Figure IV.5

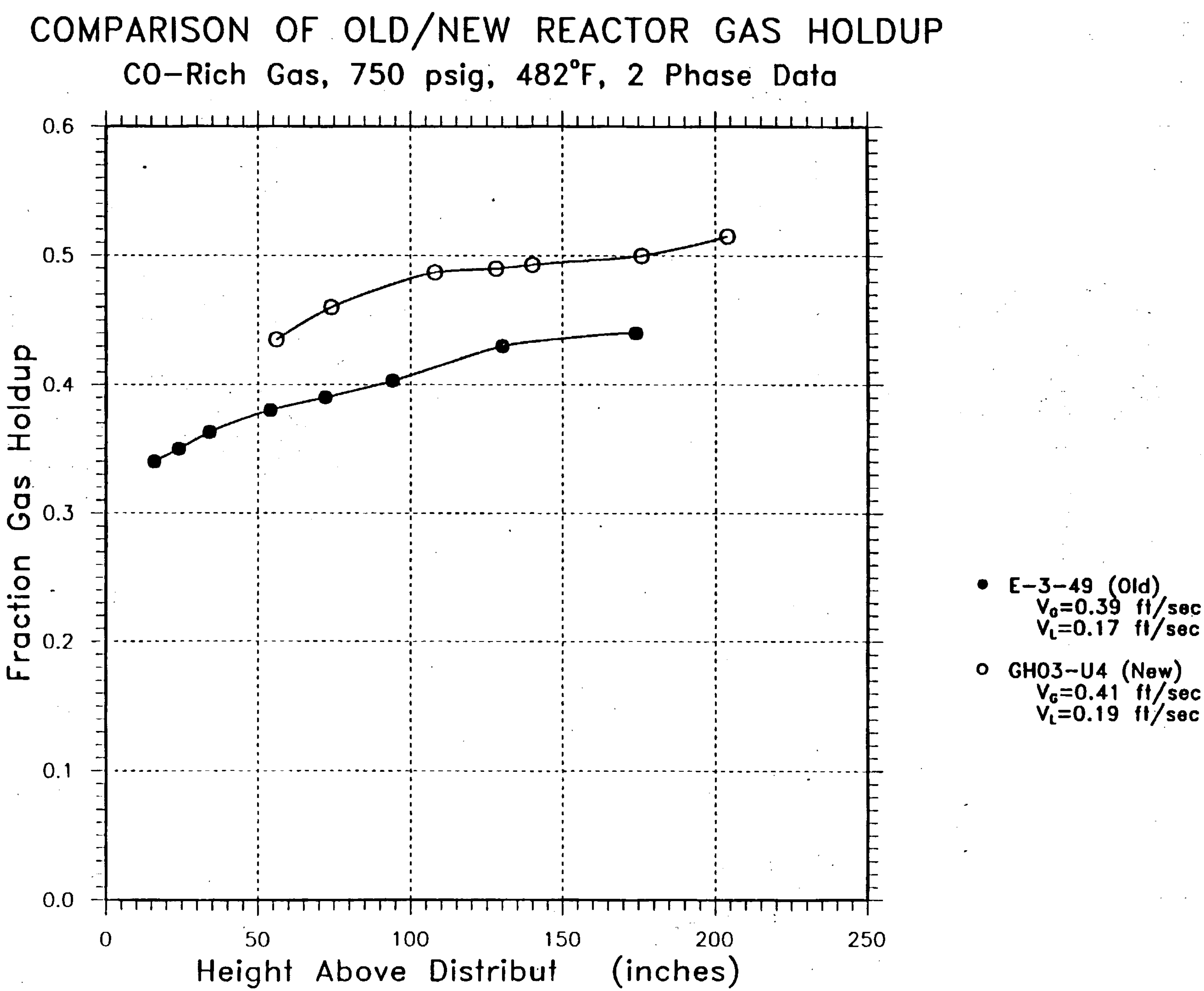

COMPARISON OF OLD/NEW REACTOR GAS HOLDUP
CO–Rich Gas, 750 psig, 482°F, 2 Phase Data
Fraction Gas Holdup
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0
50
100
150
200
250
Height Above Distribut (inches)
E–3–49 (Old)
V_G=0.39 ft/sec
V_L=0.17 ft/sec
GH03–U4 (New)
V_G=0.41 ft/sec
V_L=0.19 ft/sec

Figure IV.6

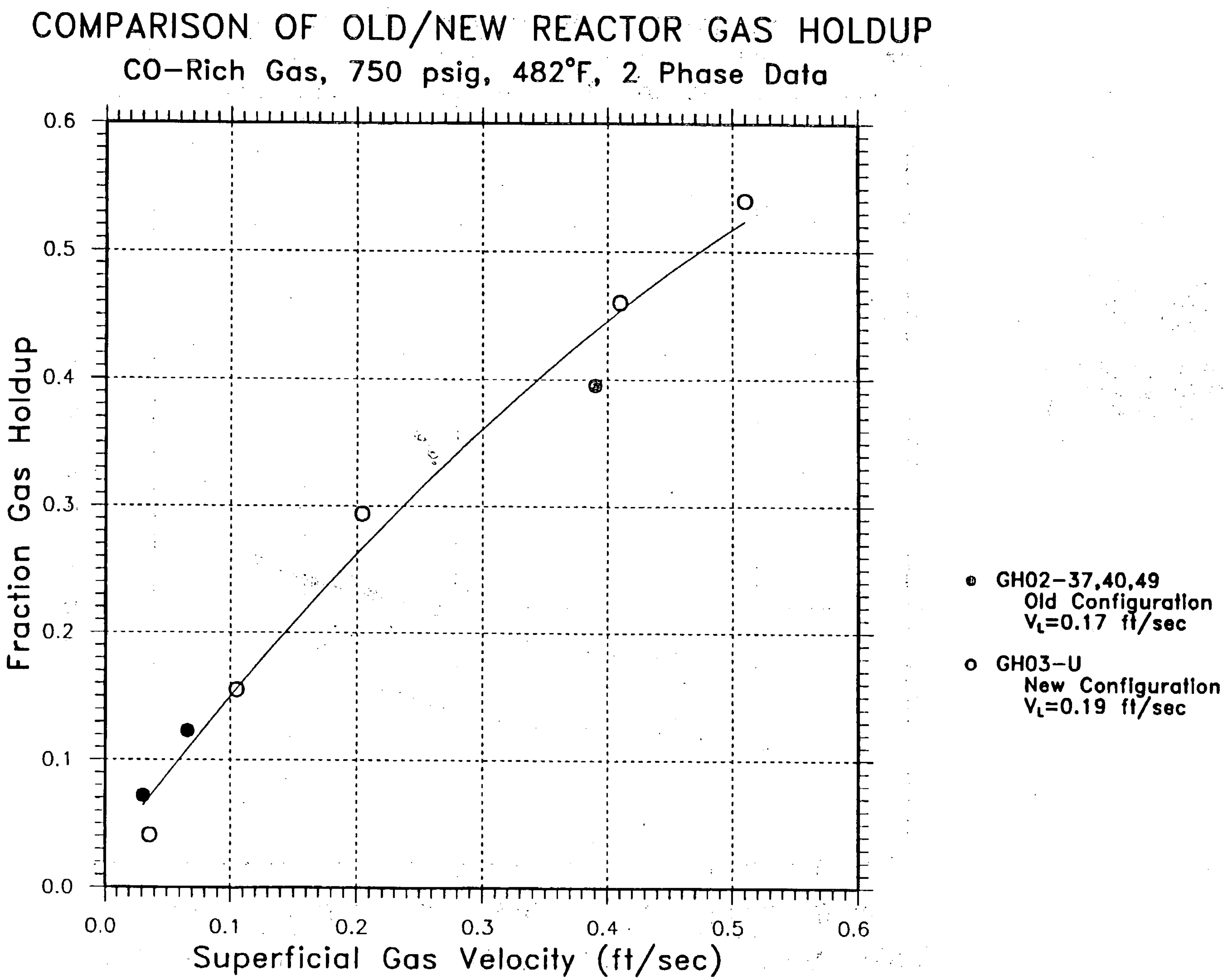
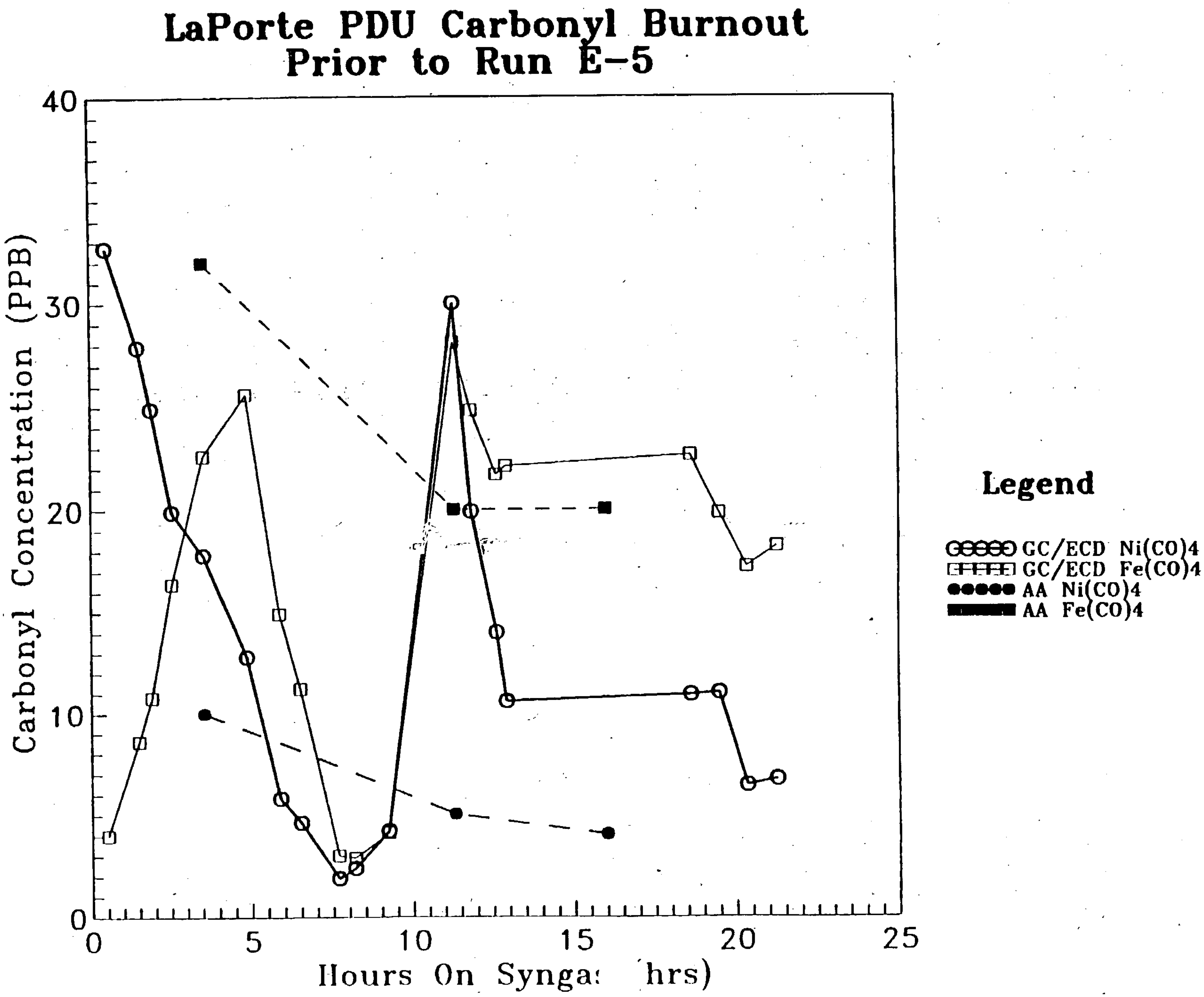

COMPARISON OF OLD/NEW REACTOR GAS HOLDUP
CO–Rich Gas, 750 psig, 482°F, 2 Phase Data
Fraction Gas Holdup
0.0
0.1
0.2
0.3
0.4
0.5
0.6
Superficial Gas Velocity (ft/sec)
GH02–37,40,49
Old Configuration
V_L=0.17 ft/sec
GH03–U
New Configuration
V_L=0.19 ft/sec

Figure IV.7


LaPorte PDU Carbonyl Burnout
Prior to Run E-5
Carbonyl Concentration (PPB)
40
30
20
10
0
0
5
10
15
20
25
Hours On Synga: hrs)
Legend
GC/ECD Ni(CO)4
GC/ECD Fe(CO)4
AA Ni(CO)4
AA Fe(CO)4

V. EQUIPMENT EVALUATION RUN (RUN E-5)

A. Introduction

Run E-5 was conducted at the LaPorte LPMEOH PDU in July, 1988. A commercially available catalyst powder (F21/OE75-44) was used. This was a new batch of the same catalyst which was used in Runs E-2, E-3, and E-4. The objective of Run E-5 was to systematically evaluate each new piece of equipment which had been added to the system: spargers, internal heat exchanger, vapor/liquid disengagement zone, demister, and cyclone. In addition, a catalyst activation with a concentrated (45 wt% oxide) slurry was conducted.

The plan to systematically evaluate each new piece of equipment was set up in four stages as shown in Figure V.1. The possible operating modes, which were identified from the previous process engineering study and resulting PDU modifications, were to be evaluated in the following order: 1) sparger gas distribution, 2) internal heat exchanger, 3) no bulk liquid circulation, and 4) shutdown test. While these operating modes were being tested, the V/L disengagement zone, demister, and cyclone would be individually evaluated.

B. In-Situ Activation

A batch of 40 wt% (oxide basis) slurry using F21/OE75-44 catalyst powder and Drakeol-10 oil was mixed in the 28.30 slurry prep tank under a nitrogen blanket and pressure-transferred to the slurry loop. The reducing gas was blended, the composition was verified (4 mol% H_2, 96 mol% N_2), and it was introduced to the reactor. The reduction gas leaving the reactor was sent to the flare. The temperature of the slurry was slowly increased during the activation process along a predefined temperature ramp using utility oil in the 21.20 external slurry heat exchanger. The inventory of oil in the slurry decreased over the course of the reduction as oil vapors were stripped from the reactor by the reduction gas. As a result, the slurry concentration increased to 45 wt% (as oxide) during the activation. Further details of the catalyst activation for Run E-5 are presented in the Catalyst Activation Chronology (see Table V-1).

The reactor slurry temperature was calculated by averaging readings from a series of thermocouples installed at various heights in the reactor. In the initial phase of the activation procedure it was discovered that the reported average slurry temperature was artificially low since the temperature calculation included a thermocouple which was not fully immersed in the liquid phase of the reactor. When this was corrected the actual activation temperature ramp rate exceeded the maximum recommended ramp rate. The reduction in temperature, reported in the chronology at 0530 on 7/15/88, was a result of cooling the slurry temperature to coincide with the predefined temperature program. There were no other major operational problems, and the NDG readings indicated a flat axial solids profile throughout the activation procedure.

However, the analytical data presented in the Results and Discussion section, indicated that the catalyst was not fully reduced during this activation procedure. The amount of hydrogen that reacted with the copper oxide to convert it to copper metal (zero valence state) was 1.37 scf of hydrogen per pound of catalyst (scf/lb). This is 15% short of the theoretical hydrogen

Figure V.1 NEW EQUIPMENT EVALUATION – SIMPLIFIED FLOWSHEET

Individually verify the performance of each new equipment item over 3–4 weeks.

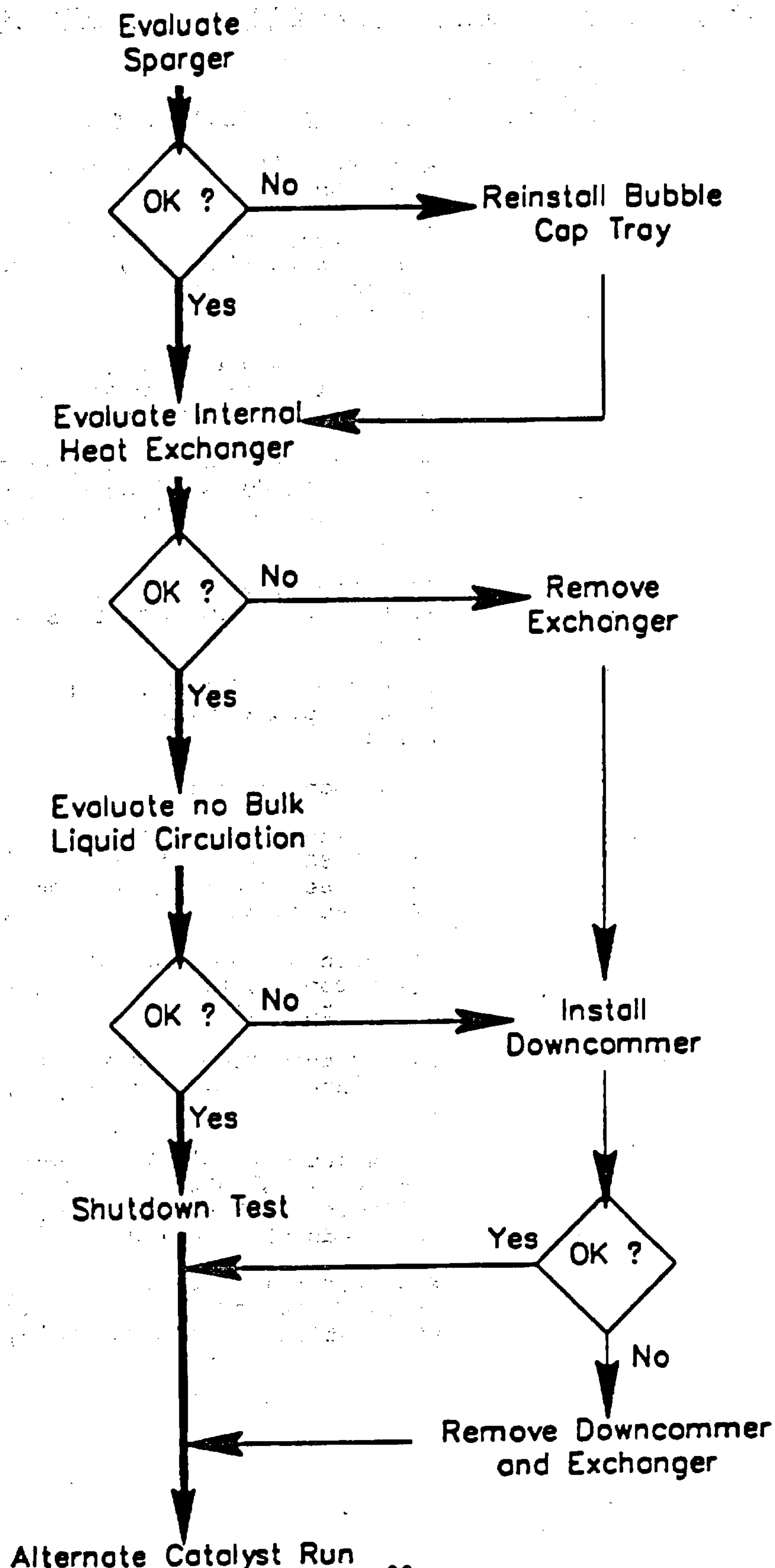


TABLE V.1

CATALYST ACTIVATION RUN ER-04 CHRONOLOGY WITH CATALYST F21/0E75-44

Date	Time	Cumulative Time On Reduction Gas (Hours)	Notes and Observations
7/14/88	0400		Charged 3990 lb of Drakeol-10 to slurry prep tank.
	0620		Pressure-transferred slurry prep tank contents to slurry loop in order to preheat oil to 250°F at a 75°F/hr maximum rate.
	1200		Oil temperature at 250°F. Transferred oil back to slurry prep tank in order to begin catalyst loading.
	1320		Oil in slurry prep tank reduced to 200°F. Loaded 2678 lb of catalyst powder (F21/0E75-44) to slurry prep tank.
	1430		Transfer final 787 lb of Drakeol-10 oil left in 27.10 reactor to slurry prep tank.
	2050		Transferred slurry from slurry prep tank to primary separator. Slurry pump started.
	2340		Reduction gas flow started to reactor.
7/15/88	0200	2 1/3	Conditions for reduction flow steady at 28,000 SCFH with the reactor at 165 psig. Began heating up the slurry from 204°F at an initial rate of 15°F/hr.
	0244	3.07	Hydrogen consumption first detected on gas chromatograph at a slurry temperature of 220°F.
7/15/88	0530	5 5/6	Temperature reduced from 262°F to 248°F to maintain maximum temperature ramp rate.
	1610	16 1/2	Temperature at 391°F. Power loss results in GC shutdown, reactor flow maintained and stable. Beginning of bulk reduction period.
7/16/88	0900	33 1/3	Bulk reduction completed; 27.10 temperature ramped down to 473 F in preparation to introducing syngas.

uptake of 1.62 scf/lb, indicating that the copper catalyst was not fully reduced during this procedure.

C. Methanol Synthesis Operation

After completion of the catalyst activation, the CO-Rich synthesis gas supplies were brought on-line and the PDU began operating under the first condition for Run E-5. The process parameters for the 6 cases of Run E-5 are summarized in Table V-2 and the detailed run chronology is listed in Table V.3. The purpose of the first case was to directly compare the performance of the new reactor in the slurry circulation mode to that of the old reactor system. In the second case, the effect of using the internal heat exchanger for heat removal was examined. The third case was a maximum flow rate test which was originally planned for the internal heat exchanger only. Because of an intentional under-design of the area in order to provide accurate heat transfer data with a 35 wt% slurry, however, it was necessary to use the external heat exchanger as a trim to remove the excessive heat load created by running at maximum rates with a 45 wt% slurry. The final three cases of this run examined the performance of the slurry reactor without the use of the external slurry loop. Overall the PDU accumulated 259.4 hours of methanol synthesis operation during this equipment evaluation Run E-5.

D. Discussion of Results

Figure V.2 shows a comparison of the results of the in-situ catalyst activation Runs ER-3 (prior to Run E-4) and ER-4 (prior to Run E-5). Runs ER-3 and ER-4 were done at nearly identical slurry catalyst concentrations and reduction gas flows. As noted previously, the sharp reduction in temperature which occurred in Run ER-4 at approximately 270°F was due to a miscalculation of the slurry temperature. The most significant difference between the two activation curves is in the total H_2 uptake, which matched autoclave predictions for Run ER-3 but fell short by 15% in Run ER-4. One possible cause for this difference is that the rapid temperature swing at the beginning of activation caused an irreversible change in the catalyst properties which prevented complete activation. A second possibility is that the reduction gas flow was marginally too low and was insufficient to remove the CO_2 and H_2O byproducts from the slurry that were generated during catalyst activation. Avoiding thermal swings and increasing the reduction gas flow while maintaining the H_2 partial pressure is recommended for future activations.

Table V.2 lists the conditions and results for the equipment evaluation Run E-5. Production of methanol was stable in Run E-5A after only 18 hours on-stream with syngas. Methanol productivity, as illustrated in Figure V.3, and gas holdup were higher than previous runs (Run E-4) at high catalyst loadings. The improvement in catalyst methanol productivity over the previous Run E-4, in spite of the poor activation, is a significant result. This demonstrated improvement is attributable to the new gas sparger. However, as expected from the poor activation, methanol productivity was still slightly below the laboratory autoclave curve. Mass transfer limitations may also have been present during this run. Catalyst productivities at these conditions indicated that the performance of the new reactor, with the gas sparger and the internal heat exchanger installed, exceeded that of the old reactor system.

TABLE V.2

CONDITIONS AND RESULTS FOR EQUIPMENT EVALUATION
(RUN E-5)

CO-Rich Feed Gas

Catalyst: F21/0E75-44; Oil: Drakeol-10

CASE:	A	B	C	D	E	F
Balance Period:						
Start Date	18 July	20 July	21 July	23 July	25 July	27 July
Start Time	1400	0100	0600	1100	0500	1600
End Date	19 July	21 July	23 July	25 July	26 July	28 July
End Time	1400	0100	0600	0000	0500	0800
Bal. Hours on Syngas	24	24	33	37	20	16
Cumm. Hours on Syngas	74.0	110.0	147.0	189.0	218.0	236.0
Configuration:						
Heat Exchanger	External	Internal	Ext/Int	Internal	Internal	Internal
Slurry Loop	External	External	External	None	None	None
Reactor Conditions:						
Temperature (/C)	250.4	250.1	250.2	250.2	250.1	250.5
Pressure (psig)	752.8	753.1	752.1	751.6	752.8	754.8
Space Velocity (Sl/hr-kg)	5297	5313	7084	5445	11356	11444
Inlet Gas Velocity (ft/sec)	0.41	0.41	0.55	0.41	0.51	0.51
Gas Holdup (vol%)	23.1	23.3	26.7	27.6	34.0	33.8
Slurry Conc. (wt% ox.)	45.0	44.9	45.9	45.0	34.1	33.9
Catalyst Inventory (kg ox.)	595	592	589	571	339	338
Conversion/Production Results:						
CO Conversion (%)	12.8	12.8	11.6	13.6	11.8	11.2
Methanol in Effluent (mol %)	8.12	8.04	6.79	8.79	7.70	7.37
Methanol Productivity (gmol/hr-kg cat ox)	14.7	15.0	18.0	16.8	30.3	29.6
Productivity as % of Autoclave	81	83	81	91	105	103
Production (TPD)	7.65	7.81	9.19	8.29	8.87	8.61

TABLE V.3

CHRONOLOGY RUN E-5 WITH CATALYST F21/0E75-44

Date	Time	Cumulative Time On Production Gas (Hours)	Notes and Observations
7/16/88	1138	0	Start syngas flow to the 27.10. Begin Run E-05.
	1145	1/6	MeOH concentration in the reactor effluent at 0.058%. First MeOH production of LPIII.
	1205	1/2	Utility oil used to cool the 27.10. Significant reaction had begun.
	1530	3 5/6	Sample from the 22.15 product/oil separator shows 1 small drop of oil in methanol product.
	1600	4 1/3	Slurry sample taken from external slurry loop. TIC-188 set at 270 F to attempt to lower the amount of oil in the product MeOH.
	2130	9 5/6	Slurry sample taken from the external circulating slurry loop.
	2330	11 5/6	TIC-188 set at 260 F to determine how much less oil will make it into the product at the lower temperatures.
7/17/88	0930	21 5/6	The slurry circulation flow was discovered to be running at higher than 300 gpm rates since beginning of run E-05. The circulation rate was lowered to 225 gpm. High liquid circulation rates gave artificially low gas holdup; after lowering flow, saw increase in gas holdup.
	1245	25 1/6	Drained 150 lb oil from 22.16 prior to transferring product methanol from the day tank.
	1400	26 1/3	Pulled a slurry sample from the external slurry loop.
	1505	27 2/5	Power outage to the control room and the computer.
	1700	29 1/3	Power restored to control room and computer restarted. Good data again.
	2300	35 1/3	Draining of the 22.16 revealed no evidence of oil in the product methanol.

TABLE V.3

CHRONOLOGY RUN E-5 WITH CATALYST F21/0E75-44

(continued)

Date	Time	Cumulative Time On Production Gas (Hours)	Notes and Observations
7/18/88	0019	36 2/3	Total power failure for about 2 minutes. HYCO flaring. 10.50 circulation pump not running and feed gas to 27.10 off.
	0027	36 5/6	10.50 circulation pump restarted and flowing at 190 gpm.
	0039	37	Gas flow to the 27.10 restarted.
	0125	37 5/6	HYCO line pressure low having trouble maintaining reactor feed flow.
	0220	38 5/6	HYCO pressure restored flow returning to normal.
	1200	48 3/6	Brief power outage taken to move control room to its own individual circuit.
	1300	49 3/6	10.50 circulation pump has developed a seal oil leak which leaks at a rate of 5 lb/hr.
	1400	50 3/6	Beginning of Case E-5A. Nominal conditions for this case are CO-Rich feed gas, 5,300 Sl/hr-kg cat space velocity, 250 °C, 750 psig, and 45 wt% catalyst. The purpose of this run is to evaluate the new gas sparger.
7/19/88	1400	74 3/6	End of case E-5A. Made 7.65 TPD MeOH and a productivity of 14.7 g-mole/hr-kg or 81% of autoclave.
	1600	76 3/6	Opened the 28.30 and accounted for 1036 pounds of slurry not in the slurry system.
	1500	77 3/6	Switching from external heat exchanger to internal heat exchanger revealed that the utility oil temperature to reactor temperature was approaching design limits at the high weight percent and low space velocity.
7/20/88	0100	87 3/6	Beginning of Case E-5B. Nominal conditions for this case are CO-Rich feed gas, 5,300 Sl/hr-kg cat, 250 °C, 750 psig, and 45 wt% catalyst. The purpose of this run is to study the performance of heat transfer using the internal heat exchanger.

TABLE V.3

CHRONOLOGY RUN E-5 WITH CATALYST F21/0E75-44

(continued)

Date	Time	Cumulative Time On Production Gas (Hours)	Notes and Observations
	1200	98 3/6	27.13 vessel was x-rayed to determine the exact liquid level in the vessel. Found level at 18.7% and LR-203 read 20-21%.
	1400	100 3/6	Heavy rains caused instability in reactor temperature and effluent methanol concentrations. Temperatures were stabilized by 1600 hours.
7/21/88	0100	111 3/6	End of Case E-5B. Production of 7.81 TPD MeOH and a productivity of 15.0 g-mole/hr-kg which is 83 % of autoclave.
	0600	116 3/6	Beginning of E-5C. Nominal conditions for this case are CO-Rich feed gas, 7,000 Sl/hr-kg cat, 250 °C, 750 psig, and 46 wt % catalyst. High space velocity (7000 l/hr-kg) and because of the safety factor on the internal heat exchanger, both internal and external heat exchangers are in service.
	1138	120	Slurry sample taken for determination of catalyst activity.
	1700	127 3/6	Line pressure from HYCO fluctuating wildly, thus feed rate to 27.10 is unstable. The line pressure did not stabilize until 1945, so approximately 3 hours of data does not reflect the actual performance of the PDU at the higher space velocities.
7/22/88	0025	134 5/6	Complete power outage. No flow to the 27.10 of either gas or slurry. GC's down.
	0032	134 7/8	Restarted 10.50 and flow of slurry resumed.
	0040	135	Gas flow restored to 27.10 but MeOH PDU running off HYCO line pressure, decided to back flow rates down to 120,000 SCFH until pressure is restored.
	0515	139 5/6	HYCO still off line and 01.10 suction pressure getting critically low. Started bringing in nitrogen to maintain suction pressure.

TABLE V.3

CHRONOLOGY RUN E-5 WITH CATALYST F21/0E75-44

(continued)

Date	Time	Cumulative Time On Production Gas (Hours)	Notes and Observations
	0600	140 3/6	HYCO reports they are up and running and pressure returning to feed hydrogen and CO. Start of ramping to high space velocity case and backing out nitrogen.
	1330	147 5/6	Restart high flow case E-5C.
7/23/88	0600	164 3/6	End of Case E-5C with a record MeOH production rate of 9.19 TPD and a productivity of 18.0 g-mole/hr-kg which is 79% of autoclave performance.
	0740	166	Begin reactor isolation; 10.50 pump speed slowed down; begin bypassing gas flow through the 27.11 demister and the 27.10 cyclone.
	0845	167	Reactor isolation complete and the level holding at 207 1/2".
	1100	169 3/6	Beginning of Case E-5D, nominal conditions of 5000 space velocity, isolated reactor, 482 F and 750 psig.
	1630	175	First slurry sample taken using the new reactor side arm slurry sampling system. Estimate that we loose approximately 5 lb of slurry in this sampling procedure.
	1740	176	The 27.10 demister pressure drop is trending upward.
7/24/88	0800	190 3/6	Noted a cyclic pattern to the %MeOH in the reactor effluent. Problem was the steam tracing on the sample line was not turned on and at night the line would cool and the MeOH would drop out.
	1230	195	The 27.10 Demister DP up to 4.25 psi. 27.12 liquid examined for solids, found to be fairly clear. Does not appear that catalyst is carrying over.
	1245	195 1/6	The 27.10 demister was back flushed with oil and the DP dropped to 1.9 psi. Cyclone DP dropped from 7.3 to 7.0 psi at the same time.
	1600	199 1/3	Since the demister wash at 1245 hours, the demister DP has climbed to 2.52 psi.

TABLE V.3

CHRONOLOGY RUN E-5 WITH CATALYST F21/0E75-44

(continued)

Date	Time	Cumulative Time On Production Gas (Hours)	Notes and Observations
	2200	203 1/3	DP on the demister dropped quickly from almost 4 psi to 1.9 psi. May have some sort of a break through.
7/25/88	0000	205 1/3	End of Case E-5D, with a MeOH production of 8.3 TPD and a productivity of 16.8 g-mole/hr-kg which is 91% of autoclave.
	0000	205 1/3	Begin dilution of reactor slurry concentration by slowly adding oil from the 27.14 to the 27.10 and allowing the slurry to spill over to the 27.13 through the cracked open V-1493s valve.
	0300	208 1/3	Dilution complete with the final slurry concentration of 33.8 weight percent solids.
	0400	209 1/3	Beginning of run E-5E, with nominal conditions of 10,000 space velocity, 35 weight percent catalyst, isolated reactor, 482 F and 750 psig.
	1000	215 1/3	Lab is seeing free oil in the MeOH sample for the first time since the high flow case. May be entrainment.
7/26/88	0500	234 1/3	Slurry sample taken from the reactor side arm slurry sampling system.
	0500	234 1/3	End of Case E-5E with a production rate of 8.9 TPD and a productivity of 30.3 g-mole/hr-kg which is 105% of autoclave.
	0750	236 1/6	Beginning of 1 hour shutdown period. HV-150-2 closed, no flow to the reactor. NDG scans taken to watch profile and estimate weight percent solids and catalyst loading.
	0850	237 1/6	HV-150-2 opened and flow restored to the reactor.
	1200	241 1/3	Gas holdup and the reactor profile have returned to the pre-shutdown conditions.
	1230	241 5/6	HV-150-2 closed for the 24 hour shut down period. No gas flow to the reactor.

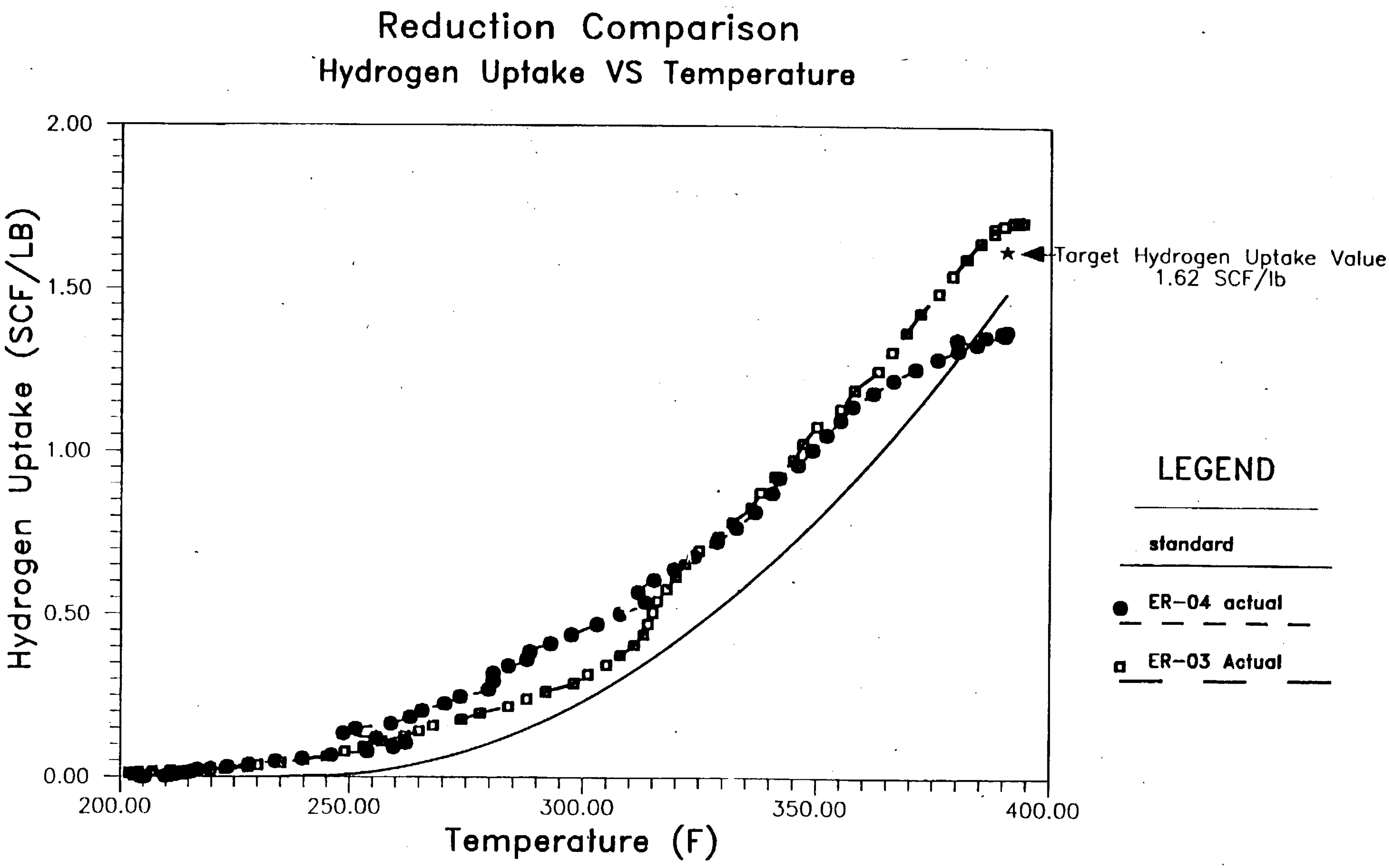
TABLE V.3

CHRONOLOGY RUN E-5 WITH CATALYST F21/0E75-44

(continued)

Date	Time	Cumulative Time On Production Gas (Hours)	Notes and Observations
7/27/88	1245	266 1/6	Utility oil temperature raised in an attempt to heat up the slurry in the 27.10.
	1308	267 1/2	HV-150-2 opened and flow to the 27.10 resumed. Flow ramped up slowly. Temperature profile in the reactor became uniform almost instantly after the gas flow was resumed.
	1540	270	Lined out in temperature and flow at 482 F and 145,000 SCFH.
	1600	270 1/3	Start recovery case E-6F with the nominal conditions of 34 weight percent solids, 10,000 space velocity, 482 F and 750 psig.
	1800	272 1/3	Severe thunderstorm that lasted until 1930 hours. Reactor temperature unstable thus performance will not be representative during this period of time.
7/28/88	0800	286 1/3	End of recovery case E-6F with a production rate of 8.6 TPD and a productivity of 29.6 g-mole/hr-kg which is 103% of autoclave. Recovery from the 24 hour shut down considered successful.
	1015	288 1/2	Beginning of final shutdown. Total correct on stream time was 259.4 hours. This on stream time excludes power outages and shutdown test where no feed gas was fed to the reactor.

Figure V.2



Reduction Comparison
Hydrogen Uptake VS Temperature
Hydrogen Uptake (SCF/LB)
2.00
1.50
1.00
0.50
0.00
200.00
250.00
300.00
350.00
400.00
Temperature (F)
Target Hydrogen Uptake Value
1.62 SCF/lb
LEGEND
standard
ER-04 actual
ER-03 Actual

The new internal heat exchanger was evaluated in Runs E-5B and E-5C. As shown in Figure V.3, removing the exothermic heat of reaction with either the external or internal heat exchanger had no observable effect on reactor performance. Again, the reactor was performing closer to the autoclave prediction than in the previous E-4 run. Operation at high space velocities and high methanol production rates was not possible using the internal heat exchanger alone. This was because the exchanger surface area was consciously designed to be low in order to achieve a measurable and accurate temperature difference between the utility oil and the reacting catalyst slurry at low production rates. The performance with high catalyst slurry loadings exceeded expectations, resulting in a high heat load on the internal heat exchanger and a large temperature differential between the slurry and heat transfer oil. Operating with large temperature differences would produce excessive thermal stresses and would exceed the design limits of the internal heat exchanger. Therefore, both the internal and external heat exchangers were used in Run E-5C to remove the heat of reaction. The new reactor system performed well in this mode of operation and daily methanol production ranged from 7.8 to 9.2 TPD, exceeding the previous production rates of 5.4 to 6.4 TPD at equivalent conditions and slurry loadings.

Run E-5D was the first test of the LaPorte reactor system operating without external slurry circulation. A higher methanol productivity was achieved in Run E-5D than in the equivalent Runs E-5A or E-5B using the external slurry loop (see Figure V.3). The increased productivity could be due to higher gas/liquid interfacial area or reduced backmixing in the absence of external liquid circulation. Clearly, in the external loop configuration the catalyst did not contribute significantly to the methanol production while circulating through the slurry loop external to the reactor. Thus, Run E-5D demonstrated that the use of the external slurry loop was not required or desirable for future PDU runs.

Runs E-5E and E-5F were completed at the end of July with a 34 wt% slurry. As shown in Figure V.3, the methanol productivity for Run E-5E improved relative to the previous cases and was slightly greater than autoclave performance. This indicated either improved mixing (reduced backmixing) and/or reduced mass transfer limitations at the more dilute slurry concentration.

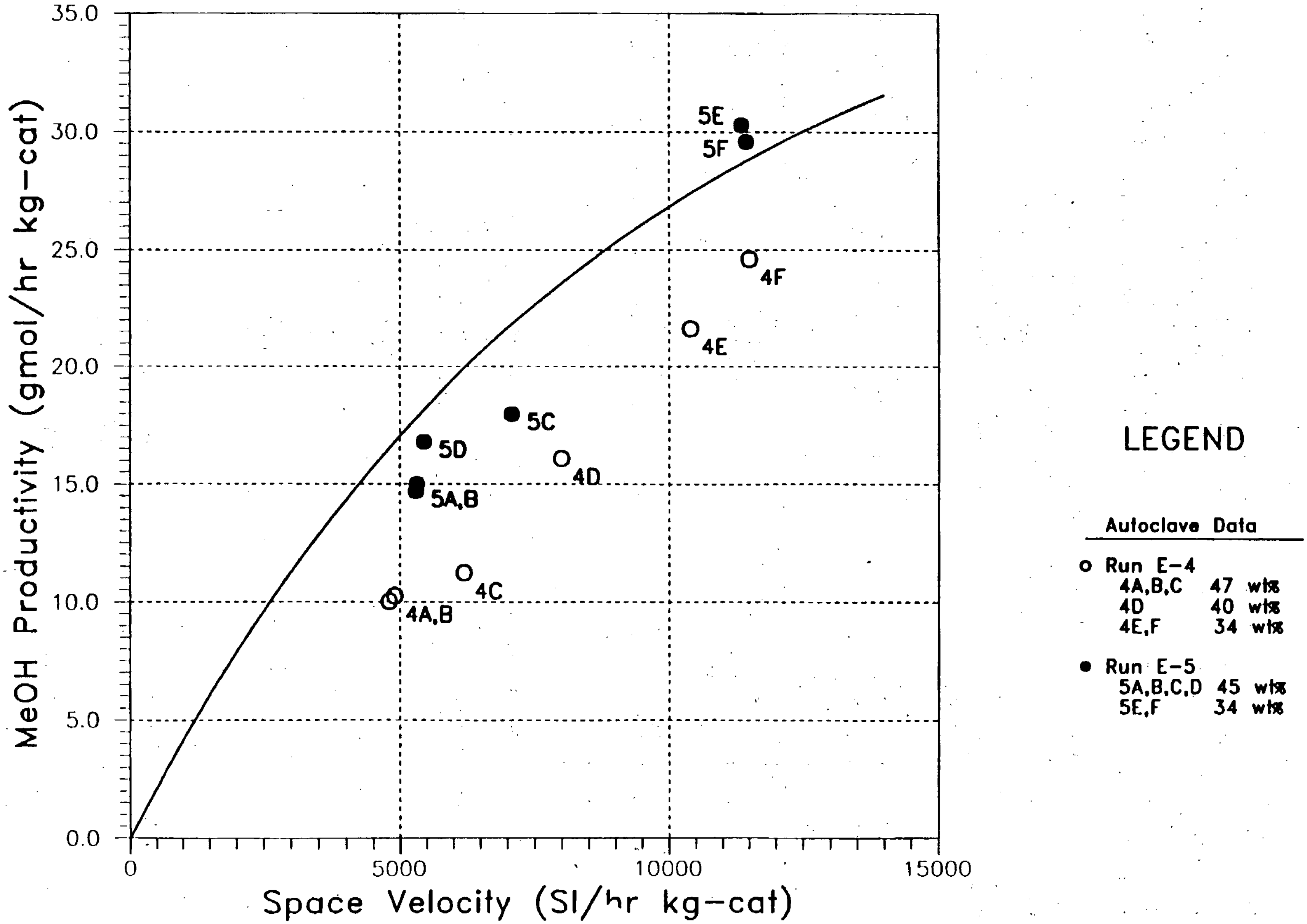
The next objective of the test program was to evaluate the ability of the new reactor system to maintain performance after either a planned or unplanned shutdown. With the elimination of the external circulation the catalyst suspension in the slurry phase is dependant solely on the agitation provided by the upward vapor phase velocity. It was possible that a temporary loss of feed gas would result in the catalyst forming a packed layer in the lower section of the reactor which would plug the inlet gas sparger and/or not refluidize. The test to evaluate this was carried out in two stages. In the first stage the reactor feed was stopped for one hour and then restarted. In the second stage the reactor was isolated for 24 hours and then restarted.

The first one-hour shutdown test was done at the completion of Run E-5E. NDG scans of slurry density as a function of reactor height immediately after the reactor feed flow was stopped, were uniform, indicating that the catalyst was evenly distributed in the slurry. Approximately 10-15 minutes into the test there was a significant density gradient, indicating that the catalyst had begun to settle out of the slurry. This density gradient continued to

Figure V.3

LAPORTE LPMEOH PDU REACTOR PERFORMANCE

Run E-5: CO-Rich Gas, 750 psia, 482°F



increase over the course of the shutdown. The feed flow was slowly reintroduced to the reactor after the one hour shutdown period without incident. The reactor temperature and feed flow were back at pre-shutdown levels within 30 minutes. These conditions were held steady for three hours, during which time the volume fraction gas holdup and the reactor effluent methanol concentration also stabilized at pre-shutdown levels.

The 24-hour shutdown test was begun immediately after it was concluded that the reactor performance had fully recovered from the one- hour test. As in the previous test, the reactor feed flow was stopped and the reactor was isolated and maintained at pressure. Shortly after shutdown the reactor slurry was cooled to 425°F using the internal heat exchanger to minimize continued reactions.

The same catalyst settling behavior was observed as seen in the previous test. The final degassed slurry height was 160 inches from the bottom head of the reactor and the nuclear density gauge was able to measure slurry densities in the region from 68 inches from the bottom head to the top liquid height. Figure V.4 illustrates the stable slurry density profile that was observed 15 hours after the feed gas to the reactor was stopped. There were two distinct regions within the settled slurry, a dense catalyst layer in the bottom 65% of the slurry with a clear oil layer on top. The dense catalyst layer varied from 42 to 45 wt% catalyst as oxide, within the observable region.

The feed flow was reintroduced to the reactor 24 1/2 hours after start of the shutdown period and the reactor conditions were stable within 2 1/2 hours. Run E-5F was done immediately after the shutdown test at the same conditions as the previous Run E-5E. As shown in Figure V.3 the reactor returned smoothly to pre-shutdown production rates.

Productivity levels achieved in Run E-5F demonstrated that the process was resilient in handling deliberate shutdowns of one hour and 24 hour durations. Four unplanned power outages during the month of July also demonstrated the reliability of this process after unscheduled shutdowns.

The external axial cyclone vapor/liquid separator performed well, with a average pressure drop of 7.6 psi. The internal demister pressure drop initially cycled between 2 and 5 psi between backflushings with clean process oil and appeared to be operating well. However, during Case E-5D the pressure drop across the demister dropped quickly from 4 psi to 1.9 psi. This was assumed to be evidence that the demister had become plugged and dislodged, which was confirmed upon post run inspection.

Internal Heat Exchanger Evaluation

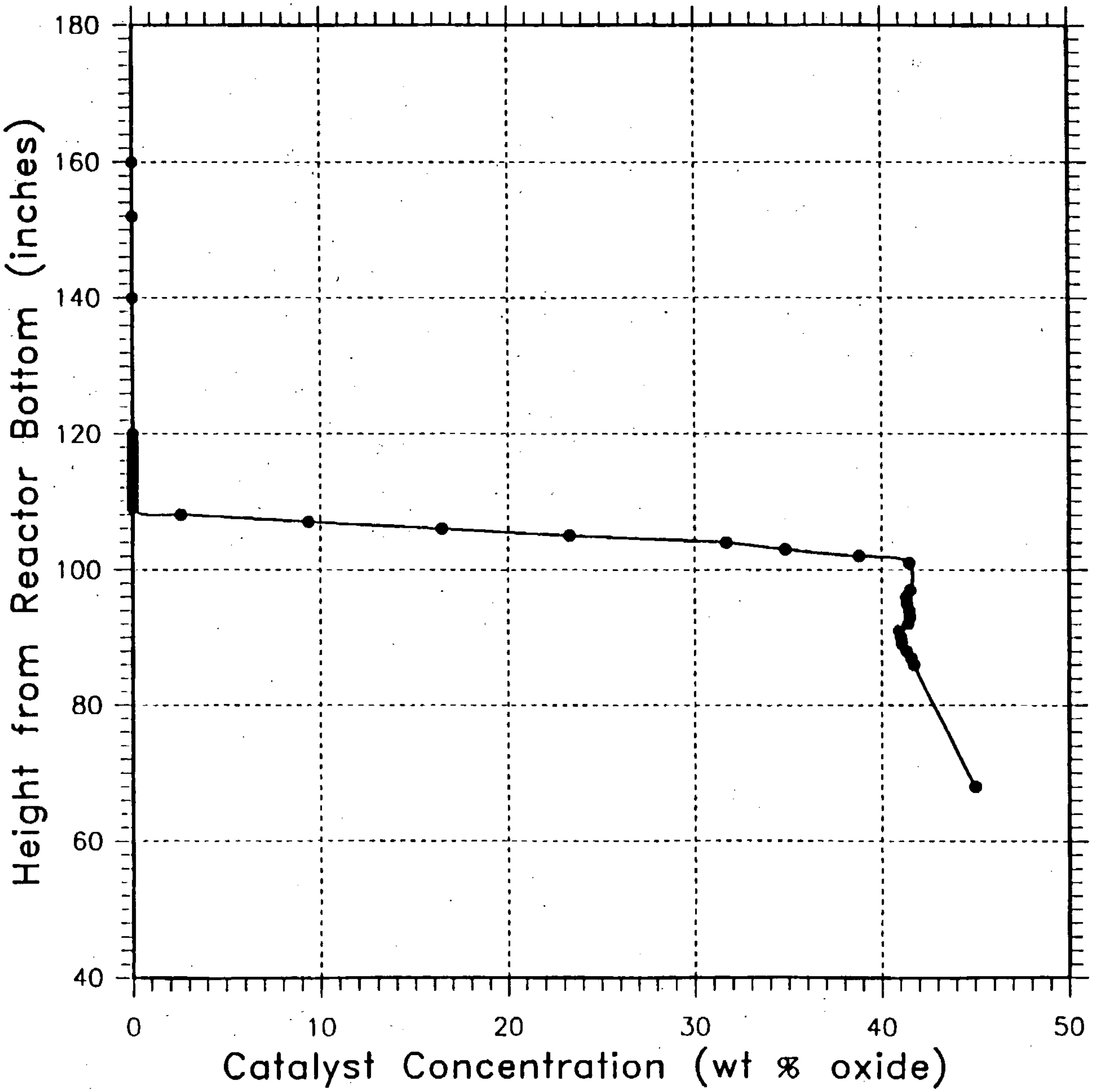
The overall heat transfer coefficient for the internal heat exchanger was calculated from the Run E-5 utility oil data. Hence, the measured overall heat transfer coefficient was based upon the actual utility oil flowrate, temperature rise, the predicted utility oil heat capacity, and the reactor temperature.

A quantitative comparison of the predicted and measured overall coefficients indicated that the average absolute error was 8.6% for the five cases of Run E-5 (see Table V.4). In all cases the uncertainty in the measured

Figure V.4

24 HOUR SHUTDOWN TEST

SETTLED SLURRY SOLIDS DENSITY PROFILE



coefficients was low from a propagation of errors analysis, at approximately 11%. Hence, the predictions of the overall coefficients, using the Sieder-Tate and Deckwer correlations, were accurate within the range of uncertainty of the plant data.

TABLE V.4

27.10B INTERNAL HEAT EXCHANGER COEFFICIENTS
(Btu/hr-ft^2-°F)

Case	E5B	E5C	E5D	E5E	E5F
Catalyst Conc. (wt%)	44.9	45.9	45.0	34.1	33.9
Overall Coefficient					
Predicted	94.3	43.8	92.1	95.5	96.3
Measured	74.2	42.0	79.1	93.5	97.8
Percent Error	-21.3	-4.1	-14.1	-2.1	1.6
Slurry Side Coefficients					
Predicted	295.5	320.5	294.8	303.1	303.0
Measured	159.7	245.5	193.3	284.4	317.6
Percent Error	-46.0	-23.4	-34.4	-6.2	4.8

These uncertainties were based on generously assumed uncertainties in the physical readings and properties of 0.5% in the heat exchanger area, 5.0% in the utility oil heat capacity, 10.0% in the utility oil flowrate, 1.0% in both the utility oil temperature change and the log mean temperature across the heat exchanger, and 15.0% in the predicted internal tube-side heat transfer coefficient. The typical oil flowrate averaged 156 gpm and the slurry to utility oil temperature difference averaged 34 to 79°F.

When the predicted overall heat transfer coefficients were plotted versus the superficial gas velocity, a narrow band of curves was produced (Figure V.5). These curves were based upon the average conditions of the five cases; Case C conditions were much different. Using only slurries of 30 to 50 wt% oxide, the predicted coefficients ranged from 82 to 85 Btu/hr-ft^2-°F at 0.10 ft/sec, to 98 to 101 Btu/hr-ft^2-°F at 0.80 ft/sec. Cases E-5E and E-5F were both just outside the band of predicted values. The predicted band was obviously within the range of uncertainty for these cases. However, Cases B, C, and D were further from the predicted values and were not correct even within their ranges of uncertainty.

Considering the physical setup of the reactor, Cases B and C would be expected to be less accurate due to the effect of the external oil circulation on heat transfer. Additionally, Case C involved the use of both the internal and external slurry heat exchanger, thereby increasing the error, since the utility oil flowrate through the exchanger was much lower. However, Cases D, E, and F should have been accurate since they had no external oil slurry circulation flow and relied entirely on the internal heat exchanger. Cases E and F were both within 3% of predicted, but Case D had an overall coefficient which was 14% low for unknown reasons.