

DOE/PC/91040-64

**ADVANCED DIRECT LIQUEFACTION CONCEPTS  
for PETC GENERIC UNITS  
Phase II**

**Quarterly Technical Progress Report  
for Period January through March 1996**

by

**University of Kentucky  
Center for Applied Energy Research**

**CONSOL Inc.**

**Hydrocarbon Technologies, Inc.**

**LDP Associates**

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**MASTER**

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## SUMMARY

### TASK 2.1 LABORATORY SUPPORT (UK/CAER)

#### Task 2.1.1 Development of a Catalyst Screening Test (CST)

The CST activity of Run 263J catalyst that was isolated from the Run 263J V-1082 ashy resid by either pressure filtration or by Soxhlet extraction was compared with the corresponding conversion of coal in the Mo-containing Run 263J solvent. The material isolated by pressure filtration of a THF slurry of V-1082 was found to contain 740 mg Mo/kg, which agrees with the calculated Mo concentration in original V-1082 ashy resid. Two samples were prepared by Soxhlet extraction of the V-1082 using THF and Panasol as the extracting solvents. The solids concentrates were blended into the CST solvent at a concentration of 300 ppm Mo/dry coal. The resid conversions were unexpectedly low, i.e., 23-24%, relative to the conversions observed in the Run 262E solvent. Although the conversions were greater than the 19% resid conversion observed in the absence of any catalyst, they were far short of the anticipated 43% resid conversion observed in Run 263J.

In order to determine whether the particular distillate and deashed resid material used in the CST experiments were retarding or otherwise influencing the catalytic activity of the solids concentrate, a sample of deashed, full-range recycle solvent from Run 258, period K, was deashed in the laboratory and substituted into the test reaction mixture. Resid conversion in the deashed Run 258K solvent was 26% versus 23% in the CST solvent. Liquefaction in these two solvents in the absence of any added solids gave essentially the same resid conversion, i.e., 18-19%. These results suggest that using the Kerr McGee deashed resid instead of the full-range resid was not overly influencing the activity measurement. Although the presence of the catalyst in these two solvents appears to make little difference in the degree of reaction, there is a very distinct difference in resid conversion, THF coal conversion, and H<sub>2</sub> yield when the catalyst is present. The activity of the samples of Mo-concentrate in distillate and deashed resid from Run 262E were also determined. The 524 ° C resid conversions obtained upon adding the Mo-rich solids concentrate to this solvent mixture were

somewhat higher in the Run 258 derived solvent, i.e., 26-30%. The conversions are still well below the value observed when the ashy resid from Run 262E was added directly into the solvent without separating out the solids concentrate.

The sensitivity of the coal reaction to Mo concentration was determined by varying the amount of Run 262E ashy resid that was added to the reaction system. In a reaction mixture comprising Run 262E distillate and ashy resid, Run 258A deashed resid was successively substituted for the ashy resid thereby reducing the Mo concentration. These runs were made over a range of Mo concentrations from 63 to 380 mg/kg mf coal. Resid conversion dropped from 43% to 30% in going from 380 to 190 ppm with a further decrease to 19% upon going to 125 ppm Mo. Further decrease in Mo concentration to 63 ppm did not affect conversion since 19% is the same level as observed in the absence of added catalyst.

Several methods for activating molybdenum precursors to make catalysts equivalent in activity to the accumulated Mo in the Run 262E ashy resid have been examined. In one run case, Mo contained in Molyvan L dispersed in solids-free Run 258A deashed resid was treated at 440 °C for 30 min in the absence of coal after which coal was added and the run continued for an additional 30 min. The activity of Molyvan L added to the coal without any pretreatment was slightly lower, than the pretreated case. A bigger difference was observed by increasing the Mo concentration from 300 ppm to 1000 ppm where resid conversion increased from 26% to 30%.

### **Task 2.1.2 Activation of Impregnated Catalysts**

Studies on activation of metal impregnated catalysts continued during this reporting period. The effect of pretreatment temperature on activating Mo, Mo + Ni, and Mo + Fe metal impregnated Black Thunder Wyodak coals was studied. The Mo-Ni and Mo-Fe metal-impregnated coals were pretreated over a range of temperatures from 275 to 375 °C and then exposed to liquefaction conditions in the CST solvent for 30 min at 440 °C in 3% H<sub>2</sub>S/H<sub>2</sub> at 1350 psig (cold). Resid conversion for the nickel-moly catalyst was greater than for the iron-moly combination over the entire

pretreatment range. Likewise, THF conversion, hydrogen consumption and hydrocarbon gas yield were also greater. Overall, the pretreatment step had a positive effect on liquefaction yields. Pretreatment at 300°C appears to improve resid conversion by about 5%.

## **TASK 2.2 LABORATORY SUPPORT (CONSOL)**

The plan for Run ALC-1 was finalized. Agglomeration conditions were found in the laboratory that provide acceptable ash rejection, organic recovery, and product size with the Run ALC-1 feed coal at an acceptable dosage of the selected agglomerating oil. The 50 gal agglomerator unit was set up and 1300 lb of cleaned coal was produced, dried, and shipped to HTI for Run ALC-1. All technical and scheduling targets were met. Equipment specifications, operating conditions, energy usage, feed consumption and product yields were measured and recorded and product characteristics will be determined to assist in the engineering and economic assessment. The request for samples from Run ALC-1 for characterization was finalized.

Ongoing and near-future activities include agglomeration and product analysis, data work-up, additional evaluation of the liquefaction reactivity of the agglomerate products, characterization of the Sandia hydrotreating samples and HTI dewaxing samples, final review of HTI's run plan, and on-site monitoring of Run ALC-1 operations.

## **TASK 3 CONTINUOUS OPERATIONS/PARAMETRIC STUDIES**

### **TASK 3.3 (HYDROCARBON TECHNOLOGIES, INC.)**

Equipment, materials and a run plan have been prepared for the first run scheduled for April. A preliminary dewaxing run was made following CONSOL's procedure using pressure filter liquid collected in PB-1 and PB-3, which was fractionated to produce a 343-524 °C vacuum still overhead (VSOH). Upon dewaxing, the PB-1 VSOH was found to contain 5.15 wt % wax, which was not much different from that of CMSL-09 VSOH. A dewaxing facility with a capacity of 80 lbs/batch

was designed and assembled. About 400 lbs of VSOH from previous bench runs was collected and will be dewaxed-hydrotreated during April in the new facility. About 116 kg of Fe-containing catalyst was prepared and tested to ensure uniformity. HTI has provided various materials to the other participants in the program. Coal, Molyvan A, and HTI's iron catalyst has been provided to CONSOL and the CAER. A 40 lb VSOH sample was shipped to Sandia for a hydrogenation study along with 100 g of Criterion 411 trilobe catalyst.

A detailed operating plan was prepared based upon CONSOL's run plan and a tentative schedule developed. A computer program/database for HTI's unit 227 is being updated to accommodate the use of oil-agglomerated feed and other unit operations, e.g., filtration/vacuum distillation and solvent dewaxing/hydrotreating. Unit start-up was tentatively scheduled to be around April 9-12.

#### **TASK 4 CONCEPTUAL PROCESS DESIGN**

##### **TASK 4.4 PRELIMINARY TECHNICAL ASSESSMENT (LDP ASSOCIATES)**

Numerous discussions were held with CONSOL regarding ALC-1. The decision to use the pressure filter was based upon the desire to control resid and solids recycle and the particular target recycle composition was based upon compositions used in recent runs at Wilsonville, HTI, and Exxon. An evaluation was conducted to determine whether the 5% wax concentration observed in the distillate solvent from CMSL-9, Condition #6 was consistent with the 20% wax concentration observed in Wilsonville Run 263J. It was determined that differences in product withdrawals, sampling and reactor severity could account for the differences in wax content. An updated base case was prepared to account for the allowable lower gasification temperature (2500 °F) and a more accurate C<sub>1</sub>-C<sub>3</sub> gas yield distribution in the upgrading units. As a result the required gasoline product selling price dropped by \$1.23/bbl from the original base case estimate. In planning ALC-1, projections were made for each of the proposed conditions and presented at the planning meeting held 19 January.

**SECTION ONE**

**Center for Applied Energy Research  
University of Kentucky**

## WORK PERFORMED

### TASK 2.1 LABORATORY SUPPORT (UK/CAER)

#### Task 2.1.1 Development of a Catalyst Screening Test (CST)

In the previous quarterly report, the composition of the catalyst screening test (CST) solvent and conditions for the test were presented. The solvent comprises distillate and deashed resid from Wilsonville Run 258, specifically distillate V-1074 from Run 258, period B, and deashed resid V-130 from Run 258, period A. Although these materials had been generated when the plant was running with a 565 °C cut point on the vacuum tower, in our test a cut point of 524 °C will be used to conform with the cut point used in the HTI pilot plant. In the CST, a solvent mix of 33% V-1074 distillate and 67% deashed resid was selected, which gives a composition having the boiling point concentrations shown in Table 1. The composition of the total mix, including dry coal, is shown in Table 2.

As reported earlier, the reaction time for the test was selected based upon the results of liquefaction of Wyodak Black Thunder coal in Wilsonville Run 262E recycle solvent. The results from reaction times of 30, 60 and 90 minute runs (Table 3) combined with results observed in Phase I at 22 minutes, led us to select 30 minutes as an appropriate reaction time for the test. The target 524 °C+ resid conversion in the test mixture, based upon the experiments in Run 262E recycle solvent, was 43% (92% on maf coal).

To check the conversion in the CST mixture, samples of solids concentrate isolated from Run 262E V-1082 ashy resid were blended with the components of the CST solvent mixture and tested with Wyodak BT coal under CST conditions. Three different samples of solids concentrate were obtained. Two were prepared by extracting the ashy resid in a Soxhlet extraction apparatus. In one case THF was used as solvent, while in the other Panasol, a mixture of alkyl naphthalenes, was used. The third sample was prepared by diluting the ashy resid with THF and filtering at ambient temperature. These solids concentrates were blended into the Run 258 solvent components at a concentration of 300 ppm Mo/dry coal in the ratios shown in Table 4. The resid conversions were unexpectedly low, i.e., 23-

24%, relative to the conversions observed in the Run 262E system described above.

To determine whether the particular samples of Run 258B V-1074 distillate and Run 258A V-1082 deashed resid used in these experiments were retarding the catalytic activity of the solids concentrate or otherwise influencing the result, a sample of deashed, full-range recycle solvent from Run 258, period K, was deashed in the laboratory and substituted into the test reaction mixture, as shown in Table 4. Comparing the two cases in which filtered solids was the source of added Mo-containing solids concentrate, shown in columns 3 and 6, resid conversions differed slightly, i.e., 23 and 26%. Liquefaction in these two solvents in the absence of any added solids, columns 2 and 5, gave essentially the same resid conversion. A run made in the filtered full-range recycle solvent from Run 258, period K, gave a resid conversion of 18% and a similar run made in the Run 258B V-1074 distillate and Run 258A V-1082 deashed resid gave 19%. In these two runs, resid conversions, THF coal conversion, and H<sub>2</sub> yields were very similar and distinctly different from runs made in which Mo-containing solids concentrate was added to the respective solvents.

Another comparison was made using V-1074 distillate and V-130 deashed resid produced in Run 262E in combination with the Mo-rich solids concentrate described above. The 524 °C- distillate fraction in each of these solvent fractions is described in Table 5 and the concentrations of the individual components in the feed to these runs is shown in Table 6. The 524 °C resid conversion numbers were somewhat higher in the Run 262 derived solvent, being 26-30%. The conversions are still well below the value observed when the V-1082 ashy resid from Run 262E was added directly into the solvent without separating out the solids concentrate.

The sensitivity of the coal reaction to the amount of accumulated Mo concentration in the reaction mixture was tested by varying the amount of Run 262E ashy resid that was added. In a reaction mixture comprising Run 262E V-1074 distillate and Run 262E V-1082 ashy resid, which had a Mo concentration of 380 mg/kg mf coal, as described above (see Table 3), Run 258A V-130 deashed resid was successively substituted for the ashy resid thereby reducing the Mo concentration. These runs were made over a range of Mo concentrations from 63 to 380 mg/kg mf coal, as shown in Table 7. Note that the run with 380 mg/kg mf coal was made at a cut point of 1050 °F while the

others were made at a cut point of 975 °F. Resid conversion dropped from 43% to 30% in going from 380 to 190 ppm. A further decrease to 125 ppm resulted in a further decrease in resid conversion to 19%, which is close to the resid conversion observed in the absence of Mo described in Table 4, runs T6-024-1 and T6-032-1. The run made at a Mo concentration of 63 ppm gave the same result as the run at 125 ppm.

#### **Activation of Molybdenum Precursors**

Several methods for activating molybdenum precursors to make catalysts equivalent in activity to the accumulated Mo in the Run 262E ashy resid has been examined. In one run (T6-082-2/3), the activation of Mo contained in Molyvan L was examined by pretreating Molyvan L dispersed in solids-free Run 258A V-130 deashed resid at 440 °C in the absence of coal (see Table 8). After the pretreat, coal was added and the run continued for an additional 30 min. The activity of Molyvan L added to the coal without any pretreatment (T6-081-1) was slightly lower, though not statistically different. A bigger difference was observed by increasing the Mo concentration. At 300 ppm the resid conversion was 26% while at 1000 ppm the resid conversion was 30%.

In the experiments that are run in the microautoclaves, the various ingredients are added separately and then heated together in the sand bath. In these runs, the temperature goes rapidly to reaction temperature over a 1-3 min interval. Whether the various ingredients become thoroughly mixed in this short interval isn't known. To test whether preliminary mixing of the Molyvan L into the resid fraction will facilitate the activation of the precursor and thereby give an active catalyst earlier in the reaction period, a run was made in which the Molyvan L was thoroughly mixed into the resid component. In this run, Run T6-086-1, the mixing was done at a relatively low temperature relative to the reaction temperature. The resulting mix was then heated to reaction temperature in the usual manner. Resid conversion in this run was 22%, which is lower than the other runs made with Molyvan L, indicating that prior mixing into the resid did not have a beneficial effect.

#### **Task 2.1.2 Activation of Impregnated Metal Catalysts**

Previously 60 minute liquefaction results on coals that had been impregnated with ammonium molybdate, Ni sulfate and ferrous sulfate were reported (see DOE/PC/91040-60, Table 6). Corrected

524 °C- distillate and 524 °C+ resid yields are presented in Table 9. The corrections do not affect the resid conversion results as previously reported.

During this reporting period, these same impregnated coals, shown in Table 10, were tested in 30 minute runs at 440 °C preceded by 30 minute pretreatment periods. Pretreatments were performed experimentally by agitating the batch microautoclave at the top of the sand bath for 30 minutes, observing the pretreatment temperature on the data screen. In general, this method allows pretreatment temperature control only to  $\pm 10$  °C (compared with  $\pm 1-2$  °C during hydroliquefaction), but obviates the need to use different sand baths operating at two different temperatures, with a cool-down and heat-up cycle between "pretreatment" and "hydroliquefaction".

The entire coal samples were impregnated (vs the vector approach). Coal feedstocks included CB-52, a Ni/Mo impregnated coal, and CB-53, a Fe/Mo impregnated coal. A second Fe/Mo impregnated coal was prepared, CB-57, to complete the pretreatment experiments. The Fe and Mo loadings matched the previous preparation, however, two of the runs contained 35% moisture, which was higher than the original moisture content of the coal due to incomplete drying of the sample. Subsequently, this sample was dried further.

Liquefaction results on the nickel-moly catalyst combination (see Table 11) has been found to be a more active catalyst than the iron-moly combination (see Table 12), as shown in THF conversion, resid conversion, hydrogen consumption and hydrocarbon gas yield. Overall, the pretreatment step had a positive effect on liquefaction yields. Pretreatment at 300°C for 30 minutes appears to improve resid conversion the most (of the low temperature pretreatments) for both coals, and THF conversion for the Ni/Mo coal [see Figures 1 and 2]. The effect found at this temperature is good, adding about 5% to resid conversions for either coal, and about the same amount to THF conversion in the case of Ni/Mo impregnated coal. However, only 1% improvement in THF conversion was seen in the case of the Fe/Mo impregnated coal. Data without pretreatment [B6-26-1 and B6-102-2] are not shown in the figures. Note that improvements from pretreatment at other temperatures are more subtle, with little impact seen at 340°C from the additional 30 minutes pretreatment time.

| Distillation Fraction | Wilsonville Run 258B<br>V-1074 Distillate | Wilsonville Run<br>258A V-130 Deashed<br>Resid | Mixture 33 wt%<br>Wilsonville Run 258B<br>V-1074 Distillate<br>plus<br>67 wt% Wilsonville<br>Run 258A V-130<br>Deashed Resid<br>(CST Solvent) |
|-----------------------|---|--|---|
| IBP-524 °C            | 3.0                                       | 8.3  | 40.9  |
| 524 -565 °C           | -   | 16.0   | -   |
| 565 °C+ Resid         | -   | 75.7   | -   |
| 524 °C+ Resid         | 97.0                                      | -  | 59.1  |

|                                    | Dry coal basis,<br>wt % |      |
|------------------------------------|-------------------------|------|
|                                    | 524                     | 565  |
| Cut point, °C                      | 524                     | 565  |
| Dry coal                           | 35                      | 35   |
| Distillate, wt% at cut point       | 26.6                    | 21.5 |
| Deashed Resid, wt% at cut<br>point | 38.4                    | 43.5 |

Table 3. Liquefaction of Wyodak Black Thunder Coal in Wilsonville

Run 262 Recycle Solvent

440 °C, 1350 psig, 2 vol% H<sub>2</sub>S/H<sub>2</sub>

| Reaction time, min               | 30                               | 60                   | 90                   |
|----------------------------------|----------------------------------|----------------------|----------------------|
| Feed Composition                 |                                  |                      |                      |
| Coal, mf, wt%                    | 30.0                             |                      |                      |
| 565 °C- Heavy Distillate, wt%    | 30.5                             |                      |                      |
| 565 °C+ Ashy Resid, wt%          | 39.5                             |                      |                      |
| Added Catalyst, wt%              | none                             |                      |                      |
| Mo Concentration, mg/kg mf coal  | 380                              |                      |                      |
| Reaction Results                 |                                  |                      |                      |
| THF Coal Conv                    | 106                              | 106                  | 107                  |
| Gas Yield                        | 22                               | 32                   | 32                   |
| 565 °C+ Resid Conv               | 43                               | 47                   | 52                   |
| 565 °C+ Resid Conv (on maf coal) | 92                               | 98                   | 110                  |
| H <sub>2</sub> consumption, wt%  | 4.6                              | 5.4                  | 5.9                  |
| Run No.                          | T5-339-1<br>T5-340-1<br>T5-341-1 | T5-310-1<br>T5-311-1 | T5-342-2<br>T5-346-1 |

Table 4. Effect of Added Wilsonville Run 262E Mo-Containing Solids Concentrate  
on Liquefaction of Wyodak BT Coal in Run 258 Derived Solvent

440 °C, 30 min, 1350 psig, 2 vol% H<sub>2</sub>S/H<sub>2</sub>

|                                       | Source of Run 262E extract |                      |                        |          |          |
|---------------------------------------|----------------------------|----------------------|------------------------|----------|----------|
|                                       | none                       | filtered             | Soxhlet<br>with<br>THF | none     | filtered |
| Feed Composition                      |                            |                      |                        |          |          |
| Run 262E extract, wt%                 | none                       | 12.4                 | 12.6                   | none     | 12.4     |
| Mo Concentration, mg/kg mf coal       | none                       | 295                  | 300                    | none     | 300      |
| Coal, mf, wt%                         | 35                         | 31                   | 31                     | 35       | 30       |
| Run 258 565 °C- Distillate, wt%       | 21.5                       | 18.8                 | 22.4                   | -        | -        |
| Run 258 565 °C+ Deashed Resid, wt%    | 43.6                       | 38.1                 | 35.3                   | -        | -        |
| Run 258 Filtered Recycle Solvent, wt% | -                          | -                    | -                      | 65       | 57.6     |
| Reaction Results                      |                            |                      |                        |          |          |
| THF Coal Conv                         | 68                         | 99                   | 101                    | 77       | 111      |
| Gas Yield                             | 21                         | 21                   | 19                     | 20       | 20       |
| 524 °C+ Resid Conv                    | 19                         | 23                   | 24                     | 18       | 26       |
| 524 °C+ Resid Conv (on maf coal)      | 41                         | 57                   | 59                     | 35       | 58       |
| H <sub>2</sub> consumption, wt%       | 1.7                        | 4.2                  | 3.9                    | 1.9      | 4.3      |
| Run No.                               | T6-024-1<br>T6-032-1       | T6-025-1<br>T6-026-1 | T6-039-1<br>T6-045-3   | T6-045-2 | T6-039-2 |

| Table 5. Distillation Cuts of Wilsonville Run 262E Solvent Components   |   |  |  |
|---|---|--|--|
|   | Distillation Fraction                                     |  |  |
|   | Wilsonville Run<br>262E V-1074<br>Distillate <sup>a</sup> | Wilsonville Run<br>262E V-130<br>Deashed Resid | Wilsonville Run<br>262E V-1082<br>Ashy Resid |
| IBP-524 °C  | 100   | 5.8  | 0  |
| 524 -565 °C   | -   | 10.2   | 2.5 <sup>a</sup>                             |
| 565 °C+ Resid   | -   | 84.0 <sup>b</sup>                              | 61.3   |
| 524 °C+ Resid   | 0   | 94.2 <sup>b</sup>                              | 58.8   |
| THF IOM   | -   | -  | 22.6   |
| SO <sub>3</sub> -free ash   | -   | -  | 16.1   |
|   |   | T6-065-1                                       | T6-057-1                                     |
| <p>a. Based on data supplied by C. Cantrell, letter to G. A. Robbins, Feb. 21, 1992.</p> <p>b. Assumes no THF insolubles in sample.</p> |   |  |  |

| Table 6. Effect of Added Wilsonville Run 262E Mo-Containing Solids Concentrate<br>on Liquefaction of Wyodak BT Coal in Run 262 Derived Solvent<br>440 °C, 30 min, 1350 psig, 2 vol% H <sub>2</sub> S/H <sub>2</sub> |                            |                  |          |                    |
|---|----------------------------|------------------|----------|--------------------|
|   | Source of Run 262E extract |                  |          |                    |
|   | none                       | THF<br>Soxhlet   | filtered | Panasol<br>Soxhlet |
| Feed Composition <sup>a</sup>   |                            |                  |          |                    |
| Run 262E extract, wt% <sup>b</sup>  | none                       | 12.4             | 12.2     | 11.8               |
| Mo Concentration, mg/kg mf coal   | none                       | 300              | 300      | 300                |
| Coal, mf, wt%   | 35                         | 31               | 31       | 30                 |
| Run 262 524 °C- Distillate, wt%   | 23.5                       | 21.0             | 32.1     | 31.0               |
| Run 262 524 °C+ Deashed Resid, wt%  | 40.0                       | 34.4             | 24.2     | 26.8               |
| THF IOM, wt%  | 1.5 <sup>c</sup>           | 1.2 <sup>c</sup> | 0.9      | 0.9                |
| Reaction Results  |                            |                  |          |                    |
| THF Coal Conv   | 60                         | 107              | 105      | 106                |
| Gas Yield   | 24                         | 23               | 21       | 21                 |
| 524 °C+ Resid Conv  | 15                         | 30               | 30       | 26                 |
| 524 °C+ Resid Conv (on maf coal)  | 34                         | 73               | 70       | 58                 |
| H <sub>2</sub> consumption, wt%   | 1.5                        | 4.5              | 4.2      | 4.3                |
| Run No.   | T6-058-1                   | T6-060-1         | T6-060-2 | T6-074-1           |
| a. Based upon lab distillation data   |                            |                  |          |                    |
| b. Includes THF IOM, mineral matter, and accumulated catalyst   |                            |                  |          |                    |
| c. Contained in Run 262 Deashed Resid   |                            |                  |          |                    |

Table 7. Effect of accumulated Mo concentration on coal reaction

440 °C, 30 min, 1350 psig, 2 vol% H<sub>2</sub>S/H<sub>2</sub>

| Feed Composition <sup>a</sup>                      |                                  |          |          |          |
|--|----------------------------------|----------|----------|----------|
| Mo Concentration, mg/kg mf coal                    | 380                              | 190      | 125      | 63       |
| Coal, mf, wt%                                      | 30                               | 30.4     | 30.3     | 30.3     |
| Run 262 Plant Distillate V-1074, wt%               | 30.5                             | 30.4     | 30.3     | 30.4     |
| Run 258 Plant Deashed Resid V-130, wt%             | none                             | 19.6     | 26.3     | 32.7     |
| Run 262 Plant Ashy Resid V-1082, wt% <sup>b</sup>  | 39.5                             | 19.6     | 13.2     | 6.6      |
| 524 °C Cut Point Distribution                      |                                  |          |          |          |
| 524 °C- Distillate, wt%                            | 30.5 <sup>c</sup>                | 32.0     | 32.5     | 33.1     |
| 524 °C+ Resid, wt%                                 | 24.2 <sup>c</sup>                | 30.0     | 32.1     | 34.0     |
| THF IOM, mineral matter, accumulated catalyst, wt% | 15.3                             | 7.6      | 5.1      | 2.6      |
| Reaction Results                                   |                                  |          |          |          |
| THF Coal Conv                                      | 106                              | 107      | 91       | 88       |
| Gas Yield  | 22                               | 23       | 22       | 23       |
| 524 °C+ Resid Conv                                 | 43 <sup>c</sup>                  | 30       | 19       | 19       |
| 524 °C+ Resid Conv (on maf coal)                   | 92 <sup>c</sup>                  | 73       | 42       | 43       |
| H <sub>2</sub> consumption, wt%                    | 4.6                              | 4.5      | 3.2      | 4.3      |
| Run No.  | T5-339-1<br>T5-340-1<br>T5-341-1 | T6-067-1 | T6-071-1 | T6-058-2 |

a. Based upon lab distillation data

b. Includes THF IOM, mineral matter, and accumulated catalyst

c. Based upon 565 °C cut point.

Table 8. Effect of Pretreatment of Molyvan L on Coal Reactivity

440 °C, 1350 psig, 2 vol% H<sub>2</sub>S/H<sub>2</sub>

| Feed Composition <sup>a</sup>                      | Run Numbers                      |               |                                       |                                       |                                   |                              |
|--|----------------------------------|---------------|---------------------------------------|---------------------------------------|-----------------------------------|------------------------------|
|  |                                  | MVL<br>30/440 | MVL<br>Pret resid<br>30/30<br>440/440 | MVL<br>Pret resid<br>30/30<br>440/440 | MVL<br>Pretreat<br>only<br>30/440 | MVL<br>Dispersed<br>in resid |
| Mo Concentration, mg/kg mf coal                    | 380                              | 300           | 300                                   | 1000                                  | 300                               | 300                          |
| Coal, mf, wt%                                      | 30                               | 34.9          | 35.2                                  | 35.0                                  | None                              | 35.0                         |
| Run 258 Plant Distillate V-1074, wt%               | 30.5 <sup>b</sup>                | 21.5          | 21.0                                  | 21.2                                  | 32.8                              | 21.2                         |
| Run 258 Plant Deashed Resid V-130, wt%             | none                             | 42.7          | 43.0                                  | 42.8                                  | 66.0                              | 43.0                         |
| Run 262 Plant Ashy Resid V-1082, wt% <sup>c</sup>  | 39.5                             | -             | -                                     | -                                     | -                                 | -                            |
| Catalyst   | -                                | 0.9           | 0.8                                   | 1.0                                   | 1.2                               | 0.8                          |
| 524 °C Cut Point Distribution                      |                                  |               |                                       |                                       |                                   |                              |
| 524 °C- Distillate, wt%                            | 30.5                             | 25.9          | 25.4                                  | 25.7                                  | 39.4                              | 25.0                         |
| 524 °C+ Resid, wt%                                 | 24.2                             | 39.1          | 39.4                                  | 39.2                                  | 60.4                              | 39.7                         |
| THF IOM, mineral matter, accumulated catalyst, wt% | 15.3                             | 0.1           | 0.1                                   | 0.1                                   | -                                 | 0.1                          |
| Reaction Results                                   |                                  |               |                                       |                                       |                                   |                              |
| THF Coal Conv                                      | 106                              | 90            | 88                                    | 89                                    | -                                 | 89                           |
| Gas Yield  | 22                               | 19            | 24                                    | 24                                    | 3.1                               | 20                           |
| 524 °C+ Resid Conv                                 | 43                               | 24            | 26                                    | 30                                    | 6.7                               | 22                           |
| 524 °C+ Resid Conv (on maf coal)                   | 92                               | 52            | 56                                    | 65                                    | -                                 | 48                           |
| H <sub>2</sub> consumption, wt%                    | 4.6                              | 3.7           | 4.4                                   | 4.9                                   | 0.9                               | 4.1                          |
|  | T5-339-1<br>T5-340-1<br>T5-341-1 | T6-081-1      | T6-082-<br>2/3                        | T6-088-<br>1/2                        | T6-085-1                          | T6-086-1                     |

a. Based upon lab distillation data

b. Used Run 262E plant distillate V-1074.

c. Includes THF IOM, mineral matter, and accumulated catalyst

| Table 9. Results of 60 minute liquefaction experiments.   |          |              |                   |          |         |          |
|---|----------|--------------|-------------------|----------|---------|----------|
|   | None     | Molyvan<br>L | Impregnated Salts |          |         |          |
|   |          |              | CB-51             |          | CB-52   | CB-53    |
| Metals added  | -        | Mo           | Mo                | Mo       | Ni/Mo   | Fe/Mo    |
| Metal Conc, mg/kg dry coal  | -        | 500          | 552               | 552      | 101/538 | 6730/478 |
| Coal moisture, wt %   | 22.9     | 22.9         | 30.1              | 30.1     | 21.1    | 22.6     |
| Pretreat time at 375 °C, mins   | 0        | 0            | 0                 | 30       | 0       | 0        |
| Products, wt% maf Coal  |          |              |                   |          |         |          |
| HC Gases  | 21.5     | 21.0         | 19.7              | 20.6     | 17.9    | 17.2     |
| CO+CO <sub>2</sub>  | 12.2     | 11.9         | 11.0              | 11.3     | 11.6    | 11.2     |
| 975 °F-   | 15.5     | 56.7         | 64.8              | 59.3     | 59.6    | 54.2     |
| 975 °F+   | 50.8     | 10.4         | 4.5               | 8.8      | 10.9    | 17.4     |
| Total   | 100.0    | 100.0        | 100.0             | 100.0    | 100.0   | 100.0    |
| THF Conv  | 66.3     | 95.1         | 90.6              | 90.6     | 92.3    | 92.5     |
| Resid Conv, wt% maf resid   | 21.8     | 40.4         | 40.8              | 38.9     | 40.7    | 36.8     |
| Material Bal index  | 97.3     | 97.1         | 97.1              | 102.3    | 89.0    | 94.2     |
| H <sub>2</sub> consumed, mg/g maf Coal  | 37.5     | 64.7         | 70.1              | 89.4     | 70.3    | 65.6     |
| Run No.   | B5-349-1 | B5-347-1     | B5-346-1          | B5-353-1 | B6-4-1  | B6-10-1  |
| a. Liquefaction experiments at 440°C for 60 minutes, using 3 g Black Thunder coal, 2.90 g V-130 deashed resid and 1.43 g V-1074 heavy distillate from Wilsonville Run 258A. 1350 psig cold charge pressure, including 3% H <sub>2</sub> S. SO <sub>3</sub> -free ash basis. |          |              |                   |          |         |          |

| Table 10. Metal impregnated Wyodak coals |                  |                  |                  |                             |
|--|------------------|------------------|------------------|-----------------------------|
| Coal sample no.                          | Mo, ppmw mf coal | Ni, ppmw mf coal | Fe, ppmw mf coal | Moisture, wt% as-determined |
| CB-51                                    | 552              | -                | -                | 30.1                        |
| CB-52                                    | 538              | 101              | -                | 21.0                        |
| CB-53                                    | 478              | -                | 6730             | 22.0                        |

Table 11

## Mid-range pretreatment of NiMo impregnated coal

|   | B6-26-1 | B6-78-1 | B6-66-1 | B6-61-1 | B6-65-2 | B6-4-1  |
|---|---------|---------|---------|---------|---------|---------|
| Run Number                              | B6-26-1 | B6-78-1 | B6-66-1 | B6-61-1 | B6-65-2 | B6-4-1  |
| RXN Temperature                         | 440     | 275/440 | 300/440 | 340/440 | 375/440 | 440     |
| Run Time                                | 30      | 30/30   | 30/30   | 30/30   | 30/30   | 60      |
| % moisture in the coal (salt free)      | 21.06%  | 21.06%  | 21.06%  | 21.06%  | 21.06%  | 21.06%  |
| Products, wt% maf Coal                  |         |         |         |         |         |         |
| HC Gases                                | 11.95%  | 13.44%  | 14.27%  | 14.28%  | 14.26%  | 17.94%  |
| CO <sub>2</sub> + CO                    | 11.46%  | 11.57%  | 11.81%  | 11.68%  | 11.69%  | 11.54%  |
| Total Gases                             | 23.41%  | 25.01%  | 26.08%  | 25.96%  | 25.95%  | 29.47%  |
| 975-                                    | 35.41%  | 39.36%  | 44.91%  | 35.47%  | 39.73%  | 59.62%  |
| PA + A                                  | 0.00%   | 0.00%   | 0.00%   | 0.00%   | 0.00%   | 0.00%   |
| 975+                                    | 41.18%  | 35.63%  | 29.01%  | 38.57%  | 34.32%  | 10.90%  |
| Total                                   | 100.00% | 100.00% | 100.00% | 100.00% | 100.00% | 100.00% |
| Resid Conversion, %                     | 26.87%  | 29.37%  | 32.41%  | 28.09%  | 29.43%  | 40.70%  |
| H <sub>2</sub> consumed, mg/g maf Resid | 54.3    | 59.6    | 60.7    | 58.2    | 70.8    | 70.3    |
| % H <sub>2</sub> consumed               | 25.8%   | 28.2%   | 28.5%   | 27.8%   | 32.3%   | 33.5%   |
| Wt of catalyst added (g)                | 0.0000  | 0.0000  | 0.0000  | 0.0000  | 0.0000  | 0.0000  |
| Initial wt of DMDS (g)                  | 0.0000  | 0.0000  | 0.0000  | 0.0000  | 0.0000  | 0.0000  |
| THF Conversion                          | 89.98%  | 93.77%  | 95.33%  | 91.70%  | 95.65%  | 92.27%  |
| Recovery efficiency, %                  | 98.01%  | 98.64%  | 99.52%  | 100.35% | 97.65%  | 88.95%  |

Table 12

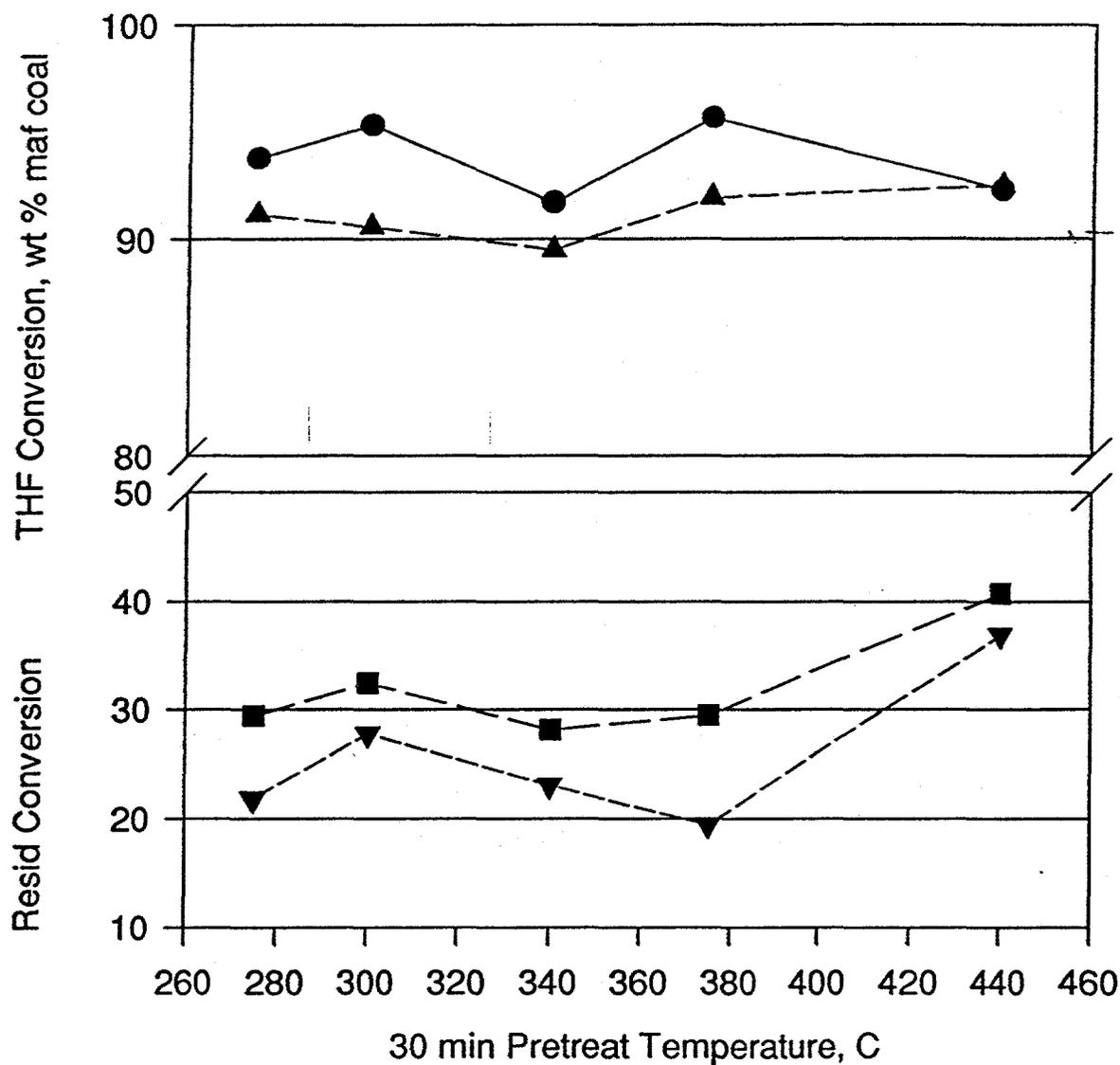
## Mid-range pretreatment of FeMo impregnated coal

| Run Number                              | B6-102-2 | B6-122-1 | B6-102-3 | B6-102-1 | B6-110-1 | B6-10-1 |
|---|----------|----------|----------|----------|----------|---------|
| RXN Temperature                         | 440      | 275/440  | 300/440  | 340/440  | 375/440  | 440     |
| Run Time                                | 30       | 30/30    | 30/30    | 30/30    | 30/30    | 60      |
| % moisture in the coal (salt free)      | 22.58%   | 35.56%   | 22.58%   | 22.58%   | 35.56%   | 22.58%  |
| Products, wt% maf Coal                  |          |          |          |          |          |         |
| HC Gases                                | 13.70%   | 14.23%   | 13.41%   | 13.12%   | 11.33%   | 17.22%  |
| CO <sub>2</sub> + CO                    | 12.43%   | 12.14%   | 11.35%   | 10.95%   | 11.71%   | 11.21%  |
| Total Gases                             | 26.14%   | 26.38%   | 24.75%   | 24.07%   | 23.04%   | 28.43%  |
| 975-                                    | 25.43%   | 27.83%   | 37.54%   | 27.63%   | 25.26%   | 54.19%  |
| PA + A                                  | 0.00%    | 0.00%    | 0.00%    | 0.00%    | 0.00%    | 0.00%   |
| 975+                                    | 48.43%   | 45.79%   | 37.70%   | 48.30%   | 51.70%   | 17.38%  |
| Total                                   | 100.00%  | 100.00%  | 100.00%  | 100.00%  | 100.00%  | 100.00% |
| Resid Conversion, %                     | 22.97%   | 21.78%   | 27.75%   | 23.00%   | 19.41%   | 36.83%  |
| H <sub>2</sub> consumed, mg/g maf Resid | 51.6     | 59.6     | 56.4     | 53.4     | 64.4     | 65.6    |
| % H <sub>2</sub> consumed               | 23.5%    | 23.2%    | 26.2%    | 24.9%    | 24.8%    | 29.2%   |
| Wt of catalyst added (g)                | 0.0000   | 0.0000   | 0.0000   | 0.0000   | 0.0000   | 0.0000  |
| Initial wt of DMDS (g)                  | 0.0000   | 0.0000   | 0.0000   | 0.0000   | 0.0000   | 0.0000  |
| THF Conversion                          | 89.86%   | 91.15%   | 90.58%   | 89.51%   | 91.94%   | 92.46%  |
| Recovery efficiency, %                  | 102.84%  | 101.23%  | 101.95%  | 102.47%  | 99.51%   | 94.21%  |

Figure 1

## Ni/Mo and Fe/Mo IW Impregnated Coal

Effect of Pretreatment Temperature



3% H<sub>2</sub>S, 1350 psig total pressure, cold

Ni/Mo coal: 500 ppm IW Mo plus 100 ppm IW Ni

Fe/Mo coal: .67% IW Fe plus 500 ppm IW Mo

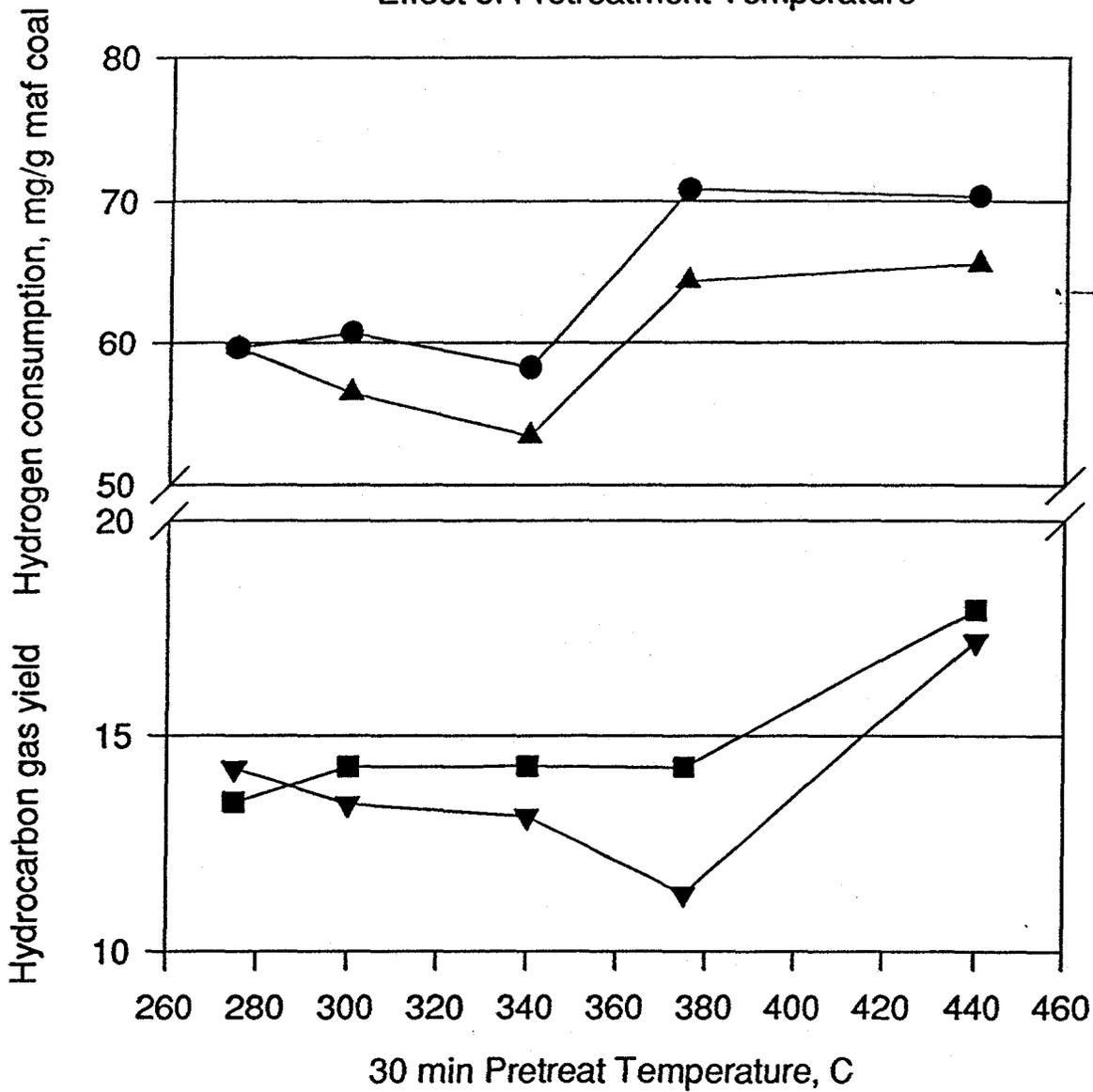
30 min hydrotreatment at 440 C, 60 min total run

- % THF Conv, Ni/Mo coal
- % Rc, Ni/Mo coal
- ▲ % THF Conv, Fe/Mo coal
- ▼ % Rc, Fe/Mo coal

Figure 2

## Ni/Mo and Fe/Mo IW Impregnated Coal

Effect of Pretreatment Temperature



3% H<sub>2</sub>S, 1350 psig total pressure, cold

Ni/Mo coal: 500 ppm IW Mo plus 100 ppm IW Ni

Fe/Mo coal: .67% IW Fe plus 500 ppm IW Mo

30 min hydrotreatment at 440 C, 60 min total run

- H<sub>2</sub> Cons, Ni/Mo coal
- H/C Gas Yield, Ni/Mo coal
- ▲ H<sub>2</sub> Cons, Fe/Mo coal
- ▼ H/C Gas Yield, Fe/Mo coal

**SECTION TWO**

**CONSOL**

August 28, 1996

Report Period: January 1 - March 31, 1996

**SUBCONTRACT TITLE AND NUMBER:**

Subcontract UKRF-4-25582-92-76 to CONSOL Inc.

Under DOE Contract No. DE-AC22-91PC91040, "Advanced Coal Liquefaction Concepts for the PETC Generic Bench-Scale Unit"

**SUBCONTRACTOR NAME:**

CONSOL Inc.

Research & Development

4000 Brownsville Road

Library, PA 15129

**SUBCONTRACT PERIOD:** November 26, 1991 - September 30, 1996

**PRINCIPAL INVESTIGATORS:** F. P. Burke, R. A. Winschel, G. A. Robbins

**SUBCONTRACT OBJECTIVES:** No change.

**SUMMARY OF TECHNICAL PROGRESS - OVERALL**

The plan for Run ALC-1 was finalized. Agglomeration conditions were found in the laboratory that provide acceptable ash rejection, organic recovery, and product size with the Run ALC-1 feed coal at an acceptable dosage of the selected agglomerating oil. The 50 gal (189 L) agglomerator unit was set up and 1300 lb (590 kg) of cleaned coal was produced, dried, and shipped to HTI for Run ALC-1. All technical and scheduling targets were met. Equipment specifications, operating conditions, energy usage, feed consumption and product yields were measured and recorded and product characteristics will be determined to assist in the engineering and economic assessment. The request for samples from Run ALC-1 for characterization was finalized.

Ongoing and near-future activities include agglomeration product analysis, data work-up, additional evaluation of the liquefaction reactivity of the agglomerate products, characterization of the Sandia hydrotreating samples and HTI dewaxing samples, final review of HTI's run plan, and on-site monitoring of Run ALC-1 operations.

## **SUMMARY OF TECHNICAL PROGRESS - BY TASK**

### **Task 2 - Laboratory Support**

The four drums of Black Thunder coal sent to us by HTI for agglomeration were sampled and analyzed. A larger composite sample also was prepared for use in the laboratory agglomeration tests. The analyses (Table 1) are similar enough that we did not blend the four drums. A fifth drum of Black Thunder Mine coal received from HTI, packed and shipped separately from the first four drums, was analyzed. It is slightly lower in moisture content than the previous drums of coal (Table 1).

We selected as the agglomerating oil, V-1074 distillate from end-of-run Wilsonville Run 263, which used Black Thunder Mine coal. About 440 lb (200 kg) of this oil is available.

Thirty-five laboratory agglomeration tests were made with the composite sample of the HTI Black Thunder Mine coal and three different oils (Table 2). The laboratory agglomeration tests were made to guide the large-scale production runs. The primary objective was to maximize ash rejection. Secondary objectives included maximizing agglomerate size and minimizing oil dosage. It is desirable to minimize oil dosage in order to minimize the use of oil generated outside of Run ALC-1. Large agglomerates are desired for ease of dewatering. Most of the runs were made at pH 1 (adjusted with sulfuric acid). Ash balances, including the filtrate ash, were consistently higher than 100% (Table 2), because the formation of calcium sulfate creates additional ash in the reject. The initial tests with the agglomeration oil selected for the production runs (Wilsonville Run 263 end-of-run V-1074) gave poorer deashing results than other oils tested (2 to 23% ash rejection vs. 42% with No. 6 fuel oil, and 31 to 38% with HTI Run CMSL-9 period 29 make-up oil). Some of the operating conditions studied during optimization included acid dosage, oil dosage, use of tap water (usually deionized water has been used), and temperature. The tests showed that agglomeration at 70 °C gives improved performance with this oil (ash rejection was increased to 37 to 44% from  $\leq 26\%$  at  $\leq 54$  °C). Most tests with the baseline oil at 70 °C gave >99% organic recovery, and well-formed agglomerates with an average size of approximately 2 mm in diameter or larger. Poorer performance is obtained at reduced oil dosage, reduced acid dosage, lower temperature, or shorter operating time. Run A75, with 41% ash rejection, was selected as the benchmark agglomeration test.

The sulfur content of the agglomerates from Run A75 is approximately double that of the feed coal when expressed on a dry coal basis. The sulfur content of an ashed filter cake is equivalent to material that is 80% anhydrite ( $\text{CaSO}_4$ ).

Agglomerated product from Run A72, and a sample of the agglomerating oil were sent to R. Anderson at CAER for use some in microautoclave tests of catalyst activity improvements.

### **Dewaxing**

The pressure filter liquid sample from HTI Run CMSL-9-29 was vacuum distilled to an 850 °F (454 °C) equivalent end point and the distillate was dewaxed with acetone at -5 and -20 °C. Wax yields were low at 4.9 and 5.3%, respectively (Table 3). Material recoveries were 98.5% or greater in both cases. The low wax yields are consistent with the low concentration of paraffinic protons in the feed oil (Table 4). If this material is representative of the VSOH that will be produced in Run ALC-1, then dewaxing may not show much of an improvement in liquefaction performance. On the other hand, some of the VSOH was purged as net product in Run CMSL-9. It is probable that in the absence of this purge, the wax content of this material would have been greater. In Run ALC-1, the purge of heavy distillate will be reduced to a minimum.

### **Characterization of Sandia Hydrotreating Samples**

The feed and eight products from Sandia hydrotreating tests are being analyzed for elemental composition and proton distribution. The feed, a vacuum overhead from the PFL from period 10 of HTI Run 227-90, contains 1.8% S, surprisingly high. Product oils vary substantially in heteroatom content. Microautoclave tests are in progress to assess the donor quality of a Sandia hydrotreated product distillate relative to the feed distillate.

### **Task 3 - Continuous Operations**

#### **Formulation of Plans for Run ALC-1**

The plan for Run ALC-1 was finalized (Table 5). It was agreed to operate the reactors at 2500 psig (17.2 MPa) total pressure (not  $\text{H}_2$  partial pressure), and it was agreed that the key indicator for adjusting process severity (i.e., space velocity) would be maintaining extinction recycle of pressure filter liquid.

#### **Coal/Oil Agglomerate Production for Run ALC-1**

The operating procedure and safety checklist for the agglomeration production runs were prepared and approved, and all site, utility, and supply arrangements were made to operate the

50 gal (189 L) batch agglomerator. The feedstocks were pulverized Black Thunder Mine coal (from HTI) and Wilsonville Run 263 end-of-run V-1074 vacuum distillate. Tap water and concentrated sulfuric acid also were used.

The target conditions for the shakedown test, Run C1, were 90 lb (41 kg) as-is coal, 23% MF oil, 22 gal (83 L) water, 0.26 gal (1 L) acid, 158 °F (70 °C), and 425 rpm, 1 h conditioning time, 90 min agglomeration time. Operations generally went smoothly; however, the agglomerate size was smaller than desired. Small incremental oil dosages up to a total of 32.1% MF, extended agglomeration time, and a reduction in stirrer speed to 350 rpm all failed to result in substantial agglomerate growth. The agglomerates were washed with fresh tap water until the wash water pH increased to 4.

Production Runs C2 through C10 were made, completing the production of material for HTI (Table 1). In order to increase product size in the production runs after C1, several operating changes were made: the slurry concentration was increased to 43% by reducing the water charge and increasing the coal charge; the stirrer speed was increased from 425 to 465 rpm; and the oil dosage was increased from 32.1% MF to 33.5% MF. These changes resulted in good agglomerate size of typically ca. 2 to 4 mm diameter, but the range of agglomerate size was from ca. 1 to 7 mm diameter. Operations were smooth, despite some run-to-run inconsistency in agglomerate size. The standard recipe for Runs C2 through C9 was 112.5 lb (51.0 kg) coal (ca. 10% moisture), 18 gal (68 L) H<sub>2</sub>O, 34.0 lb (15.4 kg) oil, 0.38 gal (1450 mL) (by weight, 5.5 lb or 2.5 kg) concentrated sulfuric acid, 158 °F (70 °C), 465 rpm stirrer speed, 60 minute acid conditioning time, 90 minute agglomeration time, and rinse to pH 4. Agglomeration Run C10, performed with coal from the last drum, required more agglomeration time (110 min vs. 90 min), perhaps because the MF oil dosage was slightly low as a consequence of the slightly lower coal moisture content.

Measurements of the current drawn by the agglomerator stirrer during Run C7 show that it consumed from 3021 to 3759 W of power, depending on the stage of the run. This corresponds to a power input of approximately 0.87 to 1.08 hp/ft<sup>3</sup> (22,910 to 28,440 W/m<sup>3</sup>) of slurry volume.

For all runs, visual observations indicate that there was near quantitative recovery of organics with the product agglomerates, which were recovered on a 48 Tyler mesh screen. The screen underflow was milky white from gypsum.

Agglomerates from production Runs C1 through C10 were dried to less than 10% moisture, 1-gallon samples were riffled from each batch, and the product was packed into five drums for shipment. A single composite product was prepared from Runs C2 through C9 for laboratory tests. Products from Runs C1 through C4 were dried in ovens at 77 °F (25 °C) for 71 hours, 100 °F (38 °C) for 20 to 27 hours, and 140 °F (60 °C) for 6 to 8 hours with a nitrogen purge flow. Products from Runs C5 through C10 were air-dried on the floor for 4.5 to 5.5 days. Rather than blending all runs, the product from each run was packed into a separate plastic bag, and the bags packed two per drum. (One drum contains an extra bag of some of the product from both Runs C6 and C7, which were accidentally mixed.) This was done because the agglomerates showed rather consistent ash content among runs and because HTI indicated that this was suitable for them. The agglomerated products from Runs C1 through C10 (ca. 1300 lb, or 590 kg) were shipped to HTI. HTI was provided with our preferred order for feeding the material during Run ALC-1; i.e., to feed agglomerates from Runs C2 through C9 in order, then the mixed Run C6-C7 agglomerates, then the agglomerates from Run C1, and from Run C10 last.

#### **Product Agglomerate Properties**

The SO<sub>3</sub>-free ash contents of agglomerated products from individual Runs C1 through C10 were fairly consistent at 3.0 to 3.6 wt % MF oil-free. SO<sub>3</sub>-free ash rejections based on these values are 37 to 47 wt %.

The analytical data on agglomerates from Runs C2 through C9 were averaged to obtain an approximate composition of the agglomerates to be fed in Run ALC-1. As a percent of the whole agglomerate, the agglomerates are composed of 8.0% moisture, 23.4% distillate, 2.4% SO<sub>3</sub>-free ash, and 66.2% MAF coal (68.6% MF coal), on the basis of the available data. These data were provided to HTI so that they could set approximate feed rates for the bench unit. The final compositions will be calculated from analyses of the riffled samples of the final agglomerate products and the composite sample. A complete suite of analyses is being conducted on the agglomerates, including ultimate, proximate, Btu, ash elemental, and particle size distribution. The feed coal, oil, and reject streams are also being analyzed. Material and elemental balances will be determined from several of the runs.

The bulk density of one batch of the agglomerates was determined to be 43 lb/ft<sup>3</sup> (688 kg/m<sup>3</sup>).

Microautoclave tests are in progress to assess the reactivity of the composite agglomerate product, relative to the feed coal. Tests completed to date indicate that the agglomerates give lower conversion to THF solubles at the conditions tested; more tests are being conducted.

The agglomerated product from Run C2 was extracted with acetone to determine the MF coal/oil ratio of the agglomerates. The recovered solubles accounted for 27% of the total dry agglomerate. The original moisture-free charge to the agglomerator was 25% oil and the oil should account for about 26% of the MF deashed product. Thus, the oil content of the agglomerates determined by the acetone wash and by the known feed mix agree to within about 1% absolute. The composite oil agglomerated coal was then separated into the coal and the agglomerating oil by this method and the two fractions were submitted for analyses to determine the form of the sulfur added during low-pH agglomeration. The sulfur content of the oil-free coal product is approximately 0.5% higher than that of the feed coal, presumably because of sulfate retention.

HTI requested a sample of the agglomerating oil to obtain distillation data prior to the run. The sample and available analytical data were sent.

### **Run ALC-1 Sample Request**

Our request for samples from Run ALC-1 for characterization was finalized. Particular care was taken to reduce perturbation of the system from the loss of internal process oils

### **Exploratory Production-Scale Agglomeration Run**

An exploratory production-scale run, Run C11, was made with the left-over coal and oil to see if residence time for agglomeration could be reduced. The slurry concentration was held at 43%, but the total slurry volume was reduced by 11% (coal charge was 100 lb, or 45.4 kg), the stirrer speed was increased to 520 rpm, the temperature was increased to 176 °F (80 °C), and the oil dosage was 38.5 wt % MF initially (incremented to nearly 40 wt %). The run was terminated after 45 minutes agglomeration time, instead of the usual 90 minutes agglomeration time. The product agglomerates were generally 2-3 mm diameter, but they had a grainy appearance resulting from the presence of a small amount of fine agglomerates. Results were favorable for reducing agglomeration time.

**Task 4 - Technical Assessment**

The abstract for the Thirteenth Annual International Pittsburgh Coal Conference entitled "Testing of Advanced Liquefaction Concepts in HTI Run ALC-1: Coal Cleaning and Recycle Solvent Treatment" was sent to the conference organizers and DOE. An abstract, "Testing of Advanced Liquefaction Concepts in HTI Run ALC-1", for a presentation at the DOE Joint Power and Fuel Systems Contractors Conference was drafted for review internally and by project participants.



F. P. Burke  
Project Manager

/s

TABLE 1

ANALYSIS OF HTI BLACK THUNDER MINE COAL FOR AGGLOMERATION

|  | Black Thunder Mine Coal, HTI No. 6213 |        |        |        |        |
|--|---------------------------------------|--------|--------|--------|--------|
|  | Drum 1                                | Drum 2 | Drum 3 | Drum 4 | Drum 5 |
| Moisture, wt % As-Determined                             | 10.00                                 | 10.00  | 9.98   | 9.65   | 8.47   |
| Ash, wt % MF, Including SO <sub>3</sub>                  | 6.12                                  | 6.31   | 6.22   | 6.28   | 6.34   |
| <u>Proximate, wt % MF, SO<sub>3</sub>-Free Ash Basis</u> |                                       |        |        |        |        |
| Ash, SO <sub>3</sub> -Free                               | 5.39                                  | 5.56   | 5.49   | 5.55   | 5.76   |
| Volatile Matter  | 43.65                                 | 43.72  | 43.63  | 43.61  | 43.69  |
| Fixed Carbon   | 50.96                                 | 50.72  | 50.88  | 50.84  | 50.55  |
| <u>Ultimate, wt % MF, SO<sub>3</sub>-Free Ash Basis</u>  |                                       |        |        |        |        |
| Carbon   | 71.00                                 | 71.21  | 71.05  | 71.05  | 69.83  |
| Hydrogen   | 4.67                                  | 4.69   | 4.68   | 4.73   | 4.67   |
| Nitrogen   | 0.92                                  | 0.92   | 0.91   | 0.91   | 0.96   |
| Sulfur   | 0.49                                  | 0.52   | 0.49   | 0.48   | 0.44   |
| Oxygen (by diff)   | 17.53                                 | 17.10  | 17.38  | 17.28  | 18.34  |
| Ash, SO <sub>3</sub> -Free                               | 5.39                                  | 5.56   | 5.49   | 5.55   | 5.76   |
| SO <sub>3</sub> , Wt % of Ash                            | 11.94                                 | 11.81  | 11.81  | 11.57  | 9.20   |
| HHV, Btu/lb (MF)   | 12,066                                | 12,061 | 12,050 | 12,098 | 11,753 |

TABLE 2. AGGLOMERATION RESULTS

| Run (a)  | Coal ID, Slurry Conc., wt % | Oil | Oil Dosage, wt % Added to MF Coal | pH, H <sub>2</sub> SO <sub>4</sub> | Temp., °C | Product Ash, wt % Dry, Oil-Free | Organic Recovery, wt % | Ash Rejection (b) (Dissolved & Physical), wt % | Physical Ash Rejection (c), wt % | Ash Balance (d), wt % | Dissolved Solids Ash Balance (e), wt % |
|--|-----------------------------|-----|-----------------------------------|------------------------------------|-----------|---------------------------------|------------------------|--|----------------------------------|-----------------------|--|
| January -March 1996 Laboratory Agglomeration Runs, SO <sub>2</sub> -Containing Ash Basis |                             |     |                                   |                                    |           |                                 |                        |  |                                  |                       |  |
| A57  | BT-6, 20%                   | P1  | 28.8                              | 1.0                                | 54        | 3.52                            | 100.0                  | 42.2   | 10.51                            | 68.28                 | 126.8                                  |
| A58  | BT-6, 20%                   | C9  | 13.1                              | 1.0                                | 54        | 4.16                            | 99.8                   | 31.3   | 15.57                            | 84.26                 | 129.3                                  |
| A59  | BT-6, 20%                   | C9  | 18.0                              | 1.0                                | 54        | 3.79                            | 100.0                  | 37.5   | 4.52                             | 66.98                 | 132.2                                  |
| A60  | BT-6, 20%                   | C9  | 22.3                              | 1.0                                | 54        | 3.95                            | 100.0                  | 34.8   | 3.43                             | 68.60                 | 138.9                                  |
| A61  | BT-6, 20%                   | C9  | 26.7                              | 1.0                                | 54        | 4.12                            | 100.0                  | 31.9   | 4.47                             | 72.52                 | 136.7                                  |
| A62  | BT-6, 20%                   | C9  | 30.4                              | 1.0                                | 54        | 3.96                            | 100.0                  | 34.6   | 5.30                             | 70.67                 | 128.4                                  |
| A63  | BT-6, 33%                   | C9  | 22.6                              | 1.0                                | 54        | 4.08                            | 100.0                  | 32.7   | 3.6                              | 70.9                  | 133.1                                  |
| A64  | BT-6, 20%                   | C10 | 26.7                              | 1.0                                | 54        | 4.97                            | 100.0                  | 17.2   | 1.2                              | 84.0                  | 132.3                                  |
| A65  | BT-6, 20%                   | C10 | 32.8                              | 1.0                                | 54        | 4.65                            | 100.0                  | 22.7   | 1.8                              | 79.1                  | 130.9                                  |
| A66  | BT-6, 20%                   | C10 | 32.3                              | 1.0                                | 40        | 5.02                            | 100.0                  | 16.3   | 0.8                              | 84.5                  | 130.5                                  |
| A67  | BT-6, 33%                   | C10 | 32.8                              | 1.0                                | 40        | 5.81                            | 99.9                   | 2.2  | 1.2                              | 99.0                  | 128.7                                  |
| A68  | BT-6, 33%                   | C10 | 31.9                              | 1.0                                | 40        | 5.40                            | 100.0                  | 9.5  | 2.1                              | 92.6                  | 128.1                                  |
| A69  | BT-6, 33%                   | C10 | 31.4                              | 1.0                                | 35        | 5.13                            | 100.0                  | 14.4   | 2.6                              | 88.2                  | 129.0                                  |
| A70  | BT-6, 20%                   | C10 | 33.7                              | 0.8                                | 54        | 4.42                            | 100.0                  | 26.8   | 3.3                              | 76.5                  | 124.5                                  |
| A71  | BT-6, 20%                   | C10 | 30.9                              | 1.0                                | 54        | 4.84                            | 100.0                  | 19.5   | 2.2                              | 82.7                  | 127.5                                  |
| A72  | BT-6, 20%                   | C10 | 31.9                              | 1.0                                | 70        | 3.65                            | 99.9                   | 40.0   | 17.1                             | 77.1                  | 127.8                                  |
| A73  | BT-6, 20%                   | C10 | 31.8                              | 1.0                                | 70        | 3.84                            | 100.0                  | 36.8   | 3.3                              | 66.5                  | 134.3                                  |
| A74  | BT-6, 33%                   | C10 | 31.0                              | 0.8                                | 70        | 3.80                            | 100.0                  | 37.5   | 10.6                             | 73.1                  | 126.4                                  |
| A75  | BT-6, 33%                   | C10 | 30.4                              | 1.0                                | 70        | 3.60                            | 99.7                   | 40.9   | 29.0                             | 88.2                  | 126.5                                  |
| A76  | BT-6, 33%                   | C10 | 29.8                              | 0.8                                | 80        | 3.46                            | 99.8                   | 43.3   | 29.4                             | 86.1                  | 105.4                                  |
| A77  | BT-6, 43%                   | C10 | 31.4                              | 1.0                                | 70        | 3.81                            | 99.4                   | 37.3   | 30.3                             | 93.0                  | 129.7                                  |
| A78 (f)  | BT-6, 33%                   | C10 | 30.2                              | 1.0                                | 70        | 3.75                            | 99.9                   | 38.4   | 30.3                             | 92.0                  | -                                      |
| A79 (f)  | BT-6, 33%                   | C10 | 30.1                              | 1.0                                | 70        | 3.88                            | 99.8                   | 50.1 (g)                                       | 35.7                             | 85.6                  | -                                      |
| A80 (f)  | BT-6, 33%                   | C10 | 30.5                              | 1.0                                | 70        | 3.91                            | 99.3                   | 35.6   | -                                | 64.4                  | -                                      |
| A81 (f)  | BT-6, 33%                   | C10 | 30.4                              | 1.0                                | 70        | 3.96                            | 100.0                  | 34.7   | 1.6                              | 66.9                  | -                                      |

| Run (a)  | Coal ID, Slurry Conc., wt % | Oil | Oil Dosage, wt % Added to MF Coal | pH, H <sub>2</sub> SO <sub>4</sub> | Temp., °C | Product Ash, wt % Dry, Oil-Free | Organic Recovery, wt % | Ash Rejection (b) (Dissolved & Physical), wt % | Physical Ash Rejection (c), wt % | Ash Balance (d), wt % | Dissolved Solids Ash Balance (e), wt % |
|--|-----------------------------|-----|-----------------------------------|------------------------------------|-----------|---------------------------------|------------------------|--|----------------------------------|-----------------------|--|
| A82 (f)  | BT-6, 33%                   | C10 | 30.5                              | 1.0                                | 70        | 3.42                            | 100.0                  | 44.0   | 4.5                              | 60.6                  | -                                      |
| A83  | BT-6, 33%                   | C10 | 30.8                              | 1.0                                | 70        | 3.52                            | 99.5                   | 42.3   | 16.9                             | 74.6                  | 123.5                                  |
| A84  | BT-6, 33%                   | C10 | 25.1                              | 1.0                                | 70        | 4.39                            | 99.8                   | 27.3   | 19.5                             | 92.2                  | 132.9                                  |
| A85  | BT-6, 33%                   | C10 | 31.1                              | 0.2                                | 70        | 3.61                            | 99.8                   | 40.7   | 24.6                             | 83.9                  | 131.9                                  |
| A86  | BT-6, 33%                   | C10 | 30.1                              | 0.6                                | 70        | 5.06                            | 99.9                   | 15.6   | 5.4                              | 89.7                  | 129.7                                  |
| A87  | BT-6, 33%                   | C10 | 30.9                              | 1.0                                | 70        | 3.82                            | 99.9                   | 37.2   | 21.5                             | 84.4                  | 130.0                                  |
| A88  | BT-6, 33%                   | C10 | 32.2                              | 1.0                                | 70        | 3.92                            | 99.6                   | 35.3   | 25.8                             | 90.4                  | 135.2(m)                               |
| A89  | BT-6, 33%                   | C10 | 31.2                              | 1.0                                | 70        | 3.18                            | 100.0                  | 48.0   | 27.4                             | 79.4                  | 116.6                                  |
| A90  | BT-6, 33%                   | C10 | 30.2                              | 1.0                                | 70(i)     | 4.37                            | 100.0                  | 27.7   | 11.8                             | 84.1                  | -                                      |
| A91  | BT-6, 43%                   | C10 | 35.0                              | 0.8                                | 70        | 3.96                            | 99.8                   | 34.7   | 13.4                             | 78.7                  | -                                      |
| A92  | BT-6, 33%                   | C10 | 33.5                              | 1.3                                | 80        | 3.93                            | 99.7                   | 35.2   | 5.5                              | 70.3                  | -                                      |
| March 1996 ALC-1 Production Agglomeration Runs, SO <sub>2</sub> -Free Ash Basis (see note below) |                             |     |                                   |                                    |           |                                 |                        |  |                                  |                       |  |
| C1   | BT-7, 33%                   | C10 | 32.1                              | 1.5                                | 70        | 3.56                            | 99.5                   | 36.6   | 45.6                             | 109.0                 | -                                      |
| C2   | BT-7, 43%                   | C10 | 33.5                              | 0.3                                | 70        | 3.27                            | 99.8                   | 41.9   | 38.0                             | 96.1                  | -                                      |
| C3   | BT-7, 43%                   | C10 | 33.4                              | 0.3                                | 70        | 3.28                            | 99.6                   | 41.7   | 36.6                             | 94.8                  | -                                      |
| C4   | BT-7, 43%                   | C10 | 33.2                              | 0.3                                | 70        | 3.46                            | 99.8                   | 38.4   | 24.0                             | 85.6                  | -                                      |
| C5   | BT-7, 43%                   | C10 | 33.3                              | 0.3                                | 80        | 3.37                            | 99.7                   | 40.2   | 38.8                             | 98.7                  | -                                      |
| C6   | BT-7, 43%                   | C10 | 33.3                              | 0.3                                | 70        | 3.23                            | 99.5                   | 42.6   | 31.3                             | 88.6                  | -                                      |
| C7   | BT-7, 43%                   | C10 | 33.3                              | 0.3                                | 70        | 3.27                            | 99.8                   | 41.9   | 10.8                             | 68.9                  | -                                      |
| C8   | BT-7, 43%                   | C10 | 33.4                              | 0.3                                | 70        | 3.25                            | 99.7                   | 42.3   | 26.3                             | 84.0                  | -                                      |
| C9   | BT-7, 43%                   | C10 | 33.6                              | 0.3                                | 70        | 3.02                            | 99.7                   | 46.5   | 22.5                             | 76.1                  | -                                      |
| C10  | BT-8 (h), 43%               | C10 | 33.1                              | 0.3                                | 70        | 3.28                            | 99.9                   | 44.5   | 30.6                             | 86.1                  | -                                      |
| C6 & C7(i)   | BT-7, 43%                   | C10 | 33.3                              | 0.3                                | 70        | 3.37                            | 100.0                  | 40.1   | 0.0                              | 59.9                  | -                                      |
| C2-C9 (j)  | BT-7, 43%                   | C10 | 33.4                              | 0.3                                | 70        | 3.27                            | 100.0                  | 42.0   | 0.0                              | 58.0                  | -                                      |
| C11 (k)  | BT-8, 43%                   | C10 | 39.7                              | 0.3                                | 80        | 3.66                            | 99.9                   | 37.9   | 27.4                             | 89.5                  | -                                      |

Coal: BT-6 = Lab aliquot of Drums 1-4 of HTI-supplied Black Thunder Mine coal (moisture content 9.42% as-determined, ash content 5.94% MF, including SO<sub>2</sub>). BT-7 = Drums 1-4 of HTI-supplied Black Thunder Mine coal (moisture content 9.91% as-determined, ash content 5.50% MF, excluding SO<sub>2</sub>). BT-8 = Drum 5 of HTI-supplied Black Thunder Mine coal (moisture content 8.47% as-determined, ash content 5.76% MF, excluding SO<sub>2</sub>).

Oil: C10 = V-1074 distillate from end-of-run Wilsonville Run 263.

- (a) Run numbers with prefix A were made in the large lab vessel (working capacity of ca. 250-600 mL slurry). Run numbers with prefix C were made in the 50-gal pilot plant vessel (working capacity of ca. 33 gal slurry).
- (b) Based on feed ash which was not recovered in the product.
- (c) Based on feed ash which was recovered in the solid reject (filter cake).
- (d) Ash balance does not include dissolved ash.
- (e) Ash balance includes dissolved ash.
- (f) Results for this run also may reflect changes in other conditions that are not explicitly shown. Examples of such conditions included different acid conditioning and agglomeration times, and intermediate rinses of agglomerates soon after oil addition.
- (g) The high ash rejection may be an artifact of other unusual run conditions.
- (h) Actual feed was 5% BT-7 and 95% BT-8.
- (i) Inadvertent blend of ca. 67% C6 product and 33% C7 product, shipped to HTI.
- (j) Lab composite of C2-C9 products, represents material most likely to be fed by HTI.
- (k) Exploratory large-scale test to reduce agglomeration time.
- (l) No conditioning time
- (m) Based partly on an estimated value.

General notes:

- pHs shown in table are nominal; usually the run conditions were controlled on acid dose.
- Acid leach times and agglomeration times were varied for some runs. Leach times ranged from 0 to 60 minutes for lab tests and were fixed at 60 minutes for production runs. Agglomeration times ranged from 60 to 180 minutes in lab tests, and ranged from 45 to 90 minutes in production runs.

Notes for Runs C1-C11:

- Organic rejection, filter ash rejection, and ash balance are considered approximate (based partially on estimates) until reject ash balance is finalized.

**TABLE 3**  
**ACETONE DEWAXING OF**  
**HTI RUN CMSL-9-29 PFL 850 °F DISTILLATE**

| Dewaxing Temp, °C   | -5   | -20  |
|---------------------|------|------|
| <b>Yields, wt %</b> |      |      |
| Dewaxed Oil         | 94.4 | 93.2 |
| Wax                 | 4.9  | 5.3  |
| Insolubles          | 0.2  | 0.2  |
| Total Recovery      | 99.5 | 98.7 |

**TABLE 4**  
**PROTON DISTRIBUTIONS OF**  
**DEWAXING SAMPLES**

|                    | Proton Distributions |             |              |             |             |            |       |
|--------------------|----------------------|-------------|--------------|-------------|-------------|------------|-------|
|                    | Cond Arom            | Uncond Arom | Cyclic Alpha | Alkyl Alpha | Cyclic Beta | Alkyl Beta | Gamma |
| Feed Oil (a)       | 13.8                 | 11.2        | 14.4         | 11.6        | 13.0        | 24.0       | 11.9  |
| -5 °C Wax          | 0.6                  | 0.4         | 1.9          | 1.6         | 4.1         | 79.4       | 11.9  |
| -20 °C Wax         | 0.5                  | 1.1         | 2.7          | 2.8         | 6.5         | 73.1       | 13.3  |
| -5 °C Dewaxed Oil  | 16.4                 | 11.3        | 17.0         | 12.9        | 14.5        | 17.1       | 10.8  |
| -20 °C Dewaxed Oil | 16.9                 | 10.9        | 18.0         | 12.6        | 14.5        | 16.7       | 10.4  |

(a) HTI Run CMSL-9-29 PFL 850 °F Distillate

**TABLE 5**

**RUN PLAN (3/19/96)  
Run ALC-1**

| <u>Condition</u>                            | 1                    | 2                      | 3                      | 4                     | 5                     |
|---|----------------------|------------------------|------------------------|-----------------------|-----------------------|
| <u>Periods</u>                              | 1-5                  | 6-11                   | 12-16                  | 17-22                 | 23-25                 |
| <u>Feed Coal Pretreatment</u>               |                      |                        |                        |                       |                       |
| Black Thunder Coal <sup>a</sup>             | Raw                  | OA                     | OA                     | Raw                   | Raw                   |
| <u>Distillate Solvent</u>                   |                      |                        |                        |                       |                       |
| Pretreatment                                | None                 | None                   | None                   | DW-HT <sup>b</sup>    | DW-HT <sup>b</sup>    |
| <u>Recycle Conditions</u>                   |                      |                        |                        |                       |                       |
| Recycle Streams <sup>c</sup>                | PFL/PFC              | PFC/VSB/<br>VSOH/(PFL) | PFC/VSB/<br>VSOH/(PFL) | PFC/VSB/<br>DWHT-VSOH | PFC/VSB/<br>DWHT-VSOH |
| Recycle (incl. buffers) to MF Coal Ratio    | 1.60                 | 1.60                   | 1.60                   | <1.60 <sup>d</sup>    | <1.60 <sup>d</sup>    |
| Recycle (excl. buffers) to MF Coal Ratio    | 1.52                 | 1.52                   | 1.52                   | <1.52 <sup>d</sup>    | <1.52 <sup>d</sup>    |
| Recycle Solids/MF Coal Ratio                | 0.20                 | 0.20                   | "Y" <sup>e</sup>       | 0.20                  | 0.20                  |
| % Solids in Toluene-Washed PFC              | ~75                  | ~75                    | ~75                    | ~75                   | ~75                   |
| <u>Still Cut Points, °F (°C)</u>            |                      |                        |                        |                       |                       |
| Continuous Atmospheric Still                | 625 (329)            | 625 (329)              | 625 (329)              | 625 (329)             | 625 (329)             |
| Vacuum Still                                | -                    | 975 (524)              | 975 (524)              | 975 (524)             | 975 (524)             |
| <u>Temperature, °F (°C)</u>                 |                      |                        |                        |                       |                       |
| Pretreater                                  | 572 (300)            | 572 (300)              | 572 (300)              | 572 (300)             | 572 (300)             |
| K-1   | 824 (440)            | 824 (440)              | 824 (440)              | 824 (440)             | 851 (455)             |
| K-2   | 842 (450)            | 842 (450)              | 842 (450)              | 842 (450)             | 869 (465)             |
| HTU   | 662-716<br>(350-380) | 662-716<br>(350-380)   | 662-716<br>(350-380)   | 662-716<br>(350-380)  | 662-716<br>(350-380)  |
| <u>Space Velocity</u>                       |                      |                        |                        |                       |                       |
| lb MF coal/h/ft <sup>3</sup> , each reactor | f                    | f                      | f                      | f                     | f                     |
| <u>Catalyst Addition Rates</u>              |                      |                        |                        |                       |                       |
| Molyvan A (as Mo), ppm MF Coal              | 100                  | 100                    | ca. 70                 | 100                   | 100                   |
| Fe-based (as Fe), ppm MF Coal               | 10000                | 10000                  | ca. 7000               | 10000                 | 10000                 |
| H <sub>2</sub> S, wt % MF Coal              | 3                    | 3                      | 3                      | 3                     | 3                     |

a. OA = low-pH oil agglomeration product.

b. DW-HT: VSOH will be dewaxed using acetone at ca. 23 °F (-5 °C), then hydrotreated at conditions to be determined by HTI and other participants.

c. See text associated with plan of 2/5/96 for details.

d. Reduced by yield of wax.

e. "Y" is same recycle ash/MF coal ratio as in the baseline Condition 1.

f. Adjust to maintain recycle requirements and extinction of 650 °F\* (343 °C\*) product, suggested starting point is 41.9 lb MF coal/h/ft<sup>3</sup> (670 kg MF coal/h/m<sup>3</sup>).

**SECTION THREE**

**HYDROCARBON TECHNOLOGIES, Inc.**

Hydrocarbon Technologies, Inc.  
P.O. Box 6047  
New York & Puritan Avenues  
Lawrenceville, New Jersey 08648  
609/394-3102 Fax 609/394-9602



*Theo L.K. Lee*  
Vice President

February 7, 1996

Dr. Ed Givens  
University of Kentucky  
Center for Applied Energy Research  
3572 Iron Works Pike  
Lexington, KY 40511-8433

RE: DOE Advanced Coal Liquefaction Concepts-Phase II Program  
Subcontract: UKRF 425582-96-152  
Progress Report: January/1996

Dear Ed:

During the month ending January 31, 1996, the following subcontract services were performed in support of Phase II of the DOE Advanced Concepts Program (DE-AC22-91PC91040):

PFLS from PB-1 and PB-2 are being fractionated to produce the required VSOH (343-524°C) for testing with dewaxed/hydrogenated solvent. The wax content of PB-1 VSOH 5.15 w% and is not too much different from that of CMSL-09 VSOH. This low level of wax content may affect the necessity of the dewaxing step. A 40 lb. VSOH sample will be shipped to Sandia National Laboratories for the hydrogenation study. Also, 100 grams of Criterion 411 trilobe catalyst had been shipped to Sandia.

We have completed the preparation of the required HTI Fe based catalyst for ALC-01. This catalyst is being characterized to ensure uniformity in composition and activity.

Sincerely yours,



Theo L.K. Lee

TLK/dms

cc: R. Winschel - Consol, Inc.  
M. Peluso - LDP Associates  
J. Hu - HTI  
F. Stevens - HTI  
D. Tanner - HTI

96/LKL/021

Hydrocarbon Technologies, Inc.  
P.O. Box 6047  
New York & Puritan Avenues  
Lawrenceville, New Jersey 08648  
609/394-3102 Fax: 609/394-9602



Theo L.K. Lee  
Vice President

March 11, 1996

Dr. Ed Givens  
University of Kentucky  
Center for Applied Energy Research  
3572 Iron Works Pike  
Lexington, KY 40511-8433

RE: DOE Advanced Coal Liquefaction Concepts-Phase II program  
Subcontract: UKRF 425582-96-152  
Progress Report: February/1996

Dear Ed:

During the month ending February 29, 1996, the following activities were performed in support of Phase II of the DOE Advanced Concepts Program (DE-AC22-91PC91040):

HTI reviewed CONSOL's detailed run plan and proposed to increase the recycle/coal(mf) ratio to 1.60 in order to keep the solid content in the feed slurry below 50 %. HTI also suggested to CONSOL that temperature of 700 °F for continuous atmospheric still (CAS) would be too high and preferred to operate at 625°F.

An in-house meeting was held to discuss operation of the ALC-1 bench run. Specific discussion was made on VSOH dewaxing and off-line hydrotreating. A detailed operating run plan based on CONSOL's technical plan is being prepared.

Per CONSOL's request, 260 lbs pulverized Black Thunder coal, 50 g of Molyvan-A and 200 g of HTI iron catalyst were shipped. Also, a 40 lbs VSOH was sent to Sandia Lab for hydrotreating test. A procedure on pretreating Criterion C411 was provided to Sandia Lab.

Sincerely yours,

Theo L.K. Lee

cc: R. Winschel-CONSOL, Inc  
M. Peluso-LDP Associate  
HTI: D. Tanner F. Stevens J. Hu

Hydrocarbon Technologies, Inc.  
P.O. Box 6047  
New York & Puritan Avenues  
Lawrenceville, New Jersey 08648  
609/394-3102 Fax 609/394-9602



Theo L.K. Lee  
Vice President

April 3, 1996

Dr. Ed Givens  
University of Kentucky  
Center for Applied Energy Research  
3572 Iron Works Pike  
Lexington, KY 40511-8433

RE: DOE Advanced Coal Liquefaction Concepts-Phase II Program  
Subcontract: UKRF 425582-96-152  
Progress Report: March/1996

Dear Ed:

During the month of March, the following activities were undertaken in support of upcoming bench run (ALC-01):

Based on CONSOL's technical plan, a detailed operating run plan has been prepared. An in-house meeting was held to discuss the operating schedule and various unit operations..

A dewaxing facility with a capacity of 80 lbs/batch was designed and assembled. About 400 lbs of VSOH from previous bench runs has been gathered and will be dewaxed-hydrotreated during the first week of April.

About 116 kg of Fe-containing catalyst has been prepared and tested to ensure uniformity in composition and activity.

The computer program/database of HTI's bench unit 227 is being updated in order to accommodate the use of oil-agglomerated feed and additional unit operations, e.g. filtration/vacuum still, and solvent dewaxing/hydrotreating. An updated run schedule is attached. Unit start-up is scheduled to be around April 9 to 12.

Sincerely,

Theo L.K. Lee

cc: R. Winschel-CONSOL, Inc  
M. Peluso-LDP Associates  
HTI: D. Tanner, F. Stevens J. Hu

## ALC-01 Run Schedule

| <b>Run Preparation</b>                        | <b>Completion Date</b> |
|---|------------------------|
| Oil-agglomerated coal from CONSOL             | 3/27                   |
| Detailed run plan                             | 3/26                   |
| Catalyst preparation                          | 4/2                    |
| Catalyst inspection & activity test           | 4/2                    |
| Dewaxing (start-up solvent for condition 4&5) | 4/3-9                  |
| Hydrotreating of dewaxed solvent              | 4/6-11                 |
| Unit preparation                              | 4/3-9 or 12            |
| <b>Unit Startup</b>                           | 4/9-12                 |
| <b>Operation</b>                              | 25 days                |
| <b>Unit Shutdown</b>                          | 5/5-8                  |

**SECTION FOUR**

**LDP ASSOCIATES**

# LDP ASSOCIATES

Michael Peluso, Proprietor  
609-586-2301

32 Albert E. Bonacci Dr.  
Hamilton Square, N.J. 08690

May 17, 1996

Dr. Ed Givens  
Center for Applied Energy Research  
3572 Iron Works Pike  
Lexington, Kentucky 40511-8433

Dear Ed:

Subject: QUARTERLY PROGRESS REPORT FOR JAN. THRU MARCH 1996

For the quarter ending March 31, 1995 the following subcontract services (UKRF-4-25582-92-75) were performed in support of the DOE Advanced Concepts Program (DE-AC22-91PC91040):

## **ALC-1 RUN PLAN**

Numerous discussions were held with CONSOL concerning the above run plan. It was decided that in order to independently control both resid and solids recycle quantities, the use of the pressure filter rather than the vacuum still was required. Recycle solvent composition was also revised after reviewing the range of recycle solvent compositions used in recent runs at Wilsonville, HTI and Exxon. The advisability of doing dewaxing was also investigated. Recommendations for changes to the ALC-1 Run Plan, including utilization of the time period originally scheduled for the Column Flotation (Condition #4) portion of the run were sent to CONSOL.

## **WAX CONCENTRATION**

HTI analysis indicated that the wax content of recycle distillate solvent used in their CMSL-9, Condition #6 Run was 5%. An evaluation was conducted to determine if this result was consistent with the 20%+ wax concentration measured for the distillate recycle stream in Wilsonville Run #263J. It was determined that differences in product withdrawals, sampling and the severity of reactor operating conditions between the two runs could account for the differences in the wax content of the recirculating distillate solvent. Furthermore, projected commercial operation at CMSL-9, Condition #6 levels would not preclude the recovery of a significant quantity of wax byproduct.

## **PROCESS SIMULATION**

Discussions were held with Prof. Eric Grulke on input data used by Bechtel in their simulation study. As a result two appendices from the Bechtel Illinois #6 report were forwarded to Grulke. In addition, copies of additional appendices from the same report which were thought to be useful, were requested from Ed Klunder at PETC. Co-ordination of the

Grulke effort with PETC's system group work was also discussed.

### **ECONOMIC ASSESSMENT**

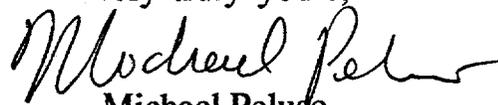
An updated Base Case (W"ville Run #263J) was prepared to account for the allowable lower gasification temperature (2,500°F) and a more accurate C<sub>1</sub> to C<sub>3</sub> gas yield distribution in the upgrading units (hydrotreating, hydrocracking & catalytic reforming). As a result the required gasoline product selling price dropped by \$1.23/Bbl. from the original Base Case estimate. The major flowrates for the updated Base Case are shown in Figure 2.

At the request of PETC economic projections were prepared for the advanced concepts proposed for testing in the first continuous bench-scale run (ALC-1) at HTI. Projections for each of the proposed conditions (#2 thru #5) were made versus the updated Base Case mentioned above. Comparative technical and economic results for the four conditions are shown in Tables 1 thru 12. The implications of these projected differences and the many assumptions upon which the evaluations were based, were discussed in detail at the ALC-1 Run Plan Meeting held in Princeton on January 19.

Based on a short article in the Chemical Marketing Reporter, the firm of Rauch Associates was contacted concerning their newsletter, "Wax Data". A complimentary sample issue of this publication contained useful data on the U. S. paraffin wax market size and pricing.

Climax Molybdenum was contacted regarding the latest pricing for moly compounds to be used as catalysts in this program (e.g. ammonium heptomolybdate, AHM). Climax advised that long term AHM pricing was expected to be in the \$7.50 to \$8.00 range which is only slightly higher than the \$7.30 price quoted by Climax in November 1993.

Very truly yours,

  
Michael Peluso  
LDP Associates

cc: F. Derbyshire @ CAER  
R. Anderson @ CAER  
R. Winschel @ CONSOL  
T. Lee @ HTI

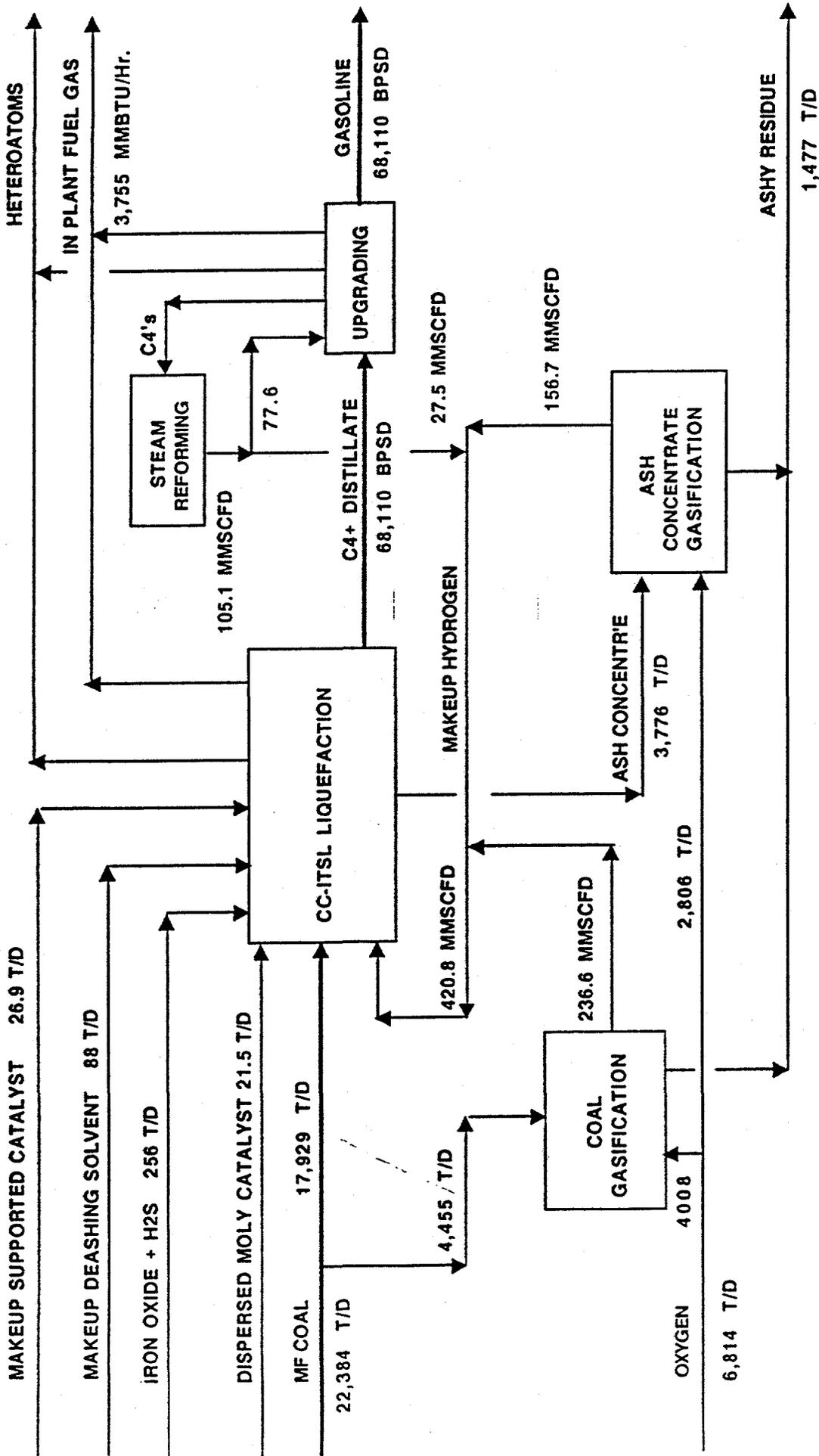


FIGURE 2  
 BLACK THUNDER LIQUEFACTION STUDY - HYBRID MODE  
 SIMPLIFIED BLOCK FLOW DIAGRAM - CONCEPTUAL COMMERCIAL PLANT  
 UPDATED BASE CASE (SO<sub>3</sub>-Free Ash, Wilsonville Run # 263J)

TABLE 1

| TECHNICAL DATA 1 OF 2           | BASE CASE | CONDITION #2 |
|---------------------------------|-----------|--------------|
| ASH in COAL FEED, Wt% MF        | 5.54      | 5.54         |
| % ASH REJECTION IN OIL AGGLOM.  | NOT USED  | 5.0          |
| FRESH IRON OXIDE RATE, Wt.%MF   | 1.00      | SAME         |
| FRESH MOLY RATE, ppm on MF Coal | 1.00      | SAME         |
| RECYCLE SOLVENT/ MF COAL        | 2.333     | SAME         |
| ASH in ASH CONC. Wt.% MF Coal   | 6.57      | 3.80         |
| ASH in RECYCLE SOLVENT, Wt.%MF  | 22.136    | 17.922       |
| RECYCLE MOLY RATE, ppm on MF    | 337       | 472          |
| SO3-Free MAF COAL CONV., WT. %  | 92        | 93.3         |
| IOM in ASH CONC. Wt.% MF Coal   | 7.557     | 6.292        |
| IOM in RECYCLE SOLVENT, Wt.%MF  | 25.463    | 29.677       |
| SOLIDS in RECYCLE SOLV., Wt.%MF | 47.599    | SAME         |
| SOLIDS in ASH CONCEN. Wt%       | 67.08     | SAME         |
| ORGANIC REJECTION, Wt.% MF Coal | 14.490    | 11.245       |

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TABLE 2

| TECHNICAL DATA 2 OF 2                         | BASE CASE | CONDITION #2<br>LOW pH OIL AGGLOM. |
|---|-----------|------------------------------------|
| MF COAL to LIQUEFACTION, T/D                  | 17929     | 17432                              |
| TOTAL MF COAL USED, T/D                       | 22384     | 23219                              |
| C1 - C3/ YIELD in LIQ'N, Wt.% MF              | 7.831     | 8.129                              |
| C4+ DISTILLATE YIELD, Wt.% MF                 | 59.546    | 62.125                             |
| C4+ DISTILLATE/H2 CONSUMED                    | 10.08     | 10.10                              |
| GASOLINE PRODUCT RATE, BPSD                   | 68110     | 71065                              |
| GASOLINE/H2 CONSUMED                          | 7.59      | 7.60                               |
| PREDICTED RELATIVE REACTOR<br>SPACE VELOCITY  | 1.00      | 0.973                              |
| PREDICTED RELATIVE REACTOR<br>VOLUME REQUIRED | 1.00      | 1.019                              |

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TABLE 3

| ECONOMIC DATA                   | BASE CASE | CONDITION #2<br>LOW pH OIL AGGLOM. |
|---------------------------------|-----------|------------------------------------|
| CAPITAL COSTS, \$ Millions      |           |                                    |
| • OIL AGGLOMERATION UNIT        | NOT USED  | + 60                               |
| • PROCESS UNITS                 | BASE      | + 83                               |
| • TOTAL                         | BASE      | + 102                              |
| OPERATING COSTS, \$/Bbl.        |           |                                    |
| • ACID for OIL AGGLOM. UNIT     | NOT USED  | + 0.38                             |
| • DISPERSED IRON CATALYST       | BASE      | - 0.04                             |
| • DISPERSED MOLY CATALYST       | BASE      | - 0.08                             |
| • 2ND STAGE SUPPORTED CATALYST  | BASE      | - 0.78                             |
| • ALL OTHER                     | BASE      | - 0.19                             |
| TOTAL                           | BASE      | - 0.71                             |
| ANNUALIZED CAPITAL COST, \$/Bbl | BASE      | - 0.16                             |
| TOTAL PRODUCT COST, \$/Bbl      | BASE      | - 0.87                             |

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TABLE 4

| TECHNICAL DATA 1 OF 2           | BASE CASE | CONDITION #3<br>LOW pH OIL AGGLOM. |
|---------------------------------|-----------|------------------------------------|
| ASH in COAL FEED, Wt% MF        | 5.54      | 5.54                               |
| % ASH REJECTION IN OIL AGGLOM.  | NOT USED  | 5.0                                |
| FRESH IRON OXIDE RATE, Wt.%MF   | 1.00      | 0.576                              |
| FRESH MOLY RATE, ppm on MF Coal | 100       | 58                                 |
| RECYCLE SOLVENT/ MF COAL        | 2.333     | 2.439                              |
| ASH in ASH CONC. Wt.% MF Coal   | 6.57      | 3.364                              |
| ASH in RECYCLE SOLVENT, Wt.%MF  | 22.136    | SAME                               |
| RECYCLE MOLY RATE, ppm on MF    | 337       | 379                                |
| SO3-Free MAF COAL CONV., WT. %  | 9.2       | 94.2                               |
| IOM in ASH CONC. Wt.% MF Coal   | 7.557     | 5.462                              |
| IOM in RECYCLE SOLVENT, Wt.%MF  | 25.463    | 35.944                             |
| SOLIDS in RECYCLE SOLV., Wt.%MF | 47.599    | 58.080                             |
| SOLIDS in ASH CONCEN. Wt%       | 67.08     | SAME                               |
| ORGANIC REJECTION, Wt.% MF Coal | 14.490    | 9.793                              |

mf  
1/19/96

**TABLE 5**

| TECHNICAL DATA 2 OF 2                         | BASE CASE | CONDITION #3<br>LOW PH OIL AGGLOM. |
|---|-----------|------------------------------------|
| MF COAL to LIQUEFACTION, T/D                  | 17929     | 17432                              |
| TOTAL MF COAL USED, T/D                       | 22384     | 23707                              |
| C1 - C3 YIELD in LIQ'N, Wt.% MF               | 7.831     | 8.265                              |
| C4+ DISTILLATE YIELD, Wt.% MF                 | 59.546    | 63.291                             |
| C4+ DISTILLATE/H2 CONSUMED                    | 10.08     | 10.09                              |
| GASOLINE PRODUCT RATE, BPSD                   | 68110     | 72400                              |
| GASOLINE/H2 CONSUMED                          | 7.59      | 7.59                               |
| PREDICTED RELATIVE REACTOR<br>SPACE VELOCITY  | 1.00      | 0.989                              |
| PREDICTED RELATIVE REACTOR<br>VOLUME REQUIRED | 1.00      | 1.034                              |

*myf*  
11/19/96

TABLE 6

| ECONOMIC DATA                   | BASE CASE | CONDITION #3<br>LOW pH OIL AGGLOM. |
|---------------------------------|-----------|------------------------------------|
| CAPITAL COSTS, \$ Millions      |           |                                    |
| • OIL AGGLOMERATION UNIT        | NOT USED  | + 60                               |
| • PROCESS UNITS                 | BASE      | + 111                              |
| • TOTAL                         | BASE      | + 135                              |
| OPERATING COSTS, \$/Bbl.        |           |                                    |
| • ACID for OIL AGGLOM. UNIT     | NOT USED  | + 0.37                             |
| • DISPERSED IRON CATALYST       | BASE      | - 0.37                             |
| • DISPERSED MOLY CATALYST       | BASE      | - 0.92                             |
| • 2ND STAGE SUPPORT'D CATALYST  | BASE      | - 0.81                             |
| • ALL OTHER                     | BASE      | - 0.29                             |
| TOTAL                           | BASE      | - 2.02                             |
| ANNUALIZED CAPITAL COST, \$/Bbl | BASE      | - 0.45                             |
| TOATAL PRODUCT COST, \$/Bbl     | BASE      | - 2.47                             |

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1/19/96

TABLE 7

| TECHNICAL DATA 1 OF 2           | BASE CASE | CONDITION #4 |
|---------------------------------|-----------|--------------|
| ASH in COAL FEED, Wt% MF        | 5.54      | 5.54         |
| % ASH REJ'N /% ORGANIC RECOV'Y  | NOT USED  | 57/77.2      |
| FRESH IRON OXIDE RATE, Wt.%MF   | 1.00      | SAME         |
| FRESH MOLY RATE,ppm on MF Coal  | 100       | SAME         |
| RECYCLE SOLVENT/MF COAL         | 2.333     | SAME         |
| ASH in ASH CONC. Wt.% MF Coal   | 6.57      | 4.219        |
| ASH in RECYCLE SOLVENT,Wt.%MF   | 22.136    | 18.693       |
| RECYCLE MOLY RATE,ppm on MF     | 337       | 443          |
| SO3-Free MAF COAL CONV., WT.%   | 92        | 93.1         |
| IOM in ASH CONC. Wt.% MF Coal   | 7.557     | 6.524        |
| IOM in RECYCLE SOLVENT,Wt.%MF   | 25.463    | 28.906       |
| SOLIDS in RECYCLE SOLV.,Wt.%MF  | 47.599    | SAME         |
| SOLIDS in ASH CONCEN. Wt%       | 67.08     | SAME         |
| ORGANIC REJECTION, Wt.% MF Coal | 14.490    | 11.796       |

mf  
1/19/94

TABLE 8

| TECHNICAL DATA 2 OF 2                         | BASE CASE | CONDITION #4<br>COLUMN FLOTATION |
|---|-----------|----------------------------------|
| MF COAL to LIQUEFACTION, T/D                  | 17929     | 17495                            |
| TOTAL MF COAL USED, T/D                       | 22384     | 23335                            |
| C1 - C3/ YIELD in LIQ'N, Wt.% MF              | 7.831     | 8.309                            |
| C4+ DISTILLATE YIELD, Wt.% MF                 | 59.546    | 63.663                           |
| C4+ DISTILLATE/H2 CONSUMED                    | 10.08     | 10.09                            |
| GASOLINE PRODUCT RATE, BPSD                   | 68110     | 71065                            |
| GASOLINE/H2 CONSUMED                          | 7.59      | 7.59                             |
| PREDICTED RELATIVE REACTOR<br>SPACE VELOCITY  | 1.00      | 0.977                            |
| PREDICTED RELATIVE REACTOR<br>VOLUME REQUIRED | 1.00      | 0.998                            |

*mf*  
1/17/96

TABLE 9

| ECONOMIC DATA                   | BASE CASE | CONDITION #4<br>COLUMN FLOTATION |
|---------------------------------|-----------|----------------------------------|
| CAPITAL COSTS, \$ Millions      |           |                                  |
| • COLUMN FLOTATION UNIT         | NOT USED  | + 90                             |
| • PROCESS UNITS                 | BASE      | + 127                            |
| • TOTAL                         | BASE      | + 153                            |
| OPERATING COSTS, \$/Bbl.        |           |                                  |
| • CHEMICALS for COL. FLOAT UNIT | NOT USED  | + 0.28                           |
| • DISPERSED IRON CATALYST       | BASE      | - 0.03                           |
| • DISPERSED MOLY CATALYST       | BASE      | - 0.09                           |
| • 2ND STAGE SUPPORTED CATALYST  | BASE      | - 0.10                           |
| • ALL OTHER                     | BASE      | - 0.04                           |
| TOTAL                           | BASE      | + 0.02                           |
| ANNUALIZED CAPITAL COST, \$/Bbl | BASE      | + 0.24                           |
| TOTAL PRODUCT COST, \$/Bbl      | BASE      | + 0.26                           |

*wpf*  
1/19/86

TABLE 10

| TECHNICAL DATA 1 OF 2            | BASE CASE | CONDITION #5<br>DEWAXING & HT |
|----------------------------------|-----------|-------------------------------|
| ASH in COAL FEED, Wt% MF         | 5.54      | 5.54                          |
| DISTILLATE in RECYCLE SOLV, Wt.% | 91.23     | 70.56                         |
| FRESH IRON OXIDE RATE, Wt.%MF    | 1.00      | 0.827                         |
| FRESH MOLY RATE, ppm on MF Coal  | 100       | 83                            |
| RECYCLE SOLVENT/MF COAL          | 2.333     | 2.128                         |
| ASH in ASH CONC. Wt.% MF Coal    | 6.57      | 6.392                         |
| ASH in RECYCLE SOLVENT, Wt.%MF   | 22.136    | 27.373                        |
| RECYCLE MOLY RATE, ppm on MF     | 337       | 354                           |
| SO3-Free MAF COAL CONV., WT. %   | 9.2       | 95.0                          |
| IOM in ASH CONC. Wt.% MF Coal    | 7.557     | 4.723                         |
| IOM in RECYCLE SOLVENT, Wt.%MF   | 25.463    | 20.226                        |
| SOLIDS in RECYCLE SOLV., Wt.%MF  | 47.599    | SAME                          |
| SOLIDS in ASH CONCEN. Wt%        | 67.08     | SAME                          |
| ORGANIC REJECTION, Wt.% MF Coal  | 14.490    | 10.178                        |

Ref 1/19/96

TABLE 11

| TECHNICAL DATA 2 OF 2                         | BASE CASE | CONDITION #5<br>DEWAXING & HT |
|---|-----------|-------------------------------|
| MF COAL to LIQUEFACTION, T/D                  | 17929     | SAME                          |
| TOTAL MF COAL USED, T/D                       | 22384     | 23931                         |
| C1 - C3 YIELD in LIQ'N, Wt.% MF               | 7.831     | 7.262                         |
| C4+ DISTILLATE YIELD, Wt.% MF                 | 59.546    | 63.931                        |
| C4+ DISTILLATE/H2 CONSUMED                    | 10.08     | 10.31                         |
| GASOLINE PRODUCT RATE, BPSD                   | 68110     | 71065                         |
| WAX PRODUCT RATE, T/D                         | NONE      | 537                           |
| PRODUCTS/H2 CONSUMED                          | 7.59      | 7.74                          |
| PREDICTED RELATIVE REACTOR<br>SPACE VELOCITY  | 1.00      | 0.895                         |
| PREDICTED RELATIVE REACTOR<br>VOLUME REQUIRED | 1.00      | 1.048                         |

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TABLE 12

| ECONOMIC DATA                   | BASE CASE | CONDITION #5<br>DEWAXING & HT |
|---------------------------------|-----------|-------------------------------|
| CAPITAL COSTS, \$ Millions      |           |                               |
| • EXTRACTION, DEWAX & HT UNITS  | NOT USED  | + 376                         |
| • PROCESS UNITS                 | BASE      | + 391                         |
| • TOTAL                         | BASE      | + 457                         |
| OPERATING COSTS, \$/Bbl.        |           |                               |
| • CHEMICALS for EXT, DW&HT UNIT | NOT USED  | + 0.18                        |
| • DISPERSED IRON CATALYST       | BASE      | - 0.15                        |
| • DISPERSED MOLY CATALYST       | BASE      | - 0.39                        |
| • 2ND STAGE SUPPORT'D CATALYST  | BASE      | - 0.06                        |
| • ALL OTHER                     | BASE      | + 0.39                        |
| • CREDIT for WAX BYPRODUCT      | BASE      | - 5.24                        |
| TOTAL                           | BASE      | - 5.27                        |
| ANNUALIZED CAPITAL COST, \$/Bbl | BASE      | + 3.19                        |
| TOATAL PRODUCT COST, \$/Bbl     | BASE      | - 2.08                        |

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