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MASTER

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

Second Interim Report
Laboratory and Pilot Plant Studies
of the Processing of SRC-I

Authors: R. F. Sullivan
B. E. Stangeland
H. C. Chen
C. E. Rudy

CHEVRON RESEARCH COMPANY
Richmond, California 94802

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I. Abstract

Solvent refined coal (SRC) from the SRC-I process is a high melting solid material that contains large amounts of nitrogen, oxygen, and metallic contaminants and is low in hydrogen content. It is a feed that is extremely difficult to convert to transportation fuels in high yields using commercial fixed bed catalytic hydroprocessing technology. In this study, it was demonstrated that SRC-I can be hydrotreated in the presence of a coal-derived solvent at moderate cracking conversions (e.g., 50% conversion to material boiling below 850°F) for at least 1000 hours without serious plugging of the catalyst bed. However, even at these relatively low conversion levels, catalyst deactivation rates are unacceptably high. With higher severity hydroprocessing, high conversions can be obtained and most of the heteroatoms can be removed. However, severe plugging occurs in the catalyst beds in a short period of time.

In subsequent studies to be described in future interim reports, we have shown that coal-derived liquids such as SRC-II and H-Coal process product are relatively attractive feeds for conversion to transportation fuels. We do not regard conversion of SRC-I to transportation fuels by the routes described here as being commercially feasible.

Encl. - See Index of Enclosures

II. Summary of Experimental Studies

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

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

A test of over 1100 hours with a 50/50 SRC-I/creosote oil blend as feed showed that, at relatively modest conversion levels, SRC-I can be processed for relatively long periods in a fixed bed hydrotreater without bed plugging. However, the catalyst fouled rapidly; and the product contained a substantial amount of 850°F+ material. Hydrogen consumption was high. The demonstrated catalyst life would not be acceptable by petroleum processing standards.

With the SRC-I/solvent at higher conversions, the maximum length of uninterrupted operation without some form of plugging was 54 hours. Catalyst bed plugging was a serious problem that would have to be solved before it could be said that SRC-I processing in a fixed bed is commercially feasible. Probably the high ash content, the high metals content, and the coke-forming tendencies of SRC-I all contribute to the plugging. It is also possible that hydrogenation of the recycle oil reduces its solvent power and that this leads to an increased tendency to precipitate asphaltene-like feed constituents within the equipment.

Various solutions could be suggested to try to correct or minimize the plugging problems. These solutions would require research beyond the scope of the present study. We concluded that alternative coal liquids such as SRC-II, H-Coal process product, and EDS product would present fewer downstream process problems than SRC-I and, therefore, should be tested in similar studies before further work is done on conversion of SRC-I to transportation fuels via the routes suggested here. Therefore, it was agreed that work be directed toward the SRC-II product. By mutual agreement of Chevron and DOE, SRC-II was added to the program; and no further pilot plant work was done on the downstream processing of SRC-I and doing engineering studies was not considered to be justified. The results on SRC-II, to be reported in the third interim report, demonstrate the feasibility of processing SRC-II and show that SRC-II is a relatively attractive feed to upgrade to transportation fuels.

III. Contract Objectives and Scope of Work

A. Objectives

The objective of the program is to determine the feasibility and estimate the costs of hydroprocessing four synthetic crude feedstocks to distillate fuels, including high octane gasoline, using presently available technology. To date two feedstocks have been studied: (1) Paraho shale oil, prepared in the indirectly heated mode, and (2) SRC of the type produced at the DOE pilot plant in Tacoma, Washington. The second feedstock was subdivided to include two types of SRC, SRC-I and SRC-II. The first interim report (FE-2315-25) described studies with Paraho shale oil. The present report discusses our work with SRC-I. A third interim report is being prepared to include the studies with SRC-II.

The feasibilities of hydroprocessing each of the synthetic crudes are to be compared through catalyst tests and evaluations, whereby commercial plant yields, hydrogen consumption, product distribution, and product inspection will be estimated. The necessary tests and evaluations for each feedstock are to 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 the proprietary commercial Chevron catalysts identified in the contract.

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

Tests are to 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 are to 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.

B. Schedule

According to the original timing estimate for an individual feedstock, the preliminary feed analyses and pilot plant program for each feedstock would require one year, followed by an additional 14 weeks for completion of the product analyses, pilot plant report, and final process design.

A general schedule was set up for each feedstock to serve as a guideline for work. It was recognized, however, that there is an exploratory nature to processing these feedstocks and that it might be desirable to alter the schedule as information on any single feedstock became available, either to emphasize or deemphasize a certain part of the program.

Figure 2 shows how the general schedule applied to our work on SRC-I. With the permission of the DOE Technical Project Officer, the actual schedule was varied somewhat from the general schedule for the reasons discussed in the next paragraph.

Because of the refractory nature of SRC-I and the plugging problems encountered with fixed bed catalyst systems at the higher conversions, processing SRC-I does not appear to be commercially feasible by the routes selected. Therefore, downstream processing studies on the product from the whole oil hydrotreater were minimized. No middle distillate hydrotreating runs were made, and no process design studies were included. A limited program on hydrocracking and catalytic cracking was included.

During the first quarter of 1978, SRC-II process product became available as a feedstock. Because the properties of SRC-II indicated it would be much more suitable for refining using existing petroleum technology, SRC-II was added to the program as Feed 2B; and the experimental work on SRC-I was discontinued. The results with SRC-II are discussed in the third interim report to be issued in the near future.

IV. Experimental Program

Task 1

A. Feedstock Analysis

Three shipments of SRC-I were received from the Pittsburg and Midway Coal Mining Company at Du Pont, Washington.

Chevron Identification No.	Quantity Drums	Shipment Date	Type of Material
WOW 3406	6	Dec. 14, 1976	SRC Flakes
WOW 3450	6	March 15, 1976	SRC Fines
WOW 3453	6	March 21, 1976	SRC Flakes

The flaked material was from the stainless steel cooling belt. The fines were from the product solidification and storage area dust collectors at the SRC pilot plant. All of our pilot plant runs were made using the flakes rather than fines. At the request of DOE, all of the SRC-I material remaining after completion of the program was shipped to the C. E. Lummus Company for their experimental studies.

Table I shows both the analyses of the SRC-I samples obtained by Chevron and by the Pittsburg and Midway Coal Mining Company. Table II shows that the metal content of SRC-I by emission spectrochemical analyses varies from batch to batch.

B. Feed Preparation

If SRC-I is to be hydroprocessed in a fixed bed reactor, the SRC-I flakes must be in a form which can be pumped at the operating temperatures of the equipment. Air Products and Chemicals, Incorporated,[1]

[1]E. J. Greskowvich, E. N. Givens, and M. A. Collura, "Quarterly Progress Report, Chemical Characterization, Handling, and Refining of SRC to liquid Fuels," ERDA Contract E(49-18)-2003, Report No. FE-2003-21, Corporate Research Department, Air Products and Chemicals, Incorporated, May 1977.

showed that a homogeneous mixture of SRC-I (50 wt %) and creosote oil (50 wt %) can be prepared and handled at the desired temperatures.

The creosote oil used in our tests was obtained from Allied Chemicals (Identification 24-CA). It was washed with water, filtered with a 15-micron filter, and distilled. The 70% overhead from distillation was identified as WOW 3366.

SRC-I; WOW 3406; and creosote oil, WOW 3366, were blended in a 50/50 weight ratio under a nitrogen blanket and heated to 370°F with stirring. Table III shows the inspections of the blend, WOW 3476, and its components. The distillation curves of creosote oil and the 50/50 blend were determined by both thermogravimetric analyses (TGA) and ASTM D 1160 distillations. The difference in data from these techniques is believed to be due to the fact that the TGA is calibrated against petroleum stocks, and the coal-derived oils have different boiling characteristics from the petroleum stocks. Figure 3 shows the distillation curves of SRC-I, creosote oil, and the blend. Table IV shows the metal content of the feed and its ash.

C. Pumping Experiment

A pumping experiment on a 50/50 wt % mixture of SRC-I/creosote oil was designed to determine the temperature needed to get a steady pumping rate and to determine whether stirring is needed to keep the melt homogeneous.

We charged 20 lb of SRC-I and 20 lb of creosote oil to a feed pot surrounded by band heaters and with an internal stirrer attached to the lid. It was placed on a scale. The rate of weight decrease indicated the feed rate. Pumping began after the feed pot was heated to 370°F with stirring for two hours. A Miloroyal pump (Model A) was used to pump the feed to an autoclave at 1500 psig. All the transfer lines were heated to 370°F. A range of steady rates from 85 g/hr to 900 g/hr was achieved.

Then the stirrer was turned off, and the feed pot was cooled to 200°F. We were still able to maintain a steady pumping rate.

Feed samples were taken near the top and the bottom of the feed pot and analyzed for homogeneity. As shown in Table V, the two samples agree within experimental error, indicating that the blend is essentially homogeneous.

D. Filtration of SRC-I/ Creosote Oil Blend

In a subsequent experiment, the 50/50 SRC-I/creosote oil feed blend, WOW 3476, was filtered with commercially available Balston 25-micron and 2-micron filters, respectively. It took three days to filter a barrel of WOW 3476 with the 2-micron filter. Table VI shows the inspections and metal content of these blends before and after filtration. Analysis shows the filtration reduced the ash from 0.11% to 0.07%. At best, only a small amount of ash was removed; and because of the difficulty in filtration, we do not regard this as a promising pretreatment step.

E. Preparation of Simulated Recycle Solvent

In the course of processing the 50/50 SRC-I/creosote oil blend in Task 2, product boiling in the 350-850°F was prepared which was separated by distillation from the rest of the hydrotreated product and used as a solvent for SRC-I in later experiments. This material is referred to as "simulated recycle solvent," and the feed blends prepared using this solvent are discussed under Task 2.

Task 2

Hydrotreating of SRC-I

Table VII lists the hydrotreating pilot plant runs made under Task 2 and Task 4 of this program. All except the last two runs listed were considered to be part of Task 2.

A. Run 87-67: Processing of 50/50 SRC-I/Creosote Oil Feeds

A preliminary pilot plant test was made with the 50/50 wt % SRC-I/creosote oil, WOW 3476, using ICR 106 catalyst at 0.2 LHSV, 2500 psig total pressure, and 10,000 SCF/bbl recycle hydrogen. The pilot plant has six reactors in series to obtain good feed dispersion. Reactor temperatures are controlled by a furnace containing a fluidized sand bath. Due to the exothermic hydrogenation reactions, this pilot plant did not operate isothermally; and temperature gradients were observed across the catalyst beds.

The first reactor contained 390 cc of porous alumina to serve as a guard bed and operated upflow. Each of the other reactors had 130 cc of ICR 106 catalyst and operated downflow. The space rate was based on 650 cc of ICR 106 catalyst.

Figure 4 shows the temperature profiles of all catalyst beds after 180 hours on stream at an average catalyst temperature of 746°F. The temperature of the first catalyst bed was highest, that of the second bed was somewhat lower, and that of the last three beds lower still.

The feed was brought in at an average catalyst temperature of 720°F. Then the catalyst temperature was raised to 747°F. At 234 hours the catalyst temperature was lowered to 725°F to compare with the initial activity. Then the catalyst temperature was raised again to 748°F. Between 80 hours and 330 hours on stream, the catalyst appeared to be relatively stable.

Tables VIII and IX show the inspections of the feed and the products to 330 hours on stream. Figure 5 shows the Ramsbottom carbon, benzene insolubles, hot heptane insolubles, total nitrogen content of the whole liquid products, and the average catalyst temperature as a function of run hour.

Table X shows the analyses for metal content in the whole liquid product from Run 87-67.

Figure 6 shows the ASTM D 1160 (2 mm) distillations of the feed and the product at 151 hours and 306 hours obtained at the same space rate and temperature. The catalyst activity with respect to 850°F+ conversion is about the same in both cases.

Tables XI-XVII, inclusive, are the yields and product properties for Run 87-67. The average chemical hydrogen consumption is about 2700 SCF/bbl at 750°F. Note, however, the scatter in hydrogen consumption results for the different yield periods.

At 330 hours, a pressure drop of 150 lb developed in the six in series reactors. The reactors were flushed with an aromatic solvent at 500°F and hexane at 200°F to remove the product on the catalyst. The sand furnace was cooled down to room temperature.

The outlet pressure of each reactor was measured at a constant inlet pressure to the first reactor. The restriction was in the third catalyst bed (No. 4 reactor).

The catalyst in No. 4 reactor was removed layer by layer. The restriction was in a 40-cc section in the middle of the catalyst bed. The fines, which were mainly the interstitial materials, contained 12.4% carbon and 3.1% iron. Typically, with petroleum stocks, carbon content of this magnitude is not enough to cause plugging in the catalyst bed. However, the 0.11% ash in the feed may have contributed to the plugging.

The Run 87-67 was restarted after replacing the plugged portion of the catalyst bed with fresh catalyst. It was plugged again within a day. No further attempt was made to unplug the unit. Run 87-67 was then shut down.

B. Run 30-27: Processing of 50/50 SRC-I/Creosote Oil Feed

In a second pilot plant test run, Run 30-27 with the 50/50 SRC-I/creosote oil blend, the filtered feed, WOW 3530-1, was used in a three-in-series reactor system which had separate temperature controllers in each reactor for isothermal conditions through all the catalyst beds. This unit also had a standby pumping system to pump creosote oil through the reactor when the feed was discontinued due to a unit upset. The feed left in the reactor could be washed out with creosote oil, and there was less danger of plugging due to coking than in the previous run.

Again, the catalyst was ICR 106. All reactors were downflow; the total volume of catalyst charged was 628 cc: 130 cc in the first reactor and 249 cc in each of the second and the third reactors. In the first reactor, 190 cc of alumina was used upstream from the catalyst bed as a guard bed to protect the catalyst from deposits of metals and ash. The process conditions were idential to Run 87-67: 0.2 LHSV, 2500 psig, and 10,000 SCF/bbl recycle hydrogen.

Run 30-27 was lined out with creosote oil followed by a feed blend of 25/75 SRC-I/creosote oil before the 50/50 blend, WOW 3530-1, was introduced.

Run 30-27 was shut down at 1407 hours when the feed blend, WOW 3530-1, was used up. There was no plugging in the reactors. The time on the 50/50 feed blend was 1103 hours.

Figure 7 is a plot of the total nitrogen content of the whole liquid product as a function of time and temperature. The volume percent of hydrogen in the recycle gas is also shown.

At 475 hours, a bursting disk was ruptured due to plugging in a cold line (external to the reactors). Since the reactor was depressurized, the catalyst may have deactivated. The hydrogen purity in the recycle gas decreased rapidly after the depressurization due to more methane produced.

At 730 hours, a recycle gas bleed equivalent to 500 SCF/bbl was started. The hydrogen purity in the recycle gas improved. The higher the hydrogen purity, the higher was the activity for nitrogen removal.

Figure 8 shows the normalized temperature necessary to maintain 0.5% total nitrogen in the whole liquid product as a function of time on stream. The fouling rate of the catalyst at 80-90% hydrogen purity is very roughly 0.2°F/hr.

Figure 9 shows the distillation curves of the products. Little change in cracking conversion is seen throughout the whole run.

Tables XVIII and XIX present the inspections of the feed blend and the whole liquid products from Run 30-27. Table XX shows the metal content of the feed and the whole liquid products. The guard bed and catalyst removed iron almost completely. Activity to remove titanium decreased with time.

Table XXI shows a yield period obtained at 742 hours on stream.

The products of Run 30-27 from 333 to 826 hours and the products of Run 87-67 from start to 330 hours were blended together and identified as SGQ 6218. The products of Run 30-27 from 826-1399 hours were blended together as SGQ 6246. Both blends were distilled to give three fractions: Start-350°F, 350-850°F, and 850°F+ bottoms. Properties of the blends and the fractions are given in Tables XXII and XXIII. Metals contents of the blends are given in Table XXIV. Further experiments with the 350-850°F fractions are described later in this report.

C. Runs 30-28, 30-29, and 30-30 Processing of SRC-I/Simulated Recycle Solvent

The 350-850°F fraction of the product from SRC-I/creosote oil feed, prepared in Runs 30-27 and 86-67 and described in the preceding paragraph, was used as the solvent to dissolve an equal weight of SRC-I. Table XXV shows the inspections of the solvent, of SRC-I, and of the blend, WOW 3567. Table XXVI gives the metals content of the blend. Figure 10 shows the distillation curves of the blend and its components.

Table XXVII compares properties of the simulated recycle solvent used for the above blend with creosote oil and with actual SRC-II recycle solvent obtained from Pittsburg and Midway Coal Mining Company.

The 50/50 SRC-I/simulated recycle solvent blend was hydrotreated in Pilot Plant Runs 30-28, 30-29, and 30-30. Target processing conditions and the catalyst were identical to those used in Runs 87-67 and 30-27.

In each run, the unit was first lined out with creosote oil feed, WOW 3366. Then, the SRC-I/solvent feed was introduced. In all cases, a substantial pressure drop developed shortly after the feed was started. When the pressure drop exceeded 500 psi, the feed was switched back to creosote oil. It was possible for a time to eliminate the pressure drop in this fashion. The SRC-I/solvent feed was then reintroduced, and the run continued until the pressure drop problem developed again. Finally, in Runs 30-28 and 30-29, it was no longer possible to eliminate the pressure drop; and the runs were shut down and the catalyst removed in layers to determine the location of the plug.

Run 30-28. Total run length was 400 hours. Of this, about 150 hours involved attempts to process the SRC-I/solvent blend with the maximum uninterrupted run length with the blend about 15 hours. The run was further complicated by heater control problems because the heater control for a transfer line between reactors burned out, caused plugging, and had to be replaced. At the end of the run, a plug in the second catalyst bed caused the shutdown.

Run 30-29. Total run length was 250 hours. Of this about 200 hours involved attempts to process the SRC-I/solvent blend. Maximum uninterrupted operation without plugging was 54 hours. Of this period, 22 hours were at 650°F average catalyst temperature and 32 hours at 700°F catalyst temperature. When the run was shut down at 248 hours, restrictions were found in the preheat section of both the second and third reactors.

Run 30-30. Total run length was 1150 hours. However, most of the run was made with creosote oil as feed. (See Section D below.) During 178 hours of attempted operation with the SRC-I/solvent blend, the unit plugged 12 times, and each time the plug was broken by pumping creosote oil. The maximum period of operation without interruption was about 30 hours during which one yield period was obtained, to be discussed in Section E. (See also Tables XXX, XXI, and XXXII.)

D. Run 30-30 Creosote Oil

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

E. Yield and Product Property Comparisons: Hydrotreating of Creosote Oil, SRC-I/Creosote Oil, and SRC-I/Simulated Recycle Solvent

Table XXX compares yields and hydrogen consumptions for "typical" operation with the SRC-I/creosote oil blend with the results obtained from the single yield period obtained with the SRC-I/solvent after a very short period of operation. Because of the short time on stream for the latter period, this sample is probably not representative of lined-out operation. However, the results do show that, at the same conditions, SRC-I/solvent conversion is much higher than with SRC-I/creosote oil. The result is shown graphically in Figure 11. This emphasizes the importance of the high hydrogen content solvent for maximum conversion.

Unfortunately, the high conversion also appears to be responsible for the serious plugging problems observed. All attempts to run at these high conversion levels resulted in plugging. Probably the coke-forming tendencies of the SRC-I and the relatively high metals content both contributed to the plugging. This problem is particularly severe if the more-easily-converted feed molecules are cracked, leaving behind the harder-to-convert feed molecules.

Table XXXI compares the feeds and product properties for SCR-I/creosote oil, SCR-I/solvent, and creosote oil alone.

Table XXXII gives detailed analyses of the fractions of the product from SRC-I/solvent.

F. Denitrification of 350-850°F Hydrotreater SRC-I/Creosote Oil

Insufficient product was available from the SRC-I/simulated recycle solvent runs for downstream processing. Therefore, downstream processing studies were made on the hydrotreated 350-850°F fraction from the SRC-I/creosote oil blend. Inspections of this feed are shown in column one of Table XXVII. This fraction was passed through a second-stage hydrotreater as shown in the schematic flow diagram in Figure 2.

The purpose of this test Pilot Plant Run 66-188 was to lower the nitrogen to less than 1000 ppm to prepare a feed for hydrocracking and catalytic cracking tests. Process conditions were 0.5 LHSV, 2500 psig total pressure; 10,000 SCF/bbl; and two average catalyst temperature levels, 650°F and 700°F. The catalyst was ICR 106.

Figure 12 shows that at 650°F, the total nitrogen content of the liquid product is approximately 800-900 ppm; and at 700°F, the nitrogen content is about 100 ppm. Table XXXIII compares inspections of the feed and products. Figure 13 shows the simulated distillation curves of the feed and products. Table XXXIV shows the yield calculations. The chemical hydrogen consumption is about 2000 SCF/bbl at 700°F.

The products at 650°F and 700°F were blended together, respectively, and identified as SGQ 6268 and SGQ 6269. Table XXXV shows the analyses on these blends and the feed, SGQ 6259. These two product blends of different nitrogen levels were used as feeds in hydrocracking and catalytic cracking tests in Task 4 and 4A.

Referring back to Table XXXI, the results of this second hydrotreatment, the product at 350 ppm nitrogen is quite similar to that obtained from the SRC-I/solvent blend in a single hydrotreating step and, therefore, for first approximations, can serve as a substitute feed for downstream tests. (There are some differences; for example, the product from the SRC-I/solvent contained 1800 ppm asphaltenes and had a lower concentration of aromatics than the twice-processed 350-850°F SRC-I/creosote oil.)

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

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

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

Recycle cut point was approximately 380°F. The feeds were difficult to hydrocrack; so per-pass conversion was low. Operating conditions are given in Table XXXVII along with the yields and hydrogen consumptions. Tables XXXVIII and XXXIX give the properties of the products. The naphtha product is of high quality.

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

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

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

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

An equilibrium catalyst, CCL-4914, withdrawn from an operating FCC Unit was used for this study. It is the same catalyst employed in the earlier catalytic cracking studies with hydrotreated Paraho shale oil. (See FE 2315-25.) It is a moderately active, moderately metal-contaminated zeolite catalyst. Catalyst inspections are shown in Table XL.

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

H/C Ratio, Hydrogen, and Nitrogen Contents of Hydrotreated SRC-I/Creosote Oil

Identification	H/C Atom Ratio	Hydrogen, Wt %	Nitrogen, ppm	FCC Conversion Below 430°F, LV %
SGQ 6268	1.27	9.58	900	48.6
SGQ 6269	1.47	10.93	350	61.7

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

Gasoline selectivity is excellent due to the low coke and light gas yields produced at this low cracking severity. The cycle oils would undoubtedly be very refractive to further cracking. Thus, the conversions achieved (50-60%) are probably close to optimum for producing maximum gasoline yields. However, probably some additional conversion would be desirable to supply sufficient coke make to maintain heat balance in existing commercial units.

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

V. Conclusions

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

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

With the SRC-I/solvent at higher conversions, bed plugging occurred within the first 60 hours on stream. This is a serious problem that would have to be solved before it could be said that SRC-I processing in a fixed bed is commercially feasible. Probably the high ash content, the high metals content, and the coke-forming tendencies of SRC-I all contribute to the plugging problems. It is also possible that hydrogenation of the recycle oil reduces its solvent power and that this leads to an increased tendency to precipitate asphaltene-like feed constituents within the equipment.

Various solutions can be suggested to correct or minimize the plugging problems. These solutions would require research beyond the scope of the present study. We concluded that alternative coal liquids such as SRC-II, H-Coal process product, and EDS product would present fewer downstream process problems than SRC-I and, therefore, should be tested in similar studies before further work is done on conversion of SRC-I to transportation fuels via the routes suggested here. Therefore, it was agreed that work be directed toward the SRC-II product. By mutual agreement of Chevron and DOE, SRC-II was added to the program; and no further work has been done on the downstream processing of SRC-I. Process engineering studies based on the experimental program were not considered to be justified.

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TABLE I

DOE CONTRACT EF-76-C-01-2315

ANALYSES OF SRC-I FROM THE PITTSBURG AND MIDWAY COAL MINING COMPANY

Sample	SRC-I Flakes[1]	SRC-I Flakes[1]	SCR-I Flakes[2]	SRC-I Flakes[2]	SRC-I Fines[3]	SRC-I Fines[3]
Data Source	Chevron Research	Pittsburg and Midway	Chevron Research	Pittsburg and Midway	Chevron Research	Pittsburg and Midway
Sample No.	WOW 3406		WOW 3453		WOW 3450	
Density, g/cc, 75°F	1.214		1.235		1.246	
Ash, Wt %	0.22	0.15	0.17	0.14	0.20	0.20
H, Wt %	6.12	5.86	5.92	5.88	6.01	5.85
C, Wt %	87.78	86.34	87.59	86.77	87.03	86.83
H/C Atom Ratio	0.83	0.81	0.81	0.81	0.80	0.80
O, Wt %	4.52	4.94	5.15	–	4.62	4.22
S, Wt %	0.89	0.71	0.69	0.74	0.67	0.66
Cl, Wt %	0.005		0.005		0.005	
Total N, Wt %	2.04	2.00	2.21	2.11	2.13	2.24
Basic N, Wt %	0.86		0.78		0.91	
Hot Heptane Insolubles, %	96.0		97.2		94.0	
Distillation	TGA[4]		TGA[4]		TGA[4]	
Start	159		163		155	
5%	943		957		935	
10%	1017		1019		1010	
30%	1161		1160		1156	
50%	1232		1241		1231	
70%						
90%						
95%						
End Point	1281		1283		1281	
Rec., %	55.2		55.4		54.8	

[1]Shipped to Chevron Research on December 14, 1976.
[2]Shipped to Chevron Research on March 21, 1977.
[3]Shipped to Chevron Research on March 15, 1977
[4]Distillation simulated by thermogravimetric analysis.

TABLE II

DOE CONTRACT EF-76-C-01-2315 (SRC-I)
METALS IN SOLVENT REFINED COAL

Sample Sample No.	SRC-I Flakes[1] WOW 3406	SRC-I Flakes[2] WOW 3453	SRC-I Fines[3] WOW 3450
Al	41	72	121
B	17	32	-
Ca	60	74	80
Cr	1	2	8
Fe	98	182	354
Mg	2	6	20
Mn	5	-	-
Si	4	24	-
Ti	147	237	323
V	3	-	-
Zn	6	-	-
Na		5	18
K		1	4

[1]Shipped to Chevron Research on December 14, 1976.

[2]Shipped to Chevron Research on March 21, 1977.

[3]Shipped to Chevron Research on March 15, 1977.

TABLE III

DOE CONTRACT EF-76-C-01-2315
PROPERTIES OF FEEDS:
SRC-I, CREOSOTE OIL, AND SRC-I/CREOSOTE OIL BLEND

Description	SRC-I[1]	400-700°F Creosote Oil[2]	50/50 SRC-I/ Creosote Oil
Chevron Identification No.	WOW 3406	WOW 3366	WOW 3476
Inspections			
Gravity, °API	-14.6	-4.9	-7.4
Sulfur, Wt %	0.89	0.64	0.90
Total Nitrogen, Wt %	2.04	0.78	1.46
Basic Nitrogen	0.86	0.45	0.46
Oxygen, Wt %	4.52	1.11	2.70
Hydrogen, Wt %	6.1	5.3/5.4/5.5/6.1	5.7
Carbon	87.8	90.6	89.9
H/C Atom Ratio	0.83	0.73	0.75
Ash, Wt %	0.22	<0.003	0.11
Pour Point, °F			115
Hot C_7 Insolubles, Wt %	96.0	0.0023	52.2
Benzene Insolubles, Wt %		<0.003	30.2
Ramsbottom Carbon, %		0.60	29.0
Chloride, ppm	50	9	30
Metals, ppm			530
Viscosity at 210°F, cSt		2.3	304.1
Molecular Weight		179	

	SRC-I	400-700°F Creosote Oil			50/50 SRC-I/ Creosote Oil	
	TGA[3]	TGA[3]	TBP[4]	ASTM D 1160	TGA[3]	ASTM D 1160
Distillation						
Start	159	147	343	489	153	483
5	943	461	406	545	573	516
10	1017	520	446	575	648	572
30	1161	638	534	628	802	659
50	1232	712	599	669	977	725
70		778	631	696	1231	
90			680	737		
95			687	771		
End Point	1281	1281	726	843	1284	1002
Recovery, %	55.2	88.8	99.0	99.0	72.2	61

[1]SRC-I supplied by Pittsburg and Midway Coal Mining Company from the Du Pont, Washington, pilot plant.
[2]70% overhead from Allied Chemicals creosote oil (Identification 24-CA), water washed and filtered through a 15-micron filter.
[3]Distillation simulated by thermogravimetric analysis.
[4]TBP distillation simulated by chromatography.

TABLE IV

DOE CONTRACT EF-76-C-01-2315
METALS IN 50/50 SRC-I/CREOSOTE OIL
BLEND AND ITS ASH

Sample Sample No. Metals	50/50 SRC-I/Creosote Oil WOW 3476, ppm	Ash From 50/50 SRC-I/Creosote Oil Ash From WOW 3476, %
Al	87	2.07
B	8	0.12
Ca	59	1.64
Cr	1	0.05
Fe	163	4.25
Mg	5	0.20
Mn	-	0.44
Si	50	10.50
Ti	154	4.58
V	3	0.11
Zn	-	1.02

TABLE V

DOE CONTRACT EF-76-C-01-2315
TESTS FOR HOMOGENEITY OF 50 WT % SRC-I AND
50 WT % CREOSOTE OIL BLEND AT 200°F
WITHOUT STIRRING

Sample	Top of Pot	Bottom of Pot
S, Wt %	0.89	0.88
Total N, Wt %	1.43	1.45
Oxygen, Wt %	2.23	2.30
Viscosity at 210°F, cSt	189.1	188.8
Ash, Wt %	0.05	0.06

TABLE VI

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF 50/50 SRC-I/CREOSOTE OIL FEED BLENDS

Sample No.	WOW 3476	WOW 3521[1]	WOW 3530-1[2]
Gravity °API	-7.4	-8.5	-7.5
Ash, Wt %	0.11	0.08	0.07
H, Wt %	5.70	5.99	5.72
C, Wt %	89.94	89.92	89.61
O, Wt %	2.70	2.81	2.57
S, Wt %	0.90	0.77	0.90
Total N, Wt %	1.46	1.46	1.49
Hot Heptane Insolubles, %	52.2	41.5	53.2
Ramsbottom Carbon, %	29.0	30.1	27.2
Viscosity at 210°F, cSt			518.6
Distillation by D 1160 (2 mm)			
Start	483	473	496
5	516	543	566
10	572	605	586
30	659	674	665
50	725	926	902
End Point	1002	990	1048
Recovered, %	61	53	57
Metals, ppm			
Al	87	44	41
B	8	6	4
Ca	59	47	56
Cr	1	1	1
Fe	163	134	55
Mg	5	2	1
Mn	-	3	4
Si	50	17	8
Ti	154	134	172
V	3	3	2
Zn	-	7	7

[1]WOW 3476 filtered with 25-micron Balston filter.
[2]WOW 3476 filtered with 2-micron Balston filter.

TABLE VII

DOE CONTRACT EF-76-01-2315
A SUMMARY OF PILOT PLANT TESTS ON SRC-I
WITH ICR 106 CATALYST

Run	Run Hr	Feed No.	Feed Description	Product Blend No.
87-67	St-333	WOW 3476	50/50 SRC-I/Creosote Oil	SGQ 6218
30-27	333-826	WOW 3530-1	50/50 SRC-I/Creosote Oil	SGQ 6218
30-27	826-1399	WOW 3530-1	50/50 SRC-I/Creosote Oil	SGQ 6246
30-28	St-445	WOW 3567	50/50 SRC-I/Simulated Recycle Solvent	-
30-29	St-260	WOW 3567	50/50 SRC-I/Simulated Recycle Solvent	-
30-30	794-976	WOW 3567	50/50 SRC-I/Simulated Recycle Solvent	Yield Period
66-188	St-390	SGQ 6259	350-850°F Product from SRC-I/Creosote Oil	SGQ 6268 (900 ppm N) SGQ 6269 (350 ppm N)
30-30	0-794,976-1156	WOW 3366	Creosote Oil	-
81-11	1294-1354	SGQ 6269	350-850°F Product (350 ppm N)	Yield Period
81-11	1354-1438	SGQ 6268	350-850°F Product (900 ppm N)	Yield Period

TABLE VIII

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-I/CREOSOTE OIL AT 0.2 LHSV,
~2000 PSIA H_2 AND 10,000 SCF/B RECYCLE H_2

	Run 87-67							
	Feed SRC-I/Creosote 50/50	Product						
Sample No.	WOW 3476							
Run Hour		79	103	127	151	175	234	258
Average Catalyst Temp., °F		719	721	749	746	746	725	751
Average LHSV		0.18	0.18	0.22	0.20	0.21	0.22	0.20
Specific Gravity	1.145	1.042	1.058	1.050	1.044	1.048	1.061	1.040
Gravity, °API	-7.9	4.3	2.2	3.3	4.0	3.5	1.9	4.5
Wt %								
H	5.70	9.80	9.08	9.54	8.85	9.04	9.49	
C	89.94	88.78	88.40	90.91	86.75	89.31	89.13	
O	2.70	0.64	0.89	0.70	-	0.85		0.53
S	0.90	0.04	0.06	0.04	0.08	0.05	0.05	0.01
Total N	1.46	0.48	0.59	0.46	0.46	0.49	0.59	0.39
Basic N	0.46	0.24						
Viscosity, cSt								
at 100°F	No Movement	86.4	134.2	65.5	56.4	60.8	105.7	35.6
at 210°F	304.1	7.35	8.41	6.50	-	6.14	7.80	4.5
Hot C_7 Insolubles, %	52.2	11.5	13.3	10.8	9.39	11.1	12.6	7.74
Ramsbottom Carbon, %	29.0	11.6	12.4	9.66	11.0	11.3	12.7	9.93
Benzene Insolubles, %	30.2	3.12	2.58	2.50	2.87	2.53	3.45	1.92

TABLE IX

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF SRC-I/CREOSOTE OIL AT 0.2 LHSV, ~2000 PSIA H_2 AND 10,000 SCF/B RECYCLE H_2

	Run 87-67			
	Feed SRC-I/Creosote 50/50	← Product →		
Sample No.	WOW 3476			
Run Hour		282	306	330
Average Catalyst Temp., °F		748	747	747
Average LHSV		0.19	0.20	0.22
Specific Gravity	1.145	1.052	1.047	1.062
Gravity, °API	-7.9	3.0	3.6	1.8
Composition, Wt %				
H	5.70	7.70	8.29	7.49
C	89.94	83.53	87.80	82.43
O	2.70	0.68	0.57	0.72
S	0.90	0.04	0.04	0.03
Total N	1.46	0.45	0.43	0.56
Basic N	0.46			
Viscosity, cSt				
at 100°F	No Movement	46.9	41.6	72.0
at 210°F	304.1	5.10	5.16	6.54
Hot C_7 Insolubles, %	52.2	9.41	8.01	11.1
Ramsbottom Carbon, %	29.0	10.4	10.5	12.5
Benzene Insolubles, %	30.2			

TABLE X

DOE CONTRACT EF-76-C-01-2315
METALS IN THE WHOLE LIQUID PRODUCT
FROM SRC-I/CREOSOTE OIL
FROM RUN 87-67

Run Hour	79	151	234
Average Cat. Temp., °F	719	746	725
Average LHSV	0.18	0.20	0.22
Metals, ppm			
Al	7.4	18.7	18.6
B	0.3	0.7	2.3
Ca	5.3	22.1	18.2
Cr	0.1	0.1	0.2
Fe	0.3	1.2	11.4
Mg	0.2	0.4	0.6
Ni	-	0.2	0.9
Si	0.6	1.5	4.7
Ti	27.2	>85	67.3
V	0.3	0.6	0.8
Zn	0.8	-	-

TABLE XI

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	
FEED	WOW 3476	
RUN HOURS	67.0- 79.0	
AVG.CAT.TEMP., F.	723.	
LHSV	0.19	
TOTAL PRESSURE,PSIG	2506.	
H2 MEAN PRESS.,PSIA	2128.	
TOTAL GAS IN,SCF/B	13454.	
RECYCLE GAS, SCF/B	10437.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.23	
C2	0.33	
C3	0.28	
I-C4	0.02	0.05
N-C4	0.16	0.32
C5-300 F.	1.99	3.07
300-550 F.	23.47	28.22
550-650 F.	25.88	29.04
650- EP F.	45.80	46.20
TOTAL C5+	97.15	106.54
ACT./NO LOSS RECOV.	101.38/103.92	
H2 CONS(GROSS),SCF/B	3017.	
H2 CONS(CHEMICAL),SCF/B	2956.	

....LIQUID PRODUCT INSPECTIONS....

C5-300 F. PRODUCT

GRAVITY, API	49.0
NITROGEN, PPM.	199.00
LOW MASS,LV.PC.	
PARAFFINS	2.5
NAPHTHENES	86.1
AROMATICS	11.4
TBP DIST., F.	
ST/ 5	153./ 178.
10/30	182./ 220.
50	241.
70/90	280./ 349.
95/99	397./ 447.

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TABLE XII

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
PRODUCT INSPECTIONS FOR 87-67

RUN	87- 67
FEED	WOW 3476
RUN HOURS	67.0- 79.0
AVG.CAT.TEMP., F.	723.

....LIQUID PRODUCT INSPECTIONS....

300-550 F. PRODUCT

GRAVITY, API	17.1
SULFUR, PPM.	10.0
NITROGEN, PPM.	615.00
MOLECULAR WEIGHT	154.
HIGH MASS,LV.PC.	
PARAFFINS	0
NAPHTHENES	37.7
AROMATICS	62.3
VISCOSITY, CS,100F.	2.591
VISCOSITY, CS,210F.	1.046
TBP DIST., F.	
ST/ 5	259./ 353.
10/30	380./ 446.
50	487.
70/90	519./ 551.
95/99	559./ 577.

550-650 F. PRODUCT

GRAVITY, API	7.2
SULFUR, PPM.	25.0
NITROGEN, PPM.	700.00
MOLECULAR WEIGHT	193.
VISCOSITY, CS,100F.	10.410
VISCOSITY, CS,210F.	4.743
TBP DIST., F.	
ST/ 5	349./ 539.
10/30	550./ 589.
50	609.
70/90	626./ 650.
95/99	658./ 672.

650- EP F. PRODUCT

GRAVITY, API	-6.8
SULFUR, WT.PC.	0.067
NITROGEN, PPM.	9900.00
MOLECULAR WEIGHT	484.
22 COMP., LV. PC.	
PARAFFINS	0
NAPHTHENES	18.1
AROMATICS	80.2
SULFUR COMPOUNDS	1.6

TABLE XIII

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	87- 67	87- 67
FEED	WOW 3476	WOW 3476	WOW 3476
RUN HOURS	91.0- 103.0	163.0- 175.0	246.0- 258.0
AVG.CAT.TEMP., F.	724.	749.	751.
LHSV	0.18	0.21	0.20
TOTAL PRESSURE,PSIG	2510.	2496.	2505.
H2 MEAN PRESS.,PSIA	2138.	2027.	2006.
TOTAL GAS IN,SCF/B	13808.	12317.	12989.
RECYCLE GAS, SCF/B	10456.	9773.	9848.

NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.	WT.PC.	VOL.PC.
C1	0.26		0.30		0.25	
C2	0.42		0.51		0.39	
C3	0.34		0.48		0.35	
I-C4	0.03	0.05	0.04	0.08	0.03	0.06
N-C4	0.18	0.35	0.31	0.61	0.22	0.42
C5-850 F.	66.72	76.63	67.42	77.03	70.71	81.47
850- EP F.	30.65	29.88	28.46	27.65	26.38	25.68
TOTAL C5+	97.38	106.51	95.88	104.69	97.10	107.15
ACT./NO LOSS RECOV.	105.42/104.37		100.00/103.28		101.55/104.10	
H2 CONS(GROSS),SCF/B	3352.		2544.		3141.	
H2 CONS(CHEMICAL),SCF/B	3292.		2472.		3092.	

....LIQUID PRODUCT INSPECTIONS....

C5-850 F. PRODUCT

GRAVITY, API	9.7	8.9	10.5
SULFUR, PPM.	115.0	110.0	< 300.0
NITROGEN, PPM.	1400.00	1400.00	1200.00
H-C7 AS-TENES,WT.PC.	0.017	0.041	0.015
RAMS. CARBON, WT.PC.	0.310	0.319	0.280
OXYGEN, WT.PC.	0.600	0.540	0.459
BENZENE INSOL.,WT PC	< 0.03	< 0.03	< 0.03
VISCOSITY, CS,100F.	7.641	6.515	5.743
VISCOSITY, CS,210F.	1.945	1.755	1.742

850- EP F. PRODUCT

GRAVITY, API	-11.0	-11.4	-11.2
SULFUR, WT.PC.	0.159	0.130	0.110
NITROGEN, PPM.	13400.00		11500.00
H-C7 AS-TENES,WT.PC.	36.900	31.680	29.989
RAMS. CARBON, WT.PC.	42.490	41.810	40.950
OXYGEN, WT.PC.	1.580	1.140	0.919
BENZENE INSOL.,WT PC	10.50	10.80	7.42

TABLE XIV

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	
FEED	WOW 3476	
RUN HOURS	282.0- 294.0	
AVG.CAT.TEMP., F.	752.	
LHSV	0.19	
TOTAL PRESSURE,PSIG	2498.	
H2 MEAN PRESS.,PSIA	1907.	
TOTAL GAS IN,SCF/B	9546.	
RECYCLE GAS, SCF/B	6512.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.35	
C2	0.51	
C3	0.46	
I-C4	0.04	0.07
N-C4	0.30	0.58
C5-643 F.	51.36	60.41
643- EP F.	45.18	48.80
TOTAL C5+	96.54	109.21
ACT./NO LOSS RECOV.	100.82/103.95	
H2 CONS(GROSS),SCF/B	3034.	
H2 CONS(CHEMICAL),SCF/B	2982.	

....LIQUID PRODUCT INSPECTIONS....

C5-643 F. PRODUCT	
GRAVITY, API	13.1
SULFUR, PPM.	65.0
NITROGEN, PPM.	695.00
HYDROGEN, WT. PCT.	10.12
CARBON, WT. PCT.	89.88
OXYGEN, WT.PC.	0.479
HIGH MASS,LV.PC.	
PARAFFINS	9.0
NAPHTHENES	26.7
AROMATICS	64.3
VISCOSITY, CS,100F.	3.804
VISCOSITY, CS,210F.	1.315

643- EP F. PRODUCT	
GRAVITY, API	2.0
SULFUR, WT.PC.	0.180
NITROGEN, PPM.	7800.00
HYDROGEN, WT. PCT.	7.77
CARBON, WT. PCT.	90.88
OXYGEN, WT.PC.	0.670
22 COMP., LV. PC.	
PARAFFINS	0
NAPHTHENES	4.1
AROMATICS	93.3
SULFUR COMPOUNDS	2.6
VISCOSITY, CS,210F.	577.700

TABLE XV

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
YIELDS AND PRODUCT PROPERTIES FOR 87-67

RUN	87- 67	
FEED	WOW 3476	
RUN HOURS	294.0- 306.0	
AVG.CAT.TEMP., F.	750.	
LHSV	0.19	
TOTAL PRESSURE,PSIG	2498.	
H2 MEAN PRESS.,PSIA	1929.	
TOTAL GAS IN,SCF/B	12579.	
RECYCLE GAS, SCF/B	10212.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.33	
C2	0.55	
C3	0.50	
I-C4	0.04	0.08
N-C4	0.30	0.58
C5-300 F.	2.18	3.32
300-550 F.	24.46	29.27
550-650 F.	26.42	29.48
650-850 F.	16.07	17.26
850- EP F.	26.42	30.25
TOTAL C5+	95.59	109.60
ACT./NO LOSS RECOV.	104.23/103.06	
H2 CONS(GROSS),SCF/B	2367.	
H2 CONS(CHEMICAL),SCF/B	2310.	

....LIQUID PRODUCT INSPECTIONS....

C5-300 F. PRODUCT	
GRAVITY, API	47.6
SULFUR, PPM.	5.1
NITROGEN, PPM.	192.00
MOLECULAR WEIGHT	104.
LOW MASS.LV.PC.	
PARAFFINS	7.5
NAPHTHENES	78.9
AROMATICS	13.7
VISCOSITY, CS,100F.	2.283
VISCOSITY, CS,210F.	0.430
TBP DIST., F.	
ST/ 5	56./ 173.
10/30	180./ 194.
50	225.
70/90	269./ 312.
95/99	330./ 358.

TABLE XVI

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
PRODUCT PROPERTIES FOR 87-67

RUN	87- 67
FEED	WOW 3476
RUN HOURS	294.0- 306.0
AVG.CAT.TEMP., F.	750.
LHSV	0.19

....LIQUID PRODUCT INSPECTIONS....

300-550 F. PRODUCT

GRAVITY, API	16.4
SULFUR, PPM.	2.2
NITROGEN, PPM.	198.00
MOLECULAR WEIGHT	162.
HIGH MASS,LV.PC.	
PARAFFINS	11.9
NAPHTHENES	24.9
AROMATICS	63.2
VISCOSITY, CS,100F.	2.462
VISCOSITY, CS,210F.	1.015
TBP DIST., F.	
ST/ 5	263./ 360.
10/30	397./ 449.
50	483.
70/90	515./ 543.
95/99	550./ 560.

550-650 F. PRODUCT

GRAVITY, API	6.4
SULFUR, PPM.	24.0
NITROGEN, PPM.	985.00
MOLECULAR WEIGHT	197.
22 COMP., LV. PC.	
PARAFFINS	0.8
NAPHTHENES	48.9
AROMATICS	50.3
SULFUR COMPOUNDS	0.0
VISCOSITY, CS,100F.	9.991
VISCOSITY, CS,210F.	2.195
TBP DIST., F.	
ST/ 5	510./ 543.
10/30	558./ 590.
50	605.
70/90	620./ 641.
95/99	649./ 660.

TABLE XVII

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
PRODUCT PROPERTIES FOR 87-67

RUN	87- 67
FEED	WOW 3476
RUN HOURS	294.0- 306.0
AVG.CAT.TEMP., F.	750.
LHSV	0.19

....LIQUID PRODUCT INSPECTIONS....

650-850 F. PRODUCT

GRAVITY, API	1.2
SULFUR, PPM.	110.0
NITROGEN, PPM.	3200.00
MOLECULAR WEIGHT	241.
H-C7 AS-TENES,WT.PC.	0.046
BENZENE INSOL.,WT PC	0.03
RAMS. CARBON, WT.PC.	0.270
22 COMP., LV. PC.	
PARAFFINS	0.2
NAPHTHENES	4.9
AROMATICS	91.8
SULFUR COMPOUNDS	3.1
VISCOSITY, CS,100F.	128.600
VISCOSITY, CS,210F.	6.902
TBP DIST., F.	
ST/ 5	543./ 638.
10/30	650./ 678.
50	700.
70/90	744./ 808.
95/99	828./ 862.

850- EP F. PRODUCT

GRAVITY, API	-9.8
SULFUR, WT.PC.	0.150
NITROGEN, PPM.	13000.00
H-C7 AS-TENES,WT.PC.	52.600
BENZENE INSOL.,WT PC	9.66
RAMS. CARBON, WT.PC.	42.540

WHOLE LIQUID PRODUCT

GRAVITY, API	3.6
SULFUR, WT.PC.	0.045
NITROGEN, PPM.	4300.00
H-C7 AS-TENES,WT.PC.	8.010
RAMS. CARBON, WT.PC.	10.500
OXYGEN, WT.PC.	0.570
VISCOSITY, CS,100F.	41.600
VISCOSITY, CS,210F.	5.200

TABLE XVIII

DOE CONTRACT EF-76-C-01-2315

HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL AT 0.2 LHSV, 2000 PSIA H_2, AND
10,000 SCF/BBL RECYCLE GAS WITH ICR 106
INSPECTIONS OF FEED AND PRODUCT

Run 30-27

Sample No.	Feed SRC-I/ Creosote 50/50 WOW 3530-1	Product							
Run Hour		345	369	526	574	718	742	790	862
Average Catalyst Temp., °F		750	749	751	750	750	750	750	766
Average LHSV		0.20	0.20	0.18	0.20	0.20	0.18	0.19	0.20
% H_2 in Recycle Gas		89	88	82	76	74	80	90	95
Specific Gravity	1.148	1.033	1.039	1.053	1.072			1.069	
Gravity, °API		5.5	4.7	2.9	0.5			0.90	
Wt %									
H	5.72	9.07	8.92				8.21		
C	89.61	89.95	89.84				90.36		
O	2.57	0.43					0.83		0.73
S	0.90	<0.03	0.04	0.05					
Total N	1.49	0.29	0.25	0.39	0.60	0.73	0.69	0.60	0.51
Viscosity, cSt									
At 100°F		32.5	22.5	53.8	92.2	159.3		102.1	49.4
At 210°F	518.6	4.18	8.27	5.62	6.81	9.12		7.35	5.13
Hot C_7 Insolubles, %	53.1	5.99	7.10	7.18	10.60	12.13	11.73	10.64	8.74
Ramsbottom Carbon, %	27.2	8.84	9.29	9.70	11.9	13.6	12.8	12.0	9.21
Benzene Insolubles, %	11.61	0.85	1.32	3.01	3.64	4.61	4.82	4.21	2.83
Ash, Wt %	0.07	0.02							

TABLE XIX

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL AT
0.2 LHSV, 2000 PSIA H_2, AND
10,000 SCF/BBL RECYCLE GAS WITH ICR 106
INSPECTIONS OF FEED AND PRODUCT

Run 30-27

	Feed SRC-I/Creosote 50/50	← Product →			
Sample No.	WOW 3530-1				
Run Hour		979	1027	1195	1387
Average Catalyst Temp., °F		781	779	778	780
Average LHSV		0.20	0.20	0.20	0.21
% H_2 in Recycle Gas		84	74		83
Specific Gravity	1.148		1.079	1.082	
Gravity, °API					
Wt %					
H	5.72				
C	89.61				
O	2.57	0.60	0.71	0.78	1.03
S	0.90		0.07	0.08	
Total N	1.49	0.48	0.68	0.75	0.64
Viscosity, cSt					
At 100°F		48.9	78.9	100.0	63.6
At 210°F	518.6	5.14	6.49	6.87	3.34
Hot C_7 Insolubles, %	53.1	8.00	18.78	11.29	12.44
Ramsbottom Carbon, %	27.2	9.88	11.60	12.59	11.47
Benzene Insolubles, %	11.61	3.34	6.12	8.91	4.85
Ash, Wt %	0.07				

TABLE XX

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL AT 0.2 LHSV,
2000 PSIA H_2, AND 10,000 SCF/BBL RECYCLE GAS WITH ICR 106
METAL ANALYSIS OF FEED AND PRODUCT

Run 30-27

	Feed SRC-I/Creosote 50/50	Product					
Sample No.	WOW 3530-1						
Run Hour		345	417	742	979	1027	1195
Average Catalyst Temp., °F		750	765	750	781	779	778
Average LHSV		0.20	0.19	0.18	0.20	0.20	0.20
Metals, ppm							
Al	41	1.1	2.3	–[1]	2.2	19.2	17.2
B	4	–[1]	–[1]	2.4	1.2	2.4	3.0
Ca	56	0.3	1.6	38	3.8	28.3	24.1
Cr	1	0.2	0.3	0.1	0.1	0.4	0.2
Fe	55	–[1]	–[1]	0.7	0.4	0.9	0.6
Mg	1	–[1]	–[1]	0.8	0.1	1.0	1.2
Mn	4	–[1]	–[1]	–[1]	–[1]	1.3	–[1]
Si	8	1.0	–[1]	6.2	–[1]	1.7	1.7
Ti	172	4.5	8.9	–[1]	11.8	70.0	67.5
V	2	–[1]	–[1]	0.8	0.5	1.3	1.2
Zn	7	–[1]	–[1]	1.5	–[1]	2.3	–[1]

[1]Metals not detected.

TABLE XXI

DOE CONTRACT EF-76-C-01-2315

HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
YIELD DATA FOR RUN 30-27

RUN	30- 27	
FEED	WOW 3530	
RUN HOURS	730.- 742.	
AVG.CAT.TEMP., F.	750.	
LHSV	0.18	
TOTAL PRESSURE,PSIG	2500.	
H2 MEAN PRESS.,PSIA	1957.	
TOTAL GAS IN,SCF/B	12943.	
RECYCLE GAS, SCF/B	10250.	
NO LOSS PROD.YIELDS	WT.PC.	VOL.PC.
C1	0.30	
C2	0.32	
C3	0.27	
I-C4	0.02	0.04
N-C4	0.14	0.28
C5-300 F.	0.68	0.98
300-550 F.	17.93	20.79
550-650 F.	29.31	31.97
650-767 F.	14.85	15.63
767- EP F.	34.03	32.46
TOTAL C5+	96.81	101.84
ACT./NO LOSS RECOV.	101.80/103.50	
H2 CONS(GROSS),SCF/B	2693.	
H2 CONS(CHEMICAL),SCF/B	2642.	

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TABLE XXIII

DOE CONTRACT NO. EF-76-C-01-2315
INSPECTION OF THE PRODUCT BLEND AND PRODUCT FRACTIONS
FROM HYDROTREATING 50/50 SRC-I/CREOSOTE OIL BLEND[1]

	SGQ 6246	Cut 1 St-350°F	Cut 2[2] 350-850°F	Cut 3 850°F+
Vol % of SGQ 6246	100	0.9	73.2	26.0
Gravity, °API	0.2	42.8	5.6	-12.3
Wt %				
Hydrogen	8.28	11.98	8.80	7.07
Carbon	90.51	81.36	91.53	87.34
Oxygen	0.76	0.51	0.64	0.82
Sulfur	0.032	0.038	0.012	
Total Nitrogen	0.63	0.118	0.292	1.30
Hot C_7 Insolubles, %	10.25		0.021	39.38
Sulfur in Hot C_7 Insolubles, %	0.20			
Ramsbottom Carbon, %	8.02		0.35	40.84
Benzene Insolubles, %	5.01		<0.03	14.20
Ash, Wt %	0.04			
Viscosity, cSt				
at 100°F			9.36	No Flow
at 210°F			2.10	No Flow
ASTM D 1160 Distillation, °F				
St/5	399/499		444/500	
10/30	531/591		541/594	
50	633		619	
70/90	729/		658/729	
95/EP	/1060		779/845	
LV % Overhead	88		99	
Group-Type Analysis, LV % (22 Component)				
Paraffins	0		0	
Naphthenes	3.9		4.7	
Aromatics	94.5		93.7	
Sulfur Compounds	(1.6)		(1.6)	

[1]Product from Pilot Plant Run 30-27 at 826-1399 hr.

[2]This cut is identified as SGQ 6259 and used as the feed in Run 66-188.

TABLE XXIV

DOE CONTRACT EF-76-C-01-2315
METAL CONTENT OF PRODUCT BLENDS
FROM HYDROTREATING
50/50 SRC-I/CREOSOTE OIL

Blend	SGQ 6218[1]	SGQ 6246[2]
Metals, ppm		
Al	24	11
B	3	2
Ca	32	15
Fe	4	1
Na	2	-
Si	3	3
Ti	84	43
V	1	1
Zn	3	-

[1]The blend of products of Run 87-67 from start to 330 hours and Run 30-27 from 333 to 826 hours.

[2]The blend of products of Run 30-27 from 826 to 1399 hours.

TABLE XXV

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF 50/50 SRC-I/SIMULATED RECYCLE SOLVENT[1]
BLEND AND ITS COMPONENTS

Description Sample	Solvent 350-850°F[1]	SRC-I WOW 3406	50/50 SRC-I/ Solvent WOW 3567
Gravity, °API	5.4	-14.6	-2.9
Wt %			
Hydrogen	8.86	6.12	6.76
Carbon	89.68	87.78	86.12
Oxygen	0.59	4.52	2.53
Sulfur	0.008	0.89	0.54
Total Nitrogen	0.22	2.04	1.29
Hot C_7 Insolubles, %	0.0128	96.0	40.8
Ramsbottom Carbon, %	0.28		28.6
Benzene Insolubles, %	<0.03		14.0
Ash, Wt %		0.22	0.09
Viscosity, cSt			
at 100°F	7.98		No Flow
at 210°F	1.98		2293
Group-Type, LV % (22 Component)			
Paraffins	0		
Naphthenes	8.2		
Aromatics	90.7		
Sulfur Compounds	(1.1)		
ASTM D 1160 Distillation, °F			
St/5	426/486		489/562
10/30	512/568		578/649
50	602		753
70/90	639/718		
95/EP	774/834		/1041
LV % Overhead	98		65

[1]The 350-850°F cut of the product blend, SGQ 6218, from Runs 30-27 and 87-67.

TABLE XXVI

DOE CONTRACT EF-76-C-01-2315
METAL CONTENT OF 50/50
SRC-I/SIMULATED RECYCLE SOLVENT[1] BLEND

Sample	WOW 3567
Metals, ppm	
Al	39
B	6
Ca	49
Cr	1
Fe	107
Ti	147

[1]The 350-850°F cut from SGQ 6218, which is a blend of products from Runs 30-27 and 87-67.

TABLE XXVII

DOE CONTRACT EF-76-C-01-2315
PROPERTIES OF CREOSOTE OIL,
TACOMA RECYCLE SOLVENT, AND
350-850°F PRODUCT FROM
50/50 SRC-I/CREOSOTE OIL

	350-850°F Product From 50/50 SRC-I/ Creosote Oil		Creosote Oil	Tacoma Recycle Solvent
Chevron ID No.	SGQ 6259[1]	Cut 2[2] ASL 4150	WOW 3366[3]	SGQ 6099[4]
Gravity, °API	5.6	5.4	-4.9	1.5
Wt %				
Hydrogen	8.80	8.86	5.63	7.05
Carbon	91.53	89.68	90.70	87.79
Oxygen	0.64	0.59	1.11	2.54
Sulfur	0.012	0.008	0.64	0.52
Total Nitrogen	0.292	0.22	0.78	0.92
Hydrogen/Carbon Atom Ratio	1.15	1.18	0.74	0.96
Hot C_7 Insolubles, %	0.021	0.013	0.0023	4.9
Ramsbottom Carbon, %	0.35	0.28	0.60	3.7
Benzene Insolubles, %	<0.03	<0.03	<0.03	1.38
Viscosity, cSt				
at 100°F	9.4	8.0		97.6
at 210°F	2.1	2.0	2.3	36.1
ASTM D 1160 Distillation, °F				
St/5	444/500	426/486	489/545	445/473
10/30	441/594	512/568	575/628	491/533
50	619	602	669	592
70/90	658/729	639/718	696/737	654/769
95/EP	779/845	774/834	771/843	864/911
LV % Overhead	99	98	99	95.5
Group-Type Analysis, LV % (22 Component)				
Paraffins	0	0		0.9
Naphthenes	4.7	8.1		3.9
Aromatics	93.7	90.8		88.9
Sulfur Compounds	(1.6)	(1.1)		6.3

[1] 350-850°F product from Pilot Plant Run 30-27 processing 50/50 SRC/creosote oil at 826-1399 hr.
[2] 350-850°F product from Pilot Plant Run 86-67 at St-330 hr and Run 30-27 at 333-826 hr.
[3] 70% overhead from Allied Chemical creosote oil.
[4] From Pittsburg and Midway Coal Mining Company, Sple 697.

TABLE XXVIII

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

Run 30-30

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

TABLE XXIX

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

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

TABLE XXX

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

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

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

TABLE XXXI

DOE CONTRACT EF-76-C-01-2315
INSPECTIONS OF CREOSOTE OIL, SOLVENT, 50/50 SRC-I/CREOSOTE OIL, 50/50 SRC-I/SOLVENT, AND THE PRODUCTS WITH ICR 106 CATALYST

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

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

TABLE XXXII

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

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

*Uncorrected for any loss in light ends.

TABLE XXXIII

DOE CONTRACT EF-76-C-01-2315
HYDROPROCESSING THE 350-850°F PORTION OF THE HYDROTREATED
PRODUCT OF 50/50 SRC-I/CREOSOTE OIL
0.5 LHSV, 2500 PSIG TOTAL PRESSURE,
10,000 SCF/BBL WITH ICR 106

Run 66-188

	Feed SGQ 6259	Product					
Run Hr		83	131	323	347	371	383
Average Cat. Temp., °F		650	700	650	702	699	700
Average LHSV		0.50	0.50	0.52	0.52	0.48	0.44
Gravity, °API	5.6	11.6	13.4	10.9	15.5	15.2	14.9
Wt %							
Hydrogen	8.48	10.25	11.0	9.72	11.12	10.79	10.92
Carbon	91.06	90.18	89.45	89.35	88.95	88.30	88.88
Oxygen	0.64	0.30	0.17	0.40	0.095	0.14	0.14
ppm							
Sulfur	120	15	20	20	42	58	100
Total Nitrogen	2920	840	143	995	46	88	118
Viscosity, cSt							
at 100°F	9.36			7.20			
at 210°F	2.10			1.98			
Hot C_7 Insolubles, ppm	207	0	0	88	9	38	33
Ramsbottom Carbon, %	0.35	0.20	0.17	0.19	0.16	0.15	0.20
Benzene Insolubles, %	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Group-Type, LV % (22 Component)							
Paraffin	0		0			0	0
Naphthenes	4.7		30.4			39.5	37.3
Aromatics	95.3		69.6			60.5	62.7

TABLE XXXIV

DOE CONTRACT EF-76-C-01-2315
HYDROPROCESSING THE 350-850°F PORTION OF HYDROTREATED
PRODUCT OF 50/50 SRC-I/CREOSOTE OIL WITH ICR 106
0.5 LHSV, 2500 PSIG TOTAL PRESSURE, 10,000 SCF/BBL RECYCLE GAS RATE

Run No. 66-188
Feed SGQ 6259

Run Hr	335-347		347-371		371-383	
Average Cat. Temp., °F	702		700		700	
LHSV	0.52		0.48		0.44	
Total Pressure, psig	2,500		2,498		2,500	
H_2 Mean Pressure, psia	2,390		2,385		2,398	
Total Gas In, SCF/Bbl	12,516		12,045		13,175	
Recycle Gas, SCF/Bbl	10,246		10,034		11,220	
No Loss Product Yields	Wt %	Vol %	Wt %	Vol %	Wt %	Vol %
Methane	0.01		0.01		0.01	
Ethane	0.11		0.09		0.09	
Propane	0.08		0.07		0.07	
Isobutane	0.01	0.02	0.01	0.01	0.01	0.01
n-Butane	0.04	0.07	0.03	0.05	0.03	0.06
Total C_5+	101.79	109.24	101.50	108.68	101.41	108.36
Act./No Loss Recovery	101.47/103.19		101.95/102.84		105.02/102.76	
H_2 Cons. (Gross), SCF/Bbl	2,269		2,011		1,955	
H_2 Cons. (Chemical), SCF/Bbl	2,165		1,930		1,872	

TABLE XXXV

DOE CONTRACT EF-76-C-01-2315
HYDROPROCESSING THE 350-850°F PORTION OF THE
HYDROTREATED PRODUCT OF 50/50 SRC-I/CREOSOTE OIL
WITH ICR 106 CATALYST
0.5 LHSV, 2500 PSIG TOTAL PRESSURE, 10,000 SCF/BBL

Pilot Plant Run 66-188

Identification	Feed SGQ 6259[1]	Product SGQ 6268[2]	Product SGQ 6269[3]
Inspections			
Gravity, °API	5.6	11.8	14.0
Wt %			
Hydrogen	8.48	9.59	10.85
Carbon	91.06	90.11	88.19
Oxygen	0.64	0.35	0.18
Sulfur, ppm	120	52	74
Total Nitrogen, ppm	2920	900	350
Viscosity cSt			
at 100°F	9.36	7.40	6.51
at 210°F	2.10	1.86	1.86
Hot C_7 Insolubles, ppm	207	64	<20
Ramsbottom Carbon, %	0.35	0.20	0.18
Benzene Insolubles, %	<0.03	<0.03	<0.03

[1]Feed, 350-850 portion of the hydrofined product of 50/50 SRC/creosote oil.

[2]The blend of products at 650°F.

[3]The blend of products at 700°F (includes some "transition" product with higher nitrogen content than typical of 700°F operation).

TABLE XXXVI

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

Identification	SGQ 6268	SGQ 6269
Inspections		
Gravity, °API	11.8	14.0
Aniline Point, °F	<32	53.9
Sulfur, ppm	52	74
Total Nitrogen, ppm	900	350
Metals, ppm	<1	<1
Hydrogen, Wt %	9.58	10.93
Carbon, Wt %	89.98	88.83
Oxygen, Wt %	0.35	0.18
Group Type, LV % (Mass Spectrometric 22-Component)		
Paraffins	0	0
Naphthenes	24.6	35.6
Aromatics	75.4	64.4
Viscosity cSt		
at 100°F	7.40	6.51
at 210°F	1.86	1.86
Hot C_7 Insolubles, ppm	64	<20
Ramsbottom Carbon, %	0.20	0.18
Benzene Insolubles, %	<0.03	<0.03
TBP Distillation, °F (Simulated by Chromatography)		
St/5	178/418	175/407
10/30	455/531	451/519
50	584	576
70/90	613/680	606/672
95/99	731/832	725/835

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

TABLE XXXVII

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

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

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TABLE XXXVIII

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

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

TABLE XXXIX

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

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

TABLE XL

DOE CONTRACT EF-76-C-01-2315
CRACKING CATALYST INSPECTIONS

Identification No.	CCL-4914
Catalyst Type	Davison CBZ-1
Source	Equilibrium Catalyst from an FCC Unit
Chevron Research Activity, LV % Conversion*	69.7
Physical Properties	
Surface Area, m/g	107
Apparent Bulk Density, g/cc	0.779
Pore Volume, cc/g	0.38
Particle Size Distribution, Wt %	
0-20 μ	0.0
20-40 μ	0.5
40-80 μ	64.0
80+ μ	35.5
Average Particle Size, μ	72.6
Metals, ppm	
Nickel	266
Vanadium	318
Iron	3020
Copper	9
Chromium	75

*Chevron Research activity is defined as the conversion (LV %) obtained in cracking a light East Texas gas oil (35.4°API; 500-700°F) in a fixed-fluidized test unit (FCTU) operated in a cyclic manner. Conversion is defined as 100- vol %, 430°F+ liquid. Test conditions are:

Reactor Temperature, °F	925
Feed Rate, ml/Min.	30
Feed Period, Min.	10
Catalyst Charge, g	600

TABLE XLI

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

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

TABLE XLII

DOE CONTRACT EF-76-C-01-2315

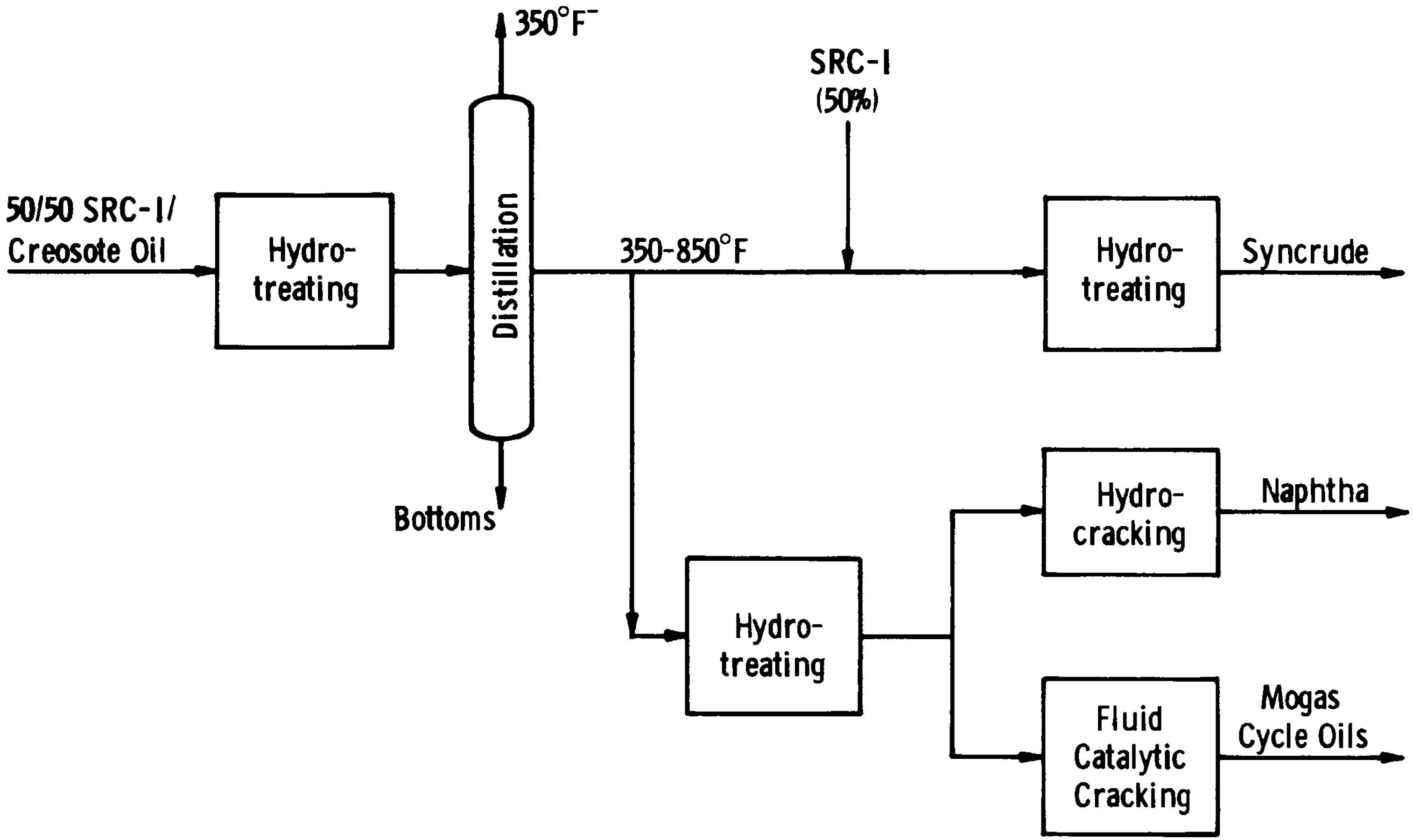
CATALYTIC CRACKING OF HYDROPROCESSED SRC-I/CREOSOTE OILS - PRODUCT INSPECTIONS

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

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

FIGURE 1

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



CHEVRON RESEARCH COMPANY
RICHMOND, CALIFORNIA

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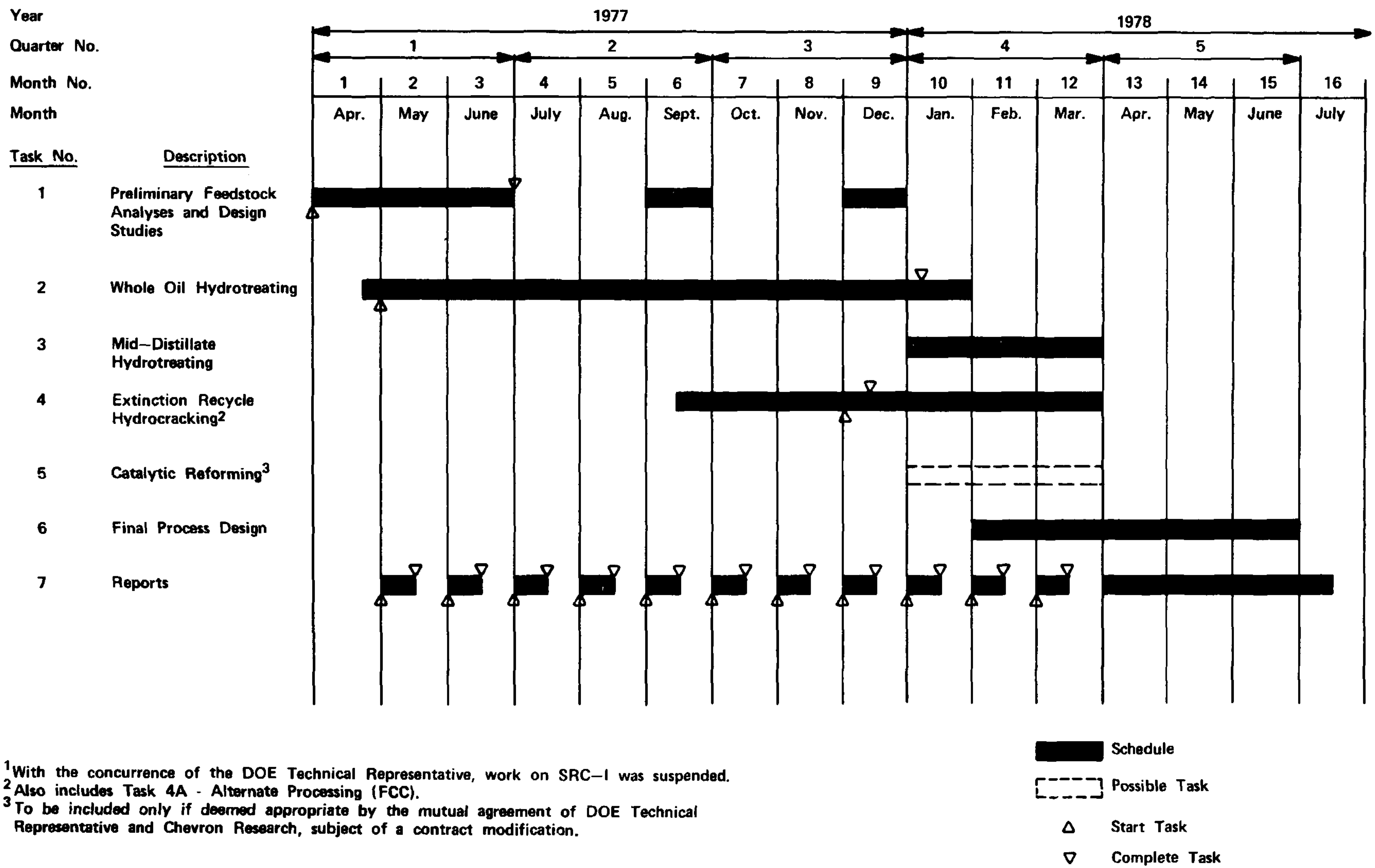
FIGURE 2

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



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

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FIGURE 3

DOE CONTRACT EF–76–C–01–2315
DISTILLATION CURVES OF SRC–I, CREOSOTE OIL, AND
50/50 SRC–I/CREOSOTE OIL BLEND

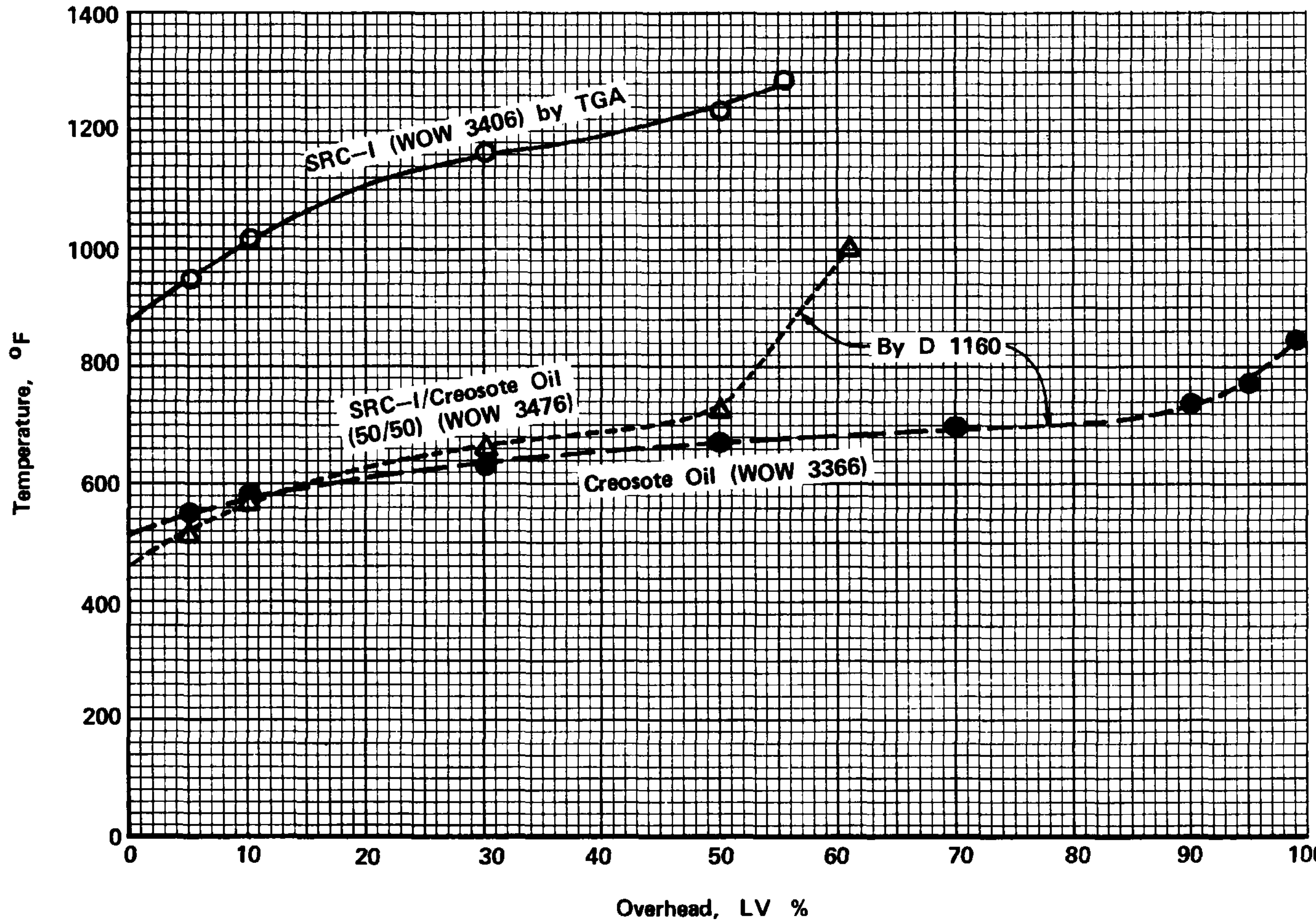

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FIGURE 4

PILOT PLANT RUN 87–67
DOE CONTRACT EF–76–C–01–2315
50/50 SRC–I/CREOSOTE OIL HYDROPROCESSING WITH ICR 106

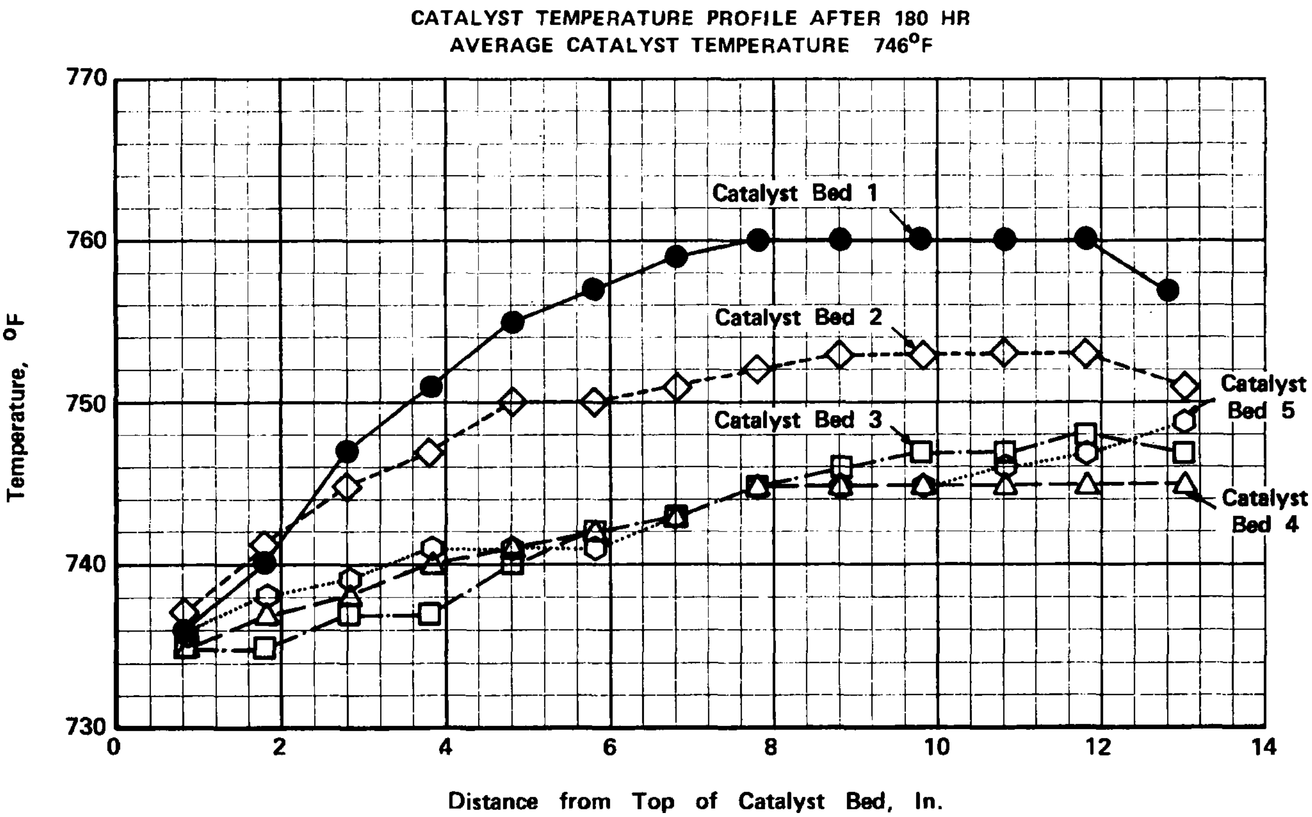


FIGURE 5

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL
WITH ICR 106 CATALYST
PILOT PLANT RUN 87-67

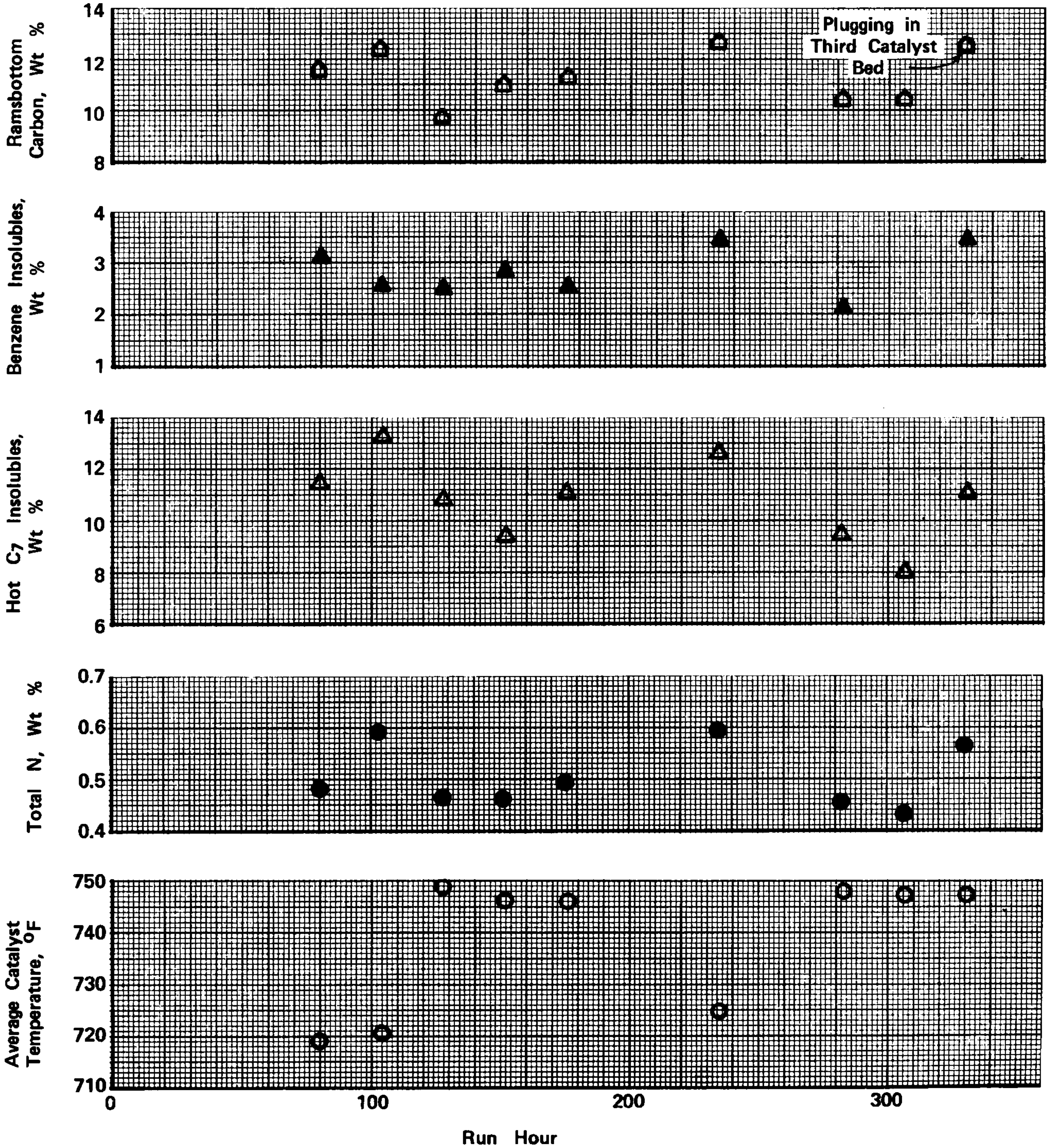


FIGURE 6

DOE CONTRACT EF-76-C-01-2315
HYDROTREATING OF 50/50 SRC-I/CREOSOTE OIL
WITH ICR 106 CATALYST
ASTM D 1160 (2 mm) DISTILLATIONS OF
FEED AND PRODUCTS OF RUN 87-67

	Run Hr	Avg. LHSV	Avg. Catalyst Temp., °F
Products	○ 103	0.18	721
	▲ 151	0.20	746
	⬡ 234	0.22	725
	△ 306	0.20	747

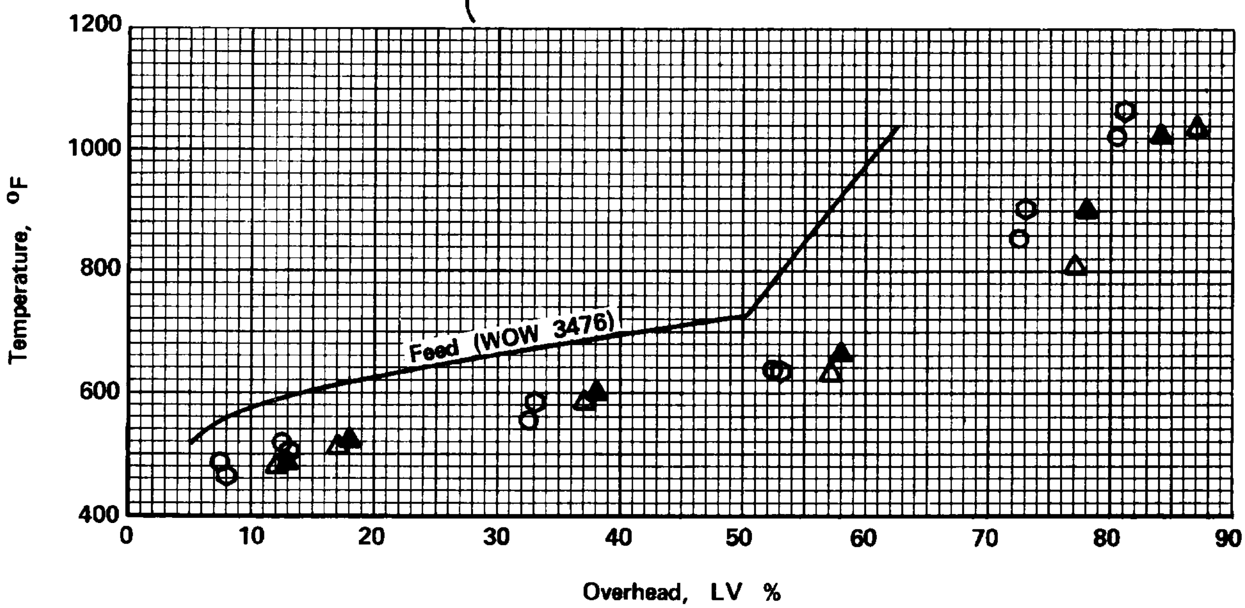
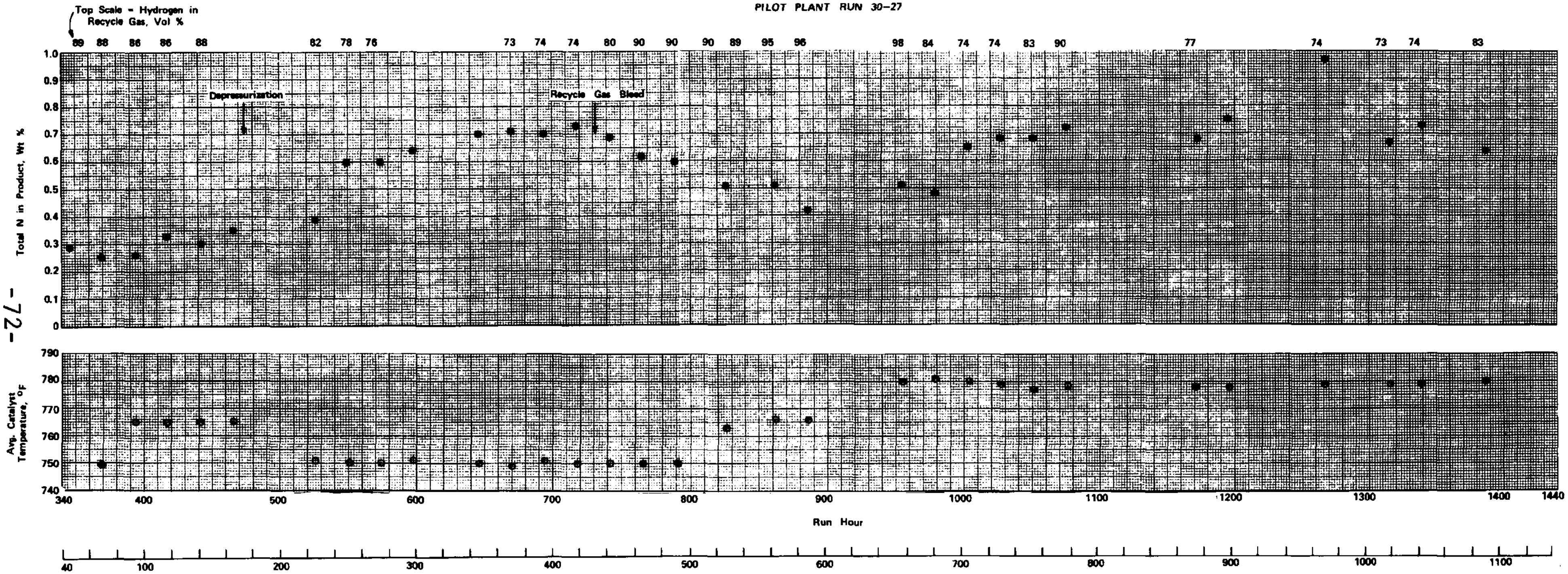


FIGURE 7

DOE CONTRACT EF-76-C-01-2315
HYDROPROCESSING 50/50 SRC-I/CREOSOTE OIL AT
0.2 LHSV, 2500 PSIG, 10,000 SCF/BBL WITH ICR 106
PILOT PLANT RUN 30-27



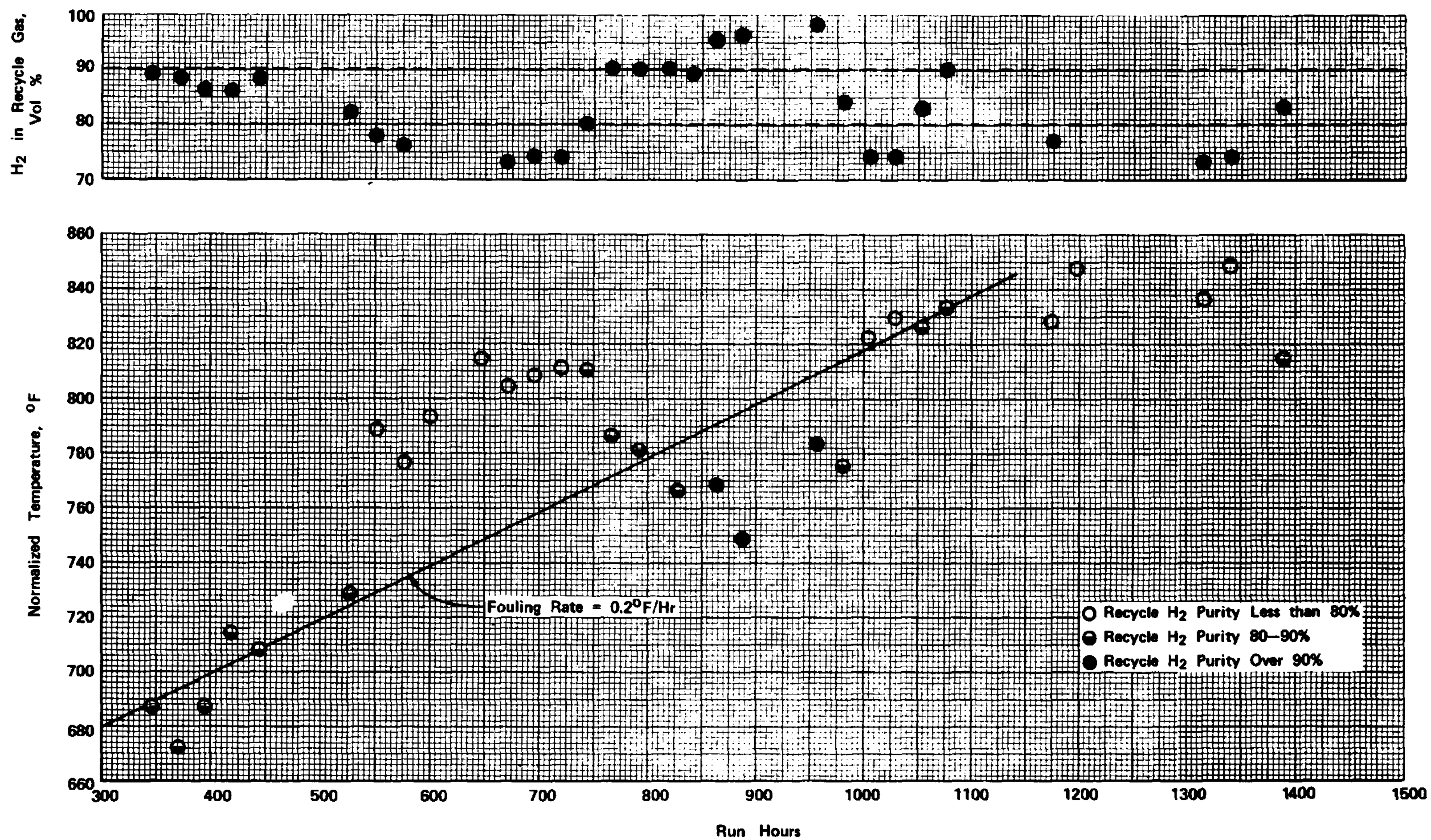
10-12-77

HCC RL 773930-1

FIGURE 8

DOE CONTRACT EF-76-C-01-2315
HYDROPROCESSING 50/50 SRC-1/CREOSOTE OIL AT
0.2 LHSV, 2500 PSIG, AND 10,000 SCF/BBL RECYCLE H_2 WITH ICR 106
RUN NO. 30-27

NORMALIZED TEMPERATURE TO 0.5% TOTAL N IN THE WHOLE LIQUID PRODUCT
(FIRST ORDER KINETICS, 18 KCAL/MOLE ACTIVATION ENERGY)



10-12-77 HCC RD 773928-1

FIGURE 9

HYDROTREATING OF 50/50 SRC–I CREOSOTE OIL
WITH ICR 106 CATALYST
DOE CONTRACT EF–76–C–01–2315
ASTM D 1160 (2 mm) DISTILLATIONS OF
FEED AND PRODUCTS OF RUN 30–27

Run Hour	Avg. LHSV	Average Catalyst Temperature, °F	H_2 in Recycle Gas, Vol %
○ 345	0.20	750	89
● 526	0.18	751	82
△ 718	0.20	750	74
◇ 790	0.19	750	90
◆ 979	0.20	781	84
▲ 1387	0.21	780	83

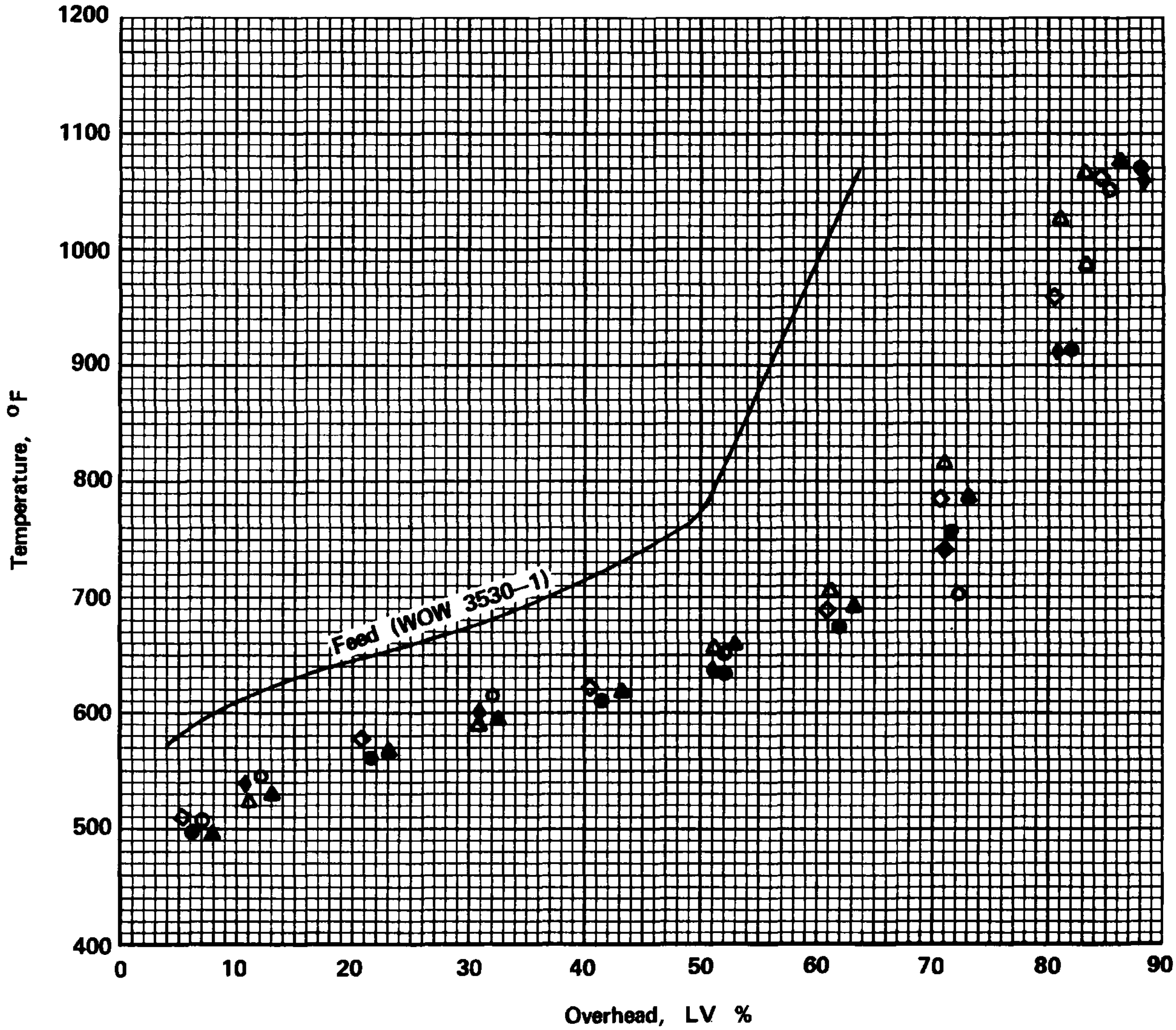


FIGURE 10

DOE CONTRACT EF-76-C-01-2315
DISTILLATION CURVES OF SRC-I, SIMULATED
RECYCLE SOLVENT, AND 50/50 SRC-I/SOLVENT

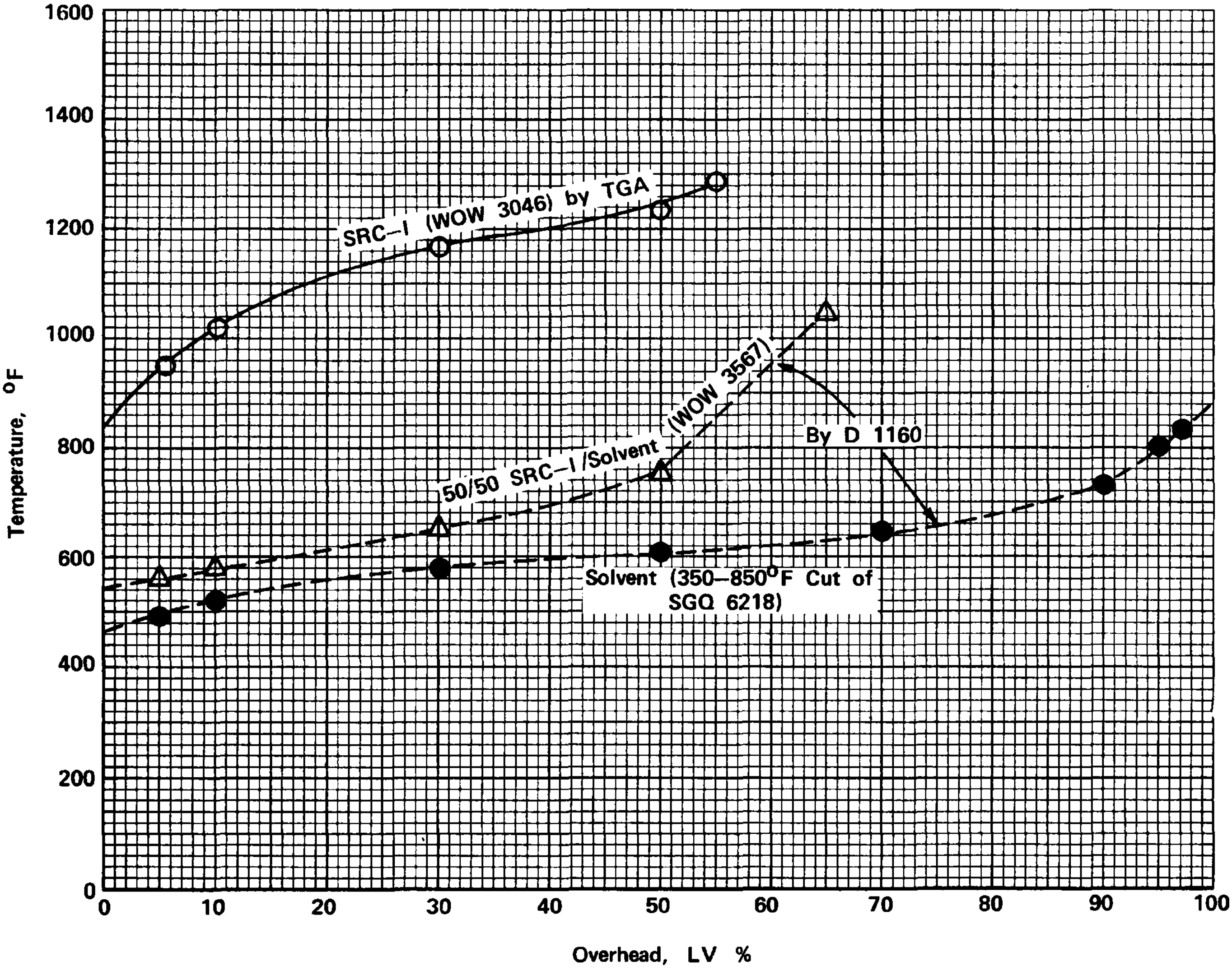
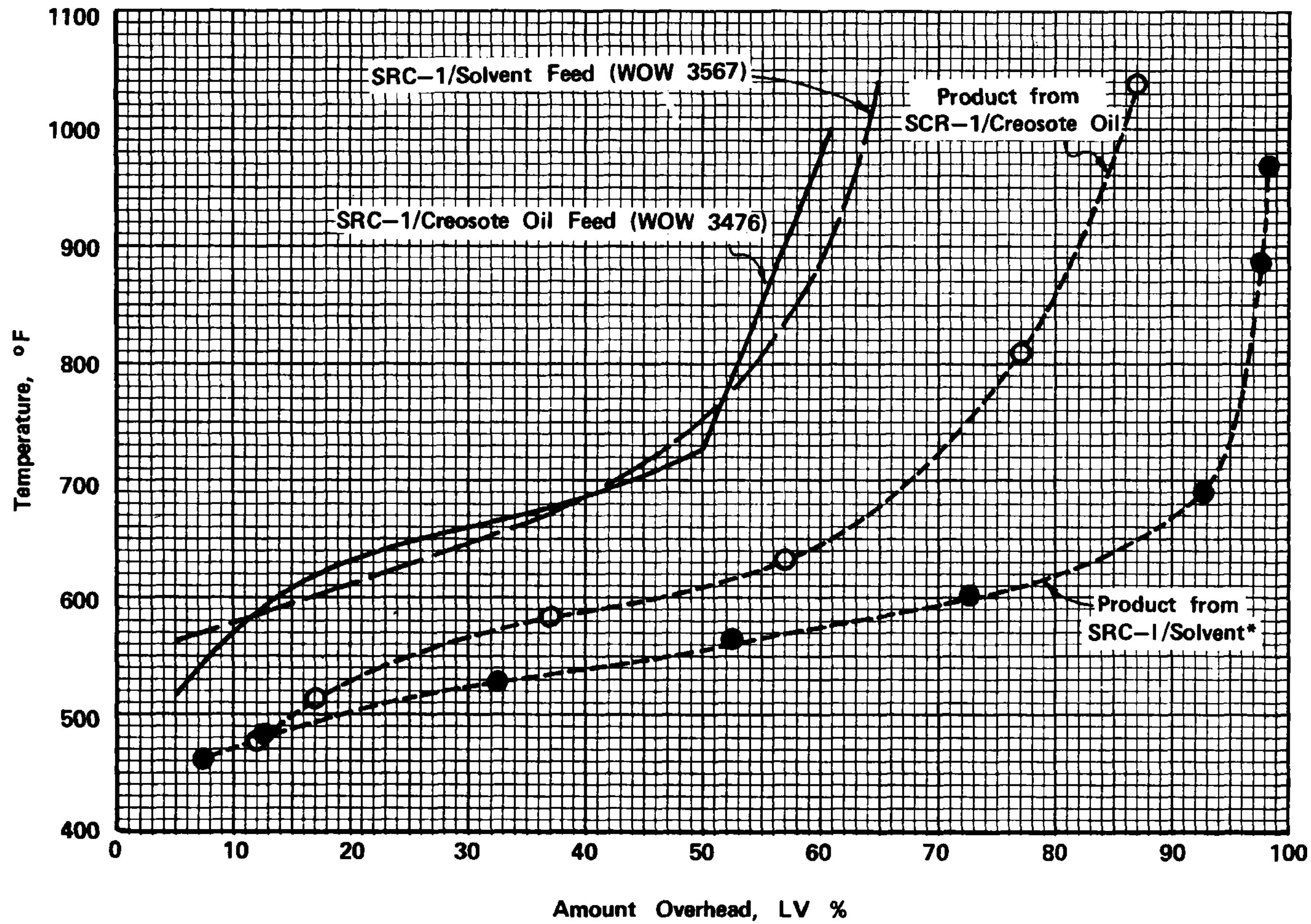


FIGURE 11

DOE CONTRACT EF–76–C–01–2315
COMPARISON OF CREOSOTE OIL AND SOLVENT IN HYDROCRACKING CONVERSIONS OF 50/50 BLENDS WITH SRC–I
ASTM D 1160 DISTILLATIONS OF FEEDS AND PRODUCTS

Run	Run Hr	Temp., °F	LHSV	Feed No.	Feed Description
87–67	294–306	750	0.19	WOW 3476	50/50 SRC/Creosote Oil
30–30	844–856	749	0.20	WOW 3567	50/50 SRC/Solvent



*Note: Short time onstream with SRC–I/Solvent Feed. Result may not be representative of lined out operation.

FIGURE 12

DOE CONTRACT EF-76-C-01-2315
HYDROPROCESSING THE 350-850°F PORTION OF THE
HYDROTREATED PRODUCT 50/50 SRC-I/CREOSOTE OIL

0.5 LHSV, 2500 PSIG TOTAL PRESSURE, 10,000 SCF/BBL WITH ICR 106
TOTAL N IN WHOLE LIQUID PRODUCT - RUN 66-188

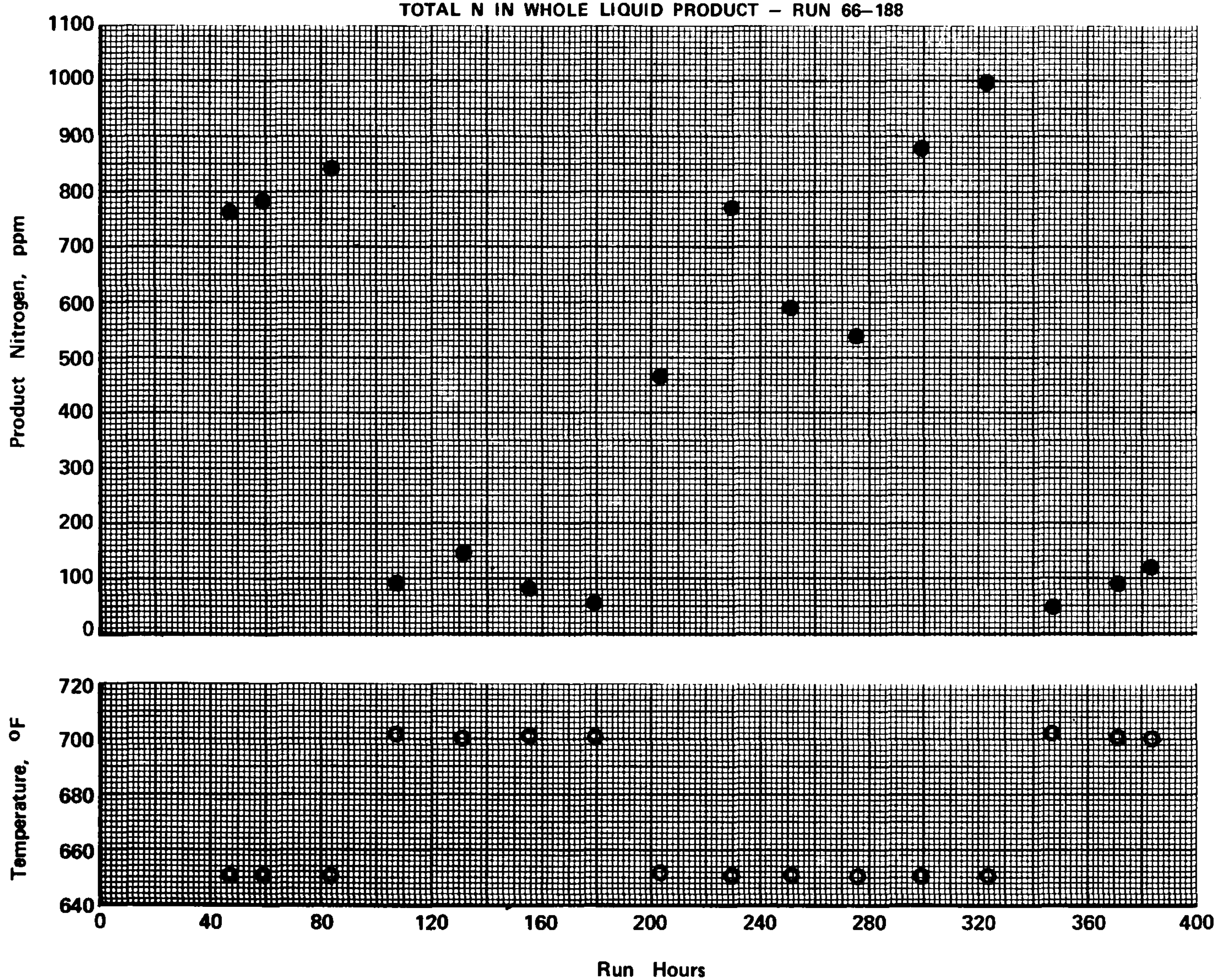
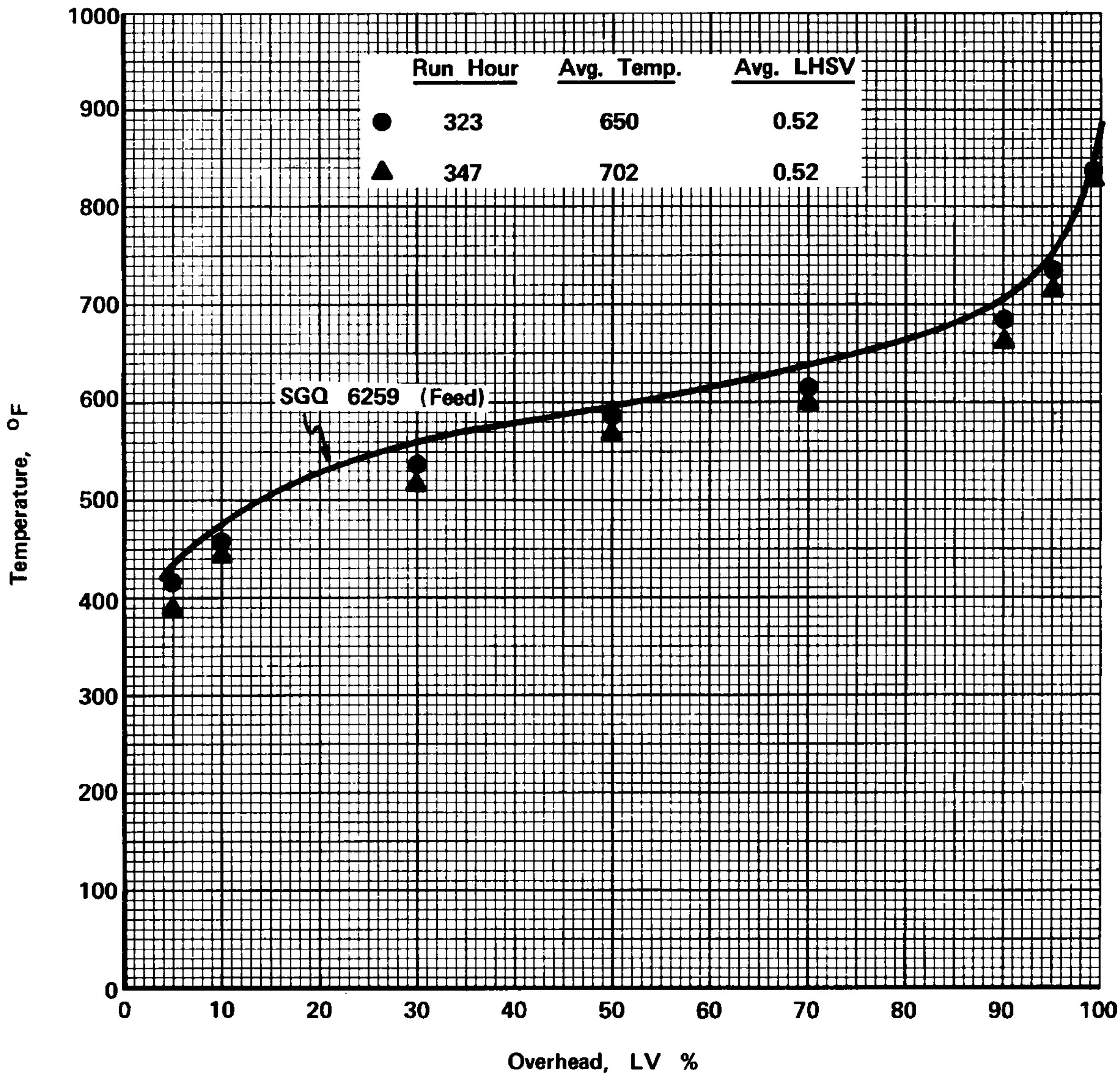


FIGURE 13

DOE CONTRACT EF–76–C–01–2315
HYDROPROCESSING THE 350–850°F PORTION OF THE
HYDROTREATED PRODUCT OF
50/50 SRC–I/CREOSOTE OIL WITH ICR 106 CATALYST

0.5 LHSV, 2,500 PSIG TOTAL PRESSURE, 10,000 SCF/BBL RECYCLE GAS RATE
SIMULATED DISTILLATION CURVES OF FEED AND PRODUCT
RUN 66–188



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