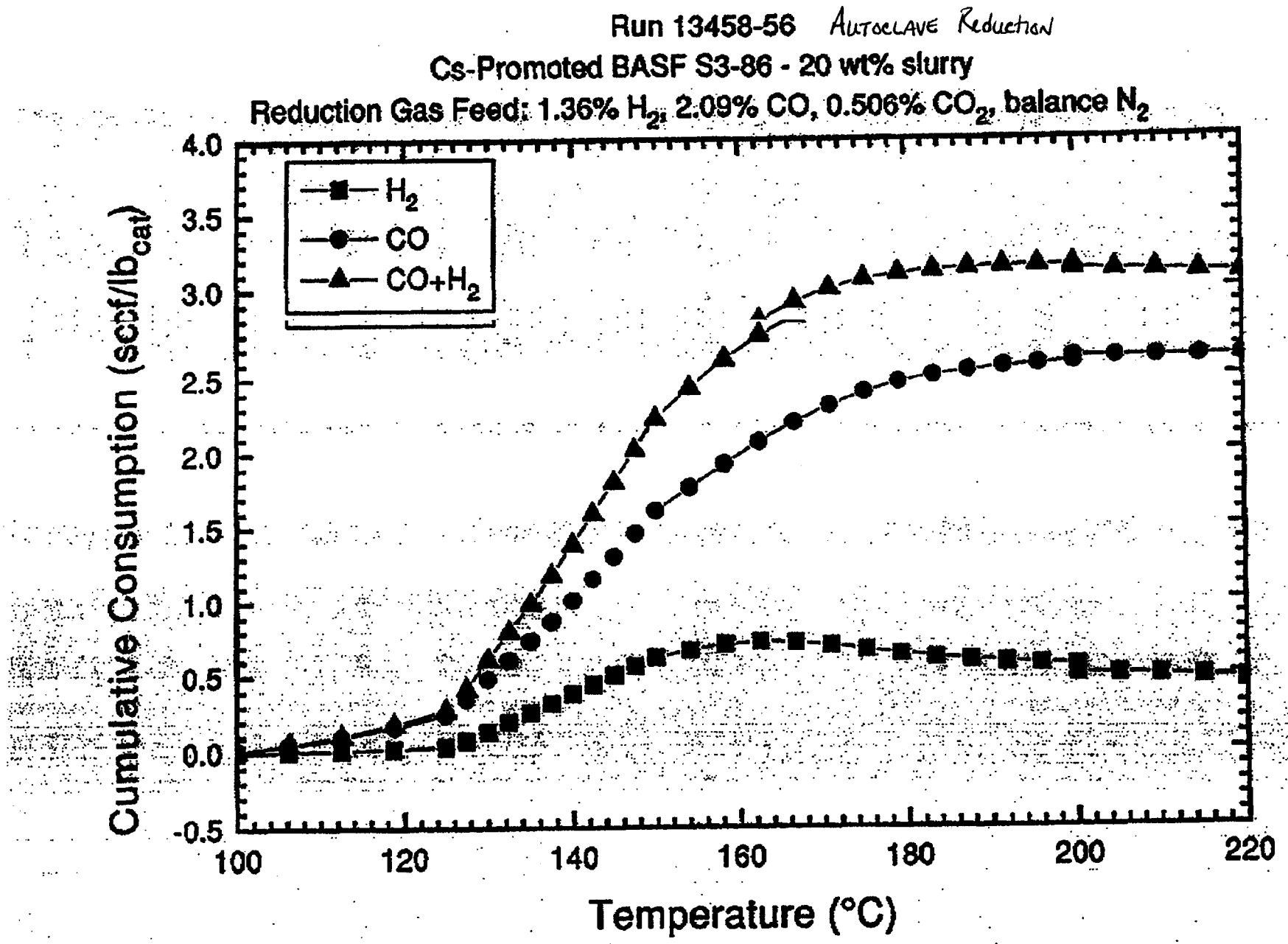
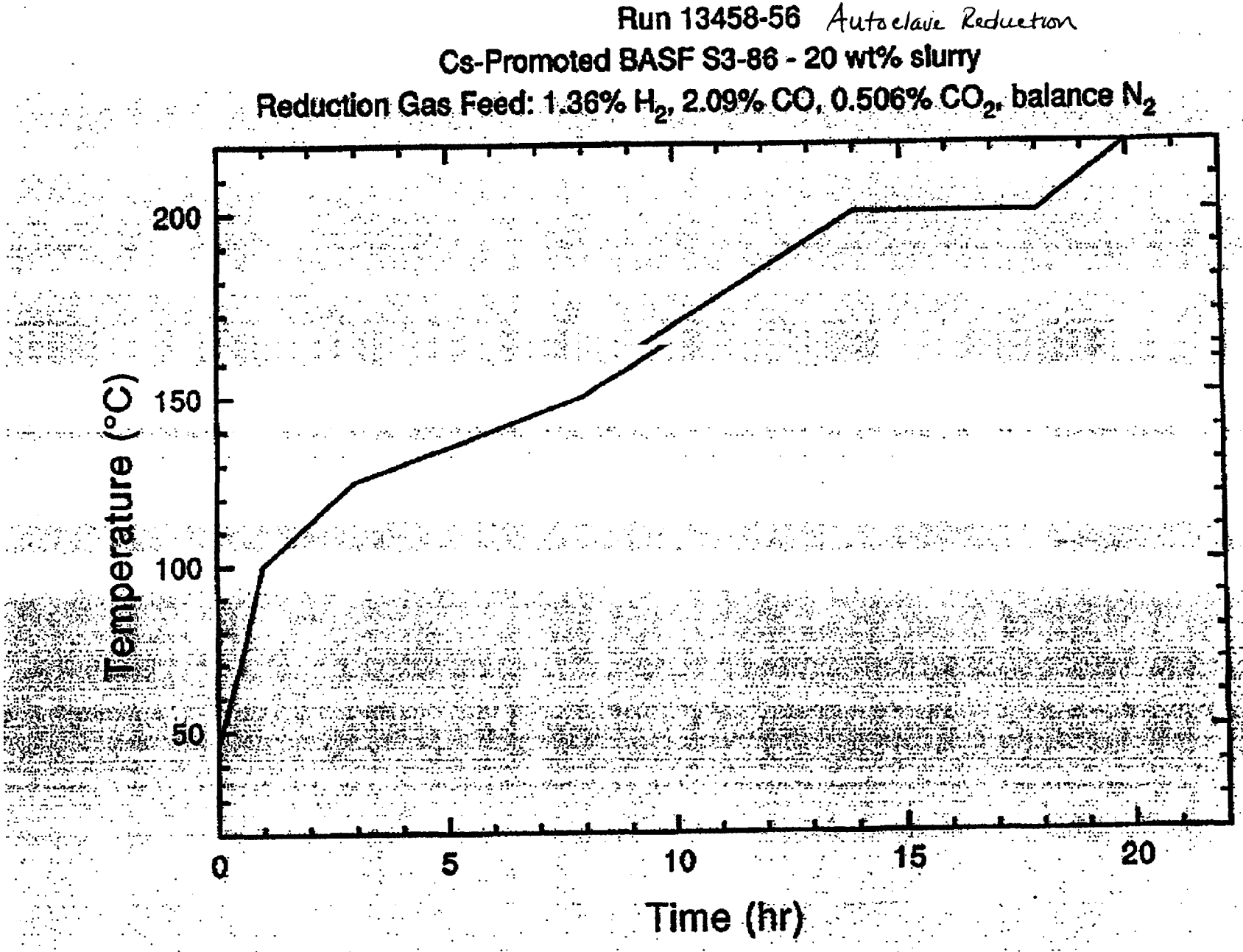
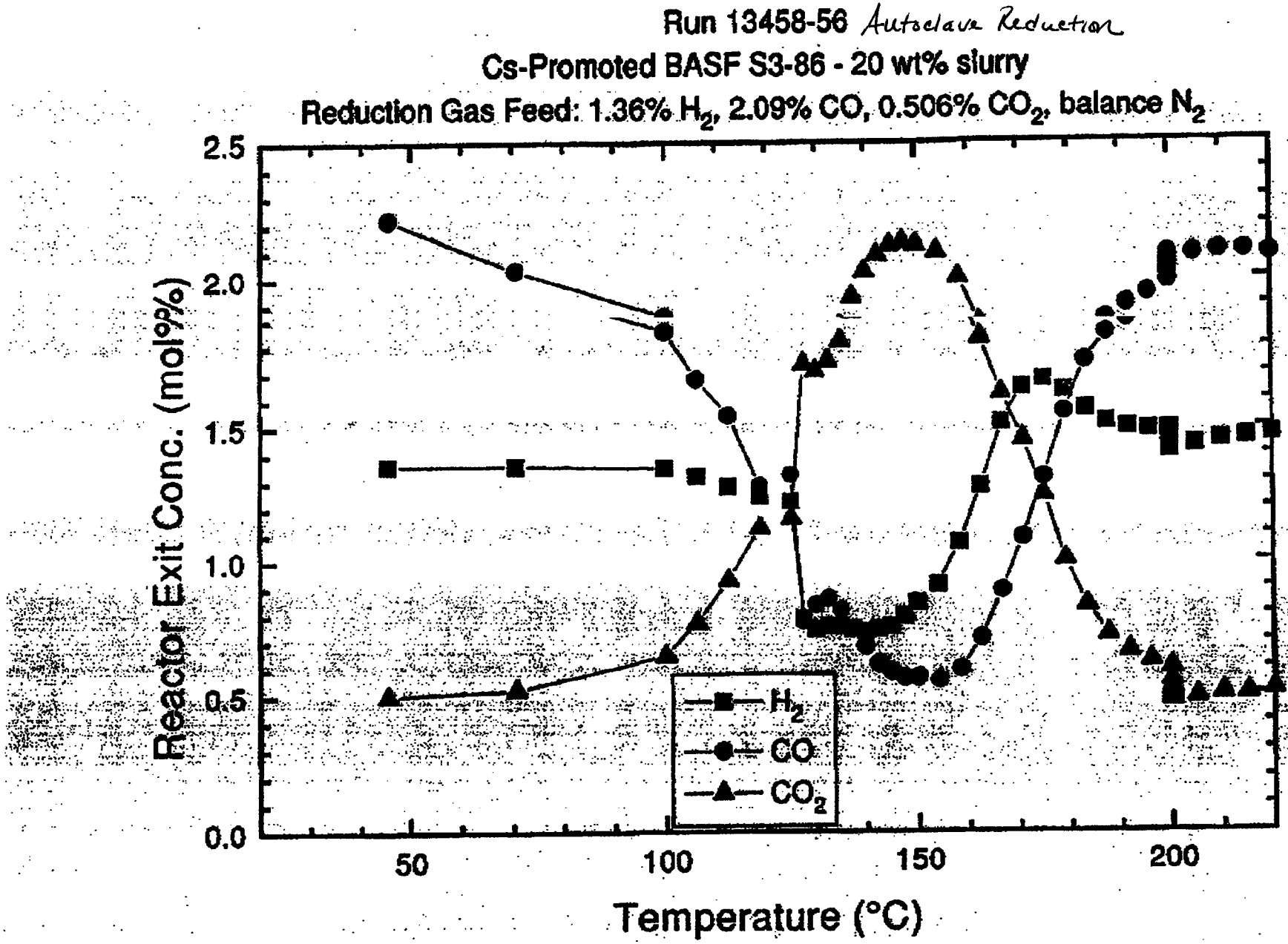
RUN PLAN FOR SPRING ..94 IBOH DEMONSTRATION

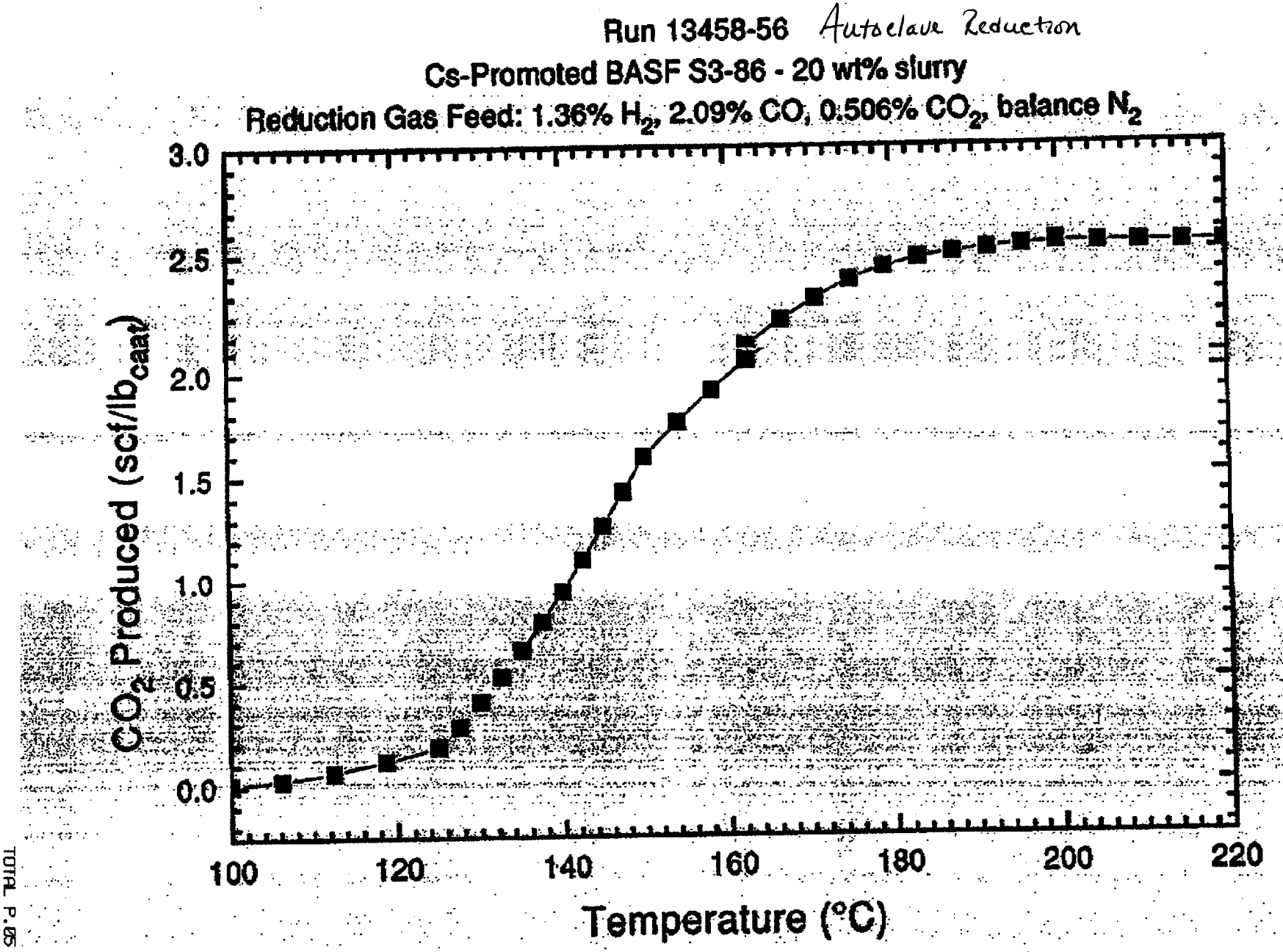
RUN				AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description		---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
21.11 Feed/Product Exchanger															
Feed Inlet Temp	TI-1257	F		---	154.1	161.2	147.3	150.0	150.0	150.0	150.0	120.0	120.0	154.1	150.0
Feed Outlet Temp	TI-1263	F		---	492.8	492.0	492.3	492.2	492.2	492.2	492.2	492.0	492.2	492.8	492.2
Total Feed to 02.63 Temp	TI-1216	F		---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	475.8	423.7	437.8	431.5
Reactor Eff. Inlet Temp	TI-1262	F		---	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0
Reactor Eff. Outlet Temp	TIC-1260	F		---	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0
Reactor Eff. Dew Temp	---	F		---	198.9	219.5	186.8	238.7	262.5	275.7	247.3	279.8	263.8	198.9	245.7
REACTOR EFFLUENT															
Total Flow	FI-196	scfh		---	86,318	49,328	143,328	131,379	124,583	43,512	78,551	93,645	83,183	86,318	47,959
H2	---	mol%		---	23.47%	22.60%	24.22%	18.26%	14.48%	13.20%	17.47%	22.24%	18.88%	23.47%	22.64%
CO	---	mol%		---	65.14%	64.82%	65.96%	65.95%	66.18%	63.55%	64.92%	55.69%	61.33%	65.14%	47.28%
N2	---	mol%		---	0.89%	0.82%	0.76%	0.62%	0.67%	0.73%	0.79%	0.66%	1.15%	0.89%	1.19%
CO2	---	mol%		---	5.37%	6.03%	4.54%	6.13%	7.18%	10.72%	7.42%	8.00%	7.89%	5.37%	18.30%
MEOH	---	mol%		---	2.98%	2.79%	3.05%	6.25%	8.33%	6.93%	5.92%	8.60%	6.61%	2.98%	7.24%
ETOH	---	mol%		---	0.13%	0.14%	0.11%	0.26%	0.38%	0.47%	0.31%	0.59%	0.43%	0.13%	0.32%
ETOH	---	mol%		---	0.13%	0.14%	0.11%	0.26%	0.38%	0.47%	0.31%	0.59%	0.43%	0.13%	0.32%
PROH	---	mol%		---	0.10%	0.14%	0.07%	0.19%	0.28%	0.50%	0.27%	0.89%	0.60%	0.10%	0.21%
C4OH	---	mol%		---	0.03%	0.04%	0.02%	0.05%	0.07%	0.15%	0.07%	0.22%	0.17%	0.03%	0.05%
IBOH	---	mol%		---	0.16%	0.30%	0.10%	0.18%	0.22%	0.46%	0.27%	0.41%	0.37%	0.16%	0.16%
C5OH+	---	mol%		---	0.26%	0.51%	0.16%	0.29%	0.34%	0.70%	0.43%	0.54%	0.52%	0.26%	0.24%
C1	---	mol%		---	1.03%	1.31%	0.65%	1.35%	1.32%	1.82%	1.56%	1.54%	1.53%	1.03%	1.20%
				---	*99.54%*	*99.50%*	*99.62%*	*99.53%*	*99.44%*	*99.22%*	*99.43%*	*99.38%*	*99.46%*	*99.54%*	*98.84%*
PRODUCT RECOVERY															
Syngas to Backend Flow	FI-682	scfh		---	83,669	47,596	139,238	122,473	112,907	39,498	73,105	83,040	76,086	83,669	None
22.11 to Flare Flow	FI-237	scfh		---	94	75	126	360	525	243	252	497	337	94	304
Main Flare Flow	FI-245	scfh		---	3,867	4,004	4,431	4,157	4,253	3,867	3,960	4,292	3,867	3,867	5,605
Product Flow	---	gpd		---	982	725	1,417	3,037	3,941	1,504	1,957	3,884	2,644	982	1,267
BACK-END															
MEOH Circulation	FIC-814	gpm		None	9.17	7.90	11.56	14.66	14.83	8.46	10.43	13.94	13.88	9.17	None
MEOH to 07.10 Temp	TI-814	F		---	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	---
07.10 OH Temp	TI-1275	F		---	-4.6	-7.2	-3.1	-3.8	-2.9	-5.5	-3.5	-4.2	-5.8	-4.6	---
07.20 OH to Flare Flow	FI-7291	scfh		---	2,288	2,377	2,419	4,332	5,046	4,079	3,652	5,356	5,218	2,288	---
07.20 Reboiler Temp	TIC-7339	F		---	296.8	296.5	296.9	296.4	296.3	296.4	296.4	296.5	296.6	296.8	---
07.22 Steam Pressure	PIC-7338	psig		---	235	235	235	235	235	235	235	235	235	235	---
07.22 Steam Usage	FI-7338	lb/hr		---	592.8	592.5	576.8	664.0	692.7	679.2	650.4	710.9	692.0	592.8	---
21.80 CO2 Usage	---	TPD		---	36.18	33.82	36.29	36.50	36.83	35.72	35.49	36.15	33.60	36.18	---
Total CO2 Usage	Fd+21.80	TPD		0.16	36.22	33.82	36.29	36.50	36.83	36.55	35.49	36.15	34.50	36.22	0.00
RECYCLE FEED															
H2	---	mol%		---	25.00%	24.65%	25.51%	20.38%	16.77%	16.18%	19.80%	26.80%	22.14%	25.00%	24.73%
CO	---	mol%		---	68.99%	70.09%	69.20%	73.09%	76.07%	76.84%	72.95%	66.39%	71.17%	68.99%	51.71%
N2	---	mol%		---	0.94%	0.90%	0.80%	0.69%	0.77%	0.89%	0.89%	0.80%	1.34%	0.94%	1.31%
CO2	---	mol%		---	3.69%	2.67%	3.58%	4.06%	4.58%	3.55%	4.27%	3.90%	3.31%	3.69%	19.52%
MEOH	---	mol%		---	0.05%	0.04%	0.05%	0.05%	0.05%	0.05%	0.05%	0.05%	0.04%	0.05%	0.84%
C1	---	mol%		---	1.05%	1.38%	0.64%	1.45%	1.46%	2.12%	1.71%	1.78%	1.72%	1.05%	1.12%
				---	*99.72%*	*99.72%*	*99.78%*	*99.73%*	*99.70%*	*99.62%*	*99.67%*	*99.72%*	*99.73%*	*99.72%*	*99.22%*

JMRepasky 3/3/94


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
H2
CO
CO+H2
Cumulative Consumption (sccf/lbcat)
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
100
120
140
160
180
200
220
Temperature (°C)


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
Temperature (°C)
200
150
100
50
0
5
10
15
20
Time (hr)


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
Reactor Exit Conc. (mol%)
2.5
2.0
1.5
1.0
0.5
0.0
50
100
150
200
Temperature (°C)
H2
CO
CO2


Run 13458-56 Autoclave Reduction
Cs-Promoted BASF S3-86 - 20 wt% slurry
Reduction Gas Feed: 1.36% H2, 2.09% CO, 0.506% CO2, balance N2
CO2 Produced (scf/lbcat)
3.0
2.5
2.0
1.5
1.0
0.5
0.0
100
120
140
160
180
200
220
Temperature (°C)

TEST AUTHORIZATION # 40

LaPorte AAlternative Fuels Development Unit (AFDU)

Sheet: 1 of 4
Date : 03/02/94
By: ESS

RUN NUMBER: AF-R10
APPROX. START DATE: 25 March, 1994

TITLE: LIQUID-PHASE-ISODBUTANOL PROCESS VARIABLE STUDIES WITH CS-BASF S3-86

OBJECTIVE:
To study the performance of cesiumm promoted methanol catalyst in a bubble column reactor at different space velocities & pressures and wiith different feed gas compositions. Two data periods will include mixed alcohol injection to demonstrtrate increased productivity of isobutanol when lower alcohol products are recycled.

SUMMARY:
Upon completion of the TA #39 (runin AF-A6), 11-12 process variable scans, covering two different reactor feed compositions (Texaco ɔ and Shell), different space velocities and pressures, will be carried-out. The reactor will be maintained d at 572°F throughout the studies.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:
Protective gear including face shielold should be worn during slurry sampling.

ENVIRONMENTAL IMPLICATIONSS:
Minimal.

SPECIAL REMARKS:
See Test Details.

AUTHORIZATIONS:

E. C. H Heydorn, Plant Mgr

E. S. Schaub, Process Engr

TEST DETAILS:

1. Prior to completion of TA #39 (r(run AF-A6) the CO2 removal section is charged with approximately 638 gallons of methanol . Follow the procedures set forth by operations. The breakdown of methanol inventory in the back-c-end is:

07.10	130 gal	(54 gals above normal liquid level of 5 ft)
07.20	207 gal	(61 gals above normal liquid level of 6 ft)
07.22	132 gal	(full)
21.45/2'21.65	117 gal	(full)
piping	52 gal	

The inventory numbers in the 07.10 and 07.20 include the normal sump liquid level plus the liquid which will be held up in the packicking once the 10.80 circulation pump is started. Upon completion of the TA #39 (run AF-A6), the 22.10, 22.15 and 22.16 should be empty, the reactor loop should be de-pressurized, and the CO2 removal section should be blocked-in.

2. Increase the reactor pressure tdo 750 psig and introduce syngas while the reactor is approximately 430-460 F. Initially control the s slurry temperature at 482°F. Slowly increase the reactor feed rate to 25,000 SCFH. The reactor fefeed composition should correspond to run AF-R10.1 conditions (Shell gas) and the effluent shoould be directed to flare (once-through operation). Slowly ramp the reactor temperature up to the tatarget of 572 F.

3. Once reactor feed flows have bbeen established, bring the CO2 removal section on-line. When the CO2 removal section is functionning properly, recycle flow may be slowly brought in and the fresh feed flows reduced so that they y match the target in the table for AF-R10.1 The process variable scans may begin.

4. PVS RUNS:

Process and control room targetets are tabulated in the attached tables. The run descriptors are presented below:

RUN No.	Gas Type	Space Velocity	Pressure (psig)	Estimated No. of Days
AF-R10.1	SHELL	5000	750	2
AF-R10.2	SHELL	3000	750	1
AF-R10.3	SHELL	8200	750	2
AF-R10.4	SHELL	8200	1300	2
AF-R10.5	SHELL	8200	1735	2
AF-R10.6	SHELL	3000	1735	1
AF-R10.7	SHELL	5000	1300	1
AF-R10.8	SHELL with fufull alcohol l injection	5000	1300	1

AF-R10.9	SHELL with partial alcohol injection	5000	1300	1
AF-R10.10	SHELL	5000	750	1
AF-R10.11	TEXACO	3000	1300	2

For each run condition a period of roughly 24 hours of steady operation constitutes a data period. Run 10.10 is optional and will be conducted if the schedule permits.

6. SPECIAL CONSIDERATIONS:

ALCOHOL INJECTION: For cases AF-R10.8 and AF-R10.9, the 10.95 pump will be used to inject alcohols into the reactor syngas feed. Before these runs start, steam flow to the 02.63 heater must be started and TSLL-12253 must be commissioned. Both of these steps will ensure that the liquids which are injected will be vaporized before entering the reactor.

CO2 REMOVAL OPERATION: The CO2 removal system will be operated during cases AF-R10.1-10.10 (Shell). Following AF-RR10.10, it will be shut down and blocked in for the final case, AF-R10.11 (Texaco).

WATER BUILD-UP IN CO2 REMOVAL SECTION: Water and higher alcohols enter the back-end of the plant through the vapor off the 22.10 and remain. Additionally, methanol escapes from the back-end with the vapors off the 07.20. As a result, the concentration of water in the circulating methanol increases with time-on-stream. Water is a poor solvent for CO2 and the effectiveness of the circulating liquid will diminish with time. Therefore, it will be necessary to increase 10.80 flow and/or increase plant purge during any given run. The composition of the methanol solvent in the CO2 removal section will be monitored regularly and, if necessary, methanol will be drained out (via the 22.11 and 22.15) and fresh methanol will be added using the 10.85 pump.

WATER FREEZE-OUT IN 21.10 TUBESIDE: Although unlikely, it is possible for the water contained in the 22.10 overhead to plate-out on the tubes of the 21.10. This process, if it occurs at all, will be slow. Indications of freeze-out include: excessive pressure drop, colder temperature at TI-233, and warmer temperature at TI-188. Corrective action is to operate the 10.85 pump to inject methanol into the 21.100 tubeside inlet.

7. Upon completion of the PVSs, this test run is done. De-pressurize the plant and proceed with shut-down.

ANALYTICAL COMMENTS:

1. Catalyst sampling requirements:

   - slurried catalyst at end-of-run.

   Exact quantities to be determined by operations, process, and research.

2. Continuous composition sampapling requirements (GC):

- fresh feed,
- reactor in, (sample pooint 4 and 15, when injecting alcohols)
- reactor out,
- 22.10 overhead,
- recycle
- 07.20 offgas

3. Periodic composition samplinqg requirements (GC):

- 22.11 off-gas (frequeiency to be determined by operations & process)

Periodic composition samplinqg requirements (LC):

- higher alcohol producct (every 8 hours)
- 10.80 suction liquid ((once every two days or as required)

4. Flow measurement requiremeents:

- fresh feed,
- 10.95 pump
- reactor in,
- reactor out,
- 22.10 overhead,
- recycle,
- purge,
- 22.11 off-gas,
- 07.20 off-gas,
- mixed alcohol product (by level)
- 10.80 flow,

REFERENCES:

1. TEST AUTHORIZATION #20 - Proocedures for reactor standby during shutdown.

2. STANDARD STARTUP PROCEDUURES FOR MeOH-ONLY OPERATION

3. STARTUP PROCEDURES FOR OOPERATION WITH CO2 REMOVAL.

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN			AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description	---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
Duration	---	days	2	2	1	2	2	2	1	1	1	1	1	2
Syngas	---	---	---	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	TEXACO
Inlet Space Velocity	---	sL/kg-hr	1,200	5,000	3,000	8,200	8,200	8,200	3,000	5,000	5,000	5,000	5,000	3,000
Reactor Pressure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
REACTOR														
Pressure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
Temperature	TI-1233	F	---	572	572	572	572	572	572	572	572	572	572	572
Heat Duty		MM BTU/hr	---	0.53	0.52	0.59	1.48	1.97	1.01	1.12	0.85	1.03	0.53	0.59
Inlet Superficial Velocity	---	ft/sec	0.640	0.611	0.366	1.002	0.583	0.438	0.160	0.355	0.390	0.366	0.611	0.213
Outlet Superficial Velocity	---	ft/sec	---	0.517	0.348	0.859	0.461	0.329	0.115	0.275	0.327	0.291	0.517	0.168
Liquid Level	LI-2142	% span	80 - 90%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
Catalyst Load	---	lb	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107
Cat Weight Fraction	---	%	40.0%	40.2%	39.1%	41.5%	42.0%	42.2%	39.8%	40.7%	41.1%	42.0%	40.2%	39.6%
Cat Weight Fraction	---	%	40.0%	40.2%	39.1%	41.5%	42.0%	42.2%	39.8%	40.7%	41.1%	42.0%	40.2%	39.6%
Vapor Void Fraction	---	%	25 - 30%	40.2%	36.4%	44.3%	46.0%	46.9%	38.7%	41.7%	43.2%	44.4%	40.2%	38.2%
FEED FLOWS														
LP H2	FIC-101	scfh	321	9,440	7,082	13,518	13,936	9,450	12,104	15,799	9,131	13,932	9,440	10,605
CO	FIC-104	scfh	481	10,016	9,086	12,582	20,790	25,311	13,715	15,670	14,682	15,712	10,016	9,476
CO2	FIC-107	scfh	114	31	0	0	0	0	607	0	0	659	31	0
N2	FIC-111	scfh	22,003	49	54	56	74	73	71	60	66	65	49	75
01.10 Total Flow	FI-726	scfh	22,919	19,536	16,222	26,157	34,800	34,835	26,497	31,529	23,879	30,367	19,536	20,156
HP H2	FIC-1200	scfh	0	0	0	0	10,019	20,322	0	0	0	0	0	0
01.20 Recycle	FIC-246	scfh	0	76,849	40,926	131,169	112,966	102,604	31,305	64,926	72,840	66,495	76,849	38,114
10.95 PUMP INJECTION														
Total Flow	FI-1221	gpm	zero	zero	zero	zero	zero	zero	zero	zero	2.14	0.70	zero	zero
MEOH	---	wt%	---	---	---	---	---	---	---	---	83.00%	69.50%	---	---
C2OH	---	wt%	---	---	---	---	---	---	---	---	5.00%	10.50%	---	---
C3OH	---	wt%	---	---	---	---	---	---	---	---	12.00%	20.00%	---	---
											100.00%	*100.00%*		
REACTOR FEED														
Target Feed Temp	TI-1253	F	---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	400.0	400.0	437.8	431.5
Feed Dewpoint	---	F	---	-6.6	-9.3	-5.2	-9.6	-14.3	-15.1	-9.4	239.5	178.4	-6.6	92.9
Total Dry Flow	FI-1216	scfh	22,920	96,375	57,133	157,308	157,750	157,727	57,770	96,434	96,692	96,859	96,375	58,268
H2	---	mol%	1.40%	29.73%	30.05%	29.87%	29.78%	29.79%	29.72%	29.71%	27.03%	28.73%	29.73%	34.37%
CO	---	mol%	2.10%	65.41%	66.11%	65.70%	65.52%	65.53%	65.38%	65.37%	59.46%	63.21%	65.41%	50.09%
N2	---	mol%	96.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%	1.00%
CO2	---	mol%	0.50%	2.97%	1.91%	2.99%	2.91%	2.98%	2.97%	2.88%	2.68%	2.87%	2.97%	12.77%
MEOH	---	mol%	0.00%	0.04%	0.03%	0.04%	0.04%	0.03%	0.03%	0.03%	7.89%	2.32%	0.04%	0.55%
ETOH	---	mol%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.33%	0.24%	0.00%	0.01%
PROH	---	mol%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.00%	0.61%	0.35%	0.00%	0.00%
C1	---	mol%	0.00%	0.84%	0.99%	0.53%	1.04%	0.95%	1.15%	1.15%	1.23%	1.15%	0.84%	0.73%
			100.00%	*99.98%*	*100.09%*	*100.13%*	*100.28%*	*100.28%*	*100.25%*	*100.14%*	*100.22%*	*99.87%*	*99.98%*	*99.53%*

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

RUN			AF-A6	AF-R10.1	AF-R10.2	AF-R10.3	AF-R10.4	AF-R10.5	AF-R10.6	AF-R10.7	AF-R10.8	AF-R10.9	AF-R10.10	AF-R10.11
Description	---	---	Reduction	IBOH-1	IBOH-2	IBOH-3	IBOH-4	IBOH-5	IBOH-6	IBOH-7	IBOH-8	IBOH-9	IBOH-10	IBOH-11
21.11 Feed/Product Exchanger														
Feed Inlet Temp	TI-1257	F	---	154.1	161.2	147.3	150.0	150.0	150.0	150.0	120.0	120.0	154.1	150.0
Feed Outlet Temp	TI-1263	F	---	492.8	492.0	492.3	492.2	492.2	492.2	492.2	492.0	492.2	492.8	492.2
Total Feed to 02.63 Temp	TI-1216	F	---	437.8	443.2	431.7	429.8	429.6	429.5	429.4	475.8	423.7	437.8	431.5
Reactor Eff. Inlet Temp	TI-1262	F	---	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0	572.0
Reactor Eff. Outlet Temp	TIC-1260	F	---	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0	280.0
Reactor Eff. Dew Temp	---	F	---	198.9	219.5	186.8	238.7	262.5	275.7	247.3	279.8	263.8	198.9	245.7
REACTOR EFFLUENT														
Total Flow	FI-196	scfh	---	86,318	49,328	143,328	131,379	124,583	43,512	78,551	93,645	83,183	86,318	47,959
H2	---	mol%	---	23.47%	22.60%	24.22%	18.26%	14.48%	13.20%	17.47%	22.24%	18.88%	23.47%	22.64%
CO	---	mol%	---	65.14%	64.82%	65.96%	65.95%	66.18%	63.55%	64.92%	55.69%	61.33%	65.14%	47.28%
N2	---	mol%	---	0.89%	0.82%	0.76%	0.62%	0.67%	0.73%	0.79%	0.66%	1.15%	0.89%	1.19%
CO2	---	mol%	---	5.37%	6.03%	4.54%	6.13%	7.18%	10.72%	7.42%	8.00%	7.89%	5.37%	18.30%
MEOH	---	mol%	---	2.98%	2.79%	3.05%	6.25%	8.33%	6.93%	5.92%	8.60%	6.61%	2.98%	7.24%
ETOH	---	mol%	---	0.13%	0.14%	0.11%	0.26%	0.38%	0.47%	0.31%	0.59%	0.43%	0.13%	0.32%
ETOH	---	mol%	---	0.13%	0.14%	0.11%	0.26%	0.38%	0.47%	0.31%	0.59%	0.43%	0.13%	0.32%
PROH	---	mol%	---	0.10%	0.14%	0.07%	0.19%	0.28%	0.50%	0.27%	0.89%	0.60%	0.10%	0.21%
C4OH	---	mol%	---	0.03%	0.04%	0.02%	0.05%	0.07%	0.15%	0.07%	0.22%	0.17%	0.03%	0.05%
IBOH	---	mol%	---	0.16%	0.30%	0.10%	0.18%	0.22%	0.46%	0.27%	0.41%	0.37%	0.16%	0.16%
C5OH+	---	mol%	---	0.26%	0.51%	0.16%	0.29%	0.34%	0.70%	0.43%	0.54%	0.52%	0.26%	0.24%
C1	---	mol%	---	1.03%	1.31%	0.65%	1.35%	1.32%	1.82%	1.56%	1.54%	1.53%	1.03%	1.20%
			---	*99.54%*	*99.50%*	*99.62%*	*99.53%*	*99.44%*	*99.22%*	*99.43%*	*99.38%*	*99.46%*	*99.54%*	*98.84%*
PRODUCT RECOVERY														
Syngas to Backend Flow	FI-682	scfh	---	83,669	47,596	139,238	122,473	112,907	39,498	73,105	83,040	76,086	83,669	None
22.11 to Flare Flow	FI-237	scfh	---	94	75	126	360	525	243	252	497	337	94	304
Main Flare Flow	FI-245	scfh	---	3,867	4,004	4,431	4,157	4,253	3,867	3,960	4,292	3,867	3,867	5,605
Product Flow	---	gpd	---	982	725	1,417	3,037	3,941	1,504	1,957	3,884	2,644	982	1,267
BACK-END														
MEOH Circulation	FIC-814	gpm	None	9.17	7.90	11.56	14.66	14.83	8.46	10.43	13.94	13.88	9.17	None
MEOH to 07.10 Temp	TI-814	F	---	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	-15.0	---
07.10 OH Temp	TI-1275	F	---	-4.6	-7.2	-3.1	-3.8	-2.9	-5.5	-3.5	-4.2	-5.8	-4.6	---
07.20 OH to Flare Flow	FI-7291	scfh	---	2,288	2,377	2,419	4,332	5,046	4,079	3,652	5,356	5,218	2,288	---
07.20 Reboiler Temp	TIC-7339	F	---	296.8	296.5	296.9	296.4	296.3	296.4	296.4	296.5	296.6	296.8	---
07.22 Steam Pressure	PIC-7338	psig	---	235	235	235	235	235	235	235	235	235	235	---
07.22 Steam Usage	FI-7338	lb/hr	---	592.8	592.5	576.8	664.0	692.7	679.2	650.4	710.9	692.0	592.8	---
21.80 CO2 Usage	---	TPD	---	36.18	33.82	36.29	36.50	36.83	35.72	35.49	36.15	33.60	36.18	---
Total CO2 Usage	Fd+21.80	TPD	0.16	36.22	33.82	36.29	36.50	36.83	36.55	35.49	36.15	34.50	36.22	0.00
RECYCLE FEED														
H2	---	mol%	---	25.00%	24.65%	25.51%	20.38%	16.77%	16.18%	19.80%	26.80%	22.14%	25.00%	24.73%
CO	---	mol%	---	68.99%	70.09%	69.20%	73.09%	76.07%	76.84%	72.95%	66.39%	71.17%	68.99%	51.71%
N2	---	mol%	---	0.94%	0.90%	0.80%	0.69%	0.77%	0.89%	0.89%	0.80%	1.34%	0.94%	1.31%
CO2	---	mol%	---	3.69%	2.67%	3.58%	4.06%	4.58%	3.55%	4.27%	3.90%	3.31%	3.69%	19.52%
MEOH	---	mol%	---	0.05%	0.04%	0.05%	0.05%	0.05%	0.05%	0.05%	0.05%	0.04%	0.05%	0.84%
C1	---	mol%	---	1.05%	1.38%	0.64%	1.45%	1.46%	2.12%	1.71%	1.78%	1.72%	1.05%	1.12%
			---	*99.72%*	*99.72%*	*99.78%*	*99.73%*	*99.70%*	*99.62%*	*99.67%*	*99.72%*	*99.73%*	*99.72%*	*99.22%*

JMRepasky 3/3/94

09:40 Nuke scan at 477,583 SCFH. Level at 255" in 27.20.
P = 751 psig; T = 5588 F; Composition = 31% H2, 68% CO

11:00 Nuke scan at 788,044 SCFH. Level at 246" in 27.20.
P = 755 psig; T = 5611 F

12:00 Nuke scan at 1001,483 SCFH. Level at 263" in 27.20.
P = 757 psig; T = 5655 F

13:35 Nuke scan at 1441,073 SCFH. Level at 313" in 27.20.
P = 753 psig; T = 5633 F; Composition = 30% H2, 68% CO
Close to 40 psi ΔP in n reactor at this flow.

16:50 Nuke scan at 366,190 SCFH. Level at 217" in 27.20.
P = 1305 psig; T = 5770 F
This was minimum coontrollable flow at these conditions
(without going to oncce-thru).

18:07 Nuke scan at 811,127 SCFH. Level at 343" in 27.20.
P = 1300 psig; T = 5667 F

19:28 Nuke scan at 1334,719 SCFH. Level at 259" in 27.20.
P = 1303 psig; T = 5558 F

20:40 Nuke scan at 1008,347 SCFH. Level at 240" in 27.20.
P = 1749 psig; T = 5663 F; Composition = 35% H2, 64% CO

22:04 Nuke scan at 1885,028 SCFH. Level at 246" in 27.20.
P = 1751 psig; T = 5448 F
This was max flow at t these conditions.

22:50 Shutdown test. Level at 63". (Nuke reading at 42" is 9.7 cps/1000)

23:51 Nuke scan at 377,307 SCFH. Level at 125" in 27.20.
P = 1753 psig; T = 5664 F

3/15/94 Tuesday

07:20 Syngas backed c out to 01.10 / 01.20.

07:45 Syngas out of ccompressors. Flowing with N2 to clean up.

07:50 Cooling plant att 60 F/hour from 482 F.

14:30 Blowdown plantit and drain oil. There was evidence of significant fines in the oil, which could be e contributing to carbonyl generation! Transferred 200 gallons of fresh oiil to prep tank for another pass at carbonyl burnout.

15:45 Transfer freshh oil to reactor. Level at 199" (5.7 cps/1000 at 186") at 104 F and 22 psig.

16:15 Started heatupp at 1 F/minute with 02.63 on. Once-thru at 20,000 SCFH.

19:15 GC compositition: 33% H2, 65% CO, 2% N2.

19:25 Changed SP1-1-MW in Bailey to 19.43.

22:00 Utility oil temmperature to 495 F and holding. Reactor temperature still increasing.

22:30 Plant lined ouut at 482 F and 750 psig.

22:52 Nuke scan at 1 21,977 SCFH (once-thru). Level at 370" in 27.20
P = 752 psig; T = 4482 F

3/16/94 Wednesday 06:53 Carbonyl analalysis starts. GC composition: 32% H2, 66% CO, 2% N2. 20,000 SCFH (oncce-thru) at 482 F and 750 psig. 02.63 heater is on.

06:58 0.017 ppm Fe'e at sample point #3A (not seeing any Ni)
07:12 0.016 ppm Fe'e at sample point #3A
07:18 0.016 ppm Fe'e at sample point #3A
07:23 0.007 ppm Fe'e at sample point #15
07:31 0.009 ppm Fe'e at sample point #15
07:36 0.009 ppm Fe'e at sample point #15
07:46 0.002 ppm Fe'e at sample point #4
07:52 0.002 ppm Fe'e at sample point #4

12:45 Plant lined ouut at 1750 psig and 572 F.

Load Drakeol oil too 28.30 prep tank:
Drum #1 = 368 llbs
Drum #2 = 374 llbs
Drum #3 = 380 llbs
Drum #4 = 378 llbs
Drum #5 = 375 llbs
Total = 18755 lbs

18:00 Began recyclele to bring plant to 58,000 SCFH.

20:10 Plant lined ouut at 58,000 SCFH, 1750 psig, and 572 F.

20:25 Nuke scan at 1 58,683 SCFH. Level at 407" in 27.20.

P = 1751 psig; T = 5772 F

23:55 High pressure carbonyl burnout complete. Begin backing out syngas and cooling at 60 F/hour.

3/17/94 Thursday

07:09 Start loading methanol catalyst to prep tank (160 F). Nice sunny day.

Drum #1 = 238 lbs
Drum #2 = 421 lbs
Drum #3 = 411 lbs
Drum #4 = 180 lbs
Total = 1250 lbs

09:00 Catalyst loading complete.

11:48 Prepare flush oil (283 lbs)

12:00 End of transfer to reactor. Level is 291". Pour flush oil into prep tank. Walls very clean, no puddles evident at bottom of tank before adding flush oil.

12:45 Begin transfer of flush oil to reactor. Level ~335".

13:00 Raise reactor pressure toward 50 psig. Level at 355".

14:25 Start bringing gas to 27.20. Starting composition:

1.2%	H2
94.4%	N2
3.2%	CO
1.0%	CO2

Average reactor T=183 F (180 F at bottom). PIC-1247 = 94.9 psig.

No GC points reading into the DEC !

14:47 Flare malfunction (high N2 content in vent gas). Dean rebooting GC computer.

15:10 Flare rupture disk blew. Stopped gas flow to reactor.

17:05 Nuke scan. Level at 309".

19:25 Start bringing reduction gas to 27.20 again. Level at 385".

19:29 Flare malfunction at 22,000 SCFH.

19:30 Flare relit with flow at 20,000 SCFH.

19:32 Starting composition: (G06) 1.04% H2
1.67% CCO
96.73% NN2
0.71% CCO2

Flow at 19,800 SCFFH, 83 psig, level at 423". Waiting for another GC scan.

19:40 New composition: (D06) 1.18% H2
1.92% CCO
96.17% NN2
0.70% CCO2

19:48 Starting heatup.

20:15 Average reactor temperature at 190 F. Still no evidence of uptake yet.

20:30 Lost flare again. Flow had crept up to 21,500 SCFH. Backing flow out to try and relight.

21:00 27.20 reactor temperature has levelled off at 194 F.

21:06 Flow stopped to 27.20. FALL-299.

21:20 Flare relit!

21:27 Re-established syngas flow to reactor.

21:45 Syngas flow set at 13,500 SCFH on FI-299.

22:00 Syngas flow up to 14,300 SCFH. Restarted heatup.

New composition: (G06) 1.06% H2
1.67% CCO
96.29% NN2
0.78% CCO2

22:10 False start on the heatup. The heaters had tripped out during the flare malfunction upset. Now they're racked back in.

23:11 G03 lost its CCO peak. GC's Gary and Dennis show good agreement otherwise.

24:00 Utility oil temperature at 232 F. Reactor temperature at 222 F. Still evidence of uptake in the outlet concentrations, but temperature hasn't taken off yet.

3/18/94 Friday

02:05 Nuke scan. Levvel at 413". Holdup=26.4%.

03:45 Noticed that GCC Gary hadn't been updating at all for over an hour. Checked the lab, and tthe unit was out of paper. Replaced paper and got a new scan (still without CO) at 4:0:05.

06:10 Told Matt to stoop temperature ramp because preliminary calculation showed uptake was fallilling below standard curve.

06:30 Matt restarted tetemperature ramp. E-beth wasn't calculating uptake right.

07:47 Dean took GC GGary off-line to investigate missing CO peak.

07:51 Nuke scan. Levvel at 390". Holdup=25.9%.

09:10 GC Gary calibraated and back on-line. GC Dennis off-line.

10:25 GC Dennis backk on-line. (10:24 reading was calibration gas.)

12:15 Reactor temperarature at 392 F and holding.

15:30 CO inlet concenntration has dropped from about 1.6% to about 1.0% in the last hour. Henry adadjusted back up.

16:00 Downloaded anond plotted cumulative reduction data. Everything still looks OK.

17:25 Nuke scan. Levvel at 354". Holdup=26.8%.

21:00 Downloaded anond plotted another set of cumulative reduction data.

3/19/94 Saturday

00:30 Begin 15 F/hr rereactor temperature ramp toward 464 F.

01:30 Matt increased tthe CO flow slightly and decreased the CO2 in the feed to match desired compposition.

03:00 Nuke scan. Levvel at 328".

03:15 Matt fired up thhe 02.62 reduction heaters to help warm up the reduction feed gas during the temmperature ramp.

03:45 Nuke scan had lclots of variability in the slurry region from 318" down. Readings do not settle c down but constantly flip values so that the recorded number is +/- 0.5 or so.

05:40 Reach 464 F. . Begin hold period.

06:42 Reduction complete. Removing syngas from plant. Reactor will be cooled at 30 F/hr to target 400 F. Low N2 bubbling through reactor.

06:43 TI-1235 (J7 nozzle in reactor) taken off-line for recalibration.

08:00 Oil added to 227.14 (12 nuts).

09:10 Begin blending syngas.

09:22 Stop N2 to 27.20 to take slurry sample. Level at 260" with no gas flow.

10:20 Syngas flow to reactor begins. Plant is pressuring up (PV-201 shut).

10:45 27.20 Avg. TT = 408 F, P = 616 psig. Level at 288" and holding. Pressure is ramping linearly, but temperature has leveled off.

10:53 Starting to increase T. We are at pressure (755 psig). Flow has been about 23,700 SCFH since startup.

11:12 Begin recycling a little. Avg. T = 413 F. Level coming up to 305".

11:22 Start seeing MeOH in reactor effluent (SP3A).

11:45 Starting to take oil from 21.11 to reactor. Level at 311". Avg. T = 425 F.

11:49 Start increasing fresh feed (about 20%) and recycle. Fresh feed is 28,600 SCFH. Reactor feed is 72,600 SCFH. Level at 318".

11:57 GC's are now shooting proper sequence.

12:02 More recycle. Reactor feed is 105,000 SCFH. Level at 347".

12:21 Bringing HP H2 into plant.

12:57 PDSHH-12000 on HP H2 tripped.

13:20 27.20 Avg. TT = 457 F, P = 749 psig. Level at 373".

13:28 About 150 gallons of liquid in 22.10. Opening LV-220 off 22.10.

14:35 Reactor temperatures cool down to 460 F after U.O. fin fan is turned on.

14:50 TIC-293 on U.OO. circuit changed to control off of TI-166 (outlet of fin fan after mixing) instead ofbf TI-293.

15:05 GC Dennis is ofbff-line for recalibration of MeOH peak.

15:49 G05MW is beinng thrown off by ~3% propane peak which Dean believes is H2O being mistakenn as propane. Problem has existed for ~3 1/4 hours. Dean claims to have solved tlthe problem.

16:13 GC Dennis is baack on-line. Both GC's should be OK for awhile now.

16:17 Henry pulls prooduct sample from 22.11. Sample appears highly contaminated with oil ((about 30% by volume). Yellow (oil) on bottom. 5.99% MeOH on SP3A.

16:32 E-beth calculateses 51.4 wt% slurry ! Level down to 317". We will now start bringing level up b by pumping oil from 27.14 / 21.11.

16:38 CW shut off to (01.34.

17:15 TI-1235 & TI-12233 were checked and found to be displaying 4 F high in the Bailey (compared tto calibrated instrument).

18:57 Level up to 420'0". Moved setpoint to 438".

19:32 G03 (SP3A) upoodate. MeOH concentration still dropping. 3.68% now, was about 5% on last sscan.

20:00 D03 (SP3A) upoodate. MeOH concentration is 2.67%, down from 4%. Level up to 465". Movved setpoint to 480".

20:27 27.20 level at 4880" and holding in automatic.

20:40 HP H2 line pressssure has dropped 20 psi in the last 4 hours (860 to 840), 10 psi in the last hour aalone. This makes feed flow difficult to control.

G03 (SP3A) update caame thru. MeOH at 3.79%.

20:55 PDSHH-1200 (HHP H2) tripped again, simultaneous with AAL-7351 (LEL monitor) - same as this s afternoon at 13:00.

22:05 D03 (SP3A) upoodate. MeOH at 3.86%.

22:31 Nuke scan. Calcdc's show 42 wt% slurry and 40 vol% gas holdup.

23:25 Matt grabbed d a liquid sample from 22.11. About 15% oil by volume. Could not detect a phhase separation in the 22.16 sight glass.

23:45 Liquid logs inndicate oil loss rate is 138 gal/day (over previous 2 hrs) and liquid product rate is s 3312 gal/day. This suggests 4% oil in the product.

3/20/94 00:20 Matt droppingg TIC-1260 (21.11 product outlet) setpoint from 260 F to Sunday 250 F to see if that afaffects the oil in the MeOH problems.

02:00 Liquid logs inndicate oil loss rate is 132 gal/day and liquid product rate is 3430 gal/day (very sisimilar to 23:45 results).

02:36 G03 (SP3A) u update. MeOH at 3.71%.

03:00 Matt took a liqiquid sample from 22.11. Oil in bottom similar to the last sample. Reduced TIIC-1260 setpoint to 245 F.

03:20 22.16 day tankk (117") transfer to trailer.

04:00 Liquid logs inndicate oil loss rate is 120 gal/day.

04:30 Nuke scan. CCalc's show 41 wt% slurry and 37 vol% gas holdup. Readings relatively sisteady throughout the liquid.

04:50 PDSHH-12000 and AAL-7351 alarms go on and off continuously.

05:30 PDSHH-12000 and AAL-7351 are back at it again ! It trips and resets itself continuously.

08:30 HP H2 flow rrose over the last 2 hours, and feed is now too rich in H2. Henry is decreasing f flow.

08:56 GC Gary is offff-line for calibration.

09:39 GC Gary backk on-line. GC Dennis taken off-line.

10:20 TIC-1260 founnd to be reading 10-20 F too high in this range. TIC-1260 back in control at 2560 F setpoint.

10:38 GC Dennis baack on-line.

14:20 22.16 day tankk transfer (100 1/8" to 19 5/8"). Begin cleaning oil out of product collection syystem by pushing all liquids thru to 22.16.

15:46 Moved TIC-12260 setpoint from 250 F to 245 F.

17:03 Henry grabbed d a liquid sample from 22.11. Oil still present but significantly reduced. l.

17:10 D03 shot lost itits MeOH peak and CO2 seems abnormally low.

18:06 More PDSHH-[-1200 alarms. HP H2 line pressure has dropped 35 psig in last 1/2 hour. Matt calalled the main plant and found out we're losing HP H2 altogether. 01.10 cannnot handle full flow with all LP H2, so Matt is backing off on total flow to maintain n composition.

18:11 D03 still missining its MeOH peak. D04 isn't totalizing real well either.

18:56 Feed compositition: (G04) 33.53% H2
(pretty much on targeget) 50.10% CO
0.64% N2 2
14.96% COO2

20:31 Nuke scan. 40) wt% slurry and 35 vol% gas holdup. Level at 478".

3/21/94 Monday

01:00 Henry blew dowwn K.O. pots on GC's. Very little liquid came out. Grabbed a liquid samnple from 22.11. There is still an oil layer in the pint jars (~3/8").

01:05 Go to reduce seetpoint on TIC-1260 further and discover that the TIC is closed and temperaturere has been running above setpoint at 251-252 F since the HP H2 tripped out at 18:00. (Good news for the 01:00 sample.

01:10 22.16 day tank t transfer to trailer (91 1/2" to 20").

02:10 Start CW to 01.1.34 cooler to bring down TI-1257 (feed inlet to 21.11) from 150 F to ~132 F wherere it was running before the H2 upset.

03:25 TI-1257 levelecd out at 121 F. TIC-1260 reading 238 F. Valve opening up with setpoint at 24.5 F.

04:30 Nuke scan. 42 ? wt% slurry and 39 vol% gas holdup. Flow has dropped off to 123,000 SCFH. . Start to put HP H2 back into the plant to bring composition and flow v in line.

05:15 Plant is swingining.

07:10 GC Dennis is ooff-line for calibration.

Level is up in the reaactor to 510". Plant has been swinging.