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EXPANDED BED HYDROPROCESSING OF SOLVENT REFINED COAL (SRC) EXTRACT

Interim
Technical Progress Report
on
Subtask II - Recycle Operation

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ABSTRACT

The feasibility of utilizing an expanded bed reactor (LC-Fining Process) for upgrading SRC-I in a recycle mode of operation has been demonstrated with respect to conversion of 850°F+ extract and denitrogenation. The objective of this subtask was to demonstrate that equilibrium recycle solvent could be produced without a concurrent accumulation of "refractory", recycled 850°F+ material.

The recycle run was made in a fashion that provided an exceptionally high conversion of fresh SRC-I material - ~87 weight percent. The demanding goal of producing a 0.3 weight percent nitrogen distillate (390-850°F) was readily achieved at this high conversion level. Equilibrium recycle solvent (500-850°F) was produced with a 9 weight percent hydrogen content after two consecutive recycle passes. No buildup of "refractory" material was discernible when recycling a 500°F+ process stream.

A calculated catalyst addition rate of one pound of nickel-molybdenum catalyst per ton of moisture free coal gave a fresh SRC-I conversion of 62 weight percent in the recycle mode of operation and 44.5 weight percent in the once-through mode of operation. This catalyst addition rate was effective in producing a superior quality product to that obtained in competing coal hydroliquefaction processes.

INTERIM

TECHNICAL PROGRESS REPORT

ON

SUBTASK II - RECYCLE OPERATION

Introduction

Several years ago discussions were initiated between Cities Service Company and the Energy Research and Development Administration (ERDA), now a part of the Department of Energy, which resulted in a government sponsored development contract. The original contract with ERDA involved process development unit (PDU) operation to determine the feasibility of operating an LC-Fining expanded bed hydroprocessing unit (LCF) to process solvent refined coal (SRC-I), and also to determine the optimum operating conditions for conversion and desulfurization. C-E Lummus, with laboratory process development units for LC-Fining located in New Brunswick, New Jersey, was chosen as the subcontractor for this project with Cities Service being the prime contractor. The choice of subcontractor was especially judicious as C-E Lummus is the exclusive worldwide licensor of the proprietary LC-Fining process and has an excellent perspective of the operating parameters.

The success of the original contract in determining the feasibility of utilizing an LC-Finer for upgrading SRC-I stimulated further interest in this method of operation by DOE. A greater emphasis was placed on nitrogen removal since good conversion and desulfurization had been adequately demonstrated. Consequently, a contract extension was granted.

Special effort was placed on the use of commercially available catalysts. It should be emphasized that the success achieved in this contract was obtained by use of a commercial petroleum refining process (LC-Fining) and commercial off-the-shelf- catalysts.

Conclusions Derived from Prior Studies

Two DOE Interim Technical Progress Reports (FE-2038-17[1] and FE-2038-25[2]) have been published and are available for distribution from NTIS, which describe, in detail, the results of the initial studies involved in expanded bed hydroprocessing of SRC-I.

This previously reported work is categorically subdivided into the following areas.

a) Preliminary 10-day operating periods to investigate the effect of solvent type and feasibility of operation;
b) First 30-day Aging Study (Co/Mo catalyst);
c) Catalyst screening;
d) 33-day operation - once-through mode, non recycle (Ni/Mo catalyst).

From these data it was concluded that:

a) Expanded bed LC-Finer processing for upgrading SRC-I is feasible at a conversion of 60+ weight percent 850°F+ and a denitrogenation of 70+ percent in the 850°F+ fraction (60+ percent in the TLP);
b) Trends have been established which provide information for optimization;
c) Proprietary LC-Fining correlations for residuum processing of petroleum are applicable to coal liquids processing;

d) Stable operation has been achieved in once-through operation for up to 33 days;

e) A distillate product (390-850°F) was made containing 0.3 weight percent nitrogen.

Objective (Subtask II)

The objective of this subtask was to determine the effectiveness of the LC-Fining Process, when operating in the recycle mode, for improved denitrogenation of SRC-I extract using a process derived recycle solvent (500-850°F) and without an accumulation of unreactive 850°F+ material. Prior studies were always conducted with the SRC-I/solvent feed blend containing fresh prehydrogenated Koppers heavy residue creosote oil (KC-Oil) as a solvent. Consequently, the liquid products contained a large percentage of hydrotreated KC-Oil as well as product from the hydroprocessing of the SRC-I. Operations with a recycle solvent would eliminate this anomaly and the liquid product would be derived entirely from the SRC-I feed.

Equipment and Unit Operation

The equipment used and the details of the PDU operation may be found in the Interim Technical Progress Report (FE-2038-17)[1].

Distillation facilities at C-E Lummus were not available for separating an 850°F- and an 850°F+ fraction in on-line operation. Hence, a operating scheme was devised which simulated one specific recycle mode - namely, total recycle of 500°F+ material which contained the unconverted 850°F+ fraction. Details of this mode of operation will be found in the section entitled "Charge Stock Preparation".

Several terms used in this report should now be defined:

a) Heavy oil basis - The PDU has three product streams, namely: Heavy Oil (H.O.), Light Oil (L.O.), and Product Gas. Rapid and representative heavy oil analyses for conversion of 850°F+ product fraction and total liquid product denitrogenation can be made on a daily basis in order to monitor unit operations.

b) Feed Basis - A more detailed analysis of the PDU product is made by combining the H.O. and L.O. together with the product gas and calculating fractional product yields and a conversion of 850°F+ based upon the feed charge. Heteroatom contents, viscosities, pour points, PONA analyses are run on the combined H.O. and L.O. product fractions.

c) Once-through Mode of Operation - This term defines PDU operation wherein only fresh SRC-I and prehydrogenated KC-Oil constitute the feed blend.

d) Recycle Mode of Operation - This term defines PDU operation wherein fresh SRC-I is added to a recycle fraction consisting of 500°F+ product. The amount of fresh SRC-I is adjusted to provide the desired SRC-I/solvent ratio in the recycle feed blend.

Charge Stock Preparation

Table I presents the LC-Fining PDU feed components and once-through feed blend for PDU Run 2LCF-3. This feed blend was used for the once-through portions of the recycle run, both during periods 1-11 and Periods 38-41. A period is defined as a 24-hour stream day.

The recycle operation was performed in the following manner. Once-through processing of fresh SRC-I/KC-Oil was undertaken for 11 periods to prepare sufficient internal recycle solvent for recycle Pass I. Distillation limitations made it necessary to achieve the solvent preparation in several batches for each pass - for example, Run Pass I: Periods 4B, 6B, 7; Periods 8, 9; and Periods 10, 11A, 11B. Each selected batch was topped to 500°F, the 500°F+ material was analyzed for the 850°F+ fraction, and sufficient fresh SRC-I was added to the 500°F+ material to make a feed blend consisting of 50/50 fresh plus unconverted 850°F+/internally generated solvent. This procedure was repeated with the product from any one recycle pass becoming the feed for the next recycle pass.

Table II presents the fractional distillation of the feed and product blends for the initial 11 periods of once-through operation and for the five recycle passes. The columns labeled "product" in Table II are the average fractional distillation values of the specific individual product blend batches found in Table III, and were used to determine the "feed" composition of the subsequent pass.

RESULTS AND DISCUSSION

Heavy Oil Analysis - Conversion and Denitrogenation

The initial data comparisons were made upon the basis of the analysis of the heavy oil fraction of the total liquid product and also upon the nitrogen content of this material. It was thus possible to obtain daily checks on the changes in the conversion of the SRC-I (850°F+) and also on the catalyst activity for denitrogenation in the heavy oil. This technique assumed that the heavy oil fraction was representative of the total liquid product since the heavy oil accounted for approximately 90% by volume of the total liquid product based on feed.

Subsequently, specific periods within each run were chosen for a complete product analysis (gas, light oil, heavy oil) and yield data was calculated on the basis of the total feed blend (Reference: Tables I-VIII in appendix A).

The denitrogenation is a function of the analytical levels of nitrogen in the feed and liquid products, and also of the absolute amounts of liquid product and fractions (feed basis). The percent denitrogenation is calculated using these two factors.

Conversion is defined as the volume percent of 850°F+ material in the liquid feed blend converted to products boiling below 850°F. The conversions reported on a daily basis are calculated on the basis of heavy oil only.

A summary of average reactor temperatures, nitrogen, and hydrogen contents of the heavy oil, gravity rise, and conversion (based upon the 850°F+ in the heavy oil) is presented in Table IV for PDU Run 2LCF-3. Table V summarizes the light oil gravity and the heavy oil product fraction yields for this run.

Figure 1 shows the conversion of 850°F+ (based upon heavy oil) for the 41 days of recycle PDU Run 2LCF-3 compared with PDU Run LCF-26 (30 days on American Cyanamid 1442B cobalt molybdenum catalyst) and Run LCF-36 (33 days on Shell 324 nickel molybdenum catalyst). Complete data for PDU Run LCF-26 and PDU Run LCF-36 will be found in Interim Reports FE-2038-17 and FE-2038-25, respectively[1,2]. The upper curve shows the behavior of the conversion as the temperature was increased and represents the actual unit data. It should be noted that after 11 periods of operation, PDU Runs LCF-36 and 2LCF-3 were essentially equivalent. However, during the recycle operation the conversion for PDU Run 2LCF-3 declined from approximately 56 weight percent at the beginning of recycle Pass I to approximately 42 weight percent at the end of recycle Pass V. During the recycle operation, the entire amount of unconverted 850°F+ plus make-up fresh SRC was processed in succeeding recycle passes. No drag stream was provided to withdraw any unconverted 850°F+, say for fuel usage, nor was any attempt made to maintain catalyst activity except through mild temperature increases.

After the fifth recycle pass, a catalyst activity check was made using fresh SRC-I plus KC-Oil solvent. Period 39 shows that at 810°F on a once-through basis, the conversion of 850°F+ is what would have been expected if the 41-day operation was entirely on a once-through basis. The actual conversion at Period 39 is on a line extrapolated from the termination of Run LCF-36. Period 41 shows that at 780°F on a once-through basis, the conversion of 850°F+ lies exactly where one would predict the conversion to be after 41 days operation at a constant temperature of 780°F. This data shows that even though the recycle conversion of 850°F+ was declining, there was no inherent damage to the catalyst. A catalyst activity check at both 780 and 810°F showed a predictable loss in activity based on once-through operation at these temperatures.

The lower curve shows the same conversion data normalized to a constant temperature of 780°F using proprietary LC-Fining correlations. The decline in conversion during the recycle passes is clearly shown as is the excellent reproducibility of the extrapolated line for PDU Run LCF-36 during the catalyst activity check for PDU Run 2LCF-3 (Periods 39 and 41).

Figure 2 shows the nitrogen content of the heavy oil product for PDU Run 2LCF-3 plotted similarly to the conversion data in Figure 1. The nitrogen level in the heavy oil product for PDU Run 2LCF-3 was very stable through Period 30. Following Period 30 there appeared to be a decline in denitrogenation activity through Period 38 (the end of recycle operation). It should be noted that there was no change in denitrogenation between the initial once-through operation and the recycle operation. Even after 34 periods of operation, the denitrogenation activity was still equivalent to once-through operation for PDU Run LCF-36. The lower curve in Figure 2 shows the actual nitrogen values at their respective run temperatures normalized to a constant temperature of 780°F.

Total Liquid Product (TLP) Analysis - Conversion and Denitrogenation

A final compilation of yield data and elemental fraction analyses for PDU Run 2LCF-3 is presented in Tables VI and VII. Table VI presents the total product distribution as a function of the feed blend charge. The component yields are determined from a combination of the three product streams - gas, light oil, and heavy oil. The weight percent values reported for ammonia and hydrogen sulfide, are determined by difference between the nitrogen and sulfur values in the feed blend and product fraction streams. The weight percent water is a measured quantity. The product fraction analyses for nitrogen and oxygen are internally checked against the comparable analysis for the total liquid product.

Table VII presents the elemental analyses for hydrogen, oxygen, nitrogen, and sulfur for the selected blend periods of the recycle Run, PDU 2LCF-3.

Figure 3 shows the conversion of 850°F+ (based upon feed and normalized to 780°F) for the 41 days of recycle PDU Run 2LCF-3 compared with PDU Run LCF-26 (30 days on American Cynamid 1442B cobalt molybdenum catalyst) and PDU Run LCF-36 (33 days on Shell 324 nickel molybdenum catalyst). The solid straight lines represent a linear regression of the data based upon feed for PDU Run LCF-36 and the recycle portion of PDU Run 2LCF-3. The dashed curved line is the data previously reported for PDU Run LCF-36, based upon heavy oil analyses only. The striking similarity of the data for PDU Run LCF-36 based upon weight percent of heavy oil only and that for weight percent of feed shows that the rapid, preliminary data interpretations based upon a heavy oil product analysis are valid and accurate. The data interpretation for PDU Run 2LCF-3 on a weight percent feed basis is the same as previously reported on a heavy oil only basis.

Figure 4 shows the nitrogen content of the total liquid product for the same three PDU runs - LCF-26, LCF-36, 2LCF-3 - plotted similarly to the conversion data in Figure 3. The nitrogen data shows that there is a decline in denitrogenation activity throughout the entire run duration for PDU Runs LCF-36 and 2LCF-3. Once again, there is a very strong similarity between the data obtained from the heavy oil analysis only and that from the total liquid product.

Nitrogen Content of Distillate Fraction (390-850°F)

One of the DOE constraints which was placed upon the product quality from LC-Fining hydroprocessing of coal extract was the achievement of a 0.3 weight percent nitrogen distillate product

(390-850°F). Table VIII shows the nitrogen content of the distillate product (390-850°F) for PDU Run 2LCF-3. During the recycle operation, a 0.3 weight percent nitrogen distillate product was produced and maintained. In the recycle mode, this product was entirely coal derived. The data obtained from once-through operation (Periods 11, 39, 41) show an increase in the nitrogen content of the distillate product over 41 days of operation (0.25 to 0.36 weight percent nitrogen), indicating a reduction in the denitrogenation function of the catalyst.

Figure 5 shows the percentage denitrogenation as a function of operating days for PDU Run LCF-36 (once-through operation in its entirety), and PDU Run 2LCF-3 (once-through followed by recycle followed by once-through operation). At period 11, both PDU Runs LCF-36 and 2LCF-3 are essentially equivalent. However, after 32 periods of operation, PDU Run LCF-36 had experienced a greater loss in denitrogenation than PDU Run 2LCF-3, evaluated on a once-through basis. It should be noted that the rate of change in denitrogenation activity for the recycle mode of PDU Run 2LCF-3 was less than the once-through mode for both PDU Runs LCF-36 and 2LCF-3. This lower rate of change in the denitrogenation function for the recycle mode of PDU Run 2LCF-3 is reflected in the denitrogenation activity at Periods 39 and 41 when once-through operation was resumed.

Product Distribution (Feed Basis)

The recycle PDU Run 2LCF-3, was made at essentially the same operating conditions as PDU Run LCF-36. This method of operation provided a comparable basis of thermal/catalytic activity for the two runs. Thus, differences between the two runs could be assigned to recycle versus once-through modes of operation. The temperature schedule for the runs is shown in Figure 6.

Both runs had catalyst activity checks made as the last set of operating conditons. In both cases, the final operating conditions in the once-through mode of operation were equivalent to the operating conditions in the once-through mode at the beginning of the run. For PDU Run LCF-36, the designated check blend periods were 11 and 32. For PDU Run 2LCF-3, the designated check blend periods were 11 and 41. The catalyst activity relationship at the beginning and end of a run was applied as a proration to the intermediate data in an attempt to remove the catalyst deactivation factor. Thus the intermediate data would then reflect the data only as a function of temperature.

However, several assumptions are inherent in this interpretation of the data. First, it is assumed that the change in the observed effect as a function of time is linear with respect to time. Thus a linear delta-effect per period of time could be established and intermediate data could be adjusted to a "fresh" activity corresponding to that observed at Period 11 and at any desired temperature. Second, it is assumed that the intermediate process parameter variations had no adverse effect on the catalyst deactivation function. In other words, operation at constant temperature for a given interval of time would produce the same catalyst deactivation as varying temperatures (within limits) over the same interval of time. Third, it is assumed that the catalyst deactivation function, observed in a once-through mode of operation, was also applicable in the recycle mode. Figures 7 through 10 present data which has been adjusted for the catalyst deactivation function just described.

Figure 7 presents a plot of the 850°F+ conversion versus the 500°F+ conversion, based on feed. At any constant weight percent conversion of 850°F+, there is no appreciable difference between once-through and recycle modes of operation. The effect of temperature on 500°F+ and 850°F+ conversion is clearly noted in PDU Run LCF-36 by noting that points 1 and 2 are at 780°F, points 3 and 4 at 790°F, and points 5, 6, and 7 at 800°F. A similar, although not as clearly defined, effect is noted for PDU Run 2LCF-3 where recycle operation at 790°F is shown by points 2 and 3, recycle operation at 800°F by points 4 and 5, and recycle operation at 810°F by point 6.

It should also be noted in Figure 7 that point 2 of PDU Run LCF-36 and point 1 of PDU Run 2LCF-3 represent the reference operation at Period 11 (780°F). These two points are never exactly equivalent in any of the product distribution plots. It will be noted in Figure 6 that PDU Run LCF-36 was brought to 780°F after only one period at 750°F. However, PDU Run 2LCF-3 ran three periods at 750°F before the temperature was raised to 780°F. Operability experience in the PDU operation has shown that a longer time at 750°F is more desirable. It is felt that the differences noted between the two runs at Period 11 is a reflection of the initial start-up procedure.

Figure 8 presents a plot of the 850°F+ conversion versus the 650°F+ conversion, based on feed. In contrast to the plot of the 500°F+ conversion, at a given 850°F+ conversion, there is a measurable difference in the 650°F+ conversion between the once-through and recycle runs. Figures 7 and 8, taken together, show that at any given 850°F+ conversion, once-through operation (PDU Run LCF-36) results in a greater production of the 500-650°F fraction, whereas the recycle operation (PDU Run 2LCF-3) results in a greater production of the 650-850°F fraction. For example:

Fraction	Assumed Feed Blend	Once-through LCF-36	Recycle 2LCF-3
500-650°F	28	32.5	29.4
650-850°F	22	23.2	26.3
850°F+	50	20	20
	100	75.7	75.7

$$850^\circ F+ \text{ Conversion} = \frac{50-20}{50} = 60\%$$

$$500^\circ F+ \text{ Conversion} = \frac{100-75.7}{100} = 24.3\%$$

$$650^\circ F+ \text{ Conversion} = \frac{72-43.2}{72} = 40\%$$

$$650^\circ F+ \text{ Conversion (2LCF-3)} = \frac{72-46.3}{72} = 35.7\%$$

Figure 9 presents a plot of the 390-500°F yield as a function of the 850°F+ conversion, based on feed. It is difficult from this plot to positively ascertain a significant difference between the two runs. The temperature effect previously described is noted for PDU Run LCF-36, but is lacking for recycle Run 2LCF-3.

Figure 10 presents a semi-logarithmic plot of the C_1-C_4 yield as a function of the 850°F+ conversion, based on feed. It should be noted that the C_1-C_4 yield is uniformly low in both the once-through and recycle modes of operation. In this plot, the C_1-C_4 yield for the once-through run (PDU LCF-36) plots uniformly as before. However, the recycle data for PDU Run 2LCF-3 show temperature deviations not heretofore observed when regressing the data through a (0,0) anchor point. The response of the C_1-C_4 yield toward 850°F+ conversion, with temperature as a parameter, is not the same for the recycle and once-through operation.

Equilibrium Recycle Solvent

PDU operation in the recycle mode for PDU Run 2LCF-3 required the recycle of a 500°F+ fraction which contained the 500-850°F solvent material. It will be recalled that the first 11 periods of operation used a prehydrogenated KC-Oil. The hydrogen content of the KC-Oil was 6.70 weight percent. The table below shows the hydrogen content of the 500-850°F fraction of the respective liquid products.

Pass	°F	Hydrogen (Wt.%) 500-850°F
Once-through	780	8.60
Recycle I	790	8.89
Recycle II	790	9.07
Recycle III	800	8.99
Recycle IV	800	9.07
Recycle V	810	8.50
Once-through	810	7.95
Once-through	780	8.35

The 500-850°F fraction of the liquid product from the initial once-through operation provided the recycle solvent for recycle Pass I with the product from recycle Pass I providing the solvent for recycle Pass II and so on. It is shown that the 500-850°F solvent had rapidly equilibrated with respect to hydrogen content after only two passes through the process development unit.

The recycle operation for PDU Run 2LCF-3 required the recycle of a 500°F+ material due to distillation limitations for separation of 850°F+ and 850°F- fractions. One of the potential problems arising from the operation of recycling unconverted 850°F+ would be the accumulation of "refractory" or non-convertible 850°F+ material. Within the period of recycle operation for PDU Run 2LCF-3, there was no apparent build-up of unconverted 850°F+ in successive recycle passes once an equilibrium conversion was established. This is shown in Table II wherein the volume percent of recycle 850°F+ in the feed blends was 15.2, 16.6, 17.8, 16.8, and 17.9, respectively, for recycle Passes I through V.

Hydrogen Consumption and Yields for Two-Step Liquefaction

In one-step liquefaction processes, the process conditions are compromised at high temperature, high pressure, and long residence times. These processes produce more naphtha and C_1-C_4 gases. Also, regeneration of the hydrogen donor solvent is accomplished at higher temperatures than in two-step liquefaction. Thermodynamics favor aromatics over hydroaromatics at higher temperatures which makes the hydrogenation of the donor solvent more difficult. Higher temperatures also favor catalyst deactivation which increases the catalyst consumption rate. High activity catalysts such as NiMoly are severely deactivated at 850°F.

A two-step liquefaction process enables the liquefaction, or coal dissolution process, to be conducted at its optimum operating conditions followed by coal extract upgrading at a different set of operating conditions. Although interdependent in two-step processing, the full advantage of the desired coal liquefaction can be maximized independently from the catalytic hydroprocessing and vice versa.

The data used for the coal liquefaction step came from published sources. The SRC-I liquefaction product yields and hydrogen consumption were taken from Moschitto's paper[3], and the LC-Finer data from our monthly progress reports. Both sets of data were standardized to the basis of one ton of moisture-free (MF) coal entering the liquefaction plant. This results in 960 pounds of SRC-I feed to the LC-Finer.

The LC-Finer recycle data was taken from PDU Run 2LCF-3 for recycle Passes II, III, IV, and V. Liquid feed and product yields were those reported for the period blends covering these passes (Table III). The gaseous yields were taken from the complete single period analyses which were reported for the corresponding recycle pass numbers (Table VI). The blended periods are more representative of the actual feeds and products than single periods. However, for the gas, data only existed for one single period within each blend.

The feed for each pass was placed on a normalized weight basis and then ratioed such that the amount of fresh SRC-I in the feed was 960 pounds. This gave a total weight of feed to the LC-Finer (recycle plus fresh SRC-I) per ton of MF coal. The weight percent (on feed) hydrogen consumption for each pass was then applied to the LC-Finer feeds to arrive at a value in pounds of hydrogen per ton of MF coal. This value plus the pounds of hydrogen consumed in the liquefaction step gave the total hydrogen consumption per ton of MF coal. The yields were a summation of the gas and liquid products from the liquefaction plant plus all the products from the LC-Finer minus the material which was recycled.

Table IX shows the combined products for the two-step liquefaction on a weight percent MF coal basis for PDU Run 2LCF-3, recycle Passes II-V. Also listed are the barrels of liquid products per ton of MF coal. Conversion of SRC-I to 850°F- material is also shown. This conversion is based on the fresh SRC-I entering the reactor and net non-recycled 850°F+ material leaving the system. It should be noted that the high conversion, about 87 weight percent, is higher than would be considered optimal for good hydrogen economy and on-stream catalyst replacement.

Since the total hydrogen consumption and total C_5-850°F distillate yields do not vary widely from recycle Passes II-V, an average of these values was used to obtain a 5.64 weight percent hydrogen consumption and a 2.83 Bbl/ton distillate yield. Table X shows the breakdown of the products from the liquefaction step and the averaged LC-Fining step. Figure 11 is a simplified material balance schematic of the two-step liquefaction operation.

Two points should be mentioned in examining the yields presented in Table X. In the recycle operation, the initial boiling point of the recycle product prior to the addition of fresh SRC was 500°F. This procedure tended to artificially increase the make of 500°F- product. Therefore, a lighter product was produced than could be expected if a heavier boiling range solvent had been used.

It is also shown in Table X that there is a net production of 850°F+ material. The yield structure was developed using the existing PDU data from Run 2LCF-3 and liquefaction data from the Fort Lewis liquefaction plant. The high quality net 850°F+ from Two-Step Liquefaction could be used as a blending component with lower quality 850°F+ material from thermal liquefaction. Also, different LC-Fining processing conditions could be utilized for higher extinction of the 850°F+. A minimum quantity of 850°F+ should always remain as a vehicle to move the ash and IOM. Neither of these alternatives was considered in this report.

For comparison purposes, a once-through LC-Fining case was generated where no material was recycled to the reactor. Prehydrogenated foreign solvent was used for blending with the SRC-I. PDU Run 2LCF-3, Period 11, was used for this case. The solvent comprised 47 weight percent of the LC-Finer reactor feed, or 851.3 pounds per 960 pounds SRC-I. This is equivalent to 2.20 barrels solvent per ton of MF coal.

Hydrogen consumption for this case was 3.29 weight percent for the LC-Finer based on the total reactor feed (59.6 pounds). This combined with the liquefaction hydrogen consumption gave a combined two-step consumption of 5.38 weight percent.

Product yields for the once-through two-step operation based on MF coal are given in Table XI. The solvent was not split out of these yields because in actual operations the liquid product comes out as a mix containing the solvent. In calculating the

C_5-850°F liquid yield, however, it is possible to draw off a volume equal to the amount of solvent added to the system. This was done in arriving at the 2.44 Bbl/ton liquid yield reported in Table XI. The actual measured yield was 4.64 Bbl/ton, but, as mentioned previously, 2.20 Bbl/ton was added as solvent prior to LC-Fining leaving 2.44 Bbl/ton as the net yield per ton of coal. Conversion of SRC-I to 850°F-material is <u>60.7 weight percent</u>, once again a very high conversion of fresh SRC-I. This calculation assumes solvent-in = solvent-out.

The breakdown of the yields between the SRC-I plant and the LC-Finer are presented in Table XII on a pounds per ton of MF coal basis. The total yield of 2851.3 pounds contains the 851.3 pounds of foreign solvent. A material balance schematic diagram similar to the one for the recycle case is drawn in Figure 12.

It should be re-emphasized that the high hydrogen consumption figures reported above are a direct result of the unusually high conversion of fresh SRC-I feed. Proprietary commercial design considerations have indicated that a two-step hydrogen consumption of 4 weight percent is a realistic value.

Catalyst Addition Rate

One of the outstanding process features of the LC-Fining expanded bed reactor process is the ease of on-stream catalyst addition and withdrawal. In the LC-Fining process, the catalyst in the reactor is in constant motion, suspended by the continuous upward flow of oil and hydrogen. There is no pressure build-up since extraneous solids easily pass through the void space between particles. When the catalyst bed is in an expanded condition, it behaves somewhat like a liquid. Therefore, on-stream catalyst addition and withdrawal is a straightforward, relatively easy procedure.

Anderson and Matthias[4] have developed a mathematical analysis procedure to predict a steady-state activity for any catalyst replacement rate from laboratory experiments using similar equipment, but with no replacement of the catalyst.

Figures 13 and 14 present, respectively, the results of this mathematical approach for PDU Run 2LCF-3 (recycle operation) and PDU Run LCF-36 (once-through operation). At a catalyst replacement rate of <u>one pound of catalyst per ton of moisture free coal</u>, the following results are noted:

PDU Run 2LCF-3 (Recycle Mode)	62 Weight % Conversion of Fresh 850°F+ 0.62 Weight % Nitrogen in TLP 0.36 Weight % Nitrogen in Distillate
PDU Run LCF-36 (Once-Through Mode)	48.5 Weight % Conversion of Fresh 850°F+ 0.65 Weight % Nitrogen in TLP 0.41 Weight % Nitrogen in Distillate

CONCLUSIONS

In conclusion, it may be stated that:

1) Recycle processing of SRC-I coal extract produced an equilibrium recycle solvent containing 9 weight percent hydrogen after two recycle passes in the PDU;

2) No "refractory" 850°F+ material was detected when recycling 500°F+ material;

3) A 850°F+ conversion of approximately 87 weight percent (based on fresh SRC-I feed) was obtained during recycle processing;

4) A distillate product (390-850°F) containing <0.3 weight percent nitrogen was routinely obtained during recycle processing at the above noted high conversion;

5) In the recycle mode of processing coal extract, a catalyst addition rate of one pound of catalyst per ton of moisture free coal gave an 850°F+ conversion of 62 weight percent (based on fresh SRC-I feed) and a nitrogen content in the distillate fraction (390-850°F) of 0.36 weight percent. A similar correlation for once-through operation showed 48.5 weight percent 850°F+ conversion and 0.42 weight percent nitrogen in the distillate fraction.

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TABLE I

LC-FINING PDU OPERATION
ONCE-THROUGH FEED BLEND PROPERTIES
PDU RUN 2LCF-3 (d)
SRC-PREHYDROGENATED KOPPERS HEAVY RESIDUE CREOSOTE OIL

Component	Solvent N-043	SRC N-023	Blend
Composition			
Solvent, Wt% (ex drum)	-	-	47.0
Description, IBP °F	500	-	500
SRC, Wt% (ex drum)	-	-	53.0
850°F+, Vol% (by distilln)	-	-	50.7
850°F+, Wt% (by distilln)	-	-	52.3
Gravity, °API	-3.6	-16.7	-14.1
, SP 60/60°F	1.1062	1.2329	1.2048
Softening Point, °F	-5 (a)	346	118
Viscosity, Kin. CS @ 100°F	17.87	-	-
Kin. CS @ 210°F	2.79	-	-
Kin. CS @ 350°F	-	-	10.88
Kin. CS @ 400°F	-	-	5.95
Elemental Content, Wt%			
Carbon	92.17	86.67	89.37
Hydrogen	6.70	5.91	6.32
Oxygen	0.59	4.45	2.52
Nitrogen	0.41	2.04	1.23
Sulfur	0.09	<0.06	0.41
Ash, Wt%	Tr	0.14	0.11
Distillate Fractions			
IBP-500°F, Vol%	11.8		
Gravity, °API	9.1		
, SP 60/60°F	1.0063		
Carbon, Wt%	-		
Hydrogen, Wt%	-		
Nitrogen, Wt%	-		
Sulfur, Wt%	<0.06		
500-650°F, Vol%	49.8		30.0(c)
Gravity, °API	-1.7		2.9
, SP 60/60°F	1.090		1.0528
Carbon, Wt%	-		-
Hydrogen, Wt%	-		-
Nitrogen, Wt%	-		0.27
Sulfur, Wt%	0.08		0.06
650-850°F, Vol%	30.8		22.0
Gravity, °API	-8.7		-6.9
, SP 60/60 °F	1.1527		1.1356
Carbon, Wt%	-		-
Hydrogen, Wt%	-		-
Nitrogen, Wt%	-		0.64
Sulfur, Wt%	0.10		0.14
850°F+, Vol%	6.5(b)		50.7
Gravity, °API	-		-17.7
, SP 60/60 °F	-		1.2430
Carbon, Wt%	-		-
Hydrogen, Wt%	-		-
Nitrogen, Wt%	-		2.00
Sulfur, Wt%	0.07		0.67

(a) Pour Point
(b) Based on assumed gravity of residue
(c) IBP-650°F
(d) Feed blend used in Periods 1-11 and 38-41

TABLE II

LC-FINING PDU OPERATION
FRACTIONAL ANALYSIS OF RECYCLE PASS FEED BLENDS
PDU Run 2LCF-3

Run Pass No.	I		II		III		IV		V		VI	
Recycle Pass No.	-		I		II		III		IV		V	
	Feed	Product	Feed	Product	Feed	Product	Feed	Product	Feed	Product	Feed	Product(a)
Dist. Fraction (b)												
IBP-500°F												
Vol%	5.0	9.2		11.2		12.9		12.6		13.0		16.2
°API	-	24.7		23.8		22.7		22.8		21.9		21.9
500-650°F												
Vol%	25.0	39.0	28.1	36.2	27.1	36.7	28.4	35.1	26.9	34.0	26.5	28.5
°API	-	9.4	9.4	12.1	12.1	12.0	12.0	12.5	12.5	12.2	12.2	10.9
650-850°F												
Vol%	22.0	30.5	21.9	30.5	22.9	27.8	21.6	30.2	23.1	30.2	23.5	27.1
°API	-6.9	0.5	0.5	2.3	2.3	2.2	2.2	1.8	1.8	1.6	1.6	0.5
850°F+												
Fresh Vol%	50.7	21.1	34.8		33.4		32.2		33.2		32.1	
°API	-17.7	-13.9	-16.5		-16.5		-16.5		-16.5		-16.5	
Recycle Vol%	-		15.2	22.1	16.6	22.9	17.8	21.9	16.8	23.0	17.9	22.1
°API	-		-13.9	-14.6	-14.6	-15.1	-15.1	-17.3	-17.3	-17.6	-17.6	-19.7
Feed Blend Properties												
Specific Gravity			1.1279		1.1207		1.1212		1.1258		1.1281	
N_2, Wt% (c)			1.09		1.10		1.13		1.12		1.14	
H_2, Wt% (c)			7.30		7.28		7.24		7.15		7.11	

(a) Period 2LCF-3/37 only
(b) Calculations based on average values for pass blend periods of Table III
(c) Average values

TABLE III

LC-FINING PDU OPERATION
FRACTIONAL ANALYSIS OF PRODUCT BLENDS
PDU Run 2LCF-3

Blend Periods 2LCF-3	Run Pass No. (a)	Used in Recycle Pass No. (a)	Distillation of Run Pass Product Blend (L.O. + H.O.)(b) IBP-500°F Vol%	IBP-500°F °API	500-650°F Vol%	500-650°F °API	650-850°F Vol%	650-850°F °API	850°F+ Vol%	850°F+ °API
4B,6B,7	I	I	9.4	25.3	38.5	10.4	31.5	1.1	20.6	-13.9
8,9			8.05	24.9	37.7	9.2	31.4	0.8	22.4	-13.0
10,11A,11B			10.2	23.9	40.9	8.7	28.5	-0.3	20.2	-14.9
13,14	II	II	10.7	24.2	31.9	12.9	33.5	3.5	24.0	-13.3
15,16			11.6	23.9	39.1	11.7	18.4	1.8	21.1	-15.2
17,18			11.6	23.4	37.6	11.6	29.7	1.7	21.2	-15.2
20,21	III	III	14.5	22.5	35.4	12.1	27.0	2.6	23.4	-14.2
19,22,23			11.2	22.8	38.0	11.8	28.7	1.7	22.3	-16.0
24B,25	IV	IV	12.2	23.8	36.0	13.0	31.9	2.2	20.0	-17.0
26,27			12.7	22.2	35.1	12.3	29.5	1.8	22.9	-17.2
27,28			13.0	22.3	34.1	12.3	29.2	1.4	22.8	-17.8
29,30	V	V	12.9	22.7	36.8	12.0	27.8	1.3	23.0	-17.8
31B,32,33			13.2	21.1	31.3	12.4	32.6	1.8	22.9	-17.3

(a) Run Pass: I, II, III, IV, V, VI, VII
Recycle Pass: - I, II, III, IV, V, -

(b) L.O. - Light Oil
H.O. - Heavy Oil

TABLE IV

LC-FINING PDU OPERATION
HEAVY OIL PRODUCT ANALYSES

SRC-I/Prehydrogenated Koppers Heavy Residue Creosote Oil
and SRC-I/Recycle Solvent
PDU Run 2LCF-3 - Shell 324 Ni/Mo

2LCF-3 Period	Reactor Temp. °F	Nitrogen Content Wt%	Hydrogen Content Wt%	Wt. Ratio of 850°F+ Feed-Prod / Feed	Gravity Rise °API
PASS I - SRC-I/PREHYDROGENATED KC-OIL					
1B(a)	750	0.33	8.79	0.45	16.4
2	750	0.37	8.76		
3(b)	750	0.43	8.35	0.45	15.9
4B(a)	780	0.44	8.23		
6A(a)	780	0.44	8.31	0.49	
7	780	0.43	8.25	0.58	16.9
8	780	0.44	8.23		
9	780	0.45	8.16	0.52	16.0
10	780	0.47	8.18		
11a	780	0.48	8.17	0.55	16.4
PASS II - SRC-I/RECYCLE SOLVENT FROM PASS I					
12	780	0.47	8.18		
13	780	0.47	8.42		
14	790	0.48	8.37		
15	790	0.45	8.35	0.54	17.4
16	790	0.43	8.36		
17	790	0.41	8.27	0.53	17.3
18	790	0.45	8.24		
PASS III - SRC-I/RECYCLE SOLVENT FROM PASS II					
19	790	0.44	8.34	0.55	17.1
20	790	0.42	8.36		
21	790	0.44	8.23	0.53	
22	790	0.43	7.63		
23	790	0.44	8.20	0.43	15.5
PASS IV - SRC-I/RECYCLE SOLVENT FROM PASS III					
24B(a)	800	0.39	8.55	0.50	
25	800	0.36	8.55	0.57	17.9
26	800	0.43	8.31		
27	800	0.47	8.00	0.48	
28	800	0.47	8.22		
PASS V - SRC-I/RECYCLE SOLVENT FROM PASS IV					
29B	800	0.44	8.05	0.45	16.9
30	800	0.46	8.07		
31B(a)	800	0.50	8.01	0.47	16.2
32	800	0.53	7.95		
33	800	0.52	7.97	0.43	14.7
PASS VI - SRC-I/RECYCLE SOLVENT FROM PASS V					
34	810	0.54	7.73		
35	810	0.53	7.66	0.44	15.0
36	810	0.54	7.73		
37	810	0.57	7.63	0.42	14.8
PASS VII - SRC-I/PREHYDROGENATED KC-OIL					
38	810	0.47	7.57		
39	810	0.45	7.65	0.64	15.3
40A	780	0.65	7.86		
40B(a)	780	0.63	7.75	0.41	13.0
41	780	0.62	7.75	0.45	13.0

(a) Spot samples of heavy oil
(b) Shut down following Period 3 due to weather emergency.

TABLE V

LC-FINING PDU OPERATION
SUMMARY OF LIGHT AND HEAVY OIL RECOVERED

SRC-I/Prehydrogenated Koppers Heavy Residue Creosote Oil
and SRC-I/Recycle Solvent
PDU Run 2LCF-3 - Shell 324 Ni/Mo

Run 2LCF-3 Period	Light Oil °API	Heavy Oil Fraction: IBP-500°F Vol%	IBP-500°F °API	IBP-650°F Vol%	IBP-650°F °API	650-850°F Vol%	650-850°F °API	850°F+ Vol%	850°F+ °API	Total °API
PASS I - SRC-I/PREHYDROGENATED KC-OIL										
1B(a)	20.7			40.7	10.6	30.1	1.8	29.5	-10.1	0.8
3(b)	20.4			41.8	10.4	28.9	1.6	29.3	-11.4	0.6
6A(a)				37.6	11.0	35.0	1.4	26.9	-13.6	0.2
7	20.3			45.5	10.9	32.5	0.7	22.0	-13.7	1.4
9	20.2			43.9	10.5	30.5	0.5	25.2	-12.9	0.8
11A	20.2			44.0	10.0	32.3	0.6	23.4	-13.7	0.9
PASS II - SRC-I/RECYCLE SOLVENT FROM PASS I										
15	21.1			42.3	13.0	33.9	2.0	23.7	-14.8	1.6
17	20.1			43.9	13.9	29.3	2.3	24.6	-14.8	1.4
PASS III - SRC-I/RECYCLE SOLVENT FROM PASS II										
19	15.3			45.9	12.6	31.4	1.6	23.4	-15.6	1.4
21	19.8			41.4	13.2	34.6	1.7	24.0	-16.2	1.0
23	19.2			41.4	12.4	28.9	1.1	29.6	-15.9	-0.5
PASS IV - SRC-I/RECYCLE SOLVENT FROM PASS III										
24B(a)	-			39.8	13.4	35.0	1.9	25.8	-16.5	-0.4
25	13.5			45.1	13.1	33.1	1.4	21.8	-17.0	1.5
27	-			41.5	13.3	32.6	1.6	26.3	-16.9	-0.3
PASS V - SRC-I/RECYCLE SOLVENT FROM PASS IV										
29B	19.7			36.3	14.5	39.0	2.8	27.7	-17.7	0.5
31B(a)(c)	16.6	8.4	20.9	37.0	11.1	27.8	1.4	27.1	-16.0	0.4
33	19.3			38.5	12.9	33.0	1.8	29.2	-17.4	-1.7
PASS VI - SRC-I/RECYCLE SOLVENT FROM PASS V										
35	19.1			38.8	12.4	33.8	0.6	27.8	-18.9	-2.1
37	17.7			41.2	10.5	29.9	-0.3	29.0	-18.9	-2.4
PASS VII - SRC-I/PREHYDROGENATED KC-OIL										
39	17.2			44.6	9.4	37.2	-2.2	18.5	-17.5	-1.2
40B(a)	16.9			39.0	7.0	30.7	-1.3	30.6	-14.9	-3.1
41	16.0			41.4	6.9	30.4	-1.5	28.7	-14.3	-3.1

(a) Spot samples of heavy oil
(b) Shut down following Period 3 for weather emergency
(c) 500-650°F cut for this period

TABLE VI

LC-FINING PDU OPERATION
YIELD DATA AS A PERCENTAGE OF FEED
PDU Run 2LCF-3

Blend Period PDU 2LCF-3	11B	18	23	28	33	37	39	41
Feed Blend	Once-through	Recycle I	Recycle II	Recycle III	Recycle IV	Recycle V	Once-through	Once-through
Average Reactor Temp., °F	780	790	790	800	800	810	810	780
Space Velocity (X times SV_0)	0.42	0.39	0.47	0.39	0.42	0.42	0.42	0.42
Conversion, Vol% 850°F+	58.4	58.1	49.4	56.8	52.1	57.2	68.7	48.1
Gravity Rise, °API	16.4	-	15.5	-	14.7	14.8	15.3	13.0
Yield - Weight % of Feed								
H_2O	3.59	2.02	1.49	1.67	1.94	2.74	3.39	2.80
H_2S	0.44	0.36	0.33	0.37	0.32	0.36	0.41	0.40
NH_3	1.07	0.77	0.79	0.92	0.8	0.88	0.93	0.73
CH_4	1.22	1.49	1.20	1.66	1.73	1.81	1.99	1.09
C_2H_6	1.13	1.49	1.24	1.74	1.27	1.88	1.99	1.09
C_3H_8	1.18	1.76	1.52	2.11	1.72	2.35	2.17	1.06
C_3H_6	0.05	0.09	0.08	0.13	0.14	0.15	0.15	0.08
$i-C_4H_{10}$	0.1	0.15	0.14	0.19	0.12	0.22	0.17	0.09
$n-C_4H_{10}$	1.26	1.72	1.42	1.75	1.51	2.21	2.01	0.99
C_4H_8	0.01	0.02	0.02	0.04	0	0.05	0.02	0.01
C_5-390°F	6.38	7.07	3.95	5.73	7.02	7.95	7.56	5.04
390-500°F	9.77	10.66	8.85	9.61	10.35	9.23	13.56	9.62
500-650°F	29.22	25.77	26.48	23.76	23.08	24.14	27.59	26.31
650-850°F	27.06	26.93	27.82	29.01	25.99	24.72	25.18	27.20
850°F+	20.81	22.45	26.80	23.84	26.29	23.80	16.19	26.18
Total	103.29	102.75	102.13	102.53	102.28	102.49	103.31	102.69
Yield - Volume % of Feed								
C_5-390°F	10.20	10.73	5.72	8.58	10.55	11.49	11.43	7.27
390-500°F	12.44	12.90	10.86	11.64	12.43	11.20	17.02	12.08
500-650°F	34.42	29.32	30.23	27.39	26.23	27.68	32.24	30.75
650-850°F	30.25	28.91	29.92	30.75	27.63	26.29	27.67	30.09
850°F+	20.78	20.93	25.29	21.59	23.93	21.42	15.63	25.94
Total	108.09	102.79	102.02	99.95	100.77	98.08	103.99	106.13
H_2 Consumption (SCF/BBL)	2611	2074	1747	1895	1718	1923	2631	2133

TABLE VII

LC-FINING PDU OPERATION
ELEMENTAL ANALYSIS OF LIQUID PRODUCT

PDU Run 2LCF-3

Blend Period PDU LCF-3	11B	18	23	28	33	37	39	41
Feed Blend	Once-through	Recycle	Recycle II	Recycle III	Recycle IV	Recycle V	Once-through	Once-through
Average Reactor Temp., °F	780	790	790	800	800	810	810	780
Space Velocity (X times SV_0)	0.42	0.39	0.47	0.39	0.42	0.42	0.42	0.42
Conversion, Vol.% 850°F+	58.4	58.1	49.4	56.8	52.1	57.2	68.7	48.1
Elemental Analysis, Wt.%								
Hydrogen								
IBP-390°F	11.84	11.78	11.53	12.18	11.34	11.01	11.01	11.06
390-500°F	10.26	10.42	10.86	10.88	10.29	9.95	9.89	9.97
500-650°F	9.08	9.51	9.61	9.71	9.77	9.32	8.43	8.84
650-850°F	8.09	8.30	8.54	8.40	8.44	7.75	7.42	7.86
850°F+	6.69	6.31	6.34	5.79	6.03	5.36	5.60	6.33
Total Liquid Product	8.35	8.48	8.30	8.23	8.18	7.93	7.80	7.94
Oxygen								
IBP-390°F	0.90	0.64	0.86	0.93	1.34	1.15	1.29	1.92
390-500°F	0.24	0.22	0.26	0.28	0.31	0.37	0.50	0.63
500-650°F	0.41	0.26	0.25	0.22	0.25	0.27	0.43	0.53
650-850°F	0.28	0.24	0.29	0.24	0.25	0.27	0.33	0.45
850°F+	1.11	0.85	1.10	0.88	0.83	0.62	0.71	1.28
Total Liquid Product	0.43	0.45	0.49	0.41	0.47	0.49	0.51	0.77
Nitrogen								
IBP-390°F	0.10	0.05	0.07	0.16	0.11	0.22	0.24	0.36
390-500°F	0.03	0.11	0.21	0.22	0.28	0.19	0.22	0.25
500-650°F	0.13	-	0.15	0.23	0.08	0.20	0.20	0.24
650-850°F	0.45	0.30	0.39	0.47	0.31	0.44	0.51	0.52
850°F+	1.21	1.13	1.21	0.97	1.21	1.29	1.29	1.22
Total Liquid Product	0.39	0.45	0.50	0.44	0.50	0.49	0.53	0.68
Sulfur								
IBP-390°F	< 0.06	<0.06	<0.06	< 0.06	< 0.06	<0.06	<0.06	<0.06
390-500°F	<0.06	<0.06	<0.06	< 0.06	<0.06	<0.06	<0.06	<0.06
500-650°F	< 0.06	<0.06	<0.06	< 0.06	<0.06	<0.06	<0.06	<0.06
650-850°F	< 0.06	<0.06	<0.06	< 0.06	<0.06	<0.06	<0.06	<0.06
850°F+	< 0.06	0.13	0.15	0.13	0.15	<0.06	<0.06	0.17
Total Liquid Product	< 0.06	<0.06	0.04	< 0.06	0.04	<0.06	0.03	0.04
Ash, Wt.%								
850°F+	0.88	0.80	0.73	0.78	0.69	0.35	0.14	0.69
Total Liquid Product	0.18	0.14	0.24	0.20	0.20	0.19	Tr	0.12

TABLE VIII

LC-FINING PDU OPERATION
NITROGEN CONTENT OF DISTILLATE FRACTION (390-850°F)
PDU Run 2LCF-3 - Shell 324 Ni/Mo

2LCF-3 Blend Period	Recycle Pass No.	Reactor Temp. °F	Distillate Fraction 390-500°F Wt% Feed	390-500°F %N	500-650°F Wt% Feed	500-650°F %N	650-850°F Wt.% Feed	650-850°F %N	390-850°F Wt% N
11B	-	780	9.77	0.03	29.22	0.13	27.06	0.45	0.25
18	I	790	10.66	0.11	25.77	(0.1)*	26.93	0.30	0.19
23	II	790	8.75	0.21	26.48	0.15	27.82	0.39	0.26
28	III	800	9.61	0.22	23.76	0.23	29.01	0.47	0.34
33	IV	800	10.35	0.28	23.08	0.08	25.99	0.31	0.22
37	V	810	9.23	0.19	24.14	0.20	24.72	0.44	0.30
39	-	810	13.56	0.22	27.59	0.20	25.18	0.51	0.32
41	-	780	9.62	0.25	26.31	0.24	27.20	0.52	0.36

* Estimated

TABLE IX

TWO-STEP LIQUEFACTION PRODUCT YIELDS AND SRC-I CONVERSION

LC-Finer Operation	Recycle			
Recycle Pass, PDU Run 2LCF-3	II	III	IV	V
Temperature, °F	790	800	800	810
Combined Yields, Wt% of MF Coal				
H_2S	1.83	1.90	1.83	1.90
H_2O	6.96	7.27	7.56	7.16
NH_3	1.04	1.24	1.06	1.27
CO_2	0.80	0.80	0.80	0.80
C_1	2.96	3.66	3.69	3.91
C_2	2.63	3.37	2.68	3.61
C_3	2.98	3.94	3.38	4.36
C_4	2.47	3.09	2.57	3.81
C_5-390°F	10.60	12.45	12.02	15.43
390-500°F	14.24	14.69	14.66	16.66
500-650°F	11.97	8.20	8.84	5.06
650-850°F	8.89	9.95	9.32	6.56
850°F+	8.07	5.28	7.02	5.37
SRC-I, ASH, IOM	30.00	30.00	30.00	30.00
Total	105.44	105.85	105.43	105.84
Bbl/Ton MF Coal				
C_5-850°F	2.82	2.87	2.83	2.79
850°F+	0.38	0.24	0.32	0.24
Conversion of Fresh SRC-I, Wt%	83.2	89.0	85.4	88.8

TABLE X

AVERAGE PRODUCT YIELDS AND HYDROGEN CONSUMPTION
FOR TWO-STEP LIQUEFACTION WITH RECYCLE LC-FINER

	SRC-I Production with 2000 Lbs. MF Coal Feed		LC-Fining Operation with 960 Lbs. SRC-I		Total Products
	Wt.%[1]	Lbs.	Wt.%[3]	Lbs.	Lbs.
H_2	-2.4	-48	-6.8[4]	-64.8	-112.8
C_1	1.4	28	4.5	43.1	71.1
C_2	1.0	20	4.3	41.4	61.4
C_3	0.9	18	5.7	55.3	73.3
C_4	0.4	8	5.4	51.7	59.7
CO_2	0.8	16	-	-	16.0
H_2S	1.4	28	1.0	9.3	37.3
H_2O	5.0	100	4.7	44.8	144.8
NH_3	N.A.	-	2.4	22.7	22.7
C_5-390°F	5.9	118	14.0	134.5	252.5
390-500°F	3.7	74	23.7	227.3	301.3
500-650°F	1.8	36	14.0	134.4	170.4
650-850°F	2.1	42	13.7	131.6	173.6
850°F+	63.0	960[2]	13.4	128.7	128.7
		300	-	-	300
Ash	9.6	192	-	-	192
IOM	5.4	108	-	-	108
		2000		960.0	2000

Hydrogen Consumption:

$$\frac{112.8 \text{ lbs. } H_2/\text{Ton}}{2000 \text{ lbs./ton}} \times 100\% = 5.64 \text{ Wt.\% } H_2$$

Conversion of Fresh SRC-I in LC-Fining: 86.6 Wt.% (very high)

NOTES:

[1] Values from Moschitto's paper[3]

[2] 300 lbs. of SRC-I are assumed needed for removing the ash and insoluble organic material (IOM) leaving 960 lbs. for upgrading

[3] Averaged values from 2LCF-3, recycle Passes II, III, IV, and V

[4] Based on 960 lbs. fresh SRC-I feed

TABLE XI

TWO-STEP LIQUEFACTION PRODUCT YIELDS AND SRC-I CONVERSION

LC-Finer Operation	Once-Through
Temperature, °F	780
Combined Yields, Wt.% of MF Coal[1]	
H_2S	1.28
H_2O	5.88
NH_3	0.69
CO_2	0.57
C_1	1.78
C_2	1.44
C_3	1.44
C_4	1.17
C_5-390°F	8.32
390-500°F	8.94
500-650°F	20.13
650-850°F	18.95
850°F+	13.42
SRC, ASH, IOM	21.37
Total	105.38
Bbl/Ton MF Coal	
C_5-850°F[2]	2.44
850°F+	0.90
Conversion of Fresh SRC-I, Wt.%	60.7

NOTE:
[1] Contains foreign solvent
[2] Net yield with 2.20 Bbl/Ton solvent removed

TABLE XII

PRODUCT YIELDS AND HYDROGEN CONSUMPTION FOR TWO-STEP LIQUEFACTION WITH ONCE-THROUGH LC-FINER

	SRC-I Production with 2000 Lbs. MF Coal Feed		LC-Fining Operation with 960 Lbs. SRC-I		Total Products
	Wt.%[1]	Lbs.	Wt.%	Lbs.[3]	Lbs.[3]
H_2	-2.4	-48	-3.29	-59.6	-107.6
C_1	1.4	28	1.22	22.1	50.1
C_2	1.0	20	1.13	20.5	40.5
C_3	0.9	18	1.23	22.3	40.3
C_4	0.4	8	1.37	24.8	32.8
CO_2	0.8	16	-	-	16.0
H_2S	1.4	28	0.44	8.0	36.0
H_2O	5.0	100	3.59	65.0	165.0
NH_3	N.A.	-	1.07	19.4	19.4
C_5-390°F	5.9	118	6.38	115.6	233.6
390-500°F	3.7	74	9.77	177.0	251.0
500-650°F	1.8	36	29.22	529.2	565.2
650-850°F	2.1	42	27.06	490.1	532.1
850°F+	63.0	960[2]	20.81	376.9	376.9
		300	-	-	300
Ash	9.6	192	-	-	192
IOM	5.4	108	-		108
				1811.3	2851.3

Hydrogen Consumption:

$$\frac{107.6 \text{ lbs. } H_2/\text{Ton}}{2000 \text{ lbs./ton}} \times 100\% = 5.38 \text{ Wt.\% } H_2$$

NOTES:

1 Values from Moschitto's paper[3]

2 See Table II

3 Includes 851.3 lbs. solvent

FIGURE 1

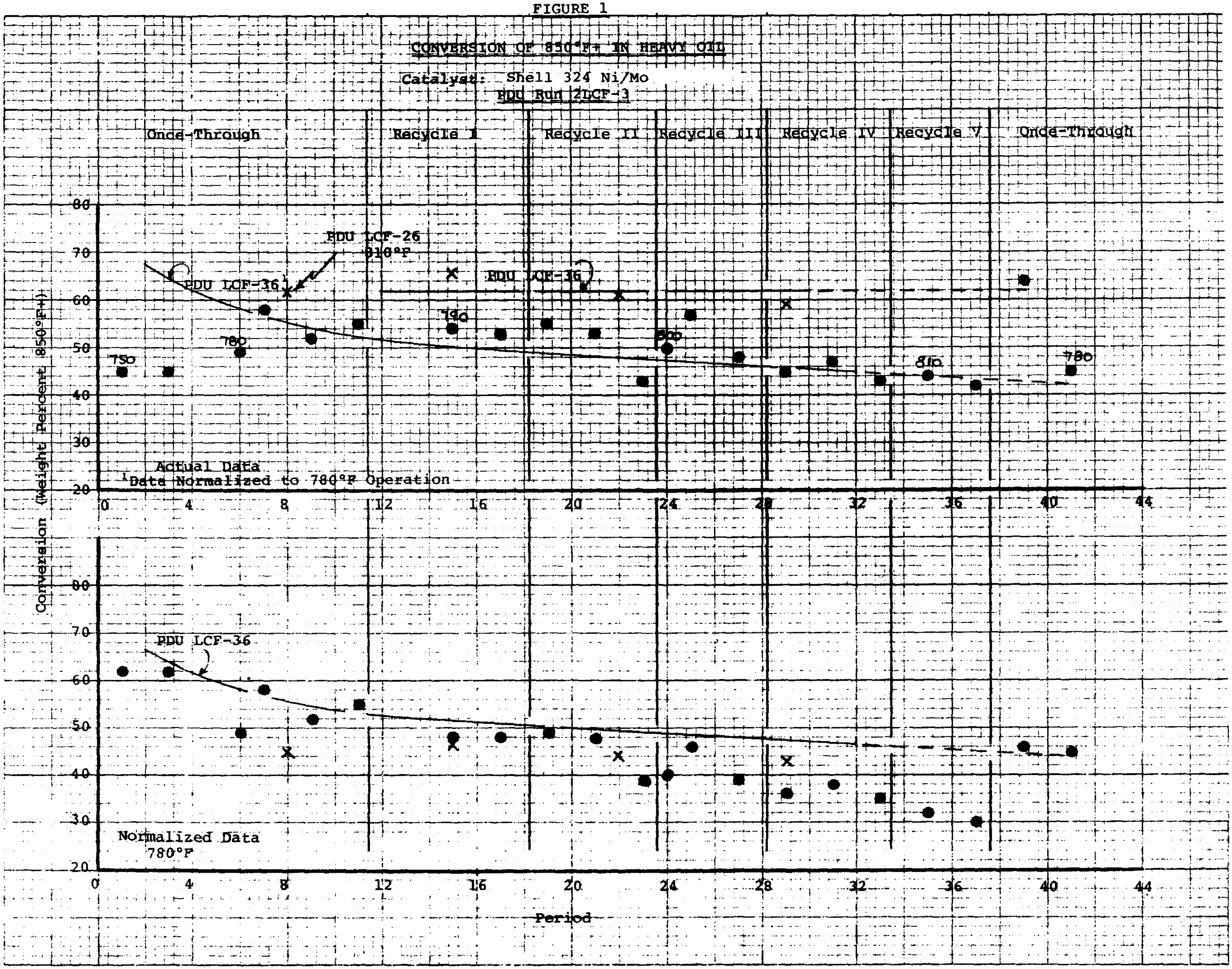

CONVERSION OF 850°F+ IN HEAVY OIL
Catalyst: Shell 324 Ni/Mo
PDU Run 2LCF-3
Once-Through
Recycle I
Recycle II
Recycle III
Recycle IV
Recycle V
Once-Through
PDU LCF-26
810°F
PDU LCF-36[1]
PDU LCF-36
750
780
790
800
810
780
Actual Data
[1]Data Normalized to 780°F Operation
Conversion (Weight Percent 850°F+)
PDU LCF-36
Normalized Data
780°F
Period

FIGURE 2

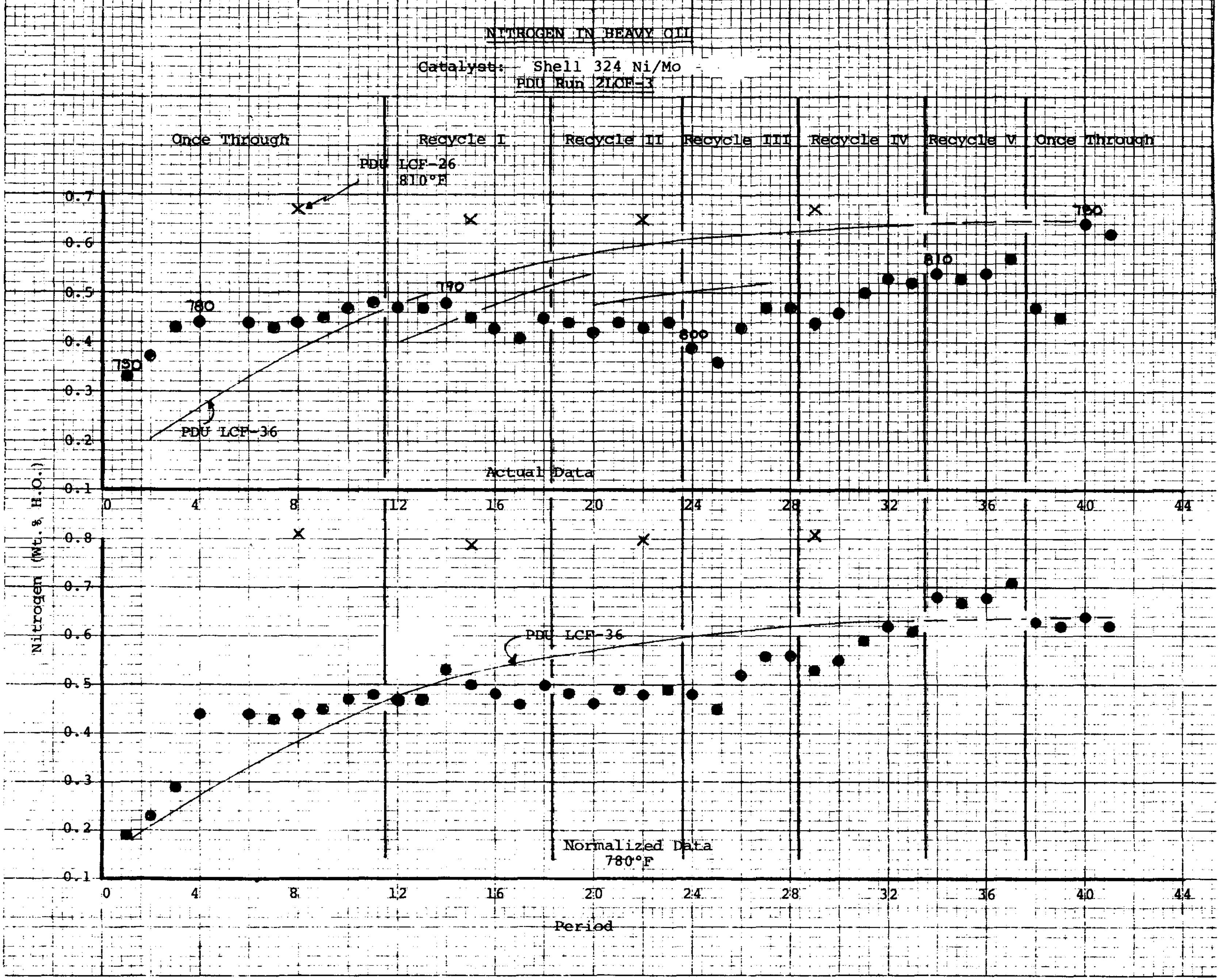

NITROGEN IN HEAVY OIL
Catalyst: Shell 324 Ni/Mo
PDU Run 2LCF-3
Once Through
Recycle I
Recycle II
Recycle III
Recycle IV
Recycle V
Once Through
PDU LCF-26
810°F
PDU LCF-36
Actual Data
750
780
770
800
810
790
Normalized Data
780°F
Nitrogen (Wt.% H.O.)
Period

FIGURE 3

CONVERSION OF 850°F+ (FEED BASIS)

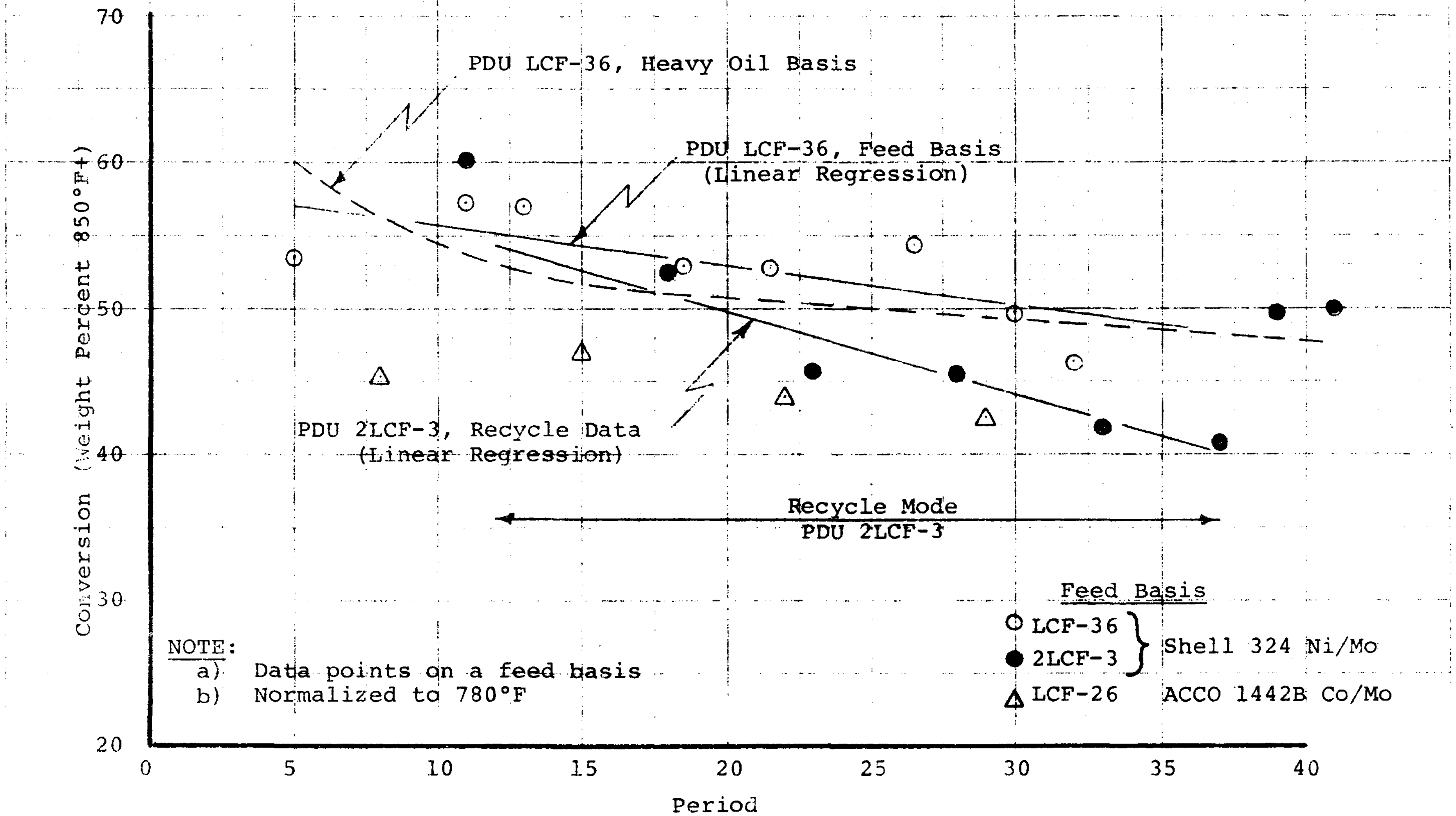
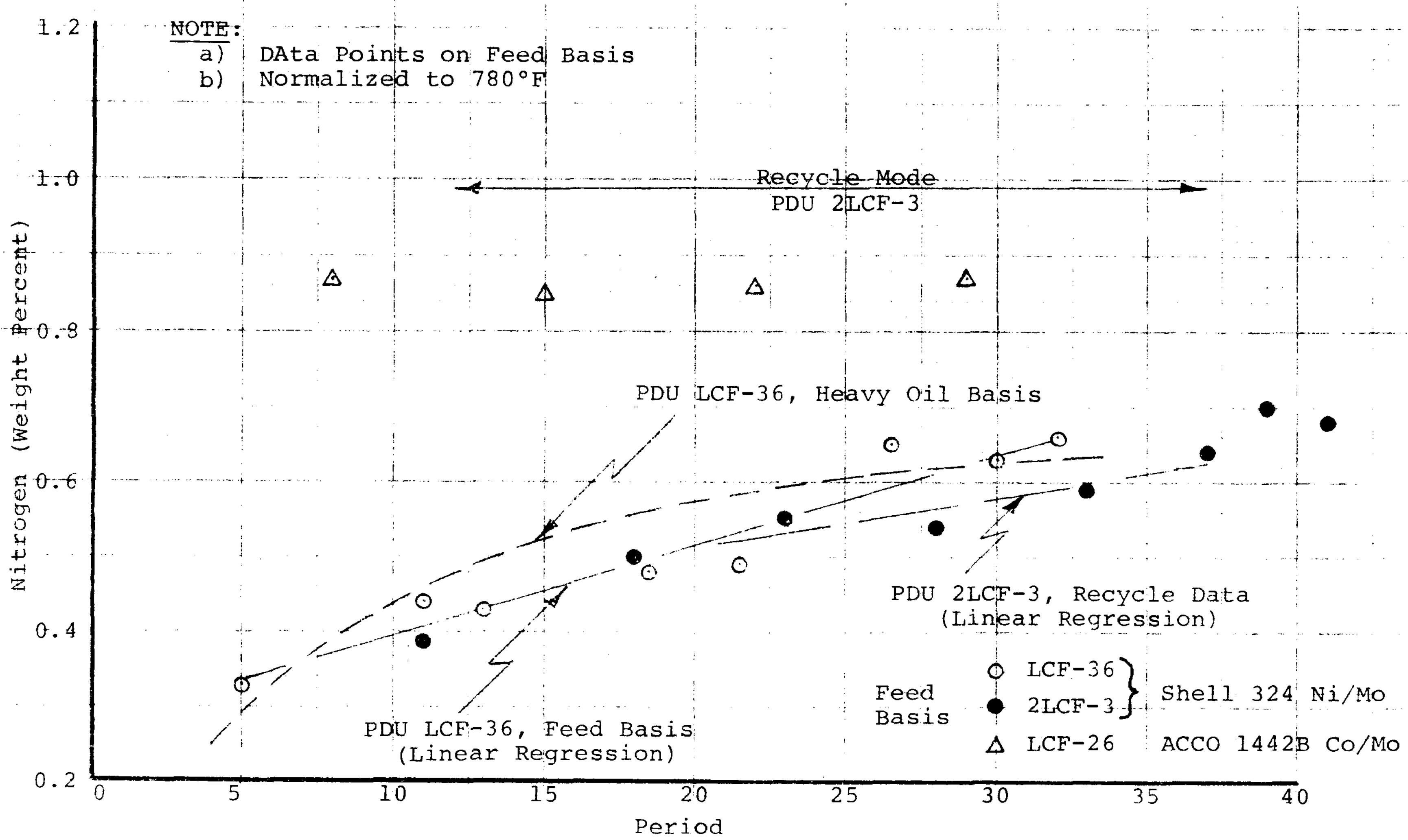


FIGURE 4

NITROGEN IN LIQUID PRODUCT (FEED BASIS)



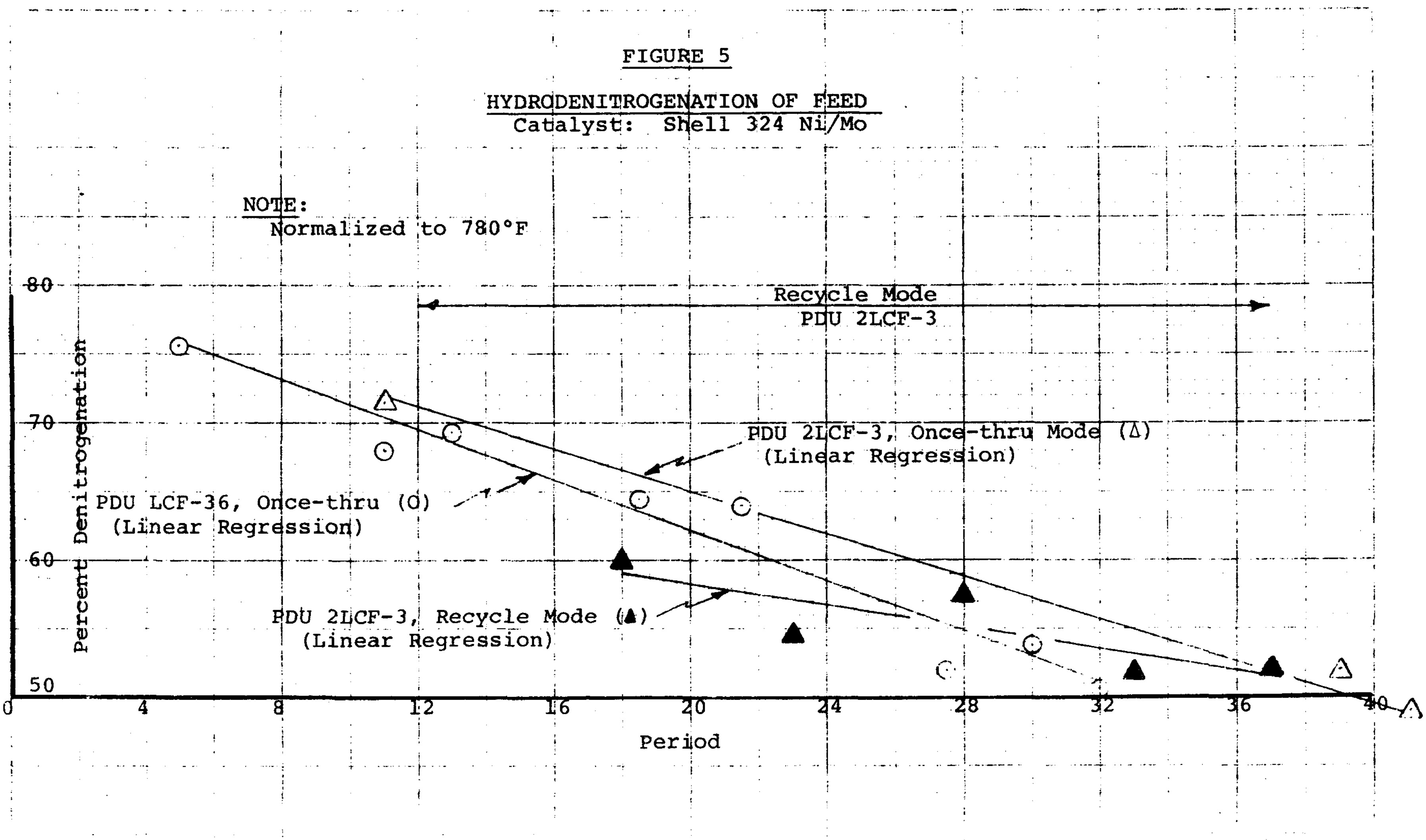


FIGURE 5
HYDRODENITROGENATION OF FEED
Catalyst: Shell 324 Ni/Mo
NOTE:
Normalized to 780°F
Recycle Mode
PDU 2LCF-3
PDU 2LCF-3, Once-thru Mode (Δ)
(Linear Regression)
PDU LCF-36, Once-thru (O)
(Linear Regression)
PDU 2LCF-3, Recycle Mode (▲)
(Linear Regression)
Percent Denitrogenation
80
70
60
50
0
4
8
12
16
20
24
28
32
36
40
Period

FIGURE 6

OPERATING TEMPERATURE PROFILES
Catalyst: Shell 324 Ni/Mo

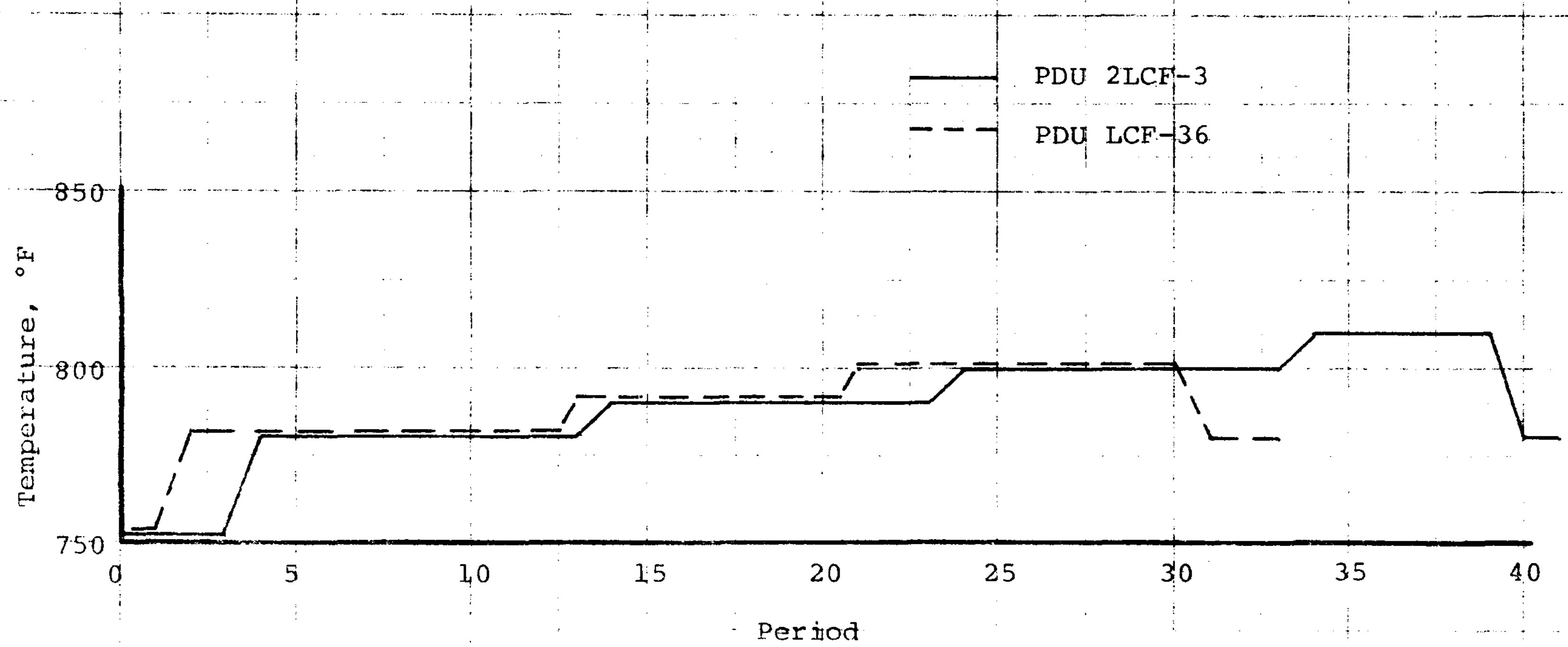


FIGURE 7

CONVERSION (850°F+ VERSUS 500°F+)
Catalyst: Shell 324 Ni/Mo

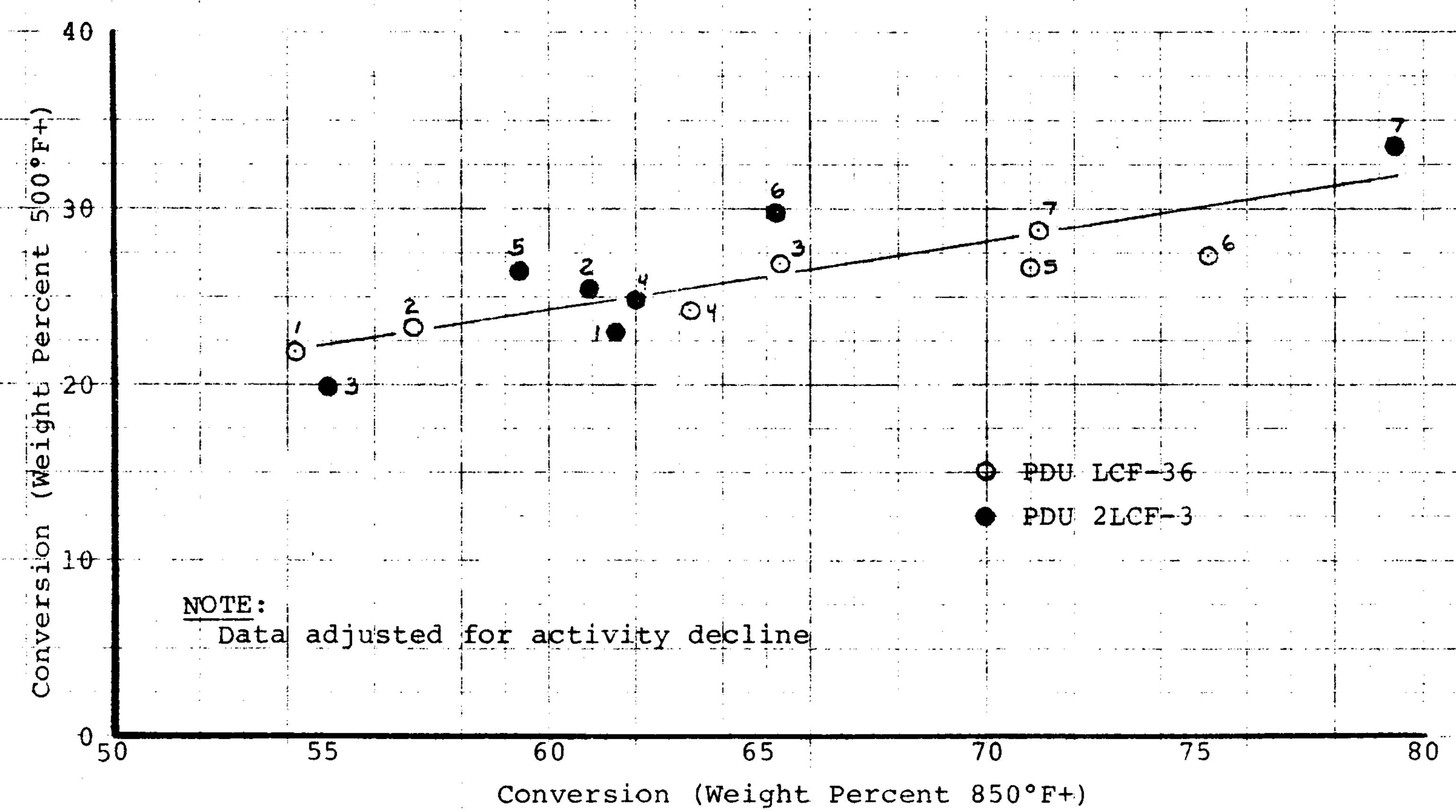


FIGURE 8

CONVERSION (850°F+ VERSUS 650°F+)
Catalyst: Shell 324 Ni/Mo

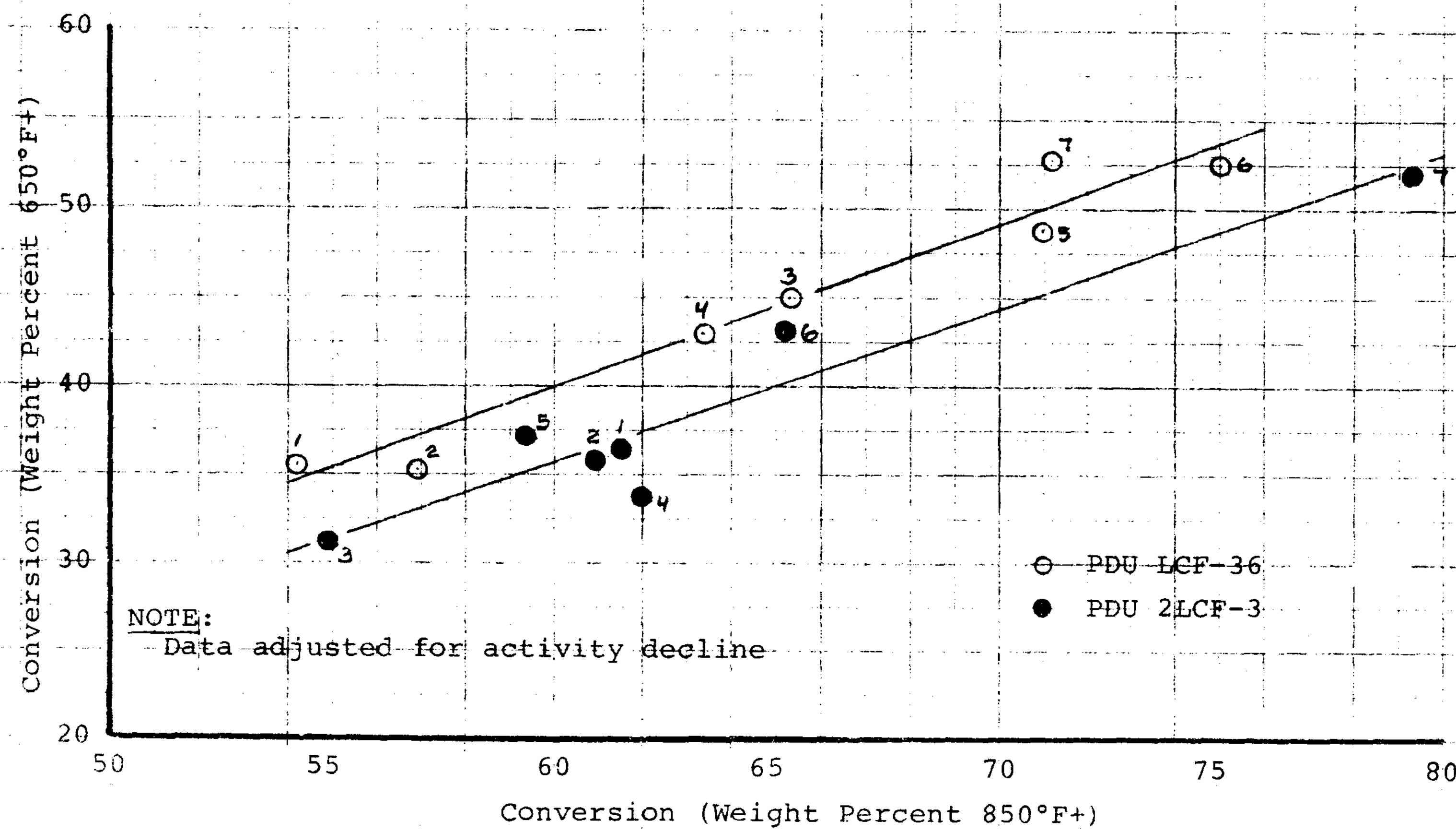


FIGURE 9

CONVERSION (850°F+) VERSUS 390-500°F YIELD
Catalyst: Shell 324 Ni/Mo

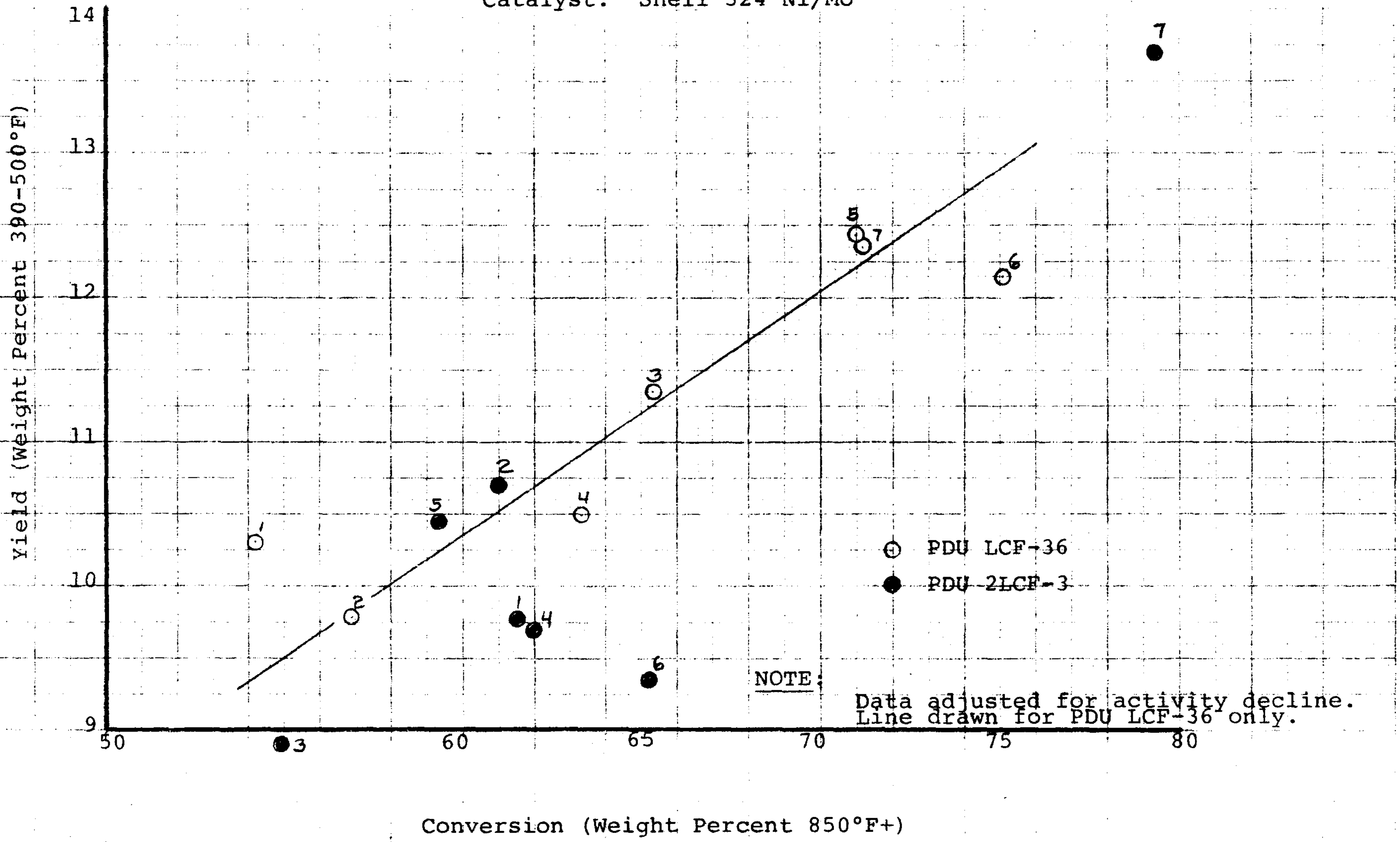


FIGURE 10

CONVERSION OF 850°F+ VERSUS C_1-C_4 YIELD
Catalyst: Shell 324 Ni/Mo

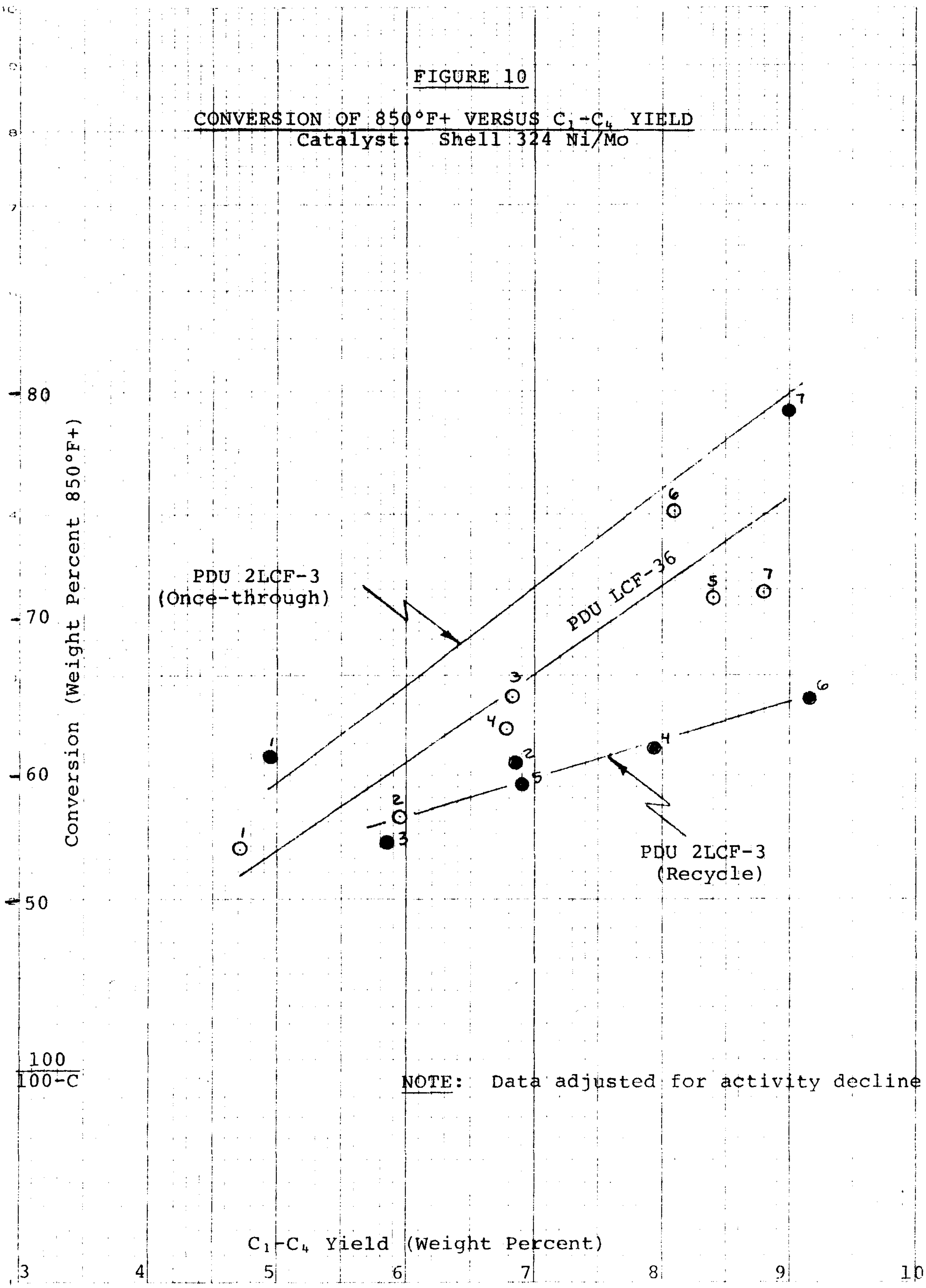


FIGURE 11

TWO-STEP LIQUEFACTION MATERIAL BALANCE

RECYCLE CASE

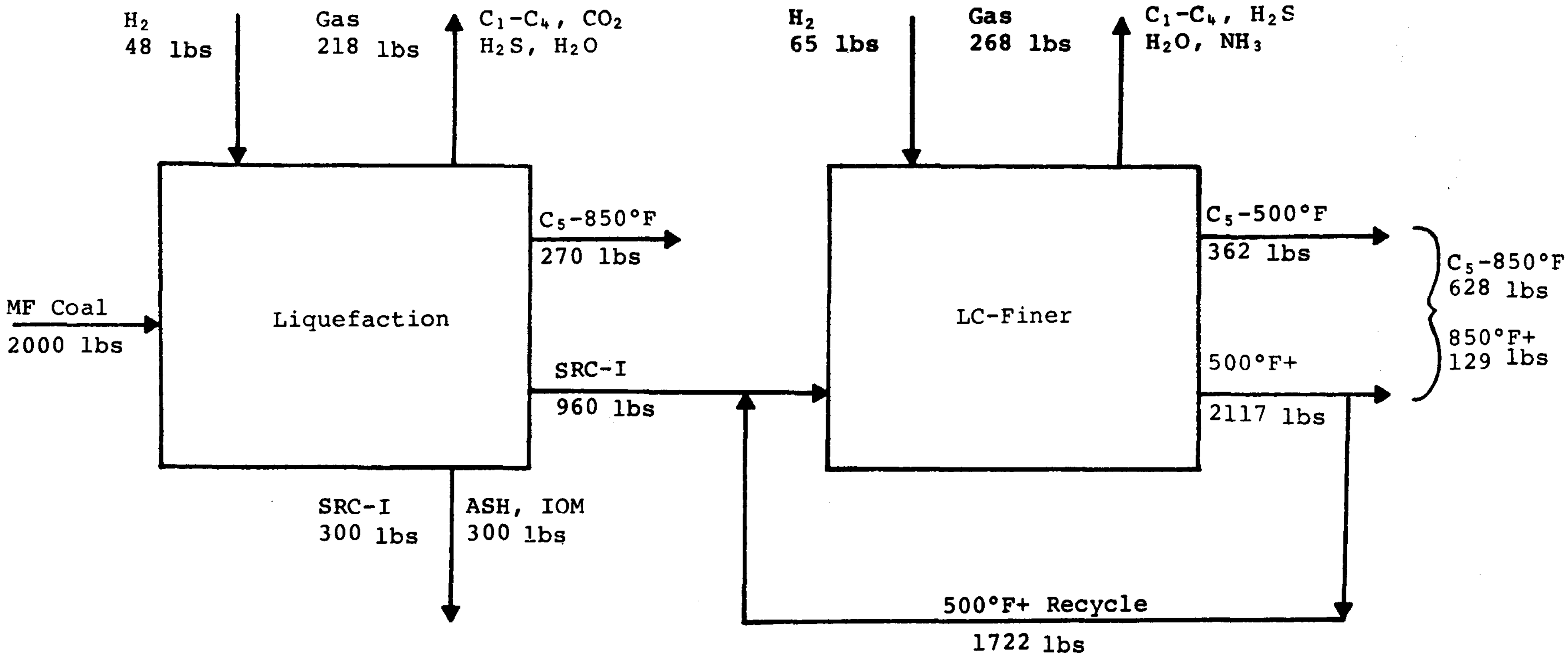
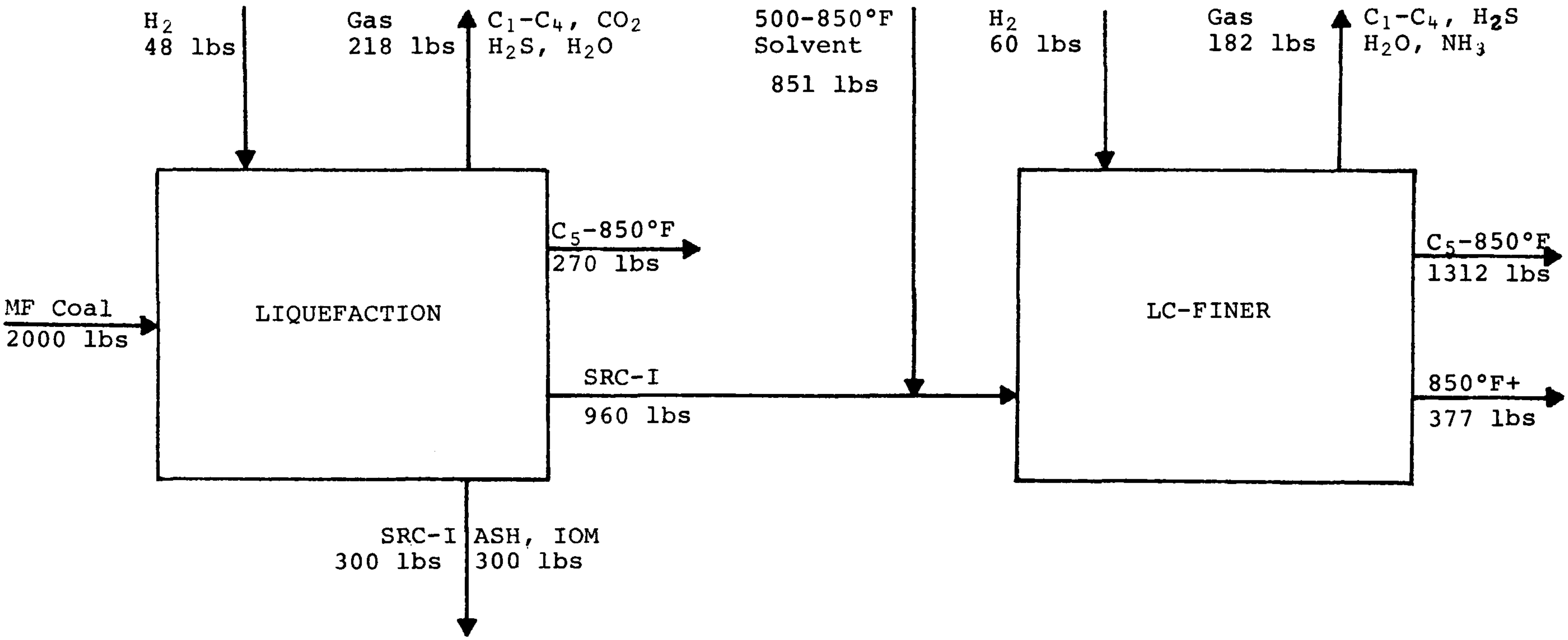


FIGURE 12

TWO-STEP LIQUEFACTION MATERIAL BALANCE

ONCE THROUGH CASE



<u>FIGURE 13</u>

<u>CATALYST ADDITION RATE</u>
PDU Run 2LCF-3

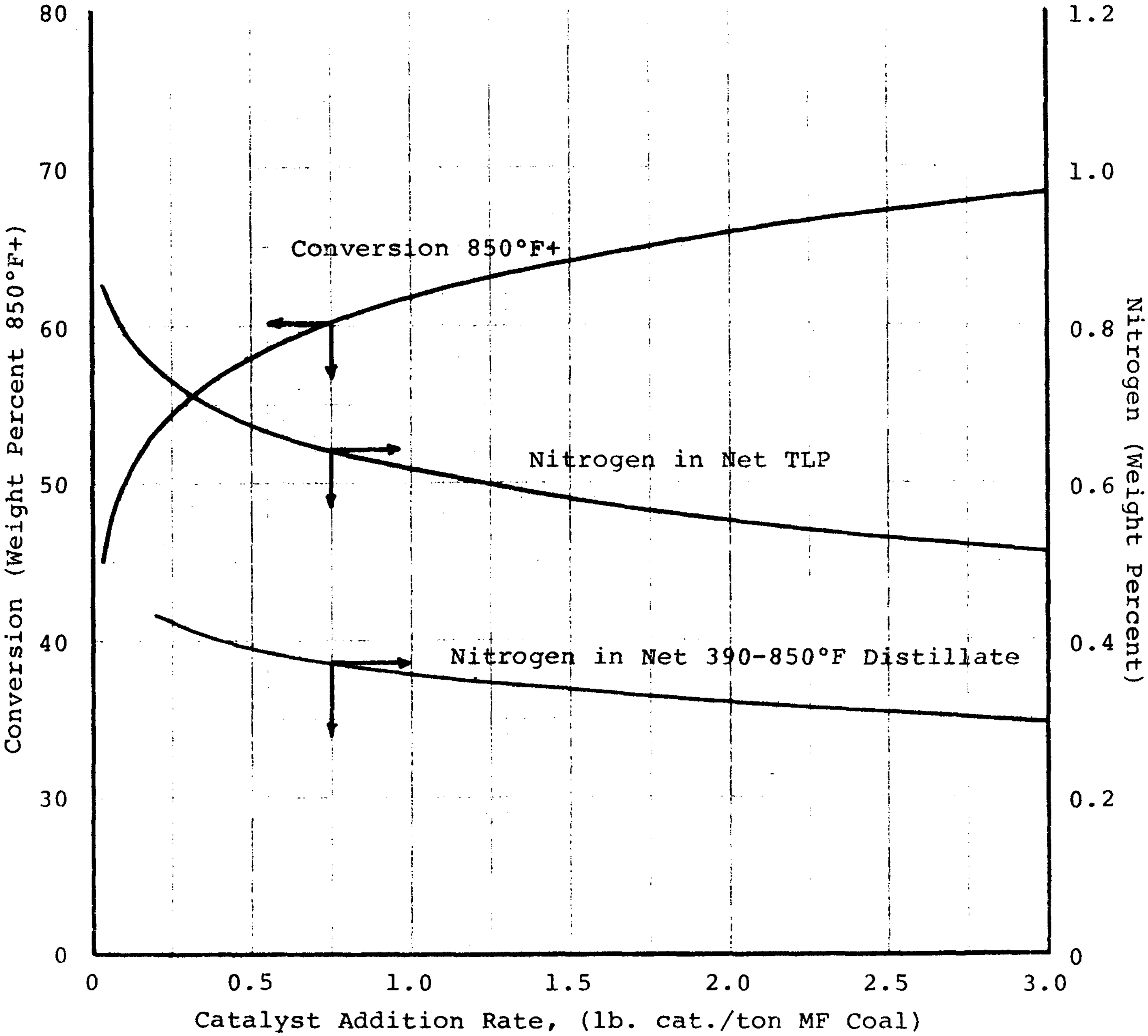
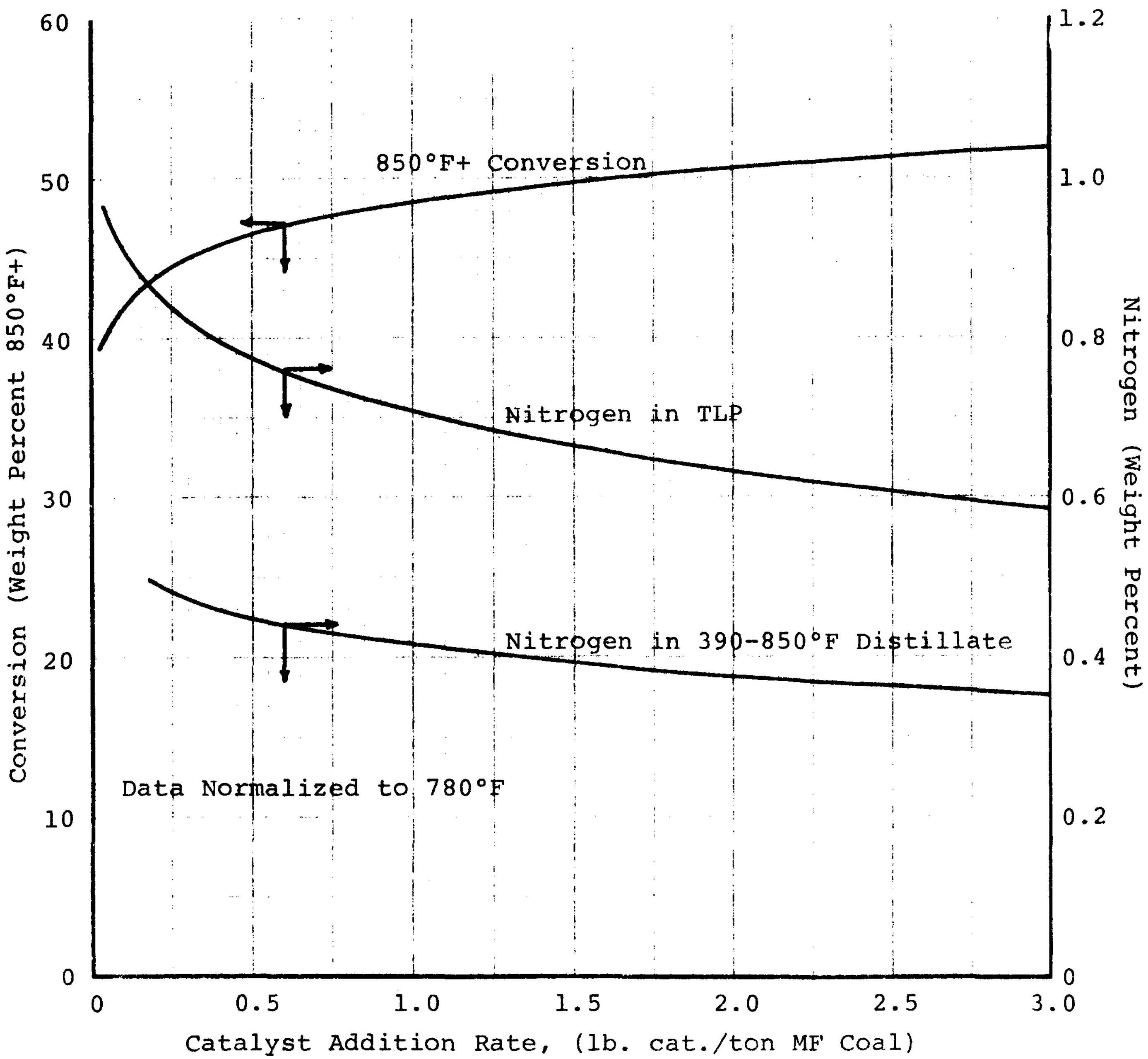


FIGURE 14

CATALYST ADDITION RATE
PDU Run LCF-36



APPENDIX

APPENDIX TABLE I

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE

PDU Run 2LCF-3 (Period 11B)

Once-through Operation

Fraction	Distillation Fractions					Total Liquid Product
	IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	
Volume Percent and	4.66(a)	12.44	34.42	30.25	20.78	102.55
Weight Percent on Fresh Feed	3.09(a)	9.77	29.22	27.06	20.81	89.95
Gravity, °API	32.9	18.5	7.5	0.4	-13.7	2.4
SP 60/60°F	0.8607	0.9433	1.0180	1.0728	1.201	1.0568
Pour Point, °F	<-16	<-10	<-14	<10	-	-5
Softening Point, °F	-	-	-	-	219	-
Viscosity, CST @ 100°F	1.13	2.22	8.17	63.32		50.32
CST @ 210°F	0.62	1.02	2.00	5.58		4.62
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	2.2	0.8				
Naphthenes	-	-				
Aromatics	33.6	68.4				
Saturates	64.2	30.8				
Elemental, Wt%						
Carbon	85.68	88.10	90.03	91.03	91.63	90.73
Hydrogen	11.84	10.26	9.08	8.09	6.69	8.35
Oxygen	0.90	0.24	0.41	0.28	1.11	0.43
Nitrogen	0.10	0.03	0.13	0.45	1.21	0.39
Sulfur	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06
Ash, Wt%					0.88	0.18

(a) C_5+ in gas are 5.54 liquid volume % and 3.28 weight % on feed

APPENDIX TABLE II

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE

PDU Run 2LCF-3 (Period 18)

Recycle Pass I

Fraction	Distillation Fractions IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	Total Liquid Product
Volume Percent and	3.85(a)	12.90	29.32	28.91	20.93	95.91
Weight Percent on Fresh Feed	2.85(a)	10.66	25.77	26.93	22.45	88.66
Gravity, °API	33.2	19.3	10.2	2.2	-15.3	2.6
SP 60/60°F	0.8591	0.9383	0.9986	1.0583	1.218	1.0552
Pour Point, °F	<-16	<-12	<-10	40	-	-10
Softening Point, °F	-	-	-	-	221	-
Viscosity, CST @ 100°F	1.07	2.33	7.58	84.43		50.32
CST @ 210°F	0.58	0.97	2.07	5.23		5.45
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	3.6	2.9				
Naphthenes	-	-				
Aromatics	41.1	71.9				
Saturates	55.3	25.3				
Elemental, Wt%						
Carbon	86.53	89.30	90.39	90.84	91.06	89.02
Hydrogen	11.78	10.42	9.51	8.30	6.31	8.48
Oxygen	0.64	0.22	0.26	0.24	0.85	0.45
Nitrogen	0.05	0.11	-	0.30	1.13	0.45
Sulfur	<0.06	<0.06	<0.06	<0.06	0.13	<0.06
Ash, Wt%					0.80	0.14

(a) C_5 in gas are 6.88 liquid volume % and 4.22 weight % on feed

APPENDIX TABLE III

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE
Run 2LCF-3 (Period 23)

Fraction	Distillation Fractions					Total Liquid Product
	IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	
Volume Percent and	3.37(a)	10.86	30.23	29.92	25.29	99.67
Weight Percent on Fresh Feed	2.51(a)	8.85	26.48	27.82	26.80	92.46
Gravity, °API	31.6	19.1	12.1	2.8	-15.8	2.3
SP 60/60°F	0.8676	0.9396	0.9854	1.0458	1.223	1.0575
Pour Point, °F	<-16	<-16	<-16	10	-	0
Softening Point, °F	-	-	-	-	230	-
Viscosity, CST @ 100°F	1.14	2.22	6.41	63.06		115.6
CST @ 210°F	0.61	0.94	1.77	4.86		8.76
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	3.4	7.8				
Naphthenes	-	-				
Aromatics	46.7	55.1				
Saturates	49.9	37.1				
Elemental, Wt%						
Carbon	86.68	88.89	89.03	91.26	90.36	89.76
Hydrogen	11.53	10.86	9.61	8.54	6.34	8.30
Oxygen	0.86	0.26	0.25	0.29	1.10	0.49
Nitrogen	0.07	0.21	0.15	0.39	1.21	0.50
Sulfur	<0.06	<0.06	<0.06	<0.06	0.15	0.04
Ash, Wt%					0.73	0.24

(a) C_5+ in gas are 2.35 liquid volume % and 1.44 weight % on feed

APPENDIX TABLE IV

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE

PDU Run 2LCF-3 (Period 28)

Recycle Pass III

Fraction	Distillation Fractions					Total Liquid Product
	IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	
Volume Percent and	3.41(a)	11.64	27.39	30.75	21.59	94.79
Weight Percent on Fresh Feed	2.57(a)	9.61	23.76	29.01	23.84	88.78
Gravity, °API	31.7	19.5	11.6	-0.15	-19.2	1.6
SP 60/60°F	0.8670	0.9371	0.9888	1.0773	1.260	1.0631
Pour Point, °F	<-16	<-16	<-16	20	-	0
Softening Point, °F	-	-	-	-	233	-
Viscosity, CST @ 100°F	1.14	2.26	7.06	70.01		74.90
CST @ 210°F	0.63	1.01	1.86	5.67		6.89
Hydrocarbon Type, Vol%						
Paraffins	5.3	-				
Olefins	3.0	2.5				
Naphthenes	41.9	-				
Aromatics	49.8	85.1				
Saturates	47.2	12.4				
Elemental, Wt%						
Carbon	87.19	89.19	89.10	91.20	92.46	89.82
Hydrogen	12.18	10.88	9.71	8.40	5.79	8.23
Oxygen	0.93	0.28	0.22	0.24	0.88	0.41
Nitrogen	0.16	0.22	0.23	0.47	0.97	0.44
Sulfur	<0.06	<0.06	<0.06	<0.06	0.13	<0.06
Ash, Wt%					0.78	0.20

(a) C_5+ in gas are 5.17 liquid volume % and 3.17 weight % on feed

APPENDIX TABLE V

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE

PDU Run 2LCF-3 (Period 33)
Recycle Pass IV

Fraction	Distillation Fractions IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	Total Liquid Product
Volume Percent and	5.91(a)	12.43	26.23	27.63	23.93	96.13
Weight Percent on Fresh Feed	4.28(a)	10.35	23.08	25.99	26.29	89.99
Gravity, °API	30.7	18.7	11.3	2.1	-17.1	1.0
SP 60/60°F	0.8724	0.9421	0.9909	1.0591	1.237	1.0679
Pour Point, °F	<-20	<-20	<-18	20	-	-10
Softening Point, °F	-	-	-	-	231	-
Viscosity, CST @ 100°F	1.11	2.15	7.12	68.98		82.37
CST @ 210°F	0.65	0.93	1.96	4.83		7.17
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	2.6	6.8				
Naphthenes	-	-				
Aromatics	58.0	84.1				
Saturates	39.4	9.1				
Elemental, Wt%						
Carbon	87.22	39.50	90.36	91.97	91.80	90.09
Hydrogen	11.34	10.29	9.77	8.44	6.03	8.18
Oxygen	1.34	0.31	0.25	0.25	0.83	0.47
Nitrogen	0.11	0.28	0.08	0.31	1.21	0.50
Sulfur	<0.06	<0.06	<0.06	<0.06	0.15	0.04
Ash, Wt%					0.69	0.20

(a) C_5+ in gas are 4.64 liquid volume % and 2.73 weight % on feed

APPENDIX TABLE VI

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE

PDU Run 2LCF-3 (Period 37)

Recycle Pass V

Fraction	Distillation Fractions					Total Liquid
	IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	Product
Volume Percent and	6.05(a)	11.20	27.68	26.29	21.42	92.64
Weight Percent on Fresh Feed	4.64(a)	9.23	24.14	24.72	23.80	86.58
Gravity, °API	29.5	18.4	10.9	0.5	-19.7	0.7
SP 60/60°F	0.8789	0.9440	0.9937	1.0716	1.266	1.0703
Pour Point, °F	<-20	<-20	<-20	50	-	-5
Softening Point, °F	-	-	-	-	242	-
Viscosity, CST @ 100°F	1.14	2.23	7.44	84.34		68.85
CST @ 210°F	0.66	0.94	1.86	5.40		5.63
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	4.3	1.9				
Naphthenes	-	-				
Aromatics	56.4	87.8				
Saturates	39.3	10.3				
Elemental, Wt%						
Carbon	87.39	90.02	89.89	90.55	92.27	89.89
Hydrogen	11.01	9.95	9.32	7.75	5.36	7.93
Oxygen	1.15	0.37	0.27	0.27	0.62	0.49
Nitrogen	0.22	0.19	0.20	0.44	1.29	0.49
Sulfur	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06
Ash, Wt%					0.35	0.19

(a) C_5 in gas are 5.44 liquid volume % and 3.32 weight % on feed

APPENDIX TABLE VII

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE

PDU Run 2LCF-3 (Period 39)

Once-through Operation

Fraction	Distillation Fractions IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	Total Liquid Product
Volume Percent and	5.74(a)	17.02	32.24	27.67	15.63	98.18
Weight Percent on Fresh Feed	4.16(a)	13.56	27.59	25.18	16.19	86.68
Gravity, °API	28.0	16.2	6.1	-2.1	-17.8	1.7
SP 60/60°F	0.8871	0.9580	1.0283	1.0934	1.245	1.0623
Pour Point, °F	<-20	<-20	<-16	40	-	-15
Softening Point, °F	-	-	-	-	247	-
Viscosity, CST @ 100°F	1.21	2.44	8.14	94.67		24.49
CST @ 210°F	0.62	0.99	1.96	5.30		3.54
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	3.7	5.1				
Naphthenes	-	-				
Aromatics	56.6	87.2				
Saturates	39.9	7.7				
Elemental, Wt%						
Carbon	86.62	90.26	90.18	91.69	92.74	89.73
Hydrogen	11.01	9.89	8.43	7.42	5.60	7.80
Oxygen	1.29	0.50	0.43	0.33	0.71	0.51
Nitrogen	0.24	0.22	0.20	0.51	1.29	0.53
Sulfur	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06
Ash, Wt%					0.14	TR

(a) C_5+ in gas are 5.69 liquid volume % and 3.40 weight % on feed

APPENDIX TABLE VIII

LIQUID PRODUCT YIELDS FROM THE PROCESSING OF SRC-I/KOPPERS CREOSOTE
PDU Run 2LCF-3 (Period 41)
Once-Through Operation

Fraction	Distillation Fractions					Total Liquid Product
	IBP-390°F	390-500°F	500-650°F	650-850°F	850°F+	
Volume Percent and	3.96(a)	12.08	30.75	30.09	25.94	102.42
Weight Percent on Fresh Feed	3.05(a)	9.62	26.31	27.29	26.18	92.36
Gravity, °API	25.9	16.5	6.3	-1.12	-14.65	-0.8
SP 60/60°F	0.8990	0.9561	1.0264	1.0853	1.211	1.0823
Pour Point, °F	<-16	<-16	<-16	30	-	5
Softening Point, °F	-	-	-	-	261	-
Viscosity, CST @ 100°F	0.91	2.52	8.35	112.62		115.6
CST @ 210°F	0.62	1.01	2.00	5.54		7.04
Hydrocarbon Type, Vol%						
Paraffins	-	-				
Olefins	2.6	4.5				
Naphthenes	-	-				
Aromatics	52.7	84.1				
Saturates	44.7	11.4				
Elemental, Wt%						
Carbon	87.27	88.61	90.48	91.13	90.81	90.12
Hydrogen	11.06	9.97	8.84	7.86	6.33	7.94
Oxygen	1.92	0.63	0.53	0.45	1.28	0.77
Nitrogen	0.36	0.25	0.24	0.52	1.22	0.68
Sulfur	<0.06	<0.06	<0.06	<0.06	0.17	0.04
Ash, Wt%					0.69	0.12

(a) C_5+ in gas are 3.31 liquid volume % and 1.99 weight % on feed

APPENDIX TABLE IX

850°F+ VISCOSITY AND TOTAL LIQUID PRODUCT METALS
PDU Run 2LCF-3

LCF-Period 2LCF-3	11b		18		23		28		33		37		39		41	
Feed Blend	Once-Through		Recycle I		Recycle II		Recycle III		Recycle IV		Recycle V		Once-Through		Once-Through	
Average Reactor Temp., °F	780		790		790		800		800		810		810		780	
Viscosity (a) 850°F+ Temperature, °F	400	450	400	450	400	450	400	450	400	450	400	450	400	450	400	450
ABS CPS at Shear Rate of																
0.2 Sec^{-1}	-	-	-	-	-	-	-	-	-	-	300	-	-	-	-	-
0.4	200	110	150	80	180	120	190	110	200	100	240	100	300	200	-	360
0.8	125	75	105	50	130	90	145	89	150	75	220	90	225	130	600	200
2.0	90	48	80	42	100	50	98	49	106	50	200	60	174	86	360	110
4.0	80	37	70	33	89	40	88	46	91	48	175	111	155	65	340	90
8.0	72.5	30.5	63	27.5	83.5	34.5	84.8	37.6	83.5	38.5	160	95	145	53.5	270	80
16.0	70	27	61.3	25	82.5	31.3	81	30	80.8	30	154	71.7	143.8	47.7	250	78.7
40.0	68.1	25.4	61.5	23	82	29.5	80.8	27.5	80.6	28.6	-	58.6	-	43.8	246	78.0
80.0	-	25.5	-	23.4	-	29.3	-	24.5	-	28.7	-	-	-	43.3	245	-
Spindle	18	18	18	18	18	18	18	18	18	18	18	34	18	18	18	18
Spindle Factor	1.32	1.32	1.32	1.32	1.32	1.32	1.32	1.32	1.32	1.32	1.32	0.28	1.32	1.32	1.32	1.32
Total Liquid Product Metals, PPM (b)																
Vanadium (c)	<20		<20		<20		<20		<20		<20		<20		<20	
Nickel	2		<2		4		2		<2		4		9		<2	
Iron (c)	<2		<2		<2		<2		2		6		22		<2	
Titanium	<20		<20		<20		<20		20		66		152		<20	
Sodium	<2		<2		<2		<2		<2		<2		<2		<2	
Potassium	<2		<2		<2		<2		<2		<2		<2		<2	
Calcium	11		16		11		15		22		15		37		6	

(a) Viscosity Measured with Brookfield Viscometer
(b) Metals analyzed by Suntech Co.
(c) 20 and 2 PPM are the detection limits for the sample size used.