

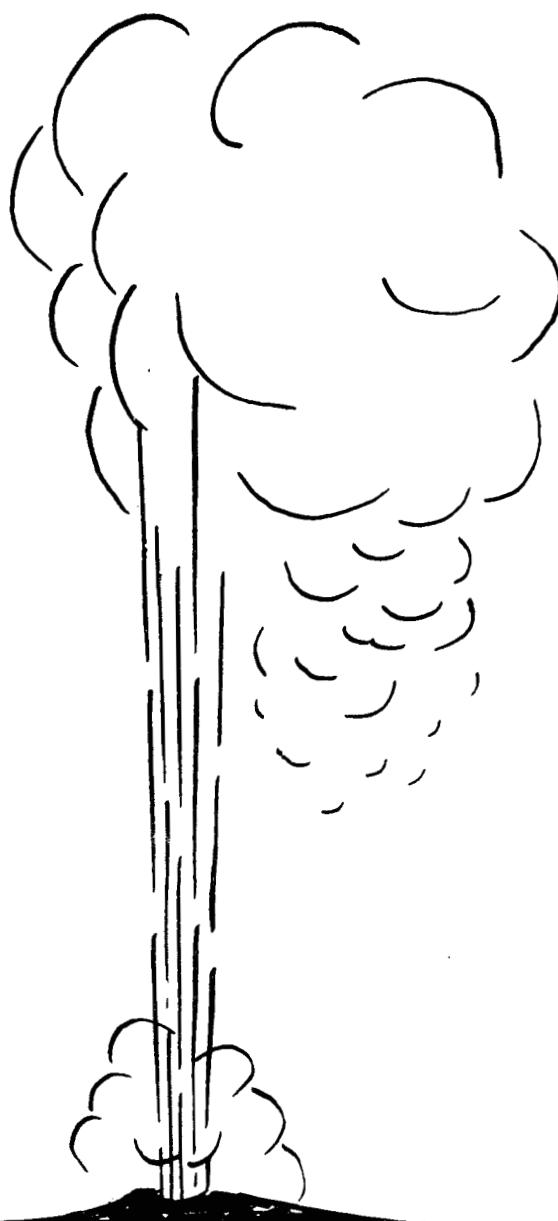
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ADDENDUM TO MATERIAL SELECTION GUIDELINES
FOR GEOTHERMAL ENERGY UTILIZATION SYSTEMS

Part I: Extension of the Field Experience Data Base
Part II: Proceedings of the Geothermal Engineering
and Materials (GEM) Program Conference
(San Diego, CA, 6-8 October 1982)

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

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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 EPDM 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 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, 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 trial 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 more so, 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) 232C (450F) 266C (510F)		x	x	x x 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.

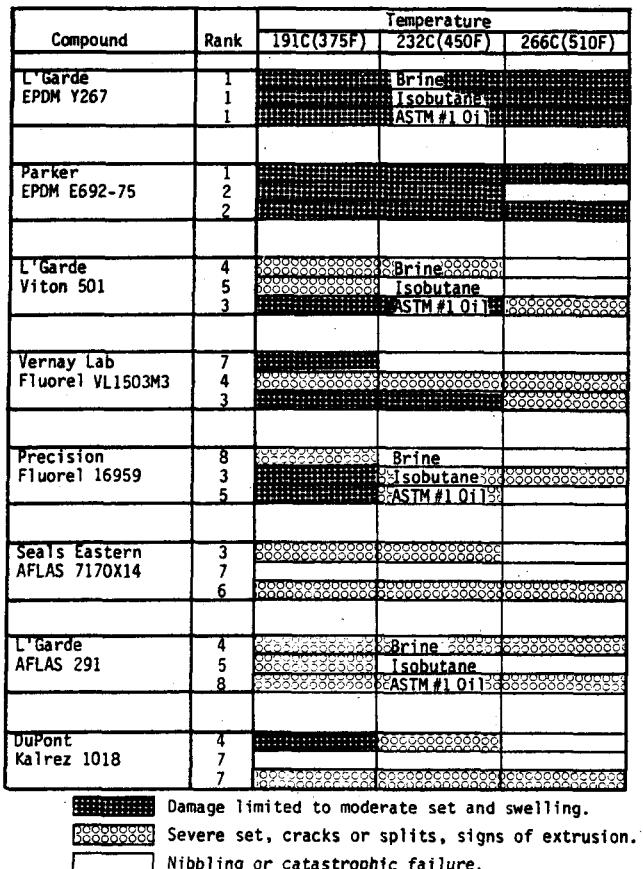


FIGURE 1. 46-HOUR O-RING TEST RESULTS,
20.7 MPa (3000 PSI) ΔP

Figure 2 summarizes the long-term O-ring test which complete the compatibility test program. The tests were run for approximately 6 months at 204°C (400°F) 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.

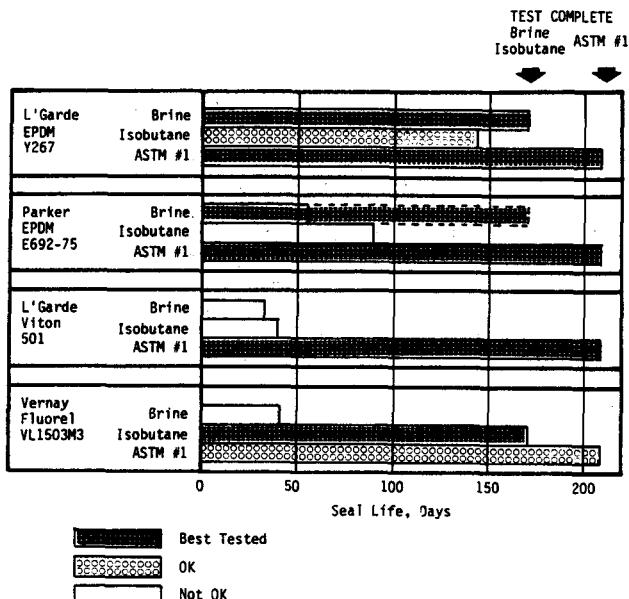


FIGURE 2. 6-MONTHS O-RING TEST RESULTS,
204C (400F), 20.7 MPa (3000 PSI) ΔP

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 Magna 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 dismantled 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 (°F)	Differential Pressure MPa (psi)	Time		
				Hours	Days	Months
Otis Lab, Packer	H ₂ O	303 (575)	20.7 (3000)	5	5	
Otis Lab, Packer	Sweet & Sour Crude	232 (450)	138 (20,000)	108	4.5	
Bakersfield, Packer	H ₂ O/Crude	204 (400)	1.7 (250)		150	
Baca Stim, Packer (2s)	Frac.	160 (320)	21.2 (3000)	12	0.5	
Baca DST, Packer (3s)	Frac/H ₂ O	171 (340)	4.1 (600)	18.5	.8	
LAML Lab, O-ring	H ₂ O/Oil	275 (525)	51.7 (7500)	40	1.7	
LAML Lab, Cement Wiper	H ₂ O	280 (536)	0 (0)	.5+	.02+	
Fenton Hill, O-ring	H ₂ O	317 (603)	41.4 (6000)	10	0.4	
Fenton Hill, Cement Wiper	H ₂ O/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 ₂ O	300 (575)	11.7 (1700)	6	.3	
Sandia Lab	H ₂ O	260 (500)	4.7 (680)	100	4.2	
Sperry Vickers Lab	H ₂ O/Hydrazine	218 (425)	2.2 (325)	85	3.5	
TerraTek Lab, O-ring	H ₂ O/Debris, Grease	288 (550)	1.4 (0/200)	104	4.3	

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