APPENDIX A

Fischer-Tropsch III Run Chronology

Fischer-Tropsch III Run - October/November 1996

4 October 1996

6:00 Loaded the reactor with oil: 80" Rxt ht, 48" on tape. Started nitrogen flow and heating up. The 10.62 pump not running overnight.

5 October 1996

8:00 Reached reactor temp of $\sim 450^{\circ}$ F. N₂ flow (Rxt feed FI-187) set @ 90,000 SCFH. Reactor level set at 210" on tape.

DEC is having communication problems with Bailey. TI-687-1 had wrong block location, fixed. No more errors in DEC, but communication problems continue. Some signals are "zero" when they should have some values, DEC gives OK signal. The obvious signal not working is PIC-201, many others. PIC-201 was working overnight, but stopped working when Nextgen was rebooted. The rebooting was done because TI-1781 A and C were not working before. They are working now!

14:30 10.62 pump seal failed. Leaked seal oil into the process oil. Will need new seals. Overnight the pump is cooling, the reactor will be de-pressurized tomorrow.

6 October 1996

16:30 New seals/parts for 10.62 received and installed. Clarification: seal was rebuilt. Began operating 10.62 pump and slowly raising temperature.

7 October 1996

- 6:00 10.62 pump still running, temp @~ 250°C and degasser is being bypassed. Nuke scan taken.
- 10:00 Attempts are being made to get flow thru the degasser loop; However, they have been unable to maintain stability.

Temperature has been reduced, filtration loop has been isolated, additional oil has been brought in to raise level in the filters.

- 12:50 Level in Rx = value in degasser 187 flow = 40000 scfh, temp 240°C
- 13:00 CH4 in Plant
- 13:25 Nuke scan
- 15:00 Flare is lit...NOMEX!
- 23:00 Decreased gas flow to reactor to decrease level to 211". (50,000 scfh 46,000 scfh). Level in 27.15 runs about 22%. At 211" in reactor. Gas holdup dropped from 43% to 39%.

9 October 1996

21:30 Felt the steam traced piping to make sure that they're hot:

- Piping around 10.62 pump NOT hot ***
- Piping around 22.62 A-D filters hot
- Piping around 27.15 degasser hot
- Piping around 28.30 tank hot
- Didn't check piping expand to light wax (e.g. 22.14, 27.13)

10 October 1996

11:30 Piping around 22.14 and 27.13 checked and hot (DURASYN - 164 = ETHYLFLO-164)

- \sim 13:00 Loaded 46.2 lb oil into 28.30 prep tank. Drum before filling = 206.2 lb. Drum after filling = 160.0 lb.
- 13:50 Loaded 5 bags of wax.
- 15:05 Loaded 14 bags of wax. Weight of amount loaded after 19 bags = 973 lbs.
- 16:15 Finished loading last 10 bags and a bucket containing 17 pounds. Total amount of wax loaded to 28.30. Prep tank = 1489 lbs.

Wax Loading

Tank T = 155°

١	٨	12	v
v	· v	$\boldsymbol{\alpha}$	

vvax			
Bag #	Weight of Bag	Weight -50	Total Deviation
4		0	
1	50	0	-
2	50	0	-
3	56	+6	+6
4	54	+4	+10
5	48	-2	+8
6	48	-2	+6
7	50	0	+6
8	52	+2	+8
9	48	-2	+6
10	55	+5	+11
11	52	+2	+13
12	54	+4	+17
13	52	+2	+19
14	50	0	+19
15	50	0	+19
16	50	0	+19
17	56	6	+25
18	50	0	+25
<u>19</u>	59	9	<u>+34</u>
20	48	-2	+32
21	54	+4	+36
22	50	0	+36
23	47	-3	+33
24	52	+2	+35
25	48	-2	+33
26	51	+1	+34
27	51	+1	+35
28	50	0	+35
29	<u>54</u>	+4	<u>+39</u>
30			
11/2:2/24	final 40 amandu.	عطاله محمط	

Weight of first 19 empty bags = 11 lbs

Weight of last 9 empty bags = 6 lbs

Amount loaded after 19 bags:

19(50) + 34 - 11 = 973 lb

Weight of 9 bags left to add

10(50) = 500 + 5 = 505 lb

Amount of 20 bags

Weight = 1478 lb

Added bucket containing 17 lbs extra

Total including bucket full = 1495 Subtract weight of empty 10 bags -6 Amount added = 1489

11 October 1996

16:30 Catalyst loading - start (Shells' CMT-25 Fischer-Tropsch Cat).

17:15 Finish catalyst loading.

1. 1st drum

> Weight catalyst, drum, funnel = 406 lb

Weight drum funnel = 127 lbÀ Weight catalyst = 279 lb

2. 2nd drum

> Weight catalyst, drum, funnel = 510 lb

Weight drum, funnel = 128 lbÀ Weight catalyst = 382 lb

3. 3rd drum

> Weight catalyst, drum, funnel = 509 lb

Weight drum, funnel = 127 lbA Weight catalyst = 382 lb

Total catalyst loaded 4.

> Weight catalyst (1st drum) = 279 lb Weight catalyst (2nd drum) = 382 lb Weight catalyst (3rd drum) = 382 lb

TOTAL catalyst loaded = 1043 lb

- 18:00 Filled drum with lb of flush oil. [Weight of drum and oil = 368 lb.]
- 19:05 Begin transferring oil/catalyst slurry from 28.30 to 27.10.
- 19:35 Finish transferring oil/catalyst slurry.

Looked inside 28.30 slurry tank and saw a lot of catalyst at the bottom.

19:50 Weight of drum (no oil) = 115 lb À Flush oil weight = 253 lb oil

- 20:00 Weight of 2nd oil drum & oil = 362 lb
- 20:20 Weight of 2nd oil drum no oil = 115 lbA Weight of 2nd oil = 247 lb
- 20:45 Currently in the catalyst drying step in the 27.10 reactor. Will begin heating the reactor to get an additional 40°F in the next 2 hours.
- 21:35 PIC-201 Rxt Pr = 505 psig

Rxt Avg temp = 243°F

Rxt feed flow (N_2 100%) = 44.69 MSCFH

23:30 Plant conditions steady for last couple of hours on dry out step.

Fresh feed flow 10,500 SCFH Nuke Scan:

Reactor feed flow 42,000 SCFH

Reactor pressure 505 psig

Reactor temperature 268°F (and climbing slowly) Level is still high (seems to be right at the top of nuke)

Gas holdup corrected later 21.8%

PDI - 1778 46 PSID PDI - 1779 51 PSID

Sparger PDI Unstable between 10 and 25 PSID

01.10 discharge pressure 533 PSIG (À total reactor $\Delta P = 28$ PSID including piping and equipment downstream of 01.10).

It seems like the PDI values are off by a factor of 10??? No - Bailey shows units of " $\underline{\text{H}_2\text{O}}$. PDI-631 across the reactor isn't reading anything - probably plugged up (although it spikes occasionally).

12 October 1996

- 00:20 Moisture probes are showing steady baselines at about 870 and 1370 PPM. But with spikes (+300 400 PPM) every 10 15 minutes.
- 00:30 Utility oil heaters tripped at about midnight. Reactor temperature lost above 5°F and then come back in about 10 minutes.
 - Reactor average temperature is now 283°R and we will continue heating at 60°/hr.
- 01:30 Looks like sparger PDI has plugged up. But essentially at the same time PDI-631 across the reactor seems to have come back.
- 03.20 Nuke scan:

Fresh feed 10,500 SCFH Reactor feed 41,400 SCFH Reactor pressure 506 PSIG

Reactor temp 410°F (still climbing)

Level still over top of nuke

Still expanding but no carryover in 22.14

Gas holpup corrected later 35.2%

This seems very high. 33% calculated from ΔP measurements.

Sparger PDI has shown 0.5 - 0.7 PSID for about the past hour. This may actually be accurate.

- 04:15 AI-1796A spiked from about 1000 PPM to about 2500 PPM (greater than reactor outlet (2000 PPM) which must be bogus) and held there.
- 04:50 Reactor temperature at 489°F. AJ cutting back to level out right at 500°F.

 ΔP measurements seem to be reasonable (including sparger and total reactor).

AI-1796B at about 2000 PPM and still climbing.

AI-1796A at about 2400 PPM and stable (doesn't make sense).

- 05:20 Reactor T at 500°F and holding.
- 06:00 Nuke Scan

Fresh feed 10,500 SCFH
Reactor feed 40.84 MSCFH
Reactor pressure 506.52 psig
Reactor temp 501°F

Level over top of nuke

Gas holdup corrected later 36.0%

- 06:35 Dave and Ray opened CW to 21.20 a little to maintain flow and avoid vapor lock. Reactor T dipped to about 485°F before recovering.
- 07:35 Reactor T back up to 500° F. Put N₂ to Panametrics and readings dropped to <100 PPM.
- 07:50 Went out to purge with N₂ again, readings went up again.
- 08:20 NDG detector position @ 4933. Checked in field and this position corresponds 214 inches. This inconsistent with calibration height.
- 09:00 A_2214_ACTVTN_GASOUT_H₂OCONC_A ~2800 PPM (27.13 Rxt In) A_2214_ACTVTN_GASOUT_H₂OCONC_B ~1700 PPM (22.14 Out)
- 10:00 10:53 PI's fluctuating a lot, difficult to get reading.

- 09:35 01.10/01.20 flow adjusted to bring in 20-21 MSCFH (Increasing purge to aid reduction of H₂O concentration in Panametrics).
- 15:15 Nuke Scan

Fresh feed (FI-126) 204.35 SCFH
Reactor feed (FI-187) 40.68 MSCFH
Reactor pressure (PIC-201) 508.77 PSIG
Reactor temp (DEC avg) 501.9°F

Level over top of nuke

Gas holdup 34.56

17:05 27.10 inlet = 1200 ppmv H_2O 22.14 outlet = 1350 ppmv H_2O

For tonight:

- 1. Our target for water in 27.10 and 22.14 is about 500 ppmv H2O. Call Bharat when this happens. He wants to be here when we start to add H2.
- 2. Until the DEC software has been installed by A. Agrawal, we need to take the Bailey readings every hour.
- 3. For the bold items in the field readings, we need to take them every two hours. This means readings at 18:00, 20:00 and 22:00......
- 4. All the rest of the items in the field readings have to be taken every four hours. This means readings at 18:00, 22:00 tomorrow at 02:00......
- 5. Nuke scans every four hours.

This means readings at 19:15, 23:15, tomorrow at 03:15......

19:17 Nuke Scan

Fresh Feed (FI-1260 = 20619 SCFH Reactor feed (FI-187) = 40.92 MSCFH Reactor pressure (PIC-201) = 508.66 psig Reactor temp (DEC) = 502°F

Level over top of nuke

Gas holdings = 33.95

- 17:05 Stopped Nextgen to remove debug lines from driver to save disk space.
- 17:15 Started Nextgen. Came up almost same as previously. Actually gained 2 data points!
- 21:00 Drying complete. Decreasing reactor temp from 500°F to 446°F.
- 23:00 Reactor level still higher than 215" on tape.

React average temp = 448.57°F

 271° F react feed in (A) = 304.20 ppmv

22.14 offgas (B) = 286.39 ppmv

23:20 Bringing in H₂ for reduction. At about 2000 SCFH.

N₂ flow is about 20,000 SCFH.

- 13 October 1996
- 00:35 GC's show 3.5% H₂
 Increasing FI-101 to 3 MSCFH 3.3 MSCFH
- 01:15 H_2 in Rxt = 9% Increasing FI-101 to 4.3 MSCFH (gal 14.1 H_2)
- 02:05 G04001 up to 14.38% H₂
- 02:15 Moving on toward 18% $\overline{H_2}$.
- 02:45 G04001 up to 18.52% H_2 . Moving on towards 25% H_2 .
- 03.20 G04001 up to 25.95% H_2 . Moving on toward 33% H_2 .
- 03:50 Holding 33% H2 and increasing reactor T by 5°F to 451°F.

 Stopped temperature increase after 2°F. Moisture concentration in 22.14 had risen from about 740 to 840 PPM. Then topped out in very direct response to the temperature hold. Increased another 2°F at 04:10, H2 in feed topping out at about 35%.
- 04:30 Not much action on the last temperature ramp. Moving up 3°F to 453°F very slowly.
- 04:50 Moving on toward 455°F. Moisture at 22:14 increasing nicely up to 935 PPM.
- 05:10 At about 453.5°F, moisture spiked up to 1200 ppm! And held.
- 06:25 Still creeping up slowly on temperature. 456.5°F right now.
- 07:55 Reactor temperature at 464°F. Continuing on to 467°F.
- 09:15 Reactor temperature raised to 473°F.
- 09:20 Gas holdup from Nuke Scan 27.31%, level still over top of Nuke.
- 10:30 Reactor temp raised to 476°F.
- 10:45 Increasing H₂ from 7.3 to 10 MSCFH (35.5% to 50% H₂) 22.14 off gas \Rightarrow ~1500 ppmv
- 12:40 Reactor level is visible! ⇒ 215" m type

NDG count = 133

16:24 Reactor temp is being increased in 2°F increments.

1:56 pm up 2 deg. to 481°F 2:22 pm up 2 deg. to 483°F 4:20 pm up 2 deg to 485°F

- 19:40 Raised feed flow to 50000 SCFH to the reactor to maintain the reactor level. Ceis approved to move.
- 19:45 Blocked in methane to the flare.
- 20:25 Raised flow to reactor to 53000 SCFH again to raise reactor level again. Ceis approved the move because the reactor is stable.
- 20:45 Completed the set up for INFI-90 (Bailey data collection) to take the 134 (?) hourly readings and the fifteen 5-minute readings. Please have Al Agrawal check the work to make sure it's set up OK.

The set-up files are found in:

C:/INFIVIEW/SETUP

- 1. AFDUFT3.STP the original file that has all the possible Bailey points.
- FT3TAG5.STP the set up file for the hourly readings.
 As instructed, I have changed the block numbers for the 17 measurements that were listed.
- 3. FT35MIN.STP the set up file for the fifteen readings taken every five minutes. The collected data is found in: C:/INFIVIEW/DATA
- 1. FT3HR1.DAT the data file for the hourly readings.
- 2. FT3MIN1.DAT the data file for the fifteen points taken every five minutes.

- I think the software is collecting data now, and the results can be seen in the trend data.
- 23:30 AJ still bumping up recycle flow to maintain level between 211" and 215". Now at 74 KSCFH total flow. Moisture level in outlet still drifting down slowly (about 80 ppm/hr). Now at 1380 ppm. Reactor temperature holding at 500°F. All we can do now is watch and wait.

14 October 1996

- 02:30 Reactor level is down below 197" and seems to be falling faster at the higher flow rate. (We've been at the target rate of 87 KSCFH since 0100.) We might be stripping out wax faster at the higher gas velocity, so we will reduce the recycle rate. We can't find carryover anywhere 22.14 still seems to be empty, but we could be building level below the sight glass. Dave's last nuke scan at 01:50 was quite uniform and steady seems to be well-mixed.
- 03:20 50 KSCFH on FI-187 to reactor. Level is down to <166". (Interfering down to 157".) Nuke scan was steady and uniform.
- 04:00 Level dropping through 157".
- 04:20 Level dropping through 154".
- 04:45 Called Ed, then Bharat.

Concluded we should:

Add ethyflo to reactor

Drop gas rates to 40 KSCFH

Maintain temperature at 500°F

- 12:25 Begin cooling step for end of reduction.
- 12:41 Temp dropped from $480^{\circ}F 450^{\circ}F$ in ~5 min.
- 13:40 Starting to heat up the 28.30 --- tank by cracking open oil to 28.30.
- 13:50 TI-1-14B dropped about 30°F pretty quickly the last few minutes.

Ref: Test Authorization #52 - meeting to discuss procedure

Filters: NV-1760 is just open/close, actually

LV-203 will throttle

3.3 m 3/h (14.7 gpm) or (126 lb/min)

(density = 1.03 g/cm3)

Eventually want to transfer control to the control valve.

Want backflush pressure higher than the pump discharge pressure.

Want to evenly distribute flow flux thru the filters.

It will be hard to control at these low rates, but we'll keep velocities low.

Possible ΔP is 10 - 60 psi, we're quessing it will be 10 psi

A 770 psi - 760 psi B 780 psi - 770 psi

700 psi - 30 psi

C 790 psi - 780 psi

D 800 psi - 790 psi

Will establish flows thru the filters with clean fluid (ethylflo)

- 15:10 Raise 27.10 reactor pressure to 710 psig.
- 15:50 Finished setting up 10.52.01.

Losing level in 27.12.

15:55 Opening NV-1760 to begin to line up filters. Going to start sending clean liquid thru filters. Start with bottom filter (D). Watching PDI-1777 (1.35 psi).

- 16:25 Now sending slurry from 27.10 to reactor to filters. Watching 10.62 pump. Pumps have failed (10.52? 10.54?).
- 16:55 27.10 reactor level is dropping.
- 17:00 10.62 pump: 128 lb/min, 40% speed, raised it to 60% speed. Line from 28.30 Prep. Tank to 10.52.01 pump is plugged. Can't remove the plug even with 300 psi N₂.
- 17:07 10:54 pump fuse has been replaced. 10.54 restated. Sending 800 psi N₂ to try to send plug bark into 28.30 prep tank.
- 17:58 UNPLUGGED used 1500 psi N₂ thru the back flush line to remove the plug.
- 18:28 Will begin re-pressuring reactor to 710 psig.
- 18:34 Start taking flow thru 21.38 economizer.
- 18:40 Filter $\Delta P = 7$ psi, which is good.
- 19:10 Valve for "D" filter (V-3574) is messed up. Will try to use "C" filter.
- 19:20 V-3574 is glassed wide open. Will back flush "C" and "D" filters. Will need to replace this valve. Closed NV-1760
- 19:26 Opening LV-203 slowly to de-pressure "C" and "D" filters.
- 19:38 Valve V-3574 already replaced.
- 20:15 Bringing CO into plant. Watch for temp. increase in reactor. Keeping total flow same (cutting back N₂). Reactor temp start at 355°, 694 psig.
- 20:20 Bring more H₂
- 20:25 Reactor temp 370°. Reacting much of CO. Bring more H2 7000 MSCFH. CO can't get flow read.
- 20:35 H₂ 8.33 MSCFH

CO 4.34 MSCFH

Reactor 392°, 668 psig

20:50 Lowering reactor level by removing wax.

Level current over nuke.

Reactor 726 psig, 383°

H₂ 12,000 MSCFH

CO 6.0 MSCFH

As level drops - increasing recycle to reactor.

21:15 Recycle 89 MSCFH

Reactor 715 psig, 360°

H₂ 10.7 MSCFH

CO 5.8 MSCFH

- 21:25 Turn off fin fan to raise reactor temperature. Reactor 359° before. Goal is 410°.
- 21:35 Adding more CO 6/7 MSCFH

H₂ - 10.9 MSCFH

- Filters <u>D</u> PDI-1777 17 PSID <u>C</u> PDI-1776 11.4 PSID <u>B</u> PDI-1775 5.6 PSID A PDI-1774 -2 backflush
- 21:40 First product GC spec in.
- 21:55 Reducing the pump 10.62 speed down from 70% to 61.5% to bring flow down to 190 lb/min.
- 23:15 Bringing reactor temp up 405° now

Filters D 15 PSID C 10 PSID B 5.5 PSID D -1 PSID Allowing temp to rise to aid in CO conversion to produce more wax. Due to small wax production - bringing down recycle ratio.

15 October 1996

00:35 21:20 bypass which was open to get reactor temp up is being closed.

00:45 Starting fin fan.

Temp. in reactor shooting through roof, 490°F Fin fan 50% ý 100% still climbing rapidly Reactor temp. peaked at about 530°F ý 790 psig

- 00:45 Compressor tripped SD-1. Low suction pressure.
- 00:50 Compressor restarted. Add N₂ to try to stabilize reactor.
- 00:55 Temperature dropping fast. Full cooling on fin fan.
- 01:45 Feed to reactor stabilized.

 Temperature reactor 358°F
- 02:10 We are going to run at very dilute H₂/CO to N₂ ratio. This hopefully will allow us to control the temperature spike. Adding N₂ with Big Joe.
- 03:00 33.5% N₂ in reactor feed. Make another move on Big Joe.
- 02:30 To increase temperature on reactor (we were stuck at 360°F). Opened more 21.20 bypass so less cooling on oil. Want to get to 420°F then start backing down on N2.
- 05:00 Reactor 428°F. Oil now cooling with fan. Fan ~50%. Temp appears stable controllable with fin fan.
- 05:05 Temperature drop slightly increase H2 flow.

Feed 14% H2 20% CO

React temp 411°F

710 psig

- 05:10 Hit with too much cooling temperature bottomed out at 408°F. Move reactor temperature back toward 420°F.
- 05:35 As control reactor continue to move back towards 2.1 H₂/CO 25% H₂
 - 17% CO in reactor feed.
- 07:05 Slowly backing out N_2 in the feed and establishing ΔT between oil system and reactor temps.
- 07:30 Draining prep tank (Dave and Ray). Raising temperature in 22.14. Still no level in 22.14 or 22.10??
- 09:30 Still no level in 22.14 or 22.10. Working to back out N_2 in the fresh feed and maintain a steady temperature. N2 is being replaced with H_2 .
- 13:00 Transfer from 28.30 prep tank to tank trailer.
 - Ran N₂ through line to make sure it was clear
 - Pressured up 28.30 prep tank to 50 psig
 - Turned agitator off
 - Changed valves so product would go to the tank trailer
 - Opened top of trailer
 - Opened HIC 512 valve
 - Emptied tank until gas came out
 - Shut HIC valve

Level was ~38

Level seen in 22.10. Height/level \cong 3 bolts (5 1/2")

22.10 site glass full.

Product carried over to 22.15 and then to the 22.16 product day tank.

The balls in the site glasses seem to be stuck. Steve Cochran is working to fix them.

- 15:00 Shut LV-203 because we are not making product.
- 16:35 Temperature has dropped form 421 to 416°F over the past few minutes.
- 16:45 Re-establish flow thru filters.
- 19:45 Reactor up to 443°F. Controls back in automatic. Operation is pretty warm and pretty stable.
- 19:48 Transferring liquid from 22:10 degasser to 22.16 day tank.
- 19:52 Trying cascade control on 27.10 reactor temp to see if it works. Target temp 443°F (TI-190-3B).

16 October 1996

00:25 Power loss in control bldg.

Bailey power stayed on.

Only partial power loss. Lost lights first, then DEC.

Powered down all unnecessary equipment to conserve.

GC's apparently lost no power.

Rocco timer not affected so we assume they all stayed on.

00:45 Gadget trying to remedy situation.

GC's still functioning.

DEC still down.

Bailey Operating.

Try to reboot when power restored.

- 01:45 Power back up
- 02:00 DEC rebooted. NG_START

Trend collector and HPGC_DRIVER are running

(GC's never went down)

Points seem to be coming back in OK. Now need to get window view going again.

02:30 Window view seems to be working again and logging data.

Will get Wendyann to check at 06:00.

03:05 Forgot to power-up the DEC printer until now. No snapshots printed since midnight. Some points seem to be "invalid" in the DEC - correction, we were looking at 22.60 filtrate parameters instead of 22.62 parameters. All seems to be well. At current wax production rates the wax take-off (via LIC-203) tends to be all or nothing

At current wax production rates the wax take-off (via LIC-203) tends to be all or nothing (1.7 lb/min or 0.1 lb/min), so level control ends up being on/off to build from 211" and drop from 215". The period is about one hour for 4" change in level.

04:30 Tried to access data point FIC-101 on laptop monitor. On hour longer.

Error: Subject out of range

Crashed. VES got running again.

Note: tracker after power loss was after this crash

B51ns4.dat

B51ins5.dat

- 04:00 Henry took a liquid sample off 22.11. Looks like skim milk.
- 06:15 Window checked and is running. It was shut down momentarily to load file with 134 pts.
- 07:30 Trying to maintain temp ~435°F

D04/B04 - Rx feed - Make sure total 100, they are close.

D02/B02 - Prod gas - Look at 4 hour trend and one hour trend of individual components to see if steady or changing (H₂ & CO). H₂/CO ratio

G04 Time scan questionable, but comp is OK. Can also compare with B04 to see if reading values are close.

A_2262_ Filtrate flow: small \u00fc in 203 (smallest move) goes to 1.6

When level gets up to 215, tell operator to close small amt. (~1 hr cycle). Once level gets down below 211, bring back up again.

Watch filter DP values

Watch HMBFLO1. Ration of 126A/701A

Compare it with 109/606 expected ration from ASPEN.

10:00 Compressor trip

Level dropped to ~188

Temp increased to ~ 443.6

Resetting H₂ at 12.8 mol.%

- 10:05 Level brought back to ~215.
- 10:10 Temp @ 426 and dropping slowly.
- 10:13 Level >215, temperature @ 424.
- 10:30 Raising temperature to 435-440 range with H₂ feed of 13 mol%.
- 12:20 Temp increased still climbing. Max of 469 reached.
- 12:25 Temp coming back down.
- 13:00 Temp leveled off \cong 440.
- 15:10 High 27.10 reactor level. Open LV-203 to transfer wax from 27.10 reactor to 28.30 prep tank.

Initial NDG reading of 30 at voltage of 4863.

Final NDG reading of 230 at voltage of 4863 (finished at 15:25).

Measured wax level/volume in trailer 5692.

- Lowered tubing to top of wax without sticking it into was.
- Marked height in pencil at bottom of metal band.
- Measured length from end of tube to pencil mark

Max capacity height = 77.75"

Empty height = 61"

Height of = 16.75" or 1'4 8/10"

Loaded wax

(wax loaded 10/15/96)

Volume loaded =1022 gal.

- 21:10 Liquid level in 27.10 reactor above 215" transferring a load of wax from 27.10 reactor to 28.30 prep tank.
- 21:50 Completed wax transfer.
- 16:30 Day tank transfer to #5390 (6251 gallon capacity @ 90%)

49 1/2"762.5

20" 240.0

515.5 transferred

17 October 1996

- 03:25 Henry took a liquid sample off 22.11. A little clearer than last night's sample.
- 12:45 Transferred heat product from 27.10 reactor to 28.30 prep tank.
- 14:00 Took a slurry sample.
- 14:40 Wax transfer from 28.30 prep tank to trailer.

Volume transferred = 547 gal. wax (54 inch from wax level to top target line.)

- 15:20 Transferred wax product from 27.10 reactor to 28.30 prep tank.
- 16:25 Completed wax transfer from 27.10 to 28.30.
- 16:45 Transferred H_2O/HC from 22.16 day tank to trailer. Starting level in 22.16 = 64.5 inches.

Ending level = 20"

density = 0.45 till 21".

then density = 0

- 17:10 Completed water/HC transfer from 22.16 to trailer. Ending level in 22.16 = 20 inches. 784 gallons transferred (total in #5390 is 1299.5 gallons).
- 17:15 Transferred wax product from 27.10 reactor to 28.30 prep tank.
- 18:30 Finished transferring wax from 27.10 to 28.30. Reactor temp has been stable all day.
- 20:00 Transfer light wax from 22.14 to 27.13. High level alarm keeps ringing.
- 20:35 Transfer wax for 27.10 reactor to 28.30 prep tank.

18 October 1996

- 02:00 Wind shifted. Cold front coming through. Heaters firing higher to maintain temperature.
- 02:45 Henry grabbed a liquid sample off 22.11.
- 12:30 Stopped data acquisition on Bailey window view to load a file that collects 5 data pts every 10 sec. to study response time. File name: XAVPTS.STP.
- 12:40 Restarted above file with FT3TAGS.STP (hourly data).
- 15:00 Transferred wax form 27.13 to wax trailer.

90.83% to 24.61% in 27.13

gal transferred.

18:30 Begin to backflush all filters.

Α

В

С

D

- 18:40 Needed to divert some utility oil to heat 28.30. Caused drop in reactor temp. took off cascade control till stabilized.
- 18:45 Downloaded data. This required stopping Bailey data acquisition.
- 19:13 Restarted Bailey data acquisition (134 tags every hour & 24 every hour).
- 19:45 Started back-flushing (28.30 ~280°F)
- 21:50 GC Dennis is showing bad compositions in just the last sequence (<1 hour). Rob is on his way in to troubleshoot. Need to exclude data from 2100 2400. It looks like D04 at about 2110 was the first shot to go bad.
- 21:15 Rob has fixed Dennis. His first comparison against G02 showed Dennis about 1% high on H2 and CO, but he will wait until morning to re-calibrate.

19 October 1996

- 02:15 AJ grabbed a liquid sample off 22.11.
- 08:00 Back-flushing filters.
- 10:05 Stopped Bailey data collection to run test.
- 10:08 Started hourly Bailey data collection and 10 sec data.
- 10:13 10 min test began

45.4 from 65.4 on TIC-293

440.6°F starting T avg on Rx

190-3B unit. Temp = 440° F

- 10:18 Tavg on Rx = 438.58.
- 10:20 Test stopped

Tavg = 437.16°F

190 - 3B = 436.2°F

11:45 Bailey data acquisition resumed.

B152AVG1.DAT

B152INS1.DAT

- 10:30 Changing cond. to R15.2.
- 12:30 Transferred 22.16 HC/H₂O to trailer.

22.16 starting level = 90.75"

density = 2.0

22.16 level @ density change = 31"

density = +0.65

22.16 ending level = 20"

- 12:55 Switching over to HP H₂, attempt 1
- 13:00 A_2710_AVG_TEMP dropped from 460.04 to ~443.
- 13:07 Went back to LP H2
- 13:45 Trying HP H₂ again
- 14:40 NP N₂ caused big deviation high 482°F low 420°F Trying to line out again.
- 16:30 Going to backflush filters with N₂

All DP's are high 80-100 psi

Wax level high. 2.4 filtrate flow

19:30 Still trying to clear filters.

Pressurize filters to ~900-1000 psi and then pop open ball valves.

- 20:00 Mildly successful. Keep running at this condition ~410°F till level drops then raise T to around 440°F.
- 20:30 Filters not filtering very well.

Keep T down ~390

Lower level as much as can. Wait till tomorrow.

20 October 1996

- 00:25 Henry grabbed a slurry sample off 10.62.
- 08:00 Turned off Bailey collection to install mouse and reconverted files B151AVG5.DAT ý B151AVG9.DAT
- 08:17 Re-started Bailey data collection.
- 09:40 Back-flushed filters with N₂ and now trying flow through them again.
- 09:50 dP increasing in D = 110

in C = 95

@ avg RxT = 395.9

in B = 81.6

in A = 66.23

Stopped test ý Mtg.

- 12:15 Attempt made to back contents into reactor and run clean wax through filters. Couldn't re-establish flow.
- 13:00 22.16 transfer complete

22.16 starting level = 50.5"

22.16 ending level = 20"

density varied ~0.6

Volume transferred 537.5 gal

Ending amt. in trailer = 3083.75

14:00 Dropping H₂ and CO feeds.

15:00 Slurry sample taken.

21 October 1996

16:45 Preparing for a shut-down test.

Rxt avg temp = 396°F

Rxt pressure = 702 psig

(187A) Rxt feed --- = 56.85 MSCFH SP4MW = 12.3

(701A) 22.10 Outlet -- = 45.72 MSCFH SP2MW = 12.38

22.10 outlet: $H_2 = 60.14 - 1$.

(D02) $N_2 = 6.18 - 1$.

 $\overline{CO} = 33.36 - 1$

CH4 = 0.27 01.

Rxt feed --- $H_2 = 60.86$

 $N_2 = 5.95$

 $C\overline{O} = 33.14$

CH4 = 0.08

Shut-down Test

17:14 Shut down gas flow

7.52 total test time

PDI 1778 ~39.7 ý~51 psi

PDI 1779 ~44 ý 56 psi

18:30 Clean liquid in filters

Product withdrawal Back valves shut flush PDI w/10.54 1772 22.16 22.31 22.28 21.21 21.21 73 18.85 18.97 19.08 18.28 18.29 74 53.67 21.78 -4.01 -6.76 -15 -0.75 28.74 10.21 75 33.09 7.21 0.7 -13 39.40 70.64 -18 76 79.15 42.85 15.66 18.17 47.68 26.13 -20 77 88.01 51.92 28.63 58.08 LV-203 7 -1. 8.1y 5.0y 4.9y 11y 0.74 0.76 DI-1761 0.73 0.73 0.74 DI-1768 0.76 0.77 0.77 0.75 0.75 FI-1768 162 163 164 162 162 FI-1761 1.38 0.89 -0.11 1.39 1.8

	19:15	19:30	19:40
PDI	Backflush w/ 10.54		

1772		20.37	20.32
73		18.5	18.43
74	-19.8	01.71	3
75	-13.47	9.51	10.86
76	-19.08	17.32	18.77
77	-24.13	27.28	28.74
LV-203		7%	10%
DI-1761		.76	.75
DI-1768		.73	.73
FI - 1768		157.03	157.05
FI - 1761		1.67	1.89

22 October 1996

- 07:30 Transferred 28.30 to trailer. Ending amt in trailer = 2438 gal. Amt added was 201 gal. 09.38 22.62 contents transferred to 27.10.
- 12:00 Drained 27.10 contents into drums.

Drum #	Empty Wt (lb)	Full Wt (lb)	<u>Δ (lb)</u>
1	45	461	416
2	46	316.5	270.5
3	46	321	275
4	46	319	273
5	46	293	247
6	46	310	264
7	46	418	372
8	46	355	309
9	46	346	350
10	46	103	57

12:15 Nextgen stopped

13:00 Filled 27.10/22.62 for flush

15:00 Draining 22.62 filters

16:00 Draining 27.10

23 October 1996

Drum height (total = 33 1/4" (from inside of cap bottom edge to ground)

Drum Diameter = 23"

Empty height within each drum:

1 7 - 3 -	-	
Drum_#	Height (in)	
1	5 1/8	5.125
2	15	50.0
3	13 7/8	13.879
4	14 1/2	14.5
5	16 1/2	16.25
6	15 1/2	15.5
7	7 3/4	7.75
8	12 1/4	12.25
9	12 3/4	12.75
10	29 1/2	29.5

Height of Catalyst in Heavy Fischer-Tropsch Wax:

Drum #	Height (in)
1	28.125
2	16.75
3	19.375
4	18.75
5	17.0
6	17.75
7	25.5
8	21.0
9	20.5
10	3.75

Density Calcs

Drum	Mass(g)	Vol (cm3)	Density (g/cc)
1	188693.44	191486.06	0.99
2	122696.1	114040.59	1.08
3	124737.25	131912.62	0.95
4	123830.07	127657.38	0.97
5	112036.73	115742.69	0.97
6	119747.76	120848.98	0.99
7	168735.48	173614.03	0.97
8	140159.31	142976.26	0.98
9	136077	139572.06	0.97
10	25854.63	25531.48	1.01

Average density = 0.99 g/cm^3

26 October 1996

Seal oil was leaking out of 10.62 pump

Maintenance done on 10.62 from ~1500 to ~2200

Note: Changed back clock early for end of daylight savings.

22:15: 10.62 Running and operating procedure for hot oil filter flush and clean oil flux test commenced. Began heating up filter loop at a rate of 87°F/hr.

23.25 Adding new oil to maintain level in 10.62. This is done frequently and seems to keep temp rate at ~78°F/hr.

27 October 1996

- 00:15 Raised reactor pressure to ~117 psig in order to maintain seal oil level.
- 00:35 TIC-293 opened 100% and temp ramp at 75°F/hr and dropping slowly.
- 00:45 Reactor P at 120 psig and level is holding steady.
- 03:00 Oil transferred from degasser to 28.30 prep tank LI-1765 reduced to 6.72%.
- 03:45 Back flush of filters began.
- 06:25 Reactor temperature peaked at 487.48 and temperature started cooling down to 300°F.
- 08:30 Flushing 22.62 logs.
- 08:49 Filter loop at 300°F, system flushed.

$$FI - 1761 = -6.36$$

$$PDI - 1775 = -0.65$$

$$PDI - 1776 = -2.26$$

$$PDI - 1777 = -2.96$$

$$FI - 1761 = 0.92$$

- C filter 09:00 PDI-1776 = 23.63 FI - 1761 = 1.0609:03 PDI - 1776 = 34.29 C filter FI - 1761 = 1.0409:04 PDI - 1775 = 15.47 B filter FI - 1761 = 1.05 09:08 PDI - 1775 = 22.67 B filter FI - 1761 = 0.9909:10 PDI - 1775 = 24.73 B filter FI - 1761 = 0.9809:12 PDI-1775 = 26.43/27.18 B filter FI - 1761 = 0.98A filter (flow 1) 09:15 PDI - 1774 = 1.25 FI - 1761 = 1.07 A filter (flow 1) 09:18 PDI - 1774 = 4.15 FI - 1761 = 1.04 09:23 PDI - 1774 = 7.6 A filter (flow 1) FI - 1761 = 1.03 09:26 PDI - 1774 = 19.17 A filter (flow 2) FI - 1761 = 1.98 09:27 PDI - 1774 = 27.23 A filter (flow 2) FI - 1761 = 1.90 009:32 PDI - 1774 = 43.3A filter (flow 2) FI - 1761 = 1.68
- 5 November 1996

Wt of impeller (10.62)After the run = 1403.32 g