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

Quarterly Report for the Period January—March 1981

By
R. F. Sullivan
D. J. O'Rear

MASTER

May 1981
Date Published

Work Performed Under Contract No. AC22-76ET10532

Chevron Research Company
Richmond, California

U. S. DEPARTMENT OF ENERGY

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PREPARED FOR THE UNITED STATES
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Under Contract No. DE-AC22-76ET10532
(Formerly EF-76-C-01-2315)

I. Abstract

Samples of SRC-II naphtha, middle distillate, and heavy distillate were received and analyzed. These samples are part of a planned study of the potential biological hazards of synthetic crudes. These oils will be hydrotreated when DOE provides blending instructions.

Five drums of EDS syncrude made from Big Brown Texas lignite were received and analyzed. The boiling range and other properties of this syncrude are very similar to the properties of the previously studied H-Coal and SRC-II syncrudes. We expect the hydrotreating severities, which were employed to upgrade the H-Coal and SRC-II syncrudes to transportation fuels, will be close to the severities needed for the EDS syncrude.

Encl. - See Index of Enclosures

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 oil 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 (except for the recently added Task 9 described below) and reported in the third interim report, FE-2315-47.
4. Work on H-Coal derived from Illinois No. 6 is complete. The fifth interim report, FE-2315-61, describing this work is being reviewed.
5. Work on H-Coal process product derived from Wyodak coal is complete. Results are also reported in FE-2315-61, now being reviewed.
6. EDS process product from Big Brown Texas lignite has arrived and the experimental program with this feedstock has begun.
7. At the request of DOE, we have added a new Task 9 to the contract. In this task, we will prepare hydrotreated SRC-II at several severities to supply DOE samples for biological studies. We received three components (naphtha, middle distillate, and heavy distillate) from Pittsburgh and Midway Mining Company on February 17. We are awaiting blending instructions from DOE before proceeding with the pilot plant work on this task. Meanwhile, we have obtained inspections on these fractions which are compared with our previous SRC-II samples in Tables I-IV, inclusive. Figure 1 compares distillation curves of the current and previous samples.

III. Analysis of EDS Syncrude

At the request of DOE, five drums of EDS syncrude made from Big Brown Texas lignite were prepared by Mr. D. F. Ryan of Exxon. Inspections of the coal were supplied by Mr. Ryan and are shown in Table V. The drums were received at Chevron Research on March 16, 1981. According to Mr. Ryan, the EDS syncrude was prepared by blending products from the Baytown 1-ton/day Coal Liquefaction Pilot Plant during Yield Periods 331 through 340. During this time period, the pilot plant was operated in a bottoms recycle mode and included vacuum gas oil recycle as well. A simplified flow scheme was provided by Mr. Ryan and is shown in Figure 2. In this configuration the liquid products are naphtha (nominally C₅ to 400°F) and excess spent solvent (nominally 400-800°F). For Yield Periods 331 to 340, the operating conditions were varied; and the yields of naphtha and solvent varied between the extremes shown below.

	Yield, Lb/100 Lb Coal (Dry, Ash-Free Basis)	
Naphtha	24.0	37.0
Solvent	<u>19.9</u>	<u>9.4</u>
Total	43.9	46.4

A weight blend ratio of 62% naphtha with 38% solvent was picked as an average yield during this operation. All the syncrude was blended in a large tank to ensure uniformity. As shown in Table VI, the inspections which have been obtained on each drum show only the variation expected for experimental error.

The available properties of the EDS syncrude are shown in Tables VII and VIII. For comparison, the properties of the previously studied H-Coal and SRC-II syncrudes are also shown in these tables. When we compare the EDS, SRC-II, and H-Coal syncrudes, it should be remembered that a direct comparison of the liquefaction processes cannot be made from this information because the syncrudes were made from different coals. The SRC-II syncrude was made from Pittsburgh Seam coal from the Blackville, West Virginia, No. 2 mine of Consolidated Coal Company; and the H-Coal syncrudes were made from Illinois No. 6 (Burning Star Mine) and Wyodak coals. The effects of the coal type on the liquefaction yields and syncrude properties are not known to us at

this time. It also should be remembered that the syncrudes refined in this study were selected as being representative syncrudes from the processes at the time of selection. However, these processes may change during their future development, and the ultimate commercial yields and syncrude properties may be different.

When the EDS, Illinois H-Coal, Wyodak H-Coal, and SRC-II syncrudes are compared, several interesting observations can be made. First, the boiling range of all three syncrudes, shown in Figure 3, are similar and almost exclusively in the distillate fuel and gasoline range. Therefore, the production of distillate fuels like jet, diesel, or heating fuels does not require a cracking process. The syncrude can be simply hydrotreated and the desired product can be distilled from the hydrotreated syncrude. The exclusive production of gasoline from these syncrudes requires a cracking process to convert the distillate products.

Most of the properties of the EDS syncrude are within the range of the properties of the previously studied syncrudes. However, the EDS syncrude has the largest amount of material which boils above 600°F and has the highest 95% point as measured by chromatography.

	Big Brown EDS	Wyodak H-Coal	Illinois H-Coal	Pittsburgh Seam SRC-II
Gravity, °API	29.0	35.1	25.8	18.6
Estimated LV % <350°F	42	49	35	26
95% Point, °F	799	602	654	642
Hydrogen, Wt %	11.25	12.74	11.28	10.46
Oxygen, Wt %	1.92	0.85	1.80	3.79
Nitrogen, Wt %	0.30	0.17	0.46	0.85
Sulfur, Wt %	<u>0.12</u>	<u>0.04</u>	<u>0.13</u>	<u>0.29</u>
Total Heteroatom, Wt %	2.34	1.06	2.39	4.93

The EDS syncrude contains hot heptane insoluble compounds (asphaltenes and ash) but not as much as the Illinois H-Coal syncrude.

	Big Brown EDS	Wyodak H-Coal	Illinois H-Coal	Pittsburgh Seam SRC-II
Hot Heptane Insolubles, ppm	1300	680	3500	469

As described in our interim report on H-Coal syncrudes (FE-2315-61), these impurities can greatly hinder the performance of the hydro-treater; but most of them can be removed by redistilling the syncrude.

Mass spectroscopic hydrocarbon analyses were obtained on the syncrudes by Chevron's nine-component or high mass method, as shown in Table VIII. We have reservations about the application of this petroleum-based method to coal-derived syncrudes because this analysis does not detect heteroatom compounds. Up to 20% of the molecules in the EDS syncrude could contain one heteroatom and would not be detected. This method is also designed to detect typical nonolefinic hydrocarbons found in petroleum which boil between 300°F and 650°F. Compounds which boil below 300°F may not be accurately measured, and hydrocarbons atypical of petroleum stocks may be ignored.

The EDS syncrude was also distilled into the following six products: St-180°F, 180-250°F, 250-300°F, 300-350°F, 350-600°F, and 600°F+. The yields from this distillation are shown in Table IX along with a similar distillation of the Wyodak H-Coal syncrude. The yields and properties of these fractions will be followed as the EDS syncrude is refined to transportation fuels.

The pilot plant work on the EDS syncrude started on March 24, 1981. Results will be reported as they become available.

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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
Group Type, LV % ¹	High Mass	Incomplete
Paraffins	4.5	
Naphthenes	17.9	
Aromatics	77.7	
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

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 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
Group Type, LV % ¹	22 Component	
Paraffins	2.1	Incomplete
Naphthenes	10.8	
Aromatics	77.4	
Sulfur Compounds	9.7	
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	1.5570
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	D 1160	D 1160
St/5	530/577	432/462
10/30	591/628	483/523
50	665	598
70/90	716/855	675/815
95/EP	884/958	881/915
% Overhead (Excl. Trap)	98	97
% In Flask	2	3
% Trap	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
 FEED COAL INSPECTIONS
BIG BROWN LIGNITE*

	<u>Composition, Wt %</u>
Carbon	57.61
Fixed Carbon	47.43
Hydrogen	4.79
Oxygen (Diff.)	23.37
Sulfur	
Total	1.20
Pyritic	0.41
Sulfate	0.06
Organic (Diff.)	0.73
Nitrogen	1.22
Chlorine	0.41
Ash (SO ₃ Free)	9.63 (8.18)
Volatile Matter	42.94
Moisture	1.77

Ash Composition, Wt %

Element	Dry	SO ₃ Free
P ₂ O ₅	0.06	0.07
SiO ₂	44.99	52.94
Fe ₂ O ₃	7.31	8.60
Al ₂ O ₃	14.19	16.70
TiO ₂	1.15	1.35
CaO	13.84	16.29
MgO	2.53	2.98
SO ₃	15.02	-
K ₂ O	0.60	0.71
Na ₂ O	0.31	0.36
Total	100.00	100.00

*Data supplied by Mr. D. F. Ryan of Exxon Research and Engineering Company.

TABLE VI

DOE CONTRACT DE-AC22-76ET10532
 COMPARABLE PROPERTIES OF INDIVIDUAL
DRUMS OF BIG BROWN EDS SYNCRUDE

Chevron Identification	WOW 4536-1	WOW 4536-2	WOW 4536-3	WOW 4536-4	WOW 4536-5
Exxon Identification	101440	101441	101442	101443	101444
Gravity, °API	28.4	29.0	28.9	29.2	29.7
Carbon by Combustion, Wt %	87.14	86.17	85.88	85.94	86.78
Hydrogen by Combustion, Wt %	11.22	11.16	11.40	11.30	11.32
Sulfur, Wt %	0.118	0.122		0.110	0.120
Oxygen, Wt %	1.21	2.24	2.30	2.35	1.50
Total Nitrogen, Wt %	0.31	0.31	0.30	0.30	0.28
Chlorine, ppm	9.8	8.2	9.1	10.7	10.4

TABLE VII

DOE CONTRACT DE-AC22-76ET10532
 PROPERTIES OF BIG BROWN EDS, ILLINOIS H-COAL,
 WYODAK H-COAL, AND SRC-II SYNCRUDES

Syncrude	Big Brown EDS	Illinois H-Coal	Wyodak H-Coal	Pittsburgh Seam SRC-II
Chevron Identification	WOW 4536	WOW 3919	WOW 4137	WOW 3666
Gravity, °API	29.0	25.8	35.1	18.6
Aniline Point, °F	(51.3)	<32		<30
Refractive Index				
At 20°C	1.4977	1.5050	1.4735	
At 80°C				1.5073
Density, g/cc				
At 20°C	0.8822	0.9015	0.8478	
At 80°C				0.9019
Molecular Weight	130	147	130	132
Viscosity, cSt				
At 100°F	1.484	1.645	1.225	2.196
At 130°F	1.176			1.627
Hot C ₇ Insolubles, Wt %	0.13	0.35	0.068	0.0469
Ash, ppm	<30	90	20	40
Ramsbottom Carbon, Wt %	0.57	0.29	0.23	0.70
Pour Point, °F				<-80
Bromine No.	38	38	26	70
Maleic Diene No., cg I ₂ /g	6.0	10.4	3.19	30.7
Acid Neut. No., mg KOH/g	0.2			0.2
Carbon by Combustion, Wt %	86.41	86.25	86.20	84.61
Hydrogen by Combustion, Wt %	11.25	11.29	12.74	10.46
Sulfur, Wt %	0.118	0.20	0.41	0.29
Oxygen, Wt %	1.92	1.80	0.85	3.79
Total Nitrogen, Wt %	0.30	0.46	0.17	0.85
Basic Nitrogen, Wt %	0.15	0.30	0.13	0.70
Chlorine, ppm	9.6	32	3	50
Hydrogen/Carbon Atom Ratio	1.55	1.56	1.76	1.47
H ₂ O, ppm	1580	2200	<500	6000
ASTM D 86/D 1160-10				
Distillation by Vol %, °F				
St/5	170/224	140/204	120/188	154/217
10/30	255/316	236/329	215/280	281/382
50	364	389	355	438
70/90	431/622	446/582	424/526	484/597
95/EP	710/852	648/704	601/650	699/850
% Received	99.0	98.5	96.0	98.0
% in Flask	1.0	1.5	4.0	0.0
% in Trap	0.0	0.0	0.0	2.0
TBP Distillation by Vol %, °F (Simulated by Chromatography)				
St/5	53/164	56/177	53/156	56/189
10/30	203/317	213/333	173/261	241/379
50	370	404	354	424
70/90	452/662	476/588	429/535	473/562
95/99	799/(1399)	654/765	602/785	642/820

Note: () = questionable result.

TABLE VIII

DOE CONTRACT DE-AC22-76ET10532
 NINE-COMPONENT MASS SPECTRUM ANALYSES OF BIG BROWN EDS,
 ILLINOIS H-COAL, WYODAK H-COAL, AND SRC-II SYNCRUDE

Synchrude Chevron Identification	Big Brown EDS WOW 4536	Illinois H-Coal WOW 3919	Wyodak H-Coal WOW 4137	Pittsburgh Seam SRC-II WOW 3666
Hydrocarbon Group Types, LV %*				
Paraffins	9.9	4.4	9.5	6.4
Naphthenes				
Cycloparaffins	20.5	11.9	27.0	4.3
Dicycloparaffins	11.5	8.4	9.1	5.1
Tricycloparaffins	<u>17.7</u>	<u>19.9</u>	<u>10.4</u>	<u>19.8</u>
Total Naphthenes	49.7	40.2	46.5	29.2
Aromatics				
Alkylbenzenes	13.9	15.7	16.3	21.1
Indans/Tetralins	16.5	21.6	20.3	19.7
Benzodicycloparaffins	1.7	3.7	2.1	2.5
Naphthalenes	4.5	9.6	3.9	17.0
Benzoindans/Benzotetralins	<u>3.8</u>	<u>4.9</u>	<u>1.4</u>	<u>4.1</u>
Total Aromatics	40.4	55.4	44.0	64.4
Avg. No. of Rings per Molecule				
Naphthenic	1.20	1.22	1.02	1.02
Aromatic	<u>0.48</u>	<u>0.69</u>	<u>0.49</u>	<u>0.85</u>
Total	1.68	1.91	1.51	1.87
Carbon Type Distribution, Wt %				
Paraffinic Carbon	8.5	9.6	16.6	1.0
Naphthenic Carbon	61.2	52.7	52.9	49.0
Aromatic Carbon	30.3	37.7	30.5	50.0
Hydrogen, Wt %				
By MS*	11.24	10.56	11.62	9.43
By Combustion	11.25	11.29	12.74	10.46

*This mass spectrum analysis detects only these types of hydrocarbons. Other hydrocarbon types and nonhydrocarbons are ignored; consequentially, the hydrogen content is often in error.

TABLE IX

DOE CONTRACT DE-AC22-76ET10532
 DISTILLATION YIELDS FROM BIG BROWN EDS
 AND WYODAK H-COAL SYNCRUDES

Cut	Big Brown EDS			Wyodak H-Coal		
	Wt %	Vol %	°API	Wt %	Vol %	°API
St-180°F	10.84	13.47	69.6	10.46	12.55	69.7
180-250°F	9.25	10.57	53.9	14.69	16.19	53.7
250-300°F	10.14	11.12	46.4	11.16	11.79	46.0
300-350°F	13.23	13.47	33.6	10.46	10.54	37.9
350-600°F	41.90	39.35	20.7	-	-	-
600°F+	14.64	12.02	1.4	-	-	-
350°F+	<u>56.54</u>	<u>51.37</u>	15.7	<u>53.23</u>	<u>48.93</u>	23.0
Total	100.00	100.00		100.00	100.00	

CHEVRON RESEARCH
 COMPANY
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FIGURE 1

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BOILING CURVES OF SRC-II
PROCESS PRODUCT FRACTIONS

FIGURE 2

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EDS FLOW DIAGRAM
VGO AND BOTTOMS RECYCLE*

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* Figure Supplied by Mr. D. F. Ryan of Exxon Research and Engineering Company

FIGURE 3

SIMULATED TRUE BOILING POINT DISTILLATIONS FOR
PITT. SEAM SRC-II, ILLINOIS H-COAL, WYODAK H-COAL, AND
BIG BROWN EDS SYNCRUDES
DOE CONTRACT DE-AC22-76ET10532

*As simulated by chromatography.