6. Run #6

Current status of the pilot plant, objectives for Run #6 and future plans for pilot plant operations were discussed at a meeting held in the DOE offices in Washington, D.C. on March 23, 1978. The objectives for Run #6 included completing the process variable scan, making a short-term, continuous run on steam-methane reformer gas and catching all available HYGAS synthesis gas during the next HYGAS run.

Personnel from Process Operators, Inc. arrived at the pilot plant on March 20 and began manning the unit with one person per shift for the duration of Run #6. This coverage supplemented the one operator per shift provided by IGT.

Hot oil was circulated for a day to remove all traces of residual catalyst from the lines and vessels. The reactor was then drained and dried with hot nitrogen. On March 24, 860 pounds of tableted catalyst #038-A were charged to the reactor. The dry settled bed height was approximately 4.5 feet.

Heatup for Run #6 catalyst reduction began with nitrogen on March 27. The reactor was maintained at 400^{o}F until hydrogen became available on March 30. Then, reduction began using a blend of 50 percent nitrogen

and 50 percent hydrogen containing 1.8 percent CO. The catalyst bed was heated to a maximum temperature of 800°F and held there for 36 hours. Hydrogen flow was then discontinued, which completed the reduction procedure.

On April 1, oil was introduced into the reactor for the first time. The reactor was maintained at 550°F and 80 psig for two days with no gas flow and only a low oil flow. On April 3, the settled bed height was measured (4.6 ft) and a liquid-only fluidization study was begun. This work was completed two days later. and a fluidization test with nitrogen and oil simulating standard reactor conditions was then run. Results of the liquid-only fluidization study are shown in Figure IV-B-14. Initially, the bed expanded up to 10 percent with increasing liquid velocity. When attempts were made to repeat these data points a few days later, the bed expansion was virtually nil. Evidently, catalyst bridging had occurred and the bed remained in a packed configuration, even at maximum available oil velocities. The addition of gas flow also did not affect the bed height.

Four filters plugged during the first week of oil-only circulation. This was substantially less plugging than had been experienced during the previous run with Calsicat catalyst.

With fluidization tests completed, the pilot plant was placed in a standby mode since no gas was available for methanation. Reactor conditions were 550°F, 100 psig and 145 GPM oil flow until hour 376, when system pressure was gradually raised to 500 psig in anticipation of the introducton of feed gas. The major events of Run #6 are summarized in Table IV-B-12.

FIGURE IV-B-14

BED POROSITY VS. LIQUID VELOCITY

LPM PILOT PLANT - RUN # 6

CATALYST # 038-A
FREEZENE - 100 OIL
NO GAS FLOW

NOTE: BASED ON 4.6 FT. SETTLED BED HEIGHT

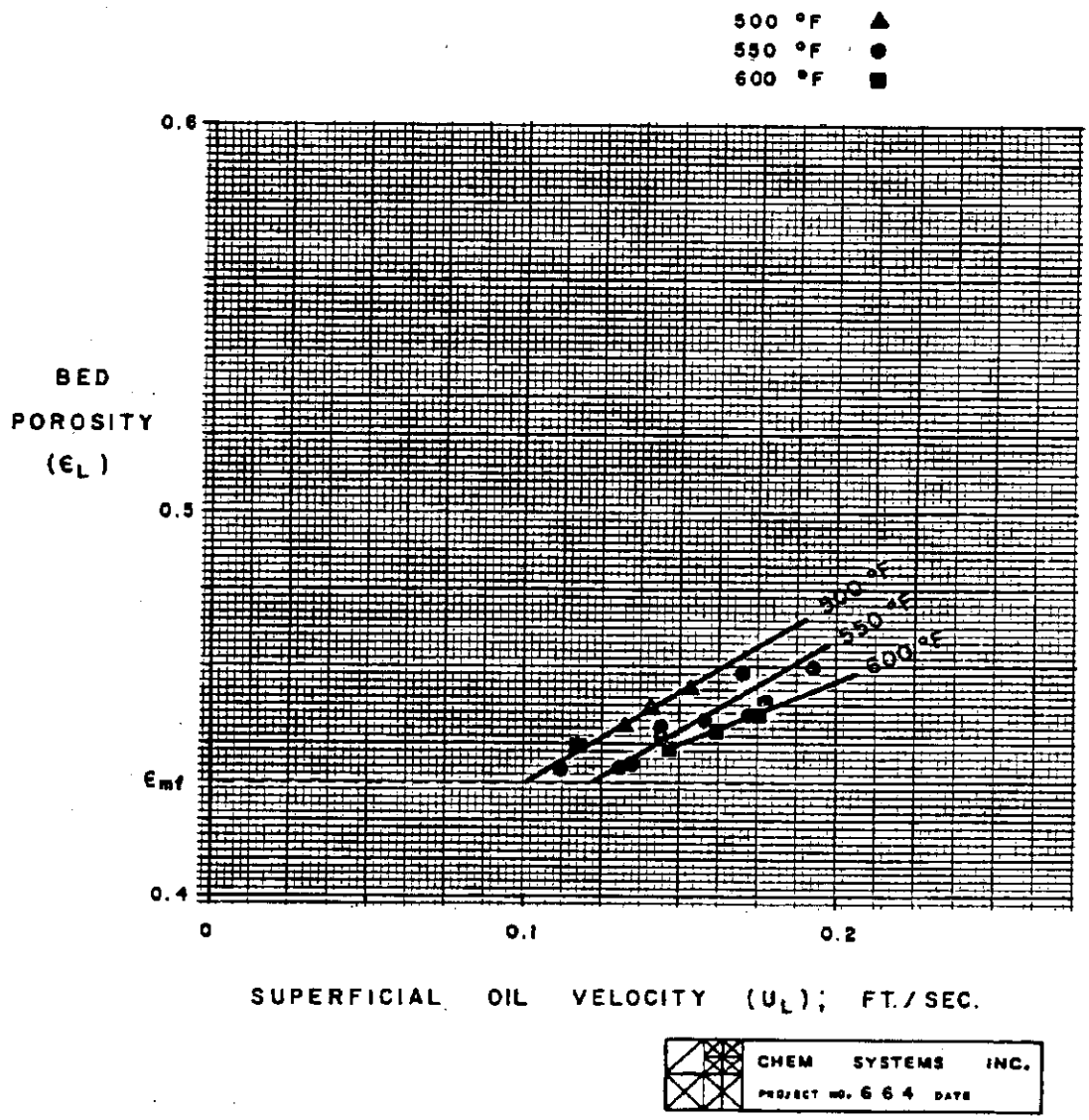

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TABLE IV-B-12
SUMMARY OF MAJOR EVENTS OF
LPM PILOT PLANT RUN #6

Hours on Stream	Accumulated Reaction Time	Date	Event
0	0	3/30/78	Started hydrogen flow into reactor.
8	0	3/30/78	Reached maximum reduction temperature of 800°F in reactor.
24	0	3/31/78	Started up circulating oil system.
44	0	4/01/78	Reduction completed and started lowering reactor to 550°F.
49	0	4/01/78	Integrated oil into reactor.
93	0	4/03/78	Settled bed height is 4.6 feet. Began liquid-only fluidization study.
97	0	4/03/78	First filter plugged.
136	0	4/05/78	Second filter plugged.
152	0	4/05/78	Completed liquid-only fluidization study.
168	0	4/06/78	Tested N_2-oil fluidization at simulated standard conditions. Bed expansion was 6.5%
169	0	4/06/78	Third filter plugged.
246	0	4/09/78	Fourth filter plugged.
336	0	4/13/78	Started up BFW system.
358	0	4/14/78	Settled bed height is 4.4 feet (823 lbs. cat).
376	0	4/15/78	Raised reactor to 500 psig, 500°F and 145 GPM oil flow.
406	0	4/16/78	Settled bed height is 4.4 feet.
435	0	4/17/78	Added hydrogen to by-passing nitrogen stream.
436	0	4/17/78	Stopped hydrogen addition.
449	0	4/18/78	Reactor cooled to 490°F due to bad weather. Started steam-methane reformer gas feed.

TABLE IV-B-12 (continued)
SUMMARY OF MAJOR EVENTS OF
LPM PILOT PLANT RUN #6

Hours on Stream	Accumulated Reaction Time	Date	Event
455	6	4/18/78	Reached 650°F, 500 psig and 35,000 SCFH feed gas.
457	8	4/18/78	Fifth filter plugged.
459	10	4/18/78	Stopped feed gas because IGT needed hydrogen. Sixth filter plugged. Started by-passing reactor.
462	10	4/18/78	Settled bed height is 4.3 ft. (804 lbs catalyst).
475	10	4/19/78	Seventh filter plugged.
488	10	4/19/78	Eighth filter plugged.
500	10	4/20/78	Ninth filter plugged.
502	10	4/20/78	Started steam-methane reformer gas feed.
504.5	12.5	4/20/78	Tenth filter plugged. Stopped feed gas to clean out fines.
529	12.5	4/21/78	Eleventh filter plugged.
543	12.5	4/22/78	Twelfth filter plugged.
565	12.5	4/23/78	Thirteenth filter plugged.
577	12.5	4/23/78	Fourteenth filter plugged.
584	12.5	4/23/78	Reactor pressure lowered to 300 psig in preparation for receiving HYGAS feed.
597.5	12.5	4/24/78	Started steam-methane reformer gas feed while by-passing filters.
600	15	4/24/78	Reached 300 psig, 600°F 30,000 SCFH gas and 180 GPM oil.
608	23	4/24/78	Reached 300 psig, 600°F, 40,000 SCFH gas and 180 GPM oil. Started BFW system.
610	25	4/24/78	Seal flush filter plugged and shut down system.
610.5	25	4/24/78	Restarted reformer gas feed at 30,000 SCFH, 550°F, 180 GPM oil and 300 psig.

TABLE IV-B-12 (continued)
SUMMARY OF MAJOR EVENTS OF
LPM PILOT PLANT - RUN #6

Hours on Stream	Accumulated Reaction Time	Date	Event
615.5	30	4/25/78	Stopped feed gas due to failure of main oil pump seals. Switched to spare.
621	30	4/25/78	Started feeding HYGAS product to reactor.
624	33	4/25/78	Reached 600°F, 300 psig, 18,000 SCFH gas and 180 GPM oil.
626	35	4/25/78	Reached 600°F, 300 psig, 28,000 SCFH gas and 180 GPM oil.
630.5	39.5	4/25/78	Increased oil flow to 210 GPM.
633.5	42.5	4/25/78	Decreased oil flow to 150 GPM.
639	48	4/26/78	Increased oil flow to 180 GPM.
644	53	4/26/78	Seal on P-102 A (condensed oil return pump) failed. Switched to spare.
645	54	4/26/78	Seal on P-102B (condensed oil return pump) failed. No spares available. Run terminated.
646	54	4/26/78	Main circulating pump (P-101) seal failed during cooldown. Entire oil system shut down.

No filters plugged during the second week of oil-only circulation. However, it was later discovered that some filter by-passing had occurred and that the oil had turned from clear amber color to opaque dark brown. The oil cleared up immediately when a new filter was placed in-line during hour 457.

The pilot plant was put on-stream during hour 449 on April 18 with steam-methane reformer gas. Conditions were brought to 500 psig, 650°F, 35,000 SCFH of feed gas and at 150 gpm of oil flow. The initial catalyst activity was 1.1 X 10^{-6} lb-mol/(sec-lb catalyst-atm), which is substantially higher than that observed in earlier pilot plant runs. Some cracking of the oil was observed when the reactor temperature rose above 650°F. Because of the high catalyst activity and the high gas flow rates, the reactor temperature overshot during initial heat-up. Bulk temperatures quickly rose to 690°F before the circulating oil cooler could be put into operation. Once in operation, the reactor overcooled to 620°F. Two more temperature cycles occurred while attempts were being made to line out the process conditions. Just as the reactor was beginning to approach steady conditions, the sixth filter plugged. Since no other filters were available and since IGT needed the hydrogen stream for HYGAS startup, feed gas was stopped after ten hours of reaction time.

During the high temperature excursions, it was observed that the product gas flow rate doubled rapidly and the product methane concentration also jumped to twice that which could be produced from the CO in the feed. Obviously, the oil was cracking and localized temperatures must have far exceeded the observed 690°F. It was decided to avoid this problem in the future by limiting the bulk reactor temperature to 600°F.

Upon completion of the ten hours of methanation, the settled bed height had decreased 7 percent (see Figure IV-B-15) for settled bed height as a function of time). Furthermore, the filters continued to plug about

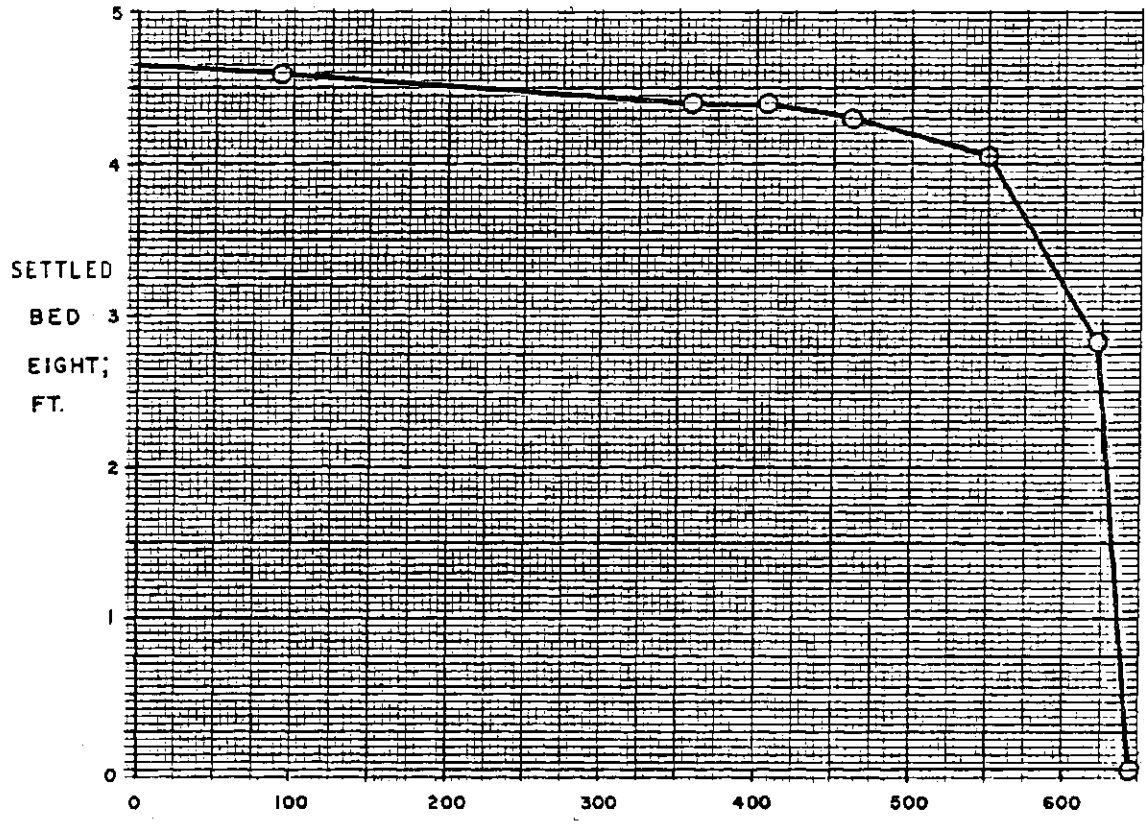
FIGURE IV-B-15

SETTLED BED HEIGHT

LPM PILOT PLANT - RUN #6

CATALYST #038 - A
FREEZENE - 100 OIL

CATALYST DENSITY : 1.09
CATALYST CHARGED : 860 LBS.
CATALYST LOADING : 187 LBS./FT.
SETTLED BED HEIGHT



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once every 15 hours on oil-only flow. The catalyst physical strength had evidently deteriorated because of its exposure to methanation or because of the high temperature excursions. Since the same problem occurred in a recent PDU run without high temperature excursions, this catalyst binder is evidently not strong enough to withstand LPM process conditions.

Feed gas was restarted on April 20, 1978. After 2.5 hours, the gas feed was stopped due to excessive filter plugging. The remainder of the week was spent filtering out the catalyst fines. Three more filters plugged in this manner, and the rate of plugging did not decrease with time.

At hour 584 on April 24, 1978, the reactor pressure was lowered to 300 psig in preparation for receiving HYGAS feed. The HYGAS gasifier was operating at 500 psig. At hour 598, steam-methane reformer gas feed was started while by-passing the main oil filters because the HYGAS feed was not ready. The initial catalyst rate constant was 0.8×10^{-6} mols/(atm-lb catalyst-sec) with 86 percent CO conversion while operating at 300 psig, 600°F and 30,000 SCFH of feed gas. Within ten hours, the catalyst rate constant increased to 4.0×10^{-6} with 100 percent CO conversion at the same conditions.

On April 25, HYGAS product was substituted in place of reformer feed. Again, the initial catalyst rate constant was low (1.2×10^{-6}), but after 20 hours it had increased to 5.0×10^{-6} at 300 psig, 600°F and 27,000 SCFH of feed gas. The enhanced catalyst activity, which was based upon the catalyst loading prior to initiation of filter by-passing (757 pounds), was caused by the breakup of catalyst particles into fines that circulated as a slurry throughout the oil system. Since the LPM Pilot Plant was not designed for slurry operation, accumulation of catalyst fines in the seal flush system eventually caused all the oil pump seals to fail and the unit had to be shut down.

At the end of the run, no integral catalyst particles were found in the reactor. Therefore, the efficient methanation results can be attributed to slurry operation. Based upon the estimated slurry concentration in the circulating oil, the catalyst rate within the reactor was approximately 40 X 10^{-6} at the end of the run.

Run #6 was terminated after 645 hours on-stream, which included 24 hours of methanation on HYGAS product and 30 hours on steam-methane reformer gas. The results, which were better than all previous pilot plant runs, are summarized in Table IV-B-13. Catalyst activity corrected to 650^{o}F is plotted as a function of reaction time in Figure IV-B-16. Each time feed gas was restarted, the rate constant was initially low and then gradually increased as more catalyst became reslurried. It should be pointed out that the initial catalyst activity, which occurred in a packed bed, was higher than the activities previously observed in the fluidized beds at the pilot plant. Less backmixing took place in the packed bed mode of operation. Of course, the final hours of methanation produced by far the best results to date at the pilot plant.

Samples of Freezene-100 oil were taken from the circulating oil system every two days. The oil remained clear amber throughout the run except when the filters were being by-passed, in which case up to 20 percent solids were found to be suspended in the oil. All samples were sent out for ASTM distillations, and results are shown in Table IV-B-14.

Upon completion of the run, all oil systems were immediately drained. Cleanout operations were begun and continued into May. The product gas separator was opened and all catalyst deposits were cleaned out. The skid area was also cleaned by an outside contractor. All instruments located on liquid lines were cleaned out and overhauled.

Work orders were written at the beginning of May for replacement of the two main circulating oil pump seals and the two condensed oil return pump

Table IV-B-13: LPM Pilot Plant Run #6 Results

Hour	456	457	458	601	602	603
Accumulated Reaction Time (Hrs)	7	8	9	16	17	18
Feed Gas:						
H_2/CO Ratio	9.05	9.01	8.75	9.51	9.26	8.85
% H_2	89.05	89.15	88.95	88.92	88.51	88.17
% N_2	0.72	0.60	0.60	0.29	0.44	0.52
% CH_4	0.34	0.31	0.22	1.29	1.33	1.18
% CO	9.85	9.81	10.17	9.35	9.56	9.96
% CO_2	0.05	0.05	0.06	0.15	0.17	0.18
% C_2^+	0.00	0.00	0.00	0.00	0.00	0.00
VHSV (Hr^{-1})	3,040	3,040	2,910	2,650	2,580	2,400
Oil Flow Rate: GPM/Ft^2	53.8	53.8	53.8	66.0	64.7	64.7
Temperature (°F)	645	642	645	601	601	602
Pressure (psig)	500	500	500	300	300	300
Product Gas:						
% H_2	79.50	82.02	83.55	85.54	84.82	84.58
% N_2	0.91	0.95	0.96	0.55	0.62	0.62
% CH_4	19.16	16.69	14.48	11.98	13.27	13.78
% CO	0.26	0.20	0.86	1.75	1.09	0.75
% CO_2	0.04	0.04	0.04	0.09	0.10	0.16
% C_2^+	0.12	0.12	0.11	0.10	0.10	0.12
MW	5.06	4.70	4.57	4.36	4.39	4.40
SCFH	18,670	21,470	23,070	22,700	21,660	20,280
CO Conversion (%)	98.62	98.79	94.35	85.62	91.43	94.31
CO_2 Conversion (%)	58.71	52.56	56.03	53.48	56.74	35.95
CH_4 Selectivity (%)	98.71	98.54	98.46	98.11	98.14	98.09
Catalyst Rate Constant:						
K_{T_R} ($\times 10^6$)	1.07	1.10	0.70	0.67	0.85	0.95
$K_{650°F}$ ($\times 10^6$)	1.09	1.14	0.72	0.84	1.06	1.19
Run Number	—	—	—	—	—	—
Bed Height (FT)	N.D.	4.6	N.D.	N.D.	N.D.	N.D.

Table IV-B-13 LPM Pilot Plant Run #6 Results (continued)

Hour	604	606	608	609	613	614
Accumulated Reaction Time (Hrs)	19	21	23	24	28	29
Feed Gas:						
H_2/CO Ratio	9.02	9.12	9.89	9.79	9.36	9.36
% H_2	88.33	88.74	89.51	89.41	89.41	89.41
% N_2	0.52	0.48	0.29	0.24	0.09	0.09
% CH_4	1.18	0.94	1.06	1.13	0.85	0.85
% CO	9.78	9.73	9.05	9.13	9.56	9.56
% CO_2	0.18	0.11	0.10	0.10	0.10	0.10
% C_2^+	0.00	0.00	0.00	0.00	0.00	0.00
VHSV (Hr^{-1})	2,460	2,530	3,130	3,220	2,580	2,670
Oil Flow Rate: GPM/Ft^2	66.0	66.0	66.0	66.0	64.9	64.9
Temperature (°F)	603	609	594	601	551	541
Pressure (psig)	300	300	300	300	300	300
Product Gas:						
% H_2	84.05	83.81	85.35	85.45	86.46	86.11
% N_2	0.64	0.62	0.49	0.42	0.13	0.21
% CH_4	14.66	15.30	13.88	13.84	12.08	13.31
% CO	0.36	0.00	0.00	0.00	0.93	0.08
% CO_2	0.16	0.14	0.14	0.14	0.25	0.16
% C_2^+	0.13	0.14	0.15	0.15	0.15	0.14
MW	4.43	4.42	4.19	4.17	4.13	4.06
SCFH	19,790	19,320	24,870	26,000	22,260	22,600
CO Conversion (%)	97.35	99.99	99.99	99.99	92.45	99.35
CO_2 Conversion (%)	38.87	14.18	0.32	—	—	—
CH_4^2 Selectivity (%)	98.03	98.07	97.67	97.59	96.35	97.54
Catalyst Rate Constant:						
K_{T_R} (x 10^6)	1.22	3.29	3.75	3.89	0.87	1.76
$K_{650°F}$ (x 10^6)	1.52	3.96	4.86	4.87	1.41	2.99
Run Number	—	—	—	—	—	—
Bed Height (FT)	N.D.	N.D.	N.D.	3.7	N.D.	N.D.

Table IV-B-13: LPM Pilot Plant Run #6 Results (continued)

Hour	615	623	624	626	627	628
Accumulated Reaction Time (Hrs)	30	32	33	35	36	37
Feed Gas:						
H_2/CO Ratio	9.36	3.13	3.26	2.97	2.95	2.91
% H_2	89.41	43.44	42.54	41.77	43.28	44.03
% N_2	0.09	16.52	16.15	15.83	16.35	15.12
% CH_4	0.85	25.37	27.53	27.21	24.95	24.93
% CO	9.56	13.88	13.04	14.07	14.66	15.15
% CO_2	0.10	0.14	0.08	0.43	0.15	0.15
% C_2^+	0.00	0.66	0.66	0.71	0.61	0.63
VHSV (Hr^{-1})	2,620	1,590	1,590	2,480	2,470	2,560
Oil Flow Rate: GPM/Ft^2	64.9	66.0	66.0	66.0	66.0	66.0
Temperature (°F)	549	600	600	601	602	608
Pressure (psig)	300	300	300	300	300	300
Product Gas:						
% H_2	86.13	15.74	15.33	18.36	17.89	19.70
% N_2	0.10	22.80	21.38	21.66	21.63	21.30
% CH_4	13.46	54.12	56.94	52.26	51.54	50.37
% CO	0.00	3.94	3.17	4.94	5.55	5.45
% CO_2	1.18	2.15	1.93	1.48	2.10	1.80
% C_2^+	0.13	1.25	1.25	1.25	1.30	1.29
MW	4.04	17.81	17.55	17.25	17.56	17.18
SCFH	22,020	11,500	11,490	19,460	18,210	19,610
CO Conversion (%)	99.99	81.56	84.20	75.27	74.90	74.85
CO_2 Conversion (%)	—	—	—	—	—	—
CH_4 Selectivity (%)	97.51	86.23	86.58	90.33	84.00	85.87
Catalyst Rate Constant:						
K_{T_R} ($\times 10^6$)	3.24	0.95	1.03	1.30	1.26	1.32
$K_{650°F}$ ($\times 10^6$)	5.28	1.20	1.29	1.63	1.57	1.59
Run Number	—	—	—	—	—	—
Bed Height (FT)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

LPM Pilot Plant
Table IV-B-13: Run #6 Results (continued)

Hour	629	630	631	632	633	634
Accumulated Reaction Time (Hrs)	38	39	40	41	42	43
Feed Gas:						
H_2/CO Ratio	2.83	2.73	2.82	2.80	2.64	2.55
% H_2	42.73	42.98	42.58	42.77	40.91	44.39
% N_2	16.15	14.89	14.58	15.02	15.37	13.41
% CH_4	25.18	25.58	26.43	25.38	27.05	24.09
% CO	15.10	15.74	15.12	15.26	15.49	17.40
% CO_2	0.21	0.15	0.65	0.98	0.59	0.18
% C_2^+	0.64	0.67	0.63	0.61	0.60	0.53
VHSV (Hr^{-1})	2,450	2,490	2,280	2,320	2,380	2,450
Oil Flow Rate: GPM/Ft^2	66.0	66.0	76.6	76.6	77.9	56.8
Temperature (°F)	608	606	606	608	605	608
Pressure (psig)	300	300	300	300	300	300
Product Gas:						
% H_2	18.10	16.80	16.16	14.43	13.37	12.31
% N_2	21.31	21.91	21.08	21.46	22.57	22.31
% CH_4	51.30	53.60	54.90	56.28	56.77	57.64
% CO	5.90	4.00	4.31	3.40	2.43	2.22
% CO_2	2.01	2.28	2.12	3.00	3.43	4.07
% C_2^+	1.38	1.41	1.39	1.43	1.48	1.45
MW	17.52	17.62	17.60	18.03	18.34	18.59
SCFH	18,360	18,190	17,240	16,910	17,890	17,430
CO Conversion (%)	73.64	82.65	80.69	85.42	89.41	91.83
CO_2 Conversion (%)	—	—	—	—	—	—
CH_4^2 Selectivity (%)	84.50	84.72	88.58	87.52	81.73	79.90
Catalyst Rate Constant:						
K_{T_R} (x 10^6)	1.22	1.68	1.41	1.69	2.16	2.47
$K_{650°F}$ (x 10^6)	1.48	2.05	1.73	2.04	2.65	3.00
Run Number	—	—	—	—	—	—
Bed Height (FT)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

Table IV-B-13: LPM Pilot Plant Run #6 Results (continued)

Hour	635	637	638	639	641	642
Accumulated Reaction Time (Hrs)	44	46	47	48	50	51
Feed Gas:						
H_2/CO Ratio	2.55	2.60	2.54	2.54	2.29	2.20
% H_2	44.40	43.18	43.60	43.60	41.81	41.10
% N_2	13.41	13.31	13.57	13.57	13.30	13.93
% CH_4	24.10	25.59	25.07	25.07	25.97	25.67
% CO	17.40	16.63	17.15	17.15	18.30	18.70
% CO_2	0.18	0.85	0.15	0.15	0.16	0.14
% C_2^+	0.51	0.45	0.46	0.46	0.46	0.46
VHSV (Hr^{-1})	2,430	2,510	2,500	2,500	2,420	2,420
Oil Flow Rate: GPM/Ft^2	55.5	57.1	57.1	57.1	66.0	66.0
Temperature (°F)	608	607	607	607	603	606
Pressure (psig)	300	300	300	300	300	300
Product Gas:						
% H_2	12.63	11.10	10.60	10.46	8.93	9.18
% N_2	22.91	21.34	21.61	21.65	21.61	21.34
% CH_4	56.87	59.88	59.81	59.66	61.43	61.61
% CO	2.34	1.97	1.68	1.94	1.08	0.98
% CO_2	3.87	4.35	4.95	4.93	5.59	5.54
% C_2^+	1.38	1.36	1.35	1.35	1.36	1.36
MW	18.57	18.68	18.92	18.97	19.26	19.17
SCFH	17,540	17,820	17,430	17,430	17,270	17,300
CO Conversion (%)	91.29	92.44	93.88	82.92	96.23	96.63
CO_2 Conversion (%)	—	—	—	—	—	—
CH_4 Selectivity (%)	80.50	82.05	76.85	76.77	75.95	76.61
Catalyst Rate Constant:						
K_{T_R} (x 10^6)	2.47	2.69	2.94	2.79	3.73	4.03
$K_{650°F}$ (x 10^6)	2.99	3.27	3.58	3.40	4.63	4.92
Run Number	—	—	—	—	—	—
Bed Height (FT)	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

Table IV-B-13: LPM Pilot Plant Run #6 Results (continued)

Hour	643	644	645			
Accumulated Reaction Time (Hrs)	52	53	54			
Feed Gas:						
H_2/CO Ratio	2.30	2.30	R			
% H_2	41.95	41.95	U			
% N_2	13.53	13.52	N			
% CH_4	25.68	25.60	T			
% CO	18.23	18.23	E			
% CO_2	0.16	0.16	R			
% C_2^+	0.46	0.46	M			
VHSV (Hr^{-1})	2,360	2,300	I			
Oil Flow Rate: GPM/Ft^2	66.0	66.0	N A T E D			
Temperature (°F)	606	603				
Pressure (psig)	300	300				
Product Gas:						
% H_2	9.06	8.81				
% N_2	22.05	22.12				
% CH_4	61.30	60.72				
% CO	0.42	1.26				
% CO_2	5.81	5.74				
% C_2^+	1.36	1.35				
MW	19.28	19.40				
SCFH	16,840	16,390				
CO Conversion (%)	98.51	95.57				
CO_2 Conversion (%)	—	—				
CH_4 Selectivity (%)	75.56	75.22				
Catalyst Rate Constant:						
K_{T_R} ($\times 10^6$)	4.64	3.35				
$K_{650°F}$ ($\times 10^6$)	5.67	4.15				
Run Number	—	—				
Bed Height (FT)	N.D.	N.D.				

FIGURE IV-B-16

CATALYST ACTIVITY VS. TIME

LPM PILOT PLANT - RUN # 6
CATALYST # 038 - A
FREEZENE - 100 OIL

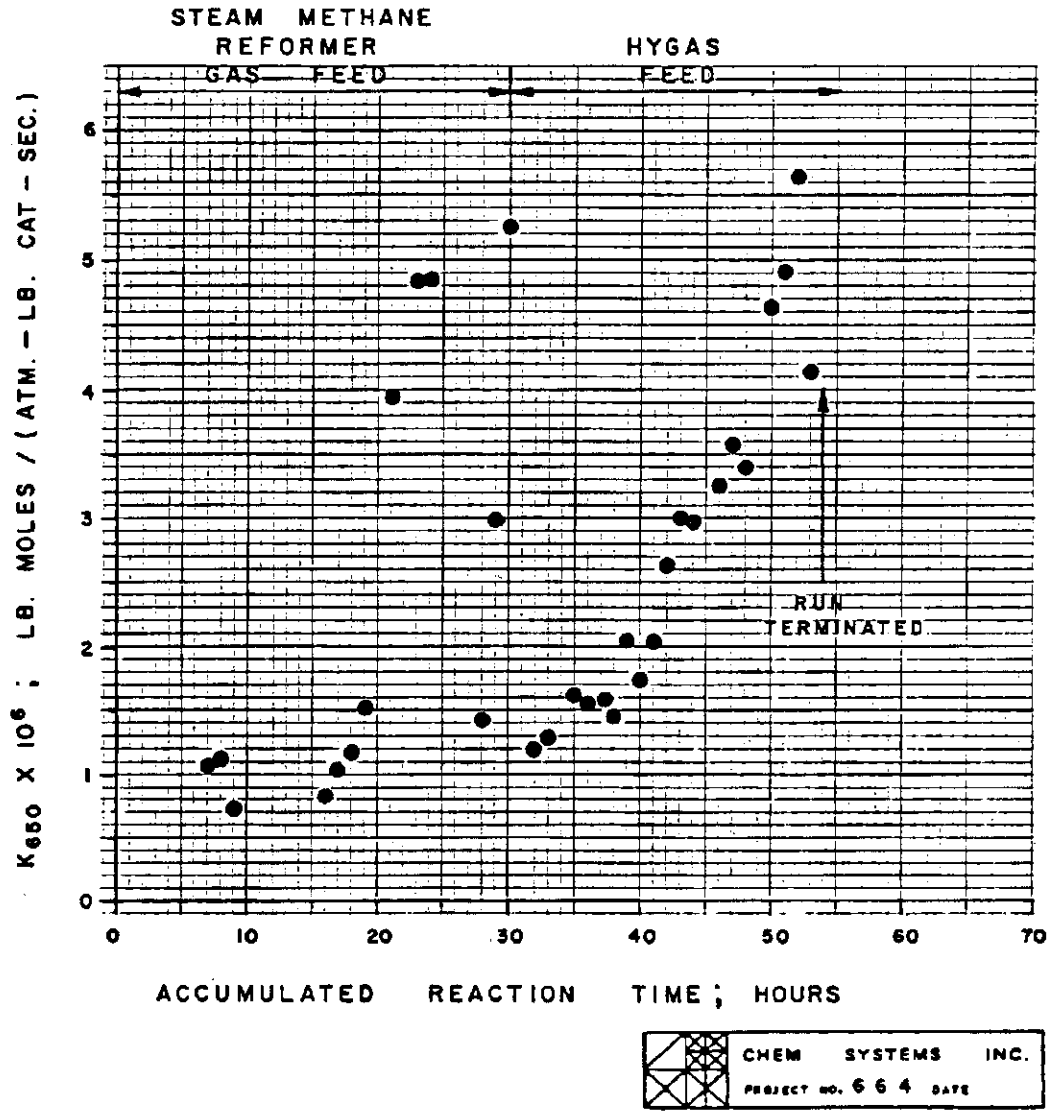

TABLE IV-B-14

ASTM DISTILLATION OF FREEZENE-100 FROM CIRCULATING OIL SYSTEM DURING PILOT PLANT RUN #6

Hour ASTM D-86 Distillation	108	156	204	314	362
IBP	519°F	524	529	477	512
5%	601	594	594	595	604
10%	617	613	612	612	618
20%	632	632	630	630	634
30%	646	644	644	647	648
40%	658	656	656	659	660
50%	670	667	667	670	673
60%	683	680	679	680	685
70%	697	692	692	685	698
80%	716	703	712	708	715
90%	725	748	748	739	739
95%	---	---	---	---	---
EP	762	762	761	760	760
% Recovery	94.5	95.0	94.5	96.0	96.0
% Residue	4.0	4.5	4.5	3.0	3.5

Hour ASTM D-86 Distillation	410	458	554	602	650
IBP	531	540	449	558	516
5%	606	610	568	602	582
10%	623	622	592	621	603
20%	637	633	612	642	624
30%	652	652	631	653	640
40%	664	664	649	670	654
50%	676	678	658	683	666
60%	689	692	669	695	679
70%	703	709	679	710	695
80%	719	725	709	730	714
90%	737	757	750	760	758
95%	756	---	---	---	---
EP	758	760	758	760	760
% Recovery	97.5	95.0	97.0	94.0	95.0
% Residue	2.0	4.0	2.5	6.0	4.5

seals. By the end of the month, nothing had been done because the IGT maintenance department was busy with other projects in the HYGAS pilot plant.