

295
12-9-81

(2)

De. 129

DOE/PC/30027-14
(DE82002154)

BINS-208
NTIS-25

MASTER

RECYCLE SLURRY OIL CHARACTERIZATION

Quarterly Report No. 3 for the Period April 1-June 30, 1981

By
F. P. Burke
R. A. Winschel

October 1981

Work Performed Under Contract No. AC22-80PC30027

Conoco Coal Development Company
Library, Pennsylvania



U. S. DEPARTMENT OF ENERGY

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.

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."

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A04
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts, (ERA)*; *Government Reports Announcements and Index (GRA and I)*; *Scientific and Technical Abstract Reports (STAR)*; and publication, NTIS-PR-360 available from (NTIS) at the above address.

RECYCLE SLURRY OIL CHARACTERIZATION

Quarterly Report No. 3

Period: April 1 through June 30, 1981

Prepared by

F. P. Burke
R. A. Winschel

Conoco Coal Development Company
Research Division
Library, Pennsylvania 15129

October, 1981

Prepared for the
United States Department of Energy
Under Contract No. DE-AC22-80PC30027

TABLE OF CONTENTS

| <u>Section</u> | | <u>Page No.</u> |
|----------------|--|-----------------|
| 1 | INTRODUCTION. | 1 |
| 2 | SUMMARY | 4 |
| | A. Recycle Oils From H-Coal PDU Run 10. | 4 |
| | B. Lummus Two-Stage Liquefaction (TSL) and LC-Fining Operations | 5 |
| 3 | EXPERIMENTAL. | 6 |
| 4 | RESULTS AND DISCUSSION. | 7 |
| | A. Recycle Oils From H-Coal PDU Run 10. | 7 |
| | B. Lummus TSL and LC-Fining Operations. | 14 |
| 5 | REFERENCES. | 52 |

LIST OF TABLES

| <u>Table No.</u> | | <u>Page No.</u> |
|------------------|--|-----------------|
| 4-1 | Proton Distributions of HO Soluble Resid. | 8 |
| 4-2 | Microautoclave Conversions With H-Coal Samples. | 9 |
| 4-3 | Concentration of Phenolic -OH by Derivatization Followed by ¹⁹ F-NMR, H-Coal PDU Run 10. | 13 |
| 4-4 | Short Contact Time Run Conditions | 15 |
| 4-5 | Component Distribution Short Contact Time Samples | 16 |
| 4-6 | Sample Distributions - Short Contact Time Pasting Solvents (PS) and Heavy Oil (HO) Products - Run SCT19 . . . | 17 |
| 4-7 | Solvent Fractionation of THF Soluble 270°C ⁺ /5 torr Resid Short Contact Time Samples. | 18 |
| 4-8 | Solubility Fractionations - Run SCT19 | 19 |
| 4-9 | Proton Distributions Short Contact Time Distillates (850°F ⁻). | 20 |
| 4-10 | Proton Distributions by ¹ H-NMR - Run SCT19 Pasting Solvent (PS) and Heavy Oil (HO) Distillates (850°F ⁻). . . . | 21 |
| 4-11 | Microautoclave Extractions of Indiana V Coal With SCT Distillates (850°F ⁻). | 23 |
| 4-12 | Correlation of Pasting Solvent Quality and Preasphaltene Content of HO Resid - Input Parameters and Correlation Constants | 26 |
| 4-13 | Comparison of Observed and Calculated Preasphaltenes in THF Soluble HO Resid. | 27 |
| 4-14 | Correlation of Pasting Solvent Composition With Heavy Oil Preasphaltenes - Correlation Constants. | 30 |
| 4-15 | Antisolvent Deasher (ASDA) Feeds, Overflows and Underflows, Component Distributions | 32 |
| 4-16 | Solubility Fractionations - Antisolvent Deasher Feeds, Overflows and Underflows, 850°F ⁺ THF Solubles | 33 |
| 4-17 | Antisolvent Deasher - Mass Balances of 850°F ⁺ Components - Forced Ash Balance. | 34 |
| 4-18 | Proton Distributions by ¹ H-NMR Antisolvent Deasher (ASDA) Distillates (850°F ⁻) | 35 |
| 4-19 | LC-Finer Run Conditions | 37 |
| 4-20 | Component Distribution LC-Finer Samples From Integrated TSL Operations. | 38 |
| 4-21 | Solvent Fractionation of THF Soluble 270°C ⁺ /5 torr Resid LC-Finer Samples From Integrated TSL Operations . . . | 39 |
| 4-22 | Proton Distributions LC-Finer Distillates (850°F ⁻) From Integrated TSL Operations | 40 |
| 4-23 | Microautoclave Conversions, LCF Distillates (850°F ⁻) From TSL Operations | 41 |
| 4-24 | LC-Finer Run Descriptions | 42 |
| 4-25 | Component Distribution of LC-Finer Samples - Operations With Wilsonville SRC Feed | 44 |

LIST OF TABLES (Cont'd.)

| <u>Table No.</u> | | <u>Page No.</u> |
|------------------|--|-----------------|
| 4-26 | Solvent Fractionation of THF Soluble 270°C ⁺ /5 torr (850°F ⁺ , atm) Resid of LC-Finer Samples - Operations With Wilsonville SRC Feed. | 45 |
| 4-27 | Proton Distributions of LC-Finer Distillates (850°F ⁻) Operations With Wilsonville SRC Feed | 46 |
| 4-28 | Feed and Total Liquid Product Analyses - LC-Finer Run 2LCF27 | 47 |
| 4-29 | Sample Distributions - Run 2LCF29. | 48 |
| 4-30 | LC-Finer Runs 2LCF27 and 2LCF29 - Hydrogenation Selectivities. | 49 |
| 4-31 | Proton Distributions by ¹ H-NMR, Feed and Product Distillates, (850°F ⁻) LC-Finer Run 2LCF27. | 50 |

LIST OF FIGURES

| <u>Figure No.</u> | | <u>Page No.</u> |
|-------------------|---|-----------------|
| 4-1 | Comparison of Calculated and Observed SCT Heavy Oil Product Resid Compositions: wt % Preasphaltenes in 850°F ⁺ THF Soluble Resid | 29 |

Section 1

INTRODUCTION

This is the third quarterly report under the subject contract reporting the results of analyses of recycle slurry oils in direct liquefaction processing. This work is similar in scope and intent to that recently completed under Contract DE-AC05-79ET-14503, "Development of the Correlation Between Slurry Oil Composition and Process Performance". That contract was devoted to the analyses of slurry recycle oils from the H-Coal process development unit operated by HRI at Trenton, New Jersey. In the current contract we expect that the majority of samples will come from the two-stage liquefaction bench scale unit operated by Lummus at New Brunswick, New Jersey. The Lummus unit consists of a short contact time coal liquefaction, followed by Lummus solvent deashing of the short residence time product. The deashed coal liquids are then upgraded in a Lummus-Cities Service LC-fining unit. The Lummus work is being conducted under DOE Contract DE-AC02-79ET-14804. DOE will also provide samples from other sources for analysis under this contract notably the H-Coal PDU and the H-Coal pilot plant in Catlettsburg, Kentucky. The work is divided into three tasks as described below:

Task 1 Analytical Evaluation

Under this task CCDC will review the operational flow schemes, existing analytical plans, and required analytical data resulting from operations of the Lummus two-stage liquefaction bench scale unit. CCDC will then develop an analytical plan for the conduct of analytical characterization on recycle slurry oils and other process oils based upon the need for a thorough process characterization. The evaluation plan will identify sampling requirements, sample gathering techniques, sampling containers, and shipment procedures. This task has been completed.

Task 2 Analytical Characterization

Samples received from the two-stage liquefaction process development unit or other samples specified by DOE will be subjected

to a series of analytical characterization tests. The procedures for analytical testing are based upon the work conducted under Contract DE-AC05-79ET-14503. The following characterization methods will be employed for some or all of the samples as deemed appropriate.

1. Distillation to quantitatively determine the distillate and resid fractions.
2. Tetrahydrofuran (THF) extraction, as appropriate, of the resid fraction to quantitatively determine the soluble residue and unconverted coal plus ash.
3. Combustion, as appropriate, of the THF insoluble residue to determine the unconverted coal and ash.
4. Microautoclave extractions of a standard batch of Old Ben #1 Mine Indiana V coal at 2/1 slurry oil/coal, 30 min, and 750°F, termed the "Equilibrium" (EQ) run, to provide a relative measure of the total hydrogen donor content of the slurry oil, and at 8/1 solvent/coal, and 10 min, termed the "Kinetic" (KIN) run, to measure the kinetic activity of the hydrogen donors, based upon THF solubles.
5. Proton nuclear magnetic resonance analysis of recycle slurry oils, process oils, and appropriate boiling range fractions thereof to determine the proton distribution (total, aromatic, aliphatic) and thus the donatable hydrogen.
6. Gas chromatographic/mass spectrometric (GC/MS) analysis of oil fractions to determine molecular composition.
7. Liquid chromatographic (LC) analyses, as appropriate, of the THF soluble residues to fractionate the sample by solubility class (LCF), chemical functionality (SESC), and molecular size (GPC).
8. ¹⁹F-nuclear magnetic resonance of fluorinated derivatives of distillate samples to measure the concentration of functional (-OH, -SH, -NH) components. These data will provide information on catalyst aging and selectivity.
9. Reverse phase liquid chromatography (RPLC) of distillate

samples to separate functional components and hydrocarbons of different degrees of alkylation.

We foresee the possibility that during the two-year duration of this project analytical methods other than those listed above may be deemed desirable. A limited number of new analytical methods may be employed during the course of this contract, if time and money allow.

Task 3 Data Interpretation and Reporting

Our intention in this work is not simply to provide analytical data, but to utilize the measurements of recycle slurry and process oil characteristics to provide assistance to DOE and to the personnel involved in DOE funded liquefaction projects for evaluating the relationship between coal liquid properties and process performance. Areas of process performance to be investigated include but are not limited to the following:

1. Changes in the slurry recycle and process oil stream compositions during start-up.
2. The manner in which the process reaches steady state operation during start-up.
3. The composition of the recycle and process oil streams during stable operation.
4. The changes in recycle oil composition in response to planned variations in process variables.
5. Changes in recycle oil compositions which cause or result from unplanned upsets in process operation.
6. Differences in recycle and process oil compositions as a function of process operating conditions.
7. Differences in recycle and process oil compositions as a function of process feed materials.

Section 2

SUMMARY

A. RECYCLE OILS FROM H-COAL PDU RUN 10

We received 69 samples of H-Coal PDU 10 recycle oils. All hydroclone overflow (HO) samples have been distilled, THF extracted and ashed. All HO soluble resids have been analyzed by solvent fractionation and the distillates by $^1\text{H-NMR}$. All clean oil tank (COT) samples have also been analyzed by $^1\text{H-NMR}$. Selected COT and HO distillate samples were analyzed by GC/MS, microautoclave (MAC) extractions and by fluorine derivatization followed by $^{19}\text{F-NMR}$. Selected HO soluble resids were analyzed by $^1\text{H-NMR}$. Planned experimental work is nearly finished. We conclude the following.

- Recycle distillates from PDU 10 (Wyodak, Syncrude) are extremely paraffinic, even able to form wax crystals at room temperature. This property along with their low concentration of hydroaromatics causes them to be poorer liquefaction media than distillates from PDU 5 (Illinois 6, Syncrude) or PDU 9 (Kentucky 11, Syncrude) in terms of both hydrogen donor strength and their ability to act as physical solvents. Their low concentrations of condensed aromatics suggests that additional hydrogenation of the distillates would not significantly improve these properties.
- The paraffin content of PDU 10 recycle distillates increased after day 20 of the run paralleling decreasing solvent quality as determined by $^1\text{H-NMR}$ and MAC.
- The declining aromaticity of the recycle distillates over the end of PDU 10 is not a result of increased hydrogenation but probably results from a decreasing removal of paraffins from the distillate recycle solvent.
- PDU 10 recycle distillates have a lower phenol content than those from PDU 5 or 9. Phenolic content increased gradually throughout the run, evidence of declining catalyst activity. The composition of the THF soluble resid is similar for Syncrude runs PDU 5, 9 and 10 regardless of feed coal.

B. LUMMUS TWO-STAGE LIQUEFACTION (TSL) AND LC-FINING OPERATIONS

We have received 116 Lummus SCT, ASDA, and LC-Fining samples as of 9/15/81. As applicable, all samples have been distilled, THF extracted, ashed and analyzed by $^1\text{H-NMR}$ and solvent fractionation. Various other analyses were performed on some of the samples. We conclude the following.

- The solvent quality of the distillate portion of the TSL pasting solvent is very good. The microautoclave tests used to characterize SRC-I and H-Coal recycle distillates are not applicable to the Lummus solvents because all of them gave essentially the maximum conversion ($\sim 90\%$) achievable with the Indiana V coal used in the test.
- The first stage (short contact time or SCT unit) product quality, as preasphaltene content, is unaffected by space velocity over the range 115-190 lb/hr/ft³. This might be expected in the largely thermal first stage reaction. Product preasphaltene content was positively correlated to feed preasphaltene content, feed distillate content and feed distillate aromaticity while it was negatively correlated to the ratios of condensed/uncondensed aromatics and cyclic/alkyl aliphatics in the feed distillate. It may be possible to use these correlations to optimize SCT operations.
- There appears to be no chemical alteration or physical fractionation of the 850°F⁺ components during their transit through the antisolvent deashing (ASDA) unit. However, additional work is planned to determine if any selective physical fractionation is performed on the 850°F⁺ solubles.
- The coal conversion to THF solubles obtained in the process does not all occur in the first stage. Additional conversion of THF insolubles to THF solubles takes place in the LC-Finer.
- In the LC-Finer operations with Wilsonville SRC feeds, low severity operation maintained product selectivity longer than high severity operation. Also, regeneration of the deactivated catalyst restored preasphaltene conversion and distillate hydrogenation but selectivity to desired products was lost.

Section 3

EXPERIMENTAL

Experimental descriptions were given in the first two quarterly reports.

Section 4

RESULTS AND DISCUSSION

A. RECYCLE OILS FROM H-COAL PDU RUN 10

Proton Distribution of the HO Soluble Resid. Proton Distributions were determined on selected THF soluble, 975°F⁺ HO resids and are given in Table 4-1. The aromaticity rises throughout the run as the concentrations of beta and gamma protons decrease. This is the opposite of the behavior of the distillates after day 15 though the magnitude of the change is much less.

Below are average proton distributions for the HO soluble resids of PDU Runs 5, 8, 9 and 10.

| <u>PDU Run</u> | <u>Run Days</u> | <u>Feed Coal</u> | <u>Mode</u> | <u>Aromatic</u> | <u>Alpha</u> | <u>Beta</u> | <u>Gamma</u> |
|----------------|--------------------|------------------|--------------|-----------------|--------------|-------------|--------------|
| 5 | 12-30 | Illinois 6 | Syncrude | 46.5 | 30.2 | 18.5 | 4.8 |
| 8 | 11-15 | Illinois 6 | Fuel Oil | 44.1 | 31.3 | 19.9 | 4.7 |
| 8 | 17-23 | Illinois 6 | Intermediate | 42.0 | 32.4 | 20.7 | 4.9 |
| 9 | 12-30 | Kentucky 11 | Syncrude | 43.7 | 32.1 | 19.9 | 4.3 |
| 10 | 17,19,26, 34,40 | Wyodak | Syncrude | 45.7 | 28.2 | 21.3 | 4.9 |

There is very little difference. ¹H-NMR spectra from the various runs appeared nearly identical except that those from PDU 10 (Wyodak) had a noticeably larger paraffin -CH₂- peak that results in a somewhat larger percentage of beta protons.

Solvent fractionation analyses previously showed the HO soluble resids from the three Syncrude runs to be very similar regardless of feed coal.

Microautoclave Extractions. Selected PDU 10 recycle distillates were used to extract Indiana V Old Ben #1 mine coal in the microautoclave (MAC). The results are reported in Table 4-2 along with average values from PDU Runs 5, 8 and 9. The values for PDU Runs 5, 8 and 9 are averages of three samples from the beginning, middle and end of the three PDU runs. These MAC runs were all made with the same batch of Indiana V coal. This is not the same coal used for the microautoclave runs with the Lummus TSL samples.

Table 4-1

Proton Distributions of HO Soluble Resid

| <u>Run Day</u> | <u>Aromatics</u> | <u>Alpha</u> | <u>Beta</u> | <u>Gamma</u> |
|--------------------|------------------|--------------|-------------|--------------|
| 5 | 41.1 | 27.5 | 25.3 | 6.2 |
| 9 | 43.0 | 26.7 | 24.1 | 5.4 |
| 14 | 45.1 | 28.0 | 22.2 | 4.7 |
| 17 | 44.5 | 28.1 | 22.4 | 5.0 |
| 19 | 44.6 | 28.4 | 21.5 | 5.5 |
| 26 | 45.1 | 28.4 | 21.9 | 4.6 |
| 34 | 47.1 | 28.1 | 20.3 | 4.5 |
| 40 | 47.3 | 27.6 | 20.5 | 4.7 |

Table 4-2

Microautoclave Conversions With
H-Coal Samples

| <u>Sample Type</u> | <u>Run Day</u> | <u>Conversion to THF Solubles, wt % MAF Coal</u> | |
|--------------------|----------------|--|-----------|
| | | <u>KIN</u> | <u>EQ</u> |
| HO Distillate ↓ | 5 | 68.7 | 58.4 |
| | 9 | 66.4 | 57.5 |
| | 14 | 65.3 | 53.4 |
| | 17 | 71.5 | 60.4 |
| | 19 | 72.6 | 69.8 |
| | 26 | 68.8 | 61.2 |
| | 34 | 70.7 | 56.8 |
| | 40 | 66.4 | 56.6 |
| | Avg. PDU 5 | 80.1 | 79.4 |
| | Avg. PDU 8 | 75.6 | 73.4 |
| Avg. PDU 9 | 78.9 | 81.1 | |
| COT ↓ | 5 | 67.5 | 53.8 |
| | 9 | 60.1 | 51.3 |
| | 14 | 61.3 | 49.0 |
| | 17 | 67.6 | 58.2 |
| | 19 | 70.8 | 63.0 |
| | 26 | 57.3 | 56.0 |
| | 34 | 64.0 | 51.9 |
| | 40 | 63.9 | 49.1 |
| | Avg. PDU 5 | 74.7 | 74.5 |
| | Avg. PDU 8 | 71.2 | 67.3 |
| Avg. PDU 9 | 74.0 | 77.9 | |

KIN = 750°F, 10 min, 8/1 S/C.

EQ = 750°F, 30 min, 2/1 S/C.

Both PDU 10 recycle distillate streams (COT and HO distillate) deteriorated in quality (conversion) between days 5 and 14 at both the KIN and EQ conditions. This could be predicted from the $^1\text{H-NMR}$ data^(1,2) because the distillates became both more aromatic and more paraffinic while becoming less hydroaromatic over that period. This is likely an effect of catalyst deactivation.

At restart on day 15, conditions were changed. The space velocity was lowered and catalyst addition rate was increased. Also, at that time, the used catalyst was washed and ca. 20% was replaced by fresh sulfided catalyst. Hydrogenated anthracene oil was used as re-startup oil. These changes produced good quality recycle distillates for day 17. On day 18, make-up hydrogenated anthracene was added resulting in good quality recycle distillates on day 19. For those samples tested in the microautoclave, liquefaction quality declined after day 19 for both recycle streams as measured by both microautoclave conditions. The COT-26 sample gave an unusually low conversion at the KIN condition and may be a bad data point. After day 19, both distillate streams became less aromatic and more paraffinic. The increasing paraffinic nature of the distillates reduced solvent quality as the run progressed.

Cursory inspections of the GC/MS of the distillates confirms that a major increase in the concentration of normal, branched and cyclic paraffins occurred from day 19 to 40. These compounds were certainly not created by ring opening hydrogenation reactions of aromatics but are primary coal products produced in much higher quantity from Wyodak coal than either Illinois 6 or Kentucky 11 coals. Paraffins may be removed from the distillate stream by cracking to light gases. In PDU 10, the yield of $\text{C}_1 \times \text{C}_7$ hydrocarbon gases decreased from day 19 to 40 paralleling the increased concentration of paraffins in the recycle distillate. Therefore, we take the increasing paraffinic nature of the distillates to be a result of a decreasing activity toward paraffin removal and not of increasing hydrogenation activity.

The average MAC conversions from PDU 5 and 9 distillates are comparable to one another as predicted by $^1\text{H-NMR}$ and are considerably higher than MAC conversions from PDU 8 distillates which in

turn are higher than MAC conversions from PDU 10 distillates. We conclude that in Syncrude mode operations, the bituminous coals produce better quality recycle distillates than Wyodak coal, in part because of their less paraffinic nature.

There were more differences than just feed coal between PDU 5, 9 and 10; notably catalyst, catalyst addition rate and space velocity but we believe these to be less of a factor (over this range) than the feed coal was toward distillate composition and quality.

For all four PDU runs we inspected, the HO distillates gave better MAC performance than the corresponding COT sample. This may be a result of the higher boiling point distribution of the HO distillates.

HRI ran daily COT samples in their microautoclave at the KIN conditions⁽³⁾. In general, their conversion values were close to ours but ca. 2% lower. The difference may be due to procedural differences or a difference in the activity of the feed coal. Our study used "A" period samples, while HRI ran MAC experiments on "B" period samples. In this study we found the EQ conditions to be more sensitive to solvent properties.

Concentration of Phenolic -OH. The concentration of phenolic -OH in the COT and several HO distillate samples were determined by derivatization with α,α,α -trifluoroacetyl chloride followed by ^{19}F -NMR. This method will also detect alcohols but none were observed. The values are given in Table 4-3. Over the first section of PDU 10, from days 4 to 10, the concentration of phenolic -OH in the COT increased from 0.4 to 0.6 meq/g. This can be accounted for by the increase in space velocity from ca. 28 to ca. 32 lbs/hr/ft³ and by catalyst deactivation that probably occurred rapidly during the beginning of the run resulting in less hydrodeoxygenation.

Space velocities for the period from day 11 to 15 averaged only ca. 20 lbs/hr/ft³. Small amounts of hydrogenated anthracene oil were added to the system on days 13 and 14. These two factors are probably responsible for the reduction in phenolic -OH concentration over that period.

Hydrogenated anthracene oil was used as the start-up oil during the re-start on day 15 which substantially reduced the phenolic concentration on days immediately following. Contributing factors were that the used catalyst was washed with fuel oil, ca. 10% fresh sulfided catalyst was added to the reactor inventory, catalyst addition rate was increased and space velocity was lowered. Make-up hydrogenated anthracene oil was added to the system on days 18 and 22 causing temporary reductions in the phenolic -OH concentration immediately thereafter.

From day 16 to 34, the phenolic concentration increased in a fairly smooth manner which may be a result of catalyst deactivation. Days 39 and 40 have low concentrations of phenols, undoubtedly due to the accidental dilution of the COT in the PDU by #2 fuel oil. Day 35 has an unexplainably low value and days 36 and 37 both have relatively low values.

Average COT phenolic concentrations are compared below for Syncrude runs PDU 5, 9 and 10.

| <u>PDU Run</u> | <u>Run Days</u> | <u>Feed Coal</u> | <u>Phenolic -OH Concentration, meq/g</u> |
|----------------|-----------------|------------------|--|
| 5 | 12-31 | Illinois 6 | 0.62 |
| 9 | 12-30 | Kentucky 11 | 0.64 |
| 10 | 16-40 | Wyodak | 0.38 |

PDU 10 COT samples are considerably less phenolic than those from the bituminous coals. Other work⁽⁴⁾ has shown Wyodak Syncrude naphtha to contain less oxygen than that from Illinois 6 coal which has less than that from Kentucky 11 coal.

The phenolic concentration in the HO distillate is somewhat lower than that of the corresponding COT sample. This was also seen for PDU 5 and 9. The COT samples have a lower boiling point distribution so this may suggest that the majority of phenols present are low molecular weight.

Table 4-3

Concentration of Phenolic -OH by
Derivatization Followed by ^{19}F -NMR
 H-Coal PDU Run 10

| <u>Run Day</u> | <u>Concentration of Phenolic -OH, meq/g</u> | |
|----------------|---|----------------------|
| | <u>COT</u> | <u>HO Distillate</u> |
| 4 | 0.39 | |
| 5 | 0.39 | |
| 6 | 0.45 | |
| 7 | 0.42 | |
| 8 | 0.55 | |
| 9 | 0.53 | |
| 10 | 0.61 | |
| 11 | -- | |
| 12 | 0.51 | |
| 13 | -- | |
| 14 | 0.40 | |
| 15 | -- | |
| 16 | 0.25 | |
| 17 | 0.36 | |
| 18 | 0.36 | |
| 19 | 0.27 | |
| 20 | 0.27 | |
| 21 | 0.36 | |
| 22 | 0.33 | |
| 23 | 0.30 | |
| 24 | 0.38 | |
| 25 | 0.39 | |
| 26 | 0.42 | 0.34 |
| 27 | 0.39 | |
| 28 | 0.46 | |
| 29 | 0.44 | |
| 30 | 0.40 | 0.39 |
| 31 | 0.41 | |
| 32 | 0.45 | |
| 33 | 0.48 | |
| 34 | 0.46 | 0.34 |
| 35 | 0.33 | |
| 36 | 0.42 | |
| 37 | 0.41 | |
| 38 | -- | |
| 39 | 0.39 | |
| 40 | 0.40 | 0.36 |

B. LUMMUS TSL AND LC-FINING OPERATIONS

1. Short Contact Time Liquefaction

The SCT unit is the first stage of the Lummus TSL process. Pasting solvents (PS), consisting of distillate, soluble and insoluble resid, and ash, are used to slurry coal which is then liquefied in a tubular reactor at residence times of 5-10 minutes. We have received samples from twenty SCT run periods; the operating conditions are given in Table 4-4. While space velocity was varied considerably, the other run conditions, except for pasting solvent composition, have been essentially invariant. For four of the runs (12, 13, 14-1 and 19-5) we received feed slurry instead of PS samples. Because the presence of the feed coal interferes with our analytical procedures these run periods are not included in our data interpretations, and most of the discussion below concerns the remaining 16 run periods.

The component distributions of the SCT samples are given in Tables 4-5 and 4-6. The "mass balance" indicates the recovery of the sample through the distillation which comprises the first part of the analytical procedure (cf. Section 3), and averages around 99%. PS distillate contents range from 46% to 78%. Substantial quantities of IOM and ash are also present in some of the PS samples.

The THF soluble resids (850°F⁺) were analyzed for oils (hexane soluble), asphaltenes (benzene soluble/hexane insoluble), and preasphaltenes (pyridine soluble/benzene insoluble). These data are given in Tables 4-7 and 4-8. Again, the PS show substantial variations, with preasphaltenes (for example) ranging from 16% to 40% of the THF soluble resid. Heavy oil (HO) products also show significant variations, with preasphaltenes ranging from 38% to 60%.

The distillate fractions were analyzed by ¹H-NMR to give the proton distributions shown in Tables 4-9 and 4-10. Since the PS are composited from LC-Finer products, a wide range of hydrogenation is possible, and is evident in these data.

Table 4-4

Short Contact Time Run Conditions

| <u>Run No., Period, Date</u> | <u>Coal Space Rate lb/hr-ft³</u> | <u>Concentration, % MF Coal</u> | <u>Preheater Outlet Temperature, °F</u> | <u>Preheater H₂ Pressure, psig</u> | |
|----------------------------------|---|-------------------------------------|---|---|---------------|
| | | | | <u>Inlet</u> | <u>Outlet</u> |
| SCT12-6-11080 | 102 | 36.0 | 861 | 2440 | 2430 |
| SCT13-5-11180 | 210 | 36.0 | 864 | 2500 | 2480 |
| SCT14-1-11280 | 126 | 36.0 | 862 | 2430 | 2420 |
| 21280 | 205 | 37.8 | 864 | 2450 | 2430 |
| 31280 | 184 | 36.0 | 865 | 2450 | 2430 |
| 41280 | 174 | 36.0 | 863 | 2430 | 2420 |
| 51280 | 133 | 36.0 | 865 | 2450 | 2440 |
| SCT15- -1181 | 116 | 36.0 | 865 | 2480 | 2470 |
| 2180 | 116 | 36.0 | 864 | 2480 | 2470 |
| 3180 | 145 | 36.0 | 863 | 2470 | 2460 |
| SCT16- -1281 | 118 | 36.0 | 865 | 2470 | 2460 |
| 2281 | 141 | 36.0 | 861 | 2470 | 2450 |
| 4-1381 | 180 | 36.0 | 867 | 2430 | 2410 |
| 5-2381 | 191 | 36.0 | 861 | 2450 | 2430 |
| 6-3381 | 185 | 36.0 | 860 | 2450 | 2430 |
| SCT19-1 | 179 | 36.0 | 866 | 2480 | 2440 |
| 2 | 147 | 36.0 | 860 | 2520 | 2460 |
| 4 | 175 | 36.0 | 865 | 2540 | 2490 |
| 5 | 176 | 36.0 | 861 | 2450 | 2390 |
| 7 | 170 | 36.0 | 863 | 2460 | 2390 |

Table 4-5

Component Distribution Short Contact Time Samples

| <u>Sample</u> | <u>270°C⁻/5 torr Distillate</u> | <u>THF Soluble Resid</u> | <u>IOM</u> | <u>Oxidized Ash</u> | <u>Mass Balance</u> |
|---------------|--|----------------------------------|------------|-------------------------|-------------------------|
| SCT12-6F11080 | 42.0 | 17.1 | 32.9 | 5.6 | 97.7 |
| 6H011080 | 45.6 | 42.7 | 4.9 | 5.4 | 98.6 |
| SCT13-5F11180 | 34.3 | 39.7 | 21.2 | 4.2 | 99.3 |
| 5H011180 | 41.7 | 47.7 | 5.3 | 4.8 | 99.6 |
| SCT14-1F11280 | 37.0 | 33.3 | 23.6 | 4.3 | 98.2 |
| PE21280 | 61.6 | 35.2 | 1.4 | 1.2 | 99.4 |
| PS31280 | 57.1 | 38.6 | 1.6 | 2.3 | 99.6 |
| PS41280 | 55.5 | 38.2 | 2.7 | 2.5 | 99.0 |
| PS51280 | 59.7 | 35.1 | 2.4 | 2.3 | 99.5 |
| 1H011280 | 41.0 | 47.8 | 6.1 | 4.7 | 99.6 |
| HO21280 | 43.4 | 45.5 | 5.8 | 4.8 | 99.5 |
| HO31280 | 40.6 | 47.5 | 6.0 | 5.0 | 99.1 |
| HO41280 | 38.8 | 48.4 | 5.9 | 5.7 | 98.7 |
| HO51280 | 37.1 | 50.4 | 6.2 | 5.7 | 99.3 |
| SCT15-PS1181 | 74.6 | 22.4 | 1.0 | 0.6 | 98.5 |
| PS2181 | 46.6 | 48.1 | 2.1 | 1.5 | 98.3 |
| PS3181 | 72.6 | 22.6 | 2.5 | 0.4 | 98.2 |
| H01181 | 46.0 | 38.3 | 5.3 | 5.1 | 94.7 |
| H02181 | 51.8 | 35.6 | 5.4 | 4.6 | 97.5 |
| H03181 | 47.7 | 40.7 | 6.4 | 4.5 | 99.3 |
| SCT16-PS1281 | 60.6 | 34.1 | 2.6 | 2.2 | 99.4 |
| PS2281 | 61.2 | 33.1 | 2.7 | 2.2 | 99.3 |
| 4PS1381 | 71.3 | 23.8 | 2.7 | 1.3 | 99.1 |
| 5PS2381 | 68.8 | 26.5 | 2.6 | 1.2 | 99.1 |
| 6PS3381 | 70.1 | 24.6 | 3.2 | 1.3 | 99.2 |
| H01281 | 47.0 | 38.7 | 7.1 | 5.0 | 97.8 |
| H02281 | 42.1 | 44.8 | 7.0 | 5.2 | 99.2 |
| 4H01381 | 41.1 | 44.9 | 7.5 | 5.5 | 99.0 |
| 5H02381 | 46.0 | 39.8 | 11.5 | 1.4 | 98.7 |
| 6H03381 | 47.5 | 39.2 | 9.4 | 3.0 | 99.1 |

Table 4-6

Sample Distributions - Short Contact Time Pasting
Solvents (PS) and Heavy Oil (HO) Products - Run SCT19

| <u>Period</u> | | <u>Distillate</u> | <u>THF Soluble Resid</u> | <u>IOM</u> | <u>Ash</u> |
|---------------|----|-------------------|------------------------------|------------|------------|
| 1 | PS | 57.7 | 36.7 | 3.0 | 2.0 |
| | HO | 49.0 | 41.0 | 3.5 | 5.7 |
| 2 | PS | 75.4 | 22.4 | 1.0 | 0.6 |
| | HO | 37.5 | 50.3 | 6.2 | 5.6 |
| 4 | PS | 71.1 | 25.4 | 1.8 | 0.7 |
| | HO | 42.6 | 47.7 | 4.1 | 4.6 |
| 5 | PS | -- | -- | -- | -- |
| | HO | 42.7 | 48.3 | 4.1 | 4.5 |
| 7 | PS | 78.5 | 20.1 | 0.6 | 0.1 |
| | HO | 51.1 | 40.5 | 3.7 | 4.3 |

Operating Conditions - Run SCT19

| <u>Period</u> | <u>Space Rate</u> | <u>Coal Concentration</u> | <u>Outlet Temperature</u> | <u>Total Pressure Inlet</u> | <u>Outlet</u> |
|---------------|-------------------|-------------------------------|-------------------------------|---------------------------------|---------------|
| 1 | 179 | 36% | 866 | 2480 | 2440 |
| 2 | 147 | ↓ | 860 | 2520 | 2460 |
| 4 | 175 | | 865 | 2540 | 2490 |
| 5 | 176 | | 861 | 2450 | 2390 |
| 7 | 170 | | 863 | 2460 | 2390 |

Table 4-7

Solvent Fractionation of THF Soluble 270°C⁺/5 torr
Resid Short Contact Time Samples

| Sample | Wt % of THF Soluble Resid | | |
|---------------|---------------------------|-------------|----------------|
| | Oils | Asphaltenes | Preasphaltenes |
| SCT12-6F11080 | 38.5 | 19.7 | 41.8 |
| 6HO11080 | 35.3 | 27.0 | 37.7 |
| SCT13-5F11180 | 51.6 | 19.0 | 29.4 |
| 5HO11180 | 32.6 | 24.6 | 42.3 |
| SCT14-1F11280 | 42.1 | 22.4 | 35.5 |
| PS21280 | 57.4 | 20.4 | 22.2 |
| PS31280 | 61.0 | 23.0 | 16.0 |
| PS41280 | 50.5 | 19.5 | 30.0 |
| PS51280 | 53.7 | 20.7 | 25.6 |
| 1HO11280 | 31.5 | 27.9 | 40.6 |
| HO21280 | 35.1 | 26.5 | 38.4 |
| HO31280 | 33.6 | 27.4 | 39.0 |
| HO41280 | 30.0 | 20.2 | 49.8 |
| HO51280 | 29.9 | 24.6 | 45.5 |
| SCT15-PS1181 | 50.4 | 21.4 | 28.2 |
| PS2181 | 41.6 | 28.4 | 30.0 |
| PS3181 | 42.4 | 23.5 | 34.1 |
| HO1181 | 30.3 | 26.8 | 42.9 |
| HO2181 | 28.7 | 22.7 | 48.6 |
| HO3181 | 25.0 | 21.0 | 54.0 |
| SCT16-PS1281 | 41.1 | 22.2 | 36.7 |
| PS2281 | 40.2 | 22.0 | 37.8 |
| 4PS1381 | 39.0 | 22.4 | 38.6 |
| 5PS2381 | 40.4 | 21.6 | 38.0 |
| 6PS3381 | 38.4 | 21.3 | 40.3 |
| HO1281 | 26.3 | 23.9 | 49.8 |
| HO2281 | 26.9 | 23.6 | 49.5 |
| 4HO1381 | 28.5 | 24.6 | 46.9 |
| 5HO2381 | 25.8 | 19.4 | 54.8 |
| 6HO3381 | 23.2 | 17.0 | 59.8 |

Table 4-8

Solubility Fractionations - Run SCT19

| <u>Period</u> | <u>Sample</u> | <u>Wt % 850°F⁺, THF Solubles</u> | | |
|---------------|---------------|---|--------------------|-----------------------|
| | | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> |
| 1 | PS | 38.2 | 25.9 | 35.9 |
| | HO | 34.3 | 22.9 | 42.8 |
| 2 | PS | 49.1 | 22.4 | 28.5 |
| | HO | 32.9 | 24.4 | 42.7 |
| 4 | PS | 46.5 | 21.2 | 32.3 |
| | HO | 29.1 | 24.0 | 46.9 |
| 5 | PS | 40.3 | 19.6 | 40.1 |
| | HO | 31.9 | 22.9 | 45.2 |
| 7 | PS | 57.4 | 21.8 | 21.5 |
| | HO | 29.5 | 22.5 | 48.0 |

Table 4-9

Proton Distributions
Short Contact Time Distillates (850°F⁻)

| <u>Sample</u> | <u>Condensed Aromatics</u> | <u>Uncondensed Aromatics</u> | <u>Cyclic α</u> | <u>Alkyl α</u> | <u>Cyclic β</u> | <u>Alkyl β</u> | <u>γ</u> |
|---------------|----------------------------|------------------------------|-----------------|----------------|-----------------|----------------|----------|
| SCT12-6F11080 | 15.7 | 7.5 | 15.6 | 8.8 | 23.5 | 18.0 | 11.0 |
| 6HO11080 | 21.8 | 7.5 | 14.6 | 8.6 | 19.8 | 16.9 | 10.7 |
| SCT13-5F11180 | 22.8 | 7.7 | 18.2 | 10.3 | 19.7 | 13.8 | 7.5 |
| 5HO11180 | 29.2 | 8.2 | 14.8 | 9.5 | 15.8 | 13.8 | 8.6 |
| SCT14-1F11280 | 25.2 | 6.6 | 16.3 | 10.0 | 18.9 | 14.7 | 8.3 |
| PS21280 | 18.8 | 7.4 | 16.0 | 8.8 | 21.8 | 17.0 | 10.3 |
| PS31280 | 10.6 | 8.1 | 15.5 | 9.7 | 24.2 | 19.3 | 12.6 |
| PS41280 | 10.0 | 8.9 | 15.4 | 10.0 | 24.0 | 19.2 | 12.6 |
| F351280 | 9.4 | 7.1 | 16.1 | 9.6 | 25.7 | 20.0 | 12.2 |
| 1HO11280 | 28.9 | 8.3 | 14.4 | 10.2 | 14.9 | 14.3 | 9.0 |
| HO21280 | 28.0 | 6.3 | 14.1 | 10.5 | 16.3 | 15.0 | 9.8 |
| HO31280 | 25.0 | 6.8 | 15.3 | 9.3 | 17.6 | 16.0 | 9.9 |
| HO41280 | 18.2 | 7.3 | 15.3 | 10.0 | 20.1 | 17.7 | 11.4 |
| HO51280 | 16.8 | 7.1 | 15.3 | 10.0 | 21.2 | 18.1 | 11.5 |
| SCT15-PS1181 | 19.6 | 8.7 | 15.9 | 8.1 | 20.8 | 16.3 | 10.6 |
| PS2181 | 18.2 | 7.1 | 15.5 | 8.4 | 21.4 | 17.4 | 11.9 |
| PS3181 | 29.1 | 8.3 | 13.4 | 10.2 | 15.8 | 13.3 | 9.8 |
| HO1181 | 22.6 | 6.9 | 15.8 | 8.8 | 19.2 | 16.1 | 10.6 |
| HO2181 | 23.4 | 7.4 | 15.0 | 8.0 | 18.6 | 15.8 | 11.7 |
| HO3181 | 30.6 | 7.5 | 14.0 | 8.3 | 14.7 | 14.4 | 10.5 |
| SCT16-PS1281 | 14.8 | 5.5 | 15.7 | 8.3 | 24.5 | 19.0 | 12.2 |
| PS2281 | 13.9 | 5.8 | 14.4 | 8.5 | 23.3 | 20.4 | 13.7 |
| 4PS1381 | 17.8 | 6.5 | 13.6 | 10.1 | 20.4 | 19.4 | 12.2 |
| 5PS2381 | 18.1 | 7.1 | 13.6 | 9.8 | 20.0 | 18.7 | 12.8 |
| 6PS3381 | 15.2 | 8.7 | 12.8 | 10.2 | 19.4 | 19.6 | 14.2 |
| HO1281 | 19.3 | 6.9 | 11.9 | 11.1 | 11.7 | 22.4 | 16.8 |
| HO2281 | 19.7 | 6.4 | 13.1 | 9.4 | 19.4 | 19.1 | 12.9 |
| 4HO1381 | 21.5 | 6.8 | 13.1 | 9.4 | 18.0 | 18.5 | 12.8 |
| 5HO2381 | 26.1 | 6.3 | 14.1 | 7.6 | 18.0 | 16.6 | 11.3 |
| 6HO3381 | 25.5 | 6.6 | 13.3 | 7.6 | 18.9 | 16.6 | 11.6 |

Table 4-10

Proton Distributions by $^1\text{H-NMR}$ - SCT Run 19
Pasting Solvent (PS) and Heavy Oil (HO) Distillates (850°F⁻)

| <u>Period</u> | <u>Sample</u> | <u>Condensed Aromatics</u> | <u>Uncondensed Aromatics</u> | <u>Cyclic α</u> | <u>Alkyl α</u> | <u>Cyclic β</u> | <u>Alkyl β</u> | <u>γ</u> |
|---------------|---------------|----------------------------|------------------------------|-----------------------------------|----------------------------------|----------------------------------|---------------------------------|----------------------------|
| 1 | PS | 20.1 | 6.3 | 17.1 | 8.0 | 22.5 | 16.2 | 9.8 |
| | HO | 27.9 | 8.7 | 16.2 | 8.5 | 18.0 | 12.2 | 8.2 |
| 2 | PS | 15.4 | 8.2 | 17.9 | 8.4 | 24.3 | 15.6 | 10.2 |
| | HO | 21.5 | 8.5 | 14.8 | 8.3 | 19.5 | 16.2 | 11.3 |
| 4 | PS | 16.1 | 7.4 | 18.3 | 8.5 | 24.5 | 15.9 | 9.3 |
| | HO | 20.2 | 7.5 | 17.2 | 8.7 | 21.3 | 15.3 | 9.7 |
| 5 | PS | -- | -- | -- | -- | -- | -- | -- |
| | HO | 24.6 | 7.8 | 16.7 | 8.6 | 18.6 | 14.3 | 9.6 |
| 7 | PS | 27.1 | 7.5 | 16.4 | 8.4 | 18.5 | 13.4 | 8.7 |
| | HO | 31.4 | 6.3 | 16.8 | 8.4 | 16.0 | 12.8 | 8.3 |

The SCT pasting solvents obviously show significant variations in composition. Given the largely invariant SCT operating conditions (T, P, slurry concentration) it should be possible to assess the effect, if any, of PS composition on SCT product composition. We have taken two approaches to this question: (1) determining the quality of PS distillates as hydrogen donor solvents in standard microautoclave tests, and (2) correlating HO compositions with PS compositions. The results are described below.

Microautoclave Extractions Using PS Distillates. The distillate fractions of the PS have been used in standard microautoclave tests previously described^(1,2,6). Two run conditions (EQ: 750°F, 2/1 S/C, 30 min and KIN: 750°F, 8/1 S/C, 10 min) have been used previously with SRC-I⁽⁵⁾ and H-Coal⁽⁶⁾ liquids and a considerable body of data exists describing a solvent's quality by its ability to convert a standard coal to THF solubles at these conditions. The coal used in these tests is the same Indiana V (Old Ben #1) used in Lummus' TSL operations. As shown in the previous quarterly report⁽²⁾ it is equivalent to that used previously with SRC-I solvents⁽⁵⁾. The results with the SCT PS samples (Table 4-11) show conversions of 80-90% at the EQ conditions for all of the PS samples tested. This is essentially the maximum conversion (to THF solubles) attainable with this coal at the conditions used. Typical SRC-I solvents give conversions of 65-75%⁽⁵⁾, and H-Coal recycle distillates (also made with mid-continent bituminous coals) give conversions of 75-85%⁽⁶⁾. Because the standard EQ test was not able to differentiate between the PS samples, the test was modified by reducing the solvent/coal ratio to 1.5/1 in an attempt to effect more complete donor hydrogen depletion of the solvents. Again, the conversions were essentially constant at 89-90% (Table 4-11). This leads us to conclude that the PS are of such high quality that the definition of solvent quality which has been applicable to H-Coal and SRC-I distillate recycle solvents is not useful in differentiating among the PS distillates.

Table 4-11

Microautoclave Extractions of Indiana V Coal
 With SCT Distillates (850°F)

| | Conversion to THF Solubles, | | |
|---------------|-----------------------------|----------------------|------------------------|
| | wt % MAF Coal | | |
| | <u>KIN</u> | <u>2/1 S/C</u> EQ | <u>1.5/1 S/C</u> EQ |
| SCT12-6F11080 | | | |
| 6H011080 | | | 88.5 |
| SCT13-5F11180 | | | |
| 5H011180 | | | 73.1 |
| SCT14-1F11280 | | | |
| PS21280 | 84.8 | 90.4 | 90.7 |
| PS31280 | 78.6 | 90.5 | 91.2 |
| PS41280 | 79.9 | 89.5 | 90.9 |
| PS51280 | 82.5 | 90.0 | 90.7 |
| 1H011280 | | | 83.8 |
| HO21280 | 81.5 | 88.2 | 84.4 |
| HO31280 | 86.3 | 88.3 | 86.8 |
| HO41280 | 81.8 | 88.7 | 88.8 |
| HO51280 | 81.3 | 88.6 | 89.2 |
| SCT15-PS1181 | 80.9 | 90.1 | 90.2 |
| PS2181 | 81.8 | 91.4 | 90.9 |
| PS3181 | 86.5 | 89.6 | 89.2 |
| H01181 | 84.5 | 87.8 | 87.4 |
| HO2181 | 83.3 | 89.8 | 87.8 |
| HO3181 | 84.7 | 87.6 | 84.1 |
| SCT16-PS1281 | 82.7 | 89.5 | 89.7 |
| PS2281 | 80.5 | 89.1 | 89.6 |
| 4PS1381 | 80.7 | 87.9 | 87.6 |
| 5PS2381 | 83.0 | | 86.3 |
| 6PS3381 | 80.9 | | 87.2 |
| H01281 | 80.2 | | 84.0 |
| HO2281 | 79.9 | | |
| 4HO1381 | 83.7 | | 83.9 |
| 5HO2381 | 81.9 | | 84.2 |
| 6HO3381 | 80.4 | | 84.5 |

Conversions at the KIN conditions, which are meant to measure hydrogen donor kinetics, average around 82% for the PS distillates. Several of the solvents do show conversions significantly above this norm, particularly SCT14-PS-2-1280 and SCT15-PS-3181. Distillates from SCT14-PS-3-1280 and SCT14-PS-4-1280 give significantly lower conversions. This is not surprising since the $^1\text{H-NMR}$ data show the former to be high in condensed aromatics and the latter to be low in condensed aromatics relative to the norm for the PS distillates (Table 4-9). Given the surplus of donor hydrogen available, as evidenced by the EQ results and further insured by the high solvent/coal ratio at the KIN conditions, it seems likely that differences in solvent quality as measured by the KIN conditions reflect differences in the physical solvent properties of these solvents. That is, more aromatic solvents are better physical solvents and will give higher KIN conversions in the presence of adequate donor hydrogen.

Typical SRC-I solvents give conversions of 75-80% at the KIN conditions⁽⁵⁾, while H-Coal recycle distillates average near 80%⁽⁶⁾. The fact that these values lie much closer to the TSL-PS values than do the EQ results indicates that the same kinds of donor hydrogen mechanisms are operative for all these solvents, and that differences are more in the quantity of donor hydrogen available. Conversion with the HO (product) distillates at the EQ conditions average several points lower than the corresponding PS distillates. At the KIN conditions, conversions are about the same for feed and product distillates. This too is evidence for the donor hydrogen mechanism implied above.

Correlation of SCT Operating Results With PS Quality. The results above establish the PS distillates as remarkably high quality hydrogen donor solvents. However, in SCT operations the PS composition includes varying amounts of distillate, and varying qualities of recycle resid. In addition, the space velocity in the SCT unit was varied over a wide range. The fact that the PS distillates were

not strictly equivalent at the KIN conditions, consistent with differences in their proton distributions, argues for an attempt to correlate overall PS composition with SCT unit performance. The wt % preasphaltene in the HO (product) resid was selected as the response variable for this interpretative effort for several reasons:

- Since SCT coal conversions (e.g., to THF solubles) are essentially constant at 92%, variations in the preasphaltene content should be a good indication of unit performance.
- The preasphaltene content of the HO resid is likely to have an impact on subsequent ASDA and LC-Finer processing.
- The preasphaltene content, on a wt % 850°F⁺ THF soluble basis, is invariant to the amount of light distillate and gases removed in the post-SCT pressure letdown and can, therefore, be established with a high degree of certainty.

The independent variables chosen for the correlation are given in Table 4-12 and fall into three categories.

1. Unit Operating Parameters. Only space velocity was used. Temperature, pressure and coal concentration were not significantly varied in the Lummus runs.
2. PS Resid Quality. The amount of distillate in the PS and the amount of preasphaltenes in the PS resid serve to scale the correlation to account for the likelihood that high preasphaltenes in the PS will produce high preasphaltenes in the product.
3. PS Distillate Quality. The three proton type ratios used here are linearly independent and have been shown in previous work⁽⁵⁾ to provide a valid quantitative description of solvent quality.

These six variables were used in a multiple linear regression with the HO resid preasphaltenes and the results are given in Table 4-13. Using all sixteen data points available gave a correlation coefficient of 0.86 ($r^2 = 0.74$) with an average (absolute) error between calculated and observed values of 2.2%. Two data points are particularly poorly correlated (SCT15-1 and SCT19-7), and eliminating

Table 4-12

Correlation of Pasting Solvent Quality and Preasphaltene Content
of HO Resid - Input Parameters and Correlation Constants

| Run | Period | Space Velocity | Resid Quality | | Distillate PS Solvent Quality* | | | % Preasphaltenes in HO Resid |
|-----|--------|-------------------|-----------------------|---------------------------------|--------------------------------|--------------------------|-----------------|---------------------------------|
| | | | % Distillate in PS | % Preasphaltenes in PS Resid | Aromatic Aliphatic | Condensed Uncondensed | Cyclic Alkyl | |
| 14 | 2 | 205 | 61.6 | 22.2 | 0.36 | 2.54 | 1.05 | 38.4 |
| | 3 | 184 | 57.1 | 16.0 | 0.23 | 1.31 | 0.95 | 39.0 |
| | 4 | 174 | 55.5 | 30.0 | 0.32 | 1.12 | 0.94 | 49.8 |
| | 5 | 133 | 59.7 | 25.6 | 0.20 | 1.32 | 1.00 | 45.5 |
| 15 | 1 | 116 | 74.6 | 28.2 | 0.39 | 2.25 | 1.05 | 42.9 |
| | 2 | 116 | 46.6 | 30.0 | 0.34 | 2.56 | 0.98 | 48.6 |
| | 3 | 145 | 72.6 | 34.1 | 0.60 | 3.51 | 0.88 | 54.0 |
| 16 | 1 | 118 | 60.6 | 36.7 | 0.25 | 2.69 | 1.02 | 49.8 |
| | 2 | 141 | 61.2 | 37.8 | 0.25 | 2.40 | 1.27 | 49.5 |
| | 4 | 180 | 71.3 | 38.6 | 0.32 | 2.74 | 0.82 | 46.9 |
| | 5 | 191 | 68.8 | 38.0 | 0.34 | 2.55 | 0.81 | 54.8 |
| | 6 | 185 | 70.1 | 40.3 | 0.31 | 1.74 | 0.73 | 59.8 |
| 19 | 1 | 179 | 57.7 | 35.9 | 0.36 | 3.19 | 1.16 | 42.8 |
| | 2 | 147 | 75.4 | 28.5 | 0.31 | 1.88 | 1.23 | 42.7 |
| | 4 | 175 | 71.1 | 32.3 | 0.31 | 2.22 | 1.27 | 46.9 |
| | 7 | 170 | 78.5 | 21.5 | 0.53 | 3.61 | 1.14 | 48.0 |

* Proton ratios from ¹H-NMR spectra of FS distillates, Tables 4-9 and 4-10.

Table 4-13

Comparison of Observed and Calculated
Preasphaltenes in THF Soluble HO Resid

| <u>Run</u> | <u>Period</u> | <u>16 Point Correlation % Preasphaltenes in HO Resid</u> | | <u>14 Point Correlation % Preasphaltenes in HO Resid</u> | |
|-------------------------|---------------|--|-----------------|--|-----------------|
| | | <u>Calculated</u> | <u>Observed</u> | <u>Calculated</u> | <u>Observed</u> |
| 14 | 2 | 41.3 | 38.4 | 39.0 | 38.4 |
| | 3 | 39.3 | 39.0 | 38.5 | 39.0 |
| | 4 | 49.3 | 49.8 | 50.0 | 49.8 |
| | 5 | 43.5 | 45.5 | 44.8 | 45.5 |
| 15 | 1 | 47.9 | 42.9 | -- | -- |
| | 2 | 44.8 | 48.6 | 44.6 | 48.6 |
| | 3 | 54.3 | 54.0 | 53.5 | 54.0 |
| 16 | 1 | 46.5 | 49.8 | 48.1 | 49.8 |
| | 2 | 49.6 | 49.5 | 51.8 | 49.5 |
| | 4 | 47.4 | 46.9 | 46.8 | 46.9 |
| | 5 | 52.8 | 54.8 | 54.2 | 54.8 |
| | 6 | 56.7 | 59.8 | 59.7 | 59.8 |
| | 7 | 42.9 | 48.0 | -- | -- |
| 19 | 1 | 45.0 | 42.8 | 43.7 | 42.8 |
| | 2 | 44.7 | 42.7 | 45.2 | 42.7 |
| | 4 | 44.6 | 46.9 | 44.4 | 46.9 |
| | 7 | 42.9 | 48.0 | -- | -- |
| Average (Calc-Obs) | | | 2.2% | 1.2% | |
| Correlation Coefficient | | | 0.86 | 0.98 | |

these gives the 14-point correlation with $r^2 = 0.96$ and an average error of 1.2%. This is about the experimental uncertainty of the preasphaltene measurement. The correlation is plotted in Figure 4-1.

The effects of the individual term in the correlation can be determined from the correlation constants, Table 4-14. The data in Table 4-14 include the signed values of the constants for both the 16 and 14 point correlations, and the average response (the constant times the average value of a variable) and response range (the standard deviation of the variable times the correlation constant) for the 14-point correlation.

The space velocity has no significant effect on the pre-asphaltenes in the HO resid. The response range (1.3%) is within experimental uncertainty.

The amount of distillate in the PS has a positive correlation with HO preasphaltenes. This suggests that a higher boiling recycle distillate is desirable in the SCT unit, the other variables being held constant. As expected, the PS preasphaltenes show a strong positive correlation with the HO preasphaltenes. We will be receiving data from Lummus which will allow us to calculate balances around the SCT unit, and we will investigate whether the PS preasphaltenes are simply carried through the SCT unit or if they are converted to lighter product.

The three parameters describing the PS distillate solvent quality have significant impacts on HO resid preasphaltenes. Increasing the cyclic/alkyl aliphatic and condensed/uncondensed aromatic proton ratios reduce preasphaltenes, at a constant aromatic/aliphatic ratio. Increased aromaticity increases the preasphaltene yield. These three parameters define the balance that must be achieved in the LC-Finer unit to optimize recycle distillate quality. Too little hydrogenation, while it may increase the condensed/uncondensed ratio, will also raise total aromaticity and reduce cyclic/alkyl aliphatics (e.g., SCT15-3). Over hydrogenation, while reducing

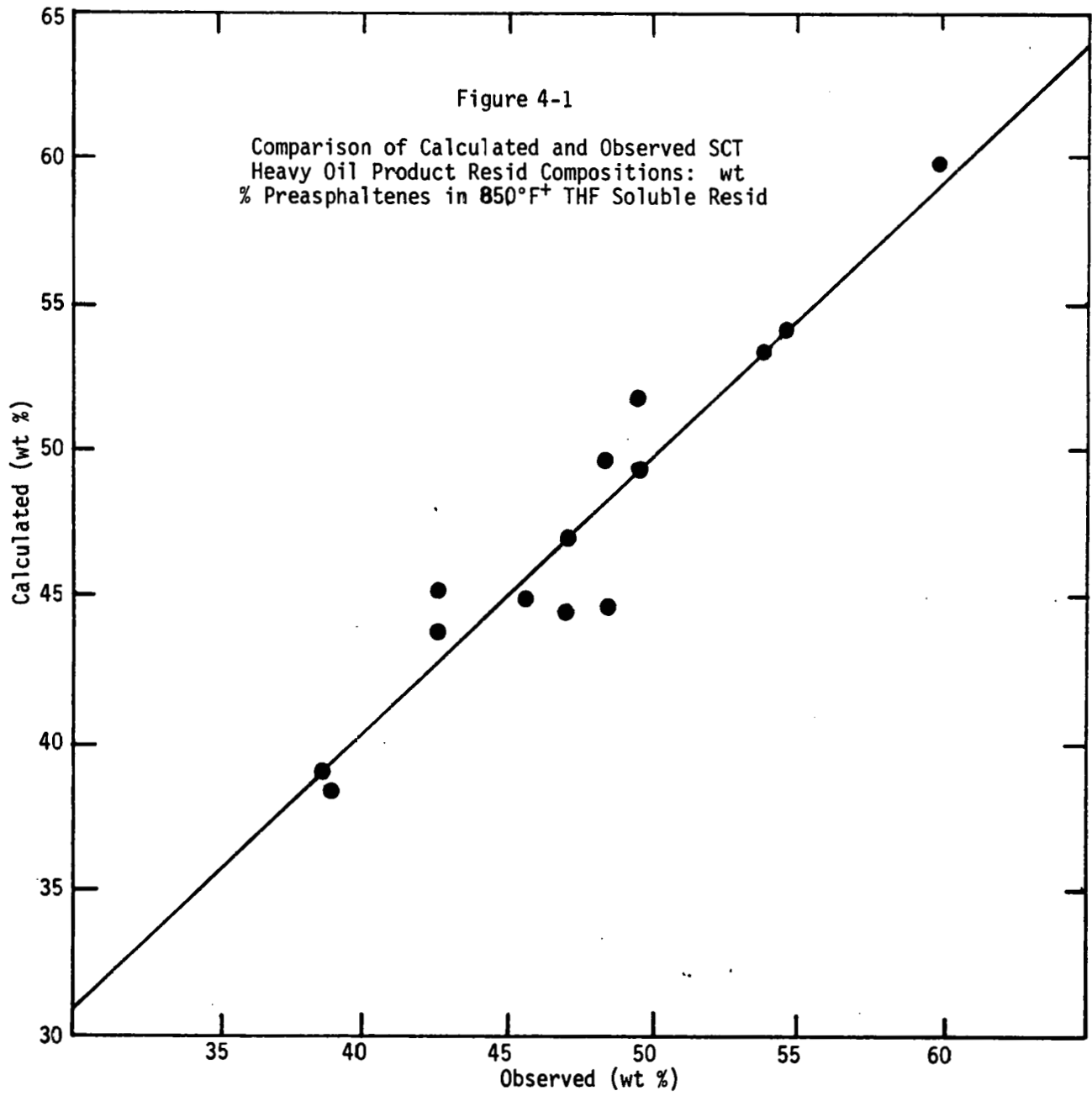


Table 4-14

Correlation of Pasting Solvent Composition With Heavy Oil Preasphaltenes - Correlation Constants

| Correlation ^a Constants (C _i) | Space Velocity | Resid Quality | | Distillate PS Solvent Quality | | | Constant (C ₀) |
|---|-------------------|-----------------------|---------------------------------|-------------------------------|--------------------------|-----------------|----------------------------|
| | | % Distillate in PS | % Preasphaltenes in PS Resid | Aromatic Aliphatic | Condensed Uncondensed | Cyclic Alkyl | |
| 16 pt. | -0.006 | 0.101 | 0.519 | 24.58 | -2.81 | -12.62 | 36.40 |
| 14 pt. | -0.022 | 0.161 | 0.646 | 18.30 | -3.60 | -16.23 | 38.99 |
| Avg. Response ^b (C _i \bar{X}) | -3.5 | 10.5 | 20.0 | 6.2 | -8.5 | -16.5 | 39.0 |
| Response Range ^b (2 σ C _i) | 1.3 | 2.9 | 9.1 | 3.8 | 5.4 | 5.4 | |

^a% preasphaltenes in HO resid = $\sum_{i=0}^6 C_i X_i$ where X_i is the value of a given variable.

^bBased on 14-point correlation.

total aromatics, will also reduce condensed aromatics and cyclic/alkyl aliphatics (perhaps seen in SCT16-6). In summary, the ideal PS would exhibit the following properties.

- The distillate content would be reduced, perhaps by increasing its IBP.
- The preasphaltene content would be reduced, if this can be done in the LC-Fining step without sacrificing donor hydrogen properties.
- The distillate hydrogenation would be controlled to promote hydroaromatic formation, but without cracking of condensed aromatics to alkylated mono-aromatics. This trade-off must be experimentally determined but can be monitored using the three proton ratios. It may, for example, be possible to improve solvent quality by reducing LC-Finer severity, although with a probable decrease in 850°F^+ conversion to 850°F^- .

2. Antisolvent Deasher (ASDA) Operation

Samples of feed, overflow and underflow from two ASDA runs (128 and 129) were analyzed to determine if any fractionation of the soluble material occurs in the unit. The idea that the antisolvent precipitates a portion of the solubilized coal, which agglomerates the ash and IOM causing it to settle more rapidly, suggests an enrichment of, probably, the preasphaltenes in the underflow.

Component distributions of the ASDA samples are given in Table 4-15. The effective removal of IOM and ash from the overflow are apparent. Table 4-16 presents the solubility fractionations of the 850°F^+ THF solubles from these samples. ASDA 128 does show a slight enrichment of preasphaltenes in the underflow, with a corresponding decrease in the overflow. The data from ASDA 129, which are very similar to the ASDA 128 data, show no apparent trend. At best, the ASDA 128 data show a very slight fractionation of preasphaltenes. While the differences are outside of the experimental uncertainty of the analytical method ($\sim 1\%$ for each solubility fraction) it is possible that sampling, sample handling, work-up, etc. could effect the small differences observed. 850°F^+ mass balances around the ASDA, based on a forced ash balance (Table 4-17) are good, indicating

Table 4-15

Antisolvent Deasher (ASDA) Feeds Overflows
and Underflows, Component Distributions

| <u>Run</u> | <u>Sample</u> | <u>Wt. % of Total Sample</u> | | | | | |
|------------|---------------|------------------------------|-------------|--------------------|-----------------------|------------|------------|
| | | <u>Distillate</u> | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> | <u>IOM</u> | <u>Ash</u> |
| ASDA 128 | Feed | 42.8 | 13.9 | 11.3 | 21.3 | 5.3 | 4.6 |
| | Overflow | 46.5 | 17.0 | 13.3 | 22.3 | 0.1 | 0.2 |
| | Underflow | 44.4 | 9.7 | 8.3 | 16.6 | 10.6 | 10.0 |
| ASDA 129 | Feed | 43.0 | 15.5 | 10.2 | 21.1 | 5.1 | 4.6 |
| | Overflow | 40.7 | 19.2 | 14.3 | 25.3 | 0.1 | 0.1 |
| | Underflow | 40.6 | 9.6 | 7.6 | 13.7 | 15.6 | 11.8 |

Table 4-16

Solubility Fractionations - Antisolvent Deasher Feeds,
Overflows and Underflows, 850°F⁺ THF Solubles

| <u>Run</u> | <u>Sample</u> | <u>Wt % 850°F⁺, THF Solubles</u> | | |
|------------|---------------|---|--------------------|-----------------------|
| | | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> |
| ASDA 128 | Feed | 29.9 | 24.2 | 45.9 |
| | Overflow | 32.3 | 25.3 | 42.4 |
| | Underflow | 28.0 | 24.1 | 48.0 |
| ASDA 129 | Feed | 33.2 | 21.7 | 45.1 |
| | Overflow | 32.6 | 24.4 | 43.0 |
| | Underflow | 31.0 | 24.6 | 44.4 |

Table 4-17

Antisolvent Deasher - Mass Balances.
of 850°F⁺ Components - Forced Ash Balance

| | (OUT/IN) x 100 | | | | |
|-----------------|----------------|--------------------|-----------------------|-------------|------------|
| | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> | <u>IOM</u> | <u>Ash</u> |
| <u>ASDA 128</u> | | | | | |
| Observed IN | 24.6 | 19.9 | 37.8 | 9.4 | 8.2 |
| Calculated OUT | <u>25.7</u> | <u>20.6</u> | <u>36.8</u> | <u>8.5</u> | <u>8.2</u> |
| % OUT/IN | 104.5 | 103.5 | 97.4 | 90.4 | 100.0 |
| <u>ASDA 129</u> | | | | | |
| Observed IN | 27.4 | 18.1 | 37.3 | 9.0 | 8.1 |
| Calculated OUT | <u>26.2</u> | <u>19.8</u> | <u>35.2</u> | <u>10.7</u> | <u>8.1</u> |
| % OUT/IN | 95.6 | 109.4 | 94.6 | 118.9 | 100.0 |

Table 4-18

Proton Distributions by $^1\text{H-NMR}$
Antisolvent Deasher (ASDA) Distillates (850°F)

| <u>Run</u> | <u>Sample</u> | <u>Condensed Aromatics</u> | <u>Uncondensed Aromatics</u> | <u>Cyclic α</u> | <u>Alkyl α</u> | <u>Cyclic β</u> | <u>Alkyl β</u> | <u>γ</u> |
|------------|---------------|--------------------------------|----------------------------------|---------------------------------------|--------------------------------------|--------------------------------------|-------------------------------------|----------------------------|
| 128 | Feed | 22.5 | 6.9 | 16.7 | 8.6 | 21.0 | 15.0 | 9.3 |
| | Overflow | 23.9 | 7.3 | 16.8 | 11.0 | 17.1 | 14.8 | 9.0 |
| | Underflow | 23.5 | 8.3 | 15.4 | 13.8 | 15.5 | 14.4 | 9.0 |
| 129 | Feed | 23.6 | 6.7 | 16.5 | 8.7 | 20.2 | 14.9 | 9.5 |
| | Overflow | 28.8 | 6.7 | 16.8 | 9.6 | 16.6 | 13.2 | 8.3 |
| | Underflow | 23.0 | 8.2 | 16.4 | 13.7 | 15.2 | 14.8 | 8.7 |

no chemical alteration of the 850°F⁺ components during their transit through the ASDA unit. We will be analyzing additional ASDA sample sets, and expect to receive feed and product rate data to allow us to calculate unforced balances. These may help to understand the ASDA phenomenon. We will also be further analyzing the whole 850°F⁺ fractions and their individual components by gel permeation chromatography to determine if molecular size differentiation occurs in the ASDA. If so, it must occur without significantly altering the solubility fractionations of these resids, which seems unlikely.

These ASDA results are enigmatic. If in fact additional work establishes that no fractionation of the soluble portion of the ASDA feed occurs, the idea that the ASDA involves precipitation of a portion of the soluble feed may need to be reconsidered.

3. LC-Finer Operation in the Integrated TSL Process

The LC-Finer unit hydrogenates ASDA product to yield the distillate product from the TSL process and to prepare the pasting solvent for the SCT unit, by a fluidized bed hydrogenation. Most of the samples we have analyzed have come from LC-Finer Run 7, and the run conditions are given in Table 4-19. Sample work-ups, solubility fractionations of the resids and ¹H-NMR of the distillates are complete (Tables 4-20, 4-21, and 4-22). We are awaiting more complete operating data before we attempt any interpretation of our results.

4. LC-Finer Operations With Wilsonville SRC Feeds

Several runs on a different LC-Finer unit than that used in the integrated operation (2LCF vs. 3LCF) were made by Lummus and Cities Service to provide data for the ICRC design of SRC-I demo plant. Samples from these runs, which fed SRC from the Wilsonville, Alabama pilot plant, were analyzed in part under contract from Cities Service, and in part under this DOE contract. Feed and product analyses from selected periods of five of these runs are reported here.

Limited data on run conditions are given in Table 4-24. Some of these data, and results on other 2LCF runs have been reported

Table 4-19

LC-Finer Run Conditions

| <u>Run No., Period, Date</u> | <u>Temperature, °F</u> | <u>Pressure, psig</u> | <u>850°F⁽¹⁾ Content in Feed, Wt %</u> | <u>Reference Space Velocity of Feed</u> |
|----------------------------------|----------------------------|---------------------------|--|---|
| 3LCF6-43-11080 | 770 | 2700 | -- | -- |
| 64-11180 | 805 | 2700 | -- | -- |
| 3LCF7-1-21280 | 750 | 2700 | 52.13 | -- |
| 3-31280 | 750 | 2700 | 52.13 | -- |
| 5-41280 | 750 | 2700 | 51.79 | -- |
| 7-51280 | 750 | 2700 | 51.79 | -- |
| 16-1281 | 770 | 2700 | 57.80 | -- |
| 30-3281 | 770 | 2700 | 54.33 | -- |
| 31-4281 | 770 | 2700 | 52.35 | -- |
| 34-5281 | 780 | 2700 | 56.44 | -- |
| 38-1381 | 780 | 2700 | 49.18 | 1.0 |
| 41-3381 | 780 | 2700 | 46.27 | 1.0 |
| 43-4381 | 780 | 2700 | 49.53 | 1.0 |
| 45-5381 | 780 | 2700 | 53.26 | 1.0 |
| 48-6381 | 780 | 2700 | 60.00 | 1.0 |
| 49-7381 | 780 | 2700 | 56.32 | 1.0 |
| 52-1481 | 780 | 2700 | 56.98 | 1.67 |
| 54-1581 | 780 | 2700 | 61.61 | 1.67 |

(1) Lummus determination.

Table 4-20

Component Distribution LC-Finer Samples
From Integrated TSL Operations

| Sample | Wt % of Total Sample | | | | Mass Balance |
|----------------|-----------------------------|------------------|------|-----------------|--------------|
| | 270°C /5 torr Distillate | Soluble Resid | IOM | Oxidized Ash | |
| 3LCF6-43F11080 | 50.1 | 45.7 | 3.0 | 0.4 | 99.2 |
| 64F11180 | 28.5 | 59.0 | 7.7 | 3.3 | 98.5 |
| 43P11080 | 55.1 | 43.5 | 0.5 | 0.5 | 99.6 |
| 64P11180 | 49.4 | 45.7 | 1.6 | 2.8 | 99.5 |
| 3LCF7-1F21280 | 43.4 | 48.7 | 5.1 | 2.2 | 99.4 |
| 3F31280 | 41.9 | 49.9 | 4.9 | 2.6 | 99.4 |
| 5F41280 | 44.0 | 48.4 | 4.2 | 2.2 | 98.7 |
| 7F51280 | 44.3 | 48.5 | 4.3 | 2.3 | 99.4 |
| 16F1281 | 34.8 | 56.2 | 5.3 | 2.8 | 99.1 |
| 30F3281 | 40.5 | 52.2 | 4.5 | 2.2 | 99.5 |
| 31F4281 | 39.9 | 53.7 | 4.0 | 2.0 | 99.6 |
| 34F5281 | 37.4 | 55.5 | 4.4 | 2.0 | 99.3 |
| 38F1381 | 46.9 | 47.9 | 3.1 | 1.8 | 99.7 |
| 41F3381 | 51.2 | 43.3 | 3.6 | 1.4 | 99.5 |
| 43F4381 | 45.8 | 47.0 | 4.4 | 2.0 | 99.3 |
| 45F5381 | 41.3 | 46.2 | 8.7 | 3.1 | 99.3 |
| 48F6381 | 37.8 | 52.5 | 6.9 | 3.0 | 100.2 |
| 49F7381 | 37.0 | 48.2 | 10.2 | 3.7 | 99.0 |
| 52F1481 | 33.0 | 49.7 | 15.8 | 1.1 | 99.6 |
| 54F1581 | 31.7 | 56.0 | 9.5 | 2.3 | 99.4 |
| 1P21280 | 56.5 | 39.2 | 1.4 | 2.2 | 99.3 |
| 3P31280 | 55.1 | 38.5 | 2.2 | 2.9 | 98.7 |
| 5P41280 | 55.8 | 38.6 | 1.8 | 2.7 | 98.9 |
| 7P51280 | 57.0 | 37.8 | 2.0 | 2.8 | 99.5 |
| 16P1281 | 52.9 | 39.8 | 2.7 | 4.0 | 99.4 |
| 30P3281 | 50.1 | 43.6 | 2.3 | 3.6 | 99.6 |
| 31P4281 | 52.4 | 41.9 | 2.0 | 2.9 | 99.2 |
| 34P5281 | 54.4 | 39.2 | 2.4 | 3.4 | 99.5 |
| 38P1381 | 61.2 | 37.1 | 0.4 | 0.7 | 99.5 |
| 41P3381 | 61.7 | 35.2 | 1.1 | 1.6 | 99.6 |
| 43P4381 | 56.7 | 38.0 | 2.1 | 2.8 | 99.6 |
| 45P5381 | 50.6 | 40.6 | 3.7 | 4.7 | 99.6 |
| 48P6381 | 50.3 | 42.2 | 2.8 | 4.3 | 99.6 |
| 49P7381 | 47.3 | 43.5 | 3.5 | 5.2 | 99.5 |
| 52P1481 | 52.3 | 43.6 | 2.4 | 1.3 | 99.5 |
| 54P1581 | 51.2 | 43.3 | 2.3 | 2.6 | 99.4 |

Table 4-21

Solvent Fractionation of THF Soluble 270°C⁺/5 torr
Resid LC-Finer Samples From Integrated TSL Operations

| <u>Sample</u> | <u>Wt % of THF Soluble Resid</u> | | |
|----------------|----------------------------------|--------------------|-----------------------|
| | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> |
| 3LCF6-43F11080 | 34.1 | 23.5 | 42.4 |
| 64F11180 | 33.1 | 23.4 | 43.5 |
| 43P11080 | 41.7 | 35.5 | 22.8 |
| 64P11180 | 56.7 | 25.2 | 18.1 |
| 3LCF7-1F21280 | 30.6 | 24.8 | 44.6 |
| 3F31280 | 29.5 | 23.5 | 47.0 |
| 5F41280 | 29.4 | 25.0 | 45.6 |
| 7F51280 | 31.3 | 26.1 | 42.6 |
| 16F1281 | 29.2 | 22.5 | 48.3 |
| 30F3280 | 32.0 | 24.4 | 43.6 |
| 31F4281 | 30.0 | 23.2 | 46.8 |
| 34F5281 | 32.2 | 22.9 | 44.9 |
| 38F1381 | 32.3 | 23.1 | 44.6 |
| 41F3381 | 27.7 | 21.5 | 50.8 |
| 43F4381 | 32.5 | 21.3 | 46.2 |
| 45F5381 | 33.5 | 19.7 | 46.8 |
| 48F6381 | 32.5 | 20.2 | 47.3 |
| 49F7381 | 35.4 | 19.4 | 45.2 |
| 52F1481 | 41.3 | 20.5 | 38.2 |
| 54F1581 | 38.2 | 22.1 | 39.7 |
| 1P21280 | 69.6 | 19.7 | 10.7 |
| 3P31280 | 50.2 | 22.5 | 27.3 |
| 5P41280 | 50.6 | 21.3 | 28.1 |
| 7P51280 | 67.7 | 18.4 | 13.9 |
| 16P1281 | 57.8 | 19.6 | 22.6 |
| 30P3281 | 49.0 | 26.6 | 24.4 |
| 31P4281 | 44.6 | 27.4 | 28.0 |
| 34P5281 | 39.6 | 21.8 | 38.6 |
| 38P1381 | 50.7 | 24.7 | 24.6 |
| 41P3381 | 46.4 | 21.8 | 31.8 |
| 43P4381 | 47.0 | 19.9 | 33.1 |
| 45P5381 | 44.6 | 22.2 | 33.2 |
| 48P6381 | 43.7 | 22.6 | 33.7 |
| 49P7381 | 46.2 | 23.5 | 30.3 |
| 52P1481 | 40.0 | 27.1 | 32.9 |
| 54P1581 | 41.1 | 27.7 | 31.2 |

Table 4-22

Proton Distributions LC-Finer Distillates (850°F⁻)
From Integrated TSL Operations

| <u>Sample</u> | <u>Condensed Aromatics</u> | <u>Uncondensed Aromatics</u> | <u>Cyclic α</u> | <u>Alkyl α</u> | <u>Cyclic β</u> | <u>Alkyl β</u> | <u>γ</u> |
|----------------|--------------------------------|----------------------------------|---------------------|--------------------|---------------------|--------------------|----------|
| 3LCF6-43F11080 | 26.4 | 7.6 | 16.5 | 9.1 | 18.6 | 13.7 | 8.2 |
| 64F11180 | 35.7 | 6.5 | 15.6 | 7.6 | 14.9 | 12.4 | 7.3 |
| 43P11080 | 14.2 | 7.3 | 15.4 | 8.7 | 24.1 | 18.6 | 11.7 |
| 64P11180 | 14.5 | 6.4 | 15.4 | 8.1 | 23.6 | 19.7 | 12.4 |
| 3LCF7-1F21280 | 28.8 | 9.0 | 14.1 | 11.2 | 14.1 | 13.8 | 9.0 |
| 3F31280 | 29.9 | 8.4 | 14.7 | 11.7 | 13.7 | 13.3 | 8.4 |
| 5F41280 | 30.6 | 8.2 | 14.5 | 11.7 | 13.1 | 13.4 | 8.5 |
| 7F51280 | 29.6 | 7.9 | 15.4 | 11.4 | 13.9 | 13.4 | 8.4 |
| 16F1281 | 31.6 | 6.2 | 16.3 | 8.9 | 15.7 | 13.2 | 8.0 |
| 30F3281 | 35.3 | 6.0 | 15.9 | 7.9 | 14.7 | 12.2 | 8.1 |
| 31F4281 | 27.7 | 8.8 | 14.9 | 8.1 | 16.6 | 14.5 | 9.3 |
| 34F5281 | 32.2 | 7.2 | 14.9 | 8.0 | 15.4 | 13.8 | 8.6 |
| 38F1381 | 23.6 | 7.6 | 13.3 | 10.8 | 17.7 | 15.5 | 11.5 |
| 41F3381 | 21.1 | 9.2 | 14.4 | 11.3 | 16.4 | 16.5 | 11.1 |
| 43F4381 | 23.3 | 8.3 | 14.3 | 10.9 | 16.5 | 15.8 | 10.9 |
| 45F5381 | 25.4 | 7.7 | 12.3 | 10.4 | 15.4 | 16.5 | 12.3 |
| 48F6381 | 32.3 | 5.2 | 14.2 | 8.1 | 15.6 | 14.4 | 10.1 |
| 49F7381 | 23.7 | 5.9 | 14.1 | 8.8 | 19.0 | 17.3 | 11.3 |
| 52F1481 | 37.4 | 6.1 | 12.2 | 7.7 | 14.0 | 13.2 | 9.4 |
| 54F1581 | 38.1 | 5.1 | 15.1 | 7.1 | 14.0 | 12.0 | 8.5 |
| 1P21280 | 7.6 | 7.7 | 14.9 | 9.7 | 25.7 | 20.3 | 14.2 |
| 3P31280 | 8.1 | 8.6 | 14.6 | 9.9 | 24.6 | 20.4 | 13.8 |
| 5P41280 | 8.7 | 7.7 | 16.9 | 10.3 | 24.9 | 19.7 | 11.8 |
| 7P51280 | 5.3 | 7.5 | 15.9 | 9.7 | 27.4 | 21.0 | 13.2 |
| 16P1281 | 9.9 | 7.8 | 16.4 | 8.3 | 24.8 | 20.3 | 12.6 |
| 30P3281 | 5.1 | 6.0 | 12.8 | 7.5 | 27.2 | 24.2 | 17.3 |
| 31P4281 | 6.0 | 5.1 | 13.5 | 7.6 | 29.8 | 23.3 | 14.6 |
| 34P5281 | 7.8 | 5.6 | 13.6 | 7.2 | 27.5 | 22.4 | 15.8 |
| 38P1381 | 7.3 | 5.5 | 12.8 | 7.8 | 27.4 | 23.7 | 15.4 |
| 41P3381 | 5.1 | 6.0 | 9.6 | 8.4 | 28.6 | 24.5 | 17.8 |
| 43P4381 | 6.3 | 5.7 | 13.4 | 8.1 | 26.1 | 24.6 | 15.9 |
| 45P5381 | 5.9 | 6.4 | 12.1 | 7.5 | 26.9 | 24.0 | 17.3 |
| 48P6381 | 7.4 | 6.0 | 14.5 | 8.1 | 27.1 | 21.3 | 15.5 |
| 49P7381 | 6.9 | 5.3 | 14.5 | 8.2 | 28.2 | 22.5 | 14.4 |
| 52P1481 | 9.7 | 6.0 | 15.7 | 8.1 | 27.2 | 20.5 | 12.9 |
| 54P1581 | 11.7 | 6.3 | 16.1 | 8.2 | 24.8 | 19.4 | 12.8 |

Table 4-23

Microautoclave Conversions, LCF Distillates
 (850°F) From TSL Operations

| | Conversion to THF Solubles, | | |
|----------------|-----------------------------|-----------------------------|-------------------------------|
| | wt % MAF Coal | | |
| | <u>KIN</u> | <u>2/1 S/C</u> <u>EQ</u> | <u>1.5/1 S/C</u> <u>EQ</u> |
| 3LCF6-43F11080 | | | 89.1 |
| 64F11180 | 86.5 | 87.5 | 83.5 |
| 43P11080 | | | 91.0 |
| 64P11180 | 81.9 | 88.8 | 89.7 |
| 3LCF7-1F21280 | | | 82.2 |
| 3F31280 | | | 80.5 |
| 5F41280 | | | 81.5 |
| 7F51280 | | | 80.1 |
| 16F1281 | | | |
| 30F3281 | | | |
| 31F4281 | | | |
| 34F5281 | | | |
| 38F1381 | | | |
| 41F3381 | | | |
| 43F4381 | | | |
| 45F5381 | | | |
| 48F6381 | | | |
| 49F7381 | | | |
| 52F1481 | | | |
| 54F1581 | | | |
| 56F2581 | | | |
| 1P21280 | | | 91.4 |
| 3P31280 | | | 90.5 |
| 5P41280 | | | 90.9 |
| 7P51280 | | | 90.6 |
| 16P1281 | | | |
| 30P3281 | | | |
| 31P4281 | | | |
| 34P5281 | | | |
| 38P1381 | | | |
| 41P3381 | | | |
| 43P4381 | | | |
| 45P5381 | | | |
| 48P6381 | | | |
| 49P7381 | | | |
| 52P1481 | | | |
| 54P1581 | | | |
| 56P2581 | | | |

Table 4-24

LC-Finer Run Descriptions

| <u>Run</u> | <u>Catalyst</u> | <u>Feed SRC</u> | <u>Solvent</u> | <u>Severity</u> |
|------------|-----------------|-----------------|----------------|-----------------|
| 2LCF22 | Ni/Mo | Pyro Mine | 8% H | |
| 24 | Ni/Mo | Pyro Mine | 7% H | |
| 27 | Ni/Mo | Fies Mine | | High |
| 28 | Co/Mo | Fies Mine | | High |
| 29 | Ni/Mo | Fies Mine | | Low |

elsewhere⁽⁷⁾. With the limited operating results available it is, nevertheless, possible to draw some conclusions concerning catalyst deactivation mechanisms in these LC-Finer operations from the analytical data. Table 4-25 gives component distributions and sample descriptions. Note that in Run 27 some catalyst was replaced and regeneration attempted after Cycle III. The last Run 27 sample, made with the recycle oil from Cycle III is referred to below as Cycle IV. Solubility fractionation data are given in Table 4-26. Proton distributions are given in Table 4-27.

Run 27 showed a significant catalyst deactivation, and the data in Table 4-26 show this in the increasing preasphaltene yield in the total liquid product. IOM yield is also on the rise. After regeneration (Cycle IV) the catalyst activity for preasphaltene conversion is apparently restored.

Run 29, made at lower severity than Run 27, shows lower distillate and higher preasphaltenes in the feed materials than does Run 27. However, the Run 29 feeds are essentially IOM free (Table 4-25).

The two runs are compared in Table 4-30 on the basis of the change in each measured component upon hydrogenation. Preasphaltene and IOM are combined as a single entity. Run 29 shows better distillate selectivity than Run 27. Oils and asphaltene selectivities are about the same for Cycles I, II and III of Run 27 and Cycles I, II and III of Run 29. The preasphaltene selectivity shows the significant decline in catalyst activity in Run 27, mentioned above. Run 29 shows a similar decline, although apparently not so severe as in Run 27. Regeneration of the catalyst in Run 27 restores preasphaltene conversion activity. However, it does not appear to improve distillate, oils or asphaltene activity, resulting in an increase in asphaltenes in Run 27, Cycle IV.

¹H-NMR data show little difference between feed and product distillates in Run 27 (Table 4-31) for Cycles I, II and III. Distillate aromaticity is steadily increasing for these three cycles, with substantial loss of donor hydrogen (Cyclic β). Catalyst regeneration

Table 4-25

Component Distribution of LC-Finer Samples -
Operations With Wilsonville SRC Feed

| Sample | 850°F ⁻ Distillate | THF Soluble Resid | IOM | Oxidized Ash | Mass Balance |
|---|----------------------------------|-------------------------|--------|-----------------|-----------------|
| 2LCF22-Feed | 22.5 | 76.1 | 0.2 | 0.02 | 98.8 |
| Initial TLP | 62.6 | 35.1 | 0.1 | 0.02 | 97.9 |
| Final TLP | 38.0 | 58.1 | 2.9 | 0.1 | 99.0 |
| 2LCF24-Feed | 20.4 | 78.8 | 0.04 | 0.01 | 99.2 |
| Initial TLP | 41.2 | 57.6 | 0.1 | 0.03 | 99.0 |
| Final TLP | 44.3 | 54.9 | 0.1 | 0.02 | 99.3 |
| 2LCF27-Once Through | 67.7 | 31.9 | < 0.01 | < 0.01 | 99.6 |
| Cycle I | 57.8 | 41.7 | 0.2 | 0.1 | 99.8 |
| Cycle II | 55.0 | 43.1 | 1.4 | 0.1 | 99.6 |
| Cycle III | 55.0 | 40.3 | 3.8 | 0.04 | 99.2 |
| Once Through With Foreign Solvent, Before Regenera- tion | 53.3 | 44.7 | 0.2 | 0.04 | 98.3 |
| After Regeneration With Recycle Oil | 60.4* | 35.1 | 2.3 | 0.05 | 97.9 |
| After Regeneration With Recycle Oil From Cycle III | 52.5 | 43.5 | 2.7 | 0.1 | 98.8 |
| 2LCF28-Once Through Feed | 22.4 | 77.8 | 0.1 | 0.02 | 100.3 |
| Cycle I Feed | 22.9 | 75.0 | 0.2 | 0.01 | 98.1 |
| Cycle I Feed | 23.6 | 74.7 | 0.1 | 0.2 | 98.6 |
| Once Through Prod. | 48.8 | 49.5 | 0.5 | 0.03 | 98.8 |
| Cycle I Prod. | 51.4 | 43.4 | 4.2 | 0.1 | 99.1 |
| Cycle I Prod. | 44.7 | 53.9 | 0.2 | 0.04 | 98.9 |
| 2LCF29-Once Through Feed | 31.4 | 67.2 | 0.2 | 0.1 | 98.8 |
| Cycle I Feed | 23.3 | 74.8 | 0.2 | 0.1 | 98.4 |
| Cycle II Feed | 23.1 | 74.3 | 1.3 | 0.1 | 98.9 |
| Cycle III Feed | 21.6 | 76.6 | 0.5 | 0.1 | 98.8 |
| Once Through Prod. | 55.4 | 43.5 | 0.1 | 0.03 | 99.0 |
| Cycle I Prod. | 52.2 | 47.0 | 0.04 | 0.02 | 99.3 |
| Cycle II Prod. | 51.0 | 47.9 | 0.1 | 0.03 | 99.1 |
| Cycle III Prod. | 46.4 | 52.4 | 0.1 | 0.1 | 99.1 |

* This sample contained 12.6% (absolute) water that reported to the distillate fraction.

Table 4-26

Solvent Fractionation of THF Soluble 270°C⁺/5 torr
(850°F⁺, atm) Resid of LC-Finer Samples -
Operations With Wilsonville SRC Feed

| Sample | Wt % of THF Soluble Resid | | |
|--|---------------------------|-------------|----------------|
| | Oils | Asphaltenes | Preasphaltenes |
| 2LCF22-Feed | 38.7 | 29.0 | 32.3 |
| Initial TLP | 67.5 | 22.4 | 10.1 |
| Final TLP | 51.2 | 26.4 | 22.4 |
| 2LCF24-Feed | 37.2 | 31.4 | 31.4 |
| Initial TLP | 41.4 | 28.6 | 30.0 |
| Final TLP | 45.0 | 31.8 | 23.2 |
| 2LCF27-Once Through | 70.1 | 20.1 | 9.9 |
| Cycle I | 63.5 | 23.9 | 12.6 |
| Cycle II | 61.6 | 25.4 | 13.0 |
| Cycle III | 50.3 | 28.0 | 21.7 |
| Once Through With Foreign Solvent Before Regenera- tion | 51.0 | 41.5 | 7.5 |
| After Regeneration With Recycle Oil | 50.6 | 39.2 | 10.2 |
| After Regeneration With Recycle Oil From Cycle III | 53.5 | 41.7 | 4.8 |
| 2LCF28-Once Through Feed | 40.3 | 25.9 | 33.8 |
| Cycle I Feed | 41.0 | 27.3 | 31.7 |
| Cycle II Feed | 36.7 | 26.0 | 37.3 |
| Once Through Prod. | 64.1 | 25.3 | 10.6 |
| Cycle I Prod. | 54.8 | 25.2 | 20.0 |
| Cycle II Prod. | 61.5 | 23.1 | 15.4 |
| 2LCF29-Once Through Feed | 40.2 | 27.0 | 32.8 |
| Cycle I Feed | 39.4 | 27.5 | 33.1 |
| Cycle II Feed | 37.9 | 27.8 | 34.3 |
| Cycle III Feed | 40.0 | 26.6 | 33.4 |
| Once Through Prod. | 63.1 | 24.5 | 12.4 |
| Cycle I Prod. | 58.4 | 26.8 | 14.8 |
| Cycle II Prod. | 55.6 | 22.6 | 21.8 |
| Cycle III Prod. | 53.4 | 24.6 | 22.0 |

Table 4-27

Proton Distributions of LC-Finer Distillates (850°F⁻)
Operations With Wilsonville SRC Feed

| Sample | % H | Cond. Ar | Uncond. Ar | Cyclic α | Alkyl α | Cyclic β | Alkyl β | γ |
|--|-------|-------------|---------------|-------------|------------|-------------|------------|------|
| 2LCF22-Feed | 8.26 | 24.0 | 8.5 | 19.1 | 10.5 | 19.2 | 11.5 | 7.1 |
| Initial TLP | 10.03 | 9.6 | 7.9 | 14.9 | 9.5 | 24.0 | 20.6 | 13.5 |
| Final TLP | 8.88 | 18.8 | 8.5 | 14.7 | 9.8 | 18.6 | 18.1 | 11.4 |
| 2LCF24-Feed | 7.22 | 36.6 | 9.8 | 17.8 | 9.7 | 12.7 | 8.8 | 4.6 |
| Initial TLP | 9.53 | 11.7 | 9.0 | 16.5 | 10.0 | 23.2 | 18.4 | 11.2 |
| Final TLP | 9.13 | 15.4 | 9.0 | 16.1 | 10.1 | 20.7 | 17.7 | 11.0 |
| 2LCF27-Once Through | 8.57 | 19.6 | 8.5 | 18.1 | 8.8 | 21.1 | 15.2 | 8.7 |
| Cycle I | 8.79 | 18.7 | 8.6 | 17.3 | 9.6 | 20.1 | 16.3 | 9.4 |
| Cycle II | 8.65 | 19.3 | 9.8 | 16.2 | 10.2 | 18.2 | 16.5 | 9.8 |
| Cycle III | 8.40 | 25.2 | 9.7 | 17.1 | 10.8 | 14.0 | 14.7 | 8.6 |
| Once Through with Foreign Solvent Before Regener- ation | 8.06 | 25.2 | 8.9 | 18.7 | 9.4 | 17.6 | 12.9 | 7.2 |
| After Regeneration with a Recycle Oil | 8.74 | 19.3 | 9.4 | 17.0 | 10.3 | 17.8 | 16.5 | 9.6 |
| After Regeneration with a Recycle Oil from Cycle III | 8.85 | 20.5 | 9.9 | 16.7 | 11.0 | 16.6 | 15.8 | 9.5 |
| 2LCF28-Once Through Feed | -- | 32.0 | 9.5 | 19.3 | 8.7 | 15.7 | 9.4 | 5.5 |
| Cycle I Feed | -- | 30.4 | 9.8 | 19.3 | 8.4 | 17.0 | 9.5 | 5.7 |
| Cycle II Feed | -- | 26.6 | 9.1 | 16.8 | 10.3 | 15.1 | 13.9 | 8.2 |
| Once Through Prod. | 8.16 | 24.1 | 10.4 | 18.2 | 11.0 | 15.8 | 13.2 | 7.4 |
| Cycle I Prod. | 7.95 | 29.4 | 9.2 | 17.8 | 10.6 | 13.7 | 12.4 | 6.8 |
| Cycle II Prod. | 8.16 | 26.8 | 9.8 | 16.9 | 11.6 | 13.7 | 13.5 | 7.8 |
| 2LCF29-Once Through Feed | -- | 30.2 | 9.8 | 19.5 | 8.7 | 16.5 | 9.4 | 5.9 |
| Cycle I Feed | -- | 19.4 | 8.6 | 18.7 | 9.8 | 19.8 | 15.0 | 8.7 |
| Cycle II Feed | -- | 19.6 | 9.2 | 17.4 | 10.2 | 18.0 | 15.4 | 10.3 |
| Cycle III Feed | -- | 15.0 | 10.9 | 16.9 | 10.3 | 19.2 | 17.0 | 10.8 |
| Once Through Prod. | 9.14 | 15.1 | 9.5 | 16.6 | 9.8 | 20.8 | 17.3 | 10.8 |
| Cycle I Prod. | 9.43 | 13.2 | 9.3 | 16.5 | 10.4 | 21.0 | 18.2 | 11.4 |
| Cycle II Prod. | 9.59 | 13.5 | 8.9 | 16.5 | 10.4 | 20.3 | 18.7 | 11.6 |
| Cycle III Prod. | 9.50 | 13.6 | 8.7 | 16.5 | 10.8 | 20.1 | 18.8 | 11.4 |

Table 4-28

Feed and Total Liquid Product Analyses - LC-Finer Run 2LCF27

| <u>Sample</u> | | <u>Wt % Total Sample</u> | | | | | |
|---------------|---------|-------------------------------|-------------|--------------------|-----------------------|------------|------------|
| | | <u>Distillate^a</u> | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> | <u>IOM</u> | <u>Ash</u> |
| Once Through | Feed | 35.8 | 27.2 | 18.4 | 17.8 | 0.2 | 0.02 |
| | Product | 67.7 | 22.4 | 6.4 | 3.2 | 0.01 | 0.01 |
| Cycle One | Feed | 34.0 | 31.9 | 17.6 | 16.3 | 0.1 | 0.01 |
| | Product | 57.8 | 26.5 | 10.0 | 5.2 | 0.2 | 0.1 |
| Cycle Two | Feed | 36.4 | 29.6 | 16.0 | 16.6 | 0.6 | 0.03 |
| | Product | 55.0 | 26.6 | 10.9 | 5.6 | 1.4 | 0.1 |
| Cycle Three | Feed | 32.2 | 29.3 | 17.0 | 18.6 | 2.5 | 0.04 |
| | Product | 55.0 | 20.3 | 11.3 | 8.7 | 3.8 | 0.04 |
| Cycle Four | Feed | 32.9 | 26.9 | 16.7 | 17.3 | 5.0 | 0.05 |
| | Product | 52.5 | 23.3 | 18.1 | 2.1 | 2.7 | 0.1 |

^a850°F.

Table 4-29

Sample Distributions - Run 2LCF29

| <u>Period</u> | | <u>Distillate</u> | <u>Oils</u> | <u>Asphaltenes</u> | <u>Preasphaltenes</u> | <u>IOM</u> | <u>Ash</u> |
|---------------|---------|-------------------|-------------|--------------------|-----------------------|------------|------------|
| Once Through | Feed | 31.4 | 27.0 | 18.1 | 22.0 | 0.2 | 0.1 |
| | Product | 55.4 | 27.4 | 10.6 | 5.4 | 0.1 | 0.0 |
| Cycle One | Feed | 23.3 | 29.5 | 20.6 | 24.8 | 0.2 | 0.1 |
| | Product | 52.2 | 27.4 | 12.6 | 7.0 | 0.0 | 0.0 |
| Cycle Two | Feed | 23.1 | 28.2 | 20.7 | 25.5 | 1.3 | 0.1 |
| | Product | 51.0 | 26.6 | 10.8 | 10.4 | 0.1 | 0.0 |
| Cycle Three | Feed | 21.6 | 30.6 | 20.4 | 25.6 | 0.5 | 0.1 |
| | Product | 46.4 | 28.0 | 12.9 | 11.5 | 0.1 | 0.1 |

Table 4-30

LC-Finer Runs 2LCF27 and 2LCF29 -
Hydrogenation Selectivities

| Run | Sample | (OUT/IN) x 100 | | | |
|--------|--------------|----------------|------|-------------|-------------------------|
| | | Distillate | Oils | Asphaltenes | Preasphaltenes + IOM |
| 2LCF27 | Once Through | 1.89 | 0.82 | 0.35 | 0.18 |
| | Cycle One | 1.70 | 0.83 | 0.57 | 0.33 |
| | Cycle Two | 1.51 | 0.90 | 0.68 | 0.41 |
| | Cycle Three | 1.71 | 0.69 | 0.66 | 0.59 |
| | Cycle Four | 1.60 | 0.87 | 1.08 | 0.22 |
| 2LCF29 | Once Through | 1.76 | 1.01 | 0.59 | 0.25 |
| | Cycle One | 2.24 | 0.93 | 0.61 | 0.28 |
| | Cycle Two | 2.20 | 0.94 | 0.52 | 0.39 |
| | Cycle Three | 2.15 | 0.92 | 0.63 | 0.44 |

Table 4-31

Proton Distributions by $^1\text{H-NMR}$, Feed and Product Distillates,
(850°F) LC-Finer Run 2LCF2⁷

| Sample | | Proton Distribution | | | | | | |
|--------------|---------|------------------------|--------------------------|--------------------|-------------------|-------------------|------------------|----------|
| | | Condensed Aromatics | Uncondensed Aromatics | Cyclic α | Alkyl α | Cyclic β | Alkyl β | γ |
| Once Through | Feed | 31.7 | 8.7 | 18.4 | 8.4 | 17.1 | 10.0 | 5.8 |
| | Product | 19.6 | 8.5 | 18.1 | 8.6 | 21.1 | 15.2 | 8.7 |
| Cycle One | Feed | 17.7 | 8.8 | 17.9 | 8.8 | 21.7 | 15.5 | 9.7 |
| | Product | 18.7 | 8.6 | 17.3 | 9.6 | 20.1 | 16.3 | 9.4 |
| Cycle Two | Feed | 21.9 | 8.7 | 17.4 | 9.0 | 18.5 | 15.1 | 9.4 |
| | Product | 19.3 | 9.8 | 16.2 | 10.2 | 18.2 | 16.5 | 9.8 |
| Cycle Three | Feed | 26.6 | 7.5 | 17.8 | 9.1 | 16.4 | 14.1 | 8.6 |
| | Product | 25.2 | 9.7 | 17.1 | 10.8 | 14.0 | 14.7 | 8.6 |
| Cycle Four | Feed | 28.2 | 7.2 | 17.9 | 9.6 | 15.6 | 13.5 | 8.0 |
| | Product | 20.5 | 9.9 | 16.7 | 11.0 | 16.6 | 15.8 | 9.5 |

restores activity for aromatic reduction (Cycle IV) but does not increase donor hydrogen. Instead the decrease in aromaticity increases the alkyl β and paraffinic alkyl α and γ protons. In Run 29 substantial decreases in distillates aromaticity between feed and product are seen in each cycle, with corresponding increases in cyclic β , alkyl β and γ protons. While it is not clear from the run data available how these differences affected or were effected by process operations, it can be concluded that significant qualitative differences did exist which should be taken into account in assessing the import of these runs. It should also be emphasized that these runs were made by reconstituting Wilsonville SRC with creosote oil distillates (in once-through operation) and with recycle LC-Finer distillates in the subsequent recycle operations. Integrated SCT-LC-Fining operations now conducted at Lummus have shown significantly better catalyst life.

Section 5

REFERENCES

1. Burke, F. P., Winschel, R. A., "Recycle Slurry Oil Characterization," Quarterly Report No. 1, DOE Contract No. DE-AC22-80PC-30027, January 1981.
2. Burke, F. P., Winschel, R. A., "Recycle Slurry Oil Characterization," Quarterly Report No. 2, DOE Contract No. DE-AC22-80PC-30027, June 1981.
3. Hydrocarbon Research, Inc., "PDU Run 10," DOE Contract No. DE-AC05-77ET-10152, Draft of Report FE-10152-67.
4. Fort, A. W., "Characterization of H-Coal Syncrude Obtained From Kentucky 11 Coal," IMMR/058, February 1981.
5. Burke, F. P., Winschel, R. A., Pochapsky, T. C., Fuel 60, 562 (1981).
6. Burke, F. P., Winschel, R. A., Pochapsky, T. C., "Development of a Correlation Between Slurry Oil Composition and Process Performance," Final Report, DOE Contract No. DE-AC05-79ET14503, November 1980.
7. Potts, J. D., Chillingworth, R. S., Hastings, K. E., Schindler, H., Burke, F. P., "LC-Fining Support Activities for Two-Stage Liquefaction," 74th AIChE National Meeting, November 1981 (New Orleans).