Sealing Simulated Leaks Technical Topical Report

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ABSTRACT

This report details the testing equipment, procedures and results performed under <u>Task</u> <u>7.2 Sealing Simulated Leaks</u>. In terms of our ability to seal leaks identified in the technical topical report, <u>Analysis of Current Field Data</u>¹, we were 100% successful. In regards to maintaining seal integrity after pigging operations we achieved varying degrees of success. Internal Corrosion defects proved to be the most resistant to the effects of pigging while External Corrosion proved to be the least resistant. Overall, with limitations, pressure activated sealant technology would be a viable option under the right circumstances.

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EXECUTIVE SUMMARY

This report details the testing equipment, procedures and results performed under Task 7.2 <u>Sealing Simulated Leaks</u>. Analysis of the data indicates that Pressure Activated Sealant Technology would be a viable repair option under the right circumstances.

We were 100% successful in the initial repair of leaks types identified in the technical topical report, Analysis of Current Filed Data². However, subsequent line pigging resulted in varying degrees of seal integrity. Internal Corrosion and Weld leaks proved to be the most resistant to the effects of pigging, while External Corrosion proved to be the least resistant.

The major factor contributing to seal failure during pig passage was the relatively low sealing pressure limits of pipelines. With a maximum available pressure differential of 9.93 MPa (1440 psi), the sealant did not develop sufficient strength to consistently resist the effects of pigging. Further testing indicated significant improvement in sealant strength at higher pressure differentials.

The optimum chance of long-term sealant success lies in pipelines which exhibit relatively high differential pressure and are not subjected to a rigorous pigging program.

EXPERIMENTAL

Establishing Leak Rates

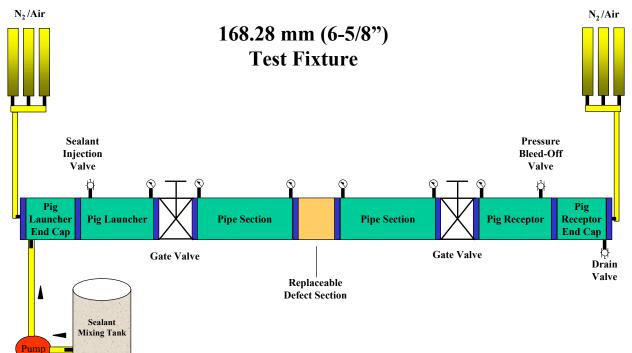
Overview / Objective

Since our experience in curing leaks in downhole applications has centered on liquid leak rates and not actual defect size we first needed to establish leak rates for each defect. We established leak rates with water, as well as nitrogen, providing a basis to correlate to past testing and operations. This data was then used to develop sealant formulations.

Apparatus

As previously reported³, the test model was constructed using 168.28 mm (6-5/8"), schedule 80 XS steel pipe with a wall thickness of 11 mm (0.432"), an internal diameter of 146 mm (5.761") and a Maximum Operating Pressure of 12.36 MPa (1,793 psi) MAOP.

Two gate valves along with twelve 25.4 mm (1") nipples were incorporated into the test model to achieve varied manipulations of pressure and isolation of sections and to allow for placement of pressure gauges, bleed-off valves, pressure pop-off valves, and ball valves for the injection and discharge of nitrogen, air, water and sealant. An overview of the test fixture is seen below in <u>Drawing 1</u>.



Drawing 1: Test Fixture

Test Fixture dimensional data is summarized below in Table 1.

Table 1:	Test Fixture Dimensions				
	OD, mm	OD, in.			
	168.280	6.625			
	ID, mm	ID, in.			
	146.000	5.761			
	Length, mm	Length, in.			
Launcher End Cap	543.000	21.375			
Pig Launcher	1780.000	70.125			
Gate Valve	565.000	22.250			
Pipe Section	2530.000	99.500			
Defect Section	1003.000	39.500			
Pipe Section	2530.000	99.500			
Gate Valve	565.000	22.250			
Pig Receptor	1780.000	70.125			
Receptor End Cap	543.000	21.375			
Total Fixture Length	11,839.000	466.000			
	11.8 meters	38 ft 10 in.			

The test model included replaceable 3 foot defect sections. Each defect section simulated a type of defect identified during the analysis stage; Defective Fabrication Weld (DFW), Defective Girth Weld (DGW), Defective Pipe Seam (DPS), External Corrosion (EC) and Internal Corrosion (IC). As previously reported⁴, these defects accounted for 75.6% of the incidents in our 205 incident base.

The DFW, DGW and DPS defects were represented by a single Weld Defect Section (<u>Photo 1</u>) that simulated common irregularities associated with welds including cracks and wormholes. Since 68.3% of the externally corroded pipe and 64.1% of the internally corroded pipe is described as either "localized pitting", "pinhole" or "pinhole with localized pitting", the EC and IC defects (<u>Photo 2</u> and <u>Photo 3</u> respectively) will simulate localized pitting with pinholes⁵. The defect section with two (2) pinholes (<u>Photo 4</u> and <u>Photo 5</u>) represents defects with higher leak rates. The dimensions of the 100 cm (3.29 ft) defect sections are summarized in SI units in <u>Table 2</u> and inches in <u>Table 3</u> with photos of the defects following.

Table 2:

168.28 mm OD / 146 mm ID Defect Dimensions (mm)

Defect Section	Corrosion			Pinhole 1		Pinhole 2	
	Length	Width	Depth	OD	Depth	OD	Depth
External Corrosion							
Defect 1	102.00	50.80	8.00	1.60	11.00	na	na
Defect 2	82.50	31.80	8.00	na	na	na	na
Internal Corrosion							
Defect 1	102.00	76.20	3.05	1.60	11.00	na	na
Defect 2	82.50	76.20	3.05	1.60	11.00	na	na
Pinhole Defect (2)	na	na	Na	1.60	11.00	1.60	11.00
Weld Defect							
Crack	50.80	1.60	11.00	na	na	na	na
Wormhole	XX	XX	11.00	na	na	na	na

Table 3:

6-5/8" OD / 5.761" ID Defect Dimensions (inches)

	_					- • •	
Defect Section	Corrosion			Corrosion Pinhole 1 Pinhole 2		ole 2	
	Length	Width	Depth	OD	Depth	OD	Depth
External Corrosion							
Defect 1	4.00	2.00	0.315	0.063	0.432	na	na
Defect 2	3.25	1.25	0.315	na	na	na	na
Internal Corrosion							
Defect 1	4.00	3.00	0.120	0.063	0.432	na	na
Defect 2	3.50	3.00	0.120	0.063	0.432	na	na
Pinhole Defect (2)	na	na	na	0.063	0.432	0.063	0.432
Weld Defect							
Crack	2.00	0.063	0.432	na	na	na	na
Wormhole	XX	XX	0.432	na	na	na	na

Photo 1: Weld Defect



Photo 2: External Corrosion Defect



Photo 3: Internal Corrosion Defect



Photo 4: Pinhole Defect



Photo 5: Pinhole Defect



Testing Procedures

Liquid leak rates were established by first filling the test model with water and continuing pumping from a marked drum. Maximum rate was determined either by maximum pressure allowed or maximum output of pump and recorded at X psi. The pumping rate was then reduced and once stabilized, the appropriate pressure and rate was recorded. We continued this process until a representative amount of data points was collected. The Weld Defect only had 2 data points due to the extremely small leak rate.

Nitrogen leak rates were established by pressuring the test model to maximum psi (limited either by pipe strength or nitrogen tanks) and recording the pressure drop over time. The leak rate was then calculated by first solving for the volume of nitrogen needed to pressure the test model at initial pressure by utilizing:

We then solved for the change in nitrogen volume due to pressure drop over time by utilizing the same formula at the final pressure. The leak rate was then calculated as the difference between <u>Initial Nitrogen Volume</u> and <u>Final Nitrogen Volume over Time</u>.

The compressibility factors used in the calculations were derived from the Beattie-Bridgeman equation of state for real gases at 20°C as shown in <u>Table 4</u>.

Table 4 Compressibility of Nitrogen at 293°K (68°F)						
Pressure (MPa)	Pressure (psi)	Z-Factor				
0.690	100	0.998				
1.379	200	0.997				
2.068	300	0.995				
2.758	400	0.994				
3.447	500	0.993				
4.137	600	0.993				
4.826	700	0.993				
5.516	800	0.993				
6.205	900	0.993				
6.895	1,000	0.994				
7.584	1,100	0.995				
8.274	1,200	0.996				
8.963	1,300	0.997				
9.653	1,400	0.999				
10.342	1,500	1.001				

Experimental Data

The results of the leak rate testing is summarized below in tables and charts for each defect type, showing liquid and gas leak rates in both SI and English units, preceded by representative photos for each defect type.



Photo 6: External Corrosion Leak Rate Testing

Photo 7: External Corrosion Leak Rate Testing



	Nitro	ogen		Water			
scf/min	scm/min	ΔP	ΔP	l/min	gpm	ΔP	ΔP
		MPa	psi			MPa	psi
66	1.870	8.320	1,206	10	2.64	9.960	1,445
48	1.360	7.490	1,087	8	2.11	6.790	985
48	1.360	6.810	987	6	1.59	4.650	675
40	1.130	6.170	895	4	1.06	1.900	275
36	1.020	5.620	815				
33	0.934	5.120	743				
30	0.850	4.660	676				
26	0.736	4.070	590				
23	0.651	3.520	511				
20	0.566	3.210	465				
19	0.538	2.920	423				
18	0.510	2.650	385				
16	0.453	2.410	350				
15	0.425	2.190	318				
13	0.368	1.990	289				
12	0.340	1.810	262				
11	0.311	1.630	237				
10	0.283	1.480	215				
9	0.255	1.340	194				
9	0.255	1.210	175				
8	0.227	1.090	158				

Table 5: External Corrosion Leak Rates

Chart 1: External Corrosion Leak Rates

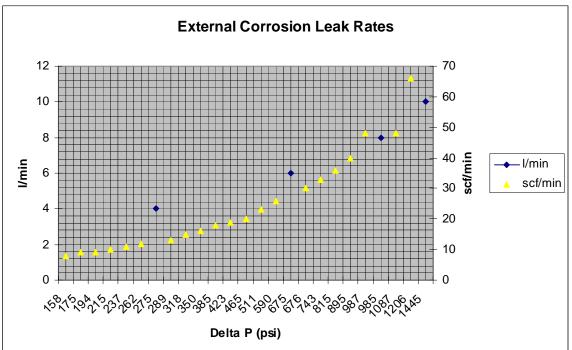


Photo 8: Internal Corrosion Leak Rate Testing



Photo 9: Internal Corrosion Leak Rate Testing



	Nitro	ogen		Water			
scf/min	scm/min	ΔP	ΔP	l/min	gpm	ΔP	ΔP
		MPa	psi			MPa	psi
79	2.240	8.230	1,193	10	2.64	9.550	1,385
56	1.590	7.250	1,052	9	2.38	6.930	1,005
47	1.330	6.510	944	6	1.59	4.520	655
42	1.190	5.870	851	5	1.32	2.550	370
35	0.991	5.050	733	4	1.06	1.620	235
30	0.850	4.320	627				
27	0.765	3.910	567				
25	0.708	3.530	512				
23	0.651	3.180	461				
20	0.566	2.870	416				
18	0.510	2.590	376				
17	0.481	2.340	339				
15	0.425	2.100	305				
13	0.368	1.800	261				
11	0.311	1.530	222				
10	0.283	1.370	199				
9	0.255	1.230	178				
9	0.255	1.100	159				

Table 6: Internal Corrosion Leak Rates

Chart 2: Internal Corrosion Leak Rates

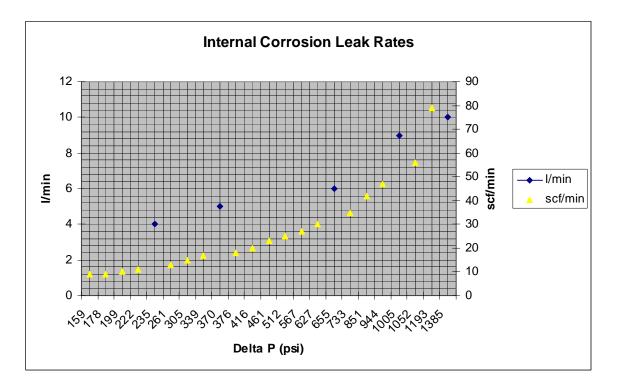


Photo 10: Pinhole Leak Rate Testing



Photo 11: Pinhole Leak Rate Testing



Table 7: Pinhole Leak Rates

	Nitro	ogen		Water				
scf/min	scm/min	ΔP	ΔP	l/min	gpm	ΔP	ΔP	
		MPa	psi			MPa	psi	
96	2.720	6.460	937	13	3.43	3.140	455	
71	2.010	5.250	761	12	3.17	2.650	385	
57	1.610	4.320	626	10	2.64	1.970	285	
47	1.330	3.560	516	10	2.64	1.900	275	
39	1.100	2.930	425	9	2.38	1.340	195	
32	0.906	2.410	349	9	2.38	1.280	185	
27	0.765	1.980	287					
23	0.651	1.610	234					
19	0.538	1.310	190					
16	0.453	1.060	154					
13	0.368	0.848	123					

Chart 3: Pinhole Leak Rates

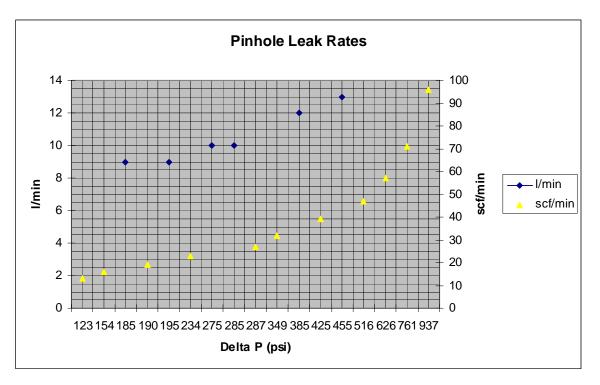
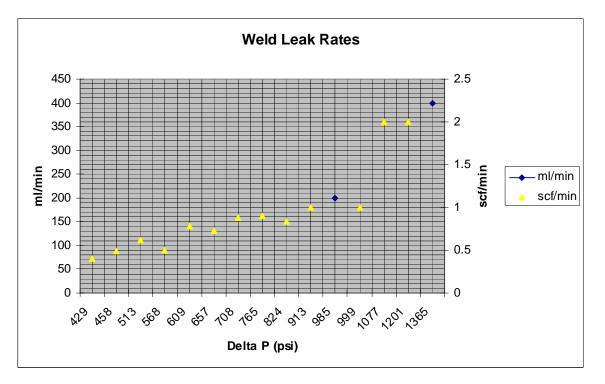


Table 8: Weld Leak Rates

	Nitro	ogen		Water				
scf/min	scm/min	ΔP	ΔP	l/min	gpm	ΔP	ΔP	
		MPa	psi			MPa	psi	
2.000	0.0566	8.280	1,201	0.4	0.11	9.410	1,365	
2.000	0.0566	7.430	1,077	0.2	0.05	6.790	985	
1.000	0.0283	6.890	999					
1.000	0.0283	6.290	913					
0.838	0.0237	5.680	824					
0.898	0.0254	5.270	765					
0.883	0.0250	4.880	708					
0.725	0.0205	4.530	657					
0.788	0.0223	4.200	609					
0.504	0.0143	3.920	568					
0.623	0.0176	3.540	513					
0.489	0.0138	3.160	458					
0.403	0.0114	2.960	429					

Chart 4: Weld Leak Rates



Data Reduction

The leaks rates, though large, were not considered to be beyond Seal-Tite's capabilities. Seal-Tite has previously cured downhole leaks in the 37.85 L (10 gal) per minute range. The critical factor was determined to be generating and maintaining a seal in a circular defect, as opposed to a split or crack. Circular defects are more difficult to seal since there is more open area than surface area. Also, the effect of pigging on seal integrity was unknown and needed to be explored in the full scale pipeline testing phase.

Full Scale Pipeline Testing

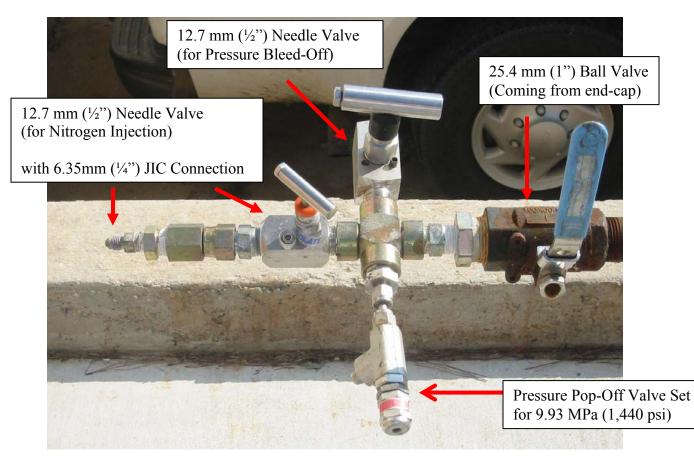
Overview / Objective

There were three objectives for the full scale pipeline testing: First, to test the feasibility of transporting the sealant between two pigs; secondly, to test sealant formulations necessary to seal the leak(s); and finally, to test the ability of the newly formed seal to withstand the effects of pigging.

Apparatus

In addition to the apparatus as described in the previous section, <u>Establishing Leak Rates</u>, each end-cap was fitted a manifold for the injection and regulation of nitrogen, as described in <u>Photo 12</u>.

Photo 12: Nitrogen Injection Manifold



Testing Procedures

- 1. With test model pressure bled to zero remove the launcher end-cap.
- 2. Close launcher gate valve and insert lead pig to gate valve.
- 3. Insert trailing pig into pipe, ensuring that pig does not cross sealant injection valve. Reinstall launcher cap.

- 4. Inject sealant volume between pigs in launcher section by pumping sealant into sealant injection ball valve.
- 5. Close sealant injection valve.
- 6. Pressure pipeline system to 200 psi with nitrogen from both receptor and launcher ends simultaneously.
- 7. Open launcher gate valve.
- 8. Move pigs & sealant train by regulating nitrogen pressure on receptor side through needle valve. Approximately 20 psi less on receptor side than launcher side moves pigs/sealant train to receptor.
- 9. When lead pig is across leak site (indicated by sealant extruding from defect in early tests indicated by electronic pig indicator on latter tests) open receptor needle valve fully to maintain equal pressure on upstream and downstream side of pigs.
- 10. Increase pressure until initial seal is formed. Shut in both receptor and launcher end-cap ball valves simultaneously to keep sealant train from moving pass leak site. Hold pressure for \underline{X} minutes.
- 11. Open both receptor and launcher ball valves simultaneously and utilize needle valves to incrementally increase pressure. Continue the pressure and hold cycles until final 9.65 MPa (1440 psi) seal is achieved.
- 12. Shut-in for cure cycle. Note initial shut-in pressure.
- 13. After designated curing time note final shut-in pressure. If final pressure is less than initial shut-in pressure retest seal by re-pressuring system to initial shut-in pressure from both receptor and launcher sides simultaneously. If seal maintained integrity proceed to Step 14. If seal broke then End Test.
- 14. Open drain valve and needle valves on receptor end and bleed pressure down to move pigs and sealant to receptor.
- 15. When pigs are in receptor (indicated by a reduction or elimination of pressure & fluid bleed-off) close drain and needle valves.
- 16. Re-pressure system from launcher side to final shut-in pressure to confirm trailing wiper pig did not destroy seal integrity. If seal maintained integrity proceed to Step 17. If seal broke then End Test.
- 17. Bleed pressure off test model through receptor side pressure bleed-off valve. Remove launcher cap and insert wiper assembly. Reinstall launcher cap.
- 18. Leave pressure bleed-off valve open (upstream of defect section).
- 19. Pressure launcher end and move pigs pass defect section.
- 20. Close pressure bleed-off valve and re-pressure system to final shut-in pressure. If seal maintained integrity proceed to Step 21. If seal broke then End Test.
- 21. Bleed pressure off test model. Remove launcher cap and insert scraper assembly.
- 22. Leaving a receptor side pressure bleed-off valve open, pressure launcher end and move pigs pass defect section.
- 23. Close pressure bleed-off valve and re-pressure system to final shut-in pressure. Record if seal maintained integrity or if seal broke. End Test.

Experimental Data

Results from Full Scale Pipeline Testing are summarized in <u>Table 9</u>, showing the defect being tested, defect orientation, type of sealant used, details on the types of pigs used, initial and shut-in pressures, shut-in duration and details and results of the test performed.

Defect Shut-In PSI Shut-In PSI Test No. Orientation Sealant Sealant Pigs (Front & Rear) Initial Shut-in Duration Final Wiper Pig Run Scraper Pig Run Pinhole Test I 7:1 Flo-Seal-P 6" OD, 5 lbs Density Foam 800 800 NA NA 7 o'clock 18 minutes Disc w/o Bullet Nose IC Test I 6" OD, 5 lbs Density Foam 6 o'clock 7:1 Flo-Seal-P 1440 18 hours + 20 minutes 1300 NA 6" OD, 5 lbs Density Foam Crisscross Wire Brush Pig with Disc w/o Bullet Nose Bullet Nose (refer to Photo 15, Page 21) EC Test I 6" OD, 5 lbs Density Foam 7 o'clock 7:1 Flo-Seal-P 1384 22 hours + 14 minutes 1047 NA 6" OD, 5 lbs Density Foam Disc Disc w/o Bullet Nose w/o Bullet Nose 6" OD, 5 lbs Density Foam Crisscross Wire Brush Pig with Bullet Nose 6" OD, 5 lbs Density Foam Disc w/o Bullet Nose EC Test II 7 o'clock 7:1 Flo-Seal-P 6" OD, 5 lbs Density Foam 1440 21 hours + 53 minutes 1336 (2) 6" OD, 5 lbs Density NA Disc w/o Bullet Nose Foam Disc w/o Bullet Nose

Table 9: Full Scale Pipeline Testing Summary

un Details / Results

Increased pressure to 257 psi. Held for 5 minutes. Increased pressure to 550 psi. Seal broke & resealed. Increased pressure to 635 psi. Held for 65 minutes. Increased pressure to 722 psi. Hammer union on pig launcher leaking. Increased pressure to 800 psi and maintained with nitrogen for 18 minutes. Moved sealant train to receptor. Seal broke when trailing sealant pig crossed defect.

- Foam Increased pressure to 232 psi. Held for 23 minutes. Increased pressure to 496 psi. Held for 15 minutes. Increased pressure to 776 psi. Seal broke & resealed. Increased pressure to 847 psi. Held for 113 minutes. Increased pressure to 1290 psi. Held for 22 minutes. Increased pressure to 1440 psi. Shut-in for 18hrs/20min. Re-pressured system to 1440 psi. Pressure held. Moved sealant train to receptor. Re-pressured system to 1440 psi. Pressure held. Ran scraper. Re-pressured system to 1440 psi. Pressure held.
- Toam DiscIncreased pressure to 203 psi. Held for 5 minutes.
Increased pressure to 515 psi. Held for 30 minutes.
Increased pressure to 880 psi. Held for 43 minutes.
Increased pressure to 1088 psi. Held for 31 minutes.
Increased pressure to 1384 psi. Nitrogen pressure
remaining in tanks was 1384 psi. Shut-in for
22hrs/14min. Replenished nitrogen supply. Re-
pressured system to 1440 psi. Pressure held.
Moved sealant train to receptor. Re-pressured
system to 1440 psi. Pressure held. Ran
wiper/scraper/wiper assembly. Seal broke at 254 psi
while attempting to re-pressure system to 1440 psi.

Increased pressure to 580 psi. Held for 15 minutes. Increased pressure to 880 psi. Held for 10 minutes. Increased pressure to 1128 psi. Held for 17 minutes. Increased pressure to 1440 psi. Shut-in for 21hrs/53min. Re-pressured system to 1440 psi. Pressure held. Moved sealant train to receptor. Repressured system to 1440 psi. Pressure held. Ran wiper assembly. Seal broke at 80 psi while attempting to re-pressure system to 1440 psi.

	Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig Run
	EC Test III	7 o'clock	7:1 Flo-Seal-P	6" OD, 5 lbs Density Foam Disc w/o Bullet Nose	1494	89 hours + 41 minutes	1270	NA	NA
F	Pinhole Test II	12 o'clock	7:1 Flo-Seal-P	6" OD, 5 lbs Density Foam Disc with Bullet Nose & Cavity Back for Magnetic Array	1440	65 hours + 6 minutes	1059	(2) 6" OD, 5 lbs Density Foam Disc with Bullet Nose & Cavity Back for Magnetic Array	NA
	Weld Test I	12 o'clock	9:1 Flo-Seal-P	6" OD, 5 lbs Density Foam Disc with Bullet Nose & Cavity Back for Magnetic Array	1440	40 hours + 14 minutes	1248	(2) 6" OD, 5 lbs Density Foam Disc with Bullet Nose & Cavity Back for Magnetic Array	6" OD, 5 lbs Density Foa w/o Bullet Nose + 6" OD, 5 lbs Density F Super Javelina Brush Pi Bullet Nose (refer to <u>Photo 16</u> , Pag

Table 9: Full Scale Pipeline Testing Summary (continued)

un Details / Results

Increased pressure to 910 psi. Held for 15 minutes. Increased pressure to 1195 psi. Held for 88 minutes. Increased pressure to 1236 psi. Held for 60 minutes. Increased pressure to 1335 psi. Held for 60 minutes. Increased pressure to 1420 psi. Held for 45 minutes. Increased pressure to 1440 psi. Held for 39 hrs/45 min. Observed sealant had filled hole like solder. Pressure dropped to 1317 psi. Increased pressure to 1440 psi. Held for 6hrs/17min. Due to thermal effects pressure had Increased to 1494 psi. Shut-in for 89hrs/41min. Re-pressured system to 1440 psi. Pressure held. Moved sealant train to receptor. While attempting to re-pressure to 1440 psi observed bubbling at defect hole at 925 psi. At 1175 psi seal completely broke indicating trailing sealant pig reduced seal integrity.

Increased pressure to 568 psi. Held for 7 minutes. Increased pressure to 597 psi. Held for 38 minutes. Increased pressure to 795 psi. Held for 73 minutes. Increased pressure to 1002 psi. Held for 52 minutes. Increased pressure to 1268 psi. Receptor side pinhole started leaking all nitrogen (no sealant). Extremely small leak. Postulating N2 passing top of pig from receptor side (passing front of bullet nose). After 10 minutes still leaking. Increased pressure to 1406 psi. Still leaking (all N2). Bled down system to 1000 psi and re-pressured to 1440 psi to try and reenergize pig. Still leaking. Shut-in for 65hrs/6min. Leak resealed at 1059 psi. Re-pressured system to 1440 psi. Pressure held. Moved sealant train to receptor. Re-pressured system to 1440 psi. Pressure held. Ran wiper assembly. Moved across defect. Re-pressured system to 1440 psi. Pressure held. Accidentally increased pressure to 1450 psi. Bubbles were observed at leak site. Must assume scraper pig would have destroyed seal integrity.

oam Disc Increased pressure to 978 psi. Held for 54 minutes. Increased pressure to 1339 psi. Held for 47 minutes. Increased pressure to 1440 psi. Shut-in for 40hrs/14min. Re-pressured system to 1440 psi.
Pressure held. Moved sealant train to receptor. Repressured system to 1440. Pressure held. Ran wiper assembly. Re-pressured system to 1440 psi.
Pressure held. Ran scraper assembly. While attempting to re-pressure system seal broke at 1100 psi. Small leak at 6 psi/min.

Data Reduction

Delivery Methods

The first two tests, which are not displayed in <u>Table 9: Full Scale Pipeline Testing</u> <u>Summary</u>, were preliminary tests to determine the volume of sealant needed to compensate for loss of sealant volume due to hoses and pump and to determine the optimum pig type to minimize sealant bypass.

The 32 kg/m³ (2 lbm/ft³) density yellow swab pig (<u>Photo 13</u>) was tested for use in multidiameter pipelines where more rigid pigs are not as easily transported. During preliminary testing, it was noted that while straddling the liquid sealant the swab pig acted like a sponge and became saturated, resulting in the nitrogen creating channels around the pigs and subsequently not allowing the nitrogen to move the sealant train. Higher gas rates that are experienced in the field most likely would have moved the sealant train.

The 80 kg/m³ (5 lbm/ft³) density foam disc pigs (<u>Photo 14</u>) on the other hand formed a tight seal against the pipe internal diameter and were very easily moved by nitrogen with only a 20 psi differential.

Photo 13 2 lbm/ft³ Yellow Swab Pig with Nose







The first five tests recorded in <u>Table 9: Full Scale Pipeline Testing Summary</u> were performed by gauging when the sealant train crossed the defect area by observation of sealant extruding through the leak site. Later tests were performed utilizing electronic pig detectors. Both methods were effective in aligning the sealant train across the defect.

Sealing Simulated Leaks

We were successful in obtaining a 9.93 MPa (1440 psi) seal in all seven tests conducted, with leak rates ranging from a low of 0.0114 scm/min (0.403 scf/min) to a high of 2.720 scm/min (96 scf/min) at 2.96 MPa (429 psi) and 6.46 MPa (937 psi) respectively, and with leak orientation varying from 6 o'clock low side to 12 o'clock high side.

Referring to <u>Table 10</u>, we can conclude that the effects of additional wiping and/or scraping are directly related to defect geometry. External Corrosion leaks with a large amount of external wall loss and an inverted funnel configuration were the least resistant to the effects of pigging; Pinhole and Weld leaks, with no loss of wall thickness were more resistant; and Internal Corrosion leaks, with a funnel configuration and an internal "valley" for sealant reserve were the most resistant.

Table 10: Results of Seal Integrity after Pigging

	Trailing Sealant Pig	Wiper Assembly	Scraper Assembly
Test 5: EC I	Passed	Not Run	Failed ¹
Test 6: EC II	Passed	Failed ²	NA
Test 7: EC III	Failed	NA	NA
Test 4: IC I	Passed	Not Run	Passed
Test 3: Pinhole I	Failed ³	NA	NA
Test 8: Pinhole II	Passed	Passed ⁴	Not Run ⁵
Test 9: Weld I	Passed	Passed ⁶	Failed ⁷

¹wiper/scraper/wiper
^{2,4,6} (2) wipers
³ Low pressure seal 5.52 MPa (800 psi) and short curing time (18 minutes)
⁵ At 10 MPa (1450 psi) leak started to bubble. Most likely seal would not have withstood effects of scraping
⁷wiper/scraper

Photo 15: 5 lbm/ft³ Foam Crisscross Wire Brush Pig with Nose



Photo 16: 5 lbm/ft³ Foam Super Javelina Brush Pig with Nose



From the data collected during this testing stage the following conclusions can be made:

- 1. Foam Disc pigs are the preferred pigs for isolation and transporting sealant to the leak site.
- 2. All leaks were successfully sealed to 9.93 MPa (1440 psi).
- 3. Leak orientation had no effect on quality of seal generated.
- 4. At the low curing pressure of 9.93 MPa (1440 psi) pigging does affect seal integrity.
- 5. Leak geometry plays a large role in maintaining seal integrity after pigging operations.
- 6. The effect of curing time on seal quality of a low pressure seal is still unknown.

Lab Tests to Determine the Effect of Curing Time on Seal Quality

Overview / Objectives

The objective of these tests was to determine if length of curing time had a favorable effect on a seal generated at 9.93 MPa (1440 psi). By conducting these tests in a controlled environment, pressure fluctuations due to temperature effects were minimized. Also any effect that "additional" curing time may have had was minimized by reducing the amount of pressure stages or cycles by immediately bringing the pressure up to 9.93 MPa (1440 psi).

Apparatus

The defect sections utilized were the same as previously described under <u>Establishing</u> <u>Leak Rates</u>. In addition, as seen in <u>Photo 17</u> and <u>Photo 18</u> below, the defect section was fitted with a blind flange on bottom and a ported flange with a ball valve, needle valve with 6.35 mm ($^{1}/_{4}$ ") JIC connection for nitrogen injection, and gauge on top.

Photo 17: Full Scale Lab Fixture Photo 18: Full Scale Lab Fixture



Testing Procedure

- 1. Install blind flange on bottom of defect section (bottom is end with defect, except for pinhole defect which had defects at each end).
- 2. Stand defect section vertical and fill to top with sealant formulation.
- 3. Install flange with ball valve, needle valve with nitrogen connection and gauge on top.

- 4. Inject nitrogen until pressure reaches 9.93 MPa (1440 psi).
- 5. Shut-in and monitor for 30 minutes.
- 6. Continue steps 4 and 5 until zero bleed-off after 30 minutes.
- 7. Shut-in for designated time (defects assign alphabetically):
 - a. External Corrosion 48 hours
 - b. Internal Corrosion 96 hours
 - c. Pinholes 144 hours
 - d. Weld 192 hours
- 8. After shut-in period re-pressure system to 9.93 MPa (1440 psi), if needed, to verify seal integrity. If pressure held proceed to step 9. If pressure didn't hold then End Test.
- 9. Bleed-off pressure and open assembly. Observe and note.

Experimental Data

Results from this testing stage are summarized in <u>Table 11</u>, Page 25, showing the defect being tested, defect orientation, type of sealant used, details on the types of pigs used, initial and shut-in pressures, shut-in duration and details and results of the test performed.

		Table 11: Effect of Curing Time on Seal Quality Testing Summary								
Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig I		
External Corrosion Test	NA	7:1 Flo-Seal-P	NA	1440	48 hours	1400	NA	NA		
Internal Corrosion Test	NA	7:1 Flo-Seal-P	NA	1440	96 hours	1400	NA	NA		
Pinhole Test	NA	7:1 Flo-Seal-P	NA	1440	144 hours	1375	NA	NA		
Weld Test	NA	9:1 Flo-Seal-P	NA	1440	192 hours	1350	NA	NA		

g Run Details / Results

Increased pressure to 1440 psi. Zero pressure bleed-off in 30 minutes. Shut-in at 1440 psi for 48 hours. Re-pressured system to 1440 psi. Pressure held. Bled-off pressure and opened assembly. Observed soft lump of sealant internally across leak site. Lump was probed with pick. A hard flake, or bit, was embedded in the sealant mass. No flake, or bit, in defect hole. No external extrusion of cured sealant.

Increased pressure to 1440 psi. Bled to 975 psi in 30 min. Increased pressure to 1440 psi. Bled to 1175 psi in 30 min. Increased pressure to 1440 psi. Bled to 1260 psi in 30 min. Increased pressure to 1440 psi. Bled to 1275 psi in 30 min. Increased pressure to 1440 psi. Bled to 1300 psi in 30 min. Increased pressure to 1440 psi. Bled to 1315 psi in 30 min. Increased pressure to 1440 psi. Bled to 1380 psi in 30 min. Increased pressure to 1440 psi. Pressure held. Shut-in. Sometime during 64 hours seal had broken and resealed at 750 psi. Increased pressure to 1400 psi. Bled to 1340 psi in 45 minutes. Increased pressure to 1440 psi. Bled to 1425 psi in 30 min. Increased pressure to 1440 psi. Zero pressure bleed-off in 30 minutes. Shut-in @ 1440 psi for 96 hours. Re-pressured system to 1440 psi. Pressure held. Bled-off pressure and opened assembly. Observed soft lump of sealant internally across leak site. Lump was probed with pick. A hard flake, or bit, was embedded in the sealant mass. No bit, or flake, in defect hole. This was the only test with cured sealant protruding externally from the leak.

Increased pressure to 1440 psi. Bled to 1100 psi in 30 min. Increased pressure to 1440 psi. Zero pressure bleed-off in 30 minutes. Shut-in @ 1440 psi for 144 hours. Re-pressured system to 1440 psi. Pressure held. Bled-off pressure and opened assembly. Observed soft lumps of sealant internally across both leak sites. Lumps were probed with pick. A hard flake, or bit, was embedded in the uppermost sealant mass. Neither pinhole had bits, or flakes, within the hole. No external extrusion of cured sealant through either pinhole.

Increased pressure to 1440 psi. Held for 30 minutes. Shut-in @ 1440 psi for 192 hours. Repressured system to 1440 psi. Held. Bled-off pressure and opened assembly. No internal sealant lump was observed. ID smooth around leak site. No external extrusion of cured sealant.

Data Reduction

This testing stage has shown that at a sealing pressure of only 9.93 MPa (1440 psi), curing time has no effect on the quality of the seal generated. The sealant formulations necessary to seal the pinhole sizes that are represented in our testing require particulates (bits or flakes) that are forced into the defect and, under pressure, expand to form a bridge that allows the polymers and monomers to create a seal. In the weld leak, sealant penetration is also required to aid in generating a seal that can withstand the effects of scraping.

The soft lump of sealant in the pinhole defects (<u>Photo 19</u>) indicates that we are not achieving penetration into the leak site with the particulates and the seal that is being generated is a superficial seal across the interior wall. The same can be concluded on the weld leak. Although there was not an internal soft lump of sealant due to the low leak rate, there was also no indication of cured sealant extruding through the leak site.

For the next stage we branched off our testing in two directions:

- 1. To evaluate our most aggressive non-particulate sealant on pinhole defects to use as a benchmark in developing other formulations.
- 2. To test less aggressive, non-particulate formulations in order to achieve deeper penetration in the weld defect.

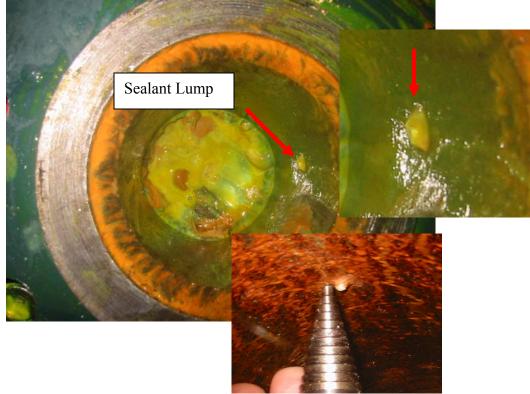


Photo 19: Soft Sealant Lump

Lab Tests to Determine Optimum Sealant for Weld Penetration

Overview / Objectives

The objective of these tests was to determine the optimum sealant formulation to penetrate the small leak rate that was exhibited by the weld defect.

Apparatus

The defect sections used were the same as previously described in <u>Lab Tests to</u> <u>Determine the Effect of Curing Time on Seal Quality</u>.

Testing Procedures

- 1. Install blind flange on bottom of weld defect section (bottom is end with defect).
- 2. Stand defect section vertical and fill above defect with sealant formulation.
- 3. Install flange with ball valve, needle valve with nitrogen connection and gauge on top.
- 4. Increase pressure until initial seal is formed. Shut in for \underline{X} minutes
- 5. Increase pressure and shut-in in increments until final 9.93 MPa (1440 psi) seal is achieved with no bleed-off.

Note: Atomization procedure injected sealant continuously until final pressure was reached

- 6. Shut-in for 24 hour cure cycle. Note initial shut-in pressure.
- 7. After designated curing time note final shut-in pressure. If final shut-in pressure is less than initial shut-in pressure retest seal by re-pressuring system to initial shut-in pressure. If seal maintained integrity proceed to next step. If seal broke then End Test.
- 8. Bleed-off pressure and open assembly.
- 9. Observe and note.
- 10. Pig defect.
- 11. Observe and note.
- 12. Reseal test fixture and re-pressure system to 9.93 MPa (1440 psi).
- 13. If re-pressure test fails record pressure and End Test. If seal retained integrity repeat steps 10, 11 & 12 with a different pig type. Continue steps until re-pressure test fails.
- 14. Inject Seal-Tite's Valve-Flush into leak to remove any cured sealant.
- 15. Set-up fixture for next test.

Experimental Data

Results from this testing stage are summarized in <u>Table 12</u>, showing the defect being tested, defect orientation, type of sealant used, details on the types of pigs used, initial and shut-in pressures, shut-in duration and details and results of the test performed.

Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig F
Weld Test I	NA	Gly-Flo "Neat"	NA	1440	24 hours	1395	6" OD, 2 lbs Density Yellow Bare Swab w/ Bullet Nose	NA
Weld Test II	NA	Gly-Flo "G"	NA	1440	24 hours	1426	(2) 6" OD, 2 lbs Density Yellow Bare Swab w/ Bullet Nose	6" OD, 5lbs De Foam Super Ja Brush Pig w Bullet Nose
Weld Test III	NA	10% Gly-Flo	NA	1440	24 hours	1420	6" OD, 2 lbs Density Yellow Bare Swab w/	NA

Table 12: Lab Tests to Determine Optimum Sealant for Weld Penetration Testing Summary

g Run Details / Results

Increased pressure to 486 psi. Pressure held for 15 min. Increased pressure to 780 psi. Pressure held for 15 min. Increased pressure to 1070 psi. Pressure held for 15 min. Increased pressure to 1418 psi. Pressure held for 15 min. Increased pressure to 1440 psi. Pressure held for 15 min. Shut-in @ 1440 psi for 24 hours. Re-pressured system to 1440 psi. Pressure held. Bled-off pressure and opened assembly. Observed 3/8" high soft lump of sealant internally around leak site. No external extrusion of any sealant, liquid or cured. After pigging soft sealant lump was completely removed. Re-pressure test failed when bubbles were observed at 1418 psi.

Density Javelina with ose Increased pressure to 484 psi. Pressure held for 15 min. Increased pressure to 786 psi. Pressure held for 15 min. Increased pressure to 1064 psi. Pressure held for 23 min. Increased pressure to 1415 psi. Pressure held for 15 min. Increased pressure to 1440 psi. Pressure held for 40 min. Shut-in @ 1440 psi for 24 hours. Re-pressured system to 1440 psi. Pressure held. Bled-off pressure and opened assembly. Observed 5/8" high lump of sealant around leak site, with a medium consistency. No external extrusion of any sealant, liquid or cured. After wiper run (2 passes) sealant lump was reduced to 1/16" in height. Repressure test held at 1440 psi. Bled-off pressure and ran scraper pig. Re-pressure test failed when bubbles were observed after pressuring back to 1440 psi. Leaking at 3-4 psi/min.

Increased pressure to 498 psi. Pressure held for 15 min. Increased pressure to 791 psi. Pressure held for 15 min. Increased pressure to 1067 psi. Pressure held for 22 min. Increased pressure to 1440 psi. Pressure held for 30 min. Shut-in @ 1440 psi for 24 hours. Re-pressured system to 1440 psi. Pressure held. Bled-off pressure and opened assembly. Observed 1/16" high strip of cured "hardened" sealant across line of defect. Observed external extrusion of liquid sealant through leak site. After pigging no visual change in appearance of internal seal. Re-pressure test failed when bubbles were observed at 850 psi.

 Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig Ru
Weld Test IV	NA	15% Gly-Flo	NA	1440	24 hours	1446	6" OD, 2 lbs Density Yellow Bare Swab w/ Bullet Nose	NA
Weld Test V	NA	Atomized 15% Gly-Flo	NA	1440	24 hours	1440	6" OD, 2 lbs Density Yellow Bare Swab w/ Bullet Nose	NA

Table 12: Lab Tests to Determine Optimum Sealant for Weld Penetration Testing Summary (continued)

Run Details / Results

Increased pressure to 501 psi. Pressure held for 90 min. Increased pressure to 785 psi. Pressure held for 60 min. Increase pressure to 1077 psi. Pressure held for 60 min. Increase pressure to 1422 psi. Pressure held for 65 min. Increase pressure to 1440 psi. Pressure held for 60 min. Shut-in @ 1440 psi for 24 hours. Pressure at 1446 psi (Thermal Effects). Bled-off pressure and opened assembly. Observed 3-1/2" long "tacky" sealant strip internally across leak site. Mainly 1/16" high with 1 spot about 3/8" high. No extrusion of liquid sealant observed during pressure stages. After pigging, re-pressure test failed at 1050 psi.

Atomize sealant into test section until pressure reached 1440 psi. Shut-in for 17hrs/12min. Pressure had dropped to 1385 psi. Atomize sealant into test section until pressure reached 1440 psi. Pressure held for 30 min. Shut-in @ 1440 psi for 24 hours. Bled-off pressure and opened assembly. Observed no visible ridge of sealant internally across leak site and no visible sealant externally extruded. Re-pressure test after pigging immediately failed at less than 100 psi.

The test data shows that only during <u>Weld Test II</u>, using Gly-Flo "G" sealant, was enough penetration achieved to withstand the effects of the wiper pigs. The scraper run failed at maximum pressure 9.93 MPa (1440 psi) with the most aggressive wire brush pig available. A run with a lesser aggressive scraper pig may have had better results.

In comparing this test to the weld defect test done on the full scale pipeline model utilizing 9:1 Flo-Seal-P as the sealant, the Gly-Flo "G" sealant had better results after scraping. With no particulates, the Gly-Flo "G" was able to achieve deeper penetration into the weld leak before activation.

It can also be noted from the results that Gly-Flo "G" had a strip of medium consistency, as seen in <u>Photo 20</u>. The other sealant formulations resulted in either a soft lump, which was wiped off with the swab wiper pig (<u>Photo 21</u> and <u>Photo 22</u>), or a hardened strip of sealant, which probably resulted in the seal being pulled out of the defect by the wiper pig.

Photo 20: Gly-Flo "G" Sealant Strip



Photo 21: Soft Sealant Lump







Flex-Plug Testing

Overview / Objectives

The objective of these tests was to utilize Flex-Plug, our most aggressive, non-particulate sealant, for:

- 1. Determining if 1.59 mm (1/16") pinholes were within our capabilities of generating a seal that could withstand the effects of scraping.
- 2. Establishing the maximum pinhole size that is within our capability for generating a seal that could withstand the effects of scraping if the 1.59 mm (1/16") pinholes tests failed.
- 3. Aiding in determining what modifications needed to be made to sealant formulations in order to enhance the ability to seal pinholes in pipe body, pipe welds, and internal corrosion defects.

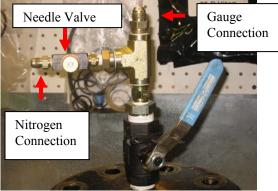
Apparatus

The internal corrosion defect used in this testing stage was the same described in <u>Establishing Leak Rates</u>. For the pinholes in varying sizes we made 114.3 mm OD x 97.2 mm ID x 152.4 mm L (4-1/2" OD x 3.826" ID x 6" L) Schedule 80 test fixtures. The fixture had a steel plate on bottom that would allow fixture to stand vertically and a ported flange on top for connection of a nitrogen injection and gauge manifold (<u>Photo 23</u> and <u>Photo 24</u>).

Photo 23: 114.3 mm (4-1/2") Test Fixture







Testing Procedures

- 1. With defect at 6 o'clock inject Flex-Plug sealant into defect hole with syringe.
- 2. Scrape internally to remove excess sealant.
- 3. Let stand to atmosphere for 24 hours.
- 4. Pressure test to 9.93 MPa (1440 psi). If pressure test fails record pressure and End Test. If seal retained integrity proceed to Step 5.
- 5. Scrape defect. Re-pressure to 9.93 MPa (1440 psi). Note and record results.

Experimental Data

Results from this testing stage are summarized in <u>Table 13</u>, showing the defect being tested, defect orientation, type of sealant used, details on the types of pigs used, initial and shut-in pressures, shut-in duration and details and results of the test performed.

Table 13: Flex-Plug Testing Summary

Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig Run	Details / Results
1/16" Pinhole in Pipe Body Test I	6 o'clock	Flex-Plug	Refer to Details / Results	Atmosphere	24 hours	Atmosphere	NA	NA	Injected sealant into defect with syringe. During injection sealant poured through defect with no observable friction. Scraped small bubble of sealant off interior wall to simulate pigging. Left open to atmosphere for 24 hours. After 24 hours pressure test failed at 550 psi when sealant slug blew out like a bullet. No remnants of sealant in pinhole.
1/16" Pinhole in Pipe Weld Test I	6 o'clock	Flex-Plug	Refer to Details / Results	Atmosphere	72 hours	Atmosphere	NA	NA	Injected sealant into defect with syringe. During injection sealant poured through defect with no observable friction. Scraped small bubble of sealant off interior wall to simulate pigging. Left open to atmosphere for 72 hours. After 72 hours pressure test failed at 1400 psi when sealant slug blew out like a bullet. No remnants of sealant in pinhole.
1/16" Internal Corrosion Pinhole Test I	6 o'clock	Flex-Plug	Refer to Details / Results	Atmosphere	24 hours	Atmosphere	NA	6" OD, 5 lbs Density Foam Super Javelina Brush Pig with Bullet Nose	Injected sealant into defect with syringe. After injecting sealant scraped ID to remove excess sealant. Left open to atmosphere for 24 hours. After 24 hours observed ID smooth and OD had 1/4" high sealant bubble. Pressure test to 1440 psi and held for 30 minutes. Ran scraper pig. Re- pressure to 1440. Pressure held.
3/64" Pinhole in Pipe Body Test I	6 o'clock	Flex-Plug	Refer to Details / Results	Atmosphere	18 hours	Atmosphere	NA	Refer to Details / Results	Injected into defect with syringe. Scraped small bubble of sealant off interior wall to simulate pigging. Left open to atmosphere for 18 hours. After 18 hours pressured fixture to 1440 psi. Pressure held. Hand scraped with wire brush. Re-pressured to 1440 psi. Pressure held.
1/32" Pinhole in Pipe Body Test I	6 o'clock	Flex-Plug	Refer to Details / Results	Atmosphere	18 hours	Atmosphere	NA	Refer to Details / Results	Injected into defect with syringe. Scraped small bubble of sealant off interior wall to simulate pigging. Left open to atmosphere for 18 hours. After 18 hours pressured fixture to 1440 psi. Pressure held. Hand scraped with wire brush. Re-pressure to 1440 psi. Pressure Held.
1/64" Pinhole in Pipe Body Test I	6 o'clock	Flex-Plug	Refer to Details / Results	Atmosphere	18 hours	Atmosphere	NA	Refer to Details / Results	Injected into defect with syringe. Scraped small bubble of sealant off interior wall to simulate pigging. Left open to atmosphere for 18 hours. After 18 hours pressured fixture to 1440 psi. Pressure held. Hand scraped with wire brush. Re-pressure to 1440 psi. Pressure held.

Four of the six tests were successful in obtaining a seal that could withstand the effects of scraping. The only 1.59 mm (1/16") pinhole that maintained integrity was on an internal corrosion defect (<u>Photo 25</u>, <u>Photo 26</u>, <u>Photo 27</u> and <u>Photo 28</u>). This confirms our conclusion under <u>Full Scale Pipeline Testing</u> that the geometry of internal corrosion defects is advantageous for resisting the effects of pigging.

The possibility of sealing 1.59 mm (1/16") pinholes in other defect types and having the seal maintain integrity after scraping will require a sealant formulation with particulates and the ability to achieve penetration into the defect before activation. Testing also showed that the 1.19 mm (3/64"), 0.79 mm (1/32") and 0.40 mm (1/64") pinholes are within our capability but new less aggressive sealant formulations that can be transported between pigs need to be developed.

Photo 25: Internal Corrosion Defect - Bare



Photo 26: Internal Corrosion Defect with Flex-Plug



Photo 27: External View before Flex-Plug



Photo 28: External View with Flex-Plug Extrusion



Lab Tests to Determine Optimum Sealant for Pinholes

Overview / Objectives

The objective of this stage was to determine the optimum sealant for curing pinholes by utilizing the data collected during the previous tests.

Apparatus

The apparatus utilized during this stage was the same as described under <u>Flex-Plug</u> <u>Testing</u>.

Testing Procedures

- 1. Stand test fixture vertical and fill with sealant formulation.
- 2. Install flange with ball valve, needle valve with nitrogen connection and gauge on top.
- 3. Increase pressure until initial seal is formed. Shut in for \underline{X} minutes
- 4. Increase pressure and shut-in in increments until final 9.93 MPa (1440 psi) seal is achieved with no bleed-off.

Note: Atomization procedure attempted to inject sealant continuously until maximum pressure is reached

- 5. Shut-in for cure cycle. Note initial shut-in pressure.
- 6. After designated curing time note final shut-in pressure. If final shut-in pressure is less than initial shut-in pressure retest seal by re-pressuring system to initial shut-in pressure. If seal maintained integrity proceed to next step. If seal broke then End Test.
- 7. Bleed-off pressure and open assembly.
- 8. Observe and note.
- 9. Pig defect with rubber disc pig (Photo 29).
- 10. Observe and note.
- 11. Reseal test fixture and re-pressure system to 9.93 MPa (1440 psi).
- 12. If re-pressure test fails record pressure and End Test. If seal retained integrity repeat steps 10, 11 & 12 with a different pig type. Continue steps until re-pressure test fails.
- 13. Inject Seal-Tite's Valve-Flush into leak to remove any cured sealant.
- 14. Set-up fixture for next test.





Experimental Data

Results from this testing stage are summarized in <u>Table 14</u>, showing the defect being tested, defect orientation, type of sealant used, details on the types of pigs used, initial and shut-in pressures, shut-in duration and details and results of the test performed.

							8	5
Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig Ru
1/16" Pinhole in Body Test I	NA "Vertical"	Tur-Flo-P	NA	1440	72 hours	750	4" Rubber Disc	NA
				4440		4000		
1/16" Pinhole in Body Test II	NA "Vertical"	Gly-Flo Flo-Seal-P Mixture	NA	1440	23 hours	1266	4" Rubber Disc	NA

Table 14: Lab Tests to Determine Optimum Sealant for Pinholes Testing Summary

3/64" Pinhole in Body Test I	NA "Vertical"	Atomized Gly- Flo "G"	NA	NA	NA	NA	NA	NA
3/64" Pinhole in Body Test II	NA "Vertical"	Gly-Flo "G"	NA	NA	NA	NA	NA	NA
3/64" Pinhole in Body Test III	NA "Vertical"	Gly-Flo "G"	NA	NA	NA	NA	NA	NA

Details / Results Run

During sealant injection stage sealant "squirted" out of leak site and pressure immediately ramped up from 0 psi to 1200 psi to 1440 psi. Shut-in for 72 hours at 1440 psi. After 72 hours re-pressured test fixture to 1440. Pressure held. Bled pressure and opened fixture. Pushed in and pulled out 4" rubber disc pig. Pig did not remove all of sealant across leak site. Re-pressure test failed at 710 psi.

Increased pressure to 1340 psi. Held for 90 minutes. Increased pressure to 1459 psi. At some point within 95 minutes seal broke and resealed at 770 psi. Increase pressure to 1400 psi. Seal broke and resealed at 350 psi. Increased pressure to 996 psi. Held for 35 minutes. Increased pressure to 1440 psi. Seal broke and resealed at 300 psi. Increased pressure to 1440 psi. Shut-in at 1440 psi for 23 hours. Note: Every time seal broke "rubbery" semi-cured sealant was expelled through leak site. After 23 hours re-pressured fixture to 1440 psi. Pressure held. Bled-off pressure and opened fixture. Observed a string of cured sealant in the pinhole but cured sealant appeared smaller than the ID of the hole. After pushing and pulling the rubber disc pig in and out of the fixture the attempt at re-pressuring the fixture failed immediately.

While atomizing sealant pressure built to 250 psi then broke. Built pressure back up to 450 psi and seal broke again. Unable to re-establish a seal.

While injecting sealant no seal was achieved until 1290 psi. Seal broke immediately after shutting-in. Unable to re-establish a seal.

Increased pressure to 225 psi. Held for 30 minutes. Increased pressure to 500 psi. Held for 60 minutes. Increased pressure to 781 psi. Seal broke. Increased pressure to 258 psi. Held for 60 minutes. Increased pressure to 520 psi. Held for 15 hours. Increased pressure to 758 psi. Held for 60 minutes. Increased pressure to 1001 psi. Seal broke. Increased pressure to 63 psi. Held for 60 minutes. Increased pressure to 250 psi. Seal broke. Unable to re-establish a seal.

Table 14: Lab Tests to Determine Optimum Sealant for Pinholes Testing Summary (continued)

_	Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig R
	1/32" Pinhole in Body Test I	NA "Vertical"	Gly-Flo "G"	NA	NA	NA	NA	NA	NA
	1/64" Pinhole in Body Test I	NA "Vertical"	Gly-Flo "G"	NA	1440	64	1447	4" Rubber Disc	NA

Run Details / Results

Increased pressure to 102 psi. Held for 20 minutes. Increased pressure to 267 psi. Held for 60 minutes. Increased pressure to 508 psi. Seal broke. Unable to generate seal again.

Increased pressure to 250 psi. Held for 75 minutes. Increased pressure to 520 psi. Held for 60 minutes. Increased pressure to 768 psi. Held for 40 minutes. Increased pressure to 1001 psi. Held for 30 minutes. Increased pressure to 1326 psi. Held for 60 minutes. Increased pressure to 1440 psi. Held for 60 minutes. Shut-in for 64 hours @ 1440 psi. After 64 hours bled-off pressure and opened fixture. Observed 1/2" x 1/4" oval tacky lump of sealant (1/8" high) across internal leak site. Pushed in and pulled out 4" rubber disc pig. Observed that pigging had removed sealant lump. No remnant of sealant in pinhole. Attempt to re-pressure fixture to 1440 psi failed immediately.

When we were able to generate a seal with a predominately non-particulate sealant (Gly-Flo "G" and Gly-Flo/Flo-Seal-P Mixture) the results after pigging were not good. The only time we were able to establish a seal that, after pigging, retained some integrity was with Tur-Flo, a particulate based sealant.

The next testing stage will test the theory that Flo-Seal-P, at higher curing pressures, would seal the 1.59 mm (1/16") pinholes, and retain integrity after pigging. If this theory proves out then we will attempt to modify the Flo-Seal formula to achieve the same results at pipeline pressures.

High Pressure Testing

Overview / Objectives

The objective of this testing stage was to confirm or refute our theory that a seal establish in $1.59 \text{ mm} (1/16^{\circ})$ pinholes with Flo-Seal-P at pressures indicative of downhole petroleum applications would withstand the effects of pigging; and if this theory was proven then proceed with modifying sealant formulation to achieve the same results at pipeline pressures.

Apparatus

An 88.9 mm OD x 73.7 mm ID x 177.8 mm L $(3-1/2" \times 2.90" \times 7")$ 4140 carbon steel test fixture was made with a 1.59 mm (1/16") pinhole in pipe body. End caps (57.2 mm long) were threaded on each end for an overall length of 190.5 mm (7-1/2"). Each end cap was threaded to accept a gauge on one end and a needle valve with a connection for nitrogen injection on the opposing end, as seen in <u>Photo 30</u> below.

Photo 30: High Pressure Test Fixture



Testing Procedures

- 1. With injection side end cap removed and pinhole at 6 o'clock fill cylinder with sealant.
- 2. Install nitrogen injection end cap and begin injecting nitrogen.
- 3. Increase pressure in increments and hold for \underline{X} minutes.
- 4. Continue pressure cycles until seal holds at 34.47 MPa (5,000 psi) (Photo 31 and Photo 32).
- 5. Bleed pressure from cylinder and open.
- 6. Drain remaining liquid sealant and observe and note seal (Photo 33 and Photo 34).
- Remove approximately ½ of seal height and re-pressure assembly to 34.47 MPa (5,000 psi). If fails then End Test. If pressure held proceed to Step 8.

- 8. Remove ½ of remaining seal and re-pressure cylinder to 34.47 MPA. If fails then End Test. If pressure held proceed to Step 9.
- 9. Remove all of remaining seal on interior wall and observe. Re-pressure cylinder to 34.47 MPa. If fails then End Test. If pressure held proceed to Step 10.
- 10. Run wire brush across defect 8 times to simulate a wire brush pig run. Observe and note. Re-pressure cylinder to 34.47 MPa. If fails then End Test. If pressure held proceed to Step 11.
- 11. Repeat the above steps with different sealant formulations to achieve the same results at pipeline pressures 9.93 MPa (1440 psi).

Photo 31: Sealant Extruding Through Pinhole



Photo 32: High Pressure Seal at 34.47 MPa (5,000 psi)



Photo 33: Draining Uncured Sealant from Cylinder



Photo 34: Sealant Across Defect



Experimental Data

Results from this testing stage are summarized in <u>Table 15</u>, showing the defect being tested, defect orientation, type of sealant used, details on the types of pigs used, initial and shut-in pressures, shut-in duration and details and results of the test performed.

Table 15: High Pressure Testing Summary

Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig Ru
 1/16" Pinhole in Body Test I	6 o'clock	7:1 Flo-Seal-P	NA	5000	24 hours + 30 minutes	5000	Poly Foam	Wire Brush

1/16" Pinhole in Body Test II	6 o'clock	7:1 Flo-Seal-P with "Smaller" Bits	NA	1440	90 min	1440	Refer to Details / Results	NA
1/16" Pinhole in Body Test III	6 o'clock	9:1 Flo-Seal-P with "Smaller" Bits	NA	1440	30 min	1440	Refer to Details / Results	NA

g Run Details / Results

sh Filled test fixture with sealant. Increased pressure to 2000 psi. Held for 32 minutes. Pressure dropped to 1750 psi. Increased pressure to 2500 psi. Pressure held for 5 minutes. Increased pressure to 3000 psi. Seal Broke. Seal resealed at 1400 psi. Held for 30 minutes. Increased pressure to 3100 psi. Seal Broke. Seal resealed at 2950 psi. Held for 30 minutes. Increased pressure to 4100 psi. Pressure held for 15 minutes. Increased pressure to 4500 psi. Pressure held for 5 minutes. Increased pressure to 5000 psi. Pressure held for 30 minutes. Shut-in at 5000 psi for 24 hours. After shut-in opened and observed mound of semi-hard sealant at internal leak site. Mound 5/8" high x 1-1/4" long. Cleaned fixture of excess sealant with water. Repressured to 5000 psi. Held for 15 minutes. Opened and removed 1/2 of mound height with pick. Re-pressured to 5000 psi and held. Removed 1/2 of remaining mound and repressured to 5000 psi. Pressure held. Poly-pigged interior defect by hand. A "bit" remained lodged in hole with cured sealant around it. Pressured to 5300 psi. Held. Open and made sure all excess sealant was thoroughly removed. Re-pressured to 5000 psi. Held. Ran 1" in length wire brush 8 times on interior defect. Re-pressured to 5000 psi. Held. Opened and pulled "bit" out of hole with needle nosed pliers. Very difficult to pull. Re-pressure attempt leaked immediately.

> Filled test fixture with sealant. Increased pressure to 1400 psi. Held for 33 minutes. Pressure dropped to 1350 psi. Increased pressure to 1440 psi. Shut-in for 90 minutes. Pressure held. After 90 minutes bled down and drained test fixture. Observed "hardened" mass of sealant across leak site internally approximately 1/2" high and 1" long. With pick removed mass from interior wall. No "bit" was observed in pinhole; wall around pinhole was completely clean. With finger no "bit" or particulate was felt in or around hole. Repressure failed immediately.

> Filled test fixture with sealant. With nitrogen increased pressure to 1440 psi. Pressure held for 30 minutes. After shut-in bled down and drained test fixture. Mound of flakes around leak site. When "scraped" away with spatula nothing remained in pinhole...no particulate or "bit" in pinhole.

Test No.	Defect Orientation	Sealant	Sealant Pigs (Front & Rear)	Shut-In PSI Initial	Shut-in Duration	Shut-In PSI Final	Wiper Pig Run	Scraper Pig
1/16" Pinhole in Body Test IV	6 o'clock	11:1Flo-Seal-P with "Smaller" Bits	NA	1440	30 min	1440	Refer to Details / Results	NA
1/16" Pinhole in Body Test V	6 o'clock	7:1 Flo-Seal II with "Standard" Bits	NA	1440	15 min	1440	Refer to Details / Results	NA
1/16" Pinhole in Body Test VI	6 o'clock	7:1 Flo-Seal II with "Smaller" Bits	NA	NA	NA	NA	NA	NA
1/16" Pinhole in Body Test VII	6 o'clock	Gelled Gly-Flo with "Smaller" Bits	NA	1440	15 min	1440	Refer to Details / Results	NA
1/16" Pinhole in Body Test VIII	6 o'clock	Gelled Gly-Flo with "Smaller" bits	NA	1440	21 Hours	1000	Refer to Details / Results	NA

Table 15: High Pressure Testing Summary (continued)

g Run Details / Results

Filled test fixture with sealant. With nitrogen increased pressure to 1440 psi. Pressure kept falling. Kept pressuring with nitrogen until seal formed at 300 psi. Held for 30 minutes. Increased pressure to 1440 psi. Shut-in 30 min. Pressure remained at 1440 psi. Observed mound of flakes and angel hair at leak site. After scraping mound away in layers observed no particulate in pinhole. Pinhole clean.

Filled test fixture with sealant. With nitrogen increased pressure to 1440 psi. Held for 15 minutes. Removed mound of sealant by hand. Re-pressure test failed immediately.

Filled test fixture with sealant. Could not establish a seal.

Filled test fixture with sealant. Increased pressure to 1440 psi. Held for 15 minutes. Removed mound of sealant by hand. Re-pressure test failed immediately.

Filled test fixture with sealant. Increased pressure to 1475 psi. Shut-in for 21 hours. Re-pressure assembly to 1440 psi. Pressure held. Removed mound of sealant by hand. No bits remained in pinhole, only some activated sealant. Re-pressure test leaked immediately.

Test I proved that the sealant formulation used during the <u>Full Scale Pipeline Testing</u> was adequate for sealing 1.59 mm (1/16") pinhole leaks with a seal that could maintain integrity after pigging if higher sealing pressures were possible. The higher pressure of 34.47 MPa (5,000 psi) would force a bit or particulate that, under pressure, would expand and form a platform for the sealant to bridge across.

During this last stage of testing we were unsuccessful in finding the right combination of bit size and sealant formula to achieve the same results with pipeline pressures of 9.93 MPa (1440 psi).

We believe that development of such a combination is possible but beyond the scope of this project.

CONCLUSION

The leaks rates, though large, were not beyond our capabilities. We successfully sealed every defect to 9.93 MPa (1440 psi) both on the full scale pipeline test model between two pigs and in the lab utilizing test fixtures of different configurations. The difficulty was achieving a seal that could resists the effects of wiper and scraping pigs for all defects.

By testing the defects in a controlled environment, at different sealant curing times, it was shown that curing time had no effect on improving seal quality at pipeline pressures; the sealant was still not penetrating the defect properly prior to activation. The testing needed to split into two directions: one, for the weld defect that exhibits a very low leak rate and another for the pinhole based defects where larger leak rates were exhibited.

With the weld defect a lesser aggressive sealant formulation was developed, Gly-Flo "G", which yielded better results than the earlier 9:1 Flo-Seal-P that was utilized on the full scale pipeline testing.

On the pinhole based leaks our most aggressive non-particulate sealant, Flex-Plug, could not maintain a seal to 9.93 MPa (1440 psi) on a 1.59 mm (1/16") pinhole, except on an Internal Corrosion defect. Flex-Plug was able to successfully seal 1.19 mm (3/64"), 0.79mm (1/32") and 0.40 mm (1/64") pinholes to 9.93 MPa (1440 psi).

From this point we tried different sealant formulations of non-particulate and non-particulate / particulate combinations with little success.

Then, going back to our past experience in sealing leaks we tested our original formulation of 7:1 Flo-Seal-P at a higher pressure of 34.47 MPa (5,000 psi) with outstanding results. From this data we knew that to accomplish the same results we needed to inject a pressure expandable particle into the defect hole at pipeline pressures to act as a platform for the sealant to bridge across. To date, we have not been successful in modifying sealant formulas and particulate sizing to achieve the same results at typical pipeline pressures of 9.93 MPa (1440 psi) or less.

In summary, pressure activated sealant technology can be considered a viable option for pipeline leak repairs under the right circumstances of pressure, leak type and pigging requirements.

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LIST OF ACRONYMS AND ABBREVIATIONS

Note: SI is an abbreviation for Le Systeme International d'Unites."

°C	Degrees Celsius
cm	Centimeters
DFW	Defective Fabrication Weld
DGW	Defective Girth Weld
DPS	Defective Pipe Seam
EC	External Corrosion
°F	Degrees Fahrenheit
ft	Foot
gal	Gallon
gpm	Gallons per Minute
ĪĊ	Internal Corrosion
ID	Internal Diameter
in	Inch
°K	Degrees Kelvin
kg/m ³	Kilograms per Cubic Meter
L, 1	Liter
lbm/ft ³	Pounds per Cubic Foot
lbs	Pounds
l/min	Liters per Minute
М	Meter
MAOP	Maximum Allowable Operating Pressure
min	Minute
mm	Millimeter
MPa	Megapascal
ml/min	Millimeter per Minute
OD	Outside Diameter
ΔP	Pressure Differential
P_1, P_2	Initial Pressure and Final Pressure
Psi	Pounds per Square Inch
scf/min	Standard Cubic Feet per Minute
scm/min	Standard Cubic Meter per Minute
V_1, V_2	Initial Volume and Final Volume
Z_1, Z_2	Initial and Final Compressibility Coefficient