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

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Quarterly Report for the
Period April-June 1978

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MASTER

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I. Abstract

SRC-I and SRC-II, two different forms of solvent refined coal, have sharply contrasting characteristics as coal-derived feeds for conversion to transportation fuels. SRC-I process product is an relatively unattractive feed for conversion to distillate fuels using commercial fixed bed catalytic hydroprocessing technology. It is necessary to dissolve the high melting SRC-I in a solvent before it can be pumped in the processing units. Catalyst bed and equipment plugging is a serious problem. The catalyst rapidly deactivates, and hydrogen consumption is very high. Refining costs for SRC-I process product are judged to be relatively high.

In contrast, preliminary results indicate that the SRC-II process product is quite an attractive feed for conversion to transportation fuels using modern petroleum hydroprocessing technology. Essentially all of the nitrogen can be removed in a single catalytic hydrotreating stage to yield a low boiling distillate. Based on limited information on the product properties, we expect to be able to convert this distillate to specification transportation fuels using conventional petroleum processing technology.

II. Contract Objectives and Scope of Work

The objective of the program is to determine the feasibility and estimate the costs of hydroprocessing four synthetic crude feedstocks to distillate fuels, including high octane gasoline, using presently available technology.

Studies of the processing of Paraho shale oil are complete and are described in an Interim Report (FE 2315-25) issued during the quarter. Studies of the processing of the second feedstock, solvent refined coal, are in progress. This study has been subdivided to include two types of solvent refined coal from the SRC process, both produced at the DOE pilot plant in Tacoma, Washington. Work on SRC-I product has been completed; our current experimental program is devoted to the processing of SRC-II product. The third feedstock will be H-Coal process product. The fourth feedstock is to be another coal-derived liquid to be selected by the mutual agreement of DOE and Chevron.

The feasibility of hydroprocessing each of the synthetic liquids mentioned above will be compared through catalyst tests and evaluations, whereby commercial plant yields, hydrogen consumption, product distribution, and product inspection will be estimated. The necessary tests and evaluations for each feedstock will be done to support "process comparison"-type estimates for each of the major refining steps. The results of the contract, insofar as hydroprocessing is concerned, will be obtained with Chevron commercial catalysts.

Catalyst activity and stability information for each feedstock will be obtained as needed to define commercial operating conditions. These data will provide the basis for the overall refining plan, plant cost estimates, utility and hydrogen requirements, etc. If tests show that refining a particular feedstock using presently existing information is not feasible, it is not intended under this program to conduct any research or development work to solve the problems encountered.

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.

III. Summary of Progress to Date

According to the original timing estimate for an individual feedstock, the preliminary feed analyses and pilot plant program for each feedstock will require one year, followed by an additional 14 weeks for completion of the product analyses, pilot plant report, and final process design. Experience shows that the actual timing on a given feedstock will vary as the program is adapted to the processing route or routes selected for a particular feed.

Delays in obtaining feedstocks have resulted in some postponements of the program beyond the original schedule.

Figure 1 shows the work completed thus far and the anticipated timing for Feedstocks 2B, 3, and 4.

At present, only a limited supply of Feedstock 3, H-Coal process product, is available. The schedule assumes sufficient additional feed will be available to complete a full program on this feedstock.

Figure 2 shows the completed schedule for processing Paraho shale oil.

Figure 3 shows the completed schedule for processing Feed 2A, SRC-I. Work on this feedstock was suspended during the first quarter of 1978.

Figure 4 shows the timing of the anticipated program for processing Feed 2B, SCR-II.

IV. Description of Technical Progress--Shale Oil Processing

The program on the processing of the first feedstock, Paraho shale oil, is now complete. The interim report covering the studies on shale oil, FE 2315-25, was transmitted to DOE in draft form during the month of April and in final form in June. The first part of this report describes the experimental laboratory program and pilot plant studies; the second part describes the engineering design studies and presents the estimated processing costs. With the permission of the DOE technical representative, the interim report was submitted instead of a quarterly report, which would normally have been due in April. (For this reason, results of SRC-I processing obtained during the first quarter of 1978 are reported in the present report.)

At the request of DOE, a paper was prepared for presentation at the Eleventh Annual Oil Shale Symposium sponsored by the Colorado School of Mines, April 12-14, 1978. The paper, "Converting Green River Shale Oil to Transportation Fuels," by R. F. Sullivan and B. E. Stangeland, summarized the pilot plant work on Paraho shale oil under this contract.

A paper, "Refining Shale Oil," by R. F. Sullivan, B. E. Stangeland, H. A. Frumkin, and C. W. Samuel was presented at the American Petroleum Institute Refining Department 43rd Midyear Meeting at the Session on "Conserving Petroleum - New Feedstocks and Fuels for Refineries" on May 10, 1978, in Toronto, Ontario, Canada. It is available as Preprint No. 25-78. This paper included information obtained under EF-76-C-01-2315, although the costs of preparation and presentation of the paper were paid by Chevron.

Results of our shale oil processing studies were presented at a seminar for representatives of DOE and the Department of Defense in Washington, D.C., on May 23, 1978.

Results of these studies were also summarized at the Naval Research Laboratory-Naval Air Systems Command Workshop on "Basic Research Needs for Synthetic Hydrocarbon Jet Aircraft Fuels" in Washington, D.C., on June 15, 1978.

V. Description of Technical Progress--Processing of SRC-I

The object of this study was to determine whether it is feasible to convert SRC-I to transportation fuels using modern commercial hydroprocessing technology. The first processing step was to be hydrotreating of the whole SRC-I in a fixed catalyst bed hydrotreater using commercial catalyst.

Figure 5 is a simplified schematic flow diagram of our pilot plant studies for processing SRC-I. Because of the very high pour point of SRC-I, it was necessary to use a solvent to pump the SRC-I in our pilot plant equipment. SRC recycle solvent was unavailable; so, therefore, at the suggestion of DOE, 50% creosote oil by weight was used as the solvent. Hydrotreating tests with the 50/50 SRC/creosote oil were made until sufficient 350-850°F product was available to serve as a "simulated recycle solvent" in subsequent tests. In downstream processing tests, the 350-850°F product prepared from SRC/creosote oil was further hydrotreated and then either (1) hydrocracked or (2) fluid catalytic cracked.

Task 1

Feed Preparation

Properties of the feedstocks were given in previous quarterly reports (FE 2315-15, -19, -22). For convenience, some of the important properties of the SRC-I, the creosote oil, and the 50/50 SRC-I/creosote oil feed are reported in Table I.

Task 2--Hydrotreating Tests

A. Creosote Oil

Creosote oil was hydrotreated alone in a "blank run" to determine how this solvent would behave at the conditions of the initial hydrotreating experiments (Run 30-30). Results shown in Tables II and III include yields, hydrogen consumptions, and product properties.

B. 50/50 SRC-I/Creosote Oil

Details of the processing of 50/50 SRC-I/creosote oil were given previously (FE 2315-15, -19, -22). Some of the analyses are included in this report for comparison with other results discussed herein.

C. 50/50 SRC-I/Simulated Recycle Solvent

Tests were made in which the diluent solvent was a simulated recycle oil separated from the product of the prior runs in which creosote oil was used as solvent. These test were complicated by an unusual amount of plugging. Only one yield period was obtained and that after less than 24 hours on stream. The results shown in Tables IV and V, thus, are not representative of lined-out operation. They do confirm the general premise that better results are obtained with a reprocessed (higher hydrogen content) solvent than with unprocessed creosote oil. Table VI shows properties of the fractions of this product.

Unfortunately, all of our attempts to process the SRC-I/simulated recycle solvent resulted in plugging, either within the catalyst bed, preheat areas, or feed lines, within the first two or three days of operation.

D. 350-850°F Hydrotreated SRC-I/Creosote Oil

Insufficient product was available from the SRC-I/simulated recycle solvent runs for downstream processing. Therefore, downstream processing studies were made on the hydrotreated 350-850°F fraction from the SRC-I/creosote oil blend. This fraction was first passed through a second-stage hydrotreater (as shown in the schematic flow diagram in Figure 5). Table VII shows the results of this second hydrotreatment. The product at 350 ppm nitrogen is quite similar to that obtained from the SRC-I/solvent blend in a single hydrotreating step (Table V) and, therefore, for first approximations, can serve as a substitute feed for downstream tests. (There are some differences; for example, the product from the SRC-I/solvent contained 1800 ppm asphaltenes and had a lower concentration of aromatics than the twice processed 350-850°F SRC-I/creosote oil.)

Task 4--Extinction Recycle Hydrocracking of 350-850°F Product from Hydrotreating SRC-I/Creosote Oil

Brief hydrocracking tests were made in which the 350-850°F fraction of the hydrotreated product from the SRC-I/creosote oil blend was hydrocracked to extinction in a single stage over ICR 106 catalyst. The catalyst had been aged previously for about 1300 hr with Arabian vacuum gas oil and the 625-850°F fraction of hydrotreated shale oil.

Two tests were made: The first feed (SGQ 6269) contained 350 ppm nitrogen; the second feed (SGQ 6268) contained 900 ppm nitrogen. Properties of the two feeds are given in Table VII.

Recycle cut point was approximately 380°F; per-pass conversion was low. Operating conditions are given in Table XIII along with the yields and hydrogen consumptions. Tables IX and X give the properties of the products. The naphtha product is of high quality.

Task 4A--Catalytic Cracking of 350-850°F Product from Hydrotreating SRC-I/Creosote Oil

The catalytic cracking characteristics of the two hydrotreated SRC-I/creosote oil blends (Table VII) were briefly explored. The limited quantities of feeds available permitted only a single four-cycle run to be made in the fixed-fluidized bed test unit (FCTU) with each of the hydrotreated blends.

The operation of the FCTU was described previously page 32 of the interim report issued in April 1978 (FE 2315-25). Nominal reactor conditions for the present work were:

Reactor Temperature, °F	975
Feed Rate, ml/Min.	60
Water (Steam) Rate, ml/Min.	4
Feed Period, Min.	5
Catalyst, g	305

An equilibrium catalyst (CCL-4914) withdrawn from an operating FCC unit was used for this study. It is the same catalyst employed in the earlier catalytic cracking studies with hydrotreated Paraho shale oil. It is a moderately active, moderately metal contaminated zeolite catalyst. Catalyst inspections were shown in Table LXX on page 126 of the interim report (FE 2315-25).

Table XI lists the cracking conditions, conversions, and product yields for the two feeds. A low cracking severity [defined as the ratio: catalyst/oil ratio (C/O) ÷ weight hourly space velocity (WHSV)] was chosen because proprietary catalytic cracking studies with hydrotreated creosote oil using the same equilibrium catalyst indicate higher severities produce only slight increases in conversion, with the incremental conversion going exclusively into coke and light gases. At constant severity, conversion increases with increasing H/C atom ratio; therefore, crackability is related to the amount of hydrogen introduced into the SRC-I/creosote oil as shown in the table below.

H/C Ratio and Nitrogen Content of
Hydrotreated SRC-I/Creosote Oil

Identification	H/C Atom Ratio	Nitrogen, ppm	FCC Conversion Below 430°F, LV %
SGQ-6268	1.27	900	48.6
SGQ-6269	1.47	350	61.7

Coke yields from cracking of these two oils were very low at 1% of feed because of the very low severity employed. Prior experience with the hydrofined creosote oils shows that coke yield increased sharply with increasing severity.

Gasoline selectivity is excellent due to the low coke and light gas yields produced at this low cracking severity. The cycle oils would undoubtedly be very refractive to further cracking. Thus, the conversions achieved (50-60%) are probably close to optimum for producing maximum gasoline yields.

Inspections of gasolines and cycle oils are shown in Table XII.

DOE Contractor's Conference,
May 16, 1978

A summary of results of our work on the processing of SRC-I was presented at the DOE Contractors Conference on Refining of Coal-Derived Liquids in Washington, D.C., on May 16, 1978.

Conclusions--SRC-I Processing

Based on these experiments, SRC-I does not appear to be an attractive feed for conversion to transportation fuels using current commercial fixed bed catalytic hydroprocessing technology. (We do not mean to imply that it is unsuitable for its originally intended use as a boiler fuel.

Our test of over 1100 hours showed that under certain circumstances, SRC-I can be processed for relatively long periods in a fixed bed without bed plugging. However, the catalyst fouled rapidly; and the product contained a substantial amount of 850°F+ material. Hydrogen consumption was high. The demonstrated catalyst life would not be acceptable by petroleum processing standards. Altering processing conditions by changes such as increasing hydrogen pressure should improve catalyst life and conversion.

With the SRC-I/solvent at higher conversions, bed plugging occurred within the first 100 hours on stream. This is a serious problem that would have to be solved before it could be said that SRC-I processing in a fixed bed is commercially feasible. Probably the high ash content, the high metals content, the high metals content, and the coke-forming tendencies of SRC-I all contribute to the plugging problems. It is also possible that hydrogenation of the recycle oil reduces its solvent power and that this leads to an increased tendency to precipitate asphaltene-like feed constituents within the equipment.

Various solutions can be suggested to correct or minimize the plugging problems. These solutions would require research beyond the scope of the present study. We believe that alternative coal liquids such as SRC-II, H-Coal process product, and EDS product will present fewer downstream process problems than SRC-I and, therefore, should be tested in similar studies before further work is done on conversion of SRC-I to transportation fuels via the routes suggested here. Therefore, it was agreed that work be directed toward the SRC-II product . By mutual agreement of Chevron and DOE, SRC-II was substituted for SRC-I to complete this part of the program.

VI. Description of Technical Progress--SRC-II Processing

As indicated in the previous section, emphasis was shifted to a different solvent refined coal product, SRC-II. Because this feed replaces SRC-I as the second feed in the program, it will be referred to as Feed 2B.

Task 1--Preliminary Feed Analysis

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

No. 1113 - Five Drums of Naphtha (Chevron Identification WOW 3631)
No. 1114 - Six Drums of Middle Distillate (Chevron Identification WOW 3632)
No. 1115 - Three Drums of Heavy Distillate (Chevron Identification WOW 3633)

Tables XIII and XIV summarize the inspections of these samples obtained by Chevron Research.

Tables XV and XVI show the inspections of a blend of the three fractions in the appropriate ratios as recommended by the DOE Technical Project Officer to constitute the whole liquid process product from "typical" SRC-II operation.

According to our information, a large portion of the SRC-II blend was prepared from Kentucky No. 9 and Kentucky No. 14 coals. Some of the product, however, was prepared from Illinois No. 6 coal (River King Mine) and a small portion from West Virginia Coal (Pitt-Seams Blackville No. 2 Mine).

Task 2--Whole Oil Hydrotreating

Our first pilot plant tests are catalyst screening to select the appropriate Chevron catalyst for hydrotreating the whole SRC-II liquid product blend to remove nitrogen, sulfur, oxygen, and metals.

The first run (Pilot Plant Run 76-165) is a test of catalyst ICR 106 containing nickel, tungsten, silica, and alumina. With the assistance of the Chevron Research Process Engineering Department, the following conditions were selected for the test:

Total Pressure, psig	2,500
H_2 Pressure, psia	2,000 Minimum
Liquid Hourly Space Velocity (LHSV), Vol. Feed/Vol. Cat/Hr	0.5
Catalyst Temperature, °F	750
Recycle Gas Rate, SCF/B	15,000

Primary kinetics are to be determined by adjusting conditions to change the product nitrogen.

At the time of writing (July 5, 1978), the run has been on stream for over 450 hours. At 350 hours, the LHSV was increased to 1.0. At 425 hours, it was further increased to 1.5

Liquid product nitrogen at 0.5 and 1.0 LHSV was below 0.5 ppm, and at 1.5 LHSV it is about 15-20 ppm. There is no evidence of catalyst fouling at this time. Gross hydrogen consumption is roughly 3000-3200 SCF/B at 0.5 LHSV, 2600-2800 SCF/B at 1.0 LHSV, and appears to be about 2100 SCF/B at 1.5 LHSV. General performance with this feed is better than expected. We plan to reduce hydrogen pressure to obtain product with higher concentrations of nitrogen and to adjust conditions to obtain some catalyst fouling.

Only limited information on other product inspections is available; this data is shown in Table XVII. Additional inspection will be presented in future reports.

A second catalyst screening test using ICR 113 catalyst containing nickel, molybdenum, silica, and alumina has been started. Pilot Plant Run 65-193 is running at 1.0 LHSV and the other conditions listed above. No analytical information is yet available for this run.

Conclusions and Program

SRC-II Processing

Preliminary results on hydrotreating of SRC-II process product with ICR 106 catalyst are encouraging. Nitrogen removal is less difficult than anticipated; product containing less than 0.5 ppm nitrogen is obtained in a single catalytic stage. During the next few weeks, we will continue to study the effects of process variables on the hydrotreating of SRC-II with ICR 106 and ICR 113. Later, we will compare hydrotreating of the entire SRC-II distillate blend (as we are doing in the present studies) to a processing scheme involving distillation to obtain a naphtha and a heavier distillate fraction followed by hydrotreating of these two fractions separately. The results will be used to determine which processing scheme is least costly.

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TABLE I

DOE CONTRACT EF-76-C-01-2315
FEED PROPERTIES

Description	SRC-I[1]	400-700°F Creosote Oil[2]	50/50 SRC-I/ Creosote Oil
Chevron Identification No.	WOW 3406	WOW 3366	WOW 3476
Inspections			
Gravity, °API	-14.6	-4.9	-7.4
Nitrogen, Wt %	2.04	0.78	1.46
Sulfur, Wt %	0.89	0.64	0.90
Oxygen, Wt %	4.52	1.11	2.70
H/C Atom Ratio	0.81	0.74	0.76
Ash, Wt %	0.22	>0.003	0.11
Hot C_7 Insolubles, Wt %	96.0	0.0023	52.2
Benzene Insolubles, Wt %		>0.003	30.2
Ramsbottom Carbon, %		0.60	29.0
Chloride, ppm	50	9	30
Metals, ppm			530

[1]SRC-I supplied by Pittsburg and Midway Coal Mining Company from the Du Pont, Washington pilot plant.

[2]70% overhead from Allied Chemical cresote oil.

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TABLE II

DOE CONTRACT EF-76-C-01-2315, HYDROPROCESSING CREOSOTE OIL WITH ICR 106 AT 2500 PSIG TOTAL PRESSURE

Run 30-30

	Feed WOW 3366	Product						
Run Hr		201	369	568	712	724	748	772
Average Cat. Temp., °F		749	750	780	779	780	749	749
Average LHSV		0.100	0.095	0.090	0.20	0.19	0.19	0.19
Gravity, °API	-4.9	27.2	27.7	24.9	19.3	19.2	16.8	16.8
Aniline Point, °F		125.9	124.3	117.0	81.1	81.1	72.9	71.8
Wt %								
Hydrogen	5.63	13.03	13.05	11.89	11.27	11.29	10.98	11.17
Carbon	90.70	85.89	85.37	87.76	88.02	88.35	88.66	88.38
Oxygen	1.11	<0.02	<0.01	0.024	0.034	0.023		0.077
Sulfur	0.64	0.0098	0.0260	0.003	0.001	0.00024	0.00027	0.00003
Total Nitrogen, ppm	7800	2.1	2.4	0.54	0.41	0.44	2.1	2.7
Viscosity, cSt								
at 100°F		3.000	2.884	3.256	3.708	3.923	4.690	4.815
at 210°F	2.33	1.205	1.200	1.313	1.376	1.457	1.816	1.714
Hot C_7 Insolubles, ppm	23	20	0	79	117	66	153	120
Ramsbottom Carbon, %	0.60	0.08	0.09	0.10	0.23	0.13	0.20	0.16
Benzene Insolubles, %	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Group Type, LV %, (22 Component)								
Paraffins		0	0	0	0.5	0	0	0
Naphthenes		91.1	93.7	82.7	66.3	49.4	40.2	39.2
Aromatics		8.9	6.3	17.3	33.2	50.6	59.8	60.8

TABLE III

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF CREOSOTE OIL WITH ICR 106
YIELDS AND HYDROGEN CONSUMPTION FOR
PILOT PLANT RUN 30-30
FEED - WOW 3366

Run Hr	688-712		712-724		736-748		760-772	
Average Cat. Temp., °F	780		780		749		750	
LHSV	0.20		0.20		0.20		0.20	
Total Pressure, psig	2,501		2,501		2,499		2,495	
H_2 Mean Pressure, psia	2,224		2,218		2,243		2,267	
Total Gas In, SCF/Bbl	14,723		14,883		13,329		13,406	
Recycle Gas, SCF/Bbl	9,717		9,952		8,624		8,817	
No Loss Prod. Yields	Wt, %	Vol, %	Wt, %	Vol, %	Wt, %	Vol, %	Wt, %	Vol, %
Methane	0.21		0.20		0.17		0.12	
Ethane	0.21		0.30		0.19		0.15	
Propane	0.37		0.35		0.17		0.14	
Isobutane	0.03	0.06	0.03	0.06	0.02	0.03	0.02	0.03
n-Butane	0.36	0.68	0.35	0.66	0.16	0.30	0.13	0.24
Total C_5+	102.64	121.88	102.58	121.72	102.81	120.04	102.81	120.02
Actual/No Loss Recovery	101.43/106.77		103.87/106.66		105.55/106.36		104.90/106.21	
H_2 Cons. (Gross), SCF/Bbl	5,006		4,930		4,704		4,589	
H_2 Cons. (Chemical), SCF/Bbl	4,950		4,876		4,653		4,541	

TABLE IV

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL
AND 50/50 SRC-I/SOLVENT WITH ICR 106
YIELDS AND PRODUCT PROPERTIES

Feed Type	SRC/Creosote Oil		SRC/Solvent*	
Feed Identification	WOW 3476		WOW 3567	
Run	87-67		30-30	
Run Hours	294-306		844-856	
Average Cat. Temp., °F	750		749	
LHSV	0.19		0.20	
Total Pressure, psig	2,498		2,502	
H_2 Mean Pressure, psia	1,929		2,256	
Total Gas In, SCF/Bbl	12,579		14,654	
Recycle Gas, SCF/Bbl	10,212		10,680	
No Loss Prod. Yields	Wt, %	Vol, %	Wt, %	Vol, %
Methane	0.33		0.09	
Ethane	0.55		0.15	
Propane	0.50		0.17	
Isobutane	0.04	0.08	0.02	0.03
n-Butane	0.30	0.58	0.12	0.22
C_5-300°F	2.18	3.32	1.45	2.18
300-550°F	24.46	29.27	46.64	55.64
550-650°F	26.42	29.48	36.05	40.21
650-850°F	16.07	17.26	12.57	13.25
850°F-EP	26.42	30.25	3.17	3.12
Total C_5+	95.59	109.60	99.90	114.41
Actual/No Loss Recovery	104.23/103.06		99.13/105.41	
H_2 Cons. (Gross), SCF/Bbl	2,367		3,973	
H_2 Cons. (Chemical), SCF/Bbl	2,310		3,905	

*Note: This yield period was taken within the first 24 hours of operation with SRC-I/solvent as feed. The previous feed was creosote oil. The product cannot be considered as representative of lined-out operation and, therefore, is not directly comparable to that shown for SRC-I/creosote oil.

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TABLE V

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF CREOSOTE OIL, SOLVENT, THEIR SRC FEED BLENDS, AND THE PRODUCTS ON ICR 106

Description	50/50 SRC-I/Creosote Oil		50/50 SRC-I/Solvent		Creosote Oil		Solvent[1]
	Feed	Product	Feed	Product	Feed	Product	
Identification	WOW 3476	Run 87-67	WOW 3567	Run 30-30	WOW 3366	Run 30-30	
Run Hr		294-306		844-856*		760-772	
Average Cat. Temp., °F		750		749		750	
Average LHSV		0.19		0.20		0.20	
Gravity, °API	-7.4	3.6	-2.9	16.1	-4.9	16.8	5.4
Wt %							
Hydrogen	5.70	8.29	6.79	10.47	5.63	11.17	8.86
Carbon	89.94	87.80	86.12	87.00	90.70	88.38	89.68
Oxygen	2.70	0.57	2.53	0.13	1.11	0.077	0.59
Sulfur	0.90	0.04	0.54	0.004	0.64		0.008
H/C Atom Ratio	0.760	1.13	0.942	1.43	0.745	1.517	1.186
Total Nitrogen, ppm	14,600	4300	12,900	240	7800	2.7	2200
Viscosity, cSt							
at 100°F		41.6		5.77		4.82	7.98
at 210°F		5.16	2293	1.74	2.33	1.71	1.98
Hot C_7 Insolubles, %	52.2	8.01	40.8	0.18	0.0023	0.012	0.013
Ramsbottom Carbon, %	29.0	10.5	28.6	0.74	0.60	0.16	0.28
Benzene Insolubles, %	30.2		14.0	0.04	<0.03	<0.03	<0.03
Conversion, %							
Oxygen		79		95		93	
Sulfur		96		99			
Nitrogen		70		98		~100	
Hydrocracking below 850°F		53		93			
Chemical Hydrogen Consumption							
SCF/Bbl		2310		3905		4541	

[1]350-850°F of SGQ 6218, which is a product blend from Run 87-67 and Run 30-27.
[2]This yield period was taken within the first 24 hours of operation with the SRC-I/solvent feed blend. Previous feed was creosote oil. The product cannot be considered representative of lined-out operation and should not be compared directly to the other products shown in this table.

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TABLE VI

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF FRACTIONS OF HYDROTREATED
SRC-I/SOLVENT FROM RUN 30-30 AT 844-856 HOURS

Fraction No.	1	2	3	4	5
Boiling Range, °F	St-300*	300-550	550-650	650-850	850+
LV % Liquid Product	1.5	48.8	35.3	11.6	2.8
Inspections					
Gravity, °API	57.6	22.5	12.5	4.6	-4.6
Sulfur, ppm	110	10	10	100	<1000
Nitrogen, ppm	19	94	173	395	4500
Oxygen, Wt %	1000	850	710	1600	6200
Hydrogen, %	12.90	11.62	10.94	9.00	8.22
Carbon, Wt %	(79.02)	(85.10)	89.44	89.13	89.22
Hydrogen/Carbon Atom Ratio					
Molecular Weight	103	186	212	238	568
Group Type, LV %	Low Mass	High Mass	High Mass	22-Component	
Paraffins	2.2	6.3	0.6	0	
Naphthenes	89.1	43.4	69.6	7.5	
Aromatics	8.7	50.3	29.7	92.5	
TBP Distillation (Simulated By Chromatography), LV %					
St/5	91/163	264/363	436/527	166/624	
10/30	169/192	389/455	545/581	637/664	
50	224	492	596	684	
70/90	254/289	522/546	613/641	708/760	
95/99	309/408	565/585	650/675	793/852	
Viscosity					
At 100°F, cSt		3.042	9.455	47.63	
At 210°F, cSt		1.188	2.294	4.745	
Smoke Point, mm		14			
Freeze Point, °F		-94			

*Uncorrected for any loss in light ends.

TABLE VII

DOE CONTRACT EF-76-C-01-2315
FEEDS FOR HYDROCRACKING AND FCC TESTS
(HYDROTREATED 350-850°F PRODUCT OF 50/50
SRC-I/CREOSOTE OIL FROM PILOT PLANT
RUN 66-188)[1]

Identification	SGQ 6268	SGQ 6269
Inspections		
Gravity, °API	11.8	14.0
Aniline Point, °F	<32	53.9
Sulfur, ppm	52	74
Total Nitrogen, ppm	900	350
Metals, ppm	<1	<1
Hydrogen, Wt %	9.59	10.85
Carbon, Wt %	90.11	88.19
Oxygen, Wt %	0.35	0.18
Group Type, LV % (Mass Spectrometric 22-Component)		
Paraffins	0	0
Naphthenes	24.6	35.6
Aromatics	75.4	64.4
Viscosity cSt		
at 100°F	7.40	6.51
at 210°F	1.86	1.86
Hot C_7 Insolubles, ppm	64	<20
Ramsbottom Carbon, %	0.20	0.18
Benzene Insolubles, %	<0.03	<0.03
TBP Distillation, °F (Simulated by Chromatography)		
St/5	178/418	175/407
10/30	455/531	451/519
50	584	576
70/90	613/680	606/672
95/99	731/832	725/835

[1]Prepared by hydrotreating product fraction containing 2920 ppm nitrogen with ICR 106 catalyst at 0.5 LHSV, 2400 psia H_2 pressure. SGQ 6268 was prepared at 700°F, SGQ 6269 includes product prepared at both 650°F and 700°F.

TABLE VIII

DOE CONTRACT EF-76-C-01-2315
YIELDS FROM EXTINCTION RECYCLE HYDROCRACKING OF THE 350-850°F PRODUCT FRACTION OF HYDROTREATED 50/50 SRC-I/CREOSOTE OIL WITH ICR 106 CATALYST
PILOT PLANT RUN 81-11

Feed No.	SGQ 6269		SGQ 6268	
Feed Nitrogen, ppm	350		900	
Run Hr	1316-1352		1364-1412	
Average Cat. Temp., °F	790		798	
LHSV	1.00		1.00	
Per-Pass Conversion	25.99		13.52	
Total Pressure, psig	2341		2351	
H_2 Mean Pressure, psia	2095		2124	
Total Gas In, SCF/Bbl	8658		8430	
Recycle Gas, SCF/Bbl	7932		7919	
No Loss Prod. Yields	Wt, %	Vol, %	Wt, %	Vol, %
Methane	0.17		0.25	
Ethane	0.40		0.58	
Propane	1.99		2.09	
Isobutane	2.51	4.33	2.07	3.62
n-Butane	2.22	3.70	1.02	1.71
C_5-180°F	17.89	25.13	28.36	40.32
180-380°F	78.59	97.33	70.72	87.94
Total C_5+	96.48	122.46	99.08	128.25
Actual/No Loss Recovery	104.91/103.98		105.91/105.56	
H_2 Cons. (Gross), SCF/Bbl	2795		3777	
H_2 Cons. (Chemical), SCF/Bbl	2547		3613	
Whole Liquid Product Nitrogen, ppm	0.13		0.73	

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TABLE IX

DOE CONTRACT EF-76-C-01-2315
PRODUCT INSPECTIONS FROM EXTINCTION RECYCLE
HYDROCRACKING OF THE 350-850°F PRODUCT FRACTION
OF HYDROTREATED 50/50 SRC-I/CREOSOTE OIL WITH
ICR 106 CATALYST - PILOT PLANT RUN 81-11

Feed No.	SGQ 6269	SGQ 6268
Feed Nitrogen, ppm	350	900
Run Hr	1316-1352	1364-1412
Average Cat. Temp., °F	790	798
Product Inspections		
C_5-180°F Product		
Gravity, °API	72.9	72.2
Group Type, LV %		
(By Chromatography)		
Paraffins	58.2	
Naphthenes	39.1	
Aromatics	2.6	
Olefins	0.1	
Octane Number		
F-1 Clear	81.8	
180-380°F Product		
Gravity, °API	48.7	46.7
Aniline Point, °F	121.2	111.2
Group Type, LV %		
(Low Mass)		
Paraffins	24.7	18.7
Naphthenes	68.2	70.5
Aromatics	7.1	10.7
Octane Number		
F-1 Clear	55.3	60.2
ASTM D 86 Distillation, °F		
St/5	220/236	220/233
10/30	241/265	241/263
50	292	293
70/90	324/351	325/348
95/EP	358/382	355/380
% Overhead, LV %	99	99
TBP Distillation, °F		
(Simulated by Chromatography)		
St/5	160/195	157/182
10/30	203/249	197/242
50	297	287
70/90	343/375	333/377
95/99	384/395	388/399

TABLE X

DOE CONTRACT EF-76-C-01-2315
DISTRIBUTION OF C_5-180°F PRODUCT FROM
HYDROCRACKING OF 350-850°F HYDROTREATED
SRC-I/CREOSOTE OIL BLEND WITH ICR 106 CATALYST
PILOT PLANT RUN 81-11

Time Onstream, Hours	1316-1350
Average Catalyst Temperature, °F	790
Composition, LV % of C_5-180°F (By Chromatography)	
Isopentane	18.6
n-Pentane	11.0
2,2-Dimethylbutane	0.2
2,3-Dimethylbutane	1.3
2-Methylpentane	9.1
3-Methylpentane	6.1
n-Hexane	9.3
Isoheptane	2.6
Total Paraffins	58.2
Cyclopentane	2.4
Methylcyclopentane	27.2
Cyclohexane	7.8
Dimethylcyclopentanes, Ethylcyclopentane	1.6
Total Naphthenes	39.0
Benzene	2.6
Total Aromatics	2.6
C_5-C_7 Olefins	0.1
Total Olefins	0.1
Octane Number, F-1 Clear (Observed)	81.8
Isopentane/n-Pentane	1.7
Iso-C_6/n-Hexane	1.8

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TABLE XI

DOE CONTRACT EF-76-C-01-2315
CATALYTIC CRACKING OF HYDROPROCESSED SRC-I/CREOSOTE OIL
CRACKING CONDITIONS AND YIELDS

Feed	SGQ-6268		SGQ-6269	
Catalyst	CCL-4914 Equilibrium Catalyst (CBZ-1)			
Run Conditions				
Reactor Temperature, °F	975		975	
WHSV	13.20		12.93	
C/O Ratio	0.910		0.928	
Severity	0.069		0.072	
Run No., FCT 5-	1134		1133	
Conversion (430°F), Wt %/LV %	46.40	48.62	59.05	61.69
Yields	Wt %	LV %	Wt %	LV %
Coke	1.01		0.99	
H_2	0.08		0.08	
Methane	0.23		0.33	
Ethane	0.20		0.29	
Ethylene	0.35		0.51	
Total C_2^- Gas	0.86		1.21	
Propane	0.94	1.82	1.62	3.10
Propylene	1.64	3.10	2.04	3.80
Total C_3's	2.58	4.92	3.66	6.90
Isobutane	1.77	3.11	2.35	4.06
n-Butane	0.67	1.13	0.78	1.30
C_4 Olefins	1.24	2.01	0.85	1.36
Total C_4's	3.68	6.25	3.98	6.72
Light Gasoline (C_5-250°F)	19.82	26.65	25.89	34.56
Heavy Gasoline (250-430°F)	18.44	20.72	23.32	25.45
Total Gasoline (C_5-430°F)	38.26	47.37	49.21	60.01
Light Cycle Oil (430-625°F)	37.60	37.34	27.46	26.76
Heavy Cycle Oil (625°F+)	16.00	14.04	13.49	11.55
Total Cycle Oil (430°F+)	53.60	51.38	40.95	38.31

TABLE XII

DOE CONTRACT EF-76-C-01-2315

CATALYTIC CRACKING OF HYDROPROCESSED SRC-I/CREOSOTE OILS - PRODUCT INSPECTIONS

Run Feed	5-1134 SGQ 6268	5-1133 SGQ 6269	5-1134 SGQ 6268	5-1133 SGQ 6269
	Light Gasoline (C_5-250°F)		Heavy Gasoline (250-430°F)	
Product Inspections				
Gravity, °API	52.4[1]	52.0[1]	29.5	27.3
Sulfur, ppm	-	-	15	20
Nitrogen, ppm	10	6.4	235	35
Bromine Number	19	15	8.1	4.9
Aniline Point, °F	82.2	77.6	<32	<32
FIAM (Group Type), LV %				
Paraffins + Naphthenes	77	76	35	26
Olefins	8	7	2	1
Aromatics	15	17	63	73
Octane Numbers				
F-1 Clear		90.4		97.5
F-2 Clear	80.3	79.5	82.0	84.9
	Light Cycle Oil (430-625°F)		Heavy Cycle Oil (625°F+)	
Gravity, °API	10.8	10.3	1.1249[2]	1.1350[2]
Sulfur, ppm	10	20	100	110
Nitrogen, ppm	75	26	154	185
Bromine Number	6.7	6.1	-	-
Aniline Point, °F	<32	<32	-	-
Pour Point, °F	<-80	-75	+70	+95
Ramsbottom Carbon, %			1.42	1.91
Viscosity, SUS				
At 100°F	38.50	36.91		-
At 130°F	33.21	33.64		88.17
FIAM (Group Type), LV %				
Paraffins + Naphthenes	10	13		
Olefins + Aromatics	90	87		

[1]Measured on 140-250°F fraction.
[2]Specific Gravity (60/60°F).

TABLE XIII

DOE CONTRACT EF-76-C-01-2315
PROPERTIES OF SRC-II PROCESS PRODUCTS

	Naphtha	Light Distillate	Heavy Distillate
Wt % of Total SRC-II Product	26	63	11
Chevron Identification	WOW 3631	WOW 3632	WOW 3633
Inspections			
Gravity, °API	40.2	13.7	-0.8
Aniline Point, °F	60.2	<32	Too Dark
Sulfur, Wt %	0.42	0.18/0.19	0.40
Total Nitrogen, Wt %	0.52	1.03	1.25
Basic Nitrogen, Wt %	0.28	0.82	0.66
Oxygen, Wt %	2.29	3.73	1.91
Carbon, Wt %	Incomplete	(82.71)	88.63
Hydrogen, Wt %	9.84	8.25	7.29
Hydrogen/Carbon Atom Ratio	Incomplete	(1.34)	0.98
Chloride, ppm	3.7	58	11
Pour Point, °F		-65	+10
Group Type, LV %	Low Mass	High Mass	22 Component
Paraffins	23.7	4.5	2.1
Naphthenes	45.7	17.9	10.8
Aromatics	31.1	77.7	77.4
Sulfur Compounds	-		9.7
Ramsbottom Carbon, Wt %			1.20
Hot Heptane Asphaltenes, Wt %			0.94
Benzene Insolubles, Wt %			0.14/0.15
Refractive Index (80°C)	1.4350	1.5165	1.5648
Ash, Wt %	120		0.06
Molecular Weight		158	228
Bromine Number	46	62	34
Viscosity, cSt			
at 100°F	0.8022	3.144	
at 210°F			4.085
ASTM Distillation, °F	D 86	D 86	D 1160
St/5	186/186	360/396	530/577
10/30	200/242	401/418	591/628
50	282	435	665
70/90	316/362	457/498	716/855
95/EP	400/465	516/558	884/958
% Overhead (Excl. Trap)	98.5	99	98
% in Flask	1.0	1	2
% Trap	0.5	0	0
TBP Distillation, °F (Simulated by Chromatography)			
St/5	41/133	216/357	248/519
10/30	161/226	382/426	543/597
50	287	464	639
70/90	344/408	506/559	693/788
95/99	445/620	576/653	844/955

TABLE XIV

DOE CONTRACT EF-76-C-01-2315
METALS IN SRC-II PROCESS PRODUCTS

	Naphtha	Light Distillate	Heavy Distillate
Wt % of Total SRC-II Product	26	63	11
Chevron Identification	WOW 3631	WOW 3632	WOW 3633
Inspections			
Metals by Emission Spec., ppm			
Aluminum	0.1	0.3	47
Boron	-	-	2.0
Barium	-	-	0.2
Calcium	0.04	0.06	10
Chromium	0.07	0.17	4.9
Copper	-	0.01	0.2
Iron	0.3	0.8	54
Magnesium	0.02	-	2.9
Molybdenum	-	-	0.3
Sodium	0.3	0.13	16
Nickel	-	0.08	1.2
Silicon	0.16	0.3	27
Titanium	-	1.9	32
Vanadium	-	-	1.7
Other Metals Analysis, ppm			
Arsenic			0.03

TABLE XV

DOE CONTRACT EF-76-C-01-2315
PROPERTIES OF WHOLE SRC-II PROCESS PRODUCTS BLEND
(26% NAPHTHA, 63% LIGHT DISTILLATE,
11% HEAVY DISTILLATE)

Chevron Identification	WOW 3666
Inspections*	
Gravity, °API	18.6
Aniline Point, °F	<30
Sulfur, Wt %	0.29
Total Nitrogen, Wt %	0.85
Basic Nitrogen, Wt %	0.7
Oxygen, Wt %	3.79
Carbon, Wt %	
Hydrogen, Wt %	
Hydrogen/Carbon Atom Ratio	
Chloride, ppm	
Pour Point, °F	Below -80
Group Type, LV %	
Paraffins	
Naphthenes	
Aromatics	
Sulfur Compounds	
Ramsbottom Carbon, Wt %	0.70
Hot Heptane Asphaltenes, ppm	468
Benzene Insolubles, Wt %	<0.03
Refractive Index (80°C)	1.5073
Ash, Wt %	0.004
Molecular Weight	132
Bromine Number	70
Viscosity, cSt	
at 100°F	2.196
at 130°F	1.617
ASTM Distillation, °F	D 86/D 1160
St/5	154/217
10/30	281/382
50	438
70/90	484/597
95/EP	699/850
% Overhead (Excl. Trap)	98
% in Flask	0
% Trap	2
TBP Distillation, °F (Simulated by Chromatography)	
St/5	56/189
10/30	241/379
50	424
70/90	473/562
95/99	642/820

*Analyses not shown are incomplete.

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TABLE XVI

DOE CONTRACT EF-76-C-01-2315
METALS IN SRC-II PROCESS PRODUCT BLEND, WOW 3666

	Analysis of Blend*	Calculated from Analyses of Components
Inspections		
Metals by Emission Spec., ppm		
Aluminum	7	5.4
Boron	-	0.2
Calcium	0.7	1.2
Chromium	1.0	0.7
Iron	12.2	6.5
Magnesium	0.5	0.3
Sodium	-	2
Nickel	0.3	0.2
Silicon	- (?)	3.2
Titanium	- (?)	5
Vanadium	-	0.2
Other Metals Analysis, ppm		
Arsenic	Incomplete	

*This analysis is being repeated.

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TABLE XVII

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
WHOLE LIQUID PRODUCT PROPERTIES
(PRELIMINARY RESULTS)
RUN 76-165
750°F - ∿2300 PSIA H_2

Time On Stream, Hr LHSV	131, 203* 0.5	419 1.0	467 1.5
Inspections			
Gravity, °API	39.4	37.0	
Aniline Point, °F	120.5		
Sulfur, ppm	(50)	10	
Nitrogen, ppm	0.19	0.4	18
Hydrogen, Wt %	13.75		
Carbon, Wt %	86.10		
TBP Distillation, °F (Simulated by Chromatography)			
ST/5	58/180	62/177	61/174
10/30	214/272	210/278	203/275
50	362	364	368
70/90	405/495	419/505	435/518
95/99	538/631	553/662	572/789

*Nitrogen and TBP distillation at 203 hr, other analyses at 131 hr.

7-13-78 RFS RE 780529-1

TABLE XVIII

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-II
WITH ICR 106 CATALYST
PROPERTIES OF PRODUCT FRACTIONS
(PRELIMINARY RESULTS)
750°F - 0.5 LHSV - ∿2300 PSIA H_2
PILOT PLANT RUN 76-165 AT 83-107

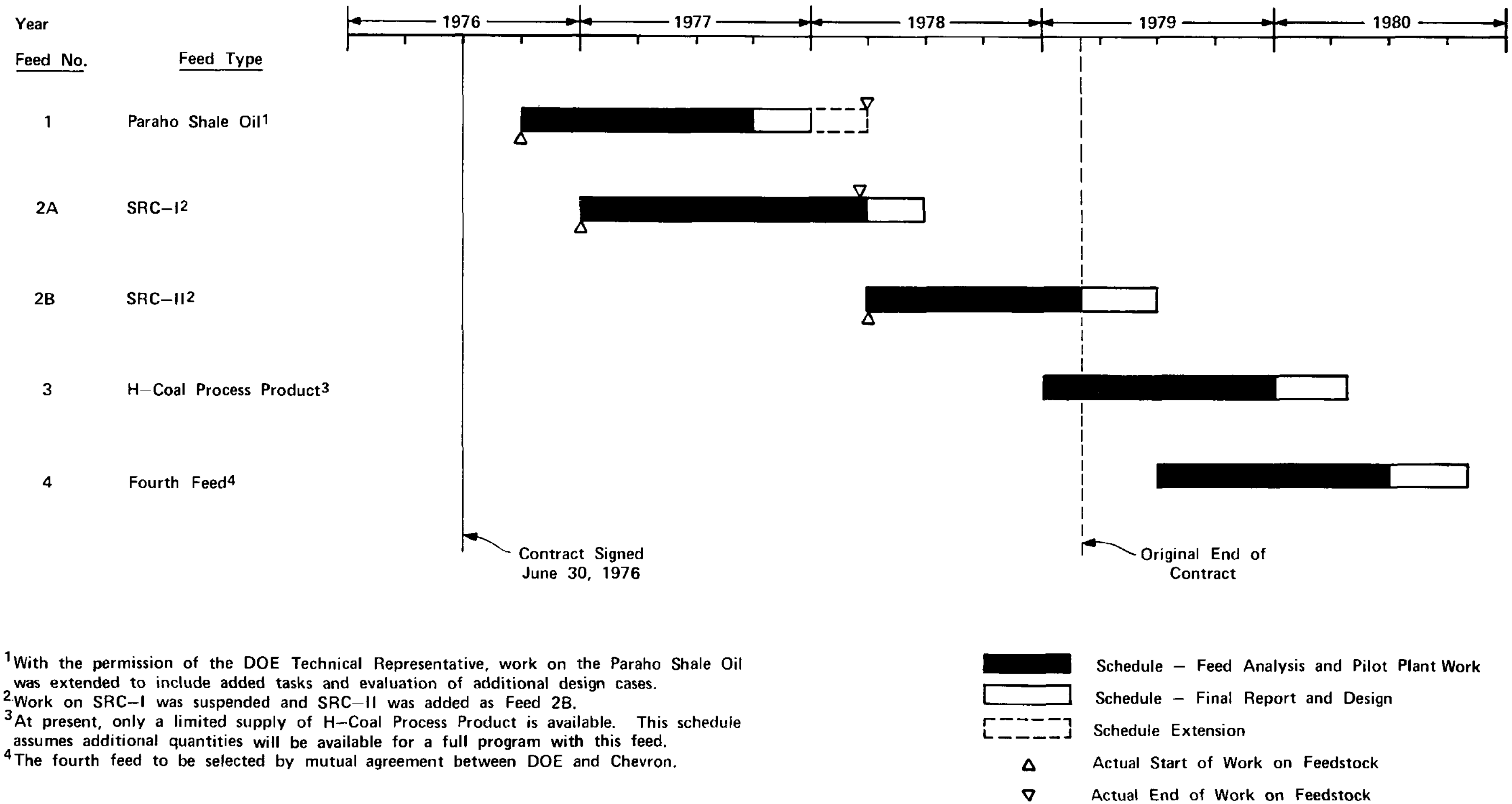
180-350°F Product[1]	
Gravity, °API	50.2
Aniline Point, °F	114.3
Octane No, F-1 Clear	59.5
350°F+ Product	
Gravity, °API	32.8
Aniline Point, °F	129.3
Cetane No.	36.2
Freeze Point, °F	-62
Viscosity	
cSt at 100°F	2.087
cSt at 210°F	0.9370
TBP Distillation, °F (Simulated by Chromatography	
ST/5	309/352
10/30	362/388
50	413
70/90	460/519
95/99	561/634

[1]Group type of 180-350°F product: Paraffins, 5.7 LV %, Naphthenes, 93.6 LV %, and Aromatics, 0.7 LV %.

7-14-78 RFS RE 780531

FIGURE 1

DOE CONTRACT EF–76–C–01–2315
FEED TIMING SCHEDULE (REVISED, JULY 1978)

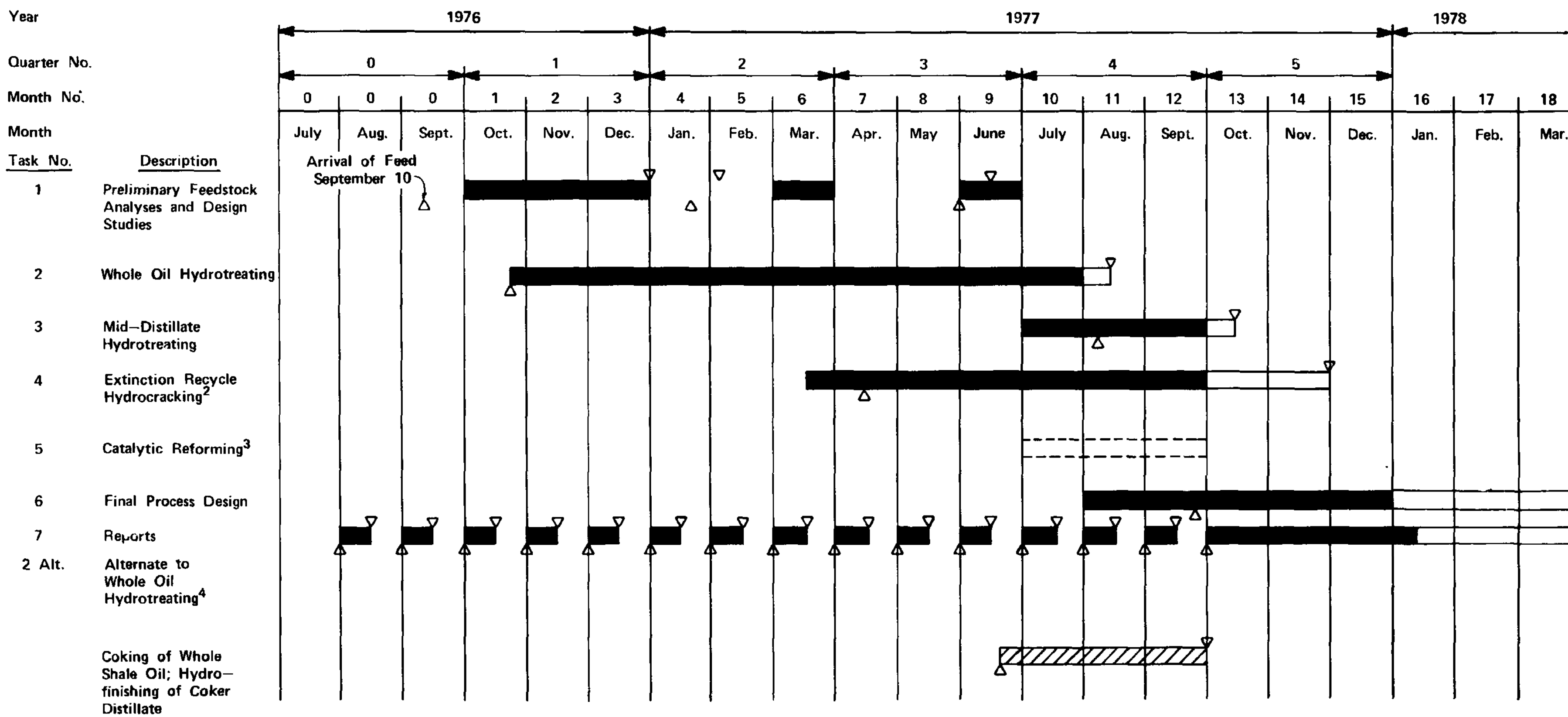


[1] With the permission of the DOE Technical Representative, work on the Paraho Shale Oil was extended to include added tasks and evaluation of additional design cases.
[2] Work on SRC–I was suspended and SRC–II was added as Feed 2B.
[3] At present, only a limited supply of H–Coal Process Product is available. This schedule assumes additional quantities will be available for a full program with this feed.
[4] The fourth feed to be selected by mutual agreement between DOE and Chevron.

7-7-78 RFS RD 780524

FIGURE 2

DOE CONTRACT EF–76–C–01–2315
REVISED SCHEDULE AS APPLIED TO
FIRST FEED (PARAHO SHALE OIL)[1]



[1]In addition to the seven major tasks, an added task (Task 8) "Distillate Shale Oil Hydrotreating" was performed in August and September, 1977, as a result of a contract modification A004.
[2]Also includes Task 4A – Alternate Processing (FCC).
[3]To be included only if deemed appropriate by the mutual agreement of the DOE Technical Representative and Chevron Research, subject of a contract modification.
[4]Described in last paragraph of Task 2, work statement.

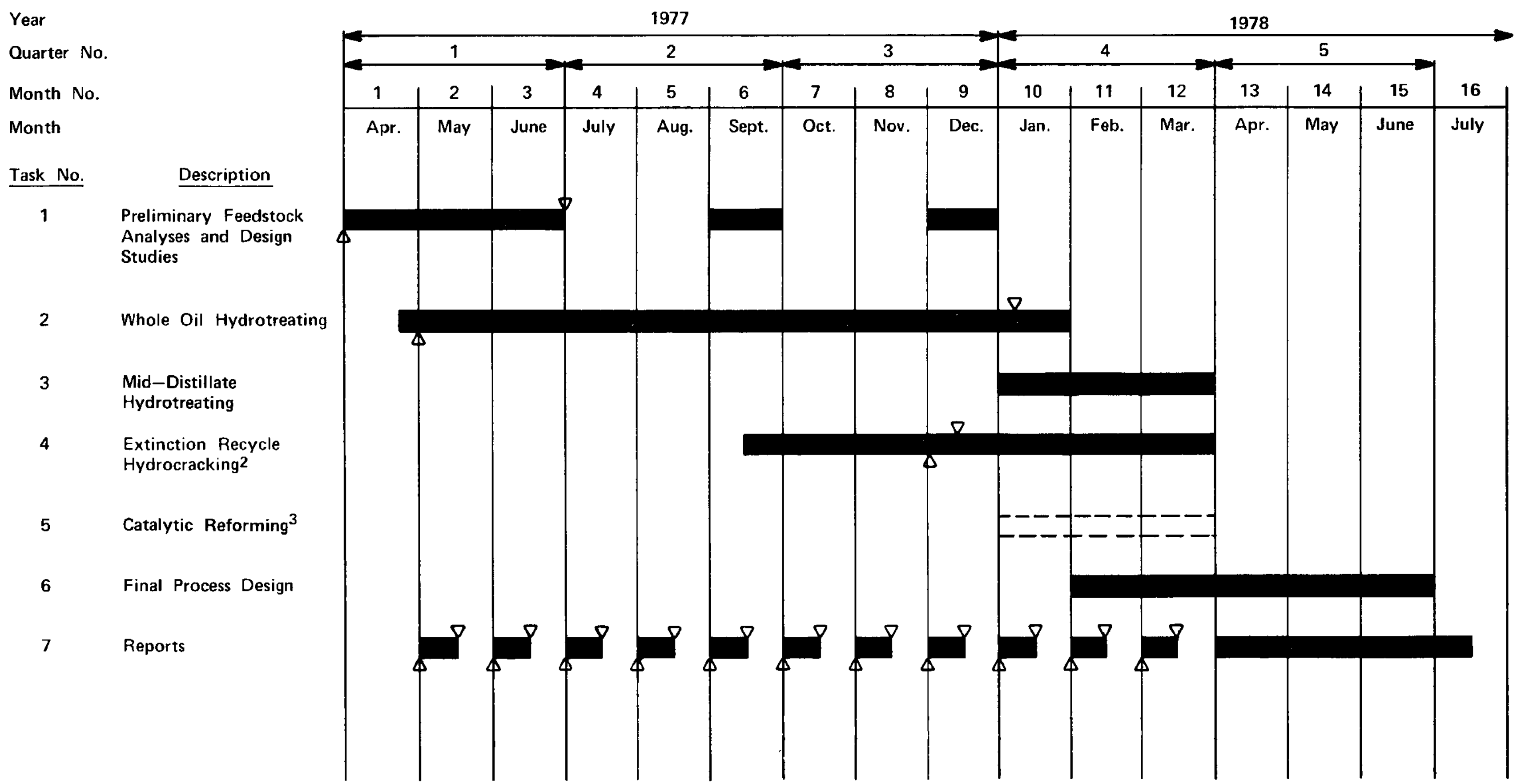
Schedule
Possible Task
Added Task
Extende´ Task
Δ Start Task
∇ Complete Task

3-29-78

RFS RD 773938-3

FIGURE 3

DOE CONTRACT EF-76-C-01-2315
SCHEDULE FOR FEED 2A (SRC-I)[1]



[1]With the concurrence of the DOE Technical Representative, work on SRC-I was suspended.
[2]Also includes Task 4A - Alternate Processing (FCC).
[3]To be included only if deemed appropriate by the mutual agreement of DOE Technical Representative and Chevron Research, subject of a contract modification.

Schedule
Possible Task
Δ Start Task
∇ Complete Task

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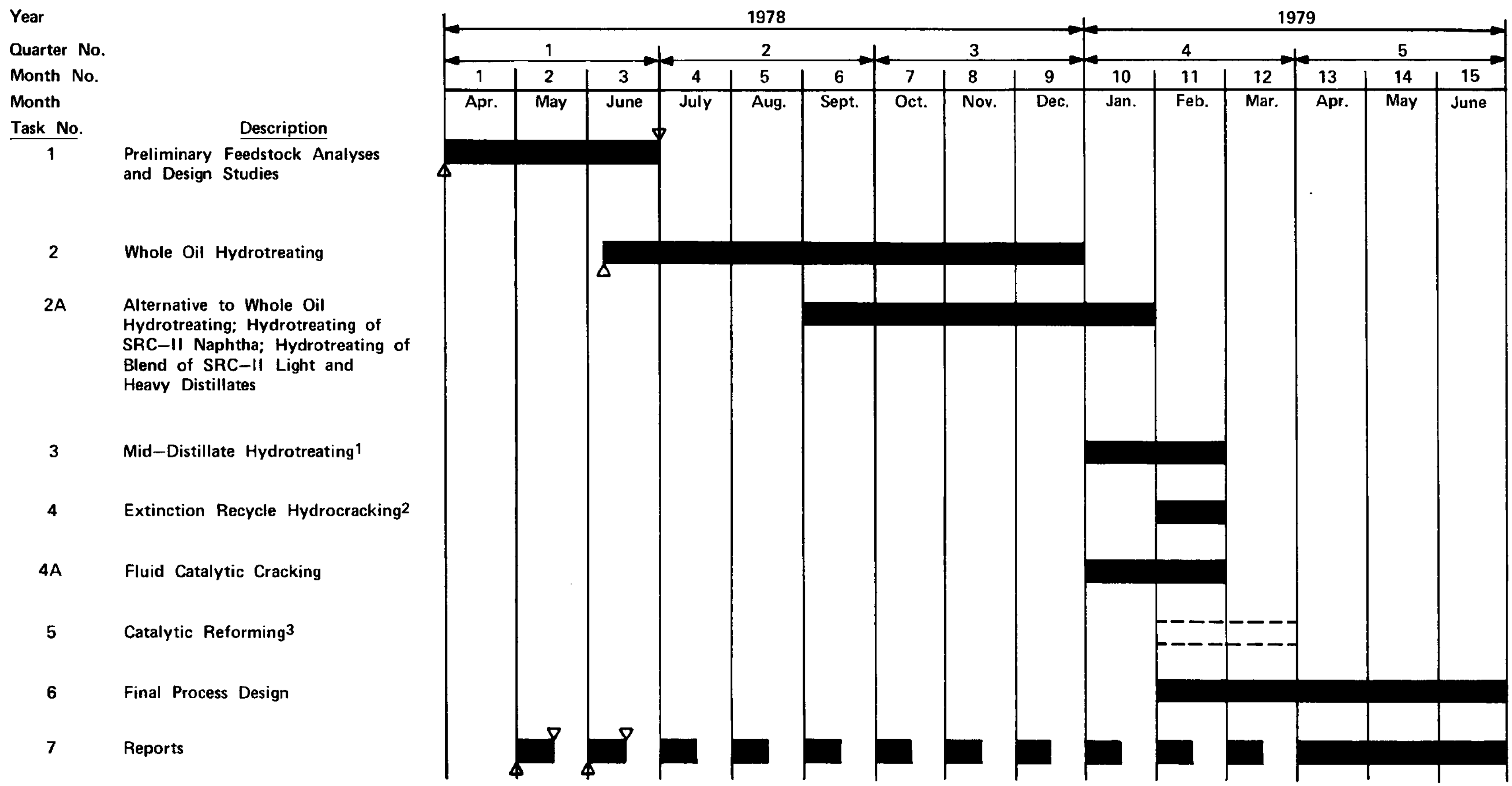
FIGURE 4

DOE CONTRACT EF-76-C-01-2315
SCHEDULE FOR FEED 2B (SRC-II)

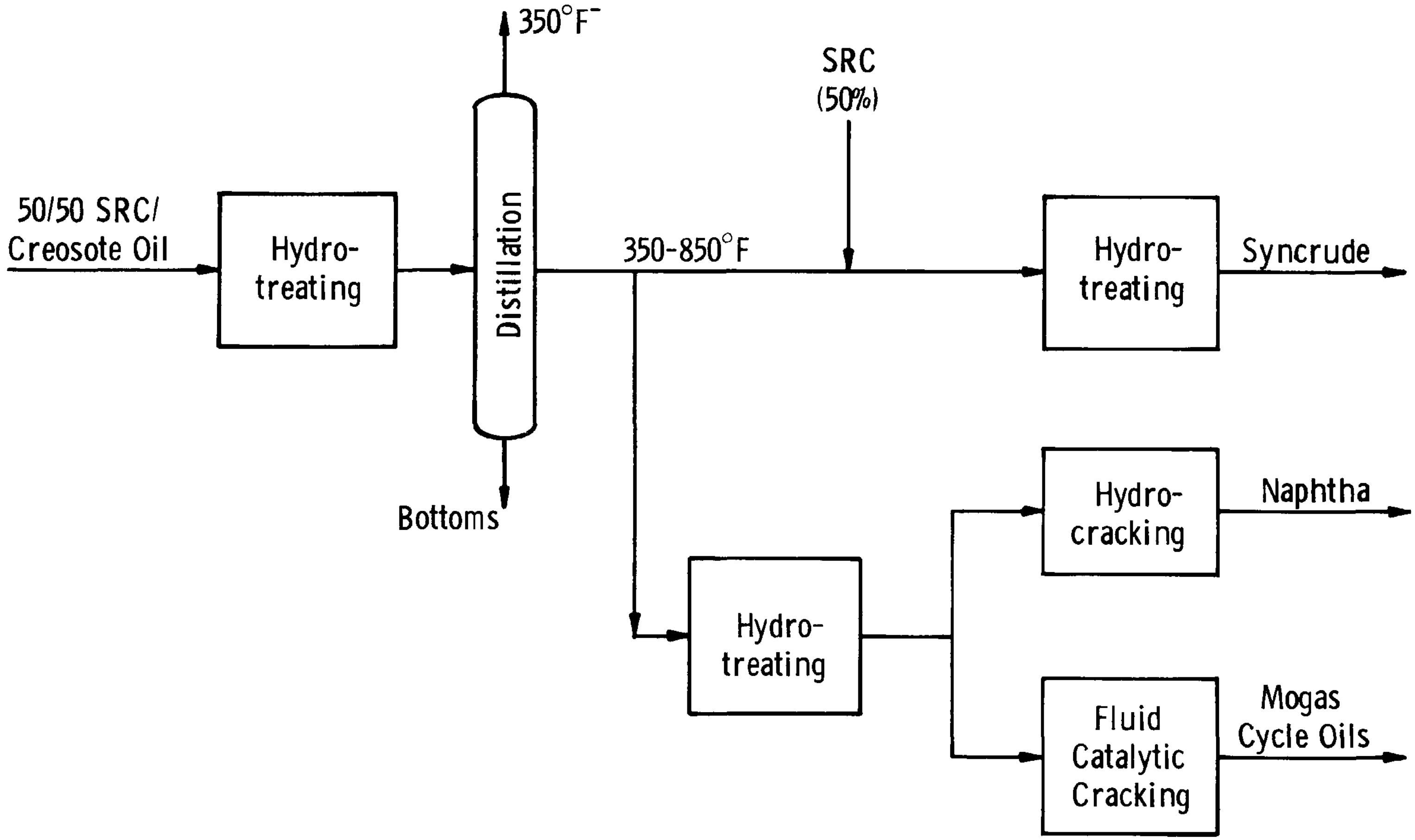


[1]Task 3 may not be necessary, depending on the outcome of Tasks 2 and 2A.
[2]Only a minimum hydrocracking program is anticipated.
[3]Task 5 is to be included only if deemed appropriate by mutual agreement of the DOE Technical Representative and Chevron Research, subject of a contract modification.

Schedule
Possible Task
Δ Start Task
∇ Complete Task

FIGURE 5

DOE CONTRACT EF-76-C-01-2315
PILOT PLANT TESTS
PROCESSING OF SRC-1



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