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BENCH-SCALE CO-PROCESSING
Contract No. DE-AC22-87PC79818

Technical Progress Report No. 21 (07/1/93-09/30/93)

by

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UOP's second co-processing contract, DE-AC22-87PC79818, began in April 1988. The major objective of this contract is to establish a database for the optimization of the co-processing concept by improving the effectiveness of the co-processing catalyst system. Two major mechanisms for improving the catalyst system are to be investigated: employment of more effective catalysts and utilization of improved catalytic environments. These two mechanisms are defined in the contract Statement of Work under Task 3.2 as Subtask 3.2.1 and 3.2.2, respectively.

This report covers the period of July 1, 1993 to September 30, 1993. During this period work on Subtask 3.2.2, Improvement in Catalytic Environment, was carried out and the bench-scale co-processing pilot plant was operated in the counter-current mode. An evaluation of the counter-current process in the presence and absence of a packing material under different operating conditions was completed. The project objective was to achieve 90+% conversion of the 510°C non-distillable at 2000 psig. However, due to plant limitations, this objective was not met. Because of the reactor size and throughput, the flow regime within the reactor is laminar. It is believed that without turbulent flow, there is insufficient mixing to keep the catalyst in solution. Once the catalyst is out of solution, it probably attaches to the reactor walls and a restriction forms which causes all the material entering the reactor to immediately exit through the reactor outlet. No additional experiments are planned with the counter-current co-processing mode and the pilot plant will be reconfigured to operate in the co-current mode.

BENCH-SCALE CO-PROCESSING PLANT

In the counter-current co-processing pilot plant the feed flows down the reactor (7/8" internal diameter, 72-3/4" total length, 20-1/2" reactor zone) and is contacted with up flow H_2. The catalyst is mixed with the feed and pumped to the reactor. The products flow upward with the

H_2 while the unreacted feed continues to flow down. The unreacted feed can be recycled back to the top of the reactor where it mixes with fresh feed. This will increase the catalyst concentration of the feed in the reactor and improve conversion. The reactor bottoms are recirculated to keep the bottoms material from setting up. In the reactor, the gas phase is continuous and the liquid phase is discontinuous.

The reactor products enter a high-pressure separator (HPS) where the gas is separated from the liquid at 110°C and 2000 psig. The liquid then flows to a low-pressure separator (LPS) where more light ends are stripped off at 135°C and 15 psig by an upflow of N_2 stream. The HPS off-gas flows into a 3-phase separator where it is contacted with water. The water removes any salts which are contained in the partially condensed HPS off-gas. The hydrocarbon phase from the 3-phase separator is sent to the LPS for further separation.

The 3-phase off-gas is recycled back into the plant. It is mixed with fresh H_2 and used as up flow H_2 in the reactor. Some of the off-gas is bled off to maintain a hydrogen purity of approximately 90%. The LPS bottoms product is collected in a drum while the overhead gas is sent to a debutanizer column. The debutanizer column bottoms are collected while the overhead gas is measured and vented. A schematic diagram of the plant flow scheme is shown in Figure 1.

EXPERIMENTAL

Feedstock

Lloydminster vacuum resid (R10, UOP 58-1625) was used as the reference for Pilot Plant 558 counter-current co-processing runs. For the set of runs discussed in this report, no coal was added to the feed. The vacuum resid was obtained from Husky Oil Lloydminster Refinery in May, 1988. The properties of the feedstock are presented in Appendix I. The feed has an API of 6.6 with 81 wt% of the feed boiling over 510°C. The resid contains 4.8 wt% sulfur, 0.6 wt% nitrogen, 13.6 wt% heptane insolubles and 17.4 wt% carbon residue.

Pilot Plant Testing

Pilot Plant 558 was used for this study (see Figure 1). In Runs 38 to 41, Lloydminster vacuum resid was processed using a catalyst concentration of 0.05 wt% Mo. Operating conditions for Runs 38, 39, 40, and 41 are listed in Tables 1, 2, 3, and 4, respectively. Plant pressure was constant in all the runs at 2000 psig. The fresh feed rate varied from 1.0 to 3.0 liquid hourly space velocity (LHSV) and reactor temperature ranged from 350°C to 420°C.

The objective of this project was to obtain 90%+ conversion of the liquid product with 0.05 wt% Mo-based catalyst in a counter-current system. Plant pressure was to be maintained at 2000 psig and reactor temperatures were not to exceed 420°C. H_2 flow rates, fresh feed flow rates and packing material were variable.

Operations

Run 38

Run 38 was started up at a fresh feed rate of 2.0 LHSV, reactor temperatures of 385°C, H_2 to feed rate of 7000 SCFB (7.10 SCFH) and a H_2 to reactor rate of 3000 SCFB (3.04 SCFH). The process conditions for the run can be seen in Table 1. The reactor was packed with 15" thin mesh Hastelloy packing located 9" below the feed inlet line. This positioned the packing completely in the middle heater zone of the reactor.

During the course of the run, the fresh feed rate (2 LHSV), H_2 rates (7.10 SCFH H_2 to feed and 3.04 SCFH H_2 to reactor), and reactor pressure (2000 psig) were kept constant. Only the reactor and preheater temperatures were varied. After 36 HOS (Period 3), the preheater and reactor temperatures were increased to 390°C. After 108 HOS, (Period 9), the reactor temperature was increased to 395°C while the preheater was maintained at 390°C. The reactor temperature was increased to 400°C at 180 HOS (Period 15) and the preheater temperature was increased to 395°C. The preheater plugged at 199 HOS and was unable to be cleared. Shutdown procedures began immediately.

The flow regime in the reactor system was in the laminar region. We believe that without turbulent flow, there is insufficient mixing to keep solids, whether in the form of catalysts and/or coal, dispersed in the liquid whereby they can be transported through the reactor without deposition.

Run 39

Run 39 was started up at a fresh feed rate of 2.0 LHSV, reactor temperature of 400°C, preheater temperature of 390°C, H_2 to feed rate of 7000 SCFB (6.73 SCFH) and H_2 to reactor rate of 3000 SCFB (3.04 SCFH). The reactor packing (thin mesh Hastelloy packing) was located in the middle heater zone, similar to Run 38. The purpose of the run was to test a new preheater on the plant. Process conditions for the entire run are listed in Table 2. Once again, the flow rates and plant pressure (2000 psig) were kept constant while the temperatures were varied.

The reactor temperature was increased to 410°C at 72 HOS (Period 6). A large ΔT was developing over the packing bed at 120 HOS (Period 10), so the reactor temperature was dropped to 405°C and the preheater temperature was increased to 400°C. To avoid coking in the preheater, the H_2 to feed rate was increased to 9000 SCFB (8.63 SCFH). The temperature differential was not eliminated so the preheater temperature was increased to 405°C at 132 HOS (Period 11). All material began going overhead and the plant was shutdown at 144 HOS.

TABLE 1
Run 38 Process Conditions

Period	H_2 Drag, cf/hr	Preheat Temp., °C	Reactor Temp., °C
1	0.5	385	385
2	0.5	385	385
3	0.5	390	390
4	0.5	390	390
5	0.5	390	390
6	0.5	390	390
7	0.5	390	390
8	0.5	390	390
9	0.5/0	390	395
10	0	390	395
11	0	390	395
12	0	390	395
13	0	390	395
14	0	390	395
15	0.5	395	400
16	0.5	395	400

Run 40

Run 40 was started at a fresh feed rate of 2.0 LHSV with no H_2 flow to either the feed or the reactor. The reactor and preheater temperatures were at 350°C to allow the feed to flow but to stop reactions from occurring. The reactor was packed with 15" of the thin mesh Hastelloy packing located in the middle heater zone. The run was designed to determine how much metals were held up in the reactor and packing. Process conditions for the entire run are summarized in Table 3. H_2 was cut into the plant at 24 HOS (Period 2) at 7000 SCFB (6.36 SCFH) to the feed and 3000 SCFB (2.72 SCFH) to the reactor. The H_2 rates were calculated based on 95% H_2 purity.

TABLE 2
Run 39 Process Conditions

Period	H_2 Drag, cf/hr	H_2 to Feed, SCFH	Preheat Temp., °C	Reactor Temp., °C
1	0	6.73	390	400
2	0	6.73	390	400
3	0	6.73	390	400
4	0	6.73	390	400
5	1.0	6.73	390	400
6	0	6.73	390	410
7	0	6.73	390	410
8	0	6.73	390	410
9	0	6.73	390	410
10	0	8.63	400	405
11	1.0	8.63	415	405

Reactor and preheater temperatures were increased to 360°C at 60 HOS (Period 5) and to 400°C at 84 HOS (Period 7). During Period 8 (96 HOS), H_2 rates were changed to allow the H_2 purity to drop to 85%. The H_2 to feed rate was changed to 3.04 SCFH (3000 SCFB) and the H_2 to reactor rate was 6.71 SCFH (7000 SCFB). Reactor and preheater temperatures were increased to 410°C at 108 HOS (Period 9). A temperature differential was observed across the packing bed so the preheater temperature was raised to 425°C at 156 HOS (Period 13).

The fresh feed rate was then dropped to 1.0 LHSV at 168 HOS (Period 14). The preheater temperature was dropped to 420°C at 192 HOS (Period 16) due to the lower feed rates. Material was passing through the reactor and not reacting so the H_2 to the reactor rate was increased to 4000 SCFB (4.059 SCFH) to increase the hydrogen availability in the reactor. Material was hanging up in the reactor at 240 HOS (Period 19) and was not able to be corrected. Shutdown began at 240 HOS.

Run 41

Prior to this run, the preheater section of the reactor was split to allow for separate temperature control of the preheater coil and the reactor outlet section.

At the start of the run, the fresh feed rate was 1.0 LHSV, the H_2 rate to the feed was 14,600 SCFB (6.36 SCFH), the H_2 rate to the reactor was 6250 SCFB (2.72 SCFH), the plant pressure was 2000 psig, the reactor temperature was 400°C, the preheater coil (top preheater) was 420°C and the reactor outlet (bottom preheater) was 410°C.

TABLE 3
Run 40 Process Conditions

Period	FF Rate, LHSV	H_2 Drag, cf/hr	H_2 to Rx, SCFH	H_2 to Feed, SCFH	Preheat Temp., °C	Reactor Temp., °C
1	2.0	0	0	0	350	350
2	2.0	1.0	2.72	6.36	350	350
3	2.0	0	2.72	6.36	350	350
4	2.0	0.3	2.72	6.36	350	350
5	2.0	0	2.72	6.36	360	360
6	2.0	0	2.72	6.36	360	360
7	2.0	0	3.04	6.75	400	400
8	2.0	0.1	3.04	6.75	400	400
9	2.0	0	3.04	6.75	410	410
10	2.0	0	3.04	6.75	410	410
11	2.0	0	3.04	6.75	410	410
12	2.0	0.5	3.04	6.75	410	410
13	1.0	0	3.04	6.75	425	410
14	1.0	0	3.04	6.75	425	410
15	1.0	0	3.04	6.75	425	410
16	1.0	0.5	3.04	6.75	420	410
17	1.0	0	3.04	6.75	420	410
18	1.0	0	1.52	6.75	420	410
19	1.0	0	1.52	6.75	420	410

The fresh feed and H_2 feed rates were hold constant throughout the run. The reactor contained no packing during this run. Once a bottoms level was established, the reactor bottoms was recycled and combined with the fresh feed prior to entering the reactor. The bottoms recycle was set to achieve a 5.0 combined feed ratio (CFR, defined as the ratio of [fresh feed + recycle] to fresh feed). This CFR was held constant during the run. The reactor bottoms looked like the feed material. The run was designed to determine the metals hold up in an empty reactor. It was theorized that the recycled bottoms material would help eliminate hold up by increasing flow through the reactor.

A summary of the run process conditions is listed in Table 4. The preheater temperature was adjusted at 24 HOS (Period 2) to achieve an isothermal profile (preheater coil = 430°C, reactor outlet = 420°C). The reactor temperature was increased to 410°C, the preheater coil temperature was increased to 435°C and the reactor outlet temperature was increased to 430°C at 48 HOS (Period 4). The majority of the material entering the reactor was going overhead and the plant was shutdown at 81 HOS. The material deposited in the reactor was dissolved in toluene and the toluene-soluble material was analyzed. As shown in Table 5, this material looks very similar to the feed material (Appendix 1). Since there was no increase in the carbon content of the deposits, one could conclude that there was no coking during the run.

TABLE 4
Run 41 Process Conditions

Period	H_2 Drag, cf/hr	Preheat Temp., °C	Reactor Temp., °C
1	0	430	400
2	0	430	400
3	0.5	430	400
4	0	435	410
5	0.5	435	410

TABLE 5
Analysis of Toluene-Soluble Deposits (Run 41)

API	10.7
Carbon, wt%	86.4
Hydrogen, wt%	9.27
Nitrogen, wt%	0.37
Sulfur, wt%	1.20
MCRT, wt%	16.26
Heptane insolubles, wt%	4.56
Toluene insolubles, wt%	0.098

RESULTS

The packing was lowered to the middle heater zone in the reactor during Run 38 to allow for the necessary preheating of the feed while allowing the preheater to be operated at a lower temperature. During this run, the preheater coked severely and was unable to be cleaned. This indicated that the preheater needed to be redesigned prior to starting up again.

It was determined that the preheater coil on the reactor was too large and that it would coke at the operating conditions that were run. The coil was 7 feet of 1/4" OD 18 gauge 316H stainless steel tubing. The feed flow was upflow within the coil. Flow characteristics are unknown within the coil but the residence time could vary from 10 minutes to 30 minutes depending on the flow regime. The new preheater coil is 2 feet of 1/4" OD 18 gauge Hastelloy tubing. Feed flows downward through the coil. This coil was designed to allow a maximum residence time of 5 minutes. This time was based on feed flow alone in the coil; it was assumed that the gas flow rate did not affect the feed flow rate.

Run 39 was the first run with the new preheater. This run was shutdown when all material began going overhead. A layer of sludge was found in the reactor when it was being unloaded. The analysis of the recovered sludge material is shown in Table 6. The slip tube covering the thermowell was coated with a greasy material. There was a build up of material on the reactor walls and the packing was coated with this material. The material was not feed or coke. There were no visible solids in the material. It was greasy and felt gritty. It is believed that the material that was built up on the reactor walls and in the packing was reacted feed. Analysis confirms this and shows that the material is high in Mo (catalyst). It was hypothesized that the metal catalyst was concentrating in the reactor.

Run 40 was performed to determine how much catalyst remained in the reactor and packing under different conditions. At low temperatures (350-360°C), <50 ppm catalyst (<10% of the feed concentration) was left in the reactor. At higher temperatures (400-410°C), as much as 65% of the catalyst in the feed was held up in the reactor (see Table 7). The reactor plugged with the material formed as catalyst accumulated on the walls and packing.

Run 41 was conducted without packing to determine if the packing was the cause of the metal hold-up alone or if the reactor walls also contributed. Once again, the reactor plugged with converted material and metals which deposited on the reactor walls. As a result, it is decided to stop operating the pilot plant in the counter-current co-processing mode. The plant will be reconfigured and operated in the co-current mode.

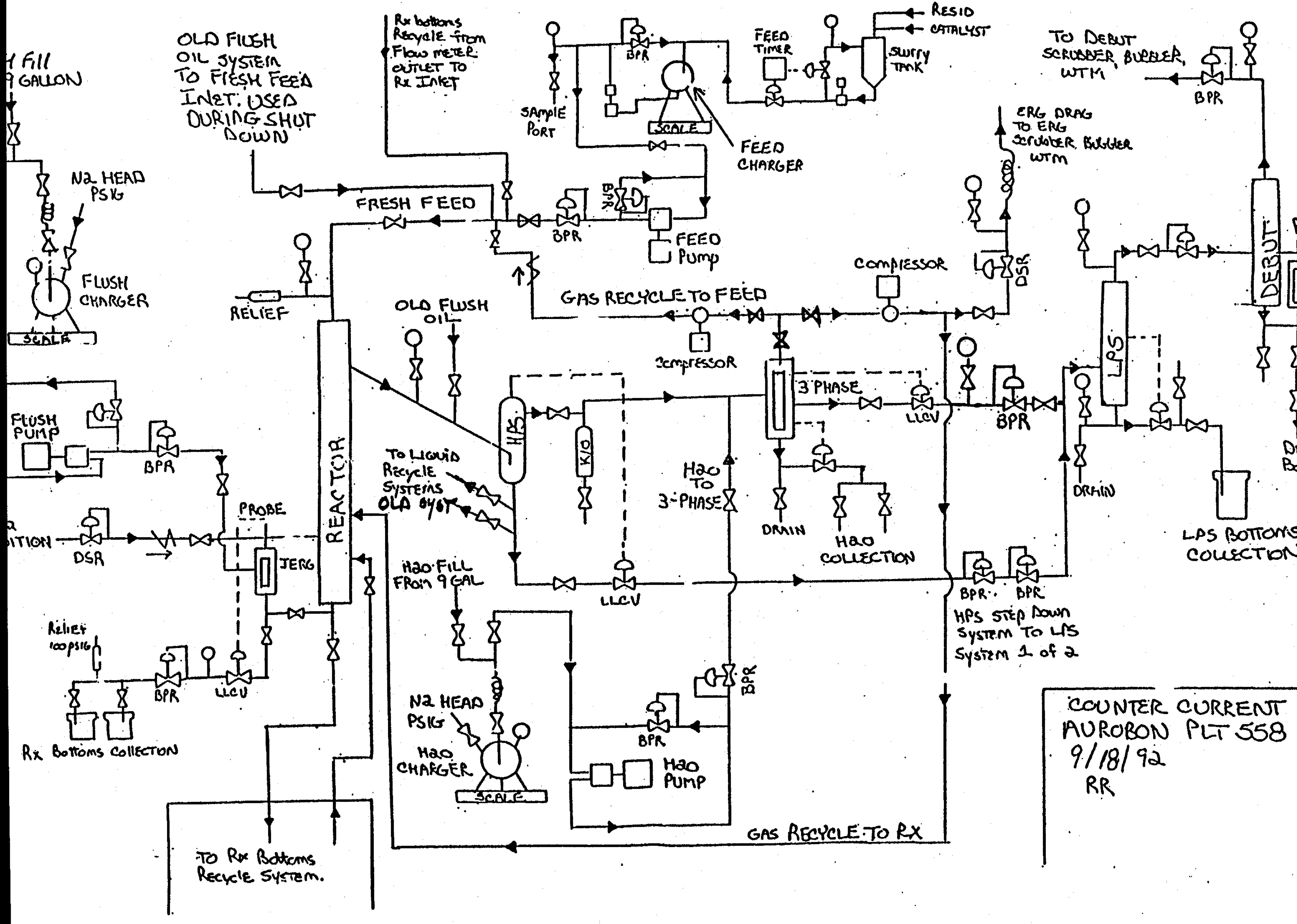
TABLE 6
Analysis of Reactor Sludge (Run 39)

API	7.7
Carbon, wt%	83.12
Hydrogen, wt%	9.23
Nitrogen, wt%	0.46
Sulfur, wt%	4.08
MCRT, wt%	18.70
Heptane insolubles, wt%	11.85
Toluene insolubles, wt%	0.02
Metals Concentration, ppm	
Molybdenum	290
Vanadium	137
Nickel	78

The flow within the reactor is laminar (Re<100). Turbulent flow within the reactor should keep the metals well distributed and eliminate deposition of the metal Unfortunately, the flow rates necessary to obtain turbulent flow within the reactor are too high for the reactor size. A new reactor will have to be designed before co-processing work can continue in Plant 558.

TABLE 7
Mo Concentrations in Reactor Bottoms (Run 40)

Period	ppm Mo	ppm V	Temp., °C
Feed	580	176	
L/O	552	175	350
1	557	177	350
2	561	179	350
3	543	177	350
4	521	172	350
5	525	173	360
6	506	163	360
7	462	156	400
8	424	139	400
9	398	132	410
10	386	129	410
11	412	138	410
12	290	104	410
13	248	98	410
14	167	78	410
17	54	42	410
18	9.4	22	410



COUNTER CURRENT
AUROBON PLT 558
9/18/92
RR
REACTOR
FRESH FEED
OLD FLUSH OIL SYSTEM TO FRESH FEED INLET. USED DURING SHUT DOWN
Rx bottoms Recycle from Flow meter outlet to Rx Inlet
SAMPLE PORT
FEED CHARGER
SCALE
FEED TIMER
SLURRY TANK
RESID
CATALYST
FEED PUMP
BPR
GAS RECYCLE TO FEED
COMPRESSOR
ERG DRAG TO ERG SCRUBBER, BUBBLER WTM
DSR
TO DEBUT SCRUBBER, BUBBLER, WTM
DEBUT
PRODUCT
DEBUT BOTTOM
LPS
LPS BOTTOMS COLLECTION
DRAIN
3 PHASE
LLCV
H2O TO 3-PHASE
H2O COLLECTION
HPS
K/O
OLD FLUSH OIL
RELIEF
To Liquid Recycle Systems OLD 84bt
H2O FILL FROM 9 GAL
N2 HEAD PSIG
H2O CHARGER
H2O PUMP
HPS step down System To LPS System 1 of 2
GAS RECYCLE TO RX
PROBE
JERG
Relief 100psig
LLCV
Rx Bottoms Collection
TO Rx Bottoms Recycle System.
FLUSH PUMP
FLUSH CHARGER
N2 HEAD PSIG
FILL 9 GALLON

FIGURE 1

APPENDIX 1

FEED ANALYSIS

Analysis of Lloydminster Vacuum Resid
UOP 58-1625

API Gravity	6.6
Specific Gravity	1.0246
Distillation, °C	
IBP	379
5	455
10	473
20	509
EP	512
% EP	22
% 510°C	81
C wt%	83.6
H wt%	10.3
S wt%	4.8
N wt%	0.6
MCRT wt%	17.4
C_7 Insol wt%	13.6
Ni ppm	82
V ppm	183
Pour Point, °C	54

APPENDIX 2

RUN SUMMARIES

PLANT NO. 558 RUN 138

FEED STOCK NO. 7896-63 LOYDMINSTER VAC BOTTOMS

Catalyst = 600 PPM MOLY Relative Density = 1.025

PER. NO.	Tot HRS	GMS FEED CHGD	GMS FLUSH CHGD	RECY/ FEED RATIO	RX INTER ABT	GMS H2 Add.	RECY TO FD SCFB	RECY TO RX SCFB	*3 LHSV	H2 Purity	* WT% H2S
1	12	1574	0.0	0.0	385.1	0.0	8038	3292.7	1.89	99.88	0.13
2	12	1439	0.0	0.0	385.5	0.4	10406	3218.6	1.73	99.78	0.00
3	12	1681	0.0	0.0	389.2	5.5	7668	3780.9	2.02	99.79	0.00
4	12	1302	0.0	0.0	389.5	9.7	ERR	ERR	1.57	ERR	0.31
5	12	1571	0.0	0.0	389.5	9.7	7391	3323.1	1.89	97.47	0.25
6	12	1397	0.0	0.0	389.7	15.4	9603	3778.8	1.68	96.53	0.43
7	12	1595	0.0	0.0	390.4	17.0	8367	3330.6	1.92	98.42	0.13
8	12	1405	0.0	0.0	391.0	17.5	10010	3741.6	1.69	94.70	0.64
9	12	1309	0.0	0.0	395.5	8.0	10984	3892.1	1.58	99.06	0.00
10	12	1663	0.0	0.0	395.4	7.3	0	0.0	2.00	0.00	0.00
11	12	1665	0.0	0.0	395.5	3.0	0	0.0	2.00	0.00	0.06
12	12	1460	0.0	0.0	396.8	2.3	0	0.0	1.76	0.00	0.00
13	12	1512	0.0	0.0	396.0	3.5	0	0.0	1.82	0.00	0.53
14	12	1420	0.0	0.0	397.3	3.9	0	0.0	1.71	0.00	0.49
15	12	1597	0.0	0.0	401.4	13.1	6491	2880.1	1.92	82.91	2.07
16	7	862	0.0	0.0	402.8	11.7	4823	1942.0	1.78	50.94	10.32

PLANT NO. 558 RUN NO. 38

(Page #2)

PER. NO.	WT% H2O	WT% H2	WT% C1	WT% C2	WT% C3	WT% C4	FRACT. OVHD	FRACT. BOTTS	WT% REC.	(g H2/ g Feed)	NOTES
1	0.00	1.03	0.01	0.00	0.00	0.00	1.8	86.7	89.7	−1.03	A
2	0.00	0.10	0.00	0.00	0.00	0.00	1.1	76.2	77.4	−0.08	B
3	0.00	0.66	0.01	0.00	0.00	0.00	0.5	89.0	89.9	−0.33	C
4	0.00	ERR	ERR	ERR	ERR	ERR	ERR	71.3	ERR	ERR	D
5	0.00	0.88	0.04	0.03	0.02	0.00	2.2	97.0	99.8	−0.26	E
6	0.00	1.72	0.10	0.05	0.08	0.00	5.3	90.3	96.9	−0.62	
7	0.00	0.83	0.04	0.01	0.00	0.00	4.5	96.4	100.9	0.23	
8	0.00	0.82	0.04	0.01	0.02	0.02	6.0	91.3	97.6	0.43	F
9	0.00	0.27	0.02	0.01	0.00	0.00	5.8	92.5	98.0	0.34	G
10	0.00	0.00	0.00	0.00	0.00	0.00	4.0	0.0	100.4	0.43	H
11	0.00	0.00	0.01	0.00	0.00	0.00	3.9	91.9	95.7	0.18	
12	0.00	0.00	0.00	0.00	0.00	0.00	4.7	95.5	100.0	0.15	I
13	0.00	0.00	0.02	0.00	0.00	0.00	4.0	93.4	97.8	0.23	J
14	0.00	0.01	0.01	0.00	0.00	0.00	6.1	96.8	103.1	0.27	K
15	0.00	0.78	0.23	0.09	0.07	0.02	5.2	82.3	90.1	0.04	L
16	0.00	0.70	0.21	0.14	0.17	0.16	57.1	61.6	128.7	0.65	M

PLANT 558 RUN 38 OPERATIONAL NOTES

*WT% H2S Through WT% Fract. Botts based upon "MAF" Feed.
*1 Fract. OVHD includes C5+ from ERG & DOH gas.
*2 WT% FRACT BOTTOMS BASED UPON FRESH FEED/ NO FLUSH
*3 THE LHSV BASED UPON RX VOID VOLUME =
*4 THE WT% REC BASED UPON FEED AND FLUSH OIL

A–D	PERIODS 1–4 WERE NOT SUMMARIZED DUE TO MANY FEED PUMP PROBLEMS. THE FEED RECYCLE PUMP HAD TO BE REPLACED ON 3 DIFFERENT OCCASIONS.
E.	DOH SPARGER PLUGGING THROUGH PERIOD, REPLACED SPARGER. LOWERED HOT BOX TEMP TO0 110 DEG C. TO AID RECYCLE PUMP EFFICIENCY.
F	INCREASED FEED RECYCLE PRESSURE TO 800 PSIG TO TRY AND HELP LINE OUT FEED RATE. BYPASSED FEED HOTBOX FILTER TO SEE IF FLOW RATE LINES OUT (130.5 HOS)
I	INCREASED FEED PUMP TEMPERATURE TO 150 C
J	157 HOS H2 DISP BOX NOT COUNTING. 159 HOS DOH blottle leaking.
K	178 HOS H2 DISP BOX NOT COUNTING.
L	CUT IN ERG DRAG AT 0.50 SCFH L/O TEMPS; PREHEAT 395 TOP 400 MID 400 BOTT 390
M	192 HOS –4 INCH RX LEVEL ROSE 6 DEGREES. 193 HOS HPS LEVEL BUILT UP TO 52 MA. 194 HOS LOST LEVEL IN HPS. 196 HOS LOST REACTOR BOTTOMS. 196.5 HOS REACTOR BOTTOMS LEVEL CAME BACK. 199 HOS PREHEATER PLUGGED AND SHUTDOWN PLANT.

PLANT NO. 558 RUN 39

FEED STOCK NO. 7896-63 LLOYDMINSTER VAC BOTTOMS

Catalyst = 600 PPM MOLY Relative Density = 1.025

PER NO.	Tot HRS	GMS FEED CHGD	GMS FLUSH CHGD	RECY/ FEED RATIO	RX INTER ABT	GMS H2 Add.	RECY TO FD SCFB	RECY TO RX SCFB	*3 LHSV	H2 Purity	* WT% H2S
1	12	1497	0	0	400	4.4	0	0	1.80	0.00	0.00
2	12	1490	0	0	400	5.1	8169	3846	1.79	97.10	0.00
3	12	1517	0	0	400	4.8	7477	3661	1.83	97.10	0.00
4	12	1501	0	0	401	6.2	7791	4091	1.81	97.10	0.00
5	12	1483	0	0	401	41.1	7651	3567	1.78	93.88	1.62
6	12	1497	0	0	410	9.9	7886	3832	1.80	97.10	0.00
7	12	1468	0	0	411	9.0	8351	3871	1.77	97.10	0.00
8	12	1503	0	0	411	11.3	7768	3672	1.81	97.10	0.47
9	12	1487	0	0	412	9.7	7672	3731	1.79	97.10	0.27
10	12	1474	0	0	410	16.2	8765	3999	1.77	97.10	0.95
11	12	1448	0	0	408	33.5	9587	1892	1.74	92.75	0.62

PLANT NO. 558 RUN NO. 39

(Page #2)

PER. NO.	WT% H2O	WT% H2	WT% C1	WT% C2	WT% C3	WT% C4	*1 WT% FRACT. OVHD	*2 WT% FRACT. BOTTS	*4 WT% REC.	H2 Consump	NOTES
1	0.00	0.00	0.00	0.00	0.00	0.00	1.2	97.7	98.6	0.29	A,B
2	0.00	0.01	0.00	0.00	0.00	0.00	2.8	97.1	99.6	0.33	C
3	0.00	0.01	0.00	0.00	0.00	0.00	2.7	95.5	97.9	0.31	D
4	0.00	0.01	0.00	0.00	0.00	0.00	2.7	96.5	98.8	0.41	E
5	0.00	1.86	0.14	0.03	0.08	0.05	2.5	96.3	99.8	0.91	
6	0.00	0.05	0.00	0.00	0.00	0.00	3.9	95.0	98.3	0.62	F
7	0.00	0.01	0.00	0.00	0.00	0.00	4.6	93.9	97.9	0.61	G
8	0.00	0.01	0.00	0.00	0.00	0.00	5.7	90.4	95.9	0.75	H
9	0.00	0.01	0.00	0.00	0.00	0.00	5.7	83.8	89.2	0.64	I
10	0.00	0.01	0.00	0.00	0.00	0.00	59.6	28.6	88.2	1.09	J
11	0.00	0.65	0.28	0.11	0.07	0.02	53.7	37.0	90.4	1.66	K

PLANT 558 RUN 39 OPERATIONAL NOTES

*WT% H2S Through WT% Fract. Botts based upon "MAF" Feed.
*1 Fract. OVHD includes C5+ from ERG & DOH gas.
*2 WT% FRACT BOTTOMS BASED UPON FRESH FEED/ NO FLUSH
*3 THE LHSV BASED UPON RX VOID VOLUME =
*4 THE WT% REC BASED UPON FEED AND FLUSH OIL

*** USED PERIOD 5 ERG ANAYSIS FOR H2 PURITY FOR ALL CALCS.

A. Preheat=390 T,M,B=400 No ERG gas. L/O F.F. rate erratic – Bled pump.

B. F.F. rate stable at +/– 5 g/hr.

C. 24 HOS H2 to Rx displacement meter not working correctly.
27 HOS Displacemnt meter working again.
36 HOS 3–phase not draining properly. Adjusted LLCV.

D. 38 HOS Slurry mixer not working properly. Reduced RPM from 500 to 350.

E. 60 HOS HPS level increased from 30 to 45 ma.
60 HOS Cut in ERG drag.
67 HOS Lost RX bottoms level. Got back in 5 minutes.

F. Preheat=390 T,M,B=410 C
73 HOS Bled P.P. through ERG to get P.P. to 2000.
81 HOS LPS low level (17 ma from 25 ma. Adjusted LLCV.

G. 85 HOS DOH Sparger leaking.
87 HOS Took F.F. and LPS samples.

H. 96 HOS –4 inch level dropped 4 C.
97 HOS DOH Sparger leaking.
106 HOS –4 inch level returned to normal.

I. 109 HOS DOH leaking.
110 HOS F.F. rate low. Bled feed pump.
110 HOS Lost 3–Phase HCBN level into LPS. Adjusted H20 take–off LLCV.
116 HOS Replaced 3–phase H20 LLCV take–off.
119 HOS –4 inch level temp increased 9 degrees.
119 HOS –7 inch temp increased 7 degrees.

J. Preheat=400 T,M,B=405
120 HOS Reactor bottoms level cycling.
120 HOS 26 degree temperature delta in paking bed.
124 HOS HPS increased from 30 to 45 ma.
125 HOS Catalst bed ABT 5 degrees higher than block temps.
125 HOS Increased preheater to 410 C.
125 HOS Increased H2 to Feed to 9000 SCFB (.63 disp time).
127 HOS HPS increased from 30 to 40 ma.
127 HOS Lost Rx bottoms level. 15 minutes later level building again.
129 HOS Lost Rx bottoms level, grove pressure low.
129 HOS 87% OVHD.

K. P,T,M,B=400
133 HOS 100% OVHD.
134 HOS Increased preheater to 415.
134 HOS Increased F.F. line temperature to 210 (+ 10).
135 HOS 3–Phase HCBN black.
136 HOS Drained 3–Phase and flushed with Toluene.
138 HOS 93% OVHD.
139 HOS Cut out the H2 to Reactor gas. Bottoms level building.
144 HOS Could not get any bottoms level with any H2 to reactor rate.
144 HOS Began Shutdown.

PLANT NO. 558 RUN 40
FEED STOCK NO. BK# 7896-83
Catalyst = 600 PPM MOLY Relative Density = 1.0246

PER NO.	Tot HRS	GMS FEED CHGD	GMS FLUSH CHGD	RECY/ FEED RATIO	RX INTER ABT	GMS H2 Add.	RECY TO FD SCFB	RECY TO RX SCFB	*3 LHSV	H2 Purity	* WT% H2S
L/O	12	1553	0	0	344	19.2	0	0	1.87	0	0.00
1	12	1575	0	0	343	5.3	0	0	1.90	0	0.00
2	12	1239	0	0	342	2.3	9314	3733	1.49	100	0.00
3	12	1448	0	0	341	3.5	7701	3953	1.74	100	0.00
4	12	1466	0	0	341	41.1	8506	3645	1.76	100	0.20
5	12	1462	0	0	352	2.1	7623	3595	1.76	100	0.00
6	12	1449	0	0	352	2.8	7554	3737	1.74	100	0.00
7	12	1421	0	0	390	4.2	8500	3963	1.71	100	0.00
8	12	1439	0	0	390	28.2	5106	2172	1.73	56	2.02
9	12	1441	0	0	399	7.4	8266	4137	1.73	99	0.00
10	12	1430	0	0	398	7.2	8371	4084	1.72	99	0.00
11	12	1455	0	0	396	8.5	7676	4042	1.75	99	0.00
12	12	610	0	0	405	23.8	18256	8287	0.73	85	5.41
13	12	731	0	0	409	6.0	17325	7821	0.88	99	0.55
14	12	875	0	0	411	7.4	14004	6499	1.05	99	0.23
15	12	179	0	0	352	5.1	72002	31549	0.22	99	0.00
16	12	680	0	0	408	25.8	17732	7900	0.82	93	0.74
17	12	732	0	0	410	12.0	15360	6804	0.88	100	0.27
18	12	708	0	0	411	14.1	15700	4097	0.85	100	0.57
19	12	702	0	0	410	28.6	18499	4276	0.84	100	1.28

PLANT NO. 558 RUN NO.: 40

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PER. NO.	WT% H2O	WT% H2	WT% C1	WT% C2	WT% C3	WT% C4	*1 WT% FRACT. OVHD	*2 WT% FRACT. BOTTS	*4 WT% REC.	H2 Consump	NOTES
L/O	0.00	0.00	0.00	0.00	0.00	0.00	0.8	81.6	81.3	1.23	A
1	0.00	0.00	0.00	0.00	0.00	0.00	−0.1	102.0	101.5	0.34	B
2	0.00	0.00	0.00	0.00	0.00	0.00	1.6	103.3	104.8	0.19	C
3	0.00	0.00	0.00	0.00	0.00	0.00	0.8	101.5	102.0	0.24	
4	0.00	0.43	0.00	0.00	0.00	0.00	−4.3	103.2	96.8	2.37	D
5	0.00	0.00	0.00	0.00	0.00	0.00	1.3	97.5	98.7	0.14	E
6	0.00	0.00	0.00	0.00	0.00	0.00	1.8	100.6	102.2	0.19	F
7	0.00	0.01	0.00	0.00	0.00	0.00	2.8	97.2	99.6	0.28	G
8	0.00	0.16	0.01	0.00	0.00	0.00	2.4	95.2	97.9	1.80	H
9	0.00	0.01	0.00	0.00	0.00	0.00	6.4	93.8	99.6	0.50	
10	0.00	0.01	0.00	0.00	0.00	0.00	5.6	94.1	99.2	0.49	
11	0.00	0.01	0.00	0.00	0.00	0.00	8.1	91.8	99.4	0.57	I
12	0.00	2.38	0.50	0.26	0.27	0.22	19.5	81.5	106.0	1.52	J
13	0.00	0.08	0.00	0.00	0.05	0.00	21.2	69.2	90.3	0.74	K
14	0.00	0.03	0.00	0.03	0.08	0.05	27.8	56.9	84.4	0.81	L
15	0.00	0.06	0.00	0.00	0.00	0.00	0.8	76.6	75.3	2.77	M
16	0.00	2.04	0.64	0.32	0.39	0.11	22.9	56.6	80.7	1.76	N
17	0.00	0.04	0.03	0.00	0.09	0.12	41.3	49.0	89.4	1.60	O
18	0.00	0.04	0.05	0.13	0.24	0.31	32.2	70.2	101.7	1.95	
19	0.00	0.10	0.22	0.35	0.56	0.61	63.1	45.9	107.7	3.98	P

PLANT 558 RUN 40 OPERATIONAL NOTES

*WT% H2S Through WT% Fract. Botts based upon "MAF" Feed.
*1 Fract. OVHD includes C5+ from ERG & DOH gas.
*2 WT% FRACT BOTTOMS BASED UPON FRESH FEED/ NO FLUSH
*3 THE LHSV BASED UPON RX VOID VOLUME =
*4 THE WT% REC BASED UPON FEED AND FLUSH OIL

A 4 HOS Lost Rx bottoms and some plant pressure.
HPS level fluctuating.

B 13.75 HOS Dumped 400 psi of plant pressure.
20.30 HOS Lost Rx bottoms and 100 psi plant P.
21.82 HOS Rx bottoms dumped and plt P down.
HPS off-gas line was blocked in. Opened and plant stabilized.

C 27 HOS Feed charger low and unable to fill.
27-29 HOS Missed readings and did not monitor plant.

D. Cut in ERG Drag.
51 HOS HPS level dropped by 2 mA. Reason unknown.
53.67 HOS Lost Rx bottoms and plant pressure.
54 HOS Drained 3-phase to remove emulsified water/oil.

E. 60 HOS Cut out ERG.
70 HOS Drained 3-phase emulsion.
70 HOS Feed rate decreased 30 g/hr for no reason.

F. 79 HOS Low feed rate.

G. Started Sampling DOH.
Increased Rx Temps to 400 C.
87 HOS Reset HPS low pressure safety switch.
93 HOS Changed recycle specs.

H. 97 HOS Dumped LPS.

I. 138 HOS 3-Phase HCBN now working.

J. Lowered F.F to 50 g/hr and cut in ERG.
146 HOS Increaed F.F. BPG to 3,000.
147 HOS HPS dumping to 26 ma. Adjusted LLCV.
147 HOS Increased preheater temps to 420.
150 HOS Increased preheater temps to 430.
151 HOS Decreased Prheheater to 425.

K. 161 HOS H2 DMS not working properly. Replaced P.P. regulator.
165 HOS Increaed F.F. spec to 60 g/hr. Feed rate still erratic.
Put insulaton on top of reactor.
Weight recovery is low (90%) and feed rate is still erratic.

L. 175 HOS Hot spot developed in -2 to 2 inch level. Increased Preheater
and top bed temps. Lowered middle temp. Profile looks better.
178 HOS Adjusted feed pump discharge to get even F.F. flow. Inadvertently
increased Zenith charger BPG and forced 300 g of F.F. into Rx. FLOODED!
178.5 HOS Shutdown F.F. and Zenith charger pumps.
179 HOS Depressured Zenith and restarted. No discharge P. Charger Plugged.
Decreased Rx and Preheater temps to 320 C.

M. 183 HOS Cleared Charger restriciton. Reconnected.
185 HOS Replaced Flex line. Leaked.
186 HOS Blew restriciton out of flex line and reconnected.
187 HOS Replaced Shear on Zenith Pump.
189 HOS Cut feed back into Plant. Drained HPS, LPS, and 3-Phase.
190 HOS Increased Temperatures to 410 C.

N. 201 HOS HPS cycling - reset LLCV.
203 HOS Zenith Bottoms recycle pump not pumping. Cleared restriciton.

O. 214 HOS Dumped pressure on Hycbn takeoff on 3-Phase

P. Shutdown plant at 240 HOS due to restriction in RX not allowing anything down

PLANT NO. 558 RUN 41
FEED STOCK NO.
Catalyst = 600 ppm Relative Density = 1.025

PER. NO.	Tot HRS	GMS FEED CHGD	GMS FLUSH CHGD	RECY/ FEED RATIO	RX INTER ABT	GMS H2 Add.	RECY TO FD SCFB	RECY TO RX SCFB	*3 LHSV	H2 Purity	* WT% H2S
1	12	677	0	4.43	395	4.4	16589	8086	0.81	97.33	0.00
2	12	650	0	4.61	396	5.1	18538	7895	0.78	97.33	0.00
3	12	815	0	3.68	395	25.4	14271	6642	0.98	94.40	0.98
4	12	672	0	4.46	403	7.4	17010	7415	0.81	94.91	0.45
5	12	1016	0	2.95	403	27.0	10903	4778	1.22	91.52	1.18

PLANT NO. 558 RUN NO. 41

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WT% H2O	WT% H2	WT% C1	WT% C2	WT% C3	WT% C4	*1 WT% FRACT. OVHD	*2 WT% FRACT. BOTTS	*4 WT% REC.	H2 Consump	NOTES
0.00	0.02	0.00	0.00	0.00	0.00	12.5	80.1	92.1	0.63	A
0.00	0.02	0.00	0.00	0.00	0.00	18.1	78.5	95.9	0.76	B
0.00	1.61	0.23	0.13	0.11	0.05	13.1	83.7	96.9	1.51	C
0.00	0.02	0.00	0.00	0.00	0.00	22.6	71.8	93.8	1.08	D
0.00	1.20	0.34	0.17	0.17	0.07	22.8	69.2	92.7	1.46	E

PLANT 558 RUN 41 OPERATIONAL NOTES

*WT% H2S Through WT% Fract. Botts based upon "MAF" Feed.
*1 Fract. OVHD includes C5+ from ERG & DOH gas.
*2 WT% FRACT BOTTOMS BASED UPON FRESH FEED/ NO FLUSH
*3 THE LHSV BASED UPON RX VOID VOLUME =
*4 THE WT% REC BASED UPON FEED AND FLUSH OIL

A	13 HOS Reduced setting on bottoms recycle B&L pump 15.5 HOS Changed actuator on H2 make−up system 21 HOS Rx Botts LLCV leaking through
B	25 HOS H2 addition DM not counting. Tightened contacts. FF rate erratic all period 30.5 HOS FF pump leaking.
C	36 HOS Cut in ERG drag 39 HOS Rx bottoms level increasing. Adjusted LLCV. 44 HOS Almost lost bottoms level. Adjusted all LLCV at least once during period. Lost no level completely.
D	48 HOS Cut out ERG drag. Increased blocks to 410 C Increased preheat top to 435 C Increased preheat bottom to 430 C 57 HOS FF pump leaking
E.	61.5 HOS Cut in ERG drag. FEED Rate erratic during period. 69 HOS Rx outlet temperature dropped 15 degrees. 71 HOS Lost Rx bottoms. Cut recycle, recirculation, and H2 to Rx. 72 HOS Rx bottoms returned. Cut in pumps and H2 flow to normal. 74 HOS Lost Rx bottoms and cut recycle, recirculation, and H2 to Rx. 79 HOS Getting 117% OVHD. 80 HOS Began plant shutdown.