Methyl Chloride Via Oxyhydrochlorination of Methane

Quarterly Technical Progress Report No.10
For the period January 1994-March 1994

August 1994

Contractor

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Prepared for the

U.S. Department of Energy
Pittsburgh Energy Technology Center

Contract No. DE-AC22-91PC91030

and the

Gas Research Institute

Contract No. 5091-222-2300

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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

In work related to the design and construction of the PDU this quarter involved further detail design and a real start to the construction activities. Status updates are given below for each discipline in the Task 2.0 and 3.0 headings. This work is progressing well, with the caveat of several small slips in the scheduling.

On the catalyst development front this quarter was extremely productive. Many catalyst screening experiments were completed and they showed that control of the reaction exotherm is going to be quite challenging under PDU conditions. The presence of much more efficient reactor design and the ability to maintain closer to isothermal conditions is expected to give a significant advantage in actual PDU operation. A major concern at the moment is the cost of La in the catalyst being used. An action plan to remedy this is being put together.

Technical Results

Task 1. Catalyst Definition and Separation Unit Operation Development.

Catalyst Definition -

The primary goal of this quarter's catalyst definition research involved the screening of some of the many catalysts that have been synthesized recently. Reactant gas flow rates and catalyst bed heights were also varied to optimize the conversion/selectivity being measured. Finally, it was discovered that a reaction that was exotherming, forming oxygenates, could be controlled by adjusting the N_2 and/or O_2 flows.

Data Set 1 is the screening of catalyst 543, an attempt to use filtering during impregnation to leave only tightly bound metals on the support. This run was uneventful and was stopped due to lack of reactivity probably due to low metals content (0.157% Cu).

Data Set 2 shows an attempt to greatly increase the reactant gas velocities using catalyst 540. However, only very small conversions were seen. The increased velocities gave too short of a residence time for the OHC reaction.

Data Set 3 again used catalyst 540, but decreased the gas flows to half of those used in the previous run. This gave a good conversion/selectivity of 12/91 at an oven set point of 315°C. However, upon boosting the set point 5°C, the activation barrier to oxygenation was overcome, and the reaction exothermed as shown on page 5 of Data Set 3.

Data Set 4 represents an attempt to increase the catalyst bed height to 30.5 cm and increase the gas velocities to give the same residence time as in Data Set 3. Problems with the online GC

caused faulty conversion/selectivity data, but page 2 of Data Set 4 again shows the reaction exotherming when the oven set point was raised to 320°C.

Data Set 5 is a repeat of the previous run, but the conversion/selectivity data is faulty due to a failure to separate the CO and CH₄ peaks.

Data Set 6 is a screening run of catalyst 544, a one-step aqueous impregnation of Cu, La, and Li into alumina. GC problems again ruined the data after the first two points. However, the first two points plus the temperature measurements shown on page 2 of Data Set 6 show that this catalyst was exotherming from the beginning even with the oven set at 315°C.

Data Set 7 is an attempt to control the exotherming using catalyst 544. However, a plug in the reactor nullified this data.

Data Set 8 shows the screening of catalyst 546, an attempt to lower the amount of La in the catalyst. With the exception of three erroneous points, little activity was observed.

Data Set 9 is the screening run for catalyst 541, substituting K for Li in the catalyst. However, this catalyst had a small Cu content, 0.239%, and did not show significant activity until combustion began. This run shows the first attempt at bringing a combustion situation under control. This was accomplished by two means: first, by adding additional nitrogen and thereby diluting the gaseous reactants while decreasing residence time over the catalyst and second, by decreasing the oxygen feed until catalyst bed temperatures lowered to reasonable levels. It is obvious that these steps had a dramatic effect on the system. Upon entering the combustion regime, an average of 15% conversion and 53% selectivity was observed. By adjusting the feed flows, a conversion/selectivity of 9%/86% was achieved.

Data Set 10 shows an attempt to measure catalyst 544, aqueous one-step catalyst, under controlled conditions. However, combustion again began before conversions over 5% were gained. At an oven set point of 310°C, 14%/57% con/sel is seen. Encouragingly though, at 320°C, a higher temperature where more combustion would be inspected, con/sel of 16%/70% was achieved with the addition of nitrogen to the feed stream.

Data Set 11 shows the beginning of an experiment to probe the effect of varying Cu metal content within the catalyst. Catalyst 550C, an aqueous one-step catalyst with 2.34% Cu, started with combustion at a 315°C oven set point. Interestingly, the amount of combustion did not increase with a 5°C boost in temperature. This is explained by the fact that the oxygen flow was accidentally set at 40 cc/min instead of 54 cc/min. It is assumed that this reaction is in fact converting all available oxygen during combustion at 315°C. That is why no further combustion is seen by increasing to 320°C.

Data Set 12 represents screening of catalyst 551, an attempt to replace La with Th in the catalyst. This was attempted because of the prohibitive cost of La. However, conversions above 5% were not achieved before combustion began.

Data Set 13 continues to probe the importance of Cu content. This run used catalyst 550A, an aqueous one-step catalyst with 0.627% Cu. Controlled data without combustion was gathered up to a 315°C oven set point.

Data Set 14 uses catalyst 550B, an aqueous one-step catalyst with 1.26% Cu. Controlled data with slightly higher conversions than catalyst 550A was measured at 305 and 310°C. Combustion set in at 315°C. Also note the temperature profiles shown on page 5 of Data Set 14. For this reaction, a second thermocouple was wired to the outside of the reactor. Having two thermocouples shows the presence of a thermal wave beginning at about 17 hours. As the wave passes thermocouple #1, thermocouple #2 begins to feel its effects.

Data Set 15 is another attempt to screen catalyst 550C. This time controlled data was measured at 305°C before combustion began at 310°C. Page 5 of Data Set 15 again shows some very interesting thermal events occurring inside the catalyst bed.

Data Set 16 shows the final probe of Cu content using catalyst 550D, an aqueous one-step catalyst with 4.05% Cu. This run was shut down quickly after observing that even the first point measured at 305°C already showed combustion.

The many catalyst screening experiments run this quarter have shown us many useful pieces of information while, of course, showing new research avenues to be pursued. Many catalysts under varying conditions showed that combustion is easy to achieve and sometimes hard to avoid. However, adjusting reactant gas flows did help to realign the reactor to more favorable conditions. The cost of La for our catalyst is still a problem that needs to be addressed. So far replacing or lowering the La content has been unsuccessful.

The startup of the OHC PDU draws near quickly. This necessitates that certain questions be answered soon. These include: deactivation/regeneration of the catalyst, confirming correlation between Cu crystallite size and methane conversion, and further analysis of catalyst 513, a Cu/Li catalyst that achieves high conversions in the absence of La.

Task 2.0 PDU Design Engineering and Task 3.0 PDU Construction and Procurement

Work this quarter involved detail design of the PDU and the beginning of construction. Status updates are given below for each discipline.

PROCESS ENGINEERING

A HAZOP session was held to look at the necessities of oxygen analyzers at the reactor inlet and outlet. The HAZOP recommendation was not that oxygen analyzers are or are not necessary, but that we need to assure that the mixtures of gases in the reactor are not explosive at any temperature or pressure we operate. The critical issues, then, were to find out what these explosive limits are and what they become under temperature and pressure. A literature search revealed two bulletins from the US Department of the Interior. Also, Chilworth Technology was contacted to do some flammability testing of OHC mixtures. Dow Corning has worked CTI in the past on these types of studies.

A sample of water with chlorocarbons was prepared and characterized by SMI. The amounts of chlorocarbons were reported to waste treatment. The quantities of methylene chloride and chloroform were a concern for the waste water treatment process, but since these are "worst case" and the WWTP Air Stripper will undergo a packing clean-up/re-pack which should increase its efficiency, no further action was taken except that WWTP requested that we sample the PDU waste water before discharge to the Chemsewer.

A feasibility study was completed by the vendor using a stream representing our reactor effluent. The report basically stated that FTIR process monitoring should not be a problem. The purchase order for the FTIR analyzer was negotiated and placed. Items included for this order are a KVB-Analect PCM-5000 FTIR, teflon coating for optibus components, Spectranet LAN cable, matrix development services, a spectrometer support kit, a spare set of ZnSe windows and Kalrez o-rings for the sample cell, and installation and training. Quite a bit of time was spent this quarter supporting the vendor's design of the analyzer.

Programming of our CAMILE data acquisition and control system continued. The programming was put on hold during January due to other priorities, however, the configuration restarted in earnest in February. This work included configuration work, the writing of several master sequences and some calculation blocks. The reactor temperature control scheme was outlined (cascade and split-range control), and some calculations for the reactor conversions and product selectivities were set up. However, the delivery of the CAMILE was delayed due to our concerns over the license contract. The legal department has found some statements in the contract that we do not usually find acceptable for purchases of this nature. We worked with the manufacturer (Dow Chemical) to find an agreement. The delayed delivery was expected to be only month at the end of the quarter, however, was not going to effect the project schedule.

A great deal of time was spent supporting equipment design, and instrument selection and data sheet preparation activities. Also, a lot of work was done reviewing the detail engineering output. For example, the phase separators (3) and storage vessels (2) were changed to glass lined instead of PFA lined. Glass lining will be less permeable to the chemicals involved and therefore more corrosion resistant. Also glass lining was cheaper. The delivery times for the vessels are also very favorable.

CIVIL ENGINEERING

Design activities for this discipline was completed at the end of quarter 4, '93. Some follow-up work and some revisions were completed this quarter.

All structural steel upgrades were made this quarter. The PDU is being installed in an existing process tower that used to house a defunct process which was demolished during quarter 3 of '93. The structural steel upgrade work primarily involved installing miscellaneous steel required to support the equipment. Other work done included repairs to the existing grating, the fabrication and installation of drip pans, equipment supports, and a platform for the reactor. This work was finished on budget with an engineering hour increase of roughly 20% due to revisions. This work was completed one week late due to the largest snow storm in Kentucky's history.

MECHANICAL ENGINEERING

The majority of the formal detail design activities for this discipline were completed this quarter.

Equipment design and specification work was completed this quarter and purchase orders were made. Several items of equipment were purchased at prices lower than what was allowed for in the estimate. In particular, substantial savings (approximately \$35M) were made as a result of choosing to use glass lining in all of our lined vessels which were originally planned to be lined with PFA Teflon. Glass lining will be less permeable to the chemicals involved and therefore more corrosion resistant. Other procurement activities this quarter involved the start of valve and piping procurement as well as other miscellaneous mechanical items.

Detailed piping isometrics were completed and the mechanical work package was delivered to our mechanical partner of February 16. Some of the piping design was done by Dow Corning personnel, however, a large portion of it was done by the construction superintendent from our mechanical partner. His input during the design was important in incorporating construction expertise in the design (we call that "constructability" information).

After several iterations, we reconciled our estimates with our mechanical partner's estimate of the mechanical work to be completed. This consisted of equipment installation, piping fabrication and installation, instrument setting and hook-up, pressure testing and clean-out. On March 22, we then agreed upon a target price for which the work was to be done. We established a bonus incentive for the contractor where we will share any money saved under the target price.

The agreement had an incremented scale and was "capped" at a maximum bonus amount. Similarly, we agreed to share any overage on an incremented scale up to a capped amount.

ELECTRICAL ENGINEERING

The electrical construction package was completed this quarter and submitted to our electrical and instrumentation (E&I) partner to prepare a "target" price estimate. Our E&I partner was involved in the preparation of the package and provided constructability input to the design. The design consisted of wiring diagrams for four motors and one electrical heater with a SCR controller and heat trace isometrics. Our E&I partner updated existing cable tray routings and schedules and helped update drawings for construction.

INSTRUMENTATION ENGINEERING

Instrumentation design continued this quarter. Several delays were experienced during this part of the design due to personnel resource limitations. The majority of the design was completed. This included junction and marshaling panel drawings, instrument block diagrams, instrument termination schedule (used in place of loop drawings), and the control room layout. At the end of the quarter, instrument data sheet and instrument requisition work was still in progress. An instrument construction package was completed and submitted to our E&I partner for a "target" price estimate.

CONSTRUCTION

The structural steel work was completed this quarter.

The two major mechanical activities for the quarter were equipment installation and carbon steel piping. The work got started by first installing some carbon steel pipe that was to be located behind some process equipment. After the pipe was installed, several pieces of equipment were installed. Some miscellaneous support steel had to be reworked due to some miscommunication in foot print details and bolt hole patterns on some equipment. Equipment installation work was delayed this quarter due to some late equipment deliveries. Work crews were kept low since the work was limited by the rate at which equipment was being delivered.

At the end of the quarter all of the carbon steel process pipe had been fabricated, delivered to the site, and painted. Except for PFA, all lined pipe, valves, and fittings had been ordered.

SAFETY

There were no safety incidents for the project this quarter.

BUDGET

At the end of the quarter we finished reconciling estimates with our mechanical contractor partner and agreed on a target price. Mechanical material and labor was projected at \$490 M versus our original estimate, including contingency of \$430 M. We suspect the difference in price may be mainly due to inaccuracies in our estimating. Minor scope changes have also added to the price. The current cost forecast for the project was \$2.498 MM, including \$185 M in assigned reserve contingency.

SCOPE

The following scope changes were made this quarter.

1. Column differential pressure indicators were added to absorber and stripper columns.
2. Change column temperature probes from remote to local indicators to save money.
3. Welding outlets eliminated to save money and were considered unnecessary.
4. Filter deleted to save money and four strainers were added to protect equipment and instruments.
5. Tubing additions for FTIR analyzer.
6. Sample point additions.
7. Stripper vent pressure control valve (PCV) bypass line added to facilitate pressure bleed down due to small size of PCV.
8. Analyzer shed upgrades. Decided to house FTIR analyzer due to sensitive nature of the analyzer and to facilitate maintenance.
9. Climate control addition to analyzer shed.
10. Methane relief valve relocation due to physical location of tube trailers to process unit.
11. Elimination of reactor feed oxygen analyzer and replacement with a Coriolis mass meter.
12. Change the absorber vent line outlet flow meter from an orifice plate meter to a Coriolis mass meter.
13. Addition of an audible alarm in process area.
14. Vapor sample point addition.

Many of these changes are the normal kinds of changes that occur during detailed design. However, all changes were documented in order to facilitate communication and also to diligently control change to the project. Note that most of these changes were made before the mechanical package was issued. Our mechanical partner assured us that they could work changes into the project during construction.

SCHEDULE

We worked hard this quarter to help make up time on the schedule. This was necessary due to an error that was made when the schedule was prepared for the process development package (PDP). This error involved using one third the amount of engineering time required and that was budgeted for the project. In order to help meet our project completion date, and at the recommendation of our mechanical partner, we decided to start construction early this quarter.

Partnering played a significant role in saving time during construction. For example, carbon steel pipe fabrication, which was included in the "target price agreement", started in our mechanical contractor's shop on February 4. This was almost two weeks before the mechanical work package was completed and six weeks before a target price was agreed to. Equipment and piping installation began on February 21, only five days after our mechanical work package was completed. We believe that through partnering, we saved at least three months on the overall project schedule. One month was saved in using a simplified construction package tailored to our partners needs. Also, one month was saved in skipping the bid package review step that is done for traditional "lump-sum not to exceed" type jobs. Finally, at least one month was saved by starting construction before all the equipment had arrived.

Construction was slowed somewhat, however, due to equipment delivery dates being scheduled by the vendors later than hoped. Also, several items were shipped late. In particular, we had several problems with the gas lined equipment not passing their spark tests indicating flaws in the lining. Delays in setting this equipment also delayed the beginning of other piping activities since the contractor was unable to get the final field measurements for piping runs. These measurements were needed to fabricate the pipe. We took this more conservative approach of pipe fabrication in order to hold down costs which result from rework.

PROJECT COORDINATION

A weekly construction review meeting was initiated where the mechanical partner representatives, Dow Corning construction inspection and scheduling representatives, engineering and project management met to discuss construction progress and future direction. These meetings proved to be very fruitful in facilitating communication.

A project luncheon was held where everyone involved with the project attended. This included representatives from research, engineering, construction scheduling and inspection, mechanical partner, E&I partner, safety, startup team members, Safety Audit Team chairman, and key managers from three different departments. To help build team spirit, a photograph of the team was made and pictures distributed to each team member.

STARTUP

Three startup team members were identified this quarter and some initial startup planning was started.

Data Appendix A
Experimental Data Sets

J0007153 OPERATOR NAME: B. CRUM DATE: 05-JAN-84
J0007153 LOG BOOK #: 11046 PAGE #: 71
J0007153 RUN #: 11048030 CATALYST #: 543
00007153 CATALYST NAME: CULALU ON ALUMINA
00007153 COMMENTS: REMAKE OF CAT #40 BUT CAT WAS FILTERED
00007153
00007153
00007153 MASS OF CATALYST (g): 2.44
00007153 REACTOR INSIDE DIAM (CM): 0.46
00007153 CATALYST BED HEIGHT (CM): 19.7

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00007040      -----
00007040      INITIAL      FINAL      ABSOLUTE
00007040      TEMP          TEMP          RAMP RATE
00007040      (DEG C)      (DEG C)      (DEG C / MIN)
00007040      -----
00007040      BOTTOM: 310      320      0.5
00007040      MIDDLE: 310      320      0.5
00007040      TOP : 310      320      0.5

```

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

CATALYST RUN WAS DISCONTINUED DUE TO LACK OF REACTIVITY.

RUN 11046030

CAT #543

00004186 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00004186
 00004186 OPERATOR NAME: B. CRUM DATE: 23-FEB-94
 00004186 LOG BOOK #: 11202 PAGE #: 74
 00004186 RUN #: 11202023 CATALYST #: 540
 00004186 CATALYST NAME: Cu/La/LI ON ALUMINA
 00004186 COMMENTS: Attempt to repeat run 11046027 at these flows.

00004186
 00004186 MASS OF CATALYST (g): 4.11
 00004186 REACTOR INSIDE DIAM (CM): 0.45
 00004186 CATALYST BED HEIGHT (CM): 19.7

00003957 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00003957
 00003957 INITIAL FINAL ABSOLUTE
 00003957 TEMP TEMP RAMP RATE
 00003957 (DEG C) (DEG C) (DEG C / MIN)
 00003957
 00003957 BOTTOM: 310 315 1 RUN TIME 24 HRS
 00003957 MIDDLE: 310 315 1 SPACE VELOCITY 50.5 CM/SEC
 00003957 TOP : 310 315 1

00003957 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00003957 CH4 FLOW RATE (CC/MIN): 450 O2 FLOW RATE (CC/MIN): 75
 00003957 HCl FLOW RATE (CC/MIN): 97.5 AUX FLOW RATE (CC/MIN): 0

00003967

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 12														
Time	A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712	
14573	0	0	0	100	0	0	0	0	1	111	2	9	8	315
19671	0	0	0	13	87	0	0	0	1	103	-2	-21	3	315
24771	0	0	0	7	93	0	0	0	1	96	4	36	10	315
29871	0	0	0	8	92	0	0	0	1	98	-10	-85	-2	315
34971	0	0	0	9	91	0	0	0	1	98	4	37	12	315
40071	0	0	0	11	89	0	0	0	1	100	2	14	10	315
45171	0	0	0	10	90	0	0	0	1	100	-2	-19	4	315
50271	0	0	0	9	91	0	0	0	2	98	5	42	11	317
56082	0	0	0	13	87	0	0	0	2	99	3	25	11	320
61191	0	0	0	11	89	0	0	0	2	97	4	34	12	321
66290	0	0	0	14	86	0	0	0	2	99	4	31	12	320
71393	0	0	0	13	87	0	0	0	2	98	5	43	13	321
76493	0	0	0	15	85	0	0	0	2	99	5	46	14	320
81594	0	0	0	12	88	0	0	0	2	97	3	25	12	340
86714	27	0	27	23	50	0	0	0	2	107	18	103	100	341
91873	24	1	24	26	50	0	0	0	2	108	19	106	100	341
AVERAGES A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712		
AVG 315	0	0	0	21	79	0	0	0	1	100	0	2	7	315
STD	0	0	0	30	30	0	0	0	0	4	5	40	4	
AVG 320	0	0	0	13	87	0	0	0	2	98	4	34	12	320
STD	0	0	0	1	1	0	0	0	0	1	1	8	1	
AVG 340	25	0	26	24	50	0	0	0	2	108	19	106	100	340
STD	2	0	1	1	0	0	0	0	0	1	0	3	0	

*****NOTE***** SELECTIVITY NUMBERS ARE SUSPECT DUE TO BAD CHROMOSORB COLUMN. CONVERSION NUMBERS ARE GOOD.*****

RUN 11202023

CAT 540

RUN 11202023
CAT 540

00004216 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00004216
 00004216
 00004216 OPERATOR NAME: B. CRUM DATE: 24-FEB-94
 00004216 LOG BOOK #: 11202 PAGE #: 76
 00004216 RUN #: 11202024 CATALYST #: 540
 00004216 CATALYST NAME:
 00004216 COMMENTS: Using half flows of 11202023 to look at effect of s.v.
 00004216
 00004216
 00004216 MASS OF CATALYST (g): 3.63
 00004216 REACTOR INSIDE DIAM (CM): 0.45
 00004216 CATALYST BED HEIGHT (CM): 19.7

00004211 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00004211
 00004211 INITIAL FINAL ABSOLUTE
 00004211 TEMP TEMP RAMP RATE
 00004211 (DEG C) (DEG C) (DEG C / MIN)
 00004211
 00004211 BOTTOM: 315 320 1
 00004211 MIDDLE: 315 320 1
 00004211 TOP : 315 320 1

00004211
 00004211 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00004211

00004211 CH4 FLOW RATE (CC/MIN): 225 O2 FLOW RATE (CC/MIN): 37.5
 00004211 HCl FLOW RATE (CC/MIN): 48 AUX FLOW RATE (CC/MIN): 0

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
16823	0	0	0	86	14	0	0	1	112	11	60	28	315
21725	0	0	0	87	13	0	0	1	113	10	51	22	315
26825	0	0	0	93	7	0	0	1	114	12	59	28	315
31922	0	0	0	92	8	0	0	1	114	12	62	23	315
37022	0	0	0	87	13	0	0	1	114	11	58	21	315
42123	0	0	0	94	6	0	0	1	115	12	59	22	315
47225	0	0	0	93	7	0	0	1	115	14	71	28	315
52325	0	0	0	92	8	0	0	2	115	14	72	24	315
58163	42	0	42	52	6	0	0	2	119	26	77	100	320
63324	29	0	29	61	11	0	0	2	127	24	92	99	320
68467	32	0	32	59	9	0	0	2				100	320
73645	34	0	34	57	9	0	0	2	118	21	76	100	320
78806	35	0	35	57	8	0	0	2	118	25	86	100	320
83964	31	0	31	60	9	0	0	2	118	25	90	100	320
89123	33	0	33	54	13	0	0	2	117	23	86	100	320
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712	
AVG 315	0	0	0	91	9	0	0	1	114	12	62	24	315
STD	0	0	0	3	3	0	0	0	1	1	7	2	
AVG 320	34	0	34	57	9	0	0	2	120	24	84	100	320
STD	4	0	4	3	2	0	0	0	4	1	6	0	

RUN 11202024

CAT 540

RUN 11202024

CAT 540

RUN 11202024

CAT 540

RUN 11202024

CAT 540

00003621
 00003621
 00003621 OPERATOR NAME: B. CRUM DATE: 05-MAR-84
 00003621 LOG BOOK #: 11202 PAGE #: 86
 00003621 RUN #: 11202028 CATALYST #: 540
 00003621 CATALYST NAME: Cu/La/Li
 00003621 COMMENTS: 6-1-1.3 higher space velocities, taller bed

00003621
 00003621
 00003621 MASS OF CATALYST (g): 6.58
 00003621 REACTOR INSIDE DIAM (CM): 0.45
 00003621 CATALYST BED HEIGHT (CM): 30.5

00003500
 00003500 INITIAL FINAL ABSOLUTE
 00003500 TEMP TEMP RAMP RATE
 00003500 (DEG C) (DEG C) (DEG C / MIN)
 00003500
 00003500
 00003500 BOTTOM: 310 315 1
 00003500 MIDDLE: 310 315 1
 00003500 TOP : 310 315 1

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 320 O2 FLOW RATE (CC/MIN): 64
 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ1712
13727	0	0	0	98	2	0	0	0	1	129	11	51	16 315
18889	0	0	0	98	2	0	0	0	1	128	10	45	16 315
24041	0	0	0	99	1	0	0	0	1	124	10	46	16 315
29141	0	0	0	98	2	0	0	0	1	131	10	48	15 315
34248	0	0	0	99	1	0	0	0	1	129	11	51	17 315
39402	0	0	0	98	2	0	0	0	2	135	16	72	16 317
45245	26	0	26	71	2	0	0	0	2	188	100	345	98 320
50403	0	18	18	77	5	0	0	0	2	188	100	394	99 320
55564	0	27	27	73	0	0	0	0	2	190	100	329	98 320
60726	0	32	32	65	3	0	0	0	2	191	100	318	98 320

*****NOTE GC PROBLEMS CAUSED BAD READINGS WITH CO AND CO2. DATA IN QUESTION.*****

RUN 11202028

CAT 540

00003687 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00003687
 00003687
 00003687 OPERATOR NAME: B. CRUM DATE: 05-MAR-84
 00003687 LOG BOOK #: 11202 PAGE #: 86
 00003687 RUN #: 11202028 CATALYST #: 540
 00003687 CATALYST NAME: Cu/La/Li
 00003687 COMMENTS: 6-1-1.3 higher space velocities, taller bed
 00003687
 00003687
 00003687 MASS OF CATALYST (g): 5.58
 00003687 REACTOR INSIDE DIAM (CM): 0.45
 00003687 CATALYST BED HEIGHT (CM): 30.5

OHC/JSS

=====

TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

=====

INITIAL TEMP (DEG C)	FINAL TEMP (DEG C)	ABSOLUTE RAMP RATE (DEG C / MIN)
BOTTOM: 310	315	1
MIDDLE: 310	315	1
TOP : 310	315	1

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00003684 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54
 00003684 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0
 00003684

Time	TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 10							ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4						
11550													
16711													
21848	0	0	0	100	0	0	0	1	133	18	72	18	318
26951	0	0	0	100	0	0	0	1	133	15	69	15	315
32050	0	0	0	100	0	0	0	1	131	14	65	12	315
37149	0	0	0	100	0	0	0	1	130	12	55	10	315
42248	0	0	0	100	0	0	0	1	130	13	57	11	315
47358	0	0	0	100	0	0	0	2	129	11	48	9	316
53170	0	0	0	100	0	0	0	2	128	12	53	8	320
58269	0	0	0	100	0	0	0	2	128	10	48	10	320
63371	0	0	0	100	0	0	0	2	127	11	49	9	320
68470	0	0	0	100	0	0	0	2	128	11	48	9	320
78610	0	0	0	100	0	0	0	2	128	11	50	10	320

*****NOTE GC DID NOT SEPERATE CO AND CH4. CONVERSION DATA IS NOT ACCURATE. TEMP DATA IS GOOD.

RUN 11202029

CAT 542

00005268 OPERATOR NAME: B. CRUM DATE: 06-MAR-84
 00005268 LOG BOOK #: 11202 PAGE #: 91
 00005268 RUN #: 11202030 CATALYST #: 644
 00005268 CATALYST NAME: Cu/La/Li auz 1step
 00005268 COMMENTS: catalyst screening run

00005268

00005268 MASS OF CATALYST (g): 4.71

00005268 REACTOR INSIDE DIAM (CM): 0.45

00005268 CATALYST BED HEIGHT (CM): 30.5

00005089 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00005089 =====

00005089 INITIAL FINAL ABSOLUTE
 00005089 TEMP TEMP RAMP RATE
 00005089 (DEG C) (DEG C) (DEG C / MIN)

00005089 =====

00005089 BOTTOM: 310 315 1

00005089 MIDDLE: 310 315 1

00005089 TOP : 310 315 1

00005089

00005089 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00005089

00005089 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 64

00005089 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

00005089

00005089 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 10

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
11744	23	15	38	51	11	0	0	0	1	129	22	72	100 316
16905	26	21	47	51	2	0	0	0	1	131	22	56	99 315
22081	0	26	26	74	0	0	0	0	1	187	97	333	98 315
27227	0	23	23	72	4	0	0	0	1	186	97	358	98 315
32401	0	24	24	72	5	0	0	0	1	186	97	360	98 315
37558	0	22	22	72	6	0	0	0	1	184	95	362	98 315
42705	0	22	22	72	6	0	0	0	2	184	95	362	98 318
48705	0	22	22	73	5	0	0	0	2	183	92	348	98 320
53865	0	21	21	75	4	0	0	0	2	189	100	379	98 320
59018	0	32	32	68	0	0	0	0	2	190	100	312	-291 320
64128	0	27	27	73	0	0	0	0	2	189	100	332	-327 320
69280	0	31	31	69	0	0	0	0	2	191	100	314	-327 320

*****NOTE GC data for run is not good due to shifting times on GC trace.
 Temperature data is accurate.*****

RUN11202030

CAT 544

00004205 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00004205 =====
 00004205
 00004205 OPERATOR NAME: B. CRUM DATE: 09-MAR-84
 00004205 LOG BOOK #: 11202 PAGE #: 96
 00004205 RUN #: 11202033 CATALYST #: 544
 00004205 CATALYST NAME: Cu/Li/La acu prep
 00004205 COMMENTS: Lower starting temp in an effort to control reaction
 00004205
 00004205
 00004205 MASS OF CATALYST (g): 3.84
 00004205 REACTOR INSIDE DIAM (CM): 0.45
 00004205 CATALYST BED HEIGHT (CM): 30.5

00004118 =====
 00004118 INITIAL FINAL ABSOLUTE
 00004118 TEMP TEMP RAMP RATE
 00004118 (DEG C) (DEG C) (DEG C / MIN)
 00004118 =====
 00004118 BOTTOM: 305 305 1
 00004118 MIDDLE: 305 305 1
 00004118 TOP : 305 305 1

00004118 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00004118

00004118 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 64
 00004118 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
10479	68	1	70	28	5	0	0	0	1	132	15	86	100 305
15628	3	67	70	30	0	0	0	0	1	110	9	992	100 305
20773	4	96	100	0	0	0	0	0	1	102	9	0	100 305
25833	4	96	100	0	0	0	0	0	1	102	9	0	100 306
31094	5	95	100	0	0	0	0	0	1	103	10	0	100 305
36253	3	86	89	11	0	0	0	0	2	103	5	134	100 306
42074	12	62	74	26	0	0	0	0	2	103	8	543	100 310
47232	0	100	100	0	0	0	0	0	2	190	97	0	100 310
52408	0	100	100	0	0	0	0	0	2	191	98	0	100 310
57555	0	100	100	0	0	0	0	0	2	189	96	0	100 310

*****NOTE Rx PLUGGED HALF WAY THROUGH SECOND SET OF DATA HCl DID NOT FLOW AFTER THIS TIME.*****

RUN 11202033

CAT 544

00018519 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00018519 *****
 00018519 OPERATOR NAME: B. CRUM DATE: 10-MAR-84
 00018519 LOG BOOK #: 11202 PAGE #: 98
 00018519 RUN #: 11202034 CATALYST #: 546
 00018519 CATALYST NAME: Cu/Li/La Lower La
 00018519 COMMENTS: Experiment to determine necessary level of La
 00018519
 00018519 MASS OF CATALYST (g): 3.67
 00018519 REACTOR INSIDE DIAM (CM): 0.45
 00018519 CATALYST BED HEIGHT (CM): 30.5

00018413 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00018413 *****
 00018413 INITIAL FINAL ABSOLUTE
 00018413 TEMP TEMP RAMP RATE
 00018413 (DEG C) (DEG C) (DEG C / MIN)
 00018413 *****
 00018413 BOTTOM: 310 315 1
 00018413 MIDDLE: 310 315 1
 00018413 TOP : 310 315 1

00018413 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00018413 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54

00018413 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
28501	0	0	0	94	6	0	0	0	1	69	5	22	17 315
33663	0	0	0	94	6	0	0	0	1	70	4	21	17 315
38807	0	0	0	95	5	0	0	0	1	69	4	18	16 315
43922	0	0	0	94	6	0	0	0	1	70	5	23	16 315
49066	0	0	0	93	7	0	0	0	2	71	5	23	16 315
54925	0	1	1	89	10	0	0	0	2	176	89	443	95 320
60059	0	26	26	70	3	0	0	0	2	173	88	312	95 320
65245	0	31	31	68	1	0	0	0	2	174	89	287	95 320
70379	0	0	0	100	0	0	0	0	2	75	3	15	92 320
75540	0	0	0	100	0	0	0	0	2	74	4	17	92 320
Averages													
Avg 315	0	0	0	94	6	0	0	0	1	70	4	21	16 315
Std	0	0	0	1	1	0	0	0	0	1	0	2	0
Avg 320	0	12	12	85	3	0	0	0	2	134	55	215	94 320
Std	0	14	14	14	4	0	0	0	0	49	42	171	1

RUN 11202034

CAT 546

—■— CONVERSION OF CH₄

RUN 11202034

CAT 546

RUN 11202034

CAT 546

RUN 11202034

CAT 546

00002404 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00002404
 00002404 OPERATOR NAME: B. CRUM DATE: 11-MAR-84
 00002404 LOG BOOK #: 11202 PAGE #: 100
 00002404 RUN #: 11202035 CATALYST #: 541
 00002404 CATALYST NAME: Cu/LI/K
 00002404 COMMENTS: Experimentm to substitute K for La

00002404
 00002404
 00002404 MASS OF CATALYST (g): 4.75
 00002404 REACTOR INSIDE DIAM (CM): 0.45
 00002404 CATALYST BED HEIGHT (CM): 30.5

00002258 OHC/JSS
 00002258
 00002258
 00002258 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7
 00002258
 00002258 INITIAL FINAL ABSOLUTE
 00002258 TEMP TEMP RAMP RATE
 00002258 (DEG C) (DEG C) (DEG C / MIN)
 00002258
 00002258
 00002258 BOTTOM: 310 315 1
 00002258 MIDDLE: 310 315 1
 00002258 TOP : 310 315 1

00002258
 00002258 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00002258

00002258 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54
 00002258 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
16599	0	0	0	100	0	0	0	0	1	74	3	12	2
21761	0	0	0	100	0	0	0	0	1	76	2	11	1
26920	0	0	0	100	0	0	0	0	1	76	3	13	1
32079	0	0	0	100	0	0	0	0	1	76	3	13	1
37238	0	0	0	100	0	0	0	0	1	76	3	13	2
42400	0	0	0	100	0	0	0	0	2	76	3	14	2
49180	0	0	0	100	0	0	0	0	2	76	4	17	4
54317	0	0	0	100	0	0	0	0	2	76	3	12	3
59418	0	0	0	100	0	0	0	0	2	76	4	18	5
64519	0	0	0	100	0	0	0	0	2				33
69625	0	0	0	100	0	0	0	0	2	76	3	15	4
74785	0	0	0	100	0	0	0	0	3	76	3	15	6
81518	0	0	0	96	4	0	0	0	3	75	3	15	9
91344	27	10	37	54	9	0	0	0	1	68	15	51	100
96519	25	15	40	52	8	0	0	0	1	69	15	47	100
101680	24	15	39	53	8	0	0	0	1	68	16	50	100
106827	25	15	41	52	8	0	0	0	1	69	16	48	100
111999	21	18	38	53	8	0	0	0	1	67	16	51	100
117159	22	16	38	55	8	0	0	0	1	71	13	42	100
127785	0	4	4	88	7	0	0	0	1	78	8	39	19
132926	0	5	5	83	12	0	0	0	1	67	11	52	31
138086	0	3	3	85	11	0	0	0	1	65	9	42	26
143246	0	3	3	84	13	0	0	0	1	66	4	18	22
148406	0	3	3	87	10	0	0	0	1	64	9	46	27
153566	0	3	3	87	10	0	0	0	1	64	9	45	28
158725	0	3	3	85	12	0	0	0	1	65	10	48	29
163886	0	3	3	86	12	0	0	0	1	66	9	47	27
AVERAGES													
AVG 320	0	0	0	100	0	0	0	0	1	76	3	13	2
	0	0	0	0	0	0	0	0	0	1	0	1	0
AVG 330	0	0	0	100	0	0	0	0	2	76	3	15	4
	0	0	0	0	0	0	0	0	0	0	0	2	1
AVG 355	24	15	39	53	8	0	0	0	1	69	15	48	100
W/O N2	2	2	1	1	1	0	0	0	0	1	1	3	0
AVG 355	0	3	3	86	11	0	0	0	1	67	9	42	26
WN2	0	1	1	2	2	0	0	0	0	4	2	10	4

****NOTE: TEMP 355 WAS RUN WITH N2 AND WITHOUT N2. THESE ARE THE TWO AVERAGES

RUN 11202035
CAT 541

RUN 11202035
CAT 541

RUN 11202035

CAT 541

RUN 11202035

CAT 541

—■— SELECTIVITY TO M1

00008225 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00008225 =====
 00008225
 00008225 OPERATOR NAME: B. CRUM DATE: 13-MAR-84
 00008225 LOG BOOK #: 11202 PAGE #: 102
 00008225 RUN #: 11202036 CATALYST #: 544
 00008225 CATALYST NAME: aqueous 1 step
 00008225 COMMENTS: trying to control aq. catalyst, dried at 350C
 00008225
 00008225
 00008225 MASS OF CATALYST (g): 4.81
 00008225 REACTOR INSIDE DIAM (CM): 0.45
 00008225 CATALYST BED HEIGHT (CM): 30.6

00008102 =====
 00008102 INITIAL FINAL ABSOLUTE
 00008102 TEMP TEMP RAMP RATE
 00008102 (DEG C) (DEG C) (DEG C / MIN)
 00008102 =====
 00008102 BOTTOM: 290 295 1
 00008102 MIDDLE: 290 295 1
 00008102 TOP : 290 295 1

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 64
 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

00008102
 00008102 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 6
 00008102

Time	A17CO	A17CO2	A17COX	A17M1	A17M2	A17M3	A17M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ1712
18314	0	0	0	100	0	0	0	0	1	80	4	16	6
21460	0	0	0	100	0	0	0	0	1	78	4	20	6
26618	0	0	0	100	0	0	0	0	1	78	4	19	6
31780	0	0	0	100	0	0	0	0	1	78	4	19	6
36941	0	0	0	100	0	0	0	0	2	78	3	16	5
42761	0	0	0	100	0	0	0	0	2	78	5	22	9
47919	0	0	0	100	0	0	0	0	2	78	3	15	7
53080	0	0	0	100	0	0	0	0	2	78	5	21	8
58238	0	0	0	100	0	0	0	0	2	77	4	18	8
63400	0	0	0	100	0	0	0	0	3	77	4	17	8
69233	0	0	0	95	5	0	0	0	3	76	5	26	12
74381	0	0	0	95	5	0	0	0	3	76	6	26	12
79542	0	0	0	95	5	0	0	0	3	77	6	27	12
84715	0	0	0	95	5	0	0	0	3	76	5	26	11
89860	0	0	0	95	5	0	0	0	4	76	5	25	11
95694	37	1	38	57	5	0	0	0	4	85	14	42	100
115674	5	12	18	68	14	0	0	0	1	85	17	74	99
120634	5	13	18	69	13	0	0	0	1	85	17	74	99
125694	4	12	16	70	14	0	0	0	1	83	13	58	99
131154	0	13	13	71	16	0	0	0	1	84	16	77	96
136300	0	8	8	74	18	0	0	0	1	82	15	76	77
141473	4	9	13	70	16	0	0	0	1	84	17	79	96
146634	6	10	17	68	15	0	0	0	1	83	17	75	98
151793	7	11	17	69	14	0	0	0	1	82	16	72	98
156953	3	11	14	71	15	0	0	0	1	83	16	76	94
AVG 295	0	0	0	100	0	0	0	0	1	79	4	18	6
STD	0	0	0	0	0	0	0	0	0	1	0	2	0
AVG 300	0	0	0	100	0	0	0	0	2	77	4	19	8
STD	0	0	0	0	0	0	0	0	0	0	1	2	0
AVG 305	0	0	0	95	5	0	0	0	3	76	5	26	12
STD	0	0	0	0	0	0	0	0	0	0	0	1	0
310 (1PT)	37	1	38	57	5	0	0	0	4	85	14	42	100
AVG 320	4	11	15	70	15	0	0	0	1	83	16	73	95
STD	2	1	3	2	1	0	0	0	0	1	1	6	7

POINTS AT 320 ARE WITH N2 ADDED TO STREAM

RUN 11202036

CAT 544

RUN 11202036

CAT 544

RUN 11202036

CAT 544

RUN 11202036
CAT 544

00002555 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00002555
 00002555

00002555 OPERATOR NAME: B. CRUM DATE: 17-MAR-94
 00002555 LOG BOOK #: 11202 PAGE #: 104
 00002555 RUN #: 11202037 CATALYST #: 550C
 00002555 CATALYST NAME: Cu/Li/La
 00002555 COMMENTS: catalyst screen
 00002555
 00002555

00002555 MASS OF CATALYST (g): 4.49
 00002555 REACTOR INSIDE DIAM (CM): 0.45
 00002555 CATALYST BED HEIGHT (CM): 30.5

00002453 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00002453
 00002453 INITIAL FINAL ABSOLUTE
 00002453 TEMP TEMP RAMP RATE
 00002453 (DEG C) (DEG C) (DEG C / MIN)
 00002453
 00002453
 00002453 BOTTOM: 310 315 1
 00002453 MIDDLE: 310 315 1
 00002453 TOP : 310 315 1
 00002453
 00002453
 00002453 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00002453
 00002453
 00002453 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 40
 00002453 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712	
11811	8	13	21	67	12	0	0	0	1	71	14	60	100	315
16980	10	13	22	67	10	0	0	0	1	71	13	53	100	315
22130														315
27302	11	13	24	66	10	0	0	0	1	73	11	43	100	315
32450	12	13	25	66	10	0	0	0	1	74	11	45	100	315
37623	12	13	24	66	9	0	0	0	1	74	15	57	100	315
42782	11	13	24	66	10	0	0	0	1	74	15	58	100	315
47941	11	13	24	67	10	0	0	0	1	74	14	56	100	315
53092	10	13	23	67	10	0	0	0	2	74	15	59	100	315
58982	9	12	22	68	10	0	0	0	2	74	15	61	100	315
64130	10	13	23	66	11	0	0	0	2	75	15	61	100	320
69277	9	13	22	68	10	0	0	0	2	75	14	58	100	320
74461	10	13	23	67	10	0	0	0	2	75	15	61	100	320
79812	10	13	22	68	10	0	0	0	2	75	15	61	100	320
84773	10	13	23	67	10	0	0	0	2	75	15	60	100	320
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712		
AVG 315	10	13	23	67	10	0	0	0	1	73	14	55	100	315
STD	1	0	1	1	1	0	0	0	0	2	1	6	0	
AVG 320	10	13	23	67	10	0	0	0	2	75	15	60	100	320
STD	0	0	0	1	0	0	0	0	0	0	0	1	0	

RUN 11202037

CAT 550C

RUN 11202037

CAT 550C

RUN 11202037

CAT 550C

RUN 11202037

CAT 550

00005621 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00005621
 00005621
 00005621 OPERATOR NAME: B. CRUM DATE: 18-MAR-84
 00005621 LOG BOOK #: 11202 PAGE #: 106
 00005621 RUN #: 11202038 CATALYST #: 551
 00005621 CATALYST NAME: Cu/Li/Th
 00005621 COMMENTS: Catalyst Screen
 00005621
 00005621
 00005621 MASS OF CATALYST (g): 4.94
 00005621 REACTOR INSIDE DIAM (CM): 0.45
 00005621 CATALYST BED HEIGHT (CM): 30.5

00005554 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00005554
 00005554 INITIAL FINAL ABSOLUTE
 00005554 TEMP TEMP RAMP RATE
 00005554 (DEG C) (DEG C) (DEG C / MIN)
 00005554

00005554
 00005554 BOTTOM: 310 310 1
 00005554 MIDDLE: 310 310 1
 00005554 TOP : 310 310 1
 00005554

00005554 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00005554 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 40
 00005554 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0
 00005554

00005554 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 8

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
11562	0	2	2	92	6	0	0	0	1	75	6	30	20 310
16725	0	0	0	95	5	0	0	0	1	74	5	26	17 311
21885	0	0	0	95	5	0	0	0	1	74	5	26	17 310
27035	0	0	0	94	6	0	0	0	1	152	66	322	17 310
32144	0	0	0	95	5	0	0	0	1	74	5	26	18 310
37296	0	0	0	95	5	0	0	0	2	73	5	24	18 312
43115	0	1	1	92	7	0	0	0	2	74	5	26	22 315
48224	0	1	1	92	7	0	0	0	2	73	4	21	22 315
53376	0	1	1	92	7	0	0	0	2	73	6	31	24 315
58475	0	2	2	90	8	0	0	0	2	74	6	27	22 315
63577	0	1	1	91	7	0	0	0	2	74	6	29	21 315
68675	0	2	2	90	8	0	0	0	3	116	4	21	-15 316
74530	28	7	35	53	12	0	0	0	3	67	12	41	100 320
79689	37	26	63	36	1	0	0	0	3	81	12	21	100 323
84824	0	0	0	100	0	0	0	0	3	80	4	19	10 329
89983	0	0	0	95	5	0	0	0	3	87	5	24	-207 344
Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
AVG	0.	0	0	94	6	0	0	0	1	87	16	76	18 310
STD	0	1	1	1	0	0	0	0	0	29	23	110	1
AVG	0	2	2	91	7	0	0	0	2	81	5	26	16 315
STD	0	0	0	1	1	0	0	0	0	16	1	4	14
IND. RUNS	28	7	35	53	12	0	0	0	3	67	12	41	100 320
IND. RUNS	37	26	63	36	1	0	0	0	3	81	12	21	100 323
IND. RUNS	0	0	0	100	0	0	0	0	3	80	4	19	10 329
IND. RUNS	0	0	0	95	5	0	0	0	3	87	5	24	-207 344

***NOTE: LAST 4 POINTS ARE INDIVIDUAL RUNS COPIED TO RESULTS SECTION. CONDITIONS WERE CHANGING DURING THIS TIME.

RUN 11202038

CAT 551

RUN 11202038

CAT 551

RUN 11202038
CAT 551

RUN 11202038
CAT 551

00008002 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00008002 =====
 00008002

00008002 OPERATOR NAME: B. CRUM DATE: 21-MAR-84
 00008002 LOG BOOK #: 11202 PAGE #: 112
 00008002 RUN #: 11202041 CATALYST #: 550A
 00008002 CATALYST NAME: Cu/Li/La
 00008002 COMMENTS: Catalyst Screening Extra TC on outside of Rx

00008002
 00008002
 00008002 MASS OF CATALYST (g): 4.62
 00008002 REACTOR INSIDE DIAM (CM): 0.45
 00008002 CATALYST BED HEIGHT (CM): 30.5

00007925 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00007925 =====
 00007925 INITIAL FINAL ABSOLUTE
 00007925 TEMP TEMP RAMP RATE
 00007925 (DEG C) (DEG C) (DEG C / MIN)
 00007925 =====
 00007925 BOTTOM: 305 305 1
 00007925 MIDDLE: 305 305 1
 00007925 TOP : 305 305 1

00007925
 00007925 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)
 00007925

00007925 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54
 00007925 HCI FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0
 00007925

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 8															
Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712		
13466	0	0	0	100	0	0	0	0	1	75	4	19	13	305	
18604	0	0	0	100	0	0	0	0	1	75	4	18	13	305	
23714	0	0	0	100	0	0	0	0	1	74	0	2	11	305	
28873	0	0	0	100	0	0	0	0	1	74	4	20	17	305	
34031	0	0	0	100	0	0	0	0	1	75	4	20	17	305	
39192	0	0	0	100	0	0	0	0	2	75	3	15	18	305	
45012	0	0	0	96	4	0	0	0	2	74	5	23	21	310	
50188	0	0	0	95	5	0	0	0	2	74	4	20	21	310	
55346	0	0	0	96	4	0	0	0	2	73	3	13	20	310	
60507	0	0	0	96	4	0	0	0	2	73	5	23	23	310	
65665	0	0	0	96	4	0	0	0	2	73	4	17	22	310	
70812	0	0	0	95	5	0	0	0	3	73	5	23	23	310	
76647	0	2	2	93	6	0	0	0	3	73	6	26	26	315	
81792	0	1	1	93	6	0	0	0	3	73	5	25	24	315	
86966	0	2	2	92	6	0	0	0	3	73	6	26	24	315	
Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712		
AVG 305	0	0	0	100	0	0	0	0	1	75	3	16	15	305	
STD	0	0	0	0	0	0	0	0	0	1	1	6	2		
AVG 310	0	0	0	96	4	0	0	0	2	73	4	20	22	310	
STD	0	0	0	0	0	0	0	0	0	0	1	4	1		
AVG 315	0	2	2	93	6	0	0	0	3	73	5	26	25	315	
STD	0	0	0	0	0	0	0	0	0	0	0	1	1		

RUN 11202041

CAT 550A

RUN 11202041

CAT 550A

—■— SELECTIVITY TO M1

RUN 11202041

CAT 550A

RUN 11202041

CAT 550A

00004789 LOG BOOK #: 11202 PAGE #: 114
 00004789 RUN #: 11202042 CATALYST #: 550b
 00004789 CATALYST NAME: Cu/LiLa
 00004789 COMMENTS: Catalyst Screening Extra TC on outside of Rx

00004789
 00004789

00004789 MASS OF CATALYST (g): 4.3
 00004789 REACTOR INSIDE DIAM (CM): 0.45
 00004789 CATALYST BED HEIGHT (CM): 30.5

00004736 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

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=====
00004736      INITIAL  FINAL  ABSOLUTE
00004736      TEMP    TEMP    RAMP RATE
00004736      (DEG C)  (DEG C)  (DEG C / MIN)
00004736      =====
00004736      BOTTOM: 305    305    1
00004736      MIDDLE: 305    305    1
00004736      TOP : 305    305    1
00004736
```

00004736 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00004736 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54

00004736 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

00004736

00004736

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 7														
Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TOI712	
16720	0	0	0	95	5	0	0	0	1	77	5	24	14	305
21867	0	0	0	94	6	0	0	0	1	79	5	25	13	305
27027	0	0	0	94	6	0	0	0	1	78	5	24	13	305
32186	0	0	0	96	4	0	0	0	2	76	5	24	13	307
38007	0	1	1	92	7	0	0	0	2	76	7	32	20	310
43168	0	1	1	91	8	0	0	0	2	76	6	32	19	310
48328	0	2	2	90	8	0	0	0	2	76	7	32	20	310
53488	0	1	1	92	7	0	0	0	2	75	7	32	19	310
58659	0	1	1	92	7	0	0	0	3	75	7	32	19	312
64550	19	13	33	55	12	0	0	0	3	69	16	57	100	315
69709	24	17	41	53	6	0	0	0	3	74	15	45	100	315
74859	24	17	41	54	5	0	0	0	3	76	16	47	100	315
80020	23	18	41	53	5	0	0	0	3	75	16	47	100	315
85191	22	18	40	54	6	0	0	0	0	78	16	49	100	315
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TOI712		
AVG 305	0	0	0	95	5	0	0	0	1	78	5	24	13	305
STD	0	0	0	1	1	0	0	0	0	1	0	0	0	
AVG 310	0	1	1	91	7	0	0	0	2	75	7	32	19	310
STD	0	0	0	1	1	0	0	0	0	0	0	0	0	
AVG 315	23	17	39	54	7	0	0	0	2	74	16	49	100	315
STD	2	2	3	1	2	0	0	0	1	2	0	4	0	

RUN 11202041

CAT 550B

RUN 11202041

CAT 550B

CAT 550B

RUN 11202042

CAT 550B

00003930 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00003930
 00003930
 00003930 OPERATOR NAME: B. CRUM DATE: 23-MAR-84
 00003930 LOG BOOK #: 11202 PAGE #: 116
 00003930 RUN #: 11202043 CATALYST #: 550c
 00003930 CATALYST NAME: Cu/Li/La
 00003930 COMMENTS: Catalyst Screening Extra TC on outside of Rx
 00003930
 00003930
 00003930 MASS OF CATALYST (g): 4.56
 00003930 REACTOR INSIDE DIAM (CM): 0.45
 00003930 CATALYST BED HEIGHT (CM): 30.5

OHC/JSS

=====

TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

=====

00003931 INITIAL FINAL ABSOLUTE
 00003931 TEMP TEMP RAMP RATE
 00003931 (DEG C) (DEG C) (DEG C / MIN)

00003931
 00003931 BOTTOM: 305 305 1
 00003931 MIDDLE: 305 305 1
 00003931 TOP : 305 305 1

(INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00003931 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54
 00003931 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0
 00003931

TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 7													
Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ712
10255	0	2	2	91	7	0	0	0	1	76	6	27	16
15416	0	0	0	94	6	0	0	0	1	77	6	28	15
20563	0	0	0	95	5	0	0	0	1	77	1	6	10
25722	0	0	0	94	6	0	0	0	1	79	4	21	13
30896	0	0	0	94	6	0	0	0	2	78	5	26	14
36761	0	3	3	82	15	0	0	0	2	82	7	35	24
41945	21	18	39	54	6	0	0	0	2	77	16	48	100
47107	26	24	50	47	3	0	0	0	2	79	11	26	100
52255	27	23	50	47	3	0	0	0	2	79	15	36	100
57415	26	24	49	48	2	0	0	0	3	78	13	32	100
63306	26	23	49	48	2	0	0	0	3	78	15	36	100
68455	27	25	52	48	2	0	0	0	3	6	15	34	99
AVERAGES AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQ712	
AVG 305	0	1	1	92	8	0	0	0	1	78	5	24	15
STD	0	1	1	5	3	0	0	0	0	2	2	9	4
AVG 310	25	22	47	49	4	0	0	0	2	78	14	35	100
STD	2	2	5	3	2	0	0	0	0	1	2	8	0
AVG 315	27	24	51	47	2	0	0	0	3	42	15	35	99
STD	1	1	1	1	0	0	0	0	0	36	0	1	0

RUN 11202043

CAT 550C

RUN 11202043

CAT 550C

—■— SELECTIVITY TO M1

RUN 11202043

CAT 550C

RUN 11202043

CAT 550C

00002601 HOOD #7 RAMPING EXPERIMENT DEFINITION
 00002601 =====
 00002601 OPERATOR NAME: B. CRUM DATE: 24-MAR-84
 00002601 LOG BOOK #: 11202 PAGE #: 118
 00002601 RUN #: 11202044 CATALYST #: 550d
 00002601 CATALYST NAME: Cu/Li/La
 00002601 COMMENTS: Catalyst Screening Extra TC on outside of Rx

00002601
 00002601

00002601 MASS OF CATALYST (g): 4.72
 00002601 REACTOR INSIDE DIAM (CM): 0.45
 00002601 CATALYST BED HEIGHT (CM): 30.5

00002547 TEMPERATURE RAMPING EXPERIMENT FOR HOOD #7

00002547

00002547 INITIAL FINAL ABSOLUTE
 00002547 TEMP TEMP RAMP RATE
 00002547 (DEG C) (DEG C) (DEG C / MIN)

00002547

00002547 BOTTOM: 305 305 1
 00002547 MIDDLE: 305 305 1
 00002547 TOP : 305 305 1

00002547

00002547 (INITIAL TEMPERATURE CAN BE GREATER THAN FINAL TEMPERATURE)

00002547

00002547 CH4 FLOW RATE (CC/MIN): 325 O2 FLOW RATE (CC/MIN): 54
 00002547 HCl FLOW RATE (CC/MIN): 71 AUX FLOW RATE (CC/MIN): 0

00002547

00002547 TIME TO MAINTAIN FINAL TEMPERATURES (HOURS): 7

Time	AI7CO	AI7CO2	AI7COX	AI7M1	AI7M2	AI7M3	AI7M4	ID7	MBC7	Q7CH4C	Q7HCLC	Q7O2C	TQI712
85056	27	3	30	62	8	0	0	1	82	15	54	100	305

*****NOTE REACTION ENDED AFTER A SHORT TIME AT TEMP 305 BECAUSE TEMPERTURE WAS OUT OF CONTROL

RUN 11202044

CAT 550D

Data Appendix B

Catalyst Synthesis and Composition Information

CATALYST 540

SUPPORT

Al₂O₃, CS331-1, UCI, Lot # 3208-S.

SOURCE

[Cu(CH₃CN)₄]{PF₆}. Lab-book 11291 page 46.

LOADING

0.45% Cu, 14.2% La, 0.61% Li, and 0.24% P.

IMPREGNATION METHOD

Al₂O₃ was left overnight to impregnate with a THF solution containing [Cu(CH₃CN)₄]{PF₆}. THF was removed with flowing nitrogen before impregnation overnight with aqueous solution of La and Li salts. Solution was then evaporated using flowing nitrogen.

PRETREATMENT

Al₂O₃ was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.

COLOR

Yellow-green.

CATALYST 541

SUPPORT

Al₂O₃, CS331-1, UCI, Lot # 3208-S.

SOURCE

[Cu(CH₃CN)₄]{PF₆}. Lab-book 11291 page 46.

LOADING

0.24% Cu, 16.3% La, 5.3% K, and 0.16% P.

IMPREGNATION METHOD

Al₂O₃ was left overnight to impregnate with a THF solution containing [Cu(CH₃CN)₄]{PF₆}. THF was removed with flowing nitrogen before impregnation overnight with aqueous solution of La and K salts. Solution was then evaporated using flowing nitrogen.

PRETREATMENT

Al₂O₃ was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.

COLOR

Yellow-green.

CATALYST 542

SUPPORT	Al ₂ O ₃ , CS331-1, UCI, Lot # 3208-S.
SOURCE	[Cu(CH ₃ CN) ₄]{PF ₆ }. Lab-book 11291 page 46.
LOADING	0.69% Cu, 0.95% Li, and 0.39% P.
IMPREGNATION METHOD	Al ₂ O ₃ was left overnight to impregnate with a THF solution containing [Cu(CH ₃ CN) ₄]{PF ₆ }. THF was removed with flowing nitrogen before impregnation overnight with aqueous solution of Li salt. Solution was then evaporated using flowing nitrogen.
PRETREATMENT	Al ₂ O ₃ was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.
COLOR	Yellow-green.

CATALYST 543

SUPPORT	Al ₂ O ₃ CS331-1, UCI, Lot # 3208-S.
SOURCE	[Cu(CH ₃ CN) ₄]{PF ₆ }. Lab-book 11291 page 48.
LOADING	0.16% Cu, 1.32% La, 0.003% Li, and 0.47% P.
IMPREGNATION METHOD	Al ₂ O ₃ was left overnight to impregnate with a THF solution containing [Cu(CH ₃ CN) ₄]{PF ₆ }. Solution was decanted via canula, and then clean THF was added leaving solution overnight. THF was decanted via canula, and catalyst dried in vacuum oven. Catalyst was then impregnated overnight with aqueous solution containing LaCl ₃ and Li salts. Catalyst was washed on Buchner funnel with water before drying in vacuum oven.
PRETREATMENT	Al ₂ O ₃ was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.
COLOR	Tan.

CATALYST 544

SUPPORT	Al_2O_3 , CS331-1, UCI, Lot # 3208-S.
SOURCE	$\text{Cu}(\text{NO}_3)_2 \times 6 \text{H}_2\text{O}$. Lab-book 11291 page 50.
LOADING	2.16% Cu, 22.7% La, 0.73% Li.
IMPREGNATION METHOD	Al_2O_3 impregnated overnight with aqueous solution containing $\text{Cu}(\text{NO}_3)_2 \times 6 \text{H}_2\text{O}$ and La and Li salts. Solution was removed by flowing nitrogen before drying in vacuum oven.
PRETREATMENT	Al_2O_3 was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.
COLOR	Yellow-green speckled brown.

CATALYST 545

SUPPORT	Al_2O_3 , CS331-1, UCI, Lot # 3208-S.
SOURCE	$\text{Cu}(\text{NO}_3)_2 \times 6 \text{H}_2\text{O}$. Lab-book 11291 page 51.
LOADING	2.11% Cu, 18.4% La, 0.67% Li.
IMPREGNATION METHOD	Al_2O_3 was impregnated overnight with aqueous solution of $\text{Cu}(\text{NO}_3)_2 \times 6 \text{H}_2\text{O}$. Sample was then dried in vacuum oven before adding aqueous solution of La and Li salts for impregnation overnight. Solution was removed with flowing air before drying catalyst in vacuum oven.
PRETREATMENT	Al_2O_3 was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.
COLOR	Yellow speckled with brown.

CATALYST 546

SUPPORT

Al₂O₃, CS331-1, UCI, Lot # 3208-S.

SOURCE

[Cu(CH₃CN)₄]{PF₆}. Lab-book 11291 page 52.

LOADING

0.64% Cu, 4.6% La, 0.82% Li, and 0.37% P.

IMPREGNATION METHOD

Al₂O₃ was left overnight to impregnate with a THF solution containing [Cu(CH₃CN)₄]{PF₆}. THF was removed with flowing nitrogen before impregnation overnight with aqueous solution of La and Li salts. Solution was then evaporated using flowing nitrogen and catalyst dried using vacuum oven.

PRETREATMENT

Al₂O₃ was ground and sifted keeping particles between 10 and 24 mesh. These were then heated under dynamic vacuum at 180°C for 4 hours and then 100°C overnight.

COLOR

Speckled brown.

CATALYST 550A

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S.

SOURCE Copper dinitrate hexahydrate, lanthanum trichloride
heptahydrate, lithium chloride - lab book 11291 page
59.

LOADING 0.627% Cu, 0.73% Li, 12.9% La.

IMPREGNATION METHOD Copper nitrate, lanthanum chloride, and lithium
chloride were dissolved in water before being added to
alumina for 16 hour impregnation. Water was then
evaporated with flowing gas and sample was dried at
120°C for 6 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Yellow.

CATALYST 550B

SUPPORT Alumina CS331-1, United Catalyst. Lot #3208-S.

SOURCE Copper dinitrate hexahydrate, lanthanum trichloride
heptahydrate, lithium chloride - lab book 11291 page
59.

LOADING 1.26% Cu, 0.67% Li, 10.8% La.

IMPREGNATION METHOD Copper nitrate, lanthanum chloride, and lithium
chloride were dissolved in water before being added to
alumina for 16 hour impregnation. Water was then
evaporated with flowing gas and sample was dried at
120°C for 6 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Dark yellow.

CATALYST 550C

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S.

SOURCE Copper dinitrate hexahydrate, lanthanum trichloride
heptahydrate, lithium chloride - lab book 11291 page
59.

LOADING 2.34% Cu, 0.68% Li, 11.6% La.

IMPREGNATION METHOD Copper nitrate, lanthanum chloride, and lithium
chloride were dissolved in water before being added to
alumina for 16 hour impregnation. Water was then
evaporated with flowing gas and sample was dried at
120°C for 6 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Tan/brown.

CATALYST 550D

SUPPORT Alumina CS331-1, United Catalyst. Lot #3208-S.

SOURCE Copper dinitrate hexahydrate, lanthanum trichloride
heptahydrate, lithium chloride - lab book 11291 page
59.

LOADING 4.05% Cu, 0.68% Li, 11.7% La.

IMPREGNATION METHOD Copper nitrate, lanthanum chloride, and lithium
chloride were dissolved in water before being added to
alumina for 16 hour impregnation. Water was then
evaporated with flowing gas and sample was dried at
120°C for 6 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Brown with some green particles.

CATALYST 551

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S.

SOURCE Copper dinitrate hexahydrate, thorium tetranitrate tetrahydrate, lithium chloride - lab book 11291 page 60.

LOADING Unknown at this time.

IMPREGNATION METHOD Copper nitrate, thorium nitrate, and lithium chloride were dissolved in water before being added to alumina for 16 hour impregnation. Water was then evaporated with flowing nitrogen and sample was dried at 120°C for 16 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Orange-yellow.

CATALYST 547A

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S

SOURCE Copper tetrakis acetonitrile hexafluorophosphate
- lab book 11291 page 53, 62.

LOADING 0.409% Cu.

IMPREGNATION METHOD Copper species dissolved in THF and transferred via
canula to dry alumina. Alumina was impregnated for
16 hours before THF was removed under flowing
nitrogen or air. Catalyst was dried for 3-4 hours at 120°C
before being sintered under argon at 720°C for 1 hour.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Tan/beige.

CATALYST 547B

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S

SOURCE Copper tetrakis acetonitrile hexafluorophosphate
- lab book 11291 page 53, 63.

LOADING 0.506% Cu.

IMPREGNATION METHOD Copper species dissolved in THF and transferred via
canula to dry alumina. Alumina was impregnated for
16 hours before THF was removed under flowing
nitrogen or air. Catalyst was dried for 3-4 hours at 120°C
before being sintered under argon at 720°C for 4 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Tan/beige.

CATALYST 547C

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S

SOURCE Copper tetrakis acetonitrile hexafluorophosphate
- lab book 11291 page 53, 64.

LOADING 0.527% Cu.

IMPREGNATION METHOD Copper species dissolved in THF and transferred via
canula to dry alumina. Alumina was impregnated for
16 hours before THF was removed under flowing
nitrogen or air. Catalyst was dried for 3-4 hours at 120°C
before being sintered under argon at 720°C for 16 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Tan/beige.

CATALYST 552

SUPPORT Alumina CS331-1, United Catalyst, Lot #3208-S.

SOURCE Copper dinitrate hexahydrate, magnesium chloride,
lithium chloride - lab book 11291 page 61.

LOADING Unknown at this time.

IMPREGNATION METHOD Copper nitrate, magnesium chloride, and lithium
chloride were dissolved in water before being added to
alumina for 16 hour impregnation. Water was then
evaporated with flowing nitrogen and sample was dried
at 120°C for 16 hours.

PRETREATMENT Alumina dried at 170°C for 16 hours.

COLOR Orange-yellow.