

10:36 GC Dennis bback on-line.

11:52 HYCO plantsts went down next door, so we lost CO. Shut off all flow to 27.20. CO and H2 t blocked in.

Nuke slump test. 4/40 wt% slurry and 6 vol% gas holdup. Level at 322".

12:40 Cooling downn plant at 50 F/hr, down to 300 F. Lining up N2 plus a little LP H2 (to maintain a a reducing atmosphere) to feed to 27.20 once-thru at about 10,000 SCFH. Level up to 334".

13:40 Lost CW fromm HYCO plant, and 01.10 keeps tripping on high discharge temperature. Can't r run compressors. We will blow down plant to 100 psig and run in reductionn mode under house line pressure.

15:00 Established NN2 flow at about 8500 SCFH. D03 is recalibrated for reduction gas and shshooting SP-13B continuously.

15:28 Feed composition: (D03) 2.1% H2
(looks good) 0.5% CO
97.8% NN2
0.0% CCO2

15:50 CO is back. (CW pumps should be back on-line by 17:00, so holding temperature here atat about 390 F. Maintenance preparing to pressure test new HP H2 tie-in line.

17:30 Reduction cirircuit to reactor shut off. Pressure test complete on HP H2 line. Will start plantt on GC Gary alone until GC Dennis can be recalibrated for regular op's. Reacttor level at 338" with no flow.

17:51 Syngas re-intatroduced to plant once-thru at about 20,000 SCFH.

17:55 Starting heat t up. P = 139 psig. Level at 369".

18:20 27.20 Avg. TT = 424 F, P = 722 psig. Level at 372". GC Gary is on-line.

18:30 Starting recycle flow from 01.20.

18:40 Compressor t trip. High discharge temperature.

19:00 Recycle floww re-established.

19:15 GC Dennis bback on-line.

20:05 Flow crossing thru 100,000 SCFH. Reactor level up to 525", so holding flow for a while. Both GC's reading consistent feed compositions.

38.6% H₂ ?
(D04 - 19:41) 50.9.9% CO
1.3% N₂ ?
9.2% CO₂

20:30 22.16 day tank transfer to trailer 103" to 19 3/4" (1467.5 gallons).

21:30 Increasing recycle flow again.

3/22/94 01:25 27.20 has lined out at steady operating conditions. Level in auto at 480".
Tuesday T = 481 F, P = 750 psig, F = 145,000 SCFH. MeOH in product at 9.6%!

04:35 Line pressure on HP H₂ has been rising steadily. Now up to 784 psig.

04:45 Re-introduced CO₂ to the fresh feed since last 2 SP4 shots have been 10.8% and 9.7%.

05:32 GC Gary 3A analysis lost its MeOH peak (plant has been steady). GC Gary analysis on feed showed 11.6% CO₂ - OK.

08:48 22.16 day tank transfer to trailer 121" to 19 7/8" (1782.5 gallons).

10:15 Matt took a liquid sample from 22.11. Slurry sample also taken.

10:30 GC Gary back on-line. GC Dennis taken off-line.

10:45 Nuke scan. Calc's show 45 wt% slurry and 47 vol% gas holdup.

12:18 GC Dennis is back on-line.

14:40 Regression of liquid logs indicates that liquid product rate was 119.5 gal/hr before the restart and 143.8 gal/hr after.

14:45 Nuke scan. Calc's show 45 wt% slurry and 47 vol% gas holdup.

16:15 Liquid logs indicate product accumulation rate equivalent to 14:40 calc. Still no oil loss.

17:00 Took a liquid product sample from 22.11. It looks completely clear.

18:55 22.16 day tank transfer to trailer 101 1/4" to 20" (1432.25 gallons).

19:35 Nuke scan. CCalc's show 45 wt% slurry and 47 vol% gas holdup.

20:55 Lined up 27.1.14 for AF-R9.2. Took a liquid sample from 22.11. It was raining at the time, so water content could be artificially high (?). Again, sample was completely clear.

21:04 Started changing conditions to AF-R9.2

3/23/94 00:05 Still helping k Henry line-out on compositions.

Wednesday

00:35 D03 shows 16.4% MeOH in reactor effluent. Wow!

01:30 Still tweaking H2 in reactor feed.

02:20 GC Gary is consistently reading lower (up to 2.5%) than GC Dennis on MeOH in reactor effluent.

03:10 Last 2 GC shots have been good on feed composition.

04:00 Took a liquid product sample from 22.11.

05:05 Nuke scan. CCalc's show 39 wt% slurry and 33 vol% gas holdup.

06:45 22.16 day tank transfer to trailer 116" to 20" (1692.2 gallons).

07:54 GC Dennis is off-line for calibration.

Sample points 6 and 8 have had little or no flow for a while. Problem corrected. Also, N2 in fresh feed is 0% often, and since recycle (SP8) is unreliable, it is difficult to close a mass balance. Adding N2 to the 01.10 feed streams. Previously we relied on N2 in the CO stream to be sufficient, but CO stream is now coming from the PSA's and N2 may be different now.

09:30 GC standards have not yet been switched to Kingsport gas standard. We need to let Deann know when we change conditions. Conclusion: data on AF-R9.2 is probably questionable to this point.

10:35 GC Dennis back on-line. GC Gary goes off-line for calibration.

12:15 GC's are back on-line.

12:25 Backed out the fresh N2 in the feed. Latest SP4 reading was 6%.

15:00 GC's differing by 3% on effluent MeOH concentration. Dean says both are very accurate in the 6% range (calibration level) and that the error will be

significant at the concentrations we're running. It is also not unusual to be totalizing >100%.

16:45 HP H2 line pressure dropped from 760 to 680 psig over last 1 3/4 hours. Feed flows and compositions starting to swing some.

16:50 AJ took a liquid sample from 22.11 (both sample bomb and glass jar).

17:11 D03 shows 18.2.2% MeOH (see entry at 15:00 above).

17:30 Nuke scan. Calal's show 39 wt% slurry and 34 vol% gas holdup.

18:00 Discovered reactor pressure was moved up to 750 psig about 1 3/4 hours ago. Matt returning setpoint to 735 psig.

18:42 22.16 day tank transfer to trailer 11 1/2" to 20" (1612.5 gallons).

22:00 Henry took a liquid sample from 22.11 (both sample bomb and glass jar).

22:25 Dean looked at the GC's. Gary and Dennis match pretty well on H2 and CO2 but differ on CO and N2. His comment was that CO and N2 come out very close and GC Dennis is getting poorer separation between the peaks. He's more inclined to believe GCC Gary.

22:35 Dean confirmed that GC Dennis is integrating weirdly. He will try to fix it.

Feed composition has been drifting slightly for some time, probably since the HP H2 line pressure problem this afternoon. Henry is making a move to increase the H2 and decrease the CO2.

22:50 Dean thinks he's fixed GC Dennis. First set of points thru looked good.

3/24/94 04:00 Took a liquid sample from 22.11 (both sample bomb and glass jar).

Thursday

06:00 22.16 day tank transfer to trailer (1612.8 gallons).

08:30 Matt took a liquid sample from 22.11 (both sample bomb and glass jar).

08:55 Data period AFF-R9.2 ends here. 21.11 testing period begins here.

09:10 Moving to Test A target: Kingsport gas, T = 482 F, P = 750 psig, SV = 6500 (F = 140,400 SCCFH).

10:25 Start 21.11 Test A.

16:27 Moving to Test B target: Kingsport gas, T = 482 F, P = 750 psig, SV = 10,000 (F = 215,8000 SCFH).

18:35 Moving to Test E target: Kingsport gas, T = 482 F, P = 850 psig, SV = 9000 (F = 194,1000 SCFH).

MeOH samples are very cloudy. After settling a few hours, there are oil bubbles in them. Decide to run here until 3-4 AM to let things settle out.

22:39 SD-1 plant trip. High discharge temperature on 01.20 (180 F). Decide to stay shutdown. Start cooling plant.

3/25/94 10:00 Level at 270" in reactor with no flow. T = ~150 F. Draining slurry.
Friday

Load Drakeol oil to 28.30 prep tank:

Drum #1 = 378 lbs
Drum #2 = 395 lbs
Drum #3 = 384 lbs
Drum #4 = 311 lbs
Drum #5 = 191 lbs
Total = 1659 lbs

16:25 Start loading IBOH catalyst to prep tank (152 F).

Drum #1 = 272 lbs
Drum #2 = 273 lbs
Drum #3 = 273 lbs
Drum #4 = 273 lbs
Drum #5 = 15 lbs
Total = 1106 lbs

17:50 Catalyst loading complete. Reactor currently being flushed with oil under N₂ flow at about 2000 F.

3/26/94 07:40 Starting to drain flush oil from reactor.
Saturday

11:10 Pressure transfer prep tank contents to reactor. Level is 243". Pressure transfer 2 more times. No noticeable change in level.

12:10 Inspect prep tank. Not as clean as MeOH transfer. There is a layer of catalyst on the walls near where liquid level was. Estimate 5-10 lbs of catalyst. Add 283 lbs of flush oil and agitate. Still a crusty layer where the initial slurry level was.

12:25 Pressure transfer flush oil to reactor. Level ~290".

12:40 Withdraw a slurry sample. Lining up reduction gas composition.

13:24 Syngas to the reactor.

13:32 T = 183 F, P = 60 psig, F = 14,500 SCFH. Level at 369".

14:00 Starting composition: (D06) 1.29% H₂
1.98% CO
95.68% N₂
0.55% CO₂

Starting heatup at 10 F/hr target rate.

14:20 GC Gary has not been totalizing well. Dean says there is liquid in the SP13b line (he sees it in the rotameters), and there was liquid in the K.O. pots. They are trying to correct problems with the electric heaters. GC Dennis is OK.

15:15 Increasing H₂ in the feed slightly. Last few analyses show 1.1%.

15:40 H₂ was a little too high (1.7%).

16:12 GC Gary is going off-line for calibration. Level at 377".

16:58 GC Gary back on-line. During calibration N₂ was reading 2% low. There may be some oil in the line which may take a while to purge out.

19:45 Level at 389". H₂ and CO uptake is underway. Earlier GC note still holds. GC Gary working well on SP6 but not on SP13b. GC Dennis still working well. Believe GC Dennis.

20:06 GC Gary seems to be improving.

20:20 Uptake appears to be increasing. Uptake vs. time matches MeOH reduction almost identically.

23:55 Level at 384".

3/27/94 02:08 Level at 363".

Sunday

03:05 Bringing the 02.622 reduction heaters online to help warm up the reduction feed gas during the temperature ramp.

04:00 Tried to increase the total flow using N₂, since it had dropped to 13,400. However, flare was flickering on one side, so we decided to not risk it.

07:30 Level at 346". Flow had dropped back to about 13,000 SCFH. Matt opened up on the valve throttled at the reactor inlet. Flow is back up to 14,000 SCFH.

11:31 Began 4 hour hold period. Level at 345". T = 393 F. Put nuke in auto control at 352".

12:55 Setpoint on nuke is now 362".

13:30 Flow at 14,800 SCFH. Reduced to approximately 14,000 SCFH.

14:35 Setpoint on nuke is now 376.5".

14:50 Oil added to 27.7.14 from 6 nuts to 16 nuts.

15:35 Heating up from 394.6 F at 18 F/hr.

20:10 Flow out of the reactor. Level at 295" with no gas. SP13 valved out; 21.11 demister line open. SBP3A is now valved in.

20:45 Henry has been trying for a slurry sample for approximately 1 hour. The sample tap (by the DP) is plugged and with 40 psig on it, he can't get a sample. We're giving up.

22:05 Syngas to the reactor. T = 451 F, P = 194 psig, F = 17,250. Level at 315". GC reading on fresh feed: 28% H₂, 67% CO, 6% CO₂.

22:33 T = 450 F, P = 500 psig, Level at 326".

22:45 T = 450 F, P = 620 psig.

GC Reading (D04):	20.04% H ₂
51.92% CO	19.57% N ₂
5.57% CO ₂	

Opening PV-201 to help purge some N₂.

22:55 Recycle flow introduced. Level at 328". T = 452 F, P = 700 psig.

23:05 T = 453 F, P = 753 psig. MeOH in effluent = 4.78% (G03 at 22:53).

23:27 T = 464 F, P = 751 psig, F = 73,990 SCFH.

23:56 Showing level in 22.10. T = 489 F, P = 744 psig.

3/28/94 00:05 Feed composition (SP4): 20.70% H₂

Monday 60.40% CO 11.85% N2
 3.61% CO2

00:25 T = 509 F, P = 7554 psig, F = 95,900 SCFH.

01:23 CO2 out of the fresh feed (CO was temporarily shut off).

01:30 T = 541 F, P = 752 psig. Level at 450". TIC-1260 (21.11 outlet) = 265 F.

01:53 Nuke level moved to 480".

02:15 27.20 level at 480". Going to cascade control. T = 565 F.

02:25 H2/CO ratio is coming up as N2 gets backed out. Increased CO from 10.0 to 10.3 MSCFH.

(D04) 32.16% H2
 58.81% CO
 5.57% N2
 3.26% CO2

03:05 Pretty well lined out on temperature. Average temp is 568 F, but control is based on TI-1232 which is right at 572 F.

04:30 Nuke scan. Calc's show 39 wt% slurry and 37 vol% gas holdup.
T = 568 F, P = 754 psig, F = 96,400 SCFH.

06:35 Still adding in CO as the N2 concentration drops.

(D04) 31.70% H2
 63.26% CO
 2.23% N2
 3.06% CO2

08:40 D04 composition looking good: 30.62% H2
 65.48% CO
 1.40% N2
 2.92% CO2

Flow slipping a little to a about 95,500 SCFH.

10:45 GC Dennis off-line for calibration. GC Gary is still on-line.

14:27 Pinhole leak in thermocouple weld. Must cool and depressurize the reactor to fix it. Started cooling at 7 FF/5 min.

17:25 T = 353 F, P = 2 263 psig.

18:30 T = 344 F, P = 2 2 psig. Thermocouple connection (TI-1235) back-welded. Leak fixed. Blending g syngas.

18:45 Syngas composition OK. Heating up.

19:07 T = 383 F, P = 5 572 psig, F = 23,000 SCFH. Back end on. Starting recycle flow.

22:20 Reactor temperature out of control. We were up to 598 F. The fin fan bypass was 50% open and HIC-1666 was 93%. We opened the HIC-166 valves to 85%, and the temperatures finally started to come back.

23:11 Noticed that none of the GC's have updated since 21:38, which is where we had a FROMHP.BAD file that was zero blocks big. All the GC's are analyzing in the lab and printing out the analyses. HP_GC_DRIVER is "hibernating", which is good. Talked with Rob and he doesn't understand why all GC's would be having this problem.

DEC ng_stop and ng_start again.

3/29/94 00:30 Rob was in to look at the GC's. There are no errors on his end, and he Tuesday doesn't want to reboot it blindly for fear of losing all his methods. So we go all night with no GC values in to the DEC. Checked printouts in lab to line-out on composition.

03:46 Took out fresh CCO₂ (was 200 SCFH on set point).

06:12 Nuke scan. Calc's show 41 wt% slurry and 42 vol% gas holdup.
T = 573 F, P = 752 psig, F = 97,000 SCFH.

07:45 10.80 pump sealal leaking. Blocking in back-end to fix the pump, while maintaining flow to reactor. Dropping temperature by 50 F in reactor.

Still getting no info from HP computer. We've given up on reconstructing the overnight data. Rob rebooted the HP and we've restopped and restarted nextgen and moved the clocks forward on both computers. (Daylight savings time is this weekend). So 9:59 became 10:59 on the DEC. Also when we first went to work on the DEC, the trend data collection routine had failed around 4:30 in the morning. We cannot access data for that time until probably 11:00 AM on March 29 when it was restarted.

12:25 (Real time) Cloudy MeOH sample from 22.11 (with drops of oil).

16:30 Back-end pressured up. 10.80 pump reinstalled and started. Heating up reactor at 60 F/hr.

17:36(DEC time) Reactor at 572 F (average) and 750 psig. Reactor PIC on cascade control. Draining liquid from the 21.11.

18:02 F = 93,800. Analyses from GC's:

(D04)29.98% H2	(D03)23.19% H2
66.16% CO	66.19% CO
1.00% N2	1.18% N2
2.69% CO2	6.03% CO2
2.28% MeOH	

Matt drained 10 gallons of liquid from the 21.11. There was a large second phase (definitely oils). Raised setpoint of TIC-1260 (21.11 outlet) to 270 F. When reactor heated up, the 21.11 bypass was not in control and the process value was 275 F even though the setpoint was 245 F. Leaving the setpoint at 270 F for now to try and get rid of two phases.

Airco loading the CO2 tank to full.

19:30 GC Gary 3A is disconnected for a while to clean out the rotameter lines with N2. Last bad file from the HHP received was at 19:53.

19:35 Reduce reactor setpoint to 574 F to meet average reactor target temperature of 572 F.

20:00 Leaks at trailer during 22.16 transfer. Called in Matlack. Plant running steady. T = 573 F, P = 748 psig, F = 95,390 SCFH. Compositions: IBOH = 0.17 - 0.18%, MeOH = 2.7 - 2.9%.

21:30 Plant running very steady.

23:20 22.16 day tank transfer to trailer 116" to 20" (1692 gallons). (Note: Bharat's logs are in real time for time period of 20:00 to 23:20. Need to add 1 hour for DEC time.) VEES is logging times as DEC-time from here on out. Everyone should follow suit.

3/30/94 03:00 Plant still very steady!

Wednesday

05:25 Henry grabbed a liquid sample from 22.11. Looks good.

07:40 Nuke scan. Calc's show 42 wt% slurry and 43 vol% gas holdup. T = 573 F, P = 753 psig, F = 96,000 SCFH.

08:48 G03 lost its H2 peak. DEC shows a bad file message from the HP at 08:54. Actual value from lab printout is 24.40. Check into deleting this point from mass balance.

09:30 FI-7291 calculation corrected in the Bailey. G/C forgot to add 14.7 to the pressure and 460 F to the temperature.

11:10 AJ grabbed a liquid sample off the 22.11. Still looks good.

13:30 Changing conditions to AF-R10.2.

AF-R10.1 data period: from 3/29/94 at 21:00 to 3/30/94 at 13:00.

14:00 AF-R10.2 condition reached.

15:45 Rob advised not to use any information from GC Gary between 12:30 and 14:30 DEC-time. Will need to correct for this when doing mass balance for AF-R10.1.

19:00 Bharat and Kerriri are investigating how to delete 08:54 bad data point from averaged data. Data point to be deleted is G03001. Last good data point for G03001 was at 07:46. Bad shoot was actually at 08:48. Next good shot was at 09:51.

21:00 Noticed DEC is not picking up some HP data. R02 is missing the methanol peak. Closed picture and signal overview and restarted. This did not help the situation. Chris DaCosta will try to work on modem.

22:15 Chris has found the problem. Butane is being sent in component #10 slot which overwrites the methanol #10 slot.

22:20 We continue to lower 10.80 MEOH flow to increase CO2 in the reactor feed to 3%.

23:15 Rob came in and deleted n-butane (#10 slot) and pentane (#11 slot) from GC's Rocco and Bharat.

3/31/94 00:00 Looks like CO2 in the feed is lined out (2.6%).

Thursday

03:40 GC's have not updated since shortly after 02:00. Message screen shows only one bad file received from HP at 02:27. HP printer still working in the lab. Closed signal overview and pictures and did a restart. Signal overview still shows flat compositions. FROMHP.BAD;81.15 (most recent) is of size 0 blocks (see 3/28/94 23:11).

04:10 Looks like exactly the same problem as the other night - solution was to reboot the HP. Called Rob. He's coming in.

04:45 Rob is shutting down GC's to reboot.

05:30 Looks like the GCC problem is fixed. DEC is starting to get new (.old) files from the HP. The new concentrations coming in are very similar to the old ones that were stuck from 3-4 hours ago. Last point went non-zero just before 07:00. Will exclude 02:00 -07:00 from data. .

05:50 Henry grabbed a liquid sample off of 22.11.

07:20 Nuke scan. Calc's show 38 wt% slurry and 36 vol% gas holdup.
T = 572 F, P = 752 psig, F = 57,680 SCFH.

09:15 22.16 day tank transfer to trailer 102 3/4" to 20" (1458.5 gallons).

10:45 Product liquid analysis is showing about 8% "others". Rich Underwood advises that it is reasonable to assume they average to C6-alcohols. Will adjust the hexanol peak.

14:20 Matt grabbed a liquid product sample off of 22.11.

15:50 AJ grabbed another liquid sample from 22.11.

17:04 Changing conditions to AF-R10.3.

AF-R10.2 data period: from 3/31/94 at 00:00 to 3/31/94 at 17:00, excluding 02:00 - 07:00 when GC's were not working correctly.

4/1/94 02:05 Plant looks steady. Feed composition is still drifting into place, though.
Friday

04:05 Still very steady. Data period begins 04:00.

07:04 Nuke scan. Calc's show 45 wt% slurry and 51 vol% gas holdup.
T = 572 F, P = 750 psig, F = 156,570 SCFH.

07:45 AJ grabbed a liquid sample off of 22.11.

10:45 22.16 day tank transfer to trailer 95 5/8" to 20" (1332.6 gallons).

14:15 Took a liquid sample from 22.11.

16:00 AF-R10.3 data period: from 4/1/94 at 04:00 to 4/1/94 at 16:00.

16:05 GC's Rocco, Bharat, and Gary will be taken off line as the plant is down.

16:25 Shutdown test. The liquid dropped from 471" to 263" in reactor.

Method 1: Assuming 0% holdup in liquid:

$$\text{Gas Holdup} = (471 - 2263)/471 * 100 = 44.2 \%$$

Method 2: Assuming calculated 6% holdup in liquid:

$$\text{Gas Holdup} = (471 - 2263 * 0.94)/471 * 100 = 47.5 \%$$

Method 3: Holdup calculated by regular nuke scan = 49.5 %

16:40 Changing conditions to AF-R10.4. 01.30 compressor is on. Increasing pressure to 1300 psig.

16:50 Reactor average temperature overshoot to 583 F, coming down now.

17:10 Reactor pressure is now 1317 psig. Another reactor temperature rise observed - peaked at 590 F.

18:10 Put reactor on level control at 480"

4/2/94 00:00 Plant seems steady. GC Gary is totalizing very poorly on SP3 and not Saturday particularly well on SFP4.

02:20 22.16 day tank transfer to trailer 103 3/4" to 20" (1475.75 gallons).

03:45 Henry grabbed a liquid sample off 22.11

04:00 Plant still steady. CO2 consumption at 3750 lb/hr.

05:01 Nuke scan. Calc's show 42 wt% slurry and 44 vol% gas holdup.
T = 573 F, P = 1302 psig, F = 157,000 SCFH.

06:05 Waiting desperately for a CO2 shipment (more than 2 hours late now).

07:15 CO2 is gone!!! Closing steam to reboiler, isolating back-end, shutting down 10.80 pump, dropping temperature in reactor at 50F/hr.

AF-R10.4 data period: from 4/1/94 at 21:00 to 4/2/94 at 07:00.

07:35 D04 at 07:20 reads 4.01% CO2 in feed. Temperature down to 554 F.

08:35 D04 at 08:22 reads 3.56 % CO2 in feed. Temperature down to 500 F.

08:40 CO₂ load finally arrived! Starting to bring back-end up again and move to data point AF-R10.5.

11:00 27.20 and 21.11 own level control.

11:45 Blocked in SP15 at root valve. Rob taking GC Gary off-line to fix it.

14:20 22.16 day tank transfer to trailer 123" to 45 1/2" (1366 gallons). During transfer the trailer overfilled, spilling liquid out of trailer and out of flare.

16:45 GC Gary back on line. Seems to be better now.

22.25 CO has been high (67%) in the reactor feed. Have found it difficult to get any attention because operators are busy with more important work. Initial, small moves had little effect, as expected. Finally convinced operators to make larger and faster moves. CO is 67% and dropping right now.

Because of overflow on the trailer, some liquid from the full trailer was transferred to empty trailer (maximum estimate is 500 gallons). We need to keep a running total of trailer inventory. Trailer capacity is 7000 gallons. A rule of thumb would be no more than 4 transfers per trailer.

22:40 22.16 day tank transfer to trailer (second) 102 3/4" to 41 1/2" (1097.5 gallons). Emtec is here doing spill cleanup.

4/3/94 00:40 22.16 day tank transfer to trailer 54 1/4" to 20" (603.5 gallons).
Sunday

03:30 Real time = Dec time! Finally!! Plant is finally steady, although CO₂ is at its upper limit in the feed (about 3%). CO₂ consumption in back-end is about 3100 lbs/hr with 35,000 lbs left in the tank. We should be okay until about 14:00 at this rate, but usage will increase if we start stripping more. Next delivery is scheduled for 10:00. R02 at 03:06 shows nothing but methane and ethane (i.e., we got part-2 but not part-1). Everything looks OK on the HP printout and no bad file messages. Previous point was OK too. Reactor temperature profile has been inverted since we started moving to this condition (more than 15 hours ago). About 580 F on bottom 2 thermocouples (1239 & 1238), 575 F on next 2 (1236 & 1235), then about 567 F thru the rest of the slurry.

04:00 R02 at 03:34 looks good again.

04:53 Nuke scan. Calc's show 41 wt% slurry and 42 vol% gas holdup.
T = 573 F, P = 1735 psig, F = 157,000 SCFH.

06:30 Matt grabbed a liquid product sample off of 22.11.

11:15 22.16 day tank transfer to trailer 117 7/8" to 20" (1723 gallons).

11:15 22.16 day tank transfer to trailer 117 7/8" to 20" (1723 gallons).

12:45 Matt took another liquid sample from 22.11.

14:25 Changing conditions to AF-R10.6.

AF-R10.5 data period: from 4/3/94 at 02:00 to 4/3/94 at 14:00.

17:50 27.14 almost empty. Added oil to go to 9 nuts (approximately 39 gallons).

21:00 Increasing CO, decreasing H₂.

22:00 Still trying to increase CO₂ in the feed.

4/4/94 01:30 Plant steady. Start data period at midnight.

Monday

02:40 22.16 day tank transfer to trailer 99 3/4" to 20" (1405.5 gallons).

04:50 Henry grabbed a liquid sample off of 22.11.

05:00 Nuke scan. Calc's show 35 wt% slurry and 28 vol% gas holdup.
T = 572 F, P = 1733 psig, F = 57,500 SCFH.

10:45 Matt caught a liquid sample from 22.11.

12:25 Changing conditions to AF-R10.7.

AF-R10.6 data period: from 4/4/94 at 00:00 to 4/4/94 at 12:00.

16:20 Lined out at 16:00, but GC Gary not on-line until now. Data period can start at 17:00.

22:30 22.16 day tank transfer to trailer 91 1/2" to 20" (1260 gallons).

4/5/94 00:35 Henry grabbed a liquid sample off of 22.11.

Tuesday

02:57 Nuke scan. Calc's show 39 wt% slurry and 37 vol% gas holdup.
T = 572 F, P = 1301 psig, F = 96,000 SCFH.
These results are the same as nuke scan at 21:40.

03:30 The plant is very steady - it must be near the end of the run!

05:30 Henry grabbed another liquid sample off 22.11.

08:45 GC's Dennis, Bharat, and Rocco are all off-line for repairs/calibration. Will take a few hours to bring them h back up.

09:30 Starting 02.63 to wwarm it up, and then they will be ready to start alcohol injection case, run AF-RR10.8.

10:00 We are hooked up p to rear compartment of alcohol injection trailer.

10:40 Started 10.95 pumppp. Having some problems.

11:15 GC Dennis is backk on-line. GC's Rocco and Bharat are still off-line.

13:35 Alcohols finally ggoing in the reactor feed.

14:50 MeOH concentratitio finally coming up in reactor feed sample (SP15).

16:05 22.16 day tank transnsfer (#1) to empty trailer 101" to 20" (1428 gallons).

19:30 Reduced 10.95 stroke rate from 100% to 95%. Methanol is currently 8.8% (target methanol = 7.9%)). Increased CO from 12.3 to 12.8.

20:45 CO increased to 133.5. 10.95 pump stroke decreased to 90%.

21:15 CO increased to 144.0.

22:40 Thunderstorm and d cold front passing through. Temperature dropped as low as 567 F in reactor. Still trying to line out the plant.

4/6/94 00:50 22.16 day tank t transfer (#2) to trailer 97" to 20" (1357 gallons).
Wednesday Henry moved 10.95 ppump back to 92% stroke from 90%.

01:30 Finally, composition is nearly lined out. Rob is putting GC Gary into its regular sequence.

02:05 Plant pretty wellll lined out, although still fighting reactor temperature because of the weather change.

03:00 G06 finally updated. All ports in GC Gary now reading new values. Temperature seems too have steadied out, too.

06:14 10.95 pump shuutdown while reconnecting hoses to facilitate the transfer from back compartment of trailer to the front.

06:16 10.95 back on-line. Stopped again for a few seconds; then back on again. Reactor temperature spiked briefly to 581 F. Pressure dropped to 1250 psig and overshot back to 13:320 psig.

06:20 10.95 stroke increased to 95%.

06:35 MeOH in G022 shot at 06:21 down to 5.95 because of upset. Excluding 06:10 to 07:00 from the data period.

07:15 Increased 10.95 stroke to 100%.

07:45 Matt grabbed a liquid sample off 22.11.

08:48 10.95 shutdown again for a minute or two. . . several times though. Temperature spiked to 587 F. Pressure dropped as low as 1240 psig.

09:40 22.16 day tank transfer (#3) to trailer 103" to 20" (1463 gallons).

12:30 Matt caught another liquid sample off of 22.11.

14:40 GC Dennis was off-line between 13:00 and 14:00. Excluding 13:40 to 15:40 for mass balance.

18:45 22.16 day tank transfer to middle compartment (1000 gallon capacity) of new trailer 93 1/8" to 46" (831 gallons).

20:30 Changing conditions to AF-R10.9.

AF-R10.8 data period: from 4/6/94 at 03:00 to 4/6/94 at 20:00.

While getting ready to change conditions: observed temperature rise in reactor (up to 587 F), and alcohol flow dropped to 0.4 gpm. Looks like the unit is changing conditions by itself! First thought we ran out of feed. Switched tank compartments, but flow is still approximately 0.4 gpm with 100% stroke. Looks like there may be another problem. The previous compartment still shows liquid level, too. Reaction temperature is back in control. Transmission fluid is low in 10.95.

23:20 Henry got 10.95 pump started again. Flow up to 0.65 - 0.70 gpm at 100% stroke.

4/7/94 03:00 10.95 still hanging in there at 0.6 - 0.7 gpm (100 % stroke). We are still Thursday adjusting feed composition (were very CO-rich), but it's slow going at 1 GC shot every half hour or so.

06:00 Finally getting closer to lining out. A few more tweaks, perhaps.

07:00 Plant is pretty well lined out, although CO₂ in the feed is a little low.

08:55 Matt grabbed a liquid product sample off of 22.11.

09:15 Matt added oil to 27.14 from 1 nut to 16 nuts (50 gallons).

11:30 Calculation of alcohol feed rates from compartment #3:

Starting level = 14688.5 gal

Final level = 13 gal

Alcohol consumed = 1455.5 gal over 11.5 hrs

Consumption rate = 22.11 gpm

Average FI-1221 reading during that period = 2.37 gpm. Therefore, the correction factor = $2.37/2.11 = 1.12$. To get 0.7 gpm for AF-R10.9, the reading on FI-1221 should be 0.778 gpm.

Improved performance of 10.95 pump. Now set at 0.78 gpm (0.7 gpm actual, per above calculation) on FI-1221.

11:35 22.16 day tank transfer to trailer 116" to 20" (1692 gallons).

15:10 Have been decreasing H₂ last 1.5 hours to desired level.

16:00 Data period for AF-R10.9 can begin now.

16:30 Matt grabbed a liquid sample off of 22.11.

19:45 22.16 day tank transfer to trailer 64 1/2" to 20" (784 gallons). Total liquid in trailer is now 6724 gallons (7000 gallon capacity). Trailer is now full.

23:30 Plant running smoothly. CO₂ in the feed is low, but no matter how much we crank down the back-end, it keeps drifting down.

4/8/94
Friday

01:20 Henry grabbed a liquid sample off of 22.11.

02:24 Nuke scan. Calc's show 39 wt% slurry and 38 vol% gas holdup.
T = 572 F, P = 1299 psig, F = 97,000 SCFH.

04:15 Henry grabbed another liquid sample off of 22.11.

05:35 05:00 mass balance indicated liquid injection rate has been 2/3 of target rate, despite calculation at 11:30 above. Also, level loss on trailer just increased for the first time, meaning that we just recently crossed thru 1/2 full point. Adjusted stroke on pump until FI-1221 read 1.26 gpm (50% increase over reading for duration of the

data period). Henry commented to me that he had decreased the stroke slightly several times during the night to maintain flow near the target. So, AF-R10.9 closes at 05:00. If we choose to run this condition for a while, it can be called AF-R10.9A.

09:30 Matt grabbed a liquid sample from 22.11.

11:30 IBOH is very low (0.06%) in effluent stream. It also looks like the DEC stopped taking data. Stopped and started NEXTGEN. Need to find the time during which the DEC was not taking data so that period can be excluded. In the meantime, the IBOH number appears to be correct. Switched GC Rocco to SP-3A, and Rocco confirms GC Bhararat's analysis. We will now calibrate both GC's. We seem to have major deactivation!!! Looking at raw GC data, it was gradual over the last 24 hours. Called Rich Underwood, but he could not explain the data.

12:45 22.16 day tank transfer to trailer 108 1/2" to 20" (1559 gallons).

14:20 Looks like we lost data on DEC after 06:00 today, so will not include 06:00 - 14:00 in any data processing.

15:00 Matt grabbed another liquid sample from 22.11.

17:00 Run No. 10.9.9A complete. Changing conditions to AF-R10.10.

19:00 Took a liquid sample from 22.11.

19:40 Henry grabbed another liquid sample off of 22.11.

20:15 27.14 is bypassed.

22:40 Henry grabbed another liquid sample.

23:15 Nuke scan. CCalc's show 41 wt% slurry and 42 vol% gas holdup.
T = 573 F, P = 752.2 psig, F = 95,600 SCFH.
Looks pretty much like AF-R10.1.

4/9/94
Saturday

00:40 Liquid production rate (from levels in product collection) is very small.

02:40 Henry grabbed another liquid sample off of 22.11.

04:40 Henry added oil into 27.14. 6 1/2 to 8 1/2 nuts (7 gallons). Oil rate appears to be about 2 gph.

05:12 Nuke scan. CCalc's show 41 wt% slurry and 42 vol% gas holdup.
T = 572 F, P = 747.7 psig, F = 96,600 SCFH.

05:40 Henry grabbed d one last liquid sample off 22.11. This was the first one that was cloudy, but the previous ones may just be leftover from the last data point.

08:15 Matt lined up carbonyl sample points 3A, 4, 15.

12:40 Fe carbonyl levels: SP4 0.006 ppm
SP3A 0.003 ppm
SP15 0.022 ppm

12:50 Started 01.30 compressor.

14:30 Matt grabbed 22.11 liquid sample.

14:45 Matt grabbed liquid sample from 21.11.

14:50 Matt grabbed sample off center compartment on trailer #SP1738 (alcohol injection source when catalyst died).

15:16 Fe carbonyl levels: SP4 0.006 ppm
SP3A 0.004 ppm
SP15 0.076 ppm

15:25 Dropping flow v from 96,000 to 58,000 SCFH.

17:52 Fe carbonyl levels: SP4 0.009 ppm
SP3A 0.006 ppm
SP15 0.069 ppm

18:20 Started shutdown. Ramping temperature down by 60 F/hr.

19:15 01.20 compressor shut down. Pressure dropped to 750 psig.

21:00 Flow switched t to go through 27.14. Bypass around 27.14 shut.

22:15 Blocked in back-end. Depressurizing process side and draining 21.80.

23:00 Stopped syngas fresh feed and brought in N₂ to purge the plant.