

LC-FINING OF SRC - A LOGICAL SECOND  
STAGE IN TWO-STEP COAL LIQUEFACTION

MASTER

Chillingworth, Richard S.  
Hastings, Kenneth E.  
Potts, John D.

Cities Service Company  
Technology Assessment Department  
Tulsa, Oklahoma 74102

Unger, Harold  
C-E Lummus Company  
A Subsidiary of Combustion Engineering, Inc.  
Engineering Development Center Annex  
New Brunswick, New Jersey 08902

Presented at the 87th National AIChE  
Meeting in Boston, Massachusetts  
August 20, 1979

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

21

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

ADDENDUM - HYDROGEN CONSUMPTION AND YIELDS  
(Text Pages 8-11)

Since this paper was written, additional information and data have become available to corroborate the statement to the effect that the hydrogen consumptions reported in the text are high and a reflection of a very high severity PDU operation.

Cities Service and C-E Lummus Company have successfully developed proprietary SRC hydroprocessing yield correlations in terms of LC-Finer (LCF) operation variables such as temperature, space velocity, and solvent to SRC feed blend ratio. A joint effort between Air Products and Chemicals, Inc. (APCI) and Cities Service/Lummus produced yield structures for Two-Step Liquefaction (TSL) using high and low severity SRC processing.

One case was structured to take into account the more natural conversion and selectivity characteristics of the hydroprocessing catalyst than those presented in this paper for the recycle operation. The SRC was less severely hydro-cracked, producing a distillate product richer in the heavier fractions, with moderate sulfur and nitrogen removal. In this case, 52 wt.% of the feed SRC was converted to clean distillates and gaseous fuels at a hydrogen consumption level of 2.5 wt.% of the SRC feed to the LC-Finer. Consequently, the hydrogen consumption for the TSL was approximately 3.8 wt.%. This compares to a hydrogen consumption of 4.8 wt.% for SRC-II at a comparable 41.0 wt.% (MAF) distillate product.

Yields

<u>Hydrocarbons</u> (Wt.% MAF Basis)	<u>TSL</u>	<u>SRC-II</u>
C <sub>1</sub> /C <sub>4</sub> Gases	13.01	18.40
Distillates	41.07	40.70
SRC	29.73	27.80
H <sub>2</sub> Consumption	3.8	4.8

Heteroatom Contents

<u>Fraction</u>	<u>LCF</u>	<u>TSL</u>	<u>SRC-II</u>
350-850°F B.P. Product			
Wt.% S	0.08	0.20	0.29
Wt.% N	0.60	0.61	1.22
SRC Product			
Wt.% S	0.26	0.26	0.59
Wt.% N	1.83	1.83	2.22

Additional data, which is beyond the scope of this paper, will be presented on comparative studies between TSL and SRC-II at the Sarnia, Ontario, Canada meeting of the Canadian Society for Chemical Engineers and the Philadelphia meeting of the AIChE.

## LC-Fining of SRC - A Logical Second Stage in Two-Step Coal Liquefaction

R. S. Chillingworth, K. E. Hastings, J. D. Potts (Speaker),  
Cities Service Company, Tulsa, Oklahoma; and H. Unger,  
C-E Lummus Company, New Brunswick, New Jersey.

### 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, a subsidiary of Combustion Engineering, Inc. 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. 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.

\* Service mark of The Lummus Company and Cities Service Company.

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.

#### Prior Studies - Conclusions

Two DOE Interim Technical Progress Reports (FE-2038-17<sup>1</sup> and FE-2038-25<sup>2</sup>) 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.

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.

Several technical presentations<sup>3,4,5,6,7</sup> have also been made which expand upon the above data and also present additional studies recently completed.

## Objective

The objective of the work described in this paper was to determine the effectiveness of the LC-Fining Process, when operating in the recycle mode, for improved denitrogenation of SRC-I extract using recycle solvent (500-850°F) derived from the LC-Finer, 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.

Also described in this paper will be the preliminary results obtained by applying the recycle mode of LC-Finer operation to two-step liquefaction. A two-step liquefaction process separates the coal dissolution process from the coal liquids upgrading process.

## 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)<sup>1</sup>.

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.

## 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

### Total Liquid Product (TLP) Analysis - Conversion and Denitrogenation

A compilation of yield data and elemental fraction analyses for PDU Run 2LCF-3 is presented in Tables IV and V. Table IV



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 by summation against the comparable analysis for the total liquid product.

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

Figure 1 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 the once-through operation of 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<sup>2</sup> for PDU Run LCF-36, based upon heavy oil analyses only. The 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 2 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 1. 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 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 VI 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.

#### 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 prehydrogenated 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.

<u>Pass</u>	<u>°F</u>	Hydrogen (Wt.%)
		<u>500-850°F</u>
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 Based Only Upon PDU Run 2LCF-3 (Recycle Mode of Operation)

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<sup>8</sup>. The LC-Finer data was obtained from one of our Interim Technical Progress Reports<sup>2</sup>, and the data of this paper. 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 IV). 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.

Table VII 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<sub>5</sub>-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 VIII shows the breakdown of the products from the liquefaction step and the averaged LC-Fining step. Figure 3 is a simplified material balance schematic of the two-step liquefaction operation.

Two points should be mentioned in examining the yields presented in Table VIII. 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 VIII 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 paper.

For comparison, a once-through LC-Fining case was generated where no material was recycled to the reactor and a 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. This combined with the liquefaction hydrogen consumption gave a combined two-step consumption of 5.38 weight percent.

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 60.7 weight percent, once again a very high conversion of fresh SRC-I. This calculation assumes solvent-in = solvent-out.

It should be re-emphasized that the high hydrogen consumption figures reported above are a direct result of the high conversion of fresh SRC-I feed. The recycle of a 500-850°F+ material prior to the addition of the fresh 850°F+ SRC will increase the make of naphtha and distillate. This lighter product will consequently

consume more hydrogen. A beneficial result of the high hydrogen consumption is the very effective heteroatom removal. Proprietary commercial design considerations have indicated that a two-step hydrogen consumption of 4 weight percent is a realistic value at 50 volume percent conversion of 850°F+.

#### 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<sup>9</sup> 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.

At a catalyst replacement rate of one pound of catalyst per ton of moisture free coal, 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  
(390-850°F)

## 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 at temperatures of 790-810°F;
- 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 at 780°F, 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.

## Acknowledgement

This work was supported by the U.S. Department of Energy under Contract No. EX-76-C-01-2038. We appreciate the technical advice from Dr. E. Moroni of DOE and Mr. R. H. Long of C-E Lummus. The excellent technical assistance of Mr. K. Moy of C-E Lummus is also gratefully acknowledged.



## REFERENCES

- 1 Potts, J. D., Hastings, K. E., Wysocki, E. D.; Interim Technical Progress Report on Expanded Bed Hydroprocessing of Solvent Refined Coal (SRC) Extract, No. FE-2038-17, November, 1977.
- 2 Potts, J. D., Hastings, K. E., Unger, H.; Interim Technical Progress Report on Expanded Bed Hydroprocessing of Solvent Refined Coal (SRC) Extract, No. FE-2038-25, August, 1978.
- 3 Hastings, K. E., Potts, J. D., and Unger, Harold; "Expanded Bed Hydroprocessing of Solvent Refined Coal (SRC-I) Extract," presented at Fifth International Conference on Coal Gasification, Liquefaction, and Conversion to Electricity, Pittsburgh, Pennsylvania (August, 1978).
- 4 Chillingworth, R. S., Hastings, K. E., Potts, J. D., and Unger, Harold; "LC-Fining Produces Low Nitrogen Distillates from Solvent Refined Coal," presented at 71st AIChE Meeting, Miami Beach, Florida (November 15, 1978).
- 5 Chillingworth, R. S., Hastings, K. E., Potts, J. D., and Unger, Harold; "Alternative Modes of Processing SRC in an Expanded Bed LC-Finer to Produce Low Nitrogen Distillates," presented at 86th National AIChE Meeting, Houston, Texas, April 3, 1979.
- 6 DOE Contract Review Meeting on Coal Liquefaction Refining, Washington, D.C., May 16, 1978.
- 7 DOE Contract Review Meeting on Refining of Syncrudes, Richmond, California, May 7-8, 1979.

- 8 Moschitto, R. D.; "Operation of the Fort Lewis, Washington Solvent Refined Coal (SRC) Pilot Plant in the SRC-I and SRC-II Processing Modes," 13th Intersociety Energy Conversion Engineering Conference, San Diego, CA, August, 1978.
- 9 Anderson, S. L., and Matthias, R. H.; "Prediction of Catalyst Activity in Fluidized Systems," IEC, 46, #6, June, 1954.

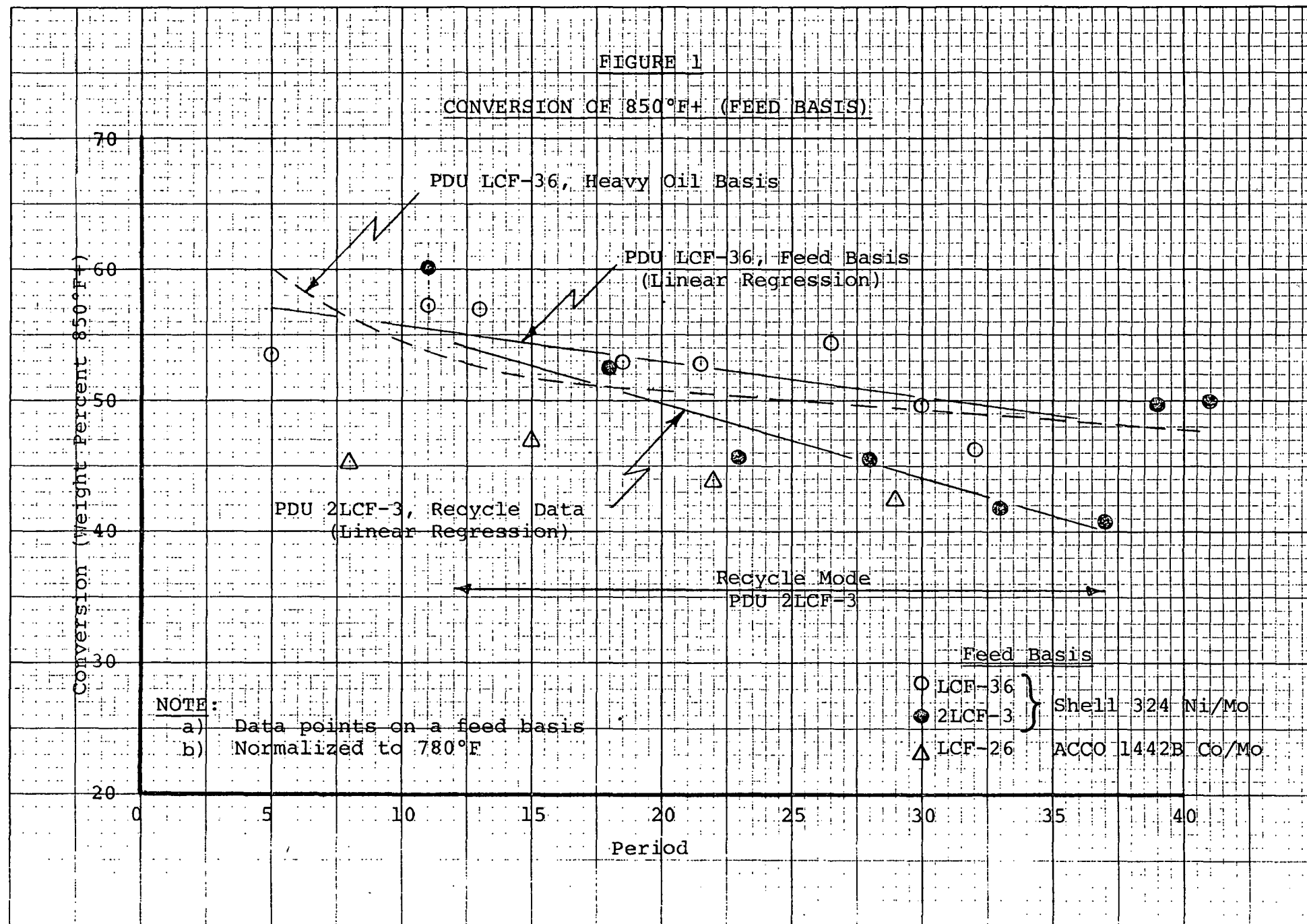


FIGURE 2  
NITROGEN IN LIQUID PRODUCT (FEED BASIS)

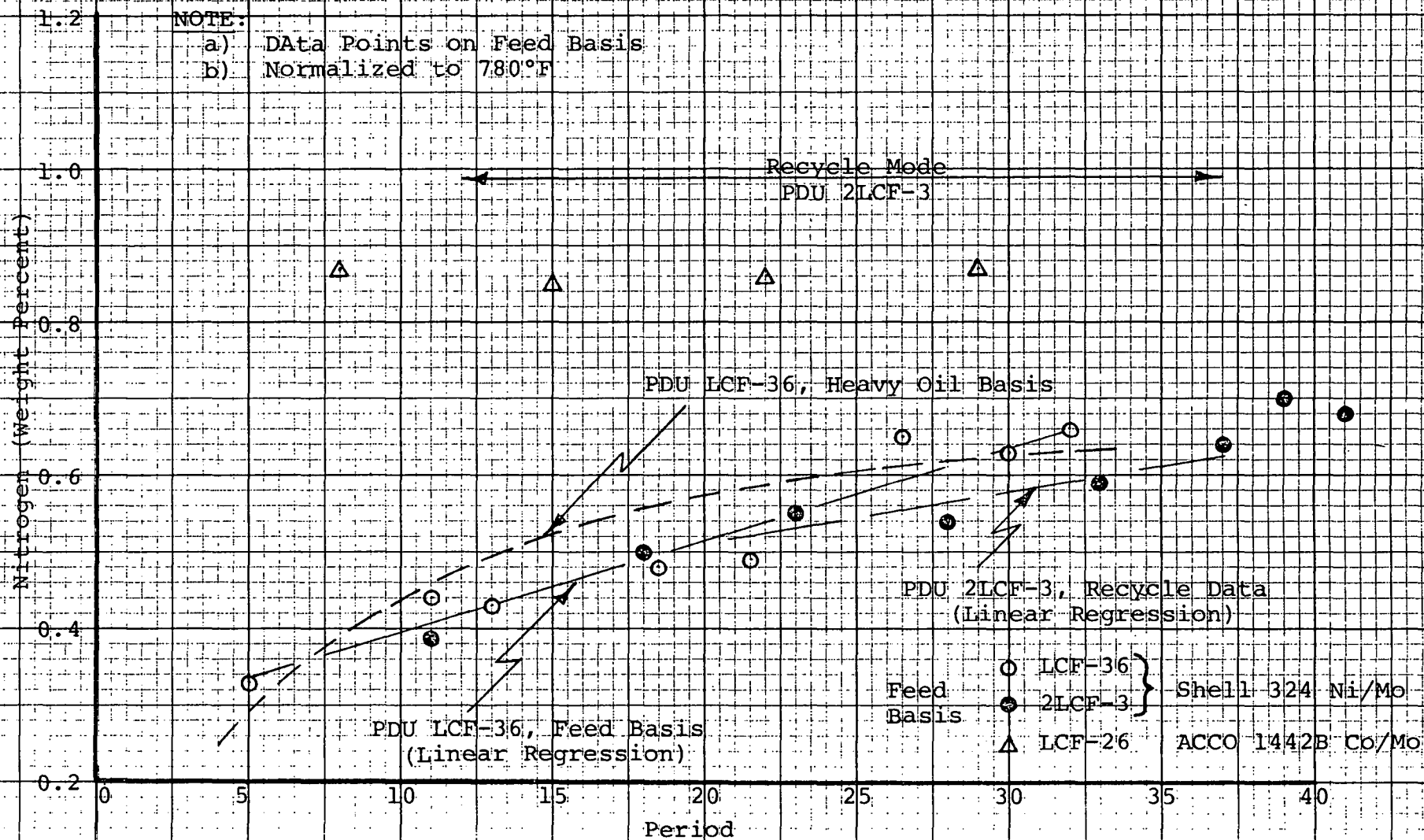


FIGURE 3

TWO-STEP LIQUEFACTION MATERIAL BALANCE

RECYCLE CASE

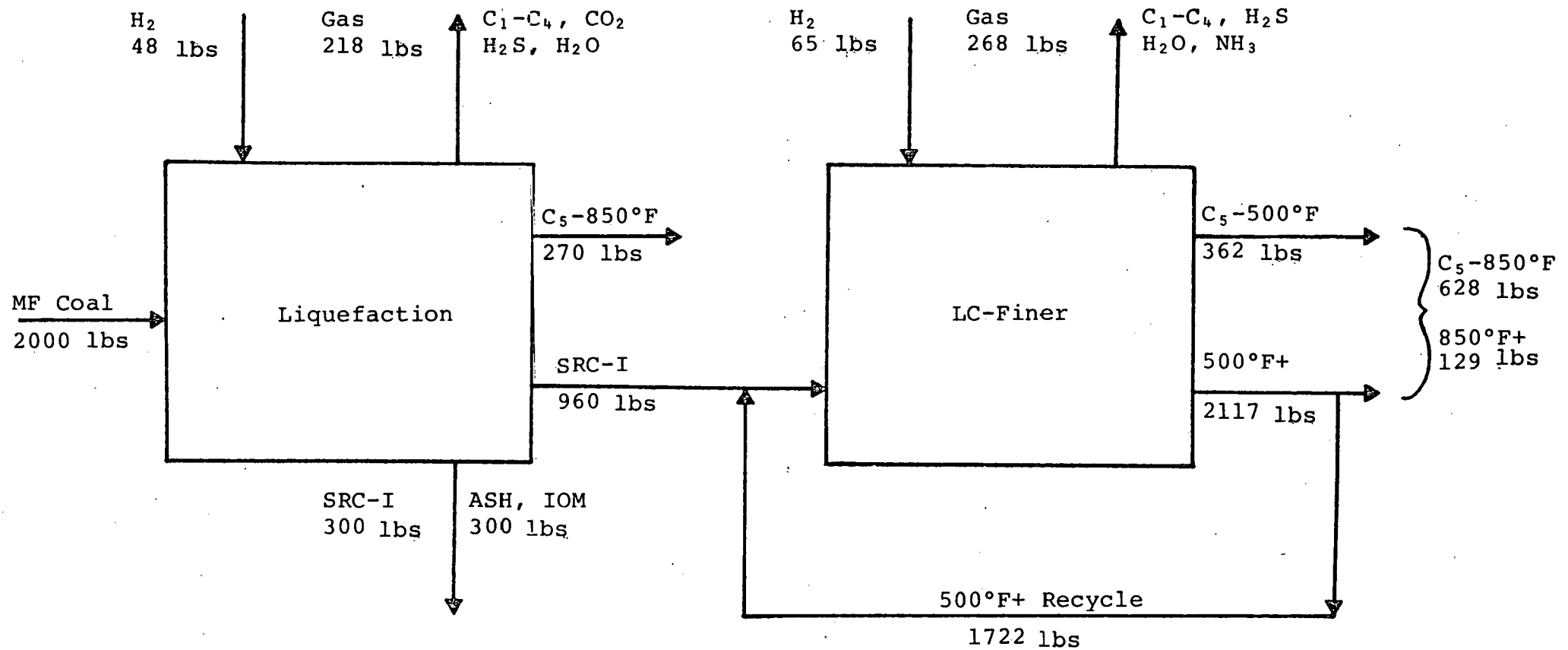


TABLE I

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. Recycle Pass No.	I -		II I		III II		IV III		V IV		VI V	
Dist. Fraction (b)	Feed	Product	Feed	Product	Feed	Product	Feed	Product	Feed	Product	Feed	Product (a)
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 <sub>2</sub> , Wt% (c)			1.09		1.10		1.13		1.12		1.14	
H <sub>2</sub> , 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		500-650°F		650-850°F		850°F+	
			Vol%	°API	Vol%	°API	Vol%	°API	Vol%	°API
4B,6B,7 8,9 10,11A,11B	I	I	9.4	25.3	38.5	10.4	31.5	1.1	20.6	-13.9
			8.05	24.9	37.7	9.2	31.4	0.8	22.4	-13.0
			10.2	23.9	40.9	8.7	28.5	-0.3	20.2	-14.9
13,14 15,16 17,18	II	II	10.7	24.2	31.9	12.9	33.5	3.5	24.0	-13.3
			11.6	23.9	39.1	11.7	18.4	1.8	21.1	-15.2
			11.6	23.4	37.6	11.6	29.7	1.7	21.2	-15.2
20,21 19,22,23	III	III	14.5	22.5	35.4	12.1	27.0	2.6	23.4	-14.2
			11.2	22.8	38.0	11.8	28.7	1.7	22.3	-16.0
24B,25 26,27 27,28	IV	IV	12.2	23.8	36.0	13.0	31.9	2.2	20.0	-17.0
			12.7	22.2	35.1	12.3	29.5	1.8	22.9	-17.2
			13.0	22.3	34.1	12.3	29.2	1.4	22.8	-17.8
29,30 31B,32,33	V	V	12.9	22.7	36.8	12.0	27.8	1.3	23.0	-17.8
			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  
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 <sub>0</sub> )	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 <sub>2</sub> O	3.59	2.02	1.49	1.67	1.94	2.74	3.39	2.80
H <sub>2</sub> S	0.44	0.36	0.33	0.37	0.32	0.36	0.41	0.40
NH <sub>3</sub>	1.07	0.77	0.79	0.92	0.8	0.88	0.93	0.73
CH <sub>4</sub>	1.22	1.49	1.20	1.66	1.73	1.81	1.99	1.09
C <sub>2</sub> H <sub>6</sub>	1.13	1.49	1.24	1.74	1.27	1.88	1.99	1.09
C <sub>3</sub> H <sub>8</sub>	1.18	1.76	1.52	2.11	1.72	2.35	2.17	1.06
C <sub>3</sub> H <sub>6</sub>	0.05	0.09	0.08	0.13	0.14	0.15	0.15	0.08
i-C <sub>4</sub> H <sub>10</sub>	0.1	0.15	0.14	0.19	0.12	0.22	0.17	0.09
n-C <sub>4</sub> H <sub>10</sub>	1.26	1.72	1.42	1.75	1.51	2.21	2.01	0.99
C <sub>4</sub> H <sub>8</sub>	0.01	0.02	0.02	0.04	0	0.05	0.02	0.01
C <sub>5</sub> -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 <sub>5</sub> -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 <sub>2</sub> Consumption (SCF/BBL)	2611	2074	1747	1895	1718	1923	2631	2133

TABLE V

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 <sub>0</sub> )		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 VI

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						390-850°F Wt% N
			390-500°F		500-650°F		650-850°F		
			Wt% Feed	%N	Wt% Feed	%N	Wt.% Feed	%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 VII

TWO-STEP LIQUEFACTION PRODUCT YIELDS  
AND SRC-I CONVERSION

LC-Finer Operation	Recycle			
	II	III	IV	V
Recycle Pass, PDU Run 2LCF-3				
Temperature, °F	790	800	800	810
<u>Combined Yields, Wt% of MF Coal</u>				
H <sub>2</sub> S	1.83	1.90	1.83	1.90
H <sub>2</sub> O	6.96	7.27	7.56	7.16
NH <sub>3</sub>	1.04	1.24	1.06	1.27
CO <sub>2</sub>	0.80	0.80	0.80	0.80
C <sub>1</sub>	2.96	3.66	3.69	3.91
C <sub>2</sub>	2.63	3.37	2.68	3.61
C <sub>3</sub>	2.98	3.94	3.38	4.36
C <sub>4</sub>	2.47	3.09	2.57	3.81
C <sub>5</sub> -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	<u>30.00</u>	<u>30.00</u>	<u>30.00</u>	<u>30.00</u>
Total	105.44	105.85	105.43	105.84
<u>Bbl/Ton MF Coal</u>				
C <sub>5</sub> -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 VIII

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.% <sup>1</sup>	Lbs.	Wt.% <sup>3</sup>	Lbs.	Lbs.
H <sub>2</sub>	-2.4	-48	-6.8 <sup>4</sup>	-64.8	-112.8
C <sub>1</sub>	1.4	28	4.5	43.1	71.1
C <sub>2</sub>	1.0	20	4.3	41.4	61.4
C <sub>3</sub>	0.9	18	5.7	55.3	73.3
C <sub>4</sub>	0.4	8	5.4	51.7	59.7
CO <sub>2</sub>	0.8	16	-	-	16.0
H <sub>2</sub> S	1.4	28	1.0	9.3	37.3
H <sub>2</sub> O	5.0	100	4.7	44.8	144.8
NH <sub>3</sub>	N.A.	-	2.4	22.7	22.7
C <sub>5</sub> -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 <sup>2</sup>	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.% (high)

## NOTES:

- <sup>1</sup> Values from Moschitto's paper<sup>3</sup>
- <sup>2</sup> 300 lbs. of SRC-I are assumed needed for removing the ash and insoluble organic material (IOM) leaving 960 lbs. for upgrading
- <sup>3</sup> Averaged values from 2LCF-3, recycle Passes II, III, IV, and V
- <sup>4</sup> Based on 960 lbs. fresh SRC-I feed