ALPINE POLYTECH

POLYMER TECHNOLOGIES
FOR THE ENERGY INDUSTRY

TEST REPORT: APT-TR-PP151027-01 Rev B
NORSOK M710 – Rev 3, ISO 23936-1:2009
Sour Aging of PEEK

NORSOK M710 QUALIFIED

Qualification Statement: The LEHVOSS supplied LUVOCOM® EOG-100, based on PEEK, is Qualified to NORSOK M710 Rev 3 and to ISO 23936-1:2009 sour aromatic multi-phase fluid ageing for high 10 % H2S classification A.3.ii per test report APT-TR-PP151027-01 Rev B.

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Accredited to the ISO/IEC 17025:2005, Field of Accreditation- Mechanical, NORSOK M710, ISO 23936
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PJLA Testing
Accreditation #88166

Approved By: _____

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1 Abstract

Alpine PolyTech evaluated LEHVOSS supplied LUVOCOM EOG-100 unfilled PEEK in a 10% H2S multiphase mixture at 215°C, 225°C, and 240°C out to 42 days (1000 hours) at each temperature. Physical property changes in tensile and volume were measured. Visual observations were made.

2 NORSOK M710 Stated Objectives

Norsok M710 defines test procedures for the prediction of progressive degradation of thermoplastics exposed to fluids at elevated pressure and temperature over an extended period of time.

The objectives:

1) Assess any physical effects of the fluid on the thermoplastic and to thermally accelerate any chemical reaction between the fluid and the thermoplastic, causing tensile and related property levels to shift systematically towards a pre-defined limit of acceptability.
2) By running exposure tests with test fluids at three different elevated temperatures above the service temperature, three different times to reach the acceptance boundary will result, with the highest test temperature producing the shortest "time to failure". Plotting the log of failure times against the reciprocal of the test temperature should result in a linear trend, enabling an estimate of service life at the operating temperature. For accelerated testing, the upper test temperature should be limited to temperatures ensuring that only degradation processes relevant for the qualification range will occur.

3 Life Estimation Statements

1) The EOG-100 was sour aged and experienced 5% volume swell and slight tensile property changes as the result of liquid absorption in the first few days. LEHVOSS and Alpine attempted to thermally accelerate chemical reaction between the PEEK and H2S by testing to 240°C but no progressive degradation of tensile properties from thermally accelerated H2S exposure was observed.
2) The tensile property changes of EOG-100 did not reach the acceptance boundary (50% change) at any time and temperature combination. The changes also do not accurately trend towards the acceptance boundary but instead are discontinuous. Therefore, EOG-100 is stable in H2S within these test parameters and a "Service Life" estimation cannot be calculated using NORSOK M710 methodology.

4 Qualification Statement

The LEHVOSS supplied LUVOCOM EOG-100, based on PEEK, is Qualified to NORSOK M710 Rev 3 and to ISO 23936-1:2009 sour aromatic multi-phase fluid ageing for high 10 % H2S classification A.3.ii per test report APT-TR-PP151027-01 Rev B.

5 Test Plan Summary

Materials	PEEK EOG-100: Modified unfilled grade
Test Temp	215°C, 225°C, 240°C (419°F, 427°F, 464°F)
Initial Pressure	6 ± .5 MPa (870 psi ± 72)
Test Duration	42 days at each temperature
Property Change	Tensile Strength, Modulus, Strain at Break, Volume/SG
Test Solutions	60% by volume hydrocarbon (70% heptane / 20% cyclohexane / 10% toluene)
	10% by volume water
	30% by volume gas phase (10% H2S / 5% CO2 / 85% CH4)

6 Materials & Methods

6.1 Material

PEEK EOG-100, modified, unfilled, Injection molded, Alpine Stock # P36.1

Figure 1 – As-received PEEK Specimens.

6.2 Test Methods & Specimens

Tensile & Volume Duplication – 5 initial and 5 conditioned

- ASTM D638 – Type V tensile bars
- ASTM D792 - Mass, Density, Volume – use Type 5 bars

6.3 Terminology

- Tensile Strength – maximum stress
- Tensile Stress at Yield – stress at zero slope or 1% offset and can also be Tensile Strength
- Tensile Stress at Beak – stress at break point and can also be Tensile Strength
- Elongation at yield – use extensometer for all materials
- Elongation at break - extensometer for glass PEEK. (Does not neck and draw)
- Nominal Strain at break - use grip separation for neat PEEK. (Does neck and draw)
- Modulus (Young's) - use extensometer for neat and filled PEEK
- Modulus (Secant 1%) - use extensometer for neat and filled PEEK
- Gage Thickness & Width – measure before conditioning and use initial values for tensile

6.4 Fluid Media

Norsok M710 Rev 3	60%	Hydrocarbon	70% heptane, 20% cyclohexane, 10% toluene
	10%	Water	distilled
	30%	Gas cap	10% H2S, 5% CO2, 85% CH4

6.5 Acceptance Criteria

This project uses the criteria from NORSOK M710 Rev 3 shown below.

- Volume: + 5 % /- 1 %
- Tensile: +/- 50 % (tensile strength, strain at break, modulus (D638 only))
- Visual inspection: No dissolution, cracking, blistering, or physical deformation permitted

Tensile test results shall be used to extrapolate the service life if applicable

6.6 Test Temperature

M710 says to run three temperatures above operating temperature. Alpine started with 215°C to identify property change trends. No degradation was observed so subsequent temperatures were increased to 225°C and then to 240°C.

6.7 Test Pressure

Pressurize the test vessel to 6 ± .5 MPa (870 psi ±72) at room temp with the test gases and then heat to the required temperature. To do this, a 10% H2S gas mixture was created based on partial pressures as shown Figure 2 and then transferred to the test vessel. The vessel was heated, vapor pressure increased and then stabilized and an example as shown in Figure 3. The Figure 2 and Figure 3 examples are from the final 22 days of soak at 225°C. This is from day 20 to day 42.

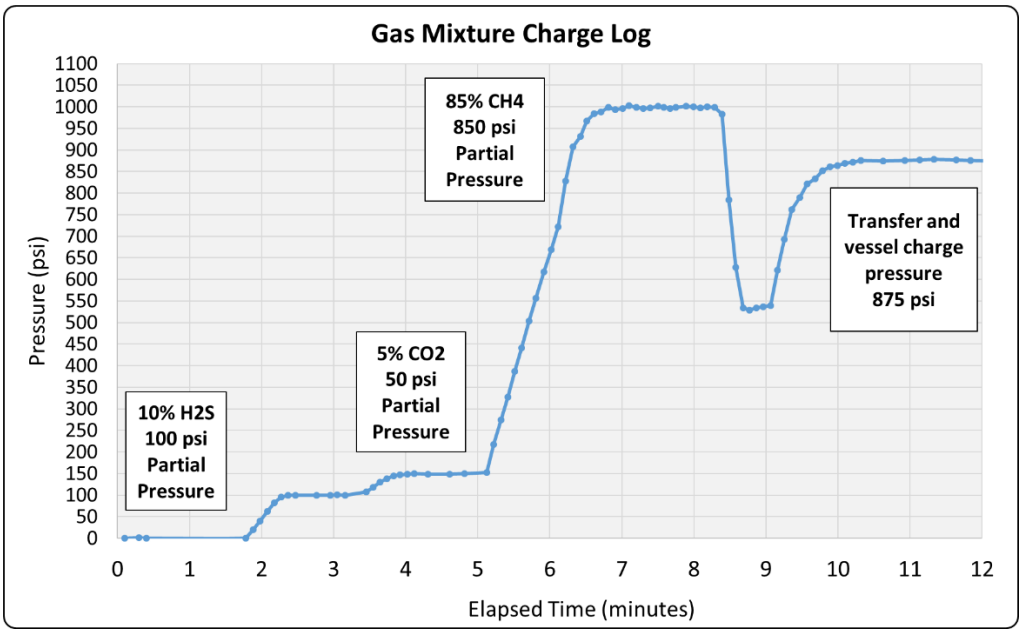

Figure 2 – 10% H2S gas mixture pressure chart for the final 22 days at 225°C

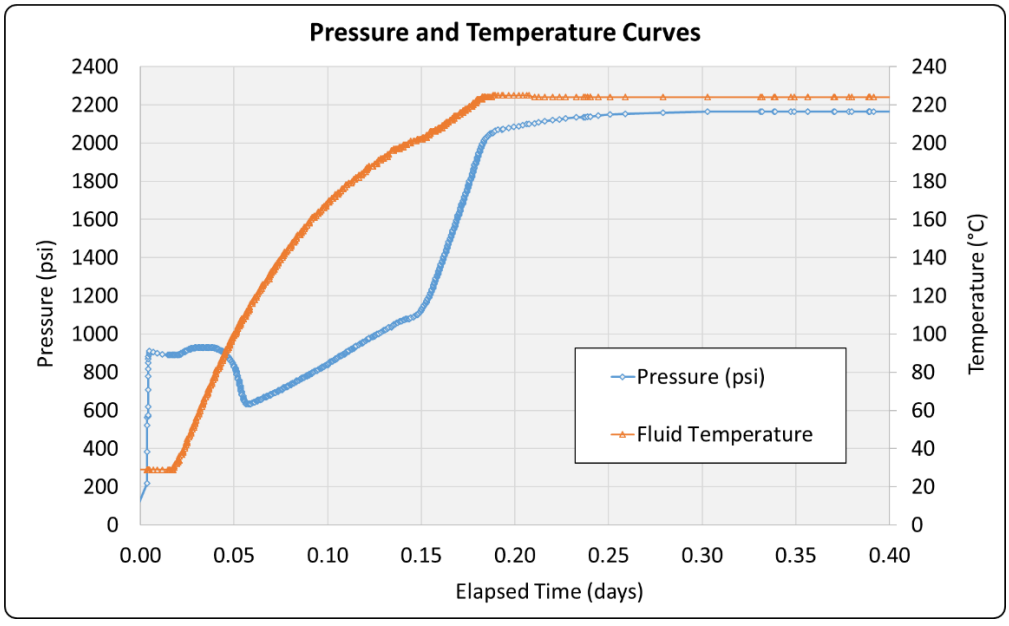

Figure 3 – Heat up log for the final 22 days at 225°C

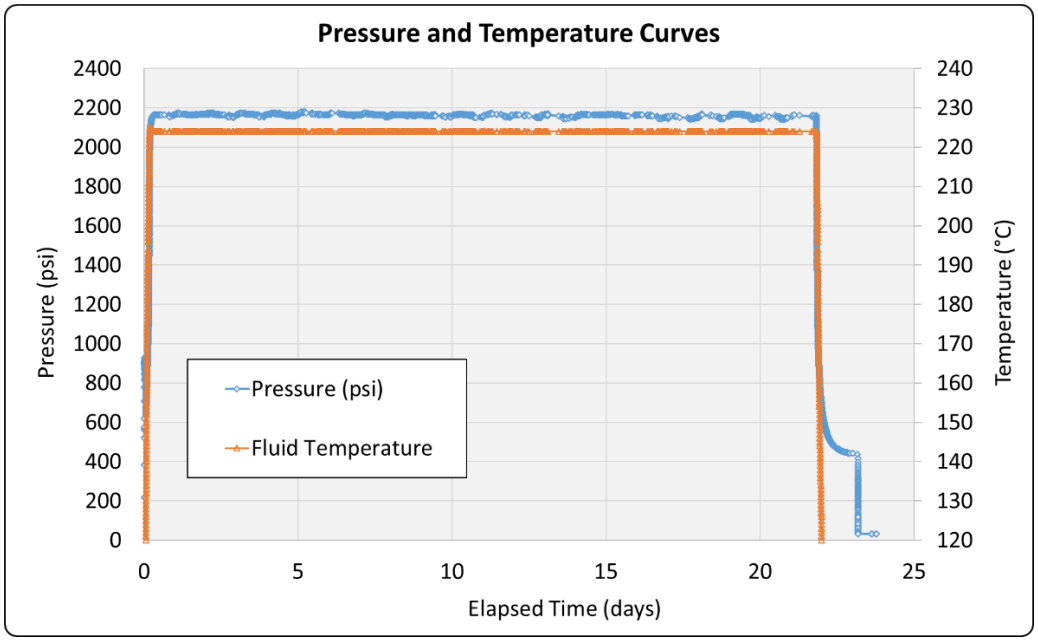

Figure 4 – Pressure and temperature data for the final 22 days at 225°C

6.8 Test Durations

Norsok M710 Rev 3 - "Several weeks may be required." The initial test durations were extended to 42 days for each temperature in an attempt to measure degradation. Prior to each H2S soak study, a 2 day saturation study without H2S present was run.

Temperaure	Saturation Duration (days)	H2S Exposure Durations (days)
215 C	2	2, 5, 10, 20, 42
225 C	2	5, 10, 20, 42
240 C	2	2, 5, 12, 20, 42

6.9 Service Life Estimate

From Norsok M710 Rev 3, Section A.4.15

"Tensile properties and mass/volume change at each test temperature shall be presented graphically as plots versus immersion time. If applicable, a graphical presentation according to Arrhenius method [logarithmic time against 1/T (absolute temperature (K))] based on trends for tensile properties shall be made. A best-fit line should be drawn to permit interpolation or extrapolation to service temperatures and the equation stated.

"ISO 23936-2 Annex D and E provide more details."

7 Test Results

The physical property data and % changes from this test program with units of measure is supplied below. No percent changes in properties reached the acceptance criteria that is used for Arrhenius life estimations. Percent changes are based on the as-received properties and changes in the median measured values.

- Tensile Strength (Maximum stress recorded) – The 2 day saturations have no impact and there were no significant changes in tensile strength over 42 days.

- Nominal Strain at Break (based on grip separation per ASTM D638) – The data hints at a long term decrease but the changes do not approach the 50% change threshold. The 42 day data at 240°C shows a plateauing of property changes so that the changes are insignificant. The extrapolations to a 50% change are discontinuous because the 215°C data trends towards an increase but the 225°C trends towards a decrease.

- Modulus (Secant 1%) – Has an initial increase of 12% from the saturation but then does not change for the remainder of the 42 day test. All changes are insignificant.

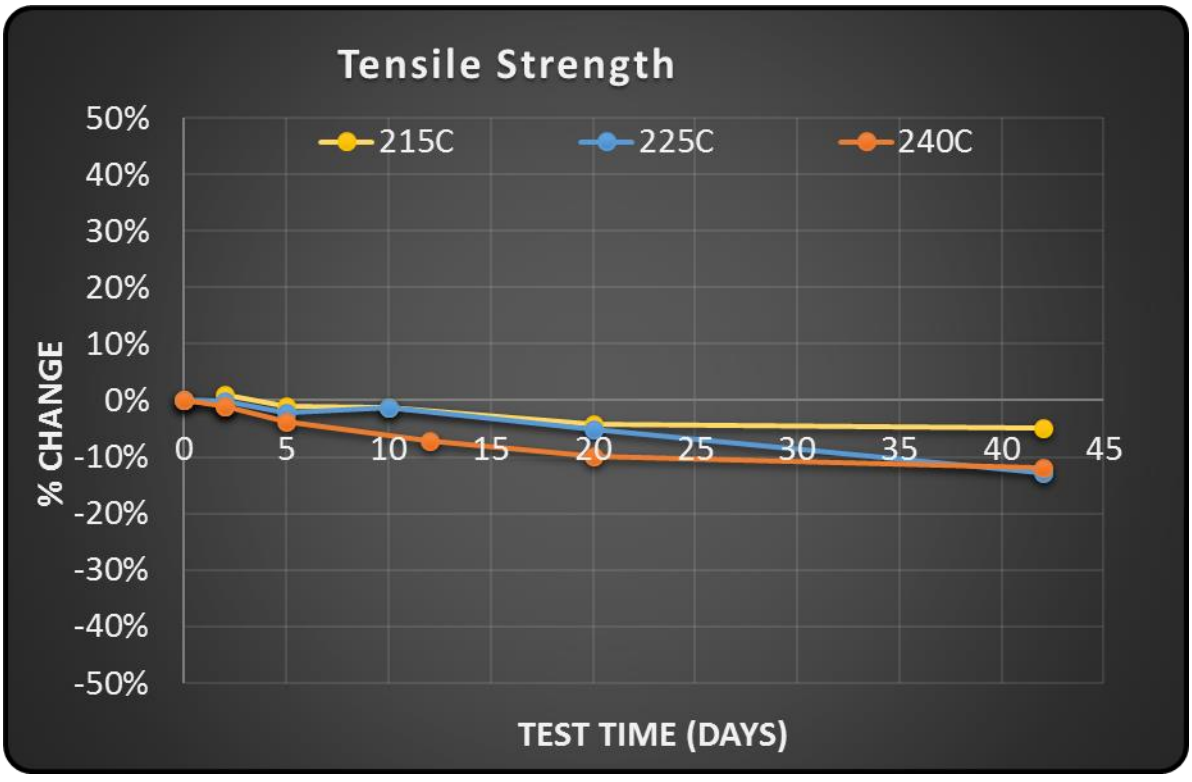

Figure 5 – Tensile Strength changes over 42 days

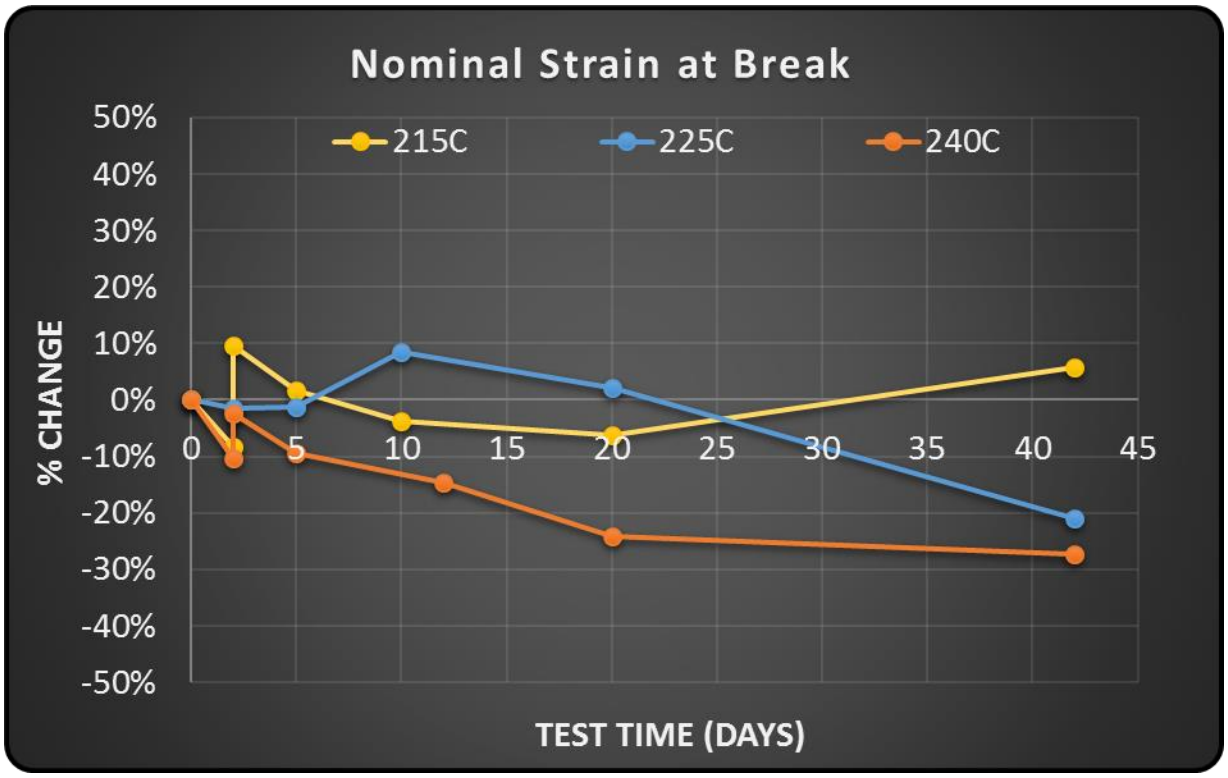

Figure 6 – Nominal strain at break changes over 42 days

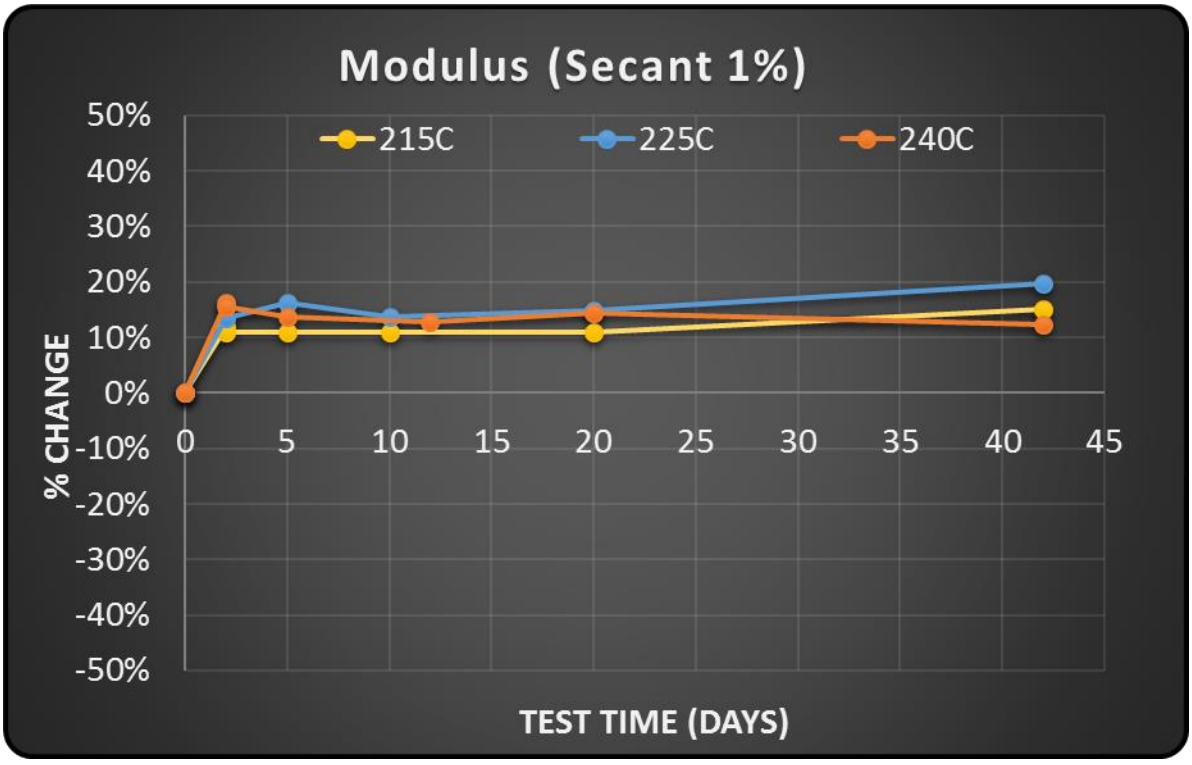

Figure 7 – Modulus changes over 42 days

Material Data	Days	Density	Tensile Strength	Elong at Break	Nominal Strain at Break	Modulus (Young's)	Modulus (Secant 1 %)
		g/ml	(psi)	%	%	(ksi)	(ksi)
m710	0	1.295	15765	90.3	50.4	539	545
215C	2	1.282	15631	76.6	46.2	594	609
Virgin	2	1.283	15933	80.6	55.3	594	605
D	5	1.283	15591	85.1	51.3	591	605
	10	1.281	15558	81.2	48.5	597	604
	20	1.281	15109	79.9	47.2	587	605
	42	1.275	15007	88.3	53.3	623	627
m710	0	1.292	15765	90.3	50.4	539	545
225C	2	1.278	15762	85.9	49.7	609	618
Virgin	5	1.280	15410	80.7	49.8	605	634
D	10	1.278	15583	86.7	54.7	621	620
	20	1.275	14953	86.1	51.5	621	626
	42	1.272	13732	67.6	39.8	650	652
m710	0	1.293	15765	90.3	50.4	539	545
240C	2	1.280	15614	80.9	48.2	616	634
Virgin	2	1.278	15603	88.1	54.3	613	630
D	5	1.277	15166	81.7	49.6	617	619
	12	1.274	14654	77.1	46.0	608	614
	20	1.274	14198	68.4	40.1	618	623
	42	1.268	13881	65.6	42.4	602	612

Material Data	Days	Mass	Density	Volume	Tensile Strength	Elong at Break	Nominal Strain at Break	Modulus (Young's)	Modulus (Secant 1 %)
		% Δ	% Δ	% Δ	% Δ	% Δ	% Δ	% Δ	% Δ
m710	0	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
215C	2	4.4%	-1.0%	5.5%	-0.8%	-15.2%	-8.3%	10.2%	11.7%
Virgin	2	3.7%	-0.9%	4.6%	1.1%	-10.7%	9.7%	10.2%	11.0%
D	5	3.7%	-1.0%	4.7%	-1.1%	-5.8%	1.8%	9.6%	11.0%
	10	3.7%	-1.1%	4.7%	-1.3%	-10.1%	-3.8%	10.8%	10.8%
	20	3.5%	-1.1%	4.7%	-4.2%	-11.5%	-6.3%	8.9%	11.0%
	42	3.2%	-1.6%	4.9%	-4.8%	-2.2%	5.8%	15.6%	15.0%
m710	0	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
225C	2	4.0%	-1.1%	5.2%	0.0%	-4.9%	-1.4%	13.0%	13.4%
Virgin	5	3.8%	-0.9%	4.7%	-2.3%	-10.6%	-1.2%	12.2%	16.3%
	10	3.8%	-1.1%	4.8%	-1.2%	-4.0%	8.5%	15.2%	13.8%
	20	3.5%	-1.3%	4.9%	-5.2%	-4.7%	2.2%	15.2%	14.9%
	42	3.3%	-1.6%	5.0%	-12.9%	-25.1%	-21.0%	20.6%	19.6%
m710	0	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
240C	2	3.8%	-1.0%	4.8%	-1.0%	-10.4%	-4.4%	14.3%	16.3%
Virgin	2	4.2%	-1.2%	5.5%	-1.0%	-2.4%	7.7%	13.7%	15.6%
D	5	4.2%	-1.2%	5.5%	-3.8%	-9.5%	-1.6%	14.5%	13.6%
	12	3.8%	-1.5%	5.4%	-7.0%	-14.6%	-8.7%	12.8%	12.7%
	20	3.9%	-1.5%	5.6%	-9.9%	-24.3%	-20.4%	14.7%	14.3%
	42	2.8%	-1.9%	4.8%	-12.0%	-27.4%	-15.9%	11.7%	12.3%

Figure 8 – Physical property data and changes after 7 day soak

8 Specimens Images

There were no visual indications of degradation, blisters, or cracks on the surface of any specimen after conditioning. Figure 9 below shows Type V PEEK specimens after 42 days at 240°C. One can see that the PEEK is visually unchanged.

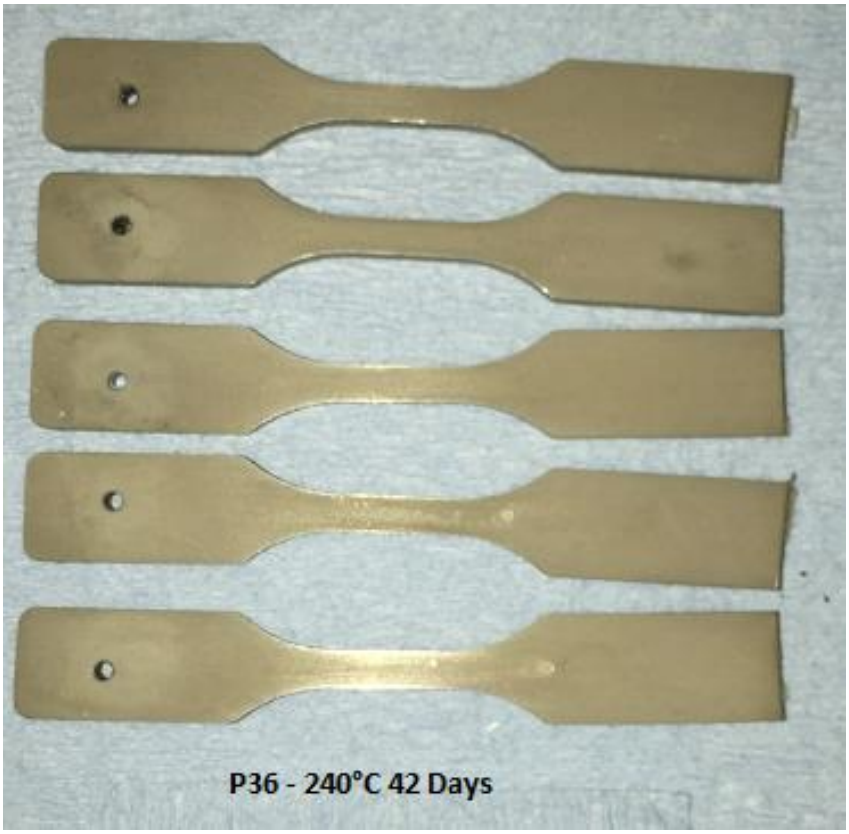

Figure 9 – PEEK materials after the 42 day 240°C H2S soak

9 Final Section

The results presented in this report relate only to the items tested.

Alpine PolyTech is accredited to the ISO/IEC 17025:2005 standard

Field of Accreditation-Mechanical, NORSOK M710, ISO 23936

PJLA Accreditation #88166