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Part IV

AN EXPLORATORY RESEARCH AND DEVELOPMENT PROGRAM  
LEADING TO SPECIFICATIONS FOR AVIATION TURBINE  
FUEL FROM WHOLE CRUDE SHALE OIL



Part IV - Production of Samples of Military Fuels From Raw Shale Oils

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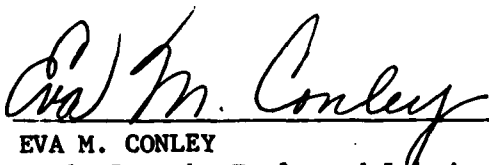
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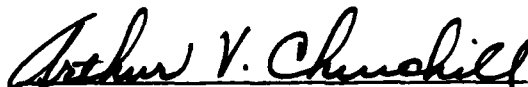
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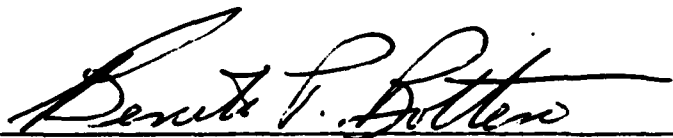
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A total of 475 gallons of specification aviation turbine fuels (JP-4, JP-5 and JP-8) were prepared from Occidental Shale Oil based on Sun Tech's upgrading concept. Processing consists of six steps: (1) hydrotreating the whole shale oil to partially reduce total nitrogen content to minimize hydrogen consumption; (2) distilling the hydrotreated product into appropriate fractions for additional processing; (3) rehydrotreating the light distillate fraction to meet product specifications; (4) treating the wide boiling gas oil fraction with anhydrous hydrogen chloride (HCl) which yields a low nitrogen content raffinate.		

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and a high nitrogen content extract phase; (5) thermally decomposing the extract to recover anhydrous HCl and a nitrogen-rich extract, which is used for generating hydrogen by partial oxidation; and (6) hydrocracking the raffinate phase to maximize aviation turbine fuel yield.

Five 5-gallon samples of specification military fuels were produced from Paraho shale oil (JP-4, JP-5, JP-8, DF-2 and DF Marine) using a modified process. Processing consists of severely hydrotreating raw shale oil followed by fractionation and finally hydrocracking the wide boiling gas oil fraction to produce the desired product slate.

Results of the individual processing steps, inspections and analyses of the products are discussed along with the process uncertainties, conclusions and recommendations.

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## FOREWORD

This report presents the results of the work performed by Sun Tech for the Air Force under Phase III of Contract F33615-78-C-2024 MOD P00010. The work program is sponsored by the Aero Propulsion Laboratory, Air Force Wright Aeronautical Laboratory, Wright-Patterson AFB, Ohio, under Project 2480, Task 00 and Work Unit 01. Ms. Eva M. Conley/AFWAL/POSF, was the assigned Air Force Project Engineer. The purpose of this phase of the contract is to:

1. Confirm the final process design estimates proposed in Phase I, and
2. Produce sample lots of military fuels for performance evaluations by AFWAL/POSF.

Work on Phase III started 1 April 1980 and was completed 30 November 1981. The results of this work were presented at the "3rd Jet Fuel From Shale Oil Technology Review" sponsored by the Aero Propulsion Laboratory, held on 17-18 November 1981 at Miamisburg, Ohio. This report was released by the Authors on 13 January 1982.

Sun Tech's program manager wishes to express his appreciation to Dr. Herbert Lander and Ms. Eva Conley for their assistance and guidance in bringing this program to a successful conclusion.

The authors gratefully acknowledge the contributions of E. J. Janoski, J. J. Vanvenrooy and K. P. Fogarty for their assistance in carrying out the pilot plant operations and technical support.

This report is Part IV of the five planned parts covering the exploratory and development program leading to specifications for military fuels from whole crude shale oil. Part I, "Preliminary Process Analyses" evaluated three different technically feasible processing schemes proposed by Sun Tech, Inc., for converting 100,000 BPCD of raw Paraho shale oil into military turbine fuels. Part II, "Process Variable Analyses and Laboratory Sample Production", incorporated pilot plant process data for three design bases for manufacturing military fuels from raw Occidental shale oil. Part III, "Production of 300 Barrels of JP-4 Turbine Fuel From Geokinetics Shale Oil" reports the results of the program carried out at Hydrocarbon Research, Inc., Lawrenceville, N.J., laboratory in their 30 barrel per day process demonstration unit under sub contract to Sun Tech. Part V, still to be issued, will cover the Phase IV program, "Overall Economic Optimization".

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## LIST OF SYMBOLS AND ABBREVIATIONS

### Symbols

Bbl/ton	Barrels per ton
°F	Degrees Fahrenheit
Mol. %	Mole percent
SCF/Bbl	Standard Cubic Feet per Barrel
v/hr/v	Volume of Liquid per hour per Volume of Catalyst
vol. %	Volume percent
wt. %	Weight percent

### Abbreviations

Bbl	Barrels
cs	Centistokes
g	Grams
H <sub>2</sub>	Hydrogen Gas
HCl	Anhydrous Hydrogen Chloride
H <sub>2</sub> S	Hydrogen Sulfide Gas
JFTOT	Jet Fuel Thermal Oxidation Tester
KV	Kinematic Viscosity
LHSV	Liquid Hourly Space Velocity
mm Hg	Millimeters of Mercury
Mo	Molybdenum
N <sub>2</sub>	Nitrogen
NH <sub>3</sub>	Ammonia Gas
Ni	Nickel
ppm	Parts per Million by Weight
psig	Pounds per Square Inch Gage Pressure
R-1	First Reactor
R-2	Second Reactor
RVP	Reid Vapor Pressure
S	Sulfur
TBP	True Boiling Point Distillation
TPO	Texaco Partial Oxidation Process

## SUMMARY

This report covers work performed by Sun Tech, Inc. in Phase III of our contract with the United States Air Force. The Phase III objectives were to: 1) confirm Sun Tech's process designs; and 2) produce component test samples of specification military fuels from raw shale oils in existing pilot plant equipment. Non-proprietary catalysts were used exclusively in the various processing steps.

Sun Tech's processing concept for refining raw Occidental shale oil consists of six distinct steps: (1) hydrotreating the whole shale oil to partially reduce the high total nitrogen content (and convert some neutral nitrogen to basic nitrogen), while minimizing hydrogen consumption; (2) distilling the hydrotreated product into appropriate fractions for additional processing; (3) rehydrotreating the light distillate fraction to meet product specifications; (4) treating the wide boiling distillate fraction with anhydrous hydrogen chloride which yields a raffinate and extract phase--the nitrogen content in the HCl raffinate is lowered and concentrated in the extract phase; (5) thermally decomposing the HCl extract to recover anhydrous hydrogen chloride--the recovered HCl-free nitrogen-rich extract fraction is used for generating hydrogen by partial oxidation; and (6) hydrocracking the raffinate fraction to maximize the yield of aviation turbine fuels. A total of 475 gallons of aviation turbine fuels were prepared from Occidental modified in-situ shale oil--170 gallons of JP-4, 150 gallons of JP-5, and 155 gallons of JP-8.

A modification of Sun Tech's processing scheme was employed to produce five additional samples of military fuels from raw Paraho shale oil. The modified processing route consists of four processing steps: (1) severely hydrotreating the raw shale oil, (2) distilling the hydrotreated material into appropriate fractions for additional processing, (3) rehydrotreating the naphtha fraction, and (4) hydrocracking the gas oil fraction to produce lower boiling distillate fuel fractions. Five 5-gallon samples of specification military fuels were produced from Paraho shale oil--JP-4, JP-5, and JP-8 jet fuels, along with Diesel Fuel No. 2 and Diesel Fuel Marine.

SECTION I  
INTRODUCTION

The purpose of the Phase III program was to demonstrate at the pilot plant scale the applicability of Sun Tech's processing scheme for refining raw shale oils.

This report covers the hydroprocessing of multi-drum quantities of Occidental in-situ shale oil to produce blending stock for a slate of jet fuels. A block flow diagram of Sun Tech's proposed process is shown in Figure 1. Hydrotreating is necessary both before and after the key extraction step to produce a feed suitable for hydrocracking. This report also covers the production of a slate of military fuels from Paraho shale oil as shown by the block flow diagram in Figure 2. In this case, a relatively severe initial hydrotreat allowed hydrocracking of the gas oil fraction to take place without any additional treatment.

The first pass with Occidental shale oil feedstock through Sun Tech's Adiabatic Pilot Plant unit took place under moderately severe hydrotreating conditions to produce a product containing 5000 ppm total nitrogen,  $N_T$ . Distillation of this material yielded a "straight-run" naphtha, a gas oil fraction (530-1000°F), and a 1000°F+ bottoms fraction. The gas oil fraction then went to the anhydrous HCl extraction step to remove basic nitrogen compounds.

The raffinate from the extraction stage was water washed and then hydrocracked. In the hydrocracking operation, the HCl raffinate was severely hydrotreated to remove residual nitrogen containing compounds as well as the organic chlorides formed during the extraction step. The straight-run naphtha was also given a second pass through the hydrotreater to eliminate any remaining nitrogen containing compounds. Relatively severe operating conditions were employed in this second hydrotreating step in order to get at those compounds which had resisted the first hydrotreating step on the whole shale oil. The hydrotreated raffinate was finally processed through the main hydrocracking reactor and separated in the unit's stripping column into an overhead hydrocracked naphtha fraction and a hydrocracked gas oil bottoms product.

The Sun Tech processing scheme used for producing military fuels from raw Paraho shale oil employs a relatively severe initial hydrotreat to produce a liquid product containing 100 ppm total nitrogen. Distillation of the hydrotreated product gave a naphtha and a gas oil fraction. The naphtha was rehydrotreated and the gas oil fraction was hydrocracked to produce lower boiling distillate fuel fractions. A block flow diagram of Sun Tech's process for preparing military fuels from Paraho shale oil is given in Figure 2.

The details of the pilot plant program for the implementation and demonstration of these processing schemes are detailed in the following sections of this report.



SECTION II  
PILOT PLANT PRODUCTION OF AVIATION TURBINE FUEL  
SAMPLES FROM WHOLE OCCIDENTAL SHALE OIL

1. Feedstock Preparation

Twenty-seven 55 gallon drums of Occidental modified in-situ shale oil were available for use in this program. The shale oil was transferred to a 1500 gallon heated storage tank equipped with a circulating blending pump system. The contents of the tank were heated to 175°F and the water that separated out was drained out the bottom. After two hours of blending by means of the circulating pump, the contents of the tank were pumped through a 5 micron Cuno filter into 55 gallon drums for storage prior to charging into Sun Tech's Adiabatic Pilot Plant. The physical and chemical properties of this material are detailed in Table 1.

2. Raw Shale Oil Hydrotreating

Sun Tech's Adiabatic Pilot Plant Unit, with a four bed 10-liter reactor in place, was used for processing about 1500 gallons of Occidental in-situ shale oil.

A simplified flow diagram of the Adiabatic Pilot Plant is shown in Figure 3. Water was used to scrub the recycle gas during each of the hydro-processing steps. Waste water was sent to the refinery separation system.

For the raw shale oil hydrotreating step the main activity was provided by Shell 324 catalyst. Shell 514 support balls were installed on top of the catalyst bed to serve as a guard section for removal of metals from the feedstock while providing sufficient void volume to forestall premature bed plugging. These support balls were also used in the various quench zones between the catalyst beds.

The initial operating conditions chosen to hydrotreat the raw shale oil to 5000 ppm  $N_T$  were:

Average Catalyst Bed Temperature, °F	675
Total Pressure, psig	1600
Liquid Hourly Space Velocity, v/hr/v	1
Recycle Gas Rate, SCF/Bbl	3000

These initial operating conditions yielded a product having 5800 ppm  $N_T$ . Accordingly, the average bed temperature was raised 15°F to 690°F. This adjustment was sufficient to drop the product  $N_T$  level to 5000 ppm as targeted. Operating problems encountered during this phase of the work were associated with loss of water feed to the recycle gas scrubbing system. When this problem occurred, ammonium sulfide quickly plugged filters, gas flow controllers and vent lines. Also, the charge line preheater plugged from coke laydown due to using the charge line for heat input.

Several material balances were performed during the raw shale oil hydrotreating step. Table 2 presents data from a typical material balance

period. The hydrogen consumption data reported represents the amount chemically consumed. Table 3 contains an analysis of the gas exiting the stripping column not including the stripping nitrogen. The so-called analytical gas also shown in Table 3 is actually the high pressure bleed gas which is taken in sufficient quantity to maintain a hydrogen purity at better than 95 mol. %. Included in this table is an analysis of the wash water used to scrub  $H_2S$  and  $NH_3$  out of the recycle gas. Finally, a summation of these miscellaneous components, i.e. other than liquid product, is also included. Water produced by the removal of oxygen from the raw shale oil is not included in material balances. Inspections and analyses of the liquid product containing 5000 ppm total nitrogen are given in Table 4. Figure 4 presents smoothed TBP distillation curves for the raw and hydrotreated Occidental shale oils.

### 3. Distillation of Hydrogenated Occidental Shale Oil

About 1600 gallons of whole crude Occidental shale oil hydrogenated to contain approximately 5000 ppm total nitrogen in the liquid effluent were distilled in Sun Tech's continuous atmospheric pressure and vacuum distillation pilot plants, shown in Figure 5.

The goal of this distillation was to prepare the following fractions:

- IBP - 530°F by atmospheric distillation
- 530 - 1000°F by vacuum distillation
- 1000°F+ bottoms from the vacuum distillation

These distillation cut points were chosen to minimize overall distillation times and costs. It was necessary to separate the 1000°F+ bottoms fraction prior to the HCl extraction step to eliminate emulsion problems encountered when water washing the raffinate.

The Initial-530°F boiling fraction was used as feedstock to the naphtha hydrotreater, where it was hydrotreated at a relatively high liquid hourly space velocity to yield a low nitrogen, stable "straight run" jet fuel blending component.

The 530-1000°F distillate fraction was HCl treated in Sun Tech's 500-gallon stirred glass lined reactor to yield raffinate feedstock for hydrocracking.

As proposed in our processing scheme, the 1000°F+ bottoms from the distillation unit would be used as a feedstock for manufacturing hydrogen via the Texaco partial oxidation process.

Inspections and analyses of the fractions from hydrotreated shale oil are shown in Table 5. Nitrogen, sulfur, and aromatic content increase with increasing boiling range.

#### 4. Naphtha Hydrotreating

The purpose of naphtha hydrotreating was to clean up this fraction sufficiently to meet final product specifications. Sun Tech's 10 liter

Adiabatic Pilot Plant was repacked with fresh Shell 324 Ni-Mo catalyst, and sulfided.

The operating conditions used for hydroprocessing the straight-run naphtha were:

Average Catalyst Temperature, °F	760
Total Pressure, psig	1500
Liquid Hourly Space Velocity, v/hr/v	1.4
Recycle Gas Rate, SCF/B	4000

Initially we attempted to run at 2.0 LHSV, but were forced to cut back to 1.4 LHSV because of the limited capacity of the feed preheater. When lined out these relatively severe operating conditions resulted in a product obtained from the straight-run naphtha having less than 5 ppm  $N_T$ . The stabilized liquid product had passed the JFTOT thermal stability test with no pressure drop and a preheater deposit code of zero.

Detailed analyses of the hydrotreated product are given in Table 6.

##### 5. Anhydrous HCl Extraction of Gas Oil Fraction

Pilot plant HCl extraction of the hydrogenated gas oil distillate fraction from Occidental whole crude shale oil was carried out batchwise in four separate runs. A simplified schematic flow diagram of Sun Tech's batch HCl extraction unit is shown in Figure 6.

The first HCl extraction run was a trial run to confirm results obtained in the laboratory. Three hundred gallons (2201.5 lbs.) of a 450-900°F distillate fraction (containing 3300 ppm total nitrogen) of hydrotreated Occidental whole crude shale oil containing 5200 ppm total nitrogen were treated with dry HCl gas in a 500 gallon, stirred, glass lined Pfaudler reactor. The reactor was pressured with dry nitrogen to 8 psig pressure. To the stirred contents of the reactor were added 49.5 lbs. of HCl from a cylinder of the compressed gas, causing the pressure in the reactor to increase from the initial of 8 to a final of 14 psig and the temperature increased from the initial of 110 to a final of 117°F. No HCl escaped during the addition. The addition required 4.5 hours, the rate of addition was limited by the rate of vaporization of HCl in the cylinder. External heating of the cylinder would have greatly increased the rate of addition of the reagent, since the reaction rate was very rapid.

The quantity of HCl added corresponded to that found in previous laboratory work. The emulsion of amine hydrochloride in hydrocarbon broke quickly when the stirring was stopped. The lower, dense amine hydrochloride phase was then drawn off. The yield of extract phase was 225 lbs. or 10.2 wt. % of the charge. Washing the extract with n-hexane removed some occluded raffinate and the corrected yield of extract was 7.3 wt. %, close to the laboratory result. The hexane-washed extract contained 11.49 wt. % chlorine and 3.84 wt. % nitrogen.

The raffinate phase, corresponding to 94.03 wt. % of the gas oil charge, was washed with two 90 gallon batches of water. After the first water

wash the raffinate was acid, but it was neutral after the second wash. No problems with emulsions were encountered. The washed and neutral raffinate phase contained 491 ppm total nitrogen and 359 ppm chloride.

The large-scale HCl treatment turned out to be a very easy straight forward operation, confirming and exceeding the results of earlier laboratory trials. The raffinate from the first large-scale HCl treatment was used as a feed to hydrocracking for producing hydrocrackate for blending into specification grades of JP-4, JP-5, and JP-8 jet fuels.

Table 7 gives an analysis of the washed raffinate from the first HCl extraction run. Note that total nitrogen content has been reduced significantly from 3300 ppm to approximately 500 ppm.

Based on the encouraging results of the initial large scale HCl extraction, HCl extraction of the three additional 325 gallon batches of the 530-1000°F distillate fraction from hydrotreated Occidental shale oil were completed. All three HCl extractions went smoothly and the results are in agreement with the pilot runs made in the laboratory. Results available from the final three large batch runs are summarized in Table 8.

Recovered undecomposed HCl extract has been held in storage for decomposition at a later date.

## 6. Hydrocracking HCl Raffinate

Hydrocracking the raffinate from the HCl extraction step required two distinct processing steps--R-1 hydrotreating to partially saturate the aromatics and to remove the remaining nitrogen and sulfur compounds from the raffinate, and R-2 hydrocracking for molecular weight reduction to produce aviation turbine fuels.

### a. R-1 Hydrotreating

Using the same catalyst loading that was employed for the naphtha hydro-treating, the raffinate from the anhydrous HCl extraction step was charged to Sun Tech's 10 liter Adiabatic Pilot Plant. However, the 10 liter Adiabatic reactor pilot plant had to be modified to provide for injecting water directly into the reactor outlet line as shown in Figure 3. This change was needed to overcome plugging problems caused by ammonium chloride deposits in the reactor effluent line. The operating conditions that were employed were identical to those used in the naphtha hydrotreating runs:

Average Catalyst Temperature, °F	760
Total Pressure, psig	1700
Liquid Hourly Space Velocity, v/hr/v	1.4
Recycle Gas Rate, SCF/B	4000



Residual chlorides and nitrogen in the raffinate feedstock are released in the reactor as HCl and ammonia gases which react to form solid ammonium chloride. Ammonium chloride decomposes at 662°F, migrates and solidifies at locations of lower temperature and plugs the system. Water injection cools and dissolves the ammonium chloride. The water phase is separated from the oil phase in the low pressure separator. This system was used successfully for the previous Phase II pilot plant work-- "Verification of Hydrocracking Model".

The effluent line from the Adiabatic reactor was fabricated from 304 austenitic stainless steel tubing and had a 90 degree bend before going to the effluent cooler. The line was subject to heat, pressure, moisture, and chlorides in the presence of water. This combination of conditions resulted in rapid failure at the 90 degree bend after two drums of feed were processed. Line failure was not catastrophic but rather occurred via a slow leak from a web of fine cracks. Because the original line had seen several years of previous service and in an effort to complete the run quickly, a new 304 stainless steel line was installed. This line also failed shortly after the unit started up. An investigation of these failures revealed that the two 90 degree bends had not been stress relieved before being placed into service. A detailed inspection of the unit downstream of the 90 degree bend did not reveal any other points of chloride stress corrosion. On the recommendation of Sun Tech's Metallurgy Laboratory, a high nickel content tubing, Inconel Alloy 600, was procured, fabricated and stress relieved before being connected to the reactor outlet. In addition, ammonia was added to the injected water to insure that there would be an excess of that needed to neutralize the HCl

as it was generated. These measures solved the chloride stress corrosion problem permitting completion of this phase of the work. The R-1 reactor product was found to contain less than 10 ppm  $N_T$ . The oil-water mixture going to the heated settling tank had a tendency to emulsify. Tretolite DS 638 demulsifier in the amount of 25 ppm was added to the oil layer to facilitate product dewatering.

R-1 feed and reactor effluent inspections from steady state operation are summarized below:

	<u>Feedstock</u>	<u>Product</u>
Boiling Range, °F	550-1000°F	$C_4$ -1000°F
API @ 60°F	30.6	31.6
Total $N_T$ , ppm	700	<10
Sulfur, ppm	30	2
Chlorides, ppm	400	nil
Aromatics, wt. %	33.1	23.5

As a result of several emergency shutdowns and startups due to line failure, some off-test product had to be recycled to meet the 10 ppm max. total nitrogen specification. About 950 gallons of HCl raffinate was processed thru R-1 for use as feed to R-2 of the hydrocracker.

b. R-2 Hydrocracking

The initial operating conditions chosen for the R-2 hydrocracking runs were as follows:

Average Catalyst Bed Temperature, °F	715°F
Total Pressure, psig	1700
Liquid Hourly Space Velocity, v/hr/v	2.0
Recycle Gas Rate, SCF/Bbl	4000

These conditions were not satisfactory since the feed preheater was undersized for this high charge rate. Reducing the feed rate to 1.6 LHSV increased the temperature of the feed to the point that the inlet portion of the catalyst bed was effectively utilized. The hydrocracking activity of the Harshaw Ni-4301 catalyst was excessive and the light ends produced were more than the gas scrubbing system could handle. In addition, the water scrubbing of the recycle gas was shutdown since the feedstock was free of heteroatoms that might cause line plugging.

Average bed temperature was adjusted downward to 700°F in an effort to adjust hydrocracking severity. After several hundred gallons of feed had been processed, a drum containing high nitrogen content was accidentally used as feedstock. It became apparent that the nitrogen poisoned the catalyst and some hydrocracking activity was lost as evidenced by an increase in the freeze point of the hydrocracked bottoms product. The average catalyst bed temperature was raised to 775°F in an attempt to offset lost activity.

Several material balance runs were obtained before the catalyst lost any significant activity. Table 9 contains the data from a typical test period of 8.0 hours duration. Table 10 contains an analysis of the gas exiting the stripping column excluding the stripping nitrogen. The

composition of the analytical gas, i.e., high pressure bleed, is also shown in Table 10 along with the summation of the gaseous components.

The overhead liquid (I-300°F) product from the stripping column amounted to 40 wt. % and the 300°F+ bottoms accounted for the other 60 wt.% of the total liquid product. No attempt was made to recycle the unconverted bottoms to extinction, since the final product distillation was carried out as a separate operation.

Typical results from combined R-1 and R-2 hydrocracking operation are shown in Table 11. Overall, a 111 volume % yield of liquid products from R-2 is attained.

#### 7. Product Blending

The three intermediate fractions available for jet fuel blending are characterized in Table 12. Note that the I-300°F boiling light hydrocrackate has F-1 clear and F-2 clear octane numbers of 69.5 and 67.9 respectively. This stream would be a suitable feedstock for catalytic reforming. These intermediate streams were distilled in a general purpose continuous distillation column, where they picked up some trace contaminants, causing excessive pressure drop in the JFTOT thermal stability tests on the final fuels. Percolation of these streams through Filtrol 24 percolation grade clay at a dosage of 250 bbl/ton of clay corrected this deficiency. No attempt was made to optimize the clay treat.

Inspections and analyses of the 170 gallon sample of JP-4 prepared from Occidental shale oil are given in Table 13. This fuel met all product specifications. Since there was little light material in the hydro-treated naphtha, only 27 volume % of the jet fuel blend was hydrotreated naphtha. The remaining 73 volume % was hydrocracked naphtha.

The 150 gallon sample of JP-5 jet fuel prepared from Occidental shale oil met all military specifications. The fuel, containing 82 volume % hydro-treated naphtha and 18 volume % hydrocracked naphtha, was precut to insure a minimum jet fuel flash point of 140°F. Inspections and analyses of the shale oil derived JP-5 are shown in Table 14.

The 155 gallon sample of JP-8 jet fuel prepared from Occidental shale oil had 57 volume % hydrotreated naphtha and 43 volume % hydrocracked naphtha. The jet fuel was distilled with a relatively low end point to assure obtaining a product meeting all military specifications. Properties of this fuel sample are given in Table 15.

Anti-icing, anti-corrosion, and anti-oxidant inhibitors were added to all fuels at a concentration slightly under the maximum limit. Table 16 summarizes the properties and functions of these inhibitors.

SECTION III  
PILOT PLANT PRODUCTION OF MILITARY FUEL SAMPLES  
FROM RAW PARAHO SHALE OIL

1. Feedstock Preparation

Two drums of Paraho shale oil obtained from a directly heated surface retort were used for this work. The contents of the drum were heated to 175°F in a hot box and the water that separated out was discarded. The dewatered shale oil was then pumped through a 5 micron Cuno filter into new 55-gallon steel drums for storage prior to charging into Sun Tech's Adiabatic Pilot Plant.

2. Raw Shale Oil Hydrotreating

Approximately 100 gallons of raw Paraho shale oil were continuously hydrotreated in Sun Tech's Adiabatic Reactor over the same catalyst loading that had previously been used for the routine production of the 1500 gallon sample of hydrotreated Occidental Shale Oil containing 5000 ppm total nitrogen. Operating conditions required to hydrorefine Paraho shale oil to 100 ppm total nitrogen were:

Average Catalyst Bed Temperature, °F	760
Total Pressure, psig	1700
LHSV, v/hr/v	0.25
Recycle Gas Rate, SCF/B	3000

Several material balance runs were performed during the hydrogenation of raw Paraho shale oil. Table 17 presents data from a typical material balance run. The characteristics of the liquid product are given in Table 18. Figure 7 presents smoothed TBP distillation curves for the raw and hydrotreated Paraho shale oils.

### 3. Distillation of Hydrotreated Paraho Shale Oil

The hydrotreated liquid product containing approximately 100 ppm total nitrogen was distilled in a Sarnia Mark II fractionator operating at 15 theoretical plates and a 5:1 reflux ratio. Listed below are the fractions by boiling range and their respective yields:

	<u>Volume %</u>
Initial-180°F	0.1
180-490°F	22.1
490-540°F	8.5
540-650°F	22.0
650-1000°F	45.3
1000°F <sup>+</sup> bottoms	2.0

These fractions were further processed in order to produce the required slate of military fuels. The Initial-490°F cut was rehydrotreated in order to produce a stable JP-4 jet fuel blendstock. The other distillate fractions were hydrocracked to produce JP-4 jet fuel blendstock, along with JP-5, JP-8, Diesel Fuel No. 2 and Diesel Fuel Marine. As proposed in our processing scheme, the 1000°F<sup>+</sup> bottoms from the distillation unit would be used as feedstock for manufacturing hydrogen via the Texaco Partial Oxidation Process.

Inspections and analyses of the fractions from Paraho shale oil hydro-treated to 100 ppm total nitrogen are given in Table 19. Overall, the fractions have a relatively low aromatic content. The basic nitrogen represents over 50% of the total nitrogen.

#### 4. Naphtha Hydrotreating

The purpose of the naphtha hydrotreating step was to cleanup the Initial-490°F boiling feedstock in order to meet final product specifications. Sun Tech's Isothermal Pilot Plant, schematic flow diagram shown in Figure 8 was used for this work. Reactor R-1 in Figure 8 was loaded with Shell 324 Ni-Mo catalyst. Operating conditions for treating the naphtha feedstock were:

Average Catalyst Temperature, °F	730
Total Pressure, psig	1500
LHSV, v/hr/v	2.0
Recycle Gas Rate, SCF/B	5000

This step may not have been required, since the feedstock was relatively free of heteroatoms before this final hydrotreat. Analyses of the feedstock and stabilized liquid product are given in Table 20.

#### 5. Hydrocracking Gas Oil Fraction

Two separate operations were employed in this step--R-1 hydrotreating to remove the remaining nitrogen compounds from the gas oil feedstock, and R-2 hydrocracking for molecular weight reduction to produce the required



samples of military fuels. Both the 490-650°F and 650-1000°F boiling fractions from the raw shale oil hydrotreater were processed in this manner, in the Isothermal Pilot Plant.

a. R-1 Hydrotreating

Using the same catalyst loading that was employed for the naphtha hydrotreating, the gas oil was passed through reactor R-1 to remove the remaining nitrogen and sulfur compounds from the feedstock. The operating conditions for R-1 hydrotreating of the 490-650°F and 650-1000°F boiling distillate were:

Average Catalyst Temperature, °F	750
Total Pressure, psig	1700
LHSV, v/hr/v	1.0
Hydrogen to Oil Ratio, SCF/B	5000

R-1 feed and reactor effluent inspections are summarized below:

	<u>Feedstock</u>	<u>R-1 Product</u>
Boiling Range, °F	650-1000	C <sub>4</sub> -1000
API Gravity @ 60°F	30.8	31.6
Total Nitrogen, ppm	163	<1
Sulfur, ppm	21	2
Yield, Vol. % Feed	100	101

b. R-2 Hydrocracking

The product from the R-1 hydrotreat was charged to reactor R-2, loaded with Harshaw Ni-4301 NiW on silica alumina hydrocracking catalyst. The R-2 hydrocracking operating conditions used were:

Average Catalyst Bed Temperature, °F	750
Total Pressure, psig	1700
LHSV, v/hr/v	2.0
Hydrogen to Oil Ratio, SCF/B	5000

Both the 490-650°F and 650-1000°F boiling gas oil fractions were hydrocracked in this manner. The overall yield of liquid products was 110 volume % of the gas oil feed. Typical results from the hydrocracking operation are shown in Table 21. The yield of 675°F + bottoms, not shown in Table 21, was 11 volume % of the gas oil feed.

6. Product Inspections and Analyses

Inspections and analyses of the five gallons of specification JP-4 jet fuel prepared from Paraho shale oil are given in Table 22. This sample contains 27 volume % hydrotreated naphtha and 73 volume % hydrocracked naphtha. The sample has a very low aromatic content and freeze point.

Inspections and analyses of the five gallon samples of JP-5 and JP-8 prepared from Paraho shale oil are shown in Tables 23 and 24. Both fuels met their respective military specifications. Freeze points on the pilot plant samples were well below the required values.

Inspections and analyses of the five gallon sample of Diesel Fuel No. 2 prepared from Paraho shale oil are given in Table 25. A cetane index of 57, a calculated value, is shown in place of cetane number, a measured value, as given in the specification.

Inspections and analyses of the five gallon sample of Diesel Fuel Marine prepared from Paraho shale oil are given in Table 26. This sample was prepared from 100% hydrocrackate. Again, all military specifications were met.

## SECTION IV

### CONCLUSIONS

1. A total of 475 gallons of specification aviation turbine fuels were prepared in the pilot plant from Occidental modified in-situ shale oil using Sun Tech's processing concept. Product volumes were 170 gallons of JP-4, 150 gallons of JP-5, and 155 gallons of JP-8.

2. Some trace contaminants were picked up during the final product distillations in a general purpose still which resulted in excessive  $\Delta P$ 's in the JFTOT tests. This problem was corrected by percolating the turbine fuels through clay.

3. A stress corrosion problem was encountered with the HCl raffinate fraction (700 ppm Cl) early in the hydrocracking operation. A leak occurred at an unannealed 90° bend in the R-1 reactor outlet line. This problem was solved by replacing the 304 type stainless steel with high nickel Inconel Alloy 600 tubing and stress relieving the bend after fabrication.

4. A total of 25 gallons of specification military fuel samples were prepared from Paraho shale oil using a modified processing scheme--severe hydrogenation and hydrocracking (five gallon samples each of JP-4, JP-5,

JP-8, DF-2 and DF Marine). This processing scheme was an outgrowth of a high severity hydrogenation run made in 1980 to produce a large volume sample of JP-4 in one step from Geokinetics in-situ shale oil. Hydrocracking was added to increase the yield of turbine fuels. No unusual processing problems were encountered during pilot plant operations.

SECTION V  
RECOMMENDATIONS

1. The chloride content in the HCl raffinate varied over a wide range from run to run--100 to 800 ppm Cl. Additional HCl extraction work is needed to determine how to minimize residual chloride concentration.
  
2. Continuous HCl treating and recovery processes have not been demonstrated. Large scale runs had to be made batchwise due to the lack of suitable continuous equipment. Continuous HCl treating should be carried out to confirm or modify Phase III data.
  
3. The main hydrotreater catalyst life at high reactor severity (100 ppm total nitrogen in product) with Paraho shale oil was not determined. It is recommended that a main hydrotreater catalyst life test at high reactor severity be initiated.
  
4. Extinction recycling of hydrocracked bottoms was not demonstrated in the pilot plant. Time permitted only once through operation with the equipment available. It is recommended that pilot plant hydrocracking with extinction recycle be run in order to firm up data generated by Sun Tech's Hydrocracking Math Model. If this type of operation is not feasible due to a buildup of aromatics or wax in the recycle oil, a drag stream may be required.

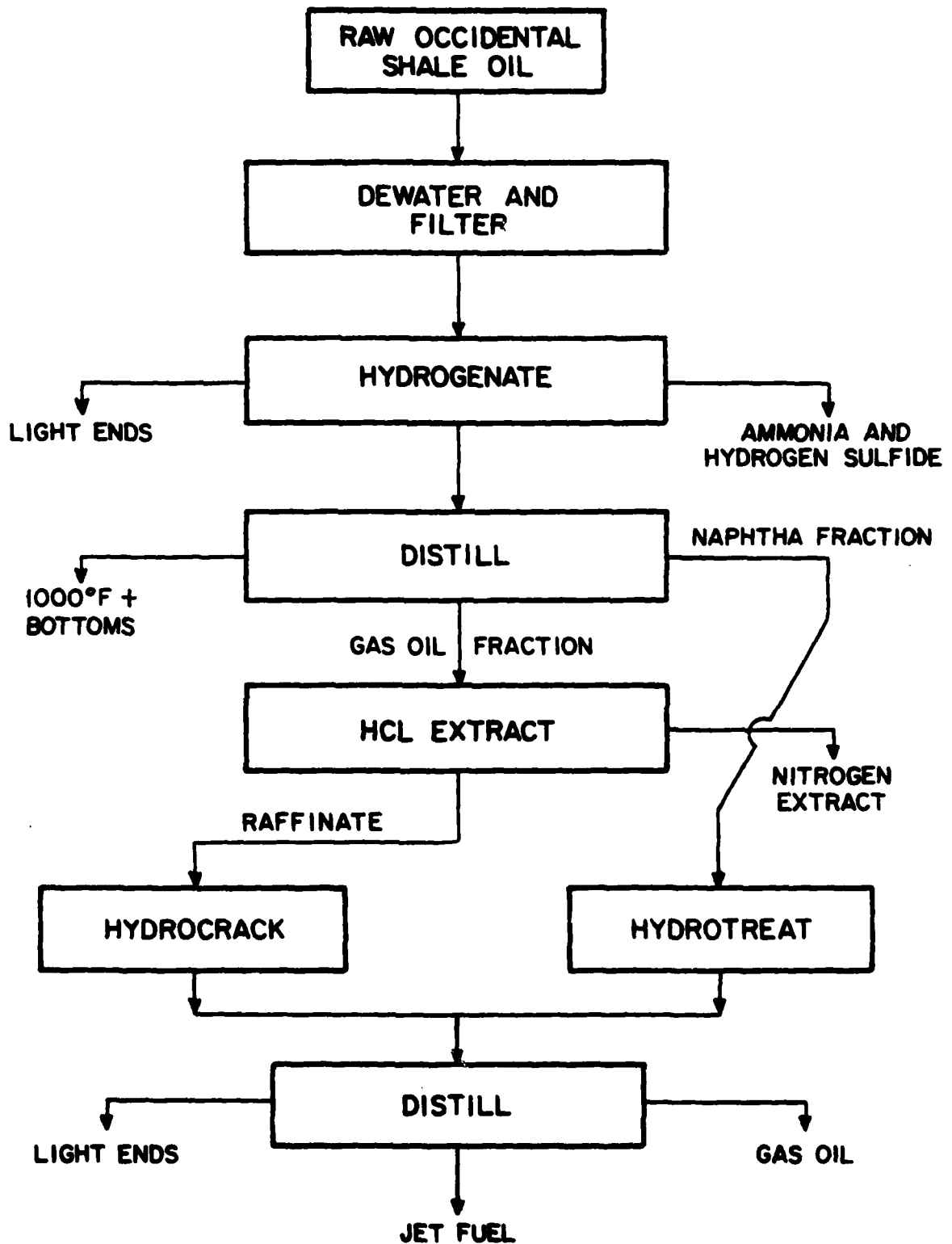


FIGURE 1  
 BLOCK FLOW DIAGRAM FOR PREPARING  
 JET FUELS FROM OCCIDENTAL SHALE OIL

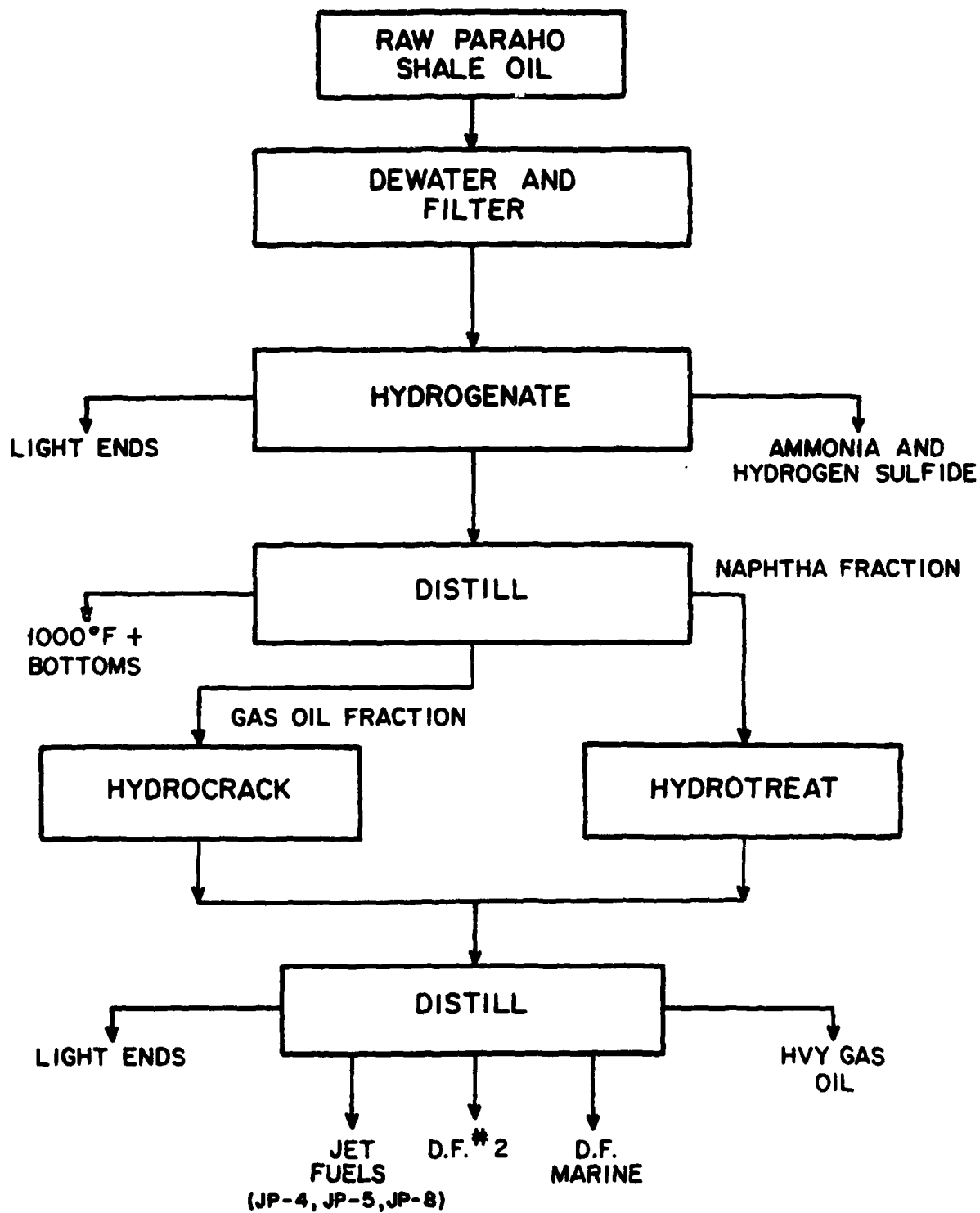


FIGURE 2  
 BLOCK FLOW DIAGRAM FOR PREPARING  
 MILITARY FUELS FROM PARAHO SHALE OIL



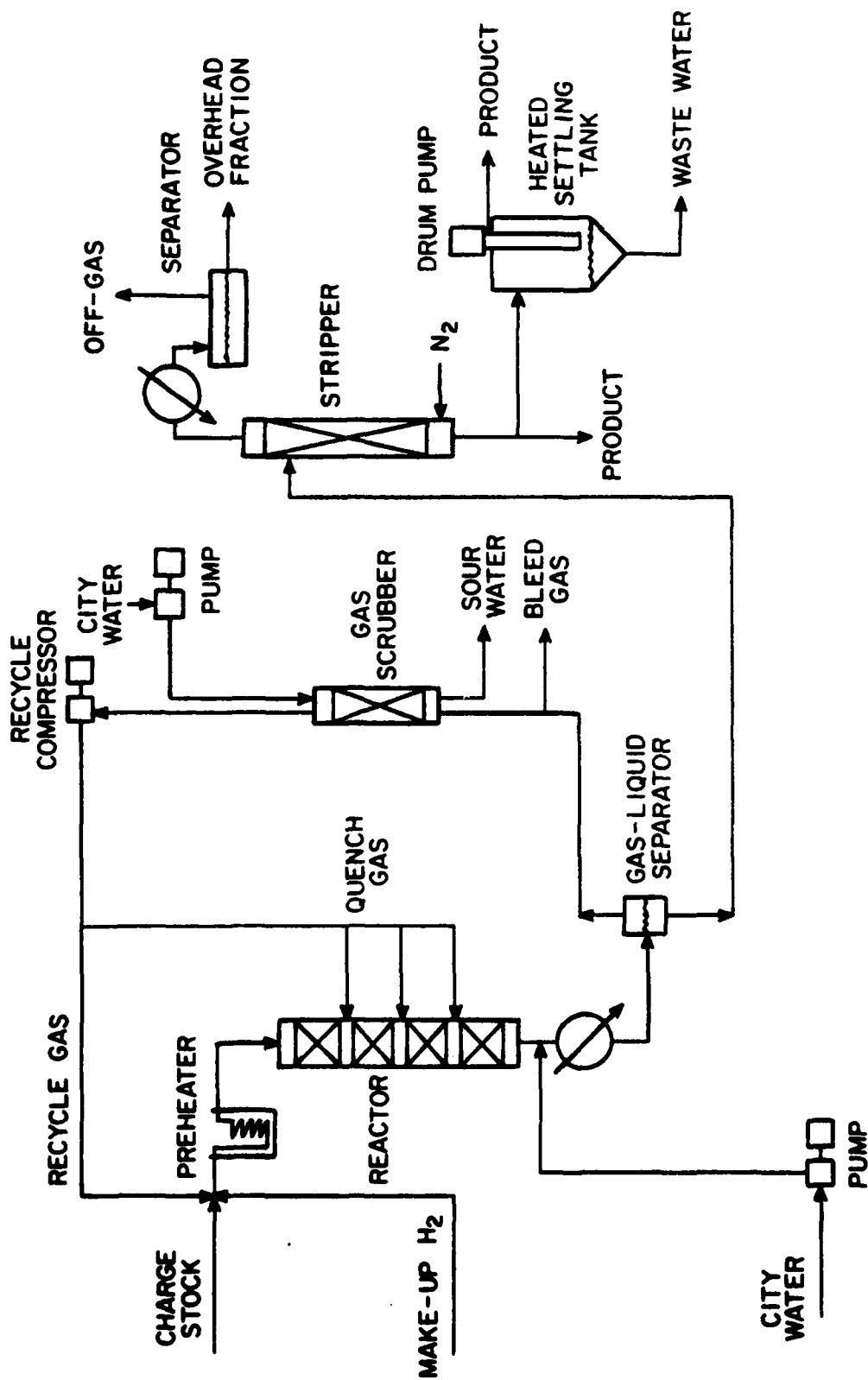


FIGURE 3  
SIMPLIFIED FLOW DIAGRAM OF ADIABATIC  
PILOT PLANT

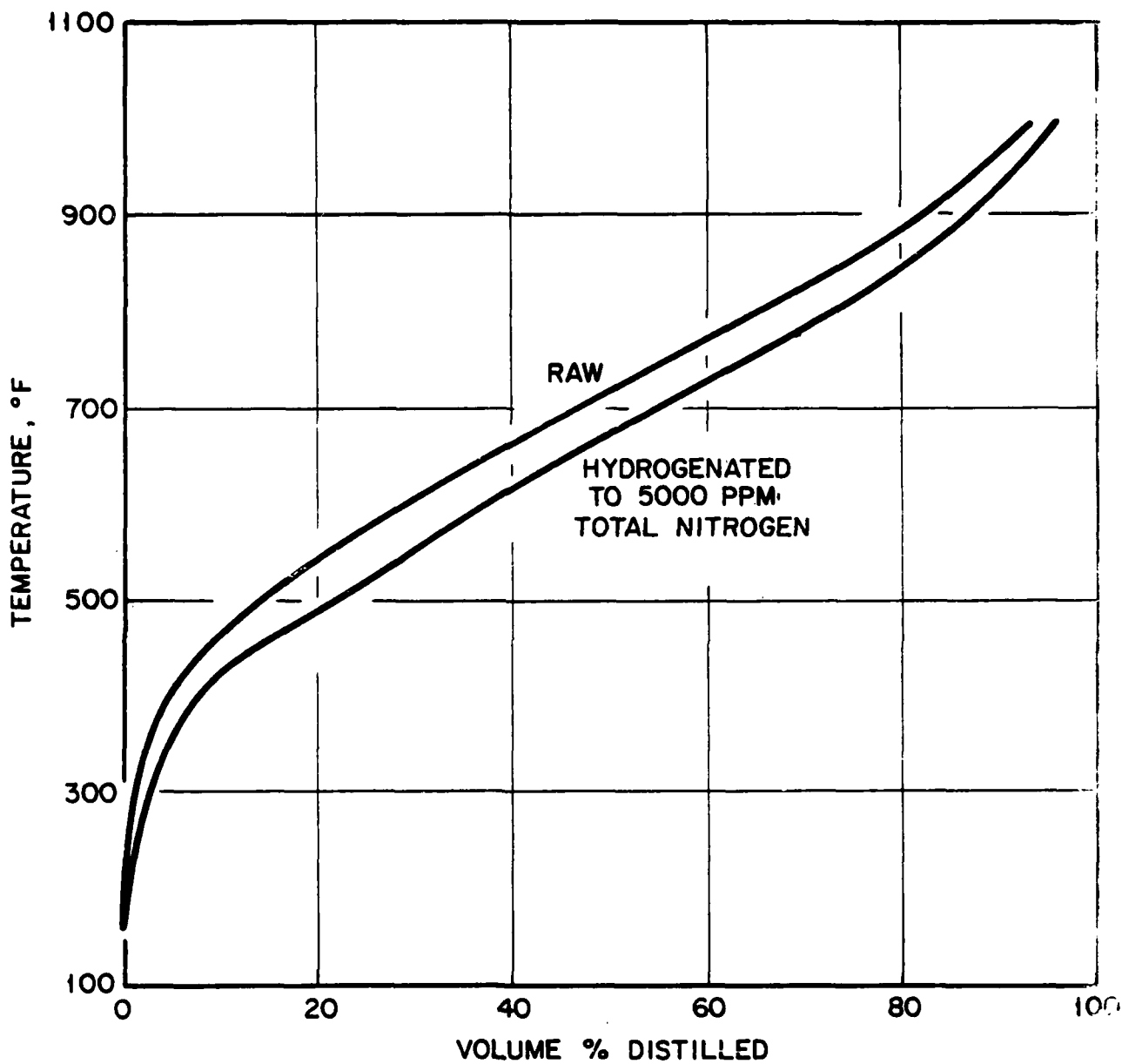


FIGURE 4  
SMOOTHED TBP DISTILLATION CURVES FOR RAW  
AND HYDROGENATED OCCIDENTAL SHALE OIL

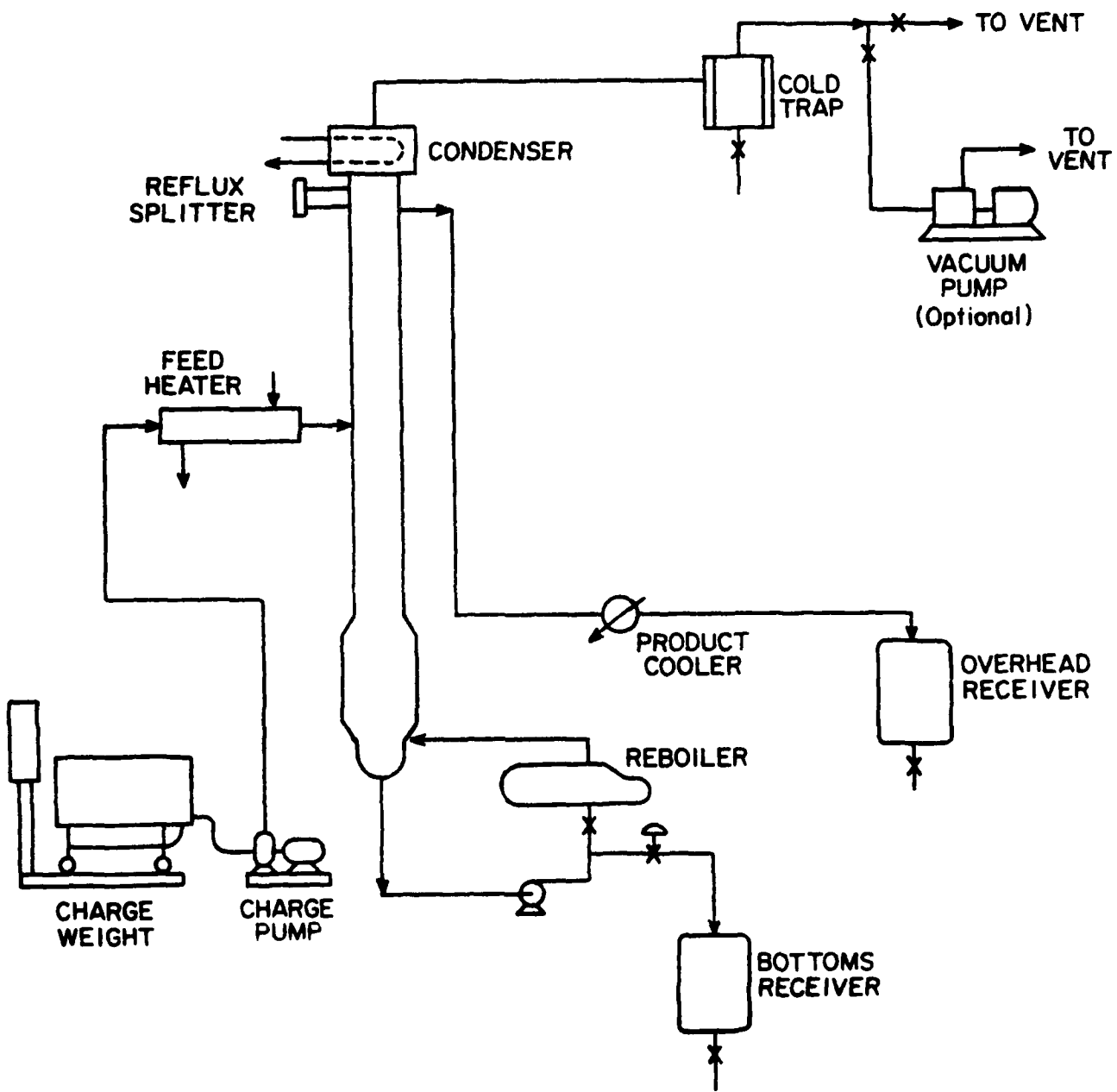


FIGURE 5  
 SCHEMATIC FLOW DIAGRAM OF DISTILLATION  
 PILOT PLANTS

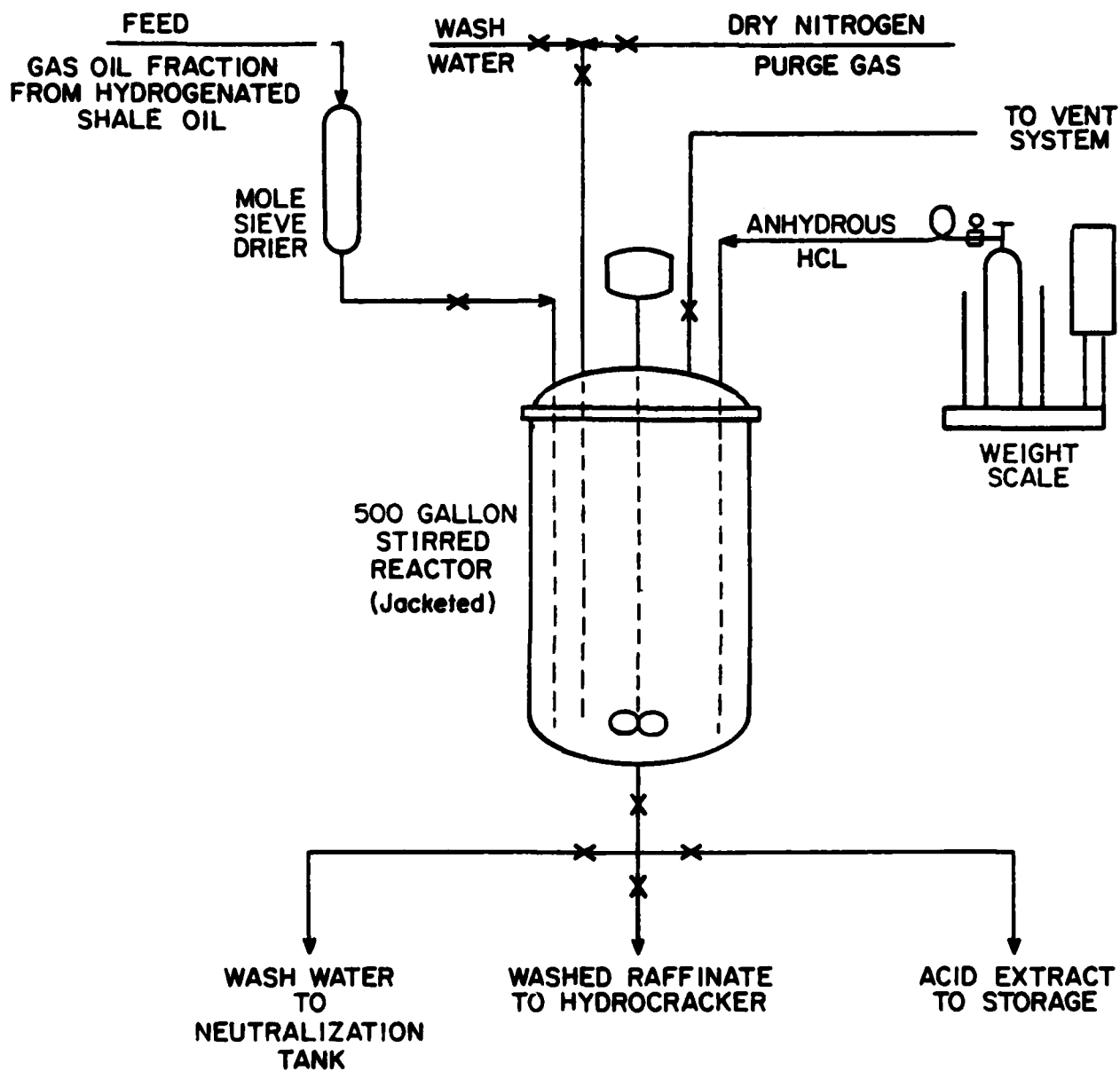


FIGURE 6  
SIMPLIFIED SCHEMATIC FLOW DIAGRAM OF BATCH HCL  
EXTRACTION UNIT

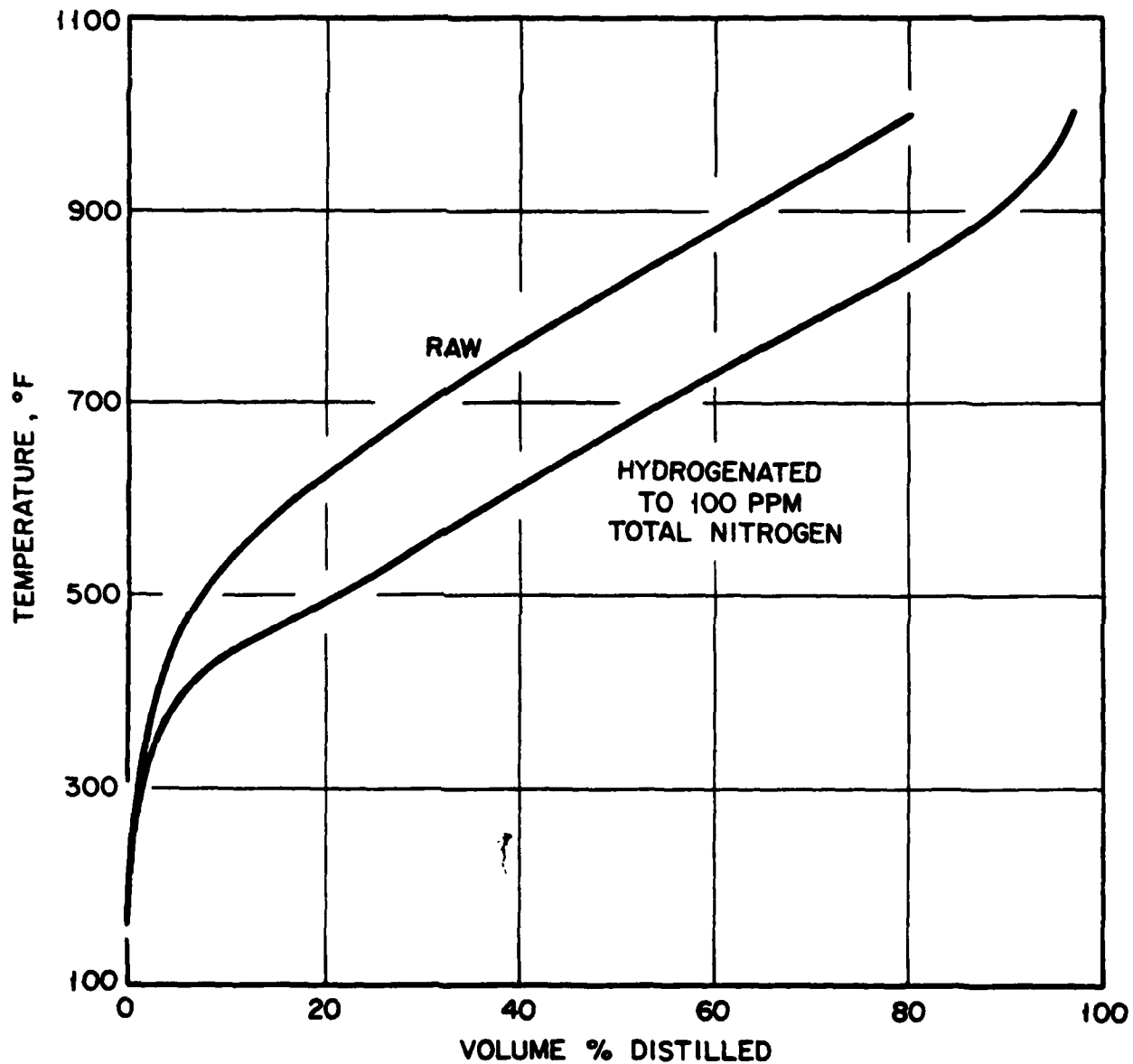


FIGURE 7  
SMOOTHED TBP DISTILLATION CURVES FOR RAW  
AND HYDROGENATED PARAHO SHALE OIL

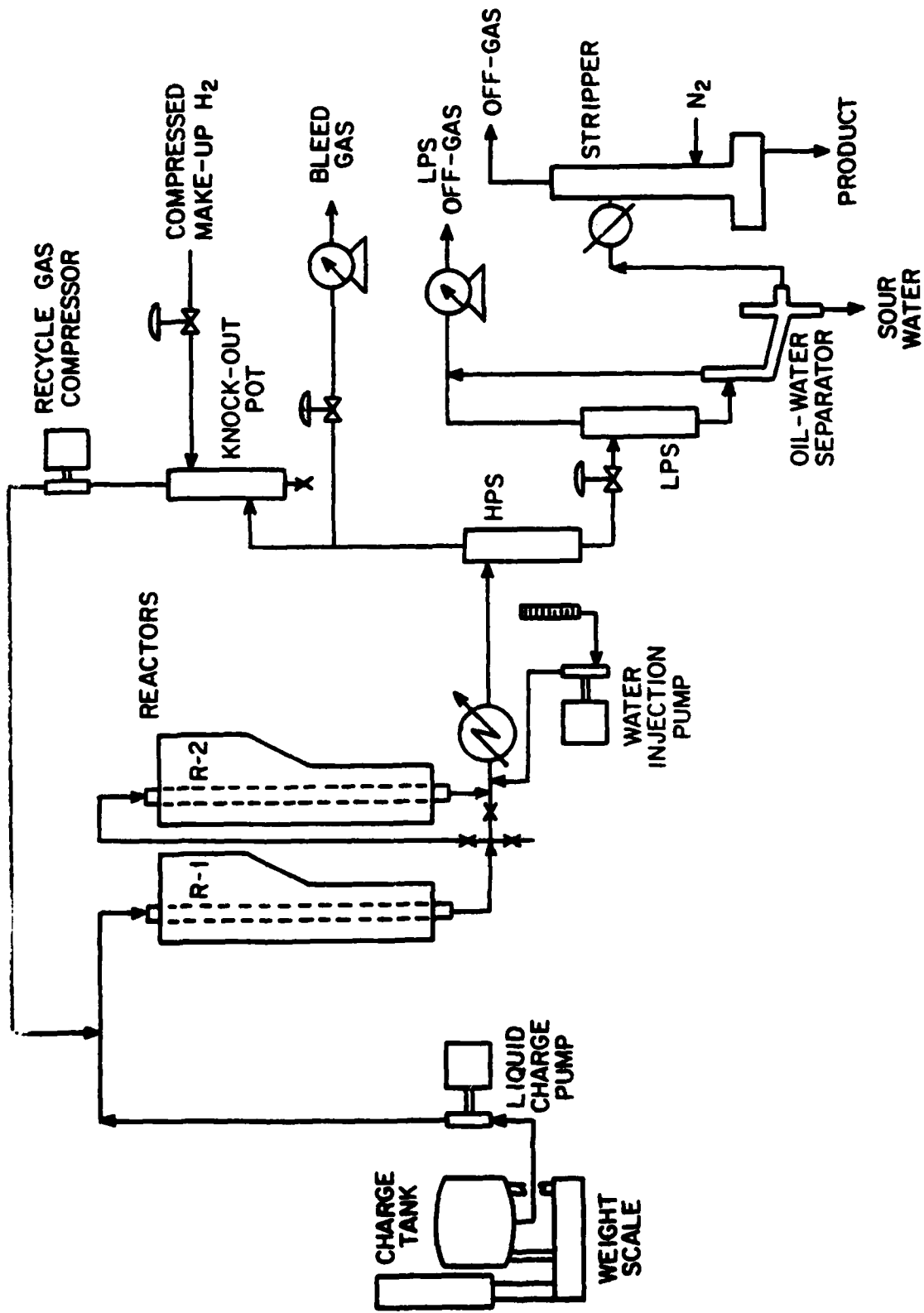


FIGURE 8  
SIMPLIFIED FLOW DIAGRAM OF ISOTHERMAL  
PILOT PLANT

**TABLE 1**  
**INSPECTIONS AND ANALYSES OF RAW OCCIDENTAL SHALE OIL**

<b>Inspection Data</b>	
API @ 60°F	23.0
Specific Gravity 60/60	0.9160
<b>Viscosities, KV</b>	
cs @ 100°F	32.3
cs @ 210°F	4.82
<b>Distillation, °F (D2887)</b>	
IBP	296
10 Vol. %	459
30 "	558
50 "	649
70 "	768
90 "	876
FBP	1071
Asphaltenes, Wt. %	2.4
<b>Chemical Composition Data, Wt. %</b>	
Carbon	84.82
Hydrogen	12.04
Oxygen	1.18
Nitrogen (Total)	1.46
(Basic)	0.81
Sulfur	0.62
Arsenic, ppm	33

TABLE 2

MATERIAL BALANCE - RAW SHALE OIL HYDROTREATER  
ADIABATIC PILOT PLANT RUN F2-569; HOURS 8.0

Reactor Conditions

Average Catalyst Bed Temperature, °F	687
Total Pressure, psig	1640
Liquid Hourly Space Velocity, v/hr/v	1.02
Recycle Gas Rate, SCF/Bbl	3000

Materials Charged

Hydrogen, g	987
Occidental Shale Oil, g	56614

Materials Produced

Gaseous products, g	515
Liquid products, g	54955
Wash Water products, g	863

Material Balance

Wt. % Closure	97.8
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Hydrogen Usage

Consumed Chemical, SCF/Bbl	865
Bleed gas, SCF/Bbl	118



TABLE 3

MATERIAL BALANCE - RAW SHALE OIL HYDROTREATER  
 PRODUCT GAS BREAKDOWN

Product Stream Breakdown - Stripper Gas

<u>Component</u>	<u>Mol. %</u>	<u>Moles</u>	<u>Grams</u>
H <sub>2</sub>	35.60	22.92	45.84
H <sub>2</sub> S	1.90	1.22	41.59
C <sub>1</sub>	3.20	2.06	31.97
C <sub>2</sub>	2.30	1.42	42.50
C <sub>3</sub>	1.5	0.97	42.50
C <sub>14</sub>	0.40	0.26	14.94
C <sub>4</sub>	1.00	0.64	37.34
C <sub>15</sub>	1.50	0.97	69.54
C <sub>5</sub>	0.10	0.06	4.64
C <sub>6</sub>	0.90	0.58	49.84
C <sub>7+</sub>	0.00	0.00	0.00
TOTAL	48.30	31.10	381.69

Product Stream Breakdown - Analytical Gas

<u>Component</u>	<u>Mol. %</u>	<u>Moles</u>	<u>Grams</u>
H <sub>2</sub>	95.60	43.94	87.88
H <sub>2</sub> S	0.00	0.00	0.00
C <sub>1</sub>	2.20	1.01	16.18
C <sub>2</sub>	0.70	0.32	9.65
C <sub>3</sub>	0.20	0.09	4.04
C <sub>14</sub>	0.10	0.05	2.67
C <sub>4</sub>	0.10	0.05	2.67
C <sub>15</sub>	0.20	0.09	6.62
C <sub>5</sub>	0.00	0.00	0.00
C <sub>6</sub>	0.10	0.05	3.95
C <sub>7+</sub>	0.00	0.00	0.00
TOTAL	99.20	45.59	133.65

TABLE 3 (Cont'd.)

Product Stream Breakdown - Gas Scrubber Water

<u>Component</u>	<u>Wt. Frac.</u>	<u>Grams</u>	<u>Moles</u>	<u>Mole %</u>
H <sub>2</sub> S	0.0074	329.2	9.1	0.0036
NH <sub>3</sub>	0.0120	533.9	31.4	0.0125
TOTAL	1.0192	45346.2	2511.8	1.0000

Summation of Product Stream Components

<u>Component</u>	<u>Grams</u>
H <sub>2</sub>	133.72
H <sub>2</sub> S	370.84
C <sub>1</sub>	49.14
C <sub>2</sub>	52.15
C <sub>3</sub>	46.54
C <sub>14</sub>	17.60
C <sub>4</sub>	40.01
C <sub>15</sub>	76.16
C <sub>5</sub>	4.64
C <sub>6</sub>	53.79
C <sub>7+</sub>	0.00
NH <sub>3</sub>	533.90
TOTAL	1378.44

TABLE 4

INSPECTIONS AND ANALYSES OF RAW AND HYDROTREATEDOCCIDENTAL SHALE OIL

	<u>Dewatered &amp; Filtered</u> <u>Feed</u>	<u>Hydrotreated</u> <u>Product</u>
API Gravity @ 60°F	23.0	30.9
Distillation, °F (D1160)		
IBP	413	169
10 vol. %	511	422
30	613	600
50	730	679
70	834	797
90	959	933
EP	-	1000
Recovery	90	97
Residue	10	2
Saturates, wt. %	31.5	63
Aromatics	42.5	37
Polar Aromatics	23.6	--
Asphaltenes	2.4	--
Chemical Analyses, wt. %		
Carbon	84.82	86.25
Hydrogen	12.04	12.77
Sulfur, ppm	6200	159
Nitrogen, total	1.46	0.51
basic	0.81	0.339
Oxygen	1.18	0.11
Arsenic, ppm	33	<1
Ash, wt. %	< 0.005	Nil

TABLE 5  
INSPECTIONS AND ANALYSES OF FRACTIONS FROM HYDROGENATED  
OCCIDENTAL SHALE OIL (5000 PPM TOTAL NITROGEN)

Units: Continuous Atmospheric and Vacuum Stills.  
(Main Hydrotreater Product)

	<u>Naphtha</u>	<u>Lt. Gas Oil</u>	<u>Hvy. Gas Oil</u>	<u>Bottoms</u>
Yield, Vol. %	14.8	12.2	63.4	9.6 <sup>1/</sup>
Boiling Range, °F	1-450	450-535	535-1000	1000+ Bot.
API Gravity @ 60°F	44.0	37.5	28.9	16.0
Specific Gravity	0.8065	0.8374	0.8822	0.9593
Distillation, °F				
IBP	296	460	535	
10 vol. %	330	472	615	
30	365	477	670	
50	385	481	740	
70	403	487	817	
90	420	497	940	
EP	448	536	1000	
Recovery	98	98	98	
Residue	1	1	1	
Viscosity, cs @ 100°F	-	-	19.7	-
210°F	-	-	3.62	1.660
Pour Point, °F	-	-	+43	+110
Ramsbottom Carbon				
Residue, wt. %	-	-	-	3.44
Aromatics, vol. %	19.5	30	41 wt.	-
Saturates, vol. %	-	-	39	-
Chemical Analyses, wt. %				
Carbon	85.91	86.16	86.25	86.39
Hydrogen	14.04	13.40	13.17	12.33
Sulfur, ppm	46	51	72	1220
Nitrogen, total	0.08	0.36	0.52	0.79
basic	0.06	0.28	0.21	0.48
Oxygen	0.03	0.17	0.14	0.84

<sup>1/</sup> Includes hold-up drained from distillation column.

TABLE 6

INSPECTIONS AND ANALYSES OF HYDROTREATED NAPHTHA

API Gravity	42.2
Distillation, °F (D-86)	
IBP	300
20 vol. %	402
50 vol. %	450
80 vol. %	511
95 vol. %	527
End Point	539
Sulfur ppm	0.4
Nitrogen (total), ppm	1.5
Aromatics, vol. %	17.5
Bromine No.	0.5
Flash, °F	125
JFTOT Test	
Tube Code Rating	0
Δ P mm Hg	0
Copper Strip	1A
Aniline Point, °F	152

TABLE 7  
INSPECTIONS AND ANALYSES OF HCl RAFFINATE (1st RUN)

ASTM D-1160 Distillation, °F	
IBP	474
5	522
10	551
20	577
30	608
40	539
50	672
60	708
70	744
80	783
90	817
95	840
Total Nitrogen, ppm	491
Basic Nitrogen, ppm	227
Sulfur, ppm	471
Carbon, wt. %	86.45
Hydrogen, wt. %	13.52
Oxygen, wt. %	0.20
Chlorine, wt. %	0.03
Specific Gravity, 60/60	0.8637
Average Molecular Weight	274

TABLE 7 (Cont'd.)

Viscosity, SUS at 100°F	61.4
Viscosity, SUS at 210°F	34.9
Viscosity Index	78
$n_D^{20}$	1.4785
$d_4^{20}$	0.8624
Aromatics, wt. %	31.3
$n_D^{20}$	1.5316
$d_4^{20}$	0.9454
Average Molecular Weight	267
Saturates	
$n_D^{20}$	1.4611
$d_4^{20}$	0.8315
Average Molecular Weight	282
Weight % of Aromatics	
Monoaromatics	68.0
Diaromatics	20.2
Triaromatics	4.2
Tetra-aromatics	3.2
Volume % of Saturates	
Paraffins	34.8
1-Ring Naphthenes	16.1
2-Ring Naphthenes	17.1
3-Ring Naphthenes	15.5
4-Ring Naphthenes	11.4
5-Ring Naphthenes	5.2

TABLE 8

TYPICAL RESULTS FROM HCl EXTRACTIONS

Feedstock: 975 Gallons of 530-1000°F Boiling Range Gas Oil.  
Fraction From Hydrogenated Occidental Shale Oil.  
(3 Runs)

Extraction Conditions: Temperature, °F 110  
Pressure, psig 8  
HCl Addition time, hours<sup>1/</sup> 4.5  
Anhydrous HCl added,  
wt.% of gas oil 2.25

	<u>Feed</u>	<u>Recovered Raffinate</u>	<u>Recovered Extract</u> <sup>2/</sup>
Yield, wt.%	100	84.8	13.3
API Gravity @ 60°F	28.9	30.6	12.9
Specific Gravity	0.8822	0.8728	0.9800
Nitrogen, Total, ppm	5200	700	3.14 wt.%
Sulfur, ppm	72	30	-
Chlorine, ppm	0	700	750
Aromatics, wt.%	41	33	-
Water Wash Loss, wt.% Feed	-	1.9	-

<sup>1/</sup> Addition time limited by vaporization rate of HCl from cylinder.

<sup>2/</sup> After thermally decomposing a small sample in the laboratory.



TABLE 9

MATERIAL BALANCE - R-2 HYDROCRACKING  
ADIABATIC HYDROCRACKER RUN F2-574; HOURS 8.0

Reactor Conditions

Average Catalyst Temperature, °F	701
Total Pressure, psig	1715
Liquid Hourly Space Velocity, v/hr/v	1.6
Recycle Rate, SCF/Bbl	4000

Materials Charged

Hydrogen, g	987
Heavy gas oil, g	49258

Materials Produced

Gaseous products, g	2276
Liquid products, g	45209

Material Balance

Wt. % Closure	95.0%
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Hydrogen Usage

Consumed Chemically, SCF/Bbl	689
Bleed gas, SCF/Bbl	118

TABLE 10  
 MATERIAL BALANCE - R-2 HYDROCRACKING  
PRODUCT GAS BREAKDOWN  
Product Stream Breakdown - Stripper Gas

<u>Component</u>	<u>Mol. %</u>	<u>Moles</u>	<u>Grams</u>
H <sub>2</sub>	24.80	21.59	43.18
H <sub>2</sub> S	0.00	0.00	0.00
C <sub>1</sub>	0.40	0.35	5.57
C <sub>2</sub>	0.70	0.61	18.28
C <sub>3</sub>	9.00	7.84	344.77
CI <sub>4</sub>	16.50	14.37	833.20
C <sub>4</sub>	5.60	4.88	282.78
CI <sub>5</sub>	6.40	5.57	401.19
C <sub>5</sub>	1.80	1.57	112.83
C <sub>6</sub>	1.50	1.31	112.31
C <sub>7</sub> <sup>+</sup>	0.00	0.00	0.00
TOTAL	66.70	58.07	2154.12

Product Stream Breakdown - Analytical Gas

<u>Component</u>	<u>Mol. %</u>	<u>Moles</u>	<u>Grams</u>
H <sub>2</sub>	96.80	31.32	62.63
H <sub>2</sub> S	0.00	0.00	0.0
C <sub>1</sub>	0.30	0.10	1.55
C <sub>2</sub>	0.20	0.06	1.94
C <sub>3</sub>	0.50	0.16	7.12
CI <sub>4</sub>	0.70	0.23	13.13
C <sub>4</sub>	0.30	0.10	5.63
CI <sub>5</sub>	0.50	0.16	11.65
C <sub>5</sub>	0.20	0.06	4.66
C <sub>6</sub>	0.50	0.16	13.91
C <sub>7</sub> <sup>+</sup>	0.00	0.00	0.00
TOTAL	100.00	32.35	122.23

TABLE 10 (Cont'd)

Summation of Product Stream Components

<u>Component</u>	<u>Grams</u>
H <sub>2</sub>	105.82
H <sub>2</sub> S	0.00
C <sub>1</sub>	7.12
C <sub>2</sub>	20.22
C <sub>3</sub>	351.89
C <sub>14</sub>	846.33
C <sub>4</sub>	288.41
C <sub>15</sub>	412.84
C <sub>5</sub>	117.49
C <sub>6</sub>	126.22
C <sub>7+</sub>	0.00
TOTAL	2276.35

TABLE 11

TYPICAL RESULTS FROM HYDROCRACKING  
HCl RAFFINATE

Feedstock: 530-1000°F. Boiling Range Raffinate From HCl Extraction

Hydrogenation Units: 10 Liter Adiabatic Reactor Pilot Plant

Catalyst: R-1 Ni Mo on Alumina  
R-2 Ni W on Silica Alumina

Operating Conditions:

	<u>R-1</u>	<u>R-2</u>
Avg. Catalyst Bed Temp., °F	760	701

Total Pressure, psig 1700 1700

Liquid Hourly Space Velocity, v/hr/v 1.41/ 1.61/

Recycle Gas Rate, SCF/B 4000 4000

	<u>Feedstock</u>	<u>R-1</u>		<u>R-2 Liquid Products</u>	
		<u>Product</u>	<u>C<sub>4</sub> - 300°F</u>	<u>300°F + Bottoms</u>	
Yield, vol.% Feed	100	101	<u>47</u>	64	
API Gravity @ 60°F	30.5	31.6	71.0	38.7	
Aromatics, wt.%	33.1	23.5	1 vol.	28	
Sulfur, ppm	30	2	1	2	
Nitrogen, Total, ppm	700	10	<1	6	
Chlorine, ppm	700	0	0	0	

1/ Throughput limited by feed preheater capacity.

TABLE 12

TYPICAL PROPERTIES OF INTERMEDIATE FRACTIONS  
FROM OCCIDENTAL SHALE OIL

	Hydrotreated Naphtha	Light Hydrocrackate	Hydrocracked Bottoms
Volume, Gallons	420	251	340
API Gravity @ 60°F	42.3	71.0	38.7
Distillation, °F			
IBP	306	84	310
10	371	129	334
50	447	204	410
90	499	257	577
EP	531	300	658
Freeze Point, °F	-31	-94	15
Flash Point, °F	125	<80	130
Aromatics, vol.%	15	1	28 wt.%
Sulfur, ppm	<1	1	2
Nitrogen, Total, ppm	3	<1	6
Octane Numbers			
F-1 Clear	-	69.5	-
F-2 Clear	-	67.9	-

TABLE 13

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLES OF JP-4 FROM RAW OCCIDENTAL SHALE OIL

	<u>Spec.</u>	<u>X190-223</u>
Aromatics, vol. % max.	25.0	2.6
Olefins, vol. % max.	5.0	2.2
Sulfur, wt. % max.	0.40	<1.0 ppm
Nitrogen (total), ppm	NA	<1.0
ASTM D-86 Distillation, °F		
IBP	Report	142
20 vol. %, max.	293	232
50 vol. %, max.	374	295
90 vol. %, max.	473	446
EP, max.	518	478
Gravity, °API min./max.	45.0/57.0	54.8
RVP (100°F), psi min./max.	2.0/3.0	2.6
Freeze point, °F max.	-72	-81
Net Ht. of comb., min.	18,400	18,846
Copper strip corrosion, max.	1B	1A
Thermal Stability		
Pressure Drop, mm Hg, max.	25	0
Heater Deposit, max.	3	0
Hydrogen, wt. % min.	13.6	14.46
Carbon, wt. %	NA	85.14
Oxygen, wt. %	NA	< 0.1

TABLE 14

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLES OF JP-5 FROM RAW OCCIDENTAL SHALE OIL

	<u>Spec.</u>	<u>X190-224</u>
Aromatics, vol. % max.	25.0	15.9
Olefins, vol. % max.	5.0	2.2
Sulfur, wt. % max.	0.40	< 1.0 ppm
Nitrogen (total), ppm	NA	< 1.0
ASTM D-86 Distillation, °F		
IBP	Report	345
10 vol. % max.	401	373
20 vol. % max.	Report	385
50 vol. % max.	Report	417
90 vol. % max.	Report	461
EP, max.	554	487
Gravity, °API	36.0/48.0	43.4
Flash Point, °F min	140	145
Freeze Point, °F max	-51	-56
Net Ht of comb., min.	18,300	18,596
Copper strip corrosion, max.	1B	1B
Thermal Stability		
Pressure drop, mm Hg max.	25	0
Heater deposit, max.	3	0
Hydrogen, wt. % min.	13.5	14.02
Carbon, wt. %	NA	85.98
Oxygen, wt. %	NA	< 0.1

TABLE 15

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLES OF JP-8 FROM RAW OCCIDENTAL SHALE OIL

	<u>Spec.</u>	<u>X190-225</u>
Aromatics, vol. % max.	25.0	11.2
Olefins, vol. % max.	5.0	1.4
Sulfur, wt. % max.	0.40	< 1.0 ppm
Nitrogen (total), ppm	NA	< 1.0
ASTM D-86 Distillation, °F		
IBP	Report	324
10 vol. %, max.	401	346
20 vol. %, max.	Report	360
50 vol. %, max.	Report	397
90 vol. %, max.	Report	452
EP, max.	572	480
Gravity, °API min./max.	37/51	45.8
Flash Point, °F min.	100	128
Freeze Point, °F max.	-58	-61
Net Ht. of Comb., min.	18,400	18,663
Copper strip corrosion, max.	1B	1B
Thermal Stability		
Pressure Drop, mm Hg max.	25	0
Heater Deposit, max.	3	0
Hydrogen, wt. % min.	13.6	14.12
Carbon, wt. %	NA	85.88
Oxygen, wt. %	NA	< 0.1



TABLE 16

INSPECTIONS AND ANALYSES OF JET FUEL INHIBITORS

<u>Inhibitor</u>	<u>Density, lb/gal</u>	<u>Concentration, max.</u>
UCAR 500/Anti Icing	--	0.12 Vol. %
DCI 4A/Anti-Corrosion	7.8	3 lb/1000 bbl.
A0995/Anti-oxidant <sup>1/</sup>	7.6 lb/gal	4 lb/1000 bbl.

<sup>1/</sup> A0995 is 60 vol. % active ingredient, 10 vol. % toluene. Density is of the active ingredient.

**TABLE 17**  
**MATERIAL BALANCE - RAW PARAHO SHALE OIL HYDROTREATER**

Target Nitrogen Level, ppm	<u>100</u>
Mass Balance, wt. % Reactants	95.8
H <sub>2</sub> Consumption, SCF/Bbl Charge	2370
Products (Corrected to 100% wt. Basis), wt. % Reactants	
H <sub>2</sub> O	1.07
H <sub>2</sub> S	0.85
NH <sub>3</sub>	2.29
C <sub>1</sub> -C <sub>3</sub>	2.15
C <sub>4</sub> -C <sub>6</sub>	1.82
<u>Liquid Product</u>	<u>91.82</u>
TOTAL	100.00

TABLE 18  
INSPECTIONS AND ANALYSES OF RAW AND  
HYDROTREATED PARAHO SHALE OIL

	<u>Dewatered and Filtered Feed</u>	<u>Hydrotreated Product</u>
API Gravity @ 60°F	20.6	37.0
Distillation, °F	D1160	TBP
IBP	133	165
10 vol. %	508	428
50	798	643
90	1057	940
EP	1065	1000
Recovery	95	97
Chemical Analyses, wt. %		
Carbon	83.83	85.69
Hydrogen	11.72	13.65
Sulfur, ppm	7500	14
Nitrogen, Total	2.13	100 ppm
Arsenic, ppm	34	<1

TABLE 19

INSPECTIONS AND ANALYSES OF FRACTIONS FROM HYDROGENATED  
PARAHO SHALE OIL (100 PPM TOTAL NITROGEN)

	<u>Naphtha</u>	<u>Lt. Gas Oil</u>	<u>Gas Oil</u>	<u>Hvy. Gas Oil</u>
Yield, vol. %	22.2	8.5	22.0	45.3
Boiling Range, °F (TBP)	1-490	490-540	540-650	650-1000
API Gravity @ 60°F	43.1	37.9	35.9	30.8
Aromatics, %	15 vol.	20 vol.	22 wt.	28 wt.
Elemental Analyses, ppm				
Nitrogen, Total	26	27	40	163
Nitrogen, Basic	19	18	28	--
Sulfur	<1	<1	4	21

TABLE 20

FINAL NAPHTHA HYDROTREATING

FEEDSTOCK: IBP-490°F Fraction From Shale Oil Hydrogenated to  
100 ppm Total Nitrogen

Catalyst: Ni Mo on Alumina

Operating Conditions:

Average Catalyst Bed Temperature °F	730
Total Pressure, psig	1500
Liquid Hourly Space Velocity, v/hr/v	2.0
Hydrogen to Oil Ratio, SCF/B	5000

	<u>Feedstock</u>	<u>Stabilized Liquid Product</u>
API Gravity @ 60°F	43.1	43.6
Aromatics, vol. %	15	4
Sulfur, ppm	1	<1
Nitrogen, Total, ppm	26	1

TABLE 21

TYPICAL HYDROCRACKING RESULTS

Feedstock: 650-1000°F Fraction From Paraho Shale Oil Hydrogenated to 100 ppm Total Nitrogen  
 Catalyst: R-1: Ni Mo on Alumina; R-2: Ni W on Silica Alumina

Operating Conditions:

	<u>R-1</u>	<u>R-2</u>
Avg. Catalyst Bed Temp., °F	750	750
Total Pressure, psig	1700	1700
Liquid Hourly Space Velocity, v/hr/v	1.0	2.0
Hydrogen to Oil Ratio, SCF/B	5000	5000

	<u>Feedstock</u>	<u>R-1 Product</u>	<u>R-2 Liquid Products</u>		
			<u>C<sub>A</sub>-310°F</u>	<u>310-550°F</u>	<u>550-675°F</u>
Yield, vol.% Feed	100	101	46	29	24
API Gravity @ 60°F	30.8	31.6	75.2	43.7	38.1
Sulfur, ppm	21	2	1	2	3
Nitrogen, Total, ppm,	163	<1	<1	<1	<1

TABLE 22

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLE OF JP-4 FROM RAW PARAHO SHALE OIL

	<u>Spec.</u>	<u>X190-76</u>
Aromatics, vol. % max.	25.0	5.2
Olefins, vol. % max.	5.0	1.4
Sulfur, wt. % max.	0.40	0.0003
Nitrogen (total), ppm	NA	< 0.1
ASTM D-86 Distillation, °F		
IBP	Report	153
20 vol. %, max.	293	214
50 vol. %, max.	374	279
90 vol. %, max.	473	430
EP, max.	518	498
Gravity, °API min/max.	45.0/57.0	56.7
RVP (100°F), psi min/max.	2.0/3.0	2.7
Freeze point, °F	-72	-80
Net Ht. of comb., min.	18,400	18,794
Copper strip corrosion, max	1B	1B
Thermal Stability		
Pressure Drop, mm HG, max.	25	0
Heater Deposit, max.	3	0
Hydrogen, wt. % min.	13.6	14.4
Carbon, wt. %	NA	85.4
Oxygen, wt. %	NA	<0.1

TABLE 23

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLE OF JP-5 FROM RAW PARAMO SHALE OIL

	<u>Spec.</u>	<u>X190-76</u>
Aromatics, vol. % max.	25.0	23.8
Olefins, vol. % max.	5.0	1.7
Sulfur, wt. % max.	0.40	0.0003
Nitrogen (total), ppm	NA	<0.1
ASTM D-86 Distillation, °F		
IBP	Report	335
10 vol. % max.	401	365
20 vol. % max.	Report	375
50 vol. % max.	Report	404
90 vol. % max.	Report	443
EP, max.	554	475
Gravity, °API min/max.	36.0/48.0	47.1
Flash Point, °F min.	140	144
Freeze Point, °F max.	-51	-69
Net Ht. of comb., min.	18,300	18,610
Copper strip corrosion, max.	1B	1B
Thermal Stability		
Pressure drop, mm HG max.	25	1
Heater deposit, max.	3	0
Hydrogen, wt. % min.	13.5	13.5
Carbon, wt. %	NA	86.4
Oxygen, wt. %	NA	0.2



TABLE 24

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLE OF JP-8 FROM RAW PARAHO SHALE OIL

	<u>Spec.</u>	<u>X190-78</u>
Aromatics, vol. % max.	25.0	23.6
Olefins, vol. % max.	5.0	1.3
Sulfur, wt. % max.	0.40	0.003
Nitrogen (total), ppm	NA	<0.1
ASTM D-86 Distillation, °F		
IBP	Report	306
10 vol. %, max.	401	333
20 vol. %, max.	Report	345
50 vol. %, max.	Report	385
90 vol. %, max.	Report	450
EP, max.	572	489
Gravity, °API min/max.	37/51	43.7
Flash Point, °F min.	100	118
Freeze Point, °F max.	-58	-80
Net Ht. of comb., min.	18,400	18,520
Copper strip corrosion, max.	1B	1B
Thermal Stability		
Pressure Drop, mm Hg max.	25	0
Heater Deposit, max.	3	0
Hydrogen, wt. % min.	13.6	13.7
Carbon, wt. %	NA	86.3
Oxygen, wt. %	NA	<0.1

TABLE 25

INSPECTIONS AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLE OF DIESEL FUEL-2 FROM RAW PARAHO SHALE OIL

<u>Diesel Fuel Properties</u>	<u>Specification Grade DF-2 Conus.</u>	<u>X190-79</u>
Gravity, °API	Report	40.4
Flash Point, °F	125	146
Cloud Point°, °F max.	--	-2
Pour Point°, °F max.	Report	-10
Kinematic viscosity @ 100°F, cSt	2.0-4.3	2.40
ASTM D-86 Distillation, °F		
IBP		344
50% evaporated	Report	494
90% evaporated, max.	640	575
EP, max.	700	594
Sulfur, wt. % max.	0.50	1 ppm
Nitrogen, ppm	--	1
Copper strip corrosion, 3 hrs. @ 122°F, max.	3	2A
Cetane Index, min.	45	57
Hydrogen, wt. %	--	13.8
Carbon, wt. %	NA	85.9
Oxygen, wt. %	NA	0.2

\* Varies with geographical and seasonal requirements.

TABLE 26

INSPECTION AND ANALYSES OF PILOT PLANT PRODUCTION  
SAMPLE OF DIESEL FUEL MARINE FROM RAW PARAHIO SHALE OIL

	<u>Spec.</u>	<u>X190-80</u>
Gravity, °API	Record	39.4
Flash Point, °F min	140	255
Cloud Point, °F max.	30	+14
Pour Point, °F max.	20	-2
Kinematic viscosity @ 100°F, cSt	1.8-4.5	3.73
ASTM D-86 Distillation, °F		
IBP	--	492
50%	Record	538
90%	675	580
EP, max.	725	601
Sulfur, wt. % max.	1.00	0.003
Nitrogen, ppm	--	1
Copper strip corrosion at 212°F, max.	1	1B
Carbon, wt. %	--	86.0
Hydrogen, wt. %	--	14.0
Oxygen, wt. %	--	<0.1
Cetane Index, min.	45	58