Methyl Chloride Via Oxyhydrochlorination of Methane

Quarterly Technical Progress Report No.13
For the period October-December 1994

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Contractor

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Contract Objective

The purpose of this contract is to develop a process for converting light alkane gases to methyl chloride via oxyhydrochlorination using highly selective, stable catalysts in fixed-bed reactors designed to remove the large amount of heat generated, so as to control the reaction temperature. Further, the objective is to obtain the engineering data base necessary for developing a commercially feasible process and to evaluate the economics of the process.

Task 1.0 Catalyst Definition and Separation Unit Operation Development

Task 2.0 Process Development Unit (PDU) Design Engineering

Task 3.0 PDU Construction and Procurement

Task 4.0 PDU Start-Up

Task 5.0 Evaluation of Packed Bed Technology

Task 6.0 Separation Optimization

Task 7.0 Process Optimization

Task 8.0 Economic Evaluation

Task 9.0 Preliminary Engineering Design Phase II

Task 10.0 Scale-Up Decision and Business Plan to Phase II

Summary of Technical Progress

Significant progress was made in six different technical areas during this quarter. These key highlights are:

- 1) Evaluation of catalyst samples from UCI led to the ordering of the OHC PDU catalyst batch. This catalyst batch arrived, was screened and found to be defective, and was reordered.
- 2) Natural gas containing higher hydrocarbons was used as a methane source. The reactant mixture formed oxygenates at temperatures lower than observed in the past. Burning at such low temperatures seems to create a product stream containing very little CH_2Cl_2 .
- 3) Although it has not been decided if the PDU will use natural gas from the plant or methane or natural gas from cylinders as a methane feed source, it was concluded that an adsorption unit to remove sulfur and higher hydrocarbons is not necessary at this time.
- 4) PDU construction was completed in December. The bulk of insulation work was completed at the end of November. Much effort has been put into pressure testing the PDU's systems. The startup team has become adept at finding and correcting such leaks.
- 5) SOP writing for the PDU was completed this quarter with communication with the software programmer to insure agreement between the software and SOP.

Technical Results

Task 1.0 Catalyst Definition and Separation Unit Operation Development.

Catalyst Definition

Catalyst research this quarter concentrated on two main issues: 1) Evaluating catalysts made by United Catalyst Inc. (UCI) and ordering the OHC PDU catalyst batch and 2) Determining the effects of using natural gas containing higher hydrocarbons as a methane feed source.

Data Set 1 shows a screening run for UCI's catalyst 2524. The results of this run show poor selectivities on a catalyst that appears to not be very active. However, a very strange phenomenon is noticed. Towards the beginning of the run, at an oven set point of 290C, negative CH_4 and HCl conversions are measured. This, not being a common occurrence, prompted us to run a calibration run which showed that the GC method was slightly off and needed to be corrected. Therefore, it was concluded that the data in Data Set 1 was invalid.

Data Set 2 shows a subsequent screening run of catalyst 2524. CH_4 conversion/MeCl selectivity of 6%/86% at 295C and 9%/76% at 300C is near what other UCI catalysts previously screened

exhibited. Next, the HCl flow was increased to the reactor to determine what effect this might produce. The increased HCl caused more reaction to take place producing a temperature spike that was followed by a gradual increase in temperature as measured by a thermocouple attached to the top of the catalyst bed. At this point, the selectivity of the reaction falls off due to the formation of oxygenates.

Data Set 3 shows a retesting of catalyst 2526, the catalyst formulation chosen for the OHC PDU catalyst batch. The results were not good, but acceptable, with a CH₄ conversion/MeCl selectivity of 7%/83% at 295C and 10%/74% at 300C. Note that these results are slightly below those reported in Quarterly Technical Progress Report No. 12. A pleasant surprise was observed when the HCl flows were increased at 300C. CH₄ conversion stayed constant at 10%, but MeCl selectivity increased to 81%. This is obviously a point to explore in future runs.

Data Set 4 is our attempt to investigate the effect of using natural gas as our CH₄ feed source. The natural gas contained approximately 2% ethane and 0.2% propane. Even at oven set points as low as 270C, the reaction was already burning. This is assumed to be due to the increased reactivity of the higher hydrocarbons. A possible advantage of this system, however, must be pointed out. With this reaction burning at such low temperatures, CH₂Cl₂ was either not being produced or was being burned at a much faster rate than the MeCl. Future experiments will track selectivities while lowering the oven set until the reaction is no longer burning.

Data Set 5 presents results of the lab screening of the OHC PDU catalyst batch that arrived from UCI. The results are obviously very poor. CH₄ conversions of only 5% were reached at 290C before the reaction began burning at 300C. This catalyst, based upon catalyst 2526 previously screened, should have performed better than was observed. Follow up analytical work discovered the problem. As stated earlier, the PDU catalyst was based on catalyst 2526 and therefore should have had a surface area of approximately 200m²/g. However, the surface area of this PDU catalyst was only 70m²/g. The only change made between catalyst 2526 and the PDU catalyst was a substitution of mixed LnCl₃ (containing 66% LaCl₃, 1% CeCl₃, 9% PrCl₃, and 24% NdCl₃) for LaCl₃. This substitution necessitates that the La spec of the catalyst be lowered approximately by one third to keep the overall metal loadings constant. This was not done. UCI left the La spec the same and therefore loaded about 25% LnCl₃ when only 17% should have been used. This overloaded the catalyst, clogged the pores with metals, and killed the catalyst selectivity. This was discussed with UCI, and a new OHC PDU catalyst batch is currently being produced.

Next quarter, two main catalyst objectives will be pursued. First, the new OHC PDU catalyst batch from UCI will be thoroughly examined when it arrives on site. This will not only involve lab screening, but also BET and DCP analyses. Second, a newly synthesized group of catalysts using spherical alumina supports from Calsicat will be screened. These spherical supports will pack nicely into the PDU reactor, and it was decided that it would be advantageous to examine competition to UCI for producing the OHC catalysts.

Task 2.0 PDU Design Engineering

ENGINEERING

Engineering activities this quarter involved some very limited construction support by the different disciplines.

PROCESS ENGINEERING

Last quarter a mixed gas standard was sent to our FTIR analyzer vendor who analyzed it using the instrument they have built for us. This was done to validate the instrument's performance. Results were received this quarter after some expediting. The test results showed problems, though, and it was decided to have the vendor do some more tests. A new standard was ordered by Dow Corning and shipped to the vendor at the end of the quarter.

Issues continued around the methane feed supply. Two main options were investigated this quarter. The first was to tie-in to the existing plant's natural gas header. Two existing idle transfer lines were identified by the team. These were inspected by the plant's Non-Destructive Testing group. Pipe thickness tests showed no thickness less than the manufacturing tolerances for the pipes. However, it was found that 75% of the welds inspected on both lines were unacceptable according to B31.3 codes due to lack of penetration on the welds. Therefore, at the end of December, we requested a bid from a local contractor to install a new transfer line (approximately 600 ft of piping). We also investigated the option of using liquid methane and natural gas in cylinders. Requests for quotations were sent to several vendors at the end of the quarter to investigate the feasibility of this option.

After further investigation, it was decided that if we used natural gas from the plant's header that we would not initially install an adsorption unit. If the header is used, tie-in valves will be provided to facilitate the installation of a unit later. This decision, not to initially add an adsorption unit, was based on the following reasons: 1) The concentration of hydrocarbons greater than C_3 (~ 0.2%) should not be large enough to significantly alter the accuracy of the FTIR analyzer results, 2) the concentration of sulfur (200 ppb) is below what is believed to be an acceptable maximum limit of 5 ppm according to information published in a patent for a oxychlorination process that is similar to ours, and 3) the fabrication and installation time for adding a unit should be short enough that it can be easily added in the future if it is deemed necessary.

Due to environmental concerns, an investigation into the concentration of chlorocarbons in our aqueous waste streams was made. Previous estimates of this waste stream used data from very "rough" tests. This data yielded results that were very high and it was decided that a more rigorous analysis of the system was necessary. Therefore, simulations of the stream's concentration were made using the Aspen PlusTM process simulator. The results show that our

waste water should be well below the plant's discharge permit limits and therefore no additional treatment will be required.

Programming of our CAMILE data acquisition and control system was completed this quarter. This work included proofreading sequences, writing shutdown and emergency stop sequences, modifying other sequences, and writing Q&A pages. Our programmer worked with the SOP writer to insure consistency between the code and the SOP.

Task 3.0 PDU Construction and Procurement

CONSTRUCTION

Construction was completed this quarter during the month of December. Insulation work was also completed this quarter with the bulk of it being finished at the end of November.

Mechanically, punchlist work was one of the main activities for the quarter as well as pressure testing. A small amount of flushing was done to the last remaining system that needed to be flushed. Several small changes were completed as well as some scope items.

There was a slow down in progress (at least two weeks) in the field due to the mechanical contractor pulling several of his men off of the job and moving them to another project in the plant that was deemed to be of higher priority by the plant management. Morale problems were also experienced in the field due to some conflict over weld inspections. One pipefitter quit. This incident also impacted the progress in the field.

After the mechanical contractor finished their work, the plant's shop personnel were used to finish us some remaining loose ends.

Instrumentation and electrical work was completed this quarter. The majority of this work had been completed during the last quarter.

BUDGET

The cost forecast for the project at the end of the quarter was \$2.99 MM. This new forecast put us @ 120% of the capital authorization request (CAR). Items that continued to contribute to the overall project spending increase included the following: additional engineering support due to construction schedule stretch, money to cover punchlist and SAT review fix-up item, field change work, increases in mechanical costs due to schedule stretch, shop support for equipment and instrument checkouts. A complete budget analysis will be presented in next quarter's report.

SCHEDULE

See the attached commissioning plan (Figure 1). The startup pace will be set by what is determined to be a safe working rate. Note that the attached plan shows actual progress up through the end of January, 1995.

Task 4.0 PDU Start-up

STARTUP

Training: The team attended a refresher course on lockout/tagout procedures. Familiarization training for the Camile control system was also conducted this quarter. Process overview sessions (class and tour of the unit) were provided to all shifts for the site safety supervisors, Safety and Loss Prevention (S&LP) technicians, Area 3 production unit technicians (our pilot plant is associated with Area 3), and also to the operating engineers of another pilot plant which will be operating in the same area and control room where our unit is located. Overview sessions were also provided to the shop maintenance personnel. Other training this quarter included; a designed experimentation course for the entire startup team, a short pump seminar, lockout/tagout training followed by a refresher course later on, blood borne pathogens awareness training, a corrosion/metals seminar, fire extinguisher training and Hazard Communication 3 training. Also, each of the startup team members spent two days each training with Area 3 technicians and engineers. This training helped to familiarize us with Area 3 operations and also to build a rapport with their technicians. This will be important for our unit as we will be relying on them for technical support in the future.

Temporary operating procedure (TOP) writing to support startup activities was completed this quarter. The TOP's were written to map out the operational steps required to startup each system and develop efficient systematic tests to verify the proper sizing and operation of the equipment and instruments. Determining this early on will help to avoid operational problems and also avoid delays during the startup.

Standard operating procedure (SOP) writing was completed at the end of this quarter. The SOP writer collaborated with the software programmer to insure consistency between the SOP and the software. Several technical operating issues were discovered and addressed during the course of the writing. The SOP was reviewed by a safety committee and only a few minor recommendations were made. After changes from the startup have been made, we will again have the safety committee review and approve our SOP for operations.

Instrument Checkouts: Instrument checkouts were completed this quarter. This work involved loop continuity checks and checkouts of the emergency shutdown system.

Pressure Testing: A considerable amount of time was spent doing operational pressure testing of the systems. The mechanical contractor had already pressure tested each system, however, their tests were not sensitive enough to detect the leaks that we found during our "operational" checks. The biggest problem that we had was making the glass lined vessel flange connections hold pressure. This work will be continued next quarter. Altogether, at least three weeks in addition to the plan were spent pressure testing.

Mechanical Issues (Gaskets): After many hours of work, milled Teflon CRT envelope gaskets were found to hold pressure on the glass lined equipment. All eight of the of the phase separator heads' had new gaskets installed. It was also found that the head gasket was bad on our 24" dia. vertical glass lined tank. It was replaced during the holiday break.

Electrical & Instrumentation issues:

MicroMotion Mass Meters: Wet calibration tests were completed and showed that the meters are working well.

Tylan Mass Meters: Wiring problems were uncovered in the I/O interface cards. There was also a problem with some of the flow control valves on the meters.

Syltherm 800 Temperature Controller: The controller was found to have been configured improperly due to a mistake made during engineering. It was shipped to the manufacturer, fixed, re-installed and checked out OK.

Control Valves: Several control valves were troubleshooted and corrected. One pressure control valve was troubleshooted and repaired by the instrument shop.

Level Probes: One probe was found to leak nitrogen through the instrument's electronic housing. It was removed and returned to the vendor for repair. This prevented us from loading Multitherm into the system. Work was also done this month to "wet set" some of the level switches.

Shift Work: Shifts have been manned with two men crews of engineers. Shift work was started in December where we worked 12 hour shifts with a four day on and two day off rotation pattern (nominally 60 hrs/wk). This schedule was found to be very taxing. With the experimental plan permitting, we are going to try working in three week campaigns followed by a few days off to recuperate.

Miscellaneous Startup Activities: Shift work was sustained throughout the month of December in spite of several obstacles. Some of the major accomplishments made this month were learning how to get around the plant at night (e.g. finding how and where to obtain supplies, spare parts, etc.), learning how to get things done by the service crew and the shops, and making the control room a reasonable place to occupy.

Pressure testing was the main activity, though, and several leaks were found. The men have learned how to be very handy in fixing the leaks. However, we have had the shops tighten all of the glass lined connections. In general, the men have been spending the evening's finding

the leaks then working with the shops during the day to get them fixed and also to have punchlist work completed.

The Syltherm XLT and the Syltherm 800 systems have been made to hold pressure, and we are very close to having the other systems pass their pressure tests. Both fluids have been loaded into their expansion tanks. The Syltherm XLT pump has been started-up and ran for an extended period of time.

Other activities completed this quarter included:

- Line labeling and instrument tagging.
- Service water system commissioned. A checklist of drain points was made and some potential freeze points were identified and corrected.
- Our double diaphragm piston pump, used to recirculate our solvent, was troubleshooted and brought on-line. Further checks will be required once Multitherm is loaded into the system.
- The pressure control valve was tuned for the absorber/reactor system.
- Miscellaneous TOP and SOP wrap-up work continued during shift work as time permitted.

SAFETY

There were no safety incidents for the project this quarter.

A final walk-through of the safety audit team was made this quarter. Only a few recommendations were made which have now been resolved. Approvals for startup have been obtained from our Safety Audit Team chairman for individual process systems. These approvals have been obtained after the construction on a particular system has been complete. A written request is made detailing the startup commissioning activities that will be performed. This system has worked very well for us.

A two gallon spill of Syltherm XLT was reported this quarter. This incident occurred when an expansion tank was over-loaded with fluid which resulted in the material spilling out of the vent line. An investigation found that the primary cause of the incident was that a packing stop was left in the vessel's level gauge preventing the float from rising. We subsequently investigated all of the other level gauges and found that two other gauges still had the packing stops installed. This situation has been corrected.

A safety checklist that required several items to be located (e.g. fire extinguishers, monitor nozzle, deluge pull station, etc.) was completed by each of the startup team members.

FILE: "OHCS\UDOBESUSCH.WQ1"

OHC STARTUP COMMISSIONING PLAN

***** = Vacation

***** = Vacation	December				January				February				March				April					
ACTIVITY	12/04	12/11	12/18	12/25	01/01	01/08	01/15	01/22	01/29	02/05	02/12	02/19	02/26	03/05	03/12	03/19	03/26	04/02	04/09	04/16	04/23	04/30
SAFETY CHECKS																						
PRESSURE TESTING				*****																		
SYLTHERM 800 COMMISSIONING																						
LEVEL PROBE WET COMMISSIONING																						
SOLVENT RECIRCULATION/HYDRAULICS																						
STRIPPER THERMOCYCLING																						
SYLTHERM XLT SYSTEM																						
ABSORBER/STRIPPER HYDRAULICS																						
FEED SYSTEM CHECKOUT																						
NITROGEN RUNS (Test SOP and Sequencing)																						
PRODUCT RECOVERY SYSTEM (MeCl Rcvry Tests)																						
FTIR STARTUP																						
SYLTHERM 800 CONDITIONING																						
CHEMICAL FEEDS ON																						

Data Appendices

Data Appendix A- Catalyst Synthesis and Composition Information

Data Appendix B- Experimental Data Sets

Data Appendix A

Catalyst Synthesis and Composition Information

CATALYST 555C

SUPPORT	Alumina 149SC, Calsicat
SOURCE	$\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ - lab book 11291 page 70.
LOADING	2.53% Cu, 11.4% La, 0.600% Li.
IMPREGNATION METHOD	Copper source, $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$, and LiCl were dissolved in 100 ml water and added to support for impregnation overnight. Solution was then left under flowing air to dry overnight.
PRETREATMENT	None.
COLOR	Light yellow-green.

CATALYST 555D

SUPPORT	Alumina 149SD, Calsicat
SOURCE	$\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ - lab book 11291 page 70.
LOADING	2.30% Cu, 5.88% La, 0.534% Li.
IMPREGNATION METHOD	Copper source, $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$, and LiCl were dissolved in 100 ml water and added to support for impregnation overnight. Solution was then left under flowing air to dry overnight.
PRETREATMENT	None.
COLOR	Light yellow-green.

CATALYST 555E

SUPPORT	Alumina 149SE, Calsicat
SOURCE	$\text{Cu}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ - lab book 11291 page 70.
LOADING	2.43% Cu, 11.8% La, 0.602% Li.
IMPREGNATION METHOD	Copper source, $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$, and LiCl were dissolved in 100 ml water and added to support for impregnation overnight. Solution was then left under flowing air to dry overnight.
PRETREATMENT	None.
COLOR	Light yellow-green.

Data Appendix B
Experimental Data Sets

HOOD #7 RAMPING EXPERIMENT DEFINITION

=====

OPERATOR NAME: B. CRUM DATE: 14-OCT-94
 LOG BOOK #: 12066 PAGE #: 26
 RUN #: 1206610 CATALYST #: PP2524
 CATALYST NAME:
 COMMENTS: 4th try at catalyst. Starting temp 290

MASS OF CATALYST (g): 4.42
 REACTOR INSIDE DIAM (CM): 0.45
 CATALYST BED HEIGHT (CM): 30.5
 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

=====

INITIAL TEMP (DEG C)	FINAL TEMP (DEG C)	ABSOLUTE RAMP RATE (DEG C / MIN)
BOTTOM: 290	290	1
MIDDLE: 290	290	1
TOP: 290	290	1

=====

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 8

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
15365	0	0	0	0	0	0	0	0	1	79	-1	0	7 290
20540	0	0	0	93	7	0	0	0	1	73	-0	-1	2 290
25699	0	0	0	94	6	0	0	0	1	74	-0	-1	2 290
30858	0	0	0	92	8	0	0	0	1	73	-0	-1	1 291
36017	0	0	0	92	8	0	0	0	1	74	0	1	1 290
41179	0	0	0	92	8	0	0	0	2	72	-1	-3	1 292
47059	0	2	2	88	10	0	0	0	2	73	1	5	5 294
52220	0	1	1	89	10	0	0	0	2	72	1	3	5 296
57389	0	2	2	88	10	0	0	0	2	72	2	6	7 296
62538	0	2	2	89	9	0	0	0	2	71	2	6	6 295
67700	0	2	2	89	10	0	0	0	2	72	2	6	6 295
72858	0	2	2	89	10	0	0	0	3	71	2	7	7 297
78739	0	3	3	83	14	0	0	0	3	69	4	15	16 300
89180	0	3	3	86	11	0	0	0	1	72	4	17	18 300
94340	0	3	3	83	14	0	0	0	1	70	4	15	16 300
99500	0	3	3	85	13	0	0	0	1	66	5	18	18 300
104657	0	2	2	87	11	0	0	0	2	61	5	18	18 301
110540	0	5	5	81	15	0	0	0	2	61	15	54	100 305
115701	0	13	13	70	18	0	0	0	2	40	15	54	100 305
120860	0	16	16	73	11	0	0	0	2	50	15	49	100 306
126020	0	26	26	60	14	0	0	0	2	68	15	44	100 305
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712	
AVG 290	0	0	0	93	7	0	0	0	1	73	-0	-1	1 291
STD	0	0	0	1	1	0	0	0	0	1	0	1	0
AVG 295	0	2	2	89	10	0	0	0	2	72	1	5	6 295
STD	0	0	0	0	0	0	0	0	0	1	0	1	1
AVG 300	0	3	3	85	12	0	0	0	2	67	4	16	17 300
STD	0	0	0	2	1	0	0	0	1	4	0	1	1
AVG 305	0	15	15	71	14	0	0	0	2	55	15	51	100 305
STD	0	8	8	8	2	0	0	0	0	10	0	4	0

RUN 1206610

CATALYST PP2524

RUN 1206610

CATALYST PP2524

Data Set 1 - Page 3

RUN 1206610

CATALYST PP2524

RUN 1206610

CATALYST PP2524

HOOD #7 RAMPING EXPERIMENT DEFINITION

OPERATOR NAME B CRUM DATE 19-OCT-94
 LOG BOOK # 12066 PAGE # 30
 RUN # 1206612 CATALYST # PR 2524
 CATALYST NAME
 COMMENTS NEW CALIBRATION HIGH HCL FLOWS

MASS OF CATALYST (g) 451

REACTOR INSIDE DIAM (CM) 0.45

CATALYST BED HEIGHT (CM) 30.5

TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

```
=====
INITIAL FINAL ABSOLUTE
TEMP TEMP RAMP RATE
(DEG C) (DEG C) (DEG C / MIN)
=====
BOTTOM: 295 295
MIDDLE: 295 295
TOP: 295 295
=====
```

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8

HCL FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 8														
Time	A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ1712	
12785	0	18	18	32	0	0	0	0	1	78	5	15	20	295
17934	0	2	2	37	11	0	0	0	1	68	8	20	18	295
23083	0	2	2	37	11	0	0	0	1	69	6	22	19	295
28244	0	2	2	38	12	0	0	0	1	67	5	19	18	295
33392	0	2	2	38	11	0	0	0	1	65	8	22	20	295
38564	0	3	3	38	12	0	0	0	2	67	6	21	21	298
45354	3	5	8	76	16	0	0	0	2	64	9	34	30	300
50515	3	5	8	76	16	0	0	0	2	83	10	38	38	301
55665	0	6	6	77	17	0	0	0	2	84	9	33	35	300
60826	0	7	7	78	18	0	0	0	2	69	9	32	33	300
65996	3	8	11	71	18	0	0	0	2	52	11	40	43	300 INCREASED HCL
71156	4	9	12	69	18	0	0	0	3	56	12	36	51	300
76374	3	4	7	77	15	2	0	0	3	43	13	38	48	300
81585	18	8	24	57	18	2	0	0	3	55	17	44	100	300
86745	9	10	19	57	24	0	0	0	3	18	18	51	100	300
91864	15	18	33	44	22	1	0	0	3	53	18	44	100	300
97127	10	12	22	62	11	5	0	0	3	28	18	48	100	300
102345	14	14	28	45	21	8	0	0	3	42	18	51	100	300
107564	13	14	27	45	22	8	0	0	4	33	17	41	100	300
112784	10	9	19	63	18	0	0	0	4	20	19	42	100	301 INCREASED HCL
117945	12	10	22	65	13	0	0	0	4	28	20	39	100	300
123165	13	10	23	41	35	0	0	0	4	33	20	49	100	301
128384	12	10	23	41	38	0	0	0	4	33	20	49	100	301
133556	13	10	23	63	14	0	0	0	4	30	20	40	100	301
138704	12	9	21	61	17	0	0	0	4	27	20	42	100	300
AVERAGES A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ1712		
AVG 295	0	2	2	66	11	0	0	1	87	6	21	19	285	
STD	0	0	0	0	0	0	0	0	1	0	1	1		
AVG 300	2	6	7	78	17	0	0	2	65	9	34	33	300	
STD	2	1	1	0	1	0	0	0	2	0	2	2		
AVG 4:18:1	10	11	20	59	19	2	0	3	43	16	44	83	300	
STD	5	4	8	11	4	2	0	0	14	3	5	25		
AVG 4:2:1	12	10	22	58	22	0	0	4	28	20	44	100	300	
STD	1	1	2	11	10	0	0	0	5	0	4	0		

RUN 1206612

CAT PP 2524

RUN 1206612

CAT PP 2524

RUN 1206612

CAT PP 2524

RUN 1206612

CAT PP 2524

HOOD #7 RAMPING EXPERIMENT DEFINITION

OPERATOR NAME: B CRUM DATE: 24-OCT-94
 LOG BOOK #: 12066 PAGE #: 32
 RUN #: 1206613 CATALYST #: PP 2528
 CATALYST NAME:
 COMMENTS: NEW CALIBRATION HIGH HCl FLOWS

MASS OF CATALYST (g): 3.82
 REACTOR INSIDE DIAM (CM): 0.45
 CATALYST BED HEIGHT (CM): 30.5

OHC/JSS

TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

INITIAL FINAL ABSOLUTE
 TEMP TEMP RAMP RATE
 (DEG C) (DEG C) (DEG C / MIN)

BOTTOM: 295 300 1

MIDDLE: 295 300 1

TOP: 295 300 1

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 HCl FLOW RATE (CC/MIN): 29.8 AUX FLOW RATE (CC/MIN): 0

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS) 8														
Time	A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ1712	STOIC
17648														295 4:1:1.3
22805														295
27968	0	4	4	83	14	0	0	1	64	8	28	28	28	295
33127	0	4	4	84	13	0	0	1	64	7	28	28	28	295
38286	0	3	3	84	12	0	0	1	64	7	27	24	24	295
43434	0	4	4	83	14	0	0	2	65	6	24	24	24	298
49278	0	7	7	75	17	2	0	2	62	10	39	40	40	300
54438	0	7	7	74	17	2	0	2	62	10	40	38	38	300
59599	0	7	7	74	17	2	0	2	62	9	34	37	37	300
64807	0	7	7	74	17	2	0	2	62	10	39	40	40	300 4:1:1.6
69979	0	7	7	74	17	2	0	2	61	10	38	41	41	300
75138	0	7	7	76	16	2	0	3	54	10	31	41	41	300
81077	0	3	3	85	11	1	0	3	43	10	29	39	39	300
86308	0	5	5	78	17	2	0	3	61	9	28	35	35	300 4:1:2
91458	3	4	7	76	15	2	0	3	58	9	29	40	40	301
96677	3	4	7	76	15	2	0	3	58	10	30	39	39	300
102197	2	3	5	77	15	2	0	3	51	9	22	35	35	300
113167	0	3	3	77	17	1	2	1	29	21	55	100	100	308
118387	0	17	17	58	26	1	0	1	57	22	52	100	100	308
123548	0	13	13	44	43	0	0	1	48	21	60	100	100	308
128787	0	14	14	72	14	0	0	1	51	21	47	100	100	308
133989	0	14	14	73	13	0	0	1	49	21	46	100	100	308
139207	0	7	7	49	44	0	0	2	8	21	62	100	100	308
146048	0	10	10	38	52	0	0	2	38	21	66	100	100	310
151208	0	10	10	38	53	0	0	2	35	21	68	100	100	310
156368	0	6	6	48	48	0	0	2	35	20	61	100	100	310
161528	0	10	10	38	52	0	0	2	34	20	62	100	100	310
AVERAGES A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ1712		
AVG 395	0	4	4	83	13	0	0	1	64	7	27	25	25	295
STD	0	0	0	1	1	0	0	0	0	0	2	1	1	
AVG 300	0	7	7	74	17	2	0	2	62	10	38	39	39	300
STD	0	0	0	0	0	0	0	0	1	1	2	2	2	
AVG 4:1:1.6	0	5	5	81	14	1	0	3	49	10	30	40	40	300
STD	0	2	2	5	3	0	0	0	6	0	1	1	1	
AVG 4:2:1	2	4	8	78	16	2	0	3	58	9	27	37	37	300
STD	1	1	1	1	1	0	0	0	3	0	3	2	2	
AVG 305	0	7	7	70	17	1	0	2	51	12	36	51	51	308
STD	0	5	5	13	13	0	1	0	18	0	6	0	0	
AVG 310	0	9	9	40	51	0	0	2	35	20	64	100	100	310
STD	0	2	2	4	2	0	0	0	1	1	2	0	0	

RUN 1206613

CAT PP2526

RUN 1206613

CAT PP2526

RUN 1206613

CAT PP2526

RUN 1206613

CAT PP2526

HOOD #7 RAMPING EXPERIMENT DEFINITION

=====

OPERATOR NAME: B. CRUM DATE 12-DEC-94
 LOG BOOK #: 12066 PAGE #: 38
 RUN #: 1206616 CATALYST #: PP2526
 CATALYST NAME:
 COMMENTS: D RTY CH4 LOW TEMP TO KEEP FROM BURNING
 RUN PLUGGED AFTER FIRST INJ AT 285C (1.5 HRS INTO RUN)

MASS OF CATALYST (g): 3.87
 REACTOR INSIDE DIAM (CM): 0.45
 CATALYST BED HEIGHT (CM): 30.5
 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

=====

INITIAL TEMP (DEG C)	FINAL TEMP (DEG C)	ABSOLUTE RAMP RATE (DEG C / MIN)
BOTTOM: 270	270	1
MIDDLE: 270	270	1
TOP: 270	270	1

=====

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

Time	TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 8								MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712	
	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7						
19705	0	82	82	18	0	0	0	0	1	69	11	7	24	270
24880	10	40	50	50	0	0	0	0	1	65	11	18	21	270
30032	9	41	50	50	0	0	0	0	1	69	9	16	18	270
35192	10	42	52	48	0	0	0	0	1	68	9	14	18	271
40360	10	44	54	46	0	0	0	0	1	69	10	16	18	270
45511	10	44	54	46	0	0	0	0	2	69	9	15	18	271
52351	12	37	49	50	1	0	0	0	2	67	11	19	28	280
57571	10	33	43	56	1	0	0	0	2	65	12	24	29	281
62796	10	35	45	53	2	0	0	0	2	159	77	145	29	280
68013	10	35	46	53	1	0	0	0	2	65	12	22	30	280
73232	7	23	30	69	1	0	0	0	2	60	12	28	29	281
78456	11	34	45	53	2	0	0	0	3	62	11	21	28	281
84385	26	22	48	51	1	0	0	0	3	81	20	36	100	285
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712		
AVG	10	42	52	48	0	0	0	1	68	10	16	18	271	
STD	0	2	2	2	0	0	0		1	1	1	1		
	10	33	43	56	1	0	0	2	80	22	43	29	280	
	2	4	6	6	0	0	0		36	24	46	0		

RUN 1206616
CAT PP2526

RUN 1206616

CAT PP2526

RUN 1206616

CAT PP2526

RUN 1206616

CAT PP 2526

Data Set 4 - Page 5

HOOD #7 RAMPING EXPERIMENT DEFINITION

=====

OPERATOR NAME: B. CRUM DATE 22-DEC-94
LOG BOOK #: 12066 PAGE #: 40
RUN #: 1206617 CATALYST #: 11291-71 SIEVED
CATALYST NAME: PDU CATALYST
COMMENTS PDU CATALYST AT 290 300 310

MASS OF CATALYST (g): 4.58
REACTOR INSIDE DIAM (CM): 0.45
CATALYST BED HEIGHT (CM): 30.5

TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

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=====
      INITIAL   FINAL   ABSOLUTE
      TEMP     TEMP     RAMP RATE
      (DEG C)   (DEG C)   (DEG C / MIN)
=====
BOTTOM: 290    290      1
MIDDLE: 290    290      1
TOP : 290     290      1
=====
```

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 8

me	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
15242	0	26	26	74	0	0	0	0	1	199	100	258	29
20413	0	6	6	82	12	0	0	0	1	71	6	19	21
25561	0	5	5	85	10	0	0	0	1	70	6	21	23
30719	0	5	5	86	10	0	0	0	1	70	4	18	20
35879	0	5	5	86	9	0	0	0	1	70	4	18	18
41035	0	5	5	86	9	0	0	0	2	70	5	18	20
47785	27	7	35	53	12	0	0	0	2	72	11	28	46
52943	25	8	33	53	14	0	0	0	2	70	11	30	46
58104	23	9	32	52	16	0	0	0	2	69	11	30	47
63265	24	9	33	52	15	0	0	0	2	70	11	31	46
68414	24	8	33	53	15	0	0	0	2	72	11	30	46
73573	22	9	31	54	15	0	0	0	3	69	11	31	46
80339	10	19	29	52	19	0	0	0	3	66	19	58	100
85499	5	17	23	61	16	0	0	0	3	56	18	56	100
90673	6	23	30	48	20	3	0	0	3	98	19	190	100
94562	0	5	5	88	7	0	0	0	3	84	4	40	39
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712	
AVG 290	0	5	5	85	10	0	0	1	70	5	18	21	290
STD	0	0	0	1	1	0	0	0	0	1	2	1	
AVG 300	24	9	33	53	14	0	0	2	70	11	30	46	300
STD	2	1	1	1	1	0	0	0	1	0	1	1	
AVG 310	7	20	27	54	18	1	0	3	73	19	101	100	310
STD	2	3	3	6	2	1	0	0	18	1	63	0	

NOTE LAST POINT IS AT MUCH HIGHER SPACE TIMES, AND IS NOT INCLUDED IN THE ABOVE DATA

RUM 1206617

CAT 11291-71 SIEVED

—■— CONVERSION OF CH₄

RUM 1206617
CAT 11291-71 SIEVED

RUM 1206617

CAT 11291-71 SIEVED

RUM 1206617

CAT 11291-71 SIEVED