VI. ACTIVITY MAINTENANCE (RUN E-3)

A. Objectives

An activity maintenance run (Run E-3), using catalyst powder F21/0E75-35 in the liquid-entrained mode of operation, began on 1 May 1985. The objectives of the run were twofold: 1) to demonstrate in-situ reduction at the PDU scale of operation, and 2) to demonstrate improved activity maintenance after metallurgical changes and chemical cleaning of the LaPorte LPMEOH PDU.

B. In-Situ Reduction

A 25 wt% slurry was prepared by charging 1,631 kg (3,597 lbs) of Freezene-100 oil to the 28.30 slurry prep tank and blending 545 kg (1,201 lb) of catalyst powder (F21/0E75-35) into the oil. Following the blending operation, the slurry was transferred from the 28.30 to the reactor slurry loop. The reduction proceeded smoothly and catalyst analyses indicated that the slurry had been properly activated using the in-situ reduction technique. Further information on the reduction activities is presented in the Supplementary Volume of this report.

C. Methanol Synthesis Operation

CO-rich synthesis gas (H_2/CO = 0.69) was brought into the reactor after the catalyst was activated in-situ, and the reactor conditions were changed to the first condition listed on Table VI-1. Two operating points were tested over the 40 days of operation. The first (Case E-3A) was held for the initial 94 hours to verify the catalyst activity and to establish baseline PDU performance. The second case (E-3B) was a brief test at a lower reactor temperature of 225°C (437°F). Reactor conditions were then returned to 250°C (482°F) (as in Case E-3A) for the remainder of the run to determine the activity maintenance characteristics of the catalyst.

TABLE VI-1

LAPORTE LPMEOH PDU

OPERATING CONDITIONS FOR RUN E-3 (3 MAY - 13 JUNE 1985)

CATALYST: F21/OE75-35

GAS TYPE: CO-RICH*

REACTOR PRESSURE: 5,270 kPa (765 PSIA)

Case	Temperature °C (°F)	Superficial Gas Velocity cm/s (ft/s)	Superficial Liquid Velocity cm/s (ft/s)	Space Velocity l/hr-kg	Slurry Conc. wt% (oxide)	Hrs. at Condition
E-3A	250 (482)	9.5 (0.31)	4.9 (0.16)	10,000	28	94
E-3B	225 (437)	9.1 (0.30)	4.9 (0.16)	11,300	25	23
E-3C	250 (482)	9.5 (0.31)	4.9 (0.16)	10,000	28	831
						948

*See Section III, Table III-2.

Table VI-2 lists the run chronology for the 40 days of operation. Overall the PDU performed well, while achieving a 97% on-stream factor and producing over 202 tons of crude methanol. A major portion of the downtime (34 hours) was due to an electrical fault in the motor for the 01.10/01.20 compressor. A replacement motor was installed, and synthesis gas was subsequently brought back into the PDU. The compressor outage, although unplanned, demonstrated the ability to maintain catalyst activity through an extended PDU shutdown. The PDU was shut down in a controlled manner on 13 June after 948 hours of operation.

D. Discussion of Results

The data summary for Run E-3 is presented in Table VI-3. Detailed data sheets generated by the Data Acquisition System are available in Appendix F. Figure VI-1 plots the CO conversion and methanol productivity as a function of space velocity for LaPorte PDU data, the autoclave data, and the results of a laboratory batch check of the catalyst powder used during Run E-3 (F21/0E75-35). The laboratory batch check was performed to check the intrinsic activity of the catalyst under conditions of negligible mass transfer resistance. The LaPorte PDU data represent the average values of the CO conversion and methanol productivity normalized to 10,000 l/hr-kg for Case E-3A. The data for Case E-3A is comparable to or slightly better than the autoclave data.

Figure VI-2 shows the CO conversion as a function of time on synthesis gas. The autoclave data are also presented in the figure for comparison. Note that the CO conversion data have been normalized to a space velocity of 10,000 l/hr-kg to provide a common basis of comparison between the LaPorte PDU data and the laboratory results. As seen in the figure, the CO conversion compares well with the autoclave predictions for the duration of the run. CO conversion decreased from 12.5% to 10.5% as compared to the predicted decline of 12.0% to 8.8% conversion based on the laboratory results.

TABLE VI-2

LAPORTE LPMEOH PDU
RUN E-3 CHRONOLOGY WITH CATALYST F21/OE75-35

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
5/3/85	1215		Reduction gas taken out of PDU. Syngas brought into the PDU. Reactor conditions T=225°C (437°F), P=5,270 kPa (765 psia).
	1430	0	Case E-3A begun: CO-rich gas, T=250°C (482°F), P=5,270 kPa (765 psia), VG=9.5 cm/s (0.31 ft/s), VL=4.9 cm/s (0.16 ft/s).
	2115	6-3/4	Methanol product flowing to storage.
	2150	7-1/3	Slurry pump (10.50) tripped; circulation restored by 2155 hours.
5/4/85	1620	25-5/6	Level visible in sight glass of Product Day Tank (22.16).
5/6/85	0100	58-1/2	High nitrogen concentration in fresh feed (0.82 mol%).
5/6/85	1230	70	Nitrogen in fresh feed returned to normal level (0.14 mol%).
5/7/85	1240	94-1/6	Case E-3A ended. Begin case E-3B. T=225°C (437°F), P=5,270 kPa (765 psia), VG=9.1 cm/s (0.30 ft/s), VL=4.9 cm/s (0.16 ft/s).
5/8/85	1115	116-3/4	Case E-3B ended. Begin case E-3C. T=250°C (482°F), P=5,270 kPa (765 psia), VG=9.5 cm/s (0.31 ft/s), VL=4.9 cm/s (0.16 ft/s).
5/14/85	0900	258-1/2	Slurry pump tripped; circulation restored by 0905 hours.
	1445	264-1/4	Slurry pump tripped; circulation restored by 1450 hours.

TABLE VI-2

LAPORTE LPMEOH PDU RUN E-3

RUN E-3 CHRONOLOGY WITH CATALYST F21/OE75-35

(continued)

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
5/23/85	0930	475	Condensed oil filter (22.51A) removed and replaced. Approximately 3.2 kg (7 lb) of oil soaked catalyst was adhering to the filter.
6/1/85	0550	687-1/3	01.10/01.20 compressor shutdown due to an electrical fault in the compressor motor.
	0915	--	Started N_2 flow through reduction circuit to complete purge of plant. Conditions: 170 Nm3/h (6,400 SCFH) N_2 at 860 kPa (125 psia), reactor temperature = 152°C (305°F).
	1000	--	Secured reduction circuit and blocked PDU in under N2.
	1312	--	Opened a mixture of 1 mol% H_2 in N_2 to PDU to offset any oxidation effects that the CO2 dissolved in the liquid phase may have on the catalyst. Conditions: Gas flow = 140 Nm3/h (5,300 SCFH), T=150°C (301°F).
	1900	--	Increased H_2/N_2 mixture flow to 175 Nm3/h (6,700 SCFH). Reactor temperature 140°C (285°F).
6/2/85	0700	--	New compressor motor being installed.
	1150	--	01.10/01.20 compressor started; plant pressure being brought up to 3,375 kPa (490 psia).
	1240	--	Beginning heat up of slurry to 220°C (428°F) in preparation for syngas addition.

TABLE VI-2

LAPORTE LPMEOH PDU RUN E-3

RUN E-3 CHRONOLOGY WITH CATALYST F21/OE75-35

(continued)

Date	Time	Cumulative Time On Synthesis Gas (Hours)	Milestone
	1550	687-1/3	Syngas brought back into plant. Resuming Case E-3C.
6/3/85	0400	699-1/2	Plant operating at steady-state conditions.
6/13/85	1230	948	Plant shutdown begun; syngas taken out of PDU. Case E-3C ended.
	1900	--	Transferred slurry to slurry prep. tank to prepare for PDU postrun inspection.

TABLE VI-3

LAPORTE LPMEOH PDU

AVERAGE DATA SUMMARY FOR RUN E-3

Run No.	Date	Time	Temp. °C	Press. kPa	Space Vel. L/Hr-Kg	Vg,S cm/s	VL,S cm/s	CO Conver. %	Appr. To Equil. %	MeOH In React. Effl. %	MeOH Prod. G-Mol/ Hr-Kg	Slurry Concen. wt% Oxide	Net MeOH Kg/D	Hrs. On Syngas
E-3A	5/4/85	0400-0800	250	5,270	9,800	9.5	4.9	13.25	71.62	7.59	28.36	29.45	5,280	13-1/2
														17-1/2
E-3A	5/4/85 5/5/85	1700-0200	250	5,270	9,800	9.7	4.9	13.60	73.51	8.01	29.13	28.91	5,490	26-1/2
														35-1/2
E-3A High N2	5/6/85	0100-	250 0700	5,270	10,000	9.8	4.9	12.65	68.38	7.04	26.35	27.69	4,740	58-1/2
E-3A	5/6/85 5/7/85	2100-0900	250	5,270	10,100	9.6	4.9	13.00	70.27	7.70	28.59	26.70	5,145	64-1/2
														78-1/2
E-3B	5/8/85	0000-1100	225	5,270	11,300	9.1	5.0	9.30	37.58	5.69	23.14	24.77	3,720	90-1/2
														105-1/2
E-3C	5/9/85	0000-2400	250	5,270	10,500	9.6	5.0	12.50	67.57	7.57	28.64	25.80	4,980	116-1/2
														129-1/2
E-3C	5/10/85	0000-2400	250	5,270	10,700	9.6	5.0	12.25	66.22	7.55	27.71	25.50	4,775	153-1/2
														153-1/2
E-3C	5/11/85	0000-2400	250	5,270	11,400	9.6	5.0	11.95	64.60	7.35	29.64	24.10	4,765	177-1/2
														177-1/2
E-3C	5/12/85	0000-2400	250	5,270	11,800	9.6	5.0	11.70	63.24	7.20	30.25	23.54	4,700	201-1/2
														201-1/2
E-3C	5/14/85	0000-0700	250	5,270	10,600	9.6	4.9	11.80	63.78	7.13	26.86	25.40	4,670	225-1/2
														249-1/2
E-3C	5/15/85	0000-2400	250	5,270	10,300	9.4	4.9	11.65	62.97	7.10	26.66	25.48	4,635	256-1/2
														273-1/2
E-3C	5/16/85	0000-2400	250	5,270	10,500	9.5	4.9	11.75	63.51	7.17	26.92	25.27	4,660	297-1/2
														297-1/2
E-3C	5/17/85	0000-2400	250	5,270	10,700	9.5	4.9	11.70	63.24	7.12	26.83	25.09	4,600	321-1/2
														321-1/2
														345-1/2

TABLE VI-3

LAPORTE LPMEOH PDU

AVERAGE DATA SUMMARY FOR RUN E-3

(continued)

Run No.	Date	Time	Temp. °C	Press. kPa	Space Vel. L/Hr-Kg	VG,S cm/s	VL,S cm/s	CO Conver. %	Appr. To Equil. %	MeOH in React. Effl. %	MeOH Prod. G-Mol/ Hr-Kg	Slurry Concen. wt% Oxide	Net MeOH Kg/D	Hrs. On Syngas
E-3C	5/18/85	0000-1600	250	5,270	10,700	9.5	4.9	11.60	62.70	7.04	26.51	25.20	4,545	345-1/2 361-1/2
E-3C	5/19/85	0000-2400	250	5,270	10,600	9.5	4.9	11.70	63.24	7.03	26.85	25.08	4,605	369-1/2 393-1/2
E-3C	5/20/85	1100-2400	250	5,270	10,400	9.5	4.9	11.55	62.43	7.16	26.75	25.20	4,650	404-1/2 417-1/2
E-3C	5/21/85	0000-2400	250	5,270	10,700	9.5	4.9	11.35	61.35	6.89	26.32	24.71	4,450	417-1/2 441-1/2
E-3C	5/22/85	0000-2400	250	5,270	10,700	9.5	4.9	11.30	61.08	6.94	26.60	24.80	4,520	441-1/2 465-1/2
E-3C	5/23/85	0000-2400	250	5,270	10,400	9.5	4.9	11.30	61.08	6.94	25.96	25.12	4,510	465-1/2 489-1/2
E-3C	5/24/85	0000-2400	250	5,270	10,500	9.5	4.9	11.20	60.54	6.93	26.24	25.14	4,540	489-1/2 513-1/2
E-3C	5/25/85	0000-2400	250	5,270	10,400	9.4	4.9	11.30	61.08	6.90	25.68	25.18	4,445	513-1/2 537-1/2
E-3C	5/26/85	0000-2400	250	5,270	10,500	9.5	4.9	11.15	60.27	6.90	26.32	25.18	4,555	537-1/2 561-1/2
E-3C	5/27/85	0000-2400	250	5,270	10,400	9.5	4.9	11.15	60.27	6.86	25.37	25.19	4,410	561-1/2 585-1/2
E-3C	5/28/85	0000-2400	250	5,270	10,500	9.5	4.9	11.15	60.27	6.85	25.48	25.23	4,410	585-1/2 609-1/2
E-3C	5/29/85	0000-1100	250	5,270	10,500	9.5	4.9	11.10	60.00	6.82	25.36	25.16	4,390	609-1/2 620-1/2

TABLE VI-3

LAPORTE LPMEOH PDU
AVERAGE DATA SUMMARY FOR RUN E-3
(continued)

Run No.	Date	Time	Temp. °C	Press. kPa	Space Vel. L/Hr-Kg	VG,S cm/s	VL,S cm/s	CO Conver. %	Appr. To Equil. %	MeOH in React. Effl. %	MeOH Prod. G-Mol/ Hr-Kg	Slurry Concen. wt% Oxide	Net MeOH Kg/D	Hrs. On Syngas
E-3C	5/30/85	0000-2400	250	5,270	10,400	9.5	4.9	10.90	58.92	6.74	25.03	25.20	4,350	633-1/2 657-1/2
E-3C	5/31/85	0000-2400	250	5,270	10,500	9.5	4.9	10.80	58.38	6.68	24.98	25.12	4,285	657-1/2 681-1/2
E-3C*	6/3/85	1200-2400	250	5,270	10,600	9.5	5.0	11.05	59.73	6.78	25.70	24.64	4,370	707-1/2 719-1/2
E-3C	6/4/85	0000-2400	250	5,270	10,700	9.5	5.0	10.85	58.65	6.70	25.76	24.67	4,360	719-1/2 743-1/2
E-3C	6/5/85	0000-2400	250	5,270	10,600	9.5	5.0	10.70	57.84	6.64	25.31	24.78	4,300	743-1/2 767-1/2
E-3C	6/6/85	0000-2400	250	5,270	10,600	9.5	5.0	10.70	57.84	6.62	25.56	24.80	4,345	767-1/2 791-1/2
E-3C	6/7/85	0000-2400	250	5,270	10,700	9.5	5.0	10.70	57.84	6.59	25.68	24.71	4,345	791-1/2 815-1/2
E-3C	6/8/85	0000-2400	250	5,270	10,700	9.5	5.0	10.55	57.03	6.58	25.69	24.63	4,345	815-1/2 839-1/2
E-3C	6/9/85	0000-2400	250	5,270	10,700	9.5	5.0	10.65	57.57	6.56	25.45	24.65	4,305	839-1/2 863-1/2
E-3C	6/10/85	0000-2400	250	5,270	10,600	9.5	5.0	10.65	57.57	6.58	25.11	24.75	4,270	863-1/2 887-1/2
E-3C	6/11/85	0000-2400	250	5,270	10,700	9.5	5.0	10.50	56.76	6.50	25.12	24.76	4,250	887-1/2 911-1/2
E-3C	6/12/85	0000-2400	250	5,270	10,700	9.5	5.0	10.40	56.22	6.40	24.71	24.74	4,180	911-1/2 935-1/2
E-3C	6/13/85	0000-1200	250	5,270	10,800	9.5	6.0	10.50	56.76	6.35	25.46	24.68	4,290	935-1/2 947-1/2

*First balance period after compressor outage.

1544S-W2

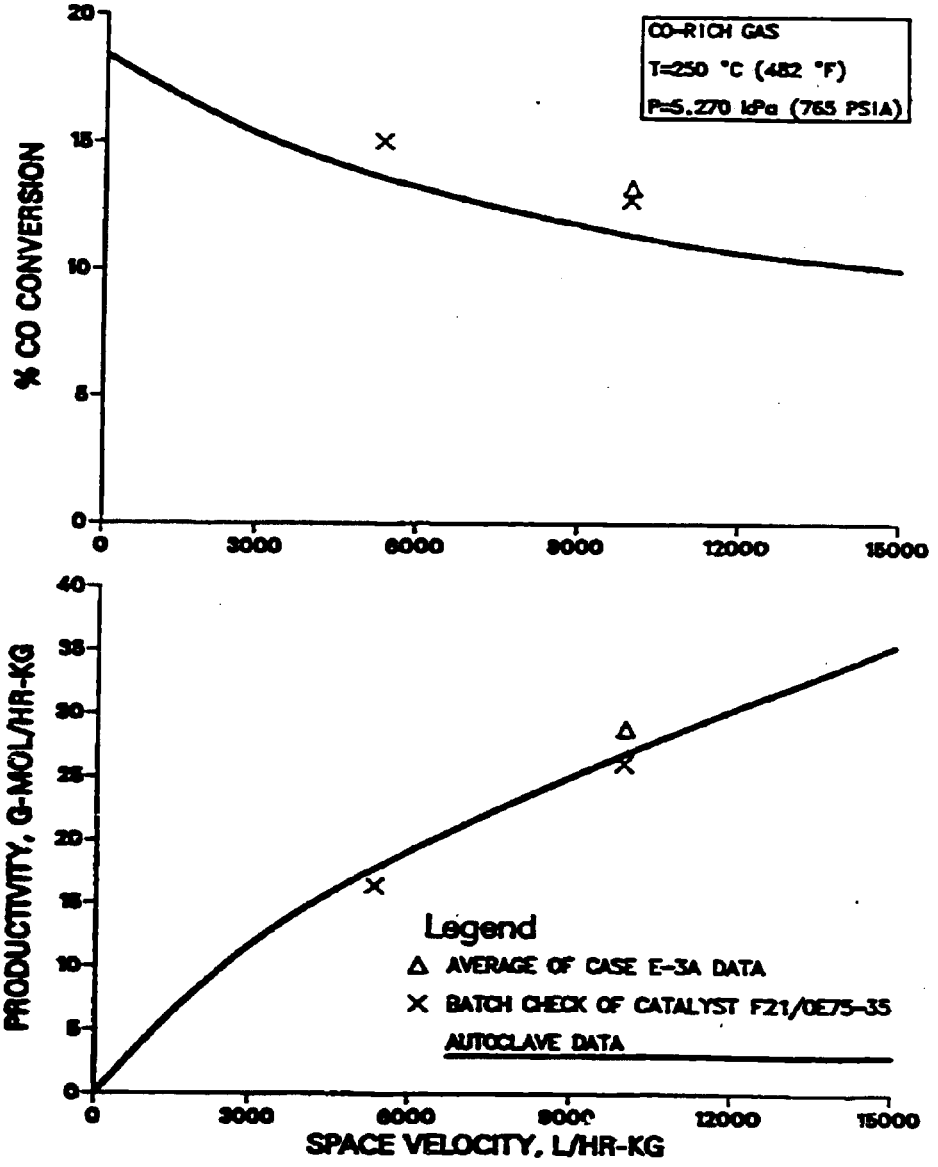

Figure VI-1. LaPorte LPMEOH PDU Laboratory and PDU Performance Comparison

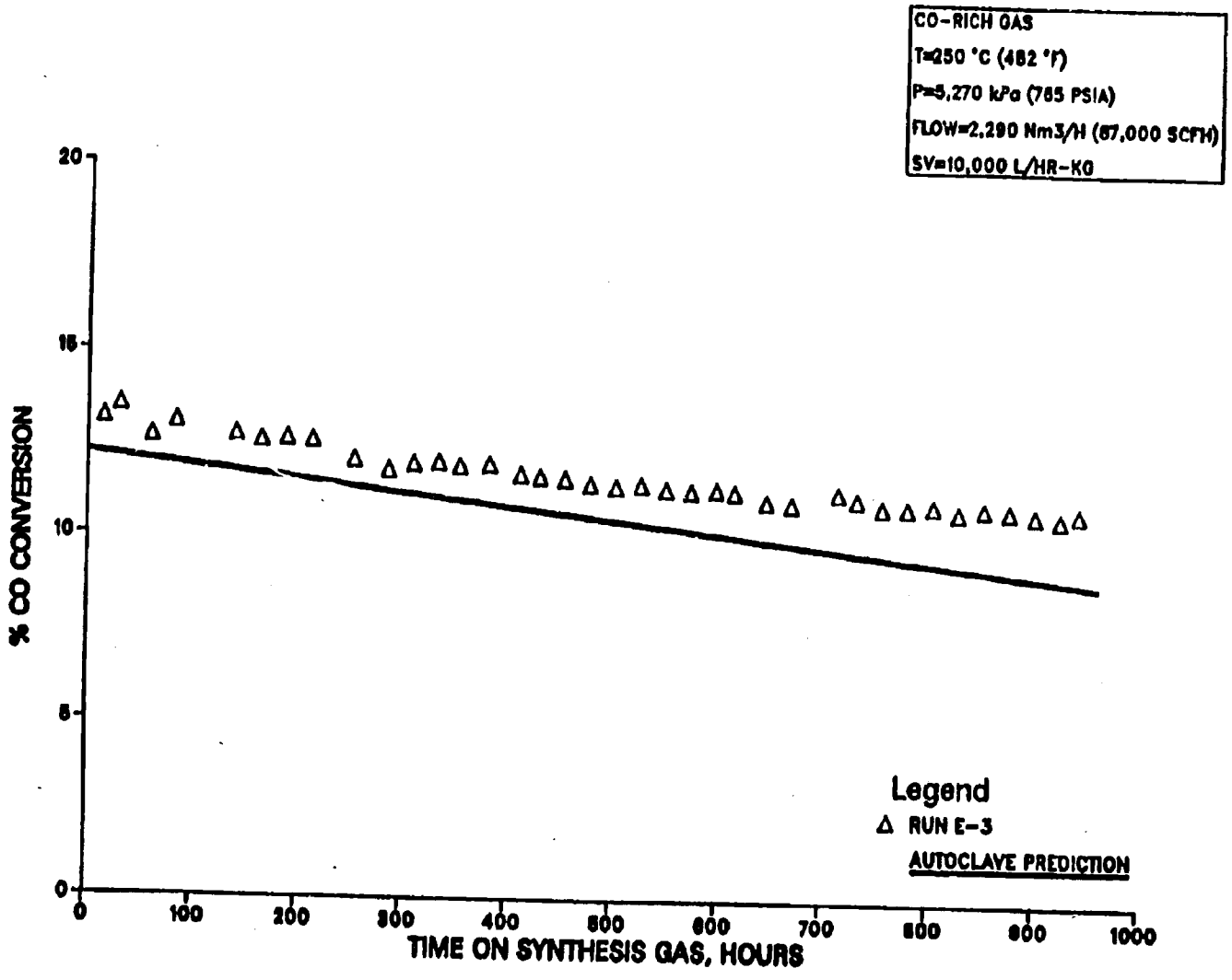

Figure VI-2. LaPorte LPMEOH PDU % CO Conversion vs.Time Normalized Data

Methanol productivity as a function time under synthesis gas is presented in Figure VI-3. The productivity data have also been normalized to 10,000 l/hr-day and the autoclave prediction line presented for comparison. The methanol productivity data indicated a 0.39% per day decline in catalyst activity. It was noted that the data for the first several days exhibited a higher activity that did not quite fit the linear decline of activity observed for the rest of the run. When these hyperactivity points were excluded, a 0.28% per day decline in productivity was obtained over the 40 days of operation. This was a significant improvement in activity maintenance over the 1.1% per day decline observed in Run E-1. Methanol productivities at the beginning of the run were approximately 28.3 g-mol/kg-hr and declined to approximately 24.7 g-mol/kg-hr at the end of the run. After the PDU was restarted following the compressor motor outage/replacement (occurred at 687 hour on synthesis gas), the methanol productivity returned to a level comparable to the performance before the outage and confirmed the suitability of a nitrogen purge containing a low H_2 content for maintaining catalyst activity during extended downtime.

Various factors can be responsible for catalyst activity decline. The exact mechanism of deactivation is not well understood, although temperature is known to be a very imp

roduction rates (~250°C). Growth of the copper and zinc crystallite size with resulting loss in metal surface area is the usual result of excessive heat at the catalyst surface. In addition, the copper-based catalysts are particularly susceptible to chemical poisons. Sulphur, halogens, and trace metals such as iron and nickel, cause rapid activity decline, even when present at the ppm level. The exact effect of these species on the active catalyst surface is complex, although the measurements we make on catalyst samples can be used to determine catalyst health.

As shown in Figure VI-3, the LaPorte PDU Run E-3 data exhibit activity maintenance performance at least equivalent to that of the autoclave, based on time-on-stream. In Figure VI-4, the history based on cumulative methanol production is depicted from Run E-3, autoclave, and from the earlier PDU activity maintenance Run E-1. The deactivation rate for E-1 was approximately four times that seen in the autoclave, while that for Run E-3 approached closely that of the autoclave. The excellent agreement between Run E-3 and the autoclave data indicates that the efficient heat removal and uniform temperatures experienced in the stirred autoclave reactors are being demonstrated equally in the LaPort LPMEOH PDU. This is a key result. The more rapid activity decline seen in Run E-1 is thought to be the result of chemical poisoning. Correlation of methanol productivity with the amount of metals (Ni plus Fe) found on the catalyst has strongly suggested this to be the case (Ref. 2, Ref. 5). The metallurgical upgrade and chemical cleaning carried out before Run E-3 are thought to be the principal reason for the much improved performance level.

Effluent methanol concentrations over time are displayed in Figure VI-5, and the results of the liquid product analyses are listed in Table VI-4. A high methanol selectivity was achieved using a CO-rich feed. The methanol concentration in the liquid product was approximately 96 wt% throughout the run. Methanol concentrations in the reactor effluent showed a slight downward trend resulting from the activity decline of the catalyst. The water concentration in the effluent increased slightly as the run proceeded, also probably due to the activity decline of the catalyst. The data for Case E-3B (the lower temperature case, 225°C (437°F)) and the comparable autoclave data results are summarized in Table VI-5. Note that the run results exceed the laboratory data; however, the difference between the run data and the laboratory data is probably not significant since the database is limited for these conditions. Further testing at a reactor temperature of 225°C (437°F) will be necessary to determine the performance of the PDU

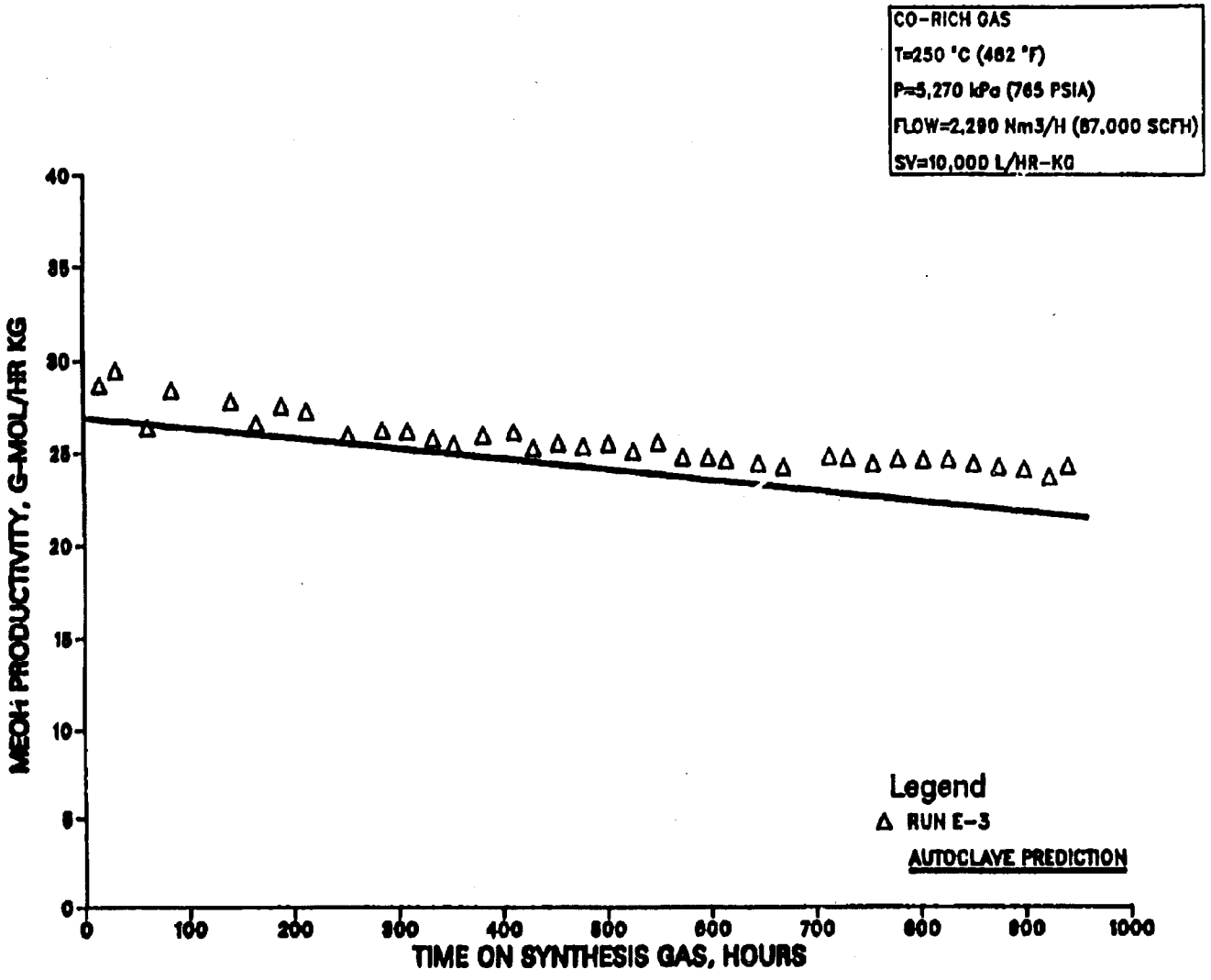

Figure VI-3. LaPorte LPMEOH PDU MeOH Productivity vs.Time
Normalized Data

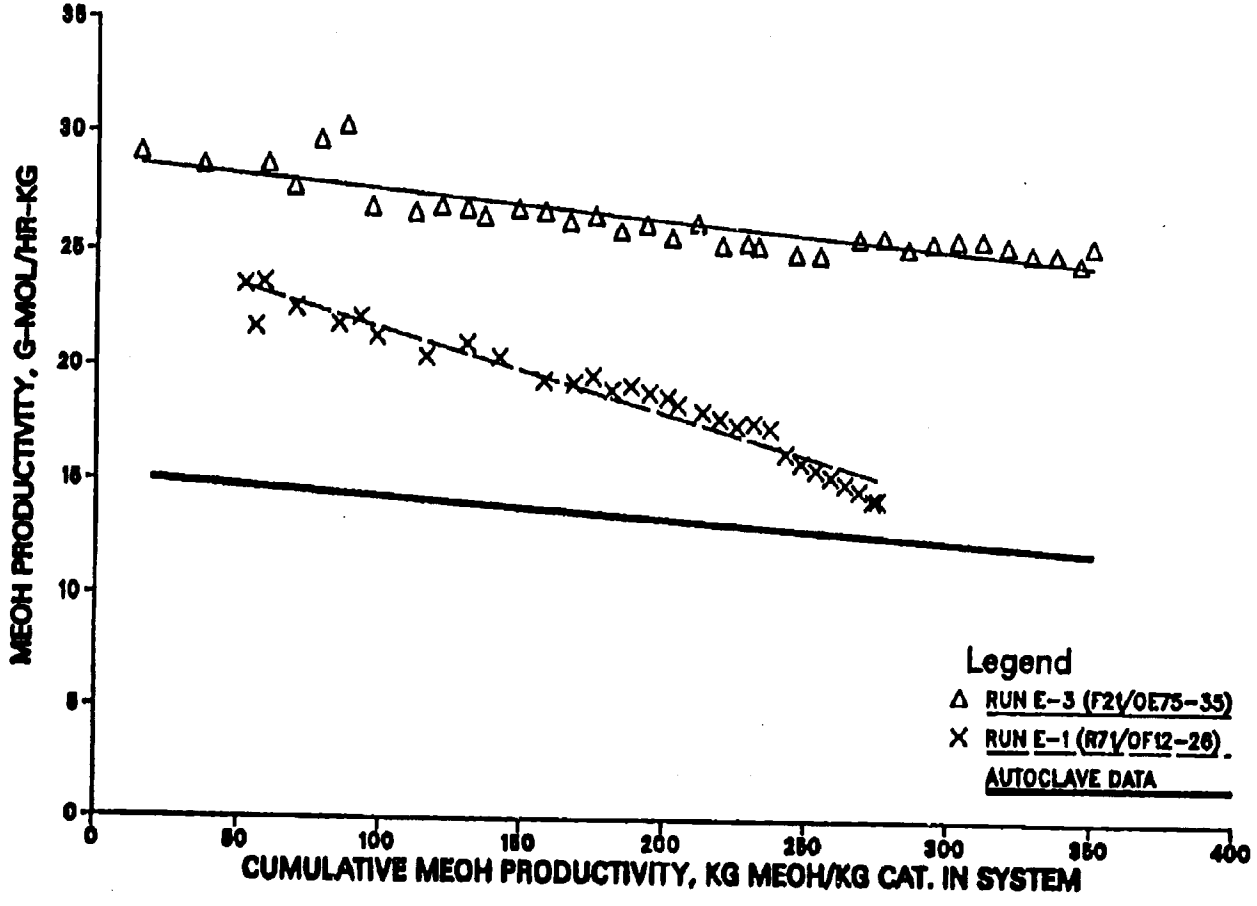

Figure VI-4. LaPorte LPMEOH PDU MeOH Productivity vs. Cumulative MeOH Productivity

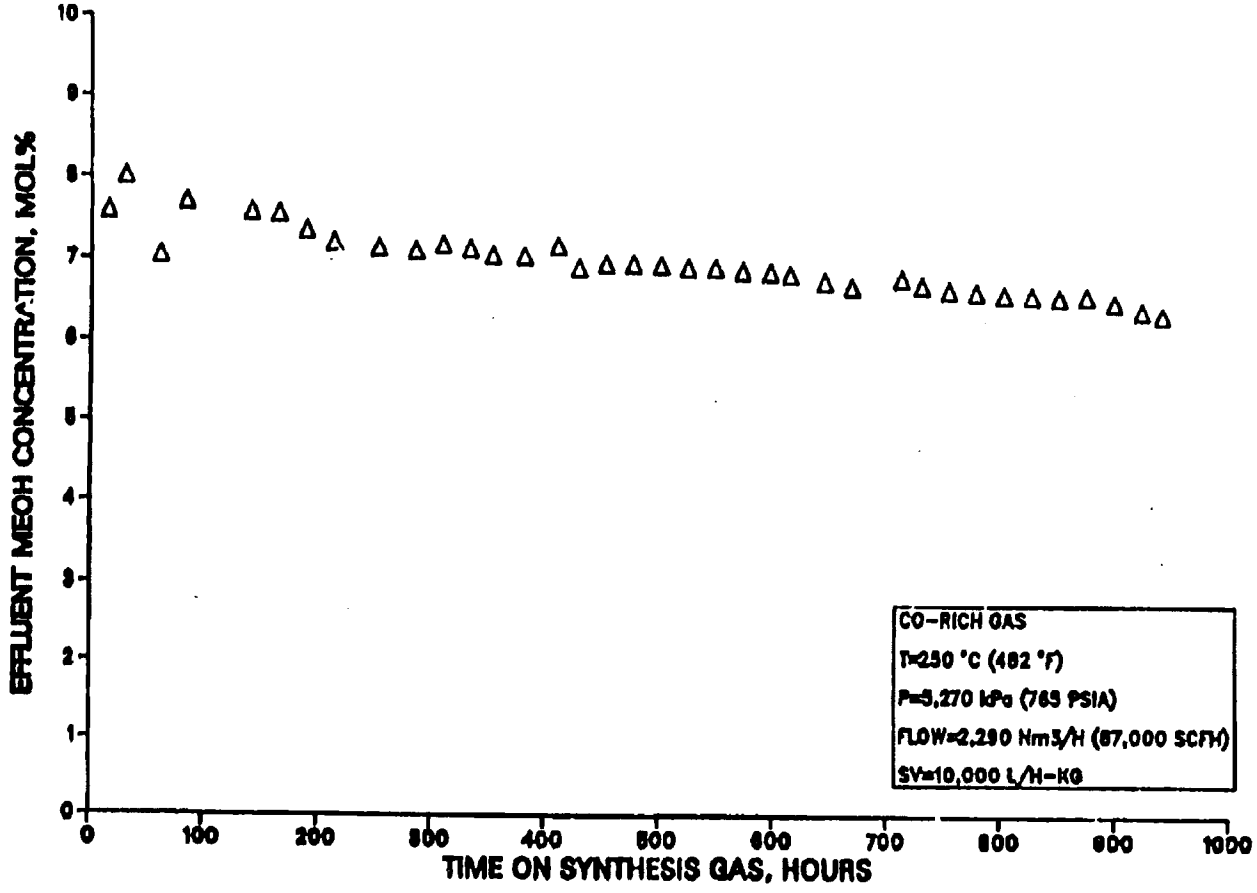

Figure VI-5. LaPorte LPMEOH PDU MeOH Concentration vs. Time

TABLE VI-4

LAPORTE LPMEOH PDU
LIQUID PRODUCT ANALYSES FOR RUN E-3

Date	MeOH wt%	C_2H_5OH wt%	C_3H_7OH wt%	C_4H_9OH wt%	$C_5H_{11}OH$ wt%	Esters wt%	Oil wt%	H_2O wt%	Total
5/6	95.33	0.95	0.30	0.31	0.10	1.02	1.30	0.69	100%
5/8*	96.80	0.26	0.08	0.07	0.04	0.61	1.30	0.85	100%
5/10	95.21	0.90	0.30	0.31	0.21	0.96	1.30	0.81	100%
5/12	95.04	0.90	0.29	0.30	0.20	0.91	1.54	0.83	100%
5/14	95.05	0.86	0.29	0.29	0.19	0.87	1.59	0.85	100%
5/16	95.20	0.79	0.27	0.27	0.16	0.91	1.54	0.86	100%
5/18	95.37	0.79	0.27	0.27	0.16	0.85	1.43	0.86	100%
5/20	95.42	0.72	0.26	0.25	0.15	0.87	1.43	0.89	100%
5/22	95.56	0.73	0.27	0.24	0.16	0.81	1.32	0.91	100%
5/24**	95.58	0.76	0.29	0.21	0.15	0.65	1.32	1.05	100%
5/26	95.61	0.75	0.31	0.19	0.14	0.65	1.32	1.05	100%
5/28	95.69	0.71	0.29	0.18	0.13	0.64	1.32	1.04	100%
5/30	95.71	0.69	0.29	0.18	0.13	0.63	1.32	1.06	100%
6/3***	95.73	0.68	0.29	0.17	0.11	0.60	1.37	1.05	100%
6/5	95.72	0.67	0.31	0.17	0.13	0.60	1.31	1.09	100%
6/7	95.75	0.66	0.27	0.17	0.12	0.59	1.34	1.10	100%
6/9	95.82	0.65	0.29	0.17	0.12	0.60	1.25	1.10	100%
6/11	95.79	0.62	0.28	0.19	0.11	0.57	1.31	1.13	100%
6/13	95.71	0.65	0.26	0.19	0.13	0.61	1.34	1.12	100%

*Condition E-3B Sample.

**New standard used in chromatograph analyses.

***Restart of PDU after compressor outage.

TABLE VI-5

LAPORTE LPMEOH PDU
CASE E-3B DATA SUMMARY

Reactor Conditions:

Temperature	225°C (437°F)
Pressure	5,270 kPa (765 psia)
Space Velocity	11,300 l/hr-kg
Slurry Concentration	25 wt% oxide based
Superficial Gas Velocity	9.1 cm/s (0.30 ft/s)
Superficial Liquid Velocity	5.0 cm/s (0.16 ft/s)

	Run E-3B Data Summary	Laboratory Data Summary
CO Conversion	9.30%	8.00%
Approach to Equilibrium	37.60%	32.34%
MeOH Productivity	23.14 g-mol/hr-kg	19.40 g-mol/hr-kg
Effluent MeOH Concentration	5.69 mol%	

compared to laboratory data. These data will be used to develop a database for temperature programming. This technique, which is used in commercial-scale reactors, holds productivity constant by increasing the reactor temperature over time, thereby balancing thermodynamic and kinetic (catalyst deactivation) effects. Hydrodynamic data from the nuclear density gauges was gathered throughout the run. These results are presented in the Supplementary Volume of this report.

Slurry samples were taken at various times throughout the 40-day operation. The samples were filtered in a nitrogen atmosphere and carefully washed with hydrohexane, dried and subjected to various physical and chemical analyses. The purpose of these analyses was to learn more about changes in catalyst properties as methanol synthesis proceeded and identify any catalyst changes that could result in changed performance. Table VI-6 summarizes all the data on catalysts obtained from Run E-3. As has been observed in other experiments, the surface Cu^{+1}/Cu° ratio changes rapidly with time on synthesis gas. The use of CO-Rich gas leads to a significant drop in CU^{+1}/CU° ratio. The ratio was nearly zero within 30-hours on synthesis gas. The surface Cu/Zn ratio also shows a decline with time on synthesis gas. Both trends are consistent with laboratory results. About 700-hours into Run E-3, the LaPorte LPMEOH PDU suffered an unscheduled shutdown. Under nitrogen purge, the preferential solubility of CO_2 is Freezene-100 oil led to a catalyst being exposed to a gas slightly richer in CO_2. A small bleed of hydrogen was introduced to the reactor to offset any harm resulting from this occurrence. A sample soon after startup, E3-59, showed an unexpected increase in CU^{+1}/Cu^{0} ratio. The intial data points on performance after restart were higher than previously. As we have seen before, a relationship between CU^{+1} and activity is suggested. However, more investigations are needed to understand why the Cu^{+1} declines so rapidly during early operation yet maintains stable performance.

TABLE VI-6

LAPORTE LPMEOH PDU
CATALYST ANALYSES FOR RUN E-3

Sample No.	Hours On Gas	ESCA/AUGER Cu^{+1}/Cu^{0}	ESCA/AUGER Cu/Zn	BET m^2/gm	XRD, Å Cu	XRD, Å ZnO	Quantitative Analysis, PPMW Fe	Ni	S	Cl
E3-1	2.00	0.30	0.18	119.50	75.80	59.70	65.00	34.00	<57.00	<100.00
E3-2	12.60			108.80	75.80	61.90				
E3-3	18.25			102.40	81.30	60.80				
E3-4	30.53	<0.10	0.12	102.10	81.30	60.80	61.00	33.00	<57.00	
E3-8	52.50	1.00	0.19		79.40	65.40	79.00	34.00	<57.00	
E3-9	73.00	0.04	0.12	102.60	83.30	61.90	76.40	37.70	69.00	
E3-17	121.92				85.50	67.00				
E3-28	195.25				83.30	68.40				
E3-34	265.93				79.40	64.40	107.00	34.00	26.00	<50.00
E3-39	297.20	0.07	0.08	79.00	85.50	67.00	138.00	37.00	22.00	<50.00
E3-42	406.25			97.30	87.70	67.00	95.90	34.00	130.00	<50.00
E3-45	473.08			85.10	95.20	68.40	77.00	36.00	50.00	<21.00
E3-46	546.00				92.60	67.00	68.00	28.00	93.00	<21.00
E3-52	617.00	<0.40	0.09	45.60	87.70	69.90	70.00	30.00	50.00	<21.00
E3-56*	702.25	<0.10	0.07	76.50	98.00	68.40	76.00	27.00		54.00
E3-59	728.25	0.82	0.07	73.80	87.70	63.10	97.00	28.00		69.00
E3-63	796.50				92.00	72.40	60.10	24.10		<50.00
E3-69	867.90				91.40	69.00	59.30	23.20		<50.00
E3-70	941.50	0.14	0.05	71.40	98.00	69.10	66.80	25.90		<50.00

*First sample after PDU outage.

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