TABLE V-1

LAPORTE LPMEOH PDU

OPERATING CONDITIONS FOR RUN E-2 (14-20 JUNE 1984)

Catalyst: F21/OE75-29
Gas Type: Balanced*
Reactor-Pressure: 6,310 kPa (915 psia)

Case	Reactor Temperature °C (°F)	Superficial Gas Velocity cm/s (ft/s)	Superficial Liquid Velocity cm/s (ft/s)	Space Velocity l/kg-hr	Slurry Conc. wt% Oxide	Hrs at Condition
E-2A	250 (482)	8.9 (0.29)	4.5 (0.15)	4,230	47.9	16.5
E-2B	250 (482)	11.7 (0.38)	4.7 (0.15)	5,000	48.9	5.5
E-2C	250 (482)	11.6 (0.38)	4.7 (0.15)	5,450	46.5	14.0
E-2D	250 (482)	11.6 (0.38)	5.9 (0.19)	5,490	46.5	5.5
E-2E	250 (482)	13.3 (0.44)	6.1 (0.20)	6,380	46.0	10.5
E-2F	250 (482)	13.5 (0.44)	5.9 (0.19)	7,250	44.0	13.0
E-2G	250 (482)	13.8 (0.45)	4.5 (0.15)	7,980	42.0	21.0
E-2H	235 (455)	13.0 (0.43)	4.3 (0.14)	8,020	41.0	12.5
E-2I	265 (509)	13.8 (0.45)	4.3 (0.14)	8,070	41.0	27.0
E-2J	250 (482)	13.6 (0.45)	5.7 (0.19)	12,600	32.0	6.0
E-2K	260 (500)	14.0 (0.46)	5.7 (0.19)	11,250	32.5	10.0
E-2L	250 (482)	7.7 (0.25)	4.2 (0.14)	6,360	32.6	4.0
						145.5

*See Section III, Table III-2.

TABLE V-2

LAPORTE LPMEOH PDU

RUN E-2 CHRONOLOGY WITH CATALYST F21/0E75-29

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
6/14/84	2100	-	Begin concentration of slurry. Pressurizing PDU to 3,550 kPa (515 psia) with N_2.
	2230	0	Synthesis gas to reactor at 2,840 Nm3/h (108,000 SCFH), T=225°C (437°F), P=5,000 kPa (725 psia).
	2330	1-0	Continuing to increase slurry concentration; reactor pressure 6,310 kPa (915 psia).
6/15/84	0200	3-1/2	Case E-3A begun T=250°C (482°F), P=6,310 kPa (915 psia), VL=4.5 cm/s (0.15 ft/s), VG=8.9 cm/s (0.29 ft/s), slurry concentration 47.9 wt% oxide.
	1145	13-1/4	Compressor trip.
	1200	13-1/2	Resume synthesis gas flow.
	1245	14-1/4	Compressor trip.
	1315	14-3/4	Resume synthesis gas flow.
	1500	16-1/2	Case E-2A ended. Begin Case E-2B. T=250°C (482°F), P=6310 kPa (915 psia), V_L=4.7 cm/s (0.15 ft/s), VG=11.7 cm/s (0.38 ft/s), slurry concentration 48.9 wt% as oxide.
	1915	20-3/4	Liquid flow rate through the reactor increased due to a nonuniform density profile in the reactor.

TABLE V-2

LAPORTE LPMEOH PDU

RUN E-2 CHRONOLOGY WITH CATALYST F21/0E75-29

(continued)

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
6/15/84	2030	22-0	End of Case E-2B. Begin Case E-2C. T=250°C (482°F), P=6,310 kPa (915 psia), VL=4.7 cm/s (0.15 ft/s), VG=11.6 cm/s (0.38 ft/s), slurry concentration 46.5 wt% oxide.
	2045	22-1/4	Increased seal flush flow to dilute slurry.
6/16/84	1045	36-1/4	End of Case E-2C. Begin Case E-2D. T=250°C (484°F), P=6,310 kPa (915 psia), VL=5.9 cm/s (0.19 ft/s), VG=11.7 cm/s (0.38 ft/s), slurry concentration 46.5 wt% as oxide. On increasing liquid rate, reactor temperature rose 4°C (7°F).
	1100	36-1/2	Reduced liquid rate back to case condition.
	1600	41-1/2	End of Case E-2D. Begin Case E-2E. T=250°C (482°F), P=6,310 kPa (915 psia), V_L=6.1 cm/s (0.19 ft/s), VG=13.3 cm/s (0.44 ft/s), slurry concentration 46.0 wt% as oxide.
	2015	45-/3/4	Slurry pump tripped; restarted within 15 minutes.
6/17/84	0215	51-3/4	End of Case E-2E. Begin Case E-2F. T=250°C (482°F), P=6,310 kPa (915 psia), VL=5.9 cm/s (0.19 ft/s), VG=13.5 cm/s (0.44 ft/s), slurry concentration 44.0 wt% as oxide.

TABLE V-2

LAPORTE LPMEOH PDU

RUN E-2 CHRONOLOGY WITH CATALYST F21/DE75-29

(continued)

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
			Unstable density gauge reading observed at 5% elevation; slurry being diluted further.
6/17/84	1530	65-0	End of Case E-2F. Begin Case E-2G. T=251°C (484°F), P=6,310 kPa (915 psia), VL=4.5 cm/s (0.15 ft/s), VG=13.8 cm/s (0.45 ft/s), slurry concentration 42.0 wt% oxide.
6/18/84	1245	86-1/4	End of Case E-2G. Begin Case E-2H. T=236°C (457°F), P=6,310 kPa (915 psia), VL=4.3 cm/s (0.14 ft/s), VG=13.0 cm/s (0.43 ft/s), slurry concentration 41.0 wt% oxide.
	1815	91-3/4	Density gauge indicates instability in bottom of reactor continues; momentarily pulsing liquid flow to reactor. Instability remains.
6/19/84	0100	98-1/2	End of Case E-2H. Begin Case E-2I. T=264°C (507°F), P=6,310 kPa (915 psia), VL=4.2 cm/s (0.14 ft/s), VG=13.8 cm/s (0.45 ft/s), slurry concentration 41.0 wt% oxide.
	0915	106-3/4	End of Case E-2I.
	1145	109-1/4	Transferring a portion of the slurry from the circulation loop to the prep tank. Beginning dilution step.
	1345	111-1/4	Slurry pump tripped.
	1400	111-1/2	Circulation re-established.

TABLE V-2

LAPORTE LPMEOH PDU
RUN E-2 CHRONOLOGY WITH CATALYST F21/OE75-29
(continued)

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
	1915	116-3/4	Slurry pump tripped.
6/19/84	1930	117-0	Circulation re-established. Continuing dilution of slurry.
	2215	119-1/4	Second slurry transfer to slurry prep tank.
6/20/84	0415	125-3/4	Begin Case E-2J. T=251°C (484°F), P=6,310 kPa (915 psia), V_L=5.7 cm/s (0.14 ft/s), VG=13.6 cm/s (0.45 ft/s), slurry concentration 32.0 wt% oxide.
			Briefly reduced slurry circulation flow rate to check effect of liquid velocity on carry-over.
	1015	131-3/4	End of Case E-2J. Begin Case E-2K. T=260°C (500°F), 6,310 kPa (915 psia), VL=5.7 cm/s (0.19 ft/s), VG=14.0 cm/s (0.46 ft/s), slurry concentration 32.5 wt% oxide.
	2000	141-1/2	End of Case E-2K. Begin Case E-2L. T=251°C (484°F), 6,310 kPa (915 psia), VL=4.2 cm/s (0.14 ft/s), VG=7.7 cm/s (0.25 ft/s), slurry concentration 32.5 wt% oxide.
6/21/84	0000	145-1/2	End of Case E-2L. Normal plant shutdown procedures begun.
	0900	-	Slurry drained to prep tank; oil rinse of slurry loop started.
	0200	-	Drained oil from oil circulation loop.

D. Discussion of Results

Solids Concentration Measurement and Density Profiles

Solids concentration measurements were performed in several ways. Slurry samples taken from the PDU slurry loop during the run could be directly measured for solids concentration. Also, the solids concentration could be estimated from nuclear density gauge (NDG) measurements under conditions of no gas flow through the reactor. The solids concentration as determined by both methods is within 1-2 wt% absolute. Table V-3 and Figure V-1 show the change in solids concentration during Run E-2.

At the beginning of methanol synthesis operation, the solids concentration was measured at 46.5 wt% (as oxide). An NDG survey (No. E-2-04) showed a uniform catalyst density profile (Figure V-2). Subsequently, the slurry concentration was further increased to 49 wt% by reducing the 10.50 pump seal flush flow. After 20 hours on synthesis gas, it was observed that the NDG reading at a position 54-in. (137-cm) above the tray had become unstable. The signal fluctuated rapidly between 210 and 250 mV as opposed to a 3-5 mV variation during normal readings. The next two NDG surveys (Numbers E-2-05 and E-2-06) revealed a distinct convex profile when the catalyst density was plotted as a function of reactor height (Figure V-2).

The apparent high catalyst density in the lower portion of the reactor could be attributed to either solids accumulation or low gas holdup. Figure V-2 was developed based on the assumption that slurry concentration was constant throughout the reactor/slurry loop and gas holdup was changing along the reactor. The change in gas

TABLE V-3

LAPORTE LPMEOH PDU

CHANGE OF SOLIDS CONCENTRATION DURING RUN E-2

Time On-Stream, Hr.		A, Measured Solid Conc. wt%	B, Solid Conc. as Reduced Cat., wt%	C, Solid Conc. as Original Oxide, wt%	Solid Conc. as Original Oxide by NDG, wt%
0	(Before Reduction)	41.14	39.47	42.48	41.7
0	(33 hrs. into Reduction)	42.19	40.51	44.48	
0	(End of Reduction)	43.75	42.05	46.05	
7.25		45.62	43.91	47.94	
21.5		46.53	44.81	48.85	
36.0		44.14	42.44	46.45	
58.5		43.10	41.41	45.40	
85.0		38.66	37.03	40.89	
107.5		39.14	37.50	41.38	
111.0					38.36
117.0					35.53
130.0		30.02	28.58	32.01	
141.0		30.47	29.02	32.48	

$$B = \frac{A/(1-0.067)}{A/(1-0.067) + (100-A)}$$

(0.067 is equal to the weight fraction change due to reduction)

$$C = \frac{B/(1-0.15)}{B/(1-0.15) + (100-B)}$$

(0.15 is the sum of the weight fraction change due to reduction and lose-on-ignition (LOI))

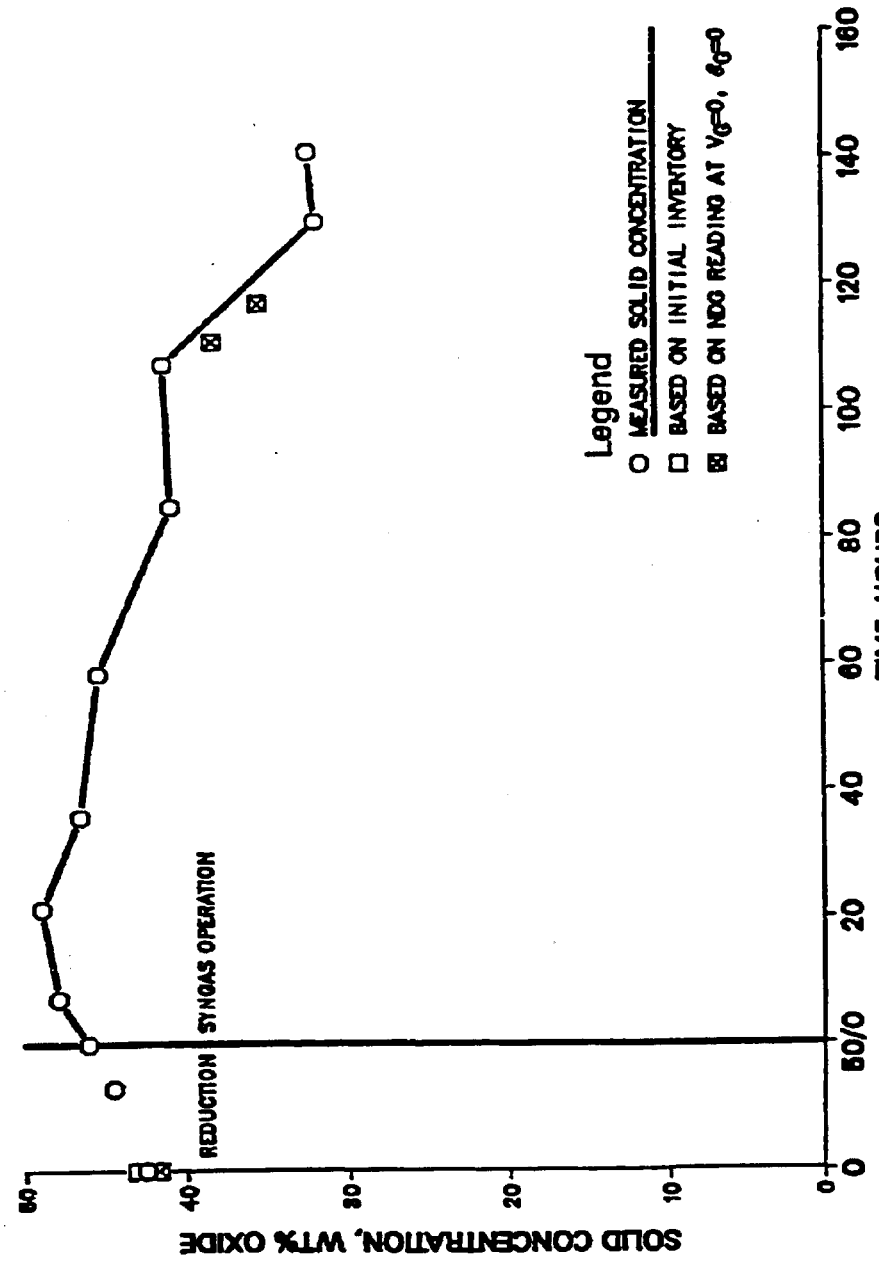
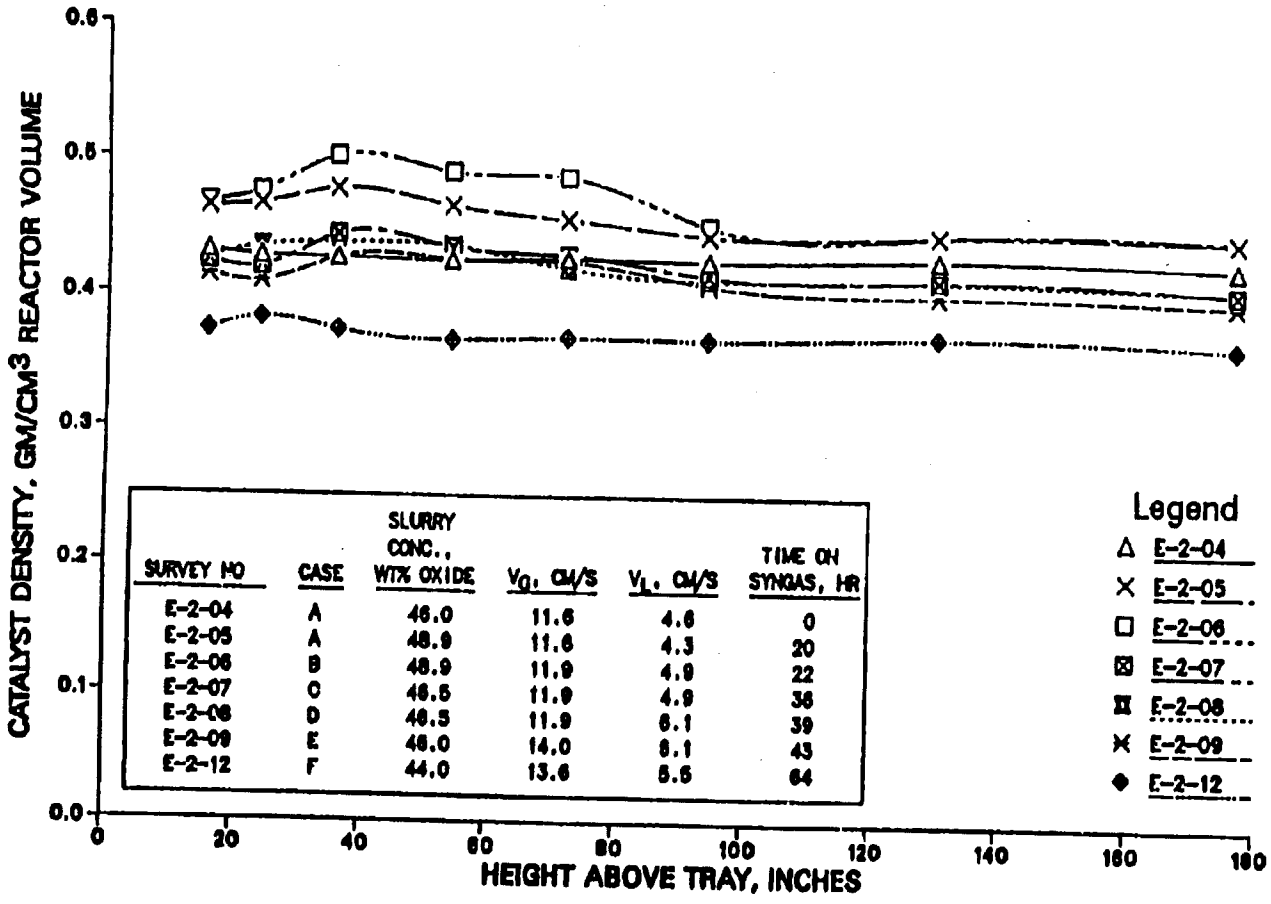


Figure V-1. LaPorte LPMEOH PDU Change of Solids Concentration During Run E-2



SURVEY NO	CASE	SLURRY CONC., WT% OXIDE	V_G, CM/S	V_L, CM/S	TIME ON SYNGAS, HR
E-2-04	A	46.0	11.6	4.6	0
E-2-05	A	48.9	11.6	4.3	20
E-2-06	B	48.9	11.9	4.9	22
E-2-07	C	46.5	11.9	4.9	36
E-2-08	D	46.5	11.9	6.1	39
E-2-09	E	46.0	14.0	6.1	43
E-2-12	F	44.0	13.6	5.5	64

Figure V-2. LaPorte LPMEOH PDU Catalyst Density Profile for Run E-2 (June 1984)

holdup through the reactor could be explained by postulating that large gas bubbles were being formed at the bubble cap tray, which would account for low gas holdup in this region. These bubbles could then break up as they rise through the reactor, thus yielding higher holdups. However, if gas holdup is assumed constant along the reactor, the NDG readings predict an unrealistic high solids concentration developing in the bottom of the reactor. It is possible, though, that a combination of the two phenomena was occurring.

In an attempt to restore a uniform density profile, the slurry concentration was diluted to 46.5 wt% (as oxide) and the superficial liquid velocity was increased to 6.1 cm/s (0.20 ft/s). The change of operating conditions reduced the density profile somewhat, but the profile was still distinctly defined (NDG surveys E-2-07 and E-2-08). A subsequent increase in the superficial gas velocity to 15 cm/s (0.46 ft/s) did not appear to have much effect on the density profile (NDG survey E-2-09). The slurry concentration was further diluted to 44 wt% (as oxide), at which time a uniform density profile in the reactor was restored (NDG survey E-2-12).

It appears that a threshold slurry concentration exists (approximately 45 wt% oxides), above which a catalyst density profile can occur in the LaPorte LPMEOH PDU reactor. The methanol synthesis reaction may also be contributing to this effect, since uniform solids concentration was detected at 46 wt% before the introduction of synthesis gas. The occurrence of a reaction appears to change the rheological properties of the slurry in some manner. The profile seemed to both develop and disappear slowly when the slurry concentration reached the threshold condition. More studies on the hydrodynamics of bubble-column reactors and the methanol synthesis catalyst slurry properties are required in order to understand the development of the density profile observed in the LaPorte PDU reactor.

LaPorte LPMEOH PDU Performance

One of the objectives of Run E-2 was to determine the performance of the LaPorte LPMEOH PDU at high slurry concentrations. Early data at 48 wt% slurry concentration indicated that the methanol productivity in the LaPorte LPMEOH PDU was not as high as projected by the autoclave data. Furthermore, nuclear density gauge surveys revealed an apparent accumulation of solids or decrease in gas holdup in the bottom of the reactor. Subsequently, a series of process variable scans was performed in order to identify the problems and to improve PDU performance. The results of the process variable scans are summarized in Table V-4. The CO conversion and methanol productivity for different operating conditions are plotted as a function of space velocity in Figures V-3 and V-4, and the detailed data sheets generated by the Data Acquisition System are attached in Appendix F.

As the run progressed, process parameters were changed to determine their impact on reactor performance. The variables included superficial liquid velocity (Cases E-2C and E-2D), superficial gas velocity (Cases E-2D and E-2E), and reactor temperature (Cases E-2G through E-2I). When these results were compared with the autoclave data, no improvement in methanol productivity was observed. The decrease in slurry concentration from 48.9 wt% (Case E-2B) to 41 wt% (Cases E-2H/I) also had no effect on performance. Further dilution to 32 wt% (Case E-2J) yielded a relative performance equaling 60% of the autoclave results. However, no further improvement was measured when reactor temperature was varied (Case E-2K). A maximum approach to the autoclave data of 65% was achieved when the space velocity was reduced by 50% (Case E-2L).

Table V-5 lists the results of the liquid product analyses performed during Run E-2. Methanol and by-product formation appeared to be typical for the balanced feed gas and insensitive to the changes in process parameters during Run E-2.

TABLE V-4

LAPORTE LPMEOH PDU
AVERAGE DATA SUMMARY FOR RUN E-2

Case	Date	Balance Period	Temp. °C	Press. kPa	VG cm/s	VL cm/s	Space Velocity l/hr-kg	Slurry Conc. wt% ox	CO Conv. %	Appr. to Equil. °C	MeOH* Prod. gmol/hr-kg	Hours on Syngas
E-2A	15 June	1300-1100	250	6,310	8.9	4.5	4,230	47.9	35.0	36.5	12.71	4-1/2 12-1/2
E-2B	15 June	1800-2400	249	6,310	11.7	4.7	5,000	48.9	21.3	51.0	9.9	19-1/2 25-1/2
E-2C	16 June	0700-1000	250	6,310	11.6	4.7	5,450	46.5	21.0	53.8	10.3	32-1/2 35-1/2
E-2D	16 June	1100-1500	251	6,310	11.7	5.9	5,490	46.5	21.1	48.2	11.36	36-1/2 40-1/2
E-2E	16 June-17 June	1600-0200	250	6,310	13.3	6.1	6,380	46.0	19.6	52.2	12.65	41-1/2 51-1/2
E-2F	17 June	0300-1500	250	6,310	13.5	5.9	7,250	44.0	19.6	51.4	13.0	52-1/2 64-1/2
E-2G	18 June	0100-1200	251	6,310	13.8	4.5	7,980	42.0	18.5	56.1	13.6	74-1/2 85-1/2
E-2H	18 June	1900-2400	236	6,310	13.0	4.3	8,020	41.0	15.6	54.8	11.2	92-1/2 97-1/2
E-2I	19 June	0400-0900	264	6,310	13.8	4.2	8,070	41.0	16.9	43.4	12.3	101-1/2 106-1/2
E-2J	20 June	0500-1000	251	6,310	13.6	5.7	12,600	32.0	18.8	56.0	20.9	126-1/2 131-1/2
E-2K	20 June	1400-2000	260	6,310	14.0	5.7	11,250	32.5	19.1	36.7	18.7	135-1/2 141-1/2
E-2L	20 June	2100-2400	251	6,310	7.7	4.2	6,360	32.5	28.6	41.6	15.5**	142-1/2 145-1/2

*Based on overall balance.
**As calculated across the reactor.

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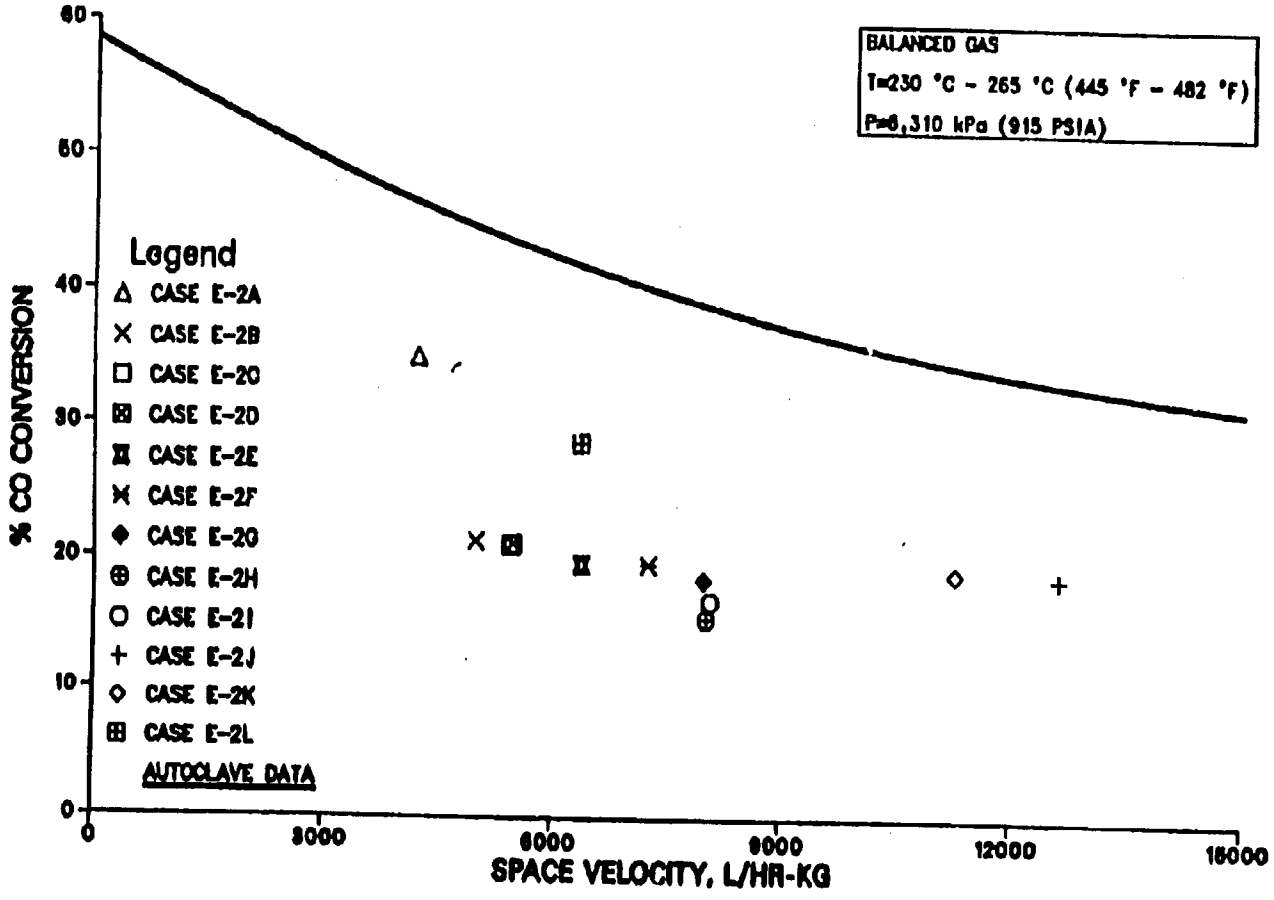

Figure V-3. LaPorte LPMEOH PDU CO Conversion vs. Space Velocity (Run E-2)

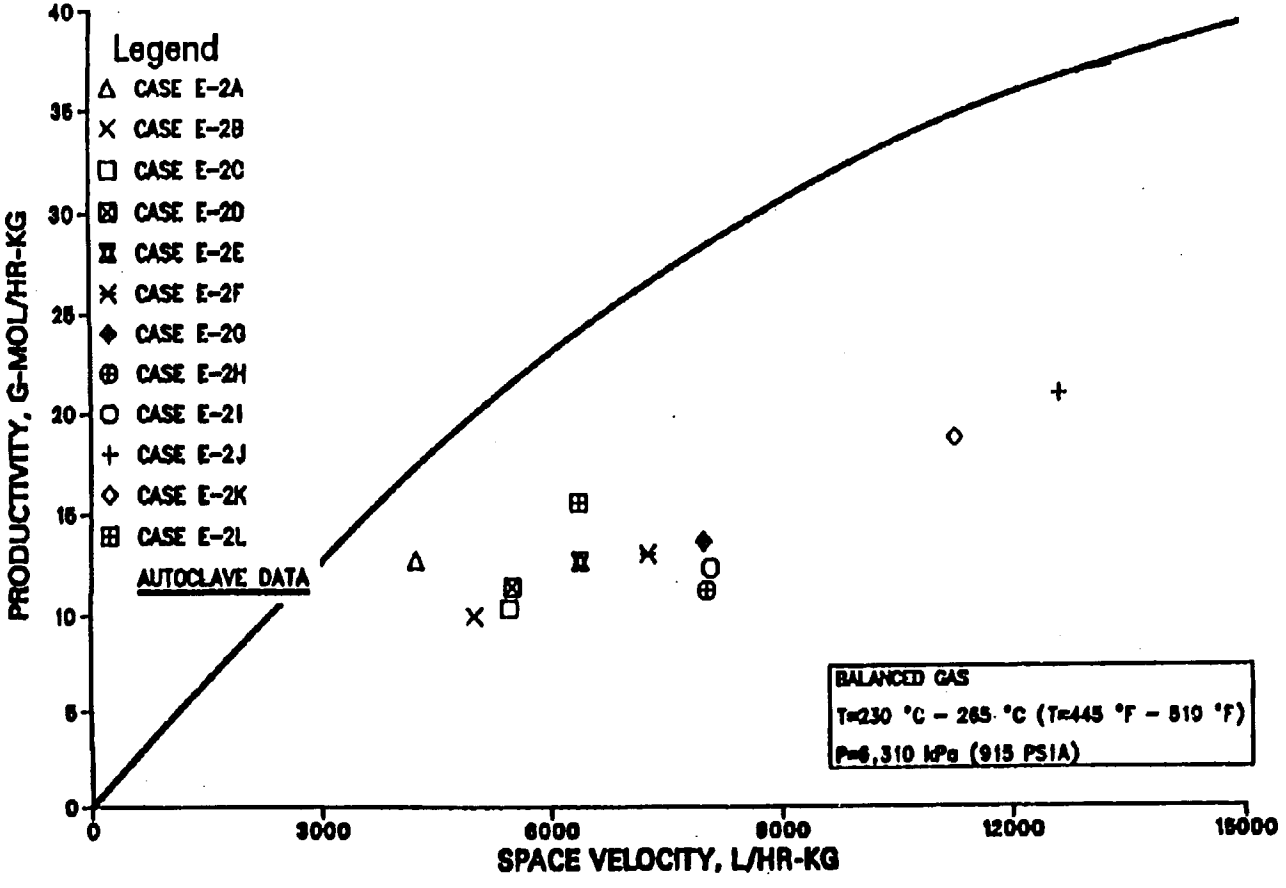

Figure V-4. LaPorte LPMEOH PDU MeOH Productivity vs. Space Velocity (Run E-2)

TABLE V-5

LAPORTE LPMEOH PDU

LIQUID PRODUCT ANALYSES FOR RUN E-2

Case	MEOH wt%	C_2H_5OH wt%	C_3H_2OH wt%	C_4H_9OH wt%	C_5H_4OH wt%	Esters wt%	Oil wt%	H_2O wt%	Total
E-2A*	95.00	-	-	-	-	-	-	1.40	96.4%
E-2B	95.52	0.09	0.02	0.01	0.00	0.27	1.36	2.72	100%
E-2C	95.56	0.09	0.02	0.01	0.00	0.27	1.33	2.72	100%
E-2D	95.56	0.09	0.02	0.01	0.00	0.27	1.33	2.72	100%
E-2E	94.86	0.07	0.02	0.00	0.00	0.25	1.46	2.34	100%
E-2F	94.15	0.06	0.02	0.00	0.00	0.24	1.59	3.95	100%
E-2G	94.25	0.06	0.02	0.00	0.00	0.25	1.54	3.89	100%
E-2H	94.27	0.04	0.01	0.00	0.00	0.25	1.46	3.97	100%
E-2I	94.28	0.03	0.01	0.00	0.00	0.23	1.36	4.10	100%
E-2J	94.60	0.06	0.02	0.00	0.00	0.24	1.48	3.60	100%
E-2K	94.60	0.06	0.02	0.00	0.00	0.24	1.48	3.60	100%
E-2L	94.64	0.06	0.02	0.00	0.00	0.20	1.48	3.60	100%

*Other component analyses not available.

A follow-up laboratory program sponsored by EPRI was conducted to discern whether mass transfer or intrinsic catalyst activity was limiting the process performance. It was found that inadequate catalyst reduction at LaPorte was a major contributor to the lower than expected performance (Reference 5). Changes in the reduction procedure were identified to remedy this problem. Mass transfer limitations may have also contributed to reduced catalyst activity at high slurry loading, but this effect was masked by the inadequate catalyst reduction. Another high slurry concentration LaPorte run will be necessary to isolate the mass transfer effects.

Carbonyl/Slurry Sample/Trace Component Analyses

On 18-20 June 1984, a series of carbonyl surveys was performed at the LaPorte LPMEOH PDU. These dates corresponded to days four through six of Run E-2 operation. There were two principal reasons for conducting the surveys. First, the copper liner for the reactor was removed after Run E-1, leading to the possibility of iron carbonyl generation from the low alloy steel walls. Second, poor catalyst performance would lead to questions on the relative impact of catalyst poisoning. As a result, the reactor feed and effluent gases were checked for iron and nickel carbonyl levels.

The results of the carbonyl surveys are given in Table V-6. Iron and nickel carbonyl levels downstream of the reactor were below the detectable limit (5.0 ppbv), suggesting that carbonyl formation from the reactor walls was minimal. Low levels of iron and nickel carbonyl were found in the reactor feed gas. This was unexpected since the reactor feed was of balanced gas composition with a low CO partial pressure. Subsequent analyses at points upstream of the 01.10 feed gas compressor showed that iron carbonyl was being formed across the shellside of the 21.10 feed/product exchanger. The source of nickel carbonyl was traced to the feed compressor unit. It was discovered that some residual nickel contamination remained in the feed compressor suction filter even after the hydroblast performed following Run E-1.

TABLE V-6

LAPORTE LPMEOH PDU
CARBONYL SURVEY (18-20 JUNE 1985) RUN E-2
(Concentrations in ppbv)

Date	Hours on Synthesis Gas	02.60B Out (Feed Gas to Reactor) Fe	Ni	27.14 Out Fe	Ni	21.10 Inlet Tubeside Fe	Ni	01.13 Out (Feed to 21.10) Fe	Ni	01.10 Out Fe	Ni	01.10 In Fe	Ni
6/18/84	83.50-87.50	36.0	10.0	<5.0	<5.0	<5.0	<5.0						
6/18/84	87.75-91.00	9.0	12.0	<5.0	<5.0	<5.0	<5.0						
6/18-19/84	87.75-91.00	480.0	27.0					8.0	20.0				
6/19/84	111.00-114.00							<5.0	6.0*	12.0	39.0*	8.0	14.0
6/19-20/84	115.00-131.00									18.0	58.0	<5.0	7.0
6/20/84	132.00-135.50							11.0	13.0				

*Recycle ratio was ~6:1 at this time in an effort to conserve CO_2.

On the basis of the data presented in Table V-6, the amount of metal carbonyls entering the reactor was sufficiently small so as to have a negligible effect on catalyst activity for a short run. However, the survey did confirm the need to replace the 21.10 feed/product exchanger (refer to Section III-D). In addition, chemical cleaning in the feed compressor area to remove residual nickel was necessary.

Catalyst analyses for Run E-2 are shown in Table V-7. Between the end of reduction and early operation on synthesis gas, the copper state parameters showed a marked drop from the reduced catalyst values. The 10-hour N_2 hold following reduction is believed to have caused this change.

Postrun Inspection

Following the completion of Run E-2, the slurry loop was drained. Clean oil was charged and circulated through the loop. After the oil was drained, the PDU was shut down and the major process piping and vessels were opened and inspected.

The interior circumference of the bottom head of the reactor contained a layer of catalyst sludge which in spots extended 4-6 inches above the gas sparger. The amount of solids in this area was more than that found at the end of the 40-day run (E-1). The center of the sparger arrangement was clear of debris. The interior of the plenum/distributor of the bubble cap tray was relatively clean. The bubble cups and risers looked clean and clear with no evidence of blockage.

TABLE V-7

LAPORTE LPMEOH PDU
CATALYST ANALYSES FOR RUN E-2

Sample No.	Date	Hrs on Stream	Solids wt% (Oxide)	ESCA/AUGER Cu/Zn	ESCA/AUGER Cu+1/Cu°	XRD Cu° (old) Å	XRD Cu° (new) Å	XRD ZnO (old) Å	XRD ZnO (new) Å	BET Surface Area m²/g	TPV cc/g	ELEMENTAL (AAS) Fe ppmv	Ni ppmv	Cl ppmv	S ppmv
OIL 119*	6/14/84	59	43.8	.13	0.5	89	110	68	80	113.4	0.541	71	22	115	44
OIL 120	6/15/84	7.5	45.6	0.07	0.06	106	136	71	84	104.9	0.619				
OIL 122	6/15/84	16.0													
OIL 124	6/15/84	21.5	46.5	0.06	0.12	112	145	72	85			72	25	69	50
OIL 126	6/16/84	36.0	44.1			114	149	74	88						
OIL 128	6/17/84	58.5	43.1	0.06	0.13	114	149	71	84			106	27	116	44
OIL 130	6/18/84	85.0	38.7	0.06	0.24	105	134	76	91	75.2	0.472				
OIL 132	6/19/84	111.0	39.1	0.05	0.10	114	149	73	86			77	34	89	62
OIL 134	6/20/84	130.0	30.0	0.06	0.07	117	153	70	82						
OIL 137	6/20/84	141.0	30.5	0.06	<0.05	123	163	74	89	82.2	0.530	85	38	104	44

*Last sample at end of reduction procedure.

1544S-W2

For the most part, the remainder of the slurry loop was clean. The 27.13 primary separator had only an oily film on the manway and vessel walls. A small amount of debris was noted on the suction line of the 10.50 slurry pump. The remainder of the loop, including the 21.20 slurry heat exchanger, appeared clean.

The 21.10 feed/product exchanger and the 27.14 intermediate separator were also inspected. The exchanger tubes looked clean, but were coated with an oil film similar to that found in the 27.13. One of the heads contained a small amount of accumulated solids. The 27.14 head had an 1/8 - 1/4-in. layer of catalyst around the standpipe; otherwise, only an oil film was present.