

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	20,863	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



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June 3, 1993

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JUN 07 1993

PROCESS ENGINEERING

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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, LO3096. 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 you 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 'DAB', with a long horizontal line extending to the right.

David A. Bucior
Senior Project Leader

DAB/jls

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BAT

February 10, 1994

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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:

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

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,

David A. Bucior
Senior Project Leader

DAB/jls

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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:

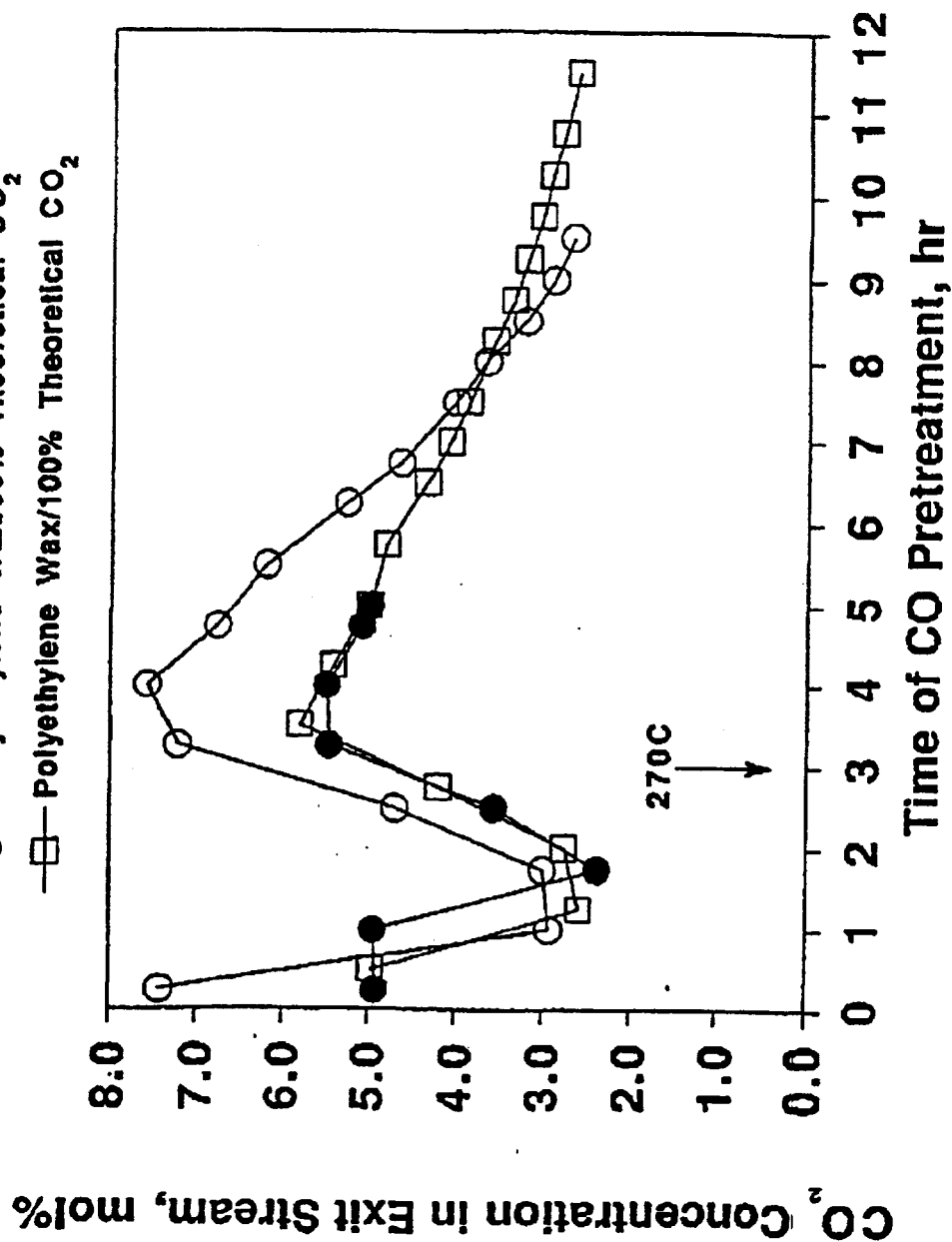
1. Gas Composition sampling requirements:
 - reactor in and out continuously
2. Flow measurement requirements:
 - reactor in at FI-126 and FI-187.
 - reactor out at FI-701.

REFERENCES:

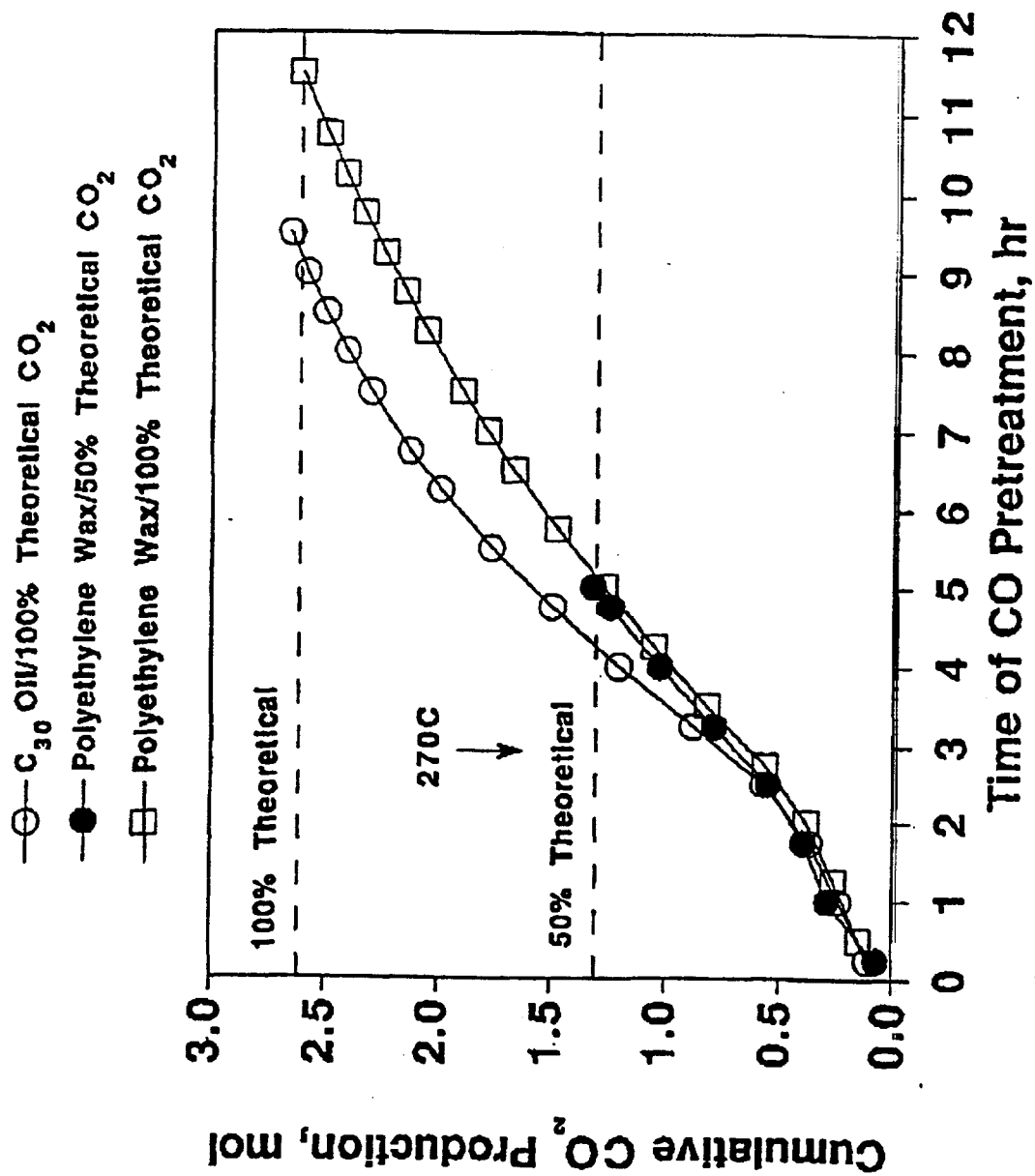
1. CAER's Activation Results (attached).

CAER AUTOCLAVE ACTIVATION

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



CAER AUTOCLAVE ACTIVATION



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.

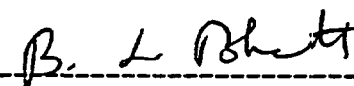
SPECIAL REMARKS:

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>
H ₂	40 %	4,718
CO	57 %	6,722
N ₂	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