APPENDIX D

Fluid Dynamic (Methanol) Run Chronology

Fluid Dynamic/Methanol Run - June, 1995
Run Chronology

2 June 1995

09:00 Loaded 28.30 Prep Tank:

Drum	Full Wt. (lb)	Empty Wt. (lb)	Net Wt. (lb)	Subtotal (lb)
1	472	121	351	351
2	482	121	361	712
3	478	121	357	1069
4	483	120	363	1432
5	456	121	335	1767

TOTAL OIL ADDED = 1767 lb

19:00 Backed syngas out of plant at conclusion of carbonyl study.

3 June 1995

08:00 Baseline Catalyst Loading and Activation
Lot # 94/15730
Colli: 859 2859
Samples of catalyst were taken from each drum (1 through 4).

Drum	Full Wt. (lb)	Empty Wt. (lb)	Net Wt. (lb)	Subtotal (lb)
1	510	128	382	382
2	512	126	386	768
3	510	128	384	1152
4			27	1179

TOTAL CATALYST ADDED = 1179 lbs

11:55 Transferred slurry from prep tank to 27.20 reactor.

12:26 Transferred 284 lb. flush oil to prep tank and flushed residual catalyst to 27.20 reactor.

14:30 Started reduction gas flow.

15:44 Started heat - up.

17:30 Stopped pump back of oil from 27.14 to 21.11.

22:20 27.20 level down from 390" to 371"; however, catalyst concentration increased only to 40%.

02:25 Reactor level at 350". Started pumping oil back from 21.11 to 27.20 to raise reactor level.

05:25 Backed down on flow rates per test authorization.

08:00 Discovered manual block valve was shut on oil make-up to 27.20. Haven't pumped backed any oil all night. Brought level up to 360".

09:15 Transferred oil from 27.14 to 21.11.

4 June 1995

10:12 Reduction complete. Swinging N2 to cool down reactor to 420°F. Liquid level of reactor at 290 (w/o flow).

11:45 Syngas once thru on to the reactor
CO = 9300 SCFH
H2 = 9400 SCFH
CO2 = 1500 SCFH
Average reactor temperature 431°F
Reactor pressure = 171 psig.

12:15 Started oil back from 21.11 to 27.20.

15:25 Reactor feed close to target. 27.20 placed on auto liquid control.

18:00 GC/DEC communication problems. Communications re-established approximately 21:30.

22:00 Rob restarted the Dennis GC because it was out of sequence (D02 was coming across as D04).

5 June 1995

00:50 Day tank transfer from 102" to 20". 1445 gals. into trailer #7486.

01:15 Bharat GC lost MeOH peak on DEC. Rocco GS still shows MeOH peak.

11:50 Day tank transfer from 121.5" to 46". 1330 gals. into trailer #7486.

16:15 Day tank transfer from 83" to 20". 1110 gals. into trailer #7486.

6 June 1995

01:30 Day tank transfer from 103.5" to 20". 1472 gals. into trailer #7486.

8:50 Day tank transfer from 54" to 20". 599 gals. into trailer #7486.

12:10 Ended condition AF-R13.1. Started AF-R13.2.

19:05 Total reactor feed flow was 89,000 SCFH. Target 80,937 SCFH. Reduced recycle.

7 June 1995

00:20 Day tank transfer from 115" to 20". 1674 gals. into trailer #7454.

01:00 Plant lined out at AF-R13.2 conditions.

09:30 Switched G02 to SP6.

11:00 Feed composition steady.

12:15 Day tank transfer from 116" to 20". 1692 gals. into trailer #7454.

14:45 Increased 21.11 product out temp from 280 to 290 °F.

16:24 Dropped pressure in 27.14 from 724 to 675 psig to stop methanol from condensing in 27.14.

8 June 1995

00:35 Day tank transfer from 120" to 20". 1762 gals. into trailer #7454.

00:21 D04 shot lost H2 peak, but other Dennis GC ports ok.

07:30 Changed TIC-1260 on 21.11 from 290 to 295 °F.

11:50 Day tank transfer from 112.5" to 20". 1630 gals. into trailer #7454.

21:00 Swapped heater/cooler order in UO circuit. Average 27.20 temperature only upset +4/-2 °F during swing.

22:35 Day tank transfer from 117" to 20". 1710 gals. into trailer #7488. DEC stopped receiving updates from HP.

22:55 Rob shutdown and restarted the lab.

9 June 1995

07:30 Performed slump test on reactor 27.20.

08:00 Started move to AF-R3.3 conditions.

12:20 Day tank transfer from 116" to 20". 1692 gals. into trailer #7488. Tried to reach high velocity 91.2 ft/sec) using TEXACO gas composition at lower pressure. Only got up to 160,000 SCFH (target 204,462 SCFH).

15:45 Changed gas composition back to Kingsport gas to reach 1.2 ft/sec. REVISED CONDITION AF-R13.3 for Kingsport gas at SV = 10,000 std. l / hr-kg. Reduced reactor pressure to 718 psig.

19:10 Day tank transfer from 88.5" to 20". 1207 gals. into trailer #7488.

10 June 1995

00:05 Day tank transfer from 89.5" to 20". 1225 gals. into trailer #7488.

02:52 Printer in lab stopped printing. DEC stopped getting updates from HP.

04:05 Rob stopped and restarted everything.

04:45 Printer dumped all its data (DEC received it all).

05:45 Day tank transfer from 99" to 20". 1392 gals. into trailer #7484.

08:00 Loaded oil in 28.30 prep tank.

Drum	Full Wt. (lb)	Empty Wt. (lb)	Net Wt. (lb)	Subtotal (lb)
1	500	121	379	379
2	500	121	379	758
3	500	121	379	1137
4	500	121	378	1516
5	372	121	251	1766

TOTAL OIL ADDED = 1766 lbs

11:00 Started Dephlegmator testing. Shut pump back from 27.14 to 21.11.

Time	27.14 (nuts)	21.11 (in)	Reactor Ht. (in)	Reading %
11:00		26.25/37.5		35.71
11:03	16			62.43
11:05			480	
11:38	17.4			66.13
11:40		21.75/33.25		29.23

11:30 Day tank transfer from 98" to 20". 1375 gals. into trailer #7484.

13:00 27.14 @ 20 nuts (74.31%)
21.11 @ 23.5" (?) (16.65%)

14:03 27.14 @ 22 nuts (80.94%)
21.11 @ 17.5" (8.25%)
27.20 @ 480"
Shut pump back from 21.11 to 27.20. Pump back from 27.14 still off.

14:21 27.20 @ 467.5" on tape.

14:26 27.20 @ 467.5" on tape.

14:44 27.20 @ 458.3" on tape.

14:46 27.20 @ 458.3" on tape.

15:07 27.14 @ 24 nuts (87.22%)
21.11 @ 20.5" (13.58%)
27.20 @ 446.8"

17:30 Day tank transfer from 102.75" to 20". 1459 gal. into trailer #7484.

22:30 Started another dephlegmator test
27.14 @ 15.75 nuts
21.11 @ 22.5/33.25"
Stopped pump back from 27.14 to 21.11 and bypassed demister.

23:27 Day tank transfer from 101" to 20". 1428 gal. into trailer #7484.

23:30 27.14 @ 18.0 nuts
21.11 @ /26.25"

11 June 1995

00:30 27.14 @ 19.75 nuts
21.11 @ /18.5"

00:35 Stopped pumpback from 21.11 to 27.20 (still bypassing the demister). Starting reactor level at 476".

00:45 PLANT TRIPPED! 01.10 ate it belts.

06:00 Day tank transfer from 46" to empty. 705 gal. into trailer #7484.

11:00 Day tank transfer from <17.5" to empty. <201 gal. into trailer #7484. Small amount of MeOH found in 22.1 and dumped into waste MeOH. Approximately 14 gal.

11:40 Blocked all feed valves from HYCO. Flare out.

12:30 Fill 21.11 with flush oil to 47".

13:00 Drained spent slurry into drums. Collected 3-5 gal. buckets of spent slurry as samples. Filled 5 drums of spent slurry.

13:30 Some rainwater (~ 1 cup) left in each 55 gal. drum before slurry 6 was drained into the drums (even though water was poured out of the drums before charging slurry). Expect that water will settle to bottom or near bottom of drums. 5 gal. "buckets" were dry before filling with spent slurry.

14:30 Draining oil from 27.14 into 55 gal. drum. Flush 27.20 reactor with oil from 21.11. Drain flush oil into 55 gal. drums.

12 June 1995

08:45 Loaded alternate methanol catalyst powder (target amount is 1177 lb).
Lot 022811 (net 50 kg; gross 63 kg). Drums were numbered: #/95.
A sample from each drum was collected.

Drum	Full Wt. (lb)	Empty Wt. (lb)	Net Wt. (lb)	Subtotal (lb)
31	162	53	109	
30	163	53	110	219
27	163	53	110	329
26	164	53	111	440
28	164	53	111	551
29	164	53	111	662
39	163.5	53	110.5	772.5
34	164	53	111	883.5
32	163	53	110	993.5
33	164	53	111	1104.5
35	126	53	73	1177.5

TOTAL CATALYST ADDED = 1778 lb

11:30 Weighed out flush oil (to flush catalyst slurry remaining in prep. tank to 27.20 reactor).
Full drum: 403 lb
Empty drum: 121 lb
FLUSH OIL: 282 lb (target: 283 lb)

13:00 Transferred slurry from 28.30 to 27.20, Liquid Ht = 241"

13:20 Transferred flush oil to 27.20, Liquid Ht = 289.4"

13:50 N_2 flow increasing.

14:30 Starting CO flow.

14:44 Started heating up.

15:00 Rxt Avg Temp = 193.3°F, Rxt Pr = 68.7 psig, FI-126A Flow = 12,569 SCFH N_2, Rxt Level = 344.7"

20:15 CO composition in feed has been creeping up. Line pressure is also running high. Matt made a move to decrease CO flow.

Uptake @ 20:00 (5.25 hr's into redtn.) was 1.2 SCF/lb.

21:00 Uptake 6.25 hours into redtn = 1.53 SCF/lb (281°F).

22:00 Uptake 7.25 hours into redtn = 1.86 SCF/lb (295°F).
Still no H_2 production, everything else steady.

22:45 Did a nuke scan. Reading did not seem a steady as they should have been at these rates.

23:00 Uptake 8.25 hours into redtn = 2.20 SCF/lb (309°F).
Still no H_2 production.

24:00 Uptake 9.25 hours into redtn = 2.40 SCF/lb (322°F).

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01:00 Uptake 10.25 hours into redtn = 2.44 SCF/lb (336°F).

02:00 Uptake 11.25 hours into redtn = 2.48 SCF/lb (351°F).

03:00 Uptake 12.25 hours into redtn = 2.52 SCF/lb (365°F).

04:00 Uptake 13.25 hours into redtn = 2.53 SCF/lb (380°F).

04:50 Cut Rates 25% per Test Authorization.

05:00 Uptake 14.25 hours into redtn = 2.59 SCF/lb (395°F).
This sudden jump up looked anomalous, so I checked the download files and found that D03 lost its H_2 peak for two consecutive shots around 0400. Deleting these points from the .dwn file resulted in an uptake calc'n of 2.55 SCF/lb.

09:45 Reduction complete. Rxt Avg. T = 464°F
Switching to once-thru syngas.

10:55 Reactor level at 288" just before syngas flow started. It was at 315" to 350" with N_2 flow through reactor.

11:17 Started 01.20 recycle flow, Reactor Level up to ~`395".

15:30 Unit almost at R14.1 condition.

15:40 Approx. 20 lbs of alternate catalyst was washed out of prep tank (<u>not</u> transferred to 27.20 reactor).

15:50 Yesterday noticed that TI-311 (21.30A shellside) was reading 98°F without hot process gas (pipe was at ambient). Ray pulled TI-311 and confirmed that it was not working. TI-311 was replaced with a new TI. Previous measurements from TI-311 should be considered unreliable!

16:00 Ray drained sight glass on 21.11. When refilled sight glass, it filled with black slurry. After approx. 5 mins, the black solids (catalyst) started to settle. It was decided not to re-drain the sight glass.

17:30 Have reached conditions for AF-R14.1. Reactor T&P are at target. Reactor feed composition and flow are also essentially at target. Suggest start data period AF-R14.1A at 18:00.

20:00 Matt pulled a liquid sample off 22.11.

14 June 1995

01:00 Day tank transfer 123.25" to 20". 1819 gallons into trailer #7486.

04:05 Matt grabbed a liquid sample of 22.11.

09:00 Dean is taking Rocco down to replace valves. Rocco had been reading low methanol (~1%). Dean thinks this is because sample is not getting in.

09:30 Dean has fixed and restarted Rocco.

09:45 271.4 level 8.25 nuts 37.94%
21.11 level 38.25" 37.21%
Stopped oil pump back from 27.14 to 21.11.
Demister (21.11) is on-line
Reactor level is 480".

10:40 <u>21.11 Temperatures</u>
Feed Gas inlet (TI-1257) = 147.7°F
Feed Gas outlet (TI-1263) = 367.9°F
Product Gas Inlet (TI-1262) = 467.7°F
Product Gas Outlet (TIC-1260) = 281.6°F

11:10 Ray pulled a liquid sample off 22.11

11:20 271.4 level 10.5 nuts 45.74%
21.11 level 21.5" 26.09%
Stopped oil pump back from 21.11 to 27.20.
Still no oil pump back from 27.14 to 21.11.
Demister (21.11) is on-line
Reactor level is 480".

11:30 Day tank transfer 118" to 20" 1727 gal. into trailer #7486

12:20 Reactor level at 450"

12:35 Reactor level at 441"

12:45 Reactor level at 436"

12:50 21.11 level at 36 3/4" 36.51%
27.14 level at ~12 1/2 nuts 53.25%
DEC/Bailey shows level being at same rate as during 9:45 to 11:20 test. Assume same rate of oil carry-over from 21.11 to 27.14.
Resume oil pumpback from 27.14 → 21.11 and 21.11 → 27.20.
Could not wait for oil level in 27.14 to rise further because concerned that level in reactor would get too low.

14:45 Swing valves to <u>bypass 21.11 demister.</u>

15:12 2.11 level 37" 35.34%
27.14 level 8+ nuts 38.59%
Reactor level at 480". Stopped oil pumpback from 27.14 to 21.11.

16:15 21.11 temperatures

Feed gas inlet	(TI-1257) 144.1°F (147°F before 15:45)
Feed gas outlet	(TI-1263) 368.3°F
Product gas inlet	(TI-1262) 466.7°F
Product gas outlet	(TIC-1260) 279.1°F (280.5°F before 15:45)

16:48 21.11 level 26" 20.41%
27.14 level 11+ nuts 47.59%
Reactor level at 480".
Stop oil pumpback from 21.11 to 27.20.
Still no oil pumpback from 27.14 to 21.11.

18:04 Reactor level at 441".

18:10 21.11 level 31 1/4" 28.94%
27.14 level 13+ nuts 55.20%
Resume oil pumpback from 27.14 → 21.11 and from 21.11 → 27.20

18:20 Day tank transfer from 83" to 20" 1110 gal into trailer #7486

18:30 Started to ramp down 21.11 product gas outlet temp (TIC-1260) from 280°F towards 260°F

18:50 Swing valves to return 21.11 demister to service.

19:30 Matt pulled a liquid sample of the 22.11.

20:00 Dec crashed! Rebooted itself.

20:30 D04 came through, so we still have HP/DEC communications.

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03:05 Matt grabbed a liquid sample off 22.11

Have been lax on liquid loss and nuke scans because Matt and I have been fighting with the plant all night. The back end is constipated and we have been fighting a losing battle with the levels in the 22.10 and 22.15.

04:45 Matt pulled a piece of junk out of LV-242. We are finally able to really unload 22.10 and 22.15 into the day tank.

05:45 Day tank transfer 121" to 20", 1780 gal. into trailer #7486.

07:20 Started dephlegmator testing with TIC-1260 set at 255°F. Plant has been running all night at this condition, but we never got a chance to do the test because of back-end problems.
21.11 level 41" (15 nuts) 40.04%
27.14 level 5 1/2 nuts 29.58%
Stop oil pumpback from 27.14 → 21.11

Reactor level is at 480"
(21.11 demister is on-line)

08:45 21.11 temperatures

Feed gas inlet	(TI-1257)	144.5°F
Feed gas outlet	(TI-1263)	348.8
Product gas inlet	(TI-1262)	467.0
Product gas outlet	(TIC-1260)	255.6

08:55 21.11 level 32 3/4" 29.18%
27.14 level 8 nuts 37.01%

09:02 Stop oil pumpback from 21.11 →27.20
Still no pumpback from 27.14 → 21.11
Reactor level at 480".

10:29 21.11 level 39 3/4" 39.68%
27.14 level 10 nuts 43.69%
Reactor level 434"
End of dephlegmator tests. Resume oil pumpback from 27.14 → 21.11 and from 21.11 → 27.20

10:30 Asked Ray to slow down sample taps because noticed lower than normal MeOH content in GC analysis of SP 3A.

10:30 Ray took a liquid sample off 22.11.

10:30 Started increasing 21.11 product gas out temp to heat up separators in preparation for Kingsport cond.

11:00 Reactor level backup to 480"

11:20 Mina did a nuke scan before slump test.

12:10 Performed slump test. Liquid level crossed 375" 11 sec after flows shut off.

12:27 Slump test complete. Making move to Kingsport, SV-4000 conditions (AF-R14.2). During slump test, reactor average temperature dropped from 481 to 473°F. TI-1240 (bottom of reactor, J11) increased from 473°F to 483°F,. TI-1235 (J7) dropped slowly from 486 to 483°F.

12:55 Day tank drained to 24 1/2" from 85 1/2" 1076 gal. into trailer #7486.

14:45 Added oil to 27.14 from <1 nut to 6 1/2 nuts (between sight glasses).

15:30 Added oil to 27.14 from 1 1/2 to 6 nuts.

17:40 Added oil to 27.14 from 4 1/2 to 10 nuts.

18:00 Very nearly at conditions for AF-R14.2.
Recommended start date period AF-R14.2 at 19:00

18:00 Ed/Dave/Ray have been fighting with flakes and grit coming out of 22.15 and plugging up the control valve, LV-242. A piping run from the 22.15 sight glass to the 22.15 exit stream filter was put in place and is now in use. Another tubing ran to bypass the 22.15 product filter and control valve directly to 22.16 was installed but is not in use so far.

20:30 Took outside liquid levels.

23:25 Nuke scan taken - stable readings.

16 June 1995

00:45 Getting set up for a day tank transfer
122" to 20" - 1797 gallons into trailer #7484

02:25 Matt grabbed a liquid sample off 22.11.

03:00 Second time the 21.40 tripped out on vibration. No effect on the process.

03:37 Another 21.40 vibration trip.

04:10 Nuke scan - liquid levels taken.

10:30 Ray collected a liquid sample off the 22.11.

11:50 Day tank transfer from 114" to 20". 1657 gallon into trailer #7484.

18:15 Day tank transfer from 72.5" to 20". 925 gallons to trailer #7484.

21:00 Nuke scan - very stable; liquid sample taken.

21:30 Outside logs taken.

17 June 1995

00:05 Liquid level logs taken.

02:40 Outside logs taken (22.16 between sight glasses at 0200).

03:10 Noticed while working on the mass balance, that FI-196 (27.14 outlet) was reading 0 during the entire mass balance period. Had to adjust the mass balance program accordingly. Matt blew down the taps on FI-196. It is now reading a proper signal again.

04:00 MeOH sample taken; Nuke scan taken.

04:40 Outside readings taken.

06:05 Day tank transfer from 116 3/4" to 20"; 1706 gal to trailer #7484. Next transfer should be no higher than 69" (873 gallons).

10:00 MeOH sample taken; nuke scan done.

12:00 Day tank transferred 69" to 20" (863 gallons). Trailer #7484 is now full.

18:00 Day tank transferred 68" to 20" (845 gallons). First transfer in trailer #7124.

18:00 MeOH sample taken

21:00 Did some cleanup on the DEC - purge files, etc.

22:25 Nuke scan
Noticed Ft-196 again was reading zero inches. Matt going up to blow down the sample taps and wrap the root valves to prevent condensation.

23:30 Finished outside logs.

18 June 1995

02:00 Liquid methanol sample collected

03:50 Nuke scan done.

04:30 Outside logs taken

05:45 Closed off on LIC-1255 (27.14 to 21.11)

06:00 Closed off oil return to reactor DIC-1242; trying to thicken up before switching conditions (level 480)

06:07 Began 22.16 transfer from 118.5 inches

06:40 Level in reactor at 473.8".

06:45 Finished 22.16 transfer, 1736 gallons.

07:00 Outside liquid levels taken end of AF14.2

07:01 Start morning to AF14.3 conditions. Level above 534" (top of nuke). TI-1255 is 492F indicates slurry up that high (2' from top face of flange)

08:30 Making fine adjustment in feed composition. Increased H_2 @ 01.10, 8.5 → 9.5 MSCFH

08:34 Discovered DEC problems! "SHO SYS" did not show "TREND_COLLECTOR" indicating DEC was not saving data. Did a "RESTART" to turn on "TREND_COLLECTOR". Did a number of downloads to figure out when DEC stopped saving data. Looked like it stopped right after 0400 on 6/18. So, 14.2B will be from 01:00 6/17 to 04:00 6/18.

09:10 Last few hours GC printer out of ink.

09:30 Switched from SPI5 to SP6 for D06 and G02.
(Purge 2 will now get correct composition).
Reactor level decreasing: In manual @ 489"
21.11 building up level
27:14 pump back started to evaporate MeOH

12:00 Composition leveling out on Rxt feed.

12:20 Transferred day tank from 100" to 50.5" (872 gallons). Trailer level switch tripped.

12:55 CO feed is being curtailed by HYCO.
Backing out CO from 18,500 to 10,000 SCFH
Decreasing Rxtor pressure to maintain velocity.

14:40 Estimated velocity = 1.07 ft/sec
@ 138,000 SCFH Rxt feed, 560 psig Rxt Pr.

15:50 High pressure H_2 switched from 01.20 suction to 01.10/01.20 discharge

17:00 Adjusting feed composition; lowered H_2
Keep CO fixed at 10,000 SCFH

18:00 Day tank transfer: 101" to 20" →1428 gallons
Unit lined out: Mass balance period can begin @ 18:00
Rxt flow = ~143,000 SCFM
Rxt Pr = ~522 psig
Rxt Temp = 482°F
Rxt liquid level = 480"
Has inlet vel ~ 1.19 ft/sec

20:30 Outside logs taken; GC's sample pots in hot box blown down. Some liquid in third from left.

21:10 Methanol sample collected

23:10 Outside liquid levels taken; sample pot 3 (from left) had liquid drops in it when I blew it down.

23:30 Trying PIC-196 in auto

PIC-247-1	01.20 suction 431.5#
PIC-196	21.11 inlet 476#
PIC 1247	reactor 524 #

2323 GC	on reactors feed
54.72%	H_2
26.59	CO
11.48	CO_2
6.12	H_2

Matt's increasing H_2 again to try bump it

19 June 1995

00:00 Matt was adjusting the manual block valves up and down stream of the HP H_2 controller PIC-1250. The H_2 flow temporarily shot up from 16 MSCFH to 60 MSCFH. PIC vent was in auto so it opened up a bit. (Maybe the blast of H_2 will help clear -- some of the N_2).

Had to reboot the PC again (3rd night in a row - not enough memory). Previously PIC 1200 was controlling 50 or more % open and still not getting flow. He opened up on the upstream block valve - now at 2-3% open but getting the flow.

00:40 Latest GC reading

0025		Pressure at 27.20 523.8
H_2	58.2	FI-1216 flow at 136
CO	25.13	
N_2	5.23	
CO_2	10.49	

Matt's making a move with H2 to get flow up higher.

00:50 Latest GC 00:43 FI-1216 138

H	58.7
CO	24.58
N_2	5.3
CO	10.2

Matt's going to increase HP H_2 again up to 20.5

00:55 Nuke scan - readings are more jumpy ± 0.5 - $\pm$ 1.0 in liquid readings.

01:40 Matt changed paper on GC computer printer. It appears (GC's) to still be communicating with the DEC.

127 GC --- reactor feed

H_2	61.59	FI-1216 at 139
CO	22.87	
N_2	4.88	
CO_2	9.64	

Reactor p at 519 (cycle between 517.5-519.5) because PIC-196 has been auto

Matt put PIC-196 in manual again to try "iron out" the cycles in the plant flows. Only way to increase flow is to increase H_2 but we're already slightly H_2 rich Hold here for a while

0145 feed GC	62.94 H_2	reactor feed flow jumped up to 143-144 MSCFH
	22.04 CO	pressure up to 52.6
	4.78N_2	PIC 196/PV-201 manual 12%

Matt's backing off HP H_2 - needs to go adjust the 01.13 manual valve

to get FIC-1200 more controllable, It has been running 2.7%
Now at 5.3% → 19.7 MSCFH

02:20 Liquid logs taken
GC pots blown; liquid in #3 again

03:10 Methanol liquid sample taken
GC's reactor feed: 247 H_2 64.9 PI-1247 down to 513 again
CO 20.81 FI 1246 CO at 142.9
N_2 9.94
CO_2 8.63
We're cutting back on H_2 once more. PV-201 still manual at 12%

05:00 Took liquid levels; blew down GC pots; droplets in #3.
Began nuke scan - readings still varying ±0.5 - ± 1.0 in the liquid region.
0451 Gary feed H_2 61.95
CO 22.47
N_2 5.04
CO_2 9.34

05:57 Begin 22.16 transfer.

06:45 1613 gallons transferred
Only room for 46 2/3" before filling up the trailer!

07:40 Outside full readings taken.

09:20 Day tank transfer: 46" to 20" (459 gallons)
Trailer #7124 now full

10:00 Ray pulled a liquid sample off the 22.11/22.15.

11:15 Full set of outside logs.

12:32 Nuke scan Nuke readings in liquid were quite jumpy: ranging ±0.5 to ±1.5

15:00 Ray pulled a liquid sample from the 22.11/22.15.

16:07 Started reactor shut down test.

16:30 Finished reactor shutdown test.
Conditions at end:
PIC-1247 719 psig
Avg temp. 474.0°F
TI-1235 483.3°F
TI-1239 476.1°F
TI-1240 478.8°F
Start move to next condition. AF-R14.4 (Texaco, inlet SV=4000 std l/hr/kg cat.)

We are still limited to 10,000 SCFM of CO and so may need to run reactor at less than the target 750 psig (lower conversion to MeOH and less makeup CO needed).

20:15 "Dennis" GC shot at 20.03 of sample 4 showed erroneous composition results (52.36%, 4.99% N2, 2.34%, CO2 0.46%). Problems with "Dennis" were confirmed with GC standard shot at 2034. This caused problems with FI measurements and FIC's that were on auto. Matt put FIC-246 on manual but we temporarily dropped pressure in the loop and reactor (reactor pressure had just reached 750 psig). We entered MW's manually into the Bailey to over write incorrect values sent by "Dennis". Called to consult with Dean.

20:54 Stopped "Dennis to prevent receiving incorrect MW's. Dean is on his way in.

21:20 Stopped pumpback from 27.14 to 21.11 since 27.14 level was at 2 1/4 nuts (piping level).

21:30 Day tank transfer begun

21.22 GC shot Gary	38.43	H_2
	44.32	CO
	2.12	N_2
	14.32	CO_2

21:45 Dean working on GC Dennis

21:55 End trailer transfer 1498 gallons transferred to new trailer.
GC Dennis back in operation at 22:39 shot. The 22.58 shot of DO5 was really a sample #4. Changed SP8MW in the Bailey temporarily. To avoid these GC's, errors, should probably start Data period at 23:00.

20 June 1995

00:00 Dennis 4 analysis - right on composition, flow - pressure targets! Outside logs taken.

00:30 Nuke scan taken.

01:00 Methanol sample collected.

02:45 MW-SP3A is incorrect happened around 145& is staying at 64 (should be ~26 or so). Doesn't affect any control valves; only FI-196 - inlet reactor composition great!
I am investigating whether liquid product rates match the GC reactor outlet predictions. GC's reading 7.9% MeOH in effluent, Predicted is 11.94%.

Day tank measurements $\frac{47" - 35"}{(02:10 - 00:00)} = \frac{723 - 511\ gal}{130\ \text{min}} = \frac{1.63\ gal}{\text{min}} = \frac{2348\ gal}{day}$

Predicted is 2337 gal/day (matches 11.5% in effluent)
Hand calc to check predictions.

$$\frac{2348\ gal}{day}\ \frac{1\ day}{24\ hr}\ \frac{50\ lb}{ft3}\ \frac{ft\,3}{7.481\ gal} = \frac{653.96\ lb}{hr}$$

$$= \frac{20.43\ lb}{hr}$$

$$reactor\ feed = \sim 81.5\ MSCFH\ \frac{lb\ mole}{386.67\ SCF}\ \frac{\sim 21\ lb}{lb\ mole} = \frac{4427\ lb\ feed}{hr}$$

$$\frac{4427}{hr}\ effluent\ \frac{lb\ mole}{\sim 26} = \frac{170.3\ lb\ mole}{h}\ outlet$$

$$\frac{20.43}{170.3} = 11.99\%\ methanol\ in\ the\ effluent\ based\ on\ 22.16\ levels$$

Both GC's off!!??
Confirm with DMECSTR calc's what expected performance is.

03:10 Another set of levels trying to confirm productn rate.
21.11 level barely in sight glass. 22.14 up to 5 nuts.
Restart pumpback between 27.14 and 27.11.
Checked GC printouts ; at 108, Bharat analysis showed a large "Pentane peak" in the summary. This would explain the MW jumping up to the 60's, since the Bharat 03 MW portion was 71 (C5H12 is 72) instead of 32
Conclusion: Bharat is not believable for SP3A.
Rocco also still shows 7-8% meoh in the effluent. There are no apparent mysterious peaks. Rocco and Bharat were both reading high concentration (10% or greater) until ~8PM. Since then, they have seen consistently 7-8 ish!

05:00 Outside logs taken.

06:00 Methanol collected.

07:00 Outside liquid levels taken.

09:25 Nuke scan

10:13 Full set of outside logs taken.

11:40 Start dephlegmator testing.
21.11 Demister is on-line
27.14 5 nuts (27.08% on lvl xmtr) 25 gal
21.11 33 1/2" (28.31% on lvl xmtr) 37.9 gal
27.20 480"

11:44 Stop oil pumpback from 27.14 → 21.11
27.20 still on auto level control

12:26 27.14 5 1/4 nuts

21.11 32 1/4 nuts

12:45 Partial set of outside logs.

13:35 27.14 6 nuts (lvl xmtr. 30.28%) 25.75 gal
21.11 27 - 30" (BG) (lvl xmtr. 24.11?) (31.2 - 34.3 gal)

14:30 Ray pulled a liquid sample from 22.11/22.15.

14.40 Reduced LP H2 feed slightly since reactor feed was becoming slightly H2 rich.

14:56 Full set of outside logs

15:20 Day tank transferred: 124' to 2-" ⇒ 1833 gallons to trailer #7406

16:40 27.14 6 1/2 nuts (BG) (lvl xmtr. 34.79%) 28.33 gal
21.11 25 3/4" (lvl xmtr. 18.00%) 29.85 gal

17:36 27.14 7 nuts (lvl xmtr. 36.24%) 33 gal
21.11 24 1/4" (lvl xmtr. 16.78%) 28.25 gal
27.20 480"

17:42 Stopped oil return 22.11 to 27.20
Still no oil return 27.14 → 21.11

18:20 27.14 7 3/4 nuts (Lvl xmtr. 37.21%) 35.25 gal $\Delta = 2.25$
21.11 26 1/2" (Lvl xmtr. 19.75%) 30.6 gal $\Delta = 2.35$
27.20 470.4"

reactor delta 10.4 in = 10.4 (1.102 $\frac{\text{gal}}{\text{in}}$) = 11.5 gal

19:25 22.11 conditions
Feed gas IN temp (TI-1257) 155.5 °F
Feed gas OUT temp (TI-1263) 313.4°F
Prod gas IN temp (TI-1262) 461.8°F
Prod gas OUT temp (TIC-1260) 274.7°F

19:40 27.14 8 1/4 nuts (Lvl xmtr. 39.09%) 36.75 gal
21.11 31 1/4" (Lvl xmtr. 26.44%) 35.55 gal
27.20 453.6 in.

19:59 End of this dephlegmator test

19:59 Return reactor to level control (oil return from 21.11 → 21.11)
Return 21.11 to level control (oil return from 27.14 → 21.11)

20:08 Full set of outside logs. Reactor level was below 480" (recovering from dephlegmator test.) Do <u>not</u> use this data point in oil balance.

21:03 Matt shut oil pumpback from 27.14 to 21.11 to lower 21.11 level in preparation for next dephlegmator test.

21:30 Matt took a liquid sample off the 22.11/22.15.

22:30 Reactor level at 480 inches.

22:35 Demister bypassed on 21.11.
21.11 31.25 in (25.85%) 27.14 - 5 1/2 nuts (30.03%)

23:05 22.11 27-30 (BG) in (24.43) 27.14 6 nuts (30.78%)

23:35 21.11 27-30 (BG) (22.96) 27.14 6 - 7 (BG) (31.78)

00:08 21.11 27-30 (BG) (21.44) 27.14 6 - 7 (BG) (32.79)

21 June 1995

00:40 Both still in-between sight glasses.

01:12 21.11 26 3/8 (18.58%) 27.14 6 - 7 (BG) (34.64)

01:47 21.11 25 3/8 (17.18) 27.14 7 (35 - 76)

02:17 21.11 24 3/4 (16.2) 27.14 8 (36.71)

02:20 Block in 21.11 return to reactor.
27.14 still blocked; reactor level at 477.6 inches.

02:43 Reactor level 474.6"
21.11 26.0 (18.33%) 27.14 8(higher than before but not quantifiable (37.54)

03:25 21.11 BG (21.43%) 27.14 8.5 (38.56) reactor 465.9
Liquid levels taken

04:35 22.11 32 (26.65) 27.14 9.0 (40.04) 447.6

04:40 Reactor level back in control at 450 inches. (Estimated height for next run condition given increased gas holdup)
21.11 back in auto at 27% level (~32 inches)
(pumpback to 21.11 and reactor is now on.)
End dephlegmator test - demister bypass shut!

05:00 Methanol sample taken.

05:30 Nuke scan done.

06:00 Outside levels taken.

06:05 Day tank transfer begun.

07:05 Last set of liquid levels taken. End of AF-R14.4. Start transition to F-R14.5.

09:00 Rxt temp reached 500°F and temp was on manual. Rxt temp on control.

10:00 Rxt lined out. Mass balance period can begin.

10:20 Adjustments to CO_2 and HP or feed flows.

11:50 Adjust CO feed 17.5 to 17.3 MSCFH.

12:55 Adjust CO 17.3 to 17.0 MSCFH
MPH_2 23.9 to 23.6 MSCFH

13:30 Ray took a liquid sample from 22.11/22.15.

13:45 Cutting back fresh feeds to reduce gas vent flow.

13:50 Day tank transfer 85" to 24 1/2" 1067 gal. into trailer #7406. Trailer is now <u>full</u>!

14:05 to 14:35 Cutting back CO from 16.4 to 15.5 MSCFH

15:25 to 16:00 Fresh CO to 16.0 MSCFH. CO_2 from 63 to 61%

15:30 FT-237A transmitter is back in service.

18:00 Ray took a liquid sample off the 22.11/22.15
Nuke scan.

18:30 Another move on CO: 16.0 to 16.2 MSCFH.

23:33 Completed nuke scan.

Between approx. 1200 and 1800 on 6/21/95 chains were installed around the 27.20 reactor to hold tracer CO (tracer study) detectors in position. Chains are located at 27, 69, 94, 120, 227, 333 and 401 inches on tape. These would not have affected nuke scan performed at 11:15 on 6/21/95. Looking at 6/21/95 18:00 and 22:43 nuke scan, it appears that readings were affected by the chains at only the 120 in. (on tape) elevation. This does <u>not</u> appear to affect avg. gas hold-up calculated.

23:40 Day tank transfer 109 1/2" to 20". 1578 gal. into trailer #7207

22 June 1995

00:05 Matt pulled a liquid sample from the 22.11/22.15.

00:28 27:14 10 1/3 N (44.86%) 21.11 38" (35.43%)
Start bypass of demister and block in LIC-1255 oil from 27.14 to 22.11

01:07 27.14 11 1/2N (48.14) 22.11 34 1/8 (29.53%)

01:10 Stop oil return to the reactor.

01:40	27.14	BG (12-13) (50.77)	21.11 35.5 (32.18)	27.20	464.8
02:09	27.14	BG (53.22)	21.11 37 1/8 (34.51)	27.20%	452 1/2
02:32	27.14	13 1/2 (55.05)	21.11 38 1/8 (35.98)	27.20	442

02:40 Demister back in line; Pumpback still blocked out

03:12	27.14 14 1/2 (58.22)	21.11 4o 1/4 (38.81) 27.20	433.6
03:45	27.14 15 1/3 (60.58)	21.11 41 3/4 (40.95).27.20	412.6

04:15 Reactor back in level control following a nuke scan at 412.6 inches.

04:22 Outside logs taken.

04:40 21.11 level has dropped back; put back in auto at 28% - begin pumpback from 27.14.

05:55 Last nuke scan done. Results very repeatable to scan done pre-dephlegmator testing.

07:05 Ed is locking out the nuke. We're running with the oil return in manual at 25.1%.

08:40 Day tank transferred: 97.5" to 20" (1366 gallons) trailer (#7207).

Tracer Study: Levels		Detector Positions Rxt Ht, Ft	" on tape
4 X	1	5	18
↓	2	10.7	86
↓	3	12.7	110
↓	4	21.7	218
↓	5	30.7	326
↓	6	36.7	398
↓	7	41.7	458
1	8	52.1	583
1	Rxt Inlet		
1	Rxt Outlet		
1	Recycle Line		
32	Total		

Run No 14.6

12:58 First gas injection made (thru valve V2462).

13:35 Second gas injection made.

15:15 Liquid injection at nozzle N_2, 4 1/2" from wall. First shot too small.

16:00 Double dose shot @ same location.

16:24 Liquid injection at nozzle N_2, wall.

17:00 Day tank transfer 95" to 20", 1322 gal into trailer #7207.

19:36 Liquid injection at nozzle N1 at "center" of reactor. Looked like a good injection, although it took " a little long".

20:02 Liquid injection at nozzle N1 at wall of reactor.

20:20 Nuke is re-set to 480" but traverse is "locked-out" so it can't be moved.

20:30 Moving to next condition AF-R14.7 (Texas gas, SV=4000 std/hr/kg cat). Ed checked position of top detector. Using metal tape, detector was at 487" = 583". This is different from previous table (previous table corrected). GC shot at 2027 (probable reflecting previous condition) showed (Dennis 04).

H_2	34.93%
CO	50.11
N_2	0.97
CO_2	12.86

23 June 1995

09:20 Day tank transferred: 122.5" to 20" (1807 gallons) to trailer #7207. Room left for one more full load: 125" max.

12:18 First gas injected Run 14.7.

12:37 Second gas injected.

13:15 Liquid Injection at N_1 - center.

13:36 Liquid injection at nozzle N_1 - at wall.

14:43 Liquid injection at nozzle N_2 - center.

15:03 Liquid Injection at nozzle N_2 - wall.

15:15 Nuke moved to 480" (inside LI) and detector shutter opened traverse locked-out and so can not be moved.

15:45 Liquid level on reactor is >480". Stop oil return from 21.11 to reactor to swing down level will rise in 27.14 and in 21.11 (because oil carry over rate from 21.11 to 27.14 is slower than 27.20 to 21.11).

16:30 Rxtor level below 480" now. Started change to R14.8. Decreasing pressure first slowly.

19:00 Matt making move to next condition.

20:30 Plant tripped SD-1 on 01.20 discharge temp high (TI-609 showed 166°F).
We had been at target pressure, near target flow, and moving in on target composition.

20:40 Plant is up and running again, flows and pressure are near target again. Adjusting to get target composition.

23:30 Gary 05 has been showing a very large DME peek (~800%) in the recycle analysis. MW is off accordingly, but since it doesn't affect a controller, we're not correcting it.

24 June 1995

00:50 Begin day tank transfer.

01:22 End transfer 1754 gallons transferred.

05:12 Outside Logs taken. GC Gary has not been totaling well (~97 - 97.5) on the reactor feed. See note above on SP4.

10:05 GC's were shut down at 09:15. A standard shot on both "Dennis" and "Gary" was accidentally sent to the DEC as SP4. To avoid upsetting flow at FI-1216A, a manual SP4 MW of 13.5 was entered into the Bailey system.

11:12 01.20 (TI-609) discharge temp has been climbing this morning (as ambient temp rises). It reached 157°F and PIC-247-2 (01.20 discharge recycle) was opened from 6.5 to 6.8% to decrease recycle feed flow and help bring down discharge temp.
Total reactor flow has dropped from 137 MSCFH to 133 MSCFH. 01.20 discharge temp. is dropping.

11:39 First gas injection.

11:56 Second gas injection.

6/21/95 LEVELS OF OIL & SETTLED CATALYST IN SLURRY DRAINED 6/11/95
Empty drum: inside height = 33.5" (rim to inside bottom)

Spent Slurry
(drums labeled: "Baseline Catalyst" 27.20 drain 6/11/95

Drum #	"Liquid" level (slurry & foam) in. from rim	"Liquid" (slurry & foam) Height (in.)	Comments
1	3 3/4"	29 3/4"	No catalyst
2	2 3/4"	30 3/4"	has settled.
3	4"	29 1/2"	Each drum has
4	2 1/2"	31"	1" to 1 1/2" of
5	3"	30 1/2"	dried slurry foam on top. Drums were last moved on 6/12/95.

Reactor flush oil
(drums labeled: 27:20 flush drain 6/11/95)
2 drums: black, transparent oil on top approx. 1/2" of settled catalyst at drum bottom

27:14 Drain oil 6/11/95
1 drum: gray but transparent oil
Re-examined 6/26/95
Liquid at top of drain: green MeOH (very fine catalyst dust?)
"Liquid" at bottom of drum: MeOH/oil mixture with distribution of catalyst particles (fines up to 2 mm diam. particles).

13:30 6/22/95 Weight of spent slurry drained 6/11/95

Empty 55 gal + drum lifter = 117 lb (empty drum = 52 16)
Empty 5 gal "bucket" = 4 lb. (but feels more like 2 lb)
Drums labeled: BASF 27.20 drain 6/11/95

Drum #	Full wt. Drum & lifter	Slurry
1	578 lb	461 lb
2	604	487
3	592	475
4	607	490
5	594	477

Bucket #	Full wt. Slurry	
1	49 lb	45 lb
2	50	46
3	est.50	46

↑sample was already shipped
TOTAL Slurry = 2527 lb.

12:08	Compressor 01.20 discharge temp	154.4°F
	Reactor press.	513.2 psig
	Reactor temp. avg.	482.1°F
	Reactor feed flow (FI-1216A)	131.6 MSCFH

12:43 Liquid injection of nozzle N_2, center of reactor.

13:05 Liquid injection at nozzle N2, wall of reactor.

13:35 Reactor press at 510 psig. Reactor feed flow at 130.3 MSCFH. 01.20 discharge temp. = 154.2°F. Unable to increase flow without raising 01.20 discharge temp. Decided to keep pressure at present value (rather than increasing to orig. target of 520 psig) to "maximize" gas velocity.

13:48 Day tank transfer 122" to 20". 1797 gal into trailer #7323.

14:02 Liquid injection at nozzle N1, reactor center.

14:36 Liquid injection at nozzle N1, reactor wall.

14:48 Reactor pressure 510.1 psig

Reactor avg. temp. 481.5°F
Reactor feed flow 129.3 MSCFH
01.20 discharge temp. 151.4°F

14:59 Nuke moved to 480" and traverse locked out. Seeing vapor at 480".

15:13 Oil levels
21.11 31 3/4 (25.41% lvl. xmtr.) 36.1 gal.
27.14 6 nuts (26.28% lvl. xmtr)
Put reactor level control in auto to bring reactor level back up to 480".

16:57 21.11 22 1/2 (13.5% lvl xmtr.) 26.5 gal
27.14 6 nuts (26.63% lvl. xmtr.)
Reactor 480"
Have added 9.6 gal oil into reactor to bring level back up to 480"
Compare to nuke scans performed under similar conditions (AF-R14.3)
At reactor level = 480", cat conc. = 44.3 wt% oxide
Oil amts = 1456 lb oil
Gas holdup = 49.0 vol%
Amt oil missing from reactor at 15:13 = 9.6 gal
Oil density at 350°F = 46.81 lb/ft3

$$(9.6\ gal.)\frac{1\ ft\,3}{7.4805\ gal}\frac{48.81\ lb}{ft3} = 60.07\ lb\ oil$$

Therefore at 15:13, cat. conc. was $\frac{1158\ lb\ oxide}{1395\ lb\ oil + 1158\ lb\ oxide} = 45.3\%$

Amount of gassed oil equivalent to 60.0 lb oil at 482°F

$$(60.0\ lb\ oil)\frac{ft\,3}{43.83\ lb} = 1.369\ ft3\ oil$$

$$(1.369\ ft3\ oil)\left(\frac{49.0\ ft3\ gas}{100 - 49.0\ ft3\ slurry}\right) = 1.315\ ft3\ gas$$

$$\therefore 60.0\ lb\ oil = 1.369 + 1.315 = 2.68\ ft3\ \text{gassed}\ oil$$

$$\frac{2.68\ ft3}{(3.1416/4)(1.5\ ft)2}\frac{12in}{1ft} = 18.2"$$

At 15:13, reactor level was ≈ 480 - 18.2" = 462"
This election compares well to the observation that the trace study detector at 458" (on tape) saw significant fluctuations in radiation intensity (counts).

18:22 Nuke scan at cond AF-R14.8

19:05 Changing condition to R14.9

19:40 At R14.9 conditioning

Rxt temp = 483.6°F	Feed		
Rxt temp = 747.4 psig	H_2	39.7%	
Rxt level = 370.4"	CO	52.2%	
H2 flow = 9.8 MSCFH	N_2	2.1%	
CO flow = 14.2 MSCFH	CO_2	5.6%	
FI1216C → 28.2 MSCFH			

20:30 H_2 10 → 9.6 MSCFH
CO 14.3 → 13.7 MSCFH
FI1216C → 27 MSCFH

20.39 H2 9.6 → 9.2
CO 13.7 → 13.1 FI1216C → 26 MSCFH
Nuke, Rxt temp and DP readings taken at 21:00, 22:00 and 23:00. All readings stable.

23:10 Shutting down !!!!
Cooling down under syngas
60°F /hr (15°F/15 mins)
Rxt Pr at 750 psig
Flow at 26 MSCFH

25 June 1995

00:20 Rxt temp at 400°F switching to N_2, dropping Rxt Pr.

00:40 Stopped pumpback from 27.14 and 21.11.

05:00 Day tank transferred 76" → 0" (1234 gallons). Done with MeOH transfer. All MeOH drained from product collection drain.

07:45 Drained 27.14 into separate drum. Oil looked clear.

07:55 Drained 21.11 into separate drum. The first ~20 sec. of flow looked like black slurry. The remainder looked cleaner.

08:15 Added clear flush oil to 27.14 filled to 17 nuts. This oil will be pumped to 21.11 and then to 27.20.

26 June 1995

11:00 Looked at drum containing 21.11 drain (6/25/95 Alternate Catalyst). ~ 1/2" layer of MeOH on top (clear). Remainder bottom layer is oil (clear). Small (~ 1/2"?) layer of catalyst particles (gritty) settled) out on bottom. Drum containing 27.14 drain (6/25/95 Alternate Catalyst). "Liquid" at bottom of drum. Mixture of MeOH and oil with dispersion of catalyst fires. "Liquid" at top of drum: black-gray colored MeOH (catalyst dust?)

6/26/95 Spent Slurry (Alternate Catalyst) drained from 27.20 on 6/25/95

Drums labeled:

"27.20 Drain 'Alternate Catalyst'. Run 6/25/95" (Drums 6, 7 mis-labeled 6/26/95)

Drum #	(with drum lifter Full Drum	(with drum lifter) Empty Drum	Slurry
1	559 lb	117 lb	422 lb
2	572 lb	117 lb	455 lb
3	600 lb	117 lb	483 lb
4	579 lb	117 lb	462 lb
5	574 lb	117 lb	457 lb
6	592 lb	117 lb	475 lb
7	260 lb	117 lb	143 lb
			Total slurry 2,917 lb

11 July 1995

DP Calibration

Water DP test on reactor:

Empty

PDT-1500	0.07
PDT-1501	0.11
PDT-1502	0.06
PDT-1503	-0.08
PDT-1504	0.11
PDT-1505	-0.03

Water level = 518" on inside indicator

PDT-1500	2.15
PDT-1501	3.15
PDT-1502	4.29
PDT-1503	10.57
PDT-1504	6.49
PDT-1505	4.28
PDT-1241	20.83

Distances between tapes:

I2 to C	45"
C to N_2	86.5"
N_2 to D	119.75"
D to N_1	120.5"
N_1 to E	120"
E to NDG	50"