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- 6. Heydorn, E. C., Schaub, E. S., Stein, V. E., Underwood, R. P. and Waller, F. J., "Recent Progress on Syngas Conversion to Isobutanol," DOE Indirect Liquefaction Contractors' Review Meeting, Peroperty, Peroperty, 1994.
- 7. Air Products and Chemicals, Innc., "Task 2.0: Run E-5, Gas Hold-up and Equipment Evaluation Studies," DOE Topipical Report under Contract No. DE-AC22-87PC90005, January 2, 1991.

APPENDIX A TEST AUTHORIZATIONS

Memorandum



Distribution

Dept./Loc.:

From:

E. S. Schaub/E. C. Heydom

Dept./Ext.:

PSG Process/LaPorte AFDU

Date:

3 March 1994

Subject:

Test Authorizations # 37,38, 389, & 40 for the LaPorte AFDU Methanol and Isobutanol Runs

Distribution:

W. C. Allen

D. P. Bernhard

B. L. Bhatt/K. G. Freidl/C. Chhen

D. M. Brown/W. R. Brown

D. P. Drown

V. E. Stein/J. M. Repasky

R. P. Underwood/B. A. Toseleland

Attached for your information are the 4 Test Authorizations for the upcoming Isobutanol/Mixed Alcohols Demonstration and the ELPMEOH run which will be used as a shakedown period prior to the Isobutanol run.

If you have any questions, pleasee contact either of us at (713) 479-5485.

E. S. Schaub/E. C. Heydorn

LaPorte A Alternative Fuels Development Unit (AFDU)

Sheet:

1 of 4

Date:

03/02/94

By:

ESS

RUN NUMBER:

AF-A5

APPROX. START DATE:

14 March, 1994

TITLE:

IN-SITU METHANODL CATALYST ACTIVATION PRIOR TO SPRING 94 RUN USING

DILUTE CO-F-RICH REDUCTION GAS

OBJECTIVE:

To activate the Liquid-Phase Methaanol (LPMEOH) synthesis catalyst.

SUMMARY:

Approximately 1250 lbs of BASF S33-86 oxide is to be slurried with Drakeol-10 oil, transferred to the 27.20 reactor and activated with dillilute CO-Rich syngas (3.5% in nitrogen). 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 gigear while loading catalyst to protect them from the dust and hot vapor which may be released from a the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

This operation will require the ventiting of unreacted hydrogen and CO. During a previous activation (performed under TEST AUTHORIZIZATION #29) the off-gas was blended with methane and burned in the flare. Previous calculations (foor TA #23) indicated that in the event a combustible mixture could not be maintained, there would be no ddanger to personnel from venting. The reduction gas flow rates to be used in this run are less than those e used in TA #23.

ENVIRONMENTAL IMPLICATIONSS:

Minimal, a flame will be maintained d at the flare. At 98% destruction efficiency, the CO emission rate would be 0.67 lb/hr.

SPECIAL REMARKS:

Hydrogen and CO concentrations ir in and out of the reactor must be monitored closely during the reduction. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The utility oil inlet temperature (TI 1244) to the 27.20 internal heat exchanger must not exceed a 200°F difference from these utility oil outlet temperature (TI-1246) or the reactor slurry temperature. These two temperature differentials are measured directly by TDI-1252 & TDI-1237. When adjusting flows or pressure, (care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. I. Heydorn, Plant Mgr

F S Schaub Process Engl

LaPorte Altilternative Fuels Development Unit (AFDU)

Sheet: Date: 2 of 4 03/02/94

By:

ESS

TEST DETAILS:

1. This reduction procedure follows previous methanol catalyst reductions from the LPIII ER-6 reduction (TEST AUTHORIZATI(ION #23), 1991 DME run (#25), and the 1992 LPSHIFT run (#29).

- 2. Charge the 28.30 prep tank withth 1875 lb of oil (265 gallons of Drakeol-10 at 80°F). The oil should be transferred to drums and weigighed using the scale for accurate measurement. As an approximation, meter the oil withth FQI-334 using a meter correction factor of actual = 1.027 * meter (meter should read 258.2 gal). I if the temperature differs from 80°F a corrected oil volume should be used. Heat this oil to 150-2000°F.
- 3. Fill the 27.14 intermediate V/L siseparator to 25 nuts on LG-358 with approximately 100 gallons of Drakeol-10 oil from storage. Noote the FQI-334 readings before and after the addition.
- 4. When the prep tank oil is at 1500-200°F, add 1250 lb of methanol catalyst (BASF S3-86- 2 full drums of Lot 851-1642 and the balance (3+) drums of Lot 553-5072). Add the catalyst very slowly to make a 40 wt% oxide slurry. | Keep the slurry well stirred to prevent agglomeration of the catalyst.
- 5. Heat the slurry to 200°F and conntinue agitation, under nitrogen, for at least 2 hours to ensure good mixing.
- 6. When the catalyst and oil have t been completely mixed, withdraw a sample of slurry.
- 7. Establish gas flow through the rereactor using nitrogen through V-2627 to prevent slurry back-flow into the distributor. Vent the gass through PV-1261.
- 8. Pressure transfer the slurry to thine reactor and verify operation by noting level with the nuclear density gauge (NDG- estimated 1 level: 25 to 28 ft.)
- 9. Flush out the prep tank with 2833 lb of oil (40 gallons of Drakeol-10 at 80°F). Measure the oil as in step 2 (meter should read approximately 38.9 gal). Pressure transfer the flush oil to the reactor and verify level with the NDG (LILI-1242).
- 10. Close V-645 to prevent utility oil il flow back to the prep tank and establish full utility oil flow through the 27.20 internal heat exchangger.
- 11. Pressurize the reactor loop to 1000 psig.
- 12. Begin heating the slurry to 200°FF, following TAVR on the DEC console. Check that the slurry temperatures are in reasonable a agreement. Verify that the slurry is well mixed by performing a NDG scan.

LaPorte A Alternative Fuels Development Unit (AFDU)

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By:

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13. Establish CO-Rich reduction ggas flow at 25,910 SCFH (on FI-126) and vent the flow through PV-170. Establish the following ecomposition:

	<u>Composition</u>	Est. Flows (SCFH)
H2	1.2%	311
CO	1.8	466
CO2	0.5	130
N2	96.5	25,003
	100.0	25,910

MW == 27.8, SCF evaluated at 70°F, 14.7 psia Target space velocity = 1 1200 sL/h-kg; Target starting inlet superficial velocity = 0.73 ft/sec

- 14. When the reactor temperaturere reaches 200°F, bring reduction gas to the reactor slowly and close the nitrogen purge (V-2627). I Establish a final flow to the reactor of 25,910 SCFH. Maintain flow and reducing gas composition n as specified in step 13. The temperature-programmed activation consists of the following steps:
 - Heat the slurry at a target rate of 15°F/hr (no more than 18°F/hr, 10°C) until the slurry temperature reaches 39'92°F (200°C).
 - Hold the slurry temperatature at 392°F for 12 hours.
 - Heat the slurry at 15°F/h/hr until the slurry temperature reaches 464°F (240°C).
 - Hold the slurry temperaturure at 464°F for 1 hour (or longer if syngas uptake is still apparent).

H2 and CO concentrations are to be measured continuously for the feed and effluent streams. As long as the cumulative H2 pluus CO consumption at a given temperature is equal to or greater than the autoclave reduction data tithen the activation is proceeding well. Figure 1 shows the consumption profile vs temperature from the labs. If the cumulative consumption curve falls below the autoclave curve, consult ththe process or research engineer to reduce the heatup rate.

If the H2+CO concentration in the effluent falls below 0.1 mole %, increase the inlet H2+CO concentration per the instructidions of the process or research engineer. The objective here is to prevent reduction gas starvatition.

During the 392°F hold period, I, it may become necessary to maintain this temperature beyond 12 hours until the difference between inlet and outlet H2+CO concentration falls below 0.05 mole %.

15. The slurry level should be maiaintained between 90 and 95% of NDG range (approximately 40 ft.) by using LIC 1242 to control ththe makeup oil rate. Note that as the reactor is heated to 464 F, the slurry will expand. At the samme time, some of the oil will be lost in the reactor effluent. If authorized by the process engineer or the plant manager, additional makeup oil can be added to the system via the 27.14 by foollowing the standard procedure; FQI-334 readings and the change in

TEST AUTHORIZATION # 37 LaPorte Altitemative Fuels Development Unit (AFDU)

Sheet:

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By:

ESS

level of the 27.14 should be reccorded before and after each addition. It is important to note that the discharge valve of the 10.522.01 and 02 pumps should be used to throttle to the 100 psig reactor pressure. The pressure a in the sump of the 21.11 should be at 150 psig or less.

- 16. Record any indication of densityty or viscosity change, such as a change in the pressure drop across the reactor or shaking of f the reactor during heatup and reduction.
- 17. When reduction has been completed, scan the reactor with the NDG. Record levels in the 21.11 and 27.14. Add fresh oil to 27.114 to bring the level up to 25 nuts on LG-358. This charge should be drawn from storage; note the e FQI-334 readings before and after addition.

TA #37 is done, consult TEST AUTH(IORIZATION #38 for the next step.

ANALYTICAL REQUIREMENTS:

- 1. Catalyst sampling requirements:
 - slurried oxide catalystst from prep tank before reduction, and,
 - from the reactor, slummed reduced catalyst

Exact quantities to be determinined by operations, process, and research.

- 2. Composition sampling requirements:
 - reactor in and out conntinuously
 - H2 and CO are criticaal
 - CO2 and N2 are also o required
- 3. Flow measurement requiremeents:
 - reactor in at FI-126 arand FI-299

REFERENCES:

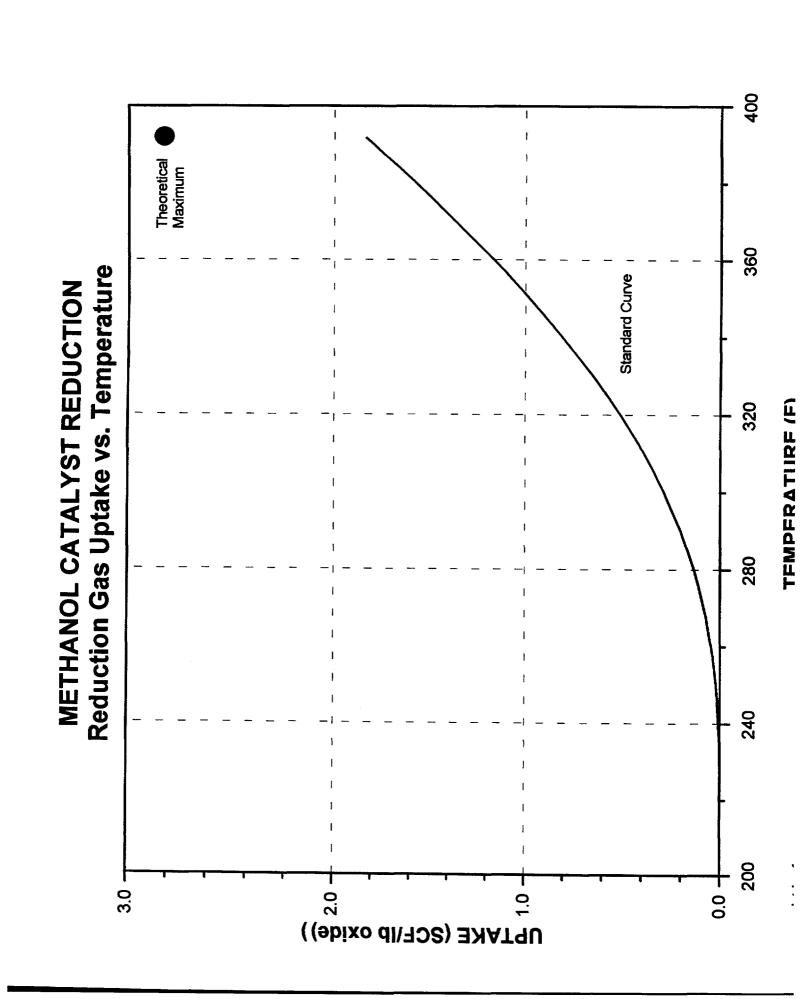
1. TEST AUTHORIZATION # 23 : Projocedure for previous in-situ activation.

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

Description			Reduction	MEOH-1	MEOH-2
Duration		days	2	2	2
Symgas	:	:	:	TEXACO	KINGSPORT
Inlet Space Velocity	:	sL/kg-hr	1,200	6,700	4,000
Reactor Pressure	PIC-1247	bisd	9	750	735
REACTOR					
Pressure	PIC-1247	psig	18 81	750	735
Temperature	TI-1233	u	:	482	482
Heat Duty	₹	MM BTU/hr		1.14	1.21
Inlet Superficial Velocity	:	f/sec	0.730	0.842	0.514
Outlet Superficial Velocity	:	ff/sec	• •	0.644	0.348
Liquid Level	LI-2142	% span	90 - 95%	100%	100%
Catalyst Load		Q	1,250	1,250	1,250
Cat Weight Fraction	:	8	40.0%	42.4%	40.0%
Car yyelyn Tiachun		\$	~\$v.o.₩°	42.4%	₩3.0%
Vapor Void Fraction		8	23 - 30%	42.9%	34.5%
FEED FLOWS					
LP H2	FIC-101	Scff	311	17,970	20,770
လ	FIC-104	scfh	466	15,581	12,694
C02	FIC-107	Scfh	130	1,192	1,065
N2	FIC-111	scfh	25,003	25	27.1
01.10 Total Flow	FI-726	scfh	25,910	34,800	34,800
ch on	EIC 4200	ng-co	c	8 685	4 027
04 20 Beamle	EIC 24R	3		101 084	46.578
OLEO RECYCLE	22-21		,	100,101	2/2/2
TO'S FOMP INSECTION	7		-		
l otal Flow	17-17		OJAZ	7010	7610
МЕОН		WL%	* * *		;
С2ОН	:	wt%			
СЗОН		wt%			
REACTOR FEED					
Target Feed Temp	TI-1253	L	1	362.2	390.7
Feed Dewpoint	:	Ŀ		0.68	78.2
Total Dry Flow	FI-1216	Scfh	25,910	145,449	86,316
H2		mol%	1.20%	34.71%	60.91%
8	:	%lom	1.80%	50.57%	24.49%
N2	:	Mol%	96.50%	1.00%	3.90%
005		mol%	0.50%	12.89%	10.03%
MEOH	;	30m	%000	0.71%	0.42%
ETOH	:	%Jom	0.00%	0.00%	0.00%
PROH		%lom	%00.0	0.00%	0.00%
C1	:	mol%	%00 O	0.03%	O 088

DEMONSTRATION
194 IBOH
SPRING
N FOR
UN PLAN
2

21.11 Feed/Product Exchanger Feed Inlet Temp Feed Outlet Temp Total Feed to 02.63 Temp Reactor Eff. Inlet Temp Reactor Eff. Outlet Temp Reactor Eff. Dew Temp Reactor Eff. Dew Temp	er	:	Reduction	MEOH-1	MEOH-2
1.11 Feed/Product Exchang Feed Inlet Temp Total Feed to 02.63 Temp Reactor Eff. Inlet Temp Reactor Eff. Outlet Temp Reactor Eff. Outlet Temp Reactor Eff. Dew Temp	er Till 1987				
Feed Inlet Temp Feed Outlet Temp Total Feed to 02.63 Temp Reactor Eff. Inlet Temp Reactor Eff. Outlet Temp Reactor Eff. Dew Temp	1207				
Feed Outlet Temp Total Feed to 02.63 Temp Reactor Eff. Inlet Temp Reactor Eff. Outlet Temp Reactor Eff. Outlet Temp	152/	ட		172.2	209.3
Total Feed to 02.63 Temp Reactor Eff. Inlet Temp Reactor Eff. Outlet Temp Reactor Eff. Dew Temp	TI-1263	Ŀ		402.3	401.6
Reactor Eff. Inlet Temp Reactor Eff. Outlet Temp Reactor Eff. Dew Temp	TI-1216	Œ		362.3	390.7
Reactor Eff. Outlet Temp Reactor Eff. Dew Temp	TI-1262	u.	:	482.0	482.0
Reactor Eff. Dew Temp	TIC-1260	ı.	3	280.0	280.0
EACTOR FEEL LIENT	:	iL.	::-	226.9	267.8
Total Flow	FI-196	scfh	1	119,495	62,776
H2	:	mol%		20.75%	46.48%
03	:	‰lou.	: :	50.50%	14.66%
N2	;	‰joш	:	1.21%	5.36%
C02	:	%lom		15.85%	14.02%
MEOH	:	%jom	1	11.20%	17.88%
ETOH	:	mol%		0.08%	0.24%
ETOIL		3612111		0.00%	0.ET26
PROH		mol%		0.02%	%80.0
C40H	::	mol%		0.01%	0.03%
ВОН	:	mol%		%00.0	0.01%
C50H+	::	%lom		0.01%	0.02%
5	:	%lour		0.05%	0.16%
				%99.66	98.95%
PRODUCT RECOVERY					
Syndas to Backend Flow	FI-682	scfh	:	None	None
22.11 to Flare Flow	FI-237	scf		875	299
Main Flare Flow	FI-245	scfh	•••	3,867	3,867
Product Flow	:	bdb		3,897	3,527
BACK-END					
MEOH Circulation	FIC-814	mdb	None	None	None
MEOH to 07.10 Temp	TI-814	Т	•		•
1	100	ı			
107.10 OH 1emp	11-12/3	-	•	-	•
07.20 OH to Flare Flow	FI-7291	Soff		:	
07.20 Reboiler Temp	TIC-7339	ட	,		
07.22 Steam Pressure	PIC-7338	psia	1 1	•	:
07.22 Steam Usage	FI-7338	ξģ		-:-	
21.80 CO2 Usage	:	TPD		:	
Total CO2 Usage	Fd+21.80	TPD	0.18	1.63	1.45
RECYCLE FEED					
H2		%lom		23.38%	82.67%
00	:	2 mol%	1 .	56.85%	18.13%
N2	:	mol%		1.36%	6.65%
203	:	mof%		17 22%	16.30%
MEOH	:	₩ol%		101%	0.78%
MICOLI C1		2016		200	0.16%
5	:	R 5		00 8592	00 K04



LaPorte AlkItemative Fuels Development Unit (AFDU)

Sheet: 1 of 3 Date: 03/02/94 **ESS** By:

RUN NUMBER:

AF-R9

APPROX. START DATE:

15 March, 1994

TITLE:

METHANOL SYNTHEESIS WITH BASF S3-86 CATALYST

OBJECTIVE:

To study the performance of S3-86 n methanol catalyst in the 27.20 reactor train.

SUMMARY:

Upon completion of the activation steep (AF-A5), the reactor feed will be adjusted to a Texaco gas composition (35% H2, 51% CO, 13%% CO2, 1% N2). For approximately 2 days, the conditions will be targeted at 750 psig, 482°F, 6,700 slsL/kg-hr space velocity, and 40 wt% oxide in oil. After 2 days, the gas composition will be switched to aa Kingsport LPMEOH gas composition for three days of operation (60.7% H2, 24.4% CO, 10.0% CO2, ; 3.89% N2). The objective is to condition the oil, allow the hyperactivity of the catalyst to decline, breaak-in the new DCS controls, and ultimately line-out at a steady rate of methanol production while collectifting data for H2-rich reactor feed gas.

TEST DETAILS:

See page 2.

ANALYTICAL COMMENTS:

See page 3.

SAFETY IMPLICATIONS:

Protective gear including face shield I should be wom during slurry sampling.

ENVIRONMENTAL IMPLICATIONS: :

Minimal.

SPECIAL REMARKS:

The high pressure hydrogen pipe linese will be in use during run AF-R9. The CO2 removal system will not be in operation. Special sample bormbs will be used to collect samples of the methanol product produced during case AF-R9.2.

AUTHORIZATIONS:

dorn, Plant Mgr E. S. Schaub, Process Engr

LaPorte / Alternative Fuels Development Unit (AFDU)

Sheet: 2 of 3 Date: 03/02/94 By: ESS

TEST DETAILS:

- Upon completion of the catalylyst activation (AF-A5), switch from reduction gas to Texaco-type gas by following the standard proocedure. The CO2 removal section should NOT be operating during this run (V-2001,V-2003,V-20004,V-2006 shut; V-2000 open). In the event of a premature shutdown, consult TA #20 (RUN EE-05) for appropriate standby conditions.
- 2. Increase the reactor pressure e to 750 psig and control the slurry temperature at 482°F. Slowly increase the reactor feed rate e to 25,000 SCFH while maintaining slurry level at 95% of NDG span. When the plant has lined out, t, the reactor feed composition should correspond closely to case AF-R9.1 (refer to Table). Once ththe compositions are lined out, slowly introduce recycle flow and back off the fresh feed flowrates unntil they match the targets outlined in the Table for case AF-R9.1. Note that the HP hydrogen pipipeline is in service during cases AF-R9.1 and AF-R9.2.
- 3. When the target feed rate has been achieved, put LIC-1242 in automatic to control slurry level at 95%. Adjust the fresh feed flow to achieve an initial purge flow rate of approximately 3,000 SCFH. Maintain reactor feed flow and reactor temperature and pressure at the case AF-R9.1 values for a nominal 24 hour period.
- 4. During the first 24 hours, the s syngas conversion across the reactor will fall as the catalyst loses it's hyperactivity. The purge flow will increase and the reactor feed composition will be changing during this period. When theese rates of change diminish, fine tune the fresh feed flow to reach the desired reactor feed compositition as specified for case AF-R9.1. The ultimate purge rate should be around 3,900 SCFH.
- 5. After the initial break-in periood, begin to increase rates to maximize production of methanol. Monitor the air-cooler loading 3 and temperature difference between the utility oil and the slurry and utility oil inlet & outlet using TITDI-1237 and TDI-1252. Both of these temperature differences must be below 200°F.
- 6. The composition of the methalanol product is to be monitored every 8 hours. The target oil content of the methanol product should be <=0.2 wt%. If the oil content is higher, lower the 21.11 effluent outlet TiC-1260 setpoint.
- 7. Maintain conditions for approximately 2 days. After conferring with the process engineer or plant manager, switch to AF-R9.2 run conditions (Kingsport gas). Run this data period for approximately 3 days.
- 8. Liquid samples of the methannol product will be collected in special sample bombs and shipped to Allentown for detailed analysisis during case AF-R9.2. The samples will be collected downstream of the 22.11 separator. Consisult with the process engineer and analytical representative for the frequency and manner of takiking the samples.

LaPorte Alalternative Fuels Development Unit (AFDU)

Sheet: 3 of 3 Date: 03/02/94 By: ESS

9. When notified by the plant mannager that case AF-R9.2 is complete, de-pressurize the plant, and drain the slurry from the 27.20 refractor using the prep tank as an intermediate hold point using the standard shutdown procedures.s. Drain the 22.10, 22.15 and 22.16. Proceed with TEST AUTHORIZATION #39.

ANALYTICAL COMMENTS:

- 1. Catalyst sampling requiremennts:
 - slurried catalyst at ennd-of-run.

Exact quantities to be determinined by operations, process, and research.

- 2. Continuous composition samppling requirements (GC):
 - fresh feed.
 - reactor in.
 - reactor out,
 - recycle
 - 22.10 overheads
- 3. Periodic composition sampling requirements (GC):
 - 22.11 off-gas (frequency to be determined by operations & process)

Periodic composition sampling requirements (LC):

- methanol product (evevery 8 hours during first two days, twice a day thereafter)
- 4. Flow measurement requiremeents:
 - fresh feed,
 - reactor in,
 - reactor out,
 - recycle.
 - purge.
 - 22.11 off-gas,
 - methanol product

REFERENCES:

- 1. TEST AUTHORIZATION #20 Prolocedures for reactor standby during shutdown.
- 2. STANDARD STARTUP PROCEDUURES FOR MeOH-ONLY OPERATION

KINGSPORT 4,000 735 MEOH-2 99.83% 20,770 12,694 1,065 60.91% 24.49% 3.90% 34,800 4,937 86,316 0.42% 1.21 0.514 0.348 1,250 40.0% 40.0% 34.5% 0.00% 0.00% 390.7 zero : 78.2 271 : : 6,700 750 8,665 101,984 145,449 34.71% 50.57% 17,970 15,581 1,192 57 34,800 AF-R9.1 MEOH-1 1.00% 12.89% 0.71% 0.00% 0.00% 0.03% 99.91% 1.14 0.842 1.250 1.250 42.4% 42.4% 42.9% 362.2 25 28 28 zero 1 ; : Reduction 90 - 95% 1,250 40.0% 40.0% 23 - 30% 0.00% 0.00% 0.00% 466 130 130 25,003 25,910 25,910 1.20% 1.80% 96.50% 0.50% 0.00% 0.730 : : 8 ! zero ; ; 00 sL/kg-hr psig % span MM BTU/hr days ff/sec f/sec psig T mol% **201 201** %lom **300** mol% scfh SCH SCH scfh wt% mol% **₩**% Scf ₽ 888 PIC-1247 FIC-104 FIC-1200 FIC-246 PIC-1247 TI-1233 LI-2142 FIC-107 FI-1216 FIC-111 FI-1221 TI-1253 ; FI-726 : ; ; : ; : ; 1 : : : : : ; Outlet Superficial Velocity Inlet Superficial Velocity Cat Weight Fraction Cat Weight Fraction Vapor Vold Fraction 10.95 PUMP INJECTION Syngas Inlet Space Velocity Target Feed Temp Feed Dewpoint Reactor Pressure 01.10 Total Flow 01.20 Recycle REACTOR FEED Total Dry Flow Catalyst Load Temperature Liquid Level Description FEED FLOWS Heat Duty Total Flow Pressure Duration REACTOR HP H2 MEOH C20H C30H MEOH ETOH PROH LP HZ 005 **CO2** ၀ 오 ၀ 呈 S ပ

RUN PLAN FOR SPRING 1994 IBOH DEMONSTRATION

Page 2 of 4

					·
Description			Reduction	MEOH-1	MECH-Z
21.11 Feed/Product Exchange					
Feed inlet Temp	TI-1257	Ŀ		172.2	209.3
Feed Outlet Temp	TI-1263	Ŧ		402.3	401.6
Total Feed to 02.63 Temp	TI-1216	Ŀ	1	362.3	390.7
Reactor Eff. Inlet Temp	TI-1262	ш		482.0	482.0
Reactor Eff. Outlet Temp	TIC-1260	L		280.0	280.0
Reactor Eff. Dew Temp		L	3	226.9	267.8
REACTOR EFFLUENT					
Total Flow	FI-196	scfh		119,495	62.776
2		%lom		20.75%	46.48%
8		%lom	1 1	5050%	14 66%
N2	: :	%Jour		1.21%	5.36%
002	:	%lom	:	15.85%	14 02%
MEOH	:	%lom		11.20%	17.88%
FTOH		2016		7000	2000
EIGH	;	RE	: :	0.00% 0.00%	0.24%
PROH		%Jour		0.02%	0.08%
C40H	:	Mof%	:	0.01%	0.03%
BOH	:	mol%		0.00%	0.01%
C50H+		% ou		001%	2000
73		2 20 00		2000	20.0
5	:	2		8000 0000	80.0
				33.00%	80.80%
Support RECOVERT	11 600	4		Mens	
Syngas to backend Flow	1-007	SCIII	•	None	None
ZZ.11 to Flare Flow	FI-23/	SCIII		6/2	/99
Main Flare Flow	FI-245	scth		3,867	3,867
Product Flow		gbd		3,897	3,527
BACK-END					
MEOH Circulation	FIC-814	mdg	None	None	None
MEOH to 07.10 Temp	TI-814	Ŀ		:	:
07.10 OH Temp	TI-1275	L	:		
07.20 OH to Flare Flow	FI-7291	scff		,	
07.20 Reboiler Temp	TIC-7339	ட			
07.22 Steam Pressure	PIC-7338	Dig B	-	:	-
07.22 Steam Usage	FI-7338	ID/hr		:	1
21.80 CO2 Usage	:	TPD			:
Total CO2 Usage	Fd+21.80	TPD	0.18	1.63	1.45
RECYCLE FEED					
H2	:	%lom		23.38%	57.67%
00		%lom		56.85%	18.13%
N2	:	%Joш	:	1.36%	6.65%
C02	;	Mot%	:	17.22%	16.30%
MEOH	:	%jom		1.01%	0.78%
2		2		10, 1	
<u> </u>	: : :	<u>و</u>		8700	0.16%

RUN PLAN FOR SPRING 1, 34 IBOH DEMONSTRATION

LaPorte A Alternative Fuels Development Unit (AFDU)

Sheet:

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Date:

03/02/94

By:

ESS

RUN NUMBER:

AF-A6

APPROX. START DATE:

24 March, 1994

TITLE:

IN-SITU ISOBUTANGOL CATALYST ACTIVATION PRIOR TO SPRING 94 RUN USING

DILUTE CO-f-RICH REDUCTION GAS

OBJECTIVE:

To activate the Liquid-Phase Mixed d Alcohols synthesis catalyst.

SUMMARY:

Approximately 1106 lbs of CS-dopeed BASF S3-86 oxide is to be slurried with Drakeol-10 oil, transferred to the 27.20 reactor and activated v with dilute CO-Rich syngas (4.0% in nitrogen). 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 g gear while loading catalyst to protect them from the dust and hot vapor which may be released from n the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

This operation will require the ventiiting of unreacted hydrogen and CO. During the previous similar activation (performed under TEST / AUTHORIZATION #29) the off-gas was blended with methane and burned in the flare. Previous calculations (for TA #23) indicated that in the event a combustible mixture could not be maintained, there would be no danger to personnel from venting. The reduction gas flow rates to be used in this run are less; than those used in TA #23.

ENVIRONMENTAL IMPLICATIONSS:

Minimal, a flame will be maintained at the flare. At 98% destruction efficiency, the CO emission rate would be 0.6 lb/hr.

SPECIAL REMARKS:

Hydrogen and CO concentrations ir in and out of the reactor must be monitored closely during the reduction. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The utility oil inlet temperature (TI 1244) to the 27.20 internal heat exchanger must not exceed a 200°F difference from these utility oil outlet temperature (TI-1246) or the reactor slurry temperature. These two temperature differentials are measured directly by TDI-1252 & TDI-1237. When adjusting flows or pressure, (care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. I. Heydorn, Plant Mgr

E. S. Schaub, Process Engr

LaPorte Altdtemative Fuels Development Unit (AFDU)

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TEST DETAILS:

1. This reduction procedure follows/s the successful ER-6 reduction (refer to TEST AUTHORIZATION #23)

- 2. Charge the 28.30 prep tank with h 1659 lb of oil (235 gallons of Drakeol-10 at 80°F). The oil should be transferred to drums and weigighed using the scale for accurate measurement. As an approximation, meter the oil with:h FQI-334 using a meter correction factor of actual = 1.027 * meter (meter should read 228.4 gal). If If the temperature differs from 80°F a corrected oil volume should be used. Heat this oil to 150-2000°F.
- 3. Fill the 27.14 intermediate V/L seeparator to 25 nuts on LG-358 with approximately 100 gallons of Drakeol-10 oil from storage. Nobte the FQI-334 readings before and after the addition.
- 4. When the prep tank oil is at 150-3-200°F, add 1106 lb of isobutanol catalyst (CS-BASF S3-86)). Add the catalyst very slowly to mmake a 40 wt% oxide slurry. Keep the slurry well stirred to prevent agglomeration of the catalyst.
- 5. Heat the slurry to 200°F and conntinue agitation, under nitrogen, for at least 2 hours to ensure good mixing.
- 6. When the catalyst and oil have bleen completely mixed, withdraw a sample of slurry.
- 7. Establish gas flow through the receator using nitrogen through V-2627 to prevent slurry back-flow into the distributor. Vent the gasis through PV-1261.
- 8. Pressure transfer the slurry to thhe reactor and verify operation by noting level with the nuclear density gauge (NDG estimated klevel: 22-25 ft.)
- 9. Flush out the prep tank with 2833 lb of oil (40 gallons of Drakeol-10 at 80°F). Measure the oil as in step 2. Meter should read approximately 38.9 gallons. Pressure transfer the flush oil to the reactor and verify level with the NDG.
- 10. Close V-645 to prevent utility oil I flow back to the prep tank and establish full utility oil flow through the 27.20 internal heat exchangeer.
- 11. Pressurize the reactor loop to 1000 psig.
- 12. Begin heating the slurry to 200°FF, following TAVR on the DEC console. Check that the slurry temperatures are in reasonable a agreement. Verify that the slurry is well mixed by performing a NDG scan.

LaPorte AAltemative Fuels Development Unit (AFDU)

Sheet: 3 of 4 Date: 03/02/94

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13. Establish CO-Rich reduction gras flow at 22,920 SCFH (on FI-126) and vent the flow through PV-170. Establish the following ecomposition (Note that this composition is slightly different than those used for typical methanol catalalyst reductions.):

	Composition	Est. Flows (SCFH)
H2	1.4%	321
CO	2.1	481
CO2	0.5	115
N2	96.0	<u>22,003</u>
	100.0	22,920

MW == 27.7, SCF evaluated at 70°F, 14.7 psia
Target space velocity = 1 1200 sL/h-kg; Target starting inlet superficial velocity = 0.64 ft/sec

- 14. When the reactor temperature reaches 200°F, bring reduction gas to the reactor slowly and close the nitrogen purge (V-2627). I Establish a final flow to the reactor of 22,910 SCFH. Maintain flow and reducing gas composition as specified in step 13. The temperature-programmed activation consists of the following steps:s:
 - Heat the slurry at a targget rate of 10°F/hr (no more than 15°F/hr) until the slurry temperature reaches 392°F (200°C). This ramp rate is slower than methanol reduction procedures. Care should be taken to contrtrol this ramp rate at the beginning of the reduction.
 - Hold the slurry temperature at 392°F for 12 hours.
 - Heat the slurry at 10°F/h/hr until the slurry temperature reaches 464°F (240°C).
 - Hold the slurry temperatature at 464°F for 1 hour (or longer if syngas uptake is still apparent).

H2 and CO concentrations are to be measured continuously for the feed and effluent streams. As long as the cumulative H2 pluus CO consumption at a given temperature is equal to or greater than the autoclave reduction data then the activation is proceeding well. Figure 1 shows the consumption profile vs temperature from the labs. If the cumulative consumption curve falls below the autoclave curve, consult the process or research engineer to reduce the heatup rate.

If the H2+CO concentration inn the effluent falls below 0.1 mole %, increase the inlet H2+CO concentration per the instructidions of the process or research engineer. The objective here is to prevent reduction gas starvatidion.

During the 392°F hold period, I, it may become necessary to maintain this temperature beyond 12 hours until the difference between inlet and outlet H2+CO concentration falls below 0.05 mole %.

15. The slurry level should be maiaintained between 80 and 90% of NDG range (estimated 35 ft.) by using LIC 1242 to control the n makeup oil rate. Note that as the reactor is heated up to 464 F, the slurry will expand. At the samme time, some of the oil will be lost in the reactor effluent. If

LaPorte Altilternative Fuels Development Unit (AFDU)

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ESS

authorized by the process enginneer or plant manager, additional makeup oil can be added to the system via the 27.14 by followining the standard procedure; FQI-334 readings and the change in the level of the 27.14 should be reccorded before and after each addition. Note that the discharge valve of the 10.52.01 and 02 puumps should be used to throttle to the 100 psig reactor pressure. The pressure in the sump of thee 21.11 should be at 150 psig or less.

- 16. Record any indication of density y or viscosity change, such as a change in the pressure drop across the reactor or shaking of f the reactor during heatup and reduction.
- 17. When reduction has been completed, scan the reactor with the NDG. Record levels in the 21.11 and 27.14. Add fresh oil to 27.114 to bring the level up to 25 nuts on LG-358. This charge should be drawn from storage; note the e FQI-334 readings before and after addition.
- 18. Withdraw a slurry sample.

TA #39 is done, consult TEST AUTH(IORIZATION #40 for the next step.

ANALYTICAL REQUIREMENTS:

- 1. Catalyst sampling requirementnts:
 - slurried oxide catalystst from prep tank before reduction, and,
 - from the reactor, slummed reduced catalyst

Exact quantities to be determinized by operations, process, and research.

- 2. Composition sampling requirements:
 - reactor in and out conntinuously
 - H2 and CO are criticaal
 - CO2 and N2 are also p required
- 3. Flow measurement requiremeents:
 - reactor in at FI-126 arand FI-299

REFERENCES:

1. TEST AUTHORIZATION # 23 : Prolocedure for previous in-situ activation.

Page 3 of 4

RATION
NBI
DEMO
IBOH
1994
BPRING
FOR
PLAN
RUN

Secretary Secr	RUN				AF-A6	AF-R10.1	AF-R10.2	_		AF-R10.5	AF-R10.6	AF-R10.7	_	AF-R10.9	AF-R10.9 AF-R10.10 AF-R10.1	AF-R10.11
Colored Colo	Description				Reduction	IBOH-1	IBOH-2	BOH-3	IBOH-4		1BOH-6	IBOH-7	BOH-8	BOH-9	IBOH-10	IBOH-11
Pic.1247 Paig 100 750 750 750 1300 1735	Duration		:	days	2	2		2	2	2	ļ	1	1	1	1	2
Pic-1247 Psig 100 750 750 750 1,300 1,735 1,	Symgas		;			SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	SHELL	TEXACO
PIC-1247 Psig 100 750 750 750 1,300 1,735 1,	Inlet Space	Velocity	:	sL/kg-hr	1,200	2,000	3,000	8,200	8,200	8,200	3,000	5,000	2,000	5,000	5,000	3,000
PIC-1247 PSig 100 750 750 750 1,300 1,735 1,	Reactor Pre	ssure	PIC-1247	psig	100	750	750	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
Pic-1247 psig 100 750 750 750 1,300 1,735 1,																
PIC-1247 Paig 100 750 750 1300 1,735 1	REACTOR															
Til-1233 F 572 5	Pressure		PIC-1247	psig	\$	750	350	750	1,300	1,735	1,735	1,300	1,300	1,300	750	1,300
MM BTUM	Temperatur		TI-1233	ıL	:	572	572	572	572	572	572	572	572	225	572	572
ty f/sec 0.640 0.611 0.366 1.002 0.583 0.481 0.105 cdy f/sec 0.517 0.348 0.859 0.461 0.329 0.1107 L12142 kspan 80-90% 40.2% 100% 1107 <th>Heat Duty</th> <th></th> <th>Š</th> <th>A BTU/hr</th> <th></th> <th>0.53</th> <th>0.52</th> <th>0.59</th> <th>-</th> <th>1.97</th> <th>1.01</th> <th>1.12</th> <th>0.85</th> <th>1.03</th> <th>0.53</th> <th>0.59</th>	Heat Duty		Š	A BTU/hr		0.53	0.52	0.59	-	1.97	1.01	1.12	0.85	1.03	0.53	0.59
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11-2142 % span 80 - 90% 100%	Outlet Supe	rficial Velocity	::	f/sec	:	0.517	0.348	0.859	0.461	0.329	0.115	0.275	0.327	0.291	0.517	0.168
1.07 1,107	Liquid Level		LI-2142	% span	80 - 90%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
Tild	Catalyst Los	2	:	۵	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107	1,107
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mol% 0.00% 0.04% 0.03% 0.04% 0.04% 0.03% 0.03% 0.03% 0.03% 0.03% 0.03% 0.00%	C02		:	%lom	0.50%	2.97%	1.91%	2.99%	2.91%	2.98%	2.97%	2.88%	2.68%	2.87%	2.97%	12.77%
mol% 0.00%	MEOH		:	%lon	0.00%	0.04%	0.03%	0.04%	0.04%	0.03%	0.03%	%E0'0	7.89%	2.32%	0.04%	0.55%
mol% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.00% 0.53% 1.04% 0.95% 1.15%	ЕТОН		:	Mol%	%00.0	0.00%	%00.0	0.00%	%00.0	0.00%	%00.0	0.00%	0.33%	0.24%	0.00%	0.01%
mol% 0.00% 0.89% 0.53% 1.04% 0.95% 1.15%	PROH		:	%Jou	%00.0	0.00%	0.00%	%00:0	0.00%	0.00%	%00'0	9.00.0	0.61%	0.35%	0.00%	0.00%
	5		;	%lom	0.00%	0.84%	0.99%	0.53%	1.04%	0.95%	1.15%	1.15%	1.23%	1.15%	0.84%	0.73%
99.98% 100.09% 100.13% 100.28% 100.28% 100.25%					100.00%	%86.66	100.09%	100.13%	100.28%	100.28%	100.25%	100.14%	100.22%	99.87%	99.98%	99.53%