

EDS COAL LIOUEFACTION PROCESS DEVELOPMENT,  
PHASE IV ,

Annual Technical Progress Report,  
~~for the Period~~  
July 1, 1978 - June 30, 1979

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EDS COAL LIQUEFACTION PROCESS DEVELOPMENT - PHASE IV  
ANNUAL TECHNICAL PROGRESS REPORT FOR THE PERIOD  
JULY 1, 1978 - JUNE 30, 1979

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ABSTRACT

This report is the second Annual Technical Progress Report for U.S. Department of Energy Cooperative Agreement No. EF-77-A-01-2893 for Exxon Donor Solvent (EDS) Coal Liquefaction Process Development - Phase IV. This report covers the period July 1, 1978 through June 30, 1979. Funding is shared by U.S. Department of Energy, The Carter Oil Company (a subsidiary of Exxon Corporation), Electric Power Research Institute, Japan Coal Liquefaction Development Company, Phillips Petroleum Company, Atlantic Richfield Company, and Ruhrkohle A.G. The agreement covers the period January 1, 1977 through December 31, 1982. The Laboratory Process Research and Development studies were conducted at various Exxon Research and Engineering Co. (ER&E) facilities: Research and Development Division at Baytown, Texas; Products Research Division at Linden, New Jersey; and the Exxon Research and Development Laboratories at Baton Rouge, Louisiana. The Engineering Research and Development studies were performed at the Synthetic Fuels Engineering and Exxon Engineering Technology Departments of ER&E at Florham Park, New Jersey. The information dealing with the Management, Detailed Engineering, and Procurement and Construction of the 250 T/D Exxon Coal Liquefaction Pilot Plant (ECLP) was generated at The Carter Oil Company, Houston, Texas, and Exxon Engineering - Project Management Department of ER&E, Florham Park, New Jersey.

Highlights from this report are summarized as follows. They are grouped according to their corresponding reporting categories in the report.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT  
LIQUEFACTION PROCESS RESEARCH

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1. OPERATION OF RECYCLE COAL LIQUEFACTION UNITS (RCLU)

- Screening studies were completed on four EDS sponsor-selected coals in the 50 pound-per-day Recycle Coal Liquefaction Unit (RCLU-1). The purpose of these screening studies was to determine the response of these coals in the EDS liquefaction process. The four coals investigated were a Pittsburgh seam bituminous coal from the Ireland mine in West Virginia, a Texas lignite from the Big Brown mine, an Illinois No. 6 bituminous coal from the Burning Star No. 2 mine, and an Australian black coal from the Wandoan area of Queensland.

Yield response to liquefaction variables, such as temperature and residence time, varied considerably and did not appear to be directly related to coal rank. Unit operability and solids deposition in the liquefaction reactor were found to be dependent on coal

rank. In general, the higher rank coals are easier to process in RCLU because of a lower viscosity bottoms product and a lower solids deposition rate during liquefaction.

- Sulfur dioxide ( $\text{SO}_2$ )/air pretreatment of Wyoming coal from the Wyodak mine was successful in significantly reducing calcium carbonate ( $\text{CaCO}_3$ ) scale formation during liquefaction in RCLU-1. Reactor scale formation was reduced over one hundred fold compared to that from untreated Wyodak coal to less than 0.001 lbs per ton of coal fed. A similar decrease in  $\text{CaCO}_3$  content of the free-flowing reactor solids was found. The pretreatment had no detrimental effects on conversion or liquid yield compared to untreated coal.
- Two liquefaction bottoms recycle studies were conducted in RCLU-1. The purpose of these studies was to evaluate the effect of bottoms recycle on Exxon Donor Solvent (EDS) liquefaction yields for Illinois No. 6 bituminous coal from the Monterey No. 1 mine and Wyoming subbituminous coal from the Wyodak mine. Vacuum bottoms from the one ton-per-day Coal Liquefaction Pilot Plant (CLPP) were fed with fresh coal to RCLU in a once-through mode to simulate recycle bottoms feed.

At 840°F, 1500 psig, 1.6 solvent-to-coal ratio and 40 minutes slurry residence time, variation of the bottoms-to-coal ratio over the range of 1:3 to 1:1 produced little variation in conversion of the bottoms in liquefaction.

Wyodak bottoms recycle required more solvent than Monterey bottoms recycle for bottoms conversion (2.4 versus 1.6 solvent-to-coal). At 2.4/1/0.5 solvent-to-coal-to-bottoms, Wyodak bottoms conversion and  $\text{C}_4$ -1000°F liquid yields were between 25-30 wt % and 22-25 wt % on 1000°F+ bottoms fed, respectively, at 840°F, 1500 psig, and 25 to 60 minute residence times. Little bottoms conversion was observed at 1.6/1/0.5 solvent-to-coal-to-bottoms.

## 2. OPERATION OF THE ONE-TON-PER-DAY COAL LIQUEFACTION PILOT PLANT (CLPP)

- Scheduled operations were completed on Wyoming subbituminous coal from the Wyodak mine during the last quarter of 1978 on the one ton-per-day Coal Liquefaction Pilot Plant (CLPP). Data were supplied from Wyodak operations to the EDS Liquefaction Engineering Division (ELED) for use in the Wyoming Coal Study Design.
- Operability on Wyoming subbituminous coal was improved by solids withdrawal on the CLPP. A withdrawal rate of approximately four weight percent on coal was sufficient to control reactor solids accumulation. Operability in the high pressure gas scrubbing system was also improved by controlling ammonium carbonate salts.



- Equipment for vacuum bottoms recycle was installed on the CLPP during the first quarter of 1979. In addition, the coal feed system was modified to give better coal feed rate controllability.
- Vacuum bottoms recycle was operated for over 550 hours with bottoms containing as low as 2-3% 1000°F-. Yield data from bottoms recycle show improved conversions compared to coal-only operations but below those expected based on tubing bomb and RCLU simulated operations.

### 3. SOLVENT HYDROGENATION STUDIES

- Additional data on the effect of carbon oxide impurities were obtained from a Ni/Mo-10 catalyst activity study. Results agree with previous findings indicating that carbon oxide impurities in solvent hydrogenation treat gas do not affect hydrogenation activity. The major effects are a slight reduction in desulfurization and denitrogenation activities.
- Solvent hydrogenation catalyst activity studies to confirm earlier observed effects attributed to feed heteroatom (sulfur, nitrogen, and oxygen) content were completed. These effects, with both Illinois No. 6 and Wyoming coal solvents over Ni/Mo-10 catalyst, were due to high initial catalyst activities and not to the lower Wyoming solvent heteroatom content. Over the ranges tested, the effects of the heteroatoms on the activity of both Ni/Mo-10 and Co/Mo-20 catalysts were negligible.
- Hydrotreating EDS spent solvent and heavy product naphtha together was demonstrated to be a feasible alternative to processing these liquids separately.

### 4. BENCH UNITS, MODELING AND CORRELATIONS STUDIES

- A bench scale kinetic study of the SO<sub>2</sub> pretreatment of Wyoming coal was conducted. It was concluded that mass transfer through the coal pores is the overall rate limiting step.
- The effectiveness of gaseous SO<sub>2</sub> pretreatment of Wyoming coal to prevent CaCO<sub>3</sub> formation during liquefaction was tested in a batch fluidized mode. This mode of treatment was observed to be effective for both Wyoming subbituminous coal and Texas Big Brown lignite. Similar pretreatment of Wyoming coal was also carried out at higher than room temperature to simulate the expected commercial unit pretreatment temperature in the fluidized bed. In this temperature range, the pretreatment effectiveness, expressed as wt % CaCO<sub>3</sub> in tubing bomb liquefaction residue ash, is somewhat lower than that at room temperature (75°F).

- The Once-Through Coal Liquefaction Unit (OTCLU) feeding Illinois No.6, Monterey No. 1 mine coal showed good agreement with RCLU yields at one operating condition (840°F/40 minutes). Validation of the unit, previously used only for operability studies, at other operating conditions and with other coals is in progress.
- Tubing bomb liquefactions were carried out on three samples of Illinois No. 6 (Burning Star No. 2) coal obtained by the Electric Power Research Institute (EPRI) from Hydrocarbon Research, Inc. The same conversion at 840°F/40 minutes was observed with this coal as with the sample currently used in the RCLU coal screening studies.
- Tubing bomb simulations of bottoms recycle for program and sponsor coals indicate a yield incentive exists for operating the EDS process in a bottoms recycle mode with each of the coals investigated. Approximately 22% of the 1000°F+ bottoms derived from bituminous coals and Wandoan subbituminous coal could be converted at 840°F and 40 minutes reaction time. Coal and bottoms showed a synergism for conversion with Wyodak subbituminous coal and Texas Big Brown lignite. The amount of synergism displayed by Wyodak coal and bottoms was affected strongly by the solvent-to-solids ratio.
- Fit of the first-pass fundamental model for coal liquefaction to its data base on Monterey coal was achieved. Final parameter values were employed to simulate RCLU yield periods not included in the data base. Good agreement between experimental data and model predictions served to further confirm the applicability of the model. The model was used to simulate RCLU bottoms recycle runs and predicted overall C<sub>1</sub>-1000°F hydrocarbon yields within 3% of the measured values. Process variable simulations covering a broad range of reaction temperatures, residence times and bottoms recycle ratios were conducted which indicate these variables have a strong influence on C<sub>1</sub>-1000°F yield and on hydrocarbon product distributions.
- The updated version of the fundamental recycle solvent hydrogenation model was completed. This involved addition of Ni/Mo-10 catalyst kinetics for heteroatom removal and an aging function for heteroatom removal. Simulations were carried out to predict product donor hydrogen and catalyst activity for different operating conditions using Ni/Mo-10 catalyst and a target of one year catalyst life. Model predictions showed that the target donor hydrogen levels could be achieved and maintained.

## 5. PRODUCT QUALITY STUDIES

- Studies with Illinois (Monterey mine) and Wyoming (Wyodak mine) coal naphthas showed that catalytic reforming feed specifications can be met by hydrotreating the coal naphthas in a conventional, single-stage hydrotreater.
- Caustic washed naphthas (to remove phenolic compounds) require lower hydrotreating severity, approximately a two-fold increase in space velocity, and lower hydrogen consumption, about 50% reduction, to meet reformer feed specifications.
- Bench scale studies indicate that oxygenated solvents (e.g. methanol/water blends) can be used to remove phenolic compounds from EDS naphthas. Potential advantages, relative to caustic washing, include the elimination of reagent consumption and solids disposal associated with caustic extraction.
- Analytical characterization and storage stability testing of Wyoming (Wyodak mine) coal liquid products were completed. Measurements of viscosity, Conradson carbon and sediment versus storage time at 150 and 210°F indicated that these coal liquids are comparable in stability to petroleum-derived reference materials.
- Combustion tests with Wyoming (Wyodak mine) 350/1000°F and 350°F<sup>+</sup> (including coker liquids) fuel oils in a 50 HP Cleaver-Brooks boiler revealed burning characteristics similar to fuel oils derived from Illinois coal, namely, low smoke and particulate emissions, but high NO<sub>x</sub> emissions consistent with high levels of fuel-bound nitrogen.
- Hydrotreating studies at the Exxon Research and Development Laboratories (Baton Rouge) were completed with Illinois (Monterey mine) and Wyoming (Wyodak mine) fuel oils, with and without coker liquids. The purpose of these studies was to define the severity and hydrogen uptake required to achieve product nitrogen targets of 0.5 and 0.2 weight percent.
- A major objective of the 1979 product quality program is to issue an interim report containing an initial assessment of preferred EDS products markets considering handling, storage, shipment, health and environment.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT  
BOTTOMS PROCESSING RESEARCH

6. COKING AND GASIFICATION STUDIES

8. SUPPLEMENTAL FLEXICOKING\* PROGRAM-Part I

- A study of the effect of dilution on the viscosity and stability of coal liquefaction bottoms was completed. Dilution of the bottoms with 1000°F<sup>-</sup> coal derived liquids reduces the initial viscosity and increases the storage stability of both Wyodak and Illinois bottoms. Data on initial viscosities of Wyodak bottoms was correlated using multiple linear regression with 1000°F<sup>-</sup> content and liquefaction severity as the independent variables.
- Viscosity measurements of the Illinois liquefaction bottoms from the liquefaction of mixtures of recycle bottoms with coal show that the initial viscosity and the thermal stability were similar to bottoms obtained from single-pass liquefaction.
- The viscosity and storage stability for two Wyoming (Wyodak) bottoms from CLPP were measured at 600°F. The initial viscosities for these Wyodak bottoms, which had low 1000°F<sup>-</sup> contents, averaged 28 poise. After 8 hours storage at 600°F, the average viscosity increased to 40 poise.
- A study of the properties of ground bottoms/solvent slurry feeds indicated that such a feed system would not be feasible for the FLEXICOKING prototype. Bottoms mixtures with raw creosote oil formed settling suspensions that would require high agitation rates and transport velocities.
- Bench coking studies of Wyodak bottoms from 1978 CLPP yield periods 264 through 284 were completed. These data give an estimate of the coke, liquid and gas yields from coking of the bottoms at base conditions. The data are used for guidance in further coking studies.
- Coking runs were made on the Continuous Stirred Coking Unit (CSCU) using heavy Wyodak coker liquids (837°F<sup>+</sup>) from the Large Stirred Coking Unit (LSCU) to simulate recycle coking operations. No evidence of production of 1000°F<sup>-</sup> liquids was found.
- An initial CSCU screening study with Big Brown coal liquefaction bottoms was completed. The net liquid yields from these Texas lignite bottoms are similar to those obtained previously for Wyodak bottoms.

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- Seven coking yield runs on coal liquefaction bottoms from Illinois No. 6 Burning Star coal were completed in the CSCU. The bottoms came from RCLU-1 operations at two conditions of liquefaction temperature and residence time. Coker liquid yields from bottoms produced at liquefaction conditions of 840°F and 40 minutes residence were higher than from 880°F/25 minute bottoms (18.6 and 10.2 wt % on dry coal, respectively). The overall total yields (liquefaction plus coking) were 42.7% and 40.6%. For both samples, an increase of coking temperature caused a decrease of liquid yield.
- Six CSCU runs were made with RCLU bottoms produced from Pittsburgh No. 8 Arkwright coal at two different liquefaction residence times. The coking yield structure over a range of coking temperatures was found to be similar to that of bottoms from Illinois No. 6 (Monterey) coal.
- Four CSCU runs were made with Wandoan coal bottoms from 840°F-40 minute RCLU liquefaction. At base coking temperature, coking liquid was estimated to contribute one-fourth of the total EDS liquid yield.
- A listing of the accumulated data on coking yields from the several coal liquefaction bottoms that were run in the CSCU reveal definite trends with coal rank. Liquid yields increase and gas yields decrease as the coal rank increases. Evidence of coking yield dependence on liquefaction conditions was observed for all types of coal liquefaction bottoms processed in the CSCU.
- CSCU liquid yields from the bottoms of liquefaction operations with mixtures of Illinois coal and recycled bottoms are very similar to those from regular Illinois coal liquefaction bottoms.
- One CSCU run was performed with a mixture of 70% Illinois bottoms and 30% vacuum gas oil. The CSCU conditions were similar to those expected on the FLEXICOKING process prototype. The results indicate that the liquid yield is the same as would be predicted from simple combination of VGO recovery plus normal coking yield.
- Three runs were made in the CSCU using ground Illinois No. 6 coal feed. Pyrolysis yields for these coals agreed well with the Fischer Assay yields. Two CSCU runs were made with a mixture of bottoms and coal. The yields were the same as would be predicted from a combination of the yields from separate coking and pyrolysis.
- Coker liquids from Illinois No. 6 Burning Star bottoms were characterized by benzene and pyridine extractions to determine the nature of the recovered liquids as a function of coking temperature. As coking temperature increased, the net 1000°F<sup>-</sup> asphaltenes increased up to RV+150°F and then decreased. This suggests that cracking of the 1000°F<sup>-</sup> asphaltenes takes place above RV+150°F.

- The Large Stirred Coking Unit (LSCU) was revamped and was operating to produce coker liquid samples for various program studies such as hydroclone tests with heavy coker liquids and combustion tests with a full-range 350°F<sup>+</sup> EDS product blend that includes coker liquid. Operations were satisfactory with both Illinois and Wyodak bottoms feeds.
- A bottoms remelt study was conducted to determine physical property data for coal liquefaction bottoms. The objective of the study was to confirm models for the mixing and pumping of non-Newtonian bottoms blends in order to provide tighter design of the feed preparation system for the FLEXICOKING prototype. Calculated heat transfer coefficients were higher than anticipated. Power consumption for mixing was low with rapid transient approach to steady-state torque. No evidence of high mixer torque or pressure impulse on start-up was noted.
- Steam and carbon dioxide gasification of IKG Illinois coke (bottoms derived from Monterey mine coal) was studied in the development of the air FLEXICOKING process for EDS bottoms. Preliminary data analysis shows a Langmuir-type rate expression may be used to correlate mild reactant inhibition although this inhibition can be neglected in short-cut calculations. The coke gasification thermal response agrees with literature values.
- Bench scale coke gasification studies of ash fusion conducted in the Small Fluidized Bed Unit (SFU) showed similar sintering behavior for Wyoming and Illinois cokes. A non-agglomerating region was determined by testing the process variables of superficial velocity, temperature, and steam/air ratio. The sintering effects were found to apply at partial gasification as well as at complete gasification. The quartz reactor used in the SFU was modified to alleviate excessive particle entrainment experienced during coke gasification runs. The addition of an enlarged disengaging section eliminated the problem.
- There was evidence from both the SFU and the Fluidized Bed Unit (FBU) that smaller coke particles were more prone to form sinter than larger particles.
- Particle size measurements at various levels of coke gasification in the SFU indicated that gasification causes gradual shrinking of the coke particles until the ash content reaches 75% where no further shrinkage occurs. The final average particle size was 80% of the initial particle size, even though all that remained was ash. The particle size shrinkage was similar for air, steam or air-steam mixtures.
- SFU tests with coke from a North Dakota lignite showed that it was much more prone to sinter than were Illinois or Wyodak cokes. The cause was attributed to a high level of sodium (10%) in the ash.

- Cold model studies with horizontal jet nozzles in models of the FBU and IKG gasifier reactors indicated that there was stagnant coke in the region of the air/steam inlet jets. This stagnant coke is thought to have been a contributing factor in the formation of sinter. These studies demonstrated that stagnant coke could be eliminated by the use of a properly designed conical distributor.
- Sinter was formed in FBU coke gasification runs at operating conditions similar to the nominal conditions in the IKG when sinter was formed. Temperature instabilities during coke gasification at low superficial velocities in the FBU were alleviated by operating at higher superficial velocities. Sintered agglomerates were formed over a range of superficial velocities with Illinois coke using a horizontal jet gas distributor. The sintering was attributed to stagnant coke near the gas inlet jets. Sintering was reduced by the use of a conical gas distributor which eliminated the stagnant coke in the FBU.
- Coke gasification studies on the Fluidized Bed Unit (FBU) provided evidence of the mechanism of sinter formation in stagnant zones. The FBU sinter is formed after all the carbon in the fluidized coke is consumed. Then, oxygen diffuses into the stagnant coke and the heat of reaction causes an increase in temperature. The FBU sinter formation appears to be caused by undissipated heat generated in the stagnant zone of coke. Elimination of the stagnant coke would likely eliminate the FBU sinter formation at target operating conditions.
- The amount of fines produced from FBU gasification of Wyodak coke was the same as for Illinois coke. Also, FBU experiments with both Illinois and Wyodak cokes showed no apparent effect of gasification temperature on fines make. Ash enrichment of the Wyodak fines was lower than for Illinois fines under similar gasification conditions. Because of the relatively low carbon content of the FBU fines from EDS cokes, the carbon rejection with the fines does not appear to be excessive.
- Coke gasification studies in the Fluidized Bed Unit (FBU) confirmed earlier indications that attrition in the overhead cyclone produced roughly half of the coke fines observed in the FBU.

## 7. CONSTRUCTION AND OPERATION OF THE 2 B/D COKING/GASIFICATION PILOT PLANT

- The Integrated Coking/Gasification Pilot Plant (IKG) was used in the development of air FLEXICOKING for EDS bottoms processing. Work this year was analysis of Illinois and Wyodak operations conducted and reported last year. Material and ash balances for the second quarter 1978 operations were much improved over previous EDS work. This enhanced the confidence in IKG data. Heater-gasifier fines production for Wyodak operation were higher than

for Illinois, but not limiting to desired gasification. Reactor fines production was unexpectedly high for Wyodak operation and is still under study in bench work. Wyodak coke was more active toward gasification than Illinois. This enabled lowering gasification temperatures about 50°F and which in turn helped avoid Wyodak coke sintering.

ENGINEERING RESEARCH AND DEVELOPMENT  
PROCESS ENGINEERING AND COST EVALUATIONS

1. COMMERCIAL STUDY DESIGN AND COST ESTIMATE

- Preliminary economics for the Illinois Coal Study Design Update Base Case and estimates of the Market Sensitivity Case were completed. The economics were developed for both 3Q87 startup (SDU Basis) and 1Q85 startup (for comparison with the 1975/76 Study Design). The total erected cost (TEC) for a plant with the Base Case configuration (steam reforming/FLEXICOKING) is estimated to be 3050 M\$ (instant plant, 4Q1978 \$) including an 8.1% process development allowance (PDA) and 25% project contingency. The estimated project cost for a 1Q1985 startup is 4035 M\$, and 4780 M\$ for a 3Q1987 startup. Based on Process Alternatives Model (PAM) screening studies, the TEC for the Market Sensitivity Case is currently assumed to be nominally 5% less than the Base Case. Required Initial Selling Prices (RISP) of coal liquids were developed over a range of effective annual differential escalation rates for product values relative to coal and operating costs.
- Thermal efficiency calculations for the Illinois Study Design Update base case were completed. Overall plant thermal efficiency is 55.6%. Additional efforts involved reconciliations of the calculated value with the 1975/76 Study Design value and identification of thermal loss locations and quantities.
- The overall plant thermal efficiency for the Illinois Market Sensitivity case is 63.6% as compared to 55.6% for the base case. This increase in efficiency reflects the recovery of C<sub>2</sub>- high Btu gas and C<sub>3</sub> LPG as products.
- Revision of the onsite design basis for the Illinois Coal Base Case was completed. The revision reflects changes in the onsite facilities which occurred during the design phase. Work was also completed on the onsite design for the Base Case. Design information packages were prepared and released to cost estimating. Final documentation of the Onsite Design Basis and the onsite design is in progress.
- The fuel system study for the Illinois Coal Base Case was completed. This study defined the basis to be used in designing a safe, operable fuel system for the plant.



- Work was completed in defining the offsite design basis for the Illinois Coal Base Case. The offsite facilities include all utilities, the fuel system, product and intermediate storage facilities, product shipping, coal transportation and preparation, wastewater treating, solid waste disposal facilities and the safety facilities for the plant. The offsite design for the plant Base Case was also completed. Design information for all the offsite facilities was prepared and released to cost estimating. The design information reflects the most restrictive operating mode of the plant.
- Results of an assessment of the air pollution impact of the Illinois Coal Base Case indicate that the plant could be located in a Class II attainment area having the typical meteorological conditions of southwestern Illinois. Location of the plant in a nonattainment area would require off-setting reductions in emissions. Results also indicate that the plant would be defined as a major pollutant source since the emission rates for all the criteria pollutants significantly exceed 100 tons per year.
- The onsite design basis for the Illinois Coal Market Flexibility Sensitivity Case was completed. Major differences in the onsite facilities are a result of substituting partial oxidation of vacuum bottoms for steam reforming of  $C_1/C_3$  as the hydrogen generation process. This change also releases the  $C_1/C_2$  for sales as high Btu gas. Work was also completed on the onsite design. Efforts were concentrated in designing the sections with major changes from the Illinois Coal Base Case. Design information packages were prepared and released to cost estimating.
- The offsite design basis and the offsite design for the Illinois Coal Market Flexibility Sensitivity Case were completed. This offsite design is in considerably less depth and detail than for the Illinois Coal Base Case. Whenever possible, the Base Case design was used. Efforts were concentrated on the fuel/steam/ power system, the water systems and solid waste disposal. Design information packages for the offsite facilities were prepared and released to cost estimating.
- Work was completed on the Onsite Design Basis for the Wyoming Coal Case. The process configuration for this case is identical to that of the Illinois Coal Market Flexibility Sensitivity Case, (i.e., partial oxidation of vacuum bottoms for hydrogen production and FLEXICOKING of the remainder of the vacuum bottoms for plant fuel production). The liquefaction conditions selected are a reactor temperature of 840°F, a residence time of 60 minutes, and a solvent-to-coal ratio of 1.6.
- The onsite design for the Wyoming Coal Case is underway. Initial efforts concentrated on generating heat and material balances for the plant. These balances will be the basis for equipment design. Preparation of the heat and material balances included the development of liquefaction yields and solvent hydrogenation yields

(at SOR and EOR). An effort was also made in improving the representation of the heavier portions of the process streams.

- Design sensitivity cases to examine process uncertainties and potential improvements for the FLEXICOKING unit in the Illinois Coal Base Case were prepared and cost estimated. The major improvement was in coke handling where potential savings approaching 10% of FLEXICOKING investment cost were identified.
- The maximum feasible vacuum pipestill (VPS) cutpoint for feeding partial oxidation for hydrogen production in the Wyoming Coal Case was identified to be 900°F. The cutpoint was reduced from the 975°F cutpoint used with Illinois coal due to the higher viscosity of Wyoming vacuum bottoms and its effect on pump performance.

## 2. COST REDUCTION AND LABORATORY GUIDANCE STUDIES

- An engineering screening study based on RCLU data on Illinois No. 6 (Monterey No. 1 mine) coal indicated a potential economic incentive for Illinois No. 6 coal for vacuum bottoms recycle to the liquefaction reactor. The potential incentive of 1-2 \$/B (2-5% on RISP 1985\$), can be achieved using ~40% bottoms recycle (defined as total bottoms plus coal feed to liquefaction) and various H<sub>2</sub>/fuel alternatives. The choice of configuration may depend on the specific coal feed and the relative stage of development of the bottoms processes. CLPP runs are underway to verify yields and hydrogen consumption during equilibrium operation.
- An engineering screening study on Wyoming (Wyodak) coal showed incentives for operating at more severe liquefaction conditions than were used in the 1975/1976 Wyoming Coal Study Design. The results of this study were used to form the basis for selecting liquefaction conditions in the Wyoming Coal Study Design Update case.
- Preliminary results were obtained from the Process Alternatives (LP) Model (PAM) evaluation of West Virginia (Pittsburgh No. 8) coal, the first sponsor coal. Results showed the most attractive cases utilize once-through FLEXICOKING for fuel/partial oxidation for hydrogen and liquefaction conditions at 840°F/60-100 minutes residence time. An economic comparison with Illinois No. 6 (Monterey) coal at 840°F/40 minutes with the same configuration showed Pittsburgh No. 8 coal to have a small potential economic advantage.
- An engineering process variables screening study was completed for Big Brown lignite, the second sponsor coal. Results showed the most attractive cases utilize once-through FLEXICOKING for fuel/partial oxidation for hydrogen, and liquefaction conditions of 840°F/25-60 minutes residence time. An economic comparison of Big Brown with Illinois No. 6 Monterey (840°F/40 minutes) and Wyodak coal (840°F/60 minutes) cases with the same configuration shows that the Illinois No. 6 Monterey coal plant in Illinois has about a 20 M\$/yr (1985\$)

advantage over the Big Brown plant in Texas and approximately a 70 M\$/yr (1985\$) advantage over a Wyodak coal plant in Wyoming when considered on a consistent economic basis and estimated location effects are included.

- Work began on an engineering screening study aimed at evaluating the economics of a coal liquefaction plant feeding Burning Star coal, the third sponsor coal, run in the Recycle Coal Liquefaction Unit (RCLU) as part of the overall sponsor coal screening program. Various liquefaction temperatures/residence time severities will be evaluated with different bottoms processing configurations. Laboratory data on the FLUID COKING response of Burning Star vacuum bottoms indicate an increased liquid recovery for Burning Star compared to Monterey No. 1 vacuum bottoms.
- An evaluation of alternative slop disposal methods in ECLP was completed. Two viable schemes of reprocessing slop in available facilities during scheduled unit downtime were identified. As a result, separate slop processing facilities, which would cost approximately 2 M\$, are not needed.
- An overall process using a methanol/water extraction for recovery of phenols from EDS naphtha is being evaluated. Based on batch extraction data, a 60 wt% methanol in water solvent and a 0.5/1.0 solvent/feed ratio was selected as being close to the optimal balance of phenol capacity and selectivity. The proposed flow scheme includes the following units: naphtha splitting, phenolic extraction, methanol recovery, phenolic concentration and phenol splitting.
- A Data Reconciliation System (DRS) model for handling ECLP process data is under development. The DRS model accomplishes reconciliation with a least squares analysis of errors that is based on the reliability (standard deviation) of each flow or analytical measurements used in generating material balances.
- Control of calcium carbonate deposition in liquefaction reactors with high-calcium coals was examined from two directions: minimizing adverse effects of the calcium carbonate once it forms (mechanical treatment) and chemical feed pretreatment to prevent calcium carbonate formation. The mechanical approach is potentially lower cost and is the preferred alternative. Confirmation in ECLP is required for the mechanical approach.
- Definitive planning of the ECLP Test Program began. Three plans are being developed: nominal 2 1/2-year test program; one-year test programs; 3-month roll-over test programs.

### 3. ENGINEERING STUDIES OF BOTTOMS PROCESSING/HYDROGEN MANUFACTURE

#### 8. SUPPLEMENTAL FLEXICOKING PROGRAM - Part I

- A bottoms process screening evaluation for the Wyoming Coal Study Design Update indicated that FLEXICOKING for fuel and partial

oxidation for hydrogen are the most economically attractive bottoms processing options. Liquefaction conditions of 840°F and 60 minutes or longer residence time will provide a pumpable vacuum bottoms feed to the partial oxidation reactor.

- Several FLEXICOKING process improvement studies are underway. Various methods for increasing the amount of solids-free coker liquids from a recycle coking operation without undue economic debit were identified. Feeding a mixture of coal and vacuum bottoms to the FLEXICOKING unit to increase liquid yield and fuel gas production is also being evaluated as a potential process improvement.
- A recent update confirms the relatively large economic incentive shown in previous studies for maximizing the VPS cut point prior to partial oxidation or other non-FLEXICOKING bottoms processes. In addition, we defined 1050°F as the maximum probable VPS cut point as limited by preheat furnace constraints and viscosity limitations with the VPS itself. Because of high bottoms viscosity, a 1050°F VPS cut point could require vacuum bottoms solidification prior to feeding downstream units.
- An initial economic screening comparison of Texaco and Shell-Koppers partial oxidation processes (TPO/SPO) on liquid EDS vacuum bottoms indicates that TPO is 0.75 \$/B and 0.25 \$/B less expensive for producing hydrogen and fuel gas, respectively. For solidified vacuum bottoms, SPO appears to have about a 1 \$/B cost advantage for hydrogen because of TPO's aqueous slurry feed. The magnitude of these cost differences for liquid vacuum bottoms is expected to decrease based on revised SPO investments due from Shell in 3Q79. The above assessment is based on non-confidential information obtained from Texaco and Shell.

ENGINEERING RESEARCH AND DEVELOPMENT  
ENGINEERING TECHNOLOGY DEVELOPEMENT

4. LIQUEFACTION ENGINEERING TECHNOLOGY

- The new Generalized Riedel (G.R.) procedure for predicting the vapor pressures of coal liquids was incorporated into the Chao-Seader correlation and the Joffe-Zudkevitch modification of the Redlich-Kwong equation of state (RKJZ) used in estimating coal liquid vapor-liquid phase distributions. The G.R. procedure significantly improved the Chao-Seader predictions (deviation in weight fraction vaporized reduced from 20.9% to 12.8%) but had little effect on the RKJZ (13.8%).
- The viscosity data for Illinois vacuum bottoms and blends of bottoms with heavy raw creosote oil were fit to a power law viscosity model. The shear thinning and shear thickening behavior are modeled as a function of shear rate and 1000°F minus content at temperatures from 524°F to 704°F on the bottoms and 332°F to 425°F on the blends.

## 5. BOTTOMS PROCESSING ENGINEERING TECHNOLOGY

- The pressure leaf filtration tests by the vendor of the equipment showed that pressure filtration without precoat or body feed will not be suitable for fines removal from EDS scrubber liquid. Filtration rates were low, less than 4 gph/ft<sup>2</sup> after six minutes, the screens were rapidly blinded, and the solids removal efficiencies were poor.
- Recent lab work on FLEXICOKING unit reactor feed nozzles demonstrated that viscous solids free feeds can be atomized satisfactorily at lower gas rates than required previously.

## 6. MATERIALS AND ENVIRONMENTAL ENGINEERING TECHNOLOGY

- The organic acid concentrations of the Wyoming (Wyodak mine) process wastewater after stripping and extraction is 2.4 times that of Illinois (Monterey mine) wastewater and the total dissolved solids (TDS) 1.75 times. The resulting higher BOD<sub>5</sub> and TOC may require a trickling filter or other roughing biological treatment upstream of an activated sludge unit. Depending on the receiving body of water, the high TDS may require tower makeup and subsequent evaporation of the blowdown.

### ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING, PROCUREMENT AND CONSTRUCTION

#### 1. PROJECT MANAGEMENT

- In late 1978 and early 1979 ER&E project management team engineers prepared detailed descriptions and scope definitions of the thirteen sections of ECLP which are to be turned over in an ordered sequence in keeping with Carter Oil's start-up plan. Further subdivisions of some of the thirteen major turnover packages were prepared by Daniel and Carter. To assist in construction planning/execution as well as final checkout of each turnover package, McKee prepared listings of equipment, instruments, electrical facilities and piping drawings for each section and defined piping materials needed to provide for the separation/safe isolation of each package. This work was completed in June 1979.

#### 2. ENGINEERING AND PROCUREMENT

- McKee's engineering and procurement work for ECLP was 96% complete at the end of June 1979, approximately 1% or 4 weeks behind the original plan. The total number of McKee engineering and procurement hours projected at the end of June 1979 was 688k, an increase of 56% over our base projection of 440k hours, and 149k hours (28%) greater than our June 1978 projections. This increase reflects the continued development of scope changes that occurred as detailed engineering progressed.

- Basic engineering work during the year consisted primarily of the development of design bases for the changes in scope required as the detailed engineering progressed and operating/model reviews were completed, and the preparation of Change Lists to formally document all modifications to the original design specifications. Among the major changes handled by the Exxon engineering design follow-up engineers with assistance from the McKee engineering staff were:
  - + Electrical heating systems modifications/definitions.
  - + Power distribution systems changes for increased load requirements.
  - + Solvent flushing and blowdown systems definition.
  - + Dowtherm heating system operating range change.
  - + Definition of modifications required by interim turnovers.
- Preparation of the ECLP plant model, a vital engineering tool, continued into the second quarter of the year. As work progressed, the model was continually studied and used to resolve both engineering and anticipated operations problems. Teams made up of Carter Oil, Exxon process and safety engineers, and ER&E project management personnel conducted formal reviews as the models of the various areas were completed. After McKee revised the models in accordance with the input, piping isometric work proceeded.
- The number of tagged equipment items increased from 470 to 516 during the year, primarily due to changes. All items were ordered and 465 were delivered to the construction site. As of June 30, 1979, commitments for all procurement totaled 37.9M\$, 93% of the forecast total for the project vs. 99% planned.
- The cost outlook for ECLP as of June 30, 1979 was 112.15M\$. (The original cost estimate for ECLP was 110M\$). The most significant increases during the year, other than increases attributable to approved changes, were in bulk materials, direct labor and field indirects. The largest material increases were in field purchased bulk materials. Direct labor and indirects increased as the result of the increased bulk materials quantities projected and an anticipated three month schedule extension.

### 3. CONSTRUCTION

- Construction of ECLP was 62% complete at the end of June 1979, about 3 months behind our original plan. Progress during the year was affected by the increased scope of work, adverse weather, and delayed completion of engineering.
- Independent, in-depth ECLP construction schedule reviews by Daniel and ER&E were completed in May 1979. The studies were based on progress/productivity trends to date and review of materials deliveries and labor projections. Based on this work, we believe that the "most probable" completion date is 15 February 1980 + 2 weeks. We have designated 25 January 1980 as our "target" date for completion.

- Major ECLP construction events from July 1978 to June 1979 were the following:

	<u>1978</u>
+ First hydrostatic test of underground piping	July
+ Completed slipforming of raw coal silo	August
+ Started tank erection	September
+ Initiated field Positive Materials Identification Program	October
+ Started area paving	October
+ Started erection of process area pipe rack steel	October
+ Started installation of aboveground piping	October
+ Started construction of cooling tower	November
+ Started installation of underground power cables	December
	<u>1979</u>
+ Received and set all eight reactors	January
+ Received and set control house instrument racks	February
+ Completed cooling tower erection	February
+ Firewater system mechanically complete	April
+ Completed erection of field fabricated tanks	April
+ Interplant lines mechanically complete	May
+ Control house mechanically complete	June
+ Main electrical substation mechanically complete	June
+ Completed underground power/instrument cables	June

#### 4. PREPARATION FOR OPERATIONS

- Over the past year ending June 1979, the ECLP staff grew to 98 members with the addition of 86 people. These staff additions included 52 people for the Process Division, 6 people for the Mechanical Division, 19 people for the Technical Division and 9 people for the Administrative Division.
- The first turnover of equipment was accomplished on schedule on April 6, 1979, with the completion of a portion of the fire water system. This first turnover package included a diesel-engine-driven fire water pump and a portion of the fire water distribution system.
- In mid-February 1979, seventeen Process Technicians on loan from Exxon's Baytown Refinery arrived at the ECLP site to begin training. Four weeks of classroom training of the Process Technicians was completed during March 1979. Twenty-three new hires, who ultimately would be assigned to ECLP as Process Technicians, reported for work

at Exxon's Baytown Refinery in March 1979, and began five weeks of basic training in the Refinery. This group began onsite ECLP training in mid-May 1979.

- An environmental health work group was formed, with the task of developing a proposal for an ECLP Occupational Health Program. Elements of the program included areas such as personal hygiene, medical surveillance, and engineering controls. The program was reviewed and approved by year-end 1978.
- All ECLP staff members attended a safety workshop. The purpose of the workshop was to inform all employees of the several components that comprise the integrated safety program for ECLP, to solicit employee input into some of the program details and to provide a stimulus for safety awareness on the part of each employee.
- The purchase option was exercised for thirty (30) 100-ton "Rapid Discharge" hopper cars from Ortner Freight Car Company of Cincinnati, Ohio. These cars are scheduled to be manufactured in July 1979. Proform, Inc. was selected as the successful bidder of 30 rail car covers.
- The prototype ECLP rail car cover system was tested at Proform's plant in Minneapolis during April 1979. The cover was operated satisfactorily for 230 cycles, which is approximately twice the number of cycles expected during the 2-1/2-years of operation. The prototype ECLP rail car test program was successfully completed June 15, 1979. The prototype car made 15 trips (6,000 miles) between The Carter Oil Company's Monterey No. 1 Mine in Illinois and a power plant during the "over-the-rail" phase of the test program.
- All sections of the ECLP engineering model were received onsite and the entire model assembled during May 1979 for use as an aid to both training and construction.
- By the end of the first quarter 1979, the ECLP General Purpose Building was 97 percent complete overall. Storehouse personnel occupied their area during March 1979 and turnover was accomplished in April 1979.
- A specification sheet and floor plan for the ECLP laboratory was prepared in mid-April 1979 for release to obtain vendor quotations. Anticipated construction and equipment delivery times are consistent with having a fully operational laboratory in October 1979.
- A service agreement was executed early in 1978 between The Carter Oil Company and Exxon Company, USA's Baytown Refinery. This agreement established a task order system which outlines a procedure for obtaining a variety of services, including utilities for ECLP, from the Refinery. In all, twelve task orders with Exxon USA's Baytown Refinery were completed. Five task orders are being processed and 28 remain to be drafted. All task orders affecting ECLP should be completed by year-end 1979.



- Engineering data submitted by ECLP to the Texas Air Control Board (TACB) were reviewed by TACB and found to be acceptable. The requirements of Special Provision No. 13 of TACB Permit No. C-6080 were thus satisfied which permitted us to install our emission abatement equipment.
- As of the end of June 1979, the ECLP Mechanical Job Plans overall were 98 percent complete and copies of the manuals were reproduced for the use by the maintenance contractor. Also, editing and revision of the safety manuals is complete and the Carter Mechanical Procedures Manual is 85 percent complete.

## 5. OPERATIONS

- Reporting under this category to start upon the start of ECLP operations.

## 6. ECLP ENVIRONMENTAL ASSESSMENT

- This task was completed and no further reports will be made.

# FLEXICOKING PROTOTYPE PROJECT MANAGEMENT DESIGN, ENGINEERING AND PROCUREMENT

## 1. INSPECTION

- Inspection of the FLEXICOKING Prototype Unit for the Class IV cost estimate for revamp of the unit was completed. Internally, the unit (which had been nitrogen-blanketed) was in very good condition. Externally, small piping was heavily corroded and insulation was deteriorated.

## 2. PROCESS DESIGN

- Work on the FLEXICOKING Prototype Unit Onsite Design Specifications started in early February 1979. By the end of June 1979, these specifications were approximately 80 percent complete. A preliminary issue of the design specifications will be released internally for comments in July, with final issue targeted for early September 1979.
- During April 1979, the FLEXICOKING Prototype Unit Offsite Design Basis Memorandum was completed. In June 1979, the Offsite Facilities Definition document was issued. This document is being used as the basis for the Class IV cost estimates. Overall, the offsite design is approximately 15 percent complete.

### 3. ENVIRONMENTAL ASSESSMENT

- Data on estimated emissions at both the FLEXICOKING Prototype Unit and the vacuum bottoms storage facility at ECLP were assimilated and applications for construction permits were made to the Texas Air Control Board.

### 4. PROJECT MANAGEMENT

- Bids were solicited from six contractors covering the detailed design, procurement, and construction of the FLEXICOKING Prototype Unit facilities. Commercial terms will be on a cost-plus-fixed fee basis. The schedule for contract award is July 1, 1979.

### 5. ENGINEERING AND PROCUREMENT

- Reporting in this category is to start after contract award.

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LABORATORY PROCESS RESEARCH AND DEVELOPMENT  
LIQUEFACTION PROCESS RESEARCH

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EDS COAL LIQUEFACTION PROCESS DEVELOPMENT - PHASE IV  
ANNUAL TECHNICAL PROGRESS REPORT FOR THE PERIOD  
JULY 1, 1978 - JUNE 30, 1979

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INTRODUCTION

This report is the second Annual Technical Progress Report for U.S. Department of Energy Agreement No. EF-77-A-01-2893 for the Exxon Donor Solvent (EDS) Coal Liquefaction Process Development - Phase IV. This agreement covers the period January 1, 1977 to December 31, 1982, and is a continuation of DOE Contract No. E(49-18)-2353-Phase IIIA covering the period January 1, 1976 to June 30, 1977. The six-month overlap in funding in the first part of 1977 applies to activities related to the 250 T/D Exxon Coal Liquefaction Pilot Plant (ECLP), to be constructed during Phase IV of the project.

Prior development of the EDS Process was conducted during the ten-year period 1966 to January 1, 1976. During this period the Exxon Donor Solvent process was selected over several other options as the preferred route to the production of coal liquids. This work was funded entirely by Exxon and was divided into Phase I, the Predevelopment Phase, and Phase II, the Planning and Design Phase. A summary of this work is given in the report, FE-2893-16, "Summary of EDS Predevelopment (1966-1975)." Phase IIIA was a continuation of the Phase I and II programs, and was divided into the following two tasks:

- Laboratory Process Research and Development, and
- Engineering Research and Development.

A description of the work performed in Phase IIIA can be found in the reports cited.(1,2) Phase IIIB which began on July 1, 1977 and Phase IV which began on January 1, 1978, include the above two tasks as well as tasks relating to project management, detailed engineering, procurement, and construction of ECLP.

Part I of the Supplemental FLEXICOKING\* development program began October 23, 1978 and is scheduled for completion December 31, 1979. It includes laboratory and engineering tasks in support of the program, as well as tasks related to revamp of the FLEXICOKING Prototype Unit. Part II, a continuation of the program, is contingent on Sponsors Management Committee approval of the program.

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(1) January-December 1976, EDS Annual Technical Progress Report (FE-2353-9)

(2) January 1976-June 1977, EDS Final Technical Progress Report (FE-2353-20)

\* "Service Mark"

A list of Technical Progress Reports prepared for U.S. Department of Energy (and also submitted to private sector participants) under this program is given in Table 1. A list of relevant publications and presentations by Exxon on coal liquefaction during this time period is given in Table 2.

During the week of 21 August, 1978, an inspection team from the Office of the Inspector General of DOE conducted a detailed inspection of the overall EDS Project. Visits were made to facilities where the work is being conducted and discussions were held with individuals involved. Sites visited were: ER&E's EDS Process Research Laboratory in Baytown, Texas, ER&E's EDS Liquefaction Engineering Division and Engineering Technology Department in Florham Park, N.J., ECLP construction site in Baytown, and McKee's engineering offices in Cleveland. Discussions were held with individuals from ER&E, Carter, McKee, and Daniel who are working on all phases of the integrated R&D program. Also, during the week of 14 August, the leader of the DOE inspection team attended meetings of the ECLP and Overall R&D Subcommittees. ER&E, Carter and the EDS Sponsors participate in these meetings.

TABLE 1  
EDS COAL LIQUEFACTION PROCESS DEVELOPMENT  
TECHNICAL PROGRESS REPORTS PREPARED FOR DOE  
PHASES IIIB AND IV

<u>Report No.</u>	<u>Type</u>	<u>Period Covered</u>
FE-2893-16	Interim	Summary of EDS Predevelopment
FE-2893-17	Annual	July 1, 1977 - June 30, 1978
FE-2893-18	Monthly	July 1 - July 31, 1978
FE-2893-19	Monthly	Aug. 1 - Aug. 31, 1978
FE-2893-20	Monthly	Sept. 1 - Sept. 30, 1978
FE-2893-21	Quarterly	July 1 - Sept. 30, 1978
FE-2893-22	Monthly	Oct. 1 - Oct. 31, 1978
FE-2893-23	Monthly	Nov. 1 - Nov. 30, 1978
FE-2893-24	Monthly	Dec. 1 - Dec. 31, 1978
FE-2893-25	Quarterly	Oct. 1 - Dec. 31, 1978
FE-2893-26	Monthly	Jan. 1 - Jan. 31, 1979
FE-2893-27	Monthly	Feb. 1 - Feb. 28, 1979
FE-2893-28	Monthly	March 1 - March 31, 1979
FE-2893-29	Quarterly	Jan. 1 - March 31, 1979
FE-2893-30	Monthly	April 1 - April 30, 1979
FE-2893-31	Monthly	May 1 - May 31, 1979
FE-2893-32	Interim	EDS Commercial Plant Study Design Update - Revised Onsite Design Basis - Illinois Coal Base Case
FE-2893-33	Interim	EDS Commercial Plant Study Design Update - Offsite Design Basis - Illinois Coal Base Case
FE-2893-34	Monthly	June 1 - June 30, 1979

TABLE 2

EXXON PUBLICATIONS ON COAL LIQUEFACTION  
JULY 1, 1978 - JUNE 30, 1979

Swabb, Jr., L. E., Vick, G. K., and Aczel, T., "The Liquefaction of Solid Carbonaceous Materials", World Conference on Future Sources of Organic Raw Materials (CHEMRAWN), Toronto, Canada, July 10-13, 1978.

Epperly, W. R. and Taunton, J. W., "Exxon Donor Solvent Coal Liquefaction Process Development", 13th Intersociety Energy Conversion Engineering Conf., San Diego, CA, August 20-25, 1978.

Epperly, W. R., "Cooperative Agreement, EDS Coal Liquefaction Project", Regional Symposium of the National Contract Management Association, Houston, TX, October 27, 1978. Also in the July 1979 issue of Contract Management.

Swabb, L. E., Jr., "Prospects for Coal Liquefaction", National Academy of Engineering Annual Meeting, Washington, D.C., November 2, 1978.

Epperly, W. R. and Taunton, J. W., "Exxon Donor Solvent Coal Liquefaction Process Development", COAL DILEMMA II - American Chemical Society, Industrial and Engineering Chemistry Division, Colorado Springs, Col., February 12-13, 1979.

Lendvai-Lintner, E. and Sorell, G., "Materials Evaluation Program for the EDS Coal Liquefaction Process", CORROSION/79, National Association of Corrosion Engineering, Atlanta, GA, March 12-16, 1979.

Vernon, L. W., "Free Radical Chemistry for Coal Liquefaction", ACS/Chemical Society of Japan Congress, Honolulu, Hawaii, April 1-6, 1979.

Zaczepinski, S., Kamienski, P. W., et al, "Upgrading of Coal Liquids", Mid-Year API Meeting, San Francisco, CA, May 14-17, 1979.

Mitchell, W. N., et al, "Performance of Low Rank Coals in the Exxon Donor Solvent Process", 1979 Lignite Symposium, Grand Forks, N.D., May 30-31, 1979.

## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### LIQUEFACTION PROCESS RESEARCH

#### 1. Operation of Recycle Coal Liquefaction Units (RCLU)

##### 1.1 Unit Operations

The Recycle Coal Liquefaction Units (RCLU's)<sup>1</sup> are continuous integrated liquefaction units with nominal coal feed rates of 50 pounds per day (RCLU-1) and 100 pounds per day (RCLU-2). The units consist of a hydrogen feed system, coal slurry feed system, a liquefaction reactor, a hydrogenation reactor, and a product recovery and solvent recycle system. The flow plan for both units is shown schematically in Figure 1-1. A detailed flow diagram and process description is given in Appendix A of the January-June, 1976, EDS Quarterly Technical Progress Report [FE-2353-2].

The units are used for process variable studies, feed coal evaluations, and process improvement studies. During this reporting period the units were used to evaluate EDS sponsor-selected coals, test sulfur dioxide pretreatment effectiveness in reducing calcium carbonate deposition during liquefaction of a Wyoming subbituminous coal, and investigate the potential of liquefaction bottoms recycle with the EDS process. The coals selected by the EDS sponsors for screening studies in RCLU were a Pittsburgh seam bituminous coal from the Ireland mine in West Virginia (Department of Energy), a Texas lignite from the Big Brown mine (The Carter Oil Company), an Illinois No. 6 bituminous coal from the Burning Star No. 2 mine (Electric Power Research Institute), and a black coal from the Wandoan area of Queensland, Australia (Japan Coal Liquefaction Development Company). The liquefaction bottoms recycle studies were done using an Illinois No. 6 bituminous coal from the Monterey No. 1 mine and a Wyoming subbituminous coal from the Wyodak mine.

The data for these studies are presented in Appendix A. The operating conditions are presented in Table A-1,<sup>2</sup> and the yields are presented in Table A-2. Inspections for all feed and product streams are shown in Tables A-3 through A-5.

Yields from these runs are based on an elemental balancing procedure. This procedure balances the total weight of feed and products and also balances the elements carbon, hydrogen, oxygen, sulfur, nitrogen, and total ash.

##### 1.2 EDS Sponsor Coal Screening Studies

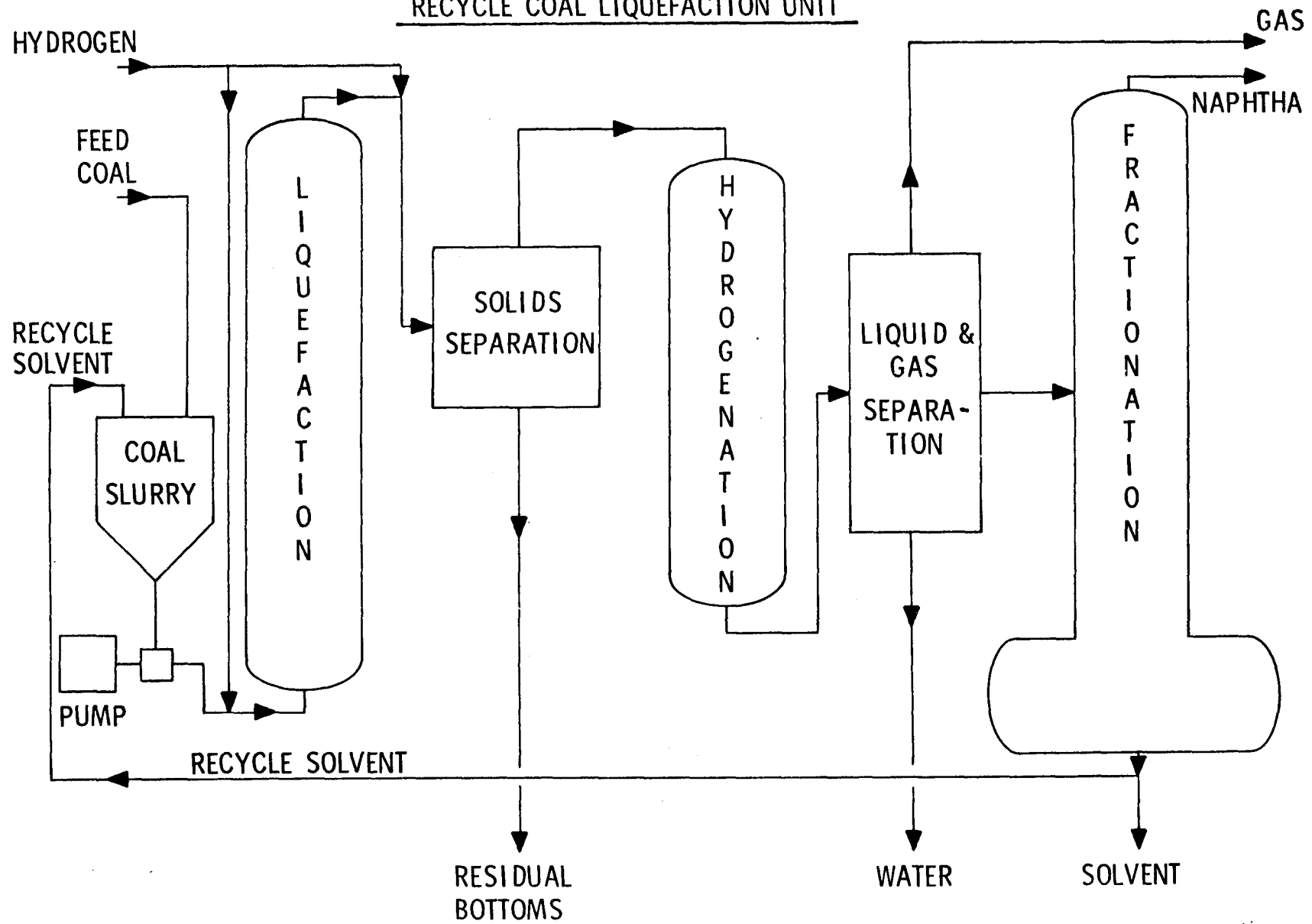
The purpose of these screening studies was to determine the response of these coals in the EDS liquefaction process. Liquefaction

(1) A list of abbreviations is included at the end of this report.

(2) Tables and figures with a letter prefix are located in the Appendices.

FIGURE 1-1

RECYCLE COAL LIQUEFACTION UNIT





yields and unit operability were investigated primarily as a function of liquefaction temperature and liquefaction residence time. Liquefaction pressure, molecular hydrogen treat rate, and solvent-to-coal ratio were also investigated briefly for some of the coals. The residual bottoms product and residual liquefaction reactor solids were also analyzed for each coal.

During the first quarter of 1979 RCLU-1 operations were devoted to a screening study with Wandoan coal from Queensland, Australia. The results of this study are presented in detail in this report. The results of the other sponsor coal screening studies have been reported previously and the data are presented in this report in a summarized fashion. For more details, see the following references.

<u>Coal</u>	<u>EDS Quarterly Technical Progress Report</u>
Ireland	July, 1977-June, 1978 [FE-2893-17]
Arkwright	July-September, 1978 [FE-2893-21]
Big Brown	July-September, 1978 [FE-2893-21]
Burning Star No. 2	January-March, 1979 [FE-2893-29]

Arkwright mine coal from the Pittsburgh seam in West Virginia was not a sponsor-selected coal, but was studied briefly because of interest in liquefaction behavior with a higher rank coal. Other coals included for comparison which were studied earlier in the EDS program are North Dakota lignite (Indian Head mine), Wyoming subbituminous (Wyodak mine), and Illinois No. 6 bituminous (Monterey No. 1 mine) coals.

#### • Wandoan Coal Screening Study

##### Study Conditions

The liquefaction conditions investigated in this study are as follows.

<u>Temperature, °F</u>	<u>Residence Time, Minutes</u>	<u>Pressure, psig</u>
800	40	1500
	100	1500
840	25	1500
	40	1500
	40	2500
	100	1500
880	25	1500

Solvent-to-coal ratio and molecular hydrogen treat rate were held constant at 1.6:1 and 4 wt% on dry coal, respectively.

## Conversion and Yields

The yields for Wandoan coal are presented in Table 1-1 and in Figures 1-2 through 1-6. The Wandoan coal was not cleaned for this study and the coal contained about 20 wt % ash. Cleaning the coal should reduce the ash content to approximately 10 wt % and increase conversion and yields. The 1000°F conversion for Wandoan coal at 840°F increases rapidly as residence time is increased from 25 to 40 minutes. Beyond 40 minutes, the 1000°F conversion is still increasing, but at a much slower rate. The corresponding C<sub>4</sub>-1000°F liquid yield shown in Figure 1-3 also increases significantly up to 40 minutes residence time. Beyond 40 minutes residence time, the liquid yield decreases with increasing coal conversion due to increased cracking of liquids to gas. The increase in C<sub>1</sub>-C<sub>3</sub> gas yield at 840°F with increasing residence time is shown in Figure 1-4.

The 1000°F conversion at 800°F parallels, but is significantly lower, than that at 840°F. However, the C<sub>4</sub>-1000°F liquid yield at 100 minutes is still increasing at 800°F in contrast to 840°F. As shown in Figure 1-4, the C<sub>1</sub>-C<sub>3</sub> gas yield at 800°F is considerably lower than at 840°F.

Coal conversion is increased significantly at 25 minutes residence time by increasing liquefaction temperature from 840°F to 880°F. However, essentially all of this increase in coal conversion at 880°F resulted in additional gas make, not liquids, compared to 840°F. Also considerably more cracking to naphtha was observed in the liquids at 880°F, as indicated in Table 1-1.

Raising liquefaction pressure from 1500 psig to 2500 psig at 840°F and 40 minutes residence time did not greatly affect conversion and yields. Conversion and C<sub>4</sub>-1000°F liquids increased by 1.8 and 2.7 lbs/100 lbs of dry coal, respectively. C<sub>1</sub>-C<sub>3</sub> gas yield decreased slightly, 0.9 lbs/100 lbs dry coal, at 2500 psig. Likewise, the percentage of naphtha in a C<sub>4</sub>-1000°F liquid dropped from 67 wt % at 1500 psig to 62 wt% at 2500 psig. Lower gas make and heavier liquids at 2500 psig versus 1500 psig indicate that cracking is suppressed during the higher pressure operations.

Hydrogen consumption for Wandoan coal is shown in Figure 1-5. As expected, hydrogen consumption increases with increasing residence time (more gas and naphtha) both at 800°F and 840°F at 1500 psig. Hydrogen consumption is lower at 800°F than at 840°F because of lower coal conversion at 800°F. Likewise, the dramatic increase in hydrogen consumption at 880°F/25 minutes compared to 840°F/25 minutes is due to the higher coal conversion and shift to a lighter product slate at 880°F. Very little additional hydrogen is consumed at 2500 psig/40 minutes compared to 1500 psig/40 minutes. Although 2.7 lbs/100 lbs dry coal more liquid is produced at 2500 psig, less C<sub>1</sub>-C<sub>3</sub> gas and a heavier liquid product reduce hydrogen consumption to about the 1500 psig level.

The hydrocarbon product yields at 840°F/1500 psig for Wandoan coal in Table 1-1 are shown graphically in Figure 1-6. Continual cracking

TABLE 1-1

SUMMARY OF AUSTRALIAN WANDOAN COAL YIELDSLiquefaction Conditions

Temperature, °F	840	800	840	800	840	880	840
Pressure, psig	1500	1500	1500	1500	1500	1500	2500
Residence Time, Minutes	40	40	100	100	25	25	40

Liquefaction Yields

(Lbs/100 Lbs dry coal)

H <sub>2</sub>	-3.1	-2.4	-4.1	-2.9	-2.6	-4.0	-3.3
H <sub>2</sub> O	10.6	10.7	12.6	10.5	10.3	12.7	11.5
CO	1.0	0.8	1.1	1.0	1.0	1.3	0.9
CO <sub>2</sub>	2.2	1.7	2.2	2.0	2.2	2.5	1.4
NH <sub>3</sub>	0.3	0.2	0.3	0.2	0.2	0.3	0.3
H <sub>2</sub> S	0.2	0.2	0.2	0.2	0.1	0.2	0.2
C <sub>1</sub>	2.7	1.2	4.8	2.7	2.0	4.7	2.3
C <sub>2</sub>	2.1	1.1	3.5	1.7	1.5	3.3	1.9
C <sub>3</sub>	2.3	1.2	3.8	1.9	1.6	3.3	2.1
C <sub>4</sub>	1.6	0.9	2.1	1.3	1.2	2.0	1.1
C <sub>5</sub> -400°F	16.9	11.4	22.7	15.3	14.1	19.2	17.7
400-700°F	5.8	5.7	-3.2	5.4	1.2	-2.0	6.2
700-1000°F	3.4	4.3	3.2	4.0	5.6	3.5	5.5
1000°F+ Bottoms	54.1	63.1	50.8	56.8	61.5	53.0	52.3
C <sub>1</sub> -C <sub>3</sub> Gas	7.1	3.5	12.1	6.3	5.2	11.3	6.3
C <sub>4</sub> -400°F Naphtha	18.5	12.3	24.8	16.5	15.2	21.2	18.8
C <sub>4</sub> -1000°F Liquids	27.7	22.4	24.8	25.9	22.0	22.7	30.4

1000°F- Conversion

(Lbs/100 Lbs dry coal)

45.9	36.9	49.2	43.2	38.5	47.0	47.7
------	------	------	------	------	------	------

Liquid Product Selectivity, wt%(C<sub>4</sub>-400°F Naphtha/  
C<sub>4</sub>-1000°F Liquids)

66.7	55.2	99.6	63.8	69.1	93.3	61.7
------	------	------	------	------	------	------

Yield Periods

625-628	629-631	632-634	635-637	638,639	641,642	643-647
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FIGURE 1-2

EFFECT OF LIQUEFACTION RESIDENCE TIME ON  
CONVERSION OF AUSTRALIAN WANDOAN COAL

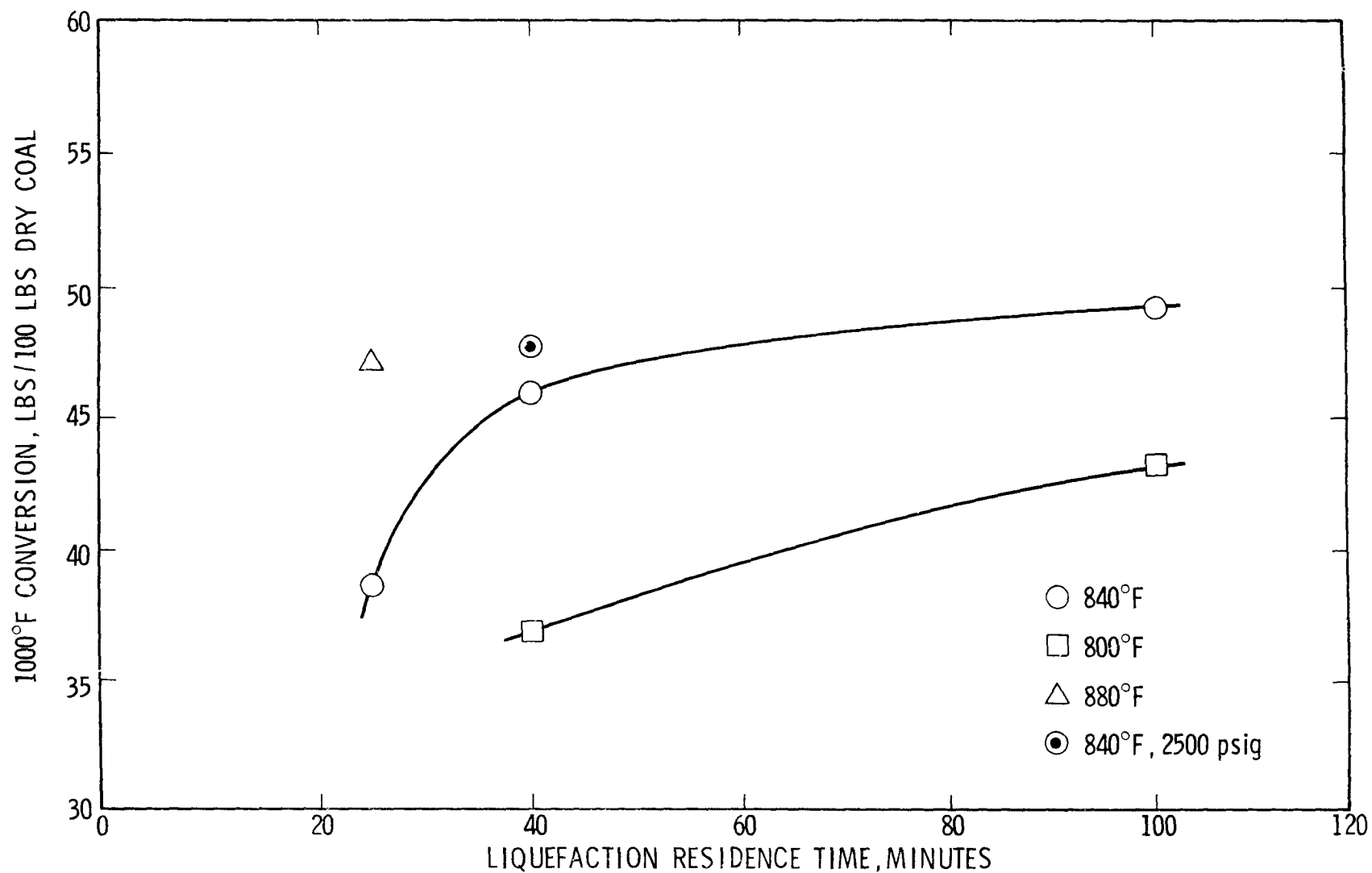


FIGURE 1-3

EFFECT OF LIQUEFACTION RESIDENCE TIME ON  
C<sub>4</sub>-1000°F LIQUID YIELD FOR AUSTRALIAN WANDOAN COAL

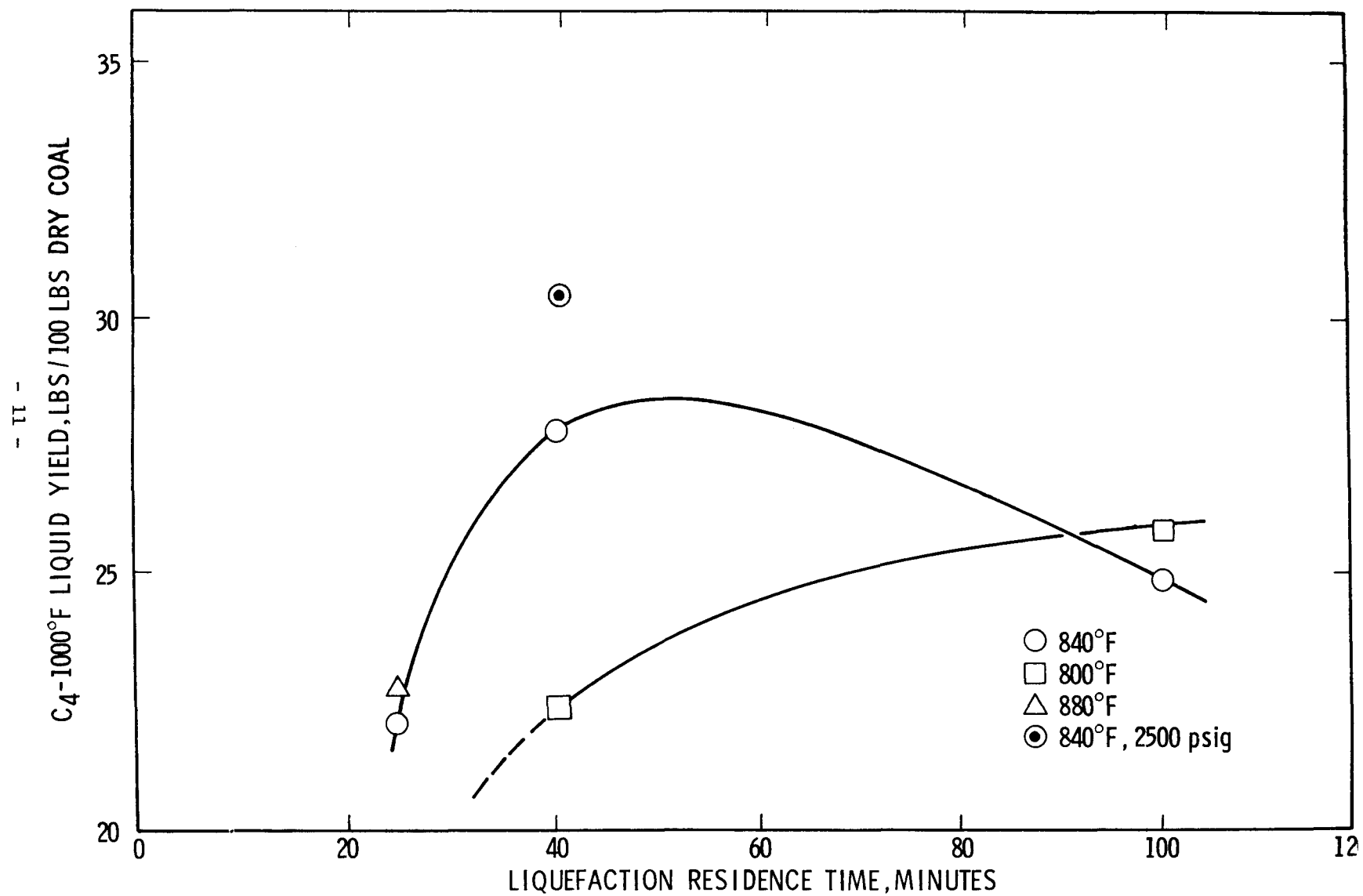
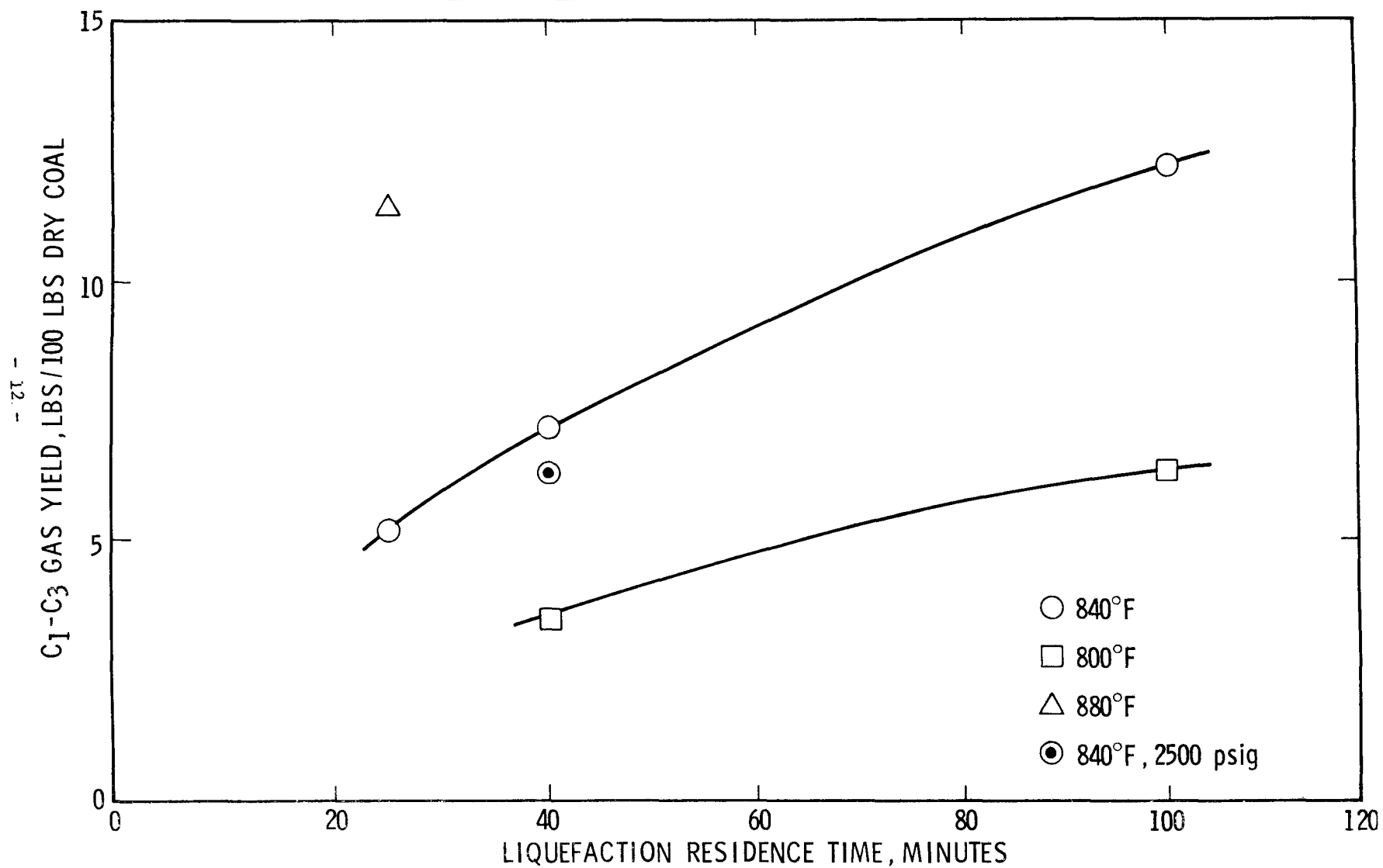


FIGURE 1-4

EFFECT OF LIQUEFACTION RESIDENCE TIME ON  
C<sub>1</sub>-C<sub>3</sub> GAS YIELD FOR AUSTRALIAN WANDOAN COAL



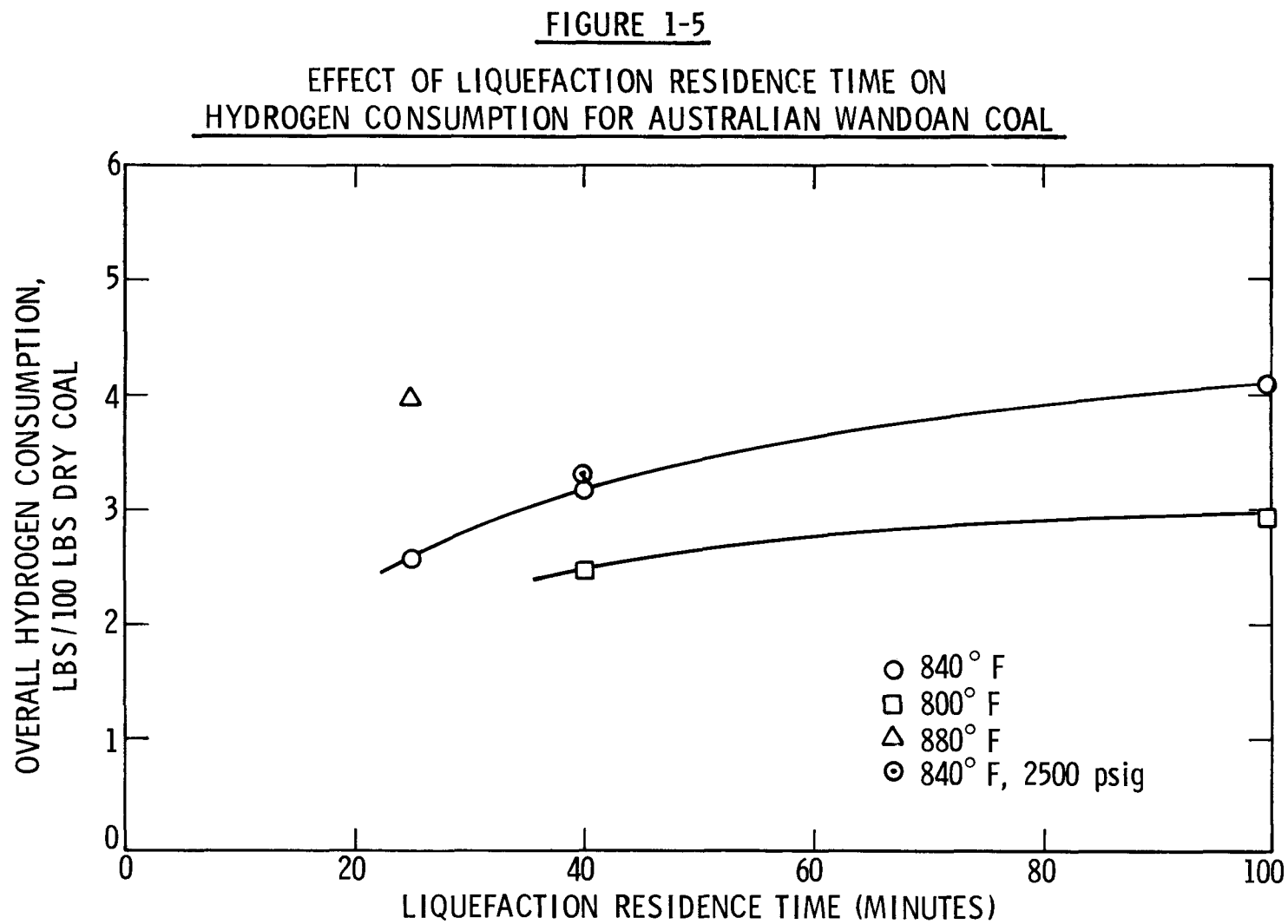
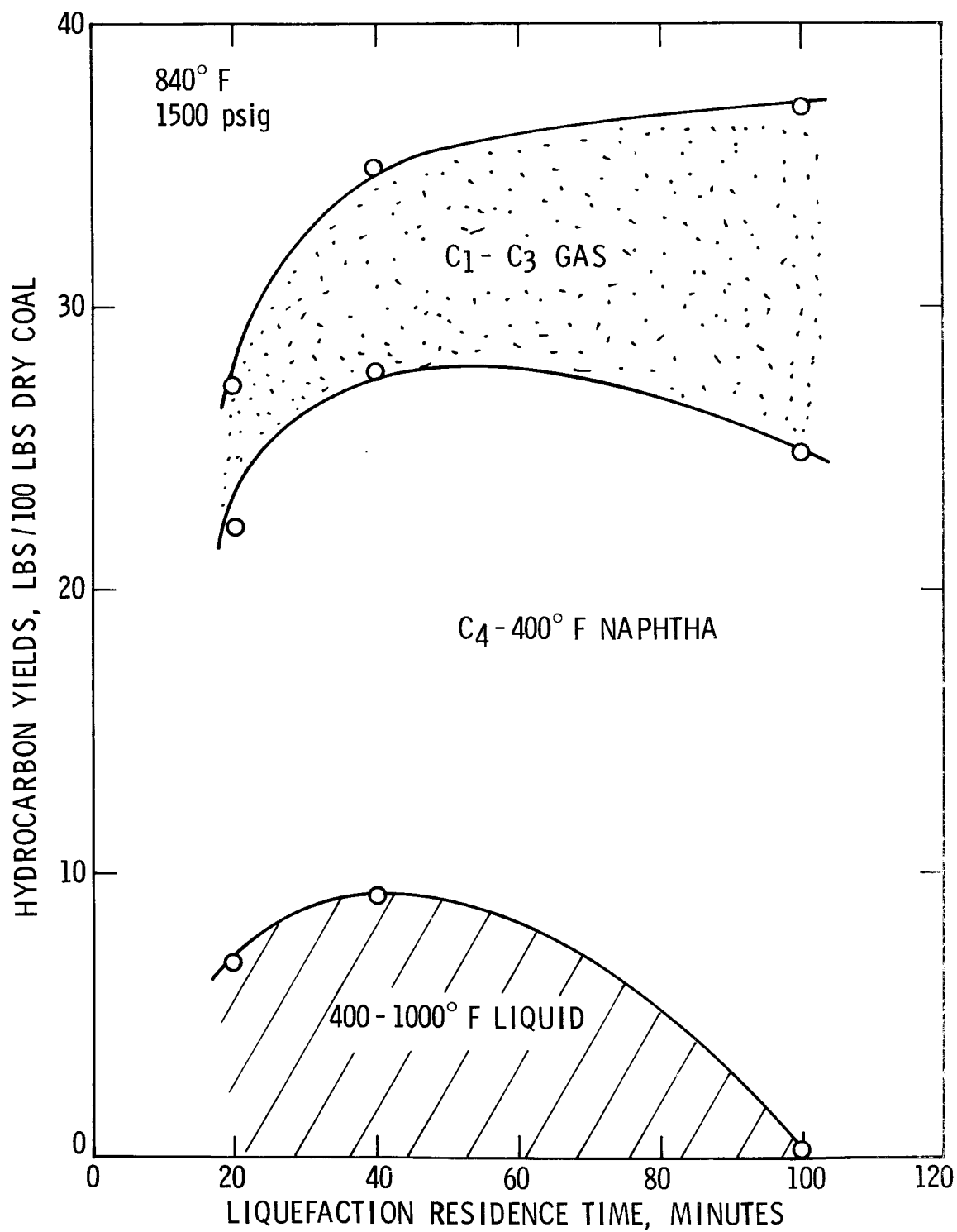


FIGURE 1-6

LIQUEFACTION PRODUCT SELECTIVITY FOR AUSTRALIAN WANDOAN COAL





to lighter products is apparent as residence time (i.e. liquefaction severity) is increased. At 25 minutes, total hydrocarbon yield is still low, 27.2 lbs/100 lbs dry coal. At this condition, the 400-1000°F liquid constitutes 25.0% of the C<sub>1</sub>-1000°F hydrocarbon product, the C<sub>4</sub>-400°F naphtha comprises 56.0%, and the C<sub>1</sub>-C<sub>3</sub> gas equals 19.0%. These proportions become 26.5%, 53.0%, and 20.5%, respectively, as coal conversion increases and more total liquid is made at 40 minutes residence time. Cracking becomes significant after about 40 minutes at 840°F/1500 psig as shown by the decrease in 400-1000°F liquid and increase in naphtha and gas. Solvent balanced operations with Wandoan coal become critical as 100 minutes residence time is approached at 840°F. Similar solvent balance concerns are apparent at 880°F/25 minutes as shown by the net loss of 400-700°F liquid and high naphtha yield in Table 1-1.

- Summary of Sponsor Coal Liquefaction Yields

The liquefaction yields for the sponsor-selected coals plus several other coals run in RCLU are presented in Table 1-2 at conditions of highest C<sub>4</sub>-1000°F liquid yield. These are not necessarily optimal liquid yields for any coal, particularly since several coals were screened on a limited basis. The number of liquefaction conditions investigated is shown for each coal.

The chemical gas and water yields are approximately constant for each coal at all conditions studied. Hydrogen consumption is dependent upon coal conversion and the distribution of hydrocarbon products, i.e. gas, naphtha, heavy liquid. In general, as can be seen, the coals which require more severe conditions to achieve high liquid yield also produce proportionately more gas and naphtha.

TABLE 1-2

HIGHEST LIQUID PRODUCT YIELDS FOR EDS PROGRAM COALS AT 1500 PSIG(RCLU Data)

	Coal							
	<u>Arkwright</u>	<u>Ireland</u>	<u>Burning Star</u>	<u>Monterey</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big Brown</u>	<u>Indian Head</u>
<u>Liquefaction Conditions</u>								
Temperature, °F	840	840	880	800	840	840	840	840
Residence Time, Minutes	100	100	25	140	40	100	25	40
<u>Number of Conditions</u>								
<u>Investigated</u>	2	10	7	16	6	10	4	3
<u>Liquefaction Yields</u> (Lbs/100 Lbs dry coal)								
Hydrogen	-4.2	-4.6	-3.4	-4.6	-3.1	-4.8	-3.1	-4.3
Water	4.6	6.0	8.2	9.8	10.6	15.1	10.4	17.5
Carbon Oxides	1.1	1.4	1.5	0.6	3.2	5.8	6.8	7.9
Ammonia	0.7	0.6	0.6	0.7	0.3	0.5	0.4	0.6
Hydrogen Sulfide	1.8	3.2	2.4	3.4	0.2	0.5	0.7	0.4
C <sub>1</sub> -C <sub>3</sub> Gas	13.5	13.5	9.5	9.0	7.1	10.1	6.2	6.8
C <sub>4</sub> -1000°F Liquid	29.9	32.7	30.4	36.1	27.7	30.9	28.0	28.1
<u>1000°F Conversion</u> (Lbs/100 Lbs dry coal)								
	47.4	52.8	49.2	55.0	46.0	58.1	49.4	57.0
<u>Liquid Product</u> <u>Selectivity, wt%</u>								
C <sub>4</sub> -400°F Naphtha	73.2	71.6	48.0	62.7	66.7	80.1	57.6	68.5
C <sub>4</sub> -1000°F Liquids								

Analyses of the feed coals run in RCLU are presented in Table 1-3. The differences in oxygen and sulfur content for these different coals are manifested in the corresponding hydrogen sulfide, carbon oxides, and water yields. Coal nitrogen contents are not sufficiently different to accurately follow the corresponding levels of ammonia yields. Water and carbon oxide yields for the different coals studied in RCLU are shown in Figure 1-7 as a function of organic oxygen (i.e. oxygen by difference) in the coals. As can be seen, water yield correlates very well with coal oxygen content. One consequence of this is greater hydrogen consumption (to make water) for the coals containing more organic oxygen. Thus, the lower rank coals will undesirably consume more hydrogen to make water than the higher rank coals. The carbon oxide yields show similar dependence on coal oxygen content. Figure 1-8 shows the hydrogen sulfide yields as a function of total sulfur content for the different coals. The Monterey and Ireland bituminous coals have the highest sulfur contents and consequently the highest hydrogen sulfide yields. The lower rank coals contain significantly less sulfur and thus much lower hydrogen sulfide yields. Wandoan coal, which is between Monterey and Wyodak coals in rank, had the lowest sulfur content (0.38 wt% DAF) of the coals investigated and also the lowest hydrogen sulfide yield (0.19 wt% DAF coal basis).

Most of the screening study data were taken at 840°F/1500 psig for the different coals, and liquid yields at these conditions are presented in Figure 1-9. As can be seen, the yield responses for the coals are for the most part unrelated to rank. For example, Monterey and Burning Star coals are close in rank (and both are Illinois No. 6 coals) but exhibit dramatically different yield behavior over the same range of conditions. Thus the different conditions shown in Table 1-2 required to obtain the highest liquid yields for each coal is not surprising.

The varying responses of C<sub>4</sub>-1000°F liquid yields for these coals also results in considerable flexibility in liquid product distribution, as shown in Figure 1-10. For most of the coals the wt % C<sub>4</sub>-400°F naphtha in the total liquid product can be varied by 15 to 20 wt % over the range of conditions shown. For Wandoan and Big Brown coals, the flexibility is much greater over the same range of conditions because both coals approach solvent balance limitations rapidly (i.e. all naphtha product) as 100 minutes residence time is approached.

These screening study results show that more detailed process variable studies will be required for each coal to determine the preferred EDS yield conditions. Results from one coal cannot necessarily be used to approximate optimum conditions for another coal, even though the coals may be close in rank.

#### • Summary of Unit Operations with Sponsor Coals

An operations summary for the coals run in the RCLU screening studies is presented in Table 1-4. Considerably more RCLU time was devoted to pre-EDS program studies with Monterey and Wyodak coals. Over 10,000 hours on coal from more than 40 runs have been logged with Monterey

TABLE 1-3

ANALYSES OF FEED COALS RUN IN RCLU

<u>Coal</u>	<u>Illinois No. 6</u> <u>Bituminous</u>		<u>Pittsburgh Seam</u> <u>Bituminous</u>		<u>Australian</u> <u>Black</u>	<u>Wyoming</u> <u>Subbituminous</u>	<u>Texas</u> <u>Lignite</u>	<u>North Dakota</u> <u>Lignite</u>
<u>Mine</u>	<u>Monterey</u> <u>No. 1</u>	<u>Burning Star</u> <u>No. 2</u>	<u>Ireland</u>	<u>Arkwright</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big Brown</u>	<u>Indian Head</u>
<u>Elemental Analyses, Dry Wt%</u>								
Carbon	70.1	70.4	74.0	78.4	59.8	68.5	62.0	63.8
Hydrogen	5.1	4.9	5.2	5.4	5.0	4.9	4.8	4.7
Oxygen (by difference)	10.6	9.9	6.3	5.1	13.4	17.2	14.5	19.2
Nitrogen	1.2	1.2	1.2	1.5	0.7	1.1	1.1	0.9
Sulfur	4.1	3.1	4.3	2.3	0.3	0.5	1.2	1.2
Ash	8.9	10.5	9.0	7.3	20.8	7.8	16.4	10.2
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
H/C Atomic Ratio	0.87	0.84	0.84	0.82	1.01	0.86	0.92	0.88
Ash (SO <sub>3</sub> -Free)	8.8	10.2	8.8	7.0	20.8	6.6	14.0	7.6
Total Oxygen	15.1	14.9	10.0	8.5	26.3	23.4	23.8	26.0
<u>Equilibrium Moisture, Wt%</u>	14.0	10.4	2.1	1.8	10.5	29.0	-	33.6
<u>Proximate Analyses, Dry Wt%</u>								
Volatile Matter	42.1	39.0	39.1	36.8	44.6	45.5	44.4	44.1
Fixed Carbon	49.0	51.2	51.9	55.9	34.6	46.7	39.2	45.7
Ash	8.9	10.5	9.0	7.3	20.8	7.8	16.4	10.2

FIGURE 1-7

WATER AND CARBON OXIDE YIELDS FOR SPONSOR COALS  
AS A FUNCTION OF OXYGEN IN COAL

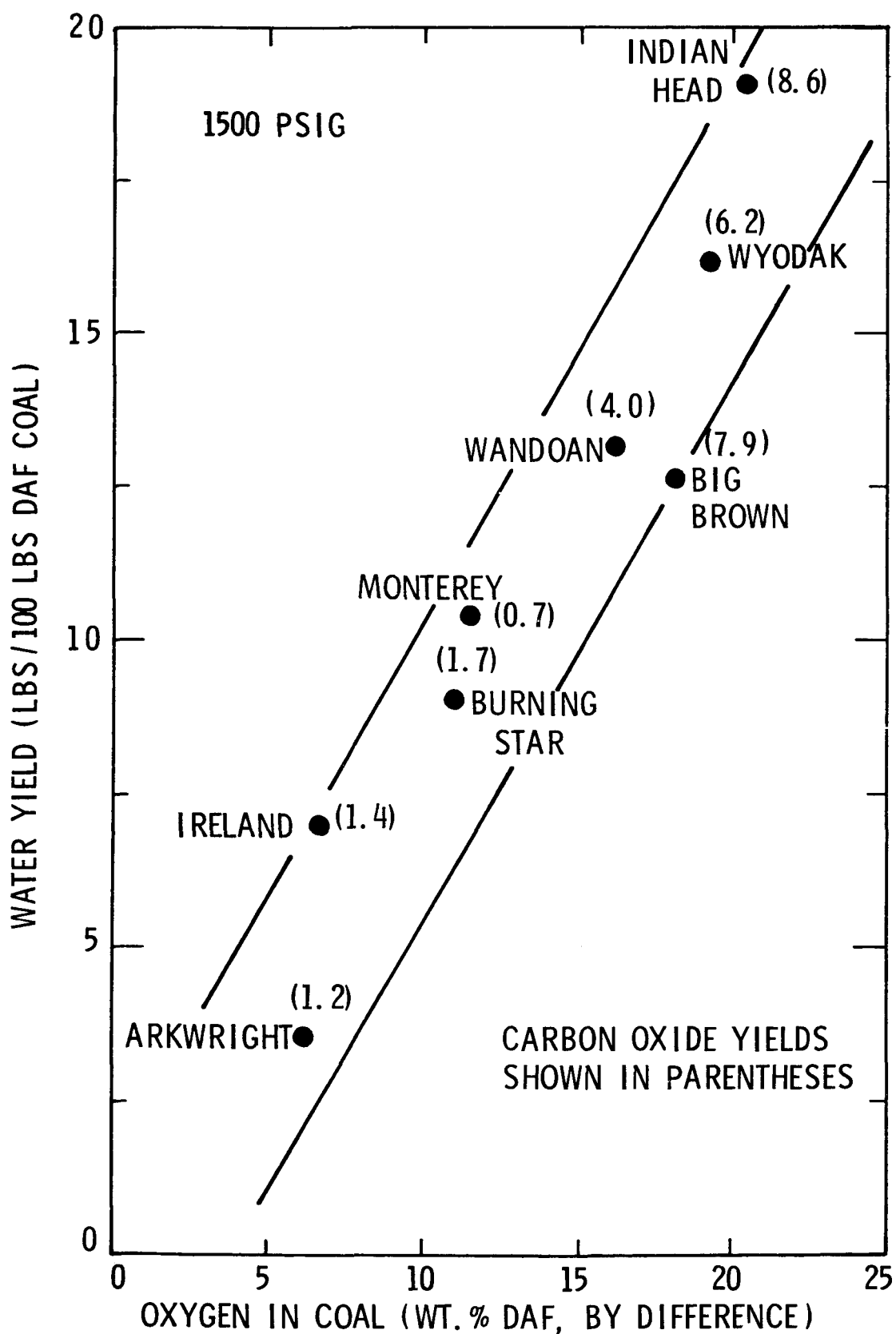


FIGURE 1-8

HYDROGEN SULFIDE YIELDS FOR SPONSOR COALS  
AS A FUNCTION OF SULFUR IN COAL

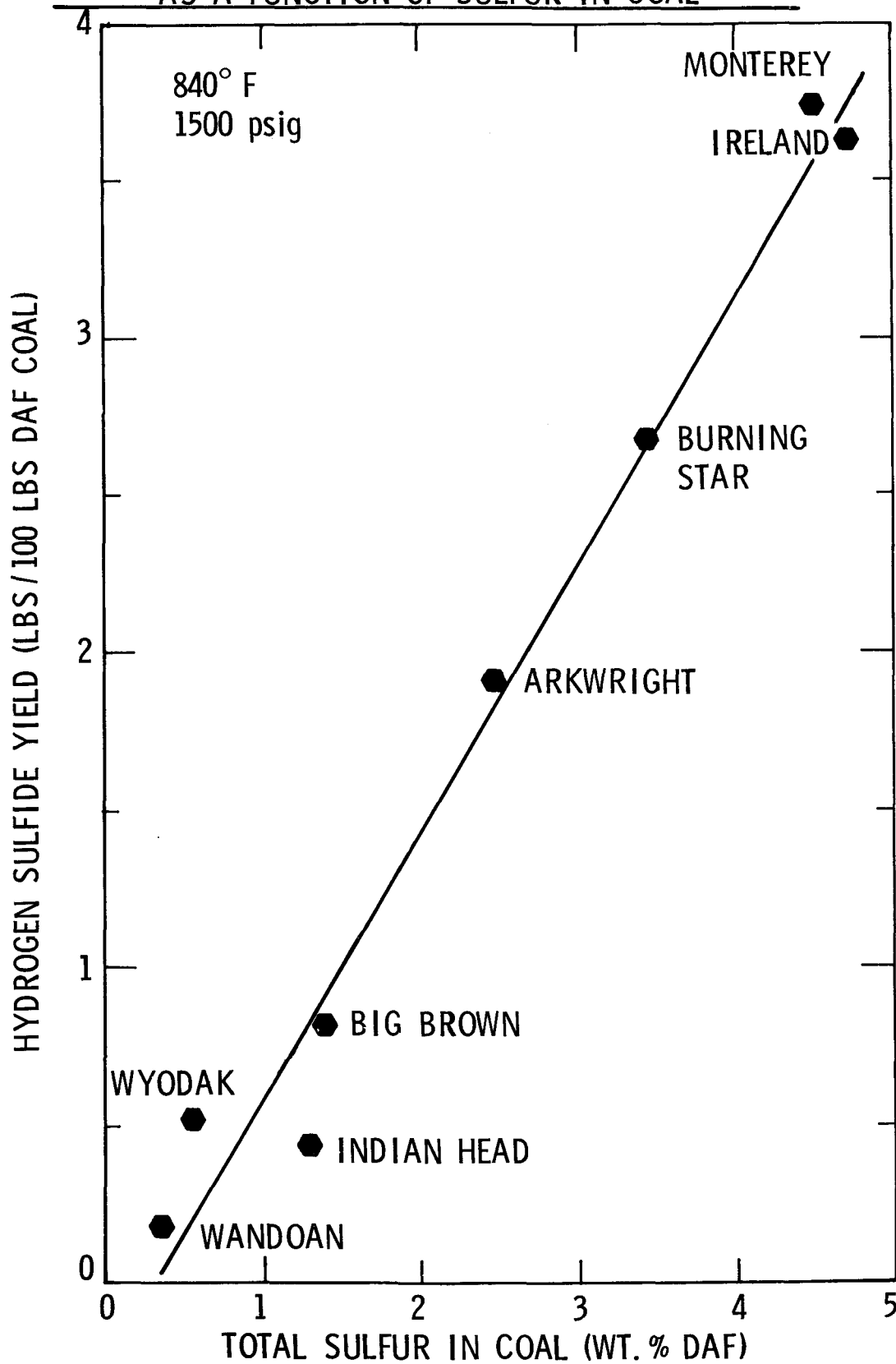


FIGURE 1-9

EFFECT OF LIQUEFACTION RESIDENCE TIME ON LIQUID YIELDS FOR EDS PROGRAM COALS

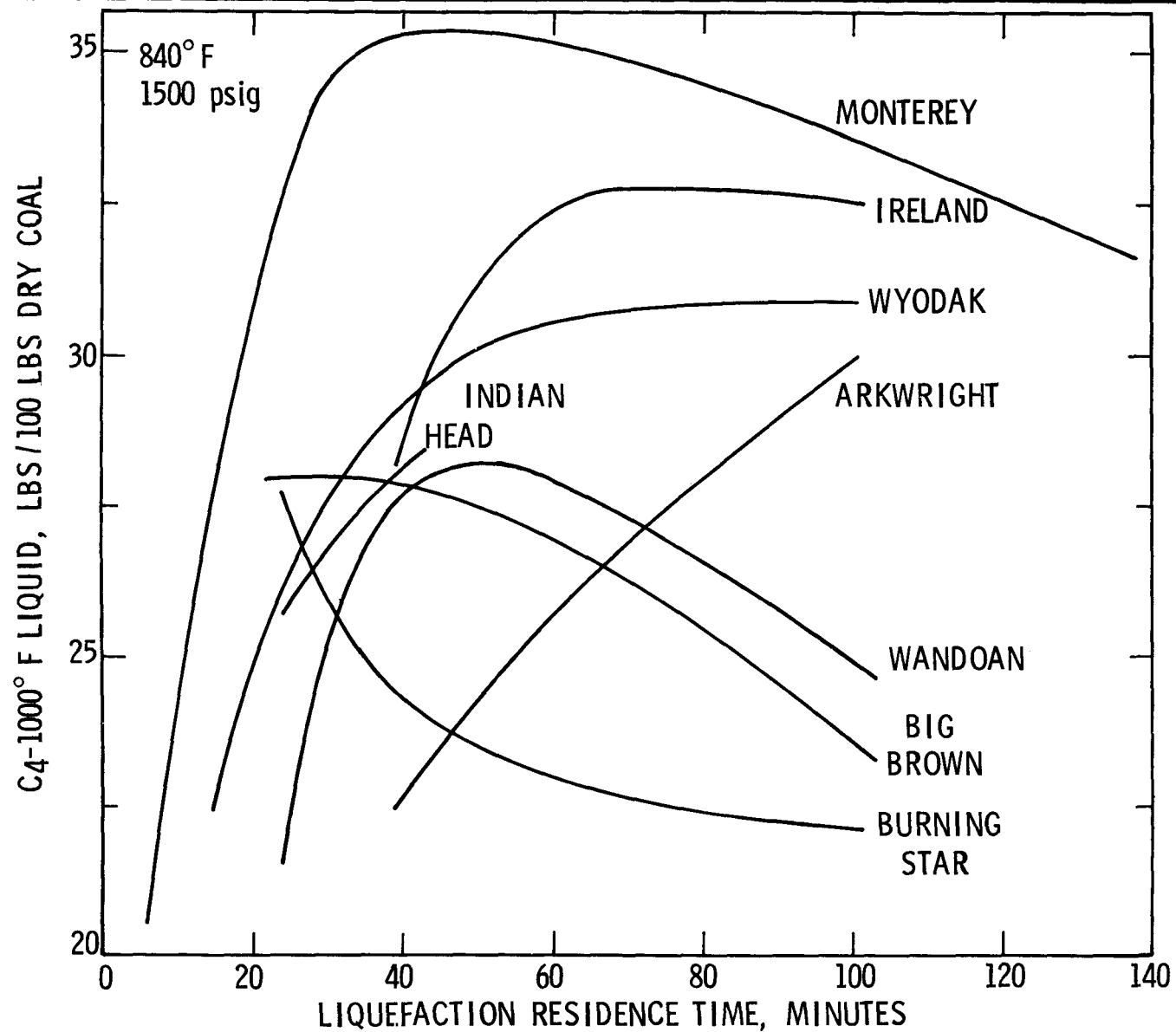
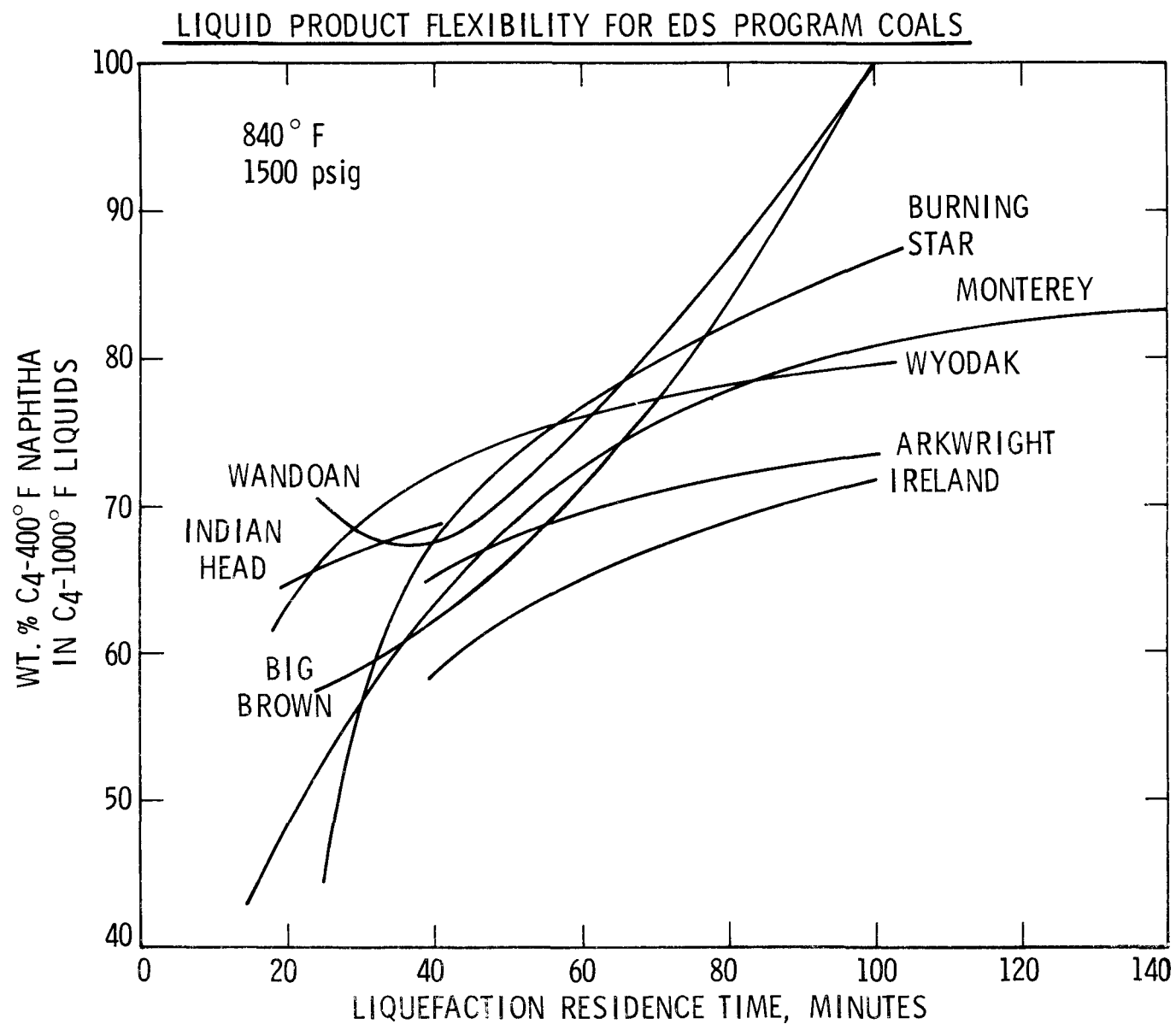


FIGURE 1-10





coal. The longest Monterey run in RCLU was 610 hours. Wyodak coal has been investigated for over 6000 hours on coal in more than 50 runs. The longest continuous run with Wyodak coal was 171 hours.

TABLE 1-4

OPERATIONS SUMMARY FOR RCLU-1 COAL SCREENING STUDIES

<u>Sponsor Coals</u>	<u>Hours in Unit</u>	<u>Longest Run, hrs.</u>	<u>Number of Runs</u>
Ireland	711	248	4
Big Brown	681	139	8
Burning Star No. 2	833	288	5
Wandoan	773	205	6
<u>Additional Coals</u>			
Arkwright	320	129	3
Indian Head	470	73	10

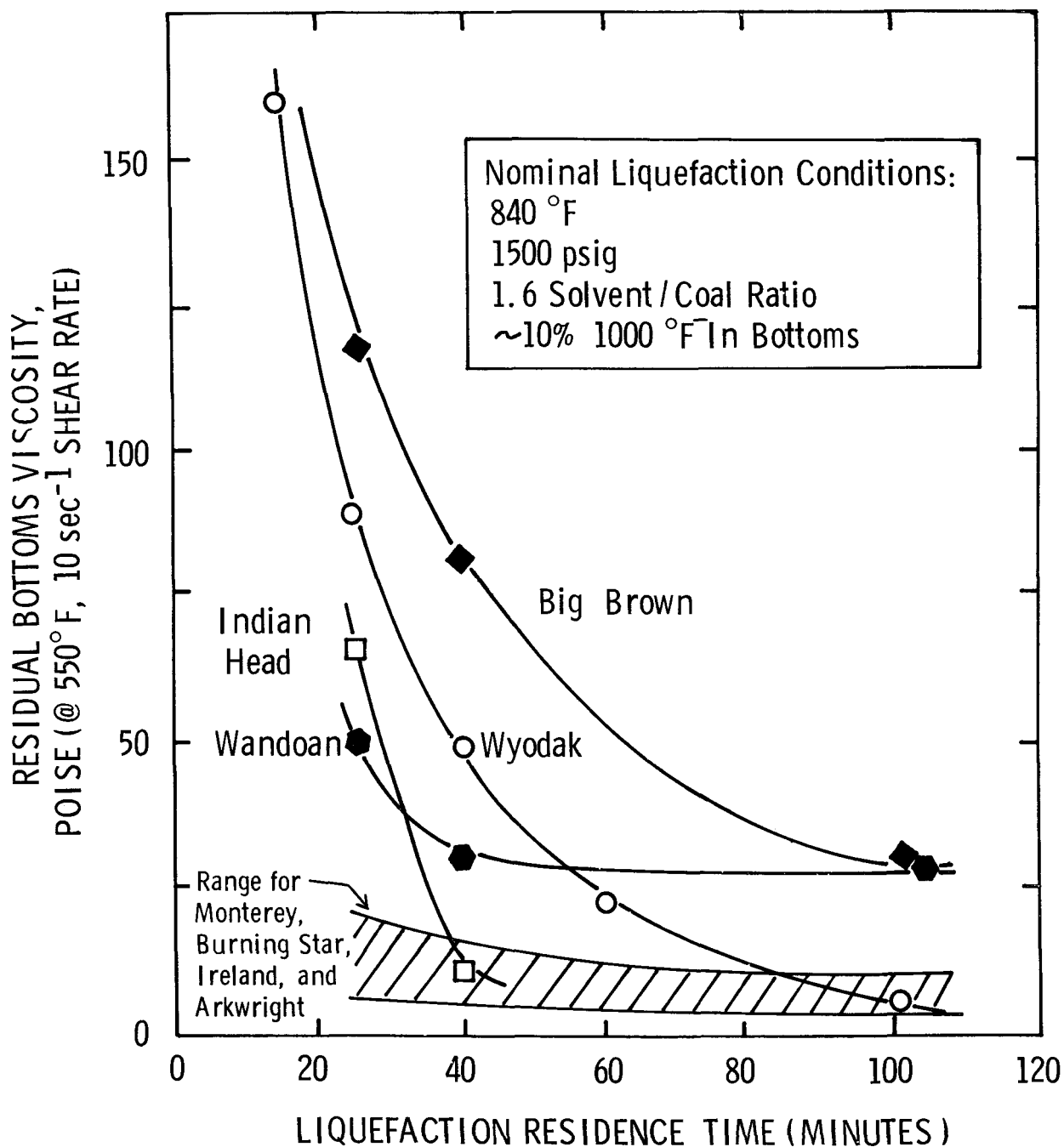
While yield structure was not for the most part related to coal rank, operability in RCLU was found to be directly affected by coal rank. The lower rank coals were more difficult to run in RCLU. Run lengths were typically shorter than for the higher rank coals, and the runs were very often ended due to plugging in the liquefaction reactor, product separator drawdown valves, and residual bottoms accumulator drawdown valve.

Residual bottoms viscosity has been shown to be an effective index to operability trends in RCLU. The lower rank coals, i.e. Big Brown, Indian Head, and Wyodak coals, exhibit much higher bottoms viscosities than the higher rank coals, i.e. Monterey, Burning Star, Ireland, and Arkwright. Furthermore, as shown in Figure 1-11, the bottoms viscosities for lower rank coals are a function of coal conversion. As liquefaction severity is increased (via increased residence time in Figure 1-11), conversion increases and the resulting bottoms viscosities decrease. Typically RCLU plugging problems occur when bottoms viscosities exceed 50-100 poise (550°F, 10 sec<sup>-1</sup> shear rate). This happens when Big Brown, Wyodak, and Indian Head coals are liquefied below about 40 minutes residence time at 840°F/1500 psig.

No plugging problems due to buildup of viscous residue in the liquefaction reactor, product separators, or residual bottoms accumulator were experienced with the higher rank Monterey, Burning Star, Ireland, and Arkwright coals. Withdrawal of residual bottoms from the bottoms accumulator was accomplished easily because of the low bottoms viscosities, as shown in Figure 1-11.

Figure 1-11

EFFECT OF RESIDENCE TIME ON RESIDUAL LIQUEFACTION  
BOTTOMS VISCOSITY FOR COALS RUN IN RCLU



Wandoan coal, which is between Monterey high volatile C bituminous and Wyodak subbituminous C in rank, exhibited operability behavior intermediate to Monterey and Wyodak behavior. Of the six RCLU runs made with Wandoan coal, two were ended by plugs forming in the liquefaction reactor. Near the end of a third run, several high pressure surges were recorded in the liquefaction reactor, indicating an incipient plugging condition. However, unit operability overall was judged better than for Wyodak coal because no problems were experienced emptying the residual bottoms accumulator even though the Wandoan bottoms were more severely stripped (3 to 7 wt % 1000°F<sup>-</sup> content) than Wyodak bottoms at comparable liquefaction conditions (8 to 12 wt % 1000°F<sup>-</sup> content).

Wandoan bottoms viscosities at 3 to 8 wt % 1000°F<sup>-</sup> content ranged from 50 to 150 poise, normally sufficient to promote drawdown valve plugging in the bottoms accumulator. These Wandoan bottoms viscosities, when evaluated at ~10 wt% 1000°F<sup>-</sup> by laboratory solvent dilution experiments, are shown to be relatively low (25-50 poise) and less sensitive to coal conversion (i.e. residence time) than the lower rank coals. Thus, better operability might be expected compared to Wyodak, Big Brown and Indian Head coals. The leveling off of Wandoan bottoms viscosity at about 25 poise beyond 40 minutes residence time compared to the lower 5-10 poise values for the other coals may be a "filler" effect due to much higher ash content of the Wandoan bottoms.

Wandoan coal operations at 2500 psig experienced no problems. The bottoms viscosities from 2500 psig operations were lower than the equivalent 1500 psig operations, 12 poise versus 26 poise at 550°F, 10 sec<sup>-1</sup> shear rate, and ~10 wt% 1000°F<sup>-</sup> content.

As in previous sponsor coal screening studies, the Wandoan bottoms produced at the various study conditions were characterized using benzene and pyridine extraction procedures. The trend in 1000°F<sup>+</sup> organics distribution shown in Figure 1-12 for Wandoan bottoms is very similar to that found for the other coals. As liquefaction severity (i.e. conversion) is increased, the asphaltenes (1000°F<sup>+</sup> benzene solubles) increase whereas the preasphaltenes (pyridine solubles/benzene insolubles) decrease. For example, increasing severity by increasing residence time at 840°F/1500 psig increases the asphaltene/preasphaltene ratio from 0.29 at 25 minutes residence time to 0.81 at 100 minutes. Likewise, increasing temperature at 25 minutes/1500 psig from 840°F to 880°F increased the asphaltene/ preasphaltene ratio from 0.29 to 0.60. The highest asphaltene content was found in the bottoms produced at 2500 psig and 840°F/40 minutes. The asphaltene/preasphaltene ratio increased from 0.38 at 1500 psig/840°F/40 minutes to 1.6 at 2500 psig. As already reported, the highest 1000°F<sup>-</sup> liquid yield was also obtained at 2500 psig.

Pyridine extracted essentially all organics except the fusinite/semifusinite (inertinite) portions of the Wandoan bottoms. Thus, the Wandoan bottoms are similar to the bottoms from the American bituminous coals studied in RCLU. The lower rank subbituminous and lignitic coal bottoms studied contain heavy organics, in addition to the inertinites, which are not extracted by pyridine (July-September, 1977, EDS Quarterly Technical Progress Report [FE-2893-3], p 16).

Figure 1-12

COMPARISON OF 1000°F<sup>+</sup> ORGANICS IN RESIDUAL  
BOTTOMS FOR AUSTRALIAN WANDOAN COAL

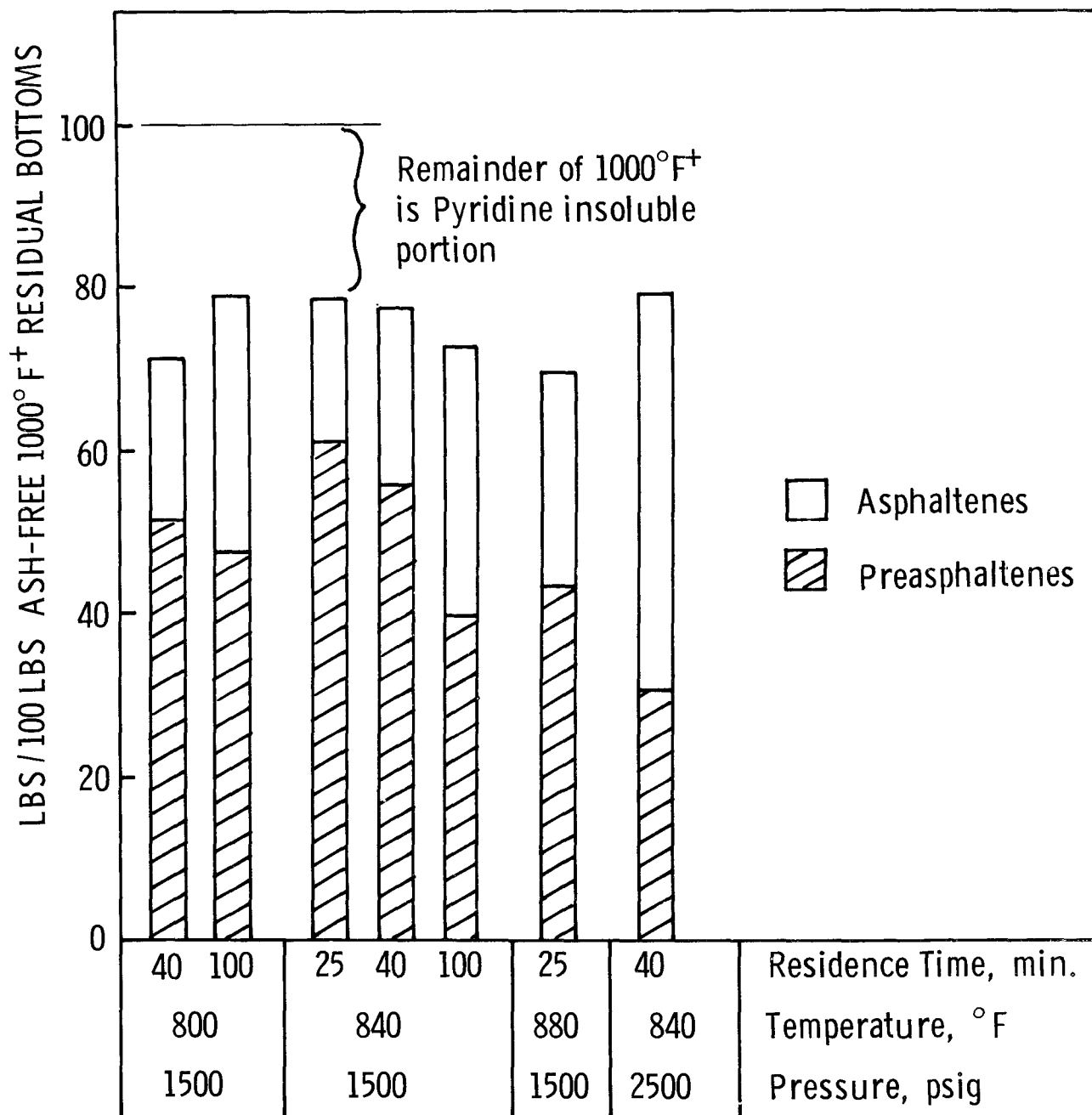


Figure 1-13 presents a summary of the 1000°F<sup>+</sup> asphaltenes in the residual bottoms for the coals studied in RCLU. As mentioned above, the trend is similar for all the coals. Asphaltenes increase in the coal bottoms with increasing liquefaction severity (i.e. conversion). These data indicate that as the 1000°F<sup>+</sup> asphaltene content becomes substantial, i.e. at 100 minute residence time in Figure 1-13, bottoms processing schemes to further convert these molecules to 1000°F<sup>-</sup> liquids may require less severe conditions than for lower residence time bottoms which contain a higher preasphaltene fraction.

● Summary of Sponsor Coal Liquefaction Reactor Solids

Inspection of the liquefaction reactor contents following liquefaction of lower rank coals in RCLU has proved important in identifying potential problems with solid deposits, e.g., the formation of calcium carbonate. Analyses of reactor residual solids following liquefaction of the higher rank coals run in RCLU were also initiated during this reporting period. The primary purpose of these studies is to elucidate differences/ similarities in reactor deposits for the different rank coals.

The reactor solids were drained from the reactor tubes and washed with toluene to remove residual oil. The reactor tube walls were then washed with methyl ethyl ketone and mechanically scraped to remove any residual scale. The reactor solids were screened to separate the +20 mesh and the -50 +100 mesh size fractions for further examination. The +20 mesh size fraction was checked for pieces of reactor scale. The -50 +100 mesh size fraction was examined for evidence of particle growth. The oolites (calcium carbonate aggregates) and iron sulfide particles which grow larger than the RCLU feed coal top size (100 mesh) are found in this size fraction for the lower rank coals.

Table 1-5 presents summarized data from liquefaction reactor inspections for the different rank coals run in RCLU. Detailed analyses for Wandoan reactor solids are given in Appendix Table A-6 of this report. The major crystalline species found in the reactor solids were essentially the same for all the coals: calcium carbonate (calcite and vaterite), iron sulfide, quartz, and kaolinite. This is consistent with the mineral elements contained in these coals, as shown in Table 1-6. Indian Head lignite also formed  $\text{Na}_2\text{Mg}(\text{CO}_3)_2$  during liquefaction, presumably because this coal contains much more sodium than the other coals. For all coals but Monterey coal, growth of iron sulfide and calcium carbonate occurs during liquefaction. Monterey coal exhibits iron sulfide growth, but no evidence of calcium carbonate growth was found. Wall scale accumulation and composition data for these coals are shown in Table 1-7.

The impact of wall scale and aggregate growth on operability should be small for the higher rank Monterey, Burning Star, Ireland, and Arkwright coals. The aggregates comprise less than 15% of the total reactor solids and wall scale growth rates are insignificant compared

Figure 1-13

COMPARISON OF ASPHALTENES IN THE RESIDUAL  
BOTTOMS OF COALS STUDIED IN RCLU

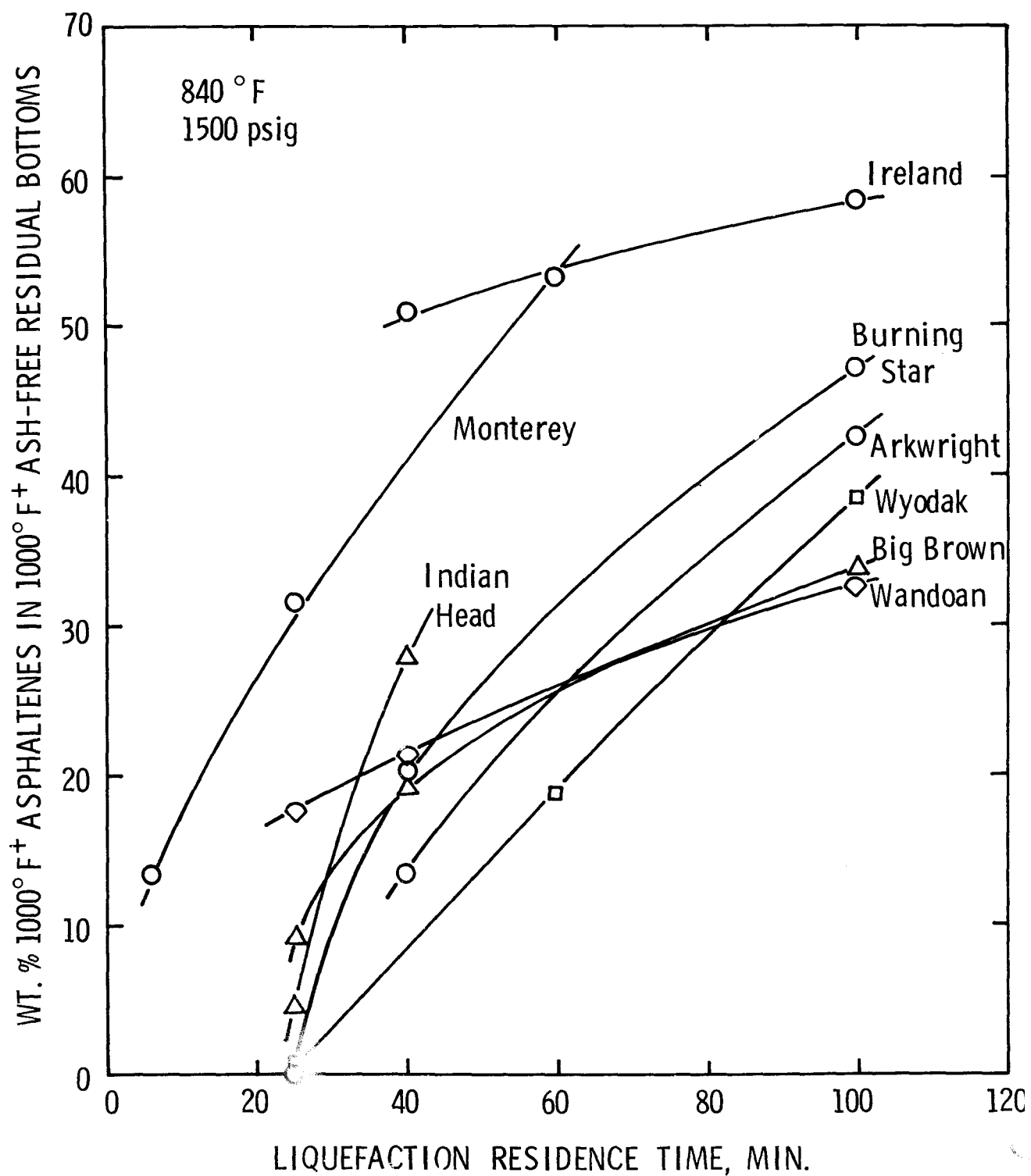


TABLE 1-5

## COMPARISON OF LIQUEFACTION RESIDUAL REACTOR SOLIDS FROM DIFFERENT RANK COALS RUN IN RCLU

Coal	Illinois No. 6 Bituminous		Pittsburgh Seam Bituminous		Australian Black	Wyoming Subbituminous	Texas Lignite	North Dakota Lignite
Mine	Monterey No. 1	Burning Star No. 2	Ireland	Arkwright	Wandoan	Wyodak	Big Brown	Indian Head
Reactor Solids <sup>1</sup>								
Accumulation, Lbs/100 Lbs Coal Fed	0.01-0.10	0.03-0.20	0.10-0.21	0.20-0.29	0.04-1.1	0.35-0.57	0.49-2.0	0.22-1.1
Composition, Ranges, Wt%								
Ash	52-69	24-71	89-90	54-73	60-76	56-70	78-82	57-67
CaCO <sub>3</sub>	4-9	7-50	5-10	3-9	43-57	24-83	67-74	17-49
Carbon	17-37	9-67	3-4	13-37	6-15	5-26	3-4	9-22
CaCO <sub>3</sub> Accumulation (Avg), Lbs/Ton of Coal Fed	0.18	0.85	0.26	0.32	3.3	5.6	12.0	6.8
Major Crystalline Species	Calcite FeS SiO <sub>2</sub>	Calcite FeS Kaolinite SiO <sub>2</sub> CaSO <sub>4</sub>	Calcite FeS Kaolinite SiO <sub>2</sub>	Calcite FeS Kaolinite SiO <sub>2</sub>	Calcite Vaterite FeS Kaolinite SiO <sub>2</sub>	Calcite Vaterite FeS SiO <sub>2</sub>	Calcite Vaterite FeS SiO <sub>2</sub>	Calcite Na <sub>2</sub> Mg(CO <sub>3</sub> ) <sub>2</sub> FeS SiO <sub>2</sub>

<sup>1</sup>Toluene washed

TABLE 1-6

## MAJOR MINERAL ELEMENTS IN RCLU COALS

<u>Coal</u>	<u>Illinois No. 6</u> <u>Bituminous</u>		<u>Pittsburgh Seam</u> <u>Bituminous</u>		<u>Australian</u> <u>Black</u>	<u>Wyoming</u> <u>Subbituminous</u>	<u>Texas</u> <u>Lignite</u>	<u>North Dakota</u> <u>Lignite</u>
<u>Mine</u>	<u>Monterey</u> <u>No. 1</u>	<u>Burning Star</u> <u>No. 2</u>	<u>Ireland</u>	<u>Arkwright</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big Brown</u>	<u>Indian Head</u>
<u>Element,</u> <u>Wt% on Dry Coal</u>								
Si	2.2	2.3	1.7	1.5	5.3	1.3	2.9	0.88
Fe	1.2	0.86	2.0	0.97	0.33	0.29	0.63	0.77
Al	0.88	1.0	0.89	0.95	3.5	0.95	1.2	0.53
Ca	0.20	0.39	0.17	0.24	0.80	1.4	2.2	1.3
Mg	0.07	0.06	0.04	0.05	0.22	0.33	0.32	0.31
Na	0.05	0.05	0.03	0.07	0.13	0.06	0.04	0.84

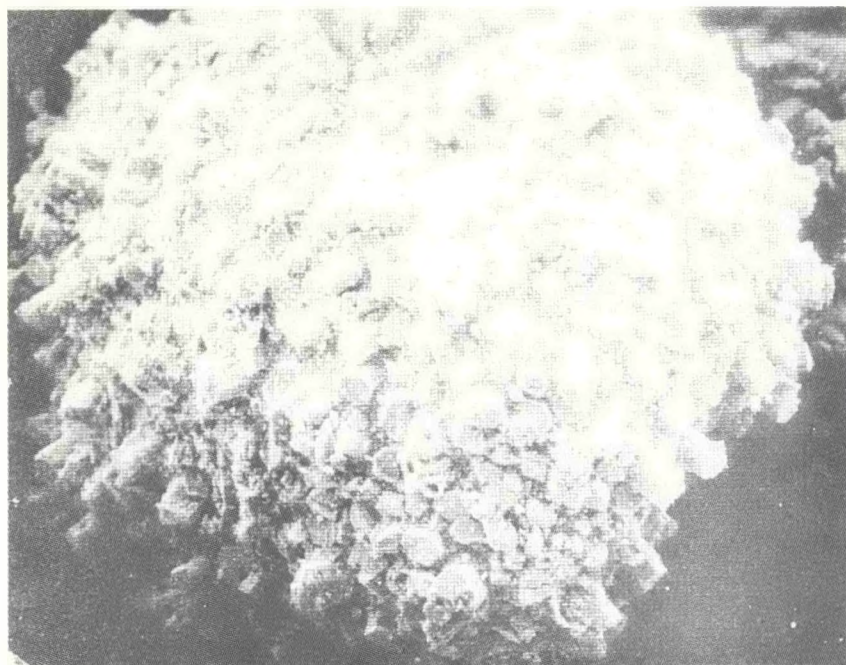


TABLE 1-7

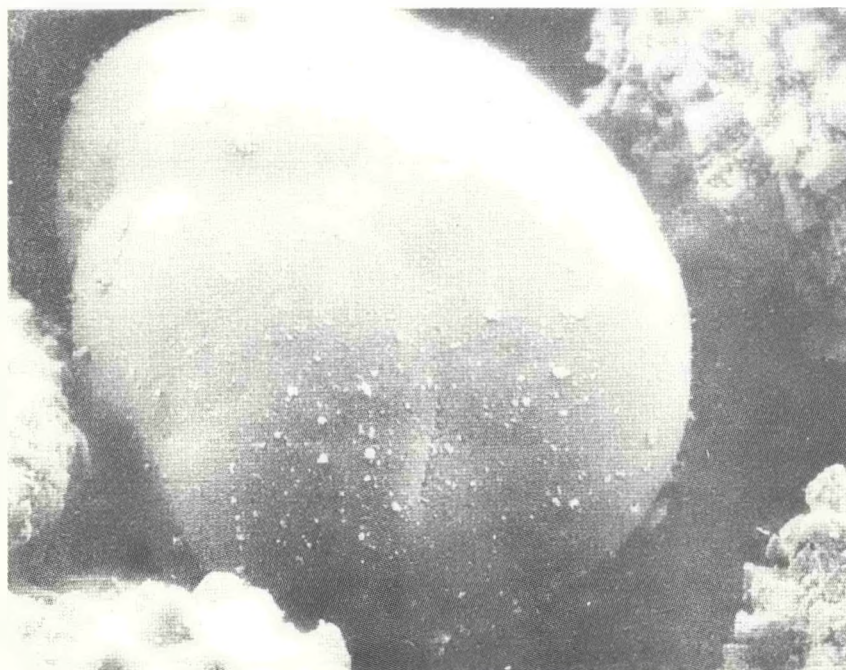
## COMPARISON OF REACTOR WALL SCALE FORMED DURING LIQUEFACTION OF DIFFERENT RANK COALS IN RCLU

Coal	Illinois No. 6 Bituminous		Pittsburgh Seam Bituminous		Australian Black	Wyoming Subbituminous	Texas Lignite	North Dakota Lignite
Mine	Monterey No. 1	Burning Star No. 2	Ireland	Arkwright	Wandoan	Wyodak	Big Brown	Indian Head
Scale Accumulation, Lbs/100 Lbs Coal Fed	Trace-0.001	0.00005-0.001	Trace-0.001	Trace-0.008	0.005-0.006	0.002-0.007	0.002-0.010	0.005-0.035
Composition, Wt%								
Ash (by TGA)	61-96	69-93	78-86	47-61	56-74	59-63	52-86	57-80
CaCO <sub>3</sub> (by TGA)	5-14	8-25	9-41	57-73	36-48	71-85	53-86	64-84
Major Crystalline Species (by X-ray Diffraction)	FeS SiO <sub>2</sub>	Calcite FeS	Calcite FeS	Calcite FeS SiO <sub>2</sub>	Calcite Vaterite FeS SiO <sub>2</sub> Kaolinite	Calcite Vaterite FeS SiO <sub>2</sub>	Calcite Vaterite FeS SiO <sub>2</sub>	Calcite FeS SiO <sub>2</sub>
Major Elements, Wt% (by X-ray Scan of Cross-Section)								
Al	2-4	0.0-4	1-2	0.1-1	0.4-4		0.1-5	
Ca	2-7	0.1-13	17-34	3-43	0.5-30	Not	0.8-38	Not
Ti	0.2-7	0.0-12	2-4	0.0-0.6	0.1-2	Analyzed	0.0-0.7	Analyzed
Fe	5-30	0.9-28	1-2	0.0-24	2-44		1-48	
S	3-25	0.7-18	0.5-1	0.1-20	0.1-26		0.2-34	
Si	2-27	0.1-4	0.4-1	0.1-0.8	0.6-6		0.4-5	

SCANNING ELECTRON PHOTOGRAPHS OF OOLITES  
FORMED DURING LIQUEFACTION OF WANDOAN COAL



Surface consists of iron sulfide crystals. Growth very similar to that observed with Wyodak coal.

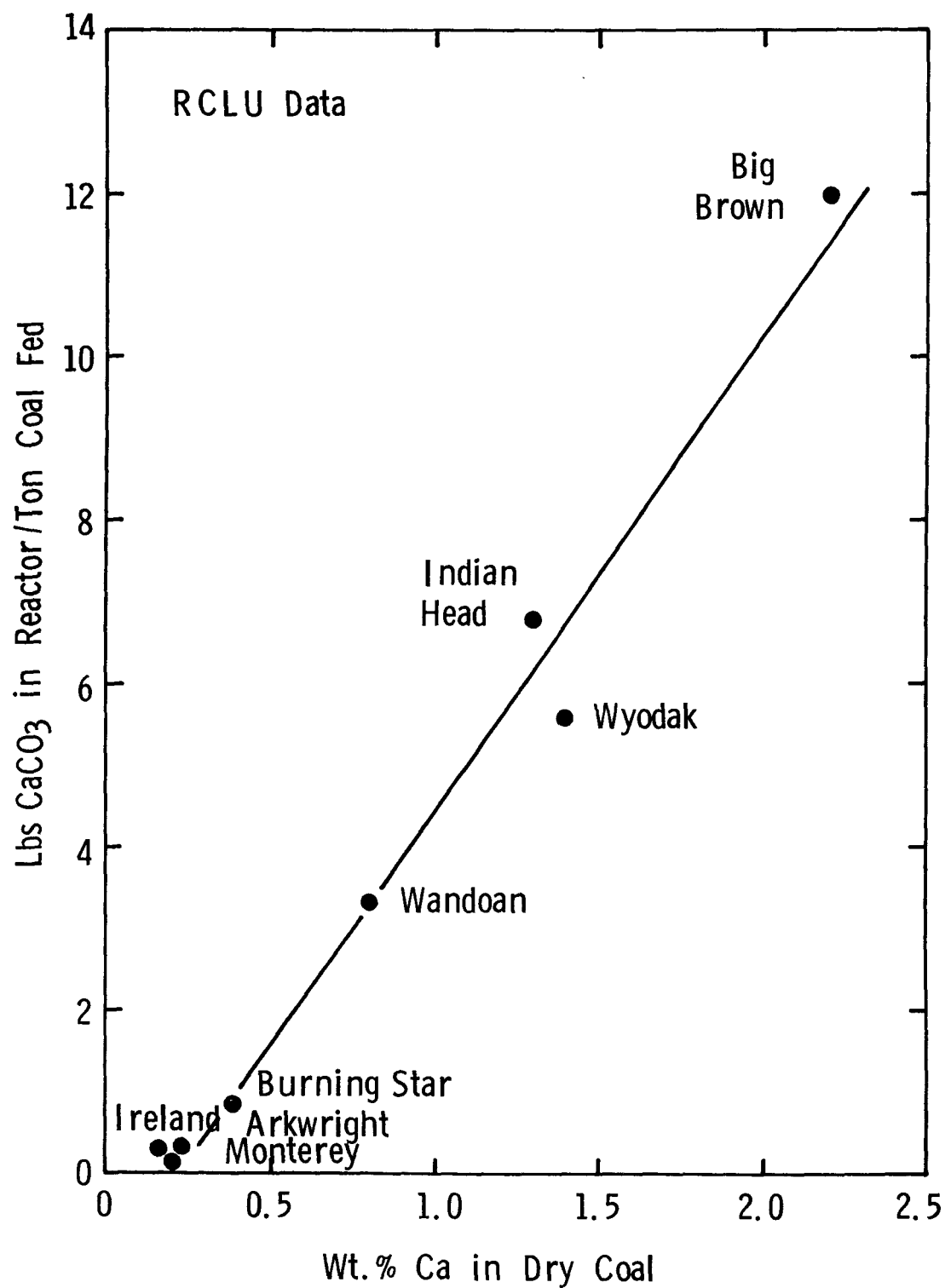


Surface very smooth; consists of calcium carbonate. Type of Oolite also prevalent in Wyodak Reactor solids

— 100 mesh —  
(150 $\mu$ )

Figure 1-15

CALCIUM CARBONATE DEPOSITION VERSUS  
CALCIUM CONTENT IN DIFFERENT RANK COALS



to the lower rank Wyodak, Big Brown, and Indian Head coals. Reactor solids from Wandoan coal are very similar to those formed from Wyodak coal (January, 1976-June, 1977, EDS Final Technical Progress Report [FE-2353-20], p. 46). Figure 1-14 shows the types of oolites (calcium carbonate aggregates) which formed during liquefaction of Wandoan coal. One type also very prevalent in Wyodak reactor solids has a very smooth surface of calcium carbonate. Less prevalent for both coals is the oolite which contains iron sulfide crystals growing over a calcium carbonate substrate.

The amount of calcium carbonate formed during liquefaction is related to the calcium content in the feed coal. Figure 1-15 shows the lbs of calcium carbonate accumulated in the reactor per ton of coal fed as a function of calcium in the coal. The linear relationship indicates that about the same proportion of calcium is available for ion-exchange in all the lower rank coals studied to date.

Based on the accumulation shown in Figure 1-15, some operability problems with calcium carbonate deposition could be expected with Wandoan coal. Because these deposits from Wandoan, Wyodak, Big Brown, and Indian Head are very similar, the solutions used for reducing/eliminating calcium carbonate deposition for one coal (e.g. sulfur dioxide pretreatment discussed in Section 1.3 of this report) will probably apply to the others as well.

### 1.3 Sulfur Dioxide Pretreatment of Wyoming Coal

Earlier RCLU operations with sulfur dioxide pretreated Wyoming coal from the Wyodak mine were not successful in completely eliminating the formation of calcium carbonate deposits during liquefaction (January-March, 1978, EDS Quarterly Technical Progress Report [FE-2893-12]). Bench studies indicated that the chemistry requires the presence of oxygen to make the treat effective. In the current test, performed during the third quarter of 1978, Wyodak coal was first treated with sulfur dioxide under conditions comparable to those for the previous RCLU study. Following the sulfur dioxide treat step, the coal was exposed to a dilute oxygen atmosphere. The effect of this oxidation step on the deposition of calcium carbonate, as well as conversion and yields, was then investigated in a continuous flow unit (RCLU-1).

#### ● S<sub>0</sub>2 Pretreatment Step

The Wyodak coal was pretreated with sulfur dioxide under conditions comparable to those for the previous RCLU study. Wet coal was pretreated in the Coal Preparation Unit Anex (CPU-A) drier. The drier, containing -1/4" mesh coal, was partially evacuated prior to introduction of the sulfur dioxide. A pressure of 15 psig of sulfur dioxide was maintained for the five days of treat.

- Oxidation Step

Bench studies showed that the unstable calcium sulfite salts formed during sulfur dioxide pretreating are stabilized when exposed to oxygen. Oxygen reacts with the sulfite salt to form calcium sulfate which is stable at liquefaction conditions. RCLU feed coal was exposed to a nitrogen/air mixture with a 4% oxygen concentration. The amount of oxygen used for the oxidation step was 10% greater than the stoichiometric need based on calcium in the feed coal.

- Results of SO<sub>2</sub>/Air Pretreatment

The results show that sulfur dioxide/air pretreatment has been successful in substantially reducing the formation of calcium carbonate deposits formed during liquefaction of Wyodak coal. A summary of RCLU operations with sulfur dioxide treated coal is presented in Table 1-8.

The data show a substantial reduction in wall scale formation for the sulfur dioxide/air pretreated runs over that of the untreated and previous sulfur dioxide pretreated cases. The major compounds in the scale that did form from the SO<sub>2</sub>/air pretreated coal were calcium carbonate and iron sulfide. More detailed solids analyses are presented in Appendix Table A-6 of the October-December, 1978, EDS Quarterly Technical Progress Report [FE-2893-25].

The data also show that there is a substantial reduction in calcium carbonate in the reactor solids for the sulfur dioxide/air treated coal runs. The amount of total solids produced per ton of coal are comparable for the untreated and SO<sub>2</sub>/air pretreated runs. However, the solids from the sulfur dioxide/air pretreated runs were high in carbon content, whereas the solids from the untreated runs were primarily calcium carbonate.

- Conversion and Yields

Sulfur dioxide/air pretreatment of Wyodak coal shows no detrimental effects on conversion or liquid yields. A comparison of yields for sulfur dioxide/air pretreated coal with untreated coal is presented in Table 1-9. The increase in conversion seen in the sulfur dioxide/air pretreated runs is due primarily to increased water and hydrogen sulfide yields. The hydrocarbon yields are comparable, with a slight increase of 0.5 percent of treated coal over the untreated. There was a slightly higher gas yield and lower C<sub>4</sub>-1000°F liquid yield for the untreated coal. Overall, the impact of sulfur dioxide/air pretreatment on Wyodak coal yields is small.

TABLE 1-8

SUMMARY OF RCLU OPERATIONS WITH SO<sub>2</sub>-PRETREATED WYODAK COAL

Liquefaction Conditions: 840°F  
 1500 psig  
 60 minutes  
 1.6 solvent/coal ratio  
 4 wt% H<sub>2</sub> on dry coal

<u>Coal Batch</u>	<u>Untreated</u>	<u>SO<sub>2</sub>- Pretreated</u>	<u>SO<sub>2</sub>/Air Pretreated</u>
<u>RCLU-1 Run No.</u>	1-3, 17-19, 23, 24, 27, 30		58, 59
<u>RCLU-2 Run No.</u>		1-4	
<u>Reactor Wall Scale</u>			
Lbs Wall Scale/Ton Coal	0.04-0.14	0.02-0.26	Trace-0.002
CaCO <sub>3</sub> in Scale, Wt. %	70-85	2-63	0.7-60
Lbs CaCO <sub>3</sub> in Scale/Ton Coal	0.02-0.12	0.003-0.038	Trace-0.0009
<u>Reactor Solids</u>			
Lbs Solids/Ton Coal	7-11	---	4.8-28
CaCO <sub>3</sub> in Solids, Wt. %	24-83	---	0.3-2.3
Lbs CaCO <sub>3</sub> in Solids/Ton Coal	1.6-9.4	---	0.02-0.08
Ca in Reactor/Ca in Feed Coal, Wt. %	2-7	---	0.078-0.35

TABLE 1-9

EFFECT OF SULFUR DIOXIDE/AIR TREATING ON  
WYODAK COAL LIQUEFACTION YIELDS

Liquefaction Conditions: 840°F  
60 Minutes Nominal Residence Time  
1500 psig  
1.6 Solvent/Coal Ratio  
4 wt% H<sub>2</sub> on Dry Coal

<u>Yield Periods</u>	<u>Untreated Wyodak Coal</u>	<u>Sulfur Dioxide/Air Treated Wyodak Coal</u>
	575,576	569,570
	577,578	571,572
	579	573,574
 <u>Liquefaction Yields, Lbs/100 Lbs Dry Coal</u>		
H <sub>2</sub>	-4.3	-4.2
H <sub>2</sub> O	13.7	14.5
CO <sub>x</sub>	7.2	7.2
NH <sub>3</sub>	0.4	0.4
H <sub>2</sub> S	0.5	1.3
C <sub>1</sub> -C <sub>3</sub> Gas	11.0	9.2
C <sub>4</sub> -1000°F Liquids	25.7	28.0
1000°F+ Bottoms	45.8	43.6
 C <sub>1</sub> -1000°F Hydrocarbons	 36.7	 37.2

● Unit Operations

Operations with the sulfur dioxide/air treated Wyodak coal were very similar to operations with untreated Wyodak coal. The run lengths for the pretreated coal were comparable to those with untreated coal. The viscosities of the liquefaction bottoms from the treated coal were somewhat lower (17 poise at 550°F, 10 sec<sup>-1</sup> shear rate) than those for the untreated Wyodak coal (37 poise at 550°F, 10 sec<sup>-1</sup> shear rate). This is primarily due to the higher conversion of the treated coal (July, 1977-June, 1978, EDS Annual Technical Progress Report [FE-2893-17], Table 1-11). At comparable conversions, the bottoms viscosities for treated and untreated coals would be nearly the same.

#### 1.4 Liquefaction Bottoms Recycle Studies

During the third quarter of 1978 a simulated bottoms recycle study with Monterey Mine No. 1 coal was performed in RCLU-1. This study was a follow-on to earlier bench tubing bomb studies which demonstrated substantial conversion of Monterey bottoms in admixture with coal. The RCLU study showed a similar bottoms conversion increase in a continuous flow unit with Monterey coal, confirming the tubing bomb data (October-December, 1978, EDS Quarterly Technical Progress Report [FE-2893-25]).

Recent bench tubing bomb studies showed similar enhanced conversion for liquefaction of Wyodak bottoms. During the fourth quarter of 1978 a study was conducted in RCLU-1 to evaluate the effect of recycling bottoms on EDS liquefaction yields and operability for Wyodak coal. The results of the simulated bottoms recycle study with Monterey coal are reviewed and compared with the results from the Wyodak bottoms recycle study.

##### ● Study Conditions

In these studies, liquefaction bottoms were fed in a once-through fashion rather than in an actual recycle mode. This was done for two reasons. Once-through operation can be accomplished rapidly without unit modifications. Secondly, once-through bottoms would be expected to show the maximum yield incentive for bottoms recycle because subsequent bottoms conversion per pass would be less. A schematic of the RCLU bottoms recycle operation employed is shown in Figure 1-16.

The bottoms used as once-through feed were vacuum tower bottoms from CLPP Monterey coal and Wyodak coal operations. The CLPP bottoms were ground to a -100 mesh particle size and mixed with fresh coal and recycle solvent. The variables investigated in the Monterey bottoms recycle study were bottoms/coal ratio and liquefaction residence time. Solvent/coal ratio and liquefaction residence time were studied in the Wyodak bottoms recycle study. The conditions for each study are given in Table 1-10.



FIGURE 1-16

RCLU ONCE-THROUGH SIMULATION OF BOTTOMS RECYCLE

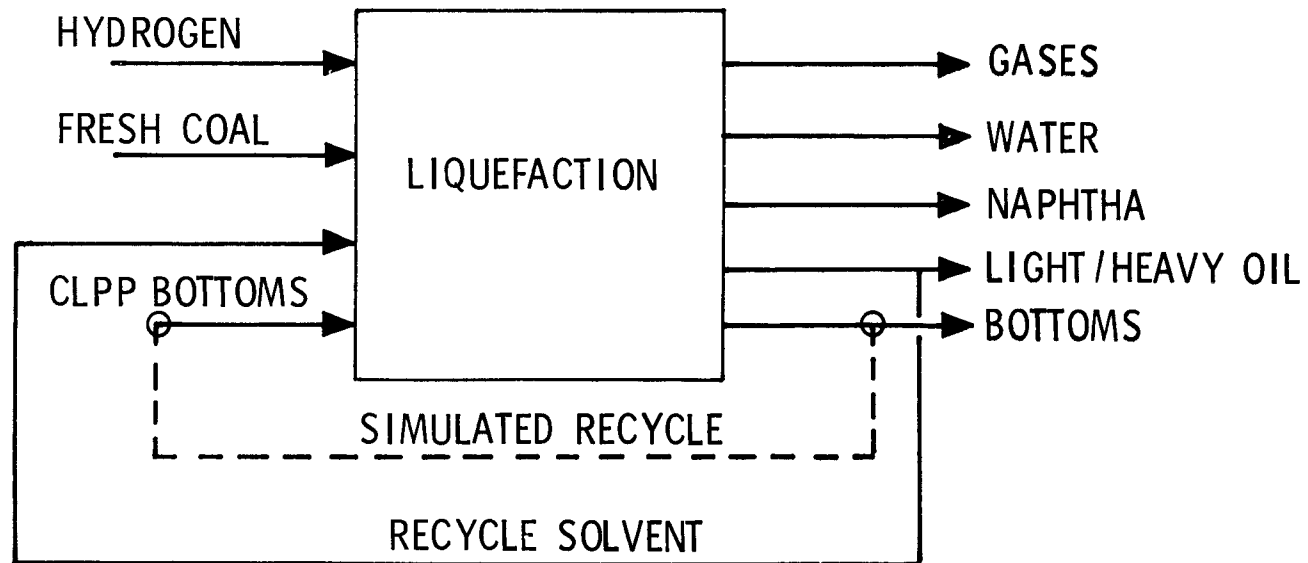


Table 1-10

LIQUEFACTION BOTTOMS RECYCLE STUDY CONDITIONS IN RCLUMonterey Coal

	<u>40-Minute CLPP Bottoms</u>			
Temperature, °F	840	840	840	840
Pressure, psig	1500	1500	1500	1500
Residence Time, Minutes	40	40	40	100
Bottoms-to-Coal Ratio	1:3	1:2	1:1	1:2
Solvent-to-Coal Ratio	1.6:1	1.6:1	2.1:1	1.6:1

Wyodak Coal

	<u>60 Minute CLPP Bottoms</u>			<u>25-Minute CLPP Bottoms</u>	
Temperature, °F	840	840	840	840	840
Pressure, psig	1500	1500	1500	1500	1500
Residence Time, Minutes	60	60	60	25	25
Solvent/Coal/Bottoms	1.6/0/1	1.6/1/0.5	2.4/1/0.5	1.6/1/0.5	2.4/1/0.5

The vacuum bottoms used were produced in CLPP at 840°F/1500 psig and the nominal residence times shown.

In addition, coal-only runs were made with both coals at bottoms recycle study conditions to provide liquefaction data for comparison with the bottoms recycle data.

- Effect of Bottoms-to-Coal Ratio on Monterey Bottoms Yields

The effect of bottoms-to-coal ratio on the liquefaction yields for Monterey once-through bottoms recycle are summarized in Figures 1-17 through 1-21. Detailed yields are presented in Appendix Table A-8 of the October-December, 1978 EDS Quarterly Technical Progress Report [FE-2873-25]. These yields represent the net yields on dry coal with the bottoms feed considered as an internal recycle stream. On this basis, the net 1000°F<sup>+</sup> bottoms yield decreases significantly as the bottoms-to-coal ratio increases, as shown in Figure 1-17. The C<sub>4</sub>-1000°F liquid yield shown in Figure 1-18 increases significantly as the bottoms-to-coal ratio increases. Estimates of the actual conversion of the bottoms show little variation with bottoms-to-coal ratio. This is shown in Table 1-14 and discussed later.

The liquid product selectivity is shown in Figure 1-19. The amounts of C<sub>4</sub>-400°F naphtha and 400-700°F liquid increase as bottoms-to-coal ratio increases, while the amount of 700-1000°F remains about constant. However, the percent of C<sub>4</sub>-400°F naphtha in the total C<sub>4</sub>-1000°F liquid remains constant at about 60 wt% for the conditions studied. The 700-1000°F liquid fraction decreases from 19.4 wt% for coal-only to

FIGURE 1-17

EFFECT OF BOTTOMS-TO-COAL RATIO ON 1000°F+ YIELD

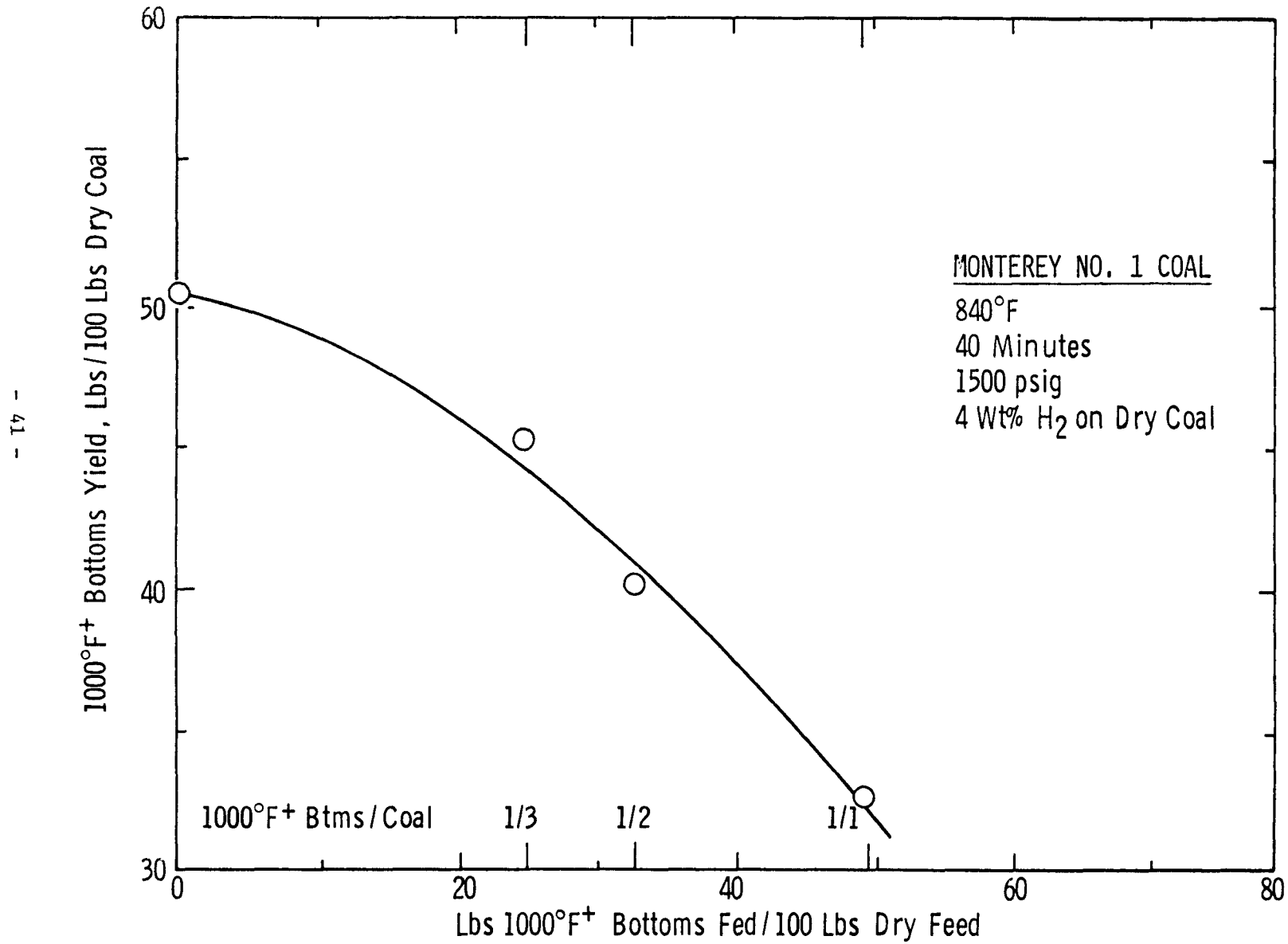


FIGURE 1-18

EFFECT OF BOTTOMS-TO-COAL RATIO ON C<sub>4</sub>-1000°F LIQUID YIELD

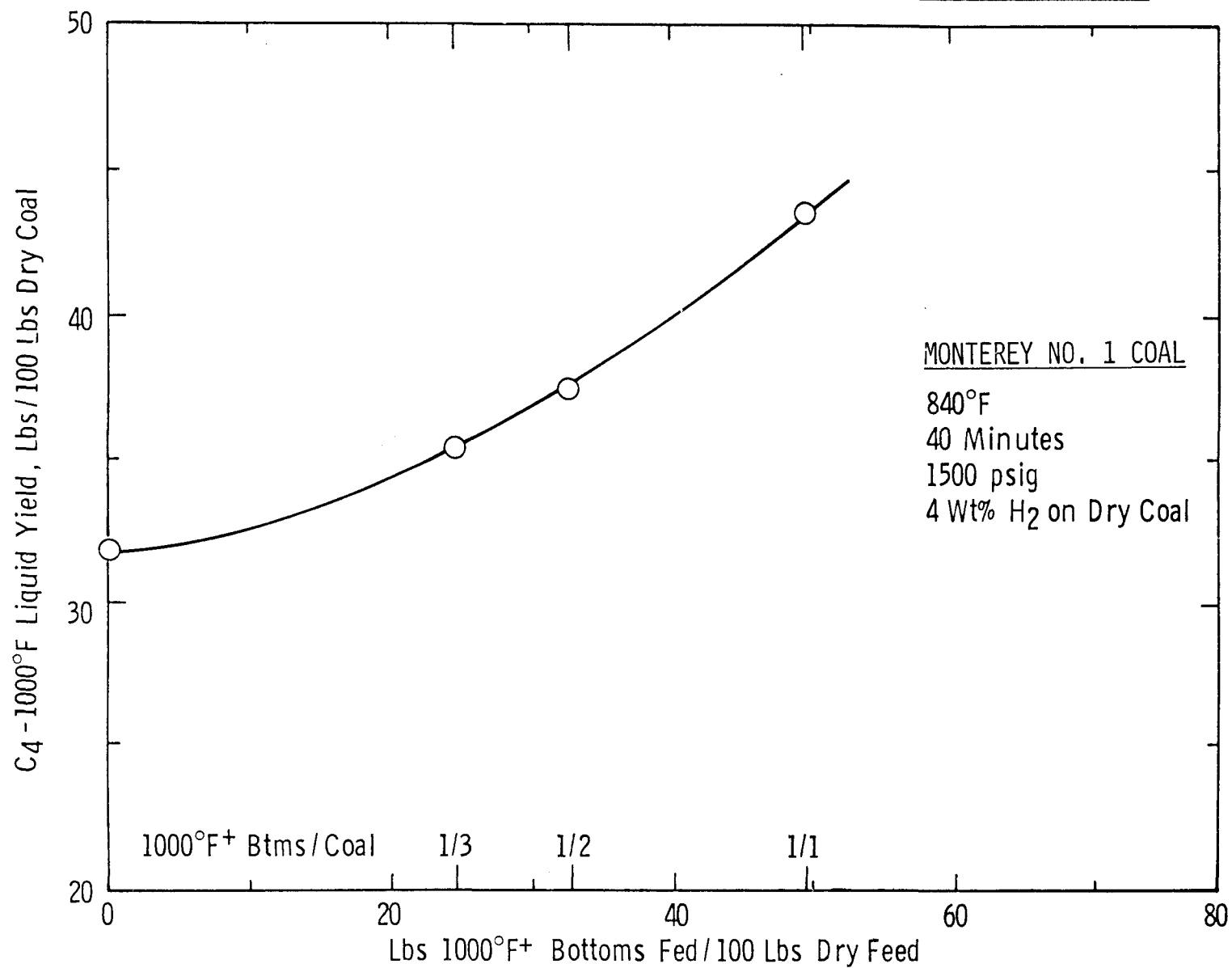
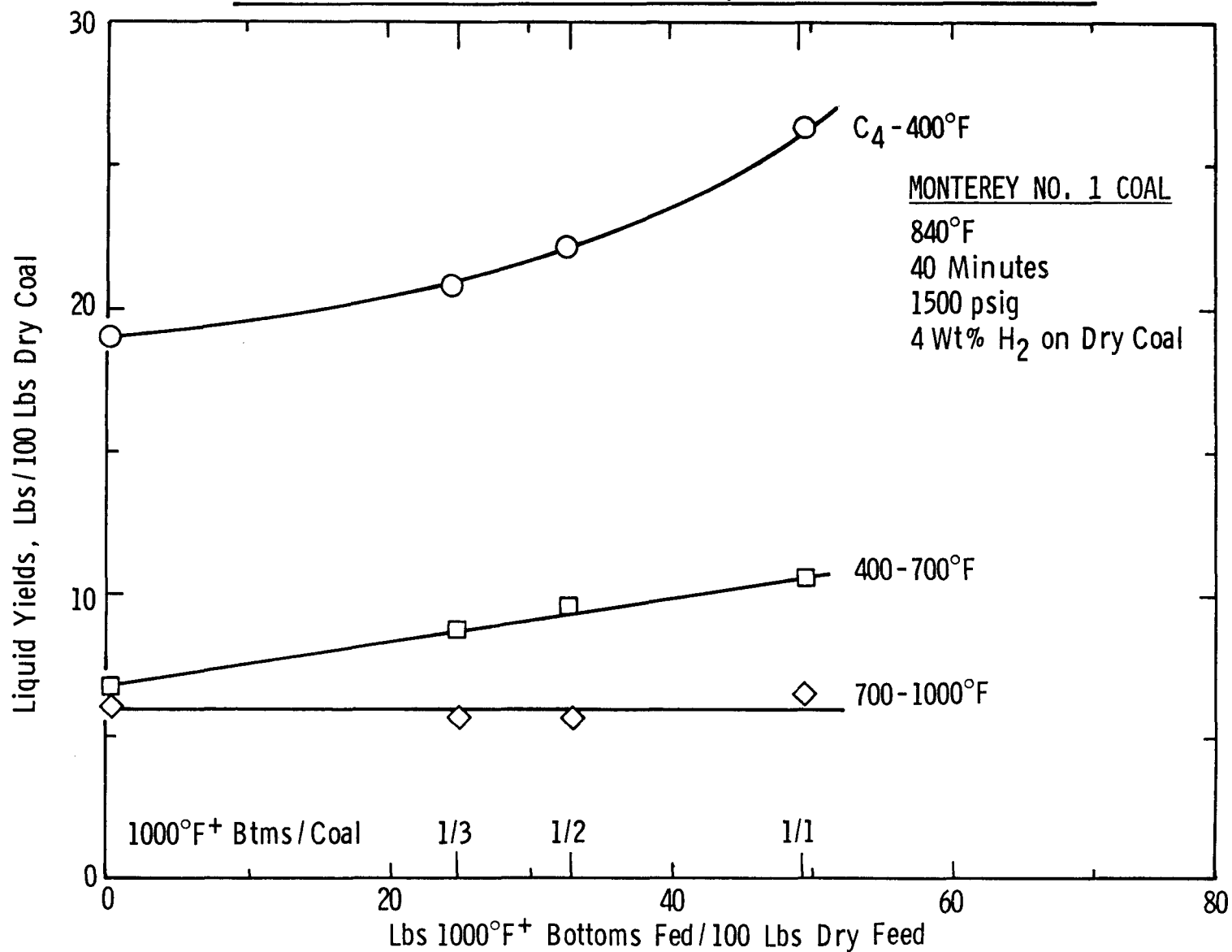


FIGURE 1-19

EFFECT OF BOTTOMS-TO-COAL ON LIQUID PRODUCT SELECTIVITY



15.5 wt% at 1:1 bottoms-to-coal ratio, while the 400-700°F liquid fraction increases from 20.7 wt% for coal-only to 24.5 wt% at 1:1 bottoms-to-coal ratio. Thus, increasing the bottoms-to-coal ratio, in addition to producing more liquids, directionally produces a lighter solvent stream for once-through Monterey bottoms recycle.

The hydrocarbon gas yields are shown in Figure 1-20. Total C<sub>1</sub>-C<sub>3</sub> gas make increases with increasing bottoms-to-coal ratio. The percent of methane in the total gas increases slightly with increasing bottoms-to-coal ratio, while ethane and propane show less response.

The chemical gas yields are essentially independent of bottoms-to-coal ratio (5.3 to 6.2 lbs/100 lbs dry coal), which means very little of these gases produced from the bottoms. Carbon oxide, ammonia, and hydrogen sulfide yields are less than 1 wt% on 1000°F<sup>+</sup> bottoms fed.

Water make also increases only slightly with increasing bottoms-to-coal ratio from 9.3 to 11.1 for 1:3 and 1:1 bottoms-to-coal ratio, respectively. The hydrogen consumption also increases as bottoms-to-coal ratio increases, as shown in Figure 1-21. Analyses of these data imply that for once-through bottoms there is no incremental benefit/debit in hydrogen consumption for the slight change in liquid product selectivity noted previously with increasing bottoms-to-coal ratio. This is because the hydrogen consumption per pound of liquid made is about constant.

• Effect of Solvent-to-Coal Ratio on Wyodak Bottoms Recycle Yields

The detailed liquefaction yields for the Wyodak once-through bottoms recycle study are presented in Appendix A, Table A-7. These yields are summarized in Table 1-11 below.

The results show a strong effect of solvent-to-solids ratio with Wyodak coal both at 60 minutes and 25 minutes residence times. At both residence times, the increase in C<sub>4</sub>-1000°F liquid yield at 1.6/1/0.5 solvent-to-coal-to-bottoms (1.07 solvent-to-solids) over coal-only liquid yield was small. The net bottoms conversion calculated by backing out the coal-only yields from the coal plus bottoms yields was only 5.6 lbs/100 lbs 1000°F<sup>+</sup> bottoms fed at 60 minutes residence time. Virtually all of this was conversion to C<sub>1</sub>-C<sub>3</sub> gas. At 25 minutes residence time, net bottoms conversion was 14.4 lbs/100 lbs 1000°F<sup>+</sup> bottoms fed of which 4.6 lbs/100 lbs was C<sub>1</sub>-C<sub>3</sub> gas and 6.3 lbs/100 lbs was C<sub>4</sub>-1000°F liquids.

It was hypothesized that this low bottoms conversion might be due to insufficient solvation of the bottoms and/or a deficiency of donor hydrogen. To test this hypothesis, RCLU runs were made with 25- and 60-minute CLPP bottoms using 50% more recycle solvent, i.e. 2.4/1/0.5 recycle solvent-to-coal-to-bottoms. This increased the solvent-to-solids ratio from 1.07/1 to 1.6/1. This increase in solvent-to-solids ratio significantly increased the conversion and liquid yields with Wyodak bottoms recycle, as shown in Table 1-11. At 60 minutes residence time,

FIGURE 1-20

EFFECT OF BOTTOMS-TO-COAL RATIO ON HYDROCARBON GAS YIELD

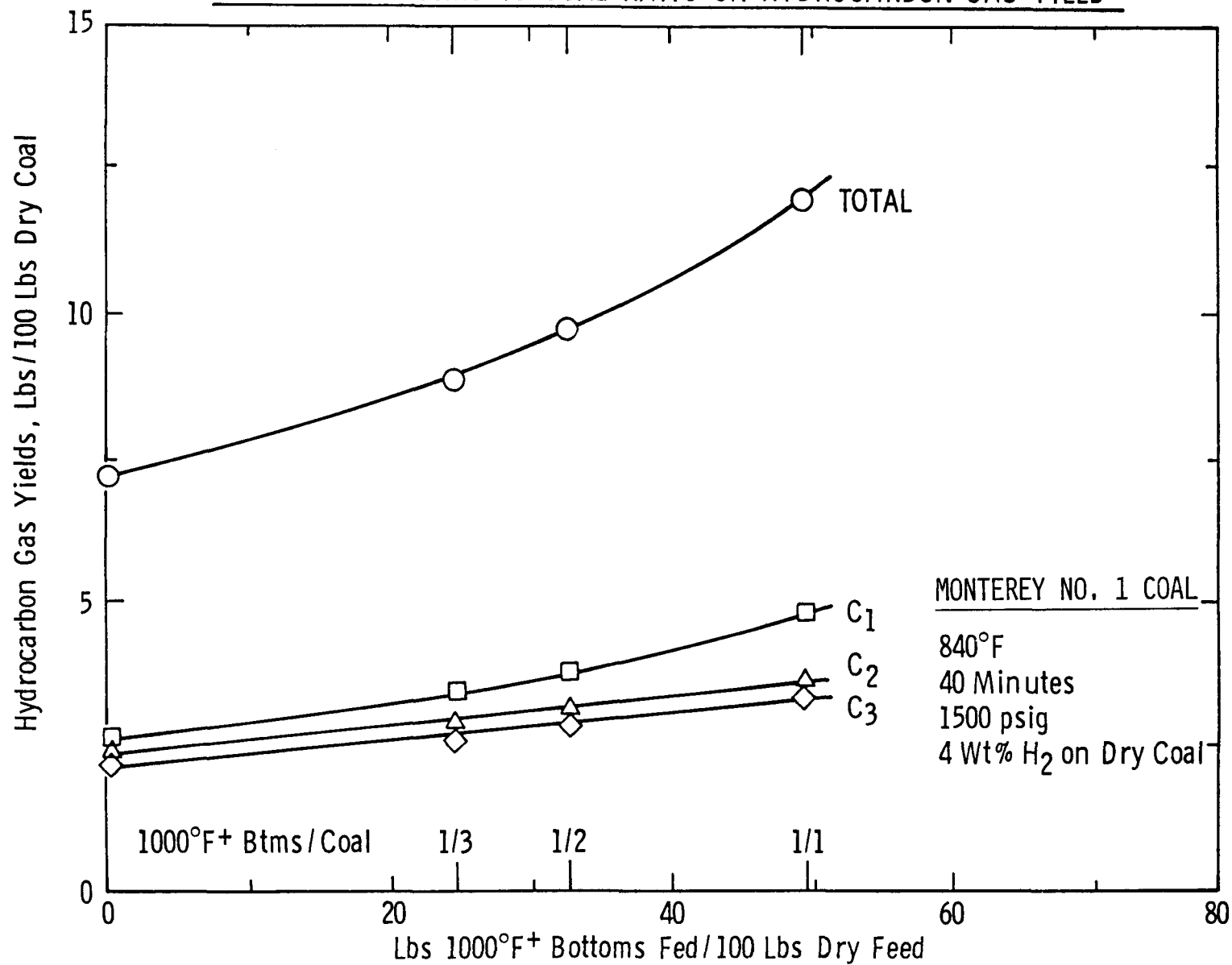


FIGURE 1-21

EFFECT OF BOTTOMS-TO-COAL RATIO ON HYDROGEN CONSUMPTION

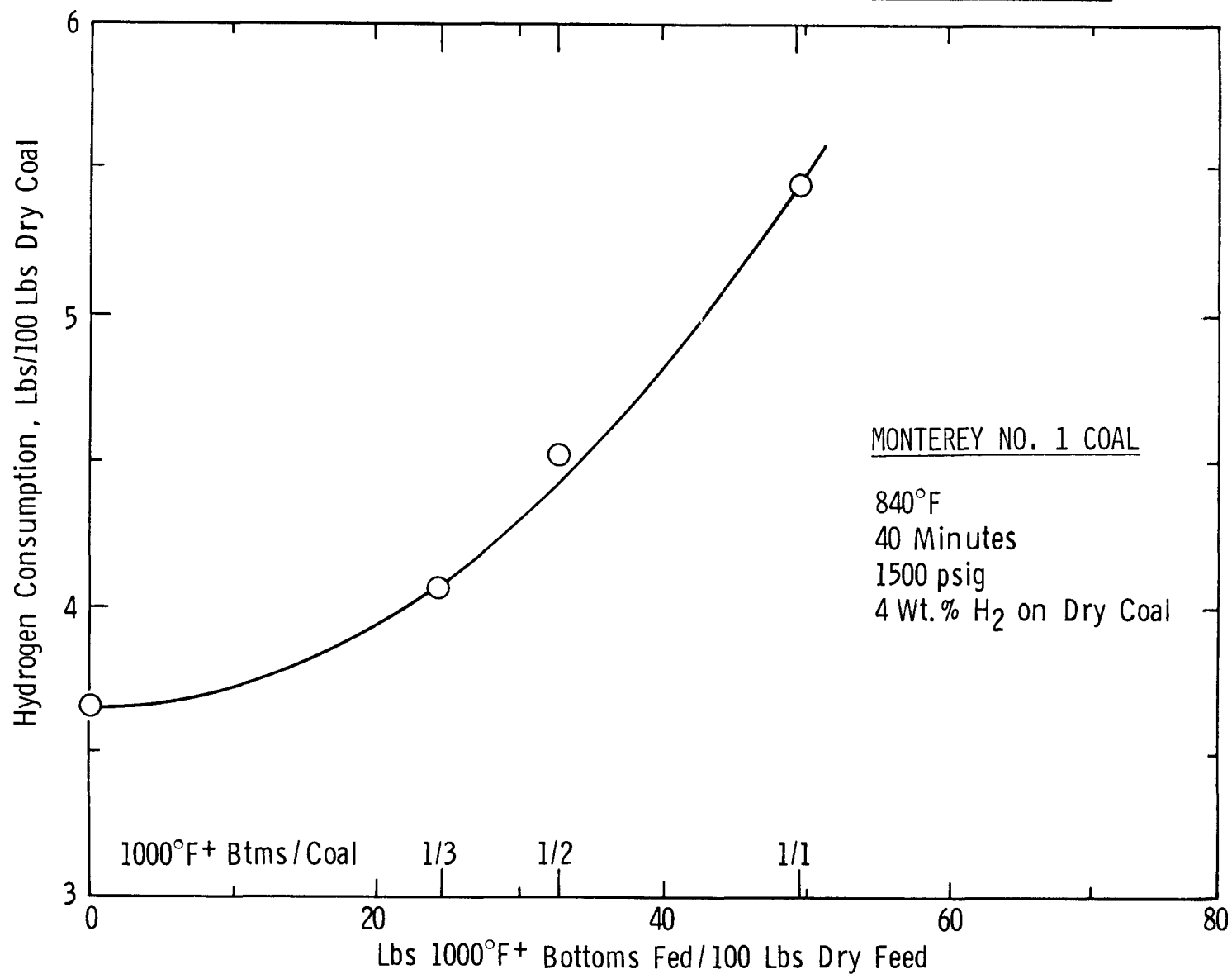




TABLE 1-11

WYODAK BOTTOMS RECYCLE STUDY YIELDS

- 840°F
- 1500 psig
- 4 wt % H<sub>2</sub> on coal

	<u>CLPP 60-Minute Bottoms</u>			<u>CLPP 25-Minute Bottoms</u>		
<u>Nominal RCLU Residence Time, Minutes</u>	-----60 -----			----- 25 -----		
<u>Solvent/Coal/Bottoms</u>	1.6/1/0	1.6/1/0.5	2.4/1/0.5	1.6/1/0	1.6/1/0.5	2.4/1/0.5
<u>Yields, Lbs/100 Lbs Dry Coal</u>						
H <sub>2</sub>	-3.9	-4.2	-6.0	-3.0	-4.1	-4.5
H <sub>2</sub> O + Chemical Gases	21.7	21.8	23.0	20.2	23.4	22.2
C <sub>1</sub> -C <sub>3</sub>	9.5	12.4	12.1	6.1	8.7	8.2
C <sub>4</sub> -1000 F	28.1	28.3	39.0	25.0	28.5	37.4
1000°F <sup>†</sup>	44.6	41.7	31.9	51.6	43.5	36.7
<u>Yield Periods</u>	606-608	603-605	617-620	609 614-616	610-613	623 624

the C<sub>4</sub>-1000°F liquid yield increased by 39% from coal-only yield (28.1 to 39.0 lbs/100 lbs dry coal). Likewise, at 25 minutes residence time C<sub>4</sub>-1000°F liquid increased by 50% from coal-only yield (25.0 to 37.4 lbs/100 dry coal). The net bottoms conversions at this increased solvent-to-solids ratio were 25.3 and 29.9 lbs/100 lbs 1000°F+ bottoms fed at 60-minute and 25-minute residence times, respectively. As can be seen in Table 1-11, essentially all this increase in conversion over the 1.07 solvent-to-solids data resulted in additional C<sub>4</sub>-1000°F liquids at both residence times.

At the same 1/0.5 coal-to-bottoms ratio and 840°F/40 minutes/1500 psig conditions, Monterey bottoms recycle study results showed an 18% increase in C<sub>4</sub>-1000°F liquids over coal-only yield (31.9 to 37.5 lbs/100 lbs dry coal). On a net bottoms basis, this represents a 21.1 lb/100 lbs 1000°F+ bottoms fed conversion of which 11.5 lbs/100 lbs was C<sub>4</sub>-1000°F liquid.

Thus, on a once-through basis, Wyodak coal shows a potential for bottoms recycle similar to Monterey coal. However, more solvent will apparently be required to achieve comparable liquid yields with Wyodak coal.

● Effect of Residence Time on Wyodak Bottoms Recycle Yields

As shown in Table 1-11 for 2.4/1/0.5 solvent-to-coal-to-bottoms, slightly more C<sub>4</sub>-1000°F liquids are produced at 60-minutes residence time (39.0 lbs/100 lbs coal fed) than at 25-minutes residence time (37.4 lbs/100 lbs coal fed). However, the 25-minute bottoms actually show more net conversion than the 60 minute bottoms. The net bottoms yields are shown in Table 1-12.

TABLE 1-12  
NET WYODAK BOTTOMS YIELDS

	<ul style="list-style-type: none"> <li>● 840°F</li> <li>● 1500 psig</li> <li>● 2.4/1/0.5 Solvent/Coal/Btms</li> <li>● 4 wt% H<sub>2</sub> on Coal</li> </ul>	
	<u>CLPP 60-Minute Btms</u>	<u>CLPP 25-Minute Btms</u>
<u>Nominal RCLU Residence</u> <u>Time, Minutes</u>	60	25
<u>Yields, Lbs/100 Lbs</u> <u>1000°F+ Bottoms Fed</u>		
H <sub>2</sub>	-4.1	-3.2
H <sub>2</sub> O + Chemical Gases	2.4	4.1
C <sub>1</sub> -C <sub>3</sub> Gas	5.2	4.2
C <sub>4</sub> -1000°F Liquid	21.8	24.8
1000°F+	74.7	70.1
<u>Yield Periods</u>	617-620	623,624

The 60-minute CLPP bottoms run at 60 minutes in RCLU show 4.6 lbs/100 lbs 1000°F+ bottoms less conversion than the 25-minute CLPP bottoms run at 25 minutes in RCLU. Most of this conversion difference is in higher C<sub>4</sub>-1000°F liquid make from the 25-minute bottoms. This result might be expected since coal conversion at 25 minutes residence time is less than at 60 minutes; therefore, the 25-minute bottoms probably contain more convertible residue than the 60-minute bottoms. Because these net yields are obtained by calculation using coal-only and coal-plus-bottoms yields the accuracy of the remaining yields is questionable, although they appear comparable for both bottoms.

• Liquid Product Selectivity with Bottoms Recycle

The liquid product selectivity with bottoms recycle operations for Wyodak and Monterey coals is shown in Table 1-13. Although the total liquid yields for Wyodak bottoms recycle were comparable for 60-minute and 25-minute recycle cases (39.0 versus 37.4 lbs/100 lbs dry coal, respectively), the liquid product distributions at 2.4/1/0.5 solvent-to-coal-to-bottoms were considerably different. As can be seen

TABLE 1-13

LIQUID PRODUCT SELECTIVITY WITH ONCE-THROUGH BOTTOMS RECYCLE

- 840°F
- 1500 psig
- 4 wt% H<sub>2</sub> on Coal

	Wyodak				Monterey No. 1	
	60-Minute		25-Minute		40-Minute	
	Coal	2.4/1/0.5	Coal	2.4/1/0.5	Coal	1.6/1/0.5
	Only	S/C/Btms.	Only	S/C/Btms	Only	S/C/Btms
<u>Wt% of C<sub>4</sub> -1000°F</u>						
<u>Liquids</u>						
C <sub>4</sub> -400°F	70.6	84.5	65.4	72.0	59.9	58.9
400-700°F	11.4	8.1	12.6	19.1	20.7	26.0
700-1000°F	18.0	7.4	22.0	8.9	19.4	15.1

in Table 1-13, for the 60-minute recycle case the percentage of C<sub>4</sub>-400°F naphtha increased from 70.6% with coal-only to 84.5% with bottoms recycle. Both 400-700°F and 700-1000°F fractions decreased significantly. At 25-minutes, the C<sub>4</sub>-400°F naphtha fraction with bottoms recycle also increases over the coal-only case, but not nearly as much as for 60-minutes. Rather than decrease, the 400-700°F liquid fraction

increases with bottoms recycle at 25 minutes to 19.1% from 12.6% with coal-only. The 700-1000°F liquid fraction decreases considerably compared to coal-only at 25-minutes recycle conditions, just as for 60-minutes recycle.

In comparison, Monterey coal showed no increase in C<sub>4</sub>-400°F naphtha at these conditions. The 400-700°F liquid fraction increased with bottoms recycle, while the 700-1000°F liquid fraction decreased. Overall, just as with Wyodak bottoms recycle, the liquid product slate also becomes lighter with recycle of Monterey bottoms.

These results indicate that, at least on a once-through bottoms basis, considerable flexibility exists in liquid product composition with Wyodak bottoms recycle operations. This flexibility is significantly greater than that observed with Monterey bottoms recycle for the conditions studied.

• Coal/Bottoms Synergism with Wyodak Recycle Operations

No synergistic effects were apparent when coal and bottoms were mixed during the Monterey bottoms recycle study. The net yields on bottoms in Table 1-14 are comparable for the different bottoms-to-coal ratios investigated and in reasonable agreement with the yields from a bottoms-only run. Net C<sub>4</sub>-1000°F liquid yields were actually somewhat less than for bottoms-only.

TABLE 1-14

COMPARISON OF NET BOTTOMS YIELDS WITH BOTTOMS-ONLY YIELD  
FOR MONTEREY RECYCLE OPERATIONS

- 840°F
- 1500 psig
- 40 minutes
- 4 wt % H<sub>2</sub> on Coal

<u>Nominal Bottoms/Coal Ratio</u>	1:3	1:2	1:1	Bottoms Only
<u>Yields, Lbs/100 Lbs 1000°F<sup>+</sup> Bottoms</u>				
C <sub>1</sub> -C <sub>3</sub> Gas	5.0	5.0	4.7	4.4
C <sub>4</sub> -1000°F Liquid	10.4	11.6	11.8	14.6
1000°F <sup>+</sup> Bottoms	84.2	79.1	82.1	79.2
<u>Yield Periods</u>	550-554	555-556	557-558 561-563	46

TABLE 1-15

COMPARISON OF NET BOTTOMS YIELDS WITH BOTTOMS-ONLY  
YIELD FOR WYODAK RECYCLE OPERATIONS

- 840°F
- 1500 psig
- 60 minutes
- 4 wt% H<sub>4</sub> on Coal
- 1.6/1 Solvent/Solids

	<u>Bottoms + Coal</u>	<u>Bottoms Only</u>
<u>Yields, Lbs/100 Lbs 1000°F+ Bottoms</u>		
C <sub>1</sub> -C <sub>3</sub> Gas	5.2	5.8
C <sub>4</sub> -1000°F Liquid	21.8	13.7
1000°F+ Bottoms	74.6	79.5

Calculation of the net yields from Wyodak bottoms recycle at 60-minutes residence time is shown in Table 1-15. A significant increase in C<sub>4</sub>-1000°F liquid yield over bottoms-only at the same liquefaction conditions was determined. Liquid yield increased from 13.7 lbs/100 lbs 1000°F+ bottoms to a calculated 21.8 lbs/100 lbs 1000°F+ bottoms when coal was added. This 8.1 lbs/100 lbs delta is indicative of a similar synergistic effect observed in bench scale tubing bomb studies with Wyodak bottoms. (See Section 4 of this report.)

Further work will be required with Wyodak coal to determine the effect of continuous bottoms recycle on the solvent-to-solids and synergistic effects observed with once-through operations.

● Unit Operations

A total of 610 hours on coal and bottoms was logged on RCLU-1 during the Monterey simulated bottoms recycle study. Operability with bottoms recycle at the conditions studied was similar to that with Monterey coal alone. No significant problems were experienced. The longest of four bottoms recycle runs was ended after 286 hours due to a plug in the feed line between the slurry feed pump and the liquefaction reactor.

Wyodak bottoms were run with coal for 563 hours in RCLU-1. The longest run was 149 hours. Five bottoms recycle runs were made, of which three were ended voluntarily. The remaining two runs were ended following plugs in the liquefaction reactor. Overall, operability with bottoms recycle was improved somewhat over that with Wyodak coal alone when additional solvent was added for the 2.4/1/0.5 solvent-to-coal-to-bottoms runs. In contrast, operability during the 60-minute bottoms-only run was not as good. Higher than usual pressure and occasional plugging in

the slurry circulation lines from the feed tank to the feed pumps were experienced. The plugging may have been due to the slow solvating action of the recycle solvent on the bottoms after circulating for several hours at ambient temperature. Softened, partially dissolved bottoms would tend to be "stickier" than coal slurry and more likely to plug the slurry circulation lines. No problems were experienced in the liquefaction reactor during the run.

The bottoms viscosities for Monterey bottoms recycle at the conditions studied are presented in Table 1-16. As can be seen, the bottoms viscosities are similar for the different bottoms-to-coal ratios run. The viscosity for bottoms from coal appears to be somewhat less than the recycle bottoms, but the corresponding 1000°F<sup>-</sup> content in the bottoms is higher. The CLPP bottoms used as recycle feed had considerably higher viscosity than the product bottoms. These high viscosities are due to the low 1000°F<sup>-</sup> content in the CLPP bottoms. Because of the varying 1000°F<sup>-</sup> content in the bottoms, no direct effects of recycle on viscosity can be determined. If these viscosities are adjusted to 10 wt% 1000°F<sup>-</sup> content in the bottoms (July-September, 1978, EDS Quarterly Technical Progress Report [FE-2893-21], Figure 6-2), all the bottoms in Table 1-16 are within the 5 to 10 poise viscosity range. Regardless, the product bottoms viscosities were well below the 50 to 100 poise which cause difficulty in emptying the RCLU bottoms accumulator.

TABLE 1-16

VISCOSITY OF LIQUEFACTION BOTTOMS FOR MONTEREY BOTTOMS RECYCLE

	Coal Only Bottoms	Nominal Bottoms/Coal Ratio			CLPP(Feed) Bottoms
		<u>1:3</u>	<u>1:2</u>	<u>1:1</u>	
<u>Wt % 1000°F<sup>-</sup> in</u>					
<u>Bottoms</u>	11	6	6	6	1.2-2.6
<u>Viscosity, poise</u>					
(550°F. 10 sec <sup>-1</sup> shear rate)	6	11	13	17	40-130

The viscosities of residual bottoms from Wyodak coal-only operations are dependent on the degree of coal conversion (July 1977-June 1978, EDS Annual Technical Progress Report [FE-2893-17], p. 12). Thus, the increased conversion of Wyodak coal afforded by recycle of bottoms might be expected to reduce the product bottoms viscosity over coal-only operations.

Evidence that reduction in Wyodak bottoms viscosity should occur for continuous bottoms recycle is shown in Figure 1-22. Compared to the bottoms feed viscosities, some reduction was noted in viscosity for bottoms-only, 1.6/1.0/0.5 solvent-to-coal-to-bottoms, and 2.4/1.0/0.5 solvent-to-coal-to-bottoms operations. The reduction in bottoms viscosity was notably less at the 1.6 solvent-to-coal condition where little additional bottoms conversion was obtained. When bottoms conversion was significant, such as with the bottoms-only and 2.4 solvent-to-coal operations, considerable reduction in feed bottoms viscosity resulted.

Pyridine and benzene extractions were also performed on the feed and product bottoms. Figure 1-23 shows the organic (i.e. ash-free) fractions which are benzene soluble (asphaltenes) and pyridine soluble/benzene insoluble (preasphaltenes). Total bar height represents pyridine solubles. The 1000°F<sup>-</sup> fraction in the bottoms was assumed to be benzene soluble in these calculations. These solubility results are consistent with the bottoms recycle 1000°F<sup>-</sup> conversion. The increase in benzene solubles in the bottoms product compared to the bottoms feed can be interpreted as conversion of 1000°F<sup>+</sup> organics from the preasphaltene fraction to asphaltenes.

For Monterey coal pyridine extracts essentially all but the fusinite fraction from the bottoms. Additional pyridine insoluble organics contribute to a much lower (60-70 wt%) pyridine soluble fraction in the Wyodak feed and product bottoms. When significant conversion to 1000°F<sup>-</sup> products occurred, the asphaltene fraction in the product bottoms also increased over that in the feed bottoms. This indicates conversion within the 1000°F<sup>+</sup> fraction also taking place. Thus, with Monterey bottoms recycle and with Wyodak bottoms recycle at 2.4 solvent-to-coal ratio, the asphaltenes increased somewhat in the product bottoms over the level in the feed bottoms. However, for the Wyodak bottoms recycle with 1.6 solvent-to-coal ratio where little net bottoms conversion was observed, the product bottoms actually decreased in asphaltene content compared to the feed bottoms. In this instance, the product bottoms can be considered less converted than the feed bottoms since the preasphaltene fraction has increased

### 1.5 Future Plans

During the second quarter of 1979, a detailed process variable study was initiated in RCLU-1 with the first of the sponsor coals, Pittsburgh seam coal from the Ireland mine. Primary variables investigated were liquefaction temperature, residence time, and pressure. The results of this portion of the study will be presented next quarter. This study is scheduled to last up to six months.

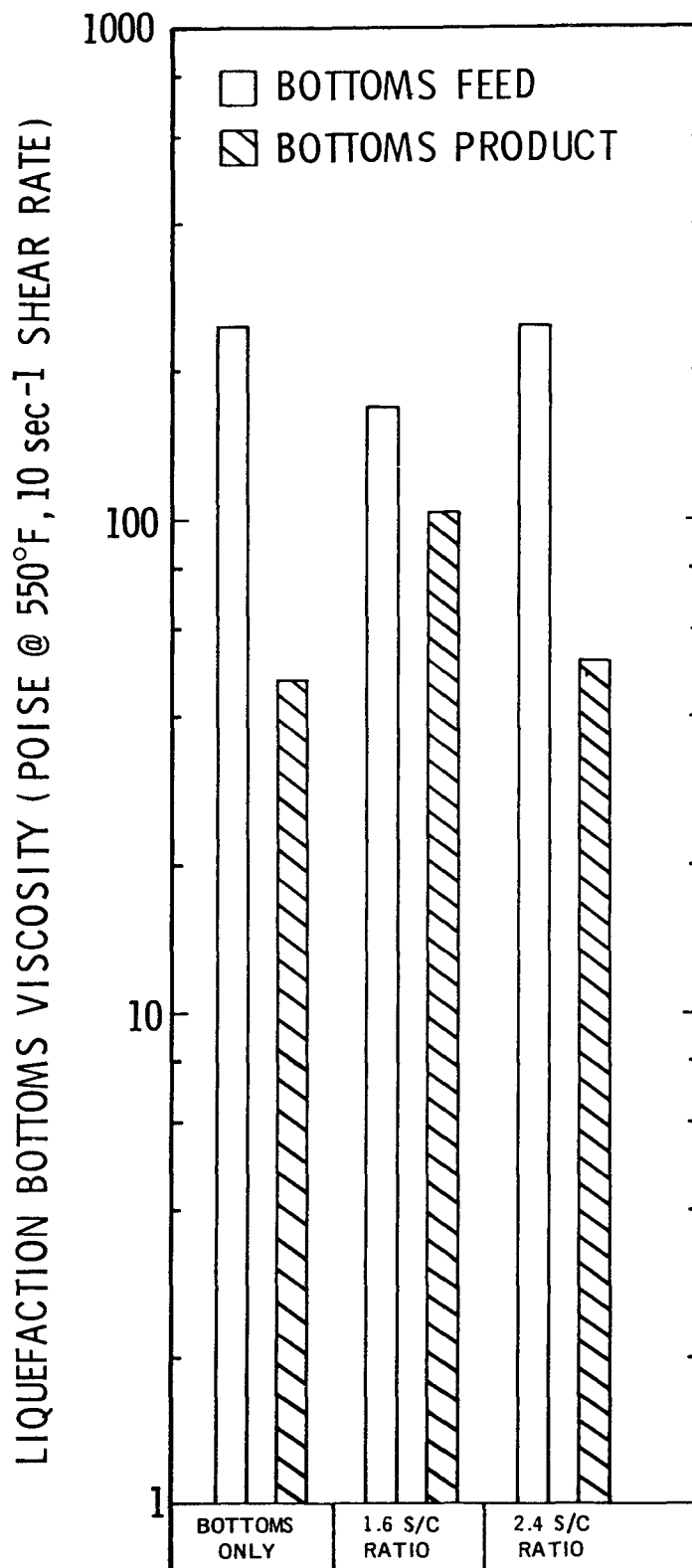
RCLU-2 will remain shut down concurrent with ongoing CLPP operations.

Initial bench liquefaction studies (see Laboratory Process Research and Development, Section 4.4) aimed at determining the applicability of bottoms recycle to different coals is complete. These results and an assessment of the possible use of RCLU for equilibrium operations with bottoms recycle (rather than the simulated operations described herein) is underway and will be discussed in the next Quarterly Technical Progress Report.



FIGURE 1-22

COMPARISON OF BOTTOMS FEED AND BOTTOMS PRODUCT  
VISCOSITIES WITH WYODAK BOTTOMS RECYCLE

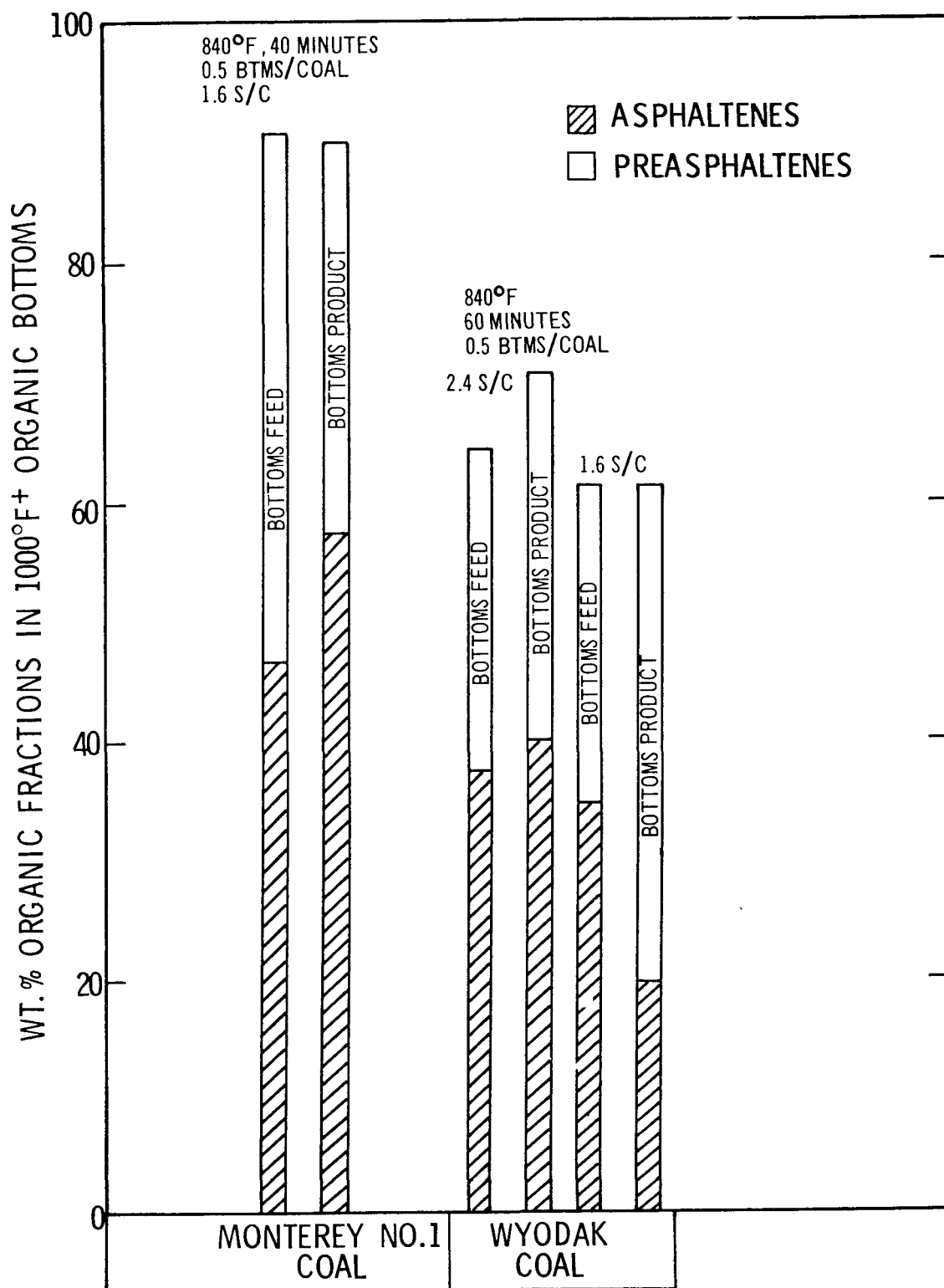


CONDITIONS:

840°F  
1500 psig/  
60 Minutes  
8-12% 1000°F -

FIGURE 1-23

COMPARISON OF BOTTOMS FEED AND BOTTOMS PRODUCT  
ORGANIC FRACTIONS FOR WYODAK AND MONTEREY NO. 1  
BOTTOMS RECYCLE



## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### LIQUEFACTION PROCESS RESEARCH

#### 2. Operation of the One-Ton-Per Day Coal Liquefaction Pilot Plant (CLPP)

The one ton-per-day Coal Liquefaction Pilot Plant (CLPP) was operated during the last half of 1978 on Wyoming subbituminous coal from the Wyodak mine. From December, 1978 to February, 1979, CLPP underwent a scheduled turnaround to do necessary maintenance and especially to install vacuum bottoms recycle equipment. Since that time CLPP has operated with Illinois No. 6 coal from the Monterey No. 1 mine in both coal-only and continuous bottoms recycle modes. A detailed description of CLPP and a process flow diagram were included in the July 1, 1976-June 30, 1977 EDS Annual Technical Progress Report (FE-2893-17). In addition, a flow diagram of the bottoms recycle portion of the CLPP was included in the January-March, 1979, EDS Quarterly Technical Progress Report (FE-2893-29).

In the last six months of 1978, CLPP operated with Wyodak coal. Thirteen elementally balanced yield periods were obtained. Full details on conditions and tabulated yield data may be found in the October 1-December 31 and January 1-March 31 EDS Quarterly Technical Progress Reports (FE-2893-25 and FE-2893-29). Studies were also conducted to characterize the liquefaction reactor solids withdrawal products and to determine the level of entrainment and fouling in the primary vacuum tower. Solids formation in the high pressure gas scrubbing system was also studied.

#### 2.1 Basis for Wyoming Coal Study Design

Data from CLPP has been supplied to the EDS Liquefaction Engineering Division (ELED) for use in setting the basis for the Wyoming Coal Study Design. The data supplied to ELED were obtained by averaging the data from Yield Periods 267, 268, 269, and 270. The overall yields and conditions are shown in Table 2-1. The longer residence time and higher solvent/coal ratio, as compared to Illinois No. 6, are necessary due to the higher viscosity of Wyoming coal liquefaction bottoms at lower conversion levels.

TABLE 2-1

YIELD STRUCTURES FOR WYOMING COAL STUDY DESIGN

Liquefaction Temp., °F	840
Nominal Residence Time, Min.	60
Liquefaction Pressure, psig	1500
Solvent-to-Coal Ratio	1.6/1
Yield Periods	267,268,269,270
<u>Overall Yields</u>	
Hydrogen	-3.68
Water	13.87
Carbon Oxides	6.39
Ammonia	0.40
Hydrogen Sulfide	0.21
C <sub>1</sub> -C <sub>3</sub> Gas	7.38
C <sub>4</sub> -400°F Naphtha	22.18
400°-700°F Middle Distillate	1.67
700-1000°F Heavy Distillate	5.05
1000°F+ Bottoms	46.53

2.2 Engineering Studies on Wyodak Coal

In the last half of 1978, engineering studies continued on Wyodak coal in the three major areas of liquefaction reactor solids withdrawal, vacuum tower fouling and entrainment, and solids formation in the high-pressure gas scrubbing system.

Since its installation, the liquefaction reactor solids withdrawal system has proven to be quite effective in eliminating reactor plugging. About 4 wt % withdrawal on coal feed controlled reactor solids buildup during normal operation. Analysis of the withdrawal products indicates that solids from the first reactor are enriched in calcium. The wall scale from the first reactor was also examined and was determined to be about 67% calcium carbonate. The scale accumulated at a rate of about 0.006 lb scale per 100 lb of coal feed. For more information, consult the October 1-December 31, 1978, EDS Quarterly Technical Progress Report (FE-2893-25).

Examination of the primary vacuum tower (F-1) has shown that very little solids entrainment and fouling occurs for Wyodak coal during normal operation. Only scattered deposits have been found on the entrainment grid suspended in the tower, and the vessel walls have shown no buildup of solids or scale.

During integrated operations with Wyodak coal, plugs occasionally have formed in the high-pressure gas scrubbing system. Analyses indicate that the plug materials were mixtures of ammonium carbonate,  $(\text{NH}_4)_2\text{CO}_3$ , and ammonium bicarbonate,  $\text{NH}_4\text{HCO}_3$ . However, plugs of these materials were minimized on the CLPP by heating or water washing the affected lines.

### 2.3 Installation and Operation of New Equipment

During the turnaround starting in December, equipment to recycle hot, molten vacuum bottoms to liquefaction was installed. This equipment has been operated successfully with bottoms from Illinois No. 6 (Monterey) coal.

A new coal feed system was also designed and installed on CLPP during the turnaround. Performance of the new system has been far superior to the old feed system. Fluctuations in coal feed rate have been reduced to within plus or minus 2% of the set rate. Additional details are included in EDS Quarterly Technical Progress Report FE-2893-29.

### 2.4 Operations With Bottoms Recycle

Initial operations of bottoms recycle equipment showed very good service and only two areas have shown erosion attributed to bottoms recycle. The first was the vacuum bottoms and coal slurry mix point, and the other was at the preheater junction near the liquefaction reactor inlet. However, both of these areas were redesigned and have not shown further erosion. These are more fully described in the April, 1979, EDS Monthly Technical Progress Report [FE-2893-30].

Since starting operations, over 550 hours of operation have been completed with bottoms recycle fully integrated on the CLPP. In addition, over 1200 hours of time have been logged with coal fed to the unit as part of the bottoms recycle study. Bottoms properties have varied to viscosities of over 50 poise with 2-3% 1000°F material in the bottoms. And only minor difficulties were encountered in pumping and recycling the higher viscosity bottoms.

Preliminary yield data with bottoms recycle indicated lower conversions than expected when compared to RCLU data. However, at least part of the low conversion was attributed to poor coal-only yields on the CLPP. Bench equipment RCLU and CLPP were used to study all liquefaction components as well as bottoms recycle until the CLPP yield structure was resolved. After a complete turnaround on the CLPP to recalibrate and reconfigure the unit, coal-only yields returned to normal and bottoms recycle studies were resumed. (For more complete details, see June, 1979, EDS Monthly Technical Progress Report [FE-2893-34]).

Bottoms recycle yields after the turnaround showed improvements over coal-only operation. Further data workup as well as additional bottoms recycle operations are planned into the next quarter before starting on Pittsburgh coal. During operations on Pittsburgh coal, the bottoms recycle information will be evaluated, and further work on bottoms recycle will be planned.

## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### LIQUEFACTION PROCESS RESEARCH

#### 3. Solvent Hydrogenation Studies

During the third quarter of 1978, solvent hydrogenation studies were made on the effects of treat gas impurities and feed solvent on catalyst activity. Illinois spent solvent and Ni/Mo-10 catalyst were used to broaden the data base on the solvent hydrotreating effects of carbon oxide impurities in the hydrogen treat gas. In addition, a Wyoming coal spent solvent was hydrotreated at various temperatures and space velocities over Ni/Mo-10 and Co/Mo-20 catalysts. This study was undertaken to define fresh catalyst performance and to select SOR conditions for an activity maintenance study with the preferred catalyst.

During the fourth quarter of 1978, solvent hydrogenation studies continued to concentrate on the effects of Wyoming spent solvent on catalyst activity. The initial catalyst activity study described above with Wyoming spent solvent over Ni/Mo-10 and Co/Mo-20 catalysts was completed. An additional scoping study was performed with the same solvent and catalysts. In this study, the solvent was spiked with heteroatoms (sulfur, nitrogen, and oxygen) separately to determine the effects of each on catalyst activity independently of the other two heteroatoms.

During the first quarter of 1979, hydrogenation of a Wyoming spent solvent/naphtha mixture was compared to the hydrogenation of a Wyoming spent solvent only. The primary purpose of this study was to determine any effects the added naphtha had on the product, solvent quality and catalyst activity. Also, brief scoping runs were carried out to study the hydrogenation of Illinois 400°F<sup>+</sup> and Wyoming 350°F<sup>+</sup> products.

During the second quarter of 1979, no work was scheduled in solvent hydrogenation.

A summary of the solvent hydrogenation studies conducted during this reporting period is shown in Table 3-1. Results are discussed in Sections 3.2 through 3-4 of this report. Data obtained from the studies completed during the first nine months are reported in detail in previous quarterly technical progress reports.

#### 3.1 Experimental Facilities and Procedures

Laboratory studies of solvent hydrogenation are conducted in small once-through fixed bed reactor systems. A simplified flow diagram of the bench scale hydrotreaters used in this program is shown in Figure 3-1. These units, designated M20, M21 and M22 are equipped with

TABLE 3-1

## SUMMARY OF SOLVENT HYDROGENATION STUDIES

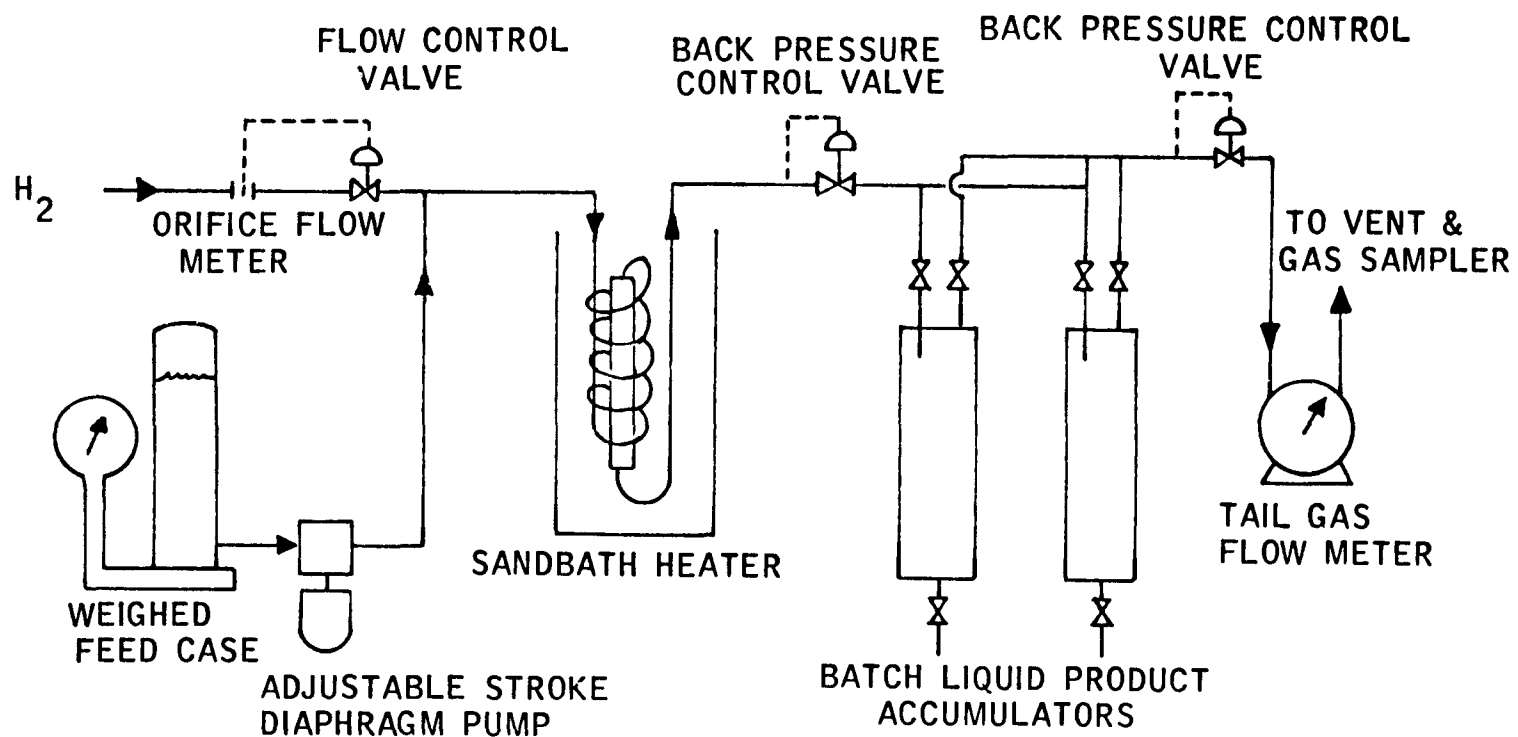
Run	Catalyst	Feed	Reactor Size, ML.	Temperature-RV °F	Pressure psig	Duration Days	Purpose of Run
13M22 A	Ni/Mo-10	Ill. MPSS	10	125-190	1500	38	Carbon Oxide Impurity Study
B	Ni/Mo-10	Ill. MPSS	10	125-190	1500	38	Carbon Oxide Impurity Study
14M22 A	Ni/Mo-10	Wyo. Spent Solvent	10	65-165	1500	73	Catalyst Activity Screening
B	Co/Mo-20	Wyo Spent Solvent	10	65-165	1500	73	Catalyst Activity Screening
15M22 A	Ni/Mo-10	Wyo. Spent Solvent	10	65	1500	45	Catalyst Activity Screening
B	Co/Mo-20	Wyo. Spent Solvent	10	65	1500	45	Catalyst Activity Screening
15M21*	Ni/Mo-10	Ill. 400°F <sup>+</sup>	20	125-145	2250	9	Total Product Hydrotreating Study
16M21*	Ni/Mo-10	Wyo. 350°F <sup>+</sup>	50	165	2250	4	Total Product Hydrotreating Study
16M22 A	Ni/Mo-10	Wyo. Spent Solvent	10	65-165	1500	39	Effects of Spent Solvent/ Naphtha Mixture Hydrotreating
B	Ni/Mo-10	Wyo. Spent Solvent/ Naphtha Mixture	10	65-165	1500	39	
17M22	Ni/Mo-10	Wyo. 350°F <sup>+</sup> Naphtha	10	215-265	1000	12	Naphtha Dehydrogenation Study

\*Fuel oil hydrotreating studies at EPRL are discussed in Laboratory Process Research and Development, Section 5.4 of this report



FIGURE 3-1

SIMPLIFIED FLOW DIAGRAM OF BENCH SCALE HYDROTREATERS



sufficient automatic monitoring and shutdown devices to allow safe and reliable overnight unattended operations. In general, these units are capable of running continuously for several months on a single catalyst charge.

One of the three bench units, M22, is operated as a dual reactor catalyst testing unit. This unit basically is similar to M20 and M21 but uses a single sandbath for isothermal temperature control of two separate reactor systems and incorporates dual feed pump and product accumulator capabilities. Smaller reactors (10 ml. catalyst volume compared to 70 ml. catalyst volume used in M20, M21) are used in this dual-train unit.

Experimental solvent hydrogenation studies begin with catalyst selection and preparation. Then the units are operated with the desired feedstock to carry out the details of the planned experiments. Solvent feed and products are characterized by mass spectrometry. Mass spectrometry provides a measure of the component distribution (both aromatics and saturates) and how this distribution is affected by the hydrogenation process. Of particular importance is the donor hydrogen characteristics of the feeds and products. These characteristics are directly related to the solvent quality index (SQI) used in determining the most effective recycle solvent.

### 3.2 Carbon Oxide Impurity Study

An experimental study was carried out to broaden the data base on the solvent hydrotreating effect of carbon oxide impurities in the hydrogen treat gas. Previous observations (see July 1977 to June 1978, EDS Annual Technical Progress Report [FE-2893-17]) showed that carbon oxides do not adversely affect catalyst activity in solvent hydrogenation.

- Experimental Conditions

Illinois coal multi-pass spent solvent (MPSS) and Ni/Mo-10 catalyst were used at additional start-of-run (SOR) conditions. Temperatures ranging from RV+125°F to RV+190°F and various space velocities were tested in the two-train catalyst testing unit, M-22, with one reactor utilizing hydrogen-only treat gas and the other 2% CO<sub>2</sub>/2% CO in hydrogen. Liquid products and tail gas samples were collected and analyzed.

- Study Results

Results from this study confirmed the absence of any effect on donor hydrogenation activity due to the presence of carbon oxides. Some loss in desulfurization and denitrogenation activity was observed, but these deactivations were apparently reversible. Approximately 20% desulfurization and denitrogenation activity losses were observed at all test conditions based on heteroatom contents of the liquid products. Examination of the tail gas samples also confirmed conversion of carbon

dioxide to carbon monoxide and methane due to water gas shift and methanation reactions. A summary of the results from this study is shown in Table 3-2.

### 3.3 Effect of Wyoming Solvent on Catalyst Activity

Two catalyst initial activity scoping studies with Wyoming coal spent solvent were completed during the third quarter of 1978. These studies were carried out, with both Ni/Mo-10 and Co Mo-20 catalysts, to obtain data on catalyst activity performance and provide a basis for selecting SOR conditions for an activity maintenance study with the preferred catalyst.

The effects of feed heteroatom (sulfur, nitrogen and oxygen compounds) content on catalyst activity were investigated by spiking a Wyoming spent solvent to heteroatom concentrations similar to those in Illinois spent solvent. Various space velocities and temperatures of RV+65°F and RV+115°F were tested in the first study (Run 14M22).

Results from Run 14M22 agreed with earlier findings which indicated that Wyoming spent solvent behaves differently than Illinois solvent. Higher solvent donor hydrogen levels were produced from Wyoming spent solvent than Illinois solvent with Ni/Mo-10 catalyst at low temperatures, but no significant difference with Co/Mo-20 catalyst at similar temperatures. The observed difference in product donor hydrogen level with Ni/Mo-10 catalyst was thought to be due to the differences in feed heteroatom content (See July-September, 1978, EDS Quarterly Technical Progress Report [FE-2893-21]). Results also indicated that sulfur and nitrogen compounds in the feed did not significantly affect catalyst activity at the experimental temperatures, but that oxygen compounds did. However, these results were considered misleading since the catalyst had already been deactivated at higher temperatures prior to this investigation. The effect of the heteroatoms on catalyst activity, if any, was expected to be more pronounced on fresh catalyst.

As a result, a second study (Run 15M22) was carried out to confirm the effects of Wyoming solvent heteroatom content on catalyst activity. Fresh Ni/Mo-10 and Co/Mo-20 catalysts were used to hydrogenate a spent solvent spiked with each heteroatom separately to the levels of the individual concentrations of Illinois solvent. In addition, space velocities were varied to broaden the data base. Thus, the effect of each heteroatom on catalyst activity was examined independently of the other two heteroatoms.

The 15M22 study was conducted only at a temperature of RV+65°F and space velocities ranging from 1.21xRV to 4.0xRV HR<sup>-1</sup>. Wyoming spent solvent was hydrogenated without any heteroatom spike, with sulfur spiked to 0.3 wt %, with nitrogen spiked to 0.3 wt %, with oxygen spiked to 1.6 wt % and with all heteroatoms spiked to the above levels.

Results of both studies are compared in Figures 3-2 and 3-3. Solvent donor hydrogen content of hydrogenated product is shown for

TABLE 3-2

RESULTS OF CARBON OXIDE IMPURITY STUDY

Run: 13M22 A&B  
 Catalyst: Ni/Mo-10  
 Feedstock: Illinois Spent Solvent  
 Pressure: 1500 psig  
 Treat Gas Impurity: 2.3% CO<sub>2</sub>/2.1% CO

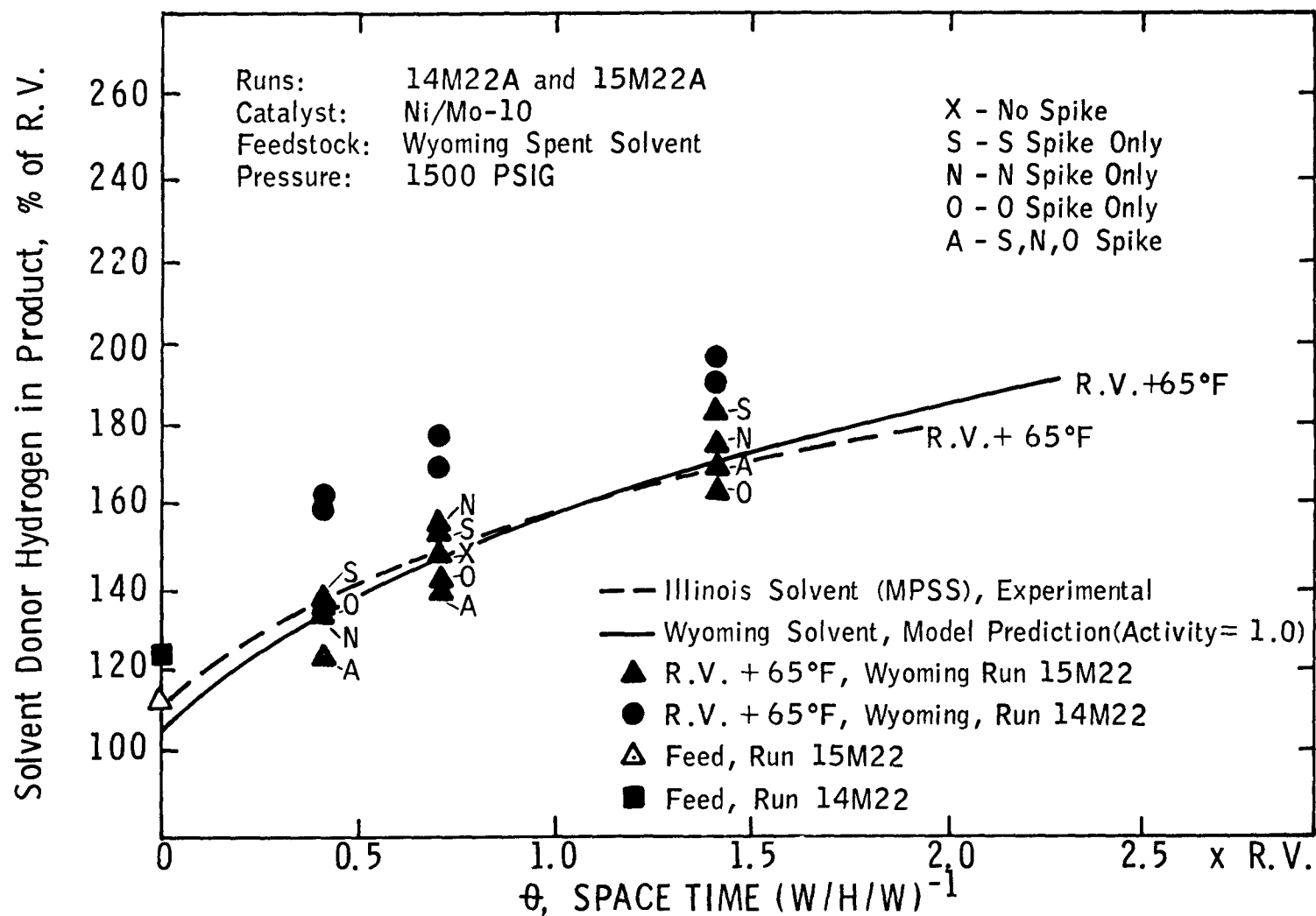
Temp.-RV °F	Space Velocity (W/H/W) % of R.V.	Treat Gas	Relative Activity	Gas Product, Mole %		
				CO <sub>2</sub>	CO	CH <sub>4</sub>
125	417	H <sub>2</sub>	100	0.01	.03	0.07
125	412	H <sub>2</sub> (CO <sub>2</sub> /CO)	98	1.04	2.37	0.61
125	125	H <sub>2</sub>	100	0.01	Trace	0.06
125	122	H <sub>2</sub> (CO <sub>2</sub> /CO)	101	0.61	1.73	1.05
165	418	H <sub>2</sub>	100	0.0	Trace	0.08
165	413	H <sub>2</sub> (CO <sub>2</sub> /CO)	100	1.86	2.24	1.31
165	125	H <sub>2</sub>	100	0.08	Trace	0.12
165	125	H <sub>2</sub> (CO <sub>2</sub> /CO)	102	1.22	2.12	1.76
190	417	H <sub>2</sub>	100	0.01	Trace	0.17
190	413	H <sub>2</sub> (CO <sub>2</sub> /CO)	99	1.22	2.03	1.10
190	253	H <sub>2</sub>	100	0.15	Trace	0.18
190	250	H <sub>2</sub> (CO <sub>2</sub> /CO)	99	1.66	2.83	1.71
190	126	H <sub>2</sub>	100	0.01	Trace	0.21
190	130	H <sub>2</sub> (CO <sub>2</sub> /CO)	103	1.27	2.17	1.37

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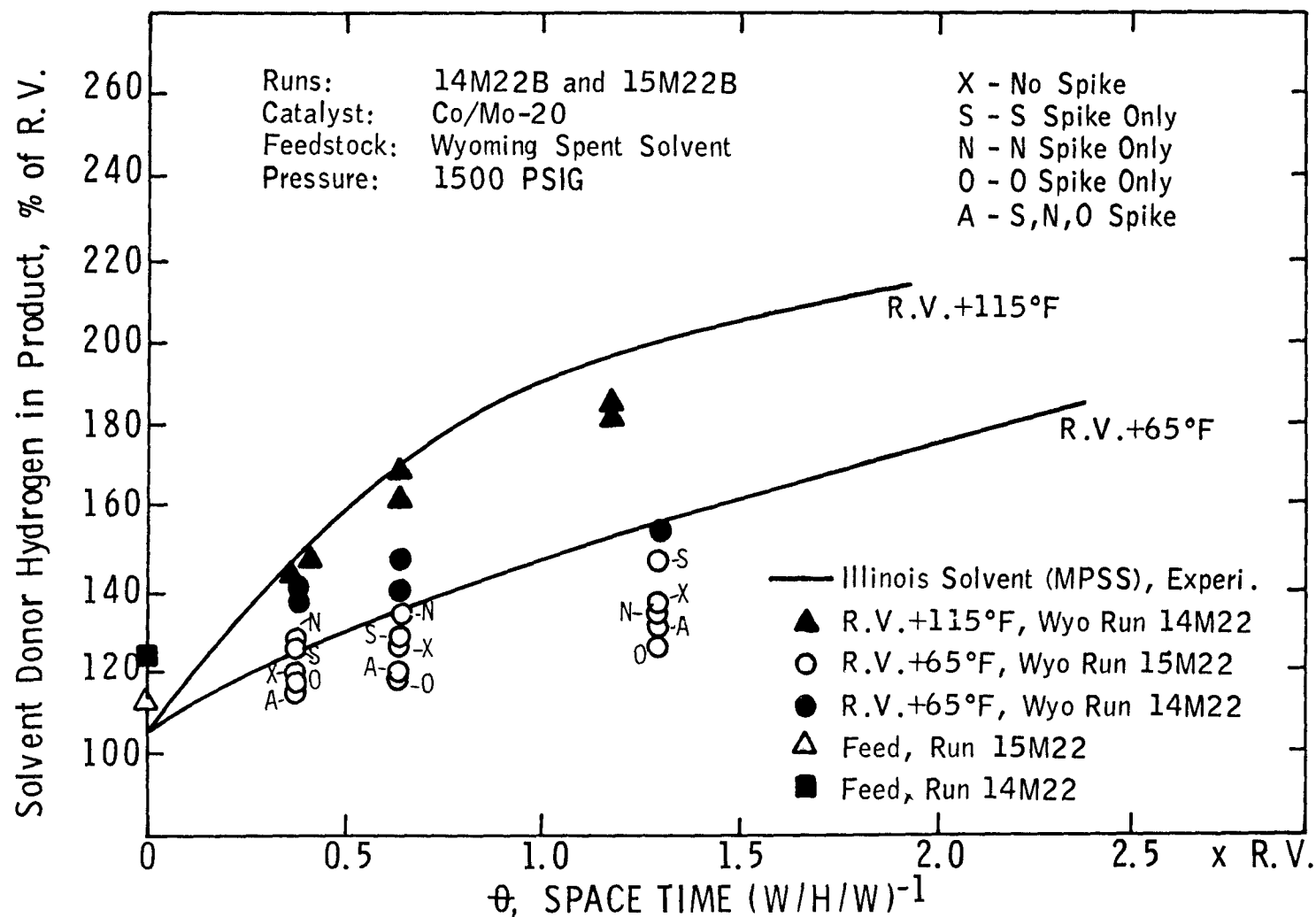

$$*R.A. = \frac{\text{Product Quality Obtained from Contaminated Hydrogen Stream}}{\text{Product Quality Obtained from Pure Hydrogen Stream}}$$

FIGURE 3-2

EFFECTS OF TEMPERATURE AND SPACE TIME ON WYOMING SPENT SOLVENT  
FOR Ni/Mo-10 CATALYST



**FIGURE 3-3**  
**EFFECTS OF TEMPERATURE AND SPACE TIME ON WYOMING SPENT SOLVENT**  
**FOR Co/Mo-20 CATALYST**



different space times at a temperature of  $RV+65^{\circ}F$  for Ni/Mo-10 and Co/Mo-20 catalysts, respectively. The solid and dashed lines represent previous Illinois solvent experimental data and Wyoming solvent model predictions for a catalyst activity of 1.0, respectively. The circular symbols are from the 14M22 study, and the triangular symbols are from the 15M22 study.

Within experimental variability, heteroatom spikes to the Illinois solvent levels did not affect the hydrogenation activity of the catalysts with Wyoming spent solvent at the above conditions. The data used in the analysis of the 15M22 study were taken after an initial catalyst stabilization period of nominally two weeks. These hydrogenation catalysts exhibit high initial activity prior to lineout after about 1 to 3 weeks on stream. The experimental Wyoming spent solvent results obtained in this study are predicted by the solvent hydrogenation model for an initial catalyst activity. Results obtained at the lower temperatures in the previous 14M22 study were collected during the first 2 to 3 weeks of catalyst life. Thus, what appeared to be increased catalyst activity due to lower heteroatom content was increased activity due to the high initial catalyst activity (prior to line-out).

### 3.4 Hydrogenation of Wyoming Solvent/Naphtha Mixture

Depending on environmental regulations, hydrotreatment of the EDS  $350^{\circ}F^{+}$  fuel oil product may be required. A relatively low cost, on-site hydrotreatment step blends heavy naphtha with the total spent EDS solvent from liquefaction, withdrawing the upgraded naphtha and excess solvent from the solvent hydrotreater product stream. In this configuration, heavy naphtha and solvent products are upgraded with incremental catalyst added to an existing solvent hydrogenation reactor. Additional upgrading of the  $350^{\circ}F^{+}$  fuel oil, if required, could be achieved in a downstream facility processing VGO and higher boiling fuel oil components.

To assess this hydrotreating option, a study was conducted in which a mixture of spent solvent and  $350^{\circ}F^{+}$  naphtha from Wyoming coal was hydrotreated. The primary purpose of this study was to determine the effects, if any, of the added naphtha on the product, solvent quality and catalyst activity. The effect on naphtha quality due to hydrotreating the mixture was also determined.

#### ● Experimental Conditions

The two-train solvent hydrogenation bench unit was used to hydrotreat a mixture of 92.5 wt % Wyoming coal spent solvent and 7.5 wt %  $350^{\circ}F^{+}$  naphtha. As a comparison, Wyoming coal spent solvent only was hydrotreated alongside the mixture at the same operating conditions. The catalyst used was Ni/Mo-10, and the inlet hydrogen partial pressure was a constant 1500 psig. The temperatures used were  $RV+115^{\circ}F$  and  $RV+165^{\circ}F$ , and the space velocities were nominally  $1.0 \times RV$  and  $2.0 \times RV$  W/H/W.

● Study Results

Donatable hydrogen levels are shown in Table 3-3 for selected hydrotreated solvents and for the corresponding hydrotreated mixtures based on the solvent fraction only (i.e., assuming the hydrotreated heavy naphtha portion contains negligible donatable hydrogen). For the operating conditions shown, the mixture donatable hydrogens, based on the solvent fraction only, equal or exceed the donatable hydrogens of the pure solvent. Thus, the 350°F<sup>+</sup> naphtha acted only as a diluent in the hydrogenation of the spent solvent. And, because these comparisons are over the life of the catalyst, it can also be concluded that the naphtha did not affect catalyst activity.

TABLE 3-3

COMPARISON OF DONATABLE HYDROGEN FOR HYDROTREATED  
SOLVENT ONLY AND FOR THE SOLVENT/NAPHTHA MIXTURE

<u>Operating Conditions</u>		<u>Donatable Hydrogen, % of RV</u>	
<u>T-RV, °F</u>	<u>W/H/W, % of RV</u>	Mixture (Based on 92.5 Wt % Solvent Only      Solvent Fraction)	
		<u>Solvent Only</u>	<u>Solvent Fraction</u>
67	98	155	178
116	100	159	164
117	98	181	182
116	198	198	207
116	211	200	205
166	198	173	182
166	182	180	185
165	200	182	191
116	198	202	202

<sup>1</sup>Values for solvent/naphtha mixture assume no donor hydrogen contribution for naphtha.

Distillations were obtained on the solvent feed, the solvent product, the naphtha and the blend. Results of these ASTM 15/5 distillations are shown in Table 3-4 for different boiling range cuts.



TABLE 3-4

DISTILLATION COMPOSITIONS OF FEEDS AND PRODUCTS  
FROM RUNS 16M22A AND 16M22B

Distillation, Liquid Volume %				
Stream	Feed Solvent	Product Solvent	Feed Naphtha	Solvent/Naphtha Product Blend
IBP/350°F	0.0	3.8	11.4	7.4
350/400°F	11.6	19.7	75.6	21.6
400/650°F	74.3	67.6	13.0	64.4
650°F+	14.1	8.9	0.0	6.6
TOTAL	100.0	100.0	100.0	100.0

These boiling pool compositions also show the amounts of solvent converted from each higher boiling range to the next. Due to hydrotreating, approximately 37% 650°F+ material was converted to 350/400°F material. And finally, 33% of the 350/400°F boiling range cut was shifted downward to the IBP/350°F boiling range material. Hence, these results show which solvent boiling range cuts were affected, and to what extent, by hydrotreating.

In addition, as shown in Table 3-4, approximately 76% of the naphtha boils in the 350/400°F range. Assuming that each higher boiling pool, when hydrotreated, is converted only to the next lower boiling pool, approximately 50% of this 350/400°F naphtha is converted to an IBP/350°F boiling pool material. This IBP/350°F boiling pool may be more valuable as catalytic reformer feed.

In summary, hydrotreating EDS spent solvent and heavy product naphtha together is feasible since neither the solvent nor naphtha quality is compromised by hydrotreating the mixture. Moreover, there were no indications of decreased catalyst activity as a result of solvent/naphtha coprocessing.

## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### LIQUEFACTION PROCESS RESEARCH

#### 4. Bench Units, Modeling and Correlations Studies

Batch tube autoclave and continuous flow reactors are employed to provide yield and operability data for a variety of coals. The yield data from the bench scale hydrotreaters and the liquefaction pilot plants are correlated to provide models for recycle solvent hydrogenation and liquefaction which are used for process simulation.

##### 4.1 Operability Studies on Wyoming Coal

During liquefaction of low rank coals, such as Wyoming sub-bituminous coal, calcium present in salts of humic acids decomposes to form calcium carbonate ( $\text{CaCO}_3$ ). This calcium carbonate accumulates in the liquefaction reactor as wall scale and free-flowing aggregates (oolites).

Calcium carbonate formation in liquefaction can be prevented by chemically altering the form of the calcium in the coal by pretreatment. One of the proposed methods of preventing liquefaction scale formation is to pretreat the coal with sulfur dioxide ( $\text{SO}_2$ ).  $\text{SO}_2$  dissolves in the pore moisture of coal and forms stable calcium salts prior to liquefaction. Since these calcium salts are stable during liquefaction, calcium tied up in this form does not produce  $\text{CaCO}_3$  scale in the liquefaction reactor.

The various methods employed to overcome the calcium carbonate deposition problem can be classified as mechanical or chemical solutions. Mechanical solutions are those in which the process conditions, process configurations or mechanical equipment are modified to alleviate the problem. Chemical solutions are those in which the chemical form of the calcium is changed by pretreatment of coal or a chemical is added to the coal slurry before it is liquefied. A detailed description of methods and results of various solutions (both mechanical and chemical in nature) tested with Wyoming coal was reported in the July 1977-June 1978, EDS Annual Technical Progress Report (FE-2893-17).

During this reporting period, an extensive study was conducted to test one of the chemical solutions, viz. gaseous  $\text{SO}_2$  pretreatment of low rank coal. Two fluidized bed units were used for this study to achieve two objectives of the project. The first objective was to study the kinetics of pretreatment and the second was to test the effectiveness of the pretreatment as a function of various process variables.

The kinetic study was carried out in a 0.7 inch diameter Bench Scale Pretreatment Unit (BSPU). In this study, ten gram samples of -8 mesh

Wyoming coal were pretreated at 5 psig in the BSPU. The major process variables covered the following conditions:

Temperature:	65°F, 85°F
SO <sub>2</sub> Gas Concentration:	5 mole %, 10 mole %
Treat Time:	5, 15, 30 and 60 minutes

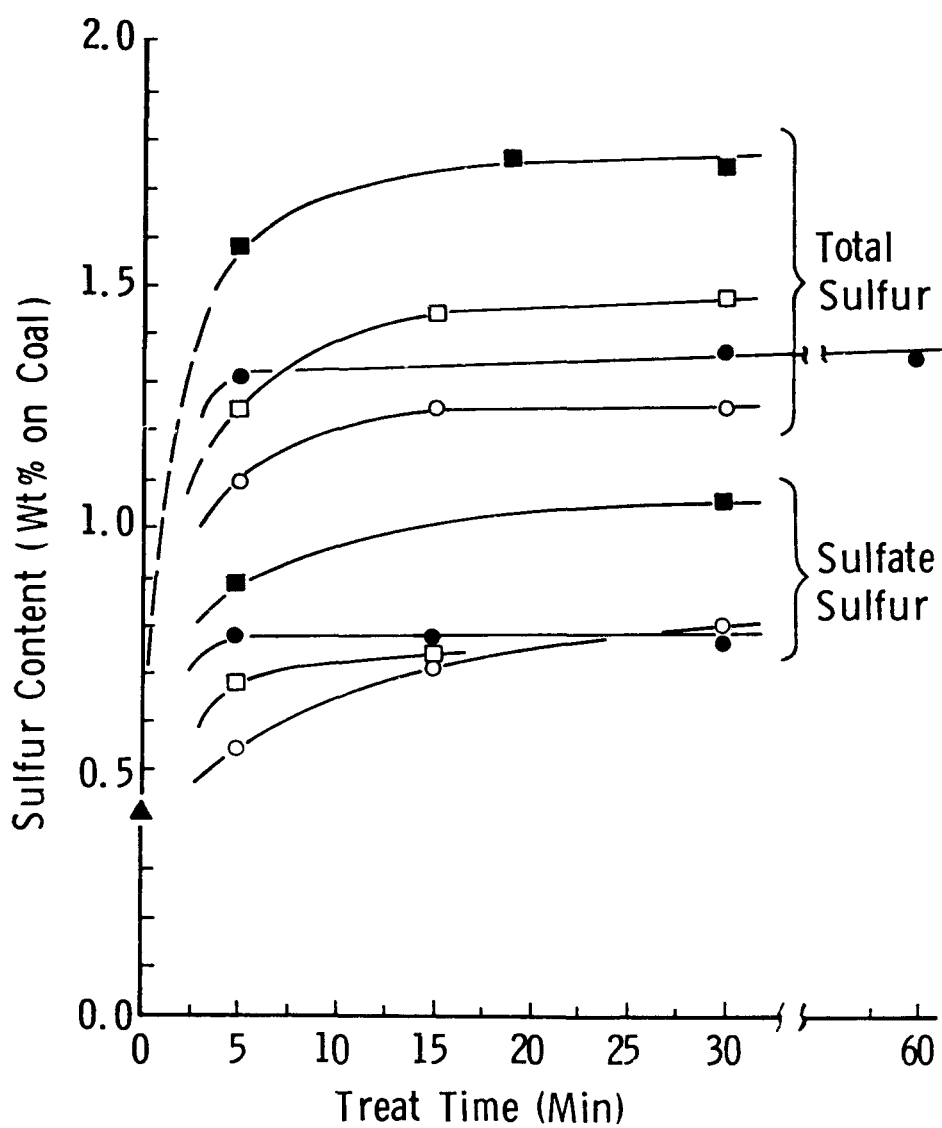
Figure 4-1 summarizes the results of the kinetic study. As pretreatment temperature increased, total sulfur and sulfate sulfur decreased. This is due to the fact that SO<sub>2</sub> is less soluble in the coal moisture at the higher temperature. With a lower HSO<sub>3</sub><sup>-</sup> (from SO<sub>2</sub> solution) concentration, the driving force for mass transfer is lower. The effect of increasing SO<sub>2</sub> concentration is similar to the effect of decreasing temperature. As treat time increased, total sulfur content increased rapidly for the first five minutes, increased more slowly to 15 minutes and then leveled off. Sulfate sulfur also increased very rapidly in the first five minutes but then continued to increase more slowly up to 30 minutes.

A 4-inch diameter plexiglass Fluidized Pretreatment Unit (FPU) was used to test the effectiveness of gaseous SO<sub>2</sub> pretreatment of high calcium coals. The pretreatment experiments were carried out with Wyoming subbituminous and Big Brown Texas lignite. The description of the unit was included in the July 1977-June 1978, EDS Annual Technical Progress Report (FE-2893-17). The effectiveness of the pretreatment was determined by carrying out tubing bomb liquefaction experiments on pretreated samples. The effectiveness is expressed as weight percent CaCO<sub>3</sub> present in the liquefaction residue ash. The results of these experiments show that this factor is significantly lower (8-10 wt %) than that of untreated coal (40-50 wt %). Thus it was established that both these high calcium coals can be effectively and uniformly treated in the FPU.

After these experiments, the FPU was modified to test the effectiveness of the pretreatment in the expected temperature range for a commercial process fluidized bed unit. Two experiments were run in this temperature range (100-120°F). Tubing bomb experiments were carried out to determine the effectiveness of these tests. The results of these experiments showed that there was 16 wt % CaCO<sub>3</sub> on liquefaction residue ash. Comparing this result with those for untreated and room temperature pretreated samples shows that although this factor is significantly lower than that of untreated coal, it is higher than the room temperature result. This finding agrees directionally with the kinetic study of SO<sub>2</sub> pretreatment made in the BSPU.

Figure 4-1

BENCH SCALE SO<sub>2</sub> PRETREATMENT KINETIC STUDY RESULTS



- 85°F, 5% SO<sub>2</sub>
- 85°F, 10% SO<sub>2</sub>
- 65°F, 5% SO<sub>2</sub>
- 65°F, 10% SO<sub>2</sub>
- ▲ Total Sulfur Content of Original Coal

#### 4.2 Modification of the Once-Through Coal Liquefaction Unit (OTCLU) and Validation of Yields

In the past, OTCLU has been extensively used to address operability problems (see January 1976-June 1977, EDS Final Technical Progress report [FE-2353-20] and July 1977-June 1978, EDS Annual Technical Progress Report [FE-2893-17]). Over this reporting period, the unit was modified to allow the generation of yield information.

With this unit modification the product slurry is stripped with hydrogen in a hot separator to recover the liquid. The bottoms product thus obtained, containing 35-55 wt % 1000°F<sup>-</sup> content by Micro-lube distillation, is collected in the bottoms storage vessel through an auto-actuated valve. The product liquid is stored in the product liquid accumulator. Figure 4-2 is a schematic representation of the unit in its present configuration.

After the initial modifications, several trial runs were carried out to debug the unit, to check overall material balance and to generate elementally balanced yield information for comparison with RCLU results at similar conditions. These runs were with Illinois No. 6, Monterey No. 1 mine coal and hydrogenated multi-pass spent solvent (MPSS). The solvent-to-coal ratio was kept at 1.6. Nominal residence time was 40 minutes and the reaction temperature was 840°F. These runs resulted in overall measured material balances ranging from 92 to 96%. The hydrogen consumption, water yield and gas yield were comparable to corresponding RCLU data. However, the C<sub>4</sub>-1000°F liquid yield was lower (✓10%) and the 1000°F<sup>+</sup> bottoms yield correspondingly higher (✓8%).

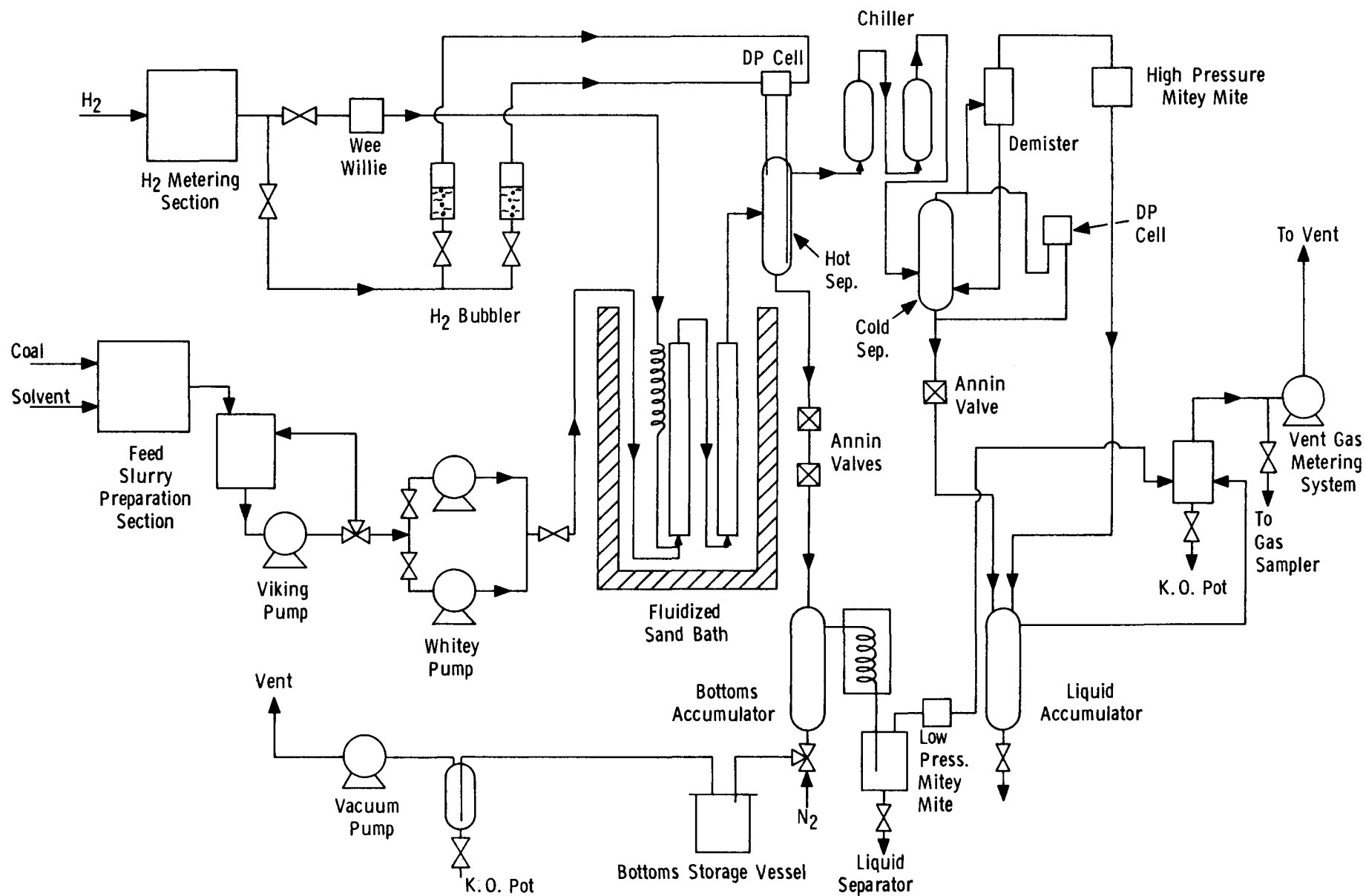
Based on this initial experience with the unit, a test program was initiated to improve the overall material balance on the unit and to reconcile differences in liquid and 1000°F<sup>+</sup> bottoms yield between OTCLU and RCLU.

As a part of this program, several further modifications were made on the unit to ensure proper accounting for the products (both liquid and bottoms). Also, a detailed operational procedure was implemented to reduce any uncertainties in measuring input and output streams. Weight scales and gas flow meters were recalibrated.

With these modifications, four yield periods have been carried out with Illinois No. 6, Monterey No. 1 mine coal at 840°F reaction temperature and 40 minutes nominal residence time. These runs resulted in very good overall measured material balance (98-100%). Results of these runs are compared in Table 4-1 with liquefaction results of similar RCLU runs. It is apparent from this table that, within experimental error, there is good agreement between OTCLU and RCLU yields in terms of gas, water, C<sub>4</sub>-1000°F<sup>-</sup> liquid and 1000°F<sup>+</sup> bottoms. Hydrogen consumptions for this unit and the liquefaction section of RCLU are comparable. A modification in the bottoms product distillation procedure also helped achieve better estimation of the 1000°F<sup>-</sup> content of the bottoms.

FIGURE 4-2

OTCLU FLOW DIAGRAM



Another series of runs with Monterey No. 1 mine coal has been completed to check the reproducibility of the unit. Analyses of the various product streams are in progress. In the future, the unit will be further validated with additional coals and at different operating conditions.

TABLE 4-1

COMPARISON OF OTCLU AND RCLU YIELDS

Run Conditions: 840°F/40 minutes  
Solvent/Coal = 1.6  
Illinois #6, Monterey Mine No. 1 coal

Yield (Elementally balanced)  
(Lbs/100 Lbs of Dry Coal)

	<u>OTCLU</u>			<u>RCLU*</u>		
	<u>YP 493</u>	<u>YP 494</u>	<u>YP 495</u>	<u>YP 496</u>	<u>Avg.</u>	<u>(Liquefaction)</u>
Overall Measured	98.4	100	100	99.9		
Material Balance						
Components						
H <sub>2</sub>	-1.4	-1.4	-1.6	-1.6	-1.5	-1.4
H <sub>2</sub> O	7.1	8.4	6.9	6.9	7.3	6.6
CO <sub>x</sub>	1.2	1.5	1.7	1.7	1.5	1.7
NH <sub>3</sub>	0.1	0.1	0.1	0.0	0.1	0.1
H <sub>2</sub> S	3.1	3.2	3.2	3.2	3.2	2.7
C <sub>1</sub> -C <sub>3</sub>	6.8	6.3	7.6	6.5	6.8	7.3
C <sub>4</sub> -1000°F	35.0	32.4	34.5	33.1	33.8	32.7
1000°F+	48.1	49.5	47.7	50.2	48.9	50.6

\*Average data for YP's 564-566 (Ref. October-December, 1978, EDS Quarterly Technical Progress Report [FE-2893-25]).

### 4.3 Correlation of Coal Conversion with Coal Properties

Stepwise multiple regression analysis of tube autoclave conversion data has been completed for six different coals and coal types. This is directed at developing a correlation expressing liquefaction conversion as a function of measurable coal properties and is similar to an effort underway at Pennsylvania State University.<sup>1</sup> Principal differences in the two studies include differences in feed coal preparation, conditions used for liquefaction, and in the definition of liquefaction conversion. As pointed out by Given, requirements for a valid statistical correlation include an extensive data base and a wide range of variability for each property studied. The work to date represents a modest beginning in this regard. More significance can be attached to the correlation as additional data are included.

Data used in the initial correlation work are given in Table 4-2. Independent variables include ultimate, proximate, and petrographic analyses. The dependent variable is taken to be the tubing bomb conversion (DAF) at 840°F and 40 minutes residence time, based on cyclohexane insoluble residue. Coals to date include two bituminous coals, two bituminous coal lithotypes, a subbituminous coal, and a lignite.

Results of single variable linear regression analyses are given in Table 4-3. Significance of each variable in explaining liquefaction behavior is expressed in terms of the percent of variation about the mean removed by regression. This is frequently referred to as the index of determination ( $R^2$ ). Percent volatile matter was found to be the most significant variable, while percent mineral matter was the least significant. Only two variables were found to be of significance when all variables were simultaneously included in a stepwise linear regression analysis. These were percent volatile matter and percent total sulfur. The resulting index of determination was 96.8. Volatile matter was highly correlated with percent carbon, H/C atomic ratio, and percent exinite while total sulfur was not highly correlated with any other independent variables.

The resulting correlation was used to predict liquefaction conversion for several additional coals for which sufficient data for comparison was available. The result is shown in Figure 4-3. Predictions were made for four Gulf Coast lignites, two Pittsburgh Seam coals, two Australian coals (one a lignite and the other subbituminous), and an Illinois No. 6 coal. While some scatter does exist about the parity line, considering the limited number of coals and the great diversity of coal types included in the data base, the results are considered to be encouraging.

Several additional coals have been acquired for inclusion in the correlation data base in order to better cover the range of variables considered in the analysis. These are listed in Table 4-4. This will be a continuing activity throughout 1979 as more data become available.

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<sup>1</sup>Given, P.H., et al., "Dependence of Coal Liquefaction Behavior on Coal Characteristics." 3. Statistical Correlations of Conversions in Coal Tetralin Interactions, FUEL, 57, February, 1978.



TABLE 4-2

COAL SCREENING DATA BASE

COAL	Pennsylvania Bituminous	Illinois #6 Bituminous	Wyoming Subbituminous	North Dakota Lignite	Illinois #6 Lithotypes	
					Vitrain	Fusain
<u>Ultimate Analysis (Wt %, DMMF)</u>						
Carbon	90.95	80.71	71.74	72.35	79.03	91.47
Hydrogen	4.84	5.15	5.44	4.66	5.44	2.99
Oxygen (Diff.)	2.22	9.31	21.50	21.54	11.64	3.88
Sulfur						
Total	0.87	5.64	0.53	0.86	3.82	2.16
Organic	0.55	3.39	0.32	0.56	2.48	1.12
Nitrogen	1.44	1.44	1.16	0.89	1.41	0.54
<u>Proximate Analysis (Wt %)</u>						
Mineral Matter (Dry)	7.8	14.5	8.8	6.2	6.6	14.07
Volatile Matter (DMMF)	19.3	43.9	48.4	46.5	44.0	17.5
H/C Atomic Ratio	0.65	0.78	0.90	0.78	0.83	0.41
<u>Petrographic Analysis (Vol. %)</u>						
Vitrinite	86.8	83.4	87.9	80.5	94.2	9.5
Exinite	0.0	5.4	4.3	2.7	2.3	0.4
Total Reactive Macerals	86.8	88.8	92.2	83.2	96.5	9.5
<u>Tubing Bomb Liquefaction Conversion</u>	9.7	56.6	51.9	47.5	59.3	19.3
(DAF) (840°F/40 Min/4 wt % H <sub>2</sub> )						

TABLE 4-3

RESULTS OF SINGLE VARIABLE REGRESSION

<u>Independent Variable</u>	<u>Percent of Variation About Mean Removed By Regression (<math>R^2</math>)</u>
Volatile Matter	88.5
% Exinite	70.6
% Carbon	66.4
H/C Atomic Ratio	61.5
% Oxygen (Org.)	46.8
% Hydrogen	40.4
% Exinite + Vitrinite	30.4
% Vitrinite	26.3
% Sulfur (Total)	21.6
% Nitrogen	9.5
% Mineral Matter	1.5

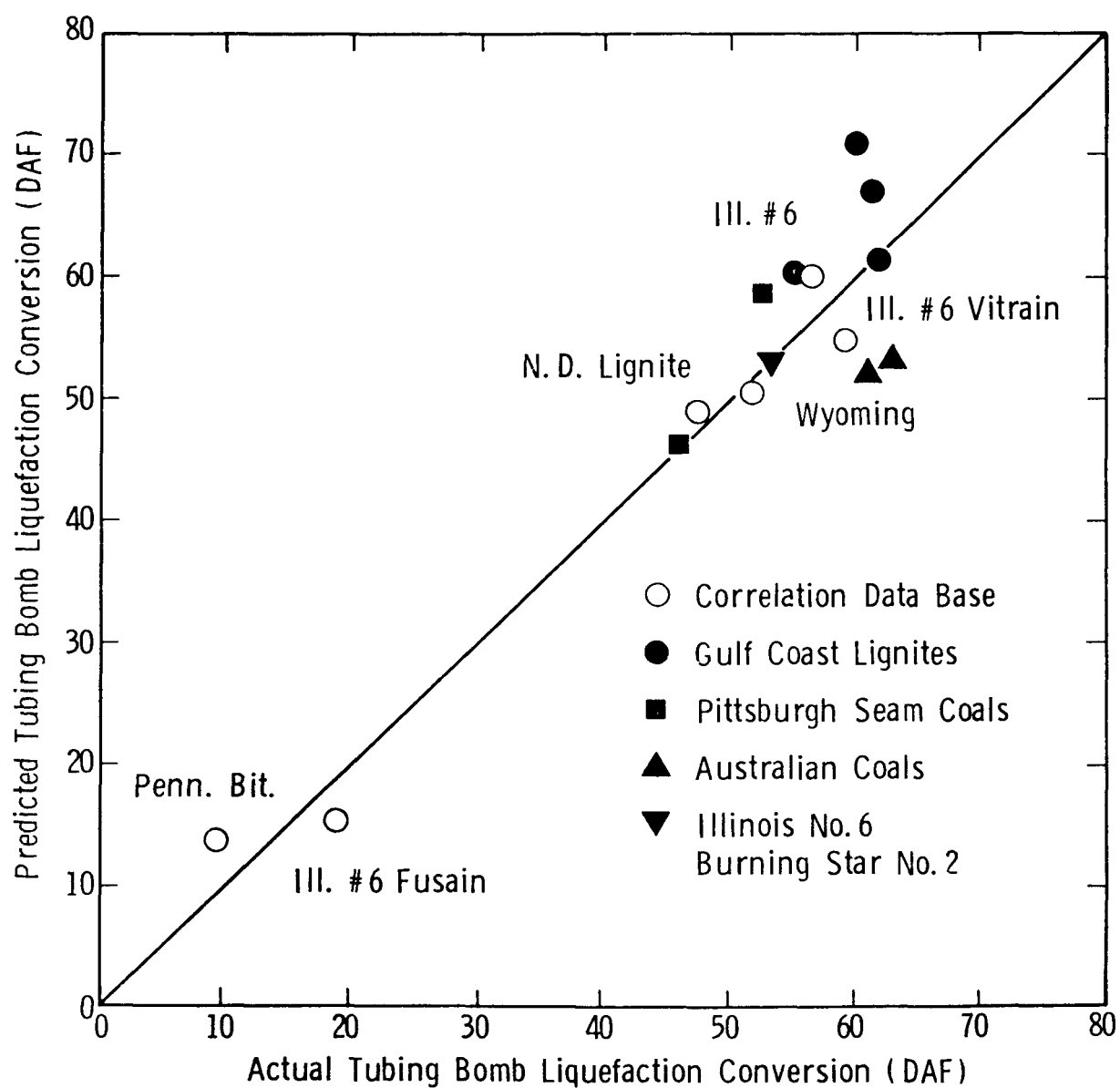
TABLE 4-4

SAMPLES ACQUIRED FOR SCREENING PROGRAM

<u>Region</u>	<u>Seam</u>	<u>County</u>	<u>State</u>	<u>ASTM Rank</u>
Rocky Mountain	#8	San Juan	NM	SBA-HVC
	Wadge	Routt	CO	HVC
	Lower Sunnyside	Carbon	UT	HVB
Western Interior	Bevier	Howard	MO	HVC-HVB
	Fleming	Bourbon	KS	HVC-HVB
	Unnamed	Jasper	IO	HVC-HVB
Appalachian	Lower Kitanning	Cambria	PA	LV
	Pittsburgh	Somerset	PA	MV-HVA
	Pittsburgh	Washington	PA	HVA
	Upper Freeport	Beaver	PA	HVA

FIGURE 4-3

PARITY PLOT FOR TWO PARAMETER CORRELATION



#### 4.4 Coal Screening

- Comparison of Burning Star and Monterey Coal Conversion

Tubing bomb liquefaction experiments were carried out, in support of RCLU operations, to determine the effects of process variables on the liquefaction conversion of two Illinois No. 6 coals. These coals were from two different mines, Burning Star No. 2 and Monterey No. 1, and were liquefied at three different temperature/ reaction time combinations with tetralin as the solvent. The solvent-to-coal weight ratio was 1.6 and molecular hydrogen was present at 4 wt % on coal. The process variable combinations used in these experiments are shown in Column 1 of Table 4-5.

The results of these experiments are also presented in Table 4-5. Both conversion and gas make are expressed as weight percent on dry coal. For all process variable combinations studied, Monterey coal shows higher conversions than Burning Star coal, which is consistent with RCLU results. At the same experimental conditions, Monterey coal conversions are approximately 3 to 8 wt % higher than Burning Star coal conversions. To summarize, Burning Star coal from the No. 2 mine gave less conversion and was less sensitive to process variable changes than was coal from the Monterey No. 1 mine.

TABLE 4-5

EFFECT OF PROCESS VARIABLES ON TUBING BOMB  
CONVERSION AND GAS MAKE FOR TWO ILLINOIS NO. 6 COALS

<u>Temp./Reaction Time</u> <u>(°F/Min.)</u>	<u>Delta Cyclohexane</u> <u>Conversion,*</u> <u>Wt % on Dry Coal</u>	<u>Delta</u> <u>Gas Make,*</u> <u>Wt % on Dry Coal</u>
840/40	3.4	-1.3
880/25	8.4	2.1
800/100	6.1	-0.1

\*Delta refers to Monterey No. 1 minus Burning Star No. 2 results.

- Tubing Bomb Liquefaction of Burning Star No. 2 Coal Sample Obtained from Hydrocarbon Research, Inc. (HRI)

Three Burning Star No. 2 coal samples (obtained by EPRI from HRI) were liquefied in tubing bomb autoclaves in order to compare these coal samples used in the H-coal development work with the Burning Star No. 2 coal sample used in the coal screening program underway in the EDS Project. The experiments were conducted at 840°F reaction temperature and 40 minutes reaction time. The solvent (tetralin)-to-coal ratio was 1.6 and molecular hydrogen was present at 4 wt % on coal.

The three samples showed essentially the same conversion and gas make on a dry coal basis. No significant differences were observed between these results and results obtained on the Burning Star No. 2 sample currently in use in the coal screening program. The proximate and ultimate analyses for the three HRI samples and the analyses of the Burning Star No. 2 sample used in the coal screening program were also comparable.

- Effect of Process Variables on Liquefaction Conversion of Burning Star No. 2 Coal

Illinois No. 6 coal from the Burning Star No. 2 mine has not shown the same degree of conversion sensitivity to changes in liquefaction severity as has been obtained for Illinois No. 6 coal from the Monterey No. 1 mine. This has been observed both in preliminary, on-line RCLU-1 results (October, 1978, EDS Monthly Technical Progress Report [FE-2893-22]) and from tubing bomb autoclave studies. Additional tubing bomb experiments were performed to determine if increased conversion is possible when operating at a higher hydrogen treat rate or with the combination of a relatively low temperature (800°F) and long holding time (140 minutes). Tetralin was used as solvent at a 1.6 solvent-to-coal ratio. Results are given in Table 4-6.

TABLE 4-6  
EFFECT OF PROCESS VARIABLES ON TUBING BOMB  
CONVERSION AND GAS MAKE FOR BURNING STAR NO. 2 COAL

	<u>Cyclohexane</u> <u>Conversion</u> <u>(Wt % on Dry Coal)</u>		<u>Gas Make</u> <u>(Wt % on Dry Coal)</u>	
<u>Hydrogen Treat</u> <u>(Wt % on Dry Coal)</u>	<u>4.0</u>	<u>6.5</u>	<u>4.0</u>	<u>6.5</u>
<u>Temp./Reaction Time</u> <u>(°F/Min.)</u>				
840/40	Base	0	Base	0
800/100	+4	---	0	---

In the presence of tetralin as solvent at 840°F/40 minutes, increasing initial hydrogen charge from 4.0 wt% to 6.5 wt% had no effect on either conversion or gas make. This result complements a RCLU study which indicated that increasing donor hydrogen availability by increasing solvent-to-coal ratio did not affect conversion or yield distribution. (See November, 1978, EDS Monthly Technical Progress Report [FE-2893-23]).

Using a low temperature with long residence time resulted in a conversion which was approximately 4 wt % higher than achieved at 840°F and 40 minutes reaction time. There was no significant change in gas make.

- Effect of Process Variables Study on Liquefaction Conversion of Pittsburgh Seam Ireland Mine Coal

A tubing bomb liquefaction process variable study was conducted for Ireland mine coal to provide guidance for the upcoming process variable study in RCLU. Five experiments were performed to test the effect of reaction time and temperature on liquefaction conversion. The reaction temperature/time combinations (°F/minutes) for these experiments were 840/40, 860/40, 880/40, 840/60 and 840/100. The solvent (tetralin)-to-coal ratio was constant at 1.6.

At 40 minutes reaction time, increasing the reaction temperature from 840 to 880°F resulted in approximate increases of 7 wt % and 4 wt % in conversion and gas make, respectively. Increasing the residence time from 40 to 100 minutes at 840°F reaction temperature resulted in the liquefaction conversion and gas make increasing by 9 wt % and 3 wt %, respectively. Assuming the hydrogen consumption and water make to be constant over these ranges of temperature and reaction time studied, then approximately 6 wt % increase in liquid make would be expected by increasing the reaction time from 40 minutes to 100 minutes at 840°F. For a constant 40 minutes reaction time, an increase in liquid yield of approximately 3 wt % would be expected when the reaction temperature is increased from 840°F to 880°F.

- Screening of Australian Coals

In order to select an Australian coal for additional EDS testing, proximate and ultimate analyses and tubing bomb liquefaction tests were conducted on two screening program candidate Australian coals. These coals were a subbituminous (Wandoan) and a brown coal.

Tubing bomb liquefaction tests were conducted at a temperature of 840°F and 40 minutes reaction time. Solvent (tetralin)-to-coal ratio was 1.6. Results of these tests were compared on a dry, ash-free basis since the Wandoan coal sample contained appreciably more mineral matter (~20 wt %) than did the brown coal (~3 wt %).

Overall conversions (DAF) were comparable at just over 60 wt % and total hydrogen consumption was about 3 wt % in each case. However, the Wandoan subbituminous coal produced 5.7 wt % more gas (C<sub>1</sub>-C<sub>3</sub> plus CO<sub>x</sub>) than did the brown coal. Since the brown coal had a higher organic oxygen content (~26 wt %) than the Wandoan coal (~16 wt %), it may be concluded that the brown coal produced more water than the Wandoan. These results did not, by themselves, support one coal as a clear-cut choice over the other for further EDS testing.

Data supplied with the two samples indicated that, on an as received basis, the Wandoan and brown coals contain approximately 7.7 wt % and 60.2 wt % moisture, respectively. Based principally on this factor, other factors being approximately equal, a recommendation has been made that the Wandoan coal be selected for additional EDS testing.

A 26 drum shipment of Wandoan coal has subsequently been received for RCLU-1 screening studies. Tubing bomb liquefaction tests were conducted on this sample to insure that the shipment was consistent with the earlier, smaller sample tested. Conversions on the two samples were essentially equal on a dry, ash-free basis.

• Liquefaction Conversion of Wyoming Coal Bottoms from CLPP

This study was conducted to test the reactivity of Wyoming coal bottoms during bottoms recycle. Tubing bomb liquefaction experiments were conducted with two CLPP bottoms samples liquefied separately in the absence of coal. The bottoms samples were produced from Wyoming coal at liquefaction residence times of 25 and 60 minutes, and are referred to as 25- and 60-minute bottoms, respectively. Wyoming coal was liquefied concurrently with each bottoms sample as a base case to ensure consistency with previous experiments.

The tubing bomb liquefaction of 25-minute bottoms was carried out at reaction times of 25 and 60 minutes. Liquefaction of 60-minute bottoms was carried out at a reaction time of 60 minutes only. Tetralin was used as the solvent for these experiments at a solvent-to-feed (either bottoms or coal) weight ratio of 1.6. The reaction temperature was constant at 840°F. These conditions are summarized with the results in Table 4-7.

TABLE 4-7

TUBING BOMB LIQUEFACTION OF WYOMING COAL BOTTOMS

<u>Operating Conditions</u>	<u>25 Min. CLPP Bottoms</u>		<u>60 Min. CLPP Bottoms</u>
Temperature, °F	840	840	840
Residence Time, Min.	25	60	60
Solvent/Coal/Bottoms	1.6/0/1	1.6/0/1	1.6/0/1
Tubing Bomb Results*			
Conversion	30.3	34.0	25.7
Gas Make	1.8	3.0	2.4

\*Wt % on dry solids. Conversion based on cyclohexane insolubles.

Conversion and gas make results for these experiments are expressed as weight percent on bottoms. At a reaction time of 60 minutes, the conversions for 25-minute bottoms and 60-minute bottoms were 34.0 and 25.7 wt %, respectively. At a reaction time of 25 minutes the liquefaction conversion for 25-minute bottoms was 30.3 wt %. Results for Wyoming coal run at the base case were consistent with previous experience. These results indicate that significant additional liquid yield can be achieved by recycling Wyoming bottoms, at least when no coal is present to compete for solvent donatable hydrogen.

A more comprehensive study of bottoms recycle potential for all program and sponsor coals was conducted subsequent to this study and is reported later in this section.

- Atmospheric Bottoms Recycle Simulation

Three sets of tubing bomb liquefaction experiments were conducted to simulate recycle of Wyoming coal-derived atmospheric tower bottoms to liquefaction. The experiments were at a 840°F reaction temperature for 40 minutes reaction time, and 4 wt % hydrogen on coal. Atmospheric tower bottoms were simulated by combining vacuum bottoms (A-1) and vacuum gas oil (VGO, A-2) samples. The A-1 and A-2 samples were obtained from CLPP yield periods which were conducted with Wyodak coal at liquefaction conditions of 840°F and 40 minutes residence time.

The feed composition and results obtained from each run are summarized in Table 4-8 (next page). Coal and tetralin were used as a base case for all three experiments. These results were used to calculate bottoms conversion and gas make. From the results of experiment #1, the bottoms conversion and percent gas make from bottoms were calculated assuming no synergistic effect between coal and bottoms. Similarly, conversion and gas make from VGO were calculated from experiment #3 assuming no synergism between coal and VGO. Bottoms conversion and gas make in the presence of VGO were calculated from experiment #2 using the results from #3. These results are presented in Table 4-9 for comparison.

TABLE 4-9

ESTIMATED BOTTOMS AND VGO CONVERSIONS AND GAS YIELDS

<u>Experiment Number</u>	<u>Bottoms Results (Wt % on Bottoms)</u>		<u>VGO Results (Wt % on VGO)</u>	
	<u>Conversion</u>	<u>Gas Make</u>	<u>Cyclohexane Insolubles</u>	<u>Gas Make</u>
1	26.6	1.0	---	---
2	21.6	5.6	---	---
3	---	---	9.4	0



TABLE 4-8

SIMULATED ATMOSPHERIC BOTTOMS RECYCLE  
FEED COMPOSITION AND OVERALL RESULTS

<u>Experiment Number</u>	<u>Tube Designation</u>	<u>Feed Composition (Grams)</u>				<u>Overall Results (Wt % on Dry Coal + Bottoms)</u>	
		<u>Coal</u>	<u>Bottoms</u>	<u>Tetralin</u>	<u>VGO</u>	<u>Conversion<sup>1</sup></u>	<u>Gas Make</u>
1	A	2	1	3.2	0	40.4	8.5
	B <sup>2</sup>	3	0	3.2	0	47.3	12.4
2	A	2	1	3.2	1	35.4	8.8
	B	3	0	3.2	0	47.4	10.4
3	A	2	0	3.2	1	42.4	12.5
	B	3	0	3.2	0	47.1	12.6

<sup>1</sup>Conversion based on cyclohexane insolubles.

<sup>2</sup>Tubes designated "B" were used as the base case.

From Table 4-9, it is seen that the estimated conversion of recycled vacuum bottoms material was found to decrease by 5 wt % when vacuum gas oil was included with the vacuum bottoms. At the same time, gas make increased by 4.6 wt %. In addition, 9.4 wt % of the vacuum gas oil was found to convert to cyclohexane insoluble material. These results are consistent with earlier results obtained for staged liquefaction of Illinois No. 6 coal in RCLU-2 (EDS 1976 Annual Technical Progress [FE-2353-9]).

● Tubing Bomb Investigation of Bottoms Recycle of Sponsor Coals

Based on optimistic results obtained simulating bottoms recycle in RCLU and tubing bombs with Illinois and Wyoming coals, a tubing bomb study to screen sponsor coals for possible yield incentives for operating the EDS process in a bottoms recycle mode was conducted. The primary objective of the project was to estimate the extent to which the liquefaction residues for the sponsor coals can be converted. A second objective of the study was to determine if a yield sensitivity to solvent-to-solids ratio can be identified in tubing bombs. Past experience with Wyodak coal bottoms recycle simulations in RCLU indicate such a sensitivity exists in the pilot units, although earlier tubing bomb studies using tetralin as solvent did not demonstrate the same sensitivity. A final objective of the study was to determine if any conversion interaction occurs between the coal and bottoms.

The seven program and sponsor coals employed in this study range in rank from lignite to bituminous. Those coals investigated included a Texas (Big Brown) lignite, Wyoming (Wyodak) and Australian (Wandoan) subbituminous coals, two Illinois No. 6 (Monterey No. 1 mine and Burning Star No. 2 mine) and two Pittsburgh seam (Ireland and Arkwright mines) bituminous coals. Liquefaction residues employed in the study had been generated on RCLU at operating conditions of 840°F and 40 minutes nominal residence time during preliminary screening studies on each of the coals.

Preliminary bottoms recycle simulations for Wyodak coal using Illinois coal-derived hydrogenated MPSS as solvent indicated the desired conversion sensitivity to solvent-to-solids ratio could be identified in tubing bombs. Increasing the solvent/coal/bottoms ratio from 1.6/1/.5 to 2.4/1/.5 resulted in a 5.1% increase in cyclohexane conversion for the mixture based on total solid feed. As a result of these findings, hydrogenated MPSS was employed as solvent throughout the study.

During the initial part of this study, neat bottoms were liquefied at a temperature of 840°F for 40 minutes reaction time, with a 4% hydrogen treat rate and 1.6 solvent-to-bottoms ratio. Results of these experiments are presented in Table 4-10. Cyclohexane conversions are based on 1000°F<sup>+</sup> content of the RCLU bottoms and gas makes are based on total bottoms feed. Different bases are used for these results because all conversion of feed bottoms to 1000°F<sup>-</sup> material is assumed to come only from 1000°F<sup>+</sup> material, whereas in this study the source of gas produced was not assumed to be necessarily solely attributed to 1000°F<sup>+</sup> material.

TABLE 4-10

TUBING BOMB CONVERSION OF RCLU LIQUEFACTION RESIDUES FOR EDS PROGRAM  
AND SPONSOR COALS AT BASE CASE\* CONDITIONS

	<u>1978</u> <u>Ireland</u>	<u>Arkwright</u>	<u>Burning</u> <u>Star</u>	<u>Monterey</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big</u> <u>Brown</u>
Cyclohexane Conversion (wt % on 1000°F+ bottoms)	26.6	22.0	21.2	23.2	20.2	16.1	11.4
Gas Make (wt % on feed bottoms)	3.8	4.6	6.5	4.4	3.8	3.6	2.6
SO <sub>3</sub> -free Ash Content of Feed Bottoms (wt % on feed bottoms)	13.8	9.0	15.4	22.3	36.2	12.0	26.8
RCLU YP	515	526	580	498	628	327	532

\*T = 840°F

t = 40 minutes

P<sub>H<sub>2</sub></sub> = 1500 psi

4% hydrogen treat rate

The tubing bomb conversion for Monterey bottoms (23%) agrees well with RCLU conversion of neat bottoms (20%, by microlube) as reported in the October-December, 1978, Quarterly Technical Progress Report (FE-2893-25). Cyclohexane conversions for all the bituminous coals and Wandoan coal are roughly equivalent to 22%, by weight. Gas makes are also approximately the same (4%). Wyodak bottoms yield only 16% conversion and Big Brown bottoms give only 11% conversion. Gas yields from these bottoms do not appear to be significantly different from the other coals. Difference between the Wyodak conversion and that reported for Wyodak bottoms earlier in this section can be explained by the different bases used to express conversion and solvent effects. The Ireland bottoms from RCLU yield period 515 gave significantly more conversion than the other liquefaction residues, lending further credence to the contention that yields obtained during the 1978 screening study may not have been representative of the coal's actual liquefaction potential.

For the remainder of the project, mixtures of coal plus bottoms in a 2/1 ratio were liquefied at 840°F and 40 minutes reaction time for solvent-to-solids ratios of 1.5 and 1.07. A set of tubing bombs containing coal at the same solvent-to-solids ratio was used as reference in each experiment. Results for the mixtures are presented in Table 4-11. Differences in gas make were observed, but are probably not significant and conversion decreased slightly for each coal when less solvent was used. Results for tubing bomb conversion of the coals at the two solvent-to-coal ratios are shown in Table 4-12. There does appear to be a sensitivity of coal conversion to solvent quantity for each of the coals.

The conversion of the bottoms can be calculated in each case by backing the results for coal only out of the results for the mixtures of coal plus bottoms. These calculated values are presented in Table 4-13. The results indicate that the conversion and gas yield for Wandoan coal and the bituminous coals are independent of the solvent-to-solids ratio (in the range studied) and are not affected by the presence of coal. Wyodak and Big Brown samples, on the other hand, showed a very strong conversion synergism of about 10 wt % (on bottoms) between coal and bottoms. In addition, this synergism for Wyodak coal plus bottoms was strongly affected by the solvent-to-solids ratio employed.

#### 4.5 Fundamental Model for Coal Liquefaction

Development of a fundamental model for coal liquefaction was conducted to gain additional insight into liquefaction behavior by identifying reaction mechanisms by which conversion can occur. The approach to this effort has been to group individual chemical species into lumped components on the basis of similar chemical reactivities. The initial first-pass formulation consists of a minimum number of lumped components and kinetic reaction equations to describe the production and/or consumption of these components. Any subsequent refinements in the model would be achieved by progressively decreasing the size of the lumps and increasing the number of corresponding kinetic reaction equations. The

TABLE 4-11

COMPARISON OF EFFECTS OF SOLVENT-TO-SOLIDS RATIO ON CONVERSION  
FOR MIXTURES OF SPONSOR COALS PLUS BOTTOMS

T = 840°F      t = 40 minutes      Coal/Bottoms = 2.0      4% Hydrogen Treat

	<u>Ireland</u>	<u>Arkwright</u>	<u>Burning Star</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big Brown</u>
<u>Solvent-to-Solids = 1.6</u>						
% Cyclohexane Conversion	38.8	32.0	35.6	32.8	39.5	38.2
Gas Make	7.8	5.8	6.0	7.4	9.8	10.2
SO <sub>3</sub> -free Ash Content of Feed	10.3	7.9	12.3	27.3	9.0	17.9
RCLU Yield Period	515	526	580	628	327	532
<u>Solvent-to-Solids = 1.07</u>						
% Cyclohexane Conversion	35.4	30.0	32.8	31.8	36.0	35.8
Gas Make	5.6	5.3	5.9	6.6	8.6	10.7
SO <sub>3</sub> -free Ash Content of Feed	10.4	7.3	12.3	27.2	9.0	17.8
RCLU Yield Period	515	526	580	628	327	532

Units on all variables are wt % on dry solid (coal + bottoms) feed.

TABLE 4-12

COMPARISON OF EFFECTS OF SOLVENT-TO-COAL RATIO ON CONVERSION FOR SPONSOR COALS

T = 840°F    t = 40 minutes    4% Hydrogen Treat Rate

	<u>Ireland</u>	<u>Arkwright</u>	<u>Burning Star</u>	<u>Monterey</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big Brown</u>
<u>Solvent-to-Coal = 1.6</u>							
% Cyclohexane Conversion	41.5	36.0	38.2	42.2	37.1	41.0	43.8
Gas Make	9.0	7.0	8.4	8.5	9.8	13.4	13.6
SO <sub>3</sub> -free Ash Content of Feed	8.6	6.7	9.6	10.2	22.5	7.5	14.0
<u>Solvent-to-Coal = 1.07</u>							
% Cyclohexane Conversion	37.0	32.4	34.7		34.6	39.6	40.0
Gas Make	6.9	6.4	8.1		8.6	11.2	14.4
SO <sub>3</sub> -free Ash Content of Feed	8.6	6.7	9.6		22.1	7.3	14.1

Units on all variables are wt % on dry coal feed.

TABLE 4-13

CALCULATED CONVERSIONS FOR SPONSOR COAL BOTTOMS BASED ON  
DATA FOR COAL AND MIXTURES OF COAL PLUS BOTTOMS

T = 840°F      t = 40 minutes      4% Hydrogen Treat Rate

	<u>Ireland</u>	<u>Arkwright</u>	<u>Burning Star</u>	<u>Wandoan</u>	<u>Wyodak</u>	<u>Big Brown</u>
<u>Solvent-to-Solids = 1.6</u>						
% Cyclohexane Conversion	25.4	17.5	23.8	21.6	28.4	19.2
Gas Make	5.4	3.4	1.2	2.6	2.6	3.4
SO <sub>3</sub> -free Ash Content	13.7	10.4	17.7	36.9	12.0	25.7
<u>Solvent-to-Solids = 1.07</u>						
% Cyclohexane Conversion	24.2	18.9	22.1	23.5	18.4	19.3
Gas Make	3.0	3.1	1.5	2.6	3.4	3.3
SO <sub>3</sub> -free Ash Content	12.9	8.7	17.7	37.3	12.4	25.4

Conversion calculation based on 1000°F<sup>+</sup> content of bottoms.  
 Other data based on entire bottoms charge.

definition of the lumped components has been formulated in a manner somewhat consistent with the fundamental solvent hydrogenation model so as to provide a basis for interaction between the two models. Definition of components for the two models are not entirely consistent due to the better degree of chemical characterization possible in the solvent hydrogenation system. This fact may establish a limitation in the manner in which the solvent hydrogenation and liquefaction models can be used interactively.

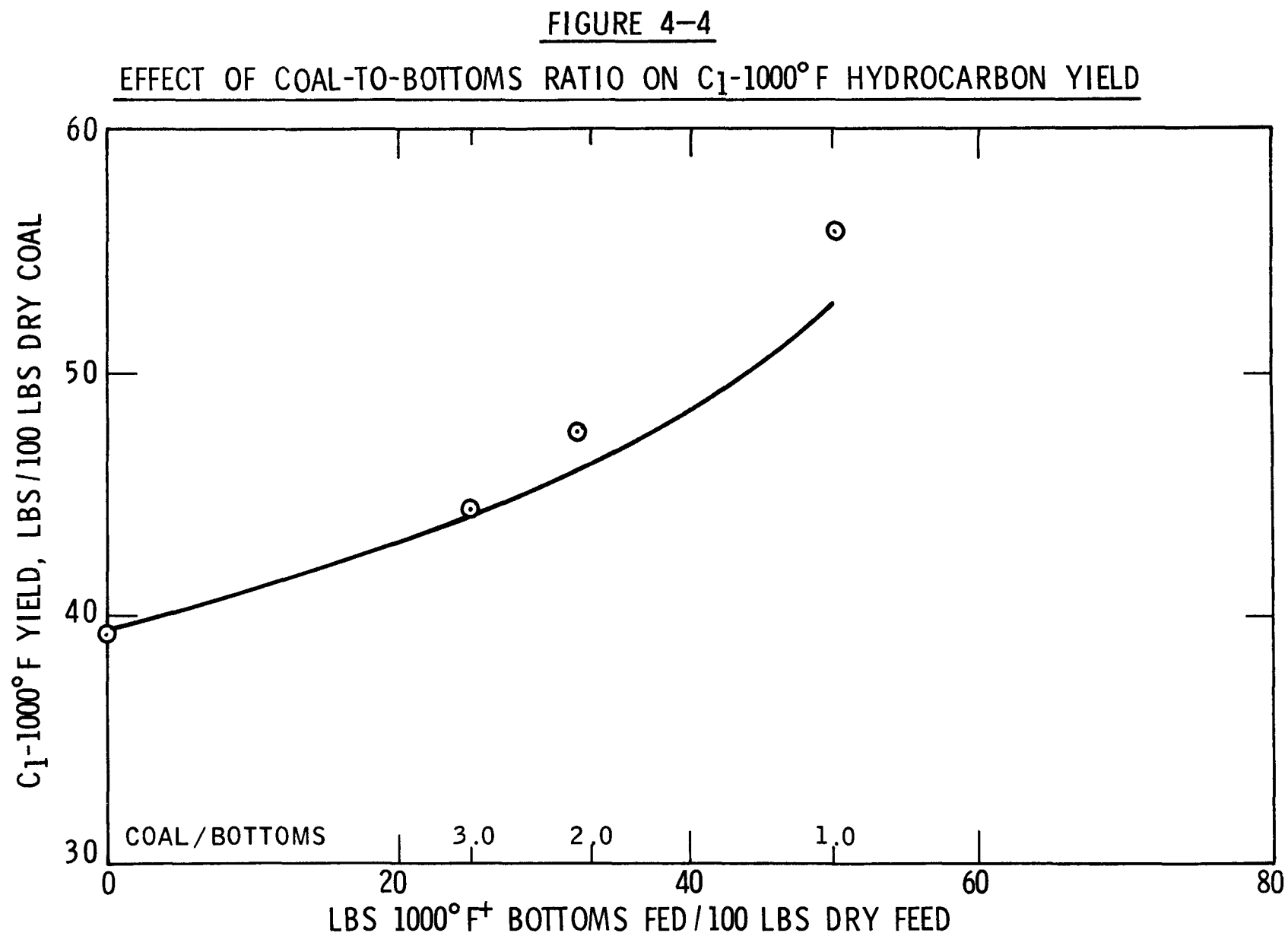
A final fit of the model to the model data base was achieved during the third quarter of 1978, as reported in the July-September, 1978, EDS Quarterly Technical Progress Report (FE-2893-21). Final parameter values were then employed by the model to simulate experimental yield periods covering a variety of conditions not included in the model data base. The generally good agreement between model predictions and the experimental results served to further confirm the validity of the model formulation and parameter estimates.

During the last quarter of 1978, this model was used to simulate modifications to the EDS Process. One set of model predictions simulated staged solvent liquefaction. Although experimental data from RCLU yield periods were not as yet worked up in a form which could be used for quantitative comparison, model predictions of increased yields over base case yields were directionally correct when compared with overall RCLU yield data.

The model was also employed to simulate RCLU yield periods during operation in a bottoms recycle mode. Conditions covered were for a liquefaction temperature of 840°F, 40 minutes space time and coal-to-bottoms ratios of 1.0, 2.0 and 3.0. Because of limitations inherent in the first-pass model, it was anticipated beforehand that predicted gas and liquid yields would be less than the corresponding experimentally observed yields. Figure 4-4 compares measured (RCLU) and predicted C<sub>1</sub>-1000°F hydrocarbon yields. The response of product yield as a function of coal-to-bottoms ratio was qualitatively accurate and was within approximately 3% agreement quantitatively on C<sub>1</sub>-1000°F yield.

Further simulations of bottoms recycle were conducted covering various temperatures (800, 820, 840, 860, 880°F) space times (25, 40, 60, 80, 100 minutes) and bottoms-to-coal ratios (0.3, 0.5, 1.0). These simulations were conducted to predict the effect of bottoms recycle operating conditions on liquefaction product yield distributions. Since pilot plant data have not been generated at these conditions, experimental verification of the model predictions is not possible at this time. The general trend predicted by the model is that C<sub>1</sub>-1000°F hydrocarbon yield increases as temperature and space time increase and as coal-to-bottoms ratio decreases. The selectivity ratio for 400-1000°F oil, defined as [400-1000°F yield/C<sub>1</sub>-1000°F yield], is predicted to decrease as temperature increases and coal-to-bottoms ratio decreases. These predictions pertain only to Monterey mine coal on a once-through recycle basis.





#### 4.6 Mathematical Modeling of Solvent Hydrogenation

During this past year, the fundamental solvent hydrogenation model was completed by the addition of Ni/Mo-10 catalyst kinetics for heteroatom removal to the updated version. With the completed model, simulations were conducted for one year Ni/Mo-10 catalyst life projections with both Illinois and Wyoming spent solvents. In addition, the solvent hydrogenation simulator was used to help explain why higher product donor hydrogen content was observed with Wyoming spent solvent than Illinois MPSS at similar operating conditions.

- Catalyst Age-Dependent Heteroatom Removal Kinetics

Heteroatom (sulfur, oxygen and nitrogen) removal activity is not only a function of operating temperature and space velocity, but also catalyst age and history. Bench unit data from the most recent Ni/Mo-10 catalyst activity maintenance study (14M21) was analyzed to determine this effect. The same non-selective deactivation model used for the solvent hydrogenation reactions, and reported in the July 1977-June 1978, EDS Annual Technical Progress Report (FE-2893-17), was applied to the heteroatom removal reactions.

Simulation of the catalyst activity maintenance study was made with and without heteroatom removal kinetics included as a function of relative catalyst activity. Experimental concentrations from Run 14M21 were compared to both sets of model-predicted concentrations over the life of the catalyst. Predictions made with catalyst activity-dependent removal kinetics provided a better fit of the experimental data than predictions made with a constant relative catalyst activity of 1.0. Thus, heteroatom removal kinetics were added to the EDS solvent hydrogenation model as a function of temperature, space velocity, catalyst age and catalyst history. Addition of Ni/Mo-10 catalyst kinetics for heteroatom removal as a function of catalyst age completed the updated version of the fundamental solvent hydrogenation model for the EDS process.

- Ni/Mo-10 Catalyst Life Simulations

Simulations using the fundamental solvent hydrogenation model were conducted to predict product donor hydrogen and catalyst activity levels for various operating conditions using Ni/Mo-10 catalyst and one year catalyst life. Different start-of-run temperatures and temperature histories were simulated for space velocities ranging from 1.32xRV to 2.63xRV using Illinois and Wyoming spent solvents as feedstocks. Model predictions indicated that desired levels of product donor hydrogen could be maintained for a year at the lower space velocity. However, desired donor hydrogen levels could not be sustained for a year at the higher space velocity.

● Wyoming MPSS Process Variable Study

A process variable study was carried out for Wyoming multi-pass spent solvent and Ni/Mo-10 catalyst at initial activity using the hydrotreater simulator. Temperatures and space velocities were varied over a wide range of operating conditions. Hydrogen partial pressure was kept constant at 1500 psig.

Results from these simulations were compared to experimental results obtained in the solvent hydrogenation bench units using the same feed and catalyst. Comparisons were made using simulated and experimentally determined product donatable hydrogen content. Experimental donatable hydrogen levels were higher than the simulated values over the ranges of temperature and space velocity studied. These results were different from those obtained for Illinois MPSS and Ni/ Mo-10 catalyst although the feed component concentrations were similar. In the Illinois study, predicted and experimental donatable hydrogens agreed reasonably well over the ranges of temperature and space velocity studied.

As shown in additional experimental solvent hydrogenation studies (see Laboratory Process Research and Development, Section 3) there was a subtle reason for the experimental donatable hydrogens being higher than the simulated values. These hydrogenation catalysts exhibit high initial activity prior to line-out after about 1 to 3 weeks off catalyst life. The experimental donor hydrogens had been obtained during the first 2 to 3 weeks of catalyst life. As a result, the simulated donor hydrogens were lower because the catalyst had a higher initial activity (prior to line-out) than was used in the simulations.

## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### LIQUEFACTION PROCESS RESEARCH

#### 5. Product Quality Studies

##### 5.1 Coal Naphtha Hydrotreating

The currently planned end use for EDS coal naphtha is catalytic reforming to make gasoline. Although these naphthas have high aromatic and naphthenic contents, thereby making them attractive reformer feeds, they also contain high levels of sulfur, nitrogen and oxygen which are reformer catalyst poisons. Coal naphthas therefore require more severe hydrotreating relative to petroleum-derived naphthas to meet reformer feed specifications.

Initial scoping studies had indicated that two-stage hydrotreating may be required to achieve the contaminant removal necessary for reforming coal naphthas (see January, 1976-June, 1977, EDS Final Technical Progress Report [FE-2353-20]). More extensive testing at the Baton Rouge Laboratories has demonstrated that reformer feed specifications can be achieved in a single-stage hydrotreater. In addition, the minimum hydrotreating conditions have been quantified along with the associated hydrogen consumption. Final documentation of these studies, including feed inspections and pilot unit data, is contained in the July-September 1978, EDS Quarterly Technical Progress Report (FE-2893-21).

Four coal naphthas were selected for hydrotreating upgrading studies. These were raw and caustic washed samples derived from CLPP operations with Illinois and Wyodak coals. Caustic washing removes phenolic compounds, thereby offering a potential two-fold process credit: reduced hydrogen consumption and severity in the hydrotreater along with product credits for the sale of recovered phenols.

Based on previous studies, which indicated nitrogen removal to be limiting, a Ni/Mo catalyst was chosen for upgrading. Conclusions from these pilot plant studies are summarized below:

- Reformer feed qualities can be met with all four coal naphtha feedstocks by hydrotreating in a conventional, single-stage hydrotreater.
- Minimum conditions required to meet reforming specifications were set by product nitrogen level. When a product nitrogen target of <1 wppm is met, sulfur and Bromine No. are sufficiently low to permit reforming with either platinum or bimetallic reforming catalysts.
- Relative difficulties of hydrotreating these feeds are demonstrated by the maximum space velocities at which <1 wppm nitrogen product is obtained. These conditions are summarized in Table 5-1.

- The Wyodak naphthas were more difficult to hydrotreat than the corresponding Illinois naphthas, requiring approximately a two-fold increase in severity. This is due to a higher final boiling point of the particular samples tested and/or the higher heteroatom content.

- Both raw naphthas exhibit the same effects of caustic washing: slightly more than a two-fold increase in space velocity with the washed naphtha along with a 50% reduction in hydrogen consumption. Thus, nearly half the hydrogen consumed by raw naphthas is required to saturate phenolic compounds.

- Nitrogen removal was affected by treat gas rate but was insensitive to reactor temperature. This suggests a denitrogenation equilibrium limitation.

- Plugs just before the reactor inlet occurred with both raw and caustic washed naphthas. These plugs were rich in iron (all wt % FeS), the remainder being a coke-like material.

TABLE 5-1

HYDROTREATING OF COAL DERIVED NAPHTHAS

Target: 1 wppm Nitrogen  
 Temperature:  $T = 238 + RV, ^\circ F$

<u>Feed</u>	<u>Pressure, psig</u>	<u>Maximum LHSV, % of RV</u>	<u>Hydrogen Consumption SCF/B</u>
Illinois Raw	800	132	620
Illinois Washed	700	368	300
Wyodak Raw	800	66	650
Wyodak Washed	700	159	300

5.2 Phenolic Coal Naphtha Upgrading

Phenolic compounds constitute approximately 10 wt % of the raw, unhydrotreated naphtha produced by the EDS coal liquefaction process. These compounds must be removed prior to reforming the EDS naphtha to high octane gasoline. Although phenols can be removed by hydrotreating, additional hydrogen uptake and catalyst are required as shown in the previous section. Extracting the phenols prior to hydrotreating, coupled with recovery/upgrading to marketable products, may be economically attractive.

Bench scale extraction studies were conducted in Phase IIIB on Illinois and Wyoming raw coal naphthas using both water and caustic solution as solvent. Data reported in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3) show that caustic treating removes 98+ percent of the phenolic compounds at the theoretical treat rate required to form sodium phenolates, whereas water washing removes only 75 percent even at a high water/naphtha ratio of 10/1. In spite of the poor selectivity using water, economic studies have indicated a \$1-2/B EDS product credit for recovery and sale of phenols versus the base case disposition ultimately to mogas. (See October-December, 1977, EDS Quarterly Technical Progress Report [FE-2893-7]).

Based on studies reported in the literature in which oxygenated solvents (e.g., methanol/water) were found to be very selective for recovering tar acids from coal tar distillates, a scoping program was initiated to see if improved recovery of phenols from raw EDS naphthas was feasible with similar solvents. Potential advantages for a more selective oxygenated solvent would be lower solvent/feed ratios relative to water-only extraction and the elimination of reagents consumption and solids disposal associated with caustic extraction. For scoping studies with EDS naphtha, methanol/water solvent blends were investigated.

Scoping studies were conducted with a heart-cut EDS naphtha derived from Illinois coal. Based on composition data versus boiling range an initial cut point of 275°F was chosen, since naphtha boiling below 275°F was found to be essentially phenol-free. An end-point of 370°F was arbitrarily chosen, since topping CLPP naphtha at this temperature would eliminate any polymer and/or particulates which might have built up during drum storage.

Distillation data obtained on the raw naphtha along with compositions have been reported in the July-September, 1978, EDS Quarterly Technical Progress Report (FE-2893-21). Extraction data with methanol/water solvent blends were also reported; subsequent data are presented in the subsequent quarterly report (FE-2893-29). A grid showing the data base is presented in Table 5-2.

The objectives of these bench equilibrium experiments were to determine distribution coefficients for the phenolic and nonphenolic naphtha components as a function of solvent composition and to determine the capacity of the solvent by reducing the solvent/feed ratios up to the solubility limit.

Equilibrium distribution coefficients for phenol and cresols are shown as a function of solvent composition in Appendix B Figure B-1. Points falling above the equilibrium curve indicate that the amount of solvent present was insufficient to affect an equilibrium distribution of phenol and cresols. It may be concluded from these data that the minimum solvent/feed ratio for batch equilibrium is about 0.5:1 by volume. The selectivity of a given solvent/naphtha system can be determined by comparing the capacity of a solvent to the distribution coefficient of the nonphenolic naphtha fraction. The latter is plotted as a function of the

extract phase water content, Figure B-2 in Appendix B. It is apparent from these two graphs that the capacity of the solvent increases with methanol content, while the selectivity decreases.

TABLE 5-2  
EXPERIMENTAL GRID OF NAPHTHA EXTRACTION EXPERIMENTS

<u>Wt % Methanol in Solvent</u>	<u>0</u>	<u>10</u>	<u>20</u>	<u>30</u>	<u>40</u>	<u>50</u>	<u>60</u>	<u>70</u>	<u>80</u>	<u>90</u>	<u>100</u>
<u>Solvent/Feed Vol. Ratio</u>											
2:1						X	X	X			
1:1				X	X	X					
1:1	X	X	X	X	X	X	X	X	X	X	
0.5:1	X	X	X		X	X	X	X	X		
0.3:1					X	X	X	X			
0.15:1					X	X	X	X			

### 5.3 Wyodak Coal Liquids Characterization Studies

Samples of Wyodak coal liquid products from the Coal Liquefaction Pilot Plant (CLPP) plus 1000°F coker liquids from the Large Stirred Coking Unit (LSCU) were blended in production ratios and fractionated to yield three major liquid product streams: IBP/350°F naphtha, 350/650°F distillate and 650/1000°F VGO. In addition, two Wyodak fuel oils, a 350/1000°F (CLPP products blend) and a 350°F<sup>+</sup> (CLPP products blend plus LSCU coker liquids), were prepared.

Product quality support activities to characterize Wyodak coal liquids were divided into four phases: Phase 1, analytical; Phase 2, storage stability; Phase 3, compatibility studies; and Phase 4, combustion studies. The majority of this work was conducted at ER&E's Products Research Division (PRD) in Linden, New Jersey.

Regarding combustion studies, tests involved primarily the two fuel oil blends and were conducted in the 50 HP Cleaver-Brooks boiler at PRD. Identical samples were sent to the Baton Rouge Laboratories for

hydrotreating studies. The purpose of the hydrotreating studies was two-fold: first, to determine the severity and hydrogen uptake required to reduce the fuel-bound nitrogen content to target levels of 0.5 and 0.2 wt %; and second, to provide product samples with which to assess the degree of improvement with respect to storage stability and compatibility when raw coal liquid fuel oils are upgraded via hydrotreatment.

The blend preparation of the total IBP/1000°F Wyodak coal liquid product along with distillation data and yields of IBP/350°F, 350/650°F and 650/1000°F fractions has been reported in the January-March, 1979, EDS Quarterly Technical Progress Report (FE-2893-29). Included in this same report are the compositions of both Wyodak fuel oil blends according to CLPP and LSCU product components. All blends were prepared using samples available at the time, and particular blends used for characterization studies will be re-examined if they are found to deviate appreciably from the Study Design Update case.

#### Phase 1: Analytical

Analytical inspections were obtained on the three major Wyodak fractions and the two fuel oil blends; these results are summarized in Table B-1 in Appendix B. Also included in this table are inspections of a partially hydrotreated IBP/350°F Wyodak naphtha produced in CLPP when part of the naphtha product was being hydrotreated. The primary difference between this sample and the raw IBP/ 350°F material distilled from the total product blend is the removal of phenolics due to hydrogenation. As expected, nitrogen and sulfur levels are also lower in the hydrotreated sample.

The raw IBP/350°F Wyodak naphtha and the narrow boiling fractions (≤20°F) obtained during distillation were submitted for more detailed analysis. Characterization included low resolution Mass Spectrometry (MS), Fluorescent Indicator Adsorption (FIA) hydrocarbon types, and oxygen by neutron activation analysis. Data have been reported in the January-March, 1979, EDS Quarterly Technical Progress Report (FE-2893-29).

#### Phase 2: Storage Stability

The storage stability test program consisted of monitoring sample product quality at two different storage temperatures, 150°F and 210°F, at intervals of 1, 2, 3 and 12 months (@150°F) and 1 month (@210°F). Viscosity, Conradson carbon and sediment were monitored for the partially hydrotreated Wyodak IBP/350°F and the 350/650°F distillate, along with two fuel oil blends, 350/1000°F and a 350°F<sup>+</sup> (including coker liquids). A reference petroleum regular sulfur fuel oil (RSFO) from Venezuelan crude was included for comparison.



Test results are summarized in Table B-2, except for 12-month stability data at 150°F. Fairly small viscosity increases were observed for all the Wyodak liquids except the 350°F<sup>+</sup> fuel oil. Future work in this program will include a 650/1000°F Wyodak gas oil, a raw IBP/ 350°F naphtha, and samples of Illinois and Wyodak fuel oils hydrotreated at the Exxon Research and Development Laboratories in Baton Rouge (ERDL).

### Phase 3: Compatibility Studies

The compatibility of EDS liquids with two petroleum-derived fuels (RSFO and No. 2 fuel oil) plus coal derived hydrogenated creosote oil was investigated in this program phase. The degree of compatibility, determined by the formation of solids upon mixing, has been measured by an Exxon proprietary test (i.e., sediment by hot filtration) developed for petroleum-derived fuels. The results of this test, presented in Table B-3, indicate that visual observations do not always agree with the measured sediment values. New compatibility tests which are meaningful for coal liquids need to be developed in order to reconcile physical observations with a quantifiable parameter.

### Phase 4: Combustion Studies

A combustion study was completed at Products Research Division (Linden, New Jersey) in a pilot scale industrial boiler (50 HP Cleaver-Brooks, 15 GPH) on raw Wyodak fuel oils. Tests were conducted with 350/1000°F and 350°F<sup>+</sup> fuel oils. Composition by CLPP/LSCU product streams and analytical inspections are summarized in Table 5-3 and in Table B-1, Appendix B, respectively.

TABLE 5-3

#### BLEND OF WYODAK FUEL OILS

<u>Blend Component</u>	<u>Weight</u>	
	<u>350/1000°F</u>	<u>350°F<sup>+</sup></u>
<u>CLPP Products</u>		
Heavy naphtha (nominal 350/400°F)	10.9	8.5
Excess solvent (A-5)	25.5	12.8
Vacuum Gas Oil (A-2)	63.6	33.4
<u>LSCU Products</u>		
Stripper K.O.	---	11.6
Condenser K.O.	---	24.3
Stripper Bottoms	---	9.4
	<u>100.0</u>	<u>100.0</u>

These combustion tests complement similar tests with Illinois fuel oil blends (October-December, 1978, EDS Quarterly Technical Progress Report [FE-2893-25]). Test results summarized below confirm previous results with the Illinois fuels, namely, that smoke and particulate emissions are low, indicative of clean-burning fuels. NO<sub>x</sub> emissions reflect the high fuel-bound nitrogen content.

<u>Test Fuel</u>	<u>350/1000°F</u>	<u>350°F+</u>		
<u>Emission Data</u>				
% CO <sub>2</sub> in flue gas	14.7	14.7		
% O <sub>2</sub> (excess)	2	2		
NO <sub>x</sub> , ppm	400	423		
<u>% N in Fuel</u>	0.48	0.86		
<u>Particulates, Wt %</u>	0.01	0.03		
<u>Effect of Excess Air on NO<sub>x</sub></u> <u>and Bacharach Smoke No.</u>				
	<u>NO<sub>x</sub></u>	<u>Smoke</u>	<u>NO<sub>x</sub></u>	<u>Smoke</u>
% Excess    5	376	2	400	2
10	402	<1	423	1
15	416	<1	442	1
20	433	<1	447	1
25	437	<<1	452	<1

Regarding the 350°F+ Wyodak blend for combustion testing, a 25/75 wt % blend was prepared from the 350/1000°F and 350°F+ blends shown in Table 5-3. Combustion tests were conducted at constant nozzle viscosity to insure similar atomization. Steam tracing of feed is used to adjust nozzle temperatures and therefore set viscosity. The 350°F+ fuel oil, as blended at EPRL, would have exceeded target viscosity even with maximum heat input via steam tracing. Consequently, dilution with the lower viscosity 350/1000°F was used to attain target viscosity.

As with the Illinois blend containing coker liquids, the Wyodak 350°F<sup>+</sup> fuel oil was filtered to <0.1 wt % ash. Since the LSCU is not designed for efficient removal of coker reactor-side fines carryover, the fines content of coker liquids is exceptionally high. Removal by filtration insures that any particulates emitted during combustion due to unburned hydrocarbons can be quantified. As with the Illinois fuel oil tests, particulates emitted agree closely with the ash contents, indicating good atomization and combustion of the hydrocarbon portion of these fuels.

#### 5.4 Hydrotreating Illinois and Wyodak Fuel Oils

Pilot plant hydrotreating studies at the Exxon Research and Development Laboratories (Baton Rouge) with Illinois and Wyodak fuel oils have been completed. Two blends (with and without coker liquids) were prepared for each coal from products produced in the Coal Liquefaction Pilot Plant (CLPP) and the Large Stirred Coking Unit (LSCU). Illinois blends were prepared from CLPP raw solvent and vacuum gas oil products in production proportion (nominally 400/1000°F) and an equivalent blend containing coker liquids (nominally 400°F<sup>+</sup>). Wyodak blends included 350°F/FBP heavy naphtha, derived from topping CLPP naphtha, along with raw solvent and vacuum gas oil products (nominally 350/1000°F). The corresponding blend containing coker liquids is referred to as a 350°F<sup>+</sup> fuel oil.

Depending on the specific end-use applications and future emission regulations, some hydrotreatment of coal-derived fuel oils may be required, especially to reduce NO<sub>x</sub> emissions by lowering the content of fuel-bound nitrogen. The purpose of the hydrotreating studies at the Baton Rouge laboratories was to define for each of these fuel oil blends the hydrogen consumption and severity required to achieve target product nitrogen levels of 0.5 and 0.2 wt %.

A final report is in progress, and a comparison of relative severities to achieve product nitrogen targets will be presented in a subsequent report. Summarized below, however, are the hydrogen consumptions required to reduce nitrogen contents to the targeted levels.

Feedstock	Feed N, wt %	H2 Consumption (SCF/B)	
		@ 0.5 wt % N	0.2 wt % N
Illinois 400/1000°F	0.69	500	1400
Wyodak 350/1000°F	0.43	---	800
Illinois 400°F <sup>+</sup>	0.86	800	2100
Wyodak 350°F <sup>+</sup>	0.87	1400	2400

Operating problems were encountered with the Illinois 400°F<sup>+</sup> fuel oil. Plugging of the reactor inlet occurred repeatedly in spite of a number of unit design changes (guard chamber, etc.), limiting time-on-oil to typically less than two-week intervals. Carbonaceous deposits, rich in iron sulfide, which limited run lengths were presumably due to iron present in the feed which is ultimately converted to the sulfide in the presence of H<sub>2</sub>S.

To test whether contamination with iron can be attributed to CLPP operations or whether it is picked up during prolonged drum storage of CLPP products, fresh samples of CLPP naphtha, solvent and VGO (from then current Wyodak operations) were collected and stored in glass containers at ambient temperature and at 150°F in the presence of iron filings. Initial levels of iron in each fraction were low, and after a two-month storage, no increase in iron content was detected. These tests indicate little, if any, metals contamination during storage.

Samples of the Illinois fuel oils have been fractionated at the Baton Rouge laboratories to assess which boiling ranges are particularly high in iron content. With both fuel oils >95% of the iron is contained in the 1000°F<sup>+</sup> boiling fraction.

#### EPRL Hydrotreating Studies

The primary products from the EDS plants, as currently envisioned, will be naphtha and a 350°F<sup>+</sup> fuel oil. The latter is a blend of heavy naphtha, excess solvent, vacuum gas oil and coker liquids derived from bottoms processing. Depending on coal type, EDS configuration/severity, and environmental regulations, some degree of additional fuel oil upgrading could be required.

Economic convention dictates that the fuel oil components which are the least costly to upgrade be processed first. Progression to the more refractory components follows, and in the extreme case, it may be necessary to upgrade all the fuel oil components (full range fuel oil upgrading).

Product hydrotreating work to date has focused on both extremes. As described in Laboratory Process Research and Development, Section 3.4 of this report, an experimental program was completed involving hydrotreating solvent/naphtha mixtures. The purpose of that study was to assess the impact on solvent hydrotreater performance when heavy naphtha is included with EDS spent solvent feed. This is a relatively inexpensive upgrading step since it requires only incremental capacity in the EDS solvent hydrotreater train.

If, after blending hydrotreated heavy naphtha and excess solvent into the fuel oil pool, additional upgrading would still be required, hydrotreating vacuum gas oil/light coker liquids would be the next least costly step. Pilot plant studies at the Baton Rouge laboratories defined

the severity required to reduce the heteroatom content of Illinois vacuum gas oil to various levels (see January 1976-June 1977 EDS Final Technical Progress Report, Phase IIIA [FE-2853-20]).

More recent studies at Baton Rouge were directed at defining the severity required to upgrade the total fuel oil product. This would be the preferred hydrotreating option if fuel oil product quality targets could not be met via a stepwise approach (i.e., each and every fuel oil component required upgrading).

A pilot plant program was completed at the Baton Rouge laboratories with each of the following feeds: Illinois 400/1000°F and 400°F+ (including coker liquids) and Wyodak 350/1000°F and 350°F+ (including coker liquids). Severities to achieve product nitrogen levels of 0.5 and 0.2 wt % were established. Operating problems were encountered only with the Illinois 400°F+ fuel oil, with repeated upsets caused by plugs rich in iron sulfide.

#### ● Experimental Conditions

In addition to the main hydrotreating program activities at Baton Rouge, very brief scoping runs on selected Illinois 400°F+ and Wyodak 350°F+ fuel oils were carried out at Baytown. The Illinois 400°F+ feed was hydrotreated at a space velocity of 0.66xRV W/H/W, and a nominal temperature of 165°F+RV. Both studies were conducted at an inlet hydrogen partial pressure of about 2250 psig using Ni/Mo-10 catalyst.

#### ● Study Results

As expected, heteroatom removal in the Illinois study was best accomplished with fresh catalyst. During the first yield period, feed nitrogen was reduced from 0.74 wt % to 0.37 wt %, while feed sulfur was reduced from 0.87 wt % to 0.14 wt %. However, as the catalyst aged, heteroatom removal became more difficult at the operating conditions studied. At end-of-run, nitrogen removal was quite low, and sulfur removal had been reduced to 68% of feed concentration. This indicates that operating condition adjustments may be required to achieve acceptable cycle length.

Iron sulfide plugs found in the Baton Rouge Illinois 400°F+ studies were not encountered. However, due to the limited feed supply, only short run durations were possible and hence these observations cannot be compared directly to the Baton Rouge experience with relatively longer run lengths.

Because of the limited number of yield periods obtained with the Wyodak 350°F+ liquids, the effects on catalyst activity of this feed are not well defined. However, nitrogen removal was not achieved to the 0.20 wt % level established at Baton Rouge. Product nitrogen concentrations ranged from 0.27 to 0.66 wt %. On the other hand, an average 73% of feed sulfur was removed over the length of this run. Moreover, an average 45% of feed oxygen was removed during this study.

As in the Illinois study, problems associated with iron sulfide plugging were not encountered in the Wyodak 350°F<sup>+</sup> experiments. But pumping the Wyoming liquids to the reactor required tape heaters on all feed lines. One of these tape heaters failed, and as a result, plugging did occur because of increased liquid viscosity.

- Metals in Coal Liquids

A screening study was conducted to determine the concentration and composition of metals contained in various EDS product streams. Wyodak fractions analyzed included a 350/650°F distillate cut, a vacuum gas oil (800/1000°F) and a 350°F<sup>+</sup> fuel oil blend containing coker liquids. The Illinois samples analyzed were: multi-pass spent solvent (MPSS, 400/800°F), vacuum gas oil (800/1000°F), and two fuel oil blends (400/1000°F and 400°F<sup>+</sup>).

The ash concentration and composition by Inductively Coupled Plasma Emission Spectroscopy (ICPES) were determined for all samples except the Wyodak VGO. The ash levels for the Wyodak and Illinois fuel oils blended for this study were less than 0.1 wt % (blends containing coker liquids were filtered to this level). The most abundant metals in the Illinois fuel oil blends were Fe, Al, Si and Ca, while in the Wyodak case Si, Fe, Al and Na were present in the highest concentrations. The ash level in the Illinois vacuum gas oil was approximately 0.02 wt %, predominantly Fe. The Illinois MPSS sample had a total metals content of 22 ppm, mostly Fe, Si, Na and Al. The Wyodak solvent sample was found to contain about 8 ppm metals, mostly Si, Na and Fe.

## 5.5 Turbine Fuel Combustion Tests

Three drums of hydrotreated multi-pass Illinois solvent were tested by Westinghouse in an EPRI-sponsored program to evaluate the performance of synthetic liquids as stationary turbine fuels. Inspections of the fuel sent to Westinghouse are summarized in Table B-4.

Test results indicated that smoke and NO<sub>x</sub> emissions and combustor wall temperature rise deviated from trend lines established for other test fuels. It was felt that a high nitrogen, hydrogen deficient 650°F<sup>+</sup> fraction may have accounted for these deviations, since the sample had not been fractionated prior to shipment and raw solvent feed to hydrotreating is typically a 700-800°F end-point CLPP product.

Unused test fuel was returned to EPRL for fractionation to yield a 350/650°FVT distillate cut for retesting at Westinghouse. By removing ~13LV% 650°F<sup>+</sup> material, a reduction in nitrogen and an increase in hydrogen content were realized as shown in Table B-4. The 350/650°FVT material was subsequently retested at Westinghouse.

EPRI plan to issue documentation of the turbine test results.

## 5.6 Interim Product Quality Report

A major objective of the 1979 product quality studies is to issue an interim report containing an initial analysis of the preferred EDS products markets considering handling, storage, shipment, health and environment. This report will be updated during the EDS development program, with a final report issued on project completion. The major topics to be covered in this report are as follows:

- I: Market Projections
  - General energy projections along with demand projections for naphtha, distillate and fuel oil
- II: Environmental/Industrial Hygiene Regulations
  - Current and projected/proposed regulations regarding  $\text{NO}_x$ , particulates, PNA's, etc.
- III: EDS Products - Characterization and Composition
  - Yield distribution versus process conditions
  - Format for interpolation/extrapolation
- IV: Product Disposition/Upgrading/Processing Options
  - Tabulation and assessment of data obtained to date affecting direct end-use applications
- V: Product Conversion Options
  - Available data from other DOE-sponsored programs (e.g. UOP, Mobil, etc.)
- VI: Future Test Work Recommendations
- VII: Comparisons with Other Coal Conversion Liquids

## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### BOTTOMS PROCESSING RESEARCH

#### 6. Coking and Gasification Studies

#### 8. Supplemental FLEXICOKING\* Program

##### 6.1 Bottoms Characterization Studies

- Effect of Diluent on Bottoms Viscosity

Results of a study of the effect of dilution on the viscosity and stability of coal liquefaction bottoms were reported in the July-September 1978 EDS Quarterly Technical Progress Report (FE-2893-21). Dilution of the bottoms with 1000°F coal-derived liquids up to 30 wt % reduced the initial viscosity and improved the storage stability of both Wyodak and Illinois bottoms. Data on initial viscosities of Wyodak bottoms was correlated using multiple linear regression with 1000°F content and liquefaction severity as the independent variables.

- Wyodak Bottoms Viscosity

The viscosity and storage stability for two Wyodak bottoms were measured at 600°F. The results were presented in the January-March, 1979, EDS Quarterly Technical Progress Report (FE-2893-29). The initial viscosities for these CLPP Wyodak bottoms, which had low 1000°F contents, averaged 28 poise. After 8 hours storage at 600°F, the average viscosity increased to 40 poise.

- Viscosity of Recycled Bottoms

Viscosity and stability determinations of Illinois coal liquefaction bottoms from the bottoms recycle studies on the Recycle Coal Liquefaction Unit (RCLU-1) were reported in the October-December, 1978, EDS Quarterly Technical Progress Report (FE-2893-25). The recycled bottoms had initial viscosities and thermal stabilities similar to bottoms obtained from single-pass liquefaction.

- Solid Bottoms/Solvent Slurry Study

A study was made of the properties of ground bottoms/solvent slurry feeds to establish the feasibility of such a feed system for the FLEXICOKING Prototype and to identify the important operating constraints. Bottoms mixtures with raw creosote oil formed settling suspensions due to the density difference between the two materials and the low viscosity of the solvent. Fairly high agitation rates and transport velocities would be required to operate a system containing bottoms as solids (see the October-December, 1978, EDS Quarterly Technical Progress Report [FE-2893-25]).

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\*Service Mark



- Bottoms Handling Studies

An experimental program was reported in the October-December, 1978, EDS Quarterly Technical Progress Report (FE-2893-25) concerning the properties of coal liquefaction bottoms when handled and stored as a bulk solid. Results indicated that the friability of solid bottoms was such that dust generation was not excessive. Also, wettability tests indicated that bottoms have little tendency to pick up moisture, so that outside storage of bottoms would be feasible from a wettability standpoint.

## 6.2 Bench Coking Studies

Bench coking results were reported in the January-March, 1979, EDS Quarterly Technical Progress Report (FE-2893-29) for Wyodak bottoms from 1978 CLPP yield periods 264 through 284. The coker liquid yields ranged from 12% to 22% of the bottoms feed.

## 6.3 Coking Yields and Qualities - Continuous Stirred Coking Unit

The Continuous Stirred Coking Unit (CSCU) is used to obtain yield and quality data from the simulated fluidized bed coking of coal liquefaction bottoms. The CSCU uses mechanical agitation to simulate the action of a fluidized bed unit. A typical CSCU run processes 4-6 kilograms of bottoms feed at a rate of about 0.9 kg/hr. Coke is deposited on an initial starting bed of tabular alumina while the vapors are condensed downstream and the gas is metered and analysed. In general, CSCU runs are coal liquefaction bottoms (CLB) resulting from the EDS process operations in CLPP or RCLU.

Detailed data from CSCU runs completed in the last quarter will be presented first (April-June, 1979). Then, the results of the CSCU program covering the last year of operations are summarized.

- Pittsburgh No. 8 Arkwright Bottoms

Pittsburgh No. 8 (Arkwright mine) coal has been liquefied at various conditions in the Recycle Coal Liquefaction Unit (RCLU-1). Bottoms samples from RCLU operations were selected at two different liquefaction conditions: (1) 840°F and 40 minutes residence time and (2) 840°F and 100 minutes residence time. The coking runs were performed at nominal coking residence time, 25% steam/feed ratio, and coking temperatures ranging from base coking temperature to 200°F above base coking temperature. A total of six runs were satisfactorily completed.

Detailed data on each CSCU run is presented in Appendix C. Included are the overall material balances, product liquid distillations, gas compositions, elemental analyses, and elemental balances. A summary of the yields is presented in Table 6-1. In addition to measured yields, yields based on ash-free 1000°F+ bottoms and yields

TABLE 6-1

## SUMMARY OF CSCU YIELDS ON RCLU PITTSBURGH NO. 8 ARKWRIGHT BOTTOMS

• Based on Microlube Distillation Technique

CSCU Run No. RCLU YP Conditions	164 840°F, 40 minutes	165 840°F, 40 minutes	174 840°F, 40 minutes	166 840°F, 100 minutes	175 840°F, 100 minutes	178 840°F, 100 minutes
Bottoms Ash Content, Wt %	10.46	9.68	9.56	12.55	11.24	11.27
Bottoms 1000°F- Content, Wt % Microlube	6.6	6.6	5.1	8.7	6.2	6.2
Coking Temperature, °F-RV	141	92	159	64	167	100
Coking Steam/Feed Ratio, %	29	24	23	18	18	16
Coking Residence Time, seconds/RV	2.28	2.12	2.62	3.3	2.7	2.6
CSCU Run Material Balance, %	103	100	102	100	99	100
Measured Coking Yields, Wt % on Feed						
Coke	60.1	61.8	64.4	67.1	67.9	70.0
Liquid	35.2	33.8	29.6	28.8	27.4	26.7
Gas	4.7	4.4	6.0	4.1	4.7	3.3
Coking Yields, Wt % on Ash Free 1000°F+ Bottoms						
Coke	60.0	62.2	64.3	69.3	68.6	71.2
Liquid	34.4	32.5	28.7	25.5	25.7	24.8
Gas	5.6	5.3	7.0	5.2	5.7	4.0
Coking Yields, Wt % on 1000°F+ Bottoms						
Coke	64.4	66.2	67.9	73.5	72.4	74.6
Liquid	30.6	29.1	25.8	22.0	22.6	21.9
Gas	5.0	4.7	6.3	4.5	5.0	3.5

based on 1000°F<sup>+</sup> bottoms are also presented in Table 6-1. Ash-free 1000°F<sup>+</sup> bottoms yields are useful in comparing the effect of coking on bottoms with different ash and 1000°F<sup>-</sup> contents or on bottoms from different types of coal. The 1000°F<sup>+</sup> bottoms yields are useful in relating coking yields to total liquefaction yields on coal.

The ash-free 1000°F<sup>+</sup> liquid yields from Pittsburgh Arkwright bottoms are plotted in Figure 6-1. The liquid yields from 840°F-40 minute bottoms show a characteristic dependence on coking temperature. Previous experience has shown that a maximum in coker liquid yield occurs around RV+70°F coking temperature. This behavior is also a function of liquefaction conditions, most notably residence time. For more highly converted bottoms (e.g., 840°F-100 minute bottoms), the yield structure shows less variation as a function of coking temperature. The same flat yield structure is also suspected for bottoms from low-conversion conditions. For comparison, the yield structure of 840°F-40 minute Illinois No. 6 Monterey bottoms is also depicted in Figure 6-1. The yield structures are similar for both the Illinois #6 and Pittsburgh No. 8 Arkwright.

In Table 6-2 information about the effect of coking on 1000°F<sup>-</sup> liquids is examined. The microlube data indicate that no 1000°F<sup>-</sup> liquid is being produced, while the HiVac-C data indicate that 1000°F<sup>-</sup> liquid is being produced 5 to 9% on feed. This trend was also observed for Illinois No. 6 Burning Star bottoms. Coking yields from Pittsburgh Arkwright bottoms will be compared to those from other coal liquefaction bottoms in the summary section.

#### + Wandoan Bottoms

Three CSCU runs were completed on bottoms from the liquefaction of Wandoan coal at 840°F and 40 minutes residence time. The detailed CSCU data are presented in Appendix C and summarized in Table 6-3. These bottoms had a particularly high ash content (36%); however, this did not appear to affect either processability or yields. The liquid yield structure corrected for ash and microlube 1000°F<sup>-</sup> liquid is depicted in Figure 6-2. The other data points give an appearance of a yield structure similar to that seen for other 840°F, 40 minute bottoms.

A comparison of the product liquid distillation results for Wandoan coal is given in Table 6-4. Both Microlube and HiVac-C methods show net production of 1000°F<sup>-</sup> material. Again, 1000°F<sup>-</sup> liquid production is seen to be a function of coking temperature, with higher net 1000°F<sup>-</sup> liquid recovered from coking between RV+65°F and RV+199°F.

TABLE 6-2

COMPARISON OF FEED 1000°F<sup>-</sup> CONTENT WITH CALCULATED 1000°F<sup>-</sup> LIQUID RECOVERED

## ● Pittsburgh Arkwright Bottoms

CSCU Run No.	165	164	174	166	178	175
Liquefaction Conditions	840°F, 40 minutes			840°F, 100 minutes		
Coking Temperature, °F-RV	92	141	159	64	100	167
<u>Microcube Distillation</u>						
Feed 1000°F <sup>-</sup> , Wt %	6.6	6.6	5.1	8.7	6.2	6.2
Product 1000°F <sup>-</sup> , Wt % on Feed	7.4	7.1	7.1	4.6	6.3	5.5
<u>HiVac-C Distillation</u>						
Feed 1000°F <sup>-</sup> , Wt %	9.9	9.9	11.9	12.4	14.6	14.6
Product 1000°F <sup>-</sup> , Wt % on Feed	19.1	19.5	19.4	20.6	19.4	20.0
Net 1000°F <sup>-</sup> , Wt %	9.2	9.6	7.5	8.2	4.8	5.4

TABLE 6-3

SUMMARY OF CSCU YIELDS ON RCLU WANDOAN BOTTOMS

- Simulated Fluidized Coking
- Microlube Distillation Basis

CSCU Run No.	170	172	189
RCLU YP Conditions	840°F, 40 minutes		
Bottoms Ash Content, Wt %	38.03	36.66	36.59
Bottoms 1000°F <sup>-</sup> Content, Wt % Microlube	4.2	4.2	7.7
Coking Temperature, °F-RV	65	199	175
Coking Steam/Feed Ratio, %	55	19	23
Coking Residence Time, Seconds/RV	2.08	3.8	3.3
CSCU Run Material Balance, %	100	96	95
Measured Coking Yields. Wt % on Feed			
Coke	76.5	76.9	70.1
Liquid	20.6	18.0	27.1
Gas	2.9	5.1	2.8
Coking Yields. Wt % on Ash Free			
1000°F <sup>+</sup> Bottoms			
Coke	66.6	68.0	60.2
Liquid	28.4	23.3	34.8
Gas	5.0	8.6	5.0
Coking Yields, Wt % on 1000°F <sup>+</sup> Bottoms			
Coke	79.9	80.3	75.9
Liquid	17.1	14.4	21.0
Gas	3.0	5.3	3.1

FIGURE 6-1

NET LIQUID YIELDS FROM COKING PITTSBURGH #8  
ARKWRIGHT BOTTOMS

- NOMINAL COKING STEAM RATE AND RESIDENCE TIME
- LIQUEFACTION CONDITIONS:
  - △ - 840°F, 100 Minutes
  - - 840°F, 40 Minutes

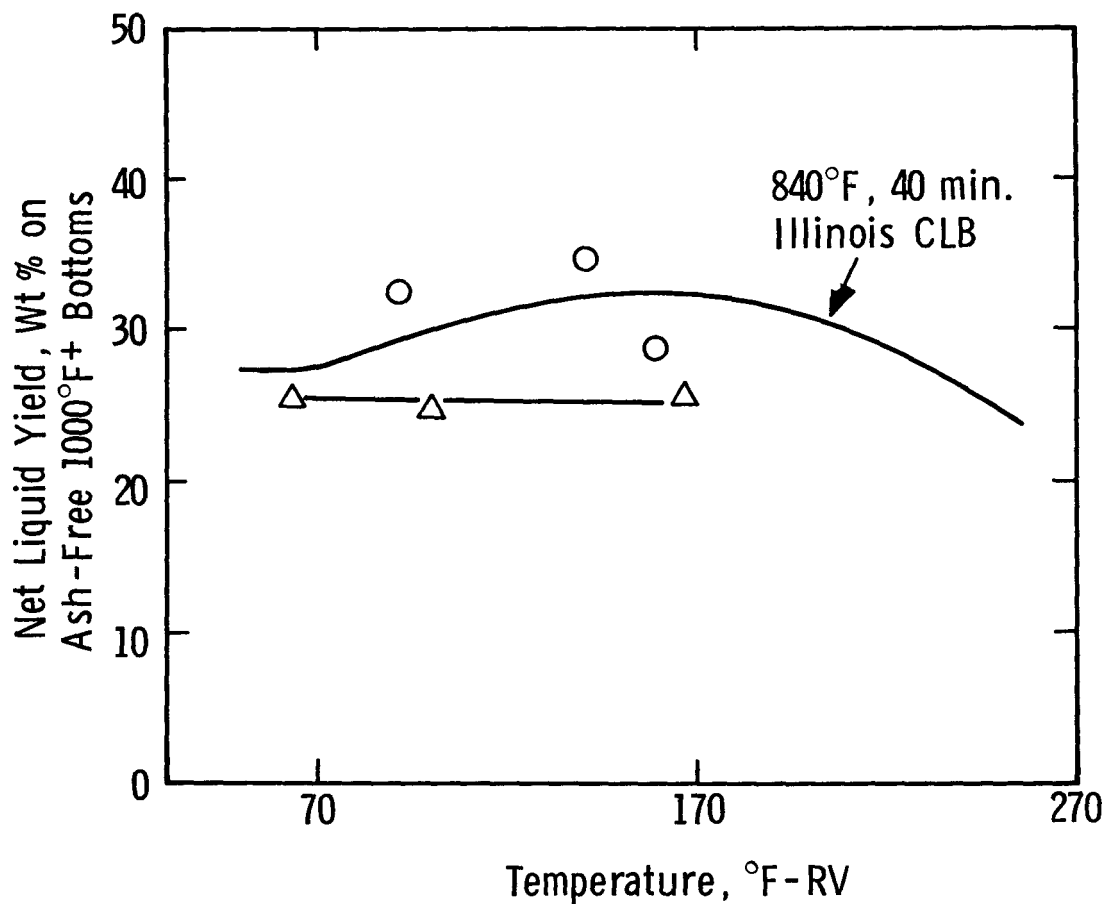


FIGURE 6-2

NET LIQUID YIELD FROM COKING WANDOAN COAL  
LIQUEFACTION BOTTOMS

- BOTTOMS PRODUCED AT 840°F, 40 MINUTES
- MICROLUBE DISTILLATION BASIS

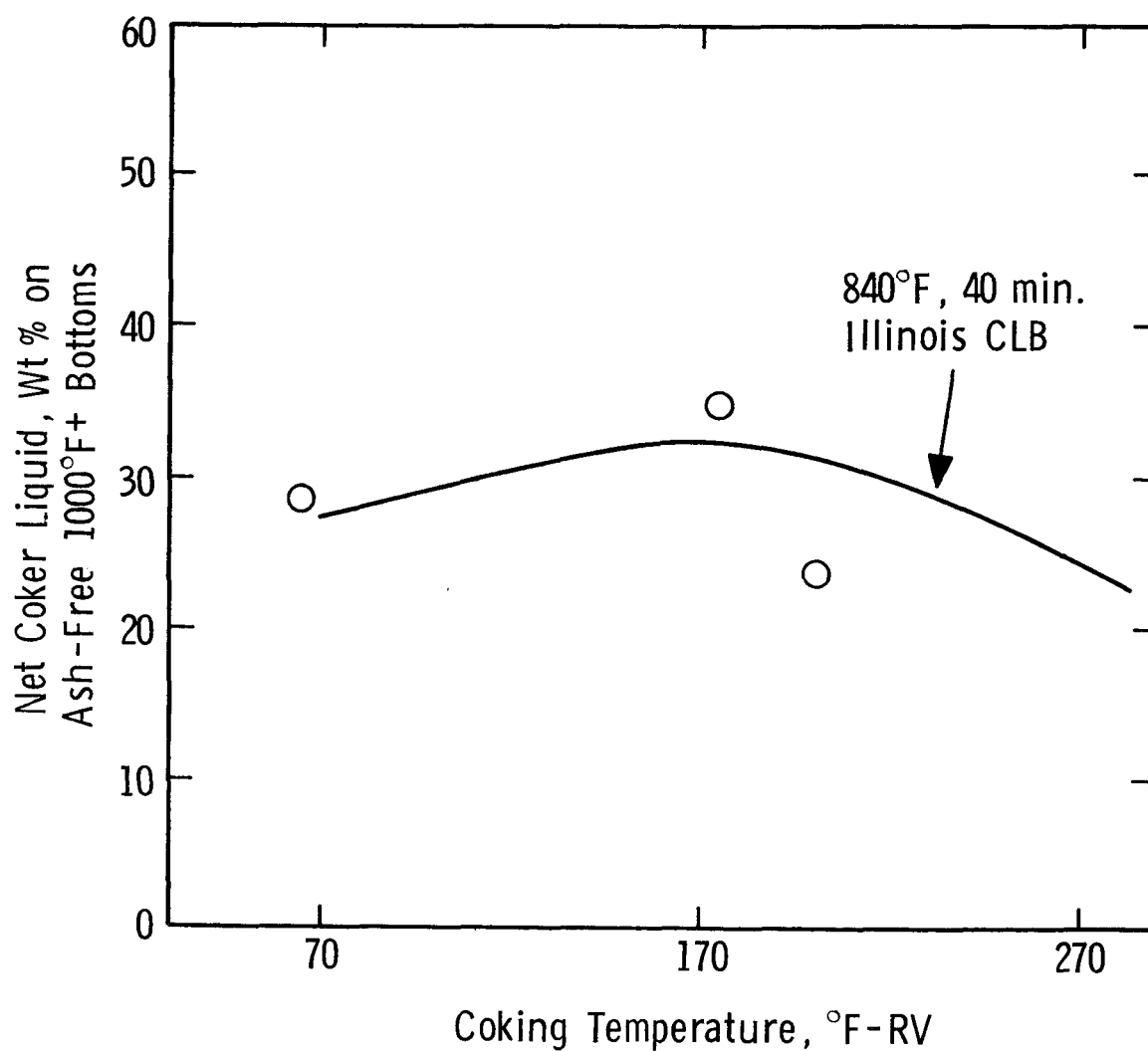


TABLE 6-4

COMPARISON OF FEED 1000°F- CONTENT WITH  
CALCULATED 1000°F- LIQUID RECOVERED

- Wandoan coal liquefaction bottoms feed
- Simulated fluidized coking processing (CSCU)

CSCU Run No.	170	171	189	172
Coking Temperature, °F-RV	65	153	175	199
<u>Microtube Distillation</u>				
Product 1000°F-, wt % on feed	6.8	12.3	10.2	5.8
Feed 1000°F-, wt %	4.2	5.9	7.7	4.2
NET	2.6	6.4	2.5	1.6
<u>HiVac-C Distillation</u>				
Product 1000°F-, wt % on feed	15.7	26.2	18.6	10.8
Feed 1000°F-, wt %	9.4	9.0	11.4	11.4
NET	6.3	17.2	7.2	-0.6

The estimated contribution of coker liquids to the overall Wandoan EDS yields is given in Table 6-5. Coking is estimated to contribute 9.2 wt % liquids on coal to the overall EDS process yield which is one-fourth of the total.

TABLE 6-5

ESTIMATED CONTRIBUTION OF COKER LIQUIDS  
TO EDS PROCESSING OF WANDOAN COAL

- Base Coking Temperature, 70°F+RV
- 840°F-40 Minute Liquefaction Conditions

<u>Liquefaction Yields</u>	
C <sub>4</sub> -1000°F, wt % on dry coal	27.9
1000°F+ Bottoms, wt % on dry coal	54.1
<u>Coker Liquid Yield</u>	
Wt % on 1000°F+ Bottoms	17.1
Wt % on Dry Coal	9.2
<u>Total Liquefaction Yield</u>	
Wt % on Dry Coal	37.1



#### + Coker Liquid Properties

Coker liquids from coking Illinois No. 6 Burning Star bottoms were further characterized by benzene and pyridine extractions. The Burning Star coker liquids were generated in a CSU screening study reported in the January-March, 1979, EDS Quarterly Technical Progress Report (FE-2893-29). This extraction study was undertaken to investigate the nature of the recovered liquids as a function of coking temperature. The results are summarized in Table 6-6. The distillation basis for the study was HiVac-C distillation because it was found that the HiVac-C was the most efficient in removing benzene solubles from the bottoms. In order to compare the coker liquid properties with the feed properties, the former are tabulated on a wt % on feed basis. The entries are arranged according to coking temperature.

As previously reported, the highest coker liquid yields occurred below RV+150°F for 840°F-40 minute bottoms. In Table 6-6, for the 40 minute bottoms, the product liquid 1000°F<sup>-</sup> asphaltenes for coking below RV+100°F are higher than those from coking at higher temperatures. The product liquid 1000°F<sup>+</sup> asphaltenes decrease as the temperature increases. Taking into account the 1000°F<sup>-</sup> asphaltenes that come in with the feed, an increase in the net 1000°F<sup>-</sup> asphaltenes can be seen up to RV+150°F and then a definite decrease. This trend can be observed in both the 40 and 100 minute bottoms. This suggests that above RV+150°F that cracking of the 1000°F<sup>-</sup> asphaltenes takes place.

Lastly, a summary of Burning Star coker liquid yields and properties is presented in Table 6-7.

TABLE 6-6

CHARACTERIZATION OF BURNING STAR COKER LIQUIDS BY BENZENE-PYRIDINE EXTRACTIONS

## ● HiVac-C Distillation Basis

<u>LIQUEFACTION CONDITIONS</u>	<u>880°F, 25 Minutes</u>			<u>840°F, 40 Minutes</u>			
CSCU Run, YT	151	156	144	149	148	157	146
Coking Temperature, °F-RV	91	136	165	60	95	149	156
<u>Coker Liquid Properties</u>							
Wt % on Feed							
Inerts	0.2	0.9	0.2	0.4	1.4	0.8	3.2
(Pyridine Insolubles)							
Pre-Asphaltenes, 1000°F <sup>+</sup>	1.8	1.9	2.0	3.1	4.3	5.5	6.8
(Pyridine Soluble-Benzene Insoluble)							
Asphaltenes							
(Benzene Solubles)							
1000°F <sup>+</sup>	4.1	4.1	4.8	5.1	4.1	3.8	1.2
1000°F <sup>-</sup>	19.0	20.7	15.9	27.3	27.4	20.4	21.3
<u>Feed Properties, Wt % on Feed</u>							
Asphaltenes (All HV 1000°F <sup>-</sup> material)	15.5	15.7	16.0	24.7	21.0	25.6	26.4
Pre-Asphaltenes	50.0	54.0	58.1	52.6	59.6	51.3	50.0
<u>Net Asphaltenes</u>							
1000°F <sup>+</sup>	4.1	4.1	4.8	5.1	4.1	3.8	1.2
1000°F <sup>-</sup>	3.5	4.9	-0.1	2.6	6.4	-5.2	-5.1
Total	7.6	9.0	4.7	7.7	10.5	-1.4	-3.9

TABLE 6-7

BURNING STAR COAL LIQUEFACTION BOTTOMS COKER LIQUID PRODUCT YIELDS

- Base coking conditions
- CSCU results on RCLU bottoms (840°-40 Minutes)
- Average of two runs

<u>Product</u>	<u>Wt % on 1000°F+ Bottoms</u>	<u>Wt % on Ash-Free 1000°F Bottoms</u>
Gas		
H <sub>2</sub>	0.4	0.5
CO, CO <sub>2</sub> , H <sub>2</sub> S, NH <sub>3</sub>	0.9	1.1
C <sub>1</sub> -C <sub>3</sub>	1.7	2.1
Liquid, C <sub>4</sub> -1000°F+	30.7	36.9
Coke	66.3	59.4

BURNING STAR COAL LIQUEFACTION BOTTOMS COKER LIQUID  
PRODUCT INSPECTIONS

- Base coking conditions

	<u>400-700°F</u>	<u>700-1000°F</u>	<u>1000°F+</u>	<u>Total Product</u>
Sp. Gr.	1.04	1.19	1.23	1.24
Con. Carbon, Wt %	0.2	12.6	44.7	35.4
BTU/lb	16300		15946	--
lb Sulfur/MBTU	0.3		0.6	--
Viscosity, cp	--	--	1.5 @ 400°F	
	--	--	0.5 @ 450°F	13 @ 270°F
Elemental Analysis, Wt %				
C	87.9	88.7	87.9	87.5
H	7.8	6.6	5.9	6.2
S	0.4	0.6	0.8	.7
N	0.7	0.9	1.5	1.3
Atomic H/C	1.10	.89	0.80	0.85

- Annual Summary of CSCU Operations

- + Coal Liquefaction Bottoms Screening Studies

During this annual reporting period, coking yield screening studies were performed on the following coal liquefaction bottoms:

<u>Coal</u>	<u>Liquefaction Conditions</u>
Big Brown (Texas Lignite)	840°F, 40 minutes
Illinois No. 6 (Burning Star)	880°F, 25 minutes 840°F, 40 minutes
Pittsburgh No. 8 (Arkwright)	840°F, 40 minutes 840°F, 100 minutes
Wandoan (Australian black coal)	840°F, 40 minutes

A comparison of the coking yields from all types of bottoms processed is given in Table 6-8. With the exception of the Pittsburgh Ireland bottoms, the coking yields are from bottoms produced at 840°F and 40 minutes residence time. The Pittsburgh Ireland yields listed in Table 6-8 were from 840°F-100 minute bottoms. Estimating from Pittsburgh Arkwright bottoms, Table 6-1. 840°F-40 minute Pittsburgh Ireland bottoms can be expected to give a liquid yield of about 33 wt % on ash-free 1000°F+ bottoms. Liquid yields can be seen to increase with coal rank, while gas yields decrease with coal rank. Evidence of coking yield dependence on liquefaction conditions has been observed for all types of coal liquefaction bottoms processed in CSCU. Coker liquid yield depends primarily on liquefaction residence time and temperature (thus conversion) and secondarily on coking temperature at nominal coking residence time and steam rates. Specific details on this effect can be found in the technical progress report in which the results of the particular screening study were presented. A summary of the contribution of coker liquids to the overall EDS process yields is given in Table 6-9. Care should be exercised in interpreting the information in Table 6-9. The yields are from various screening studies at 840°F and 40 minutes liquefaction residence time. For most of the coals listed, the optimum liquefaction C<sub>4</sub>-1000°F liquid yields occur at conditions different from 840°F and 40 minutes. Greater liquefaction conversion necessarily means lower 1000°F+ bottoms yield and often coker liquid yield also decreases for the bottoms from longer liquefaction residence time. However, total yield often stays the same. A good example of this can be seen in Table 6-10 for Pittsburgh Arkwright coal. Although the 40 minute liquefaction residence time gave a lower C<sub>4</sub>-1000°F liquid yield than 100 minute condition, the liquids not fully converted into the C<sub>4</sub>-1000°F range at 40 minutes were recovered as coker liquids. The total yield from both 40 and 100 minute operations is nearly the same. The difference is in the quality and distribution of liquids. The information in Table 6-9 indicates that at 840 F and 40 minutes the overall liquefaction yields increase with increase in coal rank from lignites to bituminous.

TABLE 6-8

COMPARISON OF COKING YIELDS FROM LIQUEFACTION BOTTOMS FROM DIFFERENT COALS

- Base case coking conditions
- Bottoms feed produced under similar liquefaction conditions of 840°F, 40 minutes residence time except Pittsburgh Ireland bottoms produced at 100 minutes residence time
- Yields expressed as weight percent on ash-free 1000°F<sup>+</sup> feed basis

	<u>Big Brown Lignite</u>	<u>Wyoming Subbituminous</u>	<u>Illinois #6 Monterey Bituminous</u>	<u>Wandoan Australian Black Coal</u>	<u>Pittsburgh Ireland Bituminous</u>	<u>Pittsburgh #8 Arkwright Bituminous</u>	<u>Illinois #6 Burning Star #2 Bituminous</u>
Coke	68.3	71.8	67.8	66.6	69.0	62.2	59.9
Liquid	24.0	23.2	26.7	28.4	27.3	32.5	37.1
Gas	7.7	5.0	5.5	5.0	3.7	5.3	3.0

TABLE 6-9

COMPARISON OF LIQUEFACTION YIELDS FROM DIFFERENT RANK COALS

- Conditions: 840°F, 40 minutes residence time, 1500 psig, 1.6 S/C, 4% H<sub>2</sub> and base coking conditions
- Liquefaction conditions not optimum for maximum C<sub>4</sub>-1000 yields in some cases

	Big Brown Texas Lignite	Wyoming Subbituminous	Wandoan Australian Black	Pittsburgh Ireland Bituminous	Pittsburgh Arkwright Bituminous	Illinois #6 Monterey Bituminous	Illinois #6 Burning Star Bituminous
Liquefaction C <sub>4</sub> -1000°F Liquid, wt % on dry coal	27.9	29.0	27.9	28.6	22.6	35.3	24.1
Liquefaction 1000°F <sup>+</sup> Bottoms, wt % on dry coal	48.3	45.5	54.1	58.1	67.3	47.0	60.1
Coker Liquid, wt % on 1000°F <sup>+</sup> bottoms	16.8	19.3	17.1	21.0*	29.1	21.0	30.9
Coker Liquid, wt % on dry coal	8.1	8.8	9.2	12.4	19.6	10.0	18.6
Total Liquefaction Yields C <sub>4</sub> -1000°F <sup>+</sup> ,	36.0	37.8	37.1	41.0	42.2	45.3	42.7

\*Estimated from coking data on 100 minute residence time bottoms.

TABLE 6-10

COMPARISON OF LIQUEFACTION YIELDS FROM PITTSBURGH ARKWRIGHT COALLiquefaction Conditions

Temperature, °F	840	840
Residence Time, minutes	40	100
<u>Yields*</u>		
Liquefaction C <sub>4</sub> -1000°F Wt % on Dry Coal	22.6	29.9
Liquefaction 1000°F+ Bottoms Wt % on Dry Coal	67.3	52.6
Coker Liquid Wt % on 1000°F+ Bottoms	29.1	22.2
Coker Liquid Wt % on Dry Coal	19.6	11.7
Total Liquefaction Yield Wt % on Dry Coal	42.2	41.6

---

\*Yields from 2/78 RCLU-1 operations

+ Special Coking Studies

Two CSCU runs were performed to simulate recycle coking of 1000°F<sup>+</sup> Wyodak coker liquid. Wyodak stripper bottoms product from the Large Stirred Coking Unit (LSCU) were distilled in a large scale HiVac-C distillation column to an equivalent 837°F endpoint. Bottoms from this distillation were used as feed for CSCU. The results of these two runs showed that no additional 1000°F<sup>-</sup> liquids could be produced by recycle coking.

To test the effect of feed diluent on coking yield, a CSCU run, YT 132, at base coking conditions was performed with a mixture of 70% CLPP Illinois bottoms with 30% Vacuum Gas Oil (VGO). CLPP VGO is a nominal 700-1000°F vacuum tower cut. Both the bottoms and the VGO were from CLPP yield period 263 at 840°F and 40 minutes residence time. The CSCU conditions were similar to those expected in the FLEXICOKING process prototype. The yield from this run is compared in Table 6-11 to 70% of the normal yield expected from CLPP Illinois bottoms plus complete recovery of the 30% VGO. The VGO is assumed to be all 1000°F<sup>-</sup> material (HiVac-C basis). The agreement between the actual and calculated yields is very good. The total liquid yields closely agree and also the split between 1000°F<sup>+</sup> and 1000°F<sup>-</sup> material agrees well. These results indicate that, in mixing VGO into bottoms to achieve lower viscosities for prototype operations, the VGO plus the normal coker liquids can be recovered from the coker.

TABLE 6-11

COMPARISON OF ACTUAL AND PREDICTED COKING YIELDS FROM  
70% BOTTOMS PLUS 30% VACUUM GAS OIL

- Illinois Coal
- Base coking temperature
- HiVac-C Distillation Basis
- Assumes all VGO HiVac-C Recoverable

	<u>Actual CSCU Yield</u>	<u>Predicted Yield</u>
	YT 132 Wt % on Feed	70% YT 122 Yield + 30% VGO Wt %
Coke	46.6	47.8
1000°F <sup>+</sup>	10.8	11.2
1000°F <sup>-</sup>	37.9	38.2
Gas	4.7	2.8
Total Liquid	48.7	49.4



In liquefaction bottoms recycle experiments, Illinois bottoms from 840°F and 40 minute operations in CLPP were mixed with Illinois coal and liquefied in RCLU at 840°F and 40 minutes residence time. The bottoms from this recycle operation, first pass bottoms recycle bottoms, were again mixed with Illinois coal and recycled through RCLU for a second pass bottoms recycle operation. Both first and second pass recycle bottoms have been coked in CSCU at base conditions. The results are compared with the coking yields from regular CLPP Illinois bottoms in Table 6-12. At base coking conditions, the net yields from all three types of bottoms are nearly the same. Previous CSCU yield data on Illinois bottoms has shown that net coker liquid yield at base coking temperature does not vary much for all types of Illinois bottoms. At 100°F above base coking temperature, coker liquid yield has been shown to depend on liquefaction residence time. Therefore, a more sensitive indicator of the effect of bottoms recycle in liquefaction would be to coke the bottoms at 100°F above base temperature. So far, the results indicate that on a bottoms feed basis no coker liquid yield debit is incurred by recycling Illinois bottoms to liquefaction.

A total of seven CSCU runs were performed to investigate the yields from the coking of Illinois coal with Illinois coal liquefaction bottoms. Three CSCU runs were completed with coal alone, two runs were completed with 15% coal in bottoms, and two runs were completed with equal parts coal, bottoms and vacuum gas oil. All runs were at base coking conditions. The three runs with coal alone used the CSCU dry coal feeder system and had poor material balances (92-96%). However, the yields did agree well with Fischer Assay at the same temperature. To simulate coking of a 1:1 coal:bottoms mixture, equal parts vacuum gas oil, bottoms and coal were fed into the CSCU reactor. The vacuum gas oil (VGO) served as a viscosity lowering diluent. The coal:bottoms yield was corrected for the VGO. The results are summarized in Figure 6-3. A slight synergistic effect in liquid yield can be seen in the 0-20% coal in bottoms range.

#### Future Plans

The CSCU operations for the next quarter include coking yield screening studies on RCLU bottoms from Wandoan and Pittsburgh No. 8 (Ireland) coals. Bottoms from a variety of liquefaction conditions are to be studied to elucidate coker yield dependency on upstream liquefaction processing. These studies will complete the CSCU operations on coal liquefaction bottoms from the sponsors' coals.

#### 6.4 Large Stirred Coking Unit (LSCU)

The Large Stirred Coking Unit (LSCU) was revamped and started up during this reporting period. The basic configuration was maintained unchanged. A simplified flow diagram of the LSCU appeared in the January-June, 1976, EDS Quarterly Technical Progress Report (FE-2353-2).

TABLE 6-12

COMPARISON OF COKING YIELDS FROM BOTTOMS RECYCLE BOTTOMS

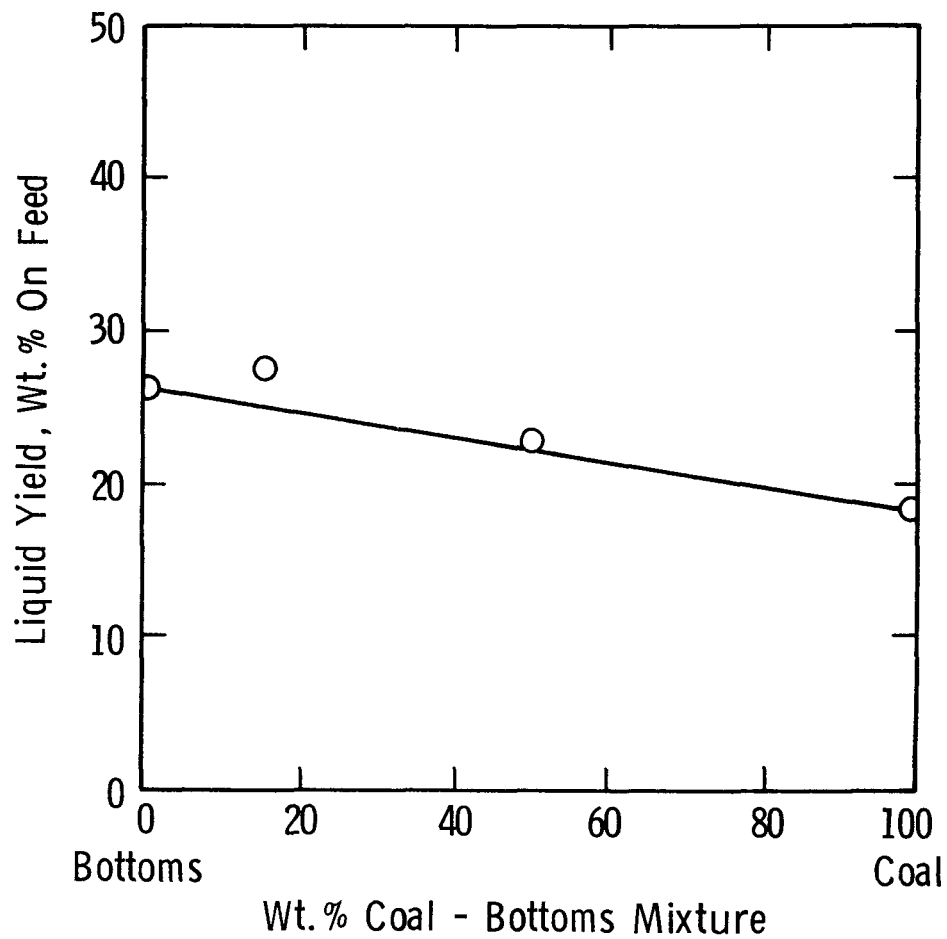
- Base coking conditions in CSCU
- 840°F and 40 minutes residence time for each liquefaction pass
- Illinois coal

<u>CSCU</u> <u>Run</u>	<u>Liquefaction</u> <u>Operation</u>	<u>Bottoms</u> <u>YP</u>	Net Yields Wt % on Ash Free 1000°F+ Bottoms			Net Yields Wt % on 1000°F+ Bottoms		
			<u>Coke</u>	<u>Liquid</u>	<u>Gas</u>	<u>Coke</u>	<u>Liquid</u>	<u>Gas</u>
122	Regular	263	68.5	25.8	5.6	75.3	20.3	4.4
129	1st Pass Recycle	561	69.6	25.7	4.7	77.3	17.2	3.5
136	2nd Pass Recycle	567	67.8	26.8	5.4	75.8	20.2	4.0

FIGURE 6-3

COKING YIELDS FROM BOTTOMS - COAL MIXTURES

- BASE COKING CONDITIONS
- AVERAGE VALUES
- FEED SOURCE - CLPP ILLINOIS BOTTOM, YP 263,  
AND ILLINOIS COAL



The objective of the current LSCU operations is to produce coker liquid samples for various program studies, such as hydroclone tests with heavy coker liquids and combustion tests with a full-range 350°F+ CLPP/LSCU production ratio blend. Both Illinois bottoms and Wyodak bottoms from CLPP will be processed to fulfill the sample needs.

Continuous operations have been achieved. Runs were usually terminated due to plugging in the product stripper inlet zone. Several mechanical modifications are being made to minimize this plugging problem. The service factor for LSCU was between 40 to 50% during this reporting period.

#### 6.5 Bottoms Remelt Study

A short-term bottoms remelt project was initiated during the last quarter of 1978. The objective of the study was to experimentally measure physical property data for bottoms blends in order to confirm mathematical models for mixing and pumping solutions of bottoms and heavy hydrogenated creosote diluent. The data collection phase of this project was completed during January, 1979, and analyses of the data were conducted throughout the remainder of the first quarter of 1979. Results from this project will be utilized to provide tighter specification of the feed preparation system for the 70 T/D FLEXICOKING prototype unit.

The variables measured fall within the headings of flow properties and mixing properties. The flow properties being determined were:

- Viscosity
- Pressure drop
- Heat transfer coefficient for counter-current heat exchanger

while the mixing properties being evaluated for the agitated melt tank were:

- Approach to perfect mixing
- Steady state stirring torque
- Transient stirring torque
- Heat transfer coefficient for the melt tank

Independent variables in the study were 1000°F- content of the blends, temperature, stir rate and pumping rate. A preliminary study was also conducted on a 1.2 wt % Natrosol solution, a synthetic analog of bottoms blends.

A schematic flow diagram of the experimental system was presented in the October-December, 1978, EDS Quarterly Technical Progress Report (FE-2893-25). The Large Stirred Coking Unit (LSCU) was modified to conduct these experiments. The fully baffled melt tank was fitted with a four-bladed, pitched turbine that was driven by a variable-speed DC

motor and provided downflow agitation of the process blends. Mixing was qualitatively evaluated using an isotopic tracer injection system. All mixing properties were measured for the melt tank and flow properties were measured within the recycle circulation loop outside the melt tank. The recycle loop consisted of two pressure drop lines of different diameters, an on-line viscometer capable of varying shear rate by two orders of magnitude and a counter-current heat exchanger. Bypasses around the viscometer and heat exchanger were provided.

Determinations of qualitative mixing behavior, stirring torques and flow heat transfer coefficients were obtained at EPRL and more detailed analyses were conducted by Exxon Engineering Technology Department (EETD). In general, mixing behavior was about as expected. Mixing power consumption was low with quick asymptotic transient build-up to steady-state torque and no evidence of torque or pressure impulse on startup. Estimates of heat transfer coefficients are higher than anticipated. Computational analyses being conducted by EETD have been completed during the second quarter of 1979.

#### 6.6 Coke Gasification Kinetics

The air FLEXICOKING process is under development in the EDS program for processing liquefaction bottoms to recover additional liquids and to provide gaseous fuel. Steam and air are fed to the FLEXICOKING gasifier to burn coke for heat and to produce fuel gas. Rate data on coke gasification is obtained from a differential type fixed-bed minigasifier and from an integral type fluidized-bed gasifier in the Integrated Coking/Gasification Pilot Plant (IKG). The minigasifier is by far the more flexible of the two and is used to study the relative reaction rates of EDS cokes with steam and/or carbon dioxide as influenced by gas partial pressures, temperature, velocity and coke particle characteristics. Relative rate information from the minigasifier will be combined with IKG rate data for EDS FLEXICOKING gasifier design.

The minigasifier program this year has included a change of technique to the acquisition of initial rate data on gasifier cokes from previous relative ranking of coke gasification activity. Some time was spent on mechanical changes to the minigasifier to eliminate radial temperature gradients and to increase the gas chromatograph sampling frequency for better rate data. Kinetics data were obtained for steam and carbon dioxide gasification of Illinois (Monterey mine) bottoms derived IKG gasifier coke. The analysis of this study coke is given in Appendix D Table D-1. Initial rate data on gasifier coke is studied to determine the responses of practical cokes with known properties, such as surface area, which are produced in integrated operation.

Preliminary data analysis shows steam and carbon dioxide gasification of IKG Illinois gasifier coke may be correlated by Langmuir-type rate expressions. However, in the range of design interest, simple linear expressions can be used for screening work to extrapolate reactant rate

effects from IKG conditions to design. Thermal response of the rate data agrees fairly well with literature values. There are indications that Illinois mineral matter gasification catalysis declines with reduction in temperature. At the temperature of commercial interest, about RV-55°F, mass transfer effects are important. So far, the data are available only for the slower carbon dioxide gasification. External mass transfer effects are definitely present as demonstrated by rate response to velocity. This information together with particle size effects on rate suggest that internal particle mass transfer is also a factor. It will be important to include both the external and internal mass transfer effects into design equations. For instance, the internal effects change the rate response in extrapolating from large particle IKG conditions to relatively small coke particle commercial conditions.

• Gasification Rates Mildly Inhibited by Reactants

It is well known that carbon gasification rates are inhibited by reactants and products. The plot of steam and carbon dioxide gasification of EDS Illinois coke in Figure 6-4 shows this inhibition effect by reactants. Carbon dioxide inhibition is somewhat stronger than the inhibition of steam, as evidenced by the greater change of the rate with pressure. This type of inhibition may be correlated by Langmuir-type rate expressions of the type:

$$r = \frac{R(P_r - \frac{P_p}{K'})}{1 + K_r P_r + K_p P_p}$$

where,

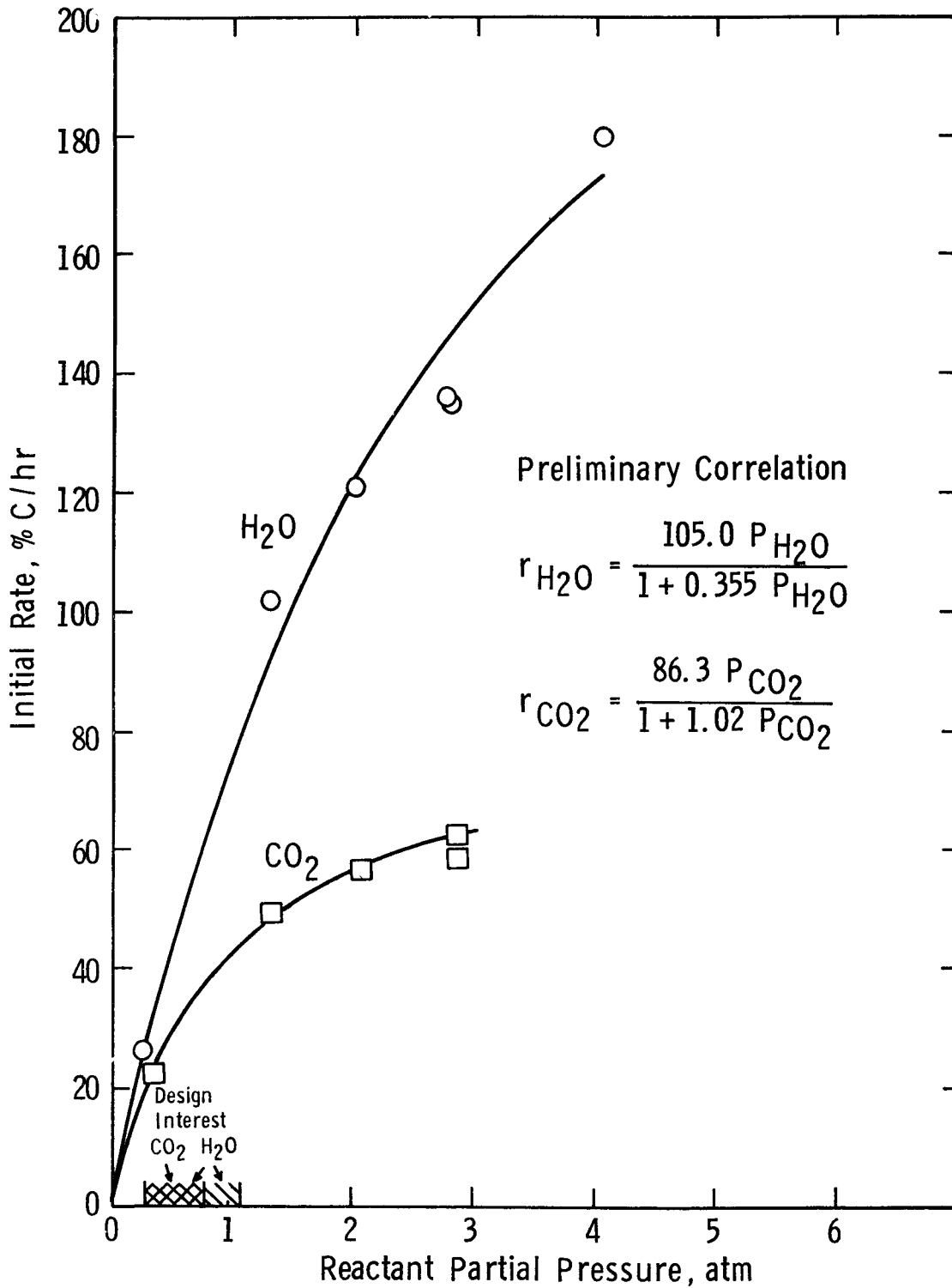
r = reaction rate, %C/hr  
 R = reaction rate constant, %C/hr·atm<sup>-1</sup>  
 K = inhibition constant, atm<sup>-1</sup>  
 K' = equilibrium constant  
 P = partial pressure, atm

with subscripts

e for equilibrium  
 r for reactant  
 p for product

Results of preliminary correlations at RV-55°F which ignore product inhibition, internal mass transfer, and the reverse reaction are given in Figure 6-4. These formulas may be quickly examined in the following comparison.

FIGURE 6-4  
MINIGASIFIER  
IKG ILLINOIS GASIFIER COKE  
RV-55°F, 50/70 Mesh



### Relative Reaction Rates

IKG Illinois Gasifier Coke  
RV-55°F, 50/70 mesh

<u>Reactant Partial Pressure, atm</u>	<u>rH<sub>2</sub>O/rCO<sub>2</sub></u>
0.0	1.2
0.5	1.6
3.0	2.3

This shows steam is always more reactive than CO<sub>2</sub> at RV-55°F. Since carbon dioxide more strongly inhibits through pressure, the reactivity ratio increases with partial pressure.

A design implication of these data is in the extrapolation of IKG gasification to commercial conditions. The range of design interest is indicated on Figure 6-4. The most probable design conditions are at pressures about 30% above IKG operations. If IKG carbon dioxide gasification were linearly extrapolated to design neglecting reactant inhibition, the predicted rates would be about 10% higher. For steam, this error would be about 3%. This suggests that shortcut extrapolations can be made neglecting product inhibition in the relatively linear design interest region. Of course, product inhibition will be included in rigorous procedures.

Thermal response provides insight to the nature of the mini-gasifier technique. Data over a range of 125°F is plotted in the form of the reaction rate constant versus the reciprocal of absolute temperature in Figure 6-5 using the well known Arrhenius expression for activation energy. The following activations are calculated assuming that reactant inhibition activation is zero as suggested by literature.

### Activation Energies, ΔE

IKG Illinois Gasifier Coke  
k cal/g mole °R

<u>Range</u>	<u>H<sub>2</sub>O</u>	<u>CO<sub>2</sub></u>
Overall	51	65
RV-130°F to RV-55°F	61	74

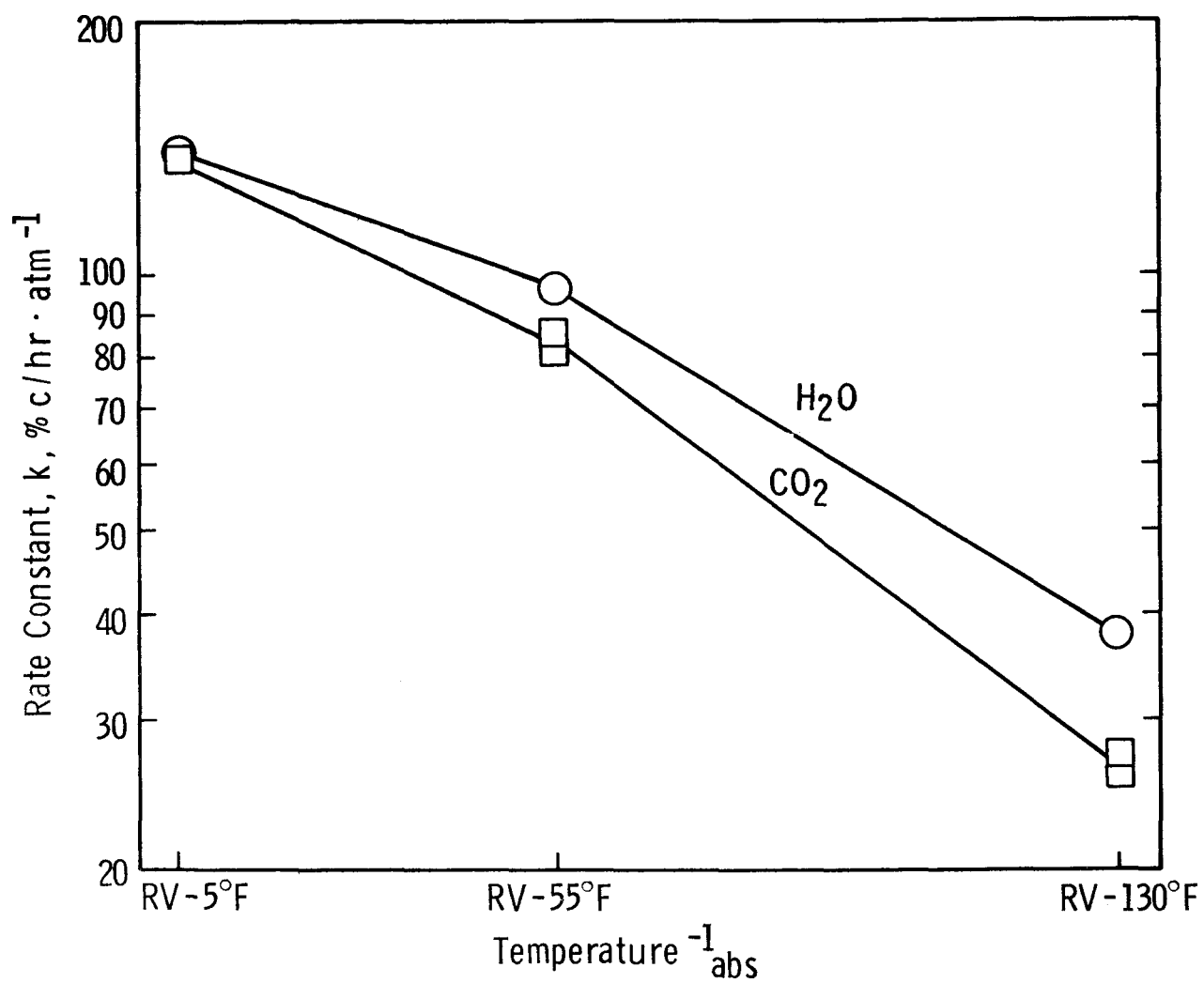


FIGURE 6-5

MINIGASIFIER

IKG ILLINOIS GASIFIER COKE

- 50/70 MESH COKE
- $\sim 2.8$  atm REACTANT PARTIAL PRESSURE



Activation energies in the literature for this temperature range are typically 50-60 kcal or close to the overall values. Since the mini-gasifier reactant gases are in high velocity down-flow to eliminate external mass transfer, these activation energies should reflect EDS coke activity. The higher activation energies over the lower temperature range for both steam and carbon dioxide are somewhat surprising. These may reflect a loss of Illinois coke mineral matter catalysis. It is noted that apparent activation energies in a fluidized bed will be lower as mass transfer effects will be present.

● Mass Transfer Effects Important

Data on velocity and particle size effects are now available for carbon dioxide gasification. As this reaction is slower than steam gasification, any apparent mass transfer effects should be even greater for steam. Previous work on velocity had shown that the high down-flow superficial velocity of about 19 cm/sec for most of this work eliminates external mass transfer limitations (see January-March, 1978, Quarterly Technical Progress Report [FE-2893-12]).

Some data were taken this year at low superficial velocity around 0.5 to 1.0 cm/sec, to investigate reaction rates at near minimum fluidization. Minimum fluidization exists in the emulsion phase of the gasifier fluidized bed and therefore is an important condition for the bulk of the coke. It was found that the low-velocity reaction rate was much lower or,

$$\frac{k_{\text{high vel.}}}{k_{\text{low vel.}}} = 3 \text{ to } 4$$

The data for this (see Appendix Table D-33) are at RV-55°F and 2.5 to 2.8 atm of carbon dioxide. Similar results had been found previously in the minigasifier and strongly support the existence of external mass transfer effects.

Data were also taken with coke particle sizes ranging from 100 to 400 microns (see Appendix Table D-33) at high velocity. One expression of these data is as follows.

Coke Particle Size Effects

IKG Illinois Gasifier Coke  
CO<sub>2</sub> Gasification, RV-55°F,  
19 cm/sec Superficial Velocity

<u>Average Coke Diameter, microns</u>	<u>Reaction Rate Constant* Dia (%C/hr)*microns*10<sup>-3</sup></u>
125	15
255	22
360	22

This suggests that reaction rate is proportional to the reciprocal of the coke diameter for larger particles but decreases for smaller particles. As external mass transfer limitations exist at low velocity, it is possible that internal mass transfer limitations are present and produces shellwise gasification near the coke surface. For smaller particles the whole particle may come into play with product inhibition effects. Additional work on gasifier coke surface area and porosity is planned to study this effect for both CO<sub>2</sub> and steam gasification data. It is noted that particle size effects are important as much of the IKG data are for 200 to 250 micron diameter cokes whereas the full-scale FLEXICOKING process is expected to yield 75 to 150 micron cokes. Therefore, diameter effects will be important in extrapolating IKG data to design.

#### ● Future Program

The near term program will be run with mixed gases, that is, reactants and products. This will be done to determine product inhibition effects which are expected to be larger than the reactant inhibition effects already studied. This will be followed by a program of steam, carbon dioxide and mixed gas runs with IKG Wyodak coke to develop the Wyodak data base. The Wyodak runs will probably be followed by rate studies of cokes that have been modified with additives to avoid sintering. Experimentation will next turn to other program cokes.

Upon completing analysis of the Illinois gasifier coke series, work will begin on modelling gasification. This will be split into short-cut modelling for scoping studies and rigorous models for design.

#### 6.7 Bench Coke Gasification - Ash Fusion Studies

The air/steam gasification of coal liquefaction bottoms coke in the FLEXICOKING process gasifier could lead to the formation of ash agglomerates which are considered to be related to potential bed sintering problems. The Small Fluidized Unit (SFU) described in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3) has been used to investigate the effect of process variables on the ash agglomeration in gasifying coal liquefaction bottoms derived cokes with steam and air. The objective is to determine potentially nonagglomerating regions in a well fluidized bed as a function of process variables such as temperature, superficial velocity and steam/feed ratio.

SFU runs were made on a -40+100 mesh sample of IKG gasifier coke from CLPP Illinois bottoms. Duplicated runs were made under each set of testing conditions. The bed agglomeration is determined by the presence of +40 mesh ash particles after the run. A conical gas distributor was used for this series of SFU runs in that it was observed to provide a well fluidized bed without any stagnant zones. Recently the 1-inch quartz reactor was modified to include an enlarged section of 2-inch diameter above the fluid bed to decrease the superficial velocity in the disengaging zone to avoid fines carryover in SFU runs.

- Mapping of Sintering/Non-Sintering Region by Process Variables

- + Sintering Conditions for Different Coal Bottoms

As reported in the October-December, 1978, EDS Quarterly Technical Progress Report (FE-2893-25), the sintering and nonsintering regions for Illinois and Wyodak bottoms coke could be mapped on a plot of gasification temperature versus superficial velocity. A common boundary of sintering/non-sintering regions can be drawn from this plot for both Illinois and Wyodak bottoms as shown in Figure 6-6. In other words, SFU results showed that sintering occurred at the same gasification conditions for both Wyodak and Illinois bottoms cokes.

The sintering behavior of different coals in the EDS process development program was investigated by gasification runs in the SFU with cokes prepared from RCLU bottoms produced from these coals. Bottoms coke samples for these SFU runs were prepared by a bench coker and ground to -40+100 mesh. So far, SFU runs were completed only on bottoms coke from North Dakota lignite. The bottoms coke from this lignite coal showed bed sintering under the gasification condition within the non-sintering region for Illinois and Wyodak bottoms cokes as illustrated in Figure 6-6. The bed defluidization appeared to be caused by particles sticking together. These agglomerates could be broken up by mechanical stirring or by pressing them with fingers. This particle agglomeration could, however, not be avoided at a low gasification temperature (RV-230°F) and the high superficial velocity in a SFU run. The high sodium content in the ash (~10%) of this lignite bottoms coke might be responsible for the severe sintering or agglomeration in SFU runs. This is being investigated by microscopic examinations and trace metal analyses on agglomerates from SFU runs.

Difficulties were encountered in preparing the other coke samples from different coals due to the high 1000°F<sup>-</sup> contents. The apparatus and/or procedure for coke preparation will be modified before proceeding with SFU runs on bottoms cokes from other coals.

- + Complete Versus Partial Gasification

The sintering/non-sintering region in gasification of Illinois (Monterey) and Wyodak bottoms cokes was defined by the data from SFU runs at 100% gasification. The gasifier of a commercial FLEXICOKING unit is generally operated at partial gasification. The EDS design target for the gasifier is about 80% carbon gasification. Additional SFU runs were, therefore, made on these bottoms cokes and were terminated before completion of gasification to see if this boundary of sintering/nonsintering region might be shifted due to the partial gasification.

As shown in Figure 6-7, results of these SFU runs at partial gasification fit well with the boundary defined by those at complete gasification. This suggests that bed sintering could occur before carbon gasification was complete. The condition for bed sintering did not appear to be appreciably affected by the gasification level.

FIGURE 6-6

MAPPING OF SINTERING ZONE FOR  
SFU RUNS WITH BOTTOMS COKE

( 10 - 15% STEAM )

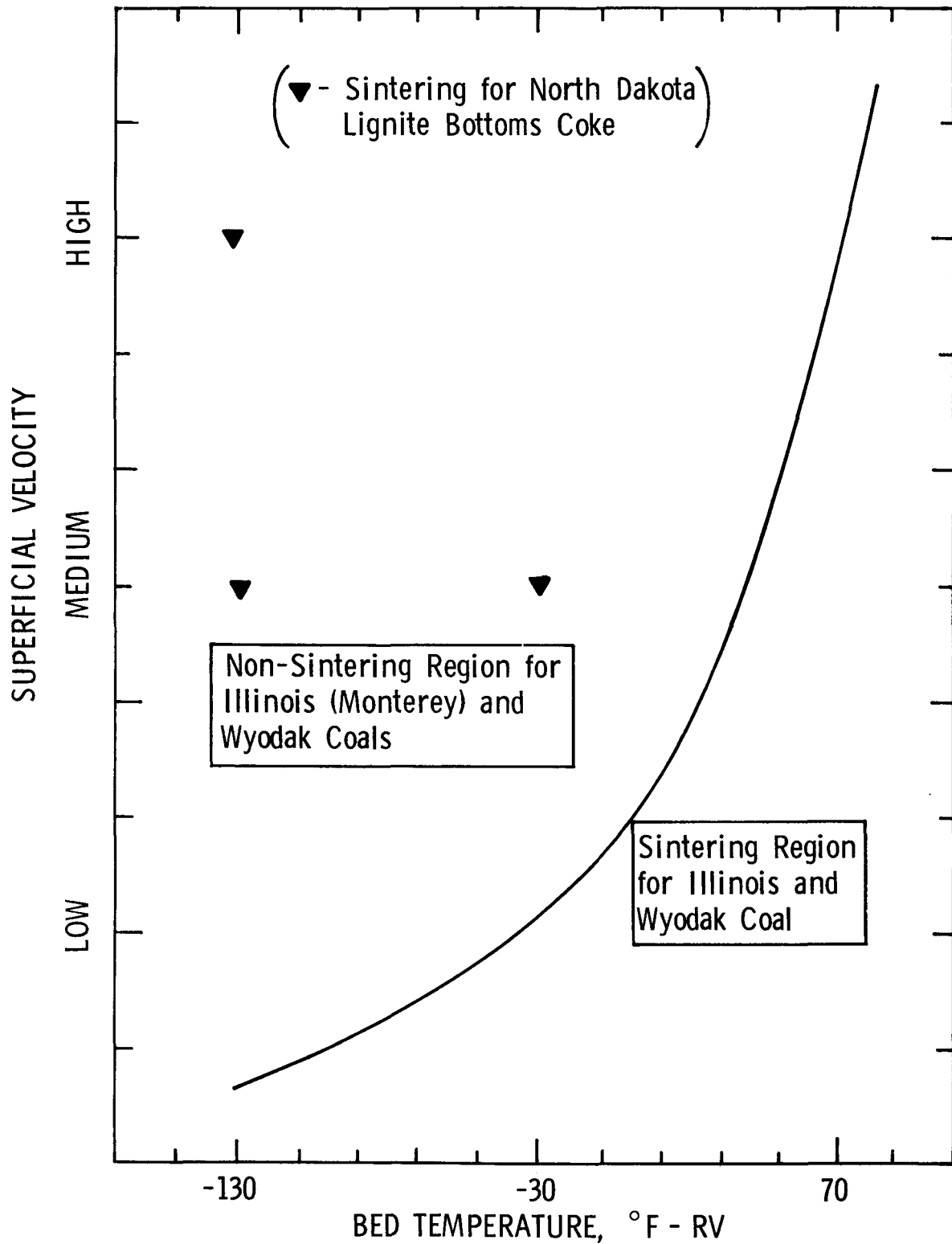
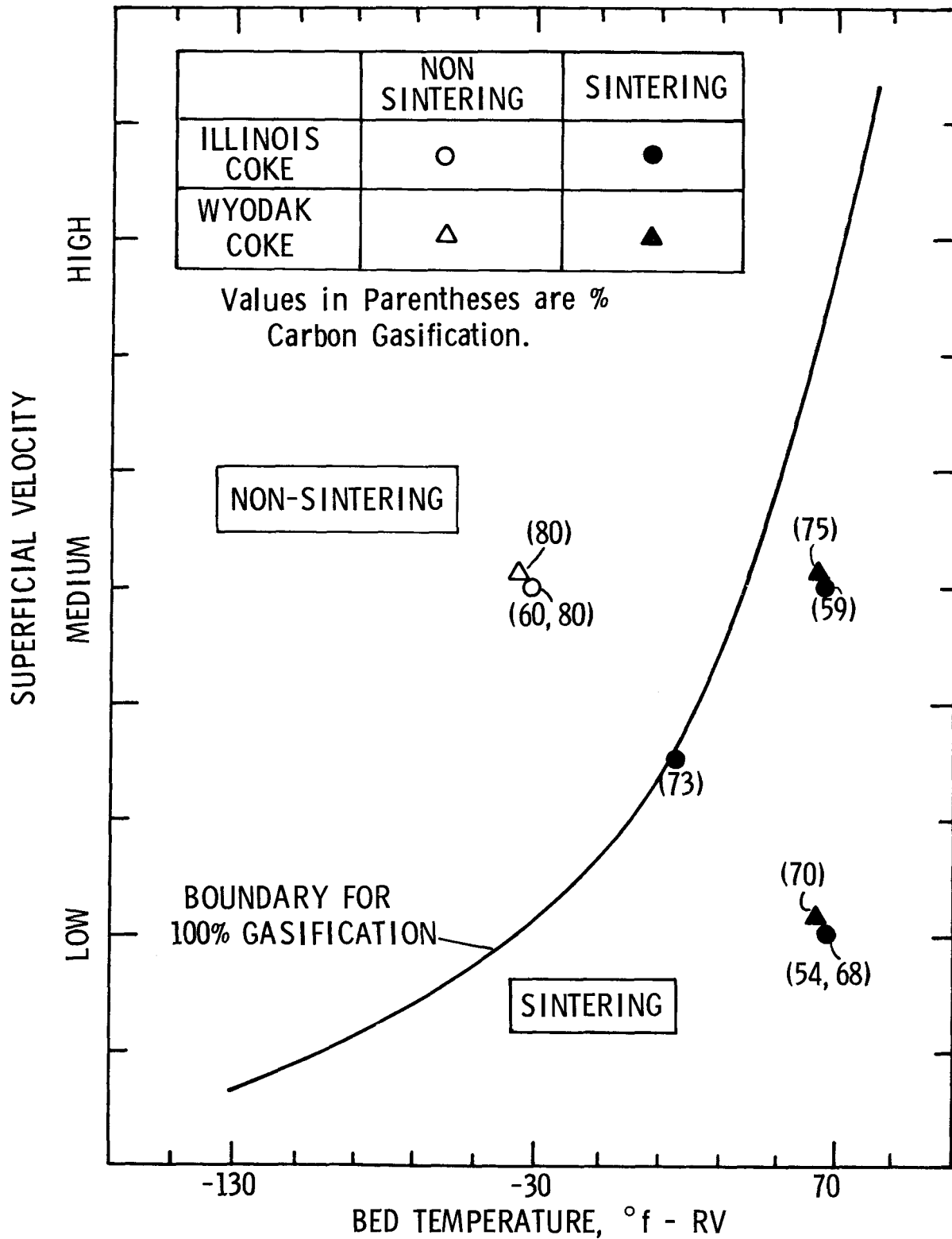


FIGURE 6-7

MAPPING OF SINTERING ZONE FOR  
SFU RUNS ON IKG BOTTOMS COKE

(PARTIAL vs. COMPLETE GASIFICATION)



# + Coke Particle Size Effect

Previous SFU runs for studying process variable effects on bed sintering were made on coke samples of -40+100 mesh. In EDS FLEXICOKING process unit study design, the coke particle mean diameter is projected to be finer than 100 mesh. SFU runs were therefore made on -80 mesh IKG cokes to see if the sintering problem might be aggravated by gasifying the finer coke particles. Due to the limited quantity of -80 mesh sample that could be sieved from IKG cokes, these additional SFU runs were made under the marginal gasification conditions for non-sintering observations in previous runs on -40+100 mesh coke samples.

As shown in Table 6-13, SFU runs showed no appreciable change of sintering condition in gasifying Wyodak bottoms coke of the finer particle size. However, bed sintering for the -80 mesh Illinois coke sample was observed at gasification conditions under which bed sintering did not occur for the -40+100 mesh coke samples. The sintered material, which appeared to be composed of agglomerated particles, was examined by the hot stage microscope. The micrographs (Figure 6-8) showed that the individual particles of agglomerates were finer than the non-agglomerated particles, but these particles melted at approximately the same temperature (2020°F). This indicates that particle dynamics plays an important role for controlling agglomeration in fluid bed gasification.

Due to the observed effect of particle size on sintering for Illinois bottoms coke, more SFU runs on -80 mesh Illinois coke will be made to gain added understanding of the interaction of particle size with process variable effects on bed sintering.

TABLE 6-13

## PARTICLE SIZE EFFECT ON BED SINTERING

• S - Sintering; NS - Non-Sintering

Gasification Temperature	RV-30°F		RV+70°F	
Superficial Velocity	Medium		Low	
Steam/Air Ratio	15		30	
Coke Particle Size, mesh	<u>-40+100</u>	<u>-80</u>	<u>-40+100</u>	<u>-80</u>
Sintering Observation:				
Illinois Coke	NS	S*	NS	S
Wyodak Coke	NS	NS	NS	NS

\*Small amount of agglomerates in bed drain (0.4 wt %).

FIGURE 6-8

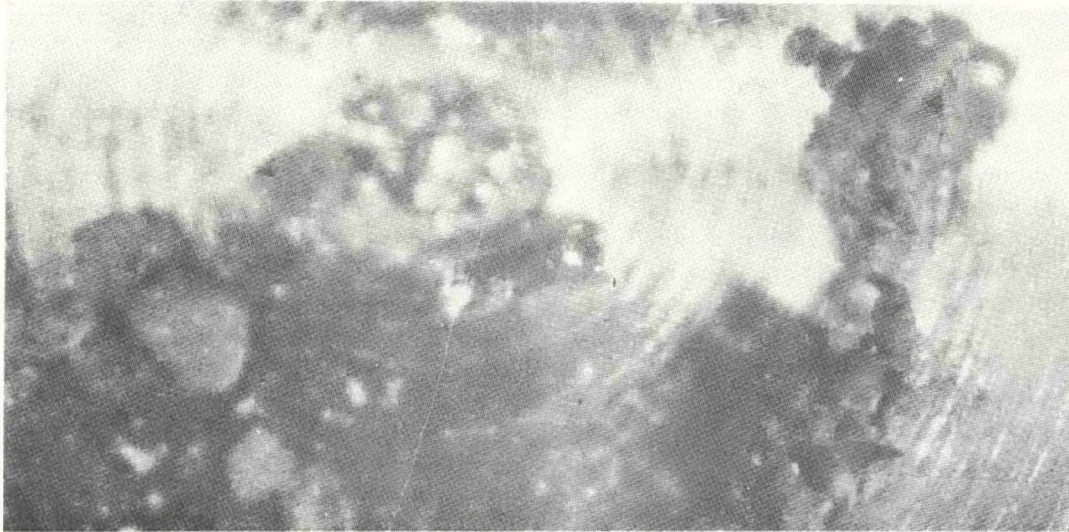
MICROGRAPHS OF HOT STAGE MICROSCOPE

- SFU RUNS ON -80 MESH ILLINOIS BOTTOMS COKE

AGGLOMERATES



SINGLE PARTICLES



BEFORE MELTING



AFTER MELTING @ 2020°F



### ● Mechanism of Bed Sintering

The nominal gasifier conditions in IKG runs on Illinois and Wyodak bottoms were within the non-sintering region as defined by SFU runs. The bed sintering in IKG is, therefore, believed to occur in the stagnant coke zone in the bed as observed by the cold model studies and the gasification runs in the Fluidized Bed Unit (see Section 6.8 following). It is not necessary to locally overheat the stagnant coke in the stagnant zone to cause sinter formation. Sintering of a material can occur, as cited in the literature, a temperature below its fusion temperature by any one or all of the following material transport phenomena: surface diffusion, volume diffusion, viscous flow and evaporation/condensation.

In a well fluidized bed, sintering can occur under more severe gasification conditions as demonstrated by the mapping of sintering/non-sintering regions with process variables in Figure 6-6. Evidences from SFU runs showed that sinter formation in a well fluidized bed might follow the following four steps:

1. Particle surface stickiness--surface softening, local melting or plastic deformation.
2. Particle agglomeration.
3. Defluidization of agglomerates.
4. Sintering of defluidized agglomerates.

The particle surface stickiness is dependent upon the particle temperature and chemical properties of the ash particles. The particle temperature in air/steam gasification could be higher than the measured fluid bed temperature due to the inadequate dissipation of the heat of combustion of the coke particles, as reported in the October-December, 1978, EDS Quarterly Technical Progress Report (FE-2893-25). SFU runs on IKG Illinois coke were made by using steam, nitrogen and CO<sub>2</sub> replacing oxygen to simulate steam/air gasification without the combustion on coke particles. Particle temperature in these runs should be close to the measured bed temperature. Results from these runs showed that the Illinois coke particles could become sticky enough to cause sinter formation at a temperature 100-200°F higher than the nominal IKG gasifier temperature. This temperature is still lower than the initial deformation temperature in ASTM fusion temperature determinations on Illinois coke ash. The formation of relatively low-melting mineral matter in gasification could also cause stickiness of ash particles. The identification of fayalite, (2FeO·SiO<sub>2</sub>, m.p. 2200°F) in Illinois coke ash by X-ray diffraction is evidence of such a chemical conversion of mineral matter in gasification atmosphere.

The agglomeration of fine particles in bed sintering has been demonstrated by the hot stage micrograph in Figure 6-8. The molten junctions between agglomerated particles could be of micron size as shown by a

Scanning Electron Micrograph (SEM) in Figure 6-9. Apparently these agglomerated particles are held together loosely. This seems to explain the possibility of reducing sintering tendency by increasing the particle momentum at the higher superficial velocities. When the agglomerates grow too large to fluidize, they settle to the bottom of the fluidized bed as stagnant material, where the sintering process progresses to form stronger agglomerates.

- Change of Particle Size, Density and Sulfur Retention at Various % Gasification

In carrying out SFU runs to various levels of gasifications, the data of size distributions, densities and elemental analyses of feed cokes and bed drains were obtained for investigating the change of particle properties with gasification. Figure 6-10 shows the decreases of particle size and density for Illinois and Wyodak bottoms cokes in SFU runs at different levels of gasification as expressed by % ash in bed coke. As can be seen, the particle shrinkage of both bottoms cokes in gasification reached an asymptotic level after the ash content in the coke was increased to about 60%. The final particle size of the coke ash was about 80% of its original size. The density of coke particles, however, decreased almost linearly with the % ash in the bed coke. The low density of Wyodak coke ash may cause the fines carryover problems in the gasifier as demonstrated in SFU runs (January-March, 1979, EDS Quarterly Technical Progress Report [FE-2893-29]).

In elemental analyses on the bed drains from SFU runs, it was noted that the Illinois bottoms coke after partial gasification still had an appreciable content of sulfur. The sulfur retention in coke after partial gasification is illustrated in Figure 6-11.

## 6.8 Coke Gasification - Operability Studies

Two major questions of gasifier operability that have emerged in the development of the EDS FLEXICOKING process are particle integrity and sinter formation. Both questions were addressed in coke gasification studies in the Fluidized Bed Unit (FBU), which is a three-inch diameter fluidized bed designed for batch gasification of coke with air and steam. A description of the FBU was given in the January-March, 1977, EDS Quarterly Technical Progress Report (FE-2353-14).

- FBU Fines Make Studies

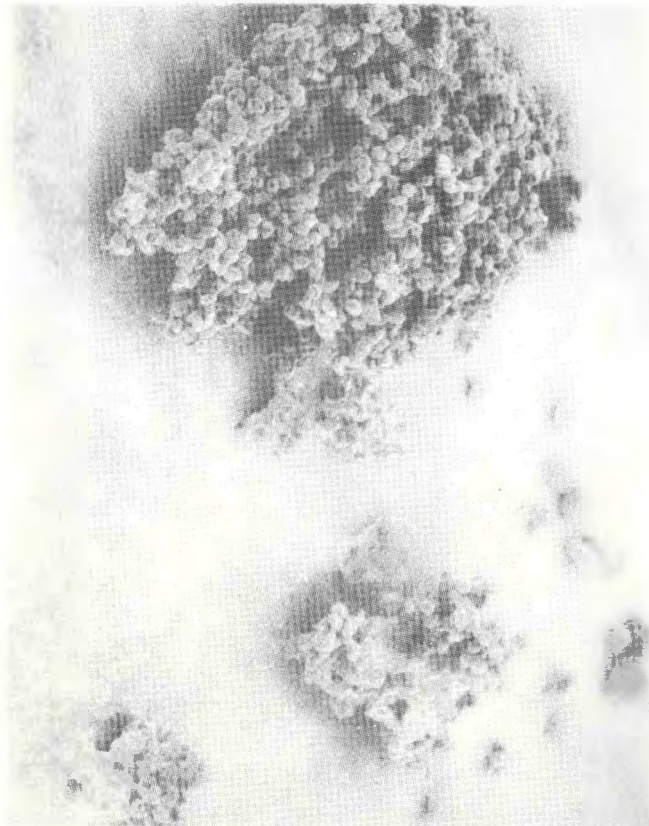
The effect of gasification on coke particle integrity was investigated by measuring the fines that were produced during gasification in the FBU. An indication of the ultimate carbon utilization can be obtained from the amount of fines produced and the ash content of the fines.

FIGURE 6-9

SCANNING ELECTRON MICROGRAPHS SHOWED MOLTEN  
JUNCTIONS BETWEEN AGGLOMERATED PARTICLES

- IKG Gasifier Coke from Illinois Bottoms After SFU Run
- Temperature: RV + 70
- Steam/Air Ratio: 45%
- Superficial Velocity: Medium

|———— 3 mm ———|



Agglomerates, 20X

|— 20  $\mu$  —|



Particle Junctions, 200X

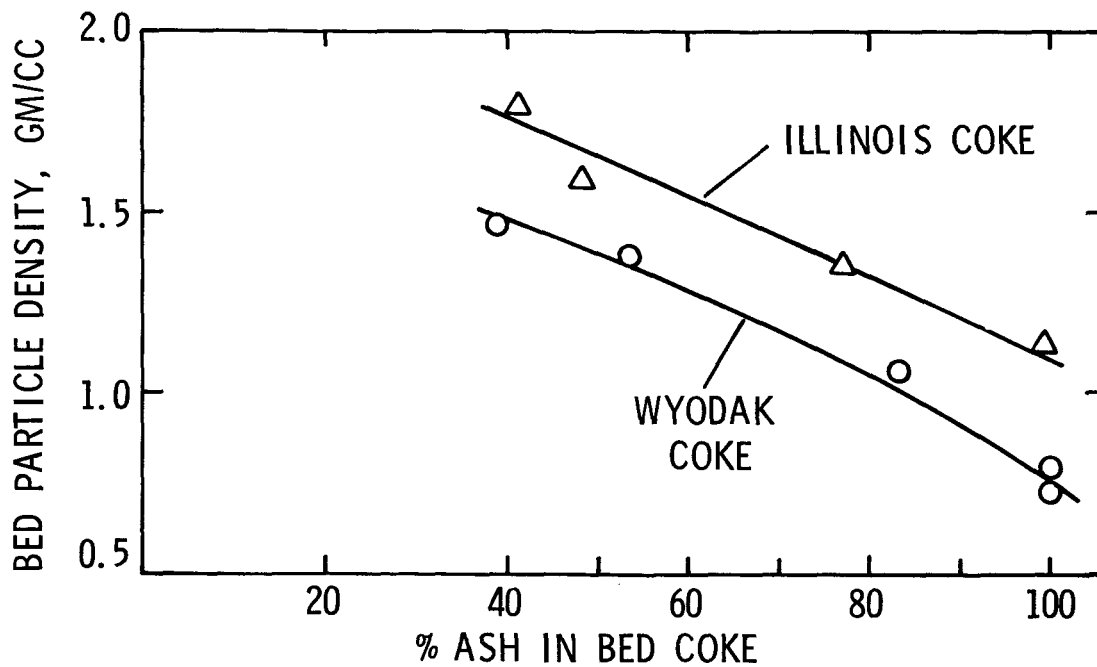
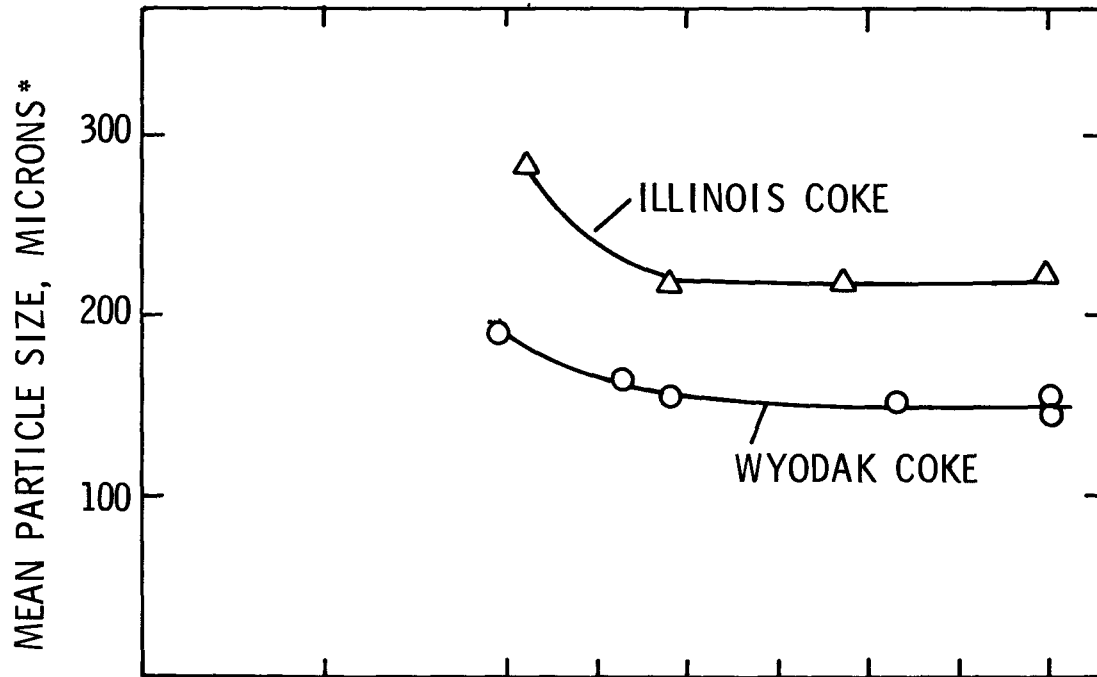
Major: Si

Minor: Fe, Al, K, Ca

FIGURE 6-10

DECREASE OF PARTICLE SIZE AND DENSITY IN SFU RUNS  
AT VARIOUS % GASIFICATION

● 1KG ILLINOIS AND WYODAK BOTTOMS COKE FEED

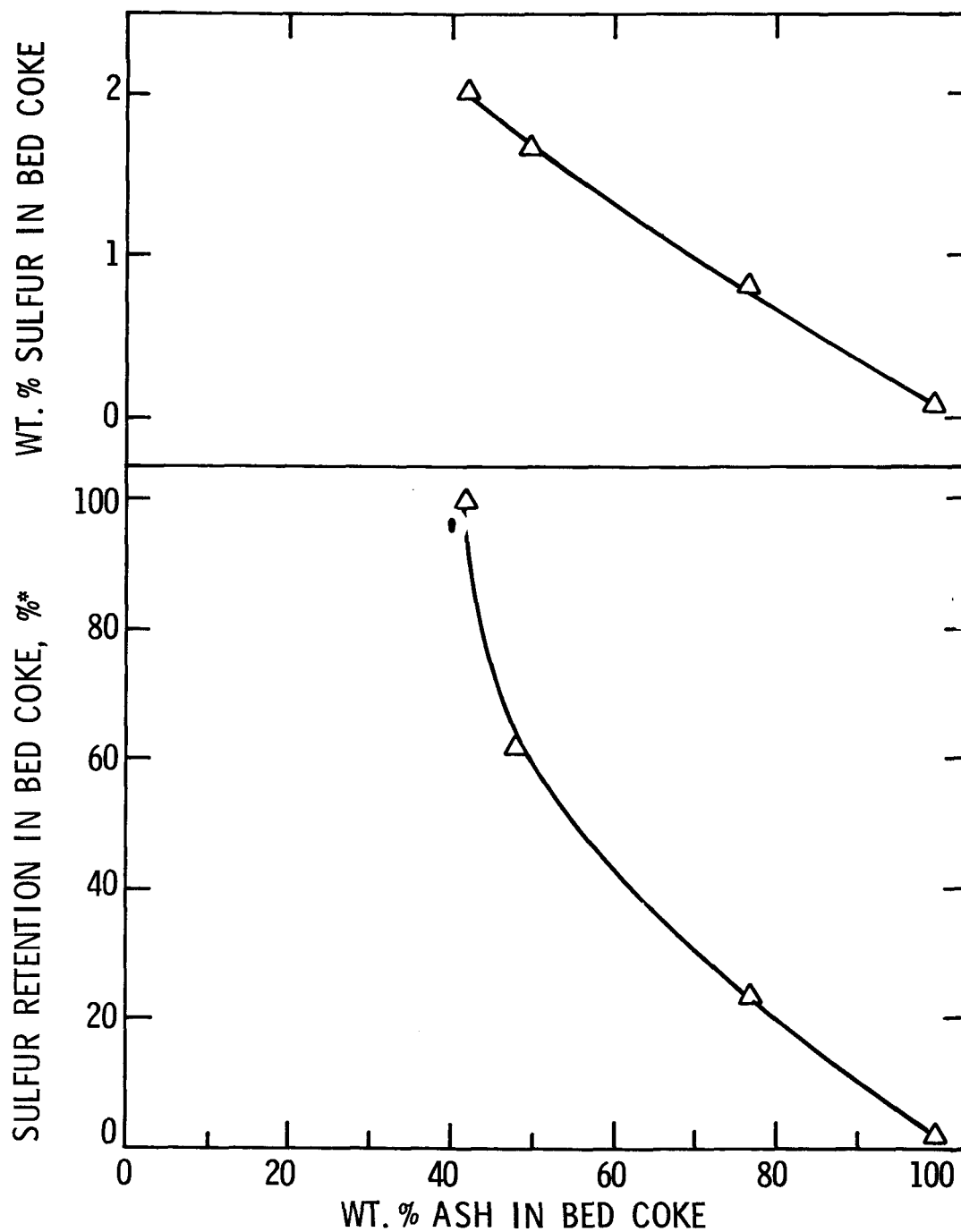


\* Volume/Surface Mean Diameter ( $d_{VS}$ ) from Sonic Sieve Analyses.

FIGURE 6-11

SULFUR RETENTION IN COKE ASH IN SFU RUNS  
AT VARIOUS % GASIFICATION

● 1KG ILLINOIS BOTTOMS COKE FEED



\* (Wt. of S in Bed Coke / Total S in SFU Feed Coke) x 100%

FBU studies indicate that coke made from EDS Illinois or Wyodak bottoms maintains sufficient particle integrity to allow gasification in the FLEXICOKING process. The fines make is higher than petroleum but the low carbon content of the fines keeps the carbon loss with the fines relatively low. The experimental evidence with EDS bottoms cokes suggests that as gasification proceeds, the outer layers of the coke particles are composed of highly gasified coke that is mostly ash. The amount of fines generated depends on the intensity of attrition and the attritability of the particles. Comparisons of the absolute amount of fines make from different units should be made with caution because of the complex dependence of fines generation on unit geometry and operating conditions, as well as on the properties of the coke itself.

+ Effect of Cyclone on FBU Fines Make

Data were reported in the July-September, 1978, EDS Quarterly Technical Progress Report (FE-2893-21) that showed that half the fines produced in the FBU were caused by attrition in the cyclone. Hot attrition tests reported in the October-December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7) also showed a strong effect of attrition in the FBU overhead system on the generation of fines. All these tests were made with EDS Illinois coke produced on either the IKG or LSCU. The high ash content of the fines that were produced by attrition supported the ash-layer theory of EDS coke attrition.

+ Effect of Gasification Temperature on Fines Make

The effect of gasification temperature on fines make was investigated by a number of runs with both Illinois and Wyodak coke. It was thought that fines make might be lower at higher temperature because sintering of the outer layer might reduce the fines make caused by attrition. Data were presented in the July-September, 1978, EDS Quarterly Technical Progress Report (FE-2893-21) that indicated there was no consistent effect of temperature on fines make over a 100°F range of temperature.

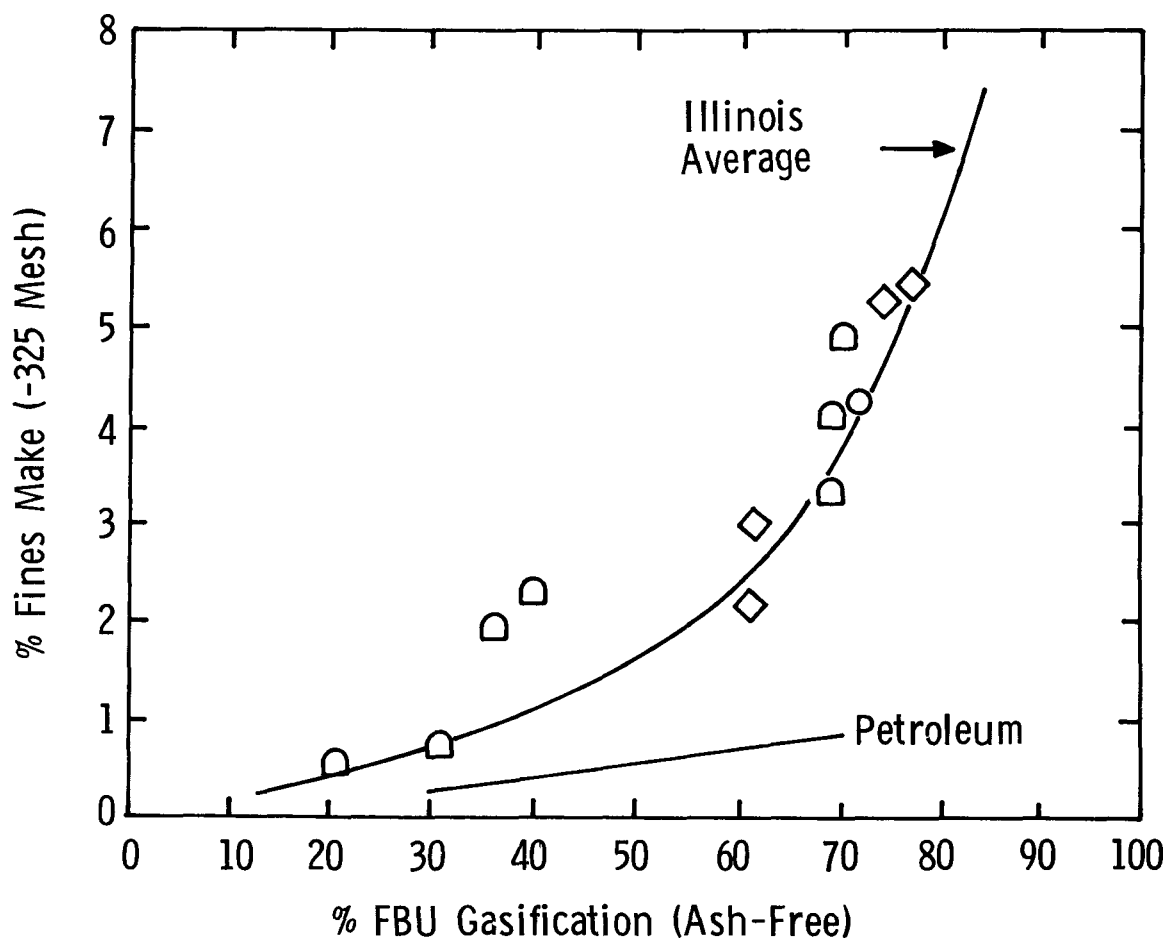
+ Comparison of Fines Makes With Illinois and Wyodak Coke

Data were presented in the July-September, 1978, EDS Quarterly Technical Progress Report (FE-2893-21) which showed that the fines makes with Illinois and Wyodak cokes were comparable. Five additional FBU runs were made with IKG Wyodak coke, especially to better define the fines makes at moderate levels of gasification. Data are shown in Figure 6-12. The curve labeled "Illinois Average" represents the average of fourteen FBU runs with Illinois coke produced in the IKG reactor when it was operated in the FLUID COKING mode. The datapoints are for coke produced in the IKG reactor when it was operated in the FLEXICOKING mode, that is, where coke gasification was an integral part of the process. The amount of fines produced in the FBU gasification runs was the same regardless of whether the coke was produced in the FLUID COKING or the FLEXICOKING mode of operation. This is surprising because one would think that the

Figure 6-12

FBU GASIFICATION FINES MAKE  
COMPARISON OF ILLINOIS AND WYODAK COKES

- IKG WYODAK RV - 30°F
- IKG WYODAK RV - 130°F
- ◇ IKG ILLINOIS RV - 30°F



gasification experienced in the FLEXICOKING process would cause the core of the coke particles to be weakened and thus more susceptible to attrition. Apparently, the layering of fresh coke on the circulating particles in the coking reactor was sufficient to maintain particle integrity. This conclusion is based on Illinois coke only because no Wyodak fluid coke was produced by the IKG.

Data are shown for two gasification temperatures because the gasification activity of Wyodak coke was greater than Illinois coke so that the standard FBU gasification temperature of Wyodak coke was 100°F lower than that of Illinois coke. As mentioned earlier, there was not a significant effect of temperature on FBU fines make for this range of temperature.

The data in Figure 6-12 show that Illinois and Wyodak cokes have similar overall fines makes. However, the two Wyodak runs at 37% and 41% gasification suggest that the Wyodak coke has higher fines makes at intermediate gasification. Additional fines make data were obtained by measuring cyclone fines at time intervals during each run. The total amount of FBU fines make data for IKG coke made in the FLEXICOKING process mode are shown in Figure 6-13. With all the intermediate data points included, it is difficult to detect any significant difference in the fines makes with Illinois and Wyodak cokes.

The two cokes show definite differences with respect to the ash enrichment of the fines. Figure 6-14 shows that the fines from Illinois coke exceeded 90% ash after only 20% gasification in the FBU while the Wyodak fines were less than 70% ash at 20% gasification. The Wyodak fines gradually increased to 90% ash after 60% of the carbon had been gasified. The ash levels of fines at 20% FBU gasification correspond to the ash levels in the IKG heater/ gasifier fines that were reported in the July-September, 1978, EDS Quarterly Technical Progress Report (FE-2893-21). The gross coke carbon gasification in the IKG was about 70%; however, due to the coke circulation between reactor and gasifier vessels, the average gasification per pass of an individual particle was considerably lower than 70%. Thus, there is some basis for comparing Illinois and Wyodak behavior at intermediate levels of gasification.

A summary of the FBU coke gasification comparison of Illinois and Wyodak cokes is that fines makes are similar and ash contents of the Wyodak fines are generally lower. At 20% FBU gasification, the FBU data show close agreement with IKG experience with respect to ash content and similar fines makes for Illinois and Wyodak. The IKG did, however, show somewhat higher heater-gasifier fines makes with Wyodak than Illinois in the gasifier.

The FBU data for fines makes and ash contents are combined in Table 6-14 to show calculated carbon losses in the fines. Though the amount of fines made with the EDS cokes is 2-5 times that found for the Billings petroleum coke, the low carbon contents of the EDS cokes keeps the carbon rejection from being excessive. In fact, for the six comparisons shown, only one condition showed a higher carbon



Figure 6-13

FBU COKE GASIFICATION FINES MAKE  
COMPARISON OF ILLINOIS AND WYODAK COKES

- IKG ILLINOIS COKE RV - 30°F
- △ IKG WYODAK COKE RV - 130°F

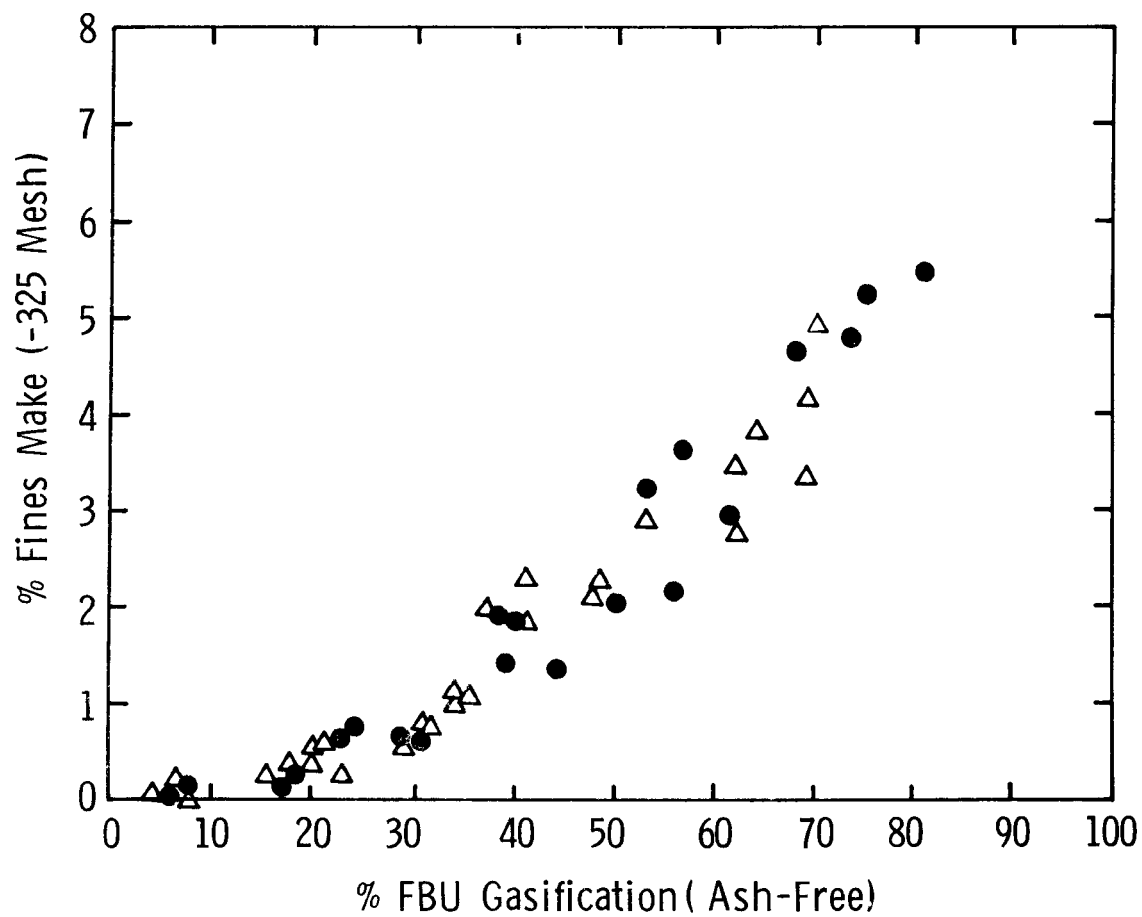
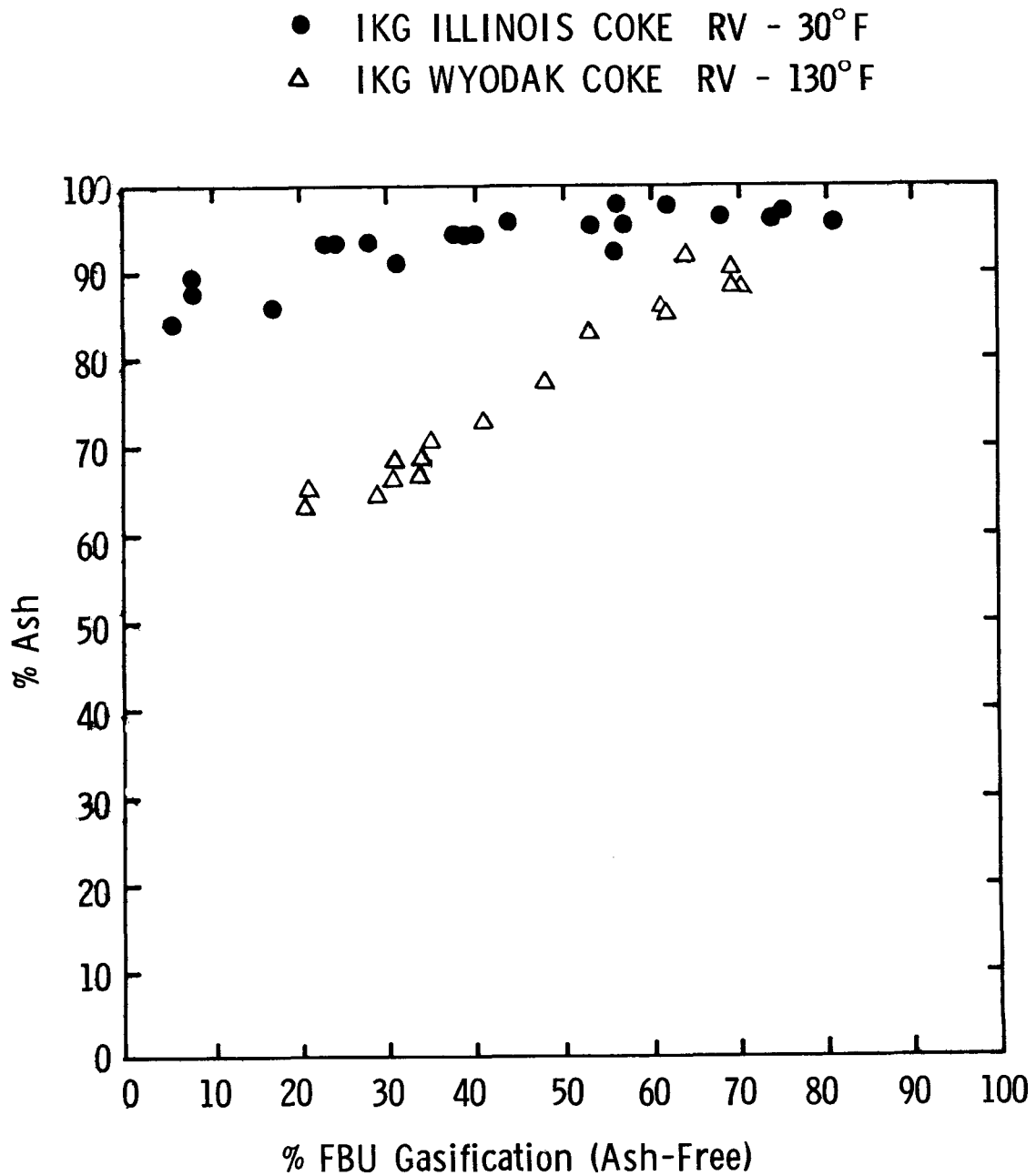


Figure 6-14

FBU COKE GASIFICATION ASH CONTENT OF FINES  
COMPARISON OF ILLINOIS AND WYODAK COKES



rejection than the Billings coke. This was Wyodak coke at 40% gasification. Extrapolation of these results to a large-scale continuous unit are not recommended. However, the results do indicate that coke particle integrity should not prevent application of the FLEXICOKING process to the bottoms from the EDS coal liquefaction process.

TABLE 6-14

FBU COKE GASIFICATION RESULTS  
CARBON REJECTION IN FINES

	<u>FBU Coke Gasification</u>		
	<u>20%</u>	<u>40%</u>	<u>60%</u>
<u>Wyodak Coke</u>			
Fines (% of feed coke)	0.5	2.0	3.3
Carbon (wt % in fines)	40.0	28.0	14.0
Carbon Rejection:			
% of feed coke	0.2	0.56	0.46
Relative to Billings	0.69	1.47	0.70
<u>Illinois Coke</u>			
Fines (% of feed coke)	0.5	1.7	3.3
Carbon (wt % in fines)	12.0	6.0	5.0
Carbon Rejection:			
% of feed coke	0.06	0.1	0.17
Relative to Billings	0.21	0.26	0.26
<u>Billings Coke</u>			
Fines(% of feed coke)	0.3	0.4	0.7
Carbon (wt % in fines)	98.0	96.0	94.0
Carbon Rejection:			
% of feed coke	0.29	0.38	0.66

+ Future Work

No additional work is planned on fines make from coke gasification. However, an increasing concern is carryover of fines into the overhead of the coking reactor. The FBU is being modified to perform coking reactor operability studies on bogging and fines.

## LABORATORY PROCESS RESEARCH AND DEVELOPMENT

### BOTTOMS PROCESSING RESEARCH

#### 7. Construction and Operation of the 2 B/D Coking/Gasification Pilot Plant (IKG)

The Integrated Coking/Gasification Pilot Plant (IKG) is being used in the development of air FLEXICOKING for EDS bottoms processing. It is the smallest unit available in ER&E for integrated operation in which fluid coke is continuously formed and gasified. The IKG is used primarily to assess operability on varying feedstocks; however, data in areas such as rate of coke gasification can also be obtained.

The IKG was not operated during the year of this report. IKG associated studies this year were data analyses conducted in the third quarter of 1978 (see July-September, 1978, EDS Quarterly Technical Progress Report [FE-2893-21] for detailed report) on second quarter operations. The operational results were discussed in the previous annual report (see July, 1977 - June, 1978, EDS Annual Technical Progress Report [FE-2893-17]). The technical findings of this year's studies are in the areas of coke particle integrity and gasification kinetics.

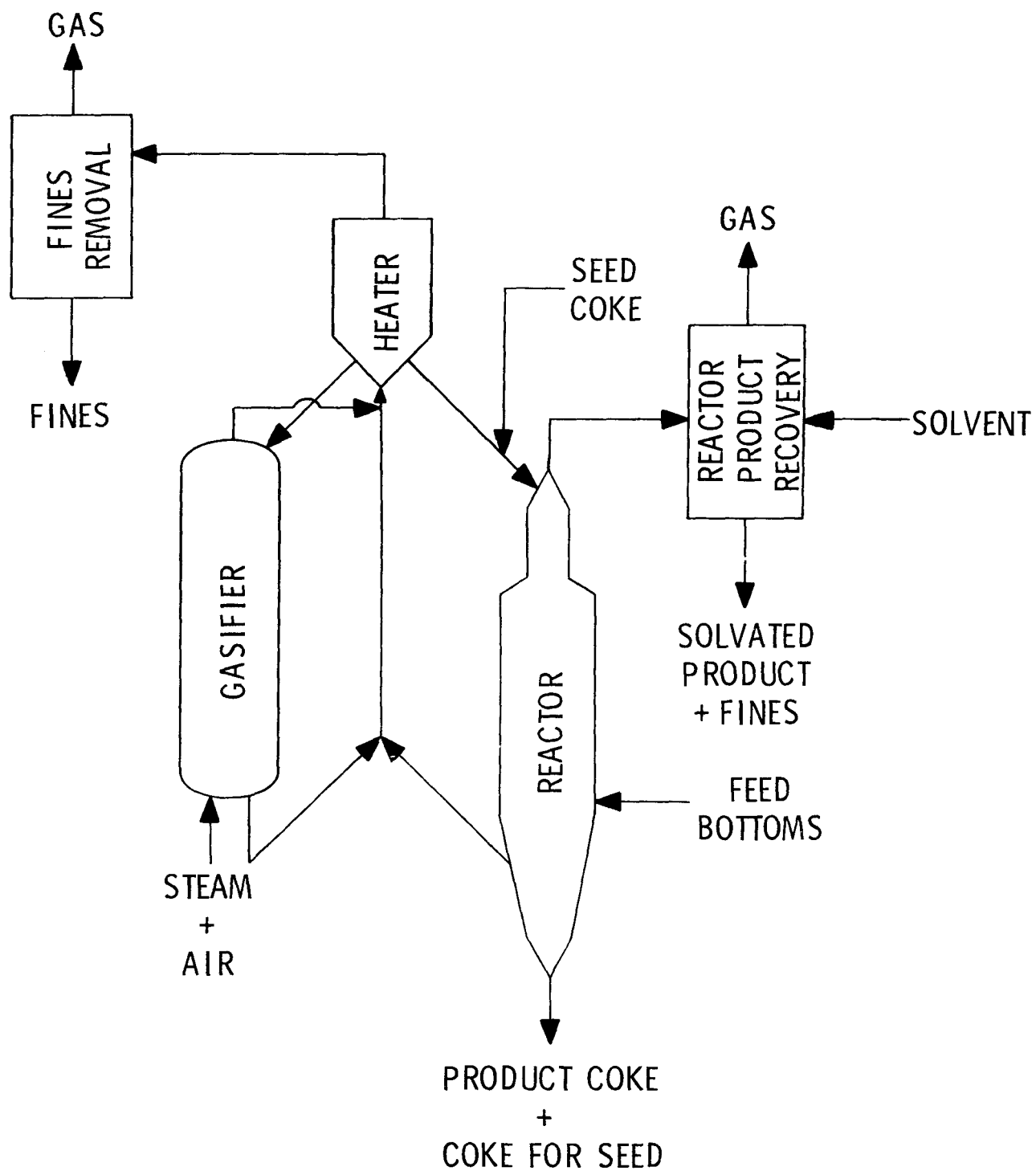
The IKG schematic in Figure 7-1 is included for reference. The bottoms feed of this study was CLPP liquefaction bottoms from Illinois (Monterey No. 1 mine) No. 6 bituminous coal operation and Wyoming (Wyodak mine) subbituminous coal operation. The bottoms were diluted with about 10 wt % creosote oil and pumped through a single feed nozzle to the reactor. The coke produced circulates between the reactor, heater and gasifier as indicated. Product coke and coke for feed grinding were withdrawn from the reactor bottom. Gristmilled seed coke was returned to the heater-reactor hot coke line. The seed coke operation is a pilot unit practice which is not done in commercial petroleum operation. Gases, liquids, and fines were taken from the system as shown.

#### 7.1 Annual Summary

##### • Unit Material Balances Show Improvement

Results from the first IKG air FLEXICOKING simulations in 1977 with EDS bottoms produced concerns about data reliability because of low ash balances. The second quarter 1978 operations yielded periods of 97% ash balance which were much better than the 1977 74% balances. This ash balance improvement is attributed to modifications of the seed coke and reactor fines return systems and dispelled many of the data reliability concerns. It was also found in regard to ash that ash elements were not selectively distributed between bed coke and fines for either Illinois or Wyodak IKG operations.

FIGURE 7-1  
IKG FLEXICOKING MODE SCHEMATIC



Feed balances were made for two periods of Wyodak operation. Product gas, liquid and cokes accounted for 97 to 98% of the feed in these periods. This further supported the IKG material balance reliability. In regard to feed balances, it was found that IKG product distributions were consistent with stirred coker (CSCU) results if the IKG's high reactor steam rates of around 400 wt % on feed were considered.

It is noted that the EDS coke of the second quarter 1978 came very close to equilibrium for IKG operation and essentially all of the starting seed coke was displaced. For Illinois operations, the starting petroleum seed coke had already been 96% displaced during 1977 operations and probably reached 97 to 98% displacement. Illinois gasifier coke ash increased to 48 wt.% from 43% at the end of 1977. The Wyodak operations began with Wyodak seed produced in the LSCU which was in turn 99% displaced. The gasifier coke ash reached 43 wt % which was very close to a projected 48% for IKG equilibrium at 78% gross coke carbon gasification. These displacement and equilibrium considerations are important in that they say such phenomena as particle attrition and gasification were occurring on near equilibrium IKG coke. This is important as the large ratio of system coke capacity to feed for the IKG makes it difficult to obtain equilibrium coke as compared to lower ratio commercial systems.

- Wyodak Fines Higher Than For Illinois

Fines producing mechanisms in the IKG differ substantially from commercial scale. The IKG's seed coke production loop is a significant illustration of this. Therefore, EDS fines data from the IKG is only indirectly relevant to design as it relates to petroleum experience where IKG to commercial practice tie-ins exist. However, EDS coke integrity issues make fines production a required study.

IKG fines production may be broken into heater-gasifier fines and reactor fines. Heater-gasifier fines are defined as -325 mesh particles. Wyodak operation resulted in 6 to 7-1/2 wt % fines on gross coke whereas Illinois operation yielded less than 6% fines. Based on carbon alone these results are 3 to 4 wt % of Wyodak gross coke carbon versus 1% in Illinois fines. Although Wyodak operation gave more carbon rejection, it was not high enough to limit operation to the commercial design level of 80% gasification. Also, carbon rejection was not unusually high compared to petroleum experience at similar levels of gross coke gasification.

The picture for second quarter 1978 reactor fines production is less clear. Illinois operations yielded reactor fines of 1 to 3 wt % on gross coke which is similar to 1977 Illinois operations. This agreement is somewhat surprising in that ash balance improvements are attributed to seed coke and reactor fines loop modifications which should have increased reactor fines make. However, Wyodak reactor fines production at 7 to 25 wt % of gross coke was unexpectedly high. It is not clear from operations data alone what factors are significant in this high Wyodak rate. Grinding tests showed Wyodak coke is more susceptible to grinding than Illinois which could lead to higher reactor fines. However, grinding tests did not

show the magnitude of difference that operating fines collection did. It is possible that more than one mechanism is important here. A bench program will soon be underway to test if reactor coke bed properties and production mechanisms could have also influenced the difference.

- Wyodak Coke Shows More Gasification Activity Than Illinois Coke

Second quarter 1978 Illinois coke gasification rates continued at the same rates as in 1977. The rates are very similar to petroleum coke rates. Wyodak coke was consistently more active toward gasification than Illinois. This increased activity permitted lowering of gasification temperatures about 50°F to avoid mineral matter sintering. It is suspected the increased activity is due to catalytic effects of the mineral matter. Coke BET surface areas do not account for the activity differences. Similar activity differences have been seen in bench gasification of the cokes and in earlier in steam gasification of the parent coal chars. The IKG EDS coke gasification data is now serving as a cornerstone to bench gasification data for constructing detailed gasification models of EDS air FLEXICOKING.

## 7.2 Future Plans

No IKG operations are scheduled for the remainder of 1979. Late in 1979 a decision will be made on 1980 IKG operations. The expected 1980 operation involves sintering control.

ENGINEERING RESEARCH AND DEVELOPMENT

PROCESS ENGINEERING AND COST EVALUATIONS

1. Commercial Study Design and Cost Estimate

EDS Study Design Update - Illinois Coal Base Case

1.1 Onsite Design Basis Revision - Illinois Coal Base Case

The onsite basis for the Study Design Update Base Case was issued as an interim report in May 1978, (FE-2893-10). Since that time, various basis changes have occurred. The major changes include the following:

- + Utilization of C<sub>1</sub>-C<sub>3</sub> as steam reforming feed whereas original basis utilized plant purge gas containing H<sub>2</sub> and C<sub>1</sub>-C<sub>3</sub> gases as feed.
- + Increase in the available FLEXICOKING capacity to 122.4% of the normal vacuum bottoms production.
- + Increase in the required FLEXICOKING scrubber bottoms recycle.
- + Additions/modifications to the tankage basis to reflect the production of a blended fuel oil with sulfur and solids specifications.
- + Reduction in vacuum bottoms tankage to reflect elimination of surge storage.

The original interim report on the Onsite Basis has been revised and will be reissued during the third quarter of 1979 with all changes made during the Study Design phase.



## 1.2 Onsites Design - Illinois Coal Base Case

Work was completed on the onsite design for the Illinois Coal Base Case of the EDS Study Design Update. The onsite sections include the following:

- + Liquefaction (section 100)
- + Product Fractionation (section 150)
- + Solvent Hydrogenation (section 200)
- + Solvent Fractionation (section 250)
- + FLEXICOKING (section 300)
- + Cryogenic Hydrogen Recovery (section 400)
- + Hydrogen Generation and Compression (section 425)
- + Gas and Water Treating (section 500)
- + Light Ends Recovery (section 600)
- + Other Onsite Support Facilities (flushing and blowdown system, CO<sub>2</sub> compression, etc.)

Heat and material balances, flowschemes, equipment design, and utility estimates were developed for all the onsite sections.

Design information packages for these sections were released to cost estimating. These packages included equipment lists, specifications of individual equipment pieces and flow diagrams. The design information reflects the effect of end-of-run operating conditions in solvent hydrogenation. The utility requirements were used in developing the offsite design basis.

Descriptions of these facilities have appeared in previous reports (refer to FE-2893-17, FE-2893-21, FE-2893-25 and FE-2893-29). Remaining efforts center on completing final documentation of the onsite designs. Final documentation will appear at a later date as an interim report covering the complete Illinois Coal Study Design Update effort.

### 1.3 FLEXICOKING Unit - Design Sensitivity Cases for The Illinois Coal Base Case

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During and following the design of the FLEXICOKING units for the EDS Study Design Update - Illinois Coal Base Case, a number of process uncertainties and potential improvements were identified. In order to assess the impact of design assumptions and the incentive for process optimization, design sensitivity cases were prepared and cost estimated.

The results of some of these sensitivities follow:

- The original coke handling facilities design was overly conservative. The design was completed without complete awareness of the cost impact on the FLEXICOKING unit. Facilities included several days onsite storage of purge coke in pressure vessels and a startup coke silo for each of the three FLEXICOKING units.

Recent reviews of the coke handling facilities resulted in a more realistic design for these facilities. The new design introduces common silos for the three FLEXICOKING units. The reduction in the number and size of the coke silos has produced potential savings of about 10% on total FLEXICOKING investment.

- There is a significant cost incentive to reduce the steam rate to the FLEXICOKING reactors. The Study Design Update incorporated a steam rate of 25% steam-on-feed based upon Continuous Stirred Coking Unit (CSCU) data.

A sensitivity case has demonstrated potential saving of 3% on FLEXICOKING investment if consumption could be lowered to 15% without significant effect on yields. Future CSCU runs will be made at lower steam rates to identify yield effects. The savings are due to a smaller reactor, fractionator and overhead condenser. Possible cost reductions in the boiler and steam distribution system have not been accounted for.

- A lower entrainment rate from the gasifier, in line with observations on petroleum feedstocks, could allow savings of about 2% by allowing a smaller gasifier vessel. However, the Integrated Coker Gasifier (IKG) and other pilot unit operations have indicated that entrainment levels may be higher with coal based feedstocks. If this observation holds for commercial units, a larger vessel diameter may be necessary to maintain coke inventory. The FLEXICOKING investment would increase by up to 4% due to increased gasifier size. Operation of the FLEXICOKING Prototype Unit is needed to define the entrainment rate and gasifier size.

- The original basis for the Study Design Update provided 15 days storage of excess vacuum bottoms produced during an unscheduled FLEXICOKING outage. In addition, surge storage capacity equivalent to 10 days feed to one FLEXICOKING unit was also provided. Total storage required 12 tanks (each 75 feet in diameter).

The present basis provides only 15 days of excess vacuum bottoms storage in 5 tanks. As discussed in the October-December, 1978 Quarterly (FE-2893-25) surge capacity was believed unnecessary. The savings in eliminating the 10 days of surge capacity has been estimated at 5% of FLEXICOKING investment.

- The Illinois Coal Base Case Study Design Update provides three stages of hydroclones for solids removal from scrubber liquids. A higher scrubber liquids viscosity, increased solids loading or more stringent fuel oil particulate emissions specification may require the use of five stages of hydroclones. The additional two stages, with associated pumping and piping, would require an additional 0.5% investment.
- During the vacuum bottoms runoff operation, coke is purged from FLEXICOKING with a high carbon content due to reduced gasification level. In the Study Design Update, this coke is sent to offsites for disposal. Providing offsite storage facilities for this coke with two concrete silos (80' diameter) could enable recovery of some of this carbon during a later turndown period. These storage facilities would require another 2% of FLEXICOKING investment. An assessment is underway to determine whether the improvement in thermal efficiency justifies the incremental investment for these storage facilities.

#### 1.4 Fuel System Study - Illinois Coal Base Case

A study was carried out to determine the basis for the fuel system in the Illinois Coal Base Case of the EDS Study Design Update. The study considered variations in plant fuel requirements and fuel availability which result from various plant operations. The results of this study defined the basis to be used in designing a safe, operable fuel system for the plant. Full documentation of this study was reported in previous Quarterly Reports (refer to FE-2893-21 and FE-2893-25).

The plant utilizes 3 different fuels, as follows:

- Low Btu gas (LBG) produced by the FLEXICOKING units for all onsite requirements and in the offsite boilers
- Vaporized C<sub>3</sub> LPG used as auxiliary and backup fuel in the onsite facilities
- Coal in the offsite boilers

For environmental considerations, any excess LBG produced may be flared/vented only during emergencies. This restriction requires that enough boiler capacity be available at all times to consume the LBG produced but not utilized in the onsite facilities. Otherwise process changes must be made to reduce the excess LBG production to a manageable level. However, a minimum amount of LBG must always be used in the offsite boilers to accommodate the short-term variability in the LBG supply/demands. As a result of the various modes of operation of the plant, the inherent short-term and longer-term variability in both the fuel supply and demand, and the restrictions on flaring/venting of the fuel, the fuel system of the plant is complex.

Tests with LBG have indicated that no supplementary fuel is required to maintain combustion. However, safety concerns require that a system be provided to protect a furnace in case of flameout. Three systems were evaluated, and it was concluded that providing LBG pilots is the preferred method for the Study Design Update. Flame scanners provide a fallback.

### 1.5 Offsite Design Basis - Illinois Coal Base Case

The Offsite Design Basis for the Illinois Coal Base Case of EDS Commercial Plant Study Design Update was completed. The offsite facilities include all utilities, the fuel system, product and intermediate stream storage facilities, product shipping, coal transportation and preparation, wastewater treating, waste disposal facilities, and facilities for personnel protection and safe operation of the plant. In keeping with the philosophy used for all onsite units, the offsite facilities were to avoid major step-outs in technology in peripheral areas. As in the onsite units, a two-train concept was to be used, as necessary, to ensure that a complete plant shutdown is not required for scheduled turnarounds.

The Offsite Design Basis, including the basis for the safety system will be issued as an interim report during the 3rd quarter of 1979. The Offsite Design Basis has also been covered in more detail in previous reports (refer to FE-2893-21 and FE-2893-25).

### 1.6 Offsites Design - Illinois Coal Base Case

The offsite design for the Illinois Coal Base Case of the EDS Study Design Update was completed. The offsite facilities include the following:

- Coal Storage and Handling
- Utilities
  - + Raw Water/Boiler Feedwater Treating
  - + Steam Generation and Distribution
  - + Plant Fuel System
  - + Cooling Water System
  - + Inert Gas/N<sub>2</sub> Systems
  - + Compressed Air System
  - + Electric Power Distribution
- Tankage, Interconnecting Lines and Product Shipping
- Chemical Storage and Handling
- Waste Solids Handling
- Wastewater Treating
- Safety and Fire Protection
- Miscellaneous (Buildings, Communications, Rail Facilities, etc.)

Design information for these facilities were released to cost estimating. The design information for each of the offsite sections reflects the most restrictive operating mode of the plant.

Descriptions of most of these facilities have appeared in previous reports (refer to FE-2893-25 and FE-2893-29). Final documentation is underway, and will be included in the Illinois Coal Study Design Update interim report.

#### 1.7 Critical Issues and Assumptions - Illinois Coal Base Case

Work has begun toward compiling a list of critical issues and assumptions for the SDU Base Case. Sensitivity of the plant economics to these issues and assumptions will be determined. In general, this list will contain basis items which were assumed or which were a result of previous screening studies. The intention is to re-examine these bases in view of the updated plant costs.

Present efforts have concentrated on compiling the list while some work has been started in evaluating the issues and assumptions.

### 1.8 Plant Thermal Efficiency - Illinois Coal Base Case

Thermal efficiency was calculated for the Illinois Coal Base Case of the EDS Study Design Update (SDU). An overall plant thermal efficiency was determined by combining individual efficiencies for six modes of plant operation, each at start-of-run and end-of-run conditions. The individual efficiencies were based on the higher heating values for all feed, product, byproduct, and waste streams and with electric power valued at 8,500 Btu/Kw-Hr. Additionally, stream day and calendar day product rates for the overall plant operation were calculated on a weight, heat, and volume basis.

An overall thermal efficiency of 56% resulted for the Illinois Coal Base Case SDU. The total C<sub>3</sub> liquid product rate was 55,380 B/CD or 51,260 FOEB/CD (one FOEB equals 6.415 MBtu), considering all the modes of operation. The higher heating value of the total C<sub>5</sub>+ liquid products was 19,960 Btu/lb compared to a value of 18,340 Btu/lb in the 1975/76 Study Design. This increase in heating value reflected the higher hydrogen consumption for the update effort which appears in the liquid products and upgrades their value. The weight recovery of C<sub>5</sub>+ liquid products for the update effort is 40.53 lb/100 dry coal versus 42.93 lb/100 dry coal for the 1975/76 Study Design. When this yield debit was combined with the heating value credit, the C<sub>5</sub>+ liquid product recovery for the update design (expressed as Btu/100 Btu dry coal feed) was essentially equivalent to that for the 1975/76 Study Design.

The plant capacity factor, expressed as the ratio of calendar day process coal feed to normal stream day process coal feed, is 86.8%. Similarly, a product recovery factor can be developed which shows that the calendar day weight recovery of C<sub>3</sub>+ liquid products is 85.3% of the normal stream day recovery. The differences between these factors reflects the consumption during FLEXICOKING unit outages and during startups.

Several factors contribute to the lower efficiency for the SDU when compared to the 1975/76 Study Design. Electric power consumption has increased in the offsite area, reflecting the increased facility definition, and in the hydrogen generation and recovery area, reflecting the 20% increase in hydrogen demand. The FLEXICOKING unit air blowers are now steam driven, resulting in a savings in electric power but increasing the need for offsite steam production and coal feed to the offsite boilers. Recovery of C<sub>3</sub> LPG as a product is decreased, reflecting consumption of C<sub>3</sub> as steam reformer feed to meet the increased hydrogen demand and consumption as backup fuel in the steam reformer furnaces during periods of FLEXICOKING outages and startups. Recovery of C<sub>4</sub> LPG is also decreased, mainly reflecting lower yields from liquefaction and solvent hydrogenation.

Consideration of the effects of various modes of operation also lowers the thermal efficiency. For example, operations at end-of-run conditions result in a 2.2% efficiency debit over start-of-run conditions.

On a yearly basis this efficiency debit is 1.1%. In addition, the effect of the FLEXICOKING outage operation in conjunction with the vacuum bottoms runoff modes causes an 0.87% efficiency debit on a yearly basis. Design changes to minimize these debits and other debits attributable to other operational effects will be areas of possible future economic studies.

In addition to the thermal efficiency calculations, an energy balance identifying plant thermal losses for normal start-of-run operation was completed. The balance indicates rates of heat lost to air fins, cooling water, flue gas, etc. and identifies areas where potentially recoverable heat (streams with a temperature  $>250^{\circ}\text{F}$ ) exists. Areas with the largest potential for heat recovery will be considered in future designs for process efficiency improvements.

Final Documentation of the plant's thermal efficiency and thermal losses will appear in the Illinois Coal Study Design Update interim report.



### 1.9 Air Pollution Impact - Illinois Coal Base Case

The air pollution impact of the Illinois Base Case of the EDS Study Design Update (SDU) has been assessed for a representative Southwest Illinois location. An earlier assessment of the air quality impact of an EDS plant was based on the 1975/1976 EDS study. Three major changes in the EDS process occurred since this earlier assessment:

- an increase in coal feed rate from 24 to 30 k tons/day;
- a switch from a fuel combination of low Btu gas (LBG) and LSFO to a combination of LBG and coal for the offsite steam boilers; and,
- a reduction in the sulfur content of the LBG from 310 to 83 vppm.

Additionally, estimates have been made of fugitive dust emissions from the coal storage piles based on recently-developed emission factors. Also included were estimates of 24 hour average ground level concentrations of SO<sub>2</sub> downstream of the plant using the prevailing wind direction and meteorological conditions for a typical southwestern Illinois location.

Only the facilities needed to process the coal from the point of coal receipt to product storage and solid waste disposal have been considered here. The atmospheric impact of coal mining, coal preparation at the mine, and coal transport from the mine to the EDS plant site have not been considered. Also, the impact of emissions on in-plant workers' environment and emissions associated with the use of the coal liquefaction products have not been considered.

Results of this evaluation indicate that the emission rates for all the criteria pollutants significantly exceed 100 tons/ year which is the threshold to be defined as a major source. Off-setting reductions in emissions from other sources would be required to locate an EDS plant in areas classified as in nonattainment of National Air Quality Standards. This essentially excludes construction of an EDS plant in a nonattainment area. An EDS plant could be located in a Class II attainment area having meteorological conditions typical of southern Illinois. A conservative buffer zone of 10 miles between the plant and Class I areas (national parks) would ensure that Class I Prevention of Significant Deterioration (PSD) increments would not be exceeded. The impact of the EDS plant relative to PSD requirements must be determined by air dispersion modeling after a specific plant location has been selected.

Full documentation of this study will appear in the Illinois Coal Study Design Update interim report.

#### 1.10 Cost Estimate - Illinois Coal Base Case

The capital cost for the Illinois Coal Base Case of the EDS Study Design Update has been estimated at 4780M\$. The cost is for a Western Illinois location and includes escalation consistent with a 3Q87 mechanical completion of the first train and 3Q88 mechanical completion of the second train. The 1975/76 EDS Study Design cost estimate was based on a 1Q85 mechanical completion. Adjusting the current Illinois Coal Base Case estimate to a 1Q85/1Q86 (Train 1/Train 2) mechanical completion basis, results in a 4035M\$ capital cost.

The above costs include a 25% project contingency and an average process development allowance of 8.1%. The project contingency is a historically based allowance required to give an equal probability of underrun/overrun for an estimate of this quality. The contingency is intended to cover process design and estimating developments that typically occur as a project develops. The contingency does not cover changes in the project scope or basis. The process development allowance is to compensate for a historically demonstrated increase in cost estimates for new technology as the technology is developed. The amount of the allowance depends on the stage of development and was determined on a section by section basis.

The capital costs presented above exclude the site specific and expensed items that are listed below. However, these costs have been incorporated in the overall project economic analysis.

- Mine and mine development costs.
- Land (1450 acres)
- Product pipelines outside the plant battery limits.
- All costs of early planning and feasibility studies by ER&E or owner.
- All right-of-way costs associated with the pipelines and conveyors.
- External power generation plant.
- Owner's non-recurring expenditures.

Table 1-1 presents a breakdown of the Total Erected Cost (TEC) by major plant sections for both the 1975/76 Study Design and the current Illinois Coal Base Case. Table 1-2 presents the development of the total erected cost for the Base Case from the 4Q78 direct costs at location.

Table 1.3 presents a reconciliation of the current Base Case to the 1975/76 Study Design. This reconciliation shows that the current estimate is 220% of the 1975/76 Study Design. This increase reflects, a general increase in Illinois construction productivity, the use of a limited site agreement and the cost increase (labor burden) incurred in going from an Eastern (in the 1975/76 Study Design) to a Western Illinois location. New estimating methods for large job field labor overheads resulted in an 11% increase. Scope changes caused an additional increase of 51% in the plant cost. The most significant scope change was the 25% increase in coal feed rate to liquefaction, which added 19% to the cost. Design and estimating developments added a further 43%. Finally, changing the mechanical completion of the plant from 1Q85 to 3Q88 has also added 13% in escalation.

Final documentation of the Illinois Coal Base Case investment will appear in the Illinois Coal Study Design Update interim report.

TABLE 1-1

EDS STUDY DESIGN UPDATE  
ILLINOIS COAL BASE CASE  
ESTIMATED BREAKDOWN BY SECTION  
TOTAL ERECTED COST, k\$

	75/76 <sup>(1)</sup> <u>Study Design</u>	EDS/SDU <sup>(2)</sup>
<u>Onsites</u>		
100 - Liquefaction	510,550	672,400
100A - Flushing & Blowdown	-	22,600
150 - Product Distillation	51,510	79,300
200 - Solvent Hydrogenation	139,560	204,600
250 - Solvent Fractionation	12,370	15,200
300 - FLEXICOKING	370,910	825,500
400 - Cryogenic, H <sub>2</sub> Recovery	64,000	113,300
400A - CO <sub>2</sub> Compression	-	6,300
425 - H <sub>2</sub> Generation, Compression	375,280	650,400
500 - Gas & Water Treating	91,370	157,700
600 - Light Ends Recovery	16,000	23,500
Common Facilities	50,590	379,200
Total Onsites	<u>1,682,140</u>	<u>3,150,000</u>
<u>Offsites</u>		
I - Coal Handling	45,850	131,800
IIA - Raw Water/BFW Treating	18,700	120,900
IIB - Steam Generation & Distribution	32,700	306,600
IIC - Fuel System	3,180	56,100
IID - Cooling Water	11,000	43,800
IIE - Inert Gas	-	17,300
IIF - Compressed Air	3,100	8,300
IIG - Electrical Power	130,700	148,200
III - Tankage, I/C Lines, Shipping	62,210	236,100
IV - Chemical Storage & Handling	3,200	9,400
V - Waste Solids Handling	42,800	192,500
VI - Wastewater Treating	58,700	190,500
VII - Safety & Fire Protection	20,400	71,600
VIII - Miscellaneous	47,360	78,900
IX - Layout & Site Prep.	8,000	18,000
Total Offsites	<u>487,900</u>	<u>1,630,000</u>
Total	2,170,040	4,780,000

NOTES:

- (1) Includes escalation for a 1Q85 mechanical completion.
- (2) Includes escalation for a 3Q87/3Q88 (Train 1/Train 2) mechanical completion.

TABLE 1-2

EDS STUDY DESIGN UPDATE  
ILLINOIS COAL BASE CASE  
ESTIMATE SUMMARY

		WESTERN ILLINOIS (M\$)			
		<u>MATERIAL</u>	<u>LABOR</u>	<u>S/C</u>	<u>TOTAL</u>
<u>DIRECT COSTS</u>					
•	Onsites	598	187	233	1,018
•	Offsites	<u>168</u>	<u>113</u>	<u>240</u>	<u>521</u>
	SUBTOTAL	766	300	473	1,539
<u>INDIRECT COSTS</u>					
•	Field Labor Overheads				332
•	Burden				177
•	Contractor's Engineering				104
•	Engineering & Erection Fee				59
•	Loss on Surplus, Insurance, and Vendor Reps.				<u>23</u>
	SUBTOTAL				2,234
<u>OTHER COSTS</u>					
•	ER&E Charges				60
•	Escalation (4Q78 to 3Q88 M.C.)				1,295
•	Project Contingency (25%)				898
•	Process Development Allowance				<u>293</u>
	TOTAL ERECTED COST "MC 3Q88"				4,780 M\$

TABLE 1-3

EDS STUDY DESIGN UPDATE  
ILLINOIS COAL BASE CASE  
ESTIMATE RECONCILIATION

	<u>TEC M\$</u>	<u>% 75/76</u>
<u>STUDY DESIGN</u>		
● 75/76 Estimate, 1Q85 M.C., Rep, U.S.	2,170	100
● Economic Basis Changes, 1Q85 W. Illinois	-	-
● Indirect Cost Method Changes	<u>230</u>	<u>11</u>
REVISSED SD, 1Q85 M.C., W. Illinois	2,400	111
<u>STUDY DESIGN UPDATE</u>		
● Scope Changes		
+ 25% Increase In Coal Feed	410	19
+ Add New Process Sections	90	4
+ Add Coal and Water Delivery Systems	140	6
+ Other	480	22
● Design Developments	590	27
● Design/Estimating Developments	240	11
● Estimating Developments	110	5
● Job Size Impact On Indirects	170	8
● Added Escalation to 3Q88 M.C. (1st Train 3Q87)	280	13
● Change In Process Development Allowance	(70)	(3)
● Reconciliation Net-by-Difference	<u>(60)</u>	<u>(3)</u>
TOTAL SDU 3Q88 M.C., W. ILLINOIS	4,780	220

### 1.11 Preliminary Economics - Illinois Coal Base Case

Preliminary economics for the Illinois Coal Study Design Update Base Case and estimates of the Market Sensitivity Case have been completed. The economics were developed for both third quarter 1987 startup (SDU Basis) and first quarter 1985 startup (for comparison with the 1975/76 Study Design). The total erected cost (TEC) for a plant with the Base Case configuration (once-through FLEXICOKING/steam reforming) is estimated to be 3050 M\$ (instant plant, 4Q 1978\$) including an 8.1% process development allowance (PDA) and 25% project contingency. The estimated project cost for a 1Q 1985 startup is 4035 M\$, and 4780 M\$ for a 3Q 1987 startup. Based on Process Alternatives Model (PAM) screening studies, the Market Sensitivity Case (once-through FLEXICOKING/partial oxidation) is currently assumed to be nominally 5% less than the Base Case, or 4540 M\$ for 3Q87 startup and 3830 M\$ for 1Q85 startup. The actual investment estimate for the Market Sensitivity Case will be available later this year.

#### Economic Basis Set from Public Sources

Table 1-4 presents the final economic basis for the Study Design Update as presented to the Overall EDS Advisory Subcommittee in early June. These bases do not represent a forecast by Exxon Corporation or any of its affiliates. The economic factors were selected from public sources as a reasonable basis for reference cost calculations of EDS coal liquids. Major basis items include a 30/31 year project life (due to one-year staging of the construction of the two trains), 48% tax rate, 100% equity financing, 13 year sum-of-the-years-digit depreciation, and a 20% investment tax credit taken in the year of expenditure. Production, investment expenditure, and startup expense schedules all reflect the staged startup.

The required initial selling price (RISP) for coal liquids is calculated using a current dollar cash flow analysis. For this analysis, product values are assumed to escalate at 9% per year for the first half of the project life and at 7 1/2% per year thereafter. Coal and operating costs are assumed to escalate at 6% per year. Sensitivities will be performed on major basis items.

Table 1-5 presents the major economic bases differences between the Study Design Update and the 1975/1976 Study Design. The major differences include raising the investment tax credit from 7% to 20%, increasing the product value escalation rate from 6% to 9/7.5% per year and the coal/operating cost escalation rates from 5% to 6% per year, and lengthening the project life from 25 to a staged 30/31 years.

TABLE 1-4

EDS COMMERCIAL PLANT STUDY DESIGN UPDATEILLINOIS COAL BASE CASE - ECONOMIC BASES

<u>Project Factors General Bases</u>	<u>Basis</u>	<u>Comments</u>
● Startup Year	3Q 1987	First train startup.
● Project Life	31/30 years	
● Capacity Factor	Coal Feed Rate & Product Recovery Factors	Annual production/(Normal stream day production x365).
● Construction		
- No. of Trains	Two	
- Staging	One Year	Second train to startup one year after first train, 3Q 1988.
- Plant Location	Western Illinois	
- Labor Market	See comments	Impact of St. Louis labor union effects, closed shop, field labor overhead, productivity
- Large Job Effect	See comments	has been developed by ER&E and is included in the project cost estimate.

Capital Expenditures

● Land	
- Lead time for expenditures for land acquisition prior to mechanical completion.	7 years
- Land Cost for plant site	
+ 3Q 1978, \$/Acre	2500



TABLE 1-4 (Cont'd)

<u>Capital Expenditures (cont'd)</u>	<u>Basis</u>	<u>Comments</u>
+ Escalation to year of purchase, %	8	
- Pipeline (Water) Right of Way Cost		
+ 3Q 1978, \$/Mile	26400	
+ Escalation to year of purchase, %	8	
- Conveyor Right of Way Cost		
+ 3Q 1978, \$/Mile	42200	
+ Escalation to year of purchase	8	
● Plant Investment Expenditure Schedule		
- Years from Mechanical Completion (1)	<u>% of TEC</u>	
-4	4.5	
-3	9.5	
-2	18.5	
-1	25.0	
0	23.5	
+1	14.5	
+2	4.5	
● Investment Escalation		
- 3Q 1978 to 3Q 1987	7	
- After 3Q 1987	7	
● Working Capital, % TEC	4.5	Taken in year 1. Recovered in year 31. Estimated from components making up working capital.

Note: (1) Mechanical Completion/Startup at end of year 0 for first train. Mechanical Completion/Startup at end of year 1 for second train. Production begins in year 1 for first train.

TABLE 1-4 (Cont'd)

	<u>Basis</u>	<u>Comments</u>
Non Recurring Expenditures, % TEC (in year of expenditure)	4.5	
• Expense Schedule, years from Mechanical Completion (1)	<u>% of Non-Recurring Expense</u>	• Startup Costs
-1	5	- Cost of labor (contractor, subcontractor or affiliate loan) for owner-supervised pre-startup operations.
0	15	- Loan startup personnel - salaries, wages, benefits, travel, moving, resettlement, etc.
+1	45	- Startup consumables
+2	35	• Preoperating Personnel Costs
		• Construction Period Costs
		• Licensing Fees (excluding EDS and FLEXICOKING)
		• Miscellaneous Others - Property tax prior to startup

<u>Plant Production Schedule</u>	<u>% of Yearly Capacity</u>
----------------------------------	-----------------------------

• Years from Mechanical Completion (1)	
0	0
+1	25
+2	81.25
+3	100.0

Taxes

• Income Taxes	48% of Gross Profit	Federal - 46%, State and Local - 4%, but re- duces to about 2% when considering Federal Income Taxes.
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Notes: (1) Mechanical Completion/Startup at end of year 0 for first train. Mechanical Completion/Startup at end of year 1 for second train. Production begins in year 1 for first train.

TABLE 1-4 (Cont'd)

<u>Taxes (cont'd)</u>	<u>Basis</u>	<u>Comments</u>
● Investment Tax Credit	20% <sup>a</sup>	In year of expenditure.
● Depreciation		
- Method	Sum of Years Digits (SYD)	
- Life	13 years	
● Property Tax Prior to Startup	1.7% of Land Cost & Improvements	Included in Non Recurring Expenditures
<u>Grants and Incentives</u>		ER&E will evaluate a number of possible alternatives.
<u>Financial Factors</u>		
● Rate of Return	15% DCF (current dollars).	Assumed Cost of Capital. Alternative methods of financing to be considered.
● Rate of General Inflation	6%/yr	Pertains to salaries, chemicals, utilities, etc.
<u>Cleaned Coal Cost, \$/ST FOB Mine</u>		
● Present cost 3Q 1978	22	Levels selected as basis from range of data available in public or literature source(s). Sensitivities will be examined. In 1975/76 Study Design 5%/yr escalation was assumed.
● Escalation rate to 3Q 1987	6	
● Escalation rate after 1987	6	
<u>Byproduct Values</u>		
● Byproducts, 3Q 1978		Level selected as basis from data available in public or literature(s). Sensitivities will be examined.
- Sulfur	51 \$/LT	
- Ammonia, Anhydrous	157 \$/ST	

TABLE 1-4 (Cont'd)

<u>Byproduct Values (cont'd)</u>	<u>Basis</u>	<u>Comments</u>
● Escalation rate to 3Q 1987	S-6%/yr, NH <sub>3</sub> 9%/yr	
● Escalation rate after 3Q87	S-6%/yr, NH <sub>3</sub> 9%/yr	
● Escalation rate after 3Q2002	S-6%/yr, NH <sub>3</sub> 7 1/2%/yr	
<u>Operating Costs (Excluding Manning Costs)</u>		
● Catalysts and Chemicals		
- Amount of each catalyst and chemical	Available	List will be issued with documentation.
- Present cost of each 3Q 1978	\$12 M/yr	
- Escalation of each 3Q 1978 to 3Q 1987	6%/yr	
- Escalation of each after 3Q 1987	6%/yr	
● Utilities		
- Purchased power		Level selected as basis from data available in public or literature(s). Sensitivities will be examined.
+ Present cost 3Q 1978, ¢/kWhr	3	
+ Escalation rate 3Q 1978 to 1987	6%/yr	
+ Escalation rate thereafter	6%/yr	

TABLE 1-4 (Cont'd)

<u>Operating Costs (Excluding Manning Costs)</u>	<u>Basis</u>	<u>Comments</u>
● Investment Related Costs, % of TEC	3Q 1978	
- Repair Material	2.1%/yr	
+ Escalation rate after 3Q 1987	6%/yr	
- Local Taxes	1.7%/yr	
+ Escalation rate after 3Q 1987	6%/yr	
● Insurance, % of Investment	0.04%	Limits - Deductible 2% of plant value. Upper limit of 20% of plant value.
- Escalation rate after 3Q 1987	6%/yr	
<u>Personnel Costs</u>		
● Salaries and Related Costs		Level selected as basis from range of data available from public or literature references. Sensitivites will be studied.
- Present costs, 3Q 1978		
+ Wage Earner, \$/man-year	20,000	
+ Salaried Personnel, \$/man-year	27,000	
+ Benefits, % of Salaries & Wages	32 1/2	
+ Overhead, supplies, etc. % of salaries and wages	20	
- Escalation of each 3Q 1978 to 1987	6%/yr	
- Escalation of each thereafter	6%/yr	

TABLE 1-4 (Cont'd)

Personnel Costs (cont'd)

Comments

● Operating and Maintenance  
Personnel Requirements

	<u>Wage</u>	<u>Management Professional Technical</u>	<u>Contract Maintenance</u>
Process	287	47	
Mechanical	699	106	
Administrative	85	116	
Total	1071	269	317

Based on most recent analysis.

Total = 1657

Pre-Operating Personnel Costs

Included in Non-Recurring Expense.

- Salary, Wages, Benefits of Staff
  - Project Coordination
  - Design/Project Services
  - Permanent Manning
- Employee/Public Relation Cost
- Training
- Manpower/Services Burden

Construction Period Costs

Included in Non-Recurring Expense.

- Temporary Rentals
- Owner's Office Operating Costs
- Services, Fees and Travel

TABLE 1-4 (Cont'd)

<u>Petroleum Product Values</u>	<u>Basis</u>	<u>Comments</u>
● Present Cost Lt Arab crude 3Q 1978 (2)	\$14.50	Crude price from National Energy Policy II used as bases.
- C <sub>1</sub> , C <sub>2</sub> 's	?	} Will be developed later for SDU.
- Gasoline	?	
- C <sub>3</sub> 's	?	
- Distillate	?	
- LSFO	?	
● Escalation 3Q 1978 to 3Q 1987	9	Escalation from Medium Case in National Energy Policy II used with some adjust- ment to smooth out escalation, and allow further escalation after 2002 at lower rate.
● Escalation after 3Q 1987	9	
● Escalation after 3Q 2002	7-1/2	
● Cost of Transportation to Illinois		To be developed.
- 1978	?	
- Escalation to 3Q 1987	7	
- Escalation after 3Q 1987	7	

Note: (2) U. S. Gulf Coast

TABLE 1-5

EDS COMMERCIAL PLANT STUDY DESIGN UPDATE  
ILLINOIS COAL BASE CASE - PRELIMINARY ECONOMICS

MAJOR ECONOMIC DIFFERENCES: 75/76 SD VERSUS SDU BASIS

<u>Selected Basis Parameter</u>	<u>1975/1976 Study Design</u>	<u>SDU Economic Basis</u>	<u>Current SDU Sensitivities</u> (1)
● Investment Tax Credit, %	7	20	10
● Product Value Escalation Rate, %	6	9/7.5	
● Op Cost/Coal Escalation Rate, %	5	6	
● Differential Escalation Rate (Product Value-Coal), %	1	3/1.5	0-5
● Project Life, years	25	30/31	
● Plant Startup	1Q85	3Q87/88	1Q85/86
● DCF Return, Current \$, %	15	15	

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Note: (1) Sensitivities examined to date. Additional sensitivities to be developed.



### Cost of Coal Liquids

Figure 1-1 shows the effects of product value escalation on RISP for EDS coal liquids. RISP is plotted versus effective annual differential escalation, or the difference between the product value escalation rate and the coal/operating costs escalation rate. The coal/operating cost escalation rate is assumed to be 6% per year. RISP's are in 1985\$ and assume 15% current \$ DCF return. When the differential escalation rate varies from 0% to 5%, RISP ranges from about 61 \$/B in the Base Case and an estimated 49 \$/B in the Market Sensitivity Case to 36 \$/B and 29 \$/B respectively. In the Market Sensitivity Case, C<sub>2</sub>- gas is priced in Btu parity with the C<sub>3</sub>+ liquids.

The effective annual differential escalation rate corresponding to the SDU economic base of 3/1.5% is represented on Figure 1-1 by a weighted average value of 2.7%. For that scenario, the required initial selling price of coal liquids for the 1Q85 plant startup is 46.85 \$/B (1985\$) for the Base Case and estimated to be 37.25 \$/B (1985\$), for the Market Sensitivity Case. The corresponding RISP's in 1987\$ for a 3Q87 plant startup are about 17% higher than for 1Q85 startup.

Table 1-6 is a 1985\$ comparison of the SDU Base Case and the estimated Market Sensitivity Case versus the 1975/76 Study Design. The SDU cases are those described above and reflect 3%/1.5% product value escalation over coal (at 6% escalation per year). The 1975/76 Study Design assumed 6% product escalation and coal/operating costs at 5% escalation per year (or a 1% differential). Shown is the impact of various costs, such as coal and capital charges, on the RISP. Also included in this table are sensitivities which show the effect on RISP of excluding Process Development Allowance (PDA) and project contingency in the total erected cost and also reducing the investment tax credit from 20% to 10%.

Table 1-7 presents a preliminary economic reconciliation. Starting with the 1975/76 Study Design the cumulative effect in RISP of each major difference to the new Study Design Update is shown in \$/B. The 1975/76 Study Design (41 \$/B, 1985\$) is adjusted first to the SDU Base Case and then to the estimated Market Sensitivity Case. The largest effects on RISP are the increased investment (2170 M\$ for the 1975/1976 Study Design to 4035 M\$ in the SDU Base Case) and the new economic bases (including the 3/1.5% product value differential escalation versus coal).

Figure 1-1

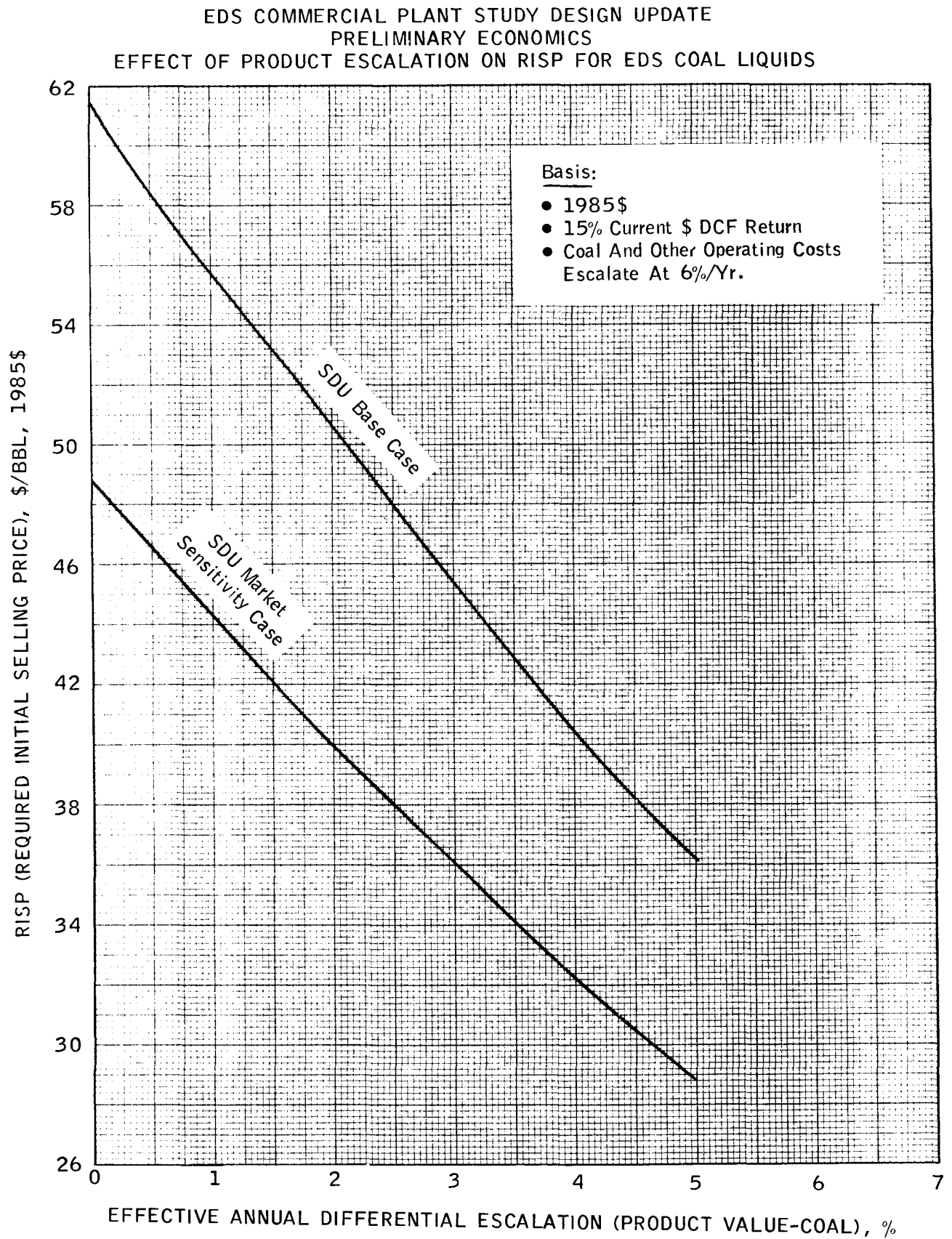


TABLE 1-6

EDS COMMERCIAL PLANT STUDY DESIGN UPDATE  
ECONOMIC COMPARISON OF SDU BASE CASE AND MARKET SENSITIVITY  
VERSUS 1975/1976 STUDY DESIGN

	1975/1976 Study Design		SDU Base Case		SDU Market Sensitivity	
Investment, M\$(1)	2170		4030		3830(2)	
10 <sup>12</sup> Btu/yr Product	96		120		144	
MB/yr C <sub>3</sub> + Product	17.5		20.2		20.7	
Effective Δ Escalation Rate (Product Value-Coal) (3)	1%		2.7%		2.7%	
<u>Costs, 1985\$</u>	<u>M\$/yr</u>	<u>\$/MBtu</u>	<u>M\$/yr</u>	<u>\$/MBtu</u>	<u>M\$/yr</u>	<u>\$/MBtu</u>
Coal	195	2.02	316	2.63	335	2.32
Byproducts						
Sulfur	(16)	(0.17)	(22)	(0.18)	(23)	(0.16)
Ammonia	(8)	(0.09)	(18)	(0.15)	(11)	(0.08)
Utilities						
Power	42	0.45	85	0.71	73	0.51
Water	1	0.01	-	-	-	-
Catalyst/Chemicals/Waste Disposal	9	0.09	23	0.19	22	0.15
Salaries & Related Costs	47	0.48	82	0.68	82	0.57
Inv. Related Op. Costs	72	0.74	155	1.29	147	1.02
Capital Charges (15% Current\$ DCF Return)	374	3.87	326	2.73	303	2.07
Total	716		947		928	
RISP, \$/MBtu		7.40		7.90		6.40
\$/B C <sub>3</sub> + Product(4)		40.91		46.85		37.25
<u>Sensitivities</u>						
- Without PDA or Contingency						
\$/MBtu		6.05		6.65		5.45
\$/B C <sub>3</sub> + Product		33.50		39.50		31.55
- 10% Inv. Tax Credit						
\$/MBtu		-		8.45		6.90
\$/B C <sub>3</sub> + Product		-		50.30		39.90

Notes:

- (1) Investment for 1Q85 plant startup obtained by deflating investment for 3Q87 plant startup (4780 M\$/yr) by 7%/yr. RISP (1987\$) for 3Q87 plant startup about 17% higher than for 1Q85 startup.
- (2) Preliminary - based on PAM screening studies investment for Market Sensitivity Case is estimated as 5% less than corresponding Base Case.
- (3) 1975/1976 Study Design Basis: 6% product escalation; coal/operating costs at 5% escalation per year. SDU Economic Basis of 3%/1.5% product value escalation delta over coal (with 6% escalation per year).
- (4) Includes C<sub>2</sub>- gas sold in Btu parity with C<sub>3</sub>+ liquids.

TABLE 1-7

EDS COMMERCIAL PLANT STUDY DESIGN UPDATE  
ILLINOIS COAL BASE CASE  
PRELIMINARY RECONCILIATION OF ECONOMICS

	\$/Bbl <u>C<sub>3</sub>+, 1985</u>	Investment <u>M\$</u>	<u>C<sub>3</sub>+</u> <u>MB/yr</u>	<u>Comments</u>
• <u>1975/1976 EDS Study Design</u>	41	2170	17.46	1975/76 Study Design Basis
• <u>Adjust 75/76 SD to SDU Base Case</u>				
- Updated forecast coal/power/manning +3		2170	17.46	Increased unit costs of coal, power & manning
- Increased investment +23		4030	17.46	
+ Larger plant size +4				25% increase in coal thruput
+ Updated Cost Est. Methods & Proj. Mgmt. Effects +6				Large job size effect, Western Illinois location, etc.
+ Scope/Basis Changes +7				Increased tankage, liqn. residence time, 3 FXC, etc.
+ Process/Design Changes +6				Increased wastewater treating, system steam pressure, etc.
- Larger Plant Size/Op Costs +7		4030	17.46	Increased coal rate, power, manning for larger SDU plant.
- New economic basis (20)		4030	17.46	20% ITC, 3/1.5% differential escalation, staging, etc.
- SDU Yields on SDU plant size (7)		4030	20.21	SDU product rate.
	47			
• <u>Adjust to SDU Market Sensitivity Case</u>				
- Reduced investment (1)		3830	20.21	5% estimated lower TEC based on PAM screening studies.
- Op cost effects ~ 0		3830	20.21	Reflects increased coal requirement, lower power, etc.
- Increased yield effect (9)		3830	20.69	Includes C <sub>2</sub> - gas sales in Btu parity with C <sub>3</sub> + liquids
	37			

EDS Study Design Update - Illinois  
Coal Market Flexibility Sensitivity Case

1.12 Onsite Design Basis - Illinois Coal Market Flexibility  
Sensitivity Case

The onsite design basis for the Illinois Coal Market Flexibility Sensitivity Case was completed. This case is a sensitivity to the Illinois Base Case. The sensitivity case produces hydrogen by partial oxidation of vacuum bottoms instead of steam reforming of methane, ethane and propane (as in the Base Case). The methane and ethane released by elimination of steam reforming in the sensitivity case will be sold as high Btu (HBG) gas to an industrial user. HBG will also be used as backup fuel (instead of C<sub>3</sub> LPG). Oxygen for the partial oxidation units is provided by oxygen plants through cryogenic fractionation of air.

Slurry drying, liquefaction, solvent hydrogenation and solvent fractionation sections are essentially the same as in the Illinois Coal Base Case. The liquefaction product distillation facilities are also similar to the Illinois Coal Base Case. The design of both atmospheric pipestills (APS) units have not changed from the Base Case. The vacuum pipestill (VPS) upstream of the FLEXICOKING units has also not changed from the Base Case (~920°F cutpoint); however, the VPS upstream of the partial oxidation units will be designed for a 975°F cutpoint. By cutting deeper in the bottoms stream, liquids which would otherwise be destroyed in the partial oxidation units are recovered as vacuum gas oil (VGO), and sent to the fuel oil pool. In FLEXICOKING, most of the 920/975 FVT liquids present in its vacuum bottoms feed are recovered in the reactor-scrubber overhead. Several modifications were also made to the FLEXICOKING units. Since only about 50% of the vacuum bottoms are processed in FLEXICOKING, only two units are needed to provide sufficient fuel for all the onsites needs and maintain the required spillover to offsites. The recycle conversion of coker scrubber liquids was changed to 15%, (compared with 30% in the Base Case). This change was possible since plant needs for LBG fuel are not as tight as in the Base Case. Also, since overall scrubber liquids recovery rate is reduced, the solids level could be increased while maintaining the same quality fuel oil product from the plant.

Full documentation of the Onsites Design Basis for the Illinois Coal Market Flexibility Sensitivity Case will appear as an interim report.

### 1.13 Onsites Design - Illinois Coal Market Flexibility Sensitivity Case

Work was completed on the onsite design for the Illinois Coal Market Sensitivity Case. Work concentrated on designing the sections with major changes from the Base Case, as follows:

- Product Fractionation (section 150)
- FLEXICOKING (section 300)
- Cryogenic Hydrogen Recovery (section 400)
- Partial Oxidation, Synthesis Gas Upgrading and Hydrogen Compression (section 425)
- Oxygen Manufacture

Heat and material balances, flowschemes, equipment design, and utility estimates were developed for all these sections. The remaining sections of the plant required only minor modifications.

Design information packages for the section which changed were released to cost estimating. These packages included equipment pieces and flow diagrams. In addition to the sections with only minor modifications, cost estimating was informed of the relevant changes. All the design information reflects the effect of end-of-run operating conditions in solvent hydrogenation.

Descriptions of these facilities have appeared in previous reports (refer to FE-2893-29). Remaining efforts are in completing final documentation of the onsite design which will be included in the Illinois Coal Study Design Update interim report.

#### 1.14 Offsite Design Basis - Illinois Coal Market Flexibility Sensitivity Case

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The offsite design basis for the Illinois Coal Market Flexibility Sensitivity Case has been completed. This case is a sensitivity case to evaluate the use of partial oxidation to produce H<sub>2</sub> and sell C<sub>2</sub>- high Btu gas (HBG) which had been processed in steam reforming in the Base Case.

The offsite design of the Market Sensitivity Case will be in considerably less depth and detail than for the Illinois Base Case. Wherever possible, the Base Case design will be used to develop the offsite design for the Market Sensitivity Case. The offsite design effort will be concentrated in the following areas:

- Fuel, steam, and power systems
- Water systems
- Transport and disposal of solid wastes

A major change is that for the Sensitivity Case, the C<sub>2</sub>- HBG will be sold via a pipeline. HBG will serve as onsite backup fuel for the plant. HBG will also serve as startup and emergency fuel for the offsite area.

There will be minimum design effort (i.e., hold constant, prorate, or use judgement of the Base Case) on the remainder of the offsites, such as the following:

- Plot Plan
- Fire Protection
- Safety Facilities
- Buildings
- Coal Receipt, Storage, and Preparation
- Catalyst and Chemicals Receipt and Storage
- Waste Treatment and Disposal
- Oil Movement and Storage
- Product Shipment
- Air Compression

Full documentation of the Offsite Design Basis - Illinois Coal Market Flexibility Sensitivity Case will appear as an interim report.

#### 1.15 Offsite Design - Illinois Coal Market Flexibility Sensitivity Cases

The design of the offsite facilities for the Illinois Coal Market Flexibility Sensitivity Case has been completed. The offsite design was carried out in considerably less depth and detail than for the Illinois Coal Base Case. Wherever possible, the Illinois Coal Base Case design was used to develop the offsite design for the Market Sensitivity Case. A description of some of the offsite facilities follows:

- Coal Storage and Handling

The capacity of the coal storage and handling equipment was increased 4% over the Base Case due to increased coal feed to the boilers. Equipment serving only the process area has not changed. The equipment serving only the boiler pulverizers has increased by 55% (2,280 t/d to 3,550 t/d).

- Raw Water/Boiler Feedwater Treating

The amount of raw water required in the Market Flexibility Sensitivity Case is 5% less than that required in the Base Case. However, the split between the major consumption areas has changed significantly. The process deaerator requirements have fallen by 70%, the offsites deaerator demands have more than doubled, and the process consumption has fallen by 33%. The softening, filtration, sludge dewatering, and cooling tower feed systems have not changed significantly.

- Steam Generation and Distribution

The offsite steam generation and distribution system for the Market Flexibility Sensitivity Case differs from the Base Case in that steam is produced only to meet the net plant requirements. Excess steam generation is not required in the Sensitivity Case since the spillover of LBG to offsites provides less than the total boiler fuel requirement. This is a direct result of increased process steam demands and a reduction in total FLEXICOKING capacity. The total offsite boiler capacity has increased approximately 55 percent in the Market Sensitivity Case.

Offsite steam generation is at the same level as the Base Case (1250 psig). The 1250 psig steam is reduced to the 600 psig and 125 psig levels, where demand exists, through the use of back pressure and extraction/back pressure turbines. These turbines are onsite drivers for the FLEXICOKING units air blowers (2), the oxygen compressors (3), and the offgas compressors (2).

- Plant Fuel System

The plant fuel system for the Market Flexibility Sensitivity Case is similar in concept to the Base Case. The two major differences in the fuel system between the cases are as follows:



- + The amount of low Btu gas (LBG) produced in the Market Sensitivity Case is about 50% of the Base Case production.
- + The Market Sensitivity Case utilizes C<sub>2</sub>- high Btu gas (HBG) instead of C<sub>3</sub> LPG (in the Base Case) for startup and backup fuel.

Low Btu Gas is the normal fuel for all onsite fuel consumers (VPS furnace, slurry furnaces, sulfur plant, solvent hydrogenation and product recovery). LBG is also used as boiler fuel and as pilot fuel for the offsite safety system flares. LBG is available at 20 psig minimum after cleanup. A vent stack on the main LBG header is specified to release LBG to the atmosphere to prevent overpressure of the LBG system during an emergency.

The fuel balance requires that varying amounts of LBG be sent to the offsite boilers because of varying process fuel usage and production during different operating modes. The LBG sent to offsite boilers is burned with coal to provide plant steam requirements. The coal firing rate will be varied to keep the boiler steam output constant as the available LBG varies. A gas holder will smooth out the rapid fluctuations in LBG sent to the offsite boilers and provide response time for coal feed adjustment.

High Btu Gas plant fuel is provided as startup and backup fuel for the slurry furnaces, and as startup and emergency fuel for the offsite boilers. HBG is also used as fuel for two small offsite fuel consumers. All HBG is available from the H<sub>2</sub> recovery area at 60 psig for normal operation and from the industrial gas pipeline for startup and emergency.

Piping is included to use some H<sub>2</sub> recovery offgas and purge gas in the offsite boilers during a H<sub>2</sub> recovery compressor outage. In the Base Case, the offgas and purge gas was sent during outages to the steam reformers as feed. When more offgas and purge gas is produced than the boilers can use, the excess is sent to the safety system.

A fuel system simulation study would be required to guarantee the operability of the LBG/coal/HBG fuel system.

- Cooling Water System

The cooling water system for the Market Flexibility Sensitivity Case is similar to the Base Case system. The major changes from the Base Case are that the process cooling water requirement has increased to 93,000 gpm and the FLEXICOKING unit air blower turbine condensate cooling has been eliminated.

- Inert Gas/N<sub>2</sub> Systems

The inert gas/H<sub>2</sub> system for the Market Flexibility Sensitivity Case has changed substantially from the Base Case. All inerting requirements for the Market Sensitivity Case are satisfied with nitrogen from the process area. Offsite N<sub>2</sub> generation is not required.

Nitrogen is available as a waste stream from the process oxygen plants at 1 psig. The N<sub>2</sub> is compressed and distributed at 150 psig by offsite equipment.

For a situation when N<sub>2</sub> is required and the oxygen plants are not operating, a backup N<sub>2</sub> system is provided. The backup N<sub>2</sub> system consists of a refrigerated storage sphere for liquid N<sub>2</sub> and a steam heated vaporizer system. The storage sphere is sized to provide the slurry driers with N<sub>2</sub> for 24 hours. The tank will be filled by truck import. The vaporizer capacity is adequate to supply the maximum simultaneous N<sub>2</sub> demand.

- Compressed Air System

The compressed air system for the Market Flexibility Sensitivity Case is similar to the Base Case system.

- Electric Power Distribution

The electric power distribution system is similar to the Base Case with the addition of the oxygen plant air compressor drivers (three). The arrangement and sizes of the substations have been modified from that in the Base Case to reflect the revised Market Sensitivity Case loads.

- Tankage, Interconnecting Lines

The total offsite storage capacity in the Market Sensitivity Case has been reduced 6% from the Base Case. This change reflects differences in product, byproduct, and intermediate stream rates. The offsite storage capacity for C<sub>3</sub> LPG has been greatly reduced since C<sub>3</sub> LPG is not used as backup fuel in the Sensitivity Case.

The offsite storage capacity for vacuum bottoms has been slightly reduced in the Market Sensitivity Case. This corresponds to a reduced rate of excess vacuum bottoms produced during a FLEXICOKING unit outage. The size of the vacuum bottoms transfer pumps and interconnecting lines have also been changed. All other facilities associated with vacuum bottoms movements and storage are unchanged from the Base Case.

- Solid Chemicals/Catalyst Receipts

All facilities in this area are the same for the Market Sensitivity Case as for the Base Case, except for minor warehouse requirements.

- Bulk Liquid Chemical Receipts

All equipment and facilities are the same for the Market Sensitivity Case as for the Base Case except for the equipment associated with chemical and catalyst handling for hydrogen generation and FLEXICOKING.

- Waste Solids Handling

The Market Flexibility Sensitivity Case offsite boilers and the onsite FLEXICOKING units produce waste solids that are handled similarly to the methods used in the Base Case. A POX (partial oxidation) unit produces a second type of onsite waste that is handled separately and disposed of in the common disposal area.

The offsite boiler waste solids system handles 55% more material than the Base Case equipment due to the increase in boiler size. The FLEXICOKING unit waste solids system handles 40% of the Base Case FLEXICOKING solids plus an additional 2% (90 ton/day) of POX fines that are similar to FLEXICOKING unit scrubber fines.

The POX unit produces a maximum of 994 ton/day of slag (0.1-7 mm diameter). The slag slurried to the offsite solids handling area where excess water is screened off and recycled, and the solids are dumped into a silo.

The blended FLEXICOKING unit fines and the POX unit slag are disposed of in the waste disposal pile. The increased production of boiler wastes results in a 9 ft deep barrier. The 50 ft high pile covers only 510 acres instead of the original 740 acres due to the higher density of POX slag and processing changes in the FLEXICOKING area that leave less carbon on the ash.

- Wastewater Treating

The wastewater treating equipment for the Market Sensitivity Case changes only slightly from the Base Case. Due to reductions in leachate and FLEXICOKING unit waste flow, the major water-treating equipment is smaller than the Base Case, but the sludge equipment remains the same. Revised waste flows from the utilities area results in a plant effluent flow that is lower than the Base Case by 380 gpm.

- Safety and Fire Protection

The safety system for the Market Sensitivity Case is similar to the Base Case. A reduced vapor release from the onsite process units (FLEXICOKING units) in the Market Sensitivity Case is offset by an increased vapor release from the offsite fuel system. The net effect of all changes from the Base Case safety system is small and considered negligible.

The fire protection system for the Market Sensitivity Case is similar to the Base Case. Changes in plant layout do not have a major effect on the firewater equipment requirements.

Final documentation of the offsite design is underway and will be included in the Illinois Coal Study Design Update interim report.

#### 1.16 Plant Thermal Efficiency - Illinois Coal Market Flexibility Sensitivity Case

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Thermal efficiency calculations were completed for the Illinois Coal Market Flexibility Sensitivity Case. The overall plant thermal efficiency is 63.6% as compared to 55.6% for the Base Case. This increase in efficiency reflects the recovery of C<sub>2</sub>- high Btu gas (HBG) and C<sub>3</sub> LPG as products. In the Base Case, all of the C<sub>2</sub>-HBG and most of the C<sub>3</sub> LPG were consumed as feed to the steam reformers for production of hydrogen or burned as auxiliary fuel in process furnaces. In the Market Sensitivity Case, hydrogen is produced through partial oxidation of vacuum bottoms and HBG is available for use as auxiliary furnace fuel.

In the Market Sensitivity Case, the vacuum bottoms feed to the FLEXICOKING units is approximately one-half of the Base Case feed rate. As a result, FLEXICOKING liquid yields are decreased. However, this debit is partially offset by an increase in vacuum gas oil recovery due to the deeper-cut vacuum pipestill located upstream of the partial oxidation units. Overall, the C<sub>5</sub>+ liquid recovery for the Market Sensitivity Case is slightly lower than that for the Base Case. When the increased C<sub>3</sub> LPG recovery is accounted for, the Market Sensitivity case recovers slightly more C<sub>3</sub>+ liquids than the Base Case.

Byproduct recovery for the Market Sensitivity Case is slightly lower than the Base Case. Offsite coal requirements are up, reflecting an increased offsite steam demand due to the loss of onsite steam production from the deleted steam reformers and an increase in steam requirements for onsite turbine drivers for the oxygen compressors and the offgas compressors. Electric power is also up somewhat, reflecting the addition of air compressors at the oxygen plants and HBG product compressors.

Final documentation of the plants thermal efficiency will appear in the Illinois Coal Study Design Update interim report.

## EDS Study Design Update - Wyoming Coal Case

### 1.17 Onsite Design Basis - Wyoming Coal Case

The Onsite Design Basis for the Wyoming Coal Case of the EDS Study Design Update has been completed. The process configuration for the Wyoming Coal Case is to be identical to that of the Illinois Coal Market Flexibility Sensitivity Case, i.e., partial oxidation of vacuum bottoms for hydrogen production and FLEXICOKING of the remainder of the vacuum bottoms for plant fuel gas production. The plant dry coal feed rate will be 25.0 kST/SD (36.3 kST/SD of "as received" coal) which is the same as that of the Illinois Coal Cases. The slurry drying facilities are expected to be substantially larger than those of an Illinois coal plant because of the higher moisture content of Wyoming coal, roughly 30% vs 17%.

The liquefaction conditions that were selected from evaluations of screening studies and laboratory data results are:

Reactor temperature	840°F
Residence time	60 min.
Solvent-to-coal wt. ratio	1.6

The longer residence time and higher solvent-to-coal ratio compared to Illinois No. 6 coal are based on laboratory data. This indicates the need for higher severity for the liquefaction for Wyoming coal. In addition, data indicate that the viscosity increase of Wyoming coal liquids is more rapid than that of Illinois liquids. Therefore, the cutpoint of the vacuum bottoms feed to partial oxidation may be limited to less than the 975°F basis used for the Illinois coal liquid. The FLEXICOKING unit hydroclone underflow fractionation may also be limited by viscosity.

The basis for controlling the potential formation of calcium carbonate scale in the liquefaction reactors is the use of screen/grids and periodic withdrawal of solids. In addition, the reactors are cleaned periodically using a dilute phosphoric acid solution.

The product fuel oil sulfur content will be lower than that of an Illinois coal based plant. Thus, there will be no need to hydrotreat excess solvent blended into fuel oil. It will also not be necessary to include the 350/400°F heavy naphtha in the fuel oil to meet sulfur specifications, although this may be desirable to control viscosity of the fuel oil pool.

Gas with a low H<sub>2</sub>S content from regeneration of the liquefaction rich DEA will need to be segregated from other higher H<sub>2</sub>S gas streams. To avoid combustion problems in the sulfur plant, the low H<sub>2</sub>S content stream will be staged into the combustion chamber after combustion has been initiated on the stream with a higher H<sub>2</sub>S content.

Full documentation of the Onsite Design Basis - Wyoming Coal Case, will appear as an interim report.

### 1.18 Onsite Design - Wyoming Coal Case

Preparation of the onsites portion of the EDS Study Design Update (SDU) for Wyoming coal has begun. Initial efforts have focused on generating detailed heat and material balances for the plant to provide the basis for equipment design as well as the necessary data to determine overall plant fuel balances and the impact these have on bottoms processing configuration.

Preparation of heat and material balances has included yield development for start-of-run and end-of-run solvent hydrogenation operations. In addition, several sensitivities involving different hydrogen consumption splits between the liquefaction and solvent hydrogenation sections were investigated in an effort to successfully simulate the laboratory results from the CLPP confirmation runs. Also, an effort was made to improve the component representation of the heavier portions of the process streams. This effort was intended to improve the prediction of process conditions necessary for the deeper-cut fractionation in the vacuum bottoms tower upstream of the partial oxidation units. The work has included comparisons of laboratory Hivac-C and microlube distillations for samples of CLPP vacuum flasher feeds during operations with Wyoming coal.

Most of the equipment in the slurry drying section of the plant has been designed based on a near-final heat and material balance for that section. The slurry drier overhead system has been modified from that in the Illinois SDU to accommodate the larger amount of water (approx. 31 wt% vs 16 wt% in the Illinois SDU) and the larger amount of hydrocarbon stripped overhead. The hydrocarbon distillate from the overhead separator distillate drum is now heated up through heat exchange with the overhead vapor stream and mixed in the slurry product stream. The overhead recycle loop has thus been eliminated, reducing the size of the overhead vapor stream and the accompanying equipment.

The slurry drier temperature has been increased to 300°F from 275°F in the Illinois SDU. This increase was necessary to reduce the residence time in the reactor by approximately 40% and avoid a possible multiplicity of slurry drier vessels.

1.19 Hydrogen Facilities for the Wyoming Coal Case -  
Selection of Vacuum Pipestill Cutpoint Feeding Partial Oxidation

A study was carried out to determine the maximum feasible vacuum pipestill (VPS) cutpoint for feeding partial oxidation for hydrogen production in the Wyoming Coal Case of the EDS Study Design Update. Previous screening studies had shown an incentive to maximize liquid recovery by cutting as deeply as possible in the VPS feeding partial oxidation. Following analysis of viscosity information from CLPP operations, a maximum cutpoint of 900°F was selected for Wyoming coal processed at 840°F/60 minutes liquefaction residence time.

The major limitation in increasing cutpoint is the corresponding increase in vacuum bottoms viscosity and its effect on pump performance. A 900°F maximum cutpoint has been selected since it produces a bottoms stream whose viscosity falls within demonstrated pumping capability of about 50 poise for reciprocating pumps. Reciprocating pumps are needed for this service since screw pumps, which can handle higher viscosities, cannot provide sufficient  $\Delta P$  to move bottoms to the high pressure partial oxidation unit.

The maximum cutpoint with Wyoming coal has been reduced from the 975°F cutpoint used with Illinois (Monterey) coal due to the higher viscosity of Wyoming coal liquefaction bottoms. A discussion of the procedures used to set the Illinois basis was given in January - March, 1978 Quarterly Progress Report (FE-2893-12).

## ENGINEERING RESEARCH AND DEVELOPMENT

### PROCESS ENGINEERING AND COST EVALUATIONS

#### 2. Cost Reduction and Laboratory Guidance Studies

##### 2.1 Illinois Coal Vacuum Bottoms Recycle Evaluation

Recycle of vacuum bottoms to the liquefaction reactor has been identified as a potential improvement for the EDS Process. Experimental data had shown that liquefaction yields increase during recycle operations using Illinois coal. A preliminary engineering evaluation had indicated a potential incentive in the order of 1 \$/B (1985) for recycle operations (refer to the October-December, 1978 Quarterly Technical Progress Report, FE-2893-25). The purpose of this study was to assess the potential incentive for vacuum bottoms recycle using various plant configurations and to determine if CLPP operations in the recycle mode were warranted. Of special interest was the effect alternative bottoms and supplemental coal processes have on the incentive. This evaluation was completed with the use of the EDS Process Alternatives LP Model (PAM).

The Study Design Update (SDU) liquefaction unit was used as the base case for the study. Two levels of bottoms recycle, 20% and 40% (1b 1000°F+ bottoms/100 lb of coal plus 1000°F+ bottoms), were evaluated. In the earlier engineering evaluation, 40% bottoms recycle was found to give the maximum incentive. The 20% recycle level was selected as a less severe operation which may maximize the incentive in a fuel-short plant configuration. All these liquefaction units have a 1.2 solvent-to-coal ratio (i.e., bottoms recycle operations were assumed to need no solvent in addition to that provided in the SDU unit). The liquefaction reactor H<sub>2</sub> partial pressure of the bottoms recycle units is maintained at ~510 psig (the SDU level) by using a larger H<sub>2</sub> treat gas rate in liquefaction.

Results of the evaluation indicate that based on the available once-through bottoms recycle data, there is a potential incentive for vacuum bottoms recycle. CLPP operation on a bottoms recycle mode using Illinois coal is required to verify yields and hydrogen consumption during equilibrium operation. Such CLPP runs are now underway. The study also confirms that 40% recycle is preferred over 20%. A bottoms recycle level somewhat higher than 40% may be attractive with some configurations. The potential incentive of 1-2 \$/B (2-5% on RISP) can be achieved using ~40% bottoms recycle and various H<sub>2</sub>/fuel alternatives. This potential incentive does not include any credits for a lighter, solids-free fuel oil product usually resulting from the 40% bottoms recycle operation. The choice of configuration may ultimately depend on the coal feed and the relative stage of bottoms process development.

Complete documentation of this study appeared in the January-March, 1979 Quarterly Report (FE-2893-29).



## 2.2 EDS Wyoming Coal Liquefaction Process Variables Study

The purpose of this study was to determine the preferred temperature and residence time for operating the liquefaction section of the EDS plant with Wyoming (Wyodak) coal. Yield information for the alternative liquefaction operating conditions used in this study was provided by EPRL. The alternative liquefaction operating conditions selected for study were at 800°F, 840°F and 880°F with residence times ranging from 15 to 120 minutes. EDS Process Alternatives LP Model (PAM) vectors at these liquefaction conditions were prepared and validated. Investments were based on using equipment patterned after the 1975/1976 Wyoming Coal EDS Study Design. The investments were adjusted as required to reflect not only the new liquefaction reactor conditions but also higher gas rates and/or increased vaporization relative to the 1975/76 Study Design. Bottoms processing alternatives available were once-through FLEXICOKING for fuel production and steam reforming or partial oxidation for hydrogen production.

Results of the study showed incentives for operating at more severe liquefaction conditions than were used in the 1975/1976 Wyoming Coal Study Design. The most attractive cases used coal in a coal-fired boiler and purge gas (feed to H<sub>2</sub> cryogenic recovery unit) to close the fuel balance. The optimum plant configuration, of the alternatives studied, utilized a once-through FLEXICOKING unit to produce fuel and partial oxidation to produce hydrogen. Although liquefaction temperatures higher than that used in the 1975/76 Study Design seemed to have a marginal economic advantage, possible operability problems and less well-defined investments at the higher temperatures resulted in the selection of liquefaction at 840°F and longer residence times for the Study Design Update.

### 2.3 EDS Pittsburgh No. 8 Coal Liquefaction

#### Process Variables Engineering Screening Study

This engineering screening study was performed to evaluate the coal liquefaction economics of a plant feeding Pittsburgh No. 8 (Ireland mine) coal. Liquefaction vectors were prepared from RCLU data obtained by EPRL for the following temperature/residence time severities: 840°F/40, 60, and 100 minutes, and 800°F/100 minutes. Evaluation of the Pittsburgh No. 8 coal liquefaction vectors was accomplished using the Process Alternatives (LP) Model developed for Pittsburgh No. 8 from the Illinois (Monterey) Coal Model.

The results of that evaluation indicate that the overall preferred case is liquefaction at 840°F for 60 or 100 minutes residence time followed by once-through FLEXICOKING of vacuum bottoms for fuel and partial oxidation of vacuum bottoms for hydrogen. A comparison of this case with the optimum Illinois (Monterey) No. 6 case in this configuration (840°F/40 minutes) shows that the Pittsburgh No. 8 plant has a small potential economic advantage, yielding a net cash flow that is 40 M\$/yr (1985\$) higher than the Illinois No. 6 plant. Sensitivities performed on location and coal price assumptions can change the Pittsburgh plant's net cash flow advantage to 20 and 72 M\$/yr., respectively.

The results of this screening study show that, while conversion of Pittsburgh No. 8 is lower than that for Illinois (Monterey) No. 6 in the liquefaction section of the plant, bottoms processing response is the same for both coals, so that additional liquids are recovered from the higher bottoms feed rate, thus narrowing the yield differences. This, coupled with lower coal moisture content and lower operating costs (higher byproduct credits) gives Pittsburgh No. 8 coal a small potential economic advantage over Illinois No. 6 coal for optimum severity processing. This advantage, however, is judged to be within the range of uncertainty defined by the limited (relative to Illinois No. 6) Pittsburgh coal data base. In addition, similar process development allowances (PDA) were applied to Pittsburgh No. 8 and Illinois No. 6 coal process blocks in this study. PDA's might directionally be higher for Pittsburgh No. 8 coal due to the limited data base, which would reduce or eliminate the small potential economic advantage for Pittsburgh No. 8 coal liquefaction.

## 2.4 Big Brown Lignite Liquefaction Engineering Process Variables Screening Study

This screening study was undertaken to evaluate the coal liquefaction economics of a plant feeding Big Brown lignite, the second sponsor coal under evaluation as part of the EDS program. Liquefaction vectors were prepared for the following temperature/residence time severities: 840°F/25, 40 and 60 minutes and 800°F/100 minutes. Vectors were also prepared for the following bottoms processing options: once-through and recycle FLEXICOKING for fuel production, partial oxidation for fuel and hydrogen production, and a coal-fired boiler. Evaluation of the Big Brown lignite liquefaction vectors was accomplished using the EDS Process Alternatives LP Model developed for Big Brown from the Wyoming Coal Ash-Free Model.

The results of this evaluation indicate that the overall preferred configuration is liquefaction at 840°F with 25-60 minutes residence time followed by once-through FLEXICOKING of vacuum bottoms for fuel and partial oxidation of vacuum bottoms for hydrogen production. An all partial oxidation case with IBG sales may be competitive with once-through FLEXICOKING/partial oxidation, but is highly dependent on the availability of a market for the IBG and product pricing. As with Western subbituminous coals, Big Brown lignite experiences high bottoms viscosity in addition to calcium carbonate deposition in the liquefaction reactors. Further, the high ash level in the Big Brown bottoms might require higher carbon rejection (i.e., lower gasification level) in FLEXICOKING. Operability considerations will be critical in ultimately choosing the optimum liquefaction severity.

An economic comparison (1985\$) of Big Brown lignite with the optimal Illinois No. 6 Monterey (840°F/40 minutes) and Wyodak (840°F/60 minutes) coal cases in the best configuration (once-through FLEXICOKING/partial oxidation) shows that the Illinois No. 6 coal liquefaction plant has a possible economic advantage of about 20 M\$/yr (1985\$) over a Big Brown plant and 75 M\$/yr (1985\$) over a Wyodak plant when potential location factor differentials are included for all coals. If, hypothetically, all location effects are excluded (e.g., all cases on a common Illinois basis), Big Brown is significantly less attractive relative to Illinois and Wyodak by ~140 M\$/yr. Under the most optimistic circumstances (with a low lignite cost of 15.50 \$/T and the location factor credit), Big Brown becomes basically a standoff with Illinois coal.

Details of this study can be found in the January-March, 1979 Quarterly Technical Progress Report and the May, 1979 Monthly Technical Progress Report (FE-2893-29, 31).

## 2.5 Burning Star Coal Liquefaction Process

### Variables Engineering Screening Study

Work has begun on an engineering screening study aimed at evaluating the economics of a coal liquefaction plant feeding Burning Star coal.

#### Liquefaction Vectors Developed From Laboratory Data

Burning Star is an Illinois No. 6 seam coal, as is Monterey coal (the coal on which both the 1975/1976 Commercial Plant Study Design and the current Study Design Update are based). Burning Star coal is mined in Perry County, Illinois; it is the third sponsor coal run in the Recycle Coal Liquefaction Unit (RCLU) as part of the overall sponsor coal screening program. Data have been obtained from RCLU for the following temperature/residence time severities:

<u>Temperature, °F</u>	<u>Residence Time, Minutes</u>
800	40, 100
840	25, 40*, 100
880	25

\* at two different solvent-to-coal ratios (S/C).

The RCLU data for Burning Star coal represent averages of several yield periods obtained for each severity. At the 840°F/100 minutes severity, all yield periods show a net consumption of the 400-700°F cut, implying an operation out of solvent balance, which is not feasible on a commercial scale. As a result, EPRL was asked to furnish a hand-calculated estimate of liquefaction/solvent hydrogenation yields for 840°F/60 minutes residence time. However, that estimate also showed an out of solvent balance situation. Initially, the 840°F/25 and 40 minutes severities will be evaluated, and those results will determine the necessity for the development of other longer residence time estimates.

Liquefaction vectors have been prepared from the data on a moisture/ash-free basis, each vector representing a temperature/residence time severity. In addition, these liquefaction vectors have been developed for both normal (925°F) and deeper (975°F) cut point operation on the vacuum pipestill. This means that for deeper cut point operation, the vacuum bottoms contain less 1000°F- material (0.8 wt% by microlube), the differential 1000°F- liquids being recovered in the liquefaction section. The basis for such deeper cut-point operation with Burning Star coal is the same as for Monterey coal. This appears to be a reasonable assumption since vacuum bottoms viscosity at 40 minutes residence time is the same for both coals.

However, Burning Star vacuum bottoms at 25 minutes residence time show a higher viscosity than Monterey vacuum bottoms, and as such the assumed 975°F cut point may not be applicable at this residence time. Deeper cut point operation has been shown to have an economic incentive for configurations utilizing partial oxidation of vacuum bottoms for fuel or hydrogen, as a result of the additional revenues for the differential 1000°F- material recovered.

Investments for the Burning Star liquefaction vectors were based on investments prepared for the 1975/1976 Commercial Plant Study Design, and were modified to reflect reductions in the slurry drying sections due to a lower feed coal moisture content (9.33 wt% for Burning Star vs. 16.5 wt% for Monterey). Utilities consumptions for the liquefaction vectors were adjusted for reduced 600 psig steam consumption in slurry drying, and for higher fuel consumption in the deeper cut point vectors.

#### Liquids Recovery From Burning Star Bottoms Higher Than For Monterey Bottoms

Data on the FLUID COKING response of Burning Star vacuum bottoms in the Continuous Stirred Coking Unit (CSCU) have indicated that vacuum bottoms from liquefaction of Burning Star coal have a higher liquids recovery in the coker than Monterey vacuum bottoms. New FLEXICOKING vectors have been developed by the EDS Bottoms Processing Section for the Burning Star screening study. These vectors represent the latest CSCU yield data and the most recent FLEXICOKING investment information for operations with 840°F/40 and 880°F/25 minutes vacuum bottoms. The 840°F/40 minutes coking yields will initially be assumed for all the 800°F and 840°F cases. Additional CSCU runs on vacuum bottoms from other liquefaction severities were carried out in May and June; an analysis of the data obtained from these runs is expected in August and the vectors will be revised as necessary.

In addition to the new FLEXICOKING vectors, the Monterey partial oxidation vectors (fuel and hydrogen) have been modified to reflect the difference in bottoms compositions between Burning Star and Monterey and the assumed change in yields of fuel gas and hydrogen. While these changes have been incorporated into the Burning Star data base, no modifications have been made to the partial oxidation investments, since it is believed that partial oxidation of Burning Star vacuum bottoms would be similar in operability to partial oxidation of Monterey bottoms.

### Case Evaluation Similar to Previous Sponsor Coal Studies

To evaluate the new liquefaction and bottoms processing vectors, a Process Alternatives LP Model (PAM) Burning Star coal model has been developed based on the Monterey coal model, but was formulated on a moisture/ash-free basis. Evaluation of the various processing configurations will follow the same outline as previous PAM screening studies:

- + partial oxidation for hydrogen, FLEXICOKING (once-through and recycle) for fuel;
- + steam reforming for hydrogen, FLEXICOKING (once-through and recycle) to feed;
- + partial oxidation for hydrogen and for fuel.

In addition, the following sensitivities will be examined:

- + Deeper cut vacuum pipestill operation (for cases using partial oxidation of vacuum bottoms for hydrogen and/or fuel);
- + C<sub>2</sub>- gas sales parity values for FLEXICOKING/partial oxidation vs. FLEXICOKING/steam reforming;
- + IBG sales in the all partial oxidation configuration as well as calculation of the resulting parity values relative to the overall best configuration and to the all partial oxidation configuration selling vacuum bottoms;
- + Several spreads between naphtha and fuel oil values for all configurations.

Once completed, Burning Star coal liquefaction will be compared to Illinois No. 6 (Monterey) and Pittsburgh No. 8 coal liquefaction on a "best-case" basis; such a comparison will give an indication of the relative profitability of operating a plant with Burning Star coal, versus either Monterey or Pittsburgh coals.

## 2.6 ECLP Slop Disposal

An investigation was conducted to assess alternative slop disposal methods other than continual run-off with feed to the slurry drier. Concern had been expressed that continual slop run-off would result in degradation of ECLP data and that this disposal method should be avoided. The investigation concluded that slop inventories could be run-off in blocked operations without the need for specification of new auxiliary facilities. Estimates of slop production rates combined with anticipated service factors for various areas of the ECLP processing sequence indicated that sufficient flexibility was already available to allow this blocked slop run-off without significantly affecting ECLP's availability for production of program data.

Among the key points brought out by the investigation was the desirability to recover the solvent portion of the slop. Solvent is required for blending with naphtha to meet sulfur specifications when excess liquid products are disposed of as fuel, to satisfy sponsor requests for ECLP products, and to provide flushing oil for normal plant operations. As a result, out-right disposal of slop to a contracted waste disposal firm should be avoided except during emergency situations.

Two alternative blocked run-off operations were identified. One operation involves use of the liquefaction and fractionation sections of the plant with slop feed going to the slurry driers during periods of controlled plant shutdowns. One month's slop inventory could be disposed of in approximately 17 hours using this technique. The second operation involves using only the vacuum furnace and fractionator portion of the plant in a blocked operation. Disposal of one month's slop inventory would take approximately two days using this technique.

A question remained regarding the ability to solidify slop bottoms at all times using the Sandvik belt at the vacuum tower. Steps which could solve or reduce the problem regarding bottoms solidification were identified and included:

- + Use of a waste disposal firm to remove slop bottoms in which solids have been concentrated.
- + Recycle of slop bottoms back to the slop storage tanks until the high viscosity bottoms/solids concentration is high enough to allow solidification.
- + Run-off of slop in a blocked operation which involves use of the vacuum furnace, atmospheric tower, and vacuum tower in series.
- + Use of the concentrated slop bottoms as flux material for the prototype FLEXICOKING unit.

To evaluate the cost-effectiveness of blocked slop run-off operations, an approximate estimate was made of the investment which would be required for auxiliary rerun facilities. An investment of the order of 1.7-2.4 M\$ would be required. Aside from the cost, other considerations were identified which would present several difficulties. Among these were the timing required to design and construct facilities for the 1980 startup, the availability of utilities to support the operation, the increased operator attention needed to run the facility, and the availability of plot space for the additional equipment.



## 2.7 Phenol Recovery from EDS Naphtha

An overall process using a methanol/water extraction for recovery of phenols from EDS naphtha is being evaluated. The flow scheme is depicted in Figure 2-1 and includes the following units: naphtha splitting, phenolic extraction, methanol recovery, phenolic concentration, and phenol splitting. Naphtha feed rates and qualities used in this study are the same as for the debutanizer bottoms stream from the light ends section of the EDS Commercial Study Design Update.

Extraction of phenolics prior to hydrotreating was investigated earlier and found to be potentially more attractive than the base case which hydrotreats and reforms the raw EDS naphtha to mogas. Relative to a phenol-free naphtha, substantially more hydrogen is consumed and additional catalyst is required during hydrotreating. Economic evaluations were completed previously for extraction processes with water and dilute caustic as solvents, revealing a potential credit of \$0.50-\$1.00 per barrel of total EDS product.

Methanol/water extraction of phenol offers advantages over water and caustic extraction. Over caustic, methanol/water eliminates the consumption of reagents and solid disposal problems associated with caustic washing. Relative to extraction with water, methanol/water offers higher recoveries at much lower solvent/feed ratios which reduces equipment size and requires less heat input.

Bench scale batch extraction studies were carried out to determine the recovery of phenolics from EDS naphtha achievable with methanol solvents. Solvent compositions (0 to 90 wt% methanol in water), and solvent/feed ratios (0.5, 1.0 and 2.0 volume basis) in single batch extractions were investigated. The experimental data base was later extended to include solvent/feed ratios below 0.5/1.0.

Results from the lab studies showed, that recoveries in excess of 90 percent can be achieved in a single extraction stage with solvents containing at least 50 wt% methanol and a solvent/feed of 2.0/1.0. Similar recoveries are obtained with solvents containing at least 70 wt% methanol and a solvent/feed ratio of 0.5/1.0. Extract purities with various solvent blends were highest at a 1.0/1.0 solvent/feed ratio, falling off as solvent/feed was increased to 2.0/1.0 or decreased to 0.5/1.0. Also, the capacity of the solvent for solubilizing phenols was reached at about 0.5/1.0 solvent/feed ratio.

Based on these batch extraction data, a 60 wt% methanol in water solvent and a 0.5/1.0 solvent/feed ratio was selected as being close to the optimal balance of phenol capacity and selectivity. A ternary phase diagram showing the equilibrium data and stream compositions for these conditions is shown in Figure 2-2.

Figure 2-1

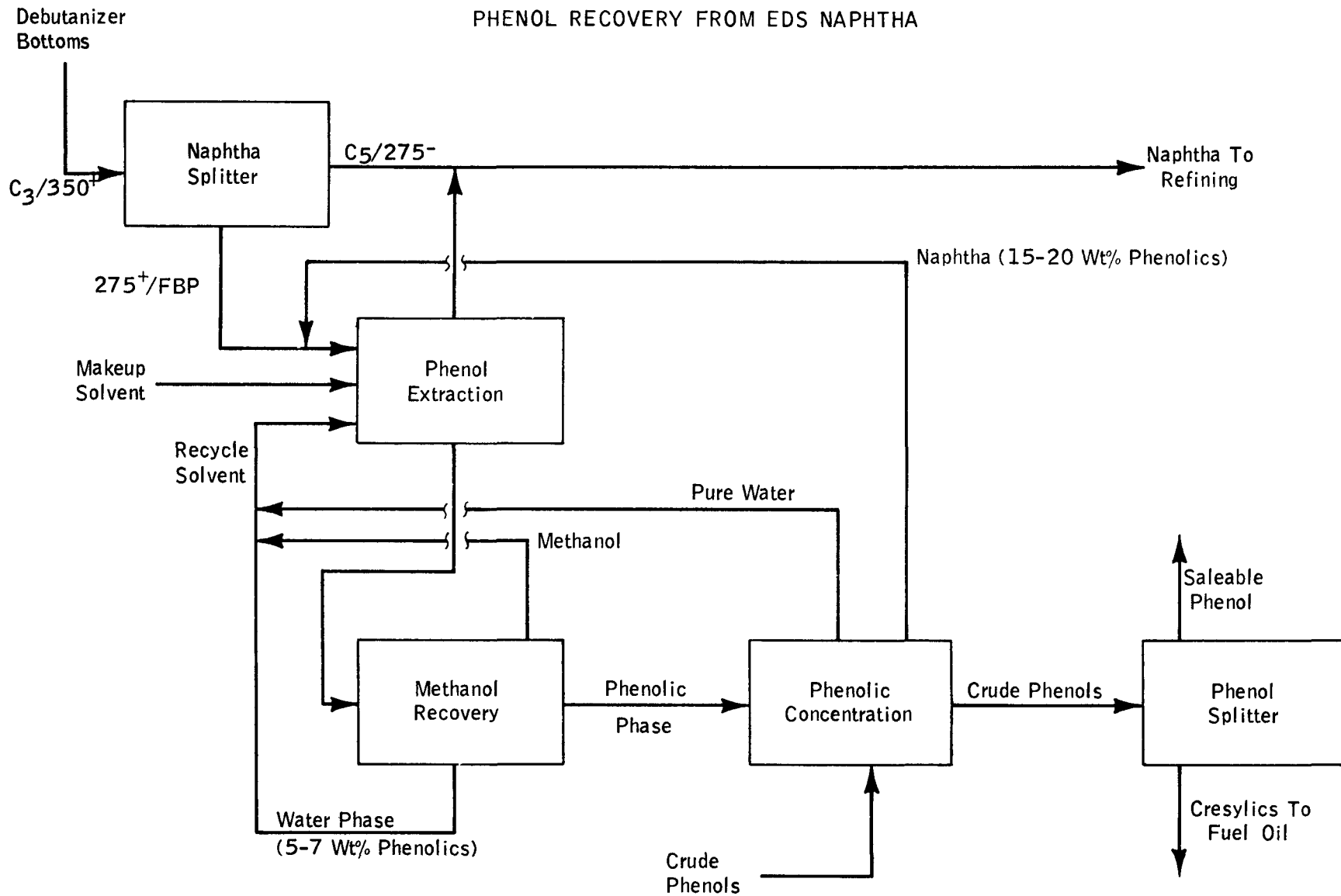
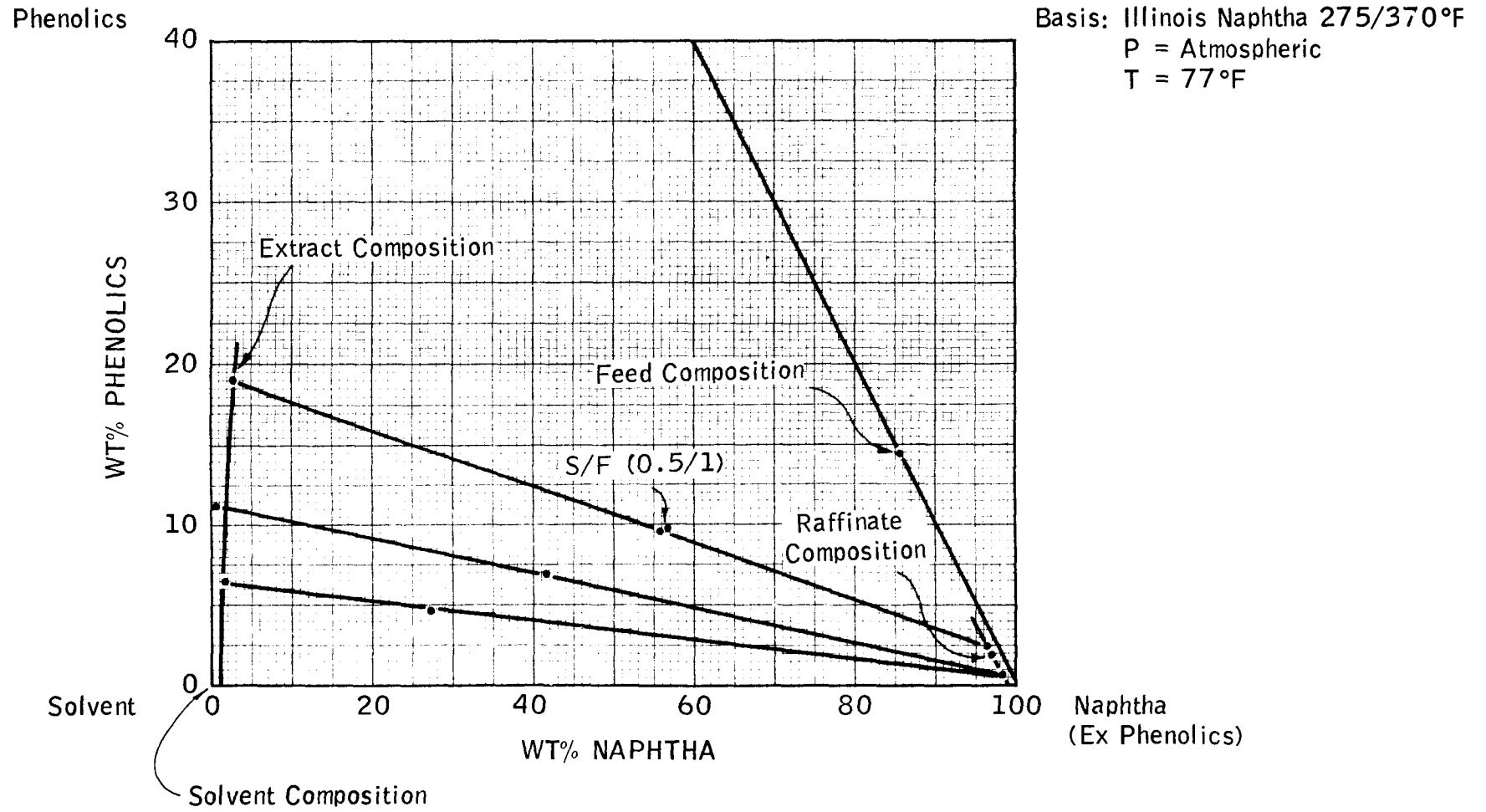


Figure 2-2

TERNARY DIAGRAM - ILLINOIS NAPHTHA  
WITH 60/40 MeOH/H<sub>2</sub>O SOLVENT



The process block flow diagram for recovery of Phenol from naphtha using methanol/water extraction is shown in Figure 2-1.

In this study debutanizer bottoms, nominally 350°F, from the light ends section are fed to a naphtha splitter where the naphtha is separated into a low phenol content distillate (C<sub>5</sub>/275°F) which is sent to refining and a 275°F/FBP naphtha (10-15 wt% phenolics) which is sent to the Phenolic Extraction system. This fractionation step substantially reduces the required capacity of downstream processing equipment.

In the Phenolic Extraction system 90 wt% of the phenolics are removed from the naphtha using a 60 wt% methanol in water solvent and a 0.5/1.0 solvent/feed ratio.

The extract rich in methanol and phenolics, is sent to the Methanol Recovery section which consists of a Methanol fractionator and a settling drum. In the Methanol Fractionation 95-99% of the methanol in the feed is recovered and recycled back to the phenolic extraction unit. Bottoms are cooled forming two liquid phases which are separated in the settling drum. The upper layer is a water phase, saturated with phenolics (5-7 wt%), which is recycled back to the extraction unit. The lower layer contains phenols/naphtha/water (70/10/20 wt%) and is sent to the Phenolic Concentrator.

In the Phenolic Concentrator, crude phenolics from waste water treating are added to the phenolic phase from the settling drum to obtain maximum phenols recovery. This concentrator fractionates water and lighter hydrocarbons from the phenolics. The water and naphtha (containing 15-20 wt% phenolics) are separated in the distillate drum and recycled back to the Extraction Unit. Naphtha is blended with extractor feed and water with recycle solvent.

The heavy naphtha/phenolics bottoms stream is then sent to a Phenol Splitter where high purity phenol is recovered overhead. Higher boiling phenols and naphtha are taken as a bottoms product and blended into fuel oil.

## 2.8 Data Reconciliation Model for ECLP

A Data Reconciliation System (DRS) model for handling ECLP process data is currently under development. DRS is a generalized computer program developed by the Exxon Communications and Computer Sciences (ECCS) Department of Exxon Corporation. Models built with DRS are able to determine more accurate and internally consistent values from raw operating data for improved process monitoring and operations analysis. The DRS model accomplishes reconciliation with a least squares analysis of errors that is based on the reliability (standard deviation) of each of the flow or analytical measurements used in generating material balances.

Mass balances that will be carried out include both total flow and components (carbon, hydrogen, nitrogen, sulfur and ash). The flow balance model has been completed. It is comprised of 35 balance areas and 151 "streams". Included in the "stream" measurements are delta inventories (level changes) in the process equipment, as well as flow meters, weigh belts and pump speed monitors.

The elemental balances will be slightly simplified with about 25 balance areas and about 100 streams. Simplified elemental balances are necessary because of laboratory analysis and sample taking constraints. However, the ECLP sections that are of greatest interest will be included. Not included are Fuel Gas Treating, DEA Regeneration, and Naphtha Weathering. These sections use conventional petroleum technology and are not representative of facilities anticipated for a commercial plant, therefore, flow reconciliation alone will be adequate.

The component balance model will be completed in July. Subsequent work will include interfacing DRS with the ECLP Data Logger and laboratory analysis results. This work will continue during the third quarter of this year.

## 2.9 $\text{CaCO}_3$ Deposition in Liquefaction Reactors With Wyoming Coal Feed

Dealing with  $\text{CaCO}_3$  deposition in liquefaction reactors with high-calcium coal feeds has been examined from two directions: (1) minimizing adverse effects of the  $\text{CaCO}_3$  once it forms and (2) chemical feed pretreatment to prevent scale formation. After completing laboratory feed pretreatment experiments to define the chemistry and exploring possible alternatives, the two preferred solutions are:

- Mechanical: Withdraw solids from the reactors, add protective screens and strainers, and chemically clean equipment during turnarounds
- Chemical: Pretreat the feed coal with  $\text{SO}_2$  to convert the calcium salts in the feed coal to  $\text{CaSO}_4$  which is stable in liquefaction

The mechanical approach is preferred because it is lowest-cost. However, its efficiency must be demonstrated in ECLP. The development approach will be to defer development of the  $\text{SO}_2$  pretreatment process pending results from the ECLP demonstration of the mechanical approach. In the event that it does not provide satisfactory control of the  $\text{CaCO}_3$  deposition problem in ECLP tests, a full commercial development program for  $\text{SO}_2$  pretreatment would be undertaken.

The commercial design modifications in the preferred approach include the following additions:

- Solids withdrawal nozzles at each distributor from both the side and through the tray.
- Wire screens with 1" openings located about three feet above each distributor.
- Y-type strainers with 1/4" screen openings upstream of the slurry letdown valves that control the reactor effluent separator bottoms flow to the atmospheric fractionator.

With these modifications the majority of the  $\text{CaCO}_3$  would be removed through the solids withdrawal nozzles. If large scale should spall from the reactor wall (scale that is too large for the solids withdrawal nozzles), the screens above the distributors will prevent the scale from plugging the distributor. The spacing between the distributor and screen assures good flow distribution even if the screens should become partially plugged. Should any scale be carried over from the liquefaction reactors by pressure surges, the Y-type strainers will protect the high-pressure letdown valves from plugging.

To prevent long-term scale build-up, the reactors would be periodically cleaned with dilute phosphoric acid. Laboratory tests on scaled reactor tubes from RCLU have demonstrated complete scale removal with this technique. The chemical cleaning operation would be carried out during slurry furnace decoking operations so that no additional downtime is needed. The cost of the chemical cleaning would be about 25 k\$ per reactor.

A general plan to test the design and operational changes in ECLP has been formulated. Necessary ECLP equipment changes will be specified after several months of initial ECLP operation on Illinois coal. This will enable the use of ECLP experience in developing equipment modification designs for both ECLP and commercial.

As mentioned earlier, if ECLP experience shows that satisfactory control is not obtained with the design and operational changes, a full commercial development program for SO<sub>2</sub> pretreatment would be undertaken. The SO<sub>2</sub> pretreatment scheme has been reported in several past quarterly and annual reports, with the latest update of the economics appearing in the July-September 1978 Quarterly.\* SO<sub>2</sub> pretreatment costs for a commercial plant are estimated to be 32 M\$/yr (2.35 \$/B, 1985). In this scheme sulfur is burned in air to form an 8 mol% treat gas which is passed through the coal in a fluidized bed. About 60% of the SO<sub>2</sub> is reacted across the fluid bed with the remainder being recycled either directly or through a Wellman-Lord scrubber/regenerator.

Limited SO<sub>2</sub> pretreatment process development tests have been carried out to answer key process feasibility operations and to prepare for commercial development if it is needed. (See Laboratory Process R&D Item 4. for a description of this at work).

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\*FE-2893-21

## 2.10 ECLP Test Program

Definitive planning of the ECLP Test Program began in the 4th Quarter of 1978. Three tiers of program plans are being prepared. These three plans, in order of increasing detail, are described below:

1. Nominal 2 1/2-year Test Program - This plan starts with coal-in and covers the entire ECLP operating period. It is an outline of ECLP testing objectives for major periods of operations which includes processing three coals: an Illinois No. 6, a sub-bituminous and a third coal to be named.
2. One-Year Test Programs - These plans cover each calendar year of operations and will be used for budget preparation and manpower planning as well as providing a framework for scheduling individual tests. The first one-year test program will cover the entire 15-month operation with Illinois No. 6 coal.
3. Three-Month Roll-Over Test Programs - A new three-month plan will be prepared every month and will incorporate results of past operations into the test schedule. These plans will serve as the basis for the ECLP organization's preparation of detailed operating plans.

To date, the 2 1/2-year Test Program and the first one-year test program (covering Illinois No. 6 operations) have been prepared. The 2 1/2-Year Test Program schedule is summarized in Figure 2-3. The two key overall objectives are to:

1. Demonstrate operability of EDS Liquefaction
2. Obtain required scaleup data for the design of a Commercial Pioneer Plant with acceptable risk.

A possible sequence of operations for the first one-year test program, which has been expanded to cover the entire 15-month Illinois No. 6 operations, is shown in Figure 2-4. This sequence has been used to provide a basis for scheduling individual component tests. In scheduling tests an attempt was made to accomplish the following:

- Conduct related tests concurrently
- Prevent interference between unrelated tests
- Provide early assessment of unanticipated results
- Bracket the range of operability as soon as practical without upsetting operations
- Allow for the simplest possible analysis of results



Figure 2-3  
SCHEDULE  
2-1/2-YEAR ECLP TEST PROGRAM

Operations with Illinois No. 6 Coal - 15 Months

- |  |          |
|--|----------|
| I. Shakedown Operations  | 5 Months |
| - Solve operating problems.  |          |
| - Turnaround for modifications.  |          |
| II. Initial Sustained Run  | 2 Months |
| - Demonstrate ability to operate for a prolonged period.                                   |          |
| - Prove out ECLP as a research/development tool.   |          |
| III. Exploration of Operability Limits   | 4 Months |
| - Determine equipment limitations.   |          |
| - Investigate alternate conditions/configurations.   |          |
| IV. Long-Term Operability  | 3 Months |
| - Demonstrate capability to operate for an extended period and obtain time dependent data. |          |
| - Simulate a commercial operating environment.   |          |
| V. Turnaround  | 1 Month  |

Operations with Sub-bituminous Coal - 9 Months

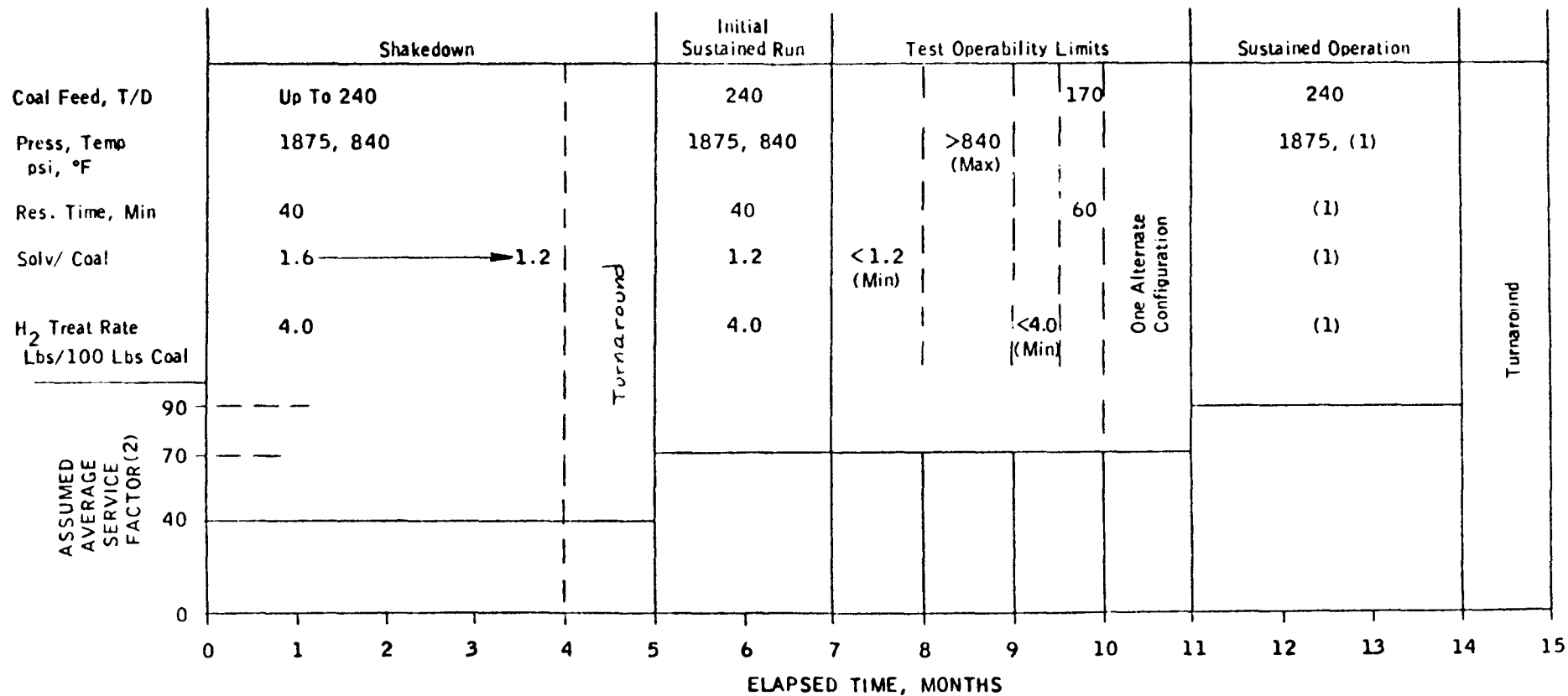
- |  |            |
|--|------------|
| I. Shakedown and Initial Testing                                   | 3 Months   |
| - Solve operating problems.  |            |
| - Evaluate operational differences (comparison to Illinois No. 6). |            |
| II. Exploration of Operability Limits                              | 2 Months   |
| - Same as Illinois No. 6.  |            |
| III. Long-Term Operability   | 2.5 Months |
| - Same as Illinois No. 6.  |            |
| IV. Confirmation of Controlled Scaling                             | 0.5 Month  |
| - Chemically clean liquefaction equipment.                         |            |
| - Inspect equipment (before and after cleaning).                   |            |
| V. Turnaround  | 1 Month    |

Operations with Third Coal - 6 Months\*

To be developed when coal is identified. Approach will be similar to sub-bituminous coal program.

\* Testing period will be shortened, if necessary, to stay within overall project budget constraints.

Figure 2-4  
ECLP TEST PROGRAM  
POSSIBLE SEQUENCE OF OPERATIONS  
FOR ILLINOIS NO. 6 COAL



Notes:

- (1) To Be Determined
- (2) Includes Both Scheduled And Unscheduled Outages.

A summary of selected development programs included in the schedule is given in Figure 2-5. The testing is broken down into eight areas, the majority of which emphasize component testing rather than yield testing. A listing of proposed testing was given in the April Monthly.\* As a result of initial scheduling, it appears that there is sufficient time available in the 15-month program to complete all of the proposed tests. The planned average service factor described in Figure 2-4 is high enough for required process runs, yet there is sufficient downtime for the needed inspections.

Near-term work in the area of ECLP Test Program development includes finalizing the first one-year program and preparation of the first three-month roll-over test program. The latter is scheduled for completion in the Fall of 1979. This program will give a detailed look at the first three months of testing. Since this is a shakedown period, there will be relatively little testing other than assuring adequate operability and identifying problem areas.

Other activities related to the ECLP Test Programs that were planned for the balance of 1979 include:

- Develop detailed planning and initiate design of plant modifications that are required for conducting tests
- Start definitive planning of pre-startup testing
- List data that are required for each test

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\*FE-2893-30

Figure 2-5  
ECLP TEST PROGRAM  
SUMMARY OF SELECTED DEVELOPMENT PROGRAMS

Liquefaction Product Fractionators

- Evaluate Applicability of Petroleum Distillation Correlations
- Determine Fouling, Entrainment and Foaming Characteristics

Liquefaction Reactors

- Develop Design Correlations

Fluid Flow - Coal Derived Liquids and Slurries

- Establish Design Correlations for Saltation and Pressure Drop

Heat Transfer - Coal Derived Slurries

- Determine Coking Rates in Furnace Tubes
- Develop Heat Transfer Correlations
- Measure Erosion of Serpentine Coil Furnace Tubes

Valves and Instruments in Slurry Service

- Establish Reliable Designs for Isolation and Check Valves
- Determine Design Characteristics of Pressure Let Down Valves
- Develop Reliable Level, Flow and Pressure (and  $\Delta P$ ) Measurement Capabilities

Pumps in Slurry Service

- Establish Design Criteria and Correlations for Both Reciprocating and Centrifugal Pumps
- Develop reliable Mechanical Seal Designs

Materials of Construction

- Determine Corrosion/Erosion Rates for Base and Alternate Materials

Air/Noise and Water Pollution Control

- Measure Pollutant Levels for Use in Future Permit Applications

## ENGINEERING RESEARCH AND DEVELOPMENT

### PROCESS ENGINEERING AND COST EVALUATIONS

#### 3. Engineering Studies of Bottoms Processing/Hydrogen Manufacture 7. Supplemental FLEXICOKING Program

In June, 1978 ERE completed a reassessment of the EDS liquefaction bottoms processing program. The goal of the EDS development program is to advance the EDS process to commercial readiness so that the design of a pioneer commercial plant could begin in 1981 with a reasonable and acceptable level of risk. In light of this goal, ERE concluded that development of vacuum bottoms processing was on the critical path. ERE proposed a program which was comprised of expanding the coal liquefaction bottoms FLEXICOKING program to include operation of a 70 T/D prototype unit and aggressively evaluating alternative bottoms processing approaches such as partial oxidation. This program was to be implemented in two parts keyed to the design and construction schedule of the 70 T/D FLEXICOKING prototype. At the conclusion of Part I, the prototype project would be ready to enter the construction phase requiring major commitment of program funds. Prior to making this commitment, the assessment of available bottoms alternatives would be reviewed to insure that proceeding with the FLEXICOKING prototype was warranted.

During the last half of 1978 and the first half of 1979, bottoms processing studies within the EDS program have therefore focused on two main objectives. First, definitive planning studies and design work have been undertaken on the FLEXICOKING prototype to better define the program cost and operating plan. Secondly, alternative bottoms processes have been extensively evaluated to determine the development status of the technology, the interface of the bottoms process with the liquefaction section and the relative product costs for various EDS process configurations. Primarily, emphasis has been on partial oxidation for fuel and hydrogen production with both the Texaco and Shell processes being investigated. Screening studies have also been undertaken to look at other alternatives, most notably direct combustion of bottoms for process fuel. The goal of this activity is to complete the reassessment of alternatives for review with the EDS sponsors in September/October, 1979. The reassessment will provide the information necessary to evaluate the various alternatives with regard to development timing and cost, process and product flexibility and product cost. From this evaluation, the EDS bottoms processing development strategy will be selected which best suits the project needs as determined collectively by the sponsors.

The activities reported herein constitute various elements of this overall reassessment. The conclusions and resulting bottoms processing development strategy will be discussed in future reports.

### 3.1 Fuel Gas Cost Comparisons

Several new or revised Process Alternatives Model (PAM) fuel gas vectors for Illinois coal were developed in support of PAM process improvement studies. These include an entrained flow partial oxidation vector based on coal feed, and a modification of the existing moving bed gasification fuel gas vector. Low Btu fuel gas costs from a FLEXICOKING unit feeding vacuum bottoms have also changed as a result of yield and investment changes identified in the Study Design Update. Because of these changes a comparison of fuel gas costs from various process options was updated.

Section 10.8 of the Phase III A Final Technical Progress Report, FE-2353-20, January 1, 1976 - June 30, 1977, reported the costs of fuel gas from several sources as a function of supplemental coal to the plant. Table 3-1 presents the current update for the component cost of fuel gas for an EDS plant configuration using Illinois #6 coal. (Coal fired boilers are available in each configuration.) The fuel costs presented are marginal values obtained from PAM results.

#### FLEXICOKING

Fuel gas from a FLEXICOKING unit is in the range of 3.00-3.75 \$/MBtu. Several changes to the FLEXICOKING unit between the 1975/1976 Study Design and Study Design Update affected LBG cost. The SDU FLEXICOKING unit reflects an investment increase due to more accurate cost estimates, and to increasing unit size. Higher product credits reflect changes in gas and liquid yield. Production of 125 psi steam from the heater overhead gas instead of 600 psi steam results in lower utility costs for the SDU FLEXICOKING unit. While capital costs have risen for the SDU FLEXICOKING unit, the marginal price of fuel has dropped from 3.74 to 3.08 \$/MBtu. The reason is lower feed costs for the SDU FLEXICOKING unit, for as FLEXICOKING costs increase, the bottoms become less valuable as coker feed.

#### Entrained Bed Gasification

The entrained bed gasifier vectors were expanded to use either vacuum bottoms or coal feed. The higher cost of fuel from coal (7.18 \$/MBtu) versus vacuum bottoms (4.61 \$/MBtu) can be attributed to higher investment and feed costs. The lower cost of vacuum bottoms vs. coal feed is due to the pricing structure in the PAM. Vacuum bottoms sales are fixed at coal parity. However, vacuum bottoms consumed within the process are valued below coal parity due to the cost of the bottoms sales facilities.

TABLE 3-1

PAM FUEL COSTS IN ILLINOIS EDS PLANTS<sup>(1)</sup>

Fuel Units:	FLEXICOKING	FLEXICOKING	Entrained Bed	Entrained Bed	MBG <sup>(2)</sup>	MBG <sup>(2)</sup>	
Fuel Source:	Bottoms	Bottoms	Bottoms	Coal	Coal	Coal	
Vector Status:	<u>Current</u>	<u>SDU</u>	<u>Current</u>	<u>Current</u>	<u>Old</u>	<u>Current</u>	
Fuel Production, kFOET/CD	1.1	1.3	1.1	1.2	1.9	1.1	1.8
<u>Costs, \$/MBtu</u>							
Investment	3.31	4.81	1.58	2.86	1.26	2.91	2.37
Feed costs	5.25	3.46	1.09	1.81	1.86	1.94	1.98
Gas and Liquid By-product Credits	(6.07)	(6.54)	-	-	(0.73)	(0.48)	(0.57)
600 psi Steam	(1.12)	0.26	(0.73)	(0.87	1.01	1.25	1.35
Oxygen	-	-	1.94	1.92	-	-	-
Utilities	1.95	0.73	0.55	1.00	0.52	1.29	1.64
Miscellaneous <sup>(3)</sup>	<u>0.42</u>	<u>0.36</u>	<u>0.18</u>	<u>0.46</u>	<u>0.86</u>	<u>0.85</u>	<u>0.52</u>
Cost of Fuel, \$/MBtu	3.74	3.08	4.61	7.18	4.78	7.76	7.29

Notes:

- (1) 1985 Economic Basis  
 (2) MBG - Moving Bed Gasification  
 (3) Includes sulfur and ammonia by-product credits, ash handling facilities, sour water facilities, etc.

### Moving Bed Gasification

The old MBG vector produced fuel at about 4.80 \$/MBtu, while the current vector fuel costs is approximately 7.30-7.75 \$/MBtu. These higher costs are attributed, in part, to a higher investment which resulted from a more detailed process basis. Decreased liquid yields, and lower tar oil product value (RSFO vs. LSFO) have reduced liquid product credits. Also, an increase in air and steam requirements for gasification have resulted in higher steam and compression costs.

### Purge Gas

Table 3-2 presents the costs associated with burning liquefaction purge gas for fuel. The costs presented have been adjusted to reflect internal component values prior to cryogenic fractionation and light ends processing. This adjustment is necessary because the PAM values C<sub>4</sub>- purge gas fuel components at their market price.



TABLE 3-2

PAM PURGE GAS FUEL COST IN ILLINOIS EDS PLANT

<u>Component of Purge Gas</u>	<u>Mole %</u>	<u>Costs, \$/MBtu of Purge Gas</u>
C <sub>2</sub>	36.1	4.00
C <sub>3</sub>	2.6	0.40
C <sub>4</sub>	1.3	0.11
H <sub>2</sub>	60.0	<u>1.06</u>
Cost of Fuel, \$/MBtu		5.57

### 3.2 Wyoming Coal Liquefaction Screening Study

The Wyoming Coal Bottoms Processing screening study evaluated a range of liquefaction conditions to select the Study Design Basis for the liquefaction of Wyoming coal (July 1-September 30, 1978 Quarterly Technical Progress Report)\*. The optimal liquefaction conditions were then considered with the following bottoms processing alternatives to determine the most attractive plant configuration:

#### Hydrogen

- Steam reforming of high Btu gas
- Partial oxidation of vacuum bottoms (POX)
- Moving-bed gasification of coal (MBG)

#### Fuel

- FLEXICOKING of vacuum bottoms (LBG)
- Partial oxidation of vacuum bottoms (IBG)
- Moving-bed gasification of coal (LBG)

#### Steam

- Coal-fired boiler
- Gas-fired boilers

The economic basis is 1985 plant startup and operation.

An overall economic comparison of the various fuel and hydrogen producing combinations was developed. The results are summarized briefly, and a detailed explanation of the results can be found in the reference cited above.

#### Overall Economic Evaluation

A comparison of the various hydrogen and fuel producing combinations is presented in Figure 3-1. This figure shows the net cash flow for the EDS plant versus the hydrogen producing process. The lines of Figure 3-1 represent three different options for providing process fuel: FLEXICOKING, partial oxidation of vacuum bottoms, and moving-bed gasification of coal. The results are presented for liquefaction conditions of 840°F and 60 minutes residence time.

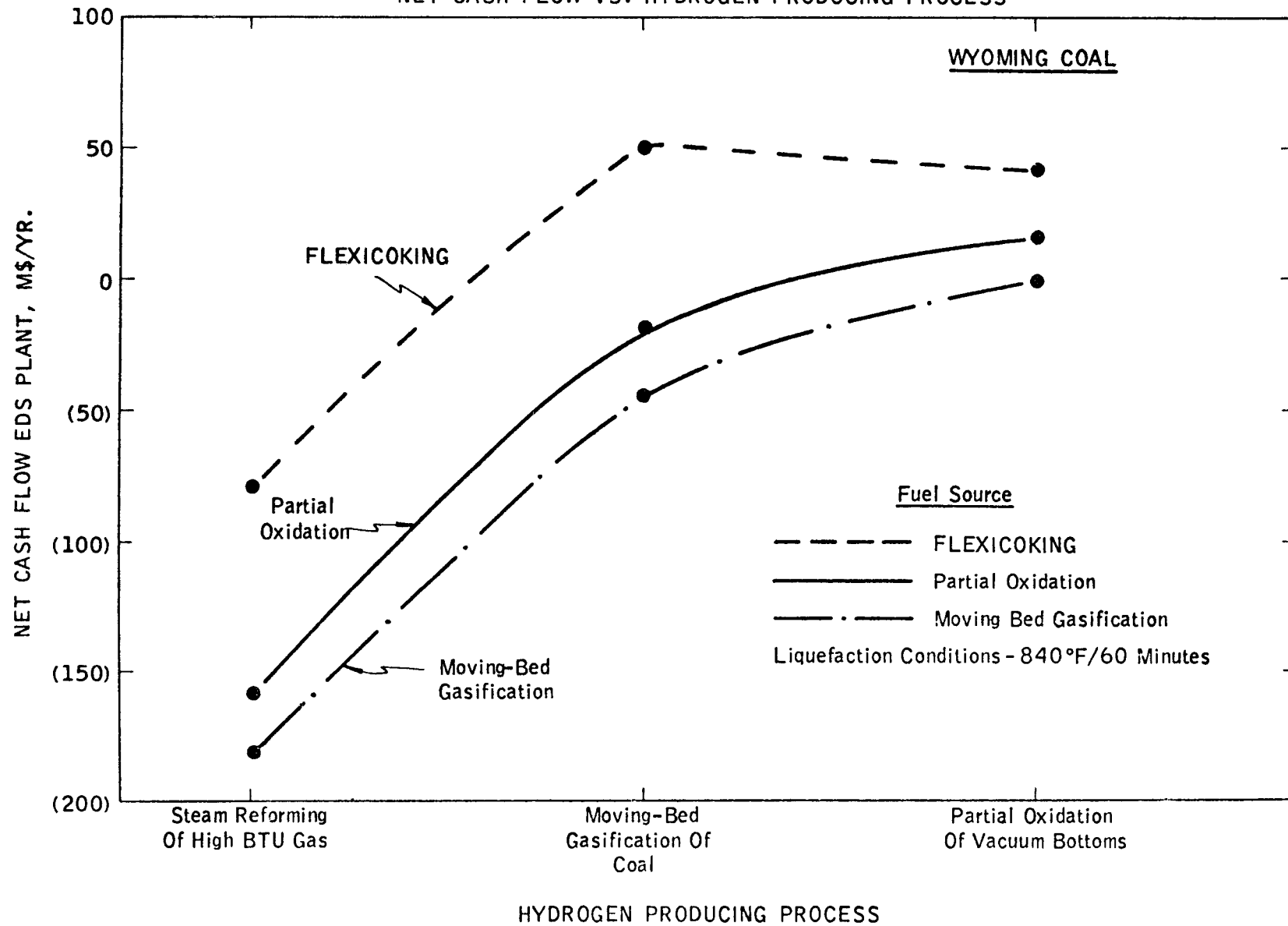
For all cases shown in Figure 3-1, high Btu gas, when it is available, is sold in parity with coal liquids distillate. A coal-fired boiler is used as needed to provide supplemental plant steam. The 1000°F-content of the vacuum bottoms is 9% in all cases.

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\*FE-2893-21

Figure 3-1

NET CASH FLOW VS. HYDROGEN PRODUCING PROCESS



### FLEXICOKING For Fuel

The dashed line in Figure 3-1 represents the use of FLEXICOKING to provide plant fuel. Hydrogen is produced by one of three processes: steam reforming, moving-bed gasification of coal, or partial oxidation of vacuum bottoms.

The major differences between the MBG and POX cases are the amount of supplemental coal to the plant, and the liquid and gas yields. In the case which uses MBG of coal, all vacuum bottoms are fed to the FLEXICOKING unit and supplemental coal is used for hydrogen production. The sale of HBG increases significantly due to the large amounts of high Btu gas produced in the MBG unit.

The cost of hydrogen is greater from the MBG unit than from the partial oxidation unit. However, added revenues from liquids recovered by FLEXICOKING the entire vacuum bottoms stream, and increased HBG production, compensates for the increased cost of hydrogen.

The other configuration which uses FLEXICOKING for fuel utilizes steam reforming of HBG to produce hydrogen. This is the least attractive case of those using FLEXICOKING for fuel. The major reason steam reforming is less attractive than either MBG or partial oxidation is that  $C_2^-$  is not available for sale.

### MBG and Partial Oxidation for Fuel

Neither MBG or POX are as attractive as FLEXICOKING for fuel. The cost differential between the partial oxidation and moving bed gasification cases is nearly constant. This 20 M\$/yr delta is due to the higher cost of fuel from the MBG unit because of higher investment, feed, and operating costs.

### Summary of Screening Cases

From this screening study, the three most attractive cases were selected for further evaluation. These are: (1) FLEXICOKING-fuel/Partial Oxidation-hydrogen, (2) FLEXICOKING-fuel/MBG-hydrogen and (3) Partial Oxidation-fuel/Partial Oxidation-hydrogen.

This second phase evaluation considered several design-related issues. One issue is the viscosity of the vacuum bottoms. A sufficiently low viscosity must be maintained when feeding vacuum bottoms to a FLEXICOKING or partial oxidation unit to avoid pumping and stability problems. Several factors affect the final viscosity of the vacuum bottoms. The 1000°F- content (which is determined by the vacuum tower cut point temperature), liquefaction severity, storage time, and storage temperature are all important. To increase liquid yield, a low 1000°F- content is preferred for feeding a partial oxidation reactor. However, high 1000°F- content aids in maintaining the low viscosities necessary for pumping.

FLEXICOKING and partial oxidation units have different feed requirements. Figure 3-2 shows the minimum viscosity for a given storage time versus the 1000°F- content of the vacuum bottoms for each unit. The minimum viscosity results from an optimized combination of initial viscosity and viscosity stability (both of which decrease with increasing temperature) such that the resulting viscosity after a given storage time is as low as possible. The viscosity of vacuum bottoms from 840°F/40 minutes and 840°F/60 minutes liquefaction conditions are indicated. A maximum viscosity of 60 poise was used as the current pumping limitation.

The design basis for the partial oxidation reactor requires a 2-hour storage time for process feed control. With liquefaction conditions of 840°F/60 minutes, an acceptable viscosity below the pumping limitation was determined with a 1000°F- content of 2%.

The design basis for vacuum bottoms storage time for a FLEXICOKING unit is 8 hours. Figure 3-2 b indicates the 1000°F- content of the vacuum bottoms necessary to keep the viscosity below the pumping limitation of 60 poise. The need to limit the feed 1000°F- content, however, is not important in this case since the FLEXICOKING reactor recovers all 1000°F- liquids remaining in the feed.

#### Base Case Configuration

The analysis of viscosity stability and vacuum pipestill cut point indicated that liquefaction residence times of 60 minutes or longer at 840°F would provide a pumpable feed to a partial oxidation reactor with lower 1000°F- contents than was assumed in our initial screening studies (9%). Therefore, the three best cases from the initial screening study (FLEXICOKING/POX for fuel/hydrogen respectively) were compared at various liquefaction residence times with a vacuum bottoms 1000°F- content of 2% (Figure 3-3). The dotted lines in Figure 3-3 below the 60 minutes residence time indicate a vacuum bottoms viscosity that exceeds pumping limitations.

The configuration at 840°F using FLEXICOKING for fuel and partial oxidation for hydrogen became the most economically attractive. The improvement of the economics of the FLEXICOKING/POX case relative to the FLEXICOKING/MBG case (Figure 3-3 vs Figure 3-1) results from the additional liquid recovered from the vacuum pipestill operating at a 2% 1000°F- vacuum bottom content.

The bottoms screening study has indicated that a range of liquefaction conditions can provide an economically optimum Wyoming plant configuration. Final liquefaction operating conditions for the Wyoming Study Design will be selected based on this study, and laboratory pilot data on bottoms viscosity and unit operability.

Figure 3-2  
MINIMUM VISCOSITY VS. 1000°F- CONTENT

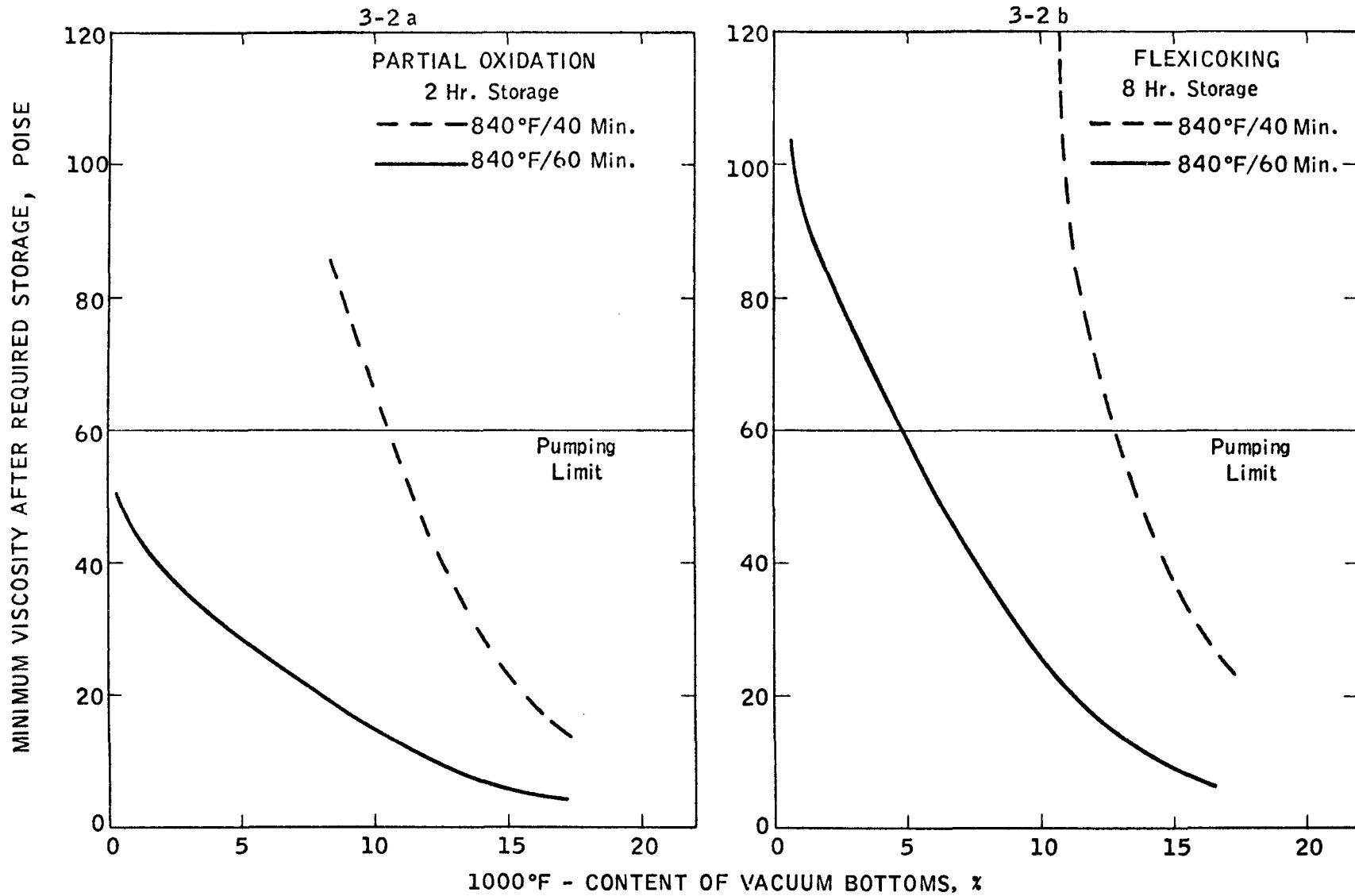
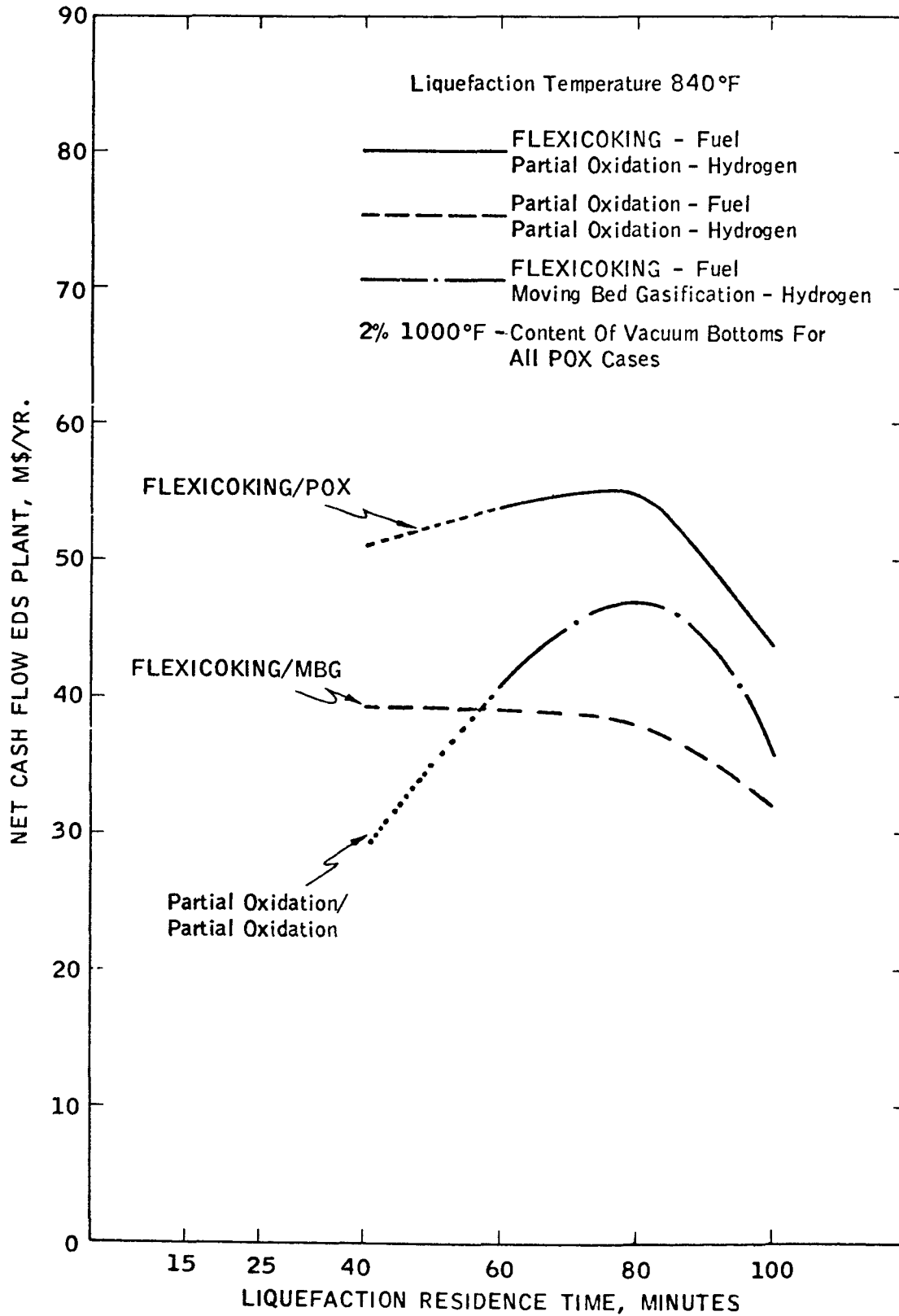


Figure 3-3

NET CASH FLOW VS. LIQUEFACTION RESIDENCE TIME



### 3.3 FLEXICOKING Improvement Studies

The study to investigate techniques for reducing the solids content of coker scrubber liquids is continuing. An EDS plant incorporating a recycle FLEXICOKING unit can produce a solids-free distillate fuel oil but at a cost higher than the SDU basis which employs a once-through FLEXICOKING unit. Presently the same scrubber design is used for once-through and recycle coking. Possible design changes are being investigated which can reduce solids entrainment from the reactor, or can increase recovery of clean coker products. Evaluation of alternate methods for solids removal from the scrubber bottoms is also underway.

Several methods of achieving a higher recycle coking scrubber cut point are being developed. Raising the scrubber vapor exit temperature will achieve a deeper cut point and permit greater liquid recovery. At elevated temperatures, coking on the scrubber sheds may occur more readily and means are being considered to minimize this problem.

Preliminary results indicate the economic debit between once-through and recycle coking can be reduced by about 30-50% by raising the scrubber cut point. Laboratory results indicate coke formation may not be a problem; however, demonstration of this higher cut point would be needed to insure coke formation is minimized.

Another approach to increase recovery of solids-free 1000°F+ liquids is to improve the scrubber design. Redesign of the FLEXICOKING scrubber would permit operation at a very high recycle cut point, and could nearly eliminate the economic delta between once-through and recycle coking. Several key technical issues concerning the modified design need to be evaluated; however, preliminary evaluations are encouraging.

The design and evaluation of the use of a scrubber liquids vacuum flash unit is also complete. Results indicate that the economic delta between once-through and recycle coking may be reduced by approximately two-thirds. Key technical issues such as coking of the scrubber liquids in the vacuum flash preheat furnace must be investigated.

Future work will concentrate on further identifying the technical issues to be resolved, and better identifying the economic incentives associated with each area.



### 3.4 Coal FLEXICOKING

The engineering evaluation of feeding a mixture of coal and vacuum bottoms to a FLEXICOKING unit to increase liquid yields and fuel gas production for the EDS liquefaction plant is continuing. Development of a vector for the Process Alternatives Model (PAM) to evaluate the economic and process impact of feeding supplemental coal to the FLEXICOKING unit is underway.

Several technical problems specific to the feeding and operation of a coal FLEXICOKING unit are being addressed. One of these is the coal feed system. The selected design consists of a dense phase feed system, which would inject the coal directly into the dense bed of the reactor. This design enables variations in coal feed rate to be handled.

In the current SDU, coal boilers are used together with gas fired boilers to produce steam, and to balance fluctuations in FLEXICOKING LBG fuel production. The use of coal FLEXICOKING would eliminate the need for these offsite coal boilers, and would require another method for handling the variations in FLEXICOKING LBG production. The use of gas fired turbines to produce electricity is the proposed means of controlling these fuel fluctuations.

Work is progressing on the development and implementation of vectors into the PAM.

### 3.5 Deep-Cut Vacuum Fractionation

As part of ongoing cost reduction studies, the feasibility of and incentives for recovering additional heavy liquid product from Illinois vacuum bottoms in the vacuum pipestill (VPS) is being updated. Earlier studies were carried out to scope the overall feasibility of deep-cut VPS operation with Illinois coal (Quarterly Technical Progress Report, FE-2353-2, January-June, 1976). Based on these studies, and subsequent bottoms viscosity data and bottoms pumping requirements, the maximum VPS vacuum bottoms cut point was defined for the Market Sensitivity Case (MSC) EDS Study Design Update (Quarterly Technical Progress Report FE-2893-12, January-March, 1978). Studies to estimate the incentive for a deeper-cut VPS operation were also reported (Final Technical Progress Report FE-2353-20, January, 1976 - June, 1977).

The present update confirms the relatively large economic incentive shown in previous studies for maximizing VPS cut point prior to partial oxidation (POX) or other non-FLEXICOKING bottoms processes. This update is based on the current bottoms distillation characterization curve used in the Market Sensitivity Case (Figure 3-4). Figure 3-4 was developed in conjunction with the EETD bottoms characterization program using Hi-Vac C distillation data. Using this distillation data, we confirmed that the 975°F vacuum bottoms cut point used in the Market Sensitivity case for liquid feed to the POX system appears to be the operable limit as determined by current pumping limitations. This is based on a vacuum bottoms viscosity of about 50 poise (at a shear rate of 200 sec<sup>-1</sup>) set by POX feed holding requirements and high-discharge pressure pumping considerations. The current study was also extended to define the maximum probable VPS severity limited by preheat furnace constraints and viscosity limitations within the VPS itself. On the basis of VPS furnace and flash zone operability, a nominal 1050°F cut point may be attainable. A VPS operation of this type would bring VPS yields to within 3-4 wt% on dry coal relative to SDU Base Case liquid yields (Figure 3-5). Economics screening studies indicate about a 3 \$/B RISP cost reduction if all bottoms were cut at 1050°F rather than 975°F.

The expected viscosity of 1050°F+ Illinois vacuum bottoms would be in the range of 10-20 poise (at a shear rate of 200 sec<sup>-1</sup> and 750°F). If cooling of these bottoms to 600°F for feed storage ahead of a POX unit is required, the viscosity would likely be unacceptably high, or the bottoms may even solidify. Therefore, feed to POX as a liquid would require holdup at a temperature above 700°F to maintain the viscosity at the 50 poise pumpability limit, and consequently, the holdup time would have to be reduced to only several minutes to prevent bottoms degradation. If this were not feasible, or if a high-discharge pressure could not be developed to handle a significantly more viscous vacuum bottoms stream, a 1050°F cut point operation would require vacuum bottoms solidification facilities with subsequent solid rather than liquid feed to POX. The potential coal liquid cost reduction based on a 1050°F cutpoint VPS operation would have to be debited for the cost of solidification and grinding facilities. These costs are currently being developed.

Figure 3-4

EDS STUDY DESIGN UPDATE-MARKET  
SENSITIVITY CASE-VACUUM PIPESTILL FEED

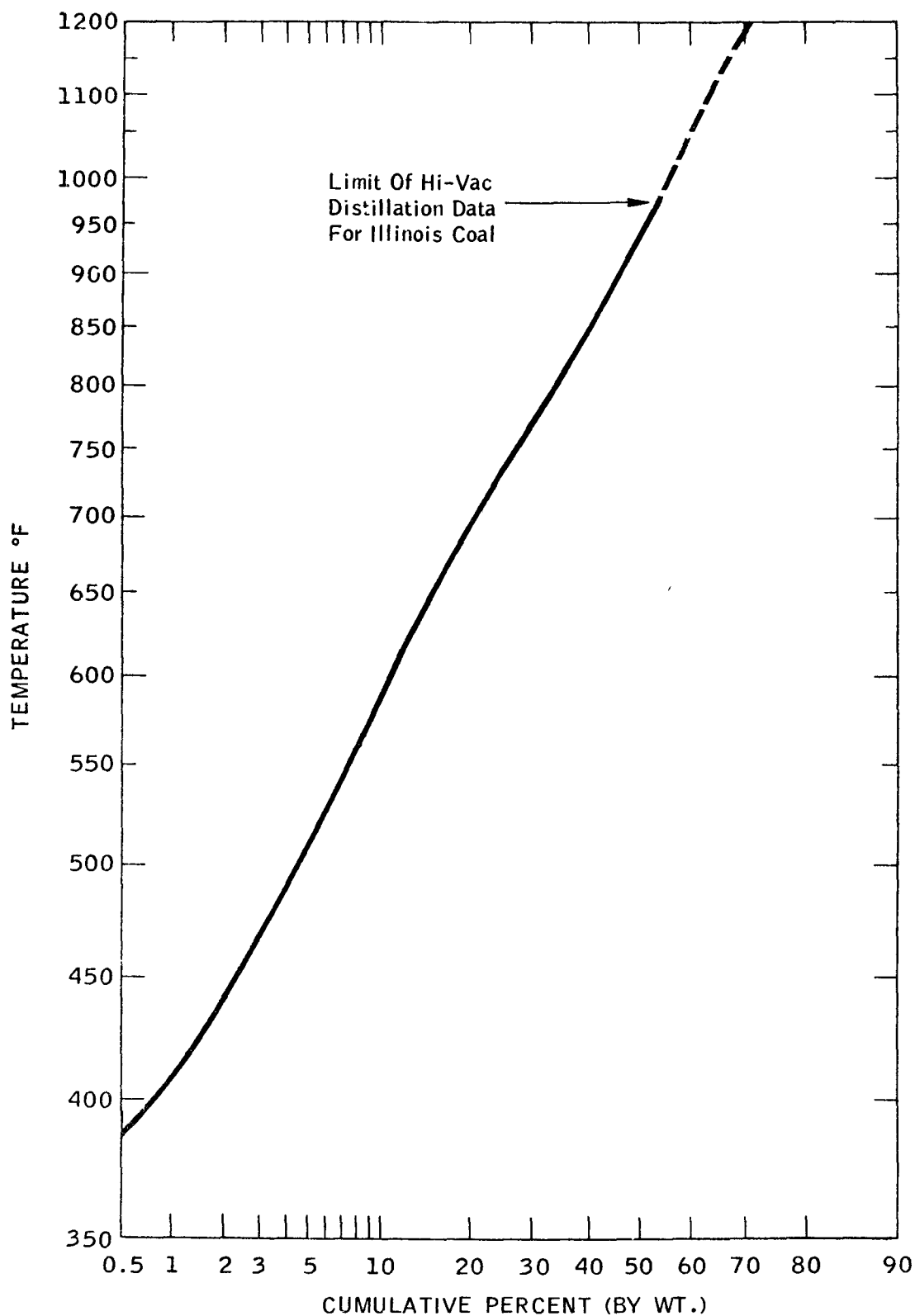
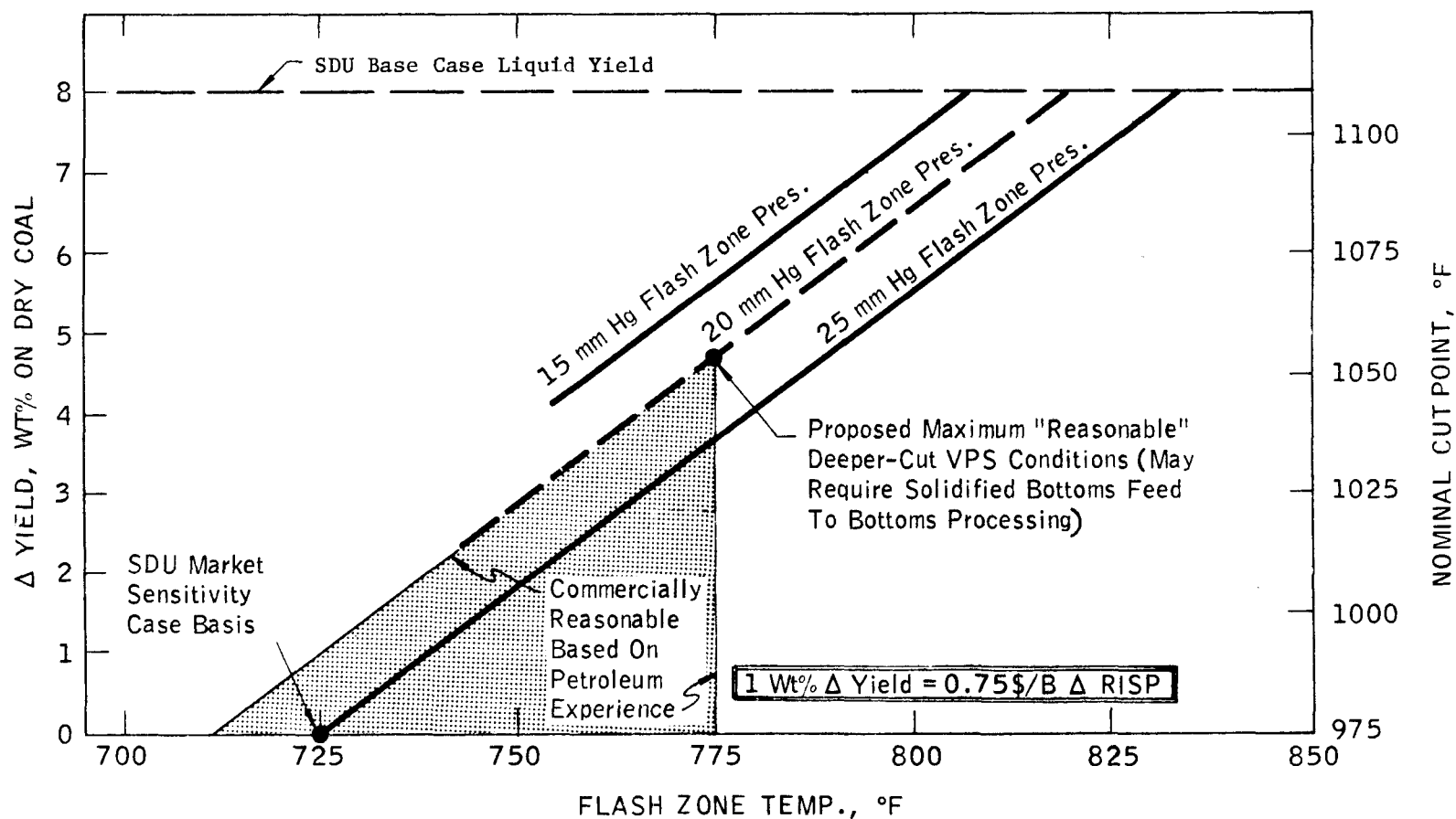


FIGURE 3-5

ADDITIONAL LIQUID YIELD FROM DEEPER  
VPS CUTPOINT RELATIVE TO EDS  
SDU MARKET SENSITIVITY CASE CONDITIONS

- Basis:
- SDU MSC VPS Feed Distillation Curve (Illinois Coal)
  - All Bottoms Deep Cut (i.e. No FLEXICOKING)
  - 2 LV% Unrecovered Overflash



To achieve a 1050°F vacuum bottoms cut point, VPS flash zone conditions of 775°F, 20 mm Hg, with 2 LV% overflash are required in a single-stage, dry VPS operation (i.e., no steam stripping). A two-stage VPS system was also considered as a means of increasing the 1050°F cutpoint, but the small additional yield recovered was judged to be insufficient to pay for the cost of the additional tower and ejectors. Also, a steam-stripped rather than dry VPS was considered. The dry operation is preferable because it is capable of recovering more liquid, has lower operating costs, and does not require stripping trays in the bottom of the VPS, which are undesirable for high-viscosity, solids containing bottoms. The selected flash zone conditions are within the upper range of petroleum-based experience. They compare to 725°F, 25 mm Hg, and 2 LV% overflash flash zone conditions required to attain the 975°F VPS cut point in the Market Sensitivity case. If coal liquids are no more susceptible to coking and thermal cracking than petroleum residue, VPS operating conditions required to achieve the 1050°F cut point should allow acceptable commercial operability, based on these constraints.

To achieve the 1050°F nominal bottoms cut point, the VPS furnace requires a coil outlet temperature (COT) of about 830°F under mixed-phase flow conditions. This temperature is somewhat higher than for typical petroleum VPS furnaces. Therefore, some uncertainty exists as to whether a typical mixed-phase outlet VPS furnace can operate at 830°F with acceptable decoking intervals. An alternative operation which may alleviate the potential coking problem would employ single-phase liquid furnace outlet conditions at 860°F COT to attain the same VPS flash zone conditions. If the coking tendency of the coal derived VPS feed is no worse than for petroleum VPS feed, there is a good probability that a commercially-operable furnace can be designed to achieve a nominal 1050°F bottoms cut point.

A brief evaluation of the potential for deeper-cut of Wyoming vacuum bottoms was also carried out. The major drawback with Wyoming bottoms is their higher viscosity, which can range from two to more than five times greater than for Illinois bottoms for a given nominal cut point. For the Wyoming Study Design Update, a nominal cut point of 900°F (rather than the 975°F cut point for Illinois bottoms) is required to provide 50 poise viscosity at the inlet to the POX feed pumps. Conceivably, a nominal cut point of 1050°F would be possible for Wyoming bottoms with respect to furnace and VPS limitations, but the viscosity would almost certainly rule out a liquid feed to subsequent POX or other bottoms processing steps. The viscosity exiting the VPS would be in the range of 100 poise (at a shear rate of 200 sec<sup>-1</sup>) which would allow no room for cooling for even minimal POX liquid bottoms feed holdup. Therefore, solidification and grinding facilities would be required.

An experimental verification program is under consideration to compare coking tendencies of coal to petroleum-derived VPS feeds. Because these data would only provide general qualitative tendencies rather than quantitative rate comparisons of coke formation, they will permit only a general assessment of the maximum commercially-operable furnace severity. ECLP testing would be required to develop a commercial design basis.

### 3.6 Burning Star Coal Screening Study - Bottoms Processing

Bottoms processing vectors for once-through and recycle FLEXICOKING have been developed for the engineering screening study of Illinois No. 6 (Burning Star No. 2 mine) coal. Process Alternative LP Model (PAM) vectors have been prepared utilizing Continuous Stirred Coking Unit (CSCU) data as well as the latest investment information on FLEXICOKING. The initial CSCU data was obtained with vacuum bottoms feed produced at liquefaction severities of 840°F/40 minutes residence time and 880°F/25 minutes residence time. FLEXICOKING vectors will be prepared at other liquefaction conditions when data from additional CSCU runs have been analyzed.

EDS operation with Burning Star coal exhibits a different yield pattern than operation with Illinois No. 6 coal from a Monterey No. 2 mine. At severities of 840°F/40 minutes residence time, liquid yields from liquefaction decreased from 35 to 24 wt% on dry coal when feeding Burning Star rather than Monterey coal. Lower liquefaction yields, however, are offset by increased liquid recovery from FLEXICOKING. FLEXICOKING yields increased by 9 wt% on dry coal (from 10 to 19 wt%) with Burning Star relative to Monterey.

Modifications have also been made to the Monterey partial oxidation vectors to account for differences in fuel gas and hydrogen yields when feeding Burning Star mine coal. These vectors are being used in PAM along with liquefaction vectors at various operating conditions to determine the most favorable plant configuration. Three configurations are being considered: FLEXICOKING for fuel/steam reforming for H<sub>2</sub>, FLEXICOKING for fuel/partial oxidation for H<sub>2</sub>, and partial oxidation for fuel/H<sub>2</sub>.

### 3.7 FLEXICOKING Prototype Unit Revamp Planning Study

For the period from July 1, 1978 to June 30, 1979, work proceeded on the EDS FLEXICOKING Prototype Unit (EDS-FPU) revamp project. Screening studies were initiated to develop a preliminary operating program for the EDS-FPU and to define preliminary heat and material balances covering operations on Illinois and Wyodak coal liquefaction vacuum bottoms. As an outcome of initial onsite screening studies, the fresh feed rate to the Prototype was set at 70 T/D. From laboratory studies identifying limiting liquid feed storage time/temperature/viscosity relationships, the desirability of decoupling the EDS-FPU from the ECLP unit was identified as a significant issue. Therefore, additional screening studies were carried out to scope out the design of feed remelt facilities. These studies identified a feed remelt system which required diluent as the melting medium. This flux, when combined with the pump seal oil, could amount to about 40% of the total feed, and a recovery system is required to minimize makeup requirements by recycling the flux and seal oil.

Gasification kinetics were also evaluated to ensure that prototype gasifier conditions would adequately simulate commercial conditions. Factors such as gasifier temperature, gas velocity, and steam rate were considered as a part of this evaluation. These studies completed the process planning for the EDS-FPU revamp and were used to set the basis for the Class V Update cost estimate.

In 4Q78, Exxon Engineering Project Management (EPPM) completed the Class V Update for the EDS-FPU revamp project showing an increase in estimated total erected cost (TEC) from 23 M\$ as shown in the original class V estimate to 33.8 M\$. This updated TEC reflected the results of the feed system alternative study. The cost increase reflected a better understanding of the feed properties and a resulting increase in the complexity of the feed storage and handling requirements. A Revamp Task Force panel was then convened to consider cost reductions to the original plan of recommissioning the Prototype FLEXICOKING Unit. A Grass Roots Task Force considered the possibility of achieving the program objective with a smaller 10 T/D mini-plant.

The Revamp Task Force identified several potential areas of cost reduction in the onsites, offsites and project execution categories (discussed in detail in the October-December, 1978 Quarterly (FE-2893-25)). At the same time, the Grass Roots Task Force developed a screening quality design for the 10 T/D mini-plant, including major vessel dimensions, elevations, lines, along with heat and material balances. From this design the task force evaluated the potential benefits of building and operating the mini-plant as opposed to the EDS FLEXICOKING Prototype Unit. The Grass Roots Task Force assessed the probability of developing significant useful scale-up information from the 10 T/D plant in critical areas of slag formation and particle behavior. Overall operability was also evaluated. In all these areas, the 70 T/D revamp was judged to be superior to the 10 T/D plant.

With the results of the two task force efforts in hand, the decision was made to proceed with the revamp effort, utilizing the modifications and cost reductions detailed in Table 3-3. In addition, it was decided that a project cost control procedure be implemented to monitor costs as the design work proceeded.

Work on the onsite Design Basis Summary (DBS) for the EDS FLEXICOKING Prototype Unit revamp was resumed with the completion of the task force efforts in mid-November. Two design base cases were selected which, together with design contingencies, cover the range of expected prototype operation. These cases were based on Illinois coal liquefaction bottoms data with once-through scrubber bottoms operation (Case 1) and Wyodak coal liquefaction bottoms data with recycle scrubber bottoms operation. A short summary of the design cases is included on Table 3-4.

Screening studies of the EDS FLEXICOKING Prototype Unit Product Recovery facilities were completed in preparation of the DBM. These studies are discussed in detail in the October-December, 1978 Quarterly Technical Progress Report (FE-2893-25). The product recovery block will play a critical role in decoupling the EDS-FPU from ECLP facilities since the remelting of solidified vacuum bottoms will require a lighter, less viscous diluent. The simulation cases considered were the two basis cases for the EDS-FPU: Illinois once-through operation at a 20 wt% feed diluent level and a Wyodak Recycle operation at a 30 wt% feed diluent level. Results of the study were used to scope the quantity of diluent (Heavy Raw Creosote Oil) needed for makeup and to provide flowrate, temperature, and pressure information for the design engineers.

In order to determine air and water pollution abatement facility design, levels of contaminants in the gas and water streams were estimated using laboratory data. Current plans call for sending solids-free sour water to the Baytown Refinery's sour water treating system and the solids laden sour water to a slurry stripper and then send the stripped slurry to the refinery settling pond. The technique used in characterizing the gas and water pollutant levels is discussed in the October-December, 1978 Quarterly Technical Progress Report (FE-2893-25). Once the gas streams were characterized, sour water estimates were made. Sour water formed as a result of water condensation was also characterized. The results of this study were included in the Design Basis Memorandum (DBM) for the EDS-FPU revamp project.

The basis for the slurry stripping facilities was set following a cooperative effort by ER&E's EDS Liquefaction Engineering Division and the Baytown refinery to define facilities to handle slurry products from the EDS-FPU. Based on the current refinery slurry and wastewater handling requirements, it was recommended that the  $\text{NH}_3$  content of the slurry streams be reduced by 99% to avoid adverse environmental effects. This sets the design of the EDS-FPU slurry stripper and was included in the DBM.

Concurrent with the ELED/Baytown Refinery efforts to design slurry handling facilities, a joint effort was undertaken by ELED, and EEPD's Gas Treating Section to define facilities for meeting the refinery sulfur



TABLE 3-3  
FLEXICOKING PROTOTYPE REVAMP  
COST REDUCTIONS

Item	Recommended Cost Change, M\$	Comment
<u>Onsite/Offsite Design Base</u>		
Eliminate ECLP Electrical Sub-station	(1.45)	• Reduced load resulting from deleted and relocated equipment
Eliminate Feed System Dust Control Equipment	(1.30)	• Lab data indicates no respirable dust problem
• Reduced Dowtherm System	(0.90)	• Eliminate need to trace flush, seal lines by using creosote oil in place of VGO
• Eliminate Sandvik Belt	(0.60)	• Dispose of excess FXC scrubber bottoms to spare refinery tank
Optimize remelt system including creosote oil as feed system flux	(0.55)	• Results in smaller mixers and remelt tanks
Others	(0.25)	• Net of several cost increase/decrease items
Subtotal	(5.05)	
<u>Project Execution</u>		
Engineering/construction by local Houston-area contractor	(3.4)	• Based on Carter/EUSA experience at Baytown
• Change in project management approach	(1.4)	
Others	(0.2)	• Net of several cost increase/decrease items
Subtotal	(5.0)	
Total	(10.1)	

TABLE 3-4

EDS FLEXICOKING PROTOTYPE CASE DESCRIPTION

	<u>Case 1</u>	<u>Case 2</u>
Coal Feedstock	Illinois	Wyodak
Mode of Operation	Once-Through	Recycle
Gasifier Process Steam	Low	High
Coke Gasified, #/hr	2563	3504
Purge Coke, #/hr	1687	1628
% Gasification on Ash-Free Coke Make	82.0	86.6

specification of 160 volume parts per million (vppm) in the FLEXICOKING unit's low Btu Gas (LBG). Various treating schemes were evaluated which use different scrubbing media and process configurations. The choice of scrubbing media was limited to those materials used by the Baytown Refinery in its gas treating processes. This work shows that conventional scrubbing with trays or packing in the existing absorber require vessel modifications to achieve 160 vppm Sulfur.

An evaluation of the refinery H<sub>2</sub>S removal tailgas cleanup facilities revealed that a slipstream of the selected H<sub>2</sub>S removal solution would not be available. Therefore, the DBS for the gas treating facilities is based on the need for a self-contained, "stand-alone" H<sub>2</sub>S removal plant. The DBS for the H<sub>2</sub>S removal facilities was prepared by ER&E's Gas Treating Section separate from the overall onsite DBM and was released in final form late second quarter, 1979.

In late December, 1978, a working copy of the onsites DBM was circulated to the petroleum process designers, petroleum process planners and the Baytown and Baton Rouge research labs for review. Feedback from the review of the DBM was incorporated into the preliminary draft of the DBM which was released late January, 1979. The goal of the preliminary DBM was to allow the design engineers to begin preparation of the onsites Design Specification. The finalized DBM was released early in the second quarter of 1979, and contained information released with the preliminary DBM plus bases for gas treating and slurry stripping facilities. With the final DBM in hand, it is anticipated that the design engineers will have the onsites Design Specification completed in the third quarter of 1979.

Concurrent with this effort, work was done to prepare an Investment Basis Memorandum (IBM) for the onsite block of the EDS-FPU revamp. Equipment to be added to the existing unit was identified and sized, while changes to the existing plant layout and piping were developed. A preliminary draft of the IBM was issued in late first quarter, 1979 for review by Exxon Research and Engineering personnel in the areas of safety, layout, onsite design, cost and scheduling, and project management. Feedback from this review was incorporated into a finalized IBM which was released early in the second quarter of 1979. The document itself provides the information necessary for the preparation of the Class IV cost estimate.

Work on the offsite facilities for the EDS FLEXICOKING Prototype Unit proceeded in parallel with the onsite work. Studies were begun in late 4Q78 to define the feed system, including requirements for solid vacuum bottoms storage, handling, and remelting. Environmental and industrial hygiene considerations played an important role in the choice of a facilities configuration. Various feed piping heat tracing systems were studied. A heat medium system was designed for remelter heating and line tracing. Utilities requirements for the onsites and offsites areas were developed. An Offsite Facilities Definition for the EDS FLEXICOKING Prototype Unit was issued on June 14 which incorporates the results of this work. This information will be used in preparation of the Class IV cost estimate.

As part of the EDS FLEXICOKING Prototype Revamp Project, work proceeded on developing a technical operating plan. The final release of the operating plan is targeted for late 4Q79. Areas to be addressed by the operating plan include gasifier ash sintering, particle properties, and coker scrubber liquids solids content. Recommendations for test programs have been solicited from various Exxon organizations. A preliminary test program submittal was made by various EETD sections. These test programs address areas of uncertainty such as particle properties, materials performance, scrubber liquids solids removal, waste stream compositions, and heat exchanger and pump operating data. The proposals are currently under review. A preliminary selection of test programs to be included in the overall operating plan will be made in 3Q79.

In preparation for the building permit application, the EDS Liquefaction Engineering Division and the General Engineering Division assembled an information package to be used by the Carter Oil Company to prepare the application for the Texas Air Control Board (TACB) permit. This package contained information on the types and sources of air pollutants which will be emitted from both the onsite and offsite portion of the EDS-FPU. Initially, a preliminary package was sent to the Carter Oil Company for their review in mid second quarter, 1979. Carter Oil Company feedback was then incorporated into the final information package with a release late in second quarter of 1979. The permit application was then prepared by Carter Oil and submitted to the TACB where it is currently under review. A response to the permit is anticipated during the third quarter of 1979.

### 3.8 Engineering Studies of Bottoms Processing/Hydrogen Manufacture

#### Economic Comparison of Texaco and Shell-Koppers Partial Oxidation on EDS Vacuum Bottoms Feed

An initial economic screening comparison of Texaco and Shell-Koppers Partial Oxidation (TPO/SPO) processes on liquid EDS vacuum bottoms indicates that TPO is 5 - 7% less expensive for producing hydrogen and fuel gas, respectively. This cost advantage for TPO corresponds to about 0.75 \$/B and about 0.25 \$/B respectively, for hydrogen and fuel gas on an average EDS liquids cost basis. When compared on solidified vacuum bottoms rather than liquid feed, SPO appears to have roughly a 1 \$/B liquid product cost advantage for hydrogen generation because of TPO's aqueous slurry feed requirement compared to dry feed used by SPO. The magnitude of these cost impact differences for liquid vacuum bottoms feed are expected to decrease based on revised investment requirements for SPO expected from Shell in 3Q79. Therefore, in light of the screening quality of the economic comparison, we have concluded that these two partial oxidation processes feeding liquid vacuum bottoms are an economic standoff in the context of an EDS process application, while for solidified vacuum bottoms feed, SPO appears to have a slight cost advantage.

#### Evaluation Based on Non-Confidential Information from Texaco and Shell

This initial economic evaluation is based on ER&E's interpretation of non-confidential, screening quality process and economic information supplied by both Texaco and Shell. This information was presented in the Quarterly Technical Progress Report\* for January - March, 1979, and is summarized in Table 3-5. This summary table, as well as this evaluation, reflects recent information from Shell in which their investments are decreased by 13% and their power requirement are decreased by 85% relative to their original basis to be consistent with the Texaco basis. Additional cost information is expected to be received from Shell in 3Q79, and is expected to show a further reduction in SPO investment requirements relative to their original basis.

This economic comparison stressed hydrogen generation from liquid vacuum bottoms, but production of intermediate Btu fuel gas was also evaluated in somewhat less detail, as was the impact of a solidified vacuum bottoms feed. Although this comparison was based on Illinois coal-derived vacuum bottoms feed, the results should be applicable for vacuum bottoms derived from a wide range of coals.

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\*FE-2893-29

TABLE 3-5

SUMMARY OF TEXACO AND SHELL-KOPPERS  
PARTIAL OXIDATION INFORMATION  
FOR HYDROGEN PRODUCTION

Basis:   ● 150 MSCF H<sub>2</sub> + CO/SD syngas production  
          ● Illinois coal derived, liquid vacuum bottoms feed

	<u>Texaco</u>	<u>Shell-Koppers</u>
<u>Process Feed Rates, T/Hr</u>		
● Vacuum Bottoms	93.0	93.8
● Steam	37.2	-
● Boiler Feed Water	-	16.4
● Oxygen	73.3	73.9
<u>Yields</u>		
● H <sub>2</sub> /CO Ratio	0.64	0.65
● Carbon Utilization, %	99.2	98.0
● Syngas Pressure, psig	960	425
<u>Investment</u>		
● Investment, M\$	9	35
- No. of Trains	1	2
- Gasifiers per Train	2	1
- Contingency, %	0	10
- Year	Jan. '77	Mid '78
<u>Utilities</u>		
● Power, MW	1.2	2
● Steam, T/Hr	-	22

### Processes are Generally Similar

The Texaco and Shell-Koppers processes are similar in concept. Both feed vacuum bottoms with oxygen and steam into a reactor at elevated temperature and pressure. A syngas consisting primarily of hydrogen and carbon monoxide is produced, which can be upgraded to hydrogen, or cleaned and used as an intermediate Btu fuel gas. A flow schematic comparing the two processes for hydrogen production is shown in Figure 3-6. The principle differences between the processes are (1) TPO's higher operating pressure (1,000 vs 450 psig), (2) TPO's use of a refractory liner versus a water jacketed reactor, (3) SPO's ability to feed solid vacuum bottoms as a dry, ground solid versus an aqueous slurry, and (4) SPO's use of a cyclone in the overhead solids recovery system, which allows disposal of soot as a dry stream compared to aqueous slurry from the TPO process.

### Hydrogen from Liquid Vacuum Bottoms 5% Cheaper via Texaco

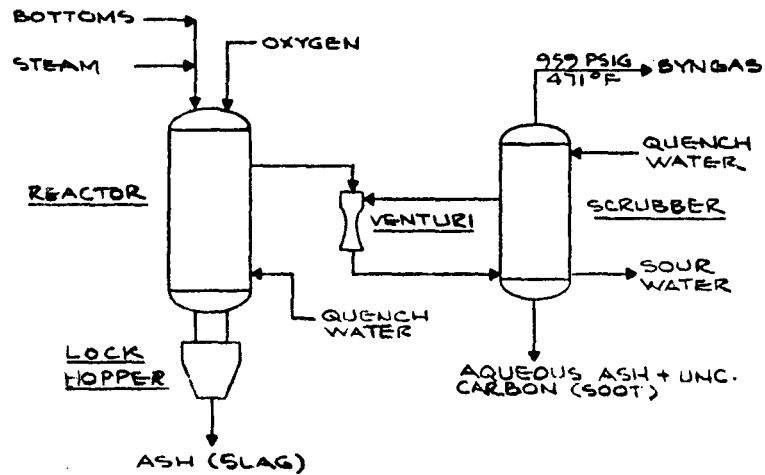
Our initial economic screening comparison indicates a 5% cost advantage for TPO vs SPO for producing hydrogen from a liquid vacuum bottoms feed. This cost advantage for TPO corresponds to a 0.75 \$/B reduction on an average EDS product cost basis.

A summary of the cost differences between TPO and SPO is shown in Figure 3-7. These are based on a typical application of both processes to the EDS process for hydrogen generation, based on EDS Study Design Update requirements. The cost comparison is shown relative to an assumed 45 \$/B base (1985\$) liquids cost for a TPO hydrogen production source. This base liquids cost is not necessarily representative of the cost expected for the EDS Study Design Update Market Sensitivity Case which employs TPO for hydrogen production. It is meant to serve as a rough estimate for purposes of this comparison only. Overall, the credit for TPO relative to SPO is on the order of 5% lower hydrogen cost, which translates to a 0.75 \$/B reduction in the overall liquid product cost.

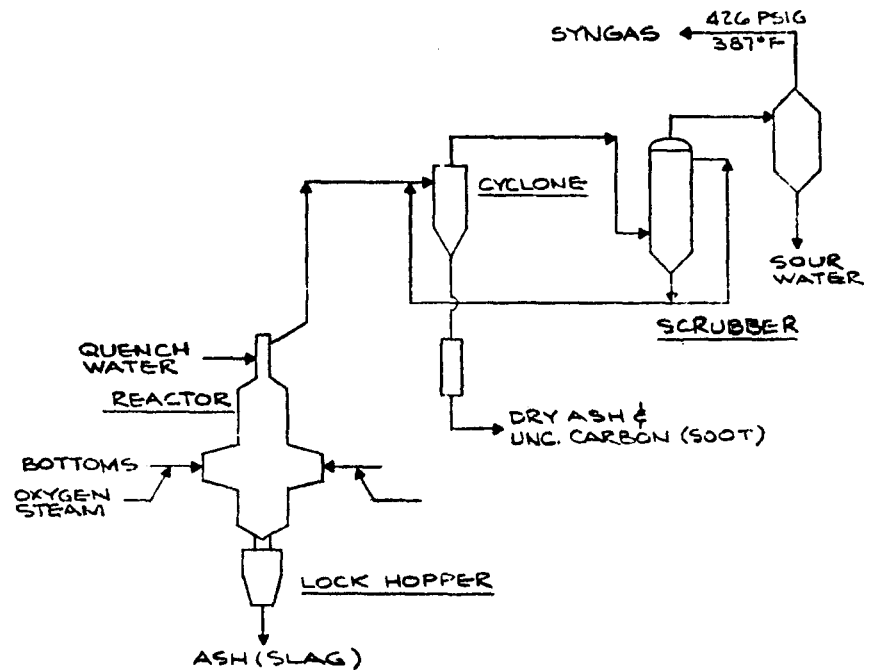
ER&E's adjustment of Texaco's and Shell's data are presented in Table 3-6, which shows the comparison based on cash flow impact and required initial selling price of the EDS product liquids (relative to a 15% DCF return). The major component of SPO's higher cost relative to that for TPO is SPO's greater investment, and the additional hydrogen compression required because of SPO's lower operating pressure. The investment, after adjustment by ER&E to a consistent basis, is over twice as great for SPO than for TPO. The less significant cost components which appear more expensive for SPO are repair materials (investment related), greater oxygen and bottoms feed requirement (resulting from a slightly lower carbon utilization efficiency), salaries and related costs, and power consumption.

FIGURE 3-6

TEXACO



SHELL - KOPPERS



EXXON RESEARCH & ENGINEERING CO.			
SYNTHETIC FUELS ENGINEERING			
P.O. BOX 161 - FLORENCE, PA. 15033			
TEXACO AND SHELL - KOPPERS			
PARTIAL OXIDATION FLOW SCHEMES			
FOR SYN GAS PRODUCTION FOR			
HYDROGEN FROM VACUUM BOTTOMS			
GPW/HLC	MAR. 21, 1979		79-474-2



FIGURE 3-7  
SHELL-KOPPERS/TEXACO PARTIAL OXIDATION COST  
DIFFERENTIAL SUMMARY FOR HYDROGEN PRODUCTION  
BASED ON EDS STUDY DESIGN UPDATE REQUIREMENT

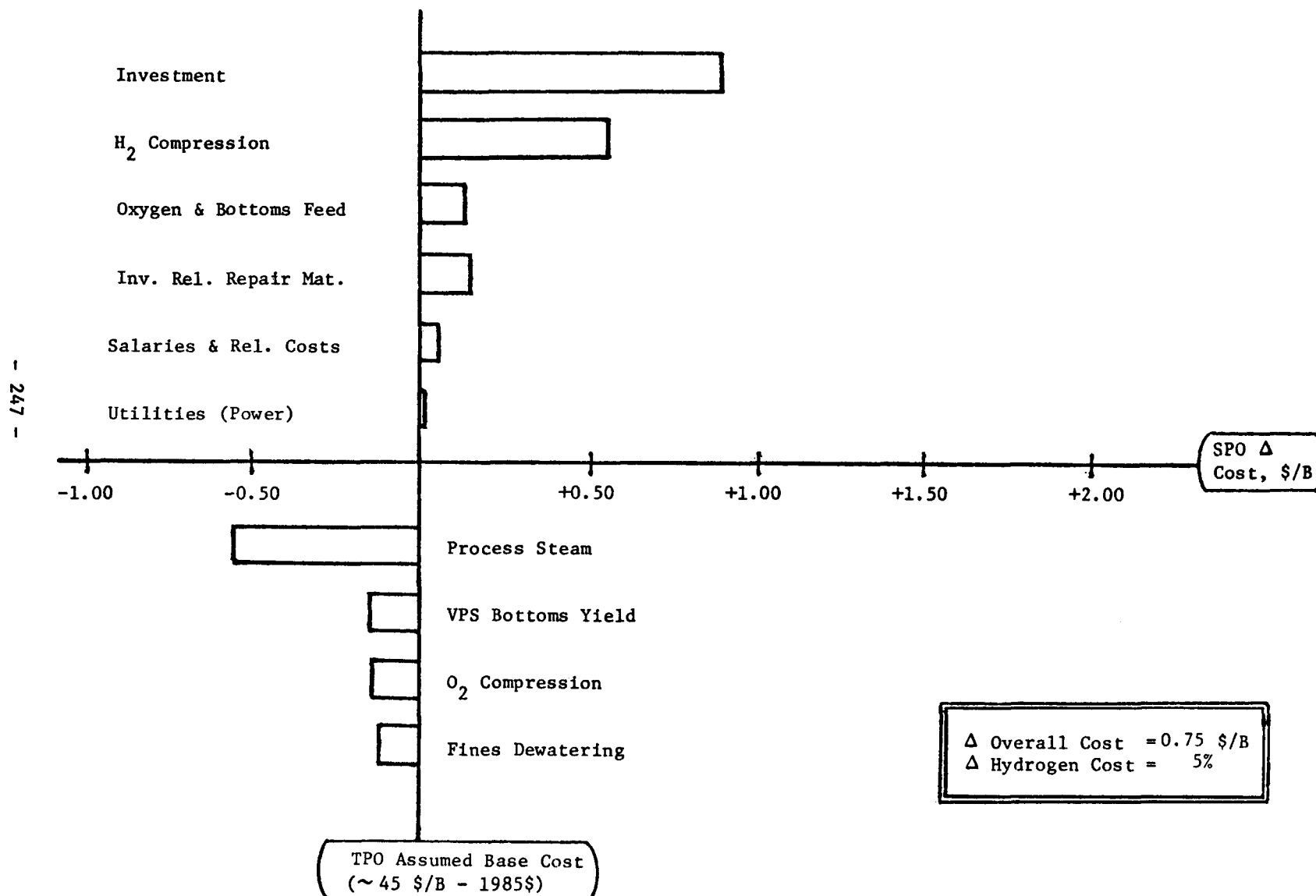


TABLE 3-6

SHELL-KOPPERS/TEXACO PARTIAL OXIDATION  
COST DIFFERENTIAL  
FOR HYDROGEN PRODUCTION

Basis: • 4 trains each 94 MSCF H<sub>2</sub> + CO/SD based on EDS  
Study Design Update requirements  
• Liquid vacuum bottoms feed  
• Economics in 1985 \$

<u>Debits for Shell-Koppers</u>	<u>Shell-Koppers</u>	<u>Texaco</u>	<u>Cost, M\$/Yr</u>
<u>Investment, M\$</u>			
- As Received (1)	35	9	-
- Adjusted (Texaco) (2)	21.5	7.7	-
- Adjusted (ER&E/Texaco) (3)	21.5	10.0	-
- SDU Basis (ER&E/Texaco) (4)	187	87	17.2
<u>H<sub>2</sub> Compression</u>			
- Pressure, psig (in/out)	400/1860	880/1860	-
- Investment, M\$	100	70	6
- Power, MW	42	25	4.2
<u>Utilities</u>			
- Power, MW (@3.5 ¢/kwhr)	5	3	0.5
- Steam, T/hr	50	0	0 (5)
<u>Investment Related</u>			
<u>Repair Materials, M\$/Yr (6)</u>			
- SDU Basis (ER&E/Texaco)	6.2	2.9	3.3
<u>Oxygen/Bottoms Feed (7)</u>			
- Oxygen, T/hr (@53 \$/T)	185.7	184.2	0.6
- Bottoms, T/hr (@47 \$/T)	235.8	233.7	0.7
<u>Salaries and Related Costs</u>			
- Operators Required (8)	9	4	0.7
<u>Acid-Gas Cleanup</u>			
- Maximum Benefit of Higher Texaco Pressure, M\$/yr	Base	-6	<u>0 (9)</u>
 TOTAL DEBITS			 33.2

TABLE 3-6 (cont'd)

SHELL-KOPPERS/TEXACO PARTIAL OXIDATION  
COST DIFFERENTIAL  
FOR HYDROGEN PRODUCTION

---

<u>Credits for Shell-Koppers</u>	<u>Shell-Koppers</u>	<u>Texaco</u>	<u>Cost, M\$/Yr</u>
<u>Process Steam</u>			
- Steam Req'd., T/hr (@ 8 \$/1000 lbs)	0 (10)	93.5	(10.4)
<u>VPS Bottoms Yield (11)</u>			
- Additional Yield, wt% Dry Coal	+0.25	Base	-
- Cut Point, °F	+10	Base	-
- Value, M\$/Yr	-	-	(3)
<u>Oxygen Compression</u>			
- Pressure, psig (in/out)	1/500	1/1100	-
- Investment, M\$	-7	Base	(1.5)
- Power, MW (@ 3.5 ¢/kwhr)	-4.9	Base	(1.2)
<u>Fines Dewatering (12)</u>			
- Fines Produced	Dry	Slurry	
- Dewatering Inv. M\$	-	+9.7	(2.2)
- Dewatering Op. Costs, M\$/yr	-	+0.1	<u>(0.1)</u>
TOTAL CREDITS			(18.4)
TOTAL DEBITS-CREDITS			15
▲ HYDROGEN COST, % (13)			5
▲ PRODUCT COST, \$/B (14)			0.75

TABLE 3-6 (cont'd)

SHELL-KOPPERS/TEXACO PARTIAL OXIDATION  
COST DIFFERENTIAL FOR HYDROGEN PRODUCTION

Notes:

- (1) Includes syngas generation system only.
- (2) Shell and Texaco "as-received" investment adjusted to common basis based on ER&E's understanding of the investment bases.
  - Texaco investment adjusted from 2 gasifiers per train to 1 assuming gasifier is 50% of investment and each is 60% capacity using 0.7 plant size exponent (9 M\$ → 7.7 M\$).
  - Shell investment put on consistent basis (40 M\$ → 21.5 M\$) adjusting following:
    - Remove 10% contingency
    - Adjust from mid-78 to Jan. 77
    - Adjust from 2 trains to 1 train
    - Subtract estimated investment for sour water stripping (3.7 M\$)
- (3) Texaco "as-received" investment increased by 30% to make it consistent with other ER&E estimates.
- (4) Shell and Texaco investments adjusted to EDS Study Design Update (SDU) basis (requires 377 MSCF H<sub>2</sub> + CO/SD) as follows:
  - Multiply 7.7 M\$ adjusted Texaco basis by:
    - Gulf Coast to Illinois location (1.15)
    - Jan. 77 - Jan. 85 (1.45)
    - Four trains at 94.2 MSCF H<sub>2</sub> + CO/SD →  $\left(\left(\frac{94.2}{150}\right)^{0.7} \times 4\right)$
    - Add 20% project contingency (1.2)
    - Add 20% process development contingency (1.2)
    - Add 2 spare gasifiers (1.25)
  - Multiply SDU basis Texaco estimate by 21.5/7.7 ratio of adjusted Shell/Texaco investments to get SDU basis Shell investment.
  - Multiply investment difference by 0.172 capital recovery factor for 15% DCF return to get cost in M\$/Yr.
  - Increase SDU basis Texaco investment by 30% as in Note 3.
- (5) Shell process not debited for utility steam. This steam estimated to be used for sour water stripping, which is not included in Texaco's basis.
- (6) At 3.3% of investment.

TABLE 3-6 (cont'd)

Notes:

- (7) Due primarily to carbon utilization, which is 98% for Shell and 99% for Texaco.
- (8) Salaries and related costs assumed to be 50% greater for Shell than for Texaco, in light of the higher reported operator requirements.
- (9) Since actual credit is uncertain depending on optimal process at each pressure, this benefit for Texaco was not included in overall total.
- (10) Shell understood to generate process steam in water-cooled reactor walls.
- (11) Shell reports 15-20 minute vacuum bottoms feed holdup time, while Texaco reports 2 hour holdup time is required. Due to bottoms viscosity increase in storage, the reduced holdup time allows deeper vacuum tower cut point while achieving the required bottoms feed pump inlet viscosity.
- (12) Texaco is debited for producing aqueous slurry fines which are assumed to require dewatering prior to disposal. Filtration system costs included for Texaco.
- (13) Based on preliminary Texaco generated hydrogen cost.
- (14) Based on assumed 45 \$/B product liquids cost with Texaco hydrogen generation.

The principal credit for SPO is the internal generation of process steam in the water-cooled reactor, in conjunction with lower process steam requirements. This credit cancels out the hydrogen compression cost debit for SPO. Also, because of Shell's lower anticipated vacuum bottoms holdup surge requirement (15 minutes versus two hours for Texaco), less degradation (i.e. viscosity increase) of the bottoms will occur. This permits a deeper cut point operation in the vacuum tower, resulting in a slightly greater liquid yield. In addition, SPO has a small credit in lower feed oxygen compression requirements due to lower operating pressure, and a credit for producing a dry fines stream for waste disposal, which must be dewatered in the TPO process prior to disposal.

#### Shell-Koppers Shows 1 \$/B Advantage for Solidified Vacuum Bottoms Feed

When compared on solidified vacuum bottoms feed rather than liquid feed, SPO appears to have roughly a 1 \$/B liquid product cost advantage over TPO for hydrogen generation. For solid feed, the TPO process uses a water slurry of solidified bottoms, whereas SPO feeds the bottoms directly as a dry, ground solid. The injection of this water as a 50 wt% solids slurry increases the oxygen requirement for TPO by roughly 35% due to the additional heat required to vaporize the water. Also, the cold gas efficiency is released by 10% (i.e., the amount of syngas produced per unit of feed). This requires additional bottoms feed, oxygen, and larger equipment to produce the equivalent amount of syngas. Consequently, the overall debit for SPO of 0.75 \$/B for liquid vacuum bottoms feed changes to a credit of 1 \$/B for solid vacuum bottoms feed.

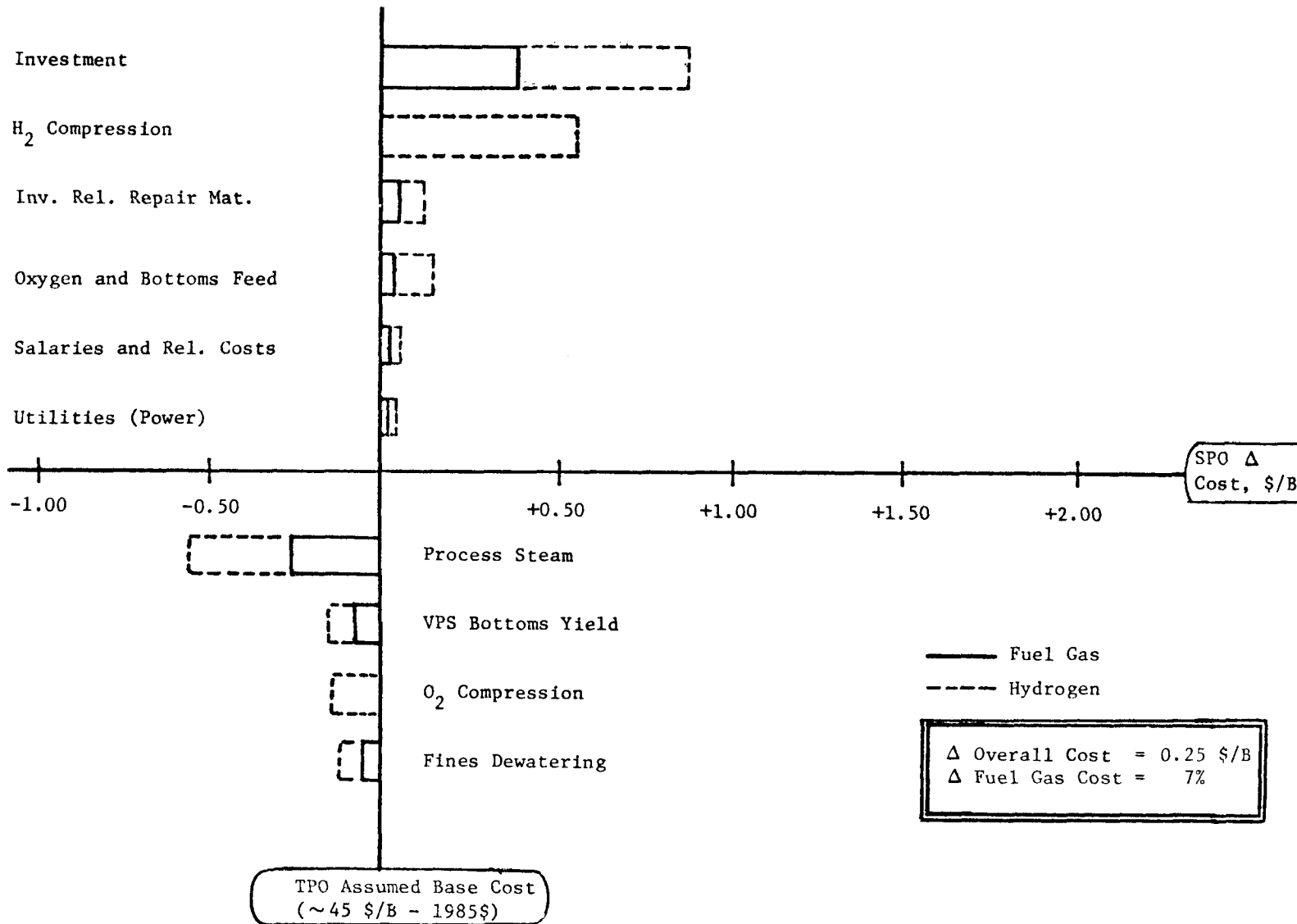
#### Fuel Gas from Liquid Vacuum Bottoms 7% Cheaper via Texaco

Although this comparison stressed hydrogen generation, fuel gas production was also considered. The results indicate that TPO is about 7% less expensive for producing fuel gas from a liquid vacuum bottoms feed. This corresponds to a 0.25 \$/B cost reduction on an average EDS liquid product cost basis. The economic comparison for fuel gas production is shown in Figure 3-8, with dotted lines representing the previous comparison for hydrogen generation.

The basis for the fuel gas production comparison is generally similar to that for hydrogen generation. The fuel gas requirement was based on producing sufficient fuel gas to meet the total onsite requirements of the EDS Study Design Update Market Sensitivity Case. Because this requirement for syngas from partial oxidation is considerably smaller than that for hydrogen generation, the cost impact of fuel gas production is less even though the differential cost per unit of fuel gas produced is 7 versus 5% for hydrogen generation. The TPO fuel gas process requirements and investments were adjusted by ER&E from the hydrogen generation case since Texaco fuel gas data were not available. Shell fuel gas requirements were used directly.

FIGURE 3-8

SHELL-KOPPERS/TEXACO PARTIAL OXIDATION COST  
DIFFERENTIAL SUMMARY FOR FUEL GAS PRODUCTION  
BASED ON EDS STUDY DESIGN UPDATE REQUIREMENT



The principal difference between production of hydrogen and fuel gas is the syngas quench system. For hydrogen generation, generally the syngas leaving the reactor is quenched with water. This water is required for subsequent carbon monoxide shift conversion. In the case of fuel gas production, this water is not desirable, so direct quench is not employed. Instead, a cooled synthesis gas recycle quench is followed by cooling in a waste heat boiler with steam generation.

The primary cost component difference is again higher SPO investment. Since fuel gas is required at a pressure well below the maximum pressure capabilities of either process, there is no compression cost difference. SPO again shows a steam credit as in the hydrogen generation case. The effect of the other components are similar, but reduced relative to hydrogen generation due to the lower syngas requirement.



## ENGINEERING RESEARCH AND DEVELOPMENT

### ENGINEERING TECHNOLOGY DEVELOPMENT

#### 4. Liquefaction Engineering Technology

##### 4.1 Coal Liquefaction VLE Data

###### Summary

The objectives of this program are to obtain VLE measurements under coal liquefaction process conditions; to analyze these data with the Chao-Seader correlation and the Redlich-Kwong-Joffe-Zudkevitch (RKJZ) equation of state; and to modify, if necessary, the VLE correlations.

We have received all the vapor pressure data on pure aromatic compounds and Wyoming coal liquid fractions. These vapor pressure data have been analyzed with the Maxwell-Bonnel correlation and the Generalized Riedel procedure. With the Generalized Riedel procedure, the vapor pressure predictions for heavy aromatics and coal liquids have been significantly improved. We have also reanalyzed the available VLE data on Illinois coal liquids by incorporating the new Generalized Riedel procedure into the Chao-Seader and RKJZ VLE correlations. Although no significant improvement was observed in the RKJZ correlation, the use of the Generalized Riedel procedure, in place of Maxwell-Bonnel, has improved the Chao-Seader correlation.

An experimental program to obtain six VLE data on mixtures of  $H_2/CH_4$  with Wyoming coal liquids is underway. For cross-checking Professor Chao's VLE measurements at Purdue University, we also initiated an experimental program with Wilco Research Company to repeat Chao's VLE measurements on mixtures of hydrogen with 1-methylnaphthalene, quinoline, and an equimolar tetralin/diphenylmethane mixture.

###### Vapor Pressure Measurements

Wilco Research Company (formerly the Thermochemical Laboratory at Brigham Young University) has completed all the vapor pressure measurements on pure aromatic compounds and coal liquid fractions. A Generalized Riedel procedure has been developed to improve the vapor pressure predictions for heavy aromatics and coal liquid fractions. It has been confirmed that the conventional Maxwell-Bonnel approach to petroleum fraction vapor pressure prediction can be significantly in error when applied to heavy aromatics, such as coal liquids, at temperatures above the normal boiling point.

Comparison of the Generalized Riedel procedure and the Maxwell-Bonnett procedure with vapor pressure on heavy aromatics is shown in Table 4-1. Table 4-1 shows that the Generalized Riedel procedure reduces the average absolute deviation from 6.30%, obtained with the Maxwell-Bonnett, to 3.70% (with literature  $T_c$  and  $P_c$ ) and 4.87% (with  $T_c$  and  $P_c$  estimated with a new procedure).

The accuracy of the Generalized Riedel procedure in predicting the vapor pressures of coal liquids has been reported in the October-December, 1978 EDS Quarterly Progress Report.<sup>(1)</sup> The results from the atmospheric bubble point to the critical point are significantly better with the Generalized Riedel procedure than with Maxwell-Bonnett. Overall deviation in vapor pressure prediction is reduced from 10.38% to 5.14% and the bias from +9.96% to +2.57%.

#### Vapor-Liquid Equilibrium Measurements

Wilco Research Company has completed the reruns of the VLE measurements on Illinois coal liquids carried out in Phase III-A. The reanalysis of the available VLE data on Illinois coal liquids by incorporating the Generalized Riedel vapor pressure procedure into the Chao-Seader and RKJZ VLE correlations<sup>(2)</sup> were discussed in the January-March, 1979 EDS Quarterly Progress Report. It indicated that the Generalized Riedel procedure significantly improved the Chao-Seader predictions; the overall deviation in predicting the weight fraction vaporized for 18 experimental points was reduced from 20.9% obtained with the Maxwell-Bonnett to 12.8%. However, no significant improvement was found in using the RKJZ with the Generalized Riedel over the RKJZ with the Maxwell-Bonnett.

Wilco Research Company has completed the six VLE runs on mixtures of  $H_2/CH_4$  with Wyoming coal liquids and three VLE runs on mixtures of hydrogen with 1-methylnaphthalene, quinoline, and an equimolar tetralin/diphenylmethane mixture. Analyses of the oil samples and data reductions remain to be done.

#### Analysis of Chao's VLE Data

As reported in the Phase III-A Final Report, 1978 Annual Report,<sup>(3)</sup> and October-December, 1978 Quarterly Report,<sup>(1)</sup> Professor K. C. Chao at Purdue University has made extensive VLE measurements on  $H_2$ /hydrocarbon and  $CH_4$ /hydrocarbon binaries as part of an EPRI-funded research project. The results of the data analysis with the RKJZ correlation were presented in the 1978 Annual Report for 8  $H_2$  binaries and in the October-December, 1978 Quarterly Report for three  $CH_4$  binaries of benzene, toluene and *n*-decane. Seven additional binaries have been investigated by Chao (3), and the results of the data analysis are summarized in Table 4-2 for  $H_2$  binaries and Table 4-3 for  $CH_4$  binaries.

(1) FE-2893-25; (2) FE-2893-29; (3) FE-2893-17

The additional systems are  $H_2$ /toluene and  $CH_4$  binaries with tetralin, 1-methylnaphthalene, diphenylmethane, m-xylene and non-hydrocarbons (quinoline and m-cresol). For all the  $CH_4$  binaries, as well as for  $H_2$ /toluene, RKJZ proved to be just as reliable a correlation as it had previously been found for the  $H_2$  binaries. Furthermore, as for the case of the  $H_2$  binaries, the optimum values of the interaction constant,  $C_{ij}$ , for the  $CH_4$ /non-hydrocarbon binaries are very close to those found for the  $CH_4$ /hydrocarbon binaries.

#### Further Work

Upon completion of the VLE measurements on the mixtures of  $H_2/CH_4$  with Wyoming coal liquids, the data will be analyzed with the Chao-Seader correlation and RKJZ equation of state using both the Maxwell-Bonnel and the Generalized Riedel procedures for vapor pressure predictions. Also the VLE data on  $H_2$  with pure components from Wilco Research Company will be cross checked against Chao's data.

TABLE 4-1

PURE HEAVY AROMATICS VAPOR PRESSURES ABOVE 1 ATMOSPHERE:  
CORRELATION DEVIATION, % AVERAGE ABSOLUTE DEVIATION

Compound	Points	Maxwell Bonnell	Generalized Riedel		Data Source
			With Literature $T_C$ and $P_C$	With New Method For $T_C$ and $P_C$	
Naphthalene	23	3.80	1.25 <sup>(a)</sup>	1.93	5,8,15,16
1-Methylnaphthalene	37	7.00	5.24 <sup>(b)</sup>	2.81	2,3,6,16
2-Methylnaphthalene	17	11.95	5.70 <sup>(c)</sup>	8.73	6,8
Tetralin	21	4.61	1.73 <sup>(d)</sup>	1.71	3,8,16
Phenylbenzene	43	6.47	2.61 <sup>(a)</sup>	2.66	4,6,8,11
Diphenylmethane	26	5.37	3.92 <sup>(e)</sup>	5.47	3,6,16
Phenylcyclohexane	7	21.73	7.26 <sup>(f)</sup>	9.36	14,16
Phenanthrene	10	3.86	6.73 <sup>(f)</sup>	5.86	12,13,16
<u>cis</u> -Decalin	28	7.85	7.01 <sup>(g)</sup>	6.99	6,16
2,4-Xylenol	35	5.55	2.63 <sup>(g)</sup>	9.50	1,6,16
Quinoline	<u>31</u>	<u>2.70</u>	<u>1.67</u> <sup>(g)</sup>	<u>3.59</u>	3,6,8,16
TOTAL	278	6.30	3.70	4.87	

Notes: (a) from Reference 9.

(b)  $T_C$  from Reference 9;  $P_C$  by fitting Wilson's data to Riedel's equation and extrapolating to  $T_C$ .

(c) from Reference 9.

(d)  $T_C$  estimated from Lydersen's method;  $P_C$  by fitting Wilson's data to Riedel's equation and extrapolating to  $T_C$ .

(e) from Reference 7.

(f)  $T_C$  and  $P_C$  estimated from Lydersen's method.

(g) from Reference 6.

TABLE 4-2

ANALYSIS OF CHAO'S VLE DATA ON H<sub>2</sub> BINARIES WITH RKJZ

<u>System</u>	<u>Points</u>	<u>t Range, °F</u>	<u>P Range, psia</u>	<u>Optimum C<sub>ij</sub></u>	<u>RMSD of<sup>(a)</sup> Both K, %</u>
HYDROCARBONS					
Toluene	25	372-576	292-3680	0.307	6.10
<u>m</u> -Xylene	27	373-588	288-3691	0.322	4.79
Tetralin	24	373-732	294-3674	0.250	6.69
1-Methylnaphthalene	27	372-803	294-3674	0.178	5.14
Diphenylmethane	27	373-803	294-3674	0.224	4.97
Bicyclohexyl	28	372-803	294-3674	0.416	6.11
NON-HYDROCARBONS					
Quinoline	27	373-803	392-3671	0.272	2.82
<u>m</u> -Cresol	41	372-732	294-3684	0.293	3.61
Thianaphthene	27	370-805	290-3670 { (b)	0.381	5.51
			(c)	0.206	3.64

Notes: (a) RMSD = root mean square deviation

(b) With specific gravity at 60/60°F of thianaphthene = 1.15

(c) With specific gravity at 60/60°F of thianaphthene = 1.22

TABLE 4-3

ANALYSIS OF CHAO'S VLE DATA ON CH<sub>4</sub> BINARIES WITH RKJZ

<u>System</u>	<u>Points</u>	<u>t Range, °F</u>	<u>P Range, psia</u>	<u>Optimum C<sub>ij</sub></u>	<u>RMSD of Both K, %</u>
HYDROCARBONS					
Benzene	18	298-442	288-3510	0.058	7.09
Toluene	26	301-518	292-3665	0.074	6.86
<u>m</u> -Xylene	22	370-588	292-2929	0.058	4.46
Tetralin	24	372-736	294-3674	0.095	4.79
1-Methylnaphthalene	28	376-807	297-3645	0.090	4.84
Diphenylmethane	25	373-805	291-3670	0.075	3.46
NON-HYDROCARBONS					
Quinoline	28	373-805	291-3670	0.100	4.43
<u>m</u> -Cresol	25	372-734	290-3674	0.117	4.11

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## 4.2 Coal Liquids Physical Properties

### Summary

The objective of this program is to develop correlations for predicting coal liquids physical properties including density, viscosity, and surface tension. Density and viscosity measurements on four Illinois coal liquids have been completed and preliminary correlations have been developed. With regard to surface tension, Brigham Young University (BYU) has reported data on three Illinois coal liquids (A-10, A-6 and A-3); data on Illinois coal liquid A-2 are expected during the last half of 1979.

In order to gain some insight into the effects of different kinds of coal on the properties of coal liquids, a similar experimental program for Wyoming coal liquids has been initiated and measurements are in progress at several laboratories including BYU, Southwest Research Institute (for density and viscosity measurements), and Exxon's Baytown Laboratory (for viscosity measurements).

### Illinois Coal Liquids

BYU has reported the surface tension data for the Monsanto reference fluid and three Illinois coal liquids--hydrotreater feed (A-3), recycle solvent (A-6), and fractionator feed (A-10), over the temperature range of 200-700°F and pressure range of 100-3000 psia. Surface tension data on Illinois heavy vacuum gas oil (A-2) remain to be done. Analysis of the data indicated that for the Monsanto fluid, the data at 100 psia show a similar convex curvature with respect to temperature as compared to the data at 14.7 psia from Imperial Oil Limited, Sarnia; however, at 3000 psia, the surface tension-temperature curve is concave and the data are probably too high at 300 and 500°F. With regard to coal liquid surface tension, comparison between our petroleum-based prediction and experimental data shows a considerable disparity. Moreover, the experimental data are somewhat scattered.

As reported in the January-March, 1979 EDS Quarterly Report,\* we have reanalyzed the experimental physical properties of Illinois coal solvent. The reanalysis was made by submitting the coal liquid fractions, with the critical properties predicted by the new procedure, as pure aromatic compounds. Although no significant differences were observed in the density and surface tension predictions, the use of new criticals, in place of the petroleum-based criticals, had a remarkably large effect on the viscosity prediction. The average deviation in predicting viscosity was reduced from 29.1% to 10.6%, and the bias from +18.4% to -5.9%.

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\*FE-2893-29



### Wyoming Coal Liquids

Arrangements have now been completed for the experimental program on four Wyoming coal liquids including unhydrotreated solvent (A-5), hydro-treated solvent (A-6), spent solvent (V-1 bottoms) and heavy vacuum gas oil (A-2). Densities in the absence of added hydrogen will be measured at Southwest Research Institute (SWRI). Viscosities, in the absence of hydrogen, will be measured at Baytown, using the slurry viscometer. Viscosities and densities of coal liquids saturated with hydrogen, as well as surface tension with, and without hydrogen, will be measured at BYU.

SWRI has completed the density measurements at 200 psig. They might have difficulty in carrying out the density measurements at 2000 psi if no suitable seal material for the test cylinder can be found. They also indicated the requested viscosity measurements were beyond the capability of their present instrumentation; accordingly, the viscosity measurements have been dropped from the SWRI program.

### Future Work

Continued work on this program will center around obtaining and analyzing the physical properties on coal liquids. Upon completion of the physical-property work at three laboratories, these data will be used as a base for developing correlations.

### 4.3 Commercial Fractionator Recovery Prediction

The objective of this program is to collect data and determine the applicability of existing petroleum distillation interconversion correlations to coal liquid samples from Illinois and Wyodak coal. These correlations are required to convert data obtained from the various analytical stills used on coal liquid products to a distillation basis consistent with our current design procedures. This permits the calculation/prediction of the distillate product recovery achievable in the commercial vacuum tower and coker fractionator. To achieve this objective, laboratory distillations were performed on samples of the feed and product streams for the CLPP Vacuum Tower and the Large Stirred Coking Unit (LSCU). A simplified flow plan of these two processes is shown in Figure 4-1.

Based on the analysis of the distillation data the existing petroleum distillation interconversion correlations for Hivac C distillations can be used for coal liquid samples from Illinois or Wyodak coal. This applies to both the CLPP Vacuum Tower and the Large Stirred Coking Unit.

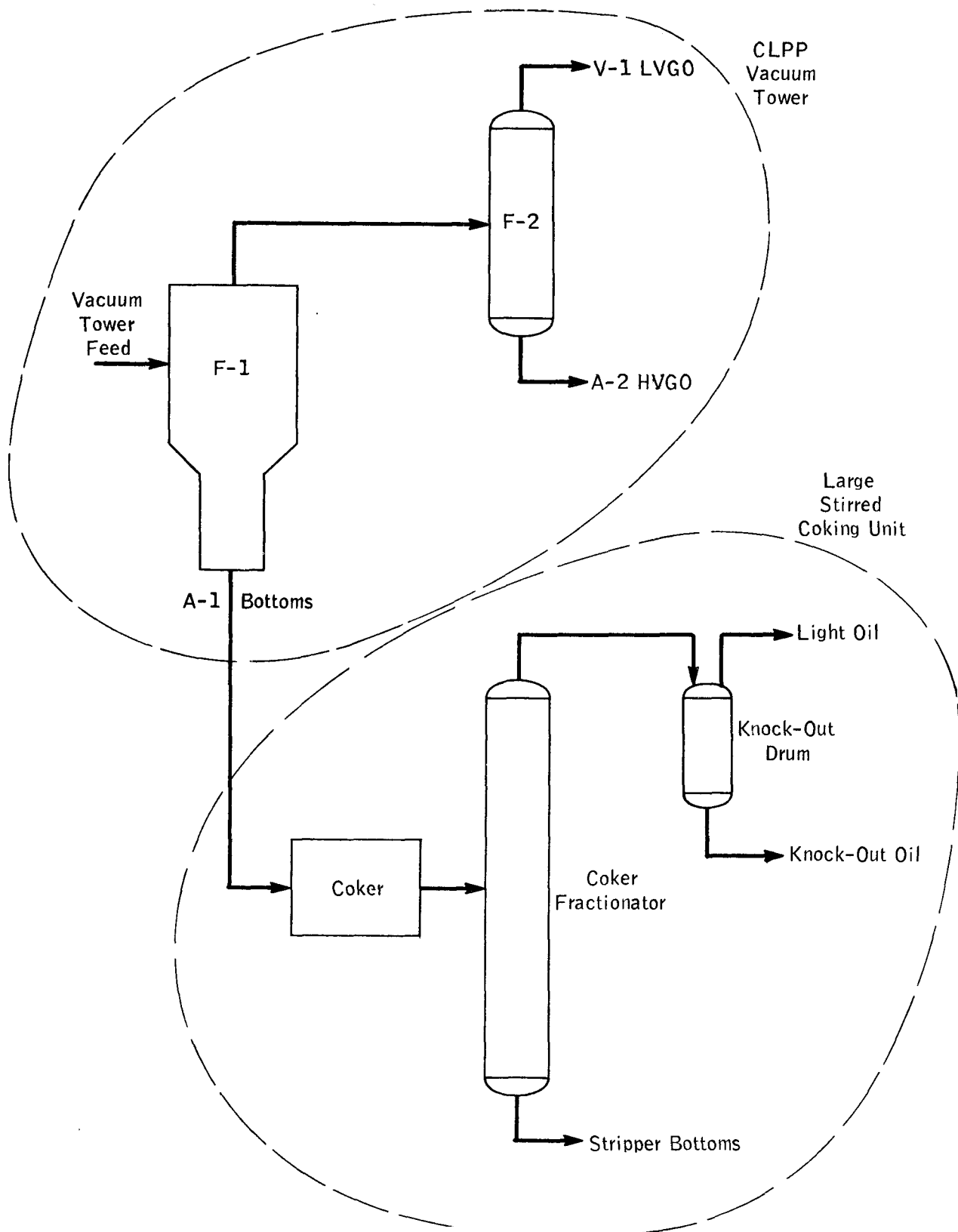
#### Data

A complete list of all the distillations performed for this project is given in Tables 4-4 and 4-5. The standard procedure for obtaining distillation data on small samples of high-boiling liquids such as coal liquids is to run an atmospheric 15/5 distillation up to a specified cutpoint. Distillation data on the remaining bottoms is then obtained via a Hivac C or microlube vacuum distillation. A Hivac C distillation is preferred since it generates a curve whereas a microlube distillation generates only a single point. When larger quantities of samples are available a metal Hivac distillation can be performed to obtain the distillation data. A metal Hivac distillation consists of running an atmospheric 15/5 distillation to a 650°F cutpoint followed by a vacuum metal Hivac distillation on the resulting bottoms.

#### Data Conversion

Depending on the type of distillation available for a particular stream, different techniques are used to obtain a total distillation curve that is consistent with our current design procedures. Three steps are required if small scale 15/5 and Hivac C distillations are available. First, the vacuum distillation temperatures are converted to atmospheric equivalent temperatures through the use of characterization corrected vapor pressure charts. Second, the Hivac C atmospheric distillation temperatures are converted to 15/5 distillation temperatures using petroleum interconversion correlations. Third, the converted Hivac C distillation is combined with the 15/5 distillation to obtain a total distillation curve. If 15/5 and metal Hivac distillation data are available the first and third steps are required to obtain a total distillation curve. Currently, if 15/5 and microlube distillation data are available a total distillation curve cannot be accurately obtained.

FIGURE 4-1  
SIMPLIFIED FLOW PLAN



To allow for a direct comparison of the 15/5 distillation temperatures, the distillation curves for the product streams were combined to give a distillation curve for the respective feed stream. A comparison of the various total distillation curves for the Vacuum Tower feed and the Coker Fractionator feed from Illinois coal is given in Table 4-6. For each feed the total distillation curves obtained from the small scale distillations are averaged and then each total distillation curve is compared to the average total distillation curve.

#### CLPP Vacuum Tower Model

In addition to a direct comparison of the distillation curves, a model was developed for the CLPP Vacuum Tower. This model consists of an adiabatic flash and is based on the following data obtained from the Illinois coal run - yield period 256.

Feed Temperature	=	702°F
Feed Pressure	=	10.9 psia
Flash Pressure	=	1.22 psia
Flash Temperature	=	662°F
V/F, Vol %	=	55.2

The 15/5 distillation curve for the A-1 Bottoms is used to represent data for the liquid stream and a combined 15/5 distillation curve of the V-1 LVGO and A-2 HVGO is used to represent data for the vapor stream. A comparison of this data with the results of flashes performed on the various total distillation curves of the feed stream is given in Table 4-7.

#### Future Work

All the distillation data have been received for this project. Remaining work includes the preparation of a final report which will contain additional details of the project along with the available results for Wyodak coal.

TABLE 4-4

DISTILLATIONS PERFORMED ON ILLINOIS COAL LIQUID SAMPLES<sup>(1)</sup>

<u>Stream</u>	<u>Small Scale Distillations</u>			<u>Metal Hivac Distillation</u>		
	<u>15/5 Cutpoint</u>	<u>Hivac C</u>	<u>Microlobes</u>	<u>15/5 Cutpoint</u>	<u>Metal Hivac</u>	<u>Hivac C<sup>(2)</sup> (each cut)</u>
Vacuum Tower	550°F	X	X	650°F	X	X
Feed	650°F	X	X			
Blended Vacuum <sup>(3)</sup>	500°F	X	X			
Tower Feed	550°F	X	X			
	650°F	X	X			
A-1 Bottoms	-	X	X			
A-2 HVG0	-	X	X			
V-1 LVGO	858°F	-	-			
Blended Coker <sup>(3)</sup>	500°F	X	X	650°F	X	X
Fractionator Feed	550°F	X	X			
	650°F	X	X			
Light Oil	500°F	X	X			
	550°F	X	X			
	650°F	X	X			
	807°F	X	X			
Knock-out Oil	650°F	X	X			
Stripper Bottoms	-	X	X			

(1) Vacuum Tower samples are from CLPP Yield Period 256.  
Coker Liquid samples are from LSCU Run #7.

(2) Hivac C distillation performed on each metal Hivac cut.

(3) Sample prepared by blending product stream samples.

TABLE 4-5

DISTILLATIONS PERFORMED ON WYODAK COAL LIQUID SAMPLES<sup>(1)</sup>

<u>Stream</u>	<u>Small Scale Distillations</u>			<u>Metal Hivac Distillation</u>	
	<u>15/5 Cutpoint</u>	<u>Hivac C</u>	<u>Microlubes</u>	<u>15/5 Cutpoint</u>	<u>Metal Hivac</u>
Vacuum Tower	550°F	X	X	650°F	X
Feed	650°F	X	X		
Blended Coker <sup>(2)</sup>	500°F	X	X	650°F	X
Factionator Feed	550°F	X	X		
	650°F	X	X		
Light Oil	500°F	X	X		
	550°F	X	X		
	650°F	X	X		
Knock-Out Oil	-	X	X		
Stripper Bottoms	-	X	X		

(1) Vacuum Tower samples are from CLPP Yield Period 238.  
Coker Liquid samples are from LSCU RUN #17.

(2) Samples prepared by blending product stream samples.

TABLE 4-6

COMPARISON OF TOTAL DISTILLATION CURVES FOR VACUUM TOWER FEED FROM ILLINOIS COAL

<u>Stream - 15/5 Cutpoint</u>	<u>Avg. Deviation<sup>(1)</sup> of 15/5 Distillation Temperatures</u>	<u>Bias<sup>(1)</sup> of 15/5 Distillation Temperatures</u>
Vacuum Tower Feed - 550°F	16.4	-11.2
Vacuum Tower Feed - 650°F	14.8	-11.1
Blended Vacuum Tower Feed - 550°F	19.5	14.8
Blended Vacuum Tower Feed - 650°F	11.8	8.5
Combination of Vacuum Tower Product Streams	<u>10.3</u>	<u>-1.0</u>
Average	14.6	0

COMPARISON OF TOTAL DISTILLATION CURVES FOR COKER FRACTIONATOR FEED FROM ILLINOIS COAL

<u>Stream - 15/5 Cutpoint</u>	<u>Avg. Deviation<sup>(1)</sup> of 15/5 Distillation Temperatures</u>	<u>Bias<sup>(1)</sup> of 15/5 Distillation Temperatures</u>
Blended Coker Fractionator Feed - 500°F	7.6	0.2
Blended Coker Fractionator Feed - 550°F	22.4	22.2
Blended Coker Fractionator Feed - 650°F	19.8	19.6
Combination of Coker Fractionator Product Streams with Light Oil Cutpoint of - 550°F	15.7	-15.6
Combination of Coker Fractionator Product Streams with Light Oil Cutpoint of - 650°F	12.5	-12.4
Combination of Coker Fractionator Product Streams with Light Oil Cutpoint of - 807°F	<u>14.3</u>	<u>-14.0</u>
Average	15.4	0

$$(1) \text{ Average Deviation} = \sum_{LV\% = 0}^{100} \frac{|T(LV\%) - T_{avg}(LV\%)|}{41}$$

$$\text{Bias} = \sum_{LV\% = 0}^{100} \frac{T(LV\%) - T_{avg}(LV\%)}{41}$$

LV% = liquid volume percent distilled in increments of 2.5%.

T(LV%) = 15/5 distillation temperature, °F, at LV% distilled for indicated stream.

Tavg(LV%) = 15/5 distillation temperature, °F, at LV% distilled for average stream.

TABLE 4-7

COMPARISON OF DATA AND MODEL PREDICTIONS FOR THE CLPP VACUUM TOWER

<u>Feed Stream - 15/5 Cutpoint</u>	<u>Δ Flash Temp, °F</u>	<u>V/F Percent Error</u>	<u>Avg. Dev. Of 15/5<sup>(1)</sup> Temp For Vapor</u>	<u>Bias Of 15/5<sup>(1)</sup> Temp For Vapor</u>	<u>Avg. Dev. Of 15/5<sup>(1)</sup> Temp For Liquid</u>	<u>Bias Of 15/5<sup>(1)</sup> Temp For Liquid</u>
Vacuum Tower Feed - 550°F	7	2.5	12.9	-1.8	17.3	0.9
Vacuum Tower Feed - 650°F	9	1.5	15.1	-3.9	13.3	-2.8
Blended Vacuum Tower Feed - 550°F	8	-8.8	12.2	-5.3	16.8	-6.4
Blended Vacuum Tower Feed - 650°F	8	-6.6	7.1	-4.1	14.2	-8.8
Combination of Vacuum Tower Product Streams	<u>9</u>	<u>-1.8</u>	<u>3.1</u>	<u>-2.4</u>	<u>7.3</u>	<u>-6.7</u>
Average	8	4.2	10.1	-3.5	13.8	-4.8

$$(1) \text{ Average Deviation} = \frac{\sum_{LV\% = 0}^{100} |T(LV\%) - T_{data}(LV\%)|}{12}$$

$$\text{Bias} = \frac{\sum_{LV\% = 0}^{100} T(LV\%) - T_{data}(LV\%)}{12}$$

LV% = liquid volume percent distilled in increments of 10%.

T(LV%) = 15/5 distillation temperature, °F, at LV% distilled for indicated stream.

T<sub>data</sub> (LV%) = 15/5 distillation temperature, °F, at LV% distilled from data.



#### 4.4 Vacuum Tower Fouling

The performance of the CLPP vacuum flasher (F-1) was monitored throughout the past year. The vacuum flasher operated satisfactorily, showing no signs of coking except that attributable to bottoms level upsets.

In April, the Glitsch grid internals in the vacuum flasher had corroded over the years to the point where the welds failed. Therefore, a new 316 SS grid was ordered, in addition to a chrome-plated grid. This chrome-plated grid will be used to qualitatively evaluate whether coke formation is inhibited by a smooth surface.

In April, the detailed ECLP Test Programs' descriptions and objectives were completed. The test programs are:

- "Vacuum Tower Transfer Line and Pipestill Simulation." This program includes a series of six tests with each coal over a range of flash zone operating conditions. The detailed heat and material balances and product quality data will be used in the simulations of the vacuum pipestill, transfer line and furnace. The simulation results will provide input for the following four EETD coal liquefaction programs:
  - + Vacuum Tower Fouling
  - + Vacuum Tower Transfer Line
  - + Thermal Cracking of Coal Liquids in Vacuum Separation
  - + Commercial Fractionation Recovery Prediction

At present, only the Vacuum Tower Fouling and Commercial Fractionation Recovery Prediction programs are active; the remaining two will commence when ECLP has begun operating.

- "Vacuum Pipestill Stripper Efficiency." A series of three tests are proposed to determine the effect of stripping steam on the vacuum flasher cutpoint. From the detailed heat and material balances and product quality data collected at various stripping steam rates, the stripping efficiency of the shed rows can be determined and the effect of stripping steam on the cutpoint quantified.
- "Adiabatic Vacuum Distillation." In the adiabatic testing program, the vacuum furnace is bypassed so that the maximum cutpoint in the absence of furnace preheat can be determined. During the testing, detailed heat and material balances and product quality data will be obtained. These data will be used in the adiabatic operation simulations of the vacuum pipestill.

#### 4.5 Coal Liquefaction Reactor Cold Model Studies

##### Summary

Studies were carried out to provide the data base for the development of correlations describing gas, liquid, and solids holdup in the ECLP reactor as a function of such variables as gas and slurry velocity, slurry concentration, and particle size. These were carried out using the 6" diameter column. With 60 mesh minus coal, gas and solids holdups were strong functions of feed coal concentration within the ECLP design range. The average residence times of these coal particles were about equal to the bulk liquid. Higher coal holdups were obtained previously using an 8 mesh minus cut at the same feed inlet concentration and flow conditions showing that the larger size cut did not fluidize in a full transport regime.

A review was made of the SRC pilot plant experience at Wilsonville, Alabama with its reactor and solids withdrawal system. Good Wilsonville operation of the distributor and solids withdrawal system indicates comparable experience in ECLP can be anticipated. Operation appeared satisfactory using a three phase distributor similar to the ECLP design at the reactor inlet. No change in the ECLP design was made as the result of this review.

Test programs were developed for evaluating the fluid dynamic performance of the ECLP reactor and solids withdrawal system.

##### Solids Holdups are Sensitive to Feed Coal Concentration and Size

As reported in the January-March, Phase IV 1979 Quarterly Report,<sup>(1)</sup> solids holdups are sensitive to the feed coal concentration for the 60 mesh minus coal tested in a 6" diameter column. Coal solids holdups in the column increased linearly with the coal feed concentration and were less sensitive to changes in gas and slurry velocities as shown in Figure 4-2. This was due to solids being nearly fully transported at the velocities tested. Higher coal holdups were obtained previously with an 8 mesh minus cut for a wide range (14 wt% to 41 wt%) of feed concentrations also shown in Figure 4-2. This indicates that the larger size particles were not fluidized in the same range of fluid velocities. An ECLP test to quantify the withdrawal operation is being considered.

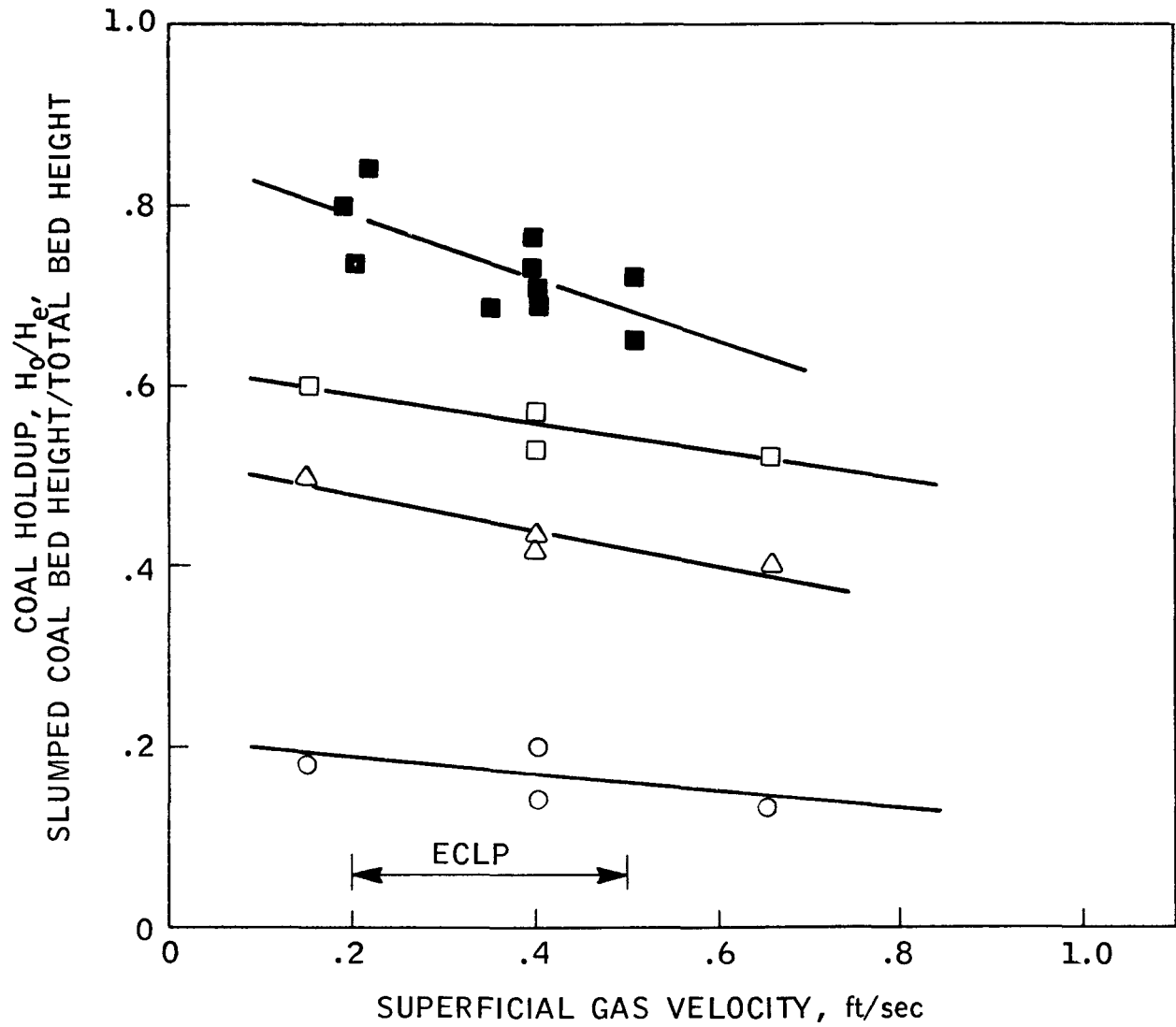
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<sup>(1)</sup>FE-2893-29

Figure 4-2  
SOLID HOLDUP IN SLURRY FEED OPERATIONS

Legend

- 8 Mesh Minus, 14-41 Wt% Coal (6" And 24" Units With Heptane/N<sub>2</sub>)
- 60 Mesh Minus, 44 Wt% Coal (6" Unit With Heptane/N<sub>2</sub>)
- △ 60 Mesh Minus, 34 Wt% Coal (6" Unit With Heptane/N<sub>2</sub>)
- 60 Mesh Minus, 12 Wt% Coal (6" Unit With Heptane/N<sub>2</sub>)



#### Gas Holdups Are Affected by Feed Coal Concentration and Size

Gas holdups were also affected by the feed coal concentration for the 60 mesh minus coal tested in a 6" diameter column. The strong influence of gas velocity on gas holdups was similar for both 8 and 60 mesh coals as shown in Figure 4-3. The holdups obtained from the 60 mesh minus cut are bounded by the results from the pure liquid and the 8 mesh minus coal slurry runs. All gas holdups obtained within the ECLP design flow velocities were acceptable for the residence time and mass transfer considerations in the reactor.

#### Good Wilsonville Process Operating Experience Augers Well for ECLP

A review was made of the SRC pilot plant experience with its reactor distributor and solids withdrawal system. Process conditions for this pilot plant and ECLP reactors are similar. Both reactor configurations have comparable internals and similar locations for the solids withdrawal lines. However, ECLP uses larger sized feed coal, employs five-fold higher slurry and gas velocities, and has a high solvent quality. The higher velocities, required to keep the coarser coal in suspension, should increase turbulence, mixing and mass transfer. Hence, in view of good Wilsonville operation on the distributor and solids withdrawal, comparable experience in ECLP can be anticipated.

#### ECLP Test Programs for Reactor and Solids Withdrawal

Test programs aimed at confirming scaleup criteria and defining operating limits for the liquefaction reactor and solids withdrawal system for ECLP have been developed for further review. These involve evaluating distributor pressure drops, turnup and turndown characteristics, reactor pressure drops and densities, holdups and particle concentrations, particle size distributions, and solids withdrawal frequency tests, and examinations of bubble caps, baffle plates and the solids withdrawal system.

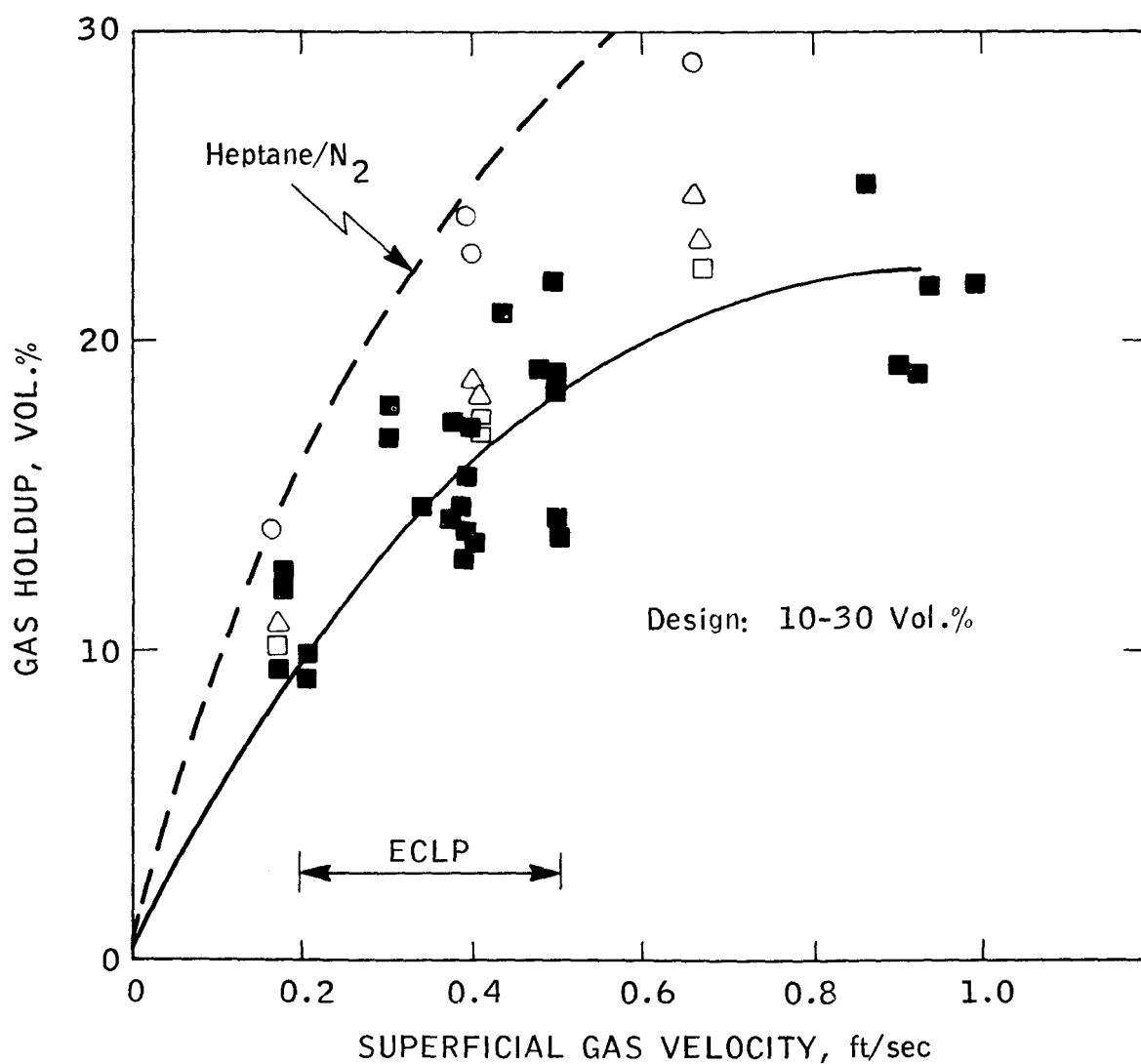
#### Future Work

Studies will continue on establishing the effect of slurry concentration and particle size in the six inch column to provide the data base for coal slurry fluidization correlations. Work will also continue on defining the details of the proposed ECLP test programs.

Figure 4-3  
GAS HOLDUP IN SLURRY FEED OPERATIONS

Legend

- Heptane/N<sub>2</sub> (6" And 24" Units, Slurry Feed = 14-41 Wt% 8 Mesh Minus)
- Heptane/N<sub>2</sub> (6" Unit, Slurry Feed = 12 Wt%, 60 Mesh Minus)
- △ Heptane/N<sub>2</sub> (6" Unit, Slurry Feed = 34 Wt%, 60 Mesh Minus)
- Heptane/N<sub>2</sub> (6" Unit, Slurry Feed = 44 Wt%, 60 Mesh Minus)



#### 4.6 Rheology of Coal-Solvent Slurries and EDS Products

##### Summary

Viscosities and pressure drops were measured for Illinois vacuum bottoms with a 5.7 wt% 1000°F minus content, heavy raw creosote oil (HRCO), and blends of bottoms and HRCO. Microlube distillations showed the blends to have 20 to 37 wt% 1000°F minus contents. The vacuum bottoms and blends exhibited the characteristic thinning or thickening behavior as functions of shear rate and 1000°F minus content. A 524°F vacuum bottoms showed shear thinning behavior at shear rates below 200 sec<sup>-1</sup> and shear thickening at higher rates. The 20 and 27 wt% 1000°F minus content blends were shear thinning at 376 and 425°F at all tested shear rates; the 37 wt% 1000°F minus content sample shear thickened at temperatures of 332 and 353°F. The HRCO and blends data were fitted to a power law viscosity model. The detailed results of these runs are reported in the January 1 through March 31, 1979 Phase IV Quarterly Report (FE-2893-29).

Viscosity measurements were also made on Illinois 30 mesh minus, feed coal/solvent slurry containing 15 wt% solids at temperatures up to 300°F. Measurements of a 30 wt% solids slurry are in progress and tests on a 45 wt% slurry are planned. Data analyses are underway and will be completed during the next quarter.

##### Future Work

The 45 wt% Illinois feed coal slurry measurements will be completed and the data from all runs will be analyzed. Similar measurements on Wyodak feed coal slurries are planned at temperatures up to 810°F and solids concentrations up to 45 wt%. Correlations will be developed for predicting the viscosities of feed slurries as a function of solids content and temperature and of bottoms streams with varying amounts of 1000°F minus material.

#### 4.7 Slurry Preheater Development

The overall objective of this program is to investigate the coking tendency of coal slurry preheater furnaces and to gain a qualitative understanding of preheater coking as it is affected by important process and design parameters. The data from this program will be used for more accurate prediction of the performance of the ECLP slurry preheater and for evaluation of data from ECLP.

#### Summary

In the past year the coking tendency of slurry feeds in the CLPP sectionalized slurry preheater has been investigated. The operating experience of slurry preheaters in the PAMCO and Southern Services pilot plants has been reviewed for implications to the ECLP slurry preheater. In addition, several test programs for the ECLP slurry preheat furnace have been developed.

The CLPP preheaters have operated virtually trouble-free with no coking being detected at conditions equal to or more severe than the ECLP design basis. A review of the PAMCO and Southern Services slurry preheaters has shown that coking has not occurred at normal operating conditions which are close to but not as severe as ECLP design conditions. The PAMCO and Southern Services preheaters have a 3 phase feed consisting of coal, solvent, and treat gas as does ECLP, but in contrast to ECLP they operate with low heat fluxes and do not demonstrate return bends.

#### CLPP Slurry Preheater Testing

The CLPP slurry preheater continued to operate satisfactorily on Wyodak coal with solvent to coal ratios (S/C) varying between 1.2 and 1.5. The slurry preheater ran for a 6 day period where both a low S/C ratio (nominal 1.5) and a high coil outlet temperature (848°F) were achieved simultaneously with Wyodak coal. To date, the preheaters have operated virtually troublefree with no coking being detected at conditions equal to or more severe than the design basis established for ECLP. This suggests that reasonable run lengths can be achieved in the ECLP preheater on Wyodak coal at design conditions. Discussions of CLPP preheater operating experiences were detailed in the Oct.-Dec. 1978 Quarterly Progress Report.\*

The operating experience of the CLPP slurry preheater with recycled vacuum bottoms is being monitored because bottoms recycle could potentially increase coking in the preheater. Since recycle operation commenced, the most significant period from a coking standpoint was a run for 261 hours from late March to mid-April. During this period CLPP operated with a 1.2 solvent/coal ratio fresh feed and 40% of the total feed containing recycled bottoms. Coking or plugging has not occurred at normal operating conditions with coil outlet temperatures of about 840°F.

\*FE-2893-25

Southern Services and  
PAMCO Slurry Preheater Review

Based on information obtained from a visit to the 6 T/D Southern Services SRC pilot plant in Wilsonville, Alabama and from a review of recent Southern Services Quarterly and Annual reports, the Southern Services slurry preheater performance has been reviewed and evaluated for its implications to ECLP. The Southern Services preheater has not experienced coking at normal operating conditions very similar to ECLP conditions except that heat flux and coil outlet temperature were lower. On occasion, Southern Services has experienced coking; in tests where the hydrogen gas partial pressure was reduced, a reduction in the solvent hydrogen donor capability was reported as the probable cause of coking. They also indicated that lighter solvents (350-800°F) have a higher tendency to form coke than heavier solvents (450-900°F), but it is not known whether this is due to increased vaporization or reduced solvent hydrogen donor capability. ECLP has a high hydrogen donor solvent and is not expected to experience rapid coking at design conditions; however, preheater coking should be closely monitored when the ECLP solvent composition changes, for example, during a plant upset.

The Southern Services preheater has been successfully steam-air decoked although twice the coil has plugged during spalling in the small diameter (1.16 inch ID) tubes. This gives increased confidence that the ECLP preheater can be steam-air decoked. Plugging is not expected to be a problem in ECLP's larger preheater tubes. There has been little apparent saltation or erosion in the Southern Services preheater. Though this is encouraging, extrapolation to ECLP cannot be made since the ECLP preheater has a larger coal particle size, higher linear velocity, and short radius return bends. Therefore, erosion and saltation will still have to be closely monitored in ECLP to establish the expected rate for commercial design.

A comparison of the PAMCO and ECLP slurry preheaters has been made based on a review of recently published information. Compared to the ECLP slurry preheater the PAMCO preheater operates at similar pressures but at coil outlet temperatures primarily 50 to 100°F lower than the ECLP design temperature of 850°F. As expected, the PAMCO preheater has not experienced coking at normal operating conditions. In the PAMCO preheater, the coal particle size is significantly smaller, and both the heat flux and linear slurry velocity are lower. However, PAMCO is planning to operate a new preheater without return bends at higher heat fluxes and linear velocity which potentially may provide data pertinent to ECLP.



### ECLP Test Program

Detailed test programs for the ECLP slurry preheat furnace have been developed to define requirements on manpower, costs, test procedures, and equipment. Test programs for the slurry preheater will investigate coking, steam-air decoking, and three-phase, high temperature inside heat transfer coefficients. These programs will provide the necessary scaleup criteria and operating limits for the EDS commercial slurry preheater. Data on the effects of film temperature, heat flux, residence time, and solvent to coal ratio on coking and run length will be obtained. Steam-air decoking of the slurry preheater will be demonstrated. Also high temperature three phase heat transfer correlations will be checked and modified for calculations of film temperature and tube metal temperature.

### Future Plans

Investigation of the coking tendency of slurry feeds in the CLPP slurry preheater will be continued with emphasis on new processing conditions and a new coal. Wyodak coal will be retested but with bottoms recycle as well as with H<sub>2</sub>S addition to the treat gas. Pittsburgh No. 8 coal is also scheduled to be tested. Operating data from the new PAMCO preheater will be analyzed when it is available and evaluated for its implications to the ECLP and commercial EDS slurry preheaters.

#### 4.8 Slurry Distributor Manifold

The overall objective of this program is to develop the technology necessary to design the slurry distributor manifold for the slurry preheat furnace in the commercial EDS Coal Liquefaction Plant. Uniform distribution of coal, solvent and treat gas to each of the parallel passes is essential to minimize coking in a multi-pass slurry preheater. In the commercial design the coal/solvent slurry and the treat gas are distributed separately to each pass. The feed laterals each have a control valve and flow measurement to provide an equal quantity of the feed stream in the individual furnace passes.

There is a concern that momentum and friction effects in the manifold could cause a maldistribution of slurry coal particles to each pass. This maldistribution is envisioned as possibly occurring either as: (1) a concentration maldistribution or (2) a particle size maldistribution. Either case could increase the possibility of coking in some passes due to higher viscosity, lower inside heat transfer coefficient, and higher film temperature.

#### Summary

A laboratory slurry distribution manifold test unit was designed to evaluate slurry distribution patterns as a function of feed coal concentration, slurry velocity in the manifold, and manifold configuration. Construction of this test unit was completed 2nd Quarter, 1978. During check-out and shakedown, which followed in the 3rd Quarter, 1978, minor instrument problems were resolved and mechanical modifications were identified and completed to minimize air trapping in the test facilities.

Preliminary testing was scheduled for 4th Quarter, 1978, but it was delayed because a portion of the test facilities was used for another study. Tests employing coal slurry in the distribution manifold is now scheduled to begin in 3rd Quarter, 1979.

#### 4.9 Slurry Fluid Dynamics In Process Use

##### Summary

The objective of this project is to assess the impact of current slurry fluid dynamics technology on the design and operation of ECLP and an EDS commercial plant. During this year, two areas of potential concern were evaluated: possible saltation problems associated with the handling of EDS bottoms slurries and the accuracy of pressure drop prediction techniques for feed coal/solvent and feed coal/solvent/treat gas systems. Laboratory studies with an 8.1 wt% slurry of 200  $\mu\text{m}$  glass beads in glycerin were carried out to assess the magnitude of possible saltation problems with EDS bottoms slurries. Saltation velocities were found to be below 1.0 ft/sec and design techniques were conservative, predicting saltation velocities of 3 ft/sec. If these results are confirmed in additional tests with smaller particles and higher solids content slurries, then long term performance of EDS slurry lines should not be a problem despite laminar flow regime operation.

Currently used pressure drop correlations for feed coal/solvent and feed coal/solvent/treat gas systems were compared to available data and found to overpredict the pressure drop by as much as 55%. However, there is little or no data available on coal systems at elevated temperatures. Therefore, an ECLP test program was developed to collect suitable data across the slurry preheater.

##### Saltation May Not be a Problem With EDS Vacuum Bottoms Slurries

EDS vacuum bottoms slurries, with viscosities probably above 10 poise, will be in the laminar flow regime. Although particle settling rates will be low, long term operation could result in particle buildup in process lines. Laboratory experiments were carried out to assess the magnitude of the problem using an 8.1 wt% slurry of 200  $\mu\text{m}$  glass beads in glycerin to simulate EDS vacuum bottoms slurries.

The experiments were done in a 3 inch diameter pipe loop that was equipped with 1 ft long glass viewing section. The slurry, which had an average viscosity of 11 poise, was circulated through the lines for 30 minutes. No settling was observed at flow rates as low as 0.2 ft/sec or a particle Reynolds number of about 5. The flow was stopped and the solids were allowed to settle. Resuspension of the particles was achieved in 10 to 15 minutes of operation at a flow velocity of 0.5 ft/sec. The predicted saltation velocity was 3 ft/sec using current design techniques. Additional experiments are planned with smaller particles and higher solids loadings. If the results are confirmed, long term operation should not be a problem.

## Pressure Drop Predictions for Feed Coal Slurry Systems are Conservative

An evaluation was made of the accuracy of pressure drop prediction techniques for feed coal/solvent and feed coal/solvent/treat gas systems. Current design techniques for coal liquids are conservative, overpredicting the pressure drop by as much as 55%. However, there is little or no data available on coal systems at elevated temperatures for validation of existing and proposed correlations. Therefore, an ECLP test program was developed to collect suitable data across the slurry preheaters.

For feed coal/solvent slurries, the Zandi correlation (ASCE Hydraulics Div. J 93, 145 [1967]) was used to predict pressure drop. This correlation was compared to data obtained in a pipeloop that had 1 inch and 2 inch diameter lines with an Illinois Coal/Creosote oil slurry at temperatures up to 280°F (Phases I and II Summary of Results [1966-1975]).\* The solids concentrations were between 15 and 45 wt%. The difference between data and predictions averaged about 13%. The correlation has not been tested on coal systems at higher temperatures where dissolution and swelling may occur. Particle degradation either by chemical or physical mechanisms cannot be accounted for with the Zandi correlation.

Three phase systems are generally handled by treating them as two phase liquid-gas flow with the slurry properties substituted for those of the liquid. The Dukler-Hughmark correlation (AIChE J. 10, 78 [1964]) was employed for predicting pressure drops. This correlation was compared to data obtained on the previously mentioned pipeloop with a coal/creosote/nitrogen system. The average difference between predicted and measured values was 11% with a maximum difference of 40%. These data were measured at temperatures below 300°F.

At elevated temperatures, data were recently collected across the CLPP preheater during a 24 hour operation on Illinois coal. The preheater is a 3/8 inch diameter 50 ft coil with a 12 ft straight run of pipe on either end. The slurry was about 37 wt% Illinois coal ground to 30 mesh minus; the gas volume fraction was 82%. Measurements were made at 810°F and 1530 psig. This temperature is higher than the 600°F value at which swelling of Illinois coal occurs. The Dukler-Hughmark correlation, corrected for the helical geometry, overpredicted the data by an average of 26% with the difference varying from 5 to 55%.

## Future Work

Additional tests are planned with smaller particles and higher loadings to confirm that saltation of EDS vacuum bottoms slurries will not be a problem. The review is continuing of relevant slurry handling experiences to establish the importance of other possible problems such as restarting and flushing of slurry pumps and the operation of safety and control valves and pulsation dampeners.

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\*FE-2893-16

#### 4.10 High Pressure Coal Slurry Separation Technology

In the EDS coal liquefaction process, coal slurries at high temperature and pressure are separated from gaseous reactor products. There is limited experience with these types of three phase separator systems. Proper design of the separators (as in CLPP) or towers (as in ECLP) after the pressure letdown system is required to avoid carry over from the drums or premature flooding in the atmospheric tower. Foaming, which can be a problem at high temperatures and after pressure letdown, may be a critical factor in obtaining good separation. In addition, the presence of fine solids which tend to stabilize foam could further aggravate the foaming tendency.

This project began with the scoping of high pressure, heavy crude processes in which foaming may exist. A comprehensive review of certain petroleum processing unit design allowances for foaming was completed. In addition, consultants for these processes were contacted for information on any persistent foam problems in the refineries. The only foam problems that occurred have been during start-up operations and disappeared soon after the units were lined out.

A review of outside coal liquefaction design considerations and operating logs for foaming or foam related problems has also been completed. Of the processes examined, only H-coal and the SRC plants have pressure letdown operations similar to that in the EDS design. However, no indication of foaming following pressure letdown has been found.

To assess the nature of foaming, if any, in the EDS separation system, the installation of a sight glass at CLPP has been proposed. This would allow direct observation of the foam, if present, and permit an estimate of foam stability. This information is required for dependable and economic design of future EDS commercial plants. The current ECLP design includes a safety factor on the sizing of the atmospheric pipestill (downstream of the pressure letdown system) for possible foam problems. If the CLPP tests show that foam is not present, future commercial plants could be designed more economically by eliminating this safety factor.

## ENGINEERING RESEARCH AND DEVELOPMENT

### ENGINEERING TECHNOLOGY DEVELOPMENT

#### 5. Bottoms Processing Engineering Technology

##### 5.1 Improve Quality of Coker Scrubber Liquid

###### Summary

An evaluation was made of non-precoated pressure filtration for removal of solids from EDS coker scrubber liquid. Vendor tests documented in the January 1 through March 31, 1979 Quarterly Technical Progress Report (FE-2893-29) indicated that non-precoated pressure filtration is unsuitable for solids removal. Filtration rates were low, less than 4 gph/ ft<sup>2</sup> after six minutes, the screens were rapidly blinded and the solids removal efficiencies were poor. These results appear to be typical of non-precoated filtration in this type of service based on a review of recent experiences with other coal liquids.

Viscosity measurements were made of Large Stirred Coking Unit (LSCU) samples at temperatures up to 700°F. These samples simulate the anticipated viscosity of scrubber liquid from the EDS FLEXICOKING unit. The viscosity of the filtered stripper bottoms was 3.7 cp at 700°F and 700 sec<sup>-1</sup> shear rate. This value is slightly higher than that previously predicted based on extrapolation of low temperature data. The stripper bottoms also exhibited a 30% increase in viscosity when held at 650°F for 43 minutes. Similar thermal stability has been observed with CLPP vacuum bottoms. The viscosity of a blend of equal amounts of unfiltered stripper bottoms and 1000°F minus material was approximately one-half that of undiluted stripper bottoms at 500°F. Based on these results, it appears that blending of EDS scrubber liquid with lighter material could reduce the viscosity to the targeted 3 to 5 cp range.

###### Multistage Hydroclone Loop Proposed for EDS FLEXICOKING Unit

A process basis was developed for a multistage hydroclone test loop at Baytown. The loop will be used to demonstrate the predicted high level of depariculation and multistage operability using EDS scrubber liquid from the FLEXICOKING unit. The loop will have three stages of 10 mm diameter Doxie hydroclones; the first and second stages have four units and the third stage has three units. Provisions are included for full or partial recycle of underflow streams from the second and third stages to the feed stream for the first stage. Both the overflow and underflow product streams will be sent back to the FLEXICOKING unit. The scrubber liquid rate will be 3 gpm at 690°F.

### Future Plans

Hydroclone separators were identified as the preferred technique for removal of solids from coal liquids in an economic and technical analysis of alternative removal schemes completed last year (Annual Technical Progress Report, FE-2893-17). Hydroclone performance was predicted using a model based on data obtained with petroleum coker liquids. To verify this performance model for coal liquids, high temperature testing of a single stage hydroclone separator is planned using LSCU liquids in the laboratory fines removal loop.

## 5.2 FLEXICOKING Unit Coke Attrition Characterization

### Summary

Particle size distribution control is critical to the operability of a FLEXICOKING unit. Attrition studies in captive fluid beds (Annual Technical Progress Report, FE-2893-17) and IKG/FBU data indicated that high ash coal derived cokes could be several fold more attritable than petroleum cokes and, hence, particle size distribution of circulating coke could be appreciably finer. Finer particle size distributions could cause excessive fines losses. The overall objective of this project is to better define the quantity and size distribution of fines to be generated in the EDS FLEXICOKING unit and to develop attrition models for incorporation in the FLEXICOKING unit solids material balance model.

### Future Work

A two foot diameter fluidized bed will be adapted for these tests. Attrition rates will be measured as a function of operating variables. Subsequently, mathematical representations for the various attrition mechanisms will be developed and incorporated into the FLEXICOKING unit solids material balance model.



### 5.3 FLEXICOKING Unit Solids Material Balance and Control

#### Summary

The FLEXICOKING solids material balance developed under this project (Annual Technical Progress Report, FE-2893-17) was used to determine the effects of design and operating parameters on equilibrium particle size distributions and entrainment rates from the reactor, heater and gasifier in both the prototype and commercial scale study designs for the EDS FLEXICOKING process program. Computer simulations indicated potentially high rates of fines carryover from the gasifier and increased fines losses from the reactor and heater cyclones when operating at turndown conditions. Recently, the model was revised to allow attrition to a spectrum of fragment sizes and account for various attrition and agglomeration mechanisms and for feeding solids to any of the process vessels. The revised model predictions match loss rates from the cyclones without the cyclone efficiency and/or fines agglomeration adjustments needed previously. Satisfactory agreement was also obtained with measured commercial FLEXICOKING fines losses and particle size distributions. Loss predictions for the commercial unit at maximum throughput are similar to those obtained with the original model; but prototype reactor losses were four times higher than with the original model and about equal to those estimated for the commercial reactor. The model is now complete and will soon be available for evaluating FLEXICOKING unit operations.

#### Revised Model Includes Improved Agglomeration/Attrition Mechanisms

In the original model, attrition was treated as a surface grinding phenomenon producing uniform size fragments. The attrition fragments in the reactor were assumed to be equal to the minimum particle size which could grow by carbon deposition and heat balance considerations. Particles smaller than this minimum size were assumed to be agglomerated to larger particles. Generation of smaller than the minimum particle size for coke deposition was assumed to take place in the heater by attrition. Agglomeration of particles was omitted and the particles in each bed were free to be entrained overhead.

The revised model allows for attrition in each vessel by specifying a maximum fragment size below which all fragments are distributed uniformly. Agglomeration, ranging from temporary clusterings in the dense phase to permanent agglomeration of fines to the surface of large particles is treated by various program options. In addition, the revised program allows for feeding solids to any of the fluidized beds.

### Revised Model Predictions

The revised model was validated using petroleum data from the FLEXICOKING prototype unit and commercial FLEXICOKING units. Satisfactory agreement was obtained with measured prototype and commercial FLEXICOKING fines losses and particle size distributions without requiring any adjustments. Model predictions for the EDS prototype and commercial scale FLEXICOKING units showed that particle size distributions were found to be very sensitive to the assumed minimum size for coke deposition. Reactor loss predictions were made by adjusting the minimum size for coke deposition to maintain a median particle size equal to petroleum experience. Loss predictions for the commercial unit at maximum throughput are similar to those obtained with the original model; but prototype reactor losses were four times higher than with the original model and about equal to those estimated for the commercial reactor. The model is now complete and will soon be available for evaluating EDS FLEXICOKING unit operations.

#### 5.4 FLEXICOKING Unit Gasifier Grid Design

##### Summary

The objective of this project is to evaluate gas/solids mixing near the grid of the FLEXICOKING unit gasifier in order to identify grid configurations which will minimize sintering and particle agglomeration. A flow visualization fluid bed unit is being modified for cold model studies. Initial tests will concentrate on characterizing the gas/solids motion in the grid region using commercial size petroleum FLEXICOKING gasifier grid caps. Subsequent testing will be directed to evaluating grid designs that may achieve more intense circulation, less stagnation and possibly greater scouring.

##### Flow Visualization Unit Modifications

An existing fluid bed unit is being adapted for grid studies. Fabrication of commercial size petroleum FLEXICOKING grid caps is near completion. The unit will be completed by August.

##### Future Work

After initial characterization of the current petroleum FLEXICOKING unit grid cap design, subsequent testing will be conducted using three standard caps as a reference and alternate cap designs at the other two locations. A fiber optics probe will be used for observations of solids motion.

## 5.5 FLEXICOKING Unit Reactor Feed Nozzle Development

### Summary

Atomization of coal liquefaction bottoms slurry in a typical FLEXICOKING unit reactor feed nozzle is difficult due to its high liquid viscosity and anticipated solids content of up to 30 wt%. Inadequate reactor feed dispersion could result in bogging and particle size control problems. Laboratory experience with typical petroleum FLEXICOKING unit feed nozzles and assessment of their operating characteristics when handling coal-derived feedstocks indicated nozzle capacity and atomization may require high pressures or very high steam dilution rates compared to typical petroleum feedstocks. Thus, this program has been planned to develop a nozzle configuration which offers adequate atomization of coal derived feedstocks with minimum gas requirements and pressure drop and without being plugged.

Tests of typical petroleum FLEXICOKING unit reactor feed nozzles with simulated solids-free and solids-containing EDS feedstocks showed severe two phase flow slugging and poor atomization at conventional petroleum FLEXICOKING flow conditions. Excessive superficial gas velocities were required for satisfactory performance. Recent lab tests have demonstrated that these feeds can be atomized satisfactorily at a third to half the gas rates required with petroleum FLEXICOKING nozzles by alternative nozzle designs. Based on these tests, a nozzle configuration embodying these modifications was recommended for the EDS FLEXICOKING prototype test program,

The detailed design and materials of construction for minimizing plugging and erosion are being defined by the Mechanical and Materials Engineering Division. A prototype nozzle will be fabricated for further testing at design solids concentrations of 20 to 30 wt%.

### Operation of Petroleum FLEXICOKING Unit Feed Nozzle was Unsatisfactory

Nozzle operation with typical petroleum feedstock was initially simulated using nitrogen and water. The effect of the high viscosity expected with coal liquefaction bottoms feedstocks was assessed using glycerin. Spray dispersion was photographed and nozzle pressure drop data were obtained over a wide range of flow conditions, and various nozzle configurations. Poor atomization and serious slugging were observed with the high viscosity feedstock at typical gas rates of petroleum FLEXICOKING unit reactor feed nozzles. High gas velocities were required to eliminate feed slugging. Modification of the nozzle tip produced only marginal improvements (October 1 through December 31, 1978, Quarterly - Technical Progress Report, (FE-2893-25). Pressure drop data gathered during the test program were used to develop a correlation to predict nozzle pressure drop for two phase flow and for high viscosity feeds.

### Gas Requirements Reduced by Improved Design

A modified nozzle configuration was tested next. Significant reductions in gas requirements, a third to a half the gas rates required previously, were achieved. Based on this configuration, three test nozzles were constructed and tested using different gas/liquid blending combinations. Also, for reference, a commercial air atomizing nozzle was tested.

### Nozzle Performance with Solids Containing Feeds Found Satisfactory

The improved nozzle was tested with glycerin containing up to 16 wt% of 200 $\mu$ m glass beads. No apparent adverse effects of solids on nozzle performance were observed. Only relatively minor increases in pressure drop were measured due to the increased slurry density. Based on these tests a prototype nozzle has been recommended for evaluation and testing in the Baytown prototype FLEXICOKING unit. It is presently being reviewed to define materials and the detailed mechanical design which will minimize plugging and erosion.

### Future Work

After the final design has been completed, a prototype nozzle will be fabricated and tested with concentrated slurries (>20 wt%) of fine (<70 $\mu$ m) glass beads. Examination of wear patterns inside the nozzle will be made to identify erosion patterns.

## 5.6 Scrubbing/Fractionation of Liquefaction Bottoms from FLUID COKING

The objective of this program is to determine whether conventional petroleum FLUID COKING design and operating criteria are applicable to the FLUID COKING of coal liquefaction bottoms or must be modified to account for any quality differences. To achieve this objective, the qualities of raw and heat soaked coker scrubber liquids from both liquefied coal and petroleum based FLUID COKING units were compared.

The Annual Technical Progress Report, covering July 1, 1977-June 30, 1978 (FE-2893-17), presented the background information on this program, the experimental procedure employed, and the results for Illinois coal based coker liquids. The current report summarizes the status of the work on Wyodak coal coker liquids.

The results of the analysis of the feed and product from the Wyodak coal based LSCU liquids heat soaking experiments have been received. The viscosity, benzene insolubles content, pyridine insolubles content, ash content and bench coking yields were measured for both the raw and heat soaked samples. A comparison of these analytical tests results with those previously obtained on Illinois coal based LSCU liquid products showed inconsistencies in several of the measurements. Therefore, additional heat soaking experiments and analytical tests on the feeds and products from these are currently in progress. The data from these additional tests will be used to resolve the inconsistencies.

## 5.7 Physical Properties of FLEXICOKING Streams

### Summary

The objective of this program is to determine whether generalized correlations for physical properties based primarily on standard inspection data are adequate for scrubber/fractionator process engineering; and, if necessary, initiate an experimental program to obtain supplementary data.

Samples of coker liquid fractions have been submitted for various inspection tests.

The specific heat data required for the preparation of a design basis model of the EDS Prototype FLEXICOKING Revamp Project have been completed. These specific heat data, along with the enthalpy data on several coal liquids measured at Colorado School of Mines(1), were used as the data base to generate a new set of parameters for the Watson and Nelson specific heat equation.

### Inspection Tests on the Coker Liquid Fractions

The narrow-cut Illinois and Wyoming coal liquid samples of FLEXICOKING products have been blended into wider cuts, 650-1000°F and 1000°F+. The physical property tests needed to characterize the streams are underway. The inspection tests will include GC distillation, density, viscosity, molecular weight, surface tension, elemental analysis, Conradson carbon, and aniline point.

### Specific Heat Correlation for Coal Liquids

Imperial Oil Limited (IOL) has completed the specific heat measurements on Illinois #6 vacuum bottoms, scrubber bottoms, heavy raw creosote oil (HRCO), and 20% HRCO/80% Illinois #6 vacuum bottoms. Enthalpy data on several coal liquids, including Western Kentucky Syncrude, Western Kentucky Syncrude light distillate, Utah Syncrude atmospheric distillate, Western Kentucky Sythoil distillate, SRC-I naphtha, and PAMCO middle distillate, have been reported by Colorado School of Mines (1). Analysis of these data has provided a check for the accuracy of new specific heat correlations and the original Watson and Nelson specific heat equation (2) to coal-based liquids. These specific heat data, covering the experimental range of 100 to 700°F, were also used as a data base to generate a new set of parameters for the Watson and Nelson equation. Results are summarized in Table 5-1. With the new parameters, the overall average absolute deviation in reproducing the specific heat is reduced from 12.53%, obtained with the original parameters, to 3.68%, and the bias from -12.23% to +0.25%.

TABLE 5-1

SPECIFIC HEATS OF COAL LIQUIDS: CORRELATION DEVIATION, %  
(TOTAL OF 63 DATA POINTS)

Method	Avg. Abs. Dev.	Bias	Max. Dev.
• New Data Book Correlation	6.46	-2.71	-18.78
• Original Watson and Nelson Equation	12.57	-12.23	-36.12
$C_p = \left( 0.35 + 0.055 K \right) \left[ 0.6811 - 0.308 S + (0.815 - 0.306 S) \frac{t}{1000} \right]$			
• Modified Watson and Nelson Equation	3.68	+0.25	+20.38
$C_p = \left( 0.465 + 0.0436 K \right) \left[ 0.4949 - 0.02479 S + (0.8117 - 0.3672 S) \frac{t}{1000} \right]$			

NOTE:  $C_p$  = specific heat, Btu/lb mol °F

$K$  = Watson characterization factor.

$S$  = specific gravity at 60°/60°F.

$t$  = temperature, °F.



### Future Work

Laboratory inspection data on the coker liquid fractions are expected by September 31, 1979. Analysis and determination of the adequacy of generalized physical property correlations is expected to be completed by end of 1979.

### References

1. Kidnay, A. J., Yesavage, V. F., "Enthalpy Measurement of Coal-Derived Liquids", FE-2035, Quarterly Technical Progress Reports to D.O.E., 1977 and 1978.
2. Watson, K. M., Nelson, E. F., "Symposium on Physical Properties of Hydrocarbon Mixtures", Ind. Eng. Chem. 25, (8), 880 (1933).

## 5.8 FLEXICOKING Unit Waste Withdrawal

Under this project, potential methods and equipment for withdrawing the solid waste streams from the FLEXICOKING Unit and preparing them for transfer to final disposal are being investigated. Work developed on this subject to date has centered in the following three areas.

### Moistened Loading of Dry Solids Streams

The prevention of fugitive dust emissions while loading out dry solids has been accomplished for many years in coal fired power plant fly ash handling by usage of a "dustless unloader". The many available devices rely upon fluffing the solids and exposing them to water sprays.

One of the simpler devices available for this purpose closely resembles a drum pelletizer in its construction and moisture requirements. The output of the device is essentially loosely agglomerated pellets at a moisture level suitable for landfill purposes.

For effective dust control, the solids feed must be maintained rigidly constant. To determine which of the available feed devices is most suitable for FLEXICOKING Unit tertiary cyclone fines and a mixture of bed/cyclone fines, a visit to a vendor was undertaken. It was determined that a fluidized orifice feeder would give the most constant and controllable solids feed rate for both materials tested.

Throughput of available dustless unloaders varies from 50 to over 150 dry tons of solids per hour depending upon the particular model and the material density. Water rate is dependent upon the solids throughput and required weight percentage. For FLEXICOKING Unit materials a 25 to 35 water weight percentage is required.

### Dry Loading of Dry Solids

In some instances it may be desired to maintain the solids dry during loading. The lime and cement industry often controls fugitive dust emissions during truck and barge loading by using extensible "dust free" loading chutes. Such devices funnel the solids into a small diameter flow stream inside an extensible fabric chute and pull a flow of dust entraining air counter to the solids flow. The dust laden air is pulled through a filter and the dust is recycled into the silo while the solids stream is caught by a funnel connected to the extensible chute. In the case of closed containers, adequate venting is required to maintain a proper airflow into the vessel and through the chute to entrain the dust.

The usual air rate in the devices is approximately twice the solids volumetric loading rate. The maximum solids loading rate can range from 130 cubic feet per minute to 3300 cfm depending upon the model chosen. Several variations on the basic design can tailor the devices to load trains, barges, trucks or drums.

#### Mixing/Drying

In some cases, gravity belt filter press (GBFP) cake (obtained by partially dewatering venturi scrubber slurry) may be required to be simultaneously mixed and dried with another solid waste stream from a FLEXICOKING Unit. The mixer/dryer is an equipment piece that can accomplish this task. Steam supplied to mixing discs on the machine provides heat for drying.

A series of vendor tests were run to attempt to a) mix GBFP cake with dry tertiary fines and dry the mixture to a handleable moisture content and b) dry GBFP cake to a handleable moisture content. Both tests were successful.

## ENGINEERING RESEARCH AND DEVELOPMENT

### ENGINEERING TECHNOLOGY DEVELOPMENT

#### 6. Materials and Environmental Engineering Technology

##### 6.1 Solids Waste Characterization for Handling and Disposal

Under this project, the handling and physical characteristics and the landfill and environmental disposal properties of the solid streams discharged from the FLEXICOKING of coal liquefaction vacuum bottoms are being determined in order to develop methods and facilities for handling and disposal of these streams from future commercial plants. Coke/ash materials from the IKG unit as well as coke from the FLEXICOKING of petroleum feed have been used to represent the streams from a commercial plant. Work developed on this project in the past year include:

##### Characterization Tests

During the past year, solids handling characterization testing was completed on dry samples of solid wastes from the integrated coker gasifier (IKG) unit at Baton Rouge as well as on three samples of boiler fly ash. The fly ash samples were tested to determine the applicability of existing fly ash handling and disposal technology. Physical and flow test data may be found in Tables 6-1 and 6-2 for the July-September, 1978 Quarterly Report. Review of the data indicates that the gasifier and reactor bed coke/ash streams are very flowable materials that should present no significant problems in design of storage and transfer systems. The overhead fines coke/ash streams, however, have poor flow characteristics and are similar to the characteristics of boiler fly ash.

Testing was also performed to determine the handling properties of one of the ashy IKG streams with various levels of moisture content. Test results with the overhead fines may be found in the July-September, 1978 Quarterly Report (FE-2893-21).

Bulk density and compressibility tests were run on mixtures of FLEXICOKING Unit bed coke and tertiary cyclone fines to determine their characteristics in combined storage. Bed coke and tertiary fines from the FLEXICOKING of petroleum feed were used. Results of these tests may be found in the January-March, 1979 Quarterly Report (FE-2893-29).

### Landfill Properties

A soils testing laboratory was engaged to conduct a series of tests on Illinois #6 and Wyodak bed/fines mixtures and power plant fly ash in order to develop data required for landfill design. The results of this test series are being reviewed and will be presented at a later date.

### Solid Wastes Environmental Testing

Leachates from materials that simulate expected EDS FLEXICOKING Unit solid waste were gathered in the laboratory, then analyzed for trace elements, organic carbon, flouride, and nitrates, and screened for polynuclear aromatic hydrocarbons (PNA's). Corrosivity of some of the leachates was also tested by measuring the corrosion rate of carbon steel in contact with the leachate. These analyses were then used to develop the potential hazard classification criteria. It must be realized that the classifications developed cannot be considered as final since neither federal nor state criteria for classifying wastes have been finalized.

Solid waste samples for the tests were obtained from the integrated coker gasifier (IKG) unit at Baton Rouge. The following samples were obtained for both Illinois #6 and Wyodak coal feeds: reactor chunk coke, gasifier purge coke, overhead fines, and overhead fines that were sieved into fractions representing tertiary cyclone fines and dewatered venturi scrubber sludge.

Two different leaching procedures were performed on each sample. They were the EPA Toxicant Extraction Procedure proposed in section 3001 of the 9/12/78 RCRA draft regulations, and the Texas Water Development Board procedure, specified in their Technical Guide No. 1 (revised 3/1/78).

To determine the hazard classification or toxicity of the leachate of each sample, the trace element concentrations of the leachates were measured against the existing EPA or Texas toxicity guideline. The two guidelines are not the same. The EPA guidelines are concerned with eight trace elements only. If the leachate concentration of any of the eight elements is higher than the standard set by the guidelines, that leachate is toxic. Texas criteria are not limited to eight elements. They include many trace elements, and the cumulative effect of the concentrations of these many elements determines the toxicity of the leachate.

Results of the tests are presented in Table 6-1. They indicate that, with one exception, each sample of tertiary cyclone fines, dewatered venturi scrubber sludge, gasifier purge coke, and reactor chunk coke leached by Texas procedures would be categorized as Class I, the most hazardous of the three Texas solid waste classes. In the Texas classification system, Illinois #6 reactor chunk coke would be categorized as Class II, an intermediate category.

TABLE 6-1

INDICATED HAZARD CLASSIFICATION

<u>SAMPLE</u>	<u>TEXAS HAZARD CLASSIFICATION</u>	<u>EPA HAZARD CLASSIFICATION</u>
<u>Wyodak EDS FLEXICOKING Unit Wastes</u>		
● Dewatered Venturi Scrubber Sludge (Simulated)	Class I <sup>(1)</sup>	No Test
● Tertiary Cyclone Fines (Simulated)	" "	No Test
● Overhead Fines (Unsieved)	" "	Non-hazardous
● Reactor Chunk Coke	" "	" "
● Gasifier Purge Coke	" "	" "
<u>Illinois #6 EDS FLEXICOKING Unit Wastes</u>		
● Dewatered Venturi Scrubber Sludge (Simulated)	Class I	No Test
● Tertiary Cyclone Fines (Simulated)	No Test	No Test
● Overhead Fines (Unsieved)	Class I	Non-hazardous
● Reactor Chunk Coke	Class II <sup>(2)</sup>	" "
● Gasifier Purge Coke	Class I	" "

(1) Most hazardous of the three Texas classifications.

(2) An intermediate classification.

In addition to trace element test results, corrosion tests and PNA screenings were performed on Texas leachates. The corrosion tests, conducted according to National Association of Corrosion Engineers' Standard TM-01-69, found that all samples tested would easily meet Texas specifications of <250 mils per year corrosion of carbon steel. PNA screening of Texas leachates detected no PNA's at the parts per billion level.

Analysis of leachates per the draft EPA procedure indicated that all the wastes tested meet the non-toxic criteria specified in the 12/18/78 draft RCRA regulations.

The conflicting answers resulting from use of the tentative EPA and Texas leaching test and analysis criteria make it unclear whether solid wastes from the FLEXICOKING of EDS vacuum bottoms will be considered toxic. As EPA and other criteria are further developed the answer should become clearer. Also, if the prototype FLEXICOKING Unit at Baytown is programmed to run on ECLP vacuum bottoms, an opportunity will develop to collect larger and more representative waste solid samples for further analysis.

## 6.2 Environmental Control - Water

### Summary

As part of the development of the Exxon Donor Solvent coal liquefaction process to a state of commercial readiness, a long range R&D program was proposed to generate the data for evaluating the impact of a commercial EDS plant on the water environment and for designing a commercial-scale wastewater treating system. The current program is shown in Figure 6-1, and reflects changes resulting from a re-evaluation of the overall program conducted in 1978 (described later in this report).

Results of the program to date support our conclusion that a proposed treatment scheme of sour water stripping, solvent extraction, equalization, secondary oil removal, air activated sludge, filtration, and carbon adsorption should be able to meet most, if not all, of the projected quality requirements for discharge to surface waters. However, heavy element discharge regulations, which appear to be getting more strict, may require the addition of a heavy element removal system to the treating sequence. In addition, if the discharge of total dissolved solids is severely restricted (e.g. by locating the plant in a water-short or sensitive environment), it may become necessary to reuse the final treated effluent in the cooling tower and to evaporate the cooling tower blowdown.

Accomplishments for the July, 1978, through June, 1979, period included the following:

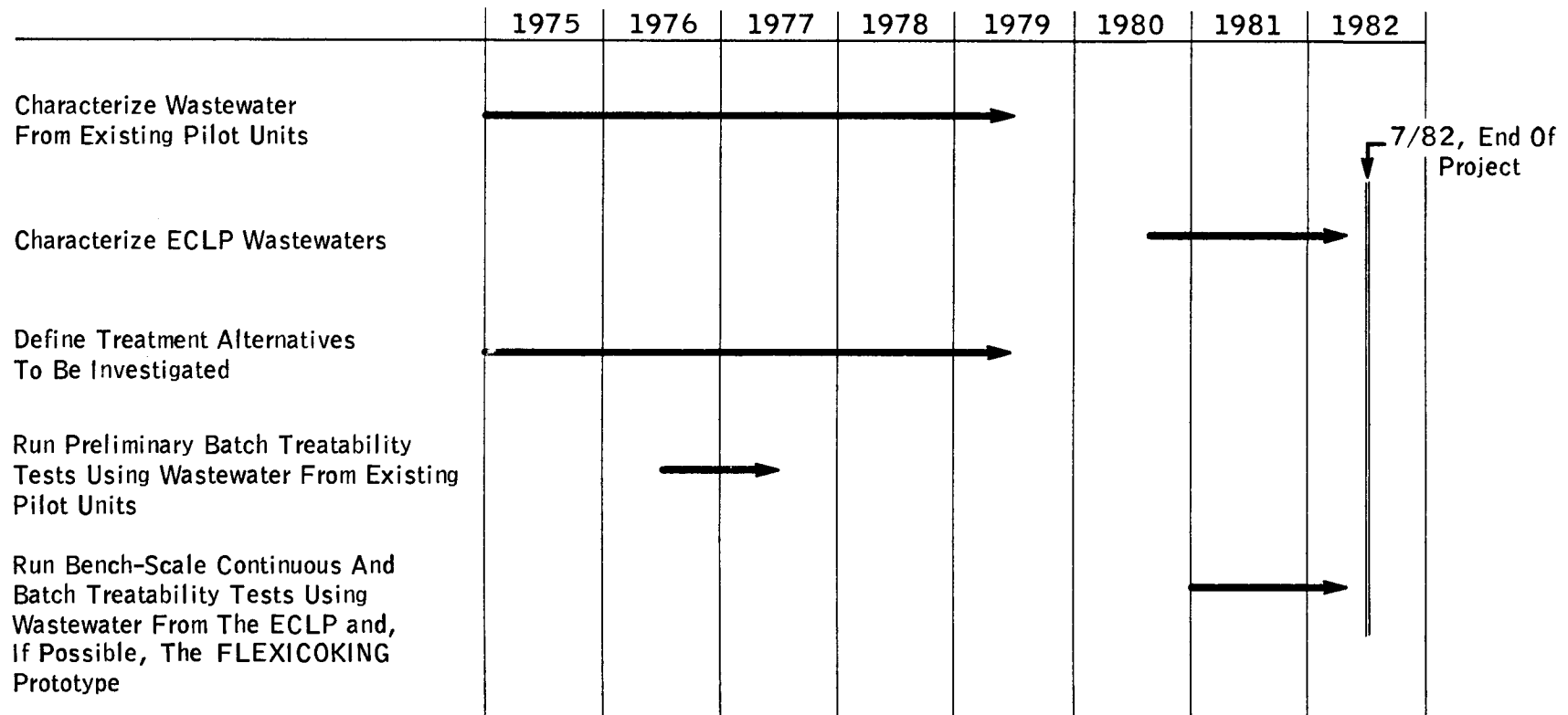
- Characterization of samples from the coal liquefaction pilot plant (CLPP), continuous stirred coker unit (CSCU), and integrated coker/gasifier (IKG) during Wyoming operations.
- Initiation of a comparison of direct aqueous gas chromatography and extraction/gas chromatography for analyzing the organic components in EDS wastewaters.
- Laboratory testing of an alternative (to isopropyl ether) solvent for liquid-liquid extraction of phenolics from EDS process wastewaters.
- Preliminary estimation of the composition of untreated wastewaters from a commercial EDS plant using Wyoming coal.
- Re-evaluation of the long-range EDS wastewater program.
- Preliminary planning of the testing to be done on samples from the ECLP.

Each of the accomplishments is discussed below:



Figure 6-1

TIMETABLE FOR COAL LIQUEFACTION WASTEWATER STUDIES



### Characterization of Samples from Existing Pilot Units during Wyoming Operations

This activity was carried out to provide data for preliminary assessment of the effect of a Wyoming-coal-fed EDS plant on the water environment, and to allow process selection for wastewater clean-up. As part of this activity, a wastewater sample (YT123) was obtained from the continuous stirred coker units during a Wyoming CLPP bottoms operation. Analysis of this sample indicated that the level of mono-hydric phenolics in this wastewater was ~40% greater than previously found. If this higher level of mono-hydric phenolics is produced in a commercial plant, it should not significantly affect the size of biological oxidation or carbon adsorption units, since the liquid-liquid extraction of process wastewater effectively removes the mono-hydric phenolics prior to downstream treatment.

In another part of this activity, analysis of a wastewater sample obtained from the integrated coke/gasifier (IKG) unit during Wyoming operations indicated that little organic contamination should be present in the heater/gasifier wastewaters from a commercial plant.

Also analyzed during this period were wastewater samples obtained from the liquefaction and solvent hydrogenation sections of the coal liquefaction pilot plant (CLPP) during Wyoming operations. These analyses showed that Wyoming and Illinois wastewater contain similar types of organic and inorganic constituents, although in different proportions. Based on these analyses, and the Phase IIIA batch treatability studies, all of the compounds identified in the samples should be effectively removed during treatment in a scheme similar to the one described earlier in this report.

### Comparison of Direct Aqueous Gas Chromatography and Extraction/Gas Chromatography

This was done to see if a change in methods would improve the accuracy and reproducibility of analyses for organic contaminants. The EPA has used the extraction GC approach during their search for priority pollutants in industrial effluents. A sample of CLPP liquefaction section(A8) wastewater, previously analyzed by direct aqueous injection gas chromatography as part of the characterization program mentioned above, was also analyzed by extracting the sample at pH = 12 and pH = 2 with methylene chloride followed by gas chromatography of the extracts. Concentrations of organics found in the CLPP wastewater sample using this method were considerably lower than concentrations previously found by direct aqueous injection gas chromatography. It is possible, however, that sample aging may have caused the decrease. Therefore, parallel tests are being run using both the direct aqueous injection and extraction methods on a sample obtained from the Large Stirred Coker Unit (LSCU) in order to more accurately compare the results obtained from the two methods.

### Laboratory Tests of an Alternative Commercially Available Liquid-Liquid Extraction Process

The present scheme for removing phenolic materials from the EDS process is isopropyl ether extraction. During this past year, a vendor performed batch tests using his alternative solvent process on unstripped samples of liquefaction section wastewater obtained from the CLPP during Wyoming operations (10/24/78). Analyses of the water both before and after extraction were done by ER&E. Results (shown in Table 6-2) indicate very good removal of the phenolic materials. The calculated distribution coefficient,  $k_D$ , for phenol averaged 93 in this test. This value exceeds the equilibrium coefficient calculated during earlier tests using isopropyl ether ( $K_D = 45$ ). Considering this result and the fact that the process is commercially proven, it should be considered as a candidate for a full-scale EDS plant. Further evaluation of this process will be done on a larger scale during the treatability tests to be conducted on ECLP wastewater.

### Preliminary Estimates of the Composition of Wastewater from a Commercial EDS Plant using Wyoming (Wyodak) Coal

Organic composition of the process wastewater from a Wyodak coal EDS plant was estimated by adjusting the available Wyodak wastewater data from the pilot units using a ratio of the composition obtained from the Illinois computer simulation studies (See January-March, 1978 Quarterly Technical Progress Report)\*to the Illinois wastewater data from the operating pilot units. Table 6-3 shows the estimated organic and total dissolved solids (TDS) composition of wastewater after stripping and extraction.

Two implications of the estimates are noteworthy. First, because of its higher BOD<sub>5</sub> concentration, the Wyodak wastewater may require the addition of a roughing biological treatment unit upstream of the activated sludge unit in the proposed treatment scheme for an Illinois coal EDS plant. Second, depending on the receiving body of water, the higher TDS of Wyodak process wastewater may cause the final effluent to exceed the allowable quality criteria. If this situation occurs, additional treatment (e.g., reuse of the treated effluent as cooling tower makeup and subsequent evaporation of the blowdown) may be needed.

### Re-evaluation of the Long-Range Wastewater R&D Program

The purpose of the re-evaluation was to develop a plan to assure that all information needed to design a reliable wastewater treating system for a commercial EDS plant would be available by mid-1982. The first conclusion from the re-evaluation was that extensive bench scale treatability testing tentatively scheduled late in the fourth quarter of 1978 should be

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\*FE-2893-12

TABLE 6-2

RESULTS OF BATCH TESTS OF AN ALTERNATIVE  
SOLVENT FOR PHENOLICS REMOVAL (1)

<u>Component</u>	<u>Untreated Wastewater</u>	<u>Treated Wastewater (1)</u>	
		<u>After Two Extractions</u>	<u>After Five Extractions</u>
Phenol (mg/l)	1600	6.6	N.D.(<0.5)(2)
Cresols (mg/l)	960	N.D.(<1)	N.D.(<1)
pH(3)	8.1	6.5	6.5

Notes:

- (1) A single wastewater sample was extracted a total of five times with a solvent in laboratory glassware. After each extraction the water phase was separated from the solvent phase and the water was re-extracted using fresh solvent. A solvent to water ratio of 0.13 by volume (0.1 by weight) was used in each extraction.
- (2) N. D. means not detected. Detection limit given in parentheses.
- (3) The wastewater pH was adjusted to 6.5 prior to extraction.

TABLE 6-3

PRELIMINARY ESTIMATES OF PROCESS  
WASTEWATER QUALITIES <sup>(1)</sup> <sup>(2)</sup>

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	<u>Wyodak</u>	<u>Illinois (Mine #1)</u>
Flow, gpm	1980	1420
Phenol, ppm	10	8
Resorcinol, ppm	12	36
Alkyl Resorcinol, ppm	5	5
Organic Acids, ppm	3600	1500
TDS, ppm <sup>(3)</sup>	4200	2400
NaHCO <sub>3</sub> , % of TDS	99	75
BOD <sub>5</sub> , ppm	3300	1410
TOC, ppm	1700	725

Notes:

- (1) After stripping and extraction.
- (2) Estimates are for a 15,000 ton/day (as received coal) EDS Pioneer plant.
- (3) The estimate for TDS included the amount of NaOH to be added to the sour water stripper to remove ammonia, and the amount of bicarbonate that would be formed by the conversion of organic materials during downstream biological treatment.

delayed until 1980-1981, when the ECLP will be running. The ECLP and the proposed FLEXICOKING prototype unit should provide wastewaters more like those in a commercial plant than can presently be obtained.

The second conclusion was that bench-scale rather than pilot-scale treating tests should be run using wastewater from the ECLP and the proposed FLEXICOKING prototype. Although the more expensive pilot-scale testing had a potential benefit-to-cost ratio of about 2:1, this ratio was judged too low to justify the substantial additional cost (~750 k\$) of the pilot-scale tests. Bench scale tests should provide enough information to design a dependable wastewater treating plant which meets effluent requirements.

#### Preliminary Planning of ECLP Tests

During the past quarter, we made preliminary plans for sampling and analyzing the wastewaters from the ECLP. Tentative plans call for the following studies:

- A study to determine the short-term variability of the composition of the ECLP wastewaters - Knowing the variability will allow us to determine the proper number of samples needed to adequately characterize the wastes. Ammonia measurements will be made to obtain information on the variability of the concentrations of inorganic constituents, while total organic carbon will be used to characterize the variability of the concentrations of organic constituents. Six process wastewater streams will be sampled hourly for thirty six hours at the beginning of steady state ECLP operations on Illinois coal.
- A study to determine the untreated wastewater characteristics from the ECLP during steady state operations - These results will form a design basis for a commercial treatment plant and will allow us to verify the computer models used to predict the untreated wastewater composition for a commercial EDS plant. Six process wastewater streams will be sampled once/shift for a maximum of seven days over a three week period during steady state ECLP operations on each of the three coals. A daily composite sample will be made for each source from the three samples collected each day. Samples will be analyzed for organic and inorganic contaminants.
- A bench-scale treating study to verify the applicability of the proposed treatment scheme and to develop design data for a commercial wastewater treating plant - This will include the collection and pretreatment of wastewaters from the ECLP and proposed FLEXICOKING prototype. Combined waters would be tested in continuous and batch bench-scale experiments after stripping, neutralization, extraction, and oil removal. Only wastewaters from an Illinois (bituminous) coal operation and a subbituminous coal operation will be tested, since this

should represent the range of wastewater qualities from the first commercial-scale EDS plant. Treatability experiments involving Illinois coal wastewaters will include activated sludge and activated carbon isotherm tests, while the treatability experiments involving the sub-bituminous coal wastewaters will include a rotating biological contactor (biodisk), activated sludge, and activated carbon isotherm tests. In addition, simulated activated carbon effluent will be examined for corrosion, scaling, and biological fouling tendencies to give a preliminary idea of the feasibility of its reuse as cooling tower makeup.

#### Future Work

Plans for the remainder of 1979 include the following:

- Detailed planning of the treating tests to be conducted on ECLP wastewater.
- Complete analysis of recently received samples of Illinois and Wyoming wastewaters from the Large Stirred Coker Unit (LSCU) during operations at steam-to-feed ratios closer to a commercial FLEXICOKING unit.
- Complete analyses of a sample of LSCU wastewater using both direct injection GC and extraction GC methods to determine the better analytical method.

### 6.3 Environmental Control - Atmospheric Emissions

#### Emissions from Combustion of Wyodak Coal Liquids Analyzed for Polynuclear Aromatics

Combustion tests were performed on two EDS heavy fuel oils produced from Wyodak coals. The results of analyses of these samples for polynuclear aromatic matter (PNA) are shown in Table 6-4. The results of previously-reported similar tests on EDS fuel oil derived from Illinois coal are included for comparison. There was concern about the earlier test conditions that the sample size was not large enough and/or that the analytical methods may not have been sensitive enough. Consequently, a newer, more sensitive method of analysis was used for the Wyodak coal liquids tests, the Single-Ion Mode GC/Mass Spectroscopy method. These analyses, performed at Battelle-Columbus Laboratories, were confirmed for one sample by the formerly-used GC/UV method of ER&E. The overall agreement between the two methods is within experimental accuracy.

The Wyodak coal-liquids produced higher concentrations of three- and four-ring compounds than the Illinois coal-liquids. The significance of these results is being evaluated.

Additional analyses were made of the EDS heavy fuel oils for PNA content to enable correlation of PNA concentration in the fuel oil with PNA emissions in the flue gas. These results are described under Product Quality Studies.

Detailed plans developed for the ECLP test program included tests of Slurry Preheat Furnace decoking, particulate emissions from coal-handling facilities, emissions passing the Venturi Scrubber for the Sandvik belt, and tests to enable accurate design of a High Energy Venturi Scrubber for the gas swept mill.

#### Noise Survey and Sampling Planned

New, more stringent, OSHA noise regulations have not yet been promulgated, and it is doubtful that they will be before the end of 1979. In 1978 the National Institute for Occupational Safety and Health (NIOSH) recommended to OSHA that new plants should be designed to a criterion of 85 dBA for 8-hour worker exposure, but no action has been taken on this recommendation.

Plans developed for the ECLP test program include noise surveys at the plant and adjacent communities. In addition, tests will be made of the gas swept mill and 600 psi let-down valve. Previous baseline community noise data will now require updating, since a large new plant will start up at Baytown before ECLP. This updating will be



accomplished economically by cooperation with a similar survey being conducted for the new Baytown Plant.

Air Pollution Impact Estimated for  
Commercial-Scale Plant

Estimates have been updated of expected emissions of conventional air pollutants ( $\text{SO}_2$ ,  $\text{NO}_x$ , CO, hydrocarbons and particulates) from a commercial-scale EDS plant. These were made for the revised plant configuration specified in the EDS Study Design Update, and are reported in Section 1.

TABLE 6-4

PNA's IN EXHAUST GASES  
FROM COMBUSTION SOURCES  
(ng/m<sup>3</sup>) (1)

COMPOUND (CARCINOGENIC ACTIVITY) (2)	NUMBER OF CARBON RINGS	MATE (3) VALUES	ANALYSIS=	WYODAK	WYODAK		EDS ILLINOIS
				350-1000°F BATTELLE	350°F+	ER&E	400°F+ ER&E
			(4)				
Phenanthrene (-)	3	1,590,000	}	106,000	>459,000	450,000	-
Anthracene (-)	3	56,000,000					-
Methyl Anthracenes (?)	3	NR	}	13,500	167,000	-	-
Phenanthrenes (-)	3	1,590,000					-
Fluoranthene (-)	3	90,000,000		24,000	187,000	164,000	15,000
Methyl Pyrene/Fluoranthene (-)	4	NR		1,360	62,400	-	-
Pyrene (-)	4	230,000,000		7,080	161,000	140,000	1,500
Benzo (c) Phenanthrene (+++)	4	27,000,000		230	2,780	-	-
Benz (a) Anthracene (++)	4	45,000	}	1,630	65,300	49,000	<450
Chrysene (+)	4	2,220,000					
Methyl Chrysenes (-)	4	1,800,000		110	16,300	-	-
7, 12-Dimethyl Benz (a) Anthracene (+++)	4	260		28	1,950	<270	<200
Benzo Fluoranthenes (- or ++)	5	1,600,000 to 900,000	}	<4	2,680	15,000	<3,800
Benzo (e) Pyrene (+)	5	3,040,000		4	3,000	7,700	<770
Benzo (a) Pyrene (+++)	5	20					
Perylene (-)	5	NR		8	-	1,500	<490
Indeno - (1, 2, 3-c, d) Pyrene (+)	5	1,630,000		4	390	<8	-
Benzo (ghi) Perylene (-)	6	NR		4	790	610	<730
Dibenz (a,h) Anthracene (+++)	6	93		8	5	<13	-
Dibenz (a,i) Pyrene (++)	6	43,000	}	8	85	-	-
Dibenz (a,h) Pyrene, (?)	6	3,700,000					
Coronene (-)	7	NR		16	28	80	50

NR = not reported

(1) Standard Conditions - Dry at 15°C, 1 atm.

(2) Plus signs indicate degree of carcinogenic activity.

Minus sign indicates no carcinogenesis has been proved in animals.

(3) Minimum Acute Toxicity of Effluent (MATE). See EPA 600/7-77-136a,b

(4) Some overlap between compounds occurs in the analysis.

## 6.4 Construction Materials - Liquefaction and FLEXICOKING

### Summary

#### 1. CLPP Materials Evaluation Program

Series V in-situ corrosion tests have been completed. Corrosion rates in the liquefaction reactor were determined in new Test Site 10, indicating appreciable attack on 5 Cr alloy steel. Flow velocity has been verified as a process variable having a marked accelerating effect on metal loss (erosion-corrosion).

#### 2. ECLP Materials Evaluation Program

The ECLP integrated materials evaluation program has been enlarged and updated. This 7-part program consists of corrosion racks, corrosion probes, component materials tests, NDT inspection, and stream sampling. For additional corrosion/erosion monitoring, two cooperative programs with National Laboratories on stress corrosion cracking and slurry erosion have been initiated.

#### 3. State-of-the-Art Materials Technology

ER&E continues to take an active role in the activities of technical bodies guiding and studying synthetic fuels materials problems. Major activities have been: preparing a coal liquids laboratory corrosion test program, presenting a paper on the EDS materials program, visiting the SRC pilot plant in Wilsonville, and attending various technical symposia.

#### 4. IKG Corrosion Tests

Corrosion tests in the IKG Unit have been completed. This integrated coking/gasification pilot unit, located at Baton Rouge, LA, simulates process conditions anticipated in EDS FLEXICOKING. Results from IKG testing indicate low rates of attack of gasifier internals and heater overhead components.

#### 5. FLEXICOKING Prototype Inspection

Inspection of the FLEXICOKING prototype showed it to be in generally good condition. Specific findings will be reflected in design specifications for revamping the prototype for EDS operation.

#### 6. High Temperature Erosion Testing

A cooperative testing program has been initiated with Lawrence Berkeley Laboratory to study high temperature gas/solids erosion of interest to EDS FLEXICOKING application. Preliminary tests are being conducted, aimed at understanding of basic test variables and determining equipment reproducibility.

## 1.0 CLPP Materials Evaluation Program

This section presents and discusses the results of materials evaluation in CLPP during the reporting period, broken down into the following three topics:

- In-Situ Corrosion Testing
- Effect of Velocity
- Component Failure Analysis

### 1.1 In-Situ Corrosion Testing

During March-December 1978, the fifth series of corrosion test samples (tubing sections and coupon specimens) were exposed in CLPP. Feed during this test period was Wyodak coal. The results from all five Test Series are tabulated in Table 6-5. Sample locations for all ten CLPP Test Sites are marked on Figure 6-2. As anticipated, rates are generally lower than with Illinois coal, and are comparable to a previous Wyodak run (Test Series III).

Test Series V contains the first evaluation of coupons exposed in the liquefaction reactor (Test Site 10). Since flow velocity is only about 0.5 ft/sec, the rates measured represent corrosion losses, as contrasted to erosion-corrosion wastage registered by tubing specimens in slurry test sites.

### 1.2 Corrosivity as a Function of Velocity

Figure 6-3 presents the effect of velocity on metal loss in liquefaction reactor slurry service. The points plotted represent data from Test Sites 1, 2, and 10. All of these are at substantially the same temperature and may contain up to 40-50% solids. Test Site 10 is located inside the liquefaction reactor, representing very low flow rates (less than 1 ft/sec). The range of velocities investigated is 0.5 fps to 35 fps.

The general trend apparent from Figure 6-3 shows a sharp increase of metal loss at higher flow velocities. Such response is characteristic of so-called erosion-corrosion where corrosion is accelerated by erosion and/or abrasion, often in a synergistic manner. As expected, stainless steel performs better than chrome steel. This improvement is attributed principally to inherently better corrosion resistance, since Cr-Ni stainless steels are not known for good erosion resistance.

TABLE 6-5

## CLPP IN-SITU CORROSION TEST SUMMARY

Test Site (See Figure 6-2)	Test Series <sup>(1)</sup>	Type Coal	Test Specimens	Tube Dimension		Process Environment	Temperature (°F)	Pressure (psig)	Exposure Time (hrs)	Material	Corrosion Rate (mils/year) <sup>(2)</sup>	
				Dia.	Wall						ΔThickness	ΔWeight
TS-1-1	1	Illinois	Tubing Sections	(")	(")	Solvent/coal slurry + H <sub>2</sub>	830	1650	282	304	93	28
TS-1-2				3/8	0.049		830	1650	282	316	93	194
TS-1-3	2	3/8		0.049	840		1650	47	5 Cr	370	120	
TS-1-4	3	Wyoming		3/8	0.065		820	1650	591	304	0	(+)
TS-1-5				3/8	0.049		820-840	1650	965	316	0	3
TS-1-6				3/8	0.049		820-840	1650	965	5 Cr	63	64
TS-1-7	5	Wyoming		5/16	0.049		820-840	1650	1407	316	19	12.5
TS-1-8				3/8	0.065		820-840	1650	1407	316	19	6.2
TS-2-1	1	Illinois	Tubing Sections	1/2	0.065	Solvent/coal slurry + H <sub>2</sub>	840	1620	282	304	0	27
TS-2-2				1/2	0.065		840	1620	282	316	(+)	(+)
TS-2-3	2	1/2		0.065	840		1620	485	5 Cr	90	29	
TS-2-4		1/2		0.065	840		1620	485	316	0	(+)	
TS-2-5	3	Wyoming		1/2	0.083		770-820	1620	965	304	5	1
TS-2-6				1/2	0.065		770-820	1620	965	316	0	~
TS-2-7				1/2	0.065		770-820	1620	965	5 Cr	18	11
TS-3-1	1	Illinois		Test rack Coupons				Liquefaction water condensate	60-100	2	282	CS
TS-3-2			60-100			2			282	12 Cr	-	3.0
TS-3-1			60-100			2			282	304	-	2.2
TS-3-2	2		60-100			2			485	Monel	-	2.7
TS-3-2			60-100			2			485	Alloy 20	-	1.9
TS-3-2			60-100			2			485	Titanium	-	3.6
TS-3-3	3	Wyoming	60-100			2			965	CS	-	0.6
TS-3-3			60-100			2			965	304	-	0.3
TS-3-3			60-100			2			965	Alloy 20	-	0.3
TS-3-4	5	Wyoming	60-100			2				CS	-	0.3
TS-3-4			60-100			2			2639	304	-	0.1
TS-3-4			60-100			2				316	-	0.2
TS-4-2	1+2	Illinois	Tubing Sections	3/4	0.035	Vacuum tower bottoms	550	0	408	316	0	4
TS-4-3	3	Wyoming		3/4	0.035		580	0	180	CS	150	23
TS-4-4	4	Illinois		3/4	0.065		580-650	0	794	CS	0	15

T A B L E 6-5 (C o n t i n u e d)

Test Site (See Figure 6-2)	Test Series <sup>(1)</sup>	Type Coal	Test Specimens	Tube Dimension Dia. Wall		Process Environment	Temperature (°F)	Pressure (psig)	Exposure Time (hrs)	Material	Corrosion Rate (mils/year) <sup>(2)</sup>	
				(")	(")						ΔThickness	ΔWeight
TS-5-1	1&2	Illinois	Test rack Coupons			Hydrotreating water condensate	60-100	2	1064	CS	-	1.6
TS-5-1							60-100	2	1064	12 Cr	-	1.4
TS-5-1							60-100	2	1064	304	-	1.1
TS-5-2	1976						60-100	2	761	Monel	-	1.4
TS-5-2							60-100	2	761	Alloy 20	-	0.7
TS-5-2							60-100	2	761	Titanium	-	0.9
TS-5-3	3	Wyoming					60-100	2	1203	CS	-	0.8
TS-5-3							60-100	2	1203	304	-	0.4
TS-5-3							60-100	2	1203	Alloy 20	-	0.3
TS-5-4	5	Wyoming					60-100	2		CS	-	0.3
TS-5-4							60-100	2	2639	304	-	0.2
TS-5-4							60-100	2		316	-	0.2
TS-6-1	1	Illinois	Tubing Sections	1/2	0.035	Coal liquid slurry to vacuum tower	600	25	249	CS	250	190
TS-6-2							600	25	249	304	0	(+)
TS-6-3				1/2	0.035		600	25	249	316	35	(+)
TS-6-4				1/2	0.035		600	25	249	12 Cr	0	36
TS-6-5				1/2	0.035		600	25	249	5 Cr	0	16
TS-6-6	3	Wyoming		1/2	0.035		700	25	180	CS	150	17
TS-6-7				1/2	0.035		700	25	180	304	240	(+)
TS-6-8				1/2	0.035		700	25	180	12 Cr	0	2
TS-6-9				1/2	0.035		700	25	180	5 Cr	0	15
TS-6-10				1/2	0.035		600-700	25	770	CS	23	33
TS-6-11				1/2	0.035		600-700	25	770	304	0	10
TS-6-12				1/2	0.049		600-700	25	770	316	23	(+)
TS-6-13				1/2	0.035		600-700	25	770	12 Cr	0	0
TS-6-14				1/2	0.035		600-700	25	770	5 Cr	0	7
TS-6-15				1/2	0.035		600-700	25	770	5 Cr	0	7
TS-6-16	4	Illinois		1/2	0.035		700	25	794	5 Cr	22	8
TS-7-1	1	Illinois	Tubing Sections	3/8	0.049	Coal liquid slurry letdown line (intermittent service)	800	1500-150	21	304	1700	(+)
TS-7-2				3/8	0.049		800	1500-150	3	316	3000	(+)
TS-7-3	2			3/8	0.049		800	1500-150	18	5 Cr	0	2000
TS-7-4				3/8	0.049		800	1500-150	20	316	(+)	900
TS-7-5	3	Wyoming		3/8	0.049		750-800	1500-150	9	316	970	120
TS-7-6				3/8	0.049		750-800	1500-150	9	5 Cr	0	2000
TS-7-7				3/8	0.065		750	1500-150	23	5 Cr	0	920
TS-7-8				3/8	0.049		750	1500-150	23	316	380	340

TABLE 6-5 (Continued)

Test Site (See Figure 6-2)	Test Series <sup>(1)</sup>	Type Coal	Test Specimens	Tube Dimension		Process Environment	Temperature (°F)	Pressure (psig)	Exposure Time (hrs)	Material	Corrosion Rate (mils/year) <sup>(2)</sup>	
				Dia. (")	Wall (")						ΔThickness	ΔWeight
TS-8-1	1&2	Illinois	Tubing Sections	1/2	0.035	Heavy vacuum gas oil	500	2	464	316	(+)	2
TS-8-2				1/2	0.035		500	2	464	CS	57	49
TS-8-3	4			1/2	0.049		500	2	795	5 Cr	11	7
TS-9-1	1	Illinois	Tubing Sections	1/2	0.065	Coal liquids/raw creosote oil	RV-55	1650	317	316	55	9
TS-9-2				1/2	0.065		RV-55	1650	317	304	0	4
TS-9-4	2			1/2	0.065		RV-55	650	747	5 Cr	35	30
TS-9-5				1/2	0.065		RV-55	650	747	316	23	5
TS-9-6	3	Wyoming		1/2	0.065		RV-55	1650	1203	304	5	3
TS-9-7				1/2	0.065		RV-55	1650	1203	316	0	(+)
TS-9-8				1/2	0.065		RV-55	1650	1203	5 Cr	9	12
TS-9-9	4	Illinois		1/2	0.083		RV-55	1650	978	304	18	6
TS-10-1	5	Wyoming	Coupons			Slurry + H <sub>2</sub> (Liquefaction Reactor)	840	1650		5 Cr	-	66.5,66.4, 66.0
TS-10-2							840	1650		304	-	3.7,4.2
TS-10-3							840	1650	452	304 SE <sup>(3)</sup>	-	5.7
TS-10-4							840	1650		316	-	6.8
TS-10-5							840	1650		321	-	8.4

NOTES: (1) Test series 1 = December 1976-February 1977  
 Test series 2 = January 1977-April 1977  
 Test series 3 = April 1977-July 1977  
 Test series 4 = July 1977-September 1977  
 Test series 5 = March 1978-December 1978

(2) (+) entries in "Corrosion Rate" column indicate measured gain in weight or wall thickness. Wall thickness measurements were discontinued after Test Series IV because they give less reliable results than weight loss measurements.

(3) SE - sensitized

Figure 6-2  
CLPP CORROSION TEST SITES

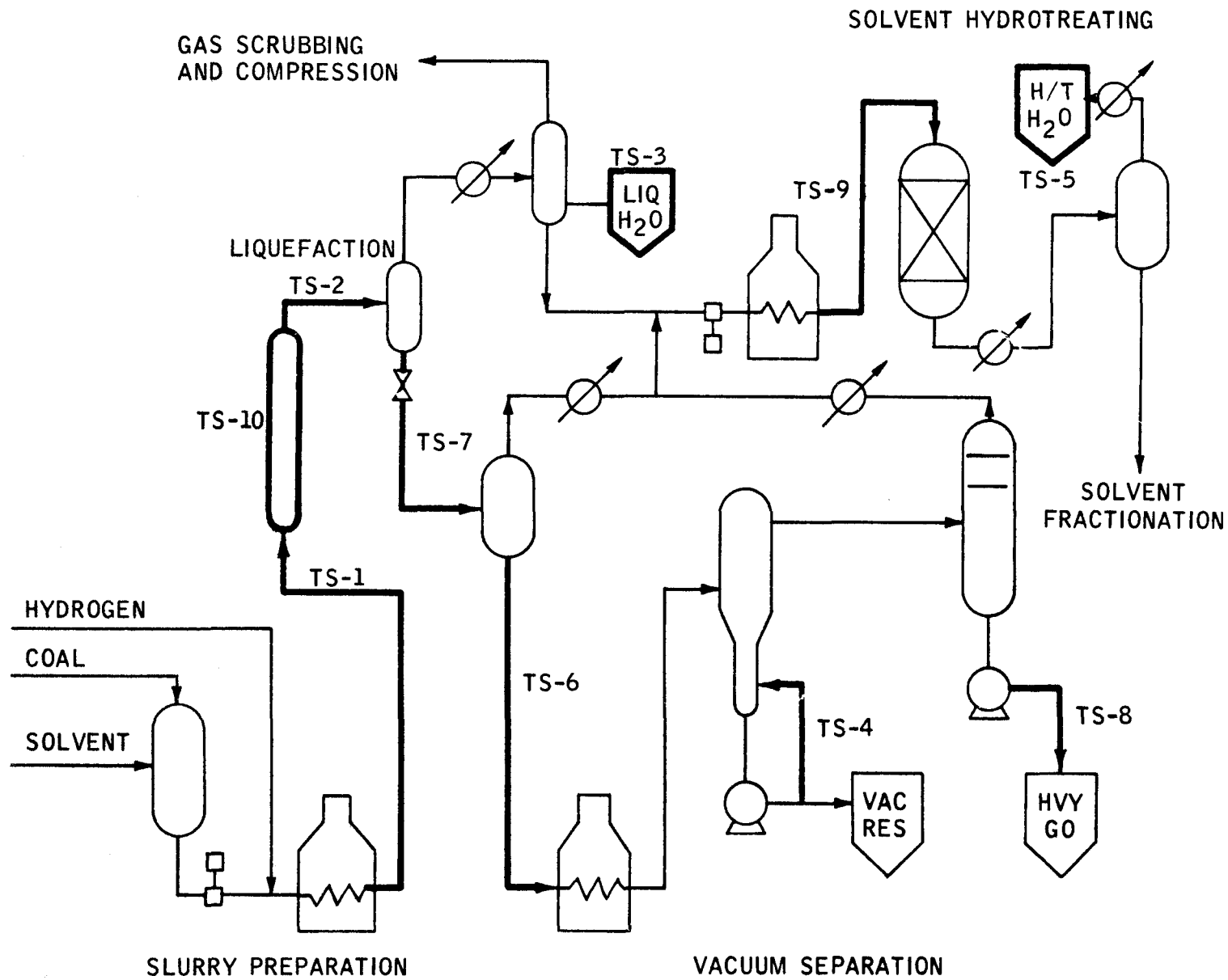
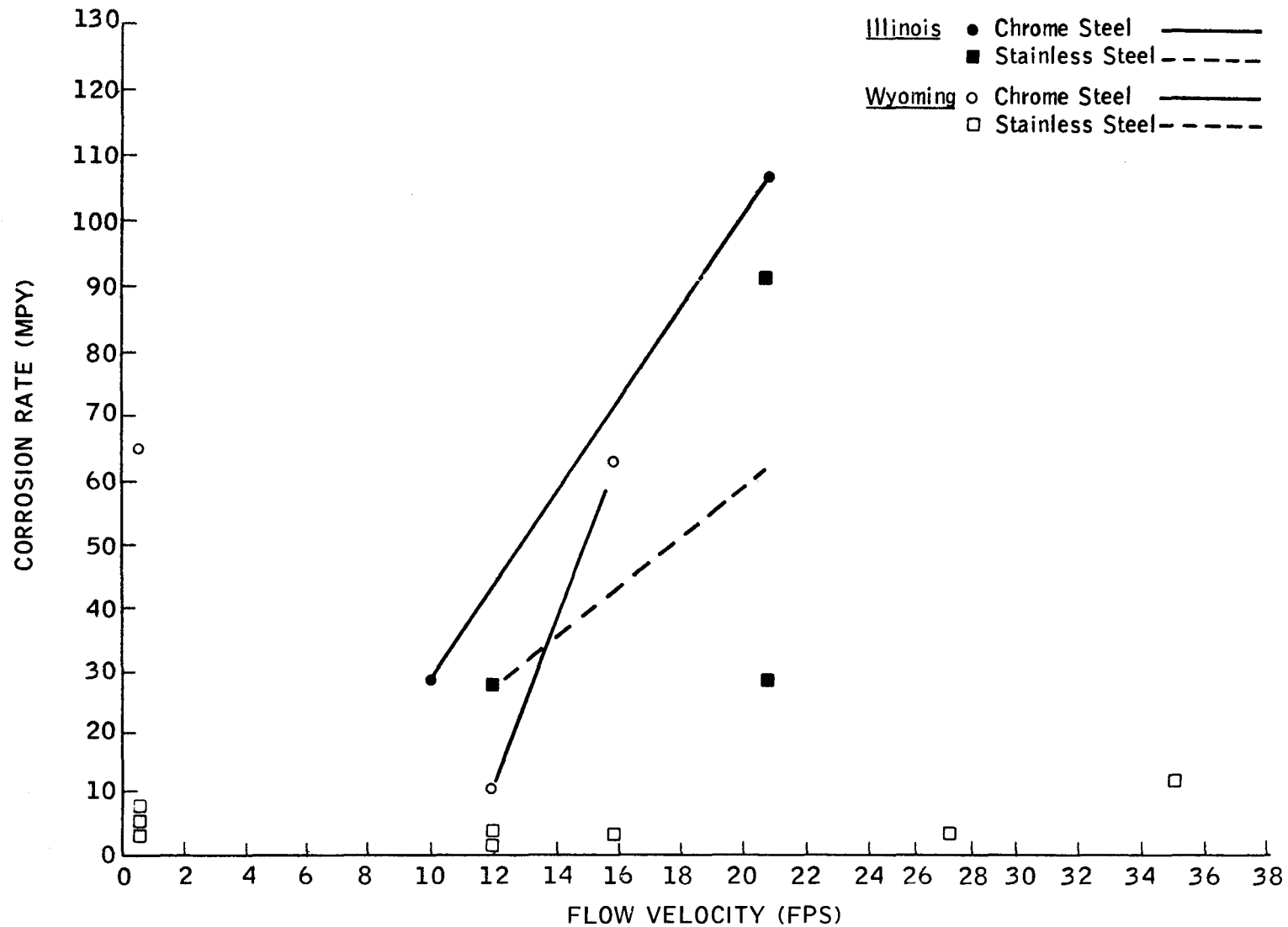




Figure 6-3  
EFFECT OF FLOW VELOCITY ON COAL LIQUIDS CORROSION



### 1.3 Component Failure Analysis

During the reporting period, one failure analysis was performed. It involved corrosion caused perforation of a carbon steel tubing specimen which was installed in the safety line off the vacuum tower pumparound system. The failure is attributed to corrosion by aqueous condensate which formed in this dead-ended location.

Failure occurred after 37 days' service in form of locally holing through the 0.035 inch wall. Temperature in the jacketed main vacuum gas oil loop (Test Site 8) was about 550°F; however, stagnant fluid trapped in the failed test section (unjacketed) was considerably cooler. Schedule pressures for the CLPP run did not permit installation of the test section in the normal TS-8 location.

Severity of corrosion of the failed tubing was considerably greater than registered on earlier TS-8 specimens. During Test Series 2 (January-April, 1977), carbon steel corroded at about 50 mils/year. By comparison, the calculated rate of penetration for the test tubing under discussion is close to 1000 mils/year. Such high rates of attack are occasionally experienced at dead zones where corrosive condensates can collect, both in pilot and commercial plants.

### 2.0 ECLP Materials Evaluation Program

An extensive materials evaluation program has been planned for ECLP to collect in-situ corrosion and erosion data, and to evaluate materials performance on working equipment components. The overall program presently consists of seven major parts, described in Table 6-6. Originally, the Materials Evaluation Program incorporated into the ECLP Design Specification consisted of only the first three parts. These have now been updated and modified. The four areas where considerable progress has been made during the reporting period and covered in this report are as follows:

- Corrosion racks
- Corrosion probes
- Miscellaneous equipment components
- Cooperative programs with National Laboratories

The other programs have been generally defined and are in the early stages of implementation. These activities will be covered in future progress reports.

TABLE 6-6

ECLP MATERIALS EVALUATION PROGRAM

Title	Purpose	Test Sites
Corrosion Racks	Obtain corrosion rates and investigate stress corrosion cracking	36
Corrosion Probes	Record corrosion rates via automated probe system	19
Slurry Letdown Valves	Evaluate developmental trim materials in actual service	2
Slurry Pumps	Evaluate optimum materials on working pump internals	8
Miscellaneous Equipment Components	Evaluate materials performance through full sized working pieces	11
Equipment Inspection	Measure metal loss of plant equipment by NDT techniques	Numerous
Stream Sampling	Identify and quantify corrosive stream constituents	29

## 2.1 Corrosion Racks

Table 6-7 is an updated listing of rack location, service environment, and test materials for all of the 36 corrosion test racks to be installed. During the reporting period, the following was accomplished:

- 1830 coupons were prepared per ASTM G-1, consisting of degreasing, grit blasting, rinsing and drying, followed by stamping with identification numbers. The coupons were subsequently measured and weighed, and assembled onto 72 racks.
- Eccentric reducers were added to contain all racks installed in small piping ( $\leq 4$  inch diameter). The reducers allow more coupons to be tested, reduce pressure drop, minimize the flow area reduction, and decrease risk of coal settling.
- 316 stainless steel was added in duplicate to each rack to serve as a control material.

## 2.2 Corrosion Monitoring (Corrosion Probes)

The prime function of the corrosion probe program is to reveal through its quick response characteristic large, unexpected fluctuations in corrosion rates. The system consists of three parts: probes/cables, data gathering equipment, and computer interface. Individual probe locations, type, and environment for all 19 probes are tabulated in Table 6-8.

Progress during the reporting period consisted of defining test installation details for the contractor, and setting software and hardware requirements for computer interface equipment.

## 2.3 Miscellaneous Component Materials Test

The purpose of these component tests is to collect materials performance data that cannot be reliably derived from racks and coupons. These supplemental tests employ a variety of working components such as piping spools, fractionation trays, and heat exchanger and furnace tubes. The latter are of particular importance since corrosion heat transfer surfaces cannot be reliably predicted from probes or coupons.

An updated summary of miscellaneous component materials tests is presented in Table 6-9. Two additional tests have been added to this updated listing. These tests evaluate fractionating trays of different materials, installed in the Atmospheric Fractionator and the Vacuum Stripper Tower.

TABLE 6-7

## ECLP CORROSION TEST RACK SUMMARY CHART

	Identification Number	Equipment & Location [1]	Service Environment	Test Rack Coupon Materials [2]
Slurry Drying & Liquefaction Section	101	F-102 outlet line (6)	Hydrocarbon	Alonized 5 Cr, 9 Cr, 12 Cr, 304, 316, 321, Alonized 321, I-800
	102	F-101 outlet line upstream of slurry mix point	Hydrocarbon	DELETED
	104	R-101D outlet upstream of D-103 (6)	Hydrocarbon	12 Cr, 304, 316, 321, Alonized 321, I-800
	105	D-103 below liquid level	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316, 321, I-800
	106	D-104 below liquid level	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316, 321, I-800
	107	E-108 shell side inlet (6)	Aqueous	CS, 12 Cr, 304, 316, I-800, I-825, A-20, 3RE60, HC, HG, Ti [3]
	108	D-105 water outlet line (6)	Aqueous	CS, 12 Cr, 304, 316, I-600, I-825, A-20, 3RE60, HC, HG, Ti [3]
	111	D-105 in water phase (2)	Aqueous	CS, 304, 316L, A-20, 3RE60, HB, HC, [3]
Product Distillation Section	201	T-201 feed line near tower (6)	Hydrocarbon	CS, 5 Cr, 9 Cr, 304, 316
	203	T-201 top pan	Hydrocarbon	CS, 316, I-825, A-20, Monel, 3RE60, Ti
	204	T-201 HGO drawoff tray	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316, I-825, HC
	205	T-201 bottom below liquid level	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316, I-825, HG
	208	D-201 water boot	Aqueous	CS, 316, I-825, A-20, 3RE60, Monel, Ti [3]
	209	T-204 feed line near furnace (4)	Hydrocarbon	CS, 5 Cr, Alonized 5 Cr, 9 Cr, 304, Alonized 304, 316
	210	T-204 feed line near tower (4)	Hydrocarbon	CS, 5 Cr, Alonized 5 Cr, 9 Cr, 304, Alonized 304, 316
	211	T-204 HVG0 drawoff tray	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316
	212	T-204 bottom "Y"	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316
	215	T-201 overhead line, near E-203 (6)	Aqueous	CS, 316, I-825, A-20, 3RE60, Monel, Ti
	216	D-206 below liquid level	Hydrocarbon	CS, 316, I-825, A-20, 3RE60, Monel, Ti [3]
	222	T-204 top above pall rings	Aqueous	CS, 316, I-825, A-20, 3RE60, Monel, Ti
Solvent Hydrogenation Section	301	E-301 shell outlet, upstream of gas mix point (6)	Hydrocarbon	CS, Alonized CS, 2 1/4 Cr, 5 Cr, 12 Cr, 304, 321, I-825, 316
	302	F-301 feed line, downstream of gas mix point (6)	Hydrocarbon	CS, Alonized 1 1/4 Cr, 2 1/4 Cr, 5 Cr, 9 Cr, 12 Cr, 304, 321, I-800, I-825, 316
	303	F-301 transfer line near F-301 (6)	Hydrocarbon	2 1/4 Cr, Alonized 2 1/4 Cr, 5 Cr, 9 Cr, 12 Cr, 304, 321, I-800, 316
	304	R-304 effluent line near E-301 (6)	Hydrocarbon	2 1/4 Cr, Alonized 2 1/4 Cr, 5 Cr, 9 Cr, 12 Cr, 304, 321, I-800, 316
	305	D-302 vapor line downstream of venturi (6)	Aqueous	CS, 2 1/4 Cr, 12 Cr, 304, 316, 321, I-825, A-20, Ti
	306	D-303 inlet line (6)	Aqueous	CS, 12 Cr, 304, 316, 321, I-600, I-825, A-20, Ti [3]
	307	D-303 water outlet line (6)	Aqueous	CS, 12 Cr, 304, 316, 321, I-600, I-825, A-20, Ti [3]
	308	T-301 water outlet line at T-301 (6)	Aqueous	CS, 12 Cr, 304, 316, 321, I-600, I-825, A-20, Ti [3]
	309	D-306 inlet line near E-304 (6)	Aqueous	CS, 12 Cr, 304, 316, 321, I-600, I-825, A-20, Ti [3]
	310	F-302 transfer line near F-302 (6)	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316
	311	T-303 bottoms line at T-303 (6)	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316
	312	T-303 at tray 15	Hydrocarbon	CS, 5 Cr, 12 Cr, 304, 316
	313	T-303 overhead line downstream of water injection (6)	Aqueous	CS, 12 Cr, 304, 316, I-600, I-825, A-20, Monel, Ti
	314	D-305 inlet line at D-305 (6)	Aqueous	CS, 12 Cr, 304, 316, I-600, I-825, A-20, Monel, Ti [3]
Fuel Gas Treating & DEA Regeneration Section	604	T-604 bottom section	Aqueous	CS, 304, 316, I-600, I-825 [3]
	606	T-603 bottom section	Aqueous	CS, 304, 316, I-600, I-825 [3]
Sour Water Collection Facilities	751	D-751 liquid	Aqueous	CS, 304, 316, I-825, A-20, Ti [3]

NOTES: [1] Parenthesized number indicates pipe size where racks are installed in piping.

[2] Materials abbreviations:

CS	- carbon steel	316	- 316 stainless steel	A-20	- Carpenter 20Cb3
1 1/4 Cr	- 1 1/4 Cr-1/2 Mo steel	316L	- 316L stainless steel	HB	- Hastelloy B
2 1/4 Cr	- 2 1/4 Cr-1/2 Mo steel	321	- 321 stainless steel	HC	- Hastelloy C-276
5 Cr	- 5 Cr-1/2 Mo steel	Monel	- Monel 400	HC	- Hastelloy G
9 Cr	- 9 Cr-1 Mo steel	I-600	- Inconel 600	3RE60	- Sandvik 3RE60
12 Cr	- 12 Cr stainless steel	I-800	- Incoloy 800	Ti	- Titanium
304	- 304 stainless steel	I-825	- Incoloy 825		

[3] Indicated test racks will have stress corrosion cracking specimens (U-bends) attached.

TABLE 6-8  
ECLP CORROSION PROBE SUMMARY

Probe#	Service/Location	Corrosion Probe			Phase /Response	
		Method <sup>(1)</sup>	Type <sup>(2)</sup>	Element		
<u>Slurry Drying &amp; Liquefaction Section</u>						
CP-109	Recycle Gas Cold Separator H <sub>2</sub> O (D-105)	Res	W40	CS	Liquid	Slow
CP-110	Recycle Gas Water Scrubber Bottoms (T-101)	Res	W40	CS	Liquid	Slow
<u>Product Distillation Section</u>						
CP-207	Distillate Condenser Outlet (E-209)	Res	T20	CS/304	Gas/Mist	Fast
CP-217	Reflux Condenser Outlet (E-203)	Pol	Corr	CS/316	Gas/Mist	Fast
CP-218	Reflux Drum Sour H <sub>2</sub> O (D-206)	Res	T20	CS/304	Liquid	Fast
CP-219	Distilate Drum Sour H <sub>2</sub> O (D-201)	Pol	Corr	CS/304	Liquid	Fast
CP-220	Vacuum Ejector Condensate (E-205)	Res	T20	CS	Gas/Mist	Fast
CP-221	Vacuum Condensate Drum Sour H <sub>2</sub> O (D-203)	Res	W40	CS	Liquid	Slow/Med
<u>Solvent Hydrogenation Section</u>						
CP-315	Reflux Drum Sour H <sub>2</sub> O (D-305)	Res	T20	CS	Gas/Mist	Fast
CP-316	Reflux Condenser Outlet (E-303)	Pol	Corr	CS	Liquid	Fast
CP-317	Distillate Condenser Outlet (E-304)	Res	T20	CS	Gas/Mist	Fast
CP-318	Distillate Drum Sour H <sub>2</sub> O (D-306)	Pol	Corr	CS	Liquid	Med/Fast
CP-319	Solvent Recycle Gas Water Scrubber Outlet (T-301)	Res	T20	CS	Liquid	Slow
CP-320	Cold Separator Drum Sour H <sub>2</sub> O (D-303)	Res	T20	CS	Liquid	Slow/Med
<u>Gas Treating &amp; DEA Regeneration Section</u>						
CP-601	Hot Rich DEA to Regenerator Tower (T-601)	Res	W40	CS	Liquid	Slow
CP-602	DEA Regenerator Overhead Condenser Outlet (E-604)	Res	W40	CS	Gas/Mist	Slow
CP-603	Regenerator Tower Bottom (T-601)	Res	W40	CS	Liquid	Slow
CP-605	Fuel Gas Condensate Separator Drum Sour H <sub>2</sub> O (D-603)	Res	W40	CS	Gas/Mist	Slow
CP-606	Regenerator Tower Reboiler Outlet (E-602)	Res	W40	CS	Liquid	Slow

NOTES:

(1) Res - Electrical resistance                      Pol - Polarization resistance

(2) W40, T20 designate Magna Corrosometer element.

Corr designates Magna Corratel

TABLE 6-9

ECLP MISCELLANEOUS COMPONENT MATERIALS TESTS

Location	Test Components
F-201 Vacuum Stripper Feed Furnace	<ul style="list-style-type: none"> <li>• Thirteen 9 Cr spare tubes and return bends</li> <li>• Two 321 spare tubes and return bends</li> <li>• Two un-Alonized 9 Cr tubes and return bends for last tubes in radiant coil</li> </ul>
E-209 Atmospheric Fractionator Overhead Distillate Condenser	<ul style="list-style-type: none"> <li>• Two AL-29-4 (Allegheny Ludlum) test tubes</li> <li>• Two AL-6X (Allegheny Ludlum) test tubes</li> <li>• Two 3RE60 (Sandvik) test tubes</li> </ul>
E-103 Atmospheric Fractionator Bottoms Cooler	<ul style="list-style-type: none"> <li>• Three carbon steel test tubes at hot end</li> </ul>
E-104 S/H Bottoms Cooler	<ul style="list-style-type: none"> <li>• Three carbon steel test tubes at hot end</li> </ul>
E-108 Cold Separator Condenser	<ul style="list-style-type: none"> <li>• Two AL-6X (Allegheny Ludlum) test tubes</li> <li>• Two 29-4 (Allegheny Ludlum) test tubes</li> <li>• Two 3RE60 (Sandvik) test tubes</li> <li>• Two Carpenter 7-Mo (Carpenter) test tubes</li> </ul>
E-302 Hot Separator Vapor Condenser	<ul style="list-style-type: none"> <li>• Two AL-6X (Allegheny Ludlum) test tubes</li> <li>• Two 29-4 (Allegheny Ludlum) test tubes</li> <li>• Two 3RE60 (Sandvik) test tubes</li> <li>• Two Carpenter 7-Mo (Carpenter Technology) test tubes</li> </ul>
T-204 Vacuum Stripper Tower Feed Line at tower inlet	<ul style="list-style-type: none"> <li>• One unlined 5 Cr removal test spool</li> <li>• One carbon steel removable test spool lined with metal fiber reinforced erosion resistant refractory castable</li> </ul>
M-101 Slurry Drier Mixer	<ul style="list-style-type: none"> <li>• Six different blade materials - CS, rubber lined CS, 12 Cr, Stellite 6, Manganese steel, tungsten carbide faced CS</li> </ul>
D-751 Sour Water Collection Drum	<ul style="list-style-type: none"> <li>• Cement test site, 6 types of cement</li> </ul>
T-201 Atmospheric Fractionator Tower	<ul style="list-style-type: none"> <li>• Alternating trays of CS, 410, 316</li> </ul>
T-204 Vacuum Stripper Tower	<ul style="list-style-type: none"> <li>• Alternating wash zone sheds and stripping section baffles of CS, 410, 316</li> </ul>

## 2.4 Cooperative Programs with National Laboratories

Two cooperative programs have been initiated with National Labs. One program addresses the question of stress corrosion cracking at ECLP; the other program involves nondestructive monitoring of slurry erosion and block valve leakage.

The stress corrosion cracking (SCC) program will be conducted cooperatively with Oak Ridge National Laboratory (ORNL). Its objective is to investigate the potential problem of SCC, principally with respect to chloride SCC of stainless steels. Toward this objective, ORNL has agreed to supply U-bend specimens which will be mounted on existing corrosion racks. ORNL will also perform post-exposure metallographic analyses. Table 6-10 summarizes locations, rack ID number, and test materials.

The second cooperative program consists of field testing of novel nondestructive testing (NDT) devices for acoustic monitoring of slurry block valve leakage, and for ultrasonic measurement of slurry erosion in piping and furnace coils. This program is to be run with Argonne National Laboratory (ANL). ANL will supply sensing devices, and furnish technical assistance for installing hardware, commissioning systems, and training ECLP staff. In addition to developing NDT techniques for future commercial plant applications, these monitoring devices will serve a useful function in ECLP operation.

## 3.0 State-of-the-Art Materials Technology

Significant effort has been directed to keeping abreast of and guiding materials engineering developments in coal conversion processes, principally through active participation in technical society/committee/workshop activities. Major activities during this reporting period have been as follows:

- Initiated coal liquids corrosion test program in Metals Properties Council Subcommittee 9.
- Chaired symposium and presented paper at National Association of Corrosion Engineer's CORROSION/79.
- Briefed Engineering Societies Commission on Energy on coal liquids materials technology.
- Attended/participated in various symposia/workshops in coal liquefaction materials.
- Visited SRC pilot plant at Wilsonville, Alabama to discuss corrosion problem.



TABLE 6-10

ECLP STRESS CORROSION CRACKING TESTS

( JOINT ER&amp;E/ORNL PROGRAM )

Area	Equipment Item	Rack ID #	SCC Potential	Test Materials <sup>(1)(2)</sup>
Liquefaction Overhead	D-105	108	High	304, 304 SE, I825, 3RE60
	E-108	107	High	304, 304 SE, I825, 3RE60
Hydrogenation Overhead	D-303	306	Medium	CS, 304, 304SE, I825, 3RE60
	T-301	308	Low	CS, 304, 304 SE, I825, 3RE60
Solvent Fractionator Overhead	D-305	314	Low	CS, 304, 304 SE
	D-306	309	Medium	CS, 304, 304 SE

Notes: (1) SE - sensitized

(2) Material                      U-bends Required

Carbon steel	8
304 stainless steel	24
Incoloy 825	8
Sandvik 3RE60	8

### 3.1 Metal Properties Council (MPC)

Since 1972, MPC has been active in generating, developing and directing broad programs on materials degradation and evaluation in coal gasification environments, under the auspices of Subcommittee 9 (Materials for Coal Gasification and Liquefaction Processes). A new Phase Group (VII) was organized in June 1977 in response to the growing interest and activities in coal liquefaction, presently chaired by ER&E. The three main areas of interest in Phase Group VII are coal liquids corrosion, coal slurry erosion, and in-situ materials testing in coal liquefaction pilot plants. Presently, the only active project is formulating the comprehensive laboratory test program on coal liquids corrosion described below.

An official MPC Project Specification for this program entitled "Materials to Resist Corrosion in Coal Liquefaction Processes" was sent out for competitive bids in April 1978. In November 1978, recommendations were made to award the work to a specific laboratory, and the program was formally submitted to DOE for funding consideration in December 1978. It is anticipated that the contract will be executed in 3Q1979.

The objectives of this comprehensive test program are outlined below. Test parameters are surveyed in Table 6-11. Detailed analytical procedures for identifying corrosive species have not yet been finalized, and therefore will not be described in this report.

Using representative process streams (coal liquids) obtained from coal hydroliquefaction pilot plants, the objectives of the test program are:

A. To conduct a series of autoclave exposure tests to:

1. measure approximate corrosion rates of common steels
2. determine the effect of temperature on corrosion
3. determine the effect of temperature on stability/concentration of corrosive species

B. To analyze these coal liquids to:

1. identify suspected corrosive species
2. measure amounts of these species before and after autoclave testing

C. To provide background for:

1. elucidating form and mechanism of corrosion
2. interpreting results from in-situ corrosion tests and component failures in coal liquefaction facilities

TABLE 6-11

TEST PARAMETERS FOR COAL LIQUIDS CORROSION TEST PROGRAM (MPC PROPOSAL No. 979-P104)	
Test Fluid Donor Process/ Location (Note 1)	CSF/Cresap, WV EDS/Baytown, TX H-Coal/Trenton, NJ SRC I/Wilsonville, AL SRC II/Fort Lewis, WA
Test Fluids (Note 2)	7 streams per plant
Test Temperatures	350-500°F (Light streams) 425-575°F (Medium streams) 500-650°F (Heavy streams)
Test Pressure (Note 3)	> 1000 psi
Test Duration	100 hours
Volume/Area Ratio (Note 4)	20 cc test fluid/cm <sup>2</sup> test specimen
Test Materials (Note 5)	Carbon steel Type 502 (5 Cr-1/2 Mo) alloy steel Type 410 (12 Cr) stainless steel Type 316 (18-8 Mo) stainless steel Hastelloy C-276 (Ni-Cr-Mo alloy)
Specimen Configuration (Note 6)	1" x 1" x 1/8-1/4" weight loss coupons
Specimen Evaluation	Weight loss Dimensional change Pitting characterization Metallography (10% of specimens) SEM and/or X-Ray analysis (5% of specimens)

- NOTES: 1) Donor process/locations are subject to change during course of program.
- 2) Test fluids will not include coal liquids with strong coking/polymerizing tendency.
- 3) Test pressure will be 1000 psi argon plus test fluid vapor pressure.
- 4) Specimens are to be fully immersed in test fluid which is to fill 75% of autoclave volume.
- 5) When several metals are tested simultaneously, CS/5 Cr/12 Cr may be tested in one group, and 316/Hastelloy in the other.
- 6) All specimens are to be tested in duplicate.

### 3.2 National Association of Corrosion Engineers (NACE)

Since its inception in 1976, NACE Technical Practices Committee T-12 (Materials for New Energy Systems) has fulfilled its objective of developing and presenting technical symposia, and providing an open forum for the identification and discussion of materials problems being experienced and anticipated in the construction and operation of coal conversion plants. ER&E participation in coal conversion directed NACE activities during the reporting period were as follows:

- Attend meeting of NACE Committee T-12 held at CORROSION/79 in Atlanta, GA.
- Chaired T-12 Symposium entitled "Materials Developments for the Coal Conversion Industry" held at CORROSION/79.
- Presented ER&E authored paper entitled, "Materials Evaluation Program for EDS Coal Liquefaction Process" by E. Lendvai-Lintner and G. Sorell, to be published in Materials Performance.

### 3.3 Engineering Societies Commission on Energy (ESCOE)

The ESCOE Engineer in Residence visited ER&E in Florham Park, N. J. in February, 1979 to discuss materials technology in coal liquefaction processes, with specific reference to EDS. The immediate purpose of this briefing was to furnish background for a forthcoming report on "Materials of Construction for Coal Conversion Systems - Coal Liquefaction", being prepared by ESCOE under DOE Contract No. EF-77-C-01-2468. Particular emphasis was placed on materials selection and testing in Exxon's 250 T/D EDS pilot plant in Baytown, TX (ECLP). A brief summary of materials of construction utilized for ECLP equipment is presented in Table 6-12.

### 3.4 Other Meetings

ER&E participated in several meetings/symposia/workshops devoted to coal conversion materials technology, as itemized below:

- Materials for Coal Conversion and Utilization - Third Annual Conference, Gaithersburgh, MD (October 1978).

Presented short briefing on integrated materials testing programs for EDS, and served as panelist in technical sessions on Materials for Coal Conversion Applications (Low Temperature).

- International Materials Congress - Materials Aspects of World Energy Needs, Reston, VA (March 1979).

TABLE 6-12  
MATERIALS SPECIFIED FOR EQUIPMENT  
250 T/D EDS PILOT PLANT

Area/Equipment	Environment	Temperature (°F)	Pressure (psi)	Equipment Materials
<u>Slurry Drying and Liquefaction Section</u>				
Slurry Drier Circuit	Hydrocarbon + solids	100-300	2000	Carbon steel
Slurry Preheat	Hydrocarbon + solids	200-850	2000	300 series stainless steel
Liquefaction Reactor	Hydrocarbon + solids	800-900	2000	2 1/4 Cr with 300 SS overlay
Reactor Separator	Hydrocarbon + solids	800-900	2000	1 1/4 Cr with 300 SS overlay
Recycle Gas Hot Separator	Hydrocarbon/aqueous fluids	300-650	2000	1 1/4 Cr with 300 SS overlay
Recycle Gas Cold Separator	Aqueous/hydrocarbon fluids	100-150	2000	Carbon steel, gunite lined
<u>Product Distillation Section</u>				
Atmospheric Fractionator	Hydrocarbon + solids	300-650	50	Carbon steel, bottom 12 Cr clad
Atmospheric Fractionator Overhead Circuit	Hydrocarbon/aqueous fluids	100-300	50	Carbon steel
Vacuum Stripper Preheat	Hydrocarbon + solids	500-750	50	5 Cr
Vacuum Stripper	Hydrocarbon + solids	300-650	5	Carbon steel, 12 Cr clad
Vacuum Stripper Overhead Circuit	Hydrocarbon/aqueous fluids	100-300	50	Carbon steel

Served as Rapporteur in workshop on "Materials Science and Technology for New Energy Sources and More Efficient Energy Conversion - Fossil Fuels".

- American Physical Society, Chicago, IL (March 1979)

Presented invited paper entitled, "Materials Technology in Coal Liquefaction - Status and Challenges", by G. Sorell at symposium on Physics and Coal Utilization.

- MPC Task Group on Extra Large Pressure Vessels for Coal Conversion Processes, New York, NY (December 1978) and Pittsburgh, PA (February 1979)

ER&E attended organizational meeting and will chair subgroup on Environmental Compatibility.

- DOE Task Force on Advanced Research and Technology Development, Washington, D.C. (April 1979)
- DOE/NACE/LBL Workshop on Corrosion/Erosion of Coal Conversion System Materials, Lawrence Berkeley Laboratory (January 1979).
- DOE/ANL Conference on Coatings for Materials Protection in Energy Systems", Argonne National Laboratory (June 1979).

#### 4.0 IKG Corrosion Tests

Although relatively little materials related work was done at the IKG unit during the reporting period, this report presents a summary of all such work to date. The rationale for this overview is based on the discontinuation of IKG operation for an indefinite period.

Materials testing in the IKG unit consisted of three tasks:

- Sour water corrosion racks
- Heater overhead condenser test tubing
- Gasifier grid specimens

To date, two sets of heater overhead tubing and one set of gasifier specimens have been exposed. In addition, results from two corrosion rack exposure periods have been obtained.

#### 4.1 Sour Water Corrosion Racks

Results from the corrosion rack exposures are tabulated in Table 6-13, together with waste water compositions. With the change from Illinois to Wyodak coal, carbon steel corrosion rates increased substantially while stainless steel corrosion rates decreased somewhat. This behavior is indicative of a possible change in corrosion mechanism. Based on the moderate corrosion rate of carbon steel coupons (8-21 mpy) ordinary steel construction would appear to be adequate.

The two sour water analyses give no clue to the higher corrosivity of Wyodak derived condensate. In fact, based on its greater S, Cl, CN and phenol content, Illinois coal derived condensate would be predicted to be the more aggressive. It is therefore concluded that these two single analyses are not representative for the entire exposure periods of the corrosion test coupons.

TABLE 6-13  
IKG UNIT CORROSION RACK DATA

EXPOSURE 1 6/9/77-12/8/77 Illinois Coal		EXPOSURE 2 2/9/78-6/21/78 Wyodak Coal		EXPOSURE 3 (Future)
<u>Rack 1 (Bottom of D-4)</u>				
Carbon Steel	3.6	Carbon Steel	15.7	Carbon Steel
304 SS	0.56	Carbon Steel	11.3	Carbon Steel
316L SS	0.73	304 SS	0.29	304 SS
Alloy 825	0.63	Welded 304 SS	0.20	Monel
Titanium	0.78	316 SS	0.26	Titanium
				Inconel 600
<u>Rack 2 (Top of D-4)</u>				
Carbon Steel	8.0	Carbon Steel	21.2	Carbon Steel
304 SS	0.48	Carbon Steel	20.6	Carbon Steel
316L SS	0.48	304 SS	0.13	304 SS
Alloy 825	0.41	Welded 304 SS	0.18	Monel
Titanium	0.51	316 SS	0.19	Titanium
				Inconel 600
567 hour exposure		1040 hour exposure		
<u>Wastewater Composition</u>				
Phenol	43 ppm	Phenol	1 ppm	
S	2.5 ppm	S	< 1 ppm	
Cl	118 ppm	Cl	20 ppm	
NH <sub>3</sub>	3700 ppm	NH <sub>3</sub>	12,600 ppm	
total CN	4.8 ppm	total CN <sup>-</sup>	undetermined	
pH	8.3	pH	8.6	

- Notes: (1) Condensate in D-4 was drained every 24 hours and replaced with fresh water.
- (2) Unit was N<sub>2</sub> purged when not operating.
- (3) Corrosion rates are in mils per year.



#### 4.2 Condenser Test Tubing

Condenser test tubing installed in the Coke-Gas Condenser (Heater Overhead system) during the Wyodak run showed appreciable corrosion. The carbon steel tube registered corrosion rates of 29 mils/year at the cold end, and 68 mils/year in the middle and hot end, plus some mild pitting throughout. This rate of attack is considerably higher than measured on D-4 coupons situated directly downstream.

The condenser tube experiments to date do not permit direct comparison between Illinois and Wyodak generated sour waters because the only tube installed during Illinois operation was 316L SS. It did not show any evidence of general corrosion or pitting.

#### 4.3 Gasifier Test Specimens

The ring test specimens installed on the gasifier grid indicated practically no corrosion. Three 310 SS specimens gave rates of 1.4, 1.7 and 2.1 mils/year and showed evidence of pitting. Two Incoloy 800 specimens registered a mere 0.2 mils/year and were devoid of pitting.

#### 4.4 Future Plans

The following test specimens have been sent to the pilot plant for the eventuality that IKG is recommissioned for EDS bottoms processing.

- Two D-4 corrosion racks (coupons listed in Table 6-13).
- 304 SS condenser test tube
- 304 SS, 310 SS, Incoloy 800 gasifier specimens

#### 5.0 EDS FLEXICOKING Inspection

An inspection was carried out on the decommissioned FLEXICOKING prototype located in Exxon's Baytown Refinery, to assess the condition of the unit after mothballing, and to determine the scope of necessary repairs to be made for reactivation of the unit for EDS operation. No major repair items were discovered which had not already been identified when the prototype was shut down in 1975. However, considerable external corrosion had occurred on insulated surfaces, notably small diameter piping. Inspection comments and repair/modification items will be incorporated into the EDS FLEXICOKING Design Specification.

## 6.0 High Temperature Erosion Testing

The first series of a cooperative LBL/ER&E R&D program to evaluate EDS FLEXICOKING erosion has been completed. These tests were conducted in Lawrence Berkeley Laboratory's erosion test facilities.

Over 80 tests were run in a 30-week period on the elevated temperature gas-solids erosion test apparatus shown in Figure 6-4. The main objective was to evaluate the reproducibility of data generated. This was accomplished by exposing specimens of 310 SS and an erosion resistant refractory at room and elevated temperatures at the conditions listed in Table 6-14. A secondary objective was to obtain a limited amount of data on the effect of temperature on erosion. The results of these tests are currently being analyzed. A second series of tests is planned for 3rd Quarter 1979.

### Future Work

The following summary of future plans for materials engineering R&D is limited to the next 6 month period, commencing July, 1979.

#### 1. CLPP Materials Evaluation Program

- Evaluate Series VI (Illinois bottoms recycle mode) and VII (first sponsor's coal specimens).
- Supply set of coupons for evaluation of second sponsor's coal.

#### 2. ECLP Materials Evaluation Program

- Update Materials Evaluation Program prior to ECLP startup.
- Assist in commissioning of corrosion monitoring system.
- Install ORNL U-bends on corrosion racks and ship to ECLP.
- Implement joint ANL/ER&E program to install and evaluate NDT devices for ultrasonic erosion monitoring.

#### 3. State-of-the-Art Materials Technology

- Initiate MPC coal liquids corrosion program pending DOE contract.

#### 4. EDS FLEXICOKING

- Prepare full materials evaluation program similar to ECLP program.

#### 5. High Temperature Erosion Testing

- Evaluate results of Phase 1 tests at LBL.
- Conduct Phase 2 tests.

Figure 6-4

LBL ELEVATED TEMPERATURE EROSION APPARATUS

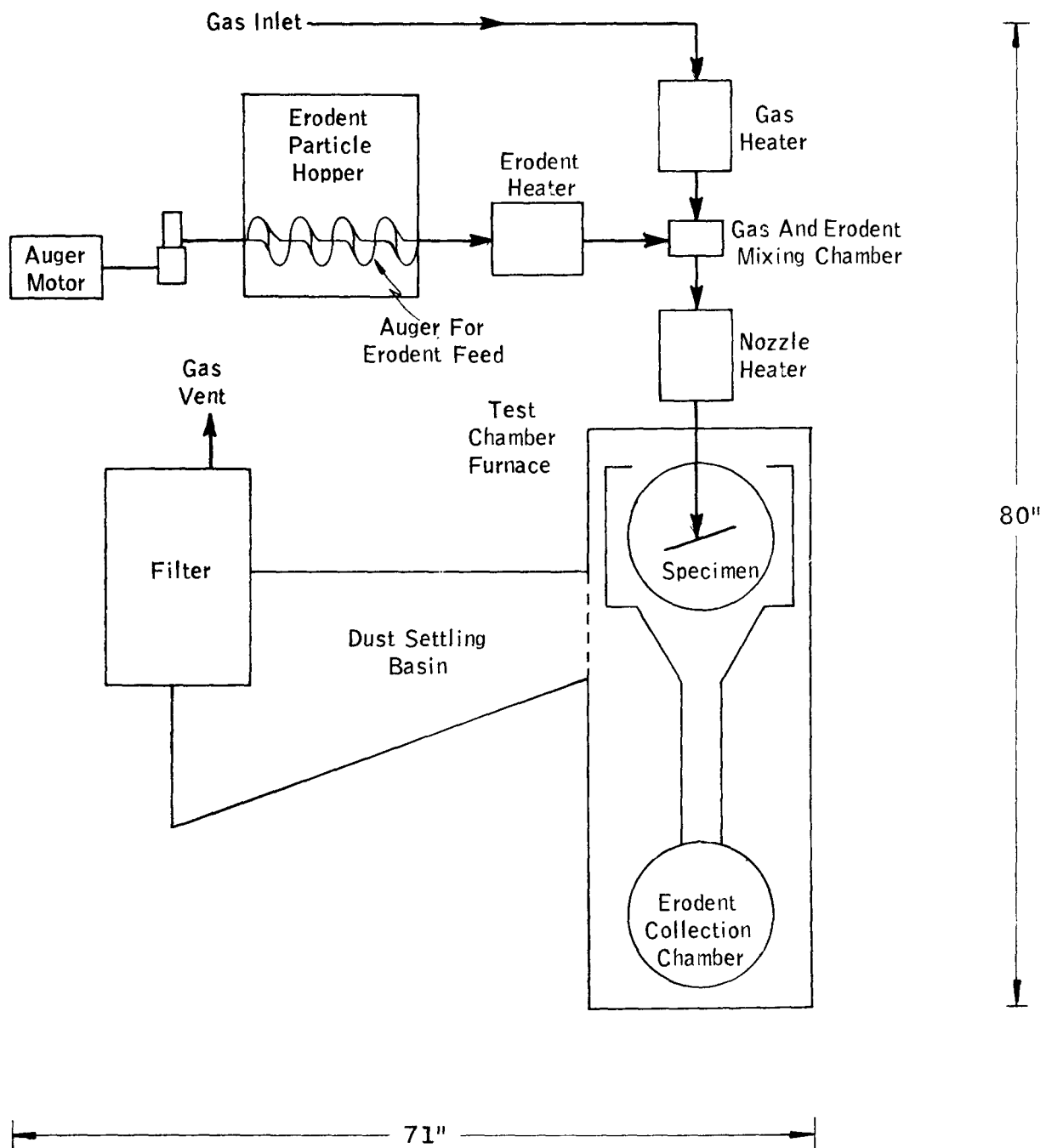


TABLE 6-14

CONDITIONS FOR LBL/ER&E EROSION TESTS  
(PHASE I)

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	Series #1	Series #2	Series #3	Series #4
Material	310 SS	310 SS	Refractory <sup>(1)</sup>	Refractory <sup>(1)</sup>
Temperature	Room	1350°F	Room	1500°F
Impingement Angle	20°	20°	90°	90°
Test Duration	20 minutes	20 minutes	2.5 minutes	2.5 minutes
Erodent <sup>(2)</sup>	SiC	SiC	SiC	SiC
Erodent Velocity	100 ft/sec	100 ft/sec	100 ft/sec	100 ft/sec

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NOTES: 1) Resco RS-17E

2) SiC grit 250-300  $\mu\text{m}$

## 6.5 Mechanical Engineering Technology

### Summary

The mechanical engineering accomplishments for the year consisted of the development of a detailed inspection and testing plan for slurry service valves and high pressure flanges at ECLP, the initiation of a valve test program at CLPP, and the development of preliminary purchase specifications for low cost slurry block valves for testing at ECLP. A detailed description of the inspection and testing plan developed for slurry service valves and critical flanges at ECLP is contained in the January-March, 1979 Quarterly Technical Progress Report (FE-2893-29) and therefore, only a summary of each test program is given below. In addition, work was started on surveying the high pressure heat exchanger industry to determine if commercial scale coal liquefaction exchangers are feasible with present technology.

### Mechanical Engineering ECLP Test Program

A detailed inspection and testing plan for slurry service block and check valves and critical high pressure flanges in ECLP was developed during this report period. The purpose of this program is to provide operational and mechanical performance data on these components in ECLP in order to allow for reliable scale up of the equipment to commercial plant size. A listing of the tests planned and the objective of each follows:

#### I. Block Valves for Slurry Service

##### A. Slurry Block Valve Leakage Tests

Objective: To determine the long-term reliability of each of the block valve types specified for slurry service.

##### B. Slurry Block Valve Flushing Requirements

Objective: To determine the extent and frequency of flushing required for each valve type.

##### C. Double Block Valve Requirements for Slurry Service

Objective: To determine if double block valves are necessary in slurry service with a design temperature greater than 400°F but less than 1000°F and a primary class rating of less than or equal to 600.

- D. Low Cost Slurry Block Valve Test (contingent on funds being available to procure and install required test valves)

Objective: To determine the reliability of the lower cost block valve design, i.e., wedge type gate valve, soft sealed ball valve, metal seated floating ball valve, and metal seated butterfly valve.

## II. Check Valves for Slurry Service

- A. Slurry Check Valve Reliability Test/Inspection

Objective: To determine the long-term reliability of each of the check valve types specified for slurry service.

## III. High Pressure Flange Joint Reliability Test

- A. Flange Joint Leak Tightness Test

Objective: To determine the relative reliability of the various high pressure flange joint configurations used in ECLP. The flange joints of particular interest are the insulated ring joint and Grayloc flanges in the liquefaction section, the uninsulated ring joint and the tongue and groove flanges on the solvent hydrogenation reactors. In addition, the merits of weather shielding (banding) the flanges in high pressure and high temperature service will be evaluated.

## Low Cost Slurry Block Valves

In conjunction with the low cost slurry block valve test program proposed for ECLP, preliminary purchase specifications have been developed for these valves. Purchase specifications were developed for a wedge type gate valve, metal seated floating ball valve and metal seated butterfly valve. These specifications are summarized below:

- Wedge Type Gate Valve - (Design pressure 200 psig, Design temperature 350°F), Class 150 carbon steel gate valve designed, fabricated, inspected and tested per API 600 and BP 3-12-2 Steel Gate Valves. Valves to have flexible wedge gate, API Trim No. 5 and valve body and bonnet purge connections (3/4 inch).
- Metal Seated Floating Ball Valve - (Design pressure 200 psig, Design temperature 350°F) Class 150 carbon steel full port metal seated ball valves, such as the following or equivalent:

- LC4 coated ball/seats or Stellite No. 6 coated ball/seats with hardness requirement per API 600.
- Ball valve with tunnel bore ball and Stellite No. 6 coated ball/seats with hardness requirements per API 600.
- Soft Sealed Floating Ball Valve - (Design pressure 200 psig, Design temperature 350°F) Class 150, carbon steel full port soft sealed ball valves per BP 3-14-1-Soft-Sealed Ball Valves, Fire Safe, Type, such as the following or equivalent:
  - Ball valve with reinforced TFE seats type HPT-2, seal code 18 (needs to be fire tested).
  - Ball valve with reinforced P.T.F.E. seal material.
- Metal Seated Butterfly Valve - (Design Pressure 50 psig, Design temperature 825°F) Class 150, 5 Cr-1/2 Mo equivalent full port design metal seated butterfly valve such as the following or equivalent:
  - Butterfly valve with a laminated metal/asbestos seal and Stellite No. 6 coated seat.

#### CLPP Valve Test Program

The valve test program at CLPP consists of the installation of two 1/2 inch trunnion mounted ball valves on the outlets of the solids accumulator drums (D-9A & D-9B). One valve is a standard ball valve design in which springs and system pressure are utilized to provide the seating force between the ball and seat rings. The other valve is a modified design such that the spring cavity will be sealed from the process stream to prevent solids and/or coke buildup which could possibly lead to a reduction in spring seating force. A principle objective of the test program is to provide data on the merits of a sealed spring cavity in slurry service.

#### High Pressure Heat Exchanger Manufacturers Survey

A program was initiated to determine if currently available design/fabrication technology and experience is adequate to provide safe reliable high pressure heat exchangers for coal liquefaction processes. In order to accomplish this, a survey form asking specific questions related to the design, fabrication, and inspection/testing of heat exchangers was developed. In addition, the survey also asks for relevant service experience with specific design details recommended by the manufacturers.

Present plans call for the sending of the survey form to about 10 leading high pressure heat exchanger manufacturers worldwide.

Also as a part of this program, a meeting was held with a leading heat exchanger manufacturer to discuss the current state of the art in high pressure heat exchanger design/fabrication. In the future, meetings with other manufacturers will be scheduled as the opportunities arise.



## 6.6 Slurry Pumping

### ECLP Equipment Procurement

Orders were placed for the two centrifugal test pumps in the ECLP Project, as follows. Both have replaceable lining elements of hard 28% chrome iron inside API type steel casings. Both are prototype designs.

<u>Tag No.</u>	<u>P-101E</u>	<u>P-216</u>
Service	Coal slurry feed booster	Atmospheric fractionator bottoms
Design temp	350°F	800°F
Mechanical shaft seal	Double	Double
Nominal speed	3560 rpm	1780 rpm

Technical proposals were received from two vendors for the application of centrifugal pumps with replaceable linings as possible replacements for the screw type pump being applied in the vacuum bottoms service, P-210. Such a substitution would not be required for the operability of the plant, but may provide an advantageous opportunity to test and demonstrate centrifugal pumps in this challenging service.

The ECLP plant will include a total of 145 pumps.

During the year the shop testing of one of the replaceable-lining type centrifugal pumps was witnessed. The shop testing of two of the plunger pumps was also witnessed.

### Hydraulic Motors for Slurry Let-Down Service

In the second quarter of 1978, inquiries were made into the use of a screw pump running backwards as a substitute for a pressure let-down valve. None of the three screw pump vendors contacted considered the idea promising and the investigation was dropped.

### Test Proposal Development for ECLP Slurry Pumps

Proposals for the testing of 12 pumps in slurry services were developed at two levels - general plans for inclusion in the overall ECLP Test Plan, and specific testing details with the five vendors of the pumps.

The general plans included estimates of manpower and parts requirements to carry out the program. The specific plans include time estimates and speed levels for each phase of the testing. Vendor advice and requests have been included in the detailed planning.

The pumps included in the slurry pump test program are summarized below:

<u>SERVICE NO.</u>	<u>SERVICE PUMPING TEMPERATURE</u>	<u>PUMP TYPE RATED SPEED</u>
P-101ABCD	Coal slurry feed booster 300°F	Centrifugal, replaceable lining 1100 rpm
P-101E	Coal slurry feed booster 300°F	Centrifugal, replaceable lining 3560 rpm
P-102AB	Coal slurry feed 300°F	Reciprocating plunger 109 rpm
P-204AB	Atmospheric fraction- ator bottoms 643°F	Reciprocating plunger 43 rpm
P-210AB	Vacuum fractionator bottoms 775°F	Screw, two-rotor 350 rpm
P-216	Atmospheric fraction- ator bottoms prototype test pump 643°F	Centrifugal, replaceable lining 1780 rpm

### Test Plans for Other ECLP Machinery

In addition to the development of test plan proposals for the 12 pumps in primary slurry service, test proposals were also developed for the following miscellaneous machinery:

- Mechanical seal serviceability and design improvement development (for all centrifugal and rotary pumps)
- Experience monitoring for other severe service pumps
- Fouling potential of the slurry drier compressor, C-102

### FLEXICOKING Prototype Feed Pump Service Designs Development

The design of the feed pumping service for feeding ECLP vacuum fractionation bottoms to the FLEXICOKING Prototype unit was developed further. The service design consists of a pair of screw pumps as boosters and a pair of plunger pumps on primary feed pumps, developing the required 600 psig.

Preliminary proposals for testing the slurry pumps in the EDS FLEXICOKING Prototype Revamp Project were developed and submitted for review.

## 6.7 Instrumentation

### Summary

Program objectives for the reporting period included keeping abreast of new developments and testing of high pressure letdown valves for coal slurries, selecting and specifying the best available materials for internal coating of venturi meters in coal slurry service, and following experience with viscometers as applied to measurement of slurry concentration. All of our objectives have been met.

### High Pressure Letdown Valves

Discussions with commercial valve manufacturers have not produced any improved designs over the valves selected for ECLP. We have been following planned testing of the EPRI sponsored valve (consolidated controls) at SRC, Wilsonville, Alabama but no test results are available. We plan to review the test data when available.

### Venturi Meters

The result of our efforts in the area of venturi flow element internal coatings has been that flow element vendors indicate that the meters used for ECLP are too small for practical application of the internal hard coatings recommended. Our recommendation is to use flame spray tungsten carbide applied to the inside of the venturi surface. This is not viewed as a significant shortcoming since meters will be used for monitoring only and degradation of metering accuracy will not reach unacceptable levels within the life of the pilot plant. Coatings can easily be applied to venturi meters in the commercial plant because the sizes are large enough for spray application of the coating.

### Viscometers for Slurry Concentration

A nuclear density gauge has been purchased for use on ECLP for this application.

Another aspect of work completed during the reporting period was documentation of ECLP Test Program Planning, cost estimating, ECLP cost projection for both engineering and test implementation and how ECLP plant tests will be applied to the future commercial design.

#### 6.8 Civil Engineering Aspects of EDS Waste Disposal

During the reporting period the initial preliminary work on the program established that outside Civil Engineering Consultants having significant experience in coal waste disposal could be advantageously used to carry out the detailed work. The extent of the proposed consultant's, participation in the preparation of the design guides for EDS waste disposal facilities was explored in a series of meetings with the consultant.

During the last quarter the consultant's proposal was reviewed and found to comply with ER&E requirements for a design guide covering site selection criteria for the facility, and engineering considerations for the safe design of secure landfills and impoundments.

ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,  
PROCUREMENT AND CONSTRUCTION

1. Project Management

Statistical highlights regarding the status of ECLP engineering and construction activities at the close of the first half of 1979 are summarized below. Each topical area is covered in more detail in later sections of this report.

		<u>% Complete</u>	
		<u>Original</u>	<u>Actual</u>
		<u>Plan</u>	<u>29 June</u>
+ <u>Progress</u>	Engineering and Procurement	97	96
	Material Commitments	99	93
	Construction	86	62
		<u>M\$</u>	
		<u>Total</u>	<u>Process Dev.</u>
		<u>Project</u>	<u>Allowance</u>
+ <u>Cost</u>	Outlook, Sept. '77	110.0	2.0
	Class II Estimate, Nov. '77	108.5	5.0
	Adjusted Control Budget, June '79	111.8	0
	Outlook, June '79	112.2	0
		<u>Mechanical</u>	
		<u>Completion</u>	
+ <u>Schedule</u>	Planned	15 November, 1979	
	Projected, Most Probable	15 February, 1980 <u>+ 2 wks</u>	

Representatives of the DOE Inspector General's staff visited both the construction site and the engineering office in August, 1978 as part of their inspection of the overall Exxon Donor Solvent program. Discussions were held with selected members of the Exxon Engineering, Daniel and McKee project and construction management staffs.

The following additions/changes to the ER&E Project and Construction Management staff groups occurred during the year.

- + New Inspection Coordinator assigned on 5 July, 1978
- + Field Engineer assigned on 18 July, 1978
- + Lead Field Inspector assigned on 4 August, 1978
- + Field Inspector assigned on 28 August, 1978
- + Field Engineer assigned on 5 September, 1978
- + Project Engineer for instrumentation, was reassigned as the Field Engineer for instrument installation on 15 December, 1978.
- + Project Purchasing Advisor, completed his assignment in mid-December and returned to the Exxon, USA Supply Department.
- + Project Engineer for piping and vessels, was reassigned as a Field Engineer on 22 January, 1979.

- + Cost/Schedule Control Engineer, relocated to the construction site on 29 January, 1979.
- + Project Manager, relocated to the construction site on 31 January, 1979

There were no significant changes to McKee's Engineering and Procurement Project Management staff during the year. Some changes to the Daniel Construction Management staff took place during the year.

Project Management tasks are described in more detail in the writeups on Engineering and Procurement, and Construction.

ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,  
PROCUREMENT AND CONSTRUCTION

2. Engineering and Procurement

Basic Engineering

Basic engineering work during the year consisted primarily of the development of design bases for the many changes in scope required as the detailed engineering progressed and operating/model reviews were completed, and the preparation of Change Lists to formally document all modifications to the original design specifications. Among the major changes handled by the Exxon engineering design follow-up engineers with assistance from the McKee engineering staff were:

- + Electrical heating systems modifications/definitions.
- + Power distribution systems changes for increased load requirements.
- + Solvent flushing and blowdown systems definition.
- + Dowtherm heating system operating range change.
- + Definition of modifications required by interim turnovers.

Change Lists issued during the year to document the above major changes as well as all other changes were as follows.

<u>Title</u>	<u>C.L. No.</u>	<u>Issue Date</u>
Slurry Drying & Liquefaction	6	April 2, 1979
Product Distillation	6	April 20, 1979
Solvent Hydrogenation	6	Jan. 29, 1979
Fuel Gas Treating and DEA Regeneration	4	Jan. 29, 1979
Hydrogen Compression Facilities	3	Jan. 29, 1979
Sour Water Collection	4	Jan. 29, 1979
Layout and Buildings	2	Aug. 9, 1978
Coal Preparation Facilities	2	Aug. 30, 1978
Utilities	2	Aug. 18, 1978
Waste Treating Facilities	2	Aug. 25, 1978
Fire Protection	2	Jan. 17, 1978
Safety Facilities	2	Aug. 9, 1978
Vacuum Bottoms Solids Handling	2	July 28, 1978

Engineering

McKee's engineering and procurement work was 96% complete at the end of June, 1979, approximately 1% or 4 weeks behind the original plan (see Figure 2-1 for Progress Charts). Design work for modifications to the Exxon Refinery VGO/DAU blending facilities and the Exxon Chemical Plant Paraxylene Absorption Unit (PAU) furnaces to accommodate certain ECLP product streams was essentially complete.

The total number of McKee engineering and procurement hours projected at the end of June, 1979 was 688k, an increase of 56% over our base projection of 440k hours and 149k hours (28%) greater than our June, 1978 projections. This increase reflects the continued development of scope changes that occurred as detailed engineering progressed and additional design/operating problems were uncovered. The following tabulation summarizes the evolution of the engineering and procurement workload for the project.



FIGURE 2-1  
ECLP PROGRESS CURVES

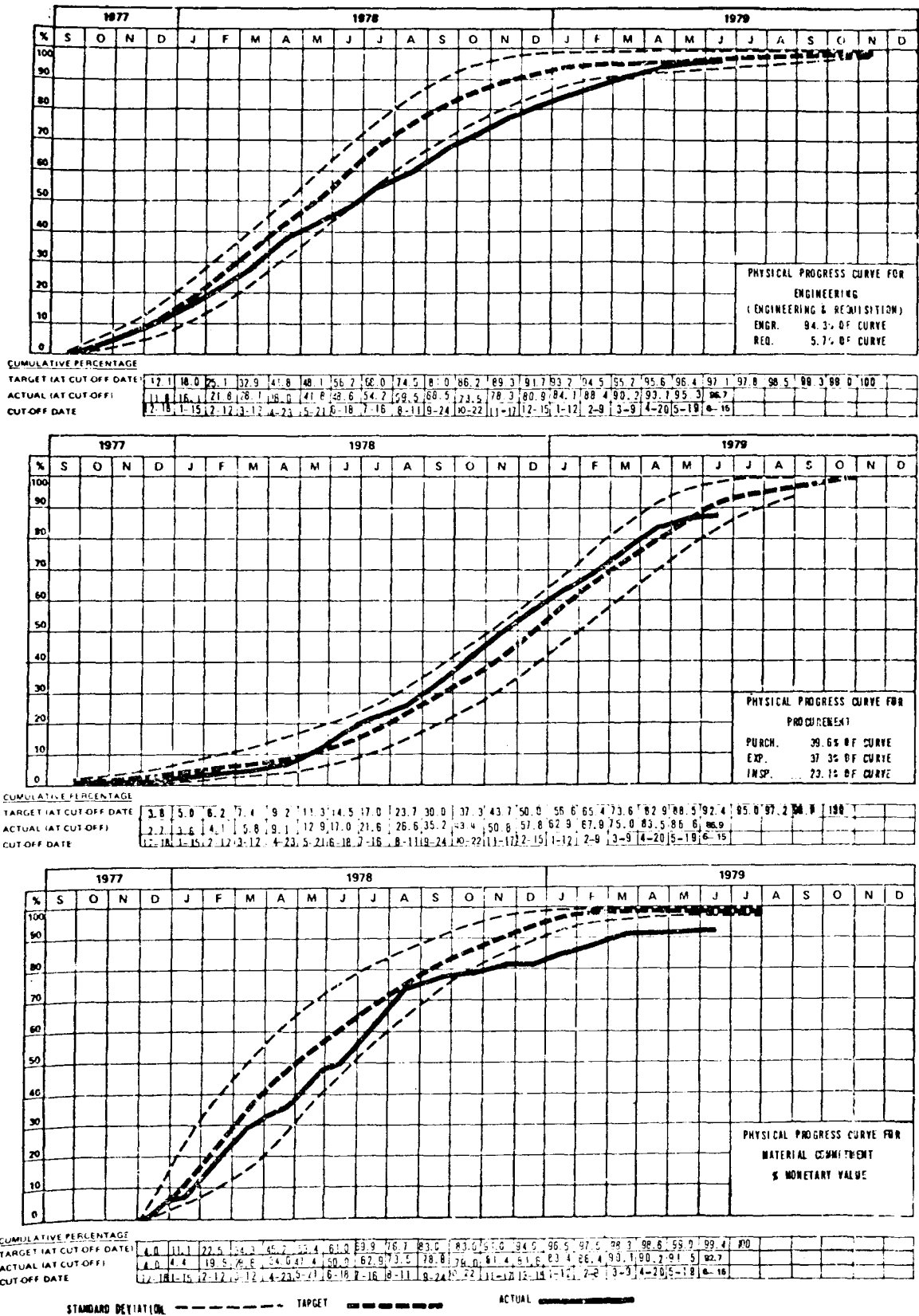
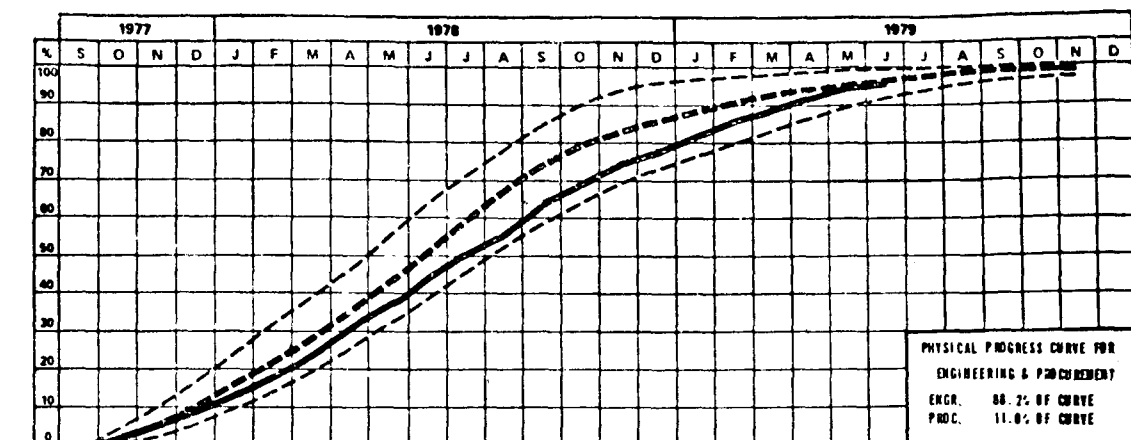
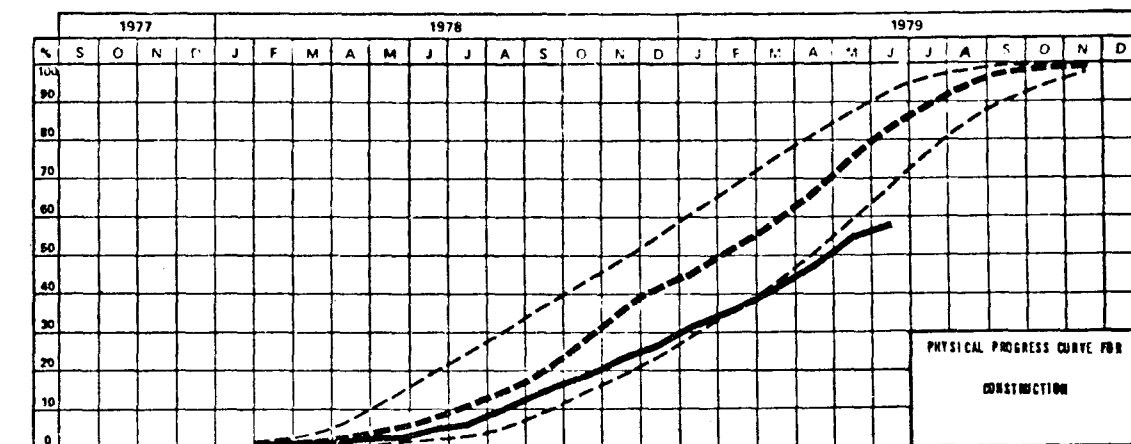


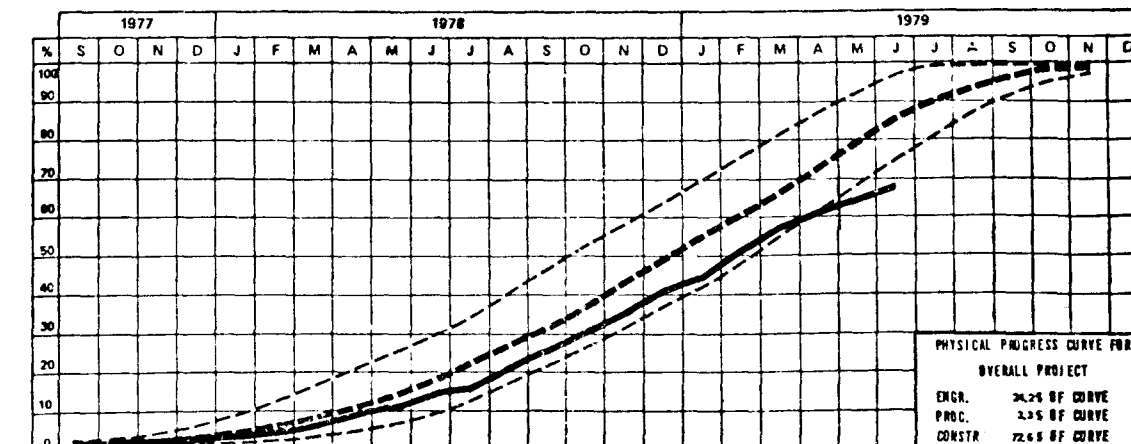
FIGURE 2-1 (cont'd)  
ECLP PROGRESS CURVES



CUMULATIVE PERCENTAGE	10.9	16.1	22.3	29.2	37.0	47.7	50.1	60.5	67.1	72.5	79.0	82.6	85.6	87.8	90.2	92.0	94.1	95.2	96.2	97.4	98.3	99.1	99.8	100.0
TARGET (AT CUT OFF DATE)	10.9	16.1	19.3	25.0	34.0	38.4	44.8	50.4	55.5	64.3	69.8	74.5	78.1	81.5	85.0	88.4	91.9	94.1	96.5					
ACTUAL (AT CUT OFF)	12.8	15.1	2.12	3.12	4.23	5.21	6.18	7.16	8.11	9.24	10.22	11.17	12.15	1.12	2.0	3.9	4.20	5.19	6.15					
CUT OFF DATE																								



CUMULATIVE PERCENTAGE	1	5	17	28	44	75	127	172	259	35	340	946	152	156	169	276	94	0	0	0	0	0	0	0
TARGET (AT CUT OFF DATE)																								
ACTUAL (AT CUT OFF)																								
CUT OFF DATE																								



CUMULATIVE PERCENTAGE	2.4	3.5	6.2	17.5	10.4	13.6	17.6	22.9	29.7	33.0	36.4	43.6	48.4	55.0	61.3	65.0	74.5	80.2	85.0	88.4	91.9	94.1	96.5	98.3
TARGET (AT CUT OFF DATE)	2.4	3.5	4.2	6.0	9.6	11.6	14.5	17.6	21.3	27.8	33	37.4	41.1	44.7	51.3	56	60.9	64.6	68.6					
ACTUAL (AT CUT OFF)	12.18	1.15	2.12	3.12	4.23	5.21	6.18	7.16	8.11	9.24	10.22	11.17	12.15	1.12	2.0	3.9	4.20	5.19	6.15					
CUT OFF DATE																								

Date	Eng./Proc. % Complete		Projected Workload		Manning Equivalent Full Time	
	Actual	Planned	k Hours	% Change	Actual*	Planned
Base	-	-	440.5	-	-	-
June, '78	47.0	55.0	538.9	22.3	229	205
Sept., '78	65.0	75.0	571.1	29.6	226	160
Dec., '78	79.0	86.0	615.5	40.0	195	72
Mar., '79	90.0	94.0	680.0	50.0	183	38
June, '79	96.0	97.0	688.2	56.0	67	25

\*Peaked at 241 in October, 1979.

At the end of June, 1979 all engineering work had been completed except for the following.

- + Piping - 250 out of a programmed 1,750 isometrics requiring minor revisions remained to be issued, 13 P&I diagrams were to be updated, and final material take-offs completed.
- + Instrumentation - Engineering requisitions for recent changes and instrument lists revisions were to be completed.
- + Electrical - Engineering of the electric heating systems, revisions to substation No. 1 layout and final vendor drawings checks remained to be completed.
- + Materials Handling and Mechanical - Only final checking of some vendor drawings remained.

Developments which added significantly to the engineering and procurement effort for the project during the year are highlighted in the following paragraphs.

- + Electric heating requirements covering a temperature range of 170°F to 850°F required the design of three separate systems (discussed in more detail later). Added engineering/procurement effort amounted to 19,920 hours. Overall costs for time and materials were 2,004 k\$. In May, 1979 the decision was made to replace the existing 7,500 kva main transformer with a 10,000 kva transformer because of the increased electrical loads. This change increased engineering hours by 2,000 and overall costs by 210 k\$.
- + Design modifications to permit the Dowtherm system to operate as low as 500°F were begun in December, 1978 and completed in May, 1979. Approximately 1700 hours were required and overall costs were increased by 97 k\$.
- + McKee began work on defining the boundaries of turnover packages in January, 1979 and completed associated material take-off work in June, 1979. About 6,830 hours were expended on this effort and total time and materials costs were 242 k\$.

- + Design basis studies and detailed engineering for the solvent blowdown and flushing system extended from March, 1978 to July, 1979. The final designs for this system included 13 pumps, two drums, and associated complex piping and instrument systems. Total hours expended were 31,410 and the overall cost for the system was 3,460 k\$.
- + The gas swept mill system was purchased as a package unit from Fuller. An estimated 10,600 engineering hours were expended in coordinating the engineering and procurement, expedite drawing, etc., over a period extending from June, 1978 through June, 1979. The additional cost for this effort was about 285 k\$.
- + Almost 13,000 valves of more than 590 different types (size, materials, rating, etc.) are required for the plant. The search for sources of supply to meet technical and schedule requirements resulted in an unusually protracted effort by engineering and procurement personnel, beginning in December, 1977 and continuing through June, 1979. Approximately 8,400 additional hours were required in this effort.

Preparation of the plant model, a vital engineering tool, continued into the second quarter of the year. As work progressed, the model was continually studied and used to resolve both engineering and anticipated operations problems. Teams made up of Carter, Exxon process and safety engineers, and Exxon project management personnel conducted formal reviews as the models of the various areas were completed. After McKee revised the models in accordance with the comments, piping isometric work was permitted to proceed. The following table summarizes the original planned and actual model review dates and shipping times for the various sections of the model.

Areas	Review Dates		Date Shipped to Field
	Original Plan	Actual	
Fuel gas, DEA regeneration	7/24/78	7/24/78	3/2/79
Utility area, pipe racks	7/24/78	7/24/78	4/21/79
Hydrogenation	9/14/78	8/4/78	5/18/79
Compressors, coal preparation	10/2/78	10/16/78	5/18/79
Liquefaction, distillation	10/9/78	9/11,18/78	5/18/79
Solvent flushing, Dowtherm	10/9/78	11/6/78	5/18/79

McKee originally estimated that 2,000 isometrics would be required for the plant, based on their historical piping data. Initial checks using Exxon's piping correlations supported this estimate. In December, 1978 McKee revised their estimate upward to 2,700 and in March, 1979 this was revised again to 3,145. The final number was 3,174, reflecting the unusual complexity of the plant. The following table highlights the changes in this critical engineering effort as final detailed piping design progressed.

Isometric Production		Actual Date	Estimated Number of Isometrics
Schedule/Revised	Schedule	of Issue	
Start	Sept., '78	Sept., '78	2,000 (original estimated number)
First issue	Nov., '78	Nov., '78	2,000 (original estimated number)
Complete	April, '79	Feb., '79	2,000 (original estimated number)
Complete	May, '79	April, '79	2,700 (revised projection 12/78)
Complete	June 1, '79	-	3,145 (revised projection 3/79)
Complete	-	June 9, '79	3,174 (actual count)

The increased scope of piping engineering work resulting from the above changes had a significant impact on the procurement of piping materials, the fabrication of pipe spools and the piping erection program for the plant.

In late 1978 and early 1979 Exxon project management team engineers prepared detailed descriptions and scope definitions of the thirteen sections of the plant which are to be turned over in an ordered sequence in keeping with Carter's start-up plan. Further subdivisions of some of the thirteen major turnover packages were prepared by Daniel and Carter. To assist in construction planning/execution as well as final checkout of each turnover package, McKee was requested to prepare listings of equipment, instruments, electrical facilities and piping drawings for each section and to define piping materials needed to provide for the separation/safe isolation of each package. This work was completed in June, 1979.

Requirements for the electrical heating of process lines and equipment could not be fully developed until the piping design work had progressed to the point where specific lines and/or potential operating problems could be fully identified. Work began in August, 1978 and continued to July, 1979 on heating systems design. The final designs include three different type systems.

- + Teflon insulated tracers for temperatures of less than 210°F (57 piping and 47 equipment circuits). Cost about 210 k\$.
- + Mineral insulated tracers for temperatures from +210°F - 600°F (85 piping and 44 equipment circuits). Cost about 272 k\$.
- + Impedance heating for critical services at temperatures in excess of 600°F (42 piping circuits). Approximate cost 224 k\$.

The scope of heat tracing required was considerably greater than defined in the design specifications and the added power needed for the systems dictated the addition of another substation and a 1125 kva standby diesel generator. The added heating load plus the power requirements for equipment added for other changes, such as the flushing and blowdown system, led to the decision in May, 1979 to replace the 7500 kva transformer at the main substation with a 10,000 kva unit. The new transformer is scheduled for delivery in December, 1979 and the changeover, which will require 1-2 days, will be made during an early turnaround.

During the year McKee completed the design/specification and procurement of the three principal components of the plant control/data collection system. In summary, the status of this work at year end was as follows.

- + Control house instrumentation was installed and the main control house was turned over to Carter for loop checking. The instrumentation for plant control/monitoring consists of split architecture Foxboro Spec 200 instruments mounted in a high density, console style, control panel 37 feet long. Signal conditioning and processing instruments are mounted in four freestanding rack sections. These racks also contain paralleled + 15 vDC instrument power supplies each of which is supplied with 120 vAC power from an uninterruptible power system with battery backup.

- + The data logger system utilized Digital Equipment Corporation PDP 11/34 and 11/04 minicomputers along with Herco multiplexers to acquire about 700 high and low level analog measurements and 128 digital status inputs. Data logger software systems provide for historical logging, alarming, real-time digital and graphic displays, data correlation, and user Fortran programming. This equipment is in final testing and will be delivered in July.
- + Automatic analyzers for the plant total 29, 15 of which are housed, along with their sample conditioning and calibration systems, in four pre-wired, pre-piped analyzer houses. Analyzers in three of the four houses had been functionally tested with calibration samples by the end of June. The fourth analyzer house was scheduled to be tested and shipped to the plant site in July. Of the remaining 14 analyzers, 13 have been delivered and 5 installed. The one remaining analyzer is scheduled for delivery by the end of August.

#### Procurement

The number of tagged equipment items increased from 470 to 516 during the year, primarily due to changes. All items have been ordered and 465 have been delivered to the construction site. As of June 30, 1979, commitments for all procurement totaled 37.9 M\$, 93% of the forecast total for the project vs. 99% planned. The status of procurement as related to tagged items through the end of June, 1979 follows.

	June 30, 1979	
	<u>Required/Purchased</u>	<u>Delivered</u>
Cooling Tower	1	1
Compressors	14	13
Drums	58	57
Exchangers	61	60
Furnaces	6	5
Pumps	146	121
Reactors	8	8
Tanks	17	17
Towers	15	15
Other	<u>190</u>	<u>168</u>
	516	465

The status of bulk materials purchased by both McKee and Daniel at the end of June, 1979 is summarized below.

	Cost, k\$		
	Projected	Committed	Committed, %
<u>McKee Purchases</u>			
Pipe and fittings	3,536	3,356	95
Valves	4,031	3,914	97
Instruments	3,174	3,071	97
Electrical	4,234	3,931	93
Steel	2,452	2,422	99
Subtotal	17,427	16,694	96
<u>Daniel Purchases</u>			
Miscellaneous	7,581	5,719	75
Total	25,008	22,413	90

Spare parts commitments through June, 1979 totaled 1,143 k\$, 92% of the total projected spares requirement on a cost basis.

As mentioned previously, almost 13,000 valves of over 590 different types are required for the project. The overall procurement status as of the end of June is tabulated below.

<u>Purchased through June, '79</u>	<u>Total</u>	<u>Shipped</u>	<u>Remaining</u>	<u>% Remaining</u>
Carbon steel	9,370	7,865	1,505	16
Slurry valves	325	130	195	60
Special valves (materials)	705	405	300	43
High pressure	1,784	717	1,067	60
Motor operated	25	5	20	80
Subtotal	12,209	9,122	3,087	25
<u>Added in June, '79</u>	22	3" and larger for turnover packages		
	68	small valves for miscellaneous changes		
	55	for pump cooling water systems		
	112	for flushing oil service		
	241	for process drains (3/4")		
	206	recommended surplus		
Total	12,913			

The valves added in June are all short delivery items and will be on order by mid-July.

Exxon delegated inspection of vessels, piping and heat exchangers to McKee. The division/status of inspection activities at the end of June, 1979 was:

		<u>Number of Orders</u>	
		<u>Main</u>	<u>Sub</u>
Exxon Inspection		47	13
McKee Inspection		171	109
Totals		218	122
Inspection Complete		140	118
Active		78	4

## Cost

The cost outlook for the project as of 30 June, 1979 was 112.15 M\$; see Table 2-1 for details. Major factors contributing to the increase were:

	Costs, k\$	
	Changes + Other	= Totals
June 30, 1978 Forecast		104,025
Materials	6299 2905	9,204
Direct Labor & Indirects	3609 7934	11,543
Subcontracts	(585) (739)	(1,324)
Engineering	2834 244	3,078
Pending & Anticipated Changes		(4,610)
Exxon Refinery/Chemical Plant Work		(335)
Exxon Engineering/Owner Expense		109
Contingency Rundown		(4,540)
Process Development Allowance Rundown		(5,000)
June 30, 1979 Forecast		112,150

The most significant increases during the year, other than increases attributable to approved changes, have been in bulk materials, direct labor and field indirects. The largest material increases were in field purchased bulk materials. Direct labor and indirects increased as the result of the increased bulk materials quantities projected and an anticipated three month schedule extension.

Two hundred eighty-one change orders have been issued to date; 269 of the changes totaling 11.794 M\$ have been approved. Summaries of approved, and pending and anticipated changes are shown in Tables 2-2 and 2-3, respectively. The continued addition of significant changes during the year caused a three month delay of mechanical completion. The rate of changes is shown graphically in Figure 2-2 which compares the cost of approved and pending changes, ex. budget shifts, to the contingency rundown. The following table compares the status of changes as of 30 June, 1979.

	30 June 1978		30 June 1979	
	Number	Cost, k\$	Number	Cost, k\$
Approved Changes	99	(713)	269	11794
Pending Changes	32	2195	12	355
Anticipated Changes	-	2740	-	180
Total Changes	131	4222	281	12329

An analysis of changes through 1979, excluding budget shifts, indicated that design basis (scope) changes accounted for 51.3% and design development 32.9% of the cost of all changes; estimate adjustments, miscellaneous and field changes totaled 15.8%. A summary of the analysis follows.



Type	Changes		Distribution, %	
	Number	Cost, k\$	Number	Cost
Design Basis: Major	33	6479.4	13.1	43.7
Minor	50	1131.4	19.8	7.6
Design Development: Major	13	3730.0	5.2	25.2
Minor	70	1140.8	27.7	7.7
Engineering Support	15	407.9	6.0	2.8
Miscellaneous	14	441.7	5.6	3.0
Estimate Adjustment	5	1276.1	2.0	8.6
Field/Start-up	33	210.0	13.1	1.4
Cancellations	19	-	7.5	-
Subtotal	252	14817.3	100.0	100.0
Budget Shifts	29	(2650.9)		
Total	281	12166.4		

Engineering overtime (excluding contract employees) at the end of June, 1979 on a cumulative basis was about 4.4%, an increase of approximately 2.3% over June, 1978. The increased overtime was necessary to minimize the impact of changes during the year. Including overtime worked by contract employees would increase the overall overtime by .25%. See Figure 2-3 for the cumulative overtime in engineering throughout the project.

The cost status of subcontracts at year end follows.

	Number	Costs, k\$	
		Projected	Committed
Directs			
Completed	11	2601	2575
In Progress	9	1648	1422
In Negotiation	3	68	-
Planned	4	83	-
Total Directs	27	4400	3997
Indirects	6	553	553
	33	4953	4550

### Schedule

A number of schedule updates were prepared during the year; highlights and conclusions resulting from updates Numbers 5 (June, 1978) through 9 (January, 1979) are summarized in Table 2-4. Following the January, 1979 update, work was begun to better define materials quantities and deliveries and to improve engineering and field labor forecasts. The schedule update planned for March, 1979 was delayed until this work could be completed. This study indicated that the overall mechanical completion date should be extended to 7 December, 1979 + 3 weeks, and that late delivery of materials, especially valves, could affect some interim completions.

A subsequent schedule update by the Daniel cost/scheduling group and an independent study by senior scheduling/cost specialists from Exxon Engineering's Project Management Department and the Project Team were completed in May. The studies were based on progress/productivity trends to date and an extensive review of the status of materials deliveries and labor projections. Results of these studies, along with the major assumptions on which they were based, are summarized below.

	Schedule Study		Original Projections
	Daniel	ER&E	
Total projected labor hours, M	1.795	1.830	1.145
Equivalent direct labor (peak)	880	715	430
Level of changes	Minimum	Minimum	-
Valve deliveries by	1 Dec.'79	1 Dec.'79	Aug.'79
All pipe spools delivered by	1 Sept.'79	1 Sept.'79	15 July,'79
Overall mechanical completion	1/25/80	2/15/80 + 2 wks	11/15/79

The ER&E study was viewed as providing the "most probable" completion date and, as such, has been used as the basis for the current cost outlook. However, the 25 January, 1980 completion date has been set as the "target" date for completion. Dates for the various interim turnovers developed in the Daniel study were also set as "targets" (see Table 2-5) and field planning/manning will be consistent with these dates with the full recognition that Daniel's ability to hit the "targets" may be affected by bulk materials deliveries, the level of future changes, and appreciable changes in productivity.

TABLE 2-1

**EXXON COAL LIQUEFACTION PILOT PLANT  
PROJECT COST SUMMARY THROUGH JUNE 30, 1979**

	Cost Outlook k\$(1)	Original Control Estimate(2) k\$	Changes, Transfers & Estimate Adjustments k\$(3)	Revised Control Estimate k\$	Forecast k\$	Underrun(-) Overrun(+) k\$	Commitments k\$
<b>McKee &amp; Daniel</b>							
Direct Material	38,100	34,062	8,570	42,632	40,800	1,832 (-)	37,737
Direct Labor	12,370	11,604	1,647	13,251	16,710	3,459 (+)	7,684
Subcontracts (4)	4,810	8,305	(2,510)	5,795	4,400	1,395 (-)	3,997
Field Indirects (5)	19,910	19,030	99	19,129	20,950	1,821 (+)	13,229
Fee (6)	2,000	1,323	192	1,515	1,550	35 (+)	1,195
Engineering (7)	13,610	12,500	3,446	15,946	15,600	346 (-)	14,960
Loss on Surplus	460	490	-	490	300	190 (-)	-
Insurance	250	600	-	600	600	-	564 (13)
Pending Changes (8)	-	-	145	145	145	-	-
<b>Total Prime Contract</b>	<b>91,510</b>	<b>87,914</b>	<b>11,589</b>	<b>99,503</b>	<b>101,055</b>	<b>1,552 (+)</b>	<b>79,366 (12)</b>
Baytown Refinery Costs(9)	-	-	3,984	3,984	2,710	1,274 (-)	1,902 (13)
Anticipated Changes	180	-	180	180	180	-	-
ER&E Services (10)	4,200	4,200	(554)	3,646	4,115	469 (+)	3,330 (14)
Owners Costs (11)	1,930	1,930	251	2,181	1,800	381 (-)	1,201 (13)
Project Contingency	10,180	9,486	(7,196)	2,290	2,290	-	-
Process Development Allowance (15)	<u>2,000</u>	<u>5,000</u>	<u>(5,000)</u>	<u>-</u>	<u>-</u>	<u>-</u>	<u>-</u>
<b>Total Project Cost</b>	<b>110,000</b>	<b>108,530</b>	<b>3,254</b>	<b>111,784</b>	<b>112,150</b>	<b>366 (+)</b>	<b>85,799</b>

Notes: See next page

NOTES:

- (1) Cost Outlook dated 9 September, 1977.
- (2) The Original Control Estimate is the Exxon Research and Engineering Class II Estimate which excludes all changes.
- (3) Changes, transfers, and estimate adjustments are alterations to the Original Control Estimate and include changes through 15 June, 1979.
- (4) Major subcontracts include tankage, piling, coal silo, cooling tower erection, railroad spur, and site preparation.
- (5) Includes burden, field supervision, construction tools, temporary facilities and vendor representatives.
- (6) Includes fee for both engineering and construction contractors.
- (7) The Original Control Estimate reflects the Class II Estimate prepared by ER&E. The forecast for engineering includes McKee's engineering only.
- (8) Includes Change Orders now being processed but not yet approved.
- (9) Includes costs associated with the Baytown Refinery and Chemical Plant for the engineering, procurement, and erection of interplant lines and the reconditioning of Burleson Street. The forecast reflects subcontract costs to Brown & Root for associated work in both the Refinery and Chemical Plant. Costs for the Baytown Chemical Plant facilities required for disposal of ECLP naphtha blend as distillate fuel, \$350k per Chemical Plant Class II estimate, and the Baytown Refinery facilities required for disposal of ECLP Vacuum Gas Oil in Dau Rock, \$110k per Refinery Class II estimate, are also included.
- (10) The Revised Control Estimate reflects a transfer of the cost of inspection services to McKee, the transfer of ER&E services for the preparation of operating guides to the Owners Account, and transfer to McKee for the work associated with the preparation of change lists.
- (11) Includes: Administration and General Purpose Building, communication system, power hookup to Houston Power & Light, additional site borings, and the encasement of Texas Eastern, Diamond Alkali, and the Houston Pipeline Co. pipelines, a transfer of the data logger programming costs from the McKee material account, a transfer of costs for the preparation of operating guides from ER&E services.
- (12) Includes commitments through 15 June, 1979.
- (13) Includes commitments through 30 June, 1979.
- (14) Includes expenditures through 31 May, 1979.
- (15) Process Development Allowance has been applied to the following approved changes, OCR-85 and OCR-185 (Partial), Flushing and Blowdown; OCR-86, Corrosion Probe Data Gathering; and OCR-114, Data Logger Calculated Variables. A transfer of 2M\$ has been made to Phase V of the EDS program budget and is no longer included in our revised control estimate or cost forecast. Revising the Class II estimate to reflect this transfer to Phase V results in an original control estimate of \$106,530k.

TABLE 2-2  
EXXON COAL LIQUEFACTION PILOT PLANT  
SUMMARY OF APPROVED CHANGES  
THROUGH JUNE 15, 1979

<u>Change No.</u> <u>OCR</u>	<u>Description</u>	<u>Type*</u>	<u>Cost</u> <u>k\$</u>	<u>Schedule</u> <u>Effect</u>
1 thru 205, 207 thru 210 212 thru 216 EFCR 1 thru 34 36, 37, 40, 41	Two hundred fifty two Previously Approved/Cancelled Changes		9885.6	+ 3 mos.
206	McKee Process Assistance in Preparing Turnover Packages and Procurement of Additional Valves	OT	241.8	None
211	Provide Alternates for Pacific and Velan Long Delivery Valves	DD	50.0	None
217	Additional Electric Heat Tracing Requirements	DD	1,270.3	None
219	Miscellaneous Piping and Relief Valve Comments	DD	20.1	None
221	Changes in Analyzer Sample Probes	DD	12.9	None
222	Increase Size of Main Transformer and Modifications to Substation #1	DB	209.9	None
223	Miscellaneous Piping and Instrument Changes	DD	54.3	None
<u>EFCR</u>				
38	Relocate Gate House Airconditioner	FC	.3	None
39	Control House Wiring Changes	FC	8.5	None
42	Change North-South Roadway to Concrete	FC	15.8	None
43	Color Drawings for Turnover Packages	OT	10.0	None
44	Add Lifting Lugs to Five Vessels	FC	2.0	None
45	Pull GTE Cable from Administration Building to General Purpose Building	FC	3.0	None
47	Add Instrument Air to Control House	FC	1.1	None
48	Provide 208V Power to Instrument Shop in Control House	FC	1.8	None
49	Remove Raised Face Flanges from Non-Oily Water Sump Pumps	FC	2.0	None
50	Provide Equipment Grout Protection	FC	4.6	None
Total Negative Approved Changes			(4507.3)	
Total Positive Approved Changes			16301.3	
Grand Total Approved Changes			11794.0**	

\*DD-Design Development; DB-Design Basis; EA-Estimate Adjustment; BS-Budget Shift; OT-Other; FC-Field Change; SC-Startup Change

\*\*Does not reflect \$350k transfer to preparation for operations (C-33)

TABLE 2-3  
EXXON COAL LIQUEFACTION PILOT PLANT  
SUMMARY OF PENDING AND ANTICIPATED CHANGES  
THROUGH JUNE 15, 1979

<u>PENDING CHANGES</u>			Execution	Cost k\$
Change No.	Description	Type*	Authority	(Order of Magnitude
OCR			Given	Note 1
218	Design Specification Change Lists	DB	Yes	10
220	Modifications to Reacceleration	DD	Yes	20
224	Miscellaneous Additional Engineering Requirements	OT	Yes	10
225	Coal Unloading Third Rail Resolution	DD	Yes	5
226	Pump Flushing System Changes	DD	Yes	10
Subtotal Office Changes				55
<u>EFCR</u>				
35	Provide Non-Slip Walkways along Conveyors	FC	No	10
46	Add Equipment Wash Rack	FC	No	10
51	Liquefaction Reactor Flange Modifications	FC	Yes	5
52	Replace Teflon Tape Thread Lubricant	FC	No	5
53	Additional Waste Water Instrument Requirements	FC	Yes	15
54	Add Lifting Lugs to Five Towers	FC	No	5
55	Additional Concrete Road Paving	DB	No	40
Subtotal Field Changes				90
Grand Total Pending Changes				145

Note 1: Order of Magnitude costs only reflects the portion of the change not already included in the direct forecast. Actual cost of the changes will not be shown until they have been approved. Attachment VII however reflects the total anticipated cost of pending changes.

\* DD-Design Development; DB-Design Basis; EA-Estimate Adjustment; BS-Budget Shift; OT-Other; FC-Field Change; SC-Startup Change

ANTICIPATED CHANGES

- Repair Baker Road	+ 60
- Oyster Shell in Tank Farm	+ 20
- Miscellaneous	+100
	+180

TABLE 2-4  
 EXXON COAL LIQUEFACTION PILOT PLANT  
HIGHLIGHTS/CONCLUSIONS OF SCHEDULE UPDATES

<u>REPORT NUMBER/DATE</u>	<u>HIGHLIGHTS/CONCLUSIONS</u>
5, June 1978	<ul style="list-style-type: none"> <li>* Original planned mechanical completion dates can be achieved.</li> <li>* Possibility of 2-3 week schedule extension</li> <li>* All valve deliveries projected by May 1, 1979</li> <li>* Changes recognized as potential for schedule slippages</li> </ul>
6, August 1978	<ul style="list-style-type: none"> <li>* Original planned mechanical completion dates can be achieved.</li> <li>* Possibility of 4-5 week schedule extension</li> <li>* Spot overtime approved to achieve turnovers</li> <li>* Valve deliveries later than May 1, 1979 identified</li> <li>* Critical vendors need to be continually expedited</li> <li>* Schedule slippages resulting from major changes appeared probable</li> <li>* Schedule effect of flushing and blowdown system to be more critically assessed</li> </ul>
7, October 1978	<ul style="list-style-type: none"> <li>* Original planned mechanical completion dates can be achieved.</li> <li>* Flushing and blowdown system assessed to have a probable 4 week delay on mechanical completion. Further study required.</li> <li>* Overtime to be utilized to achieve turnovers.</li> <li>* Underground electrical program hampered by late deliveries.</li> </ul>
8, December 1978	<ul style="list-style-type: none"> <li>* Original planned mechanical completion dates (except electrical facilities) can be achieved.</li> <li>* Overtime or double-shifting still required to obtain planned mechanical completion</li> <li>* Valves deliveries expected as late as 30 June 1979.</li> <li>* Extensive effort seen in utilizing material substitutions to meet field need dates.</li> <li>* Revised mechanical completions for substations developed.</li> <li>* Material deliveries continue to be critical</li> </ul>
9, January 1979	<ul style="list-style-type: none"> <li>* Mechanical completion dates revised for a number of turnovers, Nov. 15, 1979 still achievable.</li> <li>* Revised manpower loadings eliminate need for extensive overtime or double-shifting to achieve revised completion dates.</li> <li>* Valve and control valve deliveries major concern for meeting turnovers.</li> <li>* Substantial increase in critical field activities</li> <li>* Weather adversely affecting progress</li> <li>* Late material deliveries identified by interim turnovers.</li> </ul>

TABLE 2-5  
EXXON COAL LIQUEFACTION PILOT PLANT  
COMPARISON OF PLANNED/PROJECTED TURNOVERS

<u>Turnover Package</u> <u>No. Description</u>		<u>Completion Dates</u>			<u>Current Predicted</u>
		<u>Original Plan</u>	<u>Mar., '79 Revision</u>	<u>May, '79 Targets</u>	
1	Firewater System	4/06/79	4/06/79	Complete	-
2	Electrical Facilities				
	- Main Substation	5/25/79	5/25/79	Complete	-
	- Substation 4	5/25/79	5/25/79	7/06/79	7/13/79
	- Substation 4A	5/25/79	6/01/79	7/06/79	7/13/79
	- Substations 1,3,5	5/25/79	6/15/79	7/06/79	7/13/79
	- Substation 2	5/25/79	6/22/79	7/06/79	7/13/79
	- Substation 4B	5/25/79	8/24/79	8/24/79	8/24/79
3	Control House	6/01/79	6/01/79	Complete	-
4	Utilities	6/29/79	6/29/79	7/27/79	7/27/79 (1)
5	Waste Treating	7/13/79	7/13/79	7/27/79	7/27/79 (1)
6	Coal Receipt	7/27/79	7/27/79	7/27/79	7/27/79 (1)
7	Analyzers	8/10/79	8/10/79	8/10/79	8/10/79
8	Tankage				
	- South Tank Farm	8/10/79	8/10/79	8/17/79	8/17/79
	- North Tank Farm	8/10/79	10/12/79	10/12/79	10/12/79
	- Sponsor's Tankage	11/15/79	11/15/79	10/26/79	10/26/79
9	Coal Preparation	8/24/79	9/14/79	9/28/79	9/28/79
10	Safety Facilities	8/24/79	8/24/79	8/31/79	8/31/79
11	Fuel Gas				
	- Compressors	9/07/79	8/31/79	9/21/79	9/21/79
	- Unit	9/07/79	9/28/79	10/12/79	10/12/79
12	Hydrogenation				
	- Compressors	10/05/79	9/28/79	11/16/79	11/16/79
	- Unit	10/05/79	10/26/79	12/21/79	12/21/79
13	Liquefaction & Distillation				
	- Solidification Conveyor	11/15/79	11/16/79	10/19/79	10/19/79
	- Compressors	11/15/79	11/16/79	12/21/79	12/21/79
	- Unit	11/15/79	12/07/79	1/25/80	2/15/80 ± 2 wks

(1) Currently under study. Slippage may occur because of anticipated late deliveries of power, instrument and lighting panels.



FIGURE 2-2

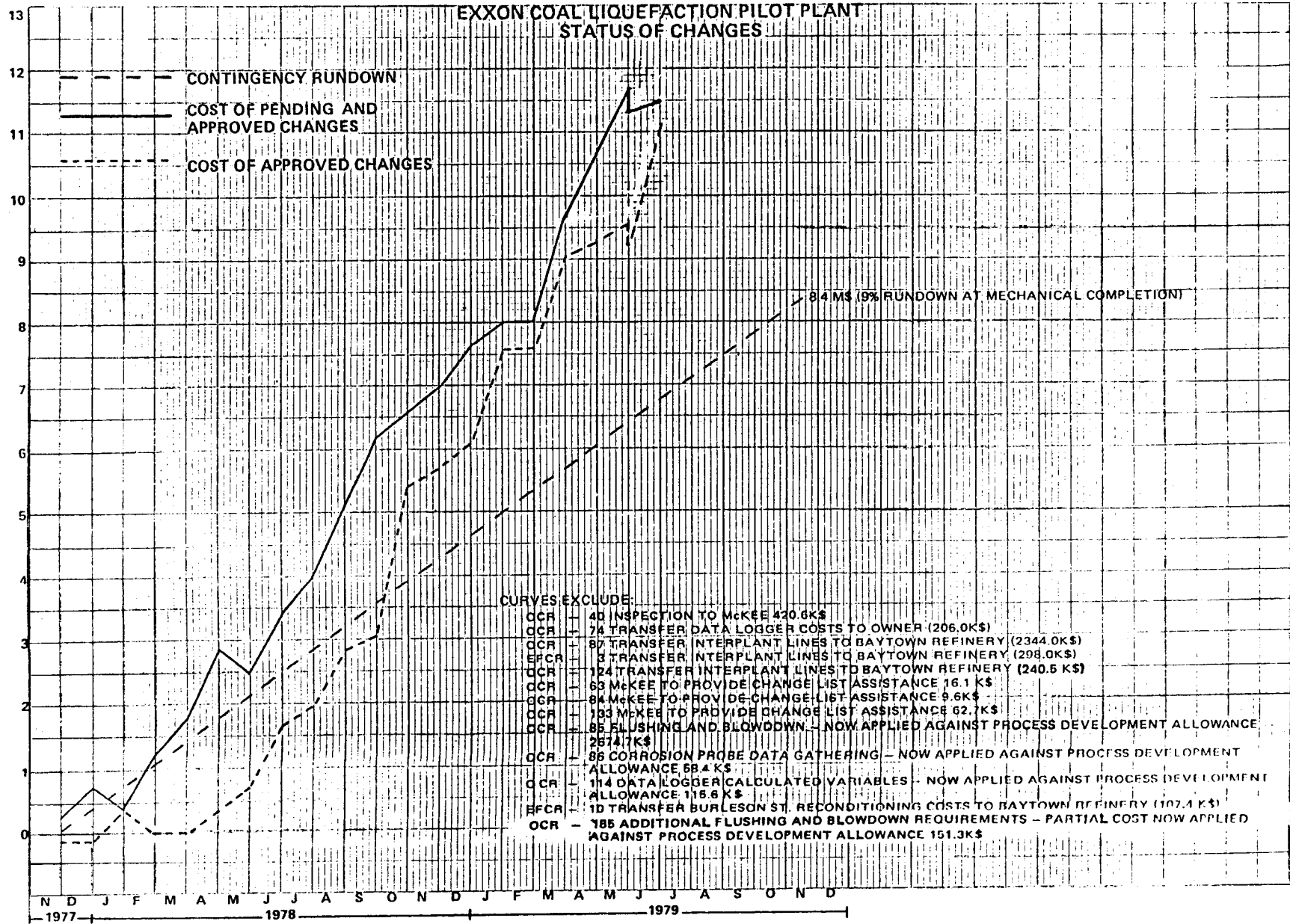
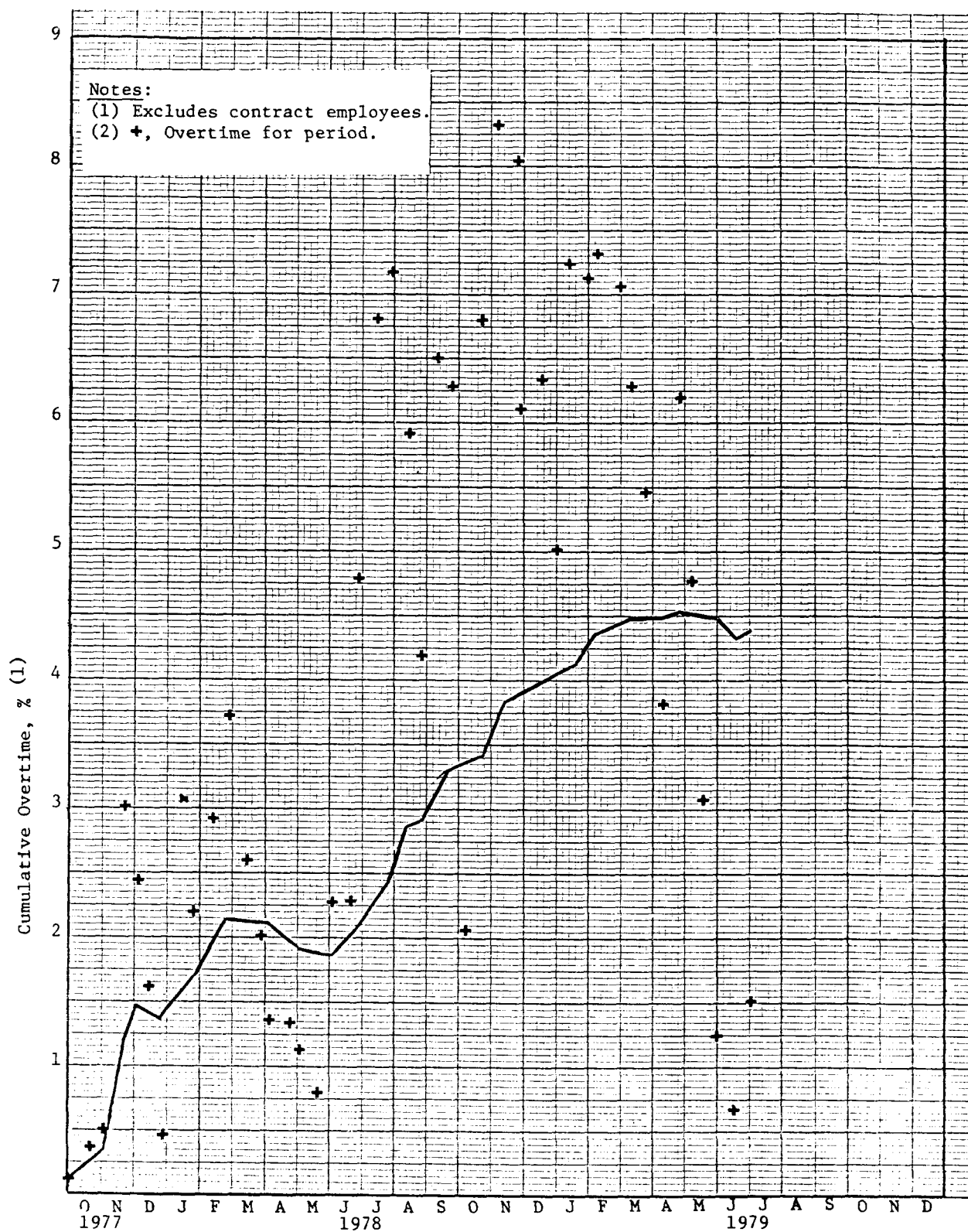


FIGURE 2-3

EXXON COAL LIQUEFACTION PILOT PLANT  
CUMULATIVE ENGINEERING OVERTIME



ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,  
PROCUREMENT AND CONSTRUCTION

3. Construction

Construction was 62% complete; about 24% or 3 months behind our original plan at the end of June, 1979. Progress during the year was affected by the increased scope of work, adverse weather, delayed completion of engineering and late delivery of materials. As shown in the following table, projected direct labor requirements very closely tracked the increases in engineering and procurement discussed earlier in this report.

Date	% Complete		Projected Direct Labor		End of Month Personnel	
	Actual	Planned	k Hours	% Change	Actual	Planned
July '78	9	12	1145	-	307	395
Aug.	13	16	1145	-	383	421
Sept.	16	22	1145	-	493	522
Oct.	20	31	1229	7	514	554
Nov.	25	37	1235	8	536	503
Dec.	29	43	1241	8	600	522
Jan. '79	33	49	1259	10	832	609
Feb.	40	55	1261	10	843	670
Mar.	45	62	1384	21	936	670
Apr.	49	70	1485	30	977	689
May	55	80	1795	57	922	654
June	62	86	1808	58	1011	622

The "actual" personnel figures shown above include all contractor personnel including direct and indirect labor, and supervision. Personnel devoted to direct activities are shown in Figure 3-1 in comparison with the original and revised projections of "equivalent" personnel.

Major construction events during the reporting period are shown below; monthly highlights are shown in Table 3-1.

	1978
First hydrostatic test of underground piping	July
Awarded purchase order prefabricated pipe	3 Aug.
Completed slipforming of raw coal silo	27 Aug.
Started tank erection	26 Sept.
Initiated field Positive Materials Identification program	2 Oct.
Started area paving	27 Oct.
Started erection of process area pipe rack steel	Oct.
Started installation of aboveground piping	Oct.
Received first shipment of prefabricated pipe	16 Nov.
Hired first apprentices	29 Nov.
Started construction of cooling tower	Nov.
Started installation of underground power cables	Dec.

	<u>1979</u>
Received and set all eight reactors	Jan.
Received and set control house instrument racks	Feb.
Completed cooling tower erection	Feb.
Firewater system mechanically complete	6 April
Completed erection of field fabricated tanks	April
Interplant lines mechanically complete	31 May
Control house mechanically complete	1 June
Main electrical substation mechanically complete	15 June
Completed underground power/instrument cables	29 June

The total effect of adverse weather on productivity cannot be measured in absolute terms; however, during the first half of 1979 it impacted heavily on construction activities. From 1 January, 1979 to 30 June, 1979 total rainfall, as measured at the Exxon Refinery in Baytown, was over 41 inches; normal annual rainfall for this location is 45 inches. During this six month period the project was completely closed down on 12 occasions because of rain and at numerous other times work was slowed by showers and extremely wet conditions.

The status of equipment receipt and erection at the end of the year is summarized below.

<u>Description</u>	<u>Total</u>		
	<u>Purchased</u>	<u>Received</u>	<u>Erected</u>
Cooling Tower	1	1	1
Compressors	14	13	13
Drums	58	57	47
Exchangers	61	60	56
Furnaces	6	5	5
Pumps	146	121	91
Reactors	8	8	8
Tanks	17	17	17
Towers	15	15	15
Other	<u>190</u>	<u>168</u>	<u>41</u>
	516	465	294

The status of piping prefabrication work by the offsite fabricator and at the field shop at year end was as follows.

<u>Fabrication</u>	<u>Materials</u>	<u>% Complete</u>	
		<u>Isos/Orthos</u>	<u>Spools</u>
		<u>Issued</u>	<u>Fabricated</u>
Shop	C.S.	100	79
Shop	Alloy	100	16
Field Shop	C.S./Alloy	99	84
Field Run	C.S.	99	20

FIGURE 3-1  
EXXON COAL LIQUEFACTION PILOT PLANT  
DIRECT LABOR

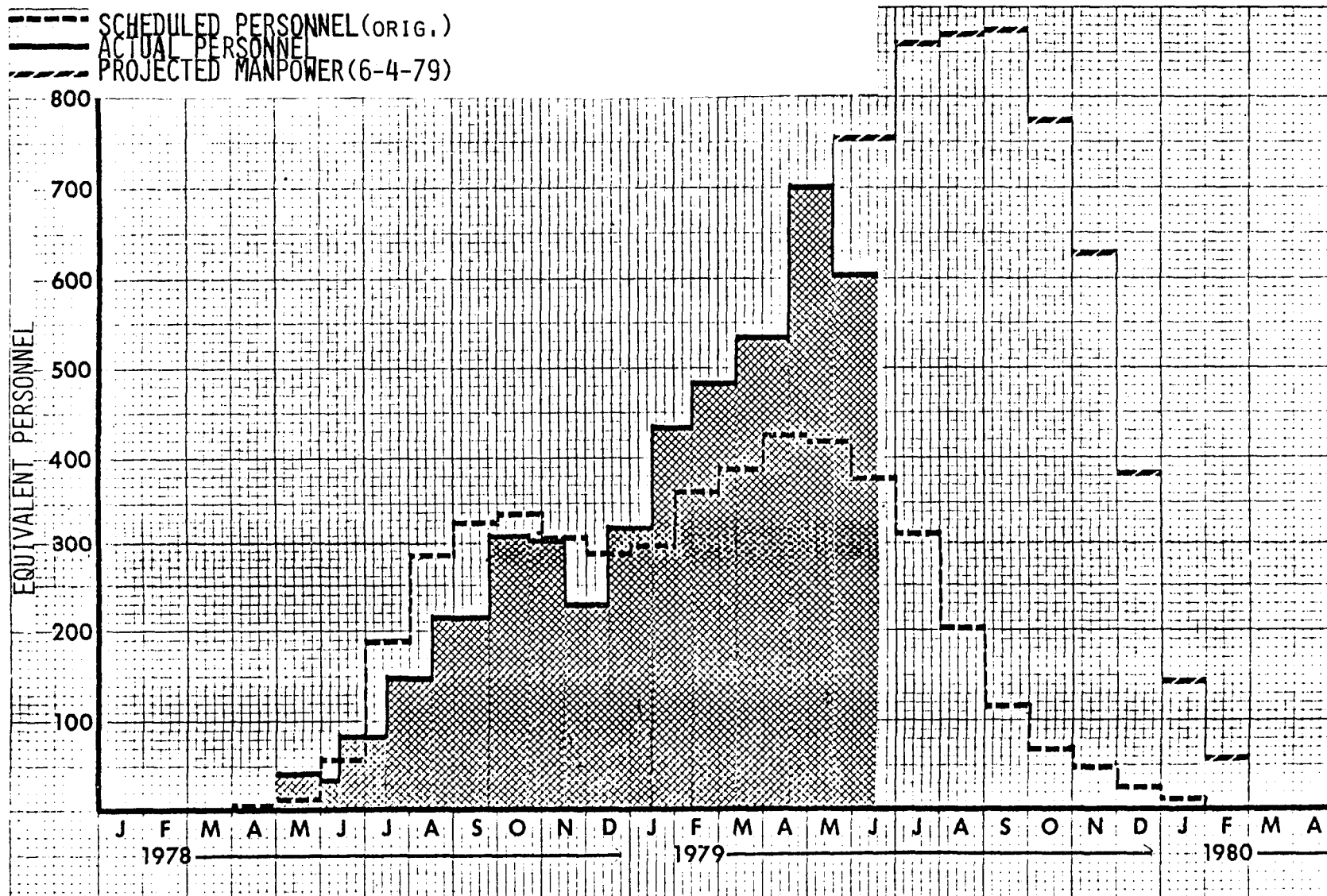


TABLE 3-1

EXXON COAL LIQUEFACTION PILOT PLANT  
SIGNIFICANT CONSTRUCTION EVENTS

Page 1 of 2

1978

- + July -
  - Sheet piling completed.
  - Piling cap for coal silo completed.
  - Raw coal silo subcontractor mobilized.
  - First structural steel arrived at site.
  - First tankage ring walls formed and poured.
  - Liquefaction furnace foundations completed.
  - Site preparation essentially completed.
  - Site fencing completed.
- + August -
  - Coal unloading hopper excavation completed and base slab started.
  - Tank base excavation and crushed stone foundations started.
  - Control house foundations started.
  - Firewater intake structure completed.
  - First pieces of major equipment placed.
  - Cooling tower foundations and basin started.
  - Oil and non-oily water sumps excavations completed.
  - Slipforming of coal storage silo shell completed.
  - Laying of rail spur tracks, ex ballast essentially complete.
- + September -
  - Tank erection started.
  - Cooling tower basin completed.
  - Raw coal silo roof poured.
  - Structural steel erection started.
  - Lower floor of coal unloading hopper poured.
  - Spur turnout from main rail line completed.
  - Precasting catch basins and manholes started.
  - Second major lift crane placed in operation.
  - Equipment preservation program initiated.
- + October -
  - Track hopper pit walls poured.
  - Track hopper pit tunnel excavation started.
  - Waste water sump first stage walls poured.
  - Installation of the support steel for the raw coal transport conveyor started.
  - Pile cap and columns for the liquefaction reactor structure poured.
  - First section of area paving completed.
  - Underground piping completed in the fuel gas area.
  - Pipe rack steel erection started in the process units.
  - Installation of rack piping started.
  - Railroad tie-in completed - spur now available for equipment deliveries.
- + November -
  - Assembly of raw coal transport conveyor support trusses completed on the ground and conveyor installation started.
  - Track unloading hopper concrete work and conveyor tunnel excavation completed.
  - Clearing and grubbing started in the sponsors' tank farm area.
  - Erection of precast wall panels and roof tees for the control house started.
  - Non-destructive testing facilities set up and X-ray examinations of welds initiated.
  - First apprentices hired for the apprentice training program.
  - Received the first shipments of prefabricated pipe on 16 November.
  - Pipe welding started in the major pipe racks.
  - Foundations for four compressors poured.
  - Largest process vessel, D-751, received and set in place.
  - Steel erection on several onsite structures initiated.
- + December 1978 -
  - Main substation transformer and 138kv circuit breaker set.
  - Installation of gas swept mill foundations started.
  - Waste water sump concrete work completed.
  - Track unloading hopper conveyor tunnel concrete work started.
  - Installation of underground power cables started.
  - First prefabricated pipe erected.
  - Started foundation work for flare and associated equipment.
  - Erection of precast walls and roof tees for the control house completed.
  - Completed structural work on the cooling tower.
  - Contracts awarded by the Exxon Refinery and Chemical Plant for the interplant piping.
- + January 1979 -
  - Equipment installation in track hopper was started.
  - Three of the four support trusses of the raw coal transport conveyor were erected.
  - The main substation was placed.
  - About 90% of the low level foundations for the gas swept mill system were completed and backfill started.
  - Excavation for impact mill foundations was started.
  - The raw coal silo elevated floor was poured.
  - Structural steel erection was started for the prepared coal bin structure.
  - All eight reactors were erected.
  - The main oily water sewer was completed to the waste water sump.

TABLE 3-1 (cont'd)

EXXON COAL LIQUEFACTION PILOT PLANT  
SIGNIFICANT CONSTRUCTION EVENTS

- + February -
  - The HVAC system in the control house was energized on temporary power.
  - The Foxboro Spec 200 instrument racks were received and set in the control house.
  - Installation of aboveground firewater piping in the firewater intake pump area was started.
  - Prefabricated substations 1, 3, and 5 were delivered and substations 1 and 3 were set in place.
  - Fireproofing of structural steel in the onsite area was started.
  - The track unloading hopper tunnel floor was poured.
  - Erection of two process unit furnaces was started.
  - Placement of pipe in offsite pipe racks was started.
- + March -
  - Preparations for turnover of the firewater facilities on 6 April were completed.
  - Area paving in utility area was started.
  - All vessels in the distillation and liquefaction separator structures were placed.
  - Substation 5 was placed.
  - Control panels from Panelmatics, Inc. were received and set in the control house.
  - Installation of permanent lighting in the fuel gas area was started.
  - The coal unloading pit tunnel was completed.
  - Completed 95% of the gas swept mill foundations.
  - Field assembly of the prepared coal bin was started.
  - Erection of the raw coal conveyor support steel was completed.
  - Substation 4B foundations were completed.
  - Completed hydrotest of eight tanks and started testing four others.
- + April -
  - The firewater system, Turnover #1, was completed as scheduled on 6 April.
  - Construction of the flare stack was started.
  - CB&I completed construction of the field erected tanks.
  - Marley completed "punch list" work on the cooling tower.
  - Control house "punch list" work was started in preparation for the scheduled 1 June turnover.
  - Piping tie-in at the ECLP boundary with the interplant pipe lines installed by the Exxon Refinery and Chemical Plant was started.
  - Impact mill and gas swept mill foundations were completed except for two small pumps.
  - The gas heater furnace for the gas swept mill was delivered and set on its foundation.
  - Erection of the prepared coal bin was completed.
  - The elevated slab for the separator structure in the liquefaction/distillation unit was poured.
  - Structural steel around the liquefaction reactors was erected.
  - Miscellaneous steel erection in hydrogenation and distillation areas was completed.
  - Completed installation/welding of the hydrogenation unit furnace tubes.
  - Foundations were poured and structural steel was erected for the Sandvik conveyor.
- + May -
  - Interplant lines were mechanically complete.
  - Strapping of tanks was completed.
  - Erection of the vacuum stripper furnace, F-201, was started.
  - The prefabricated electrical substation #2 was set.
  - Installation of underground instrument and power cables in the hydrogenation, liquefaction, distillation, and compressor areas was completed.
  - Erection of the solids withdrawal conveyor structure was completed.
  - Initial leveling and alignment work on the Ingersoll-Rand compressors was started.
  - Assembly of gas swept mill ductwork was started.
  - Testing of the main substation was completed.
- + June -
  - The control house was mechanically complete on 1 June.
  - The main substation was mechanically complete on 15 June.
  - Checkout and hydrotesting of piping was started in preparation for the late July scheduled completion date for the utility area.
  - Construction of tank farm fire dikes was started.
  - The prefabricated electrical substation #4B was set.
  - Tank mixers in the south tank farm were set.
  - Construction of the flare stack anchors was completed.
  - Installation of underground power and instrumentation cables was basically completed.
  - Erection of the coal transport conveyors in the coal preparation area started.
  - Alignment of conveyors in the coal receipt area was completed.
  - Refractory lining of the gas swept mill ducting was started.
  - Structural fireproofing in the fuel gas and liquefaction areas was completed.
  - Stacks for the slurry furnaces, F-102A/B, were set.
  - Area paving in 85% of the process area was completed.

The following table summarizes the status of piping weld quality control.

<u>Contractor</u>	<u>Material</u>	<u>Welds</u>		<u>Rejections</u>	
		<u>Number</u>	<u>Radiographed</u>	<u>Number</u>	<u>%</u>
Field	C.S./S.S.	11,106	1038	117	11.3
	*Alloy	25	19	8	42.1
Offsite Fabricator	C.S./S.S.	11,581	671	64	9.5
	*Alloy	199	132	4	3.0

\*Carbon 1/2 moly through 9 chrome, requires 100% radiograph.

A Positive Materials Identification Program (PMI) was initiated at the construction site in October of 1978. A nuclear analyzer is used to check all alloy piping materials received at the site or at the prefabrication shop. Results of this program as of 29 June, 1979 are summarized below.

<u>Material</u>	<u>Number of Items Checked</u>			
	<u>Field</u>		<u>Shop</u>	
	<u>Checked</u>	<u>Rejected</u>	<u>Checked</u>	<u>Rejected</u>
9 Chrome 1 Moly	30	0	0	0
Carbon 1/2 Moly	166	110	51	0
1 1/4 Chrome	11	0	30	0
2 1/4 Chrome	48	4	48	6
5 Chrome 1/2 Moly	232	0	189	0
304 Stainless Steel	384	1	396	0
316 Stainless Steel	864	14	15	0
321 Stainless Steel	749	1	561	0
ENCO 825	44	0	0	0
Alloy 20	36	0	70	0
Totals	2,564	130	1,360	6
Items rejected, %		5.5		0.4

The status of active subcontract work at year end is summarized below.

- + Work was completed on the raw coal silo on 11 June.
- + The tank construction subcontractor returned to the site to correct exception list items on the field fabricated tanks.
- + The electrical testing subcontractor is continuing work on substations and related electrical facilities.
- + Tank painting continued.
- + The railroad work at the coal unloading hopper is being completed.
- + The subcontractor started refractory work in the ducting for the gas swept mill. Preparations to install refractory in various process vessels and furnaces were also started.
- + The successful bidder for post weld heat treating work was awarded a unit price contract on 19 June, 1979.



- + Inquiries for subcontracting chemical cleaning work were reviewed by Exxon and will be issued by Daniel early in July.
- + A contract was awarded by the Exxon Chemical Company for the interplant line and roadway between ECLP and the Chemical Plant. Except for upgrading Burleson Street this work has been completed.
- + Work on the modifications to the Paraxylene Absorption Unit (PAU) furnaces in the Exxon Chemical Plant (to permit burning of ECLP products) and the VGO-DAU blending facilities in the Exxon Refinery should be complete as scheduled on 2 January, 1980. The design for the PAU project is complete and a subcontractor has been selected to do the construction. The DAU project is in the design stage; construction on both projects is scheduled to begin late in the summer of 1979.

ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,  
PROCUREMENT AND CONSTRUCTION

4. Preparation for Operations

ECLP Staff

Over the past year ending June 1979, the ECLP staff grew to 98 members with the addition of 86 people. These staff additions included 52 people for the Process Division, 6 people for the Mechanical Division, 19 people for the Technical Division and 9 people for the Administrative Division. The quarterly buildup rate was consistent with our projections and numbered 10 people during the third quarter of 1978, 12 people during the fourth quarter of 1978, 33 people during the first quarter of 1979 and 31 people during the second quarter of 1979.

Equipment Completion and Turnover

ECLP construction plans call for the completion and turnover of the plant by sections and/or systems in a staged fashion and spanning a period of several months. Process manpower was assigned to identify each section in detail and coordinate the turnover.

The first turnover of equipment was accomplished on schedule on April 6, 1979, with the completion of a portion of the fire water system. This first turnover package included a diesel-engine-driven fire water pump and a portion of the fire water distribution system. After running in the driver and pump and flushing the lines, this system was placed on standby status for operations when needed.

The main electrical substation had been scheduled to be completed and energized in late May. The schedule for energizing the substation slipped because of a delay in making the final hookup to the 138 kv power system. This delay was caused by some problems encountered in obtaining right-of-ways between the main line and the ECLP substation. Energizing the main substation was rescheduled for early July.

The interplant lines between ECLP and Exxon's Refinery/Chemical Plant complex are nearing completion. Coordination activity with the refinery is underway to get the lines in service as they are needed to support ECLP operations.

Training Activities

The preparation of operating manuals for ECLP is well underway. Guidelines for manual preparation and format have been prepared, and in September 1978, six members of the operating staff were engaged essentially full time in this effort. The ECLP operating manuals for each major processing area will be comprised of three sections--a basic information section, a procedures section, and an emergency section. By year-end 1978, work was completed on the first section of all manuals and the preparation

of the process procedures sections were well underway. Also, by year-end, all of the Chief Operators had visited the engineering model at McKee's offices in Cleveland, Ohio, as an aid in manual preparation.

In mid-February 1979, seventeen Process Technicians on loan from Exxon's Baytown Refinery arrived at the ECLP site to begin training. Four weeks of classroom training of the Process Technicians was completed during March 1979. By the end of March, the Chief Operators and Process Technicians jointly developed a program for detailed post training. Actual post training began in April.

Twenty-three new hires, who ultimately would be assigned to ECLP as Process Technicians, reported for work at Exxon's Baytown Refinery in March 1979, and began five weeks of basic training in the Refinery. Several members of the ECLP Process Division staff participated in various phases of their initial training activity. By mid-April the new-hire group completed five weeks of basic refinery training and were assigned to an operating department within the Refinery for four weeks of "hands on" training. This group began onsite ECLP training in mid-May.

Portable buildings (trailers) were set up to serve as classroom training facilities for the 40 Process Technicians and 9 Chief Operators. These facilities were completed and occupied by May 1979, and will be used for training activities for a period of about six months.

The "new hire" group of Process Technicians completed the orientation phase of their training program with the experienced Process Technicians serving as instructors during most of this activity. In preparing and giving this training material, the experienced technicians reinforced their own learning experience.

During May, the use of a process simulator was obtained and training was conducted on the operation of a distillation tower. The Process Technicians and Chief Operators took part in this training activity which was conducted in eight-hour sessions with four to six people in attendance at each session.

A preliminary version of the start-up procedure for the liquefaction and fractionation areas has been developed and is currently under review. Initial efforts addressed leak test procedures, optimization of inerting procedures to minimize utility requirements and identification of additional circuits to permit extended periods of near total recirculation.

#### Occupational Health Program

An environmental health work group was formed, with the task of developing a proposal for an ECLP Occupational Health Program. Elements of the program included areas such as personal hygiene, medical surveillance, and engineering controls. The work group comprised ECLP staff members, as well as representatives of the Medical and Industrial Hygiene Departments of Exxon Company, U.S.A. The program was reviewed and approved by year-end 1978.

Representatives of the National Institute of Occupational Safety and Health (NIOSH) and JRB Associates, Inc., a NIOSH subcontractor, visited Baytown on April 23, 1979 to obtain background and information regarding Exxon's activities on Occupational Health Programs for EDS as they pertain to ECLP and the smaller BARD pilot plants. This information was to aid them in preparing a Criteria Document concerning occupational exposure in coal liquefaction plants. The visit consisted of a review of the ECLP Occupational Health Program and a plant tour of ECLP, followed by a review of the BARD Occupational Health Program and a tour of CLPP, RCLU and the locker/shower facilities at the BARD site. The visitors stated that the nature and content of the discussions met their intent and needs.

Industrial hygiene test equipment selection has been completed and purchase orders will be issued shortly. Some initial testing using this equipment will be done before ECLP start-up.

### Safety

All ECLP staff members attended a four-hour safety workshop. The purpose of the safety workshop was to inform all employees of the several components that comprise the integrated safety program for ECLP, to solicit employee input into some of the program details and to provide a stimulus for safety awareness on the part of each employee. Each workshop session included presentations which described the overall safety program. Organizational responsibility for development of the various parts of the program was also discussed. The workshops also included the assignment of safety-related tasks to employee groups for discussion and recommendation of ideas and solutions which may be incorporated into an overall ECLP safety program.

### Coal Receipt and Preparation

The purchase option was exercised for thirty (30) 100-ton "Rapid Discharge" hopper cars from Ortner Freight Car Company of Cincinnati, Ohio. These cars are scheduled to be manufactured in July 1979. The Traffic Division of Exxon Company, USA's Supply Department agreed to administer ECLP's 30 rail cars during operations. TMC Engineering Services of Houston was selected as ECLP's agent to inspect the cars during their manufacture. In June 1979, representatives from Carter Oil and TMC Engineering Services visited Ortner. The purpose of the meeting was to review the specifications with Ortner, begin inspection activities, review administrative details, and tour the shop facilities.

Proform, Inc. was selected as the successful bidder of 30 rail car covers. A single prototype cover was fabricated during the first quarter 1979 and the remaining 29 covers will be fabricated following approval of the prototype design by ER&E.

A rail car was subleased from the Monterey Coal Company and moved to Proform's St. Paul plant for the prototype cover installation. The rail car identification prefix, EDSX, which will appear on the side of all ECLP cars, was approved by the Association of American Railroads. The prototype cover was installed on the test car at Proform's plant in early April 1979.

The prototype rail car cover system was tested at Proform's plant in Minneapolis during April 1979. The cover was operated satisfactorily for 230 cycles, which is approximately twice the number of cycles expected during 2-1/2-years of operation. Several minor modifications were made as a result of the test and inspection.

On May 15, 1979, representatives from Carter Oil, ER&E, and Proform met at The Carter Oil Company's Monterey No. 1 Mine in Carlinville, Illinois, to inspect the loading of the prototype covered car. It was shown that there were no clearance problems and that coal can be loaded into the car such that the cover will close over the coal heap. The car was followed on its first trip in the unit train and no problems were encountered during the unloading operation.

The prototype rail car test program was successfully completed June 15, 1979. The prototype car made 15 trips (6,000 miles) between the Monterey No. 1 Mine and a power plant during the "over-the-rail" phase of the test program. The cover system was found to be structurally sound at the final inspection of the car. The prototype car has been sent to Proform's plant in Paducah, Kentucky, for dismantling. Once the cover has been removed, it will be transferred to a rail car repair shop to return it to its previous condition before it is returned to Monterey.

The installation of dust suppression spray nozzles and the "third rail" unloading system at the ECLP track unloading hopper required a variance from the Texas Railroad Commission. An application for the variance was filed in September 1978. In November 1978, Carter representatives participated in hearings before the Texas Railroad Commission on the variance. During the first quarter of 1979, notice was received from the Texas Railroad Commission that the application had been approved.

#### Engineering Model

ECLP staff members participated in reviews of the engineering model at A.G. McKee's offices in Cleveland, Ohio. A detailed section-by-section review of piping was initiated in July 1978. Formal model review work was completed with the compressor and coal preparation areas during October 1978, and with the flushing and blowdown and Dowtherm areas during November 1978.

All sections of the engineering model were received onsite and the entire model assembled during May 1979 for use as an aid to both training and construction.

#### General Purpose Building

Bid solicitations were sent out in August 1978, for the General Purpose Building and were received by late September. This building will house the shops and stores facilities, locker rooms, and laboratory. A contract was let for the General Purpose Building and work was started in October 1978. During December 1978, the slab was poured, outer framing

completed, and sheathing started. By the end of the first quarter 1979, the General Purpose Building was 97 percent complete overall. Storehouse personnel occupied their area during March 1979 and turnover was accomplished in April 1979. Utility tie-ins and telephone installation were completed in May 1979.

#### Information Systems

A laboratory specification sheet and floor plan was prepared in mid-April 1979 for release to obtain vendor quotations. Anticipated construction and equipment delivery times are consistent with having a fully operational laboratory in October 1979.

Following bid conditioning, a laboratory furniture supplier was selected to provide furniture for the ECLP Laboratory. Bids were received from five companies who made proposals for seven lines of furniture. Also, two separate proposals were received to complete the Laboratory interior structure by providing air conditioning, partitioning, insulation, and furniture set-up.

A Carter Change Request was approved which will link ECLP with the Exxon Company, USA computer system in the Houston Headquarters Office Building. A Data 100 Corporation time-sharing terminal located in the ECLP Administration Building will provide access to Exxon's IBM 370/168 Computer System. This will allow the data stored on tape by the ECLP data logger to be transferred to the Houston computer for tape copies and storage. The system also allows for direct access to ECLP data by scientists and engineers at the various ER&E locations involved in EDS research and development activities. The linkage also permits ECLP personnel to use Exxon's Technical Computer Programs. Site preparation is in progress and should be complete to accept the system in July 1979. Work will then begin to link the system to the Houston Refinery IBM 370/168.

#### Contract Administration

A service agreement was executed early in 1978 between The Carter Oil Company and Exxon Company, USA's Baytown Refinery. This agreement established a task order system which outlines a procedure for obtaining a variety of services, including utilities for ECLP, from the Refinery. A task order has been executed for the construction of interplant lines. In addition, task order drafts have been prepared which cover the use of loaned manpower from the Refinery and any use of the Refinery warehouse which may become necessary. In all, twelve task orders with Exxon USA's Baytown Refinery have been completed. Five task orders are being processed and 28 remain to be drafted. All task orders affecting ECLP should be completed by year-end 1979.

The I&E Services contract for instrument maintenance and the rail car inspection contract with TMC Engineering Services were both executed in June 1979.

### Emission Abatement Equipment

Engineering data submitted by ECLP to the Texas Air Control Board (TACB) were reviewed by TACB and found to be acceptable. The requirements of Special Provision No. 13 of TACB Permit No. C-6080 were thus satisfied which permitted us to install our emission abatement equipment.

### Mechanical Department Activities

As of the end of June 1979, the ECLP Mechanical Job Plans overall were 98 percent complete and copies of the manuals had been reproduced for the use of the maintenance contractor. Also, editing and revision of the safety manuals is complete and the Carter Mechanical Procedures Manual is 85 percent complete. The ECLP Mechanical Job Plans include items such as vessel breakdown blinding lists, lifting requirements, a detailed tool list, documentation of job procedures and gasket lists. Contract maintenance manuals and procedures are being printed and safety valve testing procedures have been finalized.

The equipment required for calibration and loop checking of instruments was placed on order and calibration began in February 1979.

The radioactive material handling license from the Texas Department of Health was received. The radiation officer and his backup attended certification school in January 1979. There are regulatory requirements which stem from the use of radioactive material in certain types of level instruments.

Supervisors from both the general maintenance contractor and the instrument maintenance contractor arrived on site during January 1979.

ECLP personnel participated in the checkout of Foxboro Spec 200 instrument racks at the factory. The control house check began during the first week of June 1979.

## 5. Operations

Reporting under this category is to start upon start of operations.

## 6. ECLP Environmental Assessment

This task has been completed and no further reports will be made.

## ECLP TERMINATION OF OPERATIONS

### 1. Dismantling

Planning for ECLP disposition is to start January 1, 1982.

FLEXICOKING PROTOTYPE PROJECT MANAGEMENT,  
DESIGN, ENGINEERING AND PROCUREMENT

1. Inspection

Inspection of the FLEXICOKING Prototype Unit for the Class IV estimate was completed. Internally, the unit (which had been nitrogen-blanketed) was in very good condition. The only significant repairs identified will be (1) replacement of the stainless steel cone of the heater vessel, which is extensively cracked, (2) retubing of one exchanger, and (3) replacement of trays in the amine scrubber. Externally, small piping was heavily corroded and insulation has deteriorated. To reduce further corrosion, all wet and deteriorated insulation will be removed from piping and vessels.

2. Process Design

Onsite Design

Work on the Onsite Design Specifications started in early February 1979. By the end of June 1979, these specifications were approximately 80 percent complete. Design developments during May and June resulted in the deletion of some pumps, drums and condensers and the addition of onsite coker gas treating facilities. A duty specification is being prepared for these facilities. Additionally, tertiary fines from the heater overhead have been combined with the stripped wet slurry disposal steam, thus eliminating a potential housekeeping and environmental problem at the unit.

A preliminary issue of the design specifications will be released for comments in July, with final issue targeted for early September 1979.

Offsite Design

In the offsite area, design basis work is on schedule. Environmental health issues associated with storage and handling of the ECLP vacuum bottoms were resolved and a dust collection system and Occupational Health Program requirements were specified and are consistent with plans approved for ECLP.

During April 1979, the Offsite Design Basis Memorandum was completed. The offsite equipment layout was reviewed and approved for compliance with safe spacing standards and fire protection coverage.

In June 1979, the Offsite Facilities Definition document was issued. This document is being used as the basis for the Class IV cost estimates. Comments have been reviewed and will be included in the appropriate design



specifications. Also in June, initial drafts of the specifications for the vacuum bottoms storage facilities at the ECLP site and the specifications for the air compression facilities were issued. Overall, the offsite design is approximately 15 percent complete.

### 3. Environmental Assessment

Data on estimated emissions at both the FLEXICOKING Prototype and the vacuum bottoms storage facility at ECLP were assimilated for preparation of construction permit applications. By May 1979, all necessary data were available. During June 1979, applications for construction permits were made to the Texas Air Control Board (TACB). The New Source Environmental Questionnaire will be submitted to the EPA during the first half of July.

### 4. Project Management

Bids were solicited from six contractors covering the detailed design, procurement, and construction of the FLEXICOKING Prototype Unit facilities. Commercial terms will be on a cost-plus-fixed fee basis. This approach was selected in order to keep the overall implementation schedule as short as possible. The schedule for contract award is July 1, 1979, which is consistent with a mechanical completion of February 1, 1981.

By the end of May 1979, evaluation of bids from the contractors was complete and contract award work was proceeding on schedule. Work on the Class IV cost estimate was proceeding as planned, with the expected completion of this work holding the early July target.

A division of responsibility agreement was reached with Exxon Company, USA's Baytown Refinery whereby the Refinery will be responsible for design, procurement, and construction of those offsite facilities which require installation on existing Refinery pipe racks and/or in existing Refinery operating units outside the FLEXICOKING unit. Overall management and coordination will remain the responsibility of The Carter Oil Company.

### 5. Engineering and Procurement

Reporting in this category is to start after contract award.

A P P E N D I C E S

## Appendix A

### RCLU TABULATED YIELD PERIOD DATA

This appendix provides tabulated yield period data for the 50 pound-per day Recycle Coal Liquefaction Unit (RCLU-1) discussed in Laboratory Process Research and Development, Section 1 of this report. Tables A-1 and A-2 contain operating conditions, material balances, and product yields for RCLU-1 yield periods 603-647. Analyses of feed and product streams for these yield periods are given in Tables A-3 through A-5. The liquefaction reactor solids inspections for the Wandoan coal screening study are presented in Table A-6. Table A-7 contains the average liquefaction yields for the Wyodak bottoms recycle study. Also included in Table A-8 are the liquefaction-only yields for Burning Star No. 2 coal which were incomplete last quarter. The overall Burning Star yields were presented in Appendix Table A-2 in the January-March, 1979, EDS Quarterly Technical Progress Report [FE-2893-29].

TABLE A-1

## RCLU YIELD PERIOD OPERATION SUMMARY

RCLU UNIT NUMBER	1	1	1	1	1	1	1	1	1
YIELD PERIOD NUMBER	603	604	605	606	607	608	609	610	611
YIELD PERIOD LENGTH	24	24	24	24	24	24	24	24	24
TYPE OF COAL USED	--WYOMING W/CLPP A-1 BTMS--			-----WYOMING-----			-----WYOMING W/CLPP A-1 BTMS		

## LIQUEFACTION

INLET PRESSURE, PSIG	1542.	1543.	1543.	1533.	1533.	1532.	1535.	1534.	1535.
AVERAGE TEMPERATURE, °F	842.	841.	841.	839.	839.	839.	838.	838.	837.
SPACE VELOCITY, V/HR/V	0.99	1.00	1.01	1.17	1.04	1.09	2.58	2.53	2.49
SPECIFIC FEED RATE, LBS/HR/FT <sup>3</sup> *	33.	33.	32.	28.	25.	27.	64.	82.	80.
TREAT GAS RATE, LBS H <sub>2</sub> /100LBS DC	4.04	4.06	4.31	3.83	4.28	3.98	4.19	5.02	4.86
H <sub>2</sub> IN TREAT GAS, MOLE %	100.	100.	100.	100.	100.	100.	100.	100.	100.
SLURRY CONC. LBS FEED/LBS SLURRY*	0.48	0.48	0.46	0.35	0.35	0.36	0.36	0.47	0.47
DRY COAL FEED RATE, LBS/HR	2.63	2.63	2.48	3.32	3.00	3.22	3.09	2.57	2.51
LIQUEFACTION BOTTOMS FEED RATE, LBS/HR	1.37	1.38	1.40					1.51	1.49
SOLVENT QUALITY INDEX	4.28	4.29	4.55	4.69	4.82	4.67	4.65	4.38	4.42

## HYDROGENATION

OUTLET PRESSURE, PSIG	1521.	1521.	1520.	1510.	1510.	1507.	1507.	1509.	1509.
AVERAGE TEMPERATURE, °F-RV	121.	121.	121.	130.	130.	129.	129.	124.	124.
SPACE VELOCITY, V/HR/V, % RV	220.	216.	222.	314.	270.	279.	275.	212.	208.
TREAT GAS RATE, SPECIFIC/100LBS FEED, %RV	250.	261.	255.	186.	215.	209.	213.	275.	278.

## OVERALL MATERIAL BALANCE

1.5. HYDROGENATION LBS INPUT	100.12	100.13	100.34	98.26	99.09	98.73	101.94	101.47	100.18
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\*FEED = DRY COAL + LIQUEFACTION BOTTOMS

TABLE A-1 (Continued)

## RCLU YIELD PERIOD OPERATION SUMMARY

RCLD UNIT NUMBER	1	1	1	1	1	1	1	1	1
YIELD PERIOD NUMBER	612	613	614	615	616	617	618	619	620
YIELD PERIOD LENGTH	24	17	24	24	24	24	24	24	24
TYPE OF COAL USED	[WYOMING W/CLPP A-1 BTMS]-----WYOMING-----]-----WYOMING W/CLPP A-1 BTMS-----]								
LIQUEFACTION									
INLET PRESSURE, PSIG	1538.	1543.	1581.	1556.	1602.	1529.	1530.	1543.	1549.
AVERAGE TEMPERATURE, °F	837.	836.	840.	840.	839.	839.	840.	835.	834.
SPACE VELOCITY, W/HR/V	2.49	2.56	2.62	2.47	2.54	1.06	1.03	0.99	1.03
SPECIFIC FEED RATE, LBS/HR FT3*	80.	83.0	69.	64.	67.	28.	26.	26.	27.
TREAT GAS RATE, LBS H2/100 LB OC	4.26	4.06	3.85	4.19	4.04	5.12	5.40	5.40	5.22
H2 IN TREAT GAS, MOLE %	100.	100.	100.	100.	100.	100.	100.	100.	100.
SLURRY CONC. LBS FEED/LBS SLURRY*	0.47	0.47	0.38	0.38	0.38	0.38	0.38	0.38	0.38
DRY COAL FEED RATE, LBS/HR	2.51	2.61	3.32	3.07	3.21	2.22	2.10	2.09	2.18
LIQUEFACTION BOTTOMS FEED RATE, LBS/HR	1.49	1.53				1.12	1.05	1.05	1.09
SOLVENT QUALITY INDEX	4.30	4.25	4.27	4.24	4.14	7.08	7.14	6.49	6.68
HYDROGENATION									
OUTLET PRESSURE, PSIG	1511.	1516.	1505.	1512.	1513.	1521.	1521.	1534.	1541.
AVERAGE TEMPERATURE, °F-RV	121.	123.	122.	123.	123.	124.	124.	119.	117.
SPACE VELOCITY, W/4V/W, % RV	208.	212.	258.	241.	244.	262.	256.	243.	254.
TREAT GAS RATE, SCFH2/100 LB OC	272.	266.	224.	241.	240.	222.	225.	237.	228.
OVERALL MATERIAL BALANCE									
LBS OUTPUT/100 LBS INPUT	98.31	100.69	96.16	95.24	99.78	100.94	102.20	99.30	98.52

\*FEED = DRY COAL + LIQUEFACTION BOTTOMS

TABLE A-1 (Continued)

RCLU YIELD PERIOD OPERATION SUMMARY

RCLU UNIT NUMBER	1	1	1	1	1	1	1	1	1
YIELD PERIOD NUMBER	621	622	623	624	625	626	627	628	629
YIELD PERIOD LENGTH	21	24	24	17	24	24	24	24	24
TYPE OF COAL USED	--CLPP A-1 BTMS--			WYO W/CLPP A-1 BTMS			-----WANDOAN-----		
LIQUEFACTION									
INLET PRESSURE, PSIG	1541.	1547.	1525.	1531.	1515.	1512.	1514.	1514.	1521.
AVERAGE TEMPERATURE, °F	843.	843.	840.	840.	840.	840.	841.	841.	803.
SPACE VELOCITY, V/HR/V	0.98	1.10	2.23	2.21	1.59	1.56	1.52	1.49	1.53
SPECIFIC FEED RATE, LBS/HR/FT3*	23.	27.	59.	59.	42.	41.	40.	39.	40.
TREAT GAS RATE, LBS/100 LBS DC	4.62	3.98	6.07	6.06	3.84	3.96	4.10	4.18	4.02
H2 IN TREAT GAS, MOLE %	100.	100.	100.	100.	100.	100.	100.	100.	100.
SLURRY CONC. LBS FEED/LBS SLURRY*	0.35	0.35	0.38	0.38	0.38	0.38	0.38	0.38	0.38
DRY COAL FEED RATE, LBS/HR	2.76	3.20	1.88	1.87	3.35	3.26	3.20	3.13	3.22
LIQUEFACTION BOTTOMS FEED RATE, LBS/HR			0.94	0.94					
SOLVENT QUALITY INDEX	5.92	5.56	7.41	7.09	4.49	4.43	4.27	4.28	3.95
HYDROGENATION									
OUTLET PRESSURE, PSIG	1531.	1534.	1526.	1530.	1510.	1508.	1509.	1507.	1510.
AVERAGE TEMPERATURE, °F-RV	125.	125.	123.	123.	124.	124.	125.	124.	118.
SPACE VELOCITY, W/HR/W, %RV	210.	234.	214.	214.	262.	257.	251.	247.	240.
TREAT GAS RATE, SCFH2/98 LBS DC FEED, %RV	280.	249.	272.	270.	224.	231.	239.	242.	246.
OVERALL MATERIAL BALANCE									
LBS OUTPUT/100 LBS INPUT	100.11	100.70	100.99	100.41	100.02	97.93	100.65	99.18	98.16

\*FEED = DRY COAL + LIQUEFACTION BOTTOMS

TABLE A-1 (Continued)

RCU YIELD PERIOD OPERATION SUMMARY

RCU UNIT NUMBER	1	1	1	1	1	1	1	1
YIELD PERIOD NUMBER	630	631	632	633	634	635	636	637
YIELD PERIOD LENGTH	24	24	24	24	24	24	24	24
TYPE OF COAL USED	WANDUAN							

LIQUEFACTION

INLET PRESSURE, PSIG	1530.	1526.	1543.	1545.	1549.	1551.	1549.	1550.	1526.
AVERAGE TEMPERATURE, F	803.	803.	841.	841.	841.	804.	804.	805.	842.
SPACE VELOCITY, V/HR/V	1.49	1.49	0.62	0.59	0.68	0.66	0.67	0.54	2.68
SPECIFIC COAL FEED RATE, LBS/HR/FT <sup>3</sup>	39.	39.	16.	16.	18.	18.	18.	14.	70.
TREAT GAS RATE, LBS H <sub>2</sub> /100LH DC	4.05	4.14	3.64	4.15	3.59	3.65	3.65	4.56	3.91
H <sub>2</sub> IN TREAT GAS, MOLE %	100.	100.	100.	100.	100.	100.	100.	100.	100.
SLURRY CONC. LBS DC/LHS SLURRY	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38
DRY COAL FEED RATE, LBS/HR	3.15	3.14	3.28	3.12	3.59	3.51	3.52	2.83	3.35
SOLVENT QUALITY INDEX	3.97	3.71	4.48	4.48	4.43	4.35	4.30	4.29	4.24

HYDROGENATION

OUTLET PRESSURE, PSIG	1518.	1514.	1524.	1525.	1527.	1536.	1535.	1536.	1531.
AVERAGE TEMPERATURE, °F-RV	119.	118.	134.	133.	134.	123.	124.	124.	125.
SPACE VELOCITY, W/HR/W, XRV	238.	235.	264.	250.	280.	269.	273.	225.	251.
TREAT GAS RATE, SCFH <sub>2</sub> /BBL OIL FEED, XRV	246.	252.	224.	237.	211.	217.	215.	262.	238.

OVERALL MATERIAL BALANCE

LBS OUTPUT/100 LBS INPUT	98.05	96.96	101.10	100.02	98.46	97.01	98.09	102.42	100.77
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TABLE A-1 (Continued)  
RCLU YIELD PERIOD OPERATION SUMMARY

PCLD UNIT NUMBER	1	1	1	1	1	1	1	1	1
YIELD PERIOD NUMBER	639	640	641	642	643	644	645	646	647
YIELD PERIOD LENGTH	24	24	24	24	14	24	24	24	24
TYPE OF COAL USED	WANDAN								
LIQUEFACTION									
INLET PRESSURE, PSIG	1533.	1530.	1524.	1535.	2504.	2508.	2515.	2513.	2509.
AVERAGE TEMPERATURE, °F	841.	828.	875.	874.	840.	839.	834.	833.	834.
SPACE VELOCITY, V/HR/V	2.53	2.52	2.60	2.36	1.55	1.65	1.69	1.54	1.50
SPECIFIC COAL FEED RATE, LBS/HR/FT <sup>3</sup>	66.	66.	68.	62.	40.	43.	44.	40.	39.
TREAT GAS RATE, LBS H <sub>2</sub> /100 LBS DC	4.09	4.11	3.95	4.30	3.90	3.65	3.60	3.97	4.09
H <sub>2</sub> IN TREAT GAS, MOLE %	100.	100.	100.	100.	100.	100.	100.	100.	100.
SLURRY CONC. LBS DC/LBS SLURRY	0.30	0.36	0.38	0.38	0.38	0.38	0.38	0.38	0.38
DRY COAL FEED RATE, LBS/HR	3.16	3.18	3.28	2.97	3.23	3.47	3.55	3.22	3.15
SOLVENT QUALITY INDEX	4.36	4.21	3.92	4.15	4.90	4.96	4.91	4.51	4.29
HYDROGENATION									
OUTLET PRESSURE, PSIG	1535.	1525.	1520.	1528.	2463.	2487.	2492.	2492.	2488.
AVERAGE TEMPERATURE, °F-RV	125.	123.	124.	125.	132.	128.	132.	131.	130.
SPACE VELOCITY, V/HR/V, XRV	240.	231.	242.	223.	242.	257.	262.	244.	237.
TREAT GAS RATE, SCFH/100 LBS DC, XRV	245.	244.	231.	247.	225.	212.	211.	227.	235.
OVERALL MATERIAL BALANCE									
LBS OUTPUT/100 LBS INPUT	98.37	97.90	100.26	98.01	102.86	98.06	98.52	101.18	97.55



TABLE A-2  
RCLU YIELD PERIOD SUMMARY OF YIELDS

RCLU Unit Number	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Yield Period Number	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617
<u>Overall Yields,</u>															
<u>Wt % on Dry Coal</u>															
Hydrogen	-2.81	-2.81	-2.72	-3.97	-3.87	-3.94	-2.79	-2.54	-2.87	-2.48	-2.64	-3.04	-3.18	-2.80	-3.99
Water	8.62	8.53	8.08	13.97	12.30	12.12	11.80	9.51	8.91	8.60	9.78	13.42	12.44	10.77	9.77
Carbon Oxides	4.87	5.25	5.67	7.43	8.57	8.25	8.30	5.31	4.99	5.04	5.05	7.21	6.91	6.66	4.09
Hydrogen Sulfide	0.25	0.35	0.33	0.65	0.51	0.47	0.55	0.32	0.38	0.37	0.42	0.44	0.37	0.73	0.45
Ammonia	0.35	0.41	0.33	0.41	0.45	0.46	0.31	0.25	0.31	0.31	0.29	0.37	0.45	0.28	0.40
Cl-C <sub>3</sub> Gas	8.24	8.17	8.08	8.63	10.14	9.61	5.76	5.38	5.61	5.61	5.66	6.27	6.53	5.81	7.90
C <sub>4</sub> -400°F	14.77	13.39	13.87	19.82	19.83	19.77	17.66	12.76	14.49	13.12	13.11	14.28	17.09	16.32	21.58
400-700°F	2.24	3.64	3.19	4.47	2.82	2.34	0.86	3.69	2.57	2.42	2.12	3.11	3.45	5.14	3.74
700-1000°F	1.01	2.31	1.51	4.97	4.93	5.25	5.40	2.29	2.45	1.31	2.72	6.27	4.71	5.60	1.20
1000°F+	62.44	60.77	61.68	43.81	44.33	45.67	52.15	63.02	63.16	65.70	63.48	51.68	51.22	51.50	54.85
C <sub>4</sub> -1000°F	18.02	19.34	18.57	29.26	27.58	27.37	23.93	18.74	19.50	16.85	17.95	23.65	25.25	27.07	26.52
<u>Liquefaction Yields,</u>															
<u>Wt % on Dry Coal</u>															
Hydrogen	-1.11	-1.00	-1.16	-1.89	-1.57	-1.58	-0.81	-0.70	-1.30*	-0.51	-0.89*	-1.62	-1.26	-1.31	-1.61
Water	7.31	6.96	6.69	12.20	10.07	10.00	9.42	8.41	7.78	6.86	8.71	11.04	10.11	9.04	7.63
Carbon Oxides	4.87	5.25	5.67	7.43	8.57	8.25	8.30	5.31	4.99	5.04	5.05	7.21	6.91	6.66	4.09
Hydrogen Sulfide	0.16	0.22	0.23	0.50	0.44	0.32	0.41	0.26	0.31	0.30	0.34	0.23	0.20	0.49	0.18
Ammonia	0.01	0.05	0.00	0.11	0.14	0.14	0.08	0.02	0.09	0.05	0.08	0.16	0.23	0.14	0.10
Cl-C <sub>3</sub> Gas	8.24	8.17	8.08	8.63	10.14	9.61	5.76	5.38	5.61	5.61	5.66	6.27	6.53	5.81	7.90
C <sub>4</sub> -1000°F	18.07	19.59	18.81	29.20	27.90	27.60	24.70	18.29	19.36	16.95	17.58	25.02	26.05	27.68	26.86
1000°F+	62.44	60.77	61.68	43.81	44.33	45.67	52.15	63.02	63.16	65.70	63.48	51.68	51.22	51.50	54.85

\*Value for hydrogen is questionable.

TABLE A-2 (Continued)  
RCLU YIELD PERIOD SUMMARY OF YIELDS

RCLU Unit Number Yield Period Number	1 618	1 619	1 620	1 621	1 622	1 623	1 624	1 625	1 626	1 627	1 628	1 629	1 630	1 631	1 632
<u>Overall Yields,</u> <u>Wt % on Dry Coal</u>															
Hydrogen	-4.13	-3.85	-3.91	-2.73	-2.230	-3.02	-3.00	-3.17	-2.96	-3.23	-3.17	-2.35	-2.56	-2.42	-4.10
Water	10.78	9.69	10.37	2.37	2.75	10.12	9.19	10.99	9.10	12.11	10.01	10.04	11.68	10.26	12.53
Carbon Oxides	3.89	4.84	4.54	0.63	0.37	4.37	4.50	3.20	3.13	3.18	3.32	2.51	2.49	2.47	3.28
Hydrogen Sulfide	0.36	0.40	0.42	0.04	0.07	0.44	0.41	0.18	0.12	0.12	0.19	0.15	0.17	0.18	0.11
Ammonia	0.42	0.40	0.44	0.34	0.35	0.33	0.35	0.26	0.26	0.35	0.32	0.17	0.19	0.16	0.33
C1-C3 Gas	8.01	8.05	8.23	6.60	5.08	5.54	5.38	6.81	7.35	7.08	7.32	3.51	3.36	3.57	11.88
C4-400°F	23.91	21.41	20.90	13.43	10.74	17.22	18.63	18.58	18.61	18.52	18.18	12.47	12.93	11.57	25.64
400-700°F	1.59	1.37	1.72	-1.96	2.58	5.36	4.15	6.70	5.95	5.97	6.34	5.72	4.56	7.64	-4.12
700-1000°F	1.71	2.32	2.47	1.32	1.23	3.47	0.98	3.44	3.71	2.53	2.28	3.65	5.14	3.41	3.64
1000°F+	53.47	55.37	54.83	79.98	79.14	56.17	59.40	53.01	54.71	53.39	55.21	64.14	62.05	63.17	50.81
C4-1000°F	27.21	25.10	25.09	12.79	14.54	26.05	23.76	28.72	28.28	27.01	26.80	21.83	22.63	22.62	25.16
<u>Liquefaction Yields,</u> <u>Wt % on Dry Coal</u>															
Hydrogen	-1.69	-1.40	-1.81	-1.01	-0.65	-0.67	-0.32*	-0.97	-0.35*	-0.86	-1.09	-0.59	-0.44	-0.90*	-2.07
Water	9.07	7.91	8.53	1.75	1.68	8.60	6.99	8.88	7.13	9.97	7.94	8.79	9.56	8.48	11.05
Carbon Oxides	3.89	4.84	4.54	0.63	0.37	4.37	4.50	3.20	3.13	3.18	3.32	2.51	2.49	2.47	3.28
Hydrogen Sulfide	0.25	0.33	0.33	0.04	0.02	0.36	0.32	0.12	0.08	0.08	0.15	0.11	0.10	0.14	0.07
Ammonia	0.17	0.15	0.20	0.16	0.09	0.08	0.10	0.01	0.01	0.09	0.07	0.01	0.01	0.01	0.06
C1-C3 Gas	8.01	8.05	8.23	6.60	5.08	5.54	5.38	6.81	7.35	7.08	7.32	3.51	3.36	3.57	11.88
C4-1000°F	26.83	24.75	25.15	11.87	14.27	25.55	23.63	28.92	27.94	27.08	27.08	21.52	22.88	23.07	24.92
1000°F+	53.47	55.37	54.83	79.98	79.14	56.17	59.40	53.01	54.71	53.39	55.21	64.14	62.05	63.17	50.81

\*Value for hydrogen is questionable.

TABLE A-2 (Continued)

RCLU YIELD PERIOD SUMMARY OF YIELDS

RCLU Unit Number Yield Period Number	633	634	635	636	637	638	639	640**	641	642	643	644	645	646	647
<u>Overall Yields,</u> <u>Wt % on Dry Coal</u>															
Hydrogen	-4.26	-3.95	-2.95	-2.84	-3.01	-2.48	-2.63	-2.92	-4.12	-3.78	-3.42	-3.14	-3.16	-3.47	-3.43
Water	12.49	12.74	11.69	9.25	10.66	9.89	10.73	12.62	13.07	12.34	12.00	12.70	9.45	12.43	10.89
Carbon Oxides	3.40	3.16	2.77	2.98	3.16	3.18	3.26	2.96	3.92	3.52	2.43	1.95	2.32	2.46	2.31
Hydrogen Sulfide	0.19	0.14	0.15	0.15	0.22	0.10	0.17	0.10	0.17	0.13	0.19	0.17	0.16	0.16	0.20
Ammonia	0.38	0.29	0.26	0.25	0.17	0.15	0.16	0.20	0.27	0.33	0.28	0.27	0.29	0.40	0.33
C1-C3 Gas	13.24	11.26	5.54	5.81	7.55	5.01	5.34	5.89	11.46	11.21	6.99	5.22	6.50	6.66	6.03
C4-400°F	24.47	24.24	17.02	15.81	16.71	15.47	14.99	14.68	21.98	20.46	16.05	17.82	19.95	19.88	20.25
400-700°F	-1.97	-2.90	3.04	7.69	5.46	1.13	2.20	5.17	-2.61	-0.48	8.82	7.55	5.72	3.96	6.69
700-1000°F	1.82	3.70	4.57	4.27	3.12	5.56	4.73	4.51	2.15	3.98	3.94	5.43	5.92	6.48	3.78
1000°F+	50.23	51.33	57.93	56.62	55.96	61.98	61.04	56.78	53.72	52.29	52.70	52.04	52.85	51.06	52.95
C4-1000°F	24.33	25.03	24.61	27.77	25.30	22.17	21.92	24.37	21.51	23.96	28.82	30.79	31.58	30.31	30.72
<u>Liquefaction Yields,</u> <u>Wt % on Dry Coal</u>															
Hydrogen	-1.70	-1.51	-1.06	-0.74	-0.54	-0.70	-1.05	-1.16	-2.04	-1.47	-0.90	-1.32	-1.16	-1.39	-1.34
Water	10.17	11.14	10.87	7.42	8.57	7.97	8.67	10.70	11.53	10.41	9.58	10.91	7.15	10.20	8.65
Carbon Oxides	3.40	3.16	2.77	2.98	3.16	3.18	3.26	2.96	3.92	3.52	2.43	1.95	2.32	2.46	2.31
Hydrogen Sulfide	0.15	0.11	0.10	0.11	0.18	0.07	0.13	0.06	0.13	0.12	0.15	0.14	0.12	0.13	0.17
Ammonia	0.13	0.02	0.08	0.01	0.00	0.00	0.01	0.01	0.01	0.06	0.00	0.01	0.02	0.11	0.06
C1-C3 Gas	13.24	11.26	5.54	5.81	7.55	5.01	5.34	5.89	11.46	11.21	6.99	5.22	6.50	6.66	6.03
C4-1000°F	24.38	24.50	23.77	27.78	25.11	22.49	22.60	24.76	21.28	23.85	29.05	31.06	32.21	30.78	31.18
1000°F+	50.23	51.33	57.93	56.62	55.96	61.98	61.04	56.78	53.72	52.29	52.70	52.04	52.85	51.06	52.95

\*\*Questionable elemental balance; data not used in reported averages.

TABLE A-3

**RCLD YIELD PERIOD ANALYTICAL DATA SUMMARY**  
**FEED COAL, BOTTOMS FEED, RESIDUAL BOTTOMS, AND SOLVENT INSPECTIONS**

REF ID UNIT NUMBER YIELD PERIOD NUMBER	1 603	1 604	1 605	1 606	1 607	1 608	1 609	1 610	1 611
<b>FEED COAL ANALYSIS</b>									
<b>ELEMENTAL ANALYSIS, DRY WT %</b>									
CARBON	67.01	66.98	67.98	67.23	67.21	67.96	66.70	67.36	66.80
HYDROGEN	4.99	4.90	4.99	4.87	4.89	4.83	5.01	5.02	4.65
NITROGEN	0.84	0.87	0.84	0.82	0.83	0.82	0.85	0.84	0.86
SULFUR (TOTAL)	0.76	0.70	0.69	0.78	0.68	0.66	0.76	0.72	0.83
SULFATE	0.05	0.04	0.05	0.04	0.04	0.03	0.06	0.04	0.05
PYRITIC	0.15	0.17	0.11	0.17	0.18	0.12	0.17	0.15	0.18
ASH	8.88	9.26	8.39	8.66	8.91	7.87	9.07	8.73	9.05
ASH (SO3) FREE	7.31	7.68	6.83	7.11	7.32	6.35	7.42	7.20	7.52
OXYGEN	22.91	21.85	22.07	22.64	22.25	21.96	23.03	25.93	22.34
MOISTURE	0.19	1.05	7.86	10.12	8.69	6.43	5.38	4.32	5.17
<b>BOTTOMS FEED ANALYSIS</b>									
<b>ELEMENTAL ANALYSIS, DRY WT %</b>									
CARBON	75.93	76.22	75.86					74.25	73.86
HYDROGEN	4.16	4.15	4.14					4.60	4.67
NITROGEN	1.10	1.09	1.06					1.02	1.06
SULFUR	0.34	0.33	0.37					0.54	0.57
ASH	14.62	14.66	14.60					15.01	14.85
ASH (SO3) FREE	13.73	13.79	13.73					13.57	13.41
OXYGEN	9.45	10.05	10.43					11.81	12.33
<b>RESIDUAL BOTTOMS, A-1</b>									
<b>ELEMENTAL ANALYSIS, DRY WT %</b>									
CARBON	74.63	75.74	75.52	76.64	75.05	76.12	76.23	74.43	73.92
HYDROGEN	4.01	4.05	3.98	4.53	4.20	4.29	4.56	4.37	4.46
NITROGEN	0.98	0.96	1.00	0.98	0.92	0.97	1.02	1.02	1.01
SULFUR	0.59	0.40	0.47	0.35	0.41	0.42	0.41	0.53	0.57
ASH	15.62	15.97	15.81	13.46	15.54	14.53	13.61	15.23	15.40
ASH (SO3) FREE	14.61	14.92	14.87	12.58	14.58	13.55	12.61	13.91	14.11
OXYGEN	11.38	10.45	10.58	9.96	10.20	10.00	11.18	11.07	11.03
<b>DISTILLATION</b>									
1000°F-	7.00	7.96	7.76	9.40	9.50	10.40	9.30	11.20	10.60
ND IN 1000°F-	0.51	0.56	0.56						
SULFUR IN 1000°F-	0.06	0.06	0.06	0.07	0.05	0.06	0.08	0.08	0.07
<b>SOLVENT, A-2</b>									
<b>ELEMENTAL ANALYSIS, DRY WT %</b>									
CARBON	80.09	89.95	89.74	89.95	89.90	89.81	89.87	89.82	89.53
HYDROGEN	9.44	9.51	9.55	9.49	9.62	9.71	9.55	9.77	9.90
NITROGEN	0.07	0.06	0.06	0.05	0.04	0.04	0.05	0.05	0.04
SULFUR	0.002	0.001	0.001	0.002	0.001	0.001	0.002	0.002	0.006
OXYGEN	6.40	0.48	0.65	0.51	0.44	0.44	0.53	0.36	0.53
DENSITY @ 70°F, SOLVENT, % OF RV	211.	209.	207.	208.	217.	216.	217.	207.	207.
<b>DISTILLATION</b>									
5 Wt % OFF, F	399.7	398.2	399.2	398.6	396.6	394.8	393.7	394.1	397.5
15 Wt % OFF, F	411.6	409.0	410.5	409.6	407.7	404.3	403.5	420.8	407.4
25 Wt % OFF, F	441.4	434.9	437.6	437.2	433.6	425.2	429.6	452.6	436.4
50 Wt % OFF, F	519.9	506.7	511.9	510.6	504.3	489.4	501.0	531.1	511.4
75 Wt % OFF, F	603.1	592.2	602.6	594.8	589.9	573.4	583.5	606.2	595.2
95 Wt % OFF, F	768.9	735.6	775.9	745.3	746.4	697.9	711.7	771.4	753.4
400°F-, WT %	5.58	7.59	6.40	7.16	8.77	10.76	12.32	8.74	8.44
400-700°F-, WT %	84.53	84.63	83.05	84.66	83.16	84.46	81.90	81.24	82.81
700°F+, WT %	9.89	7.78	10.55	8.18	8.07	4.78	5.78	10.02	8.75
SPECIFIC GRAVITY @ 60°F/60°F	0.987	0.982	0.972	0.980	0.975	0.983	0.977	0.977	0.953

TABLE A-3 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
FEED COAL, BOTTOMS FEED, RESIDUAL BOTTOMS, AND SOLVENT INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 612	1 613	1 614	1 615	1 616	1 617	1 618	1 619	1 620
<u>FEED COAL ANALYSIS</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	67.16	66.64	67.18	67.27	67.23	66.98	67.23	66.78	67.02
HYDROGEN	5.01	4.90	4.89	4.93	5.05	4.98	5.07	4.99	5.03
NITROGEN	0.86	0.83	0.81	0.94	0.79	0.80	0.83	0.87	0.82
SULFUR (TOTAL)	0.78	7.79	0.60	0.60	1.03	0.85	0.75	0.79	0.79
SULFATE	0.05	0.05	0.04	0.04	0.07	0.05	0.04	0.04	0.04
PYRITIC	0.19	0.19	0.19	0.18	0.44	0.37	0.31	0.32	0.27
ASH	9.37	9.16	9.04	9.12	9.26	8.94	8.73	8.77	8.46
ASH (SO <sub>3</sub> ) FREE	7.72	7.74	7.45	7.57	7.25	7.06	7.00	6.99	6.84
OXYGEN	22.51	23.20	24.67	22.68	21.36	22.20	22.39	21.57	21.18
MOISTURE	5.11	3.84	0.00	0.00	0.19	0.52	0.35	0.12	0.00
<u>BOTTOMS FEED ANALYSIS</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	74.19	73.94				75.58	75.12	75.55	75.46
HYDROGEN	4.68	4.48				4.08	4.06	4.12	4.09
NITROGEN	1.11	1.07				1.03	1.09	1.08	1.09
SULFUR	0.53	0.63				0.38	0.33	0.31	0.35
ASH	15.02	14.91				15.02	14.97	15.06	14.83
ASH (SO <sub>3</sub> ) FREE	13.57	13.45				14.15	14.07	14.18	13.93
OXYGEN	12.60	12.80				10.88	10.75	11.17	11.07
<u>RESIDUAL BOTTOMS, A-1</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	74.61	74.71	76.43	76.54	75.46	73.03	73.52	73.93	74.20
HYDROGEN	4.45	4.36	4.69	4.69	4.68	4.13	4.15	4.15	4.22
NITROGEN	1.01	0.99	0.65	0.99	0.98	0.97	1.03	1.05	0.96
SULFUR	0.51	0.48	0.40	0.44	0.61	0.49	0.50	0.44	0.45
ASH	15.41	15.47	12.67	13.26	14.45	17.57	17.36	17.33	16.35
ASH (SO <sub>3</sub> ) FREE	14.10	14.12	11.70	13.12	12.72	16.19	16.12	16.12	15.31
OXYGEN	11.46	10.73	10.41	11.37	12.18	12.69	11.68	11.03	10.24
DISTILLATION									
100°F--	9.80	10.30	11.30	10.10	11.00	5.30	8.00	8.20	9.20
N <sub>2</sub> IN 100°F--									
SULFUR IN 100°F--	0.07	0.09	0.11	0.08	0.08	0.05	0.02	0.05	0.06
<u>SOLVENT, A-2</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	89.53	89.45	89.93	89.67	89.57	89.63	89.53	89.40	89.44
HYDROGEN	9.95	10.08	9.56	9.75	9.86	10.11	10.27	10.48	10.45
NITROGEN	0.03	0.03	0.06	0.04	0.04	0.03	0.02	0.02	0.02
SULFUR	0.000	0.001	0.003	0.001	0.002	0.000	0.000	0.000	0.0
OXYGEN	0.45	0.43	0.45	0.54	0.53	0.23	0.18	0.10	0.09
DENSITY @ 60°F, SOLVENT, % OF RV	201.	201.	211.	209.	204.	232.	234.	213.	220.
DISTILLATION									
5 WT% OFF, °F	378.8	395.8	393.7	379.4	396.7	393.7	393.2	391.8	391.8
15 WT% OFF, °F	389.5	406.7	403.9	388.4	406.3	403.5	402.9	402.1	402.1
25 WT% OFF, °F	416.6	435.0	433.1	416.9	435.7	434.0	431.7	430.4	426.4
50 WT% OFF, °F	493.8	507.5	508.8	491.8	509.8	499.8	496.1	493.7	488.5
75 WT% OFF, °F	583.1	595.2	586.1	570.9	591.9	582.2	579.5	576.7	568.4
95 WT% OFF, °F	779.6	761.9	712.9	730.5	767.4	722.0	722.6	726.7	719.6
400°F-- WT %	19.77	9.46	12.82	19.81	9.22	12.32	12.58	13.40	13.32
400-700°F, WT %	70.63	60.49	81.27	73.57	91.47	81.18	80.91	79.83	80.37
700°F+, WT %	9.87	9.55	5.91	6.62	9.31	6.50	6.51	6.77	6.31
SPECIFIC GRAVITY @ 60°F/60°F	0.960	0.950	0.982	0.973	0.967	0.968	0.963	0.957	0.950

TABLE A-3 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
 FEED COAL, BOTTOMS FEED, RESIDUAL BOTTOMS, AND SOLVENT INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 621	1 622	1 623	1 624	1 625	1 626	1 627	1 628	1 629
<b>FEED COAL ANALYSIS</b>									
<b>ELEMENTAL ANALYSIS, DRY WT%</b>									
CARBON	76.68*	76.70*	56.56	67.34	54.56	62.67	60.23	58.40	61.57
HYDROGEN	4.18	4.19	5.12	5.08	4.92	5.28	5.11	4.81	5.23
NITROGEN	1.10	1.10	0.81	0.86	0.66	0.71	0.73	0.66	0.67
SULFUR (TOTAL)	0.33	0.37	0.77	0.78	0.28	0.25	0.22	0.28	0.25
SULFATE	0.06	0.06	0.05	0.04	0.03	0.04	0.05	0.04	0.04
PYRITIC	0.01	0.01	0.25	0.25	0.08	0.06	0.07	0.08	0.05
ASH	13.06	13.66	8.51	6.49	22.73	18.07	20.84	22.81	18.89
ASH (SO <sub>3</sub> ) FREE	12.97	12.87	7.70	7.04	22.25	17.87	20.42	21.98	18.47
OXYGEN	10.76	10.67	24.06	25.94	25.80	24.28	26.34	24.98	26.72
MOISTURE	0.15	0.22	0.10	0.72	0.14	0.16	0.10	0.23	0.08
<b>BOTTOMS FEED ANALYSIS</b>									
<b>ELEMENTAL ANALYSIS, DRY WT %</b>									
CARBON			73.13	73.08					
HYDROGEN			4.88	4.91					
NITROGEN			1.00	1.04					
SULFUR			0.56	0.62					
ASH			14.54	14.54					
ASH (SO <sub>3</sub> ) FREE			12.94	12.93					
OXYGEN			13.22	13.29					
<b>RESIDUAL BOTTOMS, A-1</b>									
<b>ELEMENTAL ANALYSIS, DRY WT%</b>									
CARBON	75.51	75.94	74.50	73.72	52.68	54.20	54.96	52.02	58.21
HYDROGEN	4.17	4.21	4.74	4.68	3.62	3.66	3.66	3.49	4.41
NITROGEN	0.99	0.99	1.00	1.03	0.77	0.78	0.75	0.69	0.74
SULFUR	0.36	0.37	0.45	0.57	0.20	0.21	0.18	0.17	0.15
ASH	16.00	15.57	14.34	15.14	36.24	36.44	34.85	38.08	30.46
ASH (SO <sub>3</sub> ) FREE	15.12	14.70	12.67	13.37	35.79	35.41	34.43	37.57	29.57
OXYGEN	9.97	9.69	10.94	12.20	24.20	22.20	22.70	23.96	22.33
<b>DISTILLATION</b>									
1000°F-	10.50	13.70	14.10	10.90	5.90	5.80	4.20	3.30	4.30
N <sub>2</sub> IN 1000°F-	0.65			0.50					
SULFUR IN 1000°F-	0.02	0.03	0.10	0.06	0.05	0.05	0.04	0.03	0.05
<b>SOLVENT, A-2</b>									
<b>ELEMENTAL ANALYSIS, DRY WT%</b>									
CARBON	89.69	89.75	89.82	89.84	89.71	89.63	89.49	89.45	89.23
HYDROGEN	10.17	10.16	9.96	10.04	9.79	9.95	10.13	10.26	10.61
NITROGEN	0.01	0.01	0.02	0.02	0.06	0.05	0.04	0.03	0.02
SULFUR	0.000	0.0	0.0	0.0	0.001	0.000	0.000	0.000	0.000
OXYGEN	0.13	0.07	0.20	0.11	0.44	0.37	0.35	0.25	0.13
BTU/HR IN SOLVENT, % OF RV	247.	239.	245.	232.	221.	218.	211.	211.	195.
<b>DISTILLATION</b>									
5 WT% OFF, °F	390.5	391.0	389.8	390.2	383.9	393.5	400.3	394.5	396.6
15 WT% OFF, °F	399.6	400.3	399.1	400.4	398.3	402.4	410.7	405.6	408.9
25 WT% OFF, °F	425.6	423.4	425.7	428.9	429.9	434.5	444.5	437.0	438.7
50 WT% OFF, °F	487.0	483.7	492.8	495.1	511.3	511.4	521.3	514.0	511.3
75 WT% OFF, °F	558.4	550.9	575.6	577.6	590.5	591.3	605.7	605.1	601.2
95 WT% OFF, °F	685.7	680.9	697.8	713.0	752.8	749.9	780.9	782.4	776.9
400°F+, WT %	15.96	15.20	15.93	15.12	15.95	12.58	5.19	11.38	8.70
400-700°F, WT %	80.04	81.04	79.31	79.05	75.60	78.63	83.59	77.09	80.24
700°F+, WT %	4.00	3.76	4.76	5.83	8.45	8.79	11.22	11.53	11.06
SPECIFIC GRAVITY @ 60°F/60°F	0.965	0.964	0.967	0.963	0.977	0.966	0.959	0.953	0.939

\* A-1 FEED ANALYSIS

TABLE A-3 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
 FEED COAL, BOTTOMS FEED, RESIDUAL BOTTOMS, AND SOLVENT INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 630	1 631	1 632	1 633	1 634	1 635	1 636	1 637	1 638
<u>FEED COAL ANALYSIS</u>									
ELEMENTAL ANALYSIS, DRY WTC									
CARBON	60.06	59.56	61.30	59.18	59.21	57.81	61.48	60.03	60.79
HYDROGEN	5.01	5.06	5.13	5.04	5.01	4.80	5.07	4.98	5.11
NITROGEN	0.63	0.67	0.68	0.67	0.66	0.66	0.69	0.67	0.65
SULFUR (TOTAL)	0.26	0.27	0.26	0.29	0.26	0.24	0.26	0.29	0.23
SULFATE	0.04	0.04	0.04	0.04	0.03	0.04	0.04	0.04	0.04
PYRITIC	0.08	0.09	0.05	0.06	0.05	0.07	0.05	0.07	0.04
ASH	21.23	21.34	20.32	22.26	21.60	23.44	19.06	21.42	20.23
ASH (SO <sub>3</sub> ) FREE	20.76	20.90	19.86	21.80	20.99	22.57	18.57	20.90	19.77
OXYGEN	27.26	26.49	26.29	26.37	27.03	27.46	26.01	25.81	25.93
MOISTURE	0.11	0.00	0.00	0.00	0.00	0.00	0.07	0.03	0.00
RESIDUAL BOTTOMS, A-1									
ELEMENTAL ANALYSIS, DRY WTC									
CARBON	58.32	56.51	56.73	53.20	54.29	55.02	53.01	54.78	57.40
HYDROGEN	4.39	4.34	3.43	3.22	3.36	3.97	3.80	3.84	4.15
NITROGEN	0.67	0.79	0.70	0.66	0.72	0.69	0.73	0.80	0.74
SULFUR	0.14	0.14	0.27	0.21	0.22	0.16	0.18	0.14	0.18
ASH	30.22	32.02	34.34	38.13	37.41	34.71	36.66	34.78	31.07
ASH (SO <sub>3</sub> ) FREE	29.95	31.60	33.98	37.72	36.83	34.30	35.69	34.36	30.27
OXYGEN	21.35	22.70	21.90	24.00	23.00	22.80	24.20	23.50	21.90
DISTILLATION									
1000°F-	6.80	4.50	7.80	4.10	8.10	6.80	5.80	4.20	7.90
N <sub>2</sub> IN 1000°F-									
SULFUR IN 1000°F-	0.06	0.04	0.05	0.03	0.03	0.10	0.05	0.05	0.05
SOLVENT, A-2									
ELEMENTAL ANALYSIS, DRY WTC									
CARBON	89.10	83.96	89.66	89.84	89.69	89.77	89.59	89.46	89.63
HYDROGEN	10.74	10.90	9.89	9.96	10.01	9.71	9.96	10.21	9.91
NITROGEN	0.02	0.02	0.04	0.03	0.03	0.07	0.05	0.04	0.05
SULFUR	0.000	0.000	0.001	0.000	0.000	0.002	0.000	0.000	0.001
OXYGEN	0.15	0.12	0.41	0.18	0.27	0.45	0.40	0.30	0.41
DONOR H <sub>2</sub> IN SOLVENT, % OF RV	191.	183.	221.	221.	218.	214.	212.	212.	205.
DISTILLATION									
5 WT% OFF, °F	393.5	392.6	393.8	408.1	396.7	396.1	408.9	401.0	393.3
15 WT% OFF, °F	404.9	404.9	402.3	415.9	405.3	403.0	416.0	409.3	402.6
25 WT% OFF, °F	434.2	439.9	422.8	439.9	422.9	433.8	446.9	439.9	433.8
50 WT% OFF, °F	505.0	506.8	492.1	507.3	485.3	512.4	518.0	511.7	514.2
75 WT% OFF, °F	594.0	600.3	578.6	595.7	570.4	595.3	601.6	596.7	596.2
95 WT% OFF, °F	768.3	779.1	704.0	734.9	710.3	758.5	762.2	779.9	758.5
400°F-, WT %	12.09	13.35	12.95	2.00	8.44	12.38	0.46	4.20	12.49
400-700°F, WT %	77.42	74.92	81.77	90.41	85.89	78.53	90.19	85.25	77.82
700°F+, WT %	10.49	11.73	5.28	7.59	5.66	9.09	9.35	10.55	9.69
SPECIFIC GRAVITY @ 60°F/60°F	0.932	0.926	0.967	0.961	0.954	0.976	0.966	0.956	0.967

TABLE A-3 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY									
FEED COAL, BOTTOMS FEED, RESIDUAL BOTTOMS, AND SOLVENT INSPECTIONS									
RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 639	1 640	1 641	1 642	1 643	1 644	1 645	1 646	1 647
<u>FEED COAL ANALYSIS</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	57.88	63.04	59.39	58.48	58.75	57.77	62.61	57.98	58.17
HYDROGEN	4.80	5.27	4.76	4.96	4.90	4.90	5.24	4.91	4.87
NITROGEN	0.67	0.69	0.65	0.63	0.64	0.64	0.69	0.67	0.65
SULFUR (TOTAL)	0.28	0.25	0.24	0.26	0.29	0.26	0.26	0.26	0.27
SULFATE	0.05	0.04	0.06	0.05	0.05	0.04	0.03	0.04	0.04
PYRITIC	0.06	0.04	0.06	0.07	0.10	0.07	0.04	0.06	0.05
ASH	23.59	17.26	21.67	22.69	22.39	23.75	17.82	23.38	23.26
ASH (SO <sub>3</sub> ) FREE	23.06	16.60	21.33	22.17	21.86	23.13	17.12	22.81	22.71
OXYGEN	26.49	25.32	26.99	26.32	26.90	27.19	24.85	26.44	27.04
MOISTURE	0.60	0.00	0.00	0.00	0.22	0.00	0.11	0.11	0.21
<u>RESIDUAL BOTTOMS, A-1</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	56.98	53.23	55.50	54.73	52.22	52.23	54.43	55.06	49.47
HYDROGEN	4.00	3.81	3.56	3.41	3.79	3.97	4.00	4.13	3.64
NITROGEN	0.80	0.69	0.70	0.62	0.70	0.69	0.71	0.59	0.64
SULFUR	0.17	0.20	0.21	0.24	0.19	0.16	0.17	0.19	0.13
ASH	31.52	36.76	35.62	36.75	37.12	37.20	34.40	33.85	40.26
ASH (SO <sub>3</sub> ) FREE	31.03	36.17	35.14	36.02	36.75	36.84	33.43	33.49	39.64
OXYGEN	21.60	19.59	20.30	22.30	24.83	24.50	23.50	24.00	27.31
<u>DISTILLATION</u>									
1000°F-	8.10	7.50	6.10	7.10	6.50	9.60	10.00	11.20	5.70
N <sub>2</sub> IN 1000°F-				0.37					
SULFUR IN 1000°F-	0.06	0.05	0.03	0.02	0.06	0.08	0.04	0.04	0.04
<u>SOLVENT, A-2</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	89.50	89.76	89.75	89.76	89.59	89.77	89.76	89.54	89.31
HYDROGEN	10.00	9.69	9.74	9.73	9.99	9.56	9.71	10.05	10.32
NITROGEN	0.04	0.07	0.06	0.05	0.03	0.06	0.04	0.02	0.02
SULFUR	0.000	0.003	0.000	0.009	0.000	0.002	0.001	0.000	0.000
OXYGEN	0.37	0.48	0.44	0.45	0.40	0.62	0.49	0.39	0.35
DENSITY @ 70°F IN SOLVENT, % OF RV	211.	207.	192.	203.	237.	242.	241.	221.	209.
<u>DISTILLATION</u>									
5 WT% OFF, °F	394.6	397.0	397.3	395.0	398.6	396.2	395.9	393.5	393.8
15 WT% OFF, °F	404.6	410.0	408.7	404.5	407.0	402.7	401.8	401.4	402.6
25 WT% OFF, °F	436.9	447.0	440.0	430.5	434.6	432.1	435.5	433.3	434.4
50 WT% OFF, °F	518.7	530.0	517.9	503.2	503.2	504.5	512.5	507.7	506.6
75 WT% OFF, °F	604.4	615.2	599.6	590.8	586.3	582.0	587.2	586.4	587.4
95 WT% OFF, °F	788.5	804.3	771.8	758.0	726.2	700.4	718.9	737.2	749.5
400°F+, WT %	12.03	8.63	6.24	11.73	7.63	10.98	12.88	13.63	12.98
400-700°F, WT %	76.19	78.73	81.82	79.29	85.48	84.07	80.67	78.77	78.59
700°F+, WT %	11.78	12.64	9.94	8.98	6.89	4.95	6.45	7.60	8.43
SPECIFIC GRAVITY @ 60°F/60°F	0.961	0.975	0.972	0.967	0.960	0.982	0.974	0.959	0.950



TABLE A-4

ACLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
NAPHTHA AND SOUR WATER INSPECTIONS

RELIN UNIT NUMBER YIELD PERIOD NUMBER	1 603	1 604	1 605	1 606	1 607	1 608	1 609	1 610	1 611
<u>NAPHTHA, A-3</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	85.57	85.52	85.43	85.51	85.46	85.41	85.53	85.59	84.88
HYDROGEN	12.74	12.64	12.68	12.74	12.85	12.85	12.74	12.75	12.89
NITROGEN	0.06	0.06	0.06	0.04	0.04	0.03	0.04	0.07	0.04
SULFUR	0.002	0.002	0.004	0.003	0.003	0.003	0.002	0.007	0.002
OXYGEN	1.64	1.73	1.64	1.70	1.65	1.70	1.69	1.59	2.19
DISTILLATION									
5 WT% OFF, °F	168.1	169.1	165.6	165.6	164.0	168.2	181.1	188.6	181.1
15 WT% OFF, °F	209.6	212.8	213.0	211.8	210.5	213.5	216.9	223.7	216.5
25 WT% OFF, °F	233.1	239.0	240.4	239.0	235.7	240.4	246.9	263.2	246.8
50 WT% OFF, °F	312.2	317.1	317.1	317.8	310.6	312.1	320.0	324.7	313.6
75 WT% OFF, °F	362.5	362.8	362.1	364.3	361.0	361.9	364.2	364.9	357.6
95 WT% OFF, °F	383.5	384.1	383.5	384.5	382.9	384.1	384.4	393.4	377.0
400°F+, WT %	99.39	99.27	99.44	99.40	99.54	99.46	99.37	98.04	99.73
400°F+, WT %	0.61	0.73	0.56	0.60	0.46	0.54	0.63	1.96	0.27
SPECIFIC GRAVITY @ 60°F/60°F	0.831	0.833	0.833	0.830	0.826	0.828	0.832	0.831	0.829
<u>SOUR WATER, A-4</u>									
CO <sub>2</sub> , WT %	7.20	7.70	6.90	4.60	4.80	4.70	4.30	4.90	5.20
NH <sub>3</sub> , WT %	3.85	3.70	3.45	1.70	2.10	2.20	2.60	2.40	2.60
H <sub>2</sub> S, WT %	0.51	0.51	0.62	0.35	0.33	0.51	0.51	0.43	0.60
SPECIFIC GRAVITY @ 60°F/60°F	1.068	1.064	1.063	1.040	1.034	1.032	1.039	1.042	1.042

TABLE A-4 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
NAPHTHA AND SOUR WATER INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 612	1 613	1 614	1 615	1 616	1 617	1 618	1 619	1 620
<u>NAPHTHA, A-3</u>									
ELEMENTAL ANALYSIS, DRY WTE									
CARBON	84.96	85.80	85.60	85.15	85.12	86.54	86.63	86.61	86.71
HYDROGEN	12.88	12.73	12.64	12.88	12.88	13.39	13.34	13.37	13.26
NITROGEN	0.04	0.05	0.05	0.04	0.04	0.01	0.01	0.01	0.01
SULFUR	0.018	0.003	0.005	0.002	0.005	0.002	0.000	0.000	0.001
OXYGEN	2.10	1.43	1.71	1.93	1.95	0.06	0.02	0.01	0.02
DISTILLATION									
5 WTS OFF, °F	180.8	180.9	169.0	166.8	166.5	164.6	168.3	203.5	198.6
15 WTS OFF, °F	216.1	218.0	212.8	211.9	211.8	189.3	196.9	237.3	237.4
25 WTS OFF, °F	247.3	253.6	236.7	233.5	234.3	218.8	223.7	256.6	271.6
50 WTS OFF, °F	314.3	324.4	315.0	306.9	308.0	291.2	298.8	330.7	348.3
75 WTS OFF, °F	359.2	363.8	363.4	359.8	361.1	361.1	366.6	383.0	386.1
95 WTS OFF, °F	378.5	382.3	381.6	380.4	382.8	380.1	384.3	404.6	408.1
400°F, WT %	99.65	99.49	99.61	99.64	99.38	99.12	99.03	91.68	88.84
400°F, WT %	0.35	0.51	0.39	0.36	0.62	0.88	0.97	8.32	11.16
SPECIFIC GRAVITY @ 60°F/60°F	0.830	0.832	0.830	0.826	0.825	0.806	0.807	0.806	0.808
<u>SOUR WATER, A-4</u>									
CO <sub>2</sub> WT %	4.80	5.50	6.00	5.70	6.40	7.26	7.28	14.20	8.75
NH <sub>3</sub> WT %	2.31	2.38	2.87	2.84	2.95	3.70	4.30	4.24	4.02
H <sub>2</sub> S WT %	1.15	0.40	1.02	0.47	0.40	0.87	0.95	0.81	0.77
SPECIFIC GRAVITY @ 60°F/60°F	1.052	1.042	1.052	1.041	1.051	1.057	1.047	1.052	1.056

TABLE A-4 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
NAPHTHA AND SOUR WATER INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 621	1 622	1 623	1 624	1 625	1 626	1 627	1 628	1 629
<u>NAPHTHA, A-3</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	87.73	87.77	86.73	86.80	86.55	86.28	86.16	86.18	86.00
HYDROGEN	12.26	12.22	13.26	13.19	13.34	13.45	13.58	13.47	13.79
NITROGEN	0.01	0.01	0.01	0.01	0.03	0.02	0.02	0.02	0.01
SULFUR	0.000	0.0	0.001	0.0	0.000	0.000	0.000	0.000	0.000
OXYGEN	0.0	0.0	0.0	0.0	0.09	0.25	0.24	0.33	0.20
DISTILLATION									
5 WT% OFF, °F	201.2	202.2	183.1	190.8	172.0	173.8	173.0	172.5	182.0
15 WT% OFF, °F	236.8	237.9	211.9	228.3	208.5	205.5	205.8	210.8	218.5
25 WT% OFF, °F	272.2	279.6	238.7	242.1	236.6	236.2	236.4	237.9	239.6
50 WT% OFF, °F	374.7	375.1	302.6	326.7	304.9	295.7	295.6	307.6	291.0
75 WT% OFF, °F	396.0	393.3	371.2	380.8	374.3	366.6	366.7	374.1	354.8
95 WT% OFF, °F	411.0	407.7	393.6	398.9	394.7	391.7	393.6	399.4	388.5
400°F-, WT %	81.5*	86.36	98.11	95.91	97.52	98.06	97.54	95.53	98.08
400°F+, WT %	18.42	13.62	1.89	4.09	2.48	1.94	2.46	4.47	1.92
SPECIFIC GRAVITY @ 60°F/60°F	0.841	0.845	0.808	0.817	0.803	0.797	0.794	0.798	0.790
<u>SOUR WATER, A-4</u>									
CO <sub>2</sub> WT %	3.50	5.20	7.40	6.90	8.10	7.30	6.37	6.36	5.07
NH <sub>3</sub> WT %	0.67	0.65	0.36	0.34	0.70	0.49	0.84	0.68	1.41
H <sub>2</sub> S WT %	2.34	1.74	0.64	0.64	0.51	0.36	0.36	0.35	0.34
SPECIFIC GRAVITY @ 60°F/60°F	1.007	0.996	1.057	1.043	1.051	1.046	1.051	1.052	1.038

TABLE A-4 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
NAPHTHA AND SOUR WATER INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 630	1 631	1 632	1 633	1 634	1 635	1 636	1 637	1 638
<u>NAPHTHA, A-3</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	85.18	86.14	86.24	86.38	86.35	86.42	86.28	86.00	86.11
HYDROGEN	13.64	13.69	13.47	13.41	13.45	13.09	13.22	13.42	13.37
NITROGEN	0.01	0.01	0.01	0.01	0.01	0.04	0.03	0.03	0.03
SULFUR	0.000	0.000	0.000	0.000	0.000	0.002	0.000	0.000	0.000
OXYGEN	0.17	0.16	0.27	0.20	0.18	0.118	0.48	0.56	0.49
DISTILLATION									
5 WT% OFF, °F	174.3	181.7	117.2	139.7	116.8	157.6	152.9	151.3	151.6
15 WT% OFF, °F	216.6	217.3	155.0	189.7	154.8	210.2	201.3	191.8	190.2
25 WT% OFF, °F	227.7	228.1	191.6	219.5	191.5	240.4	219.5	216.3	215.5
50 WT% OFF, °F	292.6	293.2	280.6	281.8	281.2	338.3	310.8	292.3	282.9
75 WT% OFF, °F	354.3	354.3	349.4	354.5	348.7	371.1	363.7	359.4	352.8
95 WT% OFF, °F	380.1	380.3	375.4	381.2	376.6	386.6	382.1	379.9	376.0
400F-, WT %	99.63	99.39	99.98	99.64	99.95	99.24	99.01	99.53	99.62
400F+, WT %	0.37	0.61	0.02	0.36	0.05	0.76	0.99	0.47	0.38
SPECIFIC GRAVITY @ 60°F/60°F	0.795	0.794	0.792	0.792	0.791	0.815	0.808	0.799	0.802
<u>SOUR WATER, A-4</u>									
CO <sub>2</sub> WT %	5.72	6.81	7.66	7.37	7.07	6.00	6.10	6.04	6.21
NH <sub>3</sub> WT %	1.41	1.85	4.35	4.21	4.20	3.41	3.41	3.36	3.19
H <sub>2</sub> S WT %	0.27	0.32	0.47	0.37	0.37	0.32	0.32	0.34	0.36
SPECIFIC GRAVITY @ 60°F/60°F	1.032	1.037	1.061	1.056	1.057	1.041	1.041	1.050	1.051

TABLE A-4 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY									
NAPHTHA AND SOUR WATER INSPECTIONS									
RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 639	1 640	1 641	1 642	1 643	1 644	1 645	1 646	1 647
<u>NAPHTHA, A-3</u>									
ELEMENTAL ANALYSIS, DRY WT%									
CARBON	86.22	85.84	85.83	85.75	86.30	86.90	86.21	86.12	86.00
HYDROGEN	13.31	13.52	13.28	13.62	13.24	12.71	13.35	13.52	13.79
NITROGEN	0.02	0.03	0.04	0.03	0.02	0.04	0.03	0.02	0.01
SULFUR	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.000	0.026
OXYGEN	0.45	0.56	0.84	0.60	0.44	0.35	0.41	0.34	0.18
DISTILLATION									
5 WT% OFF, °F	158.5	135.7	92.7	101.8	125.7	128.5	164.8	154.2	126.6
15 WT% OFF, °F	201.4	169.6	160.6	163.5	169.8	170.8	211.6	187.0	174.0
25 WT% OFF, °F	220.7	184.7	181.4	180.1	186.4	188.4	242.0	216.1	207.4
50 WT% OFF, °F	298.7	222.0	237.8	218.7	257.1	273.2	329.6	299.2	270.0
75 WT% OFF, °F	362.0	296.1	313.0	296.7	320.8	324.5	367.4	358.8	345.2
95 WT% OFF, °F	381.0	325.7	334.0	331.3	337.8	339.8	384.9	376.9	373.4
400°F-, WT %	56.63	59.93	59.83	59.86	59.84	59.64	58.66	59.28	59.80
400°F+, WT %	3.32	0.07	0.17	0.14	0.16	0.36	1.34	0.72	0.20
SPECIFIC GRAVITY @ 60°F/60°F	0.804	0.786	0.791	0.784	0.802	0.818	0.801	0.797	0.784
<u>SOUR WATER, A-4</u>									
CO <sub>2</sub> , WT %	6.05	5.82	5.86	6.44	6.52	6.65	6.95	7.04	9.14
NH <sub>3</sub> , WT %	3.25	2.97	3.64	3.17	3.69	4.40	4.20	3.47	3.79
H <sub>2</sub> S, WT %	3.24	0.09	0.09	0.51	0.19	0.36	0.64	0.55	0.51
SPECIFIC GRAVITY @ 60°F/60°F	1.042	1.047	1.051	1.051	1.047	1.061	1.052	1.046	1.047

TABLE A-5

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
TAIL GAS INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 603	1 604	1 605	1 606	1 607	1 608	1 609	1 610	1 611
<u>TAIL GAS, MOLE %</u>									
HYDROGEN	92.54	92.48	92.05	91.83	91.44	91.15	92.30	92.33	92.12
NITROGEN	2.99	3.09	3.52	3.56	3.57	3.72	3.77	3.60	3.75
ARGON + OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0	0.0
HYDROGEN SULFIDE	0.06	0.06	0.06	0.09	0.09	0.09	0.00	0.0	0.0
SULFUR DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0	0.0
CARBON MONOXIDE	0.35	0.33	0.30	0.32	0.34	0.36	0.36	0.30	0.30
CARBON DIOXIDE	0.60	0.65	0.69	0.97	1.02	1.05	1.06	0.77	0.75
METHANE	1.84	1.76	1.62	1.58	1.76	1.82	0.89	1.40	1.47
ETHYLENE	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0	0.0
ETHANE	0.72	0.72	0.69	0.72	0.75	0.75	0.47	0.47	0.49
PROPYLENE	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0	0.0
PROPANE	0.47	0.47	0.46	0.52	0.53	0.52	0.35	0.31	0.32
N-BUTANE	0.12	0.12	0.12	0.13	0.12	0.13	0.15	0.13	0.13
I-BUTANE	0.03	0.04	0.04	0.04	0.03	0.04	0.03	0.03	0.03
1-PENTENE	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0	0.0
CIS/TRANS, 2-PENTENE	0.0	0.0	0.00	0.0	0.0	0.0	0.00	0.00	0.00
N-PENTANE	0.02	0.03	0.05	0.04	0.03	0.03	0.02	0.02	0.02
I-PENTANE	0.02	0.03	0.04	0.03	0.02	0.02	0.01	0.01	0.01
N-HEXANE	0.02	0.02	0.03	0.02	0.02	0.02	0.02	0.03	0.03
2-METHYL PENTANE	0.01	0.01	0.02	0.01	0.01	0.01	0.02	0.03	0.03
N-HEPTANE	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.02
2-METHYL HEXANE	0.0	0.0	0.00	0.00	0.0	0.0	0.00	0.01	0.01
3-METHYL HEXANE	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.00	0.01
CYCLO-PENTANE/3-MEPENTANE	0.0	0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02
METHYL CYCLO-PENTANE	0.03	0.01	0.01	0.01	0.01	0.01	0.03	0.06	0.05
CYCLO-HEXANE	0.03	0.03	0.04	0.06	0.06	0.06	0.07	0.12	0.10
METHYL CYCLO-HEXANE	0.018	0.031	0.035	0.022	0.023	0.023	0.061	0.108	0.092
BENZENE	0.0	0.0	0.0	0.0	0.0	0.0	0.000	0.0	0.0
TOLUENE	0.042	0.041	0.044	0.037	0.035	0.041	0.061	0.043	0.045
I-BUTENE	0.0	0.0	0.0	0.0	0.0	0.0	0.000	0.0	0.0
TRANS-2-BUTENE	0.009	0.004	0.019	0.0	0.0	0.0	0.010	0.030	0.031
CIS-2-BUTENE	0.0	0.0	0.000	0.0	0.0	0.0	0.000	0.001	0.001
AMMONIA	0.003	0.005	0.001	0.002	0.001	0.001	0.003	0.005	0.001
WATER	0.053	0.050	0.155	0.181	0.123	0.108	0.301	0.163	0.170

TABLE A-5 (Continued)  
RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
TAIL GAS INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 612	1 613	1 614	1 615	1 616	1 617	1 618	1 619	1 620
TAIL GAS, MOLE %									
HYDROGEN	92.08	91.63	92.09	92.81	92.94	91.32	91.68	91.93	91.59
NITROGEN	3.64	3.98	4.24	3.91	3.65	4.19	4.03	4.04	4.28
ARGON + OXYGEN	0.0	0.0	0.0	0.0	0.0	0.00	0.00	0.00	0.00
HYDROGEN SULFIDE	0.0	0.0	0.0	0.0	0.0	0.05	0.04	0.05	0.04
SULFUR DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.00	0.00	0.00	0.00
CARBON MONOXIDE	0.29	0.30	0.37	0.34	0.35	0.41	0.37	0.40	0.41
CARBON DIOXIDE	0.76	0.82	0.88	0.74	0.79	0.33	0.30	0.32	0.34
METHANE	1.50	1.61	1.11	1.05	1.01	1.61	1.51	1.55	1.61
ETHYLENE	0.0	0.0	0.0	0.0	0.0	0.00	0.00	0.00	0.00
ETHANE	0.48	0.51	0.50	0.47	0.46	0.58	0.59	0.56	0.55
PROPYLENE	0.0	0.0	0.0	0.0	0.0	0.00	0.00	0.00	0.00
PROPANE	0.31	0.34	0.34	0.32	0.31	0.44	0.43	0.42	0.41
N-BUTANE	0.15	0.16	0.12	0.08	0.14	0.27	0.24	0.26	0.25
I-BUTANE	0.04	0.04	0.03	0.02	0.04	0.06	0.05	0.06	0.06
1-PENTENE	0.0	0.0	0.0	0.0	0.0	0.00	0.00	0.00	0.00
CIS/TRANS, 2-PENTENE	0.00	0.00	0.0	0.0	0.0	0.00	0.00	0.00	0.00
N-PENTANE	0.03	0.03	0.02	0.02	0.03	0.10	0.10	0.10	0.11
I-PENTANE	0.02	0.03	0.01	0.01	0.02	0.13	0.11	0.12	0.13
N-HEXANE	0.03	0.03	0.01	0.01	0.01	0.02	0.02	0.02	0.02
2-METHYL PENTANE	0.03	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.01
N-HEPTANE	0.02	0.01	0.0	0.00	0.00	0.00	0.00	0.00	0.00
2-METHYL HEXANE	0.01	0.01	0.0	0.0	0.0	0.00	0.00	0.00	0.00
3-METHYL HEXANE	0.00	0.00	0.0	0.0	0.0	0.00	0.00	0.00	0.00
CYCLO-PENTANE/3-MEPENTANE	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.02	0.02
METHYL CYCLO-PENTANE	0.06	0.05	0.01	0.01	0.01	0.02	0.02	0.02	0.02
CYCLO-HEXANE	0.10	0.09	0.01	0.01	0.01	0.05	0.05	0.05	0.05
METHYL CYCLO-HEXANE	0.086	0.074	0.033	0.032	0.030	0.030	0.032	0.029	0.028
BENZENE	0.0	0.0	0.0	0.0	0.0	0.000	0.000	0.000	0.000
TOLUENE	0.024	0.051	0.050	0.038	0.039	0.022	0.021	0.021	0.022
I-BUTENE	0.0	0.0	0.0	0.0	0.0	0.000	0.000	0.000	0.000
TRANS 2-BUTENE	0.030	0.022	0.0	0.0	0.0	0.000	0.000	0.000	0.000
CIS 2-BUTENE	0.001	0.001	0.0	0.0	0.0	0.000	0.000	0.000	0.000
AMMONIA	0.001	0.001	0.001	0.001	0.000	0.001	0.005	0.002	0.004
WATER	0.272	0.167	0.144	0.101	0.143	0.336	0.369	0.012	0.045

TABLE A-5 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
TAIL GAS INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 621	1 622	1 623	1 624	1 625	1 626	1 627	1 628	1 629
<u>TAIL GAS, MOLE %</u>									
HYDROGEN	93.05	94.56	91.62	93.54	93.73	93.97	92.93	93.86	94.79
NITROGEN	4.17	3.27	5.73	3.81	3.01	3.03	4.32	3.59	3.38
ARGON + OXYGEN	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
HYDROGEN SULFIDE	0.0	0.00	0.05	0.05	0.0	0.0	0.0	0.00	0.0
SULFUR DIOXIDE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
CARBON MONOXIDE	0.14	0.09	0.30	0.31	0.28	0.25	0.24	0.22	0.19
CARBON DIOXIDE	0.0	0.0	0.34	0.37	0.24	0.24	0.24	0.21	0.20
METHANE	1.23	0.90	0.85	0.83	1.29	1.28	1.04	0.96	0.51
ETHYLENE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
ETHANE	0.39	0.47	0.36	0.36	0.49	0.50	0.49	0.47	0.25
PROPYLENE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
PROPANE	0.30	0.28	0.26	0.26	0.36	0.37	0.37	0.35	0.19
N-BUTANE	0.23	0.14	0.13	0.14	0.16	0.15	0.14	0.13	0.08
I-BUTANE	0.05	0.03	0.04	0.04	0.04	0.03	0.03	0.03	0.02
1-PENTENE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
CIS/TRANS, 2-PENTENE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.00
N-PENTANE	0.09	0.03	0.03	0.03	0.00	0.00	0.00	0.00	0.00
I-PENTANE	0.14	0.07	0.05	0.05	0.01	0.00	0.00	0.00	0.00
N-HEXANE	0.01	0.01	0.01	0.01	0.02	0.03	0.03	0.03	0.02
2-METHYL PENTANE	0.00	0.00	0.01	0.01	0.01	0.01	0.01	0.01	0.01
N-HEPTANE	0.0	0.0	0.0	0.0	0.00	0.00	0.00	0.00	0.00
2-METHYL HEXANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
3-METHYL HEXANE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.00	0.0
CYCLO-PENTANE/3-MEPENTANE	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
METHYL CYCLO-PENTANE	0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02	0.02
CYCLO-HEXANE	0.06	0.05	0.04	0.01	0.05	0.04	0.03	0.03	0.04
METHYL CYCLO-HEXANE	0.023	0.017	0.019	0.027	0.025	0.028	0.034	0.033	0.044
BENZENE	0.013	0.008	0.0	0.0	0.0	0.0	0.0	0.000	0.0
TOLUENE	0.000	0.012	0.025	0.030	0.026	0.015	0.013	0.019	0.000
1-BUTENE	0.0	0.0	0.0	0.0	0.008	0.004	0.004	0.003	0.006
TRANS 2-BUTENE	0.0	0.0	0.0	0.000	0.0	0.0	0.0	0.000	0.0
CIS 2-BUTENE	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.000	0.0
AMMONIA	0.063	0.020	0.005	0.004	0.006	0.001	0.001	0.000	0.003
WATER	0.007	0.000	0.107	0.104	0.200	0.009	0.031	0.013	0.240



TABLE A-5 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
TAIL GAS INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 630	1 631	1 632	1 633	1 634	1 635	1 636	1 637	1 638
<u>TAIL GAS, MOLE %</u>									
HYDROGEN	95.22	94.70	91.34	91.69	91.61	94.17	94.53	94.66	94.75
NITROGEN	3.18	3.71	3.43	3.49	2.95	2.94	2.52	2.36	3.04
ARGON + OXYGEN	0.0	0.0	0.00	0.0	0.0	0.0	0.0	0.0	0.0
HYDROGEN SULFIDE	0.0	0.0	0.00	0.0	0.00	0.0	0.0	0.0	0.0
SULFUR DIOXIDE	0.0	0.0	0.00	0.0	0.0	0.0	0.0	0.0	0.0
CARBON MONOXIDE	0.19	0.18	0.32	0.28	0.32	0.28	0.27	0.24	0.24
CARBON DIOXIDE	0.20	0.18	0.28	0.23	0.30	0.27	0.25	0.20	0.27
METHANE	0.46	0.54	2.33	2.29	2.23	1.23	1.16	1.29	0.85
ETHYLENE	0.0	0.0	0.00	0.0	0.0	0.0	0.0	0.0	0.0
ETHANE	0.26	0.24	0.94	0.84	0.94	0.46	0.42	0.40	0.33
PROPYLENE	0.0	0.0	0.00	0.0	0.0	0.0	0.0	0.0	0.0
PROPANE	0.19	0.18	0.69	0.62	0.69	0.34	0.32	0.30	0.24
N-BUTANE	0.09	0.08	0.25	0.21	0.25	0.13	0.13	0.12	0.11
I-BUTANE	0.02	0.02	0.05	0.04	0.05	0.03	0.03	0.03	0.03
1-PENTENE	0.0	0.0	0.00	0.0	0.0	0.0	0.0	0.0	0.0
CIS/TRANS, 2-PENTENE	0.00	0.00	0.00	0.00	0.00	0.0	0.0	0.0	0.0
N-PENTANE	0.00	0.00	0.00	0.00	0.00	0.03	0.03	0.03	0.02
I-PENTANE	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.02	0.02
N-HEXANE	0.03	0.02	0.04	0.03	0.03	0.02	0.02	0.02	0.02
2-METHYL PENTANE	0.01	0.01	0.03	0.02	0.02	0.01	0.01	0.01	0.01
N-HEPTANE	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00
2-METHYL HEXANE	0.0	0.0	0.00	0.00	0.00	0.0	0.00	0.00	0.00
3-METHYL HEXANE	0.00	0.0	0.00	0.0	0.00	0.0	0.0	0.00	0.00
CYCLO-PENTANE/3-MEPENTANE	0.01	0.01	0.00	0.00	0.01	0.01	0.01	0.01	0.01
METHYL CYCLO-PENTANE	0.02	0.02	0.01	0.01	0.01	0.00	0.00	0.01	0.00
CYCLO-HEXANE	0.04	0.03	0.15	0.11	0.12	0.00	0.00	0.00	0.01
METHYL CYCLO-HEXANE	0.053	0.039	0.043	0.027	0.032	0.019	0.016	0.013	0.015
BENZENE	0.0	0.0	0.000	0.0	0.0	0.0	0.0	0.0	0.0
TOLUENE	0.0	0.006	0.000	0.0	0.0	0.011	0.006	0.016	0.007
I-BUTENE	0.0	0.011	0.000	0.0	0.001	0.0	0.0	0.0	0.003
TRANS-2-BUTENE	0.000	0.000	0.002	0.001	0.001	0.0	0.0	0.0	0.0
CIS-2-BUTENE	0.000	0.000	0.000	0.0	0.000	0.0	0.0	0.0	0.0
AMMONIA	0.004	0.002	0.004	0.002	0.003	0.003	0.005	0.001	0.002
WATER	0.000	0.000	0.090	0.106	0.431	0.014	0.249	0.275	0.023

TABLE A-5 (Continued)

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY  
TAIL GAS INSPECTIONS

RCLU UNIT NUMBER YIELD PERIOD NUMBER	1 639	1 640	1 641	1 642	1 643	1 644	1 645	1 646	1 647
<u>TAIL GAS, MOLE %</u>									
HYDROGEN	45.34	52.87	90.38	91.36	92.31	92.84	93.25	91.92	91.19
NITROGEN	2.45	4.74	4.96	4.44	4.68	4.39	3.81	5.68	6.61
ARGON + OXYGEN	0.0	0.0	0.0	0.00	0.00	0.00	0.0	0.0	0.00
HYDROGEN SULFIDE	0.0	0.0	0.00	0.00	0.00	0.00	0.0	0.0	0.00
SULFUR DIOXIDE	0.0	0.0	0.0	0.00	0.00	0.00	0.0	0.0	0.00
CARBON MONOXIDE	0.22	0.25	0.34	0.31	0.26	0.24	0.28	0.22	0.20
CARBON DIOXIDE	0.27	0.24	0.33	0.24	0.12	0.12	0.13	0.14	0.11
METHANE	0.81	1.01	2.18	2.02	1.51	0.96	1.39	0.84	0.71
ETHYLENE	0.0	0.0	0.0	0.00	0.00	0.00	0.0	0.0	0.00
ETHANE	0.33	0.41	0.90	0.71	0.49	0.43	0.48	0.59	0.46
PROPYLENE	0.0	0.0	0.0	0.00	0.00	0.00	0.0	0.0	0.00
PROPANE	0.25	0.30	0.58	0.49	0.34	0.32	0.36	0.41	0.36
N-BUTANE	0.10	0.09	0.16	0.23	0.11	0.13	0.13	0.11	0.11
I-BUTANE	0.03	0.03	0.04	0.05	0.03	0.03	0.03	0.02	0.02
1-PENTENE	0.0	0.0	0.0	0.00	0.00	0.00	0.0	0.0	0.00
CIS/TRANS, 2-PENTENE	0.0	0.0	0.0	0.00	0.00	0.00	0.0	0.0	0.00
N-PENTANE	0.02	0.01	0.03	0.05	0.02	0.02	0.04	0.02	0.03
I-PENTANE	0.01	0.01	0.02	0.03	0.01	0.01	0.03	0.00	0.01
N-HEXANE	0.02	0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.01
2-METHYL PENTANE	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
N-HEPTANE	0.00	0.0	0.00	0.00	0.00	0.00	0.00	0.0	0.00
2-METHYL HEXANE	0.00	0.00	0.0	0.00	0.00	0.00	0.0	0.0	0.00
3-METHYL HEXANE	0.00	0.0	0.0	0.00	0.00	0.00	0.00	0.00	0.00
CYCLO-PENTANE/3-MEPENTANE	0.01	0.00	0.01	0.01	0.00	0.01	0.0	0.0	0.00
METHYL CYCLO-PENTANE	0.00	0.00	0.00	0.00	0.00	0.02	0.01	0.00	0.01
CYCLO HEXANE	0.01	0.00	0.00	0.00	0.01	0.02	0.02	0.02	0.01
METHYL CYCLO-HEXANE	0.000	0.002	0.003	0.010	0.006	0.003	0.007	0.008	0.006
BENZENE	0.0	0.0	0.0	0.000	0.000	0.000	0.0	0.0	0.000
TOLUENE	0.001	0.009	0.012	0.009	0.018	0.015	0.018	0.013	0.011
1-BUTENE	0.001	0.0	0.0	0.000	0.000	0.000	0.0	0.0	0.000
TRANS 2-BUTENE	0.000	0.0	0.0	0.000	0.000	0.000	0.000	0.0	0.000
CIS 2-BUTENE	0.0	0.0	0.0	0.000	0.000	0.000	0.0	0.0	0.000
AMMONIA	0.004	0.004	0.003	0.004	0.002	0.003	0.008	0.007	0.005
WATER	0.018	0.006	0.020	0.009	0.062	0.417	0.000	0.000	0.130

Table A-6

## RCLU-1 LIQUEFACTION REACTOR INSPECTIONS FROM AUSTRALIAN BLACK COAL

Source RCLU Run Number	1	2	3	Wandoan Area 4	5	6
Hours on Coal	205	93	86	85	155	149
Reactor Conditions						
Temperature, °F	800/840	840	800	840	840/880	840
Pressure, psig	1500	1500	1500	1500	1500	2500
Slurry Residence Time, Minutes	40	100	100	25	25	40
Solvent/Coal Ratio	1.6	1.6	1.6	1.6	1.6	1.6
H <sub>2</sub> Treat Rate, Wt% on Dry Coal	4	4	4	4	4	4
Shutdown Conditions						
Reactor Plug?	Voluntary	Yes	Voluntary	Yes	Voluntary	Voluntary
Feed Line Plug?	Shutdown	No	Shutdown	No	Shutdown	Shutdown
Reactor Solids (toluene washed)						
Accumulation Rate						
(Lbs/100 Lbs Coal Fed)						
Total Solids	0.20		1.1	0.44	0.04	0.21
-50+100 Mesh Solids	0.13		-	0.34	-	-
Wall Scale	0.006		-	0.005	-	-
Overall Composition, Wt%						
Ash (ASTM, SO <sub>3</sub> -free)	75.9			59.6		
(by TGA)	79.4			64.7		
CaCO <sub>3</sub> (by TGA)	30.2			48.6		
(by Acid Evolution of CO <sub>2</sub> )	42.5			56.5		
Carbon	6.38			14.97		
Hydrogen	0.79			0.89		
H/C Atomic Ratio	1.49			0.72		
Scale Composition, Wt%						
Ash (by TGA)	74.4			56.0		
CaCO <sub>3</sub> (by TGA)	48.3			36.4		
X-ray Diffraction Analyses	Calcite Vaterite FeS			Calcite Vaterite FeS Kaolinite SiO <sub>2</sub>		
Major Elements, Wt%						
(by X-ray scan of cross section)						
Al	0.4-4.2	I N S P E C T E D	A N A L Y Z E D	0.8-1.4	A N A L Y Z E D	A N A L Y Z E D
Si	0.6-5.6			1.5-3.1		
S	0.2-25.8			0.1-1.5		
Ca	0.5-29.6			15.9-28.8		
Ti	0.3-2.4			0.1-0.6		
Fe	1.8-43.9			1.7-4.6		
-50+100 Mesh Solids Comp., Wt%						
Ash (ASTM, SO <sub>3</sub> -Free)	74.3	N O T	N O T	58.8	N O T	N O T
(by TGA)	78.4			64.1		
CaCO <sub>3</sub> (by TGA)	31.4			53.3		
(by Acid Evolution of O <sub>2</sub> )	42.2			62.0		
Carbon	7.37			14.02		
Hydrogen	0.82			0.80		
H/C Atomic Ratio	1.33			0.68		
X-ray Diffraction Analyses	Calcite Vaterite FeS Kaolinite SiO <sub>2</sub>			Calcite Vaterite FeS Kaolinite SiO <sub>2</sub>		
Major Elements, Wt%						
(by X-ray scan of cross section)						
Al	0.1-26.6			0.9-11.0		
Si	0.3-33.5			1.2-11.0		
S	0.1-30.4			0.1-1.9		
Ca	0.3-63.7			5.6-32.5		
Ti	0.1-3.3			0.3-1.7		
Fe	0.3-47.8			0.6-3.7		
Yield Periods Completed						
	625	632	635	638	640	643
	626	633	636	639	641	644
	627	634	637		642	645
	628					646
	629					647
	630					
	631					

Table A-7

## AVERAGE LIQUEFACTION YIELDS FOR BOTTOMS RECYCLE OF WYODAK COAL

Feed	Coal Only	Bottoms Only	Once-through 60-Minute CLPP Btms		Coal Only	Once-through 25-Minute CLPP Btms	
	606-608	621,622	603-605	617-620	614-616	610-613	623-624
<u>Yield Periods</u>	840	840	840	840	840	840	840
<u>Temperature, °F</u>	1500	1500	1500	1500	1500	1500	1500
<u>Pressure, psig</u>	60	60	60	60	25	25	25
<u>Residence Time, Minutes</u>	1.6/1/0	1.6/0/1	1.6/1/0.5	2.4/1/0.5	1.6/1/0	1.6/1/0.5	2.4/1/0.5
<u>Nominal Solvent/Coal/Bottoms</u>	1.60/1/0	1.60/0/1	1.58/1/0.52	2.40/1/0.50	1.60/1/0	1.54/1/0.56	2.40/1/0.50
<u>Actual Solvent/Coal/1000 °F+ Btms</u>							
<u>Yields</u>							
<u>Lbs/100 Lbs Dry Coal + 1000°F+ Btms</u>							
H <sub>2</sub>	-	-	-2.78	-3.97	-	-2.63	-3.02
H <sub>2</sub> O	-	-	8.41	10.15	-	9.20	9.73
CO	-	-	1.07	1.47	-	0.88	1.43
CO <sub>2</sub>	-	-	4.20	2.87	-	4.22	2.93
NH <sub>3</sub>	-	-	0.36	0.42	-	0.29	0.34
H <sub>2</sub> S	-	-	0.31	0.41	-	0.37	0.42
C <sub>1</sub>	-	-	3.26	3.31	-	2.52	2.05
C <sub>2</sub>	-	-	2.50	2.26	-	1.55	1.66
C <sub>3</sub>	-	-	2.40	2.47	-	1.49	1.76
C <sub>4</sub> -400°F	-	-	14.01	21.95	-	13.37	17.93
400-700°F	-	-	3.02	2.10	-	2.70	4.76
700-1000°F	-	-	1.61	1.93	-	2.19	2.22
1000°F+	-	-	61.63	54.63	-	63.85	57.79
Total			100.00	100.00		100.00	100.00
C <sub>1</sub> -C <sub>3</sub>	-	-	8.16	8.04	-	5.56	5.47
C <sub>4</sub> -1000°F	-	-	18.64	25.98	-	18.26	24.91
<u>Lbs/100 Lbs Dry Coal</u>							
H <sub>2</sub>	-3.93	-	-4.22	-5.96	-2.95	-4.11	-4.53
H <sub>2</sub> O	12.73	-	12.78	15.23	12.11	14.38	14.60
CO	1.23	-	1.63	2.21	1.35	1.37	2.15
CO <sub>2</sub>	6.85	-	6.38	4.30	5.91	6.60	4.39
NH <sub>3</sub>	0.44	-	0.55	0.63	0.35	0.45	0.51
H <sub>2</sub> S	0.54	-	0.47	0.62	0.52	0.58	0.63
C <sub>1</sub>	3.59	-	4.95	4.96	2.21	3.94	3.08
C <sub>2</sub>	2.89	-	3.80	3.39	1.92	2.42	2.49
C <sub>3</sub>	2.99	-	3.65	3.71	1.97	2.33	2.64
C <sub>4</sub> -400°F	19.81	-	21.27	32.94	16.34	20.90	26.90
400-700°F	3.21	-	4.59	3.15	3.14	4.22	7.14
700-1000°F	5.05	-	2.45	2.90	5.50	3.42	3.33
1000°F+	44.60	-	41.70	31.92	51.63	43.50	36.67
Total	100.00		100.00	100.00	100.00	100.00	100.00
C <sub>1</sub> -C <sub>3</sub>	9.47	-	12.40	12.06	6.10	8.69	8.21
C <sub>4</sub> -1000°F	28.07	-	28.31	38.98	24.98	28.54	37.38
<u>Lbs/100 Lbs 1000°F+ Btms</u>							
H <sub>2</sub>	-	-2.52	-0.57	-4.05	-	-2.06	-3.16
H <sub>2</sub> O	-	2.56	0.09	5.00	-	4.03	4.97
CO	-	0.44	0.77	1.95	-	0.04	1.59
CO <sub>2</sub>	-	0.06	-0.91	-5.08	-	1.22	-3.02
NH <sub>3</sub>	-	0.34	0.21	0.38	-	0.18	0.32
H <sub>2</sub> S	-	0.06	-0.13	0.15	-	0.10	0.22
C <sub>1</sub>	-	2.35	2.62	2.75	-	3.07	1.73
C <sub>2</sub>	-	1.72	1.75	1.00	-	0.89	1.14
C <sub>3</sub>	-	1.76	1.27	1.43	-	0.64	1.34
C <sub>4</sub> -400°F	-	12.08	2.84	26.23	-	8.10	21.11
400-700°F	-	0.31	2.65	-0.12	-	1.92	8.00
700-1000°F	-	1.28	-5.02	-4.30	-	-3.69	-4.33
1000°F+	-	79.56	94.43	74.66	-	85.56	70.09
Total		100.00	100.00	100.00		100.00	100.00
C <sub>1</sub> -C <sub>3</sub>	-	5.83	5.64	5.18	-	4.60	4.21
C <sub>4</sub> -1000°F	-	13.67	0.48	21.80	-	6.33	24.77

TABLE A-8

## RCLU YIELD PERIOD SUMMARY OF LIQUEFACTION-ONLY YIELDS FOR BURNING STAR NO. 2 MINE COAL

Yield Period Number	580	581	582	583	584	585	586	587	588	589	590	591
<u>Liquefaction Yields,</u> <u>Wt% on Dry Coal</u>												
Hydrogen	-1.04	-0.92			-0.93	-1.08	-1.10	-1.84	-1.70	-1.60	-2.44*	-1.70
Water	6.95	6.85			6.52	5.95	5.76	5.87	5.92	6.81	7.02	6.14
Carbon Oxides	1.39	1.55	No	No	1.32	1.22	1.43	1.49	1.68	1.79	1.61	1.66
Hydrogen Sulfide	1.18	1.58	Samples	Samples	1.17	1.36	1.37	1.99	2.02	2.08	1.07	1.85
Ammonia	0.01	0.02	for	for	0.04	0.16	0.01	0.21	0.13	0.13	0.20	0.24
C <sub>1</sub> -C <sub>3</sub> Gas	6.55	7.63	Analyses	Analyses	3.51	4.23	4.49	11.06	10.95	11.47	6.33	6.25
C <sub>4</sub> -1000°F	24.47	23.84			24.44	24.97	26.21	25.17	21.49	20.40	27.75	27.81
1000°F+	60.49	59.45			63.93	63.19	61.83	56.05	59.51	58.92	58.46	57.75
Yield Period Number	592	593	594	595	596	597	598	599	600	601	602	
<u>Liquefaction Yields,</u> <u>Wt% on Dry Coal</u>												
Hydrogen	-1.53	-1.80	-1.88	-1.49	-0.62*	-1.45	-1.44	-1.17	-1.07	-1.12	-1.27	
Water	6.34	5.79	7.32	7.14	4.65	6.10	5.92	6.35	6.23	5.85	5.67	
Carbon Oxides	1.35	1.54	1.38	1.55	1.22	1.17	1.17	1.16	1.55	1.64	1.47	
Hydrogen Sulfide	2.07	2.14	1.41	1.44	1.33	1.76	1.69	1.67	1.63	1.91	1.64	
Ammonia	0.18	0.21	0.27	0.13	0.07	0.11	0.04	0.03	0.04	0.02	0.01	
C <sub>1</sub> C <sub>3</sub> Gas	5.65	9.90	8.84	9.98	5.52	5.69	5.56	6.92	6.94	7.18	6.95	
C <sub>4</sub> -1000 F	26.69	32.09	32.17	29.51	28.56	28.58	29.29	25.28	28.29	26.81	24.57	
1000 F+	59.25	50.13	50.49	51.74	59.27	58.04	57.77	59.76	56.39	57.71	60.96	

\*Value is questionable

## Appendix B

### TABULATED DATA - PRODUCT QUALITY STUDIES

This appendix contains tabulated data for product quality studies discussed in Laboratory Process Research and Development, Section 5 of this report.

TABLE B-1

## ANALYTICAL INSPECTION OF EDS WYODAK LIQUIDS

Test	IBP/350°F		350/650°F	650/1000°F	350/1000°F	350°F+
	Raw	Partially Hydrotreated	Raw	Raw	F.O. Blend	F.O. Blend
C, Wt.%	85.29	86.84	86.4	88.75	88.64	89.21
H, Wt.%	11.95	12.17	10.22	7.13	8.13	7.33
N, Wt.%	0.11	0.07	0.27	0.98	0.48	0.86
S, Wt.%	0.11	0.006	0.03	0.13	0.10	0.18
O, Wt.%	2.55		1.99			
Ash, Wt.%				0.03		0.07
Gravity, °API	40.95	41.5	20.6	1.15	5.7	1.12
Flash Pt., °F		73	152	435	158	200
Pour Pt., °F		-35	-35	115	+45	+75
Kinematic Viscosity						
cs @ 40°C		0.72	1.84	---	10.4	2922
cs @ 100°C		0.43	0.82	385	2.3	26
Water Content, Wt.%		0.05	0.10	0.05	0.08	0.05
Con. Carbon, Wt.%	0.0	0.0	0.06	17.2	2.85	17
Aromatics, Wt.%, (By Fla)	23.7	36.8	64.2			
Major Metals	---	---	---	Fe, Si		Fe, Si, Ca, Al
Trace Metals, ppm						
Fe	1.5					
Na	3.4		5.5			
K	0.2		24.0			
R.O.N	77.3					
M.O.N.	72.2					
Bromine No.	22.9	2.86				
Reid V.P.	1.9	1.72				
Heating Value, BTU/lb			18,085			

TABLE B-2  
STORAGE STABILITY STUDIES  
EDS WYODAK LIQUIDS

		<u>Vis., cSt</u>		<u>Con Carbon</u>	<u>SHF<sup>(1)</sup></u>	
		<u>40°C</u>	<u>100°C</u>	<u>Wt %</u>		
<u>RSFO Reference</u> <sup>(2)</sup>	Start	1005	40.8	14.53	0.05	
	<u>150°F</u>	1 mo.	1516	50.2	15.14	0.06
		2 mo.	1869	57.1	13.49	0.01
		3 mo.	3165	57.3	15.20	0.01
	<u>210°F</u>	1 mo.	2127	59.9	14.63	0.11
<u>IBP/350°F</u>	Start	0.72	0.43	0.00	0.02	
	<u>150°F</u>	1 mo.	0.75	0.47	0.02	0.02
		2 mo.	0.75	0.45	0.03	0.00
		3 mo.	0.76	0.45	0.03	0.00
<u>350/650°F</u>	Start	1.84	0.82	0.06	0.01	
	<u>150°F</u>	1 mo.	2.01	0.86	0.39	0.03
		2 mo.	2.06	0.87	0.66	0.01
		3 mo.	2.07	0.88	0.73	0.01
	<u>210°F</u>	1 mo.	2.13	0.89	0.88	0.07
<u>350/1000°F</u>	Start	10.39	2.29	2.85	0.07	
	<u>150°F</u>	1 mo.	12.35	2.46	3.61	--
		2 mo.	13.83	2.55	4.05	0.13
		3 mo.	14.79	2.80	4.36	0.15
	<u>210°F</u>	1 mo.	15.00	2.74	4.86	0.29
<u>75/25 Mix - 350°F<sup>+</sup></u> <u>and 350/1000°F</u>	Start	2922	26.00	17.01	0.22	
	<u>210°F</u>	1 mo.	6926	32.78	17.11	0.15

(1) Sediment by hot filtration is an Exxon proprietary test.

(2) Regular sulfur petroleum fuel oil.



TABLE B-3  
COMPATIBILITY STUDIES  
EDS WYODAK LIQUIDS

	<u>Sediment by Hot Filtration(2)</u>	<u>Comments and Observations</u>
	<u>Wt %</u>	
<u>EDS 350/1000°F</u>	0.07	
with 50% RSFO	0.64	
with 50% HCO <sup>(1)</sup>	1.13	Did not appear to be totally incompatible under microscope.
with 50% Home Heating Oil	0.14	Large globs of preci- pitate formed.
<u>350°F<sup>†</sup></u>	0.22	Dark viscous liquid.
with 50% RSFO	0.07	Appeared incompatible; black tar in bottom of beaker.
with 50% HCO	0.14	Appeared compatible.
with 50% Home Heating Oil	0.13	Appeared incompatible; soft tar in bottom of beaker.
<u>RCLU-1, YP551 A-1 Bottoms</u>		
with 90% RSFO	0.45	

(1) Hydrogenated creosote oil from Baytown.

(2) Exxon proprietary test. Target 0.15-0.25 depending on viscosity and sulfur content.

Table B-4

INSPECTIONS OF EDS DISTILLATE TESTED AS  
STATIONARY TURBINE FUEL BY WESTINGHOUSE  
ILLINOIS #6 MONTEREY COAL

	<u>Hydrotreated</u> <u>MPSS Solvent</u>	<u>350/650°F Portion</u> <u>(Retest Fuel)</u>
<u>Physical Properties</u>		
Gravity, °API	16.0	17.1
Flash (PM), °F	100	122
Pour Point, °F	<-36	<-36
Conradson Carbon, wt %	0.03	0.03 (10% btms.)
ASTM D-2382, BTU/Lb	18,340	18,400
Viscosity		
+ 40°F.		5.65 cs
+ 100°F	35.0 SSU	2.34 cs
+ 212°F	29.4 SSU	1.0 cs
Distillation (D-1160)		
5%	312	409
50%	454	499
95%	695	602
<u>Chemical Characterization</u>		
Carbon, wt %	89.53	89.12
Hydrogen, wt %	9.95	10.16
Nitrogen, wt %	0.081	0.044
Oxygen, wt %	0.32	0.48
Sulfur, wt %	0.031	0.02
Ash, wt %	0.001	0.001
Carbon Distribution, % Aromatic Ring (NMR)	48.4	46.8

FIGURE B-1

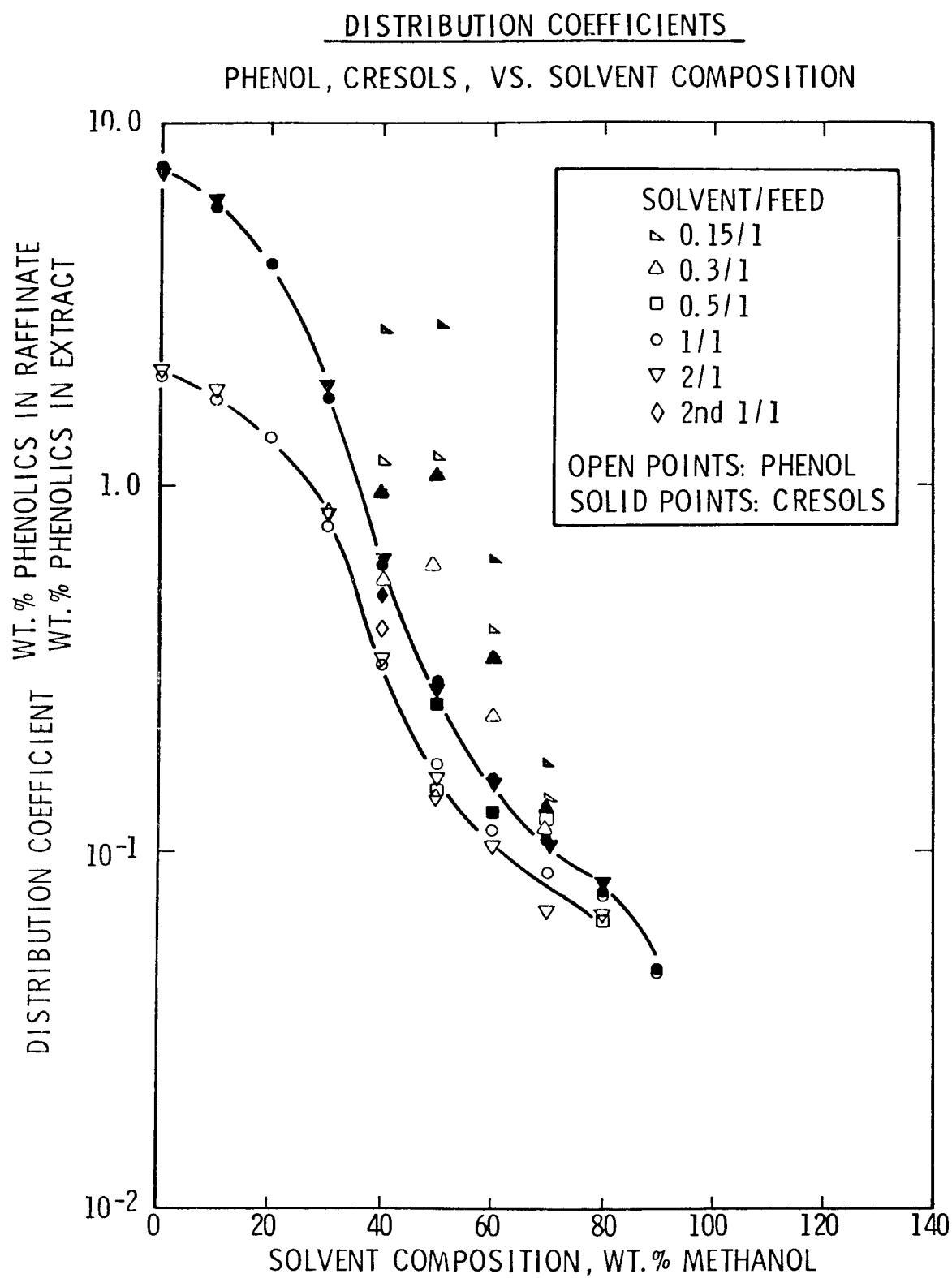
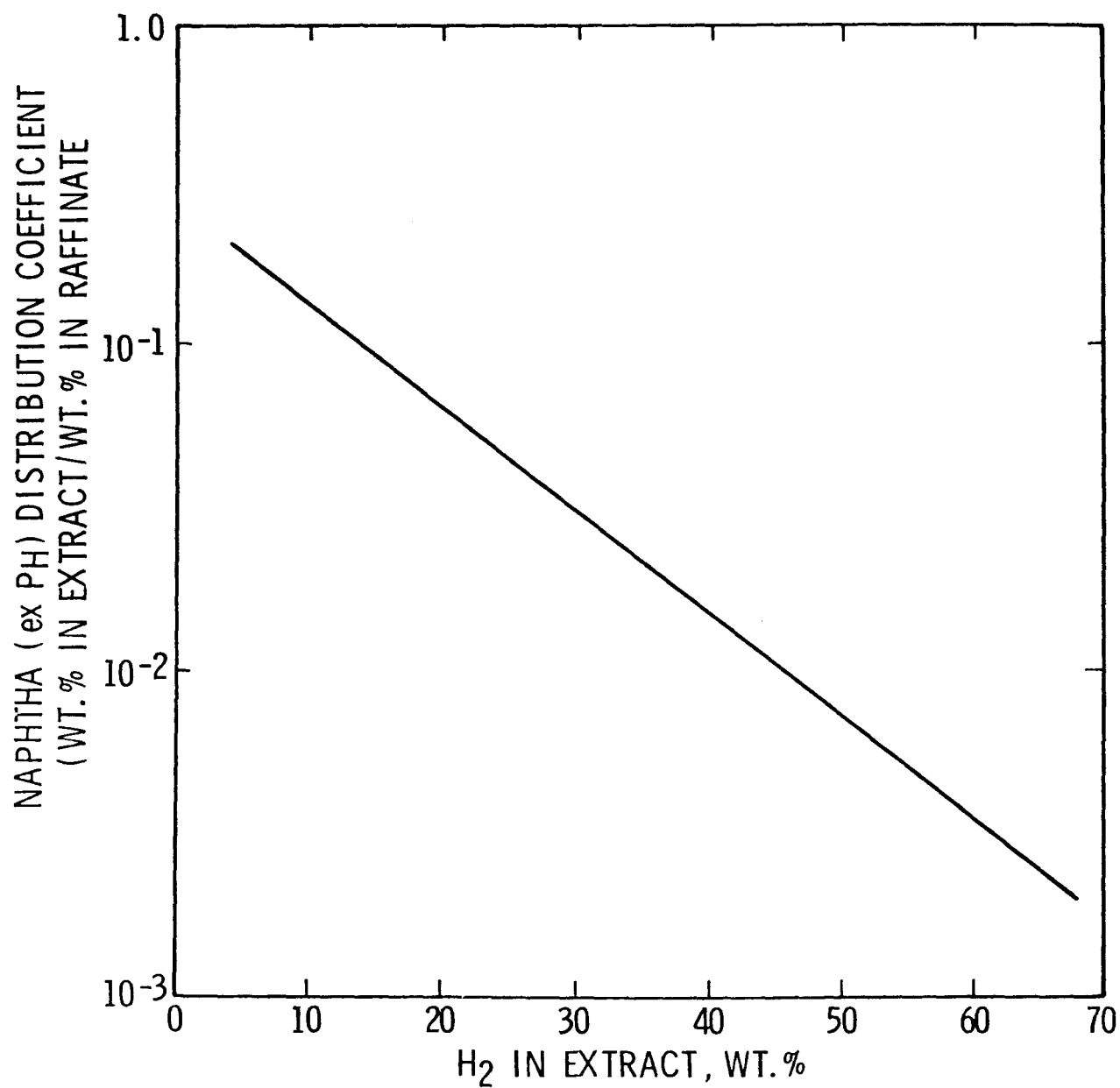


FIGURE B-2

NAPHTHA (NON-PHENOLIC) DISTRIBUTION COEFFICIENT  
VS.  
WATER CONTENT OF EXTRACT



### Appendix C

Appendix C contains data for Yield Tests 164-189 for the Continuous Stirred Coking Unit (CSCU). The information contained in these tables includes:

- Run Conditions
- Product Yields
- Liquid Product Distillations
- Gas Analysis
- Elemental and Material Balances

For each run, the results are presented on two bases; microlube distillation basis and HiVac-C distillation basis.

## CSCU RUN-164

TEMPERATURE, DEG. F                      RV + 141.00  
 PRESSURE, PSIG                              5.10  
 FEED SOURCE    RCLU YP- 527( 840 F/ 40 MIN.)  
                  PITTSBURGH #8 ARKWRIGHT BOTTOMS  
 FEED, GM.                                    5362.00  
 FEED RATE, GM/MIN.                        14.90  
 STEAM FEED RATIO, % ON FEED            28.86  
 VAPOR RESIDENCE TIME, SEC.              RV X    2.28  
 PRODUCT YIELD  
 COKE,    GMS.                                3224.96  
           % ON FEED                           60.14  
 LIQUID, GMS.                                1887.18  
           % ON FEED                           35.20  
 GAS,    GMS.                                249.87  
           % ON FEED                           4.66  
 MATERIAL BALANCE, %                      103.57  
 WATER BALANCE, %                          96.24

## GAS ANALYSES, MOL %/WT %

H2    59.49/12.19  
 CO    1.41/ 4.05  
 CO2     2.61/11.76  
 H2S     0.59/ 2.05  
 NH3     1.38/ 2.41  
 CH4     28.28/46.37  
 C2H6    3.63/11.16  
 C2H4    0.94/ 2.69  
 C3H8    0.70/ 3.16  
 C3H6    0.97/ 4.16  
 MOLECULAR WEIGHT                          9.76  
 HEATING VALUE, BTU/CU FT                612.46

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.                              0.37  
 400-700 DEG. F.                             2.76  
 700-1000 DEG. F.                            3.94  
 1000 DEG F.+                                28.09  
 1000 DEG F.-                                7.07  
 % 1000 F- IN FEED                        6.60

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.  
 C/H/S/N, WT %                              84.98/13.73/ 0.08/ 0.18  
 ATOMIC H/C                                   1.94  
 400-700 DEG F.  
 C/H/S/N, WT %                              87.75/ 7.81/ 0.35/ 0.81  
 ATOMIC H/C                                   1.07  
 700-1000 DEG F.  
 C/H/S/N, WT %                              88.45/ 6.39/ 0.46/ 1.18  
 ATOMIC H/C                                   0.87  
 1000 DEG F.-  
 C/H/S/N, WT %                              88.00/ 7.32/ 0.40/ 0.98  
 ATOMIC H/C                                   1.00  
 BTU/LB                                        16844.62  
 LB S/MBTU                                    0.24  
 1000 DEG F.+  
 C/H/S/N, WT %                              88.75/ 5.30/ 0.87/ 1.65  
 ATOMIC H/C                                   0.72  
 BTU/LB                                        15738.79  
 LB S/MBTU                                    0.55

## ELEMENTAL BALANCE

	C	H	S	N	ASH
<b>FEED</b>					
WT %	79.50	4.81	1.32	1.67	10.46
GM	4262.79	257.91	70.78	89.55	560.86
% ON FEED	100.00	100.00	100.00	100.00	100.00
<b>LIQUID PRODUCT</b>					
WT %	88.51	5.70	0.77	1.51	0.0
GM	1670.26	107.62	14.61	28.55	0.0
% ON FEED	39.18	41.73	20.65	31.88	0.0
<b>GASEOUS PRODUCT</b>					
WT %	57.11	28.12	1.93	1.99	0.0
GM	142.69	70.25	4.82	4.96	0.0
% ON FEED	3.35	27.24	6.81	5.54	0.0
<b>COKE</b>					
WT %	75.63	2.69	1.65	2.36	17.38
GM	2439.04	86.78	53.29	76.12	560.59
% ON FEED	57.22	33.65	75.29	85.01	99.95
<b>ELEMENTAL BALANCE, %</b>					
	99.75	102.62	102.74	122.43	99.95

ATOMIC RATIO OF H/C: FEED-0.73 GAS-5.91 LIQUID-0.77 COKE-0.39

## CSCU RUN-164

TEMPERATURE, DEG. F	RV + 141.00
PRESSURE, PSIG	5.10
FEED SOURCE	RCLU YP- 527( 840 F/ 40 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	5362.00
FEED RATE, GM/MIN.	14.90
STEAM FEED RATIO, % ON FEED	28.86
VAPOR RESIDENCE TIME, SEC.	RV X 2.28
PRODUCT YIELD	
COKE, GMS.	3226.89
% ON FEED	60.18
LIQUID, GMS.	1885.25
% ON FEED	35.16
GAS, GMS.	249.87
% ON FEED	4.66
MATERIAL BALANCE, %	103.57
WATER BALANCE, %	96.24

## GAS ANALYSES, MOL %/WT %

H2	59.49/12.19
CO	1.41/ 4.05
CO2	2.61/11.76
H2S	0.59/ 2.05
NH3	1.38/ 2.41
CH4	28.28/46.37
C2H6	3.63/11.16
C2H4	0.94/ 2.69
C3H8	0.70/ 3.16
C3H6	0.97/ 4.16
MOLECULAR WEIGHT	9.76
HEATING VALUE, BTU/CU FT	612.46

DISTILLATION CUT(HIVA-C) OF LIQUID, % ON FEED	
C4-400 DEG. F.	0.37
400-700 DEG. F.	2.76
700-1000 DEG. F.	16.42
1000 DEG F.+	15.61
1000 DEG F.-	19.54
% 1000 F- IN FEED	9.95

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	84.98/13.73/ 0.08/ 0.18
ATOMIC H/C	1.94
400-700 DEG F.	
C/H/S/N, WT %	87.75/ 7.81/ 0.35/ 0.81
ATOMIC H/C	1.07
700-1000 DEG F.	
C/H/S/N, WT %	88.88/ 6.33/ 0.40/ 1.41
ATOMIC H/C	0.85
1000 DEG F.-	
C/H/S/N, WT %	88.65/ 6.68/ 0.39/ 1.30
ATOMIC H/C	0.90
BTU/LB	16565.73
LB S/MBTU	0.23
1000 DEG F.+	
C/H/S/N, WT %	89.30/ 4.72/ 1.14/ 1.66
ATOMIC H/C	0.63
BTU/LB	15495.42
LB S/MBTU	0.73

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.14	4.79	1.56	1.67	10.46
GM	4243.48	256.84	83.65	89.55	560.86
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	88.92	5.81	0.72	1.46	0.0
GM	1676.31	109.51	13.56	27.58	0.0
% ON FEED	39.50	42.64	16.21	30.80	0.0
GASEOUS PRODUCT					
WT %	57.11	28.12	1.93	1.99	0.0
GM	142.69	70.25	4.82	4.96	0.0
% ON FEED	3.36	27.35	5.76	5.54	0.0
COKE					
WT %	75.59	2.69	1.65	2.36	17.42
GM	2439.35	86.79	53.29	76.13	562.18
% ON FEED	57.48	33.79	63.71	85.02	100.24
ELEMENTAL BALANCE, %	100.35	103.78	85.68	121.37	100.24

ATOMIC RATIO OF H/C: FEED-0.73 GAS-5.91 LIQUID-0.78 COKE-0.38

## CSCU RUN-165

TEMPERATURE, DEG. F                      RV + 92.00  
 PRESSURE, PSIG                              3.20  
 FEED SOURCE    HCLU YP- 527( 840 F/ 40 MIN.)  
                  PITTSBURGH #8 ARKWRIGHT BOTTOMS  
 FEED, GM.                                      5970.00  
 FEED RATE, GM/MIN.                          19.80  
 STEAM FEED RATIO, % ON FEED              23.82  
 VAPOR RESIDENCE TIME, SEC.                RV X 2.12  
 PRODUCT YIELD  
   COKE,    GMS.                              3688.18  
             % ON FEED                          61.78  
   LIQUID, GMS.                              2015.54  
             % ON FEED                          33.76  
   GAS,     GMS.                              266.28  
             % ON FEED                          4.46  
 MATERIAL BALANCE, %                        100.29  
 WATER BALANCE, %                            97.70

## GAS ANALYSES, MOL %/WT %

H2    59.71/12.27  
 CO    1.42/ 4.08  
 CO2     2.62/11.83  
 H2S     0.59/ 2.07  
 NH3     1.02/ 1.78  
 CH4     28.39/46.67  
 C2H6     3.64/11.23  
 C2H4     0.94/ 2.71  
 C3H8     0.70/ 3.18  
 C3H6     0.97/ 4.19  
 MOLECULAR WEIGHT                            9.73  
 HEATING VALUE, BTU/CU FT                  614.81

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.                              0.31  
 400-700 DEG. F.                              2.37  
 700-1000 DEG. F.                            4.74  
 1000 DEG F.+                                26.34  
 1000 DEG F.-                                7.42  
 % 1000 F- IN FEED                          6.60

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.  
 C/H/S/N, WT %                              84.62/14.53/ 0.04/ 0.09  
 ATOMIC H/C                                   2.06  
 400-700 DEG F.  
 C/H/S/N, WT %                              87.87/ 7.83/ 0.33/ 0.72  
 ATOMIC H/C                                   1.07  
 700-1000 DEG F.  
 C/H/S/N, WT %                              89.48/ 6.48/ 0.48/ 0.99  
 ATOMIC H/C                                   0.87  
 1000 DEG F.-  
 C/H/S/N, WT %                              88.76/ 7.25/ 0.41/ 0.87  
 ATOMIC H/C                                   0.98  
                  BTU/LB                              16946.02  
                  LB S/MBTU                            0.24  
 1000 DEG F.+  
 C/H/S/N, WT %                              89.35/ 5.36/ 0.77/ 1.49  
 ATOMIC H/C                                   0.72  
                  BTU/LB                              15879.05  
                  LB S/MBTU                            0.49

## ELEMENTAL BALANCE

	C	H	S	N	ASH
<b>FEED</b>					
WT %	79.14	4.79	1.56	1.67	9.78
GM	4724.66	285.96	93.13	99.70	583.87
% ON FEED	100.00	100.00	100.00	100.00	100.00
<b>LIQUID PRODUCT</b>					
WT %	89.22	5.77	0.69	1.35	0.0
GM	1798.31	116.32	13.96	27.27	0.0
% ON FEED	38.06	40.68	14.98	27.36	0.0
<b>GASEOUS PRODUCT</b>					
WT %	57.47	28.18	1.94	1.46	0.0
GM	153.04	75.05	5.18	3.90	0.0
% ON FEED	3.24	26.24	5.56	3.91	0.0
<b>COKE</b>					
WT %	76.78	2.30	1.70	1.36	17.65
GM	2831.72	84.72	62.75	50.20	650.95
% ON FEED	59.93	29.62	67.38	50.35	111.49
<b>ELEMENTAL BALANCE, %</b>					
	101.24	96.54	87.92	81.62	111.49

ATOMIC RATIO OF H/C: FEED-0.73 GAS-5.88 LIQUID-0.78 COKE-0.41



## CSCU RUN-165

TEMPERATURE, DEG. F	RV + 92.00
PRESSURE, PSIG	5.20
FEED SOURCE	RCLU YP- 527( 840 F/ 40 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	5970.00
FEED RATE, GM/MIN.	19.80
STEAM FEED RATIO, % ON FEED	23.82
VAPOR RESIDENCE TIME, SEC.	RV X 2.12
PRODUCT YIELD	
COKE, GMS.	3668.95
X ON FEED	61.46
LIQUID, GMS.	2034.77
X ON FEED	34.08
GAS, GMS.	266.28
X ON FEED	4.46
MATERIAL BALANCE, %	100.29
WATER BALANCE, %	97.70

## GAS ANALYSES, MOL %/WT %

H2	59.71/12.27
CO	1.42/ 4.08
CO2	2.62/11.83
H2S	0.59/ 2.07
NH3	1.02/ 1.78
CH4	28.39/46.67
C2H6	3.64/11.23
C2H4	0.94/ 2.71
C3H8	0.70/ 3.18
C3H6	0.97/ 4.19
MOLECULAR WEIGHT	9.73
HEATING VALUE, HTU/CU FT	614.81

## DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.31
400-700 DEG. F.	2.30
700-1000 DEG. F.	16.51
1000 DEG F.+	14.89
1000 DEG F.-	19.12
X 1000 F- IN FEED	9.95

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	84.62/14.53/ 0.04/ 0.09
ATOMIC H/C	2.06
400-700 DEG F.	
C/H/S/N, WT %	87.87/ 7.83/ 0.33/ 0.72
ATOMIC H/C	1.07
700-1000 DEG F.	
C/H/S/N, WT %	89.48/ 6.48/ 0.48/ 0.99
ATOMIC H/C	0.87
1000 DEG F.-	
C/H/S/N, WT %	89.21/ 6.77/ 0.45/ 0.94
ATOMIC H/C	0.91
BTU/LB	16729.68
LB S/MBTU	0.27
1000 DEG F.+	
C/H/S/N, WT %	89.33/ 5.35/ 0.77/ 1.49
ATOMIC H/C	0.72
BTU/LB	15874.48
LB S/MBTU	0.49

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.14	4.79	1.56	1.67	9.78
GM	4724.66	285.96	93.13	99.70	583.87
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	89.08	6.14	0.59	1.18	0.0
GM	1812.64	124.92	12.04	24.01	0.0
% ON FEED	38.37	43.69	12.93	24.08	0.0
GASEOUS PRODUCT					
WT %	57.47	28.18	1.94	1.46	0.0
GM	153.04	75.05	5.18	3.90	0.0
% ON FEED	3.24	26.24	5.56	3.91	0.0
COKE					
WT %	77.09	2.31	1.71	1.37	17.32
GM	2828.24	84.61	62.68	50.14	635.44
% ON FEED	59.86	29.59	67.30	50.29	108.83
ELEMENTAL BALANCE, %	101.47	99.52	85.79	78.29	108.83

ATOMIC RATIO OF H/C: FEED-0.73 GAS-5.88 LIQUID-0.83 COKE-0.37

## CSCU RUN-166

TEMPERATURE, DEG. F	RV + 64.40
PRESSURE, PSIG	8.50
FEED SOURCE	KCLU YP- 530( 840 F/100 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	5900.00
FEED RATE, GM/MIN.	19.70
STEAM FEED RATIO, % ON FEED	17.77
VAPOR RESIDENCE TIME, SEC.	RV X 3.34
PRODUCT YIELD	
COKE, GMS.	3959.16
% ON FEED	67.10
LIQUID, GMS.	1701.49
% ON FEED	28.84
GAS, GMS.	239.35
% ON FEED	4.06
MATERIAL BALANCE, %	100.12
WATER BALANCE, %	92.12

## GAS ANALYSES, MOL %/WT %

H2	66.32/16.29
CO	0.35/ 1.22
CO2	0.83/ 4.46
H2S	0.33/ 1.37
NH3	1.01/ 2.11
CH4	24.95/49.03
C2H6	3.95/14.55
C2H4	0.57/ 1.96
C3H8	1.02/ 5.53
C3H6	0.67/ 3.48
MOLECULAR WEIGHT	8.14
HEATING VALUE, BTU/CU FT	585.93

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.14
400-700 DEG. F.	1.04
700-1000 DEG. F.	3.37
1000 DEG F.+	24.29
1000 DEG F.-	4.55
% 1000 F- IN FEED	8.70

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	83.82/15.92/ 0.0 / 0.0
ATOMIC H/C	2.28
400-700 DEG F.	
C/H/S/N, WT %	88.98/ 7.43/ 0.19/ 0.81
ATOMIC H/C	1.00
700-1000 DEG F.	
C/H/S/N, WT %	89.97/ 6.27/ 0.38/ 1.12
ATOMIC H/C	0.84
1000 DEG F.-	
C/H/S/N, WT %	89.55/ 6.84/ 0.32/ 1.01
ATOMIC H/C	0.92
BTU/LB	16835.15
LB S/MBTU	0.19
1000 DEG F.+	
C/H/S/N, WT %	90.27/ 5.36/ 1.48/ 1.59
ATOMIC H/C	0.71
BTU/LB	16150.32
LB S/MBTU	0.91

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	78.42	4.40	1.41	1.56	12.55
GM	4626.78	259.60	83.19	92.04	740.45
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	90.16	5.59	1.30	1.50	0.0
GM	1534.05	95.11	22.04	25.47	0.0
% ON FEED	33.16	36.64	26.49	27.68	0.0
GASEOUS PRODUCT					
WT %	59.33	33.69	1.29	1.74	0.0
GM	142.01	80.65	3.09	4.16	0.0
% ON FEED	3.07	31.07	3.72	4.52	0.0
COKE					
WT %	68.12	2.34	1.53	0.97	14.46
GM	2697.10	92.56	60.64	38.30	572.63
% ON FEED	58.29	35.66	72.90	41.61	77.33
ELEMENTAL BALANCE, %	94.52	103.36	103.11	73.81	77.33

ATOMIC RATIO OF H/C: FEED-0.67 GAS-6.81 LIQUID-0.74 COKE-0.34

## CSCU RUN-166

TEMPERATURE, DEG. F	RV + 64.40
PRESSURE, PSIG	8.50
FEED SOURCE	RCLU YP- 530( 840 F/100 MIN.)
FEED, GM.	PITTSBURGH #8 ARKWRIGHT BOTTOMS 5900.00
FEED RATE, GM/MIN.	19.70
STEAM FEED RATIO, % ON FEED	17.77
VAPOR RESIDENCE TIME, SEC.	RV X 3.34
PRODUCT YIELD	
COKE, GMS.	3958.61
% ON FEED	67.10
LIQUID, GMS.	1702.04
% ON FEED	28.85
GAS, GMS.	239.35
% ON FEED	4.06
MATERIAL BALANCE, %	100.12
WATER BALANCE, %	92.12

## GAS ANALYSES, MOL %/WT %

H2	66.32/16.29
CO	0.35/ 1.22
CO2	0.83/ 4.46
H2S	0.33/ 1.37
NH3	1.01/ 2.11
CH4	24.95/49.03
C2H6	3.95/14.55
C2H4	0.57/ 1.96
C3H8	1.02/ 5.53
C3H6	0.67/ 3.48
MOLECULAR WEIGHT	8.14
HEATING VALUE, BTU/CU FT	585.93

## DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.14
400-700 DEG. F.	1.04
700-1000 DEG. F.	19.46
1000 DEG F.+	6.94
1000 DEG F.-	20.64
% 1000 F- IN FEED	12.35

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	83.82/15.92/ 0.0 / 0.0
ATOMIC H/C	2.28
400-700 DEG F.	
C/H/S/N, WT %	88.98/ 7.43/ 0.19/ 0.81
ATOMIC H/C	1.00
700-1000 DEG F.	
C/H/S/N, WT %	90.84/ 5.77/ 0.46/ 1.46
ATOMIC H/C	0.76
1000 DEG F.-	
C/H/S/N, WT %	90.70/ 5.92/ 0.44/ 1.42
ATOMIC H/C	0.78
BTU/LB	16499.71
LB S/MBTU	0.27
1000 DEG F.+	
C/H/S/N, WT %	90.66/ 4.81/ 0.74/ 1.71
ATOMIC H/C	0.64
BTU/LB	15797.97
LB S/MBTU	0.47

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	78.42	4.40	1.41	1.56	12.35
GM	4626.78	259.60	83.19	92.04	728.65
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	86.70	5.40	0.50	1.42	0.0
GM	1475.70	91.85	8.43	24.24	0.0
% ON FEED	31.89	35.38	10.14	26.34	0.0
GASEOUS PRODUCT					
WT %	59.33	33.69	1.29	1.74	0.0
GM	142.01	80.65	3.09	4.16	0.0
% ON FEED	3.07	31.07	3.72	4.52	0.0
COKE					
WT %	68.13	2.34	1.53	0.97	14.45
GM	2697.01	92.56	60.64	38.30	572.19
% ON FEED	58.29	35.65	72.90	41.61	78.53
ELEMENTAL BALANCE, %	93.26	102.10	86.75	72.47	78.53

ATOMIC RATIO OF H/C: FEED-0.67 GAS-6.81 LIQUID-0.75 COKE-0.35

## CSCU RUN-170

TEMPERATURE, DEG. F	HV + 65.00
PRESSURE, PSIG	6.00
FEED SOURCE	RCLU YP- 628( 840 F/ 40 MIN.)
FEED, GM.	WANDOAN BOTTOMS 5562.00
FEED RATE, GM/MIN.	11.60
STEAM FEED RATIO, % ON FEED	54.88
VAPOR RESIDENCE TIME, SEC.	HV X 2.08
PRODUCT YIELD	
COKE, GMS.	4257.16
% ON FEED	76.54
LIQUID, GMS.	1147.03
% ON FEED	20.62
GAS, GMS.	157.82
% ON FEED	2.84
MATERIAL BALANCE, %	100.22
WATER BALANCE, %	96.67

## GAS ANALYSES, MOL %/WT %

H2	55.14/10.24
CO	2.55/ 6.62
CO2	4.21/17.20
H2S	0.18/ 0.56
NH3	1.13/ 1.79
CH4	30.64/45.53
C2H6	4.07/11.34
C2H4	1.17/ 3.03
C3H8	0.76/ 3.09
C3H6	0.15/ 0.59
MOLECULAR WEIGHT	10.77
HEATING VALUE, BTU/CU FT	627.52

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.28
400-700 DEG. F.	1.74
700-1000 DEG. F.	4.79
1000 DEG F.+	13.81
1000 DEG F.-	6.81
% 1000 F- IN FEED	4.20

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	87.04/10.92/ 0.02/ 0.27
ATOMIC H/C	1.51
400-700 DEG F.	
C/H/S/N, WT %	88.79/ 8.33/ 0.03/ 0.40
ATOMIC H/C	1.13
700-1000 DEG F.	
C/H/S/N, WT %	89.73/ 7.00/ 0.05/ 0.65
ATOMIC H/C	0.94
1000 DEG F.-	
C/H/S/N, WT %	89.38/ 7.50/ 0.04/ 0.57
ATOMIC H/C	1.01
BTU/LB	17184.46
LB S/MBTU	0.03
1000 DEG F.+	
C/H/S/N, WT %	87.31/ 4.71/ 0.30/ 1.16
ATOMIC H/C	0.65
BTU/LB	14957.68
LB S/MBTU	0.20

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	52.14	3.49	0.23	0.69	38.03
GM	2900.03	194.11	12.79	38.38	2115.23
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	87.96	5.63	0.22	0.96	0.0
GM	1008.92	64.55	2.47	11.06	0.0
% ON FEED	34.79	33.25	19.33	28.82	0.0
GASEOUS PRODUCT					
WT %	56.38	25.32	0.53	1.47	0.0
GM	88.98	39.96	0.84	2.32	0.0
% ON FEED	3.07	20.59	6.53	6.05	0.0
COKE					
WT %	45.52	2.18	0.23	0.61	46.23
GM	1937.85	92.67	9.75	26.01	1968.15
% ON FEED	66.82	47.74	76.25	67.78	93.05
ELEMENTAL BALANCE, %	104.68	101.58	102.11	102.65	93.05
ATOMIC RATIO OF H/C:	FEED-0.80	GAS-5.39	LIQUID-0.77	COKE-0.60	

## CSCU RUN-170

TEMPERATURE, DEG. F	RV + 65.00
PRESSURE, PSIG	6.00
FEED SOURCE	RCLU YP- 628( 840 F/ 40 MIN.)
FEED, GM.	WANDOAN BOTTOMS 5562.00
FEED RATE, GM/MIN.	11.60
STEAM FEED RATIO, % ON FEED	54.88
VAPOR RESIDENCE TIME, SEC.	RV X 2.07
PRODUCT YIELD	
COKE, GMS.	4013.48
% ON FEED	72.16
LIQUID, GMS.	1390.71
% ON FEED	25.00
GAS, GMS.	157.82
% ON FEED	2.84
MATERIAL BALANCE, %	100.22
WATER BALANCE, %	96.67

## GAS ANALYSES, MOL %/WT %

H2	55.14/10.24
CO	2.55/ 6.62
CO2	4.21/17.20
H2S	0.18/ 0.56
NH3	1.13/ 1.79
CH4	30.64/45.53
C2H6	4.07/11.34
C2H4	1.17/ 3.03
C3H8	0.76/ 3.09
C3H6	0.15/ 0.59
MOLECULAR WEIGHT	10.77
HEATING VALUE, BTU/CU FT	627.52

## DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.29
400-700 DEG. F.	1.74
700-1000 DEG. F.	13.70
1000 DEG F.+	9.28
1000 DEG F.-	15.72
% 1000 F- IN FEED	9.40

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	87.11/10.82/ 0.02/ 0.27
ATOMIC H/C	1.49
400-700 DEG F.	
C/H/S/N, WT %	88.79/ 8.33/ 0.03/ 0.40
ATOMIC H/C	1.13
700-1000 DEG F.	
C/H/S/N, WT %	69.53/ 6.83/ 0.06/ 0.69
ATOMIC H/C	1.18
1000 DEG F.-	
C/H/S/N, WT %	71.98/ 7.07/ 0.06/ 0.65
ATOMIC H/C	1.18
BTU/LB	13396.79
LB S/MBTU	0.04
1000 DEG F.+	
C/H/S/N, WT %	88.10/ 5.54/ 0.21/ 1.12
ATOMIC H/C	0.75
BTU/LB	15664.01
LB S/MBTU	0.13

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	52.14	3.49	0.23	0.78	38.03
GM	2900.03	194.11	12.79	43.38	2115.23
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	77.97	6.50	0.11	0.83	0.0
GM	1084.39	90.43	1.58	11.48	0.0
% ON FEED	37.39	46.58	12.32	26.46	0.0
GASEOUS PRODUCT					
WT %	56.38	25.32	0.53	1.47	0.0
GM	88.98	39.96	0.84	2.32	0.0
% ON FEED	3.07	20.59	6.53	5.35	0.0
COKE					
WT %	47.56	2.27	0.24	0.60	43.82
GM	1908.81	91.28	9.61	24.02	1758.78
% ON FEED	65.82	47.02	75.11	55.37	83.15
ELEMENTAL BALANCE, %	106.28	114.19	93.95	87.18	83.15

ATOMIC RATIO OF H/C: FEED-0.80 GAS-5.39 LIQUID-1.00 COKE-0.44

## CSCU RUN-172

TEMPERATURE, DEG. F	RV + 198.80
PRESSURE, PSIG	9.70
FEED SOURCE	RCLU YP- 626( 840 F/ 40 MIN.)
	WANDOAN BOTTOMS
FEED, GM.	3986.00
FEED RATE, GM/MIN.	16.60
STEAM FEED RATIO, % ON FEED	18.55
VAPOR RESIDENCE TIME, SEC.	RV X 3.80
PRODUCT YIELD	
COKE, GMS.	3063.44
% ON FEED	76.85
LIQUID, GMS.	718.38
% ON FEED	18.02
GAS, GMS.	204.18
% ON FEED	5.12
MATERIAL BALANCE, %	96.29
WATER BALANCE, %	109.70

## GAS ANALYSES, MOL %/WT %

H2	53.84/ 8.87
CO	2.54/ 5.86
CO2	7.71/27.95
H2S	0.23/ 0.65
NH3	1.62/ 2.27
CH4	27.44/36.18
C2H6	3.51/ 8.69
C2H4	1.14/ 2.63
C3H8	0.69/ 2.52
C3H6	1.27/ 4.40
MOLECULAR WEIGHT	12.14
HEATING VALUE, BTU/CU FT	627.87

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.37
400-700 DEG. F.	1.66
700-1000 DEG. F.	3.74
1000 DEG F.+	12.25
1000 DEG F.-	5.77
% 1000 F- IN FEED	4.20

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.35/13.78/ 0.01/ 0.11
ATOMIC H/C	1.94
400-700 DEG F.	
C/H/S/N, WT %	89.56/ 7.60/ 0.04/ 0.57
ATOMIC H/C	1.02
700-1000 DEG F.	
C/H/S/N, WT %	90.61/ 6.14/ 0.06/ 0.78
ATOMIC H/C	0.81
1000 DEG F.-	
C/H/S/N, WT %	89.97/ 7.06/ 0.05/ 0.68
ATOMIC H/C	0.94
BTU/LB	17016.81
LB S/MBTU	0.03
1000 DEG F.+	
C/H/S/N, WT %	89.96/ 4.84/ 0.15/ 1.32
ATOMIC H/C	0.65
BTU/LB	15583.48
LB S/MBTU	0.09

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	54.20	3.61	0.21	0.78	36.66
GM	2160.41	143.89	8.37	31.09	1461.27
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	89.96	5.55	0.12	1.11	0.0
GM	646.29	39.85	0.83	8.01	0.0
% ON FEED	29.92	27.69	9.88	25.76	0.0
GASEOUS PRODUCT					
WT %	52.29	21.55	0.61	1.87	0.0
GM	106.77	44.01	1.25	3.82	0.0
% ON FEED	4.94	30.58	14.95	12.29	0.0
COKE					
WT %	42.98	2.11	0.20	0.54	55.11
GM	1316.54	64.77	6.03	16.57	1688.14
% ON FEED	60.94	45.01	71.98	53.29	115.53
ELEMENTAL BALANCE, %	95.80	103.29	96.81	91.34	115.53

ATOMIC RATIO OF H/C: FEED-0.80 GAS-4.95 LIQUID-0.74 COKE-0.51

CCCU RUN-172

TEMPERATURE, DEG. F. RV + 195.80  
 PRESSURE, PSIG 9.70  
 FEED SOURCE RECU YP- 626 (S40 F/ 40 MIN.)  
 FEED, GM. WANDON BOTTOMS 3956.00  
 FEED RATE, GM/MIN. 16.60  
 STEAM FEED RATIO, % ON FEED 18.55  
 VAPOR RESIDENCE TIME, SEC. RV X 3.80  
 PRODUCT YIELD  
 COKE, GMS. 3079.41  
 % ON FEED 77.26  
 LIQUID, GMS. 702.41  
 % ON FEED 17.62  
 GAS, GMS. 204.18  
 % ON FEED 5.12  
 MATERIAL BALANCE, % 96.29  
 WATER BALANCE, % 109.70

GAS ANALYSES, MOL %/WT %

H2 53.84/ 8.87  
 CO 2.54/ 5.86  
 CO2 7.71/27.95  
 H2S 0.23/ 0.65  
 NH3 1.62/ 2.27  
 CH4 27.44/36.18  
 C2H6 3.51/ 8.69  
 C2H4 1.14/ 2.63  
 C3H8 0.69/ 2.52  
 C3H6 1.27/ 4.40  
 MOLECULAR WEIGHT 12.14  
 HEATING VALUE, BTU/CU FT 627.87

DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F. 0.37  
 400-700 DEG. F. 1.66  
 700-1000 DEG. F. 8.72  
 1000 DEG F.+ 6.87  
 1000 DEG F.- 10.75  
 % 1000 F- IN FEED 11.40

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F. 95.35/13.75/ 0.01/ 0.11  
 C/H/S/N, WT % 1.94  
 ATOMIC H/C  
 400-700 DEG F. 39.56/ 7.60/ 0.04/ 0.57  
 C/H/S/N, WT % 1.02  
 ATOMIC H/C  
 700-1000 DEG F. 90.43/ 6.94/ 0.06/ 0.37  
 C/H/S/N, WT % 0.80  
 ATOMIC H/C  
 1000 DEG F.-  
 C/H/S/N, WT % 90.12/ 6.55/ 0.06/ 0.80  
 ATOMIC H/C 0.87  
 BTU/LB 16718.53  
 LB S/BTU 0.03  
 1000 DEG F.+  
 C/H/S/N, WT % 90.73/ 4.25/ 0.22/ 1.41  
 ATOMIC H/C 0.55  
 BTU/LB 15365.02  
 LB S/BTU 0.14

ELEMENTAL BALANCE

	C	H	S	N	ASH
<b>FEED</b>					
WT %	54.20	5.61	0.21	0.73	36.66
GM	2160.41	143.39	5.37	31.09	1461.27
% ON FEED	100.00	100.00	100.00	100.00	100.00
<b>LIQUID PRODUCT</b>					
WT %	90.36	5.65	0.12	1.04	0.0
GM	634.69	39.72	0.83	7.27	0.0
% ON FEED	29.33	27.60	9.87	23.39	0.0
<b>GASEOUS PRODUCT</b>					
WT %	52.29	21.55	0.61	1.87	0.0
GM	106.77	44.01	1.25	3.82	0.0
% ON FEED	4.94	30.55	14.95	12.29	0.0
<b>COKE</b>					
WT %	42.80	2.11	0.20	0.54	55.29
GM	1317.94	64.34	6.03	16.59	1702.65
% ON FEED	61.00	45.06	72.06	53.35	116.52
<b>ELEMENTAL BALANCE, %</b>					
	95.32	103.25	96.83	89.03	116.52

ATOMIC RATIO OF H/C: FEED=0.90 GAS=4.95 LIQUID=0.75 COKE=0.51

## CSCU RUN-174

TEMPERATURE, DEG. F	RV + 159.40
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU YP- 526( 840 F/ 40 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	6000.00
FEED RATE, GM/MIN.	19.05
STEAM FEED RATIO, % ON FEED	23.49
VAPOR RESIDENCE TIME, SEC.	RV X 2.62
PRODUCT YIELD	
COKE, GMS.	3866.25
% ON FEED	64.44
LIQUID, GMS.	1773.08
% ON FEED	29.55
GAS GMS.	360.68
% ON FEED	6.01
MATERIAL BALANCE, %	101.77
WATER BALANCE, %	95.63

## GAS ANALYSES, MOL %/WT %

H2	54.13/10.11
CO	3.72/ 9.73
CO2	2.61/10.73
H2S	0.29/ 0.92
NH3	1.58/ 2.51
CH4	31.64/47.28
C2H6	3.48/ 9.74
C2H4	0.88/ 2.31
C3H8	0.79/ 3.24
C3H6	0.87/ 3.43
MOLECULAR WEIGHT	10.71
HEATING VALUE, BTU/CU FT	631.29

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.86
400-700 DEG. F.	2.51
700-1000 DEG. F.	3.73
1000 DEG F.+	22.46
1000 DEG F.-	7.10
% 1000 F- IN FEED	5.10

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.08/13.80/ 0.10/ 0.16
ATOMIC H/C	1.95
400-700 DEG F.	
C/H/S/N, WT %	87.97/ 7.56/ 0.45/ 0.76
ATOMIC H/C	1.03
700-1000 DEG F.	
C/H/S/N, WT %	88.92/ 6.33/ 0.49/ 1.24
ATOMIC H/C	0.85
1000 DEG F.-	
C/H/S/N, WT %	88.12/ 7.67/ 0.43/ 0.94
ATOMIC H/C	1.04
BTU/LB	17102.28
LB S/MBTU	0.25
1000 DEG F.+	
C/H/S/N, WT %	88.54/ 5.35/ 1.00/ 1.74
ATOMIC H/C	0.72
BTU/LB	15744.90
LB S/MBTU	0.64

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.43	4.73	1.60	1.63	9.56
GM	4765.80	283.80	96.00	97.80	573.60
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	88.45	5.91	0.86	1.55	0.0
GM	1568.22	104.71	15.31	27.43	0.0
% ON FEED	32.91	36.90	15.94	28.04	0.0
GASEOUS PRODUCT					
WT %	57.92	25.78	0.86	2.07	0.0
GM	208.90	93.00	3.11	7.47	0.0
% ON FEED	4.38	32.77	3.24	7.63	0.0
COKE					
WT %	79.70	2.30	1.93	1.44	16.49
GM	3081.23	88.85	74.57	55.53	637.46
% ON FEED	64.65	31.31	77.68	56.78	111.13
ELEMENTAL BALANCE, %	101.94	100.97	96.86	92.46	111.13
ATOMIC RATIO OF H/C:	FEED-0.71	GAS-5.34	LIQUID-0.80	COKE-0.35	



## CSCU RUN-174

TEMPERATURE, DEG. F	RV + 159.40
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU YP- 526( 840 F/ 40 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	6000.00
FEED RATE, GM/MIN.	19.05
STEAM FEED RATIO, % ON FEED	23.49
VAPOR RESIDENCE TIME, SEC.	RV X 2.62
PRODUCT YIELD	
COKE, GMS.	3868.95
% ON FEED	64.48
LIQUID, GMS.	1770.38
% ON FEED	29.51
GAS, GMS.	360.68
% ON FEED	6.01
MATERIAL BALANCE, %	101.77
WATER BALANCE, %	95.63

## GAS ANALYSES, MOL %/WT %

H2	54.13/10.11
CO	3.72/ 9.73
CO2	2.61/10.73
H2S	0.29/ 0.92
NH3	1.58/ 2.51
CH4	31.64/47.28
C2H6	3.48/ 9.74
C2H4	0.88/ 2.31
C3H8	0.79/ 3.24
C3H6	0.87/ 3.43
MOLECULAR WEIGHT	10.71
HEATING VALUE, BTU/CU FT	631.29

## DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.86
400-700 DEG. F.	2.51
700-1000 DEG. F.	16.04
1000 DEG F.+	10.10
1000 DEG F.-	19.41
% 1000 F- IN FEED	11.90

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.				
C/H/S/N, WT %	85.08/13.80/	0.10/	0.16	
ATOMIC H/C			1.95	
400-700 DEG F.				
C/H/S/N, WT %	87.97/ 7.56/	0.45/	0.76	
ATOMIC H/C			1.03	
700-1000 DEG F.				
C/H/S/N, WT %	75.27/ 6.27/	0.60/	1.38	
ATOMIC H/C			1.00	
1000 DEG F.-				
C/H/S/N, WT %	77.35/ 6.77/	0.56/	1.25	
ATOMIC H/C			1.05	
BTU/LB			14343.17	
LB S/MBTU			0.39	
1000 DEG F.+				
C/H/S/N, WT %	89.06/ 4.79/	1.13/	1.88	
ATOMIC H/C			0.65	
BTU/LB			15504.23	
LB S/MBTU			0.73	

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.43	4.73	1.60	1.63	9.56
GM	4765.80	283.80	96.00	97.80	573.60
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	81.35	6.09	0.75	1.46	0.0
GM	1440.28	107.88	13.35	25.91	0.0
% ON FEED	30.22	38.01	13.91	26.49	0.0
GASEOUS PRODUCT					
WT %	57.92	25.78	0.86	2.07	0.0
GM	208.90	93.00	3.11	7.47	0.0
% ON FEED	4.38	32.77	3.24	7.63	0.0
COKE					
WT %	79.65	2.30	1.93	1.44	16.53
GM	3081.75	88.87	74.58	55.54	639.61
% ON FEED	64.66	31.31	77.69	56.79	111.51
ELEMENTAL BALANCE, %	99.27	102.10	94.84	90.92	111.51

ATOMIC RATIO OF H/C: FEED-0.71 GAS-5.34 LIQUID-0.90 COKE-0.32

## CSCU RUN-175

TEMPERATURE, DEG. F	RV + 166.80
PRESSURE, PSIG	9.00
FEED SOURCE RCLU YP- 529( 840 F/100 MIN.)	
PITTSBURGH #8 ARKWRIGHT BOTTOMS	
FEED, GM.	5996.00
FEED RATE, GM/MIN.	20.70
STEAM FEED RATIO, % ON FEED	18.32
VAPOR RESIDENCE TIME, SEC.	RV X 2.72
PRODUCT YIELD	
COKE, GMS.	4070.43
% ON FEED	67.89
LIQUID, GMS.	1644.00
% ON FEED	27.42
GAS, GMS.	281.57
% ON FEED	4.70
MATERIAL BALANCE, %	99.32
WATER BALANCE, %	96.18

## GAS ANALYSES, MOL %/WT %

H2	62.73/13.49
CO	5.37/16.18
CO2	2.01/ 9.53
H2S	0.39/ 1.43
NH3	1.52/ 2.77
CH4	23.40/40.25
C2H6	2.74/ 8.84
C2H4	0.60/ 1.81
C3H8	0.62/ 2.91
C3H6	0.62/ 2.80
MOLECULAR WEIGHT	9.30
HEATING VALUE, BTU/CU FT	549.50

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.20
400-700 DEG. F.	1.64
700-1000 DEG. F.	3.69
1000 DEG F.+	21.89
1000 DEG F.-	5.53
% 1000 F- IN FEED	6.20

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.70/13.06/ 0.07/ 0.27
ATOMIC H/C	1.83
400-700 DEG F.	
C/H/S/N, WT %	89.51/ 7.13/ 0.22/ 0.88
ATOMIC H/C	0.96
700-1000 DEG F.	
C/H/S/N, WT %	90.32/ 6.00/ 0.40/ 1.29
ATOMIC H/C	0.80
1000 DEG F.-	
C/H/S/N, WT %	89.91/ 6.60/ 0.33/ 1.13
ATOMIC H/C	0.88
BTU/LB	16755.91
LB S/MBTU	0.20
1000 DEG F.+	
C/H/S/N, WT %	90.89/ 5.16/ 0.49/ 1.81
ATOMIC H/C	0.68
BTU/LB	16059.07
LB S/MBTU	0.31

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.47	4.41	1.26	1.62	11.24
GM	4765.02	264.42	75.55	97.14	673.95
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	90.69	5.45	0.46	1.67	0.0
GM	1490.86	89.58	7.60	27.47	0.0
% ON FEED	31.29	33.88	10.06	28.28	0.0
GASEOUS PRODUCT					
WT %	53.12	27.08	1.35	2.28	0.0
GM	149.57	76.24	3.79	6.42	0.0
% ON FEED	3.14	28.83	5.02	6.61	0.0
COKE					
WT %	79.32	2.17	1.34	1.14	18.71
GM	3228.55	88.39	54.64	46.60	761.53
% ON FEED	67.76	33.43	72.32	47.98	112.99
ELEMENTAL BALANCE, %					
	102.18	96.14	87.40	82.87	112.99

ATOMIC RATIO OF H/C: FEED-0.67 GAS-6.12 LIQUID-0.72 COKE-0.38

## CSCU RUN-175

TEMPERATURE, DEG. F	RV + 166.80
PRESSURE, PSIG	9.00
FEEED SOURCE	RCLU YP- 529( 840 F/100 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEEED, GM.	5996.00
FEEED RATE, GM/MIN.	20.70
STEAM FEEED RATIO, % ON FEEED	18.32
VAPOR RESIDENCE TIME, SEC.	RV X 2.72
PRODUCT YIELD	
COKE, GMS.	4070.99
% ON FEEED	67.90
LIQUID, GMS.	1643.45
% ON FEEED	27.41
GAS, GMS.	281.57
% ON FEEED	4.70
MATERIAL BALANCE, %	99.32
WATER BALANCE, %	96.18

## GAS ANALYSES, MOL %/WT %

H2	62.73/13.49
CO	5.37/16.18
CO2	2.01/ 9.53
H2S	0.39/ 1.43
NH3	1.52/ 2.77
CH4	23.40/40.25
C2H6	2.74/ 8.84
C2H4	0.60/ 1.81
C3H8	0.62/ 2.91
C3H6	0.62/ 2.80
MOLECULAR WEIGHT	9.30
HEATING VALUE, BTU/CU FT	549.50

DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEEED	
C4-400 DEG. F.	0.20
400-700 DEG. F.	1.64
700-1000 DEG. F.	18.23
1000 DEG F.+	7.34
1000 DEG F.-	20.07
% 1000 F- IN FEEED	14.60

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.70/13.06/ 0.07/ 0.27
ATOMIC H/C	1.83
400-700 DEG F.	
C/H/S/N, WT %	89.51/ 7.13/ 0.22/ 0.88
ATOMIC H/C	0.96
700-1000 DEG F.	
C/H/S/N, WT %	89.92/ 5.80/ 0.43/ 1.53
ATOMIC H/C	0.77
1000 DEG F.-	
C/H/S/N, WT %	89.84/ 5.98/ 0.41/ 1.46
ATOMIC H/C	0.80
BTU/LB	16361.16
LB S/MBTU	0.25
1000 DEG F.+	
C/H/S/N, WT %	90.81/ 4.62/ 0.71/ 1.86
ATOMIC H/C	0.61
BTU/LB	15704.09
LB S/MBTU	0.45

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEEED					
WT %	79.47	4.41	1.26	1.62	11.24
GM	4765.02	264.42	75.55	97.14	673.95
% ON FEEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	90.10	5.62	0.49	1.57	0.0
GM	1480.78	92.30	8.05	25.82	0.0
% ON FEEED	31.08	34.91	10.66	26.59	0.0
GASEOUS PRODUCT					
WT %	53.12	27.08	1.35	2.28	0.0
GM	149.57	76.24	3.79	6.42	0.0
% ON FEEED	3.14	28.83	5.02	6.61	0.0
COKE					
WT %	79.31	2.17	1.34	1.14	18.72
GM	3228.66	88.39	54.64	46.61	761.96
% ON FEEED	67.76	33.43	72.33	47.98	113.06
ELEMENTAL BALANCE, %	101.97	97.17	88.00	81.18	113.06

ATOMIC RATIO OF H/C: FEEED-0.67 GAS-6.12 LIQUID-0.75 COKE-0.37

## CSCU RUN-178

TEMPERATURE, DEG. F	RV + 100.50
PRESSURE, PSIG	9.00
FEED SOURCE	RCLU YP- 529( 840 F/100 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	4195.00
FEED RATE, GM/MIN.	23.31
STEAM FEED RATIO, % ON FEED	16.22
VAPOR RESIDENCE TIME, SEC.	RV X 2.63
PRODUCT YIELD	
COKE, GMS.	2935.78
% ON FEED	69.98
LIQUID, GMS.	1120.87
% ON FEED	26.72
GAS, GMS.	138.36
% ON FEED	3.30
MATERIAL BALANCE, %	99.92
WATER BALANCE, %	91.12

## GAS ANALYSES, MOL %/WT %

H2	67.66/17.19
CO	1.43/ 5.08
CO2	1.71/ 9.56
H2S	0.23/ 1.01
NH3	1.38/ 2.98
CH4	23.78/48.33
C2H6	2.58/ 9.82
C2H4	0.37/ 1.32
C3H8	0.48/ 2.68
C3H6	0.38/ 2.03
MOLECULAR WEIGHT	7.87
HEATING VALUE, BTU/CU FT	538.52

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED	
C4-400 DEG. F.	0.21
400-700 DEG. F.	2.24
700-1000 DEG. F.	3.82
1000 DEG F.+	20.45
1000 DEG F.-	6.27
% 1000 F- IN FEED	6.20

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.69/12.79/ 0.08/ 0.36
ATOMIC H/C	1.79
400-700 DEG F.	
C/H/S/N, WT %	89.16/ 7.19/ 0.20/ 0.94
ATOMIC H/C	0.97
700-1000 DEG F.	
C/H/S/N, WT %	90.36/ 6.06/ 0.42/ 1.33
ATOMIC H/C	0.80
1000 DEG F.-	
C/H/S/N, WT %	89.77/ 6.69/ 0.33/ 1.16
ATOMIC H/C	0.89
BTU/LB	16792.01
LB S/MBTU	0.20
1000 DEG F.+	
C/H/S/N, WT %	89.46/ 5.19/ 0.71/ 1.74
ATOMIC H/C	0.70
BTU/LB	15802.51
LB S/MBTU	0.45

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.52	4.45	1.13	1.51	11.27
GM	3335.86	186.68	47.40	63.34	472.78
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	89.52	5.54	0.62	1.60	0.0
GM	1003.45	62.13	6.99	17.95	0.0
% ON FEED	30.08	33.28	14.75	28.34	0.0
GASEOUS PRODUCT					
WT %	53.95	32.79	0.95	2.46	0.0
GM	74.65	45.36	1.32	3.40	0.0
% ON FEED	2.24	24.30	2.78	5.37	0.0
COKE					
WT %	73.57	2.09	1.42	1.02	19.16
GM	2159.71	61.24	41.82	29.87	562.48
% ON FEED	64.74	32.80	88.22	47.16	118.97
ELEMENTAL BALANCE, %	97.06	90.38	105.74	80.86	118.97

ATOMIC RATIO OF H/C: FEED-0.67 GAS-7.29 LIQUID-0.74 COKE-0.42

## CSCU RUN-178

TEMPERATURE, DEG. F	RV + 100.50
PRESSURE, PSIG	9.00
FEED SOURCE	RCLU YP- 529( 840 F/100 MIN.)
	PITTSBURGH #8 ARKWRIGHT BOTTOMS
FEED, GM.	4195.00
FEED RATE, GM/MIN.	23.31
STEAM FEED RATIO, % ON FEED	16.22
VAPOR RESIDENCE TIME, SEC.	RV X 2.63
PRODUCT YIELD	
COKE, GMS.	2937.79
% ON FEED	70.03
LIQUID, GMS.	1118.85
% ON FEED	26.67
GAS, GMS.	138.36
% ON FEED	3.30
MATERIAL BALANCE, %	99.92
WATER BALANCE, %	91.12

## GAS ANALYSES, MOL %/WT %

H2	67.66/17.19
CO	1.43/ 5.08
CO2	1.71/ 9.56
H2S	0.23/ 1.01
NH3	1.38/ 2.98
CH4	23.78/48.33
C2H6	2.58/ 9.82
C2H4	0.37/ 1.32
C3H8	0.48/ 2.68
C3H6	0.38/ 2.03
MOLECULAR WEIGHT	7.87
HEATING VALUE, BTU/CU FT	538.52

## DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.21
400-700 DEG. F.	2.24
700-1000 DEG. F.	16.91
1000 DEG F.+	7.32
1000 DEG F.-	19.35
% 1000 F- IN FEED	14.60

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.69/12.79/ 0.08/ 0.36
ATOMIC H/C	1.79
400-700 DEG F.	
C/H/S/N, WT %	89.16/ 7.19/ 0.20/ 0.94
ATOMIC H/C	0.97
700-1000 DEG F.	
C/H/S/N, WT %	90.30/ 5.95/ 0.40/ 1.39
ATOMIC H/C	0.79
1000 DEG F.-	
C/H/S/N, WT %	90.12/ 6.17/ 0.37/ 1.33
ATOMIC H/C	0.82
BTU/LB	16529.48
LB S/MBTU	0.23
1000 DEG F.+	
C/H/S/N, WT %	90.98/ 4.22/ 0.90/ 1.74
ATOMIC H/C	0.56
BTU/LB	15489.13
LB S/MBTU	0.58

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	79.47	4.41	1.26	1.62	11.24
GM	3333.77	185.00	52.86	67.96	471.52
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	90.35	5.63	0.52	1.44	0.0
GM	1010.94	63.04	5.81	16.12	0.0
% ON FEED	30.32	34.07	10.99	23.72	0.0
GASEOUS PRODUCT					
WT %	53.95	32.79	0.95	2.46	0.0
GM	74.65	45.36	1.32	3.40	0.0
% ON FEED	2.24	24.52	2.49	5.00	0.0
COKE					
WT %	73.52	2.08	1.42	1.02	19.20
GM	2160.00	61.24	41.83	29.88	564.18
% ON FEED	64.79	33.11	79.13	43.96	119.65
ELEMENTAL BALANCE, %	97.35	91.70	92.61	72.68	119.65

ATOMIC RATIO OF H/C: FEED-0.67 GAS-7.29 LIQUID-0.75 COKE-0.41

## CSCU RUN-189

TEMPERATURE, DEG. F                   RV + 175.00  
 PRESSURE, PSIG                               9.70  
 FEED SOURCE       RCLU YP- 626( 840 F/ 40 MIN.)  
                   WANDOAN BOTTOMS  
 FEED, GM.                                       5625.00  
 FEED RATE, GM/MIN.                       15.60  
 STEAM FEED RATIO, % ON FEED           23.25  
 VAPOR RESIDENCE TIME, SEC.           RV X   3.32  
 PRODUCT YIELD  
   COKE,   GMS.                               3945.58  
           % ON FEED                       70.14  
   LIQUID, GMS.                               1522.44  
           % ON FEED                       27.07  
   GAS,   GMS.                               156.98  
           % ON FEED                       2.79  
 MATERIAL BALANCE, %                   95.26  
 WATER BALANCE, %                       97.52

## GAS ANALYSES, MOL %/WT %

H2                   56.03/ 9.46  
 CO                   2.01/ 4.76  
 CO2                  8.59/31.90  
 H2S                  0.25/ 0.73  
 NH3                  3.34/ 4.80  
 CH4                  24.18/32.66  
 C2H6                2.97/ 7.52  
 C2H4                1.04/ 2.45  
 C3H8                0.54/ 1.99  
 C3H6                1.05/ 3.73  
 MOLECULAR WEIGHT       11.84  
 HEATING VALUE, BTU/CU FT   582.39

## DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.                   0.26  
 400-700 DEG. F.                3.32  
 700-1000 DEG. F.               6.64  
 1000 DEG F.+                   16.84  
 1000 DEG F.-                   10.22  
 % 1000 F- IN FEED           4.20

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.  
 C/H/S/N, WT %                   86.30/11.68/ 0.03/ 0.25  
 ATOMIC H/C                       1.62  
 400-700 DEG F.  
 C/H/S/N, WT %                   88.54/ 7.73/ 0.07/ 0.50  
 ATOMIC H/C                       1.05  
 700-1000 DEG F.  
 C/H/S/N, WT %                   89.70/ 6.34/ 0.10/ 0.63  
 ATOMIC H/C                       0.85  
 1000 DEG F.-  
 C/H/S/N, WT %                   89.24/ 6.93/ 0.09/ 0.58  
 ATOMIC H/C                       0.93  
 BTU/LB                           16778.48  
 LB S/MBTU                       0.05  
 1000 DEG F.+  
 C/H/S/N, WT %                   67.58/ 5.26/ 0.22/ 0.75  
 ATOMIC H/C                       0.93  
 BTU/LB                           11375.53  
 LB S/MBTU                       0.19

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	53.62	3.63	0.25	0.79	36.59
GM	3016.12	204.19	14.06	44.44	2058.19
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	75.76	5.89	0.17	0.69	0.0
GM	1153.43	89.69	2.55	10.46	0.0
% ON FEED	38.24	43.93	18.12	23.54	0.0
GASEOUS PRODUCT					
WT %	48.18	21.27	0.68	3.95	0.0
GM	75.64	33.38	1.07	6.20	0.0
% ON FEED	2.51	16.35	7.62	13.96	0.0
COKE					
WT %	30.71	0.81	0.24	0.81	63.10
GM	1211.86	31.89	9.57	31.89	2489.74
% ON FEED	40.18	15.62	68.03	71.77	120.97
ELEMENTAL BALANCE, %					
	80.93	75.89	93.78	109.26	120.97

ATOMIC RATIO OF H/C: FEED-0.81 GAS-5.30 LIQUID-0.93 COKE-0.54

## CSCU RUN-189

TEMPERATURE, DEG. F	RV + 175.00
PRESSURE, PSIG	9.70
FEED SOURCE	RCLU YP- 626( 840 F/ 40 MIN.)
	WANDOAN BOTTOMS
FEED, GM.	5625.00
FEED RATE, GM/MIN.	15.60
STEAM FEED RATIO, % ON FEED	23.25
VAPOR RESIDENCE TIME, SEC.	RV X 3.32
PRODUCT YIELD	
COKE, GMS.	3964.72
% ON FEED	70.48
LIQUID, GMS.	1503.30
% ON FEED	26.73
GAS, GMS.	156.98
% ON FEED	2.79
MATERIAL BALANCE, %	95.26
WATER BALANCE, %	97.52

## GAS ANALYSES, MOL %/WT %

H2	56.03/ 9.46
CO	2.01/ 4.76
CO2	8.59/31.90
H2S	0.25/ 0.73
NH3	3.34/ 4.80
CH4	24.18/32.66
C2H6	2.97/ 7.52
C2H4	1.04/ 2.45
C3H8	0.54/ 1.99
C3H6	1.05/ 3.73
MOLECULAR WEIGHT	11.84
HEATING VALUE, BTU/CU FT	582.39

## DISTILLATION CUT(HIVAC-C) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.26
400-700 DEG. F.	3.32
700-1000 DEG. F.	15.05
1000 DEG F.+	8.09
1000 DEG F.-	18.63
% 1000 F- IN FEED	11.40

## ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	86.30/11.68/ 0.03/ 0.25
ATOMIC H/C	1.62
400-700 DEG F.	
C/H/S/N, WT %	88.54/ 7.73/ 0.07/ 0.50
ATOMIC H/C	1.05
700-1000 DEG F.	
C/H/S/N, WT %	89.04/ 6.28/ 0.11/ 0.90
ATOMIC H/C	0.85
1000 DEG F.-	
C/H/S/N, WT %	88.91/ 6.61/ 0.10/ 0.82
ATOMIC H/C	0.89
BTU/LB	16518.33
LB S/MBTU	0.06
1000 DEG F.+	
C/H/S/N, WT %	90.47/ 4.90/ 0.30/ 1.15
ATOMIC H/C	0.65
BTU/LB	15733.81
LB S/MBTU	0.19

## ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	53.62	3.63	0.25	0.79	36.59
GM	3016.12	204.19	14.06	44.44	2058.19
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	89.38	6.09	0.16	0.92	0.0
GM	1343.71	91.62	2.43	13.83	0.0
% ON FEED	44.55	44.87	17.30	31.13	0.0
GASEOUS PRODUCT					
WT %	48.18	21.27	0.68	3.95	0.0
GM	75.64	33.38	1.07	6.20	0.0
% ON FEED	2.51	16.35	7.62	13.96	0.0
COKE					
WT %	30.60	0.81	0.24	0.81	63.24
GM	1213.32	31.93	9.58	31.93	2507.14
% ON FEED	40.23	15.64	68.12	71.85	121.81
ELEMENTAL BALANCE, %	87.29	76.86	93.04	116.94	121.81

ATOMIC RATIO OF H/C: FEED-0.81 GAS-5.30 LIQUID-0.82 COKE-0.60

## Appendix D

### MINIGASIFIER RATE DATA

#### Reduction/Adjustment Procedures

Raw data from the minigasifier consists of coke description, time, temperature, pressure, gas volumes, and gas analyses. These raw data are illustrated by empty reactor runs 646, 647, 657 and 661. Two adjustments are applied to the coke gasification raw data before it is studied and reported as in Tables D-2 to D-32. The first adjustment is the background or empty reactor adjustment to the CO<sub>2</sub>-coke runs. The second adjustment is for carbon balance on all runs.

The background gasification adjustment is derived from Runs 617, 644 and 645. In Tables D-2, D-18, and D-19 these runs are reported as if they contained a 1-gm. charge of pure carbon. From these data are derived the first-order correction:

$$C_{uE} = C_{uE_0} e^{-k P_{CO_2} t}$$

where,

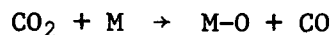
$C_{uE}$  = unevolved background carbon,  $C_{uE_0} = 0.049$  gm.

$k$  = first-order rate constant,  $4.7 \times 10^{-3} \text{ min}^{-1} \text{ atm}^{-1}$

$P_{CO_2}$  = carbon dioxide partial pressure, atm

$t$  = run time, min

The evolved carbon appears as CO. It is believed that:



where M is a component of the metal reactor. At first it was thought that M was carbon. However, empty reactor runs with steam did not yield CO or CO<sub>2</sub>, suggesting M may not be carbon. Examples of steam-empty reactor runs are given in Tables D-20, D-21, D-28, and D-31 for runs 646, 647, 657 and 661, respectively. H<sub>2</sub> is evolved for the steam-empty reactor runs, but steam run background corrections are not needed since gasification is calculated by carbon contained in the gaseous carbon oxides.

The runs reported, except the empty reactor runs, are carbon balanced. The carbon balances are based on weight charged and solid analyses because they are more accurate than gas measurements. The coke charge of



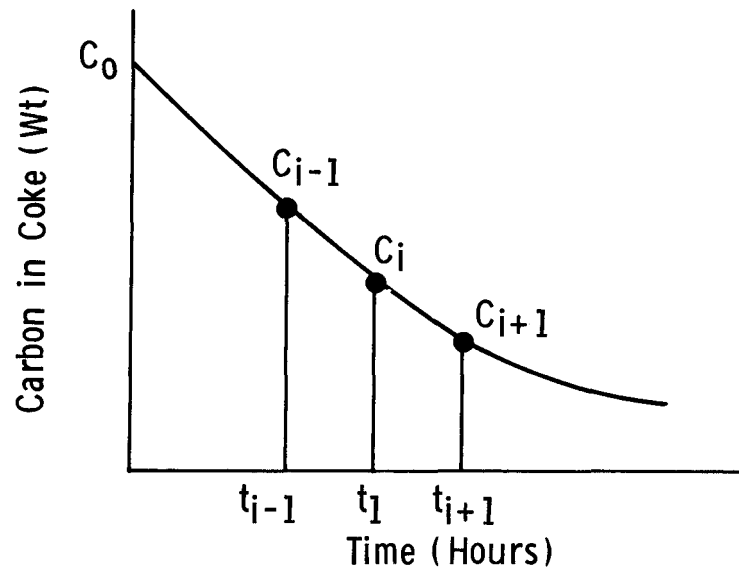
about 1 gm. is accurately measured and analyzed for carbon and ash. The gasified residue is also analyzed for carbon and ash. As some of the residue carbon is trapped in the glass wool packing, the residue carbon is calculated by ash and carbon balances. Then the gas CO content for CO<sub>2</sub> runs or CO and CO<sub>2</sub> contents for H<sub>2</sub>O runs are linearly adjusted to obtain 100% carbon balance. Usually this adjustment is small and a run is discarded if it is large.

The rate data in Tables D-2 to D-32 are next reduced to initial rate data. This is done by examining the data in several forms. For an ideal system, the instantaneous and average rates (see the following "Rate Data Representation" for definitions) reduce to the same value at zero carbon conversion. In the case of the CO<sub>2</sub> runs this is very helpful in that there is little backmixing. There is some random fluctuation in the data up to 15 or 20% carbon conversion so typically rates at 20 to 60% carbon conversion are extrapolated back to zero conversion for the initial rate.

The H<sub>2</sub>O runs are more troublesome in that the condenser, condensate collector, and drying tube (items 6, 7, and 8 in Figure D-1) are larger and induce backmixing which results in an initial lag in measured gas composition. This lag effect is eliminated in rate extrapolations by using carbon gasified versus time plots. The average steam gasification rates as reported in Tables D-22 through D-32 erroneously extrapolate 20 to 40% low at zero gasification if the lag effect is ignored.

FIGURE D-1  
MINIGASIFIER  
RATE DATA REPRESENTATION

Two Different Methods are Used to Represent the Coke Gasification Rates, as are Described Below Graphically.



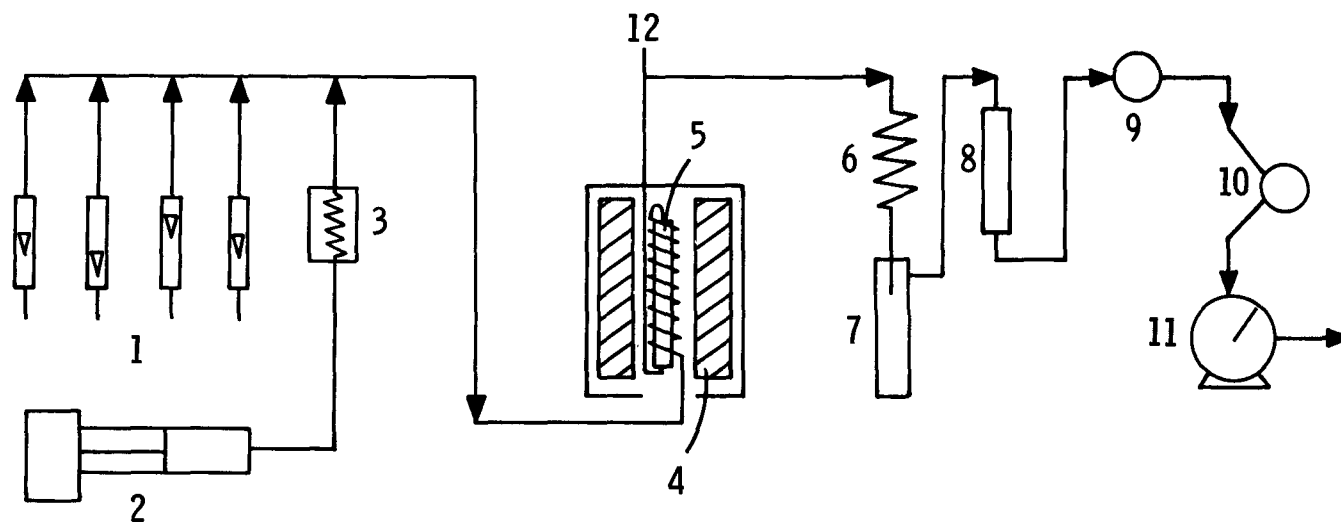
Instantaneous Rate on Remaining Carbon:

$$\left( \frac{C_i - C_{i-1}}{t_i - t_{i-1}} + \frac{C_{i+1} - C_i}{t_{i+1} - t_i} \right) \frac{1}{2 C_i}$$

Average Rate on Initial Carbon:

$$\left( \frac{C_0 - C_i}{t_i} \right) \frac{1}{C_0}$$

FIGURE D-2  
MINIGASIFIER FLOWPLAN



- |                                 |                                  |
|---------------------------------|----------------------------------|
| 1 Inlet Gas Metering            | 7 Condensate Collector           |
| 2 Water Pump (H <sub>2</sub> O) | 8 Drying Tube (H <sub>2</sub> O) |
| 3 Vaporizer (H <sub>2</sub> O)  | 9 Back Pressure Regulator        |
| 4 Reactor Heater                | 10 Gas Sampling Valve (GC)       |
| 5 Fixed-Bed, Down-Flow Reactor  | 11 Gas Meter                     |
| 6 Condenser                     | 12 Reactor Thermocouple          |

TABLE D-1

MINIGASIFIER

STUDY COKE

Source: IKG Illinois Gasifier, Run 21, Balance 9  
(See July-September, 1978, Quarterly  
Technical Progress Report [FE-2893-2],  
pp. 81-98 for discussion of operation)

Mesh Size, U.S. Sieve	<u>Analyses, Wt %</u>				IKG Unit Sample
	<u>-40+50</u>	<u>-50+70</u>	<u>-70+100</u>	<u>-100+150</u>	
Carbon	50.6	48.7	46.0	44.3	51.6
Hydrogen	0.56	0.33	0.40		0.23
Nitrogen					0.34
Sulfur		1.73			2.31
Moisture		3.12	3.50		
Carbon-Hydrogen Residue	45.1	47.3	50.2	51.1	
Ash		46.2	49.2		48.1
BET Surface Area, m <sup>2</sup> /g					74

NOTES:

1. IKG unit sample analyses were conducted at ERDL; whereas, sieve fraction sample analyses were conducted at BARD.
2. Carbon-hydrogen residue provides a check on ASTM ash determinations and is generally in close agreement for these cokes as can be seen in the table.

TABLE D-2

RUN 617

## GASIFICATION RATES FROM M11GASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.47  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 100.00  
 % ASH IN COKE 0.0  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... EMPTY REACTOR

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HK
1.3	0.3	0.67	0.04	2.86	1.4	98.3	10.6
10.5	1.7	0.41	0.03	2.88	0.9	99.0	7.7
19.8	2.6	0.26	0.02	2.89	0.6	99.4	5.0
29.2	3.2	0.20	0.01	2.89	0.4	99.5	3.9
38.5	3.8	0.21	0.01	2.89	0.3	99.5	3.5
47.8	4.3	0.16	0.01	2.89	0.2	99.6	2.9
57.2	4.6	0.11	0.01	2.90	0.2	99.7	2.2
66.5	4.9	0.10	0.01	2.90	0.2	99.8	1.8
75.9	5.2	0.09	0.01	2.90	0.2	99.8	1.6
85.2	5.4	0.08	0.00	2.90	0.2	99.6	1.3
94.5	5.6	0.04	0.00	2.90	0.1	99.9	1.1

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HK
1.3	1.4	98.3	11.8
10.5	1.2	98.6	9.7
19.8	1.0	98.9	7.8
29.2	0.8	99.1	6.6
38.5	0.7	99.2	5.9
47.8	0.7	99.3	5.3
57.2	0.6	99.3	4.9
66.5	0.5	99.4	4.4
75.9	0.5	99.4	4.1
85.2	0.5	99.5	3.8
94.5	0.4	99.5	3.5

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-3

KUM 616

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 57.00  
 SUPERFICIAL VELOCITY (CM/SEC) 18.69  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.4	1.8	1.69	0.18	4.66	3.7	96.0	82.9
10.8	15.0	1.57	0.16	4.72	3.2	96.7	96.0
20.1	27.3	1.52	0.15	4.72	3.1	96.9	106.6
29.5	39.1	1.38	0.14	4.74	2.8	97.2	118.1
38.8	49.8	1.24	0.12	4.75	2.5	97.5	129.3
48.1	59.3	1.08	0.11	4.77	2.2	97.8	141.7
57.5	67.7	0.94	0.09	4.78	1.9	98.1	154.6
66.8	74.8	0.78	0.08	4.80	1.6	98.4	163.9
76.2	80.5	0.60	0.06	4.82	1.2	98.8	165.1
85.5	84.8	0.44	0.04	4.83	0.9	99.1	161.5
94.8	88.2	0.38	0.04	4.84	0.7	99.2	169.9
103.2	90.8	0.29	0.03	4.85	0.6	99.4	168.6
112.5	92.7	0.20	0.02	4.86	0.4	99.6	149.4
121.9	94.2	0.16	0.02	4.86	0.3	99.7	142.7
131.2	95.3	0.12	0.01	4.87	0.2	99.7	128.1
140.5	96.0	0.07	0.01	4.87	0.1	99.8	121.2

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.4	3.7	96.0	79.0
10.8	3.5	96.3	83.2
20.1	3.3	96.5	81.5
29.5	3.2	96.7	79.5
38.8	3.1	96.8	76.9
48.1	2.9	97.0	73.9
57.5	2.8	97.1	70.1
66.8	2.6	97.3	67.2
76.2	2.5	97.5	63.4
85.5	2.3	97.6	59.5
94.8	2.2	97.8	55.8
103.2	2.0	97.9	52.8
112.5	1.9	98.0	49.4
121.9	1.8	98.2	46.4
131.2	1.7	98.3	43.6
140.5	1.6	98.4	41.0

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-4

RUN 620

## GASIFICATION RATES FROM MINIGASIFILM

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 27.00  
 SUPERFICIAL VELOCITY (CM/SEC) 18.67  
 COKE CHANGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE-NITROGEN  
 COKE SAMPLE..... IKG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.1	1.2	2.81	0.12	2.02	4.2	71.1	63.4
10.6	10.3	2.29	0.10	2.01	3.5	71.0	60.1
19.9	18.1	2.07	0.09	2.05	3.1	72.4	61.2
29.3	25.9	2.28	0.10	2.04	3.4	71.9	69.1
38.6	34.1	2.12	0.09	2.04	3.2	72.1	76.2
47.9	41.6	2.07	0.09	2.04	3.2	72.1	80.9
57.3	48.8	1.86	0.08	2.06	2.8	72.5	86.6
66.6	55.4	1.76	0.08	2.06	2.7	72.8	92.1
75.9	61.6	1.63	0.07	2.06	2.4	72.7	100.2
85.3	67.4	1.52	0.06	2.07	2.3	72.9	106.2
94.6	72.4	1.24	0.05	2.09	1.9	73.7	108.9
104.0	76.7	1.15	0.05	2.08	1.7	73.5	114.4
113.3	80.7	1.02	0.04	2.09	1.5	73.7	119.2
122.6	83.9	0.80	0.03	2.11	1.2	74.3	120.0
132.0	86.7	0.74	0.03	2.11	1.1	74.2	122.7
141.3	89.0	0.56	0.02	2.12	0.8	74.7	121.7
150.7	90.8	0.49	0.02	2.12	0.7	74.6	116.5
160.0	92.3	0.37	0.02	2.12	0.6	74.7	106.5
169.3	93.4	0.27	0.01	2.13	0.4	75.0	92.7
178.7	94.2	0.25	0.01	2.13	0.4	75.0	84.5
188.0	94.9	0.17	0.01	2.13	0.3	75.1	68.5
197.4	95.3	0.12	0.01	2.14	0.2	75.4	57.4

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.1	4.2	71.1	67.5
10.6	3.9	71.0	58.7
19.9	3.6	71.4	54.7
29.3	3.5	71.6	53.2
38.6	3.4	71.7	53.0
47.9	3.4	71.8	52.0
57.3	3.3	71.9	51.1
66.6	3.2	72.0	49.9
75.9	3.2	72.1	48.7
85.3	3.1	72.1	47.4
94.6	3.0	72.3	45.9
104.0	2.9	72.4	44.3
113.3	2.8	72.5	42.7
122.6	2.7	72.6	41.0
132.0	2.6	72.7	39.4
141.3	2.4	72.8	37.8
150.7	2.3	72.9	36.2
160.0	2.2	73.0	34.6
169.3	2.2	73.1	33.1
178.7	2.1	73.2	31.6
188.0	2.0	73.3	30.3
197.4	1.9	73.4	29.0

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-5

RUN 620

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (VEL. F - RV) -55.00  
 PRESSURE (PSIG) 48.00  
 SUPERFICIAL VELOCITY (CM/SEC) 16.85  
 COKE CHARGE (G) 1.00  
 X CARBON IN COKE 46.70  
 X ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 CORE SAMPLE..... ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM		GAS ANALYSIS (WY), VOL %		WAT (1)
	CARBON	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO	CO <sub>2</sub>	Σ(CARBON/HR)
1.2	1.3	2.05	0.12	2.76	4.1	95.6	65.6
8.1	8.5	2.04	0.12	2.76	4.1	95.7	66.6
15.0	15.6	2.06	0.12	2.76	4.1	95.8	72.0
21.8	22.5	1.88	0.11	2.79	3.8	96.1	77.1
28.6	29.3	1.90	0.11	2.79	3.8	96.1	84.0
35.5	36.0	1.85	0.11	2.80	3.7	96.3	90.2
42.3	42.4	1.70	0.10	2.80	3.4	96.5	93.7
49.2	46.3	1.56	0.09	2.81	3.1	96.6	95.2
56.0	53.7	1.41	0.08	2.82	2.8	97.1	98.6
62.8	58.7	1.39	0.08	2.82	2.8	97.2	106.2
69.7	63.6	1.30	0.08	2.83	2.6	97.3	112.6
76.5	68.1	1.16	0.07	2.83	2.3	97.6	115.3
83.4	72.0	1.05	0.06	2.84	2.1	97.9	118.1
90.2	75.6	0.95	0.06	2.85	1.9	98.0	124.0
97.0	78.9	0.88	0.05	2.85	1.8	98.2	133.4
103.9	82.0	0.82	0.05	2.86	1.6	98.3	143.1
110.7	84.8	0.71	0.04	2.86	1.4	98.5	149.0
117.6	87.2	0.61	0.04	2.87	1.2	98.7	152.8
124.4	89.3	0.53	0.03	2.87	1.1	98.9	158.7
131.2	91.1	0.48	0.03	2.88	1.0	99.0	169.3
138.1	92.7	0.43	0.02	2.88	0.9	99.1	183.0
144.9	94.1	0.36	0.02	2.88	0.7	99.2	192.7
151.8	95.3	0.30	0.02	2.89	0.6	99.4	197.3
158.6	96.2	0.24	0.01	2.89	0.5	99.5	202.1
165.4	97.0	0.21	0.01	2.89	0.4	99.6	208.4
172.3	97.7	0.16	0.01	2.89	0.3	99.6	205.7
179.1	98.1	0.12	0.01	2.90	0.2	99.7	188.7
186.0	98.5	0.10	0.01	2.90	0.2	99.8	175.4
192.8	98.7	0.07	0.01	2.90	0.1	99.8	164.6
199.6	98.9	0.07	0.01	2.90	0.1	99.8	165.7
206.5	99.1	0.06	0.01	2.90	0.1	99.8	169.0
213.3	99.3	0.04	0.01	2.90	0.1	99.9	171.5

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (WY), VOL %		WAT (2)
	CO	CO <sub>2</sub>	Σ(CARBON/HR)
1.2	4.1	95.6	62.7
8.1	4.1	95.7	62.9
15.0	4.1	95.7	62.7
21.8	4.1	95.8	61.8
28.6	4.0	95.9	61.3
35.5	3.9	95.9	60.9
42.3	3.9	96.0	60.2
49.2	3.8	96.1	59.0
56.0	3.7	96.2	57.5
62.8	3.6	96.3	56.1
69.7	3.5	96.4	54.8
76.5	3.4	96.5	53.4
83.4	3.3	96.6	51.8
90.2	3.2	96.7	50.3
97.0	3.1	96.8	48.8
103.9	3.0	96.9	47.4
110.7	2.9	97.0	45.9
117.6	2.8	97.1	44.5
124.4	2.7	97.2	43.0
131.2	2.7	97.3	41.6
138.1	2.6	97.4	40.3
144.9	2.5	97.4	39.0
151.8	2.4	97.5	37.7
158.6	2.3	97.6	36.4
165.4	2.2	97.7	35.2
172.3	2.2	97.8	34.0
179.1	2.1	97.8	32.9
186.0	2.0	97.9	31.8
192.8	2.0	98.0	30.7
199.6	1.9	98.0	29.7
206.5	1.8	98.1	28.6
213.3	1.8	98.2	27.9

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON



TABLE D-6

MUN 627

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - HW) -55.00  
 PRESSURE (PSIG) 26.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.23  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE-NITROGEN  
 COKE SAMPLE..... IKG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.4	1.3	3.29	0.10	1.26	3.3	43.3	52.4
8.3	7.0	2.93	0.09	1.26	3.0	43.5	52.0
15.2	12.4	2.93	0.09	1.28	3.6	44.0	52.8
22.0	17.6	2.82	0.08	1.31	2.8	43.0	55.3
28.9	22.8	2.89	0.08	1.38	2.8	47.7	59.5
35.7	28.1	2.88	0.08	1.40	2.8	48.2	63.7
42.5	33.2	2.67	0.07	1.37	2.6	47.3	64.6
49.4	37.9	2.49	0.07	1.37	2.4	47.3	65.8
56.2	42.5	2.50	0.07	1.36	2.4	46.9	68.6
63.1	46.9	2.34	0.07	1.39	2.3	47.9	70.5
69.9	51.1	2.15	0.06	1.38	2.1	47.5	71.8
76.8	54.9	2.08	0.06	1.39	2.0	47.6	74.3
83.6	58.7	2.05	0.06	1.39	2.0	47.8	77.7
90.4	62.2	1.86	0.05	1.41	1.8	48.5	79.6
97.3	65.6	1.73	0.05	1.39	1.7	48.0	81.4
104.1	68.6	1.68	0.05	1.41	1.6	48.5	84.6
110.9	71.6	1.60	0.05	1.41	1.6	48.6	89.5
117.8	74.4	1.44	0.04	1.40	1.4	48.2	90.4
124.6	76.9	1.31	0.04	1.41	1.3	48.6	91.9
131.5	79.3	1.27	0.04	1.40	1.3	48.1	96.8
138.3	81.5	1.17	0.03	1.40	1.2	48.2	99.2
145.1	83.5	1.04	0.03	1.40	1.0	48.3	99.8
152.0	85.2	0.95	0.03	1.40	0.9	48.3	100.9
158.8	86.9	0.93	0.03	1.42	0.9	48.9	108.4
165.7	88.5	0.84	0.02	1.41	0.8	48.4	114.2
172.5	89.9	0.74	0.02	1.41	0.7	48.5	115.1
179.3	91.1	0.70	0.02	1.41	0.7	48.4	116.8
186.2	92.3	0.60	0.02	1.41	0.6	48.5	115.1
193.0	93.2	0.49	0.01	1.43	0.5	49.1	112.4
199.9	94.0	0.45	0.01	1.40	0.4	48.3	116.1
206.7	94.7	0.43	0.01	1.41	0.4	48.5	114.4
213.5	95.4	0.38	0.01	1.43	0.4	49.1	117.4
220.4	96.0	0.34	0.01	1.43	0.3	49.2	122.8
227.2	96.5	0.29	0.01	1.42	0.3	48.9	119.6
234.1	96.9	0.23	0.01	1.42	0.2	48.8	110.2
240.9	97.3	0.23	0.01	1.42	0.2	49.0	106.8
247.8	97.6	0.20	0.01	1.43	0.2	49.3	110.8
254.6	97.9	0.15	0.00	1.42	0.2	48.9	96.9
261.4	98.1	0.14	0.00	1.43	0.1	49.3	91.0
268.3	98.3	0.12	0.00	1.41	0.1	48.5	96.3
275.1	98.4	0.12	0.00	1.41	0.1	48.7	82.3

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.4	3.3	43.3	53.7
8.3	3.2	43.4	50.5
15.2	3.1	43.5	48.9
22.0	3.0	43.8	47.9
28.9	3.0	44.4	47.3
35.7	2.9	45.1	47.2
42.5	2.9	45.6	46.8
49.4	2.8	45.8	46.1
56.2	2.8	46.0	45.4
63.1	2.7	46.1	44.6
69.9	2.7	46.3	43.8
76.8	2.6	46.4	42.9
83.6	2.6	46.5	42.1
90.4	2.5	46.6	41.3
97.3	2.5	46.8	40.4
104.1	2.4	46.9	39.5
110.9	2.4	47.0	38.7
117.8	2.3	47.0	37.9
124.6	2.3	47.1	37.0
131.5	2.2	47.2	36.2
138.3	2.2	47.2	35.3
145.1	2.1	47.3	34.5
152.0	2.1	47.3	33.6
158.8	2.0	47.4	32.8
165.7	2.0	47.4	32.0
172.5	1.9	47.5	31.3
179.3	1.9	47.5	30.5
186.2	1.8	47.5	29.7
193.0	1.8	47.6	29.0
199.9	1.7	47.6	28.2
206.7	1.7	47.6	27.5
213.5	1.7	47.7	26.8
220.4	1.6	47.7	26.1
227.2	1.6	47.8	25.5
234.1	1.5	47.8	24.8
240.9	1.5	47.8	24.2
247.8	1.5	47.9	23.6
254.6	1.4	47.9	23.1
261.4	1.4	47.9	22.5
268.3	1.4	48.0	22.0
275.1	1.3	48.0	21.5

NOTES:  
 (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-7

RUN 626

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DLG. F - RV) -55.00  
 PRESSURE (PSIG) 20.00  
 SUPERFICIAL VELOCITY (CM/SEC) 18.87  
 COKE CHANGE (GM) 1.00  
 % CARBON IN COKE 46.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... IRG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HK
1.7	1.1	1.61	0.11	2.79	3.7	96.1	46.6
8.6	7.6	1.69	0.11	2.79	3.6	96.1	63.0
15.4	14.5	1.98	0.11	2.79	4.0	95.9	71.4
22.3	21.6	1.98	0.11	2.79	3.9	96.0	76.4
29.1	28.5	1.89	0.11	2.79	3.8	96.2	83.0
36.0	35.1	1.77	0.10	2.80	3.5	96.4	86.8
42.8	41.3	1.70	0.10	2.80	3.4	96.5	92.4
49.6	47.4	1.65	0.10	2.81	3.3	96.6	97.6
56.5	53.0	1.45	0.08	2.82	2.9	97.0	100.0
63.3	58.1	1.37	0.06	2.82	2.7	97.2	104.5
70.2	63.0	1.31	0.06	2.83	2.6	97.3	112.4
77.0	67.6	1.22	0.07	2.83	2.4	97.5	119.3
83.8	71.8	1.07	0.06	2.84	2.1	97.8	121.9
90.7	75.5	0.95	0.05	2.85	1.9	98.1	126.1
97.5	78.9	0.90	0.05	2.85	1.8	98.2	134.0
104.4	81.9	0.79	0.05	2.86	1.6	98.4	139.3
111.2	84.6	0.68	0.04	2.86	1.4	98.6	141.5
118.0	86.9	0.59	0.03	2.87	1.2	98.8	145.0
124.9	88.9	0.54	0.03	2.87	1.1	98.9	153.3
131.7	90.8	0.49	0.03	2.88	1.0	99.0	163.8
138.6	92.4	0.41	0.02	2.88	0.8	99.1	169.9
145.4	93.7	0.35	0.02	2.88	0.7	99.3	174.9
152.3	94.9	0.30	0.02	2.89	0.6	99.4	182.0
159.1	95.8	0.25	0.01	2.89	0.5	99.5	187.4
165.9	96.7	0.21	0.01	2.89	0.4	99.6	188.6
172.8	97.3	0.16	0.01	2.89	0.3	99.7	174.1
179.6	97.7	0.13	0.01	2.90	0.3	99.7	165.0
186.4	98.1	0.11	0.01	2.90	0.2	99.8	159.5
193.3	98.4	0.08	0.00	2.90	0.2	99.8	145.3
200.1	98.6	0.06	0.00	2.90	0.1	99.8	125.7
207.0	98.8	0.05	0.00	2.90	0.1	99.9	112.6
213.8	98.9	0.04	0.00	2.90	0.1	99.9	110.4

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HK
1.7	3.7	96.1	39.9
8.6	3.7	96.1	53.2
15.4	3.8	96.1	56.2
22.3	3.8	96.0	58.0
29.1	3.8	96.0	58.7
36.0	3.8	96.1	58.5
42.8	3.8	96.2	57.9
49.6	3.7	96.2	57.3
56.5	3.6	96.3	55.3
63.3	3.5	96.4	55.1
70.2	3.5	96.5	55.9
77.0	3.4	96.5	52.7
83.8	3.3	96.6	51.4
90.7	3.2	96.7	49.9
97.5	3.1	96.8	46.5
104.4	3.0	96.9	47.1
111.2	2.9	97.0	45.6
118.0	2.8	97.1	44.2
124.9	2.7	97.2	42.7
131.7	2.6	97.3	41.3
138.6	2.5	97.4	40.0
145.4	2.5	97.5	38.7
152.3	2.4	97.6	37.4
159.1	2.3	97.6	36.1
165.9	2.2	97.7	34.9
172.8	2.2	97.8	35.8
179.6	2.1	97.9	32.6
186.4	2.0	97.9	31.6
193.3	2.0	98.0	30.6
200.1	1.9	98.1	29.6
207.0	1.8	98.1	28.6
213.8	1.8	98.2	27.6

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-2

RUN 629

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -130.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 18.82  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.2	0.4	0.78	0.05	2.85	1.6	98.1	19.0
8.9	2.7	0.52	0.03	2.87	1.1	98.8	16.0
15.9	4.3	0.57	0.03	2.87	1.2	98.7	15.9
22.7	6.1	0.65	0.04	2.86	1.3	98.6	18.9
29.5	8.3	0.68	0.04	2.86	1.4	98.6	21.1
36.3	10.5	0.69	0.04	2.86	1.4	98.6	22.6
43.2	12.9	0.71	0.04	2.86	1.4	98.5	24.1
50.0	15.3	0.71	0.04	2.86	1.4	98.5	25.3
56.8	17.8	0.69	0.04	2.86	1.4	98.6	25.5
63.7	20.1	0.65	0.04	2.87	1.3	98.7	25.3
70.4	22.3	0.64	0.04	2.87	1.3	98.7	25.5
77.3	24.6	0.63	0.04	2.87	1.3	98.7	26.2
84.1	26.8	0.63	0.04	2.87	1.3	98.7	27.2
90.8	29.1	0.63	0.04	2.87	1.3	98.7	26.6
97.7	31.5	0.66	0.04	2.87	1.3	98.6	30.3
104.5	33.8	0.64	0.04	2.87	1.3	98.7	31.4
111.4	36.2	0.64	0.04	2.87	1.3	98.7	32.2
118.2	38.5	0.61	0.04	2.87	1.2	98.7	32.5
124.9	40.7	0.59	0.03	2.87	1.2	98.8	32.7
131.8	42.9	0.57	0.03	2.87	1.2	98.8	33.0
138.5	44.9	0.54	0.03	2.87	1.1	98.9	33.3
145.4	47.0	0.54	0.03	2.87	1.0	99.0	33.7
152.2	49.0	0.51	0.03	2.87	1.0	99.0	34.9
159.0	50.9	0.51	0.03	2.87	1.0	98.9	36.6
165.9	52.9	0.52	0.03	2.87	1.0	99.0	37.8
172.7	54.8	0.50	0.03	2.88	1.0	99.0	38.9
179.6	56.8	0.49	0.03	2.86	0.9	99.1	39.7
186.4	58.7	0.47	0.03	2.88	0.9	99.1	40.3
193.1	60.5	0.32	0.03	2.88	0.9	99.1	28.3
199.9	62.2	0.31	0.03	2.88	0.9	99.1	28.0
216.6	63.8	0.42	0.02	2.88	0.8	99.2	41.0
223.4	65.5	0.39	0.02	2.88	0.8	99.2	40.9
230.1	67.0	0.38	0.02	2.88	0.8	99.2	41.3
236.8	68.5	0.36	0.02	2.88	0.7	99.3	41.5
243.6	69.9	0.35	0.02	2.88	0.7	99.3	41.5
250.3	71.3	0.33	0.02	2.89	0.7	99.3	42.4
257.1	72.6						

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.2	1.6	98.1	20.5
8.9	1.4	98.4	17.8
15.9	1.3	98.6	16.1
22.7	1.3	98.6	16.2
29.5	1.3	98.6	16.8
36.3	1.3	98.6	17.4
43.2	1.3	98.6	17.9
50.0	1.3	98.6	18.4
56.8	1.3	98.6	18.7
63.7	1.3	98.6	18.9
70.4	1.3	98.6	19.0
77.3	1.3	98.6	19.1
84.1	1.3	98.6	19.2
90.8	1.3	98.6	19.2
97.7	1.3	98.6	19.3
104.5	1.3	98.6	19.4
111.4	1.3	98.6	19.5
118.2	1.3	98.6	19.6
124.9	1.3	98.6	19.6
131.8	1.3	98.6	19.5
138.5	1.3	98.7	19.5
145.4	1.3	98.7	19.4
152.2	1.3	98.7	19.3
159.0	1.3	98.7	19.2
165.9	1.3	98.7	19.1
172.7	1.2	98.7	19.0
179.6	1.2	98.7	19.0
186.4	1.2	98.7	18.9
193.1	1.2	98.7	18.8
199.9	1.2	98.8	18.7
216.6	1.2	98.8	17.7
223.4	1.2	98.8	17.6
230.1	1.2	98.8	17.5
236.8	1.2	98.8	17.4
243.6	1.1	98.8	17.2
250.3	1.1	98.8	17.1
257.1	1.1	98.8	16.9

NOTES:  
 (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-9

MUN 650

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -5.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 16.63  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 46.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM		GAS ANALYSIS(DRY), VOL %		KATE (1)
	CARBON	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO	CO <sub>2</sub>	% CARBON/HR
1.2	2.0	3.23	0.19	2.70	6.6	93.1	102.5
8.1	13.4	3.23	0.19	2.71	6.6	93.3	115.0
14.9	24.9	3.17	0.16	2.72	6.3	93.3	126.9
21.8	35.5	2.61	0.16	2.74	5.6	94.3	139.3
28.6	45.3	2.67	0.16	2.75	5.4	94.3	149.0
35.5	54.1	2.29	0.13	2.77	4.6	95.3	155.4
42.2	61.5	1.94	0.11	2.79	3.9	96.0	159.8
49.1	68.0	1.73	0.10	2.80	3.3	96.4	166.9
55.8	73.6	1.47	0.09	2.82	3.0	96.9	170.4
62.6	76.2	1.19	0.07	2.83	2.4	97.3	172.0
69.5	82.1	1.07	0.06	2.84	2.1	97.8	181.6
76.3	85.6	0.91	0.05	2.85	1.8	98.1	190.6
83.0	88.4	0.74	0.04	2.86	1.3	98.4	196.2
89.8	90.7	0.66	0.04	2.86	1.3	98.6	212.4
96.6	92.8	0.57	0.03	2.87	1.2	98.6	232.9
103.4	94.5	0.44	0.03	2.88	0.9	99.0	236.1
110.1	95.7	0.34	0.02	2.88	0.7	99.3	236.2
116.8	96.6	0.26	0.02	2.89	0.6	99.4	244.6
123.6	97.5	0.21	0.01	2.89	0.4	99.5	247.1
130.3	98.1	0.18	0.01	2.89	0.4	99.6	255.4
137.1	98.6	0.14	0.01	2.90	0.3	99.7	269.3
143.9	99.0	0.14	0.01	2.90	0.3	99.7	360.1
151.5	99.4	0.11	0.01	2.90	0.2	99.7	340.7
158.2	99.8	0.10	0.01	2.90	0.2	99.6	1164.6

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL %		KATE (2)
	CO	CO <sub>2</sub>	% CARBON/HR
1.2	6.6	93.1	101.6
8.1	6.6	93.2	99.3
14.9	6.5	93.3	99.6
21.8	6.4	93.5	97.8
28.6	6.1	93.7	95.0
35.5	5.9	93.9	91.3
42.2	5.7	94.2	87.4
49.1	5.4	94.5	83.2
55.8	5.1	94.6	79.1
62.6	4.9	95.0	74.9
69.5	4.6	95.3	70.9
76.3	4.4	95.5	67.3
83.0	4.2	95.7	63.8
89.8	4.0	95.9	60.6
96.6	3.8	96.1	57.6
103.4	3.6	96.3	54.8
110.1	3.4	96.5	52.2
116.8	3.3	96.7	49.7
123.6	3.1	96.8	47.3
130.3	3.0	96.9	45.2
137.1	2.8	97.1	43.2
143.9	2.7	97.2	41.3
151.5	2.6	97.3	39.4
158.2	2.5	97.4	37.8

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-10

RUN 431

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -130.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 18.88  
 COKE CHARGE (G) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.2	0.4	0.81	0.05	2.84	1.8	97.9	21.1
8.0	2.9	0.68	0.04	2.86	1.4	98.3	21.1
14.8	5.1	0.72	0.04	2.86	1.5	98.4	21.1
21.6	7.4	0.73	0.04	2.86	1.5	98.4	23.4
29.3	10.3	0.80	0.05	2.86	1.6	98.3	26.4
36.2	13.2	0.85	0.05	2.85	1.7	98.2	29.5
43.0	16.1	0.84	0.05	2.85	1.7	98.2	30.7
49.8	19.1	0.81	0.05	2.86	1.6	98.3	31.1
56.7	21.9	0.79	0.05	2.86	1.6	98.4	31.7
63.5	24.7	0.78	0.05	2.86	1.6	98.4	33.0
70.4	27.6	0.79	0.05	2.86	1.6	98.4	34.4
77.2	30.4	0.78	0.05	2.86	1.6	98.4	36.1
84.0	33.3	0.78	0.05	2.86	1.6	98.4	37.6
90.9	36.1	0.76	0.04	2.86	1.5	98.4	38.7
97.7	38.9	0.73	0.04	2.86	1.5	98.5	38.7
104.6	41.5	0.68	0.04	2.86	1.4	98.6	38.7
111.4	44.1	0.68	0.04	2.86	1.4	98.6	39.4
118.2	46.6	0.66	0.04	2.87	1.3	98.6	40.6
125.1	49.0	0.64	0.04	2.87	1.3	98.7	41.9
131.9	51.4	0.65	0.04	2.87	1.3	98.7	44.1
138.8	53.9	0.64	0.04	2.87	1.3	98.7	45.8
145.6	56.3	0.60	0.04	2.87	1.2	98.8	46.5
152.4	58.5	0.58	0.03	2.87	1.2	98.8	47.2
159.3	60.7	0.57	0.03	2.87	1.1	98.8	48.2
166.1	62.8	0.54	0.03	2.87	1.1	98.9	48.6
173.0	64.8	0.51	0.03	2.87	1.0	99.0	48.7
179.8	66.7	0.48	0.03	2.88	1.0	99.0	48.8
186.6	68.5	0.45	0.03	2.88	0.9	99.1	49.4
193.5	70.3	0.45	0.03	2.88	0.9	99.1	51.0
200.3	72.0	0.43	0.03	2.88	0.9	99.1	52.9
207.2	73.7	0.43	0.02	2.88	0.9	99.1	54.8
214.0	75.3	0.41	0.02	2.88	0.8	99.2	56.4
220.8	76.8	0.39	0.02	2.88	0.8	99.2	57.7
227.7	78.3	0.37	0.02	2.88	0.7	99.2	58.7
234.5	79.7	0.35	0.02	2.88	0.7	99.3	59.5
241.4	81.1	0.33	0.02	2.88	0.7	99.3	60.1
248.2	82.3	0.31	0.02	2.89	0.6	99.4	60.3
255.0	83.5	0.29	0.02	2.89	0.6	99.4	59.9
261.9	84.6	0.27	0.02	2.89	0.5	99.4	59.3
268.7	85.6	0.25	0.01	2.89	0.5	99.5	58.8
275.6	86.5	0.23	0.01	2.89	0.5	99.5	57.8
282.3	87.4	0.21	0.01	2.89	0.4	99.6	57.0
289.1	88.1	0.20	0.01	2.89	0.4	99.6	56.8
295.8	88.9	0.18	0.01	2.89	0.4	99.6	56.5
302.6	89.6	0.17	0.01	2.89	0.3	99.7	55.4
309.3	90.2	0.15	0.01	2.90	0.3	99.7	54.5
316.0	90.8	0.15	0.01	2.90	0.3	99.7	53.5
322.7	91.3	0.13	0.01	2.90	0.3	99.7	52.1
329.4	91.8	0.12	0.01	2.90	0.2	99.8	52.8

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.2	1.8	97.9	20.3
8.0	1.6	98.2	21.5
14.8	1.6	98.3	20.5
21.6	1.5	98.3	20.6
29.3	1.5	98.3	21.1
36.2	1.6	98.3	21.9
43.0	1.6	98.3	22.5
49.8	1.6	98.3	22.9
56.7	1.6	98.3	23.2
63.5	1.6	98.3	23.3
70.4	1.6	98.3	23.5
77.2	1.6	98.3	23.6
84.0	1.6	98.3	23.8
90.9	1.6	98.3	23.9
97.7	1.6	98.3	23.9
104.6	1.6	98.4	23.8
111.4	1.6	98.4	23.7
118.2	1.5	98.4	23.6
125.1	1.5	98.4	23.5
131.9	1.5	98.4	23.4
138.8	1.5	98.4	23.3
145.6	1.5	98.4	23.2
152.4	1.5	98.5	23.0
159.3	1.5	98.5	22.9
166.1	1.4	98.5	22.7
173.0	1.4	98.5	22.5
179.8	1.4	98.5	22.3
186.6	1.4	98.5	22.0
193.5	1.4	98.6	21.8
200.3	1.4	98.6	21.6
207.2	1.3	98.6	21.3
214.0	1.3	98.6	21.1
220.8	1.3	98.6	20.9
227.7	1.3	98.7	20.6
234.5	1.3	98.7	20.4
241.4	1.3	98.7	20.2
248.2	1.2	98.7	19.9
255.0	1.2	98.7	19.6
261.9	1.2	98.7	19.4
268.7	1.2	98.8	19.1
275.6	1.2	98.8	18.8
282.3	1.2	98.8	18.6
289.1	1.1	98.8	18.3
295.8	1.1	98.8	18.0
302.6	1.1	98.9	17.8
309.3	1.1	98.9	17.5
316.0	1.1	98.9	17.2
322.7	1.1	98.9	17.0
329.4	1.0	98.9	16.7

## NOTES:

(1) BASED ON REMAINING CARBON

(2) BASED ON INITIAL CARBON

TABLE D-1

RUN 633

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.07  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE-NITROGEN  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON CO2		PARTIAL PRESSURE, ATM CO CO2		GAS ANALYSIS(DRY), VOL % CO CO2		RATE (1) %CARBON/HR
1.1	1.3	15.10	0.11	0.35	3.9	12.1	57.9
7.8	6.1	5.03	0.04	0.37	1.4	12.8	34.2
15.5	8.9	5.07	0.04	0.35	1.4	12.0	23.9
22.7	11.4	4.82	0.04	0.35	1.3	12.0	23.3
29.5	13.7	4.49	0.03	0.37	1.2	12.7	22.5
36.4	15.9	4.37	0.03	0.38	1.1	13.0	22.6
43.2	18.1	4.31	0.03	0.36	1.1	12.3	22.9
50.1	20.2	4.22	0.03	0.35	1.1	11.9	23.1
56.9	22.3	4.22	0.03	0.35	1.1	11.9	23.1
63.8	24.2	4.19	0.03	0.38	1.1	13.0	23.4
70.6	26.3	4.20	0.03	0.38	1.1	13.0	24.7
77.4	28.4	4.17	0.03	0.37	1.1	12.7	24.4
84.3	30.5	4.08	0.03	0.36	1.1	12.3	25.7
91.1	32.5	3.93	0.03	0.35	1.0	11.9	25.4
97.9	34.4	3.76	0.03	0.35	1.0	11.9	24.4
104.8	36.1	3.55	0.03	0.36	0.9	12.3	22.9
111.6	37.7	3.35	0.03	0.38	0.9	13.1	23.6
118.5	39.5	3.28	0.03	0.35	0.9	12.0	22.4
125.3	40.8	3.19	0.02	0.40	0.8	13.8	22.5
132.1	42.5	3.25	0.02	0.37	0.9	12.8	24.8
139.0	44.0	3.26	0.02	0.38	0.9	13.1	24.9
145.8	45.7	3.25	0.02	0.37	0.8	12.8	27.0
152.7	47.4	3.19	0.02	0.35	0.8	12.0	26.9
159.5	48.9	3.05	0.02	0.35	0.8	12.0	24.7
166.3	50.3	2.85	0.02	0.36	0.7	12.4	21.7
173.2	51.4	2.67	0.02	0.40	0.7	13.8	23.5
180.0	52.9	2.61	0.02	0.36	0.7	12.5	25.9
186.9	54.1	2.56	0.02	0.36	0.7	12.4	24.5
193.7	55.4	2.52	0.02	0.35	0.7	12.1	22.5
200.5	56.4	2.50	0.02	0.38	0.7	13.2	24.6
207.4	57.9	2.53	0.02	0.35	0.7	12.0	27.6
214.2	59.1	2.47	0.02	0.35	0.6	12.1	24.7
221.1	60.2	2.38	0.02	0.36	0.6	12.4	25.3
227.9	61.4	2.27	0.02	0.35	0.6	12.1	22.7
234.8	62.2	2.08	0.02	0.38	0.5	13.2	18.9

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO CO2		RATE (2) %CARBON/HR
1.1	3.9	12.1	72.1
7.8	2.8	12.4	46.4
15.5	2.1	12.4	34.4
22.7	1.8	12.2	30.3
29.5	1.7	12.3	27.9
36.4	1.6	12.4	26.2
43.2	1.5	12.4	25.1
50.1	1.5	12.4	24.2
56.9	1.4	12.4	23.5
63.8	1.4	12.4	22.8
70.6	1.4	12.4	22.4
77.4	1.3	12.5	22.0
84.3	1.3	12.5	21.7
91.1	1.3	12.5	21.4
97.9	1.3	12.4	21.1
104.8	1.3	12.4	20.7
111.6	1.2	12.4	20.3
118.5	1.2	12.4	20.0
125.3	1.2	12.5	19.5
132.1	1.2	12.5	19.3
139.0	1.2	12.5	19.0
145.8	1.1	12.5	18.8
152.7	1.1	12.5	18.6
159.5	1.1	12.5	18.4
166.3	1.1	12.5	18.1
173.2	1.1	12.5	17.8
180.0	1.1	12.5	17.6
186.9	1.1	12.5	17.4
193.7	1.0	12.5	17.2
200.5	1.0	12.5	16.9
207.4	1.0	12.5	16.7
214.2	1.0	12.5	16.5
221.1	1.0	12.5	16.3
227.9	1.0	12.5	16.2
234.8	1.0	12.5	15.9

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-12

RUN 634

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 1.04  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS (DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.1	0.0	0.27	0.01	0.29	0.4	9.9	4.0
7.3	0.9	14.92	0.79	2.03	27.3	69.9	14.6
14.2	3.2	12.32	0.72	2.13	24.6	73.4	18.9
21.0	5.0	13.50	0.69	2.17	23.7	74.7	21.5
27.9	7.9	14.73	0.67	2.20	23.0	75.6	25.7
34.7	10.4	13.26	0.69	2.18	23.7	75.0	23.0
41.5	12.6	11.73	0.68	2.19	23.4	75.4	20.4
48.4	14.5	12.28	0.65	2.22	22.5	76.5	22.8
55.2	17.0	14.09	0.65	2.22	22.4	76.6	28.0
62.1	19.8	13.39	0.66	2.22	22.6	76.5	27.6
68.9	22.1	11.37	0.66	2.22	22.7	76.4	25.3
75.8	23.9	11.46	0.64	2.25	21.9	77.3	24.3
82.6	26.3	12.98	0.61	2.27	21.1	78.2	30.0
89.4	29.0	12.61	0.62	2.27	21.3	78.1	30.3
96.3	31.2	10.96	0.62	2.26	21.4	77.9	26.1
103.1	33.1	10.35	0.59	2.30	20.2	79.2	25.9
109.9	35.1	12.20	0.57	2.31	19.8	79.6	32.9
116.8	37.9	12.35	0.58	2.31	20.0	79.4	34.6
123.6	40.0	10.16	0.58	2.31	19.9	79.6	28.8
130.5	41.9	9.55	0.55	2.34	19.1	80.4	28.0
137.3	43.8	11.10	0.53	2.36	18.3	81.2	34.9
144.1	46.4	11.26	0.53	2.36	18.3	81.3	37.5
151.0	48.3	9.64	0.53	2.36	18.4	81.2	32.4
157.8	50.2	8.82	0.51	2.38	17.6	82.0	30.8
164.7	51.8	10.00	0.49	2.40	16.9	82.7	37.4
171.5	54.3	10.39	0.49	2.40	16.8	82.8	41.1
178.3	56.1	8.89	0.49	2.40	17.0	82.6	36.0
185.2	57.9	8.26	0.48	2.41	16.5	83.1	34.7
192.0	59.5	8.77	0.46	2.44	15.7	83.8	39.4
198.9	61.5	9.47	0.45	2.44	15.6	83.8	45.0
205.7	63.4	8.40	0.46	2.43	15.7	83.7	42.0
212.5	65.0	7.16	0.46	2.43	15.8	83.5	36.8
219.4	66.4	6.95	0.44	2.44	15.3	83.9	37.0
226.2	67.9	7.77	0.42	2.46	14.5	84.7	44.8
233.1	69.6	7.65	0.42	2.45	14.6	84.5	46.5
239.9	71.1	6.52	0.43	2.45	14.7	84.3	41.1
246.8	72.4	5.76	0.42	2.45	14.6	84.4	37.6
253.6	73.4	5.48	0.40	2.47	13.8	85.1	37.8
260.4	74.6	6.60	0.39	2.48	13.5	85.4	48.5
267.3	76.2	6.67	0.40	2.48	13.6	85.2	52.0
274.1	77.5	6.54	0.40	2.47	13.7	85.2	47.0

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.1	0.4	9.9	0.3
7.3	11.4	34.4	7.0
14.2	18.1	51.5	13.6
21.0	19.8	57.9	14.4
27.9	20.8	63.0	16.9
34.7	21.4	65.6	18.0
41.5	21.7	67.0	18.1
48.4	21.9	68.1	18.0
55.2	21.9	69.3	18.5
62.1	22.0	70.2	19.1
68.9	22.1	70.8	19.2
75.8	22.1	71.2	19.0
82.6	22.0	71.8	19.1
89.4	22.0	72.3	19.4
96.3	21.9	72.7	19.4
103.1	21.8	73.1	19.3
109.9	21.7	73.4	19.2
116.8	21.6	73.9	19.5
123.6	21.5	74.2	19.4
130.5	21.4	74.4	19.3
137.3	21.3	74.7	19.1
144.1	21.1	75.1	19.3
151.0	21.0	75.4	19.2
157.8	20.9	75.6	19.1
164.7	20.8	75.9	18.9
171.5	20.6	76.2	19.0
178.3	20.4	76.4	18.9
185.2	20.3	76.7	18.8
192.0	20.2	76.9	18.6
198.9	20.0	77.1	18.6
205.7	19.8	77.4	18.5
212.5	19.7	77.5	18.4
219.4	19.6	77.7	18.1
226.2	19.5	77.9	18.0
233.1	19.4	78.1	17.9
239.9	19.2	78.2	17.8
246.8	19.1	78.4	17.6
253.6	19.1	78.5	17.4
260.4	18.9	78.6	17.2
267.3	18.8	78.8	17.1
274.1	18.7	78.9	17.0

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

RUN 636

GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 30.00  
 SUPERFICIAL VELOCITY (CM/SEC) 0.51  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

GEOS DATA

CONVERSION, % CARBON CO2		PARTIAL PRESSURE, ATM CO CO2		GAS ANALYSIS(DRY), VOL % CO CO2		RATE (1) %CARBON/HR
0.0	0.0	0.0	0.0	0.0	0.0	4.4
0.9	27.59	1.32	1.58	43.5	51.8	11.9
2.6	19.81	1.07	1.89	35.1	62.1	15.5
4.4	18.65	1.05	1.92	34.4	63.2	14.3
5.7	16.87	1.03	1.94	34.0	63.9	12.6
7.1	17.67	0.99	1.99	32.6	65.5	14.4
8.8	22.37	1.00	1.98	33.0	65.2	19.7
11.2	24.30	0.99	2.00	32.6	65.8	22.2
13.3	19.76	0.95	2.04	31.3	67.2	18.5
14.8	17.10	0.96	2.04	31.6	67.0	15.9
16.4	16.98	0.95	2.05	31.3	67.3	16.1
17.9	15.28	0.94	2.07	30.8	67.9	14.7
19.1	15.14	0.93	2.08	30.5	68.3	15.2
20.7	18.68	0.93	2.07	30.6	68.2	19.5
22.6	20.76	0.90	2.11	29.7	69.2	23.3
24.8	20.84	0.91	2.10	29.8	69.2	24.0
26.7	17.81	0.89	2.12	29.2	69.8	20.7
28.2	15.44	0.87	2.14	28.5	70.5	18.2
29.7	14.52	0.85	2.16	28.0	71.1	17.8
31.1	13.27	0.85	2.16	28.0	71.1	16.0
32.2	13.89	0.81	2.20	26.8	72.4	17.8
33.8	16.94	0.81	2.20	26.8	72.3	22.9
35.7	19.23	0.81	2.21	26.6	72.5	26.6
37.7	17.26	0.77	2.24	25.5	73.7	25.5
39.3	14.59	0.79	2.23	25.8	73.4	21.6
40.7	14.45	0.78	2.24	25.6	73.6	21.6
42.2	12.75	0.75	2.27	24.5	74.7	20.0
43.3	11.72	0.75	2.27	24.7	74.6	18.6
44.6	13.24	0.74	2.28	24.4	75.0	21.7
46.1	15.62	0.73	2.30	23.9	75.5	26.9
47.9	17.03	0.72	2.30	23.6	75.8	30.8
49.7	14.94	0.72	2.30	23.6	75.8	27.5
51.0	12.40	0.70	2.33	22.9	76.5	23.4
52.3	12.11	0.68	2.34	22.3	77.1	23.8
53.6	11.64	0.68	2.34	22.4	77.0	23.2
54.8	10.80	0.66	2.36	21.7	77.7	22.3
55.9	12.10	0.65	2.37	21.4	78.0	26.1
57.4	14.69	0.66	2.37	21.7	77.8	32.3
59.1	14.51	0.63	2.39	20.7	78.8	34.0
60.6	12.53	0.62	2.40	20.5	78.9	30.4
61.8	11.20	0.63	2.40	20.7	78.8	27.4
63.0	10.68	0.60	2.43	19.7	79.8	27.7
64.1	9.80	0.60	2.43	19.7	79.8	26.0
65.1	9.27	0.59	2.43	19.5	80.0	25.1
66.1	10.20	0.58	2.45	19.0	80.5	26.9

DATA

GAS ANALYSIS(DRY), VOL % CO CO2		RATE (2) %CARBON/HR
0.0	0.0	0.0
21.6	25.7	5.7
29.5	39.6	9.5
31.2	47.1	11.1
31.8	50.7	11.4
		11.7
		13.2
32.3	58.5	13.8
32.2	59.4	13.8
32.1	60.1	13.8
32.0	60.7	13.7
31.9	61.2	13.5
31.8	61.7	13.5
31.7	62.3	13.8
		14.1
		14.3
31.3	67.7	14.2
31.1	64.0	14.2
31.0	64.4	14.0
30.8	64.6	13.9
30.6	65.0	13.9
30.4	65.4	14.0
30.2	65.8	14.1
30.0	66.2	14.1
29.8	66.4	14.1
29.7	66.7	14.1
		14.5
		13.8
	67.8	13.8
28.7	68.1	13.9
28.6	68.4	13.8
28.4	68.6	13.8
28.3	68.8	13.7
28.1	69.0	13.6
28.0	69.2	13.5
27.8	69.5	13.5
27.6	69.8	13.5
27.3	70.0	13.5
27.2	70.2	13.4
27.0	70.4	13.3
26.9	70.7	13.3
26.7	70.8	13.2
26.6	71.0	13.1

BASED ON REMAINING CARBON  
 BASED ON INITIAL CARBON



TABLE D-14

ROR CSR

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEL. F ~ RV) -55.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 18.93  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 44.28  
 % ASH IN COKE 50.78  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (100/150 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM		GAS ANALYSIS (DRY), VOL %		RATE (1) %CARBON/HK
	CARBON	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO	CO <sub>2</sub>	
1.2	1.5	2.47	0.16	2.74	5.4	94.4	85.7
9.3	13.5	2.39	0.14	2.76	4.7	95.1	100.4
16.1	23.1	2.46	0.14	2.76	4.9	95.0	111.1
22.9	32.9	2.40	0.14	2.76	4.8	95.1	125.8
29.8	42.1	2.16	0.12	2.78	4.5	95.0	132.8
36.6	50.4	2.01	0.12	2.79	4.0	95.9	143.8
43.4	58.3	1.91	0.11	2.79	3.8	96.1	157.2
50.3	65.3	1.62	0.09	2.81	3.2	96.7	165.9
57.1	71.4	1.40	0.08	2.82	2.8	97.1	174.2
63.9	76.7	1.28	0.07	2.83	2.5	97.4	186.9
70.7	81.4	1.09	0.06	2.84	2.1	97.8	202.0
77.6	85.3	0.88	0.05	2.85	1.7	98.2	210.8
84.3	88.4	0.77	0.04	2.86	1.5	98.4	222.6
91.5	91.2	0.62	0.04	2.87	1.2	98.7	238.5
98.3	93.3	0.49	0.03	2.88	1.0	99.0	248.1
105.1	95.0	0.40	0.02	2.88	0.8	99.2	260.3
111.9	96.3	0.32	0.02	2.89	0.6	99.3	268.7
118.8	97.2	0.21	0.01	2.89	0.4	99.5	246.6
125.6	97.9	0.16	0.01	2.89	0.3	99.6	207.0
132.4	98.3	0.11	0.01	2.90	0.2	99.8	201.7

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY), VOL %		RATE (2) %CARBON/HK
	CO	CO <sub>2</sub>	
1.2	5.4	94.4	70.2
9.3	5.1	94.7	87.1
16.1	5.0	94.9	80.2
22.9	4.9	94.9	86.1
29.8	4.8	95.0	84.8
36.6	4.7	95.2	82.7
43.4	4.6	95.3	80.5
50.3	4.4	95.4	78.0
57.1	4.3	95.6	75.0
63.9	4.1	95.8	72.0
70.7	3.9	96.0	69.0
77.6	3.8	96.2	65.9
84.3	3.6	96.3	62.9
91.5	3.4	96.5	59.8
98.3	3.2	96.7	57.0
105.1	3.1	96.8	54.2
111.9	3.0	97.0	51.6
118.8	2.8	97.1	47.1
125.6	2.7	97.2	46.7
132.4	2.6	97.4	44.5

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-15

RUN 638

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 27.50  
 SUPERFICIAL VELOCITY (CM/SEC) 19.22  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 44.28  
 % ASH IN COKE 50.18  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... IKG ILLINOIS COKE (100/150 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/H
1.1	1.3	2.12	0.13	2.73	4.5	95.1	75.5
8.8	11.6	2.43	0.14	2.73	4.8	95.0	93.4
15.8	21.4	2.37	0.14	2.73	4.7	95.2	105.0
22.6	30.6	2.23	0.13	2.74	4.4	95.5	113.9
29.4	39.4	2.14	0.12	2.75	4.3	95.7	124.5
36.3	47.8	2.02	0.12	2.75	4.0	95.9	135.0
43.1	55.5	1.77	0.10	2.77	3.5	96.4	141.6
50.0	62.2	1.56	0.09	2.78	3.1	96.8	148.0
56.8	68.2	1.45	0.08	2.79	2.9	97.1	160.0
63.6	73.8	1.30	0.07	2.79	2.6	97.3	173.1
70.5	78.6	1.12	0.06	2.81	2.2	97.7	181.5
77.3	82.6	0.92	0.05	2.82	1.8	98.1	187.9
84.2	86.0	0.80	0.05	2.82	1.6	98.4	199.2
91.0	89.0	0.70	0.04	2.83	1.4	98.6	217.0
97.8	91.5	0.59	0.03	2.84	1.2	98.8	234.0
104.7	93.5	0.47	0.03	2.84	0.9	99.0	244.3
111.5	95.1	0.37	0.02	2.85	0.7	99.2	249.8
118.4	96.3	0.29	0.02	2.85	0.6	99.4	257.1
125.2	97.3	0.24	0.01	2.86	0.5	99.5	285.1
132.0	98.1	0.23	0.01	2.86	0.5	99.5	376.8
138.9	98.9	0.22	0.01	2.86	0.4	99.5	640.6

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.1	4.5	95.1	68.2
8.8	4.6	95.1	79.3
15.8	4.7	95.1	81.4
22.6	4.7	95.2	81.3
29.4	4.6	95.3	80.3
36.3	4.5	95.4	79.1
43.1	4.4	95.5	77.2
50.0	4.2	95.6	74.7
56.8	4.1	95.8	72.1
63.6	3.9	95.9	69.5
70.5	3.8	96.1	66.9
77.3	3.6	96.3	64.1
84.2	3.5	96.4	61.3
91.0	3.3	96.6	58.7
97.8	3.2	96.7	56.1
104.7	3.1	96.9	53.6
111.5	2.9	97.0	51.2
118.4	2.8	97.1	48.8
125.2	2.7	97.3	46.6
132.0	2.6	97.4	44.6
138.9	2.4	97.5	42.7

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE 5-10

RUN 640

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 30.00  
 SUPERFICIAL VELOCITY (CM/SEC) 5.06  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 44.28  
 % ASH IN COKE 50.18  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... 1KG ILLINOIS COKE (100/150 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM		GAS ANALYSIS(DRY), VOL %		RATE (1)
	CARBON	CO <sub>2</sub>	CO	CO <sub>2</sub>	CO	CO <sub>2</sub>	%CARBON/HR
1.1	0.0	0.01	0.00	0.04	0.0	1.4	4.4
8.8	1.1	14.23	0.90	2.07	29.5	68.0	16.9
16.6	4.3	14.87	0.85	2.13	28.0	70.1	28.3
23.5	7.7	15.95	0.81	2.18	26.7	71.6	32.7
30.4	11.2	14.62	0.80	2.20	26.2	72.3	30.8
37.2	14.0	12.70	0.79	2.21	25.8	72.8	26.9
44.1	16.5	13.12	0.76	2.24	25.0	73.7	29.7
50.9	19.6	14.71	0.76	2.25	24.9	73.9	35.8
57.8	23.1	14.09	0.77	2.24	25.3	73.6	34.8
64.6	25.7	11.79	0.73	2.28	24.0	75.0	30.1
71.4	28.2	12.16	0.70	2.31	23.2	75.8	32.9
78.3	31.1	13.60	0.70	2.31	23.0	76.0	39.6
85.1	34.4	12.95	0.71	2.31	23.2	75.9	38.7
91.9	36.9	10.90	0.67	2.34	22.2	77.0	33.7
98.8	39.2	11.03	0.65	2.36	21.5	77.7	36.6
105.6	42.0	12.60	0.66	2.36	21.7	77.5	43.4
112.5	45.0	11.84	0.63	2.38	20.8	78.4	43.4
119.3	47.4	9.82	0.61	2.41	20.0	79.3	38.2
126.1	49.6	10.17	0.62	2.40	20.2	79.1	40.5
133.0	52.1	11.28	0.59	2.43	19.5	79.8	47.9
139.8	54.8	10.26	0.56	2.46	18.4	80.9	47.4
146.7	57.0	9.01	0.56	2.46	18.3	81.0	43.1
153.5	59.0	9.10	0.55	2.47	18.1	81.3	45.1
160.3	61.2	9.70	0.52	2.50	17.1	82.3	52.1
167.2	63.6	9.36	0.50	2.52	16.5	83.0	54.2
174.0	65.7	8.15	0.49	2.53	16.2	83.2	49.2
180.9	67.5	7.47	0.47	2.55	15.5	83.9	47.9
187.7	69.2	8.19	0.46	2.57	15.0	84.5	56.8
194.5	71.5	8.53	0.46	2.57	15.0	84.5	63.0
201.4	73.3	7.26	0.44	2.59	14.4	85.1	57.2
208.2	74.9	6.57	0.42	2.61	13.7	85.9	56.0
215.1	76.5	7.23	0.41	2.62	13.5	86.0	65.8
221.9	78.5	7.56	0.40	2.62	13.3	86.2	74.0
228.8	80.2	6.33	0.37	2.65	12.3	87.2	68.8
235.6	81.6	5.71	0.36	2.67	11.9	87.7	66.6
242.4	83.0	6.10	0.35	2.67	11.6	87.9	77.1
249.3	84.6	6.36	0.35	2.68	11.4	88.2	88.0
256.1	86.1	5.71	0.32	2.70	10.7	88.9	88.7
262.9	87.4	4.87	0.31	2.72	10.1	89.5	84.6
269.8	88.5	4.97	0.31	2.72	10.1	89.5	92.2
276.6	89.8	5.17	0.29	2.74	9.6	90.0	114.7

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL %		RATE (2)
	CO	CO <sub>2</sub>	%CARBON/HR
1.1	0.0	1.4	0.1
8.8	13.2	31.1	7.7
16.6	20.5	49.0	15.6
23.5	22.8	56.4	19.7
30.4	23.8	60.4	22.2
37.2	24.2	62.5	22.5
44.1	24.4	64.1	22.5
50.9	24.4	65.5	23.1
57.8	24.5	66.6	24.0
64.6	24.5	67.4	23.9
71.4	24.5	68.1	23.7
78.3	24.5	68.8	23.9
85.1	24.2	69.5	24.2
91.9	24.1	69.9	24.1
98.8	24.0	70.4	23.8
105.6	23.8	70.9	23.8
112.5	23.6	71.4	24.0
119.3	23.5	71.8	23.8
126.1	23.3	72.1	23.6
133.0	23.1	72.5	23.5
139.8	22.9	73.0	23.5
146.7	22.7	73.3	23.3
153.5	22.5	73.6	23.1
160.3	22.3	74.0	22.9
167.2	22.0	74.4	22.8
174.0	21.8	74.7	22.6
180.9	21.6	75.0	22.4
187.7	21.4	75.5	22.1
194.5	21.1	75.7	22.0
201.4	20.9	76.0	21.8
208.2	20.7	76.3	21.6
215.1	20.5	76.6	21.3
221.9	20.3	76.9	21.2
228.8	20.1	77.2	21.0
235.6	19.8	77.5	20.8
242.4	19.6	77.7	20.5
249.3	19.4	78.0	20.4
256.1	19.2	78.3	20.2
262.9	18.9	78.6	19.9
269.8	18.8	78.8	19.7
276.6	18.5	79.1	19.5

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-17

RUN 641

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 28.50  
 SUPERFICIAL VELOCITY (CM/SEC) 18.81  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 50.58  
 % ASH IN COKE 44.51  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... IKG ILLINOIS COKE (40/50 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.2	0.9	1.57	0.09	2.84	3.2	96.5	45.4
9.8	7.5	1.59	0.09	2.84	3.2	96.7	51.6
16.8	13.3	1.75	0.10	2.83	3.5	96.4	59.3
23.7	19.4	1.81	0.11	2.83	3.6	96.3	67.8
30.5	25.7	1.85	0.11	2.83	3.7	96.2	75.8
37.4	32.2	1.84	0.11	2.83	3.7	96.2	82.8
44.2	38.5	1.75	0.10	2.83	3.5	96.4	88.9
51.0	44.7	1.71	0.10	2.84	3.4	96.5	93.9
57.9	50.4	1.53	0.09	2.85	3.1	96.8	98.3
64.7	55.8	1.50	0.09	2.85	3.0	96.9	103.4
71.6	60.8	1.34	0.08	2.86	2.7	97.3	108.7
78.4	65.5	1.28	0.08	2.86	2.6	97.4	115.0
85.2	69.9	1.18	0.07	2.87	2.4	97.6	121.3
92.1	73.8	1.05	0.06	2.88	2.1	97.9	126.3
98.9	77.4	0.96	0.06	2.88	1.9	98.0	131.3
105.8	80.6	0.83	0.05	2.89	1.7	98.3	136.1
112.6	83.4	0.77	0.05	2.89	1.5	98.4	140.4
119.4	85.9	0.63	0.04	2.90	1.3	98.7	142.2
126.3	88.0	0.56	0.03	2.90	1.1	98.8	144.4
133.1	89.8	0.49	0.03	2.91	1.0	99.0	146.0
140.0	91.4	0.39	0.02	2.91	0.8	99.2	143.0
146.8	92.7	0.34	0.02	2.92	0.7	99.3	138.6
153.6	93.7	0.27	0.02	2.92	0.5	99.4	130.3
160.5	94.5	0.22	0.01	2.92	0.5	99.5	118.6
167.3	95.2	0.17	0.01	2.93	0.3	99.6	106.0
174.2	95.7	0.15	0.01	2.93	0.3	99.7	95.5
181.0	96.1	0.12	0.01	2.93	0.2	99.7	83.3
187.8	96.4	0.09	0.01	2.93	0.2	99.8	67.8
194.7	96.7	0.07	0.00	2.93	0.1	99.8	59.1
201.5	96.9	0.07	0.00	2.93	0.1	99.8	59.0

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.2	3.2	96.5	43.9
9.8	3.2	96.6	45.8
16.8	3.3	96.6	47.3
23.7	3.3	96.5	49.1
30.5	3.4	96.4	50.6
37.4	3.5	96.4	51.7
44.2	3.5	96.4	52.3
51.0	3.5	96.4	52.5
57.9	3.5	96.4	52.2
64.7	3.4	96.5	51.7
71.6	3.4	96.5	51.0
78.4	3.3	96.6	50.1
85.2	3.2	96.7	49.2
92.1	3.2	96.8	48.1
98.9	3.1	96.8	46.9
105.8	3.0	96.9	45.7
112.6	2.9	97.0	44.5
119.4	2.8	97.1	43.1
126.3	2.7	97.2	41.8
133.1	2.6	97.3	40.5
140.0	2.6	97.4	39.2
146.8	2.5	97.5	37.9
153.6	2.4	97.5	36.6
160.5	2.3	97.6	35.3
167.3	2.2	97.7	34.1
174.2	2.2	97.8	33.0
181.0	2.1	97.9	31.9
187.8	2.0	97.9	30.8
194.7	2.0	98.0	29.8
201.5	1.9	98.1	28.8

## NOTES:

(1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-18

RUN 644

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.37  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 100.00  
 % ASH IN COKE 0.0  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... EMPTY REACTOR

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HK
1.2	0.1	0.39	0.03	2.88	1.0	99.0	5.5
9.4	0.9	0.18	0.01	2.89	0.4	99.6	4.2
17.7	1.3	0.15	0.01	2.90	0.3	99.7	2.7
26.0	1.6	0.14	0.01	2.90	0.3	99.7	2.5
34.4	1.9	0.13	0.01	2.90	0.3	99.7	2.3
42.6	2.3	0.13	0.01	2.90	0.3	99.7	2.3
50.9	2.5	0.12	0.01	2.90	0.2	99.8	2.1
59.1	2.8	0.11	0.01	2.90	0.2	99.8	1.9
67.3	3.1	0.09	0.01	2.90	0.2	99.8	1.7
75.6	3.5	0.08	0.00	2.90	0.2	99.8	1.5
83.9	3.5	0.08	0.00	2.90	0.2	99.8	1.4
92.3	3.6	0.08	0.00	2.90	0.2	99.8	1.4
100.5	3.8	0.07	0.00	2.90	0.1	99.9	1.2
108.8	4.0	0.06	0.00	2.90	0.1	99.9	1.1
117.0	4.1	0.05	0.00	2.90	0.1	99.9	0.9
125.3	4.2	0.04	0.00	2.90	0.1	99.9	0.7
133.6	4.3	0.03	0.00	2.90	0.1	99.9	0.6
142.1	4.4	0.03	0.00	2.90	0.1	99.9	0.5
150.5	4.4	0.02	0.00	2.90	0.0	100.0	0.4
158.9	4.5	0.02	0.00	2.90	0.0	100.0	0.4
167.4	4.5	0.02	0.00	2.90	0.0	100.0	0.3

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY), VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HK
1.2	1.0	99.0	5.4
9.4	0.7	99.3	5.5
17.7	0.5	99.5	4.3
26.0	0.4	99.6	3.7
34.4	0.4	99.6	3.4
42.6	0.4	99.6	3.2
50.9	0.4	99.6	3.0
59.1	0.3	99.7	2.9
67.3	0.3	99.7	2.7
75.6	0.3	99.7	2.6
83.9	0.3	99.7	2.5
92.3	0.3	99.7	2.4
100.5	0.3	99.7	2.3
108.8	0.3	99.7	2.2
117.0	0.2	99.8	2.1
125.3	0.2	99.8	2.0
133.6	0.2	99.8	1.9
142.1	0.2	99.8	1.8
150.5	0.2	99.8	1.8
158.9	0.2	99.8	1.7
167.4	0.2	99.8	1.6

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-19

RUN 645

## GASIFICATION RATES FROM MINIGASIFIER

TEMP. (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 28.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.10  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 100.00  
 % ASH IN COKE 0.0  
 INLET GAS..... CARBON DIOXIDE  
 COKE SAMPLE..... EMPTY REACTOR

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, % CARBON	CO <sub>2</sub>	PARTIAL PRESSURE, ATM CO	CO <sub>2</sub>	GAS ANALYSIS(DRY),VOL % CO	CO <sub>2</sub>	RATE (1) %CARBON/HR
1.1	0.1	0.23	0.02	2.89	0.5	99.5	3.6
9.3	0.6	0.18	0.01	2.89	0.4	99.6	3.5
17.7	1.0	0.19	0.01	2.89	0.4	99.6	3.1
25.9	1.4	0.15	0.01	2.90	0.3	99.7	2.8
34.1	1.8	0.14	0.01	2.90	0.3	99.7	2.4
42.3	2.1	0.12	0.01	2.90	0.2	99.8	2.1
50.6	2.3	0.11	0.01	2.90	0.2	99.8	1.9
58.8	2.6	0.10	0.01	2.90	0.2	99.8	1.7
67.2	2.8	0.09	0.01	2.90	0.2	99.8	1.6
75.5	3.0	0.09	0.01	2.90	0.2	99.8	1.5
83.7	3.2	0.07	0.00	2.90	0.1	99.9	1.3
91.9	3.3	0.06	0.00	2.90	0.1	99.9	1.1
100.3	3.5	0.05	0.00	2.90	0.1	99.9	0.9
108.7	3.6	0.05	0.00	2.90	0.1	99.9	0.9

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY),VOL % CO	CO <sub>2</sub>	RATE (2) %CARBON/HR
1.1	0.5	99.5	3.4
9.3	0.5	99.5	3.7
17.7	0.4	99.6	3.5
25.9	0.4	99.6	3.3
34.1	0.4	99.6	3.1
42.3	0.4	99.6	2.9
50.6	0.3	99.7	2.8
58.8	0.3	99.7	2.6
67.2	0.3	99.7	2.5
75.5	0.3	99.7	2.4
83.7	0.3	99.7	2.3
91.9	0.3	99.7	2.2
100.3	0.2	99.8	2.1
108.7	0.2	99.8	2.0

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-20

Run 646

Minigasifier

Empty

Temperature, RV-°F	55.0
Pressure, psig	36.0
Superficial Velocity, cm/sec	18.0
Inlet Gas	Steam

<u>Time</u> <u>Min.</u>	<u>Gas Volume</u> <u>Liters</u>	<u>O<sub>2</sub></u> <u>Vol %</u>	<u>N<sub>2</sub></u> <u>Vol %</u>	<u>CO</u> <u>Vol %</u>	<u>CO<sub>2</sub></u> <u>Vol %</u>	<u>H<sub>2</sub></u> <u>Vol %</u>	<u>CH<sub>4</sub></u> <u>Vol %</u>
0.0	0.0	0.0	100.000	0.0	0.0	0.0	0.0
3.270	0.420	0.0	95.114	0.0	0.0	4.886	0.0
9.640	1.100	0.0	96.735	0.0	0.0	3.265	0.0
15.860	1.770	0.0	98.448	0.0	0.0	1.552	0.0
22.070	2.430	0.0	99.198	0.0	0.0	0.802	0.0
28.290	3.070	0.0	99.339	0.0	0.0	0.661	0.0
34.500	3.730	0.0	99.450	0.0	0.0	0.550	0.0
40.580	4.360	0.0	99.560	0.0	0.0	0.440	0.0
46.950	5.010	0.0	99.541	0.0	0.0	0.459	0.0
53.170	5.730	0.0	99.610	0.0	0.0	0.390	0.0

TABLE D-21

Run 647

Minigasifier

Empty

Temperature, RV-°F 55.0  
Pressure, psig 36.0  
Superficial Velocity, cm/sec 18.0  
Inlet Gas Steam

<u>Time</u> <u>Min.</u>	<u>Gas Volume</u> <u>Liters</u>	<u>O<sub>2</sub></u> <u>Vol %</u>	<u>N<sub>2</sub></u> <u>Vol %</u>	<u>CO</u> <u>Vol %</u>	<u>CO<sub>2</sub></u> <u>Vol %</u>	<u>H<sub>2</sub></u> <u>Vol %</u>	<u>CH<sub>4</sub></u> <u>Vol %</u>
0.0	0.0	0.0	100.000	0.0	0.0	0.0	0.0
4.260	0.510	0.0	97.064	0.0	0.0	2.936	0.0
11.120	1.270	0.0	97.394	0.0	0.0	5.606	0.0
17.760	1.640	0.0	95.403	0.0	0.0	4.597	0.0
25.480	2.470	0.0	97.059	0.0	0.0	2.941	0.0
32.480	3.190	0.0	98.378	0.0	0.0	1.622	0.0
39.890	3.920	0.0	98.704	0.0	0.0	1.296	0.0
50.900	4.600	0.0	98.886	0.0	0.0	1.114	0.0
53.170	5.230	0.0	99.494	0.0	0.0	0.506	0.0
59.460	5.810	0.0	99.218	0.0	0.0	0.782	0.0
66.460	6.510	0.0	99.347	0.0	0.0	0.653	0.0
73.520	7.160	0.0	99.391	0.0	0.0	0.609	0.0



TABLE D-22

RUN 649

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 35.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.61  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (1) %CARBON/HF
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
4.4	1.6	3.71	0.03	0.00	0.10	2.69	23.8	0.8	74.7	0.7	55.7
11.4	11.9	5.17	0.08	0.02	0.14	2.63	29.7	6.0	63.2	1.1	133.0
18.2	28.3	4.85	0.08	0.02	0.13	2.65	34.2	9.1	55.2	1.4	189.8
25.0	42.6	4.53	0.08	0.02	0.12	2.68	35.5	8.9	54.2	1.4	220.5
31.7	56.9	3.84	0.07	0.02	0.10	2.70	35.9	8.8	53.9	1.4	261.2
38.5	68.0	3.10	0.06	0.01	0.08	2.74	36.0	9.1	53.5	1.4	283.1
45.3	77.3	2.46	0.04	0.01	0.07	2.77	35.6	9.4	53.7	1.3	315.8
52.0	84.1	1.68	0.03	0.01	0.05	2.81	35.4	9.8	53.6	1.2	321.3
58.8	88.8	1.12	0.02	0.01	0.03	2.87	34.8	10.1	54.0	1.1	290.0
65.8	91.5	0.58	0.01	0.00	0.02	2.88	34.3	10.9	53.9	0.9	229.6
73.1	93.5	0.51	0.01	0.00	0.01	2.85	33.3	10.9	55.2	0.7	189.3
80.2	94.5	0.32	0.00	0.00	0.01	2.89	28.4	8.3	63.0	0.3	114.9
87.3	95.0	0.37	0.00	0.00	0.01	2.90	20.1	5.0	74.9	0.0	60.2
94.3	95.2	0.34	0.00	0.00	0.01	2.88	12.5	2.2	85.3	0.0	46.8

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
4.4	23.8	0.8	74.7	0.7	20.9
11.4	28.6	5.1	65.2	1.0	62.4
18.2	31.5	7.2	60.1	1.2	93.3
25.0	32.7	7.7	58.3	1.3	102.3
31.7	33.5	8.0	57.3	1.3	107.5
38.5	33.9	8.1	56.7	1.3	105.9
45.3	34.1	8.3	56.4	1.3	102.5
52.0	34.2	8.4	56.1	1.3	97.0
58.8	34.2	8.5	56.0	1.3	90.6
65.8	34.2	8.5	56.0	1.3	83.4
73.1	34.2	8.6	56.0	1.3	76.7
80.2	34.1	8.6	56.1	1.3	70.7
87.3	34.0	8.6	56.2	1.2	65.3
94.3	33.8	8.5	56.4	1.2	60.6

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-23

RUN 650

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 54.00  
 SUPERFICIAL VELOCITY (CM/SEC) 22.11  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... IKG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (1) %CARBON/HR
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
3.4	2.9	3.79	0.05	0.00	0.15	3.79	26.8	1.4	70.8	1.1	93.9
10.0	17.3	3.93	0.10	0.02	0.15	3.91	31.9	5.8	60.9	1.3	187.7
16.6	37.0	3.38	0.09	0.02	0.13	3.99	35.5	8.8	54.1	1.5	252.1
23.2	52.2	2.64	0.07	0.02	0.10	4.06	36.0	9.1	53.3	1.5	270.1
29.9	65.7	2.12	0.06	0.02	0.08	4.08	36.3	9.4	52.8	1.5	311.9
37.0	76.7	1.54	0.04	0.01	0.06	4.13	36.4	9.8	52.5	1.3	340.2
43.8	84.1	0.96	0.03	0.01	0.04	4.21	36.4	10.4	52.0	1.2	327.8
50.4	88.4	0.64	0.02	0.01	0.03	4.23	36.6	11.2	51.1	1.1	300.5
57.5	92.0	0.53	0.01	0.00	0.02	4.21	34.9	10.9	53.5	0.7	302.9
64.1	93.9	0.39	0.01	0.00	0.02	4.21	30.4	9.2	59.8	0.6	291.1

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
3.4	26.8	1.4	70.8	1.1	51.4
10.0	30.8	4.9	63.1	1.3	103.6
16.6	33.1	6.7	58.8	1.4	133.8
23.2	33.9	7.4	57.3	1.4	135.0
29.9	34.3	7.8	56.5	1.4	131.7
37.0	34.6	8.0	56.0	1.4	124.4
43.8	34.7	8.2	55.6	1.4	115.2
50.4	34.8	8.4	55.4	1.4	105.2
57.5	34.8	8.5	55.4	1.4	96.1
64.1	34.7	8.5	55.5	1.3	88.0

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-24

RUN 651

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 35.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.61  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... IKG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE %CARBON
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
5.4	3.7	4.87	0.05	0.01	0.13	2.60	26.7	4.5	67.7	1.0	7
12.0	15.6	4.76	0.08	0.02	0.12	2.61	31.5	7.1	60.2	1.2	14
18.7	31.2	4.70	0.08	0.02	0.12	2.63	35.9	9.0	53.7	1.4	19
25.4	45.9	4.07	0.08	0.02	0.11	2.66	36.7	8.8	53.1	1.4	22
32.0	58.3	3.34	0.06	0.01	0.09	2.72	36.7	8.9	53.0	1.4	23
39.0	68.0	3.08	0.05	0.01	0.08	2.76	34.8	8.4	55.5	1.3	24
46.1	76.6	2.08	0.04	0.01	0.06	2.78	34.8	8.6	55.4	1.2	27
53.1	83.2	1.42	0.03	0.01	0.04	2.81	36.5	10.9	51.4	1.2	28
60.4	88.1	1.16	0.02	0.01	0.03	2.84	33.4	13.6	52.0	1.0	28
67.6	91.3	0.88	0.01	0.01	0.03	2.88	27.4	14.5	57.4	0.7	23
74.6	93.0	0.48	0.01	0.00	0.01	2.89	24.2	14.7	60.5	0.5	17
81.6	94.1	0.31	0.00	0.00	0.01	2.94	22.7	15.2	61.8	0.3	11
88.3	94.6	0.14	0.00	0.00	0.00	3.03	22.1	13.9	64.0	0.0	7

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
5.4	26.7	4.5	67.7	1.0	40.8
12.0	30.2	6.4	62.2	1.2	78.0
18.7	32.8	7.6	58.4	1.3	100.2
25.4	33.9	7.9	56.8	1.3	108.2
32.0	34.5	8.1	56.1	1.3	109.3
39.0	34.5	8.2	56.0	1.3	104.7
46.1	34.5	8.2	55.9	1.3	99.7
53.1	34.7	8.4	55.6	1.3	93.9
60.4	34.6	8.7	55.4	1.3	87.5
67.6	34.4	8.9	55.5	1.3	81.1
74.6	34.2	9.0	55.6	1.3	74.8
81.6	34.0	9.1	55.7	1.2	69.2
88.3	33.9	9.1	55.7	1.2	64.3

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-25

RUN 652

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 27.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.35  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS(DRY, N2-FREE), VOL %				RATE (1) %CARBON/HR
	CARBON	STEAM	CO	CO2	H2	H2O	CO	CO2	H2	CH4	
0.9	0.0	0.04	0.00	0.0	0.00	2.34	81.5	0.0	18.5	0.0	29.7
7.5	6.5	4.61	0.07	0.02	0.09	1.95	40.2	9.8	49.1	1.0	96.2
14.6	20.7	5.17	0.08	0.02	0.10	1.93	38.6	9.7	50.6	1.1	154.5
21.9	36.0	4.95	0.08	0.02	0.10	1.95	39.3	8.9	50.4	1.3	185.5
28.9	49.1	4.54	0.07	0.01	0.09	1.97	39.2	8.6	50.9	1.3	209.0
36.0	60.9	3.88	0.06	0.01	0.08	1.99	39.1	8.6	51.1	1.3	235.3
42.7	70.4	3.18	0.05	0.01	0.06	2.02	39.0	8.8	51.0	1.3	255.4
49.5	78.0	2.32	0.04	0.01	0.05	2.08	38.8	9.0	51.0	1.2	251.6
56.3	82.9	1.68	0.02	0.01	0.03	2.10	37.9	9.3	51.7	1.1	226.9
63.1	86.7	1.26	0.02	0.00	0.03	2.09	36.4	9.5	53.2	0.9	212.1
69.9	89.3	0.76	0.01	0.00	0.02	2.10	36.6	9.9	52.8	0.7	173.3
77.1	91.0	0.28	0.01	0.00	0.01	2.12	40.7	10.9	48.1	0.3	123.5
83.8	91.9	0.10	0.00	0.00	0.00	2.12	50.6	11.5	37.9	0.0	98.6

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY, N2-FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO2	H2	CH4	
0.9	81.5	0.0	18.5	0.0	0.0
7.5	40.2	9.8	49.1	1.0	51.9
14.6	39.1	9.7	50.1	1.1	85.4
21.9	39.2	9.4	50.3	1.2	98.5
28.9	39.2	9.2	50.4	1.2	102.0
36.0	39.2	9.1	50.5	1.2	101.5
42.7	39.1	9.0	50.6	1.2	98.8
49.5	39.1	9.0	50.6	1.2	94.4
56.3	39.0	9.1	50.7	1.2	88.4
63.1	38.9	9.1	50.8	1.2	82.5
69.9	38.8	9.1	50.9	1.2	76.7
77.1	38.9	9.1	50.8	1.2	70.8
83.8	39.0	9.1	50.7	1.2	65.8

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-26

RUN 655

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 26.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.98  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM-NITROGEN  
 COKE SAMPLE..... IKG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (1) %CARBON/HR
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
1.4	0.0	3.91	0.00	0.0	0.06	1.58	6.7	0.0	93.3	0.0	23.5
8.1	5.1	8.17	0.05	0.01	0.11	1.26	21.3	5.4	72.9	0.4	72.5
15.6	16.5	7.37	0.06	0.01	0.10	1.26	31.3	8.2	59.6	0.9	113.9
22.5	27.9	8.30	0.06	0.01	0.11	1.26	33.2	8.1	57.6	1.1	135.4
29.3	38.8	7.10	0.06	0.01	0.09	1.28	33.1	8.0	57.8	1.1	150.5
36.4	49.2	7.41	0.05	0.01	0.10	1.28	32.3	7.9	58.7	1.1	164.8
43.4	58.5	6.09	0.05	0.01	0.08	1.30	31.8	7.9	59.3	1.0	179.9
50.2	66.4	4.02	0.04	0.01	0.05	1.33	34.7	8.7	55.5	1.1	186.4
57.1	72.8	3.32	0.03	0.01	0.04	1.34	36.6	9.6	52.7	1.1	184.4
63.9	77.8	2.66	0.02	0.01	0.04	1.38	35.4	9.5	54.2	0.9	167.1
70.6	81.2	1.71	0.02	0.00	0.02	1.39	35.5	8.8	54.8	0.8	139.5
77.7	83.8	1.04	0.01	0.00	0.01	1.39	38.3	8.7	52.3	0.7	113.9
84.5	85.5	0.51	0.01	0.00	0.01	1.40	42.2	9.6	47.9	0.3	101.6

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
1.4	6.7	0.0	93.3	0.0	1.2
8.1	21.0	5.2	73.4	0.4	37.9
15.6	27.1	7.0	65.2	0.7	63.6
22.5	29.3	7.4	62.5	0.8	74.4
29.3	30.3	7.5	61.3	0.9	79.5
36.4	30.7	7.6	60.7	0.9	81.1
43.4	30.9	7.7	60.5	1.0	80.9
50.2	31.3	7.8	60.0	1.0	79.3
57.1	31.7	7.9	59.4	1.0	76.5
63.9	31.9	8.0	59.1	1.0	73.1
70.6	32.0	8.0	59.0	1.0	69.0
77.7	32.2	8.1	58.8	1.0	64.7
84.5	32.3	8.1	58.6	0.9	60.7

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-27

RUN 656

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -55.00  
 PRESSURE (PSIG) 27.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.17  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM-NITROGEN  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (1) %CARBON/HR
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
4.5	0.4	14.05	0.01	0.00	0.05	0.31	15.3	7.5	77.2	0.0	12.4
11.8	2.7	10.50	0.01	0.01	0.03	0.27	18.4	9.3	72.2	0.0	20.9
19.1	5.4	13.06	0.01	0.01	0.04	0.26	23.1	11.0	65.9	0.0	24.1
26.4	8.3	13.02	0.01	0.01	0.04	0.26	23.9	10.1	66.0	0.0	26.9
33.8	11.4	12.96	0.02	0.01	0.04	0.26	25.6	9.9	64.5	0.0	29.7
40.7	14.5	11.77	0.02	0.01	0.04	0.27	27.8	10.3	61.9	0.0	31.9
47.6	17.7	12.43	0.02	0.01	0.04	0.26	28.6	10.3	61.1	0.0	33.6
54.9	21.1	13.83	0.02	0.01	0.04	0.26	28.0	9.6	62.4	0.0	35.7
62.1	24.5	10.58	0.02	0.01	0.03	0.27	29.7	9.6	60.7	0.0	36.7
69.0	27.7	13.12	0.02	0.01	0.04	0.26	29.8	9.5	60.8	0.0	37.7
76.1	30.9	13.07	0.02	0.01	0.04	0.26	28.2	8.6	63.2	0.0	38.8
83.2	34.0	11.58	0.02	0.00	0.03	0.27	29.6	8.0	62.4	0.0	39.2
90.3	37.0	11.40	0.02	0.01	0.03	0.27	30.0	8.3	61.7	0.0	40.5
97.3	40.0	8.85	0.02	0.00	0.03	0.27	32.3	9.2	58.5	0.0	42.6
104.4	43.0	11.29	0.02	0.00	0.03	0.27	32.9	8.6	58.5	0.0	43.5
111.5	45.8	10.67	0.02	0.00	0.03	0.27	30.4	7.8	61.8	0.0	44.0
118.5	48.6	7.48	0.02	0.00	0.02	0.28	33.6	8.6	57.9	0.0	45.5
125.6	51.3	10.84	0.02	0.00	0.03	0.27	33.1	8.4	58.5	0.0	46.5
132.7	54.0	7.24	0.01	0.00	0.02	0.28	33.0	7.9	59.2	0.0	47.6
139.7	56.5	7.35	0.01	0.00	0.02	0.28	37.1	8.3	54.6	0.0	48.7
146.8	58.9	7.29	0.01	0.00	0.02	0.28	36.3	7.8	55.8	0.0	50.1

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
4.5	15.3	7.5	77.2	0.0	5.8
11.8	17.9	9.0	73.1	0.0	13.9
19.1	20.1	9.9	70.0	0.0	16.9
26.4	21.4	9.9	68.7	0.0	18.8
33.8	22.4	9.9	67.7	0.0	20.3
40.7	23.4	10.0	66.6	0.0	21.4
47.6	24.2	10.1	65.7	0.0	22.3
54.9	24.8	10.0	65.2	0.0	23.1
62.1	25.4	9.9	64.6	0.0	23.7
69.0	25.9	9.9	64.2	0.0	24.0
76.1	26.1	9.8	64.1	0.0	24.3
83.2	26.4	9.6	64.0	0.0	24.5
90.3	26.7	9.5	63.8	0.0	24.6
97.3	27.1	9.5	63.5	0.0	24.6
104.4	27.4	9.4	63.1	0.0	24.7
111.5	27.6	9.3	63.1	0.0	24.7
118.5	27.9	9.3	62.8	0.0	24.6
125.6	28.1	9.3	62.6	0.0	24.5
132.7	28.4	9.2	62.4	0.0	24.4
139.7	28.7	9.2	62.2	0.0	24.3
146.8	29.0	9.1	61.9	0.0	24.1

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-28

Run 657

Minigasifier

Empty

Temperature, RV-°F 55.0  
 Pressure, psig 35.0  
 Superficial Velocity, cm/sec 18.0  
 Inlet Gas Steam-Carbon Monoxide

<u>Time</u> <u>Min.</u>	<u>Gas Volume</u> <u>Liters</u>	<u>O<sub>2</sub></u> <u>Vol %</u>	<u>N<sub>2</sub></u> <u>Vol %</u>	<u>CO</u> <u>Vol %</u>	<u>CO<sub>2</sub></u> <u>Vol %</u>	<u>H<sub>2</sub></u> <u>Vol %</u>	<u>CH<sub>4</sub></u> <u>Vol %</u>
0.0	0.0	0.0	100.000	0.0	0.0	0.0	0.0
6.950	0.690	0.0	95.109	0.754	0.237	3.900	0.0
14.400	1.470	0.0	96.789	0.983	0.094	2.134	0.0
21.630	2.240	0.0	97.172	0.713	0.040	2.075	0.0
28.900	3.000	0.0	97.345	0.786	0.185	1.684	0.0
36.170	3.740	0.0	97.741	0.810	0.198	1.251	0.0
43.440	4.480	0.0	97.936	0.802	0.222	1.040	0.0
50.700	5.230	0.0	97.980	0.801	0.210	1.009	0.0
57.970	5.970	0.0	98.037	0.811	0.219	0.933	0.0
65.270	6.710	0.0	98.362	0.806	0.230	0.602	0.0
72.540	7.440	0.0	98.388	0.802	0.206	0.604	0.0
79.810	8.040	0.0	98.540	0.809	0.218	0.433	0.0
87.080	8.760	0.0	98.335	0.820	0.230	0.615	0.0
97.350	9.480	0.0	98.386	0.820	0.248	0.546	0.0
101.620	10.230	0.0	98.518	0.797	0.240	0.445	0.0
108.890	10.860	0.0	98.403	0.793	0.248	0.556	0.0
116.160	11.560	0.0	98.545	0.810	0.249	0.396	0.0
123.430	12.300	0.0	98.563	0.795	0.237	0.405	0.0
130.700	13.030	0.0	98.432	0.795	0.251	0.522	0.0
137.970	13.740	0.0	98.555	0.800	0.249	0.396	0.0

TABLE D-29

RUN 658

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -130.00  
 PRESSURE (PSIG) 35.50  
 SUPERFICIAL VELOCITY (CM/SEC) 19.35  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (1) %CARBON/
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
6.9	1.6	1.86	0.02	0.01	0.05	2.82	23.1	7.5	68.8	0.5	27.5
14.3	6.5	1.82	0.03	0.01	0.05	2.82	27.6	8.3	63.2	0.7	47.5
22.0	12.9	1.92	0.03	0.01	0.05	2.82	33.2	8.6	57.1	1.0	60.2
29.3	19.7	1.96	0.03	0.01	0.05	2.82	34.8	8.2	55.7	1.2	68.5
36.8	26.5	1.91	0.03	0.01	0.05	2.83	35.5	8.0	55.2	1.3	74.9
44.1	33.2	1.82	0.03	0.01	0.05	2.83	36.1	7.9	54.5	1.3	79.8
51.5	39.6	1.60	0.03	0.01	0.04	2.88	36.2	7.8	54.7	1.3	76.9
58.9	44.7	1.43	0.03	0.01	0.04	2.89	36.4	7.8	54.5	1.3	77.2
66.3	50.1	1.48	0.03	0.01	0.04	2.86	36.7	7.7	54.3	1.3	86.0
73.7	55.2	1.32	0.02	0.01	0.04	2.86	36.5	7.6	54.7	1.3	87.3
81.0	59.8	1.20	0.02	0.00	0.03	2.86	36.6	7.6	54.5	1.3	87.7
88.4	63.9	1.08	0.02	0.00	0.03	2.87	36.5	7.6	54.7	1.3	86.8
95.8	67.5	0.97	0.02	0.00	0.03	2.88	35.8	7.5	55.5	1.2	83.6
103.2	70.6	0.84	0.02	0.00	0.02	2.88	35.7	7.1	56.1	1.1	79.9
110.6	73.3	0.66	0.01	0.00	0.02	2.94	35.7	6.9	56.4	1.0	68.7
118.0	75.1	0.58	0.01	0.00	0.02	2.94	35.4	7.3	56.3	1.0	63.7
125.4	77.2	0.53	0.01	0.00	0.01	2.89	35.6	7.6	56.0	0.9	67.6
132.7	78.9	0.47	0.01	0.00	0.01	2.90	35.6	7.6	56.0	0.8	61.6
139.8	80.3	0.40	0.01	0.00	0.01	2.94	34.6	7.3	57.2	0.9	52.4
147.1	81.4	0.32	0.01	0.00	0.01	2.94	34.2	7.1	57.9	0.8	46.1
154.5	82.4	0.31	0.00	0.00	0.01	2.90	34.1	6.8	58.4	0.7	43.9
161.9	83.3	0.29	0.00	0.00	0.01	2.91	32.6	6.1	61.0	0.3	37.6
169.3	83.9	0.17	0.00	0.00	0.01	2.92	33.8	5.7	60.3	0.2	34.6

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
6.9	23.1	7.5	68.8	0.5	15.5
14.3	26.4	8.0	64.9	0.6	27.3
22.0	29.4	8.3	61.5	0.8	35.3
29.3	31.1	8.3	59.7	0.9	40.2
36.8	32.2	8.2	58.6	1.0	43.3
44.1	32.9	8.2	57.9	1.1	45.1
51.5	33.4	8.1	57.4	1.1	46.1
58.9	33.7	8.1	57.1	1.1	45.5
66.3	34.0	8.0	56.8	1.2	45.4
73.7	34.2	8.0	56.6	1.2	45.0
81.0	34.4	8.0	56.5	1.2	44.2
88.4	34.5	7.9	56.3	1.2	43.3
95.8	34.6	7.9	56.3	1.2	42.3
103.2	34.7	7.9	56.3	1.2	41.0
110.6	34.7	7.8	56.3	1.2	39.7
118.0	34.7	7.8	56.3	1.2	38.2
125.4	34.7	7.8	56.3	1.2	36.9
132.7	34.8	7.8	56.3	1.2	35.7
139.8	34.8	7.8	56.3	1.1	34.5
147.1	34.7	7.8	56.3	1.1	33.2
154.5	34.7	7.8	56.3	1.1	32.0
161.9	34.7	7.8	56.4	1.1	30.9
169.3	34.7	7.7	56.4	1.1	29.8

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON



TABLE D-30

RUN 659

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -5.00  
 PRESSURE (PSIG) 35.50  
 SUPERFICIAL VELOCITY (CM/SEC) 19.21  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... 1KG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS(DRY,N <sub>2</sub> -FREE),VOL %				RATE (1) %CARBON/HF
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
7.0	9.6	6.50	0.10	0.04	0.17	2.59	32.3	11.5	54.9	1.2	152.7
14.3	33.2	6.25	0.12	0.03	0.17	2.59	34.5	11.1	53.4	1.1	274.9
22.4	56.6	4.93	0.10	0.03	0.13	2.67	36.8	10.6	51.8	0.8	339.9
30.8	73.5	3.14	0.06	0.02	0.09	2.77	36.9	10.4	51.9	0.8	366.6
41.5	86.4	1.86	0.03	0.01	0.05	2.84	35.6	10.7	52.7	1.0	404.8
50.6	92.1	0.99	0.01	0.01	0.03	2.88	32.7	11.4	54.8	1.2	350.3
59.0	94.6	0.45	0.01	0.00	0.01	2.90	31.8	10.1	57.4	0.8	240.7
66.5	95.7	0.24	0.00	0.00	0.01	2.91	33.7	7.8	58.1	0.4	191.1

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS(DRY,N <sub>2</sub> -FREE),VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
7.0	32.3	11.5	54.9	1.2	82.1
14.3	33.8	11.2	53.8	1.1	139.1
22.4	35.0	11.0	53.0	1.0	151.4
30.8	35.4	10.8	52.8	1.0	143.4
41.5	35.5	10.8	52.7	1.0	125.0
50.6	35.3	10.9	52.9	1.0	109.3
59.0	35.2	10.8	53.0	1.0	96.1
66.5	35.2	10.8	53.1	1.0	86.2

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-31

Run 661

Minigasifier

Empty

Temperature, RV-°F 130.0  
 Pressure, psig 35.5  
 Superficial Velocity, cm/sec 18.0  
 Inlet Gas Steam

<u>Time</u> <u>Min.</u>	<u>Gas Volume</u> <u>Liters</u>	<u>O<sub>2</sub></u> <u>Vol %</u>	<u>N<sub>2</sub></u> <u>Vol %</u>	<u>CO</u> <u>Vol %</u>	<u>CO<sub>2</sub></u> <u>Vol %</u>	<u>H<sub>2</sub></u> <u>Vol %</u>	<u>CH<sub>4</sub></u> <u>Vol %</u>
0.0	0.0	0.0	100.000	0.0	0.0	0.0	0.0
1.060	0.145	0.0	99.971	0.0	0.0	0.029	0.0
8.070	0.945	0.0	95.163	0.0	0.0	4.837	0.0
15.130	1.695	0.0	98.272	0.0	0.0	1.728	0.0
22.150	2.435	0.0	99.124	0.0	0.0	0.876	0.0
29.160	3.175	0.0	99.374	0.0	0.0	0.626	0.0
36.170	3.925	0.0	99.382	0.0	0.0	0.618	0.0
43.180	4.665	0.0	99.515	0.0	0.0	0.485	0.0
50.190	5.475	0.0	99.609	0.0	0.0	0.391	0.0
57.200	5.835	0.0	99.629	0.0	0.0	0.371	0.0
64.210	6.565	0.0	99.699	0.0	0.0	0.301	0.0
71.220	7.285	0.0	99.955	0.0	0.0	0.045	0.0
78.230	8.035	0.0	99.937	0.0	0.0	0.063	0.0
85.240	8.765	0.0	99.827	0.0	0.0	0.173	0.0
92.250	9.495	0.0	99.985	0.0	0.0	0.015	0.0
99.260	10.225	0.0	99.977	0.0	0.0	0.023	0.0
106.270	10.965	0.0	100.000	0.0	0.0	0.0	0.0

TABLE D-32

RUN 662

## GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F - RV) -130.00  
 PRESSURE (PSIG) 35.00  
 SUPERFICIAL VELOCITY (CM/SEC) 19.54  
 COKE CHARGE (GM) 1.00  
 % CARBON IN COKE 48.70  
 % ASH IN COKE 46.20  
 INLET GAS..... STEAM  
 COKE SAMPLE..... IKG ILLINOIS COKE (50/70 MESH)

## I. INSTANTANEOUS DATA

TIME MIN.	CONVERSION, %		PARTIAL PRESSURE, ATM				GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (1) %CARBON/HR
	CARBON	STEAM	CO	CO <sub>2</sub>	H <sub>2</sub>	H <sub>2</sub> O	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
1.2	0.0	0.0	0.00	0.0	0.0	2.85	100.0	0.0	0.0	0.0	7.2
8.3	1.7	1.93	0.02	0.0	0.05	2.79	28.5	0.0	70.7	0.8	27.3
15.5	6.5	1.96	0.03	0.01	0.05	2.77	31.9	3.2	63.9	1.0	51.1
22.9	13.4	2.05	0.04	0.01	0.06	2.77	35.4	7.2	56.2	1.2	67.1
30.3	20.8	2.08	0.04	0.01	0.06	2.78	35.8	8.3	54.6	1.3	76.3
37.7	28.3	1.95	0.04	0.01	0.05	2.78	36.2	8.2	54.2	1.3	82.0
45.1	35.3	1.82	0.03	0.01	0.05	2.79	36.7	8.3	53.7	1.3	86.1
52.7	42.2	1.60	0.03	0.01	0.04	2.84	36.7	8.3	53.7	1.3	84.5
60.1	47.5	1.47	0.03	0.01	0.04	2.85	37.2	8.3	53.3	1.3	86.3
67.9	53.7	1.43	0.03	0.01	0.04	2.81	37.1	8.4	53.2	1.3	96.0
75.3	58.8	1.25	0.02	0.01	0.03	2.82	37.0	8.5	53.2	1.3	95.6
82.7	63.4	1.11	0.02	0.00	0.03	2.83	37.3	8.6	52.9	1.3	95.2
90.2	67.5	0.99	0.02	0.00	0.03	2.84	36.9	8.7	53.0	1.3	95.0
97.6	71.1	0.89	0.02	0.00	0.02	2.84	36.6	8.9	53.3	1.2	94.1
105.0	74.2	0.68	0.01	0.00	0.02	2.89	36.6	8.9	53.3	1.2	83.3
112.4	76.3	0.58	0.01	0.00	0.02	2.90	37.0	8.8	53.2	1.0	76.5
119.8	78.6	0.54	0.01	0.00	0.01	2.86	37.0	8.9	53.2	0.9	80.1
127.3	80.6	0.47	0.01	0.00	0.01	2.86	36.4	9.2	53.5	1.0	73.8
134.9	82.2	0.32	0.01	0.00	0.01	2.89	36.9	9.6	52.7	0.8	64.3
142.3	83.4	0.27	0.01	0.00	0.01	2.90	38.6	10.1	50.7	0.6	57.1
149.8	84.6	0.24	0.00	0.00	0.01	2.87	38.0	10.3	51.0	0.7	52.7
157.2	85.5	0.15	0.00	0.00	0.00	2.88	39.3	10.0	50.2	0.4	43.2
164.7	86.2	0.13	0.00	0.00	0.00	2.89	41.8	9.9	48.3	0.0	39.1

## II. AVERAGE DATA

TIME MIN.	GAS ANALYSIS (DRY, N <sub>2</sub> -FREE), VOL %				RATE (2) %CARBON/HR
	CO	CO <sub>2</sub>	H <sub>2</sub>	CH <sub>4</sub>	
1.2	100.0	0.0	0.0	0.0	0.1
8.3	28.5	0.0	70.7	0.8	12.3
15.5	30.9	2.2	66.0	1.0	25.0
22.9	32.9	4.5	61.5	1.1	35.0
30.3	33.8	5.7	59.3	1.1	41.1
37.7	34.4	6.3	58.1	1.2	45.0
45.1	34.8	6.7	57.3	1.2	47.0
52.7	35.1	6.9	56.7	1.2	48.0
60.1	35.3	7.1	56.4	1.2	47.4
67.9	35.5	7.2	56.0	1.2	47.4
75.3	35.6	7.3	55.8	1.2	46.9
82.7	35.8	7.4	55.6	1.2	46.0
90.2	35.8	7.5	55.5	1.2	44.9
97.6	35.9	7.6	55.3	1.2	43.7
105.0	35.9	7.6	55.3	1.2	42.4
112.4	35.9	7.6	55.2	1.2	40.8
119.8	35.9	7.7	55.2	1.2	39.4
127.3	36.0	7.7	55.1	1.2	38.0
134.9	36.0	7.7	55.1	1.2	36.6
142.3	36.0	7.8	55.0	1.2	35.2
149.8	36.0	7.8	55.0	1.2	33.9
157.2	36.1	7.8	54.9	1.2	32.6
164.7	36.1	7.8	54.9	1.2	31.4

## NOTES:

- (1) BASED ON REMAINING CARBON  
 (2) BASED ON INITIAL CARBON

TABLE D-33

MINIGASIFIER  
INITIAL CO<sub>2</sub> GASIFICATION RATE DATA

IKG Illinois Gasifier Coke

Run	Run Conditions				Initial Response		
	Temp., RV-°F	Press., atm	Superficial Velocity, cm/sec	Coke Mesh Size, U.S. Sieve	Rate %C/hr	Partial Pressures* (Bed Avg.), atm	
						CO <sub>2</sub>	CO
618	55	4.9	18.7	50/70	84	4.8	0.09
620	55	2.8	18.9	50/70	57	2.1	0.06
626	55	2.9	18.8	50/70	63	2.8	0.06
627	55	2.9	19.2	50/70	50	1.3	0.05
628	50	2.9	18.9	50/70	59	2.8	0.05
629	130	2.9	18.8	50/70	19	2.9	0.02
630	5	2.9	18.8	50/70	100	2.8	0.10
631	130	2.9	18.9	50/70	20	2.9	0.02
633	55	2.9	19.1	50/70	23	0.38	0.02
634	55	2.9	1.0	50/70	19	2.5	0.36
636	55	3.0	0.5	50/70	14	2.4	0.54
637	55	2.9	18.9	100/150	87	2.8	0.08
638	55	2.9	19.2	100/150	84	2.8	0.07
640	55	3.0	1.1	100/150	23	2.6	0.44
641	55	2.9	18.8	40/50	44	2.9	0.05

\*N<sub>2</sub> diluent in some runs.

TABLE D-34

MINIGASIFIER  
INITIAL H<sub>2</sub>O GASIFICATION RATE DATA  
IKG Illinois Gasifier Coke

Run Conditions					Initial Response					
Run	Temp., RV-°F	Press., atm	Superficial Velocity, cm/sec	Coke Mesh Size U.S. Sieve	Rate %C/hr	Partial Pressures,* (Bed Avg.), atm				
						H2O	H2	CO	CO2	CH4
649	55	3.4	19.6	50/70	136	2.8	0.07	0.04	0.01	0.002
650	55	4.7	22.1	50/70	180	4.0	0.08	0.05	0.01	0.002
651	55	3.4	19.6	50/70	135	2.8	0.06	0.04	0.01	0.002
652	55	2.8	19.4	50/70	121	2.0	0.05	0.04	0.01	0.001
653	55	2.8	20.0	50/70	102	1.3	0.05	0.03	0.007	0.001
656	55	2.8	19.2	50/70	26.5	0.27	0.02	0.009	0.003	0.0
658	130	3.4	19.4	50/70	54	2.9	0.03	0.02	0.004	0.0006
659	5	3.4	19.2	50/70	194	2.7	0.09	0.06	0.02	0.002
662	130	3.4	19.5	50/70	58	2.8	0.03	0.02	0.004	0.0008

\*N<sub>2</sub> diluent in all runs.

## LIST OF ABBREVIATIONS

APS	Atmospheric Pipe Still
ASTM	American Society for Testing and Materials
BARD	Baytown Research and Development Division (Baytown, TX)
B/CD	Barrels Per Calendar Day
B/SD	Barrels Per Stream Day
BOD <sub>5</sub>	5-Day Biological Oxygen Demand
CLPP	Coal Liquefaction Pilot Plant
Co/Mo	Cobalt/Molybdenum
CSCU	Continuous Stirred Coking Unit
DAF	Dissolved Air Flotation
DBM	Design Basis Memorandum
DCF	Discounted Cash Flow
DEA	Diethanolamine
DMMF	Dry Mineral Matter-Free
DOE	Department of Energy
ECLP	Exxon Coal Liquefaction Pilot Plant
EDS	Exxon Donor Solvent
EDS-FPU	Exxon Donor Solvent - FLEXICOKING Prototype Unit
EEPD	Exxon Engineering Petroleum Department (Florham Pk., N.J.)
ELED	EDS Liquefaction Engineering Division (Florham Pk., N.J.)
EOR	End-of-Run
EPA	Environmental Protection Agency
EPRI	Electric Power Research Institute
EPRL	EDS Process Research Laboratories (Baytown, TX)
ER&E	Exxon Research and Engineering Company
FBU	Fluidized Bed Unit
GC	Gas Chromatography
GPM	Gallons Per Minute
HBG	High Btu Gas
HCO	Hydrogenated Creosote Oil
IBG	Intermediate Btu Gas
IKG	Integrated Coking/Gasification Pilot Plant

k	Thousand
LBG	Low Btu Gas
LHV	Lower Heating Value
LP	Linear Program
LPG	Liquefied Petroleum Gas
LSCU	Large Stirred Coker Unit
LSFO	Low Sulfur Fuel Oil
M	Million
MBG	Moving-Bed Coal Gasification
MPSS	Multi-Pass Spent Solvent
NDT	Non-Destructive Testing
Ni/Mo	Nickel Molybdenum
OTCLU	Once-Through Coal Liquefaction Unit
PAM	Process Alternatives LP Model
PDA	Process Development Allowance
P&ID	Piping and Instrument Diagram
PNA	Polynuclear Aromatics
PRD	Product Research Division (Linden, N.J.)
RCLU-1	50 Pounds-per-Day Recycle Coal Liquefaction Unit
RCLU-2	100 Pounds-per-Day Recycle Coal Liquefaction Unit
RCO	Raw Creosote Oil
RISP	Required Initial Selling Price
RV	Reference Value. Under terms of this Agreement, all non-program data are coded in terms of Reference Values to prevent disclosure.
SCF	Standard Cubic Feet
SDU	Study Design Update
SEM	Scanning Electron Microscope
SFU	Small Fluidized Bed Unit (for coke gasification)
SOR	Start-of-Run
SQI	Solvent Quality Index
TDS	Total Dissolved Solids

T/D	Tons Per Day
TEC	Total Erected Cost
TIR	Temperature Increase Required
TOC	Total Organic Carbon
T/SD	Tons Per Stream Day
VGO	Vacuum Gas Oil
VLE	Vapor Liquid Equilibrium
VPS	Vacuum Pipe Still
V/Hr/V	Volumetric Hourly Space Velocity, $\text{ft}^3$ feed per hour per $\text{ft}^3$ of reactor volume.
WWT	Wastewater Treatment
YP	Yield Period
YT	Yield Test

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