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Methyl Chloride Via Oxyhydrochlorination of Methane

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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

Several key technology areas were evaluated this quarter.

The catalyst definition effort focused on the determination of the role of the Li and La promoters that have been found to be useful in enhancing Cu based oxyhydrochlorination of methane catalysts. Initial experiments show that the La acts to provide a much more active catalyst than the Cu only case. The role of the Li is ambiguous at this point. The Li enhances the stability of the La promoted catalyst, but gives only marginal improvement by itself. This work will be continued, with additional emphasis on the analysis of the catalysts to determine the structural role that the promoters may play.

The separation unit operation definition made significant progress by demonstrating in a laboratory system that a process solvent may be used to remove the product CH_3Cl from the reactor effluent stream. To date the data has been qualitative, but clear. Work will continue to gather the information possible in the laboratory to help with PDU design.

An extensive amount of testing was performed on the chosen process solvent, Multitherm. A comprehensive review of all the thermal testing and associated FTIR, UV/VIS, and physical property testing is included in this report. This work shows that Multitherm should give the desired stability and solubility that are necessary to make the separation unit operation successful.

Technical Results

Task 1. Catalyst Definition and Separation Unit Operation Development.

Catalyst Definition-

Table 1 gives an overview of the catalyst screening reactions that occurred during this time period. All reactions were performed at ambient pressure, with the %Cu on the catalyst, Temperature, Space Velocity, measured CH_4 conversion, calculated HCl and O_2 conversion, and product selectivities listed by catalyst number. Comprehensive data with associated graphs of the CH_4 conversion and CH_3Cl selectivity vs. time are given in Data Appendix A. Reactor temperature profile data is given when available. Additional catalyst synthesis information for the catalysts cited in this report is given in Appendix B.

Experimental Data Set 1 in Appendix A shows a data set obtained from a Cu-Li based catalyst labelled catalyst #513. Page 1 of this actual laboratory data set shows experimental identifiers as well as selectivity and conversion data. In the data section columns Al6CO, Al6CO₂, Al6COX, Al6M1, Al6M2, Al6M3, and Al6M4 refer to the carbon mole based selectivities to CO, CO₂, CO + CO₂, CH_3Cl , CH_2Cl_2 , CHCl_3 , and CCl_4 . Columns Q6CH₄C and Q6HClC refer to the measured CH_4 conversion and the resultant calculated HCl conversion. This will be the general format in which all raw laboratory data will be presented. Pages 2 and 3 of this data set show the conversion and selectivity vs time in graphical form. Note that this catalyst was relatively stable for the duration of this 140 hour experiment. The decrease in activity over this time was about 25%. The selectivity data has a significant variability due to some gas chromatography problems that occurred during the experiment. The temperature profile on page 4 shows that there was no significant exotherm that occurred during this reaction as measured by the difference between the thermocouple placed inside the reactor and that place on the outside wall of the reactor. Indeed the temperature profile for the entire experiment was very flat.

Data Set 2 shows the results of the first in a series of experiments designed to evaluate the impact of the Li and La promoters that are used in some oxychlorination catalysts. Catalyst #538A is simply the Cu complex used as a Cu donor which is supported on silica. As summarized in Table 1, other catalysts which are labelled #538B, #538C, and #538D were synthesized utilizing sequential impregnation. #538B has La only as a added promoter, #538C has Li only, and #538D has both Li and La added. As will be discussed, these promoters have both stability and activity impacts. The summary shown on page 2 of Data Set 2 gives the conversion of catalyst #538A as an average of 4% for 340 C and 5% for a 350 C reactor furnace set point. Note that the temperature was changed at about 100 hours into the run as seen on page 5 in the temperature profile. This data clearly indicates that the system containing only Cu does not give the desired catalyst activity. In addition, the deactivation over this 280 hour experiment is significant, as shown on pages 3 and 4 of Data Set 2.

Table 1 Copper Based Catalysts for the Oxyhydrochlorination of Methane

%Cu on Catalyst	2.3	2.5	2.5	1.8	1.8	25	2.5	2.5
Temperature (C)	340	340	350	340	340	340	340	340
Space Velocity (cm/s)	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0
CH ₄ Conversion	16	4	5	16	8	3	6	16
HCl Conversion	57	14	18	62	30	10	22	65
O ₂ Conversion	41	10	13	46	21	7	15	46
% CH ₃ Cl	81	85	77	68	81	85	85	72
%CH ₂ Cl ₂	12	8	14	19	13	7	11	20
%CHCl ₃	0	0	0	3	1	0	0	3
%CCl ₄	0	0	0	0	0	0	0	0
%CO	4	6	6	3	3	5	1	2
%CO ₂	4	1	3	6	2	2	3	3

Data Set 3 shows the impact of adding La as a promoter to this catalyst. The La was added by sequential impregnation of the same base catalyst, #538A, to form catalyst #538B. In this case, the experiment had to be aborted at about 40 hours due to plugging of the reactor system, and therefore, stability information was not obtained. However, the activity impact is quite clear. The conversion for the La promoted catalyst is four times that of the non-promoted catalyst. As shown on page 3 of the data set the selectivity was significantly lower at 16% conversion than was seen in Data Set 1 at the same conversion for the Cu-Li catalyst. Based on this limited observation it appears that the La serves to enhance the activity of the catalyst, but a selectivity penalty may be also seen. However, the selectivity penalty is not certain because the selectivity differences are of the same order as the detection ability. Another interesting fact, shown on page 4 of Data Set 3, is that a small but measurable increase in the reaction exotherm was seen during this experiment.

Data Set 4 shows another experiment with catalyst #538B. In this case, the reaction was carried out for 265 hours. This data clearly shows that the La promoter alone does not give the desired catalyst stability. The conversion of the catalyst dropped from 15% to about 7% over the course of the experiment. As seen on page 5 of Data Set 4, another temperature effect was seen in this experiment. In this case, a significant exotherm was noted in the first 20 hours of the experiment. This may have caused the observed activity loss due to catalyst deactivation at higher temperatures.

Data Set 5 is included for the sake of completeness. This is a rerun of catalyst #538A for 290 hours. Note that the conversion stayed relatively steady, but was quite low at an average of 3%. Significant portions of the data are missing due to problems in the Camile control system during this experiment. This again illustrates the lack of activity of the base Cu catalyst used in this study.

Data Set 6 gives the results for the Li promoted version of catalyst #538A, catalyst #538C. Again, the data is spotty due to instrumental problems. However, it is quite clear that this catalyst again possesses much lower activity than the La promoted version. The average conversion was 6%. As shown on the page 3 graph, the initial conversion was about 10%, and it declined steadily. In this system, there was essentially no temperature differential noted between the two monitoring thermocouples as shown on page 5.

Data Set 7 shows the initial results from catalyst #538D. This catalyst has both Li and La as promoters. The experiment lasted 210 hours and was ended due to a plug in the system. Temperature data was inadvertently monitored for another 60 hours. The page 5 thermocouple data clearly shows the "heat of reaction" by showing an approximate 7 C temperature drop when the reactant feed was stopped. This catalyst gave very good results. As shown on page 3 of the data set, the initial conversion was about 19%, dropping to about 15 % after 210 hours. Although this is still not great, this level of deactivation is consistent with the best catalysts we have evaluated thus far. The selectivity averaged 72% for the experiment. This data indicates that both the Li and the La promoters are necessary in this system for optimization of the catalyst performance. Work in the next reporting period will focus on further evaluations of these catalysts. Testing to evaluate the physical changes in the catalyst structure associated with the addition of these promoters is planned.

Separation Process Definition-

There were two significant efforts this quarter in the definition of the separation process to be used in the PDU. The first was the demonstration in the laboratory that the product, CH_3Cl , could be removed via absorption into the desired process solvent in a laboratory column.

Data Set 8 shows the results of the first successful demonstration of the laboratory separation system. This 1 page data set shows the experimental conditions and results for an oxyhydrochlorination reaction at a pressure of 30 psig followed by removal of the product chlorocarbons in an absorber column. The CH_4 conversion and product selectivities are shown, as well as the %M1 removed from the reactor effluent. This was monitored via on-line gas chromatography by the use of a switching valve that sampled the stream before and after the absorber column. This first shakedown experiment simply shows that 60+% of the CH_3Cl was removed.

Data Set 9 shows the next shakedown run for this system. In this case data was collected at a system pressure of both 30 and 40 psig. It appeared that the removal efficiency was higher, as expected, at the higher system pressure. With both Data Sets 8 and 9, it is important not to over interpret these very interesting results. These were not viewed as steady state experiments, and this work will be continued next quarter to obtain more quantitative information. It is, however, exciting to note that the increase in system pressure appears to help rather than hurt the process selectivity to CH_3Cl . This could prove to be a major advantage in the development of this technology.

The second effort this quarter in the definition of the separation process involved the evaluation of the chosen process solvent. The oxyhydrochlorination of methane process requires the use of a solvent that will prove stable through temperature and pressure changes over an extended time period. Due to the nature in which this solvent is used, it is necessary that the chosen solvent be resistant to degradation that may be caused from thermal aging, pressure fluctuations, or acid attack. The following section will describe the processes used to determine the reliability of one solvent, Multitherm 503, under these varying circumstances.

PROPERTIES OF MULTITHERM 503

<u>GENERAL CHEMICAL CLASS</u>	<u>SYNTHETIC HYDROCARBON</u>
<u>APPEARANCE</u>	<u>BRIGHT, CLEAR, COLORLESS</u>
<u>ODOR</u>	<u>NONE</u>
<u>POUR POINT</u>	<u><-75 F (<-60 C)</u>
<u>DENSITY 60 F (16 C)</u>	<u>6.65 lb/gal (0.789 g/cc)</u>
<u>FLASH POINT</u>	<u>320 F (160 C)</u>
<u>FIRE POINT</u>	<u>345 F (174 C)</u>
<u>AUTOIGNITION TEMPERATURE</u>	<u>613 F (324 C)</u>
<u>ATMOSPHERIC BOILING POINT</u>	<u>625 F</u>
<u>AVERAGE MOLECULAR WEIGHT</u>	<u>272</u>
<u>COEFFICIENT OF THERMAL EXPANSION</u>	<u>0.00054</u>

EXPERIMENTAL OVERVIEW

Several tests were conducted in an effort to better determine the reliability of Multitherm 503 while varying both time and temperature. These tests include: the Bomem Michelson series FTIR (Fourier Transform Infrared Spectroscopy) which utilizes Galactic, Inc. Lab Calc software, the Perkin Elmer Lambda 3B UV/VIS (Ultraviolet/Visible Spectrophotometer), the automatic Houllian Viscometer which utilizes ATPM Visco-Houllian software, and an experimental design that will allow for the absorption of gases into a liquid.

The FTIR, with resolution set at 2 cm^{-1} and scan number set at 50, measures peaks in the infrared region of the color spectrum. The limits placed on an FTIR spectrum range from 400-4000 wavenumbers.

The UV/VIS measures peaks in the near ultraviolet/visible region of the color spectrum. The limits placed on a UV/VIS trace range from 190-900 nanometers.

The Houllian Viscometer measures, in centistokes, the kinetic viscosity of a sample as it passes through a capillary tube. The absorption test, for our purposes, measures the amount of methyl chloride absorbed into a solvent.

DISCUSSION AND RESULTS

A. MULTITHERM 503 - THERMALLY AGED

1. RUN TIME AND TEMPERATURE

Thermal stability testing provides a way to observe the effects of thermal aging on samples of Multitherm 503. However, this testing alone only allows for a visual observation of the sample. The sample, nevertheless, can provide valuable information when exposed to other tests (i.e. FTIR, UV/VIS, viscosity, and absorption tests).

Table 2 lists the time and temperature at which each sample was thermally aged. Samples 1-8 were thermally aged at 175 C. Of these eight samples, samples 1-6 underwent a color change during the thermal aging process. As the thermal aging time for each sample increased, the yellowing of the sample intensified. However, samples 7-10 remained clear and colorless.

Samples 1-6 were run in metal containers while samples 7-10 were run in glass vials. Therefore, the discoloration of the first six samples could be due to the presence of metals during the thermal aging process.

Table 2 - Thermally Aged Multitherm Samples

Sample	Hours Thermally Aged	Temperature (C)
1	4	175
2	8	175
3	11	175
4	30	175
5	106	175
6	202	175
7	200	175
8	400	175
9	200	200
10	4	275

2. FTIR

Figures 1-7 (located in the appendix) are spectra of virgin Multitherm 503. Figure 1 shows the broad spectrum ranging from 400-4000 wavenumbers. Figures 2-7 are smaller regions of the spectrum which are enlarged for better viewing of peak formation. These figures may be used as a reference for the thermally aged spectra.

When slight changes were seen in two separate runs of clean Multitherm, a question arose as to the reproducibility of each spectra. Therefore, a baseline study was conducted in which three separate FTIR runs were performed on virgin Multitherm 503. A grassy region was seen in the spectrum ranging from 1550-2400 wavenumbers. After a comparison of this spectrum was made with the two other runs, it was decided that the scatter was probably a result of equipment noise. Nevertheless, all of the spectra peaks were located at the same wavenumber. No new peaks were seen for any of the spectra.

FTIR tests were run for samples 1-10. No significant peak changes could be found.

Figures 8 and 9 serve to illustrate this point. These are the broad spectra for samples 2 and 6. They are very similar. The peaks for both samples are located at the same wavenumbers, and no new peaks were observed.

It is necessary to divide the initial large spectrum into several smaller, "blown-up" spectra in order to better view peak activity. All sample spectra were enlarged with no new peaks being seen. For example, see **Figures 10-15**. These are spectra of samples 7 and 8. The runs have been overlayed to provide an easier way to detect peak fluctuation. Each region of the spectrum is very similar with no new pronounced peak formations. No new peaks were detected for the other thermally aged samples either.

3. VISCOSITY

Before viscosity measurements of the thermally aged samples began, eight different runs were made using clean Multitherm 503 with no thermal aging. This created a baseline with which the thermally aged samples could be compared.

The following results were obtained:

<u>SAMPLE</u>	<u>VISCOSITY (centistokes)</u>
1	7.7
2	7.4
3	7.7
4	7.4
5	7.8
6	7.8
7	7.8
8	7.8
9	7.7
10	7.7

Most of the viscosity results fell within the statistical process control limits calculated from the eight runs with clean Multitherm 503. However, samples 2 and 4 fell curiously below the calculated limits. As of

yet, the reason for this drop in viscosity is unknown. See **Figure 16** for a representative graph.

4. UV/VIS

The ultraviolet-visibility test measures the intensity of transmitted light at wavelengths between 190 - 900 nanometers. This provides data in the near ultraviolet region (<400 nm), visible region (400-800 nm), and the near infrared region (>800 nm). UV/VIS testing was conducted on the 10 thermally aged samples. No peaks were seen in the near IR or visible regions for these samples. However, for samples 1-6, the area under each curve in the near ultraviolet region gradually increased. The absorption of violet light caused these samples to appear yellow. Samples 7-10 were clear and colorless. Their UV/VIS trace varied only slightly from the trace produced from a run with clean Multitherm.

See **Figure 17**, virgin Multitherm 503, as a reference UV/VIS trace. **Figures 18-20** display the UV regions of samples 3, 4, and 6. Note the progressive broadening of the curve. **Figures 21 and 22** display the UV regions for samples 9 and 10. Note the resemblance that these traces have to the trace of virgin Multitherm. After thermal aging, these samples remained clear and colorless.

5. ABSORPTION

An absorption test was conducted on Multitherm samples 1-6, 11, and two clean Multitherm samples. Sample eleven, to be discussed shortly, involves aging Multitherm with HCl at elevated temperatures. The following results were obtained:

Table 3 - Temperature, Pressure, and Saturation Data for Methyl Chloride in Multitherm

Sample	Temperature (C)	Pressure (psig)	% Saturation
1	-3	11	15.4
2	-2	11	16.4
3	-3	10	16.0
4	3	11	16.2
5	-5	7	11.7
6	-5	10	10.9
11	0	12	19.5
clean	-3	8	12.4
clean	1	19	22.2

According to the data collected, an increase in pressure correlates with an increase in saturation point. **Figure 23** illustrates this hypothesis. Sample six, the only point not to fit the proposed pressure vs. saturation point correlation, is attributed to a variance in sample testing methods.

B. MULTITHERM 503 - THERMALLY AGED WITH HCl

1. RUN TIME AND TEMPERATURE

Since HCl is present in the oxyhydrochlorination process, it is important that its effects on Multitherm are observed. Samples 11-14 serve to fulfill this purpose. The amount of HCl to be added was determined by calculating approximately 25 weight % of the amount of Multitherm added. These samples also yellowed during the thermal aging process. It is believed that the presence of HCl causes this color change.

TABLE 4 - Thermally Aged Multitherm and HCl Samples

Sample	Hours Thermally Aged	Temperature (C)
11	100	175
12	200	175
13	400	175
14	28	200

2. FTIR

FTIR tests were run on samples 11-14. No significant peak changes were found. See **Figures 24-29**. These spectra are an overlay of samples 12 and 13. When compared to each other, no peak changes are seen. Even more importantly, when compared to a run with clean Multitherm (**Figures 2-7**), no new peak formations are noted. The other Multitherm and HCl samples display the same behavior.

3. VISCOSITY

Viscosity testing was conducted on samples 12-14. The following results were obtained:

<u>SAMPLE</u>	<u>VISCOSITY (cSt)</u>
12	7.8
13	7.8
14	7.8

These viscosity results fall within the statistical process control limits previously calculated from the eight runs with clean Multitherm. As of yet, no significant change in viscosity has been noted. See **Figure 16** for a representative graph.

4. UV/VIS

UV/VIS testing was conducted on samples 12-14. The run limits were placed at 190 (near UV region) and 900 (near IR region). No new peaks were seen in the IR or visible regions for samples 12-14. However, when compared to the clean Multitherm trace, the area under the curve in the UV region gradually increased. Again, it should be noted that these samples underwent a color change during the aging process. It is believed that the presence of HCl causes this color change.

See **Figure 17**, virgin Multitherm 503, as a reference UV/VIS trace. **Figures 30 and 31** display the UV regions of runs 12 and 14. Using the virgin Multitherm trace as a reference, note the broadening of the curve.

5. ABSORPTION

One absorption test was conducted on a Multitherm and HCl sample aged 100 hours at 175 C. The following results were obtained:

<u>TEMP. (Deg C)</u>	<u>PRESSURE (psig)</u>	<u>% SATURATION</u>
0	11.5	19.48

C. MULTITHERM 503 - THERMALLY AGED WITH METALS

1. RUN TIME AND TEMPERATURE

Samples 15-18, discussed further in TABLE 5, are samples thermally aged with Multitherm and a metal. The metals include: Hastelloy "C", Monel, Stainless Steel, and Inconel. A piece of metal tubing was placed in a vial with Multitherm and thermally aged at 175 C. After the thermal aging process was completed, the Multitherm sample was collected for further testing.

TABLE 5 - Thermally Aged Multitherm and Metal Samples

Sample	Metal	Hours Thermally Aged	Temperature (C)
15	Hastelloy C	100	175
16	Monel	100	175
17	Stainless Steel	100	175
18	Inconel	100	175

COMMENTS : Samples 16-18 turned bright yellow after thermal aging. Sample 15 remained clear and colorless.

2. FTIR

FTIR spectra were run for samples 15-18. In analyzing the data obtained, a comparison was made with an FTIR spectrum of clean Multitherm with no thermal aging. Because Multitherm aged with HCl causes no changes in the FTIR, **Figures 32 and 33** compare samples aged with metal to samples aged with HCl. Changes could be seen in all of the FTIR spectra for samples 15-18.

Multitherm and Hastelloy "C" - new peak forming around 1740 wavenumbers in the C=O region (see **Figure 32**).

Multitherm and Monel - new peak forming around 1740 wavenumbers in the C=O region (see **Figure 33**).

Multitherm and Stainless Steel - new peaks forming in the OH deformation and C-O, C=O regions. A new peak forming around 1740 wavenumbers in the C=O region (see **Figures 34-39**).

Multitherm and Inconel - new peaks forming in the OH deformation and C-O, C=O regions. A new peak forming around 1740 wavenumbers in the C=O region (see **Figures 40 and 41**).

3. VISCOSITY

No viscosity testing was conducted on samples 15-18. This test does not allow for sample recovery after testing is completed. Therefore, due to small amounts of sample, a decision was made to hold off on this test.

4. UV/VIS

The traces for samples 15-18, when compared to a trace of clean Multitherm, prove to have a significant increase in the area under the curve in the UV region. Although samples 16-18 were yellow, a characteristic that seems to correlate with the fact that the peaks in the UV region are of greater intensity than those samples visibly unaffected by thermal aging, sample 15 was clear and colorless. Yet, sample 15 displayed the same behavior as samples 16-18. This seems to indicate that the presence of metals during the thermal aging process intensifies the frequency in the UV region of the spectrum.

See **Figure 17**, virgin Multitherm, as a reference UV/VIS trace. **Figures 42-44** represent samples 15, 17, and 18. Note the broad curve in the UV regions. This is in direct contrast to the narrower curve produced from a run with clean Multitherm.

A UV/VIS test could not be conducted on sample 16, Multitherm and Monel, due to an insufficient amount of sample.

5. ABSORPTION

Absorption tests could not be run on the thermally aged Multitherm and metal samples due to the small amount of sample collected.

D. MULTITHERM 503 - THERMALLY AGED WITH METALS AND HCl

1. RUN TIME AND TEMPERATURE

Samples 19-22, discussed further in **TABLE 6**, are samples of Multitherm, metal, and concentrated HCl. The metals used include: Hastelloy "C", Monel, Stainless Steel, and Inconel. A piece of metal tubing was placed in a vial with Multitherm and concentrated HCl. The amount of HCl added to each vial was determined by calculating 25 weight % of the Multitherm added. The samples were aged for 100 hours at a temperature of 175 C.

TABLE 6 - Thermally Aged Multitherm , Metal, and HCl Samples

Sample	Metal	Hours Thermally Aged	Temperature (C)
19	Hastelloy C	100	175
20	Monel	100	175
21	Stainless Steel	100	175
22	Inconel	100	175

COMMENTS: Samples 19-22 formed some type of residue. However, the color of the Multitherm remained clear and colorless. No yellowing occurred.

Hastelloy "C" - greenish-black residue

Monel - brownish-green residue

Stainless Steel - green residue

Inconel - green residue

A clear gas, probably hydrogen, was produced during the thermal aging of these samples. A loud pop occurred when the sample vials were opened. Some solvent sprayed out of the vials and was lost upon opening.

2. FTIR

An FTIR spectrum could only be run for samples 19 and 20. Due to pressure formed within the vial during the thermal aging process, the samples containing Stainless Steel and Inconel sprayed out of the vial upon opening. Thus, no sample was collected. When both of the sample runs were compared with a spectrum of clean Multitherm, the most significant changes were seen in the C=O, C-O stretch region. See **Figures 45 and 46**.

3. VISCOSITY

No viscosity testing was conducted on samples 19-22. This test does not allow for sample recovery after testing is completed. Therefore, due to small amounts of sample, a decision was made to hold off on this test.

4. UV/VIS

A UV/VIS run was not conducted on samples 19-22 due to the small amount of sample collected.

5. ABSORPTION

Absorption tests could not be run on samples 19-22 due to the small amount of sample collected.

E. DE-GASSED MULTITHERM 503 - THERMALLY AGED WITH METALS; VACUUM SEALED

1. RUN TIME AND TEMPERATURE

Samples 23-27 are vacuum sealed samples of de-gassed Multitherm and a metal. All samples were aged 400 hours at 175 C. Before sample preparation, the Multitherm to be used was de-gassed using a bubbler and nitrogen flow. Nitrogen was allowed to bubble through the Multitherm for two hours. A syringe was then inserted through a septum on the side of the bubbler and Multitherm was withdrawn and inserted in a vial. After each vial was filled, the mouth was covered with a wax wrap. A vacuum pump was used to evacuate the head space in each vial. The vials were then sealed under a vacuum and placed in the sand bath.

TABLE 7 - Thermally Aged De-gassed Vacuum Sealed Multitherm and Metal Samples

Sample	Metal	Hours Thermally Aged	Temperature (C)
23	None	400	175
24	Hastelloy C	400	175
25	Monel	400	175
26	Tantalum	400	175
27	Zirconium	400	175

COMMENTS: All samples were clear and colorless

2. FTIR

FTIR tests were run for samples 23-27. No new peak formations were seen in any region of the spectra. The plot for each region of the spectra seemed to trace the spectrum for thermally aged, de-gassed Multitherm. Figures 47 and 48 display an overlay of two regions in each sample spectra. The spectra are unlabeled due to their obvious similarity.

3. VISCOSITY

Viscosity testing was not conducted on samples 23-27 due an insufficient amount of sample.

4. UV/VIS

UV/VIS testing was conducted on samples 23-27. Unlike previous samples that showed a broad curve in the ultraviolet region, these samples showed a very narrow curve that closely resembled the curve produced by a run with both clean Multitherm and thermally aged, de-gassed Multitherm.

Figure 49 is a trace of thermally aged de-gassed Multitherm. This trace can be used as a reference for the samples mentioned. Figures 50 and 51 are traces of samples 24 and 25. Note the narrow curve resembling the curves produced by both clean Multitherm and de-gassed thermally aged Multitherm.

5. ABSORPTION

Absorption testing was not conducted on samples 23-27 due to an insufficient amount of sample.

F. DE-GASSED MULTITHERM 503 - THERMALLY AGED WITH METALS AND HCl; VACUUM SEALED

1. RUN TIME AND TEMPERATURE

Samples 28-31 are vacuum sealed samples containing de-gassed Multitherm, a metal, and concentrated HCl. The same method of sample preparation used for the de-gassed samples void of HCl was used for the samples exposed to HCl.

TABLE 8 - Thermally Aged De-gassed Vacuum Sealed Multitherm ,Metal, and HCl Samples

Sample	Metal	Hours Thermally Aged	Temperature (C)
28	None	400	175
29	Hastelloy C	400	175
30	Tantalum	400	175
31	Zirconium	400	175

2. FTIR

FTIR tests were run for samples 28-31. No new peak formations were seen in any region of the spectra. **Figures 52 and 53** are an overlay of two regions for samples 28-31. Note the similarity of the spectra.

3. VISCOSITY

Viscosity testing was not conducted due to an insufficient amount of sample.

4. UV/VIS

UV/VIS testing was not conducted due to an insufficient amount of sample.

5. ABSORPTION

Absorption testing was not conducted due to an insufficient amount of sample.

III. SUMMARY AND CONCLUSIONS

An attempt has been made to determine the reliability of one solvent, Multitherm 503, through such systematic changes as temperature and pressure variation and exposure to concentrated acid. The following is a summary of the test results performed on Multitherm :

FTIR : No significant changes seen in the peaks formed for thermally aged samples with and without concentrated HCl. No significant changes seen in the peaks formed for vacuum sealed samples using degassed Multitherm with/without HCl. The only clear changes could be noted in samples of Multitherm thermally aged with a metal without first degassing the solvent.

VISCOSITY : No significant changes in viscosity for thermally aged samples with and without HCl.

UV/VIS : Heating Multitherm with HCl and/or metals causes a yellowing of the solvent. This yellowing was found to be preventable by degassing the solvent by bubbling nitrogen through it. It is therefore concluded that water or oxygen in the air react with Multitherm in the presence of HCl or metal to cause the yellowing.

ABSORPTION : Absorption testing was somewhat limited. However, in the samples tested with/without HCl, there were no significant changes in the absorption of MeCl.

The conclusion of this work is that there is no worrisome evidence of any sort that Multitherm will degrade during the conditions expected in the oxyhydrochlorination PDU. Any significant changes in the battery of evaluations done were found under extreme conditions that would not be found in the absorber or stripper columns of the PDU.

Task 2.0 Miniplant Design Engineering

Process Description and Definition

The major effort this quarter in the PDU Design Engineering effort was the completion of the process design package and the selection of the group that will do detail design and construction. After careful evaluation of both internal and external groups, it was decided to move forward with the project internally. The engineering support for the project will be given by the DCC small capital project group. This work is well underway.

The cost for the PDU is now much more completely defined than was possible at the earlier estimation points. At this initiation of this contract the cost was estimated to be in the \$1.0 to \$1.4 MM range. The addition of the reactor effluent separation system added significant cost and complexity to the project. The total capital cost is now estimated at \$2.3 to \$2.5 MM. Next quarter will see the start of detail engineering for this project. The estimated PDU start-up date is July 1994.

Res= 2 cm-1

FIGURE 1.
CLEAN MULTITHERM 503

Res= 2 cm-1

FIGURE 2
CLEAN MULTITHERM 503

FIGURE 3
CLEAN MULTITHERM 503

Res= 2 cm-1

FIGURE 4
CLEAN MULTITHERM 503

Res= 2 cm-1

FIGURE 5
CLEAN MULTITHERM 503

Res= 2 cm-1

Res = 2 cm-1

FIGURE 6
CLEAN MULTITHERM 503

FIGURE 7
CLEAN MULTITHERM 503

Res= 2 cm-1

FIGURE 8
MULTITHERM THERMALLY AGED 8 HRS @ 175 C
Res= 2 cm-1

FIGURE 9
MULTITHERM THERMALLY AGED 202 HRS @ 175 C

Res= 2 cm-1

FIGURE 10
Res= 2 cm⁻¹
MULTITHERM THERMALLY AGED 200 HRS @ 175 C
MULTITHERM THERMALLY AGED 400 HRS @ 175 C

FIGURE 11
Res= 2 cm-1
MULTITHERM THERMALLY AGED 200 HRS @ 175 C
MULTITHERM THERMALLY AGED 400 HRS @ 175 C

FIGURE 12 Res= 2 cm-1
MULTITHERM THERMALLY AGED 200 HRS @ 175 C
MULTITHERM THERMALLY AGED 400 HRS @ 175 C

FIGURE 13
MULTITHERM THERMALLY AGED 200 HRS @ 175 C
MULTITHERM THERMALLY AGED 400 HRS @ 175 C

FIGURE 14 **Res= 2 cm-1**
MULTITHERM THERMALLY AGED 200 HRS @ 175 C
MULTITHERM THERMALLY AGED 400 HRS @ 175 C

FIGURE 15
MULTITHERM THERMALLY AGED 200 HRS @ 175 C
MULTITHERM THERMALLY AGED 400 HRS @ 175 C

Res= 2 cm-1

SPC FOR MULTITHERM RUNS ON HOULLIAN VISCOMETER

A = CLEAN MULTITHERM
 B = MULTITHERM THERMALLY AGED 4 HRS @ 175 C
 C = MULTITHERM THERMALLY AGED 8 HRS @ 175 C
 D = MULTITHERM THERMALLY AGED 11.5 HRS @ 175 C
 E = MULTITHERM THERMALLY AGED 30 HRS @ 175 C
 F = MULTITHERM THERMALLY AGED 106 HRS @ 175 C
 G = MULTITHERM THERMALLY AGED 202 HRS @ 175 C
 H = MULTITHERM AFTER ABSORPTION

I = MULTITHERM THERMALLY AGED 4 HRS @ 275 C
 J = MULTITHERM THERMALLY AGED 200 HRS @ 200 C
 K = MULTITHERM AND CONCENTRATED HCL
 L = MULTITHERM THERMALLY AGED 28 HRS @ 200 C
 M = MULTITHERM THERMALLY AGED 200 HRS @ 175 C
 N = MULTITHERM AND HCL THERMALLY AGED 200 HRS @ 175 C
 O = MULTITHERM THERMALLY AGED 400 HRS @ 175 C
 P = MULTITHERM AND HCL THERMALLY AGED 400 HRS @ 175 C

FIGURE 16

L
11

ELMER

Chem. No. C22889-9

PERKIN-ELMER

Chem. No. C22889-9

FIGURE 18
MULTITHERM THERMALLY AGED 11.5 HRS @ 175 C

PERKIN-ELMER

Model No. C73000-0

FIGURE 19
MULTITHERM THERMALLY AGED 30 HRS @ 175 C

PERKIN-ELMER

CHART NO. CP32859 0

FIGURE 20
MULTITHERM THERMALLY AGED 202 HRS @ 175 C

FIGURE 21
MULTITHERM THERMALLY AGED 200 HRS @ 200 C

FIGURE 22
MULTITHERM THERMALLY AGED 4 HRS @ 275 C

PRESSURE VS. SATURATION POINT

- 1 = MULTITHERM THERMALLY AGED 4 HRS @ 175 C
- 2 = MULTITHERM THERMALLY AGED 8 HRS @ 175 C
- 3 = MULTITHERM THERMALLY AGED 11.5 HRS @ 175 C
- 4 = MULTITHERM THERMALLY AGED 30 HRS @ 175 C
- 5 = MULTITHERM THERMALLY AGED 106.5 HRS @ 175 C
- 6 = MULTITHERM THERMALLY AGED 202 HRS @ 175 C
- 7-10 = NO ABSORPTION TEST RUN
- 11 = MULTITHERM AND HCl THERMALLY AGED 100 HRS @ 175 C

FIGURE 23

FIGURE 24
MULTITHERM AND HCl THERMALLY AGED 200 HRS @ 175 C
MULTITHERM AND HCl THERMALLY AGED 400 HRS @ 175 C

Res= 2 cm-1

FIGURE 25
MULTITHERM AND HCl THERMALLY AGED 200 HRS @ 175 C
MULTITHERM AND HCl THERMALLY AGED 400 HRS @ 175 C

Res= 2 cm-1

FIGURE 26
MULTITHERM AND HCl THERMALLY AGED 200 HRS @ 175 C
MULTITHERM AND HCl THERMALLY AGED 400 HRS @ 175 C

FIGURE 27
MULTITHERM AND HCl THERMALLY AGED 200 HRS @ 175 C
MULTITHERM AND HCl THERMALLY AGED 400 HRS @ 175 C

Res= 2 cm-1

FIGURE 28
Res= 2 cm-1
MULTITHERM AND HCl THERMALLY AGED 200 HRS @ 175 C
MULTITHERM AND HCl THERMALLY AGED 400 HRS @ 175 C

FIGURE 29
Res= 2 cm⁻¹
MULTITHERM AND HCl THERMALLY AGED 200 HRS @ 175 C
MULTITHERM AND HCl THERMALLY AGED 400 HRS @ 175 C

PERKIN-ELMER

Chart No. C730000-0

FIGURE 30
MULTITHERM AND HCI THERMALLY AGED 200 HRS @ 175 C

FIGURE 31
MULTITHERM AND HQ THERMALLY AGED 28 HRS @ 200 C

PERKIN-ELMER
 LMD-100 CP-2000-0

FIGURE 32
 MULTITHERM AFTER THERMAL AGING WITH HASTELLOY C
 MULTITHERM AFTER THERMAL AGING WITH HCl

Res= 2 cm-1

FIGURE 33
MULTITHERM AFTER THERMAL AGING WITH MONEL
MULTITHERM AFTER THERMAL AGING WITH HCl

Res= 2 cm-1

FIGURE 34
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH STAINLESS STEEL

FIGURE 35
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH STAINLESS STEEL
Res= 2 cm-1

FIGURE 36
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH STAINLESS STEEL
Res= 2 cm-1

FIGURE 37
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH STAINLESS STEEL

Res= 2 cm-1

FIGURE 38
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH STAINLESS STEEL
Res = 2 cm-1

FIGURE 39
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH STAINLESS STEEL

Res= 2 cm-1

Res= 2 cm-1

FIGURE 40
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH INCONEL

FIGURE 41
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH INCONEL

Res= 2 cm-1

PERKIN-ELMER

Chart No. CP32858-0

FIGURE 42

PERKIN-ELMER

Chart No. CP25559-9

Chart No. CP25559-9

FIGURE 43
MULTITHERM THERMALLY AGED WITH INCONEL

.....

FIGURE 45
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH HASTELLOY C AND HCl
Res= 2 cm⁻¹

Res= 2 cm-1

FIGURE 46
CLEAN MULTITHERM
MULTITHERM AFTER THERMAL AGING WITH MONEL AND HCl

FIGURE 47
 DB-GASSED THERMALLY AGED MULTITHERM
 DE-GASSED MULTITHERM THERMALLY AGED WITH HASTELLOY
 DE-GASSED MULTITHERM THERMALLY AGED WITH MONEL
 DE-GASSED MULTITHERM THERMALLY AGED WITH TANTALUM
 DE-GASSED MULTITHERM THERMALLY AGED WITH ZIRCONIUM

FIGURE 48
 DE-GASSED THERMALLY AGED MULTITHERM
 DE-GASSED MULTITHERM THERMALLY AGED WITH HASTELLOY
 DE-GASSED MULTITHERM THERMALLY AGED WITH MONEL
 DE-GASSED MULTITHERM THERMALLY AGED WITH TANTALUM
 DE-GASSED MULTITHERM THERMALLY AGED WITH ZIRCONIUM

PERKIN ELMER

Model 142 6751 829-9

FIGURE 49
CLEAN MULTITHERM
DB - GASED/VACUUM SEALED

Chart No. CP3285-0

PERKIN-ELMER

Chart No. CP3285-0

FIGURE 50
MULTITHERM THERMALLY AGED WITH HASTELLOY -C
DE-GASSED/VACUUM SEALED

PERKIN-ELMER

Model 41200

PERKIN-EL

FIGURE 51
MULTITHERM THERMALLY AGED WITH MONEL
DB-GASSED/VACUUM SEALED

FIGURE 52

DE-GASSED MULTITHERM AND HCl THERMALLY AGED

DE-GASSED MULTITHERM THERMALLY AGED WITH HCl AND HASTELLOY "C"

DE-GASSED MULTITHERM THERMALLY AGED WITH HCl AND TANTALUM

DE-GASSED MULTITHERM THERMALLY AGED WITH HCl AND ZIRCONIUM

FIGURE 53

DE-GASSED MULTITHERM AND HCl THERMALLY AGED

DE-GASSED MULTITHERM THERMALLY AGED WITH HCl AND HASTELLOY "C"

DE-GASSED MULTITHERM THERMALLY AGED WITH HCl AND TANTALUM

DE-GASSED MULTITHERM THERMALLY AGED WITH HCl AND ZIRCONIUM

Data Appendix A
Experimental Data Sets

00013396 OPERATOR NAME: B. CRUM DATE: 01-APR-93
 00013396 LOG BOOK #: 11048 PAGE #: 8
 00013396 RUN #: 11048001 CATALYST #: 513
 00013396 CATALYST NAME:
 00013396 COMMENTS: LONG U2OQCl4 RUN SLOW RAMP
 00013398

00013398 MASS OF CATALYST (g): 1.81

00013396 REACTOR INSIDE DIAM (CM): 0.6

00013396 CATALYST BED HEIGHT (CM): 25.4

00013385 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6

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00013385      INITIAL  FINAL  ABSOLUTE  ABSOLUTE
00013385      TEMP    TEMP    RAMP RATE  RAMP RATE
00013385      (DEG C)  (DEG C)  (DEG C/MIN) (DEG C/HR)
-----
00013385      BOTTOM: 335    340    0.5    2      FLUID VELOCITY  9.0 CM/SEC
00013385      MIDDLE: 335    340    0.5    2      RUN TIME      140 HRS
00013385      TOP : 335    340    0.5    2
  
```

00013385 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00013385 CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8

00013385 HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

Time	A18CO	A18CO2	A18COX	A18M1	A18M2	A18M3	A18M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQ1812
20158	3	0	3	92	5	0	0	0	1	88	16	335
25273												
30433												
35593												
40753												
45815												
50833	2	2	3	90	7	0	0	1	116	17	61	340
51895						0	0	1	91	19	77	340
97155	3	3	6	81	13	0	0	1	81	19	69	340
102321	7	7	15	31	0	0	0	1	92	20	78	340
107472	4	4	8	75	17	0	0	1	83	20	73	340
112636	3	3	6	81	13	0	0	1	80	18	66	340
117794	3	3	6	80	13	0	0	1	82	19	68	340
122954	3	3	6	80	13	0	0	1	81	19	69	340
128113	2	2	4	89	8	0	0	1	72	18	64	340
133275	3	3	6	81	13	0	0	1	81	18	65	340
138434	3	3	6	83	11	0	0	1	78	17	62	341
143593	3	3	7	80	13	0	0	1	82	18	66	340
148753	3	3	6	82	12	0	0	1	81	18	66	340
153915	3	3	6	84	11	0	0	1	81	18	64	340
159074	2	2	5	88	8	0	0	1	82	18	62	340
164234	4	3	7	81	12	0	0	1	83	19	67	340
169393	4	3	7	81	13	0	0	1	83	17	61	340
174555	4	3	7	82	12	0	0	1	82	18	65	340
179714	1	1	2	97	1	0	0	1	69	19	63	340
184873	5	4	9	75	16	0	0	1	87	17	63	340
190035	3	2	5	86	9	0	0	1	80	18	58	340
195194	3	2	5	87	8	0	0	1	75	18	61	340
200353	5	4	9	78	15	0	0	1	89	17	61	340
205515	3	3	6	84	10	0	0	1	81	18	61	340
210674	4	3	7	81	12	0	0	1	83	17	61	340
215833	2	2	4	89	7	0	0	1	74	17	59	340
220993	3	3	6	84	10	0	0	1	81	18	61	340
226155	4	3	7	83	11	0	0	1	86	18	58	340
231314	3	2	5	88	7	0	0	1	78	18	56	340
236474	0	2	2	91	7	0	0	1	76	17	59	340
241634	0	5	5	80	16	0	0	1	91	17	63	340
246794	0	3	3	87	10	0	0	1	84	17	60	340
251955	0	1	1	95	4	0	0	1	69	17	59	340
257115	0	1	1	95	4	0	0	1	67	18	58	340
262274	0	5	5	80	15	0	0	1	90	17	63	341
267434	0	2	2	90	8	0	0	1	84	18	58	340
272594	0	5	5	80	15	0	0	1	93	15	58	340
277755	6	5	10	75	15	0	0	1	141	10	35	340
282915	0	2	2	90	8	0	0	1	86	18	58	340
288074	0	5	5	80	15	0	0	1	92	18	59	340
293235	0	5	5	80	15	0	0	1	142	14	54	340
298394	0	5	5	80	15	0	0	1	97	18	60	340
303555	0	1	1	96	3	0	0	1	61	17	57	340
308715	0	5	5	81	14	0	0	1	93	17	63	340
313873	0	5	5	81	14	0	0	1	93	18	59	340
319032	6	4	10	77	13	0	0	1	136	14	48	340
324195	0	5	5	81	14	0	0	1	91	18	58	340
329354	0	5	5	81	14	0	0	1	100	18	58	340
334515	0	5	5	81	14	0	0	1	92	17	61	340
339675	0	5	5	80	15	0	0	1	93	15	58	340
344834	0	5	5	81	14	0	0	1	92	18	59	340
349994	0	5	5	81	14	0	0	1	91	18	59	340
355155	0	5	5	80	14	0	0	1	93	15	58	340
360315	0	5	5	81	14	0	0	1	92	15	55	340
365474	0	5	5	81	14	0	0	1	92	15	56	340
370634	5	4	9	82	9	0	0	1	88	15	51	340
375794	7	6	12	78	13	0	0	1	94	16	53	340
380953	0	6	6	80	14	0	0	1	187		0	340
386115	8	6	14	73	13	0	0	1	92	15	60	340
391275	8	6	13	74	13	0	0	1	92	15	60	340
396434	8	6	14	73	13	0	0	1	94	15	48	340
401594	7	6	13	75	12	0	0	1	93	15	61	340
406755	8	6	14	72	14	0	0	1	95	15	52	340
411915	8	7	15	71	14	0	0	1	96	16	54	340
417074	8	6	14	74	12	0	0	1	94	16	52	340
422234	8	6	14	73	13	0	0	1	98	15	48	340
427393	8	6	15	72	13	0	0	1	97	14	47	340
432553	3	2	5	91	4	0	0	1	80	15	52	340
437715	8	6	14	74	12	0	0	1	91	15	61	340
442874	9	6	15	73	12	0	0	1	88	16	53	340
448034	8	7	15	72	13	0	0	1	95	15	51	340
453194	7	6	13	75	12	0	0	1	94	15	48	340
458355	8	6	15	72	13	0	0	1	108	16	55	340
463515	8	7	15	72	12	0	0	1	101	14	48	340
468674	9	7	15	72	13	0	0	1	95	15	50	340
473834	10	7	17	70	13	0	0	1	94	15	50	340
478993	3	2	5	91	4	0	0	1	77	14	48	340
484153	11	8	19	68	14	0	0	1	125	14	45	340
489315	3	2	6	90	4	0	0	1	84	13	43	340
494474	0	7	7	79	14	0	0	1	181		0	340
499634	0	2	2	93	4	0	0	1	68	18	60	340
504793	9	8	17	70	13	0	0	1	96	14	46	341
510000	8	7	15	73	12	0	0	1	95	14	47	340
Time	A18CO	A18CO2	A18COX	A18M1	A18M2	A18M3	A18M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQ1812
AVG	4	4	8	81	12	0	0	1	92	16	58	340.00
STD	3	2	5	7	4	0	0	0	21	2	11	

RUN 11046001

CATALYST 513

RUN 11046001
CATALYST 513

RUN 11046001

CATALYST 513

00008931 OPERATOR NAME: S. CRUM DATE: 08-APR-93
 00008931 LOG BOOK #: 11048 PAGE #: 10
 00008931 RUN #: 11048002 CATALYST #: 538A
 00008931 CATALYST NAME: Cu L4
 00008931 COMMENTS: Raised temp to target temp @ 1deg/min. Cat stability experiment
 00008931

00008931 MASS OF CATALYST (g): 2.71
 00008931 REACTOR INSIDE DIAM (CM): 6
 00008931 CATALYST BED HEIGHT (CM): 25.4

00008960 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6

=====													
	INITIAL	FINAL	ABSOLUTE	ABSOLUTE									
	TEMP	TEMP	RAMP RATE	RAMP RATE									
	(DEG C)	(DEG C)	(DEG C/MIN)	(DEG C/HR)									
=====													
	BOTTOM: 320	340	1	0	FLUID VELOCITY				9 CM/SEC				
	MIDDLE: 320	340	1	0	TIME				280 HRS				
	TOP: 320	340	1	0									
Time	AlaCO	AlaCO2	AlaCOX	AlaM1	AlaM2	AlaM3	AlaM4	IDS	MBC6	Q6CH4C	Q6HCLC	TCI612	
12981													
17533	5	0	5	95	0	0	0	0	1	96	5	17	340
22894	5	0	5	95	0	0	0	0	1	96	3	11	340
27882	5	0	5	95	0	0	0	0	1	96	4	11	340
33013	5	0	5	95	0	0	0	0	1	96	3	10	340
38174	5	0	5	89	6	0	0	0	1	97	5	18	340
43037	5	0	5	82	13	0	0	0	1	97	5	18	340
48487	6	0	6	86	6	0	0	0	1	97			340
53866	5	0	5	82	12	0	0	0	1	97	4	14	341
58815	6	0	6	89	5	0	0	0	1	96	4	12	340
63675	6	0	6	86	6	0	0	0	1	96	5	18	340
68136	6	1	7	88	5	0	0	0	1	97	4	12	340
74294	6	0	6	82	13	0	0	0	1	96	5	19	340
79453	6	1	7	81	6	0	0	0	1	97	5	20	340
84813	5	1	6	89	5	0	0	0	1	97	4	12	340
89836	5	0	6	77	11	0	0	0	1	97	4	17	340
94906	6	1	7	87	6	0	0	0	1	96	3	9	340
100162	6	1	7	88	5	0	0	0	1	95	3	11	340
105375	6	1	7	81	12	0	0	0	1	96	5	17	340
110587	6	1	7	81	12	0	0	0	1	96	5	17	340
115816	6	1	7	79	14	0	0	0	1	96	6	20	340
121033	6	1	8	79	13	0	0	0	1	96			340
126255	6	1	7	81	11	0	0	0	1	97	5	18	340
131473	5	2	7	78	12	0	0	0	1	96	5	21	340
136695	6	2	7	80	12	0	0	0	1	97	5	18	340
141914	6	2	8	86	7	0	0	0	1	96	5	18	340
147134	6	2	8	85	7	0	0	0	1	97	4	15	340
152352	6	2	8	73	12	0	0	0	1	96	4	18	340
157577	5	1	7	72	12	0	0	0	1	96	5	20	340
162736	5	2	8	75	6	0	0	0	1	96	4	17	340
167964	5	2	7	72	12	0	0	0	1	96	5	21	340
173175	5	2	7	72	11	0	0	0	1	95	4	15	340
178396	5	1	6	72	9	0	0	0	1	95	5	19	340
183617	5	2	7	71	11	0	0	0	1	96	5	21	340
188835	5	1	6	71	11	0	0	0	1	95	5	20	340
194056	5	1	7	74	7	0	0	0	1	95	6	24	340
199215	6	2	8	84	8	0	0	0	1	97	4	14	340
204375	6	2	9	84	7	0	0	0	1	97	5	15	340
209635	6	2	8	86	6	0	0	0	1	96	4	14	340
214741	5	2	7	80	13	0	0	0	1	96	5	17	340
219974	5	2	7	78	6	0	0	0	1	95	4	15	340
225192	5	2	7	79	5	0	0	0	1	95	3	10	340
230392	5	2	7	87	6	0	0	0	1	96	3	10	340
235514	6	2	8	83	9	0	0	0	1	96	3	9	340
240736	6	2	8	85	7	0	0	0	1	96	5	18	340
245957	6	2	8	81	11	0	0	0	1	96	3	9	340
251174	6	2	8	79	13	0	0	0	1	97	6	21	340
256393	6	2	8	80	12	0	0	0	1	97	4	18	340
261618	5	1	7	81	12	0	0	0	1	97	5	17	340
266775	7	1	8	85	8	0	0	0	1	96			340
271976	6	2	8	79	13	0	0	0	1	199			340
277154	6	2	8	86	6	0	0	0	1	96	3	9	340
282318	6	2	8	80	12	0	0	0	1	97	2	8	340
287534	6	3	9	84	8	0	0	0	1	97	6	20	340
292753	6	2	8	85	7	0	0	0	1	97	5	18	340
297913	6	2	8	80	12	0	0	0	1	95	3	9	341
303078	6	3	8	83	9	0	0	0	1	93	2	8	340
308297	6	2	8	86	6	0	0	0	1	91	6	20	340
313455	6	2	8	84	7	0	0	0	1	95	5	15	340
318675	6	2	8	81	11	0	0	0	1	95			340
323897	6	2	8	86	7	0	0	0	1	95	8	20	340
329115	6	2	8	85	7	0	0	0	1	95	3	11	340
334335	6	2	8	85	7	0	0	0	1	94	4	15	350
339537	7	2	9	81	9	0	0	0	1	93	8	27	350
344774	7	2	10	78	13	0	0	0	1	93	8	26	350
349998	7	3	10	78	12	0	0	0	1	94	6	28	350
355293	7	3	10	79	11	0	0	0	1	93	7	25	350
371098	8	3	10	77	13	0	0	0	1	93	7	24	350
376336	8	3	11	77	12	0	0	0	1	94	8	27	350
381557	8	3	11	80	9	0	0	0	1	95	7	25	350
386773	8	3	11	81	8	0	0	0	1	94	7	23	350
391998	8	3	10	81	9	0	0	0	1	93	7	24	350
397213	8	3	11	77	13	0	0	0	1	94	7	24	350
402374	7	3	10	78	12	0	0	0	1	97	5	18	350
407593	8	3	11	80	9	0	0	0	1	95	7	23	350
412755	8	3	11	81	9	0	0	0	1	95	6	21	350
423074	8	3	11	81	9	0	0	0	1	95	6	20	350
428293	8	3	11	78	11	0	0	0	1	95	7	24	350
433513	7	3	10	78	12	0	0	0	1	93	6	26	350
438735	9	3	12	77	12	0	0	0	1	90	9	32	350
=====													
449172	8	3	11	80	9	0	0	0	1	93	8	25	350
454354	7	3	10	77	12	0	0	0	1	94	6	26	350
459487	8	3	11	80	9	0	0	0	1	94	4	15	351
464713	8	3	11	81	8	0	0	0	1	94	6	21	350
469874	8	3	10	81	9	0	0	0	1	96	5	18	350
475095	8	3	11	81	9	0	0	0	1	94	8	25	350

	AI8CO	AI8CO2	AI8COX	AI8M1	AI8M2	AI8M3	AI8M4	ID8	MBC8	Q8CH4C	Q8HCLC	TQI812
480266	7	3	10	82	8	0	0	1	94	6	19	390
485478	6	3	10	79	11	0	0	1	93			390
480888	7	3	9	84	7	0	0	1	93	7	24	390
488686	8	3	11	81	8	0	0	1	118	4	14	390
501074	7	3	10	79	11	0	0	1	93	8	26	390
503293	7	3	10	83	7	0	0	1	93	7	24	390
511498	6	3	11	82	7	0	0	1	95	6	21	390
516675	7	3	10	82	8	0	0	1	94	6	21	390
521684	7	3	10	78	12	0	0	1	95	6	22	390
527118	7	3	10	79	12	0	0	1	95	6	26	391
532537	7	3	10	82	8	0	0	1	94	7	23	390
537484	7	3	10	81	8	0	0	1	95	5	18	390
542853	7	3	10	82	8	0	0	1	95	7	22	391
547875	7	3	10	77	13	0	0	1	96	7	24	390
550032	7	3	10	82	8	0	0	1	96	6	19	390
558186	7	3	10	78	12	0	0	1	95	5	18	390
563354	7	4	11	77	12	0	0	1	95	6	19	390
568614	6	4	10	78	12	0	0	1	96	5	18	390
573735	4	2	6	77	17	0	0	1	96	6	24	390
578687	7	3	9	78	15	0	0	1	96	5	17	390
584054	7	3	10	81	10	0	0	1	97	5	15	390
588814	7	3	10	83	7	0	0	1	96	6	20	391
594433	7	3	9	78	12	0	0	1	96	7	25	390
598678	7	3	10	81	8	0	0	1	97	5	16	391
604758	7	3	10	84	6	0	0	1	96	5	18	390
608817	6	3	9	86	5	0	0	1	96	5	17	390
615075	6	3	10	80	11	0	0	1	96	4	13	390
620234	7	3	10	79	11	0	0	1	96	2	7	390
625387	7	3	10	82	9	0	0	1	96	5	17	390
630613	7	3	9	82	9	0	0	1	96	7	23	390
635775	7	3	10	87	2	0	0	1	96	5	15	390
640834	7	3	11	87	3	0	0	1	96	6	18	390
646083	7	3	10	83	7	0	0	1	87	5	18	390
651257	7	3	10	83	7	0	0	1	87	5	18	390
656414	7	3	10	84	6	0	0	1	96	6	18	390
661617	6	2	9	78	13	0	0	1	96	7	23	390
666793	3	1	4	59	37	0	0	1	93	5	22	390
671956	6	3	9	83	8	0	0	1	95	5	17	390
677173	7	3	9	83	8	0	0	1	95	6	21	390
682335	6	3	10	80	11	0	0	1	96	5	16	390
687587	5	2	7	74	19	0	0	1	93	5	20	390
692758	7	3	9	83	7	0	0	1	96	6	19	390
697987	6	3	9	81	10	0	0	1	96	5	18	390
703212	7	3	9	86	5	0	0	1	96	6	18	390
708436	7	2	9	88	2	0	0	1	97	6	19	390
713653	7	3	9	86	5	0	0	1	96	5	17	390
718874	7	3	10	88	2	0	0	1	96	4	13	390
724087	7	3	9	85	6	0	0	1	96	6	18	390
729317	6	2	8	84	8	0	0	1	96	5	16	390
734474	6	3	9	80	11	0	0	1	95	5	17	390
739618	6	3	8	75	16	0	0	1	96	3	12	390
744854	6	3	9	82	9	0	0	1	96	6	21	390
750077	7	3	9	89	2	0	0	1	96	4	14	390
755294	6	2	8	81	11	0	0	1	95	5	17	348
760453	6	4	10	77	14	0	0	1	94	5	17	390
765674	6	3	9	82	9	0	0	1	94	7	22	390
770883	5	2	8	78	15	0	0	1	94	5	18	390
776113	6	2	8	76	16	0	0	1	94	5	16	391
781338	6	2	7	74	19	0	0	1	95	5	17	390
786562	5	2	7	76	17	0	0	1	95	5	19	390
791774	6	2	8	79	13	0	0	1	96	5	19	390
796934	6	3	9	75	16	0	0	1	95	3	12	390
802097	6	4	10	84	6	0	0	1	97	5	15	391
807253	6	2	8	80	12	0	0	1	96	5	16	390
812413	6	3	9	80	11	0	0	1	95	4	15	390
817635	5	2	7	72	21	0	0	1	95	4	17	390
822855	5	2	7	72	20	0	0	1	94	5	17	348
828074	5	2	7	74	19	0	0	1	94	4	14	390
833293	5	2	8	75	17	0	0	1	95	6	21	390
838517	5	2	7	74	18	0	0	1	94	4	14	390
843720	6	2	8	72	20	0	0	1	81	5	18	390
848954	5	2	7	67	27	0	0	1	93	5	19	390
854174	5	1	7	70	23	0	0	1	93	5	18	391
859337	5	3	8	77	14	0	0	1	96	4	14	390
864493	6	3	8	77	15	0	0	1	94	2	6	390
869654	5	2	8	69	23	0	0	1	94	2	6	391
874817	5	2	7	70	23	0	0	1	94	3	13	390
879975	5	2	7	71	22	0	0	1	96	3	12	390
885133	5	2	8	69	25	0	0	1	95	4	16	390
890294	4	2	6	66	28	0	0	1	95	4	15	390
895452	6	3	9	84	7	0	0	1	96	4	13	390
900613	5	3	9	81	11	0	0	1	95	3	9	391
905773	6	2	8	73	19	0	0	1	92	3	12	390
910937	5	2	7	61	32	0	0	0	85	5	19	390
916110	6	4	10	78	14	0	0	1	96	3	12	390
921278	5	3	8	88	4	0	0	1	97	4	12	390
926435	5	2	7	64	29	0	0	1	95	3	12	391
931596	4	3	7	62	31	0	0	1	95	2	6	390
936755	5	2	8	81	12	0	0	1	97	3	11	390
941916	5	3	8	75	17	0	0	1	97	3	12	390
947076	5	2	7	64	29	0	0	1	97	3	11	390
952234	6	2	8	73	19	0	0	1	96	2	9	390
957393	4	2	7	63	31	0	0	1	96	2	7	391
962554	4	2	6	61	32	0	0	1	96	2	10	390
967717	4	1	6	59	35	0	0	1	96	4	16	390
1004473	5	3	8	72	21	0	0	1	97	4	17	390
1009634	5	2	7	74	18	0	0	1	97	3	11	390
1014797	5	2	7	72	21	0	0	1	97	1	5	390
1019936	0	2	2	73	25	0	0	1	97	2	6	390
1025112	4	2	6	56	36	0	0	1	96	3	12	390
1030280	0	3	3	67	30	0	0	1	105	9	37	390
1035417	0	2	2	63	15	0	0	1	104	5	21	390
Time	AI8CO	AI8CO2	AI8COX	AI8M1	AI8M2	AI8M3	AI8M4	ID8	MBC8	Q8CH4C	Q8HCLC	TQI812
AVG 340	6	1	7	85	8	0	0	1	97	4	15	340
STD	0	1	1	6	4	0	0	1	0	8	1	4
AVG 390	6	3	9	77	14	0	0	1	95	5	18	390
STD	1	1	2	6	8	0	0	0	0	3	2	6

RUN 11046002

CATALYST 538A

—■— CONVERSION OF CH4

RUN 11046002

CATALYST 538A

RUN 11046002

TEMPERATURE PROFILE

00000568 HOOD #6 RAMPING EXPERIMENT DEFINITION
 00000568 =====

00000568 OPERATOR NAME: B. CRUM DATE: 22-APR-93
 00000568 LOG BOOK #: 11046 PAGE #: 12
 00000568 RUN #: 11046003 CATALYST #: 538B
 00000568 CATALYST NAME: Cu L4/La
 00000568 COMMENTS: PRETREATED OVERNIGHT AT 340 REST SAME AS LAST
 00000568 RUN PLUGGED AND HAD TO BE SHUT OFF AFTER 40 HRS

00000568 MASS OF CATALYST (g): 4.24
 00000568 REACTOR INSIDE DIAM (CM): 0.6
 00000568 CATALYST BED HEIGHT (CM): 25.4

00000413 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6

00000413 =====

00000413 INITIAL FINAL ABSOLUTE ABSOLUTE
 00000413 TEMP TEMP RAMP RATE RAMP RATE
 00000413 (DEG C) (DEG C) (DEG C/MIN) (DEG C/HR)

00000413 =====

00000413 BOTTOM: 340 340 3 0 FLUID VELOCITY 9.0 CM/SEC
 00000413 MIDDLE: 340 340 3 0 RUN TIME 40 HRS
 00000413 TOP : 340 340 3 0

00000413 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00000413 CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 00000413 HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

00000413 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 250

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
5914	3	14	17	65	14	3	0	1	90	18	62	
11135	2	12	14	62	20	4	0	1	82	18	68	
16293	2	10	13	64	20	4	0	1	89	16	61	
21454	2	10	12	64	20	4	0	1	83	17	68	
26613	2	8	11	65	20	4	0	1	84	17	66	
31776	2	8	10	67	21	2	0	1	85	17	66	
36933	2	7	9	67	20	4	0	1	86	16	63	
42154	3	7	9	66	21	3	0	1	89	17	67	
47373	3	6	9	67	20	4	0	1	85	17	67	
52536	3	6	9	69	20	2	0	1	86	17	67	
57756	3	6	9	69	20	3	1	1	96	16	65	
62973	3	6	8	66	20	3	1	1	85	16	66	
68178	3	5	8	68	19	4	0	1	84	17	68	
73352	3	5	8	68	19	4	0	1	86	15	60	
78512	3	5	8	68	20	4	0	1	84	15	63	
83733	3	5	8	68	20	4	0	1	89	18	72	
88957	3	5	8	68	19	4	0	1	85	15	63	
94116	3	5	8	69	19	4	0	1	87	15	60	
99335	3	4	8	69	19	4	0	1	87	16	63	
104491	3	5	8	69	20	3	0	1	86	14	54	
109714	3	4	7	70	19	4	0	1	86	15	63	
114873	4	4	7	70	20	3	0	1	87	15	57	
120035	4	4	8	71	19	2	0	1	87	15	59	
125192	4	4	7	70	20	3	0	1	88	13	52	
130414	4	4	8	70	18	4	0	1	88	11	43	
135574	4	3	7	71	18	4	0	1	88	15	60	
Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI61:
	3	6	9	68	19	3	0	1	87	16	62	
	1	3	2	2	1	1	0	0	3	2	6	

RUN 11046003

CATALYST 538B

RUN 11046003

CATALYST 538B

RUN11046003

CATALYST 538B

Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
405145	2	1	4	81	13	2	0	1	93	8	29	340
410305	2	1	4	82	13	2	0	1	93	8	29	340
415470	3	1	4	82	13	2	0	1	93	8	32	340
420827	3	1	4	82	12	2	0	1	93	1	4	340
425786	2	1	3	82	13	2	0	1	93	8	31	340
430949	3	1	4	82	13	2	0	1	93	9	34	340
436109	3	1	4	82	12	2	0	1	90	7	27	340
441287	3	1	4	81	13	3	0	1	94	7	26	340
446427	3	1	4	84	12	0	0	1	94	8	29	340
451567	3	1	4	82	12	2	0	1	94	5	20	341
456747	2	1	3	81	12	3	0	1	94	6	22	341
461604	3	1	4	82	12	2	0	1	93	6	23	340
467057	3	1	4	80	12	4	0	1	93	8	32	340
472225	3	1	4	81	12	3	0	1	94	8	31	341
477385	2	1	3	78	11	7	0	1	93	8	35	341
482546	4	1	6	80	12	3	0	1	103	10	37	340
487706	2	1	4	83	14	0	0	1	92	7	26	340
492667	3	1	4	82	12	2	0	1	93	5	18	340
498024	2	1	4	82	12	2	0	1	93	7	28	340
503185	2	1	3	81	12	3	0	1	93	8	30	340
508345	2	1	3	81	12	4	0	1	93	8	30	340
513505	2	1	3	77	11	8	0	1	92	8	32	340
518667	3	1	4	84	12	0	0	1	94	4	15	340
523825	2	1	3	79	11	6	0	1	93	6	23	340
528667	3	1	4	84	12	0	0	1	94	6	23	340
534147	3	1	4	84	12	0	0	1	94	8	28	340
539305												
544464	3	1	4	84	12	0	0	1	94	7	25	340
549627												
554769	3	1	4	84	11	0	0	1	94	6	23	340
559947	3	1	3	79	11	7	0	1	93	7	27	340
565109	3	1	4	85	12	0	0	1	94	7	26	340
570296	3	1	4	82	12	3	0	1	93	6	24	340
575425	2	1	4	77	11	8	0	1	93	6	25	340
580567	3	1	4	84	12	0	0	1	94	6	21	340
585747	3	1	4	85	11	0	0	1	93	7	27	340
590910	3	1	4	84	12	0	0	1	94	7	26	340
596087	3	1	4	84	12	0	0	1	94	7	26	339
601231	3	1	4	84	12	0	0	1	94	6	23	340
606391	3	1	4	84	12	0	0	1	94	6	23	340
611549	3	1	4	82	11	2	0	1	92	5	18	340
616709	3	1	4	85	12	0	0	1	94	6	21	340
621866	3	1	4	85	12	0	0	1	94	6	21	340
627029	3	1	4	84	11	0	0	1	94	6	20	340
632185	3	1	4	84	11	0	0	1	94	6	22	340
637348	3	1	4	84	11	0	0	1	94	6	21	340
642507	3	1	4	78	11	7	0	1	94	6	24	340
647665	3	1	4	84	12	0	0	1	94	8	29	340
652826	3	1	4	84	11	0	0	1	94	8	28	340
657967	3	1	4	84	11	0	0	1	94	7	27	340
663144	3	1	4	84	12	0	0	1	94	7	24	340
668309	3	1	4	83	12	0	0	1	94	9	33	340
673567	3	1	4	85	11	0	0	1	94	8	28	340
683726	3	1	4	84	12	0	0	1	94	7	25	340
688866	3	1	4	84	11	0	0	1	94	6	22	340
706626	3	2	5	84	11	0	0	1	96	6	33	340
713785	3	2	6	84	11	0	0	1	96	5	28	340
718948	3	2	5	84	11	0	0	1	96	5	27	340
724109	3	2	5	84	11	0	0	1	96	6	36	340
729296	3	2	5	84	11	0	0	1	96	6	37	340
734444	3	1	4	85	11	0	0	1	96	6	38	341
739605	4	2	6	84	10	0	0	1	96	5	30	341
744763	3	2	6	84	11	0	0	1	96	7	40	340
749921	3	2	5	84	11	0	0	1	96	5	31	340
755084	3	2	5	84	11	0	0	1	96	8	44	340
760242	3	2	5	84	11	0	0	1	96	3	20	340
765401	3	2	5	85	11	0	0	1	96	8	44	341
770563	3	2	5	84	11	0	0	1	97	7	39	340
775727	3	2	5	84	11	0	0	1	96	7	41	340
780885	3	2	5	84	11	0	0	1	95	5	31	340
786045	4	2	6	83	11	0	0	1	96	4	23	341
791203	4	2	6	84	11	0	0	1	97	7	39	340
796363	3	2	5	84	11	0	0	1	97	8	44	340
801521	3	2	5	85	11	0	0	1	96	7	38	341
806682	3	2	5	84	11	0	0	1	97	7	41	340
811845	3	2	5	84	11	0	0	1	97	7	42	340
817006	2	2	4	85	11	0	0	1	97	6	36	340
822162	3	2	5	84	11	0	0	1	96	8	36	340
827323	3	2	5	84	11	0	0	1	96	7	42	339
832482	3	2	5	84	11	0	0	1	96	7	38	340
837643	3	2	5	84	11	0	0	1	96	6	35	340
842802	3	2	5	84	11	0	0	1	96	7	39	340
847964	3	2	5	85	11	0	0	1	96	6	35	339
853122	3	2	5	84	11	0	0	1	96	7	41	340
858287	3	2	5	84	10	0	0	1	96	6	36	340
863442	3	2	5	85	11	0	0	1	96	6	37	340
868603	3	2	5	84	11	0	0	1	96	7	39	340
873744	4	2	6	84	11	0	0	1	96	7	38	341
878927	3	2	5	84	11	0	0	1	97	6	34	340
884085	3	2	5	84	11	0	0	1	96	6	35	340
889243	3	2	5	84	11	0	0	1	96	6	32	340
894402	4	2	5	84	10	0	0	1	96	7	41	340
899548	3	2	5	84	10	0	0	1	96	6	37	340
904722	3	2	5	84	11	0	0	1	96	7	40	340
909896	3	2	5	84	11	0	0	0	96	6	36	340
915044	3	1	4	84	12	0	0	1	95	7	25	340
943002	3	1	4	84	12	0	0	1	94	7	26	340
948162	2	1	3	83	12	2	0	1	94	7	26	341
953322	3	1	4	79	11	8	0	1	94	7	30	340
Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
AVG	3	2	4	81	13	1	0	1	94	8	35	340
STD	1	1	1	4	2	2	0	0	4	3	12	

RUN 11046004

CATALYST 538B

RUN 11046004

CATALYST 538B

—■— SELECTIVITY TO M1

RUN 11046004

CATALYST 538B

00005207 OPERATOR NAME: B CRUM DATE: 21-MAY-93
 00005207 LOG BOOK #: 11046 PAGE #: 18
 00005207 RUN #: 11048008 CATALYST #: 538A
 00005207 CATALYST NAME:
 00005207 COMMENTS: COMPUTER PROBLEM CAUSED LOSS OF DATA AT BEGINING OF RUN
 00005207 GC PROBLEMS CAUSED SOME ADDITIONAL LOSS OF CONVERSION DATA. LONG TERM RUN
 00005207
 00005207 MASS OF CATALYST (g): 2.9
 00005207 REACTOR INSIDE DIAM (CM): 6
 00005207 CATALYST BED HEIGHT (CM): 25.4

00005163 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6
 00005163 -----
 00005163 INITIAL FINAL ABSOLUTE
 00005163 TEMP TEMP RAMP RATE
 00005163 (DEG C) (DEG C) (DEG C / MIN)
 00005163 -----
 00005163 BOTTOM: 340 340 1 FLUID VELOCITY 9.0 CM/SEC
 00005163 MIDDLE: 340 340 1 RUN TIME 290 HRS
 00005163 TOP : 340 340 1
 00005163
 00005163 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00005163
 00005163 CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 00005163 HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0
 00005163
 00005163 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 260

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	M8C6	Q6CH4C	Q6HCLC	TQ612
10527	0	0	0	94	6	0	0	1	98	3	11	340
15702	4	0	4	90	6	0	0	1	101	1	4	340
20631												
25928												
31035												
36133												
41295												
46454												
51552												
56708												
61812												
66917												
72017												
77114												
82217												
87311												
92479												
97632												
102800												
107950												
113121												
118271												
123440												
128598												
133698												
138856												
144004												
149113												
154216												
159316												
164413												
169574												
174674												
179781												
184934												
190102												
195260												
200421												
205579												
210743												
215900												
221052												
226154												
231252												
236354												
241454												
246556												
251653												
256753												
261944												
267104	6	3	9	85	7	0	0	1	93	4	14	340
272262	6	3	9	84	7	0	0	1	92	3	10	341
277422	9	3	12	82	7	0	0	1	7	5	0	340
282549												
441943	4	0	4	89	6	0	0	1	93	5	16	340
447103	5	2	6	86	7	0	0	1	134	-0	-0	340
452263	4	2	6	87	6	0	0	1	91	3	11	341
457424	4	2	6	87	7	0	0	1	91	4	15	340
462584	5	2	7	88	8	0	0	1	92	5	16	340
467741	5	2	7	86	7	0	0	1	92	4	14	340
472895	5	2	6	86	7	0	0	1	91	4	12	340
478098	4	2	6	86	7	0	0	1	105	3	10	340
483285	4	2	6	86	7	0	0	1	96	5	15	340
488485	5	2	7	86	7	0	0	1	94	5	17	339
493705	5	2	8	85	7	0	0	1	108	4	12	340
498942	5	2	7	85	7	0	0	1	109	2	8	340
504161	5	2	7	86	7	0	0	1	98	4	15	340
509384	6	2	8	85	7	0	0	1	120	3	12	340
514603	5	2	7	86	7	0	0	1	90	4	14	340
519823	5	2	8	85	7	0	0	1	90	2	8	340
525042	5	2	7	85	6	0	0	1	90	5	17	340

Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQ612
530265	5	2	8	85	7	0	0	0	1	103	5	18
535484	5	2	8	85	7	0	0	0	1	106		210
540703	5	3	8	85	7	0	0	0	1	112	3	11
545923	5	2	8	86	7	0	0	0	1	95	4	12
551142	5	3	8	85	7	0	0	0	1	119	-0	-0
556364	6	3	8	86	6	0	0	0	1	105	4	13
561586	5	2	8	86	7	0	0	0	1	92	4	14
566803	5	2	8	86	7	0	0	0	1	92	3	10
572023	5	3	8	86	7	0	0	0	1	98	2	7
577246	5	3	8	87	5	0	0	0	1	105	3	10
582465	5	2	7	86	7	0	0	0	1	92	4	14
587681	5	3	8	85	7	0	0	0	1	93	4	13
592907	5	3	8	86	7	0	0	0	1	94	4	14
598126	4	3	7	87	7	0	0	0	1	110	3	11
603346	5	3	8	86	7	0	0	0	1	100	4	12
608563	4	3	7	86	7	0	0	0	1	92	4	14
613783	5	3	8	85	7	0	0	0	1	92	5	17
619005	7	3	10	83	7	0	0	0	1	111	4	12
624207	4	3	7	85	7	0	0	0	1	96	4	12
629444	5	3	8	85	7	0	0	0	1	92	4	12
634667	4	2	5	62	33	0	0	0	1	90	4	17
639886	5	3	8	85	7	0	0	0	1	94	4	12
645103	5	3	8	85	7	0	0	0	1	93	4	14
650323	5	3	8	85	7	0	0	0	1	93	3	10
655545	6	3	8	85	6	0	0	0	1	93		
660767	5	3	8	85	7	0	0	0	1	98	3	11
665986	5	3	8	86	7	0	0	0	1	92	4	12
671202	6	3	8	85	6	0	0	0	1	95	4	13
676424	6	3	8	85	7	0	0	0	1	106	4	14
681646	5	3	8	85	7	0	0	0	1	94	3	10
686868	5	3	7	86	6	0	0	0	1	106	4	14
692083	5	3	8	86	7	0	0	0	1	95	4	13
697303	5	3	8	86	6	0	0	0	1	92	3	11
702463	2	2	4	55	42	0	0	0	1	92	3	16
707682	6	3	9	85	7	0	0	0	1	106	4	12
712902	5	3	8	86	7	0	0	0	1	91	4	13
718124	6	3	8	85	7	0	0	0	1	92	4	13
723347	5	3	8	86	6	0	0	0	1	106	4	12
728563	5	3	8	86	6	0	0	0	1	95	4	12
733783	5	3	8	86	6	0	0	0	1	104	4	12
739002	5	3	8	85	6	0	0	0	1	100	4	13
744224	5	3	7	86	6	0	0	0	1	102	2	5
749426	5	3	8	86	7	0	0	0	1	97	4	13
754665	4	3	7	87	6	0	0	0	1	96	3	11
759883	5	3	8	84	9	0	0	0	1	118	1	3
765043	6	3	9	86	6	0	0	0	1	119	2	6
770187	5	3	8	86	6	0	0	0	1	92		
775421	4	3	7	87	6	0	0	0	1	92	5	16
780642	5	3	8	85	6	0	0	0	1	92	0	1
785850	6	3	8	85	6	0	0	0	1	102	4	13
791067	5	3	8	86	6	0	0	0	1	92	2	6
796289	5	3	8	86	6	0	0	0	1	92	3	9
801521	5	3	8	86	6	0	0	0	1	103	4	14

929982	4	0	4	96	0	0	0	0	1	97	3	10	340
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952902	6	3	9	86	5	0	0	0	1	124	1	5	340
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968065	5	3	8	86	6	0	0	0	1	92	2	8	341
973241	5	3	8	87	6	0	0	0	1	102	2	6	340
978404	5	3	8	86	7	0	0	0	1	106	2	7	340

986726	5	2	7	85	8	0	0	0	1	94	3	9	340
993665	5	3	8	85	7	0	0	0	1	101	1	3	339

Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQ612
AVG	5	2	7	85	7	0	0	0	1	98	3	14
STD	1	1	1	5	5	0	0	0	0	15	1	22

RUN 11046006

CATALYST 538A

—■— CONVERSION OF CH₄

RUN 11046006

CATALYST 538A

Run 11046006

Catalyst 538A

00005143 HOOD #6 RAMPING EXPERIMENT DEFINITION
 00005143 =====
 00005143
 00005143 OPERATOR NAME: B. CRUM DATE: 09-JUN-93
 00005143 LOG BOOK #: 11048 PAGE #: 22
 00005143 RUN #: 11046008 CATALYST #: 538C
 00005143 CATALYST NAME: _____
 00005143 COMMENTS: CONTINUATION OF DESIGNED EXPERIMENT.

00005143
 00005143
 00005143 MASS OF CATALYST (g): 2.93
 00005143 REACTOR INSIDE DIAM (CM): 0.6
 00005143 CATALYST BED HEIGHT (CM): 25.4

00005101 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6

00005101 =====

00005101 INITIAL FINAL ABSOLUTE
 00005101 TEMP TEMP RAMP RATE
 00005101 (DEG C) (DEG C) (DEG C / MIN)

00005101 =====
 00005101 BOTTOM: 340 340 1 FLUID VELOCITY 9.0 CM/SEC
 00005101 MIDDLE: 340 340 1 RUN TIME 160 HRS
 00005101 TOP : 340 340 1

00005101 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00005101 CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8
 00005101 HCl FLOW RATE (CC/MIN): 29.6 AUX FLOW RATE (CC/MIN): 0

00005101 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 250

Time	Al6CO	Al6CO2	Al6COX	Al6M1	Al6M2	Al6M3	Al6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
10795	12	14	26	72	2	0	0	1	98	10	25	
36593	6	2	9	82	10	0	0	1	94	10	34	
52076	6	2	8	80	12	0	0	1	84	7	26	
72654	5	3	8	80	12	0	0	1	83	10	36	
77816	5	3	8	80	12	0	0	1	87	8	27	
88376	6	3	9	79	12	0	0	1	126	2	6	
93537	6	3	8	75	17	0	0	1	103	6	21	
98698												
103893	0	4	4	75	19	2	0	1				
109078	6	3	9	80	11	0	0	1	128	4	12	
114235	7	3	9	79	11	0	0	1	104	4	14	
119454	5	3	8	78	15	0	0	1	88	10	35	
124618	5	3	8	80	12	0	0	1	83	11	39	
145437	4	5	8	71	19	2	0	1	86	10	38	
150656	5	4	9	76	16	0	0	1	86	9	32	
166134	6	3	9	79	12	0	0	1	118	11	38	
171299	0	3	3	85	12	0	0	1	82	8	28	
176515	5	3	8	81	11	0	0	1		5	18	
186894	0	3	3	85	11	0	0	1	84	12	43	
192057	0	3	3	85	12	0	0	1	84	9	32	
197276	0	3	3	82	12	3	0	1	87	9	36	
207600	0	3	3	85	12	0	0	1	84	8	30	
212720	0	3	3	81	16	0	0	1				
217901												
223076	0	3	3	85	11	0	0	1	95	7	26	
228190												

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
238498	0	3	3	84	12	0	0	1	110	4	16	3
243657	0	3	3	85	12	0	0	1	80	8	29	3
248777	0	3	3	80	11	5	0	1				3
253981	0	3	3	86	11	0	0	1	86	7	26	3
259113	0	3	3	85	11	0	0	1				3
264332	0	3	3	84	12	0	0	1				3
269520												
274679												
279859	0	0	0	65	34	0	0	1				0
285028												
290277	0	3	3	91	6	0	0	1	87	7	23	3
295401	0	3	3	85	11	0	0	1				3
300659	0	3	3	86	11	0	0	1	84	9	32	0
305818												
310938	0	3	3	85	11	0	0	1				0
316137												
321253												
328459	0	3	3	86	11	0	0	1	82	7	27	0
331617	0	3	3	85	11	0	0	1	84	8	29	0
336797	0	3	3	91	6	0	0	1	194			0
341960	0	3	3	86	10	0	0	1	188			0
347217	0	3	3	86	11	0	0	1	139	3	12	0
352438	0	3	3	86	11	0	0	1	85	4	13	0
357627	0	3	3	85	10	2	0	1				0
362786												
367937	0	3	3	86	11	0	0	1				0
373139	0	3	3	97	0	0	0	1	92		2	0
378359	0	3	3	87	10	0	0	1	87	4	13	0
383559	0	3	3	86	10	0	0	1				0
388795	0	3	3	87	11	0	0	1	89	6	23	0
389921												
399117												
404261												
409439	0	4	4	86	11	0	0	1	83	8	28	0
414600	0	3	3	87	10	0	0	1	94	5	18	0
419759	0	4	4	83	13	0	0	1	82	8	30	0
424915	0	3	3	97	0	0	0	1	90	7	21	0
430075	0	3	3	86	10	0	0	1				0
435240	0	4	4	84	12	0	0	1	93	2	6	0
440398	0	3	3	87	10	0	0	1	138			0
445529	0	3	3	97	0	0	0	1				0
450775	0	3	3	87	10	0	0	1				0
455918												
461094	0	3	3	97	0	0	0	1				0
466257	0	3	3	87	10	0	0	1				0
471478	0	3	3	87	10	0	0	1				0
476637	0	3	3	87	10	0	0	1				0
481797	0	3	3	86	10	0	0	1				0
487014	0	4	4	84	11	1	0	1				0
492179	0	3	3	87	10	0	0	1				0
497393	0	5	5	78	16	1	0	1				0
502555	0	3	3	87	10	0	0	1				0
507759	0	4	4	86	10	0	0	1	85	7	26	0
512937	0	3	3	87	10	0	0	1	135	1	5	0
518160	0	3	3	87	10	0	0	1	133	5	19	0
523318	0	3	3	87	10	0	0	1	136	1	3	0
528477	0	3	3	88	9	0	0	1	132	2	7	0
533683	0	3	3	87	9	0	0	1	91	6	21	0
538916	0	3	3	87	9	0	0	1	86	4	15	0
544118	0	3	3	87	10	0	0	1	86	4	15	0
549357	0	3	3	80	9	8	0	1	95	4	15	0
554518	0	3	3	87	9	0	0	1	85	5	17	0
559679	0	3	3	80	17	0	0	1	88	5	19	0
564835	0	3	3	97	0	0	0	1	108	4	14	0
570038	0	4	4	95	1	0	0	1	91	8	26	0
575202												
580440	0	3	3	95	2	0	0	1	89	5	18	0
585657	0	3	3	88	9	0	0	1	86	7	24	0
Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI61:
AVG	1	3	5	85	11	0	0	1	100	6	22	
STD	2	1	3	6	5	0	0	0	25	3	10	

RUN 11046008

CATALYST 538C

RUN 11046008

CATALYST 538C

RUN 11046008

CATALYST 538C

00000392 HOOD #6 RAMPING EXPERIMENT DEFINITION

00000392
00000392
00000392

00000392 OPERATOR NAME: B. CRUM DATE: 24-JUN-93

00000392 LOG BOOK #: 11048 PAGE #: 24

00000392 RUN #: 11048009 CATALYST #: 538D

00000392 CATALYST NAME:

00000392 COMMENTS: CONTINUATION OF DES. EXPERIMENT

00000392 NOTE FROM TEMP DATA--RUN PLUGGED AFTER 210 HRS--TEMP DATA CONTINUED FOR 270 HRS

00000392

00000392 MASS OF CATALYST (g): 3.83

00000392 REACTOR INSIDE DIAM (CM): 0.6

00000392 CATALYST BED HEIGHT (CM): 25.4

00000337 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #6

00000337

00000337 INITIAL FINAL ABSOLUTE
00000337 TEMP TEMP RAMP RATE
00000337 (DEG C) (DEG C) (DEG C / MIN)

00000337
00000337 BOTTOM: 340 340 1 FLUID VELOCITY 9.0 CM/SEC
00000337 MIDDLE: 340 340 1 RUN TIME 210 HRS
00000337 TOP : 340 340 1

00000337

00000337 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00000337

00000337 CH4 FLOW RATE (CC/MIN): 100 O2 FLOW RATE (CC/MIN): 22.8

00000337 HCl FLOW RATE (CC/MIN): 29.8 AUX FLOW RATE (CC/MIN): 0

00000337

00000337 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 250

Time	A16CO	A16CO2	A16COX	A16M1	A16M2	A16M3	A16M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQ1612
12142	0	6	6	68	23	3	0	1	85	18	75	340
17284	0	5	5	70	21	4	0	1	73	16	65	340
22445	0	5	5	70	22	4	0	1	121	17	72	340
37921	0	4	4	71	21	3	0	1	70	19	79	340
43078	0	4	4	71	21	3	0	1	70	19	79	340
48242	0	4	4	70	22	3	0	1	70	19	79	341
53404	0	4	4	70	22	3	0	1	70	19	82	340
58566	0	4	4	70	22	3	0	1	71	20	83	339
63725	0	4	4	75	18	3	0	1	81	18	74	340
68885	0	4	4	71	22	3	0	1	69	19	79	340
74043	0	4	4	78	16	2	0	1	84	18	72	340
78204	0	4	4	70	22	3	0	1	70	19	82	340
84366	0	4	4	73	20	3	0	1	102	16	65	340
89525	0	4	4	70	22	3	0	1	65	17	72	340
94686	0	4	4	73	20	3	0	1	81	17	71	340
99844	0	4	4	71	22	3	0	1	72	17	72	340
105005	0	4	4	71	22	3	0	1	73	16	68	340
110165	0	4	4	71	22	3	0	1	73	16	68	340
115324	0	4	4	72	21	4	0	1	68	17	70	341
120478	0	4	4	72	21	3	0	1	73	19	80	341
125704	0	4	4			2	0	1	90	19	65	340
130862	0	4	4	71	22	3	0	1	72	18	77	341
136024	0	4	4	71	22	3	0	1	95	16	68	340
141240	0	7	7	53	28	12	0	1	79	19	93	340
146407												
151558	0	3	3	72	22	3	0	1	69	18	77	340
156725	0	3	3	72	22	3	0	1	69	18	76	340
161876	0	3	3	71	22	3	0	1	71	19	78	340
167037	0	3	3	72	22	3	0	1	70	19	82	340
172200	0	3	3	71	22	3	0	1	69	18	74	341
177357	0	3	3	71	22	3	0	1	71	19	81	340
182519	0	3	3	72	22	3	0	1	71	18	76	340
187686	0	3	3	72	22	3	0	1	98	6	26	340
192836	0	3	3	72	21	3	0	1	72	17	70	340
197996	0	3	3	71	22	3	0	1	72	16	69	340
203158												
208314	0	3	3	72	22	3	0	1	66	12	52	340
213476	0	3	3	73	21	3	0	1	74	17	70	340
218647	0	3	3	72	22	3	0	1	72	19	80	340
223797	0	3	3	72	22	3	0	1	70	19	80	340
228957	0	3	3	73	21	3	0	1	77	19	78	340
234126	0	3	3	76	18	3	0	1	80	18	75	340
239277	0	3	3	73	21	3	0	1	70	18	75	340
244460												
249597	0	3	3	73	21	3	0	1	70	19	78	340
254757	0	3	3	72	22	3	0	1	70	19	81	340
259925	0	3	3	74	20	3	0	1	75	19	79	340
265085									101	18		340
270245	0	3	3	72	22	3	0	1	74	15	64	340
275397	0	3	3	72	22	3	0	1	74	17	71	340
280557	0	3	3	73	21	3	0	1	74	17	70	340
285719	0	3	3	73	21	3	0	1	74	15	65	340
290867	0	3	3	73	21	3	0	1	74	17	72	340
296036	0	3	3	73	21	3	0	1	72	17	73	340
301199	0	3	3	72	22	3	0	1	75	18	75	340

Time	AI6CO	AI6CO2	AI6COX	AI6M1	AI6M2	AI6M3	AI6M4	ID6	MBC6	Q6CH4C	Q6HCLC	TQI612
306367	0	3	3	73	21	3	0	1	79	19	79	340
311514	0	3	3	72	22	3	0	1	71	18	77	340
316686	0	3	3	73	21	3	0	1	82	17	74	340
321847	0	3	3	73	21	3	0	1	78	17	72	340
327005	0	3	3	73	21	3	0	1	104	16	67	340
332187	0	3	3	73	21	3	0	1	71	19	78	340
337387	0	4	4	61	26	9	0	1	88	19	90	340
342545	0	3	3	77	18	2	0	1	86	18	73	340
347704	0	3	3	73	21	3	0	1	89	13	54	340
352866	0	3	3	74	21	3	0	1	74	10	40	341
362166	0	3	3	75	19	3	0	1	82	16	66	340
367319	0	3	3	73	21	3	0	1	73	17	73	340
372486	0	3	3	73	21	3	0	1	73	18	76	340
377645	0	3	3	74	20	3	0	1	74	17	72	340
382806	0	3	3	74	21	3	0	1	72	16	68	340
387964	0	2	2	76	19	3	0	1	77	17	72	340
393126	0	3	3	74	21	3	0	1	71	18	75	340
398279	0	3	3	74	21	3	0	1	71	17	71	340
403462												
408622												
413781												
418941												
424063												
429259												
434422												
439589												
444750												
449903	4	2	7	71	19	3	0	1	78	13	53	341
455063	4	2	6	72	19	3	0	1	74	15	61	341
460222	4	2	7	71	20	3	0	1	73	16	62	340
465381	4	2	6	71	20	3	0	1	72	15	60	340
470517	0	3	3	75	19	3	0	1	97	5	22	340
475683	4	2	6	72	19	3	0	1	85	15	60	340
480837	4	3	6	71	20	3	0	1	71	17	71	340
486022	4	2	7	71	20	3	0	1	71	17	67	340
491181	4	2	6	73	19	3	0	1	91	16	64	340
496324	4	2	7	73	18	2	0	1	75	18	71	340
501484	4	2	6	72	19	3	0	1	70	18	71	340
506636	0	2	2	75	20	3	0	1	92	10	42	340
511805	4	2	7	75	16	2	0	1	79	17	66	340
516966	4	2	6	71	20	3	0	1	70	17	66	340
522124	4	2	6	72	19	3	0	1	74	17	67	340
527299	4	2	6	74	17	2	0	1	76	13	51	340
532446	4	2	6	72	19	3	0	1	75	16	63	340
539661	4	2	6	72	19	3	0	1	73	15	61	340
545094												
550263	4	2	6	71	19	3	0	1	74	16	64	340
555441	4	2	6	74	17	2	0	1	78	15	57	340
560603	4	2	7	72	19	3	0	1	75	15	61	340
565762	4	2	6	71	20	3	0	1	72	16	64	340
570904	4	2	6	74	17	2	0	1	79	16	64	341
576064	4	2	6	72	19	3	0	1	71	18	71	340
581242	4	2	6	72	19	3	0	1	70	16	64	340
586401	4	2	6	72	19	3	0	1	70	17	70	341
596722	4	2	6	72	19	3	0	1	70	15	60	340
601881	3		3					1	94	16	53	340
607043	4	2	6	74	18	2	0	1	75	17	66	340
612200	4	2	6	72	19	3	0	1	72	14	55	340
617363	4	2	6	73	19	2	0	1	73	15	59	340
627680	5	2	7	71	19	3	0	1	73	8	33	340
632843	4	2	6	72	19	2	0	1	72	16	64	340
637985	4	2	6	72	19	3	0	1	73	15	60	340
643145	4	2	6	73	18	2	0	1	73	16	62	340
648323	4	2	6	73	19	2	0	1	72	16	64	341
653482	4	2	6	73	18	2	0	1	72	16	61	340
658641	4	2	6	72	19	3	0	1	76	17	66	341
663801	4	2	6	72	19	2	0	1	72	17	66	340
668963	4	2	6	72	19	2	0	1	72	17	67	340
674119								1	99	18	0	340
679283	4	2	6	73	18	2	0	1	71	17	66	340
684442	4	2	6	73	18	2	0	1	71	17	65	340
689602								1	82	18	0	340
694759	4	2	6	72	19	3	0	1	84	14	56	340
699921	4	2	6	73	18	2	0	1	96	12	46	341
705080	4	2	7	72	19	2	0	1	77	14	55	340
710302	2	5	7	51	30	12	0	1	73	13	62	340
715519	2	6	8	52	29	11	0	1	77	12	56	340
720681	4	2	6	73	18	2	0	1	77	16	62	340
725841	4	2	6	72	19	2	0	1	96	9	37	340
731003	4	2	6	73	18	2	0	1	66	17	66	341
741364	4	2	6	73	18	2	0	1	66	15	61	340
746541	4	2	6	73	19	2	0	1	62	14	56	340
AVG	2	3	5	72	20	3	0	1	77	16	66	340
STD	2	1	2	4	2	1	0	0	9	3	14	

RUN 11046009

CATALYST 538D

RUN 11046009

CATALYST 538D

RUN 11046009

CATALYST 538D

HOOD 7 PERFORMANCE SUMMARY

23 APRIL 93 TO 26 APRIL 93

RUN 9521036

****NOTE****

THIS CATALYST BED WAS NOT FRESH WHEN RUN
3 RUNS HAD BEEN STARTED AND STOPPED BEFORE THIS RUN

****NOTE****

METAL HC REACTOR 1/4" OD
CAT 513
G 0.63 G
TEMP 316-325 OVEN TEMP
BED HT 10.2 CM
ID 0.46 CM

PRESSURE 30 PSIG
FLOWS ML/MIN
CH4 80
O2 18.3
HCL 23.7

ABSORBER
SOLVENT MULTITHERM 503
SOLVENT TEMP IN 4-16 C ??
MLS/MIN 4.5 AND 2.6
PACKING 1/8" RR
HEIGHT 24 INCHES
CD:PD 5:1
PRESSURE 30
L/G 32 AND 18.7

RUN DATA	CONV	M1	M2	CO	CO2	COX	ABSORBER DATA GM1/GSOLVENT	%M1 REMOVED %	SOL FLOW ML/MIN	NOTES
	12	81	8	8	5	13	3.33E-03	74		4.5 SOL FLOW 4.5 OVEN TEMP 320
	13	70	6	18	6	24	3.12E-03	74		4.5
	29	69	10	14	7	21	8.26E-03	92		4.5
	29	69	11	13	7	21	1.34E-02	85		2.6 SOL TEMP DOWN 4C SOL FLOW 2.6
	28	69	9	14	8	21	1.28E-02	84		2.6
	25	71	8	12	8	21	1.28E-02	91		2.6
	8	77	0	16	7	23	3.96E-03	78		2.6
	9	75	0	17	8	25	2.78E-03	54		2.6
	8	75	0	17	8	25	2.90E-03	61		2.6
	12	71	15	9	5	15	5.39E-03	82		2.6 OVEN TEMP 325
	11	78	0	15	7	22	4.72E-03	70		2.6
	10	77	0	16	7	23	3.91E-03	67		2.6
	10	76	0	17	8	24	3.84E-03	65		2.6
	10	76	0	17	8	24	3.63E-03	62		2.6
	10	78	0	15	7	22	3.90E-03	63		2.6
	10	76	0	16	8	24	3.71E-03	60		2.6
	10	78	0	15	7	22	3.75E-03	63		2.6
	10	80	0	12	8	20	4.16E-03	65		2.6
	9	76	0	16	7	24	3.50E-03	61		2.6
	9	77	0	16	7	23	3.46E-03	64		2.6
	8	76	0	16	8	24	2.98E-03	59		2.6
	10	78	0	15	7	22	3.74E-03	60		2.6
	10	77	0	16	7	23	3.74E-03	63		2.6
	10	76	0	16	8	24	3.97E-03	63		2.6
	10	76	0	16	8	24	3.83E-03	61		2.6
AVG AND STD CONVERSION		M1	M2	CO	CO2	COX	ABSORBER DATA GM1/GSOLVENT	%M1 REMOVED		
	13	75	3	15	7	22	5.02E-03	69		
	7	3	4	2	1	3	3.12E-03	10		

HOOD 7 PERFORMANCE SUMMARY

5 MAY 93 TO 13 MAY 93

****NOTE****

SYSTEM RAN FOR 36 HRS THEN PLUGGED. NOT SURE DATA COLLECTED AT STEADY STATE

****NOTE****

RUN 11202002

METAL HC REACTOR 1/4"OD

CAT 513

G 0.61 G

TEMP 310-320 OVEN TEMP

BED HT 10.2 CM

RX ID 0.48 CM

PRESSURE 30 TO 40 PSIG

FLOWS ML/MIN

CH4 80

O2 18.3

HCL 23.7

ABSORBER

SOLVENT MULTITHERM 503

SOLVENT TEMP IN 4-16 C ??

MLS/MIN 4.5 AND 2.6

PACKING 1/8" RR

HEIGHT 24 INCHES

CD:PD 5:1

PRESSURE 30 TO 40

L/G VARIED

RUN DATA

CO4V M1 M2 CO CO2 COX

8	93	0	0	7	7
9	85	0	9	8	15
10	86	0	7	7	14
10	83	0	10	7	17
10	84	0	9	7	16
10	84	0	10	7	16
10	85	0	8	7	15
9	84	0	8	7	16
10	84	0	9	7	16
9	85	0	8	7	15
10	84	0	9	7	16
13	77	6	10	7	17
13	77	5	11	7	18
9	89	6	16	9	25
12	75	5	12	8	20
13	76	6	11	8	18
13	80	6	7	7	14
13	77	6	11	7	18
13	76	5	11	7	18
28	54	12	21	12	33
29	67	8	17	8	25
28	46	13	27	14	42
29	66	9	16	8	25
28	66	9	16	9	25
28	65	9	17	9	26
27	64	7	20	9	29
25	61	8	23	9	32
21	80	6	5	9	14
9	83	0	12	4	17
11	85	0	11	5	15
11	84	0	11	5	16
11	80	0	14	6	20
11	75	0	17	8	25
12	73	1	18	9	26
12	71	1	19	9	28
12	75	1	14	10	24
14	71	1	19	9	28

ABSORBER DATA

GM1/GSOLVENT

%M1 REMOVED

2.76E-03	38
3.83E-03	49
4.27E-03	51
3.99E-03	51
4.21E-03	52
4.43E-03	56
4.70E-03	58
4.04E-03	52
4.73E-03	57
4.11E-03	54
4.01E-03	48
4.34E-03	30
4.56E-03	31
4.06E-04	5 *
3.45E-03	28
4.18E-03	29
3.71E-03	24
4.87E-03	33
6.39E-03	43
1.19E-03	5 *
7.08E-04	4 *
1.76E-02	63
1.71E-02	63
1.65E-02	63
0.00E+00	90
0.00E+00	53
0.00E+00	85
5.49E-03	50
6.17E-04	77
7.01E-03	77
5.58E-03	65
4.96E-03	60
4.83E-03	59
4.04E-03	48
4.70E-03	50

SOL FLOW NOTES

2.1 RX TEMP 315 SYSTEM PRESSURE 30#

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RUN DATA

CONV M1 M2 CO CO2 COX

AVG	15	76	4	13	8	21
STD	8	11	4	5	2	7

ABSORBER DATA

GM1/GSOLVENT

%M1 REMOVED

4.76E-03	53
4.17E-03	13

*DATA NOT IN AVG OR STD CALCULATION

Data Appendix B

Catalyst Synthesis and Composition Information

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 impregnation overnight. While heating the solvent removed under vacuum.
PRETREATMENT	Under vacuum for 3 hours at 350 °C. In the beginning color of the catalyst was orange but as the solvent being removed and the temperature was increased following color changes took place. Orange to yellow black to yellow to olive green to gray.
COLOR	Final color is gray. In water it becomes almost black later dark olive green.

CATALYST 538A

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$[\text{Cu}(\text{CH}_3\text{CN})_4][\text{PF}_6]$. 9947 lab-book page 91.
LOADING	2.5% Cu and 1.0% P.
IMPREGNATION METHOD	The support was heated at 400 C under full vacuum for 2 hours. Next the solution of the complex was made up in nitrogen (completely clear solution, no undissolved solid) and was added under nitrogen to the support. The suspension was left overnight at room temperature. The next day the solvent was removed by nitrogen flow.
PRETREATMENT	Under vacuum for 2 hours at 350 °C.
COLOR	Gray.

CATALYST 538B

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$[\text{Cu}(\text{CH}_3\text{CN})_4]\{\text{PF}_6\}$ and LaCl_3 . 9947 lab-book page 91.
LOADING	1.8% Cu, 0.65% P, 6.9% Cl, and 10.2% La.
IMPREGNATION METHOD	This catalyst is the same as 538A but it has been promoted with LaCl_3 . The LaCl_3 (anhydrous) was dissolved in distilled water and heat was used to enhance solubility. Suspension was gravity filtered to obtain a clear solution. Water was removed at 100 °C with flowing nitrogen.
PRETREATMENT	Under vacuum for 3 hours at 330 °C.
COLOR	Yellow.

CATALYST 538C

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE	$[\text{Cu}(\text{CH}_3\text{CN})_4]\{\text{PF}_6\}$ and LiCl. 9947 lab-book page 91.
LOADING	2.5% Cu, 1.3% Li and 1.0% P.
IMPREGNATION METHOD	This catalyst is the same as 538A, but it has been promoted with LiCl. The LiCl was dissolved in water, Catalyst 538A was impregnated overnight with the LiCl solution. After impregnation, the water was removed in flowing nitrogen.
PRETREATMENT	Under vacuum for 2 hours at 350 °C.
COLOR	Pale green blue.

CATALYST 538D

SUPPORT	Silica CS-1040, PQ Corp. Lot #1229-2201
SOURCE 91.	$[\text{Cu}(\text{CH}_3\text{CN})_4]\{\text{PF}_6\}$, LiCl and LaCl_3 . 9947 lab-book page
LOADING	2.5% Cu, 0.7% Li, 13.5% La and 0.5% P.
IMPREGNATION METHOD	This catalyst is the same as 538A , but it has been promoted with LiCl and LaCl_3 .
PRETREATMENT	Under vacuum for 2 hours at 350 °C.
COLOR	Gray.

DATE

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END