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EDS COAL LIQUEFACTION PROCESS DEVELOPMENT,
PHASES IIIB/IV

Annual Technical Progress Report, July 1, 1977-June 30, 1978

September 1978
Date Submitted

Work Performed Under Contract No. EF-77-A-01-2893

Exxon Research and Engineering Company
Florham Park, New Jersey

MASTER

U. S. DEPARTMENT OF ENERGY

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EDS COAL LIQUEFACTION PROCESS DEVELOPMENT

PHASES IIIB/IV

Annual Technical Progress Report
for the Period

July 1, 1977 - June 30, 1978

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Submitted - September, 1978

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PREPARED FOR
UNITED STATES DEPARTMENT OF ENERGY
UNDER COOPERATIVE AGREEMENT NO. EF-77-A-01-2893

A Project Jointly Sponsored By
United States Department of Energy
The Carter Oil Company
Electric Power Research Institute
Japan Coal Liquefaction Development Company
Phillips Petroleum Company
Atlantic Richfield Company

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EDS COAL LIQUEFACTION PROCESS DEVELOPMENT - PHASES IIIB/IV
ANNUAL TECHNICAL PROGRESS REPORT FOR THE PERIOD
JULY 1, 1977 - JUNE 30, 1978

ABSTRACT

This report is the first 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 - Phases IIIB/IV. This report covers the period July 1, 1977 - June 30, 1978. 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, and Atlantic Richfield Company. 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 Pilot Plant 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

1. OPERATION OF RECYCLE COAL LIQUEFACTION UNITS (RCLU)

- A process variable study with Wyoming subbituminous coal in the 50 pound-per-day Recycle Coal Liquefaction Unit (RCLU-1) showed that Wyoming coal liquid yield at 840°F increases continually with increasing residence time. This is in contrast to liquid yield from Illinois No. 6 bituminous coal, which exhibits a maximum within the same broad range of residence time.
- Liquid yield data and residual bottoms viscosity considerations strongly suggest that the optimum residence time for liquefying Wyoming coal at 840°F is between 40 and 100 minutes. Within this range of residence time, the liquid yield is nearly constant, and the residual bottoms viscosity is sufficiently low to permit easy bottoms handling.

- The effect of increased liquefaction temperature on Wyoming coal yields was investigated in RCLU-1. The maximum liquid yield at 880°F liquefaction temperature is essentially equal to the maximum liquid yield at 840°F. However, it can be obtained with at least a two-fold reduction in the liquefaction residence time over that required at 840°F.
- A solvent quality study with Wyoming subbituminous coal in RCLU-1 showed that Wyoming coal conversion and liquid yield are strongly dependent on Solvent Quality Index (SQI). Coal conversion and liquid yield are essentially independent of solvent quality above about four SQI. As SQI decreases below four, both conversion and liquid yield decrease dramatically. This response with Wyoming coal is generally similar to that found earlier with Illinois No. 6 coal.
- The effect of molecular hydrogen treat rate in liquefaction of Wyoming coal was investigated using both Recycle Coal Liquefaction Units (RCLU-1 and RCLU-2). Changing the hydrogen treat rate by ± 50 percent from four weight percent to two and six weight percent on dry coal did not significantly affect the conversion or yields.
- Pretreatment of Wyoming coal with sulfur dioxide was not successful in preventing calcium carbonate scale formation in the 100 pound-per-day Recycle Coal Liquefaction Unit (RCLU-2) runs. Reactor scale formation was reduced substantially but not eliminated, in only one out of four runs. Yields are similar to those from untreated Wyoming coal, except for slightly higher hydrogen sulfide and water make due to the pretreatment step. Work is underway to better understand the pretreatment chemistry. Ultimately, another RCLU reconfirmation run will be scheduled.
- A Ni/Mo-10 hydrogenation catalyst study was conducted in the RCLU-1. Results showed that the liquefaction yield structure and solvent quality with Ni/Mo-10 catalyst were comparable to those obtained using the Co/Mo-10 catalyst.
- A screening study with Pittsburgh No. 8 bituminous coal from the Ireland mine was conducted in the RCLU-1. For the liquefaction conditions studied (800-840°F/40-100 minutes slurry residence times), conversion and C₄-1000°F liquid yield for this bituminous coal are somewhat lower than for Illinois No. 6 bituminous coal at comparable conditions. Operability with this Pittsburgh coal was very smooth and similar to that with Illinois No. 6 coal.

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

- Successful operation of the Coal Liquefaction Pilot Plant (CLPP) was achieved during the third quarter of 1977 and second quarter of

1978. The CLPP had a scheduled downtime during the remaining six months. Twenty-six yield periods were completed during the year, 16 on Illinois No. 6 coal, and 10 on Wyodak subbituminous coal.

- No significant differences were found between RCLU and CLPP liquefaction yields in the Wyoming coal operations undertaken during the third quarter of 1977. Data workup is continuing on the more recent Wyoming coal runs at extended residence times.
- A series of ten yield periods was completed with Illinois #6 coal to confirm the yield structure at the conditions specified for the 1978 updated commercial study design (840°F and 40 minutes residence time).
- During the CLPP turnaround, modifications were made to the vacuum flash section, liquefaction reactors, and coal feed system to improve operability with Wyoming subbituminous coals.
- During the year, various engineering studies were undertaken, including evaluation of a twin-screw bottoms pump, a high severity vacuum flash study, investigation of a high initial boiling point (IBP) solvent, on-line measurement of hot slurry viscosities, a slurry preheater coking study, and a liquefaction reactor pressure cycling test.

3. SOLVENT HYDROGENATION STUDIES

- A long term catalyst activity maintenance study (311 days-on-oil) at conditions more severe than the commercial study design was completed. End-of-run data will be used to project catalyst performance at study design conditions, but preliminary indications are that a year's life can be met.
- Carbon oxide impurities in solvent hydrogenation treat gas do not affect hydrogenation activity, confirming previous results. The major effect is a slight reduction in desulfurization activity.
- The effects of CLPP spent solvent produced from Wyoming coal on Ni/Mo-10 and Co/Mo-20 initial catalyst activity were investigated. The preliminary data show that Wyoming spent solvent behaved differently than Illinois MPSS under similar solvent hydrogenation operating conditions.
- Results from the heavy solvent studies showed that higher start-of-run temperatures were required to reach the same levels of solvent donor hydrogen as those required with MPSS.

4. BENCH UNITS, MODELING AND CORRELATIONS STUDIES

- Three metal salts (ferrous sulfate, ferric sulfate and sodium carbonate) were identified as effective ion-exchange agents in preventing calcium carbonate formation with Wyoming coal. Sodium sulfate was only partially effective in ion-exchanging with Wyoming coal.
- Three reactor additives - colloidal silica, colloidal calcium carbonate and anthracene disulfonic acid - were unsuccessful in preventing calcium carbonate wall scale deposition with Wyoming coal.
- The kinetics of sulfuric acid pretreatment of Wyoming coal to prevent CaCO_3 formation during liquefaction was determined. The findings are expressed in terms of sulfur concentration remaining in the treat solution as a function of the square root of treat time divided by mean particle size.
- Bench scale studies showed that SO_2 gas is an effective agent in pretreatment of moist Wyoming coal to prevent CaCO_3 formation during liquefaction. This was tested in tubing autoclaves and the Once-Through-Coal-Liquefaction-Unit (OTCLU). A bench scale fluidized pretreatment unit was constructed to test SO_2 treatment in a fluidized bed.
- A fundamental kinetic model describing coal liquefaction is being developed. The data base used covers liquefaction of Illinois coal in RCLU and CLPP units at temperatures of 800, 840 and 880°F and space times ranging from 15 to 60 minutes. Preliminary estimates of the model parameters were obtained.
- An empirical model for liquefaction of Wyodak subbituminous coal was completed during the fourth quarter of 1977. The model is based on data from the OTCLU and RCLU pilot units, and correlates yields as functions of temperature and residence time. A computer module containing the model was developed and inserted into the EDS Process Model. The Process Model was used to generate a grid of 18 Wyodak yield vectors as input to the SFED Process Alternatives LP Model (PAM).
- The determination of reaction rate parameters for the fundamental solvent hydrogenation model with Ni/Mo-10 catalyst was completed. The data base utilized covers a wide range of solvent hydrogenation temperatures and space times.
- Simulations were undertaken to define conditions at which the solvent tends to equilibrate chemically. Results showed that chemical equilibrium would not be a limiting factor in hydrotreater operations for either Co/Mo-10 or Ni/Mo-10 catalyst.

- The improved fundamental solvent hydrogenation model was used in conjunction with the EDS Process Model to study the effects of EDS process conditions on the donatable hydrogen and saturates content of the recycle solvent during integrated steady state operation. It was determined that the effect of saturates buildup on solvent donatable hydrogen during recycle operation is not detrimental until a relatively high temperature is reached.
- A model for Ni/Mo-10 catalyst deactivation during solvent hydrogenation was chosen which assumes that each hydrogenation reaction is affected in the same way by catalyst deactivation. The model provides good fit of data from two catalyst activity maintenance studies.

5. PRODUCT QUALITY STUDIES

- Product quality studies to date concentrated on the potential disposition routes for products from the 250 ton-per-day Exxon Coal Liquefaction Pilot Plant (ECLP) and the characterization of EDS coal liquid products for direct end uses. Preliminary conclusions from these studies are summarized below.
 - + The preferred disposition routes for ECLP products in excess of Sponsors' needs are:
 - Naphtha and solvent to be blended as refinery fuel in a dedicated furnace
 - Vacuum gas oil to be blended in a special, high aromatics refinery stream
 - + Conventional, single-stage petroleum hydrofining technology can be used to upgrade EDS coal naphtha to high quality gasoline reformer feedstock.
 - + Combustion tests with 400°F⁺ EDS fuel oil in a 50 HP Cleaver-Brooks boiler indicate that smoke and particulate emissions are lower than those obtained with a typical regular or low sulfur fuel oil. NO_x emissions are consistent with predictions based on the nitrogen content.
- Future product quality studies will concentrate on end-use operability testing of EDS liquids. Tests will be conducted with raw and hydrotreated products to determine what upgrading steps are necessary to render these fuels operable in existing equipment.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT
BOTTOMS PROCESSING RESEARCH

6. COKING AND GASIFICATION STUDIES

- The bench-scale coking unit was used to perform screening studies on various EDS coal liquefaction bottoms. The yield data were used to select feedstocks for more definitive evaluation in the Continuous Stirred Coking Unit (CSCU).
- A bench coking study was conducted to determine the yields when mixtures of liquefaction bottoms and coal were coked. The yields from mixtures were similar, for most conditions studied, to the additive yields from separate coking of coal and bottoms.
- Viscosity experiments demonstrated that addition of coal to liquefaction bottoms increases the viscosity so that a maximum of 30% coal can be added before the viscosity becomes excessive. Thermal stability studies indicate that coal/bottoms mixtures exhibit poorer thermal stability than bottoms alone.
- A program was conducted to determine the effects of fluxing oils on the viscosity and storage stability of coal liquefaction bottoms. Small amounts (5%) of fluxing oil dramatically reduced bottoms viscosities and improved storage stability. However, for Wyodak bottoms, at least 20% oil is necessary to keep viscosity increases below 10%/day if the bottoms are stored at 500°F storage temperature. A computer program was developed which satisfactorily predicts blend viscosities.
- A yield study on CSCU confirmed an increase in coker liquid yield for Illinois bottoms produced at 40 minutes residence time over that produced at 25 minutes residence time at 100°F above the base coking temperature.
- The data base for coking of Wyodak coal liquefaction bottoms was expanded to include coking yields from bottoms produced at liquefaction residence times of 40, 60 and 100 minutes. The maximum coker liquid yield was obtained at 100°F above base coking temperature for 100-minute bottoms.
- Initial CSCU yields with Pittsburgh No. 8 (Ireland) coal liquefaction bottoms produced with 100 minutes liquefaction residence time are similar to yields obtained from 40-minute Illinois bottoms.
- CSCU coking yields with Illinois bottoms showed good agreement with earlier CSCU statistical data. The feed bottoms properties that best correlated the CSCU coking yields were 1000°F⁻ content of the feed and the percent 1000°F⁺ liquefaction yield. The coking process variable which consistently showed a significant correlation with coking yield was the steam/feed ratio.

- The Large Stirred Coking Unit (LSCU) produced sufficient coker liquid samples for product quality studies from both Illinois and Wyodak EDS coal liquefaction bottoms.
- Bench scale coke gasification studies of the kinetics of CO₂ and steam gasification indicate that the EDS bottoms cokes from both Illinois and Wyodak coals have higher reactivities than petroleum coke.
- Bench scale gasification studies of cokes produced in the Integrated Coking/Gasification pilot plant (IKG) showed greater reactivity for Wyodak than for Illinois coke. This was attributed to the higher concentration of calcium in the coal mineral matter of Wyodak coal.
- Scanning Electron Microscope (SEM) examination of the surfaces of bench gasified cokes suggests that some melting or fusion of the surface ash particles occurred. SEM examinations will continue in an effort to relate them to gasification kinetics, slag formation and fines generation.
- Bench scale studies of ash fusion of the mineral matter in EDS cokes revealed that agglomerate formation was dependent on superficial velocity, temperature, air/steam ratio and type of gas distributor in the Small Fluidized Bed Unit (SFU).
- Analyses of ash agglomerates removed from the IKG during Illinois bottoms runs were similar to those of Illinois coal ash.
- Coke gasification studies indicate that coke made from EDS Illinois bottoms maintains sufficient particle integrity to allow gasification in the FLEXICOKING* process. Fines makes were several-fold higher than petroleum, but the carbon content of the fines was so low that total carbon loss in the fines was low.
- Tests showed that at least half of the coke fines produced in the FBU were the result of attrition in the overhead cyclone. Thus, the FBU fines-make test measures not only the fines produced by particle size reduction due to gasification and bed attrition, but also fines produced from entrained coke by attrition in the overhead system.
- Initial FBU fines-make tests with IKG Wyodak coke indicated similar results to IKG Illinois coke, however, only two runs were completed.
- A series of runs was made in the FBU to investigate slag formation with IKG Illinois coke. Virtually complete carbon gasification

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was achieved without slag formation at nominal conditions at least as severe as the IKG nominal conditions during periods which formed slag. These results are under study.

7. CONSTRUCTION AND OPERATION OF THE 2 B/D COKING/GASIFICATION PILOT PLANT (IKG)

- The IKG was operated six months this year. These operations simulated the air FLEXICOKING process on Illinois No. 6 and Wyoming subbituminous (Wyodak) coal liquefaction vacuum bottoms. The first three and one-half months were on Illinois bottoms with the remaining time spent on Wyodak bottoms.
- Coke formation followed previous IKG petroleum experience. Pilot unit operability as related to coke particle size was satisfactory, suggesting no commercial problems in fluidizing the EDS cokes. However, extensive reactor coke agglomeration was encountered.
- Gasifier slag deposits were found after some operations. The larger deposits were found after the higher temperature run. The two lowest temperature runs on Wyodak bottoms produced no detectable deposits. Ongoing bench studies are addressing solutions to the slagging problem.
- An existing ER&E proprietary coke gasification model was successfully applied to the Illinois kinetics data. Studies in progress also indicate this model can be applied to the Wyodak data. However, there are model shortcomings which ongoing bench and computer studies are addressing.
- IKG Illinois coke particle integrity data from the areas of fines production, intensive particle properties, and particle size distributions do not suggest limitations to the application of the FLEXICOKING process to Illinois bottoms. Wyodak data still under study signal particle integrity as a possible problem area in FLEXICOKING of Wyodak bottoms.

ENGINEERING RESEARCH AND DEVELOPMENT PROCESS ENGINEERING AND COST EVALUATIONS

1. COMMERCIAL STUDY DESIGN AND COST ESTIMATE

- The onsite bases were finalized for the EDS Commercial Plant Study Design Update for Illinois coal. Coal feed rate will be 30 kT/SD with base liquefaction conditions of 840°F reactor temperature and 40 minutes residence time. Three processing configurations will be

investigated including a base case to maximize liquids, a market flexibility sensitivity case, and a technology alternative sensitivity case.

- Work is in progress on design of the liquefaction and solvent hydrogenation sections for the EDS Commercial Plant Study Design Update. A major portion of the effort involved preparation of up-to-date heat and material balances which reflect latest laboratory yield data and the results of a product quality study which identified means of improving the sulfur level of the overall plant fuel oil product.
- FLEXICOKING and hydrogen generation Study Design Update bases for the Illinois Base Case were modified to reflect more detailed information on plant fuel needs, coker scrubber liquids viscosity, and turnaround/unscheduled downtime requirements. The design capacity of each of the three once-through FLEXICOKING units was increased about 10% from 18.7 kB/SD to 20.4 kB/SD. This provides about 20% excess capacity for running off vacuum bottoms stored when one FLEXICOKING unit is down and all liquefaction trains are operating.

The average underflow recycle rate was set at 35% for hydroclones used to remove solids from coker scrubber liquids. This underflow rate satisfies the need to provide low BTU gas to offsite boilers at all times, while meeting hydroclone performance standards set by scrubber liquids viscosity. The steam reforming feed basis was changed to $C_1 - C_3$ from the purge gas feed originally contemplated because of lower fuel consumption with $C_1 - C_3$ feed. This in turn requires a change in the cryogenic separation system to recover a 95% hydrogen stream from the liquefaction purge gas.

2. COST REDUCTION AND LABORATORY GUIDANCE STUDIES

- Validation of the EDS Process Alternatives LP Model (PAM) for Wyoming coal was completed. The 1976 EDS Wyoming Study Design was represented by changing existing Illinois coal vectors and adding new vectors specific to Wyoming coal. The results of the LP simulation were compared to the Wyoming Study Design and found to be in close agreement.
- Initial results on the EDS Wyoming coal process variables study indicated that with a plant configuration utilizing a once-through FLEXICOKING unit for fuel production, partial oxidation for hydrogen production, and a coal-fired boiler, preferred liquefaction operating conditions are 840°F at 40-60 minutes residence time. Further work on bottoms viscosity and alternate configurations is proceeding.

- On February 27, 1978, the Texas Air Control Board (TACB) issued a Construction Permit for erection of the ECLP facilities. Changes to the design that were required to meet the TACB emissions standards were completed.
- Facilities were specified for preparing 2000 Bbl samples of naphtha and fuel oil products from ECLP. These samples will be transferred to the Project sponsors for their use.
- Larger, heavy particles (e.g., particles of high ash content) may build up in the ECLP liquefaction reactors because they are too heavy to be carried overhead from the upflow reactors. Facilities were designed for withdrawing these solids from the ECLP reactors.
- New facilities were specified to provide flushing oil for ECLP startup, shutdown and equipment maintenance. A blowdown system to receive the slop materials generated by these same operations was also specified.
- Three alternatives for recycling hot treat gas between the liquefaction and solvent hydrogenation units in a commercial coal liquefaction plant were compared to the base case. None of the three alternatives were sufficiently attractive to justify the operational risks and unwarranted complexity associated with the integration of the units.
- An investigation was completed to identify the areas of water usage for a commercial liquefaction plant and to determine ways to minimize fresh water requirements and the amount of sour, contaminated water sent to treating facilities. A major result of the investigation was the development of a possible water-shedding sequence which might be necessary should water not be available or should restrictions be placed on the quantity of plant effluent water.
- A study was conducted to evaluate gas treating and sulfur recovery schemes suitable for commercial coal liquefaction plants processing low sulfur Wyoming coal. The study concluded that acceptable cleanup can be achieved by using split flow DEA and Claus plants.
- A study was completed which predicts the organic and inorganic pollutant levels in the sour water streams generated from a commercial EDS Illinois coal liquefaction plant. The predictions will allow better definition of the wastewater treating facilities needed in a commercial Plant.

3. ENGINEERING STUDIES OF BOTTOMS PROCESSING/HYDROGEN MANUFACTURE

- An evaluation of hydrogen production methods for the EDS liquefaction plant showed that partial oxidation of vacuum pipestill

bottoms produced the lowest cost hydrogen when high BTU gas is valued in parity with liquids on a BTU basis. A reduction in the value of this gas by one-third makes steam reforming of high BTU gas the most attractive hydrogen production alternative.

- The maximum feasible vacuum pipestill cut point was determined to be 975°F for EDS configurations using partial oxidation and vacuum bottoms for hydrogen production. The major limitation to cutting deeper was found to be a corresponding increase in bottoms viscosity.
- A cost/benefit analysis led to the technical recommendation that operation of the 750 B/D Baytown FLEXICOKING* prototype unit on ECLP vacuum bottoms be included in the EDS program. Total first commercial plant benefits were estimated at ~300 M\$ (1980 \$), while the prototype program cost was estimated at ~30 M\$ (1980 \$).
- A study was completed to evaluate the impact of coke fines losses during EDS FLEXICOKING operations. The results show that fines losses will not limit gasification to below 83% assumed to date.
- Prototype and commercial FLEXICOKING design studies were completed. Investments were developed for both units and design data requirements were identified.

ENGINEERING RESEARCH AND DEVELOPMENT
ENGINEERING TECHNOLOGY DEVELOPMENT

4. LIQUEFACTION ENGINEERING TECHNOLOGY

- A correlation for predicting density of Illinois coal liquids was developed in the form of a series of correction factors for the effects of temperature, pressure and dissolved hydrogen on the density at 60°F and 1 atm. A preliminary correlation was developed for viscosity which is not significantly affected by dissolved hydrogen.
- The pressure drop in the three-phase -- vapor/liquid/solid -- transfer line entering the vacuum tower can be predicted by application of a correction to predicted two-phase pressure drop.
- Coal liquefaction reactor model studies confirmed the ECLP design in regard to: the solids withdrawal system, quality of inlet distribution and operable velocity range.
- Slurry rheology data is reported for liquefaction feed slurries and CLPP vacuum bottoms for both Illinois and Wyodak coals and the rheological properties reviewed. Data are also presented on CLPP low pressure separator slurry and on remelted vacuum bottoms.

- No coking of the slurry preheater or vacuum pipestill preheater was detected at conditions that meet or exceed in severity the design conditions for ECLP.

5. BOTTOMS PROCESSING ENGINEERING TECHNOLOGY

- Based on economic evaluation and operability considerations, a system of multiple hydroclone stages is the preferred technique for particulate removal from coker scrubber liquid. Alternative systems were identified, and laboratory or vendor test experimental evaluations will be performed.
- Laboratory testing showed that, at similar levels of gasification, coal-derived cokes were two to eight times more attritable than petroleum-derived cokes. Coke erosivity decreased with increasing attritability.
- A computer program to model the solids balance for the FLEXICOKING process was developed and tested against prototype and commercial data. The model is being used for EDS FLEXICOKING prototype design and the commercial plant study design.

6. MATERIALS AND ENVIRONMENTAL ENGINEERING TECHNOLOGY

- Based on projection of future regulations, continued characterization of raw wastewaters, and bench-scale testing, the proposed wastewater treating system for a commercial EDS plant should meet all future requirements. Uncertainty exists regarding the level of allowance for heavy metals in final effluent and, at what is considered the lowest level requirement, an additional treating step may be required.
- Three types of slurry block valves were identified as having the highest probability of satisfactory operating life. These three will be used in ECLP. The streamlined angle valve and choke valve, both containing trim components of cemented carbides, will be tested in high pressure slurry letdown valve service in ECLP.

ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING PROCUREMENT AND CONSTRUCTION

1. PROJECT MANAGEMENT

- Reimbursable cost, fixed fee contracts were awarded on 16 September, 1977 to Arthur G. McKee Company and Daniel Construction Company to provide engineering/procurement and construction services, respectively, for the 250 T/D, Exxon Coal Liquefaction Pilot Plant (ECLP). These contractors were selected from among the several bidders as those offering the best combination of business terms and execution potential.

- In November, 1977, an agreement was completed for Exxon Engineering's Quality Control Division and McKee's Inspection Department to share vendor plant inspection work for the project. Exxon will retain inspection on machinery, electrical equipment and instruments, and all foreign source equipment and materials. Exxon also retained overall inspection responsibility and an Exxon inspection coordinator was added to the project management team to follow this critical activity.
- A Positive Materials Identification Program was developed for the project with guidance from Exxon Engineering's Quality Control Division and Technology Department. Detailed implementation procedures for the program were issued to McKee covering vendor fabricated equipment items, and to Daniel covering bulk materials and field/shop fabricated piping. Exxon's project team inspection personnel will monitor the contractors' and fabricators' performance.

2. ENGINEERING AND PROCUREMENT

- McKee's engineering and procurement work for ECLP was 47% complete at the end of June, 1978, 8% or approximately 4 weeks behind our original plan; engineering per se was 51% complete. The Baytown Refinery Engineering Department's engineering work on the interconnecting lines was approximately 55% complete. Although the last critical path schedule update issued in mid-June indicated that the planned mechanical completion date of 15 November, 1979, is still achievable, it is highly probable that the next update in early August will indicate a schedule extension. If it is determined that an extension is indicated, we expect it will be modest -- on the order of one to two months.
- As of the end of June, 1978, inquiries were issued for 469 (99%) of the 470 tagged equipment items required for ECLP. Quotations were received for 458 items, and 441 items were purchased. Commitments for all procurement totalled 18.5 M\$, 59% of the forecasted total for the project vs 65% planned.
- The cost outlook for the ECLP Project, as of June 30, 1978, is 104 M\$. Major factors contributing to the decrease in forecasted cost of 110 M\$ were reduced materials costs, lower labor and construction indirects, and contingency rundown. These factors more than offset added costs resulting from changes.

3. CONSTRUCTION

- Site preparation work for ECLP began on 13 February, 1978. Daniel's and Exxon's construction management teams began mobilizing at the construction site on 6 March, 1978.

- Construction was 7% complete, about 3% behind our original plan, at the end of June, 1978. The major portion of work to date were in the subcontract areas of site preparation and foundation piling. Daniel's direct hire work force grew substantially and work started to accelerate on foundation and underground piping. Total Daniel manpower was 234 on 30 June; 46 salaried Daniel employees, 150 Daniel hourly employees, and 38 subcontractor employees.

4. PREPARATION FOR OPERATIONS

- The ECLP Office building was completed in early June and occupied on June 8, 1978.
- The preparation of operating manuals and other training material was initiated. A detailed work schedule for the preparation of this material is being developed.
- The ECLP staff and Refinery and Research industrial hygienists reviewed and formulated the concepts to be used for the plant. The program addresses itself to the hazards of hydrocarbons and hydrogen, H₂S and CO, dust, and polynuclear aromatic hydrocarbons. The program will have four basic elements, consisting of engineering control, environmental surveys, medical surveillance, and personal hygiene. The program is similar to that used at the Baytown R&D Division pilot plants over the last 11 years.

5. OPERATIONS

- Reporting under this category to start January 1, 1980.

6. ECLP ENVIRONMENTAL ASSESSMENT

- The ECLP Environmental Impact Negative Declaration was signed by DOE on 8 December, 1977, thus, an environmental impact statement will not be required.

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EDS COAL LIQUEFACTION PROCESS DEVELOPMENT - PHASES IIIB/IV
ANNUAL TECHNICAL PROGRESS REPORT FOR THE PERIOD
JULY 1, 1977 - JUNE 30, 1978

INTRODUCTION

This report is the first 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 - Phases IIIB and 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 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 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.

Phase IIIB which began on July 1, 1977 and Phase IV which began on January 1, 1978, include these two tasks as well as tasks relating to project management, detailed engineering, procurement, and construction of the Exxon Coal Liquefaction Pilot Plant (ECLP).

A description of the work performed in Phase IIIA can be found in the reports cited.(1,2)

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.

(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)

TABLE 1EDS COAL LIQUEFACTION PROCESS DEVELOPMENTTECHNICAL PROGRESS REPORTS PREPARED FOR DOEPHASES IIIB AND IV

<u>Report No.</u>	<u>Type</u>	<u>Period Covered</u>
FE-2893-1	Monthly	July 1 - Aug. 31, 1977
FE-2893-2	Monthly	Sept. 1 - Sept. 30, 1977
FE-2893-3	Quarterly	July 1 - Sept. 30, 1977
FE-2893-4	Monthly	Oct. 1 - Oct. 31, 1977
FE-2893-5	Monthly	Nov. 1 - Nov. 30, 1977
FE-2893-6	Monthly	Dec. 1 - Dec. 31, 1977
FE-2893-7	Quarterly	Oct. 1 - Dec. 31, 1977
FE-2893-8	Monthly	Jan. 1 - Jan. 31, 1978
FE-2893-9	Monthly	Feb. 1 - Feb. 28, 1978
FE-2893-10	Interim	Commercial Plant Study Design Update, Onsite Design Basis - Illinois Coal Case
FE-2893-11	Monthly	March 1 - March 31, 1978
FE-2893-12	Quarterly	Jan. 1 - March 31, 1978
FE-2893-13	Monthly	April 1 - April 30, 1978
FE-2893-14	Monthly	May 1 - May 31, 1978
FE-2893-15	Monthly	June 1 - June 30, 1978
FE-2893-16	Interim	Summary of EDS Predevelopment (1966-1975)
FE-2893-18	Monthly	July 1 - July 31, 1978
FE-2893-19	Monthly	Aug. 1 - Aug. 31, 1978

TABLE 2

EXXON PUBLICATIONS ON COAL LIQUEFACTION

JULY 1, 1977 - JUNE 30, 1978

Levine, D. G., Coal Conversion Technology, presented at Eighth Conference on Environmental Toxicology by the U. of Calif., October 4, 1977, Dayton, Ohio.

Swabb, L. E., Jr., Liquid Fuels from Coal: From R&D to an Industry, Science, 199, 619 (February 10, 1978)

Epperly, W. R. and Taunton, J. W., Status and Outlook of the Exxon Donor Solvent Coal Liquefaction Process Development, paper presented at the Fifth Energy Technology Conference, February 27-March 1, 1978, Washington, D.C.

Quinlan, C. W. and Siegmund, C. W., Combustion Properties of Coal Liquids from the Exxon Donor Solvent Process, paper presented at Div. of Fuel Chemistry, ACS Symposium, March 14, 1978, Anaheim, California

Fant, B. T. and Barton, W. J., Refining Coal Liquids, Preprint No. 28-78, paper presented at the API Refining Department 43rd Midyear Meeting, May 10, 1978, Toronto, Ontario, Canada

Epperly, W. R. and Taunton, J. W., Development of the Exxon Donor Solvent Coal Liquefaction Process, paper presented at the Philadelphia AIChE Meeting, June 4-8, 1978, Philadelphia, Pennsylvania.

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)⁽¹⁾ 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-2353-1).

The units are used for process variable studies, feed coal evaluations, and process improvement studies. During this reporting period the units were used for the following studies.

<u>Study</u>	<u>Coal Type</u>	<u>Mine</u>	<u>RCLU Unit No.</u>
Process Variable	Wyoming Subbituminous	Wyodak	1,2
Solvent Quality	Wyoming Subbituminous	Wyodak	1
Sulfur Dioxide Pretreatment	Wyoming Subbituminous	Wyodak	2
Solvent Hydrogen- ation Catalyst	Illinois No.6 Bituminous	Monterey No. 1	1
Screen Operability and Yields	Pittsburgh No.8 Bituminous	Ireland	1

The data for these studies are presented in Appendix A of the respective quarterly report. The operating conditions are presented in Table A-1,⁽²⁾ 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.

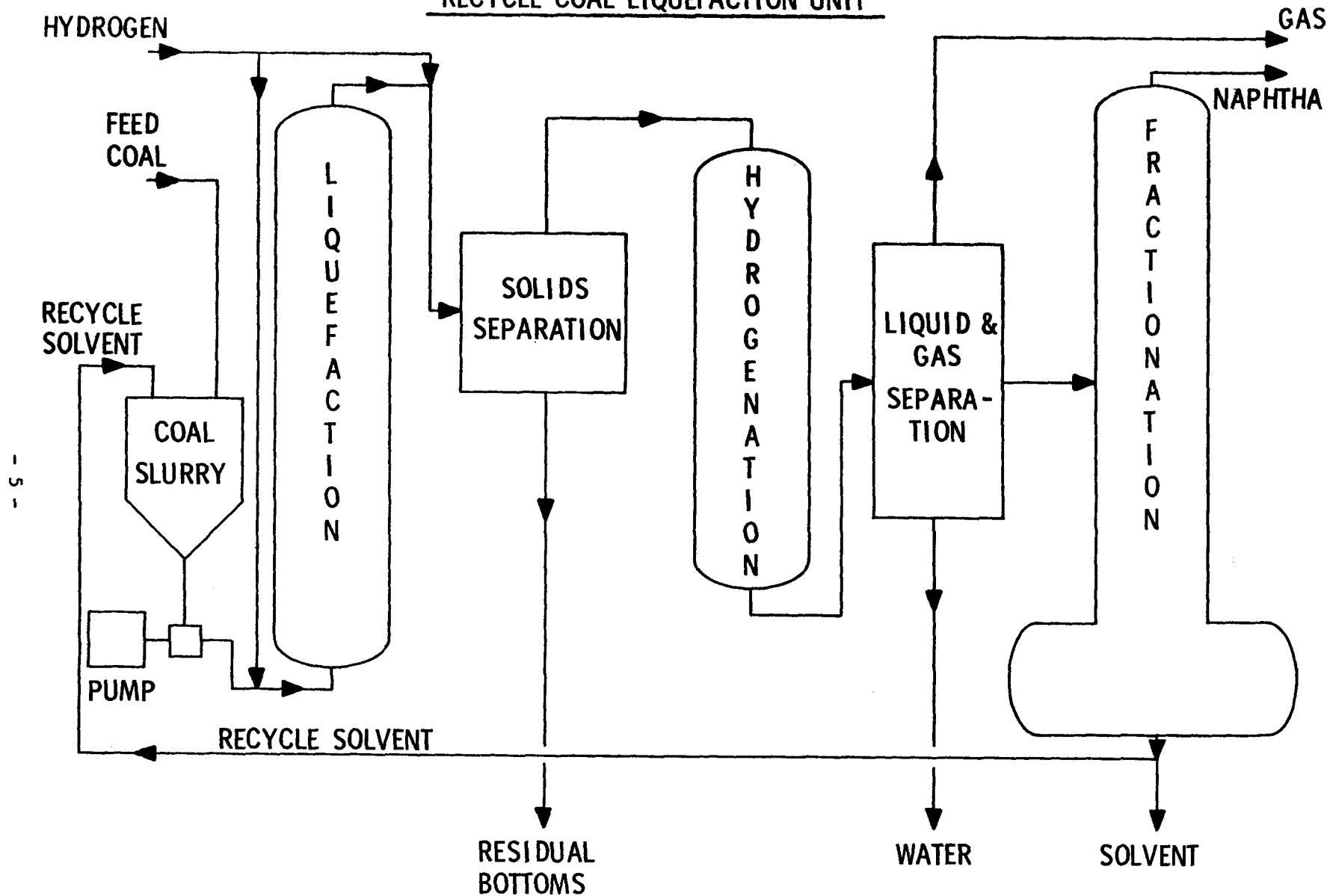
Yields from these runs are based on an elemental balancing procedure. This procedure balances the total weight of feed and products, and also balances the elements carbon, hydrogen, oxygen, sulfur, nitrogen, and total ash.

(1) 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



1.2 Wyoming Coal Process Variable Studies

The following process variable studies were completed in the Recycle Coal Liquefaction Units using Wyoming subbituminous coal from the Wyodak mine. The process variables investigated parallel those studied earlier with Illinois No. 6 coal.

- Effect of Liquefaction Residence Time on Yields
- Effect of Liquefaction Temperature on Yields
- Effect of Liquefaction Hydrogen Treat Rate on Yields

Study Conditions

The effects of liquefaction temperature and residence time on Wyoming coal yields were investigated over the ranges of 800°F to 880°F and 15 to 100 minutes, respectively. Other liquefaction conditions were 1500 psig, 1.6:1 solvent/coal ratio, and four weight percent molecular hydrogen treat rate.

In the hydrogen treat rate study, molecular hydrogen feed to the liquefaction reactor was varied by ± 50 percent of the typical rate, from four weight percent to two and six weight percent on dry coal, respectively. Other liquefaction conditions were 840°F, 1500 psig, 1.6:1 solvent/coal ratio, and 60 minutes slurry residence time.

Liquefaction Temperature and Residence Time Effect on Yields

The 1000°F conversion response to liquefaction temperature and residence time for Wyoming coal is shown in Figure 1-2. Increasing temperature and/or residence time results in increased conversion over the range of conditions studied. These data show that significant reduction in residence time is possible at constant conversion by increasing liquefaction temperature. For example, at 55 percent 1000°F conversion, the residence time can be lowered from about 115 minutes at 800°F to about 40 minutes at 840°F and 25 minutes at 880°F.

The C₄-1000°F liquid yield response to liquefaction temperature and residence time is shown in Figure 1-3. At 800°F and 840°F, the liquid yields increase with increasing residence time. The shape of the liquid yield curve is similar at both of these temperatures. However, the liquid yield response differs significantly at 880°F from that at 800°F and 840°F. At 880°F, liquid yield decreased as residence time was increased from 25 to 40 minutes.

As is evident from Figure 1-3, the maximum liquid yield is a strong function of temperature. The maximum liquid yield at 800°F occurs beyond 100 minutes residence time, while at 840°F the liquid yield reaches near maximum in the curve at about 60 minutes residence time. Clearly, the point of maximum liquid yield at 880°F occurs at a residence time below 40 minutes. The liquid yield at 880°F/25 minutes residence time is approximately equal to the maximum yield at 840°F. Thus,

FIGURE 1-2

EFFECT OF TEMPERATURE AND RESIDENCE TIME
ON WYOMING COAL 1000°F CONVERSION

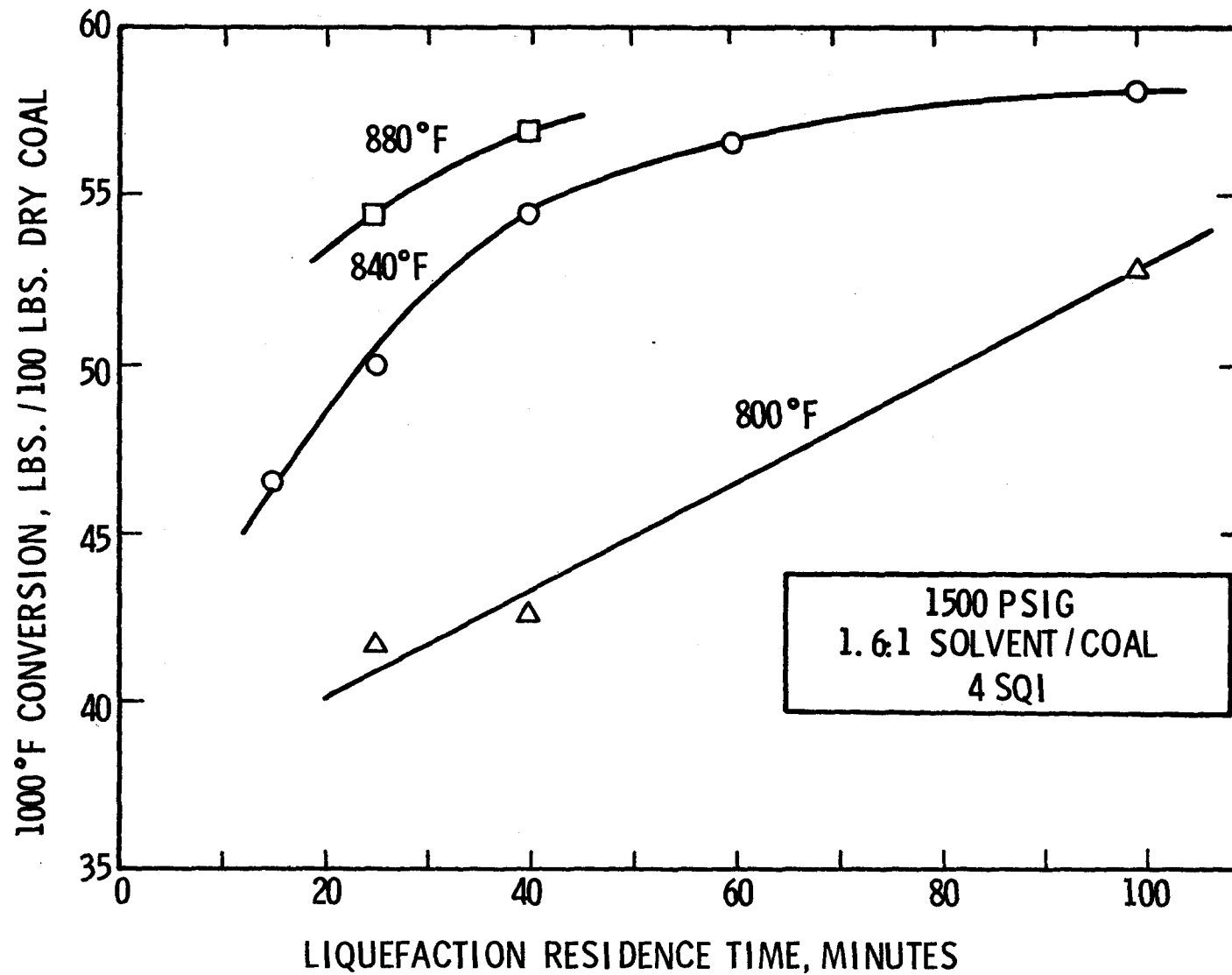
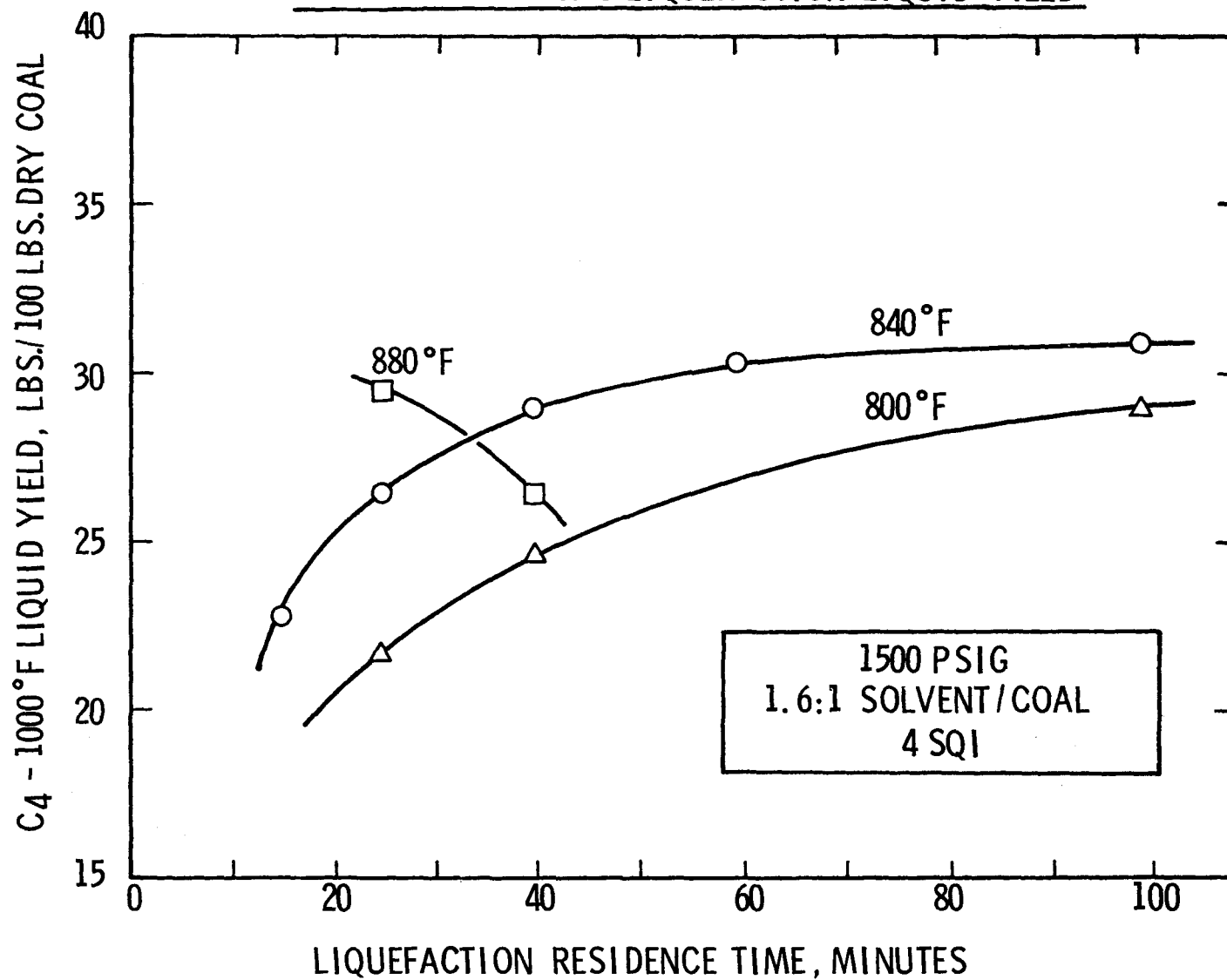


FIGURE 1-3

EFFECT OF TEMPERATURE AND RESIDENCE TIME
ON WYOMING COAL LIQUEFACTION LIQUID YIELD



if the liquefaction temperature is raised only 40°F from 840°F to 880°F, essentially the same liquid yield may be obtained by reducing the required residence time by a factor of about two. This does not address, of course, the numerous equipment design implications associated with the higher temperature operation at 880°F.

The hydrocarbon gas yields are shown in Figure 1-4. The C₁-C₃ yields increase with increasing residence time at all three liquefaction temperatures studied. The C₁-C₃ gas yield at 880°F, however, shows a much greater response to increases in residence time than the gas yields at the two lower temperatures. Apparently, much more thermal cracking of liquids to hydrocarbon gases occurs at the higher conversion levels obtained at 880°F liquefaction temperature. This increase in gas make is consistent with the decrease in C₄-1000°F liquid yield as residence time was increased from 25 to 40 minutes.

The water and chemical gas yields remained about constant over the range of temperature/residence times studied.

Hydrogen consumption is shown in Figure 1-5. These data include hydrogen consumed in both the liquefaction and hydrogenation reactions. Hydrogen consumption increases as temperature and/or residence time increases, which is consistent with the total conversion response shown in Figure 1-2. As shown, the hydrogen consumption increases dramatically as the liquefaction temperature is increased to 880°F at constant residence time. However, the hydrogen consumption for maximum C₄-1000°F liquid yield at 840°F (4.3-4.6 percent consumption at 60-80 minutes residence time) is comparable to that at 880°F for the same C₄-1000°F liquid yield (4.5 percent consumption at 25 minutes residence time).

Liquefaction Temperature and Residence Time Effect on Unit Operability (Residual Bottoms Removal)

Viscosity measurements and pyridine extractions performed on both Wyoming and Illinois residual bottoms have produced results which have increased our understanding of the processing of these coals. The liquid yield data have shown that more severe liquefaction conditions are required for Wyoming coal than for Illinois coal (July-September, 1977, EDS Quarterly Technical Progress Report [FE-2893-3]). The viscosity and pyridine extraction data also reinforce this fact from a unit operability viewpoint.

Figure 1-6 shows the residual bottoms viscosity as a function of liquefaction residence time. Residual bottoms viscosity of Illinois coal is independent of residence time (liquefaction severity) over the broad range of 6 to 100 minutes. This low Illinois bottoms viscosity is consistent with the easy withdrawal of bottoms from the residual bottoms accumulator. In contrast, Wyoming coal bottoms viscosity is much greater than for Illinois bottoms at short residence times. As residence time is increased, the Wyoming bottoms viscosity drops dramatically. Above residence times of 60 minutes, the Wyoming bottoms viscosity becomes independent of residence time and about equal to Illinois bottoms viscosity.

FIGURE 1-4

EFFECT OF TEMPERATURE AND RESIDENCE TIME
ON WYOMING COAL LIQUEFACTION GAS YIELD

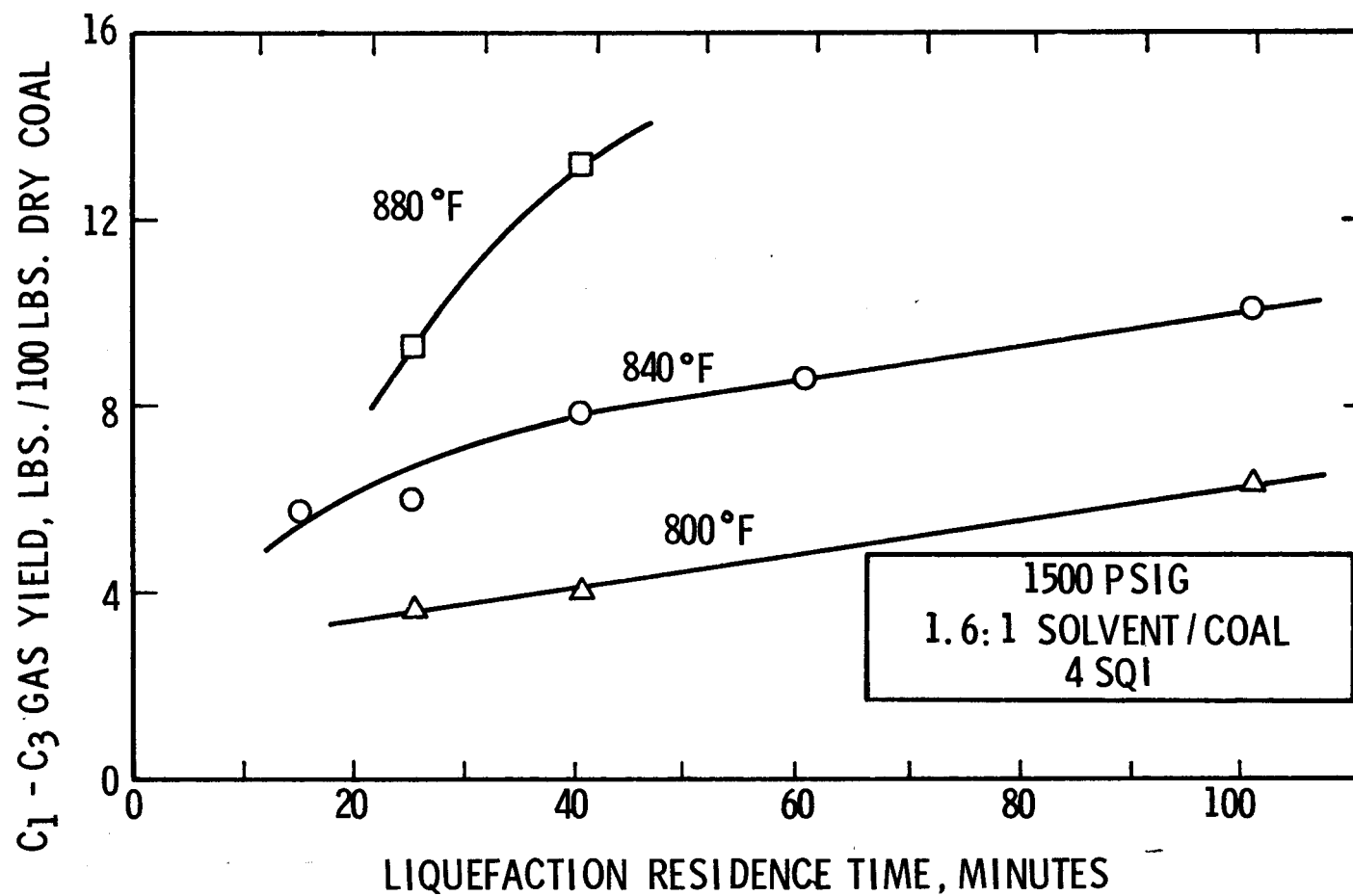


FIGURE 1-5

EFFECT OF TEMPERATURE AND RESIDENCE TIME ON
WYOMING COAL HYDROGEN CONSUMPTION

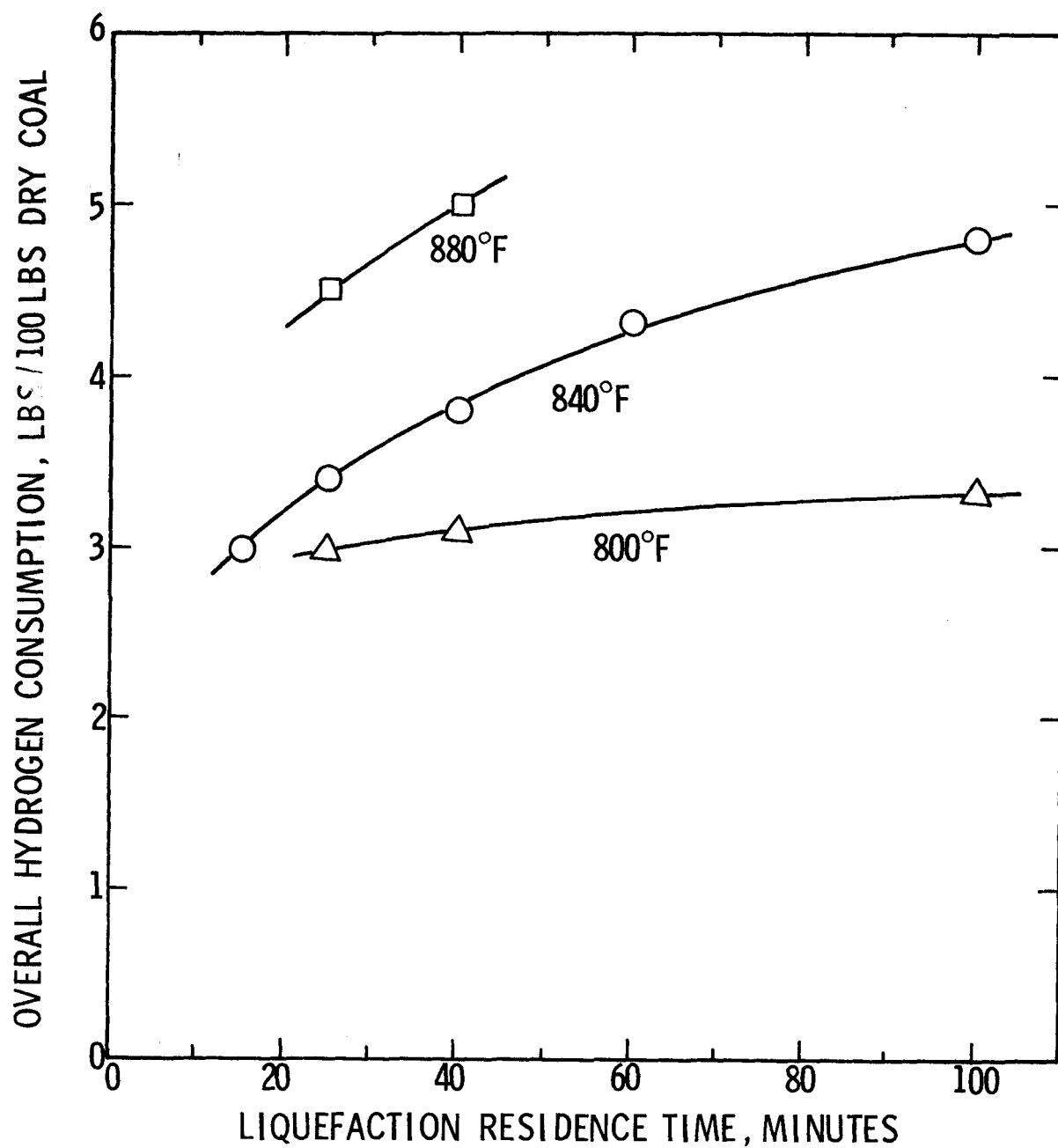
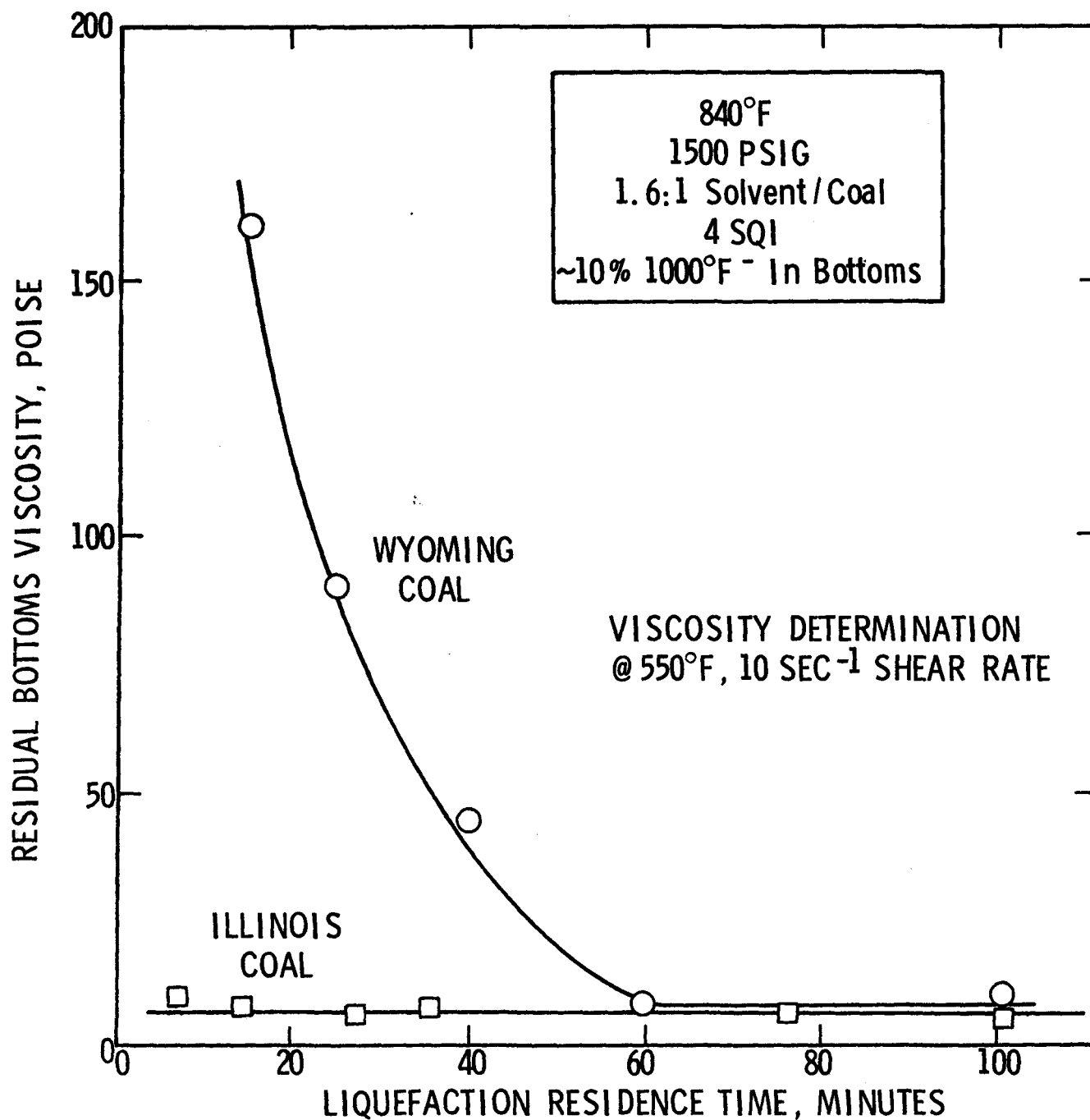


FIGURE 1-6

EFFECT OF LIQUEFACTION RESIDENCE TIME ON VISCOSITY OF
RESIDUAL LIQUEFACTION BOTTOMS FROM WYOMING COAL



These viscosity results help explain the RCLU experience in handling the residual bottoms. At residence times less than 40 minutes, the bottoms were increasingly difficult to remove from the residual bottoms accumulator. Plugs frequently developed in the accumulator outlet line and dump valve, causing the unit to shut down. At residence times greater than 40 minutes, the number of dump valve and line plugs decreased dramatically due to much lower bottoms viscosity.

Figure 1-7 shows the residual bottoms viscosity as a function of liquefaction temperature at 25 minutes residence time. These results also correlate well with RCLU bottoms handling experience. Plugs were experienced in the bottoms accumulator at 800°F and 840°F. However, no residual bottoms handling problems were encountered at 880°F and 25 minutes residence time. As shown, the bottoms viscosity at 880°F has decreased by about a factor of eight from that at 840°F. Thus, the improved bottoms accumulator operation experienced would be expected.

In contrast, at 880°F and 40 minutes residence time considerable difficulty was encountered in removing the residual bottoms from the bottoms accumulator. Four runs were terminated by plugs in the liquefaction reactor, and one was terminated by a plug in the bottom of the residual bottoms accumulator. The bottoms viscosity also increased from 12 poise at 880°F/25 minutes to 37 poise at 880°F/40 minutes. This is contrary to the Wyoming coal bottoms viscosity behavior at 800°F and 840°F. At both of these liquefaction temperatures, as liquefaction severity (residence time) increased, the bottoms viscosities decreased.

Apparently, the more severe liquefaction conditions at 880°F have produced a bottoms product of greater molecular weight. This higher molecular weight bottoms also accounts for the observed decrease in pyridine extraction conversion at 880°F as residence time is increased from 25 to 40 minutes, as shown in Figure 1-8. Except for the 880°F/40 minute data point, the pyridine conversion shows excellent agreement with the 1000°F conversion. The points on the curve represent increasing liquefaction severity conditions as conversion increases.

Liquefaction Hydrogen Treat Rate Effect on Yields

The effect of molecular hydrogen availability could be important in the liquefaction of Wyoming coal. A study performed earlier with Illinois No. 6 coal (January, 1976-July, 1977, EDS Final Technical Progress Report, [FE-2353-20]) showed the liquefaction yields were affected by hydrogen treat rate when using marginal quality solvent. No yield effects were apparent when using typical quality solvent. Since pyridine extraction data show that Wyoming coal requires higher severity liquefaction conditions than Illinois No. 6 coal to maximize liquid yield (July-September, 1977, EDS Quarterly Technical Progress Report [FE-2893-3]), molecular hydrogen could affect Wyoming coal yields moreso than for Illinois No. 6 coal.

As shown in Table 1-1, the data indicate no significant effect on yields or conversion as a result of decreasing the hydrogen treat rate from

FIGURE 1-7

EFFECT OF LIQUEFACTION TEMPERATURE ON VISCOSITY OF
RESIDUAL LIQUEFACTION BOTTOMS FROM WYOMING COAL

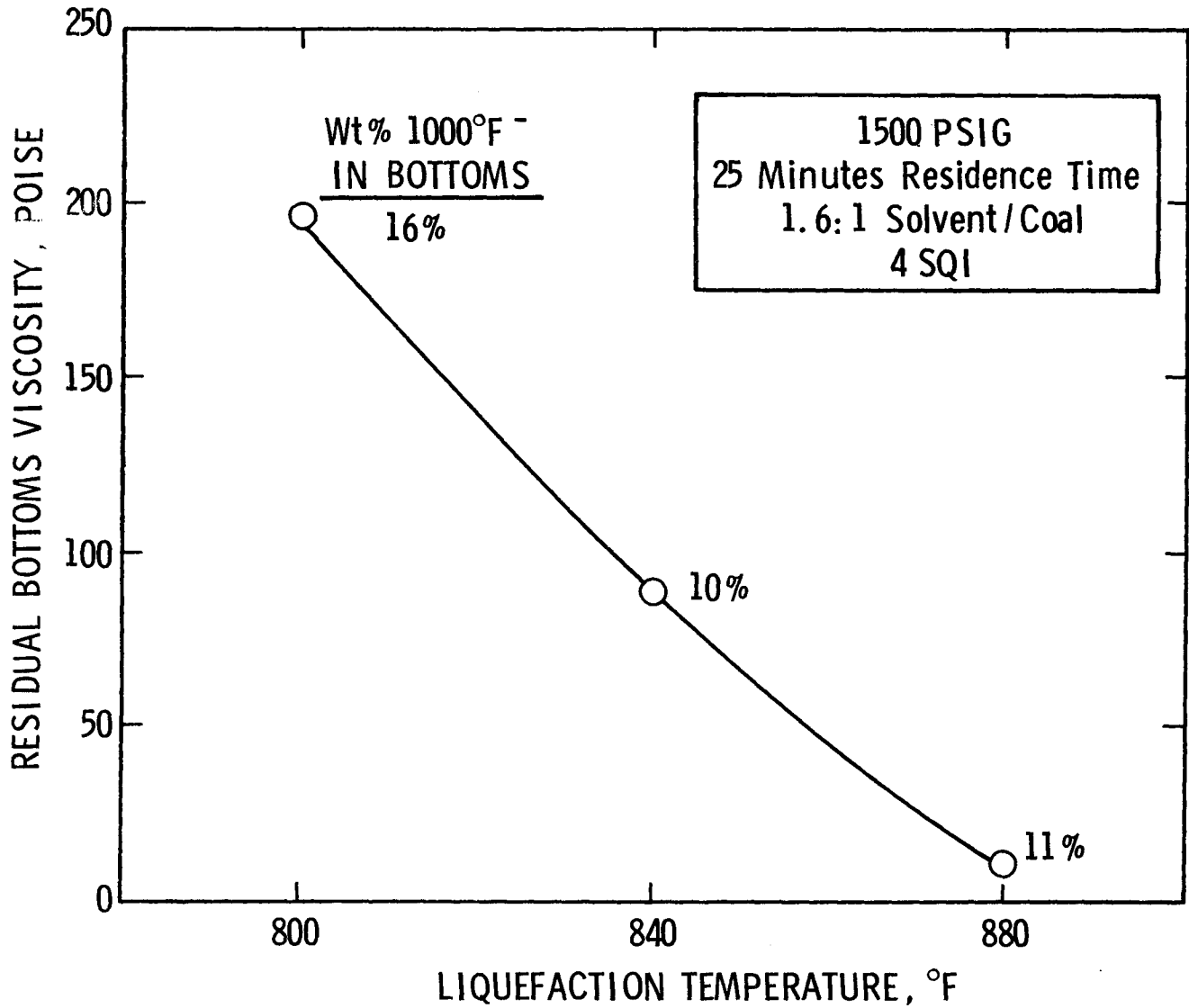
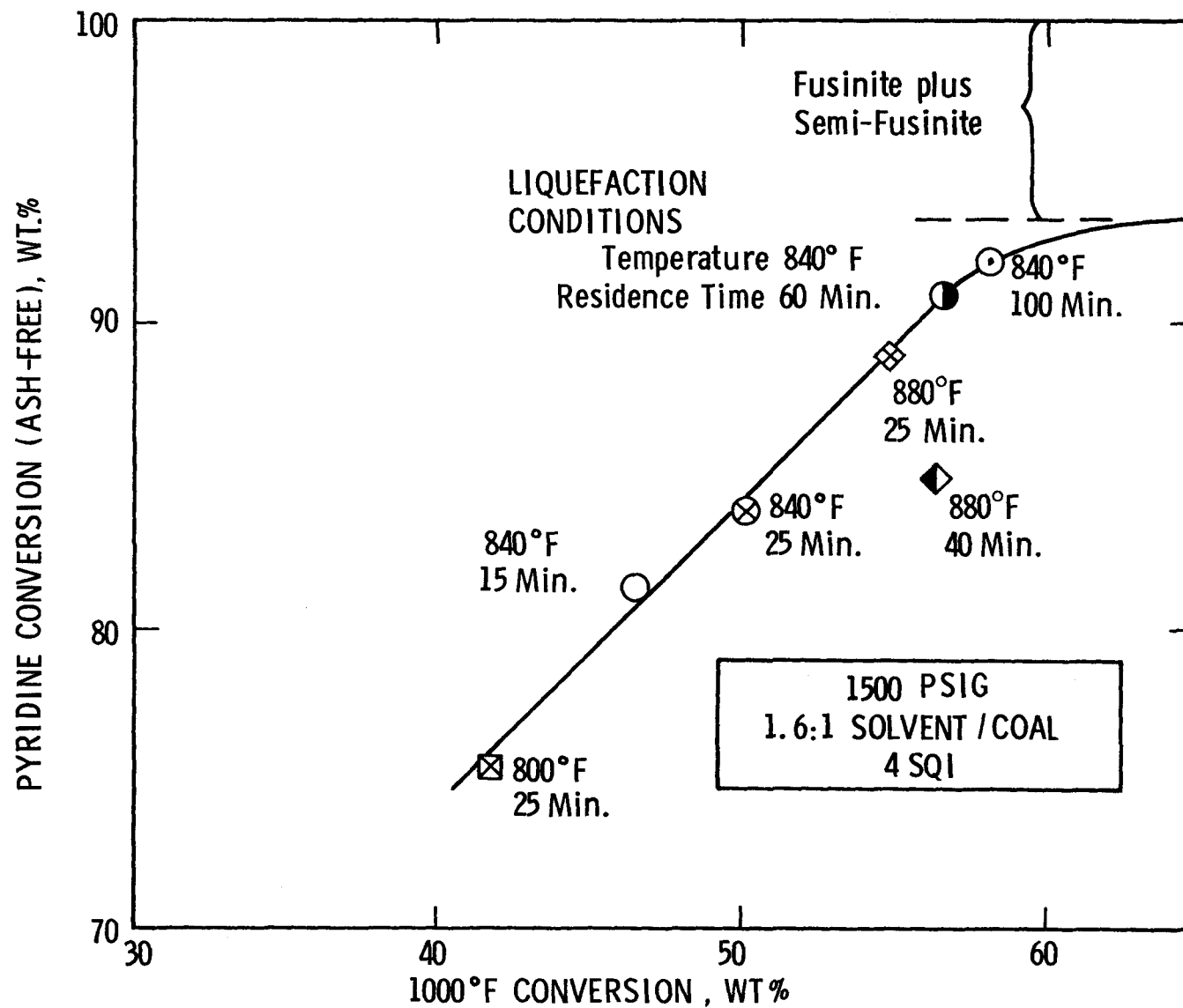


FIGURE 1-8

PYRIDINE CONVERSION VERSUS 1000°F CONVERSION
FOR WYOMING COAL



four weight percent to two weight percent. Increasing the hydrogen treat rate from four weight percent to six weight percent resulted in a decrease in C₄-1000°F liquid yield (i.e., three weight percent lower liquids yield than one standard deviation value at four percent hydrogen treat). The conversion and C₁-C₃ gas yield at six percent hydrogen treat were just outside the one standard deviation values at four percent hydrogen treat. Since only one yield period was run at six percent hydrogen treat, this data is open to conjecture. However, it is clear that increasing the hydrogen treat rate from four weight percent to six weight percent did not enhance liquid yield.

TABLE 1-1

EFFECT OF HYDROGEN TREAT RATE ON WYOMING COAL YIELDS

<u>Liquefaction Conditions</u>	840°F 1500 psig 60 minutes residence time		
<u>Yield Periods</u>	85,86 87,88	448,449 450,451	510
<u>Nominal Hydrogen Treat Rate, Wt% on Dry Coal</u>	2	4	6
<u>Overall Yields, Lbs/100 Lbs Dry Coal</u>			
C ₁ -C ₃	8.7±0.8*	8.6±1.1	9.8
C ₄ -1000°F Liquid	30.3±1.3	30.7±1.6	26.0
<u>1000°F Conversion Lbs/100 Lbs Dry Coal</u>	55.5±1.4	56.4±1.1	54.7
<u>Pyridine Conversion, Wt% (Ash Free)</u>	91	91	89
<u>Benzene Conversion, Wt% (Ash Free)</u>	69	67	71

1.3 Wyoming Coal Solvent Quality Study

Previous bench unit and RCLU studies with Illinois No. 6 coal have shown that liquefaction yields are strongly dependent on solvent quality (January-June, 1976, EDS Quarterly Technical Progress Report, [FE-2353-2]). Bench unit studies with Wyoming coal have shown a yield

*Standard Deviation

dependence on solvent quality similar to that of Illinois coal. The purpose of this study in RCLU was to ascertain the effects of solvent quality on Wyoming coal yields in a continuous flow operation with recycle solvent. As in the previous studies, Solvent Quality Index (SQI) was used as the main criterion of solvent quality.

- Study Conditions

In these studies, the SQI was controlled by adjusting the hydrotreater temperature and the solvent-to-coal ratio. The hydrotreater temperature was decreased stepwise in about 20°F increments over a range of 100°F and the recycle solvent-to-coal ratios used were 1.6:1 and 3:1. Other liquefaction conditions were 840°F, 1500 psig, and four weight percent hydrogen treat rate. Solvent quality effects on yields were determined at 25 minute and at 60 minute slurry residence times. The hydrotreating catalyst used was Co/Mo-10.

- Conversion and Yields

The effect of solvent quality on Wyoming coal conversion is shown in Figure 1-9. At SQI levels greater than four, coal conversion at 25 minutes residence time is relatively independent of solvent quality. However, when the SQI is decreased below four, the conversion decreases rapidly. These results agree closely with those from the bench unit solvent quality studies using Wyoming coal. The decrease in conversion with decreasing SQI at 60 minutes residence time closely parallels that for 25 minutes residence time over the range of SQI studied. Conversion at 60 minutes residence time is about 10 lbs/100 lbs dry coal higher than at 25 minutes residence time over the SQI range of two to four.

With Illinois No. 6 coal, the C₄-1000°F liquid yield showed a response to SQI similar to that for conversion. Figure 1-10 shows that for Wyoming coal, the C₄-1000°F liquid yield response to SQI also parallels the conversion results. At 25 minutes residence time, liquid yield is nearly independent of SQI above about four. As SQI decreases below four, the liquid yield decreases dramatically. At 60 minutes residence time, the liquid yield response is similar, and about 5 lbs/100 lbs dry coal higher than the 25 minute residence time liquid yield over the SQI range of two to four.

No significant effects of solvent quality on water, chemical gas, and C₁-C₃ gas yields were found at constant residence time. Illinois No. 6 coal also showed no effects of SQI on water, chemical gas, and hydrocarbon gas yields.

Figure 1-9 shows that by increasing liquefaction severity from 25 minutes residence time to 60 minutes residence time, a lower quality recycle solvent can be used to obtain equivalent conversion levels. Table 1-2 compares the yields at 25 minutes residence time using standard quality solvent with yields at 60 minutes residence time at equivalent coal conversion.

FIGURE 1-9

EFFECT OF SOLVENT QUALITY ON CONVERSION
OF WYOMING COAL

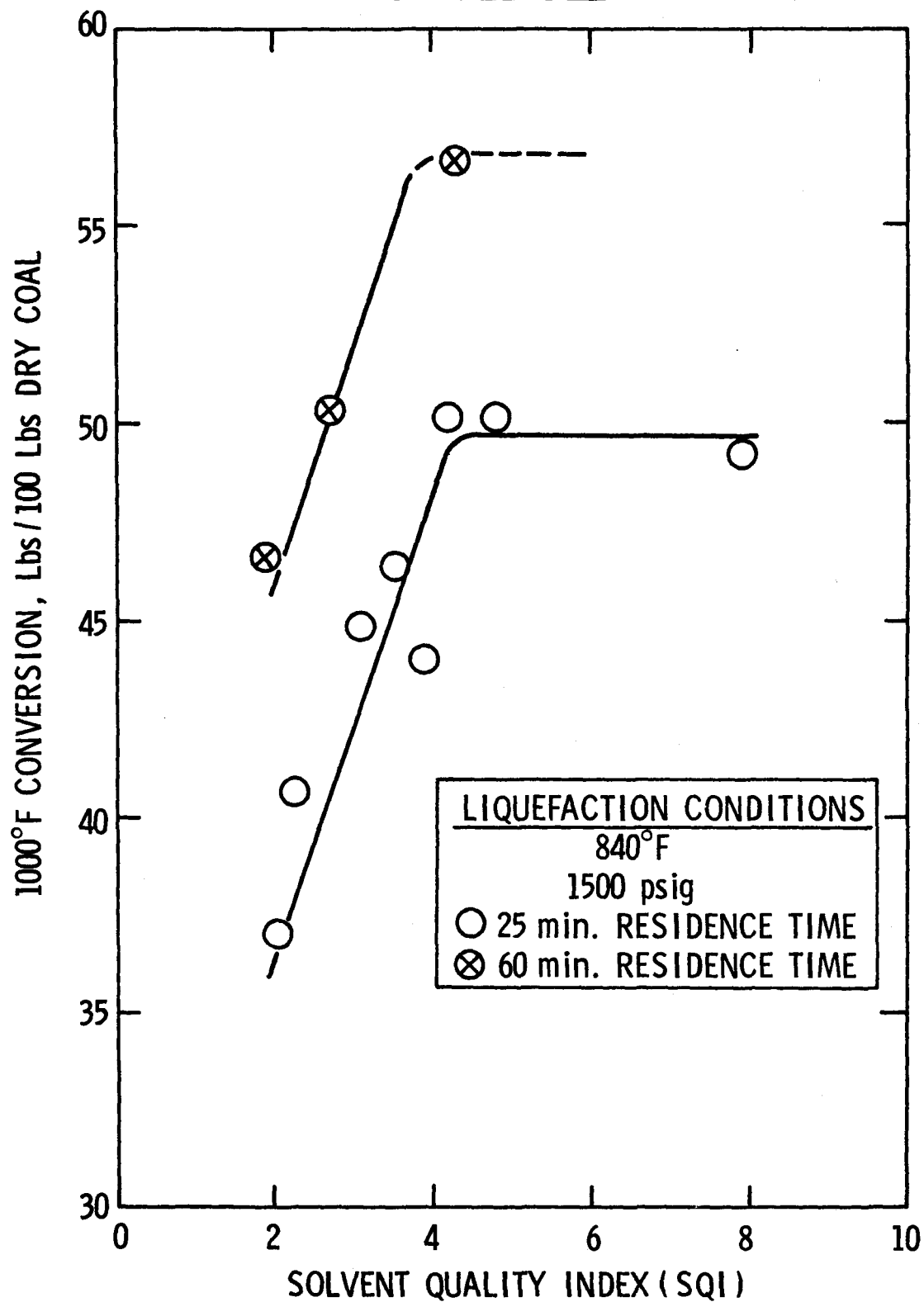
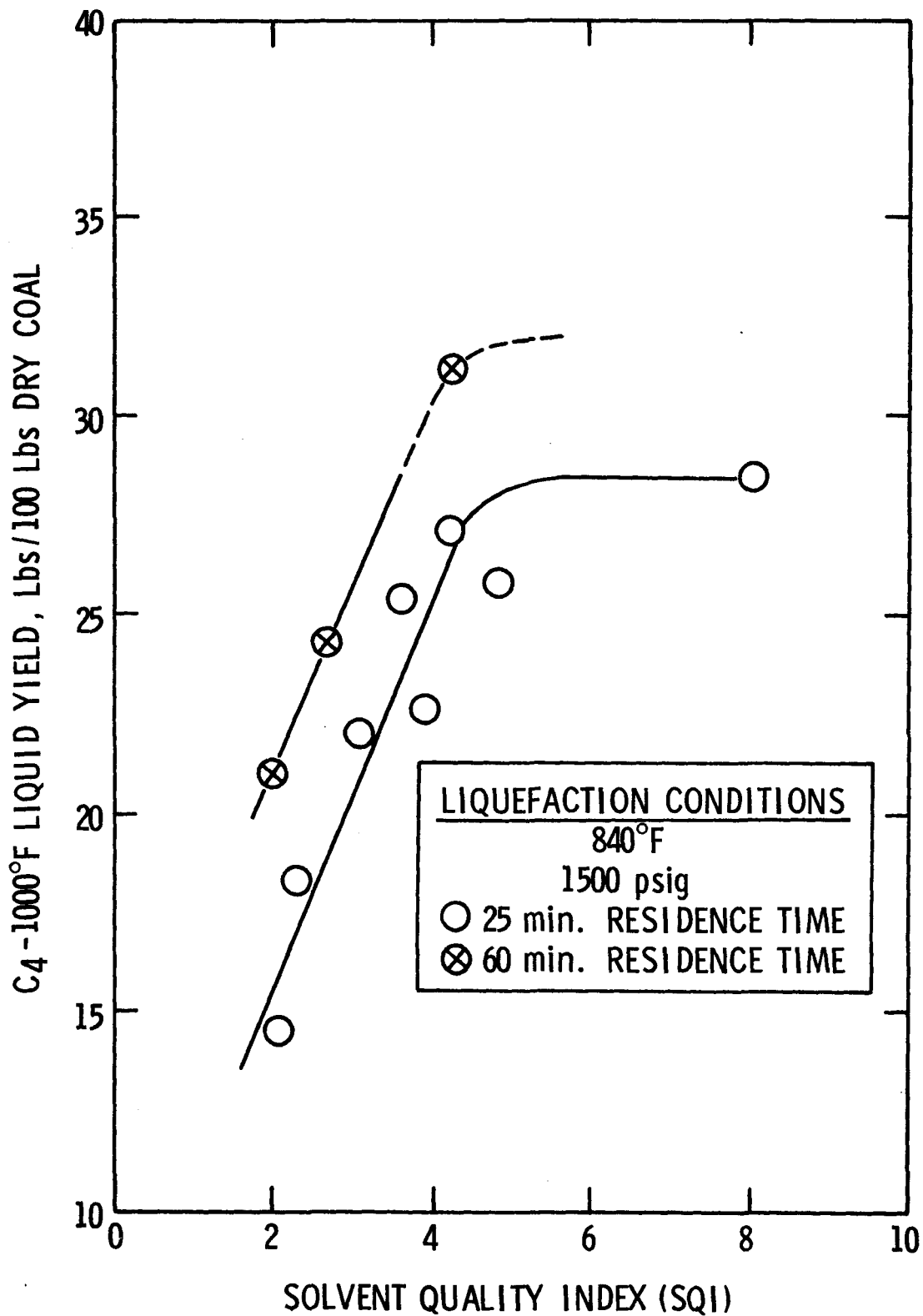


FIGURE 1-10

EFFECT OF SOLVENT QUALITY ON C₄-1000°F
LIQUID YIELD FROM WYOMING COAL



These data show that the conversion debit incurred with a low quality solvent can be regained by increasing the liquefaction severity, i.e., increasing residence time. However, the liquid yields are still somewhat lower than the liquid yields at the same conversion level using standard solvent (i.e., SQI ≥ 4). Most of this liquid yield debit at 60 minutes residence time using a low quality solvent is due to increased C₁-C₃ gas make because of the increased liquefaction severity. Very similar results were obtained from the Illinois No. 6 coal solvent quality study.

TABLE 1-2

EFFECT OF RESIDENCE TIME ON YIELDS USING LOW SQI SOLVENT

	378,379,393	
	394,395,404	
	405,406,407	485,486
<u>Yield Periods</u>	<u>408</u>	<u>487,488</u>
<u>Residence Time, min.</u>	25	60
<u>Solvent Quality Index</u>	4.5	2.7
<u>Overall Yields, Lbs/100 Lbs Dry Coal</u>		
C ₁ -C ₃	5.9	8.6
C ₄ -1000°F Liquid	26.2	24.2
<u>1000°F Conversion, Lbs/100 Lbs Dry Coal</u>	49.7	50.2

• Unit Operations

Unit operability during the solvent quality study was similar to previous RCLU experience with Wyoming coal. Nearly all the runs were terminated by reactor plugs prior to 100 hours on coal. Also the unconverted liquefaction residue was increasingly more difficult to remove from the residual bottoms accumulator as solvent quality was decreased at 25 minutes residence time. At the lowest SQI of two, considerable 1000°F liquid had to be retained in these residual bottoms so they could be removed without plugging the accumulator dump valve.

Figure 1-11 shows that the residual bottoms viscosity increases dramatically as coal conversion, i.e., SQI, decreases. Thus, the difficulties experienced in emptying the residual bottoms accumulator at 25 minutes residence time and low SQI (low conversion) would be expected based on this residual bottoms viscosity curve.

Pyridine and benzene extractions of residual bottoms also reflect the same effect of change in solvent quality. As shown in Figure 1-12, both pyridine and benzene extraction conversions decrease substantially as 1000°F conversion (SQI) decreases. These extraction data show that the organic fraction which is benzene-insoluble/pyridine-soluble

FIGURE 1-11

VISCOSITY OF RESIDUAL LIQUEFACTION BOTTOMS
VERSUS 1000°F CONVERSION FOR WYOMING COAL

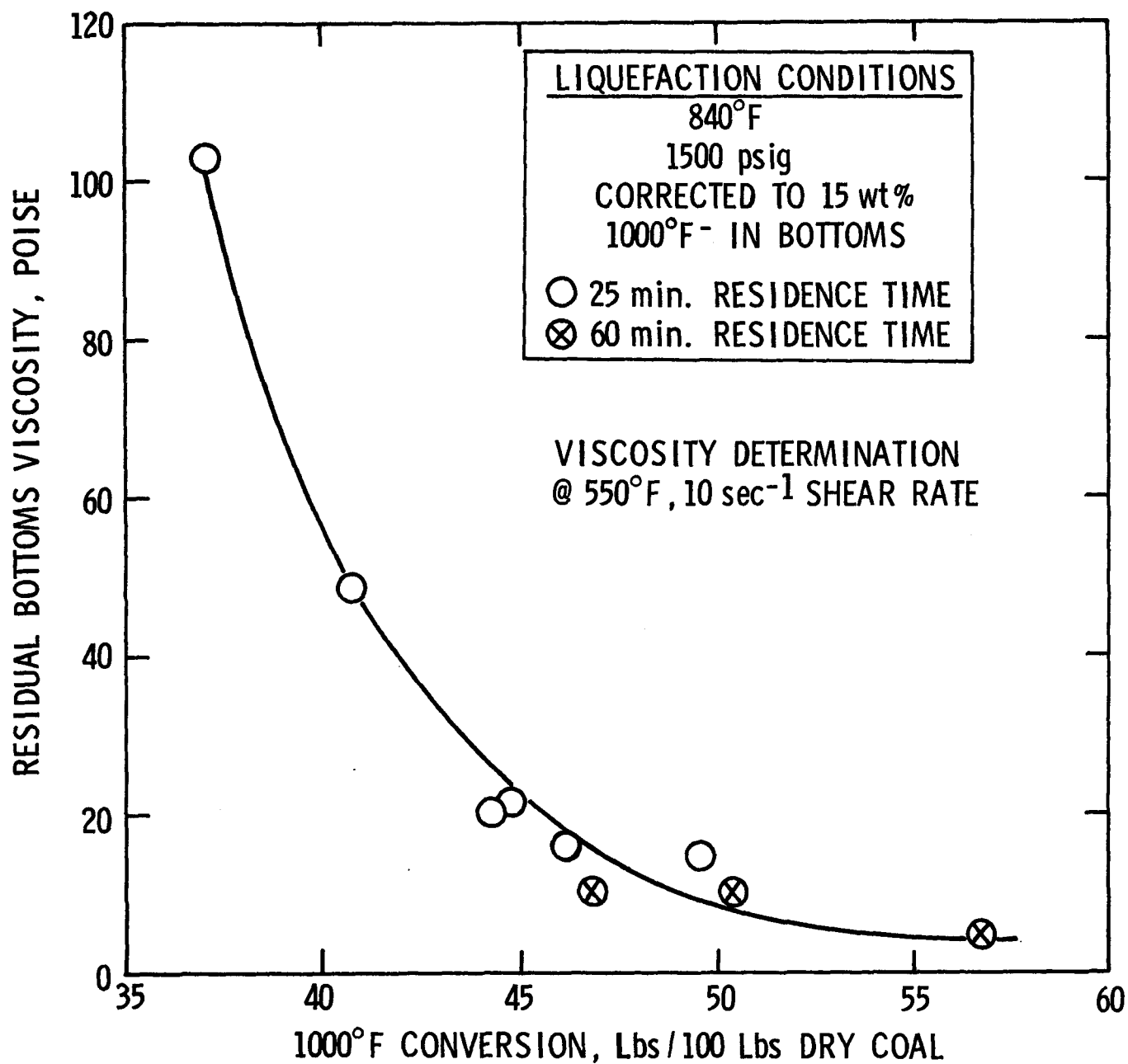
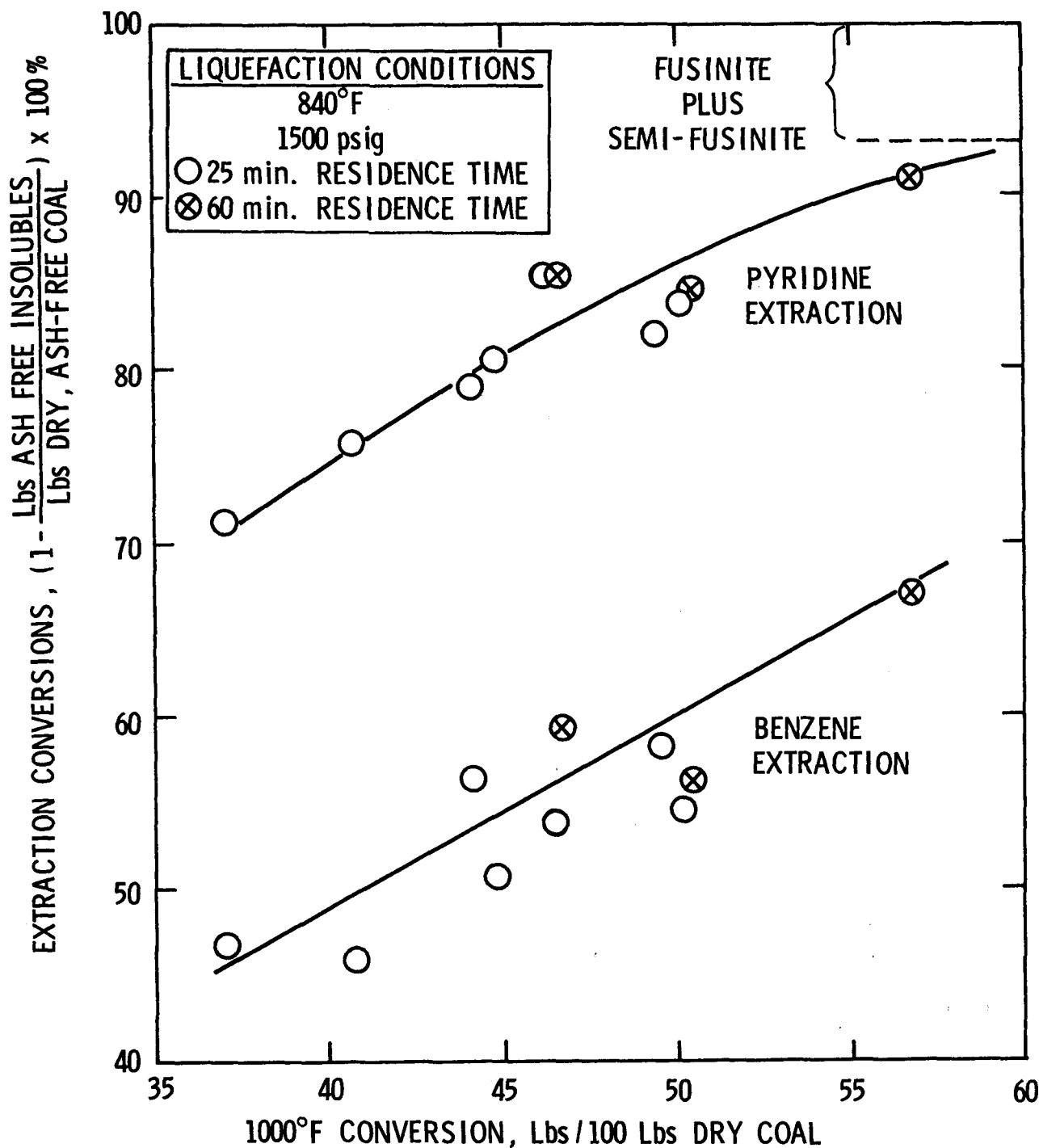


FIGURE 1-12

EXTRACTION CONVERSIONS VERSUS 1000° F CONVERSION
FOR WYOMING COAL



remains nearly constant at about 22 weight percent over the range of 1000°F conversion (SQI) studied. Thus, the decrease in coal conversion and liquid yield with decreasing SQI is due to the increase in the pyridine-insoluble organic fraction. This fraction is presumably difficult-to-convert, high molecular weight material. The residual bottoms viscosities are also consistent with these conversion results.

1.4 Wyoming Coal Sulfur Dioxide Pretreatment Study

Gas phase sulfur dioxide pretreatment of Wyoming coal, like sulfuric acid pretreatment, has reduced the formation of calcium carbonate deposits during liquefaction in small, bench scale units. Wyoming coal pretreatment with sulfur dioxide is being investigated as an alternate, less expensive method of treatment than sulfuric acid. The purpose of this study in RCLU was to determine the effect of sulfur dioxide pretreatment on Wyoming coal conversion and yields, and to further investigate the effect on calcium carbonate deposition in a recycle solvent unit.

● Study Conditions

The coal was treated with gaseous sulfur dioxide for seven days in five to ten barrel batch sizes in a large drier.

The liquefaction conditions for this study were 840°F, 1500 psig, 1.6:1 solvent-to-coal ratio, and four weight percent hydrogen treat rate. Three slurry residence times of 40, 60, and 100 minutes were investigated.

● Results of Pretreatment

The results show that pretreatment with gaseous sulfur dioxide was unsuccessful in preventing the formation of calcium carbonate deposits during liquefaction of Wyoming coal. A summary of RCLU operations with sulfur dioxide treated coal is presented in Table 1-3. Calcium carbonate formation was not substantially reduced by the pretreatment during the first three runs. However, the fourth run resulted in very little reactor scale. Also there was a significant reduction in the calcium carbonate content of the scale and the residual bottoms ash during this run as compared to untreated coal runs. Operations with the sulfur dioxide treated Wyoming coal were very similar to operations with untreated Wyoming coal. The run lengths for the pretreated coal were comparable to those with untreated coal.

An investigation is underway into possible reasons for the differences between the pretreatment experience in RCLU and earlier bench scale studies which were more successful in eliminating calcium carbonate scale formation. Such pretreatment variables as coal moisture content, treat length, sulfur dioxide partial pressure, treat temperature, and degree of sulfur dioxide/coal contacting are under study.

TABLE 1-3

SUMMARY OF RCLU OPERATIONS WITH SULFUR DIOXIDE PRETREATED WYOMING COAL

Coal Batch RCLU-2 Run No.	Untreated	Sulfur Dioxide Treated		
		No. 1 <u>1,2</u>	No. 2 <u>3</u> <u>4</u>	
<u>Yield Periods</u>		79,80 81,82 <u>83</u>	<u>89,90</u>	<u>92,93</u>
<u>Reactor Wall Scale</u>				
Total Lb/100 Lb Coal	0.002-0.007	0.013	0.010	0.001
CaCO ₃ in Scale, Wt%	70-85	43-63	6-59	2-32
CaCO ₃ in Residual	30-50	15-31	11-15	9-13
<u>Bottoms Ash, Wt%</u>				
<u>Treatment Effective?</u>	-	No	No	Marginally

• Conversion and Yields

A comparison of the yields from sulfur dioxide pretreated coal with untreated coal is presented in Table 1-4. The yields are very similar at comparable conditions, except that the hydrogen sulfide and water yields are slightly higher as a result of the pretreatment step.

TABLE 1-4

EFFECT OF SULFUR DIOXIDE TREATING ON WYOMING COAL LIQUEFACTION YIELDS

Liquefaction Conditions: 840°F
1500 psig
1.6 Solvent/Coal Ratio
4 Wt% H₂ Treat Rate

	Untreated Wyoming Coal			Sulfur Dioxide Treated Wyoming Coal		
	40	60	100	40	60	100
<u>Residence Time, min.</u>						
<u>Yield Periods</u>	329,330 <u>331</u>	448,449 <u>450,451</u>	432,433 <u>434</u>		79,80,81 82,91,92 <u>93</u>	<u>83,89</u>
<u>Liquefaction Yields, Lbs/100 Lbs Dry Coal</u>						
H ₂ O	15.0	14.8	15.5	16.0	16.6	16.4
H ₂ S	0.3	0.3	0.3	0.8	1.0	1.0
C ₁ -C ₃ Gas	7.9	8.6	10.1	7.6	9.6	12.6
C ₄ -1000°F Liquids	29.0	30.7	30.9	26.7	30.3	30.7
1000°F ⁺ Bottoms	45.5	43.6	41.9	47.9	42.2	39.1

1.5 Solvent Hydrogenation Catalyst Study

A solvent hydrogenation catalyst study was conducted in the 50 pound-per-day Recycle Coal Liquefaction Unit (RCLU-1). The primary purpose of the study was to determine the effects of Ni/Mo-10 catalyst on liquefaction yields and conversion of Illinois No. 6 coal in a recycle solvent unit. A secondary objective was to determine what effects, if any, an extended continuous run with Ni/Mo-10 catalyst would have on the recycle solvent quality. Previous RCLU operations have used Co/Mo-10 catalyst in the solvent hydrotreater.

● Study Conditions

The liquefaction conditions chosen for this study were from the updated Illinois coal commercial study design. The hydrotreating conditions used were from the commercial study design and bench unit results with Ni/Mo-10 catalyst. The commercial hydrotreater value of $2.63 \times \text{RV}$ space velocity was used in RCLU at 1500 psig. This is about 30 percent higher than the typical RCLU hydrotreater space velocity of 2.0. A $125^\circ\text{F} + \text{RV}$ start-of-run temperature was selected based on the bench unit data. This is about 50°F lower than required for comparable solvent quality with the less active Co/Mo-10 catalyst. The study conditions are given below.

Liquefaction

Temperature, $^\circ\text{F}$	840
Pressure, psig	1500
Nominal Slurry Residence Time, min.	40
Hydrogen Treat Rate, Wt% on Dry Coal	4
Solvent/Coal Ratio	1.6

Hydrotreating

Temperature, $^\circ\text{F}$	$125^\circ\text{F} + \text{RV}$
Pressure, psig	1500
Space Velocity, W/Hr/W	$2.63 \times \text{RV}$
Target solvent quality index of 4.0	

● Solvent Quality

Operations were extremely smooth throughout the study. The unit was on coal for 612 continuous hours before being shut down due to a plug in the liquefaction reactor feed inlet line. The run of 612 hours on coal set a new RCLU record for Illinois No. 6 coal. Twenty-one yield periods were completed at these conditions.

Target solvent quality was maintained throughout the run without increasing the hydrotreater temperature from the start-of-run value. The hydrotreater temperature as a function of unit run time is shown in Figure 1-13. The resulting recycle solvent quality is shown in Figure 1-14. As shown, after about 200 hours on coal, solvent quality was maintained at the target level of four SQI for the remainder of the run.

FIGURE 1-13

HYDROTREATER TEMPERATURE PROFILE DURING
Ni/Mo-10 CATALYST TEST RUN

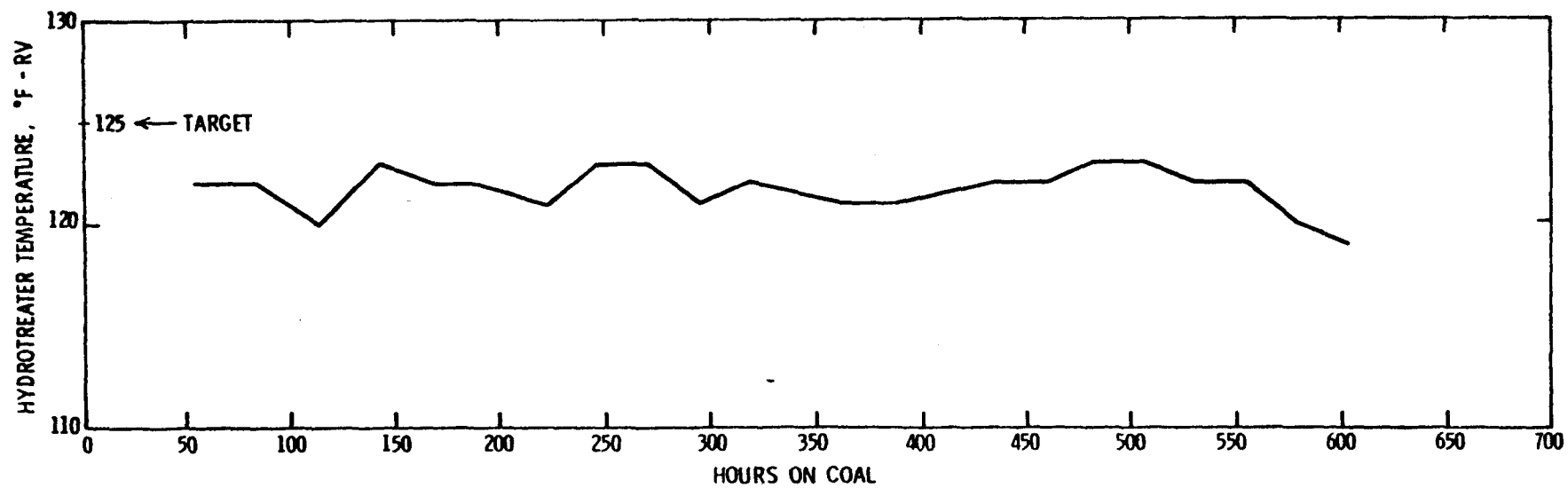
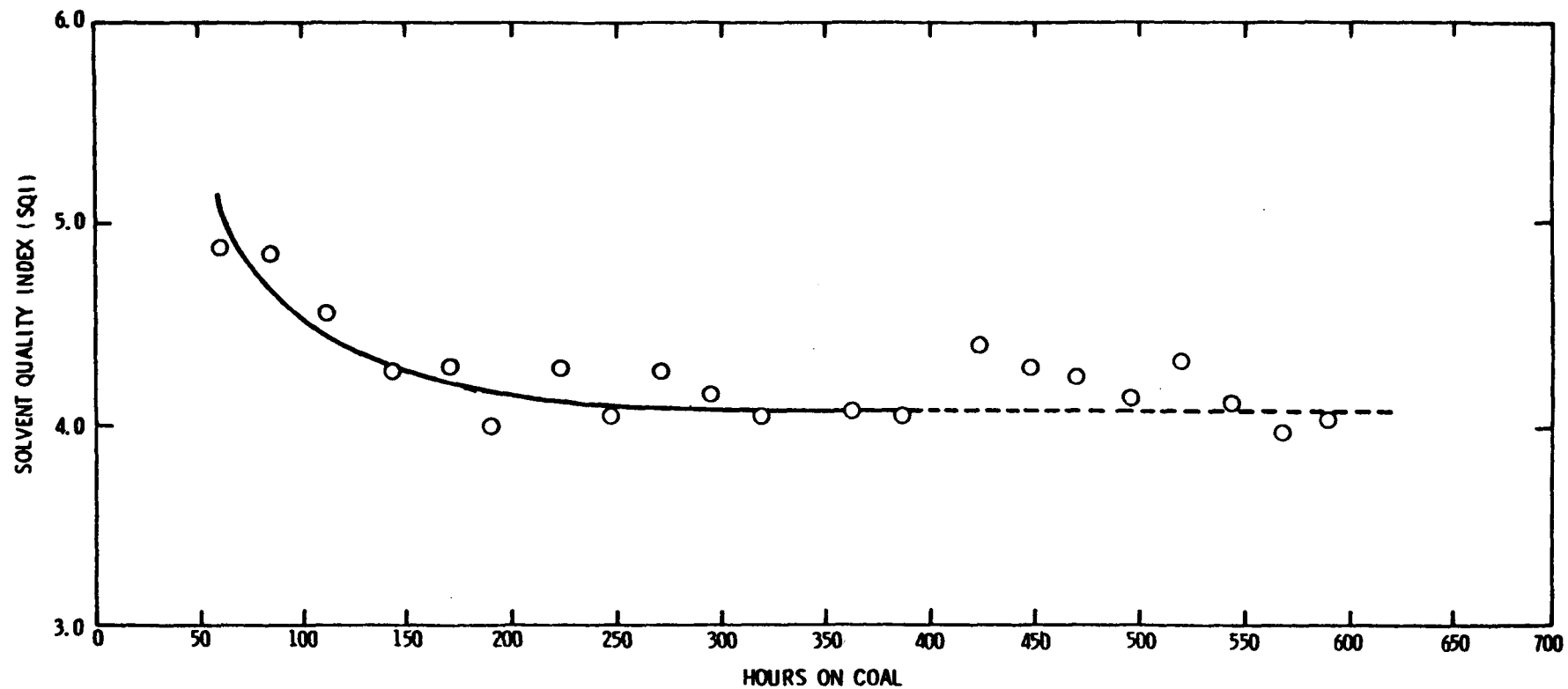


FIGURE 1-14

EFFECT OF UNIT RUN TIME ON RECYCLE
SOLVENT QUALITY USING Ni/Mo-10 CATALYST



● Conversion and Yields

The average yields from the Ni/Mo-10 catalyst run are presented in Table 1-5. No significant effect on Illinois No. 6 coal liquefaction yields is apparent due to changing from Co/Mo-10 catalyst to the more active Ni/Mo-10 catalyst.

The 1000°F conversion and C₄-1000°F liquid yield for the Ni/Mo catalyst test run are shown in Figures 1-15 and 1-16 as a function of unit run time. Both conversion and liquid yield remained essentially constant throughout the run. Data from an earlier Co/Mo-10 catalyst run with Illinois No. 6 coal are also shown for comparison. This run with Co/Mo-10 catalyst lasted about 300 hours. These data are included as part of the Co/Mo-10 results in Table 1-5. Over this 300 hour run, the Co/Mo-10 conversion and liquid yield were also constant and in excellent agreement with the Ni/Mo-10 catalyst results.

TABLE 1-5

EFFECT OF HYDROTREATER CATALYST ON
ILLINOIS NO. 6 COAL LIQUEFACTION YIELDS

<u>Hydrogenation Catalyst</u>	Co/Mo-10	Ni/Mo-10
<u>Liquefaction Conditions</u>		
Temperature	840°F	840°F
Pressure	1500 psig	1500 psig
Nominal Slurry Residence Time	40 min.	40 min.
<u>Hydrotreating Conditions</u>		
Temperature	173°F + RV	122°F + RV
Pressure	1500 psig	1500 psig
Space Velocity	1.51 x RV	2.59 x RV
<u>Solvent Quality Index</u>	3.9	4.1
<u>Yields, Wt% on Dry Coal</u>		
Hydrogen	-3.9	-4.2
Water	9.3	9.4
C ₁ -C ₃	6.8	7.6
C ₄ -1000°F	35.4	33.6
1000°F ⁺	47.0	48.3
1000°F Conversion	53.0	51.7

FIGURE 1-15

EFFECT OF HYDROTREATER CATALYST ON
ILLINOIS NO. 6 COAL CONVERSION

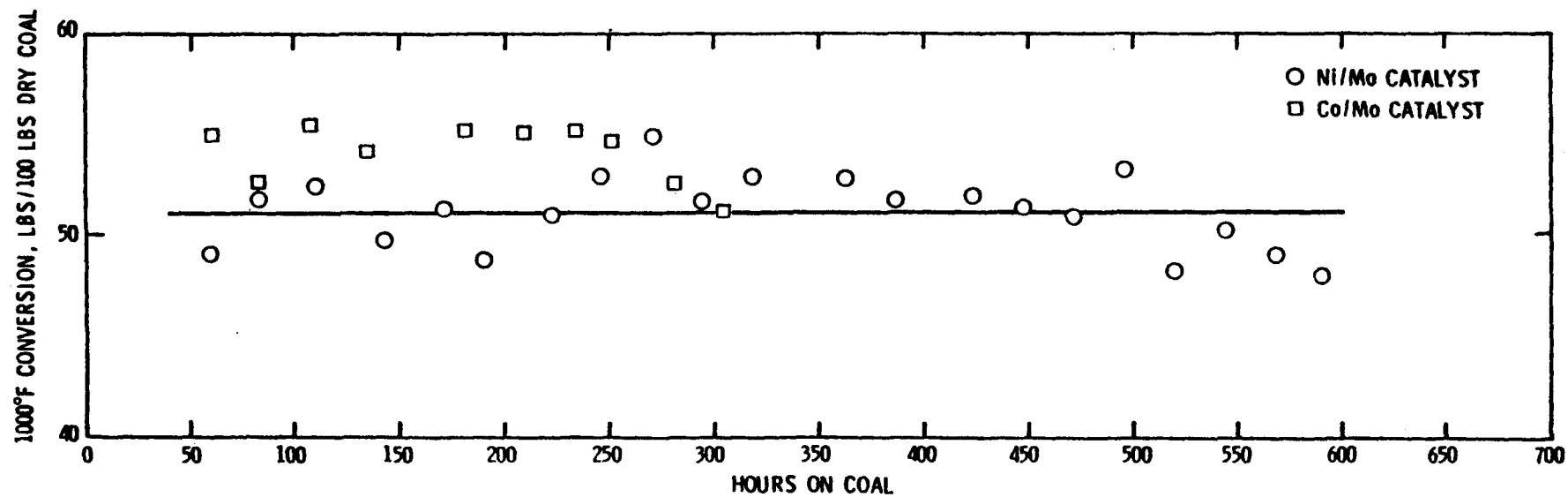
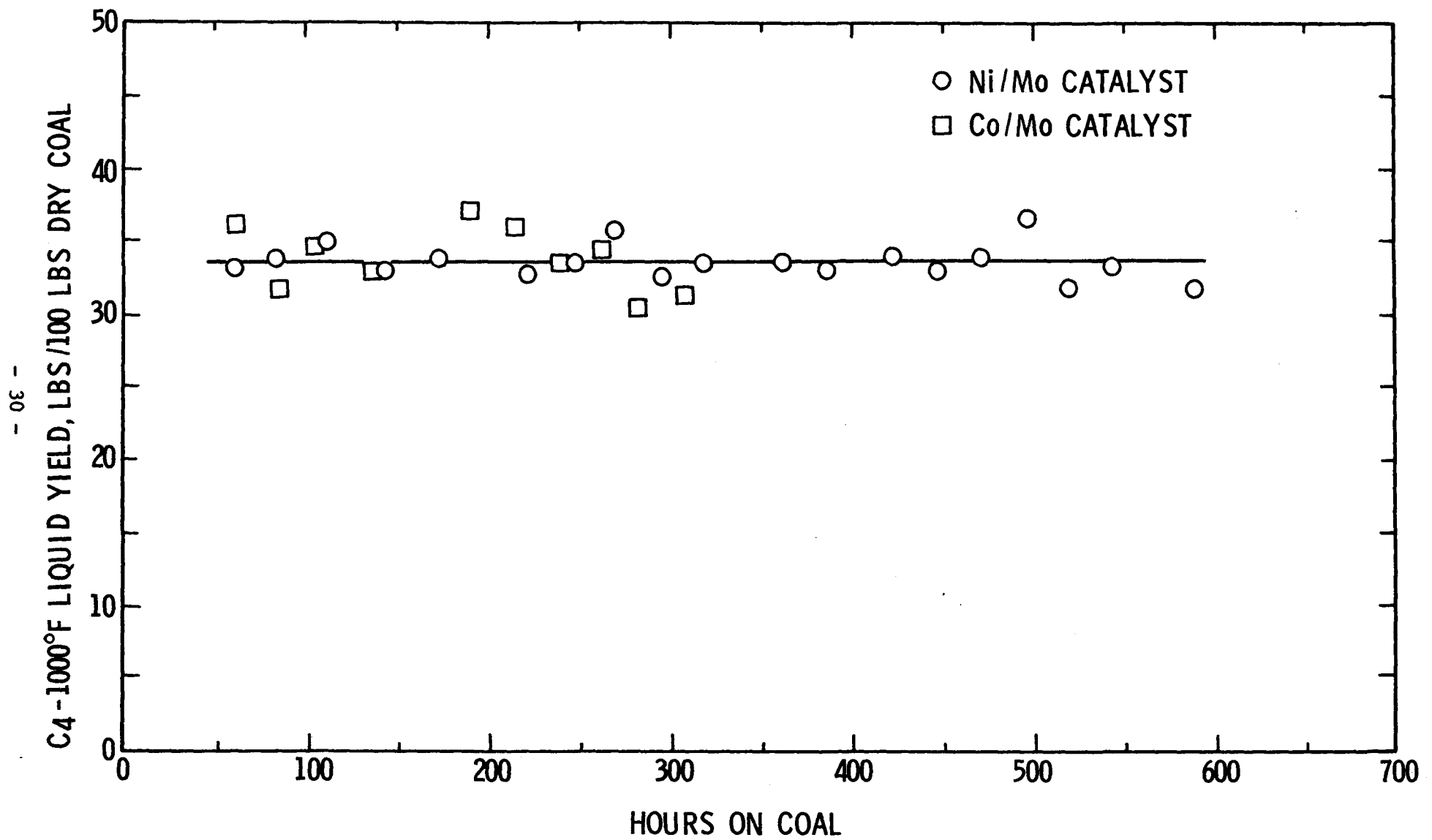


FIGURE 1-16

EFFECT OF HYDROTREATER CATALYST ON ILLINOIS NO. 6 LIQUID YIELD



1.6 Pittsburgh No. 8 Bituminous Coal Screening Study

Pittsburgh No. 8 bituminous coal from the Ireland mine is the first of several sponsor-selected coals being evaluated in a screening study in RCLU. The purpose of this limited study was to determine the response of Pittsburgh coal in the EDS liquefaction process. Liquefaction yields and unit operability were investigated as a function of temperature and residence time.

● Study Conditions

The effects of liquefaction temperature and residence time on Pittsburgh coal yields were investigated at 840°F and 40, 60, and 100 minutes residence times and at 800°F/100 minutes residence time. Other conditions were 1500 psig, four weight percent hydrogen treat rate, and 1.6:1 recycle solvent/coal ratio.

● Unit Operations

Four runs were made with Pittsburgh coal from the Ireland mine. Run lengths of 134, 160, 169, and 248 hours on coal were logged. The 134 and 248 hour runs were ended voluntarily. The other two runs were terminated due to mechanical problems unrelated to the coal type. A total of fourteen yield periods were completed during the study.

Operations were extremely smooth at all study conditions, and comparable to those experienced with Illinois No. 6 bituminous coal. No plugging problems were experienced in the liquefaction reactor or the residual bottoms accumulator. Withdrawal of residual bottoms from the accumulator was easily accomplished because of the low bottoms viscosity. For the conditions studied, the viscosities of the residual bottoms from Ireland coal were about three poise (550°F, 10 sec⁻¹ shear rate). These viscosities are comparable to Illinois No. 6 residual bottoms viscosity of about eight poise over the same range of conditions.

● Conversion and Yields

The Pittsburgh No. 8 bituminous coal yields are presented in Table 1-6 and compared to those for Illinois No. 6 bituminous coal in Figures 1-17 through 1-20. At all conditions studied, the conversion and liquid yield for Pittsburgh coal are significantly less than those for Illinois coal at equivalent liquefaction conditions. Pittsburgh coal is more difficult to convert than Illinois coal and a longer residence time is required to obtain the same degree of conversion at both 800°F and 840°F. However, at equivalent conversions, the C₄-1000°F liquid yields are only slightly lower for Pittsburgh coal.

TABLE 1-6

PITTSBURGH (IRELAND) COAL YIELDS

<u>Yield Periods</u>	511,512 513,514 515	516,517	518,519 520,521	522,523 524
<u>Operating Conditions</u>				
Reactor Temperature, °F	840	840	840	800
Pressure, psig	1500	1500	1500	1500
Nominal Residence Time, min.	40	60	100	100
Hydrogen Treat Rate, Wt% on Dry Coal	4	4	4	4
Solvent to Coal Ratio	1.6	1.6	1.6	1.6
Solvent Quality Index, SQI	4.1	4.6	4.4	4.4
<u>Overall Yields, Wt% on Dry Coal</u>				
Hydrogen	-3.1	-3.5	-4.6	-3.0
Water	5.3	5.3	6.0	6.0
Carbon Oxides	0.8	1.0	1.4	1.1
Ammonia	0.4	0.5	0.6	0.5
Hydrogen Sulfide	3.0	2.9	3.2	2.5
C ₁ -C ₃ Gas	6.9	8.2	13.5	5.7
C ₄ -1000°F Liquid	28.6	32.7	32.7	29.6
1000°F ⁺ Bottoms	58.1	52.9	47.2	57.6

At 840°F/100 minutes residence time, 1000°F conversion is still increasing for Pittsburgh coal, whereas conversion of Illinois coal has leveled off at these conditions. However, essentially all of this incremental conversion of Pittsburgh coal from 60 to 100 minutes residence time (see Figure 1-17) is due to increased C₁-C₃ gas make. The C₄-1000°F liquid make, as shown in Figure 1-18, did not increase.

C₁-C₃ gas yields are comparable for both coals, as shown in Figure 1-19. Chemical gas yields are also comparable for both coals over the range of conditions studied. Water yields are about four weight percent lower for Pittsburgh coal than those from Illinois coal at comparable conditions (July-September, 1977, EDS Quarterly Technical Progress Report, [FE-2893-3], Figure 1-6). This result is consistent with the difference in oxygen content in the two coals. The Pittsburgh coal used in RCLU had about four weight percent less oxygen (6.3 weight percent) than the Illinois coal (10.6 weight percent oxygen).

Overall hydrogen consumption is shown in Figure 1-20. The hydrogen consumption results parallel the conversion results. At comparable conditions, the Pittsburgh coal hydrogen consumption is lower than for Illinois coal.

The data also show that Pittsburgh coal yields at 800°F/100 minutes residence time are about the same as at 840°F/40 minutes residence time. This residence time/temperature yield response is similar to that experienced with Illinois No. 6 coal (January, 1976-June, 1977, EDS Final Technical Progress Report, [FE-2353-20]).

1.7 Future Plans

During the third quarter of 1978, RCLU-1 operations will be devoted to continued feed coal evaluation studies. Liquefaction of a Pittsburgh No. 8 bituminous coal from the Arkwright mine and of a Texas lignite (Big Brown mine) will be investigated. Both of these coals were selected by the sponsors as part of the coal screening program in RCLU.

RCLU-2 will remain shutdown concurrent with ongoing CLPP operations.

FIGURE 1-17

EFFECT OF LIQUEFACTION RESIDENCE TIME ON
CONVERSION OF PITTSBURGH (IRELAND) COAL

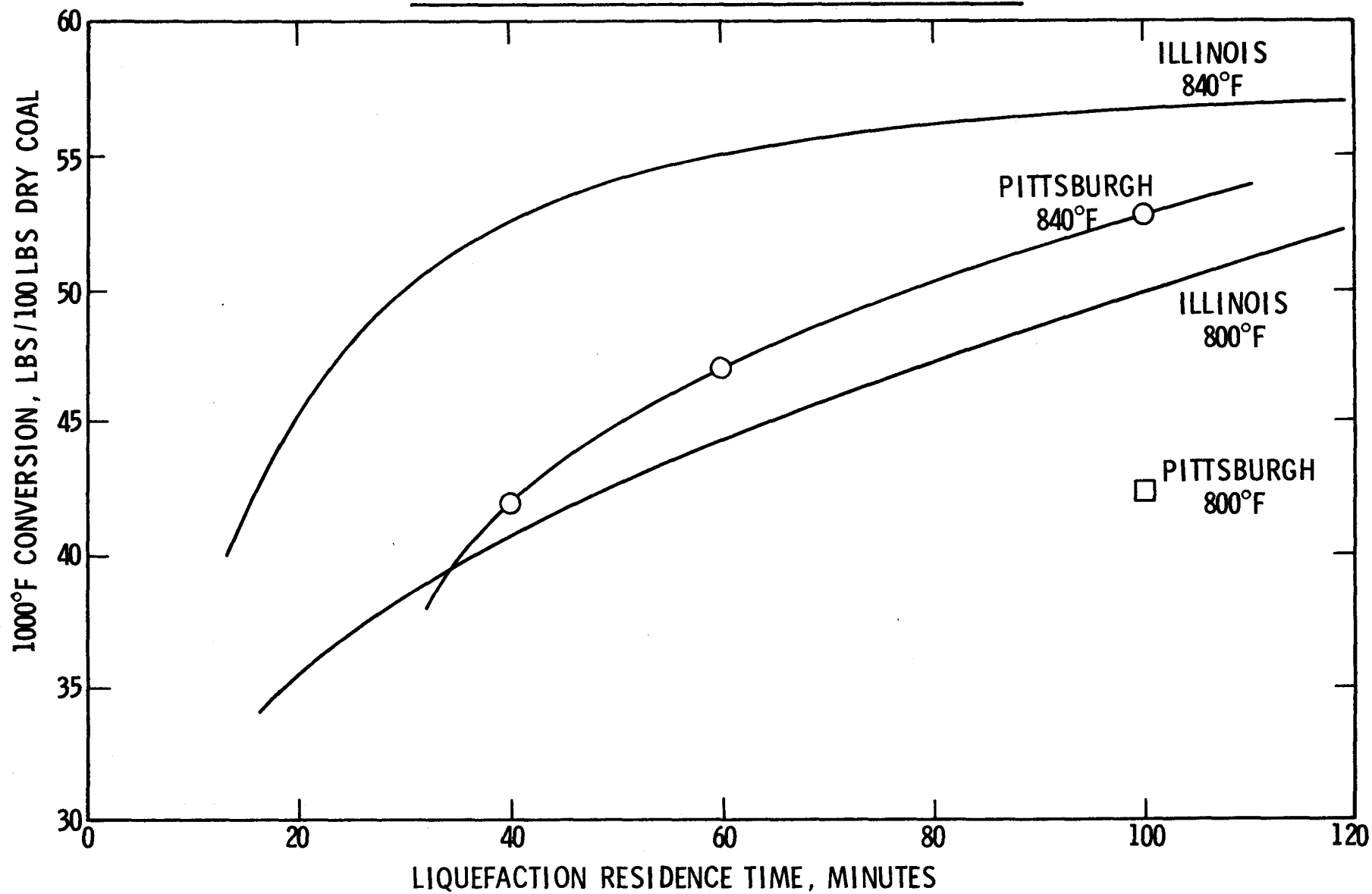


FIGURE 1-18

EFFECT OF LIQUEFACTION RESIDENCE TIME ON
PITTSBURGH (IRELAND) COAL C₄-1000°F LIQUID YIELD

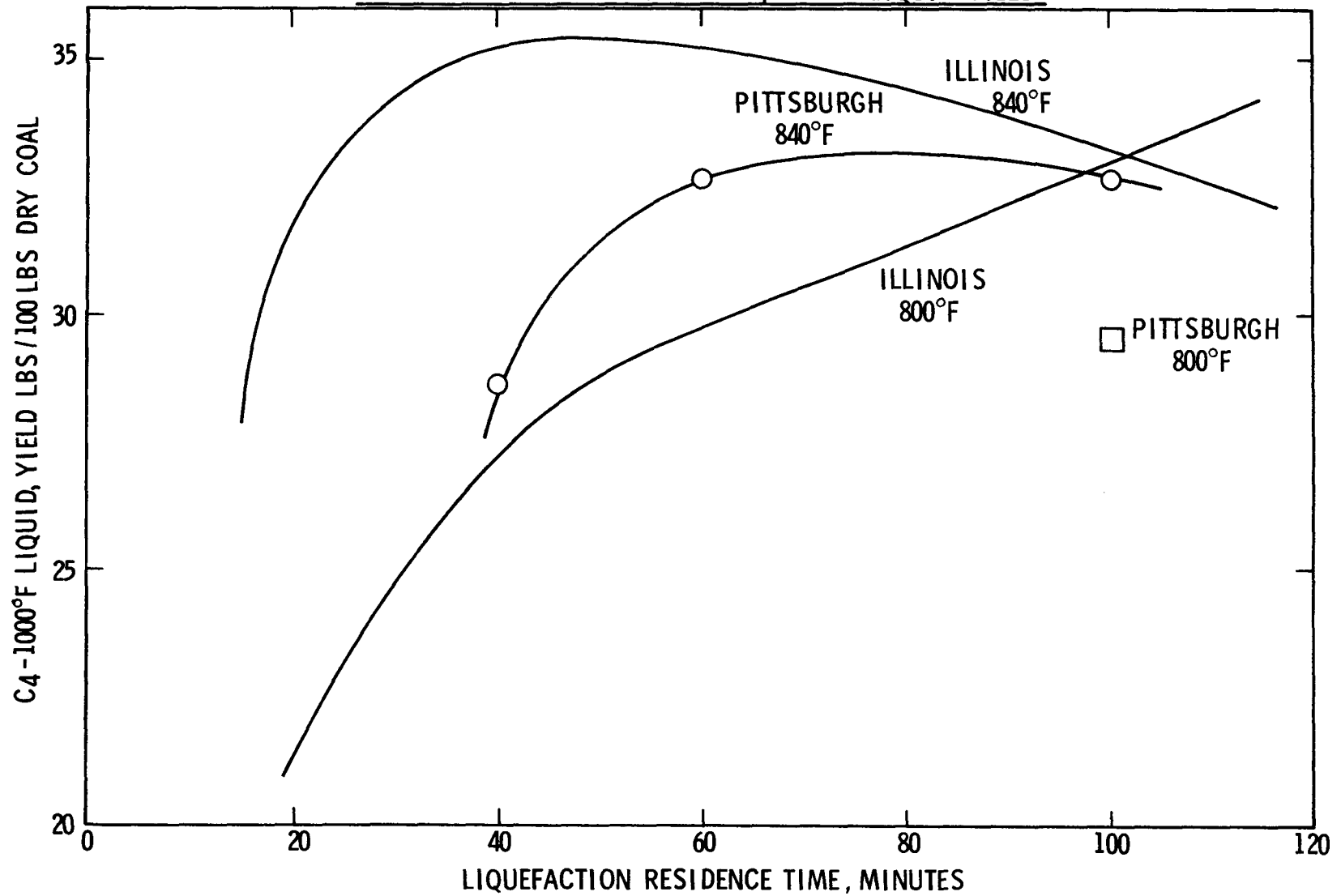


FIGURE 1-19

EFFECT OF LIQUEFACTION RESIDENCE TIME ON
PITTSBURGH (IRELAND) COAL C₁ - C₃ GAS YIELD

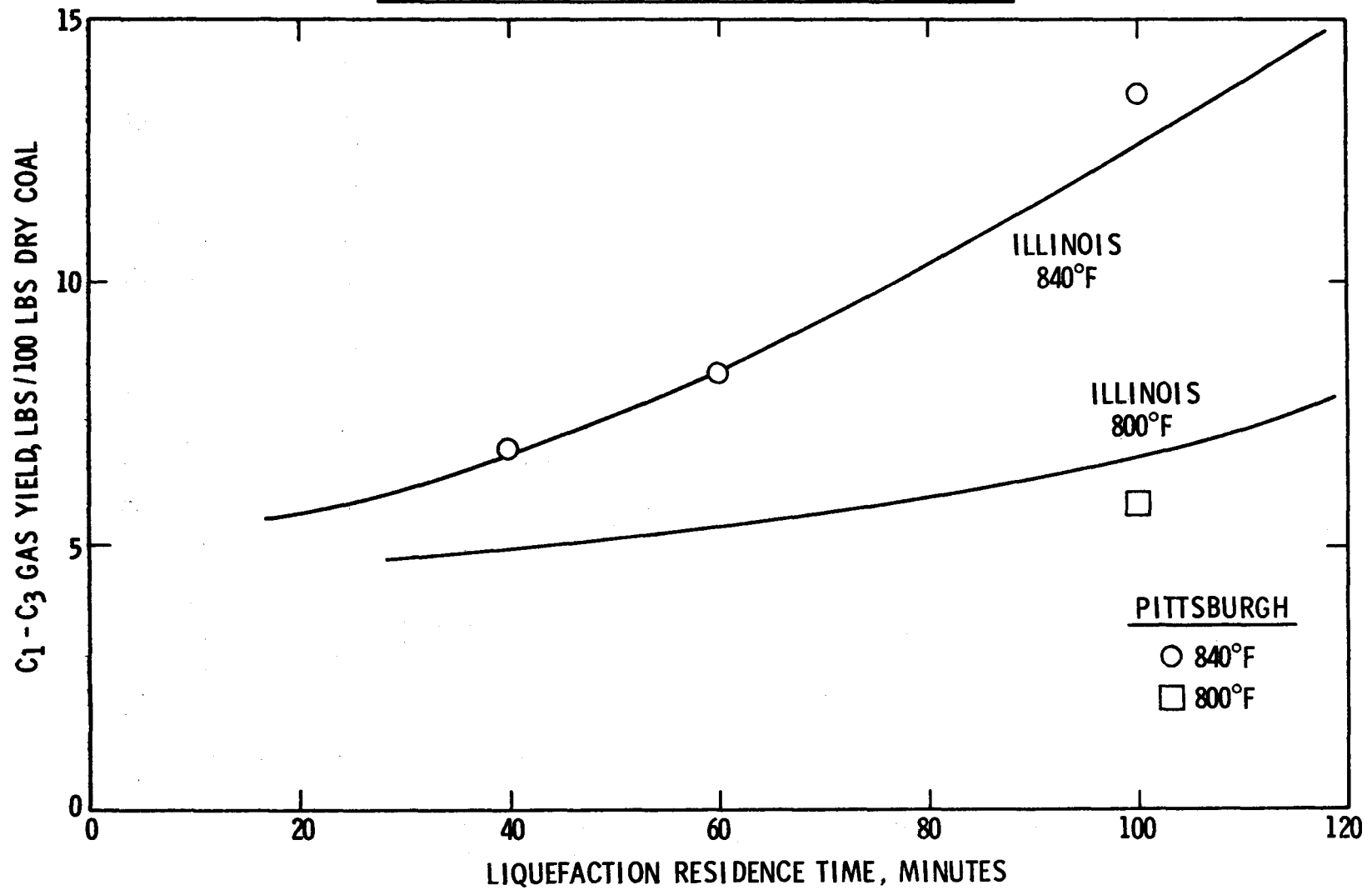
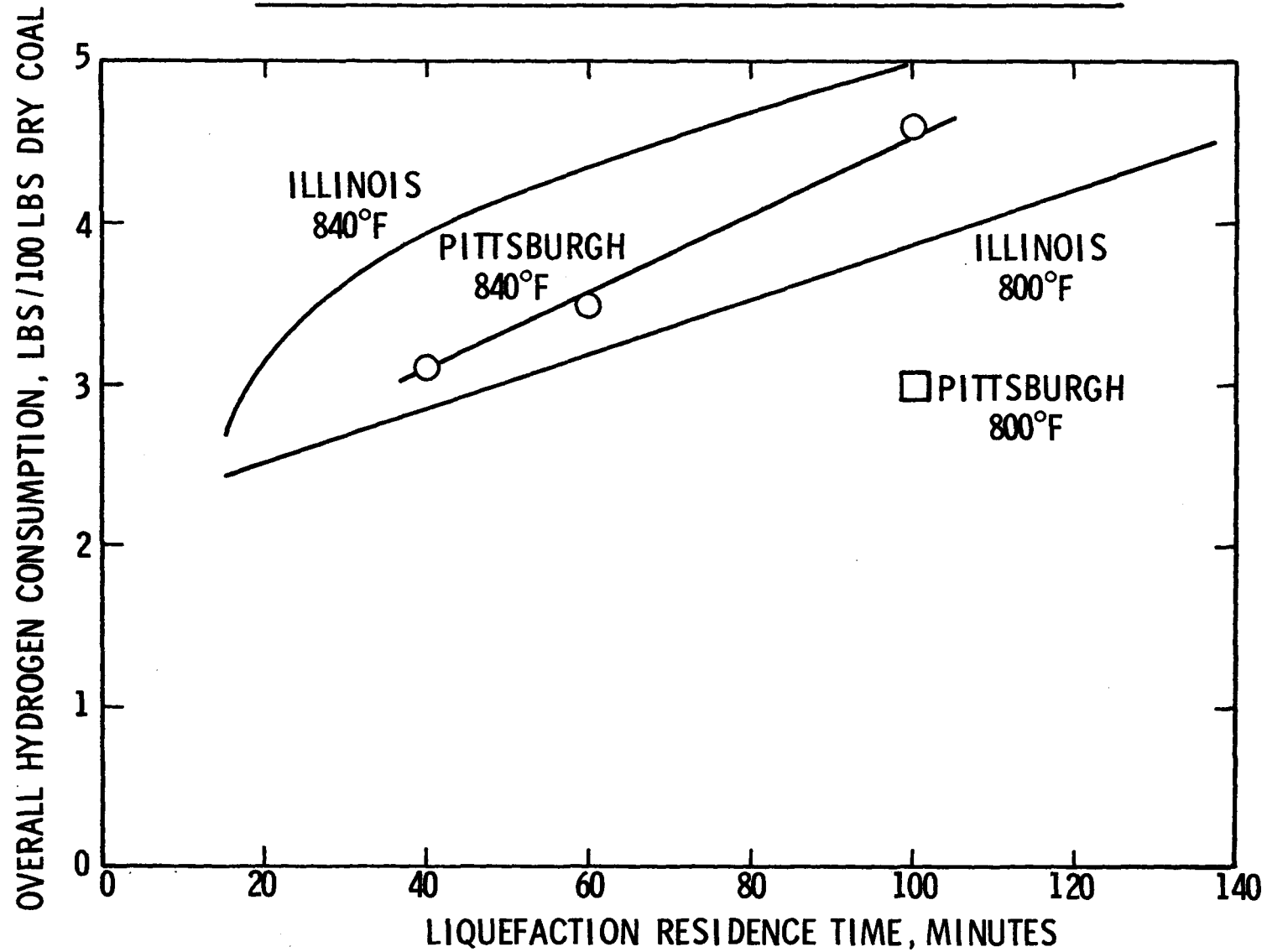


FIGURE 1-20

EFFECT OF LIQUEFACTION RESIDENCE TIME ON
PITTSBURGH (IRELAND) COAL HYDROGEN CONSUMPTION



LABORATORY PROCESS RESEARCH AND DEVELOPMENT

Liquefaction Process Research

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

The one ton-per-day Coal Liquefaction Pilot Plant (CLPP) was operated during the third quarter of 1977 on Wyodak and Illinois No. 6 feed coals. After a scheduled downtime of six months, the unit was brought up during the first quarter of 1978 on Wyodak coal at extended residence times. Wyodak operations have continued throughout the second quarter of this year. The basic process flow diagram of the unit is shown in Figure 2-1, and a detailed description of the CLPP is given in Appendix B.

CLPP was operated during the third quarter of 1977 on a Wyoming subbituminous coal and an Illinois No. 6 bituminous coal. The Wyoming coal study was completed by obtaining additional yield information at longer residence times. During these runs, three engineering studies were conducted. In the Wyoming coal run, a twin-screw pump with double mechanical seals was used as the vacuum tower bottoms pump. At the end of this run, slurry from the high temperature, low pressure separator (S-2) and bottoms from the vacuum flash tower (F-1) were fed to the CLPP slurry viscometer for viscosity measurements. Then, during the Illinois coal run the sectionalized preheaters were operated so that the slurry was maintained at 840°F for an extended residence time in the preheaters to study coking.

During the last quarter of 1977 (October only) CLPP was operated on Illinois No. 6 bituminous coal. This operation was to verify the Illinois No. 6 coal yield structure at 840°F and 40 min. nominal slurry residence time. These operating conditions will be used in the updated commercial study design. At the end of these operations, two additional studies were completed. First, the flash zone temperature of the vacuum flash tower was operated at higher than normal flash zone temperatures. Also, a special run was made to study the viscosity of the feed coal slurry preheated for short residence times.

During the first quarter of 1978, CLPP completed a scheduled five-month maintenance turnaround with a start-up in early March. No major configurational changes were attempted; however, improvements were made in several areas. Start-up operations began in the hydrotreating section to precondition a new catalyst and produce start-up solvent. Subsequently, a Wyoming subbituminous coal was introduced to the unit. Over 3000 lbs of Wyodak residual bottoms were produced for IKG operations during the preliminary shakedown period. Furthermore, it was determined that all unit modifications aimed at improving operability with Wyoming coal were successful. Yield period operations began on May 11, 1978. The primary emphasis of this series will be to pinpoint optimum operating conditions for Wyodak coal.

2.1 Confirmation of RCLU Data on Wyodak Coal

During July a run was made with a Wyoming subbituminous coal to expand the data base for this coal to longer residence times than those previously investigated on CLPP. At these longer residence times the temperature was varied from 800°F to 840°F to define the optimum operating conditions for producing maximum liquid yield. As a result of the operating techniques developed during the previous operability studies and the use of the reactor solids withdrawal system, operations during this run were made without any significant liquefaction reactor temperature excursions or loss of control of the vacuum tower bottoms viscosity. Five yield periods (YP 245 through 249) were made during an eight-day run. The detailed operating conditions, elemental balanced yields and product analyses for these yield periods are presented in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3). A comparison of the yields from these runs with comparable Recycle Coal Liquefaction Unit (RCLU) runs are presented in Figures 2-1 and 2-2. The RCLU data are represented by the solid lines and the CLPP yields are indicated by individual data points.

Total conversions from the CLPP yield periods are in good agreement with conversions from RCLU at equivalent conditions. However, the CLPP C_4^+ liquid yields are higher than RCLU liquid yields and the CLPP C_1 - C_3 gas yields are lower. These yield differences result from the fact that in CLPP only the recycle solvent stream is hydrotreated while in RCLU the entire liquefaction reactor liquid stream (which includes gas, water, naphtha, and solvent) is hydrotreated. When the liquefaction product stream is hydrotreated, its liquid content is decreased and its gas content is increased as a result of heteroatom removal and cracking reactions that occur concurrently with hydrotreating. The RCLU yields of water are subsequently higher due to the deoxygenation. Since liquid products from the liquefaction of Wyoming coal have high oxygen contents, these hydro-treating side reactions did effect differences in the liquid and gas yields from these two units.

2.2 Yield Confirmation at Updated Study Design Conditions

During the fourth quarter of 1977 (October), CLPP was operated at a set of conditions for the updated commercial study design selected by the Exxon Donor Solvent (EDS) Process Alternatives Model (PAM). These conditions are a liquefaction temperature of 840°F and a nominal slurry residence time of 4.8 x RV hours.

The final data inspection and elemental balances were completed on eight yield periods at these conditions, and the average results are presented in Table 2-1. Detailed yields and analytical data are presented in the October-December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7).

FIGURE 2-1

COMPARISON BETWEEN RCLU AND CLPP YIELDS

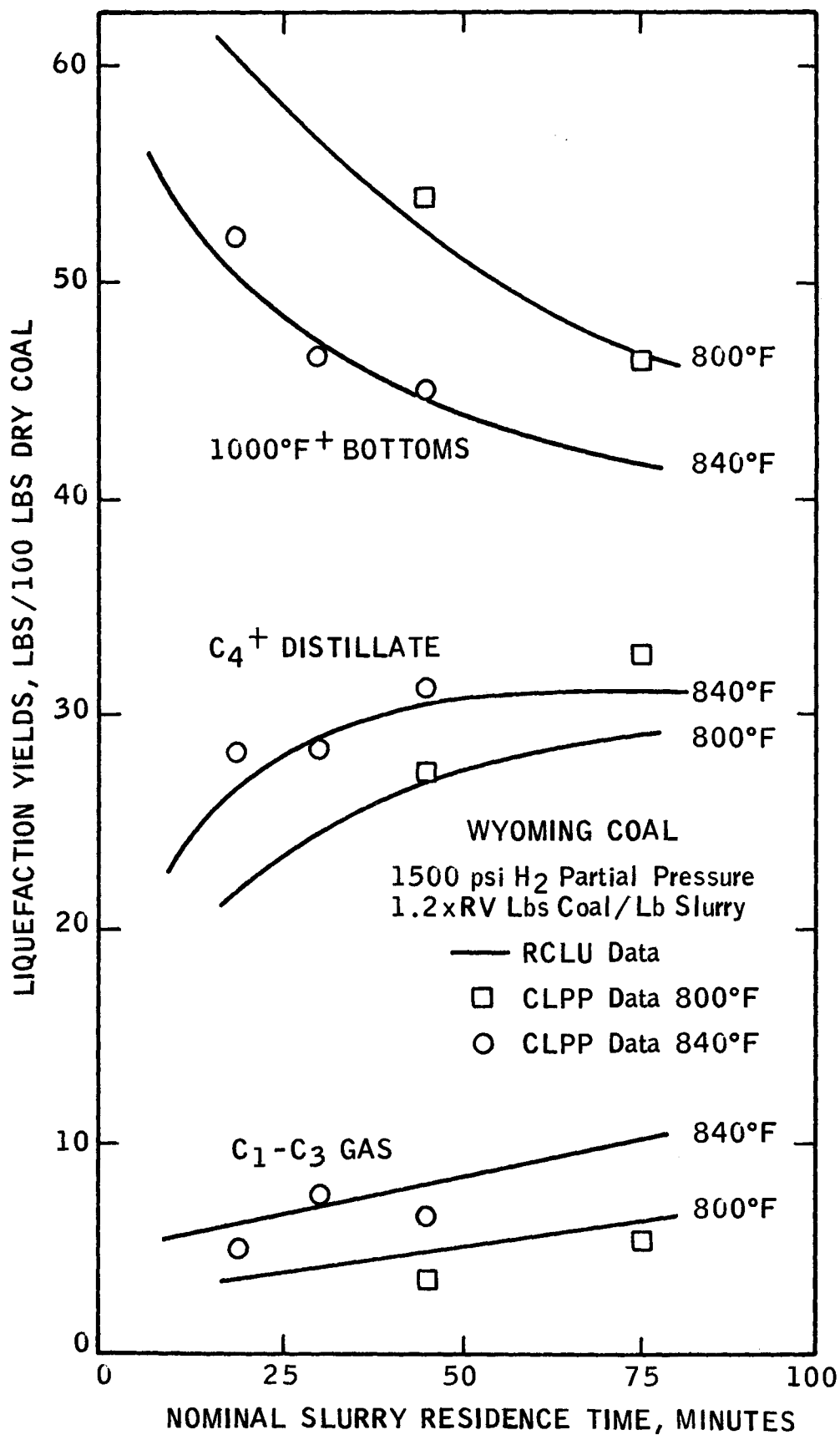


FIGURE 2-2

COMPARISON BETWEEN RCLU AND CLPP YIELDS

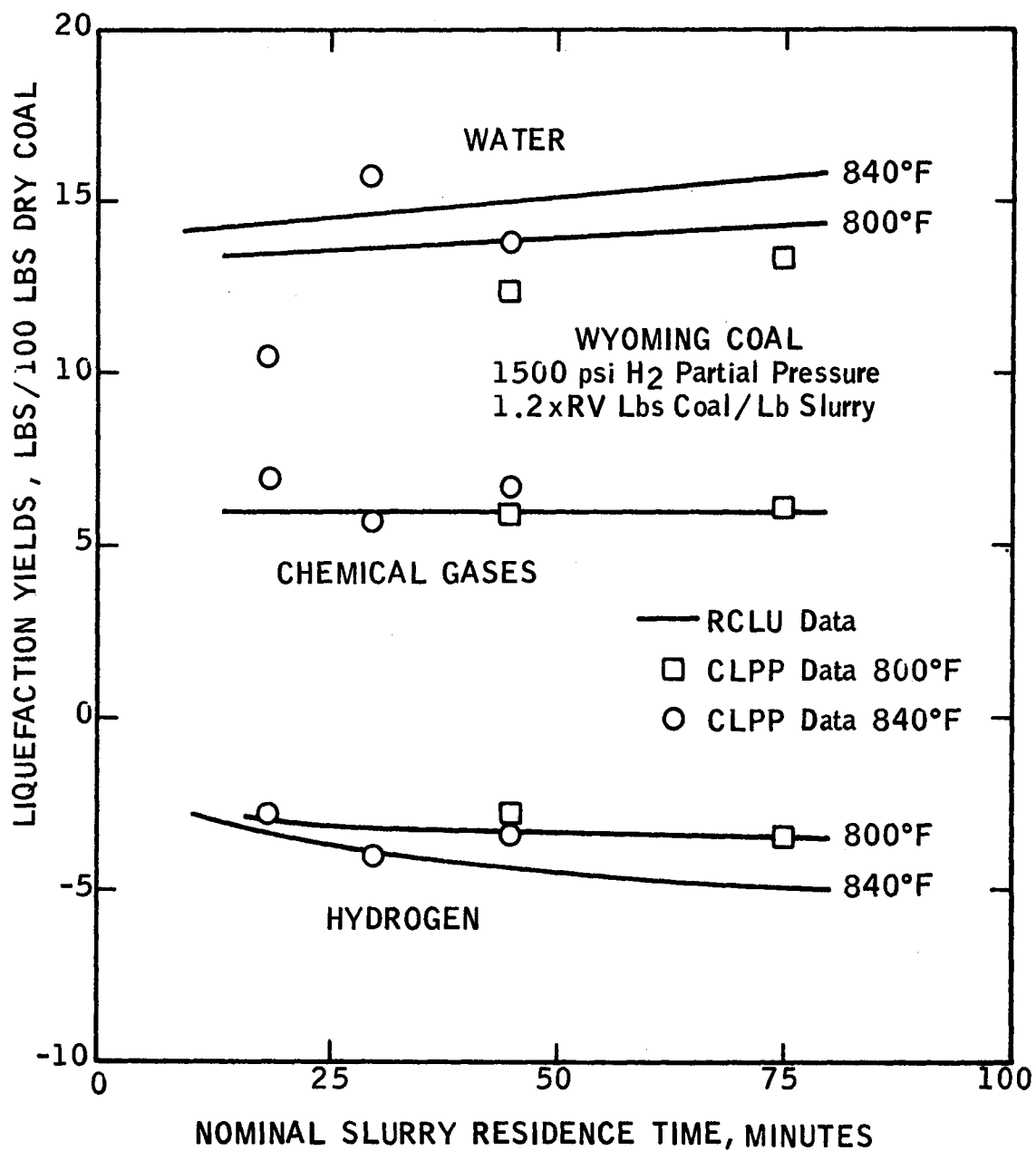


TABLE 2-1

AVERAGE CLPP ELEMENTAL BALANCED YIELDS

840°F

40 minutes Nominal Slurry Residence Time

2.8 Solvent Quality Index

11.3 wt.% Ash in Feed Coal

Overall Yields, Lbs/100 Lbs Dry Coal

H ₂	-3.3
H ₂ O	8.9
CO, CO ₂	1.4
NH ₃	0.4
H ₂ S	3.2
C ₁ -C ₃ Gas	6.6
C ₄ -400°F Naphtha	18.3
400-700°F Middle Distillate	7.6
700°F+ Heavy Distillate	7.2
1000°F+ Residual Bottoms	49.7

The results from this series form the basis for the update commercial study design now in progress. These yields form part of the basis for the commercial plant study design update done by the Synthetic Fuels Engineering Department. When corrected to a typical 9.1 weight percent ash content in feed and to 3.0 Solvent Quality Index (SQI) basis, the C₄⁺ liquid yield increases to 34.6 weight percent (from 33.1 weight percent indicated above).

2.3 Engineering Studies● Vacuum Tower Bottoms Pump

During the Wyoming coal run of third quarter 1977, a twin-screw pump with double mechanical seals was used as the vacuum tower bottoms pump. This pump is the same type of pump that is being considered for the Exxon Coal Liquefaction Pilot Plant (ECLP) and could be scaled up to commercial size. This pump was operated successfully for eight days before a plug developed in the bottoms pumparound loop. The high pressures that resulted from the plug caused the pump's mechanical seals to fail.

Inspection of the pump showed no wear on the screws or in the base. Thus, the pumping components could have operated for considerably longer than eight days in this service. However, a close examination of the seals revealed that striations in the mechanical seal faces left from the manufacturing process may have been the cause of the seal failure. To improve future operations of this pump, either the mechanical seal will be redesigned or packing will be used in place of the seal.

- Viscosity Measurements of CLPP Process Streams

At the end of the third quarter 1978 Wyoming coal operations, a run was made to supply samples of S-2 bottoms and vacuum flash bottoms to the CLPP slurry viscometer. The viscosity of these streams was measured at temperatures up to 61°F and at different shear rates. This data is needed to define the shear rate sensitivity (Newtonian, non-Newtonian) of these streams and will be used in the design of ECLP.

- Sectionalized Preheaters

The sectionalized slurry preheater study has been expanded to determine the effects associated with extended slurry residence times. Earlier data has shown that coking is not a problem at 840°F with mixed phase (slurry plus hydrogen treat gas) flow and short residence time. However, since it is generally believed that coking is a strong function of residence time at a given temperature, studies during the third quarter were at preheater residence times twice as long as those previously obtained.

Two runs were completed on Illinois No. 6 coal at a long preheater residence time; the first run lasted 48 hours and the second operated 170 hours before plugging. Since, during both of these runs, solvent to coal ratios varied considerably due to operational problems with the slurry mixing vessel, these runs represent a severe test of the preheater system. While it was not possible to define the exact location of the plug in either run, there was no evidence of coke formation in the preheaters. The preheater coil deposits were very soft and wet, which is quite surprising considering the long period the coils were held at high temperature after flow stoppage. These observations support operating evidence that this stoppage probably was external to the sectionalized preheater coils.

- High Severity Vacuum Flash Studies

A series of high severity tests were completed on CLPP to study high temperature operations of the vacuum flash section. These runs were with Illinois No. 6 coal and base case liquefaction conditions. Flash zone temperatures in the primary vacuum tower (F-1) as high as 774°F were achieved. During the highest temperature operations, the vacuum flash preheater (E/H-4) was maintained at 840°F. For normal CLPP operations the flash zone temperature is about 630°F and E/H-4 is at 675°F. The F-1 tower used was twelve inches in diameter with a one-inch diameter transfer line. Five tests were successfully finished. Each test except the last one was longer than two hours. While coming on conditions for test No. 6, the pumparound section of the vacuum tower plugged and the test was aborted.

The conditions for all six tests are summarized in Table 2-2. Weight percent 1000°F percent of vacuum tower bottoms obtained during these tests and those predicted by an empirical equation are included in the same table. The detail of this empirical relationship was included in the January-December, 1976, EDS Annual Technical Progress Report (FE-2353-9).

TABLE 2-2

RUN CONDITIONS FOR HIGH SEVERITY VACUUM FLASH STUDY

Test No.	Flash Zone Temperature, °F	Pressure, psia (mm Hg)	Effective Flash Zone Temperature**, °F	Wt% Microlube 1000°F in Bottom	
				Predicted	Experimental
1	675	0.80 (41)	905	3.6	2.7
2	681	1.22 (63)	885	4.5	4.5
3	725	1.08 (56)	940	2.1	1.1
4	735	0.85 (44)	966	1.1	0.6
5	774	1.18 (61)	982	0.5	0.5
6*	775	0.80 (41)	1008	-	-

*Pumparound loop plugged during test #6.

**These temperatures were calculated using characterization factor, K = 9.5.

● Slurry Viscosity Measurements

After the completion of the high severity vacuum flash tests, a special run was made to obtain viscosity measurements of the Illinois No. 6 coal slurry as it exits the liquefaction feed preheaters. Previous measurements of this slurry viscosity were made by heating a batch of slurry to reactor conditions (about 800°F) over a period of hours. Results of these slow heating runs indicated a pronounced viscosity peaking (due to Illinois coal swelling and gelation) in the 600-700°F temperature range. This special CLPP run was made to determine if this viscosity peaking depended on the preheating time.

For this run the CLPP sectionalized slurry preheaters were configured to provide slurry to the CLPP slurry viscometer at temperatures ranging from 550 to 815°F, with residence times comparable to those predicted for the Exxon Coal Liquefaction Plant (ECLP) slurry preheaters. The slurry was then heated to 840°F before entering the liquefaction reactor at 1600 psig. All slurry preheating was done in a separate phase mode (i.e., without hydrogen treat gas) to facilitate interpretation of viscometer data. Preliminary data indicate that the viscosity peaking did occur in the 600 to 645°F range. More detailed data analysis from this run will be reported in the Engineering Technology Development section.

2.4 Product Sample Generation

One of the continuing objectives of CLPP operations is to produce product samples for product quality and end-use testing and downstream processing studies.

During third quarter 1977, these samples were generated from operations on Illinois No. 6 bituminous coal. All of the liquid products (naphtha, solvent, and heavy vacuum gas oil) generated during operations in this quarter were unhydrotreated. A total of about 30,000 pounds of material was produced.

After the five-month unit turnaround, unit shakedown began on Wyodak subbituminous coal. During this period early in the second quarter of 1978, CLPP produced over 3000 pounds of low viscosity vacuum tower bottoms necessary for IKG operations in Baton Rouge.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

LIQUEFACTION PROCESS RESEARCH

3. Solvent Hydrogenation Studies

During this reporting period, EDS solvent hydrogenation studies concentrated on long-term catalyst performance and focused on activity maintenance as well as hydrogenation kinetics at near end-of-run conditions. In addition, several catalyst activity scoping studies were conducted with different potential feedstocks. The effect of carbon oxide impurities in hydrogen treat gas on catalyst activity was also investigated. A summary of solvent hydrogenation studies conducted during this reporting period is shown in Table 3-1.

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 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 Description of Experimental Studies

The major objectives of the spent solvent hydrogenation studies conducted during this period were:

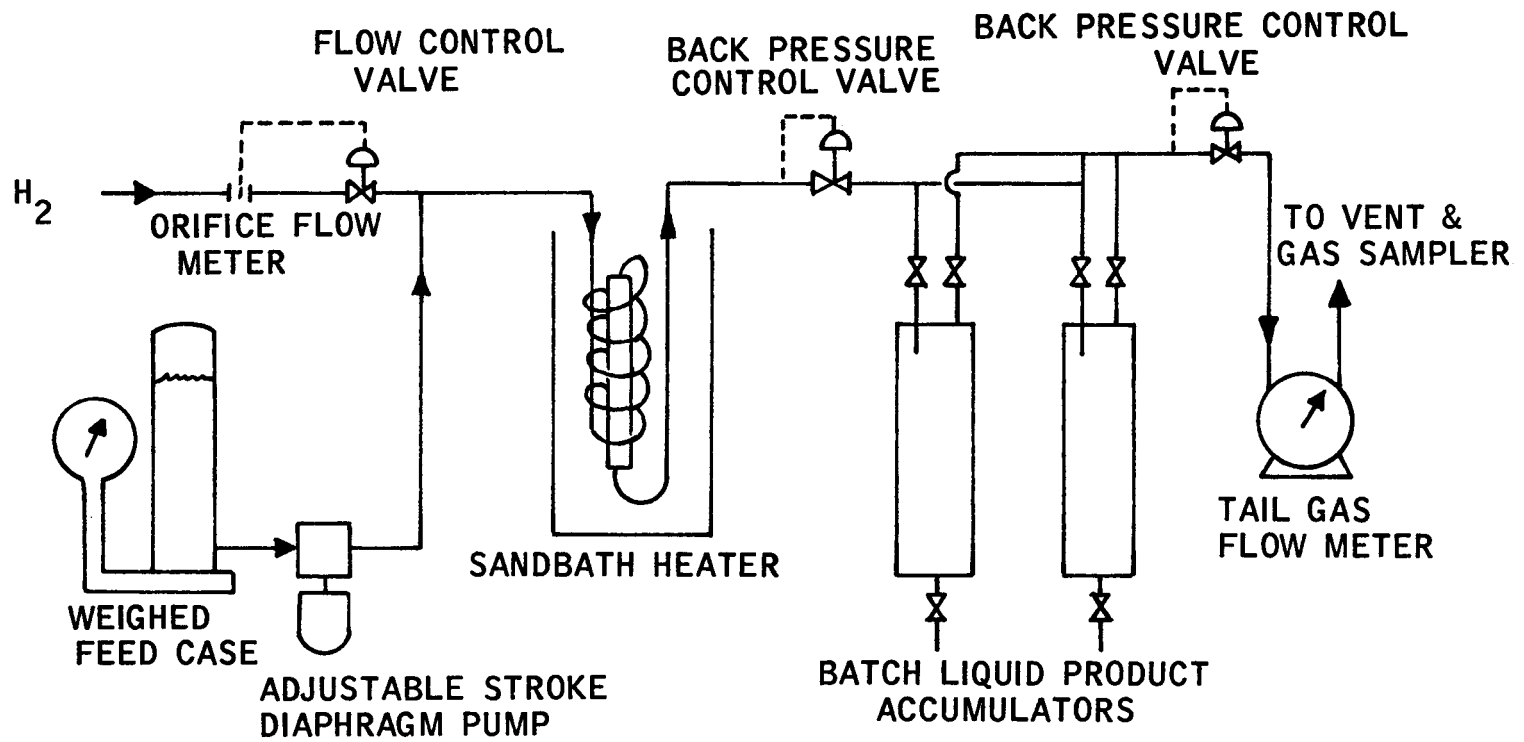
TABLE 3-1

SUMMARY OF SOLVENT HYDROGENATION STUDIES

<u>Run</u>	<u>Catalyst</u>	<u>Feed</u>	<u>Reactor Size, ML.</u>	<u>Temperature-RV °F</u>	<u>Pressure Psig</u>	<u>Duration Days</u>	<u>Purpose of Run</u>
10M22 A	Ni/Mo-10	Hvy. Solvent	10	115-215	1500	24	Catalyst activity screening
B	Co/Mo-20	Hvy. Solvent	10	115-215	1500	24	Catalyst activity screening
11M22 A	Ni/Mo-10	Wyo. Solvent	10	65-215	1500	39	Catalyst activity screening
B	Co/Mo-20	Wyo. Solvent	10	65-215	1500	39	Catalyst activity screening
12M22 A	Ni/Mo-10	Ill. Solvent	10	115-215	1500	50	Carbon oxide impurity study
B	Ni/Mo-10	Ill. Solvent	10	115-215	1500	50	Carbon oxide impurity study
13M22 A	Ni/Mo-10	Ill. Solvent	10	115-215	1500	In Progress	Carbon oxide impurity study
B	Ni/Mo-10	Ill. Solvent	10	115-215	1500	In Progress	Carbon oxide impurity study
14M21	Ni/Mo-10	MPSS	70	145-197	1500	311	Activity maintenance study
20M20	Ni/Mo-10	Hvy. Solvent	70	115-215	1500	19	Catalyst activity screening Catalyst contact study
21M20	Ni/Mo-10	Ill. Solvent	20	65-215	1500	32	Catalyst activity screening

FIGURE 3-1

SIMPLIFIED FLOW DIAGRAM OF BENCH SCALE HYDROTREATERS



- to determine the deactivation rate of Ni/Mo-10 catalyst at conditions more severe than assumed for the commercial study design.
- to assess catalyst performance at the end-of-run temperature range for Ni/Mo-10 catalyst
- to examine the effects of heavy spent solvent on initial catalyst activity
- to compare the performance of fresh Ni/Mo/Al and Co/Mo/Al catalysts with Wyoming spent solvent
- to determine the effect of treat gas impurities on catalyst activity.

The results are discussed in Section 3.3 through 3.7 of this report. The data obtained from the studies completed during the first nine months are reported in detail in previous technical progress reports. Data on the catalyst hydrogenation studies completed recently, designated as 12M22, 13M22 and 14M21, are contained in Appendix C. These tables contain the catalyst charge, solvent used, process operating conditions and product analyses for each study.

3.3 Catalyst Activity Maintenance Study

A catalyst activity maintenance study with Ni/Mo-10 catalyst was completed. The purpose of this study was to determine the deactivation rate at more severe start-of-run (SOR) conditions, and to investigate catalyst performance at the end-of-run (EOR) temperature conditions. Higher SOR temperature and a space velocity approximately 50% greater than the commercial study design value were used in this study. Multi-pass spent solvent (MPSS) produced from Illinois coal in the one ton-per-day Coal Liquefaction Pilot Plant (CLPP) served as solvent feedstock.

The experimental data indicated that the catalyst activity had declined to approximately 50% of initial activity at the termination of the run. The data also showed that constant solvent product quality had been maintained throughout the run. This should translate into a catalyst life of one year at commercial study design conditions, but a more definitive projection at design conditions is planned using EOR data from this maintenance study to extend the scope of solvent hydrogenation modeling.

Except for a brief interruption caused by a plug inside the preheat section, continuous operation of 311 days-on-oil had been logged at

the end of the run. A restriction developed inside the preheat section of the unit (on day 162); a complete plug occurred when an attempt was made to purge the restriction. The run was successfully continued after replacing the plugged section. The cause of the plug has been identified as deposits of iron sulfide.

The mechanism for formation of the iron sulfide is not clear. Soluble iron compounds and the sulfur in the solvent may react with each other in hydrogen-rich system when the mixture of the hydrogen treat gas and liquid feed enters the hot preheat section. Reaction would occur in the vapor phase resulting in the formation of insoluble salt which deposits on the inside surface of the preheat section.

The interruption discussed above occurred during operation in the projected EOR temperature range. Therefore, the run was continued at constant temperature not only to determine if the catalyst had been affected during the brief feed-out interruption but for the remainder of the run to define activity decline in terms of a change in product quality rather than a temperature increase to maintain constant product quality.

The reactor temperature was maintained at RV+191°F (i.e., the temperature prior to interruption) for approximately 50 days-on-oil. No significant changes in either solvent donor hydrogen content or heteroatom (sulfur, nitrogen, oxygen) removal level was observed during this period, indicating only a negligible decline in catalyst activity during this constant temperature operation. Figure 3-2 shows the catalyst age (weeks-on-oil) versus the reactor temperature and the corresponding product quality in terms of solvent donor hydrogen content through the life span.

Two separate process variable studies were conducted during the run following the interruption. The purpose was to test whether a "broad-based" activity parameter (i.e., a fractional multiplier on each of the hydrogenation rate constants used to model fresh catalyst performance) could be used to describe activity or whether selectivity changes had occurred, requiring more extensive modelling of the kinetics with aged catalyst. Since activity is an intrinsic property of the catalyst, its value should be independent of the process variables at which it is determined.

During the two-week test period the first process variable study revealed that catalyst activity had declined to approximately 65% of initial activity at RV+190°F and to approximately 50% of initial activity at RV+165°F (Figure 3-3). In Figure 3-3, the experimental data are plotted as donor hydrogen versus space time. The solid lines represent model predictions at various levels of activity relative to fresh catalyst. This temperature dependence of relative activity was neither expected nor understood. However, closer examination of the test conditions revealed that the results may have been influenced by decreased treat gas rate due to a leak in the system. The second process variable study was conducted to verify this observation.

FIGURE 3-2

MPSS-Ni/Mo CATALYST ACTIVITY MAINTENANCE STUDY

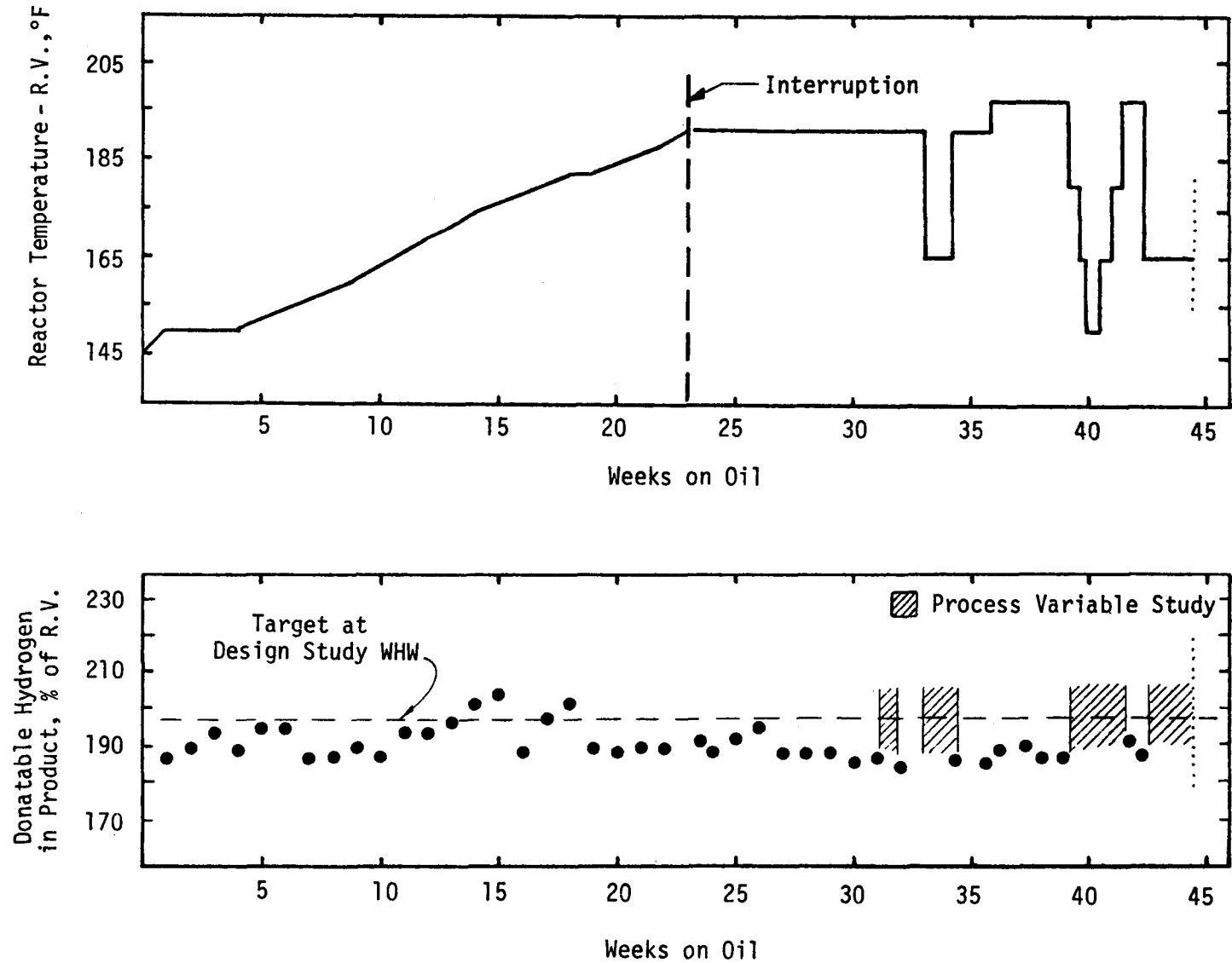
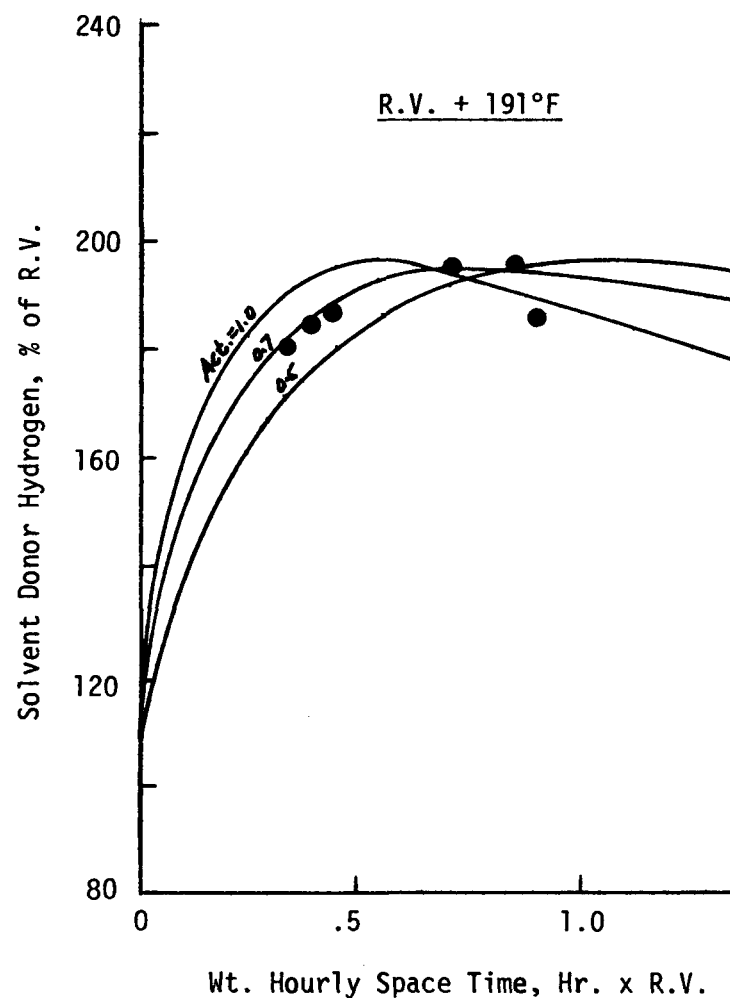
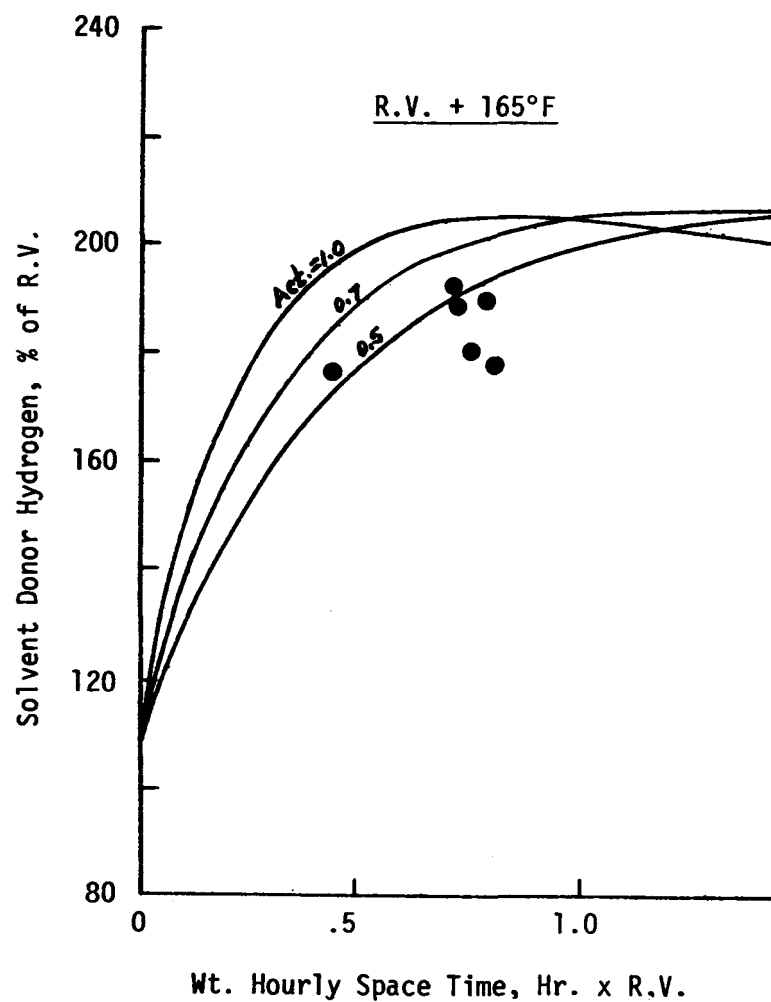


FIGURE 3-3

CATALYST ACTIVITY AS FUNCTION OF DONOR HYDROGEN AND SPACE TIME

Feed: MPSS
Catalyst: Ni/Mo-10
Age: 32-34 weeks



This study was also carried out at various space velocities and over a wider temperature range. The experimental data indicated that catalyst activity had declined to approximately 50% of initial activity at all temperature levels (Figures 3-4 and 3-5), including tests at the previous temperatures.

This catalyst activity maintenance was voluntarily terminated after the second process variable study. Inspections on the discharged catalyst are in progress to identify the primary deactivation mechanism.

3.4 Effect of Heavy Solvent on Catalyst Activity

Catalyst activity scoping studies were conducted with a heavy boiling feedstock. These studies tested the effects of a potential heavier spent solvent on initial catalyst activity. Properties of the heavy solvent, prepared by blending vacuum gas oil (CLPP A-2 liquid) with MPSS solvent, are shown in Appendix Table C-1.

Fresh Ni/Mo-10 and Co/Mo-20 catalysts were evaluated in the two-train catalyst testing unit at different space velocities and operating temperatures. The results from this study and previous MPSS studies are compared in Figures 3-6 and 3-7. Both figures show the solvent donor hydrogen content of the hydrogenated product at various operating temperatures as a function of space time.

Results from heavy solvent study show that with both catalyst SOR temperatures at least 50°F higher was required to reach the same levels of solvent donatable hydrogen as those required in the MPSS study. This temperature increase is presumably due to the presence of greater amounts of three and four-ring aromatics and/or the necessity of generating a larger fraction of donor hydrogen from these compounds.

An additional catalyst activity scoping study was performed because of concern that use of the heavy solvent might result in inadequate contact in the small (10 ml. reactor volume) two-train unit. Verification of the catalyst performance was made in the standard bench scale hydrotreating unit (70 ml. reactor volume) for Ni/Mo-10 catalyst. Test results from the standard unit confirmed those obtained with the small two-train unit.

3.5 Effect of Wyoming Spent Solvent on Catalyst Activity

Both Ni/Mo-10 and Co/Mo-20 catalysts were utilized in a catalyst initial activity study for Wyoming coal-derived solvent. CLPP spent solvent produced from Wyoming coal was investigated to provide preliminary data on initial catalyst activity. Comparison with CLPP spent solvent from Illinois coal (MPSS) shows that the Wyoming spent solvent contains more saturated compounds, but a smaller fraction of heteroatom (sulfur, nitrogen, oxygen) compounds (Appendix Table C-1).

FIGURE 3-4

CATALYST ACTIVITY AS FUNCTION OF DONOR HYDROGEN AND SPACE TIME

Feed: MPSS
Catalyst: Ni/Mo-10
Age: 39-41 weeks

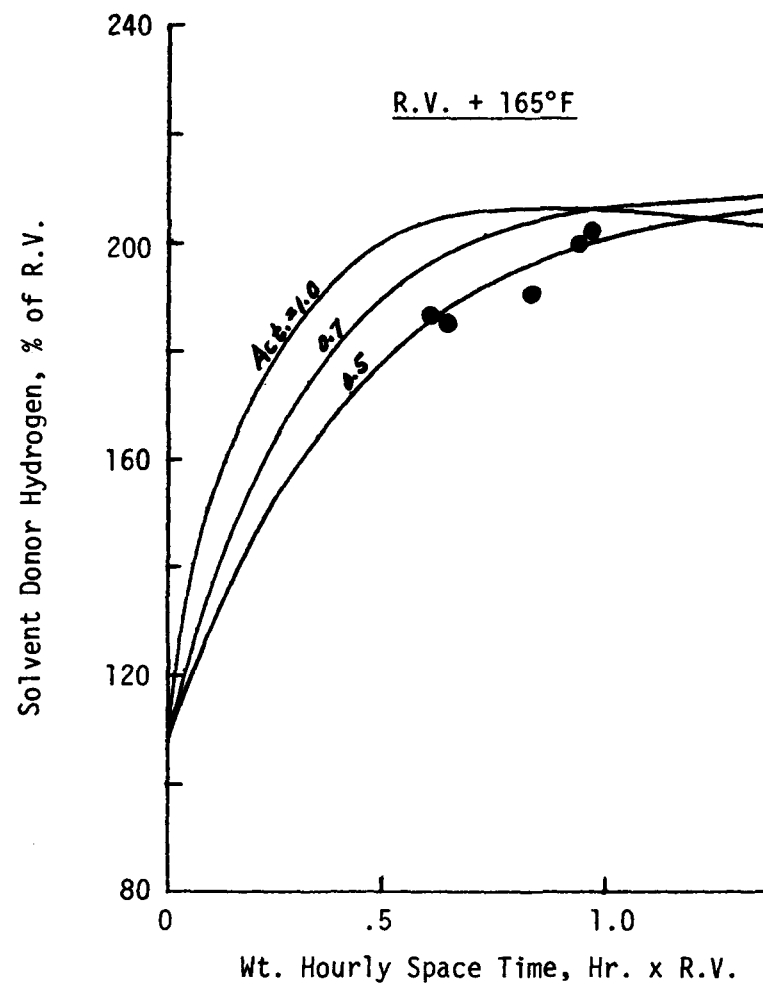
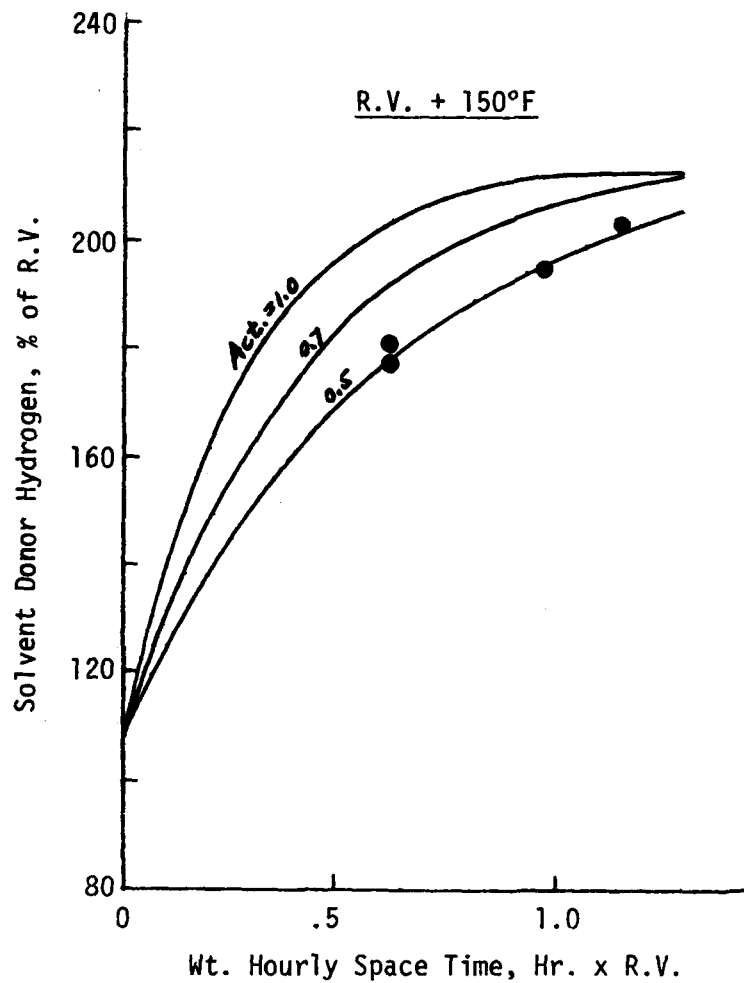


FIGURE 3-5

CATALYST ACTIVITY AS FUNCTION OF DONOR HYDROGEN AND SPACE TIME

Feed: MPSS
Catalyst: Ni/Mo-10
Age: 39-41 weeks

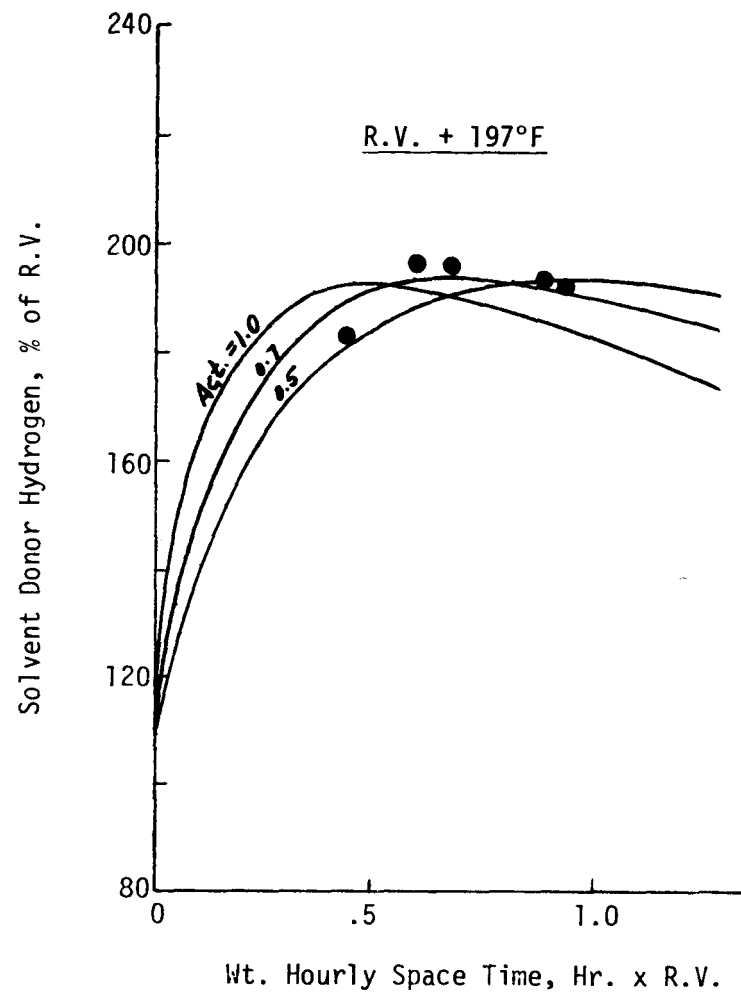
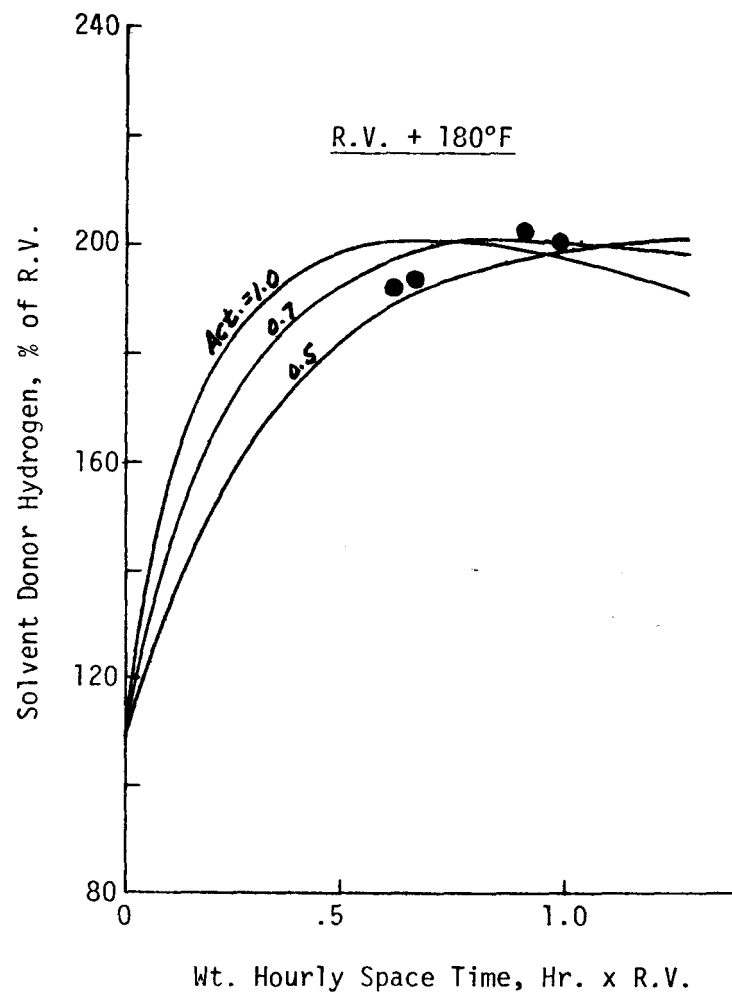


FIGURE 3-6

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

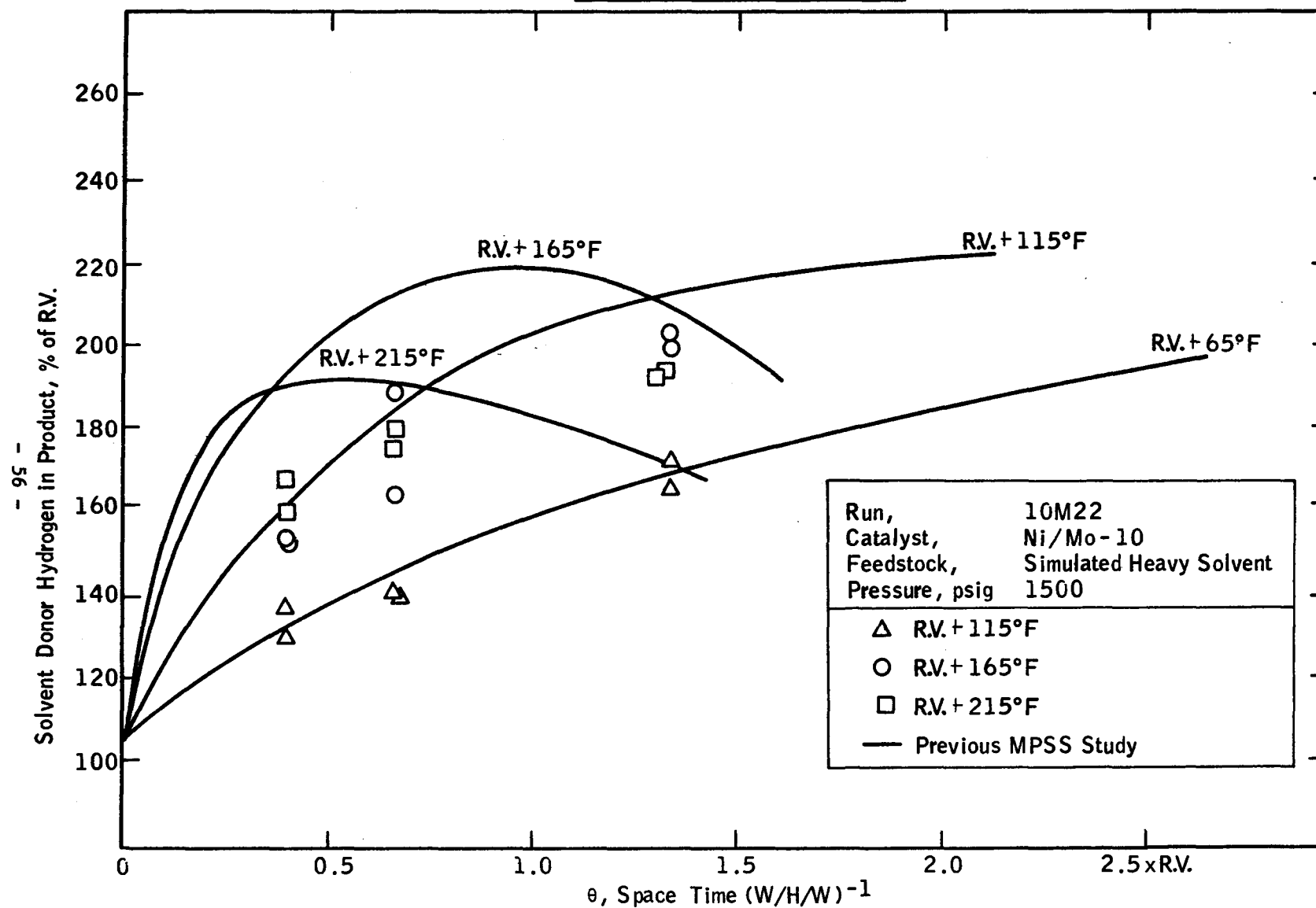
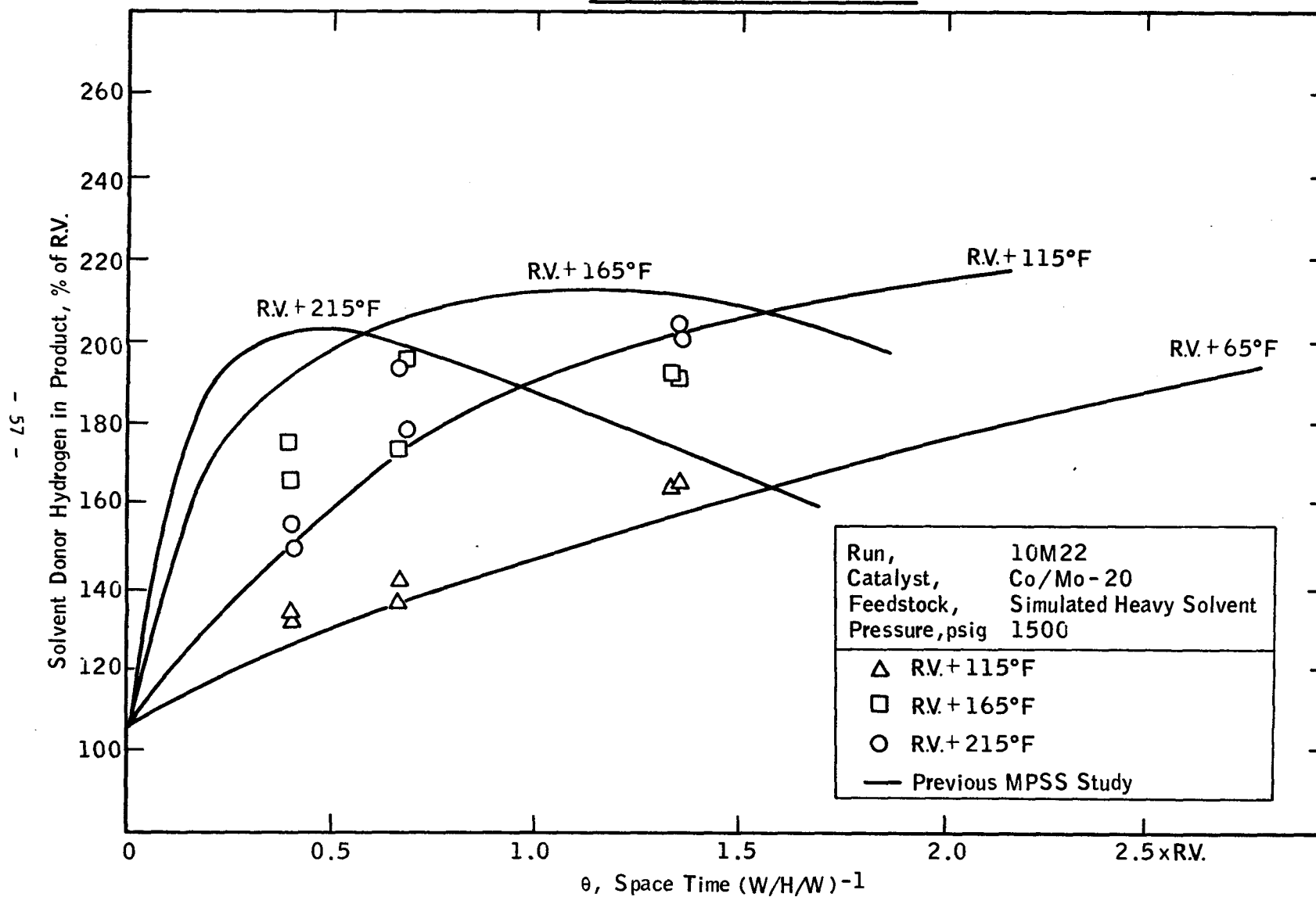


FIGURE 3-7

EFFECTS OF TEMPERATURE AND SPACE TIME ON HEAVY SOLVENT
FOR Co/Mo-20 CATALYST



Test data show that the Wyoming spent solvent behaved differently than Illinois solvent (MPSS) and the effect on catalysts was also different. Results with Wyoming spent solvent and the previous Illinois solvent are shown in Figures 3-8 and 3-9, respectively, for Ni/Mo-10 and Co/Mo-20 catalysts.

Figure 3-8 shows a higher solvent donor hydrogen content was produced from the Wyoming spent solvent with Ni/Mo-10 catalyst at the lowest temperature (RV+65°F) over the range of space times studied. The Wyoming spent solvent product donor hydrogen is about 10% higher than the Illinois (MPSS) after the difference in feed donor hydrogen is accounted for. However, the Illinois (MPSS) product donor hydrogen level is higher than the Wyoming donor hydrogen by an average 20% for space times greater than approximately 0.4 x RV hours and temperatures ranging from RV+115°F to RV+215°F.

Figure 3-9 shows the product donor hydrogen for both feeds is about the same with Co/MO-20 catalyst at the lowest temperature (RV+65°F) over the range of space times studied. However, the Illinois (MPSS) product donor hydrogen is higher than the Wyoming donor hydrogen by about 10% for space times greater than approximately 0.3xRV hours and temperatures ranging from RV+115°F to RV+215°F.

The cause of the observed difference in response to hydrotreating is not clear at this time. Additional screening studies on Wyoming solvent will be undertaken in July, 1978.

3.6 Catalyst Activity Screening Study

A catalyst activity screening study was conducted to obtain additional data with fresh Ni/Mo-10 catalyst activity at a higher operating temperature. These data will be used to extend reaction rate parameters in the fundamental molecular model for catalytic solvent hydrogenation. The study was carried out at a constant temperature of RV+125°F. A feedstock produced from Illinois coal in recent CLPP operation was utilized for this SOR activity screening study. The properties of the feedstock are shown in Appendix Table C-1.

Figure 3-10 compares the data with those obtained from a previous study utilizing MPSS feedstock. Despite differences in solvent donor hydrogen, oxygen and saturates in the feed, the products show comparable quality at all space time ranges.

3.7 Carbon Oxide Impurity Study

Economic studies have indicated incentives for the elimination of a methanator and low temperature shift converter in the EDS hydrogen plant, but effects of the resulting CO_x impurities in the hydrogen treat gas on solvent hydrotreating were not known. Consequently, studies

on the effect of carbon oxide impurities were conducted with Ni/Mo-10 catalyst and Illinois spent solvent. The purpose of the study is to confirm previous observations (see January 1976 to June 1977, EDS Final Technical Progress Report [FE-2353-20]) that carbon oxides do not adversely affect catalytic activity for solvent hydrogenation. The two-train catalyst testing unit was used in these studies.

The first study (Run 12M22) was carried out at a space velocity of 2.63xRV and temperatures ranging from RV+110°F to RV+215°F. Hydrogen only was used in one of the two trains and hydrogen spiked with 2% carbon monoxide and 2% carbon dioxide in the other. Tail gas samples and liquid products were collected and analyzed.

The results from this study confirmed previous results showing no effect on catalyst activity for solvent hydrogenation with carbon oxide impurities. Examination of the liquid products indicated that the carbon oxides did deactivate the catalyst for desulfurization, but the deactivation was apparently reversible. Desulfurization exceeded 80% at all test conditions versus 95+% without treat gas impurities. Analyses of the tail gas samples showed some conversion of carbon dioxide to carbon monoxide and methane. The results also showed that the conversion to methane was increased with increasing temperature. A summary of the results from this carbon oxide impurity study is shown in Table 3-2.

Additional SOR conditions were studied to broaden the data base in Run 13M22. Operation was carried out at various space velocities and temperatures ranging from RV+125°F to RV+190°F.

The data work up is not complete at this time. From data on hand, preliminary results also agree with the previous studies that no effect on solvent hydrogenation catalyst activity with carbon oxides in the treat gas stream.

TABLE 3-2

RESULTS OF CARBON OXIDE IMPURITY STUDY

Catalyst: Ni/Mo-10

Feedstock: Illinois Spent Solvent

Treat Gas Impurity: H₂ (+ 2.3% CO₂/2.1% CO)

Temp.-RV °F	Pressure psig	Space Velocity W/H/W % of R.V.	Treat Gas	Relative Activity*	Gas Product, Mole %		
					CO ₂	CO	CH ₄
115	1500	263	H ₂	100	Trace	Trace	.017
115	1500	263	H ₂ (CO ₂ /CO)	98	1.91	2.62	.335
165	1500	263	H ₂	100	.004	Trace	.097
165	1500	263	H ₂ (CO ₂ /CO)	101	1.51	2.94	1.13
215	1500	263	H ₂	100	.02	Trace	.30
215	1500	263	H ₂ (CO ₂ /CO)	102	1.59	2.42	2.34

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

FIGURE 3-8

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

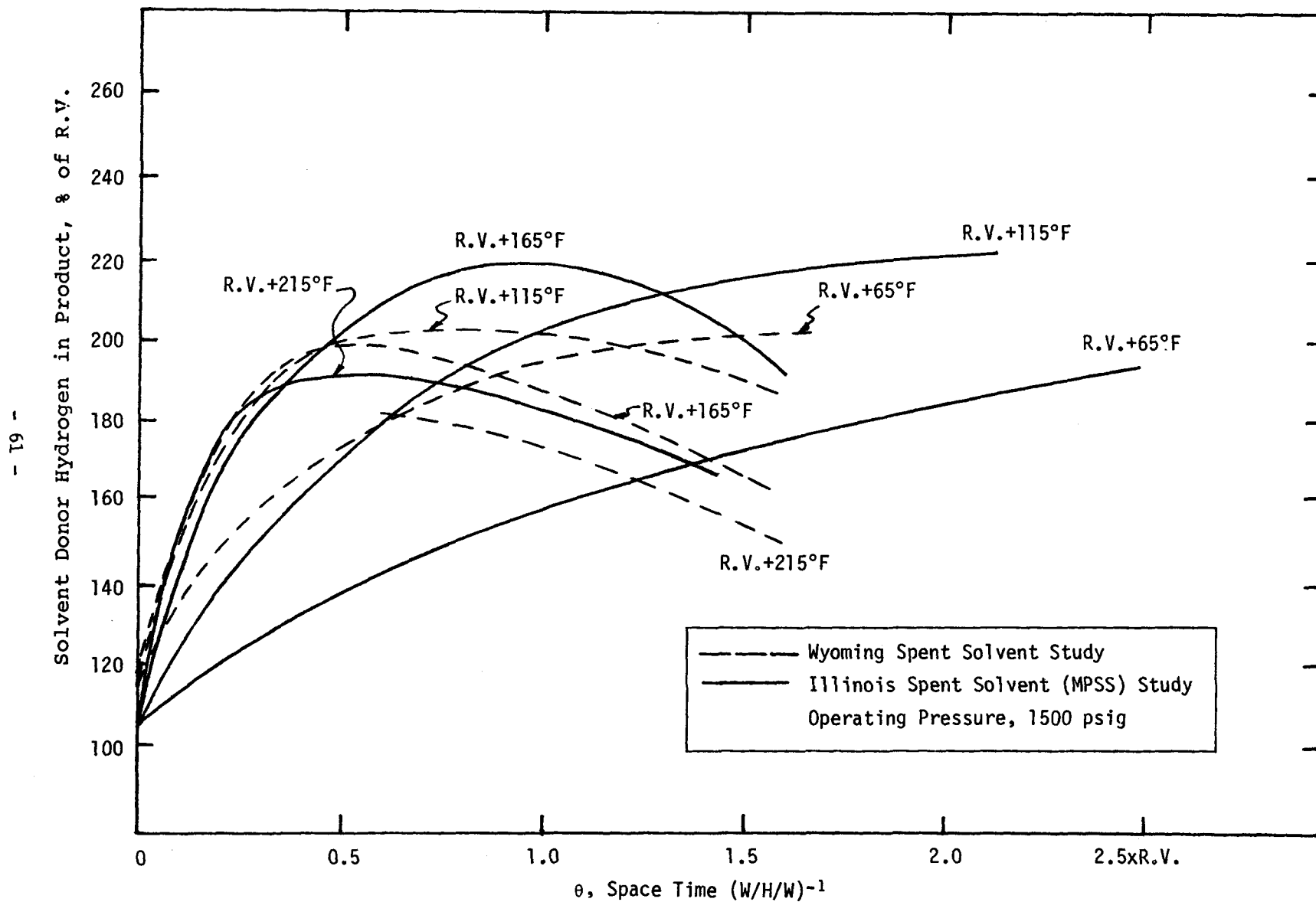


FIGURE 3-9

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

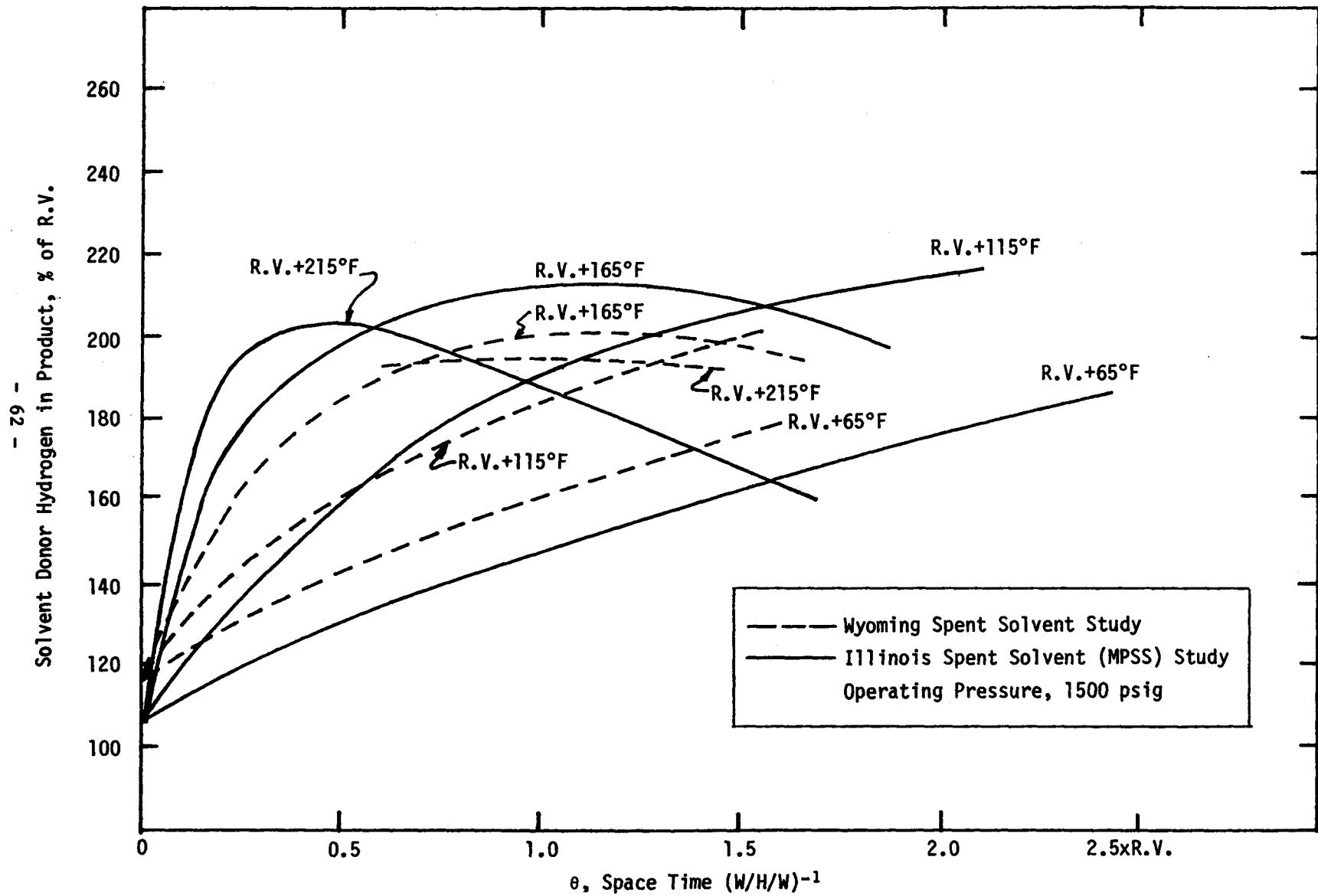
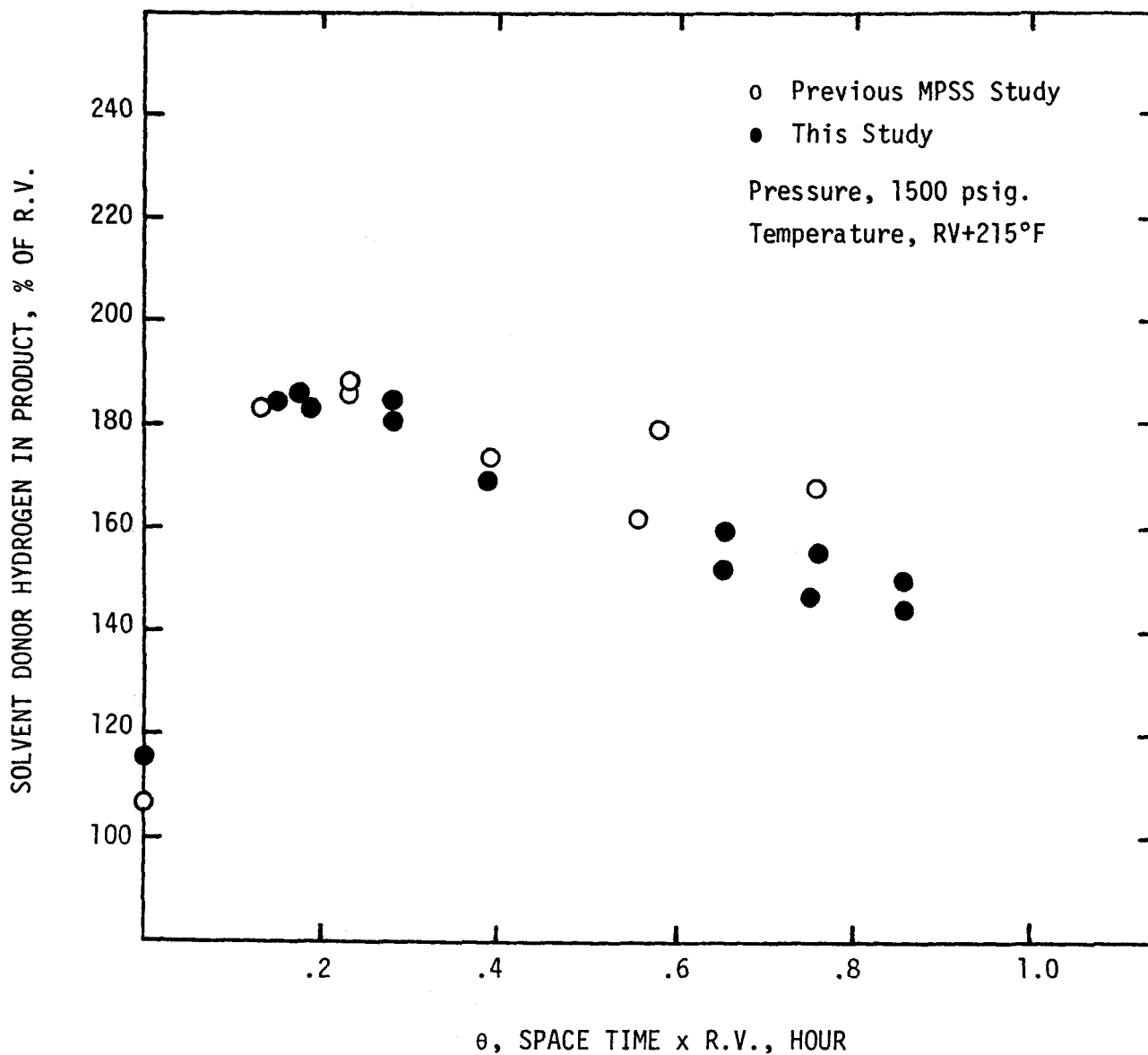


FIGURE 3-10

EFFECT OF SPACE TIME ON THE HYDROGENATION
OF ILLINOIS SOLVENT OVER Ni/Mo-10 CATALYST



LABORATORY PROCESS RESEARCH AND DEVELOPMENT

LIQUEFACTION PROCESS RESEARCH

4. Bench Units, Modeling and Correlation Studies

Batch tube autoclave and continuous flow reactors are used to provide yield and operability data for a variety of coals. The yield data from the bench scale hydrotreaters and liquefaction pilot plants are correlated to provide models for recycle solvent hydrogenation and liquefaction models which will be incorporated into the EDS Process Model.

4.1 Operability Studies with Wyoming Coal

During the liquefaction of low rank coals, such as Wyoming coal, the calcium which is contained in the form of salts of humic acids decomposes to form calcium carbonate (CaCO_3). This calcium carbonate accumulates in the liquefaction reactor as wall scale and free-flowing aggregates. The fifteen pound-per-day Once-Through Coal Liquefaction Unit (OTCLU) is used to test proposed solutions to the calcium carbonate deposition problem. These solutions can be classified as mechanical or chemical solutions. Mechanical solutions are those in which the process conditions, process configuration or mechanical equipment is modified to alleviate the problems due to the CaCO_3 deposition. Chemical solutions are those in which the chemical form of the calcium is changed by pretreatment or a chemical is added to the coal slurry before it is liquefied.

Operating conditions and feed and product inspections for these OTCLU studies are presented in Appendix D.

One mechanical solution tested in OTCLU was the liquefaction of Wyoming coal with run of mine moisture content (Ca. 30 wt. %). Batch tube autoclave liquefaction of this coal produced CaCO_3 in the form of calcite only with no vaterite which is usually produced with dry coal. Since the CaCO_3 wall scale produced from dry Wyoming coal contains only vaterite, the use of high moisture coal was proposed to eliminate the reactor wall scale. A run was made on OTCLU with -100 mesh Wyoming coal with 28 wt. % moisture. Upon completion of this run the reactor was examined for scale. Approximately twelve grams of wall scale was recovered. Since this is the same amount that would be expected from a run with dry coal, the moisture content of Wyoming coal had no effect on the amount of wall scale deposited.

Calcium carbonate formation in liquefaction can be prevented by chemically altering the form of the calcium in the coal by pretreatment. The calcium which forms CaCO_3 is in the form of a calcium humate and is easily ion-exchanged with other metal salts or acids. Various metal salts were used as ion-exchange agents. Ferrous sulfate, ferric sulfate, sodium sulfate and sodium carbonate pretreated coals were tested in OTCLU. All

salts with the exception of sodium sulfate were effective in eliminating CaCO_3 wall scale and agglomerates. Batch tube autoclave tests of the sodium sulfate treated coal indicated that only about one half of the calcium was exchanged. OTCLU runs with Wyoming coal pretreated with the other salts produced no wall scale or high calcium carbonate concentrations. With the sodium carbonate treated coal there was calcium carbonate in the reactor since this is the calcium salt formed in the ion exchange.

These results are consistent with typical ion exchange experience. Divalent and trivalent metal ions will exchange with the divalent calcium to a much greater extent than the monovalent metal ions. However, monovalent metal ions will exchange if there is the added driving force of the insolubility of the calcium salt formed (calcium carbonate in the case of sodium carbonate pretreatment).

Chemicals have also been physically added to the coal slurry to alter or interrupt the crystal growth of the CaCO_3 . Colloidal silica and calcium carbonate were used as reactor additives in separate OTCLU runs to provide additional growth sites for calcium carbonate growth. If the CaCO_3 would grow preferentially on these additives, reactor wall scale could be eliminated. Colloidal silica did reduce the amount of wall scale recovered after the OTCLU run, but did not eliminate it. The use of colloidal CaCO_3 caused numerous operational problems in the OTCLU feed system, particularly the high pressure pumps. Therefore, the use of colloidal silica or CaCO_3 is not a viable solution to wall scale deposition.

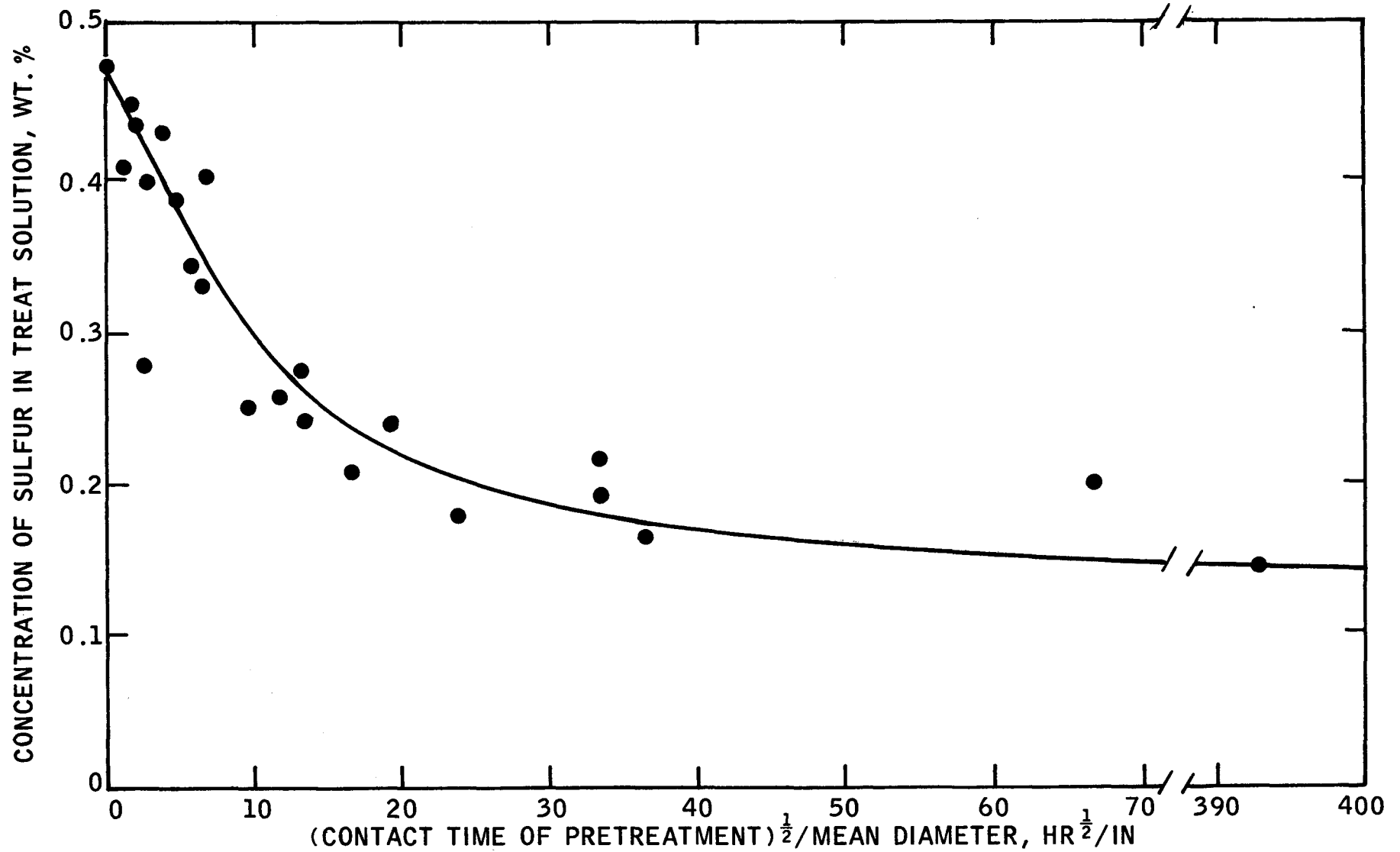
Another possible way to prevent CaCO_3 deposition is to add a surfactant to the coal slurry. This surfactant could then cause the CaCO_3 to form in a fine dispersion, preventing crystal growth, and allowing the CaCO_3 formed to flow out of the reactor. An OTCLU run was made with 1 wt.% (based on coal) anthracene disulfonic acid (ADA) added to the feed slurry. During this run nine grams of CaCO_3 wall scale was deposited in the liquefaction reactor. Thus, the use of ADA had no appreciable effect on wall scale growth.

During Phase IIIA, pretreatment of low rank coals with dilute sulfuric acid was shown to be effective in preventing CaCO_3 formation. The kinetics of H_2SO_4 pretreatment were studied with wet Wyoming coal (July-September, 1977, EDS Quarterly Technical Progress Report [FE-2893-3]). Treat effectiveness was determined by correlating the concentration of sulfur remaining in the treat solution as a function of the square root of the contact time divided by the mean particle diameter (Figure 4-1). This representation allows for easy interpolation to determine the required contact time for particles of a given size.

Pretreatment of moist Wyoming coal with SO_2 gas has also been found to be an effective means of converting calcium in coal to calcium salts which are stable during liquefaction. A six day run was made in OTCLU feeding Wyoming coal treated in this manner. No CaCO_3 wall scale was found at the end of this run, confirming the effectiveness of gaseous SO_2 as a pretreatment agent.

FIGURE 4-1

THE CONCENTRATION OF SULFUR IN THE TREAT SOLUTION
AS A FUNCTION OF $\sqrt{t}/d$ USING A 1.5 PER CENT H_2SO_4 SOLUTION



A 4-inch diameter, plexiglass Fluidized Pretreatment Unit (FPU) was constructed to study SO_2 pretreatment (See Figure 4-2). It was found that Wyoming coal can be effectively and uniformly treated in the FPU. Coal treated in the FPU, upon liquefaction in tube autoclaves, resulted in a 3-4 fold reduction in the amount of CaCO_3 present in the liquefaction residue, when compared to untreated Wyoming coal. This reduction has proven to be sufficient to indicate that no significant CaCO_3 wall scale problem would exist in a continuous flow unit.

4.2 Coal Screening

Collection of tube autoclave data on the Buelah, Cambria and Wyodak coals has been completed. These data have been added to the data base to be utilized in a correlation study in an attempt to develop a simple coal screening tool which will allow estimation of coal liquefaction performance based on measurable physical and chemical coal properties. This study will be completed in the next quarter.

4.3 Fundamental Model for Coal Liquefaction

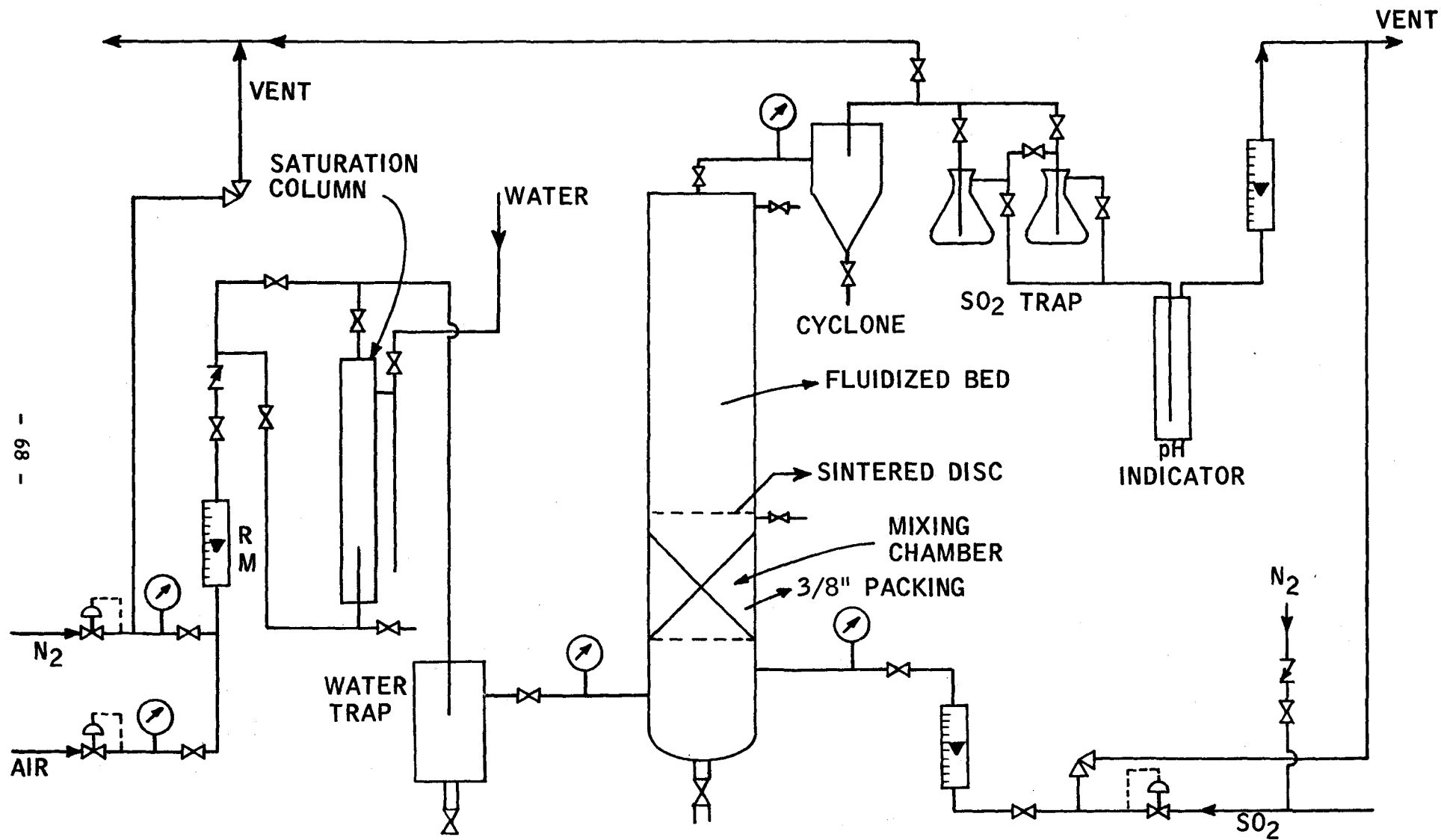
An empirical model was developed during Phase IIIA to correlate liquefaction yields for Illinois coal (see January-June, 1976, EDS Quarterly Technical Progress Report [FE-2353-2]). That model was subsequently used as a guide in developing an empirical model to describe liquefaction of Wyodak subbituminous coal. Both models postulate a kinetic scheme for conversion of coal to $1000^\circ\text{F}+$ bottoms as a function of temperature and space time, and then correlates individual component yields as functions of coal conversion. Both models have been incorporated into the EDS Process Model to generate input vectors for the SFED Process Alternatives LP Model (PAM).

To gain additional insight into the coal liquefaction process and thereby have a more flexible implement for predicting the effects of major process variables on the system, the development of a more fundamental model of liquefaction is in progress. The approach of this effort is to group individual constituents into lumped components on the basis of similar chemical reactivities. The initial formulation consists of a minimum number of lumped components and kinetic reaction equations to describe the production and consumption of the various components. After a first-pass fit of the model to the data has been obtained, subsequent refinements will be made in which the size of the lumps is progressively decreased and the number of corresponding kinetic reaction equations is increased. The definition of lumped components has been made in a manner consistent with the solvent hydrogenation model so that they can be made to interact in a closed loop simulation.

During the first quarter of 1978, efforts were directed toward fitting this liquefaction model to data for the 840°F isothermal liquefaction

FIGURE 4-2

FLUIDIZED PRETREATMENT UNIT (FPU) SCHEMATIC



of Illinois coal at space times ranging from 15 to 40 minutes. These data are for liquefaction only and were obtained by computer analysis of yield periods for the 50 pounds-per-day RCLU 1, 100 pounds-per-day RCLU 2 and one ton-per-day CLPP pilot plants. The first-pass fit of the model to the isothermal data was completed during that reporting period, and the results indicated reasonable agreement between the model and experimental data.

Primary emphasis during the second quarter of 1978 was directed toward compiling liquefaction data at temperatures of 800 and 880°F, and fitting the model to the complete data base for Illinois coal. These data cover space times ranging from 15 to 60 minutes. Efforts to analyze the data to determine the effect of temperature on the model parameters were successful in obtaining preliminary estimates of the temperature effect. Further analysis of the data will be conducted to determine the optimum set of parameter estimates.

Planning for the development of a level 2 EDS Process Model was also initiated during this quarter. The simulator will incorporate the fundamental models for coal liquefaction and solvent hydrogenation into an integrated system. Lumping/delumping modules will be developed to provide proper interaction between the liquefaction, solvent hydrotreating and separation modules. This effort will be emphasized more during the third quarter of 1978.

4.4 Liquefaction Model for Wyodak Subbituminous Coal

The liquefaction model for Wyodak subbituminous coal was completed during the final quarter of 1977. The model incorporates data from the OTCLU and RCLU pilot units into its data base, and correlates yields as functions of temperature (T) and residence time (θ). The coal conversion data were subdivided into individual component yields similar to those reported previously for the Illinois coal liquefaction model (see January-June, 1978, EDS Quarterly Technical Progress Report [FE-2893-7]).

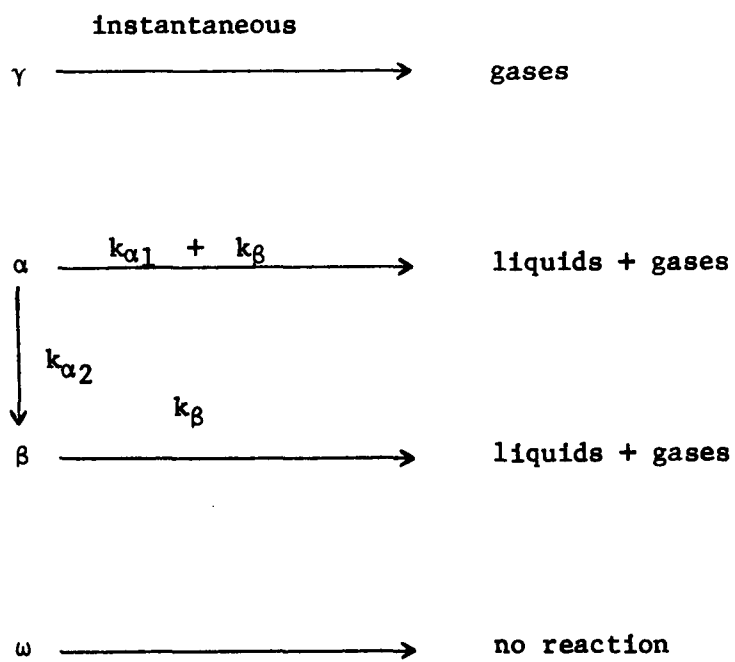
The data for liquefaction of Wyodak coal did not cover as broad a range of conditions as did the corresponding data base for Illinois coal. Therefore, the approach and form of the liquefaction model for Illinois coal was used as a guide for the Wyodak model. Thus, the Wyodak coal was divided into four reactive fractions corresponding to the same reactive classes for Illinois coal (see Figure 4-3). The volatile fraction, labeled γ , was experimentally determined by mass spectrometry to be 14 wt. % on coal. The unreactive fraction, labeled ω , was experimentally determined by petrographic analysis to be 15 wt. % on coal.

The form of the differential kinetic equations, and the analytic solution to these equations, is the same as reported previously (January-June, 1976, EDS Quarterly Technical Progress Report [FE-2353-2]) for Illinois coal. To summarize, the relationship giving 1000°F+ bottoms yield as a function of temperature and residence time is

FIGURE 4-3

LEVELS OF REACTIVITY IN COAL

Definition: $\text{Coal} \equiv \alpha + \beta + \gamma + \omega$



$$\text{Residue (1000°F+)} = \omega + (1-\omega-\gamma) \frac{\alpha+\beta}{\alpha_0+\beta_0} \quad (4-1)$$

where

$$\frac{\alpha+\beta}{\alpha_0+\beta_0} = \frac{k_{\alpha 1}}{k_{\alpha 1}+k_{\alpha 2}} \exp[-(k_{\alpha 1}+k_{\alpha 2}+k_{\beta})\theta] + \frac{k_{\alpha 2}}{k_{\alpha 1}+k_{\alpha 2}} \exp(-k_{\beta}\theta) \quad (4-2)$$

The kinetic rate constants appearing in Equation 4-2 were determined using a computerized, non-linear regression analysis to fit the experimental data. Assuming an Arrhenius form for the rate constants,

$$k_i = A_i \exp(-E_i/RT)$$

where the gas constant $R = 1.987$ cal/mole °K, the analysis gave estimates for the parameter values that were reported previously in the October-December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7). Figure 4-4 plots predicted and observed 1000°F+ bottoms yield as a function of T and θ . The predicted yield at 880°F is shown as a dashed line because there were no pilot unit data with which to compare the prediction. The standard deviation and maximum deviation of the fit are approximately 1.6% and 2.2%, respectively, based on coal, which is within the limits of experimental accuracy.

A comparison of predicted 1000°F+ BTMS yields for the Wyodak and Illinois models is plotted in Figure 4-5. It is apparent that for a given temperature and residence time Wyodak coal gives more total conversion than does Illinois coal. However, this greater conversion is due to the greater volatility of the Wyodak coal upon introduction into the liquefaction reactor. The total liquefaction liquid yield ($C_4/1000$) is higher for Illinois coal than for Wyodak coal.

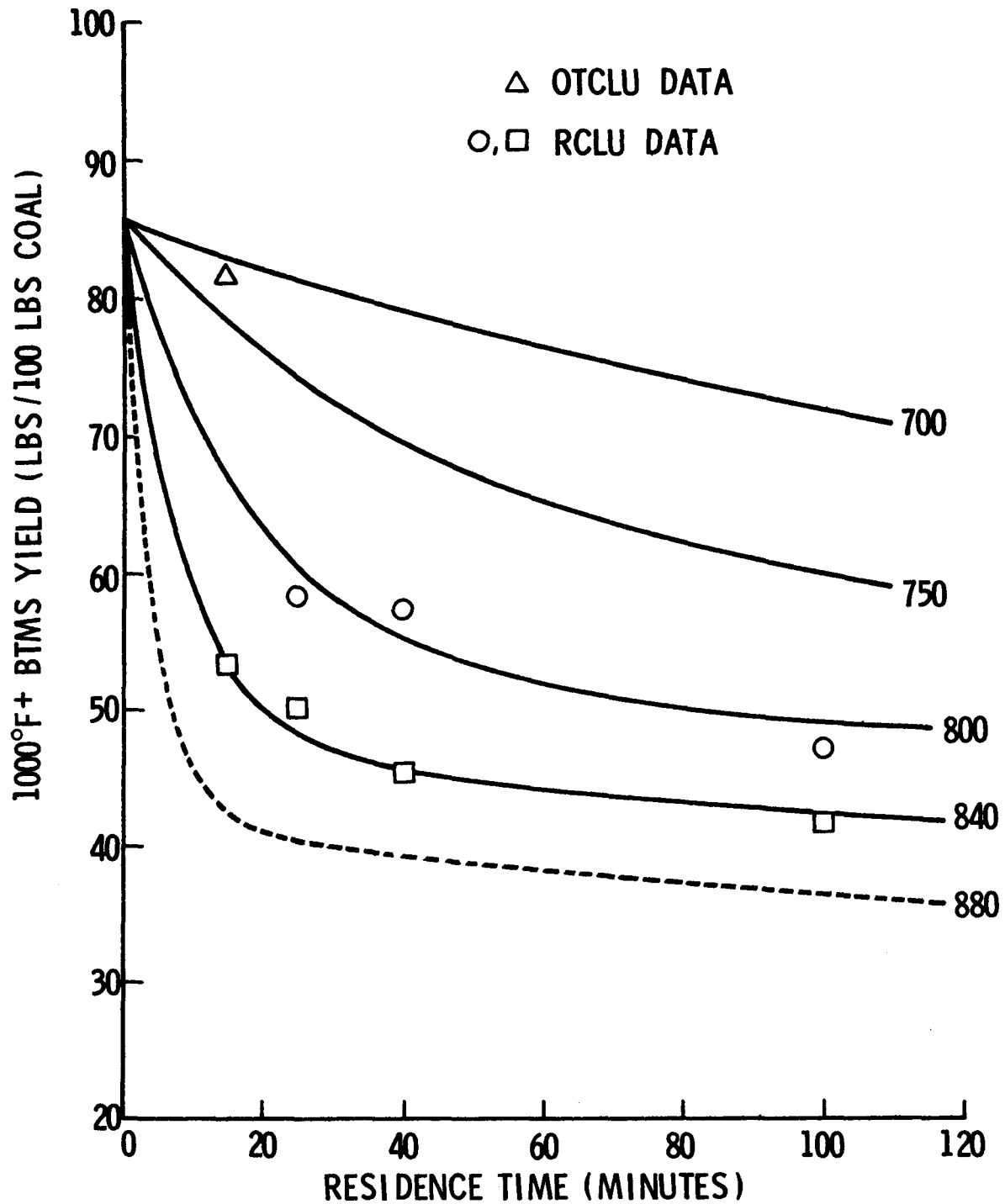
The products of liquefaction are lumped into eight component pools based primarily on similar physical characteristics (boiling points). Since the products, with the exception of 1000°F+ bottoms and a portion of the 700-1000°F liquid, pass through the hydrotreating reactor, the correlations presented are useful for the prediction of overall yields from an integrated liquefaction/solvent hydrogenation reactor system. All component yields were least squares curve fit to a quadratic equation,

$$\text{Yield}_j = a_j + b_j X + c_j X^2 \quad (4-4)$$

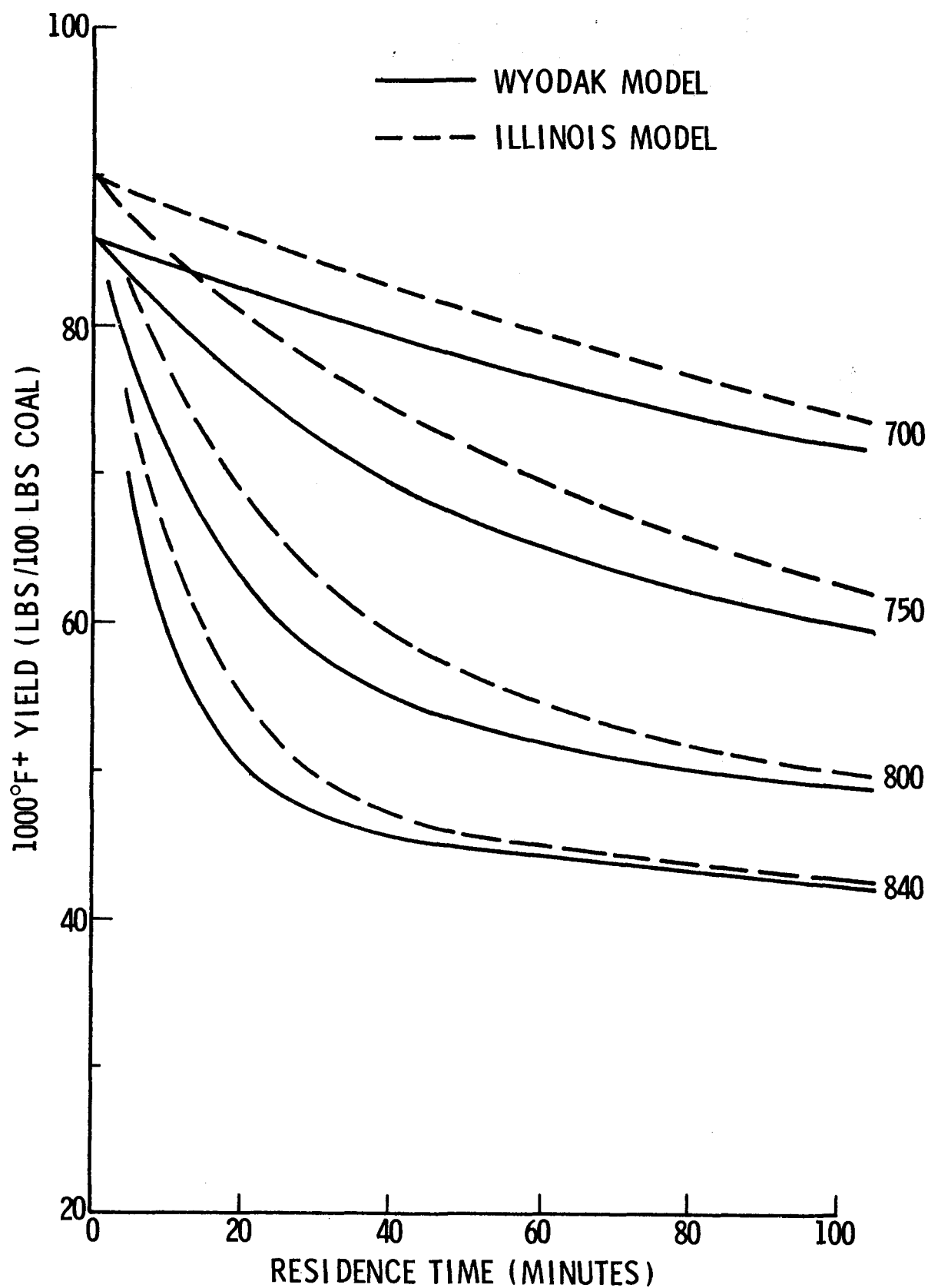
where 1000°F conversion = $100 - \% \text{ 1000°F+ bottoms yield}$.

FIGURE 4-4

LIQUEFACTION MODEL FOR WYODAK COAL
PREDICTED 1000°F+ BTMS YIELD



COMPARISON OF WYODAK MODEL AND ILLINOIS MODEL
PREDICTED 1000°F+ BTMS YIELD



The parameter estimates for yield as a function of conversion were reported previously in the October-December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7). These equations were normalized to ensure mass balances throughout the range of process yields, and they can be used with confidence for 1000°F conversions in the range 40-60 wt. % on feed coal.

A computer module containing the Wyodak coal liquefaction model was also developed during the last quarter of 1977 for insertion into the EDS Process Model. The Process Model was used to generate a grid of 18 yield vectors as input to the Process Alternatives LP Model (PAM). The yield vectors represented simulation of the following liquefaction conditions:

- Reactor temperatures of 800, 840 and 880°F
- Six residence times at each temperature. The residence times ranged from 15 to 120 minutes.

The subsequent PAM studies will be used to determine the preferred operating conditions for a balanced commercial plant operating with Wyodak coal.

4.5 Mathematical Modeling of Solvent Hydrogenation

The initial attempt at a fundamental description for solvent hydrotreating was based on a fundamental approach describing the interaction between individual component reaction paths and process variables. However, due to the limited number of components included, the initial model formulation was unable to accurately predict macroscopic solvent characteristics (e.g., total donor hydrogen).

A refined version of the initial model formulation was obtained by adding additional compounds and their reaction paths to the model. These additional components comprise a significant fraction of the solvent, and exhibit reactivities similar to species included in the initial model. The original species and the additional components were grouped into component pools based on their comparable reactivities. Components were lumped in such a way that the improved model is no more complex than the initial model, yet contains significantly more information.

The specific classes of compounds that are assumed to exhibit common reactivities are the polynuclear aromatics and their alkyl substituted homologs. For example, since all alkyl substituted tetralins react similarly to tetralin, they are all lumped together in a common pool. Lumping on the basis of common reactivity, in contrast to lumping by boiling pools, does not greatly compromise the fundamental nature of the reaction model.

The refined model was validated by simulating hydrotreater operations with partially hydrogenated creosote oil (PHCO) and Co/Mo-10 catalyst parameters. Simulations were made over a wide range of

reactor temperatures and space velocities. The refined model was found to be more accurate than the initial model in simulating experimentally measured total donor hydrogens over the range of operating conditions studied.

Reaction rate parameters for Ni/Mo-10 catalyst were also determined based on the refined solvent hydrogenation model formulation. Arrhenius type expressions were used for all rate constants. Parameters determined for the major donatable hydrogen pool reactions provide very good fit of the data, while those describing the heaviest donatable hydrogen pool reactions are acceptable. Rate constants were also determined for reactions representing the lighter and heavier components not included in the hydrogen donor pools.

Both Co/Mo-10 and Ni/Mo-10 catalyst rate parameters were incorporated into a computer program which simulates the solvent hydrogenation process in terms of the type of components discussed above. This computer program then calculates total donatable hydrogen and total saturates concentration for either catalyst based on process operating conditions such as temperature, hydrogen partial pressure, space velocity and feed composition. In addition, for Ni/Mo-10 catalyst only, heteroatom (sulfur, oxygen, nitrogen) concentrations, hydrogen consumption, chemical gas (NH_3 , H_2S , H_2O) and C_1 - C_3 hydrocarbon gas production, and relative catalyst activity are calculated.

Operating the solvent hydrotreater near solvent chemical equilibrium conditions can result in a low, undesirable donatable hydrogen level due to the conversion of hydroaromatic compounds to naphthenes (saturates). Thus, studies were undertaken to define conditions at which the solvent tends to equilibrate chemically. Simulations were carried out for both Co/Mo-10 and Ni/Mo-10 at initial catalyst activity. The approach to solvent chemical equilibrium was determined in terms of product stream donatable hydrogen. Results of the simulations showed that chemical limitations would not be a factor in hydrotreater operations for either catalyst. These results also allowed comparison of the relative activities of the two catalyst over a wide range of operating conditions.

Results from solvent hydrogenation experiments carried out in the bench scale hydrotreating units are based on once-through operations. As such they do not correspond to an integrated recycle operation. Thus, the effects of liquefaction and solvent hydrogenation processing conditions on the saturates content of the recycle solvent in the integrated EDS process at steady state conditions cannot be determined directly in the bench scale units. In addition, the saturates effect on the donatable hydrogen content of the recycle solvent during integrated operations cannot be determined on these units. As a result, a simulation of integrated EDS process operations was carried out to examine these effects.

Two models were used to simulate the interaction between liquefaction and solvent hydrogenation in the EDS recycle solvent at steady state conditions. The liquefaction process was represented by the empirical

model now used in the level 1 EDS Process Model. Solvent hydrogenation was represented by the refined fundamental model with Co/Mo-10 catalyst parameters at initial activity. Saturates content simulated by this approach indicated that saturates do build up during integrated recycle operations and decrease the solvent quality. The effect of saturates buildup, however, is not detrimental to solvent quality at the conditions studied until a relatively high temperature is reached. This simulated approach was verified by comparison with limited RCLU-1 data taken during integrated recycle operations at steady state.

Solvent hydrogenation modeling work has been concerned with construction of a catalyst deactivation model during 2Q78. The preliminary model chosen for catalyst deactivation kinetics assumes nonselective deactivation. This means that each reaction is affected in the same way by catalyst deactivation. Data from recent catalyst activity maintenance studies (Runs 19M20 and 14M21) were used to estimate catalyst activity as a function of time-on-stream, space velocity and temperature. Activity was correlated with catalyst age and hydrotreater operating conditions. The deactivation kinetic parameters obtained provide good fit of the data from the two catalyst activity maintenance studies.

The catalyst deactivation model is being validated at the present time. Additional process variable studies conducted on the bench scale hydrotreater unit are being evaluated in an effort to improve estimates of the deactivation kinetic parameters. End-of-run catalyst will be evaluated experimentally to confirm the deactivation model selected.

Future solvent hydrogenation modeling work will involve process variable studies with Wyoming coal solvent. In addition, heteroatom (sulfur, nitrogen, oxygen) removal as a function of catalyst activity will be examined.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

Liquefaction Process Research

5. Product Quality Studies

5.1 ECLP Process Stream Disposition Studies

A major element in the commercial development of the EDS process is the operation of a 250 ton-per-day large pilot plant (ECLP). Currently under construction in Baytown, Texas, ECLP will produce a naphtha, excess solvent and vacuum gas oil product. Plans are to dispose of products in excess of Project and Sponsor needs in the adjacent Exxon Refinery. To facilitate this disposition with minimum disruption of refinery operations, an acceptable disposition scheme had to be defined based on the properties of the coal liquids produced.

- Potential ECLP Product Disposition Scheme

At the end of Phase IIIA in the process development program, two potential ECLP product disposition schemes had been identified for further study. One scheme blended all of the ECLP process streams into refinery products for sale. A second scheme involved burning ECLP products as fuel for process heat with the option to blend vacuum gas oil (VGO) into heavy petroleum fuel oil for sales. Both schemes allowed for landfilling the VGO as an unattractive fallback if compatibility problems prevented blending with petroleum fuels or lighter coal liquids. Flow charts for both disposition schemes are shown in the July-September, 1977 EDS Quarterly Technical Progress Report (FE-2893-3).

- Product Disposition: Refinery Products for Sales

Blending studies carried out in Phase IIIA (see EDS Phase IIIA Final Technical Progress Report [FE-2353-20]) showed that the 300/400°F raw naphtha fraction derived from Illinois coal cannot be blended into refinery gasoline or heating oil without hydrotreating to reduce sulfur, nitrogen and oxygen levels. Additional studies in Phase IIIB (see July-September, 1977 Quarterly Technical Progress Report [FE-2893-3]) showed that this fraction cannot be blended into heavy fuel oil without significantly lowering flash point.

In addition to these limitations, test data obtained at Products Research Division indicated that raw naphtha performed poorly even if the final boiling point were limited to 275°F and concentrations in refinery gasoline were kept below 1%. These tests followed earlier data indicating that 325°F FBP naphtha might prove acceptable if concentrations were held to 1% or less. Storage stability tests with unleaded gasoline (Table 5-1) indicated these blends were unstable after 8 weeks storage (110°F) especially in the presence of 5% heat soaked steam cracker naphtha. Cutting the naphtha to a lower FBP was investigated (Table 5-2 for 300°F FBP and Table

5-3 for 275°F FBP). Test data reveal poor performance in gum testing and that most blends with coal naphtha promoted emulsification.

TABLE 5-1

110°F STORAGE STABILITY OF 325°F RAW COAL NAPHTHA BLENDS

<u>Gasoline</u>	<u>8 Week ASTM Gum, mg/100 ml</u>	
	<u>Washed</u>	<u>Unwashed</u>
Unleaded	2.7	17.2
+ 5% HS SCN*	2.1	19.6
+ 0.5% Raw 325°F Coal Naphtha	5.3	16.8
+ 0.5% Raw 325°F Coal Naphtha		
+ 5% HS SCN	2.4, 3.3	127.0, 64.0
+ 1% Raw 325°F Coal Naphtha	3.6	25.8
+ 1% Raw 325°F Coal Naphtha		
+ 5% HS SCN	5.6, 2.8	34.0, 96.0

* Heat soaked steam cracker naphtha

Table 5-2

Lab Tests - 300°F Illinois Raw Coal Naphtha in Unleaded Fuel

Composition, Vol.%

300°F Raw Naphtha	1	4	1	4	1	4	1
Heat Soaked Steam Cracker Naphtha			5	5			
Residfiner Naphtha					5	5	5
<u>Tests</u>							
ASTM Gum, mg/100 ml							
-Washed	0.2	0.8	2.2	2.6	3.0	2.4	4.0
-Unwashed	22.4	21.2	51.6	76.6	36.4	12.8	37.8
Copper Beaker Gum, mg/100	12.8	14.2	15.0	18.0	14.0	14.0	12.2
Potential Gum, mg/100 ml	26.0	84.0	25.3	33.0	17.6	78.0	24.4
110°F Storage Stability, mg/100 ml							
4 Weeks - Washed	5.5	6.0	4.5	7.8	6.2	5.0	5.5
- Unwashed	23.2	34.6	21.2	57.6	32.2	24.1	17.9
6 Weeks - Washed	5.4	4.7	4.6	5.9	3.8	8.5	6.6
- Unwashed	26.2	24.7	39.9	23.9	17.4	41.4	26.0
8 Weeks - Washed	5.4	5.0	3.4	4.0	3.4	5.2	8.9
- Unwashed	24.4	20.0	17.0	44.9	19.9	20.2	28.6
Emulsification ¹							
- Distilled Water	25	5	5	5	5	(12)	(3)
- Refinery Water Bottoms	75	75	75	1	80	(3)	50

¹ Minutes for emulsion to break or (in parentheses) % emulsion remaining after 2.5 hour.

Table 5-3

Lab Tests - 275°F Illinois Raw Coal Naphtha in Unleaded Fuel

<u>Composition, Vol.%</u>								
275°F Raw Naphtha	1	4	1	4	1	4	1	
Heat Soaked Steam Cracker Naphtha			5	5			5	5
Residfiner Naphtha					5	5	5	5
<u>Tests</u>								
ASTM Gum, mg/100 ml								
- Washed	1.4	3.2	1.2	2.2	1.2	3.4	1.2	3.0
- Unwashed	9.0	15.8	77.0	10.6	10.4	16.0	14.0	12.6
Copper Beaker Gm, mg/100 ml	15.6	22.2	17.0	25.0	13.6	12.4	20.4	21.6
Potential Gum, mg/100 ml	46.8	56.6	27.8	35.6	63.8	26.2	51.4	20.8
110°F Storage Stability, mg/100 ml								
4 Weeks - Washed	3.2	12.4	4.8	5.1	2.1	7.3	4.1	4.6
- Unwashed	20.6	72.7	51.3	16.9	13.7	27.3	12.8	13.7
6 Weeks - Washed	3.8	5.6	3.8	5.9	2.6	3.4	3.2	2.9
- Unwashed	44.6	60.8	21.9	16.5	60.9	28.1	11.8	14.7
8 Weeks - Washed	2.8	3.4	3.5	5.7	3.3	4.4	3.2	1.9
- Unwashed	21.9	24.2	20.4	21.0	12.9	28.4	15.5	13.9
<u>Emulsification¹</u>								
- Distilled Water	90	(92)	90	80	80	75	12	18
- Refinery Water Bottoms	5	5	5	5	5	5	5	5

¹ Minutes for emulsion to break or (in parentheses) % emulsion remaining after 2.5 hours.

Regarding partially hydrotreated (P/H) naphtha, earlier work had indicated that 1% and lower levels of a 400°F FBP naphtha was satisfactory in laboratory gum tests, although demulsifier is required. Lab testing was carried out on 350°F FBP P/H naphtha to determine if the removal of some of the heavy ends might permit use of a higher concentration of naphtha. The 350°F FBP naphtha was prepared from components that had been previously distilled into a series of narrow boiling fractions (see July-September, 1976 Quarterly Technical Progress Report [FE-2353-6] for distillation data). Test results summarized in Table 5-4 show that blends with 1% or more coal naphtha in unleaded fuel were borderline in ASTM gum formation and that all blends promoted emulsification.

TABLE 5-4

LAB TESTS - 350°F PARTIALLY HYDROGENATED
ILLINOIS COAL NAPHTHA IN UNLEADED FUEL

Vol. % Coal Naphtha	0	0.5	1	4
ASTM Gum, mg/100 ml				
- Washed	0.7	0.8	4.2	3.2
- Unwashed	28.7	7.6	35.0	8.6
Copper Beaker Gum, mg/100 ml	14.4	12.2	14.4	16.8
Potential Gum, mg/100 ml	33.4	38.9	30.3	27.5
Emulsification ⁽¹⁾				
- Distilled Water	5	(100)	(100)	(100)
- Refinery Water Bottoms	5	30	60	60

Evaluation of hydrotreated naphtha from Baton Rouge (see EDS Phase IIIA Final Technical Progress Report [FE-2353-20] for inspections) showed different performance in leaded and unleaded gasoline (Table 5-5). All concentrations tested in the unleaded fuel gave high gum deposits after 8 weeks storage at 110°F. The leaded fuel blends appeared generally acceptable.

(1) Minutes for emulsion to break or (in parentheses) % emulsion remaining after 2.5 hours.

TABLE 5-5

GUM TESTS - HYDROFINED COAL NAPHTHA
mg GUM/100 ml FUEL

Base Fuel	Unleaded			Leaded		
	0.5	1.0	4.0	0.5	1.0	4.0
Vol. % Coal Naphtha						
ASTM Gum						
- Washed	2.2	3.8	1.4	1.4	0.6	1.2
- Unwashed	15.4	16.8	22.6	31.0	29.8	37.8
Copper Beaker Gum	11.8	13.8	14.6	13.2	106.4	17.6
Potential Gum	26.8	109.0	25.2	16.6	38.2	34.2
110°F Storage Stability						
4 Weeks - Washed	1.5	2.7	3.0	0.4	0.7	0.2
- Unwashed	34.1	16.3	40.3	31.4	44.0	35.5
6 Weeks - Washed	11.7	3.5	3.2	1.0	1.0	2.0
- Unwashed	25.0	36.7	22.1	44.7	35.7	31.0
8 Weeks - Washed	10.5	9.5	6.0	1.5	2.2	3.2
- Unwashed	63.0	32.0	31.9	33.6	35.6	43.5

To confirm results obtained with raw Illinois naphtha, stability tests were made with raw Wyodak naphtha cut to 275, 300 and 400°F FBP. As shown in Table 5-6, two of the blends exhibited borderline performance at best. Therefore, it appears that none would be recommended for use in motor gasoline.

In summary, disposition of coal naphtha in gasoline requires that the raw naphtha be partially hydrotreated, caustic treated, and inhibited. Demulsifier is also required. In light of these results, work was directed at burning ECLP coal liquids directly as refinery furnace fuel, the second disposition route.

● Product Disposition: Refinery Fuel

Two sets of ECLP product blending studies were conducted to determine what combination of products were compatible and could be handled as a refinery furnace fuel.

- + Naphtha, solvent and/or VGO as a single stream
- + Separate component blends with low sulfur fuel oil (LSFO) or regular sulfur fuel oil (RSFO).

In the first study, blends containing both naphtha and VGO were incompatible over a wide range of compositions (see October-December, 1977 Quarterly Technical Progress Report [FE-2893-7] for details). A blend containing only solvent and naphtha showed very low sediment content as expected. Previous studies had shown that blends of 400/800°FVT solvent

Table 5-6

Gum Tests - Wyodak Raw Coal Naphtha in Unleaded Fuel
mg Gum/100 ml Fuel

Composition, Vol. %	0.5	1	4	0.5	1	4	0.5	1	4	0.5	1	4	0.5	1	4	0.5	1	4
250°F Naphtha																		
300°F Naphtha																		
400°F Naphtha																		
Heat Soaked Steam Cracker Naphtha										5	5	5				5	5	5
Tests																		
ASTM Gum	4.4	1.4	0.6	2.4	3.2	4.4	4.6	1.0	2.2	2.0	1.6	1.4	4.6	5.2	20.8	4.4	5.2	14.6
- Washed	38.0	26.0	97.4	23.0	31.4	56.6	22.2	44.0	19.8	8.4	11.8	27.6	26.6	12.4	79.4	26.2	11.4	23.0
- Unwashed	17.4	14.4	18.0	7.4	16.0	16.0	11.5	10.2	12.0	12.0	11.0	15.6	17.6	18.8	32.2	15.4	19.2	34.0
Copper Beaker Gum	35.0	36.3	46.8	36.6	43.4	50.9	55.4	17.2	39.6	36.8	29.6	33.0	27.1	23.4	61.6	29.0	25.2	51.6
Potential Gum																		
110°F Stability																		
- 4 Weeks - Washed	3.0	3.4	1.9	1.9	8.7	3.2	2.8	1.3	3.2	2.4	2.8	2.6	6.2	6.6	19.7	4.0	4.9	17.9
- Unwashed	25.4	42.5	39.7	32.0	53.7	42.7	11.5	13.9	15.6	48.6	13.2	27.0	17.2	17.9	34.4	14.6	20.4	28.7
- 6 Weeks - Washed	1.9	3.5	3.7	3.1	4.2	5.5	2.7	3.4	4.6	3.5	2.7	4.1	6.2	5.9	17.4	6.6	5.8	17.3
- Unwashed	31.0	47.4	37.9	26.0	34.5	32.7	11.8	18.1	26.1	20.8	12.6	42.9	15.4	17.4	35.1	17.6	14.4	33.0
- 8 Weeks - Washed	4.0	12.9	5.1	4.7	9.8	5.6	4.0	4.7	7.5	2.7	3.8	5.9	6.0	8.5	15.0	8.3	6.6	24.5
- Unwashed	26.5	53.5	27.9	30.9	72.0	52.4	13.6	20.1	26.7	13.6	14.3	37.6	15.6	17.4	31.3	19.7	15.4	36.3

and 800/1000°FVT VGO were compatible. All other blends containing 5 to 20% VGO with or without solvent had copious amounts of sediment. Based on these studies, therefore, a single stream is restricted to naphtha/solvent or solvent/VGO.

In the second blending study, coal liquid streams were blended into typical refinery fuel oils to determine their compatibility. Between 0.5 and 10 wt.% of either 300/400°FVT coal naphtha or 400/800°FVT solvent caused no incompatibility in either a LSFO or RSFO.

Previous studies had shown that 800/1000°FVT coal liquid VGO was incompatible with petroleum fuel oil. However, with certain very aromatic petroleum streams, partial compatibility was demonstrated. In view of this, it was felt that a coal VGO/aromatic petroleum stream blend might be compatible in a petroleum fuel oil. Blends (50/50) of VGO with two of the aromatic petroleum streams displayed reasonable compatibility with LSFO and RSFO at concentrations up to 3-5%. Followup studies have indicated that VGO may be blended with each of the two aromatic refinery streams over a wide range of concentrations. Blends were prepared with 10, 25, 50, 75 and 95 wt.% Illinois VGO in each of the two candidate streams. These blends were checked visually for incompatibility by examining films under 72X magnification. With one refinery stream the VGO blended easily at 200°F; with the other the blending temperature required a more careful blending technique to obtain homogeneous solutions. This technique required adding heated VGO (>200°F) to the heated refinery stream (>240°F) while mixing.

As a result of these studies, the following disposition schemes are planned for ECLP products:

- + Naphtha and solvent blended for use in a dedicated furnace
- + VGO blended in one or both of two aromatic refinery streams (compatibility demonstrated) with the option for disposal of the blend into RSFO or LSFO at concentration of 5% or lower.
- Flocculation Additive Selected for ECLP Coal Fines/Water Stream Disposition

In addition to liquid products ECLP will send a coal fines/water slurry (2% coal) to the adjacent Exxon Baytown refinery for disposition. Laboratory studies have indicated that "dry" coal fines from coal crushing, sizing and drying will float on the surface of the pond and affect the downstream refinery effluent water treating system.

Tests with a number of wetting agents, demulsifiers and flocculants indicated that an additive solution to the settling problem is feasible. The tests showed that a high molecular weight cationic flocculant effectively settled both Illinois and Wyoming coal fines from a water slurry. The optimum flocculant concentration was in the range of 10 to 100 ppm with the more oxygenated Wyoming coal requiring less additive. Since the additive consumption would be less than 10 gallons per stream

day, no additional screening or optimization studies were conducted. Since tests with petroleum oil/water mixtures insured no problems would occur in the refinery effluent water treating system, this flocculant is recommended as an acceptable solution for disposing of the ECLP coal fines/water stream to the refinery settling ponds.

5.2 Coal Naphtha Hydrotreating

The currently planned end use for EDS coal naphthas is catalytic reforming to make gasoline. Although the 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 considerable hydrotreating relative to a petroleum derived naphtha to meet reformer feed specifications.

Initial scoping studies indicated that two-stage hydrotreating may be required to achieve the necessary contaminant removal 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 have demonstrated that reformer feed specifications can be achieved in a single-stage hydrotreater. In addition, the minimum hydrotreating conditions required have been quantified along with the associated hydrogen consumption.

Four coal naphthas were selected for 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 associated with the sale of recovered phenols versus the lower-valued disposition to mogas as aromatics. Inspection data on each of the naphtha feedstocks are summarized in Table E-1 (Appendix E of this report).

Based on previous studies, which indicated nitrogen removal to be limiting, a Ni/Mo catalyst was chosen for the upgrading studies. At a nitrogen target of 1 wppm all other reformer specifications were met; in fact, due to extensive desulfurization in achieving the nitrogen spec, either Pt or bimetallic reforming is an option. Minimum hydrotreating conditions to meet either Pt or bimetallic reformer feed specifications are summarized in Table 5-7.

TABLE 5-7

HYDROTREATING OF COAL DERIVED NAPHTHAS

Target: <1 wppm Nitrogen
 Temperature: T°F-RV = 238

Feed	Pressure, psig	Maximum LHSV, % of RV	H ₂ Consumption SCF/B
Illinois Raw	800	132	620
Illinois Caustic Washed	700	368	300
Wyodak Raw	800	66	650
Wyodak Caustic Washed	700	159	300

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

The Wyodak naphthas were more difficult to hydrotreat than the corresponding Illinois naphthas, requiring approximately a twofold increase in severity. This is presumably due to the higher heteroatom content of the raw naphthas, the higher final boiling point of the particular sample tested and/or the possibility that the nitrogen compounds in Wyodak naphtha may be more refractory than those in Illinois.

5.3 Phenol Extraction Studies

Phenolic compounds constitute approximately 10 wt.% of the raw, unhydrotreated naphtha produced by the EDS coal liquefaction process. These phenolic 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 volume 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 carried out on Illinois and Wyoming raw coal naphthas using both water and caustic as solvents. 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 based on 100 percent stoichiometric treat rate to form sodium phenolates, whereas water washing removes only 75 percent even at a high water/naphtha ratio of 10/1.

Economic studies have indicated that extraction of phenols from EDS naphtha is more attractive than the base case disposition, namely, hydrotreating plus reforming the phenolic naphtha to mogas (see October-December, 1977, EDS Quarterly Technical Progress Report [FE-2893-7]). Based on projected 1985 investment costs and product revenues, recovery of phenolics for sales could lower the cost of coal liquids by about \$1 to \$2 per barrel.

Two leads have emerged which could provide additional incentives. First, solvent extraction using an oxygenated solvent (e.g., methanol/water blend), which has found application in recovering high purity tar acids from coal tar distillate, might be applicable to EDS naphtha. Equivalent recovery but at a much lower solvent-to-feed ratio could provide incentives relative to extraction with water. Secondly, conversion of lower-valued cresols and some xylenols to the more valuable phenol might be achieved via hydrodealkylation.

A scoping study is in progress to identify promising oxygenated solvents and a mass spectrometer procedure has been developed to analyze raffinate and extract samples derived from EDS naphtha. Followup work with promising solvents will be conducted in a continuous York-Scheibel extraction column. Future plans include updating extraction economics along with assessing the possibility of upgrading cresols to phenol for sales.

5.4 Fuel Oil Combustion Studies

A combustion study was conducted at Products Research Division in Linden, New Jersey in a pilot scale industrial boiler (50 HP Cleaver Brooks - 15 QPH nominal) on the raw 400°F⁺ fuel oil product from the EDS process. This sample was produced by blending raw solvent and vacuum gas oil (VGO) from the CLPP with coker liquid products from the Large Stirred Coking Unit (LSCU). This blend was then filtered in a laboratory filter unit to remove solids present from LSCU. Inspections obtained by Products Research Division on the 400°F⁺ sample are reported in Table E-2 (of Appendix E) along with inspections of a 400/1000°F fuel oil tested for combustion performance during Phase IIIA January, 1976 - June, 1977, EDS Final Technical Progress Report (FE-2353-20).

During these combustion tests, smoke, NO_x and particulate emissions were determined and compared with those of a reference fuel oil. Study results are reported in the October - December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7). Major conclusions are summarized below:

- Both coal liquid fuel oils gave relatively low smoke emissions at a given level of excess air. Emission levels were comparable to a LSFO and better than a RSFO.
- Particulate emissions were lower than those measured for a RSFO and lower than those expected for a LSFO.

- NO_x emissions were consistent with those expected based on the fuel oil nitrogen content. At 15% excess air, the 400°F⁺ fuel oil (0.81% N) gave 600 ppm NO_x vs. 500 ppm for the 400/1000°F (0.55% N) and 470 ppm for a petroleum fuel oil containing 0.44% N.
- Although not a primary objective of the study, observations were made on the handling problems due to the incompatibility of coal liquids with petroleum. Any accidental contact between the two types of fuel caused precipitation of copious amounts of sediment.

5.5 Coal Liquid Storage Stability Studies

Two month-long storage stability tests were run on Illinois coal liquids produced in the one ton-per-day CLPP. Tests at temperatures ranging from 100 to 300°F showed that the raw 400/700°F solvent can be stored at temperatures of 100°F for several months with no degradation if air exposure is minimized. The 700/1000°F VGO, however, exhibited significant degradation in air, as measured by Conradson carbon buildup, when stored at 300°F, the temperature required to handle this high viscosity liquid product. A 400/1000°F raw fuel oil product (prepared by blending solvent and VGO in production proportions) could be stored at 100°F for two months without significant changes in product quality. Storage at 210°F, however, resulted in increases in Conradson carbon, sediment and viscosity. Test data are reported in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3).

5.6 Future Program

Product quality studies will concentrate on areas outlined below to provide information regarding end-use application of EDS liquid products. Tests will be conducted with raw products from liquefaction along with upgraded products to define the most promising end-use applications.

- Stability

Limited storage stability information with Illinois-derived products has pointed the need for a broader-based test program. Basically, several types of storage need to be considered.

- + Long term storage at ambient temperatures for stocks that do not require heating (e.g., naphtha, 350/650°F distillate)
- + Storage for several months or longer to simulate fuels handling from plant to consumer at the pumping temperature required for fuel oil blends (e.g., 350/1000°F, 350°F⁺, 650°F⁺)

- + Short term storage at the highest temperatures and longest times anticipated during either storage, processing or final end use for fractions such as 350/650°F, 650/1000°F, 350°F⁺, 650°F⁺, and 1000°F⁺.

Tests are planned with both Illinois and Wyoming products from CLPP and LSCU. To be included at a later date are hydrotreated 400/1000°F and 400°F⁺ fractions currently being prepared at the Baton Rouge Laboratories.

Should stability of some fractions be a problem, as preliminary data suggest, followup work to improve stability (e.g., additives, special handling procedures, etc.) would be initiated. In fact, a scoping study with additives currently used for petroleum has been included as part of the stability tests described previously.

- Compatibility

The incompatibility of Illinois heavy coal liquids with petroleum fuels has been established. A broader-based set of tests, including tests with distillate stocks, will be conducted with various petroleum stocks, such as outlined below.

<u>Fraction</u>	<u>Petroleum Test Stock</u>
350/650°F	No. 2 fuel oil; light fuel oil
350/1000°F; 650/1000°F	Heavy fuel oil; special refinery streams (high aromatic content)
1000°F ⁺	Residual fuel oil; special refinery streams
350°F ⁺	Residual fuel oil

Included in the program will be tests on cross-compatibility (i.e., mixing of liquids derived from different coals) to insure that a processing facility could handle feed blends from different regions without the need for blocked operations. In addition, tests will be made with possible common solvents so that if compatibility were a problem, the same facility could process petroleum and coal-derived liquids at least in blocked operations.

Compatibility studies will also be made with hydrotreated fuel oil fractions. Should hydrotreated coal liquids be compatible with petroleum fuels, as preliminary data suggest, then the minimum hydrotreating severity to insure compatibility needs to be defined.

- Combustion Studies

Wyodak 350/1000°F and 350°F+ materials are available from CLPP/LSCU operations and will be tested in the 50 HP Cleaver-Brooks boiler to assess NO_x, smoke, particulate and PNA emissions, similar to studies with Illinois fuel oil blends.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

Bottoms Processing Research

6. Coking and Gasification Studies

6.1 Bench-Scale Coking Studies

● Bench Coking of Coal Liquefaction Bottoms

The bench-scale coking unit was used to perform screening studies on various EDS coal liquefaction bottoms; the yield data were used to select feedstocks for more definitive evaluation in the Continuous Stirred Coking Unit (CSCU). Descriptions of the bench coking unit and experimental procedure were given in the January-June, 1976, EDS Quarterly Technical Progress Report (FE-2353-2).

Bottoms produced from Wyodak and lignite coal were coked in the 1-gram-per-test bench coking unit at RV+70°F and RV+270°F. The screening-quality yield data indicated that the net coking liquid yields (1000°F⁺ boiling material) were not greatly affected by the liquefaction conditions at which the bottoms were produced. The results of this study were presented in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3).

● Coker Feedstock Evaluation

A study using the bench-scale coking unit to characterize coker feedstocks was initiated. The objective of the study is the development of a bench-scale means of predicting coking behavior of various feeds. This program has involved extending the bench coking procedure from simply providing yield data to producing coke and liquid for subsequent evaluation. Validation studies have established that bench coking products have chemical and physical properties similar to those of cokes and liquids produced in larger coking pilot units. The cokes produced in the bench unit are now being tested for gasification reactivity, slagging tendency, and particle integrity in other bench-scale tests.

● Coking of Mixtures of Coal and Liquefaction Bottoms

Coking of coal in admixture with liquefaction bottoms has been identified as a potentially attractive source of supplemental EDS process fuel. A bench coking study of Illinois, Wyodak, and North Dakota lignite coals and their respective liquefaction bottoms was performed. The viscous and storage stability properties of these mixtures were investigated. The results of this study were presented in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3).

The observed liquid yields from bench coking of coal and liquefaction bottoms in admixture is about the same for most conditions studied as the additive yields from coking coal and bottoms. Although a previously reported synergistic effect was not found in this study, it appears that the coking of coal or coal/bottoms mixtures provides a means of supplying supplemental EDS process fuel. The bench coking liquid yields for the three coals were compared to yields from the Fischer Assay test. Fischer Assay is a technique for assessing the potential pyrolysis liquid yield of coal. It appears that the Fischer Assay is conservative in predicting the amount of liquids produced in coking coal.

Viscosity experiments demonstrated that addition of coal to liquefaction bottoms increases the viscosity for all three coals studied. The mixture viscosities become prohibitively high in the 20-30 wt.% coal range. Daily viscosity measurements on heat-soaked mixtures indicated that coal/bottoms mixtures are less stable than liquefaction bottoms alone.

● Bottoms Viscosity Reduction Study

A program was conducted to study the effects of fluxing oils on the viscosity and storage stability of coal liquefaction bottoms. The objectives of the study were to identify methods of reducing the viscosity of bottoms if needed for coking pilot unit operability and to provide a data base for evaluating various bottoms handling and storage schemes.

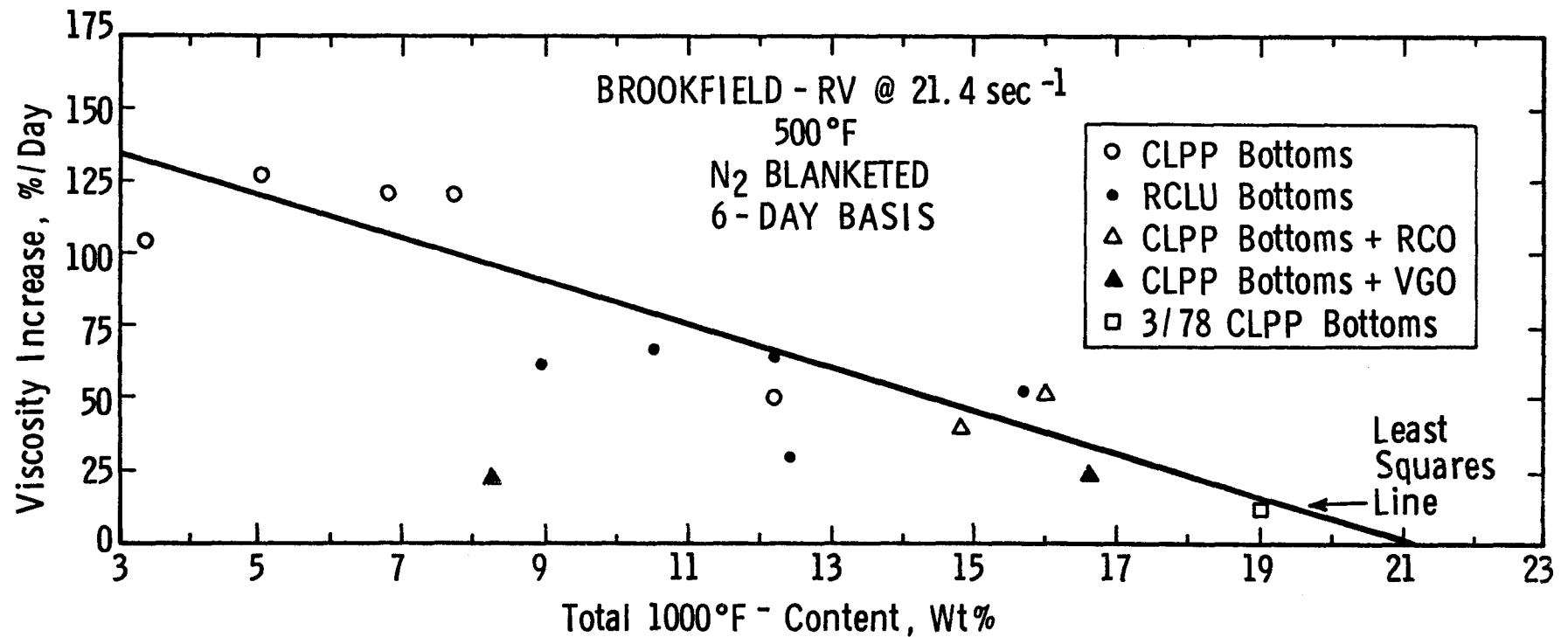
The viscosity experiments consisted of diluting bottoms from the 1 T/D Coal Liquefaction Pilot Plant (CLPP) with Raw Creosote Oil (RCO: boiling range 400-1000°F), Light Creosote Oil (LCO: naphthalene-free RCO, boiling range 450-600°F), and Vacuum Gas Oil from CLPP (VGO: boiling range 700-1000°F). The storage stability of bottoms and bottoms/fluxing oil mixtures was characterized by the percent/day viscosity increase observed in a sample maintained at 500°F for one week.

For Wyodak liquefaction bottoms, which typically have high initial viscosities, there is a dramatic reduction in viscosity for even relatively small additions of fluxing oil (5%). Wyodak bottoms have poor thermal stability. At 500°F, viscosity increases of 50 to 100%/day are typical for Wyodak liquefaction bottoms (3-12% 1000°F⁻ material). For storage of a week or more at 500°F, at least 20% total 1000°F⁻ material must be present to reduce viscosity increases to ≤10%/day. (See Figure 6-1). Thermal stability can be improved by maintaining the bottoms at temperatures below 500°F.

Similar studies with Illinois liquefaction bottoms have indicated that they have lower initial viscosities and better storage stabilities than bottoms produced from Wyodak coal. The addition of fluxing oils, however, effects the same trends with smaller added amounts. The initial viscosity of Illinois bottoms can also be lowered by raising the temperature, but the stability at elevated temperatures (550-650°F) is poor.

FIGURE 6-1

WYODAK BOTTOMS STABILITY: EFFECT OF 1000°F - CONTENT



A computer program was developed to predict the composite viscosity of coal liquefaction bottoms when blended with various solvents. The program, which is based on the Walther Equation, was found to agree with laboratory viscosity data over the temperatures and dilution levels of interest. (See Figure 6-2).

6.2 Coking Yields and Qualities - Continuous Stirred Coking Unit

The Continuous Stirred Coking Unit (CSCU) is used to obtain yield and quality data for coking of liquefaction bottoms. This type of pilot unit was utilized successfully in scale-up of yield and quality data for the FLUID COKING* process for petroleum. In the FLUID COKING process steam is generally used as the medium to fluidize the coke particles. To fluidize a short bed in a pilot unit would require a steam to feed ratio much higher than required in a commercial coker. Yield data from such a pilot plant could not be used in scale-up since steam to feed ratio is an important process variable. The stirred coker overcomes this problem by mechanically agitating the coke bed to simulate fluidization, keeping steam to feed ratio as an independent variable. Yield data can thus be obtained under conditions simulating commercial operation.

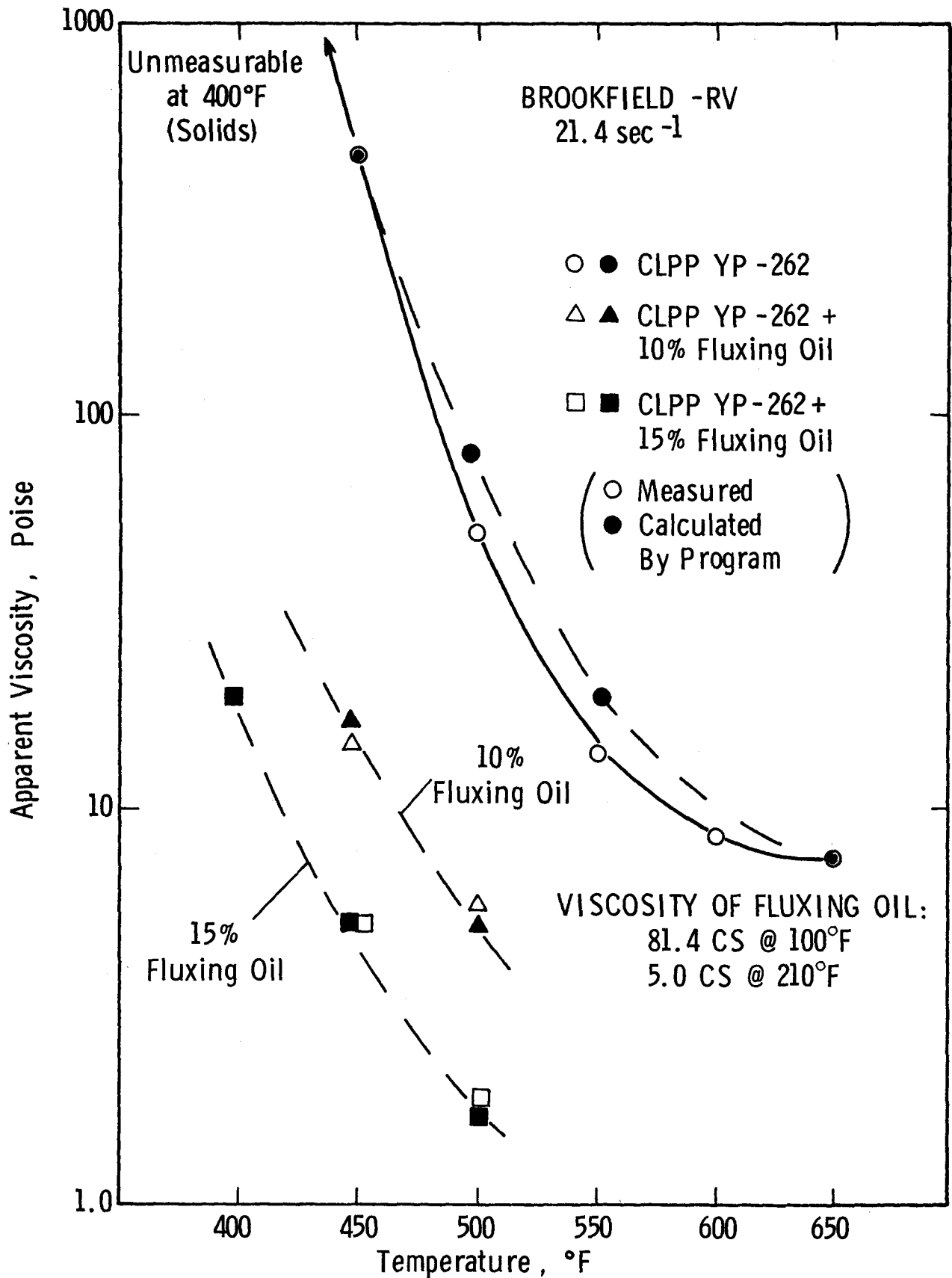
This report presents data from CSCU runs which were carried out to investigate the effect of coking temperature and liquefaction severity on coking yield. These data supplement those obtained previously in the EDS program. During this reporting period a screening study was made on Illinois coal liquefaction bottoms produced at 840°F and 40 minutes liquefaction residence time. The results of this study confirmed an increase in coker liquid yield for Illinois bottoms produced at 40 minutes residence time over those produced at 25 minutes residence time at 100°F above base coking temperature.

The data base for coking of Wyodak coal liquefaction bottoms was expanded. Wyodak bottoms from liquefaction residence times of 40, 60, and 100 minutes have been processed in the CSCU. Coking yields for Wyodak bottoms are lower than for Illinois bottoms. Results of one run suggest an effect of liquefaction residence time on coker liquid yield from Wyodak bottoms analogous to that found for Illinois bottoms. In the case of Wyodak bottoms, the increase in liquid yield was observed for bottoms produced from 100 minutes liquefaction residence time and coked at 100°F above base coking temperature.

An initial screening study of coking yields from Pittsburgh No. 8 (Ireland) coal liquefaction bottoms was completed. These bottoms were produced at 100 minutes liquefaction residence time. Coker liquid yields from these Pittsburgh bottoms are comparable to those from 40-minute Illinois bottoms.

*Service Mark

FIGURE 6-2
TEMPERATURE/FLUXING OIL EFFECT ON ILLINOIS BOTTOMS VISCOSITY



Besides the runs completed in conjunction with the above mentioned studies, two CSCU runs were completed on 1000°F⁺ coker product liquid from Wyodak bottoms. The objective of these runs was to investigate product yields from recycle coking of heavy coker liquids. The analytical work on these runs is not complete and the results will be presented in the next quarterly report.

- Illinois Coal Liquefaction Bottoms

Work has been completed on a coking yield screening study of CLPP Illinois bottoms produced at liquefaction conditions of 840°F and 40 minutes residence time. Previous results from coking similar bottoms indicated a maximum in net liquid yield occurred at 100°F above base coking temperature. Detailed information on the recent run, YT107, is presented in Appendix F. The detailed data on the previous runs of this study, YT93-97, are presented in Appendix D of the January-March, 1978, EDS Quarterly Technical Progress Report (FE-2893-12). A summary of the yields is presented in Table 6-1. From the data in Table 6-1, the following conclusions can be drawn. The recovered 1000°F⁻ liquid decreases as coking temperature increases. The 1000°F⁺ liquid recovery is significantly larger at 100°F above the base coking temperature; such that the total liquid yield, 1000°F⁺ plus 1000°F⁻, is a maximum at this temperature. Increased reactor pressure does not result in liquid yield debit at base coking conditions.

A plot of net liquid yield versus coking temperature is presented in Figure 6-3 for all the Illinois coal liquefaction bottoms processed in CSCU. At 100°F above base coking temperature, all of the data points for bottoms produced at 40 minutes liquefaction residence time lie above those for bottoms produced at 25 minutes residence time. This liquefaction residence time effect is not discernable within the scatter of the data at base coking temperature.

The impact of the interaction between increased liquefaction residence time and coking at RV+170°F is estimated in Table 6-2. This table compares coking yields at 100°F above base coking temperature on bottoms produced at 40 minutes liquefaction residence time to base case coking yields on bottoms produced at 25 minutes liquefaction residence time. From Table 6-2, it appears that about 2% more liquids on coal can be obtained by increasing the coking temperature 100°F at the longer liquefaction residence times.

TABLE 6-1

SUMMARY OF CSCU YIELDS ON CLPP ILLINOIS BOTTOMS - YP263

- Yields Expressed as Weight Percent on Ash-Free 1000°F⁺ Bottoms
- Computed Using Average Feed 1000°F⁻ Content - 3.1% ± 1.2

Run	CONDITIONS		YIELDS			
	Temperature °F - RV	Steam Feed Ratio, %*	Coke	1000°F ⁺ Liquid	1000°F ⁻ Liquid	Gas
93	259	31	63.4	17.0	3.4	16.2
94	178	40	62.0	25.0	5.9	7.1
95	78	29	66.4	20.2	6.9	6.5
96	76	35	65.0	19.9	9.0	6.1
97**	88	20	65.8	16.9	9.3	8.0
107	160	38	57.8	28.2	5.2	8.8

*Steam plus nitrogen - 25 nominal

**High Pressure Run - 20 psig

Average Liquid Yield at base coking conditions 27% ± 1

TABLE 6-2

ESTIMATE OF EFFECT ON COKING YIELD FROM INCREASING
LIQUEFACTION SEVERITY AND COKING TEMPERATURE

● Illinois Bottoms

Liquefaction Residence Time, Minutes	25	40
Coking Temperature, °F-RV	70	170
Coking Yield, Wt% on Coal	11*	13**

*Average of thirteen runs

**Average of four runs

FIGURE 6-3

CSCU YIELD VS. COKING TEMPERATURE

- Illinois Bottoms
- Nominal Coking Residence Time and Steam Rate
- Solid Symbols - Bottoms Produced at 840°F, 25 Minutes
- Open Symbols - Bottoms Produced at 840°F, 40 Minutes

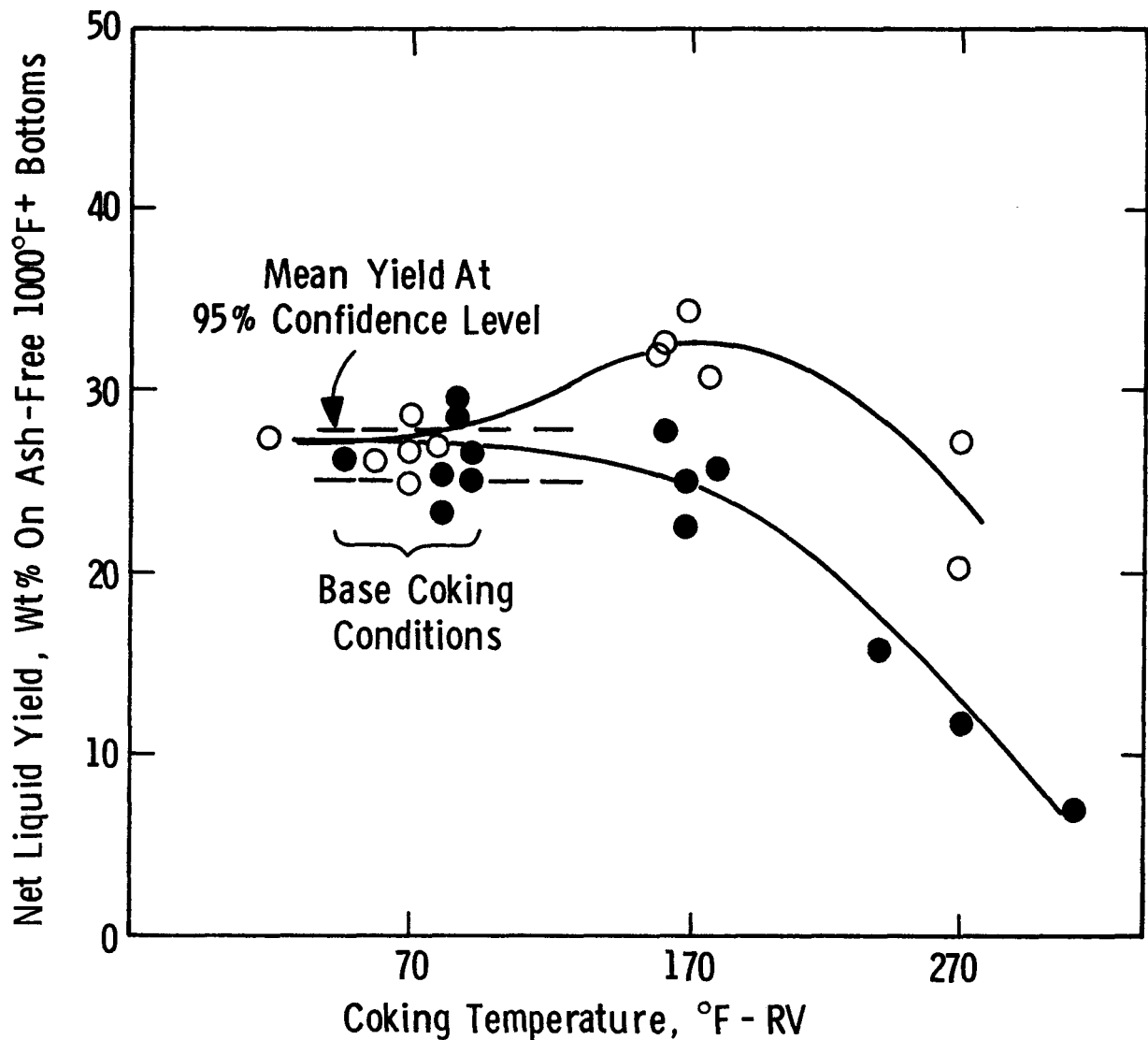


TABLE 6-3

SUMMARY OF COKING YIELDS FROM WYODAK BOTTOMS

• Continuous Stirred Coking Unit

CSCU Run	Coking		Liquefaction			Yields, wt.% on Ash Free 1000°F+ Feed			Yields, wt.% on 1000°F+ Bottoms			See Note
	Temperature °F - RV	Steam/Feed Ratio	Unit	YP	Res. Time	Coke	Liquid	Gas	Coke	Liquid	Gas	
110	73	25	RCLU	432/433	100	75.8	21.8	2.4	80.1	17.9	2.0	
109	158	25	RCLU	433	100	68.2	27.4	4.4	73.9	22.5	3.6	
108	67	25	RCLU	432	100	73.6	22.7	3.7	78.9	18.2	2.9	
102	71	95	RCLU	448	60	62.2	32.4	5.4	68.7	26.9	4.4	
101	95	20	RCLU	448	60	70.9	23.3	5.8	75.8	19.3	4.8	
100	70	31	RCLU	81	60	68.7	25.5	5.8	75.3	20.1	4.5	1
99	80	24	RCLU	450	60	75.1	19.7	5.3	79.0	16.6	4.4	
98	78	29	RCLU	451	60	66.8	26.7	6.5	72.1	22.5	5.5	2
92	270	25	CLPP	238	40	68.9	11.4	19.7	73.6	9.7	16.7	
91	270	25	CLPP	238	40	67.1	13.7	19.2	71.7	11.8	16.5	
89	167	25	CLPP	238	40	72.0	18.7	9.3	76.2	16.0	7.9	
88	67	90	CLPP	238	40	73.2	22.2	4.6	77.4	18.7	3.9	
87	72	25	CLPP	238	40	73.1	21.7	5.2	77.3	18.3	4.4	
86	-4	25	CLPP	238	40	73.9	21.2	4.4	78.0	18.3	3.7	
85	171	25	CLPP	238	40	72.4	16.4	11.2	76.8	13.8	9.4	
84	73	25	CLPP	238	40	70.5	24.1	5.4	75.1	20.3	4.6	

1 SO₂ Pretreatment of Coal

2 106% Material Balance

Average net liquid yield at base coking conditions 23.3 ± 1.8 wt% on ash-free 1000°F+ bottoms.

• Wyodak Coal Liquefaction Bottoms

A summary of the data base for coking Wyodak bottoms is presented in Table 6-3. The detailed data for the most recent runs, YT108-110, are presented in Appendix F. The bottoms for these runs were produced in the Recycle Coal Liquefaction Unit (RCLU) at 840°F and 100 minutes residence time. A plot of net liquid yield versus coking temperature is presented in Figure 6-4. The Wyodak bottoms appear to exhibit coking behavior similar to Illinois bottoms. That is, at 100°F above base coking temperature, bottoms produced at longer liquefaction residence times give higher liquid yields. In the case of Wyodak bottoms, 100 minutes liquefaction residence time is required to produce this effect (versus 40 minutes for Illinois). At comparable coking temperatures, yields from Wyodak bottoms are lower than from Illinois. At base coking conditions, the net liquid yields are 23 wt% and 27 wt% for Wyodak and Illinois, respectively (ash-free 1000°F⁺ feed basis).

Coker liquids from Wyodak bottoms are low in sulfur, 0.2 wt% or less, versus 1-2 wt% for Illinois. As liquefaction conditions become more severe, the 1000°F⁺ liquid product, the major fraction of Wyodak coker liquids, becomes more hydrogen deficient. This is illustrated in Table 6-4.

Two more CSCU runs at base coking temperature have been completed on Wyodak bottoms. These bottoms were produced from 60 minutes liquefaction residence time in the one ton-per-day Coal Liquefaction Pilot Plant (CLPP). The results of these runs will be presented in the next quarterly report.

TABLE 6-4

CARBON - HYDROGEN CONTENT OF 1000°F⁺ WYODAK COKER LIQUIDS

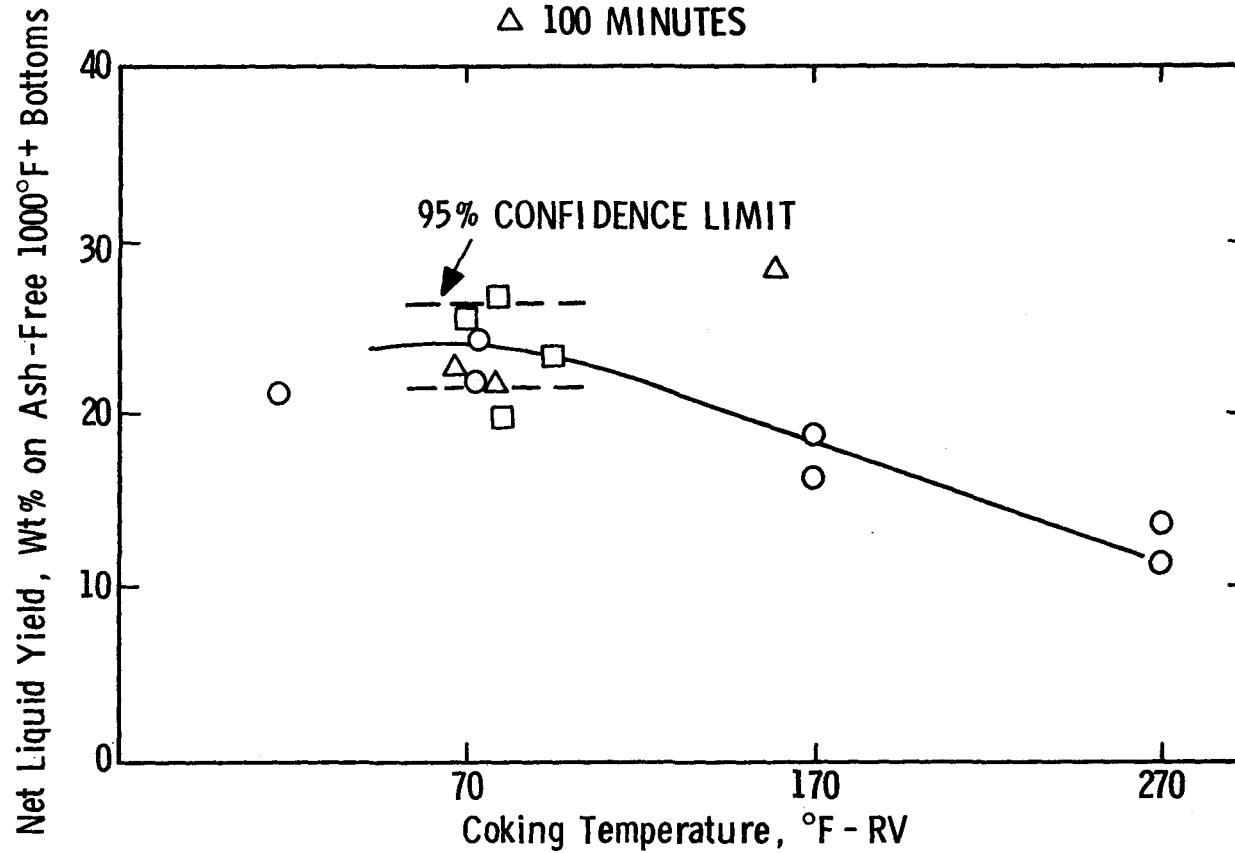
- Coker Liquid Produced At Base Coking Conditions
- 1000°F⁺ Liquid From Microlube Distillation
- Average Values

	Liquefaction Residence Time, Minutes		
	40	60	100
H/C	0.76	0.74	0.70
% C	89.4	89.6	90.1
% H	5.63	5.54	5.30

FIGURE 6-4
LIQUID YIELD VS. COKING TEMPERATURE

- WYODAK BOTTOMS
- NOMINAL COKING RESIDENCE TIME AND STEAM RATE
- LIQUEFACTION RESIDENCE TIME

○ 40 MINUTES
□ 60 MINUTES
△ 100 MINUTES



- Pittsburgh No. 8 Coal Liquefaction Bottoms

An initial coking screening study was completed on coal liquefaction bottoms from Pittsburgh No. 8 (Ireland) coal. These bottoms were produced in the RCLU at 840°F and 100 minutes liquefaction residence time. The detailed data for the CSCU runs, YTL15-118, are presented in Appendix F. The results are summarized in Table 6-5. Coking yields from these Pittsburgh bottoms produced at 100 minutes liquefaction residence time are comparable to Illinois bottoms produced from 40 minutes liquefaction residence time (compare Tables 6-1 and 6-5). On a dry coal basis, coking Pittsburgh bottoms at base coking conditions is estimated to contribute 9 wt% (on dry coal) liquids to the liquefaction process.

- Future Plans

Future runs to be performed on the CSCU will process liquefaction bottoms from Big Brown coal and bottoms from SO₂ treatment of Wyodak coal. In addition, co-processing of coal and bottoms is planned.

6.3 Coking Yield and Qualities - Correlation/Modelling

- Statistical Analysis of CSCU Yields at EDS Design Conditions

The product yield data from CSCU runs completed during EDS process development Phase IIIA on Illinois bottoms at liquefaction and coking conditions close to EDS study design conditions were analyzed statistically. The mean once-through coking yields and the confidence limits were reported in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3). Yield data of duplicate CSCU runs at the base coking conditions on CLPP bottoms from a liquefaction yield confirmation run (YP-263) are compared with these statistical data in Table 6-6. As can be seen, the yield data of single runs and the average of two runs are within the 95% confidence limit ($\pm 2\sigma$) based on CSCU data prior to EDS Phase IIIB.

The elemental analyses of liquid products from these duplicate runs for yield confirmation are compared with previous data in Table 6-7. These analytical data are also within the 95% confidence interval based on previous CSCU data. The agreement of yield data from confirmation runs with previous data enhanced the confidence of coking yields in EDS process development.

- Correlation of Coking Yields with Feed Bottom Properties

As reported in the July-September, 1977, EDS Quarterly Progress Report (FE-2893-3), the best correlation of CSCU coking yields with feed bottom properties was found to be a non-linear regression of the % 1000°F⁻ in feed bottoms and % 1000°F⁺ yield in the liquefaction section with the coke or liquid yields. These correlations were updated by including the data from recent confirmation runs as shown below:

TABLE 6-5

YIELDS FROM COKING PITTSBURGH #8 COAL LIQUEFACTION BOTTOMS

● Nominal Coking Residence Time and Steam Rates

CSCU Run	RCLU YP	Coking Temperature °F - RV	Net Yield				Net Yield			
			Wt% on Ash-Free		1000°F ⁺ Bottoms		Wt% on 1000°F ⁺ Bottoms		1000°F ⁺ Bottoms	
			Coke	1000°F ⁺	1000°F ⁻	Gas	Coke	1000°F ⁺	1000°F ⁺	Gas
115	520	58	67.6	30.2	-1.6	3.9	74.7	23.5	-1.2	3.0
116	521	131	58.3	34.1	3.2	4.5	66.2	27.6	2.6	3.7
117	521	134	59.0	33.1	2.2	5.7	67.0	26.7	1.7	4.6
118	520	49	70.5	26.9	-1.0	3.5	76.9	21.1	-0.7	2.7

TABLE 6-6

COMPARISON OF CSCU PRODUCT YIELDS OF CONFIRMATION
RUNS WITH PREVIOUS YIELD DATA

- Feed Bottoms for Confirmation Run - CLPP YP-263
- Base Coking Conditions
- Yield, Wt% on Ash-Free 1000°F+ Bottoms

	EDS Phase IIIA			Confirmation Runs		
	<u>Mean</u>	<u>σ</u>	<u>σ_2^*</u>	<u>YT-95</u>	<u>YT-96</u>	<u>Mean</u>
Coke	67.8	2.4	1.7	65.7	64.3	65.0
Liquid	26.7	1.8	1.3	27.9	29.6	28.8
Gas	5.5	1.1	0.8	6.4	6.1	6.2

Yield,
Wt% on Ash-
Free 1000°F+
Bottoms

95% Confidence Limit Based on
EDS Phase IIIA CSCU Data

	<u>Single Run</u>		<u>Duplicated Runs</u>	
	<u>Upper</u>	<u>Lower</u>	<u>Upper</u>	<u>Lower</u>
Coke	72.6	63.0	71.2	64.4
Liquid	30.3	23.1	29.3	24.1
Gas	7.7	3.3	7.1	3.9

* σ_2 = Standard deviation of duplicated run mean = $\sigma/\sqrt{2}$.

TABLE 6-7

COMPARISON OF ELEMENTAL ANALYSES OF CONFIRMATION
RUNS WITH PREVIOUS ANALYTICAL DATA OF CSCU LIQUID PRODUCTS

- Feed Bottoms For Confirmation Run - CLPP YP-263
- Base Coking Condition
- Distillation Cuts By Glass-Spiral/Microlube Distillation

Wt%	400-700°F				700-1000°F			
	EDS Phase IIIA*		Confirmation Runs		EDS Phase IIIA*		Confirmation Runs	
	Mean	σ	YT-95	YT-96	Mean	σ	YT-95	YT-96
C	85.5	0.6	86.0	86.0	86.5	0.9	86.9	86.7
H	8.0	0.4	7.8	7.8	6.7	0.2	6.5	6.5
S	0.9	0.1	0.7	0.7	1.0	0.2	0.8	0.9
N	1.0	0.04	0.8	1.0	1.2	0.1	1.4	1.3
O	4.6	0.7	4.7	4.5	4.6	0.9	4.4	4.6
H/C	1.1	0.04	1.1	1.1	0.9	0.04	0.9	0.9

Wt%	1000°F ⁺				Composite			
	EDS Phase IIIA*		Confirmation Runs		EDS Phase IIIA*		Confirmation Runs	
	Mean	σ	YT-95	YT-96	Mean	σ	YT-95	YT-96
C	86.7	0.5	86.7	86.1	86.5	0.4	86.6	86.2
H	5.7	0.2	5.4	5.4	6.2	0.2	6.2	6.1
S	1.3	0.1	0.8	1.1	1.2	0.1	0.8	1.0
N	1.7	0.05	1.7	1.9	1.6	0.1	1.5	1.6
O	4.6	0.2	5.4	5.5	4.5	0.4	4.9	5.1
H/C	0.8	0.04	0.8	0.8	0.9	0.05	0.9	0.9

*Detailed data shown in Table 6-5 of the October-December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7)

$$\begin{aligned} \text{Coke Yield,} &= 94.72 - 0.98 X_1 + X_2(0.0045X_1 - 0.383), \quad \gamma=0.996, S_e=1.6\% \quad (1) \\ \% \text{ on Feed} & \end{aligned}$$

$$\begin{aligned} \text{Liquid Yield,} &= 6.65 + 0.96 X_1 + X_2(0.274 - 0.0034 X_1), \quad \gamma=0.996, S_e=1.6\% \quad (2) \\ \% \text{ on Feed} & \end{aligned}$$

where

X_1 = % 1000°F⁻ in feed bottoms by microcube distillation,
lb/100 lb. bottoms

X_2 = % 1000°F⁺ yield in liquefaction, lb/100 lb. coal.

The standard error of estimates of these regressions are believed to be within the experimental error of CSCU runs. These empirical correlations of feed bottom property and liquefaction severity with coking yields will be useful for interrelating the liquefaction section and coking section in development of the EDS Process Model.

• Correlation of Coking Yields with Coking Conditions

The important process variables for controlling the coking yields are coking temperature, steam/feed ratio and vapor residence time. So far, steam/feed ratio was the only coking process variable which consistently showed an effect on the coking yields from Illinois bottoms, as shown in Figure 6-5. These data were not taken at a constant vapor residence time. The vapor residence time was shorter for the higher steam/feed ratio. Vapor residence time, however, does not contribute to increased liquid yield based on results of a series of CSCU runs at the base coking temperature and a constant steam/feed ratio (25% on feed), as shown in Figure 6-5 of the January, 1976 - June, 1977, EDS Final Technical Progress Report (FE-2353-20).

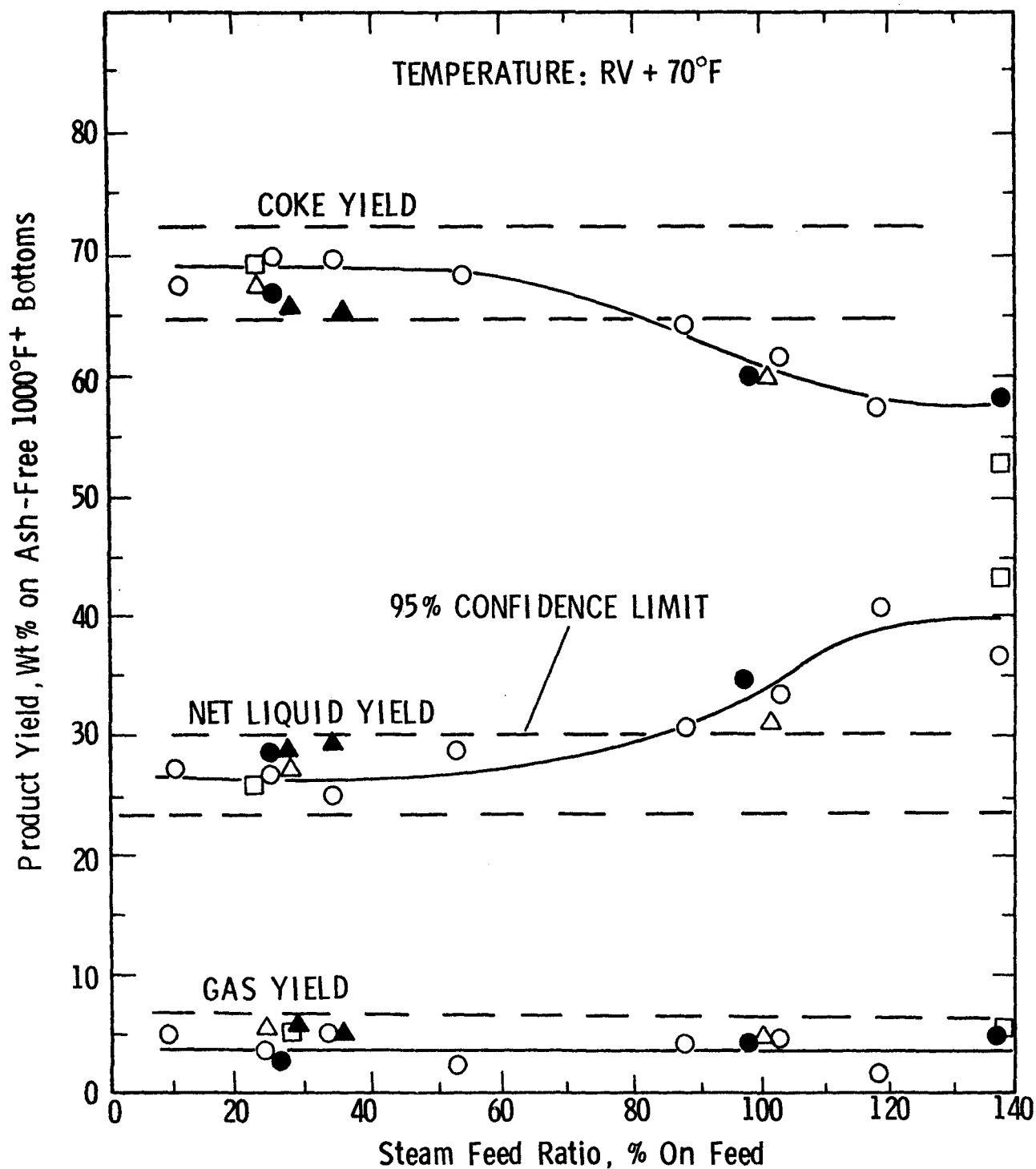
The empirical expressions for correlating the coke and liquid yields with steam/feed ratio were obtained by non-linear regression of the data shown in Figure 6-5. These expressions are combined with Equations (1) and (2) in the preceding section to give an overall expression for predicting coke yield (C) and liquid yield (L) from % 1000°F⁻ in the feed bottoms (x_1), the % 1000°F⁺ bottom yield in liquefaction (x_2) and steam/feed ratio (S):

$$C = [94.72 - 0.98x_1 + x_2 (0.0045x_1 - 0.38)] \frac{0.06S^2 + 1.058S + 0.0022}{0.194S^2 + S + 0.0071} \quad (3)$$

$$L = [6.65 + 0.962x_1 + x_2 (0.274 - 0.0034x_1)] \frac{0.0033S^2 - 0.9222S + 0.0001}{0.24S^2 - S + 0.0073} \quad (4)$$

FIGURE 6-5
STEAM/FEED RATIO SHOWED EFFECT
ON PRODUCT YIELD

- CLPP YP-221 BOTTOMS △ CLPP YP-206/208 BOTTOMS
 ● CLPP YP-207 BOTTOMS □ CLPP YP-220 BOTTOMS
 ▲ CLPP YP-263 BOTTOMS (CONFIRMATION RUNS)

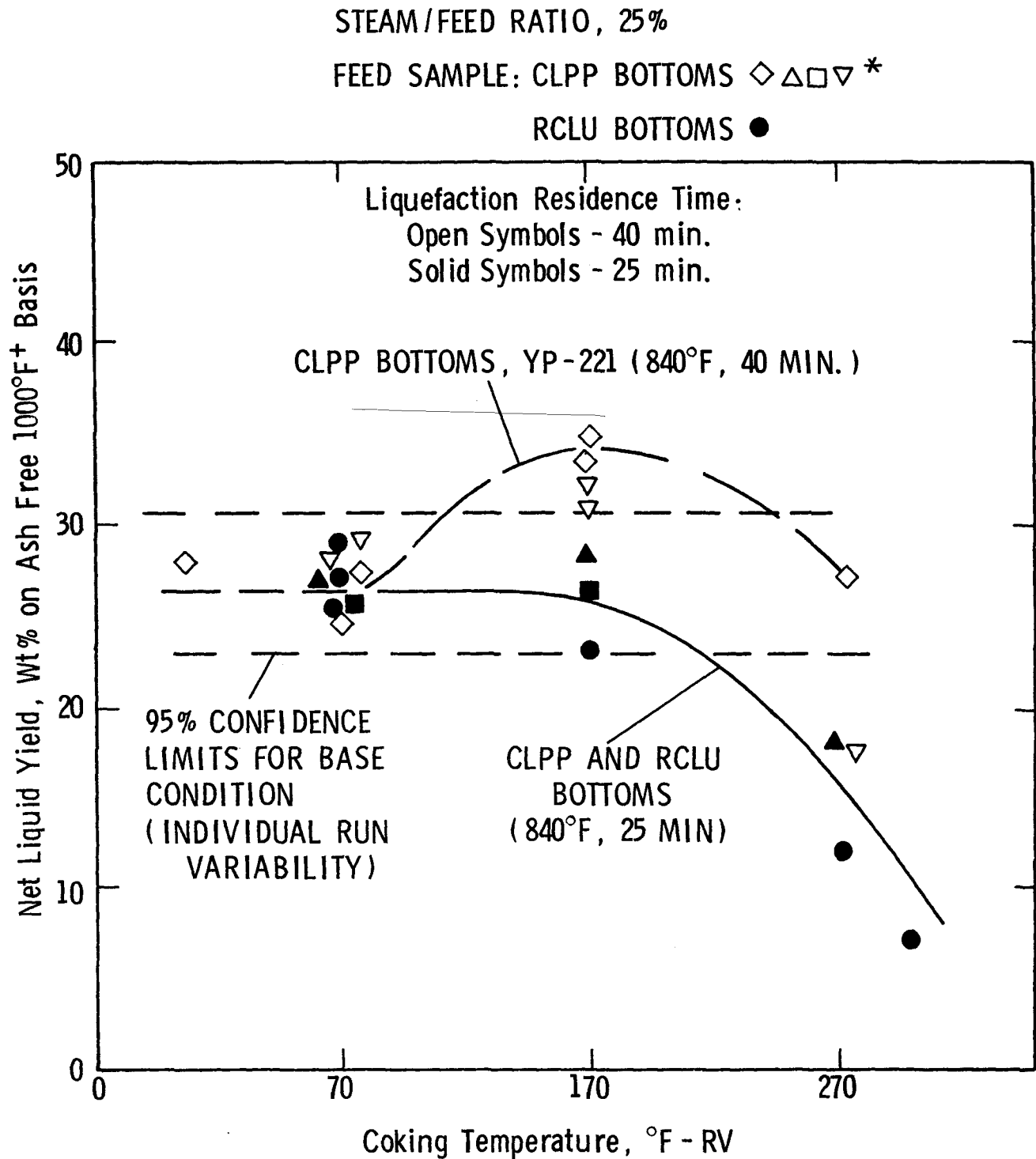


Equation (3) and Equation (4) are useful to interface the liquefaction and coking sections in developing the EDS Process Model. These correlations will be continuously updated as more CSCU data become available.

As discussed in the January 1976 - June 1977, EDS Final Technical Progress Report (FE-2353-20), the liquid yields produced at coking temperatures above the base temperature show a dependence on liquefaction conditions. This is illustrated in Figure 6-6. A maximum liquid yield is shown at 100°F above the base temperature (RV+170°F) for the CLPP YP-221 bottoms produced at the longer liquefaction residence time (40 minutes). CSCU runs on bottoms produced from a CLPP yield confirmation run (YP-262) at different coking temperatures also showed a maximum liquid yield at 100°F above the base temperature as shown in Figure 6-6. The liquid yields of these runs at two different coking temperatures are tabulated in Table 6-8. The t-statistic test showed a significant increase of liquid yield by increasing the coking temperature by 100°F. The confidence limit on the difference of liquid yields at 95% confidence level is estimated to be 2.1-6.6% on feed. The reason for this liquefaction condition/coking temperature interaction with liquid yields is not yet known. An engineering analysis is underway to assess the economic incentives for designing the coker to operate at a higher temperature than the base coking temperature. If the economic study is concluded to be favorable, the temperature effect on liquid yield will be further investigated.

Table 6-9 shows a comparison of elemental analyses of microlube distillation cuts of liquid products from CSCU runs at the base coking temperature and 100°F above. As can be seen, no appreciable difference of 1000°F⁻ liquids was noted. It is consistent that the hydrogen to carbon atomic ratio of 1000°F⁺ liquid was somewhat decreased at the higher coking temperature due to the deeper cut into the higher boiling material. The gas analyses of these runs at different coking temperatures were similar, as shown in Table 6-10. However, it is noteworthy that there is a small decrease in C₂ and C₃ gases with an increase of CO₂ and CO at the higher coking temperature. This might be attributed to the steam reforming reaction for the heavier hydrocarbon gases in the dilute phase at the higher coking temperature.

FIGURE 6-6
COKING TEMPERATURE EFFECT WAS NOT CONSISTENT



* CSCU Data From CLPP Yield Confirmation Runs (YP-263)

TABLE 6-8

COMPARISON OF LIQUID YIELDS AT DIFFERENT TEMPERATURES

- Base Coking Temperature - RV+70°F
- Feed Bottoms - EDS Design Condition
- 25% Steam/Feed Ratio

Feed Bottoms	Liquid Yield			
	% On Feed		% on Ash-Free 1000°F+	
	RV+70°F	RV+170°F	RV+70°F	RV+170°F
CLPP YP-221	24.7	30.6	26.8	34.5
CLPP YP-221	23.3	28.7	25.0	32.8
CLPP YP-263	23.9	26.7	27.9	31.2
CLPP YP-263	25.3	28.7	29.6	32.6
Mean	24.3	28.7	27.3	32.8
σ	0.9	1.6	1.9	1.4
t-statistic*	4.8 (Significant @ 99% C.L.)		4.1 (Significant @ 95% C.L.)	
Confidence Limit of Difference				
95% C.L.	2.1 - 6.6		2.7 - 8.2	
99% C.L.	1.0 - 7.7		1.3 - 9.6	

* $t_{0.01} = 4.6$, $t_{0.05} = 2.8$

TABLE 6-9

COMPARISONS OF ELEMENTAL ANALYSES OF DISTILLATION
CUTS OF CSCU PRODUCTS FROM ILLINOIS BOTTOMS

- CLPP Bottoms Produced Near EDS Design Condition
- Base Coking Temperature - RV+70°F
- Microlube Distillation

Elemental Analyses*	400°F - 700°F		700°F - 1000°F	
	RV+70°F	RV+170°F	RV+70°F	RV+170°F
Wt%				
C	85.6	85.8	87.2	86.9
H	7.8	7.6	6.6	6.4
S	0.8	0.9	0.9	0.9
N	1.0	1.1	1.3	1.3
O	4.8	4.8	4.0	4.5
Atomic H/C	1.1	1.1	0.9	0.9

Elemental Analysis*	1000°F+		Composite	
	RV+70°F	RV+170°F	RV+70°F	RV+170°F
Wt%				
C	86.1	87.5	86.4	87.2
H	5.6	5.3	6.2	5.8
S	1.1	1.1	1.0	1.1
N	1.8	1.9	1.6	1.7
O	4.9	4.2	4.8	4.2
Atomic H/C	0.8	0.7	0.8	0.8

*Average of 4 runs - YT 29, 32, 95 and 96 for RV+70°F and YT 41, 51, 94 and 107 for RV+170°F.

TABLE 6-10

COMPARISON OF GAS ANALYSES OF CSCU RUNS
AT TWO DIFFERENT COKING TEMPERATURES

- Base Coking Temperature - RV+70°F
- Feed Bottoms - EDS Design Condition
- 25% Steam/Feed Ratio

<u>Wt%</u>	<u>RV+70°F*</u>	<u>RV+170°F*</u>
H ₂	9.4	10.2
CO	5.5	7.0
CO ₂	11.5	14.6
H ₂ **	5.0	5.0
C ₁	46.4	44.2
C ₂	15.1	12.1
C ₃	7.1	6.5
	100.0	100.0

*Average of 4 runs for RV+70°F - YT-29, YT-32, YT-95, YT-96;
Average of 3 runs of RV+170°F - YT-55, YT-94 and YT-107.

**Assumed value.

6.4 Large Stirred Coking Unit (LSCU)

The Large Stirred Coking Unit was designed, built and operated to produce barrel quantities of coker liquids from Illinois and Wyodak coal CLPP liquefaction bottoms for product quality studies. A simplified unit flow diagram appeared in the January-June, 1976, EDS Quarterly Technical Progress Report (FE-2353-2). Sufficient coker liquids to fulfill commitments from both Illinois and Wyodak bottoms were produced in the fourth quarter of 1977 and no further runs were scheduled until the fourth quarter of 1978.

Runs with Illinois bottoms feed were usually terminated due to overhead line plugging. Superheated steam purging of the overhead line and line reamers were not successful in extending run lengths. No overhead line plugging problems were experienced with Wyodak bottoms feed even though no overhead line purge steam was used. Wyodak bottoms did, however, initially present feeding problems which were not experienced with Illinois bottoms.

6.5 Bench Scale Gasification - Kinetic Study

- Objectives

The final EDS processing step is the air FLEXICOKING process. It produces liquids and low-Btu gas from the hydrocarbon remaining in coal liquefaction bottoms. Although this process involves an integrated coker and air/steam gasifier, each section is being studied independently on the bench scale. This is done because a relatively large unit is needed for integrated studies and because process variables for each section are more readily studied in separate, noninteracting units.

The short-range objective of the kinetic study is to measure the gasification reactivity with CO₂ or steam of coke produced from Illinois and Wyodak bottoms and compare with that of petroleum coke. This information is useful for establishing the gasifier conditions in pilot unit operations and for the FLEXICOKING process engineering design for the EDS commercial plant. The long-range objective of bench gasification studies is to obtain a data base for a wide range of process variables for the development of a kinetic model for the gasification of cokes from EDS bottoms. This model will be combined with the coking model to complete the FLEXICOKING process model for the EDS process model.

- Experimental Apparatus and Procedure

A description of the minigasifier equipment and procedure was given in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3). Gas flow was changed from upflow to downflow to improve temperature measurement in the bed of coke particles.

A variety of coke feed samples have been tested. These are Illinois and Wyodak bottoms cokes from the 2 B/D Integrated Coking/Gasification Pilot Plant (IKG) and the Large Stirred Coking Unit (LSCU), as well as a petroleum coke which was tested as a reference. The IKG reactor cokes and the reference petroleum coke were -30 mesh particles as received. The LSCU coke was sieved and the -30 + 100 mesh portion was used as the feed sample. The elemental analyses and ash composition of IKG Illinois and Wyodak cokes are tabulated in Table 6-11. Since no detailed analyses of LSCU cokes and the petroleum coke were obtained, only their carbon, hydrogen and ash analyses are listed in Table 6-11.

● Experimental Results

Detailed data of minigasifier runs with CO₂ gasification of IKG Illinois bottoms coke, LSCU Wyodak bottoms coke and a reference petroleum coke are contained in Tables 1-15 of Appendix E in the January-March, 1978, EDS Quarterly Technical Progress Report (FE-2893-12). Additional data of minigasifier runs on these cokes and LSCU Illinois bottoms coke and IKG Wyodak bottoms cokes are tabulated in Tables 1-22 of Appendix G. Each coke was run in CO₂ or steam at two different temperatures and mostly at two different superficial velocities. The testing conditions of these runs can be identified by Run No. in the factorial design shown in Table 6-12.

● Discussion of Results

As illustrated in Figure 6-7, the average rates based on initial carbon showed a consistent trend of decreasing rate with the increase of carbon conversion for all cokes tested. The IKG bottoms cokes from both Illinois and Wyodak coals showed higher gasification rates in steam and CO₂ than the reference petroleum coke. Despite the higher ash content in bottom cokes, it was estimated that the gasification rates on the basis of coke input for bottoms cokes were somewhat higher than that for the reference petroleum coke.

It was also noted that IKG Wyodak coke gave appreciably higher gasification rate than IKG Illinois coke. Wyodak coke ash contains more calcium than Illinois coke ash as shown in Table 6-11. Calcium is known to be a gasification catalyst. Samples of Wyodak bottoms coke before and after a minigasifier run in steam were examined by Scanning Electron Microscope (SEM) to investigate how the calcium was distributed inside the coke particle. Figures 6-8A and 6-8B show the micrographs and Ca mapping by x-ray diffraction on the cross-section of these particles. No indication of migration of calcium during gasification was observed. The even dispersion of calcium on reaction sites may be responsible for the catalytic effect on gasification.

The average gasification rate on initial carbon at 80% carbon conversion is used to provide a single value to represent the intrinsic gasification reactivity for scoping studies of process variables and coke sources. Table 6-13 presents a summary of these gasification rates for all the cokes tested so far at two gasification temperatures. These data were

analyzed by a statistical analysis of variance. Results, as shown in Table 6-14, showed that variation of gasification rates for cokes from different coals and/or different pilot units was statistically significant at 95% confidence level. The superficial velocity showed no significant effect on gasification rate. This indicates that gasification is not diffusion controlled within the range of velocities tested.

The reactivities of these cokes to steam gasification can be ranked on the basis of minigasifier data shown in Table 6-13 as follows:

IKG Wyodak Coke > LSCU Wyodak Coke > IKG Illinois Coke >
LSCU Illinois Coke = Petroleum Coke.

It is evident that the reactivities were a function not only of coal source but also of the coking unit. Wyodak bottoms coke showed higher gasification rate than Illinois bottoms coke for samples from both the IKG and LSCU. It is noteworthy that the gasification rate of IKG Wyodak coke is close to that of IKG Illinois coke at a gasification temperature 100°F lower than that for gasifying Illinois coke.

The appreciable differences of gasification rates of cokes from the two different pilot units (IKG and LSCU) are rather surprising. IKG coke was from the FLEXICOKING process where coke was circulated between the coker and gasifier through the heater. The LSCU simulates the once-through FLUID COKING process without the heater. It is interesting to note that LSCU Illinois coke showed gasification rates similar to the reference petroleum coke which was produced in a commercial FLUID COKING process.

Samples of some feed cokes and gasified residues were examined by scanning electron microscope to investigate whether differences of physical characteristics of the cokes due to the difference in processing may be responsible for this observed difference of gasification reactivity. The micrographs of IKG and LSCU Illinois cokes are illustrated in Figures 6-9A and 6-9B. These two cokes had appreciably different gasification reactivities. However, no significant difference of surface topology was observed. The LSCU coke appeared to be more spherical and have a smoother surface at low magnification (100X), but the surface texture at high magnification (2000X) appeared quite similar.

The surface topology (5000X) of gasified residues from IKG Illinois and Wyodak bottom cokes was strikingly different as shown in Figure 6-10. The micro-melting structure was observed on Illinois coke residue after a minigasifier run in steam at a temperature close to IKG operation. Wyodak coke residue, however, appeared to show agglomeration of microparticles. This observed phenomenon may have impact not only on gasification kinetics, but also on the slagging tendency and particle integrity of different cokes. This will be further investigated.

- Future Plans

Minigasifier runs will be continued to obtain kinetic data for modeling EDS bottoms coke gasification. Kinetic models will be derived separately for steam and CO₂ gasification. Investigation will also be continued to elucidate the different gasification reactivities of cokes from different sources.

TABLE 6-11

ANALYSES OF FEED SAMPLES FOR MINIGASIFIER

<u>Wt% Dry Basis</u>	<u>IKG Cokes</u>		<u>LSCU Cokes</u>		<u>Petroleum Coke**</u>	
	<u>Illinois</u>	<u>Wyodak</u>	<u>Illinois</u>	<u>Wyodak</u>	<u>I</u>	<u>II</u>
C	62.7	67.8	65.3	68.4	87.8	87.3
H	0.4	0.7	2.2	2.4	1.9	2.1
S	1.6	0.2	---			
N	*	0.4	---			
O	---	0.4	---			
Ash	35.7	30.5	23.0	22.7	0.3	0.1

Ash Assay

P ₂ O ₅	0.3	1.3
SiO ₂	52.4	36.8
Fe ₂ O ₃	18.2	6.9
Al ₂ O ₃	19.6	19.5
TiO ₂	0.9	1.3
CaO	3.2	22.8
MgO	1.2	5.8
SO ₃	1.2	1.7
K ₂ O	2.6	0.7
Na ₂ O	0.7	2.4
	<u>100.3</u>	<u>99.2</u>

*Not Analyzed

**Two samples from the same commercial FLUID COKING Unit

TABLE 6-12

TESTING CONDITIONS OF MINIGASIFIER RUNS
(RUN NO. IDENTIFICATIONS)

I. CO₂ Gasification

Temperature, °F: Superficial Velocity, cm/sec:	RV-30		RV-130	
	<u>13</u>	<u>18</u>	<u>13</u>	<u>18</u>
IKG Illinois Coke	453/545*	452/455*	457	456
IKG Wyodak Coke	---	529	---	530
LSCU Wyodak Coke	463	462	465/521*	464
Petroleum Coke	461	459	466	519

II. Steam Gasification

Temperature, °F: Superficial Velocity, cm/sec:	RV-30		RV-130	
	<u>16</u>	<u>20</u>	<u>16</u>	<u>20</u>
IKG Illinois Coke	472	478	510	518
LSCU Illinois Coke	---	522	---	523
IKG Wyodak Coke	---	527	---	528
LSCU Wyodak Coke	477	475	480/520*	57
Petroleum Coke I	479	473	513	58
Petroleum Coke II	---	524	---	525

*Duplicate runs.

TABLE 6-13
INTRINSIC GASIFICATION RATES BY MINIGASIFIER RUNS

- Gm C Gasified/100 Gm Initial C/Hr
- Pressure -- 30 psig

CO₂ Gasification

Temperature, °F Superficial Velocity, cm/sec:	RV-30		RV-130	
	13	18	13	18
IKG Illinois Coke	85/90*	84/85*	38	38
IKG Wyodak Coke	---	130	---	84
LSCU Wyodak Coke	80	82	39/37*	53
Petroleum Coke I	9	12	6	7

Steam Gasification

Temperature, °F Superficial Velocity, cm/sec:	RV-30		RV-130	
	16	20	16	20
IKG Illinois Coke	151	120	38	45
LSCU Illinois Coke	---	44	---	25
IKG Wyodak Coke	---	217	---	108
LSCU Wyodak Coke	160	181	47/97*	64
Petroleum Coke I	50	56	48	55
Petroleum Coke II	---	50	---	40

*Duplicated runs. The reason for the appreciable difference of duplicated runs on LSCU Wyodak @ RV-130°F is not known.

TABLE 6-14

ANALYSIS OF VARIANCE OF INTRINSIC GASIFICATION RATES OF VARIOUS BOTTOMS COKES AT VARIOUS CONDITIONS

- Data in Table 6-13
- 3 - Factorial Analysis
- Sector - Temperature Variation; Row - Superficial Velocity Variation; Column - Coke Source Variations

I. For CO₂ Gasification

<u>Source</u>	<u>Sum of Squares</u>	<u>D.F.</u>	<u>Mean Square</u>
Sectors	4455.56	1	4455.56
Rows	22.56	1	22.56
Columns	19502.69	3	6500.90
Unexplained	1339.62	10	133.96
Total	25320.44	15	1688.03

Sectors F-Ratio = 3.325900000E+01

Degrees of freedom, numerator = 1

Degrees of freedom, denominator = 10

At the five percent level there is a significant difference between the sectors

Rows F-Ratio = 1.68000000E-01

Degrees of freedom, numerator = 1

Degrees of freedom, denominator = 10

At the five percent level there is no significant difference between the rows

Columns F-Ratio = 4.852700000E+01

Degrees of freedom, numerator = 3

Degrees of freedom, denominator = 10

At the five percent level there is a significant difference between the columns

II. For Steam Gasification

<u>Source</u>	<u>Sum of Squares</u>	<u>D.F.</u>	<u>Mean Square</u>
Sectors	18481.50	1	18481.50
Rows	0.67	1	0.67
Columns	49999.33	5	9999.87
Unexplained	13986.33	16	874.15
Total	82467.83	23	3585.56

Sectors F-Ratio = 2.114200000E+01

Degrees of freedom, numerator = 1

Degrees of freedom, denominator = 16

At the five percent level there is a significant difference between the sectors

Rows F-Ratio = 0

Degrees of freedom, numerator = 1

Degrees of freedom, denominator = 16

At the five percent level there is no significant difference between the rows

Columns F-Ratio = 1.143900000E+01

Degrees of freedom, numerator = 5

Degrees of freedom, denominator = 16

At the five percent level there is a significant difference between the columns

FIGURE 6-7
GASIFICATION RATES OF IKG COKES

- MINIGASIFIER RUNS @ RV-30, 30 psig
- ASH CONTENT - 35% IN ILLINOIS COKE
 - 30% IN WYODAK COKE

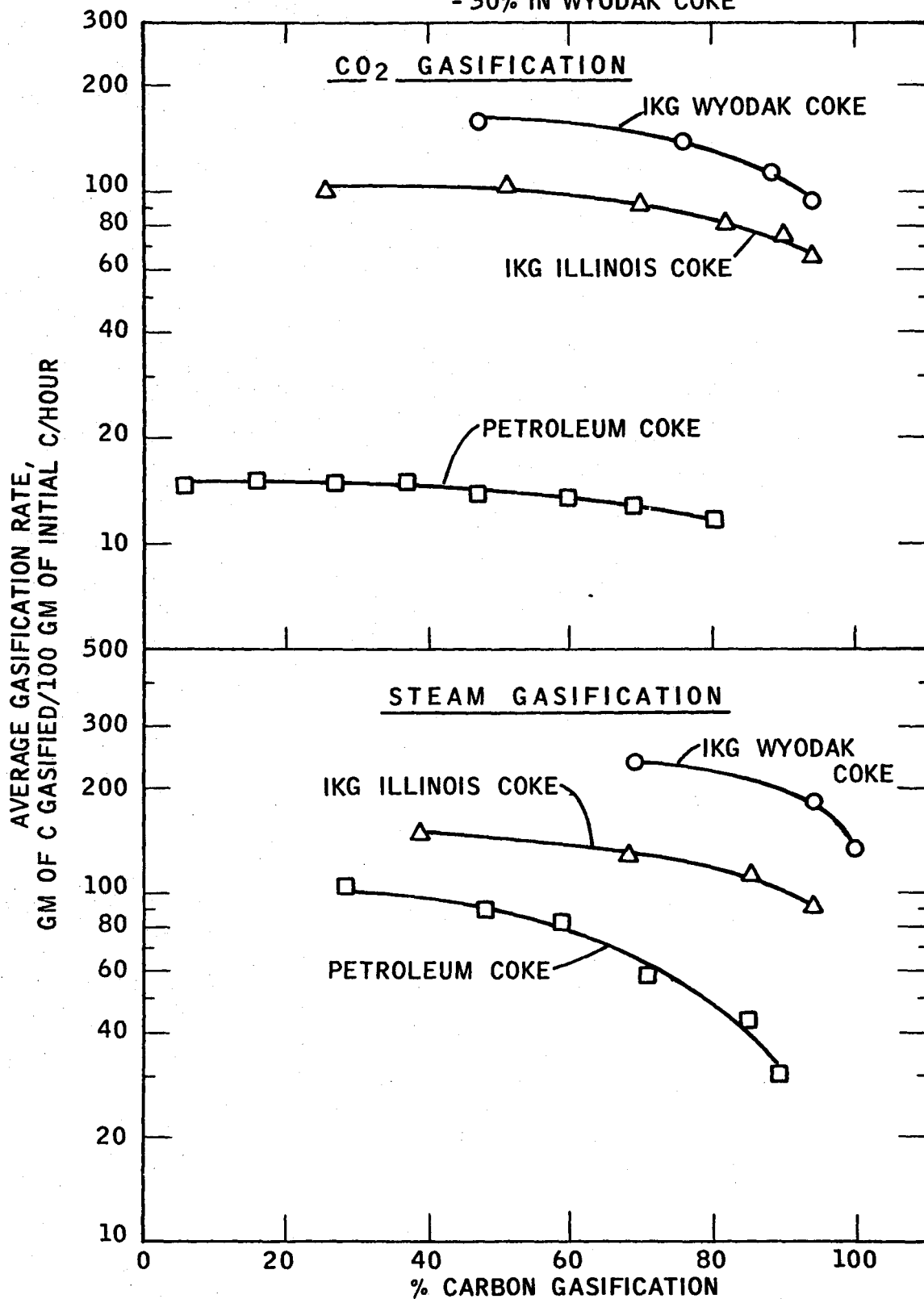


FIGURE 6-8A

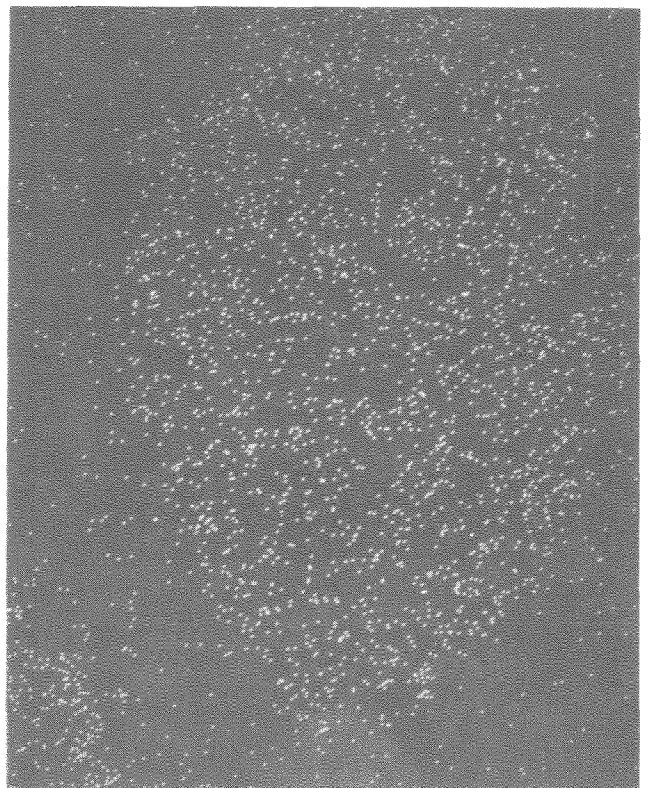
CALCIUM MAPPING OF CROSS SECTION OF WYODAK BOTTOMS
COKE BEFORE MINIGASIFIER RUN

- Steam Gasification @ RV -30°F, 30 psig
- x-Ray Diffraction for Ca-Mapping

BEFORE GASIFICATION



Micrograph, 500X



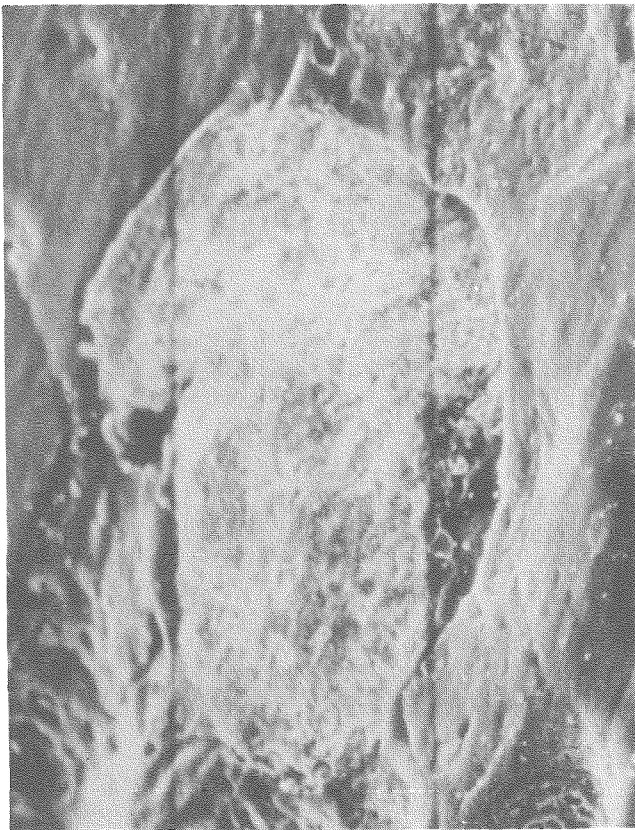
Ca-Mapping, 500X

FIGURE 6-8B

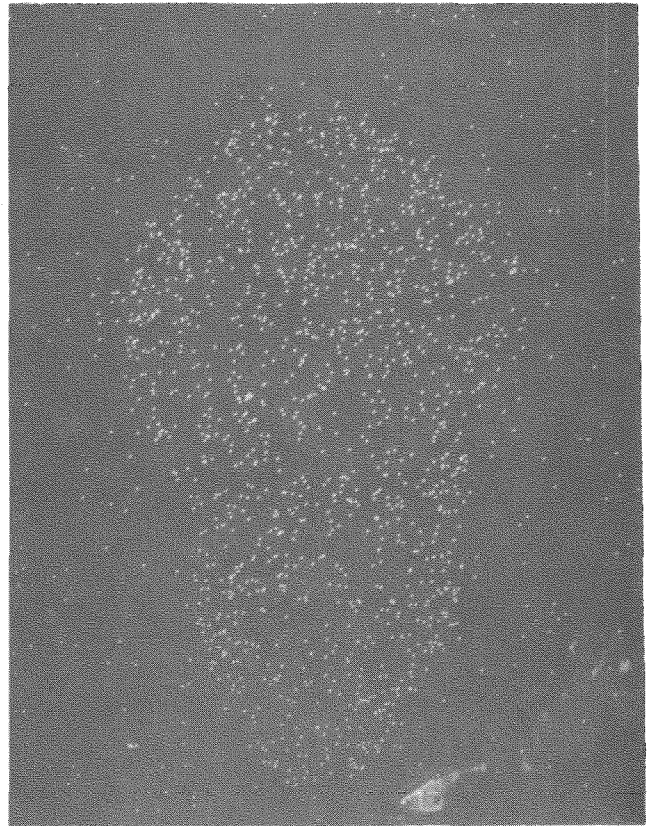
CALCIUM MAPPING OF CROSS SECTION OF WYODAK BOTTOMS
COKE AFTER MINIGASIFIER RUN

- Steam Gasification @ RV -30°F, 30 psig
- x-Ray Diffraction for Ca-Mapping

AFTER GASIFICATION



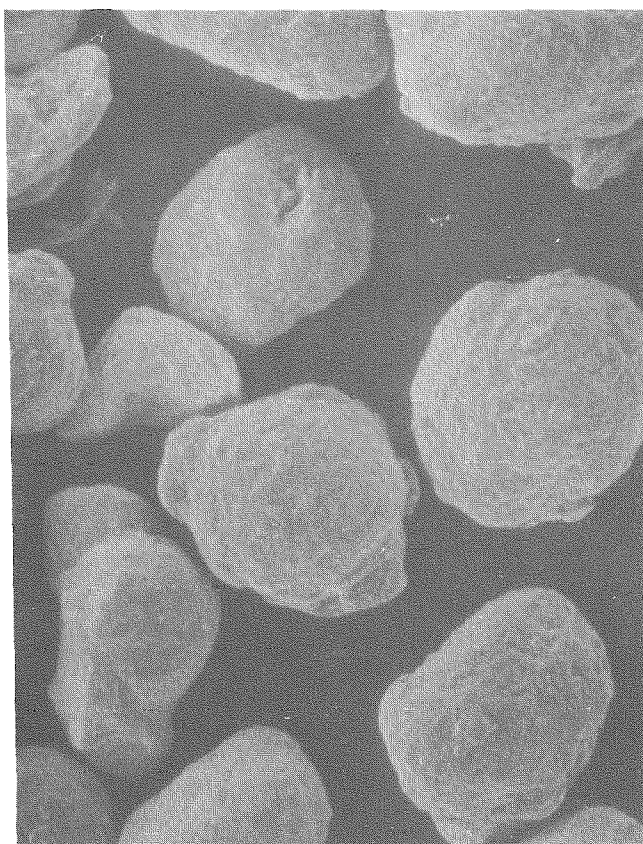
Micrograph, 500X



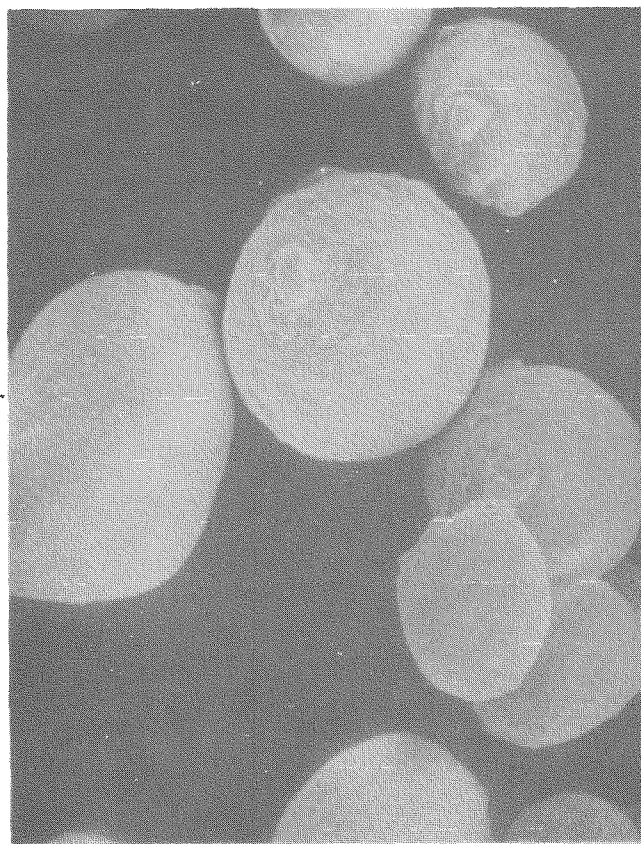
Ca-Mapping, 500X

FIGURE 6-9A

MICROGRAPHS OF BOTTOMS COKE FEEDS FOR MINIGASIFIER RUNS
BY SCANNING ELECTRON MICROSCOPE (SEM)



IKG Illinois Bottom Coke, 100X



LSCU Illinois Bottom Coke, 100X

FIGURE 6-9B
MICROGRAPHS OF BOTTOMS COKE FEEDS FOR MINIGASIFIER RUNS
BY SCANNING ELECTRON MICROSCOPE (SEM)



IKG Illinois Bottom Coke, 2000X

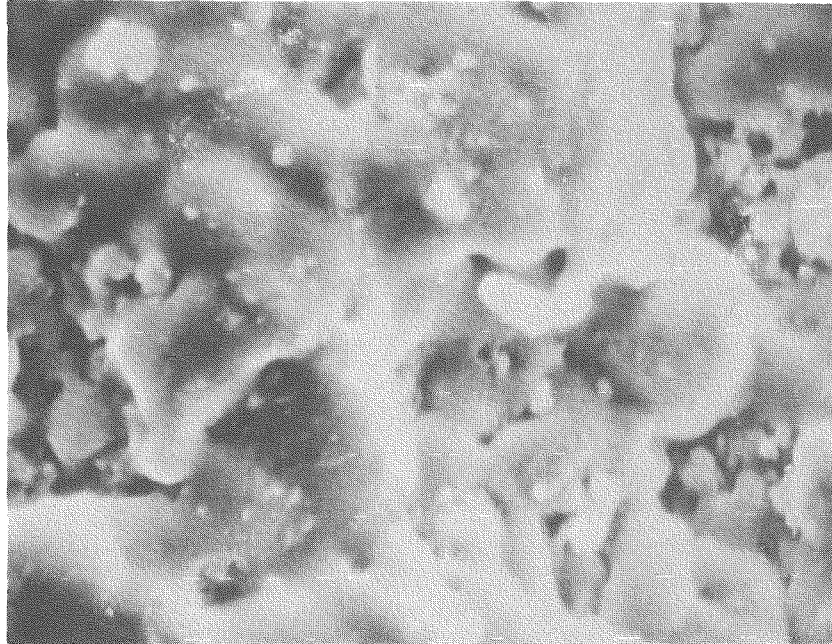


LSCU Illinois Bottom Coke, 2000X

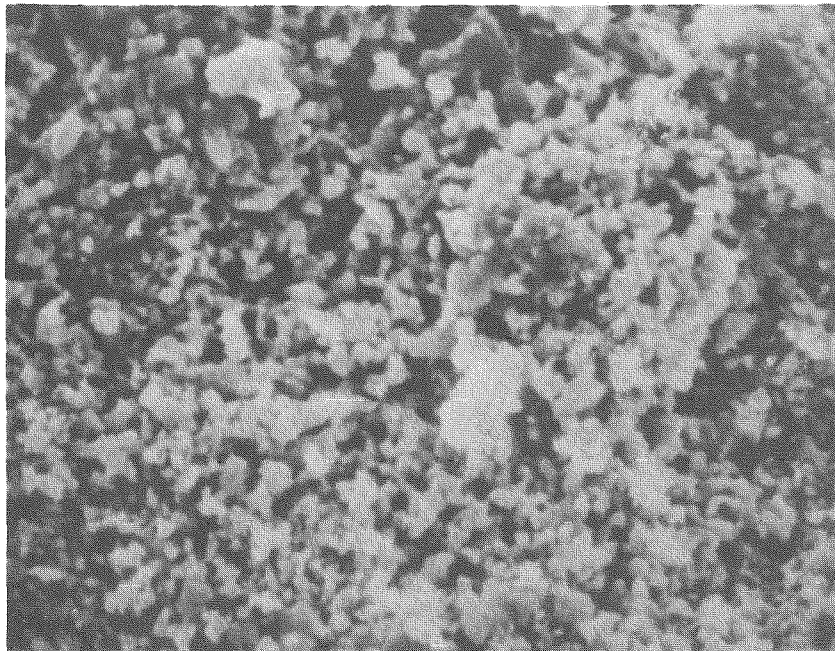
FIGURE 6-10

MICROGRAPHS OF RESIDUES FROM MINIGASIFIER RUNS ON
IKG BOTTOMS COKES BY SCANNING ELECTRON MICROSCOPE

- Steam Gasification @ RV -30°F and 30 psig



IKG ILLINOIS RESIDUE, 5000X



IKG WYODAK RESIDUE, 5000X

6.6 Bench Coke Gasification - Ash Fusion Studies

The air/steam gasification of coal liquefaction bottoms coke in the FLEXICOKING process gasifier vessel could lead to the formation of coke ash agglomerates. The Small Fluidized Bed Unit (SFU) described in the July-September, 1977, EDS Quarterly Technical Progress Report (FE-2893-3) was designed to investigate ash agglomerate-free air/steam gasification conditions. The primary operating parameters of interest in the SFU were temperature, superficial velocity and air/steam ratio.

Runs on the SFU demonstrated the necessity for good coke bed particle mixing. The original sintered quartz distributor frit permitted the formation of a stagnant coke region where ash agglomerate formation apparently was initiated. The use of a conical distributor produced ash agglomerate-free runs at identical conditions at which slagging had occurred using the sintered frit distributor. Most runs were subsequently made with the conical distributor.

Figure 6-11 is a preliminary plot of SFU operating conditions delineating coke ash agglomerating and non-agglomerating regions for IKG Illinois bottoms coke at RV+70°F. The data from duplicate runs suggests that a non-agglomerating-ash operating region can be determined for the SFU. Additional data points will be obtained to more precisely define the agglomerating/non-agglomerating regions. Similar plots are planned at other temperatures of interest.

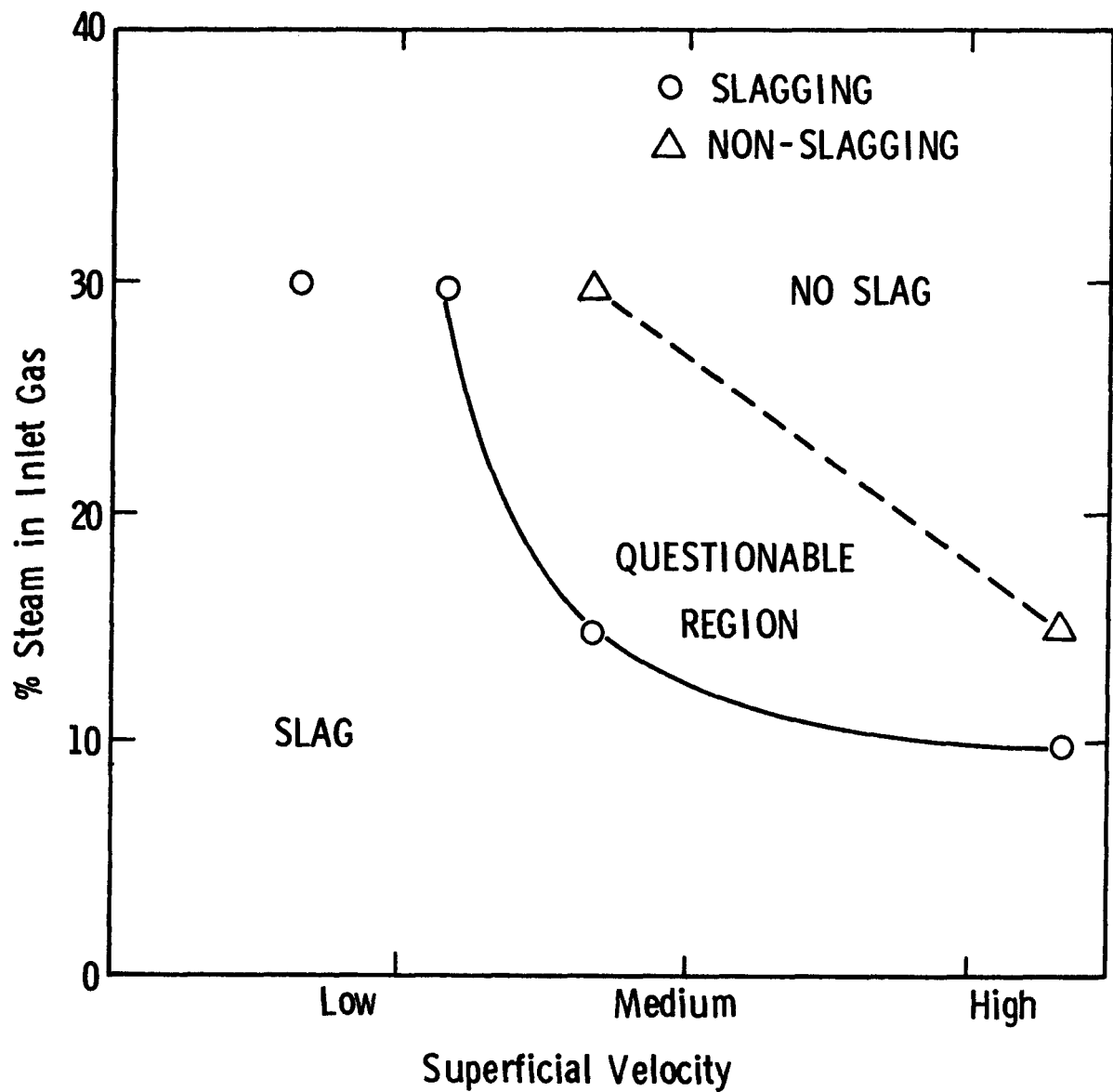
The analysis of ash agglomerates obtained from IKG runs with Illinois liquefaction bottoms coke has shown that the agglomerates have essentially the same composition as Illinois coal ash. Although significant amounts of vanadium, introduced with petroleum seed coke, were detected in agglomerates obtained in fourth quarter 1977, it is believed that the vanadium was not responsible for agglomerate formation. Later runs in 1978, after the vanadium content had decreased, still produced coke ash agglomerates. ASTM ash fusion test cones made from IKG ash agglomerates indicated ash fusion temperatures were similar to those of Illinois coal ash.

X-ray diffraction and optical microscopy analyses of ash agglomerates have produced somewhat contradictory results. X-ray patterns tend to show that little non-crystalline material is present in the agglomerates whereas optical microscopy seems to show mostly glassy constituents. This discrepancy will be investigated further.

Although the SFU has produced agglomerates that are similar to those from the IKG gasifier, questions remain as to the applicability of SFU results to larger units. Clearly, conditions in large units and the 7/8" I.D. SFU reactor are not identical. A more detailed understanding of fluidization, heat transfer and temperature distribution phenomena in both laboratory and commercial sized units is required for reliable comparisons to be made. Nevertheless, it is believed that the SFU is useful as a screening tool for determining potentially safe, non-agglomerating operating conditions.

FIGURE 6-11
SFU SLAGGING RESULTS

- IKG ILLINOIS COKE
- RV + 70°F
- CONICAL DISTRIBUTOR



6.7 Coke Gasification - Operability Studies

Coke gasification studies were made 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).

The primary purpose of the FBU coke gasification study was to determine the effect of gasification on coke particle integrity. An indication of the ultimate carbon utilization can be obtained from the amount of fines produced and the ash content of the fines. The FBU was also used for a short study of slag formation during coke gasification. Details of FBU runs are tabulated in Appendix H.

● FBU Fines Make Studies

FBU studies indicate that coke made from EDS Illinois bottoms maintains sufficient particle integrity to allow gasification in the FLEXICOKING process. The fines make is higher than petroleum but the ash content of the fines is so high that carbon loss is not excessive.

Figure 6-12 shows that fines make with IKG Illinois coke was 2-5 times higher than with Billings petroleum coke. However, the ash content of the fines was greater than 90 percent, as shown in Figure 6-13, so that the carbon content of the fines was only 3-7%. The ash-free coke lost in the fines is the product of coke content and fines make. Thus, the maximum loss of carbon with the fines was less than 1 percent.

The fines make data in Figure 6-12 show considerable variability as the gasification level increases above 60%. This was discussed in the October-December, 1977, EDS Quarterly Technical Progress Report (FE-2893-7). It was concluded 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. The fines that are produced are uniformly high in ash content.

The effect of increased attrition was tested by making hot attrition tests at the end of several gasification runs. The amount of fines was increased by as much as 100%. Also, the additional fines were greater than 96% ash. These test results agreed with the ash-layer theory of fines attrition.

It was pointed out that 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 the properties of the coke itself.

FIGURE 6-12
FBU COKE GASIFICATION RESULTS
FINES MAKE

LEGEND:

○ 1KG ILLINOIS COKE - FLUID COKING

△ 1KG ILLINOIS COKE - FLEXICOKING

x BILLINGS PETROLEUM COKE

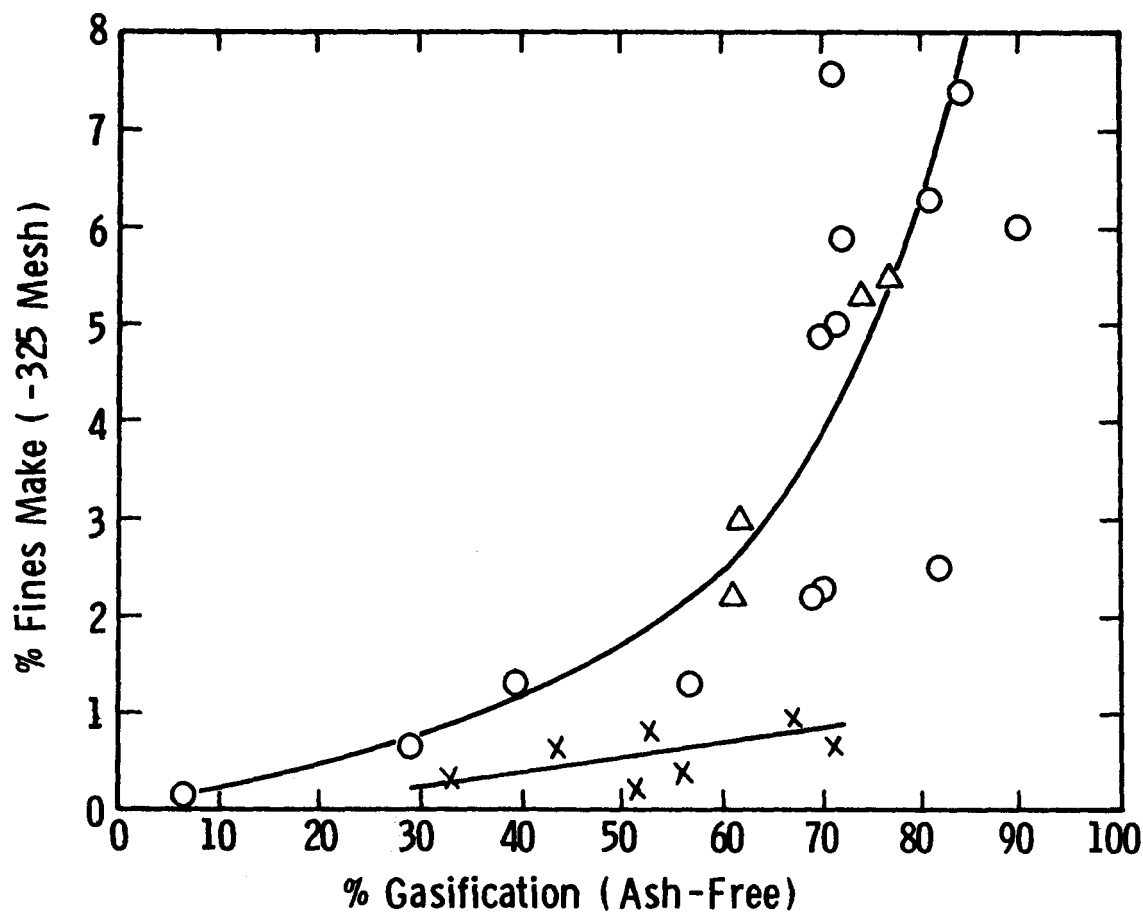


FIGURE 6-13
FBU COKE GASIFICATION RESULTS
ASH ENRICHMENT OF FINES
IKG ILLINOIS COKE

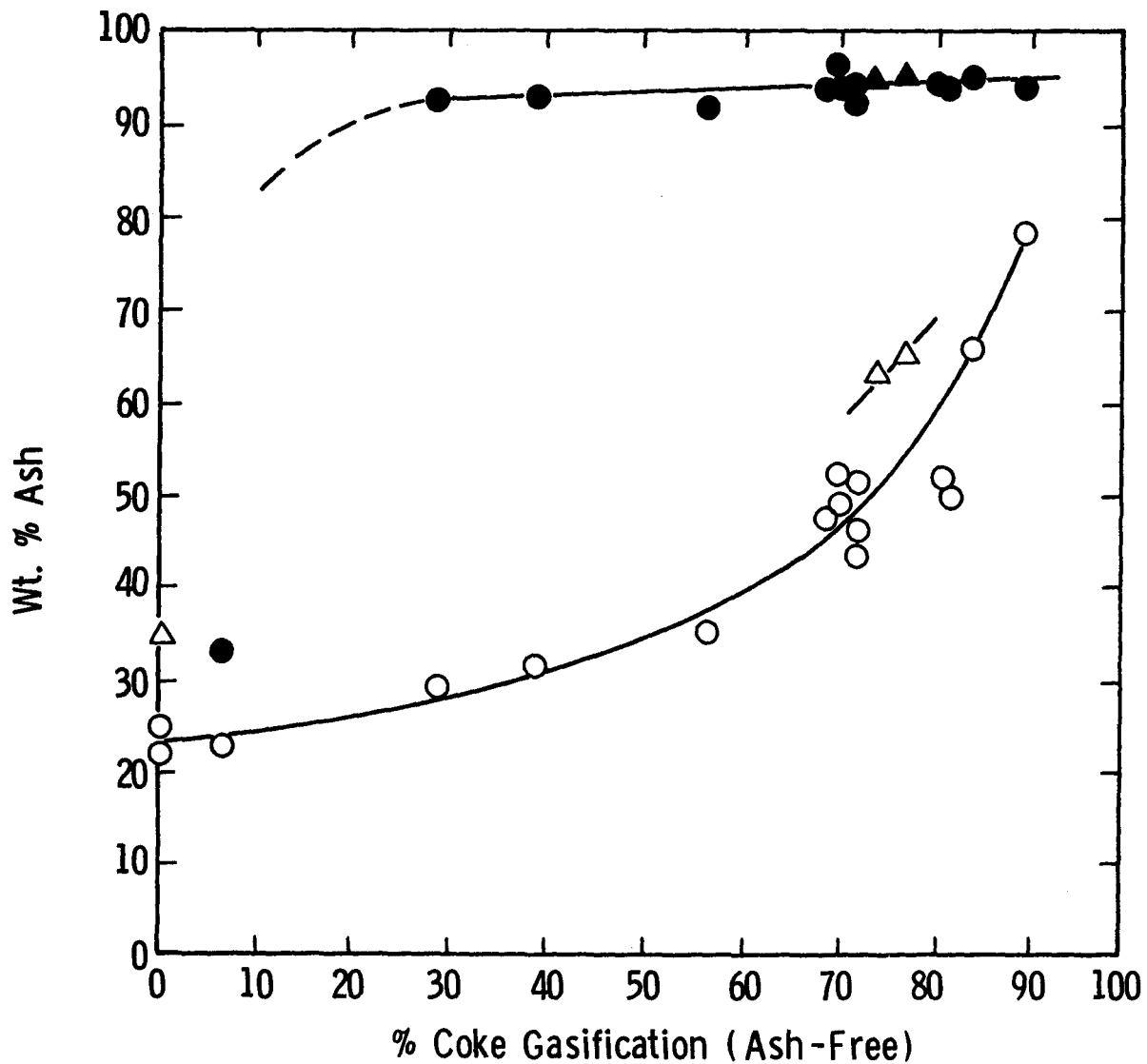
LEGEND:

○ IKG - FLUID COKING

△ IKG-FLEXICOKING

FILLED POINTS - FINES

OPEN POINTS - BED COKE



For the FBU, the highest energy device in the reactor overhead system, with respect to particle attrition, is the cyclone. The effect of the cyclone on fines generation was tested by making coke gasification runs with the cyclone bypassed. The top of the reactor was connected directly to the cloth-bag fines filter. The results are shown in Figure 6-14. Data points for individual runs are connected by lines to show the variation of fines make during each run. This is somewhat more informative than simply showing the final total cumulative fines makes.

The two runs with the cyclone bypassed showed lower fines make, particularly at intermediate gasification levels. The reason for the sharp increase of fines at the end of the runs is not known. Two possible operational causes were considered. If the reactor were depressured too abruptly, entrainment could be increased. Also, if the solids were not completely drained from the fines filter except at the end of the run, this could explain the sharp increase of fines. Neither cause is supported by the data because there was not an unusually large amount of solids in the last sample from each run. There was a sharp increase, however, in the fraction of the fines filter sample that was fines. Data for these two runs are given in Tables J-18 and J-19 of Appendix H. The coke for these runs was produced on the Large Stirred Coking Unit (LSCU) from EDS Illinois bottoms. The coke was ground and screened to provide a desirable size range and was devolatilized before the gasification runs.

The first fines-make tests with EDS Wyodak coke in the FBU were completed in June. As shown in Figure 6-15, fines make with Wyodak coke was similar to that with Illinois coke. If just the results with LSCU cokes are compared, the Wyodak fines make appears to be lower. However, the two runs with IKG Wyodak coke fall right in the band of the IKG Illinois cokes. All of these runs were made at a gasification temperature of RV-30°F except for the ones with IKG Wyodak coke, which ran at a 100°F lower temperature. The primary reason for the reduced temperature was that the activity of the IKG Wyodak coke was so high that a standard five-hour run could not be completed with a standard-sized coke charge. The activity at RV-130°F was similar to IKG Illinois coke at RV-30°F. At this time, the effect of temperature on fines make is not known, except as it relates to level of gasification. If there were any sintering of the ash layer, the ash would be more difficult to remove by attrition, so fines make would be expected to decrease as temperature was increased.

Additional runs are needed to determine the effect of cyclone attrition on fines make. Also, runs with reduced gas flows would yield data on the effect of gasification on particle size with a minimal amount of attrition.

More runs will be made with Wyodak coke over a range of gasification levels. Some fines-make runs will be made at different temperatures to determine if there is an effect of temperature on fines make.

FIGURE 6-14
FBU GASIFICATION FINES MAKES
EFFECT OF CYCLONE

Ground LSCU Illinois Coke
+ 100 Mesh, Devolatilized

○ With Cyclone
△ Cyclone Bypassed

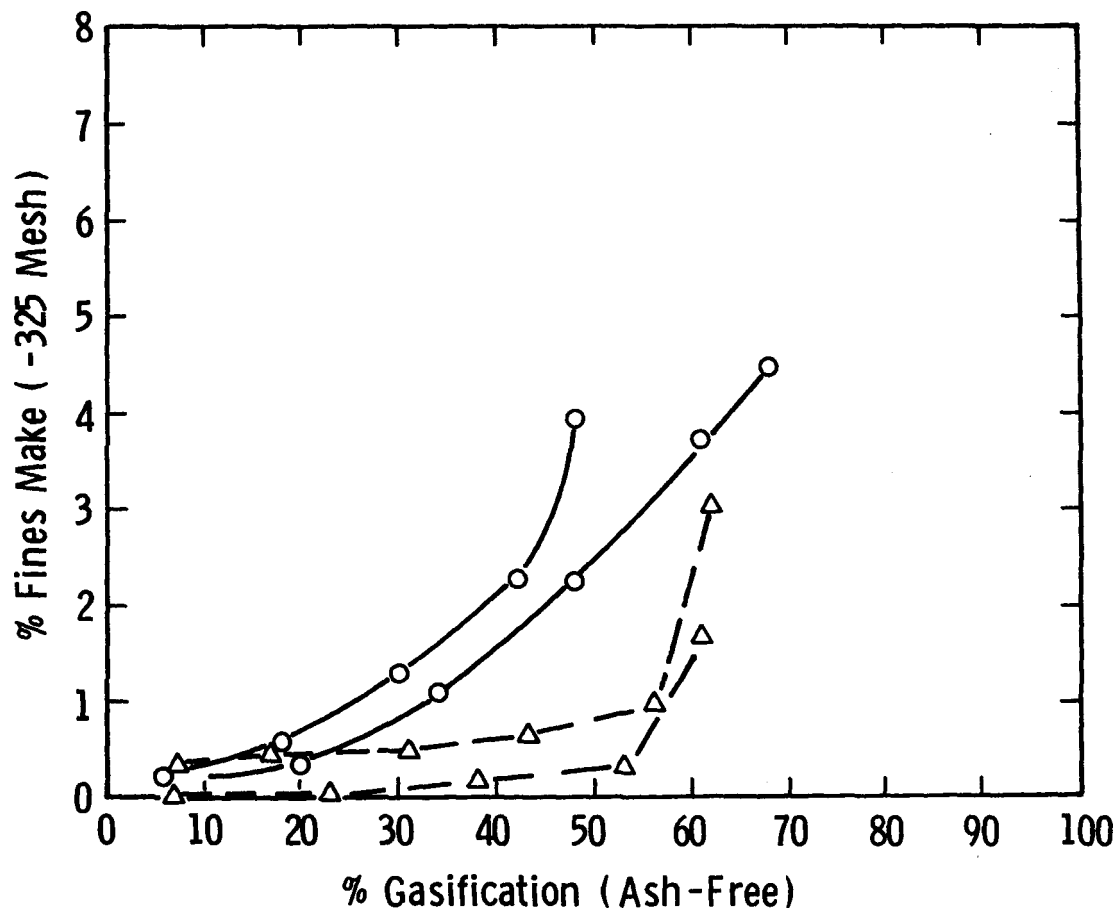
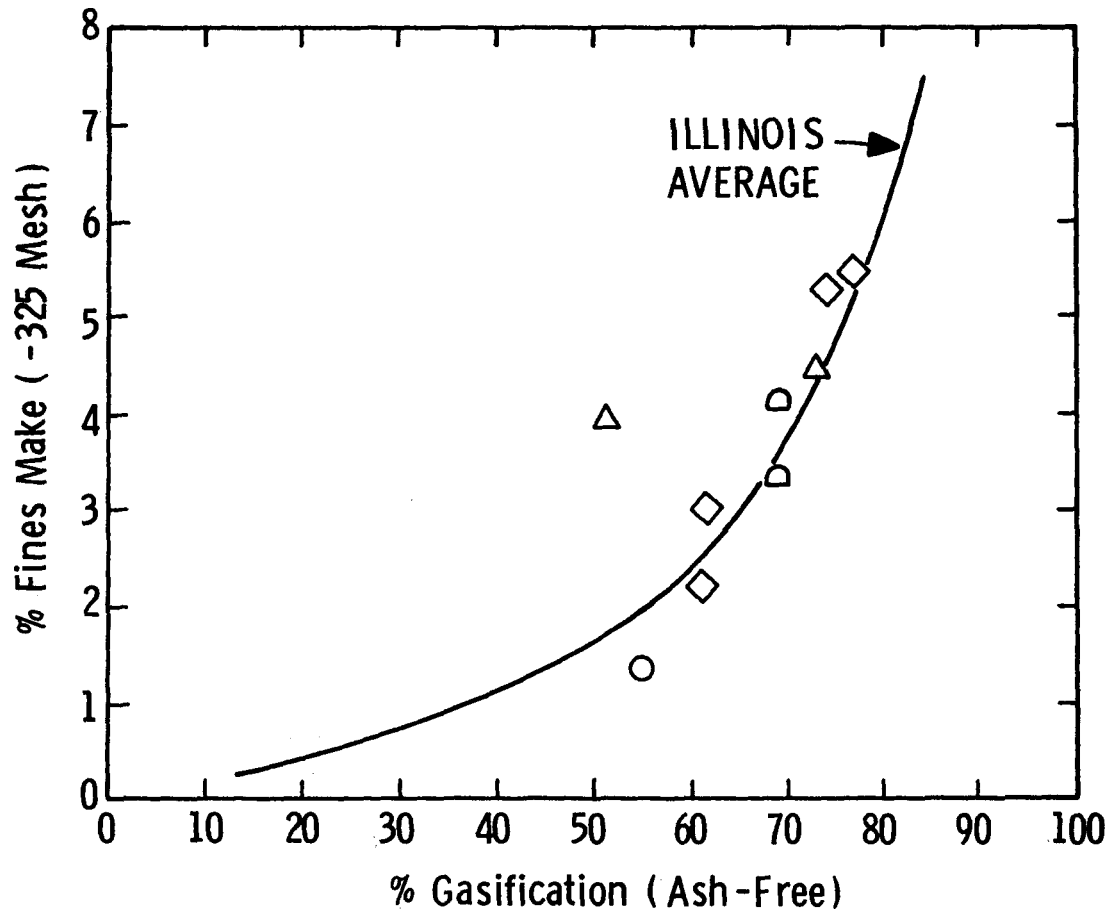


FIGURE 6-15
FBU GASIFICATION FINES MAKE
COMPARISON OF ILLINOIS AND WYODAK COKES

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



● FBU Slag Study

A series of runs was made in the FBU to investigate slag formation. In the many fines-make runs made in the FBU, no slag formation had been observed. The standard conditions used during the fines-make runs were RV-30°F, 30% steam (70% air) and a superficial velocity on the order of that expected in the commercial unit. Slag formation has been experienced in the IKG and the Small Fluidized Unit (SFU). Compared to the FBU fines-make operating conditions, both the other units had similar temperatures but lower superficial velocities and steam rates in the runs where slag was observed. Thus, the effect of those two variables was studied in the FBU. The results are summarized in Table 6-15.

Five runs were completed using the conical distributor that was used in the FBU fines-make runs. Slag was formed in one run with 15% steam, but it was believed to be due to a temperature excursion. The temperature excursion is thought to be unit-related. The small coke beds used in the slagging study provided a small bed-to-wall heat transfer area so that the heat dissipation capacity was critical. Wall temperatures had to be continually lowered during a run to provide an increased temperature differential to compensate for the reduced contact area as the coke bed became shorter. If this was not done properly, the heat generated by the carbon oxidation could exceed the heat transfer from the bed through the wall and to the gas so the bed temperature would increase uncontrollably. In subsequent runs, complete gasification of the carbon was achieved without the formation of slag at even more severe conditions: 5% steam and 0% steam (100% air).

A scaled-down version of the IKG distributor was used to see if the modified hydrodynamic and thermal conditions around the gas inlet would have any effect on slag formation. Another temperature excursion occurred in the first run with the IKG-type distributor and slag was produced as a result. Three runs were made with 15% steam with no formation of slag. Runs 77 and 78 were terminated when the bed temperature abruptly dropped. This was interpreted as indicating that the carbon had been consumed. However, subsequent analyses revealed the ash contents were only 53%. The apparent loss of reaction may have been caused by the formation of impervious ash shells on the coke particles that encapsulated the carbonaceous cores. However, Scanning Electron Microscope (SEM) examinations of cross-sections of the coke particles did not show any apparent shell formation. Additional microscopic examinations will be made in an effort to explain the runs.

One slagging run was made using ground LSCU Wyodak coke and a small piece of gray slag was produced. The temperature was higher than used in the IKG runs.

Additional analyses will be made of the slag samples and the FBU operating conditions to better understand the slagging experiments in the FBU and the relationship to studies in the SFU and the IKG. Also, cold models of the FBU will be used to study the flow patterns in the gas inlet zone with different gas distributors.

TABLE 6-15

FBU GASIFICATION SLAGGING STUDY

FBU Run No.	Maximum Temp. (T-RV°F)	Pressure (psig)	Steam (vol%)	Run Hours	Coke Charge (grams)	Slag (grams)	Bed Ash (Wt%)	
							start	end
<u>Conical Distributor:</u>								
70B	-20°	10	30	4	900	0	60.7	85.8
71	40	10	15	4	708	0	57.5	81.7
72	585	25	15	2	700	225	57.5	79.4
74	14	25	5	2.5	691	0	54.3	98.5
75	50	25	0	3.3	691	0	54.3	97.1
<u>IKG-Type Distributor:</u>								
76	700+	25	0	1	1399	9	34.1	38.6
77	0	25	15	3.5	1050	0	34.1	53.0
78	30	25	15-0	3	1048	0	34.1	52.2
79	-25	25	15	5	2993	0	35.2	44.9
<u>Ground LSCU Wyodak Coke:</u>								
80	51	25	15	1.8	1049	1.9	19.4	26.6

Coke feed was from IKG Illinois Run 20, except Run 80 used -50/+200 mesh ground LSCU Wyodak coke.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

Bottoms Processing Research

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

The IKG pilot unit was operated for three months in 1977 (August-November) and again for three months in 1978 (March-June). The objective of this work was to assess the operability of the FLEXICOKING process on Exxon Donor Solvent (EDS) coal liquefaction vacuum bottoms. The summer-fall run and the first weeks of the spring run used Illinois No. 6 coal derived bottoms whereas the last two and one-half months of the spring run used Wyoming subbituminous (Wyodak) coal bottoms. Both feeds were vacuum bottoms produced in the one ton-per-day Coal Liquefaction Pilot Plant (CLPP).

7.1 Annual Summary of Findings

The key issues of this year's study involved deposit formation in the gasifier, gasification activity, and coke particle integrity. Pilot unit operability was followed for indications of commercial operability problems. Most of the findings now available are based on the Illinois bottoms runs (see the October-December, 1977, Quarterly Technical Progress Report [FE-2893-7]). Preliminary information on the April-June 1978 Wyodak operations and details of the gasifier deposit formation experience are reported in the following Reporting Categories 7.2 and 7.3. Wyodak sample chemical analyses along with studies on gasification activity and coke particle integrity are in progress and will be reported in the July-September 1978 Quarterly Technical Progress Report.

• Some Deposit-Free Gasifier Operation

The IKG slagging experience along with some process information is given in Table 7-1. All deposits found have been chemically similar to the feedstock ashes. There is evidence that the deposits were both formed near the gasifier steam/air inlet nozzles. Two low temperature Wyodak runs have been made which were deposit-free. From this experience it appears that low gasification temperatures favor the elimination of deposits. The reader is cautioned that operating differences and uncertainties preclude any conclusions about relative slagging differences between Illinois and Wyodak mineral matters based on IKG observations. However, better gasification activity enabling lower temperature Wyodak operation helped produce the Wyodak deposit-free operation. Bench studies reported in Bottoms Processing Research Reporting Category 6 of this report further address the slagging issue.

• Gasification Rate Data Modeled

An existing ER&E proprietary coke gasification model has been successfully applied to the Illinois bottoms coke gasification data

TABLE 7-1
IKG EDS SLAGGING EXPERIENCE

<u>Feedstock/Run</u>	<u>Mineral Deposits</u>	<u>Gasifier</u>		<u>Feed to Reactor</u>	
		<u>Max. Temp., °F</u>	<u>Min. Steam, Mole % Stm+Air</u>	<u>Lbs.</u>	<u>Ash Content, Wt.%</u>
Illinois/20a	2300 Gms.	RV+147	4	2,100	16
Illinois/20b	90 Gms.	RV	13	2,500	16
Illinois/21	90 Gms.	RV-25	4	1,200	18
Wyodak/22	9 Gms	RV-25	22	2,300	12
Wyodak/23	no slag observed	RV-110	22	900	12
Wyodak/24	no slag observed	RV-110	22	3,100	12

(see October-December, 1977, Quarterly Technical Progress Report [FE-2893-7]). Studies in progress also indicate this model works equally well for Wyodak coke gasification. In both cases the model has been most successful for gas conversions in the 30 to 70% range of design interest. However, there are shortcomings with this particular model which ongoing bench and computer studies are addressing.

- Illinois Coke Particle Integrity Acceptable

Data obtained from IKG on fines production, intensive particle properties, coke particle size distributions, and pilot unit operability as influenced by particle properties provide important information on particle integrity. The positive data observed and reported for Illinois operations in all of these areas provide strong indications that coke particle integrity would not limit the application of the FLEXICOKING Process to EDS Illinois No. 6 coal liquefaction vacuum bottoms.

Some of the key features of the Illinois data supporting this position are summarized here. The carbon rejection to heater/ gasifier fines was only about 1% of gross coke carbon. Similarly, reactor fines losses were small. Intensive coke particle properties including particle density, internal surface area, and void volume indicated the Illinois cokes were well consolidated. Coke particle size distributions had followed previous petroleum operating trends with no unusual features. Consequently, pilot unit operability as influenced by coke properties was good. There were some misgivings that the first Illinois operations were near 70% gross coke carbon gasification or slightly below the 80% target. IKG runs in March were at 80% gasification and continue to support the position that IKG particle integrity data does not indicate FLEXICOKING process limitations.

The coke particle integrity information from the Wyodak operation is still under study as noted. However, the Wyodak fines losses were greater than Illinois losses and signal a potential problem area for Wyodak bottoms processing and indicate coal feedstock related particle integrity factors.

7.2 Recent IKG Operations

Simulation of the FLEXICOKING process with Illinois and Wyodak coal liquefaction bottoms was carried out in the IKG pilot unit during the period March 12 through June 15, 1978. A summary of this operation is presented in Appendix Table I-1.

A total of 1205 pounds of feed was processed in 136 hours of on-oil time during the IKG operation on Illinois bottoms. A feedstock changeover was then made and 6321 pounds of Wyodak coal liquefaction bottoms were fed during 894 on-oil hours. The resulting unit service factor for the combined operation was 46% representing nearly a 40% improvement over the 1977 IKG EDS run. The average length of an on-oil period in these studies was twice

that of the 1977 operations. The improvement in unit service was primarily a result of: 1) diluting the feed with solvent, 2) changing feed nozzles at prescribed intervals, 3) modifying the feed transfer system, and 4) increasing unit supervisory personnel.

A summary of unit downtime and its causes is presented in Table 7-2. Feed system associated problems accounted for about two-thirds of the shutdowns but for only one-third of the total downtime. Reactor backend plugging, bridging and coke agglomeration continued to be costly items per occurrence and accounted for better than a quarter of the downtime. The required feed changeover and gasifier inspections consumed another quarter of the downtime.

TABLE 7-2
SUMMARY OF IKG UNIT DOWNTIME
March-June 1978

<u>Cause of Unit Downtime</u>	<u>Number of Occurrences</u>	<u>Total Hours of Downtime</u>	<u>Percent of Total Downtime</u>
Feed Nozzle Plugged	11	89	7
Feed Pump Problems	11	128	10
Feed Transfer Delays	7	89	7
Feed Changeover	1	143	12
Voluntary Shutdown for Feed Nozzle Change	11	100	8
Reactor Backend	9	181	15
Reactor Bridging or Severe Coke Agglomeration	8	175	14
Gasifier Circulation Stopped	0	0	0
Gasifier Inspections	3	130	11
Heater Vessel or Backend	4	65	5
Other	6	<u>133</u>	<u>11</u>
Total Downtime		1233	100

Reactor coke agglomeration in the pilot unit is a function of nozzle operating conditions as well as feed characteristics. Agglomeration experience improved during this run primarily because the feed nozzles were changed at prescribed intervals thus avoiding mal-distribution of feed brought on by nozzle erosion and coking.

Feed pump problems began to surface at the higher pump rates and longer run periods. Feed transfer difficulties were much less of a nuisance than in the 1977 IKG studies because of feed dilution. However, there were still feed transfer delays brought on by the higher feed rates and the higher unit service factor.

Coke particle size control was maintained in these studies by gristmilling coke external to the unit just as in the 1977 IKG run. This form of grinding differs substantially from commercial FLEXICOKING process practice. Yet, reasonable control over coke particle size was maintained. Reactor coke particle size data are plotted in Figure 7-1. The data show that reasonable particle size control was maintained for both Illinois and Wyodak feeds despite several instances of agglomeration.

Nominal operating targets are shown in Appendix Table I-2 for all the balances in the 1978 operations. The actual operating conditions will be reported next quarter. Reactor and heater nominal temperatures remained essentially unchanged throughout the operations. Gasifier temperature specifications were changed for kinetic and slagging considerations. A few balances were targeted for below 80% gasification to provide additional coke for unit inventory and EDS bench programs.

7.3 Recent Gasifier Deposits

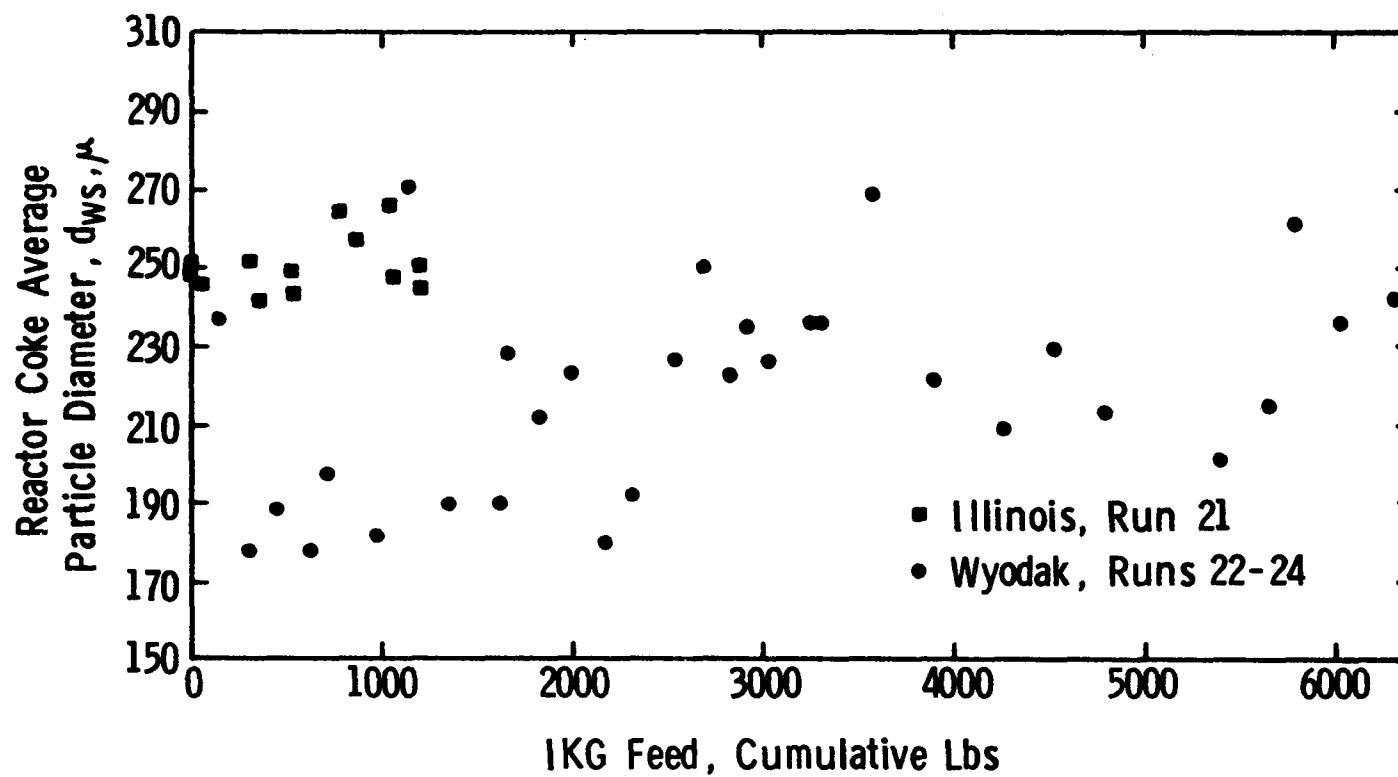
The recent IKG gasifier deposit observations are summarized in Table 7-1. Chemical analysis and ash fusion studies on the Run 21, 90 gm. Illinois deposit are discussed in the May, 1978, EDS Monthly Technical Progress Report (FE-2893-14). The Illinois deposit composition and fusion properties are similar to Illinois No. 6 coal ash. The deposit shape indicated positioning near one of the instrument bleeds or thermowell in the bottom of the gasifier close to the steam-air inlet.

The 9 gm. Wyodak deposit was mostly friable but did have one surface showing evidence of flow. X-ray diffraction analysis indicated minerals present which were also found in Wyodak deposits from Small Fluidized Unit (SFU) slagging runs.

7.4 Future Work

A three month run is planned for early 1979. The primary purpose of this run will be to test solutions to slag formation when processing coal derived bottoms.

FIGURE 7-1
REACTOR COKE SIZE
March-June 1978



ENGINEERING RESEARCH AND DEVELOPMENT

PROCESS ENGINEERING AND COST EVALUATIONS

1. Commercial Study Design and Cost Estimate

1.1 Illinois Study Design Update - Plant Configuration Study

A major study design for a commercial EDS plant utilizing Illinois No. 6 coal was completed in Phase IIIA. Using the results of this work as a basis, engineering and laboratory studies have identified potential improvements on the original base case plant. These studies have been reported in other Quarterly Reports. The EDS Process Alternatives LP Model (PAM) was utilized in this effort to determine which combination of improvements gives the optimum plant configuration(s) consistent with feasibility, operability and project/ economic basis. Preliminary evaluations identified two promising alternative types of configuration:

- A base case configuration in which high Btu gas (C_1/C_2) is not included in the product slate.
- A market flexibility configuration in which high Btu gas is included in the product slate.

The base case configuration is similar to the previous study design configuration including FLEXICOKING* of vacuum bottoms, steam reforming of C_2 - hydrocarbon gases. However, the liquefaction residence time has been increased about 60% above the previous study design value (to 40 minutes). In addition, the FLEXICOKING unit operates in the once-through mode. The H_2 makeup needed is provided by steam reforming C_2 - hydrocarbon gases. The market flexibility configuration retains the same liquefaction operating conditions as the base case. In this configuration, the H_2 makeup is provided by partial oxidation of liquefaction vacuum bottoms.

The preliminary product slate for each configuration and the corresponding plant economics with the PAM base price structure are included in the detailed documentation of this study, reported in the July - September Quarterly Report (FE-2893-3).

1.2 Illinois Study Design Update Onsite Bases Set

During the fourth quarter of 1977, the onsite bases were finalized for the EDS Commercial Plant Study Design Update (SDU) for Illinois coal. Unproven technology in peripheral areas will be avoided. The SDU will reflect the lowest cost-per-barrel plant, consistent with a logical layout of large-size units and the results of recent research and engineering work.

* Service Mark

Three process configurations were chosen for the Illinois Study Design Update. The Base Case design is a maximum liquids case including a FLEXICOKING unit to supply plant fuel and steam reforming to supply hydrogen. Sensitivity Design #1 will be a market flexibility case. A FLEXICOKING unit will recover additional liquids and produce plant fuel. Hydrogen will be produced by partial oxidation, and high Btu gas will be available for sales. Sensitivity Design #2 will be a technology alternative configuration. This case will use partial oxidation to make fuel gas and hydrogen and will sell high Btu gas. FLEXICOKING will not be included in this case.

Liquefaction conditions of 840°F reactor temperature and 40 minutes residence time were selected on the basis of previous PAM studies. These liquefaction conditions are acceptable for the base case design and first sensitivity design. Further screening work will be done for the technology alternative case (Sensitivity Design #2) to set liquefaction process conditions.

The new design basis and configuration are expected to improve the reliability of the plant over the 1975/1976 Study Design. The plant (Figure 1-1) will consist of two trains, each train consisting of two lines of liquefaction furnaces, reactors and separators with common treat gas facilities. The two-train concept gives added flexibility to meet operating demands and to keep both trains hot and running at turn-down conditions during unscheduled outages of individual units in either train.

Three FLEXICOKING units per plant are considered necessary to provide adequate continuous and reliable fuel-gas supply without the need for excessive turndown of the rest of the plant during a single FLEXICOKING unit outage. Four steam reformers are used for hydrogen generation with each unit being about the largest size currently planned for refineries. A single solvent hydrogenation unit is provided for each train.

Because of the high reliability of fractionators, and for economy of scale, the two liquefaction lines will share common fractionation facilities.

Study of the incentive for economies-of-scale for the Study Design Update has indicated a desirability to increase coal feed rate from 24 to 30 kT/SD. The Study Design Update plant will produce approximately 69 kB/SD of total products versus 60 kB/SD in the original Study Design. Table 1-1 compares the preliminary material balance and unit capacities between the 1975/ 1976 EDS Study Design and the Update cases.

Outlines of operating procedures and steps were prepared for startup, shutdown, and unit outages (both scheduled and unscheduled). This information was used in selecting the overall plant configuration, establishing intermediate and product tankage requirements, and making a preliminary estimate of the plant capacity factor. Further information on tankage and plant capacity factor is discussed elsewhere in this report and in the 4Q77 Quarterly Technical Progress Report (FE-2893-7).

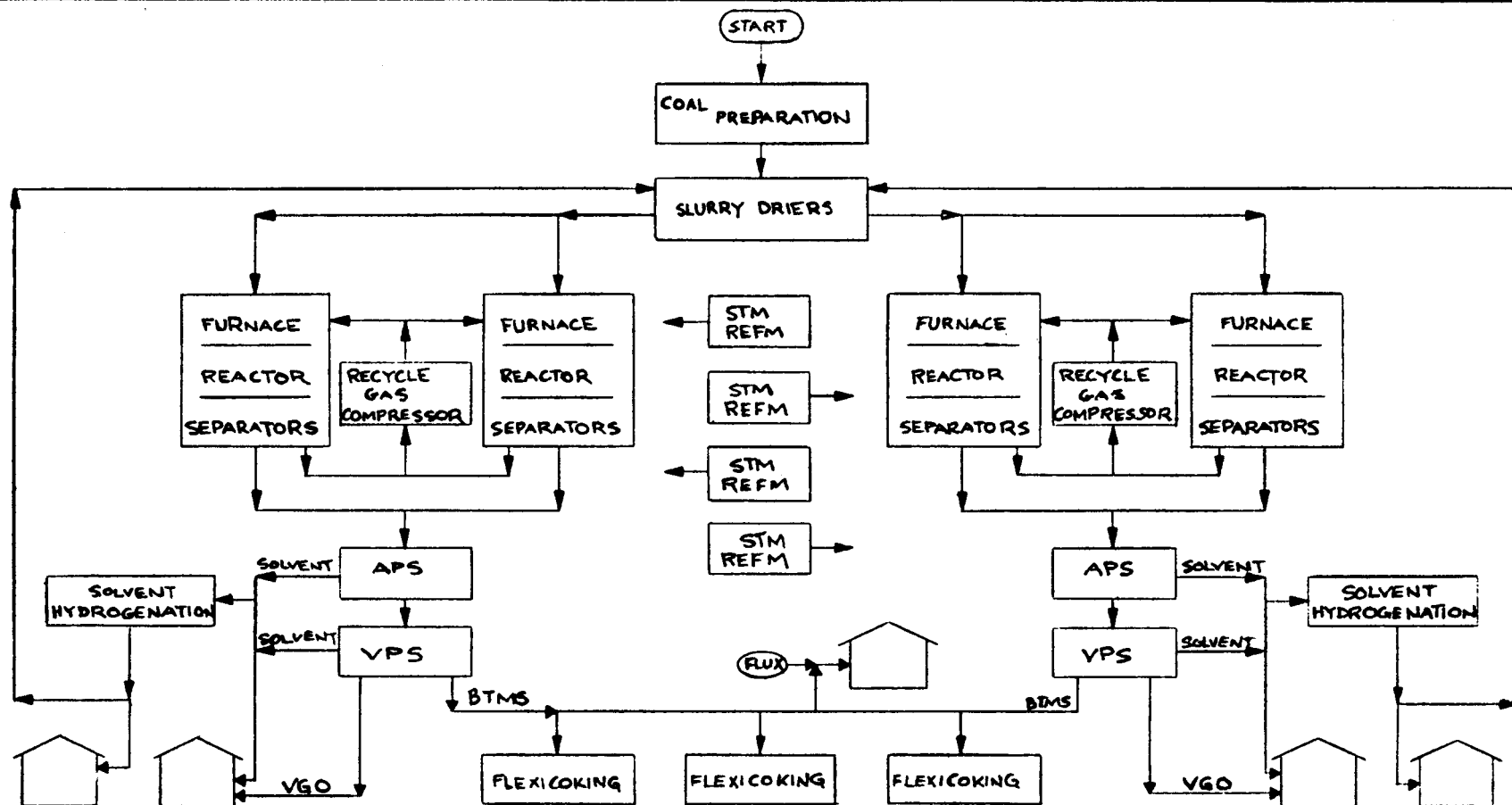


FIGURE 1-1
CONFIGURATION OF UNITS - ILLINOIS
STUDY DESIGN UPDATE (BASE CASE)

EXXON RESEARCH & ENGINEERING CO.
 SIMPLIFIED BLOCK DIAGRAM
 EDS COAL LIQUEFACTION
 COMMERCIAL PLANT
 STUDY DESIGN UPDATE
 AJN/FRD DEC 22, 1977
 77-1680-2

TABLE 1-1

1978 EDS STUDY DESIGN UPDATE - ILLINOIS BASE CASE

PRELIMINARY MATERIAL BALANCE AND UNIT CAPACITIES

	<u>1975/1976 EDS STUDY DESIGN</u>	<u>1978 EDS STUDY DESIGN UPDATE</u>
<u>COAL FEED RATE "AS RECEIVED"</u>	24 kT/SD	30 kT/SD
<u>PROCESS CAPACITIES, PER UNIT</u>		
LIQUEFACTION	12 kT/SD "AS RECEIVED" COAL	7.5 kT/SD "AS RECEIVED" COAL
ATMOSPHERIC DISTILLATION	117 kT/SD	144 kB/SD
VACUUM DISTILLATION	49 kB/SD	60 kB/SD
FLEXICOKING	21 kB/SD	19 kB/SD
HYDROGEN PLANTS	68 MSCF/SD	100 MSCF/SD
<u>PRODUCTS, kB/SD</u>		
C ₃ /C ₄ LPG	8.0	6
NAPHTHA (C ₅ /400)	22.0	30
FUEL OIL	29.8	33
TOTALS	<u>59.8</u>	<u>69</u>

Features to be Incorporated in the Onsites Basis

Based upon the results of the engineering screening studies and further laboratory data, the Study Design Update reflects the selection of economically preferred operations and process improvements to the EDS process. For example, the following features will be incorporated into the design.

- + Once-through (rather than recycle) FLEXICOKING unit
- + Increased liquefaction residence time
- + Elimination of low temperature shift in steam reforming
- + Reduced solvent endpoint
- + Increased onstream period for slurry furnaces
- + Solids withdrawal from liquefaction reactor
- + Removal of particles from coker scrubber bottoms
- + Revised yields and thermal efficiencies for liquefaction and FLEXICOKING
- + Actual CLPP feed and product data to be used
- + Added tankage to improve capacity factor
- + Coal to offsite boiler (not to FLEXICOKING feed)
- + Reuse of water
- + Sparing of coal preparation facilities to improve capacity factor

Work on the onsites design for the Illinois Study Design Update Base Case is now nearing completion.

1.3 Slurry Furnace/Train Size Study for Study Design Update

This study was carried out to examine slurry furnace valve operability, alternate methods for decoking the slurry furnace, and to determine the optimum size of the processing trains to be included in the EDS Commercial Plant Study Design Update. The results of this study, together with the results of other studies of procedures for plant startup, shutdown and unscheduled outages, and of intermediate product tankage requirements led to the selection of a plant configuration having four independent lines of liquefaction furnaces, reactors, and separators, as described in the interim report titled "Study Design Update, Onsite Design Basis - Illinois Coal Case."* The size and throughput of each line is about 60 percent of that used in the previous Study Design. More detailed information on this study can be found in the October - December, 1977 Quarterly (FE-2893-7).

*FE-2893-10

1.4 Tankage and Capacity Factor Study

The purpose of this study was to define the storage facilities which are needed for the EDS Study Design Update plant. The storage facilities will include tankage for both products and intermediate streams. Using these preliminary storage facilities and an assessment of the frequency and duration of all scheduled and unscheduled outages the capacity factor of the plant can be estimated.

Basis

The basis for this study is the preliminary base case plant configuration which has been selected for the EDS Study Design Update. This base case plant as presently envisioned includes the following:

- four liquefaction units
- four steam reforming units
- two solvent hydrogenation units
- two product fractionation units (APS/VPS)
- three FLEXICOKING units
- additional support facilities (i.e., two light ends units, a coal preparation unit, utilities, etc.)

The units in the plant are arranged into two trains (see Figure 1-1). The third FLEXICOKING unit is shared by both trains. In this study, plant products include C₃ LPG, C₄ LPG and naphtha and a single fuel oil grade. Phenol, ammonia and sulfur are also produced as byproducts.

Tankage

Product and byproduct tankage facilities were provided for the product slate previously indicated. Besides tankage for products and byproducts, the need for intermediate storage facilities for the following streams has been identified during the analysis of unit outages:

- liquefaction spent solvent
- hydrogenated recycle solvent
- vacuum bottoms
- sour water/phenolic water

Other storage facilities needed include:

- storage for FLEXICOKING unit startup feed
- light emergency storage
- cold rerun storage
- hot rerun storage
- isopropyl ether storage
- DEA storage
- caustic storage

Capacity Factor

The capacity factor of a plant defines the plant's throughput on a calendar day basis. This is the ratio of actual to theoretical yearly production. The plant's theoretical yearly production is defined as its stream day capacity times 365.

Using the frequency and duration of all unit outages, and the operating strategies during the outages, a preliminary estimate of the plant capacity factor can be made. Of the total possible capacity losses, approximately 27% are prevented by providing intermediate tankage. Another 24% of these possible losses are prevented by sparing facilities, reducing throughput, etc. Thus, the actual losses of the plant are 49% of the total possible losses. Additional losses on plant production are due to inefficiencies while operating at reduced throughput, C₃ product consumed to satisfy above normal H₂ consumption, startup fuel consumption, etc. These additional losses can be thought of as nonrecoverable losses incurred in preventing plant shutdowns but which are not related to the unit which is down. The total capacity loss during the two-year cycle is the sum of the actual losses and these additional losses or a total of 104.5 days. Thus, the preliminary capacity factor for the plant as presently envisioned is 85%.

Documentation of this study can be found in the October - December, 1977 Quarterly Report (FE-2893-7).

1.5 Study Design Update - Liquefaction/ Solvent Hydrogenation

Work continues to progress on the design of the onsite portion of the EDS Study Design Update for Illinois coal. The overall objectives and basis for the update work are documented in an interim report titled "Commercial Plant Study Design Update Onsite Design Basis - Illinois Coal Case" issued in May, 1978.* The majority of work to date has concentrated on the base case design which is intended to produce maximum liquids and which uses a FLEXICOKING unit to supply process fuel and a steam reformer to supply hydrogen.

A major portion of the effort has involved preparation of heat and material balances for the slurry drying, liquefaction, and solvent hydrogenation areas of the plant based on up-to-date yield data from laboratory catalyst life experiments (for end-of-run solvent hydrogenation yields). In addition, heat and material balances reflect results of a product quality study which investigated possible changes to the basic plant processing sequence which could improve the sulfur level of the overall plant fuel oil product relative to existing Federal New Source Performance Standards.

*FE-2893-10

The product quality study identified a need to hydrotreat all excess spent solvent in the solvent hydrogenation unit before sending it to fuel oil blending. A second recommended change involved addition of the 350/400°F portion of the liquefaction naphtha to the fuel oil pool following hydrotreating in the solvent hydrogenation unit. These two changes result in an overall plant fuel oil product which when combusted has emissions of 0.70 lb SO₂/MBtu. This meets the existing federal regulations of 0.8 lb SO₂/MBtu.

Several screening studies have been completed which deal with selection of optimum process conditions and sequencing and with selection of appropriate equilibrium data methods.

- Flash calculations on streams that contain coal and ash will be done by computer with the solids removed to insure that vapor-liquid equilibrium is not affected by solids. Enthalpy data and physical properties for the slurry streams will then be hand corrected.
- In the slurry drier area, the need for a vent gas compressor was identified which will be used to dispose of non-condensibles from the system. Also, the slurry drier overhead vapor stream will be cooled to 110°F instead of 200°F as in the original study design. This minimizes the quantity of vapor leaving the slurry drier distillate drum and therefore minimizes the quantity of water vapor in the vent gas and the size of the vent gas compressor.
- In the atmospheric and solvent fractionation sections, use of purge gas to replace stripping steam was investigated. However, results showed that the product streams would have difficulty meeting flash point specifications with use of purge gas.
- Operation of the solvent hydrogenation unit with a recycle gas compressor instead of a once-through basis with makeup hydrogen was selected as the preferred case. Neither case had a clear-cut economic incentive and therefore the recycle gas case was selected since it requires a smaller degree of integration with the liquefaction section.
- Operation of the liquefaction fractionator at 85 psia flash zone pressure has been selected as more attractive than at 35 psia flash zone pressure. An economic evaluation was made which considered equipment investments and operating costs based on simplified designs made following heat and material balance simulations of the two cases.
- Steam at a nominal pressure of 125 lb. will be used instead of 600 lb. steam as the heating medium for the slurry pumparound heaters. Due to a lower LMTD, use of 125 lb. steam requires approximately 10 times the exchanger area in this service. However, the investment debit is more than cancelled by the large operating savings for reduced steam costs.

- The solvent fractionator processing scheme will include sidestream withdrawal facilities. These facilities are needed to allow removal of 350/400°F naphtha from the recycle solvent. The 350/400°F naphtha is cut from the liquefaction naphtha, hydrotreated with spent solvent in the solvent hydrogenation unit, and then used in fuel oil blending to reduce the overall plant fuel oil sulfur level.
- Continuous water washing of the solvent hydrogenation hot separator vapor will not be included in the update design. Chlorine balances show that deposition of ammonium chloride salts should not be a problem in this area of the plant. As a result, improved heat recovery becomes possible.
- A flushing/blowdown system will be added to the plant facilities to insure removal of solids from the processing lines and equipment during shutdowns. Design of the system will be modeled on the proposed ECLP flushing/blowdown facilities.

Current Study Design Update efforts are concentrating on the design of the slurry drying, liquefaction, and solvent hydrogenation facilities including the preparation of equipment lists, specification of equipment size, development of utility requirements, and preparation of process control diagrams. In the product recovery area, preliminary heat and material balances have been prepared based on best available feed data. Final heat and material balances and equipment designs will be prepared as feed information from other areas of the plant is finalized.

1.6 Hydrogen Facilities for the Study Design Update Base Case

The hydrogen facilities basis for the Study Design Update Base Case, which had included purge gas steam reforming for hydrogen production, is being revised. The revised basis, which is similar to the 1976 EDS Study Design Basis, employs nominal C₁ - C₃ steam reforming plus a cryogenic hydrogen recovery system which removes hydrogen from the liquefaction and solvent hydro purge gases prior to their being fed to the steam reformer. This basis revision reflects a recent tightening of the overall plant fuel balance to the point where purge gas reforming, which requires over 10% more fuel than C₁ - C₃ reforming looks unattractive. A previous screening evaluation (January - March, 1978 Quarterly Technical Progress Report - FE-2893-12) had indicated that purge gas reforming had a small economic advantage over C₁ - C₃ reforming. However, that evaluation assumed a sufficient supply of low cost LBG to supply the fuel required. In the current fuel short situation, the extra LBG would have to come from additional FLEXICOKING scrubber liquids recycle which results in lower overall plant liquid yields.

Differences between the revised basis for the Study Design Update and the 1976 EDS Study Design basis exist in hydrogen plant size and hydrogen purity. The Study Design Update hydrogen plant size includes four 100 MSCF H₂/SD trains. These units are at or near maximum size based on commercial experience to date. The 1976 Study Design was based on four 68 MSCF H₂/SD trains. Hydrogen purity has been reduced from 95 to 94 mole% for both reforming and cryogenic hydrogen recovery based on a hydrogen purity screening evaluation documented in the October - December, 1977 Quarterly Technical Progress Report (FE-2893-7). This lower purity allows the economical use of a second-stage of high temperature shift rather than low temperature shift in the hydrogen upgrading section of the reforming process.

Initial heat and material balances and equipment design work is being revised to reflect the C₁ - C₃ steam reformer basis. These new heat and material balances will be based on near final liquefaction/solvent hydrogenation section balances and preliminary FLEXICOKING section balances, and should be completed by mid-July. Equipment design is expected to be completed in August.

1.7 FLEXICOKING Basis for the Study Design Update Base Case

The FLEXICOKING basis for the Study Design Update Base Case has been adjusted in response to modifications to the plant turnaround/unscheduled downtime schedule, changed requirements of the EDS plant fuel system, and additional information on the characteristics of FLEXICOKING scrubber liquids. The FLEXICOKING Design Basis has previously been discussed in the October - December, 1977 Quarterly Progress Report (FE-2893-7) and the May, 1978 Interim Report on the Onsite Design Basis for the Commercial Plant Study Design Update (FE-2893-10). As a result of this re-examination, the FLEXICOKING system will contain three once-through units of about 20 kB/SD feed rate each. This compares with three units of 19 kB/SD contemplated originally. The units are approximately 20% oversized to allow for runoff of vacuum pipestill bottoms that are stored when one FLEXICOKING unit is down and all liquefaction trains are on line. The average recycle rate of scrubber liquids returned to the reactor will be about 35% compared with 15% recycle planned previously.

Unit Downtime Revisions

Design capacities of the FLEXICOKING units have been increased from 112.5% to 122.4% of normal vacuum bottoms production rate as a result of adjustments in the time allowed for plant turnarounds and unscheduled FLEXICOKING unit downtime. Plant turnarounds have been increased from 42 days/2 years to 60 days/2 years to reflect further study of the time required to turnaround an EDS plant. The prototype design study similarly indicated that unscheduled FLEXICOKING unit downtime should be increased from 32 days/2 years per unit to 60 days/2 years for the pioneer plant. FLEXICOKING capacity was determined assuming that stored vacuum bottoms would be run off in one half of the "available time" (that time when the FLEXICOKING units are not involved in turnarounds or unscheduled downtime).

Fuel System Requirements Set 30% Minimum FLEXICOKING Recycle Rate

Plant fuel requirements indicate that a minimum recycle rate of 30% is needed for the FLEXICOKING unit to provide low Btu gas (LBG) for onsite use plus some spillover into offsite steam boilers. The quantity of spillover has been selected to minimize the impact on onsite furnace and offsite boiler operation due to normal variations in FLEXICOKING fuel gas production. EDS plant fuel requirements also increase from start-of-run (SOR) to end-of-run (EOR) due to increased hydrogen usage. When all liquefaction and FLEXICOKING units are operating and stored vacuum bottoms are not being run off, the FLEXICOKING units will have the ability to increase recycle of scrubber liquids from 30 to 45% as the EDS plant moves from SOR to EOR conditions. Increasing recycle rate leads to gasification of more material and thus provides additional LBG to satisfy EOR requirements. The relationship between LBG availability and scrubber liquids recycle rate is shown on Figure 1-2.

The average scrubber liquids recycle rate during a two-year operating period will be about 35%. This has been determined by considering turnarounds and unscheduled outages for liquefaction and FLEXICOKING units, and the time needed to run off stored vacuum pipestill bottoms.

Solids Removal from Scrubber Liquids

Hydroclones have previously been selected for reducing the solids content of FLEXICOKING scrubber liquids. Cleaned scrubber liquids are then blended with lighter liquefaction liquids and coker liquids to produce an EDS 400°F+ fuel oil product. Preliminary studies (January Monthly Technical Progress Report - FE-2893-8) indicated that for a scrubber bottoms feed with 4 wt% solids, three stages of hydroclones with an underflow (recycle) of 15% would satisfy the solids target level of 0.15 Wt% for the total 400°F+ fuel oil pool.

The 15% underflow rate was based on an anticipated scrubber liquids viscosity of about 1.5 cp. Recent measurements have indicated that viscosity of scrubber liquids is 3 - 5 cp at 700°F. The increased viscosity means that the hydroclone separation efficiency decreases, and the underflow rate must be increased to satisfy solids removal requirements.

Figure 1-3 shows predicted solids in the total fuel oil pool at a viscosity of 3 and 5 cp with three and five hydroclone stages. This figure indicates that if the scrubber liquid viscosity is closer to 3 cp, then three stages of hydroclones should provide sufficient solids removal in the 30 to 45% recycle range as required by fuel system considerations. If further testing demonstrates that scrubber liquids viscosity is closer to 5 cp, the options are to increase the number of stages or reduce the viscosity by blending in some lighter material in the hydroclone feed. The latter option could be accomplished by decreasing the scrubber overhead temperature thereby leaving lighter material in the scrubber liquids drawoff stream.

FIGURE 1-2

ILLINOIS STUDY DESIGN UPDATE
BASE CASE FUEL BALANCE

30 kT Feed to Liquefaction
SD
840°F/40 Min Liquefaction Conditions
C₂- Steam Reformers for H₂

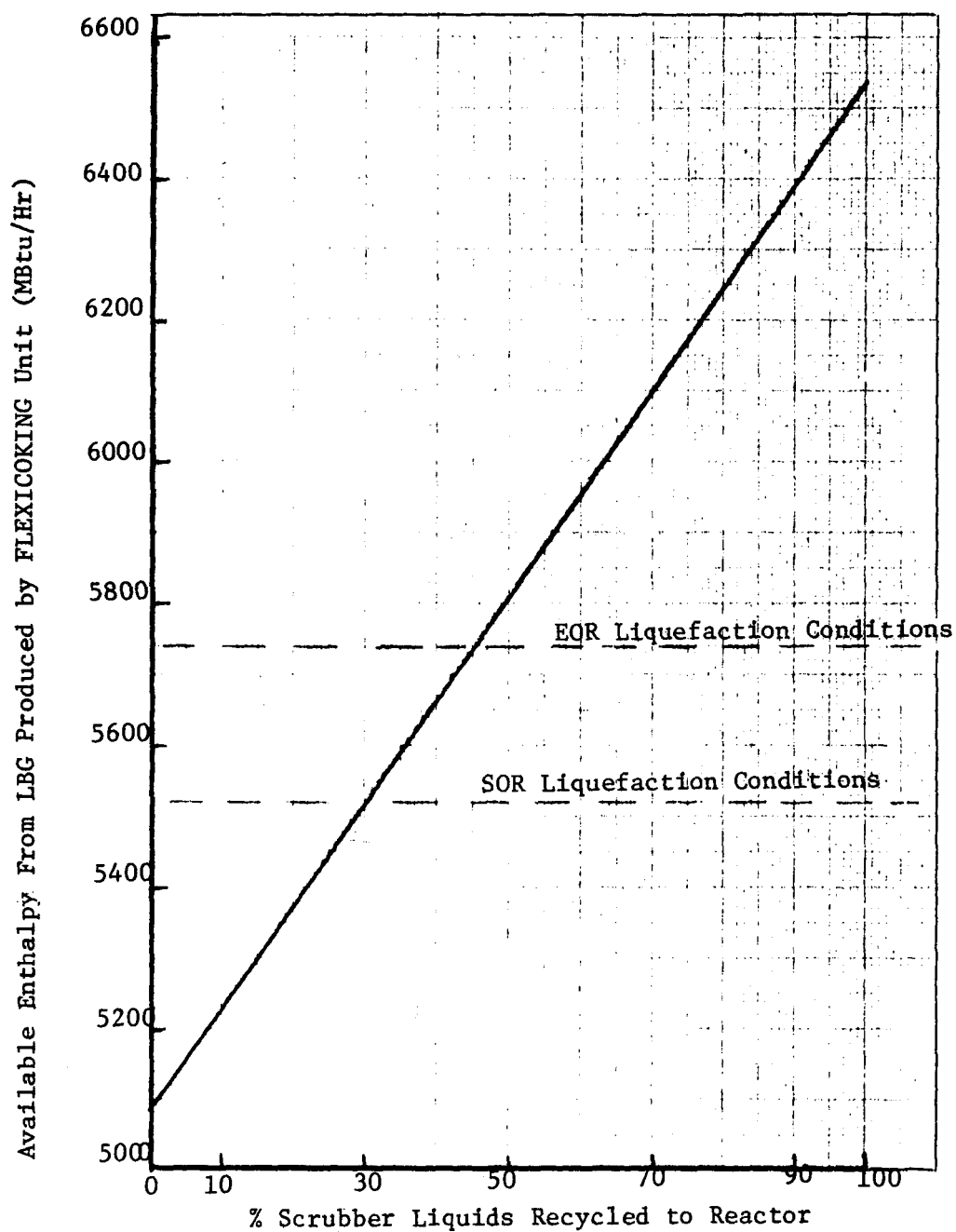
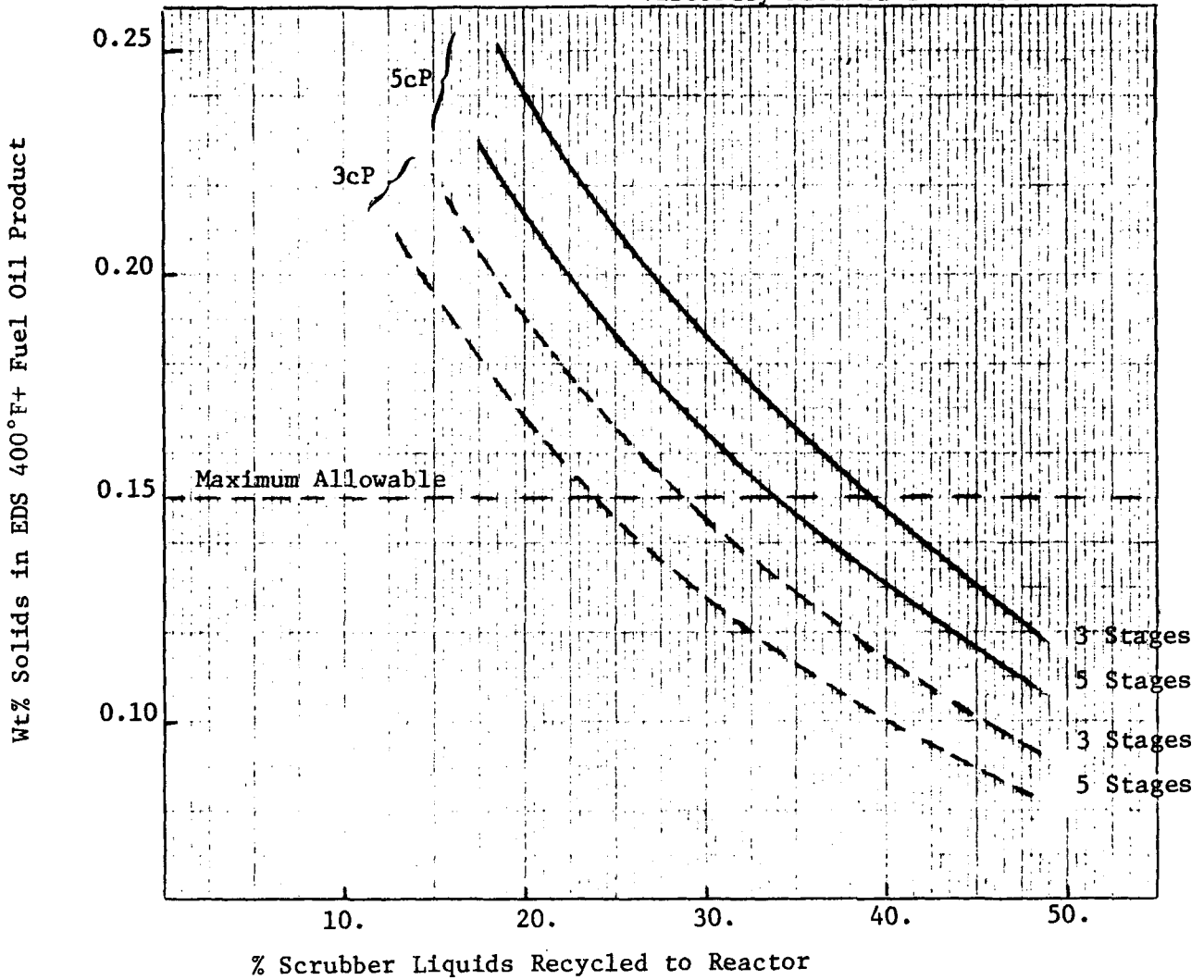


FIGURE 1-3

PREDICTED HYDROCLONE PERFORMANCE FOR
PARTICULATE REMOVAL FROM SCRUBBER LIQUIDS

Basis: 4 wt% Solids in Scrubber Liquids
Viscosity Assumed To Be 3cP or 5cP



Design work has been initiated for the FLEXICOKING unit in the Study Design Base Case operation incorporating the above basis changes.

1.8 Study Design Update - Offsite Basis

Work on the basis for the offsite facilities of the Study Design Update was started during the first quarter of 1978. Preliminary utility loads were tabulated based on the most recent PAM-LP cases. Firm loads such as major drivers and process steam were identified.

Contingencies for most of the utilities will be a function of the amount of design detail put into the onsite and offsite units. The level of these contingencies is currently under development. No contingency will be applied to onsite steam generation or process raw material (reactant) steam, such as that used in the steam reformer or FLEXICOKING unit gasifier.

A screening study for applying a packaged Rankine system to the liquefaction/solvent hydrogenation sections indicated there are potential applications such as the slurry drier and fractionator overhead streams and hot separator vapor stream. However, the net impact of these heat recoveries on EDS plant economy would be minimal. In addition, these large applications push the limits of a packaged system and add an element of technical uncertainty without commensurate economic benefit.

For these reasons, Rankine cycle applications are not included in the basis for the SDU.

1.9 EDS Study Design Update - Wastewater Treating Basis

As reported in the January - March, 1978 Quarterly Report*, work commenced on setting the basis for the wastewater treating facilities to be included in the EDS Study Design Update plant for Illinois coal. The preliminary scheme consists of units similar to those utilized in the 1975/1976 EDS Study Design, (i.e., the treatment scheme includes sour water treating, phenolic water treating, dissolved air floatation, biological oxidation, etc.). However, the exact processing basis will be determined in the offsite design phase.

One proposed operating scheme which was not in the original study design involves feeding the liquefaction cold separator sour water to the solvent hydrogenation unit. This operation was used earlier in CLPP and has been shown to remove from the offsites wastewater stream all organic acids, ketones and aldehydes which would have been present otherwise, thus reducing the required size of the offsite wastewater treatment facilities.

Evaluation of this scheme has shown that the savings in the offsite water treating facilities are insufficient to justify the increased costs in solvent hydrogenation. This option, therefore, will not be included in the Study Design Update, and further development of the concept has been dropped.

Previous studies also showed that an incentive exists for extracting phenols from the coal naphtha, (refer to the Phenolic Coal Naphtha/Wastewater Upgrading Study in the EDS Development Phase IIIA Final Report FE-2353-20). These phenols would be mixed with the phenols extracted from the wastewater and sold as petrochemical feedstock. In view of the limited experimental data available, the naphtha extraction unit will not be included in the EDS Study Design Update plant. However, the updated plant economics will use a value for the naphtha which reflects a chemical plant outlet for extractable phenols. These economics will be based on further development of this downstream recovery operation.

1.10 Fuel System for the Study Design Update Base Case

During May, work was begun in setting the basis for the fuel system of the base case of the Study Design Update. In the base case, most of the fuel used in onsite fired heaters will be low Btu gas (LBG) provided by the FLEXICOKING units. In addition, coal will be used in some of the offsite steam boilers and C₃/C₄ will be used as auxiliary/backup fuel. Major issues to be resolved include:

- The capacity required in the offsite boilers to fire LBG and coal.
- A source and volume of auxiliary fuel to accommodate normal short-interval variations in LBG heating value and plant fuel requirements.
- Transitional and lined-out responses to the impact on the fuel system of outages by large fuel gas producers or consumers.
- A strategy to cover long-term variability in the fuel balance. These longer term variations in plant fuel requirements arise mainly from the increase in solvent hydrogenation unit H₂ consumption as catalyst activity declines, (i.e. variations in start-of-run/end-of-run H₂ consumption).

An imposed restriction is that excess LBG may not be flared during normal operations. This restriction requires that the fuel system be designed such that enough LBG-fired boiler capacity is available at all times to consume all the LBG produced. Otherwise, process changes must be made to reduce the excess LBG production to a manageable level.

Preliminary fuel balances showed that the onsite fuel production (FLEXICOKING unit low Btu gas) and consumption were in rough balance during normal operations. Work to date has concentrated in developing a firm basis for the fuel balance during normal operations. A major concern then was the expected short term $\pm 5\%$ variability in the heating-value/rate of LBG. This variation is expected to occur in a continuously random fashion.

Operating the plant with a fuel balance which is too tight, may cause the variations in LBG supply to impact on onsite furnaces and heaters. Preferably, a portion of the LBG should go to the offsite boilers, such that the expected variations could occur in those boilers. The variations in LBG would be accommodated by varying the amount of coal firing used in the offsite boilers. The swings in the amount of LBG fired in the offsite boilers is also limited to about 50% due to control problems if fuel flow is too low. Thus, the preliminary assessment is that the spillover of LBG to offsites should be twice the expected net variability in the fuel system. To achieve the desired spillover of LBG into the offsite boilers, the amount of hydroclone underflow recycled to the FLEXICOKING unit will be adjusted as necessary.

It would be desirable to include both LBG firing and coal firing in the same firebox. Dual firing capability would simplify the automatic control scheme needed. However, dual firing may not be possible due to heat maldistribution when operating preferentially in one fuel. The strategy to cover the longer cycle variability in fuel requirements (start-of-run vs. end-of-run) could have a large impact on whether a dual-fired boiler can be used.

Future work will concentrate in developing a strategy to cover the larger term variability in fuel requirements and the fuel system response to outages by major producers or consumers.

1.11 EDS Plant Location Cost Study for Illinois Coal Case

The 1975/1976 EDS Coal Liquefaction Commercial Plant Study Design was based on a 24 ST/SD mine-mouth plant operating on Illinois No. 6 Bituminous coal. For the Study Design Update, a screening study was made of other locations. As a preliminary step, investment cost estimates were prepared. Next, other location-related factors which impact on the cost of producing and distributing coal liquids were evaluated. The other factors included:

- Coal and product logistics
- Maintenance costs
- Electrical power costs
- Operating costs
- Environmental factors and standards
- Real estate availability and costs
- Labor Productivity

Investment

The relative investment estimates for the various locations were based on the 1975/1976 Study Design, documented in Interim Report, Exxon Donor Solvent Coal Liquefaction Commercial Plant Study Design, FE-2353-13.

Locations chosen for evaluation were: "Rural" (Eastern) Illinois, Western Illinois, Lower Mississippi and the Gulf Coast. The estimates reflect the most recent cost estimating factors and information, and are shown in Table 1-2 along with the investment for the 1976 Study Design. A reassessment of Illinois plant construction concluded that the estimate for "Rural" Illinois was not realistic and that a "Western" Illinois cost should be used for an Illinois plant.

The estimates showed there were investment advantages for other locations, notable the Gulf Coast. The investment advantage of 590 M\$ for the Texas Gulf Coast vs. Western Illinois translates to \$6.28/Bbl of total coal liquids. This investment effect is the first listed on Table 1-2.

Coal and Product Logistics

The cost data presented in this section of the report are typical values, and do not represent official forecasts by Exxon Research and Engineering Company or any of its affiliates.

• Coal

An Illinois plant can be located adjacent to a mine supplying the liquefaction coal feed. However, for a Gulf Coast location, the 24 kST/SD of coal must be shipped nearly 1,000 miles to the plant. The following transportation options for this move have been considered:

- unit trains
- slurry pipeline
- unit trains/barge/collier (ocean-going vessel) system

For the 1985 time period, the lowest cost option is expected to be a combination of unit trains/barge and collier from the mine to the Gulf Coast Plant. The estimated 1985 cost of transporting coal from Illinois to the Texas Gulf Coast by this method \$17/ST which translates to a cost of about \$6.80/Bbl of total coal liquids. As shown in the tabulation of Table 1-3, coal transportation alone more than offsets the investment advantage of the Gulf Coast.

• Products

The next major cost consideration is that of product logistics. The two products, naphtha and fuel oil, involve different logistical and marketing considerations. Naphtha is a refinery feedstock for producing a gasoline blending component. Marginal supplies of gasoline for the Illinois area must be made available through imports, which must pass through the Gulf Coast area and incur transportation costs enroute to the Midwest. Thus, marginal gasoline costs will be higher in the Midwest, and naphtha from coal liquids in Illinois should have a higher value than naphtha produced in the Gulf Coast area. A projection of pipeline cost in 1985 is about 90¢/Bbl of naphtha, or about 35¢/Bbl of total coal liquids as listed on Table 1-3.

TABLE 1-2

EDS PLANT LOCATION COST STUDY
RELATIVE INVESTMENTS FOR VARIOUS LOCATIONS

	M\$	
	Instant Plant 3Q75	1985 Startup
<u>1976 Study Design, "Rural" Illinois</u>	1400	2170
Plant Location Cost Study (Study Design Update)		
"Rural" (Eastern Illinois)(1)	1670	2470
Western Illinois	1780	2640
Lower Mississippi River Area	1550	2300
Gulf Coast Area (Texas)	1380	2050

(1) EDS Coal Liquefaction Commercial Plant Study Design, Interim Report,
FE-2353-13.

Fuel oil logistics and marketing issues are more complex. Major markets envisioned for this product include large industrial consumers and public utilities operating oil fired boilers with several years of remaining economic life. To reach these markets, new heated supply systems must be developed. The supply system must be separate and segregated from any backup/supplementary petroleum fuel oil system used by the customer, because of incompatibility between petroleum and coal liquids.

If coal liquids can be sold into industrial or utility markets within the local area, distribution costs for both the Gulf Coast and Midwest locations should be similar. However, the local value of coal liquids as fuel oil would be higher in Illinois than in Gulf Coast if the alternative supplies of fuel for the Midwest must pass through the Gulf Coast to reach Midwest markets, thereby contributing to increased fuel oil costs for the Midwest market.

Assuming that marginal competing fuel oil supplies are produced from crude oil pipelined into the Midwest, coal-liquids fuel oil produced there may have a value 90¢/Bbl of fuel oil higher than in the Gulf Coast. This translates to about 45¢/Bbl of total coal-liquids disadvantage for the Gulf Coast, as shown on Table 1-3 under the column Fuel Oil Market Local.

The Illinois area, however, may not provide sufficient outlet for coal-liquids fuel oil. In that event, the fuel oil from an Illinois plant would be transported to the nearest adequate market. The largest potential markets for coal-liquids fuel oil are existing oil-fired power plants in Florida, the Mid-Atlantic states and the North-East. However, to supply these markets would require a heated system throughout--from plant to consumer. For an Illinois plant, the lowest cost supply system for the fuel oil volumes under consideration would include:

- Heated pipeline from the plant to the Mississippi River
- An Upper River terminal near St. Louis
- Barging to the Lower Mississippi River
- A Lower River terminal near New Orleans
- Heated tanker delivery to markets--Florida or East Coast

The cost of delivery from the plant to the lower Mississippi River is estimated to be about \$5.32/Bbl of fuel oil in 1985. The additional cost to Tampa, Florida is an estimated \$1.43/Bbl of fuel oil, or \$6.75/Bbl from the Illinois to Tampa. This translates to \$3.36/Bbl of total coal liquids.

The larger utility markets of Florida and the East Coast are less costly to reach from a Gulf Coast location. Tanker costs to Tampa are estimated to be only about \$1.72/Bbl of fuel oil, or \$0.86/Bbl of total coal liquids.

TABLE 1-3

EDS PLANT LOCATION COST STUDY

ADDED COST FOR GULF COAST LOCATION VS.
ILLINOIS BASE PLANT
(1978 STARTUP)

	<u>\$/Bbl of Total Coal Liquids</u>	
<u>Assumed Fuel Oil Market</u>	<u>Local</u>	<u>Florida</u>
Investment	(6.28)	(6.28)
Coal Transportation	+6.80	+6.80
Product Logistics - Naphtha	+0.35	+0.35
- Fuel Oil	+0.45	(2.50)
Maintenance Costs	0	0
Electrical Power	<u>+0.20</u>	<u>+0.20</u>
Net Added Cost	+1.52	(1.43)
Preferred Location	Illinois	Gulf Coast

Factors Not Evaluated in Detail

Operating Costs

Environmental Effects

Real Estate Costs

Thus, the net Illinois vs. Gulf Coast cost disadvantage for supplying fuel oil to Tampa is about \$5/Bbl of fuel oil shipped. This is equivalent to \$2.50/Bbl of total coal liquids--if there is no local market at either the Illinois or Gulf Coast location, and all fuel oil must be shipped to distant utility markets. To the extent that a portion of the fuel oil can be sold locally, the debit for an Illinois location is reduced.

Operating Costs

The higher investment for an Illinois plant location suggests that maintenance costs there may also be higher, but the effect is expected to be small and is shown as zero on Table 1-3.

The Gulf Coast power rates are expected to be higher than Midwest rates because of a higher proportion of new high cost facilities. In the 1985 to 1990 time period some projections show that the Gulf Coast industrial rate could be 3-10% higher. For other scenarios, the Gulf Coast power rate structure could be 18-20% higher than Illinois. For the current study a 10% higher industrial power cost was used for the Gulf Coast, resulting in a cost disadvantage of 20¢/Bbl as shown on Table 1-3.

Several other factors were briefly reviewed, but were not evaluated quantitatively. One problem, which would require additional development for a Gulf Coast location, is solid waste disposal. The ash residue from the plant would probably be disposed of in a remote area inland from the coastal location of the plant.

Overall Results

The net results of the economic effects of the various location factors are shown on Table 1-3. These results indicate that the sizeable apparent investment advantage for the Gulf Coast is more than offset by the anticipated additional cost of transporting the coal to that area. Product markets thereby become a crucial issue in determining the location with lower overall costs. There are reasonable expectations that local utility and industrial markets will be available for the fuel oil product from an initial plant. In these situations, a mine-mouth Illinois plant is the preferred location with an advantage of \$1.52/Bbl. However, if sufficient local markets do not develop for fuel oil, it may be necessary to ship product to more distant large consuming centers in Florida and the Northeast. If about 50% or more of the fuel oil product must be shipped over a longer heated supply system to these more distant markets, the Gulf Coast gains a cost advantage, and becomes the preferred location with up to \$1.43/Bbl advantage.

Availability of nearby markets for fuel oil could be a determining factor in establishing the preferred location for a specific plant. The net advantage for either location, Gulf Coast, or Illinois is relatively small, however, regardless of market availability. Based on the assessment of these various cost factors, an Illinois mine-mouth location has been selected as the basis for the EDS Commercial Plant Study Design Update.

ENGINEERING RESEARCH AND DEVELOPMENT
PROCESS ENGINEERING AND COST EVALUATIONS

2. Cost Reduction and Laboratory Guidance Studies

2.1 Liquefaction Solvent-To-Coal Ratio Study

Variation of the solvent-to-coal (S/C) ratio could be a potentially beneficial way of modifying the liquefaction operations. Accordingly, a study has been conducted to establish the cost of increasing this weight ratio from 1.2 to 1.6.

To facilitate the evaluation of the higher S/C ratio in plant investments and operating costs, various simplifying assumptions have been made. These assumptions confine the modifications required by the higher S/C ratio to the liquefaction and solvent hydrogenation units. The major modifications, besides the increase in solvent hydrogenation capacity, are in the liquefaction slurry preheat furnace, in the liquefaction reactor and in the fractionation facilities of the plant. These modifications increase plant investment by 26.7 M\$ (1985) over the investment at a 1.2 S/C ratio. Thus, capital recovery charges increase by 14.9 M\$/yr. In addition, operating costs for the plant increase by 17.5 M\$/yr. Of the increased operating costs, 26% are investment related, 48% are due to higher fuel requirements, 6% are caused by additional catalyst costs and 9% each are due to increased power and steam consumption. The balance is caused by small increases in other utilities. The total cost incurred in increasing the S/C ratio to 1.6 is, thus, 32.4 M\$/yr.

Background

The 75/76 EDS Study Design utilized a 1.2 solvent-to-coal (S/C) weight ratio. Much of the experimental work in the development of the EDS coal liquefaction process has been carried out at a S/C ratio of about 1.6. The driving force to lower the S/C ratio in the commercial plant is the desire to reduce the size of the solvent stream being recycled between liquefaction and solvent hydrogenation and, thus, reduce the plant investment. The basis for selecting the 1.2 S/C ratio were tests performed at the lower S/C ratio. The results of these tests indicated no significant change in yields when the lower S/C ratio was used.

The purpose of this screening study is to establish the economic cost associated with increasing the S/C ratio in the commercial study design from the present level of 1.2 to 1.6. The higher S/C ratio may improve the operability of the plant as a result of the lower solids content in the feed slurry. The improvement could be very noticeable in the liquefaction slurry preheat furnace, especially when a solvent lighter than the 75/76 study design solvent is used. The higher S/C ratio may also be needed to obtain the full benefit of some process improvements.

Basis

The basis for evaluating equipment changes at the higher S/C ratio was the Illinois base case of the 1975/1976 EDS Study Design, which utilizes a 1.2 S/C ratio. To simplify the evaluation of the modifications needed to operate at the higher S/C ratio, various assumptions have been made. The H_2 consumption in the liquefaction and solvent hydrogenation units has been assumed to be independent of the S/C ratio used and it remains unchanged when the S/C ratio is increased. It has also been assumed that neither liquefaction nor solvent hydrogenation yields are affected by the S/C ratio utilized. Thus, the overall yields and the combined product steam rates from liquefaction and solvent hydrogenation remain the same for both S/C ratios. These assumptions confine any equipment modifications to the liquefaction and solvent hydrogenation units.

Equipment Modifications

No major changes need be made in the liquefaction and solvent hydrogenation units configurations used in the 1975/1976 EDS Study Design to enable operations at a 1.6 S/C ratio. However, the size of various sections of these units must be increased to accommodate the higher solvent rate.

In liquefaction, the duty of the slurry preheat furnace must be increased by 18.5% to maintain the liquefaction temperature at 840°F. Similarly, the reactor volume must be increased by 20.8% to maintain the same liquefaction residence time at the 1.6 S/C ratio. The feed rate to the fractionation facilities in liquefaction also increases by 19.5%. The recycle gas separation equipment need not be modified since most of the additional solvent is removed from the recycle gas in the reactor effluent separator. The treat gas equipment and the slurry driers also remain unchanged from the 1.2 S/C ratio unit. The differences in the size increases for the various sections is caused by the varying amounts of vaporization experienced by the streams in question.

Whereas only some sections of the liquefaction unit are affected by the higher S/C ratio, the complete solvent hydrogenation unit must increase in size to process the higher solvent rate. Thus, the solvent hydrogenation capacity must be increased by 33%, the increase in solvent rate.

Investments and Operating Costs

The equipment modifications previously described cause the liquefaction investment for a unit using a 1.6 S/C ratio to be 49.1 M\$ (1985) higher than the investment for a unit using a 1.2 S/C ratio. The investment in both cases reflects the use of the optimum liquefaction conditions (840°F and 40 minutes residence time). About one-half of the liquefaction investment increase is caused by the larger slurry preheat furnace which must be provided. The balance is made up of the increases in reactor and fractionation facility investment (22% and 30% of the total increase in liquefaction investments, respectively). The solvent hydrogenation unit investment increases by 37.6 M\$ (1985). Thus, the total investment of the liquefaction/solvent hydrogenation block increases by 86.7 M\$ (1985). This represents an increase of 14.9 M\$/yr (1985) in the capital recovery charges for the plant.

The plant's operating costs also increase as a result of the larger increased maintenance and manpower costs, together with the higher utility consumption at the higher S/C ratio. The investment related operating costs increase by 4.6 M\$/yr (1985). These include the cost of wages, salaries, repair materials, etc. Major changes in utility costs are experienced as a result of increases in fuel consumption, electric power demand and low pressure steam. Fuel requirements at the higher S/C ratio are 162 MBtu/hr higher in view of the increased duty in the liquefaction and solvent hydrogenation furnaces. This represents an increase of 8.4 M\$/yr (1985) in the fuel costs of the plant, assuming that the additional fuel is provided by utilizing supplemental coal. Steam costs increase 1.7 M\$/yr (1985), reflecting the increased steam consumption in the solvent fractionator. Electric power costs also increase by 1.6 M\$/yr (1985) due to increased power requirements in solvent hydrogenation. Catalyst costs in solvent hydrogenation add 1.0 M\$/yr (1985) more to the operating costs and other utilities further increase it by 0.2 M\$/yr (1985). Thus, the plant operating costs increase by 17.5 M\$/yr (1985).

Table 2-1 summarizes all the changes in investments and in operating costs.

Results

The net effect of increasing the S/C ratio in the commercial study design would be to increase capital recovery charges and operating costs by a total of 32.4 M\$/yr. This represents an equivalent decrease in plant net cash flow if no yield differences are taken into consideration. The desirability of increasing the solvent circulation rate would depend on the magnitude of the increase in operability. For the higher S/C ratio to be economically attractive, the revenues derived from the higher plant capacity factor would have to be larger than 32.4 M\$/yr (1985), the costs incurred. No estimates of these revenues are available at the present time. Thus, no attempt has been made at this time to determine the incentive for using a 1.6 S/C ratio.

TABLE 2-1

LIQUEFACTION SOLVENT-TO-COAL RATIO STUDYInvestments, M\$ (1985, rural Illinois)

	<u>1.2 S/C</u>	<u>1.6 S/C</u>	<u>Δ</u>
● Liquefaction			
+ Slurry Drying	115.7	115.7	-
+ Slurry Preheat Furnace	192.6	216.2	23.6
+ Reactors	61.2	72.1	10.9
+ Recycle Gas Separation	165.7	165.7	-
+ Fractionation	44.4	59.0	14.6
Subtotal	579.6	628.7	49.1
● Solvent Hydrogenation	189.2	226.8	37.6
Total	768.8	855.5	86.7
Δ <u>Capital Recovery Charges, M\$/yr (1985)</u>		14.9	
Δ <u>Operating Costs, M\$/yr (1985)</u>			
● IROC		4.6	
● Fuel		8.4	
● Electric Power		1.6	
● Steam		1.7	
● Catalyst		1.0	
● Other		0.2	
Total Δ Operating Costs		17.5	
Δ <u>Total Plant Cost, M\$/yr</u>		32.4	

2.2 Characteristics of Illinois Vacuum Bottoms Solids

Work in EDS FLEXICOKING scale-up studies includes the development of reactor feed pumps and nozzles for both the prototype and commercial units. Laboratory and engineering evaluation of the effect of vacuum bottoms characteristics on the feed system for these units is scheduled to begin soon. To facilitate this effort, estimates of solids specific gravity, particle-size distribution, and concentration in the Illinois vacuum bottoms streams have been provided for both ECLP and for the commercial plant. This information is shown in Table 2-2 for solids normally found in the vacuum bottoms.

The information on the solids concentration and composition derived from material balances for the units. The particle specific gravity was calculated from literature values and was used during the design of ECLP and for the commercial study design. The information available at the present time on particle size distribution of the solids in the vacuum bottoms dates from pre-1970 pilot plant operations. During this period of time various solid separation schemes were tested (hydroclones, centrifuges, filters, etc.). The particle size distribution of the solids were determined to facilitate their evaluation. This size information is from operations feeding 100 mesh- coal to the unit (vs. 8 or 30 mesh- ECLP feed and 8 mesh- feed in the 75/76 study design). A major unresolved question is thus the effect of feed coal size on the particle size distribution of solids normally present in the vacuum bottoms.

Float-sink analyses of the solids in vacuum bottoms from pre-1970 operations tend to indicate that the solids normally found in the vacuum bottoms can be viewed as consisting of two types of particles. Particles derived from the indigenous mineral matter (ash) entrapped in the feed coal are usually very fine (mean size of only a few microns). A major exception could be particles rich in pyrite which seem to have a mean size about equal to that of all the solids. The ash particles ($\sim 1/3$ of the total) tend to have an intrinsic size distribution which is characteristic of the coal itself. Thus, they would be essentially unaffected by the feed coal size distribution. The second type of particle is composed mainly of unconverted coal (fusinite, etc.) but also contains appreciable amounts of mineral matter. These composite particles will be affected by the feed coal size distribution (with a coarser grinding operation yielding larger size particles). However, since as the coal swells upon heating the original organic matrix is almost totally destroyed, the effect of feed coal size in the composite particles may not be very large. Experimental verification is however needed.

In addition to the solids normally present in the vacuum bottoms, the ECLP vacuum bottoms will periodically contain the solids collected in the reactor solids withdrawal system. The purpose of the withdrawal system is to intermittently remove from the reactor any particles too large or too heavy to be carried through by the flow of material. If not withdrawn from

TABLE 2-2

CHARACTERISTICS OF ILLINOIS COAL VACUUM BOTTOMS SOLIDS

	<u>ECLP</u>	<u>Commercial Study Design</u>
● Solids concentration in vacuum bottoms wt%	36-47	31-32
● Solids composition, wt%		
+ unconverted coal (via MEK)	44.2	45.6
+ ash	55.8	54.4
● Solids specific gravity		
+ unconverted coal (via MEK)	1.4	1.4
+ ash	3.5	3.5
● Solid particle size	Max ~300 μ 98 wt% below 30 μ median ~7 μ	Assumed to be equal to ECLP

the reactors, the collected particles could cause serious operating problems. The planned operation of the withdrawal system in ECLP includes the daily disposal of the collected solids by adding them (during a 3-hour period) to the vacuum pipestill feed. The expected withdrawal rate during ECLP operation is about 2300 lb of solids (dry) per day.

The characteristics of the solids collected in the withdrawal system are for the most part unknown. However, they are thought to be composed mostly of ash, although due to the nature of the withdrawal system the collected solids may also include some relatively unreacted particles of feed coal. It is estimated that their average ash content is about 60%. Their maximum size is expected to be the same as the size of the largest feed coal particle (~ 4 mesh for 8 mesh- coal).

EDS commercial plants are expected to need reactor solids withdrawal systems. At the present time these systems are not well defined. Further information will be obtained from operations of CLPP and ECLP.

2.3 EDS Liquefaction Reactor Temperature Study

The purpose of this study is to provide a better definition of the modifications and associated costs needed to operate liquefaction at higher temperatures. One of the major operability problems associated with increasing the liquefaction reactor temperature to 880°F from 840°F is the high degree of solvent vaporization in the slurry furnace. This problem must be resolved before operation at the higher temperature can be envisioned.

Utilizing a solvent heavier than the solvent specified in the EDS Study Design (refer to the EDS Commercial Plant Study Design Interim Report No. FE-2353-13), provides a solution to the solvent vaporization problem. Evaluation of the EDS Process Alternative LP Model (PAM) of the 880°F cases, with investments reflecting the equipment modifications needed to produce the heavier solvent, shows an incentive of about 20 M\$/yr (1985\$) relative to 840°F liquefaction operation. These results are calculated for a case with liquefaction operating at 880°F, a residence time of 25 minutes and a liquefaction treat gas rate of 4 wt.% on a dry coal.

The incentive for operating at these conditions must be tempered in light of the following:

- Large quantities of supplemental coal to the FLEXICOKING unit are required to close the fuel balance.
- When the liquefaction reactor outlet H₂ partial pressure is increased to about the level of the 840°F cases by increasing the H₂ treat rate, the incentive for operating at 880°F disappears. (The above calculations assumed no increase in H₂ rate.)

Considering the potential operating problems associated with large quantities of coal in the FLEXICOKING unit feed and the uncertainty regarding liquefaction reactor H₂ partial pressure requirements, liquefaction at a temperature of 880°F appears to be, at best, only marginally more economic than operating at 840°F.

Complete documentation of this study can be found in the July-Sept. 1977 Quarterly Report (FE-2893-3).

2.4 Economics of Liquefaction Residence Time

As part of the EDS Liquefaction Process Variables Study, the net cash flow from EDS plants operating with liquefaction at 840°F and various residence times were determined for Illinois coal. The primary driving force to increase residence time is to convert vacuum bottoms (worth ~50 to 100 \$/t) to valuable liquid products and high Btu gas (worth ~300 \$/t). Results from this Illinois coal study indicate that little or no incentive exists to increase the liquefaction residence time beyond about 40 minutes. The purpose of this research guidance study is to illustrate the impact various economic factors have on the incentive to increase residence time. The economic factors of interest are:

- the value of the vacuum bottoms destroyed
- the cost of H₂ consumed to achieve the additional conversion
- operating costs (including return on investment required)
- the value of additional products recovered.

The incentive analysis was performed via cash flow balances around the liquefaction/solvent hydrogenation block. In this type of analysis, calculations are made of the incremental changes in cash flow caused by changes in product and intermediate stream rates, in utility consumption and in the operating costs (including return on investment) of the block as residence time is increased incrementally. The net incentive for the next increase in residence time is thus the sum of these cash flow changes.

The results of the present analysis indicate that the incentive to increase liquefaction residence time to about 40 minutes is strong because:

- There is a good liquefaction response in terms of a large increase in vacuum bottoms conversion relative to the incremental hydrogen consumed.
- The incremental increases in operating costs are moderate.

Little or no incentive exists for increasing residence time beyond 40 minutes due to substantially higher operating costs and moderately higher hydrogen costs.

In situations where higher cost fuel is required to balance the plant, the resulting higher vacuum bottoms value and increased hydrogen costs substantially decrease the incentive to utilize long residence times. However, this decrease in incentive does not greatly affect the optimum liquefaction residence time.

Complete documentation of this study can be found in the January-March, 1978 Quarterly Report (FE-2893-12).

2.5 Liquefaction Process Variables Study Update

Previous studies with the EDS Process Alternatives LP Model (PAM) have showed that at the optimum liquefaction condition substantial incentive exists for using supplemental coal to close the plant fuel balance. In these studies the supplemental coal was converted to gaseous fuel in a FLEXICOKING unit. Alternatively, supplemental coal could be fired in a conventional coal-fired boiler (CFB) to raise 600-psig steam. This update of the liquefaction process variables study tests the previously determined optimum liquefaction conditions for the situation where the supplemental coal is fed to a CFB.

Results from the study confirm that the maximum net cash flow is obtained when the liquefaction section is operated at 840°F and a residence time of about 40-60 minutes. Hydrogen makeup is provided by the partial oxidation of vacuum bottoms. About the same net cash flow level can also be achieved by utilizing a reactor temperature of 880°F and a shorter residence time; however, potential operating problems at the higher temperature make this case a higher risk alternative.

Thus, the optimum liquefaction conditions are the same for the scenario with supplemental coal available to the CFB's only as for the scenario with supplemental coal to a FLEXICOKING unit. However, the net cash flows for the cases with coal fired boilers burning supplemental coal are lower than those with supplemental coal fed to a FLEXICOKING unit. This reflects in part the smaller volume of total plant coal liquids because coal pyrolysis liquids are not produced when coal is sent to the CFB's.

The results of this study have also shown that the previously determined optimum liquefaction condition with partial oxidation and gas sales permitted is also valid when steam reforming of C_1/C_2 's is used to provide the H_2 required by the plant.

Complete documentation of this study can be found in the October-December 1977 Quarterly Report (FE-2893-7).

2.6 Liquefaction H₂ Treat Rate Sensitivity Study

The purpose of this screening study is to determine what savings can be realized by a 25 percent reduction of the H₂ treat rate to liquefaction assuming no reduction in liquefaction product yields or qualities. This reduction in treat gas rate could improve operations in the liquefaction unit by decreasing the vaporization in the slurry preheat furnace. The results are also expected to be useful in evaluating other process improvements.

The base case plant selected for comparison utilizes a liquefaction unit with a 4 wt% on coal treat gas rate. The principal source of hydrogen for this plant is a steam reforming unit. Utilization of the lower (3 wt% on coal) treat gas rate requires modifications in the liquefaction unit and also in the H₂ generation, recovery, and compression facilities. In determining the required modifications it has been assumed that the liquefaction H₂ consumption is independent of the H₂ treat rate.

Investment savings as a result of these modifications amount to 41.7 M\$ (1985). This represents a decrease of 7.2 M\$/yr (1985) in capital recovery charges. In addition to these savings, investment-related operating costs (IROC) are reduced by 2.2 M\$/yr and utility costs are reduced by 5.1 M\$/yr. The reduction in utility costs is mainly a result of lower power requirements in the H₂ compression facilities. Thus, the total savings, which can be achieved by using the lower H₂ treat rate, are 14.5 M\$/yr (1985).

Background

Experimental work has previously demonstrated the importance molecular H₂ has in the liquefaction unit when a lower quality solvent is used. To some extent, when a low quality solvent is used, increasing the molecular H₂ present can substitute for donor hydrogen. The use of larger-than-usual amounts of molecular H₂ under these circumstances provides yields approaching those obtained when using a high quality solvent and less molecular hydrogen. Conversely, if an even higher quality solvent (or more solvent) is used, smaller-than-usual amounts of molecular H₂ could be used without reducing the liquefaction yields.

Besides the effect it has in yields, the H₂ treat rate has a large effect in the solvent vaporization in the slurry preheat furnace. If a solvent similar to the 1975/1976 study design solvent is used, reducing the H₂ treat rate would reduce the vaporization in the slurry furnace. However, using the lower H₂ treat rate would allow the use of a lighter solvent than could be otherwise possible without substantially increasing the vaporization in the furnace.

The purpose of this study is to quantify any savings realized from a 25% reduction in the liquefaction H₂ treat rate. The driving force for

this reduction is the decrease of the treat gas circulation rate. This decrease could result in significant savings in investment and operating costs, particularly in the cryogenic H₂ recovery unit and in the H₂ compression facilities. At the same time lowering the H₂ treat rate could alleviate some of the potential problems caused by the vaporization of solvent in the slurry furnace. The results obtained from this study are expected to be useful in current and future evaluations of process improvements.

Basis

The basis for evaluating the equipment changes caused by using the low H₂ treat rate is the Illinois base case plant of the 1975/1976 EDS Study Design. This plant uses a 4 wt% on coal H₂ treat rate in liquefaction. Thus, a 25% reduction lowers the treat rate to 3 wt% on coal. The makeup H₂ is provided by steam reforming C₁/C₂ hydrocarbons and by cryogenically purifying the plant's purge gas streams. The purity of the H₂ treat gas to the liquefaction unit was maintained at 80 mole% H₂, (the value in the 1975/1976 study design). This purity was achieved by varying as necessary the amount of liquefaction purge gas sent to the cryogenic H₂ recovery unit.

To simplify the evaluation it was assumed that the liquefaction H₂ consumption is independent of the H₂ treat rate utilized. It was further assumed that the solvent quality is sufficiently high such that the no increase in donor hydrogen is needed to compensate for the lower treat rate. Thus, the lower H₂ treat rate has no effect in the H₂ consumption or in the product yields (or quality) from the unit. These assumptions will result in the largest possible incentives for reduction in treat rate.

Equipment Modifications

To operate at the lower H₂ treat rate no major changes in the 1975/1976 study design liquefaction unit are needed. However, due to changes in vaporization and heat duties, the size of some sections within the liquefaction unit have changed. The major change is a 26.4% decrease in the size of the recycle gas separation section. This reduction includes a 25% decrease in the size of the treat gas compressor. The reduction is mostly the result of using the lower treat gas rate, which for the same H₂ consumption produces a smaller amount of vapor. Thus, the amount of hot vapor available for preheating the slurry feed is much smaller than in the 1975/1976 study design. To achieve the same reactor temperature, the slurry furnace must compensate for the smaller amount of preheating. As a result, the duty of the slurry furnace increases by 1%, even though the solvent vaporization is lower. The only other liquefaction equipment change occurs in the APS/VPS where its feed increases by ~1%. This increase is caused by the lower vaporization in the separation equipment.

The feed to the solvent hydrogenation unit is unchanged since no change in liquefaction product quality or yields is assumed. It was also assumed that the quality and rate of the solvent required in the liquefaction unit remains unchanged. Thus, no equipment modifications are needed in the solvent hydrogenation unit.

Other sections of the plant affected by the lower H₂ treat rate are the H₂ generation, recovery and compression facilities. In order to achieve the desired liquefaction H₂ treat gas purity, the purge gas rate from the unit is 43.8% lower than in the 1975/1976 study design. This reduces the size of the cryogenic H₂ recovery unit by 37.7%. In addition, the steam reforming unit decreases in size by 1% due to slightly lower hydrogen losses. Overall, the H₂ make-up rate to liquefaction and solvent hydrogenation decreases by 10%. This represents an equal decrease in the size of the plant's H₂ compression facilities.

Investments and Operating Costs

The liquefaction equipment modifications described above cause the liquefaction unit investment, when using a 3 wt% on coal H₂ treat rate, to decrease by 26.6 M\$ (1985) relative to a unit using a 4 wt% on coal H₂ treat rate. The investment in both cases reflects the use of the optimum liquefaction conditions (840°F and 40 minutes). All of the liquefaction unit investment reduction (28 M\$) is due to the decrease in the size of the recycle gas separation equipment when using the lower treat rate. The net decrease in liquefaction investment is lower due to small increases in the slurry furnace (0.8M\$) and the APS/VPS (0.6 M\$). In addition, the investment for the plant's hydrogen facilities is also lower. The largest decrease is in the cryogenic unit where the smaller size reduces the investment by 9.4 M\$ (1985). The investment in the compression facilities and steam reforming decrease by 3.7 M\$ (1985) and 2 M\$ (1985), respectively. The total reduction in plant investment is 41.7 M\$ (1985). This represents a reduction of 7.2 M\$/yr (1985) in the capital recovery charges of the plant.

The plant's operating costs decrease as a result of lower maintenance costs, lower manning requirements and lower utility needs. The investment-related operating cost decrease by 2.2 M\$/yr (1985). These include the cost of wages, salaries, repair materials, etc. Utility costs decrease as a result of lower power needs in the compression facilities and a net decrease in the plant's fuel consumption. Power costs decrease by 4.9 M\$/yr (1985), while the fuel savings amount to 0.2 M\$/yr (1985). The fuel value assumes that supplemental coal is being used in the FLEXICOKING unit. Thus, the plant's operating costs decrease by 7.3 M\$/yr (1985).

Table 2-3 summarizes all the changes in the investments and operating costs of the plant.

TABLE 2-3

LIQUEFACTION H₂ TREAT RATE SENSITIVITY STUDYInvestments, M\$ (1985)

	<u>4 Wt% H₂</u>	<u>3 Wt% H₂</u>	<u>Δ</u>
● Liquefaction			-
+ Slurry Drying	115.7	115.7	-
+ Slurry Furnace	192.6	193.4	0.8
+ Reactors	61.2	61.2	-
+ Recycle Gas Separation	165.7	137.7	(28.0)
+ Fractionation	44.4	45.0	0.6
Liquefaction Total	579.6	553.0	(26.6)
● Solvent Hydrogenation	189.2	189.2	-
● Steam Reforming	327.5	325.5	(2.0)
● H ₂ Compression	85.4	81.7	(3.7)
● Cryogenic H ₂ Recovery	46.2	36.9	(9.4)
Total			(41.7)
Δ <u>Capital Recovery Charges</u>	(7.2)		
Δ <u>Operating Costs, M\$/yr (1985)</u>			
● IROC	(2.2)		
● Fuel	(0.2)		
● Electric Power	(4.9)		
	(7.3)		
Δ <u>Total Plant Cost, M\$/yr (1985)</u>	(14.5)		

Results

The net effect of decreasing the liquefaction H_2 treat rate from 4 wt% on coal to 3 wt% on coal is to decrease the total plant cost by 14.5 M\$/yr (1985). This represents an equivalent increase in plant net cash flow assuming that the product revenues remain unchanged. As indicated earlier, this reduction in Plant costs is based on the assumption that there is no reduction in Plant yields and, therefore, represents the maximum incentive for reducing treat rate.

2.7 EDS Process Alternatives LP Model (PAM): Ash-Free Formulation

During the past year, an ash-free formulation was developed for the EDS Process Alternatives LP Model (PAM). The ash-free basis involves following the ash content in coal and vacuum bottoms as a separate stream in the Model. Without this formulation, PAM is unable to differentiate between the hydrocarbon and ash content of coal and bottoms. The ash-free basis provides additional flexibility in running PAM; for example, in varying the ash content of a coal and/or changing liquefaction operating severity, which affects the ash content of vacuum bottoms. The ash-free basis affects all vectors that feed coal and vacuum bottoms such as liquefaction/solvent hydrogenation, coal-fired boiler, FLEXICOKING and partial oxidation.

In developing the ash-free model, it is necessary to distinguish between the effects of organic matter and ash on the yields, utilities and investments of coal and bottoms processes in PAM. For example, the ash level in coal has a significant effect on the investment costs in liquefaction/solvent hydrogenation. The ash has to be dried and heated in the slurry furnace and the solvent/coal ratio is based on coal plus ash. Preliminary studies indicated that ash throughput could incur about 50 to 65% of investment costs relative to organic coal feed. In the future, when new coals are evaluated in PAM, more definitive studies will need to be conducted to determine investment/utilities effects relative to ash content.

Ash-free vectors, developed for the Illinois coal base case of the 1976 EDS Study Design, were tested and validated to confirm the accuracy and reliability of the formulation. An ash-free validation was also completed for the Wyoming Coal Study Design (next item). The ash-free basis was also used in the Wyoming coal process variables study (see item 2.9).

2.8 EDS Process Alternatives LP Model Validation for Wyoming Coal

Validation of the EDS Process Alternatives LP Model (PAM) for Wyoming coal was completed early in 1978. Results of this study were presented in the January-March, 1978 Quarterly Technical Progress Report and are summarized here (FE-2893-12).

The 1976 EDS Wyoming Study Design was represented by changing existing Illinois coal vectors and adding new vectors specific to Wyoming coal. The results of the Wyoming coal LP simulation were then compared to the Wyoming Study Design to ensure that the Model results were consistent with those in the Study Design. In cases where particular Wyoming coal process blocks were assumed to be the same as or a percentage of comparable Illinois coal blocks, The Wyoming LP Model results were directly compared to the base case Illinois LP Model, after adjustment to reflect the 1976 Wyoming location factor difference on investments.

Validation Reflects a "Revised" Wyoming Study Design

During the validation, two revisions were made to the Wyoming Study Design. The first was a change in the yield basis. A study on actual plant recoveries indicated that the vacuum bottoms yield from the liquefaction section is slightly less (< 2% difference) than originally estimated. Therefore, the naphtha and LSFO yields are correspondingly higher. The second revision was a change in total investment. Validation of the Illinois LP Model (see Interim Report, FE-2353-19) revealed that, due to a change in the investment escalation factor and elimination of roundoff, the total plant investment was about 2% lower than that shown in the Illinois Study Design. Since the Wyoming total plant investment was calculated as a percentage of the Illinois investment, the Wyoming investment was also lowered.

In addition, the total plant investment in the LP Model includes two items mentioned in the Wyoming Study Design but not specifically included in the design calculations. First, due to the higher moisture content of Wyoming coal, additional slurry driers and hence, more 600-psig steam are needed as compared to the Illinois coal base case. Secondly, potential material changes resulting in investment savings can be made downstream of the liquefaction hot separator, since the separator temperature is lower than in the Illinois base case. Estimates of these two effects were included in the Wyoming coal validation.

Good Closure Between LP Model and Study Design

Closure between the Wyoming LP Model and the Revised Study Design has been very good. There is, however, a major difference between the model simulation and Study Design. A detailed fuel balance was not prepared for the Wyoming Study Design; fuel consumption was assumed to be the same as in the Illinois Study Design base case. However, with the inclusion of the additional slurry driers, fuel consumption in the Wyoming LP simulation has increased such that high-Btu gas is needed to supplement low-Btu gas for fuel, requiring the use of propane (C₃) product as a marginal reformer feed, which reduces the net propane product yield. Also, the additional slurry driers nearly double the consumption of 600-psig steam.

Results of the Wyoming Coal Study Design Validation are shown in Tables 2-4 and 2-5. Table 2-4 compares the feed and product rates. The revised Wyoming Study Design case reflects the yield changes discussed earlier. The Wyoming LP simulation and Revised Study Design feed and product rates agree closely. Table 2-5 shows the new required initial selling price (RISP) of Wyoming coal liquids based on the LP simulation. The 1975/1976 Wyoming Study Design estimated an RISP of 37.00 \$/B (high FLEXICOKING yield case). The LP simulation shows an increase to 38.06 \$/B which reflects primarily the loss of C₃ product which is needed as a feed to the steam reformer to replace high-Btu gas needed for fuel.

2.9 Wyoming Coal Process Variables Study

Work on the Wyoming Coal Process Variables Study is nearing completion. As reported previously, the EDS Process Alternatives LP Model (PAM) is being used to determine the optimal liquefaction operating conditions, with different bottoms processing configurations. The results of this study will be useful in setting the preferred operating conditions for the Wyoming Coal Study Design Update which is scheduled to begin later this year.

Liquefaction yields were determined by EPRL's EDS Process Integrated Simulation Model at the following temperatures and residence times:

- 800°F at 15, 25, 40, 60, 80, 100 and 120-minute residence times,
- 840°F at 15, 25, 40, 60, 80 and 100-minute residence times,
- 880°F at 15, 25, 40, 60, and 100-minute residence times.

Investments, utilities, and operating costs for each case were based on the 1975/1976 Wyoming Coal Study Design. The liquefaction investments were adjusted to reflect the effects of temperatures and residence times different from those in the Study Design (840°F, 25-minute residence time). Similar to the process variables study completed during Phase IIIA for Illinois coal, liquefaction investments were modified to include equipment changes and adjustments for higher vapor rates at the reactor inlet and outlet in some cases relative to the Study Design.

Various Processing Alternatives Studied

Cases evaluated thus far for this study include:

- Once-through FLEXICOKING and partial oxidation for hydrogen production at various liquefaction conditions. No supplemental coal allowed. (Type "A" cases),

TABLE 2-4

WYOMING COAL PAM-LP VALIDATION
FEED AND PRODUCT RATES

<u>Rates</u>	<u>1976 Wyoming Study Design Basis</u>	<u>Revised Wyoming Study Design</u>	<u>Wyoming LP Model Simulation</u>	<u>Δ</u>
Coal, "as received", kT/SD	20.8	20.8	20.8	-
C ₃ and C ₄ LPG, kB/SD	7.6	7.6	7.7(2)	0.1
Naphtha, kB/SD	20.4	20.9(1)	20.9	-
LSFO, kB/SD	24.7	25.3(1)	25.4	0.1
Sulfur, LT/SD	125	125	124.2	-0.8
Ammonia, ST/SD	85	52.1(3)	52.1	-

Notes:

- (1) The naphtha and LSFO yields have been modified from those previously reported due to a change in yield basis. The Wyoming Study Design assumed the same vacuum bottoms yield as the Illinois Study Design, while the Model, which is based on an elemental balance, has a slightly lower (<2%) vacuum bottoms yield. The naphtha and LSFO yields are, therefore, slightly higher when using the elemental balance yields.
- (2) Includes the C₃ used as reformer feed.
- (3) The Model yield for the liquefaction and solvent hydrogenation sections is based on the elemental balance yield of 0.25 wt% NH₃ on dry coal, while the Study Design is based on pilot plant data which indicated an NH₃ yield of 0.4 wt% on dry coal.

TABLE 2-5

WYOMING COAL PAM-LP VALIDATION
REQUIRED INITIAL SELLING PRICE

	Costs, M\$/Yr (1985)		
	1976 Wyoming Study Design(1)	Wyoming LP Model Simulation	Δ
Coal Feed @ 6.50 \$/T	54.32	54.32	-
Byproducts, (S, NH ₃) (2)	(8.31)	(6.07)	2.24(4)
Utilities	31.39	34.92	3.53(5)
Catalysts and Chemicals	8.62	7.48	-1.14
Repair Materials, Salaries and Related Costs	115.26	107.60	-7.66(6)(7)
Capital Charges (3)	<u>368.09</u>	<u>359.90</u>	<u>-8.19(7)</u>
Total Liquids Costs, M\$/Yr	569.37	558.15	-11.22
\$/B	37.00	38.06	1.06(8)
Total Liquid Products, kB/SD	52.7	50.2(8)	

Notes:

- (1) "High FLEXICOKING" yield case from 1976 Wyoming Study Design.
- (2) Byproduct sulfur at 64 \$/LT and ammonia at 228 \$/ST.
- (3) Represents a 15% DCF on a current dollar basis.

The difference in costs is due to the following factors:

- (4) Lower ammonia yields from the liquefaction and solvent hydrogenation sections in the LP simulation case.
- (5) Extra power must be purchased in the LP simulation rather than generated as in the Study Design.
- (6) Calculated as 5.54% of TEC in the Study Design and as 5.3% of TEC in the LP simulation case.
- (7) Different investment basis. The LP simulation investment is based on the revised Illinois base case investment and includes the investments for the additional slurry driers and credit for material changes downstream of the liquefaction hot separator.
- (8) Lower liquid products yield in the LP simulation case since some of the propane is used as reformer feed.

- Once-through FLEXICOKING, partial oxidation for hydrogen production, and a coal-fired boiler to meet offsite steam requirements at various liquefaction conditions (Type "B" cases),
- Once-through FLEXICOKING, steam reforming for hydrogen and coal-fired boiler at various liquefaction conditions (Type "C" cases).

Preliminary case results are presented in Figure 2-1. The reference case was considered to be the Wyoming Study Design Validation (Case 801, see Item 2.8). All cases are fuel short except at 840/15 and 800/40 conditions. Fuel short cases will choose a coal-fired boiler to satisfy offsite steam requirements. Purge gas is used to satisfy onsite fuel demands if there is insufficient low Btu gas (LBG) produced. In the type "A" cases, however, no supplemental coal is allowed, and LSFO product is burned to meet offsite fuel demands. Figure 2-1 shows, as expected, that the type "B" cases, which use a coal-fired boiler, have a substantial economic advantage over the type "A" cases since the supplemental coal reduces the need to burn valuable product as fuel. The 840°F and 880°F type "B" cases show a higher profitability than the corresponding 800°F cases since, at the less severe 800°F operation, there is a much lower ratio of product revenues to plant investment. Note that most of the type "A" and "B" cases show a higher net cash flow than the Wyoming Study Design Validation base case, since the validation case had a steam reformer for hydrogen production and used propane product as the marginal reformer feed.

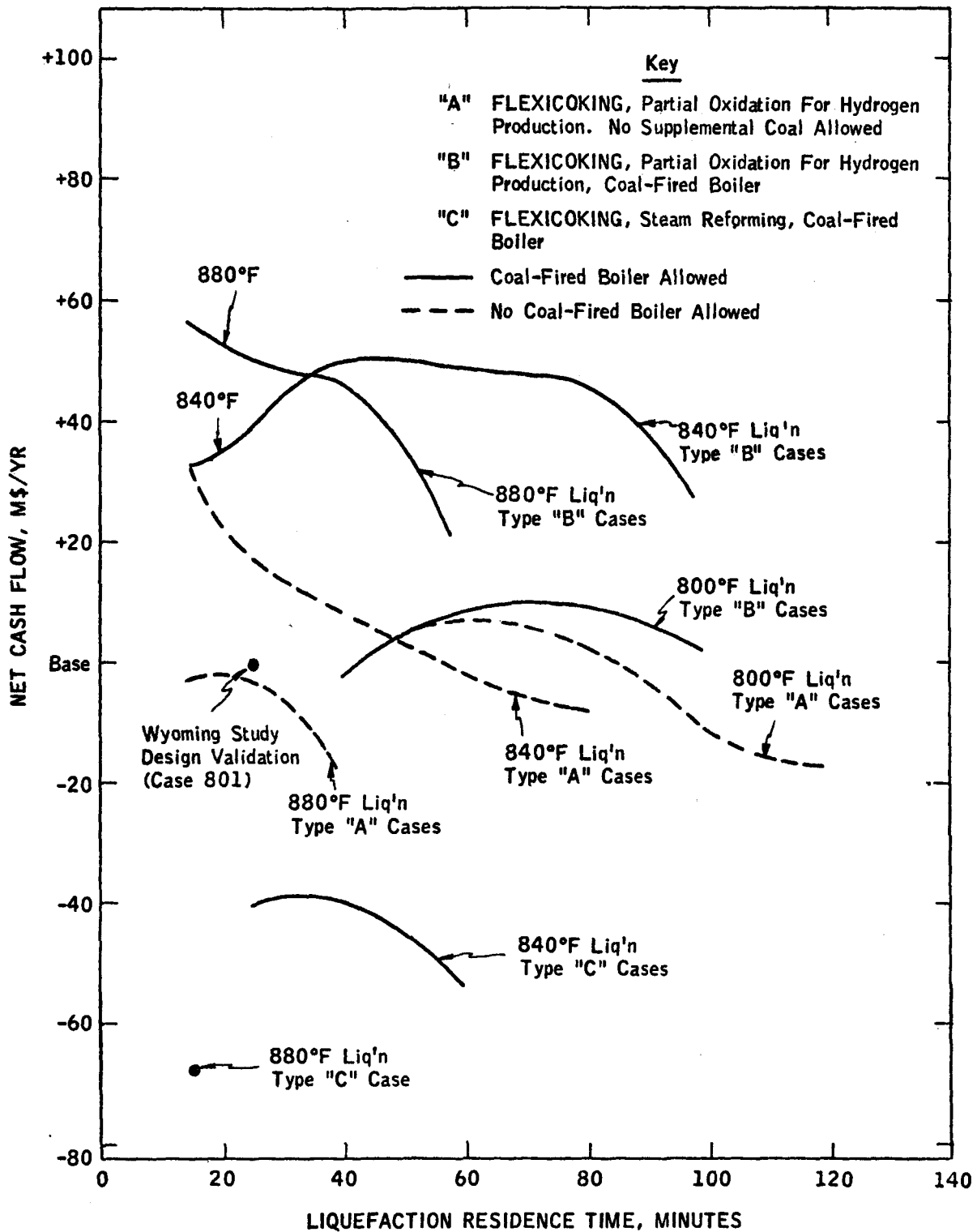
The 880°F type "B" cases at 15 and 25-minute residence times show a small economic advantage over the 840°F cases at 40-80 minute residence times, but, as was reported for Illinois coal in the Quarterly Technical Progress Report for July 1-September 30, 1977,* the same potential operability problems at 880°F could eliminate this economic advantage. For example, the use of a heavier solvent to suppress solvent vaporization at the liquefaction reactor inlet involves modification of the atmospheric pipestill overhead system. Other potential problems, such as high film temperature in the slurry furnace tubes, could affect plant service factor. In addition, a decrease in estimated product yields at 880°F would lead to decreased plant profitability. For example, a decrease of 0.5 wt% in liquid yield reduces net cash flow by 2-3 M\$/yr. Considering these potential problems and risks at an 880°F operation and the small economic debit in going to 840°F, the 880°F cases were eliminated from consideration.

Figure 2-1 shows that the 840/40 case with once-through FLEXICOKING, partial oxidation for hydrogen and a coal-fired boiler would then have the highest net cash flow. However, the question of bottoms viscosity is still to be resolved. Wyoming vacuum bottoms tend to be much more viscous than Illinois bottoms and could lead to operability/pumping problems. Increasing residence time reduces the viscosity. Should further study on the viscosity issue reveal that the liquefaction residence time must be lengthened, Figure 2-1 shows that increasing severity to 840/60 or 840/80 minutes has only a minor impact on net cash flow (less than 5 M\$/yr).

*FE-2893-3

Figure 2-1

EDS WYOMING LIQUEFACTION PROCESS VARIABLES STUDY
NET CASH FLOW VS. RESIDENCE TIME
PRELIMINARY RESULTS



Partial Oxidation for Hydrogen Shows Advantage Over Steam Reforming

It is apparent from Figure 2-1 that the once-through FLEXICOKING partial oxidation cases have a higher profitability than the corresponding FLEXICOKING/steam reforming cases (type "C"). This is due primarily to the fact that high-Btu gas (C_1/C_2) is available as an additional product and is assumed to have a plant netback value in fuel parity with low sulfur fuel oil. The steam reforming cases use most, if not all, of the high-Btu gas available as reformer feed, while the partial oxidation cases have all the high-Btu gas available (except that which is lost in the purge gas used as fuel) as product. The C_1/C_2 can yield a delta net cash flow of close to 100 M\$/yr in some cases.

Sensitivity to High-Btu Gas and Scrubber Bottoms Product Values Under Evaluation

Since the future sale of high-Btu gas is uncertain and may be regulated, a sensitivity study is being done to determine how lowering the product value of high-Btu gas changes the optimal plant configuration. The product value of the once-through FLEXICOKING scrubber bottoms is also being lowered to reflect potential marketability problems. Work to date shows that when the product values of both high-Btu gas and scrubber bottoms are lowered to about two-thirds of their original values,⁽¹⁾ net cash flow decreases, but there is no change in plant configuration. Note that the 880/15 and 880/25 cases will lose much of their small economic advantage over the 840°F cases since the higher severity 880°F cases produce more gaseous product.

When the product values of high-Btu gas and scrubber bottoms are both lowered to about one-quarter of the original values in the 880/15 cases, there is a basis change. FLEXICOKING becomes unattractive, and the vacuum bottoms not used to make hydrogen by partial oxidation are assumed to be sold as a by-product. Sales of high-Btu gas is no longer profitable; therefore, it is used as plant fuel. A coal-fired boiler is still used to meet offsite steam requirements. Since high-Btu gas sales make up about 25% of the total product revenues, the net cash flow for this case is sharply decreased. As the values of scrubber bottoms and high Btu gas are not directly related, further work is needed to define the sensitivity to the values of each of these separately.

Future Work Includes Additional Bottoms Processing Options

Work on this study is continuing in the EDS Bottoms Processing Section. New vectors being prepared include:

Note:

(1) High-Btu gas: 266.7 \$/FOET, scrubber bottoms: 216.5 \$/FOET.

- Recycle FLEXICOKING
- Air and Oxygen Moving - Bed Coal Gasification

These vectors will be used to further study configurations with Wyoming coal. In addition, the question of bottoms viscosity will be evaluated to determine the potential impact on plant operability at various liquefaction conditions.

2.10 PAM-LP Model Extension - Steam/Power Generation

Work began on extending the Process Alternatives LP Model (PAM) to evaluate alternate sources of steam/power generation. Currently, the PAM model meets makeup steam requirements in offsite boilers and has the single option to purchase electric power. The objective of this study is to add vectors to the PAM-LP model to generate total plant electric power requirements and to generate steam and power in a combined operation (cogeneration) using coal and vacuum bottoms as fuels.

The vector option for generating electricity involves a 200-MW capacity coal-fired power plant equipped with flue gas desulfurization facilities. The investment and operating costs for this vector are based on information from literature and vendor sources.

Cogeneration is a commercially used technique which produces medium-pressure steam and power from high-pressure steam. A boiler delivers high-pressure steam (~1800 psig) to a back-pressure turbo-generator thereby making electricity which can be used in the plant. The steam, now at a lower temperature and pressure (~600 psig) is piped to the process plant. Several cogeneration vectors are to be added to the LP model. One vector will generate 0.5 Mlbs/hr of 600-psig steam for an EDS plant, plus an optimum quantity of cogenerated electric power. An additional vector for a cogeneration operation will produce the approximate EDS plant requirements of both steam and power. Vectors will allow the use of coal or vacuum bottoms as fuels for cogeneration. Investment and operating costs for the facilities have been estimated from general equipment requirements, vendor contacts and the literature.

Preliminary vectors for the above steam/power alternatives have been prepared. Revised data on flue gas desulfurization costs are expected shortly and the vectors will be incorporated into PAM once this new information has been evaluated.

2.11 Hydrotreating of Heavy Coal Liquids

A screening study to examine the costs of hydrotreating a coal liquids stream consisting of liquefaction VGO (800/1000°F) and coker LSFO (400/1000°F) has been completed. Two hydrotreating modes were considered. The first, hydrodenitrogenation (HDN), reduces the feed nitrogen level from

1.18 to 0.5 wt%, with the accompanying sulfur level dropping from 0.99 to 0.08 wt%. The second, less severe mode, is hydrodesulfurization (HDS). Here, the sulfur content is reduced from 0.99 to 0.2 wt%, and the nitrogen level lowered to 0.9 wt%.

The feedstock properties are estimates based on the EDS Study Design Update. The Exxon Engineering Petroleum Department (EPPD) provided process conditions and investment parameters for the suggested RESIDFINING* operations.

Table 2-6 shows a summary of the estimated hydrotreating costs. Investments and annual costs for the two hydrotreating modes are given in 1975\$ and based on 10.3 kB/SD of feed. TEC investments range from 18 to 24 M\$ with annual operating costs of 11.9 to 18.5 M\$. Hydrotreating costs are 3.50 \$/B RESIDFINING feed for the HDS case and 5.50 \$/B RESIDFINING feed in the HDN operation. Hydrogen consumption represents 60-70% of the cost in both cases.

2.12 Phenolic Coal Naphtha Upgrading

The economics of the water extraction process to remove phenolic compounds from coal naphtha, as given in the EDS Phase IIIA Final Report, were updated to include new investment and operating costs for the phenolics recovery unit based on a vendor quotation. These costs apply specifically to a recovery unit processing the total phenolic water feed as defined in the study. As the table below shows, the revised TEC investment of 32 M\$ is significantly less than the 66 M\$ in the original case. Two changes are responsible for this difference. First, and of prime importance, the investment quotation from the vendor was almost half of the original estimate, which had been escalated from a smaller-capacity unit. (The phenolics recovery unit has greater economies-of-scale than were previously anticipated). Second, since the major investment cost of the water extraction process came from vendor information based on commercial-sized units, justification existed to reduce the Process Development Allowance (PDA) from 50% to 25%.

	<u>Original Economics</u>	<u>Revised Economics</u>
TEC Investment (1985\$)	66 M\$	32 M\$
Total Revenue from Recovered Phenolics, M\$/year	33.2	33.2
Raw Material Cost (Phenolics-in-Naphtha), M\$/year	(10.4)	(10.4)
Operating Costs, M\$/year	<u>(24.1)</u>	<u>(15.3)</u>
Net Revenue, M\$/year	(1.3)	7.5
Δ Cost/Bbl Coal Liquids, \$/B	0.07	(0.43)

* Service Mark

TABLE 2-6

HYDROTREATING OF HEAVY COAL LIQUIDS
SUMMARY OF HYDROTREATING COSTS

<u>Hydrotreating Mode</u>	<u>HDN</u>	<u>HDS</u>
Hydrogen Consumption, kSCF/B Feed	2.4	1.3
Total Erected Cost, (TEC), M\$ (1975) ⁽¹⁾	24	18
<u>Annual Costs, M\$ (1975)</u>		
Hydrogen ⁽²⁾	13.0	7.1
Capital charges	4.1	3.1
Other ⁽³⁾	1.4	1.7
Total Annual Costs, M\$ (1975)	<u>18.5</u>	<u>11.9</u>
Hydrotreating Costs, \$/B RESIDFINING feed (1975)	5.50	3.50

Notes:

- (1) Includes 10% Process Development Allowance (PDA)
- (2) Valued at 1.60 \$/kSCF.
- (3) Includes utilities and steam credit, salaries, repair and maintenance costs.

Reducing the investment and revising the utility requirements has a marked effect on the annual operating costs. The table above illustrates that, while product revenues and raw materials (phenolics) costs remain unchanged, the revised operating costs of 15.3 M\$/yr are 63% of the original costs of 24.1 M\$/yr. This lower cost gives the revised case a positive net revenue of 7.5 M\$/yr which would incrementally decrease the cost of coal liquids by about 0.43 \$/B, instead of the slight increase in cost as shown in the original case.

2.13 Economic Evaluation of Higher-Pressure Liquefaction

A brief screening study was carried out to define the economics of increasing liquefaction reactor inlet pressure in the Exxon Donor Solvent process (EDS) from about 1500-psi to 2500-psi H₂ partial pressure (3125-psig total pressure). This evaluation was based on several RCLU-II yield periods on Illinois coal generated at 25-minute reactor residence time and 840 and 880°F. The incentive to operate at the higher pressure was estimated using guidelines developed from the EDS Process Alternatives LP Model.

Although the data base are limited, this evaluation indicates that an increase in liquefaction reactor pressure to 2500-psi H₂ partial pressure is not economically attractive at either 840 or 880°. This is largely due to a very significant increase in the liquefaction reactor investment resulting from the higher pressure together with the requirement for a more costly alloy (probably 5 Cr-1/2 Mo) of reduced strength to avoid susceptibility to high pressure hydrogen attack. Furnace, separation, and hydrogen makeup compression costs are also increased. Even an assumption that the solvent hydrogenation system could be eliminated at the higher liquefaction pressure with no yield or operability debits appears insufficient to make higher-pressure liquefaction economically attractive.

2.14 Recycle Treat Gas Integration Study

The purpose of this study was to evaluate the incentives for incorporating into the EDS Commercial Plant Study Design any of three alternative schemes for recycling hot treat gas between the liquefaction and solvent hydrogenation units. The most promising of the three schemes, as determined by a preliminary study, was studied in detail including an economic evaluation.

Economic results, compared to the 1975/1976 Study Design base case, showed a saving in the cost of liquid products of about 0.05 \$/Bbl (year 1985) but sensitivity studies revealed that inaccuracies in estimating the delta investment costs could easily swing the results by $\pm$ 0.25 \$/Bbl. It was concluded that none of the three alternatives studied for integration of the recycle treat gas circuits appears sufficiently attractive compared with the base case to justify the operational risks and unwarranted complexity associated with the integration of units. More detailed information on this study can be found in the July-September, 1977 Quarterly Report (FE-2893-3).

2.15 Heat Integration Study

This study was undertaken to check the practicality of the heat integration scheme in the 1975/1976 EDS Coal Liquefaction Commercial Plant Study Design, to identify areas where heat losses have a significant impact on the economics and the overall energy efficiency of the same Study Design, and to determine means of recovering and utilizing the maximum amount of useful heat from an EDS plant.

The study showed that the existing heat integration schemes in all sections of the Study Design are fully operable. Minor changes in the liquefaction and solvent hydrogenation sections were identified which result in a 1.2 M\$ decrease in the 1985 plant TEC and a 0.66 M\$/yr reduction in plant operating costs. Overall, the cost of liquid products was reduced by 5¢/Bbl (year 1985). More detailed information on this study can be found in the July-September, 1977 Quarterly Report (FE-2893-3).

2.16 EDS Wastewater Optimization Study

An investigation has been completed to identify the areas of water usage for a commercial coal liquefaction plant and to determine ways to minimize fresh water requirements and the amount of sour, contaminated water sent to treating facilities. Emphasis was placed on identification of applications which might prove necessary for plants located in water-short Western locations such as Wyoming. The starting basis for the study was the process scheme and water rates as presented in the EDS Commercial Plant Study Design.

A major result of the investigation was the development of a possible water-shedding sequence which might be necessary should water not be available or should restrictions be placed on the quantity of plant effluent water. The water-shedding sequence is composed of three major steps.

Step 1 involves implementation of cost-effective modifications. Cost-effective is used to describe those modifications which are at worst marginally attractive when their economics are looked at individually and not as a step to reduce overall plant water consumption. Implementation of all the cost-effective modifications reduces the base case raw water makeup requirement by 21%. Implementation of these modifications also decreases the plant sour water treating load to approximately one-half of the base case rate.

Step 2 involves a major change to the Study Design basis. This step assumes production of hydrogen using partial oxidation (PO) instead of steam reforming. With such a step, water for the hydrogen production reaction no longer has to be of first-rate quality and makeup requirements can come from treated effluent water instead of from demineralized raw water. For both Illinois and Wyoming locations, raw water makeup is reduced by 15% of the base case requirement.

Step 3 is also a major step and involves the use of treated plant effluent water to supply the cooling tower makeup requirements instead of fresh water. Implementation of this step creates several operability problems for the recirculating cooling water system. However, this step can reduce the plant's raw makeup water needs by 36% of the base case requirement and is likely to become a viable alternative by the time a commercial plant is built.

More detailed information on this study can be found in the July-September, 1977 Quarterly Report (FE-2893-3).

2.17 Gas Treating and Sulfur Recovery Study

A study was undertaken to evaluate alternative gas treating and sulfur recovery schemes for a commercial Wyoming EDS coal liquefaction plant. The EDS Commercial Plant Study Design for Illinois coal incorporates a diethanolamine (DEA) gas treating system for H_2S and CO_2 removal from sour gas streams followed by a Claus plant for sulfur recovery. While the DEA/Claus plant combination is suitable for high sulfur coals such as Illinois coal, for low sulfur Wyoming coal this combination would likely be inoperable because of the low ratio of H_2S to CO_2 in the sour gas streams. The reason for this is that Claus plant operation becomes difficult when the H_2S/CO_2 mole ratio in the feed to the plant is less than 1/3, as a minimum concentration of H_2S is needed to support combustion.

A literature search and a review of in-house information was undertaken to find suitable gas treating and sulfur recovery processes for Wyoming coal. The first scheme developed involved a DEA scrubbing system with a Stretford sulfur recovery unit, as the Stretford process is able to handle any H_2S/CO_2 mole-ratio sour gas stream. The second scheme developed consisted of a split DEA system, with the low H_2S/CO_2 ratio sour gases treated by a DEA system followed by a Stretford, while the sour gases that have a H_2S/CO_2 ratio acceptable for Claus plant feed are treated in a separate DEA system followed by a Claus plant. Two schemes involving the recycle of sulfur as H_2S to the Claus plant feed to raise the H_2S/CO_2 ratio to a combustible level were also developed. However, both schemes were eliminated because of excessive costs when compared to the first two schemes, the major expense being H_2 consumption to produce the H_2S . Two solvents which selectively remove H_2S in the presence of CO_2 were also investigated as possible replacements for DEA in the DEA/Claus plant scheme. These commercially available proprietary solvents were investigated to find out if they could produce a Claus plant feed with an acceptable H_2S/CO_2 mole ratio. One process licensor advised us that his process could not economically compete with either of the first two schemes, and the other process licensor advised us that his process could not produce an acceptable Claus plant feed for our Wyoming coal case.

Screening quality designs and cost estimates were prepared for the first two schemes. The two schemes were nearly equal in cost, both representing a cost increase over a comparably sized DEA/Claus plant combination.

Exxon Engineering gas treating specialists have subsequently advised us that the split DEA system, with the low H_2S/CO_2 ratio sour gases segregated from the high H_2S/CO_2 ratio sour gases, each in their own DEA absorber/regenerator train, could yield an acceptable Claus plant feed. The Claus plant would be split flow, with the rich H_2S/CO_2 regenerator offgas being used to start combustion, and the lean H_2S/CO_2 regenerator offgas blended in downstream of the combustion stage. Their computer simulations showed that a sulfur recovery of about 94% is attainable with two catalytic converters, with additional recovery available from sulfur plant tailgas cleanup. As this scheme is viable and the most economical of all the schemes considered, it appears to be the most promising method of treating the sour gas streams from a commercial Wyoming EDS coal liquefaction plant. Further consultations with Exxon Engineering gas treating specialists will be conducted during the Wyoming Study Design Update to insure that the proposed scheme will indeed be operable.

2.18 Prediction of EDS Commercial Plant Wastewater Contaminants

Work was completed on a study to predict the organic and inorganic pollutant levels in the sour water streams from a commercial EDS Illinois coal liquefaction plant. The predicted sour water contaminant levels were developed via computer simulation and will result in better definition of the wastewater treating facilities needed in a commercial plant. The computer simulations are based upon sour water data taken from the 1 ton/day Coal Liquefaction Pilot Plant (CLPP) and recent bench scale sour water experiments. The computer simulations take into account changes in equipment, rates and conditions between experimental and commercial plant operations, and attempt to predict the effect of these changes on contaminant levels in sour water streams. The results of this study can be found in the January-March, 1978 Quarterly Report (FE-2893-12).

2.19 TACB Construction Permit Application for ECLP

Application to the Texas Air Control Board (TACB) for a Construction Permit was made on December 27, 1977, and the permit was issued to the Carter Oil Company on February 27, 1978. The permit covers construction only. Application for an operating permit must be filed upon completion of construction.

Changes to the plant design which are required to meet TACB requirements on atmospheric emissions have been made. Included was a Design Basis revision for weathering naphtha continuously and routing volatiles to the pilot plant fuel gas system instead of weathering batch-wise with volatiles routed to the flare.

TACB comments on particulate emissions from the coal preparation facilities and fugitive emissions of Non-Methane Hydrocarbons from the processing units are being reflected in the detailed design. Information was provided to the engineering contractor to facilitate his work in this area.

2.20 ECLP Support Activities

● ECLP Product Disposal

A Design Basis Memorandum was prepared for ECLP Product Disposal facilities. The scheme selected involves burning the ECLP naphtha and solvent as fuel in dedicated furnaces in the Baytown Chemical Plant. Vacuum Bottoms will either be sent to a prototype FLEXICOKING unit or be trucked to an approved landfill site. Vacuum gas oil (VGO) will either be mixed with vacuum bottoms and sent to a FLEXICOKING prototype or be sent by asphalt truck to the Baytown Refinery for blending off with one of their product streams.

Naphtha and solvent for disposal will be stored in two tanks. One will contain product which meets or exceeds the furnace fuel sulfur specification and the second will be for liquefaction naphtha which has not been hydrotreated in the Solvent Hydro unit. As much high-sulfur naphtha as possible will be blended off with low sulfur naphtha/solvent to just meet the furnace fuel sulfur specification. Any remaining high sulfur naphtha would be desulfurized in the solvent hydrogenation unit during periods when this can be accommodated. Naphtha hydrotreating alternatives will be evaluated at a later date.

Vacuum Gas Oil (VGO) produced in ECLP will be disposed of by blending off with a product from Exxon's Baytown Refinery. Laboratory tests were completed that showed that the VGO is compatible with the product over a wide range of concentrations and storage temperatures. Permission to include the VGO in shipments has been obtained from customers of this product. A design specification for the facilities required to blend the ECLP VGO into refinery product at the Refinery tank farm will be completed in July.

● Sponsors Product Sample Tankage

The design basis for use in the facilities design at ECLP to prepare, store and ship samples of product naphtha and fuel oil was completed. Bulk storage of samples will be remote from ECLP and will be the responsibility of the individual sponsors who take samples.

Samples of naphtha and fuel oil of up to 2000 Bbl each will be prepared at ECLP and shipped by truck to offsite rental tankage in the Houston/Baytown area. Facilities are provided to off-take material from either current production or ECLP product storage. Samples will be prepared by pumping specified quantities of material from selected day tanks or product storage tanks into the sample storage tanks where they will be mixed. The blended sample will then be transferred to a sponsor provided tank truck for movement to the sponsor's leased tankage.

Two sample storage tanks, each with a 2000 Bbl working capacity will be provided. One tank will be for naphtha and the second tank will be

heated and store fuel oil. Both tanks will be provided with level indicators with the output tied into the data logger. Mixing of the tank contents and provision for sampling will be included in the design.

Although solvent and VGO will be blended inline to facilitate movement and cooling of the VGO, batches of sample (2000 Bbl or less) will be made up from predetermined quantities of material that are stored in either ECLP product day tanks or storage tanks. Existing pumps have adequate capacity but need to be checked to determine if they can deliver the required head. A new cooler in the fuel oil rundown line will be required to cool the combined solvent/VGO stream to 160°F. Flow meters with totalizers will be provided with output from these flow meters transmitted to the Data Logger.

● Prediction of Contaminants in Sour Water

As part of the work in preparing information for the Environmental Assessment of the 250 ton/day Exxon Coal Liquefaction Pilot Plant (ECLP), a study to predict the composition of the phenolic sour water from the various sections of ECLP was completed. Operation of the 1 ton/day Coal Liquefaction Pilot Plant (CLPP) produced sour water with high concentrations of phenolics and other oxygenated compounds which could cause problems in wastewater treating facilities. Significant differences in the way the sour water streams are generated and handled in CLPP and ECLP made it clear that the CLPP data were not directly applicable to ECLP. The major objective of this study was to develop our best estimates of the concentrations of organic and inorganic contaminants in the sour water from ECLP for the Environmental Assessment. For this purpose, detailed composition data was developed for all of the sour water streams from ECLP by modifying available CLPP yield data through use of computer modeling of the CLPP and ECLP processing steps.

A second objective of this study was to evaluate the capability of the refinery sour water strippers to meet the expected requirements for stripped water quality while processing ECLP sour water in addition to their normal sour water feeds. The results of the evaluation showed that the refinery sour water strippers could handle the ECLP sour water in addition to their normal feeds with only a minor modification. The evaluation also provided the refinery with an estimate of the maximum amount of organic material that their wastewater treating facilities will need to handle. More detailed information on this study can be found in the July-September, 1977 Quarterly Report (FE-2893-3).

Liquefaction Reactor Solids Withdrawal

A design basis was prepared for the ECLP reactor solids withdrawal facilities. Two systems are provided which will provide enough data on the amount and type of solids withdrawn during ECLP operations to support the design of a solids withdrawal and handling system for a commercial plant.

The first of the two systems is based on Engineering Technology cold model studies (see Section 4), while the second system is similar to that used in the CLPP pilot plant. A description of the systems is given in the July-September, 1977 Quarterly Technical Progress Report (FE-2893-3).

The basic design of the ECLP Solids Withdrawal System has been completed and detailed engineering is proceeding along with engineering of the total Liquefaction Section.

Data Logger

In order to fully utilize ECLP data for scaling up the EDS process to commercial size, the list of data that will be automatically recorded by the ECLP data logger was updated. The data logger will provide automatic scanning, alarming, storing, displaying, reporting and reduction analysis of analog and digital data.

In addition to maintaining records of flow, temperature, pressure and level measurements and stream analyses, the data logger will record and correct up to 50 measured flow variables for stream properties and operating conditions. It will also monitor up to 128 plant status information digital inputs (open or closed circuits). Listings of the most critical flow rates to be corrected and plant status circuits to be monitored were forwarded to Carter for their approval and forwarding to McKee.

The flow rates to be corrected are for streams that enter into ECLP onsite material balances or are involved in custody transfer such as natural gas, steam, nitrogen, hydrogen and water which are purchased, and fuel gas, acid gas, and sour water which are transferred to the Baytown Refinery. Temperature and $S_p G_{60}$ corrections are made for liquid streams and temperature, pressure and molecular weight corrections are made for vapor streams.

The plant status information being monitored includes emergency systems (such as automatic cut-ins/cut-outs, automatic shutdown mechanisms and emergency block valves) and motors on rotating equipment in critical service. The monitoring of emergency devices will provide information for analyzing plant upsets and a record of the process' problem areas. The monitoring of motors on critical equipment will provide a record of operating time for use in equipment corrosion, and erosion and service life studies. Equipment items included in this category are slurry pumps, sour water pumps and compressors.

• Flushing and Blowdown System

A design basis specifying the requirements of a flushing and blowdown system for ECLP was prepared. The flushing system includes both

high-pressure and low-pressure headers for distributing solvent to key points in the liquefaction and product fractionation facilities. The blowdown system includes a high-pressure emergency blowdown header and a low-pressure closed drain header for collecting material flushed from equipment in the liquefaction and Solvent Hydro process blocks. Both headers discharge into an agitated collection drum, which is equipped with a pump and cooler for removal of material to the wet slop storage tanks.

2.21 Processing High Calcium Content Coals

Controlled Scaling Approach to CaCO_3 Deposition

As described in past reports, a CaCO_3 -rich deposit forms on the liquefaction reactor walls when low-rank, high-calcium coals are liquified in the EDS process. Past work has centered on defining the problem and proposing methods of preventing deposition. These methods included chemical pretreatment of feed coal, use of additives in the liquefaction reactor feed, and mechanical solutions.

This study centers on defining the reactor modifications required in order to operate with scale build-up during operations and chemically cleaning during plant shutdowns. This controlled scaling approach is based on a one-half inch per year scale deposition rate which is estimated from data obtained in RCLU and CLPP runs with Wyoming coal.

The main features of this approach are:

- Use of Y-type, in-line strainers upstream of the liquefaction reactor high-pressure letdown valves.
- High-pressure solids withdrawal from the liquefaction reactor distributors and in-line strainers.
- Chemical cleaning of the reactors with acid (i.e., phosphoric acid or sulfamic acid) during scheduled plant shutdowns. (See Table 2-7).

The major concerns in scale control are:

- Scale formation on the distributors which could cause plugging or maldistribution in the reactors.
- Large chunks of scale falling on the distributors.
- Plugging of downstream valves by large scale particles.

In this study, the cost and feasibility of the Controlled Scaling approach will be assessed. Equipment modifications and new facilities for chemical cleaning will be specified and a plan-of-attack for dealing with CaCO_3 deposition in both ECLP and a commercial plant will be formulated. The study will continue through October, 1978.

TABLE 2-7

CONCEPTUAL CHEMICAL CLEANING PROCEDURE

- Shutdown reactors.
- Flush with solvent.
- Isolate reactor and reactor separator.
- Fill with 180°F, 10% H_3PO_4 @ 500-1000 gpm by mixing (steam + water) + conc. H_3PO_4 .
- Slowly circulate and soak (~1 day) adding conc. H_3PO_4 and steam as needed.
- Bubble N_2 , air or steam thru reactors to mix up undissolved solids.
- Drain through solids withdrawal system.
- Rinse and neutralize as necessary.
- Make sure all solution is out of all lines to protect surrounding materials.
- Dispose of gases that are evolved during entire process.
- Dispose of spent solutions.

2.22 Solids Waste Disposal

The objective of the solids waste disposal study is to evaluate disposal techniques for the waste streams both from coal preparation plants (coarse and fine refuse) and from a commercial EDS plant (venturi fines from a FLEXICOKING unit and tertiary cyclone fines). As a study alternative, the inclusion of a partial oxidation unit in the EDS plant was investigated. In addition to the wastes from a FLEXICOKING unit, the partial oxidation unit would generate a slag as a slurried fine waste stream. Waste stream characteristics used in the study for the cases with and without the partial oxidation unit are described in Table 2-8.

Coal Wastes

Coal preparation plant wastes currently are disposed of by one of several methods:

- Pumping fluid refuse to a permanent impoundment.
- Depositing slurry in temporary dewatering ponds followed by excavation and burial in a landfill.
- Mechanical dewatering of the slurry followed by disposal of the refuse cake into a landfill.

The simplest disposal method is to construct an impoundment and pump the fines slurry behind it. The EPA Interim Final Rules for coal preparation plants state that there shall be "no discharge of process water" from these facilities. Therefore, with some modifications, the impoundment method may be an acceptable disposal technique for the near future.

Process Wastes

Process wastes from coal liquids (FLEXICOKING and partial oxidation units) are not produced at any commercial site in the U.S.A. Consequently, there is little or no information concerning the disposal of these type wastes. Future environmental regulations governing the discharge of wastes containing heavy metals and other chemicals will have an impact on disposal of process wastes. It is uncertain what potential environmental risks may be associated with the process wastes. Therefore, it is assumed process wastes should be segregated from coal preparation wastes, even if the EDS plant is located next to the mine. Should the process wastes be classified as hazardous, they will, as a minimum, require a secure landfill or impoundment site. Such a site would include an impervious layer or liner, and run-off or leachate collection and treatment of these collected waters.

TABLE 2-8

SOLID WASTE CHARACTERISTICS

		<u>% Ash</u> <u>% Ash</u> <u>(Dry)</u>	<u>Dry Solids</u> <u>(ST/D)</u>	<u>Water</u> <u>(ST/D)</u>
	Coal Coarse Refuse*	70	3190	98.6
	Coal Fine Refuse*	55	942	229.1
	Coal Fine Refuse*	13	1305	435.0
Case A	{ Dry Fines from FLEXICOKING Unit	67	1344	0.0
	{ Wet Fines from FLEXICOKING Unit	67	1344	4560.0
Case B	{ Partial Oxidation Slag	94	584	31.0
	{ Partial Oxidation Fines	32	118	177.0
	{ Dry Fines from FLEXICOKING Unit	67	862	0.0
	{ Wet Fines from FLEXICOKING Unit	67	862	2925.0

*Coal waste streams exist in both Case A & Case B.

Alternative Disposal Techniques

Some work has been conducted in areas of alternative disposal techniques for coal wastes. One such alternative technique is underground disposal of coal wastes. Based on European experience, this technique is technically feasible using current technology, but its economic feasibility is site specific. Underground disposal is more expensive than current surface methods of disposal. Compliance with anticipated environmental regulations will most likely require some modification to current underground disposal methods.

Potential uses for coal wastes that have been investigated to some extent are the following:

- Lightweight aggregate
- Road base and anti-skid material
- Bricks
- Mineral wool

These uses are economically questionable, and would not appear to consume significant quantities of material.

2.23 Summary of Results of Phases I and II

Prior to the start of the jointly funded EDS Process Development Program (Phases IIIA-V) in January 1976, Exxon Research & Engineering Company (ER&E) carried out privately funded research over the 10-year period 1966 thru 1975 (Phases I and II) which led to the selection of the EDS Process as the preferred route to produce liquid fuels from coal. One of the work items during the past year was the preparation of a summary description of the results of the Phases I and II work. This was issued as an interim report (FE-2893-16).

ENGINEERING RESEARCH AND DEVELOPMENT

Process Engineering and Cost Evaluations

3. Engineering Studies of Bottoms Processing/Hydrogen Manufacturing

3.1 Evaluation of Alternative Process for Hydrogen Manufacture

An evaluation of four methods of producing hydrogen for the EDS Liquefaction Plant showed that, depending on the value of high Btu gas (HBG), the hydrogen should be made either by steam reforming of HBG or by partial oxidation of vacuum pipestill (VPS) bottoms. The four hydrogen manufacturing methods considered were: 1) steam reforming of HBG, 2) partial oxidation of VPS bottoms, 3) oxygen-based processing of VPS bottoms (supplemental coal feed optional) in a FLEXICOKING unit and 4) oxygen based gasification of coal in a moving bed gasifier.

These four processes differ chiefly in the feedstock used and the method of conversion of synthesis gas. They all include similar processing to upgrade the synthesis gas to hydrogen. The steam reformer and the moving bed gasifier (MBG) are commercially demonstrated processes, whereas the other two processes will require some further development.

Table 3-1 summarizes the results. The lowest cost hydrogen producer without the supplemental coal option is partial oxidation of VPS bottoms. Hydrogen from the steam reformer costs 0.58 \$/kSCF more due to greater feed and fuel costs, whereas hydrogen from the oxygen-based FLEXICOKING unit costs 1.31 \$/kSCF more due to greater feed and utilities costs which are not totally offset by the byproduct credits.

The HBG value is the major variable which significantly affects results. This evaluation assumes a high BTU gas value in parity with liquids on a Btu basis. This value could be lower as a result of costs to bring the gas to a pipeline, or if it is discounted as an interruptable supply, or finally, if the sales price is controlled at some low level. A reduction of the price of HBG by one-third or more would make the steam reformer alternative the most attractive.

With the supplemental coal option, which effectively results in lower cost fuel and steam, the oxygen-based FLEXICOKING alternative produces hydrogen at about the same cost as partial oxidation. Partial oxidation and steam reforming maintain their relative positions, although a reduction of HBG price to two-thirds of the base case gas price will again make the steam reformer option more attractive than partial oxidation. The moving bed gasifier is not as attractive due principally to high steam costs.

TABLE 3-1

EDS PLANT HYDROGEN MANUFACTURING ALTERNATIVES
COMPONENTS OF HYDROGEN COST

(\$/kSCF H₂ Contained - 1985\$)

Unit ⁽¹⁾	O ₂			O ₂ FLEX			
	<u>S/R</u>	<u>POX</u>	<u>Btms.</u>	<u>S/R</u>	<u>POX</u>	<u>Btms./Coal</u>	<u>MBG</u>
	← No Supp. Coal →			← Supp. Coal →			
Feed							
• HBG ⁽²⁾	1.78	(0.10)	(0.55)	1.79	(0.10)	(0.84)	(1.17)
• Btms	-	1.46	2.99	-	0.95	1.00	-
• Coal	-	-	-	-	-	0.49	0.90
Fuel	1.43	-	-	0.77	-	-	-
Utilities & Misc.	(0.01)	1.14	3.24	0.13	1.07	2.50	2.91
Investment	1.13	1.25	1.26	1.13	1.25	1.29	1.29
By Prod. Liquid	-	-	(1.88)	-	-	(1.38)	(0.37)
Total H ₂ Cost	4.33	3.75	5.06	3.82	3.17	3.06	3.56

(1) Abbreviations:

S/R - Steam Reforming of high Btu gas.

POX - Partial Oxidation of vacuum bottoms.

O₂ FXC - Oxygen Based processing of vacuum bottoms and coal in a FLEXICOKING unit.

MBG - Moving Bed Gasification of Coal.

HBG - High Btu gas.

(2) HBG sold at base case gas price of 7.70 \$/MBtu. (Parity with low sulfur fuel oil).

Although the use of supplemental coal can improve all the cases, the change is greatest for the O₂ FLEXICOKING unit because the largest contributors to its H₂ cost (i.e., feed and steam) are directly reduced by the supplemental coal.

A more detailed discussion of the study basis and results may be found in the July-September, 1977 Quarterly Technical Progress Report (FE-2893-3).

3.2 Wyoming Coal Bottoms Processing Vectors

Two new Wyoming coal/bottoms processing vectors for the EDS Process Alternative LP, Model (PAM) have been developed. The process depictions are in addition to those vectors previously reported in the March, 1978 Quarterly Technical Progress Report (FE-2893-12). The first vector represents partial oxidation of vacuum bottoms for fuel production, and the second vector models moving-bed gasification of coal to produce hydrogen. The new vectors, along with those previously developed, are being used to screen bottoms processing alternatives for the Wyoming Coal Study Design Update.

● Partial Oxidation for Fuel Production (Table 3-2)

Vacuum bottoms, oxygen, and steam react at 300 psig in a non-catalytic partial oxidation reactor to form syngas. Heat and material balances were developed using assumptions similar to the partial oxidation for hydrogen case developed previously.

Hot syngas is cooled below the softening point of the ash to prevent ash deposits in the downstream waste heat boilers. This quenching is achieved by recycling a portion of the cooled product gas stream. Partially quenched syngas is further cooled in a waste heat boiler, generating 600 psi process steam. Finally, syngas is expanded to 50 psig and H₂S is removed.

Synthesis gas generation investments are based on information from a process vendor. Remaining plant investments were based on proprietary Exxon investment curves. A 25% process development allowance (PDA) was added to the syngas generation section, and a 15% PDA was added to the remainder of the unit to reflect process uncertainties. This combination results in an overall PDA of 21%.

● Moving-Bed Gasification of Coal for Hydrogen (Table 3-3)

Moving-bed gasification is representative of a commercially demonstrated coal gasification process. When oxygen and steam are used in the gasifier, an intermediate Btu gas (IBG) is produced which can be upgraded to hydrogen. The IBG upgrading facilities are also included in the process vector.

TABLE 3 -2

EDS PROCESS ALTERNATIVES MODEL
PARTIAL OXIDATION OF VACUUM BOTTOMS FOR FUEL

<u>Yields</u>		<u>Partial Oxidation</u>	
		<u>Bottoms</u>	<u>Ash</u>
		(t/t Ash Free Bottoms) ⁽¹⁾	(t/t Ash)
950°F+ Bottoms From Liquefaction		(1.0000)	(0.0750)
Intermediate BTU Gas		2.3302	
Product Sulfur		0.0039	
Sour Water		0.2471	
Residue Char			1.0750
Ash			(1.0000)
Oxygen		(0.9776)	
<u>Qualities</u>			
Intermediate BTU Gas, HHV, (Btu/lb)		5312	
<u>Utilities</u>			
Electric Power, kWh/t		56.01	
Turbine Power, kWh/t		90.34	
600 psi Process Steam Produced, t/t		1.8408	
150 psi Process Steam Consumed, t/t		0.0265	
Boiler Feed Water, t/t		2.3327	
Cooling Water, t/t		10.6430	
<u>Investments⁽²⁾</u>			
Train Size, t/SD		920.5	
Capacity Factors		80%	
Materials: Onsite, k\$/train		3266	
Labor: Onsite k\$/train		516	
Plant Size Exponent		0.70	
Process Development Allowance		21%	
<u>Operating Costs</u>			
Salaries, Wages and Related Costs		2.0% TEC	
Repair Material, Etc.		3.3% TEC	
Catalyst and Chemicals		0.0145 \$/t	

Notes:

(1) t = One metric ton = 2205 lbs.

(2) Investments are for 57-59 at standard location.

(3) Catalyst and chemicals cost are in 1985 \$.

TABLE 3-3

EDS PROCESS ALTERNATIVE MODEL
MOVING BED OXYGEN GASIFICATION PROCESS FOR HYDROGEN

Yields	Moving Bed Gasification	
	Coal (t/t Ash Free Coal) ⁽¹⁾	Ash (t/t Ash)
Coal	(1.0000)	(0.0928)
Ash		(1.0000)
Residue Char		1.0928
Oxygen	(0.2461)	
Naphtha	0.0103	
Tar and Tar Oils	0.0618	
Product Sulfur	0.0066	
Ammonia	0.0070	
Sour Water	1.0726	
95 Mole% H ₂	0.0406	
60 Mole% H ₂	0.0171	
C ₁ /C ₂	0.1003	
Qualities		
Naphtha (C ₅ /400°F)		
Sulfur, wt%	0.02	
HHV, BTU/lb	19.000	
S.G.	0.77	
Tar and Tar Oils		
Sulfur, wt%	0.23	
AHV, BTU/lb	16,500	
LHV, BTU/lb	16,000	
S.G.	1.01	
Solids, wt% ⁽²⁾	6.0	
Utilities		
Electric Power, kWh/t	30.20	
Turbine Power, kWh/t	98.49	
600 psi Process Steam Consumed, t/t	1.2840	
150 psi Process Steam Consumed, t/t	0.3381	
Cooling Water, t/t	49.04	
Boiler Feed Water, t/t	0.7877	
Process Water, t/t	6.5313	

Continued.....

TABLE 3-3 (Cont'd)

	<u>Coal</u>	<u>Ash</u>
<u>Investments⁽³⁾</u>		
Train Size, t/SD	5,655	
Capacity Factor	80%	
Materials: Onsite, k\$/train	24,001	
Labor: Onsite, k\$/train	3,237	
Plant Size Exponent	0.70	
Maximum Plant Size	6786	
Process Development Allowance	10%	
<u>Operating Costs</u>		
Salaries, Wages, and Related Costs	2.0% TEC	
Repair Materials, Etc. ⁽⁴⁾	3.3% TEC	
Catalyst and Chemicals	0.0518 \$/t	

Notes:

- (1) t = One metric ton = 2205 lbs.
- (2) Investments are for 57-59 at standard location.
- (3) Catalyst and chemicals cost are in 1985 \$.

The moving-bed gasification step is based on information published for the WESCO Gasification Project⁽¹⁾. Because of the relatively high methane content of IBG produced in the moving-bed process, hydrogen purity attainable by water-gas shift and CO₂ removal is about only 82 mole %. This is considerably lower than the 95 mole % H₂ purity which forms the basis for hydrogen consuming process vectors in PAM (i.e., liquefaction and solvent hydrogenation). However, flexibility is available in the PAM to modify the cryogenic H₂ recovery system and the purge gas/treat gas loop to maintain adequate hydrogen purities within the hydrogen consuming blocks.

● Future Work

Future work will be directed at the investigation of optimum bottoms configuration for liquefaction of Wyoming coal.

3.3 Evaluation of Moving-Bed Coal Gasification

An evaluation of moving-bed coal gasification (MBG) as a means of providing supplemental coal to an EDS plant was reported in the Phase IIIA Final Report, (FE-2352-20). Alternative products from MBG are low Btu gas (LBG) from an air-blown process or intermediate Btu gas (IBG), which can be upgraded to H₂, from an O₂-blown process. It was found that MBG for fuel gas production could be attractive providing the EDS plant was operating in a fashion which produced a large fuel imbalance.

The evaluation of MBG was continued in Phase IIIB (July-September, 1977 Quarterly Progress Report; FE-2893-3). Sensitivity cases using the EDS Process Alternatives LP Model (PAM) examined the effect on LBG value as a result of variations in liquid yield, liquid value, and steam requirements for MBG. It was determined that possible variations in these items contributed less than 10% uncertainty in LBG value between the most conservative and most optimistic representation of the MBG process. Less than a 10% uncertainty on the cost of H₂ generated by upgrading IBG from MBG was also determined due to possible variations in MBG steam and O₂ consumption.

3.4 Vacuum Tower Operation for Partial Oxidation Feed

Results from a study to determine the maximum feasible vacuum pipe-still (VPS) cut point were reported in the October-December, 1977 Quarterly

(1) WESCO FPC Application, Docket No. CP 73-211, November 16, 1973.

Technical Progress Report (FE-2893-7) and the January-March, 1978 Quarterly Technical Progress Report (FE-2893-12). The VPS cut point is important for the Study Design Update alternative designs which employ partial oxidation of vacuum bottoms for hydrogen production and/or fuel production. Screening work had shown an incentive to maximize liquid recovery by cutting as deeply as possible in the VPS upstream of partial oxidation.

The present study determined that the vacuum tower cut point should be limited to about 975°F. The major limitation to cutting deeper was found to be a corresponding increase in viscosity of the bottoms and its effect on pump performance. A 975°F cut point produces a bottoms stream whose viscosity falls within demonstrated pumping capability of 25-50 poise for plunger pumps.

3.5 Selection of Hydrogen Purity/Composition for Illinois Study Design Update

A study which determined the optimum hydrogen purity for the Study Design Update was discussed in the October-December, 1977 Quarterly Progress Report (FE-2893-7). The optimum purity for H₂ produced by steam reforming was found to be 93 to 94 mole % in comparison with a 95 mole % purity used in the 1976 EDS Commercial Plant Study Design. For H₂ produced by partial oxidation, the optimum purity continued to be about 98 mole %.

It was determined that H₂ production costs could be reduced by eliminating the low temperature shift and/or the methanation step in the upgrading section of the H₂ plant. The changes in upgrading lead to the production of a H₂ stream which contains about 2 mole % carbon oxides in place of methane. An incentive in the range of 0.25 to 0.30 \$/B of liquid product was estimated for making processing changes in steam reforming and 0.10 to 0.15 \$/B for making changes in partial oxidation.

3.6 EDS FLEXICOKING: Fines Make Prediction

The gasification level which can be obtained for an EDS FLEXICOKING Unit is influenced by the following:

- + the ash content and rate of fines loss through heater cyclones
- + the ash content and physical properties of the circulating coke.

An initial effort has been completed to predict the coke fines make for an EDS commercial FLEXICOKING Unit. The potential impact of these predictions has also been examined. The approach taken in this study was to:

- + Review petroleum FLEXICOKING Unit data on fines make for bench scale gasification (GFR), pilot plant FLEXICOKING (IKG) and FLEXICOKING in the 750 B/D prototype unit.

- + Express the fines (44 micron or smaller) as a percentage of coke gasified.
- + Examine the variability of fines make with critical operating conditions such as:
 - gasification level
 - gasifier temperature
 - gas contact time
 - air/steam ratio

The information taken from these three steps will be compared to current data from gasification (FBU) and the IKG coal liquid bottoms FLEXICOKING tests. Comparison of petroleum coke data to the initial EDS coke data provides us with a preliminary basis for estimating the fines make for a commercial EDS FLEXICOKING Unit. The available data are summarized in the following table:

<u>Feed</u>	<u>Unit</u>	<u>Fines Make as % of Coke Gasified</u>	
		<u>44 μ or smaller</u>	<u>Total</u>
Petro. Coke	GFR - pilot gasification	0.2-3.0	0.3-5.0
Petro. Resid.	IKG - pilot FLEXICOKING Unit	0.8-2.8	2.0-7.0
Petro. Resid.	Prototype FLEXICOKING Unit	1.6-4.0	2.0-5.0
EDS Coke	FBU - pilot gasification	10.0-11.0	----

These data show that the coke fines make for petroleum units all lie within the same range despite the different fines generation mechanisms inherent to each unit. The implications of these data on fines make for FLEXICOKING of coal liquefaction bottoms is shown below at 83% gasification, for a fines make bracketing the FBU data and for a likely range of fines ash composition from 70 to 100%.

● Gasification at 83% (Design Study Basis)

<u>Fines Make, % of Coke Gasified</u>	<u>Fines Composition, % Ash</u>	<u>Circulating Coke Composition, % Ash (Bed Purge)</u>	<u>Ash Purged w/Fines, % of Feed Ash</u>
7	70 to 100	65 to 62	13-21
17	70 to 100	64 to 53	30-43

Only 13% of the ash will be rejected in the fines at a fines make of 7% with an ash concentration of 70%. Even at 17% fines make and at an ash composition of 100%, only 43% of the ash is purged from the system with fines. This lowers the bed purge (and circulating coke) coke ash concentration to 53%. Based on the FBU and IKG data to date, it appears that for FLEXICOKING of EDS bottoms, a fines make of about 10% will result in an ash concentration of about 53 to 65% in the circulating coke.

An important conclusion from this study is that the currently available data do not indicate any material balance limitation to gasification levels up to 83%. The relative high estimated ash content of the circulating coke and the properties of the resultant coke could be important, however, and need further study as more data become available.

3.7 Review of Slagging in FLEXICOKING Gasifiers

A review was made of available information on ash slagging in coke gasification. This effort was aimed at providing background to assist the determination of the effect of slagging on the operating range of a FLEXICOKING unit running on Illinois coal liquefaction bottoms. The material reviewed covered two general areas of development: proprietary petroleum coke gasification and Illinois coal gasification. Three gasifier variables appear to be significant in slag formation: oxygen partial pressure, particle surface temperature, and the quality of particle fluidization. Based on this review, there would be potential for slag formation in areas where there was poor fluidization and/or high oxygen partial pressure.

3.8 Coke Gasification Kinetics in the Prototype

A study was completed comparing proprietary kinetic model predictions to Baytown previous prototype FLEXICOKING results. The model, which is based on proprietary petroleum data from other pilot plants, is one of the tools being used to determine the process basis for prototype operation on ECLP vacuum bottoms.

For this study, two prototype run periods were simulated. Simulations using these data were run with the results screened to see how well they matched the original data. The results indicate that the kinetic model does a good job of predicting gasifier holdup. Sensitivity cases using different particle properties were run to determine the effects of variations on model predictions.

3.9 FLEXICOKING Design Studies

During the period ending June 30, 1978, the EDS FLEXICOKING Prototype Cost/Benefit study was completed. This study led to the technical recommendation that the Baytown Prototype FLEXICOKING Unit (BPFU) be revamped and run with vacuum bottoms feed from ECLP. The Class V expected project cost for an 18-month prototype operating period was estimated at about 29 M\$. This revamp case was the most attractive of three alternates. Relocating the BPFU to the ECLP site or constructing a new grass roots unit at the ECLP site cost about 5 M\$ and 7 M\$ more than the base case, respectively. These were eliminated from further consideration.

The technical recommendation to include the operation of the revamped BPFU to process ECLP vacuum pipestill bottoms in the EDS development program was presented to the Overall EDS Advisory Subcommittee on June 21 and to the Sponsors Technical Committee (STC) on June 27/28. Tables 3-4, 3-5, and 3-6 attached, cover respectively: "Investment Summary - FLEXICOKING Prototype Revamp," "Prototype FLEXICOKING Unit - Costs for 18 Month Operating Period" and "EDS FLEXICOKING Prototype Revamp - Project Schedule." Based on acceptance by the STC and pending final approval by the Sponsors Management Committee, planning work is underway to resolve the issues required to develop a FLEXICOKING prototype design basis.

Based on work performed during Phase IIIA, a cost/benefit analysis was initiated to reach a decision on reactivating the prototype. The design study work associated with the cost/benefit analysis consisted of three parts. First, an "updated" study design was developed for a "base case" commercial 21.2 kB/SD once-through EDS FLEXICOKING unit for Illinois coal. Second, alternative cases were developed for the commercial plant which reflected process and equipment uncertainties. Third, a screening study design was completed for revamping the Baytown prototype FLEXICOKING unit for Illinois coal bottoms. This included specifications for reconditioning the unit, for altering some onsite equipment, and for new onsite and offsite equipment. This prototype study design served as the basis for an equipment cost estimate, which in turn was used in developing the economic and technical incentive for the Baytown FLEXICOKING operation. The details of the economic analysis used in the cost/benefit study are described in the January-March 1978 Quarterly Technical Progress Report (FE-2893-12). Highlights of this analysis are covered later in this report.

The nature of the process basis used in the commercial plant base case was discussed in the July-September, 1977 Quarterly Technical Progress Report (FE-2893-3). The underlying philosophy was to study most carefully those areas for which a prototype operation would be most helpful, for example, generation of coke dust, gasifier entrainment, and gasification kinetics. During the development of the base case equipment definition, additional technical uncertainties were uncovered. Reactor development led to a change in reactor operating conditions, as reported in the July-August, 1977 Monthly (FE-2893-1) and the October-December, 1977 Quarterly Technical Progress Report (FE-2893-7). Gasifier development led to the

TABLE 3-4

INVESTMENT SUMMARY
FLEXICOKING PROTOTYPE REVAMP

● Class V Investment Summary*, k\$	
+ Direct costs (material, labor, subcontracts)	6,900
+ Field labor overheads	2,370
+ Contractor engineering	2,900
+ ER&E charges	1,800
+ Contingencies	
- Project (25%)	4,100
- Revamp (5%)	800
- PDA (10% of prime contract)	1,400
+ Other (burden, fees, owner's charges, allowances, etc.)	2,630
Total	22,900
Call	23,000
● Major Cost Items, k\$ (TEC costs do not include pro rata share of ER&E charges, contingencies, etc.)	
+ Reconditioning (piping, major vessel reconditioning)	2,200
+ New Onsites	
- Vessels	700
- Pumps	800
- Feed System	400
- Equipment for rented LBG compressor	1,000
+ Modified Onsites (instrument reconditioning, etc.)	1,700
+ Offsites	
- Tanks, mixers, pumps, vessels	900
- Dowtherm unit	600
- Truck loading facilities	400
- Interconnecting lines	900
- Sour water treating facilities	700
- Sandvik Belt	900

*The Class V investment has a 50% chance of underrun/overrun of more than 10%.

TABLE 3-5

PROTOTYPE FLEXICOKING UNIT
COSTS FOR 18 MONTH OPERATING PERIOD

	<u>M\$</u>
● BAYTOWN REFINERY SERVICES	
+ Salary, wages, benefits	
+ Routine & TA maintenance	
+ Prorata common facilities	
+ Lab services, etc.	3.2
● UTILITIES (Net after fuel credit)	<u>0.7</u>
● TOTAL BAYTOWN SERVICES	3.9
● OTHER SERVICES	
+ Solids disposal	
+ Compressor rental	
+ Cab and Driver	0.4
● INCREMENTAL ALLOWANCE FOR MAJOR FIXUP	<u>0.4</u>
Subtotal	4.7
● ERE SITE CHARGES	<u>1.3</u>
	<u>6.0</u>
TOTAL	6.0

EDS FLEXICOKING PROTOTYPE REVAMP
PROJECT SCHEDULE

EXXON RESEARCH & ENGINEERING CO.
EXXON ENGINEERING - P.O. BOX 101 - FLORHAM PARK, N.J. 07932
PROJECT SCHEDULE
BAYTOWN FLEXICORING PROTOTYPE REVAMP

identification of special material and/or construction techniques for very large gasifier vessels. (See October, 1977 Monthly (FE-2893-4) and July-September, 1977 Quarterly Technical Progress Report (FE-2893-3). Heater grid development led to the identification of a critical temperature control problem which in turn led to the development of a lower heater temperature case.

Once the equipment specifications for the commercial base case were set, work began on defining the impact of uncertainties on the commercial unit base case. A larger diameter gasifier was studied to develop the impact of gasifier entrainment uncertainty. A shallow gasifier bed was studied in order to reflect gasification kinetics uncertainties. Changes in heater overhead equipment reflected coke fines generation uncertainties. Finally, the equipment and process impact of lower heater temperature was investigated. Base case and alternate investments are reported in Table 3-7.

The Prototype screening design was started in January, 1978 with the development of a process basis. This work identified those changes and/or additions that would be required to meet the process objectives, and investigated critical technical issues of gasifier slagging and particle integrity. Detailed discussions of the results of the prototype screening study are covered in both Monthly and Quarterly Technical Progress Report summaries from January through June, 1978. Certain aspects of the EDS FLEXICOKING process require modification of the Baytown Prototype FLEXICOKING unit. For example, incompatibility of FLEXICOKING liquid products and petroleum-based liquids dictate special consideration for feed product storage and disposition. The highly viscous, high-solids feed and scrubber-bottoms streams require special attention for the flushing and pump seal oil system, as well as consideration of a new reactor feed system. The problem of ECLP vacuum bottoms thermal instability presents another special problem for feed tankage/storage. Preliminary results of equipment screening for the prototype are reported in the January-March, 1978 Quarterly Technical Progress Report (FE-2893-12).

Since the completion of the design study work for the prototype cost/benefit study, work has been continuing on the key technical issues of storing, pumping, and feeding ECLP vacuum bottoms to the FLEXICOKING reactor. Two options currently under study include solidification and remelting of the bottoms, or hot tank storage of the bottoms. The hot tank approach requires dilution of the bottoms with lighter material since undiluted bottoms polymerize to such an extent that the liquid becomes difficult to pump. Studies are now underway to determine the dilution requirements and storage time possible while maintaining the bottoms as a pumpable liquid.

Pumping the highly viscous, high solids feed stream requires pump seal and pump flushing oil in relatively large quantities (as a percentage of feed). Earlier work tentatively identified coker fractionation as a potential source of recycle 700-1000°F material which could be used for flushing/seal-oil and for feed dilution. Work has been continuing in an attempt to identify alternative sources of material suitable for this

TABLE 3-7

SCREENING QUALITY COST ESTIMATES FOR
COMMERCIAL BASE CASE FLEXICOKING AND ALTERNATES

	<u>Prorata Investment (3 Trains)</u>
	(1980 \$)
Base Case ⁽¹⁾	560 M\$
Alt. 1 (increase diameter of gasifier)	28 M\$
Alt. 2 (decrease bed height of gasifier)	-2 M\$
Alt. 3 (higher fines production)	+4 M\$
Alt. 4 (lower heater temperature)	+17 M\$

(1) The Base Case Costs include the following facilities:

Reactor/Scrubber
Heater
Transfer Lines
Gasifier
Air Blower
Heater Overhead System
Coke Storage
Coke Gas Condenser
Fractionator
Stretford

service in an attempt to eliminate the need for a new fractionator. This pump-seal/flushing oil requirement together with bottoms dilution may result in a significant change in coker yield and scrubber performance. Coking yield studies are planned to investigate the effects of high feed dilution.

Finally, meetings with ER&E Petroleum Department design personnel have been initiated to ensure that all the issues required for the plant design have been identified and are being resolved.

3.10 FLEXICOKING Prototype Unit Cost/Benefit Process Studies

A major part of the Phase IIIB and Phase IV EDS development program relating to engineering studies of bottoms processing alternatives and hydrogen manufacture was the cost/benefit analysis work leading to a recommendation regarding operation of the Baytown prototype FLEXICOKING unit. The purpose of this study was to determine whether it was beneficial for the program to operate the prototype on EDS vacuum bottoms. The planned activities for this analysis were discussed in the July-September Quarterly Technical Progress Report (FE-2893-3). Briefly, these activities entailed developing the basis for operating a commercial plant and the prototype on Illinois coal EDS vacuum bottoms. The commercial plant basis would have to cover a large range of operating conditions due to the uncertainties in the commercial process variables. The additional costs for accommodating these uncertainties in the first commercial plant design were then compared with the cost for operating the prototype unit to see if any benefits could be realized. The following summary describes the process work that went into this analysis.

The development of the commercial FLEXICOKING process basis has been previously described (September Monthly (FE-2893-2) and July-September Quarterly (FE-2893-3). Adjustments in the process variables were made using the best laboratory data available or past petroleum experience where data for certain variables were lacking. Temperatures, pressures, space velocities, coke properties, system heat load, yields and gasification levels were considered. Other items which affected basis development were kinetics and slugging.

In November, process screening was initiated to evaluate prototype operation on EDS vacuum bottoms. Screening cases were based on limitations of the existing unit, which ran from February, 1974 to July, 1975 on petroleum feedstock. The resulting process basis includes a lower feed rate than seen in previous prototype operation (90 vs 120 tons/day) in order to maintain the expected commercial gasification level of 80%. This study also included implementing modifications to the unit to accommodate the EDS bottoms feed and developing sensitivity cases to determine the effect of variations in temperatures and kinetics on the unit.

Evaluation of the benefits for operating the prototype was initiated in April. This evaluation was carried out by covering the key issues and developing scenarios for each issue identified for the first commercial

TABLE 3-8

SUMMARY OF BENEFITS FOR OPERATION OF
FLEXICOKING PROTOTYPE UNIT

Technical Issue	Alternative Scenarios For Prototype Experience	Prototype Benefit	Benefit M\$, 1980	x Probability	Total Benefit M\$, 1980	
● Attainable gasification level is uncertain	A. Confirms initial design attains 80% gasification.	No benefit	0	0.3	0	
	B. Without Prototype initial operation would be limited to 70% gasification. Prototype allows 80% for initial operation.	Avoid burning product/coal for 5 years while operating at 70% gasification.	45	0.5	23	40
	C. Without Prototype initial operation would be limited to 50% gasification. Prototype allows increase to 80% for initial operation.	Avoid burning product/coal for 5 years while operating at 50% gasification.	135	0.1	14	
	D. Prototype shows 90% achievable.	Increase gasifier cost but reduce burning product/coal because of 90% gasification.	25	0.1	3	
● Scrubber Bottoms fines content uncertain	A. Prototype confirms once-thru operation with fines cleanup is satisfactory.	Eliminate recycle flexibility for design and reduce fines removal investment	25	0.25	6	
	B. Prototype confirms once-thru. Fines cleanup shakedown eliminated for first two years of operation.	Eliminates recycle flexibility in design and saves 2 years downgrading of scrubber bottoms.	60	0.40	24	41
	C. Prototype confirms recycle required and demonstrates method of operating satisfactorily in recycle mode.	Eliminates once-thru flexibility and avoids two years where scrubber bottoms is used for landfill.	90	0.10	9	
	D. Prototype confirms recycle required and shows initial design is operable in recycle model.	Eliminates once-thru flexibility and fines removal in design.	6	0.25	2	
● FLEXICOKING configuration	A. Prototype experience indicates 3 units at 50% capacity are required.	No benefit	0	0.5	0	45
	B. Prototype experience allows reduction to 3 units at 37.5% capacity.	Reduce train size	90	0.5	45	
● On Stream Factor Uncertain	A. Prototype does not change on stream factor for EDS plant.	No Benefit	0	.15	0	
	B. Prototype experience leads to increased EDS plant production of 3 months during first 4 years.	Increase EDS Production	100	.40	40	140
	C. Prototype experience leads to increased EDS plant production of 6 months during first 4 years.	Increase EDS Production	200	.40	80	
	D. Prototype experience leads to increased EDS plant production of 12 months during first 4 years.	Increase EDS Production	400	.05	20	
● Other Issues						
+ Gasifier Entrainment						
+ Gasification Kinetics						
+ FLEXICOKING Unit Heat Load						
+ Fines/Purge Coke Split						
+ Coke Properties						
+ Gasifier Temperature						
+ Waste Water Treatment						
					Total	44
					-	310

design (first reported in the January-March 1978 Quarterly Technical Progress Report (FE-2893-12)). The scenarios for each issue reflect various operational conditions for the first commercial plant with their degree of probability as based on current data. For each scenario a monetary benefit was computed based on what information would be realized from prototype operation. The benefit from prototype operation as pertaining to a certain issue was then calculated by multiplying the probability of each scenario by its corresponding benefit and summing these products for all scenarios of the issue. A breakdown of the benefits for each technical issue, with scenario breakdown for some important issues, is shown in Table 3-8. As seen from this table, the total benefit accrued from prototype operation by resolving these technical issues is approximately 310 million dollars (1980\$). Besides monetary benefits, other benefits could be realized from the operation of the prototype, such as large scale product samples, demonstrating the capability of meeting environmental constraints, process improvement potential and operator experience.

Comparing monetary and other benefits from prototype operation with the estimated cost of approximately 30 million dollars (1980\$) to modify and operate the prototype led to the technical recommendation that prototype operation be included in the EDS program. The recommendation was reviewed and accepted by the Sponsors Technical Committee. Pending final sponsors management approval, future work will deal with developing the design for the modified prototype.

3.11 Coke Gasification Kinetics

An analysis of petroleum and EDS coke gasification kinetics led to the following conclusions:

- + Required coke holdup for an EDS FLEXICOKING unit gasifier will be equal to or slightly greater than that required for petroleum coke gasification at equivalent operating conditions. Carbon holdup is expected to be less for EDS gasification than for petroleum operation.
- + Analysis of proprietary IKG (integrated coker-gasifier pilot unit) results for petroleum coke leads to a direct scale-up that is slightly conservative compared to commercial prototype results.
- + Analysis of IKG kinetic results on EDS coke should provide the basis to allow adjustment to the kinetic model for scale-up to a commercial EDS unit.

3.12 IKG Operations Guidance

The FLEXICOKING process studies group was involved during the year in operations guidance for the Integrated Coker-Gasifier unit (IKG). Guidance cases were generated for Runs 20 and 21 when IKG was operated on Illinois

coal bottoms. As reported in the September Monthly Technical Progress Report (FE-2893-2), observed gasification rates were higher than would be predicted using a petroleum-based kinetic model. In February, more cases were developed based on a lower gasifier temperature to take advantage of the apparent enhanced kinetics and the potential for reducing slag formation tendencies.

ENGINEERING RESEARCH AND DEVELOPMENT

ENGINEERING TECHNOLOGY DEVELOPMENT

4. Liquefaction Engineering Technology

4.1 Coal Liquefaction VLE Data

Summary

The objectives of Phases III-B and IV were to obtain additional experimental vapor pressure and VLE information in order to better understand the vaporization of coal liquids at high temperatures under both high and low pressures. Specifically, part of the Phase III-B experimental work at Brigham Young University was to repeat some of the Phase III-A VLE measurements to better establish the experimental precision above 700°F. At 1950-2000 psia, the vaporization of the hydrogenated solvent is now known reliably up to 850°F, and the RKJZ* correlation can predict it with an average error of 18% (seven data points from III-A and III-B). However, at 100 psia, the vaporization of the unhydrogenated 400-1000°F cut is well established only at 775°F; at 700°F, neither the measurements nor the predictions are satisfactory.

Also included in Phase III-B was the measurement of the vapor pressure of three pure aromatic compounds and two Wyoming coal liquid fractions. Only partial results have been reported to date due to experimental difficulties at BYU. Upon completion of this work and the data analysis, work will begin on one of the Phase IV objectives, the revision of the Maxwell-Bonnett correlation for the vapor pressure of coal liquid fractions. The experimental difficulties have also delayed another Phase IV objective, VLE measurements on mixtures of Wyoming coal liquids with H₂ and CH₄.

The advisory committee that has been formed by EPRI for the review of the ongoing experimental work at Purdue and Rice Universities met twice in the past 12 months. The first meeting was at Palo Alto, California on October 21, 1977. The committee also met at Miami, Florida on April 14, 1978.

Chao's VLE measurements have provided valuable information on the interaction of H₂ with aromatic compounds at high temperatures (up to 805°F) and pressures (up to 3700 psia). The very satisfactory fit of these data with the RKJZ VLE correlation has convincingly demonstrated the reliability of this correlation and its applicability to calculations at the extreme conditions of the EDS coal liquefaction process.

Analysis of Chao's VLE Measurements

As reported in the Phase III-A final report, Professor K. C. Chao has made extensive VLE measurements on four H₂/hydrocarbon binaries as part of an EPRI-funded research project. The results of the data analysis with

*Redlich-Kwong-Joffe-Zudkevitch equation of state.

the RKJZ correlation were presented in the Phase III-A final report. Four additional H₂ binaries have been investigated by Chao (1), and the results of the data analysis for all 8 systems are summarized in Table 4-1.

Three of the additional systems are H₂ binaries of non-hydrocarbons: quinoline (C₉H₇N), m-cresol (C₇H₈O), and thianaphthene or 1-benzothiophene (C₈H₆S). For all three, as well as for H₂/m-xylene, RKJZ proved to be just as reliable a correlation as it had previously been found for the binaries of H₂ with tetralin, 1-methylnaphthalene, diphenylmethane, and bicyclohexyl. Furthermore, the optimum values of C_{ij}, the binary constant used by RKJZ, for the non-hydrocarbon binaries are very close to those found for the hydrocarbon binaries. Thus, RKJZ is apparently unaffected by the presence of N, O, or S in the aromatic molecules.

As the two sets of results for H₂/thianaphthene in Table 4-1 show, however, RKJZ can be excessively affected by a change in the specific gravity. When the specific gravity (at 60/60°F) of thianaphthene was adjusted from 1.15 to the more accurate 1.22 (3), the optimum C_{ij} dropped from 0.381 to 0.206--and the fit of the data improved considerably. This great sensitivity may be due to the very high density of thianaphthene. It should also be noted, however, that setting C_{ij} = 0.30 would give a good fit in both cases. Indeed, with the exception of the bicyclohexyl system, the average C_{ij} value is 0.25 ± 0.04, which is a good compromise for all the aromatic binaries.

Vapor-Liquid Equilibrium Measurements

The Phase III-B VLE work aimed at better establishing the experimental precision. To that end, BYU was asked to repeat four of the VLE measurements carried out in Phase III-A. As shown in Table 4-2, two of the scheduled measurements were at 100 psia and the other two at 1950-2000 psia.

The low-pressure measurements were made on the unhydrogenated 400-1000°F cut (see Phase III-A Final Report). Along with the runs made in Phase III-A, they have established fairly well the vaporization at 775°F. Furthermore, the predictions with RKJZ and two other VLE correlations were within 9% of the experimental results. However, the rerun at 700°F was in the poor agreement with the original run. In addition, the vaporization observed in both of these runs at 700°F was substantially higher than that predicted by all three VLE correlations. Further work in Phase IV on the vapor pressure and volatility of coal liquids is planned to better establish the effect of temperature on vaporization at low pressures.

The high-pressure measurements were made on mixtures of the hydrogenated solvent (see Phase III-A Final Report) with H₂ and CH₄. Two runs had been requested 775 and 850°F, but BYU repeated the 775°F run and also made one at 880°F.

Table 4-1

ANALYSIS OF CHAO'S VLE MEASUREMENTS
ON H₂/AROMATIC BINARIES WITH RKJZ (a)

<u>Aromatic Compound</u>	<u>t Range, °F</u> <u>(no. points)</u>	<u>P Range, psia</u>	<u>Optimum C_{ij}</u> ^(b)	<u>RMSD^(c) of</u> <u>Both K's, %^(d)</u>	<u>RMSD of</u> <u>K(H₂), %</u>
HYDROCARBONS					
Tetralin	373-732 (24)	294-3674	0.250	6.69	5.81
1-Methylnaphthalene	372-803 (27)	294-3674	0.178	5.14	4.51
Diphenylmethane	373-803 (27)	294-3674	0.224	4.97	2.91
Bicyclohexyl	372-803 (28)	294-3674	0.416	6.11	6.21
<u>m</u> -Xylene	373-588 (27)	288-3691	0.322	4.79	3.70
NON-HYDROCARBONS					
Quinoline	373-803 (27)	292-3671	0.272	2.82	2.65
<u>m</u> -Cresol	372-732 (41)	294-3685	0.293	3.61	3.18
Thianaphthene	370-805 (27)	290-3670	(e)	5.51	6.48
(1-benzothiophene)			(f)	3.64	4.41

(a) RKJZ refers to the Joffe-Zudkevitch modification of the Redlich-Kwong equation of state.

(b) C_{ij} is the binary constant used by RKJZ.

(c) RMSD = root-mean-square deviation.

(d) The optimum C_{ij} value was determined by minimizing the RMSD of both K-values. A variation of 0.1 in C_{ij} raise the RMSD by about 1 unit.

(e) With specific gravity (@ 60/60°F) of thianaphthene = 1.15.

(f) With specific gravity (@ 60/60°F) of thianaphthene = 1.22.

Table 4-2

PHASE III-B VAPOR-LIQUID EQUILIBRIUM MEASUREMENTS

(A) 400-1,000°F UNHYDROGENATED CUT AT 100 PSIA

<u>t, °F</u>	<u>Weight Fraction Vaporized</u>
700	0.241
775	0.447

(B) H₂/CH₄/HYDROGENATED SOLVENT

	<u>t, °F</u>	<u>P, psia</u>	<u>Charge, wt%^(a)</u>		<u>Weight Fraction Vaporized</u>	<u>Weight K-values</u>	
			<u>H₂</u>	<u>CH₄</u>		<u>H₂</u>	<u>CH₄</u>
	775	1950	1.72	2.45	0.302	34.2	20.5
Rerun	775	1950	1.13	1.60	0.213	30.0	18.7
	850	2000	1.57	2.18	0.407	16.2	11.3
Not requested	880	2000	1.85	2.62	0.534	15.0	11.7

(a) The balance is the hydrogenated solvent; see Phase III-A Final Report.

As noted in the Phase III-A Final Report, the H_2/CH_4 /hydrogenated solvent mixture is apparently close to its critical point at 880°F (under a pressure of 1950-2000 psia). This is also illustrated in Figure 4-1, where all the data for the weight fraction vaporized from Phases III-A and III-B have been plotted. The temperature should definitely be kept below 880°F when the coal liquid is as light as the hydrogenated solvent.

If only the three runs at 775-850°F are considered, the RKJZ VLE correlation predicts the weight fraction vaporized with an average deviation of 15.3%. (The C_{1j} for the H_2 /coal liquid fractions was set equal to 0.25). When the four Phase III-A runs at 700-850°F are included, the average deviation goes up to 18.1%, with a bias of +1.8%. No particular trend with temperature was found, but the overall fit was much inferior to the 11% obtained in the analysis of the Phase II measurements (2). However, it has also been found that only at 775°F did the replicate Phase III-B measurements match those from Phase III-A.

The VLE data will be reanalyzed when the complete results of the Phase III-B experimental work are reported by BYU. It is expected that it will be possible to reduce the average error of the RKJZ predictions by correcting the vapor pressure correlation for coal liquid fractions (see below).

Vapor Pressure Measurements

BYU has not yet completed the Phase III-B experimental work on the vapor pressure of three pure aromatic compounds and two Wyoming coal liquid fractions. Only partial data have been reported for naphthalene, *cis*-decalin, and one of the fractions. Also the vapor pressure of phenanthrene is being redetermined because the Phase III-A measurements had to be discarded due to thermocouple problems. The complete results on the above as well as on 2, 4-xylene and the second fraction are expected soon and will be reported in the next quarterly report.

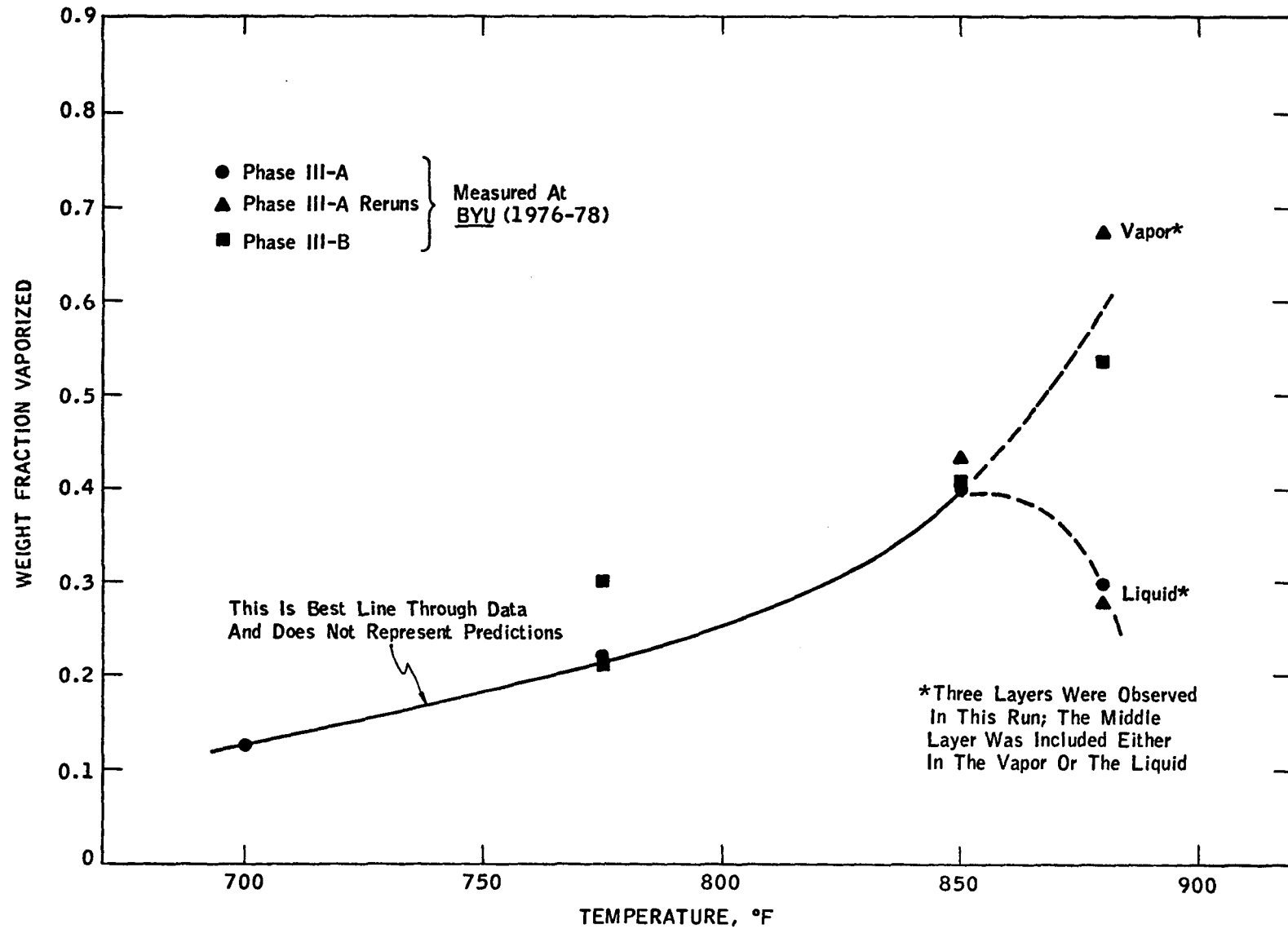
Future Work

Upon completion of the vapor-pressure work at BYU, the data will be analyzed, along with those from Phase III-A and any reliable information found in the literature. The resulting extensive data base will then be employed in the revision of the Maxwell-Bonnett correlation for the vapor pressure of coal liquid fractions. As noted in the Phase III-A final report, this correlation does not work well for the highly aromatic fractions typical of coal liquids.

The six VLE measurements on mixtures of Wyoming coal liquids scheduled for 1978 may have to be deferred to 1979 due to experimental difficulties at BYU. Scheduling of this work will be reported in the next quarterly report.

Figure 4-1

VAPORIZATION OF H_2/CH_4 /HYDROGENATED SOLVENT MIXTURES AT 1950 PSIA



References

1. Chao, K. C., "Phase Equilibrium in Coal Liquefaction Processes," RP-367, Annual Report to EPRI for 1977.
2. EDS Coal Liquefaction Process Development, Phase IIIA, Quarterly Technical Progress Report, January 1-June 30, 1976 (FE-2353-2).
3. Timmermans, J., "Physico-Chemical Constants of Pure Organic Compounds," Vol. 2, Elsevier Publishing Company, Amsterdam, 1965; p. 424.

4.2 CLPP Inspection And Physical Property Data

Summary

The objective of this program is the development of correlations for predicting physical properties of coal liquids. The properties of interest include density, viscosity, and surface tension. In order to provide a data base for the correlation development, experimental programs have been undertaken at Brigham Young University (BYU). To date, density and viscosity data on Illinois coal liquids have been reported; surface tension data are expected during the last half of 1978.

A correlation has been developed for density in the form of a series of correction factors for the effects of temperature, pressure and dissolved hydrogen on the density at 60°F and 1 atm. A preliminary correlation has also been developed for viscosity. In this case, the dissolved hydrogen does not significantly affect the viscosity.

A similar experimental program has been proposed for Wyoming coal liquids. It will be initiated as soon as it can be scheduled at an outside laboratory.

Illinois Coal Liquids

• Density

Measurements of the densities of a number of Illinois coal liquids were completed at BYU. The liquids included hydrotreater feed (A-3), recycle solvent (A-6), fractionation feed (A-10) and heavy vacuum gas oil (A-2). Measurements were made over the range 200-800°F and 300 to 3000 psia. In addition, densities of A-3 and A-6 saturated with hydrogen were also measured.

In general, our present design tool, the Exxon Data Library (EDL) is satisfactory for predicting the effects of temperature, pressure, and dissolved hydrogen on the density of coal liquids, provided a good low temperature, low pressure value is available. Maximum errors of about 5% are observed at the highest temperatures and pressures. However, the procedure requires that the coal liquid be broken down into pseudo-components, characterized by boiling point and density. The pseudo-components breakdown procedure requires some kind of distillation data.

To avoid the need for distillation data and the pseudo-component breakdown, another correlation has been developed. In this correlation the effects of temperature, pressure and dissolved hydrogen appear as correction factors on the density at 60°F and 1 atm. The functions describing the effects of pressure and dissolved hydrogen are themselves temperature dependent. The specific correlation is:

$$\begin{aligned}\rho_{T,P} = & \rho_{60} + 7.1026 \times 10^{-5} - 3.4848 \times 10^{-4} \tau - 2.36618 \times 10^{-7} \tau^2 \\ & + (3.05945 \times 10^{-6} + 5.78376 \times 10^{-11} \tau^2)P \\ & - (1.74486 + 4.385 \times 10^{-7} \tau)H\end{aligned}$$

where: $\rho_{T,P}$ = density at system temperature ($^{\circ}\text{F}$) and pressure (psia),
g cm³

ρ_{60} = density at 60 $^{\circ}\text{F}$ and 1 atm, g/cm³

$\tau = t \text{ } ^{\circ}\text{F} - 60$

P = Pressure, psia

H = dissolved hydrogen, wt. %

For 148 data points, the average deviation between observed and calculated densities is 0.7%.

• Viscosity

Measurements of the viscosities of A-3, A-6, and A-10 have been completed by BYU. Data were taken over the range 200-800 $^{\circ}\text{F}$ and 200-3000 psia. In addition viscosities of A-3 and A-6 saturated with hydrogen were also measured.

Viscosities predicted by EDL are consistently higher than observed. Deviations between observed and calculated viscosities range up to 40-50%. Moreover, viscosities predicted by EDL are virtually independent of pressure. A preliminary correlation has been developed by means of linear regression of the data, which has the form:

$$\log \mu = A + \frac{B}{T} + (C + \frac{D}{T}) P$$

where μ is the viscosity, cp

T is the temperature, $^{\circ}\text{R}$

P is the pressure, psia,

A, B, C, D are constants.

The deviations between observed and calculated viscosities are less than 10% for 56% of the points, between 10 and 20% for 27% of the points, and over 20% for 17% of the points. Note that the effects of dissolved hydrogen are apparently not significant (at least at the statistically significant level built into the linear regression computer program). Moreover, the constants A, B, C and D were essentially the same for all three stocks.

During the next six months, as additional viscosity data become available, improvement of the above correlation, and testing of other correlation forms will be carried out.

- Surface Tension

Data on the surface tension of the same coal liquids studied above have not yet been received from BYU.

Wyoming Coal Liquids

In order to gain some insight into the effects of different kinds of coal on the properties of coal liquids, an experimental program on Wyoming coal liquids similar to the one described above for Illinois coal liquids has been planned. Implementation of the program has been delayed, however, due to scheduling difficulties at BYU. Samples were sent to Exxon's Analytical and Inspection Division for "routine" inspections. The results are shown in Table 4-3.

We are seeking other laboratories to carry out our experimental programs. The work on Wyoming coal liquids will be initiated as soon as a suitable laboratory is found.

4.3 Commercial Fractionator Recovery Prediction

The overall objective of this program is to develop correlations to permit the prediction of the distillate product recovery achievable in the commercial vacuum tower and coker fractionator. A knowledge of the product recovery in the vacuum tower is required for proper design. Knowledge of FLUID COKING fractionator performance is essential to determine commercial yields. To achieve this objective, laboratory distillations were performed on the feed and product streams of the CLPP vacuum flasher.

Based on the analysis of the distillation data, the tentative conclusions are:

- Hivac C distillations at 0.2 (run in the EDS Laboratory) and 1.0 mm Hg (run in the Baytown Crude Assay Laboratory) are identical in that they yield identical atmospheric equivalent distillation curves.
- Characterization corrected petroleum normal boiling point component vapor pressures and coal normal boiling point component vapor pressures give nearly identical results when used to extrapolate vacuum distillation temperatures to atmospheric equivalent temperatures.
- The existing petroleum distillation interconversion correlations can be used for coal liquid samples when converting from a Hivac C to a 15/5 distillation.

Table 4-3

INSPECTION DATA ON WYOMING COAL LIQUIDS

	A-5 Unhydrotreated Solvent	A-6 Hydrotreated Solvent	V-1 Bottoms Spent Solvent	A-2 Heavy Vacuum Gas Oil
Specific gravity, 60°F/60°F	0.9695	0.9865	0.9975	1.0696
Molecular weight	176	186	191	242
C, weight %	85.86	89.22	89.86	88.68
H	9.09	9.25	8.87	7.71
N	<0.3	0.37	0.44	1.00
Kinematic Viscosity cSt, 100°F	3.06	3.17	4.52	109.4
210°F	1.14	1.18	1.42	6.50
Conradson Carbon, weight %	0.35	0.06	0.20	4.82
Modified naphtha insolubles, weight %	0.89	0.13	0.69	11.74
Surface Tension, 77°F, dynes/cm	35.8	37.8	36.9	30.1

Background

The procedures used for vacuum distillation tower design have the feed defined via a Normal Boiling Point (NBP) curve. The projected product yields and performance of a commercial vacuum tower and coker are based on these NBP curves. One sub-objective of this program is to define the NBP curves for the Exxon Donor Solvent coal liquefaction bottoms and heavy coal liquids to facilitate the design and prediction of the performance of these units when used for coal liquids.

For petroleum, the standard source for obtaining an NBP curve is crude assay data. Where assay-type distillation data are not available, correlations have been developed for petroleum which are used to convert data from various other distillations to a NBP curve. It is desirable to use the same procedure for coal liquids, which means coal liquid based interconversion correlations are required for the various analytical distillation methods used for coal liquids. A second sub-objective is to develop the coal liquid based interconversion correlations.

Data

The distillations listed in Table 4-4 were performed on the feed and product streams of the CLPP vacuum flasher. The samples used for these distillations were obtained from CLPP Yield Period 256. The blended feed is a blend of the three product streams -- LVGO, HVGO and vacuum flasher bottoms.

For the feed and blended feed, 15/5 distillations were performed up to the endpoint indicated with a Hivac C and microlube distillation run on the resulting bottoms. Two Hivac C distillations were run on the majority of samples, one by the Baytown Crude Assay Lab at 1.0 mm Hg and the other by the EDS Lab at 0.2 mm Hg.

Raw Data Conversion

To analyze the data it is necessary to first convert the vacuum distillation temperatures to atmospheric equivalent temperatures. For this conversion, two different methods were tried and compared. One method used characterization corrected petroleum component vapor pressures and the other method used specific gravity corrected coal component vapor pressures. (See Phase III-A, Final Report on Coal Liquefaction VLE Data, (FE-2853-20)).

Once all the distillations were converted to atmospheric equivalent temperatures, the Hivac C atmospheric distillation temperatures were converted to 15/5 distillation temperatures using the petroleum interconversion correlations. This is necessary before combining streams, since stream blending can only be done on a 15/5 distillation basis. For the feed and blended feed, the 15/5 distillations were combined with the corresponding converted Hivac C distillations to get the total feed NBP curve. This was done for each 15/5 cut point. In addition, the distillations of the

TABLE 4-4

DISTILLATION OF EDS COAL LIQUIDS

<u>Stream</u>	<u>15/5 Endpoint</u>	<u>Vacuum Distillation</u>	
		<u>Hivac C</u>	<u>Microlube</u>
F-1 Feed	550°F	X	X
	650°F	X	X
Blended Vacuum Tower Feed	500°F	X	X
	550°F	X	X
	650°F	X	X
A-1 Bottoms		X	X
A-2 HVGO		X	X
LVGO	X		

LVGO, HVGO, and bottoms were combined to produce a total feed NBP curve. All of these combinations were performed for both types of Hivac C distillations (0.2 and 1.0 mm Hg) and also for both vapor pressure methods (petroleum component and coal component) used to convert the vacuum distillation temperatures to atmospheric equivalent temperatures.

One problem with performing the combinations was that all the Hivac C distillations had to be extrapolated to an endpoint. This was due to distillation pot temperatures reaching approximately 650°F, causing cracking of the sample. This necessitated terminating the distillation before all the sample had been distilled. The extrapolation was done by extending the Hivac C distillation curves using rectangular coordinate paper. Since these extrapolations gave final boiling points over 1500°F, the boiling point limitation of our computer programs, the extrapolations were cut off at 1500°F. Therefore, the combined feed curves do not represent the entire feed, but actually represents that portion of the feed which distills below 1500°F.

Discussion of Results

By comparing the feed curves produced from these combinations, several conclusions were reached. The three figures are plots of the feed NBP curve and illustrate the three tentative conclusions that were made.

Figure 4-2 shows the results of combining a 15/5 distillation to 650°F with the converted Hivac C distillation of the resulting bottoms for the actual feed. The only difference between the two curves is the pressure under which the Hivac C distillation was run (0.2 vs. 1.0 mm Hg). From this graph it was concluded that the two Hivac C distillations produced identical atmospheric equivalent temperatures to within the accuracy of the distillation procedure.

In Figure 4-3, the two vapor pressure methods used to convert the vacuum Hivac C distillation temperatures (at 1.0 mm Hg) to atmospheric equivalent temperatures are compared. From this figure it was concluded that the characterization corrected petroleum component vapor pressures and the coal component vapor pressures give virtually identical results when used to extrapolate vacuum distillation temperatures to atmospheric equivalent temperatures.

Figure 4-4 illustrates the applicability of the petroleum interconversion correlations. The blended feed curve results from combining a 15/5 distillation to 650°F and the converted Hivac C distillation on the resulting bottoms for a blended feed sample. This is compared to a curve obtained by combining the distillations of the LVGO, HVGO, and bottoms. The percents that each individual distillation contributes to the final curve is indicated on the figure. The dashed lines indicate the part of the final curve which was obtained from the extrapolated portion of a Hivac C distillation.

Figure 4-2

COMPARISON OF HiVac C AT 0.2 mm VS HiVac C AT 1.0 mm
ATMOSPHERIC EQUIVALENT TEMPERATURES

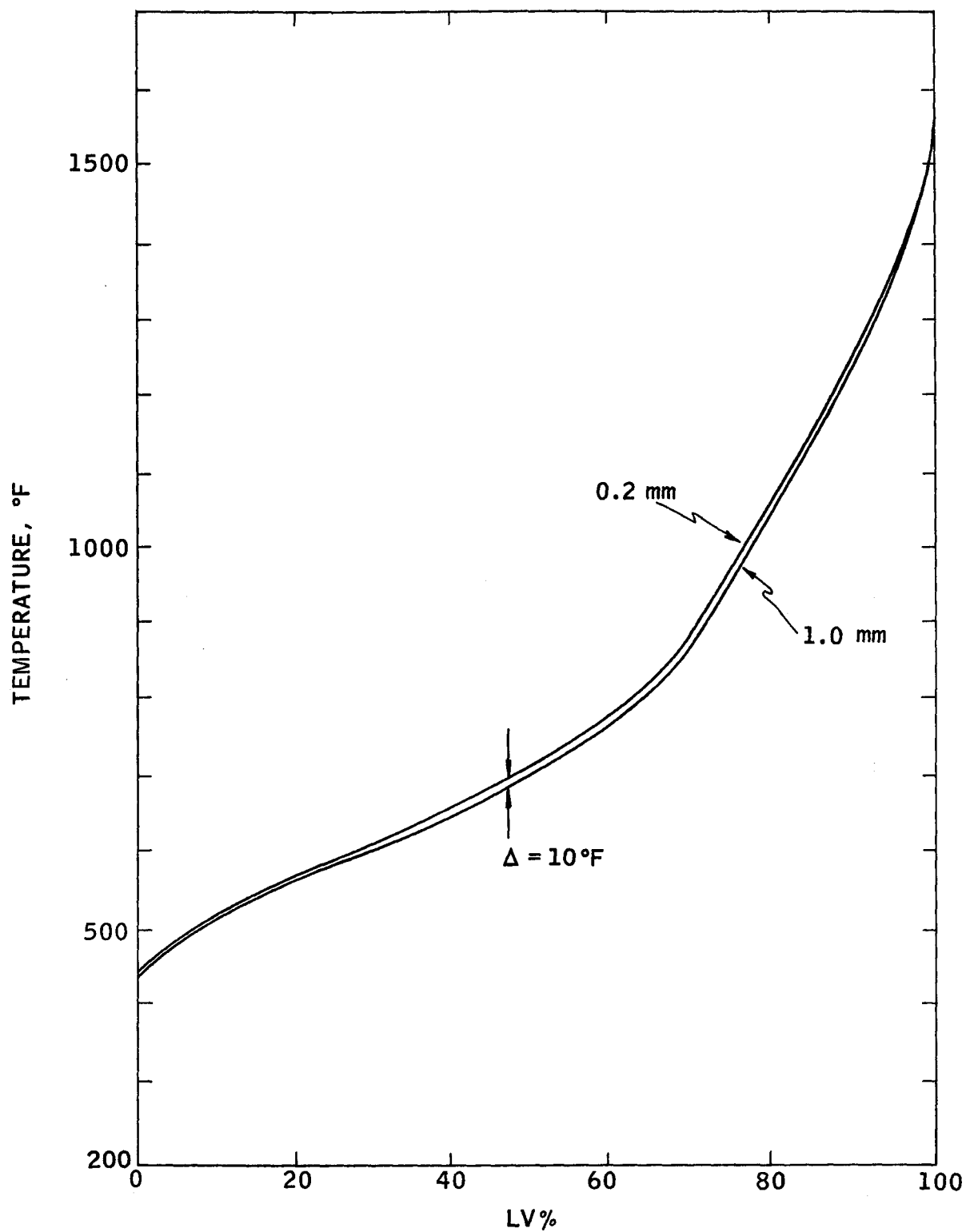


Figure 4-3

COMPARISON OF DISTILLATIONS USING PETROLEUM VS.
COAL COMPONENT VAPOR PRESSURES

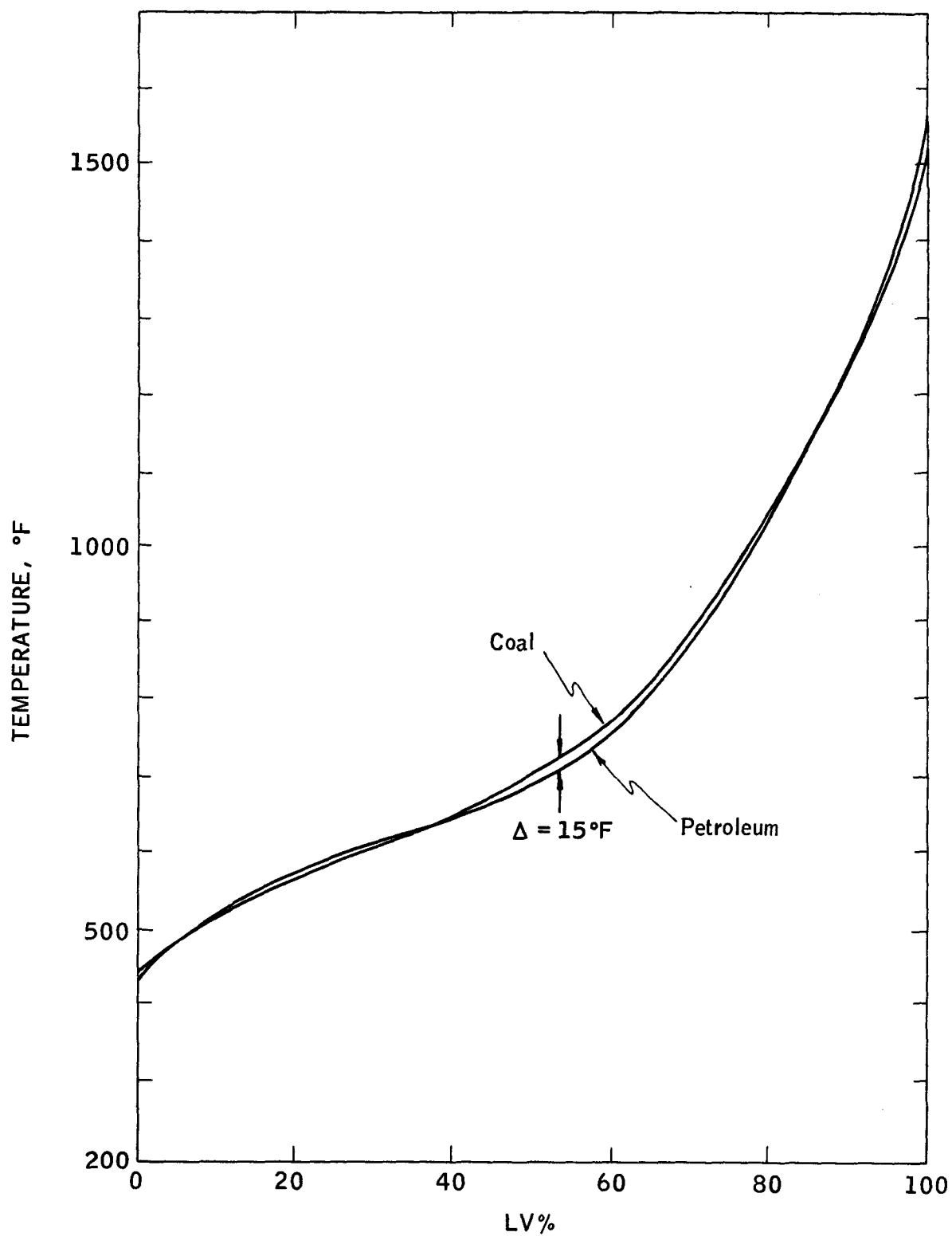
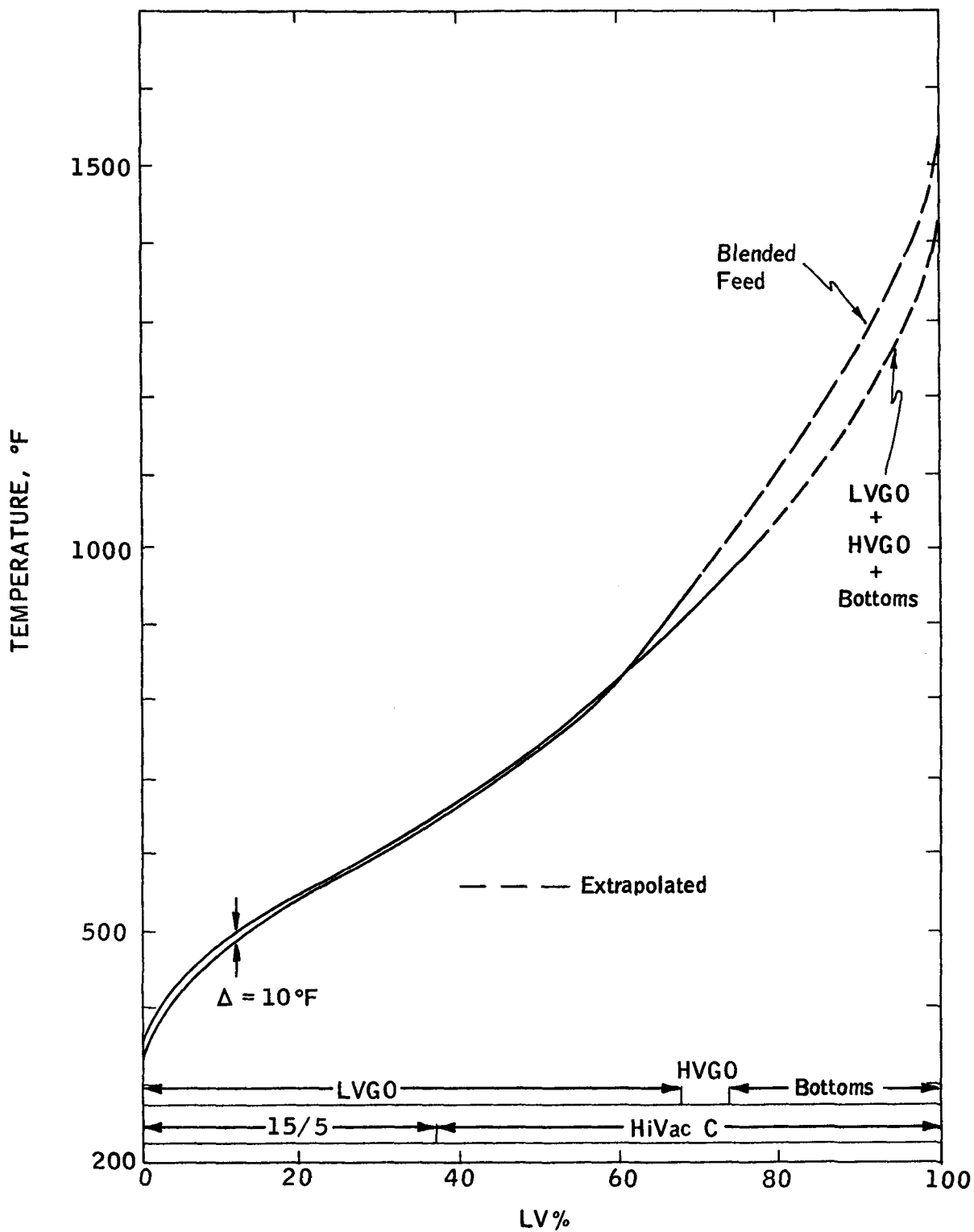


Figure 4-4

COMPARISON OF COMBINED DISTILLATIONS OF
LVGO + HVGO + BOTTOMS VS. BLENDED FEED



From the close agreement of the two curves in the non-extrapolated portion and the overall general smoothness in the curves, it was concluded that the existing distillation interconversion correlations can be used for coal liquids for converting a Hivac C to a 15/5 distillation.

Future Work

Continued work on this project will center around obtaining and analyzing: 1) distillation data on coker liquid products, and 2) data from metal Hivac distillations and Hivac C's on the metal Hivac cuts of the CLPP vacuum flasher feed for Illinois coal. Data for the coker liquid distillations will be used in a similar manner to the work just completed for the CLPP vacuum flasher for Illinois coal; i.e., suitability of interconversion correlation and use of vapor pressure extrapolations. Data from the metal Hivac and Hivac C cut distillations will be used to confirm the conclusions made from the previous round of vacuum flasher distillations. Additionally, the conclusions reached will be more fully tested by modeling the CLPP vacuum flasher. Finally, we shall attempt to develop correlations to convert data from a microlube distillation to a 15/5 distillation.

4.4 Vacuum Flasher Transfer Line Study

Summary

The primary objective of this Phase III-B study was to evaluate the transfer line pressure drop data which was obtained in Phase III-A and determine whether the existing proprietary correlations for two-phase (vapor/liquid) flow can be adjusted to predict three-phase (vapor/liquid/solid) pressure drop. Another objective of the program was to collect the necessary data to be used by EETD materials specialists to evaluate the effect of transfer line velocity on erosion in the transfer line and in the vacuum tower flash zone.

The objectives of this program were completed. A correction factor which is applied to the predicted two-phase pressure drop was found to account for the solids contribution to frictional pressure drop. This correction factor was found to be a function of the two-phase Reynolds number. The three-phase frictional pressure drop predictions were in good agreement with the observed data of Phase III-A, i.e., the average deviation was found to be $\pm 17\%$. This degree of accuracy is sufficient for commercial design of the transfer line. The correction factor also removed the underprediction bias that resulted when using the existing correlations alone.

Significant material wear was observed on the inside tower wall in the flash zone section opposite the tangential feed inlet point. However, no erosion problems were observed in the transfer lines which were in service during Phase III-A & B.

More complete information on this program was given in the Phase III-B Quarterly Technical Progress Report (Oct. 1-Dec. 31, 1977), FE-2893-7.

Transfer Line Pressure Drop Calculations

Table 4-5 summarizes the calculated CLPP transfer line pressure drops and the observed data from the Phase III-A work. A breakdown of the frictional, elevation, and acceleration pressure drop contributions is summarized in Table 4-6.

After reviewing literature correlations, a simple approach aimed at correcting the two-phase prediction (via existing correlations) was attempted and proved to be acceptably accurate. The Phase III-A data were first screened by plotting observed frictional pressure drop vs. a simplified form of the two-phase frictional drop equation as shown in Figure 4-5. The two-phase frictional pressure drop term in Figure 4-5 was based on flow properties that were taken as the average of upstream and downstream properties in the transfer line. This screening eliminated data from test #2C. The correlation in Figure 4-5 seems to fit the observed data well. However, this correlation which is a function of average flow properties would not be applicable to scale up to large commercial size lines where upstream and downstream flow properties differ more than in the smaller CLPP transfer lines.

A frictional pressure drop correction, i.e., observed pressure drop divided by calculated two-phase pressure drop ($\Delta P_{3\phi}/\Delta P_{2\phi}$), was then found via regression analysis to correlate best with the two-phase Reynolds number. The two-phase pressure drop ($\Delta P_{2\phi}$) was calculated by a rigorous computer technique with the transfer lines segmented into 1-inch sections and flow properties calculated for each section. The correlation is illustrated in Figure 4-6. A comparison between predicted and observed frictional pressure drops is shown in Table 4-7. The average deviation is + 17%. This degree of accuracy is sufficient for commercial design of the transfer line. The correction also removes the underprediction bias that results when using the existing correlations alone.

Erosion Observations

During the 1977 CLPP operations, significant erosion problems developed in the vacuum tower flash zone. Periodic tower inspections found repeated material wear (0.10 to 0.25 inches deep) on the inside of both the 5-inch and 12-inch flash zone sections. The location of this material wear was directly across from the feed tangential inlet. There was no visible sign of material wear in either the 1-inch or 0.5-inch transfer lines that were in service during Phase III-A & B. The erosion problems in the CLPP vacuum flash section were studied in another EETD program (Engineering Research and Development, Development of Construction Materials) as part of an overall CLPP materials study.

Table 4-5

SUMMARY OF TRANSFER LINE CALCULATIONS

Test No.**		1A	1B	1C	1D	2A	2B	2C	2D	2E	2F	3A	3B	4A
Distance From Tangential Inlet, in.	Vacuum Tower Tangential Inlet	-	-	-	-	-	-	-	-	-	-	-	-	-
	Downstream Location	4	4	4	4	1	1	1	1	1	1	1	1	1
	Lower Mid-Length Location	17	17	17	17	17	17	17	17	17	17	17	17	17
	Mid-Length Location	30	30	30	30	28	28	28	28	28	28	28	28	28
	Upper Mid-Length Location	43	43	43	43	40	40	40	40	40	40	40	40	40
Temperature, °F	Upstream Location	68	68	68	68	57	57	57	57	57	57	57	57	57
	Vacuum Tower Tangential Inlet	636	635	612	646	637	634	629	635	648	649	636	633	650
	Downstream Location	637	636	616	650	647	644	638	645	659	658	646	643	666
	Lower Mid-Length Location	638	639	619	653	650	646	641	648	661	660	650	646	672
	Mid-Length Location	638	640	620	654	652	647	642	649	663	661	651	647	675
Pressure, psia	Upper Mid-Length Location	639	641	621	654	653	649	643	650	664	662	653	649	677
	Upstream Location	639	642	622	656	655	650	644	652	665	663	654	650	680
	Vacuum Tower Tangential Inlet	1.03	0.96	0.77	0.67	2.63	2.38	1.79	1.61	1.80	2.00	2.42	2.08	3.00
	Downstream Location	1.05	1.05	1.06	0.91	5.89	5.39	3.98	3.80	4.36	4.41	5.27	4.66	6.09
	(measured)	(1.1)	(1.1)	(1.1)	(1.1)	-	-	-	(4.1)	(5.1)	(4.8)	(4.7)	(4.7)	-
Fluid Velocity, ft/s	Lower Mid-Length Location	1.15	1.34	1.34	1.17	7.52	6.75	4.92	4.67	5.33	5.45	6.70	5.90	7.73
	Mid-Length Location	1.21	1.43	1.43	1.25	8.33	7.48	5.42	5.15	5.86	6.02	7.45	6.55	8.59
	Upper Mid-Length Location	1.26	1.52	1.52	1.32	9.09	8.17	5.89	5.59	6.36	6.54	8.15	7.15	9.38
	Upstream Location	1.35	1.67	1.67	1.46	10.04	9.00	6.45	6.15	6.99	7.20	9.02	7.91	10.36
	(measured)	(1.7)	(1.7)	(1.7)	(1.7)	(10.5)	(9.1)	(9.1)	(10.8)*	(10.2)*	(10.4)*	(9.2)	(9.2)	-
	Vacuum Tower Tangential Inlet	173	300	383	389	573	587	411	522	595	571	541	535	636
	(% of critical velocity)	(36)	(59)	(75)	(75)	(100)	(100)	(100)	(100)	(100)	(100)	(100)	(100)	(100)
	Downstream Location	169	271	270	278	233	244	164	204	235	246	231	222	293
	Lower Mid-Length Location	134	183	183	186	181	192	128	162	189	197	177	171	224
	Mid-Length Location	128	171	171	173	161	171	113	145	171	177	156	152	198
	Upper Mid-Length Location	122	160	160	162	147	155	102	132	157	162	141	137	180
	Upstream Location	113	144	144	146	131	140	92	118	141	146	125	122	160
Enthalpy, BTU/lb (all locations)		417.30	427.53	428.28	432.95	438.22	416.57	394.85	415.91	424.22	412.48	445.43	436.54	487.19

Notes:

* "Suspect" pressure readings which may have been due to pressure tap plugging.

** Refers to Phase III-A CLPP test work.

Table 4-6

SUMMARY OF TRANSFER LINE PRESSURE DROP CALCULATIONS

Test No. ³	1A	1B	1C	1D	2A	2B	2C	2D	2E	2F	3A	3B	4A
Total	0.28	0.60	0.60	0.53	4.04	3.59	2.45	2.33	2.62	2.78	3.73	3.23	4.26
(measured)	(0.6)	(0.6)	(0.6)	(0.6)	(4.5) ¹	(3.7) ¹	(5.1) ¹	(6.7) ²	(5.1) ²	(5.6) ²	(4.5)	(4.5)	(--)
Frictional	0.28	0.53	0.50	0.47	3.60	3.37	2.31	2.15	2.41	2.58	3.28	2.89	3.54
(measured)	(0.5)	(0.5)	(0.5)	(0.5)	(4.0) ¹	(3.4) ¹	(4.9) ¹	(6.5) ²	(4.9) ²	(5.4) ²	(4.0)	(4.1)	(--)
Acceleration	0.02	0.09	0.11	0.08	0.45	0.24	0.16	0.20	0.22	0.21	0.47	0.36	0.73
(measured)	(0.1)	(0.1)	(0.1)	(0.1)	(0.5) ¹	(0.3) ¹	(0.2) ¹	(0.2) ²	(0.2) ²	(0.2) ²	(0.5)	(0.4)	(--)
Elevation	-0.02	-0.02	-0.01	-0.02	-0.01	-0.02	-0.02	-0.02	-0.01	-0.01	-0.02	-0.02	-0.01
Liquid Volume Fraction	0.001	0.001	0.001	0.001	0.0026	0.0025	0.0040	0.0021	0.0018	0.0020	0.0021	0.0021	0.0017
Liquid Holdup Factor	0.024	0.024	0.018	0.024	0.040	0.060	0.105	0.051	0.043	0.052	0.030	0.032	0.013
Two-Phase Viscosity, cP	0.068	0.069	0.060	0.065	0.071	0.095	0.139	0.080	0.076	0.086	0.063	0.071	0.027
Two-Phase Density, lb/ft ³	0.022	0.025	0.026	0.021	0.105	0.068	0.082	0.064	0.054	0.046	0.113	0.097	0.098
Two-Phase Reynolds Number	4700	7400	9400	6700	13000	6400	3700	6300	6600	5300	15300	11300	40000
Two-Phase Friction Factor	0.019	0.017	0.016	0.017	0.016	0.020	0.024	0.019	0.019	0.020	0.015	0.016	0.010
Two-Phase Velocity, ft/sec	127	188	196	190	185	185	130	164	191	200	182	175	231

Notes: (1) Estimated values.

(2) Based on "suspect" upstream absolute pressure readings.

(3) Refers to Phase III-A CLPP test work.

(4) Based on rigorous computer calculations with the transfer lines segmented into 1-inch sections and flow properties calculated for each section.

(5) Based on flow properties taken as the average of upstream and downstream properties in the transfer line.

Table 4-7

SUMMARY OF FRICTIONAL
PRESSURE DROP CORRELATION RESULTS

Test No.*	Observed Pressure Drop, psi	Predicted 2-Phase Pressure Drop, psi	Deviation, %	Predicted 3-Phase Pressure Drop, psi	Deviation, %
1A	0.5	0.28	-44	0.37	-26
1C	0.5	0.50	0	0.61	+22
1D	0.5	0.47	-6	0.60	+20
2A	4.0	3.60	-10	4.25	+6
2B	3.4	3.37	-1	4.30	+26
3A	4.0	3.28	-18	3.80	-5
3B	4.1	2.89	-30	3.46	-16
Average Deviation, %			-16 (underpredic- tion bias)		+17 (no bias)

Note: *Refers to Phase III-A CLPP test work.

Figure 4-5

FRICTIONAL PRESSURE DROP DATA SCREENING

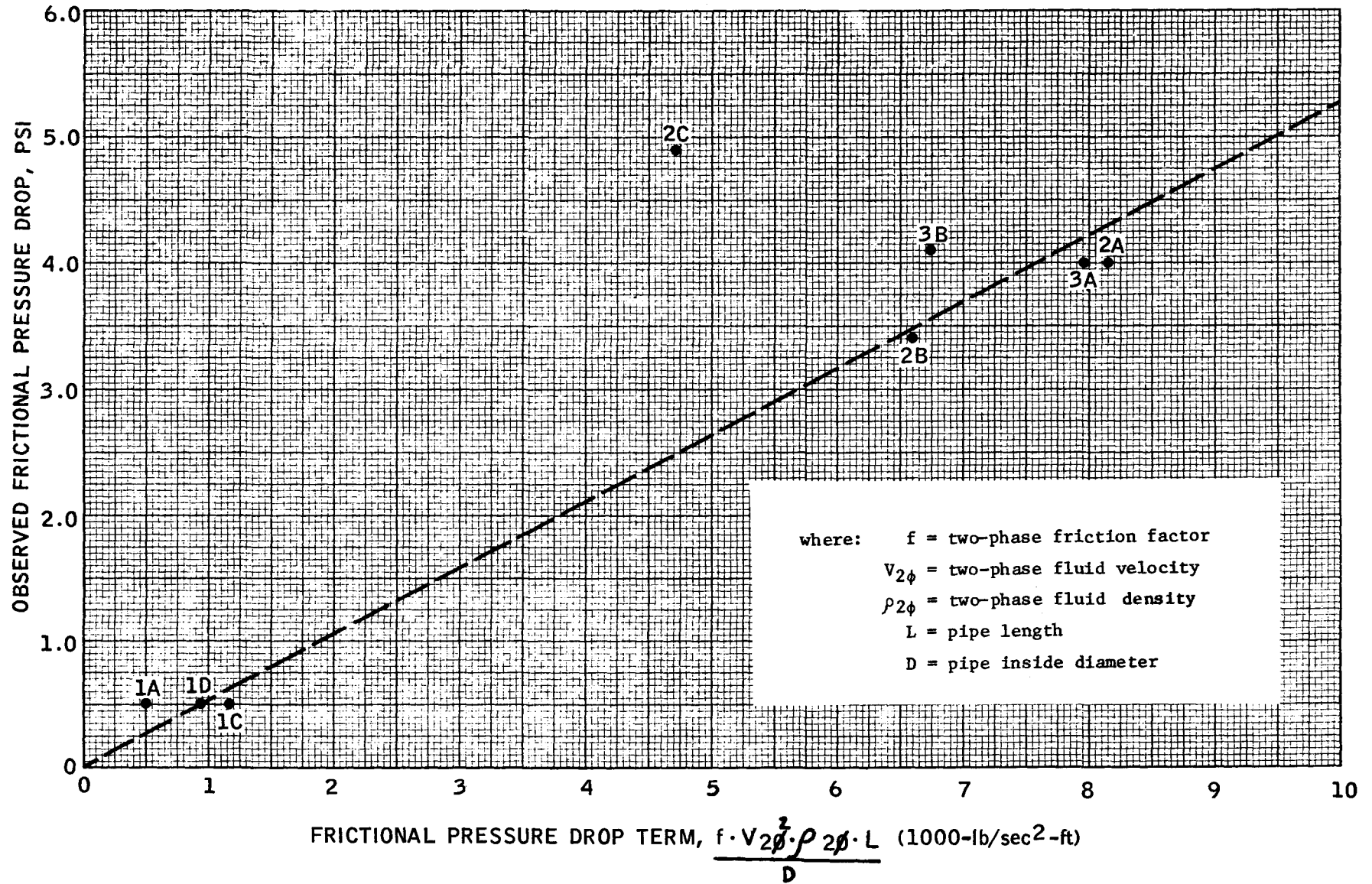
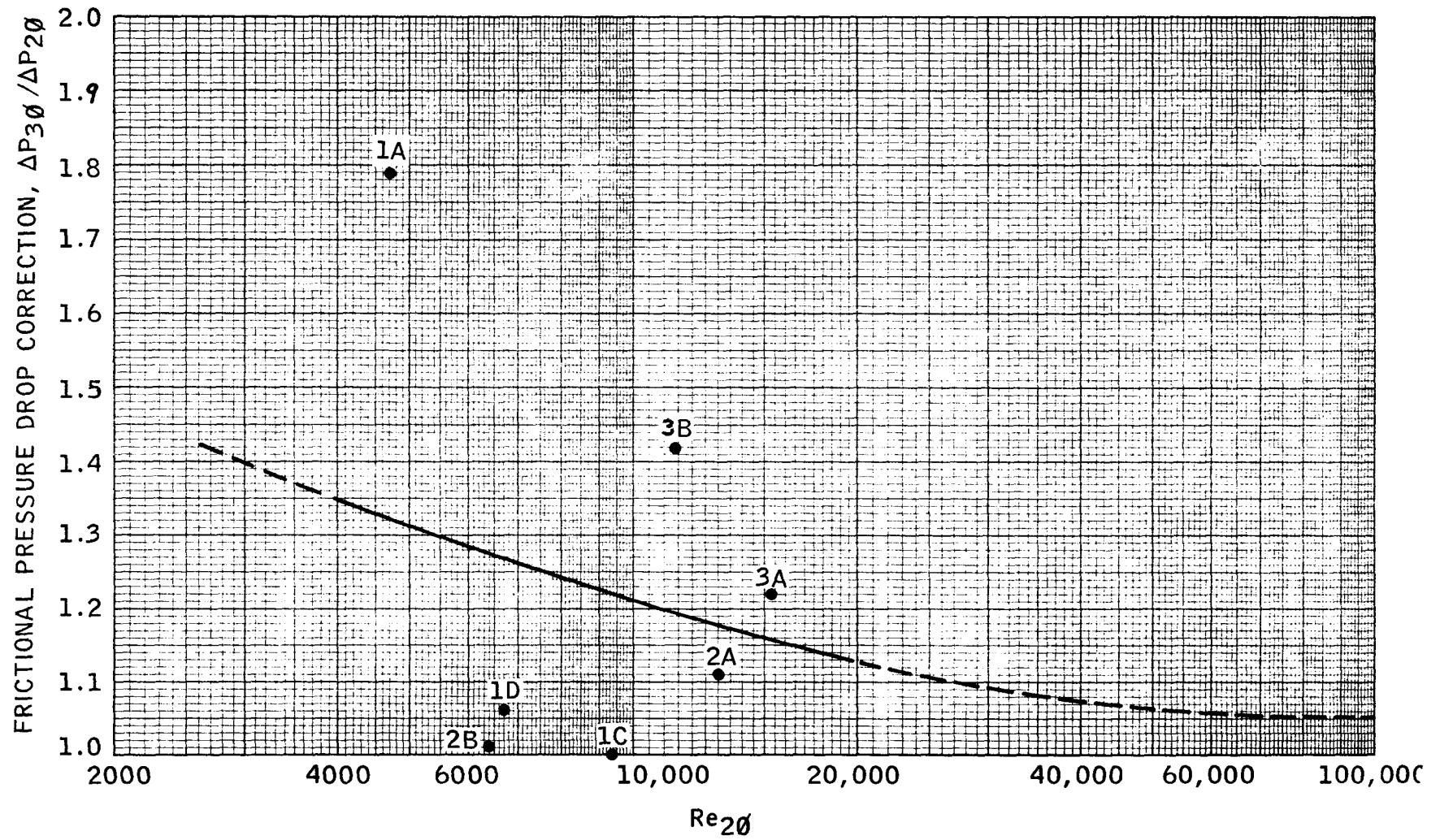


Figure 4-6

FRICTIONAL PRESSURE DROP CORRECTION
VS
TWO-PHASE REYNOLDS NUMBER



Conclusion

The results of the vacuum flasher transfer line study are:

- A correction factor which is applied to the two-phase pressure drop prediction (via existing correlations) was found to account for the solids contribution to pressure drop. The correction factor is a function of the two-phase Reynolds number.
- Predicted transfer line pressure drops are in good agreement with observed data. The average deviation is + 17%.
- Significant material wear was observed on the inside wall of the vacuum tower in the flash zone section opposite the feed tangential inlet point. However, no erosion problems were observed in the transfer lines that were in service during Phase III-A & B.

Future Work

This work completes the overall objectives of the program and no further CLPP data will be studied. Plans are to take data on ECLP to further verify the pressure drop correlations and the effect of transfer line velocity and line back-pressure on entrainment generation in the larger vacuum tower.

4.5 Vacuum Tower Fouling Study

Summary

The objective of this program, which was initiated in Phase III-A, was to determine from CLPP data whether the small amount of 1000°F+ entrainment which is always present in vacuum pipestill flash zones will foul tower internals in ECLP and in a commercial plant. Specific objectives of the Phase III-B and Phase IV vacuum tower fouling studies were (a) to evaluate the long-term (1 to 2 months) fouling characteristics of a "dry" (not washed as in commercial designs) de-entrainment grid, and (b) to compare the predicted and the observed separation efficiency of the feed tangential inlet nozzle and the collection efficiency of the de-entrainment grid. Background to the above study was described in detail in the Phase III-A final report (FE-2353-20).

In mid-July 1977, after 332 hours of intermittent service with Wyodak coal, the vacuum tower grid section was found to be heavily filled with coke-like solids while the inside wall of the tower had a thin uniform layer of coke-like deposition. Abnormally high bottoms level upsets in the tower, aggravated by the inability of the material to drain because of the high velocity through the chimney tray, resulted in severe solids accumulation and grid section fouling. The chimney tray was removed from the tower to avoid these problems in the future. However, further grid fouling test work could not be performed because the 1977 CLPP operations were curtailed in October. Thus, due to the bottoms level upsets and because of unit unavailability, data on the long-term fouling characteristics of the de-entrainment grid was not obtained in Phase III-B and this work was postponed to 1978 as part of the Phase IV program.

In the first half of 1978, the vacuum flash equipment section of CLPP operated with the "dry" de-entrainment grid in the 12-inch vacuum tower with no serious fouling problems. Two inspections of the vacuum tower and de-entrainment grid were made. Both inspections followed relatively short runs.

Entrainment observations and calculations based on the Phase III-A and III-B work showed that the tangential feed inlet nozzle is effective in disengaging most of the 1000°F+ slurry drops entering with the vapor/liquid/solid vacuum tower feed. The drop sizes of 1000°F+ entrainment from the flash zone were estimated to be <25 microns in diameter. The de-entrainment grid was found to be effective in partially removing drops as small as 5 microns in diameter.

Vacuum Tower Fouling Observations

- July 1977 Vacuum Tower/De-entrainment Grid Fouling Inspection

The CLPP vacuum tower with the five-inch flash zone section was inspected in mid-July after 332 hours of intermittent service with Wyodak feed coal. The tower grid section was found to be filled with about 90 lbs of coke-like solids. The inside wall of the entire top section of the tower was found to have about a 1/16-inch uniform layer of deposition. Abnormally high bottoms level upsets in the tower (supported by statements in the operators' log) was suspected as the reason for the severe grid section fouling. Once the bottoms slurry got above the chimney tray which was directly below the grid, the high vapor velocity through the chimney tray prevented the material from draining down the tower. This caused very high residence times and solids accumulation. The chimney tray was removed from the tower to avoid these problems in the future. However, further grid fouling test work could not be performed because the 1977 CLPP operations were curtailed in October. Thus, due to the bottoms level upsets and because of unit unavailability, data on the long-term fouling characteristics of the de-entrainment grid was not obtained in Phase III-B and was postponed to 1978 as part of the Phase IV program.

- October 1977 Vacuum Tower Fouling Inspection

Another inspection was made of the twelve-inch vacuum tower (without the grid) in mid-October after 789 hours of intermittent service with Illinois feed coal. This service period covered high bottoms cut point operations. The tower bottoms section (below the flash zone) was found to be relatively clean. However, the top section shell wall was found to be covered with coke-like solids. The average thickness of the wall fouling varied along the length of the top section of the tower (1/2 to 1-inch). The thickest part of the deposition was observed in the middle of the tower top section where wall temperatures as high as 800°F were measured. Similar fouling was also observed on an axial thermowell which extended from the top of the tower down to the flash zone. Two distinct and easily separable layers of deposition were observed on the tower shell wall and on the thermowell.

It was suggested that formation of these two layers was due to bottoms level upsets resulting from plugging of the bottoms withdrawal/pumparound loop before shutdown.

Various analyses were made on the solids samples that were collected from several locations in the vacuum tower. It was concluded that the solids deposition on the tower shell wall and on the thermowell are due to high bottoms level upsets and slurry contacting the tower top section during the high cut point operations and the resulting high temperature profile in the tower. The thickness of deposition was found to be directly related to the temperature on the shell of the vessel.

- April 1978 Vacuum Tower/De-entrainment Grid Inspection

After erratic start-up operations, a thin layer of deposition (< 1/16-inch) was found on the lower section of the de-entrainment grid with the grid top section and tower shell relatively clean. The thin layer increased in thickness uniformly from the top to the bottom section of the grid.

- May 1978 Vacuum Tower/De-entrainment Grid Inspection

After some short-term runs, a non-uniform layer of deposition was found on the grid. A 1/8 to 3/16-inch layer of deposition was found on the lower section of the grid, while the top section was found to be relatively clean. This non-uniform deposition layer was thought to be the result of the bottoms slurry stream contacting the lower grid due to level upsets. This was substantiated by the fact that prior to the May shutdown the vacuum tower bottoms pump had seized and the bottoms level may have risen above the flash zone and reached the grid. The grid was cleaned and put back into service to obtain long-term fouling data.

Vacuum Tower Entrainment

In the Phase III-A study, the entrainment rates of 1000°F+ material from the vacuum tower were found to be negligible. The measured entrainment rates under conditions of subcritical (~300 ft/sec) and critical (~500 ft/sec) feed inlet velocities showed no significant difference, i.e., 0.02 to 0.05 lb/hr. The average rate was found to be ~0.001 lb of 1000°F+ entrainment per lb of vapor leaving the tower.

During the Phase III-B CLPP operations, a series of high bottoms cut point runs were made to study higher than normal temperature operation of the vacuum flash section. Flash zone temperatures in the F-1 vacuum tower as high as 775°F (at 1.2 psia) were achieved (for several hours) at various severity levels with the 12-inch flash zone section (without grid) and with a 1-inch transfer line.

Ash carryover data and flow rate measurements taken during the high bottoms cut point runs were also utilized in this study to evaluate the 1000°F+ entrainment rates. Entrainment calculations were based on ash carryover into the A-2 heavy vacuum gas oil (HVGO) stream. Entrainment rates during the high cut point runs were found to be consistent with observations in the Phase III-A test work. The average rate was again found to be ~0.001 lb of 1000°F+ entrainment per lb of vapor.

Entrainment Removal Model Of The Tangential Inlet And Grid

An entrainment removal model was developed to determine the separation efficiency of the feed tangential inlet nozzle and the collection efficiency of the de-entrainment grid. The model predicts the separation of entrained droplets due to both the cyclonic effect of the feed inlet and impaction collection on the de-entrainment grid in the vacuum tower. Drop sizes of the 1000°F+ entrainment from the flash zone were estimated to be < 25 microns in diameter. Predicted and observed entrainment rates agreed favorably, i.e., the average deviation was ~25%. The entrainment removal model showed that almost all of the entrainment (> 99.9%) was removed by the centrifugal force imparted by the tangential feed inlet nozzle with ~4% of the remaining entrainment being removed by the grid.

• Tangential Inlet Separation Efficiency

The method for predicting the separation efficiency of the tangential inlet was based on cyclone efficiency calculations. Based on feed inlet flow conditions and physical properties, drop size distributions were calculated at the tangential inlet. These calculated drop sizes were assumed to follow an upper limit log-normal probability distribution (7) with a standard deviation of 1.7, and were based on the Weber number criteria for determining the maximum stable drop size generated at the feed tangential inlet (3, 7).

The critical drop diameter, or smallest separable drop diameter, was determined based on inlet geometry and flow conditions. Assuming that the flash zone behaves as a perfect cyclone, the distribution of droplets escaping the tangential inlet was calculated based on the cyclone collection efficiency model. The escaping drop size distribution was then used to determine the collection efficiency of the de-entrainment grid.

• Collection Efficiency of the De-Entrainment Grid

The collection efficiency of the de-entrainment grid was determined from impaction theory. The basis for these calculations was the determination of net separation efficiency for each droplet size which depends on droplet terminal velocity and vapor superficial velocity. To obtain an overall collection efficiency, the staging effect of the tiers is accounted for in the calculations, i.e., there are 6 grid tiers per ft of grid. Therefore, even though the collection efficiency is low for a single tier, the

overall efficiency is greatly increased because of the staging effect of the multiple tiers that the vapor must pass through. Figure 4-7 illustrates the overall collection efficiency curves as a function of drop size for the two vacuum tower sizes that were studied in the Phase III-A test work. For comparative purposes, similar curves are shown in Figure 4-8 for 30 grid tiers which would typically be used in a commercial tower. As illustrated in Figures 4-7 and 4-8, the de-entrainment grid is effective in partially removing drops as small as 5 microns, depending upon vacuum tower diameter.

Conclusions

The results of the Phase III-B and Phase IV work thus far are:

- In Phase III-B (second half 1977), long-term fouling data on the vacuum tower de-entrainment internals could not be obtained due to bottoms level upsets in the tower. These upsets, aggravated by the inability of the material to drain because of the high velocity through the chimney tray, resulted in the severe solids accumulation and grid section fouling. The chimney tray was removed from the tower to avoid these problems in the future. Further grid fouling test work could not be performed, however, because the 1977 CLPP operations were curtailed in October.
- In the first half of 1978, the vacuum flash equipment of CLPP has operated with the "dry" de-entrainment grid in the 12-inch vacuum tower with no serious fouling problems. Two inspections of the vacuum tower and de-entrainment grid were made. Both inspections followed relatively short run lengths.
- The tangential inlet nozzle is effective in disengaging most (> 99.9%) of the 1000°F+ slurry drops from the vapor/liquid/solid vacuum tower feed.
- Based on feed inlet flow conditions and physical properties, drop size distributions were calculated at the tangential inlet. These calculated drop sizes were assumed to follow an upper limit log-normal probability distribution (7) with a standard deviation of 1.7, and were based on the Weber number criteria for determining the maximum stable drop size generated at the feed tangential inlet (3, 7). Drop sizes of entrained 1000°F+ material were calculated to be < 25 microns.
- Entrainment rates that were predicted using the entrainment removal model for the tangential inlet and grid were in good agreement with observed rates, i.e., an average deviation of ~25%.

Figure 4-7

DE-ENTRAINMENT GRID COLLECTION EFFICIENCY, %
VS
DROP DIAMETER, MICRONS

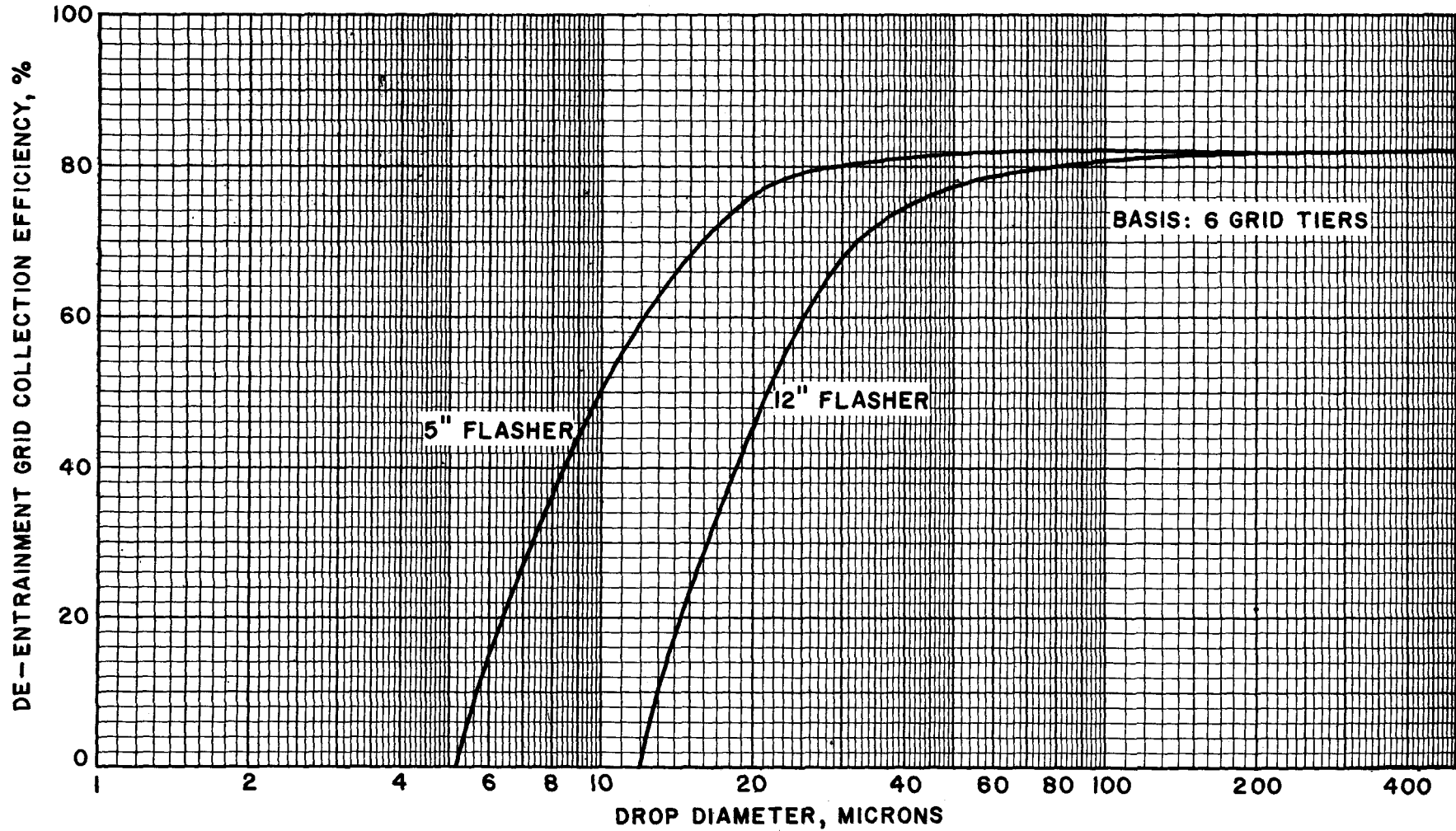
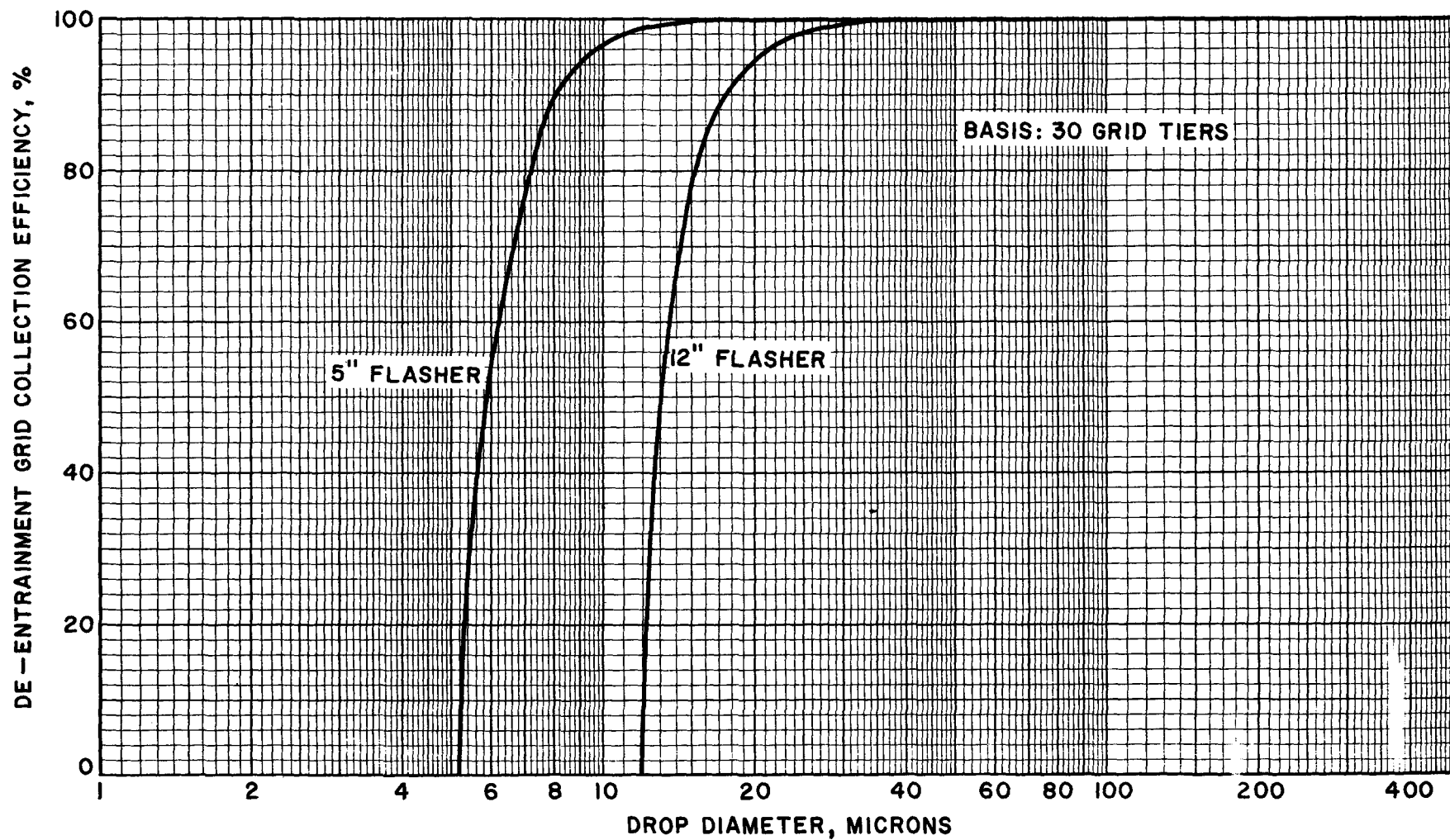


Figure 4-8

DE-ENTRAINMENT GRID COLLECTION EFFICIENCY, %
VS
DROP DIAMETER, MICRONS



Future Work

For the second half of 1978, the long-term fouling characteristics of the "dry" de-entrainment grid in the CLPP vacuum tower will continue to be monitored. Entrainment and grid fouling data with the "dry" grid (the most severe test for the de-entrainment device) in the 12-inch vacuum tower with a 1-inch transfer line will continue to be evaluated. This will determine if it will be necessary to plan alternative designs: first, a "washed" grid system similar to what would be used commercially and, secondly, if needed, shed baffle internals. If the above alternative designs are not necessary, another test would be performed with the "dry" grid in the 5-inch vacuum tower. Testing would then continue with the configuration that shows the best performance in terms of operability and de-entrainment capability until the year-end shutdown. If no fouling problems develop and time permits, grid fouling characteristics would be observed for a flash zone configuration that was modified to generate massive entrainment.

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4.6 Coal Liquefaction Reactor Model Studies

Summary

A solids withdrawal system for the two-foot diameter column was demonstrated using feed slurry concentrations up to 32 wt%. The system was proven effective in removing slurries containing oversized coal particles as large as 3.4 mm (6 mesh) and dense alumina particles with particle density of 3.8 g/cm³. Based on results of these simulation tests, a solids withdrawal system was designed for the ECLP liquefaction reactors.

The good distribution now existing within the modified plenum chamber of the ECLP cold model was further confirmed in tests carried out at the base case ECLP design gas and liquid velocities. Pressure drop measurements across the monitored distributor elements when operating with the 8 mesh minus coal slurry feed were within $\pm 10\%$.

Studies were carried out to determine the upper limit of operable velocities in the coal liquefaction reactors. Tests showed that the reactor would operate at velocities up to 2.2 x the ECLP design gas and liquid velocities without pulsing. However, increasing gas velocity accelerated bubble coalescence, and adversely affected contacting. Based on contacting limitations, the maximum operating velocity is about 1.8 x ECLP design rates.

Further experimental studies in the four and six inch diameter columns have begun to evaluate the effects of liquid viscosity on coal fluidization behavior. The comparison has been made using VARSOL* in place of heptane. VARSOL is twice as viscous as heptane but with roughly equivalent density and surface tension. Gas holdups and bed expansion were measured over the projected ECLP flow conditions for the VARSOL/N₂/coal system. Gas holdups were found insensitive to liquid viscosity, but bed expansions were higher with VARSOL than with heptane.

Oversized Ash Withdrawal Tests Completed

As reported previously in the Final Phase III-A Report, oversized (8-10 mesh) dense alumina particles were used to simulate large ash particles that may exist in the first ECLP liquefaction reactor. Tests on withdrawal of alumina particles from the column were carried out using the 1-1/4" diameter side nozzle and the 3/4" diameter "T" shape pipe inserted through the grid. Results on the side nozzle withdrawal tests were included in the Final Phase III-A Report (FE-2353-20).

Analysis of a simulation test of ash withdrawal through the 3/4" diameter pipe inserted into the grid was completed. At a pressure difference

* Trademark of Exxon Corporation.

of 33 psi between the column and the collecting drum, the slurry withdrawal rate was about 23% of the slurry feed rate to the column. As expected, this rate was lower than that previously measured with the 1-1/4" diameter nozzle at comparable conditions. Solids concentration in the withdrawn slurry was 75 wt%, and gas outflow rate was 5% of the feed gas. These data are consistent with that obtained previously using the side nozzle. Using the measured slurry withdrawal rate, ash removal from the first ECLP reactor might employ 8 minutes per 24 hour shift. Based on the results of this test program, a solids withdrawal system was designed for the ECLP liquefaction reactors.

Pressure Drop Measurements Confirmed Improved Distribution

Pressure taps were installed in the distributor for use in grid flow distribution studies to replace the transducers which have repeatedly failed during calibration. Final check of the flow distribution in the two foot diameter ECLP cold model was obtained in tests at the ECLP base case design gas and liquid velocities using a 32 wt% concentration of 8 mesh minus coal. Pressure drops across seven of the nineteen distributor elements were used to measure distributor flow uniformity. The pressure drops were within $\pm 10\%$ among the monitored elements, confirming the substantially improved distribution previously demonstrated with no coal in the heptane feed. The plenum chamber was modified during Phase III A to improve flow distribution.

Upper Limit of Operable Gas Velocity In Liquefaction Reactor Determined

Coal fluidization tests were carried out in the 24-inch diameter reactor model using heptane, 100 psig nitrogen, and 8 mesh minus coal in concentrations up to 45 wt%. Gas holdups, bed expansions, and distributor pressure drops were measured at gas and liquid velocities up to 2.2 x the projected ECLP design bases. Analysis of column pressure drop probe signals and visual observations of fluidization patterns indicated that the reactor would operate at the high gas and liquid velocities without pulsing. As shown in Figure 4-9, the measured gas holdups at 2.2 x ECLP design velocities ranged from 20-25 vol%, higher than the 10-18 vol% obtained at the projected ECLP design condition. However, large bubbles of 2-3 inches in diameter were observed near the top of the column, indicating substantial bubble coalescence at this high gas rate.

Based on a gas holdup of 22 vol% and using the observed bubble diameter of 3 inches, resistance to hydrogen transfer from treat gas to coal liquids was calculated to be significant in the liquefaction reactors at the high rates. As shown in Table 4-8, calculated hydrogen transfer rates using the modified Calderbank correlation were about 3 times the hydrogen consumption rate, whereas recommended practice is to maintain the hydrogen transfer rate at an order of magnitude higher than the consumption rate. At design

Figure 4-9
GAS HOLDUPS IN BUBBLE COLUMN

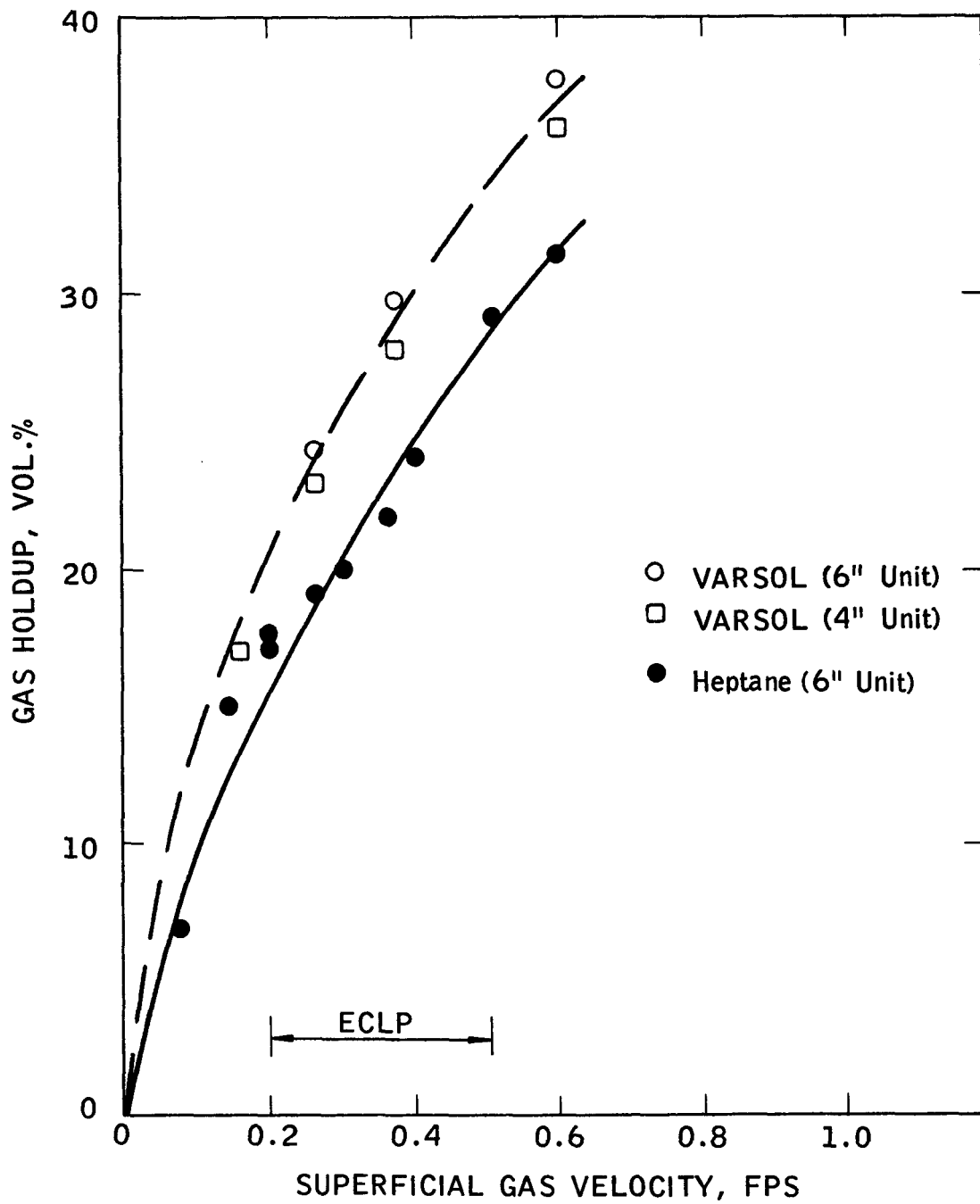


TABLE 4-8

HYDROGEN TRANSFER RATE COULD
BE LIMITING AT EXCESSIVE GAS VELOCITY

<u>(Gas Velocity</u> <u>ECLP Base Case (2)</u>	<u>Gas Holdup</u>	<u>Bubble Diameter</u>	<u>(Calc. H₂ Transfer Rate</u> ⁽¹⁾ <u>H₂ Consumption Rate</u>
	ϵ_g	d_B , in	
1.0	0.15	0.75	32
1.8	0.20	1 ⁽³⁾	10
2.2	0.22	3	3

(1) Based on mass transfer coefficient (CALDERBANK).

$$K_L = (0.31) \left[\frac{D_L \rho_L}{\mu_L} \right]^{-2/3} \left[\frac{(\rho_L - \rho_g) \mu_L g}{\rho_L^2} \right]^{1/3}$$

$$a = \frac{6\epsilon_g}{d_B}$$

where: a = gas/liquid interfacial area

K_L = mass transfer coefficient

D_L = diffusion coefficient

ρ_L, ρ_g = liquid/gas density

μ_L = liquid viscosity

ϵ_g = gas holdup

d_B = average gas bubble diameter

(2) ECLP base case gas velocity = 0.4 $\frac{\text{ft.}}{\text{sec.}}$

(3) Estimated bubble diameter

conditions with its observed 0.25 inch bubbles, hydrogen transfer was about 32 times consumption. Thus, increasing gas velocity will accelerate bubble coalescence and adversely affect contacting in the reactor. The upper limit of operable gas velocities is about 1.8 x ECLP design rate, based on ten times the consumption.

The measured bed expansion at 2.2 x ECLP design velocities was 165 vol% compared to 145 vol% obtained at design conditions. As reported previously (Final Phase IIIA Report), bed expansions were insensitive to column pressure and liquid velocity. Fluidization was satisfactory since the 30% bed expansion needed to keep all particles fluidized was achieved. Distributor pressure drop was 5 psi, as anticipated from the previously measured 2 psi at ECLP design velocities.

Gas Holdups Were Insensitive to Liquid Viscosity

Gas holdups using VARSOL instead of heptane were measured in the four and six-inch diameter columns operating without coal as bubble columns and in the six-inch column with 8 mesh minus coal at 25 wt% feed coal concentration. VARSOL has twice the viscosity of heptane, (0.8 vs. 0.4 cp), but equivalent density and surface tension. As shown in Figure 4-10, gas holdups were insensitive to liquid viscosity in slurry feed operation. Gas holdups with VARSOL alone were 17% higher than with heptane, showing a small liquid viscosity dependency (Figure 4-9). As reported previously in the Final Phase IIIA Report, these gas holdups were less than the 30 vol% upper limit of the ECLP design basis and should not limit liquid residence time in the reactor. In addition, the observed bubble diameter of 0.25 inch was comparable to that obtained with heptane. Hence, as previously discussed, hydrogen transfer rate from treat gas to coal liquids will not be a limiting factor.

Bed Expansions Increased With Increasing Liquid Viscosity

Bed expansions obtained with VARSOL/coal slurries in the six inch diameter column were compared with those previously obtained during heptane slurries (Figure 4-11). For the full range of projected ECLP design velocities, bed expansions were about 30% higher with VARSOL than with heptane. The higher, more favorable, bed expansion is believed attributable to the increased liquid viscosity, and consequently increased drag on the coal particles.

Future Work

The effects of particle size and size distribution, and feed coal concentration on fluidization behavior in VARSOL will be evaluated using the six inch diameter column, and the results compared with the previous studies with heptane. In addition, available data from EPRI's solids withdrawal and grid operability tests in Wilsonville will be evaluated for applicability to ECLP liquefaction reactors.

Figure 4-10
GAS HOLDUPS IN SLURRY FEED OPERATIONS

Legend:

■ Heptane/N₂ (6" And 24" Units, Slurry Feed = 14-41 Wt.%)

○ VARSOL/N₂ (6" Unit, Slurry Feed = 25 Wt.%)

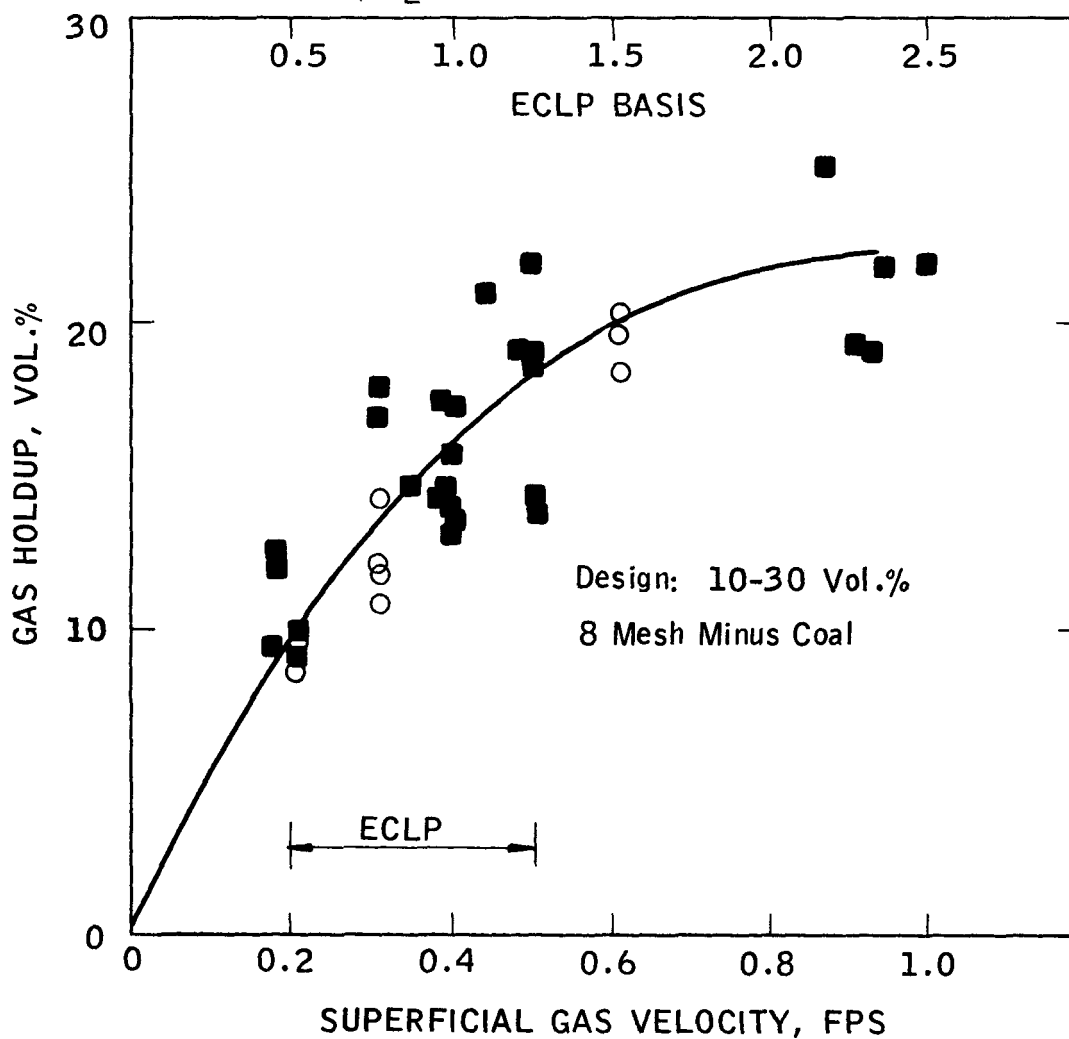
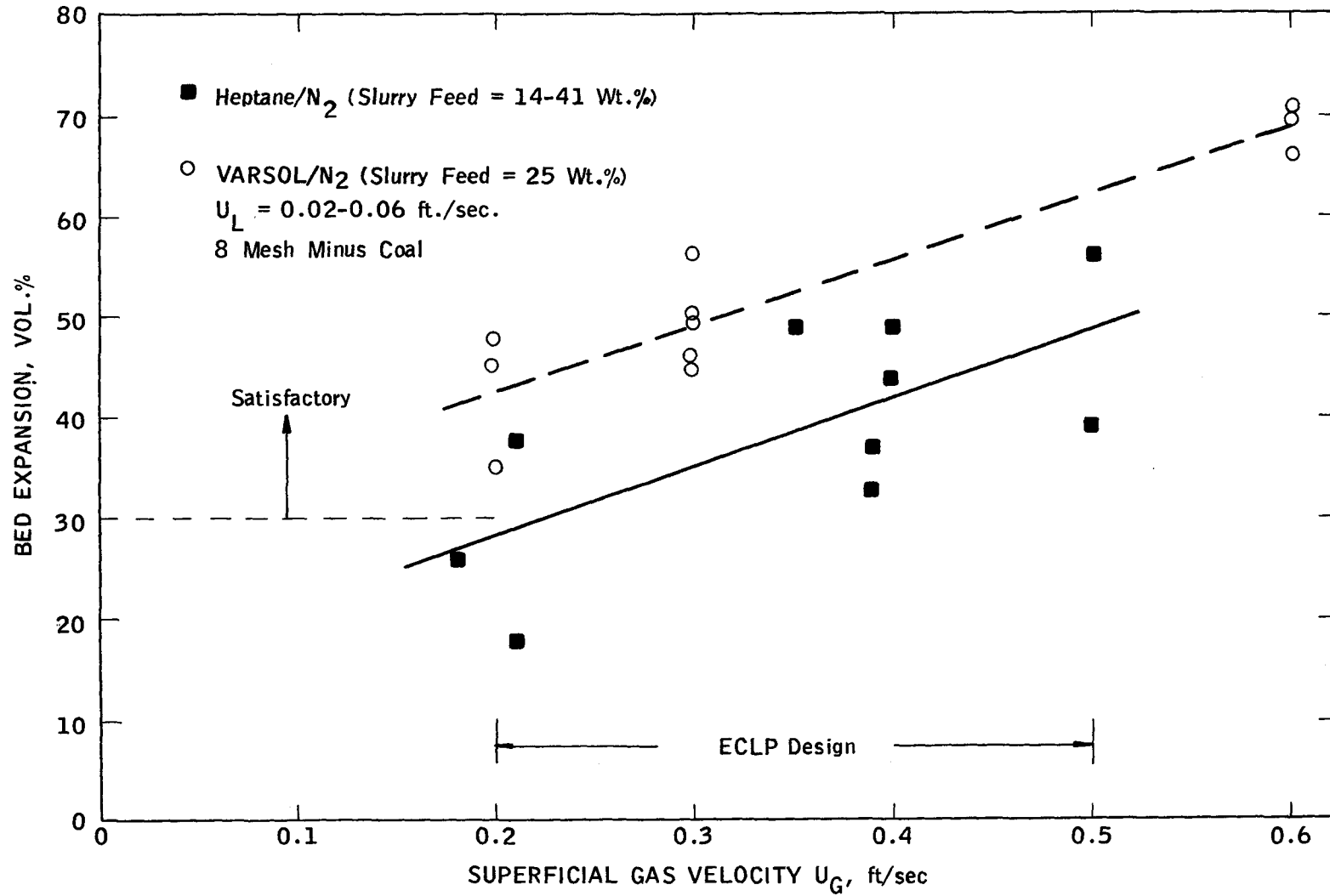


Figure 4-11

BED EXPANSIONS AT ECLP FLOW CONDITIONS



4.7 Rheology of Coal Solvent Slurries At Elevated Temperatures

Summary

Viscosities were measured for Illinois and Wyodak coal feed slurries to CLPP at temperatures up to 432°C (810°F). The effect of coal swelling on viscosity was observed at 321°C (610°F) for Illinois coal. This swelling temperature was apparently not time dependent. As expected, the Wyodak coal feed slurry exhibited no swelling characteristics.

Viscosities of the vacuum bottoms from CLPP were measured for Illinois and Wyodak coals as functions of shear rate and temperature. The Illinois coal slurries showed slight dilatant or shear thickening behavior at temperatures of 279°C (534°F) and 303°C (578°F) while shear thinning was observed at 322°C (617°F). The viscosity increased with increasing vacuum tower temperature, indicating higher solvent stripping. The Wyodak coal exhibited shear thinning behavior over the temperature range of 293°C (560°F) to 310°C (590°F) with the most pronounced effects at the lower temperatures. The viscosity of the Wyodak slurry increased with decreasing temperature.

Additional viscosity data on the CLPP low pressure separator slurry were obtained for Wyodak coal. The slurry was essentially Newtonian at shear rates below 1000 sec⁻¹, but dilatant behavior was observed at higher shear rates.

Rheological data were also collected on CLPP remelted vacuum bottoms containing 3.8 wt% 1000°F minus, CLPP bottoms with a boiling range of approximately 700 to 1000°F, and RCLU-2 bottoms containing 11.3 wt% 1000°F minus material. The CLPP vacuum bottoms data were fitted to a power law viscosity model. Analyses of the remaining data are underway. The information will be used to estimate transfer line pressure drops in the Commercial Study Design Update.

Rheological Properties of Illinois and Wyodak Coal Feed Slurries to CLPP Determined

Viscosities of Illinois coal slurries exiting from the CLPP preheater were determined at temperatures ranging from 260°C (500°F) to 432°C (810°F) with the viscometer in line with the CLPP preheater. Measurements were made at residence times of 2 and 2.8 minutes, using 30 mesh minus, 45 wt% coal slurry feed. These tests covered the range of projected coal residence times in the ECLP preheater. Coal swelling was observed at 321°C (610°F). The peak viscosity at this temperature was 28 mPa.s (cp). at a shear rate of 2125 sec⁻¹. The data are presented in Figure 4-12. Approximately the same swelling temperature was measured previously in a batch-heating system as described in the Final Phase IIIA Report. In this case, the measurement was made for a residence time of about four hours. Thus, swelling temperature for Illinois coal slurry appears to be dependent on the heating time as shown in Figure 4-13.

Figure 4-12

FEED SLURRY VISCOSITY FOR
SWELLING AND NON-SWELLING COAL

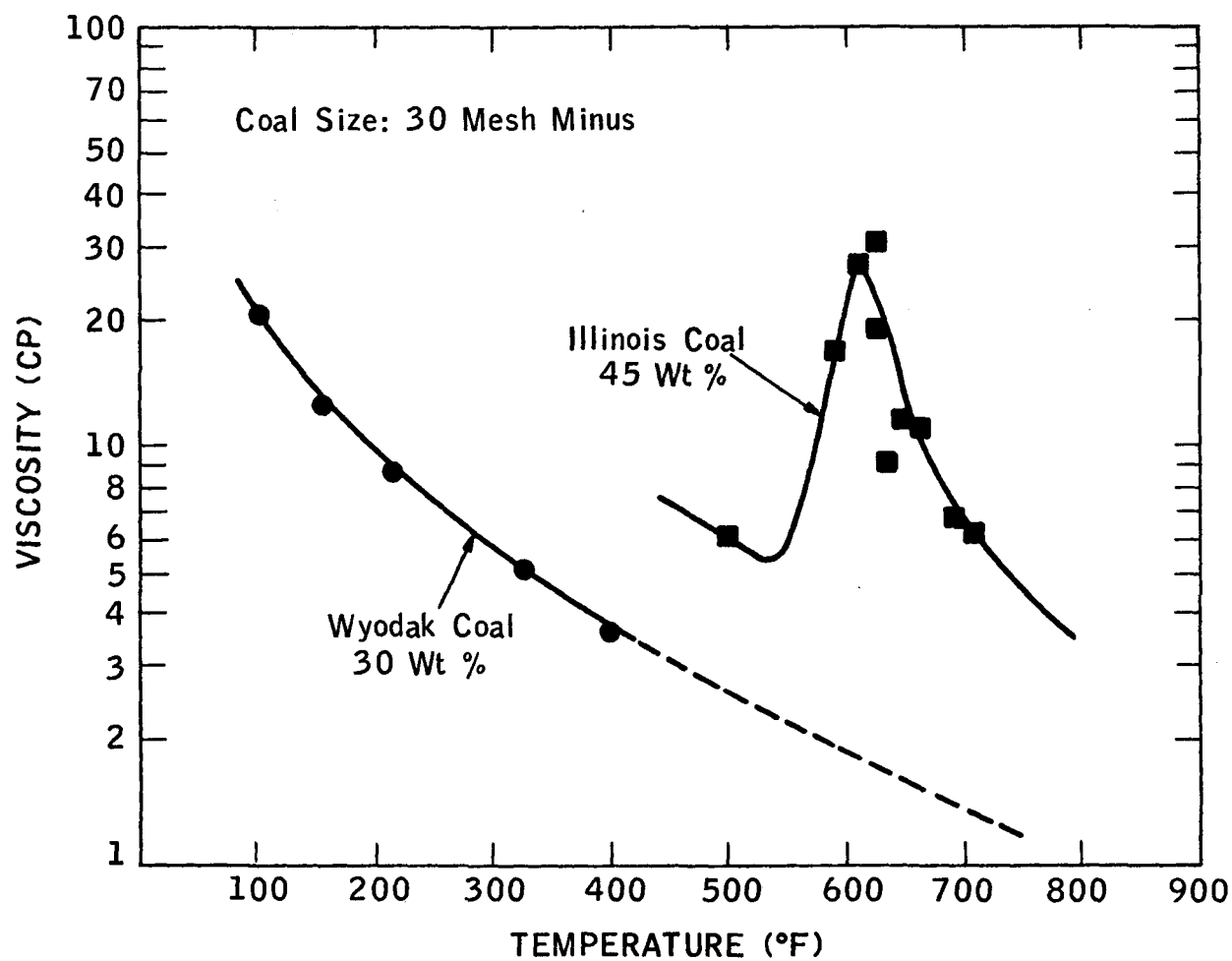
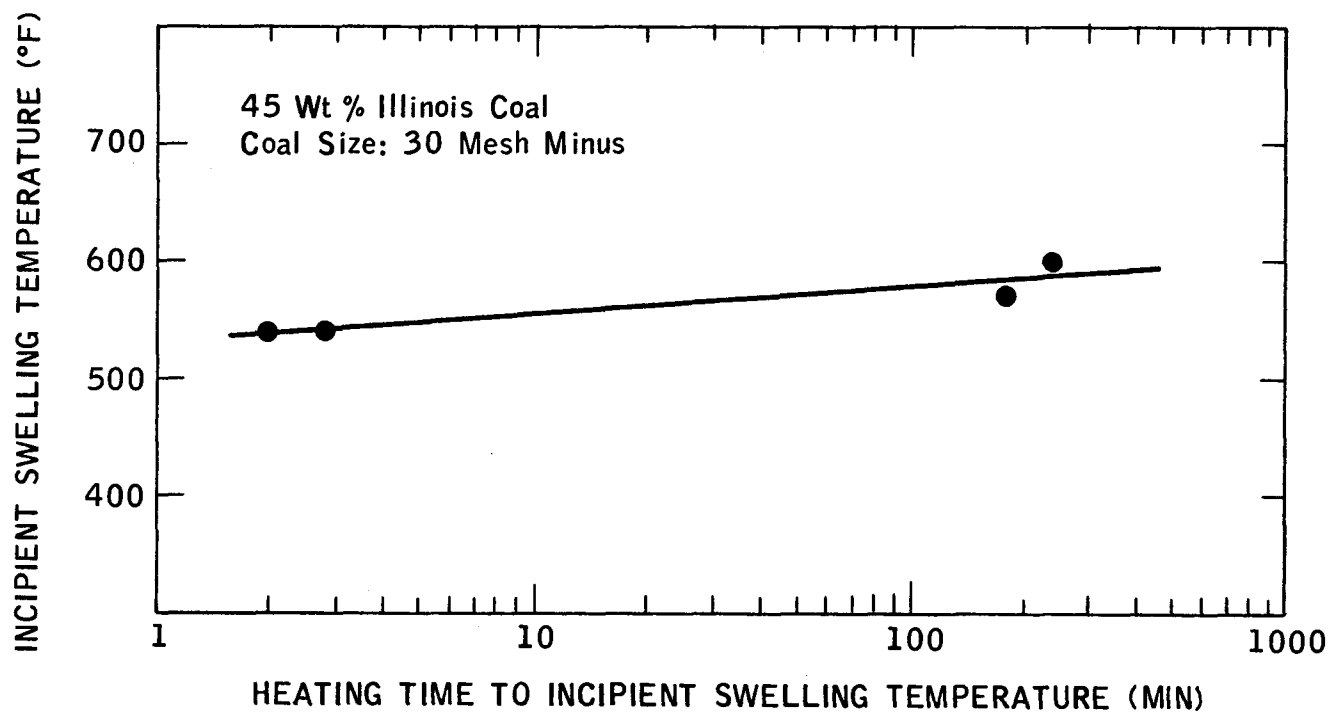


Figure 4-13

SWELLING TEMPERATURE SENSITIVITY TO
HEATING TIME FOR ILLINOIS COAL



Wyodak coal feed slurries (30 mesh minus, 45 wt% coal) were measured at temperatures ranging from 38°C (100°F) to 366°C (690°F). As expected, the Wyodak coal slurry exhibited no swelling characteristics. The viscosity continuously decreased with increasing temperature, 21 and 4 mPa.s at 38°C and 202°C (396°F), respectively. Above 202°C, the viscosity became too low for accurate measurement by the torque tube sensing element employed. The data are also shown in Figure 4-12.

Vacuum Bottoms Viscosities for Illinois and Wyodak Slurries

Viscosities of the vacuum bottoms from CLPP were measured for Illinois coal as a function of shear rate at 279°C (534°F), 303°C (578°F), and 322°C (612°F). The vacuum tower pressure was maintained at 0.7 psia. At 279°C, the slurry viscosity averaged 85 mPa.s, varying only slightly with shear rate (Figure 4-14). In contrast, when the tower temperature was raised to 322°C (612°F), the slurry viscosity increased significantly, indicating higher solvent stripping from the slurry in the tower. Analysis showed that the percentage of light material (1,000°F minus) in the slurries was 1.9 wt% at 322°C, 6.3 wt% at 303°C, and 12.0 wt% at 279°C. The slurry exhibited slight shear thinning behavior at the higher temperature. For example, the viscosity decreased from 600 mPa.s to 500 mPa.s when the shear rate was increased from about 500 to 2100 sec⁻¹.

Viscosities of Wyodak vacuum bottoms from CLPP were measured as a function of shear rate at temperatures of 293°C (560°F) and 310°C (590°F). The viscosity of the slurry increases with decreasing sample temperature. A drop of 17°C (30°F) from 310°C to 293°C increased the viscosity from 240 to 355 mPa.s at 2000 sec⁻¹; for the same temperature change the viscosity increases from 440 to 800 mPa.s at 50 sec⁻¹. The most pronounced shear thinning behavior was observed at the lower temperature. The data are shown in Figure 4-15.

Viscosities of Low Pressure Separator Slurry Measured

Low pressure separator slurry viscosities were determined as a function of shear rate for Wyodak coal at 291°C (550°F) and 297°C (566°F). The viscosity data were previously reported in a Phase IIIB Quarterly Report (July 1-September 30, 1977). The 1000°F minus contents of the samples were 68 wt% for the 291°C run and 40 wt% for the 297°C operation. At shear rates below 1000 sec⁻¹, the slurry appears to be Newtonian. However, dilatant behavior was observed at higher shear rates (i.e., viscosity increased with increasing shear rates). For example, the viscosity at 297°C increased from 13 to 18 mPa.s when the shear rate was increased from 1000 to 2000 sec⁻¹. These data are directionally consistent with previously reported viscosity of 38 mPa.s measured at a lower temperature, 280°C (540°F).

Figure 4-14

VACUUM BOTTOMS VISCOSITY FOR ILLINOIS COAL

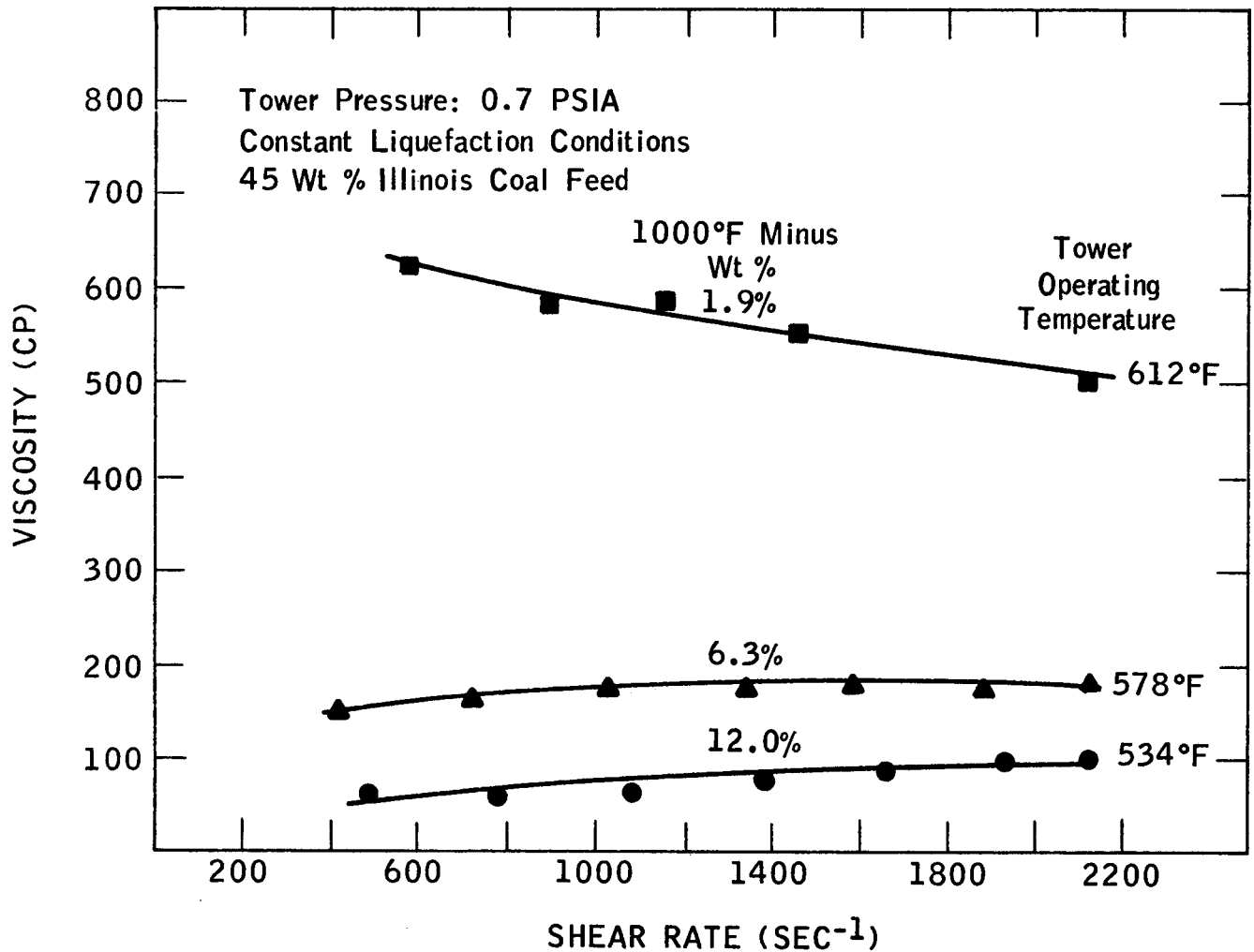
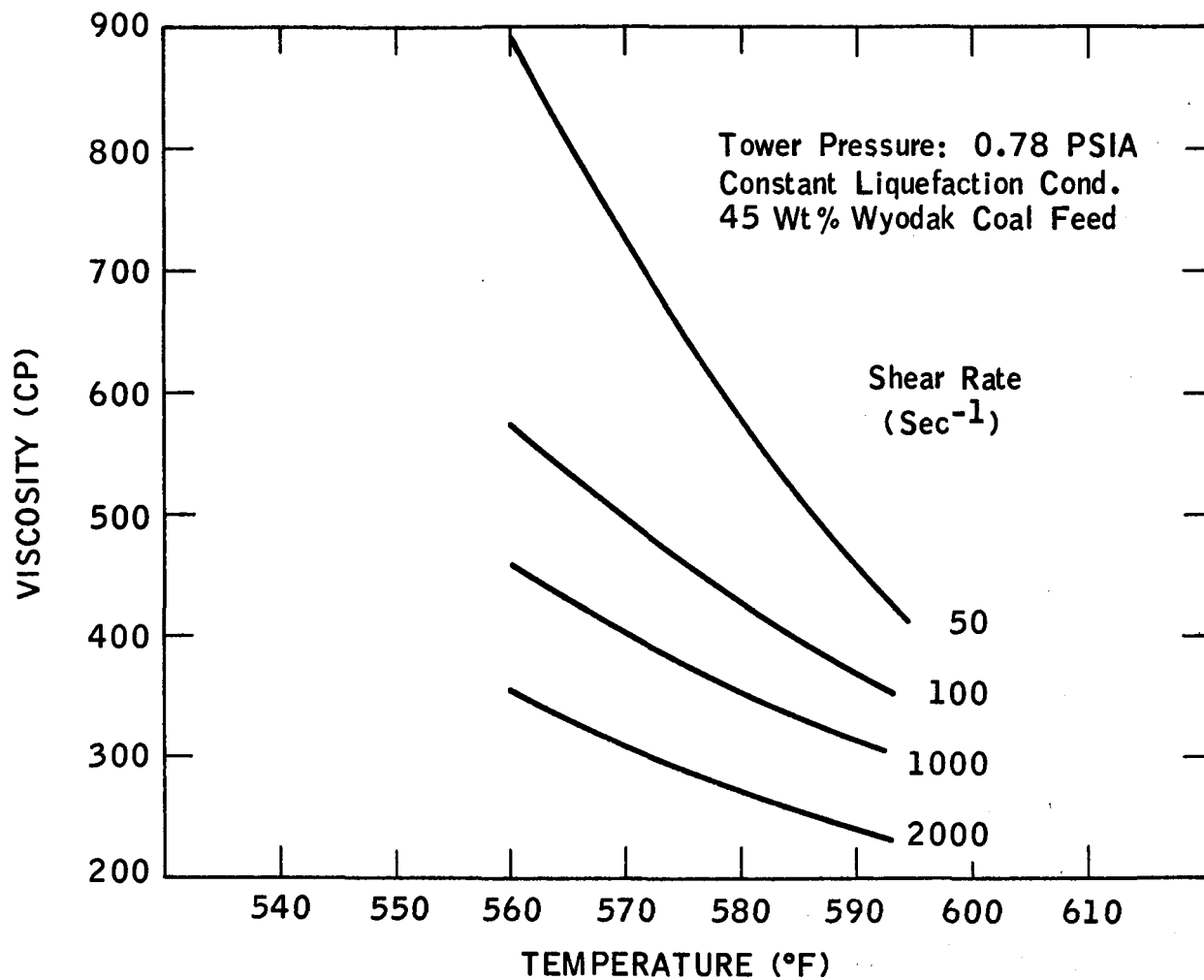


Figure 4-15

VACUUM BOTTOMS VISCOSITY FOR WYODAK COAL



Viscosity of Vacuum Bottoms As a Function of Sample Temperature

Data on vacuum bottoms slurries as a function of temperature are needed to develop pressure drop prediction techniques for EDS bottoms processing studies. The viscosity of a sample of remelted CLPP vacuum bottoms containing 3.8 wt% 1000°F minus material was measured as a function of shear rate at 260°C (500°F), 321°C (610°F) and 379°C (715°F). The data, shown in Figure 4-16, were fitted to a power law model of the form, $\tau = k \dot{\gamma}^n$ where τ is the shear stress, k is defined as the consistency index, $\dot{\gamma}$ is the shear rate, and n is the power law exponent. The resulting values of the k and n parameters are shown as functions of temperature in Figures 4-17 and 4-18. Using this power law model, pressure drop prediction equations were developed.

Additional rheological data were collected on remelted CLPP bottoms with a boiling range of 700 to 1000°F and on RCLU-2 bottoms containing 11.3 wt% 1000°F minus material. For the CLPP sample, the measurements were made at 138°C (281°F) and 211°C (412°F) as a function of shear rate. The data for the RCLU sample were obtained at 259°C (498°F) and 368°C (695°F). The data from both runs are being analyzed.

Future Work

Viscosity measurements on Illinois and Wyodak coal feed slurries will be completed. The effect of solids concentration on viscosity will be evaluated at temperatures up to 427°C (800°F) and concentrations up to 45 wt% coal. The data on feed, low pressure separator, and vacuum bottoms slurries will be correlated and put in a form suitable for design use.

4.8 Slurry Preheater Development

The objective of this program is to investigate the coking tendency of the CLPP slurry preheater feed streams to gain a qualitative understanding of preheater coking as it is affected by important process and design parameters. These parameters include residence time, film temperatures, bulk temperatures, percent vaporization, and feed composition. Operating data has been obtained using the sectionalized slurry preheaters specifically designed and installed for this purpose. These data will be used for more accurately predicting the performance of, and for evaluating data from, the ECLP slurry preheater.

Summary

During Phases IIIB and IV, additional testing has been done on both Illinois No. 6 and Wyodak coals at conditions more severe than those tested during Phase IIIA. These conditions include a slightly higher severity with a much longer run length for Illinois coal and greater severity

Figure 4-16

VISCOSITY SHEAR RATE DEPENDENCE FOR REMELTED CLPP VACUUM BOTTOMS

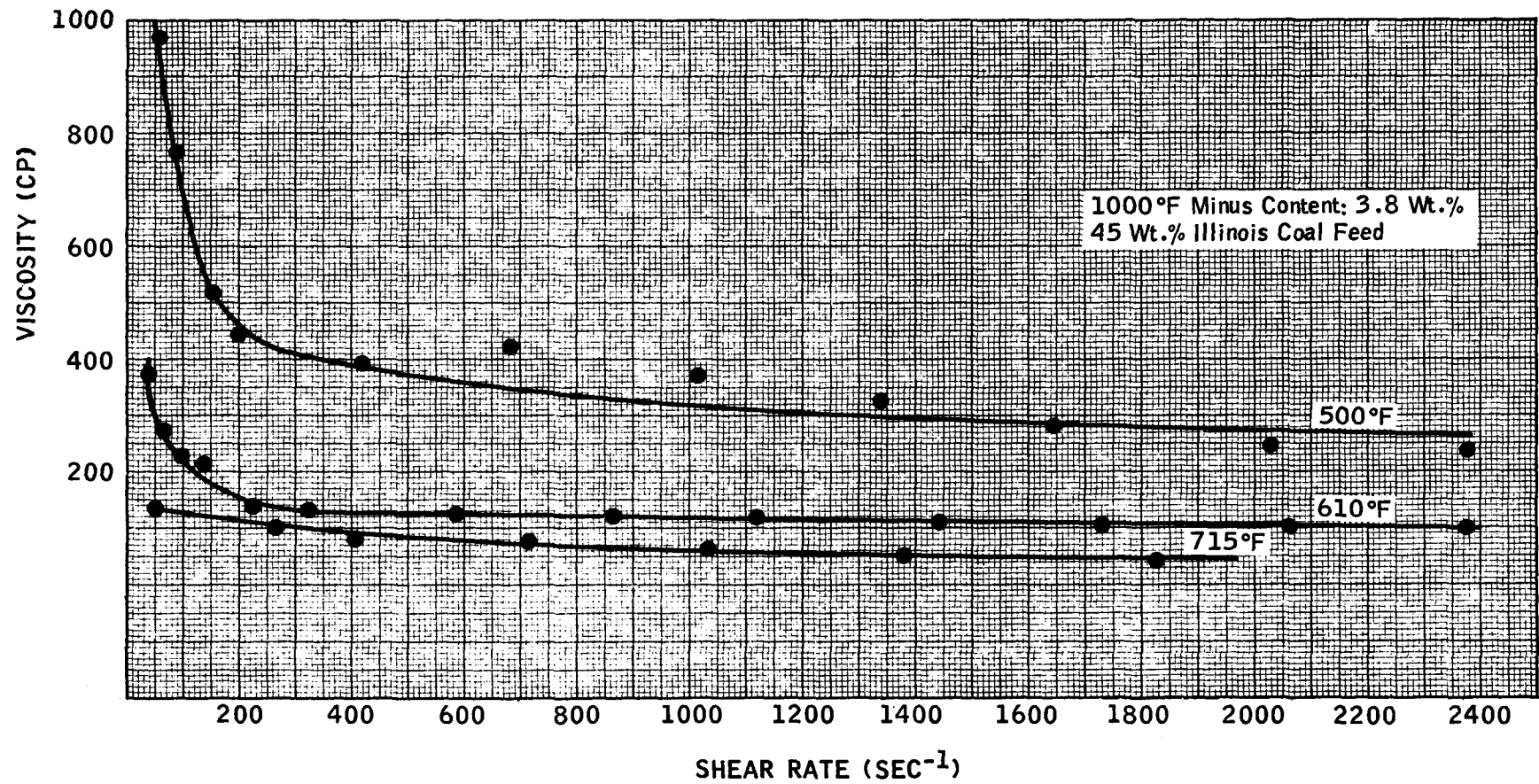


Figure 4-17
 CONSISTENCY INDEX
 TEMPERATURE DEPENDENCE
 FOR REMELTED CLPP VACUUM BOTTOMS

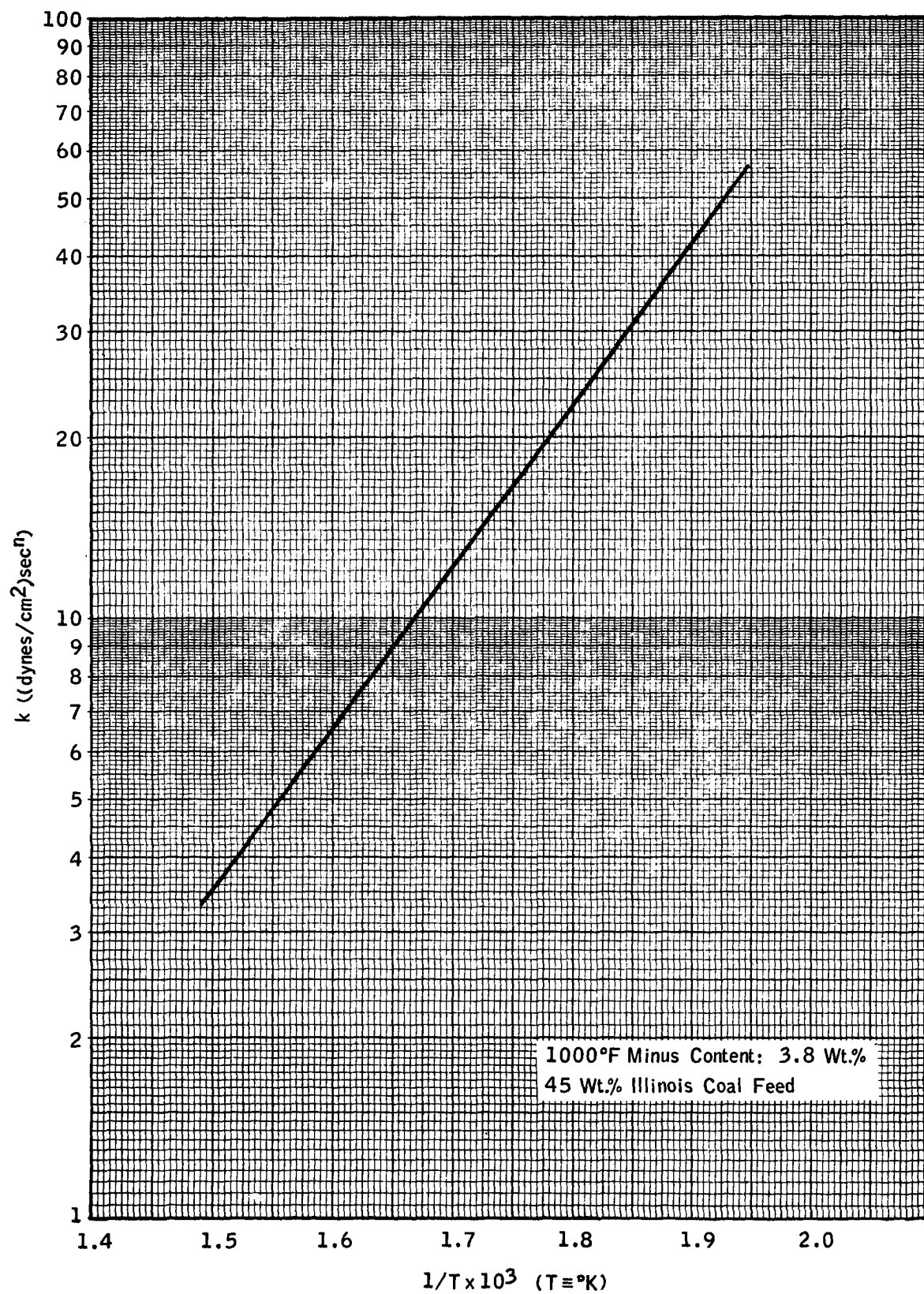
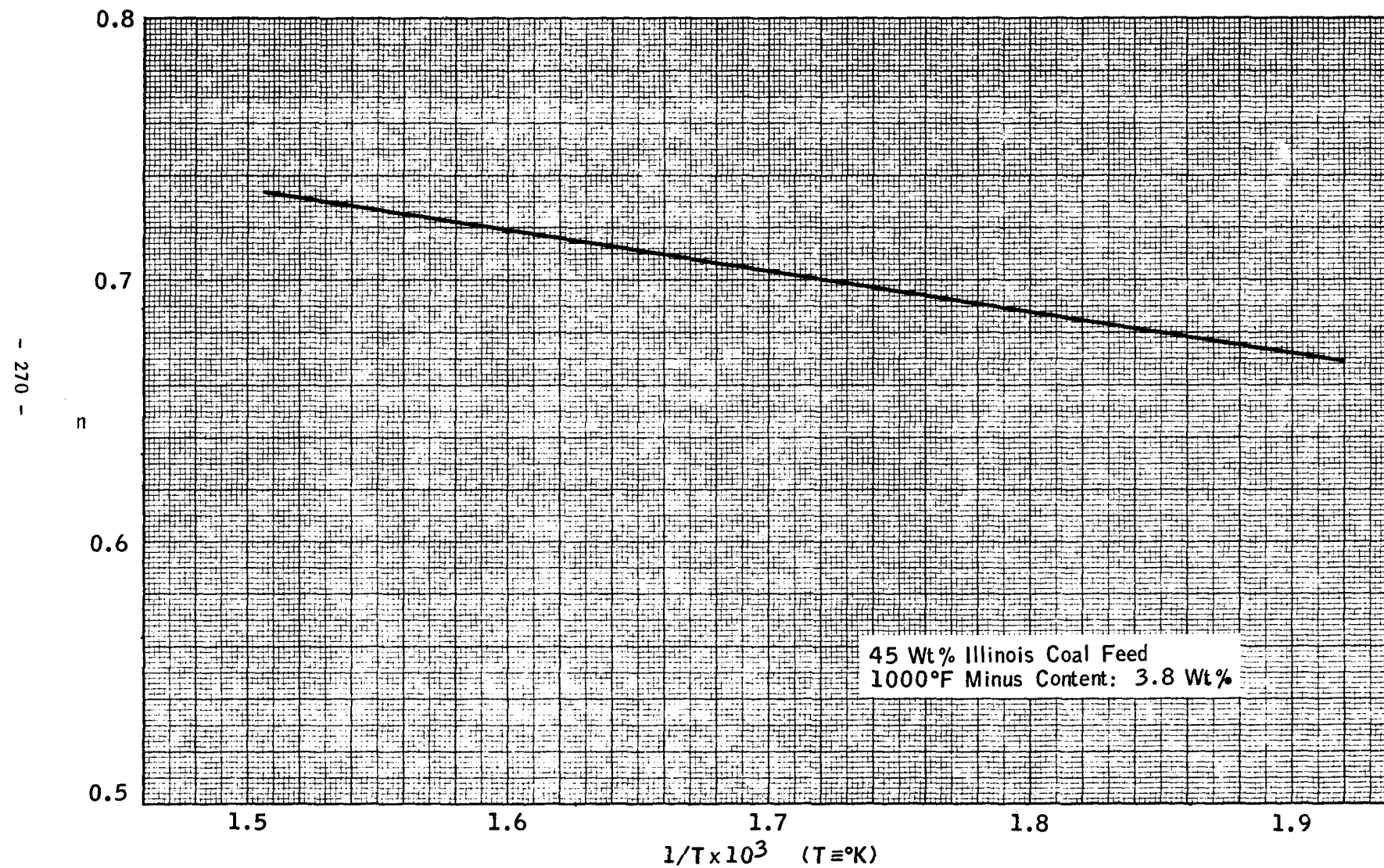


Figure 4-18

POWER LAW EXPONENT
TEMPERATURE DEPENDENCE
FOR REMELTED CLPP VACUUM BOTTOMS



at higher temperatures for Wyodak coal. Phases IIIB and IV tests were all conducted on mixed phase operation which was determined during the previous testing to be the preferred alternative for heating the slurry.

No coking was detected during these tests with the slurry preheater operating at conditions that either meet or exceed the design conditions for the ECLP slurry preheater. These include lower solvent to coal ratio (for Illinois coal only), an equal or greater severity, equal or greater vaporization, and an equivalent bulk outlet temperature. Test results for the CLPP slurry preheater are summarized in Table 4-9. Several earlier tests in Phases IIIA and ECLP design conditions are also presented for comparison.

The pilot plant has now operated satisfactorily with mixed phase heating of the slurry feed for over 3500 hours with no major problems that can be attributed to the slurry preheaters. This includes over 2100 hours on Illinois coal and over 1400 hours on Wyodak coal. The results of testing in Phases IIIB and IV continue to support our expectation that rapid coking will not occur in the ECLP preheaters at design conditions.

Slurry Preheating Test System

The slurry preheating test system has been basically the same system used for feed preheating for the CLPP liquefaction reactors during Phase IIIA. A detailed description and sketch of the system were given in the Phase IIIA Final Report (FE-2353-20).

During testing for Phases IIIB and IV, only a portion of the sectionalized preheater was used and the coil dimensions were modified depending on the specific test requirements. The general preheater (E/H-2) was used in series with two of the five sectionalized preheaters (E/H-3). The E/H-2 preheater was equipped with a 150 foot length of 5/16 inch O.D., 0.049 inch thick stainless steel tubing. To permit higher residence time and resulting greater severity for Phase IIIB testing, the sectionalized preheater coils were changed to a larger diameter and longer length tubing. For this testing, each E/H-3 preheater was equipped with a 100 foot length of 1/2 inch O.D., 0.065 inch thick stainless steel tubing. For Phase IV testing, the two E/H-3 preheaters had the normal 50 foot length of 3/8 inch O.D., 0.065 inch thick stainless steel tubing.

Phase IIIB Illinois No. 6 Coal

During the latter part of September and early October of 1977, the CLPP slurry preheater was tested on mixed phase operation with Illinois coal at a high severity for an extended time. For two nearly identical tests, the preheater operated at a severity of 343 equivalent seconds for a total of 257 hours with no coking being detected. To achieve this severity, the slurry mixture was heated to 734°F in E/H-2 and then to 830°F in two successive E/H-3 preheaters. The results of these tests are listed in Table 4-9 as Test 5/6.

TABLE 4-9

COMPARISON OF CLPP SLURRY PREHEATING TESTS

	<u>ECLP</u>	<u>CLPP Phase IIIA</u>		<u>CLPP Phase IIIB</u>	<u>CLPP Phase IV</u>
	<u>Mix. Phase</u>	<u>Mix. Phase</u> (Test 3)	<u>Mix. Phase</u> (Test 4)	<u>Mix. Phase</u> (Test 5/6)	<u>Mix. Phase</u> (Test 7)
Test Date	-	4/11/77	5/9-24/77	9/13-10/4/77	3/29-4/3/78
Coil Outlet Temp. (°F)	840 to 850	885	826	842	847
Coil Outlet Press. (psig)	1950	1640	1620	1630	1720
Coal Type	Ill. #6	Ill. #6	Wyodak	Ill. #6	Wyodak
Dry Coal in Slurry (wt. % fraction)	0.40	0.44	0.37	0.45	0.36
Treat Gas Rate (wt. % on dry coal)	18.5	5.10 to 5.92	6.85	6.11	5.33
H ₂ in Treat Gas (wt. %)	25	56	56	56	56
H ₂ O in Coal (wt. % on dry coal)	4.0	0.5	3.5	0.5	3.5
Film Temp. (°F)	875-885	900	829	844	849
Severity*	150	295	25	343	160
Sandbath Temp. (°F)	-	912	831	845	851
Solvent Vaporized, Outlet (wt. %)	60 to 64	75	54	68	74
Total Run Length (hrs.)		22	387	257 Combined	106
Comment		No Coke	No Coke	No Coke	No Coke

*Equivalent seconds at 750°F

These Phase IIIB tests were an important part of the slurry preheater testing program because they provided increased confidence in conclusions that had been drawn in the Phase IIIA Final Report. The high severity test run at similar conditions during Phase IIIA was terminated after only a brief time of 22 hours. With an equivalent residence time about 15% greater, the Phase IIIB test ran for a considerably longer time (over 10 times longer). Compared to the ECLP design case, the CLPP preheater for the Phase IIIB tests operated without coking at the design outlet temperature of 840°F, slightly higher solvent vaporization, lower solvent to coal ratio (1.22 versus design of 1.5), and an equivalent residence time severity twice as high. These facts continued to support the earlier expectation that rapid coking would not occur in the ECLP slurry preheater at design conditions.

Phase IV Wyodak Coal

From March through June of this year, the CLPP slurry preheater has operated with mixed phase on Wyodak coal at a variety of conditions. No coking was detected during an operating period totalling over 700 hours. Some of the runs were of a short duration, but this operating period also includes several runs of 4 to 6 days.

For this Phase IV period, the operating conditions were both more and less severe than those tested for Wyodak during Phase IIIA. One run, which lasted for over 100 hours, at more severe conditions is listed in Table 4-9 as Test 7. Compared with the Phase IIIA test at nearly the same solvent to coal ratio of 1.7, the significant test conditions are a higher coil outlet temperature of 847°F versus 826°F and a higher severity of 150 versus 25 equivalent seconds. The 6-fold increase in severity was produced by heating the slurry mixture to approximately 837°F in E/H-2, 847°F in E/H-3A, and then maintaining the feed at a nominal 845°F in E/H-3B. As indicated above, no coking was detected. One of the goals set for testing of the slurry preheater on Wyodak for 1978 was operation at 840°F or above. With this successful run at 847°F, this goal has been achieved.

The Phase IV mixed phase test on Wyodak was conducted at essentially ECLP design conditions: duplicate condition for coil outlet temperature, solvent vaporization, and equivalent seconds severity. However, the 1.8 solvent-to-coal ratio was higher than the design of 1.5. The results are encouraging. They indicate that rapid coking may not occur in the ECLP slurry preheater for Wyodak coal as well as Illinois coal. However, additional testing at more severe conditions are still required to determine if the same range of operating conditions that have already been satisfactorily tested for Illinois can be achieved for Wyodak.

Evaluation of Experience From SRC Slurry Preheaters

During Phases IIIB and IV, some time was spent investigating the slurry preheater performance in existing coal liquefaction pilot plants using the Solvent Refined Coal (SRC) process. This effort was concentrated

on the SRC pilot plant operation by Southern Services, Inc. in Wilsonville, Alabama, and PAMCO's pilot plant in Fort Lewis, Washington.

The review of Southern Services was based on their quarterly and semi-annual Technical Progress Reports which cover three years of operation. They reported that preheater coking had occurred at various times, but that it had not limited pilot plant operation. The available reports are not sufficiently definitive with respect to precise conditions when coking occurred to permit any detailed analysis. Therefore, direct discussions with Southern Services will be required to draw conclusions from the published information.

PAMCO's recently issued summary report for their SRC pilot plant has been briefly reviewed to determine their experience with the slurry preheater. This report covers startup and operation of the pilot plant between June, 1974 and April, 1977. No coking was reported during this period, with the preheater operating with a nominal coal concentration in the slurry of 39%, a coil outlet temperature between 750°F and 790°F, and mixed phase operation. Based on data from CLPP, coking would not have been expected at these rather mild conditions.

Future Considerations

During the remainder of 1978, testing will concentrate on investigating mixed phase operation in the slurry preheater for Wyodak coal at more severe conditions than those tested in Phase IIIA and Phase IV to date. Specific goals are to operate with a solvent to coal ratio as low as 1.2 and a severity as high as 300 equivalent seconds.

Evaluation of the operating experience from the SRC pilot plants will continue. Arrangements are being made to hold direct discussions with Southern Services concerning the coking experience with the slurry preheater in Wilsonville, Alabama.

4.9 Vacuum Pipestill Preheater Development

Coking and coil erosion have been considered potential operating problems in the EDS Vacuum Pipestill Preheater. The objective of this program was to determine the probable severity of this problem by evaluating operating data from the CLPP vacuum pipestill preheater.

High Severity Tests Demonstrate Operability of ECLP VPS Preheater

In the 4th Quarter of 1977, high severity tests were conducted on the vacuum pipestill with CLPP operating on Illinois coal. During these tests, the preheater was operated at successive outlet temperature plateaus of 695, 750, and 800°F. No coking tendency or operating problems were observed. The operation at 800°F represented the maximum that can be achieved in the preheater with the existing CLPP vacuum pipestill.

For these high severity tests, the preheater outlet and maximum film temperatures were 60 to 70°F higher than those anticipated in the ECLP slurry preheater for base case operation. Based on the satisfactory operation that was achieved in the CLPP preheater at these more severe conditions, it was concluded that reasonable run lengths should be expected from the ECLP preheater while operating at base case conditions.

Future Considerations

At the end of Phase IIIB, this program was discontinued. During the first six months of Phase IV with CLPP operating on Wyodak coal, no vacuum preheater coking or erosion problems have been reported, indicating no need to reactivate this program.

4.10 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 being a combination of two cases which are: (1) a concentration maldistribution, and (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

This program was started in Phase IIIB and has continued in 1978 as part of Phase IV. During Phase IIIB, the test unit was designed and construction was initiated. By the end of the 2nd Quarter of this year, construction of the manifold test section according to the original design was completed. Checkout and shakedown of the test unit has been initiated, and several necessary modifications have been determined. Testing of the slurry manifold will begin early in the 3rd Quarter of 1978, following completion of the modifications and final shakedown.

Description of Slurry Manifold Test Unit

A schematic diagram of the slurry distributor manifold test unit is shown in Figure 4-19. The test facilities consist of a pipe-loop with a mixing/holdup tank, a positive displacement pump, and the manifold distributor, four parallel passes, and a collection manifold. A density analyzer is included to monitor the coal concentration of the slurry being pumped to the manifold. Each parallel pass has a control valve and venturi flow meter for balancing the flow among the passes, as would be done in a commercial unit.

Each pass has a sample collection section which consists of a 54-inch long vertical piece of 3/4 inch nominal diameter schedule 80 pipe located between two fast-acting pneumatic valves. The valves in all the passes are linked together so that samples can be simultaneously trapped for each test section. The resulting samples are collected for analysis by draining the test section.

The sample sections are each provided with a solvent purge to replace the sample when it is removed. Originally, the samples were to be collected by opening an air vent at the top of the sample section and allowing gravity to drain the sample. However, the accumulation of air in the section as the sample was removed could either cause an error in a subsequent sample or become trapped in the recirculated slurry and create a three-phase flow in the manifold.

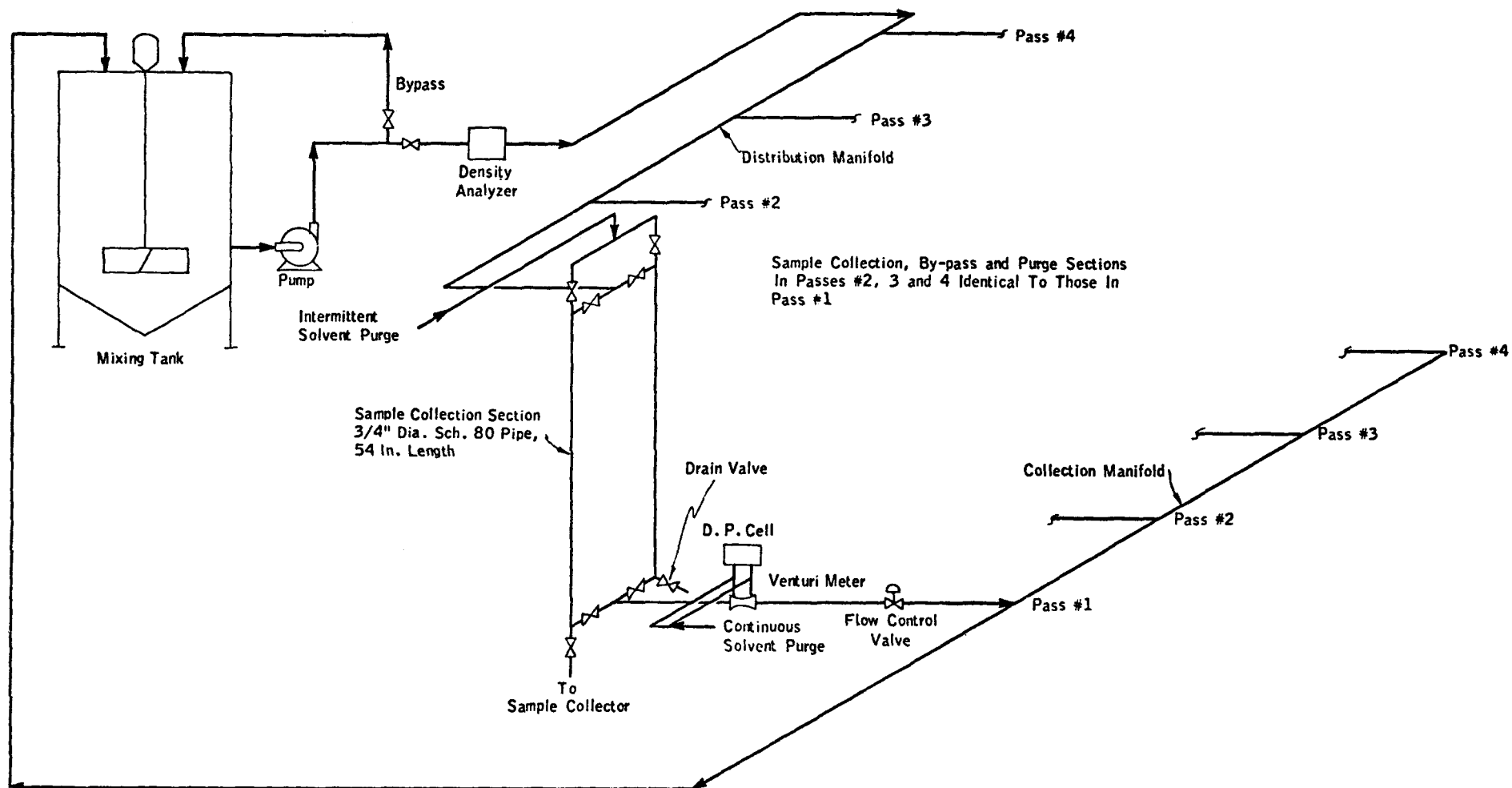
A by-pass was provided around each sample section to permit continuous flow through the test unit while the samples are trapped and collected. The by-pass lines are also provided with solvent purge to prevent the coal from settling in those lines and creating a blockage.

Outline of Test Program

During the test program, it is planned to use the manifold test unit to investigate the effect of a number of geometric variables on coal maldistribution in the parallel passes. These geometric variables include manifold orientation (horizontal vs. vertical), manifold "T" junction configurations, number of parallel laterals, and spacing between the laterals. Also during the test, the effect of variations in process flow conditions will be evaluated including solvent to coal ratio, particle size distribution, and slurry velocity.

A horizontal manifold will be the first configuration tested since it would be the most common commercial orientation. While testing the horizontal manifold, the location (top, side, or bottom) of the takeoffs will also be evaluated.

Figure 4-19
SLURRY DISTRIBUTOR MANIFOLD TEST FACILITIES
FLOW PLAN



The tests to evaluate the effect of the manifold distributor will consist of simultaneously trapping the equal volume samples in the parallel pass sample sections. The coal concentration in each sample will be determined by separating the coal from the solvent (diesel oil), drying the coal and then weighing it. The samples will then be sieved to determine the particle distribution of each sample.

Future Considerations

The extent of the test program will depend on the initial results. If no maldistribution is detected during the tests on the horizontal manifold, it may be possible to conclude this program by the end of 1978. However, it is presently anticipated that testing of the manifold distributor will have to be continued in 1979 to evaluate fully the effects of the variables.

4.11 Vacuum Bottoms - Commercial Plant Solidification

Work on the solidification of the EDS liquefaction plant vacuum bottoms was terminated in December, 1977 because revisions to the commercial EDS plant design study eliminated any requirement for solidification and solid storage of the material. A final report of progress through 1977 was presented in the EDS Phase IIIB Quarterly Report for the period October 1-December 31, 1977 (FE-2893-7).

Since preparation of that report, a previously requested proposal from a vendor of flaking equipment arrived. The vendor recommended 23 double drum flakers, each five feet in diameter by 12 feet long, fully nitrogen inerted, carbon steel, to handle a 433,000 pound per hour stream of EDS vacuum bottoms.

Work on this study was halted prior to forming conclusions on the feasibility and costs of the several potential methods for vacuum bottoms solidification.

ENGINEERING RESEARCH AND DEVELOPMENT

ENGINEERING TECHNOLOGY DEVELOPMENT

5. Bottoms Processing Engineering Technology

5.1 Improve Quality of Coker Scrubber Liquid

Summary

A base case was developed defining scrubber liquid properties and solids concentration. The base case was used for evaluating the technical feasibility and economic attractiveness of various fines removal techniques. A level of 0.15 wt% solids in EDS fuel oil was also selected as a target. Three options were identified for reducing the particulate level in EDS FLEXICOKING Unit scrubber liquid. These options are multiple hydroclone stages with further reduction in solids concentrations by blending with low sulfur fuel oil from other sources in the EDS process, a hydroclone first stage followed by a nozzle disc centrifuge second stage, and a rotary precoat filter. Based on an economic evaluation and operability considerations, the multiple hydroclone stages option is the preferred technique for particulate removal.

Sensitivity studies of the hydroclone performance show this technique is capable of reducing the solids content to the target level with moderate variation in liquid and solid properties. However, large variations in viscosity, density difference, or particle size from base case values could make the hydroclones unsuitable. Preliminary data on samples of unfiltered LSCU stripper bottoms indicate the viscosity may be higher than previously estimated. Work is underway to verify these data and to evaluate the possibility of blending scrubber liquid with 1000°F minus material to reduce the viscosity. Pending the outcome of this work, hydroclone testing is planned in the laboratory fines removal loop at elevated temperatures. Vendor tests are also scheduled to evaluate a vertical leaf pressure filter with coal liquids. This filter is reported to show unanticipated performance in removing solids from coal tar and tar pitch at elevated temperatures.

Base Case Developed for Evaluating Fines Removal Equipment

A base case was developed for evaluating the technical feasibility and economic attractiveness of various fines removal techniques. The bases of the study case were characterizations of petroleum coker liquids and preliminary viscosity data on samples of coal liquid from the Continuous Stirred Coking Unit (CSCU). The base case parameters are summarized in Table 5-1.

TABLE 5-1

BASE CASE CONDITIONS FOR
EVALUATING FINES REMOVAL EQUIPMENT

Solids Concentration:	4 wt.%
Liquid viscosity at 700°F:	1.4 cp
Liquid density:	0.983 gm/cm ³
Solid density:	1.640 g/cm ³
Particle size:	
10 wt.% less than	4 μm
20 wt.% less than	7 μm
50 wt.% less than	22 μm
100 wt.% less than	81 μm

Three Options for Particulate Removal

Three options were developed for reducing the particulate level in the scrubber liquid from the FLEXICOKING unit to acceptable levels. These options were multiple hydroclone stages with further reduction in solids concentrations by blending with low sulfur fuel oil from other sources in the EDS process; a hydroclone first stage followed by a nozzle disc centrifuge second stage; and a rotary precoat filter. Estimates of liquid losses, equipment size or number of stages, and particulate removal efficiencies were developed for all three options and used in economic evaluations to select the preferred technique.

Other fines removal techniques were found to be currently unacceptable for reducing the EDS scrubber liquid particulate concentration. These devices were electrofilters, pressure filters other than rotary precoat systems, high gradient magnetic separators, combinations of solid bowl and nozzle disc centrifuges, and antisolvent precipitation. The general reasons for rejecting these systems at this time were operability concerns at high temperatures, and low rates or low capacities. The specific reasons for rejecting each device are summarized in Table 5-2.

Multiple Hydroclone Stages Preferred Technique for Fines Removal

The technique preferred for removing particulates from the EDS FLEXICOKING Unit scrubber liquid is multiple hydroclone stages. Further reduction in the solids concentration to the target level is required but can be accomplished by blending with low sulfur fuel oil from other sources in the EDS process. Specifically, to reach a target of 0.15 wt%, three stages of hydroclones operating with a 15 wt% underflow rate appears most attractive, based on an economic evaluation and operability considerations. The other two options showed a slight economic advantage over hydroclones. However, potential operating problems and the lack of commercial experience with these devices at elevated temperatures made these options less attractive.

Calculations were also made to assess the sensitivity of hydroclone performance to physical properties of the liquid and solids. Scrubber liquid viscosity, solid-liquid density difference, particle size distribution, and flow rate per hydroclone were independently varied from the base case conditions to less optimistic values. The 0.15 wt% target value could still be achieved but with higher underflow liquid rates. For example, a viscosity increase of 14% from 1.4 to 1.6 cp would require a 17% increase in underflow, from 15 to 17.4 wt% to reach the target solids level. Further increases in viscosity to 2.0 cp would require 23.5 wt% underflow to reach the target level. At a viscosity above ~5cp, the hydroclone separators might no longer be suitable because excessive underflow might be required. Of course, some blending with 1000°F minus material could be used to adjust final viscosity.

TABLE 5-2

FINES REMOVAL DEVICES PRESENTLY REJECTED FOR
EDS FLEXICOKING UNIT SCRUBBER LIQUID SERVICE

<u>Device</u>	<u>Reason</u>
Electrofilter	• Not commercially demonstrated • Temperature limitation • Solids level limitation
Pressure filters	• Solids level limitation • Short cycles and low rates • Mechanical problems at elevated temperatures
High Gradient Magnetic Separators	• Solids level limitation • Dependent on magnetic properties of solids which are unknown
Solid bowl and Nozzle disc centrifuges	• Hydroclone - nozzle disc combination most efficient • Mechanical problems at elevated temperatures • Temperature limitations
Antisolvent Precipitation	• Not commercially demonstrated • Economic feasibility of using high treat rates of antisolvent

Preliminary Data Indicates Higher Than Predicted Scrubber Liquid Viscosity

Preliminary information from samples of unfiltered stripper bottoms from the Large Stirred Coking Unit (LSCU) indicated higher than anticipated viscosity at high temperatures. If this is confirmed, the use of hydroclone separators, the preferred technique for fines removal, may not be feasible. Blending of the EDS scrubber liquid with 1000°F minus material would be needed to reduce the viscosity to the 2-3 cp range targeted for hydroclones.

Vendor Experience With Vertical Leaf Pressure Filters Being Reviewed

Past experience with vertical leaf filters in petroleum service indicated these devices were not suitable for scrubber liquid service, even with precoat. The filters would be limited by high pressure drops and low capacities because of the highly resistant solid cake that forms during filtration of fine particulates. However, recent vendor experience with coal tar and tar pitch showed unanticipated improvements in this filter's performance with few mechanical problems. This type of filtration system could prove to be the preferred technique for removing fines from the EDS FLEXICOKING Unit scrubber liquid if target capacities and efficiencies prove attainable. Plans are being made to test a vertical leaf filter without precoat for particulate removal from LSCU coal liquids.

Future Plans

Viscosity measurements are planned at temperatures up to 700°F on samples of LSCU filtered stripper bottoms to verify the preliminary information. In addition, the viscosity of stripper bottoms mixed with 1000°F minus material will be determined. Pending the outcome of these measurements, hydroclone testing will begin with LSCU liquids in the laboratory fines removal loop at elevated temperatures. These tests are needed to verify the hydroclone performance model previously developed using petroleum coker liquids. Laboratory evaluation of a nozzle disc centrifuge is also being considered. Finally, vendor tests of the vertical leaf filter are scheduled.

5.2 Scrubbing/Fractionation of Liquefaction Bottoms from FLUIDCOKING

A first objective of this program is to determine whether conventional petroleum FLUID COKING design and operating criteria are applicable to the 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 FLUID COKING scrubber liquids from both liquefied Illinois coal and petroleum based fluid cokers were compared.

A second objective of this program is the determination of the physical properties of the liquid products from FLUID COKING to aid in their characterization.

Based on the comparison of the qualities examined, the standard criteria that are employed to design petroleum based FLUID COKING scrubber/fractionators, except those relating to liquid viscosity, should be applicable to liquefied Illinois coal based FLUID COKING units. The higher viscosity of the Illinois coal derived scrubber liquids would have to be taken into account in the design of the tower internals and external pumparound cooling circuit using standard design techniques.

Background

The vapors leaving the FLUID COKING reactor are near their dew point. It is desirable to quench them as soon as they leave the coker reactor vessel to avoid coke formation. For this reason, it has been the practice to mount a simple scrubber tower directly above the reactor. Additionally, several criteria have been developed, based on petroleum coker operating experience, to minimize operating problems. Failure to observe these criteria will usually result in excessive coke deposits in the scrubber and may limit run length.

Experimental Procedure

Side-by-side comparisons were conducted on Illinois coal and petroleum derived coker samples to determine whether any out of the ordinary operating problems are anticipated. Coker scrubber liquids were obtained from the Large Stirred Coker Unit (LSCU) operating on Illinois coal liquefaction bottoms and an operating Exxon petroleum based FLUID COKING unit. The as-received samples, as well as samples which were heat soaked at 800°F for one hour to magnify any changes which might occur in the scrubber, were subjected to a series of analytical tests. The tests included measurements of viscosity, bench coking yields, pyridine solubles, benzene solubles, specific gravity, and thermal stability. With these tests, we have attempted to assess the comparative stability of the coal based material, in terms of coking and polymerization tendency, under scrubber conditions.

A program has been initiated to characterize the liquid products from the fluid coking of EDS liquefaction bottoms. Samples of the following cuts have been requested: C₅-400°F, 400-700, 700-1000 and 1000°F+. Upon receipt of the samples, a number of inspections will be run, including ASTM distillations, API gravity, viscosity, molecular weight, surface tension, elemental analysis, Conradson carbon, and aniline point.

Discussion of Results

• Benzene Solubles

Benzene solubles were determined to give a quantitative measure of the asphaltene and oil content (material soluble in a paraffinic solvent) of the samples. A change in the benzene solubles of a sample would identify the relative polymerization tendency of the asphaltenes and oil in that sample. The results of this analysis for the various samples examined are presented in Table 5-3.

As can be seen in Table 5-3, the benzene solubles of the raw Illinois coal based sample was 87.8%, while that of the heat soaked sample was 63.8%. This decrease of 24.0% suggests that polymerization occurs in the benzene soluble material of the Illinois coal based coker liquid when subjected to conditions representing severe scrubber operating conditions. The change appears to take place early in the heat soak (benzene solubles was 77.0% after the first 5 minutes of the heat soak). Thus, we have magnified any change expected under commercial operating conditions. A petroleum sample, under similar heat soaking conditions, had very little change in benzene solubles.

The primary implication of this difference in change of benzene solubles with severe scrubber operating conditions between the coal and petroleum based samples appears to be one of viscosity change and will be discussed below under "Viscosity". This conclusion is based on evaluating this difference in combination with the measured changes in the other properties.

• Pyridine Solubles

Pyridine solubles were determined as an indication of the large molecule material content in each sample. A change in the pyridine solubles of a sample would identify the relative polymerization tendency of the large molecules in that sample. The results of this analysis for the various samples examined are also presented in Table 5-3.

The pyridine solubles of the petroleum based sample was essentially unchanged by the heat soaking. The pyridine solubles of the Illinois coal based sample increased from 92.3% to 97.2% and suggests a tendency toward slight cracking, rather than polymerization, may occur in the large molecule material.

Thus, in terms of the properties measured by pyridine solubles, there is little difference between coal and petroleum materials.

TABLE 5-3

PROPERTIES OF SCRUBBER LIQUID SAMPLES

	<u>Illinois Coal Sample</u>	
	<u>Raw</u>	<u>Heat Soaked</u>
Pyridine solubles, wt%	92.3	97.2
Benzene solubles, wt%	87.8	63.8
Bench Coking Yield, %		
Coke	85.1	88.3
Liquid	14.7	10.9
Gas	0.2	0.8
Ash, %	4.0	n.a.
Microlube Distillation		
IBP, °F	770	n.m.
1000°F Yield, %	25	n.m.
Viscosity	See Figure 5-1	

n.a. = not applicable

n.m. = not measured

- Bench Coking Yields

Bench unit coking yields were determined as an indication of coke make tendency. The higher the coke yield in this test, the greater it was assumed that the coking tendency of the material would be. The results of this analysis for the various samples examined is also presented in Table 5-3.

As can be seen in Table 5-3, the coke yield, via bench coking, increased for the Illinois coal based samples by 3.2% after heat soaking. The petroleum based sample showed a somewhat bigger increase. Thus, in terms of coking tendency as measured by bench coking yields, the Illinois coal based material has the same or possibly slightly lower tendency to form coke.

- Viscosity

Viscosity results for the raw and heat soaked scrubber liquids are shown in Figure 5-1. The viscosity of both the raw and heat soaked Illinois coal samples is higher than that of the petroleum samples. Additionally, while the viscosity of the petroleum based material decreased or remained approximately unchanged over the course of the heat soaking, the viscosity of the Illinois coal based material increased, indicating some form of polymerization is occurring. If benzene solubles are used as a measure of the extent of polymerization, it appears that the polymerization occurred in the early stages of the heat soak. This was discussed in the previous discussion on "Benzene Solubles".

The anticipated effect of the higher viscosity of the Illinois coal based material is in its flow characteristics. However, if the scrubber is designed taking into account the higher liquid viscosity, the scrubber operation should be comparable to typical petroleum based operation.

Conclusions

Based on the evaluations of the properties discussed above, the standard design criteria used to design a petroleum based FLUID COKING unit, except those relating to liquid viscosity, should be applicable to Illinois coal based feed. The higher viscosity of the coal based liquids would have to be taken into account in the design of the tower internals and external pumparound cooling circuit using standard design techniques.

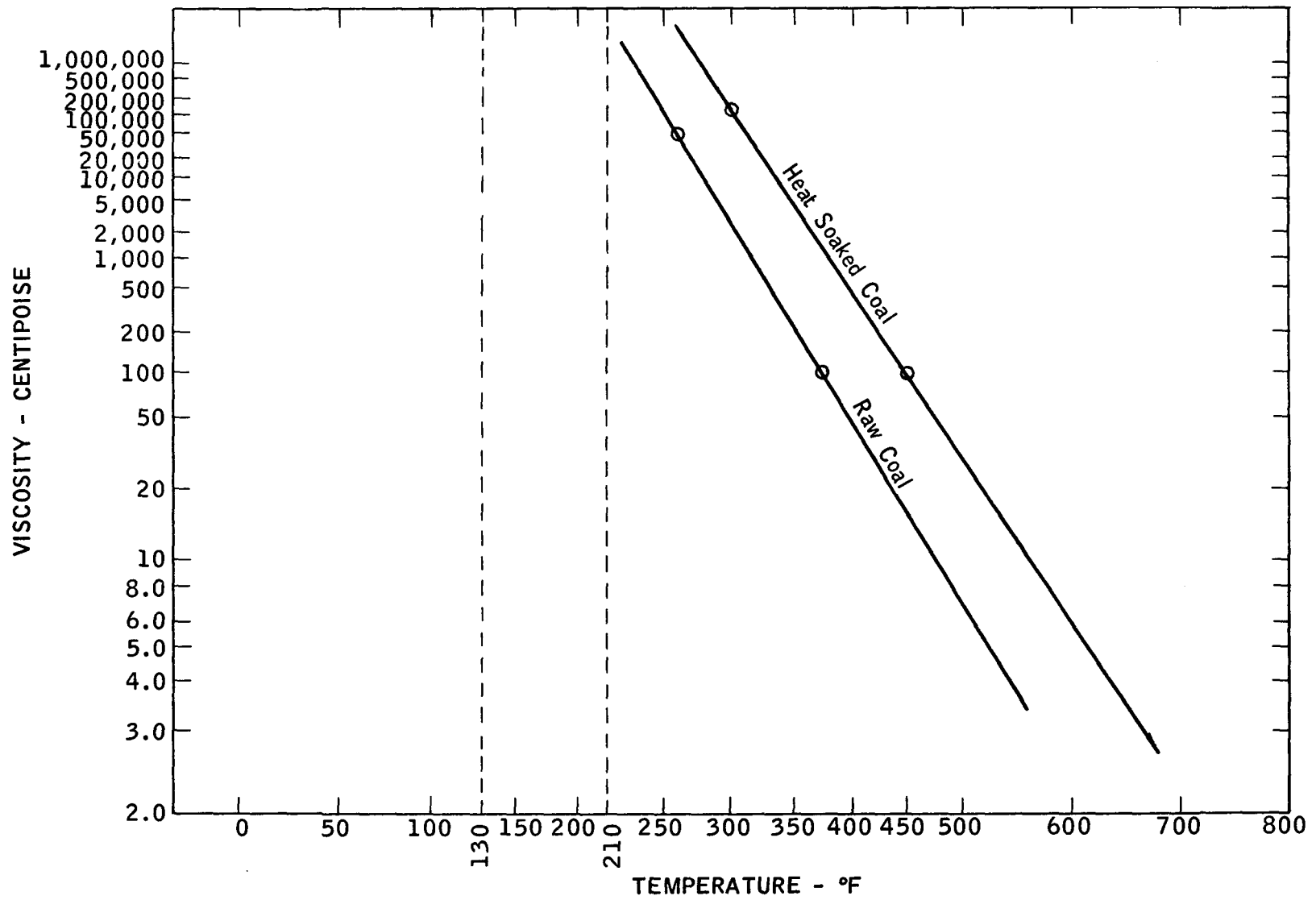
5.3 FLEXICOKING Unit Attrition and Erosion Characterization

Summary

A total of 20 coke samples from different feedstocks and processing units were tested for attritability and erosivity. Measured in terms of fines (<44 μ m diameter particles) generation, coke attritability increased with increasing extent of gasification. At similar levels of gasification,

Figure 5-1

VISCOSITY OF RAW AND HEAT SOAKED SCRUBBER SAMPLES



coal-derived cokes were approximately two to eight times more attritable than petroleum-derived cokes. A high percentage (>50%) of the fines were less than $10\mu\text{m}$ in diameter. Erosivity decreased with increasing attritability as was expected.

Coke Attritability Correlations With Ash Content and With Extent of Gasification

Of the 20 coke samples that have been attrition tested, ten were from Illinois coal liquefaction bottoms, seven from petroleum resid, and three from Tar Sands Bitumen. The coal-derived cokes were produced in the IKG unit operating either as a FLUID COKING or FLEXICOKING Unit. The petroleum cokes were produced from commercial FLUID COKING and FLEXICOKING Units as well as the prototype FLEXICOKING Unit. Tar Sands cokes were produced in the IKG unit in a FLEXICOKING mode and the prototype FLEXICOKING Unit. A majority of the 20 samples were subsequently gasified in the FBU unit. Table 5-4 presents attrition test results, classified by feedstock and processing unit, together with the ash content and level of gasification in the FBU.

Figure 5-2 shows that attritability, as measured by fines generation, increased with increasing ash content. There was no clear effect of feedstock or processing unit, given the considerable scatter in the data. Figure 5-3 shows that fines generation also increased with increasing level of gasification. Presented in this way, test results show that coal-derived cokes at identical levels of gasification are two to eight times more attritable than the petroleum-derived cokes.

FBU Attrition Results Show That Coal Fines Are Very Fine

Samples taken from the FBU overhead system were found to contain a large fraction of fines that were very fine ($<10\mu\text{m}$ in diameter). See Figure 5-4, where the lines show the general trend of the data for three types of coke: petroleum-derived, coal-derived from FLUID COKING Units, and coal-derived from FLEXICOKING Units. The amount of $<10\mu\text{m}$ material appears to be dependent on the level of gasification with coal-derived cokes having approximately an order of magnitude higher percentage of very fine material compared to petroleum-derived cokes.

Erosivity Decreases With Increasing Attritability

The results of erosivity testing are presented in Table 5-5. As would be expected, erosivity was found to decrease with increasing attritability (Figure 5-5), with no clear dependence of results on feedstock or unit type apparent.

TABLE 5-4

SUMMARY OF COKE ATTRITION TEST RESULTS *

% Fines (<44 μ m) Generated

Coking Unit Feedstock	Commercial FLUID COKING Unit	IKG FLUID COKING Unit	IKG FLEXICOKING Unit (Gasifier)	IKG FLEXICOKING Unit (Reactor)	Prototype FLEXICOKING Unit	Commercial FLEXICOKING Unit (Gasifier)
Petroleum Resid	0.0 (0.45, 0) 0.0 (0.15, 0) 0.3 (0.30, 29) 0.5 (0.34, 43)				1.1 (1.64, 0) 0.3 (2.32, 29)	3.0 (3.55, 0)
Tar Sands Bitumen			0.3 (10.5, 0) 7.4 (16.1, 43)		0.0 (16.4, 0)	
Illinois Coal Bottoms		0.4 (24.8, 0) 4.8 (32.3, 50) 4.8 (45.5, 65) 5.4 (30.7, 45) 6.5 (29.8, 36) 7.4 (52.4, 70)	1.3 (23.5, 0)	1.5 (35.7, 0) 2.5 (23.9, 0) 8.6 (63.7, 78)		

*Figures in the brackets are % ash and % gasification in FBU on an ash-free basis, respectively.

Figure 5-2

FINES ($< 44 \mu m$)
GENERATION INCREASES WITH ASH CONTENT

Symbol	Feed Stock	Unit
□	Petr. Resid	Comm. FLUID COKING
◇	" "	Prototype FLEXICOKING
○	" "	Comm. FLEXICOKING
■	Tar Sands Bitumen	IKG FLEXICOKING (Gasifier)
◆	" " "	Prototype FLEXICOKING
●	III. Coal Liq. Btms	IKG FLUID COKING
▲	" " " "	IKG FLEXICOKING (Gasifier)
▼	" " " "	IKG FLEXICOKING (Reactor)

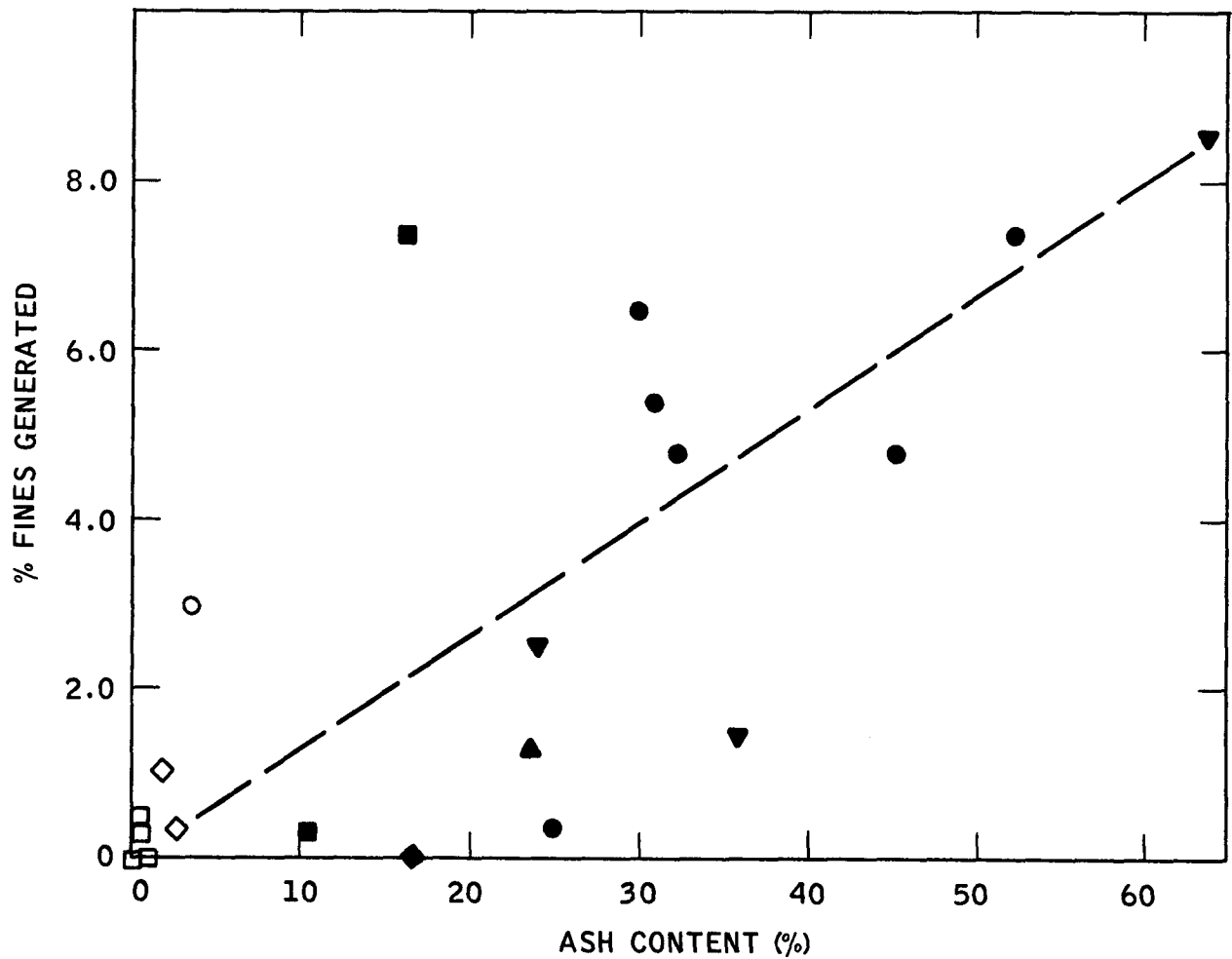


Figure 5-3

COAL-BASED COKES SHOW HIGHER ATTRITABILITY
THAN PETROLEUM COKES FOR THE SAME DEGREE OF GASIFICATION

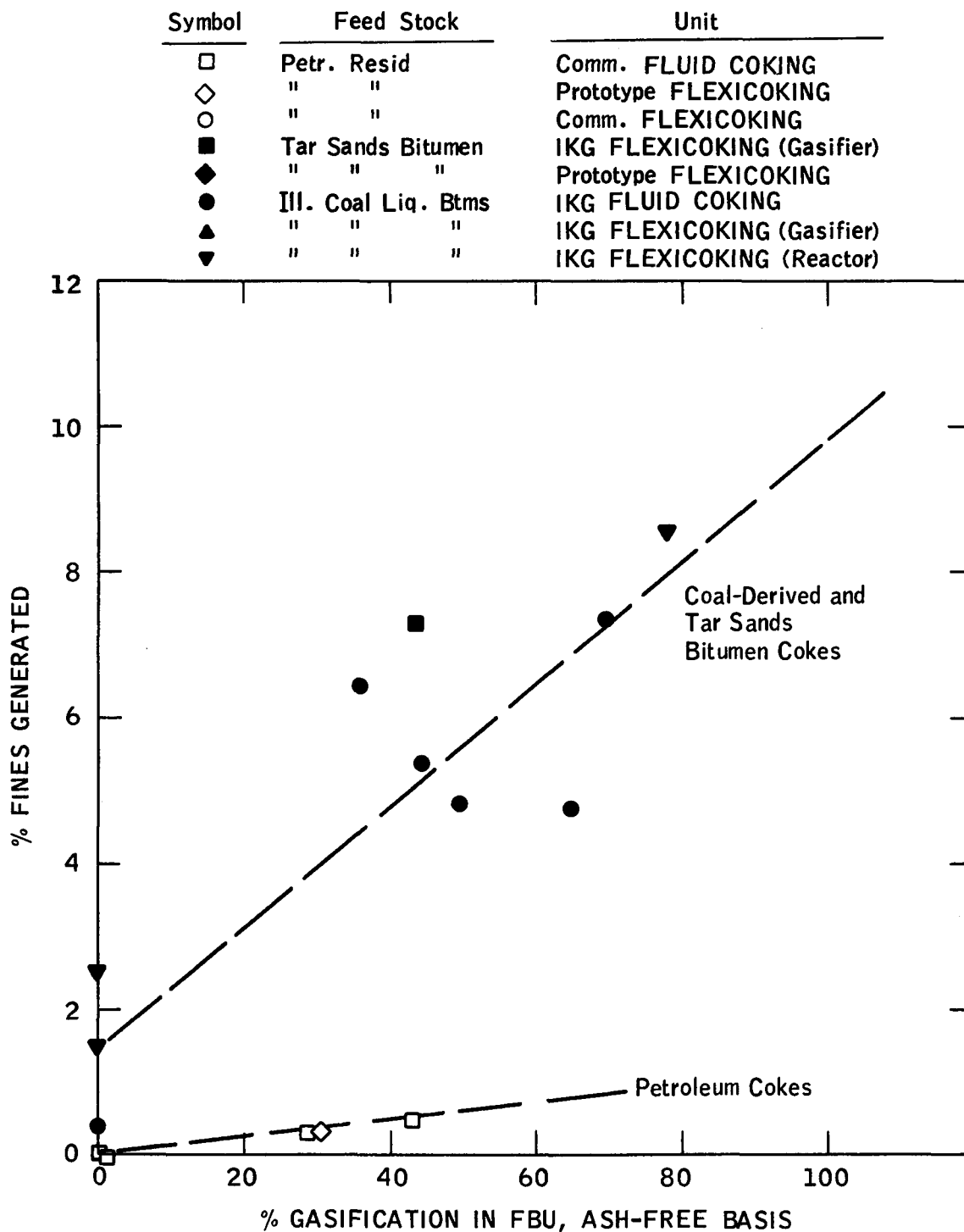


Figure 5-4

% OF FBU OVERHEAD CATCH WITH PARTICLE DIAMETER
< 10 μm VERSUS % GASIFICATION IN FBU

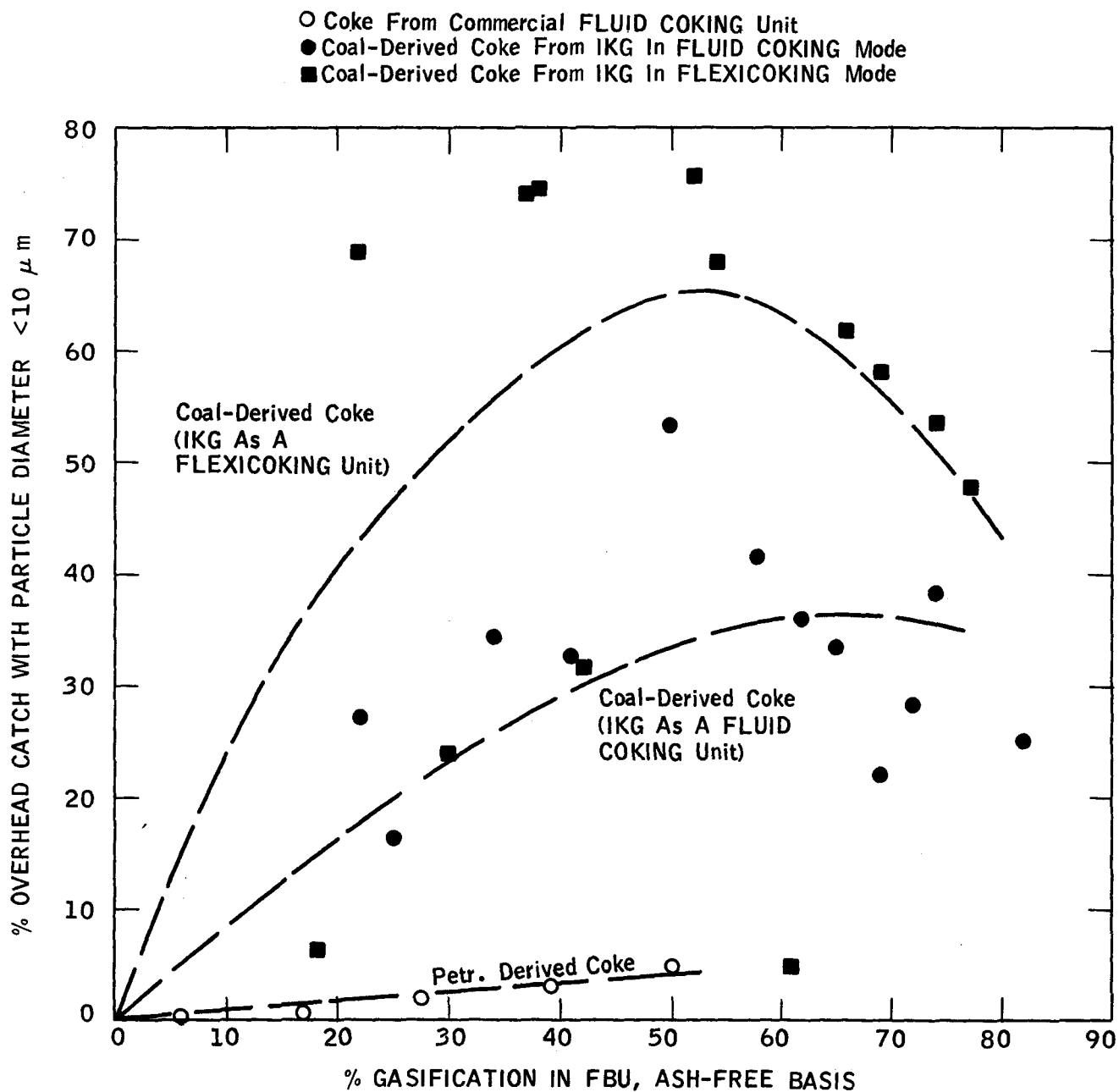


TABLE 5-5

RELATIVE EROSIVITY OF COKE PARTICLES*

Relative Erosivity**

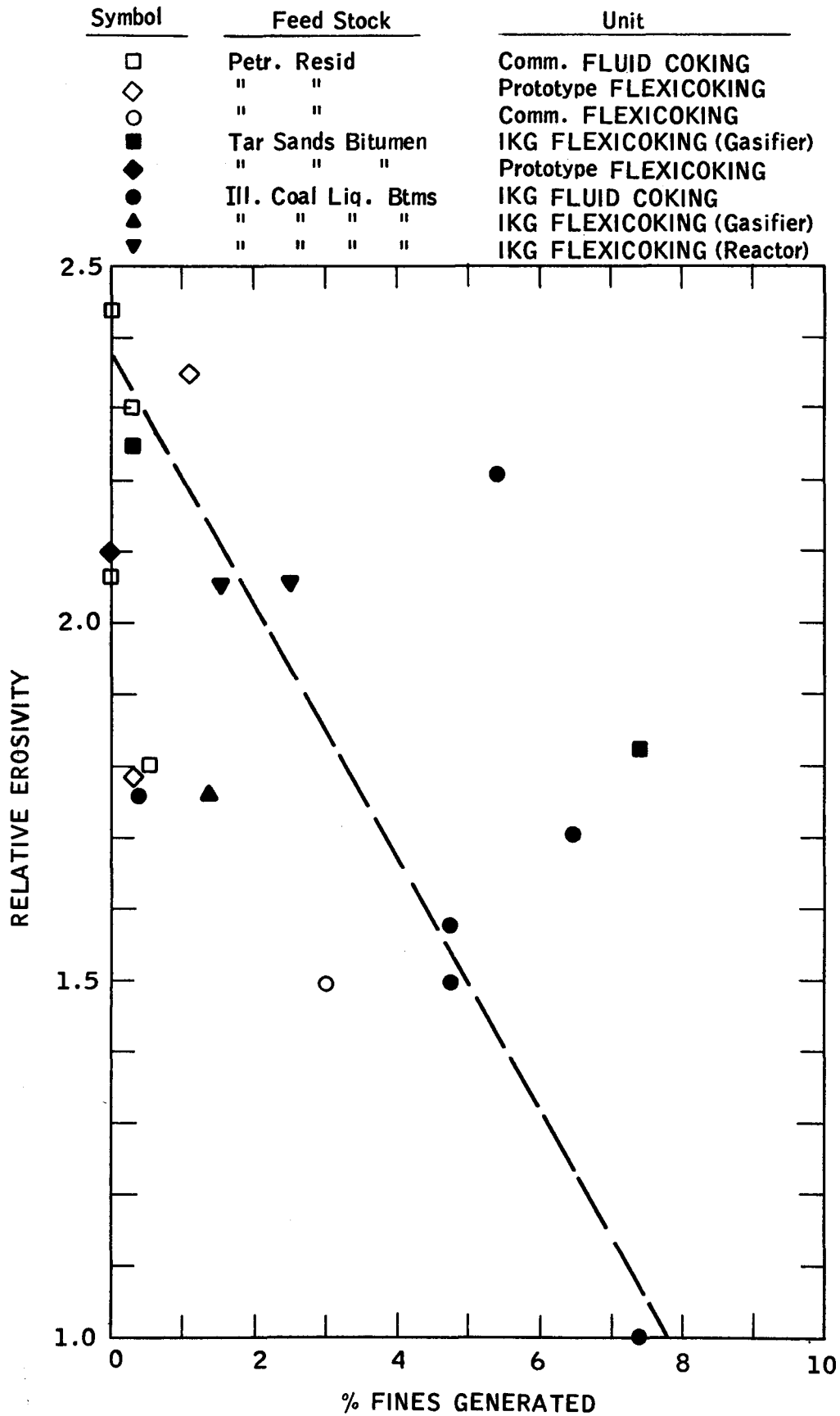
Coking Unit Feedstock	Commercial FLUID COKING Unit	IKG FLUID COKING Unit	IKG FLEXICOKING Unit (Gasifier)	IKG FLEXICOKING Unit (Reactor)	Prototype FLEXICOKING Unit	Commercial FLEXICOKING Unit (Gasifier)
Petroleum Resid	1.80 (0.5) 2.07 (0.0) 2.30 (0.3) 2.44 (0.0)				1.77 (0.3) 2.35 (1.1)	1.50 (3.0)
Tar Sands Bitumen			1.83 (7.4) 2.25 (0.3)		2.07 (0.0)	
Illinois Coal Bottoms		1.00 (7.4) 1.50 (4.8) 1.58 (4.8) 1.71 (6.5) 1.76 (0.4) 2.21 (5.4)	1.76 (1.3)	1.18 (8.6) 2.06 (1.5) 2.08 (2.5)		

*Figures in brackets are % fines generation.

**Relative Erosivity = $\frac{\text{Target wt. loss from coke particle}}{\text{Target wt. loss from a reference catalyst particle (CBZ-1)}}$

Figure 5-5

COKE EROSIVITY DECREASES WITH ATTRITABILITY



Future Work

Results from this program are being used in the program on FLEXICOKING Unit Solids Material Balance and Control. Further testing on coke from Wyoming coal may be required.

5.4 FLEXICOKING Unit Solids Material Balance and Control

Summary

A computer program to model the solids balance for the FLEXICOKING process has been developed. The program is an extension of a previously developed fluid solids material balancing program called DYNAMOD. The model was adjusted using particle size distribution and fines loss data from the prototype FLEXICOKING Unit. Model predictions for the effects of process condition changes in the prototype unit and scaleup to a commercial unit for petroleum resid feeds were checked against available data.

The model was used to simulate both prototype and commercial scale operations for coal liquefaction bottoms (CLB) feedstocks by adjusting model parameters based on laboratory kinetics and attrition data. The steady-state particle size distributions obtained were similar to petroleum experience; however, heater and reactor fines losses were somewhat higher than previously assumed. The model is currently being used in both EDS FLEXICOKING prototype design and commercial study design efforts.

Model Validated With Petroleum Based Data

The model basis and its initial testing were discussed in the first Phase IV quarterly report (January 1-March 31, 1978). To summarize, the model was developed from an existing fluid solids material balance program which solves transient equations for individual particle sizes in the major process vessels. The terms in the equations accounting for particle attrition and cyclone efficiencies were adjusted with data from a base case operating period for the prototype FLEXICOKING Unit with a petroleum feedstock. Model predictions compared well with data from the operating unit.

The effect of scaleup was evaluated by simulating the operation of a commercial unit whose feed rate was 25 times that of the prototype unit. Results were compared with available operating data. The particle size distributions measured in the large unit were somewhat finer than predicted over most of the distribution indicating either better feed dispersion or slightly smaller attrition products. Good agreement was possible by fine tuning the attrition model. The fines losses predicted from the reactor and heater cyclones were relatively insensitive to this adjustment and agreed well with the measured value.

Simulation of EDS Bottoms Feed Shows Higher Fines Losses

A commercial scale FLEXICOKING Unit with CLB feedstock was simulated based on the revised study design. Coking rates in this unit were projected to be very low leading to solids deposition rates on the order of ten times higher than in petroleum experience. Laboratory data indicated that coal-derived coke will have attrition rates two to eight times higher than those of petroleum coke. The attrition rate in the simulation was assumed to be ten times that from the petroleum simulation as a limiting case. On this basis, the model predicted a particle size profile similar to petroleum experience over most of the distribution. However, fines losses from the reactor and heater cyclones would be about two and four times greater respectively than developed in the study design. Further work to better define these losses can be done when prototype unit data is available.

A series of parametric cases were also evaluated to determine the sensitivity of model results. Lab data has indicated that the gasification mechanism for coal-derived coke may be different from that of petroleum cokes. Thus, the model was adapted to permit two mechanisms in any relative proportion. Also, the effect of varying the particle density from 80 to 120% of the nominal value was determined. The model predicted that the particle size distributions in the beds were relatively insensitive to these variables. Mean bed particle size varied only about 20 μm when density was varied over the range cited above. Since the fines content of the beds varied only slightly, loss predictions from the reactor and heater did not change significantly.

Prototype Unit Simulated With CLB Feed

The prototype unit with EDS CLB feedstock was also simulated. This case also used an attrition rate ten times that of petroleum and a 50/50 combination of the two gasification mechanisms. The resulting bed distributions were similar to petroleum experience with fines losses again being substantially higher. Equipment requirements to handle these higher fines rates are currently being developed.

Future Work

No further simulation work as part of the model development is planned. The model is being used in both the EDS prototype design and commercial study design efforts. Further simulation work may be desirable when data is available from the prototype FLEXICOKING unit.

5.5 FLEXICOKING Unit Gasifier Grid Design

Summary

Preliminary planning has begun on developing design and scaleup criteria for a Coal Liquefaction Bottoms FLEXICOKING Unit Gasifier grid. The experimental unit for cold model simulations of gas/solids mixing and contacting in the grid region of a FLEXICOKING Unit has been identified.

The unit will be modified to model the grid of a commercial FLEXICOKING unit. The experiments will aim at identifying possible grid designs that minimize solids stagnation.

Cold Model Unit Able to Simulate Interactive Effects At and Near the Grid

The experimental unit for cold model studies is an 8' wide by 1' deep by 12' high plexiglas flow visualization unit which simulates a vertical slice of the gasifier. The unit was previously used for proprietary studies. Major modifications to the grid region are required before it simulates the grid region of a gasifier. This size unit was selected to permit observing the interactive effects between grid caps. Gas/solids mixing and contacting due to grid jets will be observed and recorded photographically.

Future Work

The required unit modifications will be defined and material to charge the unit will be obtained. Initial testing now scheduled for the fourth quarter of 1978 will be aimed at characterizing mixing in the grid region and identifying possible methods for minimizing solids stagnation.

5.6 FLEXICOKING Unit Reactor Feed Nozzle Development

Summary

An assessment was made of the operating characteristics of FLEXICOKING Unit reactor feed nozzles when handling EDS coal liquefaction bottoms (CLB) feedstocks. This evaluation indicated that adequate nozzle capacity and atomization may require high pressures or very high steam dilution ratios because of the much higher viscosity of CLB feed compared to typical petroleum-derived feedstocks. The presence of substantial solids will limit the options available to overcome these problems.

An experimental program has been planned to develop suitable nozzles for CLB feedstock and construction of the required equipment is almost complete. The planned program will allow testing of nozzle performance over a wide range of flow parameters using simulated feeds.

Atomization of Gritty, Viscous Feed Will Require Severe Operating Conditions

The atomization and liquid capacity characteristics of the typical FLEXICOKING Unit feed nozzle operating with petroleum resid was chosen as the performance target for operation with coal liquefaction bottoms. This nozzle is simple in design, effective, requires little maintenance, and thus provides a good starting point for further development. However, the capacity and extent of atomization with this nozzle for CLB feeds at normal operating conditions is expected to be inadequate due to the high viscosity and solids content. Current design studies indicate that the CLB FLEXICOKING Unit feed may have a viscosity of 5 to 15 poise at operating nozzle conditions and may contain on the order of 30 to 40 wt% solids.

With petroleum-based feedstock, adequate dispersion and penetration into the fluidized bed is obtained with mixed steam/ oil flow to aid atomization. For the very viscous CLB feedstock, pressures higher than those used with petroleum resid feedstocks may be necessary to obtain adequate jet velocities for good dispersion, and avoid severe slugging in the nozzle. Alternatively, high steam-to-oil ratios might be used with lower pressure; however, capacity would be substantially reduced. Furthermore, atomization correlations developed for less viscous fluids indicated that impractically high steam rates would be required to produce acceptably sized droplets with coal liquefaction bottoms. Thus, higher pressure nozzle operation may be the more promising approach.

Experimental Program Planned for Feed Nozzle Development

Since CLB feed properties and flow behavior lie substantially outside the range of applicable experience, an experimental program to test different nozzle configurations is required. The fluid system will be simulated using nitrogen-glycerin-solids mixtures. Tests have shown that glycerin wets coke from a commercial FLEXICOKING Unit and that slurries of high solids content remain suspended for several days. The anticipated range of fluid properties will be obtained by adjusting the temperature and solids content of the slurry. The experimental unit will circulate slurries to the nozzle at pressures up to 500 psi and will be completely closed to avoid contamination of glycerin by water vapor. The viscosity of the circulating slurry will be monitored regularly and the nozzle spray characteristics will be recorded photographically.

Future Work

Initial experimental work will aim first at viewing the operation of the typical FLEXICOKING Unit nozzle with fluids simulating a petroleum feed. Then, a series of tests exploring a wide range of pressures, gas/liquid ratios, and exit velocities will be made with high viscosity feeds using various nozzle configurations. Once acceptable performance is obtained without solids, solids at different loadings and in various size ranges will be added to quantify their effect.

5.7 FLEXICOKING Unit Ash-Handling and Disposal Characterization

Characterizations of the ash-solids streams from the IKG pilot unit will provide basic data for developing handling and disposal systems for these streams from a future commercial FLEXICOKING unit running on vacuum bottoms from an EDS Coal Liquefaction Plant.

The two major streams to be handled are bed purge solids and heater tertiary cyclone fines. Particle size distribution of these two will be quite different. Flow properties of solids vary appreciably with the percentage of fine material in a mixture. The flowability of these ash streams and sensitivities to percentage fines will be tested.

It is not known whether conveying, transport, and disposal technology now used for ash from coal power plants can be applied directly to the solids from the FLEXICOKING unit due to the particle size and density differences between coal ashes and the EDS solids. If the dry solids are not comparable to coal ash, development of an appropriate technology will be required. Illinois and Wyoming coal power plant furnace ashes will be tested for comparison with the ash/coke streams.

An additional ash/coke stream is the venturi scrubber fines present as a solids slurry in water. Dewatering would allow it to be handled with solids handling technology. The solids percentage at which the material can be handled as a dry cake will be determined in the testing procedure. Similarly, if partially dewatered venturi scrubber fines are combined with the two dry streams, the effect of moisture content on the handling properties of the composite mixture will also be determined in testing.

The total ash/coke stream must be disposed of in a manner which fulfills environmental considerations. It has been assumed that a compacted landfill would be used. The permeability, strength, and compaction characteristics of the composite stream must be known in order to design and select a disposal site. These and other soils engineering factors will be determined in testing.

The individual tests, test purposes, and test outputs are listed in Table 5-6. A testing program has been devised to maximize the data output from the limited quantity of IKG Pilot Plant samples available.

The representativeness of the IKG unit operating conditions during the period when the previously supplied samples were collected have been reviewed with respect to the anticipated commercial plant operating conditions. As a result, a more representative bed purge specimen is being taken at IKG. Suitable heater overhead fines have already been received and will be used to represent both the tertiary cyclone fines and, by addition of water, venturi scrubber fines. Testing will begin upon receipt of the more representative bed purge sample and receipt of Illinois and Wyoming coal furnace fly and bottom ash samples.

TABLE 5-6

TEST PROGRAM ON ASH/SOLIDS FROM IKG UNIT

<u>Test</u>	<u>Data Output</u>	<u>Data Usage</u>	<u>Test Standards</u>
Bulk Density (loose)	the minimum dry, unpacked density	used for sizing storage and transport equipment and in solids flow characterizations	SHA test
Bulk Density (packed)	the maximum dry, packed density	used for sizing storage structural requirements and in solids flow characterizations	SHA test
Angle of Internal Friction	the dynamic angle of friction between the particles	used for sizing chute and duct outlets	SHA test
Angle of Repose	the cone angle of a loose pile	basic parameter used in conveyor and pile design and in solids flow characterizations	SHA test
Angle of Fall	the cone angle that the pile built for the angle of repose assumes when its base is disturbed	used in solids flow characterizations	SHA test
Angle of Spatula	another representation of the material's angle of internal friction	used in solids flow characterizations	SHA test
Angle of Slide	the angle of friction between a steel plate and the material	used in design of chutes and hoppers	SHA test
Cohesion	a measurement of intra-particle cohesiveness	used in solids flow characterizations	SHA test

Continued....

TABLE 5-6 (cont'd)

<u>Test</u>	<u>Data Output</u>	<u>Data Usage</u>	<u>Test Standards</u>
Disperability	a measurement of cohesiveness and dustiness	used in solids flow characterizations	SHA test
Zenz Drop Test	an indicator of potential for dense phase pneumatic conveying	-	SHA test
Pneumatic Pick-up Test	the minimum air velocity required for dilute phase pneumatic conveying	-	SHA test
Flow Behavior vs Moisture Level	observation of flow and handling behavior at increasing moisture contents	-	SHA test
Particle Size Analysis	the particle size distribution	used in solids flow characterizations and soil classifications	ASTM D-422
Particle Specific Gravity	the particle density relative to water	used in pneumatic conveying, hydraulic conveying and soils engineering	ASTM D-854
Consolidation Behavior	the coefficient of consolidation and indirectly, the permeability constant	used to predict rate and amount of settlement of soils and to determine rate of subsurface water flow and drainage capability	ASTM D-2435
Compaction Test	the optimum moisture content to reach the maximum compacted density	used to determine suitability of the soil for fill purposes	Harvard miniature
Plastic Limits	the percentage water that results in plastic behavior	used to determine the suitability of the soil for fill purposes and for soil classification	ASTM D-424

Continued....

TABLE 5-6 (cont'd)

<u>Test</u>	<u>Data Output</u>	<u>Data Usage</u>	<u>Test Standards</u>
Liquid Limit	the percentage water that results in a fluid behavior	used to determine the suitability of the soil for fill purposes and soil classification	ASTM D-423
Shear strength	the resistance of the soil to force	a basic parameter used in design of foundations and embankments	Triaxial
Particle Surface Analysis	particle shape and surface characteristics	used in solids flow characterizations, in soil classification and in calculations of surface to volume ratios	Optical Microscope and Scanning Electron Microscope

ENGINEERING RESEARCH AND DEVELOPMENT

ENGINEERING TECHNOLOGY DEVELOPMENT

6. Materials and Environmental Engineering Technology

6.1 Environmental Control - Water

Introduction

As part of the development of the Exxon Donor Solvent process, a long range R&D program was proposed to identify the characteristics of the wastewater from a commercial EDS plant and to define a cost effective treatment scheme to meet potential effluent regulations. The program sequence and timing is shown in Figure 6-1.

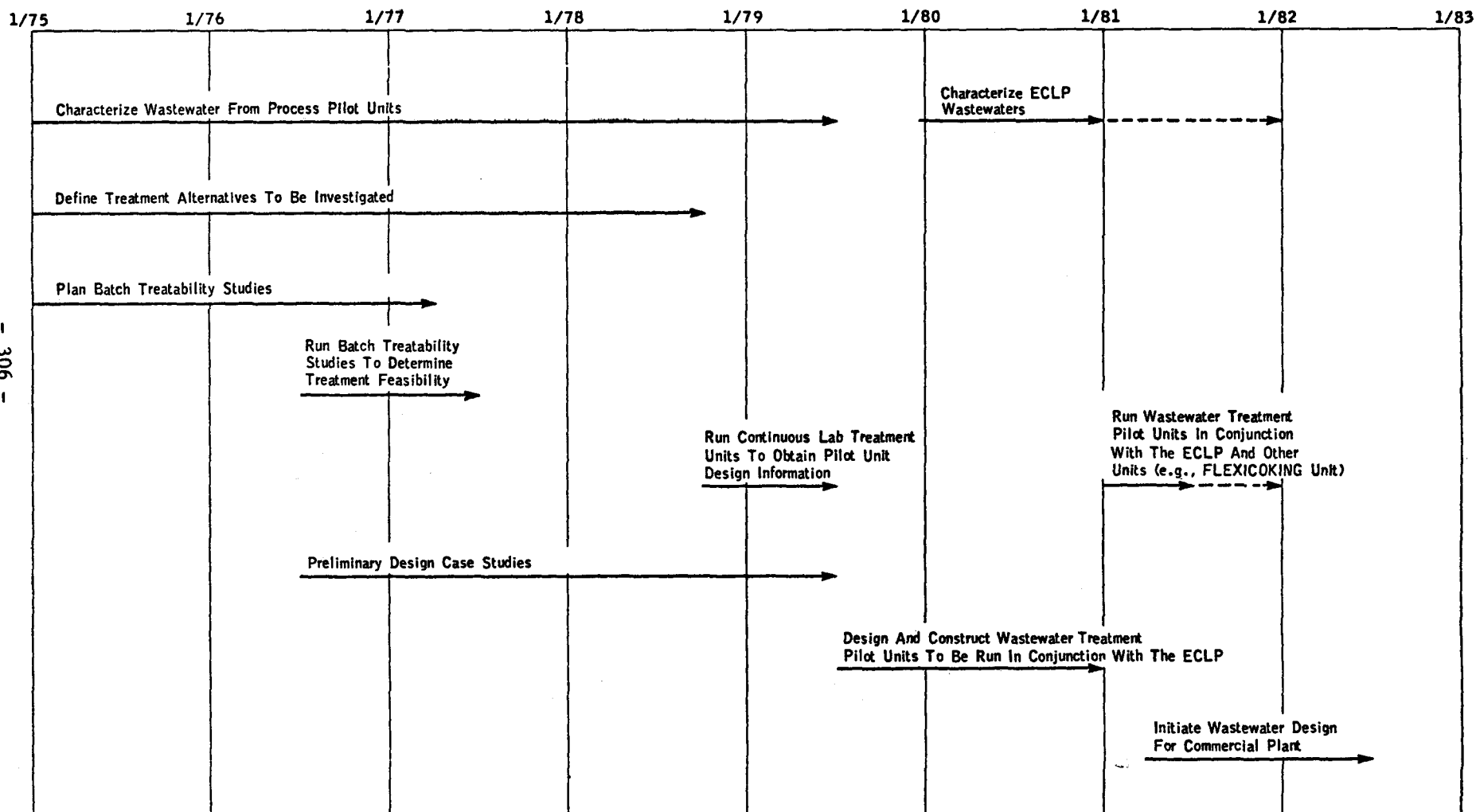
The results of the Phase IIIA program indicated that the proposed treatment scheme should meet most, if not all, anticipated government effluent regulations. The overall objective of the Phase IIIB work (July, 1977 - December, 1977) and the initial part of the Phase IV program (January - June 1978) was to further substantiate the conclusions reached during Phase IIIA work and to investigate treatment alternatives in order to reduce costs. The list below shows the major activities conducted from July, 1977 through June, 1978.

1. Characterization of wastewaters from process pilot units.
2. Estimation, using ER&E computer models, of the quantity and quality of process wastewater from and EDS commercial plant using Illinois coal as feed.
3. Preparation of preliminary designs and cost estimates for alternative wastewater treatment schemes.
4. Estimation of trace contaminant levels in the EDS process wastewater.
5. Evaluation of the possible EDS process modification in which liquefaction cold separator wastewater is hydrotreated to reduce contaminant levels.
6. Bench scale simulation of isopropyl ether extraction and carbon adsorption treatment steps in order to verify PNA removals reported in the literature.
7. Computer simulations of the EDS process sour water stripping facilities.

Current projections of future regulations indicate severe restrictions on the discharge of all pollutants. Based on earlier results, plus results from this reporting period, a basic treating sequence of sour water stripping, solvent extraction, equalization, dissolved air flotation, air activated sludge, filtration, and carbon adsorption should be able to meet

Figure 6-1

TIMETABLE FOR COAL LIQUEFACTION WASTEWATER TREATMENT STUDIES



most, if not all, of these requirements. However, heavy metals restrictions, which appear to be getting more strict, may require the addition of a heavy metal removal process to the treating sequence.

Significant results and conclusions from each area investigated during the period of July, 1977, through June, 1978, are summarized below.

Characterization of Wastewaters from Process Pilot Units

Characterization of the wastewaters from the Large Stirred Coker Unit (LSCU) resulted in a new, conservative estimate of 1500 weight parts per billion (wppb) of polynuclear aromatic hydrocarbons (PNA's) in the combined raw process wastewaters from an Illinois coal fed EDS plant, versus a previous estimate of 60 wppb. Most of the PNA's come from the FLEXICOKING fractionator distillate drum. However, even after considering the increase in PNA concentration, the basic treating sequence described earlier should be able to reduce the level of PNA's to less than 1 wppb in the treated wastewaters.

Analysis of a sample from the Integrated Coker Gasifier (IKG) unit supported earlier assumptions that only minor amounts of organic contaminants should be expected in the wastewater from the heater/gasifier side of the FLEXICOKING section.

Characterization of the wastewater from a laboratory simulation of the drier section of an EDS plant indicated higher levels of phenolics in this wastewater than had previously been found. It is now considered necessary to send to slurry drier wastewater to solvent extraction along with the other process wastewaters.

Analyses of other wastewater samples from process pilot units completed the data base for computer simulations of the wastewaters from a commercial Illinois EDS plant.

Estimates of the Quantity and Quality of Commercial EDS Process Wastewaters

ER&E computer models were used to prepare improved estimates of the flow rates of major contaminants in the process wastewaters from a commercial Illinois EDS plant. These estimates are shown in Table 6-1. In addition to providing estimated concentrations of many more contaminants than earlier studies, the new estimates reflect onsite design changes incorporated into the Illinois Study Design Update. The new estimates also reflect the 25% increase in size of the study design plant from 20,000 dry tons of coal/day to 25,000 dry tons/day (24,000 to 30,000 tons/day of cleaned, undried coal feed).

Table 6-1

ESTIMATED QUANTITY AND QUALITY OF RAW PROCESS WASTEWATER⁽³⁾
FROM AN EDS COMMERCIAL PLANT USING 25,000 DRY TONS/DAY OF ILLINOIS COAL⁽⁵⁾

New (Phase IIIB) Estimate

<u>Flow</u>	<u>2546 gpm</u>
<u>Contaminant (wppm)</u>	
H ₂ S	12,200
NH ₃	9,500
CO ₂	5,000
CN ⁻ (1)	4 (2)
SCN ⁻	10
Cl ⁻	90
monohydric phenols	5,700
dihydric phenols	600
organic acids	1,500
ketones and aldehydes	220
PNA's (4)	1.5
unknowns	not estimated

Notes: (1) Free cyanide.

(2) Conservative estimate.

(3) Excludes intermittent, small sour water sources
in product recovery section.

(4) Polynuclear aromatic hydrocarbons.

(5) 30,000 tons/day clean, undried coal feed.

One major change from previous estimates is a 40% decrease (to ~6400 wppm) in the level of phenols expected in the combined process wastewaters. Most of this decrease was due to a reduction in the estimated phenol concentration from the FLEXICOKING fractionator distillate drum. The decrease is not, however, sufficient to eliminate the need for solvent extraction in the basic process scheme.

Preliminary estimates of the concentrations of heavy elements, free cyanides, aldehydes, and ketones were calculated by hand. Estimated concentrations of free cyanides, aldehydes, and ketones are listed in Table 6-1. Estimated heavy element concentrations are listed in Table 6-2. It appears the basic treating sequence will be able to reduce the concentration of most of these contaminants to levels acceptable for discharge, though a process step for removing heavy metals may have to be added to the treating sequence.

Economic Studies

Two alternatives to the basic process sequence, following sour water stripping, of solvent extraction, equalization, dissolved air flotation, air activated sludge, filtration, and carbon adsorption were eliminated after preliminary analysis showed them to be very costly.

These included:

1. Catalytic oxidation of organic contaminants to replace solvent extraction, and, perhaps, eliminate the need for carbon adsorption in the basic sequence.
2. Eliminating solvent extraction and replacing air activated sludge in the basic process sequence with a biological treatment system consisting of trickling filters and air activated sludge.

Several other alternatives to the basic process sequence were examined more closely. Results of these studies are summarized in Table 6-3. It should be noted that the cost estimates listed in the table are non-project estimates, and exclude the costs of related offsite facilities and sour water stripping.

Considering the quality of the estimates, there appear to be no significant differences among the present value estimates for the base case and Options 1, 2, 3, and 5. The present value estimate for Option 4, however, seems to indicate a small incentive for using an oxygen rather than an air activated sludge unit.

In another economic study, wastewater treatment savings resulting from a possible modification of the EDS process, in which sour water from the liquefaction cold separator is sent to hydrotreating, were compared with the increased costs of hydrotreating. The results indicate that the increase in the hydrogenation section capital cost would far exceed the cost savings associated with wastewater treatment and, therefore, this scheme has been dropped from further consideration.

Table 6-2

ESTIMATED SOLUBLE HEAVY ELEMENT CONCENTRATIONS IN PROCESS
WASTEWATER FROM AN EDS COMMERCIAL PLANT USING ILLINOIS COAL⁽¹⁾

<u>Element</u>	<u>ppm</u>
As	N.D. ⁽²⁾
Cd	0.020
Co	0.020
Cr	0.52
Cu	0.77
Hg	N.D.
Mn	0.050
Mo	0.040
Ni	0.50
Pb	0.21
Se	N.D.
Sn	0.37
Ti	0.31
V	N.D.
Zn	1.6

(1) Excludes intermittent, small sour water sources in product recovery section.

(2) N.D. = none detectable

Table 6-3

ECONOMIC COMPARISON OF PHASE III-B WASTEWATER TREATMENT
ALTERNATIVES FOR EDS ILLINOIS WASTEWATERS FROM A 20,000 DRY TONS/DAY PLANT (6)

<u>Option</u>	<u>Description</u>	<u>1985 Total Erected Cost, M\$ (1)</u>	<u>Operating Cost, M\$/yr (2)</u>	<u>Present Value 1985 (3)</u>	<u>Δ% vs. base case</u>
base case	solvent extraction, dissolved air flotation, air activated sludge, filtration, carbon adsorption, sludge handling.	77.1	10.1	91.7	0
1	same as the base case, except the slurry drier wastewater bypasses solvent extraction.	76.3	9.9	90.4	-1
2	same as the base case, except a combination of trickling filters and air activated sludge replaces air activated sludge alone.	73.9	9.5	87.2	-5
3	same as Option 1, except solvent extraction precedes sour water stripping.	75.9	9.8	89.9	-2
4	same as the base case, except high purity oxygen activated sludge replaces air activated sludge.	67.1	9.1	80.7	-12
5	same as the base case, except resin adsorption replaces solvent extraction. (4)	71.1+	11.7	91.4+	0

Notes:

- (1) Non-project screening estimate. Excludes sour water stripping.
- (2) Includes chemical and utility costs, plus 9% of capital investment to cover salaries, maintenance, local taxes, and miscellaneous items. Excludes sour water stripping.
- (3) Based on a 15 year project life and 15% discount rate.
- (4) Acetone regenerant. System does not include dehydration tower for recovered phenolics.
- (5) Δ% represents differences among present value estimates.
- (6) 24,000 tons/day clean, undried coal feed.

Confirmation of Polynuclear Aromatic Hydrocarbon (PNA) Removals Reported in the Literature

Excellent removals of PNA's across solvent extraction and carbon adsorption have been reported in the literature, and our conclusion in the Phase IIIA report that only a negligible amount of PNA's would be discharged with treated effluent was based on these values. In order to confirm these removals, batch isopropyl ether and carbon adsorption tests were performed on an unstripped, unfiltered wastewater sample from the CLPP unit.

Analysis for PNA's before and after treatment was performed by an extraction/ultraviolet scan procedure developed by ER&E. Although absolute amounts of PNA's cannot be accurately determined by this method, using it both before and after treatment will result in an accurate estimate of percent removal. The results of these experiments indicated a 92+% and 96+% removal of PNA's via solvent extraction and carbon adsorption, respectively. These removals are similar to those reported in the literature. Based on these results, only a negligible amount of PNA's (less than one part per billion by weight) should be discharged with the treated effluent.

Computer Simulations of Sour Water Stripping

Analysis of pilot stripping data collected during Phase IIIA showed the large amount of caustic needed to achieve low levels of ammonia in the stripper effluent was due to the presence of organic acids, thiocyanates, and chlorides in the wastewater. Computer simulations of the fate of pollutants in an Illinois EDS process yielded a better estimate of the concentration of organic acids in the wastewaters from a commercial plant, as well as estimates of chlorides and thiocyanates. These estimates were incorporated into separate computer simulations of a sour water stripping system similar to the one envisioned in the 1975-76 Commercial Plant Study Design.

The results of these simulations, summarized in Table 6-4, show about 1,150 parts per million by weight (wppm) as NaOH is required to reach an effluent ammonia level of 70 wppm. This large amount of caustic reduces but does not eliminate the possibilities for reuse of treated wastewaters as make-up for a cooling tower.

During these simulations, the effect of putting solvent extraction upstream of sour water stripping, rather than downstream as currently planned, was also investigated. This change in process sequence did not reduce the caustic requirement significantly. Since this arrangement would require enlarging the sour water stripping system by about 37%, the sequence of sour water stripping followed by solvent extraction is preferred.

The possibility of allowing a higher level of ammonia in the effluent from sour water stripping, then removing this ammonia later in the treating sequence, was briefly examined. A rotating biological contactor for nitrification following air activated sludge was inserted in the process

TABLE 6-4

COMPUTER SIMULATIONS OF SOUR WATER STRIPPINGGeneral Information

Number of wastewater stripping towers	2
Effluent concentrations	5 wppm H_2S 70 wppm NH_3

First Tower (hydrogen sulfide stripper)

Number of theoretical stages	13
Steam rate, pounds/gallon of fresh feed (kg/m^3)	0.6 (72)
No distillate condenser	
Includes reboiler	
No chemical addition	

Second Tower (ammonia stripper)

Number of theoretical stages	26
Steam rate, pounds/gallon of feed (kg/m^3)	1.5 (180)
Includes distillate condenser	
Includes reboiler	
Chemical addition, wppm NaOH	1,150

Note: A more detailed description of the sour water stripping system simulated here is available in the 1975-76 Commercial Plant Study Design (FE-2353-13).

sequence, and the increase in the allowable level of ammonia in the effluent from sour water stripping was calculated. When this new target was incorporated into computer simulations of the stripping process, no significant decrease in caustic requirements was found.

Future Work

Plans for the next six months include the following:

1. Continue characterization of wastewaters from process pilot units during Wyoming coal operations, in order to develop a data base for computer estimates of the quantity and quality of wastewaters from a commercial Wyoming plant.
2. Attempt to confirm computer modelling of coal liquefaction section wastewaters by sampling wastewaters from CLPP operations with reduced primary separator temperatures.
3. Attempt to confirm computer modelling of FLEXICOKING fractionator distillate drum wastewaters by sampling wastewaters from LSCU operations at reduced steam to feed ratios.
4. Continue with efforts to reduce the raw wastewater and contaminant loads from the EDS process.
5. Continue design case studies in order to choose the best wastewater treating system for pilot studies to be run in conjunction with the ECLP.
6. Reassess the current long-range waste R&D program to assure that the capability to design the wastewater treating system for a commercial EDS plant will be available by mid-1982. Specifically, reassess the costs, benefits and possible needs for a continuous wastewater treatment pilot unit.
7. If consistent with the revised long-range R&D program (6 above), begin continuous bench-scale testing to confirm the feasibility of the proposed treatment process sequence for a commercial plant.
8. Continue to monitor regulatory developments.

6.2 Environmental Control - Atmospheric Emissions

Air Pollution Control

Atmospheric emission controls must be an integral consideration in process development to insure that a commercial EDS plant will comply with environmental regulations in a cost effective manner. Major attention in Phase III-B focused on (1) assessing fugitive dust control from coal receipt, storage, crushing, and handling; and (2) determining concentrations of

potential toxic substances emitted from combustion of coal liquefaction products. These are not the only atmospheric environmental considerations, but are those most readily addressed at this stage of development. Subsequently, for example, fugitive gaseous emissions should be characterized.

Published factors and equations have recently become available to enable estimation of the extent of coal dust emissions, fallout, and dispersion, with and without control techniques. This literature will be useful for estimating the impact of EDS plants. It would be desirable to also use such estimation techniques to guide design decisions regarding control equipment needs. Unfortunately, the range of the estimates is still quite wide, in fact, too uncertain to be used for design/cost reduction purposes. In Phase III-B our objective was only to stay abreast of these developments, since quantitative estimates are not yet required.

Adequate control methods for fugitive coal dust are available, albeit at a cost. Surfactant sprays, foams, and crusting agents are required and are usually economic. Other methods involving greater costs are covered conveyors, careful design of transfer points to minimize fines production, ducted hoods, and bag houses. A judicious mix of these techniques is probably optimal in cost effectiveness. A plant trip to a power station of a Public Service Company, allowed a review of experience with some of these control methods and lead to the following conclusions and recommendations:

- Stringent maintenance and operating procedures must be followed. Dust monitoring instrumentation, shut-down interlocks, and alarms are not yet highly developed or widely employed.
- Ramps and chutes are preferable at transfer points. At the least, "free fall" distances should be minimized to decrease dust formation by crushing and entrainment into the surrounding air.
- Fire doors leading to passageways and other enclosed areas subject to high dust concentrations (50 gr/ft³) should be equipped with alarms since explosive ignitions have been conducted through open fire doors.

In Phase III-A, we identified those toxic substances that could be potentially emitted from EDS process. In Phase III-B, pilot scale tests were carried out to sample the combustion products from burning a blend of heavy coal liquid products. This blend was a simulation of the combination of liquefaction and FLEXICOKING liquid products expected from an EDS plant and consisted of 75% liquids in the 400-1000°F boiling range and 25% coker scrubber liquid (1000°F+). A small (50 HP) packaged Cleaver Brooks boiler was used to typify industrial combustion. As reported in the January-March 1978 Quarterly Report (FE-2893-12), combustion was reasonably efficient with 10% excess air. Sampling was done with the SASS sampling train, which has been specifically designed under EPA contract for sampling toxic substances. The fuel sample available limited the test to 160 minutes. The sample rate was 1.0 scf/min.

The particulate and adsorbed vapor-phase samples were analyzed for polynuclear aromatic compounds (PNA) by a gas chromatography/ultraviolet detection method. This method is notably very positive in its identification since it uses the ultraviolet absorption spectrum of each individual peak in the gas chromatographic separation. The sensitivity of this method, compared with gas chromatography/mass spectroscopy methods is under evaluation. Only five compounds were found above detectable limits, and one of these, fluoranthene, was present in much higher amounts than the others (by an order of magnitude). Of the others present above detectable limits, only chrysene is known to be mildly carcinogenic; pyrene, triphenylene, coronene, and fluoroanthene are not. However, the analytically detectable limits for a number of other compounds was too high to permit a valid comparison with the limited data available in the literature. In general, our own data fall within the range of previously reported concentrations. The limited literature information presently available is not consistent, and efforts have not been made to date in the literature to resolve differences in the reported concentrations.

Work is clearly required to resolve these issues. We are developing a program to do so.

Noise Control

Summary

The basic objectives of the study were to assess any noise problems which are not common to Exxon refineries and chemical plants. Based on the design information reported in the EDS Study Design and ECLP Design Specifications, all mechanical equipment items, and instruments/valves were investigated for potential noise problems. It was found that all the mechanical equipment items noise sources are not unique to the EDS process. These noise problems can be eliminated by using existing ER&E in-house technology. Regarding the instruments/valves, it was found that 10 high pressure let-down valves would be potential noise sources (see Table 6-5), quantified by using ER&E's in-house technology and vendor's prediction techniques. The noise level predicted for the gas control valve is considered reliable. However, there is some concern on the accuracy of the predicted noise levels for the valves in liquid service and in slurry service. Field data from ECLP will be collected to improve the prediction confidence. General control techniques on valve noise reduction have been reviewed and are briefly discussed.

In the second half of 1978, the impact of developing State and Federal noise regulations on the design and operation of the EDS plant will be assessed. It is anticipated that the new in-plant noise regulation will be promulgated by OSHA before the end of this year. This may affect the design and operation of the EDS commercial plant.

TABLE 6-5

HIGH PRESSURE LET-DOWN VALVE NOISE IN dBA AT 3 FEET

<u>High Pressure Let-Down Valve</u>		<u>Estimated Noise Level in dBA</u>			
<u>Valve No.</u>	<u>Service</u>	<u>ER&E</u>	<u>Fisher</u>	<u>Valtec</u>	<u>Masoneilan</u>
PCV-121	Gas	100	105 (88)	110 (85)	116 (88)
FCV-306	Liquid	131.5	-	-	92 ⁺ (85)
LCV-108	Liquid	113	-	-	-
LCV-109	Liquid	114	-	-	-
LCV-112	Liquid	113	-	-	-
LCV-310	Liquid	107	-	-	-
LCV-305	Liquid	107	-	-	-
LCV-110	Liquid	105	-	-	109 ⁺ (92)
LCV-304	Liquid	115	-	-	-
LCV-106	Slurry	127	-	-	-

* Numbers in parentheses represent noise levels for quiet valve designs.

- Vendors are not able to provide predicted noise levels.

+ Low confidence.

Analyses

There are one gas control valve, eight liquid control valves and one slurry control valve that will present noise problems. These valves are listed by their corresponding ECLP control valve numbers (Table 6-5). Also shown in the table are the valve noise levels estimated by using the prediction techniques of ER&E and the vendors. All calculations were based on standard wall pipes and valves because the design for the commercial plant has not been finalized. The noise level predicted for the gas control valve is considered reliable. But there is some reservation in the predicted noise levels for the liquid control valves and the slurry control valve. These are discussed in the next two paragraphs.

At the valves in liquid services, 30-50 wt% flashing is expected. There is no reliable ER&E technique for directly estimating liquid control valve noise levels associated with massive flashing. The noise levels caused by vapor and liquid were calculated independently. The total noise level is the sum of the two. This assumption ignores the interaction between vapor and liquid upon the control valve noise level. One control valve vendor suggested that the calculated noise levels may be high by 5 dBA because the flashing tends to damp the noise level.

There is no prediction technique for estimating the noise level of a control valve in slurry service. In addition to the slurry, which contains 11.6 wt% solids, the liquid carrier is expected to produce large scale flashing. ECLP is evaluating two valve designs for its slurry valve services: a stream-lined angle valve and a choke valve. These valve design configurations have been evaluated from a noise control view-point and it was found that they should behave like standard valves (i.e., not quiet designs). To estimate the noise generation, it was assumed that the valves are in 100% liquid service. Noise data from the operation of these valves are required during the ECLP start-up and plant operation phases.

In order to gain more understanding and possible field experience information in liquid and slurry control valve noise, we contacted PAMCO in Fort Louis, Washington, and the Southern Services Pilot Plant in Wilsonville, Alabama. It was found that there were no noise problems associated with control valves because of the small capacities in these two facilities. Also, there were no data of measured noise levels available. Three vendors were also contacted for valve noise evaluation. It was found that these vendors do not have reliable techniques to estimate noise levels of slurry control valves and liquid control valves which are operated in a flashing condition.

In view of the lack of experience and techniques in predicting the noise levels of slurry control valves and liquid control valves with massive flashing, noise data from existing coal liquefaction plants should be collected

to improve prediction confidence. This work can be conducted after the start-up of ECLP.

The noise control measures to reduce valve noise include in-line silencers, pipe lagging, and quiet valve designs. In-line silencers and pipe lagging are very effective, and can reduce noise level between 10 to 25 dBA depending on specific designs. Special quiet valve designs can provide noise reduction of 6 to 28 dBA. As can be seen in Table 6-5, noise levels of quiet valves are listed in parentheses for representative control valves. Presently available quiet valve designs cannot be used in slurry service because of plugging. However, continued effort is required to evaluate any developments in quiet valve designs for slurry services.

6.3 Development of Construction Materials For Coal liquefaction and Hydrogen Manufacture

Summary

1. Continue tests aimed at evaluating corrosivity of coal liquids. Series 3 and 4 in-situ corrosion tests have been completed in CLPP. The strong effect of sulfur compounds in coal liquids corrosivity has been verified via different coal types. Selected CLPP equipment components have been analyzed to determine potential erosion behavior. The ECLP Materials Evaluation Program has been updated and turned over to the contractor for detailed engineering.
2. Evaluate potential for erosion in liquid/slurry streams. Two areas of severe potential erosion requiring special materials and design features are letdown valves and slurry pumps. Detailed materials selections for the selected ECLP letdown valves have been reviewed and spare trims recommended. In addition, promising candidate materials for developmental ceramic trim have been identified and their properties investigated. Work with slurry pump manufacturers has yielded a design which combines replaceable erosion resistant materials and good hydrodynamic design features.
3. Maintain state-of-the-art materials technology in coal liquefaction processes. ER&E has taken an active part in the activities of technical bodies guiding and studying materials utilization and development in coal liquefaction and gasification. Among ER&E's major activities have been preparing a laboratory test program on coal liquids corrosion and presenting a paper on hydrogen attack.
4. Participate in corrosion testing and analytical work on aqueous streams from Integrated Coker/Gasifier (IKG) unit. The first series of IKG exposures has been completed and analyzed. Only carbon steel exhibited any significant corrosion. Additional tests have been initiated, including a study of gasifier slagging.

5. Review industry test programs and information on refractories for FLEXICOKING unit application to coal derived stocks. Slagging and deterioration of refractories in coal gasification environments have been investigated, principally via monitoring of DOE sponsored projects in coal conversion and utilization. Commercial experience from FLEXICOKING equipment is being accumulated and analyzed.
6. Explore practicality of high temperature erosion testing in simulated EDS FLEXICOKING gas-solids streams. A series of tests have been run on Lawrence-Berkeley Laboratory's erosion test equipment. Based on ER&E's visit, it is concluded that LBL's equipment can readily be adapted to investigate FLEXICOKING erosion.
7. Plan and interpret results of in-line corrosion tests on the Large Stirred Coking Unit (LSCU). Plans for conducting materials tests in the LSCU were abandoned due to equipment and process constraints.

1.0 Corrosivity of Coal Liquids

To investigate and develop data on coal liquids corrosion, a three-part, ongoing program has been in existence since the early part of Phase III-A, consisting of:

- CLPP In-Situ Corrosion Testing
- CLPP Component Analysis
- ECLP Materials Evaluation Program

1.1 CLPP In-Situ Corrosion Test Program

During April-September 1977, the third and fourth series of corrosion test samples (tubing sections and coupon specimens) were exposed in CLPP. Series 3 was exposed during operation with Wyodak coal; Series 4 reverted back to Illinois coal. The results from all four Test Series are tabulated in Table 6-6. Sample locations for all nine CLPP Test Sites are marked on Figure 6-2.

1.2 Corrosion Test Locations and Methods

Test Sites 1, 2, 4, 6, and 7 are in high temperature slurry services, Test Sites 8 and 9 in hot solids-free coal liquids, and Test Sites 3 and 5 in water condensates. Test Site 7 is in extremely severe erosive conditions downstream of the intermittent action pressure letdown valve. Test Sites 3 and 5 are the only ones employing standard corrosion rack coupons. The other seven test sites utilize tubing specimens (1/2"-3/4" OD) which are integrated into CLPP "piping runs", not as bypass loops.

TABLE 6-6

CLPP IN-SITU CORROSION TEST SUMMARY

Test Site (See Figure 1)	Test Series ⁽¹⁾	Type Coal	Test Specimens	Tube Dimension		Process Environment	Temperature (°F)	Pressure (psig)	Exposure Time (hrs)	Material	Corrosion Rate (mils/year) ⁽²⁾	
				Dia. (")	Wall (")						ΔThickness	ΔWeight
TS-1-1	1	Illinois	Tubing Sections	3/8	0.049	Solvent/coal slurry + H ₂	830	1650	282	304	93	28
TS-1-2				3/8	0.049		830	1650	282	316	93	94
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-2-1	1	Illinois	Tubing Sections	1/2	0.065	Solvent/coal slurry + H ₂	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	4
TS-2-7			1/2	0.065	770-820	1620	965	5 Cr	18	11		
TS-3-1	1	Illinois	Test racks Coupons			Liquefaction water condensate	60-100	2	282	CS	-	12.4
TS-3-1							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-4-2	122	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
TS-5-1	182	Illinois	Test racks 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				

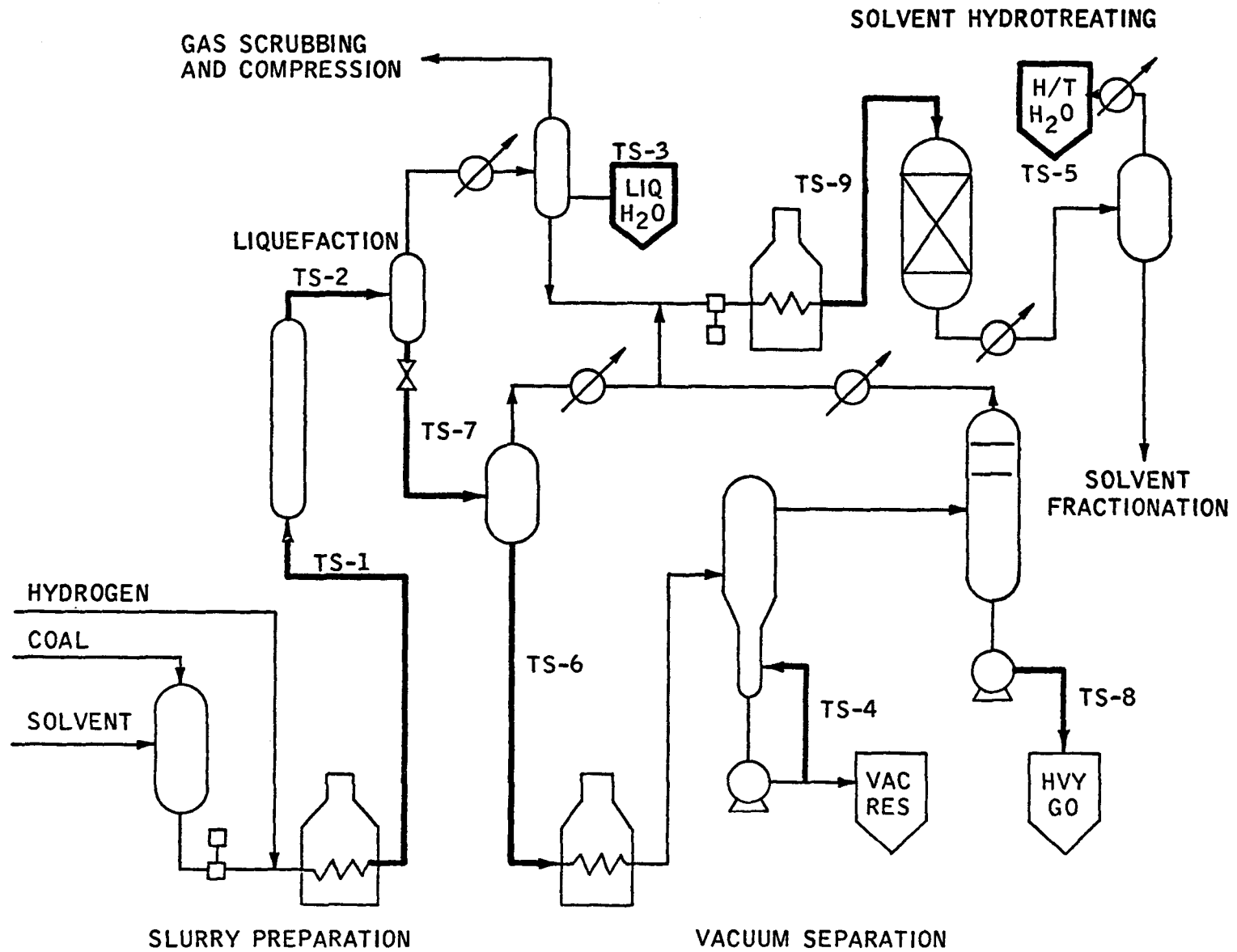
TABLE 6-6 (CONTINUED)

Test Site (see Figure 1)	Test Series (1)	Type Coal	Test Specimens	Tube Dimension		Process Environment	Temperature (°F)	Pressure (psig)	Exposure Time (hrs)	Material	Corrosion Rate (mils/year) (2)	
				Di.	Wall						ΔThickness	ΔWeight
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				1/2	0.035		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-6-9				1/2	0.035		700	25	180	12 Cr	0	2
TS-6-10				1/2	0.035		700	25	180	5 Cr	0	15
TS-6-11				1/2	0.035		600-700	25	170	CS	23	33
TS-6-12				1/2	0.035		600-700	25	770	304	0	10
TS-6-13				1/2	0.049		600-700	25	770	316	23	(+)
TS-6-14				1/2	0.035		600-700	25	770	12 Cr	0	0
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
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	650	1650	317	316	55	9
TS-9-2				1/2	0.065		650	1650	317	304	0	4
TS-9-4	2			1/2	0.065		650	650	747	5 Cr	35	30
TS-9-5				1/2	0.065		650	650	747	316	23	5
TS-9-6	3	Wyoming		1/2	0.065		650	1650	1203	304	5	3
TS-9-7				1/2	0.065		650	1650	1203	316	0	(+)
TS-9-8				1/2	0.065		650	1650	1203	5 Cr	9	12
TS-9-9	4	Illinois		1/2	0.083		650	1650	978	304	18	6

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

(2) (+) entries in "Corrosion Rate" column indicate measured gain in weight or wall thickness.

Figure 6-2
CLPP CORROSION TEST SITES



Corrosion (or erosion-corrosion) rates are determined by two methods -- thinning of the tube wall or weight loss per unit length. About 15% of the exposed tubing specimens registered an increase in weight or wall thickness; this is an indication of the large inherent error due to short exposure times and, more importantly, the tolerance spread in commercial tubing. Another consideration is the relatively short test duration, especially in environments where metal loss is small to begin with. Consequently, the results reported in Table 6-6 do not represent "hard" corrosion data, but rather a general indication of corrosion behavior.

1.3 Corrosivity as a Function of Coal Type

Corrosion rates obtained during Test Series 3 are quite low in comparison to results from Test Series 1, 2, and 4. The marked difference in corrosivity is evident from the data tabulation presented in Table 6-7. Relative metal losses are indicated by the "Corrosion Ratio", defined as the average corrosion rate with Illinois coal divided by the respective average corrosion rate with Wyoming coal. All but two of the 19 "Corrosion Ratios" are seen to exceed unity, with specific values ranging from 1.1-98. (1.1-41. for weight loss data).

The markedly disparate aggressiveness between Illinois and Wyoming coal is ascribed principally to differences in sulfur content. Illinois No. 6 coal contains 4.1 wt% sulfur vs. 0.5 wt% for Wyodak coal. Correlation between sulfur content and corrosivity has long been recognized in handling of crude oils and derived fractions at elevated temperatures.

Results from Test Sites 3 and 5 indicate lower corrosion in Wyodak coal than Illinois coal derived water condensates. As with hydrocarbon coal liquids, this is attributed to the effects of dissolved sulfides which are about an order of magnitude higher with Illinois coal. Condensate samples for TS-3 and TS-5 during Illinois coal runs ranged as high as 1.9 and 8.3% S=, respectively; corresponding levels for Wyodak runs reached only 0.1 and 1.0% S=, respectively.

The CLPP tests show corrosion to be quite mild even on carbon steel, notwithstanding the high amounts of dissolved sulfides. In contrast, sulfidic waters in petroleum refineries can be highly aggressive, especially under turbulent conditions.

1.4 Corrosivity as a Function of Metal Composition

The CLPP corrosion tests employed 8 different metals in 68 individual specimens (not counting duplicates in Test Sites 3 and 5). It is concluded that 18 Cr-8 Ni stainless steel has good corrosion resistance in all services (the high wastage rates in Test Site 1 are ascribed to slurry erosion). As expected, 18 Cr-8Ni stainless steel is far more

TABLE 6-7
COMPARISON OF CLPP CORROSION RATES
WITH ILLINOIS AND WYOMING COAL

Test Site	Material	Corrosion Rate (mils/year)								"CORROSION RATIO"	
		All Data Points				Weight Loss Data Only				Average mils/yr w/ Illinois Coal Average mils/yr w/ Wyoming Coal	
		Illinois coal ⁽¹⁾		Wyoming coal ⁽¹⁾		Illinois coal ⁽¹⁾		Wyoming Coal ⁽¹⁾		All Data Points	Weight Loss Only
		Range ⁽²⁾	Average	Range ⁽²⁾	Average	Range ⁽²⁾	Average	Range ⁽²⁾	Average		
TS-1	5 Cr 18 Cr-8 Ni	120-370[2] 28-94 [4]	250 77	63-64[2] 0.3 [4]	64 0.75	120 [1] 28-94[2]	120 61	64 [1] 0.3 [2]	64 1.5	2.5 98	1.9 41
TS-2	5 Cr 18 Cr-8 Ni	29-90[2] 0-27 [6]	60 4.5	11-18[2] 0-5 [4]	15 2.5	29 [1] 0-27 [3]	29 9	11 [1] 1-4 [2]	11 2.5	4.0 1.8	2.6 3.6
TS-3	CS 18 Cr-8 Ni Alloy 20	12 [1] 2.2 [1] 1.9 [1]	12 2.2 1.9	0.6 [1] 0.3 [1] 0.3 [1]	0.6 0.3 0.3	12 [1] 2.2 [1] 1.9 [1]	12 2.2 1.9	0.6 [1] 0.3 [1] 0.3 [1]	0.6 0.3 0.3	20 7.4 6.2	20 7.4 6.2
TS-4	CS	0-15[2]	7.5	23-150[2]	87	15 [1]	15	23 [1]	23	0.086	0.65
TS-5	CS 18 Cr-8 Ni Alloy 20	1.6 [1] 1.1 [1] 0.7 [1]	1.6 1.1 0.7	0.8[1] 0.4[1] 0.4[1]	0.8 0.4 0.4	1.6 [1] 1.1 [1] 0.7 [1]	1.6 1.1 0.7	0.8 [1] 0.4 [1] 0.4 [1]	0.8 0.4 0.4	2.0 2.7 1.8	2.0 2.7 1.8
TS-6	CS 5 Cr 12 Cr 18 Cr-8 Ni	190-250[2] 0-22 [4] 0-36 [2] 0-35 [4]	220 12 18 8.8	17-150[4] 0-15 [4] 0-2 [4] 0-240 [6]	56 5.5 0.50 45	190[1] 8-16[2] 36 [1] 0 [2]	190 12 36 0	17-33[2] 7-15 [2] 0-2 [2] 0-10 [3]	25 11 1.0 3.3	3.9 2.2 36 0.20	7.6 1.1 36 -
TS-7	5 Cr 18 Cr-8 Ni	0-2000[2] 0-3000[2]	1000 930	0-2000 [4] 120-970[4]	730 450	2000 [1] 0-900[3]	2000 300	920-2000[2] 120-340[2]	960 230	1.2 2.1	2.0 1.3
TS-8	5 Cr 18 Cr-8 Ni	30-35[2] 0-55 [8]	33 15	9-12 [2] 0-5 [4]	11 2.0	30 [1] 4-9 [4]	30 6.0	12 [1] 0-3 [2]	12 1.5	3.0 7.5	2.5 4.0

NOTES: (1) Illinois No. 6 coal data from Test Series 1, 2, & 4
Wyodak coal data from Test Series 3

(2) Bracketed figures indicate number of specimens exposed.

resistant than carbon steel and 5 Cr-1/2 Mo alloy steel. Straight chromium stainless steel (12 Cr) actually tested out better than 18 Cr-8 Ni SS in the single exposure evaluated (TS-6) but no conclusions can be drawn from such limited data.

Carbon steel and 5 Cr-1/2 Mo alloy steel appear to be suitable or at least marginally serviceable for most Wyodak derived coal liquids. 12 Cr would be expected to be intermediate between 5 Cr and 18 Cr-8 Ni; however, there are insufficient data to verify this point.

1.5 CLPP Component Analysis

To learn as much as possible from CLPP equipment components, several analyses have been performed. In particular, two potential high erosion services have been investigated. Our results can be summarized as follows:

- Sectioned carbon steel tubing in residuum pumparound circuit (Test Site 4) showed no evidence of erosion wastage after 180 hours at 1.9 ft/sec flow rate.
- Dimensional checks and metallographic analysis of vacuum flasher inlet line showed little if any evidence of erosion wastage after 750 hours at 150-250 ft/sec gas velocity.

1.6 Residuum Pumparound Slurry Erosion Evaluation

A section of carbon steel tubing in CLPP Test Site 4 (vacuum tower bottoms pumparound) has been destructively examined for evidence of slurry erosion. The tubing had been in service for 180 hours during Test Series 3 with Wyodak coal.

The sectioned tube showed no visible evidence of erosion markings. Since the calculated flow velocity is only about 1.9 ft/sec, the measured metal loss reported in Table 6-6 (TS-4-3) is therefore ascribed to corrosion rather than erosion.

1.7 Vacuum Flasher System Erosion Evaluation

Three sections of 304 stainless steel tubing used as the transfer line between the E/H-4 Preheater and the F-1 Vacuum Flasher were sectioned for dimensional checks and metallographic examination. Results from this examination together with observations on the Vacuum Flasher inlet zone were reported in Tables 6-21 and 6-22 of the Phase III-B Quarterly Progress Report, October 1 - December 31, 1977 (FE-2893-7).

Briefly, no evidence of erosion was found. The localized metal wastage on the Vacuum Flasher vessel directly at the feed inlet was caused by impingement which can be avoided by appropriate design features in larger installations.

1.8 ECLP Materials Evaluation Program

An extensive Materials Evaluation Program has been set up to collect corrosion data on alternate materials of construction and to identify areas and operating modes characterized by excessive metal loss for ECLP. This program consists of three parts:

- weight loss coupons via racks
- corrosion rate determinations via probes
- miscellaneous component tests

The basic test program was developed and incorporated into the ECLP Design Specifications during earlier phases of EDS development. The following is an update of the current status, incorporating changes and modifications completed during the reporting period.

1.9 Corrosion Racks

36 corrosion rack sites are specified in the ECLP Process Specifications. The range of candidate materials for test coupons are tabulated in Table 11-51 of Phase III-A Final Report, January 1, 1976 - June 30, 1977.* Details regarding test location, service environment, and test materials for each rack are summarized in Table 11-52 of the above referenced report.

Detailed design and fabrication of corrosion racks is being handled by the ECLP contractor. To assure a continuous test schedule, a minimum of 80 racks will be fabricated from an estimated 1700 coupon inventory. Coupon preparation, weighing, and rack assembly will be handled by ER&E. The racks are not designed for on-the-run removal because sufficient opportunities are foreseen for systematic rack removal/installation during downtime or sparing.

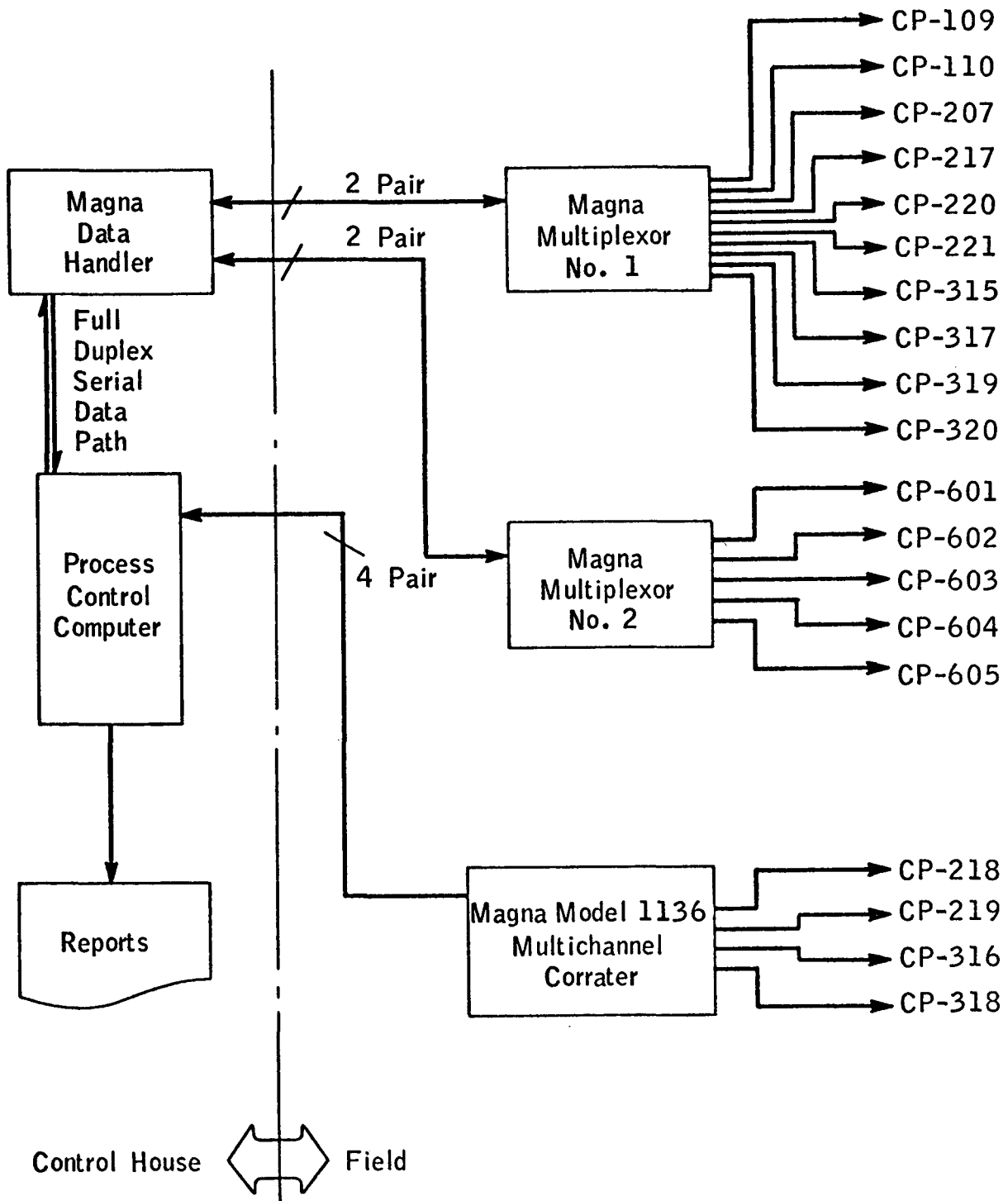
1.10 Corrosion Monitoring/Probes

The prime function of the corrosion probe program is to reveal, through its quick response characteristic, large, unexpected fluctuations in corrosion rates. The fluctuations, undetectable from time average weight loss coupon data, can arise from process related factors (e.g., type of coal) and changes in operating parameters (e.g., temperature). The system consists of three parts: probes/cables, data gathering equipment, and computer interface, as shown schematically in Figure 6-3.

*FE-2353-20

Figure 6-3

ECLP CORROSION DATA MONITORING SCHEMATIC DIAGRAM



Individual probe locations, types and environment for all 19 probes specified are tabulated in Table 6-8. Information for detailed engineering and procurement has been supplied to the ECLP contractor.

Both resistance and polarization corrosion monitoring probes have been specified; two types of resistance probes are utilized. Resistance probes operate by detecting a change in the electrical resistance of an element exposed to the corrosive environment as material is lost from the element. Polarization probes yield an instantaneous corrosion rate as opposed to the integrated material loss given by resistance probes.

1.11 Automatic Data Gathering

An integral part of the corrosion monitoring function is automatic data gathering (ADG). ADG is important for two reasons: rapid and accurate detection of corrosion incidents, and savings in manpower. The corrosion monitoring system for ECLP is not primarily designed to increase plant longevity and service factor, as with conventional systems. Rather, the principal objective is to validate materials test rack data, and help identify operating modes producing high corrosion losses.

1.12 Miscellaneous Tests

The purpose of the miscellaneous materials tests set up for ECLP is to collect materials performance data that cannot be obtained from racks and coupons. An updated summary of these tests is presented in Table 6-9. Selection of appropriate test tubes for exchangers E-108 and E-302 are not yet finalized because of limited availability of thick walled tubing. The intent is to test promising ferritic and semi-austenitic materials.

2.0 Erosion in Liquid/Slurry Streams

A review of Exxon and outside experience has identified two major areas of concern with regard to erosion and erosion-corrosion in liquid/slurry streams: these are letdown valves and slurry pumps. Letdown valve erosion can be minimized through utilization of special valve configurations and trim materials. Similarly, pump erosion can be controlled by special pump design features and materials selection.

2.1 Letdown Valve Trim Selection

Based on review of valve development programs and discussions with manufacturers, a two-phased approach has been developed. First, the selected valves were modified for optimum mechanical and hydraulic properties, and specified to be fitted with premium grades of cemented carbide trim. Second, developmental ceramic trim materials identified as the most promising candidates are being investigated with respect to mechanical, fabrication, and commercial factors.

TABLE 0-0
ECLP CORROSION PROBE SUMMARY

Probe#	Service/Location	Corrosion Probe			Comments
		Method (1)	Type (2)	Element	
<u>Slurry Drying & Liquefaction Section</u>					
CP-109	Recycle Gas Cold Separator H ₂ O (D-105)	Res	W40	CS	Slow
CP-110	Recycle Gas Water Scrubber Bottoms (T-101)	Res	W40	CS	Slow
<u>Product Distillation Section</u>					
CP-207	Distillation Condenser Outlet (E-209)	Res	T20	CS/304	Dry/Mist
CP-219	Distillation Drum Sour H ₂ O (D-201)	Pol	Corr	CS/316	Fast
CP-217	Reflux Condenser Outlet (E-203)	Res	T20	CS/304	Dry/Mist
CP-218	Reflux Drum Sour H ₂ O (D-206)	Pol	Corr	CS/304	Fast
CP-220	Vacuum Ejector Condensate (E-205)	Res	T20		Dry/Mist
CP-221	Vacuum Condensate Drum Sour H ₂ O (D-203)	Res	W40		Slow/Medium
<u>Solvent Hydrogenation Section</u>					
CP-315	Reflux Condenser Outlet (E-303)	Res	T20		Dry/Mist
CP-316	Reflux Drum Sour H ₂ O (D-305)	Pol	Corr		Fast
CP-317	Distillate Condenser Outlet (E-304)	Res	T20		Dry/Mist
CP-318	Distillate Drum Sour H ₂ O (D-306)	Pol	Corr		Medium/Fast
CP-319	Solvent Recycle Gas Water Scrubber Outlet (T-301)	Res	T20		Low severity
CP-320	Cold Separator Drum Sour H ₂ O (D-303)	Res	T20		Slow/Medium
<u>Gas Treating & DEA Regeneration Section</u>					
CP-601	Hot Rich DEA to Regenerator Tower (T-601)	Res	W40		Dry/Mist
CP-602	DEA Regenerator Overhead Condenser Outlet (E-604)	Res	W40		
CP-603	Regenerator Tower Bottom (T-601)	Res	W40		
CP-604	Regenerator Tower Reboiler Outlet (E-602)	Res	W40		Dry/Mist
CP-605	Fuel Gas Water Scrubber Sour H ₂ O (T-604)	Res	W40		

NOTES:

- (1) Res - Electrical resistance Pol - Polarization resistance
 (2) W40, T20 designate Magna Corrosometer type.
 Corr designates Magna Corratel type.

TABLE 6-9

MISCELLANEOUS MATERIALS TESTS

Location	Test Components
F-201 Vacuum Stripper Feed Furnace	• Thirteen 9 Cr spare tubes and return bends • Two 321 spare tubes and return bends • Two un-Alonized 9 Cr tubes and return bends for last tubes in radiant coil
E-203 Atmospheric Fractionator Overhead Distillate Condenser	• Two Monel test tubes • Two Incoloy 825 test tubes
E-209 Atmospheric Fractionator Overhead Distillate Condenser	• Two Monel test tubes • Two Incoloy 825 test tubes • Two titanium test tubes
E-103 Atmospheric Fractionator Bottoms Cooler	• Three carbon steel test tubes at hot end
E-104 S/H Bottoms Cooler	• Three carbon steel test tubes at hot end
E-108 Cold Separator Condenser	• Two AL-6X test tubes (Allegheny Ludlum) • Two 29-4 test tubes (Allegheny Ludlum) • Two 3RE60 test tubes (Sandvik)
E-302 Hot Separator Vapor Condenser	• Two AL-6X test tubes • Two 29-4 test tubes • Two 3RE60 test tubes
T-204 Vacuum Stripper Tower Feed Line at tower inlet	• One unlined 5 Cr removable test spool • One carbon steel removable test spool lined with metal fiber reinforced erosion resistant refractory castable
M-101 Slurry Drier Mixer	• Six different blade materials - CS, rubber, lined CS, 12 Cr, Stellite 6, Manganese steel, tungsten carbide faced CS
D-751 Sour Water Collection Drum	• Cement test site, 5 types of cement

2.2 Ceramic Trim Materials

Premium grades of hard specialty ceramics such as TiB_2 , SiC , B_4C , Si_3N_4 and Al_2O_3 have excellent erosion resistance but present serious mechanical and fabrication problems. To investigate the technical and commercial aspects of exotic trim material, an R&D proposal is being developed which will involve test installations in ECLP.

2.3 Slurry Pumps

26% Cr white cast iron such as HC-250 (Abex Corp.) has excellent erosion resistance but presents a safety problem because of sensitivity to mechanical and thermal shock. Working in conjunction with pump manufacturers, machinery and materials specialists circumvented this problem by limiting the use of HC-250 to internals, safely contained within a ductile steel casing. An added advantage of this approach is ease of replacement of these internals which are in contact with the erosive fluid.

2.4 Pump Test Programs Being Developed

Test proposals for evaluating slurry pump erosion have been received from several pump manufacturers. Based on these proposals, an EDS slurry pump program is being developed to investigate performance as a function of pump speed and materials, including specialty hard coatings.

3.0 State-Of-The-Art Materials Technology

Significant effort has been directed to keeping abreast of materials engineering developments in coal conversion processes. Major activities during this reporting period have been as follows:

- Active participation in Metal Properties Council (MPC) Subcommittee 9 (Materials for Coal Gasification and Liquefaction).
- Active participation in National Association of Corrosion Engineers (NACE) Committee T-12A (Coal Conversion and Utilization).
- Attendance at International Conference on Materials for Coal Conversion and Utilization.
- Follow-up of DOE and EPRI sponsored materials R&D through progress reports and personal contacts.

3.1 The Metal Properties Council--Subcommittee 9 (Materials for Coal Gasification and Liquefaction Processes)

In January 1978, ER&E accepted the chairmanship role of Phase Group VII (Materials for Coal Liquefaction Processes). This group has strong interest in both laboratory and in-situ test programs for studying corrosion and erosion by coal liquids and slurries.

3.2 Coal Liquids Corrosion Test Project

In Fall 1977, ER&E presented to the MPC a proposal for a comprehensive laboratory test program which is to be submitted for DOE sponsorship. A detailed work statement (Project Specification) has been developed and is being sent out for competitive bids.

The broad objectives of the proposed program are to identify the corrosive agents in coal liquids and to evaluate corrosion resistance of common steels as a function of temperature. Significantly, the test fluids are to be actual pilot plant coal liquids.

3.3 National Association of Corrosion Engineers

At CORROSION/78 symposium of NACE Committee T-12A, ER&E presented a paper entitled "High Temperature Hydrogen Damage in Petroleum Refinery Equipment". The purpose of the paper was to show the relevance of oil industry experience and practice to coal conversion plant design.

4.0 IKG Corrosion Testing

Experience with equipment in FLEXICOKING applications indicates potential materials/corrosion problems. To assess effect of coal derived feedstocks, corrosion tests are being conducted at the IKG Unit (Baton Rouge) running on CLPP vacuum bottoms.

4.1 Overhead Condensate Corrosion

Corrosion of alloys was studied by means of two corrosion test racks installed in the heater overhead scrubber. The racks were removed after 567 operating hours (1944 hours total exposure) with Illinois coal. Test results presented in Table 6-23 of the Phase III-B Quarterly Progress Report, October 1 - December 31, 1977, showed that only carbon steel suffered any appreciable corrosion. This finding is consistent with the alkaline pH and the quite low sulfide and chloride levels (3 ppm and 118 ppm respectively) dissolved in the overhead condensate. As shown in Table 6-10, a set of new specimens is currently under test in IKG's heater overhead system. Also being tested is a carbon steel tube installed in the Coke Gas Condenser.

TABLE 6-10

IKG CORROSION TEST STATUS (SERIES 2)

Test Site	Test Materials	Planned Installation Date	Comments
Heater Overhead Corrosion Test Racks (2)	CS 304 304 welded 316	February 1978	Installed February 1978
Coke Gas Condenser Tube	CS	March 1978	Installed April 1978
Gasifier Ring Specimens	310 800	March 1978	Installed May 1978

4.2 Gasifier Corrosion

Cursory inspection of the IKG gasifier showed the alumina ceramic liner and stainless steel metallic internals to be in good condition. Apparently the slagging incident was of too short duration to cause any damage. Early indications are that there is surprisingly little sulfur in the gasification product ($< 0.05\%$) which would have important corrosion implications in comparing coal vs. petroleum FLEXICOKING units.

To investigate corrosion and possible slag attack in the gasifier, a new test is being conducted at IKG. It utilizes ring type specimens of 310 SS and Alloy 800, slipped over thermowells above the gasifier grid.

5.0 Refractories for FLEXICOKING Units Employing Coal Derived Feed

Deterioration of refractories may be a greater problem with coal than petroleum derived feeds due to possibly higher slagging potential with coal liquids residue. In order to evaluate this unresolved question, experience and experiments in coal gasification and utilization environments are being investigated. Effort is concentrated on monitoring pertinent DOE/EPRI R&D programs, and on close surveillance of commercial FLEXICOKING equipment.

The broad conclusion emerging is that such limited slagging as could be tolerated from operational considerations, could be handled from a materials point of view. Premium refractories are available that could effectively resist local slag accumulations; however, no metals are as yet identified that possess adequate life.

More specific answers and guidelines could be developed if the Baytown prototype is recommissioned for EDS test runs. Appropriate materials engineering activities towards this objective are itemized in the Future Work Section.

6.0 High Temperature Erosion Testing

Several potential areas of high temperature erosion have been identified in EDS FLEXICOKING facilities. These areas include the gasifier and heater overheads, boiler feed water preheater, tertiary cyclones and heater overhead steam generators. Since materials selection cannot be based on existing information for this process, some form of testing is warranted.

Since test apparatus for running meaningful erosion tests is very costly, ER&E explored the possibility of conducting a joint test program with Lawrence Berkeley Laboratory. LBL have a special erosion laboratory which has been running various kinds of gas-solid tests since 1975. In

order to study the practicalities of such a joint program, a visit was made to LBL. During this visit, a series of 13 trial tests were conducted.

These runs confirmed that the LBL equipment was suitable for running high temperature erosion tests, and that the system is capable of feeding coal derived vacuum bottoms. All process variables such as temperature, velocity and solids loading can be varied to duplicate FLEXICOKING conditions. The only limitation of the equipment is that corrosive gas compositions cannot presently be duplicated. However, LBL are planning to modify the equipment during 1978 to accommodate reactive carrier gas compositions.

Future Work

All six of the active R&D projects described in this report are intended for continuation. In addition, it is proposed that several new projects be initiated in connection with the recommissioning of the Baytown prototype. The following summary of future plans for materials engineering R&D is for the next 6-12 months period, commencing July 1978.

1. Continue tests aimed at evaluating corrosivity of coal liquids
 - Series V exposure of CLPP test specimens (tubing) for additional data on alloy steels and effect of flow velocity.
 - CLPP reactor coupons to assess corrosion component in reactor slurry service.
 - Update ECLP Materials Evaluation Program to reflect latest experience and technology in coal conversion field.
2. Evaluate potential for erosion in liquid/slurry streams
 - Letdown valves--develop R&D proposal to evaluate ceramic trim materials.
 - Slurry pumps--develop R&D proposal(s) to evaluate abrasion resistant alloys and coatings.
3. Maintain state-of-the-art materials technology in coal liquefaction processes
 - Participate as panelist in Third Annual Conference on Materials for Coal Conversion and Utilization (October 1978).
 - Exert leadership in MPC Phase Group VII to activate coal liquids corrosion test program.
 - As Symposium Vice-Chairman, develop technical papers for NACE CORROSION/79 symposium on Materials Development & Coal Conversion.

4. Participate in corrosion testing and analytical work on IKG unit

- Evaluate Series 2 exposure of corrosion specimens currently under test in heater overhead system.
- Evaluate ring samples installed in IKG gasifier for evidence of corrosion and slagging.

5. Review of industry programs and information on refractories for coal derived FLEXICOKING units

6. Explore practicality of high temperature erosion testing in simulated EDS FLEXICOKING gas-solids streams

- Prepare R&D proposal for running tests at Lawrence Berkeley Lab.

New R&D Proposal

The recommendation to reactivate the Baytown prototype presents an opportunity for in-situ materials evaluation. Accordingly, it is proposed that materials engineering R&D projects be developed in the following areas:

- Materials Testing/Corrosion Monitoring Program--parallel to ECLP program.
- In-situ Erosion Testing--refractory evaluation in gas/solids streams.
- Gasifier and Heater Component Testing--grid caps and nozzles, instrumentation.

6.4 Mechanical Engineering Technology

The objectives of the mechanical engineering program for the period of July 1, 1977 through June 30, 1978, consisted of evaluating new and existing technology for block and check valves in slurry service and heavy wall pressure vessels and heat exchangers. The details concerning the selection of block and check valves for slurry service are contained in the January 1 - March 31, 1978 Quarterly Report (FE-2893-12).

Block Valves for Slurry Services

Six commercially available block valve types; namely, a ball valve, tapered plug valve, wedge type gate valve, through conduit type gate valve,

ram type valve and butterfly valve, were evaluated for their acceptability as block valves in slurry service. In addition, several prototype block valves, such as the choke valve, slide valve and lock hopper valve were also evaluated for their acceptability as block valves in slurry service.

Based on the data collected, it was concluded that the following valve types had the highest probability of providing a satisfactory operating life in slurry service.

- Trunnion mounted ball valve, see Figure 6-4.
- Lubricated tapered plug valve, see Figure 6-5.
- Through conduit type gate valve, see Figure 6-6.

In addition, it was concluded that the probability of the following valve designs providing an acceptable service life was either low or determinable only after in-plant testing.

- Wedge type gate valve
- Ram type valve
- Butterfly valve
- Metal seated floating ball valve

However, the construction details used in these valves indicate that they might be considerably more economical than the valves being proposed for this service. Therefore, if significant cost savings can be realized on commercial plant valve sizes, a few of these valves will be tested at specific locations in ECLP to establish the potential acceptability of these designs.

Check Valves for Slurry Service

Several types of commercially available check valves; namely, a swing check, ball check, lift check, duo check and tilting disc check, were considered for their potential acceptability as check valves in slurry service. It was concluded that for most slurry applications, a clearway type swing check valve would have the highest probability of providing an acceptable service life. However, in certain services, such as reciprocating pumps, an in-line ball check valve would be the preferred design.

Figure 6-4

TRUNNION MOUNTED BALL VALVE

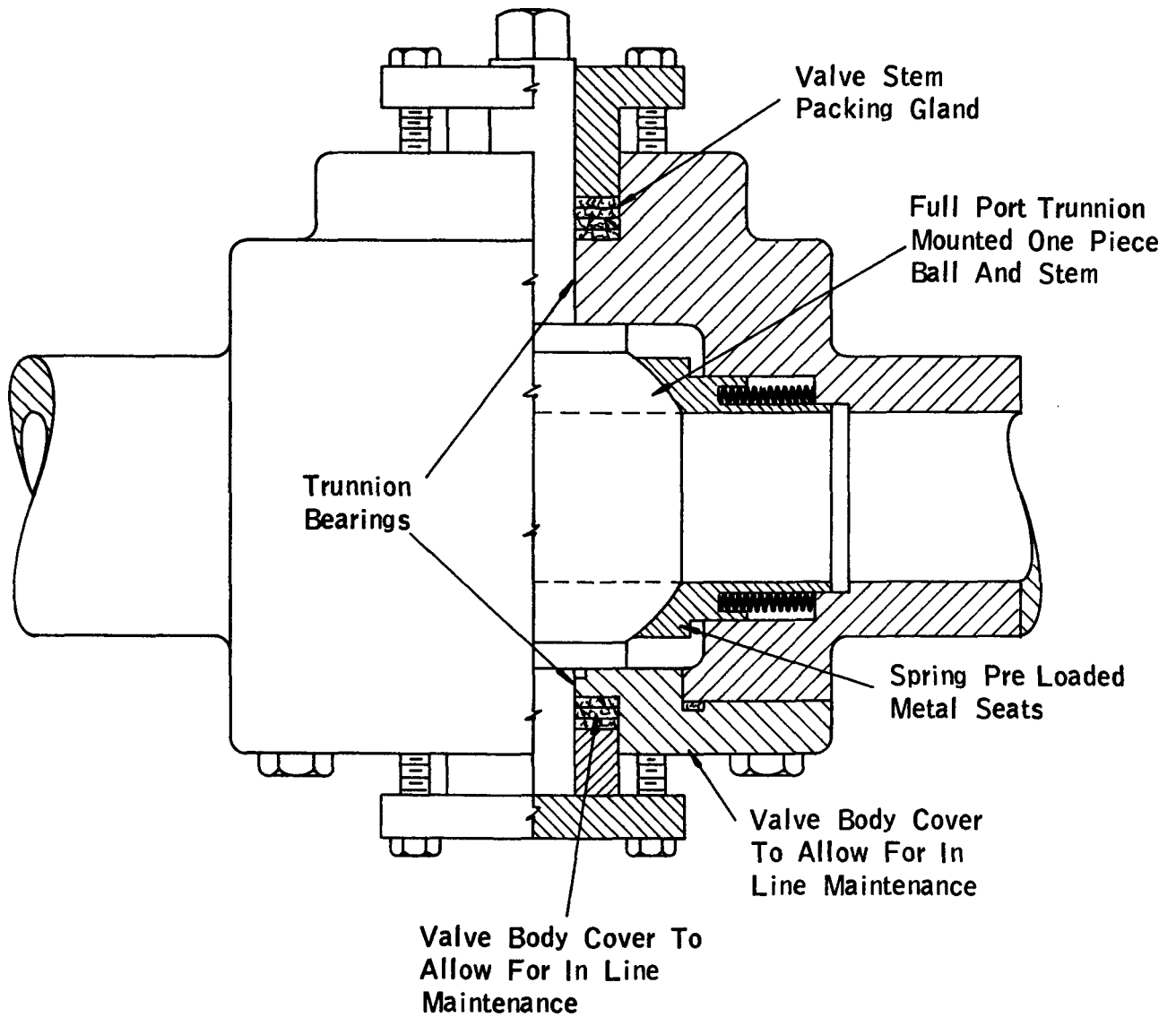


Figure 6-5

LUBRICATED TAPERED PLUG VALVE

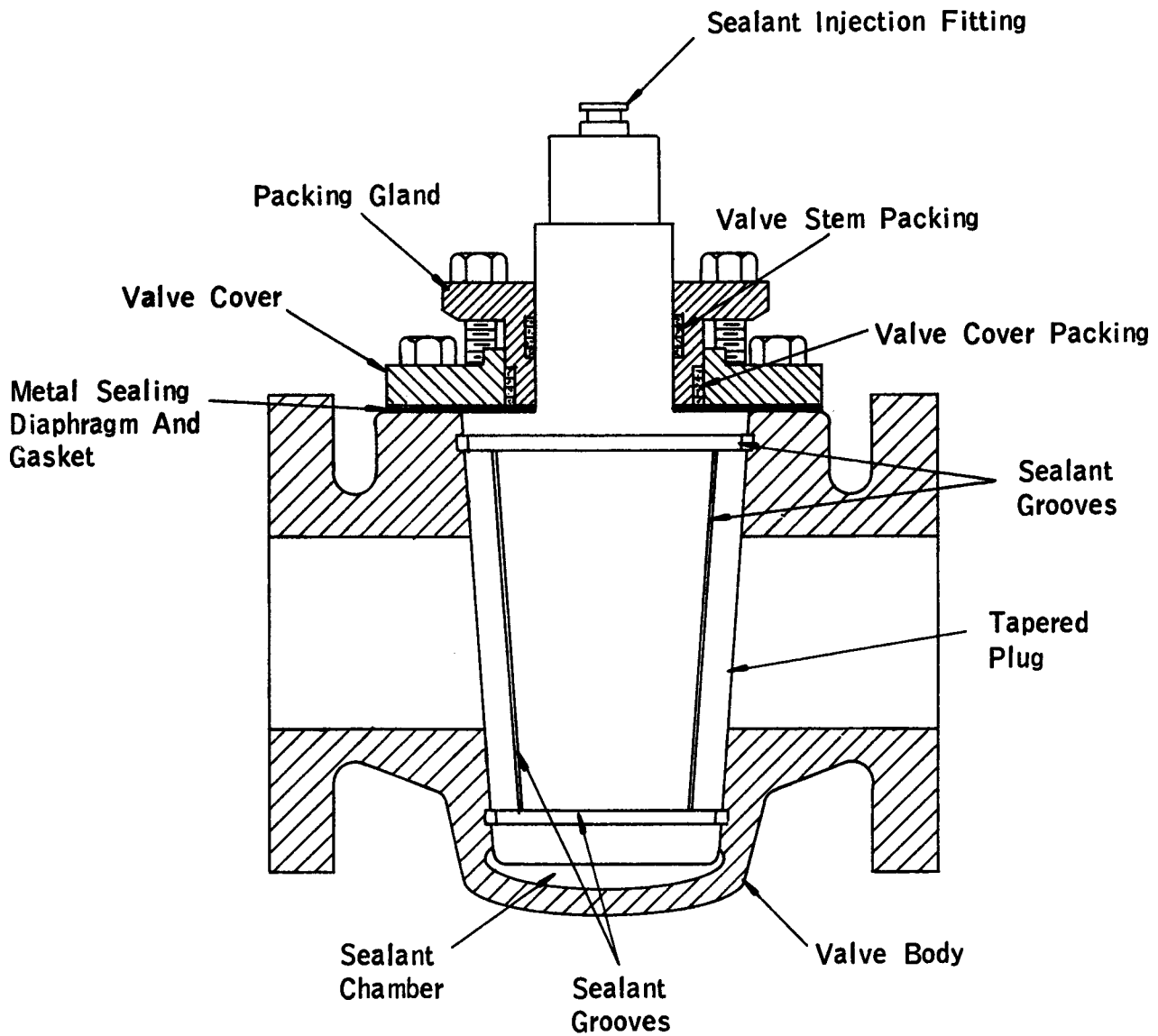
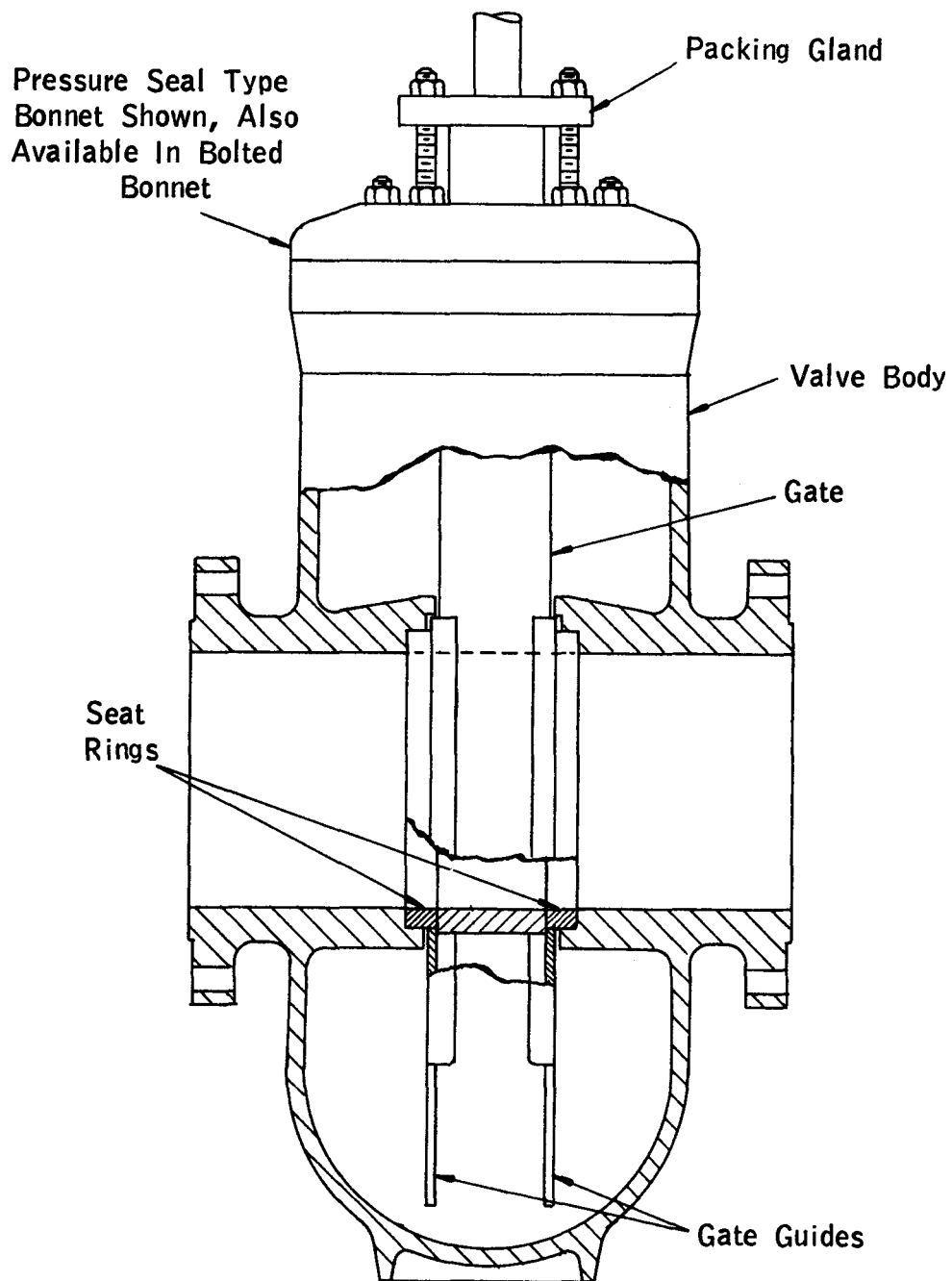


Figure 6-6

THROUGH-CONDUIT TYPE GATE VALVE



Future Work in the Area of Block and Check Valves for Slurry Service

The future work planned in the area of block and check valves for slurry service will focus on obtaining further experience in other DOE coal conversion pilot plants with the valve designs considered as having the highest possibility of providing satisfactory service life. In addition, continued followup is planned on several prototype block valves which are being developed for DOE to establish if there are any technological or economical advantages over present commercially available valves. If significant incentives exist, a few of these valves will be tested in ECLP.

Heavy Wall Pressure Vessels and Heat Exchangers

A major domestic heavy wall vessel fabricator made a presentation on pressure vessel design and fabrication at Florham Park. Discussed were the industries' current capabilities for both shop fabricated and field fabricated heavy wall pressure vessels. Acceptable nozzle and transition section details for heavy wall pressure vessels were also discussed. The information obtained from this presentation will be used to update our current requirements for the design, fabrication, inspection, and testing of heavy wall pressure vessels. Continued followup on heavy wall pressure vessel technology is planned.

6.5 Slurry Pumping

Preparation for ECLP Equipment Procurement

Discussions were held with centrifugal pump vendors to encourage them to prepare designs for the slurry pumping services specified in ECLP. A test pump position was added to the ECLP plant design in the coal slurry feed booster service with the identification "P-101E". The objective of this test pump is to determine the practicality of a single stage centrifugal pump handling feed slurry with a head requirement as high as 322 feet.

Several changes in material specification for the slurry pumps were developed, based on discussions with pump vendors. Pump casings cast in wear resistant hard-iron were judged acceptable for ECLP plant use up to 300°F. However, as discussed below under pump selection, this design was not finally selected.

Screw Pump for Vacuum Bottoms Service

The most severe slurry pumping service in the ECLP project was judged to be the vacuum fractionator bottoms service, P-210, because of the following difficult conditions:

- Pumping temperature - 775°F.
- Viscosity - 797 centipoises.
- NPSH available - 7 feet.
- Solids particles - coal up to 4 mesh and coke chips up to 1" x 1" x 1/4".
- Mechanical shaft seals are required to avoid external leakage of hazardous fluid. The seals must be of the double type because internal flushing is not allowed.

The pump type to be applied for this severe service is the rotary screw type, to be designed as follows:

- 2 rotors; external bearings and timing gears.
- Slow speed for increased wear life.
- Variable speed to permit operating at the lowest possible speed for each operating condition.
- Screw and bore surfaces with high hardness.
- Jacket heating and bearing cooling for high temperature service.
- Screw flights sized for the largest particles.
- Rotor-to-bore clearances optimized for service temperature and slurry characteristics.
- Flow direction selected to accommodate NPSH availability and to limit pressure at stuffing boxes.
- Cool sealing oil circulated between the double mechanical seals.

Maintenance difficulties must be anticipated with these pumps during the ECLP operations because:

- The pressure differential and operating temperature exceed vendors' prior experience with erosive slurries.
- The vacuum bottoms slurry tends to be more erosive than other slurry streams upstream in the plant.
- A total of 8 mechanical seals will be installed in the 4 stuffing boxes of each pump.
- Internal flushing cannot be used on the seals because flush oil would dilute the pumpage and risk atmospheric pollution when the stream is discharged for solidification at the vacuum bottoms conveyor.

ECLP Equipment Selection

The contractor's purchase recommendations were reviewed to insure basic conformance to the intent of the design specifications. Orders were placed with Worthington Pump Corporation for the reciprocating pumps in slurry service:

P-102 A&B	Coal slurry feed
P-204 A&B	Atmospheric fract. bottoms

Orders were placed with Lawrence Pumps, Inc. for the centrifugal pumps in slurry services:

P-101 A, B, C, D	Coal slurry feed booster
P-109	Slurry drier pumpout
P-206	Wash circulation

An order was placed with Worthington for a Sier-Bath pump for the one screw pump service:

P-210	Vacuum fractionator bottoms

Vendor proposals were developed and purchase recommendations were prepared for the two centrifugal test pumps:

P-101E	Coal slurry feed booster test pump 400 GPM)
P-216	Atmospheric fractionator bottoms test pump

The centrifugal pumps being supplied by Lawrence Pump, Inc. will be the same special construction style as will be used in P-101E and P-216, i.e., with replaceable hard-iron liners in steel, API-type casings. This new style was selected to allow greater nozzle loading by the suction piping than could be carried by the hard-iron casing construction which is standard for low temperature non-flammable slurry pumping.

Test Plan Development for the Centrifugal Test Pumps

The two basic aspects of centrifugal slurry pump design technology which will be developed by the test pumps, P-101E and P-216, and the Lawrence Model AL pumps are:

- The replaceable lining construction which will allow easy overhaul of the pressure casing, and with the safety of ductile steel to confine the flammable process fluid.
- The relationship of erosion rate of the pump impeller and casing lining parts to the relative velocities and rotating speed of the pump for various materials and slurry compositions.

The construction style aspects will be demonstrated and developed in the course of the mechanical design, manufacture, shop testing, and field maintenance of the pumps.

The erosion rate aspects will be established by testing the pumps at various speeds over the course of the ECLP operating period. The P-101C unit will be provided with an oversized motor (125 HP versus 50 HP for A, B, and D) and the capability to raise the head developed to twice the normal head.

The development of a specific plan for testing P-101, A, B, C, and D with various liner materials and at various speeds has been started, with Lawrence's participation. The vendor(s) selected for P-101E and P-216 will be invited to contribute to the development of similar plans for the testing program.

Hydraulic Motors for Slurry Letdown Service

An investigation was initiated on using a screw pump "running backwards" as a substitute for pressure letdown valves in slurry service. The pressure letdown service of primary interest is the letdown from the liquefaction reactor pressure level to the atmospheric fractionator pressure level. Screw type rotary pumps are commonly used as hydraulic motors in hydraulic power systems with clean, cool hydraulic oil. The slurry letdown service would be very severe, however, for a screw pump/motor because of the following conditions:

- Low viscosity (0.34 cp) at pumping temperature.
- High temperature (838°F).
- High pressure (1800-2500 psig at inlet).
- High vapor formation during pressure letdown (50-53% in outlet at 50 psia).
- Erosion due to leakage along the rotor-to-bore slip clearance due to the very high pressure differential.

Inquiries were made of three leading screw pump vendors. One response, negative, has been received.

FLEXICOKING Prototype Feed Pump Service Design Development

A design proposal was developed for a feed pump service for feeding ECLP vacuum fractionator bottoms to the Baytown FLEXICOKING prototype unit. The service design consists of a full capacity reciprocating pump as the normally operating pump, a second full capacity reciprocating pump as the normal standby, plus a full capacity rotary screw pump to serve as a backup spare and test pump. Permanent suction strainers will be required to protect the suction valves of the reciprocating pumps from large coke chips.

The design purposes of the spare screw pump are:

- To provide a ready standby pump during the time that one of the two reciprocating pumps is undergoing maintenance, to insure flow continuity to the FLEXICOKING unit.
- To provide suction booster capability for the reciprocating pumps if they experience NPSH difficulty at the high end of the feed viscosity range.
- To demonstrate the capability of a rotary screw pump as the primary feed pump for a commercial plant, with discharge pressure in the 300 to 600 psig range.

Technical proposals for this screw pump application were received from two vendors.

EDS Commercial Plant Design Update

Current recommendations were developed for the basic service design of the coal slurry feed booster service and the feed pump service for a commercial size EDS liquefaction plant. For the feed booster service, single stage centrifugal pumps with steel casings and replaceable hard-iron liners have the most promise. The most attractive choices for pump multiplicity will be two full capacity pumps or three 60% capacity pumps (two normally running in parallel).

For the feed pump service, plunger type reciprocating pumps are the most suitable type. The optimum multiplicity is expected to be four one-third capacity pumps.

Commercial Partial Oxidation Unit Feed Service Design Development

A design proposal was developed for a feed pump service to feed EDS vacuum fractionator bottoms to a partial oxidation unit in a commercial scale plant. Of special interest in the study were the limitations that very high viscosity might impose on the feed pump service design.

The service design recommended consists of booster pumps of the rotary screw type, and reciprocating pumps as primary feed pumps to develop the required 1000-2000 psig. Two or three screw pumps would be required, delivering a discharge pressure of about 60 psig. The best sizing and multiplicity of the reciprocating pumps would depend on whether a common or individual feed pump service would be provided for the four parallel lines of the partial oxidation unit.

This service design can handle viscosity up to 25,000 SSU in the EDS fractionator bottoms without special pump developments. Viscosity up to 80,000 SSU can be handled if special care is taken in the design of the reciprocating pumps, their inlet valves, and the suction systems of both the booster pumps and the feed pumps.

6.6 Instrumentation Evaluation

Program objectives for July 1, 1977 through December 31, 1977 included review of instrumentation hardware and systems used in DOE sponsored coal liquefaction pilot plants for the purpose of identifying potential problem areas, to confirm the suitability of previously recommended equipment, and to develop specific solutions for applications identified as particularly troublesome.

As a first step in our evaluation, a visit was made to the PAMCO 50 Ton/day pilot plant. Based on data obtained from this visit and DOE reports, our efforts to identify problem areas were directed toward:

- High pressure letdown valves in slurry service.
- Flow measurements in slurry service.
- Selection/development of needed process analytical instruments.

We also reviewed previously issued EDS process design specifications for ECLP to identify and prioritize those items that needed R&D work. Our work in problem areas is outlined below:

- High Pressure Letdown Valves in Slurry Service

Performance data obtained from several pilot facilities indicate that no proven high pressure letdown valve design exists for EDS application. This conclusion has been confirmed by the results of the Valve Workshop sponsored by DOE's Morgantown Energy Research Center (MERC). Based on our findings, we feel that development work on new valve designs is warranted. Unfortunately, within the time frame of EDS, this is not practical. Our recommendation is that existing valve designs must be used with little modification except for careful review and selection of valve materials.

The results of our evaluation on the valve that should be installed at ECLP and which affords the best chance of success with present technology have already been included in the design specifications. We have selected the Willis Choke valve and the stream lined angle valve (flow over the plug) as our first choices. These valves will be installed in parallel. The valves have been chosen based on extensive experience within process plants (for the streamlined angle valve) and oil well/field area (for the Willis Choke).

Technical discussions were also held with Control Components regarding the use of a DRAG valve in this service. The DRAG valve differs from conventional control valve designs in that it is designed to control the velocity of the flow stream through the valve, thereby minimizing erosion and acoustic energy output. However, the DRAG valve cannot be used in ECLP because the valve size is relatively small and the flow passages would be too small to pass the expected 0.125-0.250 inch size coal particles. The DRAG valve will be investigated further for use in a commercial coal liquefaction plant where valve sizes will be larger.

- Flow Measurement in Slurry

Work in this area has included review of available pilot plant experience. Venturi flow elements have been found to give satisfactory performance from the process and hydraulic standpoint and are the recommended flow element to use. The only problem has been one of instrument lead line plugging between the flow element (venturi) and the differential pressure sensor that sends an analog flow signal to board mounted instrumentation. We do not feel that this is a serious problem since appropriate detailed instrument piping design incorporating flushing oil to keep taps clear will solve the problem.

- Slurry Viscosity and Density Process Analyzers

Review of ECLP design specifications indicates that a vibration type viscometer will be used for Slurry Drier bottoms circuit viscosity measurement. This equipment may not be suitable for this service. This conclusion is based on experience with vibration type analyzers in petroleum and chemical process plants. Experience and theory demonstrate that vibration type analyzers are as sensitive to density as they are to viscosity and they sense only small volumes of fluid/slurry in the immediate vicinity of the probe, hence not yielding bulk property measurements. This is not critical in homogeneous fluids but becomes a matter of concern for the process condition in the slurry drier bottoms.

Cone and disc or cup and cylinder type viscometers would yield better results, but due to the abrasive nature of the process, these methods might be short lived. Additionally, stream sampling would be a problem.

Other instrumentation, including control of coal and solvent feed rates, is being relied on as the principal method of control for slurry drier bottoms composition. During ECLP operation, we will determine whether further development work is required in this area.

The major objectives for the January 1, 1978 through June 30, 1978 period are directed at high pressure slurry letdown valves. Work on letdown valves includes keeping abreast of new developments and incorporating the best available trim material and valve technology into ECLP.

- High Pressure Letdown Valve for Slurry Service Trim Selection

Trim components for these valves are subject to extreme conditions of erosion-corrosion, and therefore require exotic specialty materials. Based on limited coal conversion pilot plant experience, premium grades of cemented carbides (WC in Co-Chrome Matrix) appear to be the most durable materials. However, even these have a maximum service life measured in weeks or at best months. The design and fabrication of cemented carbide valve trim is a highly specialized skill. As a consequence, valve suppliers frequently subcontract trim fabrication to shops experienced in this area of technology. We have recommended this approach for the slurry letdown valves. Our trim recommendations are outlined below:

Streamlined Angle Valve Materials

1. Face material should be Kennametal 701. The upper plug material should be 15-5PH, with an erosion resistant coating.
2. The plug bushing should be Stellite 6. Surface to be coated with an erosion resistant coating such as TiB, SiC or Boron.
3. The seat material should be Kennametal 701.
4. The seat retainer outlet extension lining can not be fabricated in one piece. Several cylinders stacked to form the required length should be used instead. Individual cylinders should be fitted together with a machined groove and slot. The assembly should be held in place by compression placed on the end of the stack when installed flush with the bottom of the seat. This design allows individual cylinders to be exchanged as needed when liner cylinder sections break or wear out. We expect most of the erosive wear to occur on the first cylinder below the seat.

Choke Valve Materials

1. Choke valve trim materials should be Kennametal 701.

6.7 Oil/Water Interface Detection

The objective of this project to select the best commercial hardware for measuring the oil/water interface in separator drums has been achieved. Laboratory tests on CLPP samples indicated that capacitance probes would sense the interface between wet oil and oily water and that emulsions would not be a serious problem. Field reports from PAMCO and refinery operations of desalters confirmed that commercially available capacitance probes were working well in similar services. For these reasons, proposed 1978 tests have been cancelled and this project has been terminated. Implementation of the use of capacitance probes in ECLP will be handled on a normal engineering basis.

Laboratory tests on the CLPP samples consisted of mixing equal portions of oil and water in a Waring blender and measuring the capacitance of the separate wet oil and oily water phases. Values for the wet oil and oily water were found to be 0.3 and 480 microfarads respectively thus indicating a high selectivity for capacitance measurements. These tests also indicated that the oil (Specific Gravity 0.93) had low emulsion forming tendencies and that the phases separated rapidly (less than one minute).

Commercially available capacitance probes were found that met the measurement requirements for ECLP as well as the operational requirements of temperature and pressure. Further investigation found that the same probes had been recently used in similar applications at PAMCO and some of our refinery crude oil desalters.

ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,
PROCUREMENT AND CONSTRUCTION

1. Project Management

Contract Award/Planning

Reimbursable cost, fixed fee contracts were awarded on 16 September, 1977 to Arthur G. McKee Company and Daniel Construction Company to provide engineering/procurement and construction services, respectively, for the Exxon Coal Liquefaction Pilot Plant (ECLP). These contractors were selected from among the several bidders as those offering the best combination of business terms and execution potential.

Complete copies of the design specifications, vendors list and Exxon Engineering's (EE) Critical Path Schedule for the project were turned over to both contractors at contract award so work could begin immediately. An engineering kick-off meeting was held on 28 September with McKee and a joint McKee/Daniel meeting was held on 29 September to develop the initial coordination planning between the two contractors' project organizations.

The EE Project Management team began work in McKee's Cleveland office on 19 September, and the entire team was in place by 3 October.

Joint planning efforts by Exxon, McKee and Daniel project management groups during September and October, 1977 were directed toward achieving a mutually acceptable detailed division of responsibilities and developing work procedures. The following procedures manuals resulted from this combined effort:

- McKee/Exxon Procedures
- Daniel/Exxon Procedures
- McKee/Daniel Procedures
- Exxon Office Procedures

These manuals, distributed to all project team members, set the basis for the division of work, communications, team interrelationships, business practices, etc.

The Department of Labor issued a partial Wage Determination on 3 February to allow us to begin site development work as scheduled. The determination, based on previously published "Heavy and Highway" rates, includes "helper classifications for all major civil and mechanical crafts. The Wage Determination for all remaining construction was issued on 28 March. Labor classifications and minimum wages specified reflected practices for the process plant work and for all buildings. This will result in overall labor costs lower than our control budget which was based on higher scale wages and journeymen/helper ratios.

Quality Control

In November, 1977 an agreement was completed for EE's Quality Control Division and McKee's Inspection Department to share vendor plant inspection work for the project. Exxon will retain inspection on machinery, electrical equipment and instruments and all foreign source equipment and materials. Exxon also retained overall inspection responsibility and an Exxon inspection coordinator was added to the project management team to follow this critical activity.

A Positive Materials Identification Program was developed for the project with guidance from EE's Quality Control Division and Technology Department. Detailed implementation procedures for the program have been issued to McKee covering vendor fabricated equipment items, and to Daniel covering bulk materials and field/ shop fabricated piping. Exxon's project team inspection personnel will monitor the contractors' and fabricators' performance.

ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,
PROCUREMENT AND CONSTRUCTION

2. Engineering and Procurement

Basic Engineering

Exxon's design follow-up engineers, in addition to their normal consulting and advisory responsibilities, have handled, with McKee's help, a number of significant changes in the scope of work as originally defined by the design specifications for the project. For the most part these changes resulted from further study of problems which could not be fully resolved when the specifications were prepared in 1974/75. Principal changes to date are:

- Solvent Flushing and Blowdown System
- Naphtha Weathering/Sour Gas Drier/Tankage
- Standby Power Source for Slurry System
- Dowtherm System for Vacuum Bottoms
- Increased Data Logger Capabilities
- Sponsors Product Tankage and Loading Facilities

McKee has provided significant process engineering and drafting support, under the direction of Exxon engineers, to complete process designs and specifications for the above major scope changes and to prepare Change Lists to the design specifications.

Change Lists have been issued for the following Design Specifications:

● General Information & Instructions	CL5
● Slurry Drying and Liquefaction	CL5
● Product Distillation	CL5
● Solvent Hydrogenation	CL5
● Fuel Gas Treating	CL3
● Hydrogen Compression Facilities	CL2
● Sour Water Collection	CL3
● Chromatographic Analysis System	CL2
● Electrical Power System	CL2
● Tankage and Interconnecting Lines	CL1

Change Lists for eight specifications are in various stages of development; an additional four will probably not be revised.

Engineering

McKee's engineering and procurement work was 47% complete at the end of June, 1978, 8% or approximately 4 weeks behind our original plan; engineering per se was 51% complete. The Baytown Refinery Engineering Department's engineering work on the interconnecting lines was approximately 55% complete.

As indicated in the following table, McKee's projected engineering and procurement workload has increased since the start of the project. Primarily, the increase is related to the major scope changes previously mentioned, as well as a great many minor changes resulting from our increased experience/knowledge of the process and design modifications that normally evolve from detailed engineering studies.

<u>Date</u>	<u>Engr./Proc.</u> <u>% Complete</u>		<u>Projected</u> <u>Workload</u>		<u>Manning</u> <u>Equivalent Fulltime</u>	
	<u>Actual</u>	<u>Planned</u>	<u>k Hours</u>	<u>% Change</u>	<u>Actual</u>	<u>Planned</u>
Base	-	-	440.5	-	-	-
Dec. '77	12.6	12.5	442.6	.5	133	133
Jan. '78	17.8	19.4	449.8	2.1	160	163
Feb.	23.0	26.0	469.1	6.5	190	191
March	29.0	33.0	484.6	10.0	195	195
April	35.0	38.0	497.9	13.0	215	205
May	40.0	45.0	537.0	21.9	230	205
June	47.0	55.0	538.9	22.3	229	205

Physical progress in the various engineering discipline areas as of the end of June, 1978 is summarized below.

<u>% Complete</u>		<u>% Complete</u>	
● Process	94	● Mechanical	77
● Piping	43	● Instruments	38
● Civil	56	● Materials Handling	48
● Electrical	34		

Following the review of McKee's onsite plot plan drawings by EE and Carter operations and maintenance representatives in mid-December, 1977, McKee's piping group began work on the engineering piping model. The equipment location model review of the liquefaction, hydrogenation and gas treating sections of the plant was completed in early April, 1978. The compression section, furnaces, coal preparation facilities and auxiliary systems were reviewed in mid-June. The objectives of these first model reviews were:

- to finalize equipment and structure locations/spacing
- to assess safe access for routine operations and maintenance
- to review provisions for equipment lifting/handling and maintenance "laydown" areas
- to insure that emergency escape routes were adequate
- to review firefighting facilities

Final piping model reviews are planned according to the following schedule:

	1978 Week of -
• Fuel Gas Treating/DEA Regeneration	24 July
• Hydrogenation/Slurry Drier/Utilities	14 August
• Compressors (Partial)	4 Sept.
• Compressors/Coal Preparation	2 Oct.
• Liquefaction/Distillation	9 Oct.

Based on Carter's preliminary startup plan, we expect to turn over the plant in fourteen packages, beginning seven months ahead of over-all plant mechanical completion with the fire protection system, and progressing in sequence through utilities, coal handling and the onsite areas. The liquefaction and distillation areas will be the last to be handed over. Work is underway to provide Carter with a precise definition of each area turnover package by means of marked up P&I diagrams. Their review will define startup piping needs and any additional flanges and valves that may be needed to isolate the various areas so startup operations can be safely conducted before the entire plant has been completed.

Procurement

Inquiries have been issued for 469 (99%) of the 470 tagged equipment items required for the plant. Quotations have been received for 458 items, and 441 items have been purchased. Commitments for all procurement totalled 18.5 M\$, 59% of the forecasted total for the project vs. 65% planned (see Figure 2-2).

A summary of procurement related to tagged equipment items through the end of June, 1978, follows.

	Engineering <u>Requisitions</u>	<u>Equipment Items</u>			
		<u>Total Required</u>	<u>Inquiries Outstanding</u>	<u>Proposals Received</u>	<u>Orders Placed</u>
Cooling Tower	1	1	-	-	1
Compressors	6	13	-	-	13
Drums	8	58	2	5	51
Exchangers	11	56	1	-	55
Furnaces	2	6	-	1	5
Pumps	15	127	4	2	121
Reactors	2	8	-	-	8
Tanks	3	15	-	-	15
Towers	3	15	-	-	15
Other	<u>40</u>	<u>171</u>	<u>4</u>	<u>9</u>	<u>157</u>
	91	470	11	17	441

Orders totaling 4.4 M\$ have been placed for the following bulk materials.

	<u>Cost, k\$</u>		
	<u>Total Projected</u>	<u>Committed</u>	<u>% Committed</u>
Pipe/Fittings	2,863	1,099	38
Valves	3,151	271	9
Instruments	2,681	569	21
Electrical	1,977	855	43
Steel	<u>2,197</u>	<u>1,601</u>	<u>73</u>
Total	12,869	4,395	34

Included in orders placed through June are those to foreign suppliers as noted below; all others are from domestic sources.

Liquefaction and Hydrogenation Reactors	Japan Steel Works	Japan
High Pressure Heat Exchangers	Nuovo Pignone	Italy
Screw Pumps	Worthington	Canada
Valves (partial)	Kitazawa	Japan
Valves (partial)	Walworth Intl.	Italy

The search for satisfactory sources of supply and the selection of vendors for equipment and materials to meet the varied and unusual conditions of service, (i.e. high pressure, hydrogen, erosive slurry and high corrosion rates) has required a disproportionate amount of technical and procurement services. Some of the more difficult and time consuming items have been:

- Low and High Pressure Slurry Pumps
- Slurry and Special Valves
- Mix Points and High Pressure Letdown Vessels
- Gas Swept Mill
- Special Pipe and Fittings
- High Pressure Reactors
- Slurry Feed Furnaces

Cost

Exxon Engineering's Cost and Schedule Division completed the Class II control cost estimate update on 25 November, 1977. Reflecting the estimate update and including all approved, pending and anticipated changes at that time, the cost outlook remained at 110 M\$ as earlier forecasted.

As shown in Table 2-1, the cost outlook for the project as of 30 June, 1978, is 104 M\$. Major factors contributing to the decrease in forecasted costs were:

	<u>k\$</u>
Original Cost Outlook	110,000
Materials	(3,893)
Subcontracts	(626)
Labor and Construction Indirects	(1,506)
Engineering	(125)
Added Baytown Refinery Work	403
Exxon Engineering Services/Owners Expenses	(324)
Items in Original Cost Outlook Subsequently Covered by Formal Changes	(1,470)
Contingency Rundown	(2,656)
Approved, Pending and Anticipated Changes	<u>4,222</u>
Total	104,025

One hundred thirty-one Change Orders were written through 30 June, ninety-nine of these changes have been approved at a net reduction of 713 k\$. McKee is currently estimating twenty-nine pending changes with an expected cost of about 2,140 k\$. Three field changes expected to cost 55 k\$ are being estimated by Daniel. Summaries of approved, pending, and anticipated changes are shown in Tables 2-2, 2-3, 2-4, respectively. The number of changes continues to have a significant impact on the engineering and construction schedules, as well as the cost outlook. This high rate of changes is graphically shown in Figure 2-1 which compares the cost of approved and pending changes, ex budget shifts, to the contingency rundown.

A recent analysis indicated that design basis (scope) changes have accounted for 54% of all changes, while engineering design development changes came to 37% of the total. Estimate adjustments and miscellaneous changes totaled 9%.

Schedule

As mentioned previously, the EE critical path schedule was provided to both contractors at the time contracts was awarded. The schedule was used to guide the contractors' early activities until such time as a jointly prepared contractors' schedule was available. The contractors' critical path schedule was completed on 18 November, 1977, 60 days after contract award as called for in our agreements. While this first schedule study indicated a modest improvement in the mechanical completion date to be possible, we retained our original target date of 15 November 1979 for planning purposes.

The latest update of the overall project critical path schedule, which was completed in mid-June, indicates that a 15 November 1979 plant mechanical completion can still be achieved. However, because of the substantial increase in the number of critical and near critical activities indicated by this update, especially in the onsites and coal preparation areas, and the increase in the engineering workload because of recent scope changes, we believe it is probable that some overall schedule extension will be indicated. If it is determined that an extension is indicated, we expect that it will be modest - on the order of one to two months.

We are currently reviewing McKee's plans and schedule for final piping model reviews and piping isometric production to insure that the original milestones for these activities can be reached or bettered. The results of these efforts, plus Daniel's update of their detailed construction plans, and the latest delivery information on critical items of supply will be reflected in the next project critical path schedule update slated for completion in mid-August, 1978. This will be the most meaningful update since the original schedule was prepared.

Control charts to monitor the contractors' progress are shown in Figure 2-2. Target physical progress curves, with standard deviations (as control limits) based on historical experience, are plotted. Actual progress, as of 18 June, is shown.

TABLE 2-1

EXXON COAL LIQUEFACTION PILOT PLANT
PROJECT COST SUMMARY THROUGH JUNE 30, 1978

	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\$
McKee & Daniel							
Direct Material	38,100	34,062	2,271	36,333	31,596	4,737 (-)	15,805
Direct Labor	12,370	11,604	(1,204)	10,400	9,467	933 (-)	90
Subcontracts (4)	4,810	8,305	(1,925)	6,380	5,724	656 (-)	2,929
Field Indirects (5)	19,910	19,030	(659)	18,371	16,490	1,881 (-)	1,940 (1
Fee (6)	2,000	1,323	49	1,372	1,372	-	424
Engineering (7)	13,610	12,500	755	13,255	12,670	585 (-)	5,111
Loss on Surplus	460	490	-	490	490	-	-
Insurance	250	600	-	600	600	-	461 (1
Pending Changes (8)	-	-	2,195	2,195	2,195	-	-
Total Prime Contract	91,510	87,914	1,482	89,396	80,604	8,792 (-)	26,760 (1
Baytown Refinery Costs (9)	-	-	3,187	3,187	3,045	142 (-)	-
Anticipated Changes	180	-	2,740	2,740	2,740	-	-
ER&E Services (10)	4,200	4,200	(421)	3,779	3,800	21 (+)	1,365 (1
Owners Costs (11)	1,930	1,930	206	2,136	2,006	130 (-)	625 (1
Project Contingency	10,180	9,486	(2,321)	7,165	6,830	335 (-)	-
Process Development Allowance	2,000	5,000	-	5,000	5,000	-	-
Total Project Cost	110,000	108,530	4,873	113,403	104,025	9,378 (-)	28,750

See next page for notes.

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 30 June, 1978.
- (4) Major subcontracts include tankage, piling, coal silo, cooling tower erection, railroad spur, control house, 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. Includes cost for increase in 600# steam line and waste water tie-in change.
- (10) The Revised Control Estimate reflects a transfer of the cost of inspection services to McKee.
- (11) Includes: Administration and General Purpose Buildings, communication system, power hookup to Houston Power and Light, additional site borings, and the encasement of Texas Eastern, Diamond Alkali, and Houston Pipeline Co. pipelines, and a transfer of the data logger programming costs from the McKee material account.
- (12) Includes commitments through 16 June, 1978.
- (13) Includes commitments through 30 June, 1978.
- (14) Includes expenditures through 31 May, 1978.
- (15) Decreased commitments from last month's report to reflect actual commitments. Last month's report considered the total budget for burdens, travel, home office expenses, etc. as fully committed.

TABLE 2-2
EXXON COAL LIQUEFACTION PILOT PLANT
SUMMARY OF APPROVED CHANGES
THROUGH JUNE 30, 1978

<u>Change No.</u> <u>OCR</u>	<u>Description</u>	<u>Type*</u>	<u>Cost k\$</u>	<u>Schedule Effect</u>
1 thru 49 51 thru 65 67 thru 69 71 thru 78 81, 84, 87, 89, 90, 94, 96, 100, 101, 106 EFCR 1 thru 4	Eighty-nine Previously Approved/Cancelled Changes		(1,721.5)	None
50	Change List #1 to D.S. 74-126	DB	801.4	None
79	Add Naphtha Weathering Equipment to Fuel Gas Treating and Offsite Areas	DB	(34.3)	None
83	Add Condensate KO Drum and Make High Pressure Sample Outlet Changes	DD	12.0	None
91	Apply a Tungsten Carbide Overlay to Pump Shafts P-210 A-C and P-101D	DD	Cancelled	None
92	Incorporate Model Review Comments Into the Design	OT	95.9	None
93	Incorporate Carter Oil Comments Into Control House Design	DB	50.8	None
95	Include Guidelines for Level Gauge Glasses	DD	29.1	None
98	Add Digital Plant Status Inputs to Datalogger	DB	19.7	None
102	Add Metal Detectors to CO-1101B and CO-1102	DD	20.7	None
103	Revise Track Hopper Pit Sheet Piling Waler Design	DD	13.5	None
Total Negative Approved Changes			(4,122.6)	
Total Positive Approved Changes			3,409.9	
Grand Total Approved Changes			(712.7)	

*DD - Design Development; DB - Design Basis; EA - Estimate Adjustment; BS - Budget Shift; OT - Other

TABLE 2-3
EXXON COAL LIQUEFACTION PILOT PLANT
SUMMARY OF PENDING CHANGES
THROUGH JUNE 30, 1978

Change No. OCR	Description	Type*	Execution Authority Given	Cost k\$ (Order of Magnitude)
66	Add Pollution Abatement Devices to Meet TACB Requirements	DB	No	50
70	Incorporate Instrument Guidelines #1, #2 and Other Miscellaneous Instrument Items	DD	Yes	200
80	Miscellaneous Offsite Changes to Flare Header and P-1701 A/B	DD	Yes	100
82	Provide Reliable Energy Source for Critical Drivers	DB	Yes	400
85	Add Flushing and Blowdown System to the Job Spec	DB	Yes	300
86	Add Corrosion Probe Data Gathering and Computer Interfacing System	DD	Yes	50
88	Provide Pump Seal & Flushing for Environmental Control	OT	No	15
97	Provide Reversible Screw Conveyors Under Gas Swept Mill Cyclones	DB	Yes	50
99	Cost for McKee to Adopt Positive Materials Identification Program	OT	Yes	15
104	Change List #2 to D.S. 74-119	DB	Yes	N11
105	Make Modifications to D-103	DD	Yes	40
107	Incorporate Changes to the Steam System	DD	Yes	300
108	Add Paving and Sewers in the Coal Prep Area	DB	Yes	50
109	Add Monorails and Hoists Where Required	DB	No	100
110	Revise Dust Collector DC-1001 & 1101 Size	DD	Yes	20
111	Guidelines for Block Valve Selection	DD	Yes	30
112	Add Chlorinator Enclosure for Sun Protection	DD	Yes	20
113	Add Pneumatic Interlocks to F-116 - Solids Withdrawal System	DD	Yes	N11
114	Revise Datalogger Inputs and Add Associated Instruments	DD	No	100
115	Convert P-1801 to a Steam Driven Turbine	DD	No	20
116	Add Elevator to Prepared Coal Bin Structure	DB	No	80
117	Add Elevator to Vacuum Stripper Structure	DB	No	90
118	Install Two Separate Lines from Slurry Letdown Valves to T-201	DD	No	50
119	Incorporate Onsite Change Lists to the Job Spec	DB	Yes	50
120	Changes Resulting from Area 300 P&ID Review	DD	Yes	30
121	Delete Gas Swept Mill Building and Add Shelters Where Required	DB	Yes	(80)
122	Add Gay Engineering Thermowell Leak Detection in 1500 psi and Erosive Services	DD	No	10
123	Relocate P-204 A/B with Suction Ends Toward T-201	DD	Yes	50
124	Budget Shift Waste Water Line to Baytown Refinery	BS	Yes	N11
Subtotal Office Changes (OCR's)				2,140

TABLE 2-3 (Cont'd)

<u>Change No.</u> <u>EFCR</u>	<u>Field Changes</u>	<u>Type*</u>	<u>Execution Authority Given</u>	<u>Cost k\$ (Order of Magnitude)</u>
5	Add Water Hose Connections to Administration Building	FC	Yes	5
6	Make Repairs to Baker Road	FC	Yes	20
7	Extend Access Road and Accessway Shoulders for Safety	FC	Yes	<u>30</u>
Subtotal Field Changes (EFCR's)				55
Total Negative Pending Changes				(80)
Total Positive Pending Changes				2,275
Grand Total Pending Changes				<u>2,195</u>

* DD - Design Development; DB - Design Basis; EA - Estimate Adjustment; BS - Budget Shift; OT - Other; FC - Field Changes

TABLE 2-4

EXXON COAL LIQUEFACTION PILOT PLANT
SUMMARY OF ANTICIPATED CHANGES

	<u>k\$</u>
Additional Noise Control	+ 100
Alternate Scheme to Vacuum Bottoms Handling	+ 500
Daniel Positive Materials Identification Program	+ 80
Impact Test of Compressor Discharge Dampeners	+ 90
Provide Additional Flanges and Valves for Turnover Packages	+ 50
Add Slop Re-run Facilities	+1,000
Increase Solvent Flushing Facilities	+ 700
Add Coal Sampling Facilities	+ 20
Incorporate Model Review Comments	+ 100
Miscellaneous Changes to be Written Later	<u>+ 100</u>
	+2,740

FIGURE 2-1

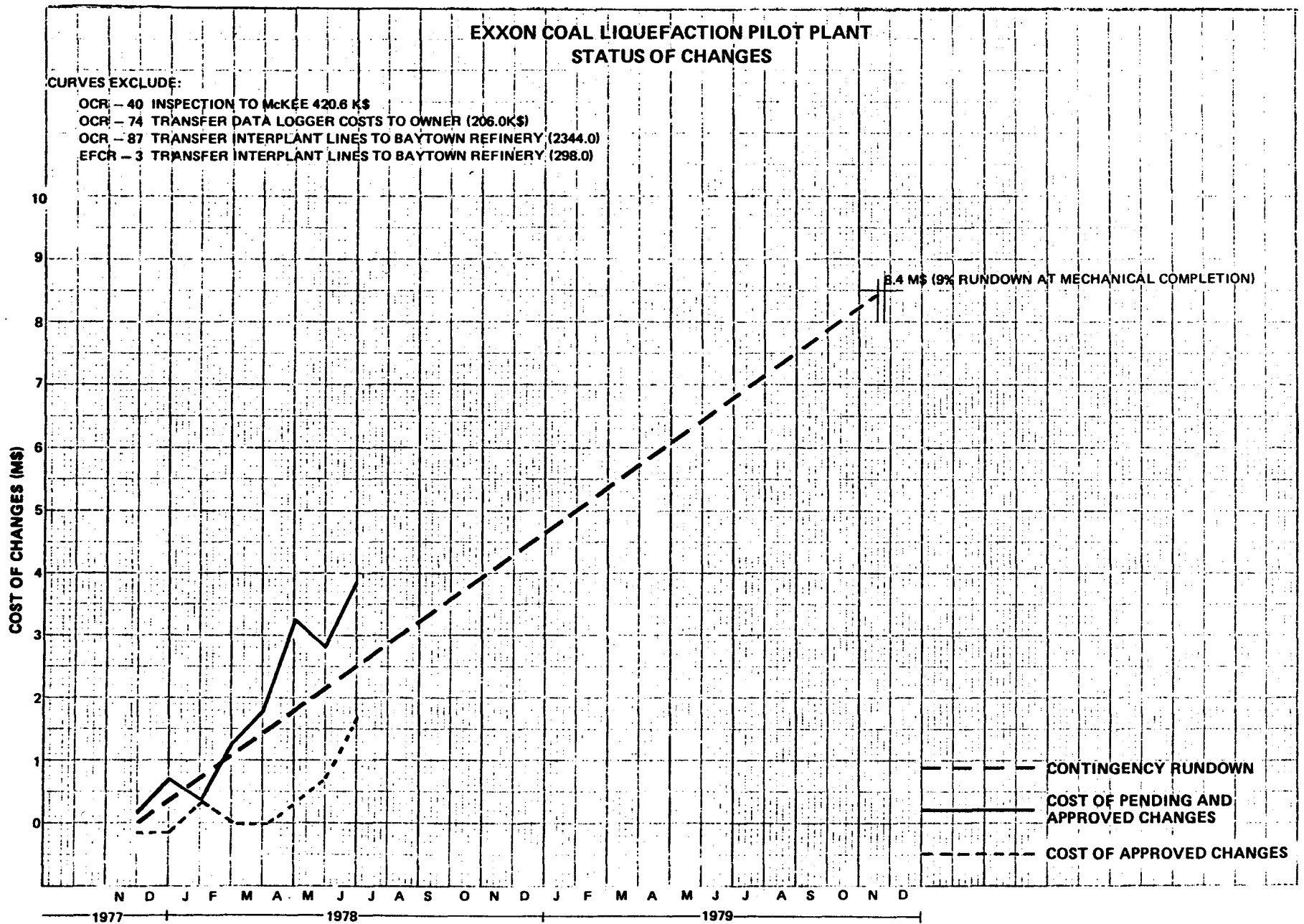
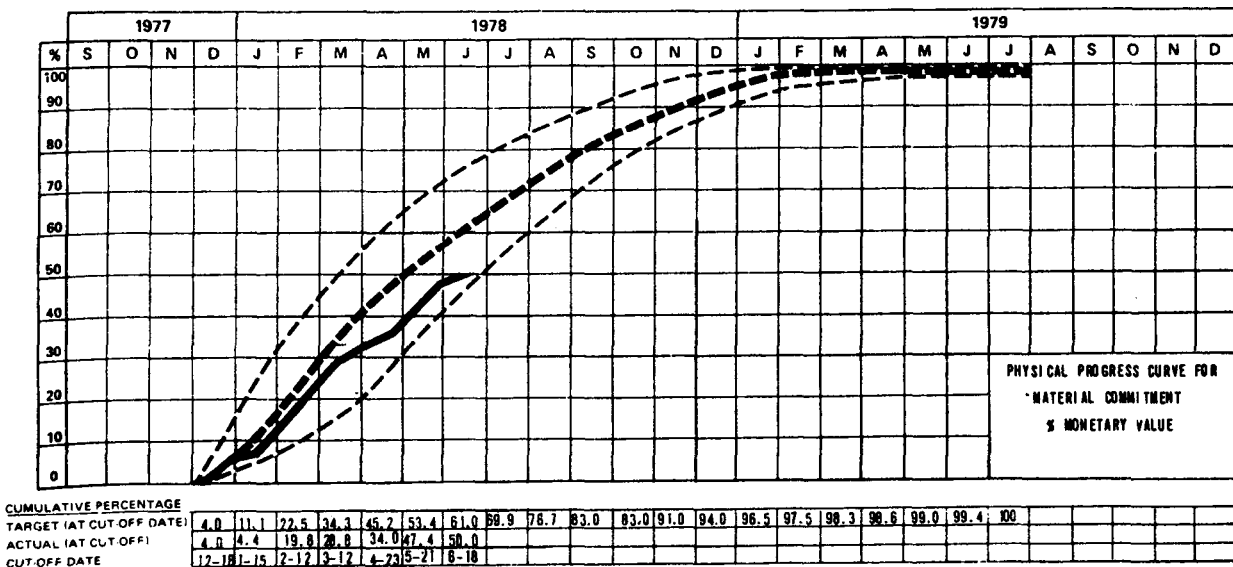
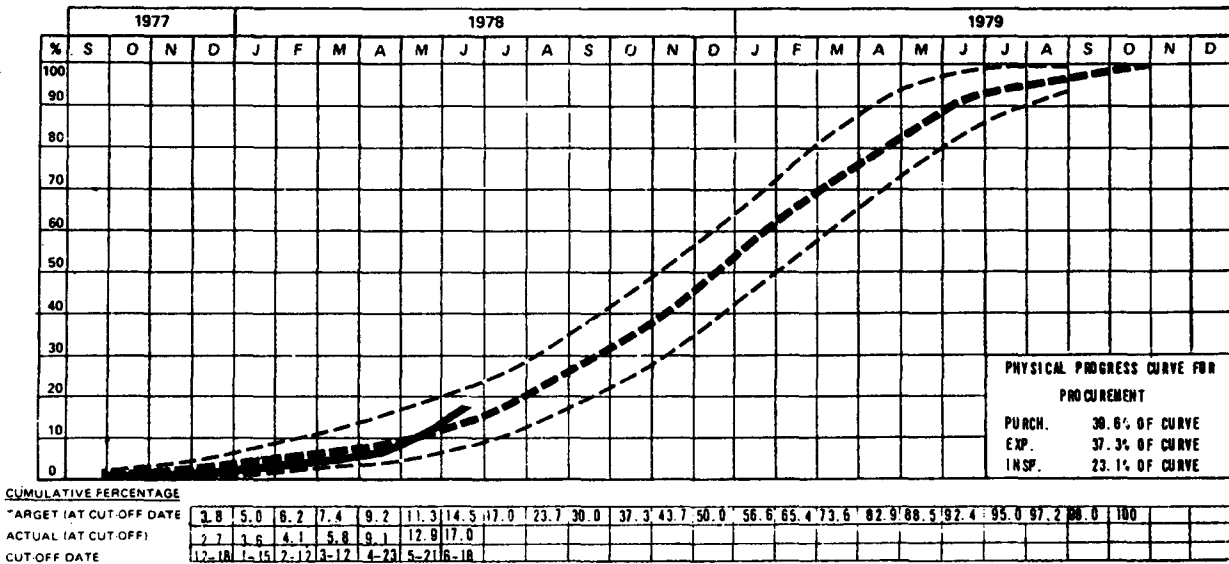
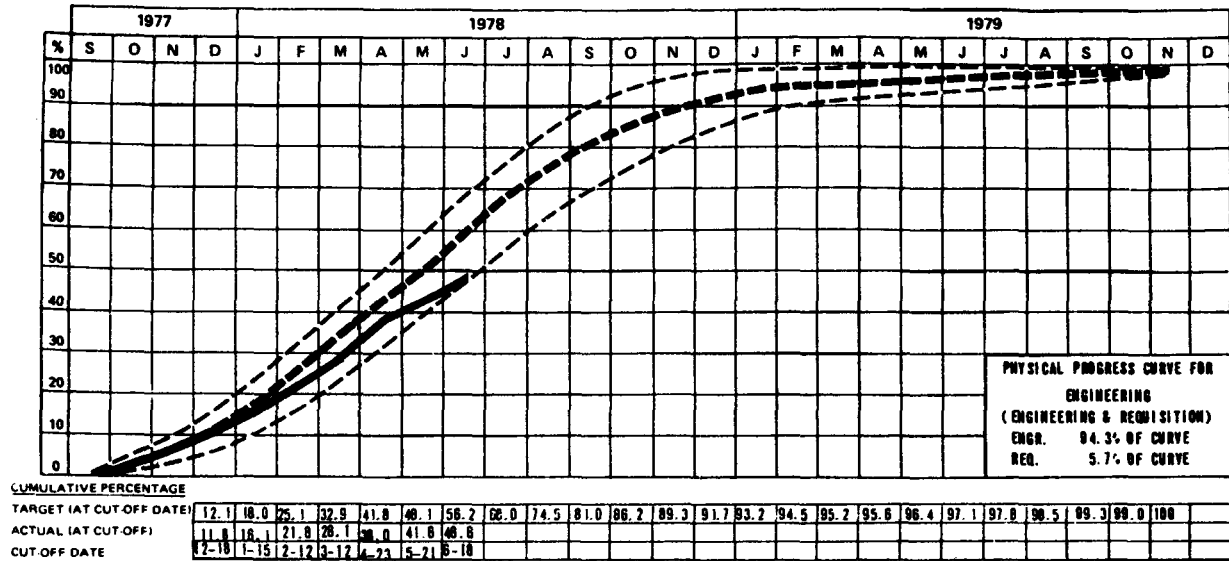
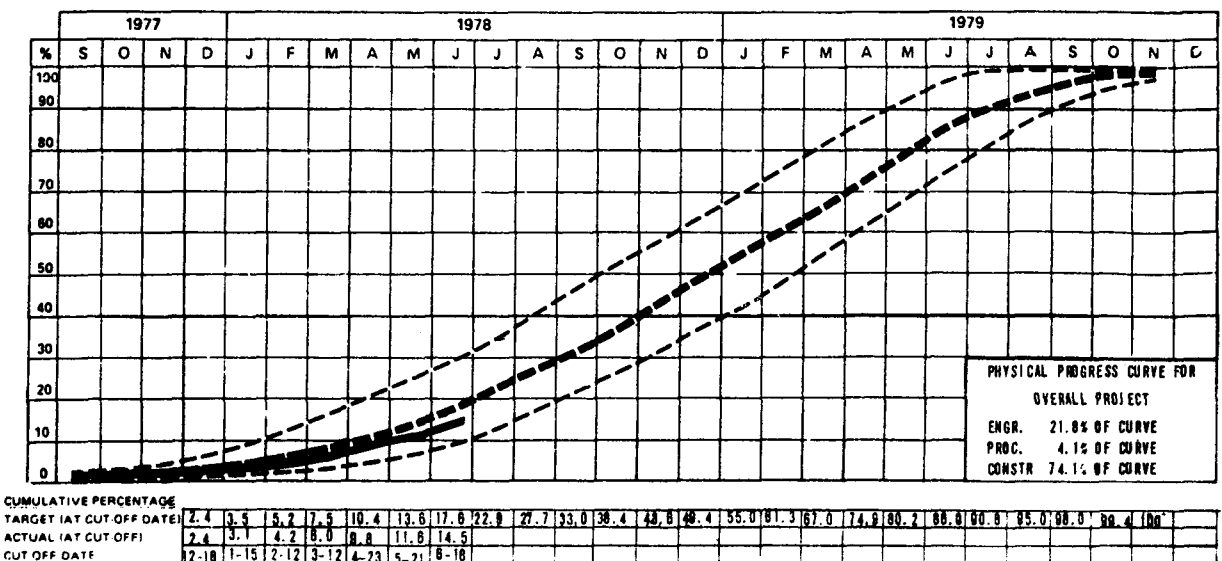
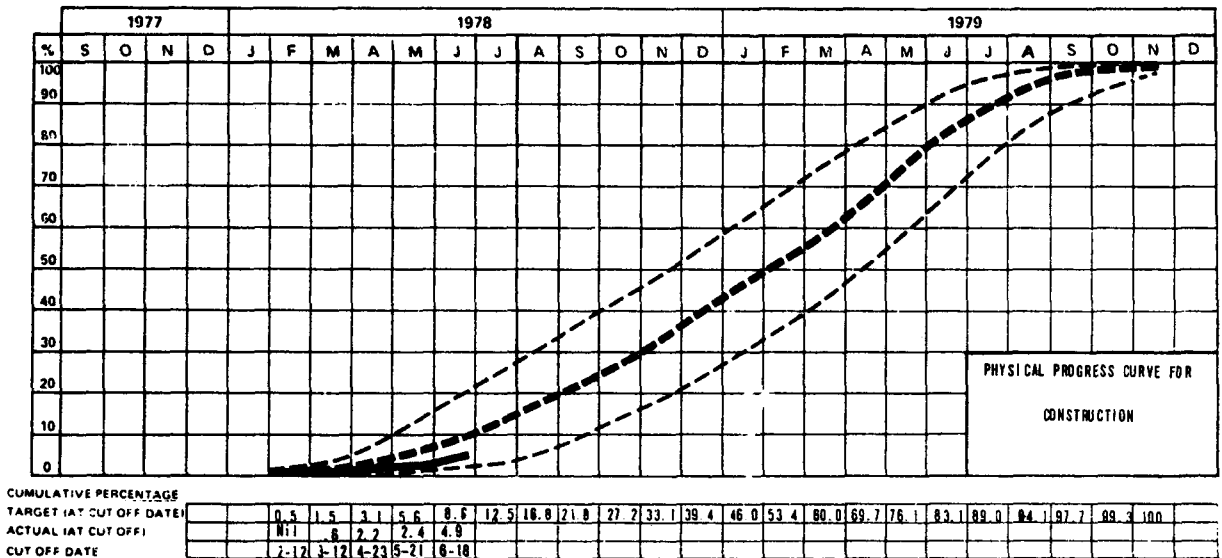
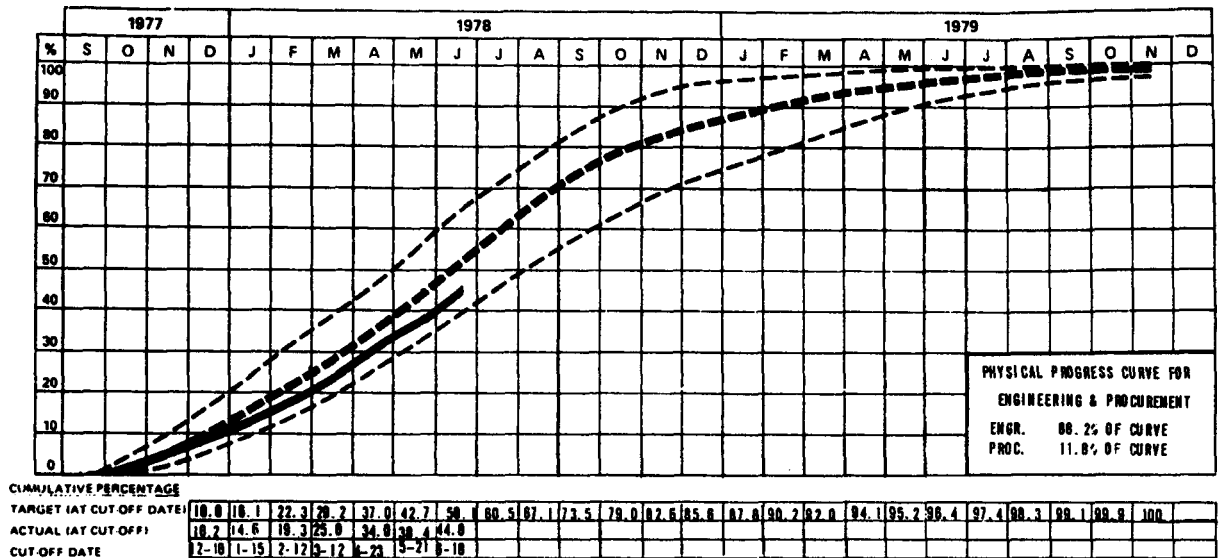


FIGURE 2-2
ECLP PROGRESS CURVES



STANDARD DEVIATION - - - - - TARGET - - - - - ACTUAL - - - - -

FIGURE 2-2 (Cont'd)
ECLP PROGRESS CURVES



ECLP PROJECT MANAGEMENT, DETAILED ENGINEERING,
PROCUREMENT AND CONSTRUCTION

3. Construction

Site preparation work began on 13 February, 1978. Daniel's and Exxon's construction management teams began mobilizing at the construction site on 6 March, 1978.

Construction was 7% complete, about 3% behind our original plan, at the end of June, 1978. The major portion of work to date has been in the subcontract areas of site prep and foundation piling. Daniel's direct hire work force has grown substantially and work has started to accelerate on foundation and underground piping. Total Daniel manpower was 234 on 30 June; 46 salaried Daniel employees, 150 Daniel hourly employees, and 38 subcontractor employees. Highlights of construction activities follow.

1978

Awarded Site Preparation Subcontract	8 Feb.
Site Preparation Started	13 Feb.
Awarded Load Bearing Piling Subcontract	7 April
Test Piles Driven	13 April
Pile Test Complete	3 May
Pile Driving for Silo Started	12 May
Construction Warehouse Erection Started	15 May
Official Ground Breaking Ceremony	16 May
Sheet Pile Driving Started	19 May
Field Office Building Erection Started	22 May
Underground Piping Started	23 May
First Concrete Footings Poured	30 May
Field Office Occupied	19 June

Subcontracts status as of June, 1978 may be summarized as follows:

Major Subcontracts Contemplated	23
Subcontracts Signed	15
Subcontracts Currently Under Negotiation or Inquiry	1
Subcontracts to be Completed September-December, 1978	7

Daniel has prepared, at our request, a proposed safety incentive awareness program for the job and we are currently reviewing their proposal with Carter management. The program will reward good safety performance at both craft and supervisory levels and will be implemented in July, 1978.

ECLP PROJECT MANAGEMENT, DET AILED ENGINEERING,
PROCUREMENT AND CONSTRUCTION

4. Preparation for Operations

ECLP Office Building

A contract was let for construction of the ECLP Administration Building in February 1978. The contractor began the fabrication of modules at his factory that same month. Onsite erection was initiated in May. The building was completed and occupied in June. The construction was accomplished on time and within budgeted funds.

Pipeline Adjustments

The ECLP site is traversed by several buried pipelines that are crossed by access roads and a rail spur. The latter are planned as part of the ECLP project. Prior to construction of ECLP, each road crossing of a pipeline required some adjustment (sleeving) of the pipeline for protection from loads bearing on the soil at the surface. Negotiations to accomplish this work was started in September 1977. There were a total of eight pipeline owner companies involved. Actual field work was started in January 1978 and was completed in May 1978 with no interference to ECLP site preparation and construction.

Contract Maintenance

Maintenance of the pilot plant during the operating period will be accomplished through use of a contractor. Discussions have been held with several potential maintenance contracting companies as part of the selection process. It is planned to solicit commercial terms from several contractors in September 1978. The selection of a contractor will be made by year-end 1978.

Rail Cars

A study was conducted to evaluate the economics of leasing versus purchasing of rail cars to be used in shipping feed coal to the ECLP site. The economics favored purchasing, and bids for 30 coal cars were solicited in early 1978. A vendor was selected and order for the cars is to be submitted in July 1978.

Manpower

On July 1, 1977, three people were assigned to ECLP--a plant manager, a mechanical superintendent, and a process superintendent. As the need developed for additional people, the appropriate staff assignments were made throughout the reporting period. By the end of June 1978, the staff had grown to 12 people, with the objective to reach 35 by year-end 1978.

Disposition of ECLP Products

Approximately 200-to-250 barrels per calendar day of liquid products will be produced in the ECLP operation. Much of this material will be shipped as large-volume samples to the various sponsors for additional testing. Acceptable dispositions will have to be found for any surplus products over the sponsors' needs. In the last half of 1977, considerable effort was devoted to the development of outlets for the liquid products from ECLP. After considering the available alternatives, a decision was reached to dispose of the naphtha and solvent boiling-range material as a liquid furnace fuel in the Baytown Chemical Plant. The heavier products (vacuum gas oil) will be blended into an asphaltic-type material, which is exported from the Baytown Refinery as a coker feed (see page 194).

Monitoring of Engineering

The Carter staff has worked closely with the Project Management Team to provide "owner" input to the engineering effort. For the most part, this effort was in the form of reviews of the engineering model, as well as the piping and instrument drawings. Comments and suggestions were submitted to the Project Management Team which reflected the owner's operating experiences.

Insurance

Builders' Risk insurance was placed in effect in April for a two-year period with coverage up to \$40 million, less \$100,000 deductible. Upon the completion of construction, the limit may be increased if ECLP Management decides additional coverage is needed. Liability coverage of \$40 million was also placed in effect in April for a two-year period. The upper limit on this coverage may also be increased at a later date. Automobile liability insurance is being carried by Daniel Construction Company for vehicles driven by Daniel employees. Insurance for vehicles driven by Carter and Exxon employees assigned to ECLP is obtained directly by Carter as vehicles are purchased.

ECLP Operating Manuals

The preparation of operating manuals and other training material was initiated in June. A detailed work schedule for the preparation of this material is being developed.

Industrial Hygiene

The ECLP staff and Refinery and Research industrial hygienists reviewed and formulated the concepts to be used for the plant. This work was also reviewed with the Sponsors Technical Committee. The program addresses itself to the hazards of hydrocarbons and hydrogen, H₂S, and CO, dust, and polynuclear aromatic hydrocarbons. The program will have four basic elements, consisting of engineering control, environmental surveys, medical surveillance, and personal hygiene. The program is similar to that used at the Baytown R&D Division pilot plants over the last 11 years.

5. Operations

Reporting under this category is to start January 1, 1980.

6. ECLP Environmental Assessment

The ECLP Environmental Impact Negative Declaration was signed by DOE on 8 December, 1977; thus, an environmental impact statement will not be required.

APPENDICES

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 489-524. Analyses of feed and product streams for these yield periods are given in Tables A-3 through A-5.

TABLE A-1

RCLU YIELD PERIOD OPERATION SUMMARY

RCLU Unit Number	1	1	1	1	1	1	1	1
Yield Period Number	489	490	491	492	493	494	495	496
Yield Period Length	24	24	23	14	24	13	24	24
Type of Coal Used	Illinois No. 6 (Monterey No. 1 Mine)							
Liquefaction								
Inlet Pressure, psig	1527	1526	1522	1520	1556	1557	1527	1532
Average Temperature, °F	832	836	837	838	838	837	837	839
Space Velocity, V/Hr/V	1.77	1.52	1.51	1.59	1.48	1.67	1.55	1.62
Specific Coal Feed Rate, Lbs/Hr Ft ³	47	40	40	42	39	43	40	43
Treat Gas Rate, Lbs H ₂ /100 Lbs Dry Coal	3.55	4.10	4.05	3.82	4.10	3.69	4.03	3.85
H ₂ in Treat Gas, Mole %	100	100	100	100	100	100	100	100
Slurry Conc., Lbs Dry Coal/Lbs Slurry	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38
Dry Coal Feed Rate, Lbs/Hr	3.72	3.22	3.19	3.34	3.11	3.48	3.24	3.42
Solvent Quality Index	4.92	4.86	4.58	4.28	4.31	4.06	4.38	4.05
Hydrogenation								
Outlet Pressure, psig	1484	1480	1480	1484	1486	1485	1482	1482
Average Temperature, °F-RV	122	122	120	123	122	122	121	123
Space Velocity, W/Hr/W, % of RV	285	252	251	260	246	273	255	269
Treat Gas Rate, SCF H ₂ /Bbl Oil Feed, % of RV	207	235	233	223	235	213	231	221

Overall Material Balance

Lbs Output/100 Lbs Input	101.38	100.40	102.27	99.86	99.74	98.62	100.90	99.36

TABLE A-1 (Cont'd)

RCLU Unit Number	1	1	1	1	1	1	1	1
Yield Period Number	498	499	500	501	502	503	504	505
Yield Period Length	24	24	24	24	24	24	24	24
Type of Coal Used	Illinois No. 6 (Monterey No. 1 Mine)							
Liquefaction								
Inlet Pressure, psig	1523	1524	1520	1514	1523	1523	1528	1526
Average Temperature, °F	840	841	840	840	840	839	838	838
Space Velocity, V/Hr/V	1.49	1.46	1.41	1.42	1.70	1.55	1.69	1.56
Specific Coal Feed Rate, Lbs/Hr/Ft ³	39	39	37	37	45	41	45	41
Treat Gas Rate, Lbs H ₂ /100 Lbs Dry Coal	4.13	4.22	4.39	4.29	3.72	4.05	3.73	3.51
H ₂ in Treat Gas, Mole %	100	100	100	100	100	100	100	100
Slurry Conc., Lbs Dry Coal/Lbs Slurry	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38
Dry Coal Feed Rate, Lbs/Hr	3.13	3.08	2.98	2.98	3.57	3.27	3.56	3.30
Solvent Quality Index	4.17	4.06	4.08	4.07	4.41	4.30	4.25	4.14
Hydrogenation								
Outlet Pressure, psig	1480	1481	1480	1485	1479	1479	1481	1484
Average Temperature, °F - RV	121	122	121	121	122	122	123	122
Space Velocity, W/Hr/W, % of RV	247	244	235	235	280	252	275	256
Treat Gas Rate, SCF H ₂ /Bbl Oil Feed, % of RV	237	241	252	247	213	236	217	198

Overall Material Balance

Lbs Output/100 Lbs Input	100.69	101.32	101.38	100.21	99.30	101.55	103.44	99.15

TABLE A-1 (Cont'd)

RCLU Unit Number	1	1	1	1	1	1	1	1	1
Yield Period Number	507	508	509	510	511	512	513	514	515
Yield Period Length	24	24	20	24	24	24	24	24	24
Type of Coal Used	Illinois No. 6			Wyoming (Wyodak Mine)	Pittsburgh No. 8 (Ireland Mine)				
<u>Liquefaction</u>									
Inlet Pressure, psig	1522	1513	1516	1539	1521	1520	1524	1528	1526
Average Temperature, °F	838	839	839	840	836	836	838	838	839
Space Velocity, V/Hr/V	1.53	1.65	1.73	1.08	1.51	1.47	1.59	1.61	1.56
Specific Coal Feed Rate, Lbs/Hr/Ft ³	40	44	46	28	40	39	42	42	41
Treat Gas Rate, Lbs H ₂ /100 Lbs Dry Coal	4.07	3.69	3.55	5.18	3.98	4.11	3.80	3.75	3.84
H ₂ in Treat Gas, Mole %	100	100	100	100	100	100	100	100	100
Slurry Conc., Lbs Dry Coal/Lbs 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	3.24	3.49	3.64	3.33	3.19	3.09	3.36	3.38	3.30
Solvent Quality Index	4.11	3.98	4.03	3.88	4.19	4.13	4.03	4.19	4.19
<u>Hydrogenation</u>									
Outlet Pressure, psig	1484	1489	1485	1501	1501	1506	1502	1504	1517
Average Temperature, °F - RV	122	120	119	125	124	123	122	124	125
Space Velocity, W/Hr/W, % of RV	254	272	282	262	227	222	240	243	236
Treat Gas Rate, SCF H ₂ /Bbl Oil Feed, % of RV	233	213	206	230	243	249	232	230	234
<u>Overall Material Balance</u>									
Lbs Output/100 Lbs Input	99.30	99.53	98.86	99.72	100.26	99.43	101.03	97.47	99.59

TABLE A-1 (Cont'd)

RCLU Unit Number	1	1	1	1	1	1	1	1
Yield Period Number	516	517	518	519	520	521	522	523
Yield Period Length	24	24	24	24	24	18	24	24
Type of Coal Used	Pittsburgh No. 8 (Ireland Mine)							
<u>Liquefaction</u>								
Inlet Pressure, psig	1528	1540	1514	1520	1519	1521	1523	1517
Average Temperature, °F	839	836	841	841	841	841	802	803
Space Velocity, V/Hr/V	1.10	1.11	0.56	0.57	0.63	0.59	0.66	0.63
Specific Coal Feed Rate, Lbs/Hr/Ft ³	29	29	15	15	16	16	17	17
Treat Gas Rate, Lbs H ₂ /100 Lbs Dry Coal	3.71	3.65	4.33	4.30	3.95	4.18	3.79	3.90
H ₂ in Treat Gas, Mole %	100	100	100	100	100	100	100	100
Slurry Conc., Lbs Dry Coal/Lbs Slurry	0.38	0.38	0.38	0.38	0.38	0.38	0.38	0.38
Dry Coal Feed Rate, Lbs/Hr	3.48	3.49	2.96	2.98	3.30	3.12	3.42	3.33
Solvent Quality Index	4.45	4.71	4.32	4.37	4.33	4.37	4.58	4.42
<u>Hydrogenation</u>								
Outlet Pressure, psig	1520	1538	1508	1520	1519	1519	1519	1504
Average Temperature, °F - RV	156	159	133	133	135	135	133	134
Space Velocity, W/Hr/W, % of RV	263	266	235	238	258	244	258	249
Treat Gas Rate, SCF H ₂ /Bbl Oil Feed, % of RV	221	216	246	243	225	238	224	232
<u>Overall Material Balance</u>								
Lbs Output/100 Lbs Input	99.30	99.17	99.35	99.57	99.72	100.38	99.76	100.06

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
Yield Period Number	489	490	491	492	493	494	495	496	497	498	499	500	501	502
<u>Overall Yields, Wt% on Dry Coal</u>														
Hydrogen	-3.54	-4.09	-4.35	-3.98	-4.75	-4.03	-4.14	-4.33	-4.61	-4.45	-4.66	-4.52	-4.41	-3.61
Water	7.72	8.54	8.53	9.46	9.85	7.69	9.56	9.73	8.71	10.80	10.58	9.17	9.56	9.30
Carbon Oxides	1.76	1.56	1.46	1.30	1.31	1.39	1.59	1.80	1.78	1.57	1.68	1.70	1.57	1.44
Hydrogen Sulfide	2.91	3.60	2.94	3.00	3.03	2.94	3.35	3.17	3.39	2.69	2.88	3.29	3.46	3.75
Ammonia	0.45	0.64	0.74	0.50	0.82	0.85	0.49	0.69	0.60	0.43	0.59	0.71	0.42	0.83
C1-C3 Gas	6.61	7.79	8.25	7.54	7.83	7.22	7.91	8.02	8.65	8.03	8.33	8.96	8.27	7.10
C4-400°F	20.00	21.22	24.87	23.94	24.76	21.54	22.99	22.41	23.02	23.97	24.64	25.62	23.45	20.57
400-700°F	5.49	6.94	4.87	2.41	4.43	5.26	3.56	5.58	7.74	4.32	5.04	3.00	4.55	6.00
700-1000°F	7.48	5.59	5.09	6.29	4.57	6.24	6.05	6.09	5.56	4.67	4.34	4.93	5.39	7.22
1000°F+	51.14	48.21	47.60	49.54	48.16	50.89	48.64	46.82	45.17	47.98	46.56	47.14	47.75	47.39
C4-1000°F	32.97	33.76	34.83	32.64	33.76	33.05	32.60	34.09	36.32	32.95	34.02	33.56	33.39	33.79

Liquefaction Yields, Wt% on Dry Coal														
Hydrogen	-0.92	-1.69	-2.11	-1.67	-2.39	-2.11	-1.48	-2.42	-2.77	-2.62	-2.85	-2.61	-2.62	-1.68
Water	5.69	6.34	6.58	6.93	7.70	5.61	6.70	7.49	6.58	8.75	8.52	7.04	7.49	7.35
Carbon Oxides	1.76	1.56	1.46	1.30	1.31	1.39	1.59	1.80	1.78	1.57	1.68	1.70	1.57	1.44
Hydrogen Sulfide	2.16	2.93	2.40	1.69	2.25	2.45	2.69	2.51	2.60	1.94	2.28	2.67	2.77	3.30
Ammonia	0.15	0.26	0.41	0.17	0.48	0.50	0.10	0.38	0.23	0.09	0.25	0.33	0.11	0.49
C ₁ -C ₃ Gas	6.61	7.79	8.25	7.54	7.83	7.22	7.91	8.02	8.65	8.03	8.33	8.96	8.27	7.10
C ₄ -1000°F	33.41	34.62	35.42	34.50	34.66	34.06	33.85	35.41	37.76	34.26	35.21	34.77	34.67	34.61
1000°F+	51.14	48.21	47.60	49.54	48.16	50.89	48.64	46.82	45.17	47.98	46.56	47.14	47.75	47.39

TABLE A-2 (Cont'd)

RCLU Unit Number	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Yield Period Number	503	504	505	506	507	508	509	510	511	512	513	514	515	516
Overall Yields, Wt% on Dry Coal														
Hydrogen	-4.07	-3.83	-4.15	-3.73	-4.06	-3.71	-3.59	-3.93	-2.93	-3.11	-3.17	-3.09	-3.18	-3.50
Water	9.92	9.73	10.98	10.04	8.88	9.18	9.39	14.85	5.35	5.04	5.32	5.09	5.44	5.05
Carbon Oxides	1.60	1.17	0.71	0.63	1.48	1.48	1.13	6.97	0.81	0.75	0.77	0.82	0.77	0.91
Hydrogen Sulfide	3.39	3.28	3.14	2.93	3.12	3.26	2.94	0.44	3.36	3.27	2.85	2.97	2.73	2.87
Ammonia	0.58	0.50	0.61	0.45	0.59	0.44	0.50	0.60	0.49	0.32	0.41	0.40	0.36	0.42
C ₁ -C ₃ Gas	7.66	6.31	5.82	6.40	7.50	7.13	6.60	9.79	6.61	6.72	6.80	7.14	7.14	8.31
C ₄ -400°F	20.28	18.77	19.23	18.92	19.98	18.62	19.06	18.12	14.80	16.68	17.17	17.04	17.76	22.37
400-700°F	4.39	8.61	11.74	7.50	8.65	8.31	6.89	1.35	6.13	5.62	5.31	2.95	5.89	3.51
700-1000°F	8.23	7.04	5.51	5.32	4.36	4.99	5.75	6.49	8.04	6.47	4.95	9.27	5.11	6.08
1000°F+	48.04	48.40	46.42	51.55	49.53	50.32	51.34	45.30	47.34	58.25	59.60	57.41	57.99	53.97
C ₄ -1000°F	32.91	34.43	36.47	31.74	32.98	31.91	31.70	25.97	28.97	28.76	27.42	29.26	28.75	31.96

Liquefaction Yields, Wt% on Dry Coal														
Hydrogen	-2.13	-1.81	-1.59	-1.55	-2.06	-1.90	-1.47	-1.27	-1.44	-1.56	-1.59	-1.25	-1.20	-1.47
Water	7.77	7.58	8.64	7.65	6.80	7.27	7.68	13.04	3.94	3.74	4.07	3.60	3.95	3.10
Carbon Oxides	1.60	1.17	0.71	0.63	1.48	1.48	1.13	6.97	0.81	0.75	0.77	0.82	0.77	0.91
Hydrogen Sulfide	2.87	2.49	2.36	2.37	2.47	2.36	2.23	0.25	2.83	2.76	2.35	2.45	2.27	2.29
Ammonia	0.27	0.20	0.18	0.07	0.28	0.20	0.19	0.38	0.31	0.14	0.20	0.12	0.16	0.09
C ₁ -C ₃ Gas	7.66	6.31	5.82	6.40	7.50	7.13	6.60	9.79	6.61	6.72	6.80	7.14	7.14	8.31
C ₄ -1000°F	33.94	35.64	37.46	32.87	34.01	33.16	32.30	25.53	29.61	29.20	27.81	29.71	28.93	32.79
1000°F+	48.04	48.40	46.42	51.55	49.53	50.32	51.34	45.31	57.34	58.25	59.60	57.41	57.99	53.97

TABLE A-2 (Cont'd)

NCLU Unit Number
Yield Period Number

1 1 1 1 1 1 1 1
517 518 519 520 521 522 523 524

Overall Yields, Wt% on Dry Coal

Hydrogen	-3.50	-4.55	-4.84	-4.44	-4.45	-3.27	-2.84	-3.03
Water	5.61	5.52	5.87	5.94	6.72	7.13	5.47	4.87
Carbon Oxides	0.97	1.43	1.28	1.16	1.65	1.31	1.10	0.77
Hydrogen Sulfide	2.94	3.08	3.32	3.24	3.01	2.96	2.84	2.72
Ammonia	0.51	0.63	0.67	0.47	0.60	0.38	0.45	0.53
C ₁ -C ₃ Gas	8.12	14.18	13.94	12.83	13.09	5.63	5.87	5.73
C ₄ -400°F	20.85	23.88	25.55	22.69	24.11	17.75	15.59	15.40
400-700°F	5.18	5.11	4.64	4.21	2.50	4.34	8.84	8.75
700-1000°F	7.36	4.03	4.97	4.94	4.37	5.79	6.17	6.08
1000°F+	51.96	46.71	44.59	48.97	48.40	57.98	56.50	58.19
C ₄ -1000°F	33.40	33.01	35.17	31.83	30.98	27.87	30.61	30.23

Liquefaction Yields, Wt% on Dry Coal

Hydrogen	-1.13	-2.47	-2.68	-2.30	-2.26	-1.29	-1.11	-1.36
Water	3.53	3.96	4.34	4.47	4.90	5.01	2.51	3.18
Carbon Oxides	0.97	1.43	1.28	1.16	1.65	1.31	1.10	0.77
Hydrogen Sulfide	2.44	2.70	2.90	2.86	2.61	1.93	1.87	1.74
Ammonia	0.16	0.23	0.29	0.08	0.26	0.04	0.18	0.24
C ₁ -C ₃ Gas	8.12	14.18	13.94	12.83	13.09	5.63	5.87	5.73
C ₄ -1000°F	33.96	33.28	35.35	31.95	31.35	29.38	33.08	31.52
1000°F+	51.96	46.71	44.59	48.97	48.40	57.98	56.50	58.19

TABLE A-3

**RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY
FEED COAL, RESIDUAL BOTTOMS, AND SOLVENT INSPECTIONS**

RCLU Unit Number Yield Period Number	1 489	1 490	1 491	1 492	1 493	1 494	1 495	1 496	1 497
Feed Coal Analysis									
Elemental Analysis, Dry Wt%									
Carbon	67.34	67.52	68.21	67.73	68.07	68.36	67.04	66.30	66.76
Hydrogen	4.89	4.86	4.99	4.98	4.96	4.94	4.98	4.86	4.73
Nitrogen	1.19	1.29	1.37	1.20	1.38	1.34	1.15	1.19	1.14
Sulfur	4.16	4.65	4.02	4.09	4.04	4.01	4.39	4.23	4.39
Ash	12.36	12.38	11.56	11.24	11.34	11.45	12.00	12.56	12.48
Ash (SO ₃) Free	11.90	12.08	11.25	10.85	10.92	11.03	11.48	12.04	11.95
Oxygen	14.78	15.43	14.80	15.97	16.38	14.10	15.89	16.60	15.34
Moisture	0.20	0.03	0.02	0.35	0.02	0.79	0.00	0.00	0.85
Residual Bottoms, A-1									
Elemental Analysis, Dry Wt%									
Carbon	69.71	67.93	68.21	69.67	68.52	69.83	68.86	68.03	66.86
Hydrogen	4.62	4.38	4.39	4.15	4.22	4.58	4.47	4.49	4.30
Nitrogen	1.41	1.42	1.44	1.40	1.36	1.14	1.36	1.20	1.33
Sulfur	2.49	2.33	2.42	2.26	2.18	2.27	2.26	2.42	2.56
Ash	19.95	21.88	21.78	20.56	20.61	19.74	20.98	21.79	23.77
Ash (SO ₃) Free	19.01	21.11	21.14	19.78	19.76	18.89	20.04	20.72	22.67
Oxygen	11.53	12.46	11.83	11.78	12.46	11.24	11.41	12.78	13.17
Distillation									
1000°F	14.20	10.40	9.30	11.70	8.80	10.90	11.50	11.40	9.00
H ₂ in 1000°F	0.88	0.82	0.69	0.90	0.91	0.91	1.07	1.00	0.90
Sulfur in 1000°F	0.77	0.68	0.67	0.60	0.57	0.59	0.55	0.54	0.52
Solvent, A-2									
Elemental Analysis, Dry Wt%									
Carbon	89.90	89.79	89.65	89.56	89.56	89.40	89.51	89.43	89.35
Hydrogen	9.70	9.91	10.04	10.08	10.18	10.32	10.22	10.28	10.32
Nitrogen	0.04	0.03	0.04	0.04	0.04	0.04	0.03	0.04	0.04
Sulfur	0.011	0.009	0.006	0.004	0.004	0.006	0.005	0.005	0.004
Oxygen	0.35	0.26	0.27	0.31	0.22	0.24	0.23	0.25	0.29
Donor H ₂ in Solvent, % of RV	241	239	225	211	212	197	212	200	211
Distillation									
5 wt% off, °F	395.3	394.5	393.6	396.5	396.7	394.8	395.5	393.8	393.3
15 wt% off, °F	415.6	408.2	407.1	405.1	406.8	408.6	405.2	406.8	405.9
25 wt% off, °F	453.9	438.1	434.7	431.9	433.5	430.1	432.1	434.9	431.6
50 wt% off, °F	537.4	513.3	507.3	499.8	500.7	498.3	500.1	504.2	500.1
75 wt% off, °F	628.7	589.0	585.2	580.8	581.6	583.1	582.3	588.0	586.5
95 wt% off, °F	827.0	740.4	742.9	725.0	726.7	724.6	722.3	752.2	761.1
400°F, wt%	9.30	10.80	11.50	10.57	9.78	8.70	10.95	11.30	11.60
400-700°F, wt%	75.80	81.70	80.80	82.75	83.46	84.50	82.47	80.30	79.50
700°F, wt%	14.90	7.50	7.70	6.68	6.76	6.80	6.58	8.40	8.90
Specific Gravity @ 60°F/60°F	0.980	0.972	0.965	0.961	0.958	0.961	0.958	0.953	0.944

TABLE A-3 (Cont'd)

NCLU Unit Number Yield Period Number	1 498	1 499	1 500	1 501	1 502	1 503	1 504	1 505	1 506
<u>Feed Coal Analysis</u>									
Elemental Analysis, Dry Wt%									
Carbon	66.48	67.56	66.65	67.85	66.56	67.19	66.74	67.48	66.04
Hydrogen	4.93	4.94	4.84	4.99	5.19	4.85	4.86	4.80	4.87
Nitrogen	1.12	1.22	1.26	1.19	1.42	1.22	1.16	1.14	1.10
Sulfur	3.83	3.99	4.43	4.87	4.86	4.82	4.54	4.28	4.20
Ash	12.31	12.20	12.12	12.11	11.62	12.17	12.86	12.55	12.46
Ash (SO ₃) Free	11.82	11.66	11.59	11.54	11.16	11.58	12.19	11.96	11.79
Oxygen	16.90	17.17	15.95	16.84	16.30	17.44	16.69	17.13	16.52
Moisture	0.27	0.17	0.07	0.24	0.33	0.19	0.19	0.07	0.02
<u>Residual Bottoms, A-1</u>									
Elemental Analysis, Dry Wt%									
Carbon	66.96	67.70	67.52	67.29	69.02	68.65	68.68	68.10	67.05
Hydrogen	4.40	4.49	4.28	4.52	4.57	4.59	4.60	4.51	4.36
Nitrogen	1.45	1.42	1.31	1.56	1.34	1.29	1.37	1.21	1.29
Sulfur	2.48	2.44	2.62	2.88	2.43	2.78	2.65	2.54	2.60
Ash	23.25	22.58	23.39	22.45	20.30	20.65	21.13	20.99	22.35
Ash (SO ₃) Free	22.26	21.73	22.30	21.37	19.36	19.71	20.00	19.84	21.20
Oxygen	11.70	12.41	12.69	12.91	12.65	12.67	12.99	12.82	12.57
Distillation									
1000°F-	8.40	8.30	9.50	10.00	13.20	14.60	12.00	9.10	8.60
N ₂ in 1000°F-	0.89	0.94	0.95	0.96	0.79	1.01	0.77	0.82	0.91
Sulfur in 1000°F-	0.52	0.49	0.59	0.51	0.63	0.55	0.61	0.62	0.59
<u>Solvent, A-2</u>									
Elemental Analysis, Dry Wt%									
Carbon	89.32	89.31	89.40	89.38	89.70	89.55	89.49	89.42	89.41
Hydrogen	10.36	10.32	10.34	10.28	9.95	10.11	10.16	10.13	10.23
Nitrogen	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.40
Sulfur	0.006	0.004	0.006	0.005	0.008	0.006	0.007	0.006	0.008
Oxygen	0.29	0.33	0.22	0.29	0.30	0.29	0.31	0.40	0.32
Donor H ₂ in Solvent, % of RV									
	205	200	201	200	217	212	209	204	213
Distillation									
5 wt% off, °F	393.5	394.8	394.8	393.2	392.0	393.4	393.4	393.5	393.1
15 wt% off, °F	406.8	404.7	410.5	407.9	403.0	406.7	410.4	407.1	406.7
25 wt% off, °F	433.3	425.3	436.8	433.3	432.4	435.2	439.9	434.8	433.4
50 wt% off, °F	502.1	489.0	506.5	500.8	509.7	507.6	515.4	505.8	503.9
75 wt% off, °F	588.6	568.8	595.0	586.8	594.0	590.1	596.9	590.8	590.6
95 wt% off, °F	764.4	707.6	769.9	744.8	760.6	750.5	758.5	759.5	763.2
400°F-, wt%	11.20	11.05	10.00	11.60	13.80	12.10	11.30	11.90	12.00
400-700°F, wt%	79.70	83.42	80.30	80.20	77.40	79.60	79.70	79.20	78.90
700°F+, wt%	9.10	5.53	9.70	8.20	8.80	8.30	9.00	8.90	9.10
Specific Gravity @ 60°F/60°F	0.948	0.946	0.950	0.949	0.965	0.960	0.962	0.957	0.956

TABLE A-3 (Cont'd)

RCLU Unit Number Yield Period Number	1 507	1 508	1 509	1 510	1 511	1 512	1 513	1 514	1 515
Feed Coal Analysis									
Elemental Analysis, Dry Wt%									
Carbon	66.93	67.83	68.59	66.26	73.41	73.24	73.16	73.90	73.01
Hydrogen	4.82	5.12	5.15	4.84	5.09	5.04	5.05	5.15	5.07
Nitrogen	1.19	1.12	1.17	1.05	1.26	1.14	1.22	1.25	1.20
Sulfur	4.26	4.42	4.28	0.65	4.92	4.88	4.49	4.60	4.48
Ash	11.19	11.63	10.70	9.70	9.48	9.43	9.52	9.20	9.55
Ash (SO ₃) Free	10.68	11.06	10.32	7.74	9.24	9.19	9.25	8.96	9.31
Oxygen	15.50	15.92	16.25	24.14	10.45	10.62	10.55	10.56	10.95
Moisture	0.03	0.04	0.01	2.39	0.03	0.20	0.05	0.10	0.01
Residual Bottoms, A-1									
Elemental Analysis, Dry Wt%									
Carbon	68.18	69.66	69.69	75.91	75.73	74.86	74.72	75.53	73.97
Hydrogen	4.39	4.56	4.51	4.34	4.91	4.80	4.86	4.89	4.73
Nitrogen	1.32	1.34	1.30	1.04	1.27	1.33	1.35	1.34	1.37
Sulfur	2.54	2.45	2.57	0.43	2.67	2.78	2.82	2.64	2.92
Ash	22.20	20.17	19.16	15.18	13.03	14.38	14.48	13.75	14.18
Ash (SO ₃) Free	21.03	19.18	18.42	14.13	12.69	13.98	14.12	13.46	13.87
Oxygen	12.16	11.77	11.79	10.65	7.61	8.44	8.00	7.86	8.27
Distillation									
1000°F-	8.50	8.70	9.70	13.50	12.30	10.10	7.50	14.20	10.70
N ₂ in 1000°F-	0.85	0.94	0.90	0.73	1.06	0.66	0.79	1.04	0.58
Sulfur in 1000°F-	0.56	0.59	0.044	0.016	0.77	0.79	0.73	0.75	0.54
Solvent, A-2									
Elemental Analysis, Dry Wt%									
Carbon	89.40	89.35	89.31	90.33	90.30	90.13	90.01	89.88	89.74
Hydrogen	10.26	10.13	10.21	9.06	9.04	9.17	9.23	9.40	9.53
Nitrogen	0.04	0.06	0.05	0.08	0.12	0.10	0.13	0.12	0.12
Sulfur	0.005	0.000	0.000	0.005	0.023	0.020	0.020	0.016	0.018
Oxygen	0.30	0.46	0.43	0.52	0.52	0.58	0.61	0.59	0.59
Donor H ₂ in Solvent, % of KV	203	196	199	187	207	203	199	205	207
Distillation									
5 wt% off, °F	397.8	401.0	397.5	394.6	397.5	397.3	394.8	394.4	400.4
15 wt% off, °F	409.6	413.9	413.1	403.7	407.4	406.8	407.4	405.9	410.8
25 wt% off, °F	434.3	436.7	435.7	427.8	436.1	436.7	442.8	436.8	437.9
50 wt% off, °F	499.6	501.7	501.2	507.4	520.2	517.5	527.6	516.1	508.1
75 wt% off, °F	583.2	585.9	586.9	592.9	602.4	596.7	613.6	600.0	591.1
95 wt% off, °F	727.9	721.3	737.5	733.5	725.0	719.3	805.3	768.8	731.5
400°F-, wt%	6.70	4.50	6.50	11.70	8.20	8.60	10.60	11.00	5.10
400-700°F, Wt%	86.20	88.70	85.70	81.30	84.80	84.90	77.70	79.50	87.60
700°F+, Wt%	7.10	6.80	7.80	7.00	7.00	6.50	11.70	9.50	7.30
Specific Gravity @ 60°F/60°F	0.953	0.958	0.957	0.991	0.996	0.990	0.988	0.984	0.977

TABLE A-3 (Cont'd)

CLU Unit Number field Period Number	1 516	1 517	1 518	1 519	1 520	1 521	1 522	1 523	1 524
<u>Feed Coal Analysis</u>									
Elemental Analysis, Dry Wt%									
Carbon	73.52	72.83	73.47	73.09	72.93	72.43	73.39	73.23	73.59
Hydrogen	5.15	5.11	5.15	5.00	5.13	5.17	5.01	5.35	5.11
Nitrogen	1.13	1.21	1.22	1.23	1.14	1.25	1.26	1.27	1.24
Sulfur	4.15	4.25	4.19	4.46	4.34	4.32	4.59	4.33	4.29
Ash	9.29	9.67	9.50	9.61	9.54	9.55	9.12	9.15	8.98
Ash (SO ₃) Free	9.07	9.45	9.17	9.28	9.47	9.33	8.29	8.96	8.70
Oxygen	9.99	10.36	10.12	10.27	10.43	11.53	12.63	10.80	10.08
Moisture	0.00	0.27	0.02	0.00	0.21	0.03	1.17	0.02	0.01
<u>Residual Bottoms, A-1</u>									
Elemental Analysis, Dry Wt%									
Carbon	74.97	74.82	73.60	73.15	74.09	73.03	74.98	74.84	74.89
Hydrogen	4.68	4.74	4.30	4.41	4.68	4.48	4.96	4.91	4.92
Nitrogen	1.29	1.32	1.38	1.36	1.38	1.39	1.42	1.41	1.25
Sulfur	2.47	2.54	2.62	2.78	2.48	2.81	2.71	2.63	2.74
Ash	14.78	14.99	17.46	17.70	16.63	17.40	14.01	14.04	13.92
Ash (SO ₃) Free	14.45	14.66	16.92	17.19	16.55	17.13	13.73	13.76	13.62
Oxygen	8.07	7.83	8.26	8.28	8.03	8.04	7.89	8.08	8.07
Distillation									
1000°F-	10.20	12.90	7.30	9.70	8.90	8.30	8.80	9.80	8.90
N ₂ in 1000°F-	0.85	0.78	1.04	0.99	1.16	0.99	1.09	1.06	0.88
Sulfur in 1000°F-	0.66	0.59	0.51	0.53	0.60	0.47	0.75	0.82	0.78
<u>Solvent, A-2</u>									
Elemental Analysis, Dry Wt%									
Carbon	90.34	90.24	90.35	90.27	90.32	90.29	89.97	89.82	89.76
Hydrogen	9.23	9.44	9.19	9.23	9.18	9.23	9.70	9.81	9.90
Nitrogen	0.10	0.07	0.12	0.12	0.13	0.12	0.08	0.09	0.09
Sulfur	0.028	0.019	0.015	0.012	0.012	0.012	0.008	0.008	0.008
Oxygen	0.30	0.23	0.32	0.37	0.36	0.35	0.24	0.27	0.25
Donor H ₂ in Solvent, % of RV	220	232	213	216	213	216	222	217	216
Distillation									
5 wt% off, °F	400.3	361.5	400.5	361.0	399.4	360.9	360.2	396.7	398.9
15 wt% off, °F	408.8	378.4	409.6	373.0	407.1	372.1	372.6	404.6	407.7
25 wt% off, °F	437.1	405.1	433.9	392.8	421.9	388.9	393.6	425.3	430.5
50 wt% off, °F	518.0	479.1	504.5	455.6	487.9	449.9	461.0	497.1	498.6
75 wt% off, °F	604.8	552.6	593.6	545.4	580.0	543.2	552.5	589.1	586.4
95 wt% off, °F	744.1	686.3	739.4	702.7	713.2	695.1	726.3	738.4	724.4
400°F-, Wt%	5.20	23.20	5.00	28.70	6.50	30.60	28.40	9.00	7.10
400-700°F, Wt%	86.60	72.70	87.00	66.20	87.40	64.80	65.20	86.80	85.90
700°F+, Wt%	8.20	4.10	8.00	5.10	6.10	4.60	6.40	4.20	7.00
Specific Gravity @ 60°F/60°F	0.991	0.986	0.990	0.986	0.987	0.983	0.967	0.964	0.961

TABLE A-4

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY
NAPHTHA AND SOUR WATER INSPECTIONS

RCLU Unit Number Yield Period Number	1 489	1 490	1 491	1 492	1 493	1 494	1 495	1 496	1 497
<u>Naphta, A-3</u>									
Elemental Analysis, Dry Wt%									
Carbon	86.24	86.24	86.12	86.16	86.20	86.40	86.20	86.01	86.06
Hydrogen	13.31	13.30	13.29	13.31	13.32	13.21	13.30	13.30	13.28
Nitrogen	0.02	0.02	0.02	0.02	0.01	0.02	0.02	0.02	0.02
Sulfur	0.008	0.011	0.007	0.002	0.012	0.006	0.008	0.005	0.008
Oxygen	0.42	0.43	0.57	0.51	0.46	0.37	0.48	0.67	0.63
Distillation									
5 wt% off, °F	147.6	156.0	157.0	156.2	158.3	167.9	168.5	161.8	156.8
15 wt% off, °F	191.9	200.8	202.9	203.4	206.4	209.7	209.9	208.4	207.0
25 wt% off, °F	213.8	216.5	218.4	219.9	226.4	242.0	241.9	232.0	231.0
50 wt% off, °F	297.0	307.1	305.8	308.2	310.2	318.7	316.5	313.7	313.8
75 wt% off, °F	354.7	362.4	359.7	361.9	361.9	365.7	363.7	363.1	363.4
95 wt% off, °F	377.5	382.1	380.1	382.1	381.6	383.5	382.7	383.0	384.0
400°F-, Wt%	99.20	99.30	99.30	99.30	99.30	99.30	99.20	99.30	99.20
400°F+, Wt%	0.80	0.70	0.70	0.70	0.70	0.70	0.80	0.70	0.80
Specific Gravity @ 60°F/60°F	0.812	0.815	0.813	0.815	0.815	0.819	0.815	0.814	0.816
<u>Sour Water, A-4</u>									
CO ₂ , Wt%	5.89	4.36	4.61	4.38	3.89	4.87	4.97	5.88	4.80
NH ₃ , Wt%	1.28	1.21	0.90	3.65	1.96	2.81	1.10	1.72	1.42
H ₂ S, Wt%	6.42	6.08	6.46	6.72	6.59	6.55	6.42	6.25	5.78
Specific Gravity @ 60°F/60°F	1.046	1.042	1.026	1.041	1.032	1.042	1.036	1.036	1.042

TABLE A-4 (Cont'd)

RCLU Unit number Yield Period Number	1 498	1 499	1 500	1 501	1 502	1 503	1 504	1 505	1 506
<u>Naphta, A-3</u>									
Elemental Analysis, Dry Wt%									
Carbon	86.02	85.78	85.86	85.85	86.03	85.76	85.82	85.59	85.69
Hydrogen	13.35	13.25	13.11	13.17	12.96	13.12	13.05	13.01	13.11
Nitrogen	0.02	0.02	0.03	0.03	0.03	0.03	0.03	0.04	0.03
Sulfur	0.006	0.010	0.005	0.006	0.006	0.006	0.004	0.010	0.008
Oxygen	0.61	0.94	0.99	0.95	0.97	1.09	1.10	1.35	1.16
Distillation									
5 wt% off, °F	155.1	155.6	156.5	160.1	160.6	149.6	159.2	158.4	167.0
15 wt% off, °F	203.3	205.0	206.0	205.6	206.6	201.8	209.2	209.2	209.5
25 wt% off, °F	221.5	227.7	245.3	240.3	247.0	228.0	238.5	241.6	240.8
50 wt% off, °F	304.7	308.0	324.5	318.1	327.9	313.9	318.4	320.5	317.0
75 wt% off, °F	356.4	357.0	366.3	363.4	366.5	362.2	365.1	365.9	363.1
95 wt% off, °F	381.2	379.3	387.0	385.6	384.5	382.7	386.3	386.8	385.3
400°F-, Wt%	99.30	99.40	99.00	99.00	99.40	99.60	99.50	99.40	99.50
400°F+, Wt%	0.70	0.60	1.00	1.00	0.60	0.40	0.50	0.60	0.50
Specific Gravity @ 60°F/60°F	0.815	0.815	0.823	0.820	0.827	0.821	0.822	0.826	0.821
<u>Sour Water, A-4</u>									
CO ₂ , Wt%	4.09	5.09	4.31	4.83	4.21	4.53	4.12	3.80	3.85
NH ₃ , Wt%	1.04	1.94	2.26	4.20	1.94	1.04	2.16	2.16	1.93
H ₂ S, Wt%	6.04	5.57	5.53	4.68	6.59	5.36	5.82	5.44	5.78
Specific Gravity @ 60°F/60°F	1.036	1.042	1.036	1.041	1.039	1.046	1.037	1.038	1.042

TABLE A-4 (Cont'd)

RCLU Unit Number Yield Period Number	1 507	1 508	1 509	1 510	1 511	1 512	1 513	1 514	1 515
<u>Naphtha, A-3</u>									
Elemental Analysis, Dry Wt%									
Carbon	85.57	85.62	85.47	85.39	86.64	86.00	86.29	86.02	85.93
Hydrogen	13.07	12.96	12.94	12.53	11.92	12.38	12.15	12.53	12.53
Nitrogen	0.04	0.04	0.05	0.06	0.10	0.09	0.08	0.08	0.08
Sulfur	0.006	0.000	0.000	0.002	0.010	0.010	0.012	0.006	0.012
Oxygen	1.32	1.39	1.53	2.01	1.33	1.52	1.47	1.37	1.45
Distillation									
5 wt% off, °F	168.8	168.6	164.5	168.9	170.4	172.8	160.3	168.6	168.0
15 wt% off, °F	210.6	210.9	210.5	211.9	213.6	215.2	203.2	211.4	211.4
25 wt% off, °F	245.0	248.7	248.6	254.3	258.2	261.3	241.5	248.4	248.3
50 wt% off, °F	317.7	324.3	324.9	328.9	330.3	329.8	319.9	322.8	323.2
75 wt% off, °F	361.9	366.5	366.5	365.2	365.4	366.8	359.0	364.8	366.0
95 wt% off, °F	383.3	386.8	387.1	382.2	380.7	382.1	375.2	382.1	384.0
400°F, Wt%	99.50	99.50	99.50	99.60	99.60	99.60	100.00	99.60	99.50
400°F+, Wt%	0.50	0.50	0.50	0.40	0.40	0.40	-0.00	0.40	0.50
Specific Gravity @ 60°F/60°F	0.823	0.826	0.827	0.835	0.840	0.838	0.837	0.834	0.833
<u>Sour Water, A-4</u>									
CO ₂ , Wt%	6.55	5.85	3.43	6.23	3.65	2.35	1.89	3.72	1.88
NH ₃ , Wt%	2.78	1.96	1.95	5.00	2.09	0.61	0.70	0.66	0.68
H ₂ S, Wt%	5.70	5.70	5.82	0.03	4.38	6.93	7.61	7.74	7.48
Specific Gravity @ 60°F/60°F	1.034	1.040	1.041	1.051	1.042	1.033	1.037	1.021	1.033

TABLE A-4 (Cont'd)

RCLU Unit Number Yield Period Number	1 516	1 517	1 518	1 519	1 520	1 521	1 522	1 523	1 524
<u>Naphtha, A-3</u>									
Elemental Analysis, Dry Wt%									
Carbon	86.51	86.70	86.96	86.76	86.90	86.74	86.52	86.36	86.31
Hydrogen	12.67	12.62	12.14	12.25	12.16	12.25	12.50	12.57	12.59
Nitrogen	0.06	0.05	0.07	0.06	0.07	0.08	0.05	0.05	0.05
Sulfur	0.008	0.007	0.008	0.003	0.007	0.005	0.007	0.006	0.006
Oxygen	0.76	0.63	0.82	0.93	0.86	0.93	0.92	1.01	1.05
Distillation									
5 wt% off, °F	149.6	168.7	179.5	158.2	171.4	149.8	162.0	162.5	161.4
15 wt% off, °F	199.4	209.3	214.4	203.9	213.6	199.4	204.0	204.6	203.3
25 wt% off, °F	227.0	242.3	243.4	232.2	239.7	229.8	242.0	240.0	239.2
50 wt% off, °F	307.1	326.5	320.3	315.4	312.3	311.4	317.2	316.2	314.5
75 wt% off, °F	356.8	366.5	365.0	362.5	363.5	357.4	358.8	359.7	357.9
95 wt% off, °F	376.4	389.5	381.8	380.8	382.2	376.4	376.6	377.8	377.0
400°F, Wt%	100.00	97.90	99.30	99.20	98.90	99.40	99.80	99.70	99.80
400°F+, Wt%	-0.00	2.10	0.70	0.80	1.10	0.60	0.20	0.30	0.20
Specific Gravity @ 60°F/60°F	0.828	0.833	0.833	0.833	0.831	0.832	0.833	0.831	0.831
<u>Sour Water, A-4</u>									
CO ₂ , Wt%	4.91	8.78	8.54	6.73	4.97	11.06	13.60	3.70	3.16
NH ₃ , Wt%	0.58	0.43	1.71	0.70	0.32	0.77	0.46	1.84	1.02
H ₂ S, Wt%	9.86	8.97	8.97	8.67	8.25	8.95	8.55	8.42	7.19
Specific Gravity @ 60°F/60°F	1.031	1.036	1.047	1.044	1.052	1.044	1.034	1.033	1.038

TABLE A-5

RCLU YIELD PERIOD ANALYTICAL DATA SUMMARY
TAIL GAS INSPECTIONS

RCLU Unit Number Yield Period Number	1 489	1 490	1 491	1 492	1 493	1 494	1 495	1 496	1 497
<u>Tail Gas, Mole %</u>									
Hydrogen	91.52	91.72	91.39	91.53	92.05	91.89	91.73	92.08	92.29
Nitrogen	4.17	4.06	3.95	3.75	3.45	3.46	3.70	3.46	3.49
Argon + Oxygen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Hydrogen Sulfide	0.66	0.62	0.66	0.65	0.58	0.62	0.60	0.61	0.55
Sulfur Dioxide	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Carbon Monoxide	0.16	0.15	0.14	0.14	0.13	0.14	0.15	0.15	0.14
Carbon Dioxide	0.14	0.11	0.11	0.11	0.10	0.11	0.12	0.14	0.12
Methane	1.57	1.53	1.66	1.63	1.60	1.62	1.63	1.68	1.61
Ethylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ethane	0.64	0.63	0.69	0.68	0.66	0.67	0.68	0.69	0.67
Propylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Propane	0.41	0.43	0.47	0.46	0.44	0.44	0.44	0.45	0.43
n-Butane	0.19	0.19	0.21	0.21	0.20	0.21	0.20	0.19	0.19
i-Butane	0.05	0.05	0.05	0.06	0.05	0.06	0.06	0.05	0.05
i-Pentene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cis/Trans, 2-Pentene	0.00	0.00	0.00	0.01	0.01	0.01	0.00	0.01	0.00
n-Pentane	0.05	0.05	0.05	0.06	0.07	0.08	0.08	0.06	0.05
i-Pentane	0.03	0.03	0.03	0.03	0.04	0.05	0.05	0.03	0.03
n-Hexane	0.02	0.02	0.03	0.04	0.04	0.05	0.03	0.04	0.03
2-Methyl Pentane	0.02	0.02	0.02	0.03	0.03	0.03	0.02	0.03	0.03
n-Heptane	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00
2-Methyl Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3-Methyl Hexane	0.00	0.00	0.10	0.12	0.00	0.00	0.00	0.00	0.00
Cyclo-Pentane/3Mepentane	0.04	0.04	0.04	0.05	0.06	0.06	0.05	0.05	0.05
Methyl Cyclopentane	0.03	0.04	0.06	0.08	0.10	0.09	0.05	0.08	0.06
Cyclohexane	0.08	0.07	0.10	0.12	0.14	0.13	0.10	0.10	0.09
Methyl Cyclohexane	0.056	0.059	0.090	0.119	0.133	0.124	0.093	0.083	0.076
Benzene	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000	0.000
Toluene	0.010	0.009	0.006	0.005	0.004	0.004	0.005	0.005	0.004
i-Butene	0.016	0.015	0.018	0.022	0.024	0.022	0.018	0.021	0.023
Tran 2-Butene	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Cis 2-Butene	0.000	0.000	0.000	0.000	0.001	0.001	0.000	0.000	0.000
Ammonia	0.002	0.002	0.003	0.002	0.001	0.003	0.001	0.001	0.002
Water	0.133	0.145	0.101	0.101	0.073	0.146	0.190	0.000	0.000

RCLU Unit Number Yield Period Number	1 498	1 499	1 500	1 501	1 502	1 503	1 504	1 505	1 506
<u>Tail Gas, Mole %</u>									
Hydrogen	92.03	92.04	92.32	92.47	92.56	92.73	93.20	95.35	96.02
Nitrogen	3.47	3.47	3.39	3.31	3.48	3.25	3.07	1.25	0.68
Argon + Oxygen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04	0.06
Hydrogen Sulfide	0.58	0.58	0.53	0.53	0.59	0.59	0.54	0.49	0.43
Sulfur Dioxide	0.80	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Carbon Monoxide	0.14	0.14	0.13	0.13	0.14	0.14	0.11	0.06	0.05
Carbon Dioxide	0.12	0.13	0.12	0.11	0.12	0.13	0.10	0.04	0.02
Methane	1.61	1.67	1.62	1.54	1.49	1.54	1.40	1.34	1.30
Ethylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ethane	0.67	0.70	0.67	0.64	0.63	0.65	0.58	0.51	0.50
Propylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Propane	0.45	0.46	0.43	0.42	0.41	0.42	0.38	0.35	0.33
n-Butane	0.19	0.20	0.18	0.19	0.18	0.19	0.18	0.18	0.17
i-Butane	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
i-Pentene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cis/Trans, 2-Pentene	0.01	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00
n-Pentane	0.06	0.06	0.06	0.06	0.05	0.06	0.06	0.06	0.05
i-Pentane	0.03	0.03	0.03	0.03	0.03	0.04	0.03	0.03	0.03
n-Hexane	0.05	0.04	0.04	0.04	0.02	0.02	0.02	0.01	0.02
2-Methyl Pentane	0.04	0.04	0.03	0.03	0.01	0.01	0.01	0.01	0.02
n-Heptane	0.01	0.03	0.02	0.02	0.00	0.00	0.00	0.00	0.00
2-Methyl Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3-Methyl Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclo-Pentane/3Mepentane	0.06	0.06	0.05	0.05	0.03	0.03	0.03	0.02	0.02
Methyl Cyclopentane	0.11	0.10	0.08	0.08	0.02	0.02	0.02	0.01	0.01
Cyclohexane	0.13	0.12	0.10	0.10	0.06	0.05	0.05	0.04	0.04
Methyl Cyclohexane	0.127	0.114	0.095	0.100	0.034	0.022	0.027	0.022	0.020
Benzene	0.000	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000
Toluene	0.003	0.003	0.004	0.005	0.006	0.010	0.009	0.011	0.008
i-Butene	0.031	0.030	0.024	0.023	0.007	0.013	0.011	0.006	0.006
Tran 2-Butene	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Cis 2-Butene	0.001	0.001	0.001	0.001	0.000	0.000	0.000	0.000	0.001
Ammonia	0.004	0.009	0.001	0.001	0.002	0.007	0.003	0.002	0.002
Water	0.016	0.022	0.012	0.037	0.066	0.019	0.118	0.114	0.145

TABLE A-5 (Cont'd)

RCLU Unit Number Yield Period Number	1 507	1 508	1 509	1 510	1 511	1 512	1 513	1 514	1 515
<u>Tail Gas, Mole %</u>									
Hydrogen	93.37	92.39	92.66	90.62	93.74	93.25	92.86	92.93	93.22
Nitrogen	2.61	3.32	3.43	4.54	3.13	3.68	3.80	3.76	3.49
Argon + Oxygen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Hydrogen Sulfide	0.53	0.61	0.46	0.08	0.50	0.48	0.50	0.50	0.50
Sulfur Dioxide	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Carbon Monoxide	0.11	0.13	0.12	0.41	0.11	0.10	0.11	0.10	0.10
Carbon Dioxide	0.09	0.12	0.10	0.72	0.04	0.03	0.04	0.04	0.04
Methane	1.52	1.65	1.58	1.85	1.27	1.19	1.29	1.32	1.28
Ethylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ethane	0.60	0.66	0.63	0.70	0.49	0.47	0.51	0.53	0.52
Propylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Propane	0.39	0.43	0.40	0.48	0.29	0.29	0.32	0.32	0.32
n-Butane	0.18	0.18	0.16	0.19	0.13	0.13	0.14	0.15	0.15
i-Butane	0.05	0.05	0.05	0.05	0.04	0.04	0.04	0.05	0.05
1-Pentene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cis/Trans, 2-Pentene	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00
n-Pentane	0.06	0.05	0.05	0.07	0.04	0.04	0.04	0.05	0.05
i-Pentane	0.04	0.02	0.02	0.06	0.03	0.02	0.03	0.04	0.04
n-Hexane	0.04	0.04	0.05	0.02	0.01	0.02	0.02	0.02	0.01
2-Methyl Pentane	0.03	0.03	0.04	0.02	0.02	0.02	0.03	0.02	0.02
n-Heptane	0.01	0.01	0.05	0.00	0.01	0.01	0.01	0.01	0.00
2-Methyl Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3-Methyl Hexane	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00
Cyclo-Pentane/3Mepentane	0.04	0.05	0.06	0.03	0.02	0.02	0.03	0.02	0.02
Methyl Cyclopentane	0.03	0.04	0.09	0.01	0.01	0.02	0.02	0.02	0.02
Cyclohexane	0.07	0.07	0.00	0.03	0.04	0.06	0.06	0.05	0.04
Methyl Cyclohexane	0.040	0.056	0.00	0.028	0.056	0.094	0.093	0.051	0.024
Benzene	0.000	0.001	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Toluene	0.009	0.007	0.010	0.014	0.011	0.011	0.011	0.010	0.025
i-Butene	0.017	0.021	0.020	0.002	0.009	0.015	0.017	0.012	0.008
Tran 2-Butene	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Cis 2-Butene	0.001	0.000	0.003	0.000	0.000	0.000	0.000	0.000	0.000
Ammonia	0.002	0.005	0.003	0.001	0.006	0.005	0.019	0.007	0.019
Water	0.144	0.047	0.000	0.081	0.013	0.000	0.000	0.000	0.045

RCLU Unit Number Yield Period Number	1 516	1 517	1 518	1 519	1 520	1 521	1 522	1 523	1 524
<u>Tail Gas, Mole %</u>									
Hydrogen	91.55	90.59	90.56	90.74	89.83	89.86	92.29	92.45	92.68
Nitrogen	4.33	4.84	3.98	3.70	4.37	4.50	4.50	4.17	4.21
Argon + Oxygen	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Hydrogen Sulfide	0.60	0.70	0.52	0.56	0.60	0.56	0.57	0.59	0.58
Sulfur Dioxide	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Carbon Monoxide	0.13	0.02	0.12	0.12	0.14	0.13	0.08	0.14	0.07
Carbon Dioxide	0.04	0.06	0.06	0.07	0.07	0.06	0.05	0.09	0.07
Methane	1.74	1.93	2.47	2.50	2.59	2.51	1.24	1.18	0.94
Ethylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Ethane	0.72	0.85	1.09	1.10	1.11	1.08	0.53	0.56	0.56
Propylene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Propane	0.48	0.56	0.69	0.71	0.69	0.68	0.30	0.33	0.34
n-Butane	0.20	0.26	0.27	0.26	0.31	0.31	0.19	0.22	0.26
i-Butane	0.05	0.07	0.07	0.07	0.09	0.09	0.07	0.07	0.08
1-Pentene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cis/Trans, 2-Pentene	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
n-Pentane	0.00	0.01	0.00	0.00	0.01	0.01	0.01	0.00	0.00
i-Pentane	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.00	0.00
n-Hexane	0.03	0.03	0.01	0.01	0.01	0.01	0.01	0.01	0.01
2-Methyl Pentane	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01
n-Heptane	0.01	0.00	0.01	0.01	0.01	0.02	0.02	0.03	0.03
2-Methyl Hexane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3-Methyl Hexane	0.00	0.00	0.00	0.00	0.01	0.03	0.00	0.00	0.00
Cyclopentane/3Mepentane	0.02	0.02	0.02	0.03	0.02	0.02	0.02	0.02	0.02
Methyl Cyclopentane	0.01	0.01	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Cyclohexane	0.01	0.01	0.04	0.04	0.02	0.00	0.02	0.03	0.01
Methyl Cyclohexane	0.003	0.004	0.020	0.024	0.022	0.019	0.019	0.018	0.022
Benzene	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000	0.000
Toluene	0.012	0.012	0.010	0.010	0.009	0.010	0.011	0.010	0.010
i-Butene	0.000	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000
Tran 2-Butene	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Cis 2-Butene	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Ammonia	0.015	0.002	0.001	0.016	0.021	0.022	0.009	0.015	0.009
Water	0.020	0.008	0.039	0.002	0.049	0.035	0.014	0.052	0.050

APPENDIX B

DESCRIPTION OF CLPP

The Coal Liquefaction Pilot Plant (CLPP) was designed to continuously process one ton of coal per day in a donor liquefaction scheme utilizing an intermediate boiling coal-derived liquid fraction as recycle solvent. Discussed below is a general description of the process steps involved in the CLPP, and a process flow diagram is presented in Figure B-1.

General Processing Sequence

Sized and dried coal feed is slurried with a recycle hydrogenated solvent oil fraction. The resulting coal slurry mixture is pressurized and preheated with the hydrogen feed stream where the coal is dissolved and hydrogenated. Excess hydrogen from liquefaction is sent to the recycle gas clean-up system and liquefaction slurry products are taken to vacuum flash separations. The primary purpose of the vacuum flash system is to separate the 1000°F and higher boiling fraction, including mineral matter, and undissolved coal, from the lower boiling fractions. An option provides for separating a 700-1000°F stream from the minus vacuum-flashed vapors.

The liquids from liquefaction separations and the solids-free 700°F minus fraction from vacuum flash separations is charged to a fractionation system for removal of the naphtha product. A portion of the middle distillate material from fractionation is charged along with feed hydrogen to a catalytic hydrotreater. Mild hydrotreating serves to replenish the hydrogen content of the recycle solvent. Excess feed hydrogen from hydrotreating is recycled to the gas clean-up system.

The recycle gas clean-up system uses a monethanol amine (MEA) absorber and an MEA regeneration system to remove acid gases from recycle gas streams. The purified recycle gas stream is supplemented with fresh make-up hydrogen, recompressed in the recycle compression system and used as fresh feed to the respective reactors.

A low pressure gas scrubbing system is used to recover light hydrocarbon liquid components from the product gas stream prior to discharge into the vent system.

Coal Feed Preparation

Coal feed to the CLPP is provided by a separate coal preparation facility. The coal is handled and prepared in an inert atmosphere. The nominal feed coal provided by this facility contains less than one weight percent moisture and has a 30 mesh top size with 60 to 95 weight percent through 100 mesh.

Coal Storage and Feed

CLPP feed coal from the coal preparation facilities is stored in an 8-ton capacity storage hopper. The storage hopper and subsequent coal feed equipment is blanketed with an inert gas (nitrogen) to inhibit air oxidation. The remotely stored CLPP feed coal is transferred via a tubular pneumatic conveyor to a continuously weighed 2-ton capacity surge hopper. Periodically, feed coal from the surge hopper is dumped into a continuously weighed one-ton capacity feed hopper (H-1). A star feeder (CF-1) continually meters coal from the feed hopper to the slurry preparation system (M-1). Coal feed rates are obtained by monitoring the change in feed hopper weight.

Slurry Preparation, Pumping and Preheat

The design feed coal rate is one ton-per-day (dry basis); however, coal feed rates of 60% to 150% of this rate have been used during the operation of the unit.

The slurry mixture is continually prepared at atmospheric pressure and temperatures normal to that of the recycle solvent (less than 125°F). Centrifugal recirculating pumps are used to maintain solids suspension in the slurry mix tank. Moyno pumps (P-1) are used to provide suction pressure for the high pressure slurry feed pumps (P-2). A viscosity measuring instrument is mounted in the slurry mix tank to detect changes and upsets in solvent to coal feed ratios.

Air fluidized, sandbath preheaters using electrical resistance immersion heating elements are used to separately preheat slurry (E/H-2) and feed hydrogen stream (E/H-1). The unit is also capable of preheating the hydrogen and slurry as a mixed phase in a single preheater. The maximum design sandbath temperature is limited to 1000°F.

Liquefaction Reactors and Product Depressurization

The donor liquefaction reactor system is designed for a 3000 psig operating pressure at 900°F. The reactors (R-1) are fabricated from four equal lengths of 316 stainless steel pipe. The residence time in the reactors can be varied by bypassing from one to three of the liquefaction reactors.

Liquefaction product depressurization is performed in two stages. The high pressure separation system (S-1) uses a cycling lock valve operation to depressure the slurry product to the second stage separator pressure. High pressure product gases, light hydrocarbons, and water are cooled in multiple stage, flooded coolers (E/C-20-21) prior to separation of the hydrocarbons and water (S-10). The presence of chloride in liquefaction overhead gases requires provisions to prevent stress chloride cracking of

metal walls at locations of incipient condensation of water. All tubing between the high pressure separator and the coolers is fabricated from carpenter 20 steel. As an additional safety precaution, a continuous recycle water stream is provided to quench the gas and flow through this section of the system. The cooled high pressure liquefaction gases are subsequently processed in the recycle gas clean-up system.

The low pressure separator (S-2) operates at 100 psig and 700°F. Hot slurry product is continuously depressured through a single control valve. Flash gases and light hydrocarbons present in the low pressure separator (E/C-1, S-3). The heavier hydrocarbons recovered from this flash are charged as part of the feed to the hydrotreater (A-3).

Vacuum Flash Separations

The vacuum flash system is designed to operate at 25 mm Hg pressure, yet has operated at considerably higher vacuum during specialized studies. The primary flash separations step (F-1) operates at a maximum feed zone temperature of 750°F and separates the mineral matter and 1000°F+ liquefaction product from the lower boiling fractions. A downflow impingement-type feed nozzle with baffles on the walls was used. The upper vessel diameter is designed for subcritical (4-6 ft/sec) upward velocities.

The primary vacuum flash separator bottoms level is monitored with a nuclear level detector, and a gear pump (P-4) is used to remove 1000°F+ material from the vessel. The bottoms is then discharged into a Sandvik belt where it is cooled and solidified.

Facilities are also provided to separate a 700°F to 1000°F side stream from the lighter material (F-2). Because of improved quality resulting from solids removal, the 1000°F minus liquefaction product allows for the use of vessel internals and packing in the side stream removal facilities. Likewise, conventional level control devices are used and a gear pump is used to handle the 700°F to 1000°F fraction. Provision is also made for continuous monitoring of the viscosity of the vacuum flash bottoms.

The 700°F minus overhead from vacuum flash is cooled and separated from noncondensable gases in a packed column (V-1). The cooling column uses one-inch metal Pall rings and a two-stage coolant recycle loop (E/C-2 and E/C-3). The condensed and cooled overhead flash products are accumulated and pumped to the hydrotreater for subsequent upgrading (A-3).

Hydrotreater and Product Flash Separations

The hydrotreater reactor (R-2) is designed for a 3000 psig operating pressure at 900°F. The reactor is fabricated from two sections of 316 stainless steel pipe and interconnected with 316 stainless steel tubing. Each reactor section has a single catalyst bed with appropriate quench

hydrogen and product oil injection points spaced along the length of each section for temperature control. Catalyst bed pressure drop measurements are taken across each section of reactor.

Facilities are provided to remove water from hydrotreater product prior to fraction (S-5).

Recycle Gas Clean-up and Comparison

High pressure product gases from liquefaction and hydrotreating are scrubbed with a water solution of monethanol amine (MEA) to remove acid gases. The MEA gas scrubber (V-4) is designed for a 2000 psig operating pressure at 150°F with operations at 1000-1500 psig. Intalox saddles are used in the scrubber to provide efficient gas/liquid contacting. MEA concentration, solution feed rate, and corresponding scrubber dimensions are chosen such that 99+ mole percent of entering hydrogen sulfide and carbon dioxide gases are removed. Concentration of gases not recoverable with MEA scrubbing, such as methane through butane hydrocarbon gases and CO are continually purged in a slip-stream prior to MEA scrubbing. The MEA scrubbing system is designed for closed loop recycle of MEA solution via a regenerator (S-9).

The recycle gas compression system (C-4, 5, 6) is designed to handle the combined streams from recycle scrubbing plus fresh hydrogen make-up. Maximum design compression capacity at 3100 psig discharge pressure and 1300 psig, 100°F suction conditions is approximately 100 SCFM (95% by volume minimum hydrogen purity).

Low Pressure Gas Scrubbing

The low pressure gas scrubbing system (V-3) recovers light condensable hydrocarbons from low pressure gas streams prior to final metering and discharge to the vent. The system is designed for 25 psig operating pressure and a temperature range from 40°F to 150°F. Rich oil from the scrubber is pumped to the naphtha/solvent fractionator.

Fractionation

The naphtha/solvent fractionator (F-4) is designed to remove the light front end components from the solvent stream prior to hydrogenation. The fractionator is designed for a 10 psig operating pressure. The fractionator is capable of producing a solvent range fraction containing less than 5 cumulative weight percent 400°F minus material with 99+ weight percent retention of the tetralin in the total fractionation feed.

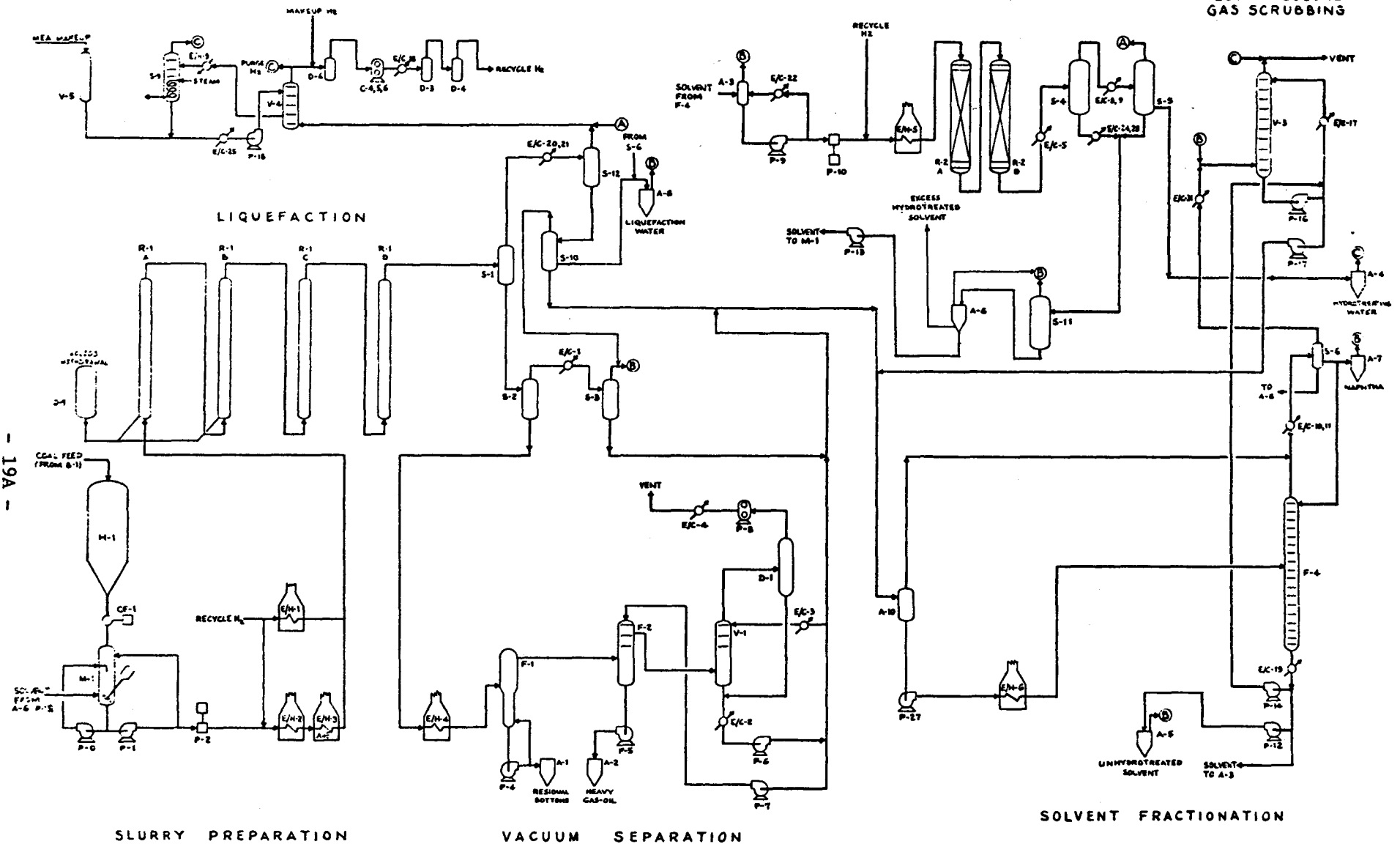
Provisions for packed zone pressure drop monitoring as well as for downstream naphtha-water separation (S-6) are included in the fractionator design.

CLPP FLOW DIAGRAM

HIGH PRESSURE GAS SCRUBBING AND COMPRESSION

SOLVENT HYDROTREATING

LOW PRESSURE GAS SCRUBBING



SLURRY PREPARATION

VACUUM SEPARATION

SOLVENT FRACTIONATION

APPENDIX C

TABULATED RESULTS - BENCH SCALE HYDROGENATION STUDIES

This appendix contains tabular data from bench-scale solvent hydrogenation studies discussed in Section 3. Data included for each run includes catalyst charge, solvent type, operating conditions and product analyses. An explanation of data table headings is given in Table C.

TABLE C

EXPLANATION OF TABLE HEADINGS FOR TABLES C-2 THROUGH C-4

Yield Period:	A yield period number is assigned for each material balance run during the life of each unit. The average length of a yield period is 17 hours.
Day:	Days on oil during run.
LMHSV, % of RV:	(Liquid Mass Hourly Space Velocity) Weight of liquid feed/hr/weight of catalyst (shown as percentage of a RV).
Average Temp. -RV, °F:	The arithmetic average of the temperatures in the catalyst bed (shown as difference from a RV).
Pressure, psig:	Reactor pressure. Where two values are given these correspond to the pressure observed at the start and end of a yield period.
Hydrogen Treat, % of RV:	Standard cubic feed of treat gas per barrel of liquid feed (shown as percentage of a RV).
Liquid Material Balance:	(Wt. of product liquid/wt. of feed liquid) x 100%.
Sp. Gr. Product:	Specific gravity 60/60 determined using hydrometers and corrected using Fisher/Tag manual.
Hydrogen Consumed SCF/bbl:	Standard cubic feed of treat gas consumed per barrel of liquid feed (calculated).
<u>Wt.% of Product Liquid</u>	
Saturates:	Wt.% saturates in product liquid, by Mass Spectrometry.
Sulfur:	Wt.% sulfur in product liquid, by X-ray Fluorescence.
Nitrogen:	Wt.% nitrogen in product liquid by Dohrman microcoulometric analysis.
Oxygen:	Wt.% oxygen in product liquid, by neutron activation analyses.
Carbon:	Wt.% carbon in product liquid, by ASTM D-3178 Method.
Hydrogen:	Wt.% hydrogen in product liquid, by ASTM D-3178 Method.
Donor Hydrogen Index, % of RV:	Determined by Mass Spectrometry (shown as percentage of a RV).

TABLE C-1

FEEDSTOCKS FOR SOLVENT HYDROGENATION STUDIES

Designation	<u>MPSS</u>	<u>Illinois Spent Solvent</u>	<u>Wyoming Spent Solvent</u>	<u>Heavy Solvent</u>
Carbon, Wt.%	88.69	89.42	89.53	87.73
Hydrogen, Wt.%	8.77	9.97	9.73	8.26
Sulfur, Wt.%	0.3	0.23	.019	0.48
Nitrogen, Wt.%	0.27	0.23	.103	0.54
Oxygen, Wt.%	1.6	1.0	0.91	2.29
Donatable Hydrogen, Wt.%	105.3 x RV	118.4 x RV	115.8 x RV	105.3 x RV
Saturates, Wt.%	19.9	27.1	23.98	18.74
1 Ring Aromatics, Wt.%	32.4	29.9	35.73	16.84
2 Ring Aromatics, Wt.%	38.8	30.3	32.87	36.26
3 Ring Aromatics, Wt.%	7.2	8.2	6.36	17.88
4 Ring Aromatics, Wt.%	1.8	3.9	1.06	8.97
GC Distillation, Wt.% overhead at T°F:				
400	10.2	10.4	17.2	7.1
500	46.5	49.7	60.9	31.6
600	71.8	74.6	80.6	48.4
700	86.6	88.0	92.4	70.2
800	94.1	95.8	97.5	85.8
900				95.2

EXPERIMENTAL RESULTS, RUN 12M22A

Yield Period		Day	WT% OF PRODUCT LIQUID					H ₂	Donor H ₂ % of RV
			<u>Saturates</u>	<u>Sulfur</u>	<u>Nitrogen</u>	<u>Oxygen</u>	<u>Carbon</u>		
562	1								
			LOST SOME OF PRODUCT ON ACCUMULATOR FROM A LEAKY FITTING						
563	2		36.75	.015	.03	.01	89.1	10.8	184
564	3								
565	4								
566	5		36.69	.009	.03	.02	89.1	10.8	191
567	6								
568	7								
569	8		31.32	.008	.09	.23	89.3	10.4	174
570	9		30.83	.007	.09	.26	89.3	10.4	178
571	10								
572	11		30.61	.007	.09	.50	89.3	10.4	171
573	12								
574	13								
575	14								
576	15								
577	16								
578	17								
579	18		30.12	.008	.10	.31	89.3	10.4	175
580	19								
581	20								
582	21								
583	22		29.46	.009	.08	.40	89.3	10.3	175
584	23								
			LOST H2 COMPRESSOR DUE TO POWER FAILURE						
585	24		30.15	.007	.08	.42	89.3	10.4	178
586	25		30.37	.005	.08	.40	89.3	10.4	172
587	26								
588	27								
589	28								
590	29		34.40	.002	.04	.01	89.1	10.7	180
591	30		34.66	.003	.05	.11	89.1	10.6	174
592	31		34.31	.003	.05	.12	89.1	10.6	180
593	32								
594	33								
595	34		33.95	.005	.05	.11	89.1	10.6	182
596	35								
597	36		38.50	.002	.01	.01	89.1	10.8	164
598	37		37.57	.002	.01	.01	89.1	10.7	157
599	38								
600	39		37.55	.01	.01	.01	89.0	10.7	164
601	40								
602	41								
603	42								
604	43		37.27	.002	.01	.01	89.0	10.7	167
605	44		36.22	.004	.01	.02	89.0	10.7	174
606	45		37.04	.004	.01	.02	89.0	10.7	167
607	46		36.95	.002	.02	.01	89.0	10.7	164
608	47		36.77	.01	.01	.01	89.1	10.7	163
			ACCUMULATORS WERE NOT SWITCHED AT START OF YP						
609	48								
610	49		36.24	.001	.02	.01	86.1	1	

EXPERIMENTAL RESULTS, RUN 12M22A

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H2 Treat SCF/bbl % of R.V.	Liquid M³ Bal.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
562	1	132	112	1435	95.3	87.1	0.933	
		LOST SOME OF PRODUCT ON ACCUMULATOR FROM A LEAKY FITTING						
563	2	133	114	1460	95.0	88.0	0.932	
564	3	130	114	1500	96.6	96.2	0.933	
565	4	128	114	1480	98.8	100.8	0.934	
566	5	133	114	1460	94.4	92.5	0.930	
567	6	262	114	1490	92.8	96.6	0.941	
568	7	261	114	1490	93.4	97.7	0.943	
569	8	263	115	1490	92.2	98.1	0.943	401
570	9	262	115	1490	95.6	98.5	0.943	432
571	10	262	115	1490	95.6	97.5	0.943	
572	11	261	114	1490	93.1	99.2	0.943	428
573	12	262	114	1490	92.8	97.7	0.944	
574	13	266	115	1490	93.8	99.2	0.944	
575	14	262	115	1460	92.8	97.3	0.943	
576	15	276	115	1490	87.8	97.8	0.945	
577	16	263	115	1490	91.6	96.6	0.944	
578	17	262	115	1490	92.8	98.5	0.943	
579	18	262	115	1480	91.6	98.5	0.946	389
580	19	261	115	1490	92.8	107.7	0.947	
581	20	262	115	1480	92.8	98.2	0.947	
582	21	259	114	1480	93.8	99.3	0.947	
583	22	263	114	1490	91.6	94.8	0.945	554
584	23	259	114	1490	93.8	98.2	---	
		LOST H2 COMPRESSOR DUE TO POWER FAILURE						
585	24	261	115	1490	93.1	98.5	0.946	402
586	25	261	115	1480	92.8	99.2	0.947	437
587	26	263	115	1480	92.2	96.2	0.946	
588	27	262	115	1490	92.8	98.5	0.947	
589	28	262	115	1490	92.8	100.0	0.947	
590	29	263	166	1490	92.2	98.5	0.933	573
591	30	263	167	1490	92.8	98.5	0.934	556
592	31	263	166	1480	92.8	93.0	0.937	594
593	32	262	166	1480	91.6	98.8	0.936	
594	33	262	167	1475	92.8	97.8	0.938	
595	34	262	166	1480	92.8	100.4	0.938	693
596	35	263	215	1490	91.6	100.0	0.925	
597	36	262	215	1490	92.8	89.3	0.925	
598	37	262	215	1490	92.8	93.9	0.927	747
599	38	262	215	1490	92.8	96.6	0.928	
600	39	263	215	1490	92.2	96.1	0.926	568
601	40	261	215	1490	93.1	97.5	0.927	
602	41	259	215	1490	93.4	95.0	0.929	
603	42	262	215	1490	94.4	96.0	0.929	
604	43	264	215	1510	93.1	95.7	0.929	608
605	44	261	215	1490	96.6	97.5	0.927	626
606	45	262	215	1500	92.8	96.6	0.927	
607	46	258	215	1490	94.4	97.8	0.930	632
608	47	261	216	1490	96.6	176.7	0.929	
		ACCUMULATORS WERE NOT SWITCHED AT START OF YP						

TABLE C-3

EXPERIMENTAL RESULTS, RUN 12M22B

Treat Gas: 2% Co + 2% CO₂ balanced with H₂

Base Feed: Illinois Spent Solvent

WT% OF PRODUCT LIQUID

Yield Period	Day	Saturates	Sulfur	Nitrogen	Oxygen	Carbon	H ₂	Donor H ₂ % of R.V.
562	1							
563	2	35.49	.011	.06	.01	89.2	10.7	176
564	3							
565	4							
566	5	35.22	.010	.04	.05	89.1	10.7	191
567	6							
568	7							
569	8	30.75	.011	.07	.30	89.3	10.4	174
570	9	30.42	.010	.10	.37	89.2	10.4	175
571	10							
572	11	30.03	.008	.09	.29	89.3	10.3	166
573	12							
574	13							
575	14							
576	15							
577	16							
578	17							
579	18	30.07	.008	.10	.35	89.3	10.3	171
580	19							
581	20							
582	21							
583	22	30.60	.012	.09	.47	89.2	10.4	167
584	23							
LOST H ₂ COMPRESSOR DUE TO POWER FAILURE								
585	24	29.53	.018	.10	.50	89.3	10.3	167
586	25	29.61	.013	.09	.46	89.3	10.3	168
587	26							
588	27							
589	28							
590	29	32.63	.007	.06	.07	89.2	10.5	175
591	30	32.34	.006	.07	.20	89.2	10.5	170
592	31	32.15	.007	.07	.17	89.2	10.5	175
593	32							
594	33							
595	34	31.81	.004	.06	.13	89.2	10.6	187
596	35							
597	36	33.28	.002	.03	.01	89.2	10.5	167
598	37	33.96	.010	.03	.01	89.2	10.5	161
599	38							
600	39	37.57	.002	.03	.01	89.0	10.7	166
601	40							
602	41							
603	42							
604	43	34.99	.002	.03	.01	89.1	10.7	176
605	44	33.80	.002	.04	.05	89.2	10.5	170
606	45	34.82	.002	.03	.01	89.2	10.6	168
607	46	37.00	.002	.03	.01	89.0	10.7	168
608	47	34.97	.005	.04	.04	89.1	10.6	164
ACCUMULATORS WERE NOT SWITCHED AT START OF YP								
609	48							
610	49	34.18	.003	.03	.01	89.2	10.6	170
611	50	34.22	.003	.03	.01	89.2	10.6	171
UNIT SHUT DOWN VOLUNTARILY								

TABLE C-3

EXPERIMENTAL RESULTS, RUN 12M22B

Catalyst: Ni/Mo-10

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H ₂ Treat SCF/bbl % of R.V.	Liquid M Bal.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
562	1	129	111	1480	95.3	100.7	0.935	
563	2	130	112	1480	95.0	99.2	0.934	
564	3	129	114	1480	95.6	100.8	0.934	
565	4	128	114	1480	96.6	100.0	0.931	
566	5	130	114	1480	95.0	95.5	0.934	
567	6	257	114	1475	92.8	98.1	0.934	
568	7	257	113	1490	92.8	98.1	0.945	
569	8	257	114	1480	92.8	100.4	0.945	513
570	9	257	114	1490	92.8	99.2	0.945	443
571	10	255	113	1490	93.1	99.3	0.945	
572	11	257	114	1490	93.1	100.0	0.945	452
573	12	257	113	1490	92.8	99.2	0.945	
574	13	257	114	1490	92.8	99.2	0.945	
575	14	257	114	1480	92.8	98.5	0.945	
576	15	271	114	1490	87.8	98.9	0.946	
577	16	254	114	1490	93.4	98.1	0.945	
578	17	257	114	1490	92.8	97.7	0.947	
579	18	262	114	1480	86.3	98.5	0.947	372
580	19	257	114	1480	92.8	107.7	0.946	
581	20	257	114	1480	92.8	98.2	0.947	
582	21	255	114	1480	93.1	99.6	0.947	
583	22	254	114	1480	93.4	97.7	0.948	427
584	23	255	113	1490	93.4	89.5	---	
LOST H ₂ COMPRESSOR DUE TO POWER FAILURE								
585	24	257	114	1490	93.1	99.6	0.950	388
586	25	258	114	1480	86.3	101.6	0.949	393
587	26	262	114	1480	92.2	99.2	0.947	
588	27	257	113	1480	92.8	98.5	0.947	
589	28	258	114	1480	92.8	101.5	0.948	
590	29	257	165	1480	92.2	99.2	0.936	559
591	30	257	165	1490	92.8	101.5	0.940	554
592	31	257	165	1460	92.8	100.4	0.940	604
593	32	257	165	1480	86.3	102.8	0.939	
594	33	257	165	1480	92.8	100.0	0.938	
595	34	257	165	1480	92.8	100.8	0.939	666
596	35	258	213	1490	86.3	108.0	0.931	
597	36	257	214	1480	92.8	98.9	0.931	542
598	37	254	214	1480	93.4	92.0	0.933	
599	38	257	214	1490	92.8	98.9	0.931	
600	39	258	214	1480	92.2	97.1	0.930	596
601	40	257	214	1490	92.8	99.7	0.932	
602	41	254	214	1480	93.4	97.7	0.932	
603	42	257	214	1470	92.8	97.6	0.927	
604	43	259	214	1470	91.6	97.6	0.930	585
605	44	255	214	1475	86.6	98.2	0.930	545
606	45	257	214	1480	92.8	98.1	0.932	
607	46	254	214	1480	87.2	99.3	0.934	693
608	47	257	214	1470	86.3	163.9	0.933	
ACCUMULATORS WERE NOT SWITCHED AT START OF YP								
609	48	257	214	1470	86.3	95.1	0.930	
610	49	258	214	1480	85.9	98.5	0.932	579
611	50	262	214	1470	91.0	94.3	0.931	
UNIT SHUT DOWN VOLUNTARILY								

TABLE C-4

EXPERIMENTAL RESULTS, RUN 13M22A

Base Feed: Illinois Spent Solvent

Yield Period	Day	WT% OF PRODUCT LIQUID						
		Saturates	Sulfur	Nitrogen	Oxygen	Carbon	H ₂	Donor H ₂ % of R ₂
612	1							
613	2							
614	3	37.06	.004	.03	.13	89.0	10.9	193
615	4							
616	5	36.58	.006	.04	.14	89.0	10.9	191
617	6	30.34	.010	.10	.42	89.3	10.4	171
618	7	30.04	.010	.10	.47	89.3	10.3	172
619	8	29.70	.010	.10	.44	89.3	10.3	170
620	9	29.67	.011	.11	.42	89.3	10.3	176
621	10	31.28	.007	.06	.32	89.2	10.6	198
622	11	31.37	.008	.07	.32	89.2	10.5	178
623	12	31.39	.008	.08	.34	89.2	10.5	176
624	13	32.55				89.2	10.5	167
625	14	36.55				89.0	10.9	195
626	15	36.06				89.0	10.7	180

TABLE C-4

EXPERIMENTAL RESULTS, RUN 13M22A

Catalyst: Ni/Mo-10

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H ₂ Treat SCF/bbl	Liquid M'Bal.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
					% of R.V.			
612	1	122	115	1490	112.5	97.7	0.925	
613	2	122	115	1500	112.5	97.8	0.932	
614	3	125	115	1490	110.0	98.5	0.933	742
615	4	125	115	1500	99.1	98.5	0.931	
616	5	125	115	1500	99.1	97.0	0.933	617
617	6	417	124	1500	89.4	97.8	0.948	327
618	7	418	124	1480	89.4	96.8	0.949	250
619	8	413	122	1480	90.3	99.3	0.948	393
620	9	416	124	1480	89.7	97.9	0.948	325
621	10	250	124	1490	92.8	98.4	0.943	513
622	11	250	124	1490	93.1	98.0	0.944	422
623	12	250	124	1490	92.5	98.8	0.943	466
624	13	250	124	1500	92.5	93.9	0.941	
625	14	125	124	1490	92.8	97.0	0.933	
626	15	126	124	1500	92.0	95.5	0.933	

TABLE C-5

EXPERIMENTAL RESULTS, RUN 13M22B

Treat Gas: 2% CO + 2% CO₂ balanced with H₂

Base Feed: Illinois Spent Solvent

WT% OF PRODUCT LIQUID								
Yield Period	Day	Saturates	Sulfur	Nitrogen	Oxygen	Carbon	H ₂	Donor H ₂ % of RV
612	1							
613	2							
614	3	37.34	.029	.03	.12	89.0	10.8	183
615	4							
616	5	36.20	.006	.04	.14	89.0	10.8	192
617	6	30.13	.053	.10	.46	89.3	10.3	164
618	7	29.70	.029	.11	.45	89.3	10.3	166
619	8	30.11	.013	.11	.46	89.3	10.3	166
620	9	29.34	.013	.11	.51	89.3	10.3	174
621	10	30.36	.013	.07	.35	89.3	10.4	184
622	11	30.53	.011	.08	.35	89.3	10.4	178
623	12	31.14	.008	.08	.36	89.2	10.4	176
624	13	32.11				89.2	10.5	172
625	14	34.48				89.1	10.7	186
626	15	34.11				89.1	10.7	186

TABLE C-5

EXPERIMENTAL RESULTS, RUN 13M22B

Catalyst: Ni/Mo-10

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H ₂ Treat SCF/bbl % of R.V.	Liquid M'Bal.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
612	1	121	115	1480	111.6	96.9	0.926	
613	2	122	116	1480	110.9	97.1	0.933	
614	3	124	115	1470	100.0	99.3	0.928	773
615	4	126	115	1480	97.5	95.5	0.931	
616	5	125	115	1480	99.1	97.7	0.933	653
617	6	412	124	1470	89.7	102.2	0.949	357
618	7	414	124	1470	89.4	96.4	0.950	186
619	8	409	124	1470	90.3	98.4	0.950	330
620	9	412	124	1470	89.7	97.5	0.950	267
621	10	249	124	1470	92.5	98.4	0.943	427
622	11	246	124	1480	93.4	100.4	0.944	520
623	12	249	124	1480	92.5	98.1	0.943	411
624	13	249	124	1480	89.7	95.1	0.943	
625	14	122	124	1470	94.4	97.7	0.937	
626	15	125	124	1470	92.0	97.0	0.934	

TABLE C-6

EXPERIMENTAL RESULTS, RUN 14M21

Base Feed: MPSS

WT% OF PRODUCT LIQUID

Yield Period	Day	Saturates	Sulfur	Nitrogen	Oxygen	Carbon	H ₂	Donor H, % of R ₂
1011	212							
1012	213							
1013	214							
1014	215							
1015	216	24.77	.011	.10	.40	89.7	9.9	187
1016	217	24.30	.010	.11	.42	89.6	9.9	184
1017	218	23.53	.015	.12	.54	89.7	9.8	179
1018	219	28.90	.005	.05	.10	89.5	10.2	196
1019	220	27.84	.006	.06	.13	89.5	10.2	192
1020	221	28.60	.009	.06	.11	89.6	10.1	184
1021	222	28.08	.008	.06	.15	89.6	10.1	182
1022	223	24.56	.013	.09	.34	89.7	9.9	187
1023	224	24.50	.013	.10	.45	89.7	9.8	182
1024	225							
1025	226							
1026	227							
1027	228							
1028	229							
1029	230							
1030	231	25.66	.018	.13	.66	89.5	9.9	167
1031	232	23.18	.019	.12	.65	89.7	9.7	170
1032	233	23.10	.017	.13	.77	89.7	9.7	175
1033	234	25.39	.012	.28	.44	89.6	10.0	192
1034	235	25.26	.011	.10	.50	89.6	9.9	184
1035	236	25.10	.010	.10	.39	89.6	9.9	189
1036	237	25.59	.014	.10	.42	89.6	10.0	191
1037	238	24.96	.015	.12	.44	89.6	9.9	180
1038	239	24.17	.018	.12	.51	89.7	9.8	182
1039	240	25.12	.010	.10	.35	89.6	9.9	187
1040	241							
1041	242							
1042	243							
1043	244							
1044	245							
1045	246							
1046	247	25.02	.009	.09	.38	89.6	9.9	189
1047	248	24.82	.010	.08	.34	89.6	9.9	186
1048	249							
1049	250							
1050	251							
1051	252	24.92	.008	.09	.37	89.7	9.9	187
1052	253	24.92	.008	.09	.32	89.6	9.9	176
1053	254							
1054	255							
1055	256							
1056	257							

TABLE C-6

EXPERIMENTAL RESULTS, RUN 14M21

Catalyst: Ni/Mo-10

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H ₂ Treat SCF/bbl % of R.V.	Liquid M ³ /Bbl.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
1011	212	405	190	1500	90.6	98.3	0.957	
1012	213	403	191	1500	91.3	98.3	0.956	
1013	214	399	190	1500	92.2	98.6	0.956	
1014	215	399	191	1500	92.5	99.5	0.955	
1015	216	403	190	1500	91.3	99.9	0.956	926
1016	217	438	191	1500	116.6	98.9	0.955	869
1017	218	501	191	1500	101.9	98.7	0.960	810
1018	219	199	189	1510	123.8	100.7	0.945	1167
1019	220	237	190	1510	103.4	92.9	0.948	1190
1020	221	188	190	1510	92.5	94.4	0.948	1206
1021	222	188	189	1510	92.5	91.5	0.948	1152
1022	223	428	190	1500	84.1	94.5	0.956	846
1023	224	417	191	1500	86.3	97.6	0.958	818
1024	225	412	191	1500	87.5	99.5	0.958	
1025	226	413	191	1500	86.9	99.0	0.959	
1026	227	407	190	1500	88.4	97.6	0.957	
1027	228	389	191	1500	92.5	98.8	0.955	
1028	229	386	191	1500	93.4	99.5	0.957	
1029	230	393	191	1500	91.6	98.0	0.956	
1030	231	396	166	1500	91.0	98.8	0.965	772
1031	232	403	165	1500	89.4	97.6	0.965	660
1032	233	399	166	1500	90.3	99.1	0.965	671
1033	234	237	165	1500	103.4	98.3	0.964	843
1034	235	233	166	1500	105.3	100.2	0.955	907
1035	236	238	166	1500	103.1	97.4	0.956	790
1036	237	213	166	1500	86.6	100.2	0.955	940
1037	238	228	166	1500	81.3	98.4	0.957	829
1038	239	211	166	1510	87.5	98.0	0.959	792
1039	240	391	191	1500	91.9	99.1	0.954	908
1040	241	363	192	1500	99.4	98.1	0.953	
1041	242	368	191	1510	97.8	98.0	0.953	
1042	243	366	191	1510	98.1	98.1	0.954	
1043	244	364	191	1500	99.0	98.0	0.954	
1044	245	355	192	1510	101.3	100.1	0.954	
1045	246	357	192	1510	101.0	98.4	0.953	
1046	247	392	192	1510	91.9	96.4	0.955	903
1047	248	397	192	1510	90.6	97.9	0.955	891
1048	249	380	192	1510	95.1	98.6	0.955	
1049	250	378	192	1510	95.3	97.5	0.954	
1050	251	367	192	1510	98.1	97.6	0.954	
1051	252	388	197	1500	92.8	96.0	0.954	875
1052	253	399	197	1510	90.3	96.1	0.955	868
1053	254	391	197	1510	91.9	97.7	0.954	
1054	255	386	197	1500	93.4	97.9	0.954	
1055	256	380	197	1500	95.3	98.9	0.954	
1056	257	380	197	1490	95.1	98.0	0.954	

TABLE C-6 (cont.)

EXPERIMENTAL RESULTS, RUN 14M21

Base Feed: MPSS

Yield Period	Day	WT% OF PRODUCT LIQUID						Donor H ₂ % of R.V.
		Saturates	Sulfur	Nitrogen	Oxygen	Carbon	H ₂	
1057	258							
1058	259							
1059	260	25.19	.009	.09	.33	89.6	9.9	184
1060	261							
HIGH TEMP. ALARM WENT OFF - NO SAMPLE								
1061	262							
1062	263							
1063	264							
1064	265	24.93	.009	.09	.42	89.6	9.9	188
1065	266							
1066	267							
SHORT YIELD PERIOD - INSTALLED NEW HIGH TEMP. CONTROL								
1067	268	24.43	.008	.10	.35	89.6	9.9	191
1068	269							
1069	270							
1070	271							
1071	272	24.47	.023	.10	.46	89.6	9.9	187
1072	273	26.97	.004	.06	.27	89.6	10.1	195
CHANGED FEED RATE								
1073	274	26.36	.007	.07	.13	89.6	10.3	192
1074	275							
1075	276	25.61	.008	.08	.26	89.6	10.0	188
1076	277	25.02	.008	.09	.46	89.6	10.0	199
1077	278	23.65	.010	.11	.61	89.6	9.8	184
1078	279	23.78	.010	.11	.49	89.6	9.9	188
1079	280	22.47	.010	.13	.79	89.6	9.7	182
1080	281	22.71	.015	.13	.81	89.6	9.7	176
1081	282	25.84	.006	.07	.54	89.5	10.1	201
1082	283	24.90	.008	.09	.53	89.5	10.1	195
1083	284	26.61	.006	.07	.59	89.5	10.1	200
1084	285	26.65	.005	.07	.35	89.5	10.1	200
1085	286	26.41	.006	.07	.39	89.5	10.1	200
1086	287	25.93	.007	.08	.47	89.5	10.0	196
1087	288	28.76	.004	.04	.20	89.5	10.2	196
1088	289	28.26	.004	.04	.20	89.5	10.2	208
1089	290	29.98	.003	.03	.18	89.5	10.3	199
1090	291	29.64	.002	.03	.19	89.5	10.3	205
1091	292							
1092	293	24.72	.008	.09	.40	89.6	10.1	191
1093	294	24.98	.008	.08	.47	89.6	9.9	188
1094	295	24.24	.010	.09	.46	89.6	9.9	187
1095	296							
1096	297	22.33				89.6	9.7	172
1097	298	22.16				89.6	9.7	179
1098	299	22.09				89.6	9.7	176
1099	300	24.65				89.5	10.0	193
1100	301	24.81				89.6	10.0	195

Catalyst: Ni/Mo-10

TABLE C-6 (cont.)

EXPERIMENTAL RESULTS, RUN 14M21

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H ₂ Treat SCF/bbl % of R.V.	Liquid M Bal.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
1057	258	372	197	1500	96.6	98.0	0.954	
1058	259	376	198	1500	95.6	97.8	0.954	
1059	260	383	198	1500	94.0	97.4	0.954	935
1060	261	382	41	1510	94.4	98.3	---	
HIGH TEMP. ALARM WENT OFF - NO SAMPLE								
1061	262	378	197	1500	95.1	100.0	0.954	
1062	263	389	197	1500	92.2	96.4	0.954	
1063	264	387	197	1500	92.8	98.5	0.955	
1064	265	391	197	1500	92.2	99.3	0.955	904
1065	266	395	197	1500	91.3	96.1	0.955	
1066	267	391	195	1500	91.9	97.6	0.955	
SHORT YIELD PERIOD - INSTALLED NEW HIGH TEMP. CONTROL								
1067	268	399	196	1500	90.3	97.5	0.955	906
1068	269	393	196	1510	91.6	97.4	0.955	
1069	270	391	196	1510	92.2	77.1	0.955	
1070	271	404	195	1510	89.1	96.7	0.955	
1071	272	400	195	1500	90.0	98.9	0.956	
1072	273	253	195	1490	99.0	96.6	0.947	1017
CHANGED FEED RATE								
1073	274	286	197	1500	87.2	96.8	0.950	1042
1074	275	247	180	1500	100.1	96.9	0.952	
1075	276	259	180	1500	96.2	98.2	0.953	903
1076	277	274	179	1500	91.0	98.0	0.955	862
1077	278	270	164	1500	92.5	97.1	0.958	748
1078	279	279	165	1500	89.7	95.1	0.958	769
1079	280	276	150	1490	90.3	95.7	0.964	668
1080	281	278	150	1490	90.0	94.9	0.964	629
1081	282	150	149	1490	128.5	96.3	0.952	773
1082	283	176	150	1500	104.0	93.3	0.955	727
1083	284	175	164	1500	105.6	95.8	0.950	762
1084	285	180	165	1500	102.2	92.4	0.950	800
1085	286	182	165	1500	101.0	95.5	0.951	785
1086	287	207	165	1500	89.1	90.0	0.952	
1087	288	171	179	1500	107.5	95.5	0.945	857
1088	289	187	179	1500	99.0	91.4	0.945	
1089	290	183	197	1500	100.3	98.1	0.942	1165
1090	291	193	196	1510	95.0	95.3	0.943	1180
1091	292	388	196	1510	92.5	96.8	0.955	
1092	293	380	197	1510	95.1	97.1	0.955	963
1093	294	378	197	1510	95.3	96.5	0.955	950
1094	295	397	196	1510	90.6	96.8	0.949	922
1095	296	387	195	1510	93.1	100.7	0.957	
1096	297	389	165	1510	92.5	96.7	0.966	
1097	298	389	165	1500	92.5	96.0	0.967	
1098	299	387	165	1510	93.1	97.2	0.965	
1099	300	212	164	1500	94.1	97.6	0.954	
1100	301	214	164	1500	92.5	96.4	0.954	

TABLE C-6 (cont.)

EXPERIMENTAL RESULTS, RUN 14M21

Base Feed: MPSS

WT% OF PRODUCT LIQUID							
Yield Period	Day	Saturates	Sulfur	Nitrogen	Oxygen	Carbon	Donor H ₂ % of R ₂
1101	302	24.81				89.6	9.9
1102	303	25.42				89.5	10.0
1103	304						
1104	305						
1105	306	25.31	.010	.10	.51	89.6	10.0
1106	307	24.98				89.6	10.0
1107	308						
1108	309						
1109	310	25.11				89.5	10.0
UNIT SHUT DOWN VOLUNTARILY							

TABLE C-6 (cont.)

EXPERIMENTAL RESULTS, RUN 14M21

Catalyst: Ni/Mo-10

Yield Period	Day	LMHSV % of R.V.	Avg. Temp. -R.V., °F	Pressure PSIG	H ₂ Treat SCF/bbl % of R.V.	Liquid M' Bal.	Sp. Gr. Product	Hydrogen Consumed SCF/bbl
1101	302	229	164	1500	86.6	95.3	0.957	
1102	303	212	164	1500	93.8	96.5	0.954	
1103	304	213	164	1490	93.1	98.5	0.955	
1104	305	225	164	1490	88.1	95.8	0.955	
1105	306	224	164	1500	88.8	93.9	0.954	832
1106	307	225	164	1500	88.1	95.5	0.954	
1107	308	218	165	1500	91.0	97.1	0.954	
1108	309	232	164	1500	86.0	91.3	0.954	
1109	310	214	164	1500	92.5	96.8	0.955	
UNIT SHUT DOWN VOLUNTARILY								

APPENDIX D

WYOMING COAL OPERABILITY STUDIES IN OTCLU

Appendix D contains analytical and operating data for runs made in the 15 pound-per-day Once-Through Coal Liquefaction Unit (OTCLU) while studying potential solutions to operability problems associated with Wyoming coal. An analysis of the data described in the tables listed below is contained in Section 4 of this report. The data reported here is only for the studies completed in the second quarter of 1978. Data for other runs discussed in Section 4 has already been reported.¹⁾

- Table D-1: Wyoming Coal Operability Studies in OTCLU Yield Period Summaries

The process conditions for the yield periods completed during this reporting period are presented in Table D-1.

- Table D-2: OTCLU Yields, Feed Analyses and Product Analyses

Yields from OTCLU have not been checked for consistency by unit material balances. However, raw measured yields are presented for 1000°F+ bottoms, C₆ -1000°F liquids and water. Also, presented in Table D-2 are analyses of the feed solvent and coal and 700°F+ bottoms, spent solvent, and naphtha.

¹July-September 1977, Quarterly Technical Progress Report, FE-2893-3.
October-December 1977, Quarterly Technical Progress Report, FE-2893-7.
January-March 1978, Quarterly Technical Progress Report, FE-2893-12.

TABLE D-1

WYOMING COAL OPERABILITY STUDIES IN OTCLU
YIELD PERIOD SUMMARIES

Hairpin Reactor at 840°F and 1500 psig
 3.0 HCO Solvent/Coal Ratio

<u>Yield Period Number</u>	<u>Date</u>	<u>Length Hours</u>	<u>Slurry Residence Time-Minutes</u>	<u>Treat Gas Wt.% on Coal</u>
447	1-6-78	18	35	
448	1-6-78	5	44	
449	1-7-78	18	38	2.9
450	1-7-78	6	33	
451	1-8-78	17.5	36	2.8
452	1-12-78	18	41	
453	1-12-78	4.5	40	
454	1-13-78	18	37	3.0
455	1-13-78	6	38	
456	1-14-78	18	40	
457	1-14-78	6	40	
458	1-15-78	18	40	
459	1-27-78	19	46	
460	1-27-78	5	40	
461	1-28-78	1.8	--	
462	2-2-78	15.5	36	
463	2-2-78	6	40	
464	2-3-78	19	40	
465	2-3-78	5.5	39	
466	2-4-78	18	40	
467	2-4-78	5	41	
468	2-5-78	20	41	
469	2-6-78	4	41	
470	2-6-78	20	40	
471	2-19-78	19	38	
472	2-19-78	5	43	
473	2-20-78	19	47	
474	2-20-78	6	46	
475	2-21-78	18	36	
476	2-22-78	18	39	
477	2-22-78	6	41	
478	2-23-78	18	42	
479	3-18-78	18.5	42	3.3
480	3-21-78	18	38	2.9
481	4-8-78	19	49	3.8
482	4-11-78	19	54	4.2

TABLE D-2

OTCLU YIELDS, FEED ANALYSES AND PRODUCT ANALYSES

Yield Periods	449	451	454	479	480	481	482
<u>Yield, Wt.%</u>							
1000°F+ bottoms	60.1	54.1	49.3	59.9	66.9	47.8	67.2
SO ₃ -free ash balance	87.9		91.8	110.8	67.4	72.4	67.3
Liquids (=C ₆ -1000°F)	31.7	27.1	22.3	16.2	10.0	28.3	16.2
Water	15.4	13.5	11.7	7.6	7.5	8.8	7.8
Gases & C ₄ -C ₅ liquids	----- Not Determined -----						
<u>Coal</u>							
Moisture				2.15	1.62	2.64	3.11
<u>Elemental Analysis,</u>							
<u>Wt.% dry coal</u>							
Carbon	58.51		62.42	68.33		67.63	67.75
Hydrogen	4.16		4.39	4.78		4.71	4.59
Nitrogen	.77		1.12	1.01	.98	.92	.90
Sulfur	2.98		1.67	0.24	.89	.85	.84
Ash	12.40		8.80	10.34	10.14	10.19	10.12
Ash (SO ₃ -free)	10.52		6.50	8.34	8.20	8.34	8.24
Total Oxygen (N.A.)	18.79		16.53	21.54	21.23	21.00	21.86
<u>Feed Solvent</u>							
Specific Gravity 60/60							
<u>Elemental Analysis, Wt.%</u>							
Carbon (MS)				90.99	91.02	91.05	
Hydrogen (MS)				8.59	8.59	8.56	
Nitrogen (Antek)							
Sulfur (x-ray)							
Oxygen (N.A.)							
Donor Hydrogen (% RV)				196	196	204	199
G.C. Distillation							
(% off @ 400°F)				8.29	16.78	10.88	
(% off @ 700°F)				92.18	89.39	89.8	
<u>700+ Bottoms</u>							
<u>Elemental Analysis, Wt.%</u>							
Carbon					82.77	82.78	83.20
Hydrogen					5.84	5.78	5.82
Nitrogen				.75	.75	.74	.78
Sulfur				.23	.39	.71	.82
Ash	11.39	10.37	7.26	11.3	5.71	7.14	
Ash (SO ₃ -free)	9.07		5.88	10.08	5.10	5.84	
Total Oxygen (N.A.)			5.42	5.97		6.58	
Microcube Distillation							
(% off @ 1000°F)	41.0	39.8	46.2	34.7	38.3	46.2	39.3
<u>Spent Solvent Analysis</u>							
Specific Gravity 60/60				1.0389	1.0209		1.0224
<u>Elemental Analysis, Wt.%</u>							
Carbon (MS)	91.21	91.14		90.99		90.56	91.16
Hydrogen (MS)	8.22	8.20		8.59		8.01	8.25
Nitrogen (Antek)	.18	.02				.02	
Sulfur (x-ray)	.029					.032	
Oxygen (N.A.)	.88					.90	
G.C. Distillation							
(% off @ 400°F)	4.35	3.88	14.19				
<u>Naphtha Analysis</u>							
Specific Gravity 60/60			0.8978	0.8973	0.8866	0.8905	0.9001
<u>Elemental Analysis, Wt.%</u>							
Carbon (MS)	85.84	86.32	85.89	87.77	88.91	87.45	90.16
Hydrogen (MS)	10.62	10.72	10.14	10.51	9.50	10.65	9.39
Nitrogen (Antek)							
Sulfur (x-ray)							
Oxygen (N.A.)							
G.C. Distillation							
(% off @ 400°F)			73.64				

APPENDIX E

Appendix E contains the following data as referenced in Section 5 of this report:

- Table E-1: Naphtha Hydrotreating Feedstock Inspections

These feedstocks were hydrotreated at the Baton Rouge Laboratories to determine the severity necessary to achieve reformer feed specifications.

- Table E-2: Inspections of EDS Fuel Oil Fractions Used in Combustion Tests

These feedstocks were used during combustion testing in a 50 HP Cleaver-Brooks boiler at Products Research Division in Linden, New Jersey.

TABLE E-1

NAPHTHA HYDROTREATING FEEDSTOCK INSPECTIONS

	<u>Illinois Naphtha</u>		<u>Wyoming Naphtha</u>	
	<u>Raw</u>	<u>Caustic Washed</u>	<u>Raw</u>	<u>Caustic Washed</u>
Gravity, °API	42.0	45.1	38.0	40.8
Br. No., cg/g	35.6	19.3	30.6	16.0
<u>Elemental Analysis (wt.%)</u>				
C	84.41	85.80	85.04	86.58
H	12.49	12.94	11.95	12.35
S	0.88	0.58	0.12	0.11
N	0.11	0.09	0.15	0.13
O	1.52	0.16	2.08	0.58
<u>ASTM D-86, °F</u>				
IBP/5	160/200	148/192	147/200	138/190
10/20	212/233	212/233	212/245	211/238
30/40	253/272	248/265	270/288	262/284
50/60	284/300	278/295	312/328	304/322
70/80	314/328	312/325	342/351	340/356
90/95	340/350	340/350	362/372	371/389
FBP	382	410	411	450

TABLE E-2

INSPECTIONS OF EDS FUEL OIL FRACTIONS
USED IN COMBUSTION TESTS

• Illinois Coal

	<u>400/1000°F</u>	<u>400°F⁺</u>
Gravity, °API	4.6	0.07
Flash Point, °F	198	204
Carbon, Wt.%	85.52	86.13
Hydrogen, Wt.%	8.03	8.09
Sulfur, Wt.%	0.60	0.87
Nitrogen, Wt.%	0.55	0.81
Total Oxygen, Wt.%	2.47	2.74
% Ash	0.027	0.03
Con-C, Wt.%	3.61	9.98
MNI, Wt.%	8.96	20.99
Sediment by Hot Filtration, Wt.%	0.04	0.64
Sediment by Ext., Wt.%	0.05	0.27
Vis. Cst. @ 100°F	21.0	440
Vis. Cst. @ 212°F	3.09	10.4
Vis. Cst. @ 250°F	---	5.8
D-1160 Distillation		
IBP °F	396	403
10%	437	441
50	592	696
90	625	1016
95	984	---
FBP	1043	1043
% Rec.	98	92.5

APPENDIX F

CSCU EXPERIMENTAL DATA

Appendix F contains the data for Yield Tests 107-110 and 115-118 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

CSCU RUN-107

TEMPERATURE, DEG. F	RV + 160.00
PRESSURE, PSIG	11.00
FEED SOURCE	CLPP YP- 263(840 F/ 40 MIN.)
FEED, CM.	4626.00
FEED RATE, CM/MIN.	13.21
STEAM FEED RATIO, % ON FEED	38.66
VAPOR RESIDENCE TIME, SEC.	RV X 3.08
PRODUCT YIELD	
COKE, GMS.	2983.78
% ON FEED	64.50
LIQUID, GMS.	1329.59
% ON FEED	28.74
GAS, GMS.	312.63
% ON FEED	6.76
MATERIAL BALANCE, %	102.34
WATER BALANCE, %	95.58

GAS ANALYSES, MOL %/WT %

H ₂	56.38/10.71
CO	2.65/ 7.04
CO ₂	3.81/15.91
H ₂ S	0.69/ 2.23
NH ₃	2.26/ 3.64
CH ₄	28.88/43.87
C ₂ H ₆	3.21/ 9.13
C ₂ H ₄	0.83/ 2.21
C ₃ H ₈	0.56/ 2.35
C ₃ H ₆	0.73/ 2.91
MOLECULAR WEIGHT	10.53
HEATING VALUE, BTU/CM FT	602.03

DISTILLATION CUT(MICROLITER) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.53
400-700 DEG. F.	2.54
700-1000 DEG. F.	4.01
1000 DEG F.+	21.66
1000 DEG F.-	7.08
% 1000 F- IN FEED	4.00

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.25/12.07/ 0.32/ 0.48
ATOMIC H/C	1.70
400-700 DEG F.	
C/H/S/N, WT %	86.74/ 7.31/ 0.75/ 1.15
ATOMIC H/C	1.01
700-1000 DEG F.	
C/H/S/N, WT %	87.49/ 6.18/ 0.91/ 1.40
ATOMIC H/C	0.85
1000 DEG F.-	
C/H/S/N, WT %	87.05/ 7.02/ 0.81/ 1.24
ATOMIC H/C	0.97
BTU/LB	16507.48
LB S/HBTU	0.49
1000 DEG F.+	
C/H/S/N, WT %	87.84/ 5.30/ 1.23/ 1.76
ATOMIC H/C	0.72
BTU/LB	15543.93
LB S/HBTU	0.79

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	69.31	4.34	2.16	1.48	20.16
CM	3206.28	200.77	99.92	68.46	932.60
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	87.65	5.72	1.13	1.63	0.0
CM	1165.37	76.10	14.96	21.66	0.0
% ON FEED	36.35	37.90	14.98	31.64	0.0
GASEOUS PRODUCT					
WT %	53.88	25.43	2.09	0.0	0.0
CM	168.44	79.51	6.55	0.0	0.0
% ON FEED	5.25	39.60	6.55	0.0	0.0
COKE					
WT %	58.25	2.06	2.36	1.31	36.02
CM	1738.12	61.43	70.42	38.96	1074.84
% ON FEED	54.21	30.60	70.48	56.90	115.25
ELEMENTAL BALANCE, %	95.81	108.10	92.01	102.23	115.25

ATOMIC RATIO OF H/C: FEED-0.75 GAS-5.66 LIQUID-0.78 COKE-0.29

CSCU RUN-108

TEMPERATURE, DEG. F	RV + 67.00
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU YP- 432(840 F/100 MIN.)
FEED, GHS.	5597.00
FEED RATE, CM/MIN.	18.66
STEAM FEED RATIO, % ON FEED	46.40
VAPOR RESIDENCE TIME, SEC.	RV X 2.11
PRODUCT YIELD	
COKE, GHS.	3937.27
% ON FEED	70.35
LIQUID, GHS.	1512.29
% ON FEED	27.02
GAS, GHS.	147.45
% ON FEED	2.63
MATERIAL BALANCE, %	97.68
WATER BALANCE, %	95.46

GAS ANALYSES, MOL %/WT %

H ₂	59.51/11.45
CO	0.99/ 2.67
CO ₂	5.10/21.58
H ₂ S	1.26/ 4.11
NH ₃	0.0 / 0.0
CH ₄	27.33/42.07
C ₂ H ₆	3.66/10.58
C ₂ H ₄	0.97/ 2.62
C ₃ H ₈	0.69/ 2.91
C ₃ H ₆	0.50/ 2.01
MOLECULAR WEIGHT	10.39
HEATING VALUE, BTU/CU FT	610.98

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.32
400-700 DEG. F.	1.96
700-1000 DEG. F.	6.20
1000 DEG F.+	18.54
1000 DEG F.-	8.48
% 1000 F- IN FEED	10.80

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	87.80/10.80/ 0.03/ 0.33
ATOMIC H/C	1.48
400-700 DEG F.	
C/H/S/N, WT %	90.51/ 6.94/ 0.06/ 0.62
ATOMIC H/C	0.92
700-1000 DEG F.	
C/H/S/N, WT %	90.68/ 6.04/ 0.13/ 1.11
ATOMIC H/C	0.80
1000 DEG F.-	
C/H/S/N, WT %	90.53/ 6.43/ 0.11/ 0.97
ATOMIC H/C	0.85
BTU/LB	16739.43
LB S/MBTU	0.07
1000 DEG F.+	
C/H/S/N, WT %	90.43/ 5.26/ 0.04/ 1.26
ATOMIC H/C	0.70
BTU/LB	15947.10
LB S/MBTU	0.03

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	74.72	4.17	0.66	1.16	17.89
GM	4182.08	233.39	36.72	64.93	1001.30
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	90.46	5.62	0.06	1.17	0.0
GM	1368.09	85.06	0.94	17.68	0.0
% ON FEED	32.71	36.45	2.56	27.23	0.0
GASEOUS PRODUCT					
WT %	53.40	25.52	3.87	0.0	0.0
GM	78.73	37.62	5.70	0.0	0.0
% ON FEED	1.88	16.12	15.52	0.0	0.0
COKE					
WT %	69.30	2.59	0.32	0.85	27.22
GM	2728.46	102.00	12.75	33.47	1071.75
% ON FEED	65.24	43.70	34.73	51.55	107.04
ELEMENTAL BALANCE, %	99.84	96.27	52.81	78.78	107.04

ATOMIC RATIO OF H/C: FEED-0.67 GAS-5.73 LIQUID-0.75 COKE-0.49

CSCU RUN-109

TEMPERATURE, DEG. F	RV + 158.00
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU YF- 433(840 F/100 MIN.)
FEED, GHS.	4196.00
FEED RATE, GH/MIN.	15.40
STEAM FEED RATIO, % ON FEED	27.29
VAPOR RESIDENCE TIME, SEC.	RV X 3.25
PRODUCT YIELD	
COKE, GHS.	2671.22
% ON FEED	63.66
LIQUID, GHS.	1394.48
% ON FEED	33.23
GAS, GHS.	130.30
% ON FEED	3.11
MATERIAL BALANCE, %	100.33
WATER BALANCE, %	95.25

GAS ANALYSES, MOL %/WT %

H ₂	58.04/ 9.73
CO	1.80/ 4.22
CO ₂	10.89/40.19
H ₂ S	0.75/ 2.15
NH ₃	0.0 / 0.0
CH ₄	24.59/32.99
C ₂ H ₆	2.34/ 5.88
C ₂ H ₄	0.68/ 1.60
C ₃ H ₈	0.36/ 1.33
C ₃ H ₆	0.54/ 1.90
MOLECULAR WEIGHT	11.93
HEATING VALUE, BTU/CU FT	574.97

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.30
400-700 DEG. F.	2.74
700-1000 DEG. F.	6.87
1000 DEG F.+	23.33
1000 DEG F.-	9.91
% 1000 F- IN FEED	13.90

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	88.21/ 9.08/ 0.06/ 0.1
ATOMIC H/C	1.24
400-700 DEG F.	
C/H/S/N, WT %	89.63/ 6.43/ 0.08/ 0.05
ATOMIC H/C	0.91
700-1000 DEG F.	
C/H/S/N, WT %	90.34/ 6.03/ 0.13/ 0.05
ATOMIC H/C	0.80
1000 DEG F.-	
C/H/S/N, WT %	90.08/ 6.34/ 0.11/ 0.78
ATOMIC H/C	0.85
BTU/LB	16577.24
LB S/MBTU	0.07
1000 DEG F.+	
C/H/S/N, WT %	90.29/ 5.17/ 0.19/ 1.1
ATOMIC H/C	0.69
BTU/LB	15682.62
LB S/MBTU	0.12

ELEMENTAL BALANCE

	C	H*	S	N	ASH
FEED					
WT %	76.11	4.40	0.57	1.15	15.61
GH	3193.58	184.62	23.92	48.25	655.00
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	90.23	5.52	0.17	1.17	0.0
GH	1258.22	77.01	2.36	16.36	0.0
% ON FEED	39.40	41.71	9.87	33.91	0.0
GASEOUS PRODUCT					
WT %	46.31	20.03	2.03	0.0	0.0
GH	60.34	26.09	2.64	0.0	0.0
% ON FEED	1.89	14.13	11.04	0.0	0.0
COKE					
WT %	70.47	2.64	0.55	0.77	25.85
GH	1882.32	70.42	14.67	20.54	690.60
% ON FEED	58.94	38.14	61.34	42.57	105.44
ELEMENTAL BALANCE, %					
	100.23	93.99	82.23	76.48	105.44

ATOMIC RATIO OF H/C: FEED-0.69 GAS-5.19 LIQUID-0.73 COKE-0.52

CSCU KUN-110

TEMPERATURE, DEG. F	RV + 73.00
PRESSURE, PSIG	9.00
FEED SOURCE	RCLU YP- 432(840 F/100 MIN.) AND 433
FEED, CH.	3953.00
FEED RATE, CH/MIN.	13.18
STEAM FEED RATIO, % ON FEED	32.93
VAPOR RESIDENCE TIME, SEC.	RV X 3.26
PRODUCT YIELD	
COKE, CHS.	2843.93
% ON FEED	71.94
LIQUID, CHS.	1039.35
% ON FEED	26.30
GAS, CHS.	69.52
% ON FEED	1.76
MATERIAL BALANCE, %	99.41
WATER BALANCE, %	96.06

GAS ANALYSES, MOL %/WT %

H ₂	75.38/22.11
CO	1.02/ 4.17
CO ₂	2.78/17.95
H ₂ S	0.26/ 1.27
NH ₃	1.35/ 3.37
CH ₄	16.69/39.17
C ₂ H ₆	1.69/ 7.42
C ₂ H ₄	0.35/ 1.43
C ₃ H ₈	0.26/ 1.70
C ₃ H ₆	0.23/ 1.40
MOLECULAR WEIGHT	6.82
HEATING VALUE, BTU/CU FT	470.44

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

G4-400 DEG. F.	0.21
400-700 DEG. F.	3.10
700-1000 DEG. F.	6.96
1000 DEG F.+	16.01
1000 DEG F.-	10.26
% 1000 F- IN FEED	10.20

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	88.16/ 9.11/ 0.05/ 0.49
ATOMIC H/C	1.24
400-700 DEG F.	
C/H/S/N, WT %	89.46/ 6.98/ 0.07/ 0.64
ATOMIC H/C	0.94
700-1000 DEG F.	
C/H/S/N, WT %	90.07/ 6.15/ 0.14/ 0.93
ATOMIC H/C	0.82
1000 DEG F.-	
C/H/S/N, WT %	89.85/ 6.46/ 0.12/ 0.83
ATOMIC H/C	0.86
BTU/LB	16611.33
LB S/MBTU	0.07
1000 DEG F.+	
C/H/S/N, WT %	89.73/ 5.34/ 0.19/ 1.28
ATOMIC H/C	0.71
BTU/LB	15875.05
LB S/MBTU	0.12

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	75.83	4.42	0.60	1.14	16.00
CH	2997.56	174.72	23.72	45.86	632.48
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	89.71	5.77	0.16	1.10	0.0
CH	932.63	68.03	1.69	11.46	0.0
% ON FEED	31.11	34.36	7.14	25.44	0.0
GASEOUS PRODUCT					
WT %	45.81	36.77	1.20	0.0	0.0
CH	31.85	24.17	0.83	0.0	0.0
% ON FEED	1.06	13.84	3.51	0.0	0.0
COKE					
WT %	73.07	2.92	0.52	1.25	21.71
CH	2078.15	83.13	14.84	35.63	617.33
% ON FEED	69.33	47.58	62.39	79.05	97.61
ELEMENTAL BALANCE, %	101.50	95.77	73.24	100.78	97.61

ATOMIC RATIO OF H/C: FEED=0.70 GAS=9.11 LIQUID=0.77 COKE=0.53

CSCU RUN-115

TEMPERATURE, DEG. F	RV + 58.00
PRESSURE, PSIG	9.50
FEED SOURCE	RCLU YP- 520(840 F/100 MIN.)
FEED, GH.	4719.00
FEED RATE, GH/MIN.	17.48
STEAM FEED RATIO, % ON FEED	24.04
VAPOR RESIDENCE TIME, SEC.	RV X 3.39
PRODUCT YIELD	
COKE, GMS.	3223.03
% ON FEED	68.30
LIQUID, GMS.	1366.10
% ON FEED	28.95
GAS, GMS.	129.86
% ON FEED	2.75
MATERIAL BALANCE, %	98.46
WATER BALANCE, %	94.13

GAS ANALYSES, MOL %/WT %

N2	67.09/16.66
CO	0.48/ 1.67
CO2	1.05/ 5.75
H2S	0.69/ 2.93
NH3	0.0 / 0.0
CH4	24.94/49.54
C2H6	3.48/12.97
C2H4	0.84/ 2.92
C3H8	0.72/ 3.91
C3H6	0.70/ 3.65
MOLECULAR WEIGHT	8.06
HEATING VALUE, BTU/CU FT	581.33

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.76
400-700 DEG. F.	1.66
700-1000 DEG. F.	5.05
1000 DEG F.+	21.48
1000 DEG F.-	7.47
% 1000 F- IN FEED	8.60

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	86.96/10.79/ 0.16/ 0.43
ATOMIC H/C	1.49
400-700 DEG F.	
C/H/S/N, WT %	88.72/ 7.14/ 0.30/ 0.82
ATOMIC H/C	0.97
700-1000 DEG F.	
C/H/S/N, WT %	89.89/ 6.25/ 0.62/ 1.12
ATOMIC H/C	0.83
1000 DEG F.-	
C/H/S/N, WT %	89.33/ 6.91/ 0.50/ 0.98
ATOMIC H/C	0.93
BTU/LB	16855.33
LB S/MBTU	0.30
1000 DEG F.+	
C/H/S/N, WT %	88.47/ 5.36/ 0.80/ 1.35
ATOMIC H/C	0.73
BTU/LB	15692.78
LB S/MBTU	0.51

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	70.54	4.07	2.36	1.31	20.18
GH	3328.78	192.06	111.37	61.82	952.29
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	88.68	5.76	0.72	1.26	0.0
GH	1211.47	78.66	9.89	17.17	0.0
% ON FEED	36.39	40.96	8.88	27.77	0.0
CASEOUS PRODUCT					
WT %	58.65	33.46	2.75	0.0	0.0
GH	76.17	43.45	3.58	0.0	0.0
% ON FEED	2.29	22.62	3.21	0.0	0.0
COKE					
WT %	62.39	2.03	2.17	1.28	27.07
GH	2010.96	65.46	70.03	41.10	872.59
% ON FEED	60.41	34.08	62.88	66.49	91.63
ELEMENTAL BALANCE, %	99.09	97.66	74.97	94.26	91.63

ATOMIC RATIO OF H/C: FEED-0.69 GAS-6.85 LIQUID-0.78 COKE-0.41

CSCU RUN-116

TEMPERATURE, DEG. F	RV + 131.00
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU YP- 521(840 F/100 MIN.)
FEED, CM.	3383.00
FEED RATE, CM/MIN.	11.87
STEAM FEED RATIO, % ON FEED	44.36
VAPOR RESIDENCE TIME, SEC.	RV X 2.60
PRODUCT YIELD	
COKE, GHS.	2072.31
% ON FEED	61.26
LIQUID, GHS.	1196.46
% ON FEED	35.37
GAS, GHS.	114.23
% ON FEED	3.38
MATERIAL BALANCE, %	96.51
WATER BALANCE, %	92.03

GAS ANALYSES, MOL %/WT %

H2	79.15/25.85
CO	0.73/ 3.32
CO2	1.87/13.46
H2S	0.55/ 3.08
NH3	0.0 / 0.0
CH4	14.75/38.54
C2H6	1.68/ 8.23
C2H4	0.55/ 2.52
C3H8	0.31/ 2.25
C3H6	0.40/ 2.75
MOLECULAR WEIGHT	6.12
HEATING VALUE, BTU/CU FT	467.67

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

C4-400 DEG. F.	0.28
400-700 DEG. F.	1.65
700-1000 DEG. F.	7.94
1000 DEG F.+	25.50
1000 DEG F.-	9.87
% 1000 F- IN FEED	7.50

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	87.13/10.01/ 0.22/ 0.58
ATOMIC H/C	1.38
400-700 DEG F.	
C/H/S/N, WT %	88.79/ 7.22/ 0.31/ 0.83
ATOMIC H/C	0.98
700-1000 DEG F.	
C/H/S/N, WT %	89.74/ 6.13/ 0.64/ 1.11
ATOMIC H/C	0.82
1000 DEG F.-	
C/H/S/N, WT %	89.51/ 6.42/ 0.57/ 1.05
ATOMIC H/C	0.86
BTU/LB	16575.49
LB S/MBTU	0.35
1000 DEG F.+	
C/H/S/N, WT %	91.86/ 5.25/ 0.82/ 1.69
ATOMIC H/C	0.69
BTU/LB	16348.09
LB S/MBTU	0.50

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	72.57	4.39	2.81	1.39	17.68
CM	2455.04	148.51	95.06	47.02	598.11
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	91.22	5.58	0.75	1.51	0.0
CM	1091.36	66.72	9.03	18.09	0.0
% ON FEED	44.45	44.93	9.50	38.47	0.0
CASEOUS PRODUCT					
WT %	46.94	38.47	2.90	0.0	0.0
CM	53.62	43.95	3.31	0.0	0.0
% ON FEED	2.18	29.59	3.48	0.0	0.0
COKE					
WT %	65.05	2.04	4.48	0.75	25.37
CM	1348.13	42.22	92.88	15.48	525.76
% ON FEED	54.91	28.43	97.70	32.92	87.90
ELEMENTAL BALANCE, %	101.55	102.94	110.68	71.39	87.90

ATOMIC RATIO OF H/C: FEED-0.73 GAS-9.83 LIQUID-0.73 COKE-0.35

CSCU RUN-117

TEMPERATURE, DEG. F	RV + 134.00
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU YP- 521(840 F/100 MIN.)
FEED, GM.	3825.00
FEED RATE, GM/MIN.	12.75
STEAM FEED RATIO, % ON FEED	34.18
VAPOR RESIDENCE TIME, SEC.	RV X 3.03
PRODUCT YIELD	
COKE, GMS.	2322.46
% ON FEED	60.72
LIQUID, GMS.	1344.27
% ON FEED	35.14
GAS, GMS.	158.27
% ON FEED	4.14
MATERIAL BALANCE, %	99.75
WATER BALANCE, %	97.55

GAS ANALYSIS, MOL %/WT %

H2	71.83/17.81
CO	1.00/ 3.46
CO2	2.57/14.04
H2S	2.25/ 9.50
NH3	0.02/ 0.04
CH4	17.81/35.33
C2H6	1.89/ 7.03
C2H4	0.58/ 2.01
C3H8	0.47/ 2.58
C3H6	1.58/ 8.21
MOLECULAR WEIGHT	8.07
HEATING VALUE, BTU/CU FT	524.60

DISTILLATION CUT(MICROLUBE) OF LIQUID, % ON FEED

G4-400 DEG. F.	0.83
400-700 DEG. F.	1.66
700-1000 DEG. F.	8.49
1000 DEG F.+	24.16
1000 DEG F.-	10.98
% 1000 F- IN FEED	9.40

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	84.75/14.48/ 0.05/ 0.12
ATOMIC H/C	2.05
400-700 DEG F.	
C/H/S/N, WT %	88.84/ 7.23/ 0.31/ 0.75
ATOMIC H/C	0.98
700-1000 DEG F.	
C/H/S/N, WT %	89.53/ 6.12/ 0.55/ 1.22
ATOMIC H/C	0.82
1000 DEG F.-	
C/H/S/N, WT %	89.06/ 6.92/ 0.48/ 1.07
ATOMIC H/C	0.93
BTU/LB	16808.48
LB S/MBTU	0.28
1000 DEG F.+	
C/H/S/N, WT %	89.67/ 5.14/ 0.83/ 1.70
ATOMIC H/C	0.85
BTU/LB	15823.99
LB S/MBTU	0.53

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	72.57	4.43	2.60	1.22	17.67
GM	2775.80	169.45	99.45	46.66	675.88
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	89.48	5.70	0.72	1.50	0.0
GM	1202.86	76.61	9.69	20.23	0.0
% ON FEED	43.33	45.21	9.74	43.35	0.0
GASEOUS PRODUCT					
WT %	48.30	30.54	8.94	0.0	0.0
GM	76.45	48.33	14.15	0.0	0.0
% ON FEED	2.75	28.52	14.22	0.0	0.0
COKE					
WT %	66.97	2.16	3.70	1.23	25.26
GM	1555.42	50.13	85.93	28.64	586.57
% ON FEED	56.03	29.58	86.41	61.38	86.79
ELEMENTAL BALANCE, %	102.12	103.32	110.37	104.84	86.79

ATOMIC RATIO OF H/C: FEED-0.73 GAS-7.59 LIQUID-0.76 COKE-0.36

CSCU RUN-118

TEMPERATURE, DEG. F	RV + 49.00
PRESSURE, PSIG	10.00
FEED SOURCE	RCLU TP- 520(840 F/100 MIN.)
FEED, CH.	4667.00
FEED RATE, CH/MIN.	17.61
STEAM FEED RATIO, % ON FEED	33.32
VAPOR RESIDENCE TIME, SEC.	RV X 2.83
PRODUCT YIELD	
COKE, CHS.	3325.03
% ON FEED	71.25
LIQUID, CHS.	1223.60
% ON FEED	26.22
GAS, CHS.	118.38
% ON FEED	2.54
MATERIAL BALANCE, %	99.25
WATER BALANCE, %	90.89

GAS ANALYSES, MOL %/WT %

H ₂	70.92/18.42
CO	0.60/ 2.18
CO ₂	1.06/ 6.08
H ₂ S	0.58/ 2.55
NH ₃	0.0 / 0.0
CH ₄	19.80/41.13
C ₂ H ₆	4.84/18.84
C ₂ H ₄	0.77/ 2.79
C ₃ H ₈	0.83/ 4.75
C ₃ H ₆	0.60/ 3.26
MOLECULAR WEIGHT	7.70
HEATING VALUE, BTU/CU FT	564.86

DISTILLATION CUT(MICROLBS) OF LIQUID, % ON FEED

50-500 DEG. F.	0.28
100-700 DEG. F.	1.43
200-1000 DEG. F.	5.00
1000 DEG F.+	19.51
1000 DEG F.-	6.71
% 1000 F.- IN FEED	7.40

ANALYSES OF LIQUID PRODUCT

C4-400 DEG F.	
C/H/S/N, WT %	85.51/12.94/ 0.10/ 0.24
ATOMIC H/C	1.82
400-700 DEG F.	
C/H/S/N, WT %	88.56/ 7.45/ 0.79/ 0.73
ATOMIC H/C	1.01
700-1000 DEG F.	
C/H/S/N, WT %	89.50/ 6.04/ 0.53/ 1.09
ATOMIC H/C	0.81
1000 DEG F.-	
C/H/S/N, WT %	89.13/ 6.63/ 0.46/ 0.98
ATOMIC H/C	0.89
BTU/LB	16619.43
LB S/HBTU	0.28
1000 DEG F.+	
C/H/S/N, WT %	87.87/ 5.47/ 0.90/ 1.64
ATOMIC H/C	0.75
BTU/LB	15671.08
LB S/HRTU	0.57

ELEMENTAL BALANCE

	C	H	S	N	ASH
FEED					
WT %	72.74	4.39	2.95	1.30	20.18
CH	3394.78	204.88	137.68	60.67	941.80
% ON FEED	100.00	100.00	100.00	100.00	100.00
LIQUID PRODUCT					
WT %	88.20	5.76	0.79	1.47	0.0
CH	1079.15	70.52	9.65	17.99	0.0
% ON FEED	31.79	34.42	7.01	29.66	0.0
GASEOUS PRODUCT					
WT %	57.58	34.34	2.40	0.0	0.0
CH	68.17	40.66	2.85	0.0	0.0
% ON FEED	2.01	19.84	2.07	0.0	0.0
COKE					
WT %	70.79	2.67	3.04	0.55	30.04
CH	2353.92	88.89	101.15	18.39	998.68
% ON FEED	69.34	43.38	73.47	30.31	106.04
ELEMENTAL BALANCE, %	103.14	97.65	82.54	59.97	106.04

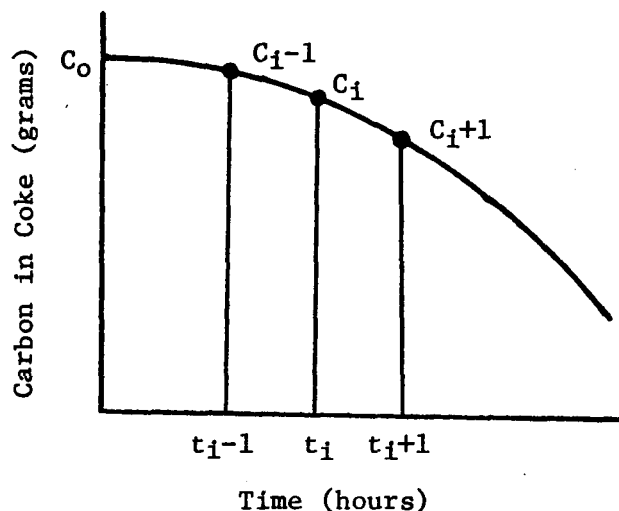
ATOMIC RATIO OF H/C: FEED-0.72 GAS-7.16 LIQUID-0.78 COKE-0.50

APPENDIX G

COKE GASIFICATION DATA FROM THE MINIGASIFIER

This appendix contains data from coke gasification runs made in the minigasifier discussed in Section 6 of this report. The experimental conditions and the measured gasification rates are presented for 22 runs. Six different coke feed samples were used: EDS Illinois and Wyodak cokes from the IKG unit, EDS Illinois and Wyodak cokes from the LSCU unit, and two petroleum coke samples.

Two different methods were used to represent the coke gasification rates, as will be described 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:

$$\left(\frac{C_0 - C_i}{t_i} \right) \frac{1}{C_0}$$

RUN FFT57

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 66.90
 % ASH IN COKE 22.20
 INLET GAS.....STEAM
 COKE SAMPLE..... LSCU WYODAK COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
16.00	25.32	5.48	0.14	0.02	0.07	2.42	114.02	94.95
30.00	42.90	4.89	0.13	0.02	0.06	2.48	128.55	85.80
44.00	59.57	3.26	0.09	0.01	0.04	2.53	140.56	81.23
59.00	70.12	2.27	0.06	0.01	0.03	2.57	132.33	71.31
73.00	78.72	1.73	0.05	0.01	0.02	2.58	144.86	64.71
88.00	84.92	1.30	0.03	0.01	0.02	2.59	153.95	57.90
102.00	89.97	1.07	0.03	0.01	0.01	2.57	190.13	52.93
116.00	93.82	0.76	0.02	0.00	0.01	2.59	217.39	48.53
131.00	96.42	0.50	0.01	0.00	0.01	2.60	247.98	44.16
145.00	98.13	0.35	0.01	0.00	0.00	2.60	312.30	40.61
159.00	99.15	0.22	0.01	0.00	0.00	2.62	375.86	37.41
174.00	99.66	0.15	0.00	0.0	0.00	2.62	453.42	34.37
188.00	99.89	0.11	0.00	0.0	0.00	2.61	672.17	31.88
202.00	100.00	0.10	0.00	0.0	0.00	2.59	*****	29.70

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
25.32	61.04	8.30	30.42	0.24	61.04	8.30	30.42	0.24
42.90	61.04	8.30	30.42	0.24	61.04	8.30	30.42	0.24
59.57	60.46	8.92	30.48	0.14	60.88	8.46	30.44	0.22
70.12	59.41	10.32	30.27	0.0	60.67	8.73	30.41	0.19
78.72	58.98	11.12	29.90	0.0	60.50	8.98	30.36	0.17
84.92	58.71	11.13	30.16	0.0	60.37	9.13	30.35	0.15
89.97	58.42	10.98	30.59	0.0	60.27	9.23	30.36	0.15
93.82	58.67	10.82	30.50	0.0	60.21	9.29	30.36	0.14
96.42	58.95	10.71	30.34	0.0	60.17	9.33	30.36	0.14
98.13	59.54	10.51	29.95	0.0	60.16	9.35	30.36	0.13
99.15	62.19	9.54	28.27	0.0	60.18	9.35	30.33	0.13
99.66	68.57	5.34	26.09	0.0	60.24	9.32	30.31	0.13
99.89	76.94	0.0	23.06	0.0	60.30	9.29	30.28	0.13
100.00	84.63	0.0	15.37	0.0	60.37	9.26	30.24	0.12

RUN FFT58

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.63
 COKE CHARGE (CM) 1.10
 % CARBON IN COKE 85.90
 % ASH IN COKE 0.29
 INLET GAS.....STEAM
 COKE SAMPLE.....PETROLEUM COKE I.

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
16.00	17.54	3.91	0.10	0.06	0.03	2.42	77.27	65.77
30.00	31.93	3.45	0.09	0.06	0.02	2.48	87.45	63.85
45.00	46.28	3.19	0.08	0.06	0.02	2.50	106.18	61.70
59.00	59.51	2.90	0.07	0.06	0.02	2.51	127.98	60.51
74.00	71.24	2.30	0.06	0.05	0.01	2.54	146.71	57.76
88.00	79.98	1.79	0.05	0.04	0.01	2.56	163.57	54.53
103.00	86.99	1.44	0.04	0.03	0.01	2.58	189.84	50.68
117.00	91.97	1.10	0.03	0.02	0.01	2.60	224.92	47.16
132.00	95.67	0.80	0.02	0.01	0.01	2.60	286.59	43.49
146.00	98.00	0.52	0.01	0.01	0.00	2.60	372.23	40.27
160.00	99.15	0.24	0.01	0.00	0.00	2.62	394.14	37.18
175.00	99.60	0.14	0.00	0.00	0.00	2.63	293.52	34.15
189.00	99.73	0.11	0.00	0.0	0.00	2.59	199.78	31.66

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS				AVERAGE			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
17.54	52.98	29.15	16.54	1.33	52.98	29.15	16.54	1.33
31.93	51.04	33.63	13.97	1.36	52.13	31.12	15.41	1.34
46.28	49.72	36.64	12.28	1.36	51.40	32.78	14.47	1.35
59.51	49.38	38.14	11.18	1.29	50.97	33.93	13.76	1.34
71.24	49.24	38.87	10.69	1.21	50.69	34.72	13.27	1.31
79.98	49.13	38.64	11.16	1.08	50.53	35.14	13.05	1.29
86.99	50.04	36.75	12.33	0.88	50.49	35.27	12.99	1.26
91.97	51.40	34.06	14.10	0.45	50.54	35.20	13.05	1.21
95.67	52.56	31.48	15.96	0.0	50.62	35.05	13.17	1.16
98.00	54.25	28.34	17.41	0.0	50.71	34.88	13.28	1.13
99.15	57.35	24.07	18.58	0.0	50.80	34.73	13.35	1.12
99.60	64.04	16.95	19.02	0.0	50.88	34.62	13.38	1.11
99.73	79.46	6.44	14.10	0.0	50.97	34.54	13.38	1.11

RUN RDW473

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CH/SEC) 20.00
 COKE CHARGE (GN) 1.00
 % CARBON IN COKE 85.70
 % ASH IN COKE 0.30
 INLET GAS.....STEAM
 COKE SAMPLE.....PETROLEUM COKE I.

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
16.00	29.62	4.76	0.13	0.09	0.02	2.63	133.72	111.06
32.00	50.19	3.	0.08	0.06	0.02	2.73	122.19	94.11
47.00	61.33	2.7	0.06	0.04	0.01	2.78	105.49	78.30
61.00	69.97	1.46	0.04	0.03	0.01	2.81	103.64	68.82
76.00	76.28	1.17	0.03	0.02	0.01	2.83	98.91	60.22
90.00	81.34	0.95	0.03	0.02	0.01	2.85	102.28	54.23
105.00	85.46	0.77	0.02	0.01	0.01	2.86	103.19	48.83
119.00	88.62	0.50	0.01	0.01	0.00	2.90	84.46	44.68
134.00	90.04	0.39	0.01	0.01	0.00	2.91	70.27	40.31
148.00	91.98	0.27	0.01	0.00	0.00	2.92	66.14	37.29
163.00	92.55	0.11	0.00	0.00	0.00	2.97	27.69	34.07
177.00	92.98	0.13	0.00	0.00	0.00	2.93	31.34	31.52
191.00	93.57	0.13	0.00	0.00	0.00	2.86	39.43	29.39

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
29.62	52.39	36.33	9.85	1.43	52.39	36.33	9.85	1.43
50.19	52.06	37.20	9.47	1.27	52.26	36.68	9.70	1.36
61.33	51.70	37.60	9.66	1.04	52.16	36.85	9.69	1.30
69.97	51.44	37.14	10.62	0.80	52.07	36.89	9.80	1.24
76.28	51.40	36.33	11.62	0.65	52.01	36.84	9.95	1.19
81.34	51.67	35.12	12.67	0.54	51.99	36.73	10.12	1.15
85.46	52.23	33.40	13.94	0.43	52.00	36.57	10.31	1.12
88.62	52.97	31.47	15.36	0.20	52.04	36.39	10.49	1.08
90.04	53.51	29.77	16.73	0.0	52.06	36.28	10.59	1.07
91.98	53.63	28.82	17.55	0.0	52.10	36.12	10.74	1.04
92.55	53.96	28.70	17.33	0.0	52.11	36.07	10.78	1.04
92.98	54.26	29.49	16.25	0.0	52.12	36.04	10.81	1.03
93.57	54.99	30.53	14.48	0.0	52.14	36.00	10.84	1.03

RUN RDW472

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 16.43
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 62.20
 % ASH IN COKE 35.40
 INLET GAS.....STEAM
 COKE SAMPLE.....IKG ILLININOIS COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
14.00	60.14	8.57	0.23	0.14	0.05	2.47	408.19	257.73
28.00	75.93	3.10	0.09	0.06	0.02	2.79	257.31	162.71
42.00	89.04	1.68	0.05	0.03	0.01	2.84	353.22	127.20
56.00	94.00	0.70	0.02	0.01	0.00	2.89	274.78	100.72
70.00	96.73	0.39	0.01	0.01	0.00	2.90	262.67	82.91
86.00	98.20	0.24	0.01	0.00	0.00	2.90	250.84	68.51
100.00	99.01	0.17	0.01	0.00	0.00	2.90	280.80	59.41
114.00	99.49	0.12	0.00	0.00	0.00	2.91	332.70	52.37
128.00	99.80	0.10	0.00	0.00	0.00	2.93	524.92	46.78
143.00	100.00	0.13	0.00	0.00	0.00	2.87	*****	41.96

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS		GAS ANALYSIS		H2	AVERAGE CO	GAS ANALYSIS		CH4
	H2	CO	CO2	CH4			CO2	CH4	
60.14	54.86	32.62	11.03	1.49	54.86	32.62	11.03	1.49	
75.93	53.90	33.86	10.88	1.35	54.68	32.85	11.00	1.47	
89.04	52.82	35.06	10.90	1.22	54.43	33.15	10.99	1.44	
94.00	52.32	35.66	10.92	1.08	54.33	33.26	10.98	1.42	
96.73	53.86	35.03	10.47	0.64	54.32	33.31	10.97	1.40	
98.20	56.57	33.68	9.75	0.0	54.35	33.32	10.95	1.38	
99.01	59.74	31.41	8.85	0.0	54.40	33.30	10.93	1.37	
99.49	63.62	28.47	7.91	0.0	54.45	33.27	10.92	1.36	
99.80	67.24	25.60	7.16	0.0	54.50	33.24	10.90	1.35	
100.00	71.13	22.49	6.38	0.0	54.55	33.21	10.89	1.35	

RUN RDW475

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.66
 COKE CHARGE (GM) 1.09
 % CARBON IN COKE 66.90
 % ASH IN COKE 22.20
 INLET GAS.....STEAM
 COKE SAMPLE.....LSCU WYODAK COKE

. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % C/HR.	
			H2	CO	CO2	H2O	INSTANTANEOUS	AVERAGE
5.00	20.31	19.23	0.51	0.06	0.21	2.12	278.61	243.76
20.00	70.38	10.19	0.27	0.03	0.12	2.37	450.99	211.14
41.00	93.79	2.84	0.08	0.01	0.02	2.67	704.72	137.26
55.00	98.60	0.88	0.02	0.00	0.01	2.75	947.53	107.56
69.00	100.00	0.32	0.01	0.00	0.00	2.65	*****	86.96

I. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
20.31	65.50	7.06	26.96	0.49	65.50	7.06	26.96	0.49
70.38	64.90	7.44	27.24	0.42	65.08	7.32	27.16	0.44
93.79	63.63	8.53	27.60	0.23	64.73	7.62	27.26	0.39
98.60	62.88	9.89	27.23	0.0	64.64	7.72	27.26	0.37
100.00	67.47	9.16	23.37	0.0	64.69	7.74	27.20	0.37

RUN RDW477

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 16.38
 COKE CHARGE (GM) 0.76
 % CARBON IN COKE 66.90
 % ASH IN COKE 22.20
 INLET GAS.....STEAM
 COKE SAMPLE.....LSCU WYODAK COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				H2O	RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2				
15.00	52.59	11.00	0.28	0.04	0.12	2.24	332.90	210.36	
30.00	78.91	5.49	0.14	0.02	0.06	2.43	392.72	157.83	
44.00	92.99	2.55	0.07	0.01	0.03	2.55	584.58	126.80	
59.00	98.41	1.14	0.03	0.00	0.01	2.61	895.27	100.08	
73.00	100.00	0.61	0.02	0.00	0.00	2.53	*****	82.19	

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
52.59	64.30	8.22	27.17	0.31	64.30	8.22	27.17	0.31
78.91	63.38	8.89	27.44	0.30	64.00	8.44	27.26	0.30
92.99	62.40	9.81	27.60	0.18	63.77	8.64	27.31	0.29
98.41	64.92	10.13	24.95	0.0	63.83	8.72	27.17	0.27
100.00	75.19	7.72	17.09	0.0	64.10	8.70	26.94	0.26

RUN RDW478

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 20.58
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 62.10
 % ASH IN COKE 35.30
 INLET GAS.....STEAM
 COKE SAMPLE.....1KG ILLINOIS COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % C/HR.	
			H2	CO	CO2	H2O	INSTANTANEOUS	AVERAGE
16.00	39.32	4.85	0.12	0.10	0.02	2.45	215.80	147.44
31.00	67.94	3.69	0.09	0.08	0.02	2.46	301.14	131.49
45.00	86.29	1.80	0.05	0.04	0.01	2.53	394.65	115.05
61.00	94.18	0.47	0.01	0.01	0.00	2.59	333.36	92.63
75.00	96.33	0.19	0.00	0.00	0.00	2.61	158.42	77.06
91.00	96.97	0.10	0.00	0.0	0.00	2.59	79.37	63.94

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS		GAS ANALYSIS		H2	AVERAGE		GAS ANALYSIS		CH4
	H2	CO	CO2	CH4		CO	CO2			
39.32	50.01	38.31	10.39	1.29	50.01	38.31	10.39	1.29		
67.94	49.95	39.23	9.59	1.23	49.98	38.70	10.05	1.27		
86.29	49.21	40.62	9.08	1.09	49.82	39.10	9.85	1.23		
94.18	48.04	42.17	9.09	0.70	49.68	39.35	9.79	1.19		
96.33	49.37	41.49	9.14	0.0	49.67	39.40	9.77	1.16		
96.97	59.67	28.28	12.05	0.0	49.75	39.31	9.79	1.15		

RUN RDW479

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 16.38
 COKE CHARGE (GM) 0.87
 % CARBON IN COKE 85.90
 % ASH IN COKE 0.29
 INLET GAS.....STEAM
 COKE SAMPLE.....PETROLEUM COKE I.

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
16.00	19.32	3.90	0.10	0.08	0.02	2.35	88.57	72.44
31.00	36.94	3.69	0.09	0.07	0.02	2.32	* 105.20	71.49
45.00	51.45	2.89	0.07	0.06	0.02	2.34	112.14	68.60
60.00	63.12	2.27	0.06	0.04	0.01	2.38	111.92	63.12
75.00	72.09	1.80	0.04	0.03	0.01	2.40	112.52	57.67
89.00	78.38	1.28	0.03	0.02	0.01	2.45	105.67	52.84
104.00	83.06	1.02	0.03	0.02	0.01	2.46	103.64	47.92
118.00	86.88	0.86	0.02	0.01	0.01	2.48	104.66	44.18
133.00	89.66	0.69	0.02	0.01	0.01	2.49	99.93	40.45
147.00	91.89	0.57	0.01	0.00	0.01	2.48	97.98	37.51
161.00	93.36	0.37	0.01	0.00	0.00	2.52	75.01	34.79
176.00	94.27	0.22	0.01	0.00	0.00	2.52	53.40	32.14
190.00	94.85	0.13	0.00	0.00	0.00	2.51	33.55	29.95
205.00	95.10	0.05	0.00	0.0	0.00	2.49	20.10	27.83

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS				AVERAGE			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
19.32	48.13	38.96	11.59	1.31	48.13	38.96	11.59	1.31
36.94	48.22	39.44	11.11	1.23	48.7	39.19	11.36	1.27
51.45	47.96	39.82	11.14	1.09	48.11	39.37	11.30	1.22
63.12	48.09	39.11	11.91	0.89	48.11	39.32	11.41	1.16
72.09	49.21	37.32	13.05	0.43	48.25	39.07	11.62	1.07
78.38	49.61	35.71	14.68	0.0	48.36	38.79	11.87	0.98
83.06	49.84	33.50	16.67	0.0	48.45	38.48	12.15	0.92
86.88	50.94	30.08	18.98	0.0	48.56	38.10	12.46	0.88
89.66	52.32	25.49	22.19	0.0	48.69	37.68	12.79	0.85
91.89	54.15	20.20	25.66	0.0	48.83	37.20	13.14	0.83
93.36	55.76	16.53	27.71	0.0	48.96	36.83	13.40	0.81
94.27	56.03	14.97	29.00	0.0	49.04	36.58	13.58	0.80
94.85	55.76	14.78	29.46	0.0	49.09	36.43	13.69	0.80
95.10	59.45	10.82	29.73	0.0	49.12	36.34	13.74	0.79

RUN RDW480

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 15.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 66.90
 % ASH IN COKE 22.20
 INLET GAS.....STEAM
 COKE SAMPLE..... LSCU WYODAK COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
19.00	22.75	6.35	0.15	0.01	0.08	2.27	91.22	71.83
33.00	38.87	5.24	0.13	0.01	0.07	2.33	96.17	70.68
48.00	50.99	3.97	0.10	0.01	0.05	2.37	96.60	63.73
62.00	61.82	3.26	0.08	0.01	0.04	2.38	104.01	59.83
77.00	70.07	2.36	0.06	0.01	0.03	2.43	98.56	54.60
91.00	76.14	1.86	0.05	0.01	0.02	2.43	99.91	50.20
105.00	81.19	1.40	0.04	0.01	0.02	2.46	97.28	46.40
120.00	84.93	1.10	0.03	0.01	0.01	2.48	94.37	42.46
134.00	88.06	0.90	0.02	0.00	0.01	2.48	94.12	39.44
149.00	90.31	0.65	0.02	0.00	0.01	2.51	85.23	36.37
163.00	92.08	0.54	0.01	0.00	0.01	2.50	85.65	33.90
178.00	93.58	0.50	0.01	0.00	0.01	2.47	92.96	31.54

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS		GAS ANALYSIS		AVERAGE	GAS ANALYSIS		CH4
	H2	CO	CO2	CH4		CO	CO2	
22.75	63.63	4.46	31.91	0.0	63.63	4.46	31.91	0.0
38.87	62.76	4.54	32.70	0.0	63.28	4.49	32.23	0.0
50.99	61.98	4.87	33.15	0.0	62.98	4.58	32.44	0.0
61.82	61.23	5.83	32.94	0.0	62.68	4.79	32.53	0.0
70.07	60.74	7.19	32.07	0.0	62.46	5.06	32.48	0.0
76.14	60.22	8.67	31.11	0.0	62.29	5.33	32.37	0.0
81.19	59.82	10.05	30.13	0.0	62.15	5.61	32.24	0.0
84.93	59.64	11.05	29.31	0.0	62.04	5.83	32.12	0.0
88.06	59.95	11.62	28.43	0.0	61.97	6.03	32.00	0.0
90.31	60.67	11.96	27.38	0.0	61.94	6.17	31.89	0.0
92.08	60.21	12.46	27.32	0.0	61.91	6.29	31.80	0.0
93.58	61.08	12.56	26.36	0.0	61.90	6.39	31.72	0.0

RUN FFT510

GASIFICATION RATES FROM MINICASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CH/SEC) 15.63
 COKE CHARGE (CH) 1.00
 % CARBON IN COKE 62.20
 % ASH IN COKE 35.40
 INLET GAS.....STEAM
 COKE SAMPLE.....IKG ILLINOIS COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAG
			H2	CO	CO2	H2O		
15.00	7.89	2.20	0.05	0.03	0.01	2.39	43.23	31.55
30.00	19.91	2.58	0.06	0.05	0.01	2.39	65.01	39.82
48.00	36.72	2.12	0.05	0.05	0.01	2.47	74.89	45.90
63.00	46.41	1.82	0.05	0.04	0.01	2.47	80.01	44.20
77.00	57.37	1.68	0.04	0.04	0.01	2.41	98.11	44.71
92.00	66.54	1.35	0.03	0.03	0.01	2.41	100.55	43.39
106.00	73.69	1.04	0.03	0.03	0.01	2.43	97.81	41.71
121.00	78.89	0.67	0.02	0.02	0.00	2.45	86.64	39.12
135.00	82.57	0.49	0.01	0.01	0.00	2.46	75.07	36.70
150.00	85.17	0.26	0.01	0.01	0.00	2.59	49.00	34.07
164.00	86.13	0.14	0.00	0.00	0.00	2.61	31.81	31.51
178.00	87.23	0.10	0.00	0.00	0.00	2.51	31.35	29.40
193.00	88.06	0.05	0.00	0.00	0.00	2.48	23.11	27.37
207.00	88.58	0.03	0.00	0.00	0.00	2.47	19.51	25.67

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE	GAS ANALYSIS			
	H2	CO	CO2	CH4		CO	CO2	CH4	
7.89	56.16	27.39	15.87	0.58	56.16	27.39	15.87	0.58	
19.91	50.60	37.00	11.31	1.09	52.96	32.91	13.25	0.87	
36.72	47.79	41.61	9.32	1.28	50.73	36.67	11.55	1.05	
46.41	46.32	43.43	8.92	1.33	49.87	37.99	11.04	1.10	
57.37	45.51	44.26	8.89	1.34	49.09	39.11	10.66	1.15	
66.54	45.19	44.79	8.82	1.20	48.59	39.84	10.42	1.15	
73.69	45.11	45.01	8.87	1.00	48.27	40.31	10.28	1.14	
78.89	44.54	45.81	9.12	0.53	48.04	40.65	10.21	1.10	
82.57	43.81	46.79	9.40	0.0	47.86	40.91	10.17	1.06	
85.17	43.88	46.68	9.44	0.0	47.75	41.07	10.15	1.03	
86.13	41.98	48.13	9.89	0.0	47.69	41.14	10.15	1.02	
87.23	37.79	51.74	10.47	0.0	47.59	41.26	10.15	1.01	
88.06	32.70	57.35	9.95	0.0	47.48	41.37	10.15	1.00	
88.58	27.20	64.80	8.00	0.0	47.39	41.47	10.14	0.99	

RUN FFT513

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 15.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 85.40
 % ASH IN COKE 0.15
 INLET GAS.....STEAM
 COKE SAMPLE.....PETROLEUM COKE I

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % C/HR.	
			H2	CO	CO2	H2O	INSTANTANEOUS	AVERAGE
58.00	46.57	3.27	0.08	0.06	0.02	2.34	91.57	48.18
72.00	58.16	2.73	0.07	0.06	0.02	2.37	107.45	48.47
87.00	68.22	5.31	0.10	0.09	0.03	1.87	258.45	47.05
91.00	76.49	4.51	0.09	0.07	0.02	1.88	324.21	50.43
106.00	83.60	1.67	0.04	0.03	0.01	2.42	159.31	47.32
120.00	89.16	1.32	0.03	0.02	0.01	2.44	187.71	44.58
135.00	93.38	1.01	0.03	0.02	0.01	2.46	226.50	41.50
149.00	96.44	0.73	0.02	0.01	0.01	2.46	304.29	38.84
163.00	98.43	0.43	0.01	0.01	0.00	2.49	402.00	36.23
178.00	99.46	0.15	0.00	0.00	0.00	2.50	548.23	33.53
192.00	99.88	0.06	0.00	0.00	0.00	2.48	957.15	31.21
206.00	100.00	0.0	0.0	0.0	0.00	2.44	*****	29.13

II. GAS ANALYSIS (DRY BASIS)

X C CONVERSION	INSTANTANEOUS		GAS ANALYSIS		H2	AVERAGE		GAS ANALYSIS	
	H2	CO	CO2	CH4		CO	CO2	CH4	
46.57	49.60	36.37	12.60	1.43	49.60	36.37	12.60	1.43	
58.16	47.80	38.88	11.96	1.36	49.31	36.77	12.50	1.42	
68.22	47.55	39.29	11.85	1.31	49.10	37.07	12.42	1.41	
76.49	47.94	38.52	12.38	1.17	49.00	37.20	12.41	1.39	
83.60	48.27	37.17	13.48	1.09	48.94	37.20	12.49	1.36	
89.16	48.87	35.42	14.79	0.92	48.94	37.10	12.62	1.34	
93.38	49.60	33.91	16.11	0.37	48.97	36.98	12.76	1.30	
96.44	49.93	32.72	17.35	0.0	48.99	36.85	12.89	1.26	
98.43	50.42	31.11	18.48	0.0	49.02	36.75	12.99	1.24	
99.46	50.10	30.65	19.25	0.0	49.03	36.69	13.05	1.23	
99.88	47.21	32.49	20.30	0.0	49.02	36.68	13.07	1.22	
100.00	45.45	30.91	23.64	0.0	49.02	36.67	13.09	1.22	

RUN 7FT518

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 62.10
 % ASH IN COKE 35.30
 INLET GAS.....STEAM
 COKE SAMPLE.....1KG ILLINOIS COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
19.00	12.62	1.93	0.05	0.04	0.01	2.47	58.81	39.84
34.00	28.35	2.16	0.05	0.05	0.01	2.43	84.16	50.03
48.00	41.80	1.75	0.04	0.04	0.01	2.48	90.22	52.26
63.00	53.64	1.30	0.04	0.04	0.01	2.50	99.41	51.09
77.00	64.10	1.28	0.03	0.03	0.01	2.51	111.28	49.95
92.00	72.87	1.03	0.03	0.03	0.00	2.53	118.09	47.53
106.00	79.63	0.77	0.02	0.02	0.00	2.55	121.57	45.07
121.00	84.77	0.59	0.02	0.01	0.00	2.56	121.18	42.04
135.00	88.59	0.40	0.01	0.01	0.00	2.57	117.70	39.37
150.00	91.21	0.26	0.01	0.01	0.00	2.59	103.42	36.49
164.00	93.00	0.17	0.00	0.00	0.00	2.59	88.31	34.03
179.00	94.18	0.10	0.00	0.00	0.00	2.60	67.70	31.57
193.00	94.92	0.06	0.00	0.00	0.00	2.59	51.59	29.51
208.00	95.43	0.04	0.00	0.00	0.00	2.60	38.26	27.53
222.00	95.77	0.0	0.0	0.00	0.0	2.57	33.89	25.88

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				H2	AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4		CO	CO2	CH4	
12.62	52.14	33.51	13.61	0.73	52.14	33.51	13.61	0.73	
28.35	48.99	39.06	10.88	1.06	50.44	36.50	12.14	0.91	
41.80	47.46	41.97	9.36	1.21	49.52	38.19	11.28	1.00	
53.64	46.80	43.29	8.68	1.23	48.95	39.27	10.73	1.05	
64.10	46.31	44.11	8.41	1.18	48.53	40.03	10.37	1.07	
72.87	46.19	44.47	8.27	1.07	48.26	40.54	10.12	1.07	
79.63	46.04	44.78	8.24	0.94	48.08	40.89	9.97	1.06	
84.77	45.97	45.31	8.25	0.47	47.96	41.15	9.87	1.03	
88.59	45.67	46.11	8.22	0.0	47.86	41.35	9.80	0.98	
91.21	44.60	47.36	8.04	0.0	47.77	41.52	9.75	0.96	
93.00	43.87	48.51	7.62	0.0	47.70	41.64	9.72	0.94	
94.18	43.02	49.86	7.12	0.0	47.65	41.73	9.69	0.93	
94.92	41.96	51.79	6.25	0.0	47.61	41.81	9.66	0.92	
95.43	40.82	53.74	5.44	0.0	47.58	41.86	9.64	0.92	
95.77	28.92	67.47	3.61	0.0	47.53	41.93	9.63	0.92	

RUN FFT520

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 15.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 67.50
 % ASH IN COKE 22.30
 INLET GAS.....STEAM
 COKE SAMPLE.....LSCU WYODAK COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % C/HR.	
			H2	CO	CO2	H2O	INSTANTANEOUS	AVERAGE
18.00	36.10	10.65	0.25	0.02	0.12	2.11	177.53	120.34
32.00	60.96	8.29	0.20	0.02	0.09	2.18	222.27	114.30
47.00	77.71	4.80	0.11	0.02	0.05	2.28	253.63	99.21
61.00	88.45	3.00	0.07	0.01	0.03	2.35	284.41	87.00
76.00	93.37	1.78	0.04	0.01	0.0	2.40	186.26	73.71
90.00	94.55	0.93	0.02	0.00	0.0	2.42	67.24	63.03
120.00	95.68	0.07	0.00	0.0	0.00	2.38	52.24	47.84

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
36.10	64.05	6.09	29.41	0.45	64.05	6.09	29.41	0.45
60.96	63.97	6.42	29.22	0.39	64.02	6.22	29.34	0.42
77.71	63.23	7.47	28.93	0.36	63.85	6.49	29.25	0.41
88.45	61.81	9.45	28.51	0.23	63.61	6.83	29.16	0.39
93.37	68.55	12.24	19.21	0.0	63.91	7.16	28.56	0.37
94.55	83.44	16.56	0.0	0.0	64.44	7.41	27.80	0.36
95.68	80.13	15.86	4.02	0.0	64.76	7.59	27.30	0.35

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 12.56
 COKE CHARGE (GH) 1.11
 % CARBON IN COKE 67.60
 % ASH IN COKE 22.50
 INLET GAS.....CARBON DIOXIDE
 COKE SAMPLE.....LSCU WYODAK COKE

I. INSTANTANEOUS RATES --- BASED ON REMAINING CARBON

TIME MIN.	% C CONVERSION	% CO2 CONVERSION	PARTIAL PRESSURE, ATM		RATE % C/HR.	GAS ANALYSIS, VOL %	
			CO	CO2		CO	CO2
20.00	16.75	2.98	0.16	2.86	60.19	5.45	94.55
35.00	29.24	3.09	0.17	2.85	70.09	5.56	94.44
49.00	40.73	2.68	0.15	2.87	76.08	5.35	94.65
64.00	50.97	2.42	0.14	2.89	80.88	4.80	95.20
78.00	59.92	2.10	0.12	2.91	87.43	4.19	95.81
92.00	67.32	1.66	0.09	2.93	85.53	3.48	96.52
107.00	73.37	1.36	0.08	2.95	85.66	2.86	97.14
121.00	78.37	1.13	0.07	2.96	88.34	2.36	97.64
136.00	82.57	0.96	0.06	2.97	92.22	1.99	98.01
150.00	86.15	0.85	0.05	2.98	102.79	1.70	98.30
164.00	89.21	0.72	0.04	2.98	113.12	1.45	98.55
178.00	91.85	0.63	0.04	2.99	128.36	1.25	98.75
192.00	94.09	0.53	0.03	3.00	149.07	1.07	98.93
206.00	95.96	0.41	0.02	3.00	170.45	0.88	99.12
221.00	97.41	0.31	0.02	3.01	202.15	0.69	99.31
235.00	98.50	0.24	0.01	3.01	262.98	0.52	99.48
249.00	99.25	0.15	0.01	3.02	354.29	0.36	99.64
263.00	99.73	0.09	0.01	3.02	566.80	0.23	99.77
278.00	99.99	0.04	0.00	3.02	11965.35	0.13	99.87

II. AVERAGE RATES --- BASED ON INITIAL CARBON

TIME MIN.	% C CONVERSION	RATE % C/HR.	GAS ANALYSIS	
			CO	CO2
20.00	16.75	50.26	5.45	94.55
35.00	29.24	50.13	5.50	94.50
49.00	40.73	49.87	5.46	94.54
64.00	50.97	47.78	5.31	94.69
78.00	59.92	46.09	5.11	94.89
92.00	67.32	43.91	4.86	95.14
107.00	73.37	41.14	4.59	95.41
121.00	78.37	38.86	4.33	95.67
136.00	82.57	36.43	4.09	95.91
150.00	86.15	34.46	3.86	96.14
164.00	89.21	32.64	3.65	96.35
178.00	91.85	30.96	3.46	96.54
192.00	94.09	29.40	3.29	96.71
206.00	95.96	27.95	3.12	96.88
221.00	97.41	26.45	2.97	97.03
235.00	98.50	25.15	2.82	97.18
249.00	99.25	23.92	2.68	97.32
263.00	99.73	22.75	2.55	97.45
278.00	99.99	21.58	2.43	97.57

RUN FFT522

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV ~ 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 20.58
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 64.40
 % ASH IN COKE 22.70
 INLET GAS.....STEAM
 COKE SAMPLE.....LSCU ILLINOIS COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
17.00	16.72	3.33	0.08	0.05	0.02	2.44	71.63	59.01
45.00	44.86	2.03	0.05	0.03	0.01	2.49	92.87	59.81
60.00	55.39	1.66	0.04	0.03	0.01	2.50	88.69	55.39
74.00	64.02	1.34	0.03	0.02	0.01	2.50	93.33	51.91
88.00	71.06	1.06	0.03	0.02	0.01	2.53	94.17	48.45
103.00	77.15	0.90	0.02	0.02	0.01	2.53	101.05	44.94
117.00	82.24	0.71	0.02	0.01	0.00	2.54	109.64	42.18
132.00	86.52	0.58	0.01	0.01	0.00	2.55	117.72	39.33
146.00	89.93	0.48	0.01	0.01	0.00	2.55	129.39	36.96
160.00	92.60	0.32	0.01	0.01	0.00	2.57	133.90	34.72
175.00	94.70	0.24	0.01	0.01	0.00	2.57	144.22	32.47
189.00	96.30	0.17	0.00	0.00	0.00	2.58	162.00	30.57
204.00	97.58	0.18	0.00	0.00	0.00	2.58	194.50	28.70
218.00	98.58	0.09	0.00	0.00	0.00	2.58	249.04	27.13
233.00	99.27	0.06	0.00	0.00	0.00	2.58	323.41	25.56
247.00	99.73	0.03	0.00	0.00	0.00	2.57	572.00	24.23
261.00	100.00	0.01	0.00	0.00	0.00	2.55	*****	22.99

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4	H2	CO	CO2	CH4
16.72	54.32	31.07	13.70	0.91	54.32	31.07	13.70	0.91
44.86	53.36	32.54	13.41	0.69	53.80	31.87	13.54	0.79
55.39	52.34	34.39	13.02	0.26	53.56	32.28	13.45	0.71
64.02	51.76	35.31	12.94	0.0	53.35	32.64	13.39	0.62
71.06	50.85	36.29	12.86	0.0	53.13	32.96	13.35	0.57
77.15	50.32	37.06	12.62	0.0	52.94	33.24	13.30	0.53
82.24	49.53	37.95	12.52	0.0	52.75	33.49	13.26	0.50
86.52	48.82	38.71	12.47	0.0	52.59	33.71	13.22	0.48
89.93	48.94	38.91	12.14	0.0	52.47	33.88	13.19	0.46
92.60	47.76	40.24	12.00	0.0	52.35	34.04	13.16	0.45
94.70	45.33	42.37	12.31	0.0	52.23	34.19	13.14	0.44
96.30	44.31	43.27	12.42	0.0	52.12	34.31	13.13	0.44
97.58	46.40	41.69	11.91	0.0	52.06	34.39	13.12	0.43
98.58	46.27	41.93	11.80	0.0	52.01	34.45	13.11	0.43
99.27	41.58	46.04	12.38	0.0	51.95	34.52	13.10	0.43
99.73	37.80	49.61	12.60	0.0	51.91	34.57	13.10	0.43
100.00	30.88	55.88	13.24	0.0	51.87	34.60	13.10	0.43

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.63
 COKE CHARGE (G/H) 1.00
 % CARBON IN COKE 64.40
 % ASH IN COKE 22.70
 INLET GAS.....STEAM
 COKE SAMPLE.....LSCU ILLINOIS COKE

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
16.00	6.12	1.68	0.04	0.02	0.01	2.53	23.28	22.96
32.00	14.16	1.84	0.05	0.02	0.01	2.53	38.94	26.55
46.00	22.72	1.93	0.05	0.03	0.01	2.53	46.99	29.64
61.00	31.70	1.82	0.05	0.03	0.01	2.52	52.16	31.18
75.00	39.95	1.66	0.04	0.02	0.01	2.53	54.48	31.96
90.00	47.47	1.55	0.04	0.02	0.01	2.54	56.31	31.65
104.00	54.25	1.48	0.04	0.02	0.01	2.55	57.60	31.30
119.00	60.16	1.35	0.03	0.02	0.01	2.55	57.30	30.33
133.00	65.30	1.12	0.03	0.01	0.01	2.56	57.06	29.46
148.00	69.70	1.03	0.03	0.01	0.00	2.57	56.77	28.25
162.00	73.62	0.98	0.03	0.01	0.00	2.56	59.67	27.27
176.00	77.04	0.90	0.02	0.01	0.00	2.58	58.14	26.26
191.00	80.05	0.82	0.02	0.01	0.00	2.58	58.24	25.15
205.00	82.66	0.68	0.02	0.01	0.00	2.58	58.17	24.19
220.00	84.91	0.61	0.02	0.01	0.00	2.59	56.87	23.16
234.00	86.82	0.55	0.01	0.01	0.00	2.59	55.75	22.26
249.00	88.44	0.54	0.01	0.00	0.00	2.59	53.79	21.31
263.00	89.83	0.48	0.01	0.00	0.00	2.58	53.84	20.49
277.00	91.00	0.49	0.01	0.00	0.00	2.59	50.38	19.71
292.00	92.01	0.44	0.01	0.00	0.00	2.59	48.81	18.91
306.00	92.89	0.36	0.01	0.00	0.00	2.60	47.11	18.21
321.00	93.63	0.37	0.01	0.00	0.00	2.60	43.66	17.50
335.00	94.23	0.35	0.01	0.00	0.00	2.60	39.40	16.88
350.00	94.72	0.24	0.01	0.00	0.00	2.59	36.67	16.24

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS				AVERAGE	GAS ANALYSIS			
	H2	CO	CO2	CH4		H2	CO	CO2	CH4
6.12	64.59	21.15	13.42	0.34	64.59	21.15	13.42	0.34	
14.16	59.85	26.77	12.68	0.71	62.05	24.17	13.02	0.77	
22.72	57.49	29.60	12.21	0.70	60.45	26.07	12.74	0.74	
31.70	56.90	30.38	11.96	0.76	59.50	27.22	12.53	0.75	
39.95	56.89	30.63	11.78	0.71	58.99	27.89	12.38	0.74	
47.47	57.13	30.63	11.59	0.65	58.71	28.31	12.26	0.73	
54.25	58.31	29.93	11.18	0.58	58.66	28.51	12.13	0.71	
60.16	59.87	29.10	10.79	0.25	58.78	28.57	11.99	0.66	
65.30	60.13	29.14	10.73	0.0	58.89	28.61	11.89	0.61	
69.70	60.22	29.11	10.67	0.0	58.98	28.65	11.81	0.57	
73.62	61.19	28.45	10.37	0.0	59.10	28.63	11.73	0.54	
77.04	62.63	27.55	9.82	0.0	59.27	28.58	11.64	0.51	
80.05	64.11	26.63	9.27	0.0	59.48	28.50	11.54	0.49	
82.66	64.28	26.67	9.05	0.0	59.65	28.43	11.45	0.47	
84.91	64.33	26.75	8.92	0.0	59.79	28.38	11.37	0.46	
86.82	65.47	26.02	8.50	0.0	59.93	28.32	11.30	0.45	
88.44	67.56	24.61	7.83	0.0	60.10	28.24	11.22	0.44	
89.83	69.50	23.25	7.25	0.0	60.29	28.14	11.14	0.43	
91.00	71.60	21.74	6.66	0.0	60.50	28.02	11.06	0.42	
92.01	74.14	19.86	6.00	0.0	60.72	27.89	10.98	0.41	
92.89	74.34	19.74	5.92	0.0	60.92	27.77	10.90	0.41	
93.63	75.50	18.87	5.63	0.0	61.10	27.66	10.84	0.40	
94.23	78.86	16.38	4.76	0.0	61.31	27.53	10.77	0.40	
94.72	79.11	16.43	4.46	0.0	61.48	27.42	10.71	0.39	

RUN FFT524

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 20.58
 COKE CHARGE (CM) 1.00
 % CARBON IN COKE 84.90
 % ASH IN COKE 0.06
 INLET GAS.....STEAM
 COKE SAMPLE.....PETROLEUM COKE II.

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	G/HR. AVERAGE
			H2	CO	CO2	H2O		
18.00	19.21	3.76	0.09	0.08	0.02	2.38	82.52	64.02
39.00	43.47	2.96	0.07	0.07	0.01	2.41	111.82	66.88
53.00	56.79	2.26	0.06	0.05	0.01	2.45	115.39	64.30
68.00	67.45	1.71	0.04	0.04	0.01	2.47	117.25	59.51
82.00	75.32	1.18	0.03	0.03	0.01	2.49	113.14	55.11
97.00	80.85	0.88	0.02	0.02	0.00	2.50	104.22	50.01
111.00	85.00	0.68	0.02	0.02	0.00	2.50	108.52	45.95
125.00	88.44	0.59	0.01	0.01	0.00	2.52	116.08	42.45
140.00	91.46	0.47	0.01	0.01	0.00	2.53	129.06	39.20
154.00	93.79	0.36	0.01	0.01	0.00	2.53	137.60	36.54
169.00	95.57	0.27	0.01	0.01	0.00	2.54	149.06	33.93
183.00	96.98	0.22	0.01	0.01	0.00	2.53	176.87	31.80
197.00	98.06	0.16	0.00	0.00	0.00	2.54	203.49	29.87
212.00	98.88	0.11	0.00	0.00	0.00	2.55	251.52	27.98
226.00	99.44	0.05	0.00	0.00	0.00	2.54	344.36	26.40
240.00	99.78	0.03	0.00	0.00	0.00	2.56	548.17	24.95
255.00	100.00	0.02	0.00	0.00	0.00	2.54	*****	23.53

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS				AVERAGE	GAS ANALYSIS			
	H2	CO	CO2	CH4		H2	CO	CO2	CH4
19.21	48.69	40.46	9.58	1.27	48.69	40.46	9.58	1.27	
43.47	48.49	41.86	8.50	1.15	48.59	41.14	9.06	1.21	
56.79	47.65	43.63	7.68	1.04	48.40	41.65	8.78	1.18	
67.45	46.67	44.64	7.76	0.93	48.16	42.07	8.63	1.14	
75.32	46.58	44.63	8.31	0.49	48.01	42.31	8.60	1.08	
80.85	46.73	44.08	9.19	0.0	47.93	42.42	8.64	1.01	
85.00	46.71	43.19	10.10	0.0	47.88	42.45	8.70	0.97	
88.44	46.09	42.98	10.93	0.0	47.81	42.47	8.78	0.93	
91.46	45.38	43.00	11.62	0.0	47.74	42.49	8.87	0.91	
93.79	45.54	42.29	12.17	0.0	47.69	42.48	8.94	0.89	
95.57	45.56	41.80	12.63	0.0	47.66	42.47	9.00	0.87	
96.98	44.74	42.26	13.00	0.0	47.62	42.47	9.09	0.86	
98.06	44.69	42.22	13.09	0.0	47.59	42.47	9.09	0.85	
98.88	44.44	42.16	13.40	0.0	47.57	42.46	9.12	0.85	
99.44	40.70	44.72	14.57	0.0	47.54	42.47	9.15	0.84	
99.78	35.96	48.25	15.79	0.0	47.50	42.49	9.17	0.84	
100.00	35.62	47.95	16.44	0.0	47.48	42.50	9.18	0.84	

RUN FFT527

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 20.58
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 67.80
 % ASH IN COKE 30.30
 INLET GAS..... STEAM
 COKE SAMPLE.....IKG WYODAK COKE COMPOSIT

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAG.
			H2	CO	CO2	H2O		
17.00	69.43	11.00	0.26	0.06	0.11	2.10	570.26	245.03
31.00	93.62	2.85	0.07	0.03	0.03	2.40	996.31	181.19
46.00	99.50	0.27	0.01	0.00	0.00	2.51	*****	129.79
60.00	100.00	0.09	0.00	0.0	0.00	2.41	*****	100.00

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				H2	AVERAGE CO	GAS CO2	ANALYSIS CH4
	H2	CO	CO2	CH4				
69.43	60.09	13.70	25.28	0.93	60.09	13.70	25.28	0.93
93.62	58.70	16.57	24.17	0.55	59.79	14.32	25.04	0.85
99.50	57.30	20.30	22.40	0.0	59.67	14.61	24.91	0.81
100.00	67.23	13.19	19.57	0.0	59.71	14.60	24.88	0.81

RUN FFT525

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 84.90
 % ASH IN COKE 0.06
 INLET GAS..... STEAM
 COKE SAMPLE.....PETROLEUM COKE II.

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				RATE, % INSTANTANEOUS	C/HR. AVERAGE
			H2	CO	CO2	H2O		
17.00	8.92	2.10	0.05	0.04	0.01	2.40	44.29	31.49
31.00	20.40	2.55	0.06	0.05	0.01	2.39	62.51	39.48
46.00	32.98	2.26	0.06	0.05	0.01	2.42	74.69	43.02
61.00	45.43	2.35	0.06	0.05	0.01	2.42	90.52	44.68
75.00	56.87	1.92	0.05	0.04	0.01	2.43	99.69	45.49
90.00	66.11	1.52	0.04	0.04	0.01	2.45	101.64	44.07
104.00	73.56	1.25	0.03	0.03	0.01	2.46	104.67	42.44
119.00	79.42	0.98	0.02	0.02	0.01	2.48	103.87	40.04
133.00	83.93	0.78	0.02	0.02	0.00	2.49	102.63	37.86
148.00	87.34	0.54	0.01	0.01	0.00	2.50	95.90	35.41
162.00	89.82	0.42	0.01	0.01	0.00	2.52	86.72	33.27
177.00	91.58	0.29	0.01	0.01	0.00	2.52	73.40	31.04
191.00	92.82	0.20	0.01	0.00	0.00	2.52	59.41	29.16
206.00	93.63	0.12	0.00	0.00	0.00	2.52	45.34	27.27
220.00	94.22	0.12	0.00	0.00	0.00	2.51	38.02	25.70
234.00	94.65	0.07	0.00	0.00	0.00	2.52	26.95	24.27
249.00	94.91	0.06	0.00	0.00	0.00	2.52	17.52	22.87
263.00	95.09	0.04	0.00	0.00	0.00	2.52	15.59	21.69
278.00	95.28	0.0	0.0	0.00	0.00	2.51	16.48	20.56

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				AVERAGE	GAS ANALYSIS			
	H2	CO	CO2	CH4		H2	CO	CO2	CH4
8.92	52.07	33.97	13.37	0.59	52.07	33.97	13.37	0.59	
20.40	49.32	38.43	11.24	1.02	50.56	36.42	12.20	0.83	
32.98	47.88	40.63	10.33	1.17	49.57	37.97	11.51	0.95	
45.43	47.23	41.97	9.63	1.17	48.95	39.03	11.01	1.01	
56.87	47.01	42.76	9.12	1.11	48.57	39.76	10.64	1.03	
66.11	46.72	43.28	9.03	0.96	48.32	40.24	10.42	1.02	
73.56	46.85	43.16	9.25	0.75	48.17	40.52	10.31	0.99	
79.42	47.42	42.58	9.65	0.35	48.12	40.67	10.26	0.95	
83.93	48.01	41.82	10.17	0.0	48.11	40.74	10.25	0.90	
87.34	47.79	41.41	10.80	0.0	48.10	40.76	10.28	0.86	
89.82	48.09	40.77	11.15	0.0	48.10	40.76	10.30	0.84	
91.58	48.68	40.15	11.18	0.0	48.11	40.75	10.32	0.82	
92.82	48.52	40.25	11.23	0.0	48.12	40.74	10.33	0.81	
93.63	48.39	40.32	11.29	0.0	48.12	40.74	10.34	0.80	
94.22	48.70	40.43	10.87	0.0	48.12	40.74	10.34	0.80	
94.65	50.88	39.18	9.94	0.0	48.14	40.73	10.34	0.79	
94.91	53.27	37.38	9.35	0.0	48.15	40.72	10.34	0.79	
95.09	56.96	35.44	7.59	0.0	48.17	40.71	10.33	0.79	
95.28	33.33	38.60	28.07	0.0	48.15	40.70	10.36	0.79	

RUN FFT528

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 19.63
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 67.80
 % ASH IN COKE 30.30
 INLET GAS.....STEAM
 COKE SAMPLE.....IKG WYO.COMP.#1

I. GASIFICATION RATES

TIME MIN.	% C CONVERSION	% STEAM CONVERSION	PARTIAL PRESSURE, ATM				H2O	RATE, % C/HR.	
			H2	CO	CO2	INSTANTANEOUS		AVERAGE	
15.00	35.36	8.20	0.20	0.03	0.09	2.19	189.57	141.45	
30.00	61.27	5.11	0.12	0.03	0.06	2.30	233.23	122.53	
44.00	79.25	2.95	0.07	0.03	0.03	2.38	290.88	108.06	
59.00	90.17	1.52	0.04	0.02	0.01	2.43	348.35	91.70	
73.00	95.96	0.71	0.02	0.01	0.01	2.46	442.78	78.87	
88.00	98.71	0.30	0.01	0.00	0.00	2.48	595.44	67.30	
102.00	99.73	0.06	0.00	0.00	0.00	2.49	*****	58.67	
117.00	100.00	0.01	0.00	0.0	0.00	2.45	*****	51.28	

II. GAS ANALYSIS (DRY BASIS)

% C CONVERSION	INSTANTANEOUS GAS ANALYSIS				H2	AVERAGE GAS ANALYSIS			
	H2	CO	CO2	CH4		CO	CO2	CH4	
35.36	62.25	6.56	30.51	0.68	62.25	6.56	30.51	0.68	
61.27	59.70	11.60	28.00	0.70	61.27	8.49	29.55	0.69	
79.25	56.61	17.96	24.73	0.70	60.34	10.37	28.59	0.69	
90.17	54.26	23.62	21.69	0.43	59.72	11.73	27.88	0.67	
95.96	52.78	27.40	19.82	0.0	59.37	12.52	27.48	0.63	
98.71	52.03	28.91	19.07	0.0	59.20	12.89	27.28	0.62	
99.73	51.13	29.13	19.74	0.0	59.14	13.03	27.22	0.61	
100.00	45.83	27.78	26.39	0.0	59.11	13.06	27.22	0.61	

RUN FFT529

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 30
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CM/SEC) 17.81
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 67.70
 % ASH IN COKE 30.20
 INLET GAS.....CARBON DIOXIDE
 COKE SAMPLE.....IKG WYO. COMP. #1

I. INSTANTANEOUS RATES --- BASED ON REMAINING CARBON

TIME MIN.	% C CONVERSION	% CO2 CONVERSION	PARTIAL PRESSURE, ATM CO	CO2	RATE % C/HR.	GAS ANALYSIS, VOL % CO	CO2
18.00	46.99	6.21	0.41	2.62	256.88	13.47	86.53
33.00	75.92	3.08	0.20	2.81	352.33	10.08	89.92
47.00	88.51	1.18	0.08	2.95	310.80	4.61	95.39
61.00	92.58	0.22	0.01	3.01	235.24	1.51	98.49

II. AVERAGE RATES --- BASED ON INITIAL CARBON

TIME MIN.	% C CONVERSION	RATE % C/HR.	GAS ANALYSIS CO	CO2
18.00	46.99	156.63	13.47	86.53
33.00	75.92	138.03	11.94	88.06
47.00	88.51	112.99	9.73	90.27
61.00	92.58	91.06	7.86	92.14

RUN FFT530

GASIFICATION RATES FROM MINIGASIFIER

TEMPERATURE (DEG. F) RV - 130
 PRESSURE (PSIG) 30.00
 SUPERFICIAL VELOCITY (CH/SEC) 16.98
 COKE CHARGE (GM) 1.00
 % CARBON IN COKE 67.90
 % ASH IN COKE 29.70
 INLET GAS.....CARBON DIOXIDE
 COKE SAMPLE.....IKGVYO. COMP. #1

I. INSTANTANEOUS RATES --- BASED ON REMAINING CARBON

TIME MIN.	% C CONVERSION	% CO2 CONVERSION	PARTIAL PRESSURE, ATM		RATE % C/HR.	GAS ANALYSIS, VOL %	
			CO	CO2		CO	CO2
17.00	31.85	4.39	0.27	2.74	147.33	9.04	90.96
37.00	61.32	2.84	0.18	2.84	193.97	7.45	92.55
51.00	75.70	2.02	0.12	2.90	211.57	4.98	95.02
65.00	85.32	1.27	0.08	2.95	222.24	3.37	96.63
79.00	90.93	0.66	0.04	2.98	199.03	2.03	97.97
94.00	93.95	0.31	0.02	3.01	142.68	1.06	98.94
109.00	95.24	0.11	0.01	3.02	109.11	0.46	99.54

II. AVERAGE RATES --- BASED ON INITIAL CARBON

TIME MIN.	% C CONVERSION	RATE % C/HR.	GAS ANALYSIS	
			CO	CO2
17.00	31.85	112.39	9.04	90.96
37.00	61.32	99.44	8.20	91.80
51.00	75.70	89.06	7.30	92.70
65.00	85.32	78.75	6.45	93.55
79.00	90.93	69.06	5.69	94.31
94.00	93.95	59.97	4.99	95.01
109.00	95.24	52.43	4.40	95.60

APPENDIX H

FBU COKE GASIFICATION DATA

Appendix H contains data obtained in coke gasification studies on the Fluidized Bed Unit (FBU). Two types of runs were made. A series of runs were made to investigate slag formation. These runs employed moderate superficial velocities and relatively high temperatures. Run conditions are listed in Table 6-15 of the body of this report. The rest of the runs were fines-make runs, that used a higher superficial velocity on the order of that expected in the commercial unit. Fines-make runs were 70A, 73 and 81-98. Temperatures were RV-30°F except for run 98, which used RV-130°F.

For runs 92 and 95, the cyclone was bypassed so that overhead solids carryover was collected in the fines filter.

The LSCU cokes that were ground and screened were still so small that, entrainment was high. For some of the runs (81, 82, 85, 91, 92, 95 and 98) data are shown for pre-cyclone and pre-FF samples. These are the solids carried over by entrainment before the reactant gases (air and steam) were started to the reactor.

TABLE H-1
FBU COKE GASIFICATION RUN 70A

		Cyclone					
		Feed	Bed	1	2	3	4
Mesh	20	0	0	0	0	0	0
(Cum.	30	0	0	0	0	0	0
Wt.%)	50	8.70	7.34	0	0	0	0
	100	97.05	96.43	1.83	1.36	2.56	2.53
	200	99.62	99.49	14.89	12.57	23.00	26.11
	325	99.71	99.61	18.71	14.87	23.73	26.49
	-325	100.00	99.98	100.00	100.00	99.99	100.00
% Fines		0.29	0.37	81.29	85.13	76.26	73.51
Ash Wt.%							
Whole		~34.1	60.73				
-325				96.67	96.86	97.51	97.61
+325				44.63	45.05	50.63	53.89
% Gasification		0	~77	~29	~48	~67	~77
% Fines Make		---	---	0.8	2.2	2.8	3.5
Total Grams		1994	~900	20	32	15	20
Elapsed Time (Hr)		0	4	1.5	2.5	3.5	4

FEED: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.

TABLE H-2
FBU COKE GASIFICATION RUN 70B

		Cyclone			
		Feed	Bed	1	2
Mesh	20	0	0	0	0
(Cum.	30	0	0	0	0
Wt.%)	50	7.34	3.51	0	0
	100	96.43	88.84	2.50	12.11
	200	99.49	98.34	10.52	26.43
	325	99.61	100.00	21.93	32.18
	-325	100.00	100.00	100.00	100.00
% Fines		0.37	0	78.07	67.82
Ash Wt.%					
Whole		60.73	85.75		
-325				97.41	97.23
+325				52.67	71.20
% Fines Make		---	---	0.5	1.4
Total Grams		~900	642	6	12
Elapsed Time (Hr)		0	4	2	4

FEED: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31
Bed from 70A.

TABLE H-4

FBU COKE GASIFICATION RUN 72

		Cyclone			
		Feed	Bed	1	2
Mesh	20	0	0.06		
(Cum.	30	0	0.06		
Wt.%)	50	8.70	6.41		
	100	97.05	90.25		
	200	99.62	91.85		
	325	99.71	98.46		
	-325	100.00	100.00		
	% Fines	0.29	1.54		
Ash Wt.%					
	Whole	57.5	79.35*	63.44	
	-325				
	+325				
Total Grams		700	348**	1.2	2
Elapsed Time (Hr)		0	2.5	2	2.5
*SO ₃ corrected			**Includes 225 g chunk of slag		

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.
Bed from Run 69.

TABLE H-3

FBU COKE GASIFICATION RUN 71

		Cyclone				
		Feed	Bed	1	2	3
Mesh	20	0	0			
(Cum.	30	0	0			
Wt.%)	50	8.70	10.68			
	100	97.05	91.70			
	200	99.62	95.41			
	325	99.71	99.65			
	-325	100.00	99.99			
	% Fines	0.29	0.34			
Ash Wt.%						
	Whole	57.5	81.74		57.17	70.40
	-325			87.08		
	+325			48.89		
Total Grams		702	552	5.8	0.7	1.2
Elapsed Time (Hr)		---	4	2	3	4

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.
Bed from Run 69.

TABLE H-5
FBU COKE GASIFICATION RUN 73

		Cyclone							
		Feed	Bed	1	2	3	4	5	6
Mesh	20	0.75	0	0	0	0	0	0	0
(Cum.	30	0.87	0	0	0	0	0	0	0
Wt.%)	50	15.78	7.92	0	0	0	0	0	0
	100	96.69	97.20	1.07	0.72	0.50	0.58	0.85	1.33
	200	97.06	97.55	70.92	12.28	1.99	5.90	3.39	15.02
	325	98.81	99.12	73.88	23.71	9.87	9.50	13.43	17.46
	-325	100.00	99.99	100.00	99.99	100.00	100.00	100.00	100.00
	% Fines	1.19	0.87	26.12	76.28	90.13	90.50	86.57	82.54
Ash Wt.%									
Whole		34.11*	54.28						
	-325			77.13	92.06*	95.50*	96.74*	96.98*	97.17*
	+325			23.02*	28.84*	38.04*	45.05*	48.76*	49.98*
% Gasification		0	0	7	21	35	49	63	70
% Fines Make		---	---	0.0	0.4	1.5	2.5	2.9	4.0
Total Grams		2991	1480	4	14	35	33	16	38
Elapsed Time (Hr)		0	5	0.5	1.5	2.5	3.5	4.5	5.0

*SO₃ corrected

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.

TABLE H-6
FBU COKE GASIFICATION RUN 74

		Cyclone				
		Feed	Bed	1	2	3
Mesh	20	0	0			
(Cum.	30	0	0			
Wt.%)	50	7.92	3.45			
	100	97.20	84.71			
	200	97.55	89.08			
	325	99.12	95.52			
	-325	99.99	100.00			
	% Fines	0.87	4.48			
Ash Wt.%						
Whole		54.28	98.5*		59.90	91.55
	-325			91.04		
Total Grams		691	382	4	2	2
Elapsed Time (Hr)		0	3.5	2	3	3.5

*SO₃ corrected

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.
Bed from Run 73.

TABLE H-7
FBU COKE GASIFICATION RUN 75

		<u>Cyclone</u>				
		<u>Feed</u>	<u>Bed</u>	<u>1</u>	<u>2</u>	<u>3</u>
Mesh	20	0	0			
(Cum.	30	0	0			
Wt.%)	50	7.92	5.77			
	100	97.20	80.72			
	200	97.55	99.80			
	325	99.12	99.90			
	-325	99.99	100.00			
% Fines		0.87	0.10			
Ash Wt.%						
Whole		54.28	97.1*	97.08		
% Fines Make		---	---	0.1	0.3	0.4
Total Grams		697	393	0.5	1.5	1.0
Elapsed Time (Hr)		0	3.5	2	3	3.5

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.
Bed from Run 73.

TABLE H-8
FBU COKE GASIFICATION RUN 76

		<u>Cyclone</u>		
		<u>Feed</u>	<u>Bed</u>	<u>1</u>
Mesh	20	0	0.62	
(Cum.	30	0	0.87	
Wt.%)	50	13.53	15.05	
	100	54.20	58.58	
	200	99.86	96.95	
	325	99.93	99.25	
	-325	100.00	100.00	
% Fines		0.07	0.25	
Ash Wt.%				
Whole		34.08*	38.64*	
% Fines Make		---	---	0.2
Total Grams		1399	1191	3
Elapsed Time (Hr)		0	2	2
*SO ₃ corrected				

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.

TABLE H-9

FBU COKE GASIFICATION RUN 77

	Feed	Bed	Cyclone					
			1	2	3	4	5	6
Ash Wt.% Whole	34.08*	53.0*						
% Gasification			5	15	24	34	44	49
% Fines Make	---	---	0.1	0.2	0.3	0.4	0.5	0.8
Total Grams	1050	705	1	1	1.3	1.3	0.8	3.0
Elapsed Time (Hr)	0	5	0.5	1.5	2.5	3.5	4.5	5
*SO ₃ corrected								

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.

TABLE H-10

FBU COKE GASIFICATION RUN 77

	Feed	Bed	Cyclone					
			1	2	3	4	5	6
Ash Wt.% Whole	34.08*	53.0*						
% Gasification			5	15	24	34	44	49
% Fines Make	---	---	0.1	0.2	0.3	0.4	0.5	0.8
Total Grams	1050	705	1	1	1.3	1.3	0.8	3.0
Elapsed Time (Hr)	0	5	0.5	1.5	2.5	3.5	4.5	5
*SO ₃ corrected								

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.

TABLE H-11
FBU COKE GASIFICATION RUN 78

		Cyclone				
		Feed	Bed	1	2	3
Mesh	20	0	0			
(Cum.	30	0	0			
Wt.%)	50	19.20	16.21			
	100	33.07	16.76			
	200	98.09	61.36			
	325	100.00	99.79			
	-325	100.00	100.01			
% Fines		0	0.22			
Ash Wt.%						
Whole		34.06*	52.19*			
-325				94.34*		
% Gasification		0	~53	~18	~35	~53
% Fines Make		---	---	~0.3	~0.6	~0.7
Total Grams		1048	675	3.3	3.1	0.5
Elapsed Time (Hr)		0	3	1	2	3

*SO₃ corrected

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.31.

TABLE H-12
FBU COKE GASIFICATION RUN 79

		Cyclone				
		Feed	Bed	1	2	3
Mesh	20	0.53	0			
(Cum.	30	0.53	0			
Wt.%)	50	18.88	15.12			
	100	96.91	98.39			
	200	100.00	99.83			
	325	100.00	99.94			
	-325	100.00	100.00			
% Fines		0	0.06			
Ash Wt.%						
Whole		35.22*	44.93*			
-325				96.83*	97.00*	97.91*
% Gasification		0	45	18	36	45
% Fines Make		---	---	---	---	---
Total Grams		2993	2100	6	12	9
Elapsed Time (Hr)		0	5	2	4	5

*SO₃ corrected

Feed: EDS Illinois Coke from IKG FLEXICOKING process reactor, MB 20.33.

TABLE H-13
FBU COKE GASIFICATION RUN 80

		<u>Cyclone</u>	
		<u>Feed</u>	<u>Bed</u>
			<u>1</u>
Mesh	20		1.04
(Cum.	30		1.04
Wt.%)	50	~1.2	2.73
	100	~35	38.93
	200	~86	90.40
	325	~96.5	96.78
	-325	100.00	100.00
% Fines		~3.5	3.22
Ash Wt.%			
Whole	19.42*	~26.59*	
-325			70.55*
+325			
% Gasification	0	57	57
% Fines Make	---	---	
Total Grams	1009	513	36
Elapsed Time (Hr)	0	2	2

*SO₃ corrected

Feed: EDS Wyodak Coke from LSCU Run 17, ground -50 +200 mesh.

TABLE H-14
FBU COKE GASIFICATION RUN 81

		<u>Cyclone</u>					<u>Pre-Cyc</u>	<u>Pre-FF</u>
		<u>Feed</u>	<u>Bed</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	
Mesh	20	0		0	0	0		0
(Cum.	30	0		0	0	0		0
Wt.%)	50	~1.2		0	0	0		0
	100	~35		19.05	22.80	36.08		10.26
	200	~86		89.09	94.92	84.62		57.91
	325	~96.5		93.03	96.16	87.94		64.35
	-325	100.00		100.01	99.99	99.99		99.99
% Fines		~3.5		6.98	3.83	15.05		35.64
Ash Wt.%								
Whole	~19.4							
-325						81.95*	20.03*	19.65*
+325				23.58*	31.36*	42.52*	19.56*	18.94*
% Gasification	0	81	13	40	66	81	---	---
% Fines Make	---	---	0.2	0.6	1.4	1.5	---	---
Total Grams	3100	157	157	243	141	3	973.4	60
Elapsed Time (Hr)	0	3	0.5	1.5	2.5	3	0	0

*SO₃ corrected

Feed: EDS Wyodak Coke from LSCU Run 17, ground -50 +200 mesh.

TABLE H-15

FBU COKE GASIFICATION RUN 82

		Cyclone						Pre-Cyc	Pre-FF
		Feed	Bed	1	2	3	4		
Mesh	20	0	0	0	0	0	0	0	0
(Cum.	30	0	0	0	0	0	0	0	0
Wt.%)	50	0.64	2.75	0	0	0	0	0	0
	100	33.57	45.30	0.28	0.71	1.50	0.68	7.27	3.81
	200	83.60	95.30	80.11	95.53	88.03	60.92	62.39	60.68
	325	91.80	97.25	98.86	98.03	89.03	65.19	90.13	92.84
	-325	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
	% Fines	8.20	2.75	1.14	1.97	10.97	34.81	9.87	7.16
Ash Wt.%									
Whole		19.25*	54.92*					20.16*	22.25*
-325						80.79*	92.24*		
+325				23.48*	31.65*	41.74*	51.01*		
% Gasification		0	67	11	34	56	67	---	---
% Fines Make		---	---	0.5	0.8	1.8	3.8	---	---
Total Grams		3460	195	209	285	153	118	1035	310
Elapsed Time (Hr)		0	3	0.5	1.5	2.5	3	0	0

*SO₃ corrected

Feed: EDS Wyodak Coke from LSCU Run 17, ground -50 +200 mesh.

TABLE H-16

FBU COKE GASIFICATION RUN 85

		Cyclone						Pre-Cyc	Pre-FF
		Feed	Bed	1	2	3	4		
Mesh	20	0	0	0	0	0	0		
(Cum.	30	0.20	0	0	0	0	0		
Wt.%)	50	0.36	0.11	0	0	0	0		
	100	25.14	46.68	0.20	0.59	1.52	1.49		
	200	83.50	98.74	88.46	96.12	92.71	87.24		
	325	96.40	99.27	98.99	99.24	93.72	88.73		
	-325	100.00	100.01	100.00	100.00	100.00	99.99		
	% Fines	3.60	0.74	1.01	0.76	6.28	11.26		
Ash Wt.%									
Whole		18.95*						20.41*	22.70*
-325						72.25*	77.42*		
+325				23.86*	29.62*	37.88*	42.51*		
% Gasification		0	55	9	28	46	55	---	---
Total Grams		2600	640	174	258	223	109	47	14
Elapsed Time (Hr)		0	3	0.5	1.5	2.5	3	0	0

*SO₃ correctedFeed: EDS Wyodak Coke from LSCU Run 17, ground -50 +200 mesh.
Bed from Runs 83 and 84.

TABLE H-17

FBU COKE GASIFICATION RUN 88

		Cyclone						
		Feed	Bed	1	2	3	4	5
Mesh	20	0	0	0	0	0	0	0
(Cum.	30	0	0	0	0	0	0	0
Wt.%)	50	0.21	2.29	0	0	0	0	0
	100	38.17	41.08	0.63	0.07	0.29	0.09	0.18
	200	98.55	98.86	83.36	84.34	79.56	74.02	69.19
	325	99.84	99.05	97.31	89.54	82.34	75.39	71.14
	-325	100.00	100.00	100.00	100.01	100.00	100.00	100.00
% Fines		0.16	0.95	2.69	10.47	17.66	24.61	28.86
Ash Wt.%								
	Whole	25.98*	87.85*					
	-325				92.96*	96.34*	97.08*	
	+325			27.92*	31.11*	33.75*	38.78*	42.65*
% Gasification		0	51	6	19	32	45	51
% Fines Make		---	---	0.2	0.6	1.3	2.3	3.9
Total Grams		2600	1107	56	83	100	102	137
Elapsed Time (Hr)		0	4	0.5	1.5	2.5	3.5	4

*SO₃ corrected

Feed: EDS Illinois Coke from LSCU Run 12, ground +100 mesh.
Bed from Runs 86 and 87.

TABLE H-18

FBU COKE GASIFICATION RUN 91

		Cyclone							
		Feed	Bed	1	2	3	4	5	6
Mesh	20	0	0	0	0	0	0	0	0
(Cum.	30	0	0.07	0	0	0	0	0	0
Wt.%)	50	0.13	5.57	0	0	0	0	0	0
	100	42.74	36.13	0.41	0.14	0.08	0.09	0	0.25
	200	92.91	99.22	87.35	89.03	82.40	71.51	67.07	93.08
	325	99.34	99.50	97.84	93.26	84.36	73.03	70.03	98.52
	-325	100.00	99.99	100.00	100.01	99.99	100.00	100.00	100.00
% Fines		0.66	0.49	2.16	6.75	15.63	26.97	29.97	1.48
Ash Wt.%									
	Whole	~25		28.45	33.99				
	-325					95.44	97.57	98.76	97.93
	+325					33.32	39.36	45.09	57.19
% Gasification		0	73	7	22	36	51	66	73
% Fines Make		---	---	0.1	0.4	1.1	2.3	3.7	4.5
Total Grams		2200	518	44	60	102	91	107	60
Elapsed Time (Hr)		0	5	0.5	1.5	2.5	3.5	4.5	5

Feed: EDS Illinois Coke from LSCU Run 12, ground +100 mesh.
Bed from Runs 89 and 90.

TABLE H-19
FBU COKE GASIFICATION RUN 92

		Fines Filter							
		Feed	Bed	1	2	3	4	5	Pre-FF
Mesh	20		0	0	0	0	0	0	0
(Cum.	30		0	0	0	0	0	0	0
Wt.%)	50		1.93	0	0	0	0	0	0.06
	100		32.00	0.38	0.07	0.08	0	0	24.77
	200		99.33	92.37	97.92	97.01	95.46	51.45	94.02
	325		99.66	99.68	99.64	97.88	96.83	53.15	99.48
	-325		99.99	100.00	100.00	100.01	100.00	100.00	100.00
% Fines			0.33	0.32	0.36	2.13	3.17	46.85	0.52
Ash Wt.%									
Whole		25.36	41.19	27.17	32.07	38.02	43.89		24.85
-325								96.40	
+325								49.58	
% Gasification		0	65	8	24	41	57	65	---
% Fines Make		---	---	0	0	0.2	0.3	1.7	---
Total Grams		2000	580	50	95	146	85	57	31
Elapsed Time (Hr)		0	4	0.5	1.5	2.5	3.5	4	0

Feed: EDS Illinois Coke from LSCU Run 12, ground +100 mesh.
Bed from Run 89.

TABLE H-20
FBU COKE GASIFICATION RUN 95

			Fines Filter							
			Feed	Bed	1	2	3	4	5	6
Mesh (Cum. Wt.%)	20	0	0.07	0	0	0	0	0	0	0
	30	0	0.14	0	0	0	0	0	0	0
	50	0.45	1.35	0	0	0	0	0	0	0
	100	57.65	35.85	0.53	0	0	0	0	0	0.11
	200	98.78	98.78	76.56	94.33	96.65	94.37	88.31	43.62	
	325	100.01	99.14	87.41	98.30	98.25	95.43	90.37	46.51	
	-325	100.01	100.00	99.99	99.99	100.00	100.00	100.00	100.00	
% Fines		0	0.86	12.58	1.69	1.75	4.57	9.63	53.49	
% Gasification		0	67	7	20	34	47	60	67	
% Fines Make		---	---	0.3	0.4	0.5	0.7	1.0	3.0	
Total Grams		2200	608	58	90	80	86	73	82	
Elapsed Time (Hr)		0	5	0.5	1.5	2.5	3.5	4.5	5	

Feed: EDS Illinois Coke from LSCU Run 12, ground +100 mesh.
Bed from Run 93.

TABLE H-21

FBU COKE GASIFICATION RUN 98

		Cyclone								Pre-Cyc
		Feed	Bed	1	2	3	4	5	6	
Mesh	20	0	0	0	0	0		0	0	0
(Cum.	30	0	0	0	0	0		0	0	0
Wt.%)	50	6.62	4.66	0	0	0		0	0	0.41
	100	78.55	69.93	0.23	0.45	1.36		0.60	0.75	50.07
	200	98.22	98.76	93.89	79.41	62.13		60.60	62.25	95.67
	325	98.35	98.76	96.98	81.47	66.44		65.06	67.12	98.51
	-325	99.99	100.00	100.00	100.00	100.00		100.00	100.00	100.00
% Fines		1.64	1.24	3.02	18.53	33.56		34.94	32.88	1.49
% Gasification		0	69	7	21	35	48	62	69	---
% Fines Make		---	---	0.2	0.6	1.7	2.3	3.5	4.2	---
Total Grams		2600	982	51	57	80	48	83	53	33
Elapsed Time (Hr)		0	5	0.5	1.5	2.5	3.5	4.5	5	0

Feed: EDS Wyodak Coke from IKG FLEXICOKING process reactor, MB 24.03.

APPENDIX I

IKG PILOT UNIT OPERATIONS

Tables 1 and 2 describe the March-June, 1978, IKG operations. Coke balances were made when feed was to the unit for the entire material balance. Table I-1 shows a unit line-out period was taken before starting a coke balanced material balance. However, material balance data were also taken during the line-out and down periods as indicated by the material balance designations.

Table I-2 summarizes nominal operating conditions.

TABLE I-1

OPERATING PERIODS

Run 21, Illinois Bottoms

Runs 22-24, Wyodak Bottoms

Run Period	Date, Time		Material Balance	Coke Balance?	Date, Time		Principal Cause of Shutdown
	SOP	EOF			SOB	EOB	
21A	3/12-1100	3/13-1930	21.00	No	---	3/12-1700	Feed nozzle oil side plug
			21.01	Yes	3/12-1700	3/13-1700	
			21.02	No	3/13-1700		
21B	3/15-2140	3/16-1845	21.02	No		3/15-2330	Heater overhead plug
			21.03	Yes	3/15-2330	3/16-1845	
			21.04	No	3/16-1845		
21C	3/21-0645	3/22-1750	21.04	No		3/21-0830	Feed exhausted, unable to transfer
			21.05	Yes	3/21-0830	3/22-1230	
			21.06	No	3/22-1230		
21D	3/23-0230	3/24-0700	21.06	No		3/23-0800	Worn feed pump
			21.07	Yes	3/23-0800	3/24-0700	
			21.08	No	3/24-0700		
21E	3/25-1830	3/26-1300	21.08	No		3/25-2100	Feed nozzle oil side plug
			21.09	Yes	3/25-2100	3/26-1300	
22A	4/5-1220	4/6-1345	22.00	No	---	4/5-1600	Feed nozzle oil side plug
			22.01	Yes	4/5-1600	4/6-1345	
			22.02	No	4/6-1345		
22B	4/6-2010	4/9-2100	22.02	No		4/7-2100	Voluntary shutdown for feed nozzle change
			22.03	Yes	4/7-2100	4/8-2100	
			22.04	Yes	4/8-2100	4/9-2100	
			22.05	No	4/9-2100		
22C	4/13-1915	4/15-0915	22.05	No		4/13-2100	Quench plugged
			22.06	Yes	4/13-2100	4/14-2100	
			22.07	Yes	4/14-2100	4/15-0915	
			22.08	No	4/15-0915		
22D	4/18-1600	4/19-0100	22.08	No			Feed exhausted, unable to transfer
22E	4/19-1315	4/21-2000	22.08	No		4/19-2000	Voluntary shutdown for feed nozzle change
			22.09	Yes	4/19-2000	4/20-2000	
			22.10	Yes	4/20-2000	4/21-2000	
			22.11	No	4/21-2000		
22F	4/22-1245	4/25-0500	22.11	No		4/22-1900	Worn feed pump
			22.12	Yes	4/22-1900	4/23-1900	
			22.13	Yes	4/23-1900	4/25-0500	
			22.14	No	4/25-0500		
22G	4/26-0300	4/28-0800	22.14	No		4/26-0800	Voluntary shutdown for feed nozzle change
			22.15	Yes	4/26-0800	4/27-0800	
			22.16	Yes	4/27-0800	4/28-0800	
			22.17	No	4/28-0800		
22H	4/28-1420	4/30-1300	22.17	No		4/28-1630	Worn feed pump, worn coke feeders
			22.18	Yes	4/28-1630	4/29-1630	
			22.19	Yes	4/29-1630	4/30-1300	
23A	5/6-0700	5/8-0830	23.00	No	---	5/6-1400	Reactor coke agglomeration
			23.01	Yes	5/6-1400	5/7-1400	
			23.02	Yes	5/7-1400	5/8-0830	
			23.03	No	5/8-0830		
23B	5/9-1430	5/10-1130	23.03	No		5/9-2100	Worn feed pump
			23.04	Yes	5/9-2100	5/10-1130	
			23.05	No	5/10-1130		

TABLE I-1
OPERATING PERIODS

CONTINUED

Runs 21-24

<u>Run Period</u>	<u>Date, Time</u>		<u>Material Balance</u>	<u>Coke Balance?</u>	<u>Date, Time</u>		<u>Principal Cause of Shutdown</u>
	<u>SOP</u>	<u>EOP</u>			<u>SOB</u>	<u>EOB</u>	
23C	5/10-2230	5/11-0545	23.05	No			Seed coke system plugged
23D	5/11-2100	5/12-1030	23.05	No		5/12-0030	Reactor cyclone/dipleg plugged
			23.06	No	5/12-0030		
23E	5/13-1230	5/15-0000	23.06	No		5/13-1900	Reactor cyclone/dipleg plugged
			23.07	Yes	5/13-1900	5/15-0000	
24A	5/21-0700	5/23-1000	24.00	No	---	5/21-2200	Voluntary shutdown for feed nozzle change
			24.01	Yes	5/21-2200	5/23-1000	
			24.02	No	5/23-1000		
24B	5/23-2130	5/25-1100	24.02	No		5/24-0300	Worn feed pump
			24.03	Yes	5/24-0300	5/25-1100	
			24.04	No	5/25-1100		
24C	5/25-1700	5/27-0800	24.04	No		5/25-2000	Voluntary shutdown for feed nozzle change
			24.05	Yes	5/25-2000	5/27-0800	
			24.06	No	5/27-0800		
24D	5/27-1400	5/29-0730	24.06	No		5/27-1900	Voluntary shutdown for feed nozzle change
			24.07	Yes	5/27-1900	5/29-0730	
			24.08	No	5/29-0730		
24E	5/29-1400	5/31-0600	24.08	No		5/29-1800	Voluntary shutdown for feed nozzle change
			24.09	Yes	5/29-1800	5/31-0600	
			24.10	No	5/31-0600		
24F	5/31-1700	6/2-0800	24.10	No		5/31-2000	Voluntary shutdown for feed nozzle change
			24.11	Yes	5/31-2000	6/2-0800	
			24.12	No	6/2-0800		
24G	6/3-0900	6/5-0600	24.12	No		6/3-1800	Voluntary shutdown for feed nozzle change
			24.13	Yes	6/3-1800	6/5-0600	
			24.14	No	6/5-0600		
24H	6/6-1115	6/8-0230	24.14	No		6/6-1430	Voluntary shutdown for feed nozzle change
			24.15	Yes	6/6-1430	6/8-0230	
			24.16	No	6/8-0230		
24I	6/8-0815	6/9-0830	24.16	No		6/8-1200	Reactor coke agglomeration
			24.17	Yes	6/8-1200	6/9-0830	
			24.18	No	6/9-0830		
24J	6/10-2030	6/12-0900	24.18	No		6/11-0100	Reactor coke agglomeration
			24.19	Yes	6/11-0100	6/12-0900	
			24.20	No	6/12-0900		
24K	6/13-1545	6/15-1400	24.20	No		6/13-2000	Feed pump mechanical failure
			24.21	Yes	6/13-2000	6/15-1400	

TABLE I-2

NOMINAL IKG OPERATING CONDITIONS

Feedstock	---- Illinois Bottoms ----			----- Wyodak Bottoms -----						
Run and Balance	21.01	21.02-08	21.09	22.01-09	22.10-19	23.01-07	24.01-03	24.04-05	24.06-21	
IKG Operating Targets:										
Reactor Temperature, °F	-----				RV+70	-----				
Heater Temperature, °F	-----				RV+70	-----				
Gasifier Temperature, °F	-----RV-55-----				RV-105	RV-130	RV-180	RV-130	RV-130	
Reactor Pressure, psig	-----				25	-----				
Gross Coke Carbon										
Gasification, %	50	-----			80	-----		50-60	60	80
Gasifier Steam on Air										
plus Steam, Mole %	-----				22	-----				

83A

LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BARD	Baytown Research and Development Division
B/CD	Barrels Per Calendar Day
B/D	Barrels Per Day
B/SD	Barrels Per Stream Day
BYU	Brigham Young University
CLB	Coal Liquefaction Bottoms
CLPP	Coal Liquefaction Pilot Plant
Co/Mo	Cobalt/Molybdenum
CSCU	Continuous Stirred Coking Unit
DCF	Discounted Cash Flow
DEA	Diethanolamine
DOE	Department of Energy
ECLP	Exxon Coal Liquefaction Pilot Plant
EDS	Exxon Donor Solvent
EOR	End-of-Run
EPA	Environmental Protection Agency
EPRI	Electric Power Research Institute
ER&E	Exxon Research and Engineering Company
FBU	Fluidized Bed Unit
G	Billion
GPM	Gallons Per Minute
HBG	High Btu Gas
IBG	Intermediate Btu Gas
IBP	Initial Boiling Point
IKG	Integrated Coking/Gasification Pilot Plant
k	Thousand
LBG	Low Btu Gas
LHV	Lower Heating Value
LP	Linear Programming
LPG	Liquefied Petroleum Gas
LSCU	Large Stirred Coker Unit

LIST OF ABBREVIATIONS (cont'd)

LSFO	Low Sulfur Fuel Oil
M	Million
MBG	Moving-Bed Coal Gasification
MPSS	Multi-Pass Spent Solvent
M20, M21, M22	Bench-Scale Hydrogenation Units
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
RCLU-1	50 Pounds-Per-Day Recycle Coal Liquefaction Unit
RCLU-2	100 Pounds-Per-Day Recycle Coal Liquefaction Unit
RCO	Raw Creosote Oil
RV	Reference Value. Under the terms of this Agreement, all non-program data are coded in terms of Reference Values to prevent disclosure.
SCF	Standard Cubic Feet
SOR	Start-of-Run
SQI	Solvent Quality Index
T/D	Tons Per Day
TEC	Total Erected Cost
TIR	Temperature Increase Required
T/SD	Tons Per Stream Day
VGO	Vacuum Gas Oil
V/Hr/V	Volumetric Hourly Space Velocity, Ft^3 feed per hour per ft^3 of reactor volume.
VLE	Vapor Liquid Equilibrium
VPS	Vacuum Pipe Still
W/Hr/W	Weight Hourly Space Velocity, lb feed per hour per lb of catalyst.
YP	Yield Period
YT	Yield Test