DOE/PC/90018--T13

**LIQUID PHASE FISCHER-TROPSCH (II) DEMONSTRATION
IN THE LAPORTE ALTERNATIVE FUELS DEVELOPMENT UNIT**

Topical Report

FINAL

(Volume VII: Appendix)

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MAR 04 1996

OSTI

Task 1: Engineering Modifications (Fischer-Tropsch II Demonstration)

and

**Task 2: AFDU Shakedown, Operations, Deactivation and Disposal
(Fischer-Tropsch II Demonstration)**

Contractor:

**AIR PRODUCTS AND CHEMICALS
Allentown, PA 18195**

**Bharat L. Bhatt
September 1995**

**Prepared for the United States Department of Energy
Under Contract No. DE-AC22-91PC90018
Contract Period October 1990 - December 1995**

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MASTER

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APPENDIX A

Reactor Temperature Stability

MAR 22 1994

PROCESS ENGINEERING

To: Bharat Bhatt

From: V. Grant Fox
21 March 1994

V. Grant Fox

PROCESS DYNAMICS
LIQUID PHASE FISCHER-TROPSCH II

SUMMARY

The Laporte AFDU utility oil loop and reactor were simulated to determine how the reactor temperature will respond to external disturbances and changes in control action. The heat exchangers were modeled as lumped systems with the capacitance being the sum of the capacitance of the oil holdup and the metal mass associated with each exchanger. The Fischer-Tropsch reaction was simplified to first order and heat generation was modeled as the product of the extent of reaction and the heat that would be released at 100% conversion of the feed. Transportation lag between units determined from volumetric flow and piping volumes was also included in the model.

CONCLUSION

- I. The system is open loop unstable. The combination of oil flow and the UA of the heat exchanger inside the reactor is insufficient to control reactor temperature. The control action of the electric heater is definitely needed to achieve stability.
- II. Process piping should be altered and the electric heater should be placed between the water cooled exchanger and the reactor in the AFDU. This placement accomplishes two things. First, stability is improved by eliminating the process lags associated with the exchangers and piping which are presently between the heater and the reactor. Secondly, the control action will no longer be attenuated by the heat exchangers. Simulation shows that the process gain through the heat exchangers is about 0.5 (i.e. increasing the temperature of the oil entering the fin/fan exchanger by one degree results in a 0.5 degree temperature rise in the oil exiting the water cooler). Thus in the present configuration about half of the control action of the heater is lost before it can be applied to the process.

MODELS

1. All models use steady state mass balances.
2. Dynamic energy balances describe the response of the oil in each process unit.
3. The dynamics of the process side of the reactor and the electric heater are modeled.
4. Steady state balances describe the response of the air and water temperatures in the fin/fan and water cooled exchangers respectively.

The expressions used to describe the process side of the reactor are shown below. As can be seen, the reaction was simplified to first order.

$$\begin{aligned} F(X_i - X_o) - R &= 0 \\ R &= K * X_o * \exp(-E/T) \\ \text{EXTENT} &= (X_i - X_o)/X_i \end{aligned}$$

$$\begin{aligned} \text{HEAT GENERATION} &= \text{EXTENT} * \text{HRXN} \\ E &= 23652 \text{ Deg Rankine} \end{aligned}$$

A first order expression is used to describe the dynamics of the oil temperature in the various heat exchangers. The time constant for the oil is shown below.

$$\text{TAU} = (M * C_{p_{\text{oil}}} + M * C_{p_{\text{metal}}}) / (W * C_{p_{\text{oil}}} + UA)$$

RESULTS

The dynamics of three process configurations were studied and are labeled c1, c2, and c3 in the plots that follow. In configurations 1 and 2 the electrical heater is placed before the fin/fan cooler in the flow sheet. In configuration 1 there is no bypass around the fin/fan and the heater is firing at 90% of capacity. In configuration 2 30% of the oil bypasses the fin/fan and the heater fires at 50% of capacity. In configuration 3 the heater is placed between the water cooler and the reactor. A small amount of oil is bypassed around the fin/fan (3%) and the heater fires at 50%.

The first disturbance to the system was a 10000 btu/hr decrease in the reactor heat leak. A controller gain of twenty was used for the proportional controller which manipulated the firing of the heater to maintain the reactor temperature at an arbitrary set point. Figure 1 shows that all three process configurations did an acceptable job maintaining reactor temperature but that configuration 3 was clearly superior. Figure 2 shows that much less control action, 5% of span vs. 10% of span, is needed for configuration 3.

The second disturbance studied was an abrupt 0.5 Deg F change in the reactor temperature set point. Figure 3 shows an overshoot of 80% of the intended change for configuration 1 and 2 and a 15% overshoot for configuration 3. Figure 4 shows again that much less control action after the initial jump is needed to control the reactor for configuration 3.

It should be kept in mind that the disturbances introduced in this study are small. It is highly likely that the control system in the AFDU will need to control much larger upsets than those studied here. Since control action is roughly proportional to the size of disturbance, I expect that in the present process configuration the control action will saturate and control could be lost. The combination of better control with less control action make configuration 3 (heater between water cooler and reactor) a much more robust process choice than configuration 1 or 2 (heater before fin/fan cooler).

DECREASE HEAT LEAK BY
10000 BTU/HR

Fig 1

DECREASE HEAT LEAK BY 10000 BTU/HR

Fig 2

INCREASE SET POINT BY
0.5 Deg F

Fig 3

INCREASE SET POINT BY
0.5 Deg F

Fig 4

APPENDIX B

Mott Cross-flow Filter Test for F-T II

mott

MOTT METALLURGICAL CORPORATION

84 Spring Lane
Farmington Industrial Park
Farmington, CT 06032-3198
Fax: 203-674-1488

Phone: 203-677-7311

Controlled Porosity For Precision Products

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DEC 09 1993

PROCESS ENGINEERING

Laboratory Test Report

Tested

November 16-19, 1993

Objective: Evaluate the flow stability of 0.2 micron porous filter media in the filtration of Fisher-Tropsch catalyst suspended in oil. Inertial filter tests were conducted using the Mott Bench Scale LSX Filter Model 7000-3/8-16". Barrier filtration tests were performed using the Mott 70 mm disc test filter and 0.2 micron media.

Customer: Air Products and Chemicals, Inc.
7201 Hamilton Ave.
Allentown, PA. 18195- 1501

Summary: Stable filtrate fluxes and filtrate clarity were obtained with 0.2 micron media with crossflow as well as barrier filtration.

Filtrate flux rates of about 0.02 gpm/ft² remained stable over a period of 3 days of testing using the Mott Hypulse LSX Inertial (crossflow) filter. Feed slurry temperatures of 140-150°F were critical to maintain these rates. Mainstream velocity was about 6-7 ft/sec with the pressure drop across the element wall ranging from 12-14 PSI. Filtrate was visibly clear within 30 minutes. 1.0 micron media did not produce clear filtrate.

Barrier filtration testing using the Mott 70 mm disc test filter resulted with a filtrate fluxrate of .023 gpm/ft² at 10 PSI. Filtrate was visibly clear. The resulting .093" thick cake was effectively backwashed using 30 PSI N₂.

Further testing is recommended at the customers pilot plant to obtain more accurate sizing data for scale up design.

WO# 41042

Job# 1145-000

FN# 709-2

Sales: KJJ

By:

Ms. Louise L. Stange

Approved:

Mr. Klaus J. Julkowski

* Approval obtained on telephone from Dr. Klaus

Julkowski(Mott) by Dr. Bhatt on 3/31/95 to release this report.

Information contained in this report is confidential to parties involved and should not be released without the consent of Air Products and Mott Metallurgical Corp.

Distribution: LHM, VJP, KJJ, LAB, REP# 004, Air Products

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Phone: 203-677-7311

Controlled Porosity For Precision Products

Laboratory Test Report

Customer: Air Products and Chemicals, Inc. **File #:**709-2
7201 Hamilton Ave.
Allentown, PA 18195-1501

Representative: Dr. Bharat L. Bhatt

MMC Sales Representative: Trist Co. Inc.

Material Tested: (3) 1 gallon samples of "unsupported" iron Fisher-Tropsch catalyst in oil supplied by the University of Kentucky. The catalyst had been deactivated and passivated for the testing .

Sample Disposition: All samples were returned to the University of Kentucky for analysis and disposal.

Summary of the Results: Inertial filter tests were performed using the Mott Hypulse LSX Inertial (cross-flow) filter with a 3/8" OD x 1/4" ID x 16" porous length element (.087 ft² area). The feed was heated to 150 °F. A media life test was conducted by recirculating the filtrate back to the feed tank. The equipment arrangement and test procedure is described on pages 9 & 10.

Unsatisfactory filtrate quality was obtained using 1.0 micron porous media. Initial tests were conducted at a mainstream velocity of 7 ft/sec and a pressure drop across the element wall of 5-7 PSI. Filtrate was visibly cloudy. Filtrate turbidity was 20 NTU. Testing was discontinued after 125 minutes when the filtrate quality did not improve. Evaluation of the particle size range of the feed slurry using the Coulter Multisizer indicated a mean particle size of 3.4 microns.

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Laboratory Test Report

Clear filtrate was obtained using 0.2 micron porous media. Tests at a mainstream velocity of 6-7 FPS and a pressure drop across the element wall ranging from 12-14 PSI resulted with a filtrate flux of about .02 gpm/ft² with slurry temperatures of 140-150°F. Filtrate was visibly clear within 30 minutes. Filtrate turbidity ranged from 0.45 - 20 NTU's. Refer to the graph of flowrate vs. cycle time in Figure 1.

At the conclusion of each day the feed slurry was removed from the tank and the system was flushed with clean oil. The filter was isolated once the oil was flushed thru the system. At start up the next morning the oil was removed and the feed slurry was added to the feed tank and temperatures were gradually increased from ambient to 150°F.

The inertial filter was backwashed once in the 3 days of testing. Backwashing did not significantly increase filtrate flux, which would indicate conditioning of the media.

Barrier filtration tests were conducted using the Mott 70 mm disc test filter and 0.2 micron media using the feed slurry sample isolated at the conclusion of Test 2. Average flux was .023 gpm/ft² running at 10 PSI constant pressure. The resulting .093" thick cake was effectively backwashed using 30 PSI N₂. Filtrate was visibly clear. Refer to Figure 5 for a schematic of equipment arrangement at test procedure.

Conclusions and Recommendations: 0.2 micron porous media is effective in both barrier and crossflow filtration in separating the unsupported iron catalyst from oil. In both cases average filtrate flux was .02 gpm/ft².

Air Products "Unsupported Iron Catalyst" Mott Hypulse LSX Testing using 0.2 um Media (.087 ft2 area)

* Test 2

Figure 1

11/16/93

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Controlled Porosity For Precision Products

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Laboratory Test Report

Discussion of the Results:

Test #1 was run using 1.0 micron porous media. Unsatisfactory filtrate quality was obtained. Testing at a mainstream velocity of 7 ft/sec and a pressure drop across the element wall of 5-7 PSI resulted with filtrate fluxrates of .01 to .02 gpm/ft². Filtrate was visibly cloudy. Filtrate turbidity was 20 NTU. Testing was discontinued after 125 minutes because filtrate quality did not improve.

Evaluation of the particle size range of the feed slurry using the Coulter Multisizer and the 100 micron aperture tube (having a range of 2.16 to 60 microns) indicated that the particle size of the feed ranged from 2.16 - 42 microns, with a mean particle size of 3-4 microns.

Test #2 was conducted using 0.2 micron porous media. Fluxrates of .02 gpm/ft² were obtained within 1 hour of running time, with feed temperatures of 140°F. Pressure drop across the element wall was gradually increased from 6 to 15 PSI. The filter was run for a total of 218 minutes without backwashing. The system was shut down, the feed was drained from the system, and clean oil was used to flush the system. The next morning the oil was removed and the same feed slurry batch from the previous day was used.

The filter was restarted when feed slurry temperatures reached about 100°F. Mainstream velocity was about 6 ft/sec. Pressure drop across the element wall was gradually increased to about 12 PSI. Filtrate was visibly clear within 2 hours. We suspect that some residual solids from Test 1 were flushed from the lines. Fluxrates were about .015 gpm/ft² with feed slurry temperatures of 100-130°F. Fluxrates of 0.2 gpm/ft² were obtained once feed temperatures reached 138-140°F.

After 680 minutes the filter was backwashed (1/2 sec pulse, 60 PSI). After backwash filtrate flux returned to .02 gpm/ft². Filtrate was slightly cloudy, but cleared up within 30 minutes.

A short cycle was run (46 minutes) to determine the effect of lowered velocity. Testing at a mainstream velocity of 5.6 ft/sec and a pressure drop across the element wall of 11-12 PSI resulted with an average flux of .0168 gpm/ft². Feed slurry temperatures ranged from 135-140°F. The filter was shut down using the same procedure as the previous day.

The filter was restarted on 11/18 and within 1 hour fluxrate of .019 gpm/ft² were obtained. After 90 minutes the pressure drop across the element wall was increased from 11.75 to 26 PSI. Mainstream velocity increased to 9 ft/sec. Filtrate flux increased

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Laboratory Test Report

to .03- .04 gpm/ft². We could not maintain this pressure for extended testing because the pump seals were leaking. After 40 minutes the pressure was decreased to 13.5 PSI and the filter was run for about 5 hours with filtrate fluxrates holding at .018 - .02 gpm/ft², with visibly clear filtrate.

At the conclusion of the inertial filter testing the feed slurry was removed from the feed tank. The particle size distribution of the feed was analyzed and compared to the feed at the start of the testing. The results are summarized in Figures 2 & 3. We suspect that the variation from start to finish is in the sampling technique.

Barrier Filtration Testing: Barrier filtration tests were conducted using the Mott 70 mm disc test filter and 0.2 micron media. Feed slurry collected at the conclusion of Test 2 was used in the evaluation. A one liter pressure vessel was used to feed the slurry to the filter at constant pressure. Average flux was .023 gpm/ft². The resulting .093" thick cake was effectively backwashed using 30 PSI N₂. Filtrate was visibly clear. Refer to the schematic and test procedure in Figure 5.

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Controlled Porosity For Precision Products

COULTER(R) Multisizer AccuComp(R) Rev. 1.10
LXUO3.#01

15:13 Tue Nov 16 1993

Filename: LXUO3.#01 Sample Number: 1
Group ID: lxuo3
Sample ID: Feed at start
Comments: cake from gravimetric analysis acetone washed
Operator: LS
Electrolyte: Isoton II
Dispersant: 1A
Aperture Size: 100 um Aperture Current: 1600 uA
Channels: 256 Kd: 991.49
Full Data Log Diameter Gain: 2
Control Method: Siphon 2000 ul
Raw Count: 10733
Coinc. Corr. Count: 10795 Variable 1: 0.000000
Variable 2: 0.000000
Acquired at: 10:49 Tue Nov 16 1993

Number Statistics (Geometric) LXUO3.#01

Calculations from 2.16 um to 42.58 um

Number	10400	Coinc. Corrected:	10795
Mean:	3.440 um	95% Conf. Limits:	3.417-3.464 um
Median:	3.174 um	Std. Dev.:	0.359 um
Mean/Median Ratio:	1.084	Variance:	0.129
Mode:	2.333 um	Coef. Var.:	29.09 %
		Skewness:	1.140e+000 Right skewed
		Kurtosis:	1.559e+000 Leptokurtic

% >	10.00	25.00	50.00	75.00	90.00
Size um	5.650	4.228	3.174	2.589	2.334

COULTER(R) Multisizer AccuComp(R) Rev. 1.10
LXUO3.#01

15:14 Tue Nov 16 1993

Filename: LXUO3.#01 Sample Number: 1
Group ID: lxuo3
Sample ID: Feed at start
Comments: cake from gravimetric analysis acetone washed
Operator: LS
Electrolyte: Isoton II
Dispersant: 1A
Aperture Size: 100 um Aperture Current: 1600 uA
Channels: 256 Kd: 991.49
Full Data Log Diameter Gain: 2
Control Method: Siphon 2000 ul
Raw Count: 10733
Coinc. Corr. Count: 10795 Variable 1: 0.000000
Variable 2: 0.000000
Acquired at: 10:49 Tue Nov 16 1993

Volume Statistics (Geometric) LXUO3.#01

Calculations from 2.16 um to 42.58 um

Volume	563.9*10 ³ um ³	Coinc. Corrected:	10795
Mean:	7.463 um	95% Conf. Limits:	7.451-7.476 um
Median:	7.033 um	Std. Dev.:	0.646 um
Mean/Median Ratio:	1.061	Variance:	0.417
Mode:	20.85 um	Coef. Var.:	32.12 %
		Skewness:	2.765e+001 Right skewed
		Kurtosis:	-7.474e+001 Platykurtic

% >	10.00	25.00	50.00	75.00	90.00
Size um	19.53	11.61	7.033	4.584	3.277

Figure 2

COULTER(R) Multisizer AccuComp(R) Rev. 1.10
 FN7092.#01
 09:57 Mon Nov 29 1993

Filename: FN7092.#01 Sample Number: 1
 Group ID: fn7092
 Sample ID: feed slurry end of testing
 Comments: Air Products Catalyst after 1290 hrs
 Operator: LS
 Electrolyte: Isoton II
 Dispersant: 1A
 Aperture Size: 100 um Aperture Current: 1600 uA
 Channels: 256 Kd: 991.49
 Full Data Log Diameter Gain: 2
 Control Method: Siphon 2000 ul
 Raw Count: 56855
 Coinc. Corr. Count: 58672 Variable 1: 0.000000
 Variable 2: 0.000000
 Acquired at: 09:56 Mon Nov 29 1993

Calculations from		Number Statistics (Geometric)	
Number	2.16 um to 51.70 um	Number	FN7092.#01
Mean	52675	Colinc. Corrected:	58672
Median	4.265 um	95% Conf. Limit:	4.249 4.282 um
Mode	3.975 um	Std. Dev.:	0.452
Mean/Median Ratio:	1.073	Variances:	0.204
	3.299	Coef. Var.:	31.14 %
		Skewness:	8.298e-001 Right skewed
		Kurtosis:	6.058e-001 Leptokurtic
Size um		Size um	
10.00		50.00	
7.965		3.975	
		75.00	
		3.000	
		90.00	
		2.495	

COULTER(R) Multisizer AccuComp(R) Rev. 1.10
 FN7092.#01
 09:57 Mon Nov 29 1993

Filename: FN7092.#01 Sample Number: 1
 Group ID: fn7092
 Sample ID: feed slurry end of testing
 Comments: Air Products Catalyst after 1290 hrs
 Operator: LS
 Electrolyte: Isoton II
 Dispersant: 1A
 Aperture Size: 100 um Aperture Current: 1600 uA
 Channels: 256 Kd: 991.49
 Full Data Log Diameter Gain: 2
 Control Method: Siphon 2000 ul
 Raw Count: 56855
 Coinc. Corr. Count: 58672 Variable 1: 0.000000
 Variable 2: 0.000000
 Acquired at: 09:56 Mon Nov 29 1993

Calculations from		Volume Statistics (Geometric)	
Volume	2.16 um to 51.70 um	Volume	FN7092.#01
Mean	8.120e+106 um	Colinc. Corrected:	58672
Median	12.00 um	95% Conf. Limit:	11.99 12.01 um
Mode	11.70 um	Std. Dev.:	0.687
Mean/Median Ratio:	1.02%	Variances:	0.472
	41.13	Coef. Var.:	27.63 %
		Skewness:	6.812e-002 Right skewed
		Kurtosis:	-6.127e-001 Platykurtic
Size um		Size um	
10.00		50.00	
7.965		11.70	
		75.00	
		7.318	
		90.00	
		4.916	

Figure 3

Laboratory Test Report

Equipment Arrangement: The equipment arrangement is described in Figure 4. The slurry was moved through the filter using a Wilden M-1 Champ diaphragm pump. The mainstream velocity thru the filter was measured using a RCM flowmeter. USG gauges were used to monitor system pressures. Filtrate turbidity was evaluated using a Hach turbidimeter. Manual ball valves were used to isolate the filter for the backwash sequence. The feed tank was agitated using a Stir-pak stirrer at a speed of 1. The feed tank was heated using a Thermolyne hot plate. The system piping was heat traced and insulated to prevent heat loss.

Backwash Technique: The inlet pressure to the filter was lowered to 10 PSI and the filtrate line was closed. The backwash gas valve was opened momentarily using 60 PSIG. The filtrate line was then opened to release trapped gas. The valve was closed and the filter was brought back to dynamic flow conditions.

**MOTT HyPulse™ LSX (INERTIAL) FILTER SYSTEM
SCHEMATIC FLOW DIAGRAM
MANUAL OPERATION**

1. MOTT CAT No. 7000 LSX	9. FILTER PRESSURE GAUGE
2. INLET PRESSURE GAUGE	10. FILTRATE RESERVOIR
3. OUTLET PRESSURE GAUGE	11. FILTRATE CONTROL VALVE
4. INLET CONTROL VALVE	12. FEED TANK
5. OUTLET CONTROL VALVE	13. AGITATOR
6. FLOWMETER	14. FILTRATE FLOWMETER
7. PUMP	15. BACKWASH VALVE
8. PUMP SHUT-OFF VALVE	16. BACKWASH REGULATOR

Figure 4

MOTT LSP/LSX FEASIBILITY TEST SCHEDULE PRESSURE TEST

Equipment

1. One liter reservoir
2. 70mm or 47mm disc fixture
3. Graduated cylinder

Test Procedure

1. Charge system with feed.
2. Run at desired flowrate and collect filtrate. Record filtrate turbidities.
3. Run the filter until the maximum Delta P is reached or the entire volume of feed is filtered.
4. Dewater, remove the upper housing and examine the cake.
5. Pressurize and backwash.
6. Inspect the disc for solids release, reassemble and resume the next cycle.

Figure 5

APPENDIX C

Fischer-Tropsch II Run Authorizations

TEST AUTHORIZATION # 41
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 1 of 4
Date : 05/07/94
By: BLB

RUN NUMBER: AF-A7
APPROX START DATE: 10 May, 1994

TITLE: IN-SITU FISCHER-TROPSCH CATALYST ACTIVATION PRIOR TO SPRING 94 RUN
(F-T II) USING CO & N2

OBJECTIVE:

To activate the Liquid-Phase Fischer-Tropsch (LPFT) synthesis catalyst.

SUMMARY:

Approximately 996 lbs of UCI L-3950 precipitated iron oxide is to be slurried with start-up wax provided by Shell, transferred to the 27.10 reactor and activated with 75% CO and 25 % N2. Approximate run time is 2 days.

TEST DETAILS:

See pages 2 to 4 for details.

ANALYTICAL COMMENTS:

See page 4.

SAFETY IMPLICATIONS:

Operators should wear protective gear while loading catalyst to protect them from the dust and hot vapor which may be released from the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

ENVIRONMENTAL IMPLICATIONS:

A flame will be maintained at the flare.

SPECIAL REMARKS:

Gas composition in and out of the reactor must be monitored closely during the activation. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The temperature difference between the reactor slurry and utility oil in the new 27.10B internal heat exchanger must not exceed 350 deg F. When adjusting flows or pressure, care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST AUTHORIZATION # 41
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 2 of 4
Date: 05/07/94
By: BLB

TEST DETAILS:

1. This activation procedure is based on CAER's bubble column activation of the catalyst (see attached).
2. Charge the 28.30 prep tank with 1132 lb of wax (176 gallons of wax at 80°F). Heat this wax to 150-200°F.
3. Fill the 22.14 intermediate V/L separator with approximately 20 gallons of Ethylflo-164 oil (1 nut on LG-688). Charge the 27.13 with approximately 50 gallons of Ethylflo-164 oil (~ 8.8 % on LIC-203).
4. When the prep tank wax is at 150-200°F, add 996 lb of UCI L-3950 precipitated iron oxide catalyst. Take about 1 lb catalyst sample from each of the four drum. Add the catalyst very slowly to make a 46.8 wt% oxide slurry. Keep the slurry well stirred to prevent agglomeration of the catalyst.
5. Heat the slurry to 200°F and continue agitation, under nitrogen, for at least 2 hours to ensure good mixing.
6. Establish gas flow through the reactor using nitrogen through V-1508 (V-2000 also open) to prevent slurry back-flow into the distributors. Vent the gas through PV-697.
7. Pressure transfer the slurry to the reactor and verify operation by noting level with the nuclear density gauge (NDG).
8. Flush out the prep tank with 136 lb of Ethylflo-164 (20 gallons of Ethylflo-164 at 80°F). Pressure transfer the flush oil to the reactor and verify level with the NDG.
9. Close V-645 to prevent utility oil flow back to the prep tank and establish full utility oil flow through the 27.10B internal heat exchanger.
10. Start 01.10/01.20 compressors with nitrogen. Pressurize the reactor loop to 150 psig. Ensure that the demister outlet (V-1476) is closed.
11. Begin heating the slurry to 302°F over a 6 hr period with a heat up rate of 16°F/hr (no more than 20°F/hr), following T-AVG on the reactor picture on the NextGen console. Check that the slurry temperatures are in reasonable agreement. Verify that the slurry is well mixed by performing a NDG scan. Maintain N₂ flow of 12,530 SCFH (FI-126, FI-187).
12. When the reactor temperature reaches 302°F (150°C), establish the activation gas flow at 20,883 SCFH (on FI-126) and vent the flow through PV-170. Establish the following composition:

TEST AUTHORIZATION # 41
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 3 of 4
Date: 05/07/94
By: BLB

	<u>Composition</u>	<u>Est Flows (SCFH)</u>
CO	75 %	15,662
N2	<u>25 %</u>	<u>5,221</u>
	100 %	20,883

MW = 28.0, SCF evaluated at 70°F, 14.7 psia

13. Wait for GC confirmation, then bring activation gas to the reactor slowly. Establish a final flow to the reactor of 20,883 SCFH. Maintain flow and activation gas composition as specified in step 12. The temperature-programmed activation consists of the following steps:

- * Heat the slurry at a target rate of 12°F/hr (no more than 15°F/hr, 8°C/hr) until the slurry temperature reaches 518°F (270°C). Due to absence of H₂ in the activation gas, Fischer-Tropsch synthesis reaction will not occur during activation. Thus, significant exothermic heat release is not expected until syngas is introduced in the reactor after activation. If the heat up rate is greater than 15°F/hr, reduce CO inlet flow rate and proportionately increase N₂ flow rate.
- * Monitor reactor feed and effluent for CO, CO₂ and N₂ continuously.
- * After reaching 518°F, hold the slurry temperature at 518°F. If the reactor reaches 527°F, reduce the CO inlet flow rate and proportionately increase N₂ flow rate. Process and operations will monitor the effluent composition. If CO₂ level in the effluent increases rapidly, a decision may be made to terminate the activation and change conditions to start process variable study. The hold time at 518°F should not exceed 12 hrs.

14. The slurry level in the reactor should be maintained between 90 and 100% of NDG range using LIC 636. During the activation, the liquid level is expected to fall due loss of lighter components from wax. Line up 22.14 separator to 27.11 and 27.12.

15. Line up liquid flow from 22.18 separator to 22.11 degasser.

16. Record any indication of density or viscosity change, such as a change in the pressure drop across the reactor or shaking of the reactor during heat up and activation.

17. When the activation is completed, scan the reactor with the NDG. Record levels in the 22.11, 22.15, 22.16 and 22.14.

When TA #41 is done, consult TEST AUTHORIZATION #42 for the next step.

TEST AUTHORIZATION # 41
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 4 of 4
Date : 05/07/94
By: BLB

ANALYTICAL REQUIREMENTS:

1. Gas Composition sampling requirements:
 - reactor in and out continuously
2. Flow measurement requirements:
 - reactor in at FI-126 and FI-187.
 - reactor out at FI-701.

REFERENCES:

1. CAER's Bubble Column Activation Results (attached).

CATALYST (UCI-78) ACTIVATION IN CAER BUBBLE COLUMN
(85 PSIG, 6 SL/HR-G FE, 0.14 FT/SEC @ 270 DEG C)

CATALYST (UCI-78) ACTIVATION IN CAER BUBBLE COLUMN
(270 DEG C, 88 PSIG, 6 SL/HR-G FE, 0.13 FT/SEC)

CATALYST (UCI-78) ACTIVATION IN CAER BUBBLE COLUMN
(270 DEG C, 88 PSIG, 6 SL/HR-G FE, 0.13 FT/SEC)

TEST AUTHORIZATION # 42
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 1 of 4
Date : 05/09/94
By: BLB

RUN NUMBER: AF-R11
APPROX START DATE: 12 May, 1994

TITLE: LIQUID PHASE FISCHER-TROPSCH II PROCESS VARIABLE STUDIES WITH UCI
L-3950 IRON OXIDE CATALYST

OBJECTIVE:

To study the performance of UCI L-3950 precipitated iron oxide catalyst in a bubble column reactor at different space velocities and pressures.

SUMMARY:

Upon the activation of the UCI catalyst (TA #41, Run AF-A7), a process variable study will be started. Four different process conditions will be tested for a total of 14 days. This includes operations at three different pressures, three different space velocities as well as a baseline check at the end. The reactor temperature will be varied in the range of 265°C to 300°C to achieve about 80% CO conversion. Following the process variable study, a 1-day tracer study and a 3-day filter test will be conducted.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:

Protective gear including face shield should be worn during slurry sampling and wax transferring/sampling.

ENVIRONMENTAL IMPLICATIONS:

Minimal.

SPECIAL REMARKS:

See Test Details.

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST AUTHORIZATION # 42
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 2 of 4
Date : 05/09/94
By: BLB

TEST DETAILS:

1. Upon completion of the catalyst activation (Run AF-A7), start the process variable study.
2. Decrease the reactor temperature to 464°F (240°C) and increase the pressure to 200 psig. Slowly increase the CO-Rich syngas flow to 25,523 SCFH (on FI-126). Establish the following composition:

	<u>Composition</u>	<u>Est Flows (SCFH)</u>
H2	40 %	10,210
CO	57 %	14,547
N2	3 %	766
	100.0 %	25,523

MW = 17.6, SCF evaluated at 70°F, 14.7 psia

Slowly increase the reactor temperature to 509°F (265°C) to reach conditions for Run AF-R11.1.

3. PVS RUNS:

Process and control room targets are tabulated in Tables 1 and 2. The run descriptors are presented below:

RUN NO.	NO. OF DAYS ON-STREAM	SPACE VEL NL/KG-FE-HR	PRESSURE PSIG	TEMPERATURE DEG C
AF-R11.1	2	2500	200	265
AF-R11.2	2	3000	400	270
AF-R11.3	5	5600	750	300
AF-R11.4	3	5600	400	300
AF-R11.5	2	3000	400	270

4. The reactor slurry level should be maintained between 80 and 100% of NDG range, as specified in the Table 1, using LIC-636 (Oxygenates mode on HS-697). Pump 10.52.02 should be on all the time to bring light wax back to the reactor from 27.11/27.12. Line up 22.14 to 27.11. Maintain 27.12 level using LIC-639. Pump 10.52.01 should also be on all the time. Excess liquid from 22.14 should be transferred in batches to 28.30 periodically. Maintain 22.14 separator between 300-400°F. Maintain 22.18 separator between 90-115°F. Maintain prep tank 28.30 at 250°F.

TEST AUTHORIZATION # 42
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 3 of 4
Date : 05/09/94
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If the catalyst does not behave as expected and the reactor slurry level increases due to significant wax production, then run the reactor in Fischer-Tropsch mode (HS-697). The reactor slurry level should be maintained between 90 and 100% of NDG range using LIC-585. Pump 10.52.02 should be on all the time to bring light wax back to the reactor from 27.13. Slurry level in 27.13 should be maintained at about 37% using LIC-203. Initially, operate LIC-203 in manual mode. Keep pump 10.60 flowing to 22.60 filters. Maintain the 22.60 filter system around 300°F and 65 psig. The 10.60 pump rate should be about 12 gpm. Maintain utility oil flow thru 21.85 heat exchanger. Monitor flow across the 22.60 filter using FI-715. Start with backflush time of 1800 seconds. If FI-715 is showing significant drop off before backflush, process and operations may make decide to increase the back-flush frequency. Maintain 100 psig backflush pressure.

5. Perform 15 min shut down tests, as necessary, to get Nuclear Density Gauge measurements for hold up and catalyst inventory estimate in the reactor. The timing of these tests will be determined by process and operations engineers.

6. SPECIAL CONSIDERATIONS:

Change of Conditions

During change of conditions, small step changes should be made to avoid temperature run-away. Also, change one parameter at a time. While changing to higher productivity conditions (AF-R11.2, 11.3, 11.4) temperature may be reduced and then slowly increased to target condition. The temperature target is a guide line, our actual target is CO conversion level (about 80% in most cases). Consult with process and operations engineers for each change of conditions.

Liquid Transfer

Liquid HC and Aqueous phases collected in day tank 22.16 should be transferred to the HC trailer as 22.16 fills up. The 22.16 liquid gauge should contain a single phase (water). The 22.16 actual level will be higher than the what the gauge indicates. The actual liquid level could be as much as 25% higher than indicated by gauge. Hence, conduct 22.16 transfers before the gauge shows 96". Follow sampling requirements described in analytical comments.

Wax Transfer

Wax from prep tank 28.30 should be transferred to drums every day. Follow sampling requirements described in analytical comments.

7. Upon completion of the PVS, refer to TA # 43 to begin the tracer study.

TEST AUTHORIZATION # 42
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 4 of 4
Date : 05/09/94
By: BLB

ANALYTICAL COMMENTS:

1. Continuous Gas Sampling (GC):
 - Fresh feed and reactor feed,
 - Main Gas Out (22.18 overhead),
2. Periodic Gas Sampling (GC):
 - Purge gas from 22.11
3. Periodic Liquid Sampling:
 - one sample/shift @ 22.11 (two 200 cc bottles)
 - two samples during liquid transfer from 22.16 to trailer
[about 150 cc each, one sample (AQ) @ beginning of transfer and
one sample (HC) @ end of transfer]
4. Periodic Wax Sampling:
 - one Sample/drum (two 20 cc bottles) after transferring wax from
28.30 prep tank to drums
5. Slurry Sampling:
 - 1 sample/day (two 200 cc bottles) @ filter system, if filters are
operated.

RUN PLAN FOR SPRING '94 F-T II RUN - TABLE 1: SUMMARY

RUN	AF-A7	AF-R11.1	R11.2	R11.3	R11.4	R11.5	R11.6A	R11.6B	R11.7
TYPE	ACTVTN	F-T	F-T	F-T	F-T	F-T	TRACER	TRACER	FILTER
DURATION, DAYS	1	2	2	5	3	2	1	1	3
FEED GAS	N ₂ , CO	SG	SG	SG	SG	SG	SG	SG	
H ₂ /CO	0	0.7	0.7	0.7	0.7	0.7	0.7	0.7	
N ₂ , %	25.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
CATALYST LOAD, LBS	996								
WAX CHARGE TO REACTOR, GALS	196								
SPACE VELOCITY, SL/HR-KG FE	2000	2500	3000	5600	5600	3000	2500	5600	
REACTOR									
PRESSURE, PSIG	150	200	400	750	400	400	200	750	
TEMPERATURE, DEG F	518	509	518	572	572	518	509	572	
INLET SUPERFICIAL VEL, FT/SEC	0.38	0.36	0.23	0.24	0.45	0.23	0.36	0.24	
OUTLET SUPERFICIAL VEL, FT/SEC	0.38	0.17	0.11	0.12	0.31	0.11	0.17	0.12	
LIQUID LEVEL, INCHES ON TAPE	174	174	179	192	186	179	174	192	
CATALYST WT FRACTION, %	44	44	44	44	44	44	44	44	
VAPOR VOID FRACTION, %	18-22	15-22	15-24	18-28	21-26	15-24	15-22	18-28	
FLOWS									
FFED GAS, SCFH	20,883	25,523	30,936	58,005	58,005	30,936	25,523	58,005	
PRODUCTS									
PRODUCT GAS, SCFH		10,555	13610	11830	6110	13610	10,555	11830	
HC LIQUID, GPD		496	610	1118	691	610	496	1118	
WATER, GPD		142	175	343	203	175	142	343	
LIGHT WAX, GPD		51	107	287	66	107	51	287	
CO CONVERSION, %		80	80	83	50	80			
CO ₂ SELECTIVITY, MOLE %		45.4	45.4	45.4	45.4	45.4			
CH ₄ SELECT. (CO ₂ FREE), WT %		6.3	6.3	6.3	6.3	6.3			
SUM C ₂ SELECT. (CO ₂ FREE), WT %		9.4	9.4	9.4	9.4	9.4			
SUM C ₃ SELECT. (CO ₂ FREE), WT %		10.5	10.5	10.5	10.5	10.5			
SUM C ₄ SELECT. (CO ₂ FREE), WT %		10.5	10.5	10.5	10.5	10.5			

RUN PLAN FOR SPRING '94 F-T RUN - TABLE 2: CONTROL TARGETS

RUN	AF-A7	AF-R11.1	R11.2	R11.3	R11.4	R11.5	R11.6A	R11.6B	R11.7
TYPE	ACTVTN	F-T	F-T	F-T	F-T	F-T	TRACER	TRACER	FILTER
FEED GAS FLOWS									
CO, SCFH	15,662	14,547	17,634	33,063	33,063	17,634	14,547	33,063	
H ₂ , SCFH	0	10,210	12,374	23,202	23,202	12,374	10,210	23,202	
N ₂ , SCFH	5,221	766	928	1,740	1,740	928	766	1,740	
TOTAL, SCFH	20,883	25,523	30,936	58,005	58,005	30,936	25,523	58,005	
FEED GAS COMPOSITION (MOLE%)									
CO	75	57.0	57.0	57.0	57.0	57.0	57.0	57.0	
H ₂	0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	
N ₂	25.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
PRODUCT GAS - 22.18 OVERHEAD COMPOSITION (MOLE%)									
H ₂		11.2	11.2	8.3	27.4	11.2			
N ₂		6.9	6.9	7.5	4.6	6.9			
CO		26.2	26.2	23.3	43.5	26.2			
CH ₄		3.2	3.2	3.6	1.4	3.2			
CO ₂		46.2	46.2	51.2	20.0	46.2			
SUM C ₂		2.5	2.5	2.7	1.1	2.5			
SUM C ₃		1.8	1.8	1.8	0.8	1.8			
SUM C ₄		1.1	1.1	1.0	0.6	1.1			
SUM C ₅		0.6	0.6	0.5	0.4	0.6			
SUM C ₆		0.2	0.2	0.2	0.2	0.2			
SUM C ₇ + C ₈		0.04	0.04	0.04	0.04	0.04			
SUM C ₉ + C ₁₀		0.003	0.003	0.004	0.003	0.003			
22.14 SEPARATOR TEMP., DEG F		300-400	300-400	300-400	300-400	300-400	300-400	300-400	
22.18 SEPARATOR TEMP., DEG F		90-115	90-115	90-115	90-115	90-115	90-115	90-115	
10.60 PUMP FLOW RATE, GPM									12
22.60 FILTER INLET PRESS., PSIG									65
21.85 HT EXCH OUTLET TEMP., DEG F									300

TEST AUTHORIZATION # 43
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 1 of 4
Date : 05/08/94
By: BLB

RUN NUMBER: AF-R11.6
APPROX START DATE: 26 May, 1994

TITLE: TRACER STUDY FOR LIQUID PHASE FISCHER-TROPSCH II
DEMONSTRATION RUN WITH UCI L-3950 IRON OXIDE CATALYST

OBJECTIVE:

To conduct a 1-day radioactive tracer study for the Fischer-Tropsch (II) synthesis run in a bubble column reactor.

SUMMARY:

A slurry of UCI precipitated iron oxide catalyst (L-3950) and start-up wax from Shell will be activated in the reactor (Test Authorization # 41). Upon completion of the activation, a process variable study will be conducted (Test Authorization # 42). A one day tracer study will follow the process variable study. Radioactive gas and liquid tracers will be injected at two process conditions to study the mixing in both the phases. A 3-day filter test will follow the tracer study.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:

Safety information available from ICI Tracerco is attached (Letters from D. A. Bucior to B. L. Bhatt, "Radiation Safety Analysis of Proposed Methanol Reactor Residence Time and Distribution Study", 3 June 1993 and "Radiation Safety Analysis of Proposed LaPorte Pilot Plant Radioactive Tracer Study", 10 February 1994.). Barricades will be erected by ICI Tracerco to prevent access to areas containing radioactive materials. Radiation film badges will be worn by all personnel present during the study.

We will have to allow 10 hours to elapse after the last liquid injection prior to draining the wax. Assuming a worst case scenario of all radioactive material depositing on one filter, 36 hours will be required after injection for the material to decay to the regulatory limit. Hence, no maintenance on the filters will be permitted for 36 hours after the last liquid injection.

ENVIRONMENTAL IMPLICATIONS:

A flame will be maintained at the flare.

SPECIAL REMARKS:

The radioactive tracer injection will be performed by ICI Tracerco personnel using their injection equipment. APCI personnel will be present during the injection and operate AFDU equipment.

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST AUTHORIZATION # 43

LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 2 of 4

Date : 05/08/94

By: BLB

TEST DETAILS:

1. Upon completion of the Process Variable Study (Run AF-R11.1 thru 11.5), start the Tracer Study. A vapor residence time distribution study will be performed by injecting Argon-41 into the inlet gas line and monitoring its progress through the reactor by several detectors. Two different gas velocities will be tested. Also, two injections of radioactive Mn_2O_3 will be made in the reactor slurry at each condition to study liquid phase mixing.

ICI Tracerco is licensed to conduct these tests and will conform to guidelines prescribed by the Texas Department of Health. Texas A&M will irradiate the Argon and Manganese on the morning of the tests, and it will be delivered to the LaPorte site by courier. The radioactive Ar-41 has a half life of 1.8 hrs and will be injected into the reactor and vented to the atmosphere in levels acceptable to the Texas Department of Health. The radioactive manganese is expected to have a half life of 2.5 hours; only small amounts acceptable to the Texas Department of Health will be injected. An irradiation test will be performed on Mn_2O_3 by Texas A&M to ensure that the radiation will decay to very low levels within several days.

2. Operating conditions of Run Nos. AF-R11.1 and 11.3 will be studied. Process and control targets for the study are tabulated in Table 1 and 2. The run descriptors are presented below:

RUN NO.	INJECTIONS	SPACE VEL SL/HR - KG FE	PRESSURE PSIG	TEMPERATURE DEG C	INLET GAS VEL, FT/SEC
AF-R11.6A	GAS-INLET (2)	2500	200	265	0.36
AF-R11.6A	LIQUID-TOP (1)	2500	200	265	0.36
AF-R11.6A	LIQUID-BOTTOM (1)	2500	200	265	0.36
AF-R811.6B	GAS-INLET (2)	5600	750	300	0.24
AF-R11.6B	LIQUID-TOP (1)	5600	750	300	0.24
AF-R11.6B	LIQUID-BOTTOM (1)	5600	750	300	0.24

3. The slurry level should be maintained between 80 and 100% of NDG range, as specified in Table 1. Leave the Nuclear Density Gauge at the normal controlling reactor height. Shut off the gauge for 10-15 minutes during the injections as cross-interference is

TEST AUTHORIZATION # 43
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 3 of 4
Date : 05/08/94
By: BLB

expected. Put LIC-636 on manual during the injections but bring it back on auto between runs. Pump 10.52.02 should be on all the time to bring light wax back to the reactor. Pump 10.52.01 should be on all the time to circulate light wax thru 22.14. Maintain 22.14 separator between 300-400°F. Maintain 22.18 separator between 90-115°F.

4. Day 1 (Approx. Date: 5/26/94)

Preparation and initial testing of the equipment will be conducted on the last day of the Process Variable Study. The electronic equipment includes 19 detectors connected to a data acquisition system. The process equipment includes a gas sample cylinder with adequate valves to allow filling of the cylinder with Ar-41 and subsequent injection into the reactor via a nitrogen flush. The Ar-41 will be injected thru valve V-1504. The radioactive manganese will be sluried in Ethylflo-164 and injected with a hand-powered piston pump. The liquid injections will be made at two locations on the side of the reactor: Top T-nozzle and Bottom T-nozzle.

During day 1, the electronic equipment will be connected and power supplied to it. A preliminary calibration will be performed to verify the equipment is operational. Arrangements will be made to support the detectors in their proper location, detectors will not be placed until day 2.

Personnel available during the study will include two persons from Tracerco, one operator, one PSG process engineer and the plant manager. A catalyst inventory will be determined during day 1 using the nuclear density gauge with no gas flow through the reactor.

Day 2 (Approx. Date: 5/27/94)

Prior to day 2, arrangements will be made to irradiate Ar-41 and Mn_2O_3 in a reactor at Texas A&M. The irradiation will take place on the morning of day 2 and be transported to LaPorte by 1 pm. The radioactive materials produced during day 2 can only be used during this day since the half life of these compounds is less than three hours.

During the morning of day 2, the Tracerco crew will calibrate and hang the 19 detectors at the LaPorte AFUDU. Each detector will be subjected to a gamma-ray source, and the response will be measured. All the detectors will then be normalized relative to the most sensitive detector.

After the calibration is complete, the detectors will be placed at specified locations. Two conditions will be studied during the day. Due to change in gas holdup for each velocity, the slurry level will change for a constant slurry concentration and catalyst inventory. The Ar-41 injections will be made into the feed gas. For each case, two injections will be made. A reasonable amount of time must exist between injections so that either Ar-41 has left the system or a steady level of radiation is available to use as a baseline. Two liquid injections (one at top and one at bottom) will be made at each condition.

TEST AUTHORIZATION # 43
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 4 of 4
Date : 05/08/94
By: BLB

At the beginning and end of each condition, liquid level, gas hold up and slurry concentration will be measured with the nuclear density gauge (NDG). Two sets of detectors below the liquid level will have to be removed for the NDG measurements.

After completing the study, Tracerco will remove their equipment from the LaPorte site.

5. Upon completion of the Tracer Study, the plant will be readied for a 3-day filter test.

ANALYTICAL COMMENTS:

Since the gas, liquid and wax will contain radioactive materials, NO sampling will be done during the tracer study.

RUN PLAN FOR SPRING '94 F-T II RUN - TABLE 1: SUMMARY

RUN	AF-A7	AF-R11.1	R11.2	R11.3	R11.4	R11.5	R11.6A	R11.6B	R11.7
TYPE	ACTVTN	F-T	F-T	F-T	F-T	F-T	TRACER	TRACER	FILTER
DURATION, DAYS	1	2	2	5	3	2	1	1	3
FEED GAS	N ₂ , CO	SG	SG	SG	SG	SG	SG	SG	
H ₂ /CO	0	0.7	0.7	0.7	0.7	0.7	0.7	0.7	
N ₂ , %	25.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
CATALYST LOAD, LBS	996								
WAX CHARGE TO REACTOR, GALS	196								
SPACE VELOCITY, SL/HR-KG FE	2000	2500	3000	5600	5600	3000	2500	5600	
REACTOR									
PRESSURE, PSIG	150	200	400	750	400	400	200	750	
TEMPERATURE, DEG F	518	509	518	572	572	518	509	572	
INLET SUPERFICIAL VEL, FT/SEC	0.38	0.36	0.23	0.24	0.45	0.23	0.36	0.24	
OUTLET SUPERFICIAL VEL, FT/SEC	0.38	0.17	0.11	0.12	0.31	0.11	0.17	0.12	
LIQUID LEVEL, INCHES ON TAPE	174	174	179	192	186	179	174	192	
CATALYST WT FRACTION, %	44	44	44	44	44	44	44	44	
VAPOR VOID FRACTION, %	18-22	15-22	15-24	18-28	21-26	15-24	15-22	18-28	
FLOWS									
FFED GAS, SCFH	20,883	25,523	30,936	58,005	58,005	30,936	25,523	58,005	
PRODUCTS									
PRODUCT GAS, SCFH		10,555	13610	11830	6110	13610	10,555	11830	
HC LIQUID, GPD		496	610	1118	691	610	496	1118	
WATER, GPD		142	175	343	203	175	142	343	
LIGHT WAX, GPD		51	107	287	66	107	51	287	
CO CONVERSION, %		80	80	83	50	80			
CO ₂ SELECTVITY, MOLE %		45.4	45.4	45.4	45.4	45.4			
CH ₄ SELECT. (CO ₂ FREE), WT %		6.3	6.3	6.3	6.3	6.3			
SUM C ₂ SELECT. (CO ₂ FREE), WT %		9.4	9.4	9.4	9.4	9.4			
SUM C ₃ SELECT. (CO ₂ FREE), WT %		10.5	10.5	10.5	10.5	10.5			
SUM C ₄ SELECT. (CO ₂ FREE), WT %		10.5	10.5	10.5	10.5	10.5			

RUN PLAN FOR SPRING '94 F-T RUN - TABLE 2: CONTROL TARGETS

RUN	AF-A7	AF-R11.1	R11.2	R11.3	R11.4	R11.5	R11.6A	R11.6B	R11.7
TYPE	ACTVTN	F-T	F-T	F-T	F-T	F-T	TRACER	TRACER	FILTER
FEED GAS FLOWS									
CO, SCFH	15,662	14,547	17,634	33,063	33,063	17,634	14,547	33,063	
H ₂ , SCFH	0	10,210	12,374	23,202	23,202	12,374	10,210	23,202	
N ₂ , SCFH	5,221	766	928	1,740	1,740	928	766	1,740	
TOTAL, SCFH	20,883	25,523	30,936	58,005	58,005	30,936	25,523	58,005	
FEED GAS COMPOSITION (MOLE%)									
CO	75	57.0	57.0	57.0	57.0	57.0	57.0	57.0	
H ₂	0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	
N ₂	25.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
PRODUCT GAS - 22.18 OVERHEAD COMPOSITION (MOLE%)									
H ₂		11.2	11.2	8.3	27.4	11.2			
N ₂		6.9	6.9	7.5	4.6	6.9			
CO		26.2	26.2	23.3	43.5	26.2			
CH ₄		3.2	3.2	3.6	1.4	3.2			
CO ₂		46.2	46.2	51.2	20.0	46.2			
SUM C ₂		2.5	2.5	2.7	1.1	2.5			
SUM C ₃		1.8	1.8	1.8	0.8	1.8			
SUM C ₄		1.1	1.1	1.0	0.6	1.1			
SUM C ₅		0.6	0.6	0.5	0.4	0.6			
SUM C ₆		0.2	0.2	0.2	0.2	0.2			
SUM C ₇ + C ₈		0.04	0.04	0.04	0.04	0.04			
SUM C ₉ + C ₁₀		0.003	0.003	0.004	0.003	0.003			
22.14 SEPARATOR TEMP., DEG F									
22.18 SEPARATOR TEMP., DEG F									
10.60 PUMP FLOW RATE, GPM									
22.60 FILTER INLET PRESS., PSIG									
21.85 HT EXCH OUTLET TEMP., DEG F									
									12
									65
									300

Tracerco

June 3, 1993

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JUN 07 1993

-PROCESS ENGINEERING

Tracerco

Suite 200
1100 Hercules
Houston, TX 77058

Telephone (713) 488-0039
Fax (713) 488-1646

Mr. Bharat Bhatt
Air Products and Chemicals
7201 Hamilton Blvd.
Allentown, PA 18195

**RE: RADIATION SAFETY ANALYSIS OF PROPOSED METHANOL REACTOR RESIDENCE
TIME AND DISTRIBUTION STUDIES**

Dear Mr. Bhatt:

The proposed radiotracer fluid distribution studies of the Methanol Reactor will be performed under ICI Tracerco's Texas Radioactive Materials License, L03096. I have included a copy of our current license for your files.

ICI Tracerco operates within strict guidelines, established by the Texas Bureau of Radiation Control, regarding how radioactive materials are to be handled, how much activity may be injected into the process system, exposure limits for non-radiation workers, and barricades around the area in which radiation is being used.

By regulation, barricades must be posted such that radiation exposure to non-radiation workers will not exceed 2 millirem in any 1 hour and/or 100 millirem in any seven consecutive seven days. Radiation workers, such as Tracerco employees, are limited to exposures of 1250 millirem per calendar quarter.

ICI Tracerco's operating standards are considerably higher than those required by law. We operate under the ALARA radiation principal. ALARA, simply put, states the any radiation exposure will be limited to As Low As Reasonably Achievable. An example of this philosophy in action is that should a Tracerco employee receive 1/10th the acceptable regulatory limit in a calendar quarter, an internal investigation will be performed to determine the cause of the "excessive" exposure. As you will appreciate, the principals of ALARA are equally applicable to possible radiation exposures of non-radiation workers. During our on-site investigation, we establish our radiation barricades such that possible exposures of non-radiation workers would be considerably less than legally allowable.

At Air Products request, radiation dosimetry was provided for each non-radiation worker on the plant site during the 1989 studies. Included is a copy of the radiation exposure analysis. There was no recordable radiation exposure to any plant employees during the study.

The other potential concern regarding the radiation safety of the project addresses the allowable concentration of the residual activity of the radiotracers and environmental impact. All accounting and

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disposal of radioactive materials are accomplished under provisions of ICI Tracerco's License. Again, ICI Tracerco is guided by regulations established by the State of Texas. (Incidentally, the applicable concentration limits are identical to those established as "fit" for human consumption.)

Two radiotracers will be used during the studies of the reactor. Vapor phase studies will be performed using Ar-41, an inert gas with a half-life of 1.8 hours. The liquid phase studies will be performed using Mn-56. Mn-56 has a half-life of 2.5 hours.

The Ar-41 radiotracer will vent the system via a 35 foot tall stack downstream of the reactor. Texas regulations allow an Ar-41 disposable concentration of 4×10^{-8} uCi/mL. This equates to an allowable injection of 2880 mCi per 8 hour day. The actual amount of radiotracer required during the previous studies was approximately 1/10 the allowable limit.

Disposal of the Mn-56 liquid tracer will be accomplished via dilution of the radiotracer within the 550 gallon liquid inventory and then via decay. Texas regulations allow a Mn-56 concentration of 3×10^{-3} uCi/mL. A strict dilution into product inventory allows injection of 6.2 mCi. The 1989 project required injection of 3 mCi Mn-56. When decay of the radioisotope is factored into the equation, the actual radiotracer concentration is considerably lower. For instance assuming 6.2 mCi were injected into the system, in 24 hours the actual concentration will be 3.8×10^{-6} uCi/mL, or 1000 times lower than the applicable regulatory limit.

I hope that I have addressed all your concerns. Please contact us if you need further information.

During the 1989 study, Air Produces provided "Manganese Oxide of a proper particle size", which we then irradiated and mixed in solution to provide the liquid radiotracer. I have limited details of the base stock, particularly regarding particle size. Can you look into providing a sample. We would want to perform a test irradiation prior to the project to insure proper decay and no undesirable by-products. I will continue researching our records as well.

Sincerely,

A handwritten signature in black ink, appearing to read 'David A. Bucior', with a long horizontal line extending to the right.

David A. Bucior
Senior Project Leader

DAB/jls

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Tracerco

CC ECH
BAT

February 10, 1994

Dr. Bharat L. Bhatt
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RE: RADIATION SAFETY ANALYSIS OF PROPOSED LAPORTE PILOT PLANT
RADIOTRACER STUDY

Dear Dr. Bhatt:

On January 25, 1994, a preliminary safety analysis associated with performing a radiotracer diagnostic flow distribution study of the reactor in the La Porte Pilot Plant was held. Present at the meeting were Dr. Bharat Bhatt, Dr. Bernie Tosland, Edward Heydorn and myself.

Three areas of concern were identified which required further investigation. These included worst case scenarios where all catalyst radiotracer injected would accumulate in 1 filter or ended up in the product stream exiting the column. The third concern was the amount of vapor phase tracer which could be absorbed into the catalyst slurry.

The calculations presented in this text are based upon consuming an identical amount of radiotracer as during a study of this equipment performed in August 1993. The catalyst radiotracer was Mn-56 with has a half-life of 2.5 hours. The vapor radiotracer was Ar-41 with a half-life of 1.8 hours.

Reactor Slurry & Product Stream

During the August studies 2 mCi of Mn-56 were consumed during each days testing. Given the reactor volume of 550 gallon the radiotracer concentration upon mixing with the reactor volume:

$$\frac{2 \text{ mCi}}{550 \text{ Gal}} * \frac{1 \text{ Gal}}{3,785 \text{ ml}} * \frac{1000 \text{ } \mu\text{Ci}}{1 \text{ mCi}} = 9.6 \text{ E-04 } \mu\text{Ci/ml}$$

The maximum concentration at which the general public may contact Mn-56 allowable under Texas Regulations is 7 E-05 $\mu\text{Ci/ml}$. The previous study showed considerable mixing occurring in the reactor. Since the product draw is located so high on the reactor I believe it safe to assume that product stream concentration would not be greater than the mixed inventory concentration.

It will be necessary to allow 10 hours to elapse after test completion prior to allowaing non-radiation personnel to come into direct contact

Dr. Bharat L. Bhatt
Air Products and Chemicals, Inc.
Page 2

with the reactor slurry and/or product stream. This will allow the injected material to have passed through 4 half-lives. After 10 hours decay the Mn-56 concentration will be $6 \text{ E-05 } \mu\text{Ci/ml}$.

Filter

Four filters are in place on the slurry stream. The following analysis is based upon all Mn-56 injected short circuiting the reactor and collecting in one filter. It is assumed that the radiotracer material is deposited on the filter in combination with a $1/8$ " thick "cake" layer of density 70 lbs/ft^3 . This would result in an accumulation of 193.17 grams of material. Thus the initial concentration would be:

$$\frac{2 \text{ mCi}}{193.17 \text{ g}} * \frac{1000 \mu\text{Ci}}{\text{mCi}} = 10.35 \mu\text{Ci/g}$$

Texas Regulations establish a concentration of $7 \text{ E-04 } \mu\text{Ci/ml}$ for solid Mn-56 as acceptable for contact by the general public. Therefore, if all material was deposited in one filter, 36 hours would be required (after injection) for the material deposited on the filters to decay to the regulatory limit. If all tracer material were trapped on four filters, 31 hours would be required (after injection) to decay to the regulator limit.

In an alternate scenario, after 4 hours of circulation (inventory turnover time), 4 hours of filter cooling time, and all four filters in operation; the filters would be acceptable for immediate handling provided less than 2.5 percent of the total tracer injected was trapped on the filters.

Vapor Tracer Solubility

A final concern was the potential for Ar-41, the vapor phase tracer, being absorbed into the catalyst slurry stream. I have insufficient information to calculate a realistic Argon absorption in the reactor. During each process rate study approximately 0.025 moles of Argon with a radiation activity of 250 mCi will be consumed. If you can calculate what percentage of the injected material might be absorbed by the process stream, the radiation concentration would be in an identical ratio. The available information indicates that Argon absorption, if any, would be minimal.

According to D. Vermeer and R. Krishna's "Hydrodynamics and Mass Transfer in Bubble Columns Operating in the Churn-Turbulent Regime", we may expect some Argon absorption. Figure 1 of the paper showed an Argon mole fraction of 0.00235 dissolved in turpentine 5 at 1 atmosphere. I assume this represented a saturation concentration. To saturate 550 gallons of slurry, we would have to inject 31 moles of argon.

A more practical representation was presented in Figure 4, which showed Residence Time Distributions (pulse injections) with tracer

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Dr. Bharat L. Bhatt
Air Products and Chemicals, Inc.
Page 3

gases of varying solubility. During these studies Argon cleared the test vessel in approximately 30 seconds.

This was also the observation during the previous studies of the reactor. If any argon was absorbed, its' concentration was lower than the detectable limits using our extremely sensitive equipment.

Argon is not typically recognized as being a soluble material. Texas Regulations for the Control of Radioactive Materials provide no concentration limits for Ar-41 being in anything other than a vapor phase, i.e.; there are no regulatory provisions which address Argon being in either a solution or solid mixture.

I trust that I have addressed your concerns. Please contact us if we may be of further service.

Sincerely,

A handwritten signature in cursive script, which appears to read 'David A. Bucior', is written over the typed name.

David A. Bucior
Senior Project Leader

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TEST AUTHORIZATION # 44
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 1 of 4
Date: 05/26/94
By: BLB

RUN NUMBER: AF-A8
APPROX START DATE: 26 May, 1994

TITLE: IN-SITU FISCHER-TROPSCH CATALYST ACTIVATION PRIOR TO SPRING 94 RUN
(F-T IIA) USING CO & N2

OBJECTIVE:

To activate the Liquid-Phase Fischer-Tropsch (LPFT) synthesis catalyst.

SUMMARY:

Approximately 480 lbs of UCI L-3950 precipitated iron oxide is to be slurried with Drakeol-10, transferred to the 27.10 reactor and activated with 75% CO and 25 % N2. Approximate run time is 1 day.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:

Operators should wear protective gear while loading catalyst to protect them from the dust and hot vapor which may be released from the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

ENVIRONMENTAL IMPLICATIONS:

A flame will be maintained at the flare.

SPECIAL REMARKS:

Gas composition in and out of the reactor must be monitored closely during the activation. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The temperature difference between the reactor slurry and utility oil in the new 27.10B internal heat exchanger must not exceed 350 deg F. When adjusting flows or pressure, care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST AUTHORIZATION # 44
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 2 of 4
Date : 05/27/94
By: BLB

TEST DETAILS:

1. This activation procedure is based on CAER's activation of the catalyst (see attached).
2. Charge the 28.30 prep tank with 947 lb of Drakeol-10 (135 gallons of wax at 80°F). Heat the oil to 150-200°F.
3. Fill the 22.14 intermediate V/L separator with approximately 20 gallons of Drakeol-10 oil (1 nut on LG-688). Charge the 27.13 with approximately 50 gallons of Drakeol-10 oil (~ 8.8 % on LIC-203).
4. When the prep tank wax is at 150-200°F, add 480 lb of UCI L-3950 precipitated iron oxide catalyst. Take about 1 lb catalyst sample from each of the two drums. Add the catalyst very slowly to make a 33.6 wt% oxide slurry. Keep the slurry well stirred to prevent agglomeration of the catalyst.
5. Heat the slurry to 250°F and continue agitation, under nitrogen, for at least 2 hours to ensure good mixing.
6. Establish gas flow through the reactor using nitrogen through V-1508 (V-2000 also open) to prevent slurry back-flow into the distributors. Vent the gas through PV-697.
7. Pressure transfer the slurry to the reactor and verify operation by noting level with the nuclear density gauge (NDG).
8. Flush out the prep tank with 228 lb of Drakeol-10 (33 gallons of Drakeol-10 at 80°F). Pressure transfer the flush oil to the reactor and verify level with the NDG.
9. Close V-645 to prevent utility oil flow back to the prep tank and establish full utility oil flow through the 27.10B internal heat exchanger.
10. Start 01.10/01.20 compressors with nitrogen. Pressurize the reactor loop to 150 psig. Ensure that the demister outlet (V-1476) is closed.
11. Begin heating the slurry to 428°F over a 6 hr period with a heat up rate of 30°F/hr (no more than 35°F/hr), following T-AVG on the reactor picture on the NextGen console. Check that the slurry temperatures are in reasonable agreement. Verify that the slurry is well mixed by performing a NDG scan. Maintain N₂ flow of 10,000 SCFH (FI-126, FI-187).
12. When the reactor temperature reaches 428°F (220°C), establish the activation gas flow at 9,822 SCFH (on FI-126) and vent the flow through PV-170. Establish the following composition:

TEST AUTHORIZATION # 44
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 3 of 4
Date: 05/26/94
By: BLB

	<u>Composition</u>	<u>Est Flows (SCFH)</u>
CO	75 %	7,367
N2	<u>25 %</u>	<u>2,455</u>
	100 %	9,822

MW = 28.0, SCF evaluated at 70°F, 14.7 psia

13. Wait for GC confirmation, then bring activation gas to the reactor slowly. Establish a final flow to the reactor of 9,822 SCFH. Maintain flow and activation gas composition as specified in step 12. The temperature-programmed activation consists of the following steps:

- * Heat the slurry at a target rate of 30°F/hr (no more than 35°F/hr, 19°C/hr) until the slurry temperature reaches 518°F (270°C). Due to absence of H₂ in the activation gas, Fischer-Tropsch synthesis reaction will not occur during activation. Thus, significant exothermic heat release is not expected until syngas is introduced in the reactor after activation. If the heat up rate is greater than 35°F/hr, reduce CO inlet flow rate and proportionately increase N₂ flow rate.
- * Monitor reactor feed and effluent for CO, CO₂ and N₂ continuously.
- * After reaching 518°F, hold the slurry temperature at 518°F. If the reactor reaches 527°F, reduce the CO inlet flow rate and proportionately increase N₂ flow rate. Process and operations will monitor the effluent composition. If CO₂ level in the effluent increases rapidly, a decision may be made to terminate the activation and change conditions to start process variable study. The hold time at 518°F should not exceed 12 hrs.

14. The slurry level in the reactor should be maintained between 90 and 100% of NDG range using LIC 636. During the activation, the liquid level is expected to fall due loss of lighter components from the oil. Line up 22.14 separator to 27.11 and 27.12.

15. Line up liquid flow from 22.18 separator to 22.11 degasser.

16. Record any indication of density or viscosity change, such as a change in the pressure drop across the reactor or shaking of the reactor during heat up and activation.

17. When the activation is completed, scan the reactor with the NDG. Record levels in the 22.11, 22.15, 22.16 and 22.14.

When TA #44 is done, consult TEST AUTHORIZATION #45 for the next step.

TEST AUTHORIZATION # 44
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 4 of 4
Date : 05/27/94
By: BLB

ANALYTICAL REQUIREMENTS:

1. Gas Composition sampling requirements:
 - reactor in and out continuously
2. Flow measurement requirements:
 - reactor in at FI-126 and FI-187.
 - reactor out at FI-701.

REFERENCES:

1. CAER's Activation Results (attached).

CAER AUTOCLAVE ACTIVATION

- C₃₀ Oil/100% Theoretical CO₂
- Polyethylene Wax/50% Theoretical CO₂
- Polyethylene Wax/100% Theoretical CO₂

TEST AUTHORIZATION # 45
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 1 of 4
Date : 05/26/94
By: BLB

RUN NUMBER: AF-R12
APPROX START DATE: 27 May, 1994

TITLE: LIQUID PHASE FISCHER-TROPSCH IIA PROCESS VARIABLE STUDIES WITH UCI
L-3950 IRON OXIDE CATALYST

OBJECTIVE:

To study the performance of UCI L-3950 precipitated iron oxide catalyst in a bubble column reactor at different space velocities and pressures.

SUMMARY:

Upon the activation of the UCI catalyst (TA #44, Run AF-A8), a process variable study will be started. Four different process conditions will be tested for a total of 11 days. This includes operations at three different pressures, two different space velocities as well as a baseline check at the end. The reactor temperature will be varied in the range of 270°C to 300°C to achieve about 80% CO conversion. Following the process variable study, a 1-day tracer study and a 2-day filter test will be conducted.

TEST DETAILS: See pages 2 to 4 for details.

ANALYTICAL COMMENTS: See page 4.

SAFETY IMPLICATIONS:

Protective gear including face shield should be worn during slurry sampling and wax transferring/sampling.

ENVIRONMENTAL IMPLICATIONS:

Minimal.

SPECIAL REMARKS:

See Test Details.

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST AUTHORIZATION # 45
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 2 of 4
Date : 05/26/94
By: BLB

TEST DETAILS:

1. Upon completion of the catalyst activation (Run AF-A8), start the process variable study.
2. Slowly increase the H₂ flow to achieve the total CO-Rich syngas flow to 11,794 SCFH (on FI-126). Establish the following composition:

	<u>Composition</u>	<u>Est Flows (SCFH)</u>
H2	40 %	4,718
CO	57 %	6,722
N2	3 %	354
	100.0 %	11,794

MW = 17.6, SCF evaluated at 70°F, 14.7 psia

Slowly increase the reactor pressure to 175 psig to reach conditions for Run AF-R12.1.

3. PVS RUNS:

Process and control room targets are tabulated in Tables 1 and 2. The run descriptors are presented below:

RUN NO.	NO. OF DAYS ON-STREAM	SPACE VEL NL/KG-FE-HR	PRESSURE PSIG	TEMPERATURE DEG C
AF-R12.1	2	2400	175	270
AF-R12.2	2.5	11700	750	300
AF-R12.3	2.5	11700	750	300
AF-R12.4	2	11700	500	300
AF-R12.5	2	2400	175	270

4. The reactor slurry level should be maintained between 90 and 100% of NDG range, as specified in the Table 1, using LIC-636 (Oxygenates mode on HS-697). Pump 10.52.02 should be on all the time to bring light wax/Drakeol-10 back to the reactor from 27.11/27.12. Line up 22.14 to 27.11. Maintain 27.12 level using LIC-639. Pump 10.52.01 should also be on all the time. Excess liquid from 22.14 should be transferred in batches to 28.30 periodically. Maintain 22.14 separator temperature as specified in Table 2. Maintain 22.18 separator between 90-115°F. Maintain prep tank 28.30 at 250°F.

TEST AUTHORIZATION # 45
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 3 of 4
Date: 05/26/94
By: BLB

If the catalyst does not behave as expected and the reactor slurry level increases due to significant wax production, then run the reactor in Fischer-Tropsch mode (HS-697). The reactor slurry level should be maintained between 90 and 100% of NDG range using LIC-585. Pump 10.52.02 should be on all the time to bring light wax back to the reactor from 27.13. Slurry level in 27.13 should be maintained at about 37% using LIC-203. Initially, operate LIC-203 in manual mode. Keep pump 10.60 flowing to 22.60 filters. Maintain the 22.60 filter system around 300°F and 65 psig. The 10.60 pump rate should be about 12 gpm. Maintain utility oil flow thru 21.85 heat exchanger. Monitor flow across the 22.60 filter using FI-715. Start with backflush time of 1800 seconds. If FI-715 is showing significant drop off before backflush, process and operations may make decide to increase the back-flush frequency. Maintain 100 psig backflush pressure.

5. Perform 15 min shut down tests, as necessary, to get Nuclear Density Gauge measurements for hold up and catalyst inventory estimate in the reactor. The timing of these tests will be determined by process and operations engineers.
6. SPECIAL CONSIDERATIONS:

Change of Conditions

During change of conditions, small step changes should be made to avoid temperature run-away. Also, change one parameter at a time. While changing to higher productivity conditions (AF-R12.2) temperature may be reduced and then slowly increased to target condition. The temperature target is a guide line, our actual target is CO conversion level. Consult with process and operations engineers for each change of conditions.

Liquid Transfer

Liquid HC and Aqueous phases collected in day tank 22.16 should be transferred to the HC trailer as 22.16 fills up. The 22.16 liquid gauge should contain a single phase (water). The 22.16 actual level will be higher than what the gauge indicates. The actual liquid level could be as much as 25% higher than indicated by gauge. Hence, conduct 22.16 transfers before the gauge shows 96". Follow sampling requirements described in analytical comments.

Wax Transfer

Wax from prep tank 28.30 should be transferred to drums every day. Follow sampling requirements described in analytical comments.

7. Upon completion of the PVS, refer to TA # 46 to begin the tracer study.

TEST AUTHORIZATION # 45
LaPorte Alternative Fuels Development Unit (AFDU)

Sheet: 4 of 4
Date : 05/26/94
By: BLB

ANALYTICAL COMMENTS:

1. Continuous Gas Sampling (GC):
 - Fresh feed and reactor feed,
 - Main Gas Out (22.18 overhead),
2. Periodic Gas Sampling (GC):
 - Purge gas from 22.11
3. Periodic Liquid Sampling:
 - one sample/shift @ 22.11 (two 200 cc bottles)
 - two samples during liquid transfer from 22.16 to trailer
[about 150 cc each, one sample (AQ) @ beginning of transfer and
one sample (HC) @ end of transfer]
4. Periodic Wax Sampling:
 - one Sample/drum (two 20 cc bottles) after transferring wax from
28.30 prep tank to drums
5. Slurry Sampling:
 - 1 sample/day (two 200 cc bottles) @ filter system, if filters are
operated.

RUN PLAN FOR SPRING '94 F-T IIA RUN - TABLE 1: SUMMARY

RUN	AF-A8	AF-R12.1	R12.2	R12.3	R12.4	R12.5	R12.6A	R12.6B	R12.7
TYPE	ACTVTN	F-T	F-T	F-T	F-T	F-T	TRACER	TRACER	FILTER
DURATION, DAYS	1	2	2.5	2.5	2	2	0.5	0.5	2
FEED GAS	N ₂ , CO	SG	SG	SG	SG	SG	SG	SG	
H ₂ /CO	0	0.7	0.7	0.7	0.7	0.7	0.7	0.7	
N ₂ , %	25.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
CATALYST LOAD, LBS	480								
OIL CHARGE TO REACTOR, GALS	168								
SPACE VELOCITY, SL/HR-KG FE	2000	2400	11700	11700	11700	2400	2400	11700	
REACTOR									
PRESSURE, PSIG	75	175	750	750	500	175	175	750	
TEMPERATURE, DEG F	518	518	572	572	572	518	518	572	
INLET SUPERFICIAL VEL, FT/SEC	0.33	0.19	0.24	0.24	0.36	0.19	0.19	0.24	
OUTLET SUPERFICIAL VEL, FT/SEC	0.33	0.09	0.14	0.14	0.24	0.09	0.09	0.14	
LIQUID LEVEL, INCHES ON TAPE	196	196/166	211	166	166	166	166	211	
CATALYST WT FRACTION, %	26	26/29	29	33	33	29	29	29	
VAPOR VOID FRACTION, %	22-27	16-26	26-40	25-38	29-38	16-26	16-26	26-40	
FLOWS									
FFED GAS, SCFH	9,822	11,794	58,005	58,005	58,005	11,794	11,794	58,005	
PRODUCTS									
PRODUCT GAS, SCFH		5,234	31851	31851	40,710	5,234	5,234	31851	
HC LIQUID, GPD		174	778	778	487	174	174	778	
WATER, GPD		63	255	255	168	63	63	255	
LIGHT WAX, GPD		66	233	233	120	66	66	233	
CO CONVERSION, %		80	64	64	43	80			
CO ₂ SELECTIVITY, MOLE %		45.4	45.4	45.4	45.4	45.4			
CH ₄ SELECT. (CO ₂ FREE), WT %		6.3	6.3	6.3	6.3	6.3			
SUM C ₂ SELECT. (CO ₂ FREE), WT %		9.4	9.4	9.4	9.4	9.4			
SUM C ₃ SELECT. (CO ₂ FREE), WT %		10.5	10.5	10.5	10.5	10.5			
SUM C ₄ SELECT. (CO ₂ FREE), WT %		10.5	10.5	10.5	10.5	10.5			

RUN PLAN FOR SPRING '94 F-T IIA RUN - TABLE 2: CONTROL TARGETS

RUN	AF-A8	AF-R12.1	R12.2	R12.3	R12.4	R12.5	R12.6A	R12.6B	R12.7
TYPE	ACTVTN	F-T	F-T	F-T	F-T	F-T	TRACER	TRACER	FILTER
FEED GAS FLOWS									
CO, SCFH	7,367	6,722	33,063	33,063	33,063	6,722	6,722	33,063	
H ₂ , SCFH	0	4,718	23,202	23,202	23,202	4,718	4,718	23,202	
N ₂ , SCFH	2,455	354	1,740	1,740	1,740	354	354	1,740	
TOTAL, SCFH	9,822	11,794	58,005	58,005	58,005	11,794	11,794	58,005	
FEED GAS COMPOSITION (MOLE%)									
CO	75	57.0	57.0	57.0	57.0	57.0	57.0	57.0	
H ₂	0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	
N ₂	25.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
PRODUCT GAS - 22.18 OVERHEAD COMPOSITION (MOLE%)									
H ₂		11.1	21.4	21.4	30.0	11.1			
N ₂		6.7	5.5	5.5	4.3	6.7			
CO		25.9	37.1	37.1	46.2	25.9			
CH ₄		3.2	2.1	2.1	1.1	3.2			
CO ₂		45.9	29.8	29.8	15.8	45.9			
SUM C ₂		2.5	1.6	1.6	0.9	2.5			
SUM C ₃		1.9	1.2	1.2	0.7	1.9			
SUM C ₄		1.3	0.7	0.7	0.5	1.3			
SUM C ₅		0.8	0.4	0.4	0.3	0.8			
SUM C ₆		0.4	0.2	0.2	0.2	0.4			
SUM C ₇ + C ₈		0.08	0.04	0.04	0.04	0.08			
SUM C ₉ + C ₁₀		0.006	0.003	0.003	0.003	0.006			
22.14 SEPARATOR TEMP., DEG F		300/280	320	320	320	280	280	320	
22.18 SEPARATOR TEMP, DEG F		90-115	90-115	90-115	90-115	90-115	90-115	90-115	
10.60 PUMP FLOW RATE, GPM									12
22.60 FILTER INLET PRESS., PSIG									65
21.85 HT EXCH OUTLET TEMP., DEG F									300

APPENDIX D

Fischer-Tropsch II Run Chronology

5/7/94 10:00

Total Amount of Weight in 28:30 = 1137 lbs

5/10/94 9:00

Drum #	Tare	Empty	Net	Subtotal
1	373 lbs	130 lbs	243 lbs	243 lbs
2	373 lbs	126 lbs	247 lbs	490 lbs
4	372 lbs	125 lbs	247 lbs	737 lbs
3	382 lbs	134 lbs	248 lbs	985 lbs

10:30

Wt of Full Ethylflo-164 Drum = 181 lbs
Wt. of empty Ethylflo-164 Drum = 45 lbs
Wt. of Ethylflo-164 rinse added = 136 lbs

13:00

Transferring slurry to 27.10 Reactor from 28.30 slurry prep tank.
Wt. of empty drum (ethylflo) = 46 lbs
Therefore, wt. of ethylflo added = 181 lbs - 46 lbs = 135 lbs

14:00 This is the start of the activation period (TOS = 0 for activation)!
Transferred ethylflo chaser to reactor.
Reactor level @ 112 - 117" level.

14:15 Started N2 Flow @ 5200 SCFH; Reactor pressure = 150 psig

14:30 Starting to bring CO in. Reactor temperature = 296 F.
Reactor level = 141 inches.
Saw a temperature rise, stopped at total flow of approximately
10,000 SCFH. Let the temperature peak go and flatten.

15:00 Increasing CO flow.

17:30 Reactor level at 157 inches.

17:50 Reactor at 3664 volts and counts = 199.

18:25 Reactor at 3582 volts and counts = 265.

19:00 Reactor at 3500 volts and counts = 179.

19:45 Made transfer from 22.14 to 22.12 (up to 9 bolts) to reactor (down
to 2 bolts). Reactor cooled, OK.

19:55 Reactor at 3623 volts and counts = 130.

20:55 Reactor at 3646 volts and counts = 160.

21:30 Reactor at 3050 volts and counts = 104. (129 inches).

21:40 We're going to add ethylflo to 22.14. We've opened V-2139 to
begin cooling 22.14 temperature (= 305 F). We're getting liquid in
22.18 and losing level in reactor.

5/11/94 01:00 Just completed adding about 10 nuts (34.3 gallons) of ethylflo
into 22.14. We're bringing level up in reactor and shooting for
156 inches.

03:00 Resting comfortably: Reactor at 450 F and beginning to see
increase in CO2 production. Holding level at 154 inches. The
slurry is thicker than at start. Going to increase level to 157
inches.

05:00 Added 3 bolts of ethylflo (9.6 gallons). 22.14 cooled to 250 F
(running set point of 280 F).

05:30 Talked with BLB and decided to raise level in reactor to 166
inches on the tape.

08:30 Reactor at approximately 518 F.

14:00 Filled 22.14 from 0 nuts to 4 nuts with ethylflo-164 (9.6 gallons).

17:40 Filled 22.16 level gauge with water.

20:00 Done with activation. Lowering temperature to 464 F.

21:00 H2 flow started (TOS = 0).

21:20 Main N2 off. H2 = 5700 SCFH, CO = 15000 SCFH.

21:30 H2 = 7800 SCFH, CO = 15000 SCFH. Temperature is rising.

21:42 Reactor temperature = 482 F, turning on fin fan, TI-190-4.

22:00 H2 = 10,200 SCFH.

23:00 Temp of reactor = 490 F, P = 200 psig, F = 28,700 SCFH.

5/12/94 00:00 Losing level in reactor too fast. Lowering setpoint to 290 F from
340 F. We are at rates and at temperature, 508 F, 200 psig.
Added 8 1/2 bolts of ethylflo into the 22.14 and charged 27.12
to 1.75 bolts from "empty" (meaning some unknown level).
Reactor at 135 inches. Will increase to 156 inches.

01:00 We are at 156 inches - on level control.

01:15 Lined up 22.18, 22.11, 22.15 (22.15 blocked in. 22.18 on LIC control).
Restarted the trend collector.

Digression: The DEC was up and down with modifications being made to files to get SPXMW up. Between 20:00 and, say, 22:00, the DEC was bad.

01:30 Ed has noticed reactor performance improved with level increase.
02:30 Added 7.5 bolts (from 3.5 to 11.0 bolts) to 22.14. Will be adding to bring reactor up to 172 inches on the tape.
02:00 Made set point changes to feed flow.
CO = 13.5 (7 % high)
H2 = 8.5 (20% high) These numbers are what it reads!
N2 = 0.5 (50% high)
Should give right flow and composition.
03:00 Lined out on level. Overshot composition correction (39 % N2) bumping up a bit.
03:08 GC "Bharat" gives 670 % C7 in effluent.
04:00 Lined out at approximately 40% N2 in feed.
07:00 Blocked in 22.18.

Digression: Summary of chat with Mike Herron

- 1) TREND_COLLECTOR was not on prior to 01:15 AM this morning. Therefore, cannot start data period prior to this time.
 - 2) Overshot composition on D05001 (H2) at 03:00 AM this morning. Therefore, wait until at least 04:00 AM to start data period.
 - 3) Bharat was showing 670% Heptane in effluent during night. This problem was not fixed until 10:00 AM on May 12th. Therefore, must exclude all Bharat information when do download on this information.
- 09:00 Brought 02.61 on line to increase reactor inlet.
11:00 Increased reaction temperature from 509 F to 518 F.
13:00 Did a nuke scan. Nuke readings not steady in liquid. (+/- 1).
Increasing linear velocity by reducing pressure to 150 psig.
Initial Nuke reading at 1096 volts equal too 13-14 counts.
Jumping to 16-17 counts after every move on pressure.
13:45 Putting N2 in the feed gas to 5000 SCFH. Initial Nuke reading at 1093 volts equal to 16 counts, and jumping around.
14:45 Diluting slurry from 157 inches to 190 inches.
16:00 Slurry sample taken from 22.18.
17:20 Reactor at 190 inches on level control.
18:20 Backing out N2: Reactor level dropped from 190 inches to 184 inches.
19:20 Added 12 nuts ethylflo to 22.14.
20:00 Completed Nuke scan at 190 inches. Begin Pressure ramp to 400 psig.
22:00 Around 300 psi on reactor, will hold. Temperature profile is hot in bottom and at top of reactor.
23:20 Begin to increase reactor levels to 211 inches on tape.

5/13/94 01:00 Going to control at 525 F. Reactor level at set point = 211 inches on the tape.

02:00 Samples from 22.15 sight glasses left and right, 22:15 sight glasses now contain aqueous. Sample from 22.18 and 22.14.

04:00 Put sample (22.14) in hot box to settle.

05:20 Reactor temperatures stabilized but falling for the last hour. Increased heater set point by 0.5 F.

05:45 Increased set point 1F.

05:55 Increased set point 1F.

08:25 Matt grabbed sample from 22.14 off 10.52.01 discharge.

08:50 Moved to H₂ rich syngas (2:1 ratio H₂/CO) to hopefully get rid of possible carbon that may have settled on catalyst.

09:00 H₂ to 12.2 SCFH, CO to 10 SCFH.

09:20 H₂ to 14.0 SCFH, CO to 7.7 SCFH.

09:40 27.11 in service.

09:45 Transferred 22.18 (11.5 nuts to 1 nut) level to 22.15.

10:00 Added 10 nuts of ethylflo to 22.14.

11:30 Installed jumper between 10.52.01 to 27.11 Hi-Point vent and 27.10 outlet Hi-Point vent to flush reactor outlet line.

13:30 Lined-up steam to 02.61.

13:50 Decided we will proceed with slump test.

14:01 Gas shut off. Started performing slump test. No liquid flow to reactor. Took nuke profile. Looks very uniform.

14:15 Starting gas again. Reactor slump test complete.

14:30 Reactor in level control at 211 inches. Reset TAH-625-2/3/4/5/6 to 590 F.

14:45 Increased 27.10 temp to 550 F.

15:00 Increased 27.10 temp to 572 F.

15:30 High Temperature Shut Down (SD-2). Some reactor temperatures were approximately equal to 626 F.

16:26 SD-2 on TI-723. Product side inlet to 21.38.

16:30 Shutting down the plant. Left syngas out, put N₂ in. Cooling the reactor down.

5/14/94 01:00 27.13 heat-up proceeds slowly because of limited flow & temperature to 21.85.

04:45 Tried a slump test. Closed HIC-150 but flow kept coming to reactor. Had to close the other HIC-150 to isolate the reactor.

07:30 Completed 22.60 ethylflo test.

27.13 PIC-202 = 30.15 psig. Filter outside PI-739 = 33.13 psig. Filter inside pressure PI-732 = 37 psig (35.45 psig). Pump speed (10.60) = 200 RPM, Filtrate flow = 250 cc/53 sec.

07:32 Reactor at 142 inches on tape, beginning transfer, N₂ flow on. Target reactor end level = 51 inches on tape.

07:35 Attempted wax transfer from 27.10 to 27.13.

08:00 Stopped wax transfer - samples too thick.

15:00 Drained slurry from 27.10 reactor directly to tote bin.

Empty tote bin weight = 955 lbs
Full tote bin weight = 3242 lbs
Total slurry weight = 2287 lbs.

15:30 Drained 22.14: Drum #1 = 361 lbs
 Drum #2 = 17 lbs
 21:00 Drained 22.16

Drum #	Tare	Gross	Net	" from Top	Vol.	Density
1	45	455	410	1.75	55.6	55.2
2	45	443	398	1.75	55.6	53.5
3	45	339	294	8.0	45.1	48.7
Total	x	x	1102		156.3	52.7

Drum Vol = 58 gallons (from calculation)
 22.15 Sight Glass - mostly HC
 Estimate 768 lbs of hydrocarbons, 334 lbs of water using
 density of hydrocarbon = 48.7 lbs/ft³ and
 density of water = 62.4 lbs/ft³

5/15/94 Dave says that they drained 20 gallons of 22.18 and 20 gallons of
 22.14 into drums (prior to 5/14 22.16 drain).

Fischer-Tropsch IIA Run - May, 1994

5/25/94 Loaded oil to 28.30
 Drum # 1 466-114 = 352 lbs
 Drum # 2 460-114 = 346 lbs
 Drum # 3 363-116 = 249 lbs
 Total = 947 lbs

5/26/94 00:00 Process back on site. Reactor has oil only and is under syngas
 at 480F. Nextgen is running . . .
 07:25 Begin cooldown to 250F. Ed tells me he added 947 lbs of oil to
 prep tank yesterday.
 10:30 Began catalyst loading to 28.30.
 Drum #5 & Catalyst = 444 lbs
 Empty Drum #5 = 125 lbs
 Total Catalyst = 319 lbs
 Drum #6 & Catalyst = 288 lbs
 Empty Drum #6 = 125 lbs
 Total Catalyst = 163 lbs
 Final Catalyst Loaded = 482 lbs
 13:30 Transferred catalyst/oil slurry from 28.30 to 27.10.
 13:45 Added 342-114 = 228 lbs of Drakeol-10 to 28.30 for flushing
 transfer lines.
 14:05 Transferred flush oil from 28.30 to 27.10.
 14:10 Added oil to vessels: 13 nuts to 27.12, 16.5 nuts to 22.14.
 14:20 27.10 at 75 psig, beginning heat up.
 15:00 Reactor average temperature = 277 F, Reactor pressure=75 psig.
 15:05 Slurry height = 136 inches on the tape.

15:10 10,000 SCFH N2 flow.
 15:20 17.5 nuts on 27.12. Vessel readings from Matt.
 15.0 nuts on 22.14.
 Began raising reactor level. Target level = 166 inches.
 15:45 Reactor at 166 inches on tape and in level control. 27.12 at 13.5
 nuts, 22.14 at 2.5 nuts.
 19:30 Reactor at 142 inches, 75 psig, 412 F on N2.
 20:20 Reactor at 433 F, bringing in CO. Will ramp to 518 at 2F/4 min.

Digression #1: Ed reports approximately 5 lbs of catalyst left in prep tank. We
 will use 477 lbs of catalyst loaded for nuke spreadsheet
 calculations.

Digression #2: As it turns out, there was substantially more than 5 lbs of
 catalyst left in the prep tank. By DMH calculations then, the
 correct number of lbs of catalyst loaded = 445 lbs.

20:35 CO at 6800 SCFH (probably 7400 actual). Making fine
 adjustments to N2 flow, will wait for GC, Reactor at 442F.

Note: For activation, TIME ON STREAM = 0 at 20:30.

21:10 T= 460F.
 23:25 Lined out at 518 F.

5/27/94 00:40 Completed nuke scan at 190 inches and will move to next level of
 197 inches (01:00).
 03:10 Nuke at 197 inches on tape.
 03:50-04:00 Opened V-2120 and bypassed reactor.
 10:15 Did nuke scan. P = 75.2 psig, T = 518 F. Level = approximately
 191 inches (actually in interference range).
 10:30 Gasoline fire on pressure washer near 27.10 unloading area.
 11:15 Took levels before bringing in H2.
 11:30 Started introducing H2 into plant. Target CO = 6250 SCFH.
 H2 = 3800 SCFH.

Note: Flow factor corrections to use for feed flows:

CO = 0.93
 H2 = 0.83
 N2 = 0.67

TIME ON STREAM = 0 at 11:30 5/27/94.

11:45 SD-1 Shutdown - 01.20 high discharge temp. (01.20 recycle
 throttled).
 11:50 Plant restarted and back at run conditions.
 13:25 Increase H2 flow: 900 SCFH
 H2 flow goes from 3800 to 4700 SCFH.
 Reactor level still at 180 inches (4515 volts).

Note: Flow correction factor for H2 probably off - more like 1.00
 than 0.83.

14:35 Added Drakeol-10 to 22.14 up to 16.5 nuts. (was at 3.5 nuts so added 13 nuts of oil).
 15:00 Reduced FIC-111 to 225 SCFH.
 16:30 22.18 at 1.5 nuts, starting to come up.
 18:10 H2 in feed has gone up. Reduced H2 from 4.7 to 4.5 MSCFH (FI-101).
 19:20 Reduced H2 from 4.5 to 4.3 MSCFH.
 21:30 Took 22.14 sample.

Digression: NUKE SCANS

		Point 9 (3599 volts)	Point 5 (2494 volts)
	23:35	16.6	17.4
5/28/94	01:25	15.3	15.9

Nuke scan at 23:55 on 5/27/94 showed significantly lower holdup than previous one.

Nuke scan at 01:25 confirms reduction in holdup.

Call BLB while discussing the concentration of CO2 in effluent begins to fall off rapidly, H2 increasing as well.

02:00 During discussion, the compositions change +/- 4 points in an hour or so.

Flow Ramp:	H2	CO	
02:30	4.1	6.25	Begin
02:35	4.5	6.9	
	5.0	7.6	Hold to get temp.
02:55	6.0	9.1	Going to let temperature come up, putting utility oil on manual.
03:00	7.2	10.9	
03:10	8.6	13.1	Pressure in reactor = 179 psig, Temp in reactor = 529 F
03:15	10.3	15.7	
03:45	12.4	18.8	37.4 MSCFH on FI-187A, Pressure in reactor = 225 psig, Temperature in reactor = 522 F.

04:00 Bringing in HP H2. Increased HP H2 (holding CO constant) until total flow returned to previous level. New reference H2 flow (on FI-1200) is 17.9 SCFH.

	H2	CO	P	T	Flow (FI-187A)
4:20	21.5	22.6	275	553	44.3
4:25			300		
4:35	25.8	27.1	380	554	51.8
4:40	31.5	33.0			

4:50 Temperature of reactor = 553F. P = 450 psig, F = 62,500 SCFH
 07:05 27.10 pressure at 750 psig.

07:45 Started transfer of 22.18 to 22.15. Then LIC-693 into Auto control (09:00).
 10:00 27.10 level into auto at 182 inches.
 13:00 Did nuke scan. Numbers are stable and uniform along length of reactor.
 13:30 Rained like cats and dogs!
 14:50 Filled 22.16 sightglass with water.
 15:15 Starting pump back to 22.14.
 15:45 Added oil to 22.14 (6.5 nuts).
 15:58 FIC-1200 tripped on pressure differential. Attempted to reset HS-1200 (repeatedly) - would not hold.
 16:20 Ed Heydorn called and authorized PIC-129-2 to be blocked-in to prevent FIC-1200 from tripping.
 16:45 PIC-201 back at 750 psig.
 17:00 27.10 back at T, P, and feed composition.
 22:20 Took 22.11 and 22.14 samples.

5/29/84 00:30 Moving to new condition. Reactor at 182 inches, Temp = 574F, P = 750 psig, % bypass = 73.5, TIC 511. So

	Temp	Bypass	TIC	
00:30	574	73.5	511F	Note: TI-190-3B (Nozzle 5)
01:00	578	74.0	512F	is lagging
	581	74.5	513F	considerably behind
01:20	584	75.0	514F	by 4 - 8 F and
01:45	587	75.5	515F	dragging down the
02:05	589	76.0	516F	average temp.
02:35	595	76.5	517F	
		77.0		

03:10 Pretty much lined out - will hold for a while. Thermocouple at bottom and top of slurry a bit hotter (+ 2 F) than internals.
 03:20 We are moving fin-fan. Bypass = 77%, TIC = 517F.
 03:40 Lined-out. Drop 22.14 to 330 F.
 04:30 Fill 22.14 from 3.5 nuts to 10 nuts.
 05:47 GC Dennis gets hung up on a port - now out of sequence.
 06:30 Changed TAH 626-2/3/4/5/6 from 590 to 605 F.
 07:50 CO line pressure dropping. Someone jumped on CO line without calling the HYCO plant. FIC-104 in manual at 100%.
 08:25 Reducing reactor temperature to 575-580 F in preparation for adding extra H2 to the feed.
 08:40 GC Dennis back on-line.
 09:45 Just noticed GC's haven't updated since 09:00. Dean is restarting his computer in the lab.
 10:15 Beginning to increase H2 flow from 23.0 MSCFH. Starting increasing H2. Change in temperature in oil (out - in) increased from 32 to 34 F.
 10:30 H2 at 31 MSCFH.
 10:45 H2 at 1:1 ration with CO. Increasing H2 again. Also, just received good GC file. GC's appear to be sending to DEC again.

11:00	H2 at 41 MSCFH.
11:07	HS-1200 tripped - - - PI-1200/PIC-247-1 differential. Throttled V-1631 to drop 01.20 suction pressure.
11:20	Plant back at run conditions.
12:45	Reducing H2 back to 23.0 MSCFH.
13:05	Adding oil slowly to 22.14. Start at 15.85% on LIC-688, 3 nuts in 22.14 sight glass, 46.15% on LIC-639 (45% set point). Ended at top of second glass on 22.14.
15:20	Preparing for shutdown test. 22.14 = 15.5 nuts, 22.18 = 7.5 nuts, 27.12 = 8.5 nuts.
15:30	Slump test.
17:25	27.10 at 211 inches.
21:00	Block-in 22.15.
23:00	Loading approximately 2 x 350 lbs of Drakeol plus ethylflo into prep tank (2 x 55 gallons).
23:30	Samples taken on 22.14 and 22.11.
5/30/94 00:55	Starting to reduce rates. Dropping flow and pressure by 25 %.
01:25	Reducing rates to 50% of original values. Swapping out HP H2 for LP.
02:10	Adding oil to 22.14 from 0 nuts to 16.5 nuts.
04:30	Lined out (pumped back too much oil during flow drop, had to thicken). 29,000 SCFH, 375 psig, 518 F, DIC-636 at 211 inches. At these conditions, the weird thermocouple (190-3B) is right in line with the others.
07:20	End of period, begin cooldown.
07:35	PIC-201 at 250 psig.
09:00	V-2590 closed - - 10.52 to 27.10.
09:30	Drained reactor slurry from 205 inches on tape to 126 inches. $\% \text{ removed} = 79 / (205 + 32) \times 100 = 33.33\%$.
10:00	HP H2 in service to 01.20. Bringing pressure, temperature, and flow up.
10:30	LP H2 blocked in. PIC-129-2 valved out and depressured to prevent HS-1200 from tripping. PIC-201 at 750 psig, CO at 31 MSCFH, H2 at 23 MSCFH.
11:15	Filled 22.14 from 2 nuts to 24 nuts.
15:30	We have been at condition with full reactor since noon. Reactor temperature is however drifting.
16:00	Filled 15.90 flare K.O. pot.
16:45	Grabbed 2 samples off of 22.11.
17:15	Had Matt take samples off of 22.11. Density analysis follows: Density (H2O phase) = $34.3507 \text{ g} / 50.00 \text{ ml} = 0.687 \text{ g / ml}$ Density (HC phase) = $55.2530 \text{ g} / 50.00 \text{ ml} = 1.1051 \text{ g / ml}$
19:00	HYCO 1 tripped, steam generator below out. Maintaining rates with HP H2 and CO pressure falling.
20:18	1200 trip. Low pressure on H2 pipeline. Swapped over to LP H2.
21:50	Back at conditions. T = 570F.
23:50	Samples taken from 22.11 and 22.14.

5/31/94 04:30 Have been cruising along but H2 flow drops out at 04:30.
08:30 Data period ends for AF-R12.3. T=578F, P = 750 psig, and
Flow = 58000 SCFH.

09:25 PIC-201 at 500 psig.
10:45 Drained slurry/catalyst out of prep tank into 5 drums.
Results of the draining of the 28.30.
Weight of 4 drums (empty), Matt and hose = 666 lbs.
Weight after 1st drum was filled = 1009 lbs.
However, approximately 3 lbs of slurry spilled as Drum #1 was
overflowed (rather, bubbled up and spilled out).
Therefore, net weight of drum #1 = 1009 - 666 + 3 = 346 lbs.

Weight of drum #2 = 1352 lbs
Net weight of drum #2 = 1352 - 1012 = 340 lbs.

Weight of drum #3 = 1698 lbs.
Net weight of drum #3 = 1698 - 1352 = 346 lbs.

Weight of drum #4 = 2054 lbs.
Net weight of drum #4 = 2054 - 1698 = 356 lbs.

New weight of 4 drums (empty), Matt and hose = 656 lbs.
Weight drum #1 = 819 lbs.
Net weight of Drum #1 = 819 - 656 = 163 lbs.

Total weight of drained slurry = 346 + 340 + 346 + 356 + 163 = 1551 lbs.

15:00 Results of 22.11 Two-Phase Liquid Samples follow:
(Note: This analysis is probably more accurate than previous one
due to more accurate volume measurements.)

Top Layer: empty 25 ml flask = 19.7915 g
full 25 ml flask = 39.8015g
Density of top layer = (39.8015 - 19.7915) / 25 ml = 0.8004 g/ml

Bottom Layer: empty 5 ml flask = 41.4154 g
full 5 ml flask = 46.2686 g
Density of top layer = (46.2686 - 41.4154) / 5.0 ml = 0.9706 g/ml

17:00 Did nuke scan.
20:20 Begin pumping to 22.14. It is probably near empty, certainly
below the glass - filled to 11 nuts by 21:00.
21:30 Took samples from 22.11 and 22.14.
23:00 End of data period AFR12.4.
23:45 Move to new condition. HP H2 blocked in. LP H2 open.
Reducing gas rates. Lowering pressure and lowering
temperature.

6/1/94 00:00 When changing conditions, level in reactor dropped from 211 inches to 191 inches. Now at pressure and temperature, level in reactor = 188 inches.

00:40 Shut down the fin fan.

01:15 At 211 inches in reactor, P = 176 psig, T = 518F.

01:40 Filled 22.14 from 4.5 nuts to 8.5 nuts.

02:30 We noticed that Dennis GC is not totalizing - we strongly suspect CO because H2 and N2 of GC Dennis and GC Gary match. Just made final composition move (on N2). We are on-line and ready.

09:00 Lowered TIC-725 set point to 300 F per Mike.

10:00 Results of density analysis of 22.11 gotten at 21:30.
Top layer: empty 50 ml = 37.3028 g
full 50 ml = 78.5273g
density of top phase = $(78.5273 - 37.3028) / 50 \text{ ml} = 0.8245 \text{ g/ml}$
Bottom layer: empty 50 ml = 37.6799 g
full 50 ml = 86.3820 g
density of bottom phase = $(86.3820 - 37.6799) / 50 \text{ ml} = 0.9740 \text{ G/ml}$

10:30 Results of density analysis of 22.11 gotten at 21:30 last night.
Top layer: empty 25 ml = 20.4728 g
full 25 ml = 40.9028g
Density of top layer = $(40.9028 - 20.4728) / 25 \text{ ml} = 0.8172 \text{ g/ml}$

13:30 Grabbed samples off of 22.11 and 22.14.

14:10 Starting to heat up reactor to 578 F.

14:10	534F	14:35	559F
14:15	539F	14:40	564F
14:20	544F	14:45	569F
14:25	549F	14:50	574F
14:30	554F	14:55	579F
		15:00	584F

15:15 Did yet another density calculation on 13:30 sample.
Top layer: empty 25 ml flask = 25.6161 g
full 25 ml flask = 45.9730 g
density of top layer = $(45.973 - 25.6161) / 25 \text{ ml} = 0.8143 \text{ g/ml}$
Bottom layer: not enough phase present to give accurate density!

17:00 Ramp from 582 F to 590 F (steady at new temperature by 17:20).

20:45 Noticed things were screwy with DEC. For instance, showing 2710_AVG_TEMP = 520 F when we know it is 580 F. Checked historian - when prompted for data from 4pm to 9pm, didn't accept 4 pm data and time. Did "sho sys" - proof that Trend collector was not running. We estimate it went out between 4 - 6 pm today, 6/1/94. Restarted the trend collector.

21:00 After further analysis of DEC, we believe we lost data at 4 AM on June 1, 1994, not 4 pm! So, DEC was down from 4 AM to 21:20 on June.1, 1994. Had to do a nextgen restart. Main terminal in control room is now locked. Had to switch to Trexlertown for remote monitoring in the back process room.

23:45 22.11 & 22.14 samples taken.

6/2/94 01:05 Slump test performed. Base line: Level = 212" (counts = 46)
T = 590 F, P = 175 psig.
01:10 H2 gas ran out. FID's not working.
01:25 Done with slump test. T = 586. Begin cooldown.
04:45 Bringing in N2.
05:20 Added oil to 22.14 (below sight glass to 4.5 nuts) and stopped
pumping back to reactor.
08:30 Drained 27.10 into drums. Slurry seems typical - not lumpy or
especially viscous.
Drum # 1 - 741 to 981 lbs = 240 lbs.
There was approximately 70 lbs of steam condensate in drum to
start with. As this boiled, drum was overfilled.
Drum # 2 - 981 to 1361 lbs = 380 lbs & 6 sample bottles.
Drum # 3 - 1361 to 1737 lbs = 376 lbs.
Drum # 4 - 1737 to 2122 lbs = 385 lbs.
Drum # 5 - 660 to 1038 lbs = 378 lbs
Drum # 6 - 1038 to 1420 lbs = 382 lbs
Drum # 7 - 1420 to 1611 lbs = 191 lbs
Drained drakeol - 1611 to 1795 lbs = 184 lbs from 22.14
08:45 22.14 unloaded.
10:00 01.10 restarted.
10:05 10.52 restarted.
10:15 PIC-201 at 200 psig.
10:30 N2 at 3-4 MSCFH.
10:40 TIC-293 at 275 F.
10:45 N2 at 9 MSCFH.

10:50	280 F	11:55	345 F
10:55	285 F	12:00	350 F
11:00	290 F	12:10	355 F
11:05	295 F	12:15	360 F
11:10	300 F	12:20	365 F
11:15	305 F	12:25	370 F
11:20	310 F	12:30	375 F
11:25	315 F	12:35	380 F
11:30	320 F	12:40	385 F
11:35	325 F	12:45	390 F
11:40	330 F	12:50	395 F
11:45	335 F	12:55	400 F
11:50	340 F		

12:00 Disconnected hoses to Arrow trailer. Purging N2 through 22.11
and 22.15 low point bleeds. Purging N2 through 22.16 low point
bleeds and transfer line to trailer bleed.

NIGHT ORDERS: Maintain plant at following conditions:

PIC-201 at 200 psig.

TIC-293 at 400 F.

N2 at 9-10 MSCFH.

27.10 at 211 inches.

Sweep LP H2, HP H2, and CO feed lines with N2.

At 01:00, 1) Reduce N2 to 4-5 MSCFH.

2) Use 10.52's to transfer 22.14 and 22.12 levels to 27.10.

3) Begin to cool 27.10 to 250 F at 60 F /hr.

Once 27.10 is at 250 F, secure 01.10/01.20, depressure the plant to 80 psig, and transfer 27.10 level to 28.30. Shut off water hose to 10.53 pumps. Set-up plant N2 purge. Secure flare. Sweep the methane line with N2. After transfer, cool utility oil system with fin-fan and 21.20 to 100 deg F.

18:00 Dean performed density analysis for 22.11 sample taken at 23:45 June 1, 1994.

Top layer: empty 10 ml flask = 41.3861 g
full 10 ml flask = 49.5091 g
density of top layer = 0.8123 g/ml

Bottom layer: empty 25 ml flask = 45.0209 g
full 25 ml flask = 20.472 g
density of bottom layer = 0.9819 g/ml

6/6/94 09:00 Drums from 1/3 slurry drained during F-T IIA on 5/31/94

Drum #	Total Height	Oil Height
1	25"	17.75"
2	28"	27"
3	27"	23.5"
4	27"	25"
5	13"	11"

Drums from final slurry drained at the end of F-T IIA on 6/2/94

Drum #	Total Height	Oil Height
5A	28.625"	11"
6	22.25"	8.375"
7	28.75"	10"
8	28.5"	15.125"
9	28.375"	11.5"
10	28.5"	11"
11	14.5"	5.5"

Drum from 22.14	12	15"	15"
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Drum Dimensions: 23" Diameter
34.5" Height

APPENDIX E
Liquid Compositions

LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE																									
DATE	TIME	CARBON NO. ----->								ALCOHOLS, WT%												TOTAL			
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22		
										22.11 HC PHASE (WT%)															
5/28/94	11:50		NO HC PHASE IN THE SAMPLE																						
5/28/94	22:20		NO HC PHASE IN THE SAMPLE																						
5/29/94	23:30	0	0	0.2	0.2	0.6	0.3	0.3	0	0	0	0	0	0	0	0	0	0	0.2	0.3	0	0	0	2.1	
5/30/94	12:00		NO HC PHASE IN THE SAMPLE																						
5/30/94	23:59	2	0	1.9	1.7	2.1	2.6	1.5	0.7	0.7	0	0	0	0	0	0	0	0	0.2	0.3	0.2	0	0	13.9	
5/31/94	12:00	1.7	1.5	0	1.3	1.7	2.3	1.3	0.7	0.1	0	0.2	0	0	0	0.2	0	0	0.4	0.4	0	0.3	0	12.1	
5/31/94	21:30	1.1	2.2	0	1.6	1.6	2.3	1.4	0.6	0.6	0.4	0.3	0	0	0	0	0	0	0.3	0.5	0	0	0	12.9	
6/1/94	13:30	0	0	0	1.1	3.1	2.1	1.3	0.9	0.6	0.3	0.2	0	0	0	0.3	0.3	0	0.5	0.5	0	0.5	0.3	12.0	
6/1/94	23:45	0	0	0	1.4	3	2.1	1.3	1	0.6	0.4	0.2	0	0.2	0	0.3	0.2	0	0.5	0.7	0	0.4	0.3	12.6	
										22.16 HC PHASE (WT%)															
6/2/94	11:30		NO HC PHASE IN THE SAMPLE																						
6/2/94	11:35		NO HC PHASE IN THE SAMPLE																						
6/2/94	11:40		NO HC PHASE IN THE SAMPLE																						
6/2/94	11:45	3.9	1.1	0.7	0	0	0	0	0	0	0.1	0	0	0	0	0.1	0	0	0.3	0	0	0	0	6.2	
6/2/94	11:50	5	1.1	1.7	0	0	0	0	0	0.3	0.1	0	0	0	0	0	0	0	0.2	0.2	0.2	0.1	0	8.9	
6/2/94	11:52	3.4	1	0.3	0	0	0.1	0	0	0.3	0.1	0	0	0	0	0	0	0	0.3	0.2	0.1	0	0	5.8	
6/2/94	11:55	2.4	1	1.6	0.9	2	0.4	0.3	0.5	0.2	0.1	0	0	0	0	0	0	0	0.2	0.2	0.1	0	0.1	10.0	
6/2/94	12:00	3.6	1	1.6	0.8	1.8	0.3	0.3	0.5	0.2	0.1	0	0	0	0	0	0	0	0.3	0.3	0.3	0.2	0	11.3	

		LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE																								
DATE	TIME	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	TOTAL			
		CARBON NO. ----->										CIS-OLEFINS, WT%														
		22.11 HC PHASE (WT%)																								
5/28/94	11:50																									
5/28/94	22:20																									
5/29/94	23:30	0.6	0.8	0.7	0.5	0.3	0.2	0.1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	3.2	
5/30/94	12:00																									
5/30/94	23:59	0.5	0.8	1	1	0.7	0.5	0.4	0.2	0.1	0.1	0	0	0	0	0	0	0	0.2	0.2	0.1	0	0	0	5.8	
5/31/94	12:00	0.4	0.6	0.8	0	0.6	0.4	0.3	0.2	0.1	0	0	0	0	0	0.1	0	0	0	0	0	0	0	0	3.5	
5/31/94	21:30	0.2	0.4	0.5	0	0.5	0.5	0.3	0.2	0.2	0	0	0	0	0.1	0	0	0	0.4	0.3	0.2	0	0	0	3.8	
6/1/94	13:30	0.2	0.3	0.5	0	0.5	0.4	0.3	0.2	0	0	0	0	0	0	0	0	0	0.4	0	0.3	0.3	0	0	3.4	
6/1/94	23:45	0.1	0.3	0.5	0.6	0.5	0.4	0.3	0.2	0.1	0	0	0	0	0.2	0.2	0	0	0.5	0	0	0	0	0	3.9	
		22.16 HC PHASE (WT%)																								
6/2/94	11:30																									
6/2/94	11:35																									
6/2/94	11:40																									
6/2/94	11:45	0.7	0.9	0.8	0.6	0.5	0.3	0.2	0	0	0	0	0	0	0	0.1	0	0	0.3	0.2	0	0	0	0	4.6	
6/2/94	11:50	0.7	0.8	0.8	0.6	0.5	0.3	0.2	0.1	0.1	0	0	0	0	0	0	0	0.3	0.2	0	0	0	0	0	4.6	
6/2/94	11:52	0.7	0.9	0.9	0.7	0.5	0.3	0.2	0.1	0	0	0	0	0	0	0.1	0.1	0.3	0.2	0	0.1	0	0	0	5.1	
6/2/94	11:55	0.6	0.8	0.8	0.6	0.5	0.3	0.2	0.1	0	0	0	0	0	0	0	0	0.3	0.2	0.2	0	0	0	0	4.6	
6/2/94	12:00	0.6	0.8	0.7	0.6	0.4	0.3	0.2	0.1	0	0	0	0	0	0.1	0.1	0.2	0.4	0.2	0.2	0	0	0	0	4.9	

LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE																											
DATE	TIME	CARBON NO. ----->										N-PARAFFINS, WT%															TOTAL
		5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27			
22.11 HC PHASE (WT%)																											
5/28/94	11:50																										
5/28/94	22:20																										
5/29/94	23:30	2.7	4.2	6.4	5.7	3.6	2.8	1.9	1.5	1.3	1.1	1	0.9	0.9	0.8	0.7	0.1	0.4	0.2	0.4	0.2	0.1	0	0			36.9
5/30/94	12:00																										
5/30/94	23:59	0.6	1.7	3.1	3.7	2.4	2.1	1.6	1.3	1	0.9	0.8	0.8	0.7	0.5	0.5	0.2	0.2	0	0	0.2	0.1	0	0			22.4
5/31/94	12:00	0.5	0.8	1.6	0.6	1.4	1.3	1	0.9	0.7	0.7	0.8	0.8	0.8	0.7	1	0.4	0.5	0	0	0	0.1	0.1	0			14.7
5/31/94	21:30	0.3	0.6	1.5	0.6	1.7	1.6	1.2	1.1	1	1	1	1.1	1.1	0.9	1.1	0.4	0.4	0.1	0	0	0.1	0	0			16.8
6/1/94	13:30	0.3	0.3	0.8	0.4	1	0.9	0.7	0.6	0.5	0.6	0.8	0.9	0.9	0.9	1.3	0.6	0.7	0	0	0	0.1	0.2	0.1			12.6
6/1/94	23:45	0.3	0.3	0.9	1	1.1	1	0.8	0.7	0.6	0.6	0.7	0.8	0.9	0.9	1.4	0.8	0.7	0.3	0	0	0	0.2	0			14.0
22.16 HC PHASE (WT%)																											
6/2/94	11:30																										
6/2/94	11:35																										
6/2/94	11:40																										
6/2/94	11:45	2.5	3.5	5.5	4.9	3.1	2.3	1.7	1.2	1	0.9	0.7	0.7	0.6	0.6	0.7	0.3	0.3	0.1	0	0	0.1	0	0			30.7
6/2/94	11:50	2.4	3.6	5.7	5.1	3.2	2.5	1.9	1.4	1.1	1	0.8	0.8	0.8	0.7	0.2	0.2	0.2	0	0	0.1	0.1	0	0			31.8
6/2/94	11:52	2.2	3.4	5.4	4.9	3	2.3	1.7	1.2	1	0.8	0.8	0.7	0.7	0.6	0.4	0.4	0.3	0	0	0	0.1	0	0			29.9
6/2/94	11:55	2.1	3.5	5.8	5	3.4	2.7	1.9	1.4	1.1	1	0.8	0.8	0.6	0.7	0.4	0.4	0.3	0	0	0	0.1	0	0			32.0
6/2/94	12:00	2.5	3.4	5.4	4.9	2.6	2.3	1.7	1.2	1	0.9	0.8	0.7	0.7	0.7	0.9	0.3	0.3	0	0	0.1	0.3	0	0			30.7

LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE																										
DATE	TIME	CARBON NO. ----->									BRANCHED ALIPHATIC, WT%													TOTAL		
		5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27		
22.11 HC PHASE (WT%)																										
5/28/94	11:50																									
5/28/94	22:20																									
5/29/94	23:30	0	0.9	1.3	1.3	1.6	1.3	1.1	0.9	0.7	0.4	0.4	0.5	0.8	0.7	0.9	0.8	0.6	1.1	1.3	0.9	0	0	0	17.5	
5/30/94	12:00																									
5/30/94	23:59	0	2.1	3	3.1	1.6	1	0.9	0.6	0.7	0.4	0.2	0	0.2	0	0	0	0.2	0.7	0.9	0.9	0.1	0	0	16.6	
5/31/94	12:00	0	1.5	2.1	2.5	1.1	0.7	1.1	0.2	0.5	0.1	0.1	0	0.7	0.5	0.9	1.1	0.8	0.8	1.5	1.1	1.2	0.5	0.1	19.1	
5/31/94	21:30	0	0.2	2.9	3.7	2.3	1	1.3	0.9	0.5	0.2	0	0	0	0	0.2	0	0.4	1.2	1.8	1.2	0	0	0	17.8	
6/1/94	13:30	0.0	0	0.9	0	0.6	0.2	0	0.2	0.1	0	0.3	0.1	0.8	0.5	0.9	1.5	1.2	1.1	2.2	1.5	1	0.7	0.5	14.3	
6/1/94	23:45	0	0	0.7	0.2	0.7	0.3	0.1	0.2	0	0	0.2	0	0.8	0.5	1.1	1.5	1.2	1.7	2.5	2	1.3	0.3	0	15.3	
6/2/94	11:30																									
6/2/94	11:35																									
6/2/94	11:40																									
6/2/94	11:45	0.0	0.8	2.1	2.9	2.5	1.7	1.1	0.8	0.3	0.4	0.1	0	0.2	0.2	0.6	0.7	0.5	1.1	1.2	0.8	0.5	0.1	0	18.6	
6/2/94	11:50	0.0	0.9	2.8	2.9	2.4	1.7	1.4	0.7	0.5	0.5	0.1	0	0.3	0.1	0.1	0.3	0.4	0.5	0.8	0.8	0.6	0	0	17.8	
6/2/94	11:52	0.0	0.9	1.8	2.8	2.3	1.8	1.1	0.6	0.5	0.6	0.1	0	0.3	0.3	0.6	0.7	0.6	0.6	0.9	0.9	0.6	0	0	18.0	
6/2/94	11:55	0.0	0.9	1.2	1.5	2.2	1.6	0.9	0.7	0.4	0.4	0.1	0	0.2	0.2	0.5	0.7	0.5	0.5	0.9	0.9	0.3	0.2	0	14.8	
6/2/94	12:00	0.0	0.8	1.2	1.4	1.9	1.4	0.7	0.7	0.3	0.4	0.1	0	0.2	0.3	0.5	0.8	0.6	0.6	0.8	0.8	0.1	0	0	13.6	

LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE																										
DATE	TIME	CARBON NO. ----->										OTHER OLEFINS, WT%												TOTAL		
		5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26			
		22.11 HC PHASE (WT%)																								
5/28/94	11:50																									
5/28/94	22:20																									
5/29/94	23:30	0	0.9	0.2	1.3	0.8	0.5	0.4	0.1	0	0	0	0	0.6	0.8	0.3	1.6	0.8	1.5	1	0.6	0	0	11.4		
5/30/94	12:00																									
5/30/94	23:59	0	1.1	1.2	1	1.5	1.2	0.7	0.4	0.3	0.2	0	0	0.2	0.3	0	0	0.3	0.8	0.7	0.7	0.7	0	11.3		
5/31/94	12:00	0	0.3	0.2	3.7	0.8	0.7	0.7	0.3	0.3	0	0	0	0.4	0.5	0.2	1	0.8	1.6	1.9	1.2	0.9	0.4	15.9		
5/31/94	21:30	0	0.8	1.2	4.4	1.9	1.8	1.5	0.6	0.4	0.2	0	0	0.3	0.2	0	0	0.6	0.9	1.5	0.6	0	0	16.9		
6/1/94	13:30	0	0.2	0.1	1.5	0.5	0.6	0.6	0.3	0.4	0.2	0	0	0.5	0.8	0.5	1.6	1.1	1.3	2.8	1.6	1.7	0.8	17.1		
6/1/94	23:45	0	0.2	0	0.5	0.7	0.7	0.7	0.3	0.3	0.2	0	0	0.5	0.8	0.3	1.4	1	1.4	3	1.6	0.9	0.2	14.7		
		22.16 HC PHASE (WT%)																								
6/2/94	11:30																									
6/2/94	11:35																									
6/2/94	11:40																									
6/2/94	11:45	0	1.5	0.5	1.9	1.1	0.7	0.6	0.3	0.1	0	0	0	0.3	0.4	0	0.6	0.5	1	0.8	1	0.4	0	11.7		
6/2/94	11:50	0	1.8	0.7	1.9	1.9	0.7	0.4	0.2	0.2	0.1	0	0	0.2	0.3	0	0.2	0.1	0.6	1.5	0.7	0.4	0	11.9		
6/2/94	11:52	0	1.3	.	1.9	1.1	0.7	0.3	0.2	0.2	0	0	0	0.3	0.4	0.1	0.5	0.2	0.6	1.2	0.6	0.4	0	10.0		
6/2/94	11:55	0	1.6	0.6	1.9	1.3	0.8	0.5	0.3	0.3	0	0	0	0.2	0.4	0	0.6	0.2	0.8	1.1	0.6	0.4	0.1	11.7		
6/2/94	12:00	0	1.6	0.5	2.2	1	0.7	0.5	0.2	0.1	0	0	0	0.3	0.3	0	0.6	0.2	0.7	1.1	0.8	0	0	10.8		

LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE																											
DATE	TIME	CARBON NO. ----->										UNIDENTIFIED, WT%										TOTAL		GRAND			
		5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27		TOTAL	
		22.11 HC PHASE (WT%)																									
5/28/94	11:50																										
5/28/94	22:20																										
5/29/94	23:30	3	0.1	0.5	1.1	0.8	0.5	0.3	0.6	0	0.1	0.3	0.1	0.3	1	1.3	1.3	1.4	1	1.7	1.5	0.6	0.1	0	17.6	99.8	
5/30/94	12:00																										
5/30/94	23:59	3	0	0.3	0.9	1.2	1.2	0.7	0.1	0.1	0	0	0	0	0	0	0.1	0.4	0.7	1.4	1.5	0.9	0	0	12.5	100.0	
5/31/94	12:00	1.4	1	0	0.6	0.6	0.8	0.3	0	0	0	0	0.1	0.3	0.6	0.9	1.2	1.7	2.2	2.9	2.6	2	0.6	0.2	20.0	100.5	
5/31/94	21:30	2	2.9	0	0.6	1.4	1.4	0.7	0.2	0	0	0	0	0	0	0.2	0.2	0.6	1.7	2.2	2.2	0.8	0.3	0	17.4	100.2	
6/1/94	13:30	1	1.2	0	0.2	0.4	0.5	0	0.1	0	0	0	0.3	0.2	1.1	1.6	1.8	2.5	3.4	4.2	3.7	2.8	1.4	0.8	27.2	101.0	
6/1/94	23:45	0.9	1.1	0	0.4	0.5	0.7	0.3	0.1	0	0	0	0.3	0.3	0.9	1.7	1.8	2.5	3.5	3.1	3.2	2.5	1.2	0.5	25.5	100.3	
		22.16 HC PHASE (WT%)																									
6/2/94	11:30																										
6/2/94	11:35																										
6/2/94	11:40																										
6/2/94	11:45	1.8	1.6	1	1	1	0.8	0.4	0.2	0	0.1	0.1	0	0.1	0.2	0.4	0.7	1.1	1.3	1.8	1.4	0.8	0.2	0.2	16.2	99.8	
6/2/94	11:50	1.9	0.2	0.4	1.1	1.1	0.6	0.4	0.2	0	0	0.1	0	0.1	0.1	0.1	0.1	0.5	1.2	1.9	1.6	0.8	0.1	0	12.5	99.9	
6/2/94	11:52	3.1	1.1	1.2	1.1	1.2	0.5	0.4	0.3	0	0	0.1	0	0.1	0.2	0.4	0.8	1.2	1.6	2.1	1.6	0.9	0.3	0	18.2	99.5	
6/2/94	11:55	1.4	0.1	1.3	1.1	1.1	0.6	0.4	0.1	0	0.1	0	0	0.1	0.1	0.3	0.4	0.8	1.5	2	1.6	0.9	0.1	0	14.0	99.8	
6/2/94	12:00	2.1	0.1	1.2	1.1	1.1	0.3	0.4	0	0	0.1	0	0	0.1	0.3	0.4	0.8	1.3	1.9	2.3	1.7	0.9	0.1	0	16.2	99.8	

					LIQUID COMPOSITION FOR FT-IIA (1994) AT LAPORTE								
DATE	TIME	Methanol	Ethanol	1-Propanol	1-Butanol	1-Pentanol	1-Hexanol	1-Heptanol	1-Octanol	2-Propanol	2-Butanol	Isobutanol	
						22.11 AQUEOUS PHASE (WT%)							
5/28/94	11:50	1.335	1.785	0.684	0.227	0.077	0.015	0	0	0.284	0.134	0.074	
5/28/94	22:20	1.93	2.94	1.181	0.343	0.094	0.017	0	0	0.477	0.215	0.114	
5/29/94	23:30	2.638	3.83	1.655	0.473	0.133	0.025	0.004	0	0.468	0.211	0.109	
5/30/94	12:00	6.686	8.906	4.219	1.715	0.771	0.244	0.082	0.025	0.624	0.349	0.111	
5/30/94	23:59	6.272	8.179	4.184	1.712	0.76	0.239	0.082	0.023	0.595	0.353	0.098	
5/31/94	12:00	5.031	7.255	3.804	1.493	0.628	0.182	0.057	0.009	0.503	0.305	0.074	
5/31/94	21:30	5.111	6.801	3.52	1.476	0.666	0.193	0.058	0.008	0.463	0.277	0.07	
6/1/94	13:30	NO AQUEOUS PHASE IN THE SAMPLE											
6/1/94	23:45	3.125	5.423	2.688	1.124	0.46	0.107	0.024	0.005	0.384	0.215	0.057	
						22.16 AQUEOUS PHASE (WT%)							
6/2/94	11:30	2.501	3.194	1.394	0.402	0.12	0.024	0.004	0	0.388	0.178	0	
6/2/94	11:35	5.66	7.501	3.514	1.124	0.358	0.081	0.02	0.005	0.561	0.286	0.108	
6/2/94	11:40	5.922	7.819	3.699	1.216	0.391	0.089	0.022	0.005	0.574	0.298	0.109	
6/2/94	11:45	NO AQUEOUS PHASE IN THE SAMPLE											
6/2/94	11:50	NO AQUEOUS PHASE IN THE SAMPLE											
6/2/94	11:52	NO AQUEOUS PHASE IN THE SAMPLE											
6/2/94	11:55	NO AQUEOUS PHASE IN THE SAMPLE											
6/2/94	12:00	NO AQUEOUS PHASE IN THE SAMPLE											

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APPENDIX F

F-T II / IIA Demonstration Mass Balances

RUN NO: AF-R11.1A

TITLE: LIQUID PHASE FISCHER-TROPSCH (II) SYNTHESIS IN LAPORTE AFDU

Start Date / Time	5/12/94	4.00	On-stream Time From Start-up (hr)
End Date / Time	5/12/94	10.00	Start 7.00
			End 13.00

Reaction Conditions:

Temperature (°F)	508.2
Pressure (psig)	200.1
Space Velocity (sL/kg-Fe hr)	2440
Superficial Gas Velocity - Inlet (ft/sec)	0.35

Slurry Data:

Catalyst Weight (lb oxide)	985
Iron content in Catalyst (wt %)	60.57
Slurry Concentration (wt %)	43.5
Slurry Level (% of NDG Span)	73.3
Slurry Height (ft)	17.13
Average Gas Holdup (vol %)	18.7

Performance Results

CO Conversion, mole %	34.7
H2 Conversion, mole %	37.9
CO + H2 Conversion, mole %	36.0

CO Conversion Rate, gmole CO converted/kg-Fe hr	21.5
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HC Production Rate, grams of HC (CH2.8) produced/kg-Fe hr	163.9
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Reactor Productivity grams of HC (CH2.8) /hr - lit of reactor vol.	36.64
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H2/CO Usage Ratio, mole/mole	0.76
H2/CO in Outlet, mole/mole	0.66

CO2 Selectivity, mole %	48.6
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HC Selectivity (CO2 free) mole % C :

CH4	19.24
C2H6	14.76
C2H4	0.62
C3H8	15.14
C3H6	5.74
SUM C4H10	7.44
SUM C4H8	7.53
SUM C5H11	8.51

RUN NO: AF-R11.1A

TITLE: LIQUID PHASE FISCHER-TROPSCH (II) SYNTHESIS IN LAPORTE AFDU

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	39.82	31.36
2	N2	3.19	4.04
3	CO	57.00	47.16
4	CH4	0.00	2.48
5	CO2	0.00	12.18
6	ETHANE	0.00	0.95
7	ETHYLENE	0.00	0.04
8	PROPANE	0.00	0.65
9	PROPYLENE	0.00	0.25
10	ISOBUTANE	0.00	0.01
11	N-BUTANE	0.00	0.23
12	T-BUTENE-2	0.00	0.10
13	BUTENE-1	0.00	0.05
14	ISOBUTYLENE	0.00	0.03
15	C-BUTENE-2	0.00	0.07
16	SUM C5	0.00	0.22
17	SUM C6	0.00	0.11
18	SUM C7	0.00	0.05
19	SUM C8	0.00	0.03
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
TOTAL		100.00	100.00
Mole Wt	lb/lb mole	17.66	22.03
Flows	SCFH	25094.55	19802.52
	lb mole/hr	64.90	51.22
	lb/hr	1146.31	1128.40

RUN NO: AF-R11.1B

TITLE: LIQUID PHASE FISCHER-TROPSCH (II) SYNTHESIS IN LAPORTE AFDU

			On-stream Time From Start-up (hr)	
Start Date / Time	5/13/94	4.00	Start	31.00
End Date / Time	5/13/94	8.00	End	35.00
Reaction Conditions:			Slurry Data:	
Temperature (°F)		521.0	Catalyst Weight (lb oxide)	985
Pressure (psig)		300.0	Iron content in Catalyst (wt %)	60.57
Space Velocity (sL/kg-Fe hr)		2433	Slurry Concentration (wt %)	41.1
Superficial Gas Velocity - Inlet (ft/sec)		0.24	Slurry Level (% of NDG Span)	91.9
			Slurry Height (ft)	20.05
			Average Gas Holdup (vol %)	24.5
Performance Results				
CO Conversion, mole %		25.7		
H2 Conversion, mole %		27.5		
CO + H2 Conversion, mole %		26.4		
CO Conversion Rate,		15.9		
gmole CO converted/kg-Fe hr				
HC Production Rate,		153.6		
grams of HC (CH _{2.8}) produced/kg-Fe hr				
Reactor Productivity		29.34		
grams of HC (CH _{2.8}) /hr - lit of reactor vol.				
H2/CO Usage Ratio, mole/mole		0.74		
H2/CO In Outlet, mole/mole		0.68		
CO ₂ Selectivity, mole %		34.8		
HC Selectivity (CO₂ free) mole % C :				
CH ₄		12.26		
C ₂ H ₆		9.83		
C ₂ H ₄		0.44		
C ₃ H ₈		11.06		
C ₃ H ₆		2.31		
SUM C ₄ H ₁₀		5.80		
SUM C ₄ H ₈		3.56		
SUM C ₅ H ₁₁		2.54		

RUN NO: AF-R12.1A

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

			On-stream Time From Start-up (hr)	
Start Date / Time	5/27/94	17.00	Start	5.00
End Date / Time	5/28/94	1.00	End	13.00
Reaction Conditions:			Slurry Data:	
Temperature (°F)		517.9	Catalyst Weight (lb oxide)	445
Pressure (psig)		175.0	Iron content in Catalyst (wt %)	61.06
Space Velocity (sL/kg-Fe hr)		2534	Slurry Concentration (wt %)	25
Superficial Gas Velocity - Inlet (ft/sec)		0.19	Slurry Level (% of NDG Span)	78.1
			Slurry Height (ft)	17.88
			Average Gas Holdup (vol %)	23.5
Performance Results				
CO Conversion, mole %		84.7		
H2 Conversion, mole %		79.6		
CO + H2 Conversion, mole %		82.5		
CO Conversion Rate,		53.1		
gmole CO converted/kg-Fe hr				
HC Production Rate,		424.1		
grams of HC (CH2.5) produced/kg-Fe hr				
Reactor Productivity		41.38		
grams of HC (CH2.5) /hr - lit of reactor vol.				
H2/CO Usage Ratio, mole/mole		0.70		
H2/CO In Outlet, mole/mole		1.00		
CO2 Selectivity, mole %		44.9		
HC Selectivity (CO2 free) mole % C :				
CH4		13.42		
C2H6		11.65		
C2H4		0.49		
C3H8		13.29		
C3H6		4.65		
SUM C4H10		7.46		
SUM C4H8		5.69		
SUM C5H11		7.92		

RUN NO: AF-R12.1A

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	41.36	17.14
2	N2	3.20	6.49
3	CO	55.44	17.14
4	CH4	0.00	7.04
5	CO2	0.00	42.79
6	ETHANE	0.00	3.06
7	ETHYLENE	0.00	0.13
8	PROPANE	0.00	2.32
9	PROPYLENE	0.00	0.81
10	ISOBUTANE	0.00	0.04
11	N-BUTANE	0.00	0.94
12	T-BUTENE-2	0.00	0.19
13	BUTENE-1	0.00	0.25
14	ISOBUTYLENE	0.00	0.11
15	C-BUTENE-2	0.00	0.19
16	SUM C5	0.00	0.83
17	SUM C6	0.00	0.36
18	SUM C7	0.00	0.14
19	SUM C8	0.00	0.04
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
TOTAL		100.00	100.00
Mole Wt	lb/lb mole	17.26	31.31
Flows	SCFH	11871.13	5857.36
	lb mole/hr	30.70	15.15
	lb/hr	529.96	474.37

RUN NO: AF-R12.2A

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

			On-stream Time From Start-up (hr)	
Start Date / Time	5/28/94	13.00	Start	25.00
End Date / Time	5/29/94	0.00	End	36.00
Reaction Conditions:			Slurry Data:	
Temperature (°F)	576.0		Catalyst Weight (lb oxide)	445
Pressure (psig)	750.0		Iron content in Catalyst (wt %)	61.06
Space Velocity (sL/kg-Fe hr)	12266		Slurry Concentration (wt %)	23
Superficial Gas Velocity - Inlet (ft/sec)	0.24		Slurry Level (% of NDG Span)	78.7
			Slurry Height (ft)	17.97
			Average Gas Holdup (vol %)	9
Performance Results				
CO Conversion, mole %	54.2			
H2 Conversion, mole %	56.3			
CO + H2 Conversion, mole %	55.0			
CO Conversion Rate, gmole CO converted/kg-Fe hr	172.2			
HC Production Rate, grams of HC (CH2.5) produced/kg-Fe hr	1405.9			
Reactor Productivity grams of HC (CH2.5) /hr - lit of reactor vol.	136.43			
H2/CO Usage Ratio, mole/mole	0.71			
H2/CO in Outlet, mole/mole	0.65			
CO2 Selectivity, mole %	43.7			
HC Selectivity (CO2 free) mole % C :				
CH4	11.78			
C2H6	11.95			
C2H4	0.39			
C3H8	14.45			
C3H6	3.66			
SUM C4H10	8.16			
SUM C4H8	3.96			
SUM C5H11	8.45			

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

RUN NO: AF-R12.2A

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	39.61	26.49
2	N2	2.29	3.52
3	CO	58.10	40.78
4	CH4	0.00	3.21
5	CO2	0.00	21.16
6	ETHANE	0.00	1.63
7	ETHYLENE	0.00	0.05
8	PROPANE	0.00	1.31
9	PROPYLENE	0.00	0.33
10	ISOBUTANE	0.00	0.03
11	N-BUTANE	0.00	0.53
12	T-BUTENE-2	0.00	0.16
13	BUTENE-1	0.00	0.07
14	ISOBUTYLENE	0.00	0.04
15	C-BUTENE-2	0.00	0.00
16	SUM C5	0.00	0.46
17	SUM C6	0.00	0.15
18	SUM C7	0.00	0.05
19	SUM C8	0.00	0.02
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
	TOTAL	100.00	100.00
Mole Wt	lb/lb mole	17.71	25.00
Flows	SCFH	57464.38	37336.89
	lb mole/hr	148.62	96.57
	lb/hr	2632.83	2414.32

RUN NO: AF-R12.3F (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Start Date / Time	5/30/94	16.00	On-stream Time From Start-up (hr)	
End Date / Time	5/31/94	8.00	Start	76.00
			End	92.00

Reaction Conditions:

Temperature (°F)	577.5
Pressure (psig)	750.0
Space Velocity (sL/kg-Fe hr)	18529
Superficial Gas Velocity - Inlet (ft/sec)	0.24

Slurry Data:

Catalyst Weight (lb oxide)	297
Iron content in Catalyst (wt %)	61.06
Slurry Concentration (wt %)	14.4
Slurry Level (% of NDG Span)	95.6
Slurry Height (ft)	20.63
Average Gas Holdup (vol %)	8.9

Performance Results

CO Conversion, mole %	24.6
H2 Conversion, mole %	32.2
CO + H2 Conversion, mole %	27.6

Mass/Elemental Balance:

	Total lb/hr	C lb/hr	H lb/hr	O lb/hr	N lb/hr
Reactor Feed Gas	2674.69	1051.73	118.31	1401.14	103.52
Main Gas Outlet	2421.12	964.87	100.01	1252.71	103.53
27.10 Reactor Wax	0.00	0.00	0.00	0.00	0.00
22.14 Light Wax	0.00	0.00	0.00	0.00	0.00
22.18 HC Phase	46.90	38.53	6.92	1.44	0.00
22.18 AQ Phase	78.80	12.29	8.88	57.63	0.00
Total Out	2546.82	1015.69	115.82	1311.78	103.53
% Balance	95.2	96.6	97.9	93.6	100.0

CO Conversion Rate, gmole CO converted/kg-Fe hr	118.7
HC Production Rate, grams of HC (CH2.5) produced/kg-Fe hr	1230.8
Reactor Productivity grams of HC (CH2.5) /hr - lit of reactor vol.	69.46

H2/CO Usage Ratio, mole/mole	0.88
H2/CO In Outlet, mole/mole	0.60

CO2 Selectivity, mole %	28.5
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HC Selectivity (CO2 free) mole % C :

CH4	5.52
C2H6	5.59
C2H4	3.13
C3H8	3.42
C3H6	11.16
SUM C4H10	2.16
SUM C4H8	8.49
SUM C5H11	6.24

Product Distribution: Selectivity (wt%)

Methane (C1)	10.3
Gas (C2 - C4)	49.9
Gasoline (C5 - C11)	33.3
Diesel (C12 - C18)	3.0
Wax (C19+)	3.5
Total	100.0

Alpha Estimate:

Carbon No.	Alpha	
C1-C16	Single	0.63

RUN NO: AF-R12.3F (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	39.09	33.48
2	N2	2.47	3.12
3	CO	58.44	55.66
4	CH4	0.00	0.72
5	CO2	0.00	5.17
6	ETHANE	0.00	0.36
7	ETHYLENE	0.00	0.20
8	PROPANE	0.00	0.15
9	PROPYLENE	0.00	0.48
10	ISOBUTANE	0.00	0.00
11	N-BUTANE	0.00	0.07
12	T-BUTENE-2	0.00	0.08
13	BUTENE-1	0.00	0.11
14	ISOBUTYLENE	0.00	0.01
15	C-BUTENE-2	0.00	0.07
16	SUM C5	0.00	0.16
17	SUM C6	0.00	0.08
18	SUM C7	0.00	0.04
19	SUM C8	0.00	0.02
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
TOTAL		100.00	100.00
Mole Wt	lb/lb mole	17.85	20.41
Flows	SCFH	57933.72	45859.61
	lb mole/hr	149.84	118.61
	lb/hr	2674.69	2421.20

RUN NO: AF-R12.3F (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Carbon No.	Compositions (wt%):			HC Phase						Aqueous Phase	
	Alcohols	Cis 2-Olefin	Trans 2-Olefin	n-Paraffin	1-Olefin	Branched Aliphatic	Other Olefins	Unidentified	Total	Compound	wt%
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%		
1	2.0								2.0	Methanol	6.3
2	0.0								0.0	Ethanol	8.2
3	1.9								1.9	1-Propanol	4.2
4	1.7								1.7	1-Butanol	1.7
5	2.1	0.5	0.7	0.6	0.7	0.0	0.0	3.0	7.6	1-Pentanol	0.8
6	2.6	0.8	1.4	1.7	1.2	2.1	1.1	0.0	10.9	1-Hexanol	0.2
7	1.5	1.0	1.7	3.1	1.2	3.0	1.2	0.3	13.0	1-Heptanol	0.1
8	0.7	1.0	1.6	3.7	1.2	3.1	1.0	0.9	13.2	1-Octanol	0.0
9	0.7	0.7	1.3	2.4	0.8	1.6	1.5	1.2	10.2	2-Propanol	0.6
10	0.0	0.5	0.9	2.1	0.5	1.0	1.2	1.2	7.4	2-Butanol	0.4
11	0.0	0.4	0.6	1.6	0.7	0.9	0.7	0.7	5.6	Isobutanol	0.1
12	0.0	0.2	0.3	1.3	0.4	0.6	0.4	0.1	3.3	2-Pentanol	0.1
13	0.0	0.1	0.3	1.0	0.3	0.7	0.3	0.1	2.8	Acetic Acid	4.9
14	0.0	0.1	0.2	0.9	0.1	0.4	0.2	0.0	1.9	Propanoic Acid	1.3
15	0.0	0.0	0.1	0.8	0.1	0.2	0.0	0.0	1.2	Butanoic Acid	0.6
16	0.0	0.0	0.0	0.8	0.0	0.0	0.0	0.0	0.8	Pentanoic Acid	0.2
17	0.0	0.0	0.1	0.7	0.0	0.2	0.2	0.0	1.2	Hexanoic Acid	0.0
18	0.2	0.0	0.1	0.5	0.0	0.0	0.3	0.0	1.1	Methyl Acetate	0.1
19		0.0	0.0	0.5	0.0	0.0	0.0	0.0	0.8	Ethyl Acetate	0.1
20		0.0	0.2	0.2	0.0	0.0	0.0	0.1	0.7	Ethyl Propanoate	0.0
21		0.0	0.2	0.2	0.1	0.2	0.3	0.4	1.4	Propyl Acetate	0.1
22		0.2	0.2	0.0	0.0	0.7	0.8	0.7	2.6	Butyl Acetate	0.0
23		0.2	0.3	0.0	0.0	0.9	0.7	1.4	3.5	Acetaldehyde	0.1
24		0.1	0.0	0.2	0.0	0.9	0.7	1.5	3.4	Propanaldehyde	0.1
25				0.1	0.0	0.1	0.7	0.9	1.8	Butanaldehyde	0.0
26				0.0	0.0	0.0	0.0	0.0	0.0	2-Propanone	0.4
27				0.0	0.0		0.0	0.0	0.0	2-Pentanone	0.1
28				0.0	0.0		0.0		0.0	2-Hexanone	0.0
29				0.0	0.0		0.0		0.0	Uncalibrated	0.4
30				0.0	0.0		0.0		0.0	Total Organics	31.0
> 30				0.0	0.0		0.0		0.0	Water by diff.	69.0
Total	13.9	5.8	10.2	22.4	7.3	16.6	11.3	12.5	100.0	Total	100.0

ALPHA ESTIMATION - RUN NO. AF-R12.3F

RUN NO: AF-R12.4 (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Start Date / Time	5/31/94	10.00	On-stream Time From Start-up (hr)	
End Date / Time	5/31/94	23.00	Start	94.00
			End	107.00

Reaction Conditions:

Temperature (°F)	577.9
Pressure (psig)	500.0
Space Velocity (sL/kg-Fe hr)	18682
Superficial Gas Velocity - Inlet (ft/sec)	0.36

Slurry Data:

Catalyst Weight (lb oxide)	297
Iron content in Catalyst (wt %)	61.06
Slurry Concentration (wt %)	15
Slurry Level (% of NDG Span)	91.8
Slurry Height (ft)	20.03
Average Gas Holdup (vol %)	13

Performance Results

CO Conversion, mole %	15.6
H2 Conversion, mole %	18.6
CO + H2 Conversion, mole %	16.8

CO Conversion Rate, gmole CO converted/kg-Fe hr	75.4
HC Production Rate, grams of HC (CH2.5) produced/kg-Fe hr	874.7
Reactor Productivity, grams of HC (CH2.5) /hr - lit of reactor vol.	50.85

H2/CO Usage Ratio, mole/mole	0.82
H2/CO in Outlet, mole/mole	0.66

CO2 Selectivity, mole %	20.0
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HC Selectivity (CO2 free) mole % C :

CH4	5.55
C2H6	4.44
C2H4	4.72
C3H8	2.23
C3H6	11.11
SUM C4H10	1.38
SUM C4H8	7.68
SUM C5H11	5.09

Alpha Estimate:

Carbon No.	Alpha	
C1-C16	Single	0.68

Mass/Elemental Balance:

	Total lb/hr	C lb/hr	H lb/hr	O lb/hr	N lb/hr
Reactor Feed Gas	2677.49	1053.04	120.80	1402.88	100.77
Main Gas Outlet	2448.36	977.64	110.12	1259.84	100.75
27.13 Reactor Wax	0.00	0.00	0.00	0.00	0.00
22.14 Light Wax	0.00	0.00	0.00	0.00	0.00
22.18 HC Phase	74.80	61.80	10.96	2.03	0.00
22.18 AQ Phase	46.50	6.48	5.20	34.82	0.00
Total Out	2569.66	1045.93	126.29	1296.69	100.75
% Balance	96.0	99.3	104.5	92.4	100.0

Product Distribution: Selectivity (wt%)

Methane (C1)	7.9
Gas (C2 - C4)	37.2
Gasoline (C5 - C11)	35.5
Diesel (C12 - C18)	6.2
Wax (C19+)	13.2
Total	100.0

RUN NO: AF-R12.4 (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	39.58	37.09
2	N2	2.38	2.77
3	CO	58.04	56.43
4	CH4	0.00	0.47
5	CO2	0.00	2.11
6	ETHANE	0.00	0.19
7	ETHYLENE	0.00	0.20
8	PROPANE	0.00	0.06
9	PROPYLENE	0.00	0.31
10	ISOBUTANE	0.00	0.00
11	N-BUTANE	0.00	0.03
12	T-BUTENE-2	0.00	0.04
13	BUTENE-1	0.00	0.07
14	ISOBUTYLENE	0.00	0.01
15	C-BUTENE-2	0.00	0.04
16	SUM C5	0.00	0.09
17	SUM C6	0.00	0.05
18	SUM C7	0.00	0.03
19	SUM C8	0.00	0.01
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
TOTAL		100.00	100.00
Mole Wt	lb/lb mole	17.72	18.86
Flows	SCFH	58413.37	50190.71
	lb mole/hr	151.08	129.81
	lb/hr	2677.49	2448.47

RUN NO: AF-R12.4 (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Carbon No.	Compositions (wt%):			HC Phase						Aqueous Phase	
	Alcohols	Cis 2-Olefin	Trans 2-Olefin	n-Paraffin	1-Olefin	Branched Aliphatic	Other Olefins	Unidentified	Total	Compound	wt%
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%		
1	1.4								1.4	Methanol	5.1
2	1.9								1.9	Ethanol	7.0
3	0.0								0.0	1-Propanol	3.7
4	1.5								1.5	1-Butanol	1.5
5	1.7	0.3	0.5	0.4	0.7	0.0	0.0	1.7	5.2	1-Pentanol	0.6
6	2.3	0.5	1.0	0.7	0.8	0.9	0.6	2.0	8.6	1-Hexanol	0.2
7	1.4	0.7	1.1	1.6	0.9	2.5	0.7	0.0	8.7	1-Heptanol	0.1
8	0.7	0.0	0.2	0.6	0.9	3.1	4.1	0.6	10.1	1-Octanol	0.0
9	0.4	0.6	1.0	1.6	0.7	1.7	1.4	1.0	8.2	2-Propanol	0.5
10	0.2	0.5	0.8	1.5	0.4	0.9	1.3	1.1	6.5	2-Butanol	0.3
11	0.3	0.3	0.5	1.1	0.5	1.2	1.1	0.5	5.4	Isobutanol	0.1
12	0.0	0.2	0.3	1.0	0.4	0.6	0.5	0.1	3.0	2-Pentanol	0.1
13	0.0	0.2	0.3	0.9	0.4	0.5	0.4	0.0	2.5	Acetic Acid	5.2
14	0.0	0.0	0.2	0.9	0.2	0.2	0.1	0.0	1.5	Propanoic Acid	1.6
15	0.1	0.0	0.1	0.9	0.2	0.1	0.0	0.0	1.3	Butanoic Acid	0.7
16	0.0	0.0	0.1	1.0	0.0	0.0	0.0	0.1	1.1	Pentanoic Acid	0.2
17	0.0	0.0	0.1	1.0	0.1	0.4	0.4	0.2	2.0	Hexanoic Acid	0.0
18	0.4	0.1	0.2	0.8	0.2	0.3	0.4	0.3	2.5	Methyl Acetate	0.1
19		0.1	0.0	1.1	0.1	0.6	0.1	0.6	2.9	Ethyl Acetate	0.1
20		0.0	0.3	0.4	0.0	0.6	0.5	0.7	2.4	Ethyl Propanoate	0.0
21		0.0	0.3	0.5	0.3	0.6	0.7	1.2	3.6	Propyl Acetate	0.0
22		0.2	0.4	0.1	0.2	1.0	1.3	2.0	5.0	Butyl Acetate	0.0
23		0.2	0.3	0.0	0.4	1.7	1.7	2.6	6.8	Acetaldehyde	0.1
24		0.1	0.3	0.0	0.2	1.2	0.9	2.4	5.0	Propanaldehyde	0.0
25				0.1	0.0	0.6	0.5	1.4	2.8	Butanaldehyde	0.0
26				0.1	0.0	0.3	0.2	0.5	1.0	2-Propanone	0.3
27				0.0	0.0		0.0	0.1	0.2	2-Pentanone	0.1
28				0.0	0.0		0.0		0.0	2-Hexanone	0.0
29				0.0	0.0		0.0		0.0	Uncalibrated	0.3
30				0.0	0.0		0.0		0.0	Total Organics	27.9
> 30				0.0	0.0		0.0		0.0	Water by diff.	72.1
Total	12.5	3.7	7.8	15.8	7.2	18.5	16.4	18.7	100.4	Total	100.0

ALPHA ESTIMATION - RUN NO. AF-R12.4

RUN NO: AF-R12.5A

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Start Date / Time	6/1/94	2.00	On-stream Time From Start-up (hr)	
End Date / Time	6/1/94	14.00	Start	110.00
			End	122.00

Reaction Conditions:

Temperature (°F)	517.8
Pressure (psig)	175.0
Space Velocity (sL/kg-Fe hr)	3665
Superficial Gas Velocity - Inlet (ft/sec)	0.18

Slurry Data:

Catalyst Weight (lb oxide)	297
Iron content in Catalyst (wt %)	61.06
Slurry Concentration (wt %)	13.8
Slurry Level (% of NDG Span)	93.0
Slurry Height (ft)	20.22
Average Gas Holdup (vol %)	6.7

Performance Results

CO Conversion, mole %	10.0
H2 Conversion, mole %	11.7
CO + H2 Conversion, mole %	10.7

CO Conversion Rate, gmole CO converted/kg-Fe hr	9.5
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HC Production Rate, grams of HC (CH _{2.5}) produced/kg-Fe hr	106.3
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Reactor Productivity grams of HC (CH _{2.5}) /hr - lit of reactor vol.	6.12
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H2/CO Usage Ratio, mole/mole	0.79
H2/CO in Outlet, mole/mole	0.66

CO ₂ Selectivity, mole %	22.4
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HC Selectivity (CO₂ free) mole % C :

CH ₄	3.87
C ₂ H ₆	3.00
C ₂ H ₄	5.24
C ₃ H ₈	1.26
C ₃ H ₆	10.03
SUM C ₄ H ₁₀	0.83
SUM C ₄ H ₈	6.47
SUM C ₅ H ₁₁	5.33

RUN NO: AF-R12.5A

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	39.23	37.80
2	N2	2.72	2.97
3	CO	58.05	57.03
4	CH4	0.00	0.19
5	CO2	0.00	1.42
6	ETHANE	0.00	0.07
7	ETHYLENE	0.00	0.13
8	PROPANE	0.00	0.02
9	PROPYLENE	0.00	0.16
10	ISOBUTANE	0.00	0.00
11	N-BUTANE	0.00	0.01
12	T-BUTENE-2	0.00	0.03
13	BUTENE-1	0.00	0.03
14	ISOBUTYLENE	0.00	0.00
15	C-BUTENE-2	0.00	0.02
16	SUM C5	0.00	0.05
17	SUM C6	0.00	0.03
18	SUM C7	0.00	0.02
19	SUM C8	0.00	0.02
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
TOTAL		100.00	100.00
Mole Wt	lb/lb mole	17.81	18.51
Flows	SCFH	11458.52	10501.25
	lb mole/hr	29.64	27.16
	lb/hr	527.92	502.78

RUN NO: AF-R12.5B (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Start Date / Time	6/1/94	18.00	On-stream Time From Start-up (hr)	
End Date / Time	6/2/94	1.00	Start	126.00
			End	133.00

Reaction Conditions:

Temperature (°F)	589.4
Pressure (psig)	175.0
Space Velocity (sL/kg-Fe hr)	3771
Superficial Gas Velocity - Inlet (ft/sec)	0.20

Slurry Data:

Catalyst Weight (lb oxide)	297
Iron content in Catalyst (wt %)	61.06
Slurry Concentration (wt %)	14.1
Slurry Level (% of NDG Span)	93.0
Slurry Height (ft)	20.22
Average Gas Holdup (vol %)	6.1

Performance Results

CO Conversion, mole %	43.6
H2 Conversion, mole %	41.7
CO + H2 Conversion, mole %	42.8

Mass/Elemental Balance:

	Total lb/hr	C lb/hr	H lb/hr	O lb/hr	N lb/hr
Reactor Feed Gas	526.81	206.00	25.45	274.44	20.92
Main Gas Outlet	502.69	194.05	22.61	265.11	20.92
27.13 Reactor Wax	0.00	0.00	0.00	0.00	0.00
22.14 Light Wax	0.00	0.00	0.00	0.00	0.00
22.18 HC Phase	2.10	1.76	0.31	0.04	0.00
22.18 AQ Phase	5.80	0.57	0.65	4.58	0.00
Total Out	510.59	196.38	23.57	269.73	20.92
% Balance	96.9	95.3	92.6	98.3	100.0

CO Conversion Rate,	41.2
gmole CO converted/kg-Fe hr	
HC Production Rate,	322.2
grams of HC (CH _{2.5}) produced/kg-Fe hr	
Reactor Productivity	18.55
grams of HC (CH _{2.5}) /hr - lit of reactor vol.	

H2/CO Usage Ratio, mole/mole	0.70
H2/CO in Outlet, mole/mole	0.76

CO2 Selectivity, mole %	46.1
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HC Selectivity (CO2 free) mole % C :

CH4	10.40
C2H6	11.25
C2H4	3.96
C3H8	7.86
C3H6	13.20
SUM C4H10	3.27
SUM C4H8	10.41
SUM C5H11	7.33

Product Distribution: Selectivity (wt%)

Methane (C1)	14.6
Gas (C2 - C4)	62.9
Gasoline (C5 - C11)	19.6
Diesel (C12 - C18)	0.7
Wax (C19+)	2.2
Total	100.0

Alpha Estimate:

Carbon No.	Alpha	
C1-C14	Single	0.49

RUN NO: AF-R12.5B (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Compositions (mole%):		REACTOR FEED GAS	MAIN GAS OUTLET
Components			
1	H2	41.31	32.70
2	N2	2.45	3.32
3	CO	56.24	43.08
4	CH4	0.00	1.86
5	CO2	0.00	15.33
6	ETHANE	0.00	1.01
7	ETHYLENE	0.00	0.35
8	PROPANE	0.00	0.47
9	PROPYLENE	0.00	0.79
10	ISOBUTANE	0.00	0.01
11	N-BUTANE	0.00	0.14
12	T-BUTENE-2	0.00	0.19
13	BUTENE-1	0.00	0.11
14	ISOBUTYLENE	0.00	0.03
15	C-BUTENE-2	0.00	0.13
16	SUM C5	0.00	0.26
17	SUM C6	0.00	0.13
18	SUM C7	0.00	0.06
19	SUM C8	0.00	0.02
20	SUM C9	0.00	0.00
21	SUM C10	0.00	0.00
TOTAL		100.00	100.00
Mole Wt	lb/lb mole	17.27	22.37
Flows	SCFH	11791.41	8688.02
	lb mole/hr	30.50	22.47
	lb/hr	526.81	502.71

RUN NO: AF-R12.5B (Rev. 1)

TITLE: LIQUID PHASE FISCHER-TROPSCH (IIA) SYNTHESIS IN LAPORTE AFDU

Carbon No.	Compositions (wt%):			HC Phase						Aqueous Phase	
	Alcohols	Cis 2-Olefin	Trans 2-Olefin	n-Paraffin	1-Olefin	Branched Aliphatic	Other Olefins	Unidentified	Total	Compound	wt%
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%		
1	0.0								0.0	Methanol	3.1
2	0.0								0.0	Ethanol	5.4
3	0.0								0.0	1-Propanol	2.7
4	1.4								1.4	1-Butanol	1.1
5	3.0	0.1	0.0	0.3	0.0	0.0	0.0	0.9	4.3	1-Pentanol	0.5
6	2.1	0.3	0.5	0.3	0.4	0.0	0.2	1.1	4.9	1-Hexanol	0.1
7	1.3	0.5	0.8	0.9	0.5	0.7	0.0	0.0	4.7	1-Heptanol	0.0
8	1.0	0.6	1.0	1.0	0.6	0.2	0.5	0.4	5.3	1-Octanol	0.0
9	0.6	0.5	1.0	1.1	0.5	0.7	0.7	0.5	5.6	2-Propanol	0.4
10	0.4	0.4	0.8	1.0	0.4	0.3	0.7	0.7	4.7	2-Butanol	0.2
11	0.2	0.3	0.5	0.8	0.4	0.1	0.7	0.3	3.3	Isobutanol	0.1
12	0.0	0.2	0.3	0.7	0.3	0.2	0.3	0.1	2.1	2-Pentanol	0.0
13	0.2	0.1	0.3	0.6	0.3	0.0	0.3	0.0	1.8	Acetic Acid	3.1
14	0.0	0.0	0.2	0.6	0.0	0.0	0.2	0.0	1.0	Propanoic Acid	1.1
15	0.3	0.0	0.1	0.7	0.2	0.2	0.0	0.0	1.5	Butanoic Acid	0.5
16	0.2	0.0	0.2	0.8	0.2	0.0	0.0	0.3	1.7	Pentanoic Acid	0.1
17	0.0	0.0	0.2	0.9	0.2	0.8	0.5	0.3	2.9	Hexanoic Acid	0.0
18	0.5	0.2	0.2	0.9	0.2	0.5	0.8	0.9	4.2	Methyl Acetate	0.0
19		0.2	0.0	1.4	0.3	1.1	0.3	1.7	5.7	Ethyl Acetate	0.0
20		0.0	0.3	0.8	0.0	1.5	1.4	1.8	5.8	Ethyl Propanoate	0.0
21		0.0	0.4	0.7	0.4	1.2	1.0	2.5	6.6	Propyl Acetate	0.0
22		0.5	0.5	0.3	0.0	1.7	1.4	3.5	8.2	Butyl Acetate	0.0
23		0.0	0.4	0.0	0.3	2.5	3.0	3.1	9.3	Acetaldehyde	0.1
24		0.0	0.4	0.0	0.5	2.0	1.6	3.2	7.7	Propanaldehyde	0.0
25				0.0	0.2	1.3	0.9	2.5	5.2	Butanaldehyde	0.0
26				0.2	0.0	0.3	0.2	1.2	1.9	2-Propanone	0.2
27				0.0	0.0		0.0	0.5	0.5	2-Pentanone	0.1
28				0.0	0.0		0.0		0.0	2-Hexanone	0.0
29				0.0	0.0		0.0		0.0	Uncalibrated	0.2
30				0.0	0.0		0.0		0.0	Total Organics	19.3
> 30				0.0	0.0		0.0		0.0	Water by diff.	80.7
Total	12.6	3.9	8.4	14.0	5.9	15.3	14.7	25.5	100.3	Total	100.0

ALPHA ESTIMATION - RUN NO. AF-R12.5B