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Mark P Dvorscak 4/25/95

Office of Intellectual Property Counsel
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Date

ADVANCED LIQUEFACTION USING COAL SWELLING AND CATALYST DISPERSION TECHNIQUES

Report Number Q-08
Quarterly Technical Progress Report
for
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NOTICE

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EXECUTIVE SUMMARY

The overall objective of this project is to develop a new approach for the direct liquefaction of coal to produce an all-distillate product slate at a sizable cost reduction over current technology. The approach integrates coal selection, pretreatment, coal swelling with catalyst impregnation, liquefaction, product recovery with characterization, alternate bottoms processing, and carrying out a technical assessment including an economic evaluation. The project is being carried out under contract to the United States Department of Energy. The primary contractor is Amoco Oil Company, and the subcontractors are Foster Wheeler Development Company, Auburn University, Pennsylvania State University, and Hazen Research, Inc.

The primary coal of this program, Black Thunder subbituminous coal, can be effectively beneficiated to about 3.5 wt% ash using aqueous sulfurous acid pretreatment. This treated coal can be further beneficiated to about 2 wt% ash using commercially available procedures. All three coals used in this study (Black Thunder, Burning Star bituminous, and Martin Lake lignite) are effectively swelled by a number of solvents. The most effective solvents are those having hetero-functionality. In addition, a synergistic effect has been demonstrated, in which solvent blends are more effective for coal swelling than the pure solvents alone.

Laboratory- and bench-scale liquefaction experimentation is underway using swelled and catalyst impregnated coal samples. Higher coal conversions were observed for the SO_2-treated subbituminous coal than the raw coal, regardless of catalyst type. Conversions of swelled coal were highest when Molyvan L, molybdenum naphthenate, and nickel octoate, respectively, were added to the liquefaction solvent. Initial laboratory and continuous flow unit (AU-51) experiments with both Illinois No. 6 and Black Thunder subbituminous coals indicate that the expected increase in coal conversion and product upgrading does not occur when the catalyst precursor, Molyvan L, is impregnated into the swelled coal. The study of a possible interaction between catalyst precursors and the swelling solvents is continuing.

The study of bottoms processing consists of combining the ASCOT process which consists of coupling solvent deasphalting with delayed coking to maximize the production of coal-derived liquids while rejecting solids within the coke drum. The asphalt production phase has been completed; representative product has been evaluated. The solvent system for the deasphalting process has been established. Two ASCOT tests produced overall liquid yields (63.3 wt% and 61.5 wt%) that exceeded the combined liquid yields from the vacuum tower and ROSE process. Direct delayed coking of the atmospheric residue resulted in a lower yield of liquids.

PROGRAM OBJECTIVES

The objective of this project is to develop a new approach to the direct liquefaction of coal to generate an all-distillate product slate at a sizable cost reduction over current technology. The approach integrates all aspects of the coal liquefaction process, including coal selection, pretreatment, coal swelling with catalyst impregnation (and parallel runs with dispersed catalyst); coal liquefaction experimentation including solvent evaluation, product recovery with characterization, alternate bottoms processing, and a technical assessment including an economic evaluation. The three tasks of this program are:

1. Task 1: Laboratory-Scale Experimentation--The goals are to obtain samples of coals and Wilsonville-derived solvents and vacuum tower bottoms and to establish experimental procedures.

2. Task 2: Laboratory-Scale Operation--The goals are to:

   (1) Reduce the concentration of mineral matter and alkali metals in low rank coals, (2) swell and impregnate coals with dispersed catalysts, (3) characterize the resulting catalyst solids, (4) evaluate the effectiveness of the procedures using liquefaction runs, and (5) study solids separation and alternative bottoms handling, which includes observing pumping characteristics, deasphalting, and coking.

3. Task 3: Technical Assessment--The goals are to analyze the data, develop descriptive models, and carry out an economic evaluation.

The research is being carried out in cooperation with the following subcontractors: Foster Wheeler Development Corporation (FWDC), Auburn University (AU), Pennsylvania State University (PSU), and Hazen Research Inc. Appropriate meetings and correspondence are being maintained to ensure effective completion of the project.

PROGRAM MILESTONES

The current milestones are those shown in Figures 1a and 1b. The first milestone was to obtain the appropriate feed stocks for the experiments, and the second was preparing the beneficiated bulk samples of coal for subsequent experiments; both were completed. The laboratory-scale coal swelling and catalyst screening program is underway. Initial bench-scale liquefaction experiments with Black Thunder and Illinois No. 6 coals were carried out. However, coal conversion with the Black Thunder coal was not adequate, so additional runs are planned. The batch deasphalting experiments and delayed coking experiments have been completed. The current program calls for program completion on May 31, 1994. A request for a no-cost extension to September 30, 1994 has been proposed.

SUMMARY OF RESULTS AND ACTIVITY

The objective of this project is to develop a new approach for the direct liquefaction of coal to produce an all-distillate product slate at a sizable cost reduction over current technology. All aspects of the process are included with emphasis upon coal pretreatment, coal swelling with catalyst impregnation, and alternate bottoms processing. Research is under way at Amoco Oil Company, Foster Wheeler Development Company, Auburn University, Pennsylvania State University, and Hazen Research, Inc. In addition, results and samples are being interchanged with Karl Vorres of Argonne Laboratories. His program involves the preparation of coals containing potentially catalytic metals that are introduced into the coals by ion exchange.

Black Thunder subbituminous coal can be effectively beneficiated to about 3.5 wt% ash using aqueous sulfurous acid pretreatment, and it can be further beneficiated to about 2 wt% ash using commercially available procedures. The three coals used in this study are effectively swelled by a number of solvents, the most effective of which are those having heterofunctionality.

Laboratory- and bench-scale liquefaction experimentation is underway using swelled and catalyst impregnated coal samples. Higher coal conversions were observed for the SO_2-treated coal than the raw coal, regardless of catalyst type. Conversions of swelled coal were highest when Molyvan L, molybdenum naphthenate, and nickel octoate, respectively, were added to the liquefaction solvent. Initial laboratory and continuous flow unit (AU-44L and AU-51L) experiments have indicated that the expected increase in coal conversion and product upgrading did not occur when the catalyst precursor, Molyvan L, was impregnated into swelled samples of both Illinois No. 6 and Black Thunder coals. Further work is underway with various solvents and catalyst precursors.

The study of bottoms processing consists of combining the ASCOT process which includes coupling solvent deasphalting with delayed coking to maximize the production of coal-derived liquids while rejecting solids within the coke drum. The asphalt production phase has been completed; representative product has been evaluated. The solvent system for the deasphalting process has been established. Two ASCOT tests produced overall liquid yields that exceeded the combined liquid yields from the vacuum tower and ROSE process. Direct delayed coking of the atmospheric residue resulted in a lower yield of liquids.

DISCUSSION OF RESULTS AND ACTIVITIES BY TASK

Project Documentation

The Milestone Schedule and budget was modified as shown in Figures 1a and 1b.

Task 1.0: Laboratory-Scale Experimentation

Subtask 1.1: Laboratory Support

The feedstocks are on hand; their analyses were reported in previous quarterlies. An additional 10-drum sample of Black Thunder Mine (Thunder Basin Coal Co./ARCO Coal Co.) was obtained to provide a fresh sample for sulfurous acid treatment followed by froth flotation tests. In the period covered by this report, experimentation was underway at Hazen Research, Inc.

Task 2.0: Laboratory-Scale Operation

Subtask 2.1: Coal Pretreatment

Bulk samples of both Martin Lake lignite and Black Thunder subbituminous coal were pretreated to provide beneficiated coal for subsequent experiments. The treatment included crushing, sizing, contacting the coarse fraction with aqueous SO_2 to remove alkali and alkaline metals, and subsequent gravity separation to recover a fraction having particularly low ash content. Two 55-gallon drums of Martin Lake (Texas) lignite and three drums of Black Thunder subbituminous coal were processed at Hazen Research, Inc. The SO_2-treated subbituminous coal was subjected to heavy media (finely dispersed magnetite) cyclone tests, but it was not possible to isolate an overhead (float) fraction having a coal recovery of about 80 wt%. Therefore, the bulk samples of raw and SO_2-treated coals were characterized and used as-prepared.

A research program to expand upon the knowledge of beneficiating low rank coal by a combination of chemical treatment and physical separation methods is being carried out at Pennsylvania State University. A procedure has been developed for producing a low-ash coal from SO_2-treated Black Thunder subbituminous coal. The procedure involved a combination of flotation and agglomeration methods to generate a clean coal product of about 2% ash. This was described in a previous Quarterly Report (Q-06). Experimentation is underway for the beneficiation of Martin Lake lignite. In confirmation of previous work with this lignite, the initial step of demineralization using a 6 wt% aqueous SO_2 solution reduced the ash content from 12.9 wt% to about 6.5 wt% with a combustible matter recovery of about 100%. Samples are being subjected to zeta potential measurements and to a series of flotation and liquid-liquid extraction (including spherical agglomeration) steps. The goals are to determine if this lignite can be beneficiated without the SO_2 treatment step and the depth of beneficiation that can be achieved with a combination of currently available approaches.

Subtask 2.2: Catalyst and Swelling Studies

The results of the simultaneous coal swelling and catalyst impregnation experiments were reported in Quarterly Report Number Q-07 (March-June, 1993). The following conclusions were drawn:

1. Simultaneous swelling and catalyst impregnation experiments conducted with SO_2-treated Black Thunder coal resulted in greater catalyst uptake compared with impregnation using a non-swelling solvent, such as toluene. The rate and extent of catalyst penetration into coal particles correlates with the ability of the solvent to swell coal, i.e., DMSO >> THF ~ IPA >> toluene.

2. Higher impregnation temperature enhances catalyst uptake, with the larger improvement obtained with less effective swelling solvents. Moreover, the effect of lower concentration of the swelling solvent can be offset by increasing the impregnation temperature.

3. Although uptake is better with smaller coal particles, particularly at short impregnation times and ambient temperature, the enhancement is lost at increased temperatures. Therefore, fine grinding of coal should not be necessary at feed slurry tank temperatures.

4. Catalyst uptake with Molyvan L as precursor is more extensive than that with molybdenum octoate. However, the instability of molybdenum octoate (at typical Mo loadings) with some swelling solvents could result in an inactive catalyst form.

Subtask 2.3: Reactivity of Swelled Coals with Dispersed Catalysts

Experimentation during this quarter focused upon evaluating the effectiveness of catalysts that have been impregnated into coal along with the coal swelling solvent. This included studying different means of introducing Molyvan L into untreated Black Thunder subbituminous coal and evaluating several other catalysts which were introduced with the coal swelling solvent or impregnated into the coal. To provide a comparison with Wilsonville runs, Molyvan L at a level of 100 ppm on a coal basis was also tested; this low molybdenum level has been shown previously to be effective in reducing coking in residuum systems.

Reactions were also performed using Wyodak coal that had been washed with nitric acid and then ion exchanged with various metals at Argonne Laboratories. The metals used were iron (Fe), cobalt (Co), molybdenum (Mo), and nickel (Ni). Molybdenum was the only metal that showed a promotional effect for coal conversion. All others appeared to be detrimental to conversion of Black Thunder coal. In fact, the ion exchanged Mo showed as high an activity for coal conversion and pyrene conversion as any system previously performed with a Mo-based catalyst.

Experimental

Materials: Liquefaction reactions were performed using untreated and SO_2-treated Black Thunder coals. The swelling solvents tetrahydrofuran (THF), methanol, and isopropanol (IPA), were obtained from Fisher and were used as received. The liquefaction reaction solvents used were 1-methylnaphthalene (1-MN) (98% purity) and 9, 10-dihydroanthracene (DHA), which were obtained from Aldrich. A coal-derived solvent V-1074 (650-1000°F) was been obtained from Wilsonville liquefaction Run 258. The catalysts were Molyvan L from Vanderbilt Chemical; Mo naphthenate, Ni octoate, and Ni acetylacetonate from Shepherd Chemical Company; and Ni naphthenate from Strem Chemical Company. Reactions using Mo naphthenate were made with added sulfur at three times the catalyst (Mo) mass. Regal 660 carbon black from Cabot Industries was used in one reaction set.

Swelling and Reaction Procedures: Untreated and SO_2-treated Black Thunder coals were swelled by introducing 1.33 g maf coal to the swelling tube and then adding 7 ml of solvent (Table 1). Molyvan L, Mo naphthenate, and Ni octoate catalysts were charged to the swelling solvent at 1.05 times the amount which had been previously used (600 to 800 ppm of active metal) when the catalyst was added directly to the liquefaction reactions. The coal was then allowed to sit unagitated in the swelling solvent for 96 hr. These experiments were designated with a "N" in the subsequent tables. Several experiments, designated with an "A" in the subsequent tables, were performed in which the coal and swelling solvent were agitated during the 96-hr preswelling period. Molyvan L was also added to the swelling solvent at 2.0 times the amount that had previously been used when the catalyst was added directly to the liquefaction reactions. These experiments in which the catalyst level was doubled are designated with a "D" in the subsequent tables. Experiments were also performed with Molyvan L where the swelling time with the catalyst present was varied: 6, 16, and 28 hr. The exact mass of catalyst added to each preswelling solvent is given in Table 2. Experiments were performed in which Molyvan L at 600-800 ppm was dissolved in 1.5 ml of THF and then the solution was applied to 1.33 g coal. Five different procedures were followed and were given the condition designations stated below:

(a) 1.5s: This procedure involved preparing a solution of 1.5 ml of THF with 600-800 ppm of Molyvan L. The solution and 1.33 g maf coal were then mixed. The coal immediately absorbed all of the solvent-catalyst mixture. After solvent absorption, the coal was immediately placed in the tubular reactors with 1-MN as solvent and reacted.

(b) 1.5s+: This procedure involved preparing the sample in the same manner as given in (a) except that after coal absorbed all of the solution, the coal was preswelled in 7 ml of THF swelling solvent. The coal with the absorbed catalyst was allowed to sit in the swelling solvent for 96 hr. The coal was removed from the swelling tube after 96 hr and allowed to sit exposed to the ambient atmosphere for 7 hr. The coal was then reacted in microtubular reactors with 1-MN as solvent.

(c) 1.5s+d: This procedure was identical to the 1.5s+ method except that after the swelling solvent was removed, the coal was allowed to dry completely and returned to its unswelled state. The coal was then placed into a tubular microreactor and reacted.

(d) 1.5s+m: This procedure involved swelling coal with 1.5 ml of THF and then introducing 1-MN into the swelling tube and simultaneously adding the catalyst. The sample was swelled for a total of 96 hr, the solvent was removed and then contacted with air for 7 hr. Then the liquefaction reaction was performed.

(e) Exposure of Molyvan L to Swelling Solvent: This procedure involved placing the catalyst Molyvan L into the THF swelling solvent for 2 hr and evaporating off the solvent. The pretreated catalyst was then placed directly into the reactor with coal that had been swelled in THF for 96 hr. The liquefaction reaction was then performed.

Liquefaction reactions using untreated Black Thunder in 1-MN were performed at 410°C for 10, 20, or 30 min. The liquefaction reactions for SO_2-treated Black Thunder coal in 1-MN and for untreated Black Thunder coal in V1074 were conducted for 30 min. Each reaction contained 1.33 g of maf coal, 2 g of solvent, 0.67 g of pyrene and residual swelling solvent that remained in the swelled coal. The amount of solvent absorbed in the coal for the different preswelling and reaction conditions is presented in Table 2. The amount of catalyst uptake is given for selected reactions. Hydrogen gas was introduced at 1250 psig at ambient temperature. The reactor was well-agitated at 450 cpm.

Products from the liquefaction reactions were removed from the reactor with THF. The conversion of the coal to THF solubles was determined. For the reactions where analysis of catalyst uptake was performed, the values for the weight percent of coal added are given. The weight percent of the coal added in the other reactions are indicated as NYD, but were in the range of 30 to 31%. The amount of pyrene hydrogenation to hydrogenated products was obtained by gas chromatographic analyses using a Varian Model 3400, a J&W DB-5 column and flame ionization detection. Pyrene hydrogenation is defined as the moles of hydrogen required to form the liquid hydrogenation products from pyrene as a percentage of the moles of hydrogen required to form the most hydrogenated product, perhydropyrene.

Liquefaction of Argonne Coals: Liquefaction reactions were performed with Argonne coals that had been ion exchanged with various metals. The Wyodak samples had been treated with Fe, Mo, Co, and Ni. All of these samples had been acid-washed prior to the ion exchange process. An acid-washed sample without ion exchange was also liquefied. The liquefaction reactions were performed thermally with 1-MN as solvent at 410°C for 30 min under well-agitated conditions. Coal conversion and pyrene hydrogenation analyses were performed as described previously.

Catalyst Screening Experiments: Untreated Black Thunder coal was swelled by introducing 1.33 g maf coal to the swelling tube and then adding 7 ml of solvent (Table 1). The slurry-phase catalysts (Molyvan L, Mo naphthenate and Ni acetylacetonate) were charged to the swelling solvent at 1.05 times the standard level of 600 to 800 ppm of active metal. When Mo naphthenate was used, sulfur was added directly to the reactor at three times the catalyst mass. The reaction solvent used in experiments was a mixture of 80% V1074 and 20% DHA. The coal was then allowed to sit unagitated in the swelling solvent for 96 hr.

Liquefaction reactions were performed using the reaction conditions given in Table 1. Liquefaction reactions using untreated Black Thunder in the V1074 and DHA mixtures were performed at 410°C for 30 min. Each reaction contained 1.33 g of maf coal, 2 g of solvent, 0.67 g of pyrene and any residual swelling solvent that remained in the swelled coal. The amount of solvent absorbed in the coal for the different swelling and reaction conditions is presented in Table 2. Hydrogen gas was introduced at 1250 psig at ambient temperature. The reactor was well agitated at 450 cpm. The products from the reaction were analyzed as described previously.

Results and Discussion

Evaluation of Molyvan L in Preswelling Solvents: Table 3 shows the liquefaction results for the various swelling procedures used to evaluate the effect of the preswelling solvent THF on Molyvan L and its activity in coal liquefaction reactions (designated by asterisks). The swelling condition "1.5s" where the THF was only contacted momentarily with the coal before reaction yielded the highest coal conversion of any catalyst introduction method where the swelling solvent contacted coal directly. Coal conversion (84.1±1%) decreased 3.2% on the average with the "1.5s" method compared to adding the catalyst directly to the reactor, which gave the highest coal conversion (87.3±1.1%, Table 6). The standard procedure of swelling coal and catalyst together for 96 hr (1.5s+) yielded 77.7±3.3% coal conversion, an average decrease of 9.6% compared to direct addition of the catalyst to the reactor. The method of 1.5 s+d, in which the coal was allowed to dry completely and collapse from the swelled state, decreased coal conversion even more to 72.8±1.2%, a 14.4% decrease compared to direct catalyst addition.

Pyrene was also reacted in the above liquefaction experiments, but pyrene was introduced into the coal after the swelling step. Pyrene was always added directly into the reactor. Previous results have shown that a catalyst was required in order to hydrogenate pyrene under these reaction conditions. Hence, whenever pyrene hydrogenation occurred, an active form of the catalyst derived from Molyvan L must be present in the system. As shown in Table 3, pyrene conversion averaged more than 20% for the reactions with the 1.5s and 1.5s+d swelling methods. The pyrene conversions with the 1.5s+ method were less and varied greatly. Hence, all of these preswelling conditions resulted in active catalyst precursors being available for hydrogenation of pyrene. Even though Molyvan L was not as active for coal conversion, its activity was not adversely affected for pyrene conversion except when a long contact time (96 hr) with THF occurred.

The product distributions from pyrene obtained during the coal liquefaction reactions are presented in Table 4. The primary product generated during the hydrogenation of pyrene was dihydropyrene (DHP) regardless of how Molyvan L was introduced into the reactor. The secondary products were tetrahydropyrene (THP) and hexahydropyrene (HHP), which were both produced in small quantities.

A comparison of coal and pyrene conversions achieved with untreated Black Thunder coal when the catalyst was first added to the swelling solvent versus those when the catalyst was added directly to the reactor after the coal was swelled in THF is given in Table 5. Coal conversion usually decreased when the coal was preswelled with solvent containing catalyst versus that occurring with direct catalyst addition to the reactor. This decrease occurred with every swelling solvent (THF, IPA, methanol). Pyrene conversion, by contrast, was positive for more than half of the reactions, indicating that the swelling solvent did not adversely affect the catalyst in terms of the resulting pyrene hydrogenation activity.

Effect of Swelling Solvents on the Liquefaction Behavior of Untreated Black Thunder Coal: Hydrogen-bonding solvents like methanol, IPA, THF, and DMSO are typically good solvents for swelling coal while non-hydrogen donors are not effective coal swelling agents. In one experiment, a hydrogen-donor swelling solvent was combined with a non-hydrogen donor swelling solvent. The coal was swelled initially with 1.5 ml of THF and then 7 ml of 1-MN was added with Molyvan L catalyst. The purpose of this experiment was to first swell the coal with a good solvent to open the coal pores, and then to contact this coal with a nonpolar solvent that would not adversely affect the catalyst. In this case, 1-MN was removed prior to reaction, and fresh 1-MN without catalyst was added before carrying out the reaction. Coal and pyrene conversions are given in Tables 3 and 4. They are designated as "1.5s+m" and with an asterisk in the tables. Coal conversion decreased to 50.8% (vs. 87.3% with direct catalyst addition) and pyrene conversion to 3.2% (vs. 18.6% for direct addition). These decreases were 36.5% (absolute) for coal conversion and 15.4% (absolute) for pyrene conversion for this method compared to the catalyst being added directly to the reactor. These conversions are only slightly higher than thermal conversions of untreated Black Thunder coal in 1-MN. The catalyst may not have been able to penetrate the THF-swollen coal when subsequently introduced in 1-MN. It is unlikely that non-polar 1-MN penetrated into the pores containing polar THF and, therefore, the catalyst did not penetrate into the pores of the coal. Since pyrene conversion was also low, it is also unlikely that Molyvan L deposited on the surface of the coal particles.

Effect of Combining Swelling Solvent with Molyvan L: On the basis of these experiments, Molyvan L appeared to be less effective as a catalyst when (1) the catalyst was impregnated into the coal and (2) when the swelling solvent was absorbed in the coal. As a further test, an experiment was performed in which Molyvan L was immersed in THF for 2 hr, and then the THF was removed. This Molyvan L was added to a liquefaction reaction with coal which had been swelled with THF for 96 hr. The coal and pyrene conversions obtained in these reactions are given in Table 6. Coal conversion decreased from 87.3±1.1% (direct addition) to 85.2±1.2%, which was within experimental error. The 2-hr contact of Molyvan L with THF had little effect on the pyrene conversion and hydrogenation to partially saturated products (Table 7).

Liquefaction Behavior of Untreated Black Thunder Coal Using Mo Naphthenate as a Catalyst: The liquefaction behavior of untreated Black Thunder coal in the presence of Mo naphthenate and sulfur was evaluated. The catalyst loading ranged from 600 to 800 ppm Mo; sulfur was added at three times the catalyst mass level; THF was used as the swelling solvent and 1-MN as the reaction solvent. Table 8 shows the results for the three reactions performed: (1) Mo naphthenate and sulfur were added directly to the reactor; (2) Mo naphthenate was added to the swelling solvent with coal present, while the sulfur was added directly to the reactor; and (3) coal was added to the swelling solvent, while both Mo naphthenate and Regal 660 carbon black were added directly into the reaction solvent.

Reference reactions with Mo naphthenate and untreated Black Thunder coal had been performed previously but without sulfur. The untreated coal had been swelled for 96 hr in THF and had also been agitated during the swelling process. The results from those experiments gave coal conversions that were quite low, averaging 62.3%; the pyrene conversions were also low giving an average of 3.5%. When sulfur and Mo naphthenate were added to the reactor with untreated Black Thunder coal preswollen in THF for 96 hr, coal conversion increased to 89.8±0.8% and pyrene conversion to 25.3±0.1%. Therefore, Mo naphthenate with added sulfur is an effective catalyst system.

When coal that had been simultaneously swollen with THF and impregnated with Mo naphthenate was liquefied in the presence of sulfur, conversions of coal and pyrene were 89.8±0.1% and 26.0±0.3%, respectively. These were similar to those obtained in the first catalyst introduction method.

In the third experiment, Regal 660 carbon black was added to the reactor to determine the effect of a solid surface being present on the reactivity of Mo naphthenate. With carbon black present, swelled coal was liquefied in the presence of Mo naphthenate and sulfur, with all of these components added directly to the reactor. Coal conversion decreased to 78.2±0.6% and pyrene conversion also decreased to 15.8±8.2% (high variance in the results).

The pyrene product distributions obtained using Mo naphthenate plus sulfur as the catalyst are presented in Table 9. The major pyrene product was DHP with THP and HHP as minor products. The distributions were the same whether the catalyst was added directly to the reactor or to the swelling solvent.

Reactions of Untreated Black Thunder Coal Using Low Levels of Catalysts: Three swelling solvents were used: THF, IPA, and methanol. Molyvan L was added to the swelling solvent at a nominal level of 100 ppm; the actual loading ranged from 93 to 153 ppm. A catalyst uptake of 91.8% was assumed based on similar runs. Low coal conversions were obtained with untreated Black Thunder coal with this low catalyst loading, as shown in Table 10. The average coal conversions for the three swelling solvents, THF, methanol and isopropanol were 53.6±1.0%, 53.1±1.6%, and 51.6±2.8%, respectively. These values were lower than those of the thermal, non-catalytic runs. Pyrene conversions were also low, ranging from 1.8 to 2.1%. These pyrene conversions were slightly greater than those of the thermal runs, so there was some limited hydrogenation activity. The two pyrene reaction products obtained were DHP and HHP, both of which were produced in low quantities of 1% (Table 11).

Liquefaction Reactions with Swelled Untreated Black Thunder Coal in V1074: Reactions were performed using untreated Black Thunder coal that had been swelled with THF and then reacted in V1074 as the reaction solvent. The conversions achieved in the reactions with the coal swelled in the presence of catalyst are compared to that of the swelled coal with catalyst added directly to the reactor in Table 12. The coal swelled with catalyst showed more variability and lower coal conversion at 76±5.7% than the swelled coal with catalyst added directly to the reactor at 82.8±1.4%. A substantial effect of coal swelling with catalyst was also observed in the pyrene conversion: 30.2±0.3% for direct catalyst addition versus 14.1±1.6% for preswelled catalyst. It is likely that the interaction of THF with the catalyst had an adverse effect on catalytic activity. Pyrene product distributions are given in Table 13. The primary product was DHP; the secondary products were THP and HHP.

Liquefaction of SO_2-Treated Coal: Liquefaction reactions were performed with SO_2-treated Black Thunder coal that had catalyst present during swelling. The reactions performed this quarter were systems of Molyvan L with methanol and Ni octoate with IPA (Table 14). The swelling of coal with a Molyvan L/methanol mixture resulted in an average drop of about 5% in conversion compared to that occurring when the catalyst was added directly to the reactor after coal preswelling. Pyrene conversion also showed a decrease (3.2%). Although pyrene conversion decreased, more than 25% of the pyrene converted to THP or HHP. When SO_2-treated coal was preswelled in methanol with Ni octoate, lower coal conversion and substantially lower pyrene conversion were obtained when compared to those runs with Molyvan L.

Similar results were observed when the coals were preswelled in IPA. Molyvan L was more effective than Ni octoate (87.6±0.7% versus 67.9±1.6%) in the case of SO_2-treated coal. The difference in the pyrene conversion was also substantial (26% versus 5%, respectively).

Table 15 provides a summary comparison of coal and pyrene conversions between the two methods of catalyst introduction. Coal conversion decreased for all the reactions except one in which SO_2-treated coal was contacted with catalyst during swelling as opposed to those in which catalyst was added directly to the reactor. In contrast, pyrene conversion was usually higher when the catalyst was introduced during preswelling.

Liquefaction Behavior of Impregnated Coals Using DHA and V1074 as Liquefaction Solvents: The purpose of these experiments was to determine the liquefaction behavior of coals swelled with THF in the presence of a catalyst in the presence of either a highly-effective hydrogen donor solvent, DHA, or a coal-derived solvent, V1074. The experiments reported above were made with impregnated coal in a non-donor liquefaction solvent, 1-MN.

The coal and pyrene conversions for untreated Black Thunder Coal reacted in V1074 are presented in Table 16. Reactions were performed thermally and catalytically, with and without swelling. The swelling solvent, when used, was THF and the catalysts were Molyvan L and Ni octoate. Four different experiments are compared: (1) thermal and catalytic reactions without prior coal swelling and the catalyst added directly to the reactor; (2) thermal and catalytic reactions with swelled coal and the catalyst added directly to the reactor; and (3) catalytic reactions with the catalyst added to the swelling solvent and introduced to the reactor by being absorbed into the coal.

The least amount of conversion of THF-swollen coal (72.0±1.2) occurred in the thermal reactions (Table 16). Catalytic reactions with Molyvan L, regardless of catalyst introduction method or prior swelling, generally yielded higher conversion than those with Ni octoate. The highest level of coal conversion that was achieved in V1074 was in runs with Molyvan L without prior swelling (84.7±0.7%). When Molyvan L was placed directly in the reactor with preswelled coal, 82.8±1.4% coal conversion was achieved. The least amount of catalytic coal conversion achieved in V1074 occurred when Molyvan L was impregnated into coal (76.0±5.7%). In the case of Ni octoate in V1074, similar coal conversions of 79.8, 76.6, and 78.7% were obtained for no prior swelling, prior swelling, and impregnated coal, respectively. Hence, in V1074 the conditions of catalyst introduction and coal swelling had a much greater effect on coal conversion with Molyvan L than with Ni octoate.

V1074 was an inherently good solvent for untreated Black Thunder coal, yielding a thermal conversion of 72%. In addition, the presence of Molyvan L improved the product quality of the liquefied coal products as evidenced by the pyrene hydrogenation observed in the reactions with Molyvan L (Table 17). Pyrene hydrogenations of 12% were achieved, while those achieved in the thermal and catalytic reactions with Ni octoate were lower than that with Molyvan L. Ni octoate was not an active catalytic hydrogenation agent for pyrene in V1074.

A similar set of reactions was performed using the highly-active donor solvent, DHA, but catalytic reactions were only performed with Molyvan L. The thermal reaction with swelled coal yielded 82.4% conversion. The addition of Molyvan L increased coal conversion. Without prior swelling, 89.7% coal conversion was achieved with Molyvan L in DHA, and 84.5% with prior swelling. When the catalyst was added directly to the reactor, coal conversion was 87.3%.

Pyrene conversion in both DHA and V1074 was greatest when the catalyst was placed directly in the reactor. The presence of THF in the reaction medium was detrimental to the conversion of pyrene to hydrogenated products. The pyrene product distributions are given in Table 18.

Catalyst Screening Reactions: The purpose of these catalyst screening experiments was to determine if other catalysts were more effective as absorbed catalysts than Molyvan L. These catalysts were introduced into the swelling solvent IPA with untreated Black Thunder coal and then the impregnated coal was liquefied. The reaction solvent was comprised of 80% V1074 and 20% DHA so that it had a reasonable hydrogen donating capability.

Since the solvent composition was changed, coal swelled with Molyvan L in THF and then reacted in the V1074/DHA mixture solvent was chosen as the base case. At this time, only two catalysts (Ni naphthenate, a slurry phase catalyst, and Ni acetylacetonate, a powder) have been tested. Both Molyvan L and Ni naphthenate were used at the nominal 600 to 800 ppm of active metal based on coal while Ni acetylacetonate was used at a lower level of 200 ppm. It is assumed that 91.8% catalyst uptake by the coal occurred.

The results from the catalyst screening experiment are presented in Tables 19 and 20. Coal conversion of the base case was 81.2±0.7%. This is essentially the same as a previous reaction in which 79.9±0.6% coal conversion was obtained for coal impregnated with Molyvan L with isopropanol as the swelling solvent and 1-MN as liquefaction solvent (Table 3). Coal conversion achieved with Ni naphthenate as the catalyst was higher yielding 85.4±1.0% conversion compared to 81% with Molyvan L. At the same catalyst loading, swelling, and reaction conditions, Ni naphthenate was more effective than Molyvan L.

Ni naphthenate also was more effective than Molyvan L in terms of pyrene conversion. Molyvan L achieved an average of 7.6±1.2% pyrene conversion while Ni naphthenate yielded 11.4±0.5%. The primary product was DHP with both catalysts with minor amounts of THP and HHP being formed (Table 20).

The results from the reaction utilizing a low level of catalyst loading using Ni acetylacetonate showed a surprising level of coal conversion. The catalyst loading level was double that of the low catalyst level experiments. However, the amount of coal conversion with Ni acetylacetonate was 77% compared to 52 to 54% for Molyvan L. This catalyst shows much promise and will be used at a higher level of active metal.

The Study of Ion Exchanged Wyodak Coal: The coal used in this study was provided by Karl Vorres of the Argonne National Laboratory. Wyodak coal was first acid-washed and then ion-exchanged with metals including Fe, Co, Mo, and Ni. A sample of untreated coal was first liquefied in DHA, V1074, and 1-MN. The results are given in Table 21. Effectiveness of the liquefaction solvents fell in the expected order: DHA > V1074 > 1-MN. Since 1-MN was not a particularly effective solvent, it was chosen as a good medium for evaluating the effect of different metals on coal conversion. The acid-washed Wyodak coal gave low conversions averaging 41.6%, while the thermal conversion of untreated Wyodak coal in 1-MN was 71.5%. The ion-exchanged coals containing Fe, Co, and Ni yielded conversions of 47.4, 46.9, and 60.7%, respectively. All of these conversions were greater than the thermal conversion with acid-washed coal, but less than those with the untreated Wyodak. Molybdenum was the only ion-exchanged metal that was effective, yielding a coal conversion of 88.3%. The only system that converted a substantial amount of pyrene was the ion-exchanged Mo, as shown in Table 22. The major product was DHP with a yield of 28.6%; more THP and HHP were produced than in any of the previously reported catalytic reactions.

Summary of Micro-autoclave Results: The liquefaction of untreated Black Thunder coal which was preswelled with Molyvan L catalyst with THF as solvent resulted in no more coal conversion than that achieved with catalyst just being introduced into the liquefaction mixture. Contacting Molyvan L with THF for 2 hr after which THF was removed also resulted in reduced coal conversion; apparently, contact between THF and Molyvan L is detrimental. The use of a nonswelling solvent such as 1-MN to impregnate Molyvan L into coal was unsuccessful even after the coal had been preswelled with THF. Ion-exchanged Wyodak coals containing Fe, Ni, or Co underwent the same levels of coal conversion as non-exchanged coal samples. Only coal having ion-exchanged Mo yielded high coal conversions. The coal conversions achieved were similar to those obtained when Molyvan L was introduced directly into the reactor. Mo naphthenate, when reacted with added sulfur, was an active slurry-phase catalyst for untreated Black Thunder coal. The method of catalyst introduction, either when the catalyst was added directly to the reactor or impregnated into coal from the swelling solvent, did not affect the activity of the catalyst.

Subtask 2.4: Bench-Scale Studies

The objectives of this subtask are (1) to evaluate the effectiveness of Illinois No. 6 coal swelled with IPA and toluene and catalyst impregnation procedures using a continuous flow liquefaction unit, (2) to generate products for solids separation trials, and (3) to provide leads for subsequent experimentation. The initial series of runs were made with Black Thunder subbituminous coal in the continuous feed AU-51L unit at relatively mild conditions (800°F and 80 min. nominal space time) to bring out the effectiveness of the catalyst and pretreatment conditions. However, as previously reported, these conditions were not severe enough to achieve high coal conversion to provide a representative product to be subjected to the solids separation procedures of FWDC. (Attempts were made to distill and recover an atmospheric bottoms product, but the solids level was high thereby leading to unrealistic poor transfer properties.)

Because additional Black Thunder coal would need to be prepared and the AU-51L unit was scheduled for other runs, the decision was made to progress to carrying out flow experiments with Illinois No. 6 coal in the AU-44L flow unit to evaluate the effect of swelling and low levels of Molyvan L addition. A series of six runs were made with the following feed blends:

1) Raw Ill No. 6 coal.
2) Raw Ill No. 6 coal with 100 ppm Molyvan L added to the feed tank.
3) Same as No. 2, but at 1/2 space velocity.
4) Ill No. 6 coal preswelled with IPA and toluene (non-catalytic).
5) Ill No. 6 coal preswelled with IPA, toluene, and 100 ppm Molyvan L.
6) Same as No. 5 with an additional 100 ppm Molyvan L added to the feed tank.

Experimental:

The Illinois No. 6 liquefaction experiments were carried out in the AU-44L single-stage pilot-scale unit that operates in a once-through continuous mode with regard to hydrogen and feed slurry. The AU-44L system is divided into feed, reaction, product separation, and product recovery sections. The slurry feed is prepared by blending screened/pulverized coal (<100 mesh), preheated solvent(s), and catalyst precursor (if desired). The blend is then charged to the feed tank having both an internal stirrer and recirculation loop. A slip stream flows from the recirculation loop through a positive displacement high pressure, Milton Roy pump. The slurry is then combined with hydrogen and fed to the bottom inlet of a 300 cc stirred Autoclave Engineer's reactor. The working liquid volume of the reactor is 273 cc. The product flows through a series of three dump valves with intermediate volumes to allow for gas expansion and pressure decrease. The product stream flows to one of two vapor/liquid separators. A small amount of light ends is carried with the gas stream to knock-out traps, after which the product gases are metered and analyzed. The product slurry is periodically withdrawn from one of the receivers.

Product analyses for these runs include GC gas analyses and Soxhlet extraction of slurry samples using the solvents toluene, THF, and hexane. The extraction cuts are defined as follows:

1. Unconverted coal - THF insolubles.
2. Preasphaltenes - THF soluble, toluene insolubles.
3. Asphaltenes - toluene soluble, hexane insolubles.
4. Oils - hexane solubles.

The amount of hexane solubles is defined as 100% of the slurry minus the level of hexane insolubles (i.e., asphaltenes, preasphaltenes, and THF insolubles). The latter definition is used to cover light ends that may be lost when the hexane is stripped from the hexane solubles.

The feed coal was Illinois No. 6 coal. The liquefaction solvent consisted of Wilsonville V1074 distillate (650-1000°F) generated from Wilsonville Run 257, which was made using this same coal as feed.

The material balances and yields were calculated using the computer program outlined in the first quarterly report issued under this project.

Results and Discussion:

A series of flow experiments with Illinois No.6 coal was carried out in the AU-44L single stage liquefaction unit to evaluate the effect of swelling and low levels of Molyvan L upon the conversion of this coal. The operating conditions, results, and stream analyses are reported in Tables 23 through 25; material balances are given in the Appendix.

At a reasonable temperature of about 825°F but with a short space time of 22 min, the conversion levels of as-received/raw Illinois No. 6 coal to THF and toluene solubles were 83 and 68%, respectively. With the addition of 100 ppm MoS_2 as Molyvan L, both levels of coal conversion increased 2 to 3% (absolute). These are nominally above the levels of reproducibility of about 1-2%. As observed in the stream analyses and the material balance tables of the Appendix, there was also a nominal increase in the level of hydrogenation (3.7 to 4.5%). The levels of denitrogenation and deoxygenation were unchanged at about 3 and 48%, respectively. There is a question of sulfur analyses, so the levels of desulfurization are not reported. The levels of sulfur remaining in the preasphaltenes and asphaltenes remained relatively high at about 1%.

When the space time was increased to about 36 min for the run with added 100 ppm Molyvan L, coal conversions increased to 91 and 81% (THF and toluene solubles, respectively). The extents of hydrogenation and deoxygenation also increased (7.7% and 62.5%). The level of denitrogenation also increased to 11%, but this is still relatively low and it is consistent with observations that a supported catalyst is needed to achieve a high level of denitrogenation.

Run 18119-01 was made with preswelled Illinois No. 6 coal to serve as a non-catalytic reference for subsequent Molyvan L runs. Considering the differences of space times, the levels of conversion of Run -01 were about those projected from the non-swelled coal runs; namely, the swelling step offered no increase of yields over that of unswelled coal.

As observed in Table 24, the addition of 100 ppm MoS_2 as Molyvan L during the swelling step resulted in the same, or perhaps lower, levels of coal conversions to THF and toluene solubles. Run 18119-04 had conversion levels of 85 and 72%, respectively, while those of Run 18119-01 were 87 and 77%, respectively. When the space times are taken into account (29 vs. 36 min) these levels are equivalent. It is interesting that the further addition of Molyvan L (100 ppm MoS_2) to the feed tank (Run 18119-05) resulted in no additional conversion. In addition, there were no changes in the levels of hydrogenation, denitrogenation and deoxygenation.

In summary, there was a nominal increase of coal conversion and hydrogenation with the addition of a low level of Molyvan L (100 ppm MoS_2) to the feed tank for the liquefaction of Illinois No. 6 coal. However, there appeared to be no increase of conversion or hydrogenation when the coal was either swelled or Molyvan L was introduced during the swelling step.

Subtask 2.5: Solids Separation/Alternative Bottoms Processing

The objective of the subtask is to develop and evaluate a solids separation and alternative bottoms processing route that includes the ASCOT process. This process couples solvent deasphalting with delayed coking to maximize the production of coal-derived liquids while rejecting troublesome solids within the coke drum. A comparison with direct delayed coking is also part of the program. (A preliminary economics assessment of the battery limits of the ASCOT process and direct delayed coking, as incorporated in an integrated processing scheme, will also be undertaken as part of Task 3.)

Experimental Results:

Two asphalt coking tests were completed this quarter to provide a comparison between the ASCOT process and direct delayed coking of the atmospheric residue from Black Thunder coal (completed the previous quarter). The analysis of Wilsonville data also provided a comparison of the ASCOT process with the bottoms product from a vacuum distillation feeding the ROSE process. Solvent deasphalting of the atmospheric residue in tandem with delayed coking of its asphalt product can be considered in direct competition with feeding the vacuum bottoms from a distillation to the ROSE process.

The two asphalt coking tests had a common feedstock, the blended asphalt produced in solvent deasphalting Runs 3039 and 3040, which were made at the same conditions. The blended yields and product characteristics from those runs are given in Table 26. The deasphalted oil yield of 40.2 wt% of the atmospheric residue represents the front end liquid yield from the ASCOT process, to which the liquid yield from coking must be added. The two other solvent deasphalting runs (3034 and 3035) summarized in the table represent candidates for the ASCOT process whose asphalt product could not be adequately fed to the coker because of their high softening points and solids loadings. If their handling problems could be overcome, they would have the potential for a greater overall liquid yield as more liquid product would be removed through non-degrading solvent deasphalting prior to the thermal reactions associated with delayed coking which also produces coke and gas.

Tables 27 and 28 summarize the operating conditions and material balances from each asphalt coking test. The overall ASCOT liquid yield (based on atmospheric residue feed) is also calculated. The two overall liquid yields attained were 63.3 wt% (Run 3047) and 61.5 wt% (Run 3048). The respective liquid yields from coking the asphalt were 23.1 wt% and 21.3 wt% of the atmospheric residue. The liquid yield of 40.2 wt% from solvent deasphalting was common to both runs.

The overall liquid yield from the ASCOT process was significantly higher than the 50 wt% from direct delayed coking of the atmospheric residue (reported in Q-07). Both asphalt coking tests resulted in ASCOT liquid yields above 60 wt% of the atmospheric residue. This firms up the conclusion that this level is readily attainable from Black Thunder coal.

The available product inspections from the coking tests are shown in Tables 29 through 31. The liquid products from the ASCOT process and direct delayed coking were essentially free of ash (≤ 0.05 wt%) and metals (< 1 ppm Ni and V, < 6 ppm Cu and Na). The level of iron was 22 to 170 ppm in the product; this is a small amount considering the atmospheric residue contained 1.6 wt% and there is carbon steel piping present in the pilot plant.

The available ASTM distillations in Tables 29 (coker distillate) and 26 (deasphalted oil) show a high temperature tail (> 850°F) that can be considered as a resid fraction (essentially the bottoms from a vacuum distillation). This fraction was about 40 vol% of the deasphalted oil and 30 vol% of the coker distillate. The theoretical resid fraction (the non-distillable fraction at 600°F and 0.1 mm Hg in the laboratory) defined in the Wilsonville program would be significantly less.

Figure 2 shows a schematic that can be used to integrate the ASCOT process with the coal liquefaction process as operated at Wilsonville. A slip stream of the atmospheric residue would be sent to the ASCOT process (or to direct delayed coking as an option) and the useful products would be recovered using the Wilsonville configured system. ASCOT's solid product (coke and ash) would be adjusted to maintain an acceptable level of solids within the system.

An analysis of Wilsonville's Run 262 data on Black Thunder coal was used in calculating the combined liquid yield from the vacuum tower and ROSE process. This combined yield of 58.6 wt% of the atmospheric residue (27.2% from vacuum distillation and 31.4% from the ROSE process) is the number that the ASCOT process should meet or exceed. Using the atmospheric residue provides more flexibility with ASCOT (solvent deasphalting can cut deeper into the feedstock than a vacuum still) than the ROSE process in tandem with a vacuum tower. Table 32 details the calculation and basis used to derive the combined vacuum tower/ROSE process liquid yield.

The two ASCOT tests produced overall liquid yields (63.3 wt% and 61.5 wt%) that exceeded the combined liquid yields from the vacuum tower and ROSE process. Direct delayed coking of the atmospheric residue fell short. Table 33 summarizes the comparison of yields from the atmospheric residue. To look at the results from another perspective, the ROSE process at Wilsonville (Run 262) produced 41.4 wt% of undesirable solid product. The two ASCOT runs produced 35.3% and 36.1 wt%.

DEVELOPING PROGRAM CONCLUSIONS AND FUTURE WORK

Experimentation is confirming some aspects of the overall process concept, but it is also raising problem areas. Coals can be beneficiated and this should result in decreased solids recovery costs. Black Thunder subbituminous coal can be effectively beneficiated to about 3.5 wt% ash using aqueous sulfurous acid pretreatment. This treated coal can be further beneficiated to about 2 wt% ash using commercially available procedures. All three coals (Black Thunder subbituminous, Burning Star bituminous, and Martin Lake lignite) are effectively swelled by a number of solvents. The most effective solvents are those having hetero-functionality. In addition, a synergistic effect has been demonstrated, in which solvent blends are more effective than the pure solvents alone. Therefore, it will be necessary to use only low levels of swelling agents and yet promote the impregnation of catalyst precursors. The rate of the impregnation of catalyst precursors into swollen coal increases greatly as the effectiveness of the solvent to swell the coal increases. The initial laboratory and continuous flow unit (AU-51L) experiments have indicated that the expected increase in coal conversion and product up-grading did not occur with the selected swelling solvents and catalyst precursor. There appears to be an interaction between the Molyvan L catalyst precursor and the swelling solvent THF that adversely effects coal liquefaction. It is also noted that the most effective swelling solvent, dimethyl sulfoxide, was detrimental to subsequent liquefaction. The flow unit data (AU-44L) indicate that there is little or no advantage for the steps of swelling and/or Molyvan L impregnation of Illinois No. 6 coal. With respect to solids separation and recovery, the solvent system for the deasphalting process has been established. Two ASCOT tests produced overall liquid yields (63.3 wt% and 61.5 wt%) that exceeded the combined liquid yields from the vacuum tower and ROSE process. Direct delayed coking of the atmospheric residue resulted in a lower yield of liquids.

Planned experimentation centers upon (1) carrying out additional micro-autoclave runs with additional catalyst precursors and solvents other than THF to determine if catalyst precursor/solvent interactions can be understood, (2) undertaking a series of Black Thunder coal (raw, swelled and catalyst impregnated) experiments in the two stage flow unit (AU-51L), and completing the solids separation aspects of the program.

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Table 1. Reaction Conditions for Untreated and SO_2 Treated Black Thunder Coals Preswelled with Catalyst

Reaction Conditions	
Coal	Untreated Black Thunder
Temperature	410°C
Agitation	500 cpm
H_2 pressure	1250 psig at ambient
Pyrene	0.67 g
Coal (maf)	~1.33 g
Solvent	2.0 g
Reaction Solvent	1-methylnaphthalene, 80% V1074, and 20% DHA
Catalyst Loading	~600-800 ppm based on maf coal
Reaction Time	30 min
Conditions for Swelling	
Solvents for Preswelling	THF, isopropanol
Amount of Preswelling Solvent	7 g
Catalyst	Mo Naphthenate, Molyvan L, Ni Octoate
Catalyst Charge to Swelling Solvent	Case 1: 1.05 x 600 to 800 ppm of active metal Case 2: 2.0 x 600 to 800 ppm of active metal
Swelling Time	96 hr
Analysis	1. Coal conversion to THF solubles 2. Pyrene conversion to hydrogenated products 3. Catalyst loading (analysis by Amoco)

Table 2. Solvent Uptake by Coal During Swelling

Catalyst	Conditions[a]	Coal Mass (g)	Solvent Mass Absorbed (g)	Catalyst Mass (g)	ID Number
Coal: untreated Reaction Time: 30 min Swelling Solvent: THF Reaction Solvent: 1-MN					
Mo Naphthenate	A	1.6492	1.2374	0.0523	1C
Mo Naphthenate	A	1.6486	1.5141	0.0523	2C
Mo Naphthenate	S	1.6476	1.3772	0.0575	1M
Mo Naphthenate	S	1.6486	1.4938	0.0499	2M
Molyvan L	N	1.6486	1.6040	0.0398	3A
Molyvan L	N	1.6496	1.6048	0.0373	2A
Molyvan L	A	1.6488	1.7701	0.0347	3C
Molyvan L	A	1.6492	1.7366	0.0427	5C
Molyvan L	D	1.6509	1.6187	0.0702	1D
Molyvan L	D	1.6505	1.5540	0.0738	5D
Molyvan L	28h	1.6492	1.6275	0.0353	1H
Molyvan L	28h	1.6505	1.3392	0.0381	3H
Molyvan L	16h	1.6522	1.4365	0.0390	2H
Molyvan L	16h	1.6492	1.3081	0.0406	5H
Molyvan L	6h	1.6502	1.4754	0.0399	4H
Molyvan L	6h	1.6487	1.3771	0.0352	6H
Molyvan L*[b]	1.5s+	1.6465	1.9551	0.0343	5J
Molyvan L*	1.5s+	1.6483	2.2133	0.0378	6J
Molyvan L*	1.5s	1.6479	1.3480	0.0377	NA
Molyvan L*	1.5s	1.6497	1.3826	0.0368	NA
Ni Octoate	N	1.6474	1.5776	0.0268	1A
Ni Octoate	N	1.6478	1.5624	0.0268	4J
Coal: Untreated Reaction Time: 30 min Swelling Solvent: 1-MN+THF Reaction Solvent: 1-MN					
Molyvan L*	1.5s+m	1.6278	1.6087	0.0385	7K
Molyvan L*	1.5s+m	1.6274	1.5201	0.0342	8K

Table 2. Solvent Uptake by Coal During Swelling (Continued)

Catalyst	Conditions[a]	Coal Mass (g)	Solvent Mass Absorbed (g)	Catalyst Mass (g)	ID Number
Coal: untreated	**Reaction Time: 20 min**	**Swelling Solvent: THF**	**Reaction Solvent: 1-MN**		
Mo Naphthenate	A	1.6470	1.2326	0.0476	4C
Mo Naphthenate	A	1.6474	1.7495	0.0500	6C
Molyvan L	N	1.6499	1.7082	0.0352	1E
Molyvan L	N	1.6484	1.5902	0.0358	3E
Ni Octoate	N	1.6532	1.2222	0.0278	3B
Ni Octoate	N	1.6506	1.2391	0.0257	5B
Coal: untreated	**Reaction Time: 10 min**	**Swelling Solvent: THF**	**Reaction Solvent: 1-MN**		
Molyvan L	N	1.6497	1.4850	0.0362	4A
Molyvan L	N	1.6487	1.4663	0.0349	5A
Coal: untreated	**Reaction Time: 30 min**	**Swelling Solvent: Methanol**	**Reaction Solvent: 1-MN**		
Molyvan L	N	1.6474	1.0783	0.0409	1B
Molyvan L	N	1.6470	1.1374	0.0367	2B
Molyvan L	D	1.6457	1.3570	0.0709	4E
Molyvan L	D	1.6487	1.1967	0.0757	5E
Ni Octoate	N	1.6490	0.9481	0.0251	1F
Ni Octoate	N	1.6463	1.0915	0.0242	4F
Coal: untreated	**Reaction Time: 20 min**	**Swelling Solvent: Methanol**	**Reaction Solvent: 1-MN**		
Molyvan L	N	1.6416	1.0018	0.0394	4B
Molyvan L	N	1.6506	0.9803	0.0362	6B
Ni Octoate	N	1.6481	0.8815	0.0228	5F
Ni Octoate	N	1.6450	1.0572	0.0255	6F
Coal: untreated	**Reaction Time: 30 min**	**Swelling Solvent: Isopropanol**	**Reaction Solvent: 1-MN**		
Molyvan L	N	1.6456	1.2749	0.0392	3D
Molyvan L	N	1.6424	1.2210	0.0408	4D
Molyvan L	D	1.6442	1.1892	0.0710	2D
Molyvan L	D	1.6413	1.2599	0.0791	6D

Table 2. Solvent Uptake by Coal During Swelling (Continued)

Catalyst	Conditions[a]	Coal Mass (g)	Solvent Mass Absorbed (g)	Catalyst Mass (g)	ID Number
Ni Octoate	N	1.6435	1.0631	0.0265	2F
Ni Octoate	N	1.6388	1.0936	0.0228	3F
Coal: untreated Reaction Time: 20 min Swelling Solvent: Isopropanol Reaction Solvent: 1-MN					
Molyvan L	N	1.6437	1.1480	0.0352	2E
Molyvan L	N	1.6431	1.1281	0.0353	6E
Ni Octoate	N	1.6461	1.1089	0.0254	1G
Ni Octoate	N	1.6415	1.0320	0.0258	3G
Coal: SO_2 treated Reaction Time: 30 min Swelling Solvent: THF Reaction Solvent: 1-MN					
Molyvan L	N	1.5314	2.1159	0.0344	2G
Molyvan L	N	1.5283	1.8332	0.0356	4G
Ni Octoate	N	1.5305	2.0377	0.0235	5G
Ni Octoate	N	1.5303	1.5671	0.0232	6G
Coal: SO_2 treated Reaction Time: 30 min Swelling Solvent: Methanol Reaction Solvent: 1-MN					
Molyvan L	N	1.5272	1.1793	0.0389	6I
Molyvan L*	N	1.5261	1.3766	0.0358	2J
Ni Octoate	N	1.5243	1.2622	0.0263	2I
Ni Octoate	N	1.5267	1.2300	0.0263	3I
Coal: SO_2 treated Reaction Time: 30 min Swelling Solvent: Isopropanol Reaction Solvent: 1-MN					
Molyvan L	N	1.5231	1.4260	0.0362	4I
Molyvan L	N	1.5278	1.5494	0.0374	5I
Ni Octoate*	N	1.5205	1.1622	0.0251	1J
Ni Octoate*	N	1.5193	1.2226	0.0236	3J
Coal: untreated Reaction Time: 30 min Swelling Solvent: THF Reaction Solvent: V1074					
Molyvan L*	N	1.6502	1.3349	0.0385	1L
Molyvan L*	N	1.6493	1.6361	0.0385	3L
Ni Octoate*	N	1.6491	1.2163	0.0270	7L
Ni Octoate*	N	1.6493	1.3427	0.0270	8L

Table 2. Solvent Uptake by Coal During Swelling (Continued)

Catalyst	Conditions[a]	Coal Mass (g)	Solvent Mass Absorbed (g)	Catalyst Mass (g)	ID Number
Coal: Untreated Reaction Time: 30 min Swelling Solvent: THF Reaction Solvent: DHA					
Molyvan L*	N	1.6487	1.3407	0.0363	5K
Molyvan L*	N	1.6497	1.4845	0.0398	6K
Coal: Untreated Reaction Time: 30 min Swelling Solvent: Isopropanol Reaction Solvent: V1074+DHA					
Molyvan L*	N	1.6456	1.1769	0.0382	1N
Molyvan L*	N	1.6435	0.9408	0.0364	3N
Ni Naphthenate*	N	1.6434	1.1496	0.0545	2N
Ni Naphthenate*	N	1.6449	1.1865	0.0544	5N
Ni Acetylacetonate*	N	1.6449	0.9229	0.0041	4N
Ni Acetylacetonate*	N	1.6451	1.0727	0.0041	6N

[a] A = agitated: Samples were agitated with catalyst for 96 hr during preswelling; S = sulfur was added to the catalyst at 3 times the catalyst mass; N = not agitated 96 hr during preswelling; D = double catalyst loading; 28 h, 16 h, 6 h = hours of catalyst solvent contact during preswelling; 1.5s catalyst impregnated into coal and reacted immediately; 1.5s+ catalyst impregneated into coal, swelled 96 hr in THF, and then reacted; 1.5s+d catalyst impregnated into coal, swelled 96 hr in THF, dried 48 hr, and then reacted; 1.5s+m 1.5 ml of THF solvent swells coal, then new solvent added with catalyst for 96 hr.

[b] *indicates that these experiments were performed this quarter.

Table 3. Coal and Pyrene Conversion for Untreated Black Thunder Coal Reacted in 1-Methylnaphthalene with Catalyst Introduced During Preswelling

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	Added Coal			Pyrene Conversion (mol %)	Coal Conversion (wt %)	Total Recovery (g) [% R]	ID No.
			%ΔV	(g)	wt%[c]				
Reaction Time: 30 min Swelling Solvent: THF									
Mo Naphthenate	NYD	A	31.8	1.6492	NYD	3.3	62.7	NYD	1C
Mo Naphthenate	683	A	38.1	1.6486	30.5	3.6	61.8	4.7470(108.6)	2C
Molyvan L	NYD	N	41.7	1.6486	NYD	13.8	80.8	NYD	3A
Molyvan L	544	N	39.1	1.6496	30.7	16.4	83.0	4.8742(112.0)	2A
Molyvan L	NYD	A	50.0	1.6488	NYD	11.1	75.7	NYD	3C
Molyvan L	NYD	A	54.5	1.6492	NYD	18.2	71.4	NYD	5C
Molyvan L	1143	D	39.1	1.6509	30.5	26.8	84.0	5.2166(119.0)	1D
Molyvan L	NYD	D	42.9	1.6505	NYD	30.3	85.6	NYD	5D
Molyvan L	NYD	28 h	45.5	1.6492	NYD	10.4	74.9	NYD	1H
Molyvan L	NYD	28 h	36.4	1.6505	NYD	7.2	73.3	NYD	3H
Molyvan L	NYD	16 h	42.9	1.6522	NYD	10.8	74.6	NYD	2H
Molyvan L	NYD	16 h	30.1	1.6492	NYD	25.3	81.7	NYD	5H
Molyvan L	NYD	6 h	42.9	1.6502	NYD	20.1	79.6	NYD	4H
Molyvan L	NYD	6 h	40.9	1.6487	NYD	10.3	74.0	NYD	6H
Molyvan L	NYD	1.5s+	52.4	1.6465	NYD	10.1	74.4	NYD	5J
Molyvan L	NYD	1.5s+	59.1	1.6483	NYD	20.7	81.0	NYD	6J
Molyvan L	692	1.5s	17.4	1.6479	30.6	21.0	82.8	5.0823(116.6)	NA
Molyvan L	675	1.5s	22.7	1.6497	30.6	24.5	85.4	5.0098(114.9)	NA
Molyvan L	NYD	1.5s+d	59.1	1.6487	NYD	20.4	71.7	NYD	1K
Molyvan L	NYD	1.5s+d	39.1	1.6493	NYD	22.4	74.0	NYD	4K
Ni Octoate	569	N	34.8	1.6474	30.7	18.8	83.9	5.1322(118.2)	1A
Ni Octoate	NYD	N	42.9	1.6478	NYD	3.3	65.7	NYD	4J
Time: 30 min Swelling Solvent: 1-MN + 1.5 ml THF									
Molyvan L	NYD	1.5s+m	26.1	1.6278	NYD	2.8	50.9	NYD	7K
Molyvan L	NYD	1.5s+m	33.3	1.6274	NYD	3.6	50.6	NYD	8K

Table 3. Coal and Pyrene Conversion for Untreated Black Thunder Coal Reacted in 1-Methylnaphthalene with Catalyst Introduced During Preswelling (Continued)

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	Added Coal			Pyrene Conversion (mol %)	Coal Conversion (wt %)	Total Recovery (g) [% R]	ID No.
			%ΔV	(g)	wt%[c]				
Reaction Time: 20 min Swelling Solvent: THF									
Mo Naphthenate	601	A	28.6	1.6470	30.5	3.0	50.7	4.6187(105.8)	4C
Mo Naphthenate	614	A	45.5	1.6474	30.5	2.3	55.4	5.0585(115.8)	6C
Molyvan L	NYD	N	40.9	1.6499	NYD	7.2	71.3	NYD	1E
Molyvan L	NYD	N	36.4	1.6484	NYD	2.3	63.7	NYD	3E
Ni Octoate	NYD	N	36.4	1.6532	NYD	2.2	58.5	NYD	3B
Ni Octoate	599	N	31.8	1.6506	30.7	2.6	63.0	4.9287(113.4)	5B
Reaction Time: 10 minutes Swelling Solvent: THF									
Molyvan L	NYD	N	40.9	1.6497	NYD	5.4	46.7	NYD	4A
Molyvan L	NYD	N	45.5	1.6487	NYD	6.7	52.0	NYD	5A
Reaction Time: 30 min Swelling Solvent: Methanol									
Molyvan L	NYD	N	21.7	1.6474	NYD	2.4	68.4	NYD	1B
Molyvan L	644	N	28.6	1.6470	30.6	3.6	68.4	4.9238(113.2)	2B
Molyvan L	1261	D	28.6	1.6457	30.3	23.8	89.1	5.2578(119.8)	4E
Molyvan L	NYD	D	23.8	1.6487	NYD	27.6	88.4	NYD	5E
Ni Octoate	NYD	N	22.7	1.6490	NYD	3.2	70.9	NYD	1F
Ni Octoate	653	N	23.8	1.6463	30.7	3.7	69.3	5.0006(115.3)	4F
Reaction Time: 20 min Swelling Solvent: Methanol									
Molyvan L	NYD	N	19.0	1.6416	NYD	3.4	67.6	NYD	4B
Molyvan L	637	N	31.8	1.6506	30.6	2.2	67.5	5.2988(121.6)	6B
Ni Octoate	608	N	23.8	1.6481	30.7	2.6	64.1	4.5322(104.4)	5F
Ni Octoate	617	N	17.4	1.6450	30.7	2.0	64.7	4.8101(110.8)	6F

Table 3. Coal and Pyrene Conversion for Untreated Black Thunder Coal Reacted in 1-Methylnaphthalene with Catalyst Introduced During Preswelling (Continued)

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	Added Coal			Pyrene Conversion (mol %)	Coal Conversion (wt %)	Total Recovery (g) [% R]	ID No.
			%ΔV	(g)	wt%[c]				
Reaction Time: 30 min Swelling Solvent: Isopropanol									
Molyvan L	626	N	17.4	1.6456	30.6	15.2	80.5	4.5367(104.2)	3D
Molyvan L	663	N	19.0	1.6424	30.5	18.5	79.3	5.0069(115.0)	4D
Molyvan L	NYD	D	23.8	1.6442	NYD	27.9	83.1	NYD	2D
Molyvan L	NYD	D	21.7	1.6413	NYD	28.4	80.6	NYD	6D
Ni Octoate	716	N	18.2	1.6435	30.6	4.3	75.8	4.7073(108.4)	2F
Ni Octoate	617	N	13.0	1.6388	30.6	3.4	74.6	4.4727(103.2)	3F
Reaction Time: 20 minutes Swelling Solvent: Isopropanol									
Molyvan L	NYD	N	19.0	1.6437	NYD	3.2	65.4	NYD	2E
Molyvan L	572	N	18.2	1.6431	30.6	5.8	71.0	5.3159(122.2)	6E
Ni Octoate	699	N	13.0	1.6461	30.6	4.0	67.4	4.8985(112.7)	1G
Ni Octoate	NYD	N	18.2	1.6415	NYD	4.6	62.2	NYD	3G

[a] NYD = Not yet determined.

[b] A = agitated: Samples were agitated with catalyst for 96 hr during preswelling; N = not agitated for 96 hr during preswelling; D = double catalyst loading; 28 h, 16 h, 6 h = hours of catalyst solvent contact during preswelling; 1.5 s catalyst impregnated into coal and reacted immediately; 1.5 s+ catalyst impregneated into coal, swelled for 96 hr in THF, and then reacted; 1.5 s+d catalyst impregnated into coal, swelled for 96 hr in THF, dried for 24 hr, and then reacted; 1.5s+m 1.5 ml of THF solvent swells coal, then new solvent added with catalyst for 96 hr.

[c] Coal percentages should be near 31%.

Table 4. Pyrene Product Distribution Using Untreated Black Thunder Coal Reacted in 1-Methylnaphthalene with Catalyst Introduced During Preswelling

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	% HYD[c] (% Coal)[d]	Product Distribution (mole %)				ID No.	% Catalyst Absorbed
				PYR	DHP	THP	HHP		
Reaction Time: 30 min Swelling Solvent: THF									
Mo Naphthenate	NYD	A	1.5 (NYD)	96.7	2.7	0	0.6	1C	NYD
Mo Naphthenate	683	A	2.0 (30.5)	96.4	2.3	0	1.3	2C	95.2
Molyvan L	NYD	N	5.4 (NYD)	86.2	12.4	0.3	1.1	3A	NYD
Molyvan L	544	N	5.9 (30.7)	83.6	15.6	0.3	0.5	2A	79.4
Molyvan L	NYD	A	4.3 (NYD)	88.9	10.0	0.3	0.8	3C	NYD
Molyvan L	NYD	A	7.0 (NYD)	81.8	16.5	0.7	1.0	5C	NYD
Molyvan L	1143	D	10.1(30.5)	73.2	24.2	1.8	0.8	1D	89.2
Molyvan L	NYD	D	11.8(NYD)	69.7	26.3	2.8	1.2	5D	NYD
Molyvan L	NYD	28h	4.3(NYD)	89.6	9.1	0.3	1.0	1H	NYD
Molyvan L	NYD	28h	2.9(NYD)	92.8	6.5	0	0.7	3H	NYD
Molyvan L	NYD	16h	4.2(NYD)	89.2	9.8	0.3	0.7	2H	NYD
Molyvan L	NYD	16h	10.1(NYD)	74.7	21.8	2.1	1.4	5H	NYD
Molyvan L	NYD	6h	7.7(NYD)	79.9	18.0	1.1	1.0	4H	NYD
Molyvan L	NYD	6h	4.1(NYD)	89.7	9.2	0.2	0.9	6H	NYD
Molyvan L	NYD	1.5s+	4.1(NYD)	89.9	9.0	0	1.1	5J	NYD
Molyvan L	NYD	1.5s+	8.0(NYD)	79.3	18.6	0.9	1.2	6J	NYD
Molyvan L	692	1.5s	7.8(30.6)	79.0	19.2	1.1	0.7	NA	NA
Molyvan L	675	1.5s	9.4(30.6)	75.5	21.8	1.8	0.9	NA	NA
Molyvan L	NYD	1.5s+d	8.0(NYD)	79.6	18.1	0.9	1.4	1K	NYD
Molyvan L	NYD	1.5s+d	8.3(NYD)	77.6	20.5	1.3	0.6	4K	NYD
Ni Octoate	NYD	N	1.8 (NYD)	96.7	2.3	0	1.0	4J	NYD
Ni Octoate	569	N	7.6 (30.7)	81.2	16.4	0.9	1.5	1A	76.8
Reaction Time: 30 min Swelling Solvent: 1-MN + 1.5 ml THF									
Molyvan L	NYD	1.5s+m	1.9 (NYD)	97.2	1.4	0.1	1.3	7K	NYD
Molyvan L	NYD	1.5s+m	2.2 (NYD)	96.4	2.1	0.1	1.4	8K	NYD

Table 4. Pyrene Product Distribution Using Untreated Black Thunder Coal Reacted in 1-Methylnaphthalene with Catalyst Introduced During Preswelling (Continued)

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	% HYD[c] (% Coal)[d]	Product Distribution (mole %)				ID No.	Catalyst Absorbed
				PYR	DHP	TH P	HH P		
Reaction Time: 20 min Swelling Solvent: THF									
Mo Naphthenate	601	A	1.8 (30.5)	97.0	1.8	0	1.2	4C	91.8
Mo Naphthenate	614	A	1.5 (30.5)	97.7	1.3	0	1.0	6C	89.4
Molyvan L	NYD	N	3.0(NYD)	92.8	6.2	0.1	0.9	1E	NYD
Molyvan L	NYD	N	1.1(NYD)	97.7	1.7	0.0	0.6	3E	NYD
Ni Octoate	NYD	N	1.2 (NYD)	97.8	1.5	0	0.7	3B	NYD
Ni Octoate	599	N	1.4 (30.7)	97.4	1.8	0	0.8	5B	84.3
Reaction Time: 10 minutes Swelling Solvent: THF									
Molyvan L	NYD	N	2.4 (NYD)	94.6	4.5	0	0.9	4A	NYD
Molyvan L	NYD	N	2.7 (NYD)	93.3	6.0	0	0.7	5A	NYD
Reaction Time: 30 min Swelling Solvent: Methanol									
Molyvan L	NYD*	N	1.3 (NYD)	97.6	1.6	0	0.8	1B	NYD
Molyvan L	644	N	2.0 (30.6)	96.4	2.3	0	1.3	2B	95.3
Molyvan L	1261	D	9.0 (30.3)	76.2	21.6	1.3	0.9	4E	97.7
Molyvan L	NYD	D	10.8 (NYD)	72.4	24.2	1.9	1.5	5E	NYD
Ni Octoate	NYD	N	1.8 (NYD)	96.8	2.1	0.0	1.1	1F	NYD
Ni Octoate	653	N	2.0 (30.7)	96.3	2.5	0.0	1.2	4F	97.5
Reaction Time: 20 min Swelling Solvent: Methanol									
Molyvan L	NYD	N	1.8 (NYD)	96.6	2.5	0	0.9	4B	NYD
Molyvan L	637	N	2.0 (30.6)	97.8	1.2	0	1.0	6B	96.0
Ni Octoate	608	N	1.6 (30.7)	97.4	1.5	0.0	1.1	5F	96.6
Ni Octoate	617	N	1.2 (30.7)	98.0	1.2	0.0	0.8	6F	87.3
Time: 30 min Swelling Solvent: Isopropanol									
Molyvan L	626	N	5.9 (30.6)	84.8	13.6	0.5	1.1	3D	87.0
Molyvan L	663	N	7.1 (30.5)	81.5	16.8	0.6	1.1	4D	88.6
Molyvan L	NYD	D	10.7 (NYD)	72.1	24.5	2.4	0.9	2D	NYD
Molyvan L	NYD	D	11.1 (NYD)	71.6	24.8	2.4	1.2	6D	NYD

Table 4. Pyrene Product Distribution Using Untreated Black Thunder Coal Reacted in 1-Methylnaphthalene with Catalyst Introduced During Preswelling (Continued)

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	% HYD[c] (% Coal)[d]	Product Distribution (mole %)				ID No.	Catalyst Absorbed
				PYR	DHP	THP	HHP		
Time: 20 min Swelling Solvent: Isopropanol									
Ni Octoate	716	N	2.1 (30.6)	95.7	3.3	0	1.0	2F	97.8
Ni Octoate	617	N	1.7 (30.6)	96.6	2.5	0	0.9	3F	97.7
Molyvan L	NYD	N	1.4 (NYD)	96.8	2.7	0	0.5	2	NYD
Molyvan L	572	N	2.4 (30.6)	94.2	5.2	0.1	0.5	6	88.1
Ni Octoate	699	N	2.1 (30.6)	96 .	2.8	0	1.2	1G	99.5
Ni Octoate	NYD	N	2.2 (NYD)	95.4	3.6	0	1	3G	NYD

[a] NYD = Not yet determined.

[b] A = agitated: Samples were agitated with catalyst for 96 hr during preswelling; N = not agitated for 96 hr during preswelling; D = double catalyst; 28 h, 16 h, 6 h = hours of catalyst solvent contact during preswelling; 1.5 s catalyst impregnated into coal and reacted immediately; 1.5 s+ catalyst impregneated into coal, swelled for 96 hr in THF, and then reacted; 1.5 s+d catalyst impregnated into coal, swelled for 96 hr in THF, dried for 24 hr, and then reacted; 1.5s+m 1.5 ml of THF solvent swells coal, then new solvent added with catalyst for 96 hr.

[c] % HYD = % hydrogenation.

[d] Coal percentages should be near 31%.

Table 5. Comparison of Coal and Pyrene Conversions With Untreated Black Thunder Coal With Different Catalyst Introduction Methods

Catalyst	Conditions	% Change in Coal Conversion[a] (weight %)	% Change in Pyrene Conversion[a] (mole %)	Swelling Index (%ΔV)
Coal: untreated Swelling Solvent: THF				
Molyvan L	N	-6.5	-4.8	41.7
Molyvan L	N	-4.3	-2.2	39.1
Molyvan L	A	-11.6	-7.5	50.0
Molyvan L	A	-15.9	-0.4	54.5
Molyvan L	D	-3.3	+8.2	39.1
Molyvan L	D	-1.7	+11.7	42.9
Molyvan L	28h	-12.4	-8.2	45.5
Molyvan L	28h	-14.0	-11.4	36.4
Molyvan L	16h	-12.7	-7.8	42.9
Molyvan L	16h	-5.6	+6.7	30.1
Molyvan L	6h	-7.7	+1.5	42.9
Molyvan L	6h	-13.3	-8.3	40.9
Molyvan L	1.5s+	-12.9	-8.5	52.4
Molyvan L	1.5s+	-6.3	+2.1	59.1
Molyvan L	1.5s	-4.5	+2.4	17.4
Molyvan L	1.5s	-1.9	+5.9	22.7
Molyvan L	1.5s+d	-15.6	+1.8	59.1
Molyvan L	1.5s+d	-13.3	+3.8	39.1
Ni Octoate	N	+6.9	+16.4	34.8
Ni Octoate	N	-11.3	+0.9	42.9
Coal: Untreated Swelling Solvent: 1-methylnaphthalene + 1.5 ml THF				
Molyvan L	1.5s+m	-36.4	-15.8	26.1
Molyvan L	1.5s+m	-36.7	-15.0	33.3

Table 5. Comparison of Coal and Pyrene Conversions With Untreated Black Thunder Coal With Different Catalyst Introduction Methods (Continued)

Catalyst	Conditions	% Change in Coal Conversion[a] (weight %)	% Change in Pyrene Conversion[a] (mole %)	Swelling Index (%ΔV)
Coal: untreated	**Swelling Solvent: Methanol**			
Molyvan L	N	-19.6	-21.9	21.7
Molyvan L	N	-19.6	-20.7	28.6
Molyvan L	D	+1.1	-0.5	28.6
Molyvan L	D	+0.4	+3.3	23.8
Ni Octoate	N	-14.6	-7.7	22.7
Ni Octoate	N	-16.2	-7.2	23.8
Coal: untreated	**Swelling Solvent: Isopropanol**			
Molyvan L	N	-7.6	-3.5	17.4
Molyvan L	N	-8.8	-0.2	19.0
Molyvan L	D	-5.0	+9.2	23.8
Molyvan L	D	-7.5	+9.7	21.7
Ni Octoate	N	-1.8	+2.1	18.2
Ni Octoate	N	-3.0	+1.2	13.0

[a] % change in conversion is the difference between the percentage of conversion when the catalyst was added directly to the reactor and the percentage conversion when the catalyst was added to the preswelling solvent.

[b] A = agitated: Samples were agitated with catalyst for 96 hr during preswelling; N = not agitated for 96 hr during preswelling; D = double catalyst loading; 28 h, 16 h, 6 h = hours of catalyst solvent contact during preswelling; 1.5 s catalyst impregnated into coal and reacted immediately; 1.5 s+ catalyst impregneated into coal, swelled for 96 hr in THF, and then reacted; 1.5 s+d catalyst impregnated into coal, swelled for 96 hr in THF, dried for 24 hr, and then reacted; 1.5 s+d catalyst impregnated into coal, swelled for 96 hr in THF, dried for 24 hr, and then reacted; 1.5s+m 1.5 ml of THF solvent swells coal, then new solvent added with catalyst for 96 hr.

Table 6. Effect of Swelling on Catalytic Activity of Molyvan L

Catalyst Type	Catalyst Loading (ppm)	Added Coal			Conversion		Total Recovery (g)[%R]	ID No.
		%ΔV	(g)	wt%	Pyrene (mol%)	Coal (wt%)		
Time: 30 min Catalyst: Added directly to reactor								
Molyvan	684	36.4	1.6485	30.6	17.7	86.2	4.2172 [96.6]	NA[a]
Molyvan	638	39.1	1.6493	30.7	19.5	88.4	4.0010 [91.9]	NA
Time: 30 min Catalyst: Placed in THF, then separated and placed into reactor								
Molyvan	672	36.4	1.6489	30.6	25.8	86.4	4.9971 [114.6]	NA
Molyvan	639	30.4	1.6486	30.6	19.9	83.9	4.5592 [104.7]	NA

[a] NA = not applicable.

Table 7. Pyrene Product Distribution from Reactions Evaluating the Catalytic Activity of Molyvan L

Catalyst Type	Catalyst Loading (ppm)	%H (% coal)	Product Distribution (mol%)				% Catalyst Absorbed	ID No.
			PYR	DHP	THP	HHP		
Time: 30 min Catalyst: Added directly to reactor								
Molyvan	684	5.9(30.6)	82.3	17.7	0	0	NA[a]	NA
Molyvan	638	6.5(30.7)	80.5	19.5	0	0	NA	NA
Time: 30 min Catalyst: Placed in THF, then separated and placed into reactor								
Molyvan	672	10.1(30.6)	74.2	22.3	2.2	1.3	NA	NA
Molyvan	639	7.9(30.6)	80.1	17.3	1.3	1.3	NA	NA

[a] NA= not applicable.

Table 8. Coal and Pyrene Conversions for Reaction Systems with Untreated Black Thunder Coal Using 1-Methylnaphthalene as the Reaction Solvent and Mo Naphthenate as Catalyst

Catalyst Type	Catalyst Loading (ppm)	Added Coal			Conversion		Total Recovery (g) [%R]	ID No.
		%ΔV	(g)	wt%	Pyrene (mol%)	Coal (wt%)		
Compound Used: Sulfur Swelling Solvent: THF Catalyst: In Reactor								
Mo Naphthenate	644	31.8	1.6490	29.5	25.4	89.0	5.1941[114.8]	NA
Mo Naphthenate	673	40.9	1.6490	29.5	24.2	90.7	5.0647[112.0]	NA
Compound Used: Sulfur Swelling Solvent: THF Catalyst: In Swelling Solvent								
Mo Naphthenate	NYD[a]	27.3	1.6476	NYD	25.6	89.8	NYD[NYD]	1M
Mo Naphthenate	NYD	42.9	1.6486	NYD	26.3	89.9	NYD[NYD]	2M
Compound Used: Sulfur + Carbon Black Swelling Solvent: THF Catalyst: In Reactor								
Mo Naphthenate	590	31.8	1.6499	28.6	7.6	77.5	5.0374[107.9]	NA
Mo Naphthenate	695	30.4	1.6477	28.5	24.0	78.8	5.0762[108.5]	NA

[a] NYD = Not yet determined.

Table 9. Pyrene Product Distribution in Reactions of Untreated Black Thunder Coal Using 1-Methylnaphthalene as Reaction Solvent and Mo Naphthenate as Catalyst

Catalyst Types	Catalyst Loading (ppm)	%H (% Coal)	Product Distribution (mol%)				% Catalyst Absorbed	ID No.
			PYR	DHP	THP	HHP		
Compound Used: Sulfur Swelling Solvent: THF Catalyst: In Reactor								
Mo Naphthenate	644	10.3(29.5)	74.6	21.6	2.1	1.7	NA	NA
Mo Naphthenate	673	10.0(29.5)	75.8	20.4	1.9	1.9	NA	NA
Compound Used: Sulfur Swelling Solvent: THF Catalyst: In Swelling Solvent								
Mo Naphthenate	NYD	10.5(NYD)	74.4	21.5	2.2	1.9	NYD	1M
Mo Naphthenate	NYD	10.8(NYD)	73.7	22.1	2.3	1.9	NYD	2M
Compound Used: Sulfur + Carbon Black Swelling Solvent: THF Catalyst: In Reactor								
Mo Naphthenate	590	3.2(28.6)	92.4	6.5	0.1	1.0	NA	NA
Mo Naphthenate	695	9.8(28.5)	76.0	20.3	1.9	1.8	NA	NA

[a] NA = not applicable. [b] NYD = not yet determined. [c] Coal percentages should be near 31%.

Table 10. Coal and Pyrene Conversions for Reaction Systems With Untreated Black Thunder Coal Using Low Levels of Catalysts

Catalyst Type	Catalyst Loading (ppm)[a]	Added Coal			Conversion		Total Recovery (g)[%R]	ID No.
		%ΔV	(g)	wt%	Pyrene (mol%)	Coal (wt%)		
Swelling Solvent: THF				Catalyst: In Swelling Solvent				
Molyvan L	100	30.4	1.6489	30.8	2.1	52.7	4.9440 [114.3]	3M
Molyvan L	153	40.9	1.6500	30.8	2.1	54.6	4.3899 [101.4]	4M
Swelling Solvent: Methanol				Catalyst: In Swelling Solvent				
Molyvan L	107	21.7	1.6467	30.8	1.8	51.5	4.7449 [109.7]	5M
Molyvan L	122	23.8	1.6475	30.8	2.4	54.7	4.7348 [101.2]	6M
Swelling Solvent: Isopropanol				Catalyst: In Swelling Solvent				
Molyvan L	93	13.6	1.6441	30.8	1.5	48.8	4.3229 [100.1]	7M
Molyvan L	139	18.2	1.6422	30.8	2.0	54.4	4.5130 [104.5]	8M

[a] Values obtained by assuming 91.8% of the catalyst adsorbed into the coal.

Table 11. Pyrene Product Distributions in Reactions of Untreated Black Thunder Coal Using Low Levels of Catalyst

Catalyst Type	Catalyst Loading (ppm)[a]	%H (% coal)	Product Distribution (mol%)				ID No.
			PYR	DHP	THP	HHP	
Swelling Solvent: THF Catalyst: In Swelling Solvent							
Molyvan L	100	1.3(30.8)	97.9	1.3	0	0.8	3M
Molyvan L	153	1.3(30.8)	97.9	1.2	0	0.9	4M
Swelling Solvent: THF Catalyst: In Swelling Solvent							
Molyvan L	107	1.1(30.8)	98.2	1.0	0	0.8	5M
Molyvan L	122	1.4(30.8)	97.6	1.6	0	0.8	6M
Swelling Solvent: Isopropanol Catalyst: In Swelling Solvent							
Molyvan L	93	1.1(30.8)	98.5	0.6	0	0.9	7M
Molyvan L	139	1.2(30.8)	98.0	1.2	0	0.8	8M

[a] Values obtained by assuming 91.8% of the catalyst absorbed into the coal.

Table 12. Coal and Pyrene Conversions for Untreated Black Thunder Coal Reacted in V1074

Catalyst Type	Catalyst Loading (ppm)	Added Coal			Conversion		Total Recovery (g) [%R]	ID No.
		%ΔV	(g)	wt%	Pyrene (mol%)	Coal (wt%)		
Reaction Time: 30 min Swelling Solvent: THF								
Molyvan L[a]	710	NA	1.6495	29.8	27.8	84.2	5.4211 [121.31]	NA
Molyvan L[a]	629	NA	1.6485	30.5	28.2	81.4	5.5136 [125.9]	NA
Reaction Time: 30 min Swelling Solvent: THF								
Molyvan L[b]	NYD	39.1	1.6502	NYD	12.5	70.3	NYD [NYD]	1L
Molyvan L[b]	NYD	37.5	1.6493	NYD	15.7	81.7	NYD [NYD]	3L

[a] Catalyst added directly to reactor.

[b] Catalyst preswelled with coal for 96 hr.

Table 13. Pyrene Product Distribution Using Untreated Black Thunder Coal With V1074 as the Reaction Solvent

Catalyst	Catalyst (ppm)	%H (% Coal)	Product Distribution (mol%)				% Catalyst Absorbed	ID No.
			PYR	DHP	THP	HHP		
Time: 30 Minutes Swelling Solvent: THF								
Molyvan L[a]	710	11.6(29.8)	72.2	23.1	2.4	2.3	NA	NA
Molyvan L[a]	629	12.0(30.5)	71.8	23.2	2.4	2.6	NA	NA
Time: 30 Minutes Swelling Solvent: THF								
Molyvan L[b]	NYD	5.5(NYD)	87.5	10.1	0.7	1.7	NYD	1L
Molyvan L[b]	NYD	6.7(NYD)	84.3	13.2	0.6	1.9	NYD	3L

[a] Catalyst added directly to reactor.

[b] Catalyst preswelled with coal for 96 hr.

Table 14. Coal and Pyrene Conversion for SO_2 Treated Black Thunder Coal Reacted in1-MN with Catalyst Introduced During Preswelling

Catalyst Type	Catalyst Loading (ppm)[a]	Conditions[b]	Added Coal			Pyrene Conversion (mol %)	Coal Conversion (wt %)	Total Recovery (g) [% R]	ID No.
			%ΔV	(g)	wt%[c]				
Reaction Time: 30 min Swelling Solvent: THF									
Molyvan L	NYD	N	88.9	1.5314	NYD	30.8	87.5	NYD	2G
Molyvan L	638	N	63.2	1.5283	31.3	31.9	84.4	4 .4536(105.3)	4G
Ni Octoate	659	N	77.8	1.5305	31.4	7.7	74.3	4.9130(116.3)	5G
Ni Octoate	NYD	N	40.0	1.5303	NYD	6.0	68.5	NYD	6G
Reaction Time: 30 min Swelling Solvent: Methanol									
Molyvan L	NYD	N	40.0	1.5272	NYD	28.7	89.1	NYD	6I
Molyvan L	NYD	N	47.4	1.5261	NYD	24.8	81.0	NYD	2J
Ni Octoate	NYD	N	36.8	1.5243	NYD	6.7	78.5	NYD	2I
Ni Octoate	NYD	N	30.0	1.5267	NYD	7.4	78.2	NYD	3I
Reaction Time: 30 min Swelling Solvent Isopropanol									
Molyvan L	NYD	N	33.3	1.5231	NYD	25.8	86.9	NYD	4I
Molyvan L	NYD	N	31.6	1.5278	NYD	26.1	88.3	NYD	5I
Ni Octoate	NYD	N	25.0	1.5205	NYD	4.8	69.5	NYD	1J
Ni Octoate	NYD	N	25.0	1.5193	NYD	5.1	66.3	NYD	3J

[a] NYD = Not yet determined.

[b] A = Agitated during preswelling; N = not agitated.

[c] Coal percentage should be near 31%.

Table 15. Comparison of Coal and Pyrene Conversions With SO_2 Treated Black Thunder Coal With Different Catalyst Introduction Methods

Catalyst	Agitated (A) Not Agitated (N) Double Catalyst (D)	% Change in Coal Conversion (weight %)	% Change in Pyrene Conversion (mole %)	Swelling Index (%ΔV)
Coal: SO_2 Treated Swelling Solvent: THF				
Molyvan L	N	-3.6	+5.4	88.9
Molyvan L	N	-6.7	+6.5	63.2
Ni Octoate	N	+1.8	+5.5	77.8
Ni Octoate	N	-4.0	+3.8	40.0
Coal: SO_2 Treated Swelling Solvent: Methanol				
Molyvan L	N	-0.9	-1.3	40.0
Molyvan L	N	-9.0	-5.2	47.4
Ni Octoate	N	-0.2	+1.8	36.8
Ni Octoate	N	-0.5	+2.5	30.0
Coal: So_2 Treated Swelling Solvent: Isopropanol				
Molyvan L	N	-4.4	+4.5	33.3
Molyvan L	N	-3.0	+4.8	31.6
Ni Octoate	N	-4.4	+0.9	25.0
Ni Octoate	N	-7.6	+1.2	25.0

Table 16. Coal and Pyrene Conversions for Untreated Black Thunder Coal Reacted in V1074

Catalyst Type	Catalyst Loading (ppm)	Added Coal			Conversion		Total Recovery (g) [%R]	ID No.
		%ΔV	(g)	wt%	Pyrene (mol%)	Coal (wt%)		
Reaction Time: 30 min Swelling Solvent: None								
Molyvan L	664	NA[a]	1.6416	30.3	12.9	85.4	4.1794[95.4]	NA
Molyvan L	639	NA	1.6410	30.2	16.8	84.0	4.4870[102.1]	NA
Ni Octoate	647	NA	1.6433	30.6	1.9	82.3	3.8885[89.6]	NA
Ni Octoate	646	NA	1.6433	30.6	1.5	74.6	4.3763[100.7]	NA
Ni Octoate	730	NA	1.6430	30.5	10.1	80.6	4.5728[104.9]	NA
Ni Octoate	650	NA	1.6437	30.6	8.5	81.7	4.3199[99.5]	NA
Reaction Time: 30 min Swelling Solvent: THF								
None	0	22.7	1.6482	30.7	5.4	73.2	5.0754[116.7]	NA
None	0	27.3	1.6486	30.7	5.2	70.8	5.0906[117.3]	NA
Reaction Time: 30 min Swelling Solvent: THF Catalyst Placed in Reactor								
Molyvan L	710	NA	1.6495	29.8	27.8	84.2	5.4211[121.31]	NA
Molyvan L	629	NA	1.6485	30.5	28.2	81.4	5.5136[125.9]	NA
Ni Octoate	660	31.8	1.6493	30.3	6.4	77.2	5.2084[118.4]	NA
Ni Octoate	666	38.1	1.6483	30.6	6.4	75.9	5.4349[124.7]	NA
Reaction Time: 30 min Swelling Solvent: THF Catalyst Placed in Swelling Solvent								
Molyvan L	NYD[b]	39.1	1.6502	NYD[c]	12.5	70.3	NYD[NYD]	1L
Molyvan L	NYD	37.5	1.6493	NYD	15.7	81.7	NYD[NYD]	3L
Ni Octoate	NYD	27.3	1.6491	NYD	3.5	77.5	NYD[NYD]	7L
Ni Octoate	NYD	26.1	1.6493	NYD	5.6	79.9	NYD[NYD]	8L

[a] NA = not applicable.

[b] NYD = not yet determined.

[c] Coal percentages should be near 31%.

Table 17. Pyrene Product Distribution Using Untreated Black Thunder Coal With V1074 as the Reaction Solvent

Catalyst	Catalyst (ppm)	%H (% Coal)	Product Distribution (mol%)				% Catalyst Absorbed	ID No.
			PYR	DHP	THP	HHP		
Reaction Time: 30 min Swelling Solvent: None								
Molyvan L	664	4.9(30.3)	87.1	11.7	0.6	0.6	NA[a]	NA
Molyvan L	639	6.4(30.2)	83.2	15.2	0.8	0.8	NA	NA
Ni Octoate	647	0.6(30.6)	98.1	1.9	0	0	NA	NA
Ni Octoate	646	0.6(30.6)	98.5	1.2	0.3	0	NA	NA
Ni Octoate	730	3.6(30.5)	89.9	9.3	0.8	0	NA	NA
Ni Octoate	650	30.0(30.6)	91.5	8.0	0.5	0	NA	NA
Reaction Time: 30 min Swelling Solvent: THF								
None	0	3.1(30.7)	94.6	3.2	0.5	1.7	NA	NA
None	0	2.9(30.7)	94.8	3.2	0.4	1.8	NA	NA
Reaction Time: 30 min Swelling Solvent: THF Catalyst: Placed in Reactor								
Molyvan L	710	11.6(29.8)	72.2	23.1	2.4	2.3	NA	NA
Molyvan L	629	12.0(30.5)	71.8	23.2	2.4	2.6	NA	NA
Ni Octoate	660	3.2(30.3)	93.6	4.6	0.4	1.4	NA	NA
Ni Octoate	629	3.3(30.6)	93.6	4.5	90.4	1.6	NA	NA
Reaction Time: 30 min Swelling Solvent: THF Catalyst: Placed in Swelling Solvent								
Molyvan L	NYD[b]	5.5(NYD)[c]	87.5	10.1	0.7	1.7	NYD	1L
Molyvan L	NYD	6.7(NYD)	84.3	13.2	0.6	1.9	NYD	3L
Ni Octoate	NYD	2.0(NYD)	96.5	2.0	0.4	1.1	NYD	7L
Ni Octoate	NYD	3.0(NYD)	94.4	3.6	0.4	1.6	NYD	8L

[a] NA = not applicable.
[b] NYD = not yet determined.
[c] Coal percentages should be near 31%.

Table 18. Pyrene Distributions for Untreated Black Thunder Coal Reactions Using Dihydroanthracene as the Reaction Solvent

Catalyst Type	Catalyst Loading (ppm)	%H (% coal)	Product Distribution (mol%)				% Catalyst Absorbed	ID No.
			PYR	DHP	THP	HHP		
Time: 30 min Swelling Solvent: None								
Molyvan L	693	6.6(30.5)	81.6	17.4	0.5	0.5	NA[a]	NA
Molyvan L	660	4.1(30.5)	88.0	11.6	0.4	0.0	NA	NA
Time: 30 min Swelling Solvent: THF								
None	0	5.9(30.9)	90.9	3.1	3.3	2.7	NA	NA
None	0	6.1(30.8)	90.6	3.3	3.2	2.9	NA	NA
Time: 30 min Swelling Solvent: THF Catalyst: Placed in reactor								
Molyvan L	635	8.1(30.6)	79.9	17.5	1.2	1.4	NA	NA
Molyvan L	681	10.7(30.6)	73.2	23.1	2.0	1.8	NA	NA
Time: 30 min Swelling Solvent: THF Catalyst: Placed in swelling solvent								
Molyvan L	NYD[b]	4.0(NYD)[c]	92.8	3.9	1.7	1.6	NYD	5K
Molyvan L	NYD	5.1(NYD)	89.5	7.6	1.1	1.8	NYD	6K

[a] NA = not applicable.

[b] NYD = not yet determined.

[c] Coal percentages should be near 31%.

Table 19. Coal and Pyrene Conversions for Reaction Systems with Untreated Black Thunder Coal Using DHA and V1074 as Reaction Solvent and Three Different Catalysts

Catalyst Type	Catalyst Loading (ppm)[a]	Added Coal			Conversion		Total Recovery (g)[%R]	ID No.
		%ΔV	(g)	wt%	Pyrene (mol%)	Coal (wt%)		
Swelling Solvent: Isopropanol Catalyst: In Swelling Solvent								
Molyvan L	645	22.7	1.6456	30.6	6.5	80.4	5.0317 [115.7]	1N
Molyvan L	607	17.4	1.6435	30.2	8.8	81.9	5.0707 [115.3]	3N
Ni Naphthenate	682	22.7	1.6434	30.2	11.9	86.5	5.1436[117.0]	2N
Ni Naphthenate	682	28.6	1.6449	30.3	10.9	84.4	5.3514[121.9]	5N
Ni Acetylacetonate	201	13.6	1.6449	30.8	5.2	77.0	5.0325[116.6]	4N
Ni Acetylacetonate	201	18.2	1.6451	30.8	5.2	77.0	4.9776[115.2]	6N

[a] Values obtained by assuming 91.8% of the catalyst absorbed into the coal.

Table 20. Pyrene Product Distribution in Reactions of Untreated Black Thunder Coal Using DHA and V1074 as the Reaction Solvent

Catalyst Type	Catalyst Loading (ppm)[a]	%H (% coal)	Product Distribution (mol%)				ID No.
			PYR	DHP	THP	HHP	
Swelling Solvent: Isopropanol Catalyst: In Swelling Solvent							
Molyvan L	645	3.1(30.6)	93.5	4.8	0.4	1.3	1N
Molyvan L	607	4.0(30.2)	91.2	7.0	0.4	1.4	3N
Ni Naphthenate	682	5.2(30.2)	88.1	9.8	0.5	1.6	2N
Ni Naphthenate	682	4.9(30.3)	89.1	8.7	0.4	1.8	5N
Ni Acetylacetonate	201	2.9(30.8)	94.8	3.1	0.6	1.5	4N
Ni Acetylacetonate	201	3.0(30.8)	94.8	2.9	0.7	1.6	6N

[a] Values obtained by assuming 91.8% of the catalyst absorbed into the coal.

Table 21. Coal and Pyrene Conversions for Treated Wyodak Coal

Coal Treatment	Added Coal		Pyrene Conversion (mol%)	Coal Conversion (mol%)	Total Recovery (g) [%R]
	(g)	wt%			
Solvent: 9,10 dihydroanthracene					
None	2.0297	28.3	4.4	85.6	6.7280[143.0]
None	2.0287	28.3	4.4	88.3	6.3865[135.8]
Solvent: V1074					
None	2.0287	28.1	4.0	84.4	5.3686[113.3]
None	3.0009	34.7	2.9	87.5	6.3743[112.2]
Solvent: 1-Methylnaphthalene					
None	2.0286	28.3	2.3	71.4	4.9975[106.3]
None	2.0277	28.3	1.8	71.5	4.9101[104.6]
Solvent: 1-Methylnaphthalene					
Iron*	2.0344	28.4	3.3	49.5	4.6773[99.4]
Iron*	2.0348	28.3	3.3	45.2	4.6777[99.3]
Solvent: 1-Methylnaphthalene					
Cobalt	2.0372	28.4	2.3	45.8	4.6783[99.4]
Cobalt	2.0358	28.4	2.3	47.9	4.6769[99.4]
Solvent: 1-Methylnaphthalene					
Molybdenum	2.0357	28.4	33.9	86.3	5.7820[122.8]
Molybdenum	2.0351	28.4	34.8	90.3	5.8877[125.0]
Solvent: 1-Methylnaphthalene					
Nickel	2.0352	28.4	5.1	62.2	4.9751[105.8]
Nickel	2.0359	28.4	5.0	59.1	4.9090[104.3]
Solvent: 1-Methylnaphthalene					
Acid Washed	2.0382	28.4	3.1	40.6	4.9198[104.4]
Acid Washed	2.0365	28.4	3.2	42.6	4.8961[104.1]

Table 22. Pyrene Product Distribution for Wyodak Coal

Coal Treatment	%N (% Coal)	Product Distribution (mol%)			
		PYR	DHP	THP	HHP
Solvent: 9,10 dihydroanthracene					
None	2.4(28.3}	95.6	2.7	0.7	1.0
None	2.6(28.3)	95.6	2.3	0.7	1.4
Solvent: V1074					
None	1.9(28.1)	96.0	2.9	0.3	0.8
None	1.4(34.7)	97.1	2.2	0.2	0.5
Solvent: 1-Methylnaphthalene					
None	1.3(28.3)	97.7	1.5	0	0.8
None	1.1(28.3)	98.2	1.0	0	0.8
Solvent: 1-Methylnaphthalene					
Iron	1.8(28.4)	96.7	2.0	0.3	1.0
Iron	1.8(28.3)	96.7	2.0	0.3	1.0
Solvent: 1-Methylnaphthalene					
Cobalt	1.4(28.4)	97.7	1.3	0.1	0.9
Cobalt	1.4(28.4)	97.7	1.3	0.1	0.9
Solvent: 1-Methylnaphthalene					
Molybdenum	14.0(28.4)	66.1	28.2	3.4	2.3
Molybdenum	14.3(28.4)	65.2	29.0	3.6	2.2
Solvent: 1-Methylnaphthalene					
Nickel	2.8(28.4)	94.9	3.4	0.2	1.5
Nickel	2.7(28.4)	95.0	3.3	0.1	1.6
Solvent: 1-Methylnaphthalene					
Acid Washed	2.1(28.4)	96.9	1.5	0.1	1.5
Acid Washed	2.2(28.4)	96.8	1.5	0.2	1.5

Table 23

Summary of AU-44 Operating Data
Illinois No. 6 Runs
(Swollen and Molyvan-L Series)
(AU-44 Unit Runs for DOE 91PC91051)

Run Number:	16103-197	16103-198-I	16103-198-II	18119-01	18119-04	18119-05
Slurry Feed:						
Coal	Raw IL	Raw IL	Raw IL	27.74% Coal	27.74% Coal	27.74% Coal
Liq'n Solvent	V1074	V1074	V1074	55.39% V1074	55.39% V1074	55.39% V1074
Sw Solv. Toluen	None	None	None	13.49%Tol	13.49%Tol	13.49%Tol
IPA	None	None	None	3.48% IPA	3.48% IPA	3.48% IPA
MV-L: To Swellin	None	None	None	None	100 ppm	100 ppm
To Feed	None	100 ppm	100 ppm	None	None	100 ppm
Operating Data:						
Initial Date	6/30/93	6/30/93	7/01/93	7/19/93	7/20/93	7/20/93
Final Date	6/30/93	7/01/93	7/01/93	7/19/93	7/20/93	7/20/93
Initial Time	1530	2315	1135	1401	1300	1800
Final Time	1832	0730	1530	1701	1600	2100
Run Time, Hr	3.03	8.25	3.92	3.00	3.00	3.00
Initial Feed Scale	35.42	43.00	25.50	17.40	42.64	36.44
Final	30.50	29.86	21.56	14.36	38.96	32.62
Feed Slurry, Lbs	4.92	13.14	3.94	3.04	3.68	3.82
Product weights						
Receiver, Lbs	4.82	12.69	3.72	2.35	3.19	3.17
Slurry Sample, L	4.36	12.61	2.38	2.23	3.19	3.37
Light Ends, g	2.80	7.15	7.70	—	—	—
Water, g	12.50	28.23	26.60	—	—	—
Feed Rate, Lb/Hr	1.64	1.59	1.01	1.01	1.23	1.27
Space Time, Min	22.2	22.7	35.9	35.6	29.4	28.3
Reactor Temp, F	824.00	823.00	825.00	826.00	824.00	825.00
Furnace Temp, F	992.00	997.00	965.00	953.00	1008.00	1001.00
Reactor Press, psig	1946	1961	1972	1981	1959	1954
Room Temp, F	72	73	75	86	82	82
Wet Test Meter, cc	1712.00	5509.00	2821.00	4776.00	2227.00	2214.00
Brooks H2 Gas, scf	14.64	14.92	15.03	15.05	14.96	15.05

Table 24

Summary of Extraction Results
Illinois No. 6 Runs at 825F
(Swollen and Molyvan-L Series)
(AU-44 Unit Runs for DOE 91PC91051)

Run Number:	16103-197 Average	16103-198-I Average	16103-198-II Average	18119-01 Average	18119-04 Average	18119-05 Average	16103-198 Fd Average
Slurry Feed:	IL No.6	IL No.6	IL No.6	Swollen IL No.6	Swollen IL No.6	Swollen IL No.6	IL No.6
	V1074	V1074	V1074	V1074	V1074	V1074	V1074
	Non-Cat	100ppm MVL	100ppm MVL	Non-Cat	W/ 100ppm MVL	W/100+100 MVL	100ppm MVL
	1.616 #/Hr	1.593 #/Hr	1.006 #/Hr	1.013 #/Hr	1.227 #/Hr	1.273 #/Hr	----
	22 Min.	23 Min.	36 Min.	36 Min.	29 Min.	28 Min.	
Extraction Data:							
THF Ins:	7.90	7.25	5.89	7.24	7.16	7.62	28.88
Toluene Ins:	12.25	11.77	8.83	10.37	10.67	10.96	31.05
Hexane Ins:	23.63	22.43	19.11	20.83	20.95	22.06	31.73
Pre-asph:	4.35	4.52	2.95	3.14	3.52	3.34	2.18
Asph:	11.38	10.65	10.28	10.46	10.28	11.10	0.67
Oils:	76.37	77.57	80.89	79.17	79.05	77.94	68.27
Extraction Yields: (MAF Coal basis)							
Conversion (THF Sol)	83.30	85.90	91.10	87.20	84.50	84.00	
Conversion (Tol Sol)	68.00	70.20	81.10	76.80	71.50	72.20	

Table 25

Summary of Stream Analyses
Illinois No. 6 Runs
(Swollen and Molyvan-L Series)
(AU-44 Unit Runs for DOE 91PC91051)

Run Number:	16103-197	16103-198-I	16103-198-II	18119-01	18119-04	18119-05	16103-198 Fd
Slurry Feed:	IL No.6 V1074 Non-Cat 1.616 #/Hr 22 Min.	IL No.6 V1074 100ppm MVL 1.593 #/Hr 23 Min.	IL No.6 V1074 100ppm MVL 1.006 #/Hr 36 Min.	Swollen IL No.6 V1074 Non-Cat 1.013 #/Hr 36 Min.	Swollen IL No.6 V1074 W/ 100ppm MVL 1.227 #/Hr 29 Min.	Swollen IL No.6 V1074 W/100+100 MVL 1.273 #/Hr 28 Min.	IL No.6 V1074 100ppm MVL ----
Analyses, wt%:							
THF Insolubles							
Carbon	43.22	42.72	44.35	50.48	50.66	43.11	65.91
Hydrogen	2.19	2.36	2.44	3.06	3.08	2.71	4.74
Nitrogen	0.77	1.01	1.12	1.06	1.40	1.02	1.05
Oxygen	9.19	9.31	7.48	6.40	6.24	7.15	13.79
Sulfur	4.07	4.18	4.18	2.98	3.08	4.36	2.86
Ash	41.48	42.74	45.00	36.33	37.79	44.71	
Total	100.92	102.32	104.57	100.31	102.25	103.06	88.35
Preasphaltenes							
Carbon	78.01	77.33	78.95	78.78	80.28	80.35	77.40
Hydrogen	5.51	5.41	5.69	6.29	6.75	6.62	6.11
Nitrogen	1.66	1.60	1.78	1.92	1.93	1.83	0.96
Oxygen	11.59	11.92	11.68	11.34	9.29	7.76	11.51
Sulfur	0.98	1.01	0.85	0.94	0.90	0.90	2.51
Total	97.75	97.27	98.95	99.27	99.15	97.46	98.49
Asphaltenes							
Carbon	83.50	83.30	84.47	84.90	83.91	83.97	81.90
Hydrogen	5.57	5.71	5.71	6.28	6.21	6.31	6.10
Nitrogen	1.75	1.67	1.73	1.92	1.88	1.94	2.34
Oxygen	6.75	6.61	5.99	5.57	6.20	5.74	7.45
Sulfur	2.10	1.17	1.05	0.98	1.08	1.11	1.68
Total	99.67	98.46	98.95	99.65	99.28	99.07	99.47
Oils (Hexane Sol)							
Carbon	88.29	87.87	88.49	89.83	86.94	88.34	88.80
Hydrogen	9.47	9.67	9.70	8.92	9.91	9.96	10.10
Nitrogen	0.30	0.33	0.34	<0.5	<0.5	<0.5	0.28
Oxygen	0.98	1.30	0.95	0.74	0.84	0.98	0.69
Sulfur	0.07	0.08	0.08	0.10	0.12	0.12	0.05
Total	99.11	99.25	99.56	99.59	97.81	99.40	99.92

Table 26

Inspection of Products from Solvent Deasphalting (Asphalt Considered a Candidate Feed to Delayed Coking)

Run No.	3039/3040 Blend	3034	3035
Deasphalted Oil (DAO)			
DAO yield, wt%	40.2	54.1	59.8
API Gravity @ 60°F	9.8	7.3	6.3
Conradson Carbon Residue, wt%	4.10	7.62	10.23
Heptane Insolubles, wt%	0.89	1.94	6.36
Ash, wt%	0.010	0.008	0.003
Carbon, wt%	87.40	87.56	87.77
Hydrogen, wt%	9.99	9.68	9.50
Sulfur, wt%	0.02	0.03	0.04
Nitrogen, wt%	0.63	0.70	0.90
Metals, ppm			
Fe	5	11	2.6
Ni	0.4	0.2	0.2
V	0.2	0.1	0.1
Cu	0.1	0.1	0.1
Na	0.3	1.6	0.5
ASTM Distillation (D-1160), °F			
IBP	537	526	532
2 vol%	603	602	618
5	665	668	687
10	717	716	728
20	753	754	765
30	779	778	791
40	801	800	820
50	827	828	853
60	859	858	894
70	912	907	963
80	996	983	1107(75%)
90	1045(83%)	1023(84%)	

(Cont) Inspection of Products from Solvent Deasphalting (Asphalt considered a Candidate Feed to Delayed Coking)

Run No.	3039/3040[1] Blend	3034	3035
Asphalt			
Yield, wt%	59.8	48.9	41.3
Specific Gravity @ 60/60°F	1.357	1.449	1.532
Conradson Carbon Residue, wt%	58.31	68.58	69.28
Asphaltenes, wt%	19.12	14.45	14.09
Toluene Insolubles, wt%	42.04	57.52	60.82
Quinoline Insolubles, wt%	33.93	49.40	51.23
Ash, wt%	19.09	24.14	27.23
Softening Point, °F	242	348	NA
Carbon, wt%	78.82	73.73	69.62
Hydrogen, wt%	6.36	5.13	4.40
Sulfur, wt%	2.01	2.50	2.73
Nitrogen, wt%	1.18	1.24	1.20
Metals, ppm			
Fe	38900	45300	43600
Ni	21	21	21
V	95	116	137
Cu	29	95	158
Na	480	637	764

NA - Not applicable. Sample decomposed before melting.

(1) - Selected Feedstock for Delayed Coking.

Table 27

Revision 1
July 1993

Delayed Coking Asphalt(1) Product From Solvent Deasphalting Wilsonville Atmospheric Residue

FWDC Run No.	3047

Operating Conditions

Coke Drum Pressure, psig	5
Throughput Ratio (Total feed/Fresh feed)	1.0
Steam/Feed Ratio, w/w	0.134
Maximum Drum Temperature, °F (Avg.)	901

Material Balance

Product Distribution-Wt%

	As Measured	Normalized
Gas	2.2	2.3
Distillate Oil	36.2	38.6
Coke	55.3	59.1
Recovery	93.7	100.0

Overall ASCOT Liquid Yield

	wt%
Solvent Deasphalting Yield	40.2
Asphalt Coking Yield (59.8 x 0.386)	23.1
Total Liquid Yield (on atm residue)	63.3
Total Liquid Yield (Solids-Free Basis)(2) (63.3/0.778)	81.4

---

(1) Asphalt produced by blending solvent deasphalting product from SDA Run Nos. 3039/3040

(2) Feedstock solids defined as Quinoline Insolubles (22.2 wt%).

Table 28

Delayed Coking Asphalt[1] Product From Solvent Deasphalting Wilsonville Atmospheric Residue

FWDC Run No.	3048

Operating Conditions

Coke Drum Pressure, psig	5
Throughput Ratio (Total feed/Fresh feed)	1.0
Steam/Feed Ratio, w/w	0.163
Maximum Drum Temperature, °F (Avg.)	962

Material Balance

Product Distribution-Wt%

	As Measured	Normalized
Gas	3.9	4.0
Distillate Oil	35.1	35.7
Coke	59.2	60.3
Recovery	98.2	100.0

Overall ASCOT Liquid Yield

	wt%
Solvent Deasphalting Yield	40.2
Asphalt Coking Yield (59.8 x 0.357)	21.3
Total Liquid Yield (on atm residue)	61.5
Total Liquid Yield (Solids-Free Basis)[2] (61.5/0.778)	79.0

---

[1] Asphalt produced by blending solvent deasphalting product from SDA Run Nos. 3039/3040

[2] Feedstock solids defined as Quinoline Insolubles (22.2 wt%).

Table 29

Inspection of Run 3042 Delayed Coking Products
Direct Delayed Coking

Distillate Oil

API Gravity @ 60°F	10.4	
Carbon, wt%	87.95	
Hydrogen, wt%	9.99	
Sulfur, wt%	0.05	
Nitrogen, wt%	0.79	
Ash, wt%	0.007	
Metals, ppm (w)		
Iron	22	
Nickel	< 1	
Vanadium	< 1	
Copper	1	
Sodium	3	
ASTM Distillation, °F	D86	D1160
IBP	352	393
2 Vol%	--	468
5	504	540
10	710	635
20		726
30		762
40		787
50		809
60		831
70		856
80		885
90		937
95		1010
EP		1023

Coke

Proximate Analysis, wt%	
Fixed Carbon	59.14
Volatile Matter	15.21
Ash	25.24
Moisture	0.41
Total	100.0

Ultimate Analysis, wt%	
Carbon	67.26
Hydrogen	3.02
Oxygen	0.09
Nitrogen	1.20
Sulfur	2.78
Ash	25.24
Moisture	0.41
Total	100.0

Table 30

Inspection of Run 3047 Delayed Coking Products
ASCOT Process

Distillate Oil

API Gravity @ 60°F	5.7
Carbon, wt%	87.68
Hydrogen, wt%	9.02
Sulfur, wt%	0.05
Nitrogen, wt%	1.01
Ash, wt%	0.018
Metals, ppm (w)	
Iron	61
Nickel	0.2
Vanadium	0.1
Copper	4
Sodium	2.2
ASTM Distillation, °F	D1160
IBP	
2 Vol%	
5	
10	
20	
30	
40	
50	
60	
70	
80	
90	
95	
EP	

Coke

Proximate Analysis, wt%	
Fixed Carbon	54.73
Volatile Matter	14.81
Ash	29.98
Moisture	0.48
Total	100.0

Ultimate Analysis, wt%	
Carbon	62.06
Hydrogen	2.55
Oxygen	0.09
Nitrogen	1.37
Sulfur	3.47
Ash	29.98
Moisture	0.48
Total	100.0

Table 31

Inspection of Run 3048 Delayed Coking Products
ASCOT Process

Distillate Oil

API Gravity @ 60°F	4.9
Carbon, wt%	86.69
Hydrogen, wt%	8.69
Sulfur, wt%	0.07
Nitrogen, wt%	0.97
Ash, wt%	0.05
Metals, ppm (w)	
Iron	170
Nickel	0.5
Vanadium	0.2
Copper	5.8
Sodium	3.4
ASTM Distillation, °F	D1160
IBP	
2 Vol%	
5	
10	
20	
30	
40	
50	
60	
70	
80	
90	
95	
EP	

Coke

Proximate Analysis, wt%

Fixed Carbon	55.42
Volatile Matter	13.51
Ash	29.77
Moisture	1.30
Total	100.0

Ultimate Analysis, wt%

Carbon	61.96
Hydrogen	2.21
Oxygen	0.15
Nitrogen	1.23
Sulfur	3.38
Ash	29.77
Moisture	1.30
Total	100.0

Table 32

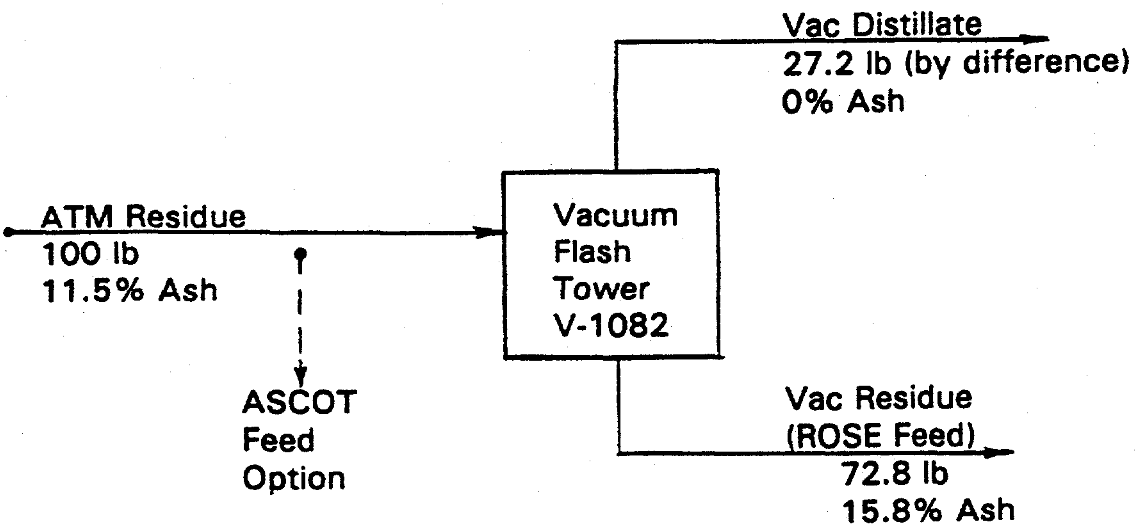
Calculation of Vacuum Tower/ROSE-SR™ Process Liquid Yield
At
Advanced Coal Liquefaction R&D Facility
Wilsonville, Alabama

Basis:

- Draft Technical Progress Report, Run 262, with Black Thunder Subbituminous Coal, by Southern Electric International Inc, 1991 (DOE Contract No. DE-AC22-90PC90033). Figures 25-30, Material Balance Flow Diagrams for Runs 262A Through 262F.
- Feed to ROSE-SR™ contains an average of 15.8% ash (Calculated from Figures 25-30)
- Solid product averages 56.9% of ROSE-SR™ feed (Calculated from Figures 25-30)
- Ash content of Atmospheric Residue 11.5 wt% (Analysis of Residue from Black Thunder subbituminous coal by FWDC)
- Ash in vacuum distillate product assumed at zero percent.

Material Balance Around Vacuum Flash Tower (V-1082)

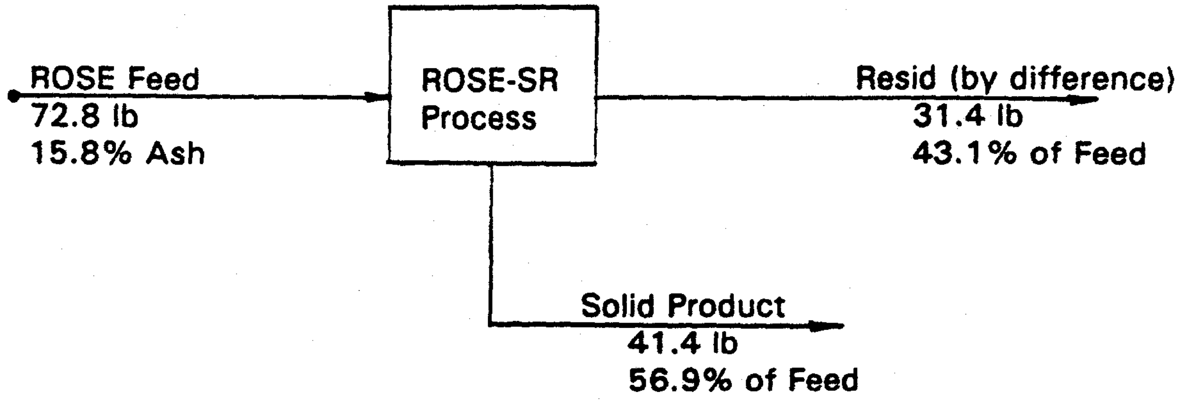
(For Portion of Vac Residue that Feeds ROSE Process)



Vacuum Residue Calculated via Ash Balance
0.115 x 100 lb/0.158 = 72.8 lb

Table 32 (cont)

Material Balance Around ROSE-SR^{TM} Process



Solid Product Calculation
72.8 lb x 0.569 = 41.4 lb

Vacuum Tower/ROSE Process Liquid Yield, % of ATM Residue

	wt %
Vacuum Distillate	27.2
Resid (ROSE Process)	31.4
Total Liquid Yield	58.6

NOTE: A technical objective of FW's ASCOT process is to meet or exceed the combined liquid yield from the vacuum tower and ROSE Process.

Table 33

Comparison of Yields from Atmospheric Residue

	Process			
	Wilsonville Run 262 Vac Tower + ROSE	Direct Delayed Coking	ASCOT 3047	ASCOT 3048
Products				
Gas	–	2.8	1.4	2.4
Oil				
Distillate	27.2	50.0	23.1	21.3
Deasphalted Oil	–	–	40.2	40.2
Resid	31.4	*	*	*
Solids/Coke	41.4	47.2	35.3	36.1
Total	100.0	100.0	100.0	100.0

---

* Included in oil product. The tail end of the ASCOT product oil would provide about two-thirds, and direct delayed coking about half the amount of Resid as the ROSE Process (Resid fraction defined as >850°F).

Figure 1a

DOE F 1332.3
(11-84)

U.S. DEPARTMENT OF ENERGY

MILESTONE SCHEDULE ☑ PLAN ☐ STATUS REPORT

FORM APPROVED
OMB NO. 1901-1400

1. TITLE Advanced Liquefaction Using Coal Swelling and Catalyst Dispersion Techniques

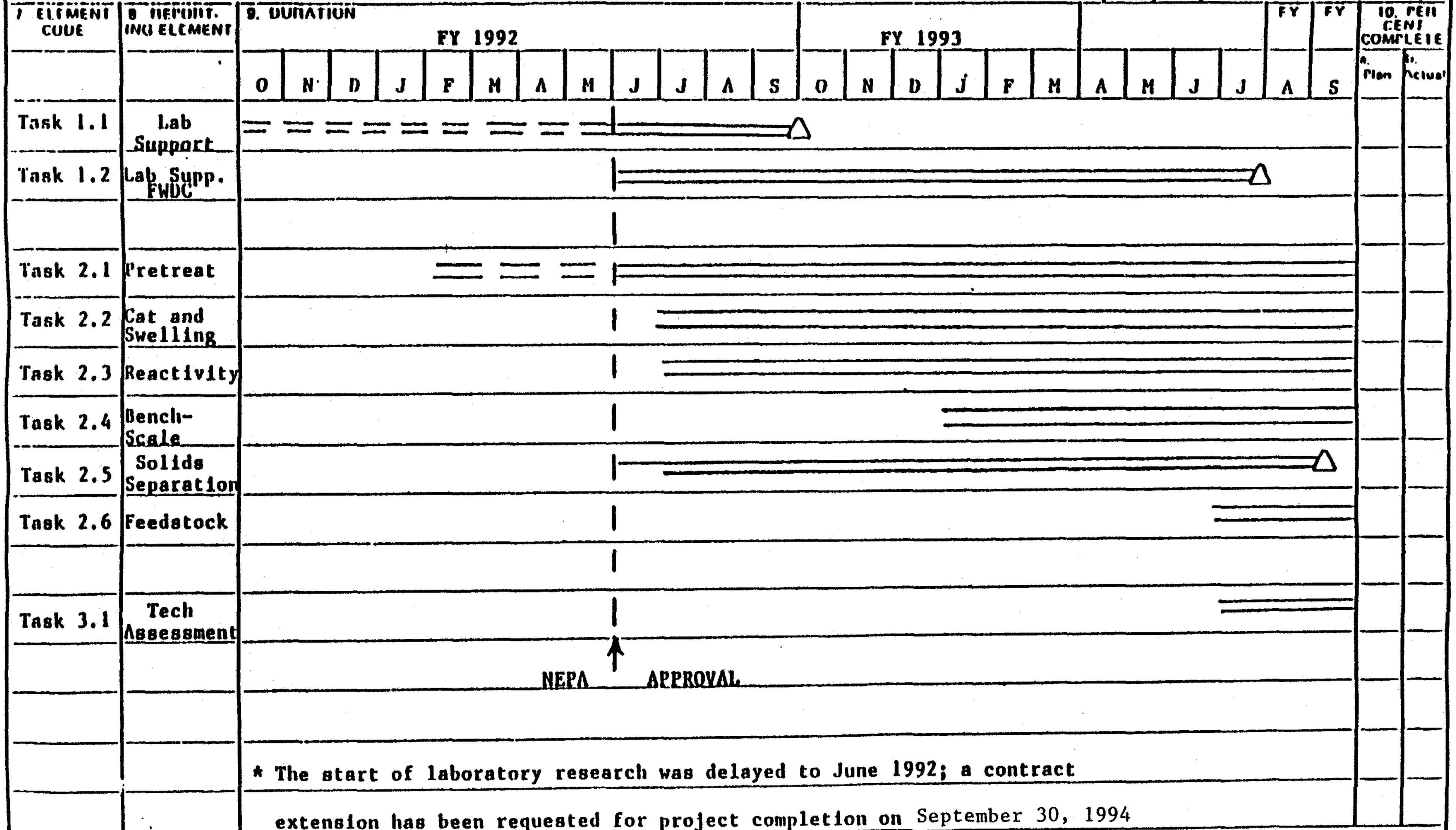
2. REPORTING PERIOD

3. IDENTIFICATION NUMBER DE-AC22-91PC91051

4. PARTICIPANT NAME AND ADDRESS
Amoco Oil Company
P. O. Box 3011
Naperville, IL 60566

5. START DATE October 1991 *

6. COMPLETION DATE May 31, 1994



* The start of laboratory research was delayed to June 1992; a contract extension has been requested for project completion on September 30, 1994

11. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE

Figure 1b

DOE F 1332.3
(11-84)

U.S. DEPARTMENT OF ENERGY

MILESTONE SCHEDULE ☑ PLAN ☐ STATUS REPORT

FORM APPROVED
OMB NO. 1901-1400

1. TITLE Advanced Liquefaction Using Coal Swelling and Catalyst Dispersion Techniques

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3. IDENTIFICATION NUMBER DE-AC22-91PC91051

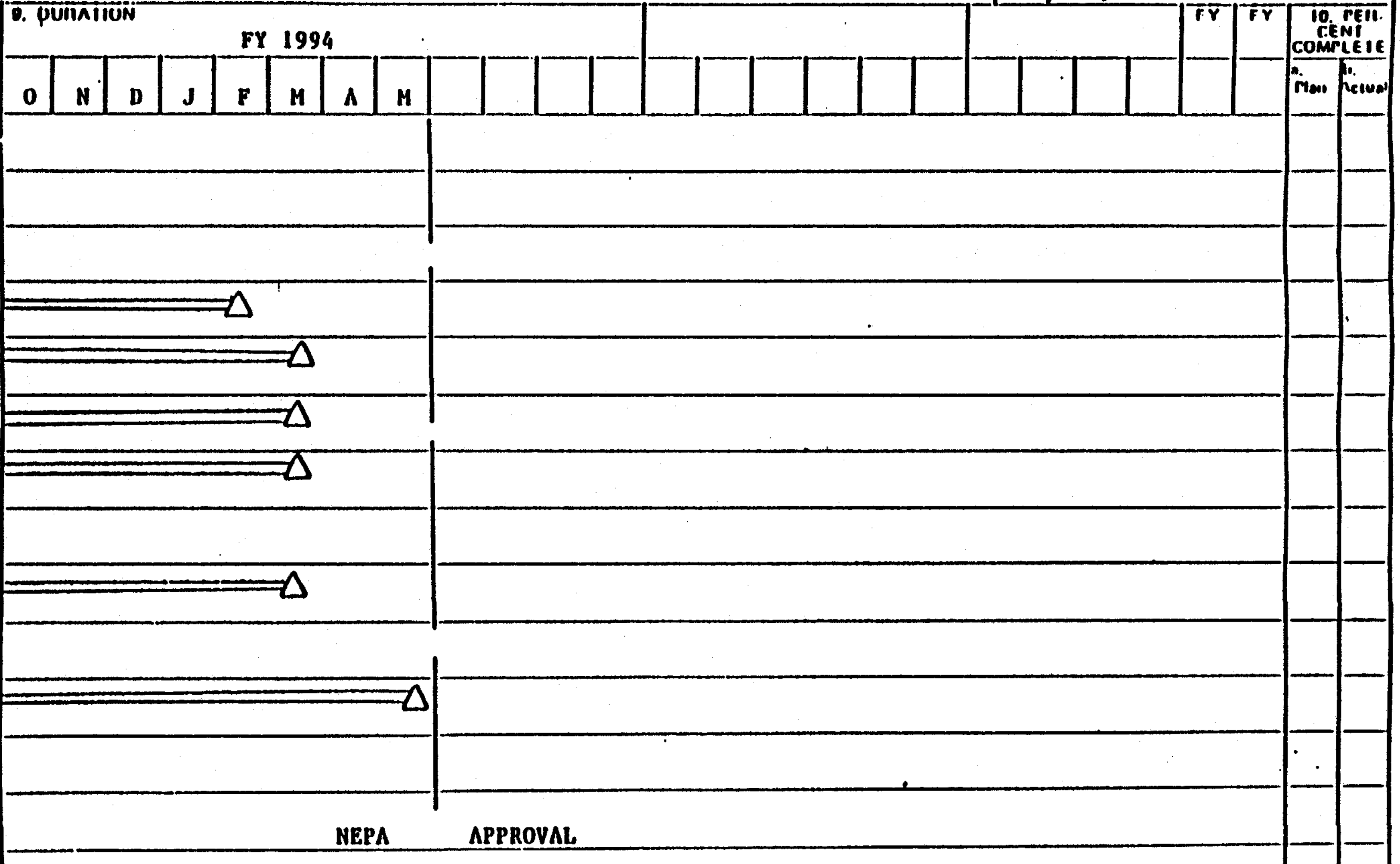
4. PARTICIPANT NAME AND ADDRESS
Amoco Oil Company
P. O. Box 3011
Naperville, IL 60566

Modified to reflect
anticipated extension
to May 31, 1994

5. START DATE October 1991 *

6. COMPLETION DATE May 31, 1994

7. ELEMENT CODE	8. REPORTING ELEMENT
Task 1.1	Lab Support
Task 1.2	Lab Supp. FWDC
Task 2.1	Pretreat
Task 2.2	Cat and Swelling
Task 2.3	Reactivity
Task 2.4	Bench-Scale
Task 2.5	Solids Separation
Task 2.6	Feedstock
Task 3.1	Tech Assessment



* The start of laboratory research was delayed to June 1992; a contract extension has been requested for project completion on May 31, 1994.

11. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE

Figure 2

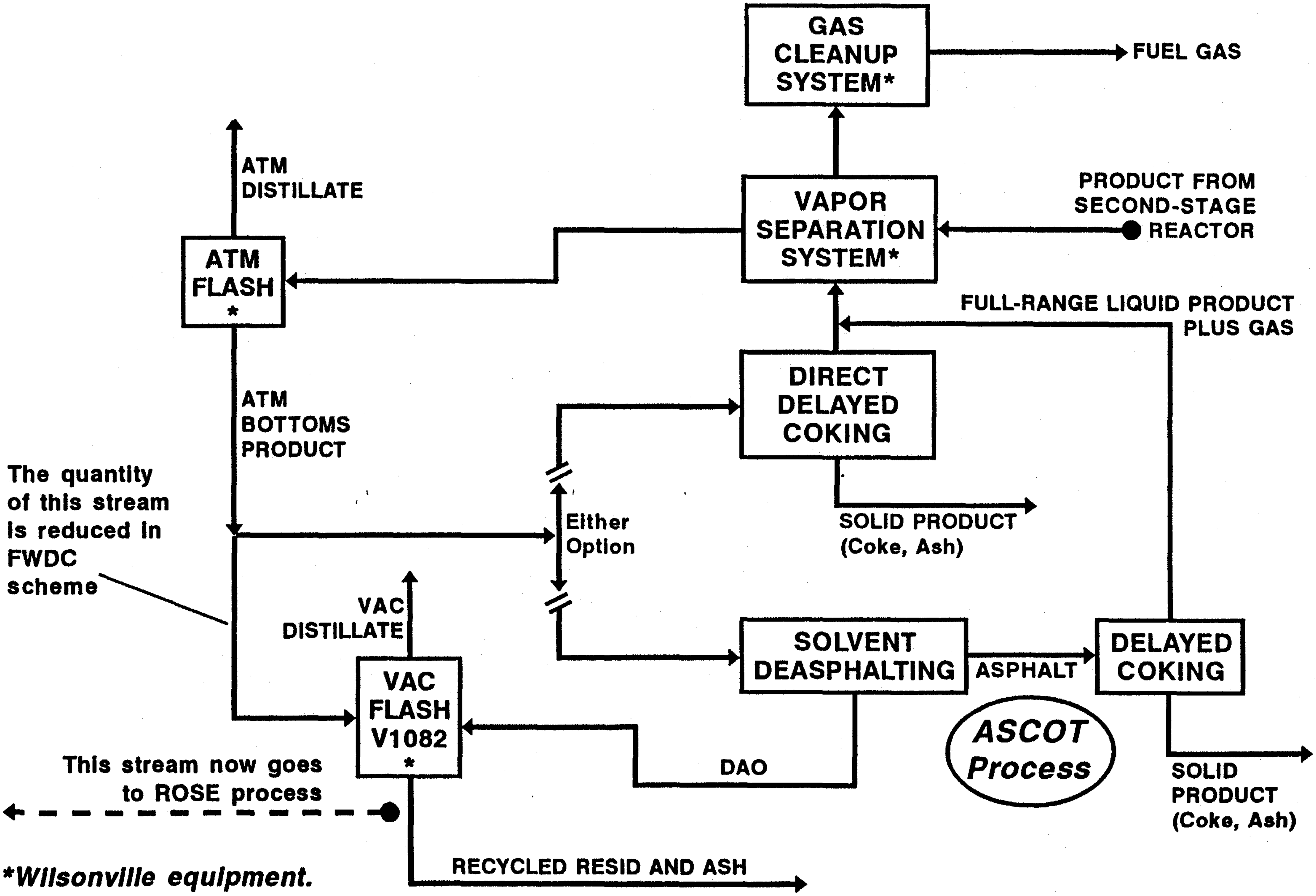


Figure 6 Scheme To Integrate ASCOT Process or Delayed Coking Into Wilsonville Close-Coupled ITSL Coal Liquefaction System Configuration

APPENDIX

```
REPORT: (SUMMARY OF EXTRACTIONS)
 BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
 CATALYST: None, or 100 ppm MV-L

REPORT:             Extraction   Normal'd
Description:        Run No.------01      04      05
----------------------------------- ------  ------  ------
CONDITIONS:                 PSIG    1981    1959    1954
 Temperature, F             (F)    826.0   824.0   825.0
 Space Time, min            Min     35.6    29.4    28.3
 Hydrogen (Brooksset)       SCFH    15.1    15.0    15.1
 Lineout Time, Hr           Hrs      0.0     0.0     0.0
 Run Time, after lineout    Hrs      3.0     3.0     3.0

MATERIAL BALANCE:
 Feed Rate (L/O+Run)      Gm/Hr  460.04  556.89  578.07
 Slurry Product           Gm/Hr  355.62  482.74  479.71
 Light Ends               Gm/Hr    0.00    0.00    0.00
 C1-C3 Gases              Gm/Hr   25.21   21.37   20.43
 C4+ Gases              . Gm/Hr   34.14   32.19   28.40
 Carbon Monoxide          Gm/Hr    0.90    0.77    0.82
 Carbon Dioxide           Gm/Hr    2.96    2.72    2.86
 Water                    Gm/Hr    0.00    0.00    0.00
 Recovery                  (%)     91.0%   96.9%   92.1%

YIELD: (Based on MAF Total Feed)
-------------------------------------------------------
 Hexane Solubles            Wt%    72.65   68.47   68.48
 Asphaltenes                Wt%    20.46   23.08   23.26

 Preasphaltenes             Wt%     3.07    3.87    3.52
 Hexane Insoluble           Wt%    27.35   31.53   31.52
 TOL Insoluble              Wt%     6.89    8.46    8.26
 THF Insoluble              Wt%     3.82    4.59    4.74

YIELD: (Based on MAF Coal Only)
------------------------------------------------------
 Asphaltenes                Wt%    68.92   77.73   78.35
 Preasphaltenes             Wt%    10.36   13.02   11.86
 THF Solubles               Wt%    87.15   84.53   84.04
 Hexane Insoluble           Wt%    92.13  106.21  106.18
 TOL Insolubles             Wt%    23.21   28.49   27.83
 THF Insolubles             Wt%    12.85   15.47   15.96
 Light Ends                 Wt%     0.00    0.00    0.00
 C1+C2+C3 Gases             Wt%    23.46   16.42   15.12
 C4+C5+C6 Gases             Wt%    31.76   24.74   21.03
 Carbon Monoxide            Wt%     0.83    0.59    0.61
 Carbon Dioxide             Wt%     2.75    2.09    2.12

EXTRACTION RESULTS:        Page: Normal'd
-------------------        -----  ------  ------  ------
 THF Insolubles             Gms    7.240   7.160   7.620
 Tol Insolubles             Gms   10.370  10.670  10.960
 Hex Ins in Tol Sol         Gms   20.830  20.950  22.060
 Hex & Tol Insol.           Gms   31.200  31.620  33.020
 HEX Solubles               Gms   79.170  79.050  77.940
 Extraction Feed            Gms  100.000 100.000 100.000
 Recovery                   (%)  110.37% 110.67% 110.96%
```

REPORT: (SUMMARY OF EXTRACTIONS)
BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

ELEMENTAL ANALYSIS: (Complete Extraction)

Unconverted Coal	(A)				
C		Frn	0.5048	0.5066	0.4311
H		Frn	0.0306	0.0308	0.0271
N		Frn	0.0106	0.0140	0.0102
O		Frn	0.0640	0.0624	0.0715
S		Frn	0.0298	0.0308	0.0438
Ash		Frn	0.3633	0.3779	0.4471
Total		Frn	1.0031	1.0225	1.0308
Preasphaltenes	(B)				
C		Frn	0.7878	0.8028	0.8035
H		Frn	0.0629	0.0675	0.6620
N		Frn	0.0193	0.0193	0.0183
O		Frn	0.1134	0.0929	0.0767
S		Frn	0.0094	0.0090	0.0251
Total		Frn	0.9928	0.9915	1.5856
Asphaltenes	(C)				
C		Frn	0.8490	0.8391	0.8397
H		Frn	0.0628	0.0621	0.0631
N		Frn	0.0192	0.0188	0.0194
O		Frn	0.0557	0.0620	0.0574
S		Frn	0.0098	0.0108	0.0111
Total		Frn	0.9965	0.9928	0.9907
Oils (Hex Sol)	(D)				
C		Frn	0.8983	0.8694	0.8834
H		Frn	0.0892	0.0991	0.0996
N		Frn	0.0030	0.0030	0.0030
O		Frn	0.0074	0.0084	0.0098
S		Frn	0.0010	0.0012	0.0012
Total		Frn	0.9989	0.9811	0.9970

MATERIAL BALANCE: DATE: 17-Aug 1992
BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L
RUN NO:01 826 F. Page .

CALCULATED ELEMENTAL CONTENT OF FEED SLURRY: (Gms/Hr)

ITEM:	H2O	Ash	MAF	Solvent	SwSolv	H2	Ex H2O H2 GAS	TOTAL
C	0.00	0.00	79.15	225.03	52.00	0.00	356.18	356.18
H	0.90	0.00	5.88	22.83	8.66	38.23	37.38	76.50
N	0.00	0.00	1.14	1.69	0.00	0.00	2.83	2.83
O	7.17	0.00	20.42	4.89	17.33	0.00	42.64	49.81
S	0.00	0.00	0.89	0.10	0.00	0.00	0.99	0.99
ASH	0.00	11.93	0.00	0.00	0.00	0.00	11.93	11.93
TOTAL	8.07	11.93	107.49	254.55	77.99	38.23	451.96	498.26
FEED:	8.07	11.93	107.49	254.56	77.99	38.23	451.97	498.27

CALCULATED ELEMENTAL CONTENT OF PRODUCT GASES AND LIQUID: (Gms/Hr)

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.38	0.81	19.70	28.25	0.00	0.00	49.1
H	31.78	0.00	0.00	5.51	5.89	0.00	0.00	43.2
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.51	2.15	0.00	0.00	0.00	0.00	2.7
S	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
ASH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
TOTAL	31.78	0.90	2.96	25.21	34.14	0.00	0.00	95.0
	31.78	0.90	2.96	25.21	34.14	0.00	0.00	95.0

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

	P/GAS	|....... .PRODUCT SLURRY.......|				SLURRY	SUB-TOT	SUB-TOT
ITEM	+L.E.	Unc.Coal	P/ASPH	ASPH.	OILS	TOTAL	-H2O	-H2&H2O
C	49.14	12.96	8.83	63.11	220.03	304.93	354.07	354.07
H	43.18	0.79	0.71	4.67	21.85	28.01	71.18	39.40
N	0.00	0.27	0.22	1.43	0.73	2.65	2.65	2.65
O	2.67	1.64	1.27	4.14	1.81	8.87	11.53	11.53
S	0.00	0.76	0.11	0.73	0.24	1.84	1.84	1.84
ASH	0.00	9.32	0.00	0.00	0.00	9.32	9.32	9.32
TOTAL	94.99	25.75	11.13	74.08	244.67	355.62	450.61	418.83
	94.99	25.75	11.13	74.08	244.67	355.62	450.6	

MATERIAL BALANCE: DATE: 17-Aug 1992
BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

RUN NO:04 824 F.
CALCULATED ELEMENTAL CONTENT OF FEED SLURRY: (Gms/Hr)

ITEM:	H2O	Ash	MAF	Solvent	MeOH	H2	Ex H2O H2 GAS	TOTAL
C	0.00	0.00	95.82	272.41	62.94	0.00	431.17	431.17
H	1.09	0.00	7.12	27.64	10.49	38.00	45.25	84.33
N	0.00	0.00	1.38	2.05	0.00	0.00	3.43	3.43
O	8.68	0.00	24.72	5.92	20.98	0.00	51.62	60.30
S	0.00	0.00	1.08	0.12	0.00	0.00	1.20	1.20
ASH	0.00	14.44	0.00	0.00	0.00	0.00	14.44	14.44
TOTAL	9.77	14.44	130.12	308.14	94.41	38.00	547.10	594.88
FEED:	9.77	14.44	130.12	308.15	94.41	38.00	547.12	594.89

CALCULATED ELEMENTAL CONTENT OF PRODUCT GASES AND LIQUID: (Gms/Hr)

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.33	0.74	16.70	26.64	0.00	0.00	44.4
H	31.41	0.00	0.00	4.67	5.55	0.00	0.00	41.6
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.44	1.98	0.00	0.00	0.00	0.00	2.4
S	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
ASH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
TOTAL	31.41	0.77	2.72	21.37	32.19	0.00	0.00	88.5
	31.41	0.77	2.72	21.37	32.19	0.00	0.00	88.5

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

	P/GAS	|.......PRODUCT SLURRY.......|				SLURRY	SUB-TOT	SUB-TOT
ITEM	+L.E.	Unc.Coal	P/ASPH	ASPH.	OILS	TOTAL	-H2O	-H2&H2O
C	44.41	17.12	13.72	85.48	292.51	408.83	453.24	453.24
H	41.63	1.04	1.15	6.33	33.34	41.86	83.50	52.08
N	0.00	0.47	0.33	1.92	1.01	3.73	3.73	3.73
O	2.42	2.11	1.59	6.32	2.83	12.84	15.26	15.26
S	0.00	1.04	0.15	1.10	0.40	2.70	2.70	2.70
ASH	0.00	12.77	0.00	0.00	0.00	12.77	12.77	12.77
TOTAL	88.46	34.56	16.94	101.13	330.10	482.74	571.20	539.79
	88.46	34.56	16.94	101.13	330.10	482.74	571.2	

MATERIAL BALANCE: DATE: 17-Aug 1992
BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

RUN NO:05 825 F.
CALCULATED ELEMENTAL CONTENT OF FEED SLURRY: (Gms/Hr)

ITEM:	H2O	Ash	MAF	Solvent	MeOH	H2	Ex H2O, H2 GAS	TOTAL
C	0.00	0.00	99.46	282.77	65.34	0.00	447.57	447.57
H	1.13	0.00	7.39	28.69	10.89	38.23	46.97	86.32
N	0.00	0.00	1.43	2.13	0.00	0.00	3.56	3.56
O	9.01	0.00	25.66	6.14	21.78	0.00	53.58	62.59
S	0.00	0.00	1.12	0.13	0.00	0.00	1.25	1.25
ASH	0.00	14.99	0.00	0.00	0.00	0.00	14.99	14.99
TOTAL	10.14	14.99	135.07	319.86	98.00	38.23	567.92	616.29
FEED:	10.14	14.99	135.07	319.88	98.00	38.23	567.93	616.30

CALCULATED ELEMENTAL CONTENT OF PRODUCT GASES AND LIQUID: (Gms/Hr)

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.35	0.78	15.96	23.50	0.00	0.00	40.6
H	31.69	0.00	0.00	4.47	4.90	0.00	0.00	41.1
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.47	2.08	0.00	0.00	0.00	0.00	2.6
S	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
ASH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
TOTAL	31.69	0.82	2.86	20.43	28.40	0.00	0.00	84.2
	31.69	0.82	2.86	20.43	28.40	0.00	0.00	84.2

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

	P/GAS	|.......	.PRODUCT SLURRY	......|		SLURRY	SUB-TOT	SUB-TOT
ITEM	+L.E.	Unc.Coal	P/ASPH	ASPH.	OILS	TOTAL	-H2O	-H2&H2O
C	40.60	15.29	8.12	89.69	284.70	397.80	438.40	438.40
H	41.06	0.96	6.69	6.74	32.10	46.49	87.55	55.85
N	0.00	0.36	0.18	2.07	0.97	3.59	3.59	3.59
O	2.55	2.54	0.78	6.13	3.16	12.60	15.15	15.15
S	0.00	1.55	0.25	1.19	0.39	3.38	3.38	3.38
ASH	0.00	15.85	0.00	0.00	0.00	15.85	15.85	15.85
TOTAL	84.21	36.55	16.02	105.82	321.31	479.71	563.92	532.22
	84.21	36.55	16.02	105.82	321.31	479.71	563.92	

FORCED ELEMENTAL BALANCE: DATE: 08/17/94

BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

RUN NO:01 826 F.
CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	|....FORCED....| H2O	H2S&NH3	SUBTOTAL EX.H-GAS	TOTAL
C		49.14	354.07	0.00	0.00	354.07	354.07
H	5.5%	11.40	39.40	4.78	-0.01	44.17	75.95
N	6.4%	0.00	2.65	0	0.18	2.83	2.83
O	79.2%	2.67	11.53	38.28	0.00	49.81	49.81
S	85.5%	0.00	1.84	0.00	-0.85	0.99	0.99
ASH		0.00	9.32	0.00	0.00	9.32	9.32

RUN NO:04 824 F.
CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	|....FORCED....| H2O	H2S&NH3	SUBTOTAL EX.H-GAS	TOTAL
C		44.41	453.24	0.00	0.00	453.24	453.24
H	8.7%	10.22	52.08	5.63	-0.09	57.62	89.03
N	0.0%	0.00	3.73	0	0.00	3.73	3.73
O	75.1%	2.42	15.26	45.04	0.00	60.30	60.30
S	124.3%	0.00	2.70	0.00	-1.50	1.20	1.20
ASH		0.00	12.77	0.00	0.00	12.77	12.77
TOTAL		57.05	539.79	50.67	-1.59	588.87	620.28

RUN NO:05 825 F.
CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	|....FORCED....| H2O	H2S&NH3	SUBTOTAL EX.H-GAS	TOTAL
C		40.60	438.40	0.00	0.00	438.40	438.40
H	10.0%	9.36	55.85	5.93	-0.13	61.65	93.34
N	0.0%	0.00	3.59	0	0.00	3.59	3.59
O	76.5%	2.55	15.15	47.44	0.00	62.59	62.59
S	170.6%	0.00	3.38	0.00	-2.13	1.25	1.25
ASH		0.00	15.85	0.00	0.00	15.85	15.85
TOTAL		52.51	532.22	53.37	-2.26	583.33	615.03

INDIVIDUAL RUN DATA BOOK#: 18119 July 93
COAL: Swelled IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

CONDITIONS:		Item: Unit:	Begin	End	Total	UOM	Yield Gm/Hr
RUN NO.01		Coal Feed	17.4	14.4	3.0	LBS	460.0
		Product Slurry	0.0	2.4	2.4	LBS	355.6
Temp.	826.0	Water (Discarded)	0.0	0.0	0.0	GMS	0.0
Res/min	35.6	Light Ends	0.0	0.0	0.0	GMS	0.0
		Total Liquid Yield			1066.9	GMS	355.6
REF:	Page 1	Lineout + Run Time			3.00	HRS	
		Offgases (wtm)			45.0	CFt	15.0
RUN NO.04		Coal Feed	42.6	39.0	3.7	LBS	556.9
		Product Slurry	0.0	3.2	3.2	LBS	482.7
Temp.	824.0	Water (Discarded)	0.0	0.0	0.0	GMS	0.0
Res/min	29.4	Light Ends	0.0	0.0	0.0	GMS	0.0
		Total Liquid Yield			1448.3	GMS	482.7
REF:	Page 2	Lineout + Run Time			3.00	HRS	
		Offgases			44.8	CFt	14.9
RUN NO.05		Coal Feed	36.4	32.6	3.8	LBS	578.1
		Product Slurry	0.0	3.2	3.2	LBS	479.7
Temp.	825.0	Water (Discarded)	0.0	0.0	0.0	GMS	0.0
Res/min	28.3	Light Ends	0.0	0.0	0.0	GMS	0.0
		Total Liquid Yield			1439.2	GMS	479.7
REF:	Page 3	Lineout + Run Time			3.00	HRS	
		Offgases			45.0	CFt	15.0
RUN NO.	0.00	Coal Feed	0.0	0.0	0.0	LBS	0.0
		Product Slurry	0.0	0.0	0.0	LBS	0.0
Temp.	0.0	Water (Discarded)	0.0	0.0	0.0	GMS	0.0
Res/min	0.0	Light Ends	0.0	0.0	0.0	GMS	0.0
		Total Liquid Yield			0.0	GMS	0.0
REF:	Page 4	Lineout + Run Time			0.00	HRS	
		Offgases			0.0	CFt	0.0

REPORT: (SUMMARY OF EXTRACTIONS)
BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

REPORT: Description:	Extraction Run No.------	Normal'd 197	1981	19811
CONDITIONS:	PSIG	1950	1960	1972
Temperature, F	(F)	824.0	823.0	825.0
Space Time, min	Min	22.2	22.7	35.9
Hydrogen (Brooksset)	SCFH	14.6	14.9	15.0
Lineout Time, Hr	Hrs	0.0	0.0	0.0
Run Time, after lineout	Hrs	3.0	8.3	3.9
MATERIAL BALANCE:				
Feed Rate (L/O+Run)	Gm/Hr	737.16	723.09	456.30
Slurry Product	Gm/Hr	722.18	698.33	430.83
Light Ends	Gm/Hr	0.92	0.87	1.96
C1-C3 Gases	Gm/Hr	10.63	10.11	14.78
C4+ Gases	Gm/Hr	9.38	11.59	13.82
Carbon Monoxide	Gm/Hr	0.75	0.66	0.93
Carbon Dioxide	Gm/Hr	2.76	2.54	3.09
Water	Gm/Hr	3.20	2.56	4.82
Recovery	(%)	101.7%	100.5%	103.1%

YIELD: (Based on MAF Total Feed)

Hexane Solubles	Wt%	66.19	68.42	75.44
Asphaltenes	Wt%	24.41	22.84	19.03
Preasphaltenes	Wt%	4.49	4.60	2.93
Hexane Insoluble	Wt%	33.81	31.58	24.56
TOL Insoluble	Wt%	9.40	8.73	5.54
THF Insoluble	Wt%	4.91	4.13	2.61

YIELD: (Based on MAF Coal Only)

Asphaltenes	Wt%	83.20	77.86	64.85
Preasphaltenes	Wt%	15.32	15.69	9.98
THF Solubles	Wt%	83.28	85.93	91.11
Hexane Insoluble	Wt%	115.24	107.61	83.71
TOL Insolubles	Wt%	32.03	29.76	18.87
THF Insolubles	Wt%	16.72	14.07	8.89
Light Ends	Wt%	0.00	0.00	0.00
C1+C2+C3 Gases	Wt%	5.18	5.03	11.64
C4+C5+C6 Gases	Wt%	4.57	5.76	10.88
Carbon Monoxide	Wt%	0.37	0.33	0.73
Carbon Dioxide	Wt%	1.35	1.26	2.44

EXTRACTION RESULTS:	Page:	Normal'd		
THF Insolubles	Gms	7.900	7.250	5.890
Tol Insolubles	Gms	12.250	11.770	8.830
Hex Ins in Tol Sol	Gms	23.630	22.430	19.110
Hex & Tol Insol.	Gms	35.880	34.200	27.940
HEX Solubles	Gms	76.370	77.570	80.890
Extraction Feed	Gms	100.000	100.000	100.000
Recovery	(%)	112.25%	111.77%	108.83%

REPORT: (SUMMARY OF EXTRACTIONS)
BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

ELEMENTAL ANALYSIS: (Complete Extraction)

Unconverted Coal	(A)				
C		Frn	0.4322	0.4272	0.4435
H		Frn	0.0219	0.0236	0.0244
N		Frn	0.0077	0.0101	0.0112
O		Frn	0.0919	0.0931	0.0748
S		Frn	0.0407	0.0418	0.0418
Ash		Frn	0.4148	0.4274	0.4500
Total		Frn	1.0092	1.0232	1.0457
Preasphaltenes	(B)				
C		Frn	0.7809	0.7733	0.7895
H		Frn	0.0551	0.0541	0.0569
N		Frn	0.0166	0.0160	0.0178
O		Frn	0.1159	0.1192	0.1168
S		Frn	0.0098	0.0102	0.0085
Total		Frn	0.9783	0.9728	0.9895
Asphaltenes	(C)				
C		Frn	0.8350	0.8330	0.8447
H		Frn	0.0557	0.0571	0.0571
N		Frn	0.0175	0.0167	0.0173
O		Frn	0.0675	0.0661	0.0599
S		Frn	0.0210	0.0117	0.0105
Total		Frn	0.9967	0.9846	0.9895
Oils (Hex Sol)	(D)				
C		Frn	0.8829	0.8787	0.8849
H		Frn	0.0947	0.0967	0.0970
N		Frn	0.0030	0.0033	0.0034
O		Frn	0.0098	0.0130	0.0095
S		Frn	0.0007	0.0008	0.0008
Total		Frn	0.9911	0.9925	0.9956

MATERIAL BALANCE: DATE: 17-Aug 1992 Total 12 Pages
BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L
RUN NO: 197 824 F.
CALCULATED ELEMENTAL CONTENT OF FEED SLURRY: (Gms/Hr)

ITEM:	H2O	Ash	MAF	Solvent	SwSolv	H2	Ex H2O, H2 GAS	TOTAL
C	0.00	0.00	151.04	436.61	0.00	0.00	587.64	587.64
H	1.71	0.00	11.22	44.30	0.00	37.09	55.52	94.32
N	0.00	0.00	2.17	3.29	0.00	0.00	5.46	5.46
O	13.69	0.00	38.97	9.48	0.00	0.00	48.45	62.14
S	0.00	0.00	1.70	0.20	0.00	0.00	1.90	1.90
ASH	0.00	22.76	0.00	0.00	0.00	0.00	22.76	22.77
TOTAL	15.40	22.76	205.10	493.88	0.00	37.09	721.74	774.23
FEED:	15.40	22.76	205.10	493.90	0.00	37.09	721.77	774.25

RUN NO: 197 824 F.
CALCULATED ELEMENTAL CONTENT OF PRODUCT GASES AND LIQUID: (Gms/Hr)

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.32	0.75	8.30	7.76	0.79	0.00	17.9
H	30.75	0.00	0.00	2.32	1.62	0.12	0.36	35.2
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.43	2.01	0.00	0.00	0.02	2.85	5.3
S	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
ASH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
TOTAL	30.75	0.75	2.76	10.63	9.38	0.92	3.20	58.4
	30.75	0.75	2.76	10.63	9.38	0.92	3.20	58.4

RUN NO: 197 824 F.
CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

	P/GAS	|.......	.PRODUCT SLURRY	.......|		SLURRY	SUB-TOT	SUB-TOT
ITEM	+L.E.	Unc.Coal	P/ASPH	ASPH.	OILS	TOTAL	-H2O	-H2&H2O
C	17.93	24.43	25.08	142.97	412.51	604.98	622.92	622.92
H	34.81	1.24	1.77	9.54	44.25	56.79	91.60	60.85
N	0.00	0.44	0.53	3.00	1.40	5.37	5.37	5.37
O	2.46	5.20	3.72	11.56	4.58	25.05	27.51	27.51
S	0.00	2.30	0.31	3.60	0.33	6.54	6.54	6.54
ASH	0.00	23.45	0.00	0.00	0.00	23.45	23.45	23.45
TOTAL	55.20	57.05	31.41	170.65	463.06	722.18	777.38	746.63
	55.20	57.05	31.41	170.65	463.06	722.18	777.4	

MATERIAL BALANCE: DATE: 17-Aug 1992
BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

RUN NO: 1981 823 F.
CALCULATED ELEMENTAL CONTENT OF FEED SLURRY: (Gms/Hr)

ITEM:	H2O	Ash	MAF	Solvent	MeOH	H2	Ex H2O, H2 GAS	TOTAL
C	0.00	0.00	148.15	428.27	0.00	0.00	576.42	576.42
H	1.68	0.00	11.00	43.46	0.00	37.85	54.46	93.99
N	0.00	0.00	2.13	3.22	0.00	0.00	5.36	5.36
O	13.43	0.00	38.23	9.30	0.00	0.00	47.53	60.95
S	0.00	0.00	1.67	0.19	0.00	0.00	1.86	1.86
ASH	0.00	22.33	0.00	0.00	0.00	0.00	22.33	22.33
TOTAL	15.10	22.33	201.19	484.45	0.00	37.85	707.96	760.92
FEED:	15.10	22.33	201.19	484.47	0.00	37.85	707.98	760.94

RUN NO: 1981 823 F.
CALCULATED ELEMENTAL CONTENT OF PRODUCT GASES AND LIQUID: (Gms/Hr)

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.28	0.69	7.90	9.59	0.74	0.00	19.2
H	31.76	0.00	0.00	2.21	2.00	0.11	0.28	36.4
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.38	1.84	0.00	0.00	0.02	2.27	4.5
S	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
ASH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
TOTAL	31.76	0.66	2.54	10.11	11.59	0.87	2.56	60.1
	31.76	0.66	2.54	10.11	11.59	0.87	2.56	60.1

RUN NO: 1981 823 F.
CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

	P/GAS	.......PRODUCT SLURRY.......				SLURRY	SUB-TOT	SUB-TOT
ITEM	+L.E.	Unc.Coal	P/ASPH	ASPH.	OILS	TOTAL	-H2O	-H2&H2O
C	19.21	21.14	25.09	132.52	406.81	585.56	604.77	604.77
H	36.08	1.17	1.76	9.08	44.77	56.78	92.85	61.09
N	0.00	0.50	0.52	2.66	1.53	5.20	5.20	5.20
O	2.24	4.61	3.87	10.52	6.02	25.01	27.25	27.25
S	0.00	2.07	0.33	1.86	0.37	4.63	4.63	4.63
ASH	0.00	21.15	0.00	0.00	0.00	21.15	21.15	21.15
TOTAL	57.53	50.63	31.56	156.63	459.50	698.33	755.86	724.10
	57.53	50.63	31.56	156.63	459.50	698.33	755.9	

MATERIAL BALANCE: DATE: 17-Aug 1992
BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

RUN NO: 19811 825 F.
CALCULATED ELEMENTAL CONTENT OF FEED SLURRY: (Gms/Hr)

ITEM:	H2O	Ash	MAF	Solvent	MeOH	H2	Ex H2O, H2 GAS	TOTAL
C	0.00	0.00	93.49	270.26	0.00	0.00	363.75	363.75
H	1.06	0.00	6.94	27.42	0.00	38.18	34.37	73.61
N	0.00	0.00	1.35	2.03	0.00	0.00	3.38	3.38
O	8.47	0.00	24.12	5.87	0.00	0.00	29.99	38.46
S	0.00	0.00	1.05	0.12	0.00	0.00	1.18	1.18
ASH	0.00	14.09	0.00	0.00	0.00	0.00	14.09	14.09
TOTAL	9.53	14.09	126.96	305.71	0.00	38.18	446.76	494.47
FEED:	9.53	14.09	126.96	305.72	0.00	38.18	446.77	494.48

RUN NO: 19811 825 F.
CALCULATED ELEMENTAL CONTENT OF PRODUCT GASES AND LIQUID: (Gms/Hr)

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.40	0.84	11.55	11.44	1.68	0.00	25.9
H	32.87	0.00	0.00	3.23	2.38	0.25	0.54	39.3
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.53	2.25	0.00	0.00	0.04	4.29	7.1
S	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
ASH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
TOTAL	32.87	0.93	3.09	14.78	13.82	1.96	4.82	72.3
	32.87	0.93	3.09	14.78	13.82	1.96	4.82	72.3

RUN NO: 19811 825 F.
CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

	P/GAS	|....... .PRODUCT SLURRY.......|				SLURRY	SUB-TOT	SUB-TOT
ITEM	+L.E.	Unc.Coal	P/ASPH	ASPH.	OILS	TOTAL	-H2O	-H2&H2O
C	25.91	10.76	10.11	70.28	275.93	367.09	392.99	392.99
H	38.73	0.59	0.73	4.75	30.25	36.32	75.04	42.18
N	0.00	0.27	0.23	1.44	1.06	3.00	3.00	3.00
O	2.82	1.82	1.50	4.98	2.96	11.26	14.07	14.07
S	0.00	1.01	0.11	0.87	0.25	2.25	2.25	2.25
ASH	0.00	10.92	0.00	0.00	0.00	10.92	10.92	10.92
TOTAL	67.45	25.38	12.67	82.33	310.45	430.83	498.27	465.41
	67.45	25.38	12.67	82.33	310.45	430.83	498.27	

FORCED ELEMENTAL BALANCE: DATE: 08/17/94

BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
CATALYST: None, or 100 ppm MV-L

RUN NO: 197 824 F.

CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

	PROCESS	COX+HC	SUBTOTAL	|....FORCED....	|	SUBTOTAL	
ITEM:	CONVRS'N	GASES	SL+LE+GS	H2O	H2S&NH3	EX.H-GAS	TOTAL
C		17.14	622.92	0.00	0.00	622.92	622.92
H	3.7%	3.94	60.85	4.33	-0.27	64.90	95.66
N	1.7%	0.00	5.37	0	0.09	5.46	5.46
O	48.3%	2.44	27.51	34.63	0.00	62.14	62.14
S		0.00	6.54	0.00	-4.64	1.90	1.90
ASH		0.00	23.45	0.00	0.00	23.45	23.45
TOTAL		23.52	746.63	38.96	-4.81	780.77	811.52

RUN NO: 1981 823 F.

CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

	PROCESS	COX+HC	SUBTOTAL	|....FORCED....	|	SUBTOTAL	
ITEM:	CONVRS'N	GASES	SL+LE+GS	H2O	H2S&NH3	EX.H-GAS	TOTAL
C		18.47	604.77	0.00	0.00	604.77	604.77
H	4.5%	4.21	61.09	4.21	-0.14	65.17	96.93
N	2.8%	0.00	5.20	0	0.15	5.36	5.36
O	47.4%	2.22	27.25	33.70	0.00	60.95	60.95
S		0.00	4.63	0.00	-2.77	1.86	1.86
ASH		0.00	21.15	0.00	0.00	21.15	21.15
TOTAL		24.90	724.10	37.92	-2.76	759.26	791.02

RUN NO: 19811 825 F.

CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

	PROCESS	COX+HC	SUBTOTAL	|....FORCED....	|	SUBTOTAL	
ITEM:	CONVRS'N	GASES	SL+LE+GS	H2O	H2S&NH3	EX.H-GAS	TOTAL
C		24.23	392.99	0.00	0.00	392.99	392.99
H	7.7%	5.61	42.18	3.05	0.01	45.24	78.11
N	11.2%	0.00	3.00	0	0.38	3.38	3.38
O	62.5%	2.78	14.07	24.39	0.00	38.46	38.46
S		0.00	2.25	0.00	-1.07	1.18	1.18
ASH		0.00	10.92	0.00	0.00	10.92	10.92
TOTAL		32.62	465.41	27.44	-0.68	492.17	525.04

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INDIVIDUAL RUN DATA                            BOOK#: 16103 June 93
COAL: Raw IL 6 + V-1074
 CATALYST: None, or 100 ppm MV-L                                     Yield
CONDITIONS:       Item:      Unit:     Begin    End     Total   UOM  Gm/Hr
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 RUN NO.  197.00  Coal Feed             35.4     30.5     4.9   LBS  737.2
                  Product Slurry         0.0      4.8     4.8   LBS  722.2
 Temp.     824.0  Water (Discarded)      0.0      9.7     9.7   GMS    3.2
Res/min     22.2  Light Ends             0.0      2.8     2.8   GMS    0.9
                  Total Liquid Yield                   2200.8   GMS  726.3
 REF:    Page 1   Lineout + Run Time                     3.03   HRS
                  Offgases (wtm)                         44.4   CFt   14.6
============================================================================
 RUN NO.1981.00   Coal Feed             43.0     29.9    13.1   LBS  723.1
                  Product Slurry         0.0     12.7    12.7   LBS  698.3
 Temp.     823.0  Water (Discarded)      0.0     21.1    21.1   GMS    2.6
Res/min     22.7  Light Ends             0.0      7.2     7.2   GMS    0.9
                  Total Liquid Yield                   5789.5   GMS  701.7
 REF:    Page 2   Lineout + Run Time                     8.25   HRS
                  Offgases                              123.1   CFt   14.9
============================================================================
 RUN NO.********  Coal Feed             25.5     21.6     3.9   LBS  456.3
                  Product Slurry         0.0      3.7     3.7   LBS  430.8
 Temp.     825.0  Water (Discarded)      0.0     18.9    18.9   GMS    4.8
Res/min     35.9  Light Ends             0.0      7.7     7.7   GMS    2.0
                  Total Liquid Yield                   1715.5   GMS  437.6
 REF:    Page 3   Lineout + Run Time                     3.92   HRS
                  Offgases                               60.0   CFt   15.3
============================================================================
 RUN NO.    0.00  Coal Feed              0.0      0.0     0.0   LBS    0.0
                  Product Slurry         0.0      0.0     0.0   LBS    0.0
 Temp.       0.0  Water (Discarded)      0.0      0.0     0.0   GMS    0.0
Res/min      0.0  Light Ends             0.0      0.0     0.0   GMS    0.0
                  Total Liquid Yield                      0.0   GMS    0.0
 REF:    Page 4   Lineout + Run Time                     0.00   HRS
                  Offgases                                0.0   CFt    0.0
============================================================================
DATE PRINTED          17-Aug ,1992
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