

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

Quarterly Report for the
Period January-March 1979

R. F. Sullivan

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CHEVRON RESEARCH COMPANY
Richmond, California

Date Published April 1979

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Under Contract No. EF-76-C-01-2315

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I. Abstract

Pilot plant tests on the hydrotreating of SRC-II process product indicate that this coal-derived feed is suitable for refining using advanced commercial petroleum processing technology. Experiments are in progress to evaluate several different combinations of refinery processes for conversion of SRC-II to transportation fuels. Nitrogen in the whole SRC-II process product can be reduced to a concentration of less than 0.5 ppm in a single catalytic stage. Sulfur and oxygen can also be reduced to low levels; and at high severity, most of the aromatic compounds are converted to naphthenes. The naphtha appears to be an excellent catalytic reformer feed, and the middle distillate meets the smoke point and stability specifications for jet fuel. As the processing severity is decreased, product nitrogen increases; and the product becomes more aromatic. In the latter case, further hydrotreating of the naphtha is required before it can be fed to the second stage of a catalytic reformer. Depending on the severity employed, the jet boiling range product must be further hydrogenated for specification jet fuel. Experiments were made to determine appropriate conditions for these processing steps. Alternate processing schemes being investigated include hydrocracking and catalytic cracking.

Encl. - See Index of Enclosures

II. Contract Objectives and Scope of Work

The objective of the program as originally defined 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 in April 1978. 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 devoted to the processing of SRC-II product will be completed within the next quarter. Work is now starting on the third feed, H-Coal process product, supplied by Hydrocarbon Research, Inc. 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 from which 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 technology 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 postponements of the program beyond the original schedule. The schedule for the second feedstock, solvent-refined coal, was further altered because SRC-II was added to the program as Feedstock 2B after completion of pilot plant tests on SRC-I, now referred to as Feedstock 2A.

Figure 1 shows the work completed thus far and the anticipated timing for Feedstocks 2B, 3, and 4. In a recent contract modification, the date for completion of the contract was extended to February 27, 1981, as shown in the figure.

Figure 2 is the schedule for processing Feed 2B, SRC-II.

Work on Feedstock 3, H-Coal process product, is starting. Sufficient feedstocks to meet the anticipated requirements for the program were received from Hydrocarbon Research, Inc., on March 23, 1979.

IV. Description of Technical
Progress - SRC-II Processing
(Feedstock 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 Pittsburgh 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)

The inspections of these samples were summarized in the April-June 1978 Quarterly Report (FE-2315-28).

Table I shows 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. Also shown in Table I are properties of the start-400°F naphtha and the 400°F+ gas oil fractions used as feedstocks in Task 2A. The analyses for chloride have been revised from the numbers originally reported. The original chloride analyses were incorrect.

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

As reported previously (FE-2315-34), the two pilot plant runs for the hydrotreating of whole SRC-II process product are now complete. The first was a 3030-hour run using ICR 106 catalyst containing nickel, tungsten, silica, and alumina. The second was a 2650-hour test using ICR 113 catalyst containing nickel, molybdenum, silica, and alumina.

Both runs were terminated voluntarily, and the catalysts were still active. Details of catalyst performance, yields, hydrogen consumptions, and product properties have been reported.

Of particular interest are possible cases in which jet fuel is the principal product. Inspections and yields were previously reported in which the product was distilled to prepare a 300-500°F jet boiling range fraction. If the boiling range is expanded to 300-550°F, a higher yield of jet fuel can be obtained. Therefore, additional distillations were made to obtain 300-500°F fractions. Yields and inspections for products distilled to give this fraction are shown in Tables II-VI, inclusive, at several sets of conditions with ICR 106 catalyst. The inspections show that the boiling range could be further expanded if maximum jet fuel is desired.

It was previously shown that the 300°F+ fraction of hydrotreated SRC-II containing 13% aromatics and 0.1 ppm nitrogen passes the stringent JFTOT thermal stability test for jet fuel at both the ASTM specification temperature of 260°C and the more severe test temperature of 280°C. Additional results for less severely hydrotreated samples are shown below:

JFTOT Analyses of Hydrotreated SRC-II Product

Boiling Range, °F	Product Nitrogen, ppm	Product Aromatics, LV %	JFTOT Rating	
			260°C Test, No. Rating	280°C Test, No. Rating
300+	0.1	13	1	1
300-550	0.1	27	1	4
300+	2.3	37	4	4

Note that the sample with 27% aromatics passes the 260°C test but no longer passes the more severe test. (A No. 4 rating is not acceptable.) The third sample fails the test at both temperatures. It is possible that if 550°F+ material were not present in this sample, the rating would be somewhat better. However, because aromatics levels above 25% are unacceptable for jet fuel, the latter sample would require more severe hydrotreatment to make acceptable jet fuel regardless of the JFTOT rating.

Among the cases being considered for SRC-II processing is a case in which a fuel such as No. 2 heating oil is a principal product. It appears that the 300°F+ fraction of partially hydrotreated SRC-II will pass most of the ASTM specifications for No. 2 heating oil except for gravity. The 1978 ASTM minimum specification for gravity is 30; if the 300°F+ fraction of SRC-II is hydrotreated to 30 gravity, it becomes acceptable jet fuel and would not be marketed as heating oil; heating oil from SRC-II is only acceptable for applications in which the low gravity is not a problem. Because of the high heating value on a volumetric basis, the low gravity may be advantageous in some applications.

Another analysis of interest for heating oil is a thermal stability test. Although there is no ASTM requirement, the test used in these studies is a Chevron adaption of Du Pont Test Method 21-61.* In Du Pont's method, a test blotter is used to rate the stability. However, because evaluation of the result is visual and subjective, Chevron has found that better repeatability is obtained by evaluating the test filters using a reflectometer. As a reference point, a reflectance of 80% or higher is recommended as a passing rating for automotive diesels. (This is comparable to a test blotter rating of No. 6 or below by the Du Pont method.) For some railroad diesels, a reflectance of 70% or above is acceptable. (The latter rating compares to a test blotter rating of No. 7 or below by the Du Pont method.)

Results for a variety of partially hydrotreated samples from SRC-II are given in Table VII. The samples selected include (1) the 300+ fraction of hydrotreated SRC-II blends at nitrogen concentrations of 13, 173, and 650 ppm, (2) the 550°F+ fraction at two nitrogen levels at 17 and 176 ppm (the molecules causing any thermal stability problems would be expected to be concentrated in this fraction), and (3) C₅+, 300°F+, and 350°F+ product at about 250 ppm nitrogen.

The stability results shown in Table VII are encouraging. No problems were observed in the 300°F+ samples up to 650 ppm nitrogen. The 550°F+ results are favorable if it is remembered that this fraction constitutes only about 10% of the total 300°F+ material.

*Du Pont Petroleum Laboratory Test Methods, E. I. du Pont de Nemours and Company, Wilmington, Delaware, June 1967

Task 2A - Alternate to
Whole Oil Hydrotreating:
Separate Hydrotreating of
SRC-II Naphtha and 400°F+
SRC-II Gas Oil

Runs were made in which the 400°F- SRC-II naphtha and the 400°F+ gas oil fractions were processed separately. Details of these runs were presented in the most recent quarterly report (FE-2315-34). Yield and inspections available to date are shown in Tables VIII-XIV, inclusive of the present report.

Task 3 - Naphtha and Middle
Distillate Hydrotreating

A. Naphtha Hydrotreater

Run 72-326 is a pilot plant run with ICR 113 catalyst containing nickel, molybdenum, silica, and alumina. In the first part of this run, the naphtha fraction from partially hydrotreated whole SRC-II was further hydrotreated to remove the remaining nitrogen. Inspections of the two feeds tested are shown in Table XV. The first feed, SGQ 6502, is a Start-400°F fraction containing 255 ppm nitrogen and 31% aromatics. The second feed, SGQ 6504, is a Start-450°F fraction containing 382 ppm nitrogen and 45% aromatics.

The run conditions were varied in the following ranges:

700-725°F average catalyst temperature

650-800 psig total pressure

500-650 psia hydrogen pressure

2.0 LHSV

3000 SCF/bbl recycle gas rate

Results of several yield periods, together with run conditions, are shown in Tables XVI and XVII. In particular, note that in most of the tests, nitrogen is removed to a low concentration; hydrogen consumption is low; and there is little saturation of aromatics.

B. Jet Hydrotreating

Two jet hydrotreating runs were made. Run 72-326 included a period in which jet boiling range (300-550°F) partially hydrotreated SRC-II was further hydrotreated with ICR 113 catalyst containing nickel, molybdenum, silica, and alumina; Run 76-168 was a similar study with ICR 106 containing nickel, tungsten, silica, and alumina.

Table XVIII shows the properties of the feedstocks used in these studies. The first, SGQ 6547, contains 12 ppm nitrogen and 56% aromatics; the second, SGQ 6581, contains 173 ppm nitrogen and 63% aromatics.

Run conditions were varied in the following range in order to select appropriate conditions for aromatics saturation.

700-775°F average catalyst temperature

1250-2100 psig total pressure

1200-1900 psia hydrogen pressure

1.0-2.0 LHSV

5600 SCF/bbl recycle gas rate

Results are given in Tables XIX-XXII, inclusive.

The results show:

1. The nitrogen is removed to concentrations well below 1 ppm with both catalysts at all conditions studied.
2. At none of the conditions tested with ICR 113 were sufficient aromatics hydrogenated to make acceptable jet fuel. (High hydrogen pressures would be required.)
3. ICR 106 makes acceptable jet fuel from the feed containing 173 ppm nitrogen and 63% aromatics at the following conditions:

2.0 LHSV

1500 psia H₂ pressure

725°F

At these conditions, the resulting product has a smoke point of 22 mm and contains 14% aromatics. The amount of cracking to 300°F- is low (7%). Hydrogen consumption is 1400 SCF/bbl.

4. With the easier feed (12 ppm nitrogen, 56% aromatics), acceptable jet fuel was obtained at a lower pressure. Conditions were:

1.0 LHSV

1225 psia H₂ pressure

725°F

At these conditions, the product contains 16% aromatics. Much cracking occurs (33%), and the hydrogen consumption is high (1650 SCF/bbl).

Task 4 - Hydrocracking
of 350°F+ SRC-II

Preliminary engineering evaluations indicate that if maximum gasoline production is desired, hydrocracking of partially hydrotreated SRC-II may be an attractive route. A brief pilot plant study is being made to determine appropriate processing conditions and catalysts. Results are incomplete.

Task 4A - Catalytic
Cracking of Partially
Hydrotreated SRC-II

Runs were made in which three hydrotreated SRC-II fractions (350°F+, 400°F+, and 450°F+) were catalytically cracked using an equilibrium catalyst withdrawn from an operating FCC unit. Feed inspections are given in Table XXIII. Results will be reported when calculations are complete.

V. Conclusions and Program

Results on hydrotreating of SRC-II process product with ICR 106 and ICR 113 catalysts are encouraging. Nitrogen removal is less difficult than anticipated; product containing less than 0.5 ppm nitrogen can be obtained in a single catalytic stage. Sulfur and oxygen also can be reduced to low levels; and at high severity, most of the aromatic compounds are converted to naphthenes. As the processing severity is decreased, product nitrogen increases; and the product becomes more aromatic. Of particular interest is the result that at high severity the product includes a jet fraction that passes the stringent JFTOT thermal stability test and appears to be of premium quality. The naphtha product should be an excellent feed to a catalytic reformer.

At comparable conditions, ICR 106 is more active for nitrogen removal and hydrogenation than is ICR 113. Also, ICR 113 deactivates at a

somewhat higher rate. However, ICR 113 is a less expensive catalyst; and the catalyst of choice will depend on the particular refining scheme selected.

Naphtha from the lower severity hydrotreating tests was further hydrotreated using ICR 113 catalyst to make a premium feed for the second stage of a catalytic reformer.

Tests were made in which the 300-550°F product from the lower severity hydrotreating tests were further hydrotreated using either ICR 113 or ICR 106 catalyst to determine the appropriate catalyst and conditions to make jet fuel. ICR 106 proved to be the catalyst of choice.

Experiments were also made in which the 400°F+ SRC-II is initially hydrotreated separately from the naphtha. Based on our results, we see little incentive for this alternative processing route.

Process Engineering studies are being made to evaluate our pilot plant results and to make recommendations for future tests to develop cases for study. The results to date suggest five cases that may be attractive:

1. A single-stage hydrotreater, operated at very high severity with ICR 106 catalyst directly producing reformer feed and acceptable jet fuel.
2. Less severe hydrotreating with ICR 106 to produce naphtha which must be further hydrotreated before reforming and jet fuel which must be further hydrotreated to make smoke point and other specifications.
3. Hydrotreating at lower severity with ICR 113 catalyst to produce a naphtha which must be further hydrotreated before reforming and a feed for a fluid catalytic cracker for gasoline production.
4. Hydrotreating followed by hydrocracking for maximum gasoline production.
5. Hydrotreating at lower severity with ICR 113 catalyst to produce a fuel such as No. 2 heating oil and a naphtha to be further hydrotreated before reforming.

The addition of the hydrocracking case to the other cases being considered extended our pilot plant testing a few weeks longer than predicted last quarter. We expect to complete the hydrocracking tests during April.

During the next quarter, we expect to complete the process engineering and cost studies for the processing of SRC-II. An interim report describing these studies is scheduled for issue at the end of the quarter.

We will start pilot plant studies on the processing of H-Coal process product during the next quarter.

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

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TABLE I

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

Description	Whole SRC-II	SRC-II Naphtha	400°F+ SRC-II
Chevron Identification	WOW 3666	WOW 3750	WOW 3751
Wt % of Whole SRC-II	100	29	71
<u>Inspections</u>			
Gravity, °API	18.6	36.2	10.7
Aniline Point, °F	<30	56.4	-
Sulfur, Wt %	0.29	0.26	0.25
Total Nitrogen, Wt %	0.85	0.42	0.99
Basic Nitrogen, Wt %	0.7		0.82
Oxygen, Wt %	3.79	3.51	3.63
Carbon, Wt %*	82.82	84.22	86.61/86.18
Hydrogen, Wt %*	9.24	12.23	8.77/9.54
Hydrogen/Carbon Atom Ratio*	1.33	1.73	1.21/1.32
Chloride, ppm	50	18/20	77/62/81
Pour Point, °F	Below -80	-	-50
<u>Group Type, LV %</u>			
Paraffins	-	23	-
Naphthenes	-	38	-
Olefins	-	5	-
Aromatics	-	34	-
Ramsbottom Carbon, Wt %	0.70	-	0.81
Hot Heptane Asphaltenes, ppm	468	-	2200
Benzene Insolubles, Wt %	<0.03	-	<0.03
Refractive Index (80°C)	1.5073	-	1.5340
Ash, Wt %	0.004	-	0.010
Molecular Weight	132	118	160
Bromine Number	70	49/52	69
<u>Viscosity, cSt</u>			
at 100°F	2.196	0.8933	4.213
at 130°F	1.617	-	-
at 210°F	-	-	1.238
<u>ASTM Distillation, °F</u>	D 86/D 1160	D 86	D 1160
St/5	154/217	151/179	393/413
10/30	281/382	197/249	427/448
50	438	290	471
70/90	484/597	332/366	506/590
95/EP	699/850	380/393	662/847
% Overhead (Excl. Trap)	98	98	99
% in Flask	0	1	1
% Trap	2	1	0
<u>TBP Distillation, °F</u> (Simulated by Chromatography)			
St/5	56/189	53/153	233/366
10/30	241/379	173/235	382/430
50	424	291	477
70/90	473/562	345/395	527/639
95/99	642/820	408/442	707/849

*Analysis being checked.

TABLE II

DOE CONTRACT EF-76-C-01-2315
 YIELDS FROM HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 PILOT PLANT RUN 76-165; FEED - WOW 3666

Run Hours	251-275*		539-563*		2,339-2,351		2,709-2,733	
Avg. Cat. Temp., °F	750		750		768		776	
LHSV	0.55		1.51		1.52		0.49	
Total Pressure, psig	2,499		2,501		2,500		2,492	
H ₂ Mean Pressure, psia	2,306		2,338		2,319		2,293	
Total Gas In, SCF/Bbl	17,054		16,938		17,452		18,889	
Recycle Gas, SCF/Bbl	13,752		14,827		15,353		15,811	
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
C ₁	0.17		0.08		0.12		0.22	
C ₂	0.29		0.16		0.20		0.37	
C ₃	0.37		0.17		0.24		0.53	
iC ₄	0.03	0.05	0.02	0.02	0.02	0.03	0.05	0.08
nC ₄	0.18	0.28	0.09	0.14	0.12	0.19	0.21	0.34
C ₅ -180°F	4.74	6.07	5.10	6.50	5.40	6.88	5.60	7.09
180-300°F	26.49	32.31	25.61	30.79	24.47	29.32	27.07	32.72
300-550°F	61.37	67.91	58.58	62.44	58.65	62.05	60.63	66.56
550°F-EP	5.81	6.11	7.86	7.97	8.42	8.34	4.45	4.66
Total C ₅ +	98.41	112.40	97.15	107.71	96.94	106.60	97.75	111.04
Actual/No Loss Recovery	105.44/105.01		100.16/103.23		99.41/103.20		103.36/104.70	
H ₂ Cons. (Gross), SCF/Bbl	3,301		2,111		2,098		3,078	
H ₂ Cons. (Chemical), SCF/Bbl	3,109		2,003		1,984		2,912	
Product Nitrogen, ppm	0.25		30		52		0.27	

*Product distillation for yield period at 251-275 hr, actually from a blend of product from 155-275 hr; for yield period at 539-563 hr, actually from blend of product from 431-563 hr.

TABLE III

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
WHOLE LIQUID PRODUCT PROPERTIES
PILOT PLANT RUN 76-165
~2300 PSIA H₂

Time Onstream, Hr	251-275	539-563	2339-2351	2709-2733
Avg. Catalyst Temp., °F	750	750	768	776
LHSV	0.55	1.51	1.52	0.49
Inspections of Whole Liquid Product				
Gravity, °API	39.3	34.1	32.7	38.4
Aniline Point, °F	116.9	67.5	54.8	105.4
Sulfur, ppm	5*	5*	21*	3*
Nitrogen, ppm	0.25	30	52	0.27
Hydrogen, Wt %	13.83*	11.99/13.37	11.60/12.14	13.88/13.28
Oxygen, ppm	40*	630*	680*	40*
Bromine Number	0.5	3.3/3.6		1.4/1.5
Chloride, ppm	0.2*	0.6*		
Group Type, LV % (High Mass)				
Paraffins	3.0*	4.1	4.5	3.9
Naphthenes	93.2	54.7	46.8	81.2
Aromatics	3.9	41.2	48.7	14.9
Density at 70°C		0.8115		0.7899
Molecular Weight	130*	134		136
Refractive Index (80°C)	1.4268*	1.4452		1.4276
TBP Distillation, °F (Simulated by Chromatography)				
St/5	65/181	59/175	66/179	65/181
10/30	215/276	209/279	213/286	215/277
50	365	370	380	364
70/90	410/499	435/518	442/528	410/498
95/99	546/648	570/685	581/705	543/657

*Results shown with an asterisk are not from these exact samples but from samples taken at about the same time and at the same processing conditions.

TABLE IV
DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
NAPHTHA PRODUCT PROPERTIES
PILOT PLANT RUN 76-165
~2300 PSIA H₂

Time Onstream, Hr	251-275	539-563	2339-2351	2709-2733
Avg. Catalyst Temp., °F	750	750	768	776
LHSV	0.55	1.51	1.52	0.49
Inspections, C ₅ -180°F Product				
Gravity, °API	60.7	59.9	59.9	58.6
Group Type, LV % (by Chromatography)	1	2		
Paraffins	31.2	30.0	32.8	26.2
Naphthenes	65.4	62.3	58.0	66.3
Aromatics	3.4	7.7	9.1	7.4
Olefins			0.1	0.1
Octane Number				
F-1 Clear	81.1	81.5	81.1	82.2
F-2 Clear	77.0	77.4		
180-300°F Product				
Gravity, °API	51.6	49.0	48.4	49.9
Aniline Point, °F	108.8	87.4	79.2	96.8
Nitrogen, ppm		2.1		
Oxygen, ppm		280		
Molecular Weight	107	107		
Octane No., F-1 Clear	66.0	69.2	72.0	68.4
Octane No., F-2 Clear	66.0	67.3	68.5	66.9
Group Type, LV % (Low Mass)				
Paraffins	7.1	10.1	11.3	10.1
Naphthenes	90.1	75.8	69.3	79.5
Aromatics	2.8	14.1	19.4	10.4
TBP Distillation, °F (Simulated by Chromatography)				
St/5	98/177	159/184	147/183	157/184
10/30	180/220	203/232	202/232	206/233
50	246	252	251	252
70/90	269/294	280/302	279/301	277/299
95/99	303/-	310/323	308/319	306/317
ASTM D 86 Distillation, °F				
St/5	212/216	214/217		213/217
10/30	220/228	220/228		221/228
50	235	236		235
70/90	246/266	247/270		246/265
95/EP	276/321	278/320		274/313
% Overhead	99.0	99		99

¹C₅-180°F PNA from sample at 275-299 hr.

²C₅-180°F PNA from sample at 563-587 hr.

TABLE V

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
300-550°F PRODUCT PROPERTIES
PILOT PLANT RUN 76-165
~2300 PSIA H₂

Time Onstream, Hr	251-275	539-563	2339-2351	2709-2733
Avg. Catalyst Temp., °F	750	750	768	776
LHSV	0.55	1.51	1.52	0.49
Inspections of 300-550°F Product				
Gravity, °API	34.6	28.5	27.3	33.3
Aniline Point, °F	122.3	60.3	41.8	110.2
Nitrogen, ppm		19		
Oxygen, ppm		970	1400	
Smoke Point, mm	22	11	11	20
Freeze Point, °F	<-94	-76	-76	-81
Group Type, LV % (High Mass)				
Paraffins	3.3	4.0	4.3	3.9
Naphthenes	92.6	47.1	36.7	81.1
Aromatics	4.0	48.8	59.0	15.0
TBP Distillation, °F (Simulated by Chromatography)				
St/5	266/304	264/313	281/318	277/312
10/30	316/365	332/382	334/386	329/373
50	404	419	421	403
70/90	451/504	460/510	459/507	442/497
95/99	520/560	526/550	522/544	516/542
ASTM D 86 Distillation, °F				
St/5	342/357	340/358	346/353	344/356
10/30	363/380	366/389	358/394	362/378
50	399	412	415	398
70/90	427/472	440/478	441/478	425/474
95/EP	488/531	494/531	491/516	495/533
% Overhead	99	99	99	99
Viscosity				
cSt at -40°F	12.79	13.17		11.92
cSt at -4°F	*	*	*	*
cSt at 100°F	*	1.591	*	1.640
Density at 70°C	0.8130	0.8443	0.8507	0.8182
Molecular Weight	155	154	152	146
Refractive Index at 80°C	1.4362	1.4611	1.4668	1.4407
Existent Gum, mg/100 ml				0
Flash Point, D 56, °F	132	142	142	134
Neutralization No. (Acid), mg/g	0.019	0.034	0.05	0.05
Napthalenes, %	0.11	0.94	1.35	0.22

*Incomplete.

TABLE VI

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 550°F+ PRODUCT PROPERTIES
 ~2300 PSIA H₂

Time Onstream, Hr	251-275	539-563	2339-2351	2709-2733
Avg. Catalyst Temp., °F	750	750	768	776
LHSV	0.55	1.51	1.52	0.49
Inspections of 550°F+ Product				
Gravity, °API	26.4	20.7	17.2	26.0
Aniline Point, °F	156.0	115.5	71.0	146.5
Nitrogen, ppm		35	68	
Oxygen, ppm		1600		
Group Type, LV % (High Mass)				
Paraffins	12.8	11.0	8.4	12.7
Naphthenes	81.1	59.3	45.3	74.9
Aromatics	6.0	29.7	46.4	12.4
Density at 70°C	0.8619	0.8934		0.8627
Molecular Weight	237	228		238
Refractive Index (80°C)	1.4625	1.4872		1.4659
TBP Distillation, °F (Simulated by Chromatography)				
St/5	398/534	378/528	427/539	346/518
10/30	550/582	547/588	551/586	541/579
50	604	616	608	602
70/90	625/655	648/685	630/658	624/653
95/99	668/694	700/722	669/691	664/681
Viscosity				
At 100°F, cSt	6.93	8.41	7.339	6.29
At 210°F, cSt	1.99	2.14		1.60

TABLE VII
DOE CONTRACT EF-76-C-01-2315
FUEL OIL/DIESEL THERMAL STABILITY OF HYDROTREATED SRC-II
FRACTIONS AT DIFFERENT NITROGEN CONCENTRATIONS

Sample Identification	SGQ 6608	SGQ 6609	SGQ 6610	SGQ 6548 ¹	SGQ 6582 ²	76-167 at 1657	76-167 at 1573	76-167 at 1585
Boiling Range, °F	300+	300+	300+	550+	550+	C ₅ +	300+	350+
Inspections								
Gravity	23.2	25.4	26.6	18.8	14.8	30.5	24.3	22.9
Nitrogen, ppm	650	173	13	17	176	282	238	232
Oxygen, %	0.71	0.26	0.07	0.13	0.26		0.26	
Group Type, LV % (High Mass)								
Paraffins	4.8	4.5	4.8	9.4	6.8	5.2	4.7	4.9
Naphthenes	28.3	33.6	41.1	51.6	45.6	39.6	33.5	30.7
Aromatics	66.9	61.9	54.1	39.0	47.6	55.2	61.8	64.4
Thermal Stability								
Reflectance, % After 90 Min. at 300°F	87.5	93.5	94.5	83.5	77.0	90.5	84.5	84.0
Filter Residue After Six- Hr O ₂ Blow	0	1	1	11	11	0	3	4
Color, As Is (ASTM D 1500)	<3.0	0.5	<0.5	<1.5	<1.5	2.0	<4.5 ³	<4.5 ³
Color, After Six-Hr O ₂ Blow	4.5	<2.0	<0.5	<2.0	4.0	<4.5	<7.0	<7.0
TBP Distillation, °F (Simulated by Chromatography)								
St/5				394/527	393/539	66/175	232/300	72/341
10/30				542/577	552/590	211/307	328/401	364/416
50				602	613	396	439	454
70/90				626/659	637/667	448/534	484/566	499/587
95/99				670/693	678/703	590/712	623/742	655/-

¹Bottom 10.4 LV % of SGQ 6610.

²Bottom 8.9 LV % of SGQ 6609.

³The samples shown in the last two columns are bottoms samples from batch distillations; they picked up considerable color during the topping operation.

TABLE VIII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II NAPHTHA WITH ICR 113 CATALYST
 PILOT PLANT RUN 65-194; FEED - WOW 3750

Run Hours	111-135	231-255		
Avg. Catalyst Temp., °F	692	699		
LHSV	1.03	0.95		
Total Pressure, psig	799	793		
H ₂ Mean Pressure, psia	642	645		
Total Gas In, SCF/Bbl	4391	4629		
Recycle Gas, SCF/Bbl	3282	3449		
	Wt %	Vol %	Wt %	Vol %
No Loss Product Yields				
C ₁	0.03		0.03	
C ₂	0.11		0.12	
C ₃	0.19		0.20	
iC ₄	0.01	0.02	0.02	0.02
nC ₄	0.10	0.14	0.10	0.15
C ₅ -EP, °F	96.75	103.00	96.83	102.46
Total C ₅ +	96.75	103.00	96.83	102.46
Actual/No Loss Recovery	92.33/101.90		91.71/102.02	
H ₂ Cons. (Gross), SCF/Bbl	1108		1108	
H ₂ Cons. (Chemical), SCF/Bbl	1056		1121	
Liquid Product Inspections				
Gravity, °API	46.9		45.8	
Aniline Point, °F	80.7		79.0	
Sulfur, ppm	11		11	
Nitrogen, ppm	0.21		<0.10	
Oxygen, ppm	90		90	
Chloride, ppm	0.5		0.6	
Octane Number				
F-1 Clear	69.4		69.2	
F-2 Clear	65.3		65.3	
Group Type, LV % (High Mass)				
Paraffins	18.4		16.8	
Naphthenes	61.4		61.3	
Aromatics	20.2		21.9	
Group Type, LV % (PNA by Chromatography)				
Paraffins	20.1		17.5	
Naphthenes	55.8		53.2	
Aromatics	24.1		29.3	
ASTM D 86 Distillation, °F				
St/5	166/188		175/198	
10/30	200/240		205/236	
50	260		261	
70/90	297/356		306/366	
95/EP	385/428		389/420	
% Overhead	98.5		98.5	
TBP Distillation, °F (Simulated by Chromatography)				
St/5	50/155		52/160	
10/30	173/214		175/217	
50	246		256	
70/90	302/374		308/377	
95/99	391/464		399/451	
Gum, mg/100 ml	1		1	

TABLE IX

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF 400°F+ SRC-II WITH ICR 106 CATALYST
 PILOT PLANT RUN 76-166; FEED - WOW 3751

Run Hours	145-169		169-193		313-337		337-361	
Avg. Catalyst Temp., °F	746		751		752		752	
LHSV	0.52		0.43		0.99		0.99	
Total Pressure, psig	2,495		2,495		2,501		2,497	
H ₂ Mean Pressure, psia	2,306		2,340		2,309		2,288	
Total Gas In, SCF/Bbl	17,591		22,376		17,055		15,446	
Recycle Gas, SCF/Bbl	13,746		18,315		14,351		12,776	
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
No Loss Prod. Yields								
C ₁	0.20		0.23		0.21		0.21	
C ₂	0.36		0.38		0.34		0.35	
C ₃	0.47		0.47		0.32		0.33	
iC ₄	0.05	0.08	0.04	0.07	0.03	0.05	0.03	0.05
nC ₄	0.16	0.26	0.15	0.26	0.10	0.17	0.10	0.17
C ₅ -180°F	1.97	2.73	2.59	3.53	1.64	2.28	2.34	3.20
180-350°F	27.58	34.68			24.71	30.90		
350°F-EP	69.40	79.40			71.08	78.32		
180-400°F			48.45	59.23			39.58	47.65
400°F-EP			48.17	54.90			55.45	60.24
Total C ₅ +	98.95	116.81	99.21	117.68	97.43	111.50	97.36	111.08
Actual/No Loss Recovery	100.90/105.70		107.38/106.01		101.11/103.95		100.15/103.89	
H ₂ Cons. (Gross), SCF/Bbl	3,844		4,061		2,704		2,670	
H ₂ Cons. (Chemical), SCF/Bbl	3,733		3,935		2,585		2,549	
Liquid Product Nitrogen, ppm	0.43		0.22		8.4		10.5	

TABLE X

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF 400°F+ SRC-II WITH ICR 106 CATALYST
 WHOLE LIQUID PRODUCT PROPERTIES
 PILOT PLANT RUN 76-166
 ~2300 PSIA H₂

Time Onstream, Hr	145-169	169-193	313-337	337-361
Avg. Catalyst Temp., °F	746	751	752	752
LHSV	0.55	0.43	0.99	0.99
Inspections of Whole Liquid Product				
Gravity, °API	35.9	35.8	31.0	30.0
Aniline Point, °F	120.3	120.5	80.8	76.5
Sulfur, ppm	57	22	34	35
Nitrogen, ppm	0.43	0.22	8.4	10.5
Hydrogen, Wt %	14.01	14.25/13.59	11.68	10.38
Oxygen, ppm	50	40	240	250
Group Type, LV % (High Mass)				
Paraffins	48.8	48.4	37.6	37.4
Naphthenes	43.3	45.8	39.8	37.7
Aromatics	7.9	5.7	22.6	24.9
TBP Distillation, °F (Simulated by Chromatography)				
St/5	75/210	75/211	95/213	89/215
10/30	216/337	221/338	241/359	242/360
50	386	387	408	411
70/90	437/514	438/514	463/540	466/545
95/99	562/656	561/647	594/710	601/719

TABLE XI

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF 400°F+ SRC-II WITH ICR 106 CATALYST
 NAPHTHA PRODUCT PROPERTIES
 PILOT PLANT RUN 76-166
 ~2300 PSIA H₂

Time Onstream, Hr	145-169	169-193	313-337	337-361
Avg. Catalyst Temp., °F	746	751	752	752
LHSV	0.55	0.43	0.99	0.99
Light Naphtha (C ₅ -180°F) Product Gravity, °API	66.0	63.3	66.8	63.1
Heavy Naphtha Product Boiling Range, °F	180-350	180-400	180-350	180-400
Gravity, °API	47.3	42.3	46.3	39.7
Aniline Point, °F	109.7	110.0	89.9	84.9
Nitrogen, ppm	(0.9)			2.0
Oxygen, ppm	110			280
Octane No., F-1 Clear	58.8	54.4	64.5	63.8
Octane No., F-2 Clear				
Group Type, LV % (Low Mass)				
Paraffins	1.1	1.0		3.7
Naphthenes	96.5	97.0		79.2
Aromatics	2.4	2.0		17.1
Group Type, LV % (By Chromatography)				
Paraffins	5.1		3.0	
Naphthenes	91.9		84.3	
Aromatics	3.0		12.7	
TBP Distillation, °F (Simulated by Chromatography)				
St/5	102/185	134/214	138/185	153/214
10/30	218/233	221/273	218/242	222/273
50	275	332	279	323
70/90	307/341	372/396	311/345	370/398
95/99	350/364	402/410	352/363	407/416
ASTM D 86 Distillation, °F				
St/5	224/231			233/245
10/30	235/249			254/282
50	264			314
70/90	285/313			342/366
95/EP	325/373			375/414
% Overhead	98			99
% Trap	1			0
% in Flask	1			1
Viscosity, cSt, 100°F			0.7964	

*Incomplete.

TABLE XII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF 400°F+ SRC-II WITH ICR 106 CATALYST
 350°F+ AND 400°F+ PRODUCT PROPERTIES
 PILOT PLANT RUN 76-166
 ~2300 PSIA H₂

Time Onstream, Hr	145-169	169-193	313-337	337-361
Avg. Catalyst Temp., °F	746	751	752	752
LHSV	0.55	0.43	0.99	0.99
Boiling Range	350°F+	400°F+	350°F+	400°F+
Inspections				
Gravity, °API	31.2	30.6	25.2	23.0
Aniline Point, °F	126.8	134.2	78.8	73.0
Nitrogen, ppm				5.2
Oxygen, ppm	190			520
Smoke Point, mm	21	20	11	8
Freeze Point, °F	-69	-42	-54	
Group Type, LV %				
(High Mass)				
Paraffins	3.4	1.0	3.9	5.2
Naphthenes	92.7	97.0	57.4	50.0
Aromatics	3.9	2.0	38.8	44.8
TBP Distillation, °F				
(Simulated by Chromatography)				
St/5	316/360	362/389	326/361	372/400
10/30	368/398	400/437	371/411	410/448
50	431	472	452	480
70/90	478/544	506/568	495/568	517/592
95/99	585/681	602/683	618/722	645/756
Viscosity				
At 100°F, cSt			2.263	
At 210°F, cSt			1.019	

TABLE XIII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF 400°F+ SRC-II WITH ICR 106 CATALYST
 PILOT PLANT RUN 76-167
FEED - WOW 3778 (WATER WASHED WOW 3751)

Run Hours	242-266		506-530		794-818	
Avg. Catalyst Temp., °F	750		752		751	
LHSV	1.00		1.01		0.99	
Total Pressure, psig	2,499		2,500		2,496	
H ₂ Mean Pressure, psia	2,332		2,303		2,293	
Total Gas In, SCF/Bbl	19,297		17,715		17,140	
Recycle Gas, SCF/Bbl	16,368		15,029		14,450	
No Loss Product Yields	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
C ₁	0.11		0.16		0.12	
C ₂	0.16		0.24		0.17	
C ₃	0.14		0.21		0.15	
iC ₄	0.01	0.02	0.02	0.03	0.02	0.03
nC ₄	0.04	0.07	0.06	0.10	0.05	0.08
C ₅ -300°F	15.78	19.97				
300-500°F	62.07	69.80				
500°F-EP	20.07	21.22				
C ₅ -180°F			1.61	2.14	0.94	1.24
180-300°F			15.37	19.39	15.10	19.05
300-550°F			70.36	78.49	71.42	79.42
550°F-EP			10.02	10.65	10.05	10.62
Total C ₅ +	97.91	111.00	97.36	110.66	97.50	110.33
Actual/No Loss Recovery	103.61/104.34		101.93/104.00		103.73/103.96	
H ₂ Cons. (Gross), SCF/Bbl	2,928		2,686		2,690	
H ₂ Cons. (Chemical, SCF/Bbl	2,829		2,608		2,582	
Liquid Product Nitrogen, ppm	4.7		16		21	

TABLE XIV

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF 400°F+ SRC-II WITH ICR 106 CATALYST
 WHOLE LIQUID PRODUCT PROPERTIES
 PILOT PLANT RUN 76-167
 ~2300 PSIA H₂

Time Onstream, Hr	242-266	506-530	794-818
Avg. Catalyst Temp., °F	750	752	751
LHSV	1.00	1.01	0.99
Inspections of Whole Liquid Product			
Gravity, °API	30.3	29.5	27.3
Aniline Point, °F	77.9	68.1	65.2
Sulfur, ppm	3.2		2.4
Nitrogen, ppm	4.7	16	21
Hydrogen, Wt %	12.55	11.66	11.98
Oxygen, ppm	200	380	460
Chloride, ppm	1.4		
Group Type, LV % (High Mass)			
Paraffins	2.1	2.4	2.5
Naphthenes	61.0	56.1	53.2
Aromatics	36.9	41.4	44.2
TBP Distillation, °F (Simulated by Chromatography)			
St/5	84/217	86/215	85/211
10/30	250/364	249/364	242/352
50	416	420	415
70/90	470/546	472/549	473/557
95/99	600/710	603/718	610/721

TABLE XV
DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF HYDROTREATED SRC-II FEEDS
FOR NAPHTHA HYDROTREATING AND FRACTIONS THEREOF

Boiling Range, °F	Whole Liquid Product ¹	St-400	St-450	180-350		350-400	350-450	St-180 ²	
Identification	WOW 3774	SGQ 6502	SGQ 6504	ASL 5660-2	ASL 5645-2	ASL 5660 Btm	ASL 5645 Btm	ASL 5660-1	ASL 5645-1
WOW 3774, Wt %	100	49.8	66.2	32.8	32.8	13.6	30.0	3.4	3.4
WOW 3774, LV %	100	53.2	68.5	35.8	35.8	13.5	28.8	3.9	3.9
Inspections									
Gravity, °API	31.1	41.6	35.4	45.4	45.0	28.3	25.2	56.1	54.3
Aniline Point, °F	42.3	67.6	48.4	80.2	79.9	33.4	<32		
Sulfur, ppm	7.5	3.7	2.9						
Total Nitrogen, ppm	410	255	382	106	121	(688)	(602)	0.61	1.1
Basic Nitrogen, ppm	383	281	390						
Oxygen, ppm	3,100	3,000	3,300	2,300		(6,100)	(5,500)		
Chloride, ppm	2	(9)	8						
Hydrogen, Wt %	11.48/11.19	Incomplete	10.72/10.62			12.03	11.10		
Octane No.									
F-1 Clear		74.4	80.0						
Group Type, LV %	High Mass	Low Mass	Low Mass			High Mass	High Mass		
Paraffins	3.9	8.2	6.6			3.8	4.1		
Naphthenes	39.2	61.0	48.4			35.0	24.6		
Aromatics	57.0	30.8	45.0			61.2	71.4		
Group Type, LV % (PNA by Chromatography)									
Paraffins		10.3		10.8					
Naphthenes		58.1		66.8					
Aromatics		31.6		22.4					
ASTM D 86 Distillation, °F									
St/5	186/226	200/216	222/243	224/234		365/370	382/387		
10/30	248/317	228/255	256/295	237/251		370/373	391/395		
50	385	286	342	266		375	401		
70/90	444/527	321/364	382/410	287/314		382/400	407/421		
95/EP	616/670	382/408	422/440	323/375		410/438	430/470		
Overhead, %	985	99	99	99		98	99		
TBP Distillation, °F (Simulated by Chromatography)									
St/5	60/170	135/184	139/193	163/180	160/199	312/336	324/359		
10/30	205/292	216/260	220/282	194/238	221/245	344/364	369/405		
50	344	309	349	274	282	385	422		
70/90	442/529	359/406	406/441	308/344	314/349	407/431	440/462		
95/99	583/710	414/436	450/469	354/369	358/371	440/(636)	469/485		
Viscosity, cSt at 100°F						1.333	1.487		
Gross Heat of Combustion, Btu/Lb						18,334	18,081		
Pour Point, °F							<-80		
Freeze Point, °F						<-94	<-94		
Smoke Point, mm						10	9		
Density at 70°C						0.8455	0.8641		
Refractive Index						1.4632	1.4752		
Molecular Weight						(170)	(163)		

¹Whole Liquid Product from which these Fractions were Distilled

²Some Loss of Start-180°F during these Distillations

TABLE XVI

DOE CONTRACT EF-76-C-01-2315
 HYDROFINING OF PARTIALLY HYDROTREATED ST-400°F
 SRC-II NAPHTHA WITH ICR 113 CATALYST
 PILOT PLANT RUN 72-326; FEED - SGQ 6502

Run Hours	138-150	222-246		
Average Cat. Temperature, °F	700	700		
LHSV	2.02	2.01		
Total Pressure, psig	648	638		
H ₂ Mean Pressure, psia	523	505		
Total Gas In, SCF/Bbl	3029	2824		
Recycle Gas, SCF/Bbl	2941	2695		
	Wt %	Vol %	Wt %	Vol %
No Loss Prod. Yields				
C ₁	0.01		0.01	
C ₂	0.01		0.01	
C ₃	0.02		0.02	
iC ₄	0.01	0.01	0.01	0.01
nC ₄	0.01	0.01	0.01	0.01
Total C ₅ +	99.84	100.20	99.80	100.20
Actual/No Loss Recovery	97.65/100.25	98.78/100.20		
H ₂ Cons. (Gross), SCF/Bbl	150	129		
H ₂ Cons. (Chemical), SCF/Bbl	134	108		
Whole Liquid Product				Feed
Inspections				Inspections (SGQ 6502)
Gravity, °API	42.2	41.8		41.6
Aniline Point, °F	72.8	70.0		67.6
Sulfur, ppm	6	4		4
Total Nitrogen, ppm	0.22	0.20		255
Oxygen, ppm ¹	70	100		3000 ²
Hydrogen, Wt %	13.14	13.28		
Octane No., F-1 Clear	70.5	71.2		74.4
Octane No., F-2 Clear	65.2	66.7		
Molecular Weight		119		
Group Type, LV % (Low Mass)				
Paraffins	8.9	8.9		8.2
Naphthenes	64.0	61.8		61.0
Aromatics	27.0	30.2		30.8
TBP Distillation, °F (Simulated by Chromatography)				
St/5	96/179	101/182		135/184
10/30	197/242	199/247		216/260
50	289	294		309
70/90	340/394	345/399		359/406
95/99	407/435	411/437		414/436
ASTM D 86 Distillation, °F				
St/5		206/214		200/216
10/30		231/258		228/255
50		290		286
70/90		327/370		321/364
95/EP		384/415		382/408
% Overhead		99		99
% Trap		1		0
% in Flask		1		1

¹No special precautions were taken to remove dissolved water and air, therefore, analyses include oxygen from these sources.

²Incomplete.

TABLE XVII

DOE CONTRACT EF-76-C-01-2315
HYDROFINING OF PARTIALLY HYDROTREATED ST-450°F
SRC-II NAPHTHA WITH ICR 113 CATALYST
PILOT PLANT RUN 72-326; FEED - SGQ 6504

Run Hours	258-270		282-294		306-318		330-342		
Average Cat. Temperature, °F	723		700		700		701		
LHSV	2.02		1.93		2.00		2.02		
Total Pressure, psig	660		803		804		645		
H ₂ Mean Pressure, psia	537		666		664		526		
Total Gas In, SCF/Bbl	2922		3224		3140		2850		
Recycle Gas, SCF/Bbl	2822		2065		3006		2764		
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	
No Loss Prod. Yields									
C ₁	0.02		0.02		0.01		0.02		
C ₂	0.02		0.02		0.01		0.02		
C ₃	0.04		0.04		0.02		0.02		
iC ₄	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	
nC ₄	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	
Total C ₅ +	99.65	100.44	99.74	100.30	99.78	100.30	99.65	100.01	
Actual/No Loss Recovery	95.23/100.14		97.55/100.22		97.96/100.22		97.58/100.11		
H ₂ Cons. (Gross), SCF/Bbl	99		158		133		86		
H ₂ Cons. (Chemical), SCF/Bbl	78		124		122		63		
Whole Liquid Product									Feed
Inspections									<u>Inspections</u>
Gravity, °API	36.7		36.3		36.2		36.0		35.4
Aniline Point, °F	54.6		54.2		54.0		52.6		48.4
Sulfur, ppm	2		2		2				3
Total Nitrogen, ppm	0.11		0.11		0.28		0.70		382
Oxygen, ppm ¹	110		80		80		80		3300
Hydrogen, Wt %	12.51		12.44		12.25		12.99		10.67
Octane No., F-1 Clear	2						76.4		80.0
Octane No., F-2 Clear							69.1		
Group Type, LV % (Low Mass)									
Paraffins	7.0		6.6		6.6		6.7		6.6
Naphthenes	47.7		49.4		49.4		48.7		48.4
Aromatics	45.4		43.3		43.9		44.5		45.0
TBP Distillation, °F									
(Simulated by Chromatography)									
St/5	68/186		132/191		128/193				139/193
10/30	219/276		217/277		218/278				220/282
50	335		339		340				349
70/90	397/440		399/437		400/437				406/441
95/99	453/481		447/469		447/472				450/469
ASTM D 86 Distillation, °F									
St/5					228/241		226/242		222/243
10/30					253/294		255/289		256/295
50					338		337		342
70/90					380/410		382/412		382/410
95/EP					421/446		424/446		422/440
% Overhead					98		98.5		99
% Trap					1		0.5		0
% in Flask					1		1		1

¹No special precautions were taken to remove dissolved water and air, therefore, analyses include oxygen from these sources.

²Incomplete.

TABLE XVIII

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF PARTIALLY HYDROTREATED 300-550°F
SRC-II FEEDS FOR JET HYDROTREATING

Identification	SGQ 6547	SGQ 6581	SGQ 6588 ¹
Inspections			
Gravity, °API	27.5	26.5	24.6
Aniline Point, °F	48.4	33.3	<22
Sulfur, ppm		2.9	7.4
Total Nitrogen, ppm	12	173	838
Basic Nitrogen, ppm		185	
Oxygen, ppm	600	2400	7400
Hydrogen, Wt %	²	11.41	²
Smoke Point, mm	11	9	8
Freeze Point, °F	-71		
Group Type, LV % (High Mass)			
Paraffins	4.2	4.3	4.7
Naphthenes	39.9	32.4	26.5
Aromatics	55.9	63.3	68.8
TBP Distillation, °F (Simulated by Chromatography)			
St/5	222/305	242/309	239/312
10/30	328/386	332/393	335/399
50	423	429	429
70/90	462/509	467/516	464/509
95/99	525/549	533/554	525/549
ASTM D 86 Distillation, °F			
St/5	360/365	340/358	
10/30	368/380	368/395	
50	402	420	
70/90	434/474	444/483	
95/EP	482/520	498/530	
% Overhead	98.5	98.5	
Viscosity			
At -40°F, cSt	12.59		16.12
At 130°F, cSt	1.219	1.229	1.250

¹No runs were made with SGQ 6588.

²Incomplete.

TABLE XIX

DOE CONTRACT EF-76-C-01-2315
HYDROFINISHING OF 300-550°F HYDROTREATED
SRC II WITH ICR 113

FEED SGQ 6547 (12 ppm N, 56% Aromatics)
Pilot Plant Run 72-326

Run Hours	402-414	426-438	450-462	462-486	
Avg. Cat. Temperature, °F	701	700	750	751	
LHSV	2.03	1.02	1.00	1.01	
Total Pressure, psig	1350	1346	1703	1700	
H ₂ Mean Pressure, psia	1215	1209	1521	1518	
Total Gas In, SCF/Bbl	5685	6041	6704	6742	
Recycle Gas, SCF/Bbl	5341	5444	5580	5542	
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %	
C ₁	0.01		0.02		
C ₂	0.03		0.05		
C ₃	0.22		0.30		
iC ₄	0.07	0.10	0.13	0.21	
nC ₄	0.06	0.09	0.09	0.14	
Total C ₅ ⁺	100.08	102.93	100.30	104.05	
Act./No Loss Recovery	97.02/100.51	99.00/100.93	100.77/101.80	100.00/101.91	
H ₂ Cons. (Gross), SCF/Bbl	343	596	1123	1200	
H ₂ Cons. (Chemical), SCF/Bbl	399	543	1056	1119	
Whole Liquid Product Inspections					Feed (SGQ 6547)
Gravity, °API	32.0	33.4	38.7	40.3	27.5
Aniline Point, °F	69.2	78.0	93.2	93.5	48.4
Sulfur, ppm	1.8				
Nitrogen, ppm	0.28	0.22	0.19	0.21	12
Oxygen, ppm	110	90	90	170	600
Hydrogen, %	12.79	12.54	13.04	13.06	
Smoke Point, mm	13	15	17	17	11
Pour Point, °F	-79	-76	-81	-83	-71
High Mass, LV %					
Paraffins	4.9	(10.5)	5.6	6.6	4.2
Naphthenes	51.3	53.1	64.6	63.7	39.9
Aromatics	43.8	36.4	29.8	29.7	55.9
TBP Distillation, °F (Simulated by Chrom.)					
St/5	33/235	85/232	38/140	37/99	222/305
10/30	295/364	286/360	174/296	168/286	328/386
50	405	401	355	348	423
70/90	442/493	437/489	408/474	402/467	462/509
95/99	515/551	510/552	499/552	494/547	525/549
ASTM D 86 Distillation, °F					
St/5	212/278				360/365
10/30	312/364				368/380
50	392				402
70/90	420/464				434/474
95/EP	483/510				482/520
% Overhead	99				98.5
Existed Gum, mg/100 ml				1	
Fraction Below 300°F, Wt % (TBP)	10.2	11.6	28.5	31.2	4.1

TABLE XX

DOE CONTRACT EF-76-C-01-2315
HYDROFINISHING OF 300-550°F HYDROTREATED
SRC II WITH ICR 113

Feed SGQ 6581 (173 ppm N, 63% Aromatics)
Pilot Plant Run 72-326

Run Hours	498-510		522-534		546-558		570-582		594-606			
Avg. Cat. Temperature, °F	750		701		725		750		775			
LHSV	1.00		0.99		1.03		1.07		0.98			
Total Pressure, psig	1713		2096		2099		2050		2099			
H ₂ Mean Pressure, psig	1535		1912		1887		1834		1874			
Total Gas In, SCF/Bbl	6533		6719		6187		5996		6478			
Recycle Gas, SCF/Bbl	5768		6120		5356		5145		5450			
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %		
C ₁	0.07		0.03		0.02		0.04		0.08			
C ₂	0.14		0.04		0.04		0.10		0.20			
C ₃	0.43		0.16		0.21		0.31		0.52			
iC ₄	0.13	0.21	0.02	0.03	0.05	0.08	0.08	0.13	0.24	0.38		
nC ₄	0.13	0.19	0.05	0.07	0.08	0.12	0.10	0.15	0.19	0.29		
Total C ₅ ⁺	100.28	105.01	100.58	102.76	100.82	103.31	100.66	103.82	100.38	105.29		
Act./No Loss Recovery	96.74/101.16		95.04/100.88		80.93/101.22		100.73/101.30		99.91/101.60		Feed (SGQ 6581)	
H ₂ Cons. (Gross), SCF/Bbl	765		598		830		850		1028			
H ₂ Cons. (Chemical), SCF/Bbl	691		520		716		764		945			
Whole Liquid Product Inspections												
Gravity, °API	33.9		29.9		30.4		31.4		34.1			24.6
Aniline Point, °F	74.3		62.4		66.8		69.3		79.7			33.3
Nitrogen, ppm	0.26		0.20		0.18		0.35					173
Oxygen, ppm	190											2400
Hydrogen, Wt %	Incomplete											11.41
Smoke Point, °F	12				12		12		13			9
Freeze Point, °F	-76		-66		-67		-76		-81			
Group Type, LV % (High Mass)												
Paraffins	4.9		4.3		4.3		4.4		5.0			4.3
Naphthenes	53.1		47.8		49.7		51.7		56.5			32.4
Aromatics	42.0		47.8		46.0		43.8		38.5			63.3
TBP Distillation, °F (Simulated by Chrom.)												
St/5	42/169		45/285		46/281		30/258		19/208			242/309
10/30	221/323		318/373		316/371		306/367		277/353			332/393
50	378		409		408		406		392			429
70/90	432/495		448/500		446/499		444/496		428/485			467/516
95/99	518/567		520/554		520/554		518/555		509/530		533/554	
Fraction Below 300°F, Wt % (TBP)	20.6		6.4		6.8		8.3		12.6		3.5	

TABLE XXI

DOE CONTRACT EF-76-C-01-2315
HYDROFINISHING OF 300-550°F HYDROTREATED
SRC II WITH ICR 106

Feed SGQ 6581 (173 ppm N, 63% Aromatics)
Pilot Plant Run 76-168

Run Hours	92-104		104-128		140-152		164-176		188-200		212-224			
Avg. Cat. Temperature, °F	724		727		727		704		704		728			
LHSV	1.02		1.00		2.00		1.99		1.91		2.01			
Total Pressure, psig	1699		1697		1696		1694		1379		1399			
H ₂ Mean Pressure, psig	1528		1528		1527		1535		1250		1251			
Total Gas In, SCF/Bbl	7153		7426		7046		6786		6869		6445			
Recycle Gas, SCF/Bbl	5371		5586		5609		5539		5833		5483			
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %		
C ₁	0.03		0.03		0.02		0.01		0.01		0.02			
C ₂	0.08		0.07		0.05		0.03		0.02		0.04			
C ₃	0.33		0.30		0.20		0.14		0.12		0.20			
iC ₄	0.19	0.30	0.18	0.28	0.06	0.09	0.02	0.03	0.02	0.03	0.05	0.07		
nC ₄	0.11	0.16	0.10	0.15	0.06	0.09	0.04	0.06	0.03	0.05	0.06	0.08		
Total C ₅ ⁺	102.19	110.58	102.35	110.69	101.97	108.44	101.80	106.71	101.50	105.35	101.19	105.43		
Act./No Loss Recovery	98.02/102.91		98.91/103.03		97.35/102.35		99.57/102.03		98.56/101.70		98.35/101.56			Feed (SGQ 6581)
H ₂ Cons. (Gross), SCF/Bbl	1782		1839		1436		1246		1035		961			
H ₂ Cons. (Chemical), SCF/Bbl	1720		1785		1386		1197		1002		920			
Whole Liquid Product Inspections														
Gravity, °API	39.4		39.3		36.5		34.1		32.5		33.1		24.6	
Aniline Point, °F	Incomplete		122.2		110.9		102.0		86.2		84.4		33.3	
Nitrogen, ppm	0.14		0.44		0.15		0.12		0.16		0.15		173	
Smoke Point, mm	24		23		22		17		14		14		9	
Freeze Point, °F	-81		-90		-84		-77		-74		-72			
High Mass, LV %														
Paraffins	5.2		5.2		4.4		4.1		4.2		4.5		4.3	
Naphthenes	89.1		88.9		82.0		75.8		63.7		61.4		32.4	
Aromatics	5.7		5.9		13.6		20.1		32.1		34.2		63.3	
TBP Distillation, °F (Simulated by Chrom.)														
St/5	50/192		30/200		31/228		45/270		42/272		28/233		242/309	
10/30	248/340		261/344		291/358		309/367		310/368		293/361		332/393	
50	376		376		388		396		402		395		429	
70/90	406/462		407/465		419/476		432/488		439/491		431/486		467/516	
95/99	486/530		490/536		500/546		508/552		512/556		509/556		533/554	
Fraction Below 300°F, Wt % (TBP)	16.5		14.3		10.8		7.9		7.7		10.4		3.5	

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TABLE XXII

DOE CONTRACT EF-76-C-01-2315
 HYDROFINISHING OF 300-550°F HYDROTREATED SRC-II WITH ICR 106
 FEED - SGQ 6547 (12 PPM N, 56% AROMATICS); PILOT PLANT RUN 76-168

Run Hours	258-268		272-296		308-320		
Avg. Catalyst Temperature, °F	728		728		726		
LHSV	2.01		2.00		0.99		
Total Pressure, psig	1400		1401		1403		
H ₂ Mean Pressure, psia	1254		1258		1234		
Total Gas In, SCF/Bbl	6960		7025		7448		
Recycle Gas, SCF/Bbl	5736		5842		5734		
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	
C ₁	0.02		0.02		0.03		
C ₂	0.07		0.07		0.13		
C ₃	0.45		0.45		0.97		
iC ₄	0.44	0.70	0.45	0.70	1.57	2.47	
nC ₄	0.18	0.27	0.19	0.28	0.56	0.86	
Total C ₅ +	100.90	109.28	100.81	108.75	99.62	111.85	
Actual/No Loss Recovery	95.74/102.02		96.82/101.95		102.88/102.85		
H ₂ Cons. (Gross), SCF/Bbl	1224		1182		1714		
H ₂ Cons. (Chemical), SCF/Bbl	1183		1141		1671		
Whole Liquid Product Inspections							Feed (SGQ 6547)
Gravity, °API	40.6		39.9		46.6		27.5
Aniline Point, °F	102.6		98.3		112.0		48.4
Nitrogen, ppm	0.12		0.08		0.67		12
Smoke Point, mm	Incomplete		15		24		11
Freeze Point, °F	Incomplete		-81		-94		-71
High Mass, LV %							
Paraffins	5.7		6.0		8.3		4.2
Naphthenes	71.4		67.5		75.3		39.9
Aromatics	22.8		26.6		16.4		55.9
TBP Distillation, °F (Simulated by Chromatography)							
St/5	18/164		25/163		26/97		222/305
10/30	201/322		198/321		165/258		328/386
50	365		366		327		423
70/90	399/453		402/454		368/418		462/509
95/99	479/524		480/523		445/494		525/549
Fractions Below 300°F (TBP), Wt %	22.1		22.3		37.6		4.1

TABLE XXIII

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF HYDROTREATED SRC-II FEEDS
FOR CATALYTIC CRACKING TESTS

Identification	WLP* WOW 3774	SGQ 6536	SGQ 6503	SGQ 6505
Boiling Range, °F		350+	400+	450+
Wt % of WOW 3774	100	63.8	50.2	33.8
LV % of WOW 3774	100	60.3	46.8	31.5
Inspections				
Gravity, °API	31.1	20.5	19.1	17.0
Aniline Point, °F	42.3	<32	<32	<32
Sulfur, ppm	7.5		8.9	14
Total Nitrogen, ppm	410	530/505/ ¹	480	520
Basic Nitrogen, ppm	383	452	389	289/307
Oxygen, ppm	3100	3600	4100	3700
Chloride, ppm	2		1.3	0.8
Hydrogen, Wt %	11.48/11.19	10.89	**	10.32
Pour Point, °F		-45	-50	-25
Group Type, LV % (High Mass)				
Paraffins	3.9	5.1	5.9	5.8
Naphthenes	39.2	28.1	25.2	30.7
Aromatics	57.0	66.8	68.9	63.6
ASTM D 86 Distillation, °F				
St/5	186/226		428/436	452/461
10/30	248/317		440/453	465/477
50	389		470	504
70/90	444/527		502/592	547/636
95/EP	616/670		662/712	698/728
% Overhead	98.5		98	98.5
TBP Distillation, °F (Simulated by Chromatography)				
St/5	60/170	323/362	348/397	401/437
10/30	205/292	382/420	405/446	445/491
50	394	456	487	539
70/90	442/529	499/583	533/622	599/691
95/99	583/710	643/763	675/796	743/852
Density at 70°C, g/ml		0.8895	0.9005	0.9142
Refractive Index, 80°C		1.4913	1.4995	1.5032
Molecular Weight		162	176	191

*Whole liquid product (WLP) from which the three catalytic cracking feeds were distilled.

**Incomplete.

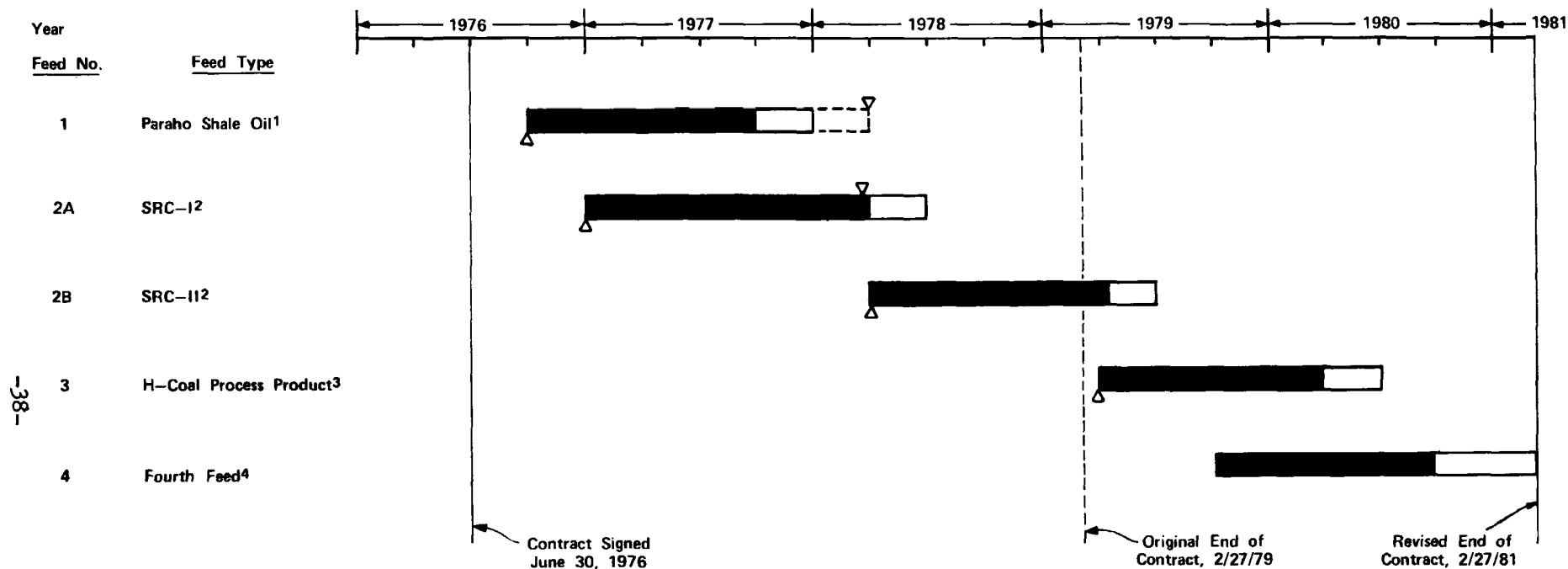
¹495

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FIGURE 1

DOE CONTRACT EF-76-C-01-2315
FEED TIMING SCHEDULE (REVISED, APRIL 1979)



¹With 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.

²Work on SRC-I was suspended and SRC-II was added as Feed 2B.

³H Coal Process Product, sufficient for the full program on this feed, was received on 3/23/79.

⁴The fourth feed to be selected by mutual agreement between DOE and Chevron.

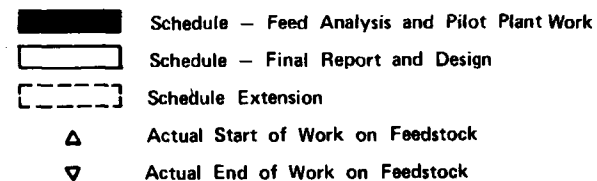
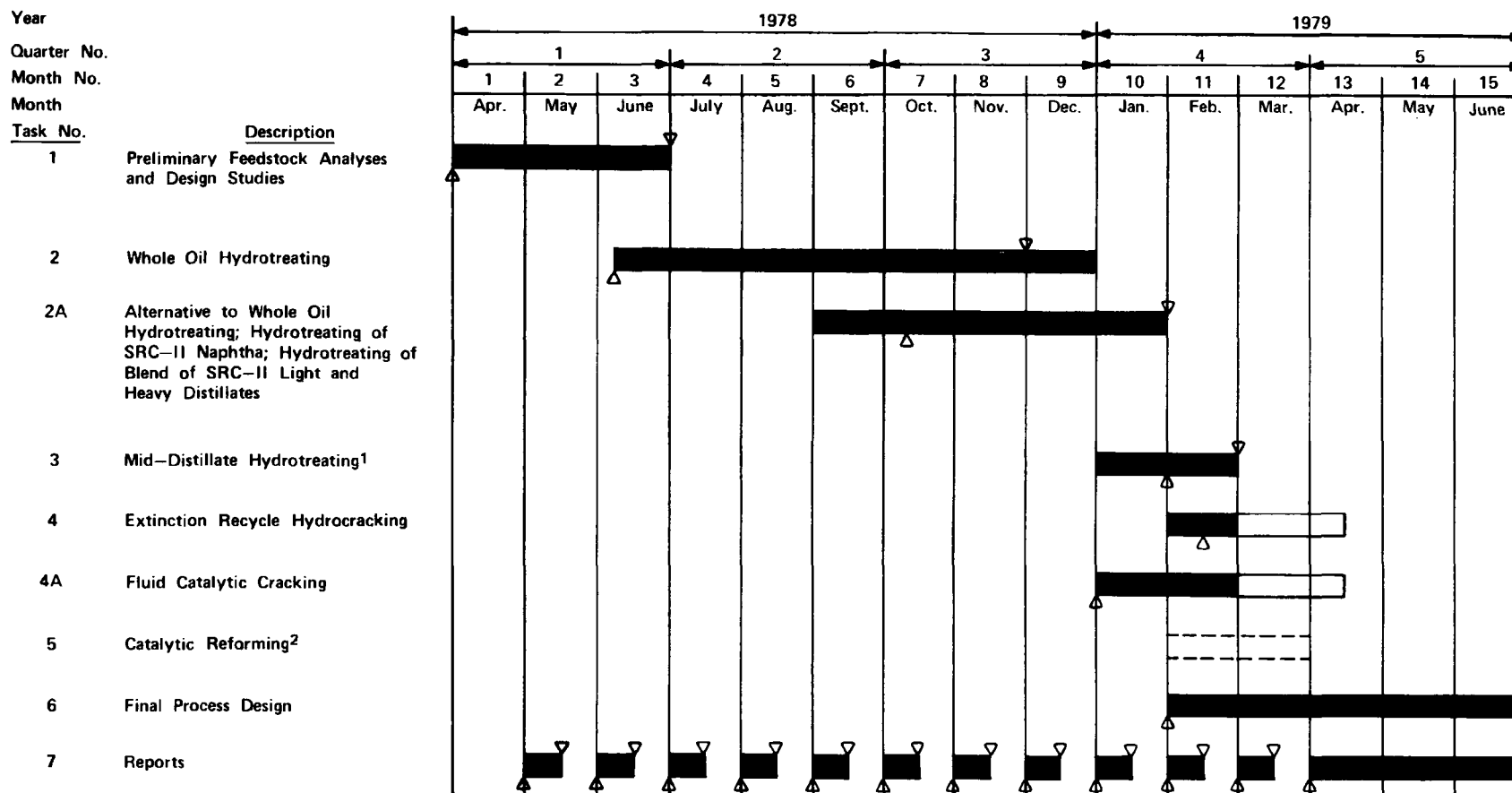


FIGURE 2

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



¹Task also includes naphtha hydrotreating.

²Task 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. (No Task 5 work is anticipated for SRC II.)

