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COMMERCIAL SCALE EXPANDED BED HYDROPROCESSING OF SOLVENT REFINED COAL (SRC) EXTRACT

Interim Technical Progress Report

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SUMMARY

The objective of this contract (ERDA RFP E(49-18)-2038) was to demonstrate that solvent refined coal (SRC) extract and expanded bed hydroprocessing (LC-Fining) can be combined to produce a valuable liquid fuel or refinery feedstock from coal, and thus expand the commercial utilization of the SRC process. For this purpose, two tasks were established. Task I involved the determination of the optimum process conditions for hydrotreating SRC extract in an LC-Fining process development unit (PDU). Task II included engineering studies and an on-site inspection of the Cities Service Lake Charles LC-Fining Unit which would lead to the eventual processing of 15,000 barrels of SRC extract (Task III).

PDU results showed that 60-65% of the solvent refined coal is converted to distillates in one pass through two expanded catalyst beds in series. A 30-day catalyst aging run (one batch of catalyst) showed no appreciable catalyst aging at the above conversion level. Sufficient sulfur removal was achieved with a 90+% reduction in the sulfur contained in SRC converted to distillates and a 85% reduction in the sulfur contained in unconverted SRC. The nitrogen removal of 60-80% in SRC converted to distillates is insufficient for processing of distillates in other conventional refinery catalytic processes or for direct fuel usage. These require a distillate nitrogen content of less than 0.3 wt.%.

The inspection of the Lake Charles commercial unit showed the high pressure circuit to be in good mechanical shape. Installation of new feedstock pumps could lower the minimum feedstock rate to 500 BPD. This rate corresponds to the lowest space velocity used in our PDU work. A 50% SRC/50% foreign solvent blend can easily be handled in the steam tracing and pumping equipment available at Lake Charles.

Analysis of PDU data has provided the following additional information:

1. Solvent refined coal can be redissolved in native process solvent, and selected foreign solvents, without adverse effects. The SRC can be further processed in an LC-Fining Unit using a commercial catalyst

2. Coal derived solvents were more compatible with the SRC than petroleum based solvents.
3. Conversion, denitrogenation, and oxygen removal in LC-Fining decrease with increasing SRC level in the feed blend and increasing space velocity. Oxygen removal variations with process parameters were not as marked as with nitrogen removal.
4. Desulfurization was excellent at all levels of the operating variables.
5. There was no apparent catalyst aging effect on conversion, nitrogen, and sulfur removal. Oxygen removal showed a declining rate with catalyst age.
6. The viscosity of the total liquid product showed no significant increase with catalyst age when processing a 52/48 weight percent SRC/solvent blend at 810°F. However, runs made at a higher temperature (830-845°F), twice the space velocity, and more SRC in the feed blend showed a definite increase in the viscosity of the total liquid product as the catalyst aged.
7. A small viscosity increase of the feed blends was noted after 60 days at 210°F in a nitrogen atmosphere.

INTRODUCTION

Solvent Refined Coal (SRC) extract is a hydrocarbon mixture containing significant amounts of oxygen, nitrogen, sulfur, and ash. SRC extract has properties and composition similar to heavy and sour petroleum crude and process derived residua. These are problem stocks which must be upgraded by treating with hydrogen at elevated temperatures and pressures before they can be further refined to finished products. Application of expanded bed hydroprocessing (LC-Fining) to upgrading of SRC extract appears to be a logical and straightforward merging of petroleum refining and coal liquefaction technology.

LC-Fining is a commercially proven process for upgrading heavy residual petroleum oils to clean distillate stocks. The process reacts residual petroleum oils with hydrogen in an expanded catalyst bed at elevated temperatures and pressures. The heavy oils are cracked and hydrogenated over a proprietary commercial catalyst to distillate stocks which can be further processed to clean burning fuels. Sulfur and nitrogen impurities are reacted to hydrogen sulfide (H_2S) and ammonia (NH_3). These impurities are removed from the process by recycle gas scrubbing. The expanded bed design essentially solves fixed bed problems, including plugging, catalyst deactivation, temperature drift, heat dissipation, and catalyst servicing.

Cities Service has shown in two commercial installations that continuous operation can be maintained for long periods of time. The process was first demonstrated by construction of a 2500 BPD unit at Cities Service's Lake Charles, Louisiana refinery. This unit was later expanded to 6000 BPD. The process was then licensed to Petroleos Mexicanos (PEMEX) and a 18,500 BPD unit was built at PEMEX's Salamanca refinery in Mexico.

DEFINITION OF PROGRAM

Active work on the Commercial Scale Expanded Bed Hydroprocessing of Solvent Refined Coal (SRC) Extract, under contract number RFP E(49-18)-2038, commenced on June 1, 1976, and the third of three 30-day catalyst aging runs was completed on April 2, 1977. The primary contract has since been extended for an additional 18 months, commencing June 1, 1977, under Contract No. EX-76-C-01-2038.

The objectives of the primary contract are threefold. First, expanded bed conditions for hydrotreating SRC extract are to be determined in order to optimize denitrogenation and desulfurization with minimal gas production at the maximum conversion of 850°F+ SRC feed. Second, a production quantity of 15,000 barrels of SRC extract from the Fort Lewis, Washington SRC pilot plant is to be upgraded using commercial scale expanded bed hydroprocessing equipment located at Cities Service Company's Lake Charles, Louisiana refinery. Lastly, and more importantly, it will be demonstrated that SRC and expanded bed hydroprocessing can be combined to provide a valuable liquid fuel or refinery feedstock from coal and thus expand the commercial utilization of the SRC process.

The work objectives required two distinct tasks to be performed. The first task involved PDU operation, and the second task involved engineering studies for the Lake Charles expanded bed refinery unit prior to the commercial scale operation of the unit in an as yet unfunded program.

The original work statement was modified, with the agreement of the ERDA Program Manager and permission of the ERDA Procurement Officer, to encompass the following. Task I work included the PDU studies necessary to establish commercial feedstock and operating conditions. The initial PDU studies allowed for processing of higher ratios of SRC/solvent than the 35/65 ratio first specified, and also provided for investigation of foreign solvents. The four 10-day pilot screening runs were:

(a) Solid SRC extract redissolved in native recycle solvent at the Fort Lewis filtrate concentration level (35% SRC/65% solvent);

(b) Solid SRC extract redissolved in foreign solvent, Neville LX-745 Aromatic Oil, at the same concentration as (a);

(c) Solid SRC extract redissolved in foreign solvent, Neville LX-745 Aromatic Oil, at an SRC concentration level higher than in (b);

(d) Solid SRC extract redissolved in foreign solvent, Koppers Heavy Residue Creosote Oil (KC-Oil), at the same concentration level as (c).

In conjunction with the investigation of solvent responses to process parameters as in the above four runs, an SRC/solvent aging study was conducted. SRC/solvent blends were prepared and aged for up to 60 days in the search for a solvent blend that would permit a high SRC/solvent ratio with low solids formation, and still meet viscosity requirements.

A second series of PDU runs consisted of three 30-day catalyst aging runs to demonstrate varying degrees of conversion and denitrogenation/desulfurization at two levels of SRC/solvent ratios.

Task II included all the engineering studies required to define the effort and resources needed to adapt a commercial expanded bed unit at the Lake Charles Refinery to process an SRC/solvent feed mixture. A prerequisite for the engineering study was a complete unit inspection since the unit had been out of service for several years.

The combined work of the above tasks will ultimately determine if it is feasible to proceed to modify the commercial LC-Fining Unit at Cities Service's Lake Charles Refinery and upgrade 15,000 barrels of SRC.

TASK I PROCESS STUDIES

Equipment and Unit Operation

The LC-Fining PDU consisted of three expanded bed reactors in series with adequate process piping to operate in either a two- or three-reactor upflow mode. The two-reactor series mode was employed in the runs described in this report because this corresponds to the Lake Charles operation. The desired feed blend and hydrogen passed through separate preheaters before entering the bottom of the first reactor. The catalyst bed was maintained in an expanded state in each reactor by the external circulation of process liquids around each reactor. The size constraints of the PDU reactor necessitated the external circulation which simulates the internal circulation of a commercial reactor system. The unit operation was on a once through basis with no overall recycle of liquid product or gas from the separators.

The product streams pass through a series of high and low pressure separators and scrubbers to provide a light and heavy oil liquid product plus a gas stream. Light oil, a minor fraction of the total product, is the condensate from the unit's recovery section and has an average end point of 600°F. The heavy oil is the liquid product from the unit and generally is composed of material boiling above 400°F. The end point of the light oil and the initial boiling point of the heavy oil overlap due to the simple condenser type separation of the two product fractions. No total product fractionation was employed. Detailed analyses of the feed and heavy liquid products include CHONS, ash, density, melting or pour point, viscosity, metals, and weight/volume fractions. A PONA analysis was run on the 500°F minus fractions of the liquid product. The used catalyst was examined for metals content and a screen analysis.

A commercially available catalyst was charged into the reactors. A run consisted of lining out the PDU on solvent with the catalyst bed in an expanded state until a temperature was reached approximately 50°F lower than the desired operating temperature. The appropriate feed blend was then introduced and operation was continued on a 24-hour basis, gradually increasing temperature to the run condition. Each 24-hour day of operation constituted a period during which at least one set of samples was removed for analysis. The analyses for a given period represent equilibrium operation for that period.

Charge Stock Components

Detailed analyses of the three solvents used in the four 10-day runs, together with the SRC analysis, are presented in Table 1. The Neville Aromatic Oil, a foreign solvent, was quite similar to the Native Process Solvent with the exception of the oxygen, nitrogen, and sulfur content. Both solvents had a relatively high hydrogen content which should enhance their ability to perform as a good hydrogen donor. Considerable evidence is available in the literature to substantiate the fact that a good hydrogen donor function is advantageous in the choice of a coal solvent. However, the Neville Aromatic Oil solvent was petroleum based and exhibited an incompatability towards the SRC with respect to solids formation. Consequently, a second foreign solvent was chosen for investigation.

The second foreign solvent (Koppers Heavy Residue Creosote Oil - referred to as KC-Oil) was more similar to the Native Process Oil with respect to nitrogen and sulfur, but was quite different in hydrogen and oxygen contents (Table 1). The KC-Oil was a coal-derived product which made it more compatible with the SRC. But, on a fresh feed, once-through basis the KC-Oil, based upon its hydrogen content, will have less ability to perform as a good hydrogen donor solvent. Prehydrogenation of the KC-Oil will be undertaken in future PDU work.

Solvent, native or foreign, is defined as all material boiling below 850°F and SRC is defined as all material boiling above 850°F.

Feed Blends

Detailed analyses of the feed blends for Runs LCF-19 through LCF-26 are presented in Table 2. The elemental contents of the feed blend (carbon, hydrogen, oxygen, nitrogen, sulfur) are in good agreement with the blend composition (SRC/solvent ratio). The nitrogen and sulfur levels of the distillate fractions, on the whole, increase from lighter to heavier boiling fractions as shown below:

	IBP-650°F		650-850°F		850°F	
	N	S	N	S	N	S
LCF-19	-	0.31	-	0.50	-	0.80
20	-	1.57	0.19	2.30	1.99	0.77
21	0.09	1.34	0.50	1.99	2.19	0.73
22	0.74	0.54	1.07	0.57	2.01	0.83
24	0.66	0.52	1.05	0.68	2.12	0.71
25	0.65	0.56	1.01	0.65	2.13	0.72
26	0.64	0.54	0.94	0.67	2.07	0.76

Effect of Solvent Type on Conversion

Comparisons of the data were made in two ways. In the first comparison, all the LC-Fining PDU runs were compared using composite samples from Periods 7 through 10. The catalyst was still relatively fresh at this point in time, yet any catalyst initial activity should have been removed. The data would then represent equilibrium operation with fresh catalyst. This comparison was used in the following sections of the report.

Effect of Solvent Type on Conversion
Effect of SRC/Solvent Feed Blend Ratio on Conversion
Effect of Temperature
Effect of Space Velocity on Conversion
Nitrogen, Oxygen, Ash Removal
Sulfur Removal
Hydrocarbon Type

A second comparison was made using the data from the 30-day aging studies. Selected blend intervals (usually three periods) were taken at various times through a run (usually four) to obtain equilibrium data as a function of catalyst age. This comparison was used in the Catalyst Aging section of the report.

The three solvent types tested (Native Process Solvent, Neville Aromatic Oil, and KC-Oil) are evaluated in part by comparing Runs LCF-19, 20, 21, and 22 (Table 3).

The feed blend distillate fractions of Run LCF-19 and 20 are virtually identical (Table 2) except that the feed blend for Run LCF-20 has a larger amount of SRC in the blend. The non-coal derived Neville Aromatic Oil solvent provided a higher conversion of the 850°F+ material than the coal based Native Process Solvent (45.8 versus 42.2 Vol% 850°F+ respectively)

The feed blend SRC/solvent ratios for Runs LCF-21 (SRC/Neville) and LCF-22 (SRC/KC-Oil) are also similar. The feed blend for Run LCF-22 contains a larger amount of the 650-850°F fraction and less of the IBP-650°F fraction than the corresponding fractions of Run LCF-21 (Table 2). Since the KC-Oil contains more of the 650-850°F fraction, this could account for the blend differences. The non-coal derived Neville solvent provided a higher conversion of the 850°F+ material than the coal based KC-Oil solvent (36.1 versus 33.5 Vol% 850°F+ respectively).

Conversion is defined as the volume percent of 850°F+ material in the liquid feed converted to products boiling below 850°F. The 850°F cut point was selected because this is the highest cut point obtained in the SRC equipment at Fort Lewis, Washington. It also coincides with a readily reproducible cut point in the PDU vacuum distillation equipment.

The yield data from the four screening runs provides the following conclusions:

1. Solvent refined coal can be redissolved in native process solvent and selected foreign solvents without adverse effects.
2. The non-coal derived foreign solvent (Neville Aromatic Oil) provided a higher conversion of the 850°F+ material at a given level of SRC concentration than the foreign coal based solvent (KC-Oil).

Even though the Neville Aromatic Oil solvent appeared to be superior to the KC-Oil solvent based on conversion, aging tests on the two solvents showed the converse to be true. Specifically, the Neville Aromatic Oil solvent formed a noticeable quantity of insoluble material in comparison to the amount of solids formed when the SRC was redissolved in Native Process Solvent. The KC-Oil solvent provided a feed blend with the lowest viscosity and lowest solids formation. One explanation for the better solvent qualities of the KC-Oil can be attributed to the fact that the KC-Oil is a coal derived oil.

Effect of SRC/Solvent Feed Blend Ratio on Conversion

The effect of the SRC/solvent feed blend ratio on the conversion of 850°F+ charge is best illustrated by comparing two runs each on two different solvents - Neville Aromatic Oil, Runs LCF-20/21, and KC-Oil, Runs LCF-25/26 (Table 3). In both cases the run with the lower fraction of SRC in the SRC/solvent feed blend gave the highest conversion.

The increase in lighter materials from the greater conversion of 850°F+ material was reflected primarily in an increase in the 390-500°F and 500-650°F distillate fractions as shown below:

LCF-	20	21	26	25
Period	7,8,9	8,9,10	7,8,9	9B,10B
Solvent	Neville		KC-Oil	
SRC/Solvent (Wt%)	37.1/62.9	78.6/21.4	52/48	67/33
SV	0.97 SV_0	0.89 SV_0	0.44 SV_0	0.44 SV_0
Product Fractions (Vol% Feed)				
390-500°F	23.3	9.4	13.7	9.4
500-650°F	40.9	19.7	30.3	27.7
650-850°F	16.1	18.3	28.1	28.5
850°F+	17.7	46.7	19.2	27.2
Conversion (Vol% 850°F+)	45.8	36.1	62.4	57.8

The effect noted was applicable at two widely separated space rates - 0.89-0.97 SV_0 versus 0.44 SV_0.

Effect of Temperature

The effect of temperature on conversion cannot be clearly defined from the available data. Whenever a temperature change is noted, it is confounded by another process variable change. From a practical point of view, the LC-Fining PDU had fewer operational problems when the reactor temperature was kept at, or below, 810°F.

Effect of Space Velocity on Conversion

The effect of space velocity is best described by a comparison of Runs LCF-24 and LCF-25 - two 30-day catalyst aging runs. The actual base space velocity (SV_0) used in the LC-Fining unit is proprietary with the Cities Service Company. Run LCF-24 was made at an average space velocity of 0.94 SV_0, and Run LCF-25 was made at an average space velocity of 0.46 SV_0 (Table 3). The net effect of reducing the space velocity in half (0.87 SV_0 to 0.44 SV_0) was to increase the conversion from 42.9 to 57.8 Vol% 850°F+. The analysis of the distillate fractions of the liquid product indicate that the incremental increase in lighter material from the increased conversion of 850°F+ material was spread throughout the 390-850°F boiling range. This differs from the effect noted for varying the SRC/solvent ratio where the increase in lighter material was primarily in the 390-500°F boiling range. Thus the two process variable changes, SRC/solvent ratio and space velocity, do not have the same effect on the liquid product distribution (<850°F).

Nitrogen, Oxygen, Ash Removal

The denitrogenation and deoxygenation of SRC/solvent blends was affected by several operating factors, namely:

a) choice of solvent
b) level of SRC content in SRC/solvent feed blend
c) space velocity

The above three factors were identified from the available data on the Neville Aromatic Oil and KC-Oil solvents. Sufficient nitrogen analyses were obtained (Table 4) to describe not only the level of denitrogenation in the total liquid product, but also the level of denitrogenation in selected fractions of the liquid product.

(SEE TABLE NEXT PAGE)

LCF-	20	21	22	24	25	26
Period	7,8,9	8,9,10	7,8,9	7AB,8	9B,10B	7,8,9
Solvent	Neville	Neville	KC	KC	KC	KC
SRC/Solvent (Wt%)	37.1/62.9	78.6/21.4	76.2/23.8	67/33	67/33	52/48
SV	$0.97SV_0$	$0.89SV_0$	$0.93SV_0$	$0.87SV_0$	$0.44SV_0$	$0.44SV_0$
Nitrogen Removal (% of Feed)						
650-850°F	-	-	-4.25*	8.15	31.15	43.92
850°F+	61.17	4.63	39.66	53.61	72.52	74.29
Total Liq. Prod.	36.57	37.16	26.05	32.77	61.89	61.36
Oxygen Removal (% of Feed)						
Total Liq. Prod.	80.13	75.30	64.91	79.87	88.21	84.78
Ash Removal (%)						
Total Liq. Prod.	37	21.6	26.6	43.8	52.5	62.8

*(Negative value indicates addition of nitrogen to the specified product fraction)

Runs LCF-20 and LCF-21 made on an SRC/Neville Aromatic Oil blend show that a decreased amount of SRC in the feed blend produced a greater degree of nitrogen removal in the 850°F+ fraction of the feed blend - that is, nitrogen compounds in SRC are harder to remove. A comparison of Runs LCF-21 and LCF-22 show that the Neville Aromatic Oil solvent gave an enhanced nitrogen removal over that of KC-Oil, all other conditions being essentially equivalent. The space velocity in Run LCF-25 was approximately one-half of that in Run LCF-24, and the nitrogen removal in the total liquid product was correspondingly increased to 61.89 percent from 32.77 percent of the feed. This showed that decreasing the space velocity will increase the nitrogen removal in the total liquid product. The temperature effect on nitrogen removal (compare Runs LCF-22/LCF-24 and LCF-25/LCF-26) was obscured by more drastic changes in both the space velocity and SRC/solvent blend ratio.

A comparison of the nitrogen removal of the distillate fractions of the liquid product shows that the greatest degree of denitrogenation occurs in the 850°F+ fraction. The 650-850°F fraction of the liquid product for Run LCF-22 shows an increase in nitrogen content over the corresponding fraction of the SRC/solvent feed blend. A possible explanation for this behavior would be the ring scission and hydrocracking of the outer non-nitrogen containing aromatic rings of compounds like carbazole (BP 671°F) and phenanthridene (BP 680°F) or acridene (BP 655°F) to yield compounds like indole (BP 489°F) and quinoline (BP 460°F). A competitive reaction mechanism is set up between a simple ring scission and a ring scission accompanied by denitrogenation. Further exploratory work would have to be undertaken to verify this concept.

The oxygen removal from SRC/solvent blends was not affected by the process parameters to the same extent as the previously discussed nitrogen removal. For example, the data for nitrogen removal showed that halving the space velocity (Runs LCF-24/25) caused an increase in nitrogen removal from 32.77 to 61.89 percent. The same variable change increased the oxygen removal from 79.87 to 88.21 percent, a marked contrast to the nitrogen removal data. It is expected that the response of nitrogen removal to a variable change will be tempered as the level of nitrogen removal is increased to that for the oxygen removal.

The amount of nitrogen and oxygen removal is, therefore, influenced by the following:

a) A more highly hydrogenated solvent will increase the degree of denitrogenation and deoxygenation.

b) A larger fraction of SRC in the SRC/solvent feed blend and an increase in the space velocity will decrease the degree of denitrogenation and deoxygenation.

The level of ash in the total liquid product appears to follow the trends previously established for nitrogen and sulfur removal with the exception that the Neville Aromatic Oil solvent has no apparent advantage over the KC-Oil solvent. However, the data trends can be markedly affected by small variations in the ash content analysis. For example, if the reported ash in the total liquid product for Run LCF-21 was 0.11 weight percent instead of 0.12 weight percent as reported, the ash removal percentage would be changed from 21.6 to 28.1 weight percent. This would make the ash removal virtually equivalent for the Neville and KC-Oil solvents at the conditions of Run LCF-21/22. Different interpretations would logically follow if the ash contents for the other runs were similarly perturbed. The data presented for ash removal are indicative only of apparent trends because of the lack of reproducibility in ash analyses.

Sulfur Removal

The total liquid product desulfurization results obtained from the data of Table 4 (processing SRC/KC-Oil feed blends) are uniformly high. The lower limit of detection of the test method used for sulfur determination was 0.06 weight percent sulfur and, hence, the actual level of desulfurization is higher than reported.

Hydrocarbon Type

Table 5 presents a comparison of the hydrocarbon type (paraffins, olefins, aromatics, saturates) for the two lightest fractions of the total liquid product (IBP-390°F and 390-500°F). It is obvious that the aromatics/saturates ratio for the two fractions are exactly opposite each other - high saturates, low aromatics in IBP-390°F fraction and low saturates, high aromatics in 390-500°F fraction. This result is important when deciding upon the required cut-points for a recycle solvent.

The low boiling fraction, IBP-390°F, should be removed from a process solvent before the solvent is recycled back to the expanded bed. The lighter materials, being more paraffinic, are less compatible with the SRC. They can act as an anti-solvent which agglomerates and settles out the SRC in solution tending to increase plugging problems. Furthermore, these fractions do not contain any significant amounts of hydrogen donor molecules.

Catalyst Aging

Three runs were made through 30-day periods on blends of SRC/KC-Oil solvent. Different levels of operating severity were employed in order to develop trends toward optimization of process conditions. Tables 3 and 6 present a detailed comparison of the three runs using data from selected periods for each run. The behavior of the run results for the various operating parameters through period 10 has been previously discussed. This section of the report will consider the run behavior throughout the entire 30-day period.

The conversion level for Runs LCF-24 and LCF-25 did not indicate any appreciable change with time on stream through a 30-day period, although several operational upsets did occur. The reported trends in product properties were not affected by the operational disturbances. The operating parameters for Run LCF-26 were chosen to effectively eliminate the upsets which had previously occurred. Only a small disturbance occurred during Periods 18 and 19. Table 7 presents a comparison of the observed conversion for Run LCF-26 versus a calculated conversion developed from proprietary expanded bed correlations. Two details may be observed from this table. An erratic feed pump rate caused flow disturbances which are reflected in variations in the space velocity. This problem was corrected during periods 18 and 19, and a much smoother space velocity pattern is noted for the remainder of the run. Secondly, it is shown that through Period 18 there is a good relationship between the observed conversion and the calculated conversion. Following Period 18, the observed conversion was always less than the calculated conversion. It is speculated that a premature catalyst aging occurred during the operational disturbance of Periods 18/19 which was either permanent or being remedied at an extremely slow rate during subsequent operation.

Table 6 shows that there was no appreciable change in the elemental analysis for hydrogen, nitrogen, and sulfur in the distillate fractions and the total liquid product for Runs LCF-24, 25, 26. This would indicate that the rate of nitrogen and sulfur removal was relatively constant throughout each run although not necessarily the same for each run. The data shown below substantiates this statement for Run LCF-26 by showing no appreciable change in the nitrogen removal for the total liquid product and distillate fractions.

LCF-26

Period	7,8,9	14,15,16	21,22,23B	28,30B
Nitrogen Removal (%)				
Total Liq. Product	61.36	62.47	61.81	61.57
850°F+	74.29	76.14	72.72	71.59
650-850°F	43.92	45.73	44.17	44.52

However, the oxygen content of the total liquid product and distillate fractions for each run did show an increase with time.

Run LCF-26 experienced good operation throughout the entire 30-day period. It represents the adoption of many of the process variable trends previously established. Not only did the run perform satisfactorily from a mechanical viewpoint, but it also experienced the highest level of conversion and nitrogen and sulfur removal (65.5, 62, and 91 percent respectively).

Viscosities, Total Liquid Product

The most significant effects of process parameters on the viscosity of the total liquid product are found in Runs LCF-24, 25, 26 - the three 30-day aging runs (Table 8). Runs LCF-24 and 25 show an increase in the viscosity of the total liquid product with catalyst age. Decreasing the space velocity in Run LCF-26 (more severe operation) resulted in a higher conversion and a less viscous product when compared to Run LCF-24. However, Run LCF-26, which is representative of good operation throughout the 30-day period, showed no significant increase in viscosity over the entire run. Run LCF-26 was not only run at a lower temperature than either Run LCF-24 or LCF-25, but the feed blend contained the lowest amount of SRC. It is not possible at this time to separate the effect of temperature and SRC content in the feed blend. Suffice it to say that the most stable run, from an operating and yield viewpoint (Run LCF-26), provided a liquid product with a nearly constant viscosity over the run period of 30 days.

Table 9 shows that the viscosity increase of the total liquid product with catalyst age is due primarily to an increase in the viscosity of the 850°F+ fraction (Run LCF-24). As shown below, comparable data for Run LCF-26 show a greatly reduced rate of viscosity increase in the 850°F+ fraction of the total liquid product.

Run LCF-26 Period	Viscosity, Abs CS at Shear Rate of 0.8 Sec^{-1} (400°F)
7,8,9	180
14,15,16	362
21,22,23B	420
28,30B	440

The amount of the 850°F+ in Run LCF-26 is also less than that in Run LCF-24 (20 versus 38 weight percent) which would contribute to a lower viscosity of the total liquid product.

SRC/Solvent Feed Blend Aging Study

Three SRC/solvent feed blends were aged for a total of 60 days at 210°F under a nitrogen atmosphere to check for possible feed degradation and settling. The three feed blends were:

a) 34.5/65.5 Vol% SRC/Neville Aromatic Oil
b) 50/50 Vol% SRC/Neville Aromatic Oil
c) 70/30 Vol% SRC/KC-Oil

Table 10 presents the aging data obtained from the above three blends.

There appears to be a slight degradation of the blends as shown by an increasing viscosity over the 60-day period. However, the viscosities were measured with a Saybolt Furol Second Viscometer (ASTM-D88) which was not as good a technique as the Kinematic viscosity method (ASTM-D445). Still, the SFS viscosity method does indicate an increase in viscosity with time. During the 30-day aging runs (LCF-24, 25, 26) no feed difficulties were encountered which were attributable to feed degradation. There was no correlation between product properties and feed degradation.

Metals Analyses

Considerable difficulty has been experienced with two independent analytical laboratories in providing reproducible, accurate metals analyses. Therefore, the metals analyses for used catalysts, feed, and product streams will be provided as an addendum to this report at a future date.

TASK II

Lake Charles LC-Fining Inspection

A detailed mechanical inspection of the LC-Fining Unit at Cities Service's Lake Charles Refinery was required in order to determine the state of the equipment and to supply information required in calculating the cost of Task III. Without this detailed inspection, the cost estimate for Task III could have been in error by as much as ± 50%. The equipment in the LC-Fining Unit, which had been "mothballed" since February 15, 1974, was emptied and opened for inspection during September and October, 1976.

Extensive work was required to prepare the LC-Fining Unit for inspection. For example, a passivating solution had to be removed and the unit flushed, blinds installed for pressure testing, sand blasting to facilitate visual inspection, erection and removal of temporary scaffolds, welding as required, etc. After the inspection and pressure testing, the unit had to be again placed in the "passive" state to prevent formation of the deleterious polythionic acid.

The high pressure portion of the unit - reactors, fired heaters, exchangers, absorber tower, recycle hydrogen and separator drums, and the interconnecting stainless steel piping - was carefully checked. Hydrostatic tests (up to 4000 psig) were applied to many of the items in the high pressure system. The reactor heads were dye-tested after sandblasting and visually inspected, but the reactors were not pressure tested. In order to pressure test a reactor, a minimum temperature of 310°F would be required to prevent brittle fracture of the metal. Prior to start-up for SRC processing, a hot test of both reactors will be required. No serious cracking was found in either of the reactors at cold inspection conditions.

A fin-fan cooler in the intermediate pressure system was tested at 4000 psig. The overall condition of the tubes, however, was not good and their replacement will be required before putting the cooler in service again.

The LC-Fining Unit was originally designed for operation at 2500 BPD and modified in 1971 to 6000 BPD. A small investment in new feed pumps could lower the unit feed rate to 500 BPD which is necessary for SRC processing.

Adequate heat can be applied to the process transfer lines such that liquids with the viscosity of a 50/50 SRC/solvent blend can be transported. The low pressure flash separator temperatures of the existing equipment can be controlled at approximately 400°F.

The completion of Task II has been temporarily suspended pending additional data from the PDU program in the contract extension.

CONCLUSIONS

PDU experiments have shown that it is feasible and practical to process solvent refined coal (SRC) in an expanded bed reactor (LC-Fining). It was found that SRC requires a dilution of equal volumes of solvent for reactor operability and maximization of conversion to distillates. Sufficient quantities of natural solvent will not be available for the Lake Charles commercial run. Two foreign solvents were tested as potential replacements for natural solvent. The two solvents were both acceptable replacements for natural solvent. Koppers Heavy Creosote Oil (KC-Oil) was selected over Neville LX-745 Aromatic Oil due to better compatibility with SRC. It is recommended that hydrogenated KC-Oil be used for future PDU experiments.

A successful 30-day PDU run was demonstrated with very little catalyst aging. During this run constant SRC conversion levels of 60-65 volume percent were achieved with 90+% sulfur removal and 60+% nitrogen removal. This PDU run demonstrated results at a space velocity which is equivalent to a 500 BPD feedrate at Lake Charles. The 500 BPD feedrate at Lake Charles has been found to be operable with the purchase of new feed pumps.

Analysis of the PDU data provided the following additional conclusions:

1) A higher conversion was obtained with a lower fraction of SRC in the SRC/solvent feed blend and a decrease in space velocity. The petroleum derived solvent provided a higher conversion of the 850°F+ material at a given level of SRC concentration than the coal derived solvent.

2) A higher level of nitrogen, oxygen, and ash removal was obtained with a lower fraction of SRC in the SRC/solvent feed blend and a decrease in space velocity.

3) Nitrogen and oxygen removal was increased by the use of the more highly hydrogenated solvent (larger number of potential hydrogen donors) - Neville Aromatic Oil versus KC-Oil. The choice of solvent had little effect on ash removal.

4) The oxygen removal response to the process variable changes was not as pronounced as the nitrogen removal response.

5) The level of desulfurization (≈90+%) was uniformly high throughout the three 30-day aging runs.

6) Paraffins were the predominant hydrocarbon type in the IBP-390°F fraction of the liquid product, whereas aromatics predominated in the 390-500°F fraction. This will be important in the future in choosing the proper distillation cut point to prepare a process recycle solvent.

7) No appreciable change was noted for conversion, nitrogen removal, and sulfur removal during the 30-day catalyst aging runs.

8) The viscosity of the total liquid product showed no significant increase with catalyst age when processing a 52/48 weight percent SRC/solvent blend at 810°F. However, runs made at a higher temperature (830-845°F), twice the space velocity, and more SRC in the feed blend showed an increase in the viscosity of the total liquid product as the catalyst aged.

9) SRC/solvent feed blends show a slight increase in viscosity when aged at 210°F for 60 days in nitrogen, possibly due to some feed degradation.

TABLE 1

CHARGE STOCK ANALYSES

Description	Native Process Solvent	Neville LX-745 Aromatic Oil	Koppers Hvy. Res. Creosote Oil	Solvent Refined Coal
Sample No.	N-011	N-012	N-013	N-009
Gravity,Sp.,60/60 °F	1.069	1.0497	1.1486	1.230
Pour Point, °F	-10	-24	10	-
Softening Point, °F	-	-	-	353
Viscosity, SUS @ 100°F	129 (a)	53.3 (a)	-	-
SUS @ 210°F	37.1 (a)	32.3 (a)	38.4	-
Elemental Content, Wt%				
Carbon	87.16	91.20	91.48	86.17
Hydrogen	7.46	7.73	5.81	5.66
Oxygen	2.95	0.23	1.07	4.39
Nitrogen	0.96	0.03	1.18	2.09
Sulfur	0.51	1.83	0.56	0.80
Ash, Wt%	0.014	0.0	0.02	0.15
Water Content, Wt%	0.1	Trace	Trace	0.0
Distillation, °F				
IBP	472	446	323	620
5 Vol%	490	514	488	850
10	500	530	530	-
20	512	550	575	-
30	533	564	607	-
40	560	589	633	-
50	592	612	650	-
60	626	635	674	-
70	665	663	706	-
80	716	700	752	-
90	820	742	815	-
95	-	805	845	-
Final Temperature, °F	850	850	850	850
Distillate Recovered, Vol%	92.8	97.0	96.4	5.1
Residue, Vol%	6.6	0.8	3.1	92.3
Loss, Vol%	0.6	2.2	0.5	2.6
Distillate Fractions				
IBP-500°F, Vol%	-	-	6.2	-
Gravity, Sp,60/60 °F	-	-	1.0740	-
500-650 °F, Vol%	-	-	44.2	-
Gravity, Sp,60/60 °F	-	-	1.1089	-
650-850 °F, Vol%	-	-	46.0	-
Gravity, Sp,60/60 °F	-	-	1.1725	-
850 °F+	-	-	3.1	-
Gravity,Sp,60/60 °F	-	-	1.272	-

(a) Converted from Kinematic Determinations

FEED BLENDS

Run No. (LCF-)	19	20	21	22	24	25	26
Blend							
Wt% SRC	32.2	37.1	78.6	76.2	67.0	67.0	52.0
Wt% Solvent	67.8	62.9	21.4	23.8	33.0	33.0	48.0
Solvent	Native	Neville	Neville	Koppers	Koppers	Koppers	Koppers
Gravity, Sp 60/60 °F	1.134	1.1281	1.2220	1.252	1.2432	1.2502	1.2290
Pour Point, °F	65	-	-	-	-	-	-
Softening Point, °F	-	108	244	233	206.5	212	136
Viscosity, Unit @ Temp 1	655(a)	238(b)	-	-	-	-	291.8(c)
Temp 2	220(a)	47(b)	-	-	-	-	58.6(c)
Temp 3	-	-	559(b)	498(b)	140.3(c)	124.8(c)	18.9(c)
Temp 4	-	-	90.6(b)	76.1(b)	39.2(c)	37.5(c)	-
Elemental Content, Wt%							
Carbon	88.30	88.72	87.94	88.06	89.01	89.77	89.12
Hydrogen	6.93	6.79	6.09	5.71	5.70	5.65	5.73
Oxygen	3.40	1.76	3.89	3.61	3.56	3.48	2.82
Nitrogen	1.35	0.73	1.66	1.80	1.69	1.66	1.55
Sulfur	0.58	1.50	0.92	0.80	0.76	0.71	0.69
Ash, Wt%	0.05	0.03	0.14	0.15	0.14	0.18	0.12
Water Content, Wt%	Trace	0.0	-	-	-	-	-
Distillation, °F							
IBP	469	467	469	441	450	447	441
5 Vol%	488	510	546	581	578	560	535
10	499	532	594	630	623	615	590
20	523	561	702	745	695	692	645
30	555	595	-	-	773	750	680
40	610	630	-	-	-	-	730
50	660	665	-	-	-	-	818
60	750	730	-	-	-	-	-
Final Temperature, °F	850	850	850	850	850	850	850
Distillate Recovered, Vol%	68.2	67.2	27.8	29.7	36.6	38.5	52.2
Residue, Vol%	31.6	32.4	72.6	72.7	65.1	64.5	51.0
Loss, Vol%	0.2	0.4	+0.4	+2.4	+1.7	+3.0	+3.2
Distillate Fractions							
IBP-650°F, Vol%	48.5	47.6	15.9	11.7	13.6	13.9	20.7
Gravity, Sp 60/60 °F	1.037	1.0298	1.0217	1.0958	1.0920	1.1008	1.0893
Sulfur, Wt%	0.31	1.57	1.34	0.54	0.52	0.56	0.54
Nitrogen, Wt%	-	-	0.09	0.74	0.66	0.65	0.64
650-850 °F, Vol%	19.7	19.6	11.9	18.0	23.0	24.6	31.5
Gravity, Sp 60/60 °F	1.1208	1.1107	1.1071	1.1602	1.1612	1.1642	1.1579
Sulfur, Wt%	0.50	2.30	1.99	0.57	0.68	0.65	0.67
Nitrogen, Wt%	-	0.19	0.50	1.07	1.05	1.01	0.94
850°F+	31.6	32.4	72.6	72.7	65.1	64.5	51.0
Gravity, Sp 60/60 °F	1.244	1.255	1.2506	1.258	1.2547	1.2566	1.2551
Sulfur, Wt%	0.80	0.77	0.73	0.83	0.71	0.72	0.76
Nitrogen, Wt%	-	1.99	2.19	2.01	2.12	2.13	2.07

(a) SUS @ 180°F, 210°F

(b) SFS @ 180°F, 210°F, 350°F, 400°F

(c) KIN, CST @ 250°F, 300°F, 350°F, 400°F

TABLE 3

YIELD DATA AS A PERCENTAGE OF FEED

Run No. (LCF-)	19	20	21	22	24			25				26			
Blend															
Wt% SRC	32.2	37.1	78.6	76.2	67.0			67.0				52.0			
Wt% Solvent	67.8	62.9	21.4	23.8	33.0			33.0				48.0			
Solvent	Native	Neville	Neville	Koppers											
Average Reactor Temp.,°F	805	805	805	805	845	845	840	830	830	820	820	810	810	810	810
Space Velocity(X times SV_0)	0.94	0.97	0.89	0.93	0.87	0.96	0.99	0.46	0.44	0.44	0.49	0.44	0.40	0.43	0.4
Conversion, Vol% 850°F+	42.2	45.8	36.1	33.5	42.9	43.1	40.6	51.3	57.8	52.9	58.1	62.4	65.5	61.2	59.
Gravity Rise, °API	9.6	10.7	9.1	7.9	9	8.3	8	10.8	13.7	12.8	14.1	18.2	18.4	16.5	16.
Blend Period	6 8, 9	7 8, 9	8 9, 10	7 8, 9	7AB, 8	12, 13B	19C 20B, 21B	6B	9B, 10B	18, 19	26, 27	7 8, 9	14 15, 16	21 22, 23B	28,
Yield-Weight % of Feed															
H_2O	3.4	1.2	3.8	4.4	2.8	3.0	2.6	3.1	3.5	3.3	3.3	3.1	3.5	3.2	3.4
H_2S	0.6	1.3	0.7	0.7	0.7	0.6	0.6	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7
NH_3	0.9	0.5	0.6	0.6	0.8	0.8	0.7	1.2	1.2	1.2	1.1	1.1	1.2	1.2	1.2
CH_4	0.8	1.6	2.1	1.8	3.4	3.1	3.0	4.0	4.0	3.3	3.3	2.5	2.8	2.5	2.5
C_2H_6	0.9	1.0	1.5	1.3	2.8	2.4	2.2	3.4	3.4	2.7	2.6	2.2	2.5	2.3	2.2
C_3H_8	0.9	0.8	1.3	1.2	2.4	2.0	1.9	3.4	3.2	2.6	2.6	2.2	2.4	2.2	2.1
C_3H_6	-	0.02	0.05	0.05	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
$i\text{-}C_4H_{10}$	0.07	0.09	0.1	0.1	0.2	0.2	0.2	0.3	0.2	0.2	0.2	0.2	0.2	0.2	0.1
$m\text{-}C_4H_{10}$	0.6	0.6	1.0	0.9	1.6	1.4	1.5	2.7	2.0	1.8	2.1	1.8	2.0	1.8	1.7
C_4H_8	-	-	-	-	0.03	0.03	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.0
C_5-390°F					2.1	3.4	3.3	2.0	2.7	2.4	5.0	8.0	7.9	7.6	7.9
390-500°F	54.9	57.7	27.2	22.5	4.4	7.2	6.9	3.8	7.3	7.0	10.0	10.8	11.0	10.7	9.5
500-650°F					17.2	18.0	16.2	17.6	22.7	20.8	19.8	25.9	26.1	25.5	23.9
650-850°F	18.6	18.0	17.1	21.4	25.8	22.1	23.8	30.3	25.3	27.9	25.6	25.2	25.2	25.5	26.2
850°F+	19.4	18.8	47.2	47.9	37.9	37.8	39.1	31.7	27.5	30.6	27.4	19.5	18.1	20.4	21.4
Total	101.1	101.6	102.7	102.9	102.2	102.1	102.1	104.3	103.8	104.6	103.8	103.3	103.7	103.9	102.
Yield-Volume % of Feed															
C_5-390°F	6.3	3.6	4.4	3.7	2.6	3.7	3.4	1.8	3.9	3.4	6.2	11.4	12.1	11.0	11.2
390-500°F	17.1	23.3	9.4	6.9	5.6	9.0	8.7	4.9	9.4	9.1	12.9	13.7	13.0	13.6	12.0
500-650°F	38.4	40.9	19.7	16.7	19.7	19.5	18.7	20.8	27.7	25.2	23.5	30.3	30.8	30.0	27.8
650-850°F	19.5	16.1	18.3	21.9	28.8	24.7	26.6	34.4	28.5	31.8	29.2	28.1	28.0	28.0	29.2
850°F+	19.0	17.7	46.7	50.1	37.2	36.9	38.5	31.4	27.2	30.4	27.0	19.2	17.6	19.8	20.9
Total	100.3	101.6	97.0	97.9	93.2	93.8	95.1	92.7	96.0	98.7	98.1	102.6	101.2	102.4	100.
H_2 Consumption (SCF/BBL)					2390	2110	2110	3080	3080	2840	2930	2710	2860	2670	2570

TABLE 4

ELEMENTAL ANALYSIS OF LIQUID PRODUCT

Run No. (LCF-)	19	20	21	22	24	25	26
Blend							
Wt% SRC	32.2	37.1	78.6	76.2	67.0	67.0	52.0
Wt% Solvent	67.8	62.9	21.4	23.8	33.0	33.0	48.0
Solvent	Native	Neville	Neville	Koppers ⟶			
Average Reactor Tempreature,°F	805	805	805	805	845	830	810
Space Velocity (X times SV_0)	0.94	0.97	0.89	0.93	0.87	0.44	0.44
Conversion, Vol% 850°F+	42.2	45.8	36.1	33.5	42.9	57.8	62.4
Gravity Rise, °API	9.6	10.7	9.1	7.9	9	13.7	18.2
Blend Period	6,8,9	7,8,9	8,9,10	7,8,9	7AB,8	9B,10B	7,8,9
Elemental Analysis, Wt%							
Hydrogen							
IBP-390°F	10.23	9.77	10.24	10.45	10.75	10.70	11.96
390-500°F	9.21	9.43	9.48	9.10	9.17	9.43	9.51
500-650°F	8.35	8.74	8.75	7.96	7.78	8.27	8.30
650-850°F	7.87	7.65	7.65	7.12	6.80	7.06	7.08
850°F+	6.26	6.27	5.90	5.75	5.38	5.37	5.66
Total Liquid Product	8.03	8.19	7.06	6.63	6.69	6.92	7.34
Oxygen							
IBP-390°F	1.05	0.33	1.99	3.46	1.59	0.79	0.43
390-500°F	1.17	0.66	0.58	1.45	0.88	0.28	0.21
500-650°F	1.08	0.28	0.50	0.83	0.85	0.58	0.63
650-850°F	0.77	0.31	0.64	0.69	0.51	0.31	0.33
850°F+	1.15	0.92	1.47	1.52	0.99	0.71	0.63
Total Liquid Product	0.99	0.37	1.05	1.38	0.82	0.48	0.48
Nitrogen							
IBP-390°F	0.70	0.35	0.85	1.03	0.75	0.72	0.42
390-500°F	0.61	0.25	0.52	1.16	1.02	0.50	0.49
500-650°F	0.42	0.06	0.43	0.56	0.59	0.44	0.38
650-850°F	0.99	0.35	0.83	0.87	0.81	0.63	0.62
850°F+	1.74	1.50	1.87	1.85	1.72	1.38	1.42
Total Liquid Product	0.91	0.49	1.14	1.45	1.3	0.74	0.67
Sulfur							
IBP-390°F	<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
500-650°F	<0.06	0.31	0.28	0.09	<0.06	<0.06	<0.06
650-850°F	<0.06	0.82	0.42	0.09	0.07	<0.06	<0.06
850°F+	<0.06	0.10	0.29	0.29	0.18	0.10	0.10
Total Liquid Product	<0.06	0.28	0.24	0.23	0.11	<0.06	0.06
Ash, Wt%	0.05	0.02	0.12	0.12	0.09	0.10	0.05

TABLE 5

HYDROCARBON TYPE

Run No. (LCF-)	19	20	21	22	24	25	26
Blend							
Wt % SRC	32.2	37.1	78.6	76.2	67.0	67.0	52.0
Wt % Solvent	67.8	62.9	21.4	23.8	33.0	33.0	48.0
Solvent	Native	Neville	Neville	Koppers	———	———	→
Average Reactor Temperature, °F	805	805	805	805	845	830	810
Space Velocity (X times SV_o)	0.94	0.97	0.89	0.93	0.87	0.44	0.44
Conversion, Vol% 850°F+	42.2	45.8	36.1	33.5	42.9	57.8	62.4
Gravity Rise, °API	9.6	10.7	9.1	7.9	9	13.7	18.2
Blend Period	6,8,9	7,8,9	8,9,10	7,8,9	7AB,8	9B,10B	7,8,9
Hydrocarbon Type, Vol%							
IBP-390°F							
Paraffins	37.6	22.6	25.1	36.1	--	--	--
Olefins	6.6	5.7	0.0	2.4	0.0	1.5	0.5
Aromatics	55.8	71.7	74.9	61.5	68.4	57.6	43.9
Saturates	--	--	--	--	31.6	40.9	55.6
390-500°F							
Paraffins	5.5	3.0	6.3	6.0	--	--	--
Olefins	4.4	0.0	0.0	0.0	0.0	0.0	0.3
Aromatics	90.1	97.0	93.7	94.0	97.6	93.0	92.5
Saturates	--	--	--	--	2.4	6.5	7.2

TABLE 6

ELEMENTAL ANALYSIS OF LIQUID PRODUCT (30-Day Aging Runs)

Run No. (LCF-)	24			25				26			
Blend											
Wt% SRC	67							52			
Wt% Solvent	33							48			
Solvent	Koppers										
Average Reactor Temp., °F	845	845	840	830	830	820	820	810	810	810	810
Space Velocity(X times SV_0)	0.87	0.97	0.99	0.46	0.44	0.44	0.49	0.44	0.40	0.43	0.42
Conversion, Vol% 850°F+	42.9	43.1	40.6	51.3	57.8	52.9	58.1	62.4	65.5	61.2	59.0
Gravity Rise, °API	9	8.3	8.5	10.8	13.7	12.8	14.2	18.2	18.4	16.5	16.5
Blend Period	7AB,8	12,13B	19C 20B,21B	6B	9B,10B	18,19	26.27	7,8,9	14 15,16	21 22,23B	28,30B
Elemental Analysis, Wt%											
Hydrogen											
IBP-390°F	10.75	9.94	10.03	10.53	10.70	10.75	10.65	11.96	12.18	11.82	11.69
390-500°F	9.17	8.74	8.90	9.29	9.43	9.42	9.42	9.51	9.45	9.31	9.19
500-650°F	7.78	7.83	7.61	8.19	8.27	8.25	8.15	8.30	8.15	7.99	8.05
650-850°F	6.80	6.72	6.78	7.19	7.06	7.24	7.36	7.08	6.89	7.04	7.00
850°F+	5.38	5.42	5.40	5.48	5.37	5.50	5.44	5.66	5.49	5.47	5.48
Total Liquid Product	6.69	6.49	6.50	6.80	6.92	6.93	6.98	7.34	7.27	7.16	7.05
Oxygen											
IBP-390°F	1.59	2.76	3.00	0.87	0.79	1.09	1.18	0.43	0.47	0.85	1.06
390-500°F	0.88	1.11	1.21	0.22	0.28	0.37	0.40	0.21	0.32	0.42	0.55
500-650°F	0.85	0.99	1.01	0.55	0.58	0.61	0.65	0.63	0.63	0.70	0.75
650-850°F	0.51	0.57	0.64	0.31	0.31	0.36	0.37	0.33	0.34	0.37	0.40
850°F+	0.99	1.13	1.36	0.66	0.71	0.63	0.82	0.63	0.68	0.72	0.76
Total Liquid Product	0.82	1.16	1.46	0.85	0.48	0.58	0.63	0.48	0.47	0.56	0.63
Nitrogen											
IBP-390°F	0.75	0.55	0.77	0.97	0.72	0.75	0.83	0.42	0.45	0.42	0.47
390-500°F	1.02	0.93	0.96	0.61	0.50	0.65	0.55	0.49	0.51	0.46	0.58
500-650°F	0.59	0.50	0.47	0.40	0.44	0.34	0.34	0.38	0.40	0.37	0.37
650-850°F	0.81	0.77	0.67	0.69	0.63	0.59	0.60	0.62	0.60	0.61	0.59
850°F	1.72	1.78	1.83	1.42	1.38	1.50	1.51	1.42	1.42	1.44	1.43
Total Liquid Product	1.3	1.15	1.16	0.86	0.74	0.77	0.81	0.67	0.65	0.66	0.67
Sulfur											
IBP-390°F	<0.06	<0.06	<0.06	<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	<0.06	<0.06	<0.06
500-650°F	<0.06	0.06	0.07	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06
650-850°F	0.07	0.11	0.12	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06	<0.06
850°F+	0.18	0.22	0.26	0.10	0.10	0.15	0.17	0.10	0.11	0.10	0.13
Total Liquid Product	0.11	0.13	0.15	0.08	<0.06	0.07	0.08	0.06	<0.06	<0.06	<0.06
Ash, Wt%	0.09	0.09	0.09	0.15	0.10	0.13	0.13	0.05	0.07	0.06	0.05

TABLE 7

CALCULATED VERSUS OBSERVED CONVERSION

SRC-KOPPERS CREOSOTE

RUN LCF-26

Period	Space Velocity	Observed Conversion Vol. %	Calculated Conversion Vol. %	(Obs. - Calc.)
1	0.39 SV_o	69.9	67.9	2.
2	0.41 SV_o	73.3	66.3	7.
3	0.40 SV_o	65.2	66.7	-1.5
4	0.41 SV_o	67.3	66.2	1.1
5	0.39 SV_o	64.9	67.5	-2.6
6	0.44 SV_o	65.6	64.2	1.4
7	0.46 SV_o	64.7	62.8	1.9
8	0.43 SV_o	64.3	64.4	-0.1
9	0.43 SV_o	65.0	64.5	0.5
10	0.39 SV_o	67.4	67.9	-0.5
11	0.41 SV_o	65.9	65.9	0.0
12	0.40 SV_o	68.4	68.8	-0.4
13 (a)	-	-	-	-
14	0.38 SV_o	68.5	70.3	-1.8
15	0.41 SV_o	66.0	67.6	-1.6
16	0.42 SV_o	68.1	67.2	0.9
17	0.41 SV_o	66.0	67.7	-1.7
18	0.45 SV_o	63.8	64.3	-0.5
19 (a)	-	-	-	-
20	0.41 SV_o	57.0	67.7	-10.7
21	0.43 SV_o	59.2	66.5	-7.3
22	0.43 SV_o	61.5	66.8	-5.3
23B	0.43 SV_o	63.1	65.7	-2.6
24	0.43 SV_o	60.8	66.0	-5.2
25	0.43 SV_o	60.2	66.0	-5.8
26	0.43 SV_o	62.4	66.0	-3.6
27B	0.42 SV_o	62.3	65.4	-3.1
28	0.40 SV_o	60.2	68.7	-8.5
29 (a)	-	-	-	-
30B	0.44 SV_o	61.2	65.7	-4.5

(a) The products from these periods were not representative due to operational upsets.

TABLE 8

VISCOSITIES - FEED BLEND & TOTAL LIQUID PRODUCT

Run No. (LCF-)	19	20	21	22	24			25				26			
Blend															
Wt% SRC	32.2	37.1	78.6	76.2	67.0			67.0				52.0			
Wt% Solvent	67.8	62.9	21.4	23.8	33.0			33.0				48.0			
Solvent	Native	Neville	Neville	Koppers											
Average Reactor Temperature, °F	805	805	805	805	845	845	840	830	830	820	820	810	810	810	810
Space Velocity (X times SV_0)	0.94	0.97	0.89	0.93	0.87	0.96	0.99	0.46	0.44	0.44	0.49	0.44	0.40	0.43	0.40
Conversion, Vol% 850°F+	42.2	45.8	36.1	33.5	42.9	43.1	40.6	51.3	57.8	52.9	58.1	62.4	65.5	61.2	59.0
Blend Period	6,8,9	7,8,9	8,9,10	7,8,9	7AB,8	12,13B	19C, 20B,21B	6B	9B,10B	18,19	26,27	8,9	14, 15,16	21, 22.23B	28,30B
Viscosity															
Feed Blend, CS @ 180°F	140.6	499.5	-	-	-			-				-			
CS @ 210°F	49.8	97.2	-	-	-			-				-			
CS @ 250°F	-	-	-	-	-			-				291.8			
CS @ 300°F	-	-	-	-	-			-				58.6			
CS @ 350°F	-	-	559(a)	498(a)	140.3			124.8				18.9			
CS @ 400°F	-	-	90.4(a)	76.1(a)	39.2			37.5				-			
Viscosity															
Total Liquid Product, CS @ 100°F	27.1	22.6	-	-	-	-	-	-	-	-	-	-	-	-	-
CS @ 150°F	4.2	4.0	-	-	290.1	1312.2	1692.9	81.87	23.13	40.33	117.6	9.185	8.68	9.05	10.88
CS @ 210°F	-	-	-	591.2	108.6	154.0	190.0	17.81	7.02	10.92	51.7	3.25	2.91	3.31	4.15

(a) Saybolt Furol Seconds

TABLE 9

VISCOSITIES-DISTILLATE FRACTIONS OF TOTAL LIQUID PRODUCT

RUN LCF-24

Period	7AB,8		12,13B		19C,20B,21B	
Fraction	650-850°F	850°F+	650-850°F	850°F+	650-850°F	850°F+
Weight Percent on Fresh Feed	25.8	37.9	22.1	37.8	23.8	39.1
Viscosity						
CS @ 100°F	85.2	-	72.4	-	81.3	-
CS @ 400°F (a)	-	3400	-	3300	-	18,000

(a) Abs. CS at shear rate of 0.8 sec^{-1} measured with a Brookfield Viscometer

TABLE 10

AGING STUDY

SRC/SOLVENT FEED BLENDS

TEMPERATURE 210° F

AGING TIME (DAYS)	0	30	60
Blend No. 1 (34.5 vol.% SRC in Neville Aromatic Oil Solvent)			
Specific Gravity @ 60/60° F	1.1303	1.1371	1.1307
Viscosity, SFS			
@ 140° F	170.7	—	--
@ 180° F	101.9	240.6	122.4
@ 210° F	29.8	53.6	41.0
@ 250° F	--	18.5	16.7
Softening Point, °F	108.0	111.5	99.0
Blend No. 2 (50.0 vo.% SRC in Neville Aromatic Oil Solvent)			
Specific Gravity @ 60/60° F	1.1764	1.1776	1.1780
Viscosity, SFS			
@ 210° F	971.0	1143.0	1578.0
@ 250° F	140.1	171.6	183.8
@ 275° F	62.7	67.8	83.9
Softening Point, °F	145.0	142.0	145.0
Blend No. 5 (70.0 vol.% SRC in KC-Oil Solvent)			
Specific Gravity @ 60/60° F	1.2340	1.2340	1.2420
Viscosity, SFS			
@ 300° F	589.0	700.0	>800.0
Softening Point, °F	206.0	200.0	210.0

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