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ADVANCED LIQUEFACTION USING COAL SWELLING
AND CATALYST DISPERSION TECHNIQUES

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Pittsburgh, Pennsylvania

By

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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 4 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. 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.

Both laboratory- and bench-scale liquefaction experimentation is underway using swelled and catalyst impregnated coal samples. Higher coal conversions were observed for the SO₂-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. The initial laboratory and continuous flow unit (AU-51) experiments have indicated that the expected increase in coal conversion and product up-grading did not occur when the catalyst precursor, Molyvan-L, was impregnated into the swelled coal. There is likely an interaction between the Molyvan-L catalyst precursor and swelling solvent that effects coal liquefaction. This aspect and that of evaluating other precursors is underway.

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 batch deasphalting screening tests have been completed; pentane alone at 380°F provided an oil yield (63.6 wt%) that was desired for subsequent tests. An asphalt stream containing 21% ash has been handled successfully in the transport tests, and delayed coking tests are underway using this stream.

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 will integrate 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

A modification of the keystones has been approved to those shown in Figures 1a and 1b. (This modification was a result of obtaining a delayed approval of the authorization of the NEPA form to start experimentation.) The first key 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. The feedstocks have been obtained, and the beneficiation of a single sample of Martin Lake lignite and two samples of Black Thunder subbituminous coal have been done by Hazen Research Inc. A sample of froth floated, sulfurous acid-treated Black Thunder Coal has been prepared. The laboratory-scale coal swelling and catalyst screening program has started. The initial bench-scale liquefaction experiments with Black Thunder coal were carried out in January and February, 1993. The batch deasphalting experiments have been completed and the delayed coking experiments are underway. The coking experiments should be essentially complete by the end of the seventh quarter. The current program calls for program completion on May 31, 1994.

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.

Black Thunder subbituminous coal, can be effectively beneficiated to about 4 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 hetero-functionality. The rate of the impregnation of catalyst precursors into swollen coal increases greatly as the effectiveness of the solvent to swell the coal increases.

Laboratory- and bench-scale liquefaction experimentation is underway using swelled and catalyst impregnated coal samples. Higher coal conversions were observed for the SO₂-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. The initial laboratory and continuous flow unit (AU-51) experiments have indicated that the expected increase in coal conversion and product up-grading did not occur when the catalyst precursor, Molyvan-L, was impregnated into the swelled coal. There is likely an interaction between the Molyvan-L catalyst precursor and swelling solvent that effects coal liquefaction. This aspect and that of evaluating other precursors is underway.

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 batch deasphalting screening tests have been completed; pentane alone at 380°F provided an oil yield (63.6 wt%) that was desired for subsequent tests. An asphalt stream containing 21% ash has been handled successfully in the transport tests, and delayed coking tests are underway using this stream.

Activity is now centering upon (1) studying the liquefaction behavior of swollen coals that have been impregnated with catalyst precursors, and (2) undertaking coking experiments.

DISCUSSION OF RESULTS AND ACTIVITIES BY TASK

Project Documentation

A request for a modification of the Milestone Schedule and budget was submitted to DOE/PETC in the period following this quarter; the revised Milestone Schedule is attached as Figures 1a and 1b for reference. (The new schedule has been approved pending signing of formal documents.)

Task 1.0: Laboratory-Scale Experimentation

Subtask 1.1: Laboratory Support

The feedstocks are on hand, and analyses are complete as reported below and 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 planned for Hazen Research, Inc.

Task 2.0: Laboratory-Scale Operation

Subtask 2.1: Coal Pretreatment

Initial Coal Preparation:

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 returned to Amoco for characterization and use. These results and those of subsequent laboratory-scale experiments using heavy chlorinated solvents for gravity (density) separation were discussed in previous quarterly reports.

Flotation/Agglomeration Experiments:

Research underway at Pennsylvania State University is directed at beneficiating coal by a combination of chemical treatment and physical separation methods. A procedure has been developed for producing a low ash coal from SO_2 -treated Black Thunder subbituminous coal, that was prepared at Hazen Research, Inc. The procedure involves a combination of flotation and agglomeration methods. The method for producing a clean coal product of about 2% ash is described below:

Grinding: To obtain liberation of the ash-forming minerals, it was necessary to micronize coal. When grinding was done in a laboratory ball

mill with steel media, an increase in the ash content of the material was observed. Such contamination was coming from the grinding mill and media; the results were discussed in a previous report. In the laboratory tests contamination was decreased by grinding coal in an agate planetary mill and media. The agate mill was relatively inefficient, and it was necessary to use long grinding times. Alternate grinding procedures and mills might be considered for larger scale tests. It was observed that fine grinding is necessary for liberation of the ash-forming minerals as will be discussed in a subsequent section.

Spherical Agglomeration: In the previous quarterly report the benefits of agglomerating coal with oil prior to flotation were discussed. In the preliminary tests an ultrasonic bath was used to promote deaggregation and dispersion of the ash-forming minerals. However, the ultrasonic stage was found to be unnecessary. The desired amount of oil and the surfactant, when used, were mixed with the coal slurry in a blender. The surfactant used in this series of tests was a block co-polymer, designated as BCl, obtained from BASF Corporation (Pluronic 25R1). We are presently investigating an alternate method of mixing the oil in which the oil is pre-emulsified before mixing it with the coal slurry; the results will be reported later.

Flotation: The agglomerated slurry from the blender was transferred to a flotation cell, and it was diluted with an equal amount of water. The clean coal was floated for predetermined times. The froth products were filtered, dried, weighed and analyzed. The results are discussed below.

Effect of Grinding Time: Spherical agglomeration followed by flotation was carried out on three samples of different sizes produced by varying the grinding time. The amounts of material passing 400 mesh for these samples were about 58%, 83% and 96%, respectively. The results are given in Tables 1-3. The sample with 96% passing 400 mesh yielded a better product with an ash content of 2.10% and with a combustible matter recovery (CMR) of 93.7%. The lack of improvement in the product quality for other two samples suggests poor liberation and a necessity of further grinding.

Effect of Solids Concentration: Four different solids concentrations, namely 1.25%, 2.5%, 5.0% and 10%, were investigated using the sample with 95% passing 400 mesh. The results are given in Tables 4-6, respectively. Solid concentrations were based on the amount of solids in a slurry of 500 ml in the blender. Based on 4 minutes of flotation, selectivity decreased with increasing concentration of solids. A clean coal product with an ash content of 1.85% ash with a CMR of 84.47% was obtained for a solids concentration of 1.25%.

Effect of Oil and Surfactant Concentration: The amount of oil was varied while keeping the surfactant to oil ratio the same at 1 to 10. A solids concentration of 1.25% was used in these tests. The feed size was 96% passing 400 mesh. The amount of oil added was 1125 μ l, 2250 μ l, 4500 μ l and 9000 μ l for the four tests conducted. The results for these tests are given in Tables 7-10, respectively. Both the selectivity and the CMR were low, at 2.49% and 75%, respectively, for the lowest oil amount, 1125 μ l. Oil levels of 2250 μ l and higher produced better products with ash contents between 1.96 and 1.99% and with CMR's between about 87 and 91%.

Effect of Addition of Dispersant: A dispersant, sodium hexametaphosphate, was added in varying amounts during the grinding stage to increase selectivity. The amount of dispersant, calgon, was varied from 0, 20, 100, 500 and 2000 mg per 100 grams of coal for these tests. The coal feed size was 96% passing 400 mesh and the solids concentration was 2.5%. The results are given in Tables, 3, 11, 12, 13 and 14 for the respective amounts of calgon. The addition of calgon resulted in a small increase in the selectivity while the CMR decreased slightly.

Effect of Ultrasonic Treatment: All the previous tests summarized above were carried out following a pre-treatment of the slurry in an ultrasonic bath for 30 minutes prior to spherical agglomeration. The effect of ultrasonic treatment on the product quality was investigated, and the results for several times of ultrasonic treatment are given in Tables 15, 16, 17, 3, and 18. Corresponding time of ultrasonic treatment for these tests were 0, 4, 10, 30 and 60 minutes, respectively. A solids concentration of 2.5% was used. The coal feed size was 96% passing 400 mesh. The results show no significant benefits from the ultrasonic treatment. Both the ash content and the CMR remained essentially the same for all these tests.

Summary and Recommendations from the Flotation/Agglomeration Experiments:

The effect of operating variables such as grinding time, solids concentration, oil and surfactant concentration, chemical and mechanical dispersion was investigated. It was found that almost all of the sample had to be ground to pass 400 mesh for adequate mineral liberation. At such fine sizes flotation alone did not work satisfactorily, and it was necessary to use selective agglomeration prior to flotation (note the previous quarterly report). A solids concentration of 2.5% was found to be optimum from the current studies. It was observed that neither mechanical nor chemical dispersion helped significantly, most probably due to the fact that the strong agitation in the blender helped dispersion of the ash material while agglomerating the coal. A clean coal product with an ash content of about 2.0% with a CMR of about 90% should be capable of being produced in commercial equipment.

The following recommendations are made for planned pilot-scale experiments to be carried out at Hazen Research, Inc.:

Grinding:

Grind Size - 96% passing 400 mesh.

Spherical Agglomeration:

Solids concentration - 2.5% by weight.

Oil concentration - 300 kg/T of coal.

Surfactant concentration - 8 kg/T of coal.

Flotation:

Solids concentration - 1.25% by weight.

Frother - 3 kg/T of coal.

Subtask 2.2: Catalyst and Swelling Studies

Literature:

The literature concerning coal swelling, both alone and in combination with coal liquefaction, was reported in Quarterly Report Number Q-03 (April-June, 1992).

Coal Swelling Results:

The results of most of the coal swelling experiments were reported in Quarterly Report Number Q-04 (July-September, 1992). Additional runs are being made as part of the laboratory-scale liquefaction experiments; they will be reported in the appropriate section when generated.

The following is a summary of the previously generated conclusions:

1. Experimental reproducibility of the swelling experiments was good.
2. Solvent type strongly affected coal swelling with the greatest effect observed with solvents having hetero-functionality.
3. There was little effect of coal particle size upon the swelling ratio.
4. SO₂-treated coal swelled to a greater extent than untreated coal.
5. Solvent blends were more effective than individual solvents.

Catalyst Precursor Impregnation - Experimental:

The procedure used for the coal swelling/catalyst impregnation experiments consisted of mixing 100 g of SO₂-treated coal with 500 g of swelling solvent containing the catalyst precursor, normally 100 ppm metal as an organo-metallic. The resulting slurry was blended in a 1 L flask with baffles in a rotavapor unit (Brinkman R110) for the desired time and temperature. The treated coal was recovered by filtration, and in selected cases the level of metal in the coal was determined. Rather than analyze the coal samples, it was more cost effective to determine the concentration of metals remaining in the liquid using either inductively coupled plasma (ICP) or X-ray fluorescence (XRF).

Catalyst Precursor Impregnation - Results:

Effect of Swelling Solvent on Catalyst Penetration: If swelling alone determines diffusion rates of the catalyst precursor into the coal particle, the rate of metal catalyst uptake should follow the order dimethyl sulfoxide (DMSO) > tetrahydrofuran (THF) > isopropanol (IPA). This general trend is demonstrated in Figures 2a and 2b in which the ratio of Mo concentration in the remaining solvent to that of the initial solvent is shown for selected Molyvan-L impregnation experiments. Specifically, Molyvan-L loss into the toluene swelled coal is small, while that penetrating into the polar solvent swelled coals is much greater.

That of DMSO-swelled coal is also greater than that achieved with either the IPA- or THF-swelled coal. At short times (< 20 hr), penetration was better with the IPA-swollen coal than that with the THF-swollen coal, but they were about equal after two days.

Effect of Impregnation Temperature on Penetration: As shown in Figure 2b, 3a and 3b, catalyst impregnation is promoted by increased temperature. Moreover, the combination of a good swelling solvent (like DMSO) and a 150°F impregnation temperature results in 90% penetration after 30 minutes. At room temperature, this same penetration with DMSO was achieved after about 2 hours.

Effect of Particle Size on Penetration: Figures 4a, 4b, and 4c demonstrate that penetration is better with finer coal particles, especially at short impregnation times. For THF-swollen coal, the results of the -70 mesh and -200 mesh coals are the same within experimental error.

Effect of Catalyst Precursor on Penetration: Figure 5 indicates that Molyvan-L penetrates more effectively than Mo octoate for IPA-swollen coal. Additional experiments with Mo octoate are planned, and, if warranted, other precursors (like water-soluble and other Molyvan additives) will be tested.

Effect of Initial Precursor Concentration: Figure 6 indicates that penetration at higher initial concentration and long times is not as extensive as that obtained with a lower initial concentration. This difference could be due a partial saturation of deposition sites.

Stripping of Catalyst Precursor: A sample of SO₂-treated coal that was contacted with Molyvan-L was subsequently contacted with fresh THF for a two week period. As confirmed by both liquid and coal analyses, no precursor was removed from the coal.

Modelling of the Precursor Penetration Results: Simple diffusion/immobilization models which utilize Langmuir adsorption isotherms have been examined. It appears that the initial rates can be described by the limiting case of strong sorption. However, the adsorption appears to be irreversible in that Molyvan-L deposited on the swollen coals cannot be stripped from the coal. The particle geometry does not seem to be important, either. The modelling aspect will be studied further as more data is generated.

Catalyst Precursor Impregnation - Summary:

The following conclusions are drawn from simultaneous swelling and catalyst impregnation experiments conducted with SO₂-treated Black Thunder coal:

1. The rate and extent of catalyst penetration into coal particle correlates with the ability of solvent to swell coal.

-DMSO >> THF > IPA

-95% (equilibrium) penetration was attained after 6 hours swelling with DMSO at ambient temperature.

2. Higher impregnation temperatures enhance catalyst penetration, and the improvement is larger with less effective swelling solvents.

-95% penetration with THF/toluene can be achieved with 48-hour swelling at 120°F.

-the combination of a good swelling solvent (like DMSO) and 150°F impregnation temperature results in 90% penetration after 30 minutes.

3. The effect of lower concentration of the swelling solvent can be offset by increasing the impregnation temperature.
4. The rate of penetration is higher with smaller coal particles, especially at short impregnation times. There was no difference in results for -70 mesh and -200 mesh particles.
5. Molyvan-L penetrates more effectively than Mo octoate into IPA-swollen coal.

Subtask 2.3: Reactivity of Swelled Coals with Dispersed Catalysts

The objective of the research performed this quarter was to determine the effect of preswelling coal on the liquefaction behavior of SO₂-treated and untreated Black Thunder coal. The following four swelling solvents were used: methanol, isopropanol, tetrahydrofuran (THF) and dimethylsulfoxide (DMSO). After swelling, liquefaction reactions were performed using 1-methylnaphthalene as the reaction solvent. Pyrene was added to the liquefaction reaction as a probe hydrogenation model. Both thermal and catalytic reactions were performed. The catalyst precursors were Molyvan-L and nickel octoate. The effect of the preswelling was determined by comparing the amount of coal conversion to THF solubles and the amount pyrene conversion to hydrogenated products with both swelled and unswelled coals.

Experimental: Liquefaction reactions were performed in a sand bath micro-reactor using untreated and SO₂-treated Black Thunder coal, the analysis of which are presented in Table 19. The swelling solvents were methanol, isopropanol, tetrahydrofuran and dimethylsulfoxide, all of which were obtained from Aldrich and were used as received. The reaction solvent was 1-methylnaphthalene (98% purity) also from Aldrich.

Swelling: Both treated and untreated coals were swelled prior to reaction by introducing 2 g of coal to a swelling tube and then adding 10 ml of swelling solvent. After 2 hr in the swelling solvent, the coal swelling was determined by measuring the height of the coal and comparing that to the height of the coal prior to swelling. The change in volume was

calculated by

$$\% \Delta V = \frac{\pi r^2 h_1}{\pi r^2 h_0} \times 100\% = \frac{h_1}{h_0} \times 100\%$$

where, ΔV = the volume change of the coal
 r = the inside radius of the tube
 h_0 = the initial height of the coal in the tube in an unswelled state
 h_1 = the height of the coal in the tube in a swelled state

The coal was left in the swelling solvent for a total of 24 hr at the conditions given in Table 20. This solvent was then filtered from the coal, which was then weighed and placed in a liquefaction reactor. The amount of coal introduced was calculated using the following equation

$$W_{TB} = (T_C - T_0) - \frac{(T_F - T_0)(T_C - T_0)}{(T_{CS} - T_0)}$$

where, W_{TB} = mass of coal transferred to the tubular reactor in terms of its mass in the unswelled state
 T_0 = mass of the swelling tube without coal
 T_C = mass of the swelling tube with coal
 T_{CS} = mass of the swelling tube plus swelled coal after solvent removal
 T_F = mass of the swelling tube after removal of the swelled coal

Liquefaction Reactions: The liquefaction reactions using SO_2 -treated and untreated Black Thunder coal were performed in a sand bath micro-reactor at $410^\circ C$ for 30 min. as given in Table 20. Each reaction contained 1.33 g of maf coal, 2 g of 1-methylnaphthalene as solvent, 0.67 g of pyrene. The coal contained any residual solvent that remained after swelling. Hydrogen gas was introduced at 1250 psig at ambient temperature. The catalyst precursors were Molyvan-L and nickel octoate, each introduced at a level of 600 to 800 ppm of active metal. The reactor was well-agitated at 500 cpm.

The liquefaction products were removed from the reactor with THF. The conversion of the coal to THF solubles was determined. The amount of hydrogenation of pyrene to hydrogenated products was determined by gas chromatographic analyses using a Varian Model 3400, a SGE HT-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 those required to form the most hydrogenated product, perhydropyrene.

Results and Discussion - Swelling of Coal Samples: The results of the recent swelling runs, that are being carried out as part of the liquefaction experiments, are consistent with those generated in the initial swelling runs reported in the previous quarterlies. The averages

of the swelling ratios of both sets of runs are given in the following table:

<u>Swelling Ratio</u>		
<u>Solvent\Coal</u>	<u>Untreated</u>	<u>SO₂-Treated</u>
Methanol	1.23	1.31
Isopropanol	1.22	1.40
Tetrahydrofuran	1.46	1.97
Dimethylsulfoxide	2.10	2.58

The amount of swelling solvent remaining in the swollen coal samples after the surface solvent was drawn off is given in Table 21. Methanol and isopropanol levels remaining in the coal ranged from 0.9 to 1.3 g for 1.6 to 1.7 g of coal for both the raw and SO₂-treated coals. The level of THF was in the range of 1.45 to 1.7 g in 1.6 to 1.7 g of untreated coal, and about 2 g in the case of SO₂-treated coal. DMSO, which had the highest swelling ratio, ranged between 3.1 and 4.3 g in 1.62 g of untreated coal and between 4.0 and 5.7 in the case of SO₂-treated coal.

Results and Discussion - Liquefaction Results Using Swelled Coals: A detailed description of the thermal and catalytic reactions of untreated and SO₂-treated Black Thunder with and without swelling solvent is presented in Tables 22 and 24. All of the listed experiments were performed using 1-methylnaphthalene as the reaction solvent and pyrene as the probe hydrogenation agent. Note that the liquefaction runs were made with the catalyst precursor being charged just before the reaction, so that there wasn't time for precursor penetration into the swollen coal prior to liquefaction.

Summary tables for the liquefaction behavior of untreated and SO₂ treated coals using 1-methylnaphthalene as the reaction solvent and THF, methanol, isopropanol, and DMSO as the swelling solvents are given in Tables 26 and 27. Based on these overall averages, there was a positive effect on subsequent thermal coal conversion when untreated coal was swollen with THF, isopropanol, or methanol. There was only a nominal positive effect as a result of the similar swelling of SO₂-treated coal. The swelling solvents also showed a nominal positive effect upon the conversions of both untreated and SO₂-treated coal with the addition of Molyvan-L. The combination of methanol with nickel octoate stood out as being particularly effective for coal conversion, even though nickel octoate was less effective than Molyvan-L in all other cases.

Even though DMSO swelled the untreated and SO₂-treated Black Thunder coal greatly, it exhibited a major negative effect upon subsequent liquefaction for both coals. As a result of swelling with DMSO, thermal coal conversion decreased from about 50% to 30%. Even the activity of the Molyvan-L catalyst was affected adversely by DMSO. Coal conversion with nickel octoate was the most adversely affected by DMSO (~76% versus ~50%).

The presence of all of the swelling solvents in the coal samples was detrimental for pyrene conversion during the liquefaction runs with untreated coal except for the combination of nickel octoate with methanol. This adverse effect also occurred with SO₂-treated Black Thunder coal. Methanol, isopropanol, and THF were very detrimental to pyrene conversion except when Ni octoate was present. The primary reaction product from pyrene for reactions with both untreated Black Thunder coal (Table 23) and SO₂-treated Black Thunder coal (Table 25) was dihydropyrene (DHP). For all the reactions regardless of the presence or type of swelling solvent, the highest level of pyrene conversion occurred with Molyvan-L as the catalyst. For untreated Black Thunder coal, the yield distribution of DHP produced with Molyvan-L was the following: no swelling solvent, methanol > THF, isopropanol > DMSO. The ranking for DHP production with SO₂-treated Black Thunder was the following: no solvent, THF, methanol > isopropanol > DMSO. Reactions with Molyvan-L also produced small amounts of tetrahydropyrene (THP). For some reactions, a very small amount of hexahydropyrene (HHP) was also formed.

Subtask 2.4: Bench-Scale Studies

The objectives of this subtask are (1) to evaluate the effectiveness of the coal swelling 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 run periods were made 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. (As will be shown, these conditions were too mild.)

Five continuous flow unit liquefaction runs were completed using the following operating periods:

- 1) SO₂-treated coal and Wilsonville solvent (V1074); Run No. 93,
- 2) SO₂-treated coal with Molyvan-L catalyst (100 ppm Mo based on feed coal) and Wilsonville V1074 solvent; Runs 95 & 102,
- 3) SO₂-treated coal that was swollen with 30/70 THF/toluene and then liquefied with Wilsonville V1074 solvent containing 20% resid (the run was non-catalytic); Run 113,
- 4) SO₂-treated coal that was mixed with THF, Wilsonville V1074 solvent, resid, and Molyvan-L (100 ppm Mo based on feed coal), Run 115, Periods 1 & 2,
- 5) The same system as 4, but without sufficient time for catalyst penetration prior to being used; Run 115, Period 3.

Experimental:

The liquefaction experiments were carried out in the AU-51L two-stage pilot-scale unit that operates in a once-through continuous mode with regard to hydrogen and feed slurry. A schematic is shown in Figure 7.

The AU-51 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, Bran-Lubbe pump. The slurry is then combined with hydrogen and fed to the inlet of the first of two 1-liter stirred reactors. The liquid volume of the two reactors are 271 and 270 cc, respectively as a result of using dip-legs. The first stage product flows to the second through an overflow line. The second stage product flows to one of two vapor/liquid separators. Pressure is controlled by regulators in the vent gas stream. The product gases are metered and analyzed. The product slurry is periodically sampled by cycling the two slurry receivers.

Product analyses for these runs include GC gas analyses and Soxhlet extraction of slurry samples using the solvents: toluene, tetrahydrofuran (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 (including 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 either untreated or SO₂-treated Black Thunder coal; representative analyses are given in Table 19. The liquefaction solvents consisted of either Wilsonville V1074 distillate (650-1000°F), or a blend of V1074 with 20 wt% Wilsonville resid. The analyses are given in Table 28.

Two approaches were followed in calculating material balances and yields. As-recorded data and flows were used in the first, and in the second case, the product slurry rate was adjusted for an overall 100.0% material balance. It was assumed that the feed rates were accurate and the recovery of product slurry was then adjusted. This assumption appears valid, because there was some material remaining on the walls of the product vessels and there was some splattering of product out of the collection cans.

The swollen coal for Run 113 was prepared by blending a mixture of one part pulverized, SO₂-treated coal with two parts of 20 wt% THF with 80 wt% toluene. The slurry was mildly agitated for two days, and then filtered. The solids underwent limited drying. A liquefaction feed slurry was blended for a concentration of approximately 30 wt% (dry) coal with V-1074. The resulting feed slurry was too thin to establish a uniform feed through the Bran-Lubbe piston pump; therefore, resid was added to the feed. The resulting feed blend consisted of 27.4% coal (dry), 17.0% swelling solvent, 44.4% V-1074, and 11.2% resid.

The first two periods of Run 115 were carried out using a feed blend of THF, V-1074, resid, SO₂-treated coal, and Molyvan-L in the following percentages: 11.7%, 47.1%, 11.8%, 29.4%, and 100 ppm Mo (based on coal). This blend was mixed for two days at about 110°F prior to the run. The third period of Run 115 was carried out using the same slurry concentrations, but with only three hours of blending before the run period.

Results and Discussion:

Reference Run: The reference run (93) was carried out using a 2/1 slurry blend of V1074 solvent with SO₂-treated Black Thunder coal at reaction conditions of 800°F and 2000 psig pressure. The nominal space time of the feed slurry (calculated at ambient conditions) was 80 minutes. The normalized results are given in Table 29, and the results and operating of both calculation approaches are given in Appendix A. Product analyses are given in Tables 30 and 31.

The level of coal conversion to THF solubles was low at 79.2%. The yield of pre-asphaltenes was high at about 10.3% on MA feed coal. Of particular significance, the yield of asphaltenes was in excess of 70% indicating that little up-grading of coal-derived material was occurring. In fact, a portion of the V-1074 solvent, that contains only 1.3% hexane insolubles, reacted with the coal-derived material to form additional insolubles. The yield of C₁* gases was reasonable at 8.1% on MA feed coal, as was that of carbon oxides at 4.4%.

Run with Molyvan-L Catalyst: As shown in Table 29, the addition of 100 ppm Mo (as Molyvan-L) in Run 102 resulted in a nominal increase in conversion of coal to THF solubles (81 vs. 79%). However, there was a sizeable increase in the yields of pre-asphaltenes and asphaltenes. Considering past Wilsonville results and those of our previous DOE-sponsored study, these results are suspect. Perhaps the run should be repeated or rerun as part of a series at more severe conditions. It is also noted that the yield of hydrocarbon gases was high at 12.7%.

Run with Swollen Coal: Run 113 was carried out using SO₂-treated coal that had been swollen with a 20/80 mixture of THF in toluene. In addition, resid from Wilsonville was added to the feed to make the slurry more pumpable (thin coal slurries result in plugging of the pump checks).

The resulting level of coal conversion to THF solubles was again nominally greater than the reference run (82 vs. 79%). The yield of pre-asphaltenes was reasonable at about 10% after provision is made for the additional level in the feed resid. The yield of asphaltenes was again very high, even after subtracting the amount in the feed resid. The level of retrogressive reactions at these reaction conditions was obviously high.

Runs with Swollen and Impregnated Coal: Due to difficulties in preparing for the above run (113), the initial coal slurry (first two periods of Run 115) was prepared by charging in succession coal, THF, V-1074 and resid to the feed tank. The percentages were 11.7%, 29.4%, 47.1%, and 11.8%, respectively. The slurry was blended for about one hour and Molyvan-L was added at a level of 98 ppm Mo based on coal. This feed slurry was then

mixed for two days at about 80°F prior to the start of the run. The third period of Run 115 was carried out using a feed blend of material remaining in the feed tank with added slurry that was freshly prepared. The ratio of fresh to remaining slurry was 3.4/1, so the feed slurry can be considered only partially catalyst impregnated.

The level of coal conversion to THF solubles for the three periods of Run 115 was low at 72%. A total of 8 extractions were done, and there were no differences between the results from periods with feed slurries blended for two days and that with fresh slurry. There were also high yields of pre-asphaltenes, asphaltenes, and gases. Therefore, the approach must be studied further and improved.

Product Analyses: The analyses of the individual product fractions of the above runs are given in Table 30; the averages are given in Table 31.

The analyses followed expected trends. Hydrogen contents increased in the following order: THF insolubles > pre-asphaltenes > asphaltenes > oils. The nitrogen and oxygen contents of the pre-asphaltenes were equal to or greater than those of the THF insolubles. The levels of nitrogen and, in most cases, oxygen in the asphaltenes were high, even when there were high levels of retrogressive reactions between the coal-derived species and the solvents. It appeared that there was insufficient hydrogenation severity for both effective liquefaction and hydrocracking. As typical of most coal liquefaction, the level of desulfurization was reasonable for all fractions.

Observations/Conclusions on AU-51 Unit Runs: The above results indicate that the liquefaction runs were not carried out with sufficient reaction severity. In addition, the swelling of coal in combination with the use of low levels of catalyst precursor was not as effective as desired at the chosen reaction conditions. In fact, retrogressive reactions appeared to be promoted. With respect to coal conversion, this result is consistent with current batch autoclave liquefaction experiments.

There are a number of possible causes that will be explored first in batch autoclave experiments. In addition, selected runs will be made in flow units at short space times but higher temperatures to observe initial liquefaction reactions. Of particular interest are the possible interactions of (1) swelling solvent with catalyst precursor reactivity, (2) swelling solvent with the coal during liquefaction, and (3) between the solvent donor solvent quality and the catalyzed reaction of the swollen/impregnated coal.

Subtask 2.5: Solids Separation/Alternative Bottoms Processing

The objective of this subtask is to evaluate and develop 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 preliminary economics assessment of the battery limits of solvent deasphalting units, delayed coking units, or both, as incorporated in an integrated processing scheme, will be then be undertaken.

The batch deasphalting screening tests were completed and reported in the previous quarterly (Report Number Q-05). It was shown that the controlling variable was the type of solvent (i.e., paraffin or aromatic). The deasphalted oil (DAO) yield was essentially independent of temperature and solvent/feed ratio. The yield averaged 61.3 wt% for the C₅-C₇ paraffin solvents and was 80.6 wt% for the single test with xylene. These numbers were consistent with the feedstock composed of 65.9 wt% oil (paraffin-soluble material) and 76.8 wt% soluble in toluene. None of these solvents provided the means for readily achieving lower yields and achieving the desired 40 to 60 wt% DAO yield range. n-Butane was the first solvent that readily permitted the use of temperature as a controlling variable. Butane's solvent power, however, limited the maximum yield to about 50 wt% DAO. In the case of butane/pentane blends, as the pentane level was increased above about 50 vol%, the DAO yield peaked and then decreased. It appears that as more oil was extracted with successively higher concentrations of pentane in the blend, the asphalt phase became too viscous for effective extraction at the test temperature (290°F). While the oil yield at 290°F for n-butane/pentane would not exceed 55 wt%, pentane alone at 380°F provided an oil yield (63.6 wt%) that met the desired goal.

The production of asphalt for the transport tests was undertaken with target deasphalted oil yields of 40, 50 and 60 wt% of feed. This would produce asphalt with ash levels ranging from 20 to 30 wt% for the transport tests. The results for asphalt production are shown below:

Asphalt Production for Transport Tests

Solvent	S/F Ratio (V/V at 60°F)	Temp. (°F)	Yields (wt%)			Ash (wt%)	
			Oil	Asphalt	Recov.	Oil	Asphalt
90%C ₄ , 10%C ₅	4	290	41.0	58.0	99.0	0.001	21.03
Pentane	4	340	54.1	48.9	103.0	0.008	24.14
Hexane	4	400	59.8	41.3	101.1	0.003	27.23

The asphalt from the butane/pentane blend, containing 21.03 wt% ash, was selected for the first transport test. A test setup was assembled to include the following:

1. A steam heated, pressurized feed tank mounted on a shaker assembly.
2. An electrically-heated flexible metal hose as suction line to the metering pump.
3. A Zenith precision gear metering pump applicable for suspended fine solids slurry transport.
4. A pressure-control valve and controller to maintain the pump discharge pressure above that of the feed tank.

5. A steam-heated and enclosed receiving tank to collect transport product.

The asphalt was prepared by crushing and screening through a No. 50 sieve ($\sim 300 \mu\text{m}$). It was then blended at transport temperature ($\sim 275^\circ\text{F}$) with an equal volume of solvent (90% C_4 , 10% C_6) for three (3) hours before conducting the pumping test over a four (4) hour period. The test was successful and represents the first demonstration that a coal-derived asphalt with a heavy solids loading could be handled in FWDC pilot plant equipment.

Two transport tests using the asphalts containing the higher ash levels (24.14 wt% and 27.23 wt%), were undertaken using the above unit at $\sim 275^\circ\text{F}$. Neither test was able to demonstrate an ability to properly transport these materials. Visual examination suggested that the asphalt never reached a stage of fluidity where the solvent could effectively aid in its transport through the system.

Therefore, the test equipment was modified to reach temperatures in the range of $350\text{--}400^\circ\text{F}$. This was accomplished by adding three independently controlled electrical trace heaters to the batch deasphalter, the suction line to the pump and a portion of the line downstream of the pump, respectively, and installing additional thermocouples to monitor transport temperatures. Again, neither test was able to demonstrate an ability to properly transport these materials. The operating parameters reached the point where the pressure ratings on some equipment and frequent failure of the flexible metal hose connection limited any further experimental increases of temperature. The asphalt containing approximately 21 wt% ash, which has been transported successfully, was therefore selected for the delayed coking experimental program.

CONCLUSIONS AND FUTURE WORK

Experimentation is confirming some aspects of the overall process concept, but it is also raising problem areas. Black Thunder subbituminous coal can be effectively beneficiated to about 4 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-51) 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 may be an interaction between the Molyvan-L catalyst precursor and swelling solvent that effects coal liquefaction. It is also noted that the most effective swelling solvent, dimethyl sulfoxide, was detrimental to subsequent liquefaction. Therefore, more extensive research is needed to develop an

effective process. With respect to solids separation and recovery, the solvent system for the deasphalting process has been established. An asphalt stream containing 21% ash has been handled successfully in the transport tests, and delayed coking tests are underway using this stream.

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7/30/93

Table 1.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 10 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	36.13	36.13	2.39	2.39	36.37	36.37
1 min.	15.97	52.10	2.38	2.39	16.07	52.45
2 min.	16.81	68.91	2.33	2.37	16.93	69.38
4 min.	0.00	68.91	0.00	2.37	0.00	69.38
8 min.	0.00	68.91	0.00	2.37	0.00	69.38
Refuse	31.09	100.00	4.50	3.03	30.62	100.00
Total	100.00		3.03		100.00	

Table 2.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 20 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	29.85	29.85	2.49	2.49	30.02	30.02
1 min.	14.18	44.03	2.41	2.46	14.27	44.30
2 min.	14.18	58.21	2.35	2.44	14.28	58.58
4 min.	6.72	64.93	2.44	2.44	6.76	65.34
8 min.	0.00	64.93	0.00	2.44	0.00	65.34
Refuse	35.07	100.00	4.20	3.06	34.66	100.00
Total	100.00		3.06		100.00	

Table 3.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 µl
Collector: Dodecane 4500 µl
Frother: MIBC 50 µl

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	45.69	45.69	1.95	1.95	46.03	46.03
1 min.	21.55	67.24	2.27	2.05	21.64	67.68
2 min.	18.10	85.34	2.13	2.07	18.21	85.88
4 min.	7.76	93.10	2.44	2.10	7.78	93.66
8 min.	0.00	93.10	0.00	2.10	0.00	93.66
Refuse	6.90	100.00	10.52	2.68	6.34	100.00
Total	100.00		2.68		100.00	

Table 4.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 100 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	68.06	68.06	1.77	1.77	69.04	69.04
1 min.	15.28	83.33	2.20	1.85	15.43	84.47
2 min.	0.00	83.33	0.00	1.85	0.00	84.47
4 min.	0.00	83.33	0.00	1.85	0.00	84.47
8 min.	0.00	83.33	0.00	1.85	0.00	84.47
Refuse	16.67	100.00	9.80	3.17	15.53	100.00
Total	100.00		3.17		100.00	

Table 5.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 500 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	28.69	28.69	2.28	2.28	28.85	28.85
1 min.	20.72	49.40	2.15	2.23	20.87	49.72
2 min.	21.12	70.52	2.18	2.21	21.26	70.98
4 min.	16.33	86.85	2.13	2.20	16.46	87.44
8 min.	0.00	86.85	0.00	2.20	0.00	87.44
Refuse	13.15	100.00	7.17	2.85	12.56	100.00
Total	100.00		2.85		100.00	

Table 6.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 µl
Collector: Dodecane 4500 µl
Frother: MIBC 50 µl

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 500 ml. All the slurry was taken to conduct the experiment. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	12.85	12.85	2.83	2.83	12.89	12.89
1 min.	7.83	20.68	2.65	2.76	7.87	20.76
2 min.	13.86	34.54	2.69	2.73	13.92	34.67
4 min.	15.26	49.80	2.52	2.67	15.35	50.03
8 min.	20.08	69.88	2.51	2.62	20.21	70.24
Refuse	30.12	100.00	4.26	3.12	29.76	100.00
Total	100.00		3.12		100.00	

Table 7.

Experiment type:	Spherical Agglomeration/Flotation	
Material Type:	SO₂ treated sample from AMOCO	
Surfactant:	BC1	125 μl
Collector:	Dodecane	1125 μl
Frother:	MIBC	50 μl
Conditions:	<p><i>Grinding:</i> Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.</p> <p><i>Spherical Agglomeration:</i> Slurry from the mill was completed to 1000 ml. 100 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.</p> <p><i>Flotation:</i> 1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.</p>	
Feed Ash:	3.2%	

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	55.13	55.13	2.35	2.35	55.61	55.61
1 min.	19.30	74.43	2.89	2.49	19.35	74.96
2 min.	0.00	74.43	0.00	2.49	0.00	74.96
Refuse	25.57	100.00	5.21	3.19	25.04	100.00
Total	100.00		3.19		100.00	

Table 8.

Experiment type:	Spherical Agglomeration/Flotation	
Material Type:	SO₂ treated sample from AMOCO	
Surfactant:	BC1	250 μl
Collector:	Dodecane	2250 μl
Frother:	MIBC	50 μl
Conditions:	<p><i>Grinding:</i> Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.</p> <p><i>Spherical Agglomeration:</i> Slurry from the mill was completed to 1000 ml. 100 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.</p> <p><i>Flotation:</i> 1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.</p>	
Feed Ash:	3.2%	

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	71.21	71.21	1.83	1.83	72.23	72.23
1 min.	15.74	86.96	2.53	1.96	15.85	88.08
2 min.	0.00	86.96	0.00	1.96	0.00	88.08
Refuse	13.04	100.00	11.52	3.20	11.92	100.00
Total	100.00		3.20		100.00	

Table 9.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 500 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*
Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.
Spherical Agglomeration:
Slurry from the mill was completed to 1000 ml. 100 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.
Flotation:
1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.
Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	75.18	75.18	1.82	1.82	76.22	76.22
1 min.	14.37	89.55	2.88	1.99	14.41	90.63
2 min.	0.00	89.55	0.00	1.99	0.00	90.63
Refuse	10.45	100.00	13.15	3.16	9.37	100.00
Total	100.00		3.16		100.00	

Table 10.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 1000 μ l
Collector: Dodecane 9000 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*
 Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.
Spherical Agglomeration:
 Slurry from the mill was completed to 1000 ml. 100 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.
Flotation:
 1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.
Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	45.33	45.33	1.97	1.97	45.86	45.86
1 min.	23.68	69.00	1.88	1.94	23.98	69.84
2 min.	16.67	85.67	2.21	1.99	16.82	86.66
Refuse	14.33	100.00	9.82	3.11	13.34	100.00
Total	100.00		3.11		100.00	

Table 11.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 µl
Collector: Dodecane 4500 µl
Frother: MIBC 50 µl

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill in the presence of 20 mg Calgon per kg of coal.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	44.09	44.09	1.82	1.82	44.68	44.68
1 min.	21.26	65.35	2.00	1.88	21.50	66.19
2 min.	17.32	82.68	2.02	1.91	17.52	83.70
4 min.	9.45	92.13	2.47	1.97	9.51	93.21
8 min.	0.00	92.13	0.00	1.97	0.00	93.21
Refuse	7.87	100.00	16.50	3.11	6.79	100.00

Table 12.

Experiment type: Spherical Agglomeration/Flotation
 Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
 Collector: Dodecane 4500 μ l
 Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill in the presence of 100 mg Calgon per kg of coal.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	43.07	43.07	2.00	2.00	43.53	43.53
1 min.	18.25	61.31	1.94	1.98	18.46	61.99
2 min.	16.79	78.10	2.04	1.99	16.96	78.95
4 min.	8.76	86.86	2.36	2.03	8.82	87.78
8 min.	0.00	86.86	0.00	2.03	0.00	87.78
Refuse	13.14	100.00	9.80	3.05	12.22	100.00

Table 13.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill in the presence of 500 mg Calgon per kg of coal.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	38.58	38.58	1.87	1.87	39.07	39.07
1 min.	17.32	55.91	2.03	1.92	17.51	56.58
2 min.	20.47	76.38	1.87	1.91	20.73	77.31
4 min.	14.96	91.34	2.13	1.94	15.11	92.42
8 min.	0.00	91.34	0.00	1.94	0.00	92.42
Refuse	8.66	100.00	15.20	3.09	7.58	100.00

Table 14.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill in the presence of 2000 mg Calgon per kg of coal.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 30 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	41.41	41.41	1.93	1.93	41.91	41.91
1 min.	16.41	57.81	1.92	1.93	16.61	58.52
2 min.	18.75	76.56	2.00	1.95	18.97	77.49
4 min.	14.06	90.63	2.11	1.97	14.21	91.70
8 min.	0.00	90.63	14.20	1.97	0.00	91.70
Refuse	9.38	100.00	14.20	3.12	8.30	100.00

Table 15.

Experiment type: Spherical Agglomeration/Flotation
 Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
 Collector: Dodecane 4500 μ l
 Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 00 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	40.74	40.74	1.94	1.94	41.12	41.12
1 min.	19.26	60.00	1.96	1.95	19.43	60.55
2 min.	19.26	79.26	2.03	1.97	19.42	79.97
4 min.	10.37	89.63	2.34	2.01	10.42	90.39
8 min.	0.00	89.63	0.00	2.01	0.00	90.39
Refuse	10.37	100.00	9.95	2.83	9.61	100.00
Total	100.00		2.83		100.00	

Table 16.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 04 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	39.84	39.84	1.94	1.94	40.20	40.20
1 min.	22.66	62.50	2.00	1.96	22.85	63.05
2 min.	17.97	80.47	1.96	1.96	18.13	81.18
4 min.	10.94	91.41	2.24	1.99	11.00	92.18
8 min.	0.00	91.41	0.00	1.99	0.00	92.18
Refuse	8.59	100.00	11.54	2.82	7.82	100.00
Total	100.00		2.82		100.00	

Table 17.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 μ l
Collector: Dodecane 4500 μ l
Frother: MIBC 50 μ l

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 10 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	39.84	39.84	1.91	1.91	40.19	40.19
1 min.	21.09	60.94	1.99	1.94	21.26	61.45
2 min.	18.75	79.69	1.98	1.95	18.90	80.35
4 min.	11.72	91.41	2.51	2.02	11.75	92.09
8 min.	0.00	91.41	0.00	2.02	0.00	92.09
Refuse	8.59	100.00	10.52	2.75	7.91	100.00
Total	100.00		2.75		100.00	

Table 18.

Experiment type: Spherical Agglomeration/Flotation
Material Type: SO₂ treated sample from AMOCO

Surfactant: BC1 100 µl
Collector: Dodecane 4500 µl
Frother: MIBC 50 µl

Conditions: *Grinding:*

Wet grinding of a 50 g. sample in 100 ml of water for 40 min in a planetary mill.

Spherical Agglomeration:

Slurry from the mill was completed to 1000 ml. 200 ml slurry was taken and completed to 500 ml. This slurry was kept in an ultrasonic bath for 60 min. It was mixed in a blender for 1 min. at a setting of 2. Oil + surfactant was added and the slurry was blended for 3 more min. at a setting of 4. A final blending of 2 min. at a setting of 2 was applied.

Flotation:

1 min. conditioning – MIBC addition; 1 min. conditioning – flotation.

Feed Ash: 3.2%

Product	Yield(%)	Cumulative Yield(%)	Ash(%)	Cum. Ash (%)	CMR (%)	Cumulative CMR (%)
30 sec.	37.96	37.96	2.04	2.04	38.28	38.28
1 min.	18.25	56.20	2.05	2.04	18.40	56.68
2 min.	21.17	77.37	1.98	2.03	21.36	78.04
4 min.	12.41	89.78	2.18	2.05	12.50	90.53
8 min.	0.00	89.78	0.00	2.05	0.00	90.53
Refuse	10.22	100.00	10.01	2.86	9.47	100.00
Total	100.00		2.86		100.00	

Proximate and Ultimate Analysis of Black Thunder and SO₂ Treated Black Thunder Coals*

	Proximate Analysis			
	Black Thunder		SO ₂ Treated Black Thunder	
	As Received	Dry Basis	As Received	Dry Basis
% Moisture	11.20		9.88	
% Ash	5.43	6.12	3.08	3.42
% Volatile	44.53	50.15	41.74	46.32
% Fixed Carbon	38.84	43.73	45.30	50.26
Total	100.00	100.00	100.00	100.00
Btu/lb	10606	11944	10993	12198
% Sulfur	0.39	0.44	0.73	0.81
maf Btu		12723		12630
	Ultimate Analysis			
	Black Thunder		SO ₂ Treated Black Thunder	
	As Received	Dry Basis	As Received	Dry Basis
% Moisture	11.20		9.88	
% Carbon	61.87	69.67	63.57	70.54
% Hydrogen	4.33	4.88	4.46	4.95
% Nitrogen	0.81	0.91	0.80	0.89
% Sulfur	0.39	0.44	0.73	0.81
% Ash	5.43	6.12	3.08	3.42
% Oxygen (diff)	15.97	17.98	17.48	19.39

* Commercial Testing and Engineering Co., Birmingham, AL laboratory.

Reaction Conditions for Coal Swelling Reactivity Test Using Swelled Coals

Coal	Black Thunder (untreated and SO ₂ treated)
Temperature	410°C
Agitation	500 cpm
H ₂ Pressure	1250 psig
Pyrene	0.67 g
Coal	1.33 g
Solvent	2.0 g
Solvent for Reaction	1-methylnaphthalene
Catalyst Loading	600-800 ppm
Reaction Time	30 min
Solvent for Coal Swelling	Methanol
Swelling Time	24 hr
Analysis	(1) Coal products removed from reactors with THF
	(2) GC analysis performed for pyrene products
	(3) Coal conversion to THF solubles determined

Table 21

**Mass of Swelling Solvent in SO₂ Treated and Untreated Coals
Using 1-Methylnaphthalene as the Reaction Solvent**

Catalyst	Catalyst Loading (ppm)	Coal Mass (g)	Solvent Mass Absorbed (g)	% ΔV
Swelling Agent: THF Coal: untreated				
None	0	1.6481	1.4497	29.2
None	0	1.6452	1.6434	39.1
Molyvan L	684	1.6485	1.6400	36.4
Molyvan L	638	1.6493	1.5791	39.1
Ni Octoate	619	1.6505	1.6921	41.7
Ni Octoate	611	1.6472	1.5637	40.9
Swelling Agent: Methanol Coal: untreated				
None	0	1.6391	1.1640	27.3
None	0	1.6405	1.0405	26.1
Molyvan L	676	1.6085	0.9976	18.2
Molyvan L	703	1.6210	0.8984	19.0
Ni Octoate	648	1.6316	0.9552	18.2
Ni Octoate	747	1.6105	0.9537	19.0
Swelling Agent: Isopropanol Coal: untreated				
None	0	1.6342	1.1045	13.0
None	0	1.6363	1.3059	20.8
Molyvan L	596	1.6329	1.2173	13.0
Molyvan L	649	1.6400	1.1612	22.7
Ni Octoate	657	1.6331	0.9619	19.0
Ni Octoate	659	1.6318	1.2779	13.6

Table 21
(cont.)

Mass of Swelling Solvent in SO₂ Treated and Untreated Coals
Using 1-Methylnaphthalene as the Reaction Solvent (Continued)

Catalyst	Catalyst Loading (ppm)	Coal Mass (g)	Solvent Mass Absorbed (g)	%ΔV
Swelling Agent: DMSO Coal: untreated				
None	0	1.6245	3.8707	109.1
None	0	1.6249	4.0666	114.3
Molyvan L	594	1.6241	4.3110	117.4
Molyvan L	658	1.6107	3.1809	114.3
Ni Octoate	643	1.6211	3.6091	95.7
Ni Octoate	643	1.6239	3.5189	109.1
Swelling Agent: THF Coal: SO ₂ treated				
None	0	1.5271	2.2323	85.7
None	0	1.5312	2.0231	89.5
Molyvan L	720	1.5311	1.9879	80.0
Molyvan L	645	1.5274	2.0580	78.9
Ni Octoate	681	1.5310	2.1304	100.0
Ni Octoate	648	1.5279	2.2049	94.7
Swelling Agent: Methanol Coal: SO ₂ treated				
None	0	1.5049	1.1282	40.0
None	0	1.5238	1.0295	36.8
Molyvan L	659	1.5123	0.8919	31.6
Molyvan L	684	1.5161	1.0070	42.1
Ni Octoate	656	1.4995	1.0274	26.3
Ni Octoate	653	1.4998	0.9251	36.8

Table 21
(cont.)

Mass of Swelling Solvent in SO₂ Treated and Untreated Coals
Using 1-Methylnaphthalene as the Reaction Solvent (Continued)

Catalyst	Catalyst Loading (ppm)	Coal Mass (g)	Solvent Mass Absorbed (g)	%ΔV
Swelling Agent: Isopropanol Coal: SO ₂ treated				
None	0	1.5047	1.3465	45.0
None	0	1.5140	1.5310	45.0
Molyvan L	624	1.5105	1.3264	35.0
Molyvan L	659	1.5095	1.3122	42.1
Ni Octoate	692	1.5167	1.2375	27.8
Ni Octoate	655	1.5192	1.3097	36.8
Swelling Agent: DMSO Coal: SO ₂ treated				
None	0	1.5128	5.6584	195.0
None	0	1.4968	4.7448	157.9
Molyvan L	797	1.4969	4.0154	178.9
Ni Octoate	661	1.4851	5.1239	163.2
Ni Octoate	744	1.3060	5.3900	180.0

Coal and Pyrene Conversions for Untreated Black Thunder Coal under Swelled and Unswelled Conditions

Catalyst	Catalyst Loading (ppm)	Change in Volume (%ΔV)	Coal (weight %)	Pyrene Conversion (mole %)	Coal Conversion (weight %)	Total Recovery (%)
No Swelling ■ 1-Methylnaphthalene Reaction Solvent						
None	0	NA	30.8	4.3	49.8	101.3
None	0	NA	30.8	2.7	48.7	105.4
Molyvan L	681	NA	30.5	26.2	80.3	95.2
Molyvan L	736	NA	30.5	33.8	84.5	98.6
Ni Octoate	599	NA	30.7	7.0	67.8	95.9
Ni Octoate	608	NA	30.5	6.6	73.6	97.6
THF Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	29.2	30.8	0.0	66.0	91.8
None	0	39.1	30.8	0.0	53.4	96.0
Molyvan L	684	36.4	30.6	17.7	86.2	96.6
Molyvan L	638	39.1	30.7	19.5	88.4	91.9
Ni Octoate	619	41.7	30.7	2.4	77.0	124.5
Ni Octoate	611	40.9	30.7	2.3	76.9	107.7
Methanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	27.3	30.7	1.2	55.8	108.6
None	0	26.1	30.8	0.8	51.9	106.0
Molyvan L	676	18.2	30.1	23.0	87.3	89.4
Molyvan L	703	19.0	30.3	25.5	88.6	97.2
Ni Octoate	648	18.2	30.5	11.2	85.5	92.3
Ni Octoate	747	19.0	30.3	10.6	85.4	105.7

**Coal and Pyrene Conversion for Untreated Black Thunder Coal
under Swelled and Unswelled Conditions (continued)**

Catalyst	Catalyst Loading (ppm)	Change in Volume (%ΔV)	Coal (weight %)	Pyrene Conversion (mole %)	Coal Conversion (weight %)	Total Recovery (%)
Isopropanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	13.0	30.7	1.6	60.9	105.7
None	0	20.8	30.7	0.0	62.5	95.6
Molyvan L	596	13.0	30.5	18.1	88.3	109.9
Molyvan L	649	22.7	30.5	19.2	87.8	105.2
Ni Octoate	657	19.0	30.5	2.5	76.8	106.8
Ni Octoate	659	13.6	30.5	1.9	78.3	99.5
DMSO* Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	109.1	30.6	0.8	32.1	96.1
None	0	114.3	30.6	1.5	26.4	89.5
Molyvan L	594	117.4	30.4	11.6	78.5	111.7
Molyvan L	658	114.3	30.2	11.5	81.2	109.9
Ni Octoate	643	95.7	30.4	5.5	61.0	103.9
Ni Octoate	634	109.1	30.4	4.4	66.1	102.2

NA = not applicable

* DMSO requires 48 hours for swelling

Table 23

Product Distributions for Pyrene Hydrogenation for Reactions with Untreated Black Thunder Coal

Catalyst	Catalyst Loading (ppm)	% HYD	Product Distribution (mole %)			
			PYR	DHP	THP	HHP
No Swelling ■ 1-Methylnaphthalene Reaction Solvent						
None	0	2.5	95.7	2.7	0.0	1.6
None	0	1.6	97.3	1.7	0.0	1.0
Molyvan L	681	9.5	73.8	23.9	2.3	0.0
Molyvan L	736	12.9	66.2	30.1	2.5	1.2
Ni Octoate	599	2.3	93.0	7.0	0.0	0.0
Ni Octoate	608	2.2	93.4	6.6	0.0	0.0
THF Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	0.0	100	0.0	0.0	0.0
None	0	0.0	100	0.0	0.0	0.0
Molyvan L	684	5.9	82.3	17.7	0.0	0.0
Molyvan L	638	6.5	80.5	19.5	0.0	0.0
Ni Octoate	619	1.1	97.6	1.9	0.0	0.5
Ni Octoate	611	1.2	97.7	1.6	0.0	0.7
Methanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	0.9	98.8	0.4	0.0	0.8
None	0	0.6	99.2	0.3	0.0	0.5
Molyvan L	676	7.7	77.0	23.0	0.0	0.0
Molyvan L	703	9.3	74.5	23.0	2.5	0.0
Ni Octoate	648	3.7	88.8	11.2	0.0	0.0
Ni Octoate	747	4.2	89.4	9.5	0.0	1.1

Product Distribution for Pyrene Hydrogenation for Reactions with Untreated Black Thunder Coal (continued)

Catalyst	Catalyst Loading (ppm)	% HYD	Product Distribution (mole %)			
			PYR	DHP	THP	HHP
Isopropanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	1.2	98.4	0.6	0.0	1.0
None	0	0.0	100.0	0.0	0.0	0.0
Molyvan L	596	6.4	81.9	17.1	0.7	0.3
Molyvan L	649	6.8	80.8	18.2	0.5	0.5
Ni Octoate	657	1.2	97.5	1.9	0.0	0.6
Ni Octoate	659	1.0	98.1	1.3	0.0	0.6
DMSO Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	0.5	99.2	0.4	0.0	0.4
None	0	0.8	98.5	1.0	0.0	0.5
Molyvan L	594	4.6	88.4	10.3	0.3	1.0
Molyvan L	658	4.5	88.5	10.3	0.3	0.9
Ni Octoate	643	2.7	94.5	4.1	0.0	1.4
Ni Octoate	634	1.9	95.6	3.7	0.0	0.7

Table 24

Coal and Pyrene Conversions for SO₂ Treated Black Thunder Coal Under Swelled and Unswelled Conditions

Catalyst	Catalyst Loading (ppm)	Change in Volume (% Δ V)	Coal (weight %)	Pyrene Conversion (mole %)	Coal Conversion (weight %)	Total Recovery (%)
No Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	NA	31.6	0.7	48.1	101.7
None	0	NA	31.6	2.3	46.5	117.3
Molyvan L	614	NA	31.4	29.9	89.0	97.7
Molyvan L	631	NA	31.3	25.7	88.0	95.4
Ni Octoate	595	NA	31.4	10.0	73.3	96.8
Ni Octoate	797	NA	31.4	15.0	79.2	95.7
THF Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	85.7	31.5	0.0	64.7	89.9
None	0	89.5	31.6	0.0	51.2	96.0
Molyvan L	720	80.0	31.3	25.3	90.1	101.1
Molyvan L	645	78.9	31.2	25.4	92.2	103.3
Ni Octoate	681	100.0	31.4	2.4	68.4	114.6
Ni Octoate	648	94.7	31.4	1.9	76.6	108.8
Methanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	40.0	31.2	1.5	43.5	90.6
None	0	36.8	31.5	1.4	40.1	109.8
Molyvan L	659	31.6	31.1	26.9	90.9	103.3
Molyvan L	684	42.1	31.1	33.1	89.0	111.9
Ni Octoate	656	26.3	31.0	5.5	77.9	105.9
Ni Octoate	653	36.8	31.0	4.3	79.4	102.9

Table 24
(cont.)

Coal and Pyrene Conversions for SO₂ Treated Black Thunder Coal Under Swelled and Unswelled Conditions (continued)

Catalyst	Catalyst Loading (ppm)	Change in Volume (%ΔV)	Coal (weight %)	Pyrene Conversion (mole %)	Coal Conversion (weight %)	Total Recovery (%)
Isopropanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	45.0	31.2	1.7	58.5	111.0
None	0	45.0	31.3	1.5	54.3	110.8
Molyvan L	624	35.0	31.0	23.0	91.7	109.9
Molyvan L	659	42.1	31.1	19.6	90.8	110.1
Ni Octoate	692	27.8	31.2	3.4	70.3	105.0
Ni Octoate	655	36.8	31.3	4.3	77.5	110.4
DMSO Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	195	31.3	1.0	32.0	92.2
None	0	158	31.2	1.5	27.0	98.3
Molyvan L	797	179	30.8	13.7	78.1	115.3
Ni Octoate	661	163	30.8	6.3	43.3	105.7
Ni Octoate	744	180	28.3	7.8	56.8	98.2

NA = not applicable

**Product Distributions for Pyrene Reactions with SO₂ Treated
Black Thunder Coal**

Catalyst	Catalyst Loading (ppm)	%HYD	Product Distribution (mole %)			
			PYR	DHP	THP	HHP
No Swelling ■ 1-Methylnaphthalene Reaction Solvent						
None	0	0.2	99.3	0.7	0.0	0.0
None	0	1.2	97.7	1.6	0.0	0.7
Molyvan L	614	10.8	70.1	27.4	2.5	0.0
Molyvan L	631	9.9	74.3	23.4	2.3	0.0
Ni Octoate	595	3.3	90.0	10.0	0.0	0.0
Ni Octoate	795	5.0	85.0	15.0	0.0	0.0
THF Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	0.0	100	0.0	0.0	0.0
None	0	0.0	100	0.0	0.0	0.0
Molyvan L	720	9.2	74.7	23.1	2.2	0.0
Molyvan L	645	9.0	74.6	23.8	1.6	0.0
Ni Octoate	681	1.3	97.6	1.6	0.0	0.8
Ni Octoate	648	1.1	98.1	1.3	0.0	0.6
Methanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	1.0	98.5	0.7	0.0	0.8
None	0	1.0	98.6	0.7	0.0	0.7
Molyvan L	659	10.1	73.1	24.3	1.5	1.1
Molyvan L	684	12.2	66.9	30.3	2.2	0.6
Ni Octoate	656	2.2	94.5	5.0	0.0	0.5
Ni Octoate	653	1.4	95.7	4.3	0.0	0.0

Table 25
(cont.)

Product Distribution for Pyrene Reactions for Pyrene Reactions with SO₂
Treated Black Thunder Coal (continued)

Catalyst	Catalyst Loading (ppm)	%HYD	Product Distribution (mol %)			
			PYR	DHP	THP	HHP
Isopropanol Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	1.1	98.3	0.9	0.0	0.8
None	0	1.0	98.5	0.8	0.0	0.7
Molyvan L	624	8.8	77.0	20.7	1.4	0.9
Molyvan L	659	7.2	80.4	18.2	0.8	0.6
Ni Octoate	692	2.1	96.6	1.9	0.0	1.5
Ni Octoate	655	2.1	95.7	3.4	0.0	0.9
DMSO Swelling Solvent ■ 1-Methylnaphthalene Reaction Solvent						
None	0	0.6	99.0	0.7	0.0	0.3
None	0	0.8	98.5	1.0	0.0	0.5
Molyvan L	797	5.7	86.5	11.7	0.6	1.4
Ni Octoate	661	2.9	93.7	5.1	0.0	1.2
Ni Octoate	744	3.0	92.2	7.1	0.0	0.7

Tables
26, 27

**Summary Table for Untreated Black Thunder Coal using
1-Methylnaphthalene as the Reaction Solvent***

Swelling Solvents	Percentage Change in Coal Conversion (weight %)			Percentage Change in Pyrene Conversion (mole %)			Swelling Index (ΔV%)
	Catalyst			Catalyst			
	None	Molyvan L	Ni Octoate	None	Molyvan L	Ni Octoate	
THF	+10.5	+4.9	+6.3	-3.5	-11.4	-4.5	37.3
Methanol	+4.6	+5.6	+14.8	-2.5	-5.8	4.1	21.3
Isopropanol	+12.5	+5.7	+6.9	-2.7	-11.4	-4.6	17.0
DMSO	-20.0	-2.6	-7.2	-2.4	-18.5	-1.9	110.0

- * All percentages were averaged for each solvent-catalyst system.
+ Means that the percentage increased; - means that the percentage decreased.

**Summary Table for SO₂ Treated Black Thunder Coal using
1-Methylnaphthalene as the Reaction Solvent***

Swelling Solvents	Percentage Change in Coal Conversion (weight %)			Percentage Change in Pyrene Conversion (mole %)			Swelling Index (ΔV%)
	Catalyst			Catalyst			
	None	Molyvan L	Ni Octoate	None	Molyvan L	Ni Octoate	
THF	+10.7	+2.7	-3.8	-1.5	-2.5	-10.4	88.1
Methanol	-5.5	+1.5	+2.4	-0.1	+2.2	-7.6	35.6
Isopropanol	+9.1	+2.8	-2.4	+0.1	-6.5	-8.7	38.6
DMSO	-17.6	-10.4	-26.2	-0.3	-14.1	-5.5	175.0

- * All percentage are averaged for each solvent-catalyst combination.
+ Means that the percentage increased; - means that the percentage decreased.

Table 28

SOLVENT ANALYSES

Designation	V-1074 (650-1000°F)	FSN-113 (Resid)	Blend 80/20
Elemental Analyses, Wt%			
Carbon	87.23	89.56	88.41
Hydrogen	9.68	8.26	8.97
Nitrogen	0.53	0.80	0.66
Oxygen	2.52	1.33	1.92
Sulfur	0.04	0.05	0.04
Distillation, wt%			
IBP-650 F	--	8.8	--
650-975 F	--	53.7	--
975+ F	--	37.5	--
Solubility, wt%			
THF Insolubles	0.01	0.09	0.05
Toluenes Insolubles	0.12	1.31	0.71
Hexane Insolubles	1.32	15.33	8.32

Table 29

RESULTS OF THE CONTINUOUS AU-51 UNIT RUNS
(Based on normalized results)

<u>Run Number</u>	93	102	113	115
Coal (1)	--	--	Swollen	Swollen
Catalyst	None	MV-L	--	MV-L
Solvent	V-1074	V-1074	80/20	80/20
			V-1074/Resid	V-1074/Resid
Space Time, Min	80.7	89.0	78.7	71.4
<u>Conversion, %MAF(2)</u>				
THF Solubles	79.2	81.4	82.2	72.0
Tol Solubles	68.9	63.0	69.8	48.8
Hexane Solubles	-8.1	-45.5	-37.9	-96.0
C1-C3 Gases	5.4	9.1	8.4	13.9
C4+ Gases	2.7	3.6	2.4	12.3
CO, CO2	4.4	5.4	4.8	6.8

Notes:

1. In Runs 93 & 102 SO₂-Treated Black Thunder coal was used.
In Run 113, SO₂-treated BT coal preswelled with 80/20toluene/THG.
In Run 115, THF was added to the liquefaction solvent before the run.
2. Conversions are based on Moisture, Ash Free feed coal.

Table 30

ANALYSES OF AU-51 PRODUCT STREAMS

Run Number 16103-93C

Sample	Solids	Pre-Asph	Asphaltenes	Oils
Yield,%	20.8	10.3	77.0	---
Elemental				
Carbon	74.28	84.14	87.59	89.41
Hydrogen	4.03	5.61	6.41	9.05
Nitrogen	1.30	1.58	1.22	0.40
Oxygen	7.66	7.35	3.65	1.29
Sulfur	0.95	0.28	0.18	0.07
Ash		--	--	--
Total	88.22	98.96	99.05	100.22

Run Number 16103-102C

Sample	Solids	Pre-Asph	Asphaltenes	Oils
Yield,%	18.6	18.4	100+	---
Elemental				
Carbon	81.22	85.26	87.45	89.78
Hydrogen	4.20	5.49	5.53	8.58
Nitrogen	1.36	1.33	1.52	0.49
Oxygen	5.75	5.87	4.91	1.39
Sulfur	0.47	0.19	0.18	0.09
Ash		--	--	--
Total	93.00	98.14	99.59	100.33

Run Number 16103-113C

Sample	Solids	Pre-Asph	Asphaltenes	Oils
Yield,%	17.8	12.4	100+	---
Elemental				
Carbon	77.45	85.46	88.27	89.42
Hydrogen	3.99	5.73	6.02	8.77
Nitrogen	1.28	1.42	1.29	0.43
Oxygen	6.20	6.04	3.43	1.06
Sulfur	0.78	0.12	0.16	0.09
Ash		--	--	--
Total	89.70	98.77	99.17	99.77

Run Number 16103-115C

Sample	Solids	Pre-Asph	Asphaltenes	Oils
Yield,%	28.0	23.2	100+	---
Elemental				
Carbon	78.98	86.68	88.60	89.22
Hydrogen	4.01	5.54	6.32	8.99
Nitrogen	1.23	1.30	1.16	0.41
Oxygen	5.09	5.49	3.19	1.20
Sulfur	0.77	0.19	0.17	0.52
Ash		--	--	--
Total	90.08	99.20	99.44	100.34

Table 31

AVERAGED ANALYSES OF AU-51 PRODUCT STREAMS
Run Numbers 16103-93C, 102C, 113C, 115C
(DCC entered 6/14/93)

Sample	Solids	Pre-Asph	Asphaltenes	Oils	Feed Coal SO ₂ -Treated	20/80 solvent
Carbon	77.98	85.39	87.98	89.46	71.26	88.41
Hydrogen	4.06	5.59	6.07	8.85	5.29	8.97
Nitrogen	1.29	1.41	1.30	0.43	1.03	0.66
Oxygen	6.18	6.19	3.80	1.24	18.38	1.92
Sulfur	0.74	0.20	0.17	0.19	0.80	0.04
Ash		--	--	--	3.24	----
Total	90.25	98.77	99.31	100.17	100.00	100.00

Elemental Ratios of the Fractions:

H/C	0.625	0.787	0.829	1.188	0.892	1.219
N/C	0.014	0.014	0.013	0.004	0.012	0.006
O/C	0.059	0.054	0.032	0.010	0.193	0.016
S/C	0.004	0.001	0.001	0.001	0.004	0.000

Figure 2a
Effect of Solvent Upon Molyvan-L Penetration

-70 mesh SO₂-treated Black Thunder
~100 ppm Mo Initial, 70 F

Figure 2b
Effect of Solvent Upon Molyvan-L Penetration

Figure 3a
Effect of Temperature Upon Molyvan-L Penetration
(THF and Toluene With -70 Mesh Coal)

Figure 3b
Effect of Temperature Upon Molyvan-L Penetration
(THF and DMSO With 8x60 Mesh Coal)

Figure 4a
Effect of Coal Size Upon Molyvan-L Penetration
(THF/Toluene at Ambient Temperature)

SO₂-treated Black Thunder
 ~100 ppm Mo Initial

Figure 4b
Effect of Coal Size Upon Molyvan-L Penetration
(IPA/Toluene at Ambient Temperature)

~100 ppm Mo Initial
 SO₂-treated Black Thunder coal

Figure 4c
Effect of Coal Size Upon Molyvan-L Penetration
(THF/Toluene at Elevated Temperature)

Figure 5
Effect of Catalyst Precursor Upon Penetration
(IPA/Toluene at Ambient)

Figure 6
Effect of Initial Catalyst Concentration Upon
Molyvan-L Penetration

8 x 60 mesh SO₂-treated Black Thunder
IPA/Toluene at Ambient

FIGURE 7
SCHEMATIC OF THE AU-51 FLOW UNIT

APPENDIX A
AU51 RESULTS

REPORT: (SUMMARY OF EXTRACTIONS)

DATE: 07/09/93

BOOK#: 16103-93 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: None

REPORT:	Extraction No	143A,B	143A,B
Description:	Run No.-----	093C	093C*
-----	-----	-----	-----
CONDITIONS:	PSIG	2000	2000
Temperature, F	(F)	800.0	800.0
Space Time, min	Min	80.7	80.7
Hydrogen (Brooksset)	SCFH	10.0	10.0
Lineout Time, Hr	Hrs	0.0	0.0
Run Time, after lineout	Hrs	11.0	11.0

MATERIAL BALANCE:			
Feed Rate (L/O+Run)	Gm/Hr	401.99	401.99
Slurry Product	Gm/Hr	412.72	375.99
Light Ends	Gm/Hr	0.00	0.00
C1-C3 Gases	Gm/Hr	6.35	6.35
C4+ Gases	Gm/Hr	3.20	3.20
Carbon Monoxide	Gm/Hr	0.63	0.63
Carbon Dioxide	Gm/Hr	4.58	4.58
Water	Gm/Hr	0.00	11.36
Recovery	(%)	106.3%	100.0%

YIELD: (Based on MAF Total Feed)

-----	-----	-----	-----
Hexane Solubles	Wt%	63.86	67.17
Asphaltenes	Wt%	25.68	23.40
Preasphaltenes	Wt%	3.43	3.12
Hexane Insoluble Fee	Wt%	36.14	32.83
TOL Insoluble MAF Fee	Wt%	10.45	9.44
THF Insoluble MAF Fee	Wt%	7.02	6.31

YIELD: (Based on MAF Coal Only)

-----	-----	-----	-----
Asphaltenes	Wt%	84.62	77.08
Preasphaltenes	Wt%	11.29	10.29
THF Solubles	Wt%	76.86	79.20
Hexane Insoluble	Wt%	119.05	108.17
TOL Insolubles	Wt%	34.43	31.09
THF Insolubles	Wt%	23.14	20.80
Light Ends	Wt%	0.00	0.00
C1+C2+C3 Gases	Wt%	5.41	5.41
C4+C5+C6 Gases	Wt%	2.72	2.72
Carbon Monoxide	Wt%	0.53	0.53
Carbon Dioxide	Wt%	3.90	3.90

EXTRACTION RESULTS:	Page:	143A,B	143A,B
-----	-----	-----	-----
THF Insolubles	Gms	5.386	5.386
Tol Insolubles	Gms	7.700	7.700
Hex Ins in Tol Sol	Gms	17.337	17.337
Hex & Tol Insol.	Gms	25.037	25.037
HEX Solubles	Gms	36.040	36.040
Extraction Feed	Gms	72.040	72.040
Recovery	(%)	84.78%	84.78%

REPORT:

BOOK#: 16103-93 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: None

ELEMENTAL ANALYSIS: (Complete Extraction)

Unconverted Coal (A)

C	Frn	0.7428
H	Frn	0.0403
N	Frn	0.0130
O	Frn	0.0766
S	Frn	0.0095
Ash	Frn	0.1178
Total	Frn	1.0000

Preasphaltenes (B)

C	Frn	0.8414
H	Frn	0.0561
N	Frn	0.0158
O	Frn	0.0735
S	Frn	0.0028
Total	Frn	0.9896

Asphaltenes (C)

C	Frn	0.8759
H	Frn	0.0641
N	Frn	0.0122
O	Frn	0.0365
S	Frn	0.0018
Total	Frn	0.9905

Oils (Hex Sol) (D)

C	Frn	0.8941
H	Frn	0.0905
N	Frn	0.0040
O	Frn	0.0129
S	Frn	0.0007
Total	Frn	1.0022

MATERIAL BALANCE:

DATE: 09-Jul 1992

BOOK#: 16103-93 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: None

RUN NO: 093C

800 F.

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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	86.44	234.94	0.00	0.00	321.38	321.38
H	1.29	0.00	6.42	26.07	0.00	25.40	32.49	59.18
N	0.00	0.00	1.24	1.43	0.00	0.00	2.67	2.67
O	10.29	0.00	22.30	6.79	0.00	0.00	29.09	39.38
S	0.00	0.00	0.97	0.11	0.00	0.00	1.08	1.08
ASH	0.00	3.69	0.00	0.00	0.00	0.00	3.69	3.70
TOTAL	11.58	3.69	117.38	269.34	0.00	25.40	390.41	427.40
FEED:	11.58	3.69	117.38	269.34	0.00	25.40	390.41	427.39

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.27	1.25	4.96	2.64	0.00	0.00	9.1
H	22.57	0.00	0.00	1.39	0.55	0.00	1.26	25.8
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.36	3.33	0.00	0.00	0.00	10.10	13.8
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	22.57	0.63	4.58	6.35	3.20	0.00	11.36	48.7
	22.57	0.63	4.58	6.35	3.20	0.00	11.36	48.7

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	 Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	 OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	9.12	22.92	11.27	87.83	240.24	362.26	371.39	371.39
H	24.51	1.24	0.75	6.43	24.32	32.74	57.25	34.68
N	0.00	0.40	0.21	1.22	1.07	2.91	2.91	2.91
O	3.69	2.36	0.98	3.66	3.47	10.47	14.16	14.16
S	0.00	0.29	0.04	0.18	0.19	0.70	0.70	0.70
ASH	0.00	3.64	0.00	0.00	0.00	3.64	3.64	3.64
TOTAL	37.31	30.86	13.25	99.32	269.29	412.72	450.04	427.47
	37.31	30.86	13.25	99.32	269.29	412.72	450.0	

MATERIAL BALANCE: DATE: 09-Jul 1992
 BOOK#: 16103-93 June 93
 COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.
 CATALYST: None

RUN NO: 093C* (Modified 100% 800 F. Page 1 of 12
 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	86.44	234.94	0.00	0.00	321.38	321.38
H	1.29	0.00	6.42	26.07	0.00	25.40	32.49	59.18
N	0.00	0.00	1.24	1.43	0.00	0.00	2.67	2.67
O	10.29	0.00	22.30	6.79	0.00	0.00	29.09	39.38
S	0.00	0.00	0.97	0.11	0.00	0.00	1.08	1.08
ASH	0.00	3.69	0.00	0.00	0.00	0.00	3.69	3.70
TOTAL	11.58	3.69	117.38	269.34	0.00	25.40	390.41	427.40
FEED:	11.58	3.69	117.38	269.34	0.00	25.40	390.41	427.39

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.27	1.25	4.96	2.64	0.00	0.00	9.1
H	22.57	0.00	0.00	1.39	0.55	0.00	0.00	24.5
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.36	3.33	0.00	0.00	0.00	0.00	3.7
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	22.57	0.63	4.58	6.35	3.20	0.00	0.00	37.3
	22.57	0.63	4.58	6.35	3.20	0.00	0.00	37.3

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	SLURRY OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	9.12	20.88	10.27	80.02	218.86	330.02	339.14	339.14
H	24.51	1.13	0.68	5.86	22.15	29.83	54.33	31.76
N	0.00	0.37	0.19	1.11	0.98	2.65	2.65	2.65
O	3.69	2.15	0.90	3.33	3.16	9.54	13.23	13.23
S	0.00	0.27	0.03	0.16	0.17	0.64	0.64	0.64
ASH	0.00	3.31	0.00	0.00	0.00	3.31	3.31	3.31
TOTAL	37.31	28.11	12.07	90.48	245.32	375.99	413.31	390.74
	37.31	28.11	12.07	90.48	245.32	375.99	413.3	

FORCED ELEMENTAL BALANCE:

DATE: 07/09/93

BOOK#: 16103-93 June 93
 COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.
 CATALYST: None

RUN NO: 093C

800 F.

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CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	FORCED....		SUBTOTAL EX.H-GAS	TOTAL
				H2O	H2S&NH3		
C		9.12	371.39	0.00	0.00	371.39	371.39
H	3.5%	1.94	34.68	3.15	0.02	37.85	60.42
N	0.0%	0.00	2.91	0	0.00	2.91	2.91
O	64.0%	3.69	14.16	25.22	0.00	39.38	39.38
S	35.4%	0.00	0.70	0.00	0.38	1.08	1.08
ASH		0.00	3.64	0.00	0.00	3.64	3.64
TOTAL		14.74	427.47	28.38	0.41	456.25	478.82

RUN NO: 093C* (Modified 100% Matl Bal)

800 F.

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CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	FORCED....		SUBTOTAL EX.H-GAS	TOTAL
				H2O	H2S&NH3		
C		9.12	339.14	0.00	0.00	339.14	339.14
H	1.1%	1.94	31.76	3.27	0.03	35.07	57.64
N	0.7%	0.00	2.65	0	0.02	2.67	2.67
O	67.2%	3.69	13.23	26.16	0.00	39.38	39.38
S	41.1%	0.00	0.64	0.00	0.45	1.08	1.08
ASH		0.00	3.31	0.00	0.00	3.31	3.31
TOTAL		14.74	390.74	29.43	0.50	420.66	443.23

REPORT: (SUMMARY OF EXTRACTIONS)

BOOK#: 16103-102 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: 100 ppm Mo as Molyvan-L

REPORT:	Extraction	Normal'd	
Description:	Run No.-----	102C	102C*
CONDITIONS:	PSIG	2000	2000
Temperature, F	(F)	800.0	800.0
Space Time, min	Min	89.0	89.0
Hydrogen (Brooksset)	SCFH	10.0	10.0
Lineout Time, Hr	Hrs	0.0	0.0
Run Time, after lineout	Hrs	6.0	6.0

MATERIAL BALANCE:

Feed Rate (L/O+Run)	Gm/Hr	364.71	364.71
Slurry Product	Gm/Hr	253.48	335.35
Light Ends	Gm/Hr	0.00	0.00
C1-C3 Gases	Gm/Hr	9.65	9.65
C4+ Gases	Gm/Hr	3.82	3.82
Carbon Monoxide	Gm/Hr	0.95	0.95
Carbon Dioxide	Gm/Hr	4.79	4.79
Water	Gm/Hr	0.00	10.17
Recovery	(%)	74.8%	100.0%

YIELD: (Based on MAF Total Feed)

Hexane Solubles	Wt%	66.87	55.85
Asphaltenes	Wt%	24.88	32.92
Preasphaltenes	Wt%	4.23	5.59
Hexane Insoluble	Wt%	33.13	44.15
TOL Insoluble	Wt%	8.25	11.23
THF Insoluble	Wt%	4.02	5.63

YIELD: (Based on MAF Coal Only)

Asphaltenes	Wt%	82.01	108.50
Preasphaltenes	Wt%	13.93	18.43
THF Solubles	Wt%	86.74	81.43
Hexane Insoluble	Wt%	109.21	145.50
TOL Insolubles	Wt%	27.19	37.00
THF Insolubles	Wt%	13.26	18.57
Light Ends	Wt%	0.00	0.00
C1+C2+C3 Gases	Wt%	9.07	9.07
C4+C5+C6 Gases	Wt%	3.59	3.59
Carbon Monoxide	Wt%	0.89	0.89
Carbon Dioxide	Wt%	4.50	4.50

EXTRACTION RESULTS:	Page:	Normal'd	Normal'd
THF Insolubles	Gms	6.900	6.900
Tol Insolubles	Gms	12.750	12.750
Hex Ins in Tol Sol	Gms	34.440	34.440
Hex & Tol Insol.	Gms	47.190	47.190
HEX Solubles	Gms	52.810	52.810
Extraction Feed	Gms	100.000	100.000
Recovery	(%)	100.00%	100.00%

REPORT:

BOOK#: 16103-102 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: 100 ppm Mo as Molyvan-L

ELEMENTAL ANALYSIS: (Complete Extraction)

Unconverted Coal (A)

C	Frn	0.8122
H	Frn	0.0420
N	Frn	0.0136
O	Frn	0.0575
S	Frn	0.0047
Ash	Frn	0.0700
Total	Frn	1.0000

Preasphaltenes (B)

C	Frn	0.8526
H	Frn	0.0549
N	Frn	0.0133
O	Frn	0.0587
S	Frn	0.0019
Total	Frn	0.9814

Asphaltenes (C)

C	Frn	0.8745
H	Frn	0.0553
N	Frn	0.0152
O	Frn	0.0491
S	Frn	0.0018
Total	Frn	0.9959

Oils (Hex Sol) (D)

C	Frn	0.8978
H	Frn	0.0858
N	Frn	0.0049
O	Frn	0.0139
S	Frn	0.0009
Total	Frn	1.0033

MATERIAL BALANCE: DATE: 09-Jul 1992 Total 12 Pages
 BOOK#: 16103-102 June 93
 COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.
 CATALYST: 100 ppm Mo as Molyvan-L
 RUN NO: 102C 800 F. Page 1 of 12
 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	78.39	213.18	0.00	0.00	291.57	291.57
H	1.17	0.00	5.82	23.66	0.00	25.40	29.48	56.05
N	0.00	0.00	1.13	1.30	0.00	0.00	2.42	2.42
O	9.34	0.00	20.22	6.16	0.00	0.00	26.38	35.72
S	0.00	0.00	0.88	0.10	0.00	0.00	0.98	0.98
ASH	0.00	3.37	0.00	0.00	0.00	0.00	3.37	3.37
TOTAL	10.50	3.37	106.45	244.39	0.00	25.40	354.21	390.11
FEED:	10.50	3.37	106.45	244.39	0.00	25.40	354.21	390.11

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.41	1.31	7.54	3.16	0.00	0.00	12.4
H	22.46	0.00	0.00	2.11	0.66	0.00	0.00	25.2
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.54	3.48	0.00	0.00	0.00	0.00	4.0
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	22.46	0.95	4.79	9.65	3.82	0.00	0.00	41.7
	22.46	0.95	4.79	9.65	3.82	0.00	0.00	41.7

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	12.42	14.21	12.88	76.66	119.79	223.53	235.95	235.95
H	25.23	0.73	0.83	4.85	11.45	17.86	43.09	20.63
N	0.00	0.24	0.20	1.33	0.65	2.42	2.42	2.42
O	4.02	1.01	0.89	4.30	1.85	8.05	12.07	12.07
S	0.00	0.08	0.03	0.16	0.12	0.39	0.39	0.39
ASH	0.00	1.22	0.00	0.00	0.00	1.22	1.22	1.22
TOTAL	41.67	17.49	14.83	87.30	133.86	253.48	295.15	272.69
	41.67	17.49	14.83	87.30	133.86	253.48	295.2	

MATERIAL BALANCE: DATE: 09-Jul 1992 Total 12 Pages

BOOK#: 16103-102 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: 100 ppm Mo as Molyvan-L

RUN NO: 102C# 800 F.

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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	78.39	213.18	0.00	0.00	291.57	291.57
H	1.17	0.00	5.82	23.66	0.00	25.40	29.48	56.05
N	0.00	0.00	1.13	1.30	0.00	0.00	2.42	2.42
O	9.34	0.00	20.22	6.16	0.00	0.00	26.38	35.72
S	0.00	0.00	0.88	0.10	0.00	0.00	0.98	0.98
ASH	0.00	3.37	0.00	0.00	0.00	0.00	3.37	3.37
TOTAL	10.50	3.37	106.45	244.39	0.00	25.40	354.21	390.11
FEED:	10.50	3.37	106.45	244.39	0.00	25.40	354.21	390.11

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.41	1.31	7.54	3.16	0.00	0.00	12.4
H	22.46	0.00	0.00	2.11	0.66	0.00	1.13	26.4
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.54	3.48	0.00	0.00	0.00	9.04	13.1
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	22.46	0.95	4.79	9.65	3.82	0.00	10.17	51.8
	22.46	0.95	4.79	9.65	3.82	0.00	10.17	51.8

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	SLURRY OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	12.42	18.79	17.04	101.42	158.48	295.73	308.15	308.15
H	25.23	0.97	1.10	6.41	15.15	23.63	48.86	26.40
N	0.00	0.31	0.27	1.76	0.86	3.21	3.21	3.21
O	4.02	1.33	1.17	5.69	2.45	10.65	14.67	14.67
S	0.00	0.11	0.04	0.21	0.16	0.51	0.51	0.51
ASH	0.00	1.62	0.00	0.00	0.00	1.62	1.62	1.62
TOTAL	41.67	23.14	19.62	115.49	177.10	335.35	377.02	354.56
	41.67	23.14	19.62	115.49	177.10	335.35	377.0	

FORCED ELEMENTAL BALANCE:

DATE: 07/09/93

BOOK#: 16103-102 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110.

CATALYST: 100 ppm Mo as Molyvan-L

RUN NO: 102C

800 F.

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CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	FORCED.....		SUBTOTAL EX.H-GAS	TOTAL
				H2O	H2S&NH3		
C		12.42	235.95	0.00	0.00	235.95	235.95
H	-6.6%	2.77	20.63	2.96	0.04	23.62	46.09
N	0.0%	0.00	2.42	0	0.00	2.42	2.42
O	69.5%	4.02	12.07	23.65	0.00	35.72	35.72
S	60.4%	0.00	0.39	0.00	0.59	0.98	0.98
ASH		0.00	1.22	0.00	0.00	1.22	1.22
TOTAL		19.21	272.69	26.60	0.63	299.92	322.38

RUN NO: 102C*

800 F.

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CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	FORCED.....		SUBTOTAL EX.H-GAS	TOTAL
				H2O	H2S&NH3		
C		12.42	308.15	0.00	0.00	308.15	308.15
H	-1.5%	2.77	26.40	2.63	0.03	29.06	51.52
N	0.0%	0.00	3.21	0	0.00	3.21	3.21
O	59.6%	4.02	14.67	21.05	0.00	35.72	35.72
S	47.6%	0.00	0.51	0.00	0.47	0.98	0.98
ASH		0.00	1.62	0.00	0.00	1.62	1.62
TOTAL		19.21	354.56	23.68	0.50	378.73	401.19

REPORT: (SUMMARY OF EXTRACTIONS)

BOOK#: 16103-113 June 93

COAL: Swollen SO2-BT; SOLVENT: V-1074 + Resid, V-203

CATALYST: None

REPORT: Extraction Normal'd
Description: Run No.----- 113C

CONDITIONS: PSIG 2000
Temperature, F (F) 800.0
Space Time, min Min 78.7
Hydrogen (Brooksset) SCFH 10.0
Lineout Time, Hr Hrs 0.0
Run Time, after lineout Hrs 6.0

MATERIAL BALANCE:
Feed Rate (L/O+Run) Gm/Hr 412.38
Slurry Product Gm/Hr 394.97
Light Ends Gm/Hr 0.00
C1-C3 Gases Gm/Hr 8.41
C4+ Gases Gm/Hr 2.41
Carbon Monoxide Gm/Hr 1.12
Carbon Dioxide Gm/Hr 3.71
Water Gm/Hr 0.00
Recovery (%) 99.6%

YIELD: (Based on MAF Total Feed)

Hexane Solubles Wt% 58.15
Asphaltenes Wt% 32.68
Preasphaltenes Wt% 3.77
Hexane Insoluble Wt% 41.85
TOL Insoluble Wt% 9.18
THF Insoluble Wt% 5.41

YIELD: (Based on MAF Coal Only)

Asphaltenes Wt% 107.68
Preasphaltenes Wt% 12.42
THF Solubles Wt% 82.18
Hexane Insoluble Wt% 137.93
TOL Insolubles Wt% 30.24
THF Insolubles Wt% 17.82
Light Ends Wt% 0.00
C1+C2+C3 Gases Wt% 8.43
C4+C5+C6 Gases Wt% 2.41
Carbon Monoxide Wt% 1.12
Carbon Dioxide Wt% 3.72

EXTRACTION RESULTS: Page: Normal'd

THF Insolubles Gms 5.300
Tol Insolubles Gms 8.440
Hex Ins in Tol Sol Gms 27.220
Hex & Tol Insol. Gms 35.660
HEX Solubles Gms 64.340
Extraction Feed Gms 100.000
Recovery (%) 100.00%

REPORT:

BOOK#: 16103-113 June 93

COAL: Swollen SO2-BT; **SOLVENT:** V-1074 + Resid, V-203

CATALYST: None

ELEMENTAL ANALYSIS: (Complete Extraction)

Unconverted Coal (A)

C	Frn	0.7745
H	Frn	0.0399
N	Frn	0.0128
O	Frn	0.0620
S	Frn	0.0078
Ash	Frn	0.1030
Total	Frn	1.0000

Preasphaltenes (B)

C	Frn	0.8546
H	Frn	0.0573
N	Frn	0.0142
O	Frn	0.0604
S	Frn	0.0012
Total	Frn	0.9877

Asphaltenes (C)

C	Frn	0.8827
H	Frn	0.0602
N	Frn	0.0129
O	Frn	0.0343
S	Frn	0.0016
Total	Frn	0.9917

Oils (Hex Sol) (D)

C	Frn	0.8942
H	Frn	0.0877
N	Frn	0.0043
O	Frn	0.0106
S	Frn	0.0009
Total	Frn	0.9977

MATERIAL BALANCE:

DATE: 17-Jul 1992

Total 12 Pages

BOOK#: 16103-113 June 93

COAL: Swollen SO2-BT; SOLVENT: V-1074 + Resid, V-203

CATALYST: None

800 F.

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RUN NO: 113C

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	73.52	202.61	60.67	0.00	336.79	336.79
H	1.09	0.00	5.46	20.56	6.56	25.40	32.58	59.08
N	0.00	0.00	1.06	1.51	0.00	0.00	2.57	2.57
O	8.76	0.00	18.97	4.40	3.12	0.00	26.49	35.25
S	0.00	0.00	0.83	0.09	0.00	0.00	0.92	0.92
ASH	0.00	3.14	0.00	0.00	0.00	0.00	3.14	3.14
TOTAL	9.85	3.14	99.84	229.17	70.35	25.40	402.50	437.76
FEED:	9.85	3.14	99.84	229.19	70.35	25.40	402.53	437.78

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.48	1.01	6.57	1.99	0.00	0.00	10.1
H	22.41	0.00	0.00	1.84	0.42	0.00	0.00	24.7
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	0.64	2.70	0.00	0.00	0.00	0.00	3.3
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	22.41	1.12	3.71	8.41	2.41	0.00	0.00	38.1
	22.41	1.12	3.71	8.41	2.41	0.00	0.00	38.1

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	SLURRY OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	10.06	16.21	10.73	95.69	227.76	350.40	360.46	360.46
H	24.66	0.84	0.72	6.53	22.34	30.42	55.08	32.67
N	0.00	0.27	0.18	1.40	1.10	2.94	2.94	2.94
O	3.34	1.30	0.76	3.72	2.70	8.47	11.81	11.81
S	0.00	0.16	0.02	0.17	0.23	0.58	0.58	0.58
ASH	0.00	2.16	0.00	0.00	0.00	2.16	2.16	2.16
TOTAL	38.06	20.93	12.40	107.51	254.13	394.97	433.04	410.63
	38.06	20.93	12.40	107.51	254.13	394.97	433.0	

FORCED ELEMENTAL BALANCE:

DATE: 07/12/93

BOOK#: 16103-113 June 93

COAL: SO2-Black Thunder; SOLVENT: V-1074 Run 258, FSN-110

CATALYST: None

RUN NO: 113C

800 F.

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CALCULATED ELEMENTAL BALANCE: (Gms/Hr)

ITEM:	PROCESS CONVRS'N	COX+HC GASES	SUBTOTAL SL+LE+GS	FORCED.....		SUBTOTAL EX.H-GAS	TOTAL
				H2O	H2S&NH3		
C		10.06	360.46	0.00	0.00	360.46	360.46
H	1.9%	2.25	32.67	2.93	0.02	35.62	58.03
N	0.0%	0.00	2.94	0	0.00	2.94	2.94
O	68.0%	3.34	11.81	23.44	0.00	35.25	35.25
S	36.9%	0.00	0.58	0.00	0.34	0.92	0.92
ASH		0.00	2.16	0.00	0.00	2.16	2.16
TOTAL		15.65	410.63	26.37	0.36	437.35	459.76

REPORT: (SUMMARY OF EXTRACTIONS)

BOOK#: 16103-115 June 93

COAL: Swollen SO2-BT; SOLVENT: V-1074 + Resid (80/20).

CATALYST: Molyvan-L

REPORT:	Extraction	Normal'd	Normal'd
Description:	Run No.-----	115C	115C*
CONDITIONS:	PSIG	2000	2000
Temperature, F	(F)	800.0	800.0
Space Time, min	Min	71.4	71.4
Hydrogen (Brooksset)	SCFH	10.0	10.0
Lineout Time, Hr	Hrs	0.0	0.0
Run Time, after lineout	Hrs	39.7	39.7

MATERIAL BALANCE:

Feed Rate (L/O+Run)	Gm/Hr	454.91	454.91
Slurry Product	Gm/Hr	352.83	403.37
Light Ends	Gm/Hr	0.00	0.00
C1-C3 Gases	Gm/Hr	16.48	16.48
C4+ Gases	Gm/Hr	14.61	14.61
Carbon Monoxide	Gm/Hr	1.82	1.82
Carbon Dioxide	Gm/Hr	6.26	6.26
Water	Gm/Hr	0.00	12.50
Recovery	(%)	86.2%	100.0%

YIELD: (Based on MAF Total Feed)

Hexane Solubles	Wt%	47.28	39.59
Asphaltenes	Wt%	39.74	45.43
Preasphaltenes	Wt%	5.58	6.38
Hexane Insoluble	Wt%	52.72	60.41
TOL Insoluble	Wt%	12.98	14.98
THF Insoluble	Wt%	7.40	8.59

YIELD: (Based on MAF Coal Only)

Asphaltenes	Wt%	129.56	148.11
Preasphaltenes	Wt%	18.21	20.81
THF Solubles	Wt%	75.89	71.99
Hexane Insoluble	Wt%	171.87	196.94
TOL Insolubles	Wt%	42.31	48.83
THF Insolubles	Wt%	24.11	28.01
Light Ends	Wt%	0.00	0.00
C1+C2+C3 Gases	Wt%	13.92	13.92
C4+C5+C6 Gases	Wt%	12.34	12.34
Carbon Monoxide	Wt%	1.53	1.53
Carbon Dioxide	Wt%	5.29	5.29

EXTRACTION RESULTS:	Page:	Normal'd	Normal'd
THF Insolubles	Gms	9.150	9.150
Tol Insolubles	Gms	15.260	15.260
Hex Ins in Tol Sol	Gms	43.480	43.480
Hex & Tol Insol.	Gms	58.740	58.740
HEX Solubles	Gms	41.260	41.260
Extraction Feed	Gms	100.000	100.000
Recovery	(%)	100.00%	100.00%

REPORT:

BOOK#: 16103-115 June 93

COAL: Swollen SO2-BT; SOLVENT: V-1074 + Resid (80/20).

CATALYST: Molyvan-L

ELEMENTAL ANALYSIS: (Complete Extraction)

Unconverted Coal (A)

C	Frn	0.7898
H	Frn	0.0401
N	Frn	0.0123
O	Frn	0.0509
S	Frn	0.0077
Ash	Frn	0.0992
Total	Frn	1.0000

Preasphaltenes (B)

C	Frn	0.8668
H	Frn	0.0554
N	Frn	0.0130
O	Frn	0.0549
S	Frn	0.0019
Total	Frn	0.9920

Asphaltenes (C)

C	Frn	0.8860
H	Frn	0.0632
N	Frn	0.0116
O	Frn	0.0319
S	Frn	0.0017
Total	Frn	0.9944

Oils (Hex Sol) (D)

C	Frn	0.8922
H	Frn	0.0899
N	Frn	0.0041
O	Frn	0.0120
S	Frn	0.0052
Total	Frn	1.0034

MATERIAL BALANCE:

BOOK#: 16103-115 June 93

COAL: S02-Black Thunder; SOLVENT:

CATALYST: None

RUN NO: 115C

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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	87.20	236.57	35.65	0.00	359.42	359.42
H	1.30	0.00	6.48	24.01	5.94	25.40	36.42	63.12
N	0.00	0.00	1.26	1.78	0.00	0.00	3.04	3.04
O	10.38	0.00	22.50	5.14	11.88	0.00	39.52	49.90
S	0.00	0.00	0.98	0.11	0.00	0.00	1.09	1.09
ASH	0.00	3.74	0.00	0.00	0.00	0.00	3.74	3.74
TOTAL	11.68	3.74	118.41	267.60	53.47	25.40	443.22	480.31
FEED:	11.68	3.74	118.41	267.61	53.47	25.40	443.23	480.32

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.78	1.71	12.88	12.09	0.00	0.00	27.5
H	21.13	0.00	0.00	3.60	2.52	0.00	0.00	27.2
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	1.04	4.55	0.00	0.00	0.00	0.00	5.6
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	21.13	1.82	6.26	16.48	14.61	0.00	0.00	60.3
	21.13	1.82	6.26	16.48	14.61	0.00	0.00	60.3

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	SLURRY OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	27.45	25.50	18.84	136.69	129.44	310.47	337.92	337.92
H	27.25	1.29	1.20	9.75	13.04	25.29	52.54	31.41
N	0.00	0.40	0.28	1.79	0.59	3.06	3.06	3.06
O	5.59	1.64	1.19	4.92	1.74	9.50	15.09	15.09
S	0.00	0.25	0.04	0.26	0.75	1.31	1.31	1.31
ASH	0.00	3.20	0.00	0.00	0.00	3.20	3.20	3.20
TOTAL	60.29	32.28	21.56	153.41	145.58	352.83	413.12	392.00
	60.29	32.28	21.56	153.41	145.58	352.83	413.1	

MATERIAL BALANCE:

BOOK#: 16103-115 June 93

COAL: SO2-Black Thunder;

CATALYST: None

RUN NO: 115C

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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	87.20	236.57	35.65	0.00	359.42	359.42
H	1.30	0.00	6.48	24.01	5.94	25.40	36.42	63.12
N	0.00	0.00	1.26	1.78	0.00	0.00	3.04	3.04
O	10.38	0.00	22.50	5.14	11.88	0.00	39.52	49.90
S	0.00	0.00	0.98	0.11	0.00	0.00	1.09	1.09
ASH	0.00	3.74	0.00	0.00	0.00	0.00	3.74	3.74
TOTAL	11.68	3.74	118.41	267.60	53.47	25.40	443.22	480.31
FEED:	11.68	3.74	118.41	267.61	53.47	25.40	443.23	480.32

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

ITEM	H2	CO	CO2	C1-C3	C4+	LT ENDS	H2O	TOTAL
C	0.00	0.78	1.71	12.88	12.09	0.00	0.00	27.5
H	21.13	0.00	0.00	3.60	2.52	0.00	1.39	28.6
N	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.0
O	0.00	1.04	4.55	0.00	0.00	0.00	11.11	16.7
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	21.13	1.82	6.26	16.48	14.61	0.00	12.50	72.8
	21.13	1.82	6.26	16.48	14.61	0.00	12.50	72.8

CALCULATED ELEMENTAL CONTENT OF PRODUCT SLURRY (Gms/Hr)

ITEM	P/GAS +L.E.	Unc.Coal	PRODUCT P/ASPH	SLURRY ASPH.	SLURRY OILS	SLURRY TOTAL	SUB-TOT -H2O	SUB-TOT -H2&H2O
C	27.45	29.15	21.54	156.27	147.99	354.94	382.39	382.39
H	27.25	1.48	1.38	11.15	14.91	28.91	56.16	35.04
N	0.00	0.45	0.32	2.05	0.68	3.50	3.50	3.50
O	5.59	1.88	1.36	5.63	1.99	10.86	16.45	16.45
S	0.00	0.28	0.05	0.30	0.86	1.49	1.49	1.49
ASH	0.00	3.66	0.00	0.00	0.00	3.66	3.66	3.66
TOTAL	60.29	36.91	24.65	175.38	166.43	403.37	463.66	442.54
	60.29	36.91	24.65	175.38	166.43	403.37	463.7	

FORCED ELEMENTAL BALANCE:

BOOK#: 16103-115 June 93
 COAL: Swollen SO2-BT; SOLVENT: V-1074 + Resid (80/20).
 CATALYST: Molyvan-L

RUN NO: 115C

800 F.

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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		27.45	337.92	0.00	0.00	337.92	337.92
H	-1.7%	6.12	31.41	4.35	-0.01	35.75	56.88
N	0.0%	0.00	3.06	0	0.00	3.06	3.06
O	76.0%	5.59	15.09	34.81	0.00	49.90	49.90
S	-19.9%	0.00	1.31	0.00	-0.22	1.09	1.09
ASH		0.00	3.20	0.00	0.00	3.20	3.20
TOTAL		39.17	392.00	39.16	-0.23	430.93	452.06

RUN NO: 115C*

800 F.

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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		27.45	382.39	0.00	0.00	382.39	382.39
H	1.2%	6.12	35.04	4.18	-0.03	39.19	60.32
N	0.0%	0.00	3.50	0	0.00	3.50	3.50
O	72.5%	5.59	16.45	33.45	0.00	49.90	49.90
S	-37.1%	0.00	1.49	0.00	-0.40	1.09	1.09
ASH		0.00	3.66	0.00	0.00	3.66	3.66
TOTAL		39.17	442.54	37.63	-0.43	479.74	500.87

**DATE
FILMED**

10 / 25 / 93

END