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DOE/ET/10532-T5
(DE82013235)

**REFINING AND UPGRADING OF SYNFUELS FROM COAL AND
OIL SHALES BY ADVANCED CATALYTIC PROCESSES**

**Sixth Interim Report Task 9: Hydrotreating 400°F+ SRC-II Oil for
Biological Studies**

By
R. F. Sullivan

April 1982
Date Published

Work Performed Under Contract No. AC22-76ET10532

Chevron Research Company
Richmond, California

U. S. DEPARTMENT OF ENERGY

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DOE/ET/10532--T5

DE82 013235

DOE/ET/10532-T5

(FE-2315-74)

(DE82013235)

Distribution Category UC-90d

REFINING AND UPGRADING OF SYNFUELS
FROM COAL AND OIL SHALES
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Sixth Interim Report
Task 9: Hydrotreating
400°F+ SRC-II Oil for
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R. F. Sullivan

CHEVRON RESEARCH COMPANY
Richmond, California

Date Published April 1982

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Under Contract No. DE-AC22-75ET10532
(Formerly EF-76-C-01-2315)

Abstract

400°F+ SRC-II oil derived from Pittsburgh Seam coal was hydrotreated to provide DOE samples for subsequent biological testing at the Oak Ridge National Laboratory. Samples containing about 500 ppm nitrogen, 2000 ppm nitrogen, and 5000 ppm nitrogen were prepared. These samples do not represent finished products, but conditions were selected to provide a wide range of processing severities. The feedstock was somewhat higher boiling and more difficult to hydrotreat than another 400°F+ SRC-II oil studied previously.

Encl. - See Index of Enclosures

I. Summary

The object of work under this task was to prepare samples of hydro-processed 400°F+ SRC-II oil for subsequent biological testing by DOE at the Oak Ridge National Laboratory. Sample preparation conditions were purposely selected to obtain a broad range of processing severities. These samples, therefore, do not represent specification products nor are the processing conditions necessarily representative of conditions that might exemplify commercial refining conditions. With this particular synthetic oil, DOE requested that we obtain product at three hydrotreating severities defined by the nitrogen in the whole liquid product, as follows:

<u>Hydrotreatment Severity Level</u>	<u>Whole Hydrotreated Liquid Product Nitrogen, ppm</u>
Low	5000
Medium	2000
High	500

The products were made at 750°F and 2250 psia hydrogen using Chevron commercial catalysts. Space velocity was adjusted to produce the desired product nitrogen. The particular SRC-II oil used in this study was found to be harder to process than a 1978 sample of 400°F+ oil derived from the same coal. The newer feedstock was somewhat higher boiling and contained more hot heptane insolubles than the previous oil.

II. Contract Objective and Status

The objective of this program is to determine the feasibility and to estimate the costs of hydroprocessing synthetic crude feedstocks to distillate fuels using modern commercial petroleum processing technology. Tests will be conducted only to the extent needed to enable making reasonable estimates of commercial plant performance and only to the extent a commercial plant is feasible using presently existing technology, subject to the mutual agreement of DOE and Chevron Research. Tests will be made for each whole synthetic oil and, where appropriate, for the fractions derived therefrom. Tests will not be carried out for processes which can be reliably estimated.

The feedstocks under this contract and the status of the work on each are as follows:

1. Work on the Paraho shale is complete and reported in the first interim report, FE-2315-25. Refining cost for shale oil is updated in the fourth interim report, FE-2315-50.
2. Work on SRC-I is complete and reported in the second interim report, FE-2315-45.
3. Work on SRC-II process product derived from Pittsburgh Seam coal is complete and reported in the third interim report, FE-2315-47.
4. Work on H-Coal process product derived from Illinois No. 6 is complete and reported in the fifth interim report, FE-2315-61.
5. Work on H-Coal process product derived from Wyodak coal is complete. Results are also reported in FE-2315-61.
6. The pilot plant program with EDS process product from Big Brown Texas lignite is complete; analysis of the results is in progress.
7. At the request of DOE, we added Task 9 to the contract. In this task, we hydrotreated 400°F+ SRC-II oil at several severities to supply DOE samples for biological studies. The pilot plant program is complete and the product has been sent to Oak Ridge National Laboratory. This report describes the work under Task 9.
8. At the request of DOE, we added Task 10 to the contract. In this task, we will hydrotreat three additional coal-derived oils to supply DOE with product for biological studies. The first two oils (from the EDS liquefaction process and the H-Coal process) have been received, were analyzed, and are being processed.

III. Feedstock Preparation and Analyses

A. Analysis of SRC-II Oil Components

At the request of the DOE Technical Representative, the following samples of SRC-II process product were sent to Chevron from the Solvent Refined Coal Pilot Plant of the Pittsburg and Midway Coal Mining Company, Du Pont, Washington:

Sample No. 2263 - Two drums of naphtha (Chevron Identification NA 2553 WOW 4519)

Sample No. 2264 - Two drums of middle distillate (Chevron UN 1137 Identification WOW 4520)

Sample No. 2265 - One drum of heavy distillate (Chevron UN 1137 Identification WOW 4521)

These samples were drawn on April 4-6, 1980, and shipped on February 6, 1981. These SRC-II process products were derived from Pittsburgh Seam coal from the Blacksville, West Virginia, No. 2 Mine of the Consolidated Coal Company.

Tables I-IV inclusive show inspections of these samples as analyzed by Chevron. Results are compared to previous samples prepared in February 1978 and used in studies described in FE-2315-47. Figure 1 shows the boiling curves for both sets of samples.

DOE requested that we blend the middle distillate and heavy distillate in a ratio of 1.7/1 middle/heavy by weight and process the resulting 400°F+ blend rather than the whole syncrude. This was done after the water washing steps described in the next section.

B. Water Washing of SRC-II Middle and Heavy Distillates

The middle distillate fraction contained 110 ppm chlorine by weight; the heavy distillate contained 360 ppm chlorine. These chlorine-containing compounds react to form HCl during hydrotreating. Subsequently, ammonium chloride plugging could occur in the reactor exit lines. Furthermore, chloride can cause the corrosion of stainless steel processing equipment.

Our previous experience showed that the chlorine-containing compounds in SRC-II are water soluble. By water washing the 400°F+ fraction of SRC-II three times at 170°F, we decreased the chlorine content from 73 ppm to 1 ppm. (See DOE Report FE-2315-47.) Similarly, a single water wash of whole H-Coal syncrude at ambient temperature lowered the

chlorine content from 32 ppm to 1 ppm. (See FE-2315-61.) Therefore, with the permission of the DOE Technical Project Officer, the feed fractions were water washed to remove the chlorine compounds.

It was calculated that the 1.7/1 ratio blend of middle and heavy distillate would have a gravity of 10°API, which is the same as water. Therefore, to avoid separation problems that could result from mixing the blend directly with water, we water washed the middle distillate and heavy distillate fractions separately.

SRC-II middle distillate (755 lb) was mixed with an equal weight of distilled water under an inert atmosphere (nitrogen) and stirred for 2 hr at ambient temperature (about 65°F). The mixture was allowed to stand for 1 hr. A relatively good separation occurred, and the bottom layer (foul water) was drawn off until 760 lb had been removed and the interface was reached. An additional 755 lb of water was mixed with the oil remaining in the kettle and again mixed for 2 hr at ambient temperature, then allowed to stand 1 hr as before. Again the water layer (763 lb) was removed to the interface. An additional 6 lb of the interface material ("cuff") appeared to be an emulsion and was kept separately. The remaining oil (732 lb) was labeled SGQ 7560, sampled for inspections, and stored under an inert atmosphere.

The material balance was as follows:

	<u>Weight, Lb</u>
Input	
SRC-II Middle Distillate	755
First Water Wash	755
Second Water Wash	<u>755</u>
Total Input	2265
Recovery	
SRC-II Middle Distillate	732
Foul Water (First Wash)	760
Foul Water (Second Wash)	763
Interface Emulsion ("Cuff")	<u>6</u>
Total Recovery	2261
Unaccounted For	4

SRC-II heavy distillate (414 lb of WOW 4521) was charged to a kettle under an inert atmosphere (nitrogen), mixed with distilled water (423 lb), and stirred for 2 hr at 120°F. The mixture was allowed to stand for 1 hr; however, a good separation did not occur in that period. The mixture was then heated to 170°F, mixed for 2 hr, and again allowed to stand for another hour. Again, the emulsion did not separate within an hour. Fifty milliliters of a commercial additive

used to break emulsions in desalting (a nonionic surfactant, Oronite OGA 473) was added to aid in the separation. The mixture was stirred 1 hr, allowed to settle overnight (14 hr) at 163°F, and a separation was obtained. The oil layer (bottom layer) of 392 lb was drawn off until an emulsion ("cuff") was reached. This emulsion (27 lb) was drawn off and held separately. Because of the difficulty in separating the layers, a second wash was not performed. The material balance is as follows:

	<u>Weight, Lb</u>
Input	
SRC-II Heavy Distillate (WOW 4521)	414
Distilled Water	<u>423</u>
Total Input	837
Recovery	
SRC-II Heavy Distillate (SGQ 7561)	397
Foul Water	414
Emulsion ("Cuff")	<u>27</u>
	838

C. Blending and Analysis

The water washed fractions were blended in the weight ratio of 1.7 parts of middle distillate to one part heavy distillate. Three gallons of the blend, identified as WOW 4551, was shipped to Dr. Bruce Clark at the Oak Ridge National Laboratory. Also sent to Dr. Clark were 5 gal. of the combined foul wash water, identified as SGQ 7572, and a 3-gal. blend of the components before water washing, identified as SGQ 7559.

Our analyses of the components before and after water washing are given in Tables I and II. Samples of the unwashed and washed blends are given in Table III. Table III also includes analyses of the 1978 material for comparison. Figure 2 compares the distillation of the present blend with the 1978 blend. The results show that the 1978 material and the present blend are quite similar. The new blend contains somewhat more high boiling material, however, and has a considerably higher concentration of hot heptane insolubles.

It is concluded that the water washing was somewhat less effective than in our previous experience. The overall reduction of chlorine for the blended fractions is 81% compared to almost 99% for the previous 400°F+ SRC-II and 96% for the whole H-Coal syncrude. The differences in the results are presumably due to the different procedures used in the present study and are believed to be related to the difficulty in mixing and separating the water and heavy distillate.

Compared to the previous procedures with 400°F+ SRC-II, the present method involved separate washing of the individual fractions, a lower temperature wash of the middle distillate fraction, and fewer washings. In the present study, water washing of the middle distillate at ambient temperature was effective and the chlorine content was reduced from 110 ppm to 2 ppm. However, the washing of the heavy distillate was not nearly as effective as previously when it was diluted with lighter material. The high viscosity of the heavy distillate made contacting less thorough than when lower viscosity components are included in the blend.

To verify the conclusion that better contacting would remove the remaining chlorine, a small-scale test was made by washing the blend of middle and heavy distillate three times at 170°F with distilled water. The chloride content was reduced from 48 to 1.7 ppm.

Rather than repeat the water washing with the blend, we elected to process the original washed blend containing 48 ppm chloride. By using comparatively large diameter exit lines from the reactor for the hydrotreated product, ammonium chloride plugging was avoided.

Tables I, II, and III indicate only minor changes in most of the properties of the fractions before and after the water wash. Most of the properties appear to be within experimental error of the analyses before water washing. In addition to the change in chlorine content, one notable exception is the iron content. The concentration of iron is reduced by a factor of about 10 by the wash. Table III also indicates a 50% reduction in hot heptane insolubles.

IV. Pilot Plant Tests

A. Introduction

DOE requested us to select appropriate refining conditions to prepare samples with three different nitrogen concentrations:

<u>Hydrotreatment Severity Level*</u>	<u>Whole Hydrotreated Liquid Product Nitrogen, ppm</u>
Low	5000
Medium	2000
High	500

*Note that these severity levels are defined differently than those in our previous studies with SRC-II oil. See FE-2315-47.

Three pilot plant runs (90-157, 90-158, 90-159) were made to prepare these samples and to determine the performance of two commercial catalysts: (1) ICR 106 containing nickel, tungsten, silica, and alumina; (2) ICR 113 containing nickel, molybdenum, silica, and alumina. The catalyst samples were sulfided prior to the tests by a procedure simulating commercial sulfiding.

The runs will be described in more detail in subsequent sections of this report.

Table VIII lists run numbers, run hours, conditions to produce the samples, and the catalyst used in each case.

Table IX gives the properties of the three blends sent to DOE.

Table X gives typical yield periods obtained during the time the samples were produced.

Tables XI, XII, and XIII give inspections of start to 350°F, 350-550°F, and 550°F+ fractions of each of the blends.

B. Pilot Plant Run 90-157

Pilot Plant Run 90-157 was a 1700-hr pilot plant test that was started as a screening test to determine appropriate conditions for processing the SRC-II oil to given nitrogen levels using ICR 106 catalyst and continued to prepare product at "medium" severity. The run was made at the following target conditions: 750°F, 2500 psig total pressure, about 2250 psia hydrogen partial pressure, and a recycle gas rate of 15,000 SCF/bbl. The space velocity was varied to change the product nitrogen concentration. The initial space velocity of 0.5 volumes_i of

feed per volume of catalyst per hour was selected for direct comparison with previous Run 76-166 with the 1978 sample 400°F+ SCR-II reported in FE-2315-47, page 29. The 1978 feed contained less material boiling above 700°F and fewer hot heptane insolubles. As will be shown, pilot plant results indicate the earlier feed was considerably easier to process than the present feed.

The catalyst appeared to lose activity rapidly in the early part of the run. This deactivation is, at least in part, believed to be due to the hot heptane insolubles in the feed. However, some catalyst damage may have resulted from several temperature excursions to over 1000°F. The excursions were caused by a combination of a faulty temperature controller and the highly exothermic nature of the hydrogenation reactions of aromatic compounds in the feed. In particular, the reaction was difficult to control at high feed rates until the catalyst was partially deactivated.

After about 500 hr onstream, the catalyst appeared to be relatively stable. At 1.0 LHSV, the product nitrogen was about 2000 ppm and this space velocity was selected for preparation of the "medium" severity product. Product from lined-out portions of the run between 513 and 1037 hr onstream was blended and sent to Oak Ridge National Laboratory.

Table XIV shows two yield periods obtained during this operating period. For comparison, a yield period obtained at similar conditions using the 1978 feed is shown. The 1980 feed is much harder to process than the 1978 feed, as indicated by the the higher product nitrogen and lower hydrogen consumption at a given space velocity.

Hydrogen consumptions at a variety of product nitrogen concentrations are shown in Figure 3. Both the 1978 and 1980 feeds require about the same amount of hydrogen for a given product nitrogen.

Following the preparation of product at "medium" severity, the space velocity was reduced to 0.7 LHSV to obtain product at about 500 ppm nitrogen. However, product from another run was inadvertently included in the blend, making it unsuitable for DOE's purpose. Therefore, the preparation was repeated in Run 90-159.

After the 0.7 LHSV test, the space velocity was decreased to 0.5 to check catalyst activity versus the initial activity at the same conditions. At 350 hr onstream, the product nitrogen had been 15 ppm; at 1500 hr the product nitrogen was 250 ppm. This shows that significant catalyst deactivation occurred between 350 and 1500 hr onstream. Over a similar period of time and operating severities in our previous tests, very little catalyst deactivation occurred with the 1978 feed. Again, this indicates the 1980 feed is more difficult to process than the earlier sample.

C. Pilot Plant Run 90-158

Run 90-158 was a 400-hr pilot plant test, the purpose of which was to make product at 5000 ppm nitrogen ("low" severity). Partially deactivated ICR 113 was selected as the catalyst. The run was started at 1.5 LHSV. The product nitrogen lined out at 3000 ppm. The first yield period shown in Table XVI gives results at 1.5 space velocity. Table XVII shows properties of product fractions.

To obtain the target of 5000 ppm, the space velocity was increased to 2.5 volumes of feed per volume of catalyst per hour. Yield periods at this condition are also given in Table XVI.

The run was terminated after 400 hr when sufficient hydrotreated product to send to DOE had been made.

D. Pilot Plant Run 90-159

Pilot Plant Run 90-159 was a 700 hr test, the primary purpose of which was to prepare product at 500 ppm nitrogen ("high" severity) using ICR 106 catalyst.

The run was started at 1.0 LHSV. As before, the temperature was hard to control early in the run due to the highly exothermic nature of the hydrogenation reaction. The first 200 hr of the run were characterized by erratic operation. The product nitrogen control increased rapidly. After 250 hr, the space velocity was reduced to 0.7 and the product nitrogen lined out at about 500 ppm. The run was continued at these conditions until sufficient product had been prepared to supply to DOE.

Following the test at 0.7 LHSV, the LHSV was lowered to 0.33 for a brief test at even higher severity. Product nitrogen was about 15-20 ppm. Yield periods obtained during Run 90-159 are given in Table XVIII.

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TABLE I

DOE CONTRACT DE-AC22-76ET10532
PROPERTIES OF SRC-II NAPHTHAS

Date Drawn	February 3, 1978	April 4-5, 1980
Date Shipped	February 6, 1978	February 6, 1981
Identification No. (As Received)	1113	2263
Chevron Identification	WOW 3631	WOW 4519
Inspections		
Gravity, °API	40.2	49.7
Aniline Point, °F	60.2	81.0
Sulfur, Wt %	0.42	0.32
Total Nitrogen, Wt %	0.52	0.33
Basic Nitrogen, Wt %	0.28	0.083
Oxygen, Wt %	2.29	1.37
Carbon, Wt %	84.33	85.29
Hydrogen, Wt %	12.44	12.69
Hydrogen/Carbon Atom Ratio	1.76	1.74
Chloride, ppm	3.7	19
Group Type, LV % ¹	Low Mass	Low Mass
Paraffins	23.7	27.5
Naphthenes	45.7	51.5
Aromatics	31.1	20.9
Molecular Weight	120	109
Bromine No.	46	39
Viscosity, cSt		
At 100°F	0.8022	0.6303
ASTM Distillation, °F	D 86	D 86
St/5	186/186	118/150
10/30	200/242	168/203
50	282	230
70/90	316/362	264/301
95/EP	400/465	318/348
% Overhead (Excl. Trap)	98.5	98
% in Flask	1.0	2
% Trap	0.5	0
TBP Distillation, °F		
(Simulated by Chromatography)		
St/5	41/133	42/100
10/30	161/226	140/185
50	287	234
70/90	344/408	282/329
95/99	445/620	338/379

¹Reliability of this mass spectrometric group-type analysis for coal-derived liquids is not known. Nitrogen, oxygen, and sulfur compounds are not analyzed by this method.

TABLE II

DOE CONTRACT DE-AC22-76ET10532
PROPERTIES OF SRC-II MIDDLE DISTILLATES

Date Drawn	February 3, 1978	April 6, 1980
Date Shipped	February 6, 1978	February 6, 1981
Identification No. (As Received)	1114	2264
Chevron Identification	WOW 3632	WOW 4520
Inspections		
Gravity, °API	13.7	13.6
Aniline Point, °F	<32	<17
Sulfur, Wt %	0.18/0.19	0.26
Total Nitrogen, Wt %	1.03	0.99
Basic Nitrogen, Wt %	0.82	0.74
Oxygen, Wt %	3.73	4.31
Carbon, Wt %	86.28	85.30
Hydrogen, Wt %	8.77	9.15
Hydrogen/Carbon Atom Ratio	1.21	1.28
Chloride, ppm	58	110
Pour Point, °F	-65	<-60
Freeze Point, °F		-23
Arsenic, ppm		0.01
Group Type, LV % ¹	High Mass	22 Component
Paraffins	4.5	5.2
Naphthenes	17.9	14.4
Aromatics	77.7	77.2
Sulfur Compounds	-	3.2
Density (80°C)		0.9233
Refractive Index (80°C)	1.5165	1.5120
Molecular Weight	158	170
Bromine No.	62	80
Viscosity, cSt		
At 100°F	3.144	3.520
ASTM Distillation, °F	D 86	D 86
St/5	360/396	376/396
10/30	401/418	400/426
50	435	451
70/90	457/498	478/522
95/EP	516/558	561/590
% Overhead (Excl. Trap)	99	100
% in Flask	1	0
% Trap	0	0
TBP Distillation, °F		
(Simulated by Chromatography)		
St/5	216/357	277/338
10/30	382/426	364/412
50	464	458
70/90	506/559	501/544
95/99	576/653	554/579

¹Reliabilities of these mass spectrometric group-type analyses for coal-derived liquids are not known. Nitrogen and oxygen compounds are not analyzed by these methods. Sulfur is not analyzed by the "high mass" method.

TABLE III

DOE CONTRACT DE-AC22-76ET10532
PROPERTIES OF SRC-II HEAVY DISTILLATES

Date Drawn	February 3, 1980	April 4-6, 1980
Date Shipped	February 6, 1980	February 6, 1981
Identification No. (As Received)	1115	2265
Chevron Identification	WOW 3633	WOW 4521
Inspections		
Gravity, °API	-0.8	4.0
Sulfur, Wt %	0.40	0.31
Total Nitrogen, Wt %	1.25	0.95
Basic Nitrogen, Wt %	0.66	0.58
Oxygen, Wt %	1.91	2.61
Carbon, Wt %	89.11	87.87
Hydrogen, Wt %	7.33	8.26
Hydrogen/Carbon Atom Ratio	0.98	1.12
Chloride, ppm	17	360
Pour Point, °F	+10	-10
Arsenic, ppm		0.09
Group Type, LV %¹		
Paraffins	22 Component	22 Component
Naphthenes	2.1	3.8
Aromatics	10.8	6.9
Sulfur Compounds	77.4	83.3
	9.7	5.9
Ramsbottom Carbon, Wt %	1.20	2.33
Hot Heptane Asphaltenes, Wt %	0.94	2.72
Benzene Insolubles, Wt %	0.14/0.15	
Refractive Index (80°C)	1.5648	1.557
Ash, Wt %	0.06	0.07
Molecular Weight	228	215
Bromine No.	34	53
Viscosity, cSt		
At 100°F	52.89	24.33
At 210°F	4.085	3.146
ASTM Distillation, °F		
St/5	D 1160	D 1160
10/30	530/577	432/462
50	591/628	483/523
70/90	665	598
95/EP	716/855	675/815
% Overhead (Excl. Trap)	884/958	881/915
% in Flask	98	97
% Trap	2	3
	0	0
TBP Distillation, °F		
(Simulated by Chromatography)		
St/5	248/519	251/425
10/30	543/597	445/515
50	639	584
70/90	693/788	657/788
95/99	844/955	857/(1008)

¹Reliability of this mass spectrometric group-type analysis for coal-derived liquids is not known. Nitrogen and oxygen compounds are not analyzed by this method.

TABLE IV

DOE CONTRACT DE-AC22-76ET10532
METALS IN SRC-II HEAVY DISTILLATE

Date Sample Drawn	February 3, 1978	April 4-6, 1980
Chevron Identification	WOW 3633	WOW 4521
Inspections		
Metals by Emission Spec., ppm		
Aluminum	47	23.
Boron	2.0	2.4
Barium	0.2	-
Calcium	10	2.9
Chromium	4.9	-
Copper	0.2	-
Iron	54	410.
Magnesium	2.9	1.4
Manganese	-	7.4
Molybdenum	0.3	-
Sodium	16	-
Nickel	1.2	-
Silicon	27	27
Tin	-	2.0
Titanium	32	13
Vanadium	1.7	1.0
Other Metals Analysis, ppm		
Arsenic	0.03	0.09

TABLE V

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF SRC-II MIDDLE DISTILLATE BEFORE
 AND AFTER WATER WASHING TWICE AT
 AMBIENT TEMPERATURE

Description	As Received	After H ₂ O Wash
Chevron Identification	WOW 4520	SGQ 7560
Inspections		
Gravity, °API	13.6	13.8
Sulfur, Wt %	0.26	0.27
Total Nitrogen, Wt %	0.99	0.97
Basic Nitrogen, Wt %	0.74	0.73
Oxygen, Wt %	4.31	4.17
Carbon, Wt %	85.30	85.32
Hydrogen, Wt %	9.15	9.27
Hydrogen/Carbon Atom Ratio	1.28	1.29
Chloride, ppm	110	2
Bromine No.	80	84
Viscosity, cSt at 100°F		3.48
TBP Distillation, °F (Simulated by Chromatography)		
St/5	277/338	276/356
10/30	364/412	374/419
50	458	460
70/90	501/544	500/543
95/99	554/579	553/583
Arsenic, ppm	0.01	0.01

TABLE VI

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF SRC-II HEAVY DISTILLATE BEFORE
 AND AFTER WATER WASHING ONCE AT 170°F

Description Chevron Identification	As Received	After Water Wash
	WOW 4521	SGQ 7561
Inspections		
Gravity, °API	4.0	4.1
Sulfur, Wt %	0.31	0.37
Total Nitrogen, Wt %	0.95	1.01
Basic Nitrogen, Wt %	0.58	0.57
Oxygen, Wt %	2.61	3.44
Carbon, Wt %	87.87	87.49
Hydrogen, Wt %	8.26	7.69
Hydrogen/Carbon Atom Ratio	1.12	1.05
Chloride, ppm	360	48
Ramsbottom Carbon, Wt %	2.33	2.03
Hot Heptane Insolubles, Wt %	2.72	2.60
Viscosity, cSt		
At 100°F	24.33	23.42
At 210°F	3.146	2.889
TBP Distillation, °F (Simulated by Chromatography)		
St/5	251/425	129/425
10/30	445/515	448/517
50	584	587
70/90	657/708	660/794
95/99	857/(1008)	863/(997)
Arsenic, ppm	0.1	0.1

TABLE VII
DOE CONTRACT DE-AC22-76ET10532
PROPERTIES OF BLEND OF SRC-II
MIDDLE AND HEAVY DISTILLATES BEFORE
AND AFTER WATER WASHING

Description Chevron Identification	1978 Sample		1980 Sample	
	Before Wash	After Wash ¹	Before Wash	After Wash ²
	WOW 3751	WOW 3778	SGQ 7559	WOW 4551
Inspections				
Gravity, °API	10.7	11.3	9.9	10.4
Sulfur, Wt %	0.25	0.26	0.41	0.32
Total Nitrogen, Wt %	0.99	1.08	0.95	0.98
Basic Nitrogen, Wt %	0.82	0.83		0.67
Oxygen, Wt %	3.63	3.91	3.68	3.95
Carbon, Wt %	85.86			85.88
Hydrogen, Wt %	9.27			8.88
Hydrogen/Carbon Atom Ratio	1.29			1.23
Chloride, ppm ³	73	1	205	50
Pour Point, °F	-50		-50	-45
Ramsbottom Carbon, Wt %	0.81		1.21	1.09
Hot Heptane Insolubles, ppm ³	2200		10,500	5600
Ash, Wt %	0.01			0.01
Bromine No.	69			64
Viscosity, cSt				
At 100°F	4.213		6.056	5.754
At 130°F	-		-	3.477
At 210°F	1.238		1.500	1.433
D 1160-10 Distillation by LV %, °F				
St/5	393/413			292/409
10/30	427/448			420/461
50	471			499
70/90	506/590			539/661
95/EP	662/847			755/891
% Overhead (Excl. Trap)	99			99
% in Flask	1			1
% in Trap	0			0
TBP Distillation by LV %, °F (Simulated by Chromatography)				
St/5	233/366	269/365	215/361	290/363
10/30	382/430	380/427	379/444	383/456
50	477	470	489	490
70/90	527/639	519/628	538/666	538/660
95/99	707/849	701/865	755/920	743/892
Maleic Diene Value, cg I ₂ /g	37.3	18.0		27.0
Water by Distillation, Vol %				0.45
Acid Neutralization No., mg KOH/g				<0.1
Arsenic, ppm	0.005			0.05
Metals (Emission Spec.), ppm				
Aluminum	4	(No Analysis)	12	6
Chromium	0.5		0.5	0.5
Copper	-		0.1	0.1
Iron	5		89	9
Manganese	0.2		0.4	0.1
Nickel	0.1		6	-
Silicon	2		7	8
Titanium	4		3	3
Vanadium	0.1		5	0.2

¹Washed three times at 160-170°F.

²Middle distillate fraction washed twice at 65-70°F. Heavy distillate fraction washed once at 170°F.

³Average of two analyses.

TABLE VIII

SUMMARY OF RUN CONDITIONS FOR HYDROTREATED
SRC-II SAMPLES SHIPPED TO OAK RIDGE NATIONAL LABORATORY

Severity	"Low"	"Medium"	"High"
Target Product Nitrogen, ppm	5,000	2,000	500
Actual Product Nitrogen in Blend, ppm	5,300	1,710	505
Run No.	90-158	90-157	90-159
Run Hr. for Product Blend	189-309, 345-393	513-603, 747-1037	262-538
Catalyst	ICR 113	ICR 106	ICR 106
Product Blend Identification	SGQ 7779	SGQ 7711	SGQ 7795
Run Conditions			
Average Catalyst Temperature, °F	750	748	750
Total Pressure, psig	2,500	2,500	2,500
Hydrogen Partial Pressure, psia	2,250	2,250	2,250
LHSV, Vol Feed/Vol Catalyst/Hr	2.5	1.0	0.7
Recycle Gas Rate, SCF/Bbl	15,000	15,000	15,000
Approximate Hydrogen Consumption, SCF/Bbl	975	1,450	1,850

TABLE IX
 PROPERTIES OF RAW AND HYDROTREATED SRC-II
 SAMPLES SENT TO OAK RIDGE NATIONAL LABORATORY

Description	Raw Feed	Low Severity Product	Medium Severity Product	High Severity Product
Chevron Identification	WOW 4451	SGQ 7779	SGQ 7711	SGQ 7795
Gravity, °API	10.4	17.5	22.3	24.4
Aniline Point, °F	-	-	23.0	44.0
Sulfur, ppm	3200	68	51	<10
Total Nitrogen, ppm	9800	5300	1710	505
Basic Nitrogen, ppm	6700	3900	1300	456
Oxygen, Wt %	3.95	1.96	1.03	0.43
Hydrogen, Wt %	8.88	10.02	10.86	11.14
Carbon, Wt %	85.88	87.47	87.94	88.38
Chloride, ppm	50	1.1	1.1	0.8
Hot Heptane Insolubles, ppm	5600	466	225	<20
Ramsbottom Carbon, %	1.09	0.33	0.31	0.25
Water, ppm	4500	2646	598	427
Bromine No.	64	45	22	10
Maleic Diene No., cg I ₂ /g	27	11.1	0.9	2.2
True Boiling Point Distillation, °F (Simulated by Chromatography)				
St/5	290/363	163/293	144/234	164/237
10/30	383/456	349/424	294/400	292/398
50	490	478	459	455
70/90	538/660	531/648	517/614	512/601
95/99	743/892	734/909	687/834	667/798

HYDROTREATING OF 400°F+ SRC-II OIL
TYPICAL YIELD PERIODS AT THREE PROCESSING SEVERITIES

Severity	"Low"		"Medium"		"High"	
Catalyst	ICR 113		ICR 106		ICR 106	
Run	90-158		90-157		90-159	
Feed	WOW 4551		WOW 4551		WOW 4551	
Run Hr	285-297		915-939		298-322	
Average Catalyst Temperature, °F	753		749		751	
LHSV	2.49		0.97		0.70	
Total Pressure, psig	2,503		2,501		2,496	
H ₂ Mean Pressure, psia	2,271		2,255		2,265	
Total Gas In., SCF/Bbl	13,705		16,962		17,770	
Recycle Gas, SCF/Bbl	12,650		15,430		15,811	
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
No Loss Product Yields						
C ₁	0.08	-	0.17	-	0.14	-
C ₂	0.08	-	0.20	-	0.17	-
C ₃	0.07	-	0.17	-	0.13	-
iC ₄	0.01	0.01	0.01	0.02	0.01	0.02
nC ₄	0.02	0.04	0.05	0.09	0.04	0.07
C ₅ -350°F	10.44	12.66	17.35	21.26	17.76	22.00
350-550°F	61.95	65.31	57.81	62.42	60.19	65.88
550-End Point, °F	23.22	22.81	20.45	20.52	18.41	18.67
Total C ₅ +	95.62	100.79	95.62	104.20	96.37	106.55
Actual/No Loss Recovery	104.11/101.48		100.22/102.19		102.71/102.82	
H ₂ Consumption (Gross), SCF/Bbl	1,055		1,531		1,958	
H ₂ Consumption (Chemical), SCF/Bbl	976		1,438		1,852	
Whole Liquid Product Nitrogen, ppm	4,900		1,660		455	

TABLE XI

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF FRACTIONS OF HYDROTREATED PRODUCT
 FROM 400°F+ SCR-II OIL, SGQ 7779, NITROGEN CONTENT
 OF WHOLE LIQUID PRODUCT = 5300 PPM ("LOW" SEVERITY)

Boiling Range, °F	St-350	350-550	550+
LV % of Whole Liquid Product	11.9	62.7	25.4
Inspections			
Gravity; °API	40.1	18.1	9.6
Aniline Point, °F	73.0	<32	-
Sulfur, ppm	16	14	227
Total Nitrogen, ppm	2040	5000	5200
Basic Nitrogen, ppm	1150	4500	3000
Oxygen, Wt %	1.61	2.19	1.00
Hydrogen, Wt %	12.46	10.04	9.60
Carbon, Wt %	85.73	87.27	88.85
TBP Distillation (Simulated by Chromatography)			
St/5	96/180	330/369	534/545
10/30	214/252	387/427	554/591
50	304	462	635
70/90	336/359	497/534	699/832
95/99	366/382	546/660	912/1037
Ramsbottom Carbon, %	-	-	0.89
Hot Heptane Insolubles, Wt %	-	-	0.51

TABLE XII

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF FRACTIONS OF HYDROTREATED PRODUCT
 FROM 400°F+ SRC-II OIL, SGQ 7711, NITROGEN CONTENT
OF WHOLE LIQUID PRODUCT = 0.17 WT % ("MEDIUM" SEVERITY)

Boiling Range, °F	St-350	350-550	550+
LV % of Whole Liquid Product	20.3	60.0	19.7
Inspections			
Gravity, °API	42.2	21.7	10.9
Aniline Point, °F	64.7	-	-
Sulfur, ppm	5	5	-
Total Nitrogen, ppm	590	1870	2300
Basic Nitrogen, ppm	522	1500	964
Oxygen, Wt %	0.76	1.35	0.73
Hydrogen, Wt %	12.67	10.05	10.21
Carbon, Wt %	86.51	88.41	88.83
	Low Mass	High Mass	22-Component
Group Type, LV %			
Paraffins	3.9	3.5	7.3
Naphthenes	70.1	24.6	15.1
Aromatics	26.0	71.8	77.6
TBP Distillation (Simulated by Chromatography)			
St/5	84/178	331/365	409/551
10/30	212/244	384/424	569/601
50	290	459	646
70/90	326/349	494/531	715/777
95/99	358/369	541/554	823/935
Ramsbottom Carbon, %	-	-	0.61
Hot Heptane Insolubles, Wt %	-	-	0.21

TABLE XIII

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF FRACTIONS OF HYDROTREATED PRODUCT
 FROM 400°F+ SRC-II OIL, SGQ 7795, NITROGEN CONTENT
 OF WHOLE LIQUID PRODUCT = 505 PPM ("HIGH" SEVERITY)

Boiling Range, °F	St-350	350-550	550+
LV % of Whole Liquid Product	20.0	61.1	18.9
Inspections			
Gravity, °API	44.0	23.3	13.3
Aniline Point, °F	78.7	<32	68.0
Sulfur, ppm	2	3	14
Total Nitrogen, ppm	152	595	615
Basic Nitrogen, ppm	137	530	288
Oxygen, Wt %	0.15	0.51	0.34
Hydrogen, Wt %	13.26	11.08	10.59
Carbon, Wt %	86.58	88.35	89.01
Group Type, LV %			
Paraffins	3.8	4.1	4.9
Naphthenes	77.6	29.6	17.7
Aromatics	18.7	66.2	77.4
TBP Distillation (Simulated by Chromatography)			
St/5	134/185	335/359	526/552
10/30	204/253	372/418	565/594
50	289	459	629
70/90	322/354	496/534	674/768
95/99	362/379	544/564	818/920
Ramsbottom Carbon, %	-	-	0.42
Hot Heptane Insolubles, ppm	-	-	555

TABLE XIV

DOE CONTRACT DE-AC22-76ET10532
YIELDS FROM HYDROTREATING 400°F+ SRC-II WITH ICR 106

	1980 Feed				1978 Feed	
	90-157 WOW 4551 843-867 749 0.96	90-157 WOW 4551 915-939 749 0.97	90-157 WOW 4551 915-939 749 0.97	90-157 WOW 4551 915-939 749 0.97	76-167 WOW 3778 794-818 751 0.99	76-167 WOW 3778 794-818 751 0.99
Run						
Feed						
Run Hours						
Avg. Catalyst Temperature, °F						
LHSV						
Total Pressure, psig	2,503	2,501	2,501	2,501	2,496	2,496
Hydrogen Mean Pressure, psia	2,272	2,255	2,255	2,255	2,293	2,293
Total Gas In, SCF/Bbl	17,186	16,962	16,962	16,962	17,140	17,140
Recycle Gas, SCF/Bbl	15,613	15,430	15,430	15,430	14,450	14,450
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
No Loss Product Yields						
C ₁	0.17		0.17		0.12	
C ₂	0.20		0.20		0.17	
C ₃	0.19		0.17		0.15	
iC ₄	0.02	0.03	0.01	0.02	0.02	0.03
nC ₄	0.08	0.13	0.05	0.09	0.05	0.08
C ₅ -350°F	17.52	21.52	17.35	21.26	26.37	32.80
350-550°F	57.69	62.28	57.81	62.42	61.09	66.91
550-EP, °F	20.40	20.47	20.45	20.52	10.05	10.62
Total C ₅ +	95.62	104.28	95.62	104.20	97.50	110.33
Actual/No Loss Recovery	101.32/102.24		100.22/102.19		103.73/103.96	
H ₂ Consumption (Gross), SCF/Bbl	1,573		1,531		2,690	
H ₂ Consumption (Chemical), SCF/Bbl	1,472		1,438		2,582	
Nitrogen in Whole Liquid Product, ppm	1,995		1,660		21	

TABLE XV

DOE CONTRACT DE-AC22-76ET10532
 ADDITIONAL YIELDS FROM PILOT PLANT RUN 90-157;
 FEED - WOW 4551 (400°F+ SRC-II OIL);
 CATALYST - ICR 106

Severity	Above High		High		High		Above High	
Nitrogen in Liquid Product, ppm	15		575		780		250	
Run Hours	339-363		1,155-1,179		1,227-1,251		1,491-1,515	
Avg. Catalyst Temperature, °F	749		752		751		750	
LHSV	0.49		0.69		0.73		0.52	
Total Pressure, psig	2,500		2,501		2,492		2,505	
H ₂ Mean Pressure, psia	2,258		2,277		2,247		2,246	
Total Gas In., SCF/Bbl	17,386		16,354		15,138		14,228	
Recycle Gas, SCF/Bbl	14,974		14,529		13,361		12,176	
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
No Loss Product Yields								
C ₁	0.21		0.19		0.17		0.25	
C ₂	0.42		0.25		0.22		0.32	
C ₃	0.46		0.22		0.19		0.37	
iC ₄	0.04	0.07	0.02	0.03	0.01	0.02	0.03	0.06
nC ₄	0.20	0.34	0.07	0.12	0.06	0.10	0.18	0.30
C ₅ -180°F	2.16	3.00						
180-350°F	21.20	26.37						
350°F-EF	72.85	80.04						
Total C ₅ +	96.23	109.41	96.46	105.88	96.54	105.77	96.10	105.78
Actual/No Loss Recovery	109.30/103.53		103.16/102.59		103.32/102.52		93.93/102.90	
H ₂ Consumption (Gross), SCF/Bbl	2,411		1,825		1,777		2,052	
H ₂ Consumption (Chemical), SCF/Bbl	2,317		1,702		1,656		1,905	
Whole Liquid Product Inspections								
Gravity, °API	28.4		24.2		23.9		24.4	
Aniline Point, °P	68.5							
Sulfur, ppm	24							
Total Nitrogen, ppm	15		575		780		250	
Oxygen, Wt %	0.07							
Hydrogen, Wt %	11.83							
Carbon, Wt %	88.10							
Group Type, LV %	High Mass							
Paraffins	2.6							
Naphthenes	53.6							
Aromatics	43.8							
TBP Distillation, °F								
St/5	61/216		149/220		161/233		166/239	
10/30	253/372		276/395		289/402		288/398	
50	439		452		458		454	
70/90	498/585		510/599		515/606		511/602	
95/99	651/809		667/799		676/809		671/812	

TABLE XVI

DOE CONTRACT DE-AC22-76ET10532
 YIELDS FROM PILOT PLANT RUN 90-158;
 FEED - WOW 4551 (400°F+ SRC-II OIL);
 CATALYST - ICR 113

Severity	Between Low and Medium		Low		Low		Low		Low	
Liquid Product Nitrogen, ppm	0.30		0.57		0.49		0.50		0.49	
Run Hours	153-177		237-249		285-297		297-309		369-393	
Average Catalyst Temperature, °F	756		754		753		754		756	
LHSV	1.49		2.48		2.49		2.51		2.47	
Total Pressure, psig	2,494		2,495		2,503		2,513		2,491	
H ₂ Mean Pressure, psia	2,278		2,284		2,271		2,314		2,288	
Total Gas In., SCF/Bbl	15,311		13,910		13,705		15,243		15,380	
Recycle Gas, SCF/Bbl	13,915		12,836		12,650		14,237		14,410	
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
No Loss Product Yields										
C ₁	0.10		0.07		0.08		0.08		0.08	
C ₂	0.11		0.08		0.08		0.08		0.08	
C ₃	0.11		0.07		0.07		0.07		0.07	
iC ₄	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.00	0.01
nC ₄	0.04	0.08	0.02	0.04	0.02	0.04	0.02	0.04	0.02	0.04
C ₅ -350°F	14.22	17.45			10.44	12.66				
350-550°F	58.53	62.57			61.95	63.31				
550°F-EP	23.17	23.09			23.22	22.81				
Total C ₅ +	95.93	103.12	97.95	102.82	95.62	100.79	97.97	102.68	97.97	102.90
Actual/No Loss Recovery	101.21/102.00		97.95/101.51		104.11/101.48		102.19/101.41		102.18/101.36	
H ₂ Consumption (Gross), SCF/Bbl	1,395		1,073		1,055		1,005		969	
H ₂ Consumption (Chemical), SCF/Bbl	1,314		995		976		929		894	
Whole Liquid Product Properties										
Gravity, °API	20.5		17.4		17.5				16.6	
Sulfur, ppm	36				58					
Total Nitrogen, Wt %	0.30		0.57		0.49		0.50		0.49	
Basic Nitrogen, Wt %					0.39					
Oxygen, Wt %	1.44				2.46					
Hydrogen, Wt %	10.91				10.10					
Carbon, Wt %	88.35				86.94					
Chlorine, ppm	1.0				0.4					
Hot Heptane Asphaltenes, ppm					545					
Ramsbottom Carbon, %					0.41					
TBP Distillation, °F (Simulated by Chromatography)										
St/5	168/252		163/294						164/300	
10/30	316/409		350/423						349/419	
50	466		476						472	
70/90	521/625		528/632						522/626	
95/99	700/847		709/858						701/843	

TABLE XVII

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF FRACTIONS OF HYDROTREATED PRODUCT
 FROM 400°F+ SRC-II OIL, SGQ 7755 (90-158 AT 139-177 HR)
 NITROGEN CONTENT OF WHOLE LIQUID PRODUCT = 0.30 WT %
 (BETWEEN "LOW" AND "MEDIUM" SEVERITY)

Boiling Range, °F	St-350	350-550	550+
LV % of Whole Liquid Product	16.7	60.9	22.4
Inspections			
Gravity, °API	42.2	20.2	9.9
Sulfur, ppm	26	25	130
Total Nitrogen, ppm	795	3100	4300
Oxygen, Wt %	0.93	1.63	0.84
Hydrogen, Wt %	-	10.42	9.68
Carbon, Wt %	-	87.64	89.09
TBP Distillation (Simulated by Chromatography)			
St/5	98/178	321/365	518/536
10/30	211/237	387/426	547/590
50	282	461	632
70/90	323/354	496/532	684/785
95/99	363/382	544/579	839/950

TABLE XVIII

DOE CONTRACT DE-AC22-76ET10532
 YIELDS FROM PILOT PLANT RUN 90-158;
 FEED - WOW 4551 (400°F+ SRC-II OIL);
 CATALYST - ICR 106

Severity	High		High		High		Above High		Above High	
Liquid Product Nitrogen, ppm	520		455		625		20		14	
Run	90-159		90-159		90-159		90-159		90-159	
Feed	WOW 4551		WOW 4551		WOW 4551		WOW 4551		WOW 4551	
Run Hours	274-298		298-322		490-514		586-610		634-658	
Avg. Catalyst Temperature, °F	751		751		751		752		752	
LHSV	0.69		0.70		0.68		0.32		0.33	
Total Pressure, psig	2,505		2,496		2,492		2,501		2,500	
H ₂ Mean Pressure, psia	2,267		2,265		2,289		2,216		2,232	
Total Gas In., SCF/Bbl	16,684		17,770		17,388		18,152		17,638	
Recycle Gas, SCF/Bbl	14,702		15,811		15,468		15,585		15,105	
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
No Loss Product Yields										
C ₁	0.15		0.14		0.14		0.23		0.18	
C ₂	0.17		0.17		0.16		0.27		0.24	
C ₃	0.13		0.13		0.14		0.24		0.20	
iC ₄	0.01	0.02	0.01	0.02	0.01	0.02	0.02	0.03	0.01	0.02
nC ₄	0.04	0.07	0.04	0.07	0.04	0.07	0.07	0.13	0.06	0.11
C ₅ -350°F			17.76	22.00						
350-550°F			60.19	65.88						
550°F-EP			18.41	18.67						
Total C ₅ +	96.91	106.69	96.37	106.55	97.04	106.77	96.95	108.31	97.04	108.40
Actual/No Loss Recovery	106.00/102.85		102.71/102.82		106.02/102.75		111.90/103.75		110.80/103.71	
H ₂ Consumption (Gross), SCF/Bbl	1,982		1,958		1,919		2,567		2,532	
H ₂ Consumption (Chemical), SCF/Ebl	1,874		1,852		1,809		2,461		2,435	
Whole Liquid Product Properties										
Gravity, °API			24.7				27.2		27.0	
Aniline Point, °F			42.5				64.0		63.1	
Sulfur, ppm			18				3		4	
Total Nitrogen, ppm	520		455		625		12		22	
Basic Nitrogen, ppm			383							
Oxygen, Wt %			0.42				0.04		0.08	
Hydrogen, Wt %			11.21				12.3		12.3	
Carbon, Wt %			88.32				87.7		87.7	
Group Type, LV %										
Paraffins							1.7		2.1	
Naphthenes							49.9		50.2	
Aromatics							48.4		47.7	
TBP Distillation, °F (Simulated by Chromatography)										
St/5	152/236		155/224		152/236		144/220		149/230	
10/30	295/402		283/396		293/400		271/382		280/390	
50	458		453		457		446		448	
70/90	516/608		511/602		515/610		504/590		504/590	
95/99	678/817		669/801		680/820		652/777		654/789	

FIGURE 1

DOE CONTRACT DE-AC22-76ET10532
BOILING CURVES OF SRC-II
PROCESS PRODUCT FRACTIONS

FIGURE 2

COMPARISON OF 1.7/1 BLEND OF
SRC-II MIDDLE/HEAVY DISTILLATE TO
PREVIOUS 400°F+ SRC-II OIL
DOE CONTRACT DE-AC22-76ET10532

FIGURE 3

PRODUCT NITROGEN VERSUS
HYDROGEN CONSUMPTION FOR
400°F+ SRC-II OIL
DOE CONTRACT DE-AC22-76ET10532