

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

Quarterly Report for the
Period October-December 1978

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I. Abstract

Pilot plant tests on the hydrotreating of SRC-II process product indicate that this coal-derived feed is a suitable feed 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.

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 is devoted to the processing of SRC-II product. Assuming that the contract is extended as shown in Figure 1, 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 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, if the contract is extended.

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

Work on Feedstock 3, H-Coal process product, is scheduled to begin next quarter, as soon as the contract is extended.

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 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)

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.

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 were for hydrotreating of the whole SRC-II process product blend to remove nitrogen, sulfur, oxygen, and metals using fixed catalyst beds.

The pilot plant tests are designed: (1) to evaluate two Chevron catalysts, ICR 106 (containing nickel, tungsten, silica, and alumina) and ICR 113 (containing nickel, molybdenum, silica, and alumina), (2) to select appropriate processing conditions, (3) to determine yields and hydrogen consumptions, (4) to determine product properties, and (5) to determine catalyst deactivation rates.

Each pilot plant test is run downflow with a fixed bed of 130 cc of catalyst. The catalysts are sulfided in situ according to a procedure simulating commercial sulfiding. Then feed is introduced at 500°F, and

temperatures are gradually increased over a period of eight hours to 750°F. The recycle gas is scrubbed with water to remove gaseous ammonia and hydrogen sulfide. The liquid product is contacted with water to remove dissolved ammonia and hydrogen sulfide.

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. Each catalyst lost a maximum of about 25°F activity during the fouling rate portion of the test and showed no significant activity change in the portions of the run at higher pressure.

Tables II and III summarize the conditions and results of the tests. More details on the earlier parts of the runs, including inspections and yield periods for the first 2000 hours of the run, were presented previously (FE-2315-31).

The remaining yields and product inspections for Run 76-165 with ICR 106 catalyst are summarized in Tables IV-XI, inclusive. In particular, note the breakdown by carbon number of the 180-300°F naphtha shown in Table VIII. The very high yield of cyclics shows that this material would produce a high octane gasoline upon reforming. Furthermore, the exceptionally high yields of C₆-C₉ aromatics upon reforming would make this naphtha an unusually attractive feed for petrochemicals production if a use other than as a transportation fuel is desired.

One product inspection of particular interest is the JFTOT thermal stability rating of the jet boiling range product (ASTM D 1660). Table XI shows that the 300°F+ product from the ICR 106 tests easily passes this test at 280°C with a No. 1 rating. Current jet specification requires a No. 1 rating at 260°C, a temperature considerably less severe than the 280°C used here. The smoke point of 18 mm is at the minimum range of the current commercial specification; therefore, smoke point appears to be the limiting specification. Any jet product of lower smoke point would require additional low severity hydrotreating. As a result, jet fuel produced from SRC-II should pass the thermal stability specification at any condition that the smoke point is acceptable.

The following table summarizes some of the current commercial (Jet A) specifications compared to the properties of jet fuel from the severely hydrotreated SRC-II. The properties of the jet fuel from SRC-II have been adjusted to an appropriate boiling range by process design estimates and are subject to revision as more data become available.

Comparison of Jet Fuel
from SRC-II With
Current Jet A Commercial Specification

Property	Jet A Specification (ASTM D 1655-78)	Preliminary Estimate: Properties of Jet Fraction from Severely Hydrotreated SRC-II Oil
Gravity, °API	37-51	~35
Hydrogen, Wt %	None as Yet	13.8
Net Heating Value Btu/Lb	18,400 Minimum	18,450
Btu/Gal.	-	130,500
Thermal Stability, JFTOT Break Point, °C	260 Minimum	>280
Smoke Point	18 Minimum	18-20
Aromatics, LV %	25 Maximum	5-15
Freezing Point, °F	-40	<-60
Existent Gum, mg/100 ml, Max.	7	2
Corrosion, Copper Strip, 2 Hours at 212°F	No. 1	No. 1

One property outside current limits is the API gravity. This specification was originally set because of limitations of available fuel flow controllers. Today's controllers are no longer limited in this way, but there has been no incentive to lower the specification because until now there was no available product below 37°API which met the other specifications.

The gravimetric heat of combustion is only slightly above the current specification. This quantity determines the range of newly designed airplanes. The volumetric heat of combustion, which determines the range of an existing plane with fixed fuel tanks, is quite high because the fuel is unusually dense.

The specifications on smoke point and aromatics content should be qualified as follows. If a Jet A product has a smoke point of less than 20 mm but not less than 18 mm and a maximum of 3 LV % of naphthalenes, it is salable provided the supplier notifies the purchaser of these properties under conditions mutually agreeable to both parties. Otherwise, the smoke point specification is a minimum of 20 mm. Similarly, an aromatics content of over 20 LV % and less than 25 LV % is permitted by mutual agreement between supplier and purchaser. These qualifications are subject to reapproval in 1979.

Additional yields and product inspections for Pilot Plant Run 65-193 using ICR 113 catalyst are given in Tables XII-XXIII, inclusive. Some inspections are incomplete and will be reported at a later date. (Table XII showing yield information for ICR 113 was shown in a previous report but is repeated here because product inspections were not available at that time.)

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

The SRC-II whole process product blend, WOW 3666, was distilled into two fractions: (1) start-400°F naphtha and (2) 400°F+ gas oil. Hydrotreating pilot plant runs were made using each fraction as feed to determine whether it is preferable to process the whole SRC-II process product or to process the naphtha and gas oil fractions separately. Feed inspections are shown in Table I. Selection of the 400°F cut point between fractions is arbitrary and would be most applicable for cases for maximum gasoline production. However, the general conclusions obtained from these experiments should also apply to other cut points.

Initial pilot plant conditions were chosen after consultation with the Chevron Process Engineering Department and Catalytic Reforming Process Development Group.

A. Hydrotreating of
400°F+ SRC-II

Run 76-166

In Pilot Plant Run 76-166, the 400°F+ gas oil was processed initially with ICR 106 catalyst at the same conditions used for our first hydrotreating test with the whole SRC-II:

Total Pressure, psig	2,500
Average H ₂ Partial Pressure, psia	2,300
Liquid Hourly Space Velocity (LHSV), Vol Feed/Vol Catalyst/Hour	0.5
Catalyst Temperature, °F	750
Recycle Gas Rate, SCF/Bbl	15,000

At these conditions, as with the whole SRC-II, the product nitrogen is less than 0.5 ppm.

After 250 hours, the space velocity was increased to 1.0. The nitrogen content of the whole liquid product increased to about 10 ppm and appeared to be lining out in the 10-20 ppm nitrogen range. This nitrogen level is roughly the same as that observed for processing the whole SRC-II at 1.5 LHSV. The 400°F- naphtha is about a third of the

whole SRC-II. Therefore, if the naphtha is removed, it is necessary to reduce the space velocity by about a third to maintain the same product nitrogen level. This shows that the naphtha is easy to denitrify compared to the 400°F+ fraction. In other words, if only the reactor size is considered, there is little or no penalty in catalyst activity as a result of processing the naphtha together with the 400°F+ portion of the SRC-II. (There are other considerations such as the sizes of compressors and feed pumps, as well as concerns about operating flexibility which must be considered in determining which processing scheme is preferable.)

At about 380 hours onstream, plugging problems developed in the exit line from the reactor due to the formation of ammonium chloride. The run was continued for another 240 hours with intermittent plugging. The product is water washed to remove ammonium chloride; however, the plug occurred in a segment of tubing upstream from the point water is injected.

The yield data and product inspections collected during this run are being tabulated and will be presented in a future report.

Water Washing of 400°F+ SRC-II

It was previously reported that our initial attempts to remove chloride from the whole SRC-II by water washing at room temperature were not successful. Because of continued plugging problems, additional water washing experiments were made with the 400°F+ SRC-II. In the latter case, because the light ends were no longer present, it was more convenient to work at higher temperatures without the concern of loss of light ends.

Experiments at both 160-170°F and 190-200°F indicate that the chloride can be removed to about 1 ppm by water washing.

Meanwhile, we have discovered an analytical error in the earlier chloride analysis of the water-washed SRC-II. It may be that we will be able to remove chloride from whole SRC-II by water washing at a lower temperature; this result is being checked.

Run 76-167

Run 76-167 is a repeat of Run 76-166 using the water-washed 400°F+ SRC-II from which the chloride has been removed to about 1.0 ppm. Conditions are the same as the previous run except that the space velocity has been maintained at 1.0 throughout. (In Run 76-166, the LHSV was 0.5 for the first 250 hours.)

This run has now been onstream for about 1000 hours with no plugging problem.

Initial product nitrogen was 1-2 ppm. During the first 1400 hours onstream, the product nitrogen increased to about 15 ppm and has remained at that concentration for the subsequent 600 hours. Therefore, this run verifies the activity of ICR 106 catalyst observed in Run 76-166 prior to the plugging problems and shows that the ICR 106 catalyst is stable at these conditions.

B. Hydrotreating of
SRC-II Naphtha

Run 65-194

The start-400°F naphtha was processed with ICR 113 catalyst at the following conditions in Run 65-194:

Total Pressure, psig	800
Average H ₂ Partial Pressure, psia	650
Liquid Hourly Space Velocity (LHSV), Vol Feed/Vol Catalyst/Hour	1.0
Catalyst Temperature, °F	700
Recycle Gas Rate, SCF/Bbl	3500

At these conditions, product nitrogen is less than 0.5 ppm. After about 300 hours, some plugging problems developed. Originally, it was thought that this plugging was also due to ammonium chloride formation at the reactor exit. However, in this case, in contrast to the result with the 400°F+ SRC-II, there appeared to be some plugging in the preheat section due to coke formation.

Yields and product inspections for this run will be presented in a future report.

Run 65-195

The SRC-II naphtha feed was water washed to remove the chloride. In bench-scale experiments, it was shown that the chloride concentration of the naphtha can be reduced from 50 ppm to 2 ppm by water washing five times at room temperature with equal volumes of naphtha and distilled water. Only a minor improvement was observed by washing at higher temperatures.

Run 65-195 was a repeat of Run 65-194 except that the water-washed naphtha was used as feed. The preheat section, in this experiment, consisted of 12 mesh nonporous alumina. Again, plugging was noted in the preheat section of the reactor; in this case, plugging occurred during the first 100 hours onstream.

These results show that the plugging for the naphtha hydrotreating experiments is not necessarily the result of ammonium chloride but can also be caused by the formation of a carbonaceous deposit in the preheat section of the reactor. A similar problem was noted by

Tan and deRosset* in the processing of a naphtha derived from H-Coal. These workers attributed the carbonaceous deposit to the presence of gum in the naphtha.

Our results for the processing of whole SRC-II and 400°F+ SRC-II gave no indication of a similar preheat plugging problem. Because results to date show no particular advantage of initially hydropyrolysis the naphtha separately from the 400°F+ SRC-II, we do not intend to pursue this plugging problem further.

The reason for the difference between the results for SRC-II naphtha and the higher boiling feedstocks may be related to the considerably higher pressure used in the runs with the heavier stocks. Also, because of its low boiling point, the naphtha is expected to be entirely vaporized in the preheat zone. Therefore, any traces of residue may deposit in a small section of the preheat rather than be carried further into the reaction zone. In contrast, if the feed is heavier and less volatile, the remaining liquid phase can carry these coke precursors into the reaction zone for hydrotreatment.

Assuming the latter explanation is correct, several possible solutions can be suggested to solve the plugging problem:

1. A lower end point naphtha could be selected as feed. Such a naphtha would be less likely to contain coke precursors.
2. The fractionation should be sharper when the naphtha is prepared. Although the naphtha in these experiments had a nominal end point of 400°F, the TBP distillation shows 7-8% boiling above 400°F.
3. Coal-derived naphthas are known to be unstable. Although we attempted to nitrogen blanket the stocks and protect them from light, some exposure either to air or light may have occurred. These stocks may require even more careful handling.
4. The feedstocks should be freshly prepared to minimize any instability problems. The feed used in this study was prepared over a year ago.
5. If diolefins are present in petroleum naphtha feedstocks, preheat plugging of this type is a common experience. This problem is solved commercially by passing the feed through a prereactor containing a hydrogenation catalyst at a temperature sufficiently high to saturate the diolefins but below the temperature at which plugging occurs. Assuming the problem with coal-derived naphthas is similar, the same solution could be applied.

*Gim Tan and Armand J. deRosset, "Upgrading of Coal Liquids," DOE Interim Report FE-2566-12, March 1978.

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.

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 four 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 (FCC) for gasoline production.
4. 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.

Another case could include hydrocracking; however, once the nitrogen is removed, there is very little material remaining above jet cut point. Therefore, there is little incentive for a hydrocracking case; and we do not plan to include one.

We are completing experiments in which the 400°F+ SRC-II is hydro-treated separately from the naphtha. Based on our results to date, we see little incentive for this alternate processing route. Furthermore,

at the lower operating pressures for the naphtha hydrotreater applied in this scheme, a carbonaceous deposit in the preheat section of the reactor caused plugging problems.

We have distilled hydrotreated SRC-II to prepare feeds for FCC tests to be completed in the next few weeks.

During the next quarter, we plan to complete the pilot plant studies on SRC-II and start process engineering and cost studies for the processing of SRC-II.

We also expect to start pilot plant studies on the processing of H-Coal process product during the next quarter, as soon as the contract is extended.

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

Description	Whole SRC-II WOW 3666	SRC-II Naphtha WOW 3750	400°F+ SRC-II WOW 3751
<u>Chevron Identification</u>			
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	Indeterminate	0.82
Oxygen, Wt %	3.79	3.51	3.63
Carbon, Wt %	84.68	84.22	86.61/86.18
Hydrogen, Wt %	10.54	12.23	8.77/9.54
Hydrogen/Carbon Atom Ratio	1.48	1.73	1.21/1.32
Chloride, ppm*	190/102/63*	51*	280*
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 (Simulated by Chromatography)</u>			
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

*Due to an analytical problem (neutron activation analysis), these chloride analyses show much scatter and are now believed to be high. More reliable numbers will be reported when the problem has been resolved.

TABLE II

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 RUN CONDITIONS AND PRODUCT NITROGEN
 PILOT PLANT RUN 65-193
 AVERAGE CATALYST TEMPERATURE 750°F

Time Onstream, Hr	LHSV	Avg. Cat. Temp., °F	Pressure		Nitrogen Content of Whole Liquid Product, ppm	Comments
			Total, psig	H ₂ , psig (Approx.)		
0-490	1.0	750	2500	2300	~10	Lines Out at 10 ppm Product Nitrogen After Initial Transient Higher Activity
490-1660	1.0	750	2000	1700-1800 ²	60-700	Fouling Rate Test; See FE 2315-31
1660-1860	1.0	750	2500	2300	130	No Fouling Observed
1860-2115	1.0	775	2500	2300	10	No Fouling Observed
2115-2650	0.5	775	2500	2300	0.5	No Fouling Observed

¹Recycle gas rate at 2,500 psig, 15,000 SCF/bbl; at 2,000 psig, 8,000 SCF/bbl.

²Intermittent periods of partial plugging in reactor exit lines due to formation of ammonium chloride resulted in lower recycle gas rates and lower hydrogen pressures.

TABLE III

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 RUN CONDITIONS AND PRODUCT NITROGEN
 PILOT PLANT RUN 76-165

Time Onstream, Hr	LHSV	Avg. Cat. Temp., °F	Pressure		Nitrogen Content of Whole Liquid Product, ppm	Comments
			Total, psig	H ₂ , psia ¹ (Approx.)		
0-350	0.5	750	2500	2300	<0.5	No Fouling Observed
350-425	1.0	750	2500	2300	<0.5	No Fouling Observed
425-620	1.5	750	2500	2300	20	No Fouling Observed
620-2100	1.5	750	2000	1700-1800 ²	100-1500	Fouling Rate Test; See FE 2315-31
2100-2300	1.5	750	2500	2300 ²	450	No Fouling Observed
2300-2500	1.5	775	2500	2300 ²	20	Erratic Operation
2500-3030	0.5	775	2500	2300	<0.1	No Fouling Observed

¹Recycle gas rate at 2,500 psig, 15,000 SCF/bbl; at 2,000 psig, 8,000 SCF/bbl.

²Intermittent periods of partial plugging in reactor exit due to formation of ammonium chloride resulted in lower recycle gas rates and lower hydrogen pressures.

TABLE IV

DOE CONTRACT EF-76-C-01-2315
 YIELDS FROM HYDROTREATING SRC-II WITH
 ICR 106 CATALYST AT ~2300 PSIA H₂
 PILOT PLANT RUN 76-165
 FEED - WOW 3666

Time Onstream, Hr	2123 - 2147	2219 - 2243	2315 - 2339	2661 - 2685	2805 - 2829
Avg. Cat. Temp., °F	750	749	774	776	775
LHSV	1.51	1.47	1.50	0.51	0.49
Total Pressure, psig	2493	2506	2499	2501	2499
H ₂ Mean Pressure, psia	2314	2327	2317	2292	2303
Total Gas In, SCF/Bbl	16,050	16,453	16,922	17,596	18,290
Recycle Gas, SCF/Bbl	14,155	14,625	14,851	14,475	15,180
	Wt %	Vol %	Wt %	Vol %	Wt %
<u>No Loss Prod. Yield</u>					
C ₁	0.06		0.09		0.19
C ₂	0.16		0.18		0.30
C ₃	0.21		0.21		0.42
iC ₄	0.02	0.03	0.02	0.03	0.04
nC ₄	0.13	0.20	0.12	0.19	0.26
C ₅ -180°F	5.01	6.48	5.86	7.49	4.78
180-350°F	32.75	38.55			
350°F-EP	58.97	60.15			
180-300°F					27.30
300-500°F					55.15
500°F-EP					10.80
180-400°F			43.11	50.04	68.20
400°F-EP			47.62	47.87	35.41
Total C ₅ +	96.73	105.16	96.60	105.40	98.13
Actual/No Loss Recovery	99.41/102.87		99.80/102.77	99.52/103.16	104.33/104.81
H ₂ Cons. (Gross), SCF/Bbl	1894		1828	2071	3120
H ₂ Cons. (Chemical), SCF/Bbl	1781		1721	1958	2981
Liquid Product Nitrogen, ppm	468		388	32	<0.1
					<0.1

TABLE V

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 WHOLE LIQUID PRODUCT PROPERTIES
 PILOT PLANT RUN 76-165
 FEED - WOW 3666
 ~2300 PSIA H₂

Avg. Catalyst Temperature	750	749	774	776	775
LHSV	1.5	1.5	1.5	0.5	0.5
Time Onstream, Hr	2123 - 2147	2219 - 2243	2315 - 2339	2661 - 2685	2805 - 2829
<u>Whole Liquid Product Inspections</u>					
Gravity, °API	31.5	31.9	33.2	38.0	38.1
Aniline Point, °F	44.7	47.2	55.2	103.9	105.8
Sulfur, ppm	14	8	21	3	4
Total Nitrogen, ppm	486	388	32	<0.1	<0.1
Basic Nitrogen, ppm	383	378	26		
Hydrogen, Wt %	12.11	12.14	11.60	13.88	13.28
Oxygen, ppm	3400	3100	680	40	40
<u>Group Type, LV % (High Mass)</u>					
Paraffins	4.5	4.5	4.5	3.9	3.8
Naphthenes	41.1	42.7	46.8	80.9	82.5
Aromatics	54.5	52.8	48.7	15.2	13.6
<u>TBP Distillation, °F (Simulated by Chromatography)</u>					
St/5	58/178	58/178	54/177	54/177	53/177
10/30	216/299	215/294	213/288	212/275	212/275
50	398	394	382	363	363
70/90	448/539	446/537	444/531	411/499	410/499
95/99	599/721	594/718	585/703	543/645	542/643

TABLE VI

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 PROPERTIES OF 180-350°F AND 350°F+ PRODUCTS
 PILOT PLANT RUN 76-165
 750°F - 1.5 LHSV - ~2300 PSIA H₂

Run Hours 2123 - 2147

180-350°F Product Inspections

Gravity, °API	45.2
Aniline Point, °F	78.9
Octane No., F-1 Clear	71.2
Nitrogen, ppm	135
Oxygen, Wt %	0.27

Group Type, LV %
 (By Chromatography)

Paraffins	10.7
Naphthenes	66.3
Aromatics	23.0

TBP Distillation, °F

St/5	158/185
10/30	216/238
50	276
70/90	306/344
95/99	356/370

350°F+ Product

Gravity, °API	21.6
Aniline Point, °F	<32
Nitrogen, ppm	447
Oxygen, Wt %	0.40
Smoke Point, mm	7

Group Type, LV %
 (High Mass)

Paraffins	5.1
Naphthenes	29.0
Aromatics	65.9

TBP Distillation, °F (Simulated
 by Chromatography)

St/5	325/363
10/30	379/419
50	456
70/90	501/586
95/99	645/760

TABLE VII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 PILOT PLANT RUN 76-165 AT 2805-2829 HR
PRODUCT PROPERTIES*

180-300°F Product Inspections

Gravity, °API	50.4
Aniline Point, °F	94.6

Group Type, LV %
(By Chromatography)

Paraffins	10.4
Naphthenes	79.3
Aromatics	10.3

300-500°F Product Inspections

Gravity, °API	34.3
Aniline Point, °F	108.3
Smoke Point, mm	19
Freeze Point, °F	-89

Group Type, LV %
(High Mass)

Paraffins	3.2
Naphthenes	82.4
Aromatics	14.5

Existent Gum, ppm	2
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500°F+ Product Inspections

Gravity, °API	25.2
Aniline Point, °F	135.8

*Note: Some inspections are incomplete and will be reported later.

TABLE VIII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106
 PILOT PLANT RUN 76-165 AT 2805-2829 HR
 DETAILED COMPOSITION OF 180-300°F PRODUCT
 BY TWO CHROMATOGRAPHIC TECHNIQUES

Method	A	B
	Procedure 1001	PNA By Chromatography
<u>Composition, LV %</u>		
<u>Paraffins</u>		
C ₆ -	0.5	0.5
i-C ₇	1.7	{ 3.6 }
n-C ₇	1.8	
i-C ₈	2.1	{ 3.9 }
n-C ₈	1.8	
i-C ₉	(5.2)*	{ 2.1 }
n-C ₉	0	
C ₁₀ 's	(1.2)*	0.2
C ₁₁ 's	-	0.1
Total Paraffins	(14.3)*	10.4
<u>Naphthenes</u>		
Cyclopentane	0.04	0.04
Methylcyclopentane	0.7	{ 8.3 }
Cyclohexane	7.7	
C ₇ -Cyclopentanes	6.3	{ 32.6 }
Methylcyclohexane	27.1	
C ₈ -Cyclopentanes	3.7	{ 30.6 }
C ₈ -Cyclohexanes	22.5	
C ₉ -Cyclopentanes	1.7	{ 7.8 }
C ₉ -Cyclohexanes	4.2	
Total Naphthenes	(73.9)*	79.3
<u>Aromatics</u>		
Benzene	0.4	0.4
Toluene	4.9	4.6
Ethylbenzene	1.7	{ 5.1 }
Xylenes	4.0	
C ₉ Aromatics	-	0.2
Total Aromatics	11.0	10.3
<u>Unidentified</u>		
Unidentified	0.7	-

*Although Method A gives a more detailed breakdown, some C₈-C₉ naphthenes are misidentified as C₉-C₁₀ paraffins by this method.

TABLE IX

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 PROPERTIES OF 180-400°F PRODUCT
 PILOT PLANT RUN 76-165
 ~2300 PSIA H₂

Avg. Catalyst Temperature, °F LHSV	749 1.5	774 1.5	776 0.5
Run Hours	2219 - 2243	2315 - 2339	2661 - 2685
<u>180-400°F Product Inspections</u>			
Gravity, °API	42.7	42.9	43.3
Aniline Point, °F	73.6	73.5	98.3
Nitrogen, ppm	233	14.3	
Oxygen, Wt %	0.36		
Octane No., F-1 Clear	72.9	67.0	59.2
Octane No., F-2 Clear			58.0
<u>Group Type, LV % (Low Mass)</u>			
Paraffins		8.1	6.8
Naphthenes		67.0	82.5
Aromatics		24.9	10.7
<u>Group Type, LV % (By Chromatography)</u>			
Paraffins	9.5		
Naphthenes	62.5		
Aromatics	28.0		
Viscosity at 100°F, cSt	0.8194		
<u>TBP Distillation, °F</u>			
St/5	164/187	165/197	153/188
10/30	217/249	220/249	219/254
50	291	294	301
70/90	340/381	343/386	359/391
95/99	394/407	400/410	399/410
<u>ASTM D 86 Distillation, °F</u>			
St/5	222/232		
10/30	238/261		
50	286		
70/90	318/351		
95 EP	363/405		
% Overhead	99.5		

TABLE X

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 106 CATALYST
 PROPERTIES OF 400°F+ PRODUCT
 PILOT PLANT RUN 76-165
~2300 PSIA H₂

Avg. Catalyst Temperature LHSV	749 1.5	774 1.5	776 0.5
Run Hours	2219 - 2243	2315 - 2339	2661 - 2685
<u>400°F+ Product</u>			
Gravity, °API	19.4	21.0	29.8
Aniline Point, °F	52	36.0	118.7
Nitrogen, ppm	393	41	
Oxygen, Wt %	0.28		
Smoke Point, mm	7		
Pour Point, ASTM, °F	-40		
<u>Group Type, LV % (High Mass)</u>			
Paraffins	Incomplete	5.9	7.2
Naphthenes		32.0	78.2
Aromatics		62.1	14.6
<u>TBP Distillation, °F (Simulated by Chromatography)</u>			
St/5	373/401	357/399	369/394
10/30	408/444	407/445	405/439
50	477	476	470
70/90	518/605	514/592	504/568
95/99	660/776	647/760	610/700
<u>ASTM D 86 Distillation, °F</u>			
St/5	426/433		
10/30	438/450		
50	464		
70/90	495/575		
95/EP	642/700		
% Overhead, LV %	99		

TABLE XI

DOE CONTRACT EF-76-C-01-2315
PROPERTIES OF 300°F+ PRODUCT FROM SRC-II

Feed - WOV 3666
 Catalyst - ICR 106

Conditions

775°F, ~2300 psia H₂; 0.5 LHSV, 15,000 SCF/Bbl Recycle Gas

Pilot Plant Run 76-165 at
 2841-2877 Hr

300°F+ Product is 70 Wt %, 66.6 LV % of the Whole Liquid Product

Inspections of 300°F+ Product*

Gravity, °API	30.9
Nitrogen, ppm	0.1
Oxygen, ppm	<50
Hydrogen, Wt %	13.85
JFTOT Rating	No. 1 (Pass, 270°C)
Flash Point, pm (°F)	112
Smoke Point, mm	18
Freeze Point, °F	-53

Group Type, LV % (High Mass)

Paraffins	4.3
Naphthenes	82.4
Aromatics	13.3

Group Type, LV % (FIAM)

Paraffins and Naphthenes	91
Olefins	0
Aromatics	9
Gross Heat of Combustion, Btu/Lb	19,238
Density at 70°C, g/ml	0.8171
Refractive Index (80°C)	1.4409
Molecular Weight	221
Viscosity, cSt, at 100°F	1.659

ASTM D 86 Distillation, °F

St/5	304/323
10/30	335/370
50	399
70/90	433/500
95/EP	544/608
90 Overhead	99

TBP Distillation, °F
 (Simulated by Chromatography)

St/5	179/269
10/30	299/368
50	403
70/90	452/523
95/99	569/665

*Note: Relatively low efficiency batch distillation used to avoid overheating sample. Therefore, some 300°F- material remains. Also, no attempt was made to remove the 550°F+ heavy ends.

TABLE XIII

DOE CONTRACT EF-76-C-01-2315
 YIELDS FROM HYDROTREATING OF SRC-II WITH ICR 113 CATALYST AT 1.0 LHSV, ~1750 PSIA H₂
 PILOT PLANT RUN 65-193; FEED - WOW 3666

Run Hours	674-698	698-722	842-866	1370-1394	1586-1610
Avg. Cat. Temp., °F	750	750	750	750	750
LHSV	0.97	1.00	1.00	1.01	1.04
Total Pressure, psig	1995	2001	1997	2003	2001
H ₂ Mean Pressure, psia	1764	1763	1762	1750	1770
Total Gas In, SCF/Bbl	9855	9914	9599	9556	9517
Recycle Gas, SCF/Bbl	7968	7985	7731	7735	7657
No Loss Prod. Yields	Wt %	Vol %	Wt %	Vol %	Wt %
C ₁	0.08		0.09		0.10
C ₂	0.17		0.17		0.19
C ₃	0.19		0.19		0.19
1C ₄	0.02	0.03	0.02	0.02	0.03
nC ₄	0.09	0.14	0.09	0.15	0.14
C ₅ -180°F	5.27	6.78	5.49	7.04	5.96
180-350°F	34.47	40.88			
350°F-EP	57.05	58.35			
180-300°F			25.00	30.05	
300-500°F			49.86	52.82	
500°F-EP			16.52	15.95	
180-400°F					43.63
400°F-EP					50.69
					47.14
Total C ₅ +	96.80	106.00	96.87	105.85	96.73
					105.80
Actual/No Loss Recovery	99.59/102.91		100.38/102.99		99.98/102.87
H ₂ Cons. (Gross), SCF/Bbl	1887		1929		1868
H ₂ Cons. (Chemical), SCF/Bbl	1813		1855		1782
Liquid Product Nitrogen, ppm (12-Hour Samples)	59		91		149
					149
					535
					535
					412
					412

TABLE XIII

DOE CONTRACT EF-76-C-01-2315
 YIELDS FROM HYDROTREATING OF SRC-II WITH ICR 113 CATALYST AT ~2300 PSIA H₂
 PILOT PLANT RUN 65-193; FEED - WOW 3666

Run Hours	1802 - 1826	1946 - 1970	1994 - 2018	2258 - 2282	2306 - 2330					
Avg. Cat. Temperature, °F LHSV	749 1.05	776 1.02	775 1.00	775 0.50	775 0.51					
Total Pressure, psig H ₂ Mean Pressure, psia	2,497 2,336	2,505 2,314	2,501 2,311	2,501 2,301	2,502 2,298					
Total Gas In, SCF/Bbl	17,106	16,429	16,473	17,977	18,130					
Recycle Gas, SCF/Bbl	14,942	14,221	14,278	15,120	15,262					
	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
<u>No Loss Prod. Yields</u>										
C ₁	0.08		0.09		0.11		0.11		0.10	
C ₂	0.15		0.17		0.19		0.21		0.21	
C ₃	0.18		0.20		0.22		0.29		0.30	
iC ₄	0.02	0.02	0.02	0.03	0.02	0.03	0.03	0.05	0.03	0.04
nC ₄	0.09	0.14	0.08	0.13	0.09	0.14	0.11	0.17	0.13	0.21
C ₅ -180°F	5.88	7.50	4.12	5.31	**	**	4.39	5.65	5.56	7.14
180-350°F	34.08	40.39	33.57	39.79						
350°F-EP	57.27	58.03	59.60	61.14						
180-300°F								25.77	31.02	
300-500°F								49.87*	54.12*	
500°F-EP								16.90*	17.22*	
180-400°F					49.05**	57.39**	50.70	58.81		
400°F-EP					48.12	52.09	43.00	44.95		
Total C ₅ +	97.23	105.91	97.28	106.24	97.16	109.49	98.09	109.40	98.10	109.50
Actual/No Loss Recovery	100.08/103.33		100.69/103.40		100.01/103.38		102.54/104.41		101.38/104.43	
H ₂ Cons. (Gross), SCF/Bbl	2164		2208		2194		2856		2867	
H ₂ Cons. (Chemical), SCF/Bbl	2063		2111		2097		2740		2750	
Liquid Product Nitrogen, ppm (12-Hour Samples)	164		13		8		0.38		0.63	

*Actual cut point closer to 480°F than target of 500°F.

**C₅-180°F material included in this fraction. Perhaps some loss of light ends.

TABLE XIV

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 WHOLE LIQUID PRODUCT PROPERTIES
 PILOT PLANT RUN 65-193
 750°F - ~1750 PSIA H₂ - 1.0 LHSV

Time Onstream, Hr	674 - 698	698 - 722	842 - 866	1370 - 1394	1586 - 1610
<u>Whole Liquid Product Inspections</u>					
Gravity, °API	32.6	32.3	32.1	29.9	30.9
Aniline Point, °F	50.1	49.4	46.8	38.1	41.2
Sulfur, ppm	4	5	8	8	8
Total Nitrogen, ppm	62	84	148	371	(306)
Basic Nitrogen, ppm	56	73	130	367	322
Hydrogen, Wt %	12.08	11.66	11.12	11.24	11.93
Oxygen, ppm	880	1000	1600	2800	3200
<u>Group Type, LV % (High Mass)</u>					
Paraffins	4.1	4.0	4.3	3.9	4.1
Naphthenes	43.7	40.4	41.7	40.1	39.8
Aromatics	52.2	55.6	54.0	55.9	56.1
Molecular Wt	141	142	142	145	147
Refractive Index (80°C)	1.4529	1.4545	1.4560	1.4609	1.4596
<u>TBP Distillation, °F (Simulated by Chromatography)</u>					
St/5	52/177		49/176	59/181	59/180
10/30	211/282		211/282	216/309	215/305
50	384		388	401	400
70/90	442/532		441/531	454/545	453/544
95/99	587/709		585/710	608/746	608/745

TABLE XV

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 WHOLE LIQUID PRODUCT PROPERTIES
 PILOT PLANT RUN 65-193
~2300 PSIA H₂

Avg. Catalyst Temperature, °F LHSV	749 1.0	776 1.0	775 1.0	775 0.5	775 0.5
Time Onstream, Hr	1802 - 1826	1946 - 1970	1994 - 2018	2258 - 2282	2306 - 2330
<u>Whole Liquid Product Inspections</u>					
Gravity, °API	31.3	31.8	32.3	34.8	35.5
Aniline Point, °F	48.0	54.3	54.6	82.7	83.5
Sulfur, ppm	5			11	4
Total Nitrogen, ppm	164	13	8	0.38	0.63
Basic Nitrogen, ppm	154	8	5	-	-
Hydrogen, Wt %	11.57	11.47	11.43	12.18	12.64
Oxygen, ppm	1500	440	390	90	90
<u>Group Type, LV % (High Mass)</u>					
Paraffins	4.3	3.8	3.9	3.6	3.8
Naphthenes	43.3	47.0	47.1	65.4	65.7
Aromatics	52.5	49.1	48.9	31.0	30.5
<u>TBP Distillation, °F (Simulated by Chromatography)</u>					
St/5	55/177	49/178	48/177	48/177	45/176
10/30	214/288	215/297	215/294	214/290	212/282
50	393	393	389	373	369
70/90	445/538	449/536	447/534	434/515	430/514
95/99	595/715	591/710	588/705	563/668	562/670

TABLE XVI

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 PROPERTIES OF 180-350°F PRODUCT
 PILOT PLANT RUN 65-193; 1.0 LHSV

	750 1764 674 - 698	750 1750 1370 - 1394	750 1770 1586 - 1610	749 2336 1802 - 1826	776 2314 1946 - 1970
<u>180-350°F Product Inspections</u>					
Gravity, °API	46.5	45.5	46.2	46.4	46.4
Aniline Point, °F	83.2	79.1	80.8	82.3	81.7
Nitrogen, ppm	16	126	107	52	1.5
Oxygen, ppm	350	1700		730	90
Octane No., F-1 Clear	Incomplete	69.3	69.6	68.4	68.7
Octane No., F-2 Clear	64.9	67.1			
<u>Group Type, LV % (Low Mass)</u>					
Paraffins	8.2				6.3
Naphthenes	74.8				77.7
Aromatics	17.0				16.0
<u>Group Type, LV % (By Chromatography)</u>					
Paraffins		8.0	9.9	Incomplete	
Naphthenes		69.2	70.0		
Aromatics		22.8	21.1		
<u>TBP Distillation, °F</u>					
St/5	163/181	161/182	163/188	164/183	157/182
10/30	213/227	213/232	214/230	211/234	213/228
50	266	271	269	273	269
70/90	295/334	301/337	298/338	301/340	298/336
95/99	342/358	347/362	351/-	349/363	345/358
<u>ASTM D 86 Distillation, °F</u>					
St/5			220/230		
10/30			236/247		
50			262		
70/90			280/310		
95/EP			321/352		
% Overhead			99		

TABLE XVII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 PROPERTIES OF 350°F+ PRODUCT
 PILOT PLANT RUN 65-193; 1.0 LHSV

Average Catalyst Temperature, °F	750 1764	750 1750	750 1770	749 2336	776 2314
H ₂ Pressure, psia	674 - 698	1370 - 1394	1586 - 1610	1802 - 1826	1946 - 1970
<u>350°F+ Product</u>					
Gravity, °API	22.0	20.7	20.9	20.6	22.5
Aniline Point, °F	32	32	32	32	36.9
Nitrogen, ppm	88	529	542	220	15.5
Oxygen, ppm	1200	3400		2200	610
Smoke Point, mm	7	8		7	8
Cetane No.	21				
Freeze Point, °F	-38			-36	
<u>Group Type, LV % (High Mass)</u>					
Paraffins	5.0	4.7	4.5	5.1	4.2
Naphthenes	29.9	26.6	26.8	29.6	33.3
Aromatics	65.1	68.8	68.8	65.4	62.5
<u>Viscosity, cSt</u>					
At 100°F	2.152				
At 210°F	0.9289				
<u>TBP Distillation, °F (Simulated by Chromatography)</u>					
St/5	328/360	329/362	326/363	328/362	329/361
10/30	375/413	381/417	382/421	378/417	374/411
50	449	452	456	455	446
70/90	494/580	497/586	501/590	500/586	490/568
95/99	641/751	648/765	653/777	645/765	622/730
Cu Strip Test, ASTM D 130					
Neutralization No., ASTM D 974, mg KOH/g	Incomplete 0.02				
Molecular Weight			181		177
Refractive Index, 80°C			1.4928		1.4839
Density at 70°C, g/ml			0.8901		0.8767

TABLE XVIII

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 PILOT PLANT RUN 65-193
 PROPERTIES OF 180-300°F PRODUCT

Avg. Catalyst Temperature, °F	750	775
H ₂ Pressure, psia	1763	2298
LHSV	1.0	0.5
Run Hours	698 - 722	2306 - 2330
<u>180-300°F Product Inspections</u>		
Gravity, °API	48.9	49.2
Aniline Point, °F	84.1	87.7
Nitrogen, ppm	4	
Oxygen, ppm	200	70
Octane No., F-1 Clear	70.9	69.7
Octane No., F-2 Clear	68.0	
<u>Group Type, LV % (Low Mass)</u>		
Paraffins		7.4
Naphthenes		79.1
Aromatics		13.5
<u>Group Type, LV % (By Chromatography)</u>		
Paraffins	12.0	10.0
Naphthenes	73.1	75.2
Aromatics	14.9	14.8
<u>TBP Distillation, °F (Simulated by Chromatography)</u>		
St/5	161/178	151/177
10/30	201/219	190/223
50	240	240
70/90	268/289	269/290
95/99	298/326	298/314
<u>ASTM D 86 Distillation, °F</u>		
St/5	215/222	
10/30	224/230	
50	238	
70/90	248/268	
95/EP	276/309	
% Overhead	99.5	

TABLE XIX

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 PROPERTIES OF 300-500°F PRODUCT
 PILOT PLANT RUN 65-193

Avg. Catalyst Temperature, °F	750	775
H ₂ Pressure, psia	1763	2298
LHSV		
Run Hours	698 - 722	2306 - 2330*
<u>300-500°F Product Inspections</u>		
Gravity, °API	27.5	31.4
Aniline Point, °F	31.3	78.0
Nitrogen, ppm	98	
Oxygen, ppm	1200	50
Smoke Point, mm	8	13
Freeze Point, °F	-80	-94
<u>Group Type, LV % (High Mass)</u>		
Paraffins	3.7	2.6
Naphthenes	31.6	58.0
Aromatics	64.7	39.4
Viscosity at 100°F, cSt	1.431	
<u>TBP Distillation, °F (Simulated by Chromatography)</u>		
St/5	278/315	276/310
10/30	331/381	326/366
50	413	398
70/90	446/481	426/462
95/99	492/506	475/494
<u>ASTM D 86 Distillation, °F</u>		
St/5	351/361	
10/30	366/386	
50	405	
70/90	428/454	
95/EP	465/510	
% Overhead	99	
Cu Strip Test, ASTM D 130	1A	

*End point somewhat below target of 500°F for period at 2306-2330 hr.

TABLE XX

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SCR-II WITH ICR 113 CATALYST
 PROPERTIES OF 500°F+ PRODUCT
 PILOT PLANT RUN 65-193

Avg. Catalyst Temperature, °F	750	775
H ₂ Pressure, psia	1763	2298
LHSV	1.0	0.5
Run Hours	698 - 722	2306 - 2330*
<u>500°F+ Product Inspections</u>		
Gravity, °API	13.4	21.4
Aniline Point, °F	38.7	109.9
Nitrogen, ppm	152	
Oxygen, ppm	350	170
Pour Point, °F	+5	-5
<u>Group Type, LV % (22 Component)</u>		
Paraffins	6.1	
Naphthenes	18.0	
Aromatics	75.9	
Viscosity at 100°F, cSt	8.777	
Viscosity at 210°F, cSt	2.114	
<u>TBP Distillation, °F (Simulated by Chromatography)</u>		
St/5	446/521	446/489
10/30	533/560	501/531
50	592	559
70/90	634/710	595/663
95/99	757/849	699/780
<u>ASTM D 1160 Distillation, °F</u>		
St/5	544/559	
10/30	563/575	
50	593	
70/90	633/718	
95/EP	755/857	
% Overhead	99	

*Inspections show that actual initial is somewhat below 500°F for period at 2306-2330 hr.

TABLE XXI

DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 PROPERTIES OF 180-400°F PRODUCT
 PILOT PLANT RUN 65-193

Avg. Catalyst Temperature, °F	750	775	775
H ₂ Pressure, psia	1762	2311	2301
LHSV	1.0	1.0	0.5
Run Hours	842 - 866	1994 - 2018*	2258 - 2282
<u>180-400°F Product Inspections</u>			
Gravity, °API	42.9	44.0	42.6
Aniline Point, °F	75.3	78.3	84.8
Total Nitrogen, ppm	113	1.1	
Basic Nitrogen, ppm	115		
Oxygen, ppm	80	100	70
Octane No., F-1 Clear	69.6	68.0	64.9
<u>Group Type, LV % (Low Mass)</u>			
Paraffins		8.2	5.8
Naphthenes		69.8	75.8
Aromatics		21.9	18.3
<u>Group Type, LV % (By Chromatography)</u>			
Paraffins	9.1		
Naphthenes	63.6		
Aromatics	27.3		
<u>TBP Distillation, °F</u>			
St/5	160/200	60/170	148/189
10/30	219/252	176/221	222/263
50	293	275	311
70/90	341/384	328/374	360/395
95/99	399/410	390/401	405/413
<u>ASTM D 86 Distillation, °F</u>			
St/5	226/242		
10/30	248/266		
50	290		
70/90	324/362		
95/EP	377/440		
% Overhead			

*Product at 1994-2018 hr contains some 180°F material also.

TABLE XXII
 DOE CONTRACT EF-76-C-01-2315
 HYDROTREATING OF SRC-II WITH ICR 113 CATALYST
 PROPERTIES OF 400°F+ PRODUCT
 PILOT PLANT RUN 65-193

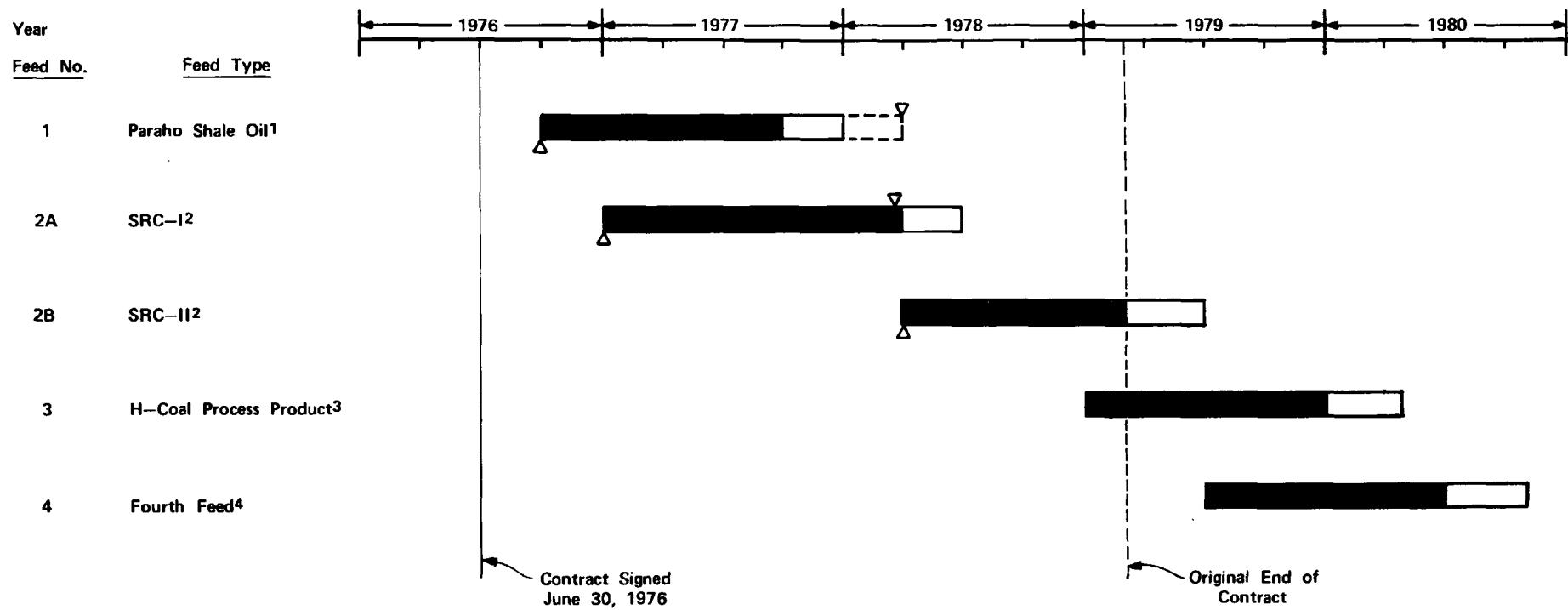
Avg. Catalyst Temperature, °F H ₂ Pressure, psia	750 1762 1.0 842 - 866	775 2311 1.0 1994 - 2018	775 2301 0.5 2258 - 2282
<u>400°F+ Product</u>			
Gravity, °API	19.7	21.0	25.4
Aniline Point, °F	<32	33.3	83.9
Total Nitrogen, ppm	187	19.4	
Basic Nitrogen, ppm	151		
Oxygen, ppm	2000	900	170
Smoke Point, mm	7		10
Freeze Point, °F			-36
<u>Group Type, LV % (High Mass)</u>			
Paraffins	3.3	4.6	5.3
Naphthenes	24.0	30.6	56.0
Aromatics	72.6	64.8	38.6
<u>TBP Distillation, °F (Simulated by Chromatography)</u>			
St/5	379/403	372/399	361/396
10/30	410/446	405/440	405/440
50	480	468	468
70/90	522/611	506/581	503/572
95/99	667/784	637/743	622/732
Molecular Weight	180		180
Refractive Index at 80°C	1.4893		1.4718
Density at 70°C, g/ml	0.8985		0.8643

TABLE XXIII

DOE CONTRACT EF-76-C-01-2315
 DISTRIBUTION OF C₅-180°F PRODUCT FROM SRC-II
 WITH ICR 113 CATALYST AT 750°F AND 1.0 LHSV
 PILOT PLANT RUN 65-193

Time Onstream, Hr	266 - 290	314 - 338	1586 - 1610
Avg. Hydrogen Partial Pressure, psia	2328	2323	1770
<u>Composition, LV % of C₅-180°F (By Chromatography)</u>			
Propane	0.2	-	0.2
Isobutane	0.2	-	0.2
n-Butane	2.3	0.04	2.1
Isopentane	1.8	0.06	2.6
n-Pentane	6.2	2.7	7.0
2,2-Dimethylbutane	0.02	0.01	0.02
2,3-Dimethylbutane	0.4	0.4	0.4
2-Methylpentane	4.0	3.6	3.8
3-Methylpentane	2.8	2.7	2.6
n-Hexane	10.5	10.5	9.5
Isoheptanes	2.5	2.0	2.9
n-Heptane	0.3	0.1	0.2
C ₈ Isoparaffins	0.05	0.01	0.03
Total Paraffins	31.3	22.6	31.6
Cyclopentane	3.6	2.5	{ 14.4 }
Methylcyclopentane	11.7	12.8	
Cyclohexane	43.0	52.8	43.9
Dimethylcyclopentanes,	0.7	0.9	0.8
Ethylcyclopentane			
Methylcyclohexane	1.4	0.4	1.2
Total Naphthenes	60.4	69.4	60.3
Benzene	8.2	8.0	8.0
Toluene	0.1	0.04	0.1
Total Aromatics	8.3	8.0	8.1
Octane Number, F-1 Clear	81.0	82.0	Incomplete
Gravity, °API	60.0	60.0	
			60.9

FIGURE 1
DOE CONTRACT EF-76-C-01-2315
FEED TIMING SCHEDULE (REVISED, JULY 1978)



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.

³At 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.

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

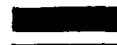
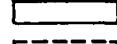
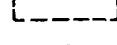
-  Schedule - Feed Analysis and Pilot Plant Work
-  Schedule - Final Report and Design
-  Schedule Extension
-  Actual Start of Work on Feedstock
-  Actual End of Work on Feedstock

FIGURE 2

DOE CONTRACT EF-76-C-01-2315
SCHEDULE FOR FEED 2B (SRC-II)Year
Quarter No.
Month No.
Month
Task No. Description

1 Preliminary Feedstock Analyses and Design Studies

2 Whole Oil Hydrotreating

2A Alternative to Whole Oil Hydrotreating; Hydrotreating of SRC-II Naphtha; Hydrotreating of Blend of SRC-II Light and Heavy Distillates

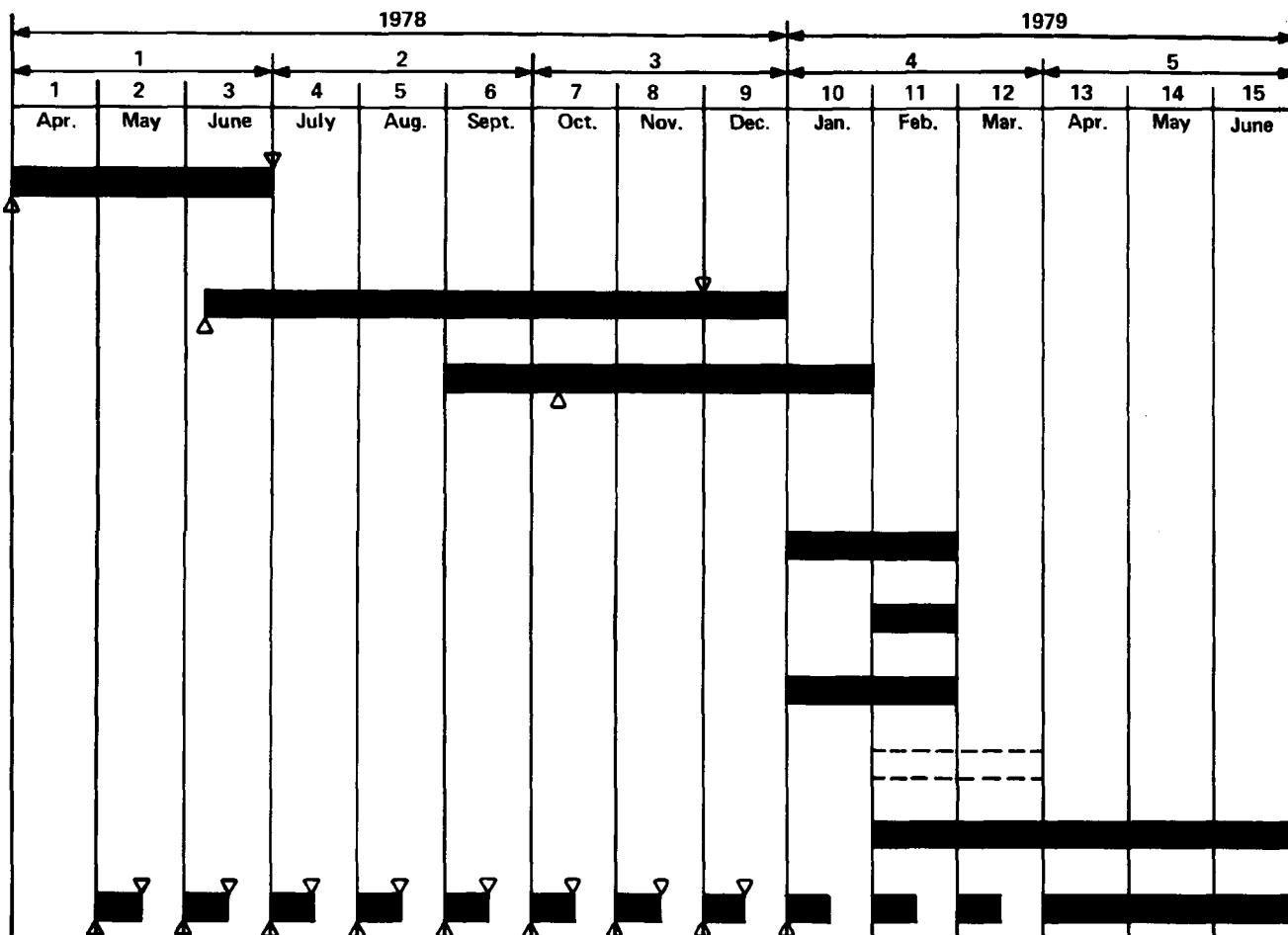
3 Mid-Distillate Hydrotreating¹4 Extinction Recycle Hydrocracking²

4A Fluid Catalytic Cracking

5 Catalytic Reforming³

6 Final Process Design

7 Reports

¹Task 3 may not be necessary, depending on the outcome of Tasks 2 and 2A.²At present, no hydrocracking work is anticipated.³Task 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
 Start Task
 Complete Task