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Methyl Chloride Via Oxyhydrochlorination of Methane

Quarterly Technical Progress Report No. 5
For the period October-December 1992

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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 Miniplant Design Engineering

Task 3.0 Miniplant 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

At a review meeting in Carrollton on October 21, a detailed overview of the proposed PFD for this unit was described to the project management committee as well as other representatives from Dow Corning, PETC, Gas Research Institute, and Texas Gas. As a result of this meeting and subsequent detailed internal Dow Corning reviews, the PDU PFD is currently being expanded into a P&ID with a subcontractor. This work is preliminary to construction and procurement of the PDU. A determination of the final scope of the PDU and the beginning of detail design is targeted for Q1 1993.

The key development this quarter was the near finalization of the PFD for the PDU. This unit will be the cornerstone of this contractual effort. Significant technical hurdles, primarily the definition of the separation unit operation, have been passed in order to reach this point. A new time schedule that will point to a late 1993 startup of this unit is under development at this time.

Technical Results

Task 1. Catalyst Definition and Separation Unit Operation Development.

Catalyst Definition-

Work continues to develop a stable highly selective catalyst for the oxyhydrochlorination of CH_4 . A review of this activity to date with emphasis on progress made this quarter is given in this section.

Experimental Data Set 1 in Appendix A shows a data set obtained from a CuO based catalyst labelled catalyst 163A. Page 1 of this actual laboratory data set shows experimental identifiers as well as selectivity and conversion data. In the data section columns Al6CO, CO₂ COX, M1, M2, M3 and M4 refer to the carbon mole based selectivities to CO, CO₂, CO +₂, CH₃Cl, CH₂Cl₂, CHCl₃, and CCl₄. Columns Q6CH₄C and Q6HCLC refer to the measured CH₄ conversion and the resultant calculated HCl conversion. This will be the general format in which all raw laboratory data will be presented.

Page 2 of Data Set 1 shows the plotted results for CH₄ conversion as a function of experiment time for this data set, followed on the next page by a similar plot of CH₃Cl selectivity. This rather uninspiring data shows a fairly large degree of variability, but the basic results are clear. In order for this catalyst to obtain even 10% CH₄ conversions at the given feed rate, a reactor furnace set temperature of about 330° C was necessary.

One key working hypothesis for our catalyst development work is the premise that we need very tight control of the temperature *inside of the catalytic reactor*. Previous work in our laboratory has shown that if a significant exotherm occurs in the catalyst bed, significant catalyst degradation occurs. This theory was the impetus for us to place a tiny thermocouple inside of our packed bed to monitor the bed temperature and track it independently of the external reactor furnace set point. Data set number 2 shows one of our first attempts at monitoring this temperature profile. The set points for the reactor furnace in this experiment are given in the last column labelled TQI612. In the experiment the external reactor furnace set point was systematically varied. This data shows results for one of our most promising Cu catalysts, labelled 536.

This experiment shows reasonably good temperature control inside of the reactor at temperatures up to 340° C. Note on Page 2 of Data Set 2, the relative temperature independence of the conversion as the temperature ramps from 340° through 352° C. On Page 4 of Data Set 2, the temperature profiles are graphed, notice that the internal bed temperature is continually rising independently of the temperature ramping rate during this period from 50 to 80 hours into the experiment. The key here is to note that the amount of oxygenated by-products is increasing during this time. Figure 1 shows the results of an activation energy study done earlier in the contract. The data shows that the activation energies for CO and CO₂ formation are roughly twice those for CH₃Cl and CH₂Cl₂ formation. This fact, combined with the 3 to 5 times greater heat of reaction, supports that the observed temperature increase was caused by the increase in the heat of reaction. It was exacerbated by the increase in activation energy for the combustion by-products. This shows the need for adequate heat transfer in the PDU reactor design.

Experimental Data Set 3 shows the first attempt at measuring the useful lifetime for the same catalyst 536. This is a "pseudo"-isothermal experiment in which the external reactor furnace was controlled to 340° C for the entire run. This 183 hour run showed a gradual and continuous reduction in CH₄ conversion from 16-17% to approximately 10%. The selectivity correspondingly rose from 82% to 90%. Note on the temperature traces from page 5 of this data set that the "feeds on" point was not time zero. The 7°C rise in internal bed temperature was caused by the initiation of the reactants. This temperature differential remained quite stable over the 8 day experiment.

Experimental Data Set 4 shows a very different behavior for a high loading CuO catalyst, 163C. In this case the reactant flow and isothermal 340° C set point were identical to those in data set 3. Here another thermocouple was wired to the immediate outside of the reactor tube. Note on Page 4 of this data set, the 120°C exotherm in the catalyst bed over the 340° C set point ! This huge temperature excursion is typical of what high metal loading catalysts will do under these experimental conditions. The resulting data must be viewed with caution, as it is obviously the result of a very unstable system. This experiment was aborted due to catalyst decomposition which caused plugging of the screening experiment.

In an attempt to put our current catalyst development status into perspective Tables 1, 2, and 3 are included. A significant portion of this work was done in Q3 and Q4 of this contract. In these tables numerous experiments are shown with the temperature, space velocity (cm/sec), CH₄ conversion, HCl conversion, O₂ conversion, and selectivities to various species all listed. The form of copper used for catalyst synthesis and the support are also shown. All of this data was obtained at atmospheric pressure. The key conclusion from this data is that there are many copper based catalyst systems that do not give the desired selectivity to CH₃Cl. This is clear from the wide range of selectivities observed over this range of experiments. Details on the various catalyst compositions are given in Data Appendix B.

Figure 2 shows a composite of all of the experiments done to date that have CH₄ conversions in the 15 to 25% range. This is currently our design target for the PDU and is the area that selectivity improvements are being focused on. It is useful to remember that an initial goal of 20% CH₄ conversion and 80% selectivity to CH₃Cl is our initial target. Presenting the data in this fashion allows us to use the graph as a screening tool to evaluate the factors that promote positive increases in CH₃Cl selectivity. Three trends emerge as this is done:

1. There is a one to one correlation between experiments done at the highest laboratory fluid velocities and the points that have good selectivity. That is, every time an experiment has yielded 15<x<25% CH₄ conversion and the fluid velocity has been higher than 8.5 cm/sec, the resulting selectivity has been good

compared with other data.

2. Experiments with very low fluid velocities, as low as 0.1 cm/sec, have given good selectivities as long as the temperature of the experiment is kept very low and the space time high. This is presumably due to the severe reaction rate limitation that occurs with low temperatures combined with severe mass transfer resistances at these very low fluid velocities.

3. Initial pressure studies show the effect of increased pressure. A pressure of 40 psi yields equivalent selectivity and conversion to a low pressure experiment at the same fluid velocity and residence time. The key is that the reaction temperature is lowered by 20°- 30° C.

The outcome of the work to date is that we have several different catalyst's which perform within the same range. Work in Q6 will focus on stability issues and these systems will be characterized by activity loss in 1-3 week stability experiments. In addition the effects of increasing pressure and maintaining the reaction at the absolute minimum temperature will be explored.

Separation Process Definition-

Work that began last quarter on the development of a absorber/stripper unit operation to remove CH_3Cl from the large non-condensable effluent stream of the OHC reactor continued this quarter. Two different efforts are underway. The first is to measure key solvent parameters in a batch mode, the second is to test the absorber column operation on the laboratory scale. Both of these efforts will be reviewed.

The key requirements for a solvent to be a candidate for successful implementation in this technology are:

1. Low Volatility
2. Strong Absorption Characteristics for C1 chlorocarbons
3. Resistance to $\text{HCl}(\text{aq})$
4. Insoluble with H_2O
5. Stable over a temperature range of -20 °C to 180 °C
6. Preferably possess a low viscosity

Our approach to identifying a group of potential solvents follows:

1. Information gathering- a database of over 50 solvents with all of the pertinent physical data has been collected.
2. Procurement of potential solvents- Approximately 20 different solvents that on paper look to have high potential for success have been ordered or received.
3. Conduct CH_3Cl saturation experiments- This work has been and will be done in a batch system with only CH_3Cl as an adsorbate. The CH_3Cl absorbance vs pressure curves are measured. This ongoing work will be summarized.
4. Assess solvents' resistances to acid attack, perform simple phase separation studies and viscosity

measurements- In both open ampule and sealed tube experiments solvents are evaluated by contacting with various strength acid solutions and evaluated for breakdown. These are done at ambient and high temperature for varying lengths of time. Simple assessments of phase separation ability with H_2O are made by measuring time to settle under controlled conditions. Viscosity vs temperature curves are measured.

5. Study key solvents that look to be the most promising in laboratory absorber.

Table 4 shows a summary of the CH_3Cl single component absorption experiments that have been obtained to date. These results show the results from 6 of the potential solvents we are evaluating. The weight % CH_3Cl , experimental temperature, experimental pressure, and the corresponding vapor pressure of CH_3Cl at the experimental temperature. These 14 experiments done over a fairly small temperature range show a range of CH_3Cl absorbance of 11 to 24%. All of these values are within reason for use in a absorber/stripper system. Clearly, the higher the value the smaller the necessary amount of solvent to recover a given amount of CH_3Cl . This was one screening study used to evaluate solvents. These results are shown graphically in Figure 3.

Figure 4 shows viscosity vs temperature curves for several of the solvents evaluated. As is seen in a comparison of the results for solvent A vs those for solvent D and E, a wide range of viscosity correlations were noted. Figure 5 shows an enlarged view of the results for solvents D and E. These solvents show a very little temperature dependence with the viscosity change of only 30 to 40 cP with a drop in temperature from 25 C to -20C.

In addition to the previously referred to acid stability studies, the next work in solvent determination will be evaluation of several solvents using an actual multicomponent reactor effluent. Figure 6 shows a rough schematic of a laboratory based absorber system in which the composition of the gas phase can be measured on-line before and after passing through an absorber column. Q6 effort in this area of the project will be dedicated to the start up of this system.

Task 2.0 Miniplant Design Engineering

Process Description and Definition

Work on finalizing the pre-engineering work for the PDU continued this quarter. By quarter's end a "final" P&ID was sent to Xytel for the purpose of bidding a modular system to build off of our site. Xytel is a potential subcontractor that specializes in the design and construction of modular systems of this scale. The following list summarizes the key pieces of equipment included in the current definition of PDU scope:

Reactor Section-

- OHC Reactor
- Reactor Hot Oil Expansion Tank
- Reactor Hot Oil Knock Out Pot
- Knock Out Cooler
- Reactor Hot Oil Pump
- Reactor Hot Oil Cooler
- Reactor Hot Oil Heater

Reactor Effluent Recovery

- Desuperheater
- Acid Condenser
- Acid Condenser Separator
- Condensed CH_3Cl Pump
- CH_3Cl Absorber Column
- Absorber Bottoms Separator
- HCl Adsorption Column
- Solvent Chiller
- Absorber Feed Pump
- Stripper Feed Pump
- Solvent Storage Tank

Steam Stripping

- Stripper Preheater
- Stripping Column
- Stripping Column Bottom Separator
- Condensate Reboiler
- Overhead Condenser
- Stripping Column Overhead Separator
- Crude CH_3Cl Pump
- Water Recycle Pump
- Solvent Cooler

CH_3Cl Purification

CH₃Cl Column and Reboiler

CH₂Cl₂ Cooler

Crude CH₂Cl₂ Tank

CH₃Cl Condenser

Reflux Accumulator

CH₃Cl Reflux Pump

Syltherm Utilities

Glycol Chiller

Glycol Pump

Glycol Expansion Tank

Secondary Hot Oil Pump

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Figure 2 - OHC Data Summary

Figure 3 - Methyl Chloride Absorbance for OHC Absorber/Stripper Solvents

Figure 4 - Temperature vs. Viscosity Data for OHC Absorber/Stripper Solvents

Figure 5 - Temperature vs. Viscosity Data for OHC Absorber/Stripper Solvents

Figure 6 - Schematic Diagram for OHC Laboratory Absorber Apparatus

FIGURE 1

RESULTS OF TEMPERATURE DEPENDENCE STUDY

	Eact (kJ/mol)	Hrxn (kJ/mol)
CH ₄ ---> CH ₃ Cl	121	-157
CH ₄ ---> CO	225	-500
CH ₄ ---> CO ₂	240	-781
CH ₃ Cl ---> CH ₂ Cl ₂	126	-153
CH ₃ Cl ---> CO	187	-343
CH ₃ Cl ---> CO ₂	197	-624

* HEATS OF CHLORINATION STEPS CAN INITIATE COMBUSTION REACTIONS

' CH₃Cl SELECTIVITY IS STRONGLY DEPENDENT UPON HEAT REMOVAL

Oxyhydrochlorination of Methane

Cumulative through period ending 12/31/92
 All Experiments with Methane Conversion $15 < x < 25$
 Variable Space Time, Fluid Velocity and Catalyst

Average MeCl Absorbance of OHC Absorber/Stripper Solvents

Temperatures between -3.6 to 0.5C

FIGURE 3

FIGURE 4

OHC Absorber/Stripper Solvents

Temperature vs. Viscosity

— Solvent A — Solvent C — Solvent D — Solvent E

Viscosity curves for A & C determined experimentally. Data for D & E supplied by manufacturer. No data for solvents B & F.

Temp (C)	Visc (cP)			
	Solvent A	Solvent C	Solvent D	Solvent E
25	84	51.5	6	8.85
15	157	78.8	8.4	11
5	336	129	12.8	13.9
-5	791	220	20	18
-15	1974	408	34	22
-20	3111	580	48	25

FIGURE 5

OHC Absorber/Stripper Solvents

Temperature vs. Viscosity

Temp (C)	Visc (cP)	
	Solvent D	Solvent E

25	6	8.85
15	8.4	11
5	12.8	13.9
-5	20	18
-15	34	22
-20	48	25

FIGURE 6

List of Tables

Table 1 - Copper (II) Based Catalysts for the Oxyhydrochlorination of Methane

Table 2 - Copper (II) Based Catalysts for the Oxyhydrochlorination of Methane

Table 3 - Copper-Aluminum Based Catalyst for the Oxyhydrochlorination of Methane

Table 4 - Methyl Chloride Absorbance of Oxyhydrochlorination Experimental Solvents

Table 1 Copper (II) Based Catalysts for the Oxyhydrochlorination of Methane

Catalyst Number	503	505	507	511	512	513	514
%Cu on Catalyst	< 0.1	< 0.1	1.37	0.99	1.12	2.27	1.98
Temperature (C)	365	365	365	350	350	350	350
Space Velocity (cm/s)	3.6	3.6	3.6	7.7	7.7	7.7	7.7
CH ₄ Conversion	1	13	41	19	23	47	11
HCl Conversion	0	5	53	21	22	69	13
O ₂ Conversion	3	32	74	36	43	83	92
% CH ₃ Cl	15	28	47	57	53	41	51
%CH ₂ Cl ₂	0	2	26	21	17	30	25
%CHCl ₃	0	0	6	4	3	12	5
%CCl ₄	0	0	1	0	0	2	0
%CO	65	43	13	12	16	10	13
%CO ₂	20	27	7	6	11	5	6

* all data in this table taken at atmospheric pressure

Table 2 Copper (II) Based Catalysts for the Oxyhydrochlorination of Methane

Catalyst Number	508A	508B	509C	509E	509F
%Cu on Catalyst	2.88	15.4	< .1	.43	1.81
Temperature (C)	365	350	350	350	350
Space Velocity (cm/s)	3.6	7.7	7.7	7.7	7.7
CH ₄ Conversion	16	18	5	19	14
HCl Conversion	10	15	4	23	27
O ₂ Conversion	30	43	6	29	18
% CH ₃ Cl	50	36	72	49	67
%CH ₂ Cl ₂	5	19	0	23	25
%CHCl ₃	1	5	0	8	0
%CCl ₄	0	0	0	0	0
%CO	42	37	20	12	7
%CO ₂	3	3	9	8	1

* all data in this table taken at atmospheric pressure

Table 3 Copper-Aluminum Based Catalyst for the Oxyhydrochlorination of MethaneExperimental Record 10346-001; 1.86% Cu, 0.14%Al on SiO₂; Space Velocity 7.7 cm/sec

Catalyst Number	518	518	518	518	518
Temperature (C)	315	325	335	350	390
CH ₄ Conversion	8	11	15	27	25
HCl Conversion	9	12	18	38	18
O ₂ Conversion	10	13	20	42	48
% CH ₃ Cl	72	66	61	48	38
%CH ₂ Cl ₂	18	22	24	29	14
%CHCl ₃	0	0	0	0	0
%CCl ₄	0	0	0	0	0
%CO	8	10	10	10	39
%CO ₂	10	12	12	11	46

* all data in this table taken at atmospheric pressure

Table 4 Methyl Chloride Absorbance of Oxyhydrochlorination Experimental Solvents

Solvent ID	Trade Name	% Wt. CH ₃ Cl	Temperature (C)	Pressure (psia)	CH ₃ Cl Saturation Pressure (psia)
A	Cerechlor 50LV	15.6	3	35	42
A	Cerechlor 50LV	15.4	25	28	41
A	Cerechlor 50LV	14.4	1	30	39
A	Cerechlor 50LV	19	-3.3	25	33
B	Cerechlor 49T	16.2	-3.6	24	33
B	Cerechlor 49T	18.1	-3.6	24	33
C	Mobil Oil SHC-624	13.2	0	33	38
C	Mobil Oil SHC-624	11.9	-12	15	24
D	Multitherm 503	11.6	-3	23	34
D	Multitherm 503	13	-3	23	34
E	Syltherm 800	21.7	0.5	27	38
E	Syltherm 800	23.5	0.5	29	38
F	Linear Dimethylpolysiloxane	20.5	0	35	38
F	Linear Dimethylpolysiloxane	27.6	0	33	38

Data Appendices

Data Appendix A- Experimental Data Sets

Data Appendix B- Catalyst Synthesis Information

Data Appendix A
Experimental Data Sets

00062175 OPERATOR NAME: B. CRUM DATE: 21-NOV-92
 00062175 LOG BOOK #: 10348 PAGE #: 102
 00062175 RUN #: 10348043 CATALYST #: 163A

00062175
 00062175 COMMENTS:

00062175
 00062175

00062175 MASS OF CATALYST (g): 6.59
 00062175 REACTOR INSIDE DIAM (CM): 0.6
 00062175 CATALYST BED HEIGHT (CM): 25

00062211 =====
 00062211 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6
 00062211 =====
 00062211 INITIAL FINAL ABSOLUTE
 00062211 TEMP TEMP RAMP RATE
 00062211 (DEG C) (DEG C) (DEG C / MIN)
 00062211 =====
 00062211 BOTTOM: 310 316 2
 00062211 MIDDLE: 310 316 2
 00062211 TOP : 310 316 2
 00062211
 00062211 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00062211
 00062211 CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 00062211 HCl FLOW RATE (CC/MIN): 29.6 ALX FLOW RATE (CC/MIN): 0

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	MBO6	Q6CH4C	Q6HCLC	TQI612
69692	0.00	0.00	0.00	100.00	0.00	0.00	0.00	1.00	132.65	134.12	1.28	4.33	3
74901	0.00	0.00	0.00	100.00	0.00	0.00	0.00	1.00	140.12	151.54	2.98	10.09	3
80104	0.00	0.00	0.00	100.00	0.00	0.00	0.00	1.00	139.87	152.06	3.94	13.33	3
85382	0.00	0.00	0.00	100.00	0.00	0.00	0.00	1.00	138.97	153.68	4.34	14.70	3
90651	0.00	0.00	0.00	100.00	0.00	0.00	0.00	2.00	136.27	146.77	-1.52	-8.58	3
96223	0.00	0.00	0.00	100.00	0.00	0.00	0.00	2.00	137.53	148.03	4.02	20.34	3
101442	0.00	0.00	0.00	100.00	0.00	0.00	0.00	2.00	137.30	146.35	0.15	0.75	3
106640	0.00	0.00	0.00	100.00	0.00	0.00	0.00	2.00	137.03	149.48	16.60	91.17	3
111877	0.00	0.00	0.00	100.00	0.00	0.00	0.00	2.00	139.45	150.38	6.34	21.44	3
117144	0.00	0.00	0.00	100.00	0.00	0.00	0.00	3.00	140.47	151.68	-0.45	-1.53	3
122752	0.00	0.00	0.00	87.46	5.76	6.78	0.00	3.00	135.29	139.24	7.42	30.02	3
127956	0.00	0.00	0.00	100.00	0.00	0.00	0.00	3.00	138.50	142.51	5.41	18.26	3
133151	0.00	0.00	0.00	88.65	0.00	11.15	0.00	3.00	135.62	141.25	0.38	1.57	3
138431	0.00	0.00	0.00	91.59	8.41	0.00	0.00	3.00	138.90	144.48	2.62	9.60	3
143676	0.00	0.00	0.00	91.38	0.00	8.62	0.00	4.00	137.33	141.45	2.49	9.88	3
149264	0.00	1.78	1.78	94.97	3.25	0.00	0.00	4.00	135.95	134.97	10.87	37.31	3
154460	0.00	1.51	1.51	91.33	2.97	4.19	0.00	4.00	132.09	128.61	13.35	49.47	3
159695	0.00	1.65	1.65	93.78	4.58	0.00	0.00	4.00	133.79	128.63	8.17	28.47	3
164960	0.00	1.36	1.36	91.81	2.32	4.50	0.00	4.00	131.11	124.80	9.21	34.35	3
170222	0.00	1.43	1.43	95.31	3.25	0.00	0.00	5.00	133.46	127.57	9.52	32.73	3
175810	0.00	1.97	1.97	92.81	5.22	0.00	0.00	5.00	132.70	111.55	13.05	45.52	3
181080	0.00	2.79	2.79	91.07	6.14	0.00	0.00	5.00	19827.87	55.44	11.68	0.15	3
AVG & STD AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	MBO6	Q6CH4C	Q6HCLC	TQI612	
AVG 310	0.00	0.00	0.00	100.00	0.00	0.00	0.00	1.20	137.58	147.63	2.20	6.77	
	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.40	2.82	7.14	2.14	8.47	
AVG 316	0.00	0.00	0.00	100.00	0.00	0.00	0.00	2.20	138.35	149.18	5.33	26.43	
	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.40	1.36	1.85	6.16	33.75	
AVG 322	0.00	0.00	0.00	91.86	2.83	5.31	0.00	3.20	137.13	141.79	3.66	13.87	
	0.00	0.00	0.00	4.36	3.57	4.55	0.00	0.40	1.46	1.71	2.47	9.65	
AVG 328	0.00	1.55	1.55	93.44	3.27	1.74	0.00	4.20	133.28	128.92	10.22	36.46	
	0.00	0.15	0.15	1.62	0.73	2.13	0.00	0.40	1.65	3.33	1.79	7.10	

RUN 10346043

CATALYST 163A

RUN 10346043

CATALYST 163A

OPERATOR NAME: S CRUM DATE: 25-NOV-82
 LOG BOOK #: 10346 PAGE #: 116
 RUN #: 10346050 CATALYST #: 538
 CATALYST NAME:
 COMMENTS: EXTENDED RUN TEMP IN BED
 MASS OF CATALYST (G): 3.6
 REACTOR INSIDE DIAM (CM): 0.6
 CATALYST BED HEIGHT (CM): 25.4

TEMPERATURE RAMPING EXPERIMENT FOR HOOO #6

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=====
INITIAL FINAL ABSOLUTE
TEMP TEMP RAMP RATE
(°C) (°C) (°C / MIN)
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BOTTOM: 310 310 2
MIDDLE: 310 310 2
TOP: 310 310 2
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(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 H2 FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	O6CH4C	O6HCLC	TO612	
15464	0.00	1.14	1.15	93.51	5.36	0.00	0.00	0.00	1.00	99.88	6.46	22.88	310
20548	0.00	0.00	0.00	96.28	1.74	0.00	0.00	0.00	1.00	99.78	6.77	26.14	310
25632	0.00	2.22	1.93	96.67	0.90	0.00	0.00	0.00	1.00	99.73	6.22	23.83	310
30736	0.00	2.26	1.95	96.81	0.94	0.00	0.00	0.00	1.00	99.76	6.18	23.70	311
35840	0.00	2.24	2.13	96.79	0.98	0.00	0.00	0.00	2.00	100.58	5.88	20.72	313
41308	0.00	2.58	2.29	95.85	1.47	0.00	0.00	0.00	2.00	99.81	6.85	25.73	316
46407	0.00	2.85	2.72	95.82	1.43	0.00	0.00	0.00	2.00	99.93	6.31	20.73	318
51508	0.00	2.89	2.72	93.67	3.34	0.00	0.00	0.00	2.00	99.41	7.27	24.82	318
56624	0.00	2.52	2.55	92.89	4.95	0.00	0.00	0.00	2.00	99.18	6.50	22.46	318
61888	0.00	2.78	2.78	92.28	4.98	0.00	0.00	0.00	3.00	99.47	6.88	23.28	319
67188	0.00	2.81	2.84	90.32	6.87	0.00	0.00	0.00	3.00	97.80	9.31	32.88	323
72267	0.00	2.93	2.87	90.28	6.81	0.00	0.00	0.00	3.00	97.88	9.48	33.08	323
77365	0.00	2.87	2.89	90.11	7.02	0.00	0.00	0.00	3.00	97.88	9.87	34.28	322
82468	0.00	2.83	3.18	90.08	7.01	0.00	0.00	0.00	3.00	98.07	5.23	17.09	322
87556	0.00	2.99	3.25	90.08	6.96	0.00	0.00	0.00	4.00	98.08	9.15	29.78	324
93026	0.00	4.33	5.03	82.88	12.78	0.00	0.00	0.00	4.00	99.32	13.35	43.04	328
98128	0.00	3.78	4.30	84.60	11.58	0.05	0.00	0.00	4.00	98.21	13.88	45.12	328
103228	0.00	3.82	4.40	84.17	11.85	0.05	0.00	0.00	4.00	98.08	13.10	42.81	328
108327	0.00	3.83	4.41	84.12	11.88	0.05	0.00	0.00	4.00	98.02	14.79	47.88	328
113427	0.00	3.94	4.54	83.82	12.18	0.05	0.00	0.00	5.00	95.35	14.75	47.28	330
118906	0.00	7.57	7.85	72.70	17.14	2.59	0.00	0.00	5.00	91.81	22.28	62.14	335
123986	0.00	17.17	17.67	62.08	17.73	3.07	0.00	0.00	5.00	89.34	24.84	63.21	334
129103	0.00	19.85	24.81	60.87	16.77	2.81	0.00	0.00	5.00	91.08	21.08	53.67	334
134212	0.00	6.74	11.04	71.30	17.32	2.84	0.00	0.00	5.00	93.88	20.12	60.71	334
139371	0.00	6.24	10.38	71.80	17.24	2.82	0.00	0.00	5.00	93.82	21.88	66.78	334
144446	0.00	7.77	9.77	72.83	17.13	2.47	0.00	0.00	5.00	94.32	20.14	61.18	334
149563	0.00	7.87	9.88	72.84	16.87	2.51	0.00	0.00	5.00	94.08	20.12	61.92	335
154684	0.00	7.45	9.38	73.07	16.84	2.54	0.00	0.00	5.00	93.71	20.38	62.04	334
159784	0.00	7.28	9.14	73.22	16.82	2.56	0.00	0.00	6.00	93.81	21.88	67.88	337
165234	0.00	14.81	18.78	64.83	16.14	2.22	0.00	0.00	6.00	93.70	22.82	62.28	340
170324	0.00	17.58	22.08	62.70	17.57	2.18	0.00	0.00	6.00	92.67	22.88	59.88	340
175424	0.00	16.24	20.48	63.78	17.80	2.18	0.00	0.00	6.00	92.88	23.88	64.38	340
180524	0.00	14.88	16.91	65.47	17.38	2.25	0.00	0.00	6.00	92.88	22.38	61.48	341
185625	0.00	14.05	17.71	66.07	17.84	2.24	0.00	0.00	6.00	92.88	21.88	61.01	341
190725	0.00	13.44	16.88	66.47	17.85	2.25	0.00	0.00	6.00	92.88	24.07	67.88	341
195840	0.00	13.02	16.94	66.91	17.67	2.78	0.00	0.00	6.00	92.83	22.47	62.88	341
200986	0.00	12.38	15.75	67.08	17.75	2.82	0.00	0.00	6.00	93.38	23.38	67.47	340
206086	0.00	12.21	15.44	67.88	17.42	2.88	0.00	0.00	7.00	93.33	21.88	62.28	348
221811	0.00	16.50	23.37	61.39	17.35	2.78	0.00	0.00	7.00	92.08	24.88	63.84	347
232063	0.00	17.84	22.21	62.58	17.14	2.88	0.00	0.00	7.00	92.98	22.75	60.08	348
237163	0.00	17.22	21.78	62.88	17.30	2.78	0.00	0.00	7.00	92.47	22.88	60.34	347
242246	0.00	16.88	21.28	63.17	17.28	2.67	0.00	0.00	7.00	92.28	22.77	60.34	347
247385	0.00	16.88	21.34	63.04	17.43	2.84	0.00	0.00	7.00	92.18	24.80	68.08	347
252485	0.00	16.94	20.88	63.45	17.33	2.88	0.00	0.00	8.00	92.35	22.70	60.48	348
257523	0.00	22.80	29.82	54.88	19.38	2.98	0.00	0.00	8.00	98.58	23.88	58.74	352
262638	0.00	19.85	25.07	60.37	16.88	2.70	0.00	0.00	8.00	92.58	23.88	60.84	352
268184	0.00	19.74	24.85	60.43	17.13	2.71	0.00	0.00	8.00	92.34	23.37	59.02	352
273281	0.00	22.00	28.81	55.88	19.40	3.02	0.00	0.00	8.00	98.48	23.88	58.71	352
288844	0.00	16.88	23.70	61.43	17.07	2.85	0.00	0.00	8.00	92.17	23.48	60.78	352
293944	0.00	16.37	23.08	61.87	17.01	2.88	0.00	0.00	8.00	91.87	22.88	58.81	352
299044	0.00	17.83	22.44	62.38	17.11	2.87	0.00	0.00	8.00	92.18	23.81	61.74	352
304144	0.00	17.80	22.08	62.84	16.88	2.83	0.00	0.00	9.00	92.81	23.88	60.78	348
309247	0.00	9.42	11.67	72.22	16.20	2.15	0.00	0.00	9.00	92.38	19.81	58.78	340
314348	0.00	8.31	10.30	73.28	16.22	2.18	0.00	0.00	9.00	92.90	22.98	68.22	340
319448	0.00	7.82	9.88	73.87	16.08	2.13	0.00	0.00	9.00	92.88	21.08	64.18	340
324548	0.00	8.22	10.18	73.38	16.27	2.18	0.00	0.00	9.00	92.20	20.67	62.41	340
329648	0.00	7.80	9.78	74.02	15.88	2.08	0.00	0.00	9.00	92.88	21.48	65.02	341
330184	0.00	7.82	9.32	74.31	15.73	2.14	0.00	0.00	9.00	92.88	18.78	57.08	341
335239	0.00	8.13	10.08	73.78	15.82	2.18	0.00	0.00	9.00	92.38	20.88	63.08	340
340348	0.00	8.18	10.08	73.83	16.05	2.17	0.00	0.00	9.00	92.40	20.38	61.71	340
AVG & STD A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	O6CH4C	O6HCLC	TO612	311	
AVG 310	0.00	1.57	1.43	96.48	1.98	0.00	0.00	0.00	1.20	99.85	6.30	23.48	311
SVG 316	0.00	0.88	0.79	1.57	1.71	0.00	0.00	0.00	0.40	0.32	0.30	1.78	1
AVG 322	0.00	2.84	2.81	94.13	3.24	0.00	0.00	0.00	2.20	99.28	6.72	23.38	317
AVG 328	0.00	0.00	0.18	1.58	1.57	0.00	0.00	0.00	0.40	0.23	0.33	1.73	1
AVG 334	0.00	2.81	3.02	90.18	6.83	0.00	0.00	0.00	3.20	97.88	8.56	28.40	323
AVG 340	0.00	0.08	0.18	0.11	0.08	0.00	0.00	0.00	0.40	0.18	1.87	6.33	1
AVG 346	0.00	3.84	4.54	83.82	12.10	0.04	0.00	0.00	4.20	96.84	13.88	45.18	329
AVG 352	0.00	0.21	0.28	0.37	0.38	0.02	0.00	0.00	0.40	1.47	0.88	2.18	1
AVG 358	0.00	10.32	12.35	70.00	17.13	2.65	0.00	0.00	5.11	92.80	21.40	66.84	335
AVG 364	0.00	4.50	5.23	4.70	0.27	0.17	0.00	0.00	0.31	1.80	1.38	9.41	1
AVG 370	0.00	14.37	18.08	65.82	17.71	2.40	0.00	0.00	6.11	93.07	22.78	63.28	341
AVG 376	0.00	1.88	2.07	1.51	0.19	0.28	0.00	0.00	0.31	0.35	0.79	2.58	1
AVG 382	0.00	17.28	21.81	62.71	17.30	2.70	0.00	0.00	7.17	92.31	23.28	61.78	347
AVG 388	0.00	0.84	0.81	0.88	0.00	0.08	0.00	0.00	0.37	0.17	0.88	2.25	1
AVG 394	0.00	19.84	24.88	58.88	17.82	2.73	0.00	0.00	8.13	93.28	23.48	59.81	352
AVG 400	0.00	1.78	2.82	2.87	1.01	0.15	0.00	0.00	0.33	1.88	0.34	1.78	1
AVG 406	0.00	8.22	10.18	73.57	16.08	2.15	0.00	0.00	9.00	92.81	20.67	62.88	340
AVG 412	0.00	0.40	0.81	0.80	0.17	0.03	0.00	0.00	0.00	0.23	1.08	3.28	0

RUN 10346050

CATALYST 536

—■— CONVERSION OF CH₄

RUN 10346050

CATALYST 536

RUN 10346050

CATALYST 536

— REACTOR INSIDE TEMPERATURE

00062628 OPERATOR NAME: B. CRUM DATE: 03-DEC-92
 00062628 LOG BOOK #: 10346 PAGE #: 120
 00062628 RUN #: 10346052 CATALYST #: 536

00062628
 00062628 COMMENTS: CHECKING TEMP RAISE WHEN FLOWS STARTED-LONG TERM RUN AT 340

LONG TERM RUN

00062415 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6
 00062415 =====
 00062415 INITIAL FINAL ABSOLUTE
 00062415 TEMP TEMP RAMP RATE
 00062415 (DEG C) (DEG C) (DEG C / MIN)
 00062415 =====
 00062415 BOTTOM: 340 340 2
 00062415 MIDDLE: 340 340 2
 00062415 TOP : 340 340 2

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
68393	0.00	16.15	16.15	78.08	5.77	0.00	0.00	1.00	102.20	15.89	47.84	34
73476	0.00	3.22	3.22	83.36	13.42	0.00	0.00	1.00	92.55	16.00	59.86	34
78575	0.00	3.36	3.36	83.24	13.40	0.00	0.00	1.00	92.38	15.92	59.43	34
83666	0.00	3.45	3.45	82.80	13.74	0.00	0.00	1.00	91.94	16.01	59.77	34
88778	0.00	3.54	3.54	82.69	13.77	0.00	0.00	1.00	92.26	16.42	60.87	34
93873	0.00	3.47	3.47	82.99	13.54	0.00	0.00	1.00	91.45	16.76	62.66	34
98975	0.00	3.51	3.51	82.98	13.52	0.00	0.00	1.00	90.88	15.92	58.66	34
104076	0.00	3.54	3.54	82.90	13.56	0.00	0.00	1.00	90.90	16.25	60.15	34
109174	0.00	3.58	3.58	82.83	13.59	0.00	0.00	1.00	90.67	15.96	59.13	34
114275	0.00	3.74	3.74	82.30	13.96	0.00	0.00	1.00	90.46	15.88	59.39	34
119376	0.00	3.61	3.61	83.01	13.38	0.00	0.00	1.00	91.11	16.14	59.67	34
124474	0.00	3.63	3.63	82.77	13.60	0.00	0.00	1.00	90.86	15.82	57.81	34
129575	0.00	3.72	3.72	82.51	13.76	0.00	0.00	1.00	89.83	15.78	58.82	34
134675	0.00	3.88	3.88	82.27	13.84	0.00	0.00	1.00	89.53	16.59	61.78	34
139773	0.00	3.73	3.73	82.85	13.42	0.00	0.00	1.00	89.61	16.65	61.34	34
144874	0.00	3.79	3.79	82.59	13.62	0.00	0.00	1.00	89.69	16.85	62.60	34
149966	0.00	3.74	3.74	82.48	13.78	0.00	0.00	1.00	88.98	16.38	60.60	34
155074	0.00	3.74	3.74	82.64	13.63	0.00	0.00	1.00	89.19	16.52	61.23	34
160174	0.00	3.99	3.99	82.12	13.89	0.00	0.00	1.00	88.63	15.96	59.05	34
165274	0.00	3.96	3.96	82.40	13.64	0.00	0.00	1.00	89.06	17.02	62.89	34
170376	0.00	3.87	3.87	82.73	13.41	0.00	0.00	1.00	89.25	16.33	60.11	34
175474	0.00	3.84	3.84	82.74	13.42	0.00	0.00	1.00	89.00	15.44	58.96	34
180575	0.00	3.71	3.71	83.30	12.99	0.00	0.00	1.00	89.63	15.92	58.92	34
185674	0.00	3.80	3.80	83.01	13.19	0.00	0.00	1.00	89.71	15.53	57.50	34
190767	0.00	3.88	3.88	82.74	13.38	0.00	0.00	1.00	90.24	15.59	57.47	34
195874	0.00	3.91	3.91	82.68	13.41	0.00	0.00	1.00	88.95	15.95	59.26	34
200975	0.00	3.61	3.61	83.64	12.75	0.00	0.00	1.00	89.90	16.38	59.91	34
206073	0.00	3.87	3.87	83.21	12.92	0.00	0.00	1.00	89.38	16.10	59.59	34
211167	0.00	3.76	3.76	83.25	12.99	0.00	0.00	1.00	89.08	16.36	60.29	34
216274	0.00	3.85	3.85	83.16	12.99	0.00	0.00	1.00	90.24	15.36	58.80	34
221375	0.00	3.72	3.72	83.61	12.67	0.00	0.00	1.00	89.56	16.54	61.15	34
226473	0.00	3.91	3.91	83.15	12.94	0.00	0.00	1.00	89.69	15.66	57.91	34
231575	0.00	3.90	3.90	83.13	12.97	0.00	0.00	1.00	90.19	15.34	56.21	34
236676	0.00	3.79	3.79	83.32	12.89	0.00	0.00	1.00	89.91	15.75	58.33	34
241774	0.00	3.82	3.82	83.60	12.58	0.00	0.00	1.00	90.60	15.68	57.78	34
246875	0.00	3.85	3.85	83.51	12.64	0.00	0.00	1.00	90.54	15.36	56.63	34
251973	0.00	3.84	3.84	83.58	12.58	0.00	0.00	1.00	90.32	14.89	54.52	34
257074	0.00	3.55	3.55	84.07	12.38	0.00	0.00	1.00	90.95	15.18	55.83	34
262175	0.00	3.79	3.79	83.77	12.43	0.00	0.00	1.00	91.04	14.61	53.90	34
267274	0.00	3.54	3.54	84.12	12.33	0.00	0.00	1.00	91.04	14.93	55.09	34
272375	0.00	3.52	3.52	84.49	12.00	0.00	0.00	1.00	91.60	14.89	54.71	34
277473	0.00	3.68	3.68	84.26	12.06	0.00	0.00	1.00	91.77	15.06	55.25	34
282575	0.00	3.59	3.59	84.61	11.80	0.00	0.00	1.00	91.92	14.86	54.58	34
287668	0.00	3.73	3.73	84.12	12.14	0.00	0.00	1.00	91.63	14.93	54.93	34
292775	0.00	3.74	3.74	84.33	11.94	0.00	0.00	1.00	91.85	15.07	55.33	34
297874	0.00	3.72	3.72	84.28	12.00	0.00	0.00	1.00	91.70	14.42	52.76	34
302975	0.00	3.55	3.55	84.88	11.57	0.00	0.00	1.00	92.01	14.00	51.18	34
308076	0.00	3.49	3.49	85.15	11.36	0.00	0.00	1.00	91.74	14.12	51.17	34
313174	0.00	3.52	3.52	85.09	11.40	0.00	0.00	1.00	92.09	14.46	52.38	34
318276	0.00	3.63	3.63	84.95	11.42	0.00	0.00	1.00	91.65	12.24	44.53	34

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
323374	0.00	3.51	3.51	85.11	11.38	0.00	0.00	1.00	91.58	12.99	47.14	34
328475	0.00	3.60	3.60	84.98	11.42	0.00	0.00	1.00	91.87	14.21	51.58	34
333590	0.00	3.64	3.64	84.93	11.43	0.00	0.00	1.00	92.42	14.24	51.97	34
338692	0.00	3.65	3.65	84.95	11.40	0.00	0.00	1.00	91.55	13.73	49.82	34
343774	0.00	3.54	3.54	85.33	11.13	0.00	0.00	0.00	91.67	13.53	49.01	34
351454	0.00	3.68	3.68	84.84	11.50	0.00	0.00	1.00	92.51	13.14	48.01	34
356556	0.00	3.53	3.53	85.45	11.02	0.00	0.00	1.00	91.88	13.64	49.89	34
361654	0.00	3.61	3.61	85.27	11.13	0.00	0.00	1.00	91.50	12.99	47.02	34
366755	0.00	3.64	3.64	85.26	11.09	0.00	0.00	1.00	91.42	13.30	48.37	34
371856	0.00	3.17	3.17	85.78	11.05	0.00	0.00	1.00	91.91	12.73	46.69	34
376954	0.00	3.67	3.67	85.18	11.15	0.00	0.00	1.00	91.24	13.53	48.98	34
382056	0.00	3.55	3.55	85.89	10.76	0.00	0.00	1.00	91.94	13.65	49.58	34
387171	0.00	3.40	3.40	86.14	10.46	0.00	0.00	1.00	92.00	14.08	50.77	34
392255	0.00	3.60	3.60	85.82	10.77	0.00	0.00	1.00	91.46	13.94	50.71	34
397356	0.00	3.56	3.56	85.84	10.61	0.00	0.00	1.00	91.64	13.17	47.37	34
402471	0.00	3.51	3.51	86.09	10.41	0.00	0.00	1.00	92.15	13.42	48.69	34
407547	0.00	3.44	3.44	86.27	10.29	0.00	0.00	1.00	91.98	13.85	50.13	34
412656	0.00	3.57	3.57	86.06	10.37	0.00	0.00	1.00	91.49	12.50	45.04	34
417756	0.00	3.59	3.59	86.00	10.41	0.00	0.00	1.00	91.52	13.42	48.54	34
422854	0.00	3.40	3.40	86.34	10.26	0.00	0.00	1.00	91.85	13.10	47.41	34
427955	0.00	3.53	3.53	86.19	10.28	0.00	0.00	1.00	91.64	12.71	45.67	34
433055	0.00	3.54	3.54	86.24	10.22	0.00	0.00	1.00	92.07	13.22	47.74	34
438157	0.00	3.48	3.48	86.56	9.96	0.00	0.00	1.00	92.26	12.70	45.89	34
443255	0.00	3.46	3.46	86.48	10.07	0.00	0.00	1.00	91.90	13.43	48.36	34
448355	0.00	3.31	3.31	86.67	9.82	0.00	0.00	1.00	92.10	12.95	46.55	34
453454	0.00	3.48	3.48	86.55	9.97	0.00	0.00	1.00	91.26	12.48	44.64	34
458571	0.00	3.54	3.54	86.47	9.99	0.00	0.00	1.00	91.78	11.89	42.97	34
463653	0.00	3.23	3.23	87.07	9.70	0.00	0.00	1.00	91.68	13.07	46.90	34
468755	0.00	3.47	3.47	87.03	9.51	0.00	0.00	1.00	91.93	11.98	43.00	34
473855	0.00	3.29	3.29	87.06	9.65	0.00	0.00	1.00	91.94	12.78	45.58	34
478955	0.00	3.49	3.49	86.99	9.53	0.00	0.00	1.00	92.04	12.99	46.74	34
484055	0.00	3.50	3.50	86.98	9.52	0.00	0.00	1.00	91.99	12.61	45.01	34
489155	0.00	3.61	3.61	86.84	9.74	0.00	0.00	1.00	91.81	12.21	44.06	34
494255	0.00	3.42	3.42	87.24	9.34	0.00	0.00	1.00	92.14	12.61	45.32	34
499355	0.00	3.47	3.47	87.38	9.15	0.00	0.00	1.00	92.49	11.95	42.92	34
504455	0.00	3.41	3.41	87.38	9.21	0.00	0.00	1.00	92.44	12.10	42.99	34
509555	0.00	3.50	3.50	87.30	9.20	0.00	0.00	0.00	92.57	12.64	45.19	34
517294	0.00	3.49	3.49	87.19	9.32	0.00	0.00	1.00	93.10	12.26	44.02	34
522394	0.00	3.46	3.46	87.60	8.94	0.00	0.00	1.00	93.56	11.73	41.88	34
527495	0.00	3.53	3.53	87.36	9.11	0.00	0.00	1.00	93.52	12.23	43.53	34
532594	0.00	3.50	3.50	87.37	9.13	0.00	0.00	1.00	93.14	11.38	40.38	34
537694	0.00	3.53	3.53	87.61	8.86	0.00	0.00	1.00	94.10	11.43	40.85	34
542794	0.00	3.44	3.44	87.96	8.60	0.00	0.00	1.00	93.27	11.69	41.38	34
547894	0.00	3.68	3.68	87.05	9.27	0.00	0.00	1.00	81.60	14.22	51.05	34
552996	0.00	4.89	4.89	83.65	11.46	0.00	0.00	1.00	83.30	13.39	48.47	34
558096	0.00	3.39	3.39	87.79	8.83	0.00	0.00	1.00	92.66	12.11	43.47	34
563197	0.00	3.49	3.49	87.91	8.61	0.00	0.00	1.00	93.56	11.69	41.45	34
568294	0.00	3.49	3.49	88.02	8.49	0.00	0.00	1.00	91.78	13.09	46.16	34
573394	0.00	3.42	3.42	88.21	8.36	0.00	0.00	1.00	94.24	11.97	42.53	34
578495	0.00	3.63	3.63	87.66	8.72	0.00	0.00	1.00	93.35	11.19	39.68	34
583595	0.00	3.39	3.39	88.44	8.17	0.00	0.00	1.00	92.53	11.79	41.91	34
588695	0.00	3.51	3.51	88.03	8.46	0.00	0.00	1.00	94.98	10.17	36.13	34
593795	0.00	3.40	3.40	88.48	8.12	0.00	0.00	1.00	95.40	11.19	39.85	34
598895	0.00	3.32	3.32	88.70	7.99	0.00	0.00	1.00	95.09	11.01	39.10	34
603995	0.00	3.37	3.37	88.69	7.94	0.00	0.00	1.00	99.89	10.67	37.63	34
609095	0.00	3.28	3.28	88.67	7.85	0.00	0.00	1.00	95.71	10.49	37.13	34
614195	0.00	2.93	2.93	89.32	7.78	0.00	0.00	1.00	95.80	9.74	34.63	34
619295	0.00	3.38	3.38	88.74	7.88	0.00	0.00	1.00	95.61	10.31	36.47	34
624395	0.00	3.39	3.39	89.06	7.53	0.00	0.00	1.00	95.97	10.57	37.30	34
629495	0.00	3.45	3.45	88.90	7.65	0.00	0.00	1.00	95.41	10.20	35.77	34
634596	0.00	3.46	3.46	88.82	7.72	0.00	0.00	1.00	95.45	10.60	37.66	34
639696	0.00	2.54	2.54	89.97	7.49	0.00	0.00	1.00	95.65	10.34	36.76	34
644796	0.00	3.24	3.24	89.41	7.35	0.00	0.00	1.00	95.67	9.91	34.75	34
649893	0.00	3.44	3.44	88.97	7.59	0.00	0.00	1.00	95.34	10.48	37.01	34
654986	0.00	3.44	3.44	89.14	7.42	0.00	0.00	1.00	95.16	9.50	33.20	34
660095	0.00	3.39	3.39	89.37	7.24	0.00	0.00	1.00	95.35	9.89	34.86	34

RUN 10346052

CATALYST 536

RUN 10346052

CATALYST 536

RUN 10346052

CATALYST 536

OPERATOR NAME: B. CRUM DATE: 11-DEC-92
 LOG BOOK #: 10346 PAGE #: 122
 RUN #: 10346053 CATALYST #: 163C

COMMENTS: CHECKING TEMPS 2 TC'S IN BED(OUT&IN) ACT@340 RUN 340
 NOTES: RUN DIED AT END SHUT OFF DUE TO HIGH PRESSURE
 BEFORE RUN DIED-ALL O2 FED WAS BEING USED
 MASS OF CATALYST (g): 5.1
 REACTOR INSIDE DIAM (CM): 0.6
 CATALYST BED HEIGHT (CM): 24

TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6

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

 BOTTOM: 340 340 2
 MIDDLE: 340 340 2
 TOP : 340 340 2

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

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

Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQ1612
11942												34
17042	0.00	31.50	31.50	64.31	4.19	0.00	0.00	1.00	91.50	18.54	45.83	34
22140	0.00	34.52	34.52	58.39	7.08	0.00	0.00	1.00	86.82	19.15	47.03	34
27241	0.00	47.06	47.06		12.75	0.00	0.00	1.00	99.82		44.35	34
32341	0.00	28.74	28.74	63.12	8.14	0.00	0.00	1.00	86.40	19.37	51.96	34
37442	0.00	24.97	24.97	66.09	8.94	0.00	0.00	1.00	92.00	20.03	56.94	34
42539	0.00	41.83	41.83		13.99	0.00	0.00	1.00	104.63	21.51	52.37	34
47641	0.00	26.78	26.78	63.93	9.29	0.00	0.00	1.00	86.77	22.05	61.86	34
52741	0.00	27.59	27.59	62.20	10.21	0.00	0.00	1.00	87.14	22.47	62.85	34
57841	0.00	27.60	27.60	62.15	10.25	0.00	0.00	1.00	88.57	20.81	57.80	34
62942	0.00	23.37	23.37	66.16	8.46	0.00	0.00	1.00	85.45	19.34	55.79	34
68043	0.00	21.63	21.63	70.85	7.52	0.00	0.00	1.00	86.62	19.64	56.91	34
73140	0.00	20.66	20.66	71.44	7.91	0.00	0.00	1.00	84.70	20.71	61.18	34
78241	0.00	21.03	21.03	71.36	7.60	0.00	0.00	1.00	85.27	20.94	61.06	34
83341	0.00	23.94	23.94	66.70	10.36	0.00	0.00	1.00	88.99	22.13	64.40	34
88441	0.00	36.24	36.24		16.29	0.00	0.00	1.00	104.17	22.87	61.77	34
93541	0.00	19.59	19.59	71.18	9.23	0.00	0.00	1.00	82.76	23.02	69.82	34
98642	0.00	20.73	20.73	69.67	9.39	0.00	0.00	1.00	80.56	23.08	69.27	34
103743	0.00	40.91	40.91		17.79	0.00	0.00	1.00	106.19	22.51	58.30	34
108841	0.00	25.58	25.58	62.47	11.95	0.00	0.00	1.00	75.75	21.64	63.45	34
113942	0.00	26.96	26.96	60.54	12.50	0.00	0.00	1.00	82.44	23.65	68.03	34
119042	0.00	26.52	26.52	60.63	12.85	0.00	0.00	1.00	90.60	23.19	67.39	34
124147	0.00	24.88	24.88	63.60	11.51	0.00	0.00	1.00	88.37	23.81	69.42	34
129242	0.00	19.84	19.84	70.90	9.26	0.00	0.00	1.00	85.85	24.68	74.66	34
134343	0.00	24.77	24.77	63.29	11.94	0.00	0.00	1.00	89.55	24.67	72.95	34
139441	0.00	26.51	26.51	60.49	13.00	0.00	0.00	1.00	74.77	24.38	70.96	34
144525	0.00	35.19	35.19		17.51	0.00	0.00	1.00	101.96	24.24	67.18	34
149641	0.00	24.95	24.95	62.32	12.73	0.00	0.00	1.00	94.82	24.49	72.35	34
154724	0.00	21.76	21.76	67.16	11.08	0.00	0.00	1.00	93.27	24.57	73.65	34
159839	0.00	20.13	20.13	69.69	10.18	0.00	0.00	1.00	74.45	25.70	77.87	34
164940	0.00	36.42	36.42		18.97	0.00	0.00	1.00	103.83	25.39	70.66	34
170050	0.00	22.15	22.15	66.27	11.58	0.00	0.00	1.00	94.05	25.09	75.99	34
175225	0.00	19.99	19.99	69.62	10.40	0.00	0.00	1.00	86.94			34
180344	0.00	32.88	32.88		18.59	0.00	0.00	1.00	84.35	26.57	76.69	34
185461	0.00	19.78	19.78	69.99	10.23	0.00	0.00	1.00	93.83	24.83	75.93	34
190569	0.00	25.06	25.06	61.24	13.70	0.00	0.00	1.00	89.38	26.15	78.61	34
195703	0.00	20.10	20.10	67.83	12.07	0.00	0.00	1.00	87.80	25.78	80.47	34
200821	0.00	36.06	36.06		20.16	0.00	0.00	1.00	104.61	26.04	74.28	34
205922	0.00	22.22	22.22	65.25	12.53	0.00	0.00	1.00	91.62	25.35	77.08	34
211008	0.00	19.73	19.73	69.84	10.43	0.00	0.00	1.00	88.41			34
216129	0.00	18.97	18.97	70.69	10.14	0.00	0.00	1.00	73.43	26.27	80.66	34
221289	0.00	39.84	39.84		21.42	0.00	0.00	1.00	115.77	25.88	71.48	34
226440	0.00	18.79	18.79	71.02	10.19	0.00	0.00	1.00	92.25	26.35	81.63	34
231540	0.00	21.37	21.37	67.82	11.00	0.00	0.00	1.00	84.47	26.51	80.50	34
236656	0.00	19.24	19.24	70.14	10.62	0.00	0.00	1.00	89.78	25.67	78.83	34
241801	0.00	18.94	18.94	70.77	10.29	0.00	0.00	1.00	86.61	25.62	79.32	34
246941	0.00	19.23	19.23	70.13	10.65	0.00	0.00	1.00	86.31			34
252035	0.00	41.55	41.55		22.72	0.00	0.00	1.00	112.76	24.91	66.53	34
257145	0.00	19.63	19.63	69.39	10.98	0.00	0.00	1.00	88.67	26.33	80.90	34
262245	0.00	23.10	23.10	64.26	12.63	0.00	0.00	1.00	80.58	25.72	77.97	34
AVG & STD												
AVG	0.00	26.34	26.34	66.52	11.90	0.00	0.00	1.00	90.12	23.59	67.99	
	0.00	7.35	7.35	3.86	3.78	0.00	0.00	0.00	9.12	2.34	10.09	

RUN 10346053

CATALYST 163C

Data Set 4 - Page 2

—■— SELECTIVITY TO M1

GC BASELINE PROBLEMS CAUSED IRREGULAR DATA

RUN 10346053

CATALYST 163C

RUN 10346053

CATALYST 163C

Data Appendix B
Catalyst Synthesis and Composition Information

CATALYST 503

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	Copper phthalocyanine-3,4',4'',4'''-tetrasulfonic acid tetrasodium salt. 9947 lab-book page 20.
LOADING	< 0.1% Cu , < 0.1% Na.
IMPREGNATION METHOD	The aqueous clear blue solution of the cluster was left with the support about 10-20 minutes. The solution was decanted and the catalyst was washed with water.
PRETREATMENT	Under full vacuum at 100 ° ^{6,36} for about 2 hours.
COLOR	Blue.

CATALYST 505

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	Copper phthalocyanine-3,4',4'',4'''-tetrasulfonic acid tetrasodium salt. 9947 lab-book page 22.
LOADING	< 0.1% Cu.
IMPREGNATION METHOD	The aqueous clear dark blue solution of the cluster was left with the support about 23 hours. The solution was decanted and the catalyst was washed with water.
PRETREATMENT	Thermal treatment 350 C , 1 hour flowing dry nitrogen.
COLOR	White.

CATALYST 507

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ Mallinckrodt Lot 4824 KVRK. See 9947 lab-book page 24.
LOADING	1.37% Cu, 1.82% Cu.
IMPREGNATION METHOD	The saturated clear olive green acetonitrile solution of CuCl_2 was left with the support for impregnation overnight. The solvent was removed by a flow of nitrogen.
PRETREATMENT	Under vacuum for 5 hours at 120 °C .
COLOR	Dark olive green.

CATALYST 508A

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$\text{Cu}(\text{BF}_4)_2$. See 9947 lab-book page 27.
LOADING	2.88% Cu, < 0.1% B.
IMPREGNATION METHOD	The 45% aq. solution of $\text{Cu}(\text{BF}_4)_2$ was left with the support for impregnation overnight. Most of the solvent water was removed by a flow of air.
PRETREATMENT	Under vacuum for several hours at 80 °C .

CATALYST 508B

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$\text{Cu}(\text{BF}_4)_2$. See 9947 lab-book page 27.
LOADING	15.4% Cu, <0.1% B.
IMPREGNATION METHOD	The 45% aq. solution of $\text{Cu}(\text{BF}_4)_2$ was left with the support for impregnation overnight. Most of the solvent water was removed by a flow of air.
PRETREATMENT	Under vacuum for several hours at 80 °C .

CATALYST 509C

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	Copper phthalocyanine-3,4',4'',4'''-tetrasulfonic acid tetrasodium salt. See at 9947 lab-book page 30.
LOADING	< 0.1% Cu, Na = 0.35%.
IMPREGNATION METHOD	The aq. solution of the salt was left with the support for impregnation overnight. Most of the solvent water was removed by rotavapor at 100 °C .
PRETREATMENT	Under vacuum overnight at 100 °C .
COLOR	Blue

CATALYST 509E

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	Copper phthalocyanine-3,4',4'',4'''-tetrasulfonic acid tetrasodium salt. See at 9947 lab-book page 30.
LOADING	0.43% Cu, Na = 0.92%.
IMPREGNATION METHOD	The aq. solution of the salt was left with the support for impregnation overnight. Most of the solvent water was removed by rotavapor at 100 °C .
PRETREATMENT	Under vacuum overnight at 100 °C .
COLOR	Blue

CATALYST 509F

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	Copper phthalocyanine-3,4',4'',4'''-tetrasulfonic acid tetrasodium salt. See at 9947 lab-book page 30.
LOADING	1.81% Cu, Na = 3.34%.
IMPREGNATION METHOD	The aq. solution of the salt was left with the support for impregnation overnight. Most of the solvent water was removed by rotavapor at 100 °C .
PRETREATMENT	Under vacuum overnight at 100 °C .
COLOR	Blue

CATALYST 511

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. 9947 lab-book page 34.
LOADING	0.992% Cu, 1.34% Cl.
IMPREGNATION METHOD	Very short impregnation period. While heating the solvent was evaporated using a nitrogen flow.
PRETREATMENT	Under vacuum for 3 hours at 140 °C, and R.T. overnight under static vacuum.
COLOR	Green.

CATALYST 512

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. 9947 lab-book page 35
LOADING	Cu = 1.12% and Cl = 0.99%
IMPREGNATION METHOD	The acetonitrile solution of the salt was added to the support for impregnation overnight. While heating the solvent was evaporated using a nitrogen flow.
PRETREATMENT	Under vacuum for 3 hours at 140 °C, and R.T. overnight under static vacuum.
COLOR	Dark olive green.

CATALYST 513

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	Li_2CuCl_4 . 9947 lab-book page 36
LOADING	2.27% Cu, 0.50% Li, 3.58% Cl.
IMPREGNATION METHOD	The THF solution of the salt was added to the support for impregnation overnight. While heating the solvent was removed under vacuum.
PRETREATMENT	Under vacuum for 3 hours at 350 °C. In the beginning the color of the catalyst was orange but as the solvent was being removed and the temperature was increased the following color changes took place. Orange to yellow to black to yellow to olive green to gray.
COLOR	Final color is gray. In water it becomes almost black and later dark olive green.

CATALYST 514

SUPPORT	Fumed Silica, Cabot MS-75
SOURCE	Li_2CuCl_4 . 9947 lab-book page 38.
LOADING	1.98% Cu, 0.1% Li, 5.12% Cl.
IMPREGNATION METHOD	The THF solution of the salt was added to the support for impregnation overnight. While heating the solvent was removed under vacuum.
PRETREATMENT	Under vacuum for 3 hours at 400 °C. In the beginning the color of the catalyst was orange but as the solvent was being removed and the temperature was increased the following color changes took place. Orange to yellow to black to yellow to olive green to gray.
COLOR	Gray.

CATALYST 518

SUPPORT	Silica (CS-1040)
SOURCE	"Cu ₆ Al" cluster. 9947 lab-book page 43 and 54.
LOADING	1.86% Cu, 0.14% Al.
IMPREGNATION METHOD	The acetonitrile solution of the cluster was left with the support overnight. The pale blue color of the solution was decanted and the catalyst was dried (see pretreatment).
PRETREATMENT	Under full vacuum A. 50-60 °C for 1 hour B. 200 °C for 2 hours C. 350 °C for 2 hours
COLOR	Black

CATALYST 536

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	CuF ₂ ·2H ₂ O, LiCl, LaCl ₃ . 9947 lab-book page 90.

**DATE
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1 / 27 / 94

END