

## [HOT OIL SAMPLE STATION] SILANE 4.0 DISTILLATION

Customer: PTRHTF10093	System Information	Sample Information
REC GROUP 3322 ROAD N N.E. MOSES LAKE, WA 98837 USA Attn: Sam Bright Tel: (509)766-8902 E-Mail: sam.bright@recsilicon.com	System Volume: 50000 gal Bulk Operating Temp: 420F / 216C Heating Source: Blanket: Fluid: PETRO CANADA CALFLO AF Make: COEN	Lab No: 02287112 Analyst: Ron LeBlanc Sample Date: 05/14/19 Received Date: 05/24/19 Completed: 05/27/19

Recommendation: Re-Sample in 1 months. Pentane Insolubles increased which might indicate a poorly drawn sample. (GCD) 90% distillation might be due to poorly drawn sample. Silicon at 8 ppm is double over last sample. Look closely at next sample to see if silicon rises again which could indicate a process leak.

Comments: (GCD) 90% Distillation Point is marginally low.

Sample Date	Received Date	Fluid Age	Sample Location	Flash Point (COC)	Water (KF)	Viscosity (40°C)	Acid Number	Solids	GCD 10%	GCD 50%	GCD 90%	GCD % < 335°C
	mm/dd/yy			°F/°C	ppm	cSt	mg/KOH/g	%wt	°F/°C	°F/°C	°F/°C	%
05/14/19	05/24/19	28m	TCS INLET VENT	396 / 202	5.7	30.7	0.065	0.175	690 / 366	781 / 416	869 / 465	0.00
01/16/19	01/25/19	28m	TCS INLET VENT	394 / 201	43.9	31.0	0.06	0.072	669 / 354	771 / 411	870 / 465	3.46
03/22/18	03/29/18	18m		399 / 204	9.5	31.1	0.10	0.069	700 / 371	791 / 422	895 / 479	0.00
02/16/18	02/22/18	17m		399 / 204	8.7	30.8	0.12	0.046	691 / 366	794 / 424	889 / 476	2.19
02/08/18	02/15/18	17m		421 / 216	2.6	30.7	0.034	0.049	728 / 387	800 / 427	888 / 476	0.00
01/30/18	02/07/18	16m		414 / 212	5.3	31.0	0.044	0.074	692 / 367	792 / 422	888 / 476	1.57
05/31/17	06/05/17	8m	DISTO HOT OIL	406 / 208	17.0	30.9	0.060	0.052	692 / 367	797 / 425	897 / 481	1.60
09/14/16	09/19/16	58m	HOT OIL SMPL STATION	424 / 218	184.2	31.4	0.12	0.713	694 / 368	795 / 424	898 / 481	1.30
07/28/16	08/02/16	58m	PRV VENT NR TCS VAP	288 / 142	11.3	19.3	0.05	0.051	609 / 321	788 / 420	893 / 479	10.84
07/22/16	07/26/16	58m	H/O DCS REBOILER S	288 / 142	5.8	20.1	0.08	0.068	646 / 341	786 / 419	891 / 477	8.75
07/19/16	07/21/16	58m	PRV VENT NEAR TCS	237 / 114	43.3	19.8	0.13	0.508	552 / 289	777 / 414	878 / 470	13.13
07/15/16	07/21/16	58m	DISTO HOT OIL RESULT	252 / 122	25.4	17.6	0.09	0.190	548 / 287	790 / 421	901 / 483	13.01
07/08/16	07/14/16	49m	PRV VENT NR TCS VAP	270 / 132	0.00	24.0	0.08	0.115	645 / 340	787 / 420	889 / 476	8.79
07/07/16	07/12/16	49m	OIL LOOP DCS REBOILR	198 / 92	96.4	18.0	0.05	0.066	640 / 338	793 / 423	936 / 502	9.21
01/18/16	01/25/16	49m	PRV VENT NEAR TCS VP	392 / 200	30.2	28.0	0.16	0.136	684 / 362	796 / 425	898 / 481	4.13
11/21/15	11/27/15	0m		408 / 209	195.8	21.5	0.10	0.097	671 / 355	789 / 420	891 / 477	5.41
07/22/15	07/29/15	41m	PRV VENT NEAR TCS VP	367 / 186	0.00	28.9	0.041	0.101	682 / 361	798 / 426	895 / 479	4.55
04/13/15	04/21/15	41m	PRV VENT NEAR TCS VP	378 / 192	14.0	28.2	0.04	0.055	672 / 356	783 / 417	869 / 465	4.95
01/20/15	01/26/15	37m	PRVVENT NEAR TCSVAP	363 / 184	11.4	28.2	0.029	0.042	687 / 364	775 / 413	868 / 465	0.52
01/06/15	01/07/15	37m	PRV VENT NEAR TCS VA	378 / 192	7.3	28.4	0.12	0.356	654 / 346	773 / 412	963 / 517	6.92
11/13/14	11/25/14	34m	PRV VENTNEARTCSVAP	385 / 196	12.0	28.3	0.04	0.082	674 / 356	791 / 422	904 / 484	5.31
07/03/14	07/16/14	31m	PRV VENT	388 / 198	7.3	28.9	0.04	0.131	669 / 354	785 / 419	896 / 480	5.69
04/12/14	04/22/14	22m	PRV VENT	385 / 196	35.1	28.6	0.029	0.051	681 / 361	798 / 426	895 / 479	4.49
03/25/14	04/02/14	27m	PRV VENT NEAR TCS VP	396 / 202	17.0	28.5	0.02	0.054	683 / 362	798 / 425	896 / 480	4.29







PRV VENT NEAR TCS





























PRV VENT NEAR TCS VA



























PRV VENT NEAR TCS VP



























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PRV VENT NEAR TCS























































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PRV VENT NEAR TCS



























































































































































































































































































































































































REBOILER















































































































































































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N2 PURGE @100C 5DAYS





























CARBON FILTERED























































































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TCS VAPORIZER OUTLET









































































































































































































































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PUMP DISCHARGE





























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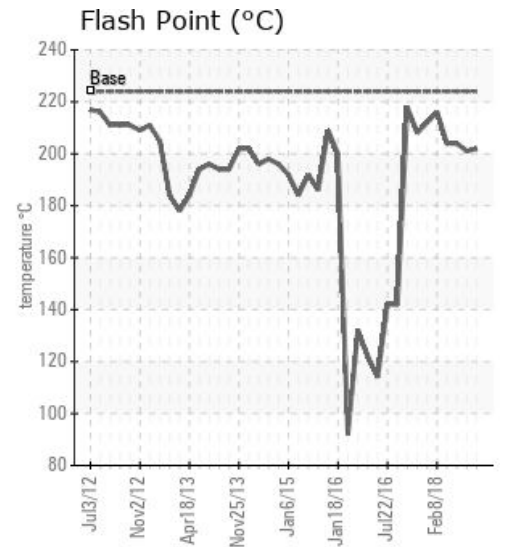
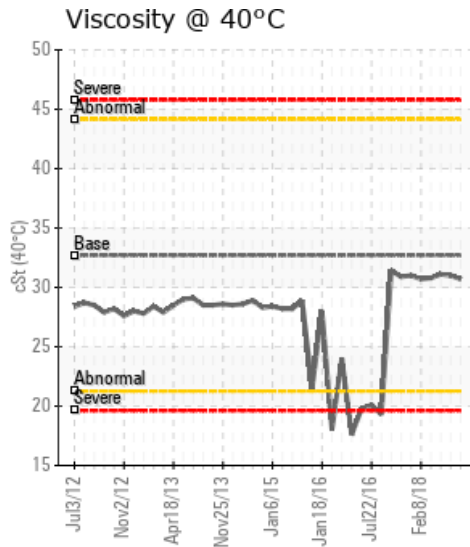
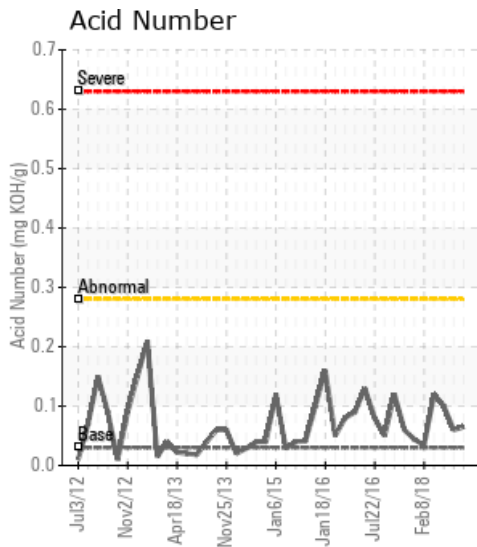
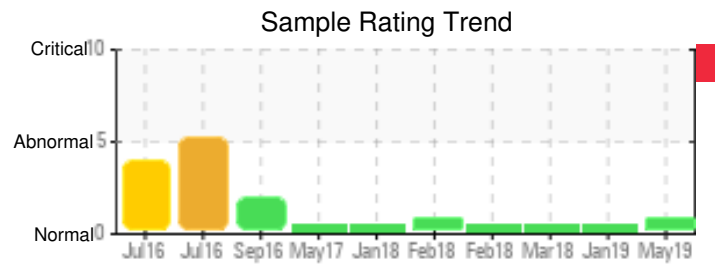
















































































































































































































































































































































































































































































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Elemental analysis results (above) in parts per million (ppm). [10,000 ppm = 1.0%]

Historical Comments	
01/16/19	Sample appears normal. Re-sample at normal interval.
03/22/18	Sample looks normal. Pentane insoluble increased slightly from previous sample.
02/16/18	Sample appears close to normal. The silicon stayed relatively the same as the last 3 samples. The (GCD) 10% distillation point dropped to normal this sample.
02/08/18	Pentane insoluble have dropped over the last 3 samples. The viscosity has dropped slightly over the last 3 samples as well. The (GCD) 10% distillation point is elevated approximately 20 degrees over last 2 samples. (GCD) 10% Distillation Point is marginally high.
01/30/18	Pentane insoluble dropped significantly in this sample. Debris dropped from moderate to very lite.
05/31/17	Sample appears normal. Resample in 3 months.
09/14/16	Pentane is elevated indicating sediment. Filter cart should be run on oil to clean it up. Pentane Insolubles levels are severely high.

07/28/16



07/19/16

COC Flash Point is severely low. Visc @ 40°C is abnormally low. (GCD) % < 335°C is marginally high. COC Flash Point is severely low. Visc @ 40°C is abnormally low. (GCD) % < 335°C is marginally high.

07/15/16

(GCD) 10% Distillation Point is severely low. COC Flash Point is severely low. (GCD) % < 335°C is abnormally high. Visc @ 40°C is abnormally low. Compared to the previous sample the viscosity improved slightly, the COC flash point lowered slightly, the (GCD)10% is approximately 55C lower than normal. Pentane appears to be increasing significantly over the last 4 samples. Decision needs to be made if the fluid can continue to be run with the low viscosity and low COC flash point. Pentane Insolubles levels are abnormally high. (GCD) 10% Distillation Point is severely low. COC Flash Point is severely low. (GCD) % < 335°C is abnormally high. Visc @ 40°C is abnormally low.

07/08/16

(GCD) 10% Distillation Point is severely low. COC Flash Point is severely low. Visc @ 40°C is severely low. (GCD) % < 335°C is abnormally high. Pentane appears to be increasing significantly over the last 3 samples. Decision needs to be made if the fluid can continue to be run with the low viscosity and low COC flash point. (GCD) 10% Distillation Point is severely low. COC Flash Point is severely low. Visc @ 40°C is severely low. (GCD) % < 335°C is abnormally high.

07/07/16

Viscosity has improved significantly compared to last sample. Flash point has also raised to 132 which is a significant improvement over previous sample. (GCD) 90% distillation point has returned to specifications. Resample in 1 week to monitor condition. Flash point remains low. As oil is circulated it might improve. Adding new oil to the system could improve Flash Point as well. COC Flash Point is severely low. (GCD) % < 335°C is marginally high.



01/18/16

The oil viscosity has dropped significantly (35%) on this last sample. The drop of flash point to ~200F (from ~400F) and steep increase in low boilers could be 2 things. The oil was contaminated with a lighter material or it was thermally degraded. The rapid degradation due to the fire may cause such a rapid degradation of the fluid. If the entire charge of the system fluid looks like this sample it brings the difficult decision that the fluid should probably be replaced or do a significant sweetening to restore the flash point and other properties. (GCD) 90% Distillation Point is severely high. COC Flash Point is severely low. Visc @ 40°C is severely low. (GCD) % < 335°C is marginally high.

11/21/15

Sample appears normal. Si has elevated compared to last sample but is still low compared to previous samples. Resample at normal interval.

07/22/15

\*\*\* NOTE: water result retested. Now normal \*\*\*Viscosity has decreased since last sample. Resample at normal interval.

04/13/15

Si & Na have increased since last sample. COC Flash Point is marginally low.

01/20/15

Sample appears normal. Sample again in 3 months. COC Flash Point is marginally low.

01/06/15

The overall condition of the oil has improved since the previous sample. The flash point has dropped slightly and is marginally low. The (GCD) 90% distillation point has improved slightly. The Si has decreased significantly. Sample in 1 month suggested. COC Flash Point is marginally low. (GCD) 90% Distillation Point is marginally low.

11/13/14

Si increased nearly 4 times since last sample (11/13/2014). A process leak is indicated. The (GCD) 90% Distillation Point is severely high. COC Flash Point is marginally low. Resample immediately to monitor oil condition. Silicon ppm levels are abnormally high. (GCD) 90% Distillation Point is severely high. COC Flash Point is marginally low.

07/03/14

Sample appears normal. Continue to sample at normal intervals.



04/12/14

Oil condition looks normal and consistent with previous reports. Pls try to sample every quarter at least to monitor silicon levels.

03/25/14

Silicon level remains unchanhed. All other properties remain normal.

11/25/13

Silicon is unchanged at ~9-10ppm. Other properties are normal. We're glad to hear regular sampling will resume on this system.

10/29/13

Silicon is stable at 8-10 ppm. Re-sample at next scheduled interval.

09/05/13

Silicon is stable at 10 ppm. Other properties are acceptable. Re-sample at next scheduled interval. COC Flash Point is marginally low.

08/12/13

The oil is in good condition and suitable for further use. Silicon has dropped slightly (within the repeatability of the test) to 8.6 ppm. Please continue to sample at the regular interval. COC Flash Point is marginally low.

06/25/13

Silicon remains steady at 9.3 ppm. All other properties remain the same. Please continue to sample at the regular interval.

04/18/13

Silicon has risen slightly from 9.2 to 10 ppm (decimal places are rounded in the report). All other properties remain the same. Please continue to sample at the regular interval. COC Flash Point is marginally low.



01/17/13

Sample appears relatively unchanged since Jan 2013. Silicon remains constant since Dec 2012 at 9 ppm. Please continue to sample at the regular interval. COC Flash Point is marginally low.

12/10/12

The sample looks identical to the previous one from a week ago. Keep re-sampling to accumulate data with the new lab and monitor the fluid condition COC Flash Point is abnormally low.

11/28/12

This is a retest of the sample from early January that Polaris did not analyze fully. Results are comparable. Silicon is a couple ppm higher but this could be due to the different lab testing it. Let's continue to monitor the system. COC Flash Point is marginally low.

11/12/12

Silicon is stable at 6-7 ppm. Other properties look normal.

11/02/12

There is a surge in TAN (acidity) but it is not accompanied with a rise in Silicon so we do not suspect a leak. Re-sample at next normal sampling interval. Happy Thanksgiving !

09/04/12

The oil condition remains good. Silicon is stable at the 6-7 ppm level.

08/15/12

Results are consistent with previous samples. Silicon is stable at 7 ppm.

07/30/12

The results are sort of in line with previous results. This may not be founded but the TAN has risen very slightly and the viscosity has very slowly decreased (keep in mind the TAN has an uncertainty level of +/- 45%). Silicon is stable at 6 ppm. Some venting of the low boilers will help restore the flash point and viscosity closer to fresh oil value.



07/23/12

The results are sort of in line with previous results. This may not be founded but the TAN has risen very slightly and the viscosity has very slowly decreased (keep in mind the TAN has an uncertainty level of +/- 45%). Silicon is stable at 6 ppm. Some venting of the low boilers will help restore the flash point and viscosity closer to fresh oil value.

07/03/12

Silicon is stable at 5-6 ppm level. Other properties virtually unchanged.

06/25/12

Results are consistent with previous samples. Silicon stable at ~5 ppm

06/14/12

Results are virtually unchanged from the last sample. Silicon is stable at 7 ppm. Please advise if anything abnormal is noticed on the operations side. The missing COC Flash Point is due to not having enough sample.

06/07/12

The results show no major change and Silicon is stable.

05/29/12

Baseline sample from June 2012. No significant difference between sample from June 2012 and April 2013. Silicon remains constant at 9 ppm. Please continue to sample at the regular interval. COC Flash Point is marginally low.

04/30/12

Virtually no change from the last sample. Re-sample at next regular interval

04/16/12

Fluid properties and silicon results look stable. Re-sample at next normal interval.



04/14/12

Silicon rose from 5 to 9 ppm. We would like to see further samples before concluding it's rising because of the test uncertainty at single digit ppms.

03/19/12

Silicon is stable around 5 ppm. Other properties look normal.

03/05/12

This is a temporary report because the COC flash point instrument is down. all properties are stable and Silicon remains at 5ppm level. Nothing to report based on these results.

02/20/12

Everything seems stable, Silicon is the same as previous samples. We are not certain why the flash point is suddenly higher but suspect uncertainty in testing. Samples are taken regularly so no reason to be concerned here.

02/06/12

The results are consistent with recent TCS Vaporizer samples. Silicon is unchanged at 5 ppm.

02/06/12

The oil condition didn't changed much since the last sample. The phosphorous dropped suddenly to 10 ppm from 270 ppm which is surprising. We will see from the specialized testing from Mississauga if indeed the additive contributing to the phosphorous has depleted.

01/09/12

The oil properties look normal and consistent with previous samples. silicon rose from 4 to 9 ppm but this may be due to the analysis, as this is very low level and uncertainty may be high at this level.

12/27/11

Results look very much like previous sample. Silicon is stable around 4-5ppm.



12/13/11

This sample show normal results and no sudden change in Silicon, TAN or solids. Let's keep monitoring the oil in this plant.

11/29/11

The previous sample had Silicon jump from 6 ppm to 42 ppm, taken from a location described as "Reboiler". This sample was taken from "TCH Columbia Reboiler RRV" is now back down to 6 ppm Silicon. Please verify with operating parameters to see if indeed a leak occurred or not.

11/22/11

It's unclear if this sample was taken from the same reboiler as the last sample but the Silicon jumped from 6 to 42 ppm, which is very significant in just 1 week. Please analyze the data gathered from your process and vent detection to assist in the diagnosis of a possible major leak.

11/16/11

Results look slightly different (better) in this Reboiler North vs the traditional expansion tank or vaporizer samples. Silicon remains at 6 ppm and consistent with recent samples.

11/13/11

Silicon remains very low (~6ppm) which is a good thing. All other properties remain constant from the last samples after the cleaning and flushing.

11/09/11

This sample show normal results and no sudden change in Silicon, TAN or solids. Let's keep monitoring the oil in this plant.

11/01/11

Silicon remains very low (~6ppm) which is a good thing. All other properties remain constant from the last samples after the cleaning and flushing.

10/18/11

Results look identical to 2 weeks ago. No rise in silicon. Let's keep monitoring the fluid.



10/02/11

The properties look very much like Calflo AF. There is a trace amount of diesel present left from the cleaning as seen in the 4.3% low boilers, but overall this baseline sample looks good. Let's see what the R&D analysis shows. Silicon has dropped down to 8 ppm.











08/15/11

















































03/12/11

The results look like the last few samples whereas the TAN stabilized around 0.15 and the Silicon lingers at ~300 ppm. We see traces of Barium which is unseen before but we will wait to see if it's the beginning of a trend or a one-off result before commenting further. Re-sample at next normal interval.

03/10/11

same comments as previous sample. Encouraging results with TAN dropping which means the hydrochloric acid is not posing as much of a threat anymore.

02/25/11

The silicon is still present (~300 ppm) however the acidity of the oil has dropped dramatically. The TAN dropped from nearly 1.0 down to 0.15. Not certain if it's safe to say the silicon that is there will remain but at least there doesn't seem to be formation of hydrochloric acid anymore. Let's keep monitoring the situation.

02/08/11

The encouraging news is that the TAN dropped from 0.95 to 0.15 mg KOH/g which might mean that the acidic form of contamination (hydrochloric acid ?) was greatly removed. The silicon remains high at 327 ppm, still lower than the 400 ppm at the peak of the contamination but with the low TAN it appears the form of silicon lingering around may not have been removed by filtration. Keep up the good work.



02/07/11

Silicone decreased again on this sample so a drop from 398 ppm a couple weeks ago to 328 now. Hopefully this is a representation of the entire system and let's watch carefully in coming weeks now that the reboilers have been cleaned down to bare metal.

01/24/11

There is a decline in Silicon for the first time, let's see if it's sustainable, after teh reboilers have been cleaned with caustic.



01/14/11

TAN is now above 1 and silicone at 334 ppm.



01/04/11

TAN and silicone rising

12/17/10

Monitor Silicon and TAN levels

12/17/10

Silicone seem to have stabilized around 300 ppm, little lower than a couple months ago. The tin may come from the pump bearings attacked by the acids.





11/30/10

Not sure what actions were taken since the last sample. Silicon is at 323 ppm while the Total Acid Number is 0.63.



11/19/10

As shared before, the TAN is lower in the sample that was purged with nitrogen to release the chlorosilane. However, the carbon filtered sample shows lower silicone.







10/15/10

Compared to other TCS Vaporizer samples the Silicon is still rising and so is the Acid Number. We are starting to suspect the Acid Number is not rising because of the fluid oxidizing but rather because of the hydrochloric acid formation because the viscosity and distillation properties still look like fresh oil despite the very high AN.



10/15/10

The tin dropped all of a sudden. Silicon is still high and so is Acid Number.

10/04/10

Same comments as other sample

10/04/10

Silicon is still rising to unprecedented levels and now we see a clear trend of increasing acid number.

10/02/10

Silicon is still rising at unprecedented levels and now we see a clear trend of increasing acid number.

10/01/10

Refer to comments on the next sample

10/01/10

Silicon is still rising in the Vaporizer

09/02/10

The silicon is still rising but now we see Tin shooting up.

09/02/10

The results from the Vaporizer look very much alike the ones from this bottom of the tank sample. Rising Acid Number, rising Silicone to levels not experienced before.



08/19/10

The Silicon is still rising and so is the Acid Number (acidity)

08/19/10

The silicon is rising for the "bottom of tank" samples, now at 88 ppm.

08/13/10

Just like the bottom of tank samples, these Vaporizer samples see a rise in Silicon.

08/13/10

The GCD instrument is down so full report to follow later. Results are consistent with past samples with Silicon hovering around 50 ppm while Chlorine test and GCD results to follow soon.

08/04/10

Results released without the GCD data to expedite. Chlorine testing is also being done. Results are consistent with previous samples with Silicon around 50 ppm and Acid Number around 0.13.

08/04/10

Results are confusing. Silicon dropped slightly but chlorine is higher. Next sample taken today will help monitor trend

07/22/10

Follow up sample will help monitor

07/22/10

The GCD instrument is down so full report to follow later. Silicon has risen slowly but chlorine is lower than the bottom of tank sample



07/15/10

This bottom of tank sample does not look as bad as the previous bottom of tank sample from 7/15. There was not enough sample to run the GCD.

07/15/10

The oil looks like new Calflo AF.

07/15/10

We will ask for chlorine testing on the tank bottom sample. Tank bottom appears to have more solids (oil insoluble material), more water but not more acidic

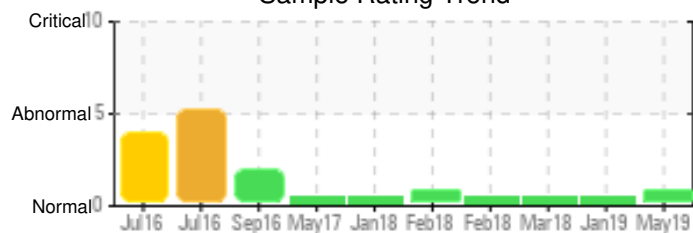
07/12/10

Wait for next sample for comparison

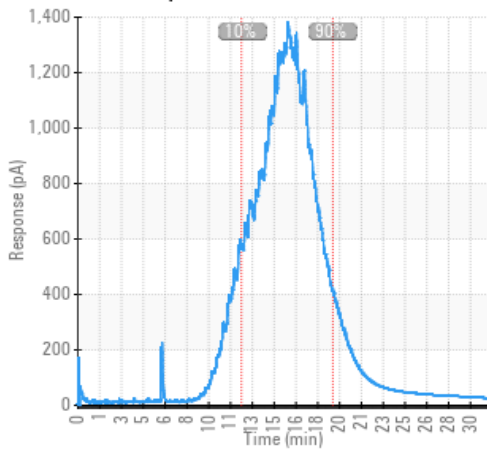
01/15/10

A process leak and contamination from left-over water from the initial pressure oil in such a short time, which may indicate the presence of acids (hydrochloric < 50 ppm. Water content is 108 ppm which is only about twice what fresh oil n

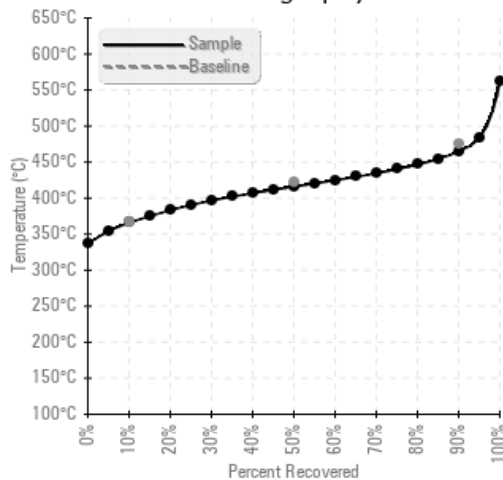
### Sample Rating Trend



### GCD Spectrum



### Gas Chromatography Distillation



### % Boiling < 335°C

