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Volume IX

PRODUCTION OF JET FUELS FROM COAL DERIVED LIQUIDS

VOL IX - RESULTS OF BENCH - SCALE AND PILOT PLANT TESTING

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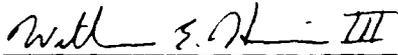
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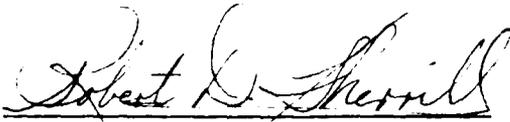


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SUMMARY

Amoco and Lummus Crest are contracted with the Department of Energy to develop an upgrading scheme for the liquid by-products (tar oil, phenols, and naphtha) produced by the Great Plains Coal Gasification plant in Beulah, North Dakota. These streams are currently burned in the utility boilers and steam superheaters in the Great Plains plant. Tasks 1 through 3 of the contract are complete. Task 1 results were reported previously (AFWAL-TR-87-2042 Volume VI), and the results of Tasks 2 and 3 are reported here.

The objectives of Task 2 are to confirm process simulation results obtained in Task 1, to determine optimum process conditions for operation of the pilot plant in Task 3, and to produce small samples of jet fuels. However, after initial experiments failed to confirm the Task 1 plan, development of a viable process became the primary objective to Task 2. Further bench scale experiments provided a basis for an improved process, established pilot plant conditions for Task 3, and a preliminary design basis for Task 4, and produced small samples of JP-4, JP-8, and JP-8X aviation turbine fuels.

Pilot plant operation in Task 3 confirmed the viability of the Task 2 process by demonstrating extensive recycle of high boiling materials by hydrocracking. Two barrels of JP-8 jet fuel were produced for evaluation by the United States Air Force, and other contractors to the Department of Energy and the Air Force.

Hydrocracking experiments in Task 2 suggested the coal-derived liquids deactivate catalysts more rapidly than petroleum-derived stocks. This should be verified and the cause determined in future studies. The results of Tasks 1 through 3 will be applied to a preliminary process design and to a production run recommendation in Task 4 and 5, respectively.



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## FOREWORD

In September 1986, the Fuels Branch of the Aero Propulsion Laboratory at Wright-Patterson Air Force Base, Ohio, commenced an investigation of the potential for production of jet fuel from the liquid by-product streams produced by the gasification of lignite at the Great Plains Gasification Plant located in Beulah, North Dakota. Funding was provided to the department of Energy (DOE) Pittsburgh Energy Technology Center (PETC) to administer the experimental portion of this effort. This report details the effort of Amoco Oil Company, who is a contractor to DOE (DOE Contract Number DE-AC22-87PC90015), conducted a preliminary analysis of upgrading alternatives for the production of turbine fuels from the Great Plains liquid by-product streams. DOE/PETC was funded through Military Interdepartmental Purchase Request (MIPR)-FY1455-86-NO657. Mr. William E. Harrison III was the Air Force Program Manager, Mr. Gary Stiegel was the DOE/PETC Program Manager, and Mark Furlong was the Amoco Program Manager.

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## SECTION I

### INTRODUCTION

The Great Plains Coal Gasification Plant in Beulah, North Dakota, produces about 145 MM SCF/D of substitute natural gas (SNG) from lignite. The plant also produces three liquid by-products: about 2,900 B/D of tar oil, 830 B/D of crude phenols, and 650 B/D of naphtha. These liquids are all products from the devolatilization of lignite in the Lurgi gasifiers. Currently, the by-products are burned in the plant's boilers and superheaters to produce steam. The economic viability of the plant might be improved by producing marketable products, rather than steam, from these by-product liquids. To this end, Amoco and Lummus Crest, under a contract with the United States Department of Energy, are investigating the technical and economic feasibility of converting the by-product liquids to jet fuels and other saleable products. Jet fuels are of particular interest because of the close proximity of Great Plains to several U.S. Air Force bases, and the obvious strategic interest in maintaining a constant, source of jet fuel for those bases.

## SECTION II

### PROJECT OVERVIEW

As shown in Figure 1, this project is divided into five major tasks: Process Concept Definition, Bench Scale Testing, Pilot Plant Testing, Preliminary Process Design and Economics, and Production Run Recommendation. The results of Tasks 2 and 3 are reported here.

The first task, Process Concept Definition, includes three subtasks: Liquid By-Product Analysis, Process Modelling and Design, and Economic Modelling. The first of these subtasks (1.1), By-Product Analyses, involves analytical characterizations of samples of each by-product taken at six-week intervals. The results from this program, which provide an indication of the average quality of each stream and the variability of that quality over time, are an important input to the second subtask include limited experimental processing data on the Great Plains by-products by the Western Research Institute (WRI),<sup>(1)</sup> Amoco's petroleum refining process models and linear programming technology, Lummus' process simulation and design programs and a market analysis of by-products from Great Plains developed by Sinor Consultants.<sup>(2)</sup> In addition, throughout Task 1, ANG Coal Gasification Company provided valuable input and advice on all fronts. The major objective of subtask 1.2 is to produce seven conceptual designs and associated capital and operating costs for facilities to refine the Great Plains by-products. These include designs for maximizing productions of each grade of jet fuel (JP-4, JP-8, JP-8X), designs for profitable schemes which produce the various jet fuel grades, and a scheme for maximizing profits. In subtask 1.3 the results generated by Amoco and Lummus are subjected to economic analysis.

The two products from Tasks 1 were the design and economic results for each of the seven designs and a plan for bench scale testing (Task 2) to confirm any assumptions made in Task 1. The final report for Task 1 was issued by the U.S. Air Force in September 1988.<sup>(3)</sup> Based on the design and economic results from Task 1 and preliminary results from Task 2, the Department of Energy and the Department of Defense decided on a preferred processing scheme for the Great Plains liquids, the "profitable JP-8" case developed in Task 1. Amoco has carried out pilot plant testing (Task 3) of the process design from Tasks 1 and 2 and has provided barrel quantities of product for testing by the United States Air Force and associated contractors. The pilot plant results are being used by Amoco and Lummus to develop a preliminary process design (Task 4) for a plant to upgrade the liquid by-products at Great Plains. Finally, in Task 5, Lummus will suggest existing facilities where the processing scheme might be carried out on a scale sufficient to provide jet fuel for aircraft testing.

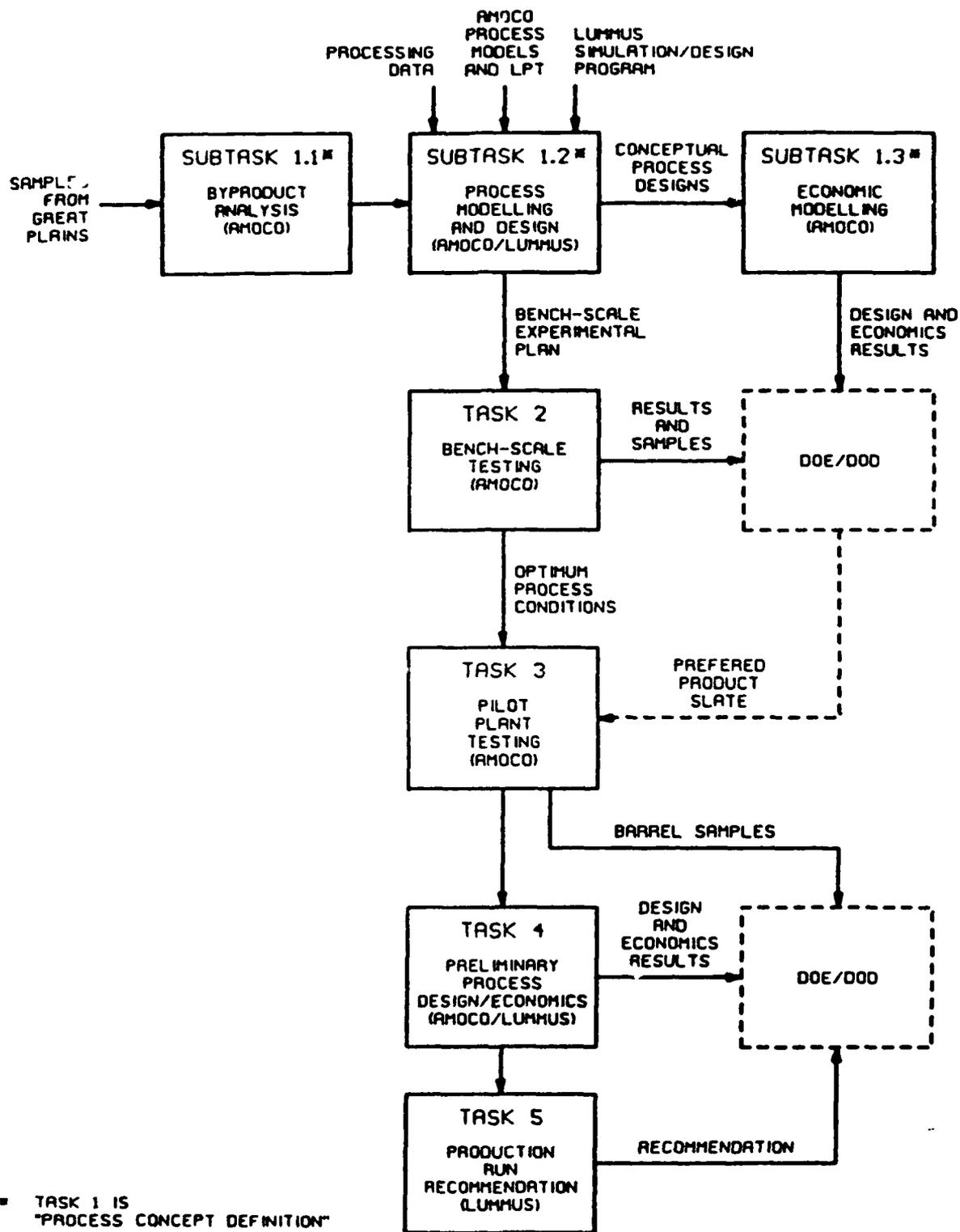


FIGURE 1  
 PRODUCTION OF JET FUEL  
 FROM COAL DERIVED LIQUIDS:  
 AMOCO/LUMMUS ACTIVITIES

### SECTION III

#### SOURCE OF BY-PRODUCT LIQUIDS

Tar oils, crude phenols, and naphtha are produced at the Great Plains Gasification Plant; a schematic of the plant is shown in Figure 2. The plant currently produces about 145 MMSCFD of synthetic natural gas (SNG) from North Dakota lignite. The SNG is composed almost entirely of methane, which is derived mostly from synthesis gas ( $H_2 + CO$ ) produced in the Lurgi Mark IV gasifiers and methanated in downstream reactors. The liquid by-products (tar oil, phenolics, and naphtha) are produced during lignite devolatilization in the gasifiers.

The tar oil and phenolics are condensed from the product gas along with water vapor to form a gas liquor. This condensation takes place in heat exchangers located in the gasifier quench, shift converter, gas cooling, and Rectisol units. The liquor is routed to the gas liquor separation unit, where the tar oil is recovered by gravity separation. The heaviest portion of the tar oil, which contains about 20 percent coal dust, is recycled to the gasifiers. The recycle rate of this "dusty tar" is about 1800 B/D. The remaining tar oil, which contains 2-6 percent dust, is produced at a rate of 2900 B/D. The phenolics are recovered from the gas liquor by extraction with isopropyl ether in the Phenolsolvan unit. The resulting crude phenol stream, which is produced at a rate of about 830 B/D is composed mostly of phenol, cresol, and xylenol, with the remainder being water and neutral oils. The naphtha is condensed from the gasifier raw gas by contacting the stream with cold methanol in the Rectisol unit. The naphtha is produced at a rate of 650 B/D.

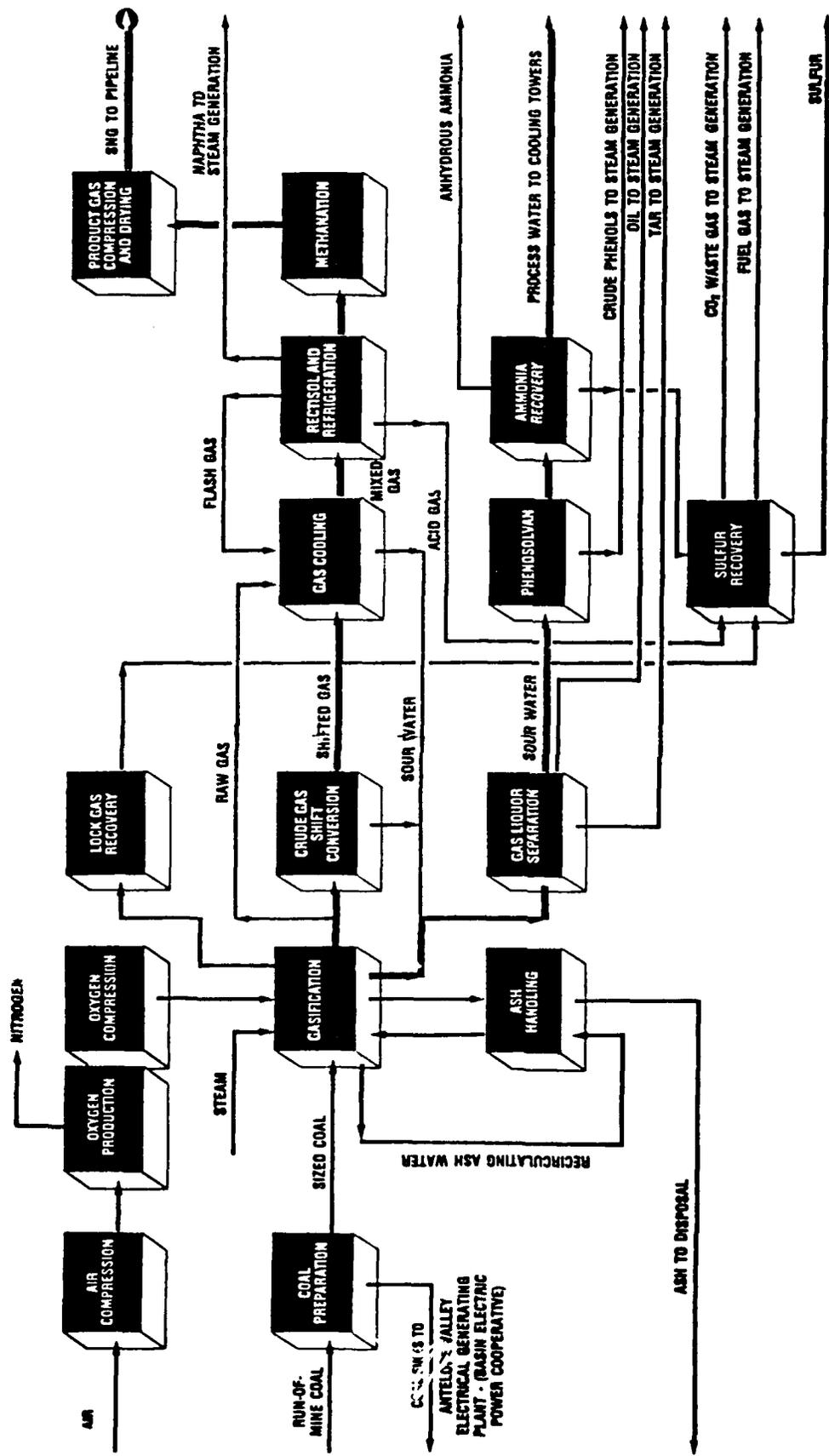


FIGURE 2. GREAT PLAINS GASIFICATION PROCESS BLOCK FLOW DIAGRAM

## SECTION IV

### TASK 2 RESULTS

As outlined in the Task 1 recommendations (3) and the project contract (4), the objectives of Task 2 are to confirm process simulation model results obtained in Task 1, to determine optimum process conditions for operation of the pilot plant in Task 3, and to produce small samples of jet fuels.

Experiments to confirm the Task 1 plan showed that the recommended process could not produce specification jet fuels. Accordingly, bench-scale experiments to develop a viable process became the primary objective of Task 2. Optimization, particularly data analysis confirming the reaction kinetics used in the Task 1 models, was not completed. However, raw data necessary for this analysis are presented here, and were used to specify process design conditions.

An overview of this process development work is presented below, followed by more detailed discussions of the experimental procedures and results which defined conditions for a working tar-oil conversion process. Analyses of the small samples of JP-4, JP-8, and JP-8X jet fuels produced during these runs are also discussed.

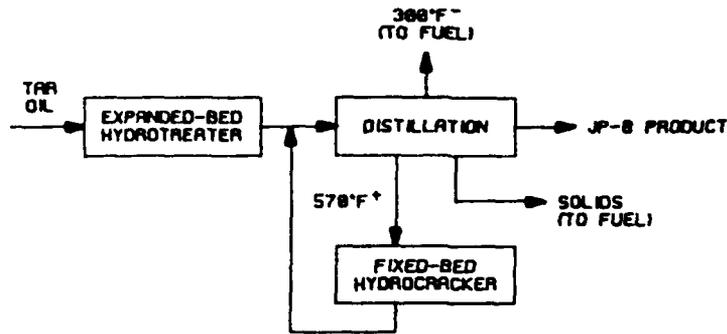
#### A. Overview of Process Development Work

As a part of Task 1, a process for the production of jet fuel from tar oil was developed in the "Profitable JP-8 Case". The Task 1 recommendation was to process the tar oil in an expanded-bed hydrotreater to lower aromatics content and nitrogen levels in the feed and then to hydrocrack the material boiling over 570°F to extinction in a fixed-bed hydrocracker (Figure 3). The expanded-bed hydrotreater was recommended to handle the high solids content in the tar oil and high releases expected during hydrotreating.

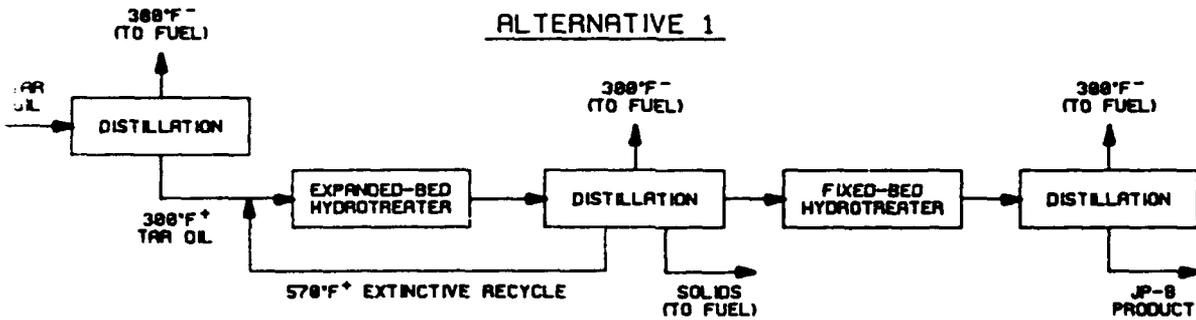
The initial goal of Task 2 was to verify this scheme recommended in Task 1 and to provide data on appropriate operating conditions and product yields and qualities. Selection of the expanded-bed hydrotreater for the first stage was verified when attempts to remove the solids by distillation resulted in losses of 25 percent of the tar oil to decomposition in the still pot. Since the solids could not be removed by distillation without significant losses and since fixed-bed reactors cannot handle solids without plugging, an expanded-bed reactor is required for the first stage of hydrotreating. However, the process is not feasible as recommended. The expanded-bed hydrotreater, although fine for handling solids and high heat releases, has significant backmixing and cannot lower the aromatics content of the feed sufficiently to meet jet fuel specifications. Consequently, alternative processes were developed.

Alternatives 1 through 3, shown in Figure 3, represent progressively more severe and more capital intensive approaches for the conversion of the tar oil to JP-8. All three cases contain a distillation pretreatment step to remove water and materials boiling below 300°F from the tar oil before

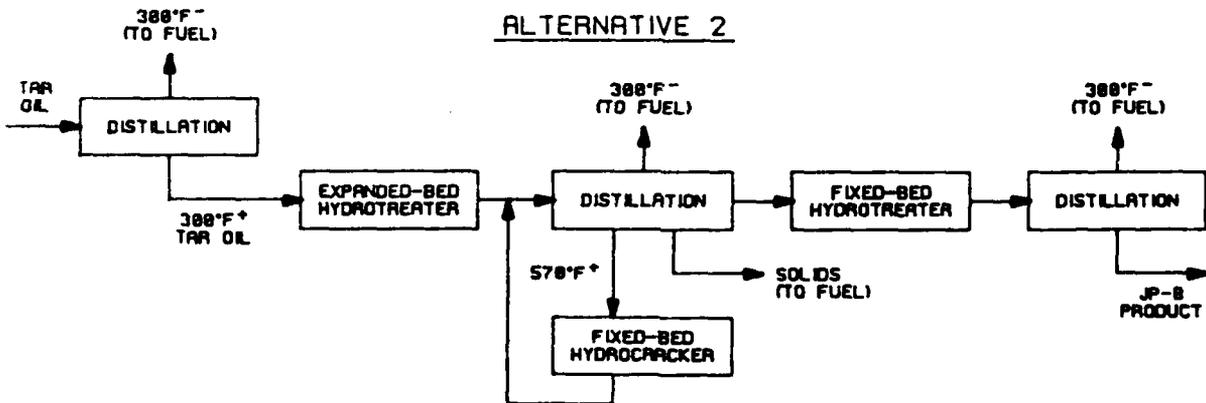
TASK 1 RECOMMENDATION



ALTERNATIVE 1



ALTERNATIVE 2



ALTERNATIVE 3

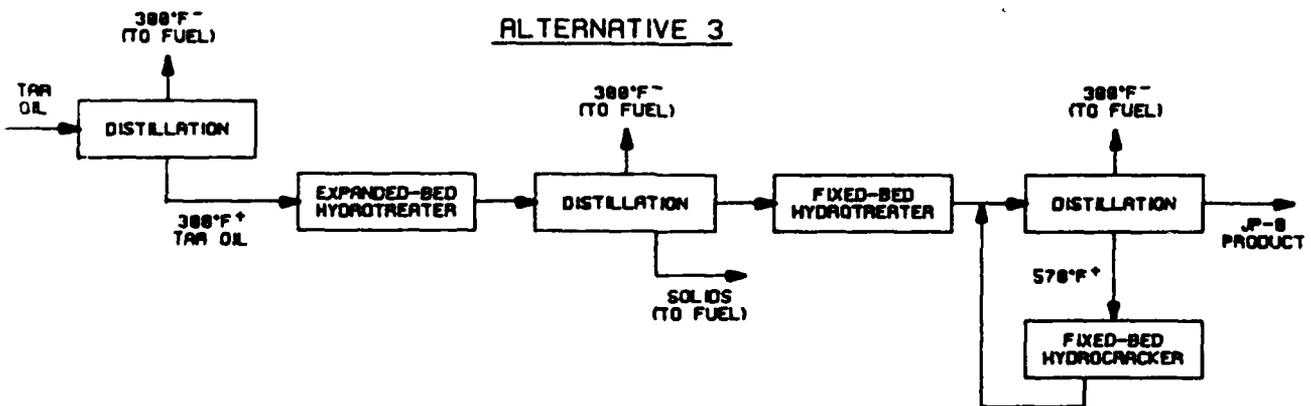


FIGURE 3. TAR OIL PROCESSING ALTERNATIVES

hydroprocessing. This reduces first-stage hydrotreater capacity. Removal of the lighter materials lowers the total hydrogen consumptions much as 4 percent. Furthermore, the light products from the 300°F minus feed would be unsuitable for jet fuel. Process optimization may recommend taking a heavier cut which could improve the economics.

Of the three alternatives, Alternative 1 is the simplest, requiring only two stages of hydrotreating and no hydrocracker. In Alternative 1, the first stage hydrotreater operates in a recycle mode. The material boiling over 570°F is recycled back to the expanded bed hydrotreater to lower its boiling range. After distillation to remove the light ends and solids, neither of which are suitable as jet fuel substituents, the remaining liquids are treated over a fixed-bed hydrotreater to further lower the aromatics and nitrogen contents to meet jet fuel specifications. Alternative 2 is similar to the Task 1 recommendation with the addition of a second hydrotreater after the hydrocracking stage. The second hydrotreater will lower the nitrogen and aromatics contents of the product to jet fuel specifications. Finally, Alternative 3 requires two stages of hydrotreating, followed by a hydrocracker. The first, expanded-bed hydrotreater lowers the nitrogen and aromatics contents and saturates olefins in the tar oil which permits solids removal by distillation without significant losses to retrograde reactions, which the raw tar oil demonstrated. After the first stage of hydrotreating, the solids and light ends are stripped from the hydrotreated product and sent to a second-stage, fixed-bed hydrotreater to further reduce the nitrogen level and aromatics content. Finally, the 570°F+ fraction is hydrocracked to extinction in a final, hydrocracker stage.

Alternative 1 was tested first, since it has the lowest capital cost and a hydrocracker is not required. Instead of lowering the boiling point to jet fuel range by hydrocracking, the 550°F+ material is recycled to extinction in the first-stage hydrotreater. Bench scale testing showed that there was virtually no conversion of the 550°F recycle material in the recycle mode. Although 35 percent of the fresh feed was converted in one pass at 0.6 weight-hourly space velocity (WHSV), the recycle material was not converted in the hydrotreater. Even at space velocities as low as 0.2 and 0.1 conversion of the recycle stream was undetectable. Alternative 1, then, was not effective.

Alternative 2 was tested next as it is the next most cost effective design. Alternative 2 requires a fixed-bed hydrocracker between the two hydrotreater stages used in Alternative 1. Because the heavy materials are hydrocracked first the fixed-bed hydrotreater can operate at relatively mild conditions and will be less expensive than a second hydrotreater installed in front of the hydrocracker (as in Alternative 3).

Alternative 2 failed because rapid catalyst deactivation occurred in the hydrocracker with hydrotreated 550°F+ tar oil containing as little as 200 ppm nitrogen. Some petroleum hydrocrackers are routinely operated at these nitrogen levels. This deactivation may be caused by nitrogen-containing materials peculiar to coal-derived liquids which poison the active sites on the catalyst. The nitrogen compounds in the coal-derived material are different when compared to petroleum-derived stocks of similar boiling range. A comparative examination of nitrogen compound types in the tar oil and petroleum feedstocks might explain deactivation and differences in

hydrocracking of the two feedstock types. For example, the addition of denitrogenation catalyst above the hydrocracking catalyst beds may make Alternative 2 viable.

Alternative 3 is the most severe of the alternatives and was successful at converting the tar oil to jet fuel. Since the fixed-bed hydrotreater processes the entire expanded-bed effluent stream the size of the fixed-bed hydrotreater is nearly doubled relative to Alternative 2. The second stage hydrotreater lowers the nitrogen level to 10 ppm before the hydrocracker, which gives stable hydrocracker operation and reasonable hydrocracker catalyst life. It may be possible to install hydrotreater beds above the hydrocracker beds in a single vessel, eliminating the separate fixed-bed hydrotreater. The advantage of Alternative 3 is that less hydrogen is consumed and better reactor control is possible with this design, than a system with denitrogenation beds added to the hydrocracker (modification to Alternative 2).

Compared with the scheme described in Task 1, Alternatives 2 and 3 would be considerably more expensive, as there are two more distillation and one more hydrotreater involved. However, the bench-scale and pilot-plant work verified the need for the more severe design of Alternative 3. Although it is more capital intensive, Alternative 3 has better operability, more flexibility, and the capability to process the tar oil to meet JP-8 specifications.

The experimentally-verified version of Alternative 3 uses nickel-molybdenum-phosphorous on alumina catalyst operating at 760°F and 2,000 psig for denitrogenation in the first-stage hydrotreater. The second-stage operates at the same conditions with nickel-tungsten-phosphorous on alumina catalyst to promote aromatics saturation while removing most of the remaining nitrogen. The hydrocracker uses a commercial nickel-tungsten catalyst on "y" sieve. More detailed catalyst compositions are given below.

Figure 4 and Table I show the experimentally-verified process based on Alternative 3. This is the recommended basis for the preliminary process design to be developed in Task 4 of this project and the basis for production of the larger samples of JP-8 for Task 3.

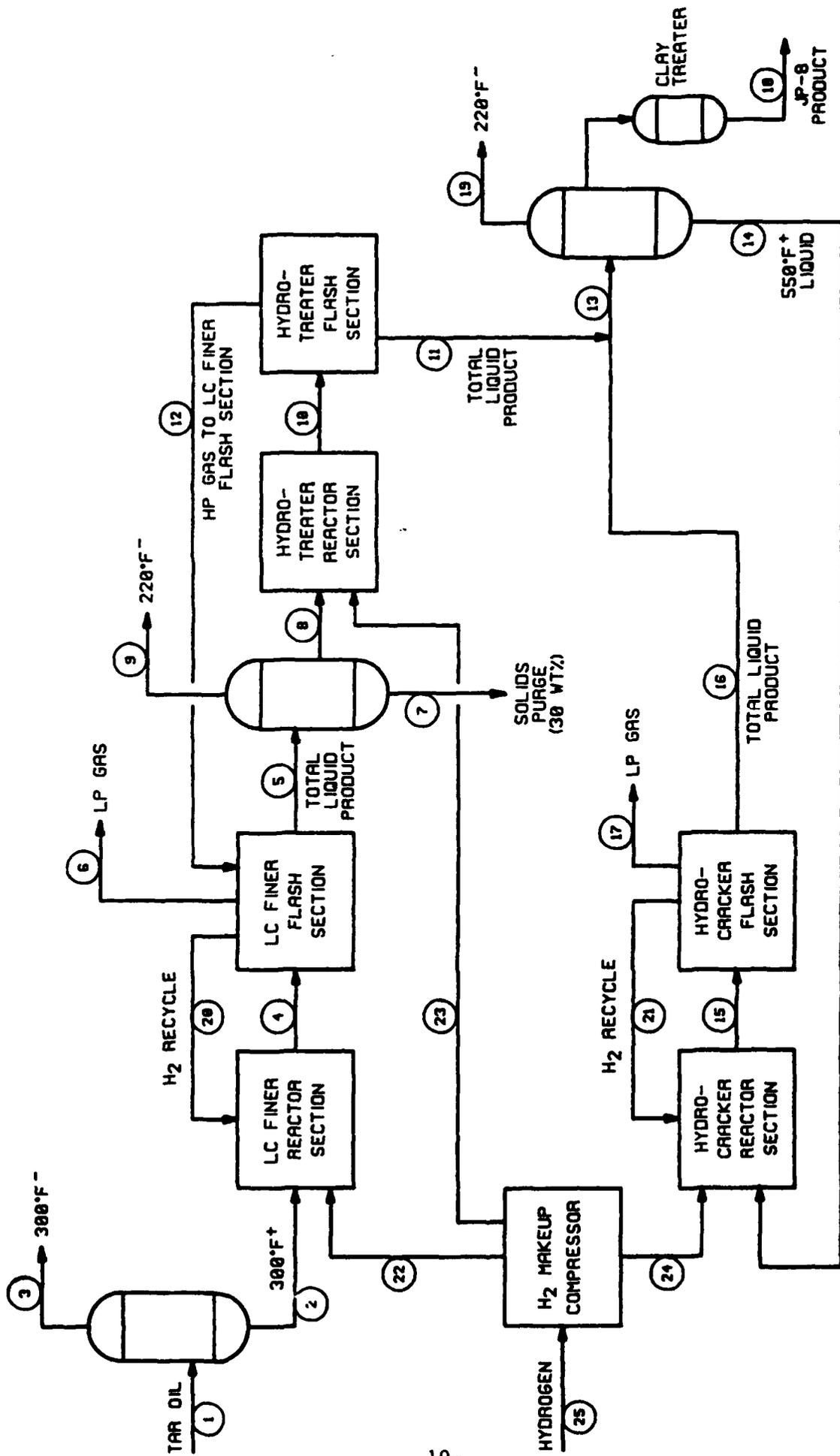


FIGURE 4

RECOMMENDED PROCESS DESIGN BASIS

TABLE I

MATERIAL BALANCE FOR RECOMMENDED PROCESS

Stream No.	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>	<u>9</u>
Stream Description	Tar Oil	300°F+ Tar Oil	300°F- Tar Oil	LC Finer Effluent	LC Finer Tot. Liq.	LP Gas Vent	Solids Purge	Hydro- Treater Feed	Light Ends Purge
Components, lbs/hr									
Carbon	40,077	38,763	1,314		37,508	1,751	1,094	35,339	1,075
Hydrogen	4,090	3,936	154		5,333	1,051	122	5,000	211
Nitrogen	385	333	52		8	333		8	
Sulfur	257	226	31		4	226		4	
Oxygen	2,861	2,505	356		18	2,505		18	
Solids	Incl.	Incl.			521		521		
Total	47,670	45,763	1,907		43,392	5,866	1,737	40,369	1,286
Flow Rate, gpm									
Temperature, °F									
Pressure, gpm									
Viscosity Liq., cs									
Sp. Gr. Liquid	1.015	1.028			0.881			0.879	
Gas Density, lb/ft <sup>3</sup>									
Vapor Pressure, psia									

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TABLE I (Cont.)

MATERIAL BALANCE FOR RECOMMENDED PROCESS

Stream No.	<u>10</u>	<u>11</u>	<u>12</u>	<u>13</u>	<u>14</u>	<u>15</u>	<u>16</u>	<u>17</u>	<u>18</u>
Stream Description	Hydro- treater Effluent	Hydro- treater Tot.Liq. Product	Hydro- treater Vent Gas	Fraction- ator Feed	Hydro- Cracker Feed	Hydro- Cracker Effluent	Hydro- Cracker Tot.Liq. Product	LP Gas Vent	JP-8 Product
Components, lbs/hr									
Carbon		34,843	496	51,140	17,397		16,297	1,100	31,976
Hydrogen		5,207	839	7,645	2,241		2,438	339	5,033
Nitrogen			8						
Sulfur			4						
Oxygen			18						
Solids									
Total		40,050	1,365	58,785	19,638		18,735	1,439	37,009
Flow Rate, gpm									
Temperature, °F									
Pressure, psig									
Viscosity Liq., cs									
Sp. Gr. Liquid									
Gas Density, lb/ft <sup>3</sup>									
Vapor Pressure, psia									

MWF/df/89625

5/10/89

TABLE I (Cont.)

MATERIAL BALANCE FOR RECOMMENDED PROCESS

Stream No.	<u>19</u>	<u>20</u>	<u>21</u>	<u>22</u>	<u>23</u>	<u>24</u>	<u>25</u>
Stream Description	Light Ends Purge	LC Finer H <sub>2</sub> Recycle	Hydro- cracker H <sub>2</sub> Recycle	H <sub>2</sub> To LC Finer	H <sub>2</sub> To Hydro- treater	H <sub>2</sub> To Hydro- cracker	Total H <sub>2</sub> Makeup
Components, lbs/hr							
Carbon	1,767						
Hydrogen	371			1,609	1,046	536	3,191
Nitrogen							
Sulfur							
Oxygen							
Solids							
Total	2,138			1,609	1,046	536	3,191
Flow Rate, gpm							
Temperature, °F							
Pressure, psig							
Viscosity, Liq., cs							
Sp. Gr. Liquid							
Gas Density, lb/ft <sup>3</sup>							
Vapor Pressure, psia							

WF/df/89625

1/10/89

## B. Distillation Pretreatment of Tar Oil Feed

Table 2 shows analyses of tar oil before and after distillation to remove water and low-boiling (<300°F) hydrocarbons. The 300°F cut point represented less than 5 percent of the as-received tar oil, but removed about two-thirds of the water and most of the low-boiling hydrocarbons. These low-boiling hydrocarbons included about 10 percent of the nitrogen and oxygen, 8 percent of the sulfur, and 3 percent of the "aromatics" (presumably volatile pyridinics, oxygenates, and mercaptans).

Distillation pretreatment decreases consumption of hydrogen that would otherwise be required to convert these low-boiling heteroatomic compounds to NH<sub>3</sub>, H<sub>2</sub>S, and light hydrocarbons. All of these consume more hydrogen than do aromatics saturation reactions. Pretreatment decreases overall hydrogen consumption by about 4 percent, reducing the cost of JP-8 product by about 2 percent.

## C. Hydrotreating Scoping Runs

As-received tar oil from the Great Plains plant was hydrotreated in a bench-scale autoclave reactor to determine heat release and to provide denitrogenation kinetic data. Table 3 shows selected properties of tar oil and petroleum heavy virgin gas oil feeds used in these experiments. Whole tar oil taken from Great Plains on November 16, 1987, was the feed for these experiments. Later runs on other units used tar oil taken on January 18, 1988, distilled to remove materials boiling below 300°F.

Whole tar oil was hydrotreated in an automated, high pressure isothermal basket autoclave reactor system, shown schematically in Figure 5. The reactor, Figure 6, is a nominal one-liter heavy wall vessel with a center-positioned magnetically-driven mixer designed to minimize solids accumulation on the catalyst. A cylindrical basket suspended between the vessel wall and mixer holds up to 150 cc of catalyst.

126 grams of Shell 424 catalyst, 1/16 inch extrudate with an average length of about 1/4 inch, was sulfided then conditioned for about 120 hours with a standard petroleum heavy vacuum gas oil (HVGO) before a base-case run to monitor catalyst activity. Table 4 shows the catalyst composition for all three of the reactor systems.

Table 5 shows the run conditions and corresponding product analyses, and Table 6 summarizes material balances for the runs, closed by mathematically adjusting feed input to provide carbon balance. For all runs, temperature and system pressure were held constant at 720°F and 1,800 psig while varying the apparent weight-hourly space velocity (WHSV) between 0.5 and 4. Baseline runs with HVGO feed before and after the tar oil runs showed that catalyst remained suitably active for these experiments so that no adjustments for catalyst activity decline were necessary.

The material balance results show that hydrogen uptake, C<sub>1</sub>-C<sub>4</sub> gas production, heteroatom gas make, denitrogenation, and desulfurization all increased as expected with decreased space velocity. However, the desulfurization results are somewhat erratic, suggesting that hydrogen sulfide may have recombined

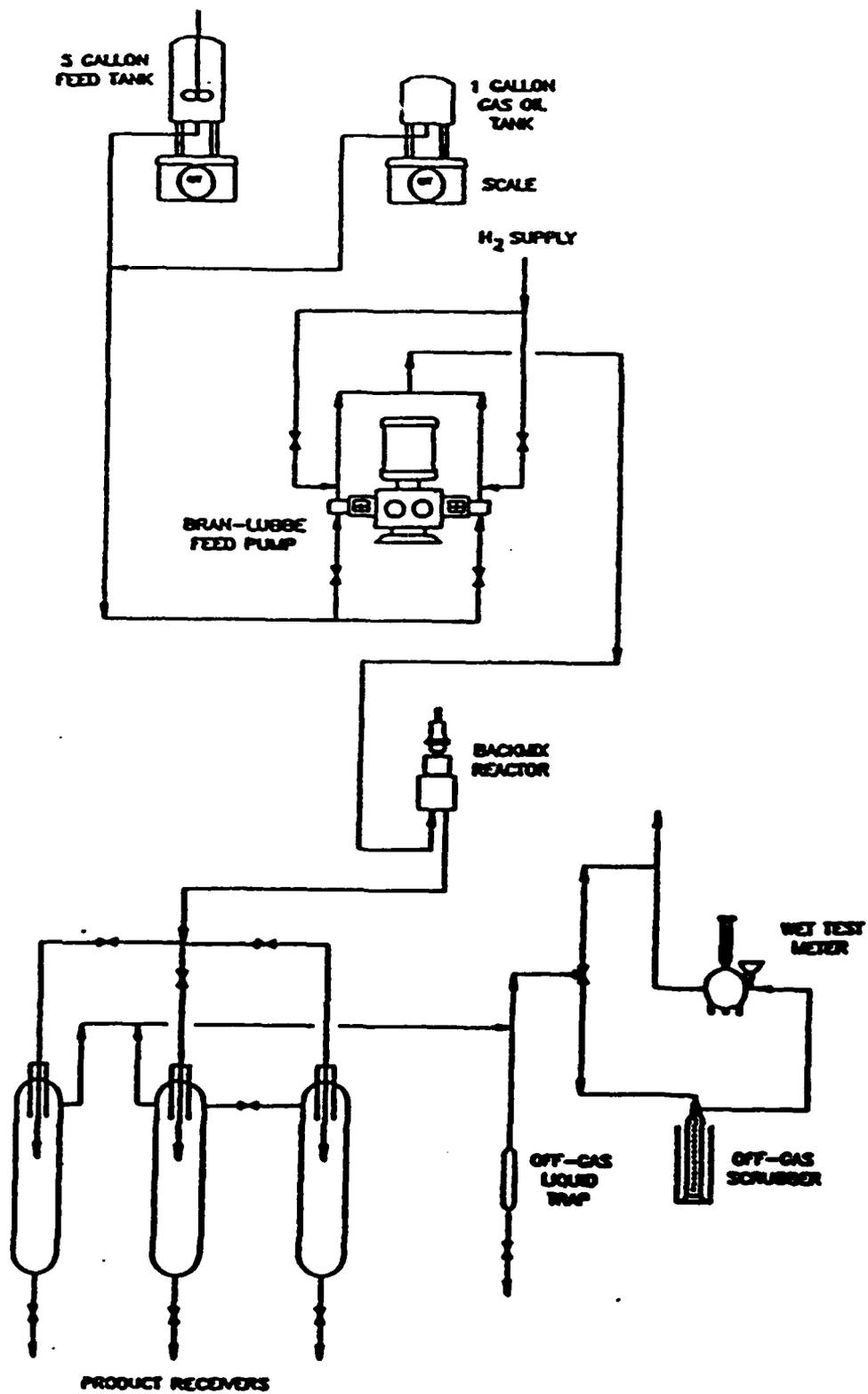


FIGURE 5  
HYDROTREATING PILOT PLANT--BASKET AUTOCLAVE

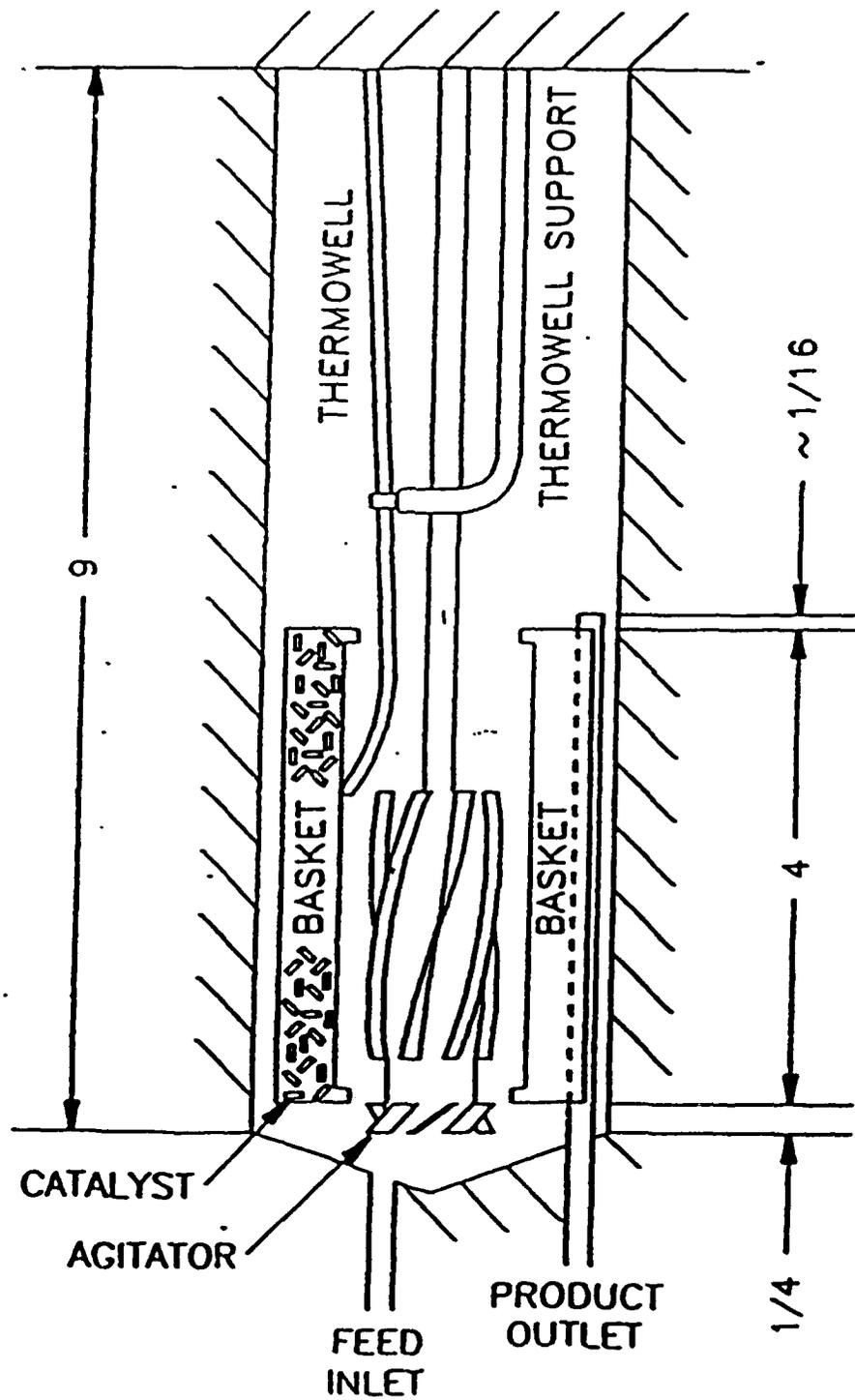


FIGURE 6

BASKET AUTOCLAVE REACTOR INTERNALS

TABLE 2  
 SELECTED PROPERTIES OF TAR OIL FEED  
 BEFORE AND AFTER DISTILLATION PRETREATMENT

Sample Description:	1/18/88 Whole Tar Oil	1/18/88 300F+ Tar Oil
Elemental Analysis, As Received:		
Water, wt.%	1.55	0.59
Elemental Analysis, Dry Basis:		
Carbon, wt.%	84.07	84.71
Hydrogen, wt.%	8.58	8.60
Nitrogen, ppm	8,065	7,283
Sulfur, ppm	5,383	4,929
Oxygen, ppm (diff.)	60,012	54,739
Atomic H/C Ratio	1.22	1.21
API Gravity	7.9	6.2
Specific Gravity	1.015	1.028
Mass Spectral Results, wt.%		
Saturates	36.3	37.9
Paraffins	8.3	10.3
NCond. Cycp'fins	6.5	7.6
Cond. 2-Ring "	0.0	0.0
Cond. 3-Ring "	21.6	20.0
Isoparaffins	0.0	0.0
Naphthenes	0.0	0.0
Aromatics	63.7	62.1
Mono Species	35.9	34.3
Di "	19.1	19.5
Tri "	3.1	2.5
Tetra "	1.0	0.8
Penta "	0.5	0.4
Thiopheno "	3.9	4.3
unidentified	0.3	0.3
Total CA, wt. %	27.1	27.4

G. C. Simulated Distillation, ASTM Method D-2887

Weight % Distilled	Temperature, F	
IBP	167	279
5	281	336
10	338	371
20	390	414
30	440	468
40	486	498
50	544	556
60	586	603
70	674	683
80	760	765
90	881	877
95	961	948
FBP	1,018	1,018

TABLE 3

SELECTED PROPERTIES OF TAR OIL AND  
AMOCO HEAVY VACUUM GAS OIL

Sample Description:	Tar Oil 11/16/87	HVGO
Water, wt.%	1.41	0.00
Elemental Analysis, Dry Basis:		
Carbon, wt.%	84.19	85.28
Hydrogen, wt.%	8.56	11.79
Nitrogen, ppm	8,297	1,338
Sulfur, ppm	4,730	28,200
Oxygen, ppm (diff.)	59,462	-238
Atomic H/C Ratio	1.21	1.65
API Gravity	7.8	19.8
Specific Gravity	1.016	0.935
Chlorides, ppm	1.0	--
Ash Oxide, wt. %	0.03	--
Filtered Solids, wt.%	0.16	--
PSD (Microtrac), %		
<4 microns	7.4	--
4.4-6.6	6.8	--
6.6-9.4	9.0	--
9.4-13	10.4	--
13-19	13.4	--
19-27	11.0	--
>27 microns	41.5	--

TABLE 4  
TYPICAL CATALYST PROPERTIES

	<u>First Hydrotreater</u>	<u>Second Hydrotreater</u>	<u>Hydrocracker</u>
Supplier No.	Shell 424	Shell 354	Davison SMR 6-1881
Shape	Tri-lobed	Tri-lobed	Cylindrical
Nominal Size, in.	1/16	1/8	1/8
Composition, wt.%			
Nickel	3.0	3.0	1.6
Tungsten	0.0	25.0	14.2
Molybdenum	13.0	0.0	0.0
Phosphorus	--	2.5	0.0
Physical Properties			
Surface Area, m <sup>2</sup> /gm	162	135	325
Pore Volume, ml/gm	0.47	0.39	0.42
Support	Alumina	Alumina	"y" Sieve

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TABLE 5  
HYDROTREATING SCOPING EXPERIMENTS--  
RUN CONDITIONS AND PRODUCT ANALYSES

Run No.	1	2	3	4	5	6	7
Feed	HVGO	Tar Oil	HVGO				
Nominal MHSV	2.2	4.6	1.9	2.3	1.0	0.5	2.2
Temperature, F	720	720	720	720	720	720	720
Pressure, psig	1,800	1,800	1,800	1,800	1,800	1,800	1,800
Water, wt. %	0.01	0.41	0.11	0.15	0.04	0.00	0.07
Elemental Analysis, Dry Basis:							
Carbon, wt. %	86.84	86.95	87.23	87.15	87.55	87.76	86.80
Hydrogen, wt. %	12.73	10.67	11.23	11.15	11.62	11.81	12.63
Nitrogen, ppm	300	4,749	3,254	3,205	1,721	980	550
Sulfur, ppm	3,839	889	2,359	800	958	665	6,364
Oxygen, ppm (diff)	153	18,215	9,827	12,987	5,660	2,655	--
Atomic H/C Ratio	1.75	1.46	1.53	1.52	1.58	1.60	1.73
API Gravity	25.9	23.0	26.2	25.4	28.8	29.8	25.3
Specific Gravity	0.8990	0.9190	0.8974	0.9016	0.8827	0.8775	0.9024
Mass Spectral Results, wt. %							
Saturates	32.8	44.9	49.7	48.4	52.6	54.9	--
Paraffins	32.8	12.8	12.7	12.2	13.0	12.9	--
NCond. Cycp'fins	--	20.5	25.3	24.4	26.8	28.3	--
Cond. 2-Ring "	--	3.6	5.4	5.5	7.5	6.7	--
Cond. 3-Ring "	--	8.0	6.2	6.3	5.3	5.0	--
Isoparaffins	--	0.0	0.0	0.0	0.0	0.0	--
Naphthenes	--	0.0	0.0	0.0	0.0	0.0	--
Aromatics	7.0	55.1	50.3	51.6	47.4	45.1	--
Mono Species	--	38.9	38.6	38.9	37.3	36.2	--
Di "	--	12.5	9.4	10.0	7.3	6.7	--
Tri "	--	2.2	1.9	1.8	1.5	1.5	--
Tetra "	--	0.4	0.2	0.2	0.5	0.2	--
Penta "	--	0.2	0.2	0.2	0.1	0.1	--
Thiopheno "	--	0.6	0.1	0.4	0.5	0.2	--
Unidentified	--	0.3	0.0	0.2	0.1	0.1	--
Total CA, wt. %	--	21.5	18.8	19.9	20.7	19.7	--

JDF  
11/17/88

TABLE 6

HYDROTREATING SCOPING EXPERIMENTS--  
MATERIAL BALANCE SUMMARIES AND HEAT RELEASE

Run No.	1	2	3	4	5	6	7
Feed	HVGO	Tar Oil	HVGO				
Nominal WHSV Temperature, F	2.2 720	4.6 720	1.9 720	2.3 720	1.0 720	0.5 720	2.2 720
Yield Summary, wt. %							
Hydrogen	-0.91	-2.40	-3.05	-2.87	-3.48	-3.69	-0.80
Heteroatom Gas	2.72	5.41	6.40	5.64	7.28	7.70	2.43
C1 - C4 Gas	0.29	0.16	0.18	0.16	0.22	0.23	0.28
C5+ Liquid	97.94	102.97	103.37	132.53	97.73	103.59	98.15
Total	100.04	106.14	106.89	135.46	101.75	107.83	100.06
Hydrogen, SCFB	562	1,608	2,044	1,925	2,334	2,472	493
Heteroatom gas, SCFB	104	393	474	412	536	566	92
C1-C4 Gas, SCFB	9	6	6	6	8	9	9
Conversion, wt. %							
Denitrogenation	76.0	41.1	59.6	48.9	79.8	87.8	59.7
Desulfurization	86.7	80.7	48.6	77.6	80.2	85.4	77.9
Heat of Reaction, BTU/Bbl*							
Denitrogenation	2,915	10,338	14,978	12,286	20,057	22,075	2,228
Desulfurization	19,870	3,368	2,028	3,240	3,349	3,568	17,851
Deoxygenation	0	48,037	56,188	49,857	63,624	66,902	0
Saturation	20,066	61,685	82,454	84,272	92,721	97,757	17,515
Total	42,851	123,429	157,648	149,655	179,752	190,302	37,594

\* 85 BTU/SCF of H2 consumed for saturation, 70 for other reactions.

with product in the receivers. (All samples were washed with zinc chloride solution before analysis to remove dissolved  $H_2S$  and  $NH_3$ .)

Heat release was calculated for each run based on hydrogen consumption using 70 BTU/SCF for removal of nitrogen, oxygen, and sulfur, and 85 BTU/SCF for aromatics saturation reactions. For tar oil, the latter value is probably low because it accounts only for aromatics saturation; heats of reaction for saturation of olefins, about 140 BTU/SCF, were not included. Table 5 shows that substantially more heat is released for tar oil hydrogenation than for Amoco's standard HVGO feed.

Hydrogen consumption ranged from 1,600 to 2,500 SCFB, substantially more than 1,400 SCFB for petroleum resid hydroprocessing where expanded-bed reactors facilitate temperature control. Expanded-bed reactors will be necessary for temperature control in commercial tar oil hydrotreating. Because this reactor type can tolerate solids, pretreatment of feed for solids removal is avoided. However, removal of water and light ends upstream of the hydrotreater is recommended, as discussed in Section IV-B.

#### D. Hydrotreating Experiments

Figure 7 shows the automated, high pressure, isothermal bench-scale unit used to study conditions for first-stage hydrotreating. The reactor, a heavy-wall tube of type 304 stainless steel, 0.625 inch I.D. by 30.5 inches long, fitted with a center-mounted 1/8-inch thermowell, is enclosed by a 1-inch pipe wrapped with electrical heaters which serves as a four-zone furnace. A Ruska pump controls the oil rate and a Brooks mass flow meter the hydrogen rate. Piping was arranged for upflow of oil and hydrogen providing rough simulation of an expanded-bed reactor. Two high-pressure receivers collect product at reactor pressure. Reactor off gas exits the receivers via a pressure control valve, scrubber, and wet test meter.

102 grams of Shell 424 catalyst, 1/16 inch extrudate with an average length of about 1/4 inch, was sulfided then conditioned for about 735 hours with tar oil before a base-case run (Run 3) to monitor catalyst activity decline. Table 4 shows the catalyst composition.

Figure 8 shows the partial factorial experimental design used to study removal of aromatics, nitrogen, and the other heteroatoms from 300°F+ tar oil. Reaction temperatures ranged from 670 to 760°F, pressure from 1,800 to 2,000 psig, and weight-hourly space velocities from 0.6 to 1.2. Table 7 lists conditions and product analyses for each run, and Table 8 summarizes the material balances, closed by adjusting feed input to provide carbon balance.

In the process recommended in Task 1, the high boiling fraction of hydrotreated product is hydrocracked and blended with the lighter hydrotreater fraction to yield specification jet fuel product (containing less than 25 percent aromatics). Table 7 shows that about 65 percent of the lowest aromatics content hydrotreater product (Run 8) boils below 550°F, in the JP-8 fuel range. However, Table 9 shows that this fraction contains about 39 percent aromatics. Even if the higher boiling fraction were hydrocracked to aromatics-free material, the final JP-8 product would still contain over 25 percent aromatics after blending. Light ends removal, necessary to meet

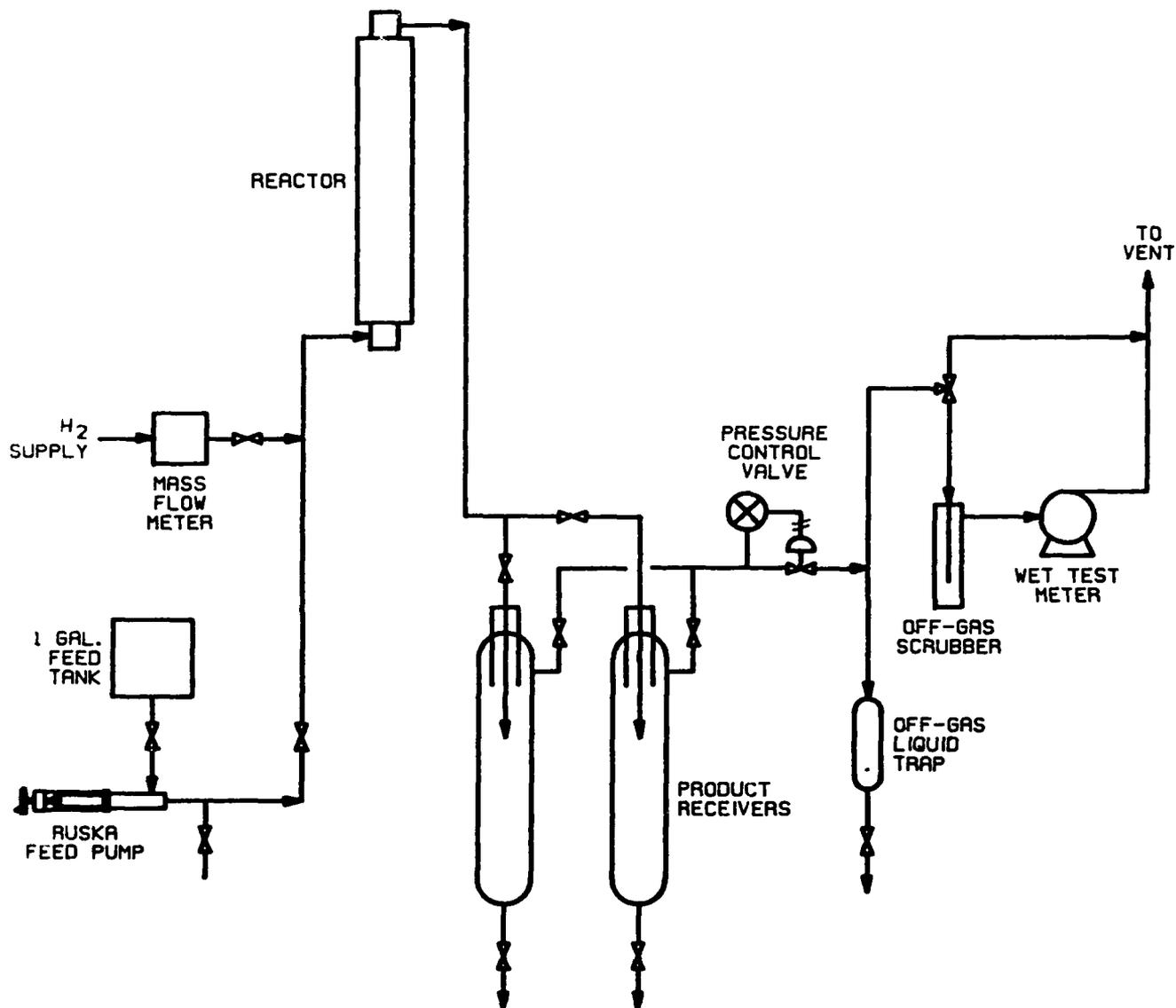


FIGURE 7

HYDROTREATING PILOT PLANT--FIRST HYDROTREATER



TABLE 7

FIRST HYDROTREAT--RUIN CONDITIONS AND PRODUCT ANALYSES

Run No.	1	2	3	4	5	6	7	8	9	10	11	12
Moist wt. %	0.6	0.6	0.6	1.2	1.2	0.6	0.6	0.6	1.2	2.6	0.6	1.2
Temperature, °F	700	646	680	700	700	730	700	730	730	760	760	760
Pressure, psig	1,800	1,800	2,000	2,000	2,000	1,800	1,800	1,800	1,800	1,800	2,000	2,000
Water, wt. %	0.06	0.13	0.07	0.18	0.09	0.03	0.05	0.02	0.06	0.05	0.05	0.04
Elemental Analysis, Dry Basis:												
Carbon, wt. %	87.11	87.11	87.34	87.26	86.93	88.18	87.88	87.64	87.96	88.13	88.06	88.06
Hydrogen, wt. %	11.27	11.27	11.66	11.10	11.28	12.06	11.94	12.16	11.50	11.92	12.13	11.92
Nitrogen, ppm	1,901	2,653	1,951	3,166	2,382	542	2,211	210	1,161	180	180	490
Sulfur, ppm	693	219	61	152	100	39	192	53	148	102	25	85
Oxygen, ppm	2,367	5,844	4,078	7,100	6,300	1,833	4,456	1,122	3,567	-364	-364	1,544
Atomic H/C Ratio	1.54	1.54	1.59	1.52	1.55	1.63	1.56	1.65	1.56	1.61	1.64	1.61
API Gravity	26.1	24.1	26.0	22.3	24.3	28.5	25.9	30.0	25.1	30.0	30.7	28.7
Specific Gravity	0.8978	0.9092	0.8986	0.9200	0.9081	0.8845	0.8987	0.8760	0.9033	0.8762	0.8726	0.8834
Mass Spectra Results, wt. %												
Saturates	53.4	50.9	54.2	49.5	51.1	56.8	51.7	58.2	48.9	55.4	56.7	53.0
Paraffins	9.2	12.7	8.2	12.5	11.4	9.5	9.7	11.1	11.2	12.3	12.3	10.7
NCond. Cyc'ring	30.6	23.9	31.9	22.5	25.6	31.9	29.6	31.8	24.0	18.2	30.3	29.9
Cond. 2-Ring	8.3	6.9	8.4	6.4	7.4	10.8	7.2	11.6	8.2	10.2	11.0	8.8
Cond. 3-Ring	5.3	7.4	5.7	8.1	6.8	4.6	5.2	3.8	5.5	2.5	3.0	3.6
Isoparaffins	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ashthenes	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Aromatics	46.6	49.1	45.8	50.5	48.9	43.2	48.3	41.8	51.1	44.6	43.3	47.0
Monospecifics	36.9	38.5	37.5	36.3	38.7	37.1	36.8	36.0	40.8	36.4	36.1	38.7
Dic'ring	7.7	7.7	5.6	8.0	7.5	4.2	8.6	4.0	7.9	6.1	7.3	6.3
Tri'ring	1.7	1.9	1.5	2.0	1.8	1.1	1.9	1.2	1.7	1.6	1.4	1.4
Tetra	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.0	0.2	0.1	0.0
Pen'ta	0.2	0.1	0.1	0.2	0.2	0.1	0.2	0.0	0.1	0.1	0.1	0.0
Triopheno	0.4	0.5	0.7	0.8	0.6	0.6	0.3	0.5	0.4	0.1	0.3	0.4
Unidentified	0.3	0.3	0.4	0.5	0.2	0.1	0.4	0.1	0.2	0.1	0.1	0.1
Total CA, wt. %	19.1	19.9	18.0	18.4	17.7	15.0	17.1	16.6	19.1	18.1	18.3	17.3
G.C. Simulated Distillat. on, Method D-2887												
Weight % Distilled								Temperature, F				
0	155	177	155	177	136	117	129	129	174	115	115	113
1	174	209	174	209	173	154	156	156	177	151	154	144
5	215	253	215	253	214	212	213	213	241	212	212	194
10	268	308	268	308	267	282	281	281	281	212	212	260
20	346	376	346	376	346	346	332	332	354	296	296	307
30	406	427	406	427	406	376	398	364	409	354	354	368
40	471	480	471	480	470	465	466	466	475	406	407	430
50	508	513	508	513	501	488	489	483	477	475	475	483
60	549	576	549	576	548	543	544	540	505	507	506	531
70	655	662	647	662	647	605	643	605	569	579	574	594
80	743	747	739	747	739	688	736	688	729	647	647	693
90	880	883	889	883	889	840	885	840	885	801	801	830
95	913	961	913	961	918	930	971	931	976	864	864	920
99	--	--	--	--	--	--	--	--	--	--	1,008	--
100	--	--	--	--	--	--	--	--	--	--	1,018	--

TABLE 8

FIRST HYDROTREATER--MATERIAL BALANCE SUMMARIES

Run No.	1	2	3	4	5	6	7	8	9	10	11	12
Nominal MSV	0.6	0.6	0.6	1.2	1.2	0.6	0.6	0.6	1.2	0.6	0.6	1.2
Temperature, F	700	666	680	680	700	700	700	730	730	760	760	760
Pressure, psig	1,800	1,800	2,000	2,000	2,000	1,800	1,800	1,800	1,800	1,800	2,000	2,000
Yield Summary, wt. %												
Hydrogen	-3.50	-3.11	-3.53	-2.91	-3.06	-3.95	-3.34	-4.09	-3.38	-3.82	-4.03	-3.84
Water vapor	7.25	5.71	6.40	5.67	5.48	7.50	6.69	7.34	6.95	7.54	7.55	7.50
C1-C4 Gas	0.41	0.66	0.64	0.67	0.28	0.60	0.21	0.76	0.83	0.72	0.69	0.89
C5+ Liquid	95.82	96.66	96.43	96.49	97.23	95.37	96.20	95.98	95.57	95.48	95.58	95.41
Total	99.98	99.93	99.94	99.92	99.90	99.72	99.96	99.98	99.96	99.92	99.79	99.96
Hydrogen, SCFB	2,378	2,109	2,396	1,978	2,073	2,683	2,267	2,778	2,293	2,591	2,732	2,605
H <sub>2</sub> atom gas, SCFB	537	418	470	415	400	555	508	542	513	558	558	555
C1-C4 Gas, SCFB	27	41	40	41	15	45	14	43	47	42	41	47
Conversion, wt. %	73.0	63.2	72.5	53.2	66.4	92.4	68.8	97.0	83.7	97.5	97.4	93.1
Denitrogenation	85.4	93.5	96.7	96.9	97.9	99.2	96.0	98.9	96.9	97.9	99.5	98.2
Desulfurization												
Carbon Balance, %	93.3	96.7	96.8	98.7	95.2	96.8	96.8	95.8	91.5	94.6	93.2	95.7

TABLE 9

FIRST HYDROTREATER--RUN 8:  
GC/MS ANALYSES OF IBP TO 550°F FRACTION

## Saturates, wt. percent:

Paraffins	20.4
N'Cond. Cycp'fins	17.9
Cond. 2-Ring	14.1
Cond. 3-Ring	9.0
Isoparaffins	0.0
Naphthenes	0.0
Total Saturates	61.4

## Aromatics, wt. percent:

Mono Species	27.8
Di "	5.5
Tri "	2.6
Tetra "	0.3
Penta "	0.1
Thiopheno "	2.3
Unidentified	0.0
Total Aromatics	38.6

flash point specifications, will further increase aromatics content beyond the 25 percent maximum allowed in JP-8 fuel.

Therefore, a second hydrotreater is necessary to saturate aromatics to the required level. Addition of a second hydrotreater allows operation of the first at more severe conditions to improve denitrogenation performance. Accordingly, first-stage hydrotreater conditions were recommended based on the best denitrogenation run (Run 12). In Task 3 of this project, operation of the large expanded-bed pilot plant at these conditions made product of the predicted quality.

#### E. Second Stage Hydrotreating Experiments

In these experiments, effluent from the first hydrotreater was distilled then hydrotreated again in a bench-scale fixed-bed unit to study effects of reaction conditions on aromatics saturation and denitrogenation.

Figure 9 shows the reactor, a heavy-wall tube of type 304 stainless steel, 0.815 inch I.D. by 32 inches long, fitted with a center-mounted 3/16 inch thermowell which is wrapped with electrical heaters to provide five heating zones. A Bran-Lubbe pump controls the oil rate and a Brooks mass flow meter the hydrogen rate. Piping was arranged for downflow of oil and hydrogen, with reactor effluent collected in three high-pressure product receivers. Gas vented from the receivers exits via a scrubber and wet test meter.

Conditions studied ranged from 650 to 760°F reactor temperature at 1,500 and 2,000 psig and weight-hourly space velocities of 0.5 and 1.0. Table 10 shows the feed composition, Table 11 lists conditions and product analyses, and Table 12 summarizes the material balances, closed by adjusting feed input to provide carbon balance.

As discussed in Section IV-A, the purpose of the second hydrotreater is to decrease nitrogen to less than 10 ppm in hydrocracker feed, and to provide the degree of aromatics saturation necessary. Table 11 shows that 2,000 psig and 700°F satisfied denitrogenation requirements and maximized aromatics saturation. However, 2,000 psig at 760°F was recommended for the design basis because this further lowered nitrogen in the product, enhancing hydrocracker catalyst life and performance. This recognizes that additional aromatics saturation will take place in the hydrocracker to meet jet fuel specifications.

A run at the recommended conditions yielded about ten gallons of product similar to the product from Run 9 described in Table 11. Distillation yielded about 35 weight percent of 550°F+ material for the hydrocracking study discussed in Section IV-F.

#### F. Hydrocracking Experiments

Two units were used in these runs, the second hydrotreater described in Section IV-E, and a smaller unit with the reactor cooled by a molten salt bath. Table 4 shows composition of the hydrocracking catalyst used in both units. Stabilized reactor operation could not be achieved during the experiments in the first unit, however, using the high-nitrogen feed. The second unit was fed only with low-nitrogen (less than 10 ppm) material and performed well.

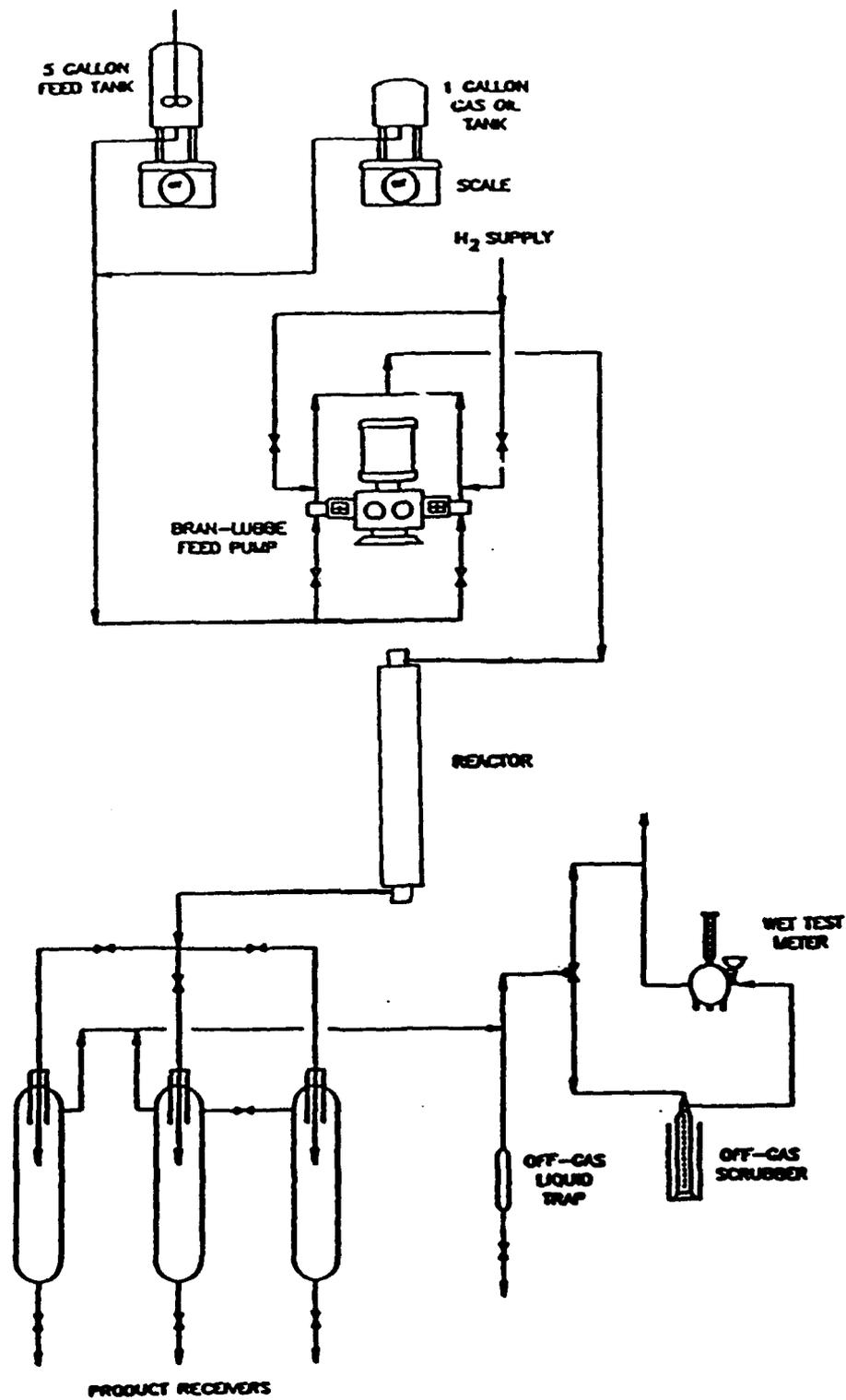


FIGURE 9  
HYDROTREATING PILOT PLAN--SECOND HYDROTREATER

TABLE 10

## SECOND HYDROTREATER--FEEDSTOCK ANALYSES

## Elemental Analysis, Dry Basis:

Carbon, wt.%	87.79
Hydrogen, wt.%	12.49
Nitrogen, ppm	120
Sulfur, ppm	270
Oxygen, ppm	1,000
Atomic H/C Ratio	1.70
API Gravity	29.6
Specific Gravity	0.879

## Mass Spectral Results, wt.%

Saturates	65.3
Paraffins	9.6
NCond. Cycp'fins	29.1
Cond. 2-Ring "	18.5
Cond. 3-Ring "	8.2
Isoparaffins	0.0
Naphthenes	0.0
Aromatics	34.7
Mono Species	29.6
Di "	3.6
Tri "	0.8
Tetra "	0.1
Penta "	0.0
Thiopheno "	0.6
Unidentified	0.0
Total CA, wt.%	12.9

## G.C. Simulated Distillation, Method D-2887

Wt. % Distilled	Temperature, F
0	172
1	179
5	241
10	279
20	350
30	395
40	439
50	479
60	521
70	574
80	632
90	729
95	792
99	901
100	939

RELATIONSHIP BETWEEN OPERATING CONDITIONS AND RESULTS

Run No.	1	2	3	4	5	6	7	8	9
WHSV	0.5	0.5	1.0	1.0	1.0	1.0	0.5	0.5	1.0
Temperature, F	700	760	760	760	700	650	650	650	760
Pressure, psig	2,000	2,000	2,000	1,500	1,500	1,500	1,500	2,000	2,000

Elemental Analysis, Dry Basis:

Carbon, wt. %	84.17	86.95	87.02	87.42	87.22	87.60	87.69	87.16	87.65
Hydrogen, wt. %	13.06	13.41	13.06	12.64	12.72	12.42	12.66	12.90	12.77
Nitrogen, ppm	1	2	1	4	6	29	18	16	4
Sulfur, ppm	16	11	16	27	25	21	15	9	12
Oxygen, ppm	200	200	200	200	300	300	200	200	200
Atomic H/C Ratio	1.85	1.84	1.79	1.72	1.74	1.69	1.72	1.76	1.74
API Gravity	33.7	33.1	33.2	31.5	31.2	29.7	30.6	31.7	31.6
Specific Gravity	0.857	0.849	0.859	0.868	0.870	0.878	0.873	0.867	0.867

Mass Spectral Results, wt. %

Saturates	90.5	89.8	81.1	70.2	72.4	63.1	68.5	77.9	73.6
Paraffins	10.8	13.4	9.4	9.4	9.1	8.9	7.5	7.3	9.4
NCond. Cycp'fins	35.3	35.6	35.8	32.0	32.3	31.0	33.2	35.5	34.8
Cond. 2-Ring "	31.2	30.1	27.3	22.2	23.2	17.6	21.4	26.3	22.6
Cond. 3-Ring "	13.2	10.8	8.7	6.5	7.8	5.6	6.4	8.8	6.9
Isoparaffins	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Napthenes	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Aromatics	9.5	10.2	18.9	29.8	27.6	36.9	31.5	22.1	26.4
Mono Species	7.4	8.5	16.6	24.9	24.8	33.3	28.3	19.3	23.2
Di "	0.6	0.6	1.4	3.4	1.6	1.1	1.7	1.2	2.0
Tri "	1.1	0.8	0.7	0.9	0.7	0.5	0.7	0.7	0.9
Tetra "	0.2	0.1	0.1	0.3	0.1	0.0	0.0	0.0	0.1
Penta "	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Thiopheno "	0.2	0.1	0.0	0.3	0.3	0.6	0.8	1.0	0.2
Unidentified	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total CA, wt. %	4.37	4.90	6.57	11.19	9.40	12.66	10.64	7.62	9.27

G.C. Simulated Distillation, Method D-2887

Weight % Distilled	Temperature, F
0	100
1	142
5	211
10	258
20	329
30	384
40	446
50	486
60	530
70	584
80	677
90	761
95	893
99	1,007
100	1,018

TABLE 12

## SECOND HYDROTREATER--MATERIAL BALANCE SUMMARIES

Run No.	1	2	3	4	5	6	7	8	9
WHSV	0.5	0.5	1.0	1.0	1.0	1.0	0.5	0.5	1.0
Temperature, F	700	760	760	760	700	650	650	650	760
Pressure, psig	2,000	2,000	2,000	1,500	1,500	1,500	1,500	2,000	2,000
Yield Summary, wt. %									
Hydrogen	-1.27	-1.21	-0.84	-0.36	-0.46	-0.08	-0.32	-0.67	-0.48
Heteroatom Gas	0.13	0.13	0.13	0.13	0.12	0.12	0.13	0.13	0.13
C1 - C <sub>4</sub> Gas	1.30	1.63	1.45	1.43	1.27	0.66	1.13	1.41	1.55
C5+ Liquid	103.08	99.49	99.59	99.15	99.51	99.67	99.12	99.47	98.78
Total	103.25	100.04	100.33	100.34	100.45	100.37	100.06	100.34	99.99
Hydrogen, SCFB	734	702	486	209	264	44	188	386	276
H <sub>2</sub> atom gas, SCFB	8	8	8	8	7	7	8	8	8
C1-C <sub>4</sub> Gas, SCFB	56	72	66	64	56	26	48	60	74
Conversion, wt. %									
Denitrogenation	99.1	98.3	99.2	96.7	95.0	76.1	85.2	86.8	96.7
Desulfurization	93.9	96.0	94.1	90.1	90.8	92.3	94.5	96.7	95.6
Carbon Balance, %	90.9	78.9	90.2	99.6	99.5	99.6	95.4	99.6	97.7

Rapid catalyst deactivation occurred in the first unit when feeding once-hydrotreated 550°F+ tar oil containing 250 to 300 ppm nitrogen. Presumably, nitrogen-containing materials peculiar to coal-derived liquids had poisoned active sites in the catalyst, requiring increasingly higher reactor temperatures to effect conversion.

Organic nitrogen compounds are thought to act as permanent catalyst poisons in petroleum hydrocracking, but the ammonia produced by reaction of organic nitrogen does not affect the catalyst permanently (5). For coal-derived liquids, nitrogen conversion to ammonia may occur more slowly than for petroleum-derived stocks, thus accounting for a greater degree of catalyst deactivation. More study is necessary to verify and determine the cause of this apparent difference in hydrocracking performance of petroleum and coal-derived liquids.

Several short scoping runs were completed in the second unit. With twice-hydrotreated feed containing about 5 ppm nitrogen, about 70 percent of the 550°F+ material was converted to lower boiling 550°F minus material. This provided the recommended design basis for hydrocracker conditions: 650°F, 1,600 psig, and 0.7 space velocity. Good catalyst performance in these runs was attributed to low nitrogen content of the feed.

The recommended hydrocracker conditions were further confirmed in the Task 3 work described later. In particular, the effects of hydrocracker recycle on product quality are discussed.

#### G. Production of Jet Fuel Samples

Because of the difficulties met in developing viable hydrocracker conditions, there was not sufficient good quality hydrocracked material available to blend into the small samples in Task 2. However, hydrocracked material was included in the large sample of JP-8 produced in Task 3 of this project.

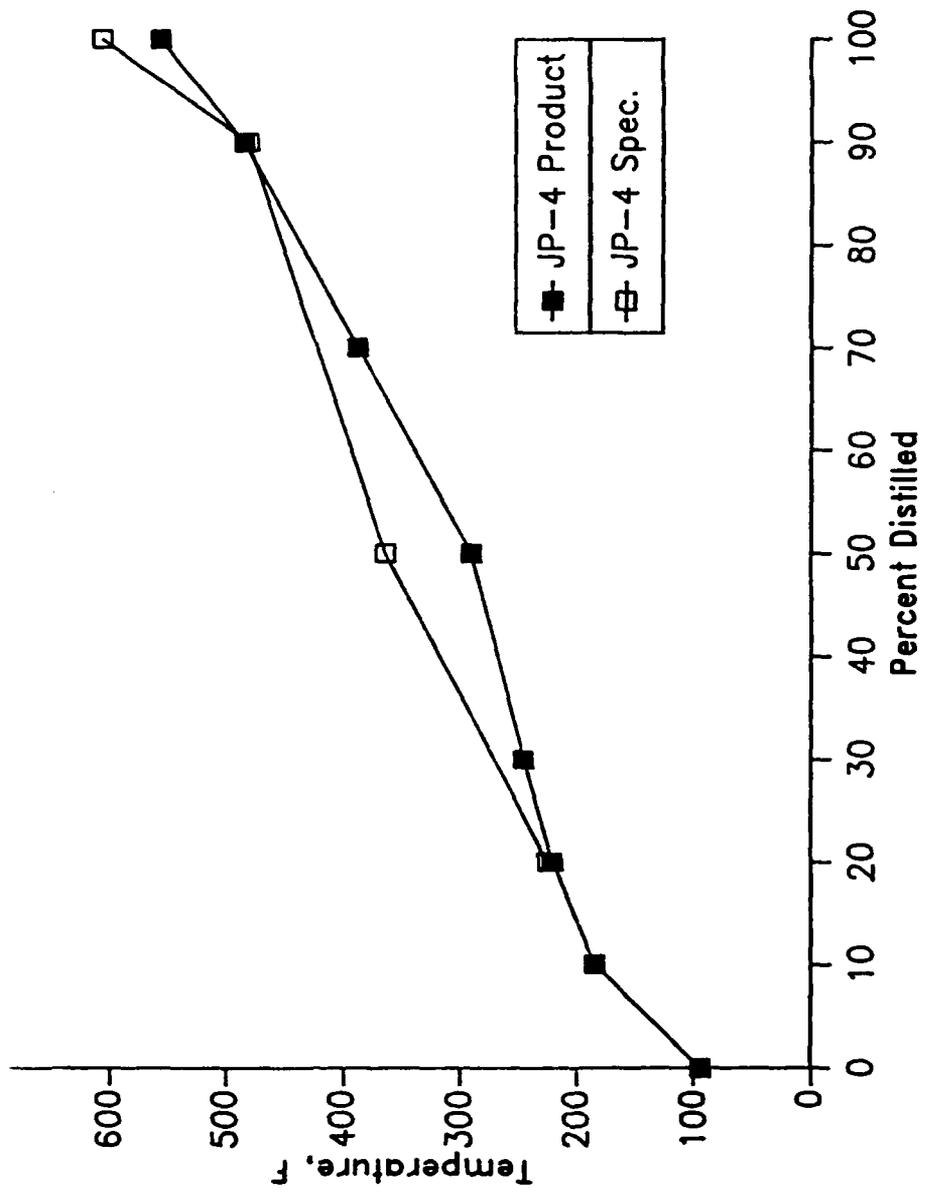
Table 13 shows how small samples of JP-4, JP-8, and JP-8X were prepared by blending distillation cuts of hydrotreated tar oil. Distillation results shown in Figures 10 through 12 confirm that the blended fuels meet boiling range specifications. Tables 14 and 15 compare the remaining inspection results against specifications for the respective fuels. Note that several analyses which require large aliquots of fuel could not be run because of limited sample quantity.

The JP-4 product met all specifications for which analyses were performed except for copper strip corrosion and heating value. The JP-8 and JP-8X products failed copper strip corrosion, existent gum, and flash point specifications. However, as specified by the client (USAF), none of these samples was clay-treated or treated with specified antioxidant additives, which may explain failure to pass the copper strip corrosion and gum content tests. Inclusion of too much material boiling in the IBP to 300°F range may explain the slightly low JP-4 heating value and the failure of JP-8 to meet the flash point specification.

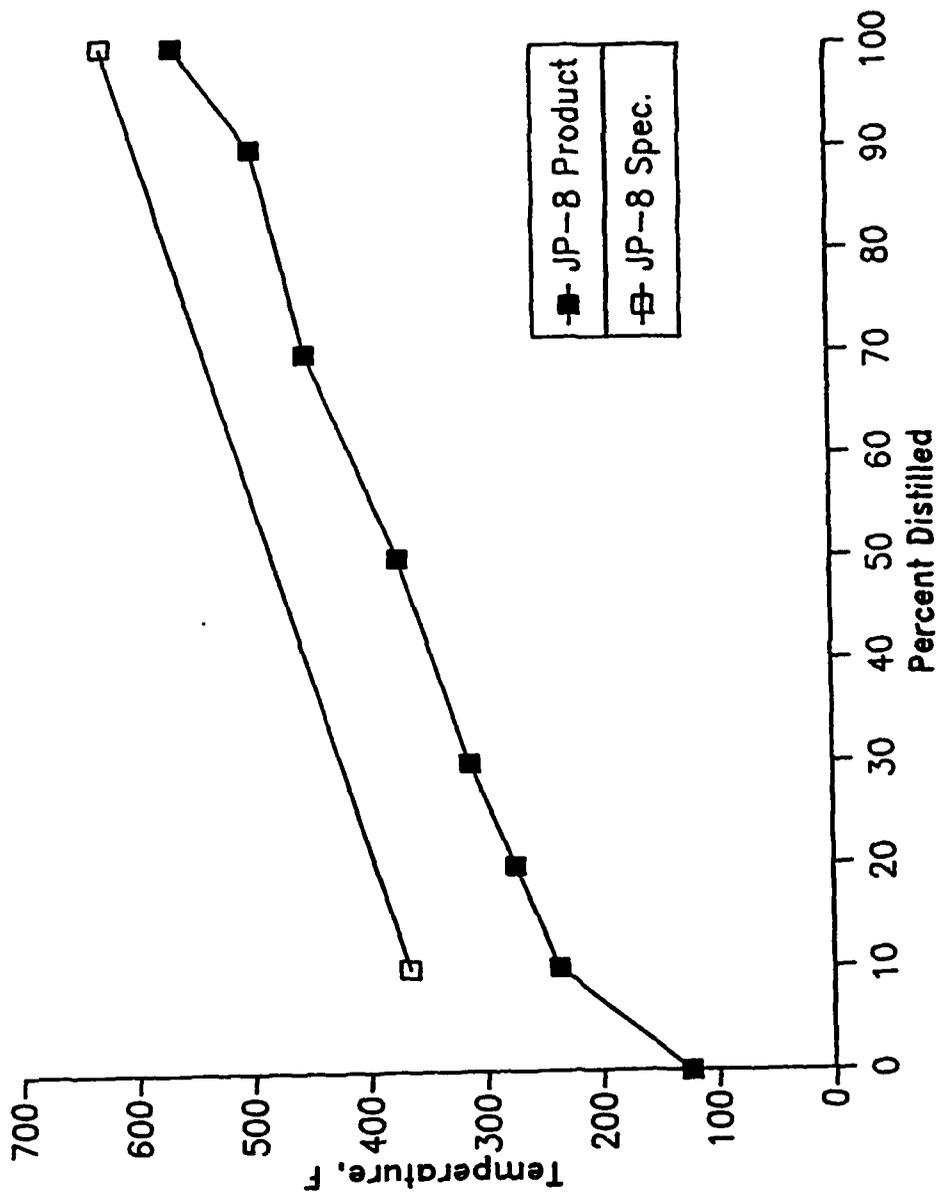
TABLE 13

## Blending for Jet Fuel Products

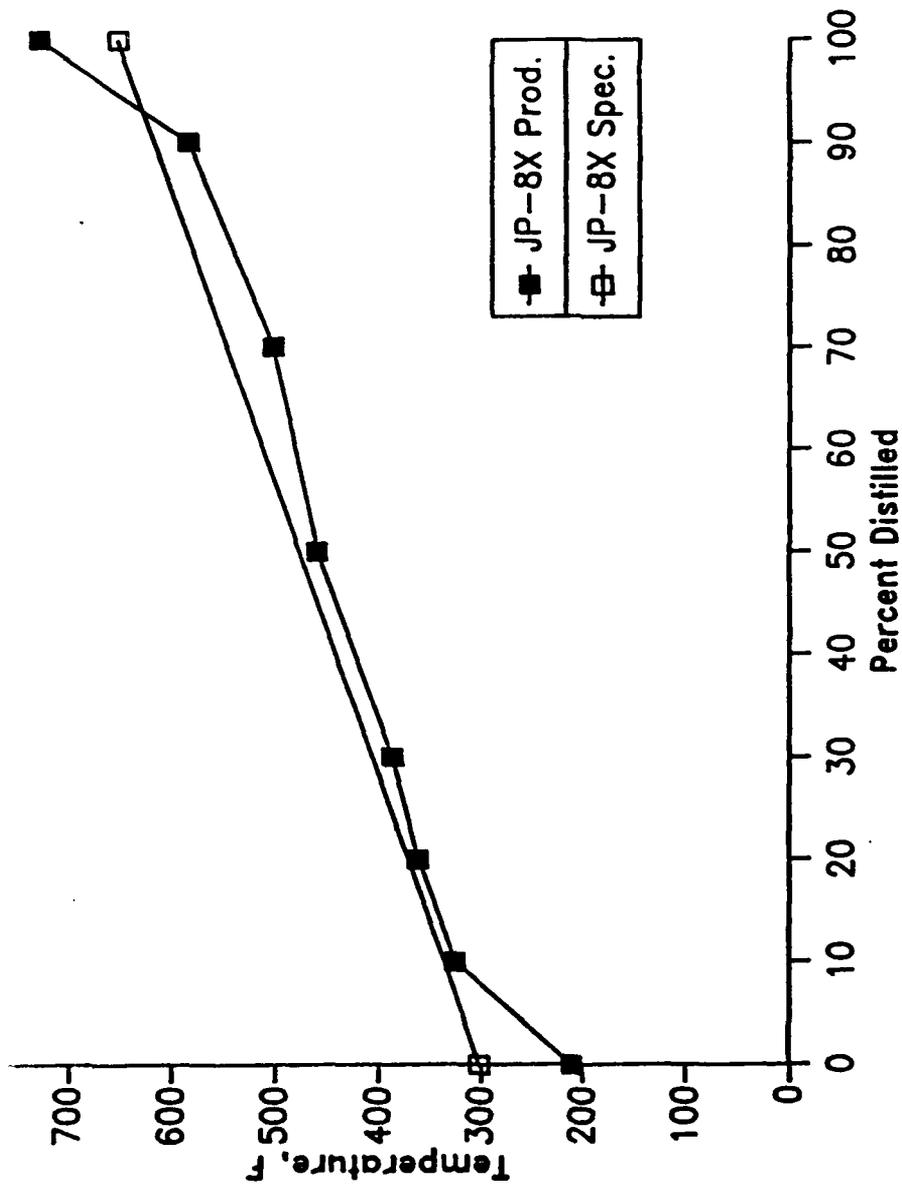
Cut No. -----	Boiling Range, F -----	Weight, gms. -----		
		JP-4 -----	JP-8 -----	JP-8X -----
1	IBP-300	3,886	2,862	--
2	300-375	26	5,421	1,797
3	375-400	--	--	5,323
4	400-475	1,304	6,006	2,932
5	475-520	--	--	1,113
6	520-575	--	--	3,123
Total		5,213	14,289	14,288
Est. Volume, gal.		1.7	4.6	4.4



**FIGURE 10  
BOILING RANGE OF JP-4 PRODUCT COMPARED TO SPECIFICATION**



**FIGURE 11**  
**BOILING RANGE OF JP-8 PRODUCT COMPARED TO SPECIFICATION**



**FIGURE 12**  
**BOILING RANGE OF JP-8X PRODUCT COMPARED TO SPECIFICATION**

TABLE 14

INSPECTIONS OF JP-4 PRODUCT

<u>Requirements</u>	<u>JP-4 Specification</u>	<u>JP-4 Inspection</u>
Color, Saybolt	Report	
Total acid number, mg KOH/g. max.	0.015	0.00
Aromatics, vol %, max.	25.0	10.0
Olefins, vol % max.	5.0	2.0
Mercaptan sulfur, wt.% max.	0.002	0.0001
Sulfur, total wt.% max.	0.40	0.002
Distillation temperatures, C		
Initial boiling point	Report	87.2
10 percent recovered, max.	Report	105
20 percent recovered, max.	145	111
50 percent recovered, max.	190	128
90 percent recovered, max.	245	221
End Point, max.	270	253
Residue, vol %, max	1.5	
Loss, vol %, max	1.5	1.5
Flash Point, C	No spec	
Density, kg/l, min. at 15 C	0.751	
Density, kg/l, max. at 15 C	0.802	
Vapor Pressure, 37.8 C, kPa	14 to 21	15.9
Freezing Point, C, max.	-58	-79
Viscosity, at -20 C, c'stokes max	No spec	0.9 @ 38 C
Heating value, Aniline--gravity product, min,	5,250	4,965
Hydrogen content, wt.%, min. or Smoke Point, mm, min.	13.6 20.0	30
Copper strip corrosion, 2 hr at 100 C, max.	1b	2.3
Thermal stability: Change in pressure drop, mm of Hg., max. Preheater deposit code, less than	25 3	
Existent gum, mg/100 ml, max	7.0	1.8
Particulate matter, mg/liter, max	1.0	
Filtration time, minutes, max	10	
Water Separometer Index, modified, min.	85	79
Fuel System icing inhibitor, vol.%	0.10-0.15	
Electrical conductivity, CU	200-600	
Cetane Index	No spec	
Peroxide number, wt. ppm, max.	No spec	

TABLE 15

INSPECTION OF JP-8 AND JP-8X PRODUCTS

<u>Requirements</u>	<u>JP-8 Specification</u>	<u>JP-8 Inspection</u>	<u>JP-8X Inspection</u>
Color, Saybolt	Report	30	
Total Acid Number, mg KOH/gm, max.	0.015	0.00	0.00
Aromatics, Vol%, max.	25.0	16.5	19.0
Olefins, Vol%, max.	5.0	3.0	4.0
Sulfur, Mercaptan, Wt%, max.	0.001	0.001	0.0001
Sulfur, Total, Wt%, max.	0.4	0.002	0.002
Distillation Temperature, °C			
Initial Boiling Point	Report	105	184°C
10% Recovered, max.	186	137	187
20% Recovered, max.	Report	151	193
50% Recovered, max.	Report	193	211
90% Recovered, max.	Report	256	268
End Point, max.	330	268	291°C
Residue, Vol%, max.	1.5		
Loss, Vol%, max.	1.5	2.3	2.5
Flash Point, °C, max.	38 (100)	26	35
Gravity, °API or	37-51	37.3	32.7
Density, kg/m <sup>3</sup> at 15°C	775-840	838	862
Freezing Point, °C, max.	-50	-56	-45
Viscosity at -20°C, C'stokes, max.	8.0 (8.0)		1.8@100°F
Net Heat of Combustion, MJ/kg min.	42.8		
Combustion:			
(1) Hydrogen Content, wt% min.,	13.6		
or (2) Smoke Point, mm min.	25		
(3) Smoke point, mm min. and	20		
Napthalenes, Vol% max.	3.0	0.36	0.56
Copper Strip Corrosion,	NDT 1b	2.3	
2 Hr @ 100°C (212°F)			
Thermal Stability:			
Change in Pressure Drop, mm Hg max.	25	0	0
Heater Tube Deposit Visual Rating	Less than Code 3	-1	-1
Existent Gum, mg/100 ml, max.	7.0	13.4	15.0
Particulate Matter, mg/Liter, max.	1.0		
Water Separation Index, Modified min.	70	75	70
Fuel System Icing Inhibitor, Vol%	0.1-0.15		

## SECTION V

### TASK 3 RESULTS

As outlined in Section II, the objectives of Task 3 are to carry out pilot plant testing to confirm the process design, and provide barrel quantities of product for testing by the Air Force. The technical approach consists of two stages of hydrotreating followed by distillation and hydrocracking of the higher boiling fractions to yield JP-8 jet fuel as outlined in the process flow diagram, Figure 4.

#### A. First-Stage Hydrotreating

As discussed in Section IV-C, expanded-bed reactors will be necessary for temperature control in the hydrotreaters for commercial tar oil processing. Because Amoco's expanded-bed pilot plant was unavailable within the required project schedule, Amoco requested that Lummus Crest Incorporated (LCI) perform the hydrotreating work. LCI maintains an expanded-bed pilot plant of appropriate size for this operation as part of their LC-Fining (tm) process studies.

LCI completed the hydrotreating operations at conditions specified by Amoco, based on the bench scale experiments discussed in Section IV-D of this report. Appendix A presents LCI's report on the hydrotreating operations, including a brief description of the unit.

Amoco distilled the hydrotreated product (excluding that collected during an upset condition at LCI) to remove water and other materials boiling below 300°F. Table 16 shows analyses of this 300°F+ hydrotreated tar oil, used as feed to the second hydrotreater.

#### B. Second-Stage Hydrotreating

Second-stage hydrotreating was completed in Amoco's pilot plant, shown schematically in Figure 13. The unit features two thirteen-foot long reactors with multiple heating zones, four hydrogen quench points, and travelling thermocouples in each reactor. Bran-Lubbe pumps control the oil feed rate and Brooks mass flow meters the hydrogen rates. The piping was arranged for downflow of oil and hydrogen through the first reactor only, bypassing the second reactor, with the effluent passing on level control to a product receiver via a high pressure gas-liquid separator. Gas vented from the separator and receiver exits through a scrubber and wet test meter.

The reactor was controlled at the conditions recommended in Section IV-E, 760°F, 2,000 psig, and 1.0 WHSV, except that throughput was increased to about 1.5 WHSV for most of the run to expedite sample production. Table 4 shows the catalyst composition, the same as for the bench-scale experiments. Table 17, a typical control analysis, shows that product quality is similar to bench-scale runs at similar conditions, specifically, Runs 3 and 9 in Table 11. Elemental analyses and material balances were not routinely performed during this production run.

The product was distilled to yield light ends, a 160 to 550°F heartcut, and 550°F+ material for hydrocracker feed. The respective yields were 0.2, 71.4, and 28.4 weight percent. Table 18 shows compositions of the heartcut and hydrocracker feed fractions.

TABLE 16

## SECOND HYDROTREATER--TYPICAL FEED PROPERTIES

## Elemental Analysis, Dry Basis

Carbon, wt%	87.79
Hydrogen, wt%	12.49
Nitrogen ppm	120
Sulfur, ppm	270
Oxygen, ppm	1000

Atomic H/C Ratio	1.70
------------------	------

API Gravity	29.6
Specific Gravity	0.879

## Mass Spectral Results, wt%

Saturates	65.3
Paraffins	9.6
NCond. Cycp' fins	29.1
Cond. 2-Ring fins	18.5
Cond. 3-Ring fins	8.2

Aromatics	34.7
Mono Species	29.6
Di Species	3.6
Tri Species	0.8
Tetra Species	0.1
Penta Species	0.0
Thiopheno Species	0.6

Unidentified	0.0
Total CA, wt%	12.9

## G. C. Simulated Distillation, Method D-2887

<u>Wt% Distilled</u>	<u>Temperature, °F</u>
0	172
1	179
5	241
10	279
20	350
30	395
40	439
50	479
60	521
70	574
80	632
90	729
95	792
99	901
100	939

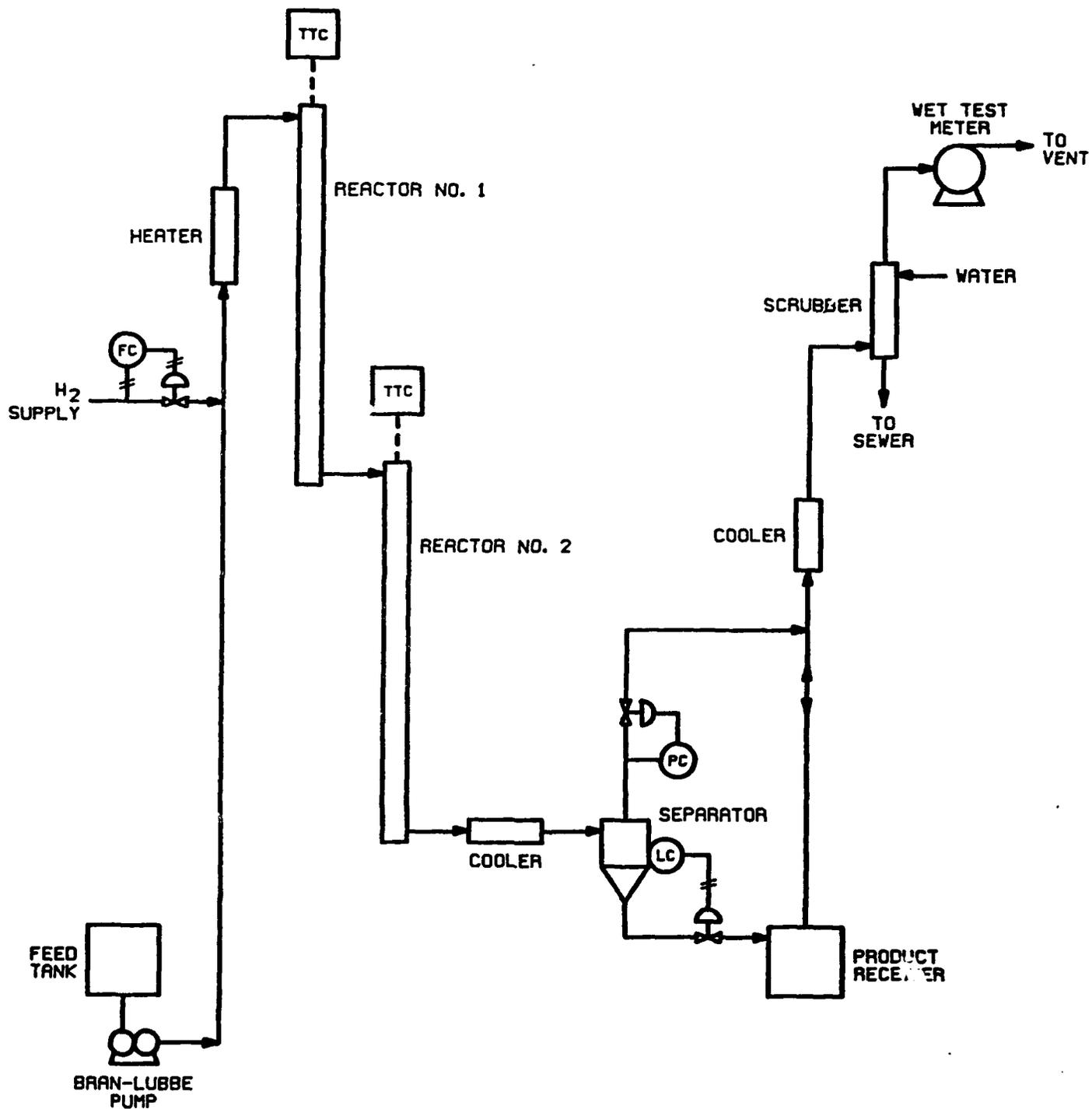


FIGURE 13  
 HYDROTREATING/HYDROCRACKING PILOT PLANT

TABLE 17

SECOND HYDROTREATER--TYPICAL PRODUCT ANALYSESMass Spectral Results, Wt%

Saturates	72.6
Paraffins	9.0
NCond. Cycloparaffins	33.4
Cond. 2-Ring Cycloparaffins	22.9
Cond. 3-Ring Cycloparaffins	7.3
Aromatics	27.4
Mono Species	24.3
Di Species	1.8
Tri Species	0.8
Tetra Species	0.3
Penta Species	0.0
Thiopheno Species	0.3
Unidentified	0.0
Total CA, Wt%	9.9

GC Simulated Distillation, Method D-2887

Wt% Distilled	Temperature, °F
0	135
1	169
5	214
10	261
20	331
30	378
40	433
50	481
60	511
70	570
80	658
90	774
95	881
99	1,005
100	1,018

### C. Hydrocracking

Hydrocracking was carried out in the second reactor of the pilot plant shown in Figure 13, over catalyst diluted 50 percent by volume with non-porous alumina chips to aid heat dissipation. Table 4 shows the catalyst composition, the same as for the bench-scale experiments discussed in Section IV-F.

Hydrotreated 550°F+ material was fed to the hydrocracker, initially at the conditions suggested by the bench-scale experiments, 650°F, 1,600 psig, and 0.7 WHSV. However, it was necessary to increase temperature to 670°F to achieve the targeted 70 percent conversion of 550°F+ to lower boiling materials. After hydrocracking about half of the available 550°F+ material, the unit was temporarily shut down and the product distilled to provide 550°F+ hydrocrackate for a recycle experiment.

During feed blending for the recycle run, a malfunctioning relief valve resulted in losses of hydrotreated 550°F+ material. As a result, feed for the recycle run contained about 45 percent of the hydrocracked 550°F material instead of 29 percent as implied by material balances in Section IV-A of this report. Therefore, the effects of recycle shown by these results, while directionally correct, may be amplified somewhat.

Tables 19 and 20 show the effects of recycle on product quality and yields. Although only slightly higher temperature (674 vs. 670°F) was required for 70 percent conversion with recycle, gases and light liquid yields increased dramatically. Table 20 shows that gas production more than doubled and that fifty percent more light liquid was produced in the recycle case, at the expense of desirable heartcut material.

However, with recycle the feed contained about ten percent more saturates, mostly paraffinic, that might be expected to increase gas yields in hydrocracking. In an optimized process a heavier heartcut might be taken downstream of the hydrotreaters and the conversion per pass decreased in the hydrocracker to improve liquid yields at the expense of gas makers. However, these represent minor operational rather than design changes to the process and will not significantly affect equipment sizing in Task 4.

These results confirm the recommended design basis by showing the viability of extensive recycle in the hydrocracker, albeit at higher than design concentration. The lower hydrocracker temperature (650°F) recommended in the design basis remains preferred over the 670 to 674°F used in these runs, because this will decrease severity and improve liquid yields.

### D. Jet Fuel Production--Blending, Clay Treating, and Inspection

Table 21 shows the inspection analyses of the two jet fuel samples that were produced, designated JP-8 HC and JP-8 HT. The JP-8HC fuel contains heartcuts of hydrotreated and hydrocracked stocks, blended according to the design basis in Section IV-A. HT fuel contains only hydrotreated stock, because there was not enough hydrocrackate for blending because of the losses during the recycle run discussed in Section V-C.

TABLE 18

SECOND HYDROTREATER--ANALYSES OF PRODUCT FRACTIONS

<u>Elemental Analysis, Dry Basis</u>	<u>Heartcut 160-550°F</u>	<u>Hydrocracker Feed 550°F+</u>
Carbon, wt%	86.91	87.49
Hydrogen, wt%	12.75	12.31
Nitrogen, ppm	3	8
Sulfur, ppm		26
Atomic H/C Ratio		1.68
API Gravity	35.0	24.5
Specific Gravity	0.850	0.907
<u>Mass Spectral Results, wt%</u>		
Saturates	76.0	66.3
Paraffins	8.5	20.7
NCond. Cycloparaffins	34.9	17.6
Cond. 2-Ring Cycloparaffins	26.6	16.0
Cond. 3-Ring Cycloparaffins	6.0	11.9
Aromatics	24.0	33.7
Mono Species	23.0	21.2
Di Species	0.81	6.1
Tri Species	0.00	3.3
Tetra Species	0.00	0.7
Penta Species	0.00	0.1
Thiopheno Species	0.03	2.1
Unidentified	0.00	0.2
Total CA, wt%	12.5	14.2
<u>GC Simulated Distillation, Method D-2887</u>		
<u>wt% Distilled</u>		
0	173	488
1	175	500
5	217	564
10	250	590
20	302	651
30	349	690
40	380	730
50	416	788
60	466	845
70	487	889
80	507	927
90	560	993
95	586	--
99	658	--
100	674	--

TABLE 19

HYDROCRACKER--EFFECT OF RECYCLE

	<u>Without Recycle</u>		<u>With Recycle*</u>	
	<u>Feed</u>	<u>Product</u>	<u>Feed</u>	<u>Product</u>
Carbon, wt%	87.49	86.20	87.10	85.97
Hydrogen, wt%	12.31	13.93	12.78	14.09
Nitrogen, ppm	8			1
Sulfur, ppm	26	2	35	<1
Atomic H/C Ratio	1.68	1.92	1.75	1.95
API Gravity	24.5			
Specific Gravity	0.907	0.828	0.894	0.793
<u>Mass Spectral Results, wt%</u>				
Saturates	66.3	85.7	73.6	90.1
Paraffins	20.7	27.4	34.4	45.3
NCond. Cycloparaffins	17.6	33.8	15.6	32.5
Cond. 2-Ring Cycloparaffins	16.0	15.0	12.0	7.8
Cond. 3-Ring Cycloparaffins	11.9	9.5	11.5	4.5
Aromatics	33.7	14.3	26.4	9.9
Mono Species	21.2	13.1	14.4	0.0
Di Species	6.1	0.0	5.0	0.0
Tri Species	3.3	0.8	3.5	0.0
Tetra Species	0.7	0.1	1.0	0.0
Penta Species	0.1	0.0	0.2	0.0
Thiopheno Species	2.1	0.2	2.3	0.0
Unidentified	0.2	0.0	0.0	0.0
Total CA, wt%	14.2	4.5	11.6	5.3
<u>GC Simulated Distillation, Method D-2887</u>				
<u>wt% Distilled</u>				
0	488	91	485	76
1	500	114	495	79
5	564	191	550	129
10	590	226	577	187
20	651	290	633	234
30	690	359	674	279
40	730	433	705	331
50	788	500	739	399
60	845	588	793	506
70	889	691	851	674
80	927	809	900	786
90	993	912	960	900
95	--	976	1,017	969
99	--	--	--	--
100	--	--	--	--

\*Feed contains 45% hydrocracked 550°F+ material.

TABLE 20

EFFECT OF RECYCLE ON HYDROCRACKER YIELD

	<u>Without Recycle</u>	<u>With Recycle</u>
Nominal WHSV	0.7	0.7
Temperature, °F	670	674
Pressure, psig	1,600	1,600
550°F+ Conversion, %	70.1	70.3
Hydrogen, SCFB	1,165	1,289
Reactor Effluent, % of Feed		
C1	0.006	0.004
C2	0.065	0.053
C3	0.485	0.526
C4	1.41	3.80
C5	1.51	4.94
C6-210°F	8.21	12.9
210-550°F	65.3	54.1
550°F+	25.1	25.9
C1-C4 Gas	1.97	4.38
Total	102.1	102.1

TABLE 21

INSPECTION OF JP-8 PRODUCTS

<u>Requirements</u>	<u>JP-8 Specification</u>	<u>JP-8 HT Inspection</u>	<u>JP-8 HC Inspection</u>
Color, Saybolt	Report		
Total Acid Number, mg KOH/gm, max.	0.015		
Aromatics, Vol%, max.	25.0	23.5	20.0
Olefins, Vol%, max.	5.0	1.5	1.5
Sulfur, Mercaptan, Wt%, max.	0.001	0.000	0.000
Sulfur, Total, Wt%, max.	0.4	0.0	0.0
Distillation Temperature, °C			
Initial Boiling Point	Report		
10% Recovered, max.	205		
20% Recovered, max.	Report		
50% Recovered, max.	Report		
90% Recovered, max.	Report		
End Point, max.	300		
Residue, Vol%, max.	1.5		
Loss, Vol%, max.	1.5		
Flash Point, °C, max.	38	33	27
Gravity, °API or	37-51	--	--
Density, kg/m <sup>3</sup> at 15°C	775-840	850	832
Freezing Point, °C, max.	-50	-50	-50
Viscosity at -20°C, C'stokes, max.	8.0	1.45@100°F	1.42@100°F
Net Heat of Combustion, MJ/kg min.	42.8		
Combustion:			
(1) Hydrogen Content, wt% min.,	13.6	--	--
or (2) Smoke Point, mm min.	25	--	--
or (3) Smoke point, mm min. and	20	22	20
Naphthalenes, Vol% max.	3.0	0.73	0.70
Copper Strip Corrosion,	1b	1a	1a
2 Hr @ 100°C (212°F)			
Thermal Stability:			
Change in Pressure Drop, mm Hg max.	25	1	1
Heater Tube Deposit Visual Rating	Less than Code 3	-2	-2
Existent Gum, mg/100 ml, max.	7.0	4.8	5.2
Particulate Matter, mg/Liter, max.	1.0	--	--
Water Separation Index, Modified min.	70	89	87
Fuel System Icing Inhibitor, Vol%	0.10-0.15	--	--

Note: "HC" product contains hydrotreated and hydrocracked stocks;  
 "HT" product contains only hydrotreated stock.

Both samples were treated over Fluorex LVM clay in a 2 inch diameter by 12 inch high column at a velocity of 1.0 feet per minute before adding 2.8 ppm of UOP U-344 antioxidant. The benefits of clay treatment may be seen by comparing these with properties of the untreated fuels shown in Table 15. Apparently, the clay removes gums and acidity to improve existent gum, copper strip corrosion, and water separation index results.

All of the specifications for JP-8 were met, except the flash point of both samples are low because too much low boiling material was added during blending. The low boiling material was added to satisfy the density requirement. Also, the HT fuel failed the density specification because no lower-density hydrocrackate was added.

The high density of these fuels is attributed to operation of the second hydrotreater at higher throughputs than specified in the recommended design during the pilot plant run, primarily to expedite production. The aromatics content of product hydrotreated at the correct rate would have been lower, allowing the final product to meet both density and flash point requirements.

## SECTION VI

### SUMMARY AND CONCLUSIONS

After initial experiments in Task 2 failed to confirm the plan outlined in Task 1, development of a viable process became the primary objective. Bench scale experiments provided the basis for an improved process plan, established pilot plant conditions for Task 3 and a preliminary design basis for Task 4, and produced small quantities of JP-8, JP-8X, and JP-4 jet fuels.

In the improved process, a distillation pretreatment step removes heteroatom-containing light ends from the tar oil feed to reduce hydrogen consumption, decreasing jet fuel cost by about 2 percent. It was necessary to add a second hydrotreating stage to decrease aromatics to the level specified for jet fuels.

Pilot plant operation in Task 3 confirmed the viability of the Task 2 process by demonstrating extensive recycle of high boiling materials in the hydrocracker. Two barrels of JP-8 jet fuel were produced for evaluation by the Air Force.

Optimization, particularly the data analysis outlined in the Contract for confirming the reaction kinetics used in the Task 1 models, was not completed. Instead efforts were focused on developing a viable process. However, the raw data necessary for this analysis are presented herein, and these were used to specify the process design conditions.

## SECTION VII

### RECOMMENDATIONS

Hydrocracking experiments suggested that coal-derived liquids deactivate catalysts more rapidly than petroleum-derived stocks. This should be verified and the cause determined. Examination of heteroatomic species distributions (particularly nitrogen) would be the logical starting point.

The information from Tasks 2 and 3 provide sufficient data to evaluate a preliminary design basis for the production of JP-8 from the Great Plains tar oil. These data should be used in Tasks 4 and 5.

## REFERENCES

1. Smith, E. B., "Jet Fuels from Coal; Third Quarterly Report," U.S. Department of Energy Research Contract No. DE-FC21-86MC11076, May 1987.
2. Sinor, J. E., "Production of Jet Fuel from Coal-Derived Liquids, Vol 1, Market Assessment for Liquid By-Products from the Great Plains Gasification Plant," AFWAL-TR-87-2042, August 1987.
3. Fleming, B. A., et al., "Production of Jet Fuels From Coal-Derived Liquids, Vol VI, Preliminary Analysis of Upgrading Alternatives for the Great Plains Liquid By-Product Streams," AFWAL-TR-87-2042, September 1988.
4. U.S. Department of Energy, Contract No. DE-RP-22-86PC90015, "Production of Jet Fuel from Coal-Derived Liquids," Effective May 1, 1987.
5. Gary, J. H., and G. E. Handwerk, "Petroleum Refining Technology and Economics," Marcel Dekker, Inc., 1984, page 140.

APPENDIX A

BRIEF DESCRIPTION OF LCI PILOT PLANT

## APPENDIX A

### BRIEF DESCRIPTION OF LCI PILOT PLANT

#### FEED SYSTEMS

The total hydrogen flow is measured using an orifice meter. The individual hydrogen feed rates to the reactor, instrument sensing lines, etc., are measured with rotameters. All the catalytic hydrogen is introduced at the bottom of the reactor.

The oil feed is metered into the reactor loop using a positive displacement pump. The feed rate is monitored by measuring the weight loss in the run tank which is mounted on an electronic scale.

#### EXPANDED BED REACTOR

The LC-Fining reactor system consists of (1) a two-stage, expanded-bed in series reactor system, (2) a recycle surge bomb for each reactor stage, and (3) a duplex remote head recycle pump for each reactor stage. The liquid hourly space velocity is calculated based on either the total thermal reactor volume including the recycle lines and surge bomb or on the settled-bed catalyst volume. The reactor and reactor loop are heated electrically with nine temperature controlled circuits for each reactor stage, which provide almost isothermal operation.

The internal recycle rate is set to provide a bed expansion of 10-35% depending upon catalyst and feed properties. The connecting line to the remote check assembly is purged intermittently with a small quantity of gas oil from the stripping column.

#### RECOVERY SECTION

The net liquid and gas from the reactor loop are separated in the high temperature-high pressure (HT-HP) separator. The light oil overhead from the HT-HP separator is condensed and collected in the low temperature-high pressure (LT-HP) separator where it is removed by level control. Quench water is introduced periodically upstream of the condenser to prevent the buildup of sulfides in the lines. The quench water is taken off in a decanter following the LT-HP level controller. The vapors from the LT-HP separator pass through the main pressure control valve which reduces the pressure to atmospheric. The low pressure gas is chilled, scrubbed, sampled, metered and vented. The hydrogen is not recycled back to the reactors.

The heavy oil from the HT-HP separator is let down through the level control valve to the low pressure-high temperature separator, which is maintained at about 55 psig. The net flow of heavy oil through the recovery section is low. Heavy oil is recycled from the HT-LP separator back to the HT-HP separator to (1) eliminate dead spots which may lead to overheating or phase separation and (2) increase the flow through the level control valve. A small quantity of stripping gas is introduced into the separator to remove a fraction of the gas oil from the heavy oil. The vapors from the HT-LP separator are let down to atmospheric pressure and stripped. The stripped gas oil, removed from the

stripper by level control, is combined with stripper overhead product and the liquid from the cold trap to form the light oil product. The heavy oil is removed from the HT-LP separator by level control, collected and weighed on an electronic scale.

#### SAMPLING AND OPERATING PROCEDURE

Daily operations are usually divided into 12-hour test periods. Routine maintenance and changes in operating conditions are usually made during the day shift. The cold trap liquid is blended in with the light oil receiver liquid prior to analysis. The gas analysis is done on a composite of equal volume hourly samples. The hydrogen consumption is calculated from the normalized feed and product elemental analyses. The yields of  $H_2S$ ,  $NH_3$  and  $H_2O$  are usually obtained from the elemental balances for sulfur, nitrogen and oxygen, respectively.

**REACTOR LOOP**

Total Volume: 1.38 Gallon / stage

Pressure: 2000 psig

**HIGH TEMPERATURE-  
PRESSURE SEPARATOR**

Pressure: 2000 psig

**HIGH TEMPERATURE-  
LOW PRESSURE  
SEPARATOR**

Pressure: 50 psig

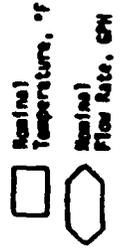
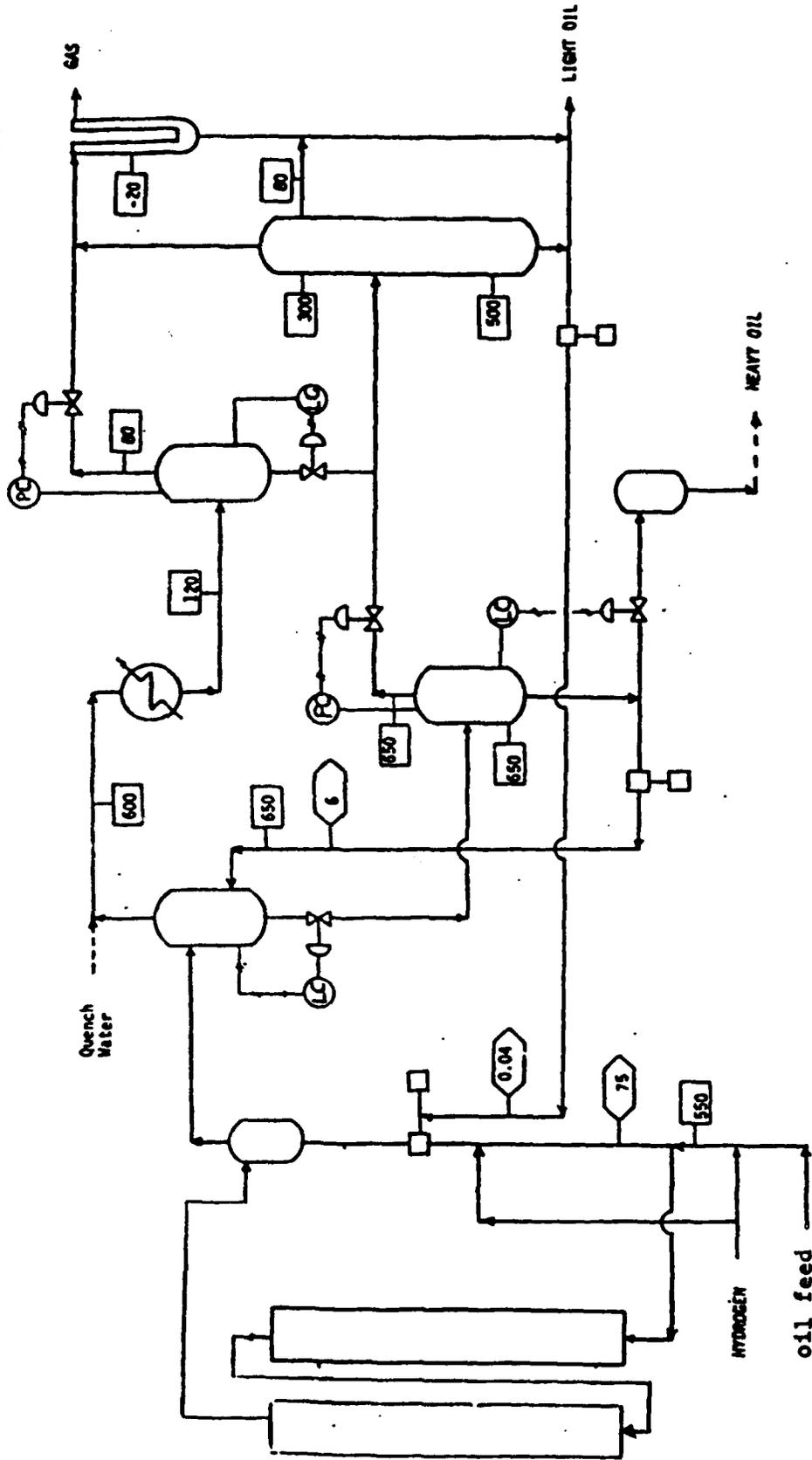
**LOW TEMPERATURE-  
HIGH PRESSURE  
SEPARATOR**

Pressure: 2000 psig

**LIGHT OIL  
STRIPPER**

Pressure: 0 psig

**COLD  
TRAP**



LC1 PILOT PLANT CONFIGURATION

LCI TEST REPORT ON EXPANDED-BED  
HYDROPROCESSING OF 300°F+ GREAT PLAINS TAR OIL

Summary

The six drums (300 gal.) of Great Plains 300°F+ tar oil supplied by Amoco were successfully hydrotreated in LCI's two-stage, expanded bed pilot plant. The tar oil was processed over a twelve-day period at operating conditions recommended by Amoco. The objective of the hydrotreatment was to reduce the nitrogen and other heteroatoms and to maximize hydrogen uptake. These objectives were achieved. The daily nitrogen removals ranged from 95% to 99%. After blending by Amoco, the composited total liquid product should have a nitrogen content of about 150 to 200 ppm. The hydrogen consumption for the entire run averaged about 3200 SCF/bbl. The operating conditions, product properties, yields and conversions for each of the twelve operating days are attached. Six drums of the hydrotreated product were sent to your attention along with the daily 1 quart total liquid product (TLP) samples.

Pilot Plant Operation and Results

A general description of the LC-Fining pilot plant equipment and the sampling and operating procedure have already been provided in earlier communications and will not be repeated here. A brief summary of this specific pilot plant run is given below.

Each of the two LC-Fining reactors was charged with 3500 cc of dried, screened Shell 324 M catalyst. The total catalyst charged was 5760 gms which was used as the basis for the weight hourly space velocity calculation. The catalyst was presulfided with 5%  $\text{HS}_2/\text{H}_2$  at 400°F to 700°F over a 16-hour period following a standard procedure for Shell catalysts. After cooling to 500°F and increasing the pressure to 1500 psig, the tar oil feed was started and adjusted to achieve 0.6 WHSV. The reactor temperatures were increased to 700°F and maintained there for the first day, pending analytical results. As shown in the attached results, the TLP nitrogen content was 365 ppm. This was higher than the 200 ppm N desired by Amoco. The reactor temperatures were increased to 760°F and the total pressure was increased to 2000 psig while the space velocity was maintained at  $0.6 \text{ hr}^{-1}$ . The TLP nitrogen was reduced to 90 ppm. The hydrogen uptake had increased from 2900 SCF/bbl to 3600 SCF/bbl. This exceeded the product specification and in order to process the maximum amount of material in the allotted time, the space velocity was increased to  $0.8 \text{ hr}^{-1}$ . At these conditions the TLP nitrogen was 151 ppm and the hydrogen uptake was 3200 SCF/bbl.

During period 3, the recycle pump on the first reactor shut off (due to electrical high temperature shutoff). The bottom reactor temperature increased to more than 930°F before the pump could be restarted. Since about 2 gal. decant oil had to be added to the pump connecting lines following this incident, the product for the next 6 hours was taken offstream and was not included in the drummed product. There was some increase in the  $\Delta P$  across the reactor following the high temperature excursion and it was thought this might be due to coke formation. However, inspection of the reactor after the run indicated that the pressure drop was primarily due to catalyst which had worked its way through the distributor and into the bottom screen.

LCI TEST REPORT ON EXPANDED-BED HYDROPROCESSING  
OF 300F+ GREAT PLAINS TAR OIL

The TLP nitrogen for period 4 at 0.64 WHSV was 134 ppm so the space velocity was increased to 0.7 hr<sup>-1</sup> and then 0.75 hr<sup>-1</sup> for periods 5 and 6, respectively. During period 8 about 2 quarts decant oil was added to the recycle pump connecting lines accounting for the 104% material balance for the period. This product was kept isolated so Amoco could choose whether or not to blend it. The space velocity was maintained at 0.75hr<sup>-1</sup> up to period 9 when, after the TLP nitrogen had increased to 275 ppm, it was reduced to 0.65 hr<sup>-1</sup> for the remainder of the run.

Throughout the run the recycle line was maintained about 20°F cooler than the reactor temperature. This in combination with the high recycle rate (35-40 gal/hr) facilitated removal of the exothermic heat and provided very effective and stable temperature control. Except for the loss of the recycle pump during period 3, no problems were encountered in controlling the reaction exotherm.

By *V. A. Strangio*  
V. A. Strangio

Approved *M. I. Greene*  
M. I. Greene

Job No. 5-2730

Date: August 25, 1988

**FEEDSTOCK INSPECTION  
COAL DERIVED LIQUIDS**

	LCI	AMOCO
SAMPLE NUMBER	3602	
DESCRIPTION	300 F+	
GRAVITY, DEG API	5.1	6.6
SP.GR. @ 60/60	1.0359	1.028
ELEMENTAL ANALYSIS		
CARBON, WT%	84.29	84.71
HYDROGEN, WT%	8.55	8.60
SULFUR, WT%	0.42	0.49
NITROGEN, PPM	7309	7283
OXYGEN, WT% (BY DIFF)	6.01	5.47
TOTAL	100.00	100.00
SIMDIS (WT%)		
IBP	* 335	279
5	375	336
10	402	371
20	434	414
30	482	468
40	540	498
50	591	556
60	653	603
70	733	683
80	825	765
90	943	877
95	1024	948
FBP	1212	1018

\* SIMDIS was calibrated with pure aromatic standards. It was not calibrated for this particular feedstock. These SIMDIS values should be used only for relative comparisons with the products.

DAILY EXPERIMENTAL RESULTS

PERIOD NUMBER	1	2	3	4	
DATE	1988	8/10	8/11	8/12	8/13
TEMPERATURE, DEG F					
FIRST STAGE TOP	691	758	789	761	
"      "      MID	687	759	796	766	
"      "      BOT	666	740	757	744	
SECOND STAGE TOP	700	754	765	750	
"      "      MID	694	743	761	751	
"      "      BOT	673	733	745	738	
TOTAL PRESSURE, PSIG	1500	2000	2000	2000	
WHSV, LBS/LB/HR	.63	.597	.802	.635	
CATALYTIC H2, SCF/BBL	7300	7700	5700	7200	
CARBON BALANCE, WT% RECOVERY	97	96	99	100	
MATL. BALANCE, WT% RECOVERY	100	101	100	101	
TLP PROPERTIES					
API GRAVITY	27.3	31.6	30.2	29.4	
CARBON, WT%	87.70	86.93	87.60	87.52	
HYDROGEN, WT%	12.17	12.89	12.37	12.46	
SULFUR, WT%	0.03	0.01	0.01	0.01	
NITROGEN, PPM	365	90	151	134	
OXYGEN, WT% (BY DIF)	0.06	0.16	0.00	0.00	
TOTAL	100.00	99.99	99.98	99.99	
* SIMDIS (WT%)					
IBP	194	178	172	176	
10	286	259	249	253	
20	364	337	323	325	
30	406	380	379	385	
40	458	423	419	430	
50	508	468	469	476	
60	561	518	515	528	
70	633	577	572	597	
80	718	656	653	676	
90	853	775	779	797	
FBP	1146	1049	1075	1060	
550 F+ (WT%)	41.8	34.0	33.3	36.2	
DAILY PRODUCT YIELDS, WT% FEED					
OFFGAS H2S (CALC)	0.42	0.44	0.44	0.44	
OFFGAS NH3 (CALC)	0.85	0.88	0.87	0.87	
OFFGAS H2O (CALC)	6.69	6.59	6.76	6.76	
OFFGAS C1	0.36	1.61	0.64	0.56	
OFFGAS C2	0.00	0.62	0.95	0.77	
OFFGAS C3	0.22	0.92	0.44	0.40	
OFFGAS C4	0.16	0.52	0.46	0.41	
OFFGAS C5+	0.40	0.99	1.43	0.80	
TLP	95.07	92.69	92.60	93.60	
TOTAL	104.16	105.26	104.58	104.61	
H2 CONSUMPTION, SCF/BBL					
2900	3600	3200	3200		
WT% 550 F+ CONVERSION					
32	46	47	42		
% DESULFURIZATION					
93	98	98	98		
% DENITROGENATION					
95	99	98	98		
% DEOXYGENATION					
99	98	100	100		

\* SIMDIS to be used for relative comparisons only.

DAILY EXPERIMENTAL RESULTS

PERIOD NUMBER	5	6	7	8	
DATE	1988	8/14	8/15	8/16	8/17
TEMPERATURE, DEG F					
FIRST STAGE TOP	758	756	761	754	
"      "  MID	761	751	759	754	
"      "  BOT	730	729	730	725	
SECOND STAGE TOP	756	755	755	754	
"      "  MID	751	750	748	751	
"      "  BOT	737	738	737	735	
TOTAL PRESSURE, PSIG	2000	2000	2000	2000	
WHSV, LBS/LB/HR	.718	.751	.752	.736	
CATALYTIC H2, SCF/BBL	6400	6100	6100	6200	
CARBON BALANCE, WT% RECOVERY	99	97	97	104	
MATL. BALANCE, WT% RECOVERY	100	98	98	104	
-----					
TLP PROPERTIES					
API GRAVITY	28.8	28.8	28.8	28.2	
CARBON, WT%	87.53	87.56	87.77	87.32	
HYDROGEN, WT%	12.42	12.40	12.55	12.27	
SULFUR, WT%	0.01	0.01	0.01	0.01	
NITROGEN, PPM	155	161	199	250	
OXYGEN, WT% (BY DIF)	0.02	0.01	0.00	0.38	
-----					
TOTAL	100.00	100.00	100.35	100.00	
* SIMDIS (WT%)					
IBP	178	178	172	174	
10	255	254	256	258	
20	330	328	333	342	
30	388	388	394	396	
40	435	436	443	451	
50	481	484	492	501	
60	532	537	543	552	
70	605	612	617	626	
80	683	691	695	707	
90	810	824	825	831	
FBP	1083	1105	1093	1088	
550 F+ (WT%)	37.0	37.7	38.8	40.4	
-----					
DAILY PRODUCT YIELDS, WT% FEED					
OFFGAS H2S (CALC)	0.44	0.44	0.44	0.44	
OFFGAS NH3 (CALC)	0.87	0.87	0.86	0.86	
OFFGAS H2O (CALC)	6.73	6.75	6.76	6.36	
OFFGAS C1	0.52	0.51	0.52	0.51	
OFFGAS C2	0.58	0.44	0.44	0.34	
OFFGAS C3	0.37	0.36	0.36	0.35	
OFFGAS C4	0.39	0.37	0.35	0.37	
OFFGAS C5+	1.11	0.68	0.99	0.94	
TLP	93.56	94.10	93.58	94.20	
-----					
TOTAL	104.57	104.51	104.30	104.37	
-----					
H2 CONSUMPTION, SCF/BBL	3100	3100	3200	3000	
WT% 550 F+ CONVERSION	41	39	39	35	
% DESULFURIZATION	98	98	98	98	
% DENITROGENATION	98	98	97	97	
% DEOXYGENATION	100	100	100	94	

\* SIMDIS to be used for relative comparisons only.

**DAILY EXPERIMENTAL RESULTS**

PERIOD NUMBER	9	10	11	12	
DATE	1988	8/18	8/19	8/20	8/20
TEMPERATURE, DEG F					
FIRST STAGE TOP		760	756	762	762
"      "      MID		758	751	761	760
"      "      BOT		728	729	738	739
SECOND STAGE TOP		754	755	754	753
"      "      MID		748	750	749	748
"      "      BOT		734	738	735	734
TOTAL PRESSURE, PSIG		2000	2000	2000	2000
WHSV, LBS/LB/HR		.732	.636	.648	.667
CATALYTIC H2, SCF/BBL		6300	7200	7100	6900
CARBON BALANCE, WTX RECOVERY		98	96	97	100
MATL. BALANCE, WTX RECOVERY		99	97	98	101
<b>TLP PROPERTIES</b>					
API GRAVITY		29	29.2	29.4	29
CARBON, WTX		87.27	87.86	87.27	87.57
HYDROGEN, WTX		12.33	12.63	12.36	12.40
SULFUR, WTX		0.01	0.01	0.01	0.01
NITROGEN, PPM		275	178	92	148
OXYGEN, WTX (BY DIF)		0.36	0.00	0.35	0.01
TOTAL		100.00	100.52	100.00	100.00
* SIMDIS (WTX)					
IBP		174	174	175	175
10		257	254	254	256
20		336	333	329	333
30		396	393	392	393
40		446	442	440	443
50		495	490	486	491
60		544	540	536	540
70		616	612	605	610
80		693	690	682	687
90		822	814	803	810
FBP		1084	1078	1062	1076
550 F+ (WTX)		39.0	38.3	37.6	38.2
<b>DAILY PRODUCT YIELDS, WTX FEED</b>					
OFFGAS H2S (CALC)		0.44	0.44	0.44	0.44
OFFGAS NH3 (CALC)		0.86	0.87	0.88	0.87
OFFGAS H2O (CALC)		6.38	6.76	6.39	6.75
OFFGAS C1		0.51	0.51	0.62	0.58
OFFGAS C2		0.41	0.47	0.52	0.39
OFFGAS C3		0.36	0.35	0.42	0.40
OFFGAS C4		0.35	0.35	0.41	0.42
OFFGAS C5+		1.13	1.02	1.17	1.06
TLP		94.02	93.45	93.68	93.63
TOTAL		104.45	104.21	104.52	104.54
H2 CONSUMPTION, SCF/BBL		3100	3200	3100	3100
WTX 550 F+ CONVERSION		37	39	40	39
% DESULFURIZATION		98	98	98	98
% DENITROGENATION		96	98	99	98
% DEOXYGENATION		94	100	95	100

\* SIMDIS to be used for relative comparisons only.