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L'GARDE, INC.

GEOTHERMAL ELASTOMERIC MATERIALS TECHNOLOGY TRANSFER (GEM-TT) PROGRAM
FINAL REPORT

LTR-82-AH-111
December 1982

Prepared For
Brookhaven National Laboratory
Subcontract BNL 490316-S

DISCLAIMER

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A. R. Hirasuna
G. J. Friese
C. A. Stephens

L'Garde, Inc.
1555 Placentia Avenue
Newport Beach, California 92663
(714)645-4880

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FORWARD

The technical information reported herein has been developed by L'Garde, Inc., Newport Beach, CA under the Geothermal Elastomeric Materials Technology Transfer (GEM-TT) Program, Brookhaven National Laboratory (BNL) Subcontract BNL 490316-S and DOE Prime Contract EY-76-C-02-0016.

The work was sponsored by the DOE Division of Geothermal Energy through BNL. The Program Manager was Dr. Larry Kukacka at BNL and the cognizant managers at DOE were Mr. Clifton McFarland and Dr. Robert Reeber.

The effort is directed for L'Garde by Mr. Alan Hirasuna. The development of this report and the technology transfer achieved under the GEM-TT Program are also attributable to the following personnel: Mr. Gilbert Friese (Technology Transferee Selection), Mr. Clifford Stephens (Rubber Chemistry), Mr. Francisco Dy (Fabrication and Evaluation Testing). Invaluable support in preparing this report was provided by Mmes. Pat Wall and Judy Winslow.

The authors acknowledge the outstanding support provided by the three transferee rubber product companies and their staffs. At BJ-Hughes Rubber Products Division the liasons were Messrs. Victor Lopez, Lloyd Logsden, Ted Falkard, and William Long. At Parker Seals the liasons were Messrs. Robert Barbarin and Jerry Barsoumian. And at Precision Rubber Products the liasons were Messrs. Howard Gillette and Joe Beard. The relationships with the transferee companies and staffs were outstanding and much of the success can be attributable to their open-mindedness and unbiased pursuit of technical excellence.

STATEMENT OF OBJECTIVES

Promote broad industry use of Geothermal Elastomeric Materials developments by DOE/L'Garde through transferring the expertise to industry, substantiating the viability of the technology, and disseminating information regarding the technology.

ABSTRACT

This Final Report is a comprehensive description of the work performed by L'Garde, Inc. on the Geothermal Elastomeric Materials Technology Transfer (GEM-TT) Program performed under the Brookhaven National Laboratory Subcontract BNL 490316-S and the DOE Prime Contract EY-76-C-02-0016.

The primary objective, to promote broad use of the earlier developed elastomers technology appears to have been successfully accomplished. The results will become clear within the next couple of years. The expertise was transferred to three rubber products manufacturers, and is currently commercially available. Significant substantiation of the viability of the technology was fostered through supporting and tracking numerous test efforts in various industry laboratories and out in the field. Numerous papers were presented on the technology and information was also disseminated verbally and by providing data packages. The formal and informal technology transfer effort are described.

Several secondary spin-offs also resulted. Steps toward a better understanding of the complex technology transfer process were achieved. The experience provides a data point illustrating one way that technology transfer can be accomplished and a data point which can be used to evaluate its effectiveness. And finally studies were made assessing the potential of elastomers to perform at even higher temperatures.

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1.0 INTRODUCTION AND SUMMARY

During the period of 1976-1979, L'Garde, Inc. developed Geothermal Elastomeric Material (GEM) compounds for the U.S. DOE Division of Geothermal Energy. At the conclusion of that development, test results were beginning to indicate that the technology was a significant advance in the state-of-the-art. In view of the evolving importance of the technology, it was important to assess whether the effort should be concluded with the Final Report. In that one of the prime national objectives is to engender widespread use of Government developed technology, a valid question existed regarding disproportionate benefit that may arise out of a small additional effort to explicitly encourage transfer of the technology to the private sector. Stopping with the Final Report could have been analogous to dropping the ball at the 5 yard line.

Given all the various factors, it was decided that it was a good investment of resources to explicitly transfer the GEM technology to industry. As a result, the Geothermal Elastomeric Materials Technology Transfer (GEM-TT) Program was awarded to L'Garde, Inc. by Brookhaven National Laboratory to accomplish the transfer. This report comprehensively documents the work performed.

1.1 PROGRAM ACCOMPLISHMENTS

Though it is too early to absolutely determine, it appears that the GEM-TT Program will accomplish what it set out to do. The technology is currently available to industry through three sources who manufacture standard and custom elastomeric products. Between them they can provide the full size range of elastomeric parts from a few ounces to hundreds of pounds.

During the performance of this program, the viability of the technology was confirmed such that it is clear that the GEM technology constitutes a breakthrough. In addition, throughout the performance of this contract, information regarding the technology was continuously disseminated such that excellent product recognition was accomplished. Y267 EPDM is widely known.

The final result of all these prior efforts and the only pertinent measure of success of the overall effort, is the extent of use of the technology by the general community. Only time will tell whether the ultimate objective was accomplished, however, all present indicators lead to this conclusion.

1.2 ELASTOMER STATE-OF-THE-ART

One of the GEM developments, Y267 EPDM, has proven to be eminently successful and therefore was a focal point of this technology transfer program. The Y267 EPDM elastomer has been independently tested by other organizations in a variety of field and laboratory applications. The following are some examples. The same Y267 EPDM O-rings worked with no leaks as logging tool seals for multiple trips to 4600M (15K ft.) at 320C (608F) BHST. A packer element performed flawlessly for five months in a 204C (400F) continuous steam injection well and was retrieved at the end of the test in an as-new condition. A high-pressure Y267 EPDM packer test was performed with complete success at 232C (450F) for a 7.5 day test in sour crude with differential pressures to 138 MPa (20 ksi) and the seal condition was only very slightly changed by the test. Comprehensive compatibility testing of 34 compounds from 15 companies in geothermal brine, isobutane, and oil at 191C-266C (375-510F) showed that the Y267 EPDM was best of the 34 in all three fluids. The Y267 EPDM is clearly a state-of-the-art elastomer and at the cutting edge of the high-temperature technology.

1.3 TECHNOLOGY TRANSFER OVERVIEW

The subject or science of technology transfer (TT) is clearly complex. Being intimately involved in a TT effort provided a unique opportunity to observe it from the inside out. A preliminary effort was spent to provide a beginning understanding of the fundamentals of TT. Three critical factors are identified -- 1) Level of General Knowledge of the Technology, 2) Competitive Advantage Transferee Will Obtain, and 3) Potential of the Technology. These critical factors, their interrelationships, and specific examples are discussed.

1.4 FORMAL TECHNOLOGY TRANSFER

A formal structured technology transfer was performed. It included all steps beginning with designing a process to solicit and select the transferees, and ending with completion of the transfer such that the transferees could reliably produce parts based on the technology.

The solicitation process included public advertisements and ultimately mailings to about 500 individuals. The selection process with carefully adopted selection criteria resulted in the following three companies to which the technology was transferred:

- BJ-Hughes Rubber Products Division
- Parker Seal
- Precision Rubber Products

The transfer process was accomplished over a period greater than a year. As a result, these three companies are now commercial sources of standard and custom rubber products which are made from compounds based on the L'Garde/DOE GEM technology.

1.5 INFORMAL TECHNOLOGY TRANSFER

Though not immediately obvious, informal technology transfer is probably as important as the formal process. The informal transfer includes activities such as responding to inquiries regarding the technology, supporting tests being run by other organizations, presenting papers, participating in various pertinent forums, etc.

The informal transfer effects dissemination of information regarding the technology and promotes experiments using the technology. These activities have the dual effect of stimulating the demand for the technology while at the same time motivating the supply through substantiating the viability of the technology and the market.

1.6 HIGHER TEMPERATURE CAPABILITY ASSESSMENT

When a goal is achieved it is human nature to begin to almost immediately consider improving on the goal that was just achieved. Elastomer compounds were developed which perform well at 260C (500F), and the question that follows is what about 315C (600F)?

It was known that 260C was approaching the ultimate capability of polymers, however, it was deemed worthwhile to spend a limited effort addressing the question. Three polymers were investigated -- EPDM, a new peroxide cured fluoroelastomer (Viton), and an ethylene-acrylic (Vamac). There were no startling results. The EPDM shows serviceability at 315C, but both the Viton and Vamac do not.

2.0 PERTINENT ACCOMPLISHMENTS OF THE GEOTHERMAL ELASTOMERIC MATERIALS (GEM) TECHNOLOGY TRANSFER PROGRAM

The overall objective of transfer of nonmilitary Government technology is to engender widespread utilization of the technology. Widespread utilization of technology is beneficial to America in many ways. It creates jobs, provides a technology advantage to the USA, provides exportable products, furthers realization of national goals such as energy independence, etc. Technology transfer of the GEM technology was accomplished in two ways by directly transferring the technology to industry and by disseminating information regarding the breakthrough developments throughout the community.

The direct transfer of the GEM technology to BJ-Hughes Rubber, Parker Seal, and Precision Rubber has worked out extremely successfully. Each of these three companies is a ready commercial source for rubber parts based on Y267 EPDM technology. In addition, technology regarding the other successful polymer systems developed by L'Garde -- FKM (Viton), EPDM/FKM blend, and propylene/TFE (AFLAS) has been transferred and if the need arises that technology is in place. Establishing commercial sources where parts based on the GEM technology are readily available is critical to the utilization of the technology because an interested individual soon becomes disinterested if he cannot readily obtain experimental parts.

The GEM Technology Transfer Program enabled the DOE and Brookhaven through L'Garde to track, encourage, and report several applications of the technology. The Technology Transfer contractor provides a ready source for information and access to the technology. Furthermore, by tracking uses of the technology the results can be documented and clearly related back to the original technology. Successful experiences greatly enhance the viability of the technology especially as the successes become more numerous. The technology transferees have reported an extremely good product identification of Y267 EPDM in the geothermal, and oil and gas communities.

In summary the following pertinent results were yielded from the technology transfer effort.

- Three commercial sources for GEM technology were developed.
    - BJ-Hughes Rubber Products Division
      P.O. Box 2709, Terminal Annex
      Los Angeles, CA 90051
      (213)726-4090

    - Parker Seal
      10567 Jefferson Blvd.
      Culver City, CA 90230
      (213)837-5101

    - Precision Rubber Products
      Hartmann Drive
      Lebanon, TN 37087
      (615)444-0191

- A source for GEM and related technologies for R&D situations is available from
    - L'Garde, Inc.
      1555 Placentia Avenue
      Newport Beach, CA 92663
      (714)645-4880

- Strong confirmation was established that Y267 EPDM is breakthrough technology.

- Good general knowledge of the existence of the technology was established in the geothermal community, Y267 EPDM is widely known.

- The stage is now set for the free enterprise system to exploit the benefits of the GEM technology.

3.0 ELASTOMERS STATE-OF-THE-ART, OCTOBER 1982

Multiple elastomer development efforts were initiated under Department of Energy sponsorship. Most were not able to advance to the state where commercially applicable technology was yielded given the ever tightening budget trend. However, the Geothermal Elastomeric Materials Program[1] was able to yield commercially applicable technology. The resulting developments are at the cutting edge of elastomer technology for hostile environments for geothermal and hot oil and gas applications. This section reports many of the important accomplishments of the technology which was transferred to industry under the GEM Technology Transfer Program and is commercially available.

3.1 Y267 EPDM EMINENTLY SUCCESSFUL

The elastomer compound development by L'Garde resulted in compounds from four different polymer systems which exceeded the original contract requirements. The polymer systems include: EPDM (Nordel), FKM (Viton), EPDM/FKM Blends, and propylene/TFE (AFLAS). The packer requirements include the following environments:

Temperature - 260C (500F)
Differential - 20.7-27.6 Mpa (3000-4000 psi)
Fluid - Sour Brine
Time - 24 Hours

Though compounds from all four polymer systems exceeded the requirements, one of the compounds is eminently successful because it far exceeds the requirements and it has good processing qualities, extremely good thermochemical stability, extremely good mechanical properties, and finally, low-cost constituents. The compound, Y267 EPDM, now has significant laboratory and downhole experience as shown in the case histories given below. An additional virtue of the compound is its uncharacteristically good performance in hydrocarbons. Three experiences are quoted below, where Y267 EPDM was exposed to oil at high temperatures.

At this point in time all testing shows that there is no better elastomer than Y267 EPDM for high-temperature/pressure aqueous reducing environments. Data is building which indicates it may also be superior for high-temperature/pressure hydrocarbon reducing environments and that its inherent swell in hydrocarbon may be a positive characteristic in opposition to the traditional thinking.

3.2 EPDM IS NOT EPDM

Unfortunately, it has been common in the industry to specify an elastomer by the polymer and its hardness, e.g. 70 durometer nitrile. This is adequate for benign environments, however, in hostile environments which are becoming more and more commonplace today, this simple specification is grossly inadequate.

Generally speaking an elastomer compound is made up of about 50% polymer, i.e. EPDM, and about 50% other ingredients. Hence, there is wide latitude to vary the characteristics with the other 50%, and it is not unusual for one of the very minor ingredients to markedly change the characteristics. In addition, with EPDMs there are about seven American manufacturers and each has about ten different EPDM polymers which have varying characteristics. It is true that a polymer will afford general characteristics virtually independent of the specific compound, however, when the requirements demand the ultimate capability to be yielded, the specific compound is critical and very probably specific tailoring for the application is required. Fortunately, the Y267 EPDM is showing superior performance in a wide variety of applications beyond the original intent, static geothermal seals. When Y267 EPDM works well outside the realm for which it was designed, the opportunity remains for even better performance with specific tailoring as was done for the geothermal static seals. Given this insight, it becomes obvious that specifying polymer and hardness is grossly inadequate for hostile environments, i.e. an EPDM is not an EPDM.

Y267 EPDM specifies a recipe and a manufacturing procedure, both of which are critical to its ultimate performance. There have been several direct comparisons between Y267 EPDM technology and other EPDM compounds by L'Garde and other companies all indicating the superiority of Y267 EPDM. Otis Engineering Corp. tested over ten other EPDM compounds from three companies as 7-inch packer seals, none of which compared favorably to Y267 EPDM. Parker Seals directly compared their previous generation E692-75 EPDM against their new E962-85 EPDM which is based directly on the Y267 EPDM technology. Table 3-I shows the characteristics after ageing in steam at 288C (550F) and 316C (600F) for 168 hours. Significant differences show up at 288C and complete deterioration of the old EPDM occurs at 316C.

TABLE 3-I. Ageing in Steam by Parker Seal

TEST	Parker E692-75[a]	Parker E962-85[b]
<u>168 Hours @ 288C (550F)</u>		
Hardness, Type A, PTS (Chg., PTS)	67(-7)	82(-5)
Tensile Strength, PSI (Chg., %)	1330(-33)	1740(-19)
Elongation, % (Chg., %)	261(+47)	120(+6)
Modulus @ 100%, PSI (Chg., %)	376(-50)	1710 --
Volume Change, %	-5.3	+2.1
Compression Set, %	85.7	46.7
<u>168 Hours @ 316C (600F)</u>		
Hardness, Type A, PTS (Chg., PTS)	Deteriorated	65(-22)
Tensile Strength, PSI (Chg., %)	Deteriorated	506(-76)
Elongation, % (Chg., %)	Deteriorated	109(+14)
Modulus @ 100%, PSI (Chg., %)	Deteriorated	466 --
Volume Change, %	Deteriorated	+4.0
Compression Set, %	Deteriorated	85.7

a. Prior generation EPDM

b. Based on Y267 EPDM Technology

3.3 CASE HISTORIES

The following case histories document field and laboratory test results on Y267 EPDM which are almost entirely performed by organizations independent of L'Garde. L'Garde's participation with these companies was to provide the molded Y267 EPDM parts for their experiments.

3.3.1 Otis Engineering Corporation Packer Tests

Y267 EPDM packer seals were molded by L'Garde for tests by Otis Engineering Corporation. The seals are for a 7-inch casing packer which Otis tested in 302C (575F) and 20.7 MPa (3000 psi) differential pressure water in its laboratory simulation tester. The elements were successfully sealing after 5 days when the test was terminated. The seal was in excellent condition considering the environment and did not show any signs of reversion softening or scission. Figure 3-1 shows the post-mortem seal.

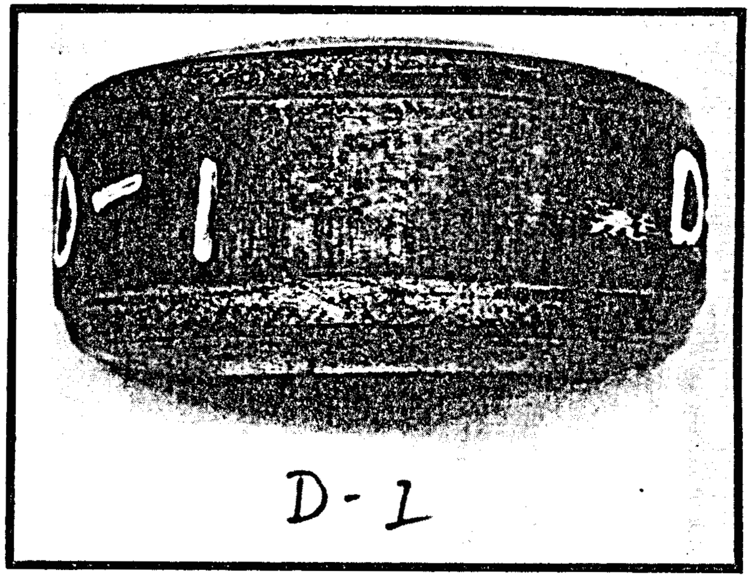


Figure 3-1. Post Mortem Y267 EPDM Seal after 5 days of testing at 302C (575F) and 20.7 MPa (3000 psi). Note the excellent condition considering the extreme severity of the environment.

Prior to this test Otis tested an EPDM from another firm but at a lower temperature, 288C (550F), and the seal failed because it lost strength due to reversion softening of the elastomer. The post-mortem seal did not have any form and did not resemble its prior molded configuration whatsoever. Otis has since tried more than ten other competing EPDM compounds from three different companies and none has yet equaled the performance of the Y267 EPDM.

In addition to the above tests, Otis also tested Y267 packer seals twice in sweet and once in sour crude. The more severe sweet crude test was at 232C (450F) and 103 MPa (15,000 psi) for 6 days. The sour crude test went to even higher pressure. It was run at 232C (450F) for 3 days at 69 MPa (10,000 psi) and then the pressure was increased for 4.5 days to 138 MPa (20,000 psi). Otis reports that the seals were undamaged and just showed only very slight swell and softening. They did not expect any elastomeric seal to hold up at 138 MPa (20,000 psi). They conclude that the Y267 EPDM is a definite candidate for this extraordinarily extreme environment.

3.3.2 Bakersfield Continuous Steam Injection

The Y267 EPDM element on an Otis R&D packer was fielded in a Bakersfield heavy-oil, continuous-steam injection well. The steam is 246C (475F) at the boiler and 204C (400F) at the wellhead.

The packer was pulled after 5 months of continuous steaming because the well required sand removal. Typically, difficulty is encountered in retrieving thermal packers, but this packer was retrieved with no problems whatsoever. Usually difficulty is encountered with the seal bonding to the casing or hardening in place, requiring the seal to be broken to enable packer retrieval.

The appearance of the recovered seal was excellent. It looked virtually like new after 5 months continuous operation, and it was apparent that it could have remained in operation indefinitely. Figure 3-2 is a photograph of the packer seal after it was removed from the Bakersfield well. The good condition is particularly significant since the Y267 EPDM seal was in contact with crude oil which covered the packer and element when it was retrieved. EPDM's are generally considered inappropriate for a hydrocarbon environment.

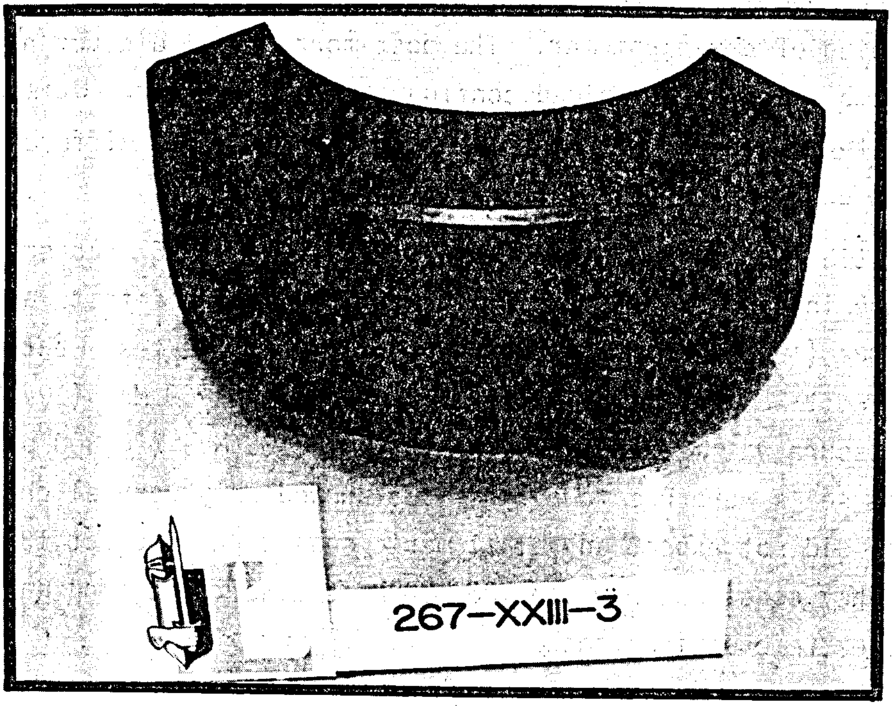


Figure 3-2. Post-Mortem Y267 EPDM Seal after 5 months in Bakersfield continuous-steam injection well at 204C (400F). Seal is virtually like new.

3.3.3 Baca, New Mexico - Geothermal Hydraulic Stimulations

Union Geothermal/Republic Geothermal - DOE performed a hydraulic fracture experiment at the Baca power plant demonstration site. The packer was set at about 915m (3000 ft.) where the formation is normally about 232C (450F). Two Otis packers with L'Garde Y267 EPDM elements were used for the fracture treatment and then subsequently for drill stem tests. This procedure was successfully performed in March and then again in October of 1981.

Normally for stimulations with temperatures above 149-177C (300-350F), Republic Geothermal takes special precautions to protect the marginal seal elements made from standard elastomer compounds. Republic first cools the well, circulates ambient water while the packer is being run, and leaves the packer unset until the moment it is needed so that ambient water circulation can be continued. Then, just prior to pumping, the packer is set.

The above procedure, required for packers with standard elastomer compounds, is unnecessary for geothermal packers with Y267 EPDM elements deployed in wells even above 260C (500F). Keeping the packer in the balancing act through this crucial period just prior to pumping is eliminated, since the packer can be set when it is run, checked out for leakage, and then forgotten, thus freeing personnel to concentrate on other critical problems. In addition, if the standard elastomeric packers fail to pack off just before pumping, expensive equipment must stand by until successful sealing is accomplished. Hypothetically, for the Baca case, if a standard elastomeric packer would have been run, and if it had needed to be replaced, it would have cost about $18,000 in standby time.

The Baca well was cooled somewhat when the stimulation packer was run, since ambient water was injected to assure that the well would remain killed. Hence, the maximum temperature the stimulation packer experienced was 160C (320F) during the 12 hours it was packed off. The maximum pressure during stimulation was 21.2 MPa (3080 psi). The drill stem test packer only saw 171C (340F) and 4.1 MPa (600 psi) because of the cooling from 8000 barrels of fracture fluid, and it remained packed off for 18.5 hours. Conditions for the second stimulation were about the same.

A total of 5 packers were set and retrieved, and as with the Bakersfield packers, all were retrieved on the first attempt with no problems whatsoever -- textbook retrievals. All 5 packer seals looked like new, which was expected at these temperatures for the Y267 EPDM, since this is well below its capability. However, the same conditions are considered to be a challenge for standard elastomer compounds. Republic commented that even with precautions taken with standard elastomers under the same conditions, the post-mortem seals would be expected to be extruded and/or cracked. Figure 3-3 shows segments of the hydraulic fracture and drill stem test seals after the first stimulation next to a new seal. Figure 3-4 shows the hydraulic fracture packer just after removal from the well.

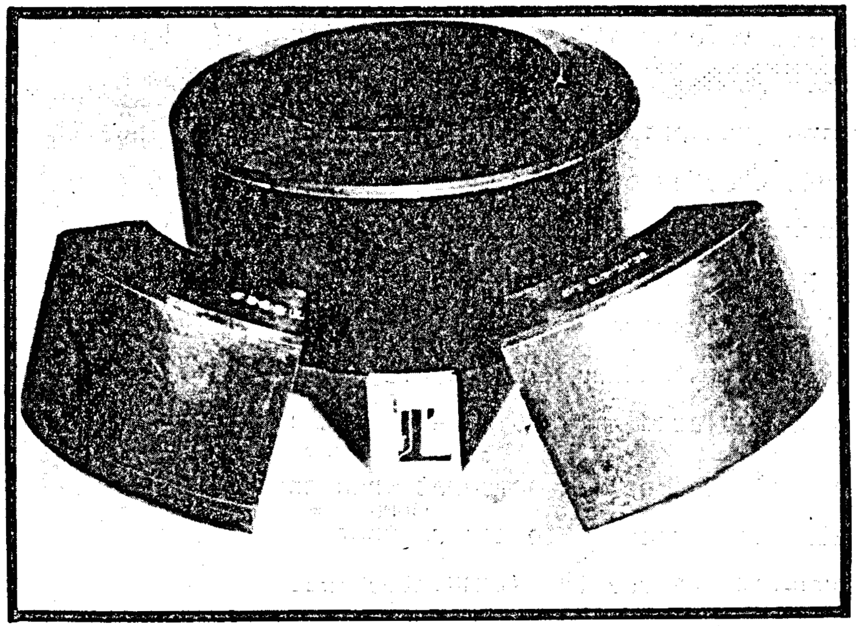


Figure 3-3. Post-Mortem Y267 EPDM Seals after 12 hours in Baca geothermal hydraulic fracture at 160C (320F) and 18.5 hours in drill-stem tests at 171C (340F). Seals are like new and are compared to a new seal element.

Figure 3-4. Post-Mortem Packer with Y267 EPDM seal after Baca hydraulic fracture. Seal is like new

3.3.4 Los Alamos National Laboratory (LANL) Tests

LANL is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock Project. LANL has shown feasibility of the concept at 200C (392F) and is currently preparing to demonstrate the concept with deeper wells and higher temperatures, 275C (525F) minimum. Because of the higher temperatures, LANL is concerned about elastomeric parts in various well and logging tools. L'Garde was awarded a contract to support LANL with the developed high-temperature elastomer expertise.

L'Garde fabricated O-rings and a cable-bend protector from the Y267 EPDM packer seal compound for the LANL cablehead. LANL tested cablehead O-rings in an autoclave in water and Mobil One oil. Twenty-four hour cycles were run with the temperature and pressure on for 8 hours and off for 16; simulating tripping in and out of the hole. The best prior performance achieved was with commercial fluoroelastomer O-rings which literally disintegrated after one cycle.

LANL ran the Y267 EPDM in water for 5 cycles or 5 days before stopping the test to examine the seals. This included a 24-hour run on the fifth day which provided a total test time of about 56 hours for this one seal. The temperature was nominally 275C (525F), and the pressure was nominally 51.7 MPa (7500 psi). The O-ring looked excellent after this test and obviously could have continued further cycling.

LANL also ran the Y267 EPDM in Mobil One oil for 4 cycles or 4 days and stopped the tests to examine the seals. The temperature was nominally 275C (525F) with an excursion to 380C (716F) during one of the 24-hour cycles. LANL estimates that the seal was exposed to approximately 340C (644F) for about one hour during the anomally. The pressure was nominally 51.7 MPa (7500 psi). The seals looked good and were sealing after 4 cycles, a.though there was some swelling from the high-temperature oil and some permanent deformation as would be expected for these conditions. LANL hopes to go ten cycles operationally before having to change out the O-rings.

These tests are landmark tests because this is the first time the Y267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range, the first time the Y267 EPDM was tested in 100% oil, the first time the Y267 EPDM was tested in oil at extreme temperatures (excursions to 340C (644F)), and the first time the Y267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDMs are generally not used in oil environments because they are vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the Y267 EPDM is serviceable and a practical option in oil.

In addition to the cablehead tests, LANL also spent significant effort on cementing wiper plugs. Poor cement jobs at Fenton Hill place question on the performance of prior elastomeric cementing wipers. Through an extended effort LANL was able to procure Dowell wipers made from Y267 EPDM.

As a matter of course LANL immersion tests wiper plugs in their autoclave. Typically they heat the autoclave to 280C (536F) at 17.2 MPa (1500 psi), hold it for 30 minutes, and then let it cool down. The autoclave and test specimen are at elevate temperature for about 12 hours. Changes in appearance and hardness are recorded.

LANL tested several wipers with different elastomers. All prior wipers showed degradation in this test, with the elastomer on one disappearing completely. The Y267 wiper has been the exception; it was unaffected by the test. Post test it appeared as new, and the hardness did not change. Shore A hardness was measured at 10 different locations before and after the test.

3.3.5 Fenton Hill, New Mexico - Logging in EE-2

Subsequent to the autoclave testing described in the previous section, LANL equipped its high-temperature cablehead with L'Garde Y267 EPDM O-rings and cable-bend protector. The cablehead was run with a temperature probe into the new EE-2 well which is about 4600M (15,000 ft.) deep with a bottomhole temperature of 317C (603F). The longest service experienced by Y267 EPDM parts at this writing is 10 hours at bottomhole and 15 hours at greater than 200C (400F). The parts sealed and looked excellent after this set of trips with the most major problem being only some permanent deformation of the O-rings. LANL plans to try for as many as 10 trips on a single set of elastomeric parts when the opportunity avails itself.

Figure 3-5 shows the O-rings which were downhole in the 317C (603F) EE-2 well.

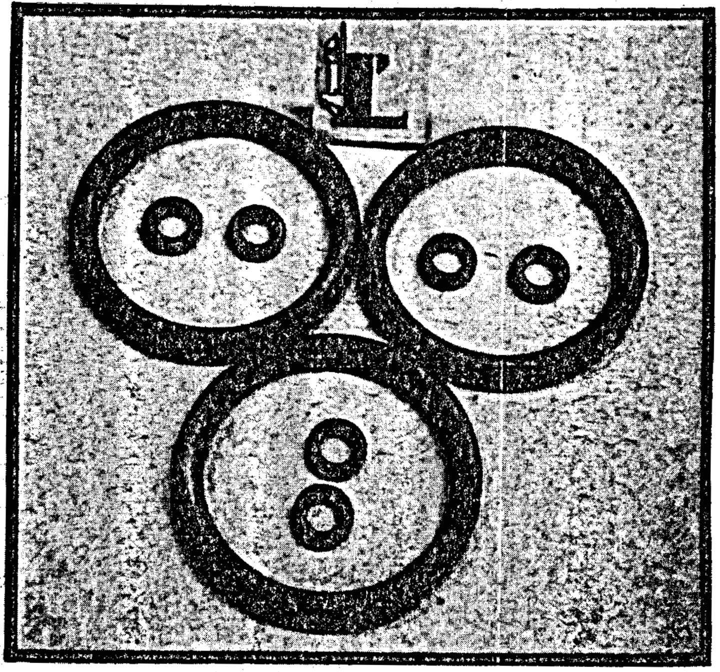


Figure 3-5. Post-Mortem Y267 EPDM cablehead O-rings after multiple trips into 317C (603F) EE-2 well at 4600M (15,000 ft.).

At this writing, one Y267 EPDM Dowell cementing wiper plug was run at Fenton Hill. A scab liner was cemented into EE-2 at 4481M (14,700 ft.) where the temperature is about 300C (575F). Though it is impossible to deduce precisely how the Y267 EPDM performed in the depths of the well, it is known that a good cement job was achieved.

3.3.6 Binary Plant Compatibility Tests

Because of the absence of compatibility data of high-performance elastomers, significant expense is often incurred as each individual project attempts by trial and error to find the right elastomer for its requirements. Recognizing this, the DOE/Brookhaven National Laboratory contracted L'Garde to run compatibility tests for elastomers for the binary power plant application.

A typical plant might be located in the Imperial Valley in an area where the brine temperature is 191C (375F) and use isobutane/isopentane as the working fluids. In addition, there may be other equipment such as downhole electrical pumps which need a high-temperature oil. Hence, static seal compatibility tests were run for synthetic brine, isobutane (represents isopentane also), and oil at nominally 191C (375F) with some testing up to 266C (510F). The oils tested are ASTM No. 1, ASTM No. 3, Chevron Cylinder 460X, and Pacer DHT-185M.

Given the test conditions, it was doubtful that any one compound would work satisfactorily in all three fluids. Nevertheless, this was highly desirable and the testing was structured to reveal this should any compound have that capability. Several of the molded-rubber product manufacturers were solicited for their most promising compound(s) for the test. Ultimately 34 compounds from 15 different companies were selected for testing. A full spectrum of high-temperature polymers were included: EPDM, Kalrez, PNF, Viton/Fluorel, Nitrile/Buna N, and AFLAS.

Immersion testing in each of the three fluids was run to screen the 34 candidates down to 8 for further evaluation. The immersion tests were run at 191C (375F) for 5 days and evaluated on the basis of change in hardness, ultimate tensile strength, ultimate elongation, weight, volume, and resilience. This screening-down process was done with keen awareness that immersion tests only test the effect of temperature and chemistry on the elastomer. At these higher temperatures, the missing mechanical environment due to differential pressures, seal setting, etc., is just as important, if not more so, than temperature and chemistry. Not surprisingly, what looked the best based on immersion tests did not look the best after subsequent full-environment static seal tests.

After selection, the 8 compounds were then further evaluated. They were immersion tested at 191C (375F) for longer periods, and tested as O-rings up to 266C (510F). Based on these tests, 4 compounds were O-ring tested at 204C (400F) and 20.7 MPa (3000 psi) for 6 months. The overall summary matrix of tests is shown in Table 3-II.

Figure 3-6 summarizes the short-term O-ring tests. All testing was done at 20.7 MPa (3000 psi), and Figure 3-6 shows only the EPDMs performing reliably up to the 266C (510F) level. Catastrophic failure occurred with many of the compounds, especially in isobutane and brine. Both the AFLAS 7170X14 and Kalrez 1018 catastrophically failed at the minimum test temperature, 191C (375F), in isobutane. The E692-75 EPDM failed in isobutane, hence, does not show across-the-board reliability like the Y267 EPDM. Figures 3-7, 3-8, and 3-9 show the Y267

TABLE 3-II. Summary Matrix
Binary Power Plant Compatibility Testing

Number Tested	Immersion (6 Fluids)			O-ring (3 Fluids)	
	5D	2 mos.	6 mos.	2D	6 Mos.
34 Compounds					
191C (375F)	X				
8 Selected					
191C (375F)		X	X	X	
232C (450F)				X	
266C (510F)				X	
4 Selected					
204C (400F)					X

EPDM after testing in the static O-ring test with a differential pressure and at the indicated temperatures. Figures 3-7, 3-8, and 3-9 are for brine, isobutane, and oil, respectively. Figure 3-10 shows the Parker E692-75 EPDM for isobutane. This compound performed quite well in general; however, results indicate that its capability is being approached for the more severe conditions. Similar static O-ring tests in 260C (500F) brine reported in Reference 1 indicate nibbling of the E692-75 in brine at 28.3 MPa (4100 psi). Hence, these conditions are about the upper limit for the E692-75, while the capability of the Y267 EPDM is indicated to be higher since it has not experienced breakage or nibbling.

Six-month, long-term O-ring tests were run completing the compatibility test program. The tests were run for approximately six months at 204C (400F) and 20.7 MPa (3000 psi) differential pressure. The long-term tests were run on a different fixture than the short-term tests. The short-term tests were strictly static-seal tests while the long-term tests permitted slow sliding, up to a cycle per day. This design was adopted strictly because of economic considerations. The four best compounds (first four in Figure 3-6) from the short-term O-ring tests were tested. Catastrophic failure occurred in isobutane and brine, again the most difficult fluids to seal. Because of the relatively mild test conditions, 204C, more choices became available, e.g. Y267, E692-75, and 501 all performed satisfactorily in ASTM #1 oil.

Compound	Rank	Temperature		
		191C(375F)	232C(450F)	266C(510F)
L'Garde EPDM Y267	1	▦	▦ Brine	▦
	1	▦	▦ Isobutane	▦
	1	▦	▦ ASTM #1 Oil	▦
Parker EPDM E692-75	1	▦	▦	▦
	2	▦	▦	☐
	2	▦	▦	▦
L'Garde Viton 501	4	○	○ Brine	☐
	5	○	☐ Isobutane	☐
	3	▦	▦ ASTM #1 Oil	○
Vernay Lab Fluorel VL1503M3	7	▦	☐	☐
	4	○	○	○
	3	▦	▦	○
Precision Fluorel 16959	8	○	☐ Brine	☐
	3	▦	○ Isobutane	○
	5	▦	○ ASTM #1 Oil	☐
Seals Eastern AFLAS 7170X14	3	○	○	☐
	7	☐	☐	☐
	6	○	○	○
L'Garde AFLAS 291	4	○	○ Brine	○
	5	○	☐ Isobutane	☐
	8	○	○ ASTM #1 Oil	○
DuPont Kalrez 1018	4	▦	○	☐
	7	☐	☐	☐
	7	○	○	○

▦ Damage limited to moderate set and swelling.

○ Severe set, cracks or splits, signs of extrusion.

☐ Nibbling or catastrophic failure.

Figure 3-6. O-ring test results. 20.7 MPa (3000 psi) ΔP.

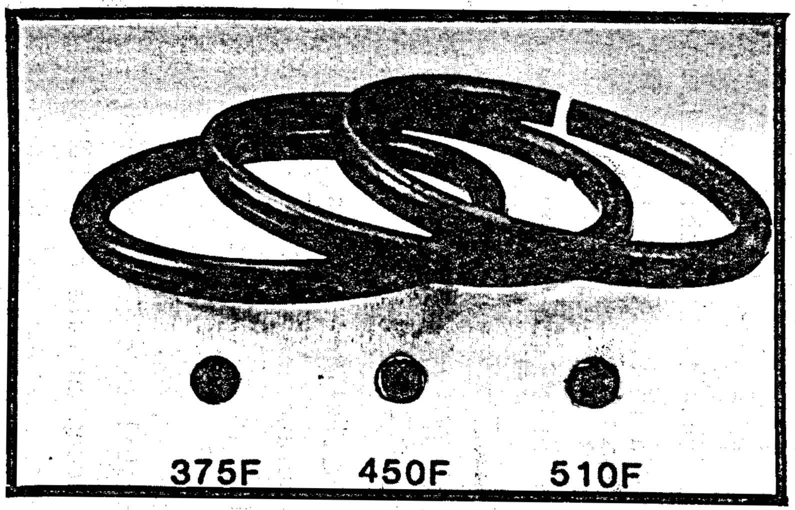


Figure 3-7. L'Garde Y267 EPDM after 2 days brine at 20.7 MPa (3000 psi).

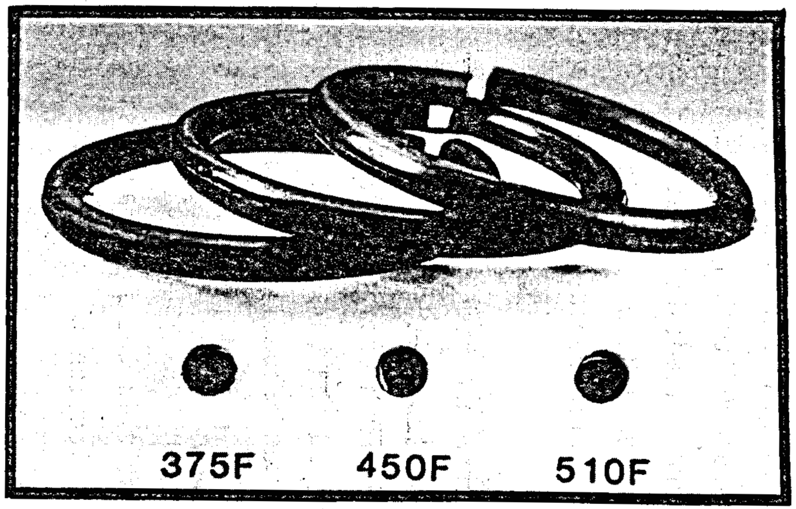


Figure 3-8. L'Garde Y267 EPDM after 2 days in isobutane at 20.7 MPa (3000 psi).

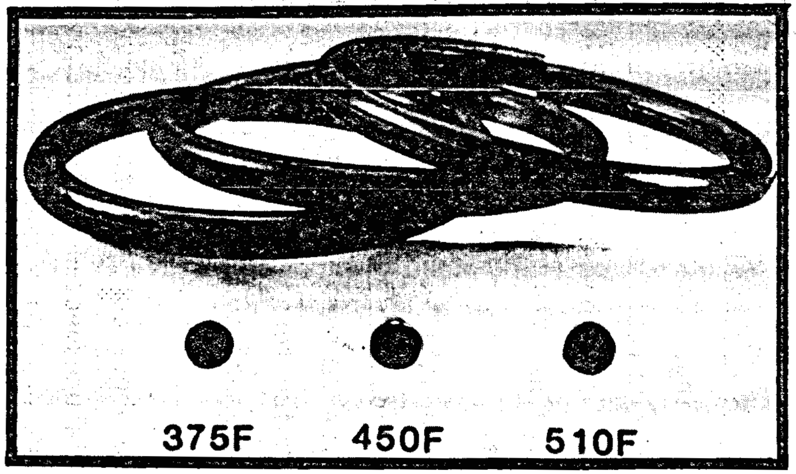


Figure 3-9. L'Garde Y267 EPDM after 2 days in ASTM No. 1 oil at 20.7 MPa (3000 psi).

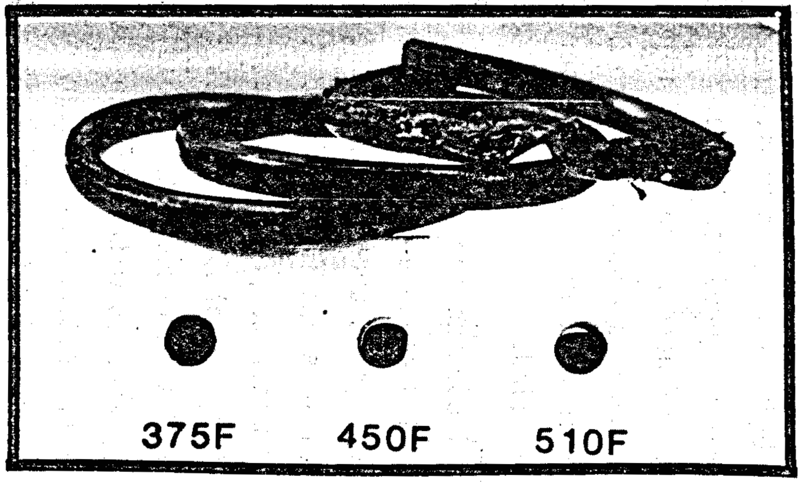


Figure 2-10. Parker E692-75 EPDM after 2 days in isobutane at 20.7 MPa (3000 psi).

In brine for six months, the Y267 looks the best which is not surprising because it was designed for brine. The Parker E692-75 EPDM test fixture failed after 56 days because a non-test O-ring in the fixture failed; but based on previous data, there is every reason to believe that it would have performed equally at this relatively mild temperature. Both the fluoroelastomers became brittle and failed.

In isobutane for six months, both E692-75 EPDM and 501 Viton are "not OK", they failed at 90 and 41 days respectively consistent with previous tests. There is significant uncertainty, however, as to Y267 EPDM and VL1503M3 Fluorel. For the conditions VL1503M3 is best because it survived the entire test, 171 days, but suffered moderate permanent set, while the Y267 suffered slight permanent set and it cracked over about 10% of its circumference but stayed intact. The Y267 crack may have originated with a molding defect and, in any event, it could probably be prevented with a backup ring. Consequently in the final analysis, these and other factors would enter into the final selection for isobutane. More testing is needed to resolve the uncertainty.

In ASTM #1 oil for six months, a broader choice exists as all four candidates survived the 209-day test period. The VL1503M3 Fluorel became brittle, suffered severe set, and slight nibbling. The EPDMs suffered some swell and moderate nibbling. The 501 Viton suffered severe set and slight nibbling, however, it did not get brittle as did the VL1503M3 or as it did in Chevron Cylinder Oil. A trade exists here between severe set and potential of getting brittle for the 501 Viton, against the moderate nibbling of the EPDMs which can be corrected with backup rings.

These data are particularly valuable because of the comparison between multiple candidates under controlled broad spectrum of environments. In overall conclusion for temperatures in excess of 232C (450F), the Y267 EPDM is the best indicated choice for all three fluids -- brine, isobutane, and ASTM #1 oil. At more benign temperatures below 204C (400F), more choices exist particularly in ASTM #1 oil.

The above conclusions regarding the serviceability of EPDMs in hydrocarbon oil is unexpected because it is a generally accepted rule that EPDMs should not be used with hydrocarbon oil. This rule is derived from immersion tests and even though the Y267 is less vulnerable to oil than most EPDMs, it does swell, and lose strength and hardness. Nevertheless, after less than five days in the environment the degradation reaches a plateau which has been measured to be maintained at least six months. The plateau properties still provide an excellent static seal. There is some evidence, in fact, that swell may be a desirable property for seals in hostile environments.

3.3.7 Other Miscellaneous Tests

Other miscellaneous tests on Y267 EPDM have been run for a variety of environments and by several organizations. These are summarized in this section.

Battelle Northwest ran into rubber seal problems on an inline corrosion probe they are testing. They ran autoclave tests in 200C (400F) brine on the Y267 EPDM prior to installing it in the field. They have tested several compounds and confirm that the Y267 EPDM stands up better than any other elastomer they have tested. They subsequently ran excellent comparative tests at the Magma Plant in the Imperial Valley. A test loop is available at the feed to the plant where several instruments can be tested simultaneously. Battelle ran corrosion probes with fluoroelastomer (FKM) perfluoroelastomer (FFKM), and Y267 EPDM seals side-by-side. Both other seals failed within 2 weeks; the FKM became brittle, and the FFKM cracked and split. The Y267 EPDM was still running after 8 months when the plant was shut down. This is the longest performance logged for this application for any elastomeric seal.

LANL/Union Oil ran an explosive stimulation experiment at the Geysers. They encountered seal problems during preliminary hot water tests with their timer/detonator vessel which is immersed in the explosive during operation. Immersion tests were subsequently run in the explosive at 246C (475F) and 6.9 MPa (1000 psi) with four different elastomer compounds and the Y267 EPDM was clearly the best performer. Tests were also successfully rerun in 260C (500F) water in an autoclave. Y267 EPDM seals were ultimately installed in both pressure vessels; the timer/detonator, and the overall container. The timer was set for 48 hours for the downhole tests and the detonation occurred at the second implying that the seals must have performed flawlessly.

LANL ran some temperature surveys for Union at the Baca using their cablehead equipped with Y267 EPDM seals. The well temperature was approximately 300C (575F) and the Y267 EPDM parts were exposed for a maximum of 4 to 6 hours. The rubber parts were changed whenever the cablehead was dismantled for other reasons.

Sandia ran autoclave tests in the vapor phase of water at 270C (500F) for 100 hours plus heat-up and cool-down. The cable BOP environment was being stimulated. The Y267 EPDM was the best of 7 compounds in this environment. It retained about 90% of its tensile strength after ageing and was recommended by Sandia for this application.

Sperry Vickers ran long-term compatibility tests of elastomers in an aqueous solution of hydrazine at 218C (425F). They saw no change in the Y267 EPDM after 85 days.

TerraTek ran drill bit seal tests on several elastomers including Y267 EPDM. The test exposes the seal to grease on one side and rock cuttings, sand, and water on the other. The specimen is raised to a presoak temperature to simulate tripping into the well, cooled to 150C (302F) to simulate introduction of cold drilling fluid, and then the dynamic test is initiated. The test provides a rotating motion and on each cycle radial and longitudinal jogs are superimposed on the motion. At the time they ran Y267 EPDM, a 40 to 50 hour run was quite respectable. With the Y267 a presoak temperature of 288C (550F) was chosen; this was a first for this high a test temperature. Both TerraTek and L'Garde were pleasantly surprised when the Y267 EPDM was still sealing after 104 hours. TerraTek remarked that the Y267 appeared excellent after the test, less degraded than all other elastomers tested.

4.0 TECHNOLOGY TRANSFER OVERVIEW

This section documents some observations regarding the technology transfer process. The process is fundamentally complex and this complexity is compounded because the subjective judgement of all people involved can make or break the effort independent of the methods or techniques employed. This section was created to document some of the few absolutes which are observed to pervade the process.

4.1 TECHNOLOGY TRANSFER MOTIVATION

The motivation to implement technology transfer exists in most segments of the private sector and Government. There is general agreement that it is desirable to have Government technology transferred into practical application in industry because of the multiple benefits to the general American economy in terms of more jobs, increased U.S. technology advantage, more exportable products, further realization of national goals such as energy independence, etc. So the fact that Government technology transfer should occur does not create debate, but the how and to what extent Government should foster transfer stimulates intense debate.

4.2 CRITICAL FACTORS

As indicated, agreement quickly deteriorates into conflict when considering the "how" of technology transfer. The debate usually centers around the extent of Government involvement required or justified to effect the transfer of technology. Generally when objective intelligent people disagree, the issue is highly complex such that each is considering a different combination of parameters or factors which can each be individually valid while leading to opposing conclusions. The following is a first attempt at understanding the factors that govern technology transfer.

There are three critical factors which are observed to affect the technology transfer process. These three factor are:

- Level of General Knowledge of the technology
- Competitive Advantage transferee will obtain
- Potential of the Technology

At the onset of the transfer process the Potential of the Technology is given, however, the Level of General Knowledge can be increased during or as part of the transfer process, and the Competitive Advantage the transferee will have is determined by the arrangement with the Government.

The level of effort required to effect transfer is strongly a function of the interrelationship of the three critical factors. To illustrate this a critical factors matrix is constructed and shown in Table 4-I.

TABLE 4-I. Critical Factors Matrix, Examples

Level of Knowledge Required	L	L	H	M
Competitive Advantage	H	M	M	L
Potential of Technology	M	H	M	H

The matrix shows various plausible combination levels of the critical factors quantified as High (H), Medium (M), or Low (L). Please note that low potential technology was not considered since this would probably not be the subject of transfer.

The extent to which Government must participate in the transfer is implied by the Level of General Knowledge required to effect transfer. If only low Level of Knowledge is required to effect transfer, Government has to do very little. If high Level of Knowledge is required to prime the transfer pump, Government has a significant effort if transfer is to be achieved. The Level of Knowledge required is affected by both the Competitive Advantage and the Potential of the Technology. Technology transfer will quickly become self propelling at a low Level of Knowledge if either or preferably the combination of the Competitive Advantage and Potential of Technology factors is high. If a favorable market position exists and/or if the technology offers high potential, little Government effort is required to transfer the technology because the more significant the opportunity the keener the interest.

In conclusion, there is a difficult trade-off that exists for each technology transfer project. Given the Potential of the Technology which is difficult to assess in the first place especially in its infant stages, there then exists a trade-off between the degree of Competitive Advantage the Government affords the transferee vs the level of effort the Government will need to expend to increase the Level of Knowledge sufficiently to effect the transfer.

This is an apples and oranges trade-off between philosophy and the realities of life, consequently the ultimate decision reduces to a subjective judgement and the correct judgement is different for each combination of circumstances.

The following paragraph provides more detail considerations of the critical factors.

4.3 DISCUSSION ELUCIDATING CRITICAL FACTORS MATRIX

This paragraph addresses various subjects which feed into the understanding of the interrelationships between the critical factors.

4.3.1 Reports

Generally speaking dissemination of reports is an inadequate mode of establishing sufficient Level of Knowledge to effect transfer. However, if the Potential of the Technology is high and the developments are advantageously presented by the developers in public forums during the normal course of the development, a small percentage of the cases will result in sufficient Level of Knowledge to effect transfer by just performing the normal tasks required by the contract.

4.3.2 Incentive Depends on Competitive Advantage and Potential of Technology

Incentive is the gasoline that powers the technology transfer car. The amount of incentive can range from a full tank where the normal contract functions are enough of a nudge to start the engine. If the tank is empty, pushing ad infinitum will not result in the engine starting. The incentive or level to which the tank is filled on any project depends on a combination of Competitive Advantage granted by the Government and the Potential of the Technology.

When the incentive is good enough, very little Government effort will be required for the technology transfer to be self propelling. The situation where little or no effort, not even reports, is required to make interested parties aware of the technology is when the interested parties are those involved with the development of the technology. The involved parties can either exploit the technology in their present organization, join another established firm, or they can form a new company around the technology.

The prior discussion regards the extremes where the incentive is either very high or very low. Obviously the majority of the cases are in between the empty and full tanks, where either the Potential of the Technology is uncertain and/or the Competitive Advantage is not clear enough to create a self propelling technology transfer. In these cases Government must foster technology transfer if it is to be achieved. It has the option of increasing the Level of Knowledge to a broader population such that a transferee is uncovered and/or it can grant greater Competitive Advantage such that there is a higher probability of a member of a smaller population becoming a transferee.

4.3.3 Knowledge Building Period

Increasing the Level of Knowledge can have three objectives. The objectives may be to uncover a transferee, to stimulate the market, or to confirm the Technology Potential. Given a transfer effort where the Government is building the Level of Knowledge, an obvious mode of efficiently disseminating information and increasing general knowledge of the technology is through presenting papers at the appropriate symposia. Implied as part and parcel to presenting the papers is generation of new data that will continue to hold the interest of the audience at subsequent symposia. New successful data regarding the technology is invaluable for building credibility of the technology.

Once transferees are identified, the knowledge building period becomes critical to transcending them into genuine bona fide transferees thru stimulating the market and confirming the Technology Potential. For these cases where Level of Knowledge needs to be increased, at first the transferee is probably mostly motivated to participate by wanting to make sure he doesn't "miss the boat". His participation is tenuous by definition because the Technology Potential is uncertain, otherwise there would be a self-propelled transfer. Consequently, the transferor is in the process of selling the transferee. The transferee can fulfill his obligations by just going through the motions of transfer or he can be sold to the point where he is a bona fide transferee and he continues forth once the formal transfer is complete to exploit the full potential of the technology. During this tenuous period the transferee must be continually increasingly convinced of the viability of the technology as well as the market. If he becomes dubious in either area, he is likely to drop out as an earnest transferee. To this end generation of new data, presentation of these data at appropriate foums is invaluable. This provides increased substantiation of the Technology Potential while also stimulating the market into making inquiries which provide a positive feedback on market potential.

4.3.4 Financial Ability

Another perturbation regards level of resources of the transferee. All other things being equal, the resources available to the transferee will affect the extent of Government involvement required for transfer. The level of resources will depend on how much a company is willing or able to allocate for these types of investments. For small companies, the level of resources can have an absolute ceiling resulting from its small size and for all concerns it will be limited by what they judge is prudent for the project.

Hence, the level of the Government involvement needed may vary due to secondary considerations. With very small companies this consideration may be completely independent of the technology.

4.3.5 GEM Case

With the GEM technology the Potential of the Technology is high, but limited because of the relatively low volume market. The Level of Knowledge was medium and the Competitive Advantage low.

Because of the limit to the Potential and the low Competitive Advantage, Government effort was required to transfer the technology. It was in the Government's interest to transfer this technology because it adds significantly to the ability of the geothermal community to efficiently develop the geothermal resource.

The Potential of the Technology is limited because the numbers of parts that will be required, though significant, is minuscule compared to say the automotive market where thousands of tons of rubber are used each year. The low Competitive Advantage results from the fact that the technology is reported in public documents and is impractical to patent. If a rubber recipe is patented, by that very act it will become public information. Once the recipe is public, it takes little effort to produce effectively the same product with a modified recipe.

The only way to have created a self propelled technology transfer in the GEM situation would have been to allow competitive advantage by allowing the recipe to be trade secret. This is what occurs in the rubber industry and this practice maintains the technology advantage for some uncertain period. Then, the secret is leaked through employees or, more likely, through suppliers of the critical components. At this point in time, there is no way to maintain Government technology trade secret.

Consequently, it was desired and justified to transfer the GEM technology because of the general national advantages stated in 4.1, in addition to the benefits that would be afforded the geothermal and, oil and gas resource development.

5.0 FORMAL TECHNOLOGY TRANSFER

Generally "Technology Transfer", implies a formal structured handover of technology from one entity to another. There is significantly more to successful transfer of technology than the obvious formal structured process. Sections 4.0 and 6.0 discuss those other aspects while this section discusses the formal structured portion of the GEM-TT Program.

5.1 OBJECTIVE OF TECHNOLOGY TRANSFER PROGRAM

In 1979, L'Garde completed DOE sponsored elastomer compound development for geothermal applications. All testing of the new developments indicated that the technology could be an important contribution to the advance of the geothermal energy alternative. In practically every phase of the development of the geothermal resource, from exploration through production, elastomeric seals play an important role. In instances where old elastomer technology is in use, special expensive and time consuming procedures are practiced to compensate for the weak-link, old-technology elastomeric seals or nonuse prevails because the result, though desired, is not worth the extensive effort.

Since the GEM technology can significantly help advance the state of development of the geothermal resource by providing increased capability in numerous obvious and subtle applications, a formal transfer of this technology was sponsored by the DOE through Brookhaven National Laboratory. Given the state of the Critical Factors for the GEM technology discussed in Section 4.0, i.e. the relatively low volume market limits the Technology Potential, and the Competitive Advantage is low because the technology is public information, it was apparent there was a need to foster the technology transfer to assure that industry would ultimately take advantage of the developments.

Because of the low Competitive Advantage it was not attractive for the technology developer, L'Garde, to phase into manufacture of rubber parts using the developed technology. It was reasoned that once a proven market was developed, then large companies with established marketing networks would obtain the technology which is documented in the public domain and compete for that same market. The only practical way a Competitive Advantage could have been created would have been to allow the recipes and processes to remain trade secrets, however, this is not a consideration under current Government contract regulations. Then the developer would have incentive to independently take the technology to the market place. Under the present circumstances he does not.

Given the circumstances, it was critical to foster transfer to assure that the technology would ultimately be commercially available. It was important that the technology L'Garde developed be transferred as completely as possible to commercial manufacturers so they would be in a good position to make elastomeric parts based on the GEM technology.

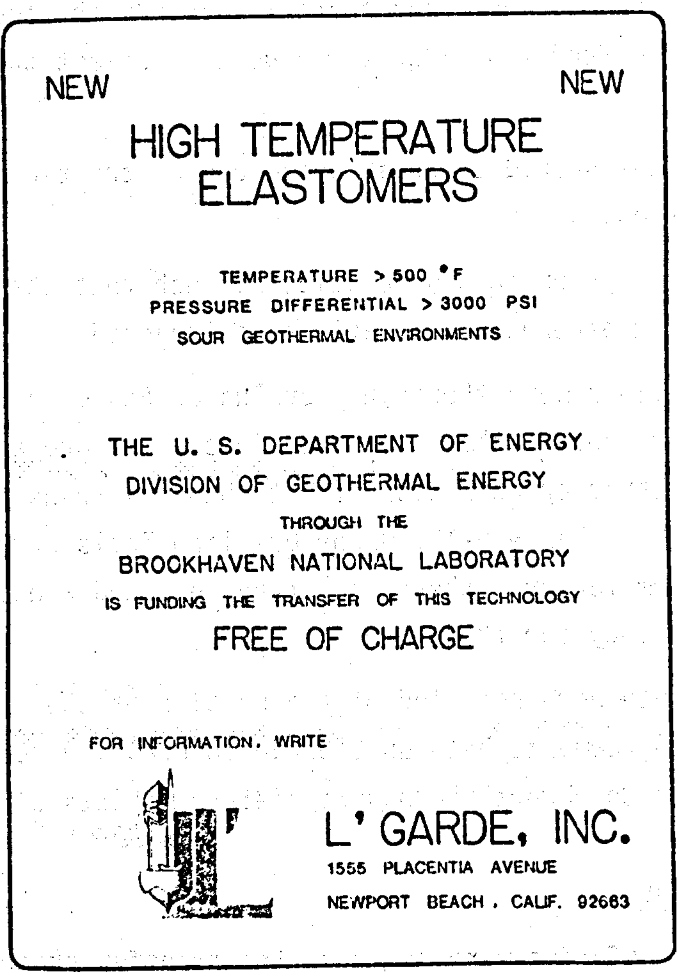
So the basic end objective of the Formal Technology Transfer was to successfully place the developed expertise into commercial companies such that parts based on the GEM technology would be available from them for industry.

5.2 TRANSFEREE SELECTION PROCESS

The transferee selection process was one of the more challenging subtasks of the formal technology transfer. There was a need to solicit applications from a very broad population and then select the optimum transferees from those that applied.

To satisfy the requirement for broad coverage and to uncover candidates from areas which may have been wrongly preconceived as not germane, public advertisements were placed. The ad shown in Figure 5-1 was placed in the Petroleum Engineer International and the Geothermal Energy Magazine. The ad ran in the November 1980 issue of each of the periodicals. In addition to the public advertisement, mailing lists were developed from the Geothermal Resources Council annual meeting attendance list, various DOE industry lists, and the Composite Catalogue.

These steps resulted in technology transfer application packages being sent to 338 companies with approximately 500 individual mailings resulting from more than one mailing sent to different addresses for a single company. The technology transfer application package is included in Appendix A and includes a letter, a description of the technology transfer program, a paper describing the technology, and an application for technology transfer.



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TEMPERATURE > 500 °F
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THE U. S. DEPARTMENT OF ENERGY
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BROOKHAVEN NATIONAL LABORATORY
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FREE OF CHARGE

FOR INFORMATION, WRITE

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1555 PLACENTIA AVENUE
NEWPORT BEACH, CALIF. 92663

Figure 5-1. Public Advertisement of Technology Transfer

The compaign resulted in nearly 100 inquiries by phone, mail, or reader inquiry card. A total of 34 companies submitted applications to participate.

Once the applications were received the other difficult aspect was addressed, i.e. selection of three companies for the technology transfer and three back-up companies who would additionally receive the technology if funds became available. The fundamental criteria used to select the transferees are the following:

- transferees should engender broad use of the technology once transferred,
- transferee group should be selected such that the broadest array of geothermal applications is impacted.

The applications were thoroughly evaluated and each organization was contacted by telephone for further clarifications. The process finally resulted in the selection of three organizations for technology transfer and three as back-ups. They are listed in Table 5-I. Oncor Drilling Tools purchases all their elastomeric seals from vendors, therefore, they selected Precision Rubber Products to receive the technology for their benefit.

All applicants were notified of the results of the application and selection process. Agreements between L'Garde (BNL/DOE) were executed committing each of the companies to report the nonproprietary aspects of the transfer.

5.3 TRANSFER PROCESS

Once the transferees were selected the transfer process was relatively straightforward, insofar as the obvious steps are concerned. The transfer occurred with all three companies in parallel.

5.3.1 Groundrules

The groundrules which governed the technology transfer were kept as simple and straightforward as possible. The following discussion documents their essence.

The financial responsibility of the effort was shared by the DOE and the transferees. All the expenses incurred by the transferor, were sponsored by the DOE through Brookhaven National Laboratory. All of the expenses incurred

TABLE 5-I. GEM Technology Transferees

Selected Transferees
BJ-Hughes, Rubber Products Division; Commerce, California
Oncor Drilling Tools; Houston, Texas Precision Rubber; Lebanon, Tennessee
Parker Seal; Culver City, California
Back-Up Transferees
NL Rig Equipment Rubber Plant; Stafford, Texas
Tri-State Oil Tool, Inc.; Boosier City, Louisiana
Vernay Laboratories; Yellow Springs, Ohio

by the transferees was self funded by each transferee.

Through the entire transfer, there was a conscious effort by the transferor to accommodate any sensitivities regarding confidentiality. There was a basic agreement that there was no requirement to report anything that is proprietary. The transferor was careful to assure that any sensitive information which was reported to the DOE did not flow outside the DOE program management circles. He was also very careful to maintain each transferees data isolated from the others and avoided passing on any benefits produced by one transferee's effort to the other transferees. From the standpoint of technology advance, it would have been most expeditious to provide full crossfeed of progress. However, from the standpoint of fostering commercialization, crossfeed is not desirable because each company is then highly motivated to line out his production and to get a better product into the market first. This follows the principles of free enterprise, allowing the company who puts forth the greatest effort to reap the greatest reward.

The obligations on the part of the transferees were few in number and simple. They were obligated to self support their effort, provide a report on their part of the technology transfer and any data during the subsequent year, and allow that information to be included in this final report and presented at the DOE conference. Proprietary data was not to be reported. The reports are in the form of papers presented at the conference described in 5.5 below.

5.3.2 Transfer Process

The steps to transfer the knowledge and developed expertise are obvious and straightforward. However, care was taken to assure that each phase of the elastomers processing was transferred such that the results could be reproduced by the transferees. Processing specifications which spell out the recipe, mixing procedure, and molding and postcuring procedures were provided. Then the series of procedures was performed by the transferee in stages such that benchmark check points were established to assure that there was reproducibility at progressive points in the overall procedure. L'Garde provided the transferee with finished test slabs and mixed rubber. The transferee then established a battery of tests which characterized the rubber processed by L'Garde alone, by L'Garde and the transferee, and by the transferee alone. These tests included a minimum of tensile properties and hardness. All measurements were made on the transferees' equipment and by his personnel to eliminate those variables.

First the transferee establishes a reference set of characteristics using the finished slabs provided by L'Garde. Second the transferee molded and postcured slabs using mixed rubber provided by L'Garde. The measured characteristics were then compared with the reference characteristics. This process was continued until there was a match implying that the molding and post curing process was reproducible by the transferee. Then the transferee performed the entire process -- mixing rubber, molding test slabs, and post curing the test slabs, and measuring the characteristics which were then compared with the reference. In general, there were adjustments which were required before the characteristics could be consistently reproduced.

Because of the eminently successful results L'Garde experienced with the Y267 EPDM compound, the transfer process was initiated with that compound and much of the effort was focused on it. In addition to the transfer steps the transferees also made parts for test with the Y267 EPDM. BJ-Hughes made drill pipe protectors, Parker Seal made O-rings for a Centrilift downhole electric pump, and Precision made lip seals for an Oncor drilling equalizer or shock sub.

Drill pipe protectors need to be upgraded to perform at higher temperatures. Generally for geothermal applications they are not used because none exist which will not hold up to geothermal temperatures. Consequently, if the drilling penetration rate slows, the probability of wearing through the casing is high. This is especially problematic for drilling in the Imperial Valley where expensive alloys are being used for casing as an interim solution to the corrosion problem. Consequently this is a perfect application for the Y267 EPDM.

BJ-Hughes has a simulation test facility for drill pipe protectors, consequently, it logically followed that they make some Y267 EPDM protectors and test them. One of the aspects of the drill pipe protector is that a good rubber/metal bond be achieved. This in itself presents a technology problem because the commercial rubber/metal coupling systems cannot withstand the high temperature, 288C (550F), used for the postcure of Y267 EPDM. This problem was an impediment to the completion of successful drill pipe protector tests, however, as reported at the DOE Conference, described in paragraph 5.5, BJ-Hughes is developing a rubber/metal coupling system and is meeting with success. Given this accomplishment, development of a geothermal drill pipe protector based on Y267 EPDM technology should be readily achievable.

Towards the beginning of the transfer effort the DOE was aware of the problems that were occurring with the downhole electric pumps. The problems ultimately surfaced as disabled pumps due to electrical shorts after very short periods in the well. This occurred even in relatively benign environments such as Raft River where the temperatures are less than 149C (300F) and the water is potable. Commercial fluoroelastomer O-rings experienced significant permanent set after being in the Raft River environment and were strongly suspected to be a source of leak.

Consequently, as an initial effort in the Technology Transfer Program, a cooperative exercise between L'Garde, Parker Seal, and Centrilift occurred. L'Garde provided mixed rubber to Parker, Parker molded 112 O-rings, L'Garde post cured them, and Parker performed final inspection. The O-rings were then delivered to Centrilift for testing in one of their downhole electric pumps. This testing was successful and Centrilift reported that the Y267 EPDM O-rings was identified as acceptable for high-temperature applications.

Geothermal drilling is generally tougher than oil and gas drilling because generally oil and gas is found in soft sedimentary formations while geothermal energy is found in tough faulted metamorphic formations. In addition, lighter fluids which afford less damping are used in geothermal drilling. Consequently, geothermal drilling can benefit more than oil and gas from use of a shock sub or shock absorber to damp out the damaging hammering action of the drill bit buttons colliding with the formation. Again, as with the drill pipe protectors, the current oil field technology will not perform at geothermal temperatures, the weak link being the elastomeric seals.

Given the obvious need, Precision Rubber cooperated with Oncor Corporation who manufacturers an oil field shock sub or what they term an "equalizer". Precision molded seals from Y267 EPDM for the Oncor 16.5 cm (6.5") O.D. shock sub. It was run in Summit County, Utah in what is reported to be an extremely tough formation. The test tool drilled 701M (2301 ft.) to a depth of 4430M (14,534 ft.) accumulating 312 rotating hours. The temperature is reported to be 107C (225F). The shock sub was still operating successfully when it was removed for inspection. Based on L'Garde's inspection of the post-mortem seals -- none had failed, no wear was apparent on the contacting lips, however, some nibbling occurred at the heel of the seals. The nibbling is attributable to the large diametral clearance in the tool design, .4 to .5 mm (15 to 20 mils), which can be easily modified. In general the Y267 EPDM technology indicates promise for this application.

In addition to transfer activity centering around the Y267 EPDM, there was transfer activity on the compounds based on the other three polymers system. Those compounds are the 255 Viton, 266 EPDM/Viton blend, and the 291 AFLAS. Similar structured transfer activity occurred with these three compounds as with the Y267 EPDM. The process specifications for all four technology transfer compounds are contained in Appendices B thru F.

5.3.3 Monthly Progress

In order to maintain the momentun and to disseminate new information on the developed elastomers two activities were regularly performed. This was a bilateral monthly verbal reporting of progress, and the maintenance and distribution of a milestone schedule for each transferee.

On a minimum of monthly frequency, L'Garde contacted each transferee for a bilateral verbal reporting of progress. These conversations consisted of L'Garde describing the latest events with respect to the technology and the transferee describing his progress during the previous month. L'Garde judges this to be a critical activity in that new reports of testing which substantiates the technology stimulates continuing motivation in the transferee and an increasingly stronger commitment to take the technology to the market place.

In addition to the verbal reports, part way through the process milestone schedules were developed for each of the transferees where objectives were plotted. This provided a constant reminder of the dates by which the goals were to be achieved which helped to keep the activities moving at a desirable pace.

Some form of both activities, monthly bilateral reporting and milestone schedules, are considered to be important to successful technology transfer.

5.4 GRAND FINALE TESTS (GFT)

The Grand Finale Tests were conceived as a confirmation that the technology was successfully transferred to each transferee. The GFT simultaneously tests static O-rings made by each of the transferees and L'Garde to confirm consistent performance.

5.4.1 GFT Test Summary

Each company furnished O-rings made from compounds based on Y267 EPDM technology for test in L'Garde's O-Ring Test Facility originally developed for the Environmental Compatibility Test Program.[2] These O-rings were subjected to 260C (500F) brine at 28.3 MPa (4100 psi) differential pressure. The temperature was cycled and the differential pressure was cycled and reversed submitting the O-rings to an extremely severe test.

The first test (lasting 13 days) showed that

a) the Precision and L'Garde O-rings were about the same;
b) the BJ-Hughes Rubber O-rings were inferior; and
c) the Parker O-rings did not see a high enough temperature (248C instead of 260C) to draw conclusions.

It was decided to repeat the test because of the latter two results. BJ-Hughes Rubber made a new batch of their compound for the second test.

The second test was run for 306 hours (13 days) -- 291 hours at 260C and 28.1 MPa differential pressure, seven hours near room temperature and 28.1 MPa differential pressure during three pressure-temperature cycles, and the remaining eight hours for heating and cooling at zero differential pressure.

After the second test it was concluded that

a) the BJ-Hughes, L'Garde and Precision O-rings were essentially identical. All held the brine at 260C but leaked slightly due to permanent set and thermal contraction near room temperature after about 10 days of testing.
b) The Parker O-rings tested were not as good as the others in 28.1 MPa differential pressure, 260C brine. The Parker O-rings started leaking after 140 hours of testing and failed to hold any differential pressure after 190 hours of testing.
c) It is important to realize that these tests provide a very limited data base and it would be a mistake to make firm conclusions strictly from these data. Given the circumstances L'Garde recommends for any

critical application, that rubber parts procured from at least two of the transferees be evaluated. There is a distinct likelihood that for one given set of circumstances one company's parts may out perform anothers and for another set of circumstances the vice versa, though both were based on Y267 technology. Differences in processing, equipment, or modifications of the recipe exist. In addition, every company occasionally makes a bad batch.

Figures 5-2 through 5-5 show the O-rings after Test 2. Parker's O-rings were nibbling away; considerable residue was downstream. All experienced permanent set; that is, their cross sections squared which is expected at these extreme conditions, 260C and 28.1 MPa.

5.4.2 Test Set-Up

Figure 5-6 shows the pressure control and instrumentation. The N_2 Tank 2 is the source for the front face pressure. Nitrogen pressure in the pump drive cylinder results in a 11.7 pressure amplification in the displacement pump. This intensified pressure is transmitted to the four accumulators by hydraulic fluid. Each of the four piston accumulators contains 115 cc of the test fluid and serves one of the elastomers being tested.

Should one of the compounds fail, the piston accumulator bottoms out, forcing its contents past the failed O-ring into the back pressure accumulator. The back face fluid is held at a constant pressure by the N_2 Tank 1.

The test section (Figure 5-7) is heated by eight strip heaters on the outside of the Inconel pressure vessel. Temperature feed-back for control is provided by a single thermocouple. A controller automatically adjusts the voltage to the heaters so that heating is constant.

Figure 5-7 shows eight O-rings -- two of each compound. The high pressure test fluid comes up each of four passages in the inner stainless steel cylinder and is led to a small passage in the stainless steel O-ring holder between two O-rings of the same compound. At failure, the fluid moves to the center of the O-ring holder and out through the cap.

The as-measured dimensions of the O-ring interfaces are provided in Figure 5-8. The downstream gap is 0.09 mm (3.4 mils) or a diametral gap of 0.175 mm (6.9 mils).

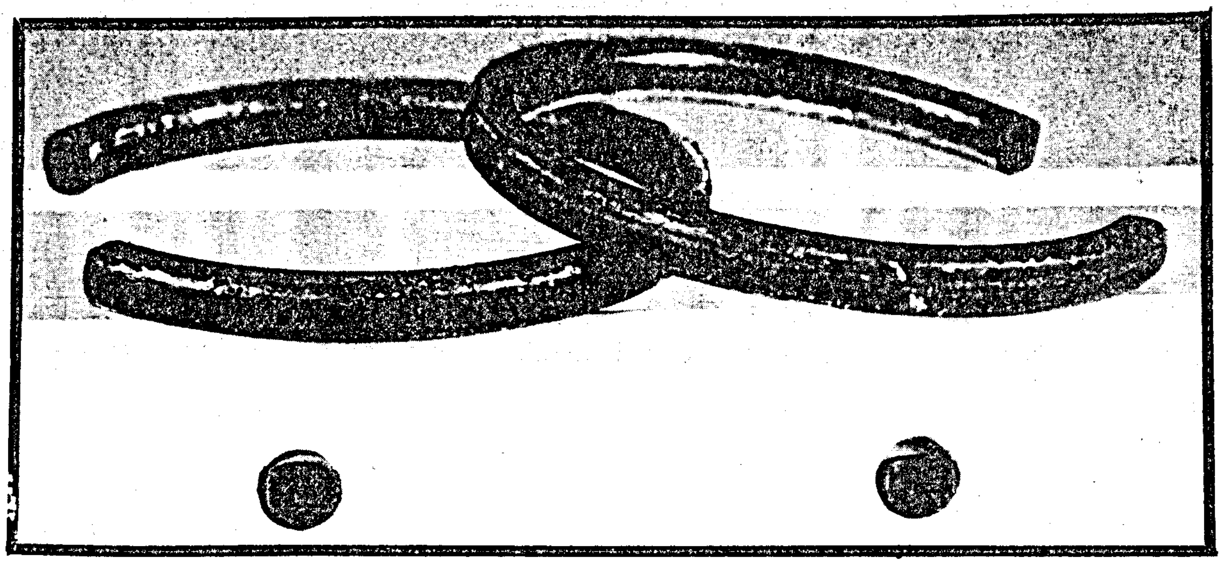


Figure 5-2. L'Garde EPDM Y267

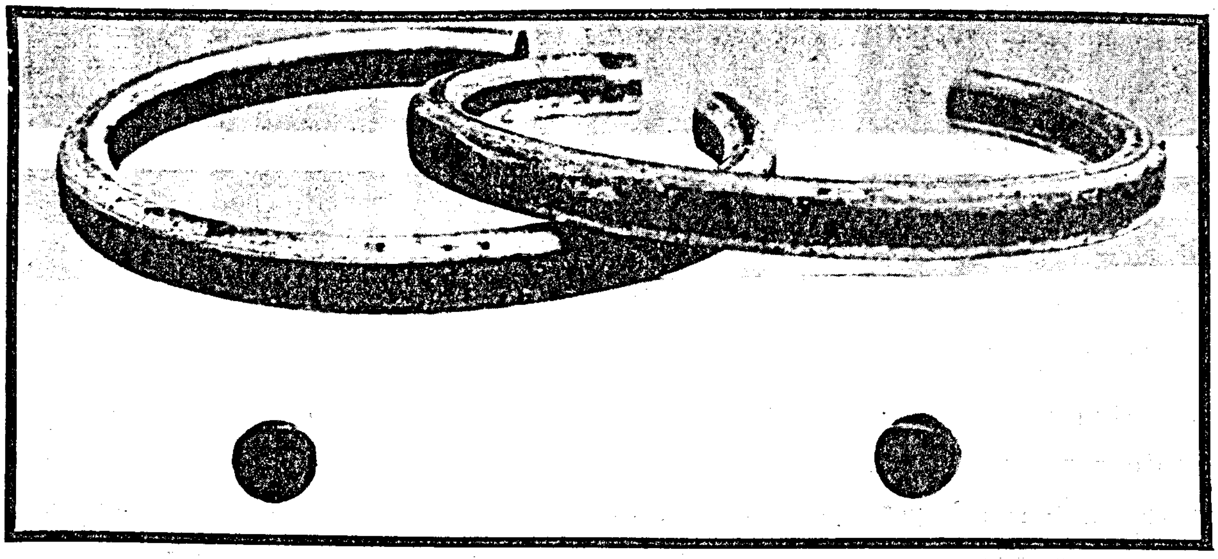


Figure 5-3. Precision Rubber EPDM 42679

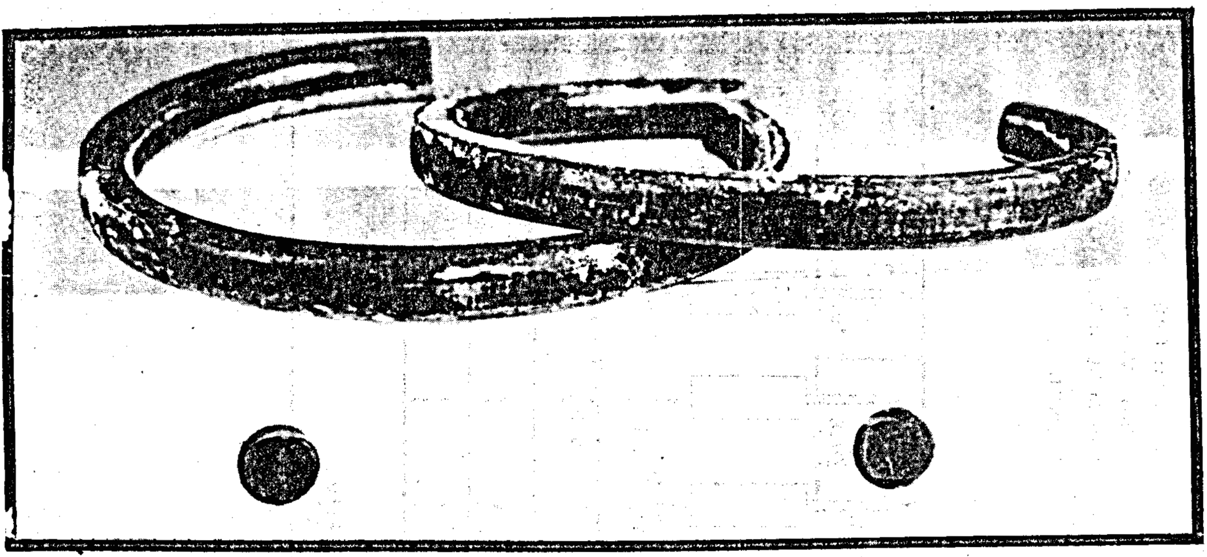


Figure 5-4. BJ-Hughes Rubber EPDM N-22-1

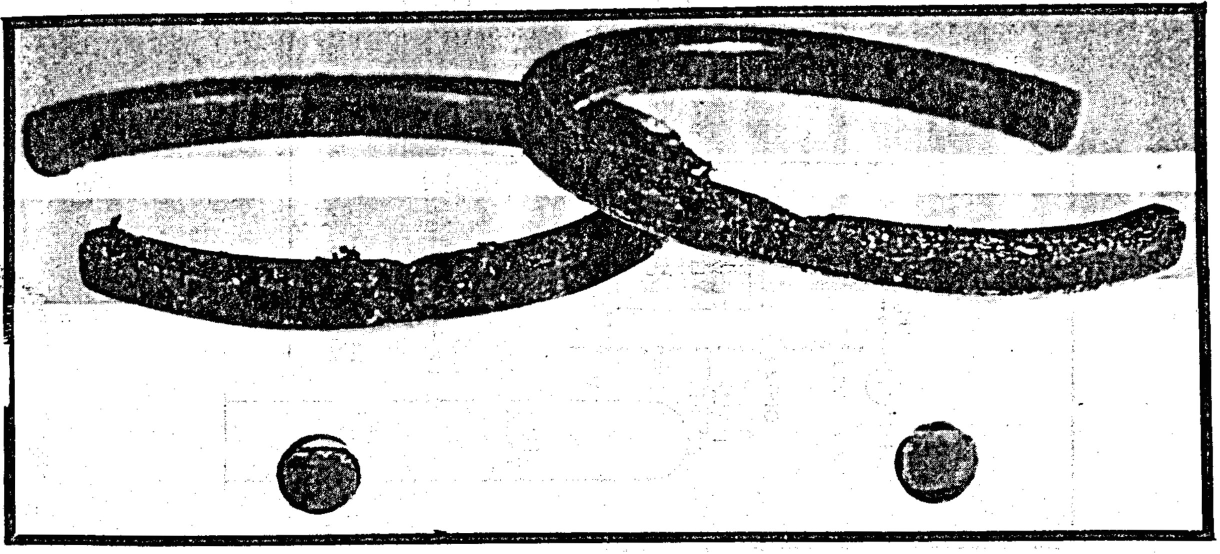


Figure 5-5. Parker Seal EPDM E962-85

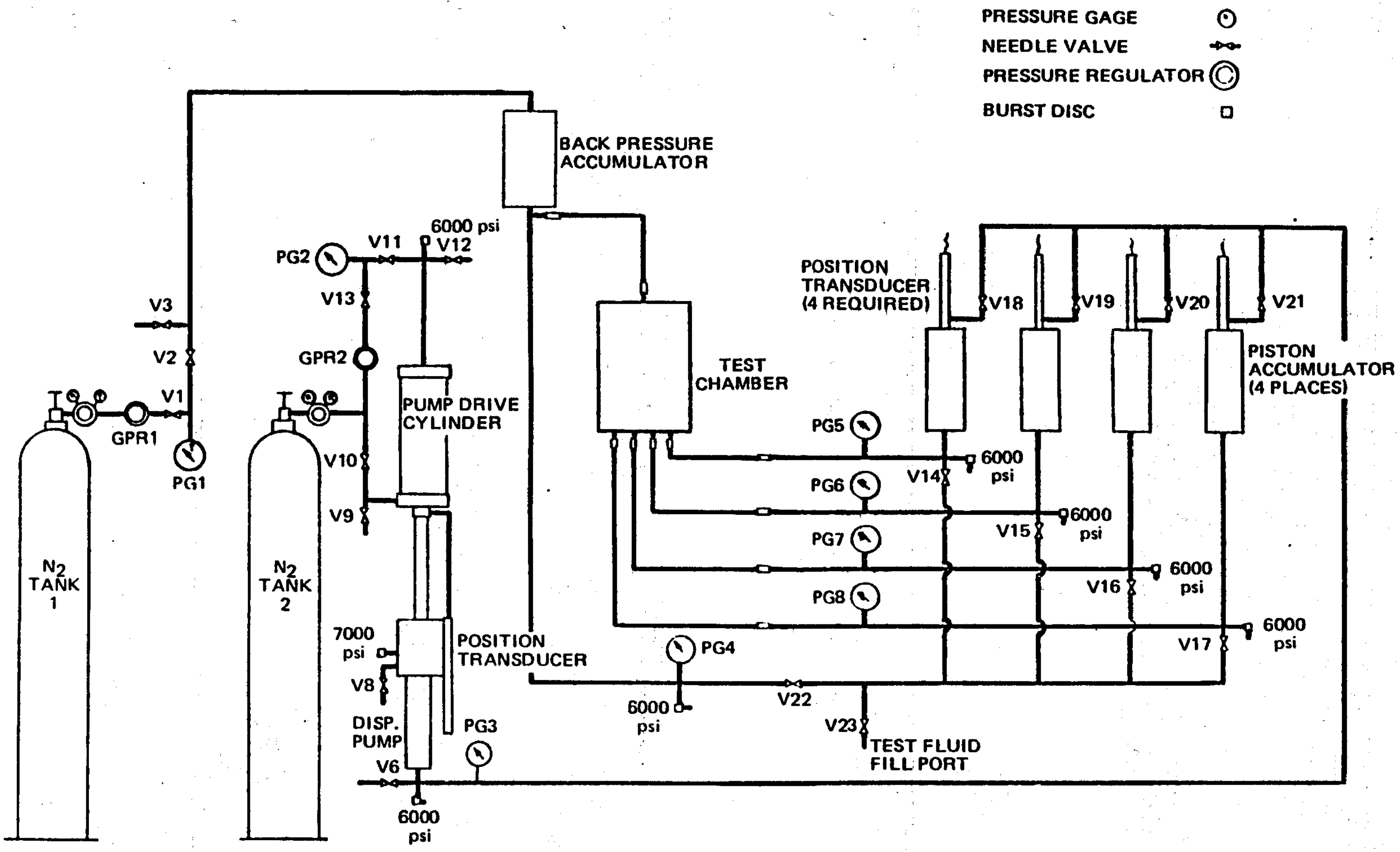


Figure 5-6. O-Ring Test Schematic

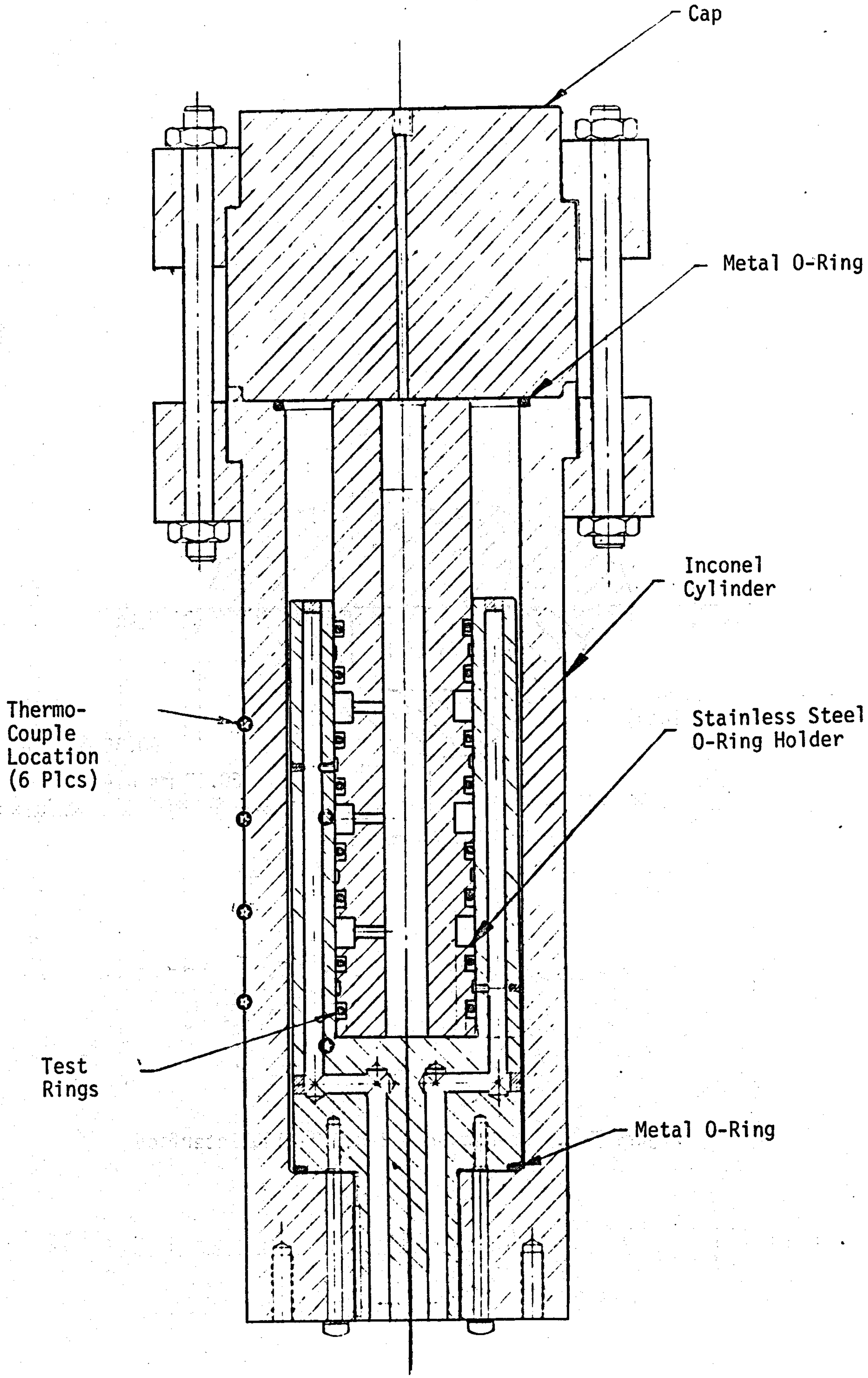


Figure 5-7. Test Section

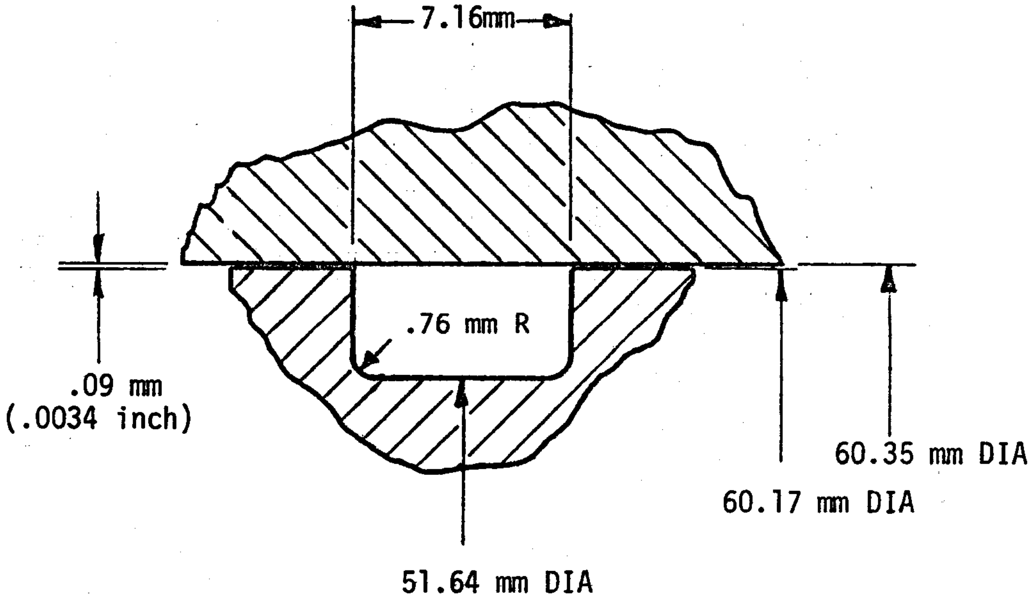


Figure 5-8. As-Measured 2-329 O-Ring Interface

Instrumentation consists of eight pressure gages (Figure 5-6), six thermocouples (Figure 5-7), and four accumulator piston position locators (Figure 5-6). The lateral distances from the thermocouples to the center of the O-ring grooves are 41.9 and 6.9 mm for the outer and inner thermocouples, respectively. The thermocouples and pressure gages are read and recorded manually. The piston positons are recorded on a strip chart so that leak rate and/or time of catastrophic failure is easily determined.

5.4.3 Test 1

Test 1 resulted in no catastrophic failures, but there was leakage during the temperature cycles. Based on this and upon post-test O-ring measurements:

a) It was concluded that BJ-Hughes N-22-1 O-rings were not as good as L'Garde Y267 O-rings for the conditions tested.

b) It was concluded that the Precision 42679 and L'Garde Y267 O-rings were essentially identical.

c) No conclusions could be made regarding Parker E962-85 because its temperature was 11-12C lower than the others. Per Arrhenius, this is equivalent to 40% of the exposure time at 260C (5½ days instead of 13 days). At the lower temperature, 248C, the Parker O-rings did not shrink as much as the others, nor did they leak.

It was recommended that the test be repeated:

a) With newly made BJ-Hughes O-rings, to validate the above conclusions, and

b) With the Parker and Precision O-ring positions on the holder interchanged so the former sees the full test temperature.

This was done. Test 2 is summarized in Section 5.4.4. Test 1 is summarized below.

5.4.3.1 Test Procedure

a) Determined the location of the compounds on the O-ring holder by chance using a random number generator.

b) Installed the O-rings on the holder and installed the holder in the test section.

c) Evacuated the system and back-filled with brine -- such that the fluid was on both sides of the O-rings.

d) Turned on heat.

e) Applied the front face pressure after one hour of heating (which was the approximate time to reach the test temperature). This was considered the start of the test.

f) Monitored the test and make adjustments.

g) Changed differential pressure to about -2 MPa for 5 minutes after 125 hours (5.2 days) and again after 219 hours (9.1 days) from start of test. This was done to encourage failure.

h) Turned off heat 287 hours after Step 4 and air cooled with fan; allowed pressure to decay with reducing temperature.

i) Repressurized when the O-rings were approximately 57C (135F), 2 hours after start of cooling. Monitored leak rates for 1-2 hours.

j) With pressure on, turned on heat 290.5 hours from start of test.

k) Turned off heat 309 hours from start of test and air cooled with fan. After an hour, reduced differential pressure to zero.

l) With temperature at 68C and decreasing, applied 28.3 MPa (4100 psi) front face pressure (zero back face pressure) and monitored leak rate for 2.7 hours while the temperature dropped to 29C.

m) Depressurized.

n) Removed O-ring holder, recorded observations, and photographed.

5.4.3.2 O-Ring Temperatures

While nominal test temperature was 260C, actual temperatures are different because of a) temperature changes with time, and b) temperature gradients along the test section. Six thermocouples were used to determine actual average temperatures. Four were located under the strip heaters and two are located close to the O-rings as previously illustrated in Figure 5-7.

Thermocouple T6, which is 6.9 mm outboard of the O-rings and near the center of the 8 O-rings, was used to plot the temperature time history shown in Figure 5-9. Readings were taken manually. All readings are connected by straight lines. The average T6 temperature was calculated at 259C (499F) and only varied by ±2C at the extremes.

The thermal gradients are reconstructed in Figure 5-10. T1 through T4 are 41.9 mm from the O-ring groove cylinder, while T5 and T6 are 6.9 mm away. The latter are close enough to the O-rings to represent their temperatures. Extrapolation from T6 was done parallel to the T1-T4 temperatures.

From Figure 5-10, the temperatures of the compounds were:

Precision 42679	260C
L'Garde Y267	259C
BJ-Hughes N-22-1	259C
Parker E962-85	248C

The Parker O-ring was located closest to the base of the test fixture where the largest heat loss occurs. This probably contributed to the lower temperature. The difference is greater than that seen in all prior tests, where historically the maximum difference has been 2C.

5.4.3.3 Pressure

The nominal front face pressure was 34.5 MPa (5000 psia) and the nominal back face pressure was 6.2 MPa (900 psi). The back face pressure kept the H_2S and CO_2 in solution.

Due to the high design differential pressure, the displacement pump has a packed seal which has fairly high friction. Figure 5-11 provides the pressure history as recorded. Data are connected by straight lines. If there were continuous data, it is unlikely that the curves would look like those in the figures. The main thing that is obtained from these curves is the probable differential pressure extremes of 26.4 to 29.3 MPa. The average pressure was 28.1 MPa (4075 psi). The test equipment pressure tolerance was therefore about ± 6%.

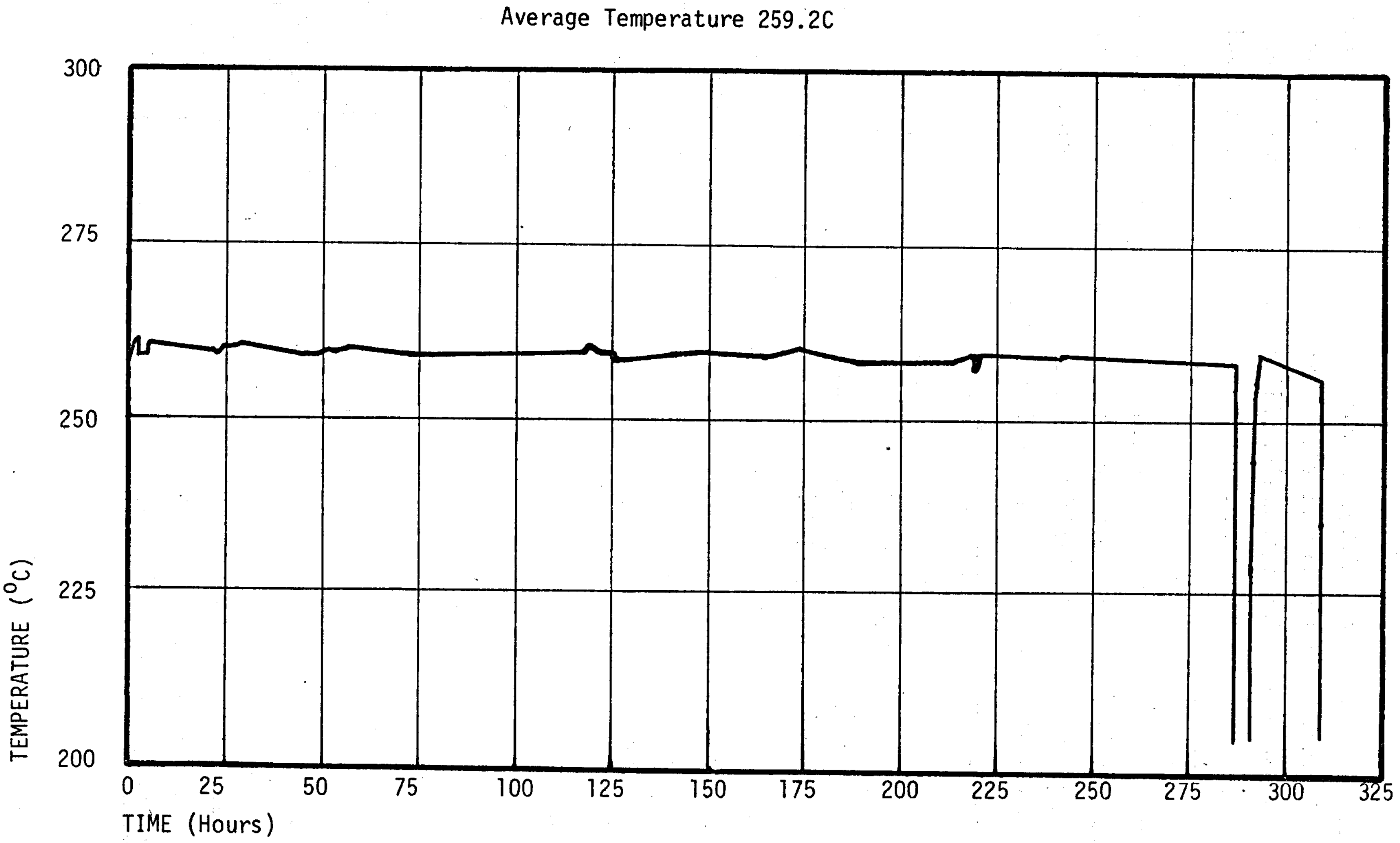


Figure 5-9. Thermocouple T6 Temperature History

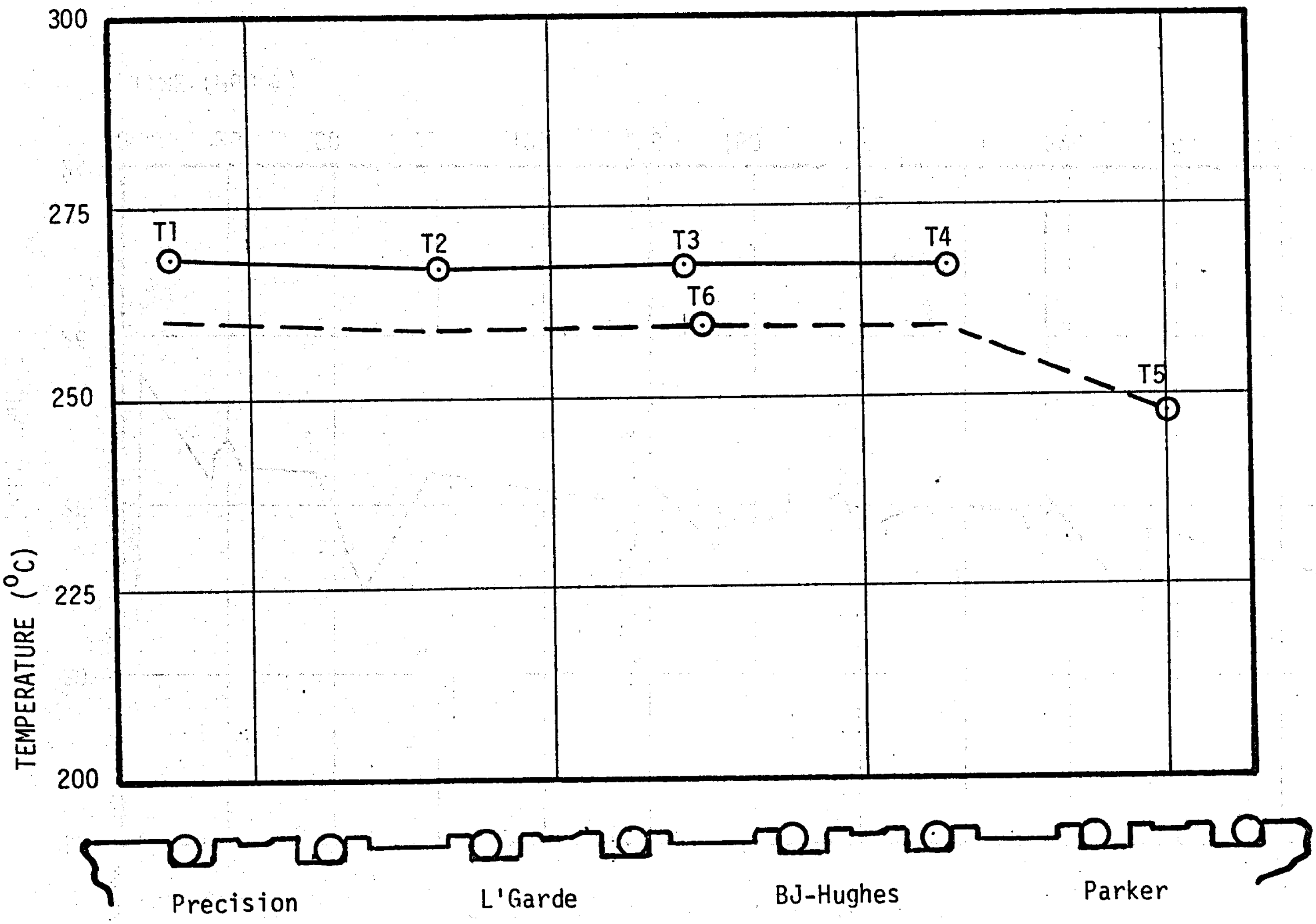


Figure 5-10. Test Temperature Gradients

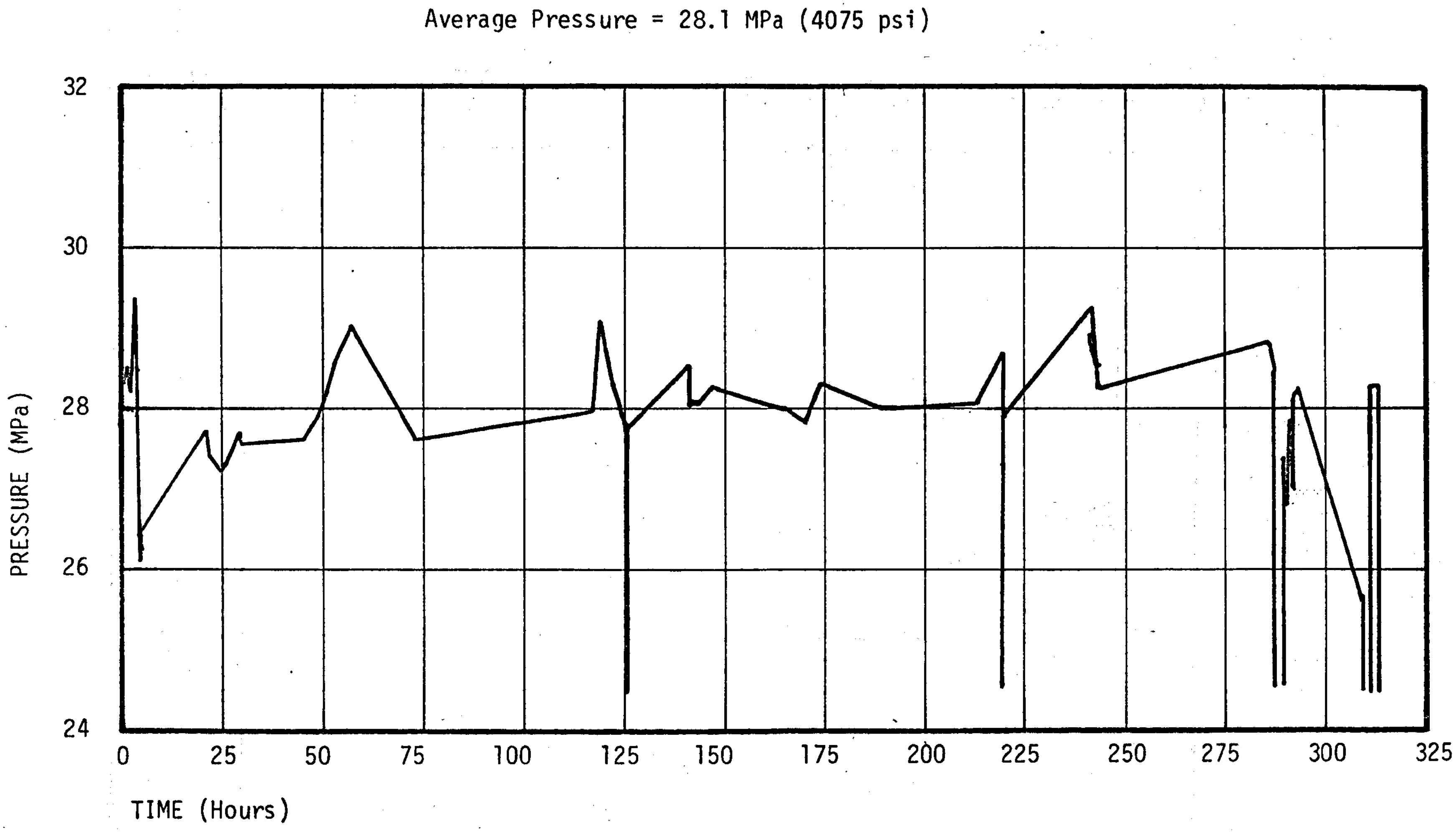


Figure 5-11. Test Pressure History

Shown on Figure 5-11 are the times (125 and 219 hours) at which the system differential pressure was reversed and held for about five minutes. This was done to flex the O-rings and encourage failure. Also shown at 287 and 309 hours is the pressure drop temporarily associated with temperature cycling (cooling to near room temperature). See Steps 8 through 12 of the Test Procedure (Paragraph 5.4.3.1) for clarification of the latter.

5.4.3.4 Test Results and Evaluation

There were no catastrophic failures; however, fluid did leak past the O-rings, as seen in Table 5-II, during this thirteen day test.

During the first 125 hours at 260C and 28 MPa differential pressure, there was no leakage. Then, the differential pressure was changed from +28 MPa to -2 MPa to move the O-ring and encourage failure. Shortly after the 28 MPa pressure was re-applied, the BJ-Hughes O-ring started to leak at 10 cc per day (24 hours).

At 219 hours (94 hours later), the pressure was again reversed for five minutes. No changes occurred during the 68 hours after pressure was re-applied. The BJ-Hughes O-ring continued to leak at 10 cc/day.

At 287 hours, the heat was turned off. Two hours later the front face pressure was dropped to 6 MPa so that the differential pressure was zero. Then it was increased back to 28 MPa. At this time, the temperature of the O-rings was about 61C. Over the next hour, all compounds except Parker's leaked (Table 5-II). Precision's and L'Garde's O-rings leaked at around 100 cc/day while BJ-Hughes leaked at about 600 cc/day.

Pressure was maintained and the heat was turned back on. All but BJ-Hughes stopped leaking as before. The cooling procedure was then repeated with about the same results as before also.

The O-rings on the holder were examined. All were permanently set such that the three sides facing metal were almost flat. This permanent set is expected at these hostile conditions and especially without form-fitting back-up rings. The high pressure was used in this test to highly stress the compounds in order to encourage failure.

TABLE 5-II. Test 1 Leak Rates

(28 MPa Differential Pressure)

TIME PERIOD (Hours)	TEMPERATURE (^{o}C)	Leak Rate (cc/day)			
		PRECISION	L'GARDE	BJ-HUGHES	PARKER
0 - 125	260	0	0	0	0
Reverse ΔP					
125 - 219	260	0	0	10	0
Reverse ΔP					
219 - 287	260	0	0	10	0
Zero ΔP					
289 - 290	61-54	130	110	550	0
290 - 309	260	0	0	10	0
Zero ΔP					
311-314	68-29	50	90	670	0

The diameters of the O-rings were measured while on the holders. Also the thicknesses of virgin O-rings (but not the ones tested) were also measured. The results are shown in Table 5-III.

The BJ-Hughes O-rings initially were thicker than the others and yet they shrunk the most -- ending up with a gap of as much as 0.07 mm at room temperature between the O-ring and the Inconel outer wall. The 10 cc/day leakage at 260C (Table 5-II) is attributable to this shrinkage. Therefore, it must be concluded that the BJ-Hughes O-rings tested is not as good as L'Garde's Y267 for the conditions tested.

Table 5-III shows that the L'Garde O-ring was probably thicker than Precision's by 0.08 mm. The last column shows that L'Garde's "interference" was also greater by about 0.08 mm. Therefore, the two compounds contracted by about the same amount. Table 5-II showed that the two compounds performed about the same -- not leaking at 260C, but leaking at about 100 cc/day cold. The discrepancy is that L'Garde's sealing performance should be better than Precision's because of the greater "interference". It is concluded that the Precision 42679 and L'Garde Y267 compounds are essentially identical.

Parker's O-rings were among the thinnest and yet contracted the least (Table 5-III) and did not leak during the test (Table 5-II). Unfortunatelyl, the Parker O-rings ran at a lower temperature than the others (248C versus 259C-260C) as seen in Figure 5-10. This 11C-12C (20F-22F) difference is significant. Per Arrhenius, 13 days at 248C is equivalent to 5½ days at 260C. Therefore, a conclusion cannot be made regarding Parker E962-85.

5.4.4 Test 2

Test 2 was conducted to determine if the BJ-Hughes N-22-1 compound used in Test 1 was a bad batch, and to derive conclusions for Parker E962-85 compound. BJ-Hughes made a new set of O-rings for this test. Also, more insulation was added to the L'Garde O-Ring Test Facility to reduce the temperature gradient observed in Test 1.

It was concluded from Test 2 that, for the test conditions,

a) the L'Garde Y267, Precision and BJ-Hughes N-22-1 O-rings were essentially equal, and

TABLE 5-III. Test 1 Dimensional Analysis

Compound	Thickness of Virgin O-ring * (mm)	Diameter of Aged O-ring on Holder (mm)	Diameter of Male (mm)	Interference on the Radius ** (mm)
Precision 42679	5.24	60.36 to 60.44	60.34	+0.02 to +0.10
L'Garde Y267	5.32	60.44 to 60.53	60.34	+0.10 to +0.19
BJ-Hughes N-22-1	5.36	60.27 to 60.34	60.34	-0.17 to 0.00
Parker E962-85	5.27	60.56 to 60.60	60.34	+0.22 to +0.26

*The thickness of a 2-329 O-ring is required to be between 5.21 and 5.46 mm. Therefore, all O-rings are within specification.

**"Interference on the Radius" is equal to ½ ("Diameter of Aged O-ring on Holder" - "Diameter of Female").

b) the Parker E962-85 O-rings did not perform as well as the other three compounds.

5.4.4.1 Test Procedure

a) Installed the O-rings on the holder such that the Precision compound would be in the coolest spot and installed the holder in the test section. (The Precision compound had proved itself in Test 1.)

b) Evacuated the system and back-filled with brine -- such that the fluid was on both sides of the O-rings.

c) Turned on heat.

d) Applied the front face pressure after one hour of heating (which was the approximate time to reach the test temperature). This is considered the start of the test.

e) Monitored the test and made adjustments.

f) At 135 hours, turned off heat, brought the front face pressure below the back face pressure, and then equalized the pressure on both sides of the rings.

g) At 137 hours (with the temperature at 71C), applied a differential pressure of 28.3 MPa.

h) At 139 hours, equalized pressures and turned on heat.

i) At 141 hours, applied a differential pressure of 28.3 MPa.

j) Repeated Steps 6 through 9 at 231, 233, 236, and 237 hours, respectively. (Second pressure-temperature cycle)

k. Repeated Steps 6 and 7 at 303 and 304 hours, respectively. (Third cool-down)

l) Depressurized.

m) Removed O-ring holder, recorded observations, and photographed.

5.4.4.2 O-Ring Temperatures

As with Test 1, temperatures were recorded manually. The temperature of the T6 thermocouple (located 6.9 mm outboard of the O-rings near the center of the set) was calculated from the data to be at an average of 260C (500F) over the high temperature portion of the test (291.4 hours). Except for very brief excursions to as low as 242C and to as high as 264C early in the test, the 260C was held to within ± 1C.

A major problem with the first test was that there was a large thermal gradient near the base of the test fixture. The difference between T5 and T6 was 11C. To reduce this, 50% more insulation was placed under the fixture, and the insulation around the above the fixture was more than doubled.

Because of this, the gradient was reduced to 5C. This was considered still high, so one of the eight strip heaters near the top of the fixture was turned off after the system reached temperature. The gradient dropped to a reasonable 2C.

Figure 5-12 shows the temperature gradients with and without one strip heater off. The scale is the same as that of Figure 5-10 for comparison. The extra insulation clearly helped since the outside thermocouples (like T3) and the inside thermocouples (like T6) are much closer in temperature (3C instead of 8C).

With the heater on top of T2 off, that thermocouple was lower than adjacent ones by 2½C. This gradient will disappear by the time it reaches the O-ring. Figure 5-7 shows that the energy must pass through two metal parts as before reaching the O-rings. The distance is 41.9 mm (1.65 inches).. Therefore, the extrapolation shown on the lowest curve of Figure 5-12 is considered reasonable.

The temperatures of the O-rings are deduced to have been:

BJ-Hughes	260C
L'Garde	260C
Parker	260C
Precision	258C

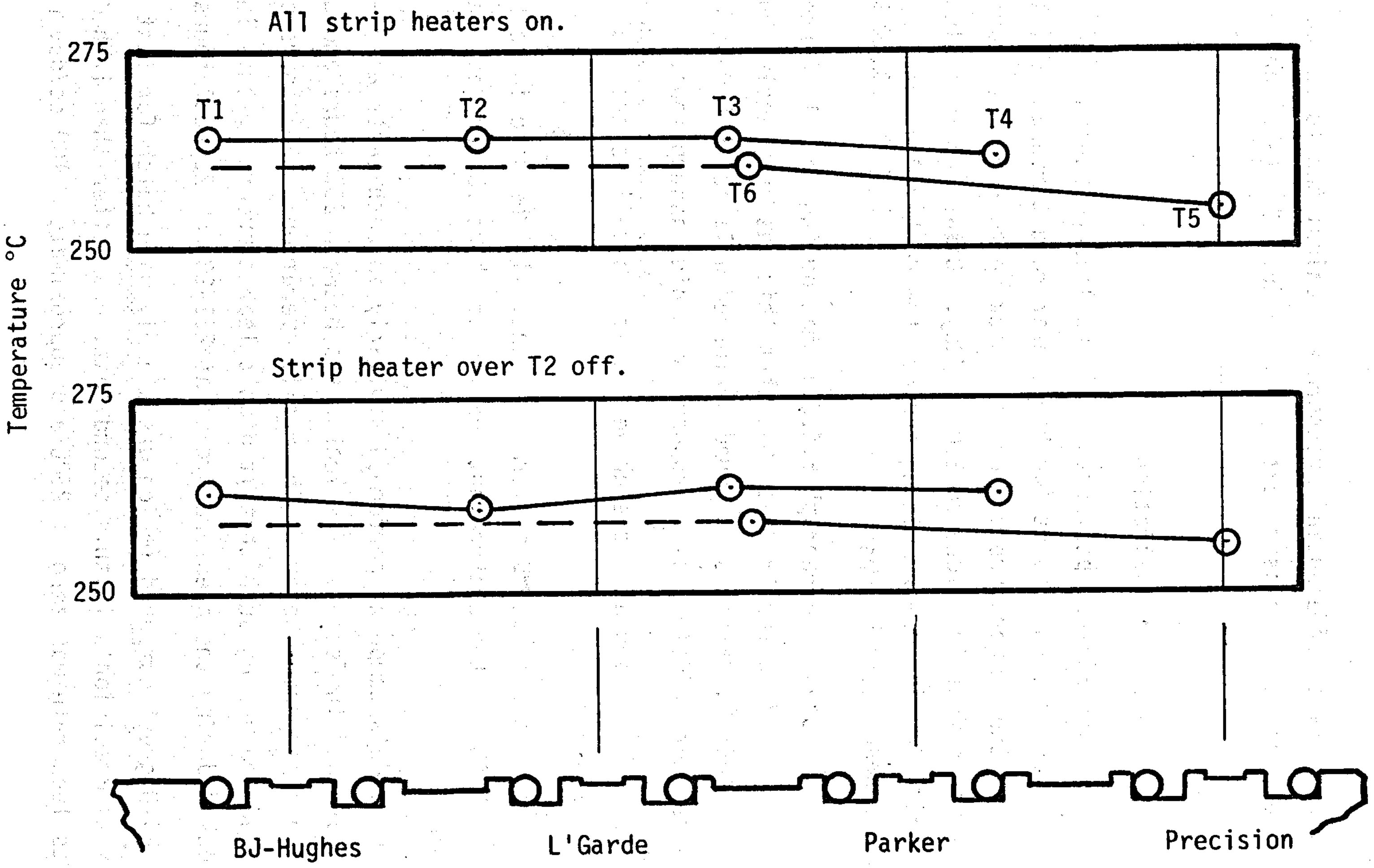


Figure 5-12. Test 2 Temperature Gradients

5.4.4.3 Pressure

The pressure history was similar to that reported in Section 5.4.3.3 and Figure 5-11.

The average differential pressure was calculated to be 28.1 MPa (4076 psi). The differential pressure extremes were 26.2 MPa (-7%) and 32.2 MPa (+15%).

5.4.4.4 Test Results and Evaluation

The Parker E962-85 performed well (no leakage) during the first 135 hours at 260C and the subsequent two hours near room temperature (71-32C). However, after the temperature was brought back up to 260C, it started leaking at various rates from 30 cc/day to 2300 cc/day. Catastrophic failure occurred at 190 hours. Through the remaining cycles, the Parker O-rings were unable to hold any differential pressure.

It was noted in Paragraph 5.4.3.4 that during Test 1, Parker's compound performed well at 248C for 13 days, but that this was only equivalent to 5½ days at 260C. In Test 2, Parker's compound performed well for 5.8 days at 260C, but failed thereafter. This tends to support Arrhenius assumptions.

Table 5-IV tabulates the above results as well as the leak rates for the other three compounds.

Precision's compound performed essentially the same in both tests -- holding the brine at temperature but leaking at about 100 cc/day near room temperature.

L'Garde's compound performed similarly in both tests also -- perhaps not quite as good in Test 2. The leak rates between 237 hours and 305 hours are higher than experienced before. The refill operation at 305 hours perhaps repositioned the O-ring such that it didn't leak thereafter; this implies that it was locked during the pressure cycles at 231-233 or 236-237 hours.

BJ-Hughes' compound (which was a newly made batch for Test 2) performed similarly to Precision's in this second test, and better than it had in the first test. Its leak rate was zero at 260C and approximately 100 cc/day near room temperature.

TABLE 5-IV. Test 2 Leak Rates (28 MPa Differential Pressure*)

Time Period (Hours)	Temperature (°C)	Leak Rate (cc/day)			
		Precision	L'Garde	BJ-Hughes	Parker
0-135	260	0	0	0	0
137-139	71-32	120	0	70	0
141-142	260	0	0	0	100
142-143	↓	↓	↓	↓	2300 refill
143-168					0
168-183					30
183-184					880
184-190					30
190					Fail
190-231					
233-236	68-36	180	40	130	
237-303	260	0	10	0	
304-305	93-53	110	660 refill	60	
305-306	49-46	110	0	60	

*Pressure was 6.2 MPa (900 psi) front face and back face (no differential pressure) during cooling and heating.

The O-rings on the holder were examined (Figure 5-13). As in Test 1, all were permanently set such that the three sides facing metal were almost flat. Both Parker O-rings were being nibbled away; the residue appeared downstream (as seen in Figure 5-13).

As with Test 1 (Table 5-III), the diameters of the O-rings were measured while on the holders to determine the post-test, room temperature interference with the female part:

	Interference on The Radius (mm)	
	Test 1	Test 2
BJ-Hughes N-22-1	-0.17 to 0.00	+0.13 to +0.14
L'Garde Y267	+0.10 to +0.19	+0.06 to +0.08
Parker E962-85	+0.22 to +0.26	+0.01 to 0.08
Precision 42679	+0.02 to +0.10	+0.04 to +0.06

The BJ-Hughes batch clearly was an improvement over that used in Test 1. L'Garde's interference decreased which may account for the higher leak rate at the end of Test 2. Parker's interference decreased not only because it saw the right temperature in Test 2 but also because a significant volume nibbled away. Precision's interference and performance stayed the same.

It is concluded that:

a) he BJ Hughes, L'Garde and Precision O-rings were essentially identical; and

b) the Parker E962-85 O-rings were not as good as the others in 260C brine at a 28.1 MPa differential pressure.

c) The data are too limited to make firm conclusions. Given the circumstances, L'Garde recommends for any critical application that rubber parts procured from at least two of the transferees be evaluated. There is a distinct likelihood that for one given set of circumstances one company's part may out perform another's and for another set of circumstances the vice versa, though both are based on Y267 EPDM technology. Difference in processing, equipment, or modifications of the recipe exist. In addition, every company occasionally makes a bad batch.

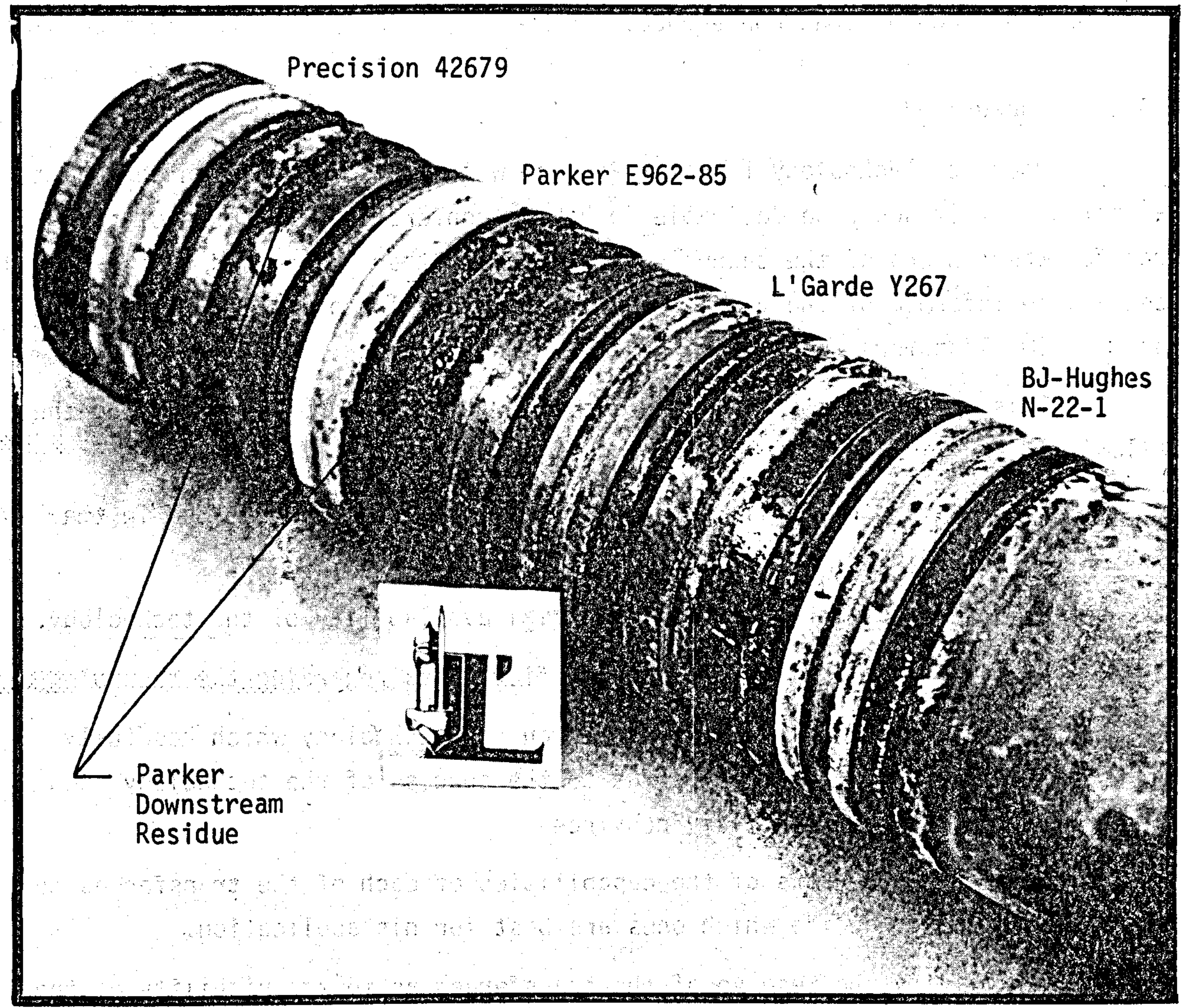


Figure 5-13. Post-Test Holder and O-Rings

5.5 DOE 1982 SAN DIEGO CONFERENCE

The elastomers session of the DOE conference entitled, "Applying Current Geothermal Corrosion/Materials Technology to Today's Projects" provided a perfect conclusion to the GEM Technology Transfer Program. This paragraph describes the elastomers portion of that conference.

5.5.1 Background

When the Technology Transfer Program was in its formulation phases it was noted that it would be desirable to have a conference at the end of the transfer where users of the technology along with other interested parties could meet and be informed of the milestone, i.e. the creation of commercial sources for the GEM Elastomers technology.

This meeting was anticipated to serve multiple purposes including the following:

- Creating a forum limited to those specifically interested in the technology.
- Inform the forum of the commercial availability of the technology.
- Inform the forum of the latest field data regarding the technology.
- Provide multiple field reports on the technology which hopefully all reinforce each other as to the success of the technology such that users are solidly convinced.
- Inform the users of the capabilities of each of the transferees to help him decide which ones are best for his application.
- Solidify the resolve of the transferees as to the viability of the market for the technology.

It was agreed that a conference would enhance the transfer and would be desirable. The final destiny, of if and when, was then a matter of the DOE budget.

5.5.2 Conference Particulars

A conference was ultimately organized for the DOE by Radian Corporation. The conference was held at the Sheraton Airport Hotel in San Diego, California on 6-8 October 1982. The conference included subjects which included corrosion, instrumentation, polymer concretes, metals, and elastomers. The subjects were divided into three sessions of which one was devoted to elastomers.

The session on elastomers included the following presentations:

- "Pitfalls of Elastomer Compatibility Testing", Gilbert J. Friese, L'Garde, Inc.
- High Temperature Y267 EPDM Elastomer Field and Laboratory Experiences, September 1982", Alan R. Hirasuna, Gilbert J. Friese, and Clifford Stephens, L'Garde, Inc.
- "Elastomers for Service in Deep Well Environment", L. R. Logsden and W. C. Long, BJ-Hughes Rubber.
- "Improved Energy Sealing Capability", Jerry L. Barsoumian, Parker Seal.
- "A Fabrication Report on L'Garde Y267 Geothermal Compound", Howard Gillette, Precision Rubber.
- "Summary of Geothermal Elastomeric Materials (GEM) Technology Transfer", Alan R. Hirasuna.

These papers are attached as Appendix G.

5.5.3 Conference Conclusion

In general the conference was successful and worthwhile. The elastomers session occurred on the last day and was the only subject for that day which implies that those who were in attendance were interested in elastomers. The attendance was fair, about ninety in all sessions, but the audience seemed to change somewhat during the progression of the conference. There were several comments that some people came primarily for the elastomers sessions.

Fortunately, the conference results coincided with the desired purposes or benefits stated in 5.5.1. There was a forum of people specifically interested in elastomers. The transferees were all prepared to fill commercial orders for the Y267 EPDM technology and in fact had each been doing so for several months. The three transferees and their customer experiences with the Y267 EPDM technology, without question, all substantiated that it is at the cutting edge. The latest information was exchanged, the users became fully aware of the technology performance and the manufacturers, and the manufacturers became more fully aware of the market.

6.0 INFORMAL TECHNOLOGY TRANSFER

The informal technology transfer is differentiated from the formal technology transfer of Section 5.0 in that it encompasses all other related activities except for the "formal" structured transfer process taking place with the three technology transferees. This section describes the other related activities.

6.1 IMPORTANCE OF INFORMAL TECHNOLOGY TRANSFER

The informal aspect of the technology transfer played an invaluable role in the GEM Technology Transfer Program. As discussed in 4.3.5, the GEM Technology could not be a self propelling transfer because the Technology Potential was limited by the low volume market and by the low Competitive Advantage which exists because the technology is in the public domain. Consequently, an informal effort was required to help effect transfer of the technology by stimulating the market through more widespread knowledge and additional confirmation of the viability of the technology. The informal activities included support of laboratory and field test trials and general dissemination of information regarding the technology. The resulting new successful test results and stimulation of inquiries from the community played an invaluable role in solidifying each transferee's committment to the effort. The informal Technology Transfer activities were critical to promoting and maintaining the strength and quality of the formal transfer.

6.2 TECHNICAL PRESENTATIONS

Presentation of papers at pertinent symposia provides a highly efficient vehicle for increasing the Level of Knowledge by reaching a large number of people at once. After any given paper L'Garde has received several inquires itself and there were unsolicited reports by the transferees of a flurry of inquiries. In addition to the primary mode of dissemination the conferences provide, there are important secondary disseminations of the information. The paper becomes part of the literature as part of the technical proceedings and can also be published in technical journals and summarized in news features. Probably the most important dissemination instrument is when the Level of Knowledge increases sufficiently that discussion of the technology becomes part of the cloak room talk.

During the course of the GEM Technology Transfer Program six presentations of the technology were made at various symposia. The reference information regarding each is listed in Table 6-I and the papers are included in Appendices H thru M. In addition, the papers presented at the concluding conference discussed in Paragraph 5.5 are included in Appendix G.

6.3 GENERAL INQUIRIES

Having a knowledgeable person able to respond to spontaneous general inquiries is extremely critical to successful technology transfer, i.e. someone under contract or an organization with a vested interest so they can justify supporting such an activity. If an individual is interested in the technology it is important that he be able to communicate with someone who can be responsive in a timely manner. If he cannot call or write someone for additional information, and/or samples, he will give up pursuit of the technology. Most times people pursuing technology are in the process of solving a problem. If information regarding a particular candidate technology is not readily available, he will seek other solutions so that he can make a conclusion within the time frame of his assignment. In addition, he is likely to need a sample of the product so that he can test it in his application.

Specific circumstances regarding the GEM-TT rendered contract support of the response to inquiries extremely essential. In that L'Garde would not ultimately be involved in selling the technology once it is transferred, it had no justification to self-support responses to general inquiries. This is the general circumstance in which any contractor would be who is employed to transfer the technology for the Government. If the technology is being transferred directly to a contractor for his ultimate use, he would then be justified in self-supporting responses to general inquiries.

The general inquiries came via all the expected modes -- telephone, mail, and first-person verbal. People have heard about the developed technology through the literature, word of mouth, and paper presentations; and made inquiries regarding the technology. The inquirer is generally given a summary of the development program, the technology transferees, and the results of the developments. Then the discussion generally turned to the inquirer's specific

TABLE 6-I. GEM-TT Papers and Publications

Date	Conference/Journal	Location	Title
Sept. 1979	GRC Annual Meeting	Reno, NV	Results of Geothermal Casing Packer Seal Elastomer Compound Development.
Dec. 1979	High-Temperature Electronics & Instrumentation Seminar	Houston, TX	Same as Sept. 1979.
May 1980	Rubber Division, American Chemical Society	Las Vegas, NV	High-Temperature Geothermal Elastomer Compound Development.
Jan. 1981	International Conference, Drilling & Completion Technology	Albuquerque, NM	High-Temperature Elastomer Compound Development.
Apr. 1981	NACE, Corrosion 81	Toronto, Ontario	High-Temperature Elastomer Compounds Developed for Unusually Severe Geothermal Environment.
Dec. 1981	NACE, Materials Performance		April 1981 Paper

specific interests and how the technology might apply. In addition to the discussions, various papers and in some cases samples were sent out. On the Technology Transfer Program 185 such requests were responded to in addition to the other responses under the original development program.

The inquirers included the following types of organizations:

- Contractors on other DOE projects
- U.S. Government Laboratories
- Geothermal Exploration Companies
- Fishing Tool Companies
- Instrumentation Companies
- Geothermal Developers
- BOP Companies
- Wellhead Companies
- Cementing Companies
- Pump Companies
- Drill Bit Companies
- Downhole Components Companies
- Molded Rubber Parts Companies
- Solar Energy Components Companies
- Rubber Constituent Manufacturers
- Geothermal Publications
- Downhole Instrumentation Manufacturers
- Power Companies
- A&E Contractors
- Cable Manufacturers
- Seal Manufacturers
- Logging Companies
- Research Institutes
- University Research Centers
- Pipe Coupling Manufacturers

Most of the inquirees were American but some were foreign which include the following countries.

- Indonesia
- Taiwan
- Italy
- Japan
- Trinidad
- India
- Sweden
- Canada
- Holland
- Switzerland

6.4 ASTM GEOTHERMAL COMMITTEE

In that several elements of the original development program[1] required testing and procedures that provide valuable input for geothermal ASTM standards, it was important to participate in the appropriate ASTM committee to pass-on the developed technology. Tests pertinent to geothermal elastomers and a synthetic geothermal test brine were developed in support of the geothermal elastomers development and proved to be quite pertinent and successful.

In the interest of transferring the technology to the ASTM committee, L'Garde participated in the following meetings:

St. Louis, MO	6 Dec 77	D-11.36
Boston, MA	27 June 78	D-11.36
Philadelphia, PA	17 Oct 79	E-45
Denver, CO	18 May 80	E-45.10.02
Reno, NV	18 Nov 80	E-45.10.02

The first meetings of the D-11.36 seals subcommittee were preparatory to the organization of the E-45 Committee which is devoted to geothermal energy. Once the E-45 committee was formed, L'Garde participated in the E-45.10.02 subcommittee which was addressing nonmetallic materials related subjects.

To facilitate the transfer of the GEM Program developments, L'Garde prepared a document which summarizes those aspects which are pertinent to geothermal elastomers and testing. The document is entitled "Specification For Standard Geothermal Fluid, Documentation of Some Previous Work", dated 19 May 1980. This document is included as Appendix N to this report. This document was presented at the Denver meeting on 18 May 1980.

7.0 HIGHER TEMPERATURE CAPABILITY ASSESSMENT

The GEM elastomer compounds were previously exposed to synthetic geothermal brine for 22 hours at 315C (600F) indicating some higher temperature capability. Though the performance is definitely approaching the capability ceiling of the polymers, it was deemed worthwhile to spend a limited effort to determine if further stabilization can be achieved, thus rendering higher temperatures practicable. Data indicate that EPDM is a worthwhile candidate. In addiiton a new Viton polymer recently released from the DuPont laboratory since the GEM development, VTX-5362 or Viton GF, has a better hot tear and tensile strength than do the prior peroxide cured Viton polymers. It also has good fluids resistance and replaced VTR-4590 which is the Viton polymer that L'Garde narrowed in on and concentrated on. In addition, a relatively new family of ethylene/acrylic polymers by DuPont, Vamac, warranted investigation. These three polymer systems were evaluated and are reported in this section.

7.1 APPROACH

Sensitivity studies were performed to determine elastomer potential at 315C. Compounds and processing which are offshoots of the GEM Y267 EPDM, and the GEM 255 Viton using the new Viton GF polymer were investigated as well as Vamac. These compounds were immersion tested at 315C in the GEM synthetic geothermal brine which has the following constituency:

H_2S	300 ppm
CO_2	1000
NaCl	25,000
H_2O	balance

The immersion ran for 22 hours and the ultimate tensile strength, ultimate elongation, Shore A hardness, weight change, and volume change were evaluated for the virgin and aged specimens.

7.2 EPDM STUDY

Evaluations by L'Garde and other laboratories in tests simulating "downhole" well conditions, as well as in the wells themselves, have shown exceptional life in both brine and in oil, in thick parts such as packers, and in small parts such as O-rings. Some of these tests were performed at 315C or higher.

This project was undertaken to investigate optimization of the Y267 compound for use at 315C. The conclusions of this limited study are as follows:

- All specimens, regardless of composition, showed very little or no degradation in brine at 315C for 22 hours. In fact, there was very little difference between the results of these tests and previous tests at 260C (500F).
- These tests confirm tests by other laboratories that Y267 will be useful in applications at 315C.
- Hypalon 20 reduced the amount of softening of Y267 in brine. The softening was probably not due to reversion since the tensile strength was not degraded. Increasing the amount of Hypalon 20 to 10 phr as in compound 505 might be of help to reduce O-ring nibbling and packer extrusion.
- Increasing or decreasing the thermal stabilizers Thermoguard S or Cyanox 2246 had no effect on ageing in brine.
- Post curing at 315C gave a softer compound (undesirable) although its recovery from deformation was faster, more resilient.
- 10 minute press cures at 177C (350F) were equivalent to 60 minutes in test sheets.

7.2.1 Hypalon 20

This is a chlorosulfonated polyethylene elastomer at 24% chlorine and 1.4% sulfur. Use of Hypalon 20 along with Thermoguard S as a stabilizer was based on some early work by the Air Force.

In these tests below, Hypalon 20 reduced the amount of softening of Y267 in brine. The softening was probably not due to reversion since the tensile strength was maintained.

Differences are not great, so the present level in Y267 of 5 phr should not be changed unless a special problem such as O-ring nibbling or packer extrusion occurs. The data are presented in Table 7-I.

TABLE 7-I. EPDM/Hypalon 20 Data

Compound No. Hypalon 20 level	504 0	Y267 5	505 10
Tensile Strength			
Original	1610	1620	1580 psi
Brine Aged	1420	1430	1330
Change	-11.8	-11.7	-15.8%
Elongation at Break			
Original	200	190	300%
Brine Aged	300	270	290
Change	+50	+42	+3%
Shore A Hardness			
Original	89	90	90
Brine Aged	85	89	90
Change	-4	-1	0 duro.
Weight Change	-.6	-.6	-.6%
Volume Change			
Width	0	+4.5	0%
Thickness	0	0	+1.6

7.2.2 Thermoguard S

This is antimony trioxide. Thermoguard S is the counterpart to Hypalon forming the stabilization system developed by the Air Force.

Neither decreasing nor increasing the amount of Thermoguard S in Y267 had any significant effect on the aged properties. This is not surprising since antimony trioxide is completely soluble in water and would be leached from the compound.

Since we have no data on hot air ageing, no change in the level in Y267 is recommended. The test data are presented in Table 7-II.

TABLE 7-II. EPDM/Thermoguard S Data

Compound No. Thermoguard S Level	506 0	Y267 5	507 10
Tensile Strength			
Original	1640	1620	1870 psi
Brine Aged	1250	1430	1630
Change	-23.8	-11.7	-12.8%
Elongation at Break			
Original	280	190	170%
Brine Aged	380	270	240
Change	+35.7	+42	+41.2%
Shore A Hardness			
Original	90	90	93
Brine Aged	86	89	91
Change	-4	-1	-2 duro
Weight Change	-1.5	-.6	-1.0
Volume Change			
Width	-16.7	+4.5	+1.7
Thickness	+2.0	0	+1.3

7.2.3 Cyanox 2246

This is 2,2 methylene bis (4-methyl-6-tert. butyl phenol). It has found use as a strong antioxidant in several types of elastomers. It is only slightly soluble in water or oil. It is also the subject of some early Air Force work.

No improvement can be expected by either increasing or decreasing the amount of Cyanox 2246 in Y267. It is quite likely it has no effect on ageing in brine. Until there is data on air ageing in Y267 at high temperatures, the amount of Cyanox 2246 in Y267 should not be changed. The data are presented in Table 7-III.

TABLE 7-III. EPDM/Cyanox 2246 Data

Compound No. Cyanox 2246 Level	508 0	Y267 .5	509 1.5	510 3.0
Tensile Strength				
Original	2010	1620	1510	1490 psi
Brine Aged	1600	1430	1280	1180
Change	-20.0	-11.7	-15.1	-14.9%
Elongation at Break				
Original	190	190	210	240%
Brine Aged	250	270	300	330
Change	+18.8	+42	+20.9	+23.9%
Shore A Hardness				
Original	92	90	93	95
Brine Aged	88	89	92	93
Change	-4	-1	-1	-2 duro
Weight Change	-.4	-.6	-.5	-.9%
Volume Change				
Width	-1.0	+4.5	-6.3	-12.8%
Thickness	+.6	0	+1.2	-1.3

7.2.4 Press Cure Times

The original GEM development used press cures of 60 minutes at 177C for test sheets and O-rings, and 90 minutes at 177C for packers. Such long press cure times would be costly in production. The purpose of the press cure is to shape the part and develop enough strength to prevent internal sponginess when the pressure is released and the gases come out of solution.

For this work press cures of 60, 30, 10 and 5 minutes at 177C were investigated for test sheets and O-rings. Freshly cut surfaces were washed with alcohol and examined at 3X and 18X magnification with grazing light at about 15°. None of the specimens showed any sponginess.

Physical properties before and after ageing in brine at 315C (600F) for 22 hours showed no significant differences between the 10 minute and the 60 minute press cures. Of course, for thick parts such as packers, a longer press cure time than 10 minutes may be required.

7.2.5 Oven Post Cure Conditions

Four variations were tried. All were press cured 10 minutes at 177C. The oven was heated to 204C (400F) prior to loading the nitrogen purged cell containing the test specimens.

Cure 1:	Raise temperature to 315C (600F) as fast as oven will go. Then 6 hours at 315C.
Cure 2:	1 hour at each of 204 and 260C, and then 5 hours at 315C.
Cure 3:	1 hour at each of 204C (400F), 232C (450F), 260C (500F), 288C (550F), then 5 hours at 315C.
Cure 4:	1 hour at each of 204, 232, 260C, then 5 hours at 288C.
Control:	Same as Cure 4 except press cured 60 minutes at 177C.

Hand examination showed that Cure 1 specimens recovered faster when bent or stretched and felt more resilient.

Physical properties and brine ageing were run on Cures 1, 4 and Control. See Table 7-IV.

Even though Cure 1 seemed more resilient than any of the others, comparison of physical properties with Cure 4 or the Control showed Cure 1 to be significantly softer with lower tensile and higher elongation. Changes due to brine ageing were contradictory. It could be that 315C is too high a temperature for curing causing thermal reversion. In any case the best oven post cure was the Control.

TABLE 7-IV. EPDM/Post Cure Data

	Cure 1	Cure 4	Control
Press Cure	10	10	60 min.
OPC Temperature	600	550	550F
Tensile Strength			
Original	1065	1620	1620 psi
Brine Aged	740	1360	1430
Change	-30.5	-16.0	-11.7%
Elongation at Break			
Original	430	220	190%
Brine Aged	440	290	270
Change	+2	+32	+42%
Shore A Hardness			
Original	86	90	90
Brine Aged	81	86	89
Change	-5	-4	-1 duro
Weight Change	-.5	-.1	-.6%
Volume Change			
Width	+2.7	0	+4.5%
Thickness	-3.7	+1.3	0

7.3 VITON STUDY

Extensive work was done by L'Garde, Inc. to evaluate the use of peroxide cured Viton compounds to make geothermal well packers and O-ring seals for use in a synthetic brine for 22 hours at 260C (500F). The best Viton compound was L'Garde's compound 255 based on DuPont's VT-R-4590. Viton 4590 was obsoleted by DuPont and replaced with VT-X-5362 which has since been commercialized as Viton GF.

The present work was undertaken to demonstrate whether VT-X-5362 would be an acceptable replacement for VT-R-4590. The conclusions of this limited study are as follows:

- VT-X-5362 with increased curatives produces a compound equivalent to VT-R-4590.
- Curative level should be 3 phr for Luperco 101XL and for Diak 7.
- For O-rings and test sheets and other thin parts, use a press cure of 10 minutes at 177C (350F) and an oven post-cure in nitrogen of 22 hours at 260C (500F).

- For packers and other thick parts, use a press cure of 15 minutes at 177C, and an oven post-cure of 1 hour at 177C, 204C, 232C, and 17.5 hours at 260C.
- Use Luperco 101XL as the peroxide at 3 phr in place of Luperco 130XL.
- VT-X-5362 does not appear to enable 315C service, all compounds were blistered and brittle after 22 hours at 315C in synthetic brine.

7.3.1 Test Formulas

Three formulas were compared -- the 255 which is the old GEM VT-R-4590 compound which was used as the control, and two new formulas based on VT-X-5362. The formulas are presented in Table 7-V.

TABLE 7-V. Test Formulas

L'Garde Number	255	300	501
Viton 4590	100	-	-
Viton 5362	-	100	100
Austin Black	10	10	10
FEF Black	25	25	25
Maglite K	3	3	-
Litharge	-	-	3
Diak 7	1.5	2.25	3
Luperco 130XL	1.5	2.25	-
Luperco 101XL	-	-	3
Press Cure	15'/177C		10'/177C
Oven Post Cure in nitrogen	1 hr/177C 1 hr/204C 1 hr/232C 17.5 hrs/260C	Same as 255	22 hrs/232C

7.3.2 Test Results

Immersion tests in synthetic geothermal brine were run at 260C (500F) and 315C (600F) for 22 hours for the three peroxide cured Biton compounds. All specimens aged at 315C were blistered and brittle, therefore no data are reported. Compounds 255, 300 and 501 were run in parallel. At 260C compounds 501 and 255 were equivalent within experimental error. Test sheets all molded without internal ruptures indicating that a one-step oven post-cure can be used for thin parts such as O-ring seals.

In this first run the properties of compound 300 were so far from expectations that it was obvious some error had been made. These results are reported, but discounted. The first run data are presented in Table 7-VI.

A second run was made of compounds 255 and 300 in parallel. This time the two compounds were equivalent within experimental error. However, compound 300 was 6 Shore A points softer than 255 with consequent higher elongation at break indicating (but not proving) that the peroxide and Diak 7 should probably be increased somewhat. These data are presented in Table 7-VII.

TABLE 7-VI. Test Data - First Run

L'Garde Number	255 Typical	255 this run	300	501
Tensile Strength				
Original	1320-2200	2400	2390	2780
Chem Aged	950-1590	1800	1650	2040
Retained %		79	69	73
Elongation				
Original	135-225	240	95	210
Chem Aged	135-225	230	340	280
Retained %		96	362	134
Hardness at 260C (500F)				
Original	74-84	70	93	78
Chem Aged		75	67	67
Change, duro		+5	-26	-11
Weight Change				
Chem Aged	+2.4%	+.05	+.35	-1.8
Dimension Change Chem Aged				
Width	+1.9	+.7	+.15	0
Thickness	+2.8	+7.9	+4.3	+.7

TABLE 7-VII. Test Data - Second Run

L'Garde Number	255	300
Tensile Strength		
Original	2450	2680
Chem Aged	1615	1700
Retained %	66	64
Elongation		
Original	195	260
Chem Aged	170	285
Retained %	87	110
Hardness at 260C (500F)		
Original	81	75
Chem Aged	84	71
Retained, duro	+3	-4
Dimension Change Chem Aged		
Width	+4.2%	+2.2
Thickness	+.5	+2.5

7.4 VAMAC STUDY

Vamac is an ethylene-acrylic elastomer by DuPont introduced in 1976. It is a copolymer of ethylene and methyl acrylate with a small amount of carboxyl group (-COOH) to provide curing sites. This polymer belongs to a large family of acrylate co- and terpolymers produced for a long time by American Cyanimide, Goodrich Chemical, Uniroyal Chemical and Thiokol.

In general, these all have a saturated backbone which gives them heat, ozone, and oxidation resistance, with methyl, ethyl or butyl side groups to give better low temperature flexibility in that order. Some have (-Cl) or (-CN) groups to give better oil resistance and to provide curing sites. Most are very difficult to mold because of sticking or because they become fluid at molding temperatures.

As a class, they are satisfactory for long-term use at 177C (350F), especially in petroleum lubricants. The maximum usable temperature for short-term use is 200C (400F). DuPont's bulletin EA 000.2, page 5 on Vamac shows life in hot air as follows:

4 weeks at 177C (350F)
10 days at 191C (375F)
5 days at 204C (400F)

In bulletin EA 110.1 (R-1), page 2, they note that Vamac compounds slowly decompose in air at 315C (600F). The decomposition products are very toxic.

However, based on previous work it was expected that Vamac would not be hydrolytically stable because of the carboxyl group. In spite of the questionable potential for Vamac at 315C, very limited investigation of one compound was pursued in case the preconceptions were invalid.

The conclusion of this limited study is that Vamac is not a viable candidate for synthetic geothermal brine at 260C (500F) or 315C.

7.4.1 Test Formula

Due to the improbability of Vamac, one formulation was considered. MDA is 4,4-methylene dianiline sold by DuPont as LD2729 and DPG is diphenyl guanidine which is widely available. The formulation is shown in Table 7-VIII.

TABLE 7-VIII. Test Formula

L'Garde Number	503
Vamac B-124	124 phr
SAF Black	80
MDA	1.25
DPG	4
Press Cure	20'/177C (350F)
Oven Post Cure in N_2	4 hr/177C

7.4.2 Test Results

Immersion tests in synthetic geothermal brine were run at 260C (500F) and 315C (600F). Specimens at 260C were very brittle and blistered, however, some measurements were possible. The specimens at 315C were destroyed, therefore, no data are reported.

The original properties are quite respectable, but even at 260C the degradation is catastrophic with the ultimate elongation going to 38%. The resulting data are shown in Table 7-IX.

TABLE 7-IX. Vamac Data

L'Garde Number	503
Tensile Strength, psi	
Original	2318
Chem Aged (260C)	842
Retained %	36
Elongation, %	
Original	291
Chem Aged (260C)	38
Retained %	13
Hardness @ 260C	
Original	80
Chem Aged (260C)	75
Retained %	94
Weight Change	
Chem Aged	+4.1%
Dimension Change Chem Aged	
Width	-1.5%
Thickness	+12.8%

8.0 REFERENCES

1. A. R. Hirasuna, et. al., "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976-30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U. S. Department of Energy-Division of Geothermal Energy, July 1979.

2. G. J. Friese, "Short and Long-Term Tests of Elastomers With Hot Hostile Fluids," L'Garde, Inc., Newport Beach, CA 92663, Brookhaven National Laboratory/U. S. Department of Energy-Division of Geothermal Energy, to be published.

APPENDICES

APPENDIX A

L'GARDE, INC.

1555 PLACENTIA AVENUE
NEWPORT BEACH, CALIFORNIA 92663
TELEPHONE (714) 546-4671 & (714) 645-4880

L-79-AH-408
5 December 1979

TYPICAL LETTER

Subject: Invitation to Apply For Participation in a High-Temperature Elastomer Technology Transfer Program

We have developed significantly improved high-temperature elastomers for use in geothermal well environments under contract to the U.S. Department of Energy. The Brookhaven National Laboratory is now funding us to transfer this technology to a limited number of companies for their particular application(s).

The enclosed paper describes the improved elastomers. Also enclosed is a description of the technology transfer effort. If you are interested in obtaining this elastomer technology free of charge, please complete and return the enclosed application.

Sincerely,

Alan R. Hirasuna
Program Manager

ARH:pw
5513

Enclosures: Results of Geothermal Casing Packer Seal Elastomer Compound Development
GEM Technology Transfer Program
Application

ENGINEERING CONSULTING • RESEARCH AND DEVELOPMENT • LIGHT MANUFACTURING

Geothermal Resources Council, TRANSACTIONS, Vol. 3 September 1979

RESULTS OF GEOTHERMAL CASING PACKER SEAL ELASTOMER COMPOUND DEVELOPMENT

Alan R. Hirasuna
Richard W. Sedwick
Clifford A. Stephens

L'Garde, Inc.
Newport Beach, CA 92663

ABSTRACT

Reliable casing packer seal elastomers for the unusually severe geothermal environment at 260°C (500°F) did not exist in 1976. A contract to develop elastomer compounds for this specific application was awarded to L'Garde, Inc. by the Department of Energy. Elastomer compounds were successfully developed which as packer seals maintain seal at 260°C for more than 24 hours in synthetic geothermal brine (aqueous solution of H_2S, CO_2, and NaCl).

The final results of the elastomer compound development are presented. The successful compounds are based on the following polymer systems: EPDM (Nordel^R), fluoroelastomer (Viton^R), EPDM/fluoroelastomer blend, and propylene-TFE (AFLAS^R). Evaluation was performed on full-scale packer seals as well as O-rings by L'Garde. L'Garde ran comparison tests against commercially available elastomers including Kalrez^R. L'Garde's test results were also corroborated independently by three other companies.

INTRODUCTION

In late 1976 L'Garde, Inc. was awarded a contract by the Department of Energy (DOE), DE-AC03-77ET28309 (formerly EG-77-C-03-1308), the Geothermal Elastomeric Materials (GEM) Program. By that time the DOE concluded that there was a general need for improved elastomers for various seal applications which were required to operate in the unusually severe geothermal environment. Logging equipment could not be run for fear of losing seals and damaging the instrumentation. Packers could not be run without first cooling the well. Rotating well head seals and BOP seals required frequent replacement. There was a general need for higher temperature, more hydrochemically resistant elastomers and for that matter the need is still not satisfied in practice today in the geothermal field.

L'Garde performed an initial task to identify what elastomer application when upgraded to geothermal requirements would be of greatest benefit to the geothermal community. The formation packer was identified for two functions, hydraulic stimulation and interval testing in support of overall reservoir characterization. However, though high temperature geothermal formation packers are needed, development of elastomers for this application was judged too ambitious a goal for the first effort. Therefore, it was recommended and approved to first develop improved elastomers for casing packer seal elements. In addition, rubber development for the casing packer element "kills two birds with one stone", as those elastomers which work successfully for the packer seal will most probably work well for static O-ring seals. The O-ring application is less severe because both surfaces are machined, the clearances are small and the required deformation of the O-ring is small compared to a casing packer seal element.

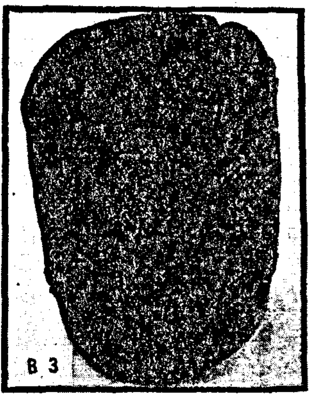
This paper presents the results of the elastomer compound development for high temperature geothermal casing packer seals.

PACKER SEAL SIMULATION EVALUATION AND DEVELOPMENT

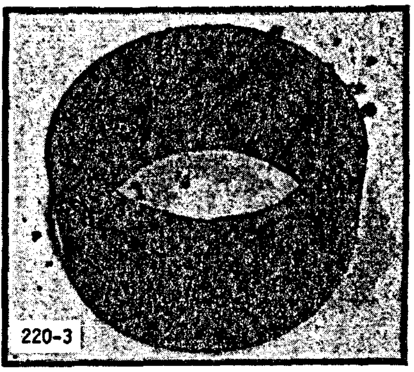
The primary objective of the Program was successfully met. Though meeting the fundamental objective, to develop a casing packer seal elastomer compound which will operate in a 260°C (500°F) geothermal environment, was highly challenging and carried with it a definite probability of failure, L'Garde was fortunate to be able to conclude the program with very positive results. Packer seals which perform well in the oil field up to temperatures of about 150°C (302°F) run into reliability problems at higher temperatures especially when they get as high as 260°C. Though the stresses induced into the elastomeric element are substantially the same as the lower temperatures, the loss in elastomer strength at 260°C is substantial and it cannot sustain the stress levels. Other common problems include hardening of the seal at higher temperatures and associated failure. The geothermal elastomeric packer seal problem at 260°C is significant and even the less demanding static O-ring seal is still a problem. Figure 1 rather lucidly illustrates the starting point of the geothermal packer seal development. These post mortem test specimens were run at 260°C in synthetic brine. At these unusually severe conditions, the seals did one of three things; extruded severely (B3), could not withstand the pack-off and cracked without sealing (220-3), or extruded and broke catastrophically (D3 and E3).

FIGURE 1 -- SIM TEST POST MORTEM SPECIMENS TYPICAL EARLY RESULTS

Company B EPDM -- This and L'Garde EPDM remained elastomeric and survived full 24 hours. Only difference L'Garde EPDM extruded 2.5 in. vs company B's 4.0 in.



L'Garde Peroxide-Cured Fluoroelastomer -- Failed because of inability to elongate sufficiently during seating.



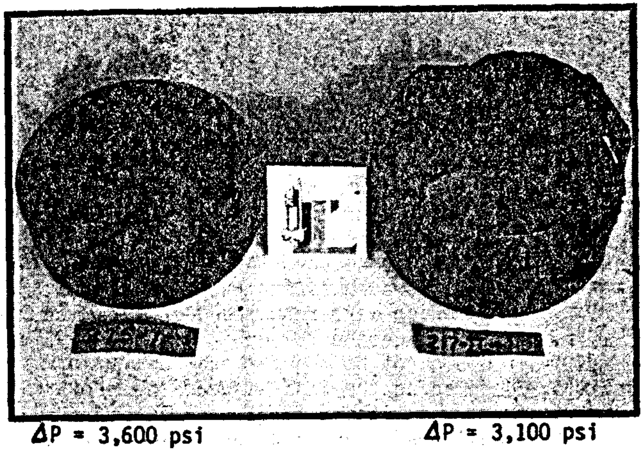
Company D Epichlorohydrin -- Lost all structural strength and became soft and crumbly before seating.

Company E Non-Peroxide-Cured Fluoroelastomer -- Failed by breaking in the high stress region. Survived 3.75H with slow leak, then failed.

For about the first 2/3 of the contract, it was doubtful that elastomer improvements alone would provide reasonable performance at the test conditions at 260°C.

Upon conclusion of the development, compounds based on four (one was required) separate elastomer systems molded into full-scale packer seals successfully sealed in laboratory simulated packer tests for the prescribed duty cycle - temperature of 260°C, in synthetic geothermal brine, differential pressure of 3100 to 4100 psi, and for 24 hours. The four systems were based on the following polymers: Ethylene Propylene Diene Terpolymer (EPDM, Nordel^{R}), Fluoroelastomer (FKM, Viton^{R}), EPDM/FKM Blend (Nordel^{R}/Viton^{R}), and Propylene-TFE (AFLAS^{R}). All four elastomer systems successfully met the Program objectives and show definite improvement over elastomer compounds which are commercially available. Measurable improvement of the hydrothermochemical stability was achieved in the synthetic geothermal brine which includes an aqueous solution of H_2S, CO_2, and NaCl at the following levels: 300 ppm, 1,000 ppm, and 25,000 ppm respectively. For these conditions, the Nordel^{R} compound is the most hydrothermochemically stable showing no effect after 22 hours. These results represent an effort which included formulating and testing 129 compounds and running 96 seal Simulation Tests.

FIGURE 2 -- POST MORTEM PACKER SEALS AFTER 22 HRS. IN SYNTHETIC GEOTHERMAL BRINE CONTRASTING EARLIER AND CURRENT BEST FORMULATIONS



ΔP = 3,600 psi ΔP = 3,100 psi

Figure 2 shows post mortem test specimen of one of the better, earlier Nordel^{R} EPDM compounds, seal 217-II-1. This element sealed successfully at 260°C, but did suffer a significant level of extrusion. In contrast, one of the latest compounds is shown, post mortem seal 275-1 which is a Nordel^{R}/Viton^{R} Blend. Only a minor amount of permanent deformation resulted; the seal had an excellent appearance when removed from the test. This element sealed successfully under the same conditions as 217-II-1 except even a higher differential pressure, 3600 vs. 3100 psi.

Compound 267 packer seals were molded for tests by a packer company. Molded seals for 7-inch, 23 lb/ft casing were provided and tested at 302°C (575°F) and 3000 psi differential pressure in their laboratory packer simulation tester. The elements sealed successfully for five days when the test was stopped and their comment was, "after the test they bounced off the floor just like before the test." They are currently planning to test these elements downhole.

CORROBORATING O-RING SIMULATION TESTS

A good quantitative demonstration of the accomplishments is seen through examination of the O-ring SIM Test data and comparing the developed compounds against commercially available compounds. A test was fabricated to enable static simulation testing of O-rings. The O-rings were tested at 260°C in the synthetic geothermal brine starting at 4100 psi differential pressure. The test was set for 22 hours and if a compound failed before 22 hours, the differential pressure was decreased by 1000 psi and the test rerun. The reduction in differential pressure progressed to 2100 psi where the testing stopped if the material failed.

Because this is an unusually severe environment, the GEM compounds have a distinct advantage in that they were developed specifically for this condition. Figure 3 summarizes the capability for each of the compounds. The GEM Viton^R and Nordel^R compounds performed better than their commercial counterparts. The GEM 255 Viton^R held at 3100 psi while the commercial counterparts both broke at 2100 psi. The GEM 267 Nordel^R looked excellent after the 4100 psi run and is capable of higher differential pressures while its counterparts showed incipient failure. There were no commercially comparable counterparts to the Nordel^R/Viton^R Blend or the AFLAS^R, both ran the full 22 hours at the full differential pressure. The Blend looked as good as the 267 Nordel^R and is also capable of higher differential pressures and the AFLAS^R suffered minor extrusion. There was no counterpart to the DuPont Kalrez^R, however, it is of keen interest because it is one of the more chemically stable polymers. It is very expensive but its expense is justifiable in some situations because of the longer life it renders. For the synthetic geothermal brine environment the 3074 Kalrez^R did not fare as well as the four developed compounds in the O-ring test, and earlier coupon ageing tests were made on 3073 Kalrez^R which indicate that it is not as hydrothermochemically stable as the developed compounds. Specific compounding for the geothermal brine environment may result in similar improvements as was herein accomplished.

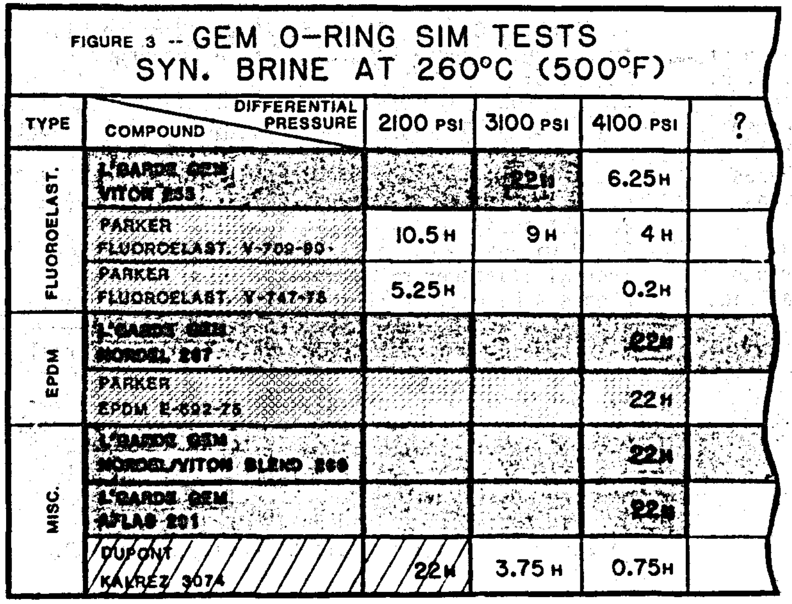


FIGURE 3 -- GEM O-RING SIM TESTS SYN. BRINE AT 260°C (500°F)

TYPE	COMPOUND \ DIFFERENTIAL PRESSURE	2100 PSI	3100 PSI	4100 PSI	?
FLUOROELAST.	L'GARDE GEM VITON 255		22H	6.25H	
	PARKER FLUOROELAST. V-709-90	10.5H	9H	4H	
	PARKER FLUOROELAST. V-747-75	5.25H		0.2H	
EPDM	L'GARDE GEM NORDEL 267			22H	
	PARKER EPDM E-692-75			22H	
MISC.	L'GARDE GEM NORDEL/VITON BLEND 266			22H	
	L'GARDE GEM AFLAS 291			22H	
	DUPONT KALREZ 3074	22H	3.75H	0.75H	

Figure 4 is a composite photograph of the more interesting seals; L'Garde GEM Nordel^R 267, Parker EPDM E-692-75, L'Garde GEM Nordel^R/Viton^R Blend 266, and the DuPont Kalrez^R 3074. All seals ran successfully for 22 hours at 4100 psi differential pressure except the Kalrez^R 3074 which ran at 2100 psi differential pressure. Note that the Nordel^R 267 and the Nordel^R/Viton^R Blend 266 look excellent, although some permanent squaring of the O-ring did occur. Both the Parker EPDM E-692-75 and the DuPont Kalrez^R 3074 are seen to have damage.

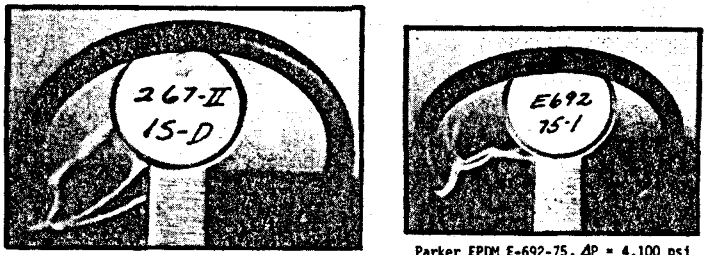
HYDROTHERMOCHEMICAL STABILITY TESTING

Another good quantitative demonstration of the accomplishments is seen through a comparison of compound characteristics for early compounds against commensurate ones at the completion of the program. Table I illustrates the improvements as indicated by the percent of each of the four critical characteristics retained after ageing in brine for 22 hours. Table II lists the virgin and chemically aged values which are represented by the percentage numbers.

Note that there is significant improvement reflected by the latest compounds. The 267 Nordel^R is virtually unchanged by the 22 hour ageing in the synthetic brine with respect to all four characterization parameters. Vast improvement was finally accomplished with the 255 Viton^R. There is room for improvement on the Extrusion Resistance especially, as is reflected by the Packer Seal Simulation Tests, however, the extrusion resistance is adequate to meet the requirements. The 266 Nordel^R/Viton^R is very stable with some minor degradation of the ultimate elongation. The 291 AFLAS^R shows some minor hydrothermochemical instability in the ultimate elongation. In general, all of the latest formulations showed very good stability considering the unusual severity of environment.

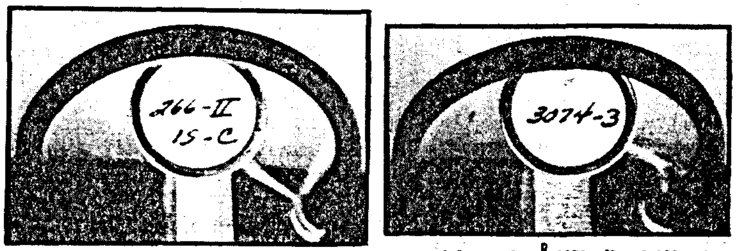
The above data are all comprehensively reported in Reference 2.

FIGURE 4 -- POST MORTEM O-RINGS SIM TEST SEALS AFTER 22 HRS.
IN SYNTHETIC GEOTHERMAL BRINE @ 500°F



L'Garde GEM Nordel^R 267 EPDM, ΔP = 4,100 psi

Parker EPDM E-692-75, ΔP = 4,100 psi



L'Garde GEM Nordel^R/Viton^R Blend, ΔP = 4,100 psi

DuPont Kalrez^R 3074, ΔP = 2,100 psi

TABLE I -- PROGRESS OF HYDROTHERMOCHEMICAL STABILITY
OF GEOTHERMAL ELASTOMERS

Elastomer	Ult. Tensile @ RT		Ult. Elong. @ RT		Shore A Hard @ 260°C		Extrusion Resist. @ 260°C	
	Early	Present	Early	Present	Early	Present	Early	Present
EPDM								
Early-E263	75%		--		•85%		57%	
Present-267		97%		105%		100%		103%
Viton								
Early-115	14%		46%		73%		32%	
Present-255		72%		101%		100%		55%
EPDM/Viton								
Early-235	80%		63%		110%		128%	
Present-266		96%		84%		101%		96%
AFLAS								
Early-268	77%		112%		90%		84%	
Present-291		93%		118%		100%		87%

TABLE II -- GEOTHERMAL ELASTOMERS PROGRESS

Elastomer	Ult. Tensile @ RT. psi		Ult. Elong. @ RT %		Shore A Hard @ 260°C		Extrusion Resist @ 260°C, psia	
	V	CA	V	CA	V	CA	V	CA
EPDM								
Early-E263	1363	1020	>840	>840	61	52	9.6	5.5
Present-267	1610	1554	141	148	92	92	17.1	17.6
Viton								
Early-115	1699	244	162	74	80	58	11.2	3.6
Present-255	2195	1591	224	226	79	79	11.4	6.3
EPDM/Viton								
Early-235	1882	1506	681	431	67	74	8.3	10.6
Present-266	1596	1531	193	163	88	89	18	17.3
AFLAS								
Early-268	1673	1289	394	442	69	63	6.7	5.6
Present-291	2335	2162	134	158	95	95	17.4	15.2

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research and development and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory was especially helpful.

REFERENCES

1. Arnold, C. Jr., 19 April 1979, "Memo to Distribution," Sandia Laboratories, Albuquerque, NM 87115.
2. Hirasuna, A.R., et al., July 1979, "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976 - 30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy (To Be Published).
3. Hirasuna, A.R., et al., December 1977, "Geothermal Elastomeric Materials - Twelve-Months Progress Report, October 1, 1976 - September 30, 1977," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy.
4. Hirasuna, A.R., et al., 25-27 July 1978, "Important Conclusions Based on Results of Geothermal Casing Packer Seal Elastomers Testing," Transactions of Geothermal Resources Council Annual Meeting, Hilo, HI.
5. Winzenried, R., 15 June 1979, "Letter to Hirasuna," Terra Tek, Salt Lake City, UT 84108.

GEM TECHNOLOGY TRANSFER PROGRAM

High-temperature elastomer compounds were developed by L'Garde for the Department of Energy on the Geothermal Elastomeric Materials (GEM) Program (Contract DE-AC03-77ET28309). The government, through Brookhaven National Laboratory, is interested in putting these advancements into use, and is funding L'Garde to transfer the technology to a limited number of companies.

BACKGROUND

The primary GEM Program objective, to develop a geothermal packer elastomer to withstand 260°C (500°F) geothermal brine with 300 ppm H_2S, 1,000 ppm CO_2, 25,000 ppm NaCl in aqueous solution for 24 hours, was exceeded. Four elastomeric polymer systems were successfully developed; this provides good flexibility for other applications and environments. While the compounds were developed for the casing packer seal application, they were also demonstrated for use in static O-ring and other elastomer applications in similarly severe environments. Also, drill-bit seal tests show that the basic compounds will be adaptable to high-temperature dynamic seal applications.

A comprehensive description of the work is reported in Geothermal Elastomeric Materials (GEM) Program, L'Garde, Inc., DOE Publication No. SAN/1308-2, July 1979. Copies should soon be available from the National Technical Information Service, U. S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. However, there is risk that the report information is insufficient and/or would be misused to the point of failure; the developed elastomer technology is complex and involves a significant degree of art. Therefore, L'Garde has been funded to directly transfer the technology to interested companies.

BENEFIT TO YOU

Participation in this program assures that you will achieve inhouse capability to produce these advanced elastomers earlier and cheaper. You will receive L'Garde's expertise and labor free of charge. There will be no government or commercial contracts, restrictions, et cetera involved.

COMPANY SELECTION

The goal of the GEM Technology Transfer Program is to get these advanced materials into wide use. L'Garde is mailing an invitation to participate to anyone that can be identified as having a potential interest. If you don't have such an interest but know someone who might, please share this information with him.

Company selection criteria has not yet been established. It is anticipated that both large and small companies will be included, and that each company selected will have a different primary application. It may be necessary to resort to random selection.

Current funding limits direct participation to 4 to 6 companies. To further broaden the use of the technology, the DOE hopes to sponsor a symposium/workshop at the end of technology transfer effort to disseminate results. All applicants would be invited.

TECHNOLOGY TRANSFER

The effort will occur during the first half of 1980. L'Garde will present the latest GEM results to each selected company. This presentation will be followed by an interchange of requirements and applicable technologies for the company's particular application. Recommendations will be made as to the best initial compound.

L'Garde will prepare a molded part(s), using a supplied mold, and provide them to the company for testing. Assuming the part provides satisfactory results, L'Garde will provide detailed compounding and curing process information to the company. Alternately, L'Garde may provide uncured rubber and work on-location with the company or its supplier. If the company experiences problems producing the part, L'Garde will provide assistance for the duration of the contract.

SYMPOSIUM/WORKSHOP

Should a symposium/workshop be held, the selected companies will be invited to document their experience and results, and will also be invited to present such papers. Emphasis will be on the results and the improvement in performance. This provides product and company publicity.

COST AND SECURITY

L'Garde is being fully funded by the Brookhaven National Laboratory. The selected company will pay for their own employees' time, a mold if otherwise unavailable, and travel costs to the symposium/workshop if required.

L'Garde will keep confidential any proprietary or sensitive information supplied (and marked as such) by a company, and will not use such information for any purpose. L'Garde, Inc. is a government-funded research and development firm.

APPLICATION

Geothermal Elastomeric Materials Technology Transfer

Company: ______________________________

Address: ______________________________

____

Person to Contact: ______________________________ Phone: ____________

Mark any information below that must be kept confidential. Use additional sheets if desired.

1. Approximate Employment. Total ________

   In product line requiring special elastomers ____________________

2. Your current products/applications that could potentially use GEM elastomers:

Product Type/Description	1979 Market Number or $

3. Assuming the GEM elastomers have the properties given in the summary plus other necessary properties that your current elastomer has, roughly project the performance gains that GEM elastomers might provide your product/application (Example: Service life from ________ to ________; additional applications for ___________; et cetera). Limit to two products.

   ____

   ____

   ____

   ____

4. For the Item 3 products/applications, list other necessary elastomer properties or environments (that you are unsure the GEM elastomers have).

   ____

   ____

   ____

   ____

5. Which product or application would you propose modifying through this technology transfer program? ____________________

6. How would you test the L'Garde-furnished product; i.e., special laboratory equipment? in the field? Please estimate the time required.

____

____

7. What additional information or conditions would you need before you would participate in this transfer program?

____

____

____

8. If you decide to participate and are selected, would you (at your own cost):
   a) Loan L'Garde a mold? ____________________
   b) Test the L'Garde-furnished item? ____________________
   c) Write a paper at the end of the technology transfer? __________
   d) Attend and support a symposium? ____________________

9. Would you object to L'Garde publishing the technology transfer effort and results? __________. If yes, why? ____________________

____

____

10. What changes to the suggested transfer approach would you suggest, and why?

____

____

____

____

11. What new products/applications would you consider developing with the GEM elastomers?

____

____

____

____

12. Please provide pertinent brochures, catalogues, et cetera to help understand your products and their use.

APPLICATION		REVISION			
NEXT ASSY	USED ON	LTR	DESCRIPTION	DATE	APPROVED
		A	Corrections and changes to Table I and II.	1 Jun 79	

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ARE: FRACTIONS ± DECIMALS .XX ± .XXX ± ANGLES ±	CONTRACT NO.	
	APPROVALS	DATE
	DRAWN R. Wallstrom Rw	8 Nov 78
MATERIAL	CHECKED	
FINISH		
DO NOT SCALE DRAWING		

L'GARDE, INC.

1555 PLACENTIA AVENUE
NEWPORT BEACH
CALIFORNIA 92660

Procedure for Preparation of Fluoroelastomer Compound, GEM 255

SIZE	CODE IDENT NO.	DRAWING NO.
A	1F668	10590

SCALE

SHEET 1 OF 9

1. SCOPE

This procedure establishes the formulation, mixing methodology and steps for preparing the GEM 255 rubber compound intended for use as Geothermal Packer seal elements.

2. APPLICABLE DOCUMENTS

The following documents form a part of this specification of the extent specified herein. Later issues of these documents may be used at the option of the supplier providing no degradation of the product ensues.

Federal

Q-A-51	Acetone, Technical

American Society for Testing and Materials

ASTM D2516	Nomenclature for Rubber-Grade Carbon Blacks
ASTM D15	Compound and Sample Preparation for Physical Testing of Rubber Products
ASTM D412	Tension Testing of Vulcanized Rubber
ASTM D2240	Indentation Hardness of Rubber and Plastics By Means of a Durometer, Test for

3. REQUIREMENTS

3.1 Material. All materials for the preparation of these rubber compounds shall be formulated and processed to meet the requirements of this procedure. All materials shall be homogeneous, clean and suitable for the purpose intended.

3.2 Processing. The compounds shall be prepared according to this procedure.

3.3 Mechanical Properties. The compounded rubber shall have properties according to this procedure.

3.4 Workmanship. Each batch of rubber shall be prepared, handled and stored to minimize contamination. Contamination is herein defined as the presence of any material in the compound which is visible to the unaided eye. Contaminated rubber may be removed from the compound if it does not exceed 1% by weight of the compound at the time of removal. If the contaminated rubber exceeds 1% by weight, the compound must be discarded.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance. The supplier of materials under this procedure shall certify that each batch conforms to all requirements of the procedure. Certification shall include the actual results of all laboratory tests used to determine conformance. A batch is defined herein as the quantity of homogeneous material compounded on a rubber mill at one time.

4.2 Rejection. A batch of material which does not meet the requirements of one or more tests under this procedure may be retested once. Failure to meet the requirements with the second testing shall be cause for rejection of the material. Criteria for rejection are established by Table I, Table II and paragraph 3.4.

4.3 Test Methods. Qualification and acceptance testing of the material to the requirements of this procedure shall be in accordance with the following.

4.3.1 Tensile Strength and Elongation. The tensile strength and ultimate elongation shall be determined in accordance with ASTM D412 using Die "C" for the specimens. Results shall conform to the requirements of Table II.

4.3.2 Hardness. The hardness shall be determined in accordance with ASTM D2240. Results shall conform to the requirements of Table II.

4.4 Mixing. This compound shall be formulated according to the recipes and tolerances given in Table I.

TABLE I

FORMULATION GEM 255

Item	Seq. No.	Component Description	phr	Tolerances
a	1	VT-R-4590 (DuPont)	100	±2%
b	2	Austin Black (Cities Service)	10	±1%
c	2	Statex M-568 (N-568) (Cities Service)	25	±2%
d	2	Maglite K (C. P. Hall)	3	±.5%
e	2	Diak #7 (DuPont)	1.5	±.5%
f	2	Luperco 130 XL (Ram Chemicals)	1.5	±.5%

NOTE: The tolerances show the degree of accuracy required for weighing each individual component and the required material balance for the total compound (±5%). This latter value includes losses due to removal of contaminated material. Adjustments to the tolerances may be warranted as more experience is accumulated.

The compound shall be mixed on a two-roll rubber mill precleaned with solvent such as acetone. All parts of the equipment which would come in contact with the compound during the mixing operation shall be cleaned. The floor adjacent to the mill shall be swept to remove contaminants.

4.5 Mixing Procedure

4.5.1 Premix

4.5.1.1 Alternately sprinkle in item f into d and mix until well dispersed.

4.5.1.2 Add b to e until mixture is dry; breakup lumps until mixture is powdered. If all of b is used up before mixture is dry, add c until dry state is reached.

4.5.1.3 Add d and f to b and e sprinkling one into the other, mixing until well dispersed.

4.5.1.4 Take half of the remaining b and c and sprinkle into the above mixture of d + f + b and e.

4.5.1.5 Take last half of b and c and alternately sprinkle into mixture form in 4.5.1.4., above, and mix until well dispersed.

4.5.2 Final Mix. Add mixture formed in 4.5.1.5. to the bulk rubber (component a in Table I) while it is banded on mill.

4.6 Curing Procedures

4.6.1 Test Sheets

4.6.1.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.1.2 Remove stock from mill when approximate thickness of 100 mils is attained.

4.6.1.3 Cut out a 5.75" by 5.75" square from stock making sure to mark mill mixing direction.

4.6.1.4 Put sample into a preheated 75 mil ASTM sheet mold such that mill direction is identifiable.

4.6.1.5 Place loaded mold into heat platens at 350°F for 15 minutes at 20 tons force. After one minute at force, "bump" sample 3 times by releasing pressure to almost zero and then repressurizing to 20 tons.

4.6.1.6 After step 4.6.1.5, remove sample from mold and follow post-curing procedure.

4.6.2 Packer Seals

4.6.2.1 Freshen stock by banding on mill for several minutes until stock is warm and pliable.

4.6.2.2 For a 2.75 inch OD packer seal, the following procedure is used. Place loaded mold onto heat platens at 300°F for 30 minutes at 50 tons force. After one minute subject sample to a "bump" procedure consisting of 3 cycles of pressure change from 50 tons to just above zero, then repressuring to 50 tons.

4.6.2.3 Raise cure temperature to 350°F. Upon reaching 350°F cure for 30 additional minutes.

4.6.2.4 After 4.6.2.3, remove sample from mold and follow post-curing procedure.

4.6.3 O-Rings

4.6.3.2 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.3.2 For a compression mold which makes 4 each 2-329 O-rings the following procedure is used. Place loaded mold into heat platens at 300°F for 30 minutes at 50 tons pressure. After one minute subject sample to a "bump" procedure consisting of 3 cycles of pressure change from 50 tons to just above zero, then repressuring to 50 tons.

4.6.3.3 Raise cure temperature to 350°F. Upon reaching 350°F continue curing for another 15 minutes.

4.6.3.4 After 4.6.3.3, remove sample from mold and follow post-curing procedure.

4.7 Post Curing Procedure

4.7.1 Immediately upon removing cured sample from mold, place sample into 350°F preheated, nitrogen atmosphere oven chamber and continue temperature environment as follows:

Period	Chamber Temp.
1st hour	350°F
1 - 2 hours	400°F
2 - 3 hours	450°F
3 - 20.5 hours	500°F

4.7.2. Remove hot sample from chamber to cool.

4.7.3 Upon cooling, the sample is ready for intended application.

4.8 <u>Properties</u>. Properties of the resultant compounds shall conform to the values and tolerances shown in Table II.

TABLE II

TYPICAL PROPERTIES OF COMPOUND GEM 255

Property Units	Median Value/Tolerance*	
	Virgin	Chem. Aged**
Ultimate tensile strength @ RT, psi	1760 + 440	1270 + 320
Ultimate elongation at break, @ RT, %	180 + 45	180 + 45
Shore A hardness @ 260°C	79 ± 5	79 ± 5
Extrusion resistance @ 260°C, psia	9.1 + 4	5.0 + 3

*Median values have been selected based on test data and tolerances have been selected arbitrarily. It is recommended that these selected values be reviewed and adjusted after more experience is generated.

**Chemical Effects Test Procedure TP10582

4.9 <u>Packaging</u>. When cooled to ambient room temperature (hand test) the rubber shall be cut into appropriate size slabs, wrapped in plastic film and boxed. The box shall be closed to prevent contamination during subsequent handling and storage.

4.10 Marking. The box shall be identified in a clear manner with the following information:

Compound Number
Date of Compounding
Supplier's Name or Other Identification
"STORE IN A COOL, DRY PLACE"

APPLICATION		REVISION			
NEXT ASSY	USED ON	LTR	DESCRIPTION	DATE	APPROVED

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ARE: FRACTIONS ± DECIMALS .XX ± .XXX ± ANGLES ±	CONTRACT NO.	**'GARDE, INC.**	1555 PLACENTIA AVENUE NEWPORT BEACH CALIFORNIA 92660
	APPROVALS / DATE DRAWN R. Sedwick 11 JUN 79 CHECKED	Procedure for Preparation of EPDM/Fluoro-elastomer Blend Compound GEM 266	
MATERIAL		SIZE A	CODE IDENT NO. 1F668
FINISH		DRAWING NO. 10668	
DO NOT SCALE DRAWING		SCALE	SHEET 1 OF 7

1. SCOPE

This procedure establishes the formulation, mixing methodology and steps for preparing GEM 266 rubber compounds intended for use as geothermal packer seal elements.

2. APPLICABLE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. Later issues of these documents may be used at the option of the supplier providing no degradation of the product ensues.

Federal

Q-A-51	Acetone, Technical

American Society for Testing and Materials

ASTM D2516	Nomenclature for Rubber-Grade Carbon Blacks
ASTM D15	Compound and Sample Preparation for Physical Testing of Rubber Products
ASTM D412	Tension Testing of Vulcanized Rubber
ASTM D2240	Indentation Hardness of Rubber and Plastics By Means of a Durometer, Test for

3. REQUIREMENTS

3.1 Material. All materials used for the preparation of these rubber compounds shall be formulated and processed to meet the requirements of this procedure. All materials shall be homogeneous, clean and suitable for the purpose intended.

3.2 Processing. The compounds shall be prepared according to this procedure.

3.3 Mechanical Properties. The compounded rubber shall have properties according to this procedure.

3.4 Workmanship. Each batch of rubber shall be prepared, handled and stored to minimize contamination. Contamination is herein defined as the presence of any material in the compound which is visible to the unaided eye. Contaminated rubber may be removed from the compound if it does not exceed 1% by weight of the compound at the time of removal. If the contaminated rubber exceeds 1% by weight, the compound must be discarded.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance. The supplier of materials under this procedure shall certify that each batch conforms to all requirements of the procedure. Certification shall include the actual results of all laboratory tests used to determine conformance. A batch is defined herein as the quantity of homogeneous material compounded on a rubber mill at one time.

4.2 Rejection. A batch of material which does not meet the requirements of one or more tests under this procedure may be retested once. Failure to meet the requirements with the second testing shall be cause for rejection of the material. Criteria for rejection are established by Table I, Table III and paragraph 3.4.

4.3 Test Methods. Qualification and acceptance testing of the material to the requirements of this procedure shall be in accordance with the following:

4.3.1 Tensile Strength and Elongation. The tensile strength and ultimate elongation shall be determined in accordance with ASTM D412 using Die "C" for the specimens. Results shall conform to the requirements of Table II.

4.3.2 Hardness. The hardness shall be determined in accordance with ASTM D2240. Results shall conform to the requirements of Table II.

4.4 Mixing. This compound shall be formulated according to the recipes and tolerances given in Table I.

TABLE I

FORMULATION OF GEM 266 COMPOUND

Item	Seq. No.	Component Description	phr	Tolerances
a	1	Nordel 1660 (DuPont)	50	±2%
b	2	VT-R-4590 (DuPont)	50	±2%
c	3	Hypalon 20 (DuPont)	2.5	±.5%
d	5	Austin Black (Cities Service)	7.5	±1%
e	5	Maglite K (C. P. Hall)	2.5	±.5%
f	4	Statex 160 (N110)(Cities Service)	52.5	±2%
g	5	Diak #7 (DuPont)	3.0	±.5%
h	5	Luperco 130XL (Ram Chemicals)	1.5	±.5%
i	5	Cyanox 2246 (American Cyanamid Co.)	.25	±.5%
j	5	Thermoguard S (M & T Chemicals Inc.)	2.5	±.5%

NOTE: The tolerances show the degree of accuracy required for weighing each individual component and the required material balance for the total compound (±5%). This latter value includes losses due to removal of contaminated material. Adjustments to the tolerances may be warranted as more experience is accumulated.

The compound shall be mixed on a two-roll rubber mill precleaned with solvent such as acetone. All parts of the equipment which could come in contact with the compound during the mixing operation shall be cleaned. The floor adjacent to the mill shall be swept to remove contaminants.

4.5 Mixing Procedure

4.5.1 Premix

4.5.1.1 Add d to g until mixture is dry; break lumps until mixture is powdered. If all of d is used before mixture is dry, add f until dry state is reached.

4.5.1.2 Combine e and h, then add i, then j, then d and g sprinkling one into the other, mixing until well dispersed after the addition of each component.

4.5.2 Final Mix

4.5.2.1 Combine items a, b and c on mill and run several minutes to make the banded rubber warm and pliable.

4.5.2.2 Slowly add f then the mixture resulting from 4.5.1.2. As each component of combination of components is added to the mill, frequent cross-cutting of the rubber should be accomplished for uniform mixing.

4.6 Curing Procedures

4.6.1 Test Sheets

4.6.1.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.1.2 Remove stock from mill when approximate thickness of 100 mils is attained.

4.6.1.3 Cut out a 5.75" by 5.75" square from sheet making sure to mark mill mixing direction.

4.6.1.4 Put sample into a preheated 75 mil ASTM sheet mold, such that mill direction is identifiable.

4.6.1.5 Place loaded mold into heat platens at 350^{O}F for 60 minutes at 20 tons force. After one minute at force "bump" sample 3 times by releasing force to almost zero and then reapplying it to 20 tons.

4.6.1.6 After step 4.6.1.5, remove sample from mold and follow post-curing procedures.

4.6.2 Packer Seals

4.6.2.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.2.2 For a 2.75 inch OD packer seal, the following procedure is used. Place loaded mold into heat platens at 350°F for 90 minutes at 50 tons force. After one minute subject sample to a "bump" procedure consisting of 3 cycles of force change from 50 tons to just above zero, then repressuring to 50 tons.

4.6.2.3 After 4.6.2.2, remove sample from mold and follow post-curing procedure.

4.6.3 O-Rings

4.6.3.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.3.2 For a compression mold which makes 4 each 2-329 O-rings the following procedure is used. Place loaded mold into heat platens at 350°F for 60 minutes at 50 tons force. After one minute subject sample to a "bump" procedure consisting of 3 cycles of force change from 50 tons to just above zero, then repressuring to 50 tons.

4.6.3.3 After 4.6.3.2, remove sample from mold and follow post-curing procedure.

4.7 Post-Curing Procedure

4.7.1 Immediately upon removing cured sample from mold, place sample into 350°F preheated, nitrogen atmosphere oven chamber and continue temperature environment as follows:

Period	Chamber Temp.
1st hour	400°F
1 - hours	450°F
2 - 3 hours	500°F
3 - 8.0 hours	550°F

After 8 hours cool to at least 300°F in nitrogen oven.

4.7.2 Upon cooling the sample is ready for intended application.

4.8 Properties. Properties of the compound shall conform to the values and tolerances shown in Table II.

TABLE II

TYPICAL PROPERTIES OF COMPOUND GEM 266

Property, Units	Median Value/Tolerance*	
	Virgin	Chem Aged**
Ultimate Tensile Strength @ RT, psi	1280 + 320	1220 + 305
Ultimate Elongation at Break, @ RT, %	150 + 38	130 + 33
Shore A Hardness @ 260°C	88 ± 6	89 ± 6
Extrusion Resistance @ 260°C, psia	14 + 5	14 + 5

* Median Values have been selected based on test data and tolerances have been selected arbitrarily. It is recommended that these selected values be reviewed and adjusted after more experience is generated.

** Chemical Effects Test Procedure TP10582

4.9 Packaging. When cooled to ambient room temperature (hand test) the rubber shall be cut into appropriate size slabs, wrapped in plastic film and boxed. The box shall be closed to prevent contamination during subsequent handling and storage.

4.10 Marking. The box shall be identified in a clear manner with the following information:

Compound Number
Date of Compounding
Supplier's Name or Other Identification
"STORE IN A COOL, DRY PLACE"

APPENDIX D

APPLICATION		REVISION			
NEXT ASSY	USED ON	LTR	DESCRIPTION	DATE	APPROVED

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ARE:	CONTRACT NO.	
FRACTIONS ± DECIMALS .XX ± .XXX ± ANGLES ±	APPROVALS	DATE
	DRAWN G. Friese GJF	4/16/80
MATERIAL	CHECKED	
FINISH		
DO NOT SCALE DRAWING		

'GARDE, INC.

1555 PLACENTIA AVENUE
NEWPORT BEACH
CALIFORNIA 92660

PROCEDURE FOR MIXING L'GARDE EPDM COMPOUND Y267

SIZE	CODE IDENT NO.	DRAWING NO.
A	1F668	10416

SCALE		SHEET 1 OF 4

BISHOP GRAPHICS, INC.
REORDER NO. 20500

1.0 SCOPE

This procedure establishes the formulation and mixing process for preparing L'Garde Y267 rubber compound.

2.0 APPLICABLE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. Later issues of these documents may be used at the option of the supplier providing no product degradation results.

<u>Federal</u>

Q-A-51	Acetone

<u>American Society for Testing and Materials</u>

ASTM D2516	Nomenclature for Rubber-Grade Carbon Blacks

3.0 REQUIREMENTS

3.1 <u>Material</u>. All materials used for the preparation of these rubber compounds shall be formulated and processed to meet the requirements of this procedure. All materials shall be homogeneous, clean and suitable for the purpose intended.

3.2 <u>Processing</u>. The compounds shall be prepared according to this procedure.

3.3 <u>Workmanship</u>. Each batch of rubber shall be prepared, handled and stored to minimize contamination. Contamination is herein defined as the presence of any material in the compound which is visible to the unaided eye. Contaminated rubber may be removed from the compound if it does not exceed 1% by weight of the compound at the time of removal. If the contaminated rubber exceeds 1% by weight, the compound must be discarded.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 <u>Acceptance</u>. The supplier of materials under this procedure shall certify that each batch conforms to all requirements of the procedure.

A batch is defined herein as the quantity of homogeneous material compounded on a rubber mill at one time.

4.2 Rejection. A batch of material which does not meet the requirements of this procedure shall be cause for rejection. Criteria for rejection are established by 3.3 and Table I.

4.3 Mixing. The compound shall be formulated per Table I. The tolerances show the degree of accuracy required for weighing each individual component. The material balance for the total compound shall be ±5%. This latter value includes losses due to removal of contaminated material.

TABLE I
Y267 Compound Formulation

Item	Component Description	phr	Tolerances
a	Nordel 1660 (DuPont)	100.0	±2.0%
b	Statex 160 (N110) (Cities Service)	75.0	±2.0%
c	Cyanox 2246 (American Cyanamid Co.)	0.5	±0.5%
d	Di-Cup R (Harwick Chemical Corp.)	3.5	±0.5%
e	Hypalon 20 (DuPont)	5.0	±1.0%
f	Thermoguard S (M & T Chemicals Inc.)	5.0	±1.0%
g	Polybutadiene, #6081 (Polysciences, Inc.)	20.0	±1.0%

4.3.1 Pre-Mix

4.3.1.1 Dry b at 250°F.

4.3.1.2 Heat b and g to 150°F. Add just enough of b to g to produce a dry mix. Mix thoroughly, and set aside.

4.3.1.3 Thoroughly mix c and f. Slowly add the remaining b while continuing to mix.

4.3.2 Final Mix. The compound shall be mixed on a two-roll rubber mill precleaned with solvent such as acetone (per Q-A-51). All parts of the equipment which could come in contact with the compound during the mixing operation shall be cleaned. The floor adjacent to the mill shall be swept to remove contaminants. (Development was performed on a laboratory basis only -- using a 8" x 18" mill.)

4.3.2.1 Combine a and e on mill. Add 4.3.1.3 mixture slowly. Add 4.3.1.2 mixture at a rate such that the rubber remains banded.

4.3.2.2 Mix and cut for six minutes. Cigar roll six times.

4.3.2.3 Add d.

4.3.2.4 Mix and cut for four minutes. Cigar roll six times.

4.4 Packaging. When cooled to room temperature, the rubber shall be cut into appropriate size slabs, wrapped in plastic film and boxed. The box shall be closed to prevent contamination during subsequent handling and storage.

4.5 Marking. The box shall be identified in a clear manner with the following information:

Compound Number
Date of Compounding
Supplier's Name or Other Identification
"STORE IN A COOL, DRY PLACE"

APPENDIX E

APPLICATION		REVISION			
NEXT ASSY	USED ON	LTR	DESCRIPTION	DATE	APPROVED

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ARE: FRACTIONS ± DECIMALS .XX ± .XXX ± ANGLES ±	CONTRACT NO.		**L'GARDE, INC.** 1555 PLACENTIA AVENUE NEWPORT BEACH CALIFORNIA 92660
	APPROVALS	DATE	
	DRAWN G. Friese GJF	4-14-80	PROCEDURE FOR COMPRESSION MOLDING L'GARDE EPDM COMPOUND Y267
MATERIAL	CHECKED		
FINISH			SIZE A · CODE IDENT NO. 1F668 · DRAWING NO. 10417
DO NOT SCALE DRAWING			SCALE · SHEET 1 OF 4

1. SCOPE

This procedure establishes the process for compression molding parts made from L'Garde Y267 rubber compound.

2. APPLICABLE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. Later issues of these documents may be used at the option of the supplier providing no product degradation results.

American Society for Testing and Materials

ASTM D15	Compound and Sample Preparation for Physical Testing of Rubber Products.
ASTM D412	Tension Testing of Vulcanized Rubber
ASTM D2240	Indentation Hardness of Rubber and Plastics By Means of a Durometer, Test for

3. REQUIREMENTS

3.1 Material. Y267 rubber shall be mixed per L'Garde specification 10416.

3.2 Processing. The parts shall be prepared according to this procedure.

3.3 Mechanical Properties. The cured rubber shall have properties specified herein.

3.4 Workmanship. Uncured rubber shall be handled and stored to minimize contamination. Contamination is herein defined as the presence of any material in the compound which is visible to the unaided eye. Contaminated rubber may be removed from the compound if it does not exceed 1% by weight of the compound at the time of removal. If the contaminated rubber exceeds 1% by weight, the compound must be discarded.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance. The supplier of parts under this procedure shall certify that they conform to all requirements of the procedure. Certification shall include the actual results of all laboratory tests used to determine conformance.

4.2 Rejection. Material which does not meet the requirements of one or more tests under this procedure may be retested once. Failure to meet the requirements with the second testing shall be cause for rejection of the material. Criteria for rejection are established by 3.4 and 4.6.

4.3 Test Methods. Qualification and acceptance testing of the material to the requirements of this procedure shall be in accordance with the following:

4.3.1 Tensile Strength and Elongation. The tensile strength and ultimate elongation shall be determined in accordance with ASTM D412 using Die "C" for the specimens. Results shall conform to the requirements of 4.6.

4.3.2 Hardness. The hardness shall be determined in accordance with ASTM D2240. Results shall conform to the requirements of 4.6.

4.4 Curing.

4.4.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.4.2 Preheat the compression mold to 350°F and load. (The completed part will be approximately 3% smaller than the mold, both at room temperature.)

4.4.3 Place loaded mold into heat platens at 350°F and close press as rapidly as possible, with at least 3,000 psi pressure over the part plus overflow area.

4.4.4 After one minute, quickly mold to three (3) bump cycles -- each cyle consists of reducing the compressive force to near zero and then increasing it back to the 4.4.3 pressure.

4.4.5 Keep mold at 350°F and the 4.4.4 pressure for one hour for parts up to ¼-inch thick and up to two hours for thicker parts. (No effort has been expended to investigate optimum cure time.)

4.4.6 Remove part from the mold, and immediately start post-curing.

4.5 Post-Curing.

4.5.1 Immediately after removing cured part from mold, place part into a nitrogen atmosphere oven chamber preheated to 350°F. The parts may be left at 350°F for up to eight hours while additional cured parts are added. Post-cure as follows for parts up to ¼-inch thick; add another hour at 400°F for thicker parts:

Period	Set Temp.
0 - 1 hour	400°F
1 - 2 hours	450°F
2 - 3 hours	500°F
3 - 8 hours	550°F

4.5.2 Cool the parts to less than 300°F, while in the nitrogen environment, within one hour.

4.5.3 Remove from nitrogen atmosphere.

4.6 Properties. Based upon limited data, properties of the compound should conform to the following values:

Ultimate Tensile Strength @ RT (psi)	1900 ±200
Ultimate Elongation @ RT (%) (grip travel)	190 ±20
Shore A Hardness @ 500°F	87 ±4

APPLICATION		REVISION			
NEXT ASSY	USED ON	LTR	DESCRIPTION	DATE	APPROVED

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCES ARE: FRACTIONS ± DECIMALS .XX ± .XXX ± ANGLES ±	CONTRACT NO.		**L'GARDE, INC.** 1555 PLACENTIA AVENUE NEWPORT BEACH CALIFORNIA 92660
	APPROVALS	DATE	
	DRAWN R. Sedwick	11 Jun 79	Procedure for preparation of Propylene-TFE Compound GEM 291
MATERIAL	CHECKED		
FINISH			SIZE A · CODE IDENT NO. 1F668 · DRAWING NO. 10670
DO NOT SCALE DRAWING			SCALE · SHEET 1 OF 8

1. SCOPE

This procedure establishes the formulation, mixing methodology and steps for preparing the GEM 291 rubber compound intended for use as Geothermal Packer seal elements.

2. APPLICABLE DOCUMENTS

The following documents form a part of this specification of the extent specified herein. Later issues of these documents may be used at the option of the supplier providing no degradation of the product ensues.

Federal

Q-A-51	Acetone, Technical

American Society for Testing and Materials

ASTM D2516	Nomenclature for Rubber-Grade Carbon Blacks
ASTM D15	Compound and Sample Preparation for Physical Testing of Rubber Products
ASTM D412	Tension Testing of Vulcanized Rubber
ASTM D2240	Indentation Hardness of Rubber and Plastics By Means of a Durometer, Test for.

3. REQUIREMENTS

3.1 Material. All materials for the preparation of these rubber compounds shall be formulated and processed to meet the requirements of this procedure. All materials shall be homogeneous, clean and suitable for the purpose intended.

3.2 Processing. The compounds shall be prepared according to this procedure.

3.3 Mechanical Properties. The compounded rubber shall have properties according to this procedure.

3.4 Workmanship. Each batch of rubber shall be prepared, handled and stored to minimize contamination. Contamination is herein defined as the presence of any material in the compound which is visible to the unaided eye. Contaminated rubber may be removed from the compound if it does not exceed 1% by weight of the compound at the time of removal. If the contaminated rubber exceeds 1% by weight, the compound must be discarded.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance. The supplier of materials under this procedure shall certify that each batch conforms to all requirements of the procedure. Certification shall include the actual results of all laboratory tests used to determine conformance. A batch is defined herein as the quantity of homogeneous material compounded on a rubber mill at one time.

4.2 Rejection. A batch of material which does not meet the requirements of one or more tests under this procedure may be re-tested once. Failure to meet the requirements with the second testing shall be cause for rejection of the material. Criteria for rejection are established by Table I, Table II and paragraph 3.4.

4.3 Test Methods. Qualification and acceptance testing of the material to the requirements of this procedure shall be in accordance with the following:

4.3.1 Tensile Strength and Elongation. The tensile strength and ultimate elongation shall be determined in accordance with ASTM D412 using Die "C" for the specimens. Results shall conform to the requirements of Table II.

4.3.2 Hardness. The hardness shall be determined in accordance with ASTM D2240. Results shall conform to the requirements of Table II.

4.4 Mixing. This compound shall be formulated according to the recipes and tolerances given in Table I.

TABLE I
FORMULATION GEM 291

Item	Seq. No.	Component Description	phr	Tolerances
a	1	AFLAS 100 (Asahi Glass)	100	±2%
b	2	Statex 160 (N110) (Cities Service)	50	±2%
c	3	Maglite K (C.P. Hall)	3.0	±.5%
d	3	Luperco 130 XL (RAM Chemicals)	5.0	±1%
e	3	Diak #7 (DuPont)	5.0	±1%
f	4	Polybutadiene, #6081 (Polysciences, Inc.)	5.0	±1%

NOTE: The tolerances show the degree of accuracy required for weighing each individual component and the required material balance for the total compound (±5%). This latter value includes losses due to removal of contaminated material. Adjustments to the tolerances may be warranted as more experience is accumulated.

The compound shall be mixed on a two-roll rubber mill precleaned with solvent such as acetone. All parts of the equipment which would come in contact with the compound during the mixing operation shall be cleaned. The floor adjacent to the mill shall be swept to remove contaminants.

4.5 Mixing Procedure

4.5.1 Premix

4.5.1.1 Add b to e until mixture is dry; break lumps until mixture is powdered.

4.5.1.2 Heat b and f to 125°F, then mix thoroughly, while still hot, enough b to f to make a dry mix.

4.5.1.3 Combine c and d, then add b + e sprinkling one into the other, mixing until well dispersed after the addition of each component.

4.5.2 Final Mix

4.5.2.1 Add b, then mixture resulting from 4.5.1.3, then b + f to banded rubber on mill. As each component is added to the mill, frequent cross-cutting of the rubber should be accomplished for uniform mixing.

4.6 Curing Procedures

4.6.1 Test Sheets

4.6.1.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.1.2 Remove stock from mill when approximate thickness of 100 mils is attained.

4.6.1.3 Cut out a 5.75" by 5.75" square from stock making sure to mark mill mixing direction.

4.6.1.4 Put sample into a preheated 75 mil ASTM sheet mold such that mill direction is identifiable.

4.6.1.5 Place loaded mold into heat platens at 350°F for 20 minutes at 20 tons force. After one minute at force, "bump" sample 3 times by releasing pressure to almost zero and then repressurizing to 20 tons.

4.6.1.6 After step 4.6.1.5, remove sample from mold and follow post-curing procedure.

4.6.2 Packer Seals

4.6.2.1 Freshen stock by banding on mill for several minutes until stock is warm and pliable.

4.6.2.2 For a 2.75 inch OD packer seal, the following procedure is used. Place loaded mold onto heat platens at 250°F for 30 minutes at 50 tons force. After one minute subject sample to a "bump" procedure consisting of 3 cycles of pressure change from 50 tons to just above zero, then repressuring to 50 tons.

4.6.2.3 Raise cure temperature to 350°F. Upon reaching 350°F cure for 60 additional minutes.

4.6.2.4 After 4.6.2.3, remove sample from mold and follow post-curing procedure.

4.6.3 O-Rings

4.6.3.1 Freshen basic stock by banding on mill for several minutes until stock is warm and pliable.

4.6.3.2 For a compression mold which makes 4 each 2-329 O-rings the following procedure is used. Place loaded mold into heat platens at 250°F for 30 minutes at 50 tons pressure. After one minute subject sample to a "bump" procedure consisting of 3 cycles of pressure change from 50 tons to just above zero, then repressuring to 50 tons.

4.6.3.3 Raise cure temperature to 350°F. Upon reaching 350°F continue curing for another 20 minutes.

4.6.3.4 After 4.6.3.3, remove sample from mold and follow post-curing procedure.

4.7 Post Curing Procedure

4.7.1 Immediately upon removing cured sample from mold, place sample into 350°F preheated, nitrogen atmosphere oven chamber and continue temperature environment as follows:

Period	Chamber Temp.
1st 4½ hours	350°F
4½ - 5 hours	400°F
5 - 5½ hours	450°F
5½ - 8 hours	500°F

After 8 hours cool to at least 300°F in nitrogen oven.

4.7.2 Upon cooling, the sample is ready for intended application.

4.8 Properties. Properties of the resultant compounds shall conform to the values and tolerances shown in Table II on the following page.

TABLE II

TYPICAL PROPERTIES OF COMPOUND GEM 291

Property, Units	Median Value/Tolerance*	
	Virgin	Chem Aged**
Ultimate Tensile Strength @ RT, psi	1870 + 470	1730 + 430
Ultimate Elongation at Break, @ RT, %	110 + 28	130 + 33
Shore A Hardness @ 260°C	95 ± 6	95 ± 6
Extrusion Resistance @ 260°C, psia	14 + 5	12 + 5

*Median values have been selected based on test data and tolerances have been selected arbitrarily. It is recommended that these selected values be reviewed and adjusted after more experience is generated.

**Chemical Effects Test Procedure TP10582.

4.9 Packaging. When cooled to ambient room temperature (hand test) the rubber shall be cut into appropriate size slabs, wrapped in plastic film and boxed. The box shall be closed to prevent contamination during subsequent handling and storage.

4.10 Marking. The box shall be identified in a clear manner with the following information:

Compound Number
Date of Compounding
Supplier's Name or Other Identification
"STORE IN A COOL, DRY PLACE"

CONFERENCE PROCEEDINGS

"APPLYING CURRENT GEOTHERMAL CORROSION/MATERIALS TECHNOLOGY TO TODAY'S PROJECTS"

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PITFALLS OF ELASTOMER COMPATIBILITY TESTING

Gilbert J. Friese

L'Garde, Inc.
1555 Placentia Avenue
Newport Beach, California 92663

ABSTRACT

An extensive compatibility test program was conducted starting with 34 compounds and six 190C fluids. Both immersion tests and simulation tests were conducted for time periods ranging from 46 hours to over six months. Deficiencies in both types of tests were determined. Immersion tests, while useful for reducing the number of candidate compounds, can easily lead to incorrect conclusions. It is essential that simulation tests be conducted before a final elastomer is selected for use in a critical design.

INTRODUCTION

In early 1980, Brookhaven National Laboratory sponsored an extensive test program to determine the best elastomers for use in geothermal binary power-plant applications. The program, now complete, met its objectives. In addition, much was learned concerning the idiosyncrasies of such compatibility testing. The purpose of this paper is to discuss main observations and conclusions regarding elastomer testing in hostile environments.

TEST PROGRAM APPROACH

The program plan is shown in Figure 1. The program started with the short-term testing of a large number of elastomers, and ended with the long-term testing of only a few believed to be the best.

Equipment and elastomeric parts manufacturers were contacted to find the best currently available high-temperature elastomers for the binary power plant application. Tensile specimens of 34 such compounds were immersion tested for five days (120 hours) in six 190C (375F) fluids of interest: isobutane, brine, ASTM No. 1 oil, ASTM No. 3 oil, Pacer DHT-185M synthetic oil, and Chevron Cylinder Grade 460X oil. The best eight were selected based upon the least change in weight, volume, ultimate tensile strength and elongation, and hardness.

These eight were then simultaneously tested a) by immersion in five 190C (375F) fluids for six months and b) as O-rings for 46 hours at 190C, 230C, and 265C (375F, 450F, 510F) (accelerated ageing) in three fluids and at a differential pressure of 21 MPa (3000 psi).

Based upon these O-ring tests, four compounds were selected for testing as O-rings for approximately six months in three fluids at 204C (400F) and 21 MPa differential pressure.

IMMERSION TEST PITFALLS

Tensile specimens of each compound were tested in autoclaves generally at the vapor pressure of the fluid. Such tests are economical, but can lead to incorrect conclusions.

Mechanical Property Changes

In the immersion testing; weight, volume, ultimate tensile properties, and hardness are measured before and after ageing the elastomer in the heated fluid of interest. If these properties don't change, one would assume that the elastomer is immune to that fluid and would be an excellent choice for the application.

However, changes are likely to occur -- so the obvious thing to do is to select those that change the least. This is not always valid because the properties that can be measured in immersion tests are not necessarily those required for a good seal material.

Table I shows the percentage changes for eight compounds after immersion for 120 hours in 190C ASTM No. 1 oil. (These eight were selected from the 34 tested as the most promising for one or more of the six fluids of interest.)

The only easy polymer to delete is EPDM (which the immersion tests indicated was good in brine and isobutane). These eight compounds were then tested as O-rings with ASTM No. 1 oil at a 21 MPa (3000 psi) differential pressure for 46 hours; tests were conducted at 190C, 230C, and 265C (375F, 450F, and 510F).

The EPDMs were ranked the best of the eight. The EPDMs did swell about 10%, but remained circular in cross section. All others exhibited various degrees of permanent set -- the worst being almost square in cross section. In addition, the AFLAS and Kalrez compounds extruded slightly.

Later in the 6-month long O-ring tests, the EPDM O-ring did experience very slow nibbling. (More about this later.) However, they are still ranked best or nearly so.

Immersion tests submit the specimens to the thermochemical effects of a hot fluid environment. With experience, one can sort through the large quantity of data and narrow the list candidates.

However, as the above illustration shows, immersion tests alone are insufficient for selecting the correct elastomer for a hostile environment. Simulation tests which produce the mechanical effects as well as the thermochemical effects are essential.

Test Duration

Immersion tests usually are conducted for less than five days. Results from the two and six-month tests showed that even five days can be insufficient for long-term applications.

The five-day tests gave no indication that any of the eight compounds were becoming brittle in any of the oils. (Table I for instance.) However, at two months, Precision Fluoroelastomer 16959 was brittle after immersion in both ASTM No. 1 oil and Chevron Cylinder Grade 460X oil. After six months, L'Garde Viton 501 and Vernay Laboratory VL1503M3 in Chevron Cylinder Grade 460X oil joined the brittle group. In addition, the ultimate elongations of the Precision and Vernay fluoroelastomers in Pacer DHT-185M oil had decreased 58% -- indicating they too might be heading for the brittle column.

Figure 2 shows average ultimate elongation changes for three polymers (2 compounds each in three oils). The loss in elasticity for the fluoroelastomers is clear -- but only after 2 months or more of testing. The EPDM and AFLAS compounds look all right.

Therefore, short-term immersion tests may lead to erroneous conclusions also.

Rarely does one have the luxury of testing for the duration of the product's desired life. Accelerated ageing tests may be the solution. Arrhenius observed that reaction rates increased with increasing temperature.

The problem with accelerated ageing is that new reactions may start at some higher temperature. (As an analogy, accelerated ageing of ice above 0°C would give invalid results.) Therefore, accelerated ageing might result in dismissing a candidate that could outperform others at a lower temperature. It is important then to stay within the reactivity continuum and not transcend thresholds to new reactions.

Correct Test Fluid

The tests in oil also showed that immersion testing should be conducted in the fluid of interest -- not in a "similar" fluid.

In reviewing the short-term immersion test data, it appeared that ASTM No. 1 oil, Chevron Cylinder Grade 460X oil and Pacer DHT-185M oil had about the same affect on the elastomers. (An exception noted was that the Pacer oil affected EPDM compounds less.) The longer term immersion tests showed that Pacer oil generally affected all elastomers ~~least~~ (of the three oils) and Chevron Cylinder Grade 460X oil affected them generally the most. Once observing this, another review of the short-term data supported this conclusion.

The relative rankings of elastomers can change from oil to oil also. A dramatic example of this was with Kalrez 1018 which tends to be a superior compound in oil immersion tests. In the Pacer and ASTM No. 1 oils, it was superior; it could almost be considered immune to these 190C (375F) fluids. After two months in Chevron Cylinder oil, however, it absorbed the oil gaining weight and volume, developed numerous longitudinal cracks resulting in reduced ultimate strength and elongation, and lost 20% of its hardness.

Charts showing performance of polymers or specific compounds in generic type fluids can provide valuable guidance. For hostile environments, however, it is essential to test the compounds in the specific fluids of interest -- and as noted earlier, it is important to use immersion tests strictly for screening, and simulation tests for making the final decision. Field tests should be used for confirmation.

ACCELERATED AGEING O-RING TEST PITFALLS

Although the immersion tests were all conducted at 190C (375F), the O-ring tests were conducted at 190C, 230C, and 265C (375F, 450F, 510F) for 46 hours to simulate 190C (375F) for approximately 2 days, 1 month and 1 year, respectively, at a 21 MPa (3000 psi) differential pressure. In general, and as expected, damage such as permanent set, nibbling, extrusion (and of course failure) increased with temperature. (Also as expected, there was no indication that thresholds to new reactions were transcended over this temperature range.)

Two fluoroelastomers and two EPDMs were then tested in three of the fluids at 204C (400F) for six months. Failures occurred in less time predicted by the short-term accelerated O-ring tests and the Arrhenius equation. Inspection of the O-rings after testing, showed that nibbling or extrusion was more common in the long-term tests.

For instance, the EPDMs tested with isobutane and ASTM No. 1 oil for 46 hours showed no visible signs of damage except for the Parker EPDM E692-75 in 265C (510F) isobutane; a large section had been nibbled away. In the long-term tests, both EPDMs showed some degree of nibbling. In isobutane, it was enough to cause catastrophic failure. In 204C (400F) ASTM No. 1 oil for 209 days (6.8 months), there was moderate nibbling and no failures. (In the latter tests, L'Garde Viton 501 and Vernay Fluoroelastomer VL1503M3 both experienced slight nibbling, severe permanent set and no failures. The Vernay compound was brittle as was seen in the long-term immersion tests.)

Based upon all the O-ring test data, mechanical damage (such as permanent set, nibbling and extrusion) does increase with temperature. However, this is somewhat "proportional" to the thermochemical degradation produced by accelerated ageing. In addition, there can be mechanical damage that is time dependent only. (Nibbling is visualized as rubber entering the gap, tearing off because of tension or shear, and being replaced by new rubber to continue the process.)

Therefore, accelerated ageing (increased temperature) simulation tests can result in less mechanical damage than will be seen at the operating temperature. Perhaps mechanical damage can be accelerated more realistically by higher pressure, but it would take considerable work to develop such a relationship. Accelerated aged simulation tests are still the most valuable tool for expediently evaluating long-term elastomer-fluid compatibility.

SUMMARY CONCLUSIONS

Immersion tests provide a lot of data economically. However, results must be interpreted with caution using analysis and experience.

1. Based upon the results of EPDM compounds in oil, screening out polymer systems based on immersion test data alone may be a mistake -- unless perhaps all compounds within the group become brittle. It is essential that simulation testing be conducted for accurate evaluation for hostile environments.

2. For long-term applications, immersion testing must be conducted over a long time period or at some limited but increased temperature. Careful judgement must be exercised to avoid extrapolating short-term data which can be extremely erroneous.

3. Immersion testing should be conducted in the exact fluid of interest. Compatibility data from similar fluids can provide guidance, but the exact fluid is essential for critical applications.

Simulation testing is essential for accurate evaluation of elastomers for hostile environments. It is superior to immersion testing (although more expensive) in that it provides the mechanical (stresses) damage mechanisms as well as thermochemical effects. If long-term simulation tests aren't practical, carefully executed increased temperature (accelerated aged) simulation tests are recommended even though mechanical damage may be less severe than in actual use.

ACKNOWLEDGEMENTS

The Environmental Compatibility Testing Program required considerable outside cooperation. People from seventeen companies contributed technical information as well as elastomer specimens, oil or back-up rings. It would have been difficult to perform this type of program without such support.

Dr. Larry Kukacka of Brookhaven National Laboratory provided valuable technical assistance and suggestions in the course of monitoring the program. Alan Hirasuna of L'Garde provided considerable information concerning the problems of elastomers and compatibility testing; having such experience available is essential to cost effective research.

BIBLIOGRAPHY

Friese, G. J., "Elastomer Compatibility With Brine, Isobutane, and Oils", 1981 Annual Meeting, Geothermal Resources Council, 25-29 October 1981, Houston, Texas.

Friese, G.J., Performance of Elastomers in Hot Brine, Isobutane and Oils, L'Garde, Inc. Publication Number LTR-81-GF-102, September 1981.

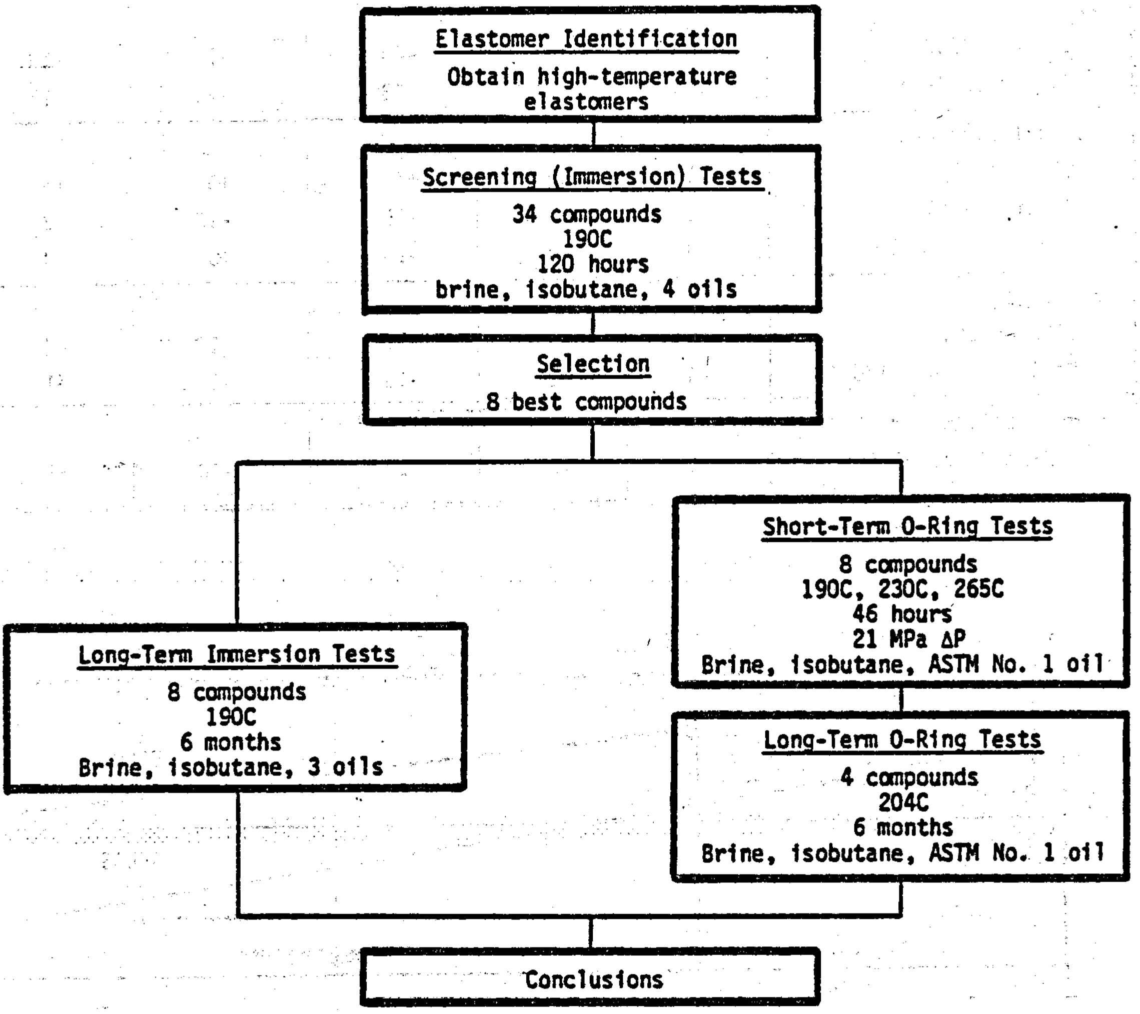

FIGURE 1. PROGRAM APPROACH

TABLE I. EFFECT OF 190C ASTM NO. 1 OIL FOR 120 HOURS

Compound	Wt. Chg. (%)	Swell (%)	Ult. Stress (%)	Ult. Elong. (%)	Hardness (%)
EPDM:					
L'Garde Y267	+58	+69	-48	-7	-19
Parker E692-75	+44	+61	-38	-21	-17
Fluoroelastomers:					
L'Garde 501	-1	+0	+7	+3	+6
Precision 16959	-1	+0	-11	-12	+3
Vernay VL1503M3	-0	+0	-1	+0	+4
AFLAS					
L'Garde 291	+2	+4	-11	+19	-4
Seals Eastern 7170X14	-0	+2	-18	-1	+1
Perfluoroelastomer					
DuPont Kalrez 1018	-1	+1	+1	-10	+6

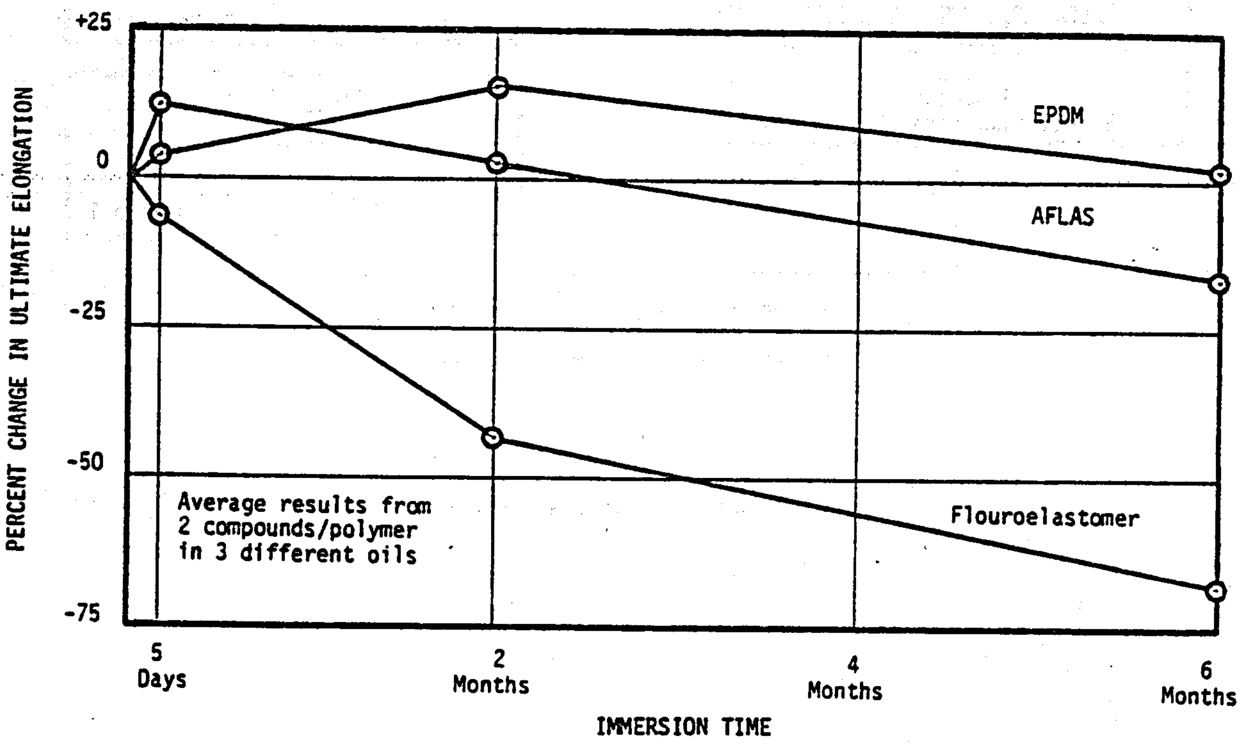

FIGURE 2. FLUOROELASTOMER COMPOUNDS BECOMING BRITTLE IN OIL

HIGH-TEMPERATURE Y267 EPDM ELASTOMER
FIELD AND LABORATORY EXPERIENCES, SEPTEMBER 1982

Alan R. Hirasuna
Gilbert J. Friese
Clifford A. Stephens

L'Garde, Inc.
Newport Beach, California 92663

ABSTRACT

High performance elastomers were developed for hostile geothermal environments which clearly advance the state-of-the-art. The Y267 EPDM compound is eminently successful and has accumulated broad laboratory and field test experience. Over 15 separate tests are reviewed with about 95% performed independently by other organizations. The tests include a broad spectrum of environments with temperatures in excess of 320C (608F), differential pressures up to 138 MPa (20,000 psi) and in fluids including brine, oils, isobutane, and others.

INTRODUCTION

In 1976, reliable elastomers for the unusually severe geothermal environment at 260C (500F) did not exist. L'Garde, Inc. developed geothermal elastomer compounds during the period of 1976-1979 under U.S. Department of Energy-Division of Geothermal Energy (DOE-DGE) contract DE-AC0-77ET28309 [1]. The resulting developments yielded compounds from four polymer systems which successfully exceeded the contract requirements.

Since completion of the compound development, widespread laboratory and downhole experience occurred. Though all developments exceeded the requirements of the development contract, data show that the Y267 EPDM compound is eminently successful. This paper summarizes over 15 laboratory and field tests of the Y267 EPDM under various extreme conditions. 95% of the tests were performed independent of L'Garde by other organizations. These and other substantiating data clearly show that Y267 DPDM significantly advances the state-of-the-art of high-temperature elastomers.

OTIS ENGINEERING CORP. LAB PACKER TESTS

Y267 EPDM packer seals were molded by L'Garde for tests by Otis Engineering Corp. [2]. The seals are for a 7-inch casing packer which Otis tested in 302C (575F) and 20.7 MPa (3000 psi) differential pressure water in their laboratory simulation tester. The elements were successfully sealing after 5 days when the test was terminated. The seal was in excellent condition considering the environment and did not show any signs of reversion softening or scission.

Prior to this test Otis tested an EPDM from another firm but at a lower temperature, 288C (550F), and the seal failed because it lost strength due to reversion softening of the polymer. The post mortem seal did not have any form and looked like a blob of tar. Otis has since tried several other competing EPDM compounds and none has yet equaled the performance of the Y267 EPDM.

In addition to the above tests, Otis also tested Y267 packer seals twice in sweet and once in sour crude. The severe sweet crude test was at 232C (450F) and 103 MPa (15,000 psi) for 6 days. The sour crude test went to even higher pressure. It was run at 232C (450F) for 3 days at 69 MPa (10,000 psi) and then the pressure was increased for 4.5 days to 138 MPa (20,000 psi). Otis reports that the seals were undamaged and just showed only very slight swell and softening. They did not expect any elastomeric seal to hold up at 138 MPa (20,000 psi). They conclude that the Y267 EPDM is a definite candidate for this extraordinarily extreme environment. [3]

BAKERSFIELD CONTINUOUS STEAM INJECTION

The Y267 EPDM element on an R&D Otis packer was fielded in a Bakersfield heavy oil continous steam injection well. The steam is 246C (475F) at the boiler and 204C (400F) at the wellhead.

The packer was pulled after 5 months of continuous steaming because the well required sand removal. Typically, difficulty is encountered in retrieving thermal packers but this packer was retrieved with no problems whatsoever, where usually difficulty is encountered with the seal bonding to the casing. or hardening in place.

The appearance of the recovered seal was excellent. It looked virtually as new after 5 months continuous operation and it was apparent that it could have remained in operation indefinitely. The good condition is particularly significant since the Y267 EPDM seal was in contact with crude oil which covered the packer and element when it was retrieved. EPDM's are generally considered inappropriate for hydrocarbon environments.

BACA, NEW MEXICO GEOTHERMAL HYDRAULIC STIMULATION

Union Geothermal/Republic Geothermal - LANL/DOE performed two separate hydraulic fracture experiments at the former Baca power plant demonstration site. The packer was set at about 915M (3000 ft.) where the formation is normally about 232C (450F). Two Otis packers with L'Garde Y267 EPDM elements were used, one for the fracture and then one for drill stem tests. This procedure was successfully performed in March and then again in October of 1981.

Normally for stimulations with temperatures above 149-177C (300-350F), Republic Geothermal takes special precautions to protect marginal seal elements made from standard elastomer compounds [4]. They first cool the well, circulate cold water while the packer is being run, leave the packer unset until the moment it is needed so that cold water circulation can be continued, and then just prior to pumping the packer is set.

The above procedure required for packers with standard elastomer compounds is unnecessary for geothermal packers with Y267 EPDM elements deployed in wells even above 260C (500F). Having to keep the packer in the balancing act through this crucial period just prior to pumping is eliminated since the packer can be set as soon as it is run in, checkout out for leakage, and then forgotten, thus freeing personnel to concentrate on other critical problems. In addition, if the standard elastomeric packers fail to pack-off just before pumping, expensive equipment must stand by until successful sealing is accomplished. Hypothetically, for the Baca case, if a standard elastomeric packer would have been run and if it had to be replaced, it would have cost about $18,000 in standby time.

The Baca well was cooled somewhat when the stimulation packer was run since cold water was circulated to assure that the well would remain killed. Hence, the maximum temperature the first stimulation packer experienced was 160C (320F) during the 12 hours it was packed off. The maximum pressure during stimulation was 21.2 MPa (3080 psi). The drill stem test packer only saw 171C (340F) and 4.1 MPa (600 psi) because of the cooling from 8000 barrels of fracture fluid and it remained packed off for 18.5 hours. Conditions for the second stimulation were about the same.

A total of 5 were retrieved, as with the Bakersfield packers, on the first attempt with no problems whatsoever, textbook retrievals. All packer seals looked as new which was expected at these temperatures since this is well below the Y267 EPDM capability. However, these same conditions are a challenge for standard elastomer compounds and Republic commented that even with precautions taken with standard elastomers under these same conditions, they would expect the post mortem seals to be extruded and/or cracked.

LOS ALAMOS NATIONAL LABORATORY (LANL) LAB TESTS

LANL is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock Project. They have shown feasibility of the concept at 200C (392F) and are currently preparing to demonstrate the concept with deeper wells and higher temperatures, 275C (525F) minimum. They had many elastomer problems at 200C and, hence, were especially concerned about elastomers for the hotter wells.

L'Garde fabricated O-rings and a cable bend protector from the Y267 EPDM packer seal compound for the LANL cablehead. LANL tested cablehead O-rings in an autoclave with water and Mobil One Oil [5]. They ran 24-hour cycles with the temperature and pressure on for 8 hours and off for 16, this simulates tripping in and out of the hole. The best prior performance they achieved was with commercial fluoroelastomer O-rings which literally disintegrated after one cycle.

LANL ran the Y267 EPDM in water for 5 cycles or 5 days before stopping the test to examine the seals. This included a 24-hour run on the fifth day which provided a total test time of about 56 hours for this one seal. The temperature was nominally 275C (525F) and the pressure was nominally 51.7 MPa (7500 psi). The O-ring looked excellent after this test and obviously could have continued further cycling.

They also ran the Y267 EPDM in Mobil One Oil for 4 cycles or 4 days and stopped the tests to examine the seals. The temperature was nominally 275C (525F) with a one hour excursion to 380C (716F) during one of the 24-hour cycles. LANL estimates that the seal was exposed to approximately 340C (644F). The pressure was nominally 51.7 MPa (7500 psi). The seals looked good and were sealing after 4 cycles, although there was some swelling from the high-temperature oil and some permanent deformation as would be expected for these conditions. They hope to go ten cycles operationally before they must change out the O-rings.

These tests are landmark tests because this is the first time the Y267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range, the first time the Y267 EPDM was tested in 100% oil, the first time the Y267 EPDM was tested in oil at extreme temperatures (excursions to 340C (644F), and the first time the Y267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDM's are generally not used in oil environments because they are extremely vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the Y267 EPDM is extremely serviceable in oil.

In addition to the cablehead tests, LANL also spent significant effort on cementing wiper plugs. [6] Poor cement jobs at Fenton Hill place question on the performance of prior elastomeric cementing wipers. Through an extended effort LANL was able to procure Dowell wipers made from Y267 EPDM.

As a matter of course LANL immersion tests wiper plugs in their autoclave. Typically they heat the autoclave to 280C (536F) at 17.2 MPa (2500 psi), hold it for 30 minutes, and then let it cool down. The autoclave and test specimen are at elevated temperature for about 12 hours. Changes in appearance and hardness are recorded.

LANL tested several wipers with different elastomers. All prior wipers showed some degradation in this test, with the elastomer on one disappearing completely. The Y267 wiper has been the exception; it was unaffected by the test. Post test it appeared as new, and the hardness did not change. Shore A hardness was measured at 10 different locations before and after the test.

FENTON HILL, NEW MEXICO ELASTOMERS IN WELL EE-2

Subsequent to the autoclave testing described in the previous section, LANL equipped their high-temperature cablehead with L'Garde Y267 EPDM O-rings and cable bend protector. The cablehead was run with a temperature probe into the new EE-2 well which is about 4600M (15,000 ft.) deep with a bottomhole temperature of 320C (608F). The longest service experienced by Y267 EPDM parts at this writing is bottomhole for 10 hours and above 200C (400F) for 15 hours. The parts sealed and looked excellent after this set of round trips with the most major problem being only some permanent deformation of the O-rings. LANL plans to try for as many as 10 trips on a single set of elastomeric parts when the opportunity avails itself. They have now converted the seals for all logging tools over to Y267 EPDM.

At this writing, one Y267 EPDM Dowell cementing wiper plug was run at Fenton Hill. A scab liner was cemented into EE-2 at 4481M (14,700 ft.) where the temperature is about 300C. Though it is impossible to deduce precisely how the Y267 EPDM performed in the depths of the well, it is known that a good cement job was achieved.

BINARY PLANT COMPATIBILITY LAB TESTS [7]

Because of the absence of adequate compatibility data of elastomers for hostile applications, significant expense is often incurred as each individual project attempts to find the right elastomer for its requirements by tril and error. Recognizing this the DOE/Brookhaven National Laboratory contracted L'Garde to run compatibility tests for elastomers for the binary power plant application.

A typical plant might be located in the Imperial Valley, in an area where the brine temperature is 191C (375F), and use isobutane/isopentane as the working fluids. In addition, there may be other equipment such as downhole electrical pumps which need a high temperature oil. Hence, static seal compatibility tests were run in synthetic brine, isobutane (represents isopentane also), and oil at nominally 191C (375F) with some testing up to 266C (510F). The oils tested are ASTM No. 1, ASTM No. 3, Chevron Cylinder 460X, and Pacer DHT-185M.

Given the test conditions, it was doubtful that any one compound would work satisfactorily in all three fluids. Nevertheless, this was highly desirable and the testing was structured to reveal this should any compound have that capability. Several of the molded rubber product manufacturers were solicited for their most promising compound(s) for the test. Ultimately 34 compounds from 15 different companies were selected for testing. A full spectrum of high performance polymers were included: EPDM, Kalrez, PNF, Viton/Fluorel, Nitrile/Buna N, and AFLAS.

Immersion testing in each of the three fluids were run to screen the 34 candidates down to 8 for further evaluation. The immersion tests were run at 191C (375F) for 5 days and evaluated on the basis of change in hardness, ultimate tensile strength, ultimate elongation, weight, volume, and resilience. This screening down process was done with the keen awareness that immersion only tests the effect of temperature and chemistry on the elastomer. At these higher temperatures, the mechanical environment due to differential pressures, seal setting, etc., are just as important, if not moreso, than temperature and chemistry. Not surprisingly, what looked the best based on immersion tests did not look the best after full-environment static seal tests.

After selection, the 8 compounds were then further evaluated. They were immersion tested at 191C (375F) for longer periods, and tested as O-rings up to 266C (510F). Based on these tests 4 compounds were O-ring tested at 204C (400F) and 20.7 MPa (3000 psi) for 6 months. The overall summary matrix of tests is shown in Table I.

TABLE I. SUMMARY MATRIX
BINARY POWER PLANT COMPATIBILITY TESTING

Number Tested	Immersion (6 Fluids)			O-ring (3 Fluids)	
	5d	2 mos.	6 mos.	2d	6 mos.
34 Compounds					
191C (375F)	X				
8 Selected					
191C (375F)		X	X	X	
232C (450F)				X	
266C (510F)				X	
4 Selected					
204C (400F)					X

Figure 1 summarizes the short-term O-ring tests. All testing was done at 20.7 MPa (3000 psi) differential pressure and Figure 1 shows only the EPDM's performing reliably up to the 266C (510F) level. Catastrophic failure occurred with many of the compounds especially in isobutane and brine. The AFLAS 7170X14 and Kalrez 1018 both catastrophically failed at the minimum test temperature, 191C (375F), in isobutane. The E692-75 EPDM failed in isobutane, hence, does not show across the board reliability that Y267 EPDM shows. This compound performed quite well in general; however, results indicate that its capability is being approached for the more severe conditions. Similar static O-ring tests in 260C (500F) brine reported in Reference 1, indicate nibbling of the E692-75 in brine at 28.3 MPa (4100 psi). Hence, these conditions are about the upper limit for the E692-75 while the capability of the Y267 EPDM is indicated to be somewhat higher since it has not experienced breakage or nibbling.

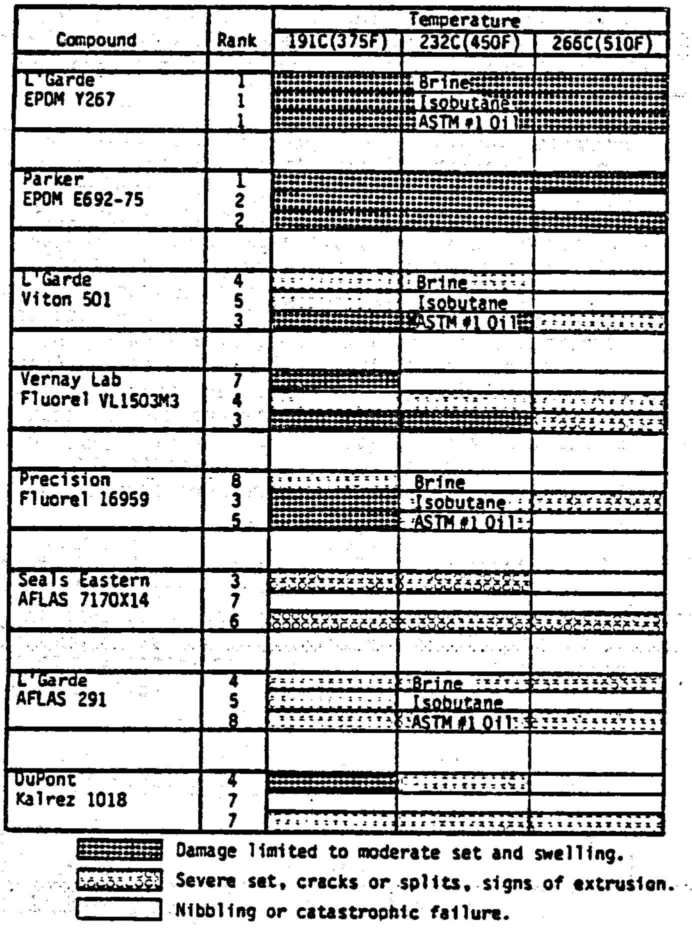

FIGURE 1. 46-HOUR O-RING TEST RESULTS, 20.7 MPa (3000 PSI) ΔP

Figure 2 summarizes the long-term O-ring test which complete the compatibility test program. The tests were run for approximately 6 months at 204C (400F) and 20.7 MPa (3000 psi) differential pressure. The long-term tests were run on a different fixture than the short-term tests. The short-term tests were strictly static seal tests while the long-term tests permitted slow sliding, up to a cycle per day. This design was adopted strictly because of economic considerations. The four best compounds from the short-term O-ring tests were tested. Figure 2 shows a relative ranking of compounds by shading and their life for each of the three fluids. Catastrophic failure occurred in isobutane and brine, again the most difficult fluids to seal.

In brine the Y267 looks the best which is not surprising because it was designed for brine. The Parker E692-75 EPDM test fixture failed after 56 days because a non-test O-ring in the fixture failed, but based on previous data there is every reason to believe that it would have performed equally at this relatively mild temperature. Both the fluoroelastomers became brittle and failed. In isobutane, both E692-75 EPDM and 501 Viton are "not OK"; they failed at 90 and 41 days respectively

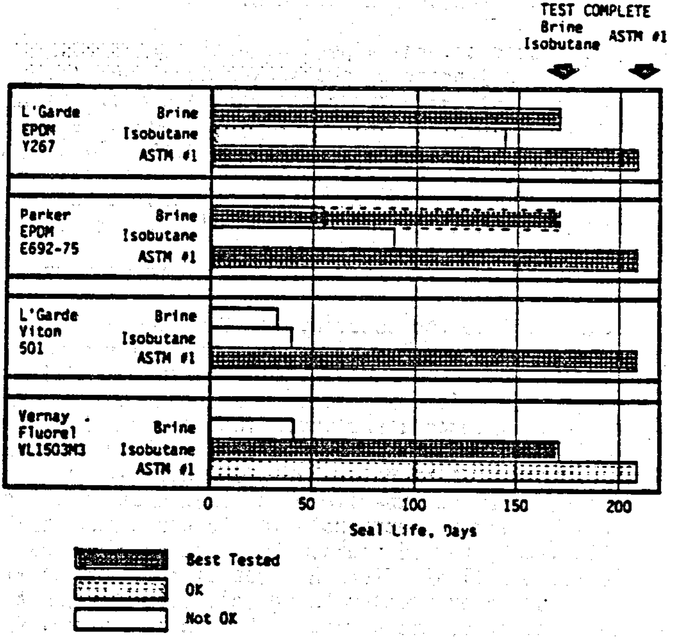

FIGURE 2. 6-MONTHS O-RING TEST RESULTS, 204C (400F), 20.7 MPa (3000 PSI) ΔP

consistent with previous tests. There is significant uncertainty, however, as to Y267 EPDM and VL1503M3 Fluorel. For the conditions VL1503M3 is best because it survived the entire test, 171 days, but suffered moderate permanent set, while the Y267 suffered slight permanent set and it cracked over about 10% of its circumference but stayed intact. The Y267 crack may have originated with a molding defect and, in any event, it could probably be prevented with a backup ring. Consequently in the final analysis, these and other factors such as availability would enter into the final selection of isobutane. In ASTM No. 1 oil a broader choice exists as all four candidates survived the 209-day test period. The VL1503M3 Fluorel became brittle, suffered severe set, and slight nibbling. The EPDMs suffered some swell and moderate nibbling. The 501 Viton suffered severe set and slight nibbling, however, it did not get brittle as did the VL1503M3 or as it did in Chevron Cylinder Oil. A trade exists here between severe set and potential of getting brittle for the 501 Viton, against the moderate nibbling of the EPDMs which can be corrected with backup rings.

In overall conclusion for the 46-Hour O-ring Test conditions, the Y267 EPDM looks the best for brine, isobutane, and ASTM No. 1 oil. The Parker E692-75 looks as good for brine and almost as good for ASTM No. 1 oil, however, other test data indicate that it is performing near its maximum capacity under these test conditions while Y267 EPDM has some additional capacity. For the 6-months O-ring test conditions, the same conclusion applies as for the 46-hour test for brine. However, for isobutane, there is a trade between Y267 EPDM and VL1503M3 Fluorel. In ASTM No. 1 oil, there is a trade between the EPDMs and 501 Viton. The 501 suffered severe set and gets brittle in Chevron

Cylinder Oil while the EPDMs suffered moderate nibbling which can be prevented with a backup ring. The comment on the 46-Hour O-ring Test regarding Y267 and E692-75 capacities also applies here.

The above conclusions regarding the serviceability of EPDMs in hydrocarbon oil is unexpected because it is a generally accepted rule that EPDMs should not be used with hydrocarbon oil. This rule is derived from immersion tests and even though the Y267 is less vulnerable to oil than most EPDMs, it does swell, and lose strength and hardness. Nevertheless, after several days in the environment the degradation reaches a plateau which has been measured to exist to at least 6 months and the remaining properties still provide an excellent static seal. There is some evidence, in fact, that swell may be a desirable property for seals in hostile environments.

OTHER MISCELLANEOUS TESTS

Other miscellaneous tests on Y267 EPDM have been run for a variety of environments and by several organizations. These are summarized in this section.

Battelle Northwest [8] ran into rubber seal problems on an inline corrosion probe they are testing. They ran autoclave tests in 200C (400F) brine on the Y267 EPDM prior to installing it in the field. They have tested several compounds and confirm that the Y267 EPDM stands up better than any other elastomer they have tested. They subsequently ran excellent comparative tests at the Magma Plant in the Imperial Valley. A test loop is available at the feed to the plant where several instruments can be tested simultaneously. Battelle ran corrosion probes with fluoroelastomer (FKM) perfluoroelastomer (FFKM), and Y267 EPDM seals side-by-side. Both other seals failed within 2 weeks; the FKM became brittle, and the FFKM cracked and split. The Y267 EPDM was still running after 8 months when the plant was shut down. This is the longest performance logged for this application for any elastomeric seal.

LANL/Union Oil [9] ran an explosive stimulation experiment at the Geysers. They encountered seal problems during preliminary hot water tests with their timer/detonator vessel which is immersed in the explosive during operation. Immersion tests were subsequently run in the explosive at 246C (475F) and 6.9 MPa (1000 psi) with four different elastomer compounds and the Y267 EPDM was clearly the best performer. Tests were also successfully rerun in 260C (500F) water in an autoclave. Y267 EPDM seals were ultimately installed in both pressure vessels; the timer/detonator, and the overall container. The timer was set for 48 hours for the downhole tests and the detonation occurred at the second implying that the seals must have performed flawlessly.

LANL [10] ran some temperature surveys for Union at the Baca using their cablehead equipped with Y267 EPDM seals. The well temperature was approximately 300C (575F) and the Y267 EPDM parts were exposed for a maximum of 4 to 6 hours. The rubber parts were changed whenever the cablehead was dismanteled for other reasons.

Sandia [11] ran autoclave tests in the vapor phase of water at 270C (500F) for 100 hours plus heat-up and cool-down. The cable BOP environment was being stimulated. The Y267 EPDM was the best of 7 compounds in this environment. It retained about 90% of its tensile strength after ageing and was recommended by Sandia for this application.

Sperry Vickers [12] ran long-term compatibility tests of elastomers in an aqueous solution of hydrazine at 218C (425F). They saw no change in the Y267 EPDM after 85 days.

TerraTek [13] ran drill bit seal tests on several elastomers including Y267 EPDM. The test exposes the seal to grease on one side and rock cuttings, sand, and water on the other. The specimen is raised to a presoak temperature to simulate tripping into the well, cooled to 150C (302F) to simulate introduction of cold drilling fluid, and then the dynamic test is initiated. The test provides a rotating motion and on each cycle radial and longitudinal jogs are superimposed on the motion. At the time they ran Y267 EPDM, a 40 to 50 hour run was quite respectable. With the Y267 a presoak temperature of 288C (550F) was chosen; this was a first for this high a test temperature. Both TerraTek and L'Garde were pleasantly surprised when the Y267 EPDM was still sealing after 104 hours. TerraTek remarked that the Y267 appeared excellent after the test, less degraded than all other elastomers tested.

AVAILABILITY OF Y267 EPDM

A major objective of the DOE effort was to assure that the developed technology is available for widespread field use. To this end, L'Garde was contracted to transfer the technology. After an application and careful evaluation process, BJ-Hughes Rubber, Oncor/Precision Rubber, and Parker Seal were selected for transfer. The transfer is now complete and molded parts based on Y267 EPDM technology are available in commercial quantities from BJ-Hughes Rubber, Parker Seal, and Precision Rubber. The full spectrum of large and small parts are available from these suppliers. In addition, the technology is available for R&D efforts directly from L'Garde.

CONCLUSION

Downhole and laboratory trials of L'Garde's Y267 EPDM were successful. Table II summarizes those 17 experiences and they indicate that Y267 EPDM is the superior elastomer for high-temperature aqueous reducing environments. Test data is also building which indicates superiority for high-temperature hydrocarbon reducing environments.

Parts based on Y267 EPDM technology are available in commercial quantities from:

BJ-Hughes Rubber
Parker Seal
Precision Rubber

Y267 EPDM technology, hostile environment seal technology, and hostile environment testing technology is available from L'Garde.

TABLE II. SUMMARY OF Y267 EPDM LABORATORY AND FIELD EXPERIENCE

Test	Fluid	Temperature C(F)	Differential Pressure MPa (psi)	Time		
				Hours	Days	Months
Otis Lab. Packer	H_2O	302 (575)	20.7 (3000)		1	5
Otis Lab. Packer	Sweet & Sour Crude	232 (450)	138 (20,000)	108	4.5	
Bakersfield, Packer	H_2O/Crude	204 (400)	1.7 (250)		150	
Baca Stim. Packer (2x)	Frac.	160 (320)	21.2 (3080)	12	0.5	
Baca DST. Packer (3x)	Frac/H_2O	171 (340)	4.1 (600)	18.5	.8	
LANL Lab. O-ring	H_2O/Oil	275 (525)	51.7 (7500)	40	1.7	
LANL Lab. Cement Wiper	H_2O	280 (536)	0 (0)	.5+	.02+	
Fenton Hill, O-ring	H_2O	317 (603)	41.4 (6000)	10	0.4	
Fenton Hill, Cement Wiper	H_2O/Cement	300 (572)	0 (0)			
L'Garde Lab. O-ring	Brine,Oil,Isobutane	266 (510)	20.7 (3000)	48	2	
L'Garde Lab. O-ring	Brine,Oil,Isobutane	204 (400)	20.7 (3000)		171+	5.7+
Battelle Lab	Brine	200 (392)	4.1 (600)		4	
Magma, Seal	Brine	180 (356)	N/A		180+	6+
Union/Physics Int'l Lab	Explosive	246 (475)	6.9 (1000)	24	1	
Baca, Log O-ring	H_2O	300 (575)	11.7 (1700)	6	.3	
Sandia Lab	H_2O	260 (500)	4.7 (680)	100	4.2	
Sperry Vickers Lab	H_2O/Hydrazine	218 (425)	2.2 (325)	86	3.6	
TerraTek Lab, O-ring	H_2O/Debris,Grease	288 (550)	1.4 (0/200)	104	4.3	

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Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory and Dr. J. C. Rowley of Los Alamos National Laboratory were especially helpful.

REFERENCES

[1] A. R. Hirasuna, et al., July 1979, "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976 - 30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy.

[2] D. Taylor, Private Communication, Otis Engineering Corp., Dallas, TX.

[3] J. Bostock, Otis Test Report Number 1395-31982, dated 19 March 1982.

[4] R. Verity, Private Communication, Republic Geothermal, Santa Fe Springs, CA.

[5] J. Archuleta and B. Dennis, Private Communication, Los Alamos National Laboratory, Los Alamos, NM.

[6] R. Pettitt, J. Rowley, Private Communication, Los Alamos National Laboratory, Los Alamos, NM.

[7] G. J. Friese, "Elastomer Compatibility With Brine, Isobutane, and Oil," 1981 Annual Meeting, Geothermal Resources Council, 25-29 October 1981, Houston, TX.

[8] D, Shannon, M. Danielson, Private Communication, Battelle Northwest, Richland, WN.

[9] C. Allen, Private Communication, Union Oil Research, Brea, CA.

[10] B. Dennis, B. Todd, Private Communication, Los Alamos National Laboratory, Los Alamos, NM.

[11] C. Arnold, Jr., 19 August 1979, Memo to Distribution, Sandia National Laboratories, Albuquerque, NM.

[12] D. Nowicki, Private Communication, Sperry Vickers, Jackson, MS.

[13] R. Hendrickson, R. Winzenreid, Private Communication, TerraTek, Salt Lake City.

ELASTOMERS FOR SERVICE IN DEEP WELL ENVIRONMENTS

L.R. Logsdon and W.C. Long

BJ-HUGHES INC.
7355 E. Slauson Ave.
Commerce, CA 90040

For the last two decades the rubber division of BJ-Hughes, has been in the forefront of rubber technology as applied to products for the oil-well drilling industry.

We welcomed the opportunities presented when we were selected to participate in the GEM program and extend our appreciation to L'Garde Inc., Department of Energy and Radian Corporation.

Our first efforts, approximately 15 months ago were to duplicate the four compounds recommended by L'Garde and verify our ability to obtain equal physical properties. After some minor problems in obtaining exact duplication of the chemicals, we did succeed in matching the properties obtained by L'Garde.

Aflas, a copolymer of teflon and propylene manufactured in Japan by Asahi Glass and representedby Xenox, Inc., is the basis elastomer for GEM formulation 291.

Advantages of Aflas are high temperature resistance up to 550°F. (Short term), resistance to H_2S and amine environment, acids, basis, many solvents and hydrocarbons. It also has excellent resistance to steam and will not hydrolize when exposed to high temperature and high pressure steam. It has excellent physical properties including long term compression-set resistance at high temperature.

We have submitted two products manufactured from GEM 291 to customers for evaluation. One was an accumulator bladder for a hydraulic pumping system with hot oil on one side and nitrogen on the other. However, our customer reported that its service life was no better than they experienced with epichlorhydrin.

The second application was an "O" ring for sealing a bearing in a drill bit. Compound 291 was one of a group of compounds and did not perform as well as several of the others. At the present time we are not planning any further investigation of this compound unless some application would seem to require Aflas over other elastomers.

Viton, which is a DuPont fluoroelastomer, forms the basis for GEM formulation 255. Although the particular viton used in the original work is no longer available, a currently available material is equal or better in all respects. Most applications of Viton involve a sealing function of some sort. Lasting sealability when exposed to high heat, pressure, and aggressive chemicals or hydrocarbons is the strong point of Viton. Compared to the earlier compound, 255 shows superior resistance to oils and organic fluids and very good resistance to hot water, steam and aqueous acids. We did extensive work with the compound, evaluating various modifications and adjusting for several possible applications. Sample products submitted to customer for evaluation include packers, stabilizers, O'rings and bladders. We have also submitted compression-set buttons and tensile slabs at prospective users request. Of these, the stabilizers and O'rings were outperformed by other materials, while results with the packers and bladders have not been completed.

We feel 255 is an excellent compound and that along with our variations, these Viton materials will be very useful in future products.

GEM formulation 266 (a blend of Viton and EPDM) was also the basis of extensive investigation for us. We checked many variations, including DuPonts' recommendations for such blends and in general, we feel such compounds are a promising compromise for some difficult applications. So far we have submitted drill-bit O'rings which did not prove resistant enough to abrasion; and stabilizers, which are still to be evaluated.

GEM formulation 267, an EPDM compound, has evoked more interest than all the others put together.

Let's consider each of the L'Garde formulas and the comparison in physical properties as processed by both companies.

VITON
FORMULATION GEM 255

	PHR
VTR 5362 (now Viton GF)	100.00
Austin Black	10.00
N568 Black	25.00
Maglite K	3.00
Diak #7	1.50
Luperco 130 XL	1.50
	141.00

Logsdon and Long

Press cure 45'/350°F	BJ-HUGHES	L'GARDE
Durometer, Shore A	91	90
Tensil Strength, PSI	2870	2680
Ultimate Elongation, %	130	105
Tear Strength, Die C, PPI	230	-

VITON - EPDM BLEND
FORMULATION GEM 266

	PHR
Nordel 1660	50.00
VTR 5362 (Viton GF)	50.00
Hypalon 20	2.50
Austin Black	7.50
Maglite K	2.50
N110 Carbon Black	52.50
Antioxidant 2246	0.25
Thermogard S	2.50
Diak #7	3.00
Luperco 130XL	1.50
	172.25

Press cure 60'/350°F	BJ-HUGHES	L'GARDE
Durometer, Shore A	94	92
Tensile strength, PSI	2360	2290
Ultimate Elongation, %	130	130
Tear Strength, Die C, PPI	190	-

EPDM
FORMULATION GEM Y267

	PHR
Nordel 1660	100.00
Hypalon 20	5.00
Thermogard S	5.00
N110 Black	75.00
A.O. 2246	0.50
Polyscience #6081 (HYSTIL-B-3000)	20.00
Dicup R	3.50
	209.00

Press cure: 30'/350°F Post-cure: 1 HR/400°F, 1 HR/400°F, 1 HR/500°F, 5 HR/550°F

	BJ-HUGHES	L'GARDE
Durometer, Shore A	90	91
Tensile Strength, PSI	2280	2170
Ultimate Elongation, %	160	170
Tear Strength, Die C, PPI	240	260

AFLAS
FORMULATION GEM 291

	PHR
Aflas 100	100.00
N110 Black	50.00
Maglite K	3.00
Polybutadiene #608 (Hyptil-B-3000)	5.00
Diak #7	5.00
Luperco 130 XL	5.00
	168.00

Press cure: 20'/350°F Post-cure 4.5 HR/350°F, 0.5 HR/400°F, 0.5 HR/450°F, 2.5 HR/500°F

	BJ-HUGHES	L'GARDE
Durometer, Shore A	96	95
Tensile strength, PSI	2640	2330
Ultimate Elongation, %	120	130
Tear Strength, Die C, PPI	305	-

Our company feels that since the program was conducted under the auspices of the Department, and with the need for energy which we face in this nation today, we wish to disclose all the pertinent facts concerning our part in the transferee phase, as long as the information is not proprietory.

We would, therefore, like to disclose our experiences in working with the L'Garde formulas.

A. Processing at BJ-HUGHES

1. The Viton formula processed well and since we had developed similar compounds, the experiences already learned were transferable.
    a. Mixing can be optimized by making sure that the internal mixer temperature is above 150°F. We made several "Dust" batches in our Lab Banbury prior to discovering this.
    b. Milling, extruding, or barwelling of the compound was normal when preparing the material for molding.
    c. Molding procedures were those usually used with any Viton formulation. We have compression and transfer molded it, and encountered no surprises.

2. The Viton-EPDM blend was similar to the Viton, except that we did find processing to be improved over the completely Viton formula.
    a. Mixing was easier with the blend and preheating of the Laboratory Banbury was found to be unnecessary. The EPDM apparently allowed the polymers to accept the cold carbon black more readily.
    b. Milling, extruding, or barwelling the blend was easier, probably because of of the reduced compound viscosity.
    c. The compound flowed well during molding; whether we were compression or transfer molding. Chemically bonding to steel was also no problem after we found the proper adhesives.

3. The L'Garde Aflas compound can be used and/or blends with other polymers. We again found that preheating of internal mixers is helpful.
    a. The mixing was similar to that of any fluorocarbon polymer.
    b. Milling is the only technique which we have used in preparing the compound for molding.
    c. Molding presented no problems, and we have both compression-molded and transfer-molded parts.

4. The EPDM formula (Y267) processes well in most respects, but there are certain techniques which we would recommend.
    a. "Up-side-down" mixing appears to be better than "right-side-up" mixing. We feared that we might form carbon black agglomerates when mixing "up-side-down", but this doesn't seem to be true; infact, we have obtained slightly higher original original properties with this method over the conventional method.
    b. This compound can be prepared for molding by milling, extruding or barwelling, but care should be exercised that no voids (air-pockets) are left in the 'preparation'; especially when it is to be used in compression molding. Some incidents of 'non-knitting have been experienced.
    c. The molding qualities are quite good, but there are some precautions to be taken. We found that in transfer molding, the size and number of sprues and bleeders must be determined for each different part. In compression molding, an adequate amount of compound must be used to obtain good "packing density"; especially when molding "O" rings or flat washers.
    d. The post-curing of Y267 is of great importance. The times and temperatures used-determine, in large part, the final physical properties and part-life of the final product.

B. Testing at BJ-HUGHES.

HIGH TEMPERATURE TESTING OF EPDM
ROOM TEMPERATURE PHYSICAL PROPERTIES

(Y267)	N22-2	N25-6
DUROMETER	89	91
TENSILE	2180	2650
ELONGATION	150	180
100% MODULUS	720	1220

PHYSICALS @ 347°F

COMPOUND	N22-2	N25-6
DUROMETER	87	88
TENSILE, PSI	610	790

Lodsdon and Long

PHYSICALS @ 482°F		
COMPOUND	N22-2	N25-6
DUR @ 77°F	87	90
DUR @ 482°F	78	81
TENSILE, PSI	540	585

RUBBER-TO-METAL BONDING AT 350°F

DATE 9-14-82
USE Various
PROJECT GEM

Rubber section: .075" X 1" X 6"
Steel section: .062" x 1" X 6"

ADHESIVE SYSTEM	SPECIMEN	COMPOUND	CURE	TYPE OF FAILURE (%) RUBBER	CEMENT	METAL	PULL (LB/IN)	REMARKS
G	1.	N22-2	20'/325°F	100	-	-	12.5	Bond stronger than rubber
	2.			100	-	-	13.	Bond stronger than Rubber
	3.			100	-	-	35.	Bond stronger than Rubber
Y	1.	N25-1	30'/325°F	100	-	-	56.	Bond stronger than Rubber
	2.			100	-	-	55.	Bond stronger than Rubber
	3.			100	-	-	55.5	Bond stronger than Rubber
Z	1.	N25-1	30'/325°F	100	-	-	33.	Bond stronger than Rubber
	2.			100	-	-	26.	Bond stronger than Rubber
	3.			100	-	-	38.	Bond stronger than Rubber
AA	1.	N25-1	30'/325°F	100	-	-	47.5	Bond stronger than Rubber
	2.			100	-	-	40.	Bond stronger than Rubber
	3.			100	-	-	70.	Bond stronger than Rubber

RUBBER-TO-METAL BONDING AT 482°F

DATE 9-15-82
USE Various
PROJECT GEM

Rubber section: .075" X 1" X 6"
Steel section: .062" X 1" X 6"

ADHESIVE SYSTEM	SPECIMEN	COMPOUND	CURE	TYPE OF FAILURE (%) RUBBER	CEMENT	METAL	PULL (LB/IN)	REMARKS
G	1.	N22-2	30'/325°F	100	-	-	8.	Bond stronger than Rubber
	2.			100	-	-	7.5	Bond stronger than Rubber
Y	1.	N25-1	30'/325°F	100	-	-	45.	Bond stronger than Rubber
	2.			100	-	-	45.	Bond stronger than Rubber
Z	1.	N25-1	30'/325°F	100	-	-	6.	Bond stronger than Rubber
	2.			100	-	-	30.	Bond stronger than Rubber
	3.			100	-	-	8.5	Bond stronger than Rubber
T	1.	N22-2	30'/325°F	100	-	-	5.	Bond stronger than Rubber
	2.			100	-	-	3.	Bond stronger than Rubber
	3.			100	-	-	3.	Bond stronger than Rubber

ADHESIVE SYSTEM	SPECIMEN	COMPOUND	CURE	TYPE OF FAILURE (%)			PULL (LB/IN)	REMARKS
				RUBBER	CEMENT	METAL		
AA	1.	N25-1	30'/325°F	100	-	-	27.	Bond stronger than Rubber
	2.			100	-	-	26.5	Bond stronger than Rubber
	3.			100	-	-	34.5	Bond stronger than Rubber

C. Laboratory Testing by B.J. Hughes' Customers.
   1. There has been very little reporting by our customers as to their data.

D. Field tests by B.J. Hughes customers.
   1. Our customers have been periodically testing parts manufactured by us for approximately a year. In most cases, these tests have been positive and the parts did the job for which they were intended. We have also experienced some failures.
      a. Customer "A" used a packer made with Y267 compound in a deep geothermal well, and it outperformed all of the other parts which they tested (90).
      b. Customer "A" Lab-tested another type of packer which was also made with Y267, and our part failed to maintain a seal before the desired time of test was reached. A second test was run, and this time our part passed the test. New parts are now at the well-site awaiting "down-hole" testing.
      c. Customer "B" has ordered "O" rings made with Viton, a Viton-EPDM blend, and EPDM. All parts are being tested.
      d. Customer "C" has received cured ASTM slabs and uncured compounds of both Viton and EPDM. Testing is in progress.
      e. Customer "D" ordered and received Viton bladders and tests are being run.
      f. Customer "E" has received ASTM slabs and compression-set buttons made with Viton and EPDM compounds. Testing is being done.
      g. Customer "F" ordered EPDM and Viton-EPDM blend stabilizers. They are still being tested.
      h. Customer "G" has received a solid block, packers and chevron seals - all made with the EPDM compound. Testing has just begun.

E. General Comments
   1. We feel that the technology initiated by L'Garde for elastomeric materials in deep-well environments has been very worth-while for the well-drilling industry.
   2. The interest in, and use of this technology is escalating, as you can see from the varied types of parts that customers are requesting.
   3. Our company is more than willing to attempt the manufacture of large, small, custom, or any reasonably shaped parts and invite customer's inquiries.

IMPROVED ENERGY SEALING CAPABILITY

Jerry L. Barsoumian

Parker Hannifin Corp. - Seal Group
10567 Jefferson Boulevard
Culver City, California 90230

ABSTRACT

In response to the need for tapping national energy resources, an improved high temperature sealing material has been developed through the sponsorship of the Department of Energy. Parker Seal was selected as one of the technology transferees from L'Garde Inc. and has optimized this transferred technology for further improved performance capabilities and acceptable plant processing. This paper summarizes Parker Seal's testing and evaluation efforts on L'Garde's Y267 transferred technology for a new geothermal and steam service material. This new product, Parker's E962-85 is described in this paper.

INTRODUCTION

In an effort to tap the earth's high temperature geoenergy resources more readily, it is imperative to develop increased sealing capabilities for dealing with these hostile chemical, pressure, and temperature conditions.

Seal material developed in the past for well logging, drilling, and oil production applications have become technically deficient in handling the increased performance requirements introduced by deeper and more varied drilling needs. Today, it is not uncommon to encounter geothermal well temperatures ranging as high as 350°C.

By proper design of sealing materials, seal shapes and seal systems, it is becoming possible to consider elastomeric seal service at these extreme temperatures and pressures.

PHYSICAL PROFILES

Additional research and development efforts were made to the transferred technology in order to improve plant processing, reduce mold fouling and improve properties. Improvements were achieved in original physical properties, properties in steam including compression set and extrusion resistance in water using customer fixture. Refer to Table I for comparative test data.

When tested in various other media relating to service in geothermal and oil field applications, E962-85 material exhibits outstanding physical and chemical properties.

In steam at 550°F for 168 hours, this material shows little loss in properties. At 600°F after 168 hours, it retains its overall physical integrity but with relatively significant changes in hardness, tensile, and compression set.

In oil contaminated steam at 550°F after 168 hours, the oil contributes to an accelerated rate of deterioration and yet the overall profile would still be acceptable in a number of sealing applications. Refer to Table II for test data.

Test data in Table III shows resistance to ASTM #1 oil, ASTM #3 oil, diesel #2 , NACE B amine inhibitor with diesel #2 as the diluent and indicates in a similar way that E962-85 material retains its overall physical integrity. The high swell can be capitalized upon in several static sealing applications due to the positive seal force exerted by the sealing surface on the gland.

After 70 hours at 158°F in a 28% hydrochloric acid medium, it exhibits a good profile as a sealing material. Hydrochloric acid conditions are often encountered in hostile environments in oil wells. These acid conditions are often neutralized by amine inhibitors. E962-85 showed a very good resistance in water based amine inhibitor NACE A fluid.

APPLICATIONS

A sealing package including E962-85 was able to successfully seal under the following conditions: The package consisted of an E962-85 V-ring and Parker high temperature experimental thermoplastic material XP2589-2 for backup.

a. 15,000 psi/500°F in diesel #1 for 70 hrs.
b. 15,000 psi/550°F in water for 70 hours

The V-ring test fixture is set in a static pressure arrangement with a floating gap of .012".

Some of the current applications and field testing evaluations underway by customers, both nationally and internationally, include E962-85 seals for workover blow out preventor (BOP) hydraulic systems, seals used for monitoring and logging tools used in the induction and drilling phase of geothermal wells, and seals for pressure compensator systems in drill bits. In addition, evaluations are underway for a seal nipple packing encountered in steam stimulated, huff and puff applications. In each case mentioned above, several sizes are typically required to seal the system with seal configurations using O-rings, V-rings, and other packings.

Feedback from two major U. S. firms indicates Parker E962-85 is the top qualifier based on their testing and evaluation of competitive sealing products.

Parker Seal field testing and evaluations with E962-85 is now widespread, with the participation of major U.S. oil and other related companies active in both geothermal and oil business.

Current international activities include evaluations of Parker E962-85 in Iceland, The Philippines, Venezuela, Japan, France, and Sweden.

CONCLUSION

Parker E962-85 is an optimized formulation based on L'Garde Y267 transferred technology which has improved processing characteristics and physical profile.

It has been shown to perform satisfactorily up to 550°F and 15,000 psi in a variety of media. Parker E962-85 is currently being evaluated and sold to oil and geothermal related companies throughout the world.

ACKNOWLEDGEMENTS

Parker Seal extends its appreciation to L'Garde for selecting Parker Seal as one of the technology transferees. Furthermore, we thank the technical personnel at L'Garde for their extensive and responsive efforts leading to this successful program.

In addition, Parker Seal appreciates the cooperation by all the companies who have and are participating in this effort.

TABLE I

	Y267	E962-85
Original Physical Properties (2-214)		
Hardness, Type A, pts.	88	86
Tensile Strength, psi	1680	1930
Elongation, %	136	135
Modulus @ 100%, psi	1220	1590
Specific Gravity	1.10	1.11
Aging in Steam (2-214) 70 Hours @ 200°C (392°F)		
Hardness, Type A, pts. (Chg., pts.)	88(0)	86(0)
Tensile Strength, psi (Chg., %)	1400(-17)	2000(+4)
Elongation, % (Chg., %)	108(-21)	121(-10)
Modulus @ 100%, psi (Chg., %)	1290(+6)	1570(-1)
Volume Change, %	+4.1	+4.3
Compression St, %	51.4	44.7
Extrusion Test (2-336) H_2O @ 350°F Eccentric Gap, 0.015"		
Pressure to rupture, psi	19,000	23,000

TABLE II

	E962-85 2-214
Original Physical Properties	
Hardness, Type A, pts.	87
Tensile Strength, psi	2150
Elongation, %	96
Modulus @100%, psi.	--
Aging in Steam **168 Hours @288°C (550°F)**	
Hardness, Type A, pts. (Chg., pts.)	82(-5)
Tensile Strength, psi (Chg., %)	1740(-19)
Elongation, % (Chg., %)	102(+6)
Modulus @100%, psi (Chg., %)	1710 --
Volume Change, %	+2.1
Compression Set, %	46.7
Aging in Steam **168 Hours @316°C (600°F)**	
Hardness, Type A, pts. (Chg., pts.)	65(-22)
Tensile Strength, psi (Chg., %)	506(-76)
Elongation, % (Chg., %)	109(+14)
Modulus @100%, psi (Chg., %)	466 --
Volume Change, %	+4.0
Compression Set, %	85.7
Aging in Steam with 10% MIL-H-5606D Oil **168 Hours @288°C (550°F)**	
Hardness, Type A, pts. (Chg., pts.)	72(-15)
Tensile Strength, psi (Chg., %)	1030(-52)
Elongation, % (Chg., %)	98(+2)
Modulus @100%, psi (Chg., %)	--
Volume Change, %	+16.6
Compression Set, %	76.5

TABLE III

	E962-85
Original Physical Properties	
Hardness, Type A, pts.	87
Tensile Strength, psi	1960
Elongation, %	101
Modulus @ 100%, psi	1900
Aging in 28% HCL - 72% H_2O 70 Hours @ 158°F	
Hardness, Type A, pts. (Chg., pts.)	87(0)
Tensile Strength, psi (Chg., %)	2330(+19)
Elongation, % (Chg., %)	112(+11)
Modulus @ 100%, psi (Chg., %)	2050(+8)
Volume Change, %	+0.9
Compression Set, %	28.0
Aging in ASTM #1 70 Hours @ 350°F	
Hardness, Type A, pts. (Chg., pts.)	76(-11)
Tensile Strength, psi (Chg., %)	1520(-22)
Elongation, % (Chg., %)	114(+13)
Modulus @ 100%, psi (Chg., %)	1200(-37)
Volume Change, %	+56.3
Compression Set, %	20.6
Aging in ASTM #3 70 Hours @ 350°F	
Hardness, Type A, pts. (Chg., pts.)	76(-11)
Tensile Strength, psi (Chg., %)	1090(-44)
Elongation, % (Chg., %)	94(-7)
Modulus @ 100%, psi (Chg., %)	--
Volume Change, %	+86.2
Compression Set, %	2.9
Aging in Diesel #2 (Texaco) 70 Hours @ 350°F	
Hardness, Type A, pts. (Chg., pts.)	74(-13)
Tensile Strength, psi (Chg., %)	656(-67)
Elongation, % (Chg., %)	74(-27)
Modulus @ 100%, psi (Chg., %)	--
Volume Change, %	+90.8
Compression Set, %	0.0
Aging in NACE A 5% + 95% H_2O 168 Hours @ 212°F	
Hardness, Type A, pts. (Chg., pts.)	83(-4)
Tensile Strength, psi (Chg., %)	2070(+6)
Elongation, % (Chg., %)	129(+28)
Modulus @ 100%, psi (Chg., %)	1470
Volume Change, %	+9.6
Compression Set, %	16.2
Aging in NACE B 5% + 95% Diesel #2 168 Hours @ 212 °F	
Hardness, Type A, pts. (Chg., pts.)	75(-12)
Tensile Strength, psi (Chg., %)	796(-59)
Elongation, % (Chg., %)	85(-16)
Modulus @ 100%, psi (Chg., %)	--
Volume Change, %	+98.6
Compression Set, %	0.0

A FABRICATION REPORT ON L'GARDE Y267 GEOTHERMAL COMPOUND

Howard Gillette

Precision Rubber Products Corporation

Abstract

A technology transfer was made from L'Garde to Precision Rubber Products for the fabrication of L'Garde geothermal seal compound Y267. Precision Rubber reports their experience as fabricators and gives field reports from three of their customers. Most of the experience has been very successful.

Introduction

In January, 1980, we at Precision entered the L'Garde program and began setting up the equipment and procedures necessary in the transfer of this technology.

There are several unique features in this program. One of them is the curing of the final product in an atmosphere of nitrogen. This of course, required a special oven.

We designed an oven from a 13 inch section of aluminum pipe, put ends on it, heating bands, and heavily insulated it. With this fairly simple arrangement we pass nitrogen through at 30 Std. CU. FT. per hour.

The other unusual aspect of the program is the formula which combines the toughest ethylene propylene compound with the hardest carbon black used by our industry.

Even though this is a tough combination, the compound molds quite well in every type of part we have made. This may be due to a good slug of liquid polybutadiene.

At first we only mill mixed. Then we made a banbury masterbatch and added the cure and part of the black on the mill. I am happy to report that we have now learned to make complete banbury batches with superior physical properties. We have seen 2800 psi tensile from the banbury mix.

Many sample 0-rings and other parts have been supplied since the inception of the program. Of the many sample programs, I will report the field result from just three who used three different types of parts.

The Following Field Report is From WKM Div. ACF Industries, PACKERS

Applications of the Y267 in our 3" valve removal tool service equipment has been a complete success. Our analysis of a compound does not involve chemicals or physicals in either before or after conditions, but rather how it performs under simulated and then real field conditions. Visual appearance after a test sequence or a field usage is the real story teller. All lab tests for the 3" equipment were done on actual field equipment, but with a static steam source of 500°F saturated. Due to a thermostatically controlled heat source, system pressures varied from 600 to 750 psig. Test procedures were to duplicate field operations, and then allow the seal to remain in place one hour before releasing the seal and retrieving the plug. Several compounds of EPDM and AFLAS from different vendors were tested in-house. With the exception of one AFLAS compound, all seal samples performed well enough, but physical deterioration was least in the L'Garde material.

Field trials of our 3" service equipment in the Geysers were most pleasing. Under controlled conditions at 400 psia, W-K-M's field service personnel demonstrated the system would work by installing the plug in a test spool mounted on a customer's well. Thirty minutes later the plug was retrieved. The customer and representatives of three other oil companies present seemed satisfied and approval was given to change both wing valves on the test well. Another plug and seal was used for one valve, and the first plug and seal was used to change the second valve. The valve replacing process takes less than one hour with the seal being in place 35 to 45 minutes.

The second company to be serviced has a well on line flowing into a PG & E power plant at 140,000 pounds per hour. Conditions were 367°F at 167 psi outlet pressure. The second plug was used without changing the seal. The valve changing process was completed without problems. The seal showed little signs of deterioration and could be used again. The third company was serviced with the first plug and seal, again with smooth success. Conditions of this well were reported to be 320 psi and 410°F. This seal, used three times, shows only slight deterioration and can be used again.

K. W. Lazarus

Report From Baker Service Tool, 0-rings

Baker Service Tools began testing L'Garde 267 0-rings in August 1981 for use on a high temperature wireless setting tool. This tool is an arrangement of hydraulic cylinders containing several chambers at atmospheric pressure and a gas generating power charge. It is used in hot oil, gas, or geothermal wells to set bridge plugs, packers and other downhold equipment.

The seal requirements were zero leakage static seal at 550° and 20,000 psi for 8 hrs., and less than 2 cubic inch per hour leakage dynamic seal at 550° and 20,000 psi for 1 hour. The Fluids could be water, oil base drilling mud, waterbase drilling mud, or synthetic oil.

The L'Garde 0-rings were tested with back-up rings under these conditions and looked reusable. In subsequent testing the back-up rings were gradually refined and the temperature and pressure increased.

Our final series of tests were at 600° and 30,000 psi with water, motor oil and synthetic oil. Some of these tests were run for up to 24 hrs. Leakage both static and dynamic was too slow to measure. Testing was stopped at this point because we had reached the limits of our testing equipment.

Several high temp setting tools have been built and extensively lab tested at 600° and 20,000 psi. As of August 8, 1982, they have been run 5 times in deep hot gas wells. There have been no seal failures in the lab or field and there have been no failures at all in the field. These tools are commercially available through any Baker Service Tools District.

Report from Oncor Drilling Tools, Lip Type Seals

Application: Dynamic Sealing in high temperature (above 300°F) abrasive and contaminated drilling fluids.

Test Equipment: 6 1/2" O.D. Equalizer

Equipment Function: The Equalizer is a hydraulic actuated downhole shock tool that maintains a more closely controlled down weight on the drill bit. The pressure generated by the mud pumps reacts on pistons within the Equalizer to maintain a dynamic fluid coupling between the drill bit and the drill stem that resists bit load variations and dissipates destructive forces.

Test Location: Chase Drilling Rig #5 Summit County, Utah 14 - 4 N - 7 E

Drilling Conditions: The test occurred in an 8 1/2" hole drilling through the Weaver and Madison formations. Weight on bit was 45,000 pounds at 50 to 65 revolutions per minute to a depth of 15,900 feet. It was then decreased to 25,000 pounds and rotational speed was increased to 70 revolutions per minute.

The mud type was low solids non dispersed with funnel viscosity of 45-50 seconds and weight of 9.2 pounds per gallon.

Results: The test tool was run into the hole to a depth of 14,534 feet. It drilled 2301 feet accumulating 312 rotating hours. No seal failures occurred. Seal lips were worn and slight heel nibble was typical on all seals. Temperature at surface was 225°F.

Conclusion: Although the test was relatively successful as compared to other elastomeric compounds, further evaluation is needed to verify performance characteristics in oilfield drilling applications.

A New Test Site Has Been Located For A Second Test In The Near Future.

In conclusion, we at Precision feel this is an excellent program and will continue to furnish our 42679, (Y267) compound to any potential user of oil field specialties.

H. Gillette

SUMMARY OF
GEOTHERMAL ELASTOMERIC MATERIALS (GEM) TECHNOLOGY TRANSFER

Alan R. Hirasuna

L'Garde, Inc.
1555 Placentia Avenue
Newport Beach, California 92663

ABSTRACT

High temperature elastomer technology which significantly advances the state-of-the-art was developed by L'Garde under DOE sponsorship. DOE exercised foresight and sponsored direct transferral of this technology to industry. Consequently, the technology can be readily purchased today from three commercial sources in the form of finished elastomeric parts. This paper provides a summary and conclusion of this effort which transcended important technology from a report gathering dust to three ready sources of elastomeric parts providing the benefits of Y267 EPDM technology.

BACKGROUND

During the period from 1976 to 1979, L'Garde, Inc. was contracted by the US-DOE to develop high-temperature geothermal elastomer compounds. At the conclusion of the development, there were several indications that the developments significantly improved the state-of-the-art of high-temperature elastomers.

In pursuit of the basic DOE objective to have developed DOE technology in broad use so that it provides maximum benefit to the American industry, the DOE opted to sponsor L'Garde, Inc. to transfer the high-temperature elastomers technology to industry. Subcontract number BNL 490316S was awarded to L'Garde by Brookhaven National Laboratory.

TECHNOLOGY TRANSFEREE SELECTION

One of the more critical tasks was to select a set of companies to which the technology would be transferred. An application was designed and sent out to a mailing list of over 350 companies. In addition, ads soliciting technology transfer applicants were placed in Petroleum Engineer International and Geothermal Energy Magazine.

There were more than 30 applicants for technology transfer and three were selected after consideration of many factors. The factors had mainly to do with the amount of usage the companies would engender and coverage in the various aspects of drilling and completion.

The three companies that were selected were:
BJ-Hughes, Rubber Products Div.-Los Angeles, CA
Oncor Corporation-Houston, TX
Parker Seals-Culver City, CA
In that Oncor buys their seals from rubber products vendors, they selected Precision Rubber Products-Lebanon, TN to receive the technology in their behalf.

In addition to the prime transferees, three back-ups were selected should additional technology transfer have become possible. The three back-up companies were:
NL Rig Equipment-Stafford, TX
Tri-State Oil Tool, Inc.-Bossier City, LA
Vernay Laboratories, Inc.-Yellow Springs, OH
Unfortunately, no additional funds became available to transfer the technology to any of the back-up companies.

TECHNOLOGY TRANSFER

Under the earlier elastomer compound development contract, L'Garde developed compounds from four polymer systems which exceeded the requirements of the contract, EPDM, fluoroelastomer (Viton), EPDM/Viton blend, and propylene-TFE (AFLAS). The objective of the technology transfer was for L'Garde to transfer the knowledge accumulated during compound development to the transferees such that they are able to reproduce and improve on the development compounds. Of the compounds developed the Y267 EPDM was eminently superior because it has the best hydrothermochemical resistance, it is the easiest to process and its components are the least expensive. Consequently, the technology transfer effort concentrated on the Y267 EPDM with minor efforts spent on the other three polymer systems.

The transfer occurred over several months and each company began making parts for their customers. They are each reporting their experiences at this same conference.

GRAND FINALE TESTS

In conclusion of the technology transfer effort, comparative tests were run between O-rings fabricated by the transferees and L'Garde fabricated Y267 EPDM O-rings. This would have provided a simple conclusion had the transferee O-rings performed equivalent to or better than L'Garde's Y267 EPDM, unfortunately, this was not the case.

L'Garde has a static O-ring test rig which was designed for the Elastomeric Compatibility Testing Program performed for Brookhaven National Laboratory/DOE[1] and provided a perfect testbed for the Grand Finale Tests.

This test rig has the capability of testing O-rings in various hostile fluids at temperatures to 340C (650F) and differential pressures to 34.5 MPa (5000 psi). The ID groove has standard commercial dimensions with a 6 mil diametral gap. Backup rings were NOT run. Four O-ring compounds are tested simultaneously and the leak for each is continuously monitored.

Four sets of size AS 568A-329 O-rings comprised of one each from the transferees and one from L'Garde were tested at 260C (500F) and 28.3 MPa (4100 psi) in synthetic geothermal brine which was developed during the earlier compound development program[2]. Two thirteen-day tests were run with periods where the pressure and/or temperature were cycled.

The Test 1 temperature/pressure duty cycle is shown on Figure 1. The temperature was held constant out to 287 hours. During this period the differential pressure was slightly reversed to -2 MPa (-290 psi) at 125 and 219 hours. Then the temperature and pressure were cycled partially at 287 and then fully to zero differential pressure at 309 hours.

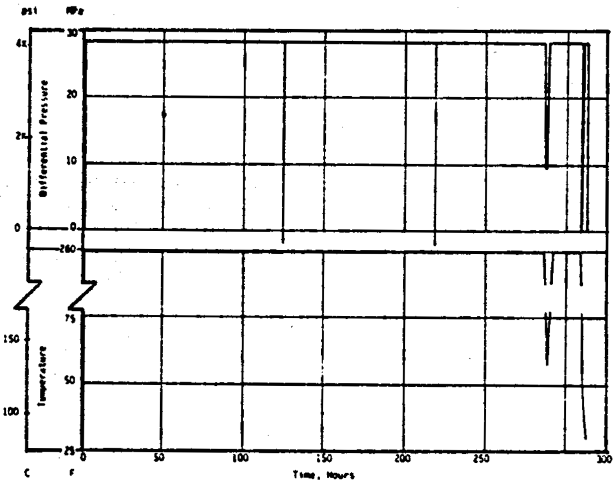


Figure 1. Test 1 Pressure/Temperature History

Test 1 resulted in no catastrophic failures, but there was leakage during the temperature cycles. Based on this and upon post-test O-ring measurements

1. it was concluded that BJ-Hughes N-22-1 tested was not as good as L'Garde Y267 EPDM for the conditions;
2. it was concluded that the Precision 42679 and L'Garde Y267 EPDM compounds tested performed essentially identically;
3. no conclusions could be made regarding Parker E962-85 because its temperature was 11-12C lower than the others. Per Arrhenius, this is equivalent to testing only 40% of the time experienced by the others (5½ days instead of 13 days). Under the less severe environment, the Parker O-rings at 248C did not shrink as much as the others, nor did they leak.

It was recommended that the test be repeated

1. with newly made BJ-Hughes O-rings to validate the above conclusions; and
2. with the Parker and Precision O-ring positions on the holder interchanged so the former sees a hotter temperature.

The Test 2 temperature/pressure duty cycle is shown in Figure 2. For this test the temperature was cycled down to less than 50C (122F) and the differential pressure was slightly reversed and reduced to zero at 135, 231, and 303 hours. At each of these times the differential pressure was reverse cycled to -2 MPa (-290 psi) at the start of cool-down. When at approximately room temperature, the differential pressure was raised to 28.3 MPa for two hours for a leak check. After the leak check the differential pressure was returned to zero, reheating initiated, and full differential pressure reapplied when up to test temperature. This same cycling of temperature and pressure occurred at 135, 231, and 303 hours except at the end of the test at 303 hours, reheating and subsequent full pressurization did not occur.

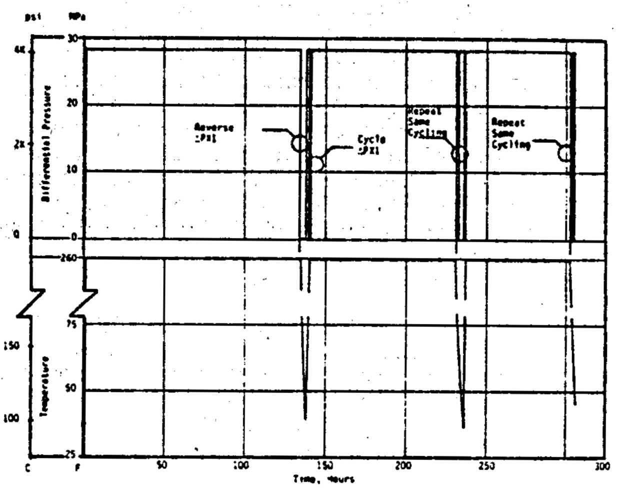


Figure 2. Test 2 Pressure/Temperature History

The new BJ-Hughes N-22-1 O-rings in Test 2 tested equivalent to L'Garde's Y267 EPDM. The Precision 42679 O-rings repeated its equivalent performance with Y267 EPDM. However, the Parker E962-85 O-rings now started leaking after 135 hours and failed at 190 hours at 260C (500F). The test cycle in Test 2 was more severe in that the temperature AND pressure were cycled twice before 287 hours in addition to slightly reversing the differential pressure, and the differential pressure was reversed at the end of the test.

It was concluded from Test 2 that, for the test conditions,

1. the L'Garde Y267 EPDM, Precision, and BJ-Hughes N-22-1 compounds were essentially equal; and
2. the Parker E962-85 compound did not perform as well as the other three compounds.

The duty cycle imposed on the Grand Finale Tests is extreme. Both the temperature and the differential pressures are extremely high especially without the benefit of back-up rings. Under these conditions there will be some permanent set with any polymeric seals, however, less with these compounds than any others known. Permanent set will be shown to greatest disadvantage through reverse pressurization and pressure testing at reduced temperature, as was done. Reducing the temperature results in thermal contraction which further reduces the sealing action already impaired by the permanent set. Consequently, these tests which were purposely made to be extreme to induce failure, do not represent operational reality. In practice means to alleviate the seal environment such as use of back-up rings, replacement of seals after each cycle and use of redundant seals are used.

It is unfortunate that the results of the Grand Finale Tests are muddied. It would have been simple if all transferee compounds tested equal to L'Garde's implying that equivalent product is available from all. At the same time it is important to realize that the data from these two tests are not conclusive and it would probably be erroneous to conclude that one company will supply consistently better parts than another based on these tests alone. All companies have problems with bad batches of parts, hence, L'Garde recommends that parts from more than one manufacturer be tried whenever testing this technology for the first time on critical applications.

CONCLUSION

The DOE GEM Technology Transfer has worked out extremely successfully. There is clearly sufficient test data to conclusively show that the Y267 EPDM has significantly advanced the state-of-the-art for high-temperature elastomers.

The association with the three transferees has been outstanding where each was vitally interested in any technology that can improve their products. They were not hindered in the least with the "Not Invented Here (NIH)" syndrome even though each in his own right is an expert in the elastomer field.

Because of the foresight Brookhaven/DOE exercised in placing this technology transfer effort, Y267 EPDM technology is currently readily available and accessible to industry at a reasonable price. The technology transfer has transcended the developments beyond a report collecting dust in bookcases.

ACKNOWLEDGEMENTS

The author wishes to acknowledge various people for their critical contributions to the project. Mr. G. Friese of L'Garde was responsible for the difficult task of selecting the technology transferees and also the Grand Finale Tests. Mr. C. McFarland and Dr. R. Reeber of DOE-Washington, D.C. were instrumental in providing the opportunity to carry out this important effort. Dr. L. Kukacka of Brookhaven and Mr. A. Adduci of DOE-San Francisco Operations provided vital continual support and guidance throughout the effort.

REFERENCES

[1] Friese, G.J., "Elastomer Compatibility With Brine, Isobutane, and Oil", 1981 Annual Meeting, Geothermal Resources Council, 25-29 October 1981, Houston, TX.

[2] Hirasuna, A.R., et. al., July 1979, Geothermal Elastomeric Materials (GEM) Program-Final Report for Period 1 October 1976-30 June 1979", L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Division of Geothermal Energy.

APPENDIX H

Geothermal Resources Council, TRANSACTIONS, Vol. 3 September 1979

RESULTS OF GEOTHERMAL CASING PACKER SEAL ELASTOMER COMPOUND DEVELOPMENT

Alan R. Hirasuna
Richard W. Sedwick
Clifford A. Stephens

L'Garde, Inc.
Newport Beach, CA 92663

ABSTRACT

Reliable casing packer seal elastomers for the unusually severe geothermal environment at 260°C (500°F) did not exist in 1976. A contract to develop elastomer compounds for this specific application was awarded to L'Garde, Inc. by the Department of Energy. Elastomer compounds were successfully developed which as packer seals maintain seal at 260°C for more than 24 hours in synthetic geothermal brine (aqueous solution of H_2S, CO_2, and NaCl).

The final results of the elastomer compound development are presented. The successful compounds are based on the following polymer systems: EPDM (Nordel^R), fluoroelastomer (Viton^R), EPDM/fluoroelastomer blend, and propylene-TFE (AFLAS^R). Evaluation was performed on full-scale packer seals as well as O-rings by L'Garde. L'Garde ran comparison tests against commercially available elastomers including Kalrez^R. L'Garde's test results were also corroborated independently by three other companies.

INTRODUCTION

In late 1976 L'Garde, Inc. was awarded a contract by the Department of Energy (DOE), DE-AC03-77ET28309 (formerly EG-77-C-03-1308), the Geothermal Elastomeric Materials (GEM) Program. By that time the DOE concluded that there was a general need for improved elastomers for various seal applications which were required to operate in the unusually severe geothermal environment. Logging equipment could not be run for fear of losing seals and damaging the instrumentation. Packers could not be run without first cooling the well. Rotating well head seals and BOP seals required frequent replacement. There was a general need for higher temperature, more hydrochemically resistant elastomers and for that matter the need is still not satisfied in practice today in the geothermal field.

L'Garde performed an initial task to identify what elastomer application when upgraded to geothermal requirements would be of greatest benefit to the geothermal community. The formation packer was identified for two functions, hydraulic stimulation and interval testing in support of overall reservoir characterization. However, though high temperature geothermal formation packers are needed, development of elastomers for this application was judged too ambitious a goal for the first effort. Therefore, it was recommended and approved to first develop improved elastomers for casing packer seal elements. In addition, rubber development for the casing packer element "kills two birds with one stone", as those elastomers which work successfully for the packer seal will most probably work well for static O-ring seals. The O-ring application is less severe because both surfaces are machined, the clearances are small and the required deformation of the O-ring is small compared to a casing packer seal element.

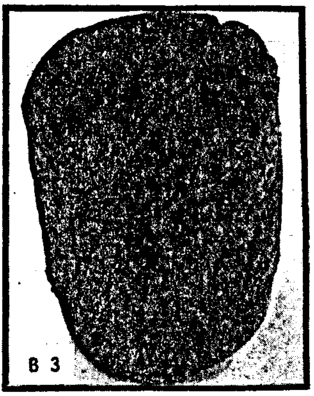
This paper presents the results of the elastomer compound development for high temperature geothermal casing packer seals.

PACKER SEAL SIMULATION EVALUATION AND DEVELOPMENT

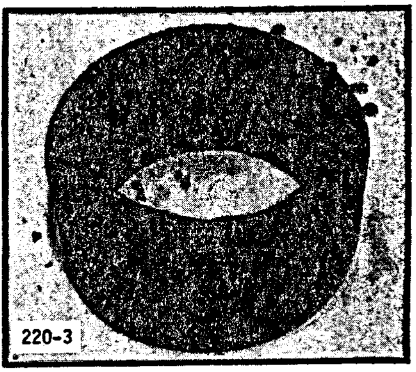
The primary objective of the Program was successfully met. Though meeting the fundamental objective, to develop a casing packer seal elastomer compound which will operate in a 260°C (500°F) geothermal environment, was highly challenging and carried with it a definite probability of failure, L'Garde was fortunate to be able to conclude the program with very positive results. Packer seals which perform well in the oil field up to temperatures of about 150°C (302°F) run into reliability problems at higher temperatures especially when they get as high as 260°C. Though the stresses induced into the elastomeric element are substantially the same as the lower temperatures, the loss in elastomer strength at 260°C is substantial and it cannot sustain the stress levels. Other common problems include hardening of the seal at higher temperatures and associated failure. The geothermal elastomeric packer seal problem at 260°C is significant and even the less demanding static O-ring seal is still a problem. Figure 1 rather lucidly illustrates the starting point of the geothermal packer seal development. These post mortem test specimens were run at 260°C in synthetic brine. At these unusually severe conditions, the seals did one of three things; extruded severely (B3), could not withstand the pack-off and cracked without sealing (220-3), or extruded and broke catastrophically (D3 and E3).

FIGURE 1 -- SIM TEST POST MORTEM SPECIMENS TYPICAL EARLY RESULTS

Company B EPDM -- This and L'Garde EPDM remained elastomeric and survived full 24 hours. Only difference L'Garde EPDM extruded 2.5 in. vs company B's 4.0 in.

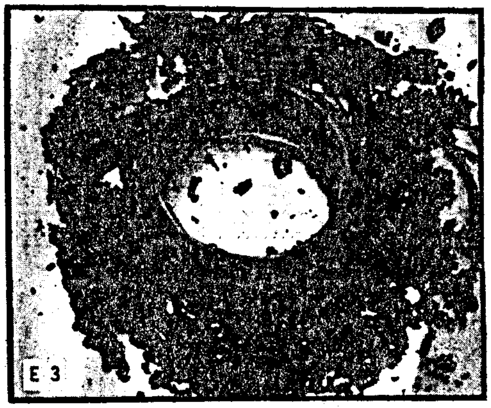


L'Garde Peroxide-Cured Fluoroelastomer -- Failed because of inability to elongate sufficiently during seating.



Company D Epichlorohydrin -- Lost all structural strength and became soft and crumbly before seating.

Company E Non-Peroxide-Cured Fluoroelastomer -- Failed by breaking in the high stress region. Survived 3.75H with slow leak, then failed.



For about the first 2/3 of the contract, it was doubtful that elastomer improvements alone would provide reasonable performance at the test conditions at 260°C.

Upon conclusion of the development, compounds based on four (one was required) separate elastomer systems molded into full-scale packer seals successfully sealed in laboratory simulated packer tests for the prescribed duty cycle - temperature of 260°C, in synthetic geothermal brine, differential pressure of 3100 to 4100 psi, and for 24 hours. The four systems were based on the following polymers: Ethylene Propylene Diene Terpolymer (EPDM, Nordel^R), Fluoroelastomer (FKM, Viton^R), EPDM/FKM Blend (Nordel^R/Viton^R), and Propylene-TFE (AFLAS^R). All four elastomer systems successfully met the Program objectives and show definite improvement over elastomer compounds which are commercially available. Measurable improvement of the hydrothermochemical stability was achieved in the synthetic geothermal brine which includes an aqueous solution of H_2S, CO_2, and NaCl at the following levels: 300 ppm, 1,000 ppm, and 25,000 ppm respectively. For these conditions, the Nordel^R compound is the most hydrothermochemically stable showing no effect after 22 hours. These results represent an effort which included formulating and testing 129 compounds and running 96 seal Simulation Tests.

FIGURE 2 -- POST MORTEM PACKER SEALS AFTER 22 HRS. IN SYNTHETIC GEOTHERMAL BRINE CONTRASTING EARLIER AND CURRENT BEST FORMULATIONS

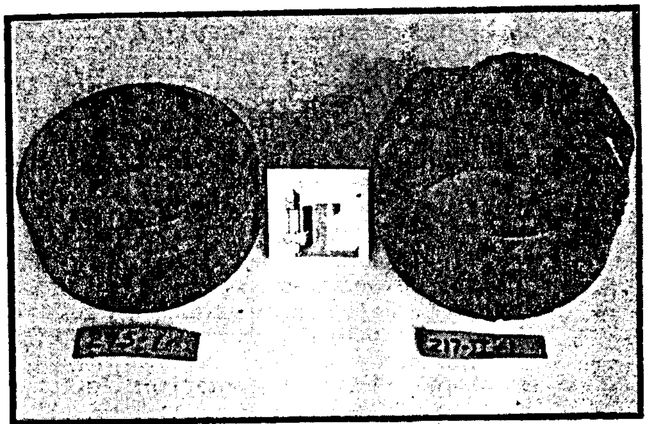


Figure 2 shows post mortem test specimen of one of the better, earlier Nordel^R EPDM compounds, seal 217-II-1. This element sealed successfully at 260°C, but did suffer a significant level of extrusion. In contrast, one of the latest compounds is shown, post mortem seal 275-1 which is a Nordel^R/Viton^R Blend. Only a minor amount of permanent deformation resulted; the seal had an excellent appearance when removed from the test. This element sealed successfully under the same conditions as 217-II-1 except even a higher differential pressure, 3600 vs. 3100 psi.

Compound 267 packer seals were molded for tests by a packer company. Molded seals for 7-inch, 23 lb/ft casing were provided and tested at 302°C (575°F) and 3000 psi differential pressure in their laboratory packer simulation tester. The elements sealed successfully for five days when the test was stopped and their comment was, "after the test they bounced off the floor just like before the test." They are currently planning to test these elements downhole.

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Because this is an unusually severe environment, the GEM compounds have a distinct advantage in that they were developed specifically for this condition. Figure 3 summarizes the capability for each of the compounds. The GEM Viton^R and Nordel^R compounds performed better than their commercial counterparts. The GEM 255 Viton^R held at 3100 psi while the commercial counterparts both broke at 2100 psi. The GEM 267 Nordel^R looked excellent after the 4100 psi run and is capable of higher differential pressures while its counterparts showed incipient failure. There were no commercially comparable counterparts to the Nordel^R/Viton^R Blend or the AFLAS^R, both ran the full 22 hours at the full differential pressure. The Blend looked as good as the 267 Nordel^R and is also capable of higher differential pressures and the AFLAS^R suffered minor extrusion. There was no counterpart to the DuPont Kalrez^R, however, it is of keen interest because it is one of the more chemically stable polymers. It is very expensive but its expense is justifiable in some situations because of the longer life it renders. For the synthetic geothermal brine environment the 3074 Kalrez^R did not fare as well as the four developed compounds in the O-ring test, and earlier coupon ageing tests were made on 3073 Kalrez^R which indicate that it is not as hydrothermochemically stable as the developed compounds. Specific compounding for the geothermal brine environment may result in similar improvements as was herein accomplished.

Figure 4 is a composite photograph of the more interesting seals; L'Garde GEM Nordel^R 267, Parker EPDM E-692-75, L'Garde GEM Nordel^R/Viton^R Blend 266, and the DuPont Kalrez^R 3074. All seals ran successfully for 22 hours at 4100 psi differential pressure except the Kalrez^R 3074 which ran at 2100 psi differential pressure. Note that the Nordel^R 267 and the Nordel^R/Viton^R Blend 266 look excellent, although some permanent squaring of the O-ring did occur. Both the Parker EPDM E-692-75 and the DuPont Kalrez^R 3074 are seen to have damage.

HYDROTHERMOCHEMICAL STABILITY TESTING

Another good quantitative demonstration of the accomplishments is seen through a comparison of compound characteristics for early compounds against commensurate ones at the completion of the program. Table I illustrates the improvements as indicated by the percent of each of the four critical characteristics retained after ageing in brine for 22 hours. Table II lists the virgin and chemically aged values which are represented by the percentage numbers.

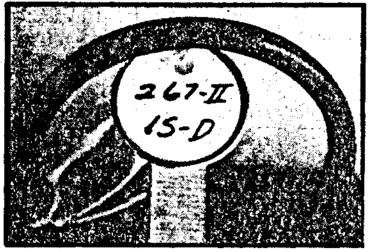
FIGURE 3 -- GEM O-RING SIM TESTS
SYN. BRINE AT 260°C (500°F)

TYPE	COMPOUND / DIFFERENTIAL PRESSURE	2100 PSI	3100 PSI	4100 PSI	?
FLUOROELAST.	L'GARDE GEM VITON 255	▨	22H	6.25H	
	PARKER FLUOROELAST. V-709-90	10.5H	9H	4H	
	PARKER FLUOROELAST. V-747-75	5.25H		0.2H	
EPDM	L'GARDE GEM NORDEL 267	▨	▨	22H	▨
	PARKER EPDM E-692-75	▨	▨	22H	
MISC.	L'GARDE GEM NORDEL/VITON BLEND 266	▨	▨	22H	▨
	L'GARDE GEM AFLAS 291	▨	▨	22H	
	DUPONT KALREZ 3074	22H	3.75H	0.75H	

Note that there is significant improvement reflected by the latest compounds. The 267 Nordel^R is virtually unchanged by the 22 hour ageing in the synthetic brine with respect to all four characterization parameters. Vast improvement was finally accomplished with the 255 Viton^R. There is room for improvement on the Extrusion Resistance especially, as is reflected by the Packer Seal Simulation Tests, however, the extrusion resistance is adequate to meet the requirements. The 266 Nordel^R/Viton^R is very stable with some minor degradation of the ultimate elongation. The 291 AFLAS^R shows some minor hydrothermochemical instability in the ultimate elongation. In general, all of the latest formulations showed very good stability considering the unusual severity of environment.

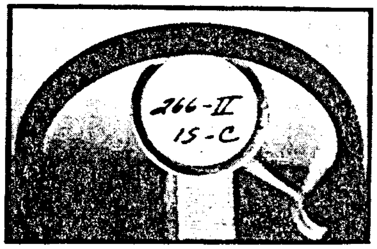
The above data are all comprehensively reported in Reference 2.

FIGURE 4 -- POST MORTEM O-RINGS SIM TEST SEALS AFTER 22 HRS. IN SYNTHETIC GEOTHERMAL BRINE @ 500°F

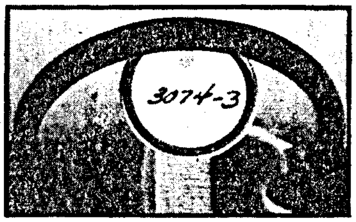


L'Garde GEM Nordel^R 267 EPDM, ΔP = 4,100 psi

Parker EPDM E-692-75, ΔP = 4,100 psi



L'Garde GEM Nordel^R/Viton^R Blend, ΔP = 4,100 psi



DuPont Kalrez^R 3074, ΔP = 2,100 psi

TABLE I -- PROGRESS OF HYDROTHERMOCHEMICAL STABILITY OF GEOTHERMAL ELASTOMERS

Elastomer	Ult. Tensile @ RT		Ult. Elong. @ RT		Shore A Hard @ 260°C		Extrusion Resist. @ 260°C	
	Early	Present	Early	Present	Early	Present	Early	Present
EPDM								
Early-E263	75%		--		•85%		57%	
Present-267		97%		105%		100%		103%
Viton								
Early-115	14%		46%		73%		32%	
Present-255		72%		101%		100%		55%
EPDM/Viton								
Early-235	80%		63%		110%		128%	
Present-266		96%		84%		101%		96%
AFLAS								
Early-268	77%		112%		90%		84%	
Present-291		93%		118%		100%		87%

TABLE II -- GEOTHERMAL ELASTOMERS PROGRESS

Elastomer	Ult. Tensile @ RT. psi		Ult. Elong. @ RT %		Shore A Hard @ 260°C		Extrusion Resist @ 260°C, psia	
	V	CA	V	CA	V	CA	V	CA
EPDM								
Early-E263	1363	1020	>840	>840	61	52	9.6	5.5
Present-267	1610	1554	141	148	92	92	17.1	17.6
Viton								
Early-115	1699	244	162	74	80	58	11.2	3.6
Present-255	2195	1591	224	226	79	79	11.4	6.3
EPDM/Viton								
Early-235	1882	1506	681	431	67	74	8.3	10.6
Present-266	1596	1531	193	163	88	89	18	17.3
AFLAS								
Early-268	1673	1289	394	442	69	63	6.7	5.6
Present-291	2335	2162	134	158	95	95	17.4	15.2

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research and development and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE.

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REFERENCES

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APPENDIX I

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High Temperature Electronics and Instrumentation Seminar Proceedings December 3-4, 1979

Editors
Anthony F. Veneruso, Richard S. Simpson, and Charles Arnold

Prepared by Sandia National Laboratories, Albuquerque, New Mexico 87185
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May 1980

Sandia Laboratories

RESULTS OF GEOTHERMAL CASING PACKER SEAL ELASTOMER COMPOUND DEVELOPMENT

Alan R. Hirasuna, Richard A. Sedwick
Clifford A. Stephens

L'Garde, Inc.
Newport Beach, CA 92663

ABSTRACT

Reliable casing packer seal elastomers for the unusually severe geothermal environment at 260°C (500 F) did not exist in 1976. A contract to develop elastomer compounds for this specific application was awarded to L'Garde, Inc. by the Department of Energy. Elastomer compounds were successfully developed which as packer seals maintain seal at 260°C for more than 24 hours in synthetic geothermal brine (aqueous solution of H_2S, CO_2, and NaCl).

The final results of the elastomer compound development are presented. The successful compounds are based on the following polymer systems: EPDM (Nordel^R), fluoroelastomer (Viton^R), EPDM/fluoroelastomer blend, and propylene-TFE (AFLAS^R). Evaluation was performed on full-scale packer seals as well as O-rings by L'Garde. L'Garde ran comparison tests against commercially available elastomers including Kalrez^R. L'Garde's test results were also corroborated independently by three other companies.

INTRODUCTION

In late 1976 L'Garde, Inc. was awarded a contract by the Department of Energy (DOE), DE-AC03-77ET28309 (formerly EG-77-C-03-1308), the Geothermal Elastomeric Materials (GEM) Program. By that time the DOE concluded that there was a general need for improved elastomers for various seal applications which were required to operate in the unusually severe geothermal environment. Logging equipment could not be run for fear of losing seals and damaging the instrumentation. Packers could not be run without first cooling the well. Rotating well head seals and BOP seals required frequent replacement. There was a general need for higher temperature, more hydrochemically resistant elastomers and for that matter the need is still not satisfied in practice today in the geothermal field.

L'Garde performed an initial task to identify what elastomer application when upgraded to geothermal requirements would be of greatest benefit to the geothermal community. The formation packer was identified for two functions, hydraulic stimulation and interval testing in support of overall reservoir characterization. However, though high temperature geothermal formation packers are needed, development of elastomers for this application was judged too ambitious a goal for the first effort. Therefore, it was recommended and approved to first develop improved elastomers for casing packer seal elements. In addition, rubber development for the casing packer element "kills two birds with one stone", as those elastomers which work successfully for the packer seal will most probably work well for static O-ring seals. The O-ring application is less severe because both surfaces are machined, the clearances are small and the required deformation of the O-ring is small compared to a casing packer seal element.

This paper presents the results of the elastomer compound development for high temperature geothermal casing packer seals.

PACKER SEAL SIMULATION EVALUATION AND DEVELOPMENT

The primary objective of the Program was successfully met. Though meeting the fundamental objective, to develop a casing packer seal elastomer compound which will operate in a 260°C (500°F) geothermal environment, was highly challenging and carried with it a definite probability of failure, L'Garde was fortunate to be able to conclude the program with very positive results. Packer seals which perform well in the oil field up to temperatures of about 150°C (302°F) run into reliability problems at higher temperatures especially when they get as high as 260°C. Though the stresses induced into the elastomeric element are substantially the same as at lower temperatures, the loss in elastomer strength at 260°C is substantial and it cannot sustain the stress levels. Other common problems include hardening of the seal at higher temperatures and associated failure. The geothermal elastomeric packer seal problem at 260°C is significant and even the less demanding static O-ring seal is still a problem. Figure 1 rather lucidly illustrates the starting point of the geothermal packer seal development. These post mortem test specimens were run at 260°C in synthetic brine. At these unusually severe conditions, the seals did one of three things; extruded severely (B3), could not withstand the pack-off and cracked without sealing (220-3), or extruded and broke catastrophically (D3 and E3). For about the first 2/3 of the contract, it was doubtful that elastomer improvements alone would provide reasonable performance at the test conditions at 260°C.

Upon conclusion of the development, compounds based on four (one was required) separate elastomer systems molded into full-scale packer seals successfully sealed in laboratory simulated packer tests for the prescribed duty cycle - temperature of 260°C, in synthetic geothermal brine, differential pressure of 3100 to 4100 psi, and for 24 hours. The four systems were based on the following polymers: Ethylene Propylene Diene Terpolymer (EPDM, Nordel^R), Fluoroelastomer (FKM, Viton^R), EPDM/FKM Blend (Nordel^R/Viton^R), and Propylene-TFE (AFLAS^R). All four elastomer systems successfully met the program objectives and show definite improvement over elastomer compounds which are commercially available. Measurable improvement of the hydrothermochemical stability was achieved in the synthetic geothermal brine which includes an aqueous solution of H_2S, CO_2, and NaCl at the following levels: 300 ppm, 1,000 ppm, and 25,000 ppm, respectively. For these conditions, the Nordel^R compound is the most hydrothermochemically stable showing no effect after 22 hours. These results represent an effort which included formulating and testing 129 compounds and running 96 seal simulation tests.

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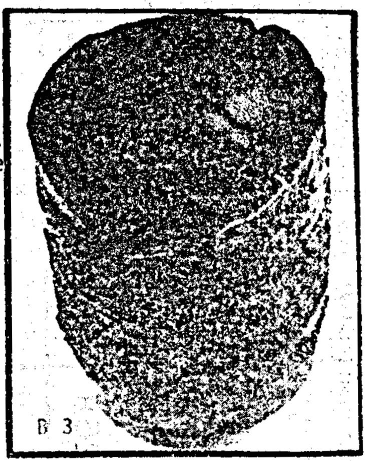
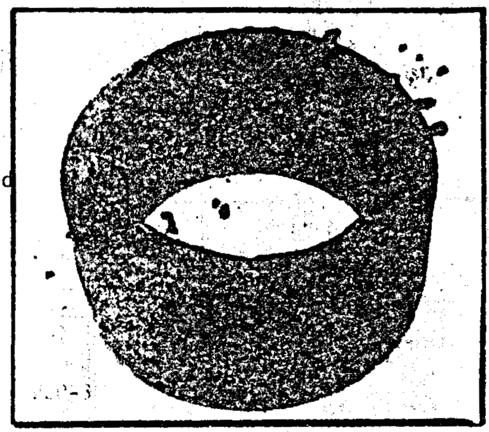
FIGURE 1 -- SIM TEST POST MORTEM SPECIMENS TYPICAL EARLY RESULTS

Company B EPDM -- This and L'Garde EPDM remained elastomeric and survived full 24 hours. Only difference L'Garde EPDM extruded 2.5 in. vs company B's 4.0 in.

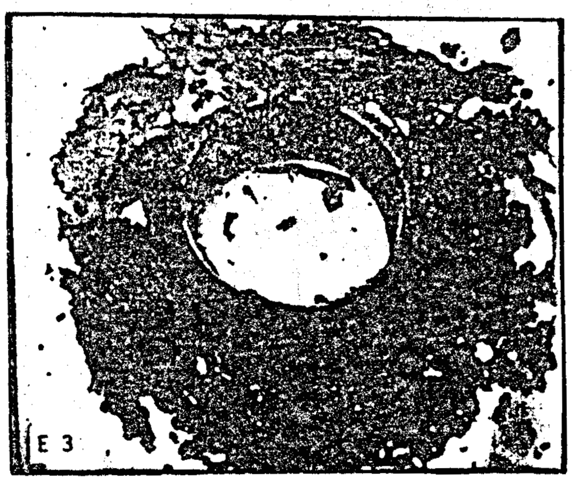


L'Garde Peroxide-Cured Fluoroelastomer -- Failed because of inability to elongate sufficiently during seating.



Company D Epichlorohydrin -- Lost all structural strength and became soft and crumbly before seating.

Company E Non-Peroxide-Cured Fluoroelastomer -- Failed by breaking in the high stress region. Survived 3.75H with slow leak, then failed.



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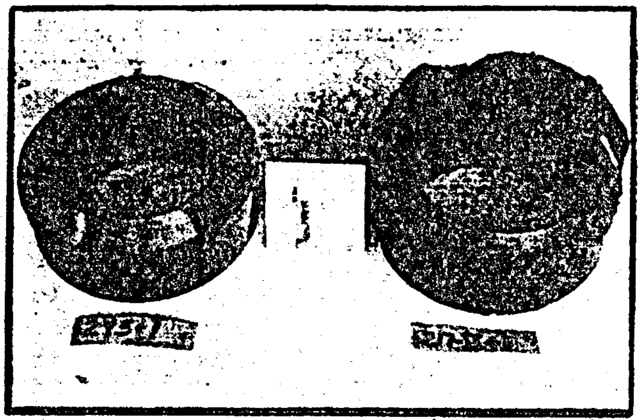

ΔP = 3,600 psi ΔP = 3,100 psi

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	PARKER FLUOROELAST. V-769-90	10.5 H	9 H	4 H	
	PARKER FLUOROELAST. V-747-75	5.25H		0.2H	
EPDM	L'GARDE GEM NORDEL 267			22H	
	PARKER EPDM E-692-75			22H	
MISC.	L'GARDE GEM NORDEL/VITON BLEND 266			22H	
	L'GARDE GEM AFLAS 291			22H	
	DUPONT KALREZ 3074	22H	3.75 H	0.75H	

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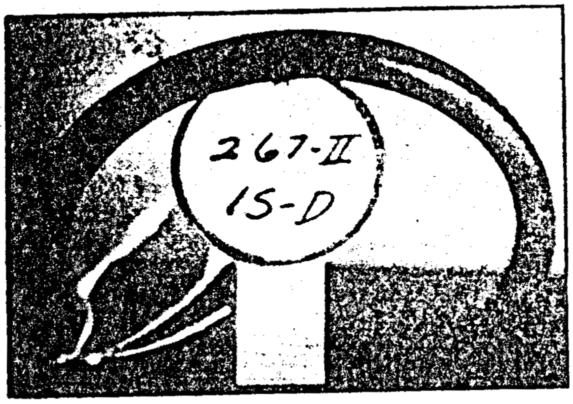
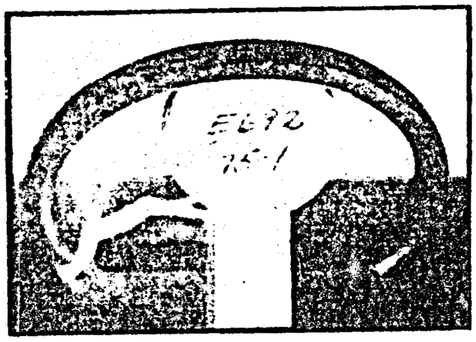
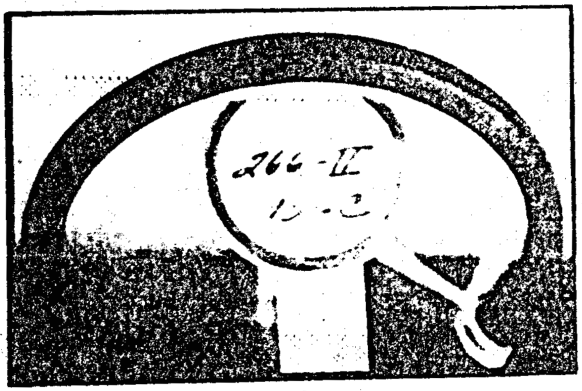


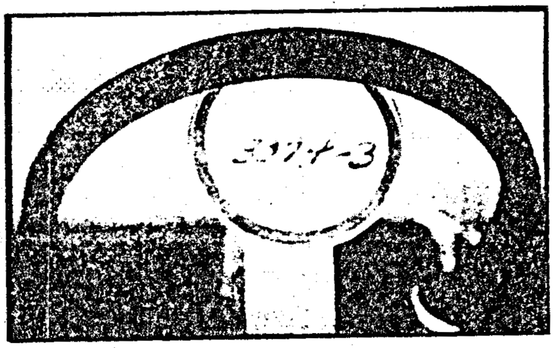
L'Garde GEM Nordel^R 267 EPDM, ΔP = 4,100 psi



Parker EPDM E-692-75, ΔP = 4,100 psi



L'Garde GEM Nordel^R/Viton^R Blend, ΔP = 4,100 psi



DuPont Kalrez^R 3074, ΔP = 2,100 psi

Paper No. 36

HIGH TEMPERATURE GEOTHERMAL ELASTOMER COMPOUND DEVELOPMENT*

A.R. Hirasuna, R.A. Sedwick, and C.A. Stephens
L'Garde, Inc., Newport Beach, CA 92663

*Presented at a meeting of the Rubber Division, American Chemical Society, Las Vegas, NV, May 20-23, 1980. Based on a paper presented at a meeting of the Geothermal Resources Council, Reno, NV, September 24-27, 1979.

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Reliable casing packer seal elastomers for the unusually severe geothermal environment at 260°C (500°F) did not exist in 1976. In late 1976 L'Garde, Inc. was awarded a contract by the Department of Energy (DOE), DEAC03-77-ET28309 (formerly EG-77-C-03-1308), the Geothermal Elastomeric Materials (GEM) Program.[1, 2, 3, 4] By that time the DOE concluded that there was a general need for improved elastomers for various seal applications which were required to operate in the unusually severe geothermal environment. Logging equipment could not be run for fear of losing seals and damaging the instrumentation. Packers could not be run without first cooling the well. Rotating well head seals and BOP seals required frequent replacement. There was a general need for higher temperature, more hydrochemically and extrusion resistant elastomers and for that matter the need is still not satisfied in practice today in the geothermal field.

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This paper presents the results of the elastomer compound development for high temperature geothermal casing packer seals. Elastomer compounds were successfully developed which as packer seals maintain seal at 260°C for more than 24 hours in synthetic geothermal brine (aqueous solution of H_2S, CO_2, and NaCl). The successful compounds are based on the following polymer systems: EPDM (Nordel^R), FKM (Viton^R), EPDM/FKM (Nordel/Viton) blend, and propylene-TFE (AFLAS^R). Evaluation was performed on full-scale packer seals as well as O-rings by L'Garde. L'Garde ran comparison tests against commercially available elastomers including Kalrez^R. L'Garde's test results were also corroborated independently by three other companies.[5, 6, 7]

PACKER SEAL SIMULATION, EVALUATION, AND DEVELOPMENT

The primary objective of the Program was successfully met. Though meeting the fundamental objective, to develop a casing packer seal elastomer compound which will operate in a 260°C (500°F) geothermal environment, was highly challenging and carried with it a definite probability of failure, L'Garde was fortunate to be able to conclude the program with very positive results.

Packer seals which perform well in the oil field up to temperatures of about 150°C (302°F) run into reliability problems at higher temperatures especially when they get as high as 260°C. Though the stresses induced into the elastomeric element are substantially the same as the lower temperatures, the loss in elastomer strength at 260°C is substantial and, typically, the stress exceeds the reduced strength and failure occurs. Other common problems include hardening or reversion softening of the elastomer at higher temperatures with associated failure. The geothermal elastomeric packer seal problem at 260°C is significant and even the less demanding static O-ring seal is still a problem. Figure 1 rather lucidly illustrates the starting point of the geothermal packer seal development. These post mortem test specimens were run at 260°C in synthetic brine. At these unusually severe conditions, the seals did one of three things; extruded severely (B3), could not withstand the pack-off and cracked without sealing (220-3), or extruded and broke catastrophically (D3 and E3). For about the first 2/3 of the contract, it was doubtful that elastomer improvements alone would provide reasonable performance at the test conditions at 260°C.

The GEM Program was initiated by developing a program around packer seal elastomer compound development and evaluation. For this unusually severe environmental requirement, L'Garde feels that good simulation tests are essential. A battery of simple tests were defined for broad brush screening of compounds. All screening tests used simple specimens cut from slabs and comparisons between virgin specimens and specimens aged in synthetic geothermal brine at 260°C were made to assess the hydrothermochemical stability of the compounds. A special test was designed and fabricated which measures the extrusion resistance of a specimen about the size of a quarter.

Those compounds which performed well in the screening were then graduated to full scale packer seal tests. A laboratory simulator was designed and fabricated which subjects a full scale 10.2 cm (4-inch) packer seal to simulated downhole environments, in the laboratory. In this test the seal is simultaneously subjected to the chemistry, temperature, mechanical forces, pressure forces, and limited exposure of the seal just as it is on an operational packer. This provides the full spectrum of interactive operational environments, the combination of which is important when evaluating materials being stressed to their ultimate capacity.

The above evaluation scheme proved invaluable and enabled efficient evaluation of 129 compounds with quick feedback for subsequent compounding efforts. Ninety-six (96) seal simulation tests were run which provide proof-of-pudding data with little need for uncertain extrapolation to judge whether it will perform operationally. The testing and evaluation scheme is detailed in References 2 and 4.

Upon conclusion of the development, compounds based on four (one was required) separate polymer systems molded into full-scale packer seals successfully sealed in laboratory simulated packer tests in excess of the prescribed duty cycle - temperature of 260°C, in synthetic geothermal brine, differential pressure of 21.4-28.3 MPa (3100 to 4100 psi), and for 24 hours. The four systems were based on the following polymers: EPDM, FKM, EPDM/FKM Blend, and Propylene-TFE. All four elastomer systems successfully exceeded the Program objectives and show definite improvement over elastomer compounds which are commercially available. Measurable improvement of the hydrothermochemical stability was achieved in the synthetic geothermal brine which includes an aqueous solution of H_2S, CO_2 and NaCl at the following levels: 300 ppm, 1,000 ppm, and 25,000 ppm respectively.

Figure 2 shows post mortem test specimen of one of the better, earlier EPDM compounds, seal 217-II-1. This element sealed successfully at 260°C, but did suffer a significant level of extrusion. In contrast, one of the latest compounds is shown, post mortem seal 275-1 which is EPDM/FKM Blend. Only a minor amount of permanent deformation resulted; the seal had an excellent appearance when removed from the test. This element sealed successfully under the same conditions as 217-II-1 except even a higher differential pressure, 24.8 vs. 21.4 MPa (3600 vs. 3100 psi).

PACKER COMPANY LABORATORY AND FIELD EXPERIENCE

Compound 267 EPDM packer seals were molded for tests by Otis Engineering Corp.[6] Molded seals for 7-inch, 26 lb/ft casing were provided and tested at 302°C (575°F) and 20.7 MPa (3000 psi) differential pressure in their laboratory packer simulation tester. The elements sealed successfully for five days when the test was stopped and their comment was, "After the test they bounced off the floor just like before the test."

Subsequent to the laboratory tests, this packer company installed a 267 EPDM element on a new R&D packer and ran it in a heavy-oil field well in Bakersfield. The well has 7-inch casing and is used for continuous steam injection. The steam is about 246°C (475°F) at the boiler and 204°C (400°F) at the well head.

The packer was pulled after 5 months of successful sealing to perform well maintenance. Tracer tests confirmed that the packer was still sealing prior to removal. The packer was pulled with no problems whatsoever, where usually difficulty is encountered with the seal jamming and either being rolled or ripped off the packer.

The appearance of the recovered seal was excellent with virtually no permanent deformation whatsoever. It looked like a new seal and obviously could have remained in operation indefinitely. There was no apparent degradation from the crude oil which covered the packer when it was removed. Figure 3 is a photograph of the packer seal element after removal from the Bakersfield well.

CORROBORATING O-RING SIMULATION TESTS

A good quantitative demonstration of the accomplishments is the O-ring SIM Test data which compares the developed compounds against a limited number of commercially available compounds. The packer simulation test was modified for static simulation testing of O-rings. The O-rings were tested at 260°C in the synthetic geothermal brine starting at 28.3 MPa (4100 psi) differential pressure. The test was set for 22 hours and if a compound failed before 22 hours, the differential pressure was decreased by 6.9 MPa (1000 psi) and the test rerun with a new seal. The reduction in differential pressure progressed to 14.5 MPa (2100 psi) where the testing stopped if the compound failed.

Because this is an unusually severe environment, the GEM compounds have a distinct advantage in that they were developed specifically for this condition.

Figure 4 summarizes the capability for each of the compounds. The GEM 255 FKM and 267 EPDM compounds performed better than their commercial counterparts. The 255 FKM held at 21.4 MPa (3100 psi) while the commercial counterparts both broke at 14.5 MPa (2100 psi). The GEM 267 EPDM looked excellent after the 28.3 MPa (4100 psi) run and is capable of higher differential pressures while its counterparts showed incipient failure. There were no commercially comparable counterparts for the EPDM/FKM Blend or the Propylene-TFE, both ran the full 22 hours at the full differential pressure. The Blend looked as good as the 267 EPDM and is also capable of higher differential pressures and the Propylene-TFE suffered minor extrusion. There was no counterpart to the DuPont Perfluoroelastomer (Kalrez^R), however, it is of keen interest because it is one of the more chemically stable polymers. It is very expensive but its expense is justifiable in some situations because of the longer life it renders. For the synthetic geothermal brine environment the 3074 Perfluoroelastomer did not fare as well as the four developed compounds in the O-ring test, and earlier coupon ageing tests were made on 3073 Perfluoroelastomer which indicate that it is not as hydrothermochemically stable as the developed compounds. Specific compounding for the geothermal brine environment may result in similar improvements as was herein accomplished.

Figure 5 is a composite photograph of the more interesting seals; 267 EPDM, E-692-75 EPDM, 266 EPDM/FKM Blend, and the Perfluoroelastomer 3074. All seals ran successfully for 22 hours at 28.3 MPa (4100 psi) differential pressure except the Perfluoroelastomer 3074 which ran at 14.5 MPa (2100 psi) differential pressure. Note that the 267 EPDM and the 266 EPDM/FKM Blend look excellent, although some permanent squaring of the O-ring did occur. Both the E-692-75 EPDM and the Perfluoroelastomer 3074 are seen to have damage.

HYDROTHERMOCHEMICAL STABILITY TESTING

Another good quantitiative demonstration of the accomplishments is seen through a comparison of compound characteristics for early compounds against commensurate ones at the completion of the program. Table I illustrates the improvements as indicated by the percent of each of the four critical characteristics retained after ageing in synthetic brine for 22 hours. Table II lists the virgin and chemically aged values which are represented by the percentage numbers.

Note that there is significant improvement reflected by the latest compounds. The 267 EPDM is virtually unchanged by the 22 hour ageing in the synthetic brine with respect to all four characterization parameters. Vast improvement was finally accomplished with the 255 FKM. There is room for improvement on the Extrusion Resistance especially, as is reflected by the Packer Seal Simulation Tests, however, the extrusion resistance is adequate to meet the requirements and the results are quite respectable. The 266 EPDM/FKM is very stable with some minor degradation of the ultimate elongation. The 291 Propylene-TFE shows some minor hydrothermochemical instability in the ultimate elongation. In general, all of the latest formulations showed very good stability considering the unusual severity of environment.

The above data are all comprehensively reported in Reference 1.

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research

and development and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory was especially helpful.

REFERENCES

[1] Hirasuna, A.R., et al., July 1979, "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976 - 30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy (To Be Published).

[2] Hirasuna, A.R., et al., December 1977, "Geothermal Elastomeric Materials - Twelve-Months Progress Report, October 1, 1976 - September 30, 1977," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-1, U.S. Department of Energy, Geothermal Energy.

[3] Hirasuna, A.R., et al., "Important Conclusions Based on Results of Geothermal Casing Packer Seal Elastomers Testing," presented at a meeting of the Geothermal Resources Council, Hilo, Hawaii, 25-27 July 1978, Summary in Transactions, page 279.

[4] Hirasuna, A.R., et al., "Evaluation Tests for Geothermal Casing Packer Seal Elastomers," presented at a meeting of the American Institute of Chemical Engineers, Philadelphia, Pennsylvania, 4-8 June 1978.

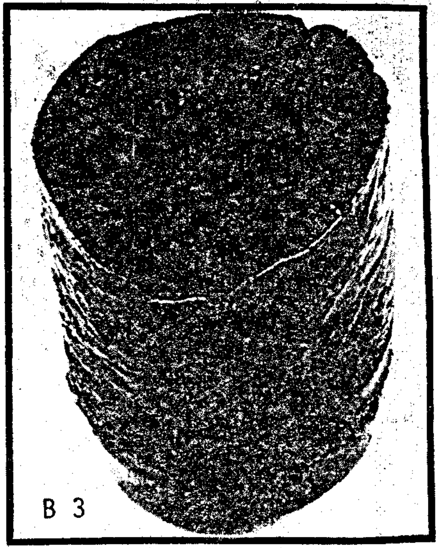
[5] Arnold, C., Jr., "Memo to Distribution," Sandia Laboratories, 19 April 1979, Albuquerque, New Mexico.

[6] Taylor, D., Private Communication, Otis Engineering Corp., 23 April 1979, Dallas, Texas.

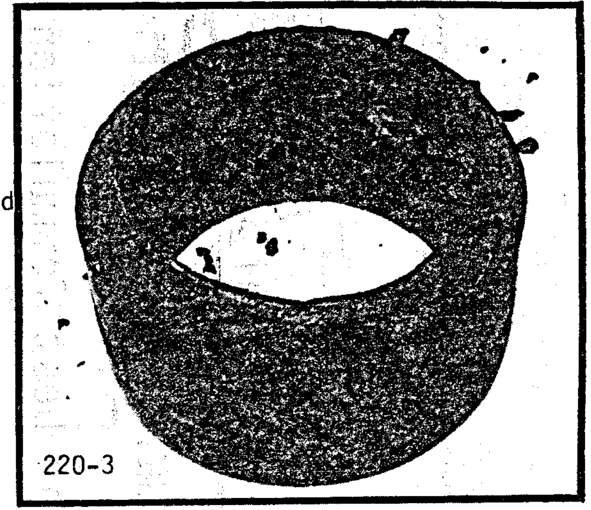
[7] Winzenried, R., "Letter to Hirasuna," Terratek, 15 June 1979, Salt Lake City, Utah.

Fig. 1 -- SIM Test post mortem specimens typical early results.

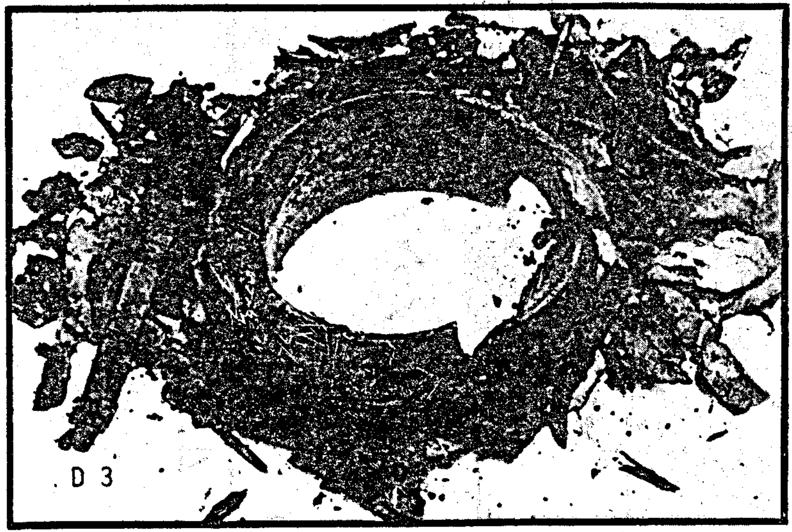
Company B EPDM -- This and L'Garde EPDM remained elastomeric and survived full 24 hours. Only difference L'Garde EPDM extruded 2.5 in. vs company B's 4.0 in.


B 3

L'Garde Peroxide-Cured Fluoroelastomer -- Failed because of inability to elongate sufficiently during seating.


220-3

Company D Epichlorohydrin -- Lost all structural strength and became soft and crumbly before seating.


D 3

Company E Non-Peroxide-Cured Fluoroelastomer -- Failed by breaking in the high stress region. Survived 3.75H with slow leak, then failed.

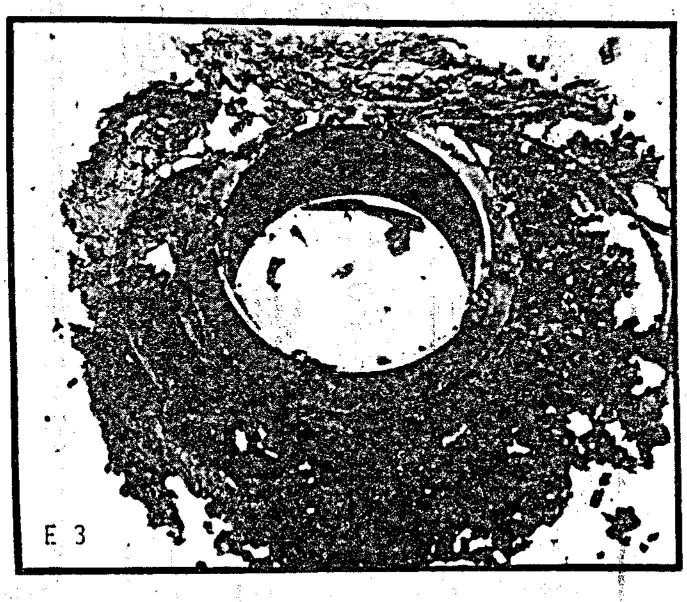
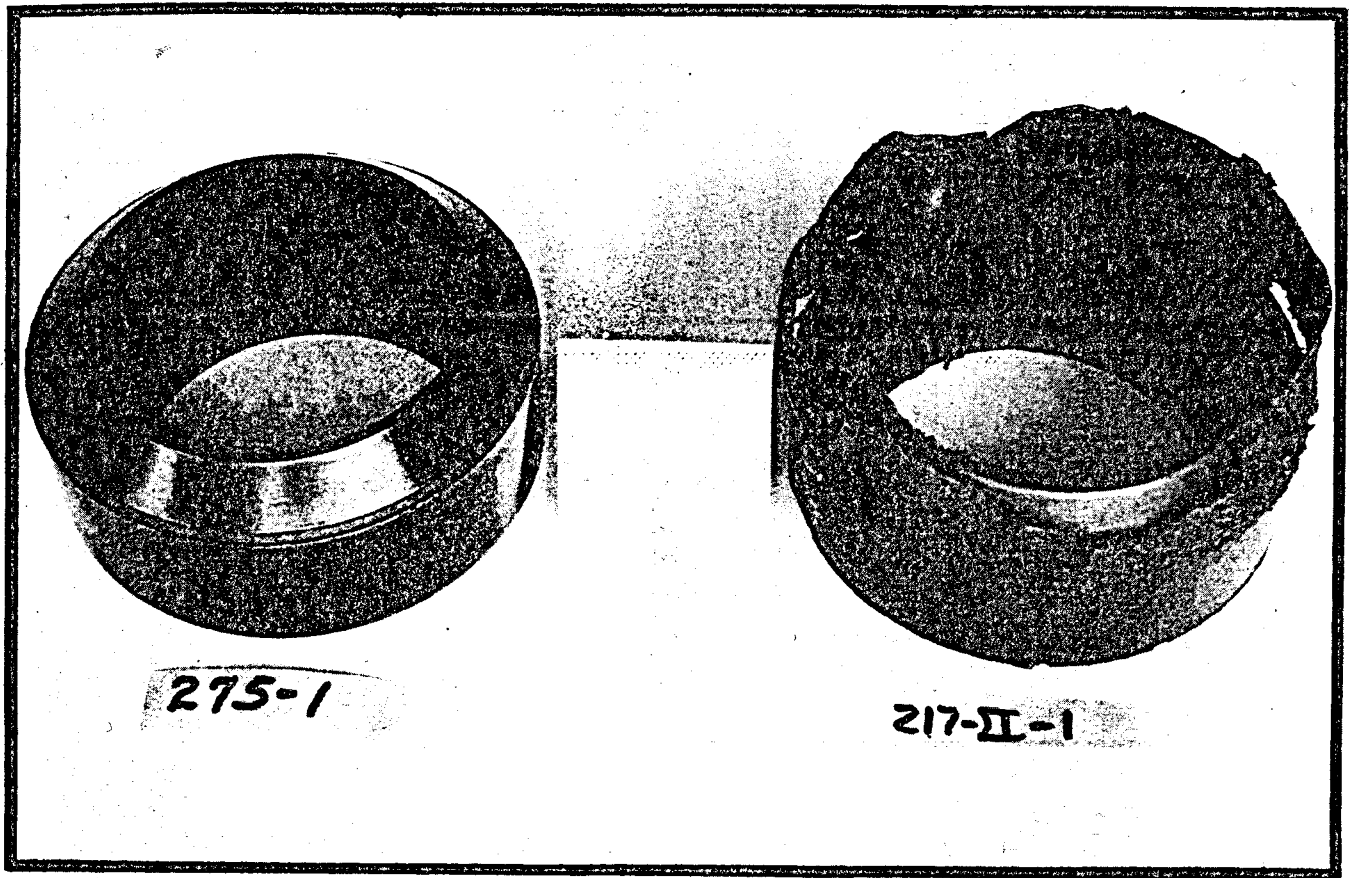

E 3

Fig. 2 -- Post mortem packer seals after 22 hrs. in synthetic geothermal brine contrasting earlier and current best formulations.



ΔP = 3,600 psi ΔP = 3,100 psi

Fig. 3 -- EPDM after 5 months in Bakersfield steam injection well for heavy oil.

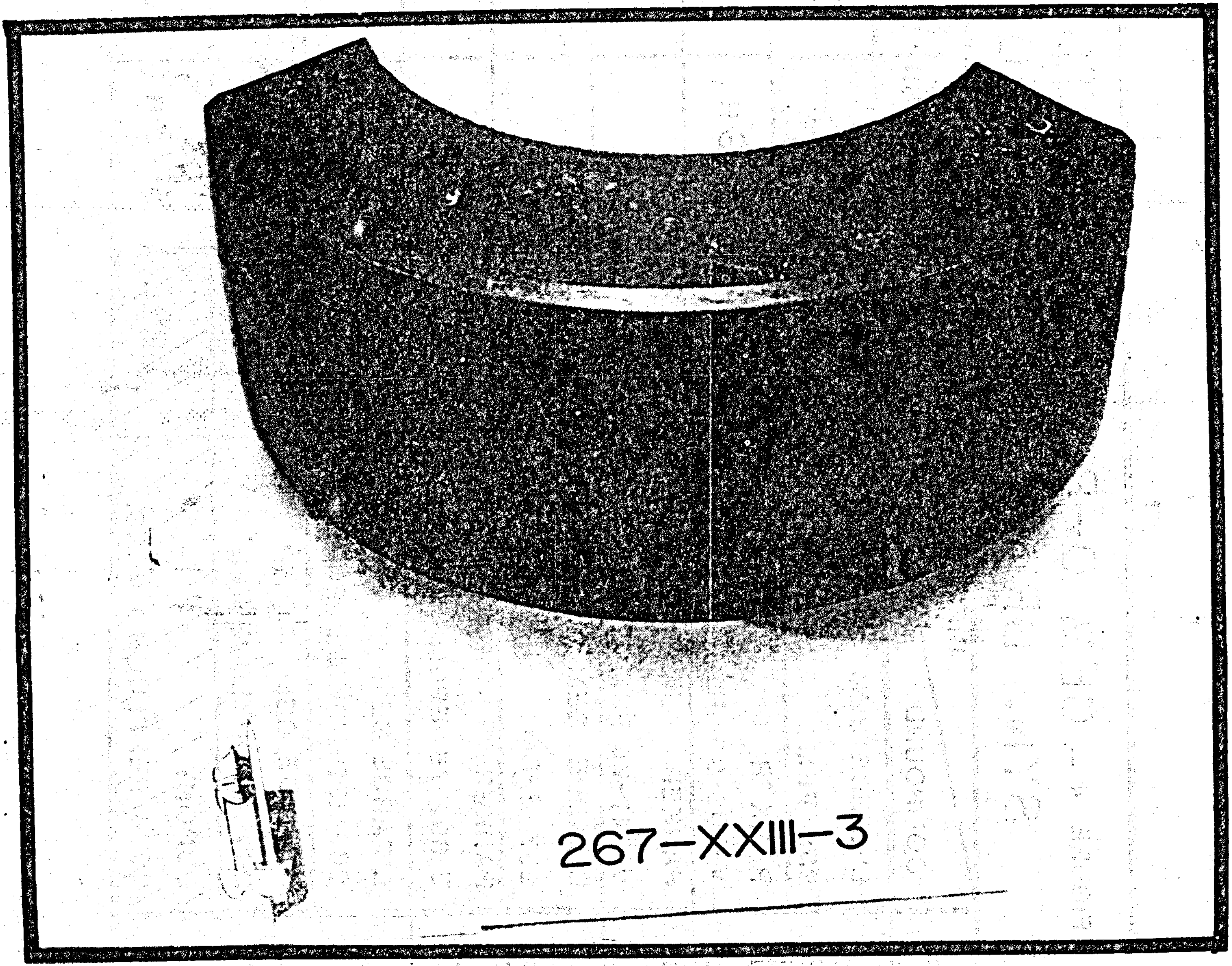
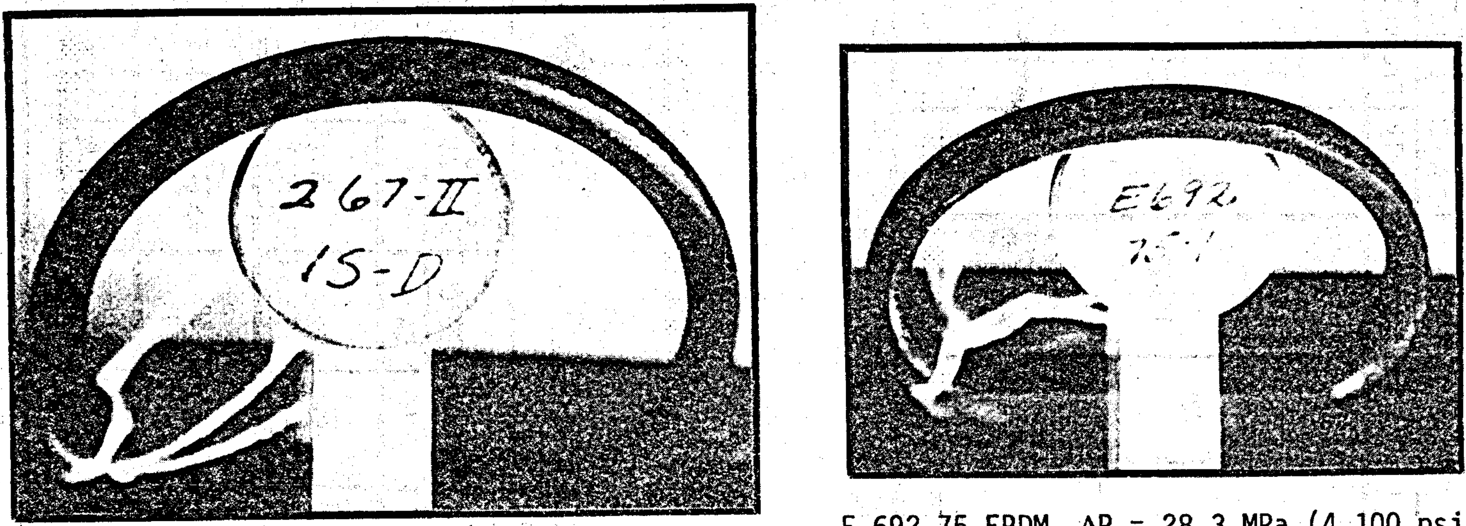


FIGURE 4 -- GEM O-RING SIM TESTS SYN. BRINE AT 260°C (500°F)

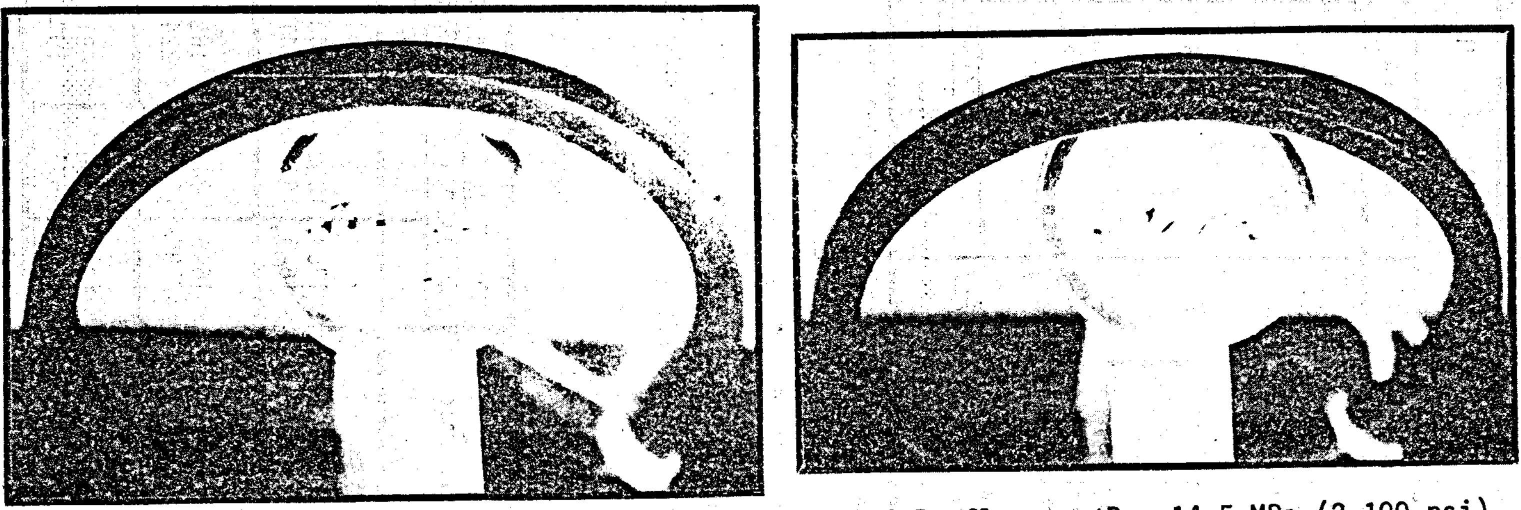
TYPE	COMPOUND / DIFFERENTIAL PRESSURE	14.5 MPa (2100 PSI)	21.4 MPa (3100 PSI)	28.3 MPa (4100 PSI)	?
FLUOROELAST.	L'GARDE GEM 255 FKM		22H	6.25H	
	PARKER V-709-90 FKM	10.5H	9H	4H	
	PARKER V-747-75 FKM	5.25H		0.2H	
EPDM	L'GARDE GEM 267 EPDM			22H	
	PARKER E-692-75 EPDM			22H	
MISC.	L'GARDE GEM 266 EPDM/FKM BLEND			22H	
	L'GARDE GEM 291 PROPYLENE-TFE			22H	
	DUPONT 3074 PERFLUORO ELAS.	22H	3.75H	0.75H	

Fig. 5 -- Post mortem o-rings SIM Test seals after 22 hrs. in sour synthetic geothermal brine @ 500^{0}F.



267 EPDM/FKM Blend, ΔP = 28.3 MPa (4,100 psi)

E-692-75 EPDM, ΔP = 28.3 MPa (4,100 psi)



266 EPDM/FKM Blend, ΔP = 28.3 MPa (4,100 psi)

3074 Perfluoro, ΔP = 14.5 MPa (2,100 psi)

Table I

Progress of hydrothermochemical stability of geothermal elastomers.

Elastomer	Ult. Tensile @ RT		Ult. Elong. @ RT		Shore A Hard @ 260°C		Extrusion Resist. @ 260°C	
	Early	Present	Early	Present	Early	Present	Early	Present
EPDM								
Early-E263	75%		--		85%		57%	
Present-267		97%		105%		100%		103%
FKM								
Early-115	14%		46%		73%		32%	
Present-255		72%		101%		100%		55%
EPDM/FKM								
Early-235	80%		63%		110%		128%	
Present-266		96%		84%		101%		96%
Propylene-TFE								
Early-268	77%		112%		90%		84%	
Present-291		93%		118%		100%		87%

Table II

Geothermal elastomers progress.

Elastomer	Ult. Tensile @ RT. psi		Ult. Elong. @ RT %		Shore A Hard @ 260°C		Extrusion Resist @ 260°C, psia	
	V	CA	V	CA	V	CA	V	CA
EPDM								
Early-E263	1363	1020	>840	>840	61	52	9.6	5.5
Present-267	1610	1554	141	148	92	92	17.1	17.6
FKM								
Early-115	1699	244	162	74	80	58	11.2	3.6
Present-255	2195	1591	224	226	79	79	11.4	6.3
EPDM/FKM								
Early-235	1882	1506	681	431	67	74	8.3	10.6
Present-266	1596	1531	193	163	88	89	18	17.3
Propylene-TFE								
Early-268	1673	1289	394	442	69	63	6.7	5.6
Present-291	2335	2162	134	158	95	95	17.4	15.2

APPENDIX K

PROCEEDINGS

OF THE

INTERNATIONAL CONFERENCE ON

GEOTHERMAL DRILLING

AND

COMPLETION TECHNOLOGY

Co-sponsored by
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HIGH TEMPERATURE GEOTHERMAL ELASTOMER COMPOUND DEVELOPMENT

Alan R. Hirasuna
L'Garde, Inc.

United States

ABSTRACT

Reliable casing packer seal elastomers for the unusually severe geothermal environment at 260°C (500°F) did not exist in 1976. L'Garde, Inc., was awarded a contract to fulfill this need by the United States Department of Energy. Successful development was completed in 1979. Compounds based on four different polymer systems were developed, all of which exceed the contract requirements. Successful laboratory tests above 300°C (575°F) have been performed with packer seals. Field tests to temperatures as high as 317°C (603°F) have been performed on static O-rings in a cablehead. Successful, dynamic, drill bit seal tests were run with a presoak temperature of 288°C (550°F).

The successful compounds are based on the following polymer systems: EPDM (Nordel®), FKM (Viton®), EPDM/FKM (Nordel®/Viton®) blend, and propylene-TFE (AFLAS®). Evaluation was performed on full-scale packer seals as well as on O-rings by L'Garde. L'Garde ran comparison tests against commercially available elastomers including Kalrez®. Case histories of other organizations which have laboratory and/or downhole experience with one or more of the developed compounds are presented. The applications cited include blowout preventors, dynamic drill bit seals, packers, and cableheads. Finally, an effort to actively transfer this technology to the industry is described.

INTRODUCTION

In late 1976 L'Garde, Inc. was awarded a contract by the United States Department of Energy (DOE), DEAC03-77-ET28309 (formerly EG-77-C-03-1308), the Geo-Elastomeric Materials (GEM) Program [1,2]. By that time the DOE concluded that there was a general need for improved elastomers for various seal applications which were required to operate in the unusually severe geothermal environment. Logging equipment could not be run for fear of losing seals and damaging the instrumentation. Packers could not be run without first cooling the well. Rotating well head seals and BOP seals required frequent replacement. There was a general need for higher temperature, more hydrochemically and extrusion resistant elastomers and for that matter the need is still not satisfied in practice today in the geothermal field.

L'Garde performed an initial task to identify what elastomer application when upgraded to geothermal requirements would be of greatest benefit to the geothermal community. The formation packer was identified for two functions, hydraulic stimulation and interval testing in support of reservoir and well characterization. However, though high temperature geothermal formation packers are needed, development of elastomers for this application was judged too ambitious a goal for the first effort. Therefore, it was recommended and approved to first develop improved elastomers for casing packer seal elements. In addition, rubber development for the casing packer element "kills two birds with one stone", as those elastomers which work successfully for the packer seal will most probably work well for static O-ring seals. The O-ring application is less severe because both surfaces are machined, the clearances are small and the required deformation of the O-ring is small compared to a casing packer seal element.

In 1979 after the completion of the elastomer compound development, L'Garde was awarded a contract to actively transfer the developed technology to industry. The transfer is sponsored by the DOE through Brookhaven National Laboratory under contract BNL 490316-S, Geothermal Elastomeric Materials Technology Transfer.

This paper presents the results of the elastomer compound development for high temperature geothermal casing packer seals, case histories of laboratory and field experience by other organizations, and a description of the present technology transfer effort.

GEOTHERMAL PACKER ELASTOMER COMPOUND DEVELOPMENT

The objectives of the elastomer development contract were successfully met. Though meeting the fundamental objective, to develop a casing packer seal elastomer compound which will operate in a 260°C (500°F) geothermal environment, was highly challenging and carried with it a definite probability of failure, L'Garde was fortunate to be able to conclude the program with very positive results.

Packer seals which perform well in the oil field up to temperatures of about 150°C (302°F) run into reliability problems at higher temperatures especially when they get as high as 260°C. Though the stresses induced into the elastomeric element are substantially the same as at the lower temperatures, the loss in elastomer strength at 260°C is substantial and, typically, the stress exceeds the reduced strength and failure occurs. Other common problems include hardening or reversion softening of the elastomer at higher temperatures with associated failure. The geothermal elastomeric packer seal problem at 260°C is significant and even the less demanding static O-ring seal is still a problem. Figure 1 rather lucidly illustrates the starting point of the geothermal packer seal development. These post mortem test specimens were run at 260°C in synthetic brine. At these unusually severe conditions, the seals did one of three things; extruded severely (B3), could not withstand the pack-off and cracked without sealing (220-3), or extruded and broke catastrophically (D3 and E3). For about the first 2/3 of the contract, it was doubtful that elastomer improvements alone would provide reasonable performance at the test conditions at 260°C.

The 2-phase Screening and Simulation Test evaluation scheme with the special tests proved invaluable and enabled efficient evaluation of 129 compounds with quick feedback for subsequent compounding efforts. 96 seal Simulation Tests were run which provide data which is closely correlated with operational performance with little need for uncertain extrapolation to judge how compounds will perform operationally. The testing and evaluation scheme is described below and is detailed in References 1 and 2.

Screening Tests -- A battery of simple tests were defined for broad brush screening of compounds. All screening tests used simple specimens cut from standard test slabs and comparisons between virgin specimens and specimens aged in synthetic geothermal brine at 260°C were made to assess the hydrothermochemical stability of the compounds. The synthetic geothermal brine was defined and developed specifically for the elastomers development. The constituency levels include almost the entire population of known geothermal resource areas. This constituency specifically avoids extremely high levels measured in some locations which correspond to a small fraction of the population. The synthetic brine is an aqueous solution of 300 ppm H_2S, 1000 ppm CO_2 and 25,000 ppm NaCl. The starting pH is about 5. To specifically address the important problem, extrusion, a special test was designed and fabricated which measured the extrusion resistance of a specimen about the size of a quarter.

A good quantitative demonstration of the compounding accomplishments is seen through a comparison of characteristics for early compounds against commensurate ones at the completion of the program. This can be clearly seen by examining the data generated under the Screening Tests. The most pertinent parameters measured are ultimate tensile strength, ultimate elongation, Shore A hardness, and extrusion resistance. The tensile and elongation were measured at room temperature while the hardness and extrusion resistance were measured at 260°C. Table I illustrates the improvements as indicated by the percent of each of the four critical characteristics retained after ageing in synthetic brine for 22 hours. Table II lists the virgin and chemically aged values which are represented by the percentage numbers.

Note that there is significant improvement reflected by the latest compounds. The 267 EPDM is virtually unchanged by the 22 hour ageing in the synthetic brine with respect to all four characterization parameters indicating phenomenal hydrothermochemical stability in the synthetic brine. Vast improvement was finally accomplished with the 255 FKM. There is room for improvement on the extrusion resistance especially, as is reflected by the packer seal Simulation Tests, however, the extrusion resistance is adequate to meet the requirements and the results are quite respectable. The 266 EPDM/FKM is very stable with some minor degradation of the ultimate elongation. The 291 Propylene-TFE shows some minor hydrothermochemical instability in the ultimate elongation. In general, all of the latest formulations showed very good stability considering the unusual severity of environment.

The above data and tests are all comprehensively reported in Reference 1.

Packer Seal Simulation Tests -- Those compounds which performed well in the screening were then graduated to full-scale packer seal tests. A laboratory simulator was designed and fabricated which subjects a full-scale 10.2 cm (4-inch) packer seal to simulated downhole environments, in the laboratory. In this test the seal is simultaneously subjected to the chemistry, temperature, mechanical forces, pressure forces, and limited surface exposure of the seal just as it is on an operational packer. This provides the full spectrum of interactive operational environments, the combination of which is important when evaluating materials being stressed to their ultimate capacity.

Upon conclusion of the development, compounds based on four (one was required) separate polymer systems molded into full-scale packer seals successfully sealed in laboratory packer SIM Tests in excess of the prescribed duty cycle -- temperature of 260°C, in synthetic geothermal brine, differential pressure of 21.4-28.3 MPa (3100 to 4100 psi), and for 24 hours. The four systems were based on the following polymers: EPDM, FKM, EPDM/FKM Blend, and Propylene-TFE. All four elastomer systems successfully exceeded the contract objectives and show definite improvement over elastomer compounds which are commercially available. Over the duration of the program, measurable improvement of the hydrothermochemical stability was achieved in the synthetic geothermal brine.

Figure 2 shows post mortem test specimen of one of the better, earlier EPDM compounds, seal 217-II-1. This element sealed successfully at 260°C, but did suffer a significant level of extrusion. In contrast, one of the latest compounds is shown, post mortem seal 275-1, which is an EPDM/FKM Blend. Only a minor amount of permanent deformation resulted and no extrusion; the seal had an excellent appearance when removed from the test. This element sealed successfully under the same conditions as 217-II-1 except even a higher differential pressure, 24.8 vs. 21.4 MPa (3600 vs. 3100 psi).

O-Ring Simulation Tests -- Another good quantitative demonstration of the accomplishments is the O-ring SIM Test data which compares the developed compounds against a limited number of commercially available compounds. The packer SIM Test was modified for static simulation testing of O-rings. The 2-329 O-ring groove is of standard commercial dimensions with no back-ups. The measured clearance is 0.229 mm (0.009 in.), quite severe for the test conditions. The O-rings were tested at 260°C in the synthetic geothermal brine starting at 28.3 MPa (4100 psi) differential pressure. The test was set for 22 hours and if a compound failed before 22 hours, the differential pressure was decreased by 6.9 MPa (1000 psi) and the test rerun with a new seal. The reduction in differential pressure progressed to 14.5 MPa (2100 psi) where the testing stopped if the compound failed.

Because this is an unusually severe environment, the GEM compounds have a distinct advantage in that they were developed specifically for this condition. Figure 3 summarizes the resulting capability measured for each of the compounds. The GEM 255 FKM and 267 EPDM compounds performed better than their commercial counterparts. The 255 FKM held for the entire test period at 21.4 MPa (3100 psi) while the commercial counterparts both broke at 14.5 MPa (2100 psi). The GEM 267 EPDM looked excellent after the 28.3 MPa (4100 psi) run and is capable of higher differential pressures while its counterparts showed incipient failure. There were no commercially comparable counterparts for the EPDM/FKM Blend or the Propylene-TFE, both ran the full 22 hours at the full differential pressure. The Blend looked as good as the 267 EPDM and, hence, is also capable of higher differential pressures and the Propylene-TFE suffered minor extrusion. There was no counterpart to the DuPont Perfluoroelastomer (Kalrez^R), however, it is of keen interest because it is one of the more chemically stable polymers. It is very expensive but its expense is justifiable in some situations because of the longer life it renders. For the synthetic geothermal brine environment the 3074 Perfluoroelastomer did not fare as well as the four developed compounds in the O-ring test, and earlier coupon ageing tests were made on 3073 Perfluoroelastomer which indicate that it is not as hydrothermochemically stable as the developed compounds. Specific compounding for the geothermal brine environment may result in similar improvements as was herein accomplished.

Figure 4 is a composite photograph of the more interesting seals; 267 EPDM, E-692-75 EPDM, 266 EPDM/FKM Blend, and the Perfluoroelastomer 3074. All seals ran successfully for 22 hours at 28.3 MPa (4100 psi) differential pressure except the Perfluoroelastomer 3074 which ran at 14.5 MPa (2100 psi) differential pressure.

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Note that the 267 EPDM and the 266 EPDM/FKM Blend look excellent, although some permanent squaring of the O-rings did occur. Both the E-692-75 EPDM and the Perfluoroelastomer 3074 are seen to have suffered damage.

CASE HISTORIES OF OTHER ORGANIZATIONS

Since the completion of the elastomer compound development there have been opportunities for other organizations to expose the favored compounds to various environments. Test slabs, O-rings, and packer seals were molded by L'Garde and provided to the other organizations for independent testing.

Sandia National Laboratories, Laboratory BOP Simulation -- Sandia [3] investigated promising elastomers for the cable blowout preventor (BOP) application. They tested tensile specimens in the vapor phase of steam at 260°C (500°F) for 100 hours plus a substantial heat-up and cool-down time of about 14 hours. On their summary test they exposed 7 compounds, 4 of which were provided by L'Garde.

The compounds tested are shown in Table III which is excerpted from Reference 3. The 4 compounds provided by L'Garde were the best 4 out of 7 tested. The E60C FKM and the 914-80 both became so brittle they could not be measured without their breaking. The only other nonL'Garde compound which survived was the GH FKM which experienced a 72% loss of tensile strength. The L'Garde compounds ranged from 9 to 40% loss of tensile strength.

TerraTek, Laboratory Drill Bit Seal Simulation -- TerraTek [4] is a DOE contractor and has developed a dynamic drill-bit seal tester. It tests a rotating seal simulating the journal seal of a tricone bit. On each revolution a jog is superimposed on the rotational motion. Tests are typically run at 90 RPM with rock cuttings, sand, and water on one side of the seal, and grease on the other.

O-ring seals made from the L'Garde static packer seal compounds were provided to TerraTek for dynamic drill bit seal testing. There was concern about testing the static packer seal compounds in this environment because there was no requirement or attention paid to dynamic seal properties when they were compounded. Furthermore, there was concern about the exposure to hydrocarbon grease, another environmental consideration outside the realm of the geothermal packer seal development.

Because of the concern about the exposure to hydrocarbon grease it was first recommended that TerraTek test the 266 EPDM/FKM Blend as EPDM's are generally vulnerable to hydrocarbon, hence, testing blend compounds seemed prudent to gain the oil resistance of the FKM. The 266 Blend compound was tested at a time when 40 to 50 hours of successful sealing was very good performance. To the pleasant surprise of everyone concerned the static packer seal Blend compound sealed for 47 hours.

Additional tests were then made with the 267 EPDM compound. A presoak temperature of 288°C (550°F) was agreed to, this was the first time that the test was run at this high a temperature. Apparently the grease and temperature did not adversely affect the 267 EPDM seal as it was successfully sealing after 104 hours when the test was stopped. The post test seal was described by TerraTek personnel as looking surprisingly unaffected by the environment. It did, however, abrade and wear significantly on the debris side of the seal.

Otis Engineering Corp., Packer Laboratory and Downhole Tests -- Compound 267 EPDM packer seals were molded by L'Garde for tests by Otis Engineering Corp. [5]. Molded seals for 7-inch, 26 lb/ft casing were provided and tested by Otis at 302°C (575°F) and 20.7 MPa (3000 psi) differential pressure in their laboratory packer simulation tester. The elements sealed successfully for five days when the test was stopped. The seal was still elastomeric and did not show any signs of reversion softening or scission. Prior to this test Otis tested an EPDM from another firm at a lower temperature, 288°C (550°F), and the seal failed because it lost strength due to reversion. The post mortem seal did not have any form and looked like a lump of tar.

Subsequent to the laboratory tests, Otis installed a 267 EPDM element on a new R&D packer and ran it in a heavy oil field in Bakersfield. The well has 7-inch casing and is used for continuous steam injection. The steam is about 246°C (475°F) at the boiler and 204°C (400°F) at the well head.

The packer was pulled after 5 months of successful sealing to perform well maintenance. Tracer tests confirmed that the packer was still sealing prior to removal. The packer was pulled with no problems whatsoever, where usually difficulty is encountered with the seal jamming and either being rolled or ripped off the packer.

The appearance of the recovered seal was excellent with virtually no permanent deformation whatsoever. It looked like a new seal and obviously could have remained in operation indefinitely. There was no apparent degradation from the crude oil which covered the packer when it was removed. Figure 5 is a photograph of the packer seal element after removal from the Bakersfield well.

Los Alamos Scientific Laboratory (LASL), Laboratory and Downhole Cablehead Tests -- LASL [6] is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock Project. They have shown feasibility of the concept at 200°C (392°F) and are currently going to demonstrate the concept with deeper wells and higher temperatures, 275°C (525°F) minimum. Because of the higher temperatures LASL is concerned about elastomeric parts in various well and logging tools. They awarded L'Garde a contract to support them with the developed high-temperature elastomer expertise.

L'Garde fabricated O-rings and a cable bend protector from the 267 EPDM compound for the LASL cablehead. LASL tested the cablehead in an autoclave with Mobil One oil inside the cablehead and water outside of it. They ran 24 hour cycles with the temperature on for 8 hours and off for 16, this simulates tripping in and out of the hole. The best prior performance they achieved was with commercial fluoroelastomer O-rings which literally disentegrated after one cycle. They ran the 267 EPDM for 4 cycles or 4 days and stopped the tests to examine the seals. The temperature was 275°C (525°F) minimum with an excursion to 340°C (644°F) during one of the 24 hour cycles. The pressure was 51.7 MPa (7500 psi). The seals looked good and were sealing after 4 cycles, although there was some swelling from the high-temperature oil and some permanent deformation as would be expected for these conditions. They hope to go ten cycles operationally before they must change out the O-rings.

Subsequent to the autoclave tests LASL ran the cablehead with the 267 EPDM parts on a temperature probe in the new hotter well. They measured a near bottomhole temperature of 317°C (603°F) with the probe. After this run the O-rings and bend protector were examined and reported to be in excellent condition for the imposed environment.

These tests are landmark tests because this is the first time the 267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range, the first time the 267 EPDM was tested in 100% oil, the first time the 267 EPDM was tested in oil at extreme temperatures (excursions to 340°C (644°F)), and the first time the 267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDM's are generally not used in oil environments because they are extremely vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the GEM 267 EPDM is less vulnerable to oil than other EPDM's.

TECHNOLOGY TRANSFER

Toward the final stages of the geothermal compound development, it was becoming evident that the technology developed represented significant progress in high-temperature elastomer capability. The standard procedure is to conclude an effort such as this with publication and distribution of a final report. However, in this instance the DOE felt it worthwhile to expend additional effort to transfer the technology to industry to further assure that benefit to the industry will be more generally widespread.

Applications for technology transfer were solicited from companies in the well tool industry. There were advertisements placed in the Geothermal Energy Magazine and the Petroleum Engineer International, and there was a mailing list of over 300. Applications were received and three companies were selected for technology transfer and three additional companies were placed on a back-up list should funds be available. The companies are as follows:

Selected For Transfer

B. J. Hughes Rubber Division, Commerce, CA
Oncor Drilling Tools, Houston, TX
(Precision Rubber, Lebanon, TN)
Parker Seal, Culver City, CA

Back-Up List

NL Rig Equipment, Stafford, TX
Tri-State Oil Tool, Inc., Bossier City, LA
Vernay Laboratories, Yellow Springs, OH

In that Oncor Drilling Tools purchases their molded rubber parts from vendors, they have selected Precision Rubber to receive the technology in their behalf. L'Garde is transferring the technology directly to Precision.

The transfer process is one where progressively more and more of the processing is done by the transferee with checks to assure that the elastomer physicals are being reproduced. When there is confidence that reproducibility exists with elastomer that is mixed, molded, and post-cured by the transferee with his raw materials, equipment, and personnel; then full-scale parts will be fabricated and tested under laboratory conditions and/or in the actual downhole environment. The applications that are currently planned for testing are downhole electric pump seals, drill bit seals, drilling shock-sub seals, drill pipe protectors and blow-out preventor ram rubbers.

Molded rubber components made from the developed compounds are anticipated to be available to industry on a commercial basis through the technology transferees. In addition, a workshop discussing the transfer case histories is tentatively planned for 1981 for all who are interested.

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the United States Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research and development, and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE as did Mr. A. J. Adduci of the San Francisco office of DOE as the technical administrator. Dr. L. E. Kukacka is currently providing valuable guidance on the Technology Transfer effort.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory and Dr. J. C. Rowley of Los Alamos Scientific Laboratory were especially helpful.

REFERENCES

[1]Hirasuna, A. R., et al., July 1979, "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976 - 30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U. S. Department of Energy, Geothermal Energy.

[2]Hirasuna, A. R., et al., "Evaluation Tests for Geothermal Casing Packer Seal Elastomers," presented at a meeting of the American Institute of Chemical Engineers, Philadelphia, Pennsylvania, 4-8 June 1978.

[3]Arnold, C., Jr., "Memo to Distribution," Sandia Laboratories, 19 April 1979, Albuquerque, New Mexico.

[4]Winzenried, R., "Letter to Hirasuna," TerraTek, 15 June 1979, Salt Lake City, Utah.

[5]Taylor, D., Private Communication, Otis Engineering Corp., 23 April 1979, Dallas, Texas.

[6]Archuleta, J. and Dennis, B., Private Communications, Los Alamos Scientific Laboratory, 9 July 1980, 4 August 1980, Los Alamos, New Mexico.

Table 1
Progress of Hydrothermochemical Stability of Geothermal Elastomers

Elastomer	Ult. Tensile @ RT		Ult. Elong. @ RT		Shore A Hard @ 260°C		Extrusion Resist. @ 260°C	
	Early	Present	Early	Present	Early	Present	Early	Present
EPDM Early-E263 Present-267	75%	97%	--	105%	85%	100%	57%	103%
FKM Early-115 Present-255	14%	72%	45%	101%	73%	100%	32%	55%
EPDM/FKM Early-235 Present-266	80%	96%	63%	84%	110%	101%	128%	96%
Propylene-TFE Early-268 Present-291	77%	93%	112%	118%	90%	100%	84%	87%

Table 2
Geothermal Elastomers Progress

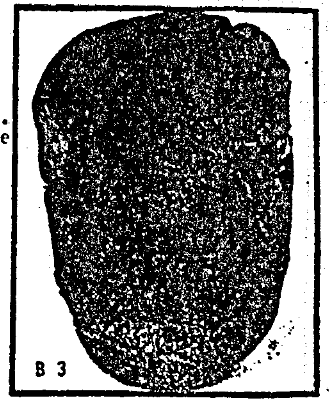
Elastomer	Ult. Tensile @ RT. psi		Ult. Elong. @ RT %		Shore A Hard @ 260°C		Extrusion Resist @ 260°C, psia	
	V	CA	V	CA	V	CA	V	CA
EPDM Early-E263 Present-267	1363 1610	1020 1554	>840 141	>840 148	61 92	52 92	9.6 17.1	5.5 17.6
FKM Early-115 Present-255	1699 2195	244 1591	162 224	74 226	80 79	58 79	11.2 11.4	3.6 6.3
EPDM/FKM Early-235 Present-266	1882 1596	1506 1531	681 193	431 163	67 88	74 89	8.3 18	10.6 17.3
Propylene-TFE Early-268 Present-291	1673 2335	1289 2162	394 134	442 158	69 95	63 95	6.7 17.4	5.6 15.2

Table 3
Sandia Cable BOP Tests

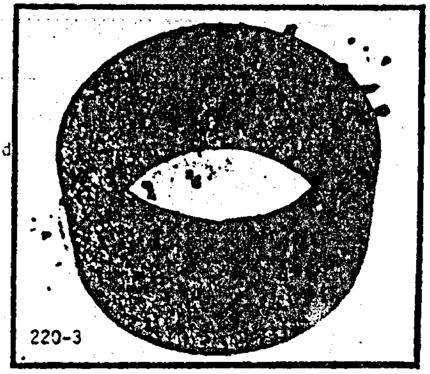
Elastomer Designation	Source	Control		Aged		Comparison	
		Tensile Str. (psi)	Elong. (%)	Tensile Str. (psi)	Elong. (%)	Δ Tensile (%)	Δ Elong. (%)
267 EPDM	L'Garde	1395	70.2	1261	60.2	-9	-14.9
266 EPDM/FKM Blend	L'Garde	1596	111	1410	71.2	-11	-36.7
291 Propylene/PTFE	L'Garde	2321	170	1799	87.4	-22	-48
255 FKM	L'Garde	2312	121	1376	135	-40	+11.6
GH FKM	DuPont Formula	1301	172	364	45	-72	-74
E60C FKM	DuPont Formula	864	145	BROKE	BROKE	BROKE	BROKE
914-80?	BOP Manufact.	882	148	BROKE	BROKE	BROKE	BROKE

Fig. 1 -- SIM Test post mortem specimens typical early results.

Company B EPDM -- This and L'Garde EPDM remained elastomeric and survived full 24 hours. Only difference L'Garde EPDM extruded 2.5 in. vs company B's 4.0 in.



L'Garde Peroxide-Cured Fluoroelastomer -- Failed because of inability to elongate sufficiently during seating.



Company D Epichlorohydrin -- Lost all structural strength and became soft and crumbly before seating.

Company E Non-Peroxide-Cured Fluoroelastomer -- Failed by breaking in the high stress region. Survived 3.75H with slow leak, then failed.

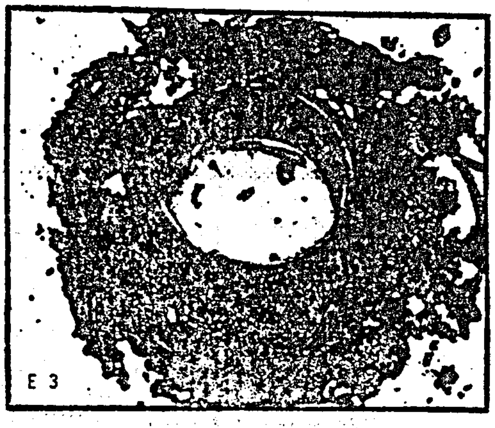


Fig. 2 -- Post mortem packer seals after 22 hrs. in synthetic geothermal brine contrasting earlier and current best formulations.

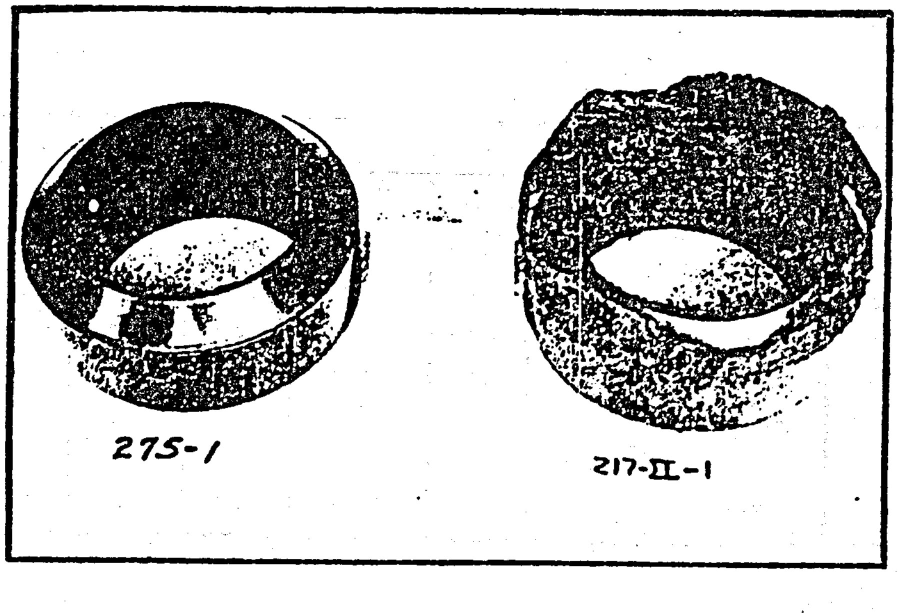


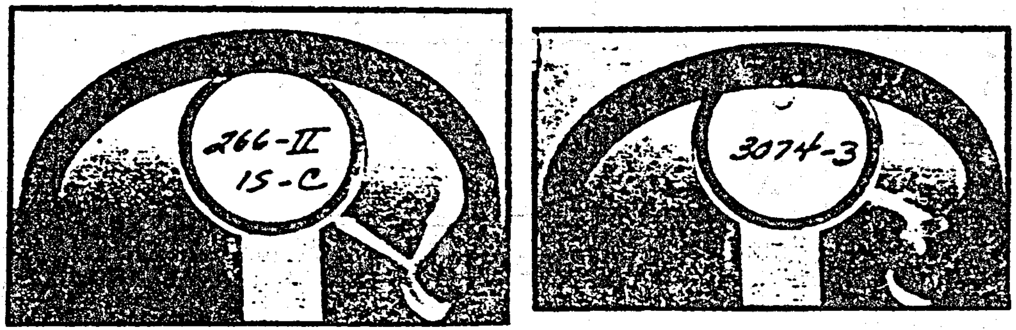
FIGURE 3 -- GEM O-RING SIM TESTS
SYN. BRINE AT 260°C (500°F)

TYPE	COMPOUND / DIFFERENTIAL PRESSURE	14.5 MPa (2100 PSI)	21.4 MPa (3100 PSI)	28.3 MPa (4100 PSI)	?
FLUOROELAST.	L'GARDE GEM 255 FKM.		22H	6.25H	
	PARKER V-709-90 FKM	10.5H	9H	4H	
	PARKER V-747-75 FKM	5.25H		0.2H	
EPDM	L'GARDE GEM 267 EPDM			22H	
	PARKER E-692-75 EPDM			22H	
MISC.	L'GARDE GEM 266 EPDM/FKM BLEND			22H	
	L'GARDE GEM 291 PROPYLENE-TFE			22H	
	DUPONT 3074 PERFLUORO ELAS.	22H	3.75H	0.75H	

Fig. 4 -- Post Mortem O-rings SIM Test Seals After 22 Hrs. In Sour Synthetic Geothermal Brine @ 500^{o}F.

267 EPDM, ΔP = 28.3 MPa (4,100 psi)

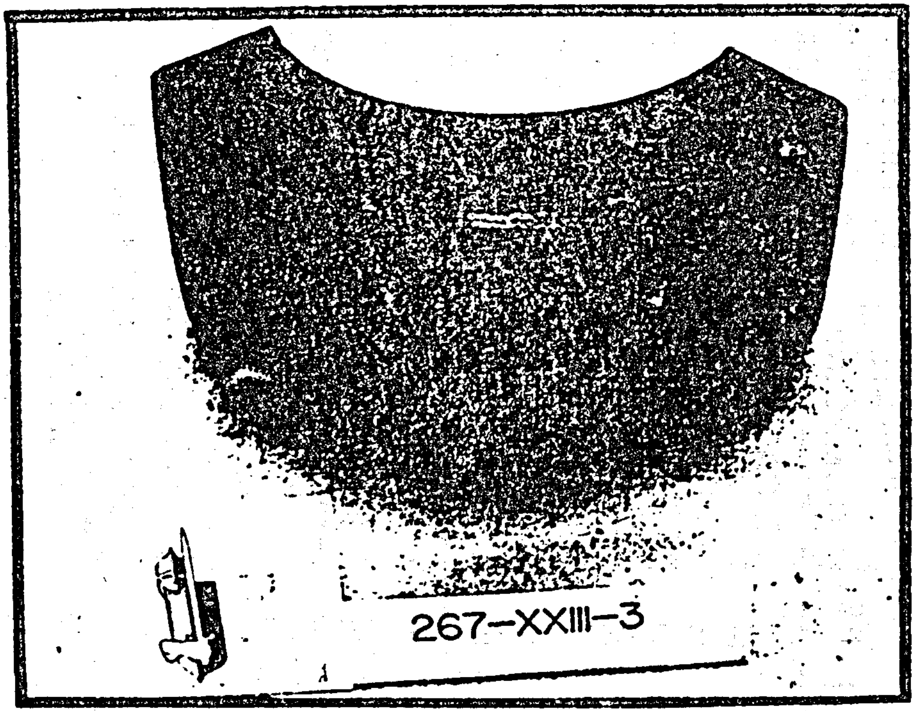
E-692-75 EPDM, ΔP = 28.3 MPa (4,100 psi)



266 EPDM/FKM Blend, ΔP = 28.3 MPa (4,100 psi)

3074 Perfluoro, ΔP = 14.5 MPa (2,100 psi)

Fig. 5 -- EPDM After 5 Months In Bakersfield Steam Injection Well For Heavy Oil.



APPENDIX L

CORROSION/81
National Association of Corrosion Engineers
Toronto, Ontario, Canada

6-10 April 1981

This paper was subsequently published in Materials Performance, See Appendix M.

APPENDIX M

AN OFFICIAL NACE PUBLICATION

A Reprint from Materials Performance

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Development of Elastomer Compounds for High Temperature Service

ALAN R. HIRASUNA and CLIFFORD A. STEPHENS

• P. O. Box 218340 • Houston, Texas 77218

Development of Elastomer Compounds for High Temperature Service*

ALAN R. HIRASUNA and CLIFFORD A. STEPHENS*

Reliable casing packer seal elastomers for the unusually severe geothermal environment at 260 C (500 F) did not exist in 1976. Successful development was completed in 1979. Compounds based on four different polymer systems were developed which all exceed the contract requirements. Successful laboratory tests above 300 C (575 F) have been performed with packer seals. Field tests to as high as 317 C (603 F) have been performed as static O-rings in a cablehead. Successful dynamic drill bit seal tests were run with a presoak temperature of 288 C (550 F). The successful compounds are based on the following polymer systems: EPDM (Nordel®), FKM (Viton®), EPDM/FKM (Nordel®/Viton®) blend, and propylene-TFE (AFLAS®). Evaluation was performed on full-scale packer seals as well as O-rings. We ran comparison tests against commercially available elastomers including Kalrez®. Case histories of other organizations which have laboratory and/or downhole experience with one or more of the developed compounds are presented. The applications include blowout preventors, dynamic drillbit seals, packers, and cableheads. Finally, an effort to actively transfer this technology to the industry is described.

Introduction

BY LATE 1976, THE DEPARTMENT OF ENERGY (DOE) concluded that there was a general need for improved elastomers for various seal applications which were required to operate in the usually severe geothermal environment. Logging equipment could not be run for fear of losing seals and damaging the instrumentation. Packers could not be run without first cooling the well. Rotating well head seals and BOP seals required frequent replacement. There was a general need for higher temperature, more hydrochemically and extrusion resistant elastomers, and for that matter, the need is still not satisfied in practice today in the geothermal field.

L'Garde was selected to develop improved elastomer compounds for the geothermal needs. An initial task was performed to identify what elastomer application when upgraded to geothermal requirements would be of greatest benefit to the geothermal community. The formation packer was identified for two functions, hydraulic stimulation and interval testing in support of reservoir and well characterization. However, though high temperature geothermal formation packers are needed, development of elastomers for this application was judged too ambitious a goal for the first effort. Therefore, it was recommended and approved to first develop improved elastomers for casing packer seal elements. In addition, rubber development for the casing packer element "kills two birds with one stone," as those elastomers whch work successfully for the packer seal will most probably work well for static O-ring seals. The O-ring application is less severe because both surfaces are machined, the clearances are small, and the required deformation of the O-ring is small compared to a casing packer seal element.

This paper presents the results of the elastomer compound development for high temperature geothermal casing packer seals which was completed in 1979, case histories of laboratory and field experience by other organizations, and a description of the present technology transfer effort.

Geothermal Packer Elastomer Compound Development

Packer seals which perform well in the oil field up to temperatures of about 150 C (302 F) run into reliability problems at high temperatures especially when they get as high as 260 C. Though the stresses induced into the elastomeric element are substantially the same as at the lower temperatures, the loss in elastomer strength at 260 C is substantial and, typically, the stress exceeds the reduced strength and failure occurs. Other common problems include hardening or reversion softening of the elastomer at higher temperatures with associated failure. The geothermal elastomeric packer seal problem at 260 C is significant, and even the less demanding static O-ring seal is still a problem. Figure 1 rather lucidly illustrates the starting point of the geothermal packer seal development. These post mortem test specimens were run at 260 C in synthetic brine. At these unusually severe conditions, the seals did one of three things: extruded severely (B3); could not withstand the packoff and cracked without sealing (220-3); or extruded and broke catastrophically (D3 and E3). For the first 2/3 of the contract, it was doubtful that elastomer improvements alone would provide reasonable performance at the test conditions at 260 C.

The 2-phase screening and simulation test evaluation scheme with special tests proved invaluable, and enabled efficient evaluation of 129 compounds with quick feedback for subsequent compounding efforts. Ninety-six seal simulation tests were run which provide data which is closely correlated with operational performance with little need for uncertain extrapolation to judge how compounds will perform operationally. The testing and evaluation scheme is described below and is detailed in References 1 and 2.

Screening Tests

A battery of simple tests were defined for broad brush screening of compounds. All screening tests used simple specimens cut from standard test slabs and comparisons between virgin specimens and specimens aged in synthetic geothermal brine at 260 C were made to assess the hydrothermochemical stability of the compounds. A synthetic geothermal brine was developed specifically for the elastomer development. The constituency levels include almost the entire population of known geothermal resource areas. This constituency specifically avoids extremely high levels measured in some locations which correspond to a small fraction of the

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•Presented during Corrosion/81 (Paper No. 226), April, 1981, Toronto, Ontario.

*L'Garde, Inc., Newport Beach, California.

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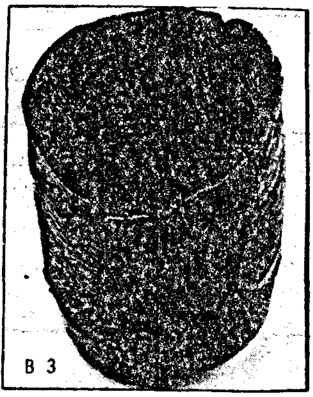
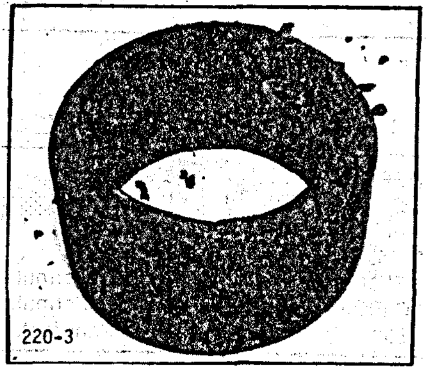
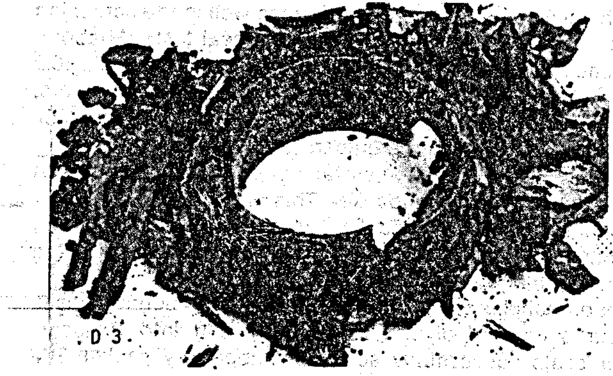


Our Peroxide-Cured Fluoroelastomer—Failed because of inability to elongate sufficiently during seating.



Company D Epichlorohydrin—Lost all structural strength and became soft and crumbly before seating.



Company E Non-Peroxide-Cured Fluoroelastomer—Failed by breaking in the high stress region. Survived 3.75H with slow leak, then failed.

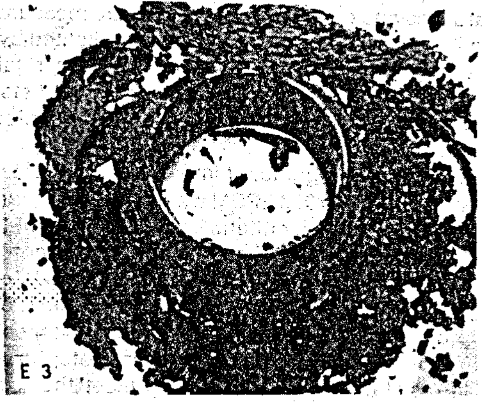


FIGURE 1 — SIM test post mortem specimens typical early results.

TABLE 1 — Progress of Hydrothermochemical Stability of Geothermal Elastomers

Elastomer	Ult. Tensile @ RT Early (%)	Ult. Tensile @ RT Present (%)	Ult. Elong. @ RT Early (%)	Ult. Elong. @ RT Present (%)	Shore A Hard @ 250 C Early (%)	Shore A Hard @ 250 C Present (%)	Extrusion Resist. @ 260 C Early (%)	Extrusion Resist. @ 260 C Present (%)
EPDM								
Early-E263	75		—		85		57	
Present-267		97		105		100		103
FKM								
Early-115	14		46		73		32	
Present-255		72		101		100		55
EPDM/FKM								
Early-235	80		63		110		128	
Present-266		96		84		101		96
Propylene-TFE								
Early-268	77		112		90		84	
Present-291		93		118		100		87

population. The synthetic brine is an aqueous solution of 300 mg/L H_2S, 1000 mg/L CO_2, and 25,000 mg/L NaCl. The starting pH was about 5. To specifically address the important problem, extrusion, a special test was designed which measured the extrusion resistance of a specimen about the size of a quarter (25 × 1.5 mm).

A good quantitative demonstration of the compounding accomplishments is seen through a comparison of characteristics of early compounds against commensurate ones at the completion of the program. This can be clearly seen by examining the data from the screening tests. The most pertinent parameters measured were ultimate tensile strength, ultimate

TABLE 2 — Geothermal Elastomers Progress

Elastomer Designation	Source	Control Tensile Str. (psi)	Control Elong. (%)	Aged Tensile Str. (psi)	Aged Elong. (%)	Comparison △ Tensile (%)	Comparison △ Elong. (%)
267 EPDM	L'Garde	1395	70.2	1261	60.2	−9	−14.9
266 EPDM/FKM Blend	L'Garde	1596	111	1410	71.2	−11	−36.7
291 Propylene/PTFE	L'Garde	2321	170	1799	87.4	−22	−48
255 FKM	L'Garde	2312	121	1376	135	−40	+11.6
GH FKM	DuPont Formula	1301	172	364	45	−72	−74
E60C FKM	DuPont Formula	864	145	broke	broke	broke	broke
914-80?	BOP Manufact.	882	148	broke	broke	broke	broke

elongation, Shore A hardness, and extrusion resistance. The tensile and elongation were measured at room temperature, while the hardness and extrusion resistance were measured at 260 C. Table 1 illustrates the improvements—indicated by the percent of each of the four critical characteristics retained after aging in synthetic brine for 22 hours. Table 2 lists the virgin and chemically aged values which are represented by the percentage numbers.

Note that there was significant improvement reflected by the latest compounds. The 267 EPDM was virtually unchanged by the 22 hour aging in the synthetic brine with respect to all four characterization parameters, indicating phenomenal hydrothermochemical stability in the synthetic brine. Vast improvement was finally accomplished with 255 FKM. There is room for improvement on the extrusion resistance especially, as is reflected by the packer seal simulation tests; however, the extrusion resistance was adequate to meet the requirements and the results were quite respectable. The 266 EPDM/FKM was very stable with some minor degradation of the ultimate elongation. The 291 Propylene-TFE showed some minor hydrothermochemical instability in the ultimate elongation. In general, all of the latest formulations showed very good stability considering the unusual severity of environment.

The above data are all comprehensively reported in Reference 1.

Packer Seal Simulation Tests

Those compounds which performed well in the screening were then graduated to full-scale packer seal tests. A laboratory simulator was fabricated which subjects a full-scale 10.2 cm (4 inch) packer seal to simulated downhole environments, in the laboratory. In this test, the seal is simultaneously subjected to the chemistry, temperature, mechanical forces, pressure forces, and limited surface exposure of the seal just as it is on an operational packer. This provided the full spectrum of interactive operational environments, the combination of which is important when evaluating materials being stressed to their ultimate capacity.

Upon conclusion of the development, compounds based on four separate polymer systems molded into full-scale packer seals successfully sealed in laboratory packer SIM Tests in excess of the prescribed duty cycle—temperature of 260 C, in synthetic geothermal brine, differential pressure of 21.4 to 28.3 MPa (3100 to 4100 psi), and for 24 hours. The four systems were based on the following polymers: EPDM, FKM, EPDM/FKM blend, and Propylene-TFE. All four elastomer

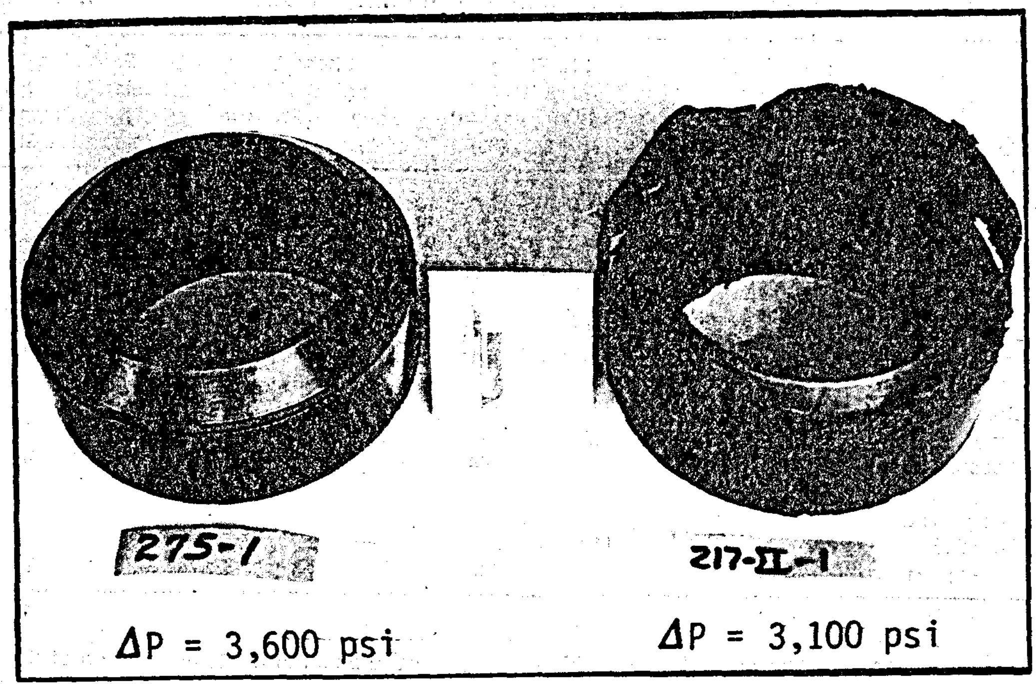


FIGURE 2 — Post mortem packer seals after 22 hours in synthetic geothermal brine contrasting earlier and current best formulations.

systems successfully exceeded the contract objectives and show definite improvement over elastomer compounds which are commercially available. Over the duration of the program, measurable improvement of the hydrothermochemical stability was achieved in the synthetic geothermal brine.

Figure 2 shows post mortem test specimen of one of the better, earlier EPDM compounds, seal 217-II-1. This element sealed successfully at 260 C, but did suffer a significant level of extrusion. In contrast, one of the latest compounds is shown, post mortem seal 275-1, which is an EPDM/FKM blend. Only a minor amount of permanent deformation occurred and no extrusion; the seal had an excellent appearance when removed from the test. This element sealed successfully under the same conditions as 217-II-1 except even a higher differential pressure, 24.8 vs 21.4 MPa (3600 vs 3100 psi).

O-Ring Simulation Tests

Another good quantitative demonstration of the accomplishment is the O-ring SIM test data which compares the developed compounds against a limited number of commercially available compounds. The packer SIM test was modified for static simulation testing of O-rings. The 2-329 O-ring groove was of standard commercial dimensions with no backups. The measured clearance was 229 μm (0.009 inch), quite severe for the test conditions. The O-rings were tested at 260 C in the synthetic geothermal brine starting at 28.3 MPa (4100 psi) differential pressure. The test was set for 22 hours and if a compound failed before 22 hours, the differential pressure was decreased by 6.9 MPa (1000 psi) and the test rerun with a new seal. The reduction in differential pressure progressed to 14.5 MPa (2100 psi) where the testing stopped if the compound failed.

Because this was an unusually severe environment, the developed compounds have a distinct advantage in that they were developed specifically for this condition. Figure 3 summarizes the resulting capability measured for each of the compounds. The 255 FKM and 267 EPDM compounds performed better than their commercial counterparts. The 255 FKM held for the entire test period at 21.4 MPa (3100 psi), while the commercial counterparts both broke at 14.5 MPa (2100 psi). The 267 EPDM looked excellent after the 28.3 MPa (4100 psi) run and is capable of higher differential pressures while its counterparts showed incipient failure. There were no commercially comparable counterparts for the EPDM/FKM blend or the propylene-TFE, both ran the full 22 hours at the full differential pressure. The blend looked as good as the 267 EPDM and, hence, is also capable of higher differential pressures and the propylene-TFE suffered minor extrusion. There was no counterpart to the DuPont perfluoroelastomer (Kalrez®); however, it is of keen interest because it is one of the more chemically stable polymers. It is very expensive but its expense is justifiable in some situations because of its longer life. For the synthetic geothermal brine environment, the 3074 perfluoroelastomer did not fare as well as the four developed compounds in the O-ring test, and earlier coupon aging tests were made on 3073 perfluoroelastomer which indicate that it is not as hydrothermochemically stable as the compounds developed in the project. Specific compounding for the geothermal brine environment may result in similar improvements as was herein accomplished.

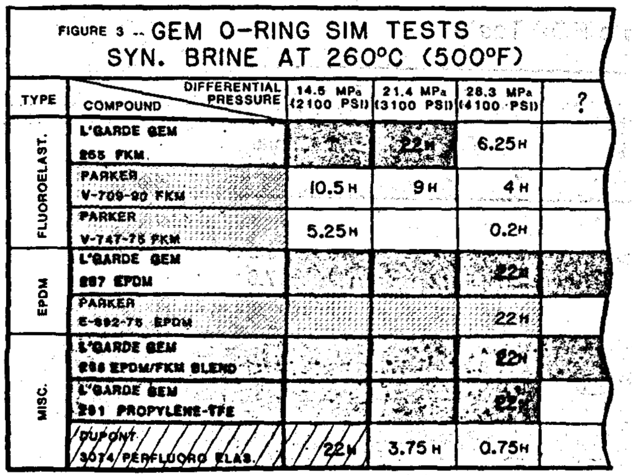


FIGURE 3 -- GEM O-RING SIM TESTS
SYN. BRINE AT 260°C (500°F)

TYPE	COMPOUND \ DIFFERENTIAL PRESSURE	14.5 MPa (2100 PSI)	21.4 MPa (3100 PSI)	28.3 MPa (4100 PSI)	?
FLUOROELAST.	L'GARDE GEM 255 FKM		22H	6.25H	
	PARKER V-709-90 FKM	10.5H	9H	4H	
	PARKER V-747-75 FKM	5.25H		0.2H	
EPDM	L'GARDE GEM 267 EPDM			22H	
	PARKER E-692-75 EPDM			22H	
MISC.	L'GARDE GEM 266 EPDM/FKM BLEND			22H	
	L'GARDE GEM 291 PROPYLENE-TFE			22H	
	DUPONT 3074 PERFLUORO ELAS.	22H	3.75H	0.75H	

FIGURE 3 — GEM O-ring SIM tests syn. brine at 260 C (500 F).

Figure 4 is a composite photograph of the more interesting seals; 267 EPDM, E-692-75 EPDM, 266 EPDM/FKM blend, and the perluoroelastomer 3074. All seals ran successfully for 22 hours at 28.3 MPa (4100 psi) differential pressure except the perfluoroelastomer 3074 which ran at 14.5 MPa (2100 psi) differential pressure. Note that the 267 EPDM and the 266 EPDM/FKM blend look excellent, although some permanent squaring of the O-rings did occur. Both the E-692-75 EPDM and the perfluoroelastomer 3074 are seen to have suffered damage.

Case Histories of Other Organizations

Since the completion of the elastomer development, there have been opportunities for other organizations to expose the favored compounds to various environments. Test slabs, O-rings, and packer seals were molded by us and provided to the other organizations for independent testing.

Sandia National Laboratories, Laboratory BOP Simulation

Sandia[3] investigated promising elastomers for the cable blowout preventor (BOP) application. They tested tensile specimens in the vapor phase of steam at 260 C (500 F) for 100 hours plus a substantial heat-up and cool-down time of about 14 hours. On their summary test, they exposed seven compounds, four of which were provided by us.

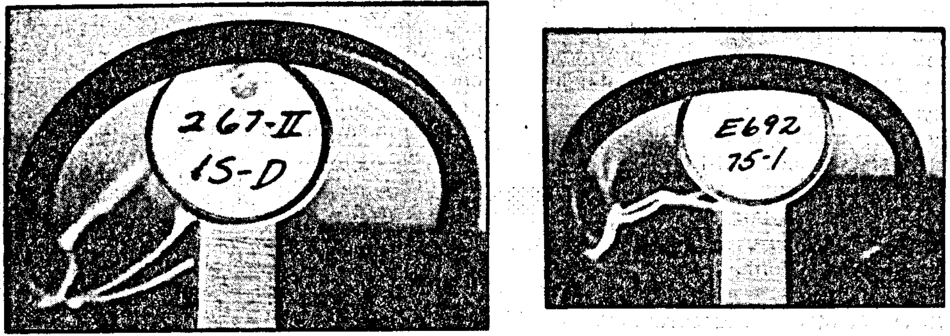
The compounds tested are shown in Table 3 which is excerpted from Reference 3. The four compounds from this project were the best four out of seven tested. The E60C FKM and the 914-80 both became so brittle they could not be measured without their breaking. The only compound not from this project which survived was the GH FKM which experienced a 72% loss of tensile strength. The compounds developed herein ranged from 9 to 40% loss of tensile strength.

TerraTek, Laboratory Drill Bit Seal Simulation

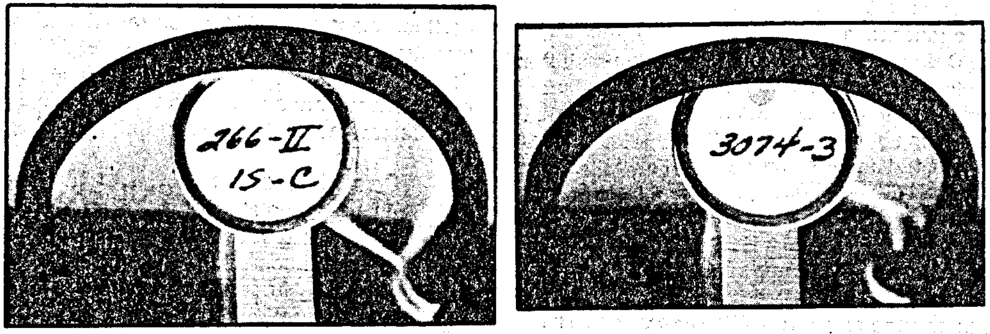
TerraTek[4] is a DOE contractor who has developed a dynamic drill-bit seal tester. It tests a rotating seal simulating the journal seal of a tricone bit. On each revolution, a jog is superimposed on the rotational motion. Tests are typically run at 90 rpm with rock cuttings, sand, and water on one side of the seal, and grease on the other.

O-ring seals made from our new static packer seal compounds were provided to TerraTek for dynamic drill-bit seal testing. There was concern about testing the static packer seal compounds in this environment because there was no attention paid to dynamic seal properties when they were compounded. Furthermore, there was concern about the exposure to hydrocarbon grease, another environmental consideration outside the realm of the geothermal packer seal development.

Because of the concern about the exposure to hydrocarbon grease, it was first recommended that TerraTek test the 266 EPDM/FKM blend as EPDM's are generally vulnerable to hydrocarbon, hence, testing blend compounds seemed prudent to gain the oil resistance of the FKM. The 266 blend compound was tested at a time when 40 to 50 hours of successful sealing was very good performance. To the pleasant surprise



267 EPDM, ΔP = 28.3 MPa (4100 psi)   E-692-75 EPDM, ΔP = 28.3 MPa (4100 psi)



266 EPDM/FKM Blend, ΔP = 28.3 MPa (4100 psi)   3074 Perfluoro, ΔP = 14.5 MPa (2100 psi)

FIGURE 4 — Post mortem O-rings SIM test seals after 22 hours in sour synthetic geothermal brine @ 500 F.

TABLE 3 — Sandia Cable BOP Tests

Elastomer	Ult. Tensile @ RT psi V	Ult. Tensile @ RT psi CA	Ult. Elong. @ RT % V	Ult. Elong. @ RT % CA	Shore A Hard @ 260 C V	Shore A Hard @ 260 C CA	Extrusion Resist @ 260 C, psia V	Extrusion Resist @ 260 C, psia CA
EPDM								
Early-E263	1363	1020	>840	>840	61	52	9.6	5.5
Present-267	1610	1554	141	148	92	92	17.1	17.6
FKM								
Early-115	1699	244	162	74	80	58	11.2	3.6
Present-255	2195	1591	224	226	79	79	11.4	6.3
EPDM/FKM								
Early-235	1882	1506	681	431	67	74	8.3	10.6
Present-266	1596	1531	193	163	88	89	18	17.3
Propylene-TFE								
Early-268	1673	1289	394	442	69	63	6.7	5.6
Present-291	2335	2162	134	158	95	95	17.4	15.2

of everyone concerned, the static packer seal blend compound sealed for 47 hours.

Additional tests were then made with the 267 EPDM compound. A presoak temperature of 288 C (550 F) was agreed to; this was the first time that the test was run at this high a temperature. The grease and temperature did not adversely affect the 267 EPDM seal as it was successfully sealing after 104 hours when the test was stopped. The post test seal was described by TerraTek personnel as looking surprisingly unaffected by the environment. It did, however, abrade and wear significantly on the debris side of the seal.

Otis Engineering Corp., Packer Laboratory and Downhole Tests

Compound 267 EPDM packer seals were molded by us for tests by Otis Engineering Corp.[5] Molded seals for 7 inch, 26 lb/ft casing were provided and tested by Otis at 302 C (575 F) and 20.7 MPa (3000 psi) differential pressure in their laboratory packer simulation tester. The elements sealed successfully for five days when the test was stopped. The seal was still elastomeric and did not show any signs of reversion softening or scission. Prior to this test, Otis tested an EPDM from another source at a lower temperature, 288 C (550 F), and the seal failed because it lost strength due to reversion. The post mortem seal did not have any form and looked like a lump of tar.

Subsequent to the laboratory tests, Otis installed a 267 EPDM element on a new R&D packer and ran it in a heavy oil field in Bakersfield. The well has 7 inch casing and is used for continuous steam injection. The steam is about 246 C (475 F) at the boiler and 204 C (400 F) at the well head.

The packer was pulled after 5 months of successful sealing to perform well maintenance. Tracer tests confirmed that the packer was still sealing prior to removal. The packer was pulled with no problems whatsoever, where difficulty is usually encountered with the seal jamming and either being rolled or ripped off the packer.

The appearance of the recovered seal was excellent, with virtually no permanent deformation. It looked like a new seal and obviously could have remained in operation indefinitely. There was no apparent degradation from the crude oil which covered the packer when it was removed. Figure 5 is a photograph of the packer seal element after removal from the Bakersfield well.

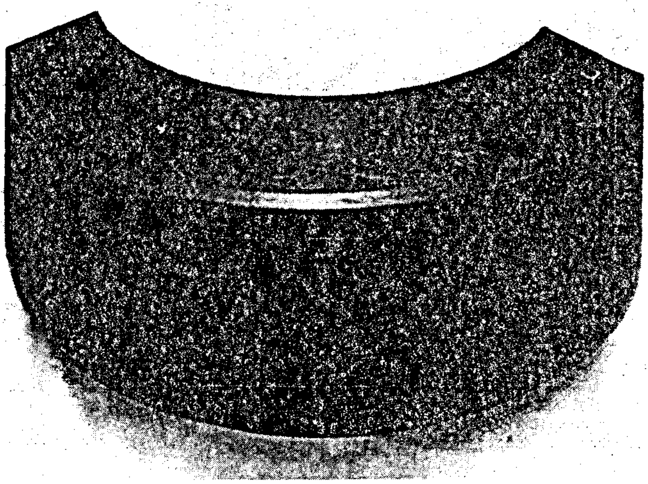


FIGURE 5 — EPDM after 5 months in Bakersfield steam injection well for heavy oil.

Los Alamos Scientific Laboratory (LASL), Laboratory and Downhole Cablehead Tests

LASL[6] is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock Project. They have shown feasibility of the concept at 200 C (392 F) and are currently going to demonstrate the concept with deeper wells and higher temperatures, 275 C (525 F) minimum. Because of the higher temperatures, LASL is concerned about elastomeric parts in various well and logging tools. They awarded a contract to our company to support them with the developed high temperature elastomer expertise.

We fabricated O-rings and a cable bend protector from the 267 EPDM compound for the LASL cablehead. LASL tested the cablehead in an autoclave with Mobil One oil inside the cablehead and water outside of it. They ran 24 hour cycles with the temperature on for 8 hours and off for 16; this simulates tripping in and out of the hole. The best prior performance they achieved was with commercial fluoroelastomer O-rings which literally disintegrated after one cycle. They ran the 267 EPDM for 4 cycles or 4 days and stopped the tests to examine the seals. The temperature was 275 C (525 F) minimum with an excursion to 340 C (644 F) during one of the 24 hour cycles. The pressure was 51.7 MPa (7500 psi). The seals looked good and were sealing after 4 cycles, although there was some swelling from the high temperature oil and some permanent deformation as would be expected for these conditions. They hope to go ten cycles operationally before they must change out the O-rings.

Subsequent to the autoclave tests, LASL ran the cablehead with the 267 EPDM parts on a temperature probe in the new hotter well. They measured a near bottomhole temperature of 317 C (603 F) with the probe. After this run, the O-rings and bend protector were examined and reported to be in excellent condition for the imposed environment.

These are landmark tests because this is the first time the 267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range; the first time the 267 EPDM was tested in 100% oil, the first time the 267 EPDM was tested in oil at extreme temperatures (excursions to 340 C (644 F), and the first time the 267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDM's are generally not used in oil environments because they are extremely vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the 267 EPDM is less vulnerable to oil than other EPDM's.

Technology Transfer

Active transfer to industry of the developed technology is currently taking place. The objective is to achieve more widespread knowledge, use and benefit of the developments within the commercial industrial community.

Applications for technology transfer were solicited from companies in the well tool industry. Advertisements were placed in the *Geothermal Energy Magazine* and the *Petroleum Engineer International*, and there was a mailing list of over 300. From the applications received, three companies were selected for technology transfer and three additional companies were placed on a backup list should funds be available.

The transfer process is one where progressively more and more of the processing is done by the transferee with checks to assure that the elastomer physicals are being reproduced. When there is confidence that reproducibility exists with an elastomer that is mixed, molded, and post cured by the transferee with his raw materials, equipment, and personnel; then full-scale parts are fabricated and tested under laboratory conditions and/or in the actual downhole environment. The applications that are currently planned for testing are downhole electric pump seals, drill bit seals, drilling shock-sub seals, drill pipe protectors, and blow-out preventor ram rubbers.

Molded rubber components made from the compounds developed are anticipated to be available to industry on a commercial basis through the technology transferees. In addition, a workshop discussing the transfer case histories is tentatively planned for 1981 for all who are interested.

Summary

Elastomers from four different polymer systems were developed which exceed the original requirements for geothermal casing packer seals at 260 C (500 F). Based on testing by the developer and other organizations, potential was demonstrated for additional application to geothermal BOP's, drill bit seals, and logging tools; and to heavy oil steam injection packers. Other application tests outside the original intended use which are planned include downhole electric pump seals, drilling shock-sub seals, drill pipe protectors, and blow-out preventor ram rubbers.

Acknowledgments

The project described in this paper was sponsored by the United States Department of Energy, Division of Geothermal Energy. J. V. Walker and R. R. Reeber were instrumental in providing the opportunity to engage in this research and development, and provided valuable guidance throughout the effort. In addition, P. E. Cassidy of Texas Research Institute contributed

significantly as technical coordinator for DOE as did A. J. Adduci of the San Francisco office of DOE as the technical administrator. L. E. Kukacka is currently providing valuable guidance on the Technology Transfer effort.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. J. K. Sieron of the Air Force Materials Laboratory and J. C. Rowley of Los Alamos Scientific Laboratory were especially helpful.

References

1. Hirasuna, A. R. et al. July, 1979, Geothermal Elastomeric Materials (GEM) Program—Final Report for Period October 1, 1976-June 30, 1979, L'Garde, Inc. Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy.
2. Hirasuna, A. R. et al. Evaluation Tests for Geothermal Casing Packer Seal Elastomers, presented at a meeting of the American Institute of Chemical Engineers, Philadelphia, Pennsylvania, June 4-8, 1978.
3. Arnold, C, Jr. Memo to Distribution, Sandia Laboratories, April 19, 1979, Albuquerque, New Mexico.
4. Winzenried, R. Letter to Hirasuna, TerraTek, June 15, 1979, Salt Lake City, Utah.
5. Taylor, D. Private Communication, Otis Engineering Corp., April 23, 1979, Dallas, Texas.
6. Archuleta, J. and Dennis, B. Private Communications, Los Alamos Scientific Laboratory, July 9, 1980, August 4, 1980, Los Alamos, New Mexico.

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APPENDIX N

SPECIFICATION FOR STANDARD SYNTHETIC GEOTHERMAL FLUID
DOCUMENTATION OF SOME PREVIOUS WORK

19 May 1980

Prepared For

ASTM E45.10.02
MATERIALS TASK GROUP

Prepared By

Alan R. Hirasuna
Clifford A. Stephens

L'GARDE, INC.
Newport Beach, CAlifornia 92663

ASTM E45.10.02 -- MATERIALS TASK GROUP

SPECIFICATION FOR STANDARD SYNTHETIC GEOTHERMAL FLUID
DOCUMENTATION OF SOME PREVIOUS WORK

19 May 1980

Objective To develop a specification and recipe for standard synthetic geothermal fluid(s) for environmentally testing or ageing materials for geothermal applications. This may be one general fluid for all testing or a set of fluids each pertinent to a certain class of materials or wells. Accomplishment will allow comparative evaluations of results from different laboratories, and will preclude the necessity of redeveloping a fluid on each effort enabling maximum energy to be spent on the primary objective of the particular project.

Introduction I was asked at the 17 October 1979 meeting in Philadelphia to take the first steps toward the above objective. L'Garde was faced with the same but less general problem associated with the development of elastomer compounds under the Geothermal Elastomeric Materials (GEM) Program contracted by the Department of Energy - Division of Geothermal Energy (DOE-DGE). On that program a minor effort was expended to determine a representative geothermal environment for which elastomers were specifically compounded.

The various geothermal resources which imply different classes of environments can be broadly categorized as follows:

1) hydrothermal
2) geopressured
3) Hot Dry Rock (HDR)

The work on the GEM Program addressed the nearest term resource, hydrothermal, and in addition HDR. Hydrothermal resources can be subcategorized into vapor dominated wells, e.g. The Geysers and liquid dominated wells, e.g. the Imperial Valley. The vapor dominated wells tend to be chemically benign relative to the hotter liquid dominated wells. The HDR resources can use a heat transfer fluid of choice, probably nearly potable water, hence, what works for hydrothermal should work for HDR. The geopressured wells exist along the US Gulf Coast and are characterized by high levels of dissolved methane, extreme pressures (~100-138 MPa (14.5-20.0 Ksi)), high concentrations of H_2S (~35-40), and moderately high temperatures (<200°C (392°F)). These conditions markedly differentiate the geopressured resource environment from the others.

Because of the need for a test fluid for elastomer development, L'Garde developed a synthetic geothermal fluid representative of the hydrothermal resource which also encompasses the HDR resource. The important considerations made, and references consulted are described to provide benefit of previous work related to the present Objective. A chemical environment pertinent to ELASTOMERS was defined and a practical synthetic fluid recipe was developed. Because of lack of information, and budget and schedule constraints, this was not an exhaustive effort by any means, and in no way, should be treated as irrefutable.

The synthetic fluid was used in conjunction with development of high-temperature geothermal packer elastomer compounds. Compounds from four different polymer systems successfully met the objectives of the contract (260°C (500°F)) and one was successfully tested as high as 302°C (575°F). Only limited field test data exists at this time, nonetheless, it is quite encouraging.[1,2]

Developed GEM Program Environmental Requirements The following simplified requirements were reduced from a broader host of complex requirements as interpreted to be pertinent to elastomers development for high-temperature geothermal casing packers.

Application:	Casing Packer, nonproduction	
Temperature:	260°C (500°F)	
Diff. Press:	20-28 MPa (2900-4060 psi)	
Residence:	24 hours	
Chemistry:	H_2S	300 ppm
	CO_2	1000 ppm
	NaCl	25,000 ppm
	H_2O	balance

High-Temperature Packer Seal -- Modes of Failure High-temperature downhole seals tend to fail from extrusion and/or hardening. The more interesting geothermal wells have temperatures of 260°C (500°F) and higher, and it is not unusual to have compositions of the fluid which are unusually reactive to elastomeric compounds.

260°C (500°F) causes most rubber compounds to harden by thermal breaking of double bonds and forming new crosslinks to form a three dimensional matrix with less and less ability to yield and recover when deformed. This action proceeds quite rapidly in air (oxygen). In water or steam at high temperatures the hot water acts as an active swelling and softening agent, sufficient to break crosslinkages causing most cured elastomers to devulcanize or revert and to become soft and weak. The geothermal well fluid often contains sulfur in the form of hydrogen sulfide. At high temperatures sulfur rapidly breaks double bonds and forms crosslinks until the rubber becomes hard and brittle. The fluid also can contain considerable concentrations of metallic salts completely ionized. The effects of such salts on elastomeric compounds is not known.

Early Considerations of Elastomers The development and evaluation of elastomers for the GEM Program was confined to commercially available elastomers, reinforcing fillers and curing systems. The most common and lowest cost elastomers are the butadiene copolymers. These elastomers are copolymers of butadiene and styrene or butadiene and acrylonitrile (the latter is used in nearly all oil well seals because of its resistance to degradation by crude oil). These polymers have unsaturated backbone structure - C = C - C - C = C - etc., the double bond being used to get fast curing with sulfur. Unfortunately only a few of the double bonds are used to form the sulfur crosslinkages; consequently sulfur in the geothermal fluid continues to break those remaining double bonds to form more sulfur crosslinks until the seal compound becomes very hard and weak, crumbling or cracking during use.

A logical step would be to consider elastomers free of excess double bonds in the backbone. There are a considerable number of these as discussed below.

Butyl rubber is a copolymer of isobutylene and small amounts of isoprene to give an elastomer with most of the backbone as single bonds. It has a long history of use for the curing bags where it is exposed to water at 177°C (350°F); however, it softens severely at 260°C (500°F) and is slowly devulcanized by the hot water.

Epichlorohydrin polymers (chlorinated epoxy propane) have the required saturated backbone. The high concentration of pendent chlorine groups makes it very resistant to solvents and fuels. There was no information about resistance to high-temperature brine. A commercial formulation was selected for evaluation although softening at 260^{o}C (500^{o}F) and extrusion failure was expected and occurred.

The polyacrylics (copolymers of ethyl acrylate and chlorovinyl ethyl ether or butyl acrylate and acrylonitrile) have the desired saturated backbone and are highly resistant to heat oxidation; however, neither are resistant to water or steam at high temperature.

The chloroprene elastomers have good oxidation resistance, but poor water resistance.

Ethylene propylene diene terpolymers are similar to the butyl rubbers in properties. They have the desired saturated backbone with a few double bonds to facilitate curing. They are widely used in tire curing bags where resistances to water at 177°C (350°F) is required.

Silicone elastomers have the best high-temperature oxidation resistance and maintain their physical properties better at 260^{o}C than any of the elastomers. The backbone is -Si-O-Si-O- which is thermally stable. No applicable information about resistance to sulfur or water or high temperatures was found. However, small amounts of oil cause severe degradation. Fluorosilicone elastomers are similar to the silicones except they have been fluorinated to give the compounds some resistance to oils. According to Dow Corning, applicability of silicone rubber to the GEM environment is uncertain, therefore, simple Screening Tests to determine the hydrolytic and sulfur resistance were accomplished.

The fluoroelastomers (Vitons, Fluorels) are fluorinated hydrocarbons with completely saturated -C-C- backbone to give the best resistance to heat, oxidation and attack by sulfur. Peroxide cured Vitons were selected for compounding, evaluation of cure systems and reinforcing fillers principally because there was a considerable amount of data reported in the literature indicating good prospects in steam and fluids environments and, the Air Force Materials Laboratory has been experiencing success with them at 260^{O}C for high performance aircraft hydraulic cylinder applications.

The GEM elastomer development was centered around the casing packer seal element operating at 260^{O}C. The polymers first chosen to be developed for this application were the then newly introduced peroxide cured family of Viton fluoroelastomers. After a fair amount of development, little success was experienced with the peroxide cured Vitons. Hence, other alternates were investigated in the interest of developing some improvements over what was then being used, primarily nitriles (Buna N). Table I shows all the basic polymer types which were finally investigated under the GEM Program. Within each polymer type, several specific polymers may have been investigated. For example, there were 9 fluoroelastomer polymers investigated - Viton GH, Viton GLT, VT-R-4590, Viton E-60C, Fluorel 2174, Fluorel 2175, Fluorel 2178, Fluorel 2179, and Fluorel 2181.

TABLE I -- ELASTOMERIC POLIMERS COMPOUNDED UNDER GEM PROGRAM

Common Chemical Designation	Trade Name	Manufacturer	Comment
Fluorelastomer (FKM)	Viton	DuPont	
	"E" type		Bisphenol cured
	"G" type		Peroxide/Triazine cures
	Fluorel	3M	
	2170's 2181		Bisphenol cured
Ethylene Propylene Diene (EPDM)	Nordel	DuPont	Peroxide cured
Propylene-TFE	AFLAS	Asahi Glass	Peroxide/Triazine cured
Perfluoroelastomer	Kalrez	DuPont	Finished parts only
Dimethylsilicone (VMQ)	Silastic	Dow Corning	
Fluorosilicone (FVMQ)	Silastic	Dow Corning	Resistant to hydrocarbons

<u>Representative Fluid Constituency and GEM Recipe</u> The Earth Sciences Division of the Lawrence Berkeley Laboratory, University of California, was under contract to compile available data on fluids from liquid dominated geothermal resources in the United States. The compilation is quite thorough but they were limited on their comprehensiveness because of proprietary data that was not available. In 1976 there was also some question as to the accuracy of the data especially regarding gasses. A common practice was to collect the samples in a polyethene bottle and transport it several miles to an analytical lab.

It's quite conceivable that during this interim significant amounts of gas permeated out of the bottle. L'Garde is not aware of the resolution of this problem and whether the later published data is without this uncertainty. Special sampling techniques were under consideration to assure that the assayed sample was truly representative.

Attachment 1 shows a typical data sheet for Baca 11A and 11C wells. This data sheet was taken from Reference 3.

Based on reports and discussions with Dr. J. Apps at the Lawrence Berkeley Laboratory in 1976, a specification was developed in 1976 for a synthetic geothermal fluid. The approach was to avoid simply compiling all the maximums for each constituent that has been measured as this may result in unrealistically severe chemistry, rather to apply judgement and generate a specification which covers the majority of potential geothermal wells. For example total dissolved solids can run as high as several hundred thousand ppm in the Salton Sea Area, however, the great majority of known potential wells are included at levels below 25,000 ppm. Dr. Apps was extremely helpful providing information and judgements on what generally exists in the overall geothermal resource.

The constituency specification which evolved for elastomers and was used for the GEM Program testing is as follows:

H_2S	300 ppm
NaCl	25,000
CO_2	1,000
H_2O	balance

The H_2S and CO_2 are formed from $Na_2S.9H_2O$ and $NaHCO_3$ respectively. The test uses a finite amount of H_2S thus limiting the potential exposure to personnel. If all of the H_2S were to escape into the room including the excess leftovers, the resulting concentration would be about 1 ppm as compared to OSHA's maximum allowable to 10 ppm. This approach is preferred over one where bottled H_2S is used and potential for lethal concentrations of H_2S exist. The recipe for the synthetic fluid is as follows:

$Na_2S.9H_2O$	5.29 gms.
$NaHCO_3$	4.77 gms.
NaCl	56.64 gms.
H_2O distilled	1430.00 gms.
HCl(0.1N)	1.0 liter

If this recipe is used, care should be exercised in chosing the pressure vessel to contain it. At 260°C (500°F) the saturation pressure is about 5.2 MPa (754 psi) and it increases very, very, rapidly with temperature. And because of the chloride and hydrogen ions, vessel materials vulnerable to stress cracking and hydrogen embrittlement should be avoided or considered with extreme care.

The above recipe was very practical, resulting in an economical procedure which enabled comprehensive sour brine testing of over 129 elastomer compounds. One possible drawback, however, is that the brine is not refreshed, rather, it starts with the specified constituency which continually changes during the autoclave ageing. Considering a refreshing system is a pertinent cost and safety trade-off. However, real uncertainties exist as to what the materials are actually exposed in operation. It is conceivable that obscure seals may in fact experience an environment which is not refreshed and therefore refreshing may create unrealistic environments for some applications. The expedient philosophy of "some H_2S exposure is more realistic than none" may be appropriate here, assuming funding to fully investigate the question is not available.

Other Laboratory Environment Efforts Laboratory environmental testing in simulated geothermal environment is happening across the nation, some of which L'Garde is aware. A lot of the testing is in water or pure salt water. Fewer tests are done with H_2S as a constituent because it can be so hazardous. Several organizations within the geothermal community who are doing H_2S testing are using the recipe developed under the GEM Program and would not represent different approaches or information.

Two organizations who were involved in similar laboratory testing were very helpful to L'Garde at the time it was developing the GEM recipes. They were and perhaps still are engaged in sour brine laboratory testing which includes refreshing systems. They are

Battelle-Northwest
Richland, Washington
D. W. Shannon

Case Western Reserve University
Cleveland, Ohio
A. R. Troiano

In addition, work is being done by Shell Development and probably competitor oil companies at high concentration of H_2S, simulating the geopressured environment.

Summary The objective of this presentation is to document previous related work performed for DOE-DGE by L'Garde, Inc. This information should provide some of the building blocks required to meet the overall committee objective, of creating an ASTM specification for standard geothermal laboratory test fluid(s).

L'Garde developed high-temperature packer elastomer compounds for the DOE-DGE, and, associated with the design and fabrication of the support evaluation tests, L'Garde developed a recipe for synthetic geothermal test fluid. Economy dictated that an abbreviated effort be spent on developing the constituency specification and the recipe so any more exhaustive development should reinvestigate the earlier premises.

The synthetic fluid developed enabled L'Garde to achieve a greater degree of operational simulation in the laboratory. Early results exceed the contract requirements and indicate that a significant advance was made. However, the final substantiation will only exist after broad field experience is developed.

The presentation includes the thinking used in developing the elastomer test fluid as well as references which were invaluable. The references provided information on what environments and chemical constituencies could be expected in geothermal wells as well as previous experience on laboratory methods of creating a synthetic geothermal fluid.

References

1. Hirasuna, A. R., et.al, "High-Temperature Geothermal Elastomer Compound Development", Paper No. 36, Rubber Division, American Chemical Society, Las Vegas, NV, May 20-23, 1980.

2. Hirasuna, A. R., et.al, "Geothermal Elastomeric Materials (GEM) Program, Final Report, 1 October 1976 - 30 June 1979", Report Number SAN/1308-2, L'Garde, Inc., Newport Beach, CA, July 1979.

3. Cosner, S. R., and Apps, J. A., "A Compilation of Data on Fluids from Geothermal Resources in the United States", Report Number LBL-5936, Lawrence Berkeley Laboratory, Berkeley, CA, May 1978.

ATTACHMENT 1

LBL-5936
UC-66b
TID-4500-R66

COMPILATION OF DATA ON FLUIDS FROM GEOTHERMAL RESOURCES IN THE UNITED STATES

by

S. R. Cosner and J. A. Apps

Earth Sciences Division
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

Prepared for the U.S. Department of Energy under
Contract W-7405-ENG-48

TABLE OF CONTENTS

```
                                        RECORD    1
                                        CODE NAME=BACA 11A
                                        SAMPLE TYPE=WATER

WELL BACA 11
BACA LOCATION NO. 1 KGRA
LOCATION-- T19N, R3E, SEC. 12, 1900FT S, 1500FT W, FROM NE CORNER.
SANDOVAL COUNTY, N.M., USA

WELL INFORMATION
  OWNER-- UNION OIL CO.
  DATE DRILLED-- 19 SEP 73 - 13 NOV 73

SAMPLING INFORMATION
  SAMPLE NUMBER, LABORATORY-- SAMPLING PERIOD A

PHYSICAL DATA
  PH= 6.60
  SP. CONDUCTANCE=  11160.00 MICROMHOS/CM
  TOT DISS SOLIDS=   7593.00 PPM
  OTHER DATA--
  SUSPENDED SOLIDS = 638 PPM

BRINE DATA
  UNITS-- PPM

  CONSTIT-    CONCEN-
   UENT       TRATION    COMMENT
  -------   ----------- ---------------------------------------
  SIO2         640
  NA          2200
  K            550
  CA            46
  CL          4400
  SO4           50
  HCO3          97
  CO3            0
  S              1.9

 BIBLIOGRAPHIC DATA
   SOURCES--
    TONEY 76B
    WITMAN 76
    TONEY 76

 OTHER NOTES
   SEE CODE NAME = BACA 11B FOR NONCONDENSABLE GAS ANALYSIS.
```

```
                                        RECORD    2
                                        CODE NAME=BACA 11B
                                        SAMPLE TYPE=NONCONDENSABLE GASES

WELL BACA 11
BACA LOCATION NO. 1 KGRA
SANDOVAL COUNTY, N.M., USA

SAMPLING INFORMATION
  SAMPLE NUMBER, LABORATORY-- SAMPLING PERIOD A
  CONDITION OF SAMPLE-- NONCONDENSABLE GASES = 2.44 PERCENT BY WT.
        IN STEAM PHASE.

PHYSICAL DATA
  OTHER DATA--
  PH OF CONDENSATE = 4.9

BRINE DATA
  UNITS FOR GASES-- PPM

  CONSTIT-    CONCEN-
   UENT       TRATION    COMMENT
  -------   ----------- ---------------------------------------
  CO2        33700
  H2S          290
  NH3            4
  CH4            0
  H2             2
  N2            39

  GASES IN STEAM PHASE ONLY.

 BIBLIOGRAPHIC DATA
   SOURCES--
    TONEY 76B
    TONEY 76

 OTHER NOTES
   SEE CODE NAME = BACA 11A FOR FLUID PHASE ANALYSIS.
```

```
                                        RECORD    3
                                        CODE NAME=BACA 11C
                                        SAMPLE TYPE=WATER

WELL BACA 11
BACA LOCATION NO. 1 KGRA
SANDOVAL COUNTY, N.M., USA

SAMPLING INFORMATION
  SAMPLE NUMBER, LABORATORY-- SAMPLING PERIOD B

PHYSICAL DATA
  PH= 6.80
  SP. CONDUCTANCE=  10630.00 MICROMHOS/CM
  TOT DISS SOLIDS=   7034.00 PPM
  OTHER DATA--
  SUSPENDED SOLIDS = 688 PPM

BRINE DATA
  UNITS-- PPM

  CONSTIT-    CONCEN-
   UENT       TRATION    COMMENT
  -------   ----------- ---------------------------------------
  SIO2         792
  NA          2000
  K            463
  CA            27
  CL          3400
  SO4           70
  HCO3         128
  CO3            0
  S              6

 BIBLIOGRAPHIC DATA
   SOURCES--
    TONEY 76B
    TONEY 76

 OTHER NOTES
   SEE CODE NAME = BACA 11D FOR NONCONDENSABLE GAS ANALYSIS.
```

```
                                        RECORD    4
                                        CODE NAME=BACA 11D
                                        SAMPLE TYPE=NONCONDENSABLE GASES

WELL BACA 11
BACA LOCATION NO. 1 KGRA
SANDOVAL COUNTY, N.M., USA

SAMPLING INFORMATION
  SAMPLE NUMBER, LABORATORY-- SAMPLING PERIOD B
  CONDITION OF SAMPLE-- NONCONDENSABLE GASES = 3.99 PERCENT BY WT.
        IN STEAM PHASE.

PHYSICAL DATA
  OTHER DATA--
  PH OF CONDENSATE = 4.5

BRINE DATA
  UNITS FOR GASES-- PPM

  CONSTIT-    CONCEN-
   UENT       TRATION    COMMENT
  -------   ----------- ---------------------------------------
  CO2        47390
  H2S          567
  CH4            1.5
  H2             4
  N2           109

  GASES IN STEAM PHASE ONLY.

 BIBLIOGRAPHIC DATA
   SOURCES--
    TONEY 76B
    TONEY 76

 OTHER NOTES
   SEE CODE NAME = BACA 11C FOR FLUID PHASE ANALYSIS.
```