LABORATORY PROCESS RESEARCH AND DEVELOPMENT

LIQUEFACTION PROCESS RESEARCH

4. Bench Units, Modeling and Correlation Studies

Batch tubing bomb autoclaves and continuous flow reactors were employed to provide yield and operability data for a variety of coals. The yield data from the bench scale hydrotreaters and the liquefaction pilot units were correlated to provide models for recycle solvent hydrogenation and liquefaction which were used for process simulation.

Bench unit studies from the beginning of Phase IIIB (3Q77) to the completion of the EDS program (4Q82) are outlined in Table 4-1. Bench studies performed on OTCLU have been listed separately from those conducted in tubing bomb autoclaves.

4.1 Tubing Bomb Autoclave Studies

Tubing bombs (Figure 4-1) have principally been used for bench scale research in the various areas of coal liquefaction classified below:

- o Direct comparison of reactivity of coals in terms of overall conversion to both liquids and gas. Tubing bombs are useful tools for coal screening studies prior to pilot plant testing.
- o Evaluation of potential process improvements, e.g., staged temperature, staged liquefaction, bottoms recycle, and coal pretreatment effectiveness.
- o Process variable studies to determine the effects of temperature, holding time, hydrogen partial pressure, solvent quality and solvent/coal ratio on liquefaction conversion.

The advantages of using the tubing bomb autoclave as a research tool are: small sample requirement, rapid results, low cost per data point, and well-defined operating conditions. However, because the tubing bomb autoclave is a batch system, continuous flow units are needed to address steady state process concerns.

The important tubing bomb studies performed during Phases IIIB-V of the EDS program are summarized below and reported in Table 4-1.

4.1.1 Correlations of Coal Conversion with Coal Properties

Stepwise multiple regression analysis of tubing bomb autoclave conversion data was performed to develop a correlation expressing liquefaction

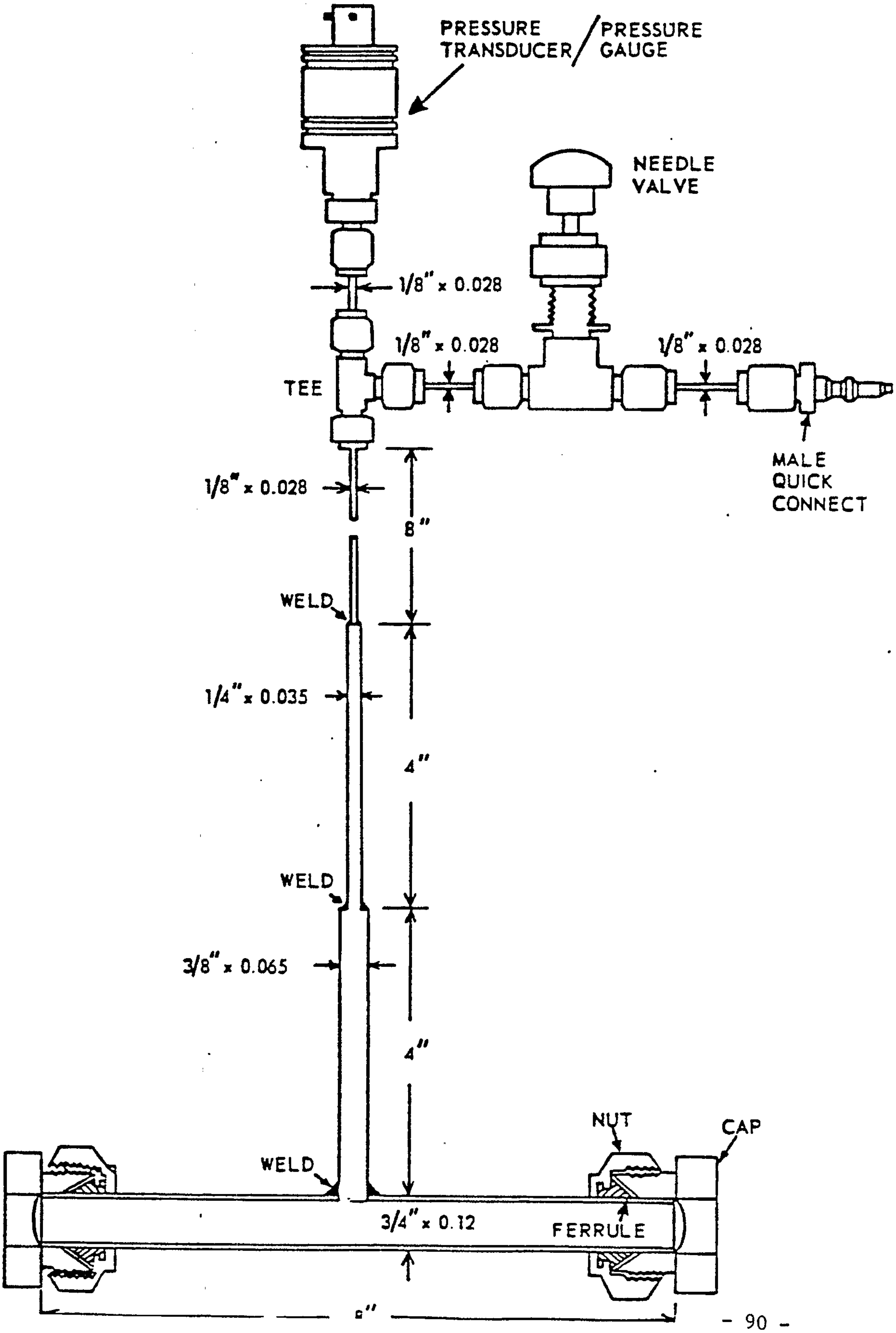
Table 4-1

Bench Unit Experimental Programs Conducted During the EDS Program

Unit	Study	References
• Tubing Bomb Autoclaves	+ Correlation of Coal Conversion with Coal Properties	3Q78 [FE-2893-21] 4Q78 [FE-2893-25] AN78-79 [FE-2893-35] 3Q79 [FE-2893-41] 1Q80 [FE-2893-49] AN79-80 [FE-2893-53]
	+ Liquefaction Conversion of Wyoming Coal Bottoms from CLPP	4Q78 [FE-2893-25] AN78-79 [FE-2893-29]
	+ Tubing Bomb Investigation of Bottoms Recycle of Sponsor Coals	1Q79 [FE-2893-29] AN78-79 [FE-2893-35] 3Q79 [FE-2893-41]
	+ Heavy Liquid Recycle-Tubing Bomb Simulation	4Q79 [FE-2893-45] 1Q80 [FE-2893-49] AN79-80 [FE-2893-53]
	+ Solvent Cracking Studies	CA 3Q79 [FE-2893-41] CA AN79-80 [FE-2893-53]
	+ Coal Screening and Process Variable Studies;	
	- Illinois No. 6 (Burning Star No. 2 mine) Bituminous Coal	3Q78 [FE-2893-21] 4Q78 [FE-2893-25] 1Q79 [FE-2893-29] AN78-79 [FE-2893-35] 3Q81 [FE-2893-78]
	- Pittsburgh (Ireland Mine) Bituminous Coal	1Q79 [FE-2893-29] AN78-79 [FE-2893-35] 3Q79 [FE-2893-41] AN79-80 [FE-2893-53]
	- Australian (Wandoan) Black Coal	4Q78 [FE-2893-25] AN78-79 [FE-2893-35] 3Q80 [FE-2893-57] AN80-81 [FE-2893-74]
	- Illinois No. 6 (Monterey No. 1 Mine) Bituminous Coal	4Q79 [FE-2893-45] AN79-80 [FE-2893-53]
	- Wyoming (Wyodak Mine) Subbituminous Coal	4Q79 [FE-2893-45] AN79-80 [FE-2893-53]
	- Texas (Big Brown Mine) Lignite	1Q80 [FE-2893-49] AN79-80 [FE-2893-53]
	- Texas (Martin Lake Mine) Lignite	4Q81 [FE-2893-83] 1Q82 [FE-2893-89] AN81-82 [FE-2893-93]
	+ Tubing Bomb Evaluation of Solvent Quality Effects on Bottoms Recycle Coal Conversion	3Q80 [FE-2893-57] 4Q80 [FE-2893-64] AN80-81 [FE-2893-74]
	+ Reactor Hydrodynamic Support Study	3Q81 [FE-2893-78] 4Q80 [FE-2893-83] AN81-82 [FE-2893-93]
	+ Gas-Swept Mill Coal Drying Study	3Q80 [FE-2893-57] 1Q81 [FE-2893-70] AN80-81 [FE-2893-74] 1Q82 [FE-2893-89] AN81-82 [FE-2893-93] 3Q82 [FE-2893-99] 4Q82 [FE-2893-103]
	+ Slurry Drying Study	4Q82 [FE-2893-103]
o OTCLU	+ Validation of OTCLU Yields	AN78-79 [FE-2893-35] 3Q79 [FE-2893-41] 4Q79 [FE-2893-45] AN79-80 [FE-2893-53]
	+ Operability Study Wyoming Coal	3Q77 [FE-2893-3] 4Q77 [FE-2893-7] 1Q78 [FE-2893-12] AN77-78 [FE-2893-17] 3Q78 [FE-2893-21] 4Q78 [FE-2893-25] 1Q79 [FE-2893-29] AN78-79 [FE-2893-35]
	+ High Pressure, Coal-only Study on OTCLU	4Q79 [FE-2893-45] 1Q80 [FE-2893-49] AN79-80 [FE-2893-53] 3Q80 [FE-2893-57] 4Q80 [FE-2893-64] AN80-81 [FE-2893-74]
	+ Solvent Cracking Study	CA AN79-80 [FE-2893-53]
	+ Liquefaction of Neat Bottoms	1Q80 [FE-2893-49] AN79-80 [FE-2893-53] 3Q80 [FE-2893-57] AN80-81 [FE-2893-74]
	+ Vacuum Gas Oil (VGO) Conversion Study	3Q80 [FE-2893-57]

FIGURE 4-1

SCHEMATIC DIAGRAM OF TUBING BOMB AUTOCLAVE



conversion and yields as functions of measurable coal properties. Independent variables included ultimate, proximate, and petrographic analyses. The dependent variable was tubing bomb conversion (dry, ash-free) at 840°F/40 minutes/1500 psig hydrogen partial pressure and 1.6/1 tetralin-to-coal ratio. A total of 16 coals ranging from bituminous to lignitic were included in the data base (4Q78 [FE-2893-25]).

The following observations were made based on the correlations:

+ percent volatile matter was the most significant variable
  - Volatile matter was highly correlated with percent carbon, atomic H/C ratio, and percent exinite.
+ total sulfur content, in addition to volatile matter, was significant in correlating conversion.
+ C_1-C_3 gas make correlated highly with H/C ratio and volatile matter content.
+ total gas make correlated well with percent carbon, percent oxygen, and atomic O/C ratio.

Tubing bomb conversions for coals not in the data base were predicted well by the derived statistical correlation (1Q80 [FE-2893-49], AN79-80 [FE-2893-53]). Tubing bomb conversion trends were in reasonable agreement with continuous pilot plant results.

4.1.2 Liquefaction Conversion of Wyoming Coal Bottoms from CLPP

This study was conducted to test the reactivity of Wyoming (Wyodak mine) coal bottoms during bottoms recycle. Tubing bomb liquefaction experiments were conducted with two CLPP bottoms samples liquefied separately in the absence of coal. The results indicated that significant additional liquid yield could be achieved by recycling Wyoming bottoms (4Q78 [FE-2893-25]). Subsequent to this study, all program and sponsor coals were investigated for bottoms recycle potential.

4.1.3 Tubing Bomb Investigation of Bottoms Recycle of Sponsor Coals

A tubing bomb study to screen sponsor coals for possible yield incentives for operating the EDS process in a bottoms recycle mode was conducted. The primary objective of the study was to estimate the extent to which the liquefaction residues for the sponsor coals could be converted. The seven program and sponsor coals employed in this study were Texas (Big Brown mine) lignite, Wyoming (Wyodak mine) subbituminous coal, Australian (Wandoan) black coal, two Illinois No. 6 (Monterey No. 1 mine and Burning Star No. 2 mine) and two Pittsburgh seam (Ireland and Arkwright mines) bituminous coals. Liquefaction residues employed in the study had been generated on RCLU at operating conditions of 840°F and 40 minutes nominal residence time during preliminary screening studies on each of the coals.

During the initial part of this study, neat bottoms were liquefied at a temperature of 840°F for 40 minutes reaction time, with a 4 wt% hydrogen treat rate and 1.6 solvent (hydrogenated MPSS)-to-bottoms ratio. Cyclohexane conversions for all the bituminous coals and Wandoan coal were roughly equivalent to 22 wt%. Wyodak bottoms yielded only 16 wt% conversion, and Big Brown bottoms gave only 11 wt% conversion.

For the remainder of the study, mixtures of coal plus bottoms in a 2/1 ratio were liquefied at 840°F and 40 minutes reaction time for solvent-to-solids ratios of 1.6 and 1.07. The results indicated that the conversion and gas yield for Wandoan and the bituminous coals were independent of the solvent-to-solids ratio in the range studied. On the other hand, Wyodak and Big Brown samples showed a strong conversion synergism of about 10 wt% (on bottoms) between coal and bottoms. In addition, the synergism for Wyodak coal plus bottoms was strongly affected by the solvent-to-solids ratio (AN78-79 [FE-2893-35]).

4.1.4 Tubing Bomb Simulation of Heavy Liquid Recycle

The recycle of heavy liquid products to liquefaction provides potential for process and product slate flexibility. Two such alternatives, vacuum gas oil (VGO) recycle and coker liquid recycle, were screened in tubing bombs.

+ Vacuum Gas Oil (VGO) Recycle

Tubing bomb liquefaction experiments were carried out with VGO (CLPP A-2) derived from CLPP operations with Wyodak coal. Various temperature/residence time combinations were tested in tubing bombs in the presence of 2000 psig hydrogen partial pressure. No donor solvent was added. Gas chromatographic distillation on feed and products revealed that heavy liquid fractions (700°F$^+$) were converted into lighter fractions (400°F$^-$ and 400-700°F) as liquefaction severity increased. Gas make, comprised of C_1-C_3 hydrocarbons, CO_2 and H_2S, also increased (1Q80 [FE-2893-49]).

The results indicated that 700-1000°F VGO could be recycled to extinction in liquefaction, yielding additional 700°F$^-$ liquid and gas products. This study was extended in the Once-Through Coal Liquefaction Unit (OTCLU) and is summarized separately in Section 4.2.

+ Coker Liquid Recycle

Coker liquids derived from coking operations at two different coking temperatures (RV-60°F and RV+70°F) were tested. This study complemented an earlier brief tubing bomb study and a more extensive RCLU study. The results of the earlier studies are reported in the January, 1976-June, 1977, EDS Final Technical Progress Report (FE-2353-20).

The purpose of this study was to see if the temperature at which the coker liquids were produced had an effect on their conversion under liquefaction conditions. Reaction temperature was kept constant while the initial hydrogen pressure, solvent-to-coker liquids ratio and residence time were varied.

Coker liquids derived at low coking temperature were more reactive in liquefaction that the high temperature derived coker liquids. However, since low temperature coking produces less coker liquids, the overall impact on net 1000°F⁻ conversion products was unchanged. Assuming no interactions existed between coal and coker liquids conversion in liquefaction, recycle of coker liquids to liquefaction resulted in approximately 3-4 wt% extra conversion to 1000°F⁻ liquids based on dry feed coal. This increased conversion estimate was independent of the coking temperature.

Coker liquids conversion increased with increased liquefaction severity. The solvent-to-coker liquids ratio was the most sensitive variable. However, on a dry coal feed basis, this sensitivity resulted in only small additional conversion incentive (~1 wt% on coal) (1Q82 [FE-2893-49], AN79-80 [FE-2893-53]).

4.1.5 Solvent Cracking Study in Tubing Bombs

A series of tubing bomb experiments was conducted to determine the extent to which solvent cracking occurs under liquefaction conditions. The solvent samples used were hydrogenated multipass spent solvent (MPSS) from CLPP, a saturates-rich raffinate, and an aromatics-rich extract from sulfolane extraction of the MPSS. Each of these samples was run at four different sets of liquefaction conditions in order to assess the effect of residence time and temperature on cracking.

Results show that gas make increases with liquefaction severity and is greatest for the raffinate (which has the highest saturates content). The results indicated that the cracking of hydroaromatics, as well as saturates, is significant. Furthermore, hydroaromatics decreased with increasing severity, indicating that the loss of solvent quality is due both to dehydrogenation and cracking (3Q79 [FE-2893-41]). Solvent cracking characteristics were identical with and without the presence of mineral matter (AN79-80 [FE-2893-53]).

4.1.6 Coal Screening and Process Variable Studies

Coal screening and process variable studies in tubing bombs were normally performed prior to testing a coal in the Recycle Coal Liquefaction Unit (RCLU). The scope and depth of the studies varied from coal to coal, but the primary purpose was to provide guidance for subsequent pilot plant operations.

Two level factorial designs were used to screen the effects of reaction temperature, reaction time, hydrogen partial pressure, solvent-to-coal ratio and bottoms-to-coal ratio. Process variable studies in the coal-only operating mode were conducted for Illinois No. 6 (Burning Star No. 2 mine) coal (4Q78 [FE-2893-25]), AN78-79 [FE-2893-35], and Pittsburgh (Ireland mine) coal (4Q79 [FE-2893-45]). Process variable studies incorporating bottoms-to-coal ratio included Australian (Wandoan mine) coal (4Q78 [FE-2893-25], 3Q80 [FE-2893-57]), Illinois No. 6 (Monterey No. 1 mine) coal (3Q79 [FE-2893-41]), Wyoming (Wyodak mine) coal (4Q79 [FE-2893-45]), Texas (Big Brown mine) lignite (1Q80 [FE-2893-49]), AN79-80 [FE-2893-53]) and Texas (Martin Lake mine) lignite (1Q82 [FE-2893-89], AN81-82 [FE-2893-93]). Results indicated that significantly improved liquid yields were achievable with bottoms recycle compared to coal-only operations.

4.1.7 Tubing Bomb Evaluation of Solvent Quality Effects on Bottoms Recycle Coal Conversion

A tubing bomb study was conducted to investigate the effects of solvent quality index (SQI) on coal conversion with simulated bottoms recycle. The three coals employed in this study were Illinois No. 6 (Monterey No. 1 mine), Wyoming (Wyodak mine), and Texas (Big Brown mine) lignite. All experiments were done at constant temperature, time, and solvent-to-coal-to bottoms. Two levels of pressure were examined.

Conversion response plateaued at higher SQI with bottoms recycle liquefaction than with once-through liquefaction (4Q80 [FE-2893-64], AN80-81 [FE-2893-74]). The results were used in planning future RCLU solvent quality investigations.

4.1.8 Reactor Hydrodynamic Support Study

A tubing bomb support study for selection of a suitable liquid phase radioactive tracer to be used in characterizing liquefaction reactor hydrodynamics was conducted. The criteria used in the tubing bomb study for selecting a liquid phase tracer were:

- compatability with a coal, solvent, and hydrogen atmosphere at liquefaction conditions.
- ability to disperse in coal liquids.
- distribution in the gas, liquid, and solid phases at liquefaction conditions.

Using these criteria, several organic and inorganic compounds were tested. Early tests identified 1-bromoadamantane and 9-bromophenanthrene as possible candidates for testing in RCLU (3Q81 [FE-2893-78], 4Q81 [FE-2893-83]). Further testing identified tetraphenylgermanium as the best candidate for the soluble radioactive liquid phase tracer (AN81-82 [FE-2893-93]).

4.1.9 Gas Swept Mill Coal Drying Study

A tubing bomb investigation of coal drying effects in the gas-swept mill (GSM) was conducted due to unexpected low coal conversions observed in ECLP. Early tubing bomb results showed no conversion debit due to GSM processing for Illinois No. 6 (Monterey No. 1 mine) coal (3Q80 [FE-2893-57]). However, tests in tubing bombs and RCLU identified a 5 wt% debit for a sample of GSM processed Wyoming (Wyodak mine) coal (1Q82 [FE-2893-89]), prompting this investigation.

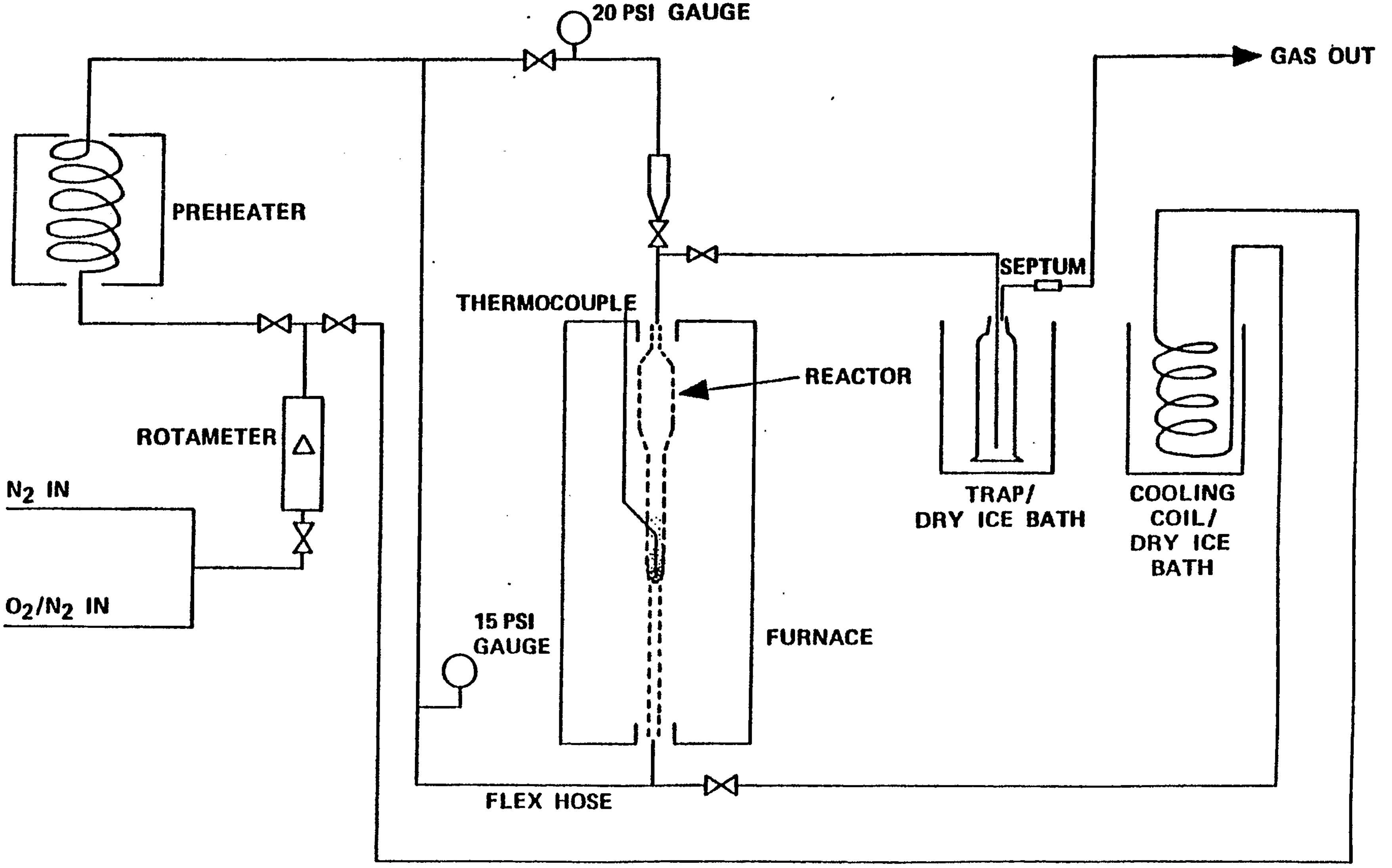
Coal was prepared for liquefaction in the GSM, a process which first crushes coal and then dries the crushed coal particles in a hot flue gas. This drying procedure was shown to be detrimental to coal liquefaction yields due to the high temperature (often > 600°F) and large oxygen concentration (8-12 mole %) of the flue gas. The bench program utilized a small fluidizing unit (SFU), shown in Figure 4-2, to determine the controlling variables. Bench tests revealed that extremely severe drying conditions could produce liquefaction conversion liquid yield debits as large as 20 wt% for both Wyodak subbituminous coal and Martin Lake lignite. However, ECLP drying conditions typically produced debits in the 2 to 5 weight percent range. The debits resulted from pyrolysis and oxidation of the coal, which occurred while drying coal in the mill. With the ECLP operating scheme, debits for Wyodak coal were initiated at 575°F inlet gas temperature and debits for Martin Lake lignite were initiated at 620°F inlet gas temperature (3Q82 [FE-2893-99]).

Conventional analytical tests failed to detect any chemical changes in the dried coal although the mild pyrolysis/oxidation induced liquefaction debits as large as 10-12%. Coal-only liquefaction in tubing bombs proved to be the most sensitive test for detecting the effects of mild oxidation/pyrolysis. This result had significant impact on the evaluation procedures used to determine the effects of coal preparation processes on subsequent liquefaction.

A safe operating range for the gas-swept mill was determined that would avoid the liquefaction debits. Control strategy centered on reducing the coal feed rate, but several design options are available for a commercial unit that would not restrict the coal feed rate. Furthermore, bottoms-recycle liquefaction significantly reduced the debits due to GSM processing for Martin Lake lignite, but had little effect on Wyodak coal debits (4Q82 [FE-2893-103]). The coal conversion debits associated with ECLP gas-swept mill drying are not a concern for a commerical process.

FIGURE 4-2

SCHEMATIC OF SFU APPARATUS



4.1.10 Slurry Drying Study

Due to conversion debits observed with gas-swept mill coal drying, a short experimental program was conducted to determine whether similar debits could occur during slurry drying. Bench tests were performed on Wyoming (Wyodak mine) coal and Texas (Martin Lake mine) lignite. Both coals showed no subsequent conversion debits after being slurry dried at nominal ECLP conditions at 3.4 SQI. Furthermore, no deleterious effect was observed when Wyodak coal was slurry dried at 2.0 SQI (4Q82 [FE-2893-103]). Based on the laboratory results from tubing bomb experiments, slurry drying at nominal ECLP conditions poses no commercial concerns.

4.2 Design and Operation of 15 pound-per-day Once-Through Coal Liquefaction Unit (OTCLU)

A 15 pound/day Once-Through Coal Liquefaction Unit (OTCLU) was designed and built to fill a gap between small batch reactors and the fully integrated pilot unit facilities. The unit is low-cost, easy to operate, and large enough to produce products for distillation, but small enough to conduct a complete screening study on less than one barrel of coal.

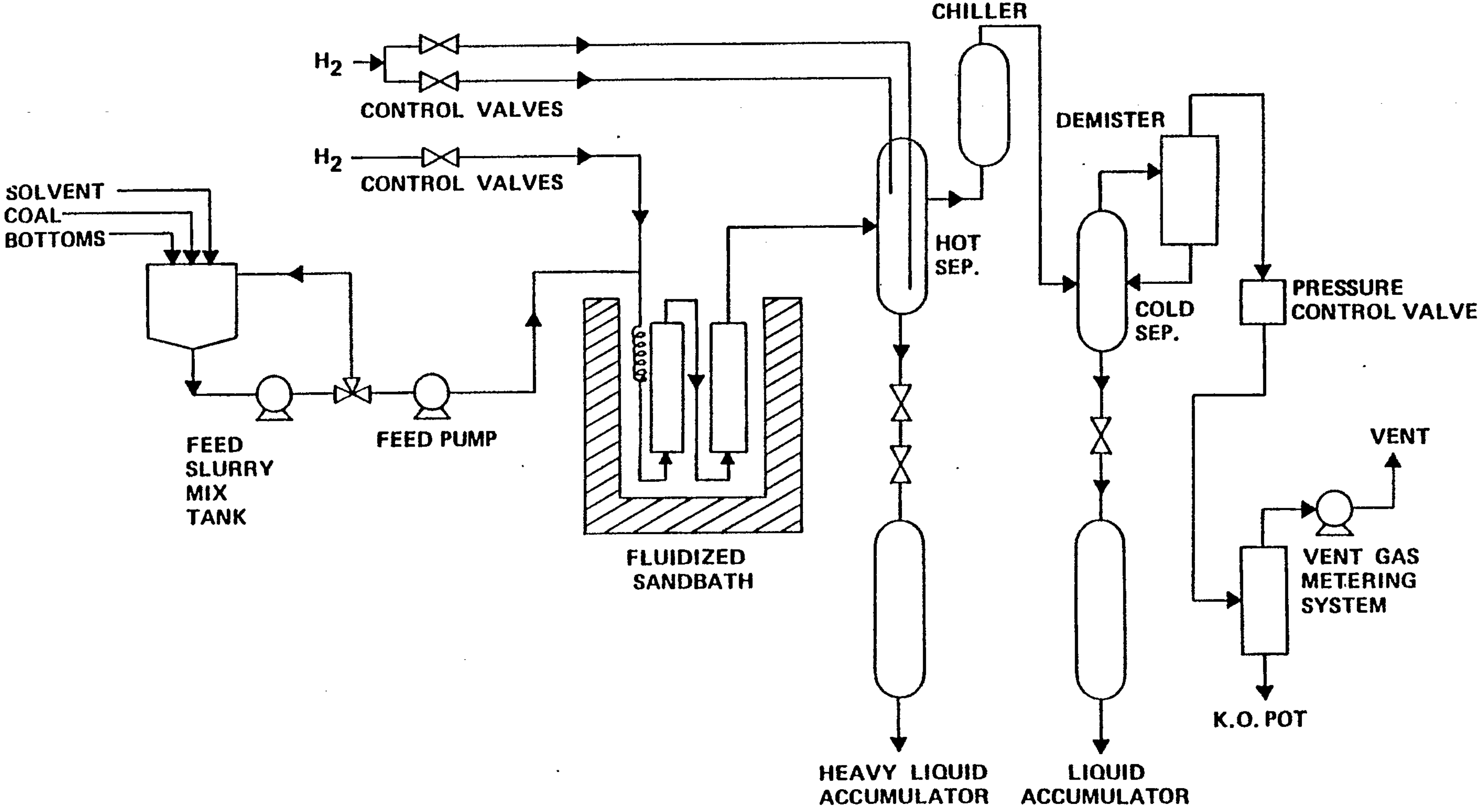
A schematic flow plan of the OTCLU is given in Figure 4-3. Feed slurry and hydrogen are metered through separate preheaters to the up-flow liquefaction reactors which are contained in a fluidized sandbath. The reactors are designed to operate at temperatures up to 1000°F and pressures up to 3000 psig. The total gas, liquid and solid products exit the liquefaction reactors into two high pressure accumulators where the solids and liquids are collected for each yield period. The gas continuously leaves the accumulator through a pressure let-down valve and is measured in a dry test meter.

Several validation experiments were carried out with the OTCLU using various coals and at different operating conditions (AN78-79 FE-2893-35, 3Q79 FE-2893-41, 4Q79 FE-2893-45, AN79-80 FE-2893-53). Objectives of these experiments were to check the reproducibility of the unit and to test the operability limitations of the unit. Coals used for these experiments were Illinois No. 6 (Monterey Mine No. 1), Pittsburgh seam (Ireland mine) bituminous coal, Wyoming (Wyodak mine) subbituminous coal and Texas (Big Brown) lignite.

The results indicated that the unit generates reproducible data, and that the yields obtained were in reasonable agreement with the 50 pound/day Recycle Coal Liquefaction Unit (RCLU-1) at comparable conditions. Operational problems were experienced only in once-through coal operations with Big Brown lignite, due to the high viscosity of the product slurry.

FIGURE 4-3

OTCLU FLOW DIAGRAM



4.2.1 Operability Study with Wyoming Coal

During the liquefaction of low rank coals, such as Wyoming (Wyodak mine) coal, the calcium contained in the form of salts of humid acids decomposes to form calcium carbonate ($CaCO_3$). This calcium carbonate accumulates in the liquefaction reactor as wall scale and free-flowing aggregates (oolites). The OTCLU was used to test proposed solutions to the calcium carbonate deposition problems. These solutions can be classified as mechanical or chemical solutions. Mechanical solutions are those in which the process conditions, process configuration or mechanical equipment are 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.

During EDS 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 (3Q77 [FE-2893-3]). Pretreatment of moist Wyodak coal with SO_2 gas was also found to be an effective means of converting calcium in coal to calcium salts which are stable during liquefaction (4Q77 [FE-2893-7]). Calcium carbonate formation in liquefaction was prevented by another means, e.g., ion-exchange with various metal salts. Ferrous sulfate, ferric sulfate, and sodium carbonate were all effective in eliminating $CaCO_3$ wall scale and agglomerates (AN77-78 [FE-2893-17]). No mechanical solutions were found.

4.2.2 High Pressure, Coal-only Study

The purpose of this study was to generate coal-only liquefaction yields at high pressure to be used for developing yield correlations for bottoms recycle operations. Experiments were conducted with Illinois No. 6 (Monterey No. 1 mine), Pittsburgh seam (Ireland mine), Wyoming (Wyodak mine), and Texas (Big Brown mine) coals. Considerable data scatter and operational problems were encountered during this study, especially with the lower rank coals, Wyodak and Big Brown (1Q80 [FE-2893-49], AN79-80 [FE-2893-53], 3Q80 [FE-2893-57]). Results showed practically no effect of pressure in the range 2000-2500 psig for Monterey No. 1 and Wyodak coals (1Q80 [FE-2893-49], AN79-80 [FE-2893-53]), and only a slight effect on Big Brown lignite (AN80-81 [FE-2893-74]). OTCLU experienced lower conversions than RCLU for Ireland coal (AN79-80 [FE-2893-53]). The OTCLU results at high pressure were implemented into the EDS coal liquefaction model.

4.2.3 Solvent Cracking Study on the Once-Through Coal Liquefaction Unit (OTCLU)

A process variable study analyzing solvent cracking characteristics was carried out on OTCLU with unhydrotreated MPSS from CLPP and hydrogenated raw creosote oil (HCO). The results supported the earlier conclusions based on a similar tubing bomb solvent cracking study.

The main conclusions from this study were (CA AN79-80):

- As the temperature was increased from 750 to 950°F, naphtha and gas make increased, whereas saturates content and solvent quality decreased. Both CLPP MPSS and HCO have essentially the same temperature response regarding gas and naphtha makes and reduction of solvent quality.

- The increase in gas and naphtha makes and decrease in saturates and donor hydrogen contents were more dramatic at temperatures above 850°F and were practically insignificantly at 750°F.

- For the same reaction temperature, an increase in pressure from 1500 to 2500 psig did not result in any significant effect on either gas and naphtha make, or saturate depletion.

- The percentage change in concentration of specific hydroaromatics and aromatics components also demonstrated strong dependence on temperature at 850°F and above, and practically no dependence on pressure from 1500 to 2500 psig.

4.2.4 Liquefaction of Neat Bottoms

In support of continuous bottoms recycle operations, and to aid in developing yield correlations for bottoms recycle, an OTCLU process variable study was conducted on the liquefaction of neat bottoms from CLPP operations on Illinois No. 6 (Monterey No. 1 mine) coal. Results indicated that liquid make (C_4-1000°F) increased as the pressure increased for recycled bottoms, but not for single-pass bottoms (AN79-80 [FE-2893-53]). A similar study for Texas lignite (Big Brown mine) bottoms was aborted due to operational difficulties (3Q80 [FE-2893-57], AN80-81 [FE-2893-74]).

4.2.5 Vacuum Gas Oil (VGO) Conversion Study

A process variable study was carried out on OTCLU to assess vacuum gas oil (VGO) conversion trends at various process conditions (3Q80 [FE-2893-57]). The VGO sample was obtained from CLPP during Illinois No. 6 (Monterey No. 1 mine) coal operations.

Several conclusions have been drawn based on the results:

- There is a net conversion of 700°F$^+$ fractions in VGO to a lighter distillate fraction (700°F$^-$). This result is directionally comparable with an earlier VGO conversion study conducted in tubing bombs (see Section 4.1).

- The 1000°F conversion is independent of pressure for 1500 and 2500 psig but is dependent on temperature/residence time (°F/minutes) combinations. The 1000°F conversions are equal at 840/40 and 880/25, but is significantly lower at 800/100.

Hydrogen pressure has a positive effect on lighter liquid (IBP-700°F) yield. This indicates that VGO undergoes hydrocracking to form lighter products.

Gas yield was very small (~1 wt% on VGO) for all process variable combinations, except at 880°F/25 minutes/1500 psig and 880°F/25 minutes/2500 psig. The gas make at these two conditions was about 4 wt% on VGO.

4.3 EDS Coal Liquefaction Model Development

The conversion of coal into useful hydrocarbon products through hydrogenation reactions is a very complicated system. The hydrogen comes either directly from molecular hydrogen or indirectly through a "donor" solvent. In the EDS process, spent solvent is recycled through a solvent hydrotreating unit to upgrade the solvent quality and restore its donor capability. Both coal and the donor solvent are very complex and consist of many components. The process is even more complicated with the incorporation of bottoms recycle operations. Therefore, it became important to develop a means of correlating pilot plant yields for both coal-only and bottoms recycle operations. To accomplish this required that the contributions of individual reactants to the product slate be identified, and the effects of process variables on the contributions be determined.

Table 4-2 summarizes the EDS liquefaction modeling activities during EDS Phases IIIB-V. The earliest model developed was an empirical model which correlated liquefaction yields with coal conversion. In an effort to more fully understand the chemistry of coal conversion, a fundamental model was developed. Finally, a semi-empirical model was developed which can accurately predict the product slate at processing conditions within the data base for RCLU and CLPP.

An integrated liquefaction yield model was then developed by linking the semi-empirical model and the solvent hydrogenation model to predict steady state solvent quality and liquefaction yields. With the accumulated learnings from this research and development program, the semi-empirical model was successfully upgraded to a generic model which will be used in the EDS licensing package to provide predictions of EDS liquefaction yields for any coal with known properties.

4.3.1 Empirical Coal Liquefaction Model

The mechanisms of the donor solvent liquefaction reactions were formulated into an empirical kinetic model. The model involved the use of an empirical classification of reactive coal types and lumped kinetic parameters. Coal contains several types of chemical bonds and thus exhibits a wide distribution of reactivities. It is convenient to lump portions of the coal into reactive types. In this model, the feed coal is lumped into four classes according to reactivity.

Table 4-2

EDS COAL LIQUEFACTION MODELING ACTIVITY

Model	Description	References
1. Empirical Coal liquefaction Model	Coal conversion is function of Temperature, Reaction time, and hydrogen pressure. Component yields are expressed in terms of coal conversion. Developed for coal-only operations for Illinois and Wyoming coals.	FE-2353-2 FE-2893-17
2. Fundamental Coal liquefaction Model	Coal is separated into chemically similar groups. Each group is treated as a pure substance. Developed for coal-only operations.	FE-2353-9 FE-2353-17 FE-2353-35
3. Solvent hydrogenation Model	Solvent is separated into groups of chemical homologs and each homolog series has its unique rate constant.	FE-2353-6 FE-2353-9 FE-2893-17 FE-2893-35
4. Semi-Empirical Coal liquefaction Model	Coal conversion and component yields are all correlated as functions of processing conditions following a proposed reaction scheme. Applies to both coal-only and bottoms recycle operations.	FE-2893-53 FE-2893-74 FE-2893-93
5. Integrated EDS liquefaction Model	The solvent hydrogenation model and the semi-empirical model are combined through a saturates balance routine. This model produces liquefaction yields and solvent quality simultaneously.	FE-2893-93
6. EDS Generic Coal Liquefaction Model	Coal conversion and yields are functions of coal properties and processing conditions. Formulation similar to the semi-empirical model.	FE-2893-103

This model predicts 1000°F+ liquefaction yields for a given liquefaction reactor temperature, nominal residence time and total pressure in the reactor. The yields of the other components: C_1-C_3 gas, H_2, H_2O, chemical gases, (H_2S, NH_3, CO, CO_2) C_4-400°F liquids, 400-700°F liquids, 700-1000°F liquids are obtained by fitting quadratic equations based on conversion.

These kinetic parameters were calculated to insure a best fit of the RCLU pilot unit once-through liquefaction data for Illinois and Wyoming coals, and thus reflect RCLU measured yields of solids, liquids, and gases. This model provides a means for extrapolating the data to other conditions, as well as a means for optimizing liquefaction conditions. The data cover a wide range of liquefaction conditions: 700-900°F, 1500-2500 psig, and a ten-fold variation in residence time.

This model gave an acceptable correlation of the Recycle Coal Liquefaction Unit (RCLU) once-through data and was incorporated into the Exxon Donor Solvent (EDS) Process Model to provide yield information for coal-only operations. Also, it can be used to optimize liquefaction conditions. A disadvantage of the model was that it did not predict the effects of process variables on product slate distribution. Further details of this model have been presented in the January-June, 1976, EDS Quarterly Technical Progress Report (FE-2353-2) and the July 1977-June 1978, EDS Annual Technical Progress Report (FE-2893-17).

4.3.2 Fundamental Coal Liquefaction Model

To gain a better understanding of the chemistry of coal liquefaction and to predict more precisely the product distribution, a more fundamental coal liquefaction model was developed. Because coal and the liquefaction donor solvent are both very complex systems made up of many components, it is impossible to write down each chemical reaction explicitly. For this reason, classes of chemical species and reactions are lumped together and represented by one equation and one rate constant. The chemical species and reactions chosen are based on the known chemistry of coal, coal liquids, and donor solvent. In this model, eight reactions with hydrogenation, polymerization, and hydrocracking of nine chemical species were formulated.

From these reactions, rate equations are derived and integrated numerically to give concentrations of the chemical species as a function of temperature, reaction time and solvent-to-coal ratio. The model was developed based on RCLU once-through liquefaction data for Illinois coal, and gave acceptable fit to the data base. This model agrees qualitively with RCLU and CLPP pilot plant data not included in the data base, especially in the shape of the net solvent fraction (400-700°F) and heavy liquid yield curves. This model was also used to successfully predict results of low pressure (1500 psig) bottoms recycle simulations on RCLU.

The major disadvantage of the model was the extent of analytical description required for the data base, making extension of the model to other coals very costly. Details of this model are presented in the January-December 1976, July 1977-June 1978, and July 1978-June 1979 EDS Annual Technical Progress Report (FE-2353-9, FE-2893-17, and FE-2893-35).

4.3.3 Semi-Empirical Coal Liquefaction Model

A semi-empirical coal liquefaction yield model was developed which can be used to predict coal conversion and liquefaction yields with coal-only and bottoms recycle operations. The objectives of this activity were to guide process development, facilitate data interpretation, and develop a tool for evaluation of EDS project coals. This correlation predicts conversion and yields as functions of 7 key process variables (pressure, temperature, nominal residence time, solvent-to-coal ratio, bottoms-to-coal ratio, solvent quality index, and hydrogen treat rate) and identifies contributions of the individual reactants to the overall product slate. Data for these correlations were produced on the 75 pounds-per-day Recycle Coal Liquefaction Units (RCLU-1 and RCLU-2) and the 1 ton-per-day Coal Liquefaction Pilot Plant (CLPP). The data base covers a wide range of processing conditions.

The formulation of this model is based on the following concepts:

- All reactions are single step reactions and follow first order, irreversible kinetics.
- Coal can be separated into five distinct fractions.
- Solvent hydrocracks in the liquefaction reactor to form C_1-C_3 gas and C_4-400°F naphtha.

Predictions of coal conversion, hydrogen consumption, C_1-C_3 gas yield, C_4-400°F naphtha yield, C_1-1000°F total hydrocarbon yield, 700-1000°F VGO yield and water make are directly obtained from correlations, while 400-700°F distillate yield is calculated by difference in order to satisfy material balance criteria. The correlation coefficients (R^2) for the key component yields, i.e., coal conversion, C_1-C_3 gas yields, C_4-1000°F total hydrocarbon liquid yields, and hydrogen consumption, are all high (>.90). Less satisfactory correlations were observed for the chemical gas and VGO yields. This model has been used extensively to provide operating guidance on RCLU, CLPP, and ECLP operations.

Details of this model development are summarized in the July 1980-June 1981 and July 1981-June 1982 Annual Technical Progress Reports (FE-2893-74 and FE-2893-93).

4.3.4 Solvent Hydrogenation Model

A model capable of accounting for the possible variations in solvent composition has been developed for the EDS process based on a set of proposed reaction paths of individual molecular species. This model assumes that all reactions follow first order kinetics.

The effects of catalyst deactivation are also incorporated. This model was developed for both Co/Mo-10 and Ni/Mo-10 catalysts at 1500 psig hydrogen pressure. Heteroatoms removal reactions are also included for the Ni/Mo-10 model.

4.3.5 Integrated EDS Coal Liquefaction Yield Model

Although liquefaction and solvent hydrotreating are two independent unit operations, they are intimately connected via the recycling of process solvent. The performance of either unit depends not only on its own processing conditions, but also on the conditions of the other unit. For this reason, an integrated model was developed which links the liquefaction and solvent hydrogenation models together and predicts the steady state process yields and solvent quality for a given set of operating conditions.

The link between the liquefaction model and the solvent hydrogenation model is a saturates balance routine which equates the saturates depletion rate in the liquefaction reactor to the saturates production rate in the solvent hydrotreater. The saturates cracking rates were derived from tubing bomb autoclave tests 4Q82 (FE-2893-103). This cracking algorithm proved to be valid, showing excellent agreement with results from an extended 21 yield period RCLU operations (YP's 489-509).

This model has been successfully used to explain the observed high saturates and low quality solvent during the initial operations of ECLP. Effective solutions were suggested by employing this model in planning ECLP operations. In addition, this model has been used to establish an operating window for each of the project coals in terms of the maximum attainable solvent quality (FE-2893-93).

The integrated EDS coal liquefaction model was also used to simulate the hydrogen partial pressure profile in the ECLP liquefaction reactors to determine the operating temperature limits for Monterey coal bottoms recycle operations. Results indicated that the hydrogen pressure drops rapidly in the first reactor and then gradually decreases to its final value through the successive reactors. This information has been used by ECLP together with vapor-liquid equilibrium (VLE) data and the Nelson curve (API publications No. 941, Operating Limits for Steels in Hydrogen Services) to establish an upper bound of liquefaction temperature for ECLP operation (FE-2893-93).

Details of the integrated yield model were presented in the July 1981-June 1982 EDS Annual Technical Progress Report (FE-2893-93).

4.3.6 EDS Generic Coal Liquefaction Model

A generic coal yield model for EDS coal liquefaction process has been developed which can be used to predict the yield responses for any new coal with rank in the range from lignitic to high volatile bituminous. The data base used for this model development is mainly from 6 EDS program coals tested in RCLU: Illinois No. 6 bituminous coal (Monterey No. 1 mine); West Virginia bituminous coal (Ireland mine); Illinois No. 6 bituminous coal (Burning Star No. 2 mine); Wyoming sub-bituminous coal (Wyodak mine); Australian black coal (Wandoan mine); and Texas lignite (Big Brown mine). The data base contains 172 processing conditions with 711 yield periods and covers a wide range of processing conditions varying from low severities at coal-only operations to high severities with bottoms recycle operations.

The basic formulations used in this generic model development, except for coal conversion, are identical to the ones used in the semi-empirical yield model development. However, the coal fractions and rate constants are set as functions of coal properties, including the elemental, proximate, and petrographic analysis. A new formulation has been developed for the coal conversion correlation, which treats the bottoms as residues from coal and assumes that they still retain the characteristics of their precursors.

This model fits the experimental results well with high correlation coefficients and low standard deviations. The validity of this model has been checked using the RCLU results on Texas (Martin Lake mine) lignite. This model will be used in the licensing activities for the EDS coal liquefaction process. With this model, a complete product slate at preferred processing conditions can be predicted for a given coal with known properties. This information then can be used in preliminary economic screening studies to determine if this coal merits further evaluation.

Details of this model are summarized in the October-December 1982 EDS Quarterly Technical Progress Report (FE-2893-103).

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

LIQUEFACTION PROCESS RESEARCH

5. Product Quality Studies

5.1 Summary of Research Results

The specific objectives of the EDS Product Quality Program during Phases IIIB-V were to conduct product quality studies, define potential end-use applications for coal liquids, and evaluate the properties of these coal liquids relative to requirements of these potential applications. The ultimate product of these studies is an identification of the most attractive markets for coal liquids. The process can also produce LPG (C_3/C_4) and high Btu gas (C_1/C_2) as co-products. However, these C_4-minus co-products are adequately characterized by their molecular compositions and therefore were not included as part of the Product Quality Program.

The EDS coal liquefaction process exhibits broad feed coal and product slate flexibility. It handles a wide range of coals (bituminous, subbituminous, and lignitic), and can produce a product slate varying from predominantly heavy fuel oil through a clean product slate (naphtha/distillate) to a high naphtha yield slate[1], [2].

Recent advances in EDS process understanding permit varying the product slate through control of key process variables. This flexibilty, combined with the necessary economic information on costs of producing a given product state, allow for judicious selection of process conditions/configuration. In light of this flexibility, it is helpful to highlight the product quality characteristics of EDS-derived products to these three broad categories (naphtha, distillate, fuel oil).

Highly cyclic EDS naphthas represent unique and outstanding catalytic reforming feedstocks. High volumes of high octane motor gasoline blendstock are produced while liberating a considerable quantity of high purity molecular hydrogen. These qualities have been demonstrated in DOE-sponsored programs conducted by Universal Oil Products, Inc. (Contract No. EF-77-C-01-2566) and more recently by Chevron Research Company (Contract No. DE-AC22-76ET10532). These exceptional properties suggest a definite role for naphtha product.

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[1] EDS Commercial Plant Study Design Update, Illinois Coal, Interim Report, FE-2893-61, February, 1981.

[2] EDS Wyoming Coal Bottoms Recycle Design, EDS Monthly Technical Progress Report, July 1, 1982-July 31, 1982, FE-2893-94.

Without external upgrading, EDS distillates satisfy most of the performance characteristics for application as a home heating oil and as a stationary gas turbine fuel. While not meeting the current petroleum-based gravity specifications for ASTM No. 2 fuel oil and emitting somewhat higher NO_x levels (although there is no current specification for domestic heating oil), EDS distillates have been successfully demonstrated in both of these potential applications.

The properties of EDS distillates can be enhanced through hydrotreating. Extensive work has been carried out within the project to define the extent of hydrogreating required to meet any desirable distillate qualities, including products which meet all current specifications for No. 2 petroleum fuel oil and auto diesel fuel.

In stationary gas turbine applications EDS distillates are considered satisfactory and could be used commercially. These conclusions are based on studies addressing this potential application conducted by Westinghouse under EPRI-sponsorship (Program No. RP989-1). Non EDS-funded studies of EDS distillates are currently underway for low and medium speed diesel engines.

EDS-derived fuel oils vary considerably in properties depending on actual blend components used. Product quality studies with EDS fuel oils have demonstrated acceptable combustion characteristics of Illinois and Wyoming coal-derived blends. At the same time, however, several technical concerns have been identified. These relate to relatively high nitrogen content (NO_x emissions), incompatibility with many petroleum products, and handling concerns (wall deposits, water separation, potential toxic nature). Relative to petroleum, EDS fuel oils have exhibited high levels of PNA emissions based on small-scale combustion tests. However, more recent commercial scale tests have indicated PNA emissions levels similar to those from petroleum fuels. Regarding handling, certain fuel oil blends may not meet ASTM No. 6 fuel oil specifications and would have to be marketed as specialty fuels. Depending on compatibilty, handling of fuel oil blends in separate facilties may be required.

Improvement in fuel oil characteristics is possible through hydrotreatment. In addition, alternate and possibly more attractive product slates of naphtha and distillate fractions produced directly from the process have been investigated.

The above considerations, in addition to the major conclusions presented in Table 5-1, demonstrate the wide diversification of EDS products and their potential suitability for meeting a broad range of petroleum product needs.

5.2 End-Use Applications of EDS Products

A broad range of end-use applications for EDS product streams have been tested in the product quality program. Table 5-2 summarizes the most likely preferred disposition of individual product streams. In cases

Table 5-1

MAJOR CONCLUSIONS OF THE EDS PRODUCT QUALITY PROGRAM

- Consideration of future market needs, combined with generic properties of EDS-derived products, may suggest a clean product slate (naphtha/distillate) as the most desirable, long-range EDS product objective.

- Properties of lighter EDS product streams (naphtha, distillates) are essentially independent of liquefaction operating conditions for a given coal. This applies to both coal-only and botoms recycle operations.

- With the exception of heterocyclic content, primarily sulfur, properties of lighter EDS product streams (naphtha, distillates) are very nearly coal-independent.

Table 5-2

EDS PRODUCT DISPOSITION/PRIMARY PROCESSING OPTIONS

Stream	Disposition	Primary Processing
C_1/C_2	Pipeline Gas	--
C_3/C_4	LPG	--
C_2/C_4	Ethylene, Propylene	Steam Cracking
C_4/160°F Naphtha	Mogas	Hydrotreating
160/350°F Naphtha	Mogas Chemicals (BTX)	Hydrotreating/Reforming Extraction
350/430°F Naphtha	Mogas	Hydrotreating
Middle Distillate	Turbine Fuel	Direct Use
350/750°F and/or 430/750°F Solvent	Low, Medium Speed Diesel Fuel Home Heating Oil Blendstock No. 6 Fuel Oil Blendstock	Direct Use Likely Limited Direct Use Direct Use
	Home Heating Oil	Direct Use With Burner Adjustment
	Auto Diesel Blendstock	Moderate Hydrotreating
	Jet/Auto Diesel/No. 2 Fuel Oil	Severe Hydrotreating/ Hydroconversion
750/1000°F VGO	Fuel Oil Blendstock FCC or Hydroconversion Feed	Direct Use/Hydrotreating Hydrotreating
Coker Gas Oil and/or Scrubber Liquids	Fuel Oil Blendstock FCC or Hydroconversion Feed Fuel Oil Blendstock	Direct Use/Hydrotreating Hydrotreating Direct Use/Hydrotreating

where such disposition requires immediate processing the primary processing technology is identified. Any C_4^-minus co-products produced can be processed to meet pipeline gas (HGB) and LPG specifications or serve as chemical feedstocks for ethylene/propylene production.

It is expected that following hydrotreating and reforming, EDS naphtha (160°F-350°F) will be used almost exclusively as a prime mogas blendstock. Alternatively, or in conjunction with mogas disposition, the EDS naphtha can serve as a source of valuable chemicals. For example, raw naphtha is an abundant source of phenol/cresols, while the reformate contains high concentrations of benzene, toluene and xylenes (BTX). Extractive processing can be used to liberate these and many other desirable chemicals.

The use of 350-430°F hydrotreated heavy naphtha as a direct mogas pool blendstock has also been demonstrated. This stream is the front end of the 350-750°F middle distillate (solvent) fraction shown in Table 5-2. The solvent can be viewed as a fuel for several applications. Without additional upgrading, potential applications include fuel for stationary gas turbines and low or medium diesel engines, as well as No. 2 or No. 6 fuel oil blendstocks. Moderate hydrotreating improves this product stream for these applications and provides the upgrading needed for use as an auto diesel blendstock. Severe hydrotreating is required to meet all current specifications for jet, No. 2 diesel and No. 2 fuel.

The last three product streams, if present in a given EDS configuration, can be blended into the fuel oil pool. Contingent upon specific blend situations, they normally require no separate upgrading. In other situations, upgrading of these streams, or more likely of the total fuel oil blend, may be required to meet the applicable nitrogen and/or sulfur specifications. Finally, it is noteworthy that conversion studies by UOP, Inc. and Chevron Research indicate that the EDS mid-distillate and vacuum gas oil may be used as potential conversion feedstocks through the use of catalytic cracking or hydrocracking processing.

Except for C_4^-minus co-products, discussion of each of the applications identified in Table 5-2 comprises the major portion of this Product Quality Report.

5.3 Future Product Quality Needs

One of the key objectives of the product quality program was to identify areas pertaining to product quality requiring further development.

Table 5-3 summarizes potential end-use applications of EDS products and the associated constraints and limitations which have been identified as a result of product quality testing. In addition to those recognized limitations, there are technical uncertainties pertaining to several potential applications which could not be explored within the scope of the EDS program.

Table 5-3

EDS PRODUCT QUALITY ASSESSMENT

Stream/Product	Application	Identified Limitations/Constraints	Technical Uncertainty
A. C_1/C_2	Pipeline Gas	None	Likely None[1]
B. C_3/C_4	LPG	None	Likely None[1]
C. C_2/C_4	Chemical Feedstock	None	Likely None[1]
D. C_4/160°F Naphtha	Gasoline Blendstock	Hydrotreating Required to Eliminate Gas Forming Tendencies	Engine Performance
E. 160/350°F Naphtha	1. Extraction (Phenol, Cresols)	Raw Naphtha Exhibits Poor Storage Stability	1. Scale-up/Equipment
	2. Hydrotreating (Pre-Reforming)		2. Activity Maintenance
F. 160/350°F Reformate	Gasoline Blendstock	None	Engine Performance
G. 350/430°F Naphtha	Gasoline Blendstock	1. Hydrotreating Required (EDS Solvent H/T) 2. Low Octane; Volatility	None
H. 350/750°F Distillate	1. Gas Turbine Fuel	1. H-Content Lower than Petroleum ◆ Higher Aromaticity ◆ More Luminous Flame H-Content Higher than Petroleum	1. Large Scale Demonstration
	2. Low, Medium Speed Diesel Fuels	2. Not Yet Tested	2. Demonstration
	3. Home Heating Oil Blendstock	3. API Gravity <30 ◆ Burner Adjustment Required Higher NO_x Emissions	3. Long Term Performance
	4. No. 6 Fuel Oil Blendstock	4. None	4. None
	5. Auto Diesel Blendstock	5. Hydrotreating Required ◆ Filtration May be Required	5. Activity Maintenance
	6. Jet/Auto Diesel/No. 2 Fuel Oil	6. Severe Hydrotreating Required	6. Activity Maintenance Engine/Burner Performance
	7. Extraction of Aromatics	7. High Aromatics Content Reverse of Petroleum	7. Scale-up/Equipment/Solvent
I. 750/1000°F Vacuum Gas Oil	1. Specialty Fuel Oil	1. Segregated Storage/Handling Facilities ◆ Limited Compatibility With Petroleum ◆ Specific Gravity >1.0 High PNA - Handling High N-Level - NO_x Emissions	1. Large Scale Demonstration
	2. Hydrotreating Feedstock	2. Guard Chamber Likely Required	2. Activity Maintenance
	3. No. 6 Fuel Oil Blendstock	3. Hydrotreating Required Large H_2 Consumption Required For Low N, Compatible Fuel	3. None with Adequate Hydrotreating

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[1] Conventional processing of light hydrocarbons to meet interstate specifications likely adequate.

Technical uncertainties for the most part can be classified within the following three categories.

- Engine Performance

  End-use applications such as hydrotreated EDS light naphtha (C_4/160°F) and reformate (160/350°F) as gasoline blendstocks, hydrotreated EDS distillate as an auto diesel blendstock, or severely hydrotreated/converted distillate as jet or auto diesel fuel must be considered as potential applications until certified through extensive engine test programs.

- Large Scale Demonstration

  Utilization of EDS distillate as a gas turbine fuel, a low or medium speed diesel fuel or even a fuel oil blendstock appears promising based on laboratory or sub-scale tests of relatively short duration. However, extensive testing to assess long-term equipment performance and life is ultimately required.

- Catalyst Activity Maintenance

  Upgrading studies (e.g., hydrotreatment) have focused primarily on the effects of process variables using relatively fresh catalysts. Acceptable long-term activity maintenance, a concern with coal-derived products generically high in heterocyclic content, has not been included in these programs.

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

BOTTOMS PROCESS RESEARCH

6. Coking and Gasification Studies

The objective of bottoms coking and gasification studies performed during Phases IIIB-V of the EDS program was, initially, to investigate the feasibility of FLEXICOKING^R for EDS bottoms processing. After completion of IKG runs on CLPP Illinois (Monterey) and Wyoming (Wyodak) bottoms, further laboratory studies investigated the potential problems observed during IKG operation. Extensive data of coking yields, feed bottoms properties and gasification kinetics were obtained to support the study design of a FLEXICOKING unit and the planning of the prototype pilot plant demonstration.

Coke formation in a coal slurry preheater was observed during early ECLP operation. Laboratory bench studies were carried out to identify the causes and important process variables responsible for coke formation in the preheater. A continuous flow unit was also developed to simulate ECLP slurry preheater operation. Operation of this unit allowed identification of operating conditions which are conducive to coke formation in the slurry preheater for various feed coals.

6.1 EDS FLEXICOKING Studies

6.1.1 Experimental Facilities and Procedures

Severe bench units and pilot units were developed to simulate the individual reactors of the FLEXICOKING unit for bottoms processing. The schematic diagrams and operating procedures of these units are described in the EDS Technical Progress Reports referenced in Table 6-1. The functions of these units are briefly described as follows:

- The Minicoker is a fixed-bed bench unit used to obtain preliminary coking yield data using one gram bottoms samples. The unit is also useful for process variable screening studies, especially for bottoms samples of limited availablility.
- The Continuous Stirred Coking Unit (CSCU) is a pilot unit with the capability of feeding molten bottoms continuously at a rate of approximately 20 grams per minute. This unit was used to obtain coker product distributions at various coking conditions for study design and planning the prototype pilot plant demonstration.
- The Fluid Coker Bogging Unit (FCBU) is a 3" diameter fluid bed unit with continuous feeding of molten bottoms into a fluidized bed of bottoms coke. This unit was used to determine the bogging limit in simulations of a fluid coker using bottoms feed.

Table 6-1

BENCH AND PILOT UNITS FOR COKING/GASIFICATION STUDIES

Unit	EDS Progress Report Reference
Minicoker Bench Unit	1,2Q76 (FE-2353-2)
Continuous Stirred Coking Unit (CSCU)	1,2Q76 (FE-2353-2)
Fluid Bed Bogging Unit (FCBU)	1,2Q76 (FE-2353-2)
Large Stirred Coking Unit (LSCU)	1,2Q76 (FE-2353-2)
Fluid Bed Gasifier (FBG)	1Q77 (FE-2353-14)
Minigasifier Bench Unit	3Q77 (FE-2893-3)
Integrated Coking/Gasification Pilot Plant (IKG)	3Q77 (FE-2893-3)
Small Fluid Bed Unit (SFU)	3Q78 (FE-2393-21)

- The Large Stirred Coking Unit (LSCU) is a scale-up of the CSCU and is designed to produce about 2-1/2 barrels of coker liquid products per week. These products were used for product quality evaluation and burning studies.

- The Fluid Bed Gasifier (FBU) is a 3" diameter fluid bed gasifier. This unit is designed to allow batch gasification of coke samples (approximately 4 lbs charge) with air and steam. FBU was used to determine the effect of gasification on coke particle integrity and to investigate the effect of bed stagnation on sintering.

- The Minigasifier is a fixed bed bench unit using a charge of 1-2 gram. This unit was used to determine the relative gasification reactivity of various bottoms cokes. The data were used to formulate gasification kinetic models.

- The Small Fluidized Bed Unit (SFU) is a bench gasifier unit with a 1" diameter fluid bed reactor. This unit was used to study the process variable effects on sintering and coke particle attrition under simulated gasification conditions.

- The Integrated Coking/Gasification (IKG) pilot plant used for developing the petroleum FLEXICOKING^R process was modified to allow feeding of CLPP bottoms. The primary objective of IKG operation was to assess FLEXICOKING^R operability on EDS vacuum bottoms from Illinois and Wyodak coals.

6.1.2 Major Conclusions and Results

Results of these coking/gasification studies in various bench and pilot units have been reported in EDS Technical Progress Reports, as tabulated in Table 6-2. The major conclusions are discussed here briefly.

Coking yields were obtained in CSCU runs on bottoms from all EDS program coals. The coker liquid yields from bottoms for various coals were not appreciably different. Once-through coking operations without 1000°F$^+$ recycle were found preferable because of the low thermal conversion of the bottoms feed. The coking temperature of 70+RV$^{\circ}$F and 25% steam/feed ratio were recommended for the FLEXICOKING^R study design. Bottoms/coal blends were also found feasible with a product yield equivalent to the total contribution from coal and bottoms coked separately. Bottoms feed showed a lower tendency to bog than the reference petroleum residuum in FCBU runs on Illinois (Monterey) and Wyodak bottoms. Bed bogging should not present an operating problem because the Exxon proprietary bogging correlation based on petroleum feeds can be used to size the coking reactor. In coker product quality evaluations, sufficient liquid products from Illinois (Monterey) and Wyodak bottoms were produced from LSCU operations. Results of the product evaluation studies have been reported in the EDS Product Quality Final Report (FE-2893-97, p. 215, September 1982).

Table 6-2

Page 1 of 3

SUMMARY OF TOPICS COVERED IN EDS PROGRESS REPORTS - COKING/GASIFICATION STUDIES

Report	Topics and Results Covered
1,2Q76 (FE-2353-2)	● Description of design and procedure of a bench-scale coking unit (minicoker) ● Results of coking yields at various temperatures by minicoker tests ● Description of design and operating procedures for Continuous Stirred Coking Unit (CSCU) ● Results of CSCU runs to show the process variable effect (temperature, steam/feed ratio and vapor residence time) on coker liquid yields ● Description of design and start-up of Fluid Coker Bogging Unit (FCBU) for fluid coking bogging study ● Description of design and planning of Large Stirred Coking Unit (LSCU) for producing coker products for quality evaluation
3Q76 (FE-2353-6)	● Discussion of CSCU runs for recycle simulation, process variable effects and liquefaction/coking interaction ● Validation of FBCU by bogging tests with petroleum residuum ● Preliminary evaluation of viscometric behavior of coker liquids ● Progress of LSCU construction
76 Annual (FE-2353-9)	● Summary of CSCU results ● Bench study of heavy hydrocarbon carryover during coke circulation of fluid coker by minicoker tests ● Completion of bogging study by FCBU runs for Illinois bottoms ● Description of a bench-scale minigasifier for studying the gasification kinetics of bottoms coke ● Results of IKG fluid coking runs on CLPP bottoms
1Q77 (FE-2353-14)	● Bench coking study to investigate the effect of sulfuric acid treating of Wyodak coal on coking yield ● CSCU runs on CLPP Illinois bottoms ● Correlation of coke and liquid yield with CSCU runs in 1976 ● Discussion of difference between Microlube and HIVAC-C distillation of coker liquids
1,2Q77 (Phase IIIA Final, FE-2353-20)	● Summary of bench-scale minicoker test results ● Summary of coking results from CSCU runs ● Summary of bogging results from FCBU runs ● Correlation of feed bottoms properties with coking yield ● Correlation of heating value with elemental analysis for coker liquids
3Q77 (FE-2893-3)	● Minicoker runs to study liquefaction severity on coking yield and the feasibility of feeding coal/bottoms mixture ● CSCU runs on CLPP Wyodak bottoms ● Correlation of bottoms properties with coking yield ● Minigasifier runs to determine gasification rates of bottoms coke samples ● Initiation of FBU runs to study coke particle integrity ● Results of initial operation of IKG FLEXICOKING mode; observation of sinter formation in gasifier bed ● Laboratory inspection of IKG sintered deposits

Table 6-2 (Continued)

SUMMARY OF TOPICS COVERED IN EDS PROGRESS REPORTS - COKING/GASIFICATION STUDIES

Report	Topics and Results Covered
4Q77 (FE-2893-7)	• CSCU runs on CLPP Illinois bottoms under updated study design condition on RCLU under longer residence time • Summary of coking product quality from Illinois and Wyodak bottoms • Discussion of initial LSCU runs • Results of initial minigasifier runs on Illinois bottoms coke sample from IKG • Results of FBU runs for investigating the particle integrity under simulated gasification condition • Summary of results from IKG FLEXICOKING runs
1Q78 (FE-2893-12)	• Effect of temperature on coker liquid yield by CSCU runs • Results of gasification rate of Illinois and Wyodak botoms cokes by minigasifier runs • Study of fines generation under the gasification condition by FBU runs • Start-up of IKG pilot plant for continuation of FLEXICOKING runs
1978 Annual (FE-2893-17)	• Study of bottoms viscosity reduction by fluxing oils for preparation of bottoms feed for FLEXICOKING pilot plants • Summary of coking yields from CLPP once-through Illinois and Wyodak bottoms by CSCU runs • Correlation of coking yields with coking condition and feed bottoms properties • Gasification kinetic study by minigasifier for Illinois and Wyodak bottoms cokes • FBU runs for study of fines generation and sinter formation in gasifier • Summary of results from IKG runs
3Q78 (FE-2893-21)	• CSCU runs on CLPP Wyodak and Big Brown bottoms, CLPP Illinois recycled bottoms and Illinois bottoms with fluxing oils or 15% coal • Sintering studies by Small Fluidized Unit (FBU) runs • Effect of percent gasification on fines make by FBU runs • Cold model studies to investigate the fluidization behavior in IKG • Product yields, coke properties and gasification rates of IKG runs
4Q78 (FE-2893-25)	• Study of handling bottoms as bulk solids - friability (drop shatter test) and moisture susceptibility • CSCU runs on Illinois bottoms with coal and Big Brown bottoms • Gasifier bed sintering studies by SFU and FBU runs
1Q79 (FE-2893-29)	• Coking yields from CLPP Wyodak bottoms by minicoker runs • CSCU runs on RCLU bottoms from Burning Star coal • Plan of LSCU runs to prepare coker liquid samples for various program studies • Gasifier bed sintering studies by SKU and FBU runs • Study of gasification rate of Wyodak bottoms coke by minigasifier runs

Table 6-2 (Continued)

SUMMARY OF TOPICS COVERED IN EDS PROGRESS REPORTS - COKING/GASIFICATION STUDIES

Report	Topics and Results Covered
1979 Annual (FE-2893-35)	• CSCU runs on RCLU Arkwright bottoms and Wandoan bottoms • Progress of LSCU run to prepare coker liquids for various program studies • Modeling of coke gasification kinetics • Discussion of gasifier bed sintering from results of SFU runs • Review of carryover fines study by FBU • Review of IKG operation
3Q79 (FE-2893-41)	• Comparison of CSCU and minicoker product yields • CSCU runs on RCLU Burning Star bottoms • Bogging study for bottoms feed diluted with fluxing oil by FCBU • Study of bottoms coke kinetics by minigasifier runs • Study of sinter formation in gasifier or burner bed by SFU runs • Effect of additive in bottoms feed on sinter formation in gasifier by SFU runs
4Q79	• CSCU runs on RCLU Ireland and Wandoan bottoms • Bogging results of Monterey bottoms feed diluted with 20% creosote oil by FCBU runs • Relative sintering tendency of bottoms coke from program coals by SFU runs • Effect of CaO additive in bottoms feed on sinter formation
1Q80 (FE-2893-49)	• CSCU runs on feed bottoms with 20% HRCO • Study of fines retention at a temperature close to bogging (sticky bed operation) by FCBU
1980 Annual (FE-2893-53)	• CSCU runs on CLPP Illinois and Wyodak recycled bottoms • Summarized FDBU run result for studying fines retention by "sticky" bed • Coke particle attrition study under the simulated coking and gasifier condition by SFU runs • Effect of MgO additive in bottoms feed on sinter formation by SFU runs
3Q80 (FE-2893-57)	• Bogging tests on recycled bottoms feed by FCBU • SFU attrition under simulated gasification condition
4Q80 (FE-2893-70)	• SFU attrition tests under simulated combustion and gasification conditions • Updated correlation of coking yield with the feed bottoms properties

IKG pilot plant operation demonstrated FLEXICOKER operability by processing approximately 6000 pounds each of CLPP Monterey and Wyodak bottoms. Ash sintering and fines generation were identified as potential problem areas in applying bottoms FLEXICOKING on a commercial scale. FBU runs identified bed stagnation around the gas inlet as the major cause of sinter formation in the gasifier bed. In addition, a non-sintering region mapped with process variables was defined by SFU runs for bottoms cokes produced from various feed coals. Laboratory studies also showed that fines generation increased with the increase of carbon gasified. The fines loss could be controlled at a tolerable level by limiting the carbon gasification to less than 70%.

6.2 Refractory - Coal Ash Interactions Study

A study was undertaken to determine the effect of slag chemical corrosion on refractories under conditions expected in a partial oxidation (POX) gasifier. A commercial unit will have temperatures up to 3000°F with a reducing atmosphere of hydrogen, carbon monoxide, and steam under pressures up to 1000 psig. Under these severe conditions, refractory life will also be greatly affected by erosion and by spalling from thermal shock, in addition to chemical disintegration of the refractory caused by molten ash. The current goal for refractory life in a commercial slagging gasifier is a minimum of two years ("Design, Engineering, and Evaluation of Refractory Linkings for Slagging Gasifiers," FE-2785-T2, p. 1). Currently operating POX pilot plants have not reported achievement of this goal.

In addition to having superior resistance to chemical attack by coal ash constituents, a refractory suitable for POX slagging gasifiers must have low porosity to minimize slag penetration. Other necessary refractory properties include a high melting temperature (no liquid phases below 3000°F), low thermal conductivity, extreme hardness to resist erosion, resistance to spalling by thermal shock, and resistance to decomposition by hydrogen and carbon monoxide in the reducing atmosphere gas. In all probability, a single refractory will not have the optimum of each of these individual properties. Thus, a trade-off may be necessary in certain areas (e.g., porosity vs. spalling) in developing or choosing the best overall refractory for a slagging gasifier environment.

The seven commercially-available refractories that were examined in this study are listed in Table 6-3, along with a brief description of the individual compositions. The first three refractories are fused cast at very high temperatures and show relatively low porosity. The last four materials are sintered or partially sintered blocks that have not gone through a molten state. The relative porosity of these samples is much greater than that for the fused cast refractories (see Table 6-3).

The three ash materials used in this experimental program are derived directly from EDS bottoms from the Coal Liquefaction Pilot Plant (CLPP). The coal residues and CLPP yield periods are Illinois No. 6

Table 6-3

REFRACTORIES EXAMINED IN ASH/REFRACTORY STUDY

	Type	Description	Apparent Porosity, Percent
1.	Fused Cast Alumina (99%)	Mostly a-Al_2O_3 with some "b-Al_2O_3"	1.1
2.	Fused Cast Chromia (80%)-(80%)-Magnesia (8%)-Alumina (4%)-Ferria (8%)	A Complex Chrome Spinel	5.4
3.	Fused Cast Alumina (60%)-Chromia (30%)-Magnesia (6%)	An Alumina-Chromia Solution within a Magnesia-Chromia-Alumina Spinel	4.2
4.	Alumina (90%) - Chromia (10%)	An a-Al_2O_3 Matrix Bond Bonded with Alumina-Chromia Solution	16.0
5.	Magnesia (55%) - Chromia (20%) - Alumina (8%) - Ferria (11%)	Magnesium Oxide with Magnesium-Chrome Spinel	14.0
6.	Alumina (90%) - Chromia (10%)	Similar to No. 4	15.8
7.	Alumina (86%) - Chromia (9%) - Silica (2%)	Similar to No. 4, Silica added to Increase Spalling Resistance	18.6

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* Data Supplied by Refractory Manufacturer.

(Monterey) from YP-311, Wyoming (Wyodak) from YP-269, and Big Brown lignite from YP-336. The ash element analyses for these three materials are given in Table 6-4.

The initial phase of this project involved the testing and subsequent examination of ash/refractory combinations exposed in an air-atmosphere furnace for three days. The samples were prepared by placing a pressed pellet of ash (5-10 gm) in a 3/4" diameter hole (ca. 1/2" deep) that had been drilled into a small block of refractory (ca. 2" x 1-1/2" x 1"). All twenty-one ash-refractory combinations were tested at 2500°F, 2650°F, and 2800°F.

The effect of temperature was most evident upon visual examination of the individual slag/refractory samples. As the temperature increased from 2500°F to 2800°F, both the Wyodak and Big Brown slags showed increased penetration into the refractory. The Monterey slag has a higher viscosity at these same temperatures in air atmosphere, and generally showed less penetration than the other two ashes. The effect of slag penetration was also dependent upon the porosity of the refractory. One of the most porous samples, refractory 5 (see Table 6.3), allowed all three types of slag to penetrate readily at all three temperatures tested. The three low-porosity, fused-cast refractories (1, 2, and 3) showed the least penetration. This reduced slag penetration in refractories, 1, 2, and 3 was also accompanied by an increase in thermal cracking, especially for Sample 2.

After the high-temperature exposure is concluded, the sample is sliced into sections and polished for subsequent microscopic examination. In addition, selected fractions of the slag and slag/refractory layers are ground to a fine powder for X-ray diffraction analysis in order to identify the individual crystalline phases present, including reaction products formed at the slag/refractory interface.

Examination of the slag-refractory interface by optical and electron microscopy reveals a layer formation on the refractory surface ~~for some of the samples.~~ This is ~~particularly evident with refractory~~ sample 3 with Monterey ash. The layer is of a spinel of mixed-stoichiometry $(Fe, Mg)O \cdot (FE, Al, Cr)_2O_3$ and was evidently formed as a reaction product of slag and refractory constituents. It appears that the effect of the spinel layer is to act as a barrier to slow down further refractory degradation. Similar spinel-type layers have been identified in other samples, particularly those containing alumina and chromia. The spinel layer "barrier" is well-formed on the surface of the low-porosity refractories, especially samples 2 and 3. For the more porous samples such as 4, 6, and 7, this spinel layer shows more discontinuity. Typically, the spinel layer was approximately twice as thick for the samples exposed at 2800°F compared to those at 2500°F. Refractory reference standards showed virtually no change over this temperature range.

In addition to the three-day tests described above, a twelve-week, long-term test at 2500°F was undertaken in the air atmosphere furnace. Again, all twenty-one combinations of seven refractories and three

TABLE 6-4

81B-2-327

ASH MATERIALS BEING EXAMINED IN THIS STUDY

ASH ELEMENTS	ILLINOIS NO. 6	WYODAK	BIG BROWN LIGNITE
SiO_2	51.34	39.04	50.91
Al_2O_3	18.60	17.46	17.85
Fe_2O_3	19.54	5.19	7.38
CaO	5.09	26.89	17.48
MgO	0.90	6.63	3.47
Na_2O	1.29	1.92	0.55
K_2O	2.04	0.36	0.65
TiO_2	0.86	1.05	1.59
P_2O_5	0.35	1.46	0.11
SOFTENING TEMPERATURE °F, ASTM, TYPICAL			
REDUCING	2030	2180	2130
OXIDIZING	2400	2230	2200
B/A RATIO	0.41	0.71	0.42

NOTE: ASHES WERE PREPARED BY MILD OXIDATION OF EXTRACTED EDS BOTTOMS ACQUIRED FROM ACTUAL CLPP YIELD PERIODS. ASH ANALYSIS IS GIVEN ON AN "SO_3-FREE" BASIS.

ashes were tested along with reference standards of each refractory. For most of the long duration samples, the spinel layer buildup was a little greater than twice that of the short-term tests, even though the exposure time was almost thirty times as long. This observation tends to support the role of the spinel layer as a chemically resistant barrier to further refractory decomposition.

The second phase of this experimental program consisted of studies of similar ash-refractory combinations in a high temperature, controlled atmosphere furnace that will allow similation of reducing atmosphere compositions to be found in a partial oxidation gasifier. A representative diagram of the controlled atmosphere furnace is given in Figure 6-1. This two-inch diameter tube furnace is capable of sustained temperatures greater than 3000°F. The refractory/ash samples were prepared in the same way as for the air atmosphere samples.

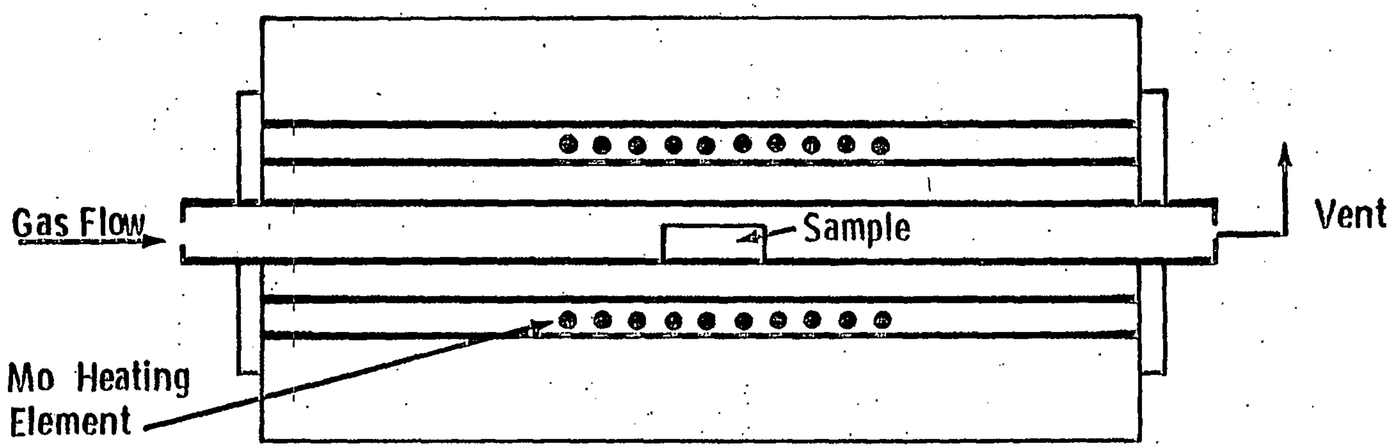
The first series of tests in the controlled atmosphere furnace were at 2650°F for a period of three days under a reducing atmosphere of 52% CO, 40% H_2, and 8% CO_2 (similar to the product gas composition (water-free) from a POX gasifier). For samples containing Illinois No. 6 ash, some metallic iron was observed to have formed in addition to an iron-rich spinel layer similar to that reported earlier in air atmosphere runs. In addition, the Illinois slag was much more fluid under this reducing atmosphere than under air atmosphere, resulting in greater slag penetration problems (see the ASTM softening temperatures in Table 6-4). Wyodak and Big Brown ashes showed similar behavior, although less metallic iron was present. All twenty-one combinations of three ashes and seven refractories were run under the above conditions.

Another series of tests were carried out at 3000°F in the same highly reducing 52% CO/40% H_2/8% CO_2 atmosphere. Refractories 1, 2, and 3 (all fused-cast with low porosity) and one of the sintered 90% alumina - 10% chromia materials were tested under these conditions as an upper temperature limit of a POX gasifier environment. In combination with the three ash materials, a total of twelve samples were tested. During the 24 hour test period at 3000°F, greater decomposition at the refractory surface was more apparent than at 2650°F. Refractory 1 (99% alumina) showed worse degradation than the other refractories, particularly with Wyodak and Big Brown slags. Refractory 3 (60% alumina - 30% chromia - 6% magnesia) appeared to perform best at 3000°F and also displayed little slag penetration. A well-defined spinel layer was also present at the slag/refractory interface.

A final series of tests was performed in the controlled atmosphere furnace under a less reducing atmosphere of 50% CO/50% CO_2 at 2650°F for three days. Under these conditions no metallic iron was observed to have formed in the slag/refractory samples. Magnetite, Fe_3O_4, was the primary iron-containing phase in the slag. Most of the samples tested under the 50% CO/50% CO_2 atmosphere also showed a well defined iron-rich spinel layer at the slag/refractory interface.

FIGURE 6-1

CONTROLLED ATMOSPHERE HIGH TEMPERATURE FURNACE



- CONCENTRIC ALUMINA TUBE DESIGN
- MAXIMUM TEMPERATURE 3200 °F
- CAPABILITY OF HIGH VACUUM, REDUCING, OR OXIDIZING ENVIRONMENT
- PROGRAMMABLE HEATING AND COOLING RATE

The following conclusions can be made from this work:

- Formation of a spinel layer on certain chromium-containing refractories can act as a chemically resistant barrier to further decomposition,
- Refractories #3 (60% Al_2O_3-30% Cr_2O_3-6%MgO) and #4 (90% Al_2O_3-10% Cr_2O_3) show good chemical resistance to slag degradation,
- Slag viscosity and refractory porosity are major factors in determining the rate of refractory decomposition.

6.3 Laboratory Studies of ECLP Preheater Coking

6.3.1 Bench Slurry Coking Studies

Tubing bomb tests were used to simulate EDS coal slurry preheater conditions. The procedure for these tests was described in the July 1, 1980-June 30, 1981 Annual Technical Progress Report (FE-2893-74). A coking bomb test was also used to simulate coke formation on the preheater wall while allowing for solvent evaporation. This test was described in the 1981 July - September EDS Technical Progress Report (FE-2893-78). Results from these laboratory tests were reported in EDS Technical Progress Reports as listed in Table 6-5.

Inspection of ECLP preheater coke samples by polarized light microscopic examinations showed that the coke adjacent to the preheater wall consisted of highly anisotropic carbon with large domains, as evidenced by the mosaic morphology. The observation of mesophase spherules in the coal residues from the tubing bomb tests confirmed the coke formed in the preheater via a liquid crystal mesophase. Coke is not formed as the coal slurry flows through the preheater furnace within the nominal residence time. Rather, coke formation is believed to be initiated with the deposition of mesophase spherules and/or coal slurry agglomerates on the preheater tube wall. These deposits subsequently coke.

Coking severity is dependent on tube wall temperature, time, and solvent quality. Coking does not occur at appreciable rates below about 800°F, and the time required for coking to occur decreases rapidly as the tube wall temperature is increased. Improving solvent quality decreases coking tendency at a given temperature for both Monterey and Wyodak coals. Further studies performed to evaluate the role of solvent parameters in slurry preheater coking show that hydrogen donating ability is more important than solvency in reducing coking severity. Polar or heterocyclic aromatic constituents in EDS solvents are not major contributing factors to coking in the ECLP slurry preheater.

6.3.2 Slurry Preheater Coking Studies in a Flow Unit

The small flow unit shown in Figure 6-2 was used to determine the relative coking tendencies of EDS program coals in the bottoms recycle mode at 0.5 bottoms/coal ratio. The flow unit simulates the time/temperature/chemical environment of the ECLP slurry preheater. The coals tested

Table 6-5

SUMMARY OF TOPICS COVERED IN EDS PROGRESS REPORTS - SLURRY PREHEATER COKING STUDIES

Report	Topics and Results Covered
81 Annual (FE-2893-74)	• Laboratory inspections of ECLP preheater coke plug • Solvent quality effect on coking tendency by thermal gravimetric analysis • Tubing bomb tests under simulated preheater condition
3Q81 (FE-2893-78)	• Evidence of the presence of mesophase spherules under the simulated ECLP preheater condition • Effect of temperature/time on coke formation by coking bomb tests • Microscopic studies of ECLP preheater coke deposits
4Q81 (FE-2893-83)	• Relative sticky tendency of coal, bottoms and HVGO • Study of chemical changes involved in coke formation • Effect of temperature and solvent quality on coking severity • Initiation of slurry preheater coking studies in a flow unit
1Q82	• Effect of phenol content in solvent on coking tendency • Effect of solvent solvency on coking tendency • Mapping of SQI/tube temperature for Wyodak coal and Martin Lake coal slurries by CCLU runs • Microscopic examinations of coke deposits
2Q82	• Proposed mechanism for coke formation in ECLP slurry preheater • Study of solvency effect by model solvents • Mapping of SQI/tube temperature for Illinois and Wyodak coal slurries with bottoms recycle by CCLU tests • Microscopic examination of coke desposits
3Q83	• Study of solvency effect on coking severity with model solvents by coking bomb tests • Mapping of SQI/tube temperature for Burning Star coal slurry on CCLU tests • Laboratory studies on ECLP hot separator deposits
4Q82	• Study of donor hydrogen and solvency effect on coking of coal derived asphalthenic materials • Study of temperature effect on hot separator deposits • CCLU runs on Ireland, Wandoan and Burning Star coal slurries

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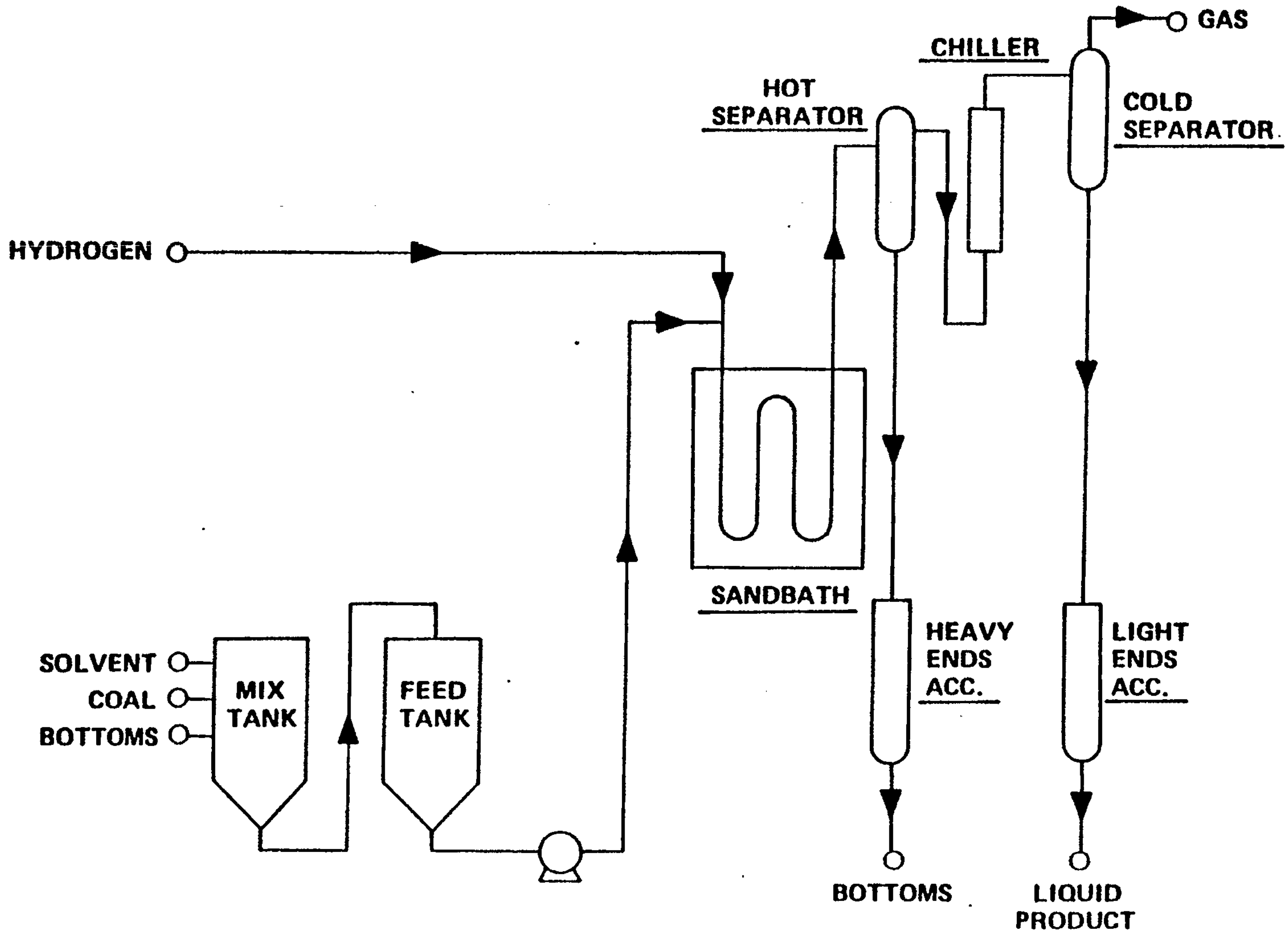
FIGURE 6-2

FLOW UNIT FOR PREHEATER COKING STUDIES



in the flow unit were Illinois No. 6 (Monterey and Burning Star No. 2 mines), Pittsburgh No. 8 (Ireland mine), Wyoming subbituminous (Wyodak mine), Texas lignite (Martin Lake mine), and Australian black (Wandoan mine). A list of the various studies undertaken and the appropriate references are given in Table 6-6.

Data obtained from polarized light microscopic analysis of each flow unit preheater tube were used to define the coking and non-coking regions of each coal as functions of tube temperature and solvent quality index. The coking maps for the EDS coals tested in the flow unit are given in Figures 6-3 through 6-7.

- Monterey Coal

  Coking with Monterey slurries (Figure 6-3) begins at 875°F at low (< 3.5) SQI. At higher SQI, the temperature required to produce coking increases dramatically. Coking with Monterey slurries is characterized by a rapid, complete plugging of the flow unit preheater tube.

- Wyodak Coal

  The coking behavior of Wyodak slurries (Figure 6-4) is also a strong function of SQI. At low (< 3.0) SQI, Wyodak slurries begin to coke at 800°F. As the SQI is increased, the maximum allowable tube temperature rises to 900°F at 4.5 SQI. Coking with Wyodak slurries is characterized by the formation of annular deposits, which build to form complete cross-sectional plugs at severe (> 900°F, low SQI) conditions.

- Martin Lake Lignite

  Coking with Martin Lake slurries is unique. Two coking regions have been defined for this coal, both of which are invariant with SQI (Figure 6-5). The partial coking region (> 920°F tube temperature) is similar to Wyodak coking in that annular deposits were formed. However, catastrophic coking was not observed with Martin Lake slurries. The minimal coking region (825-920°F) is unique to Martin Lake lignite. In this region, the coke formed is thin, an order of magnitude or less in thickness than in the partial coking region, and is limited to the first third of the flow-unit preheater tube. The exact impact of operating in the minimal coking region in a large unit is not clear.

- Ireland Coal

  A short study (11 tests) was conducted to determine the coking behavior of Ireland coal slurries (Figure 6-6). Ireland slurries begin to coke when the tube temperature reaches 950-975°F. Because the study was short, the effect of high SQI on coking was not determined. However, given the high maximum allowable tube temperature (950°F), the response of the coking boundary to SQI is of less importance than it is with coals such as Wyodak or Martin Lake.

Table 6-6

SUMMARY OF FLOW UNIT PREHEATER COKING STUDIES

Coal	Reference	
Wyodak	4Q1981	FE-2893-83
	1Q1982	FE-2893-89
Monterey	4Q1981	FE-2893-83
	1Q1982	FE-2893-89
Martin Lake	1Q1982	FE-2893-89
	2Q1982	FE-2893-93
Burning Star	3Q1982	FE-2893-99
	4Q1982	FE-2893-103
Ireland	4Q1982	FE-2893-103
Wandoan	4Q1982	FE-2893-103

FIGURE 6-3

MONTEREY COKING MAP

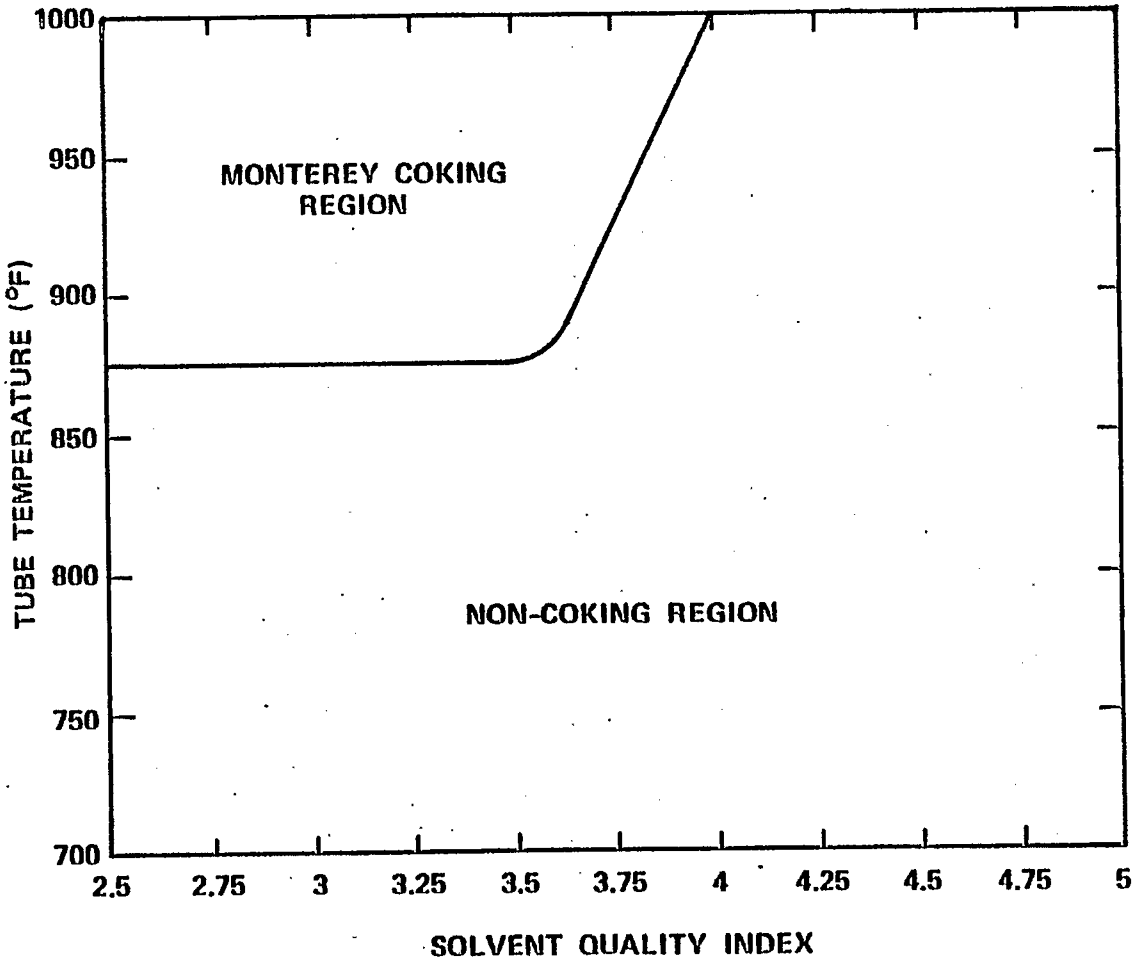


FIGURE 6-4

WYODAK COKING MAP

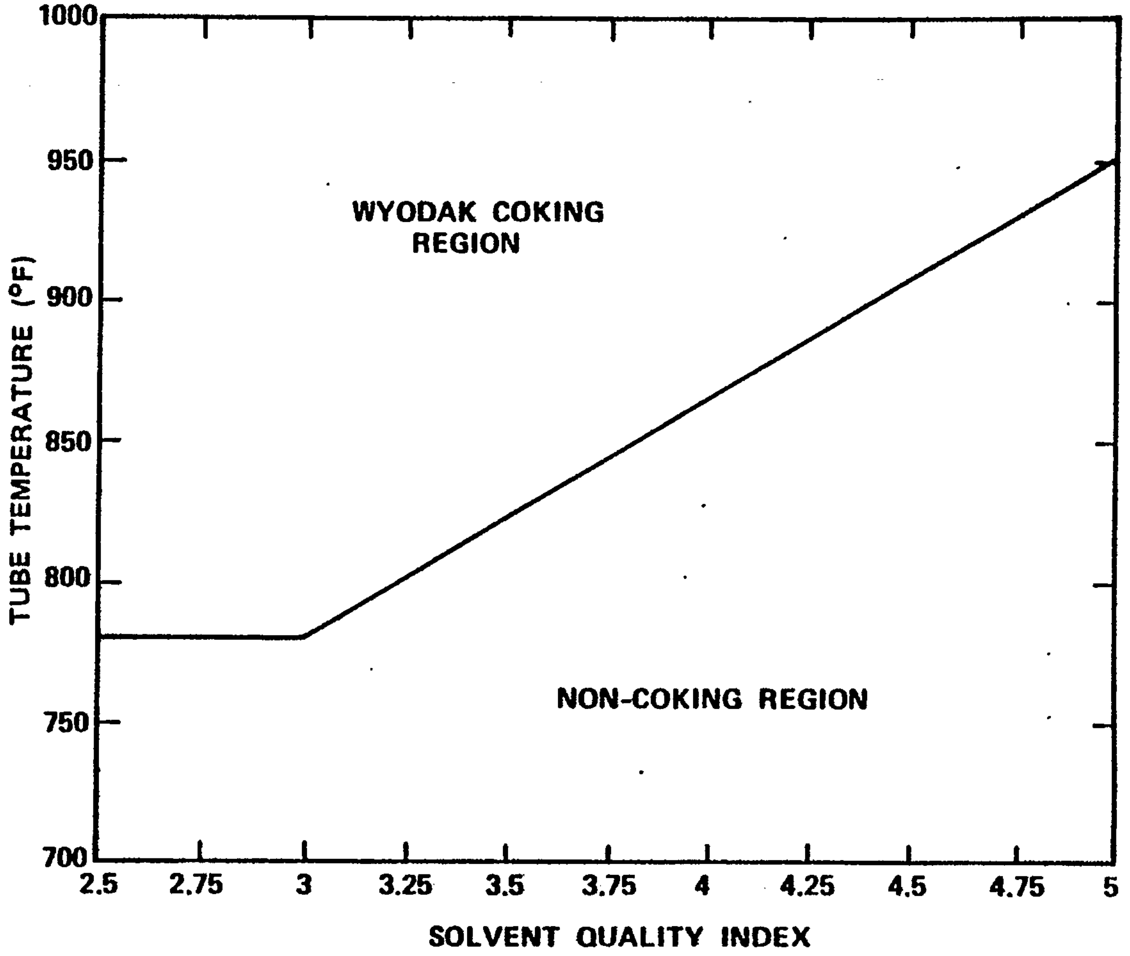


FIGURE 6-5

MARTIN LAKE COKING MAP

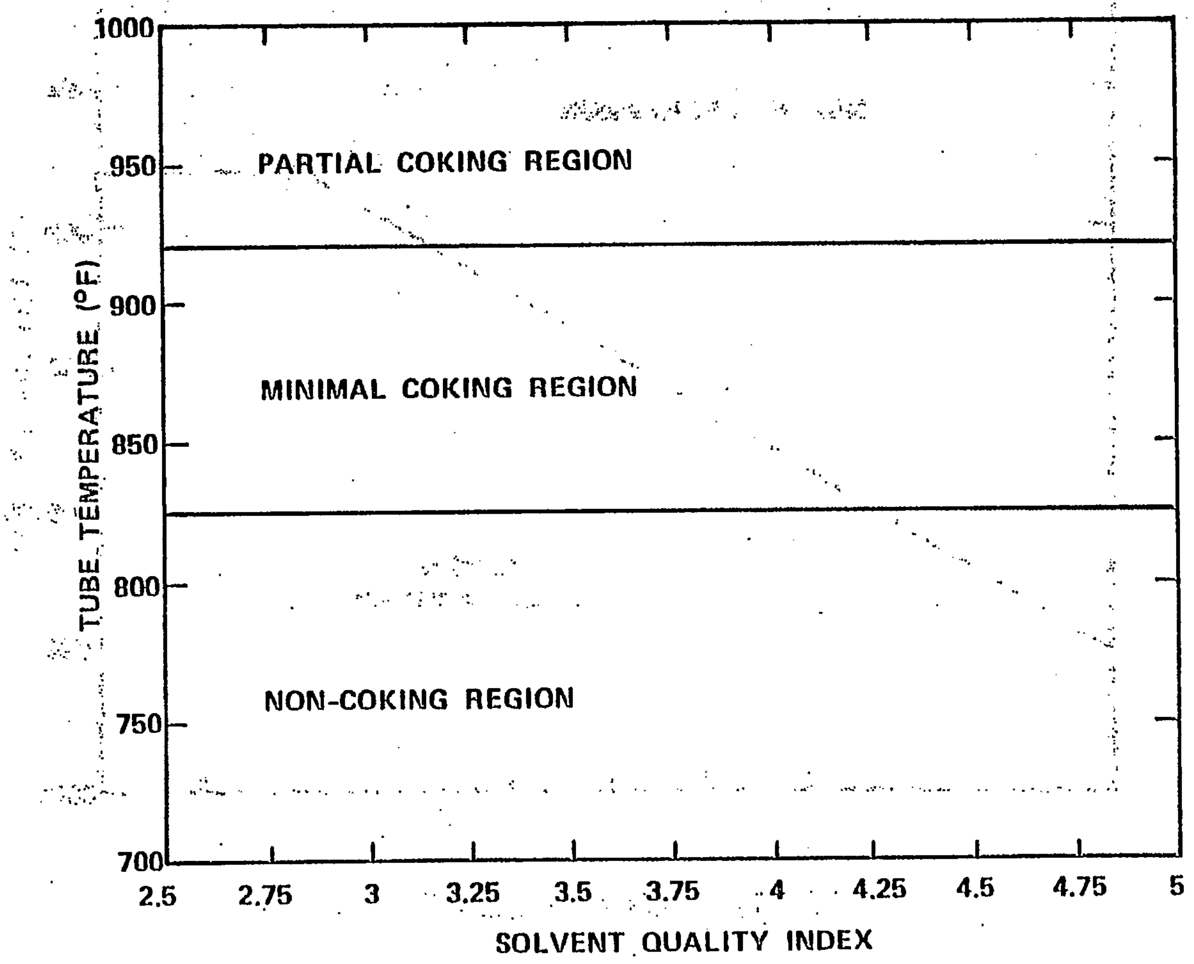


FIGURE 6-6

IRELAND COKING MAP

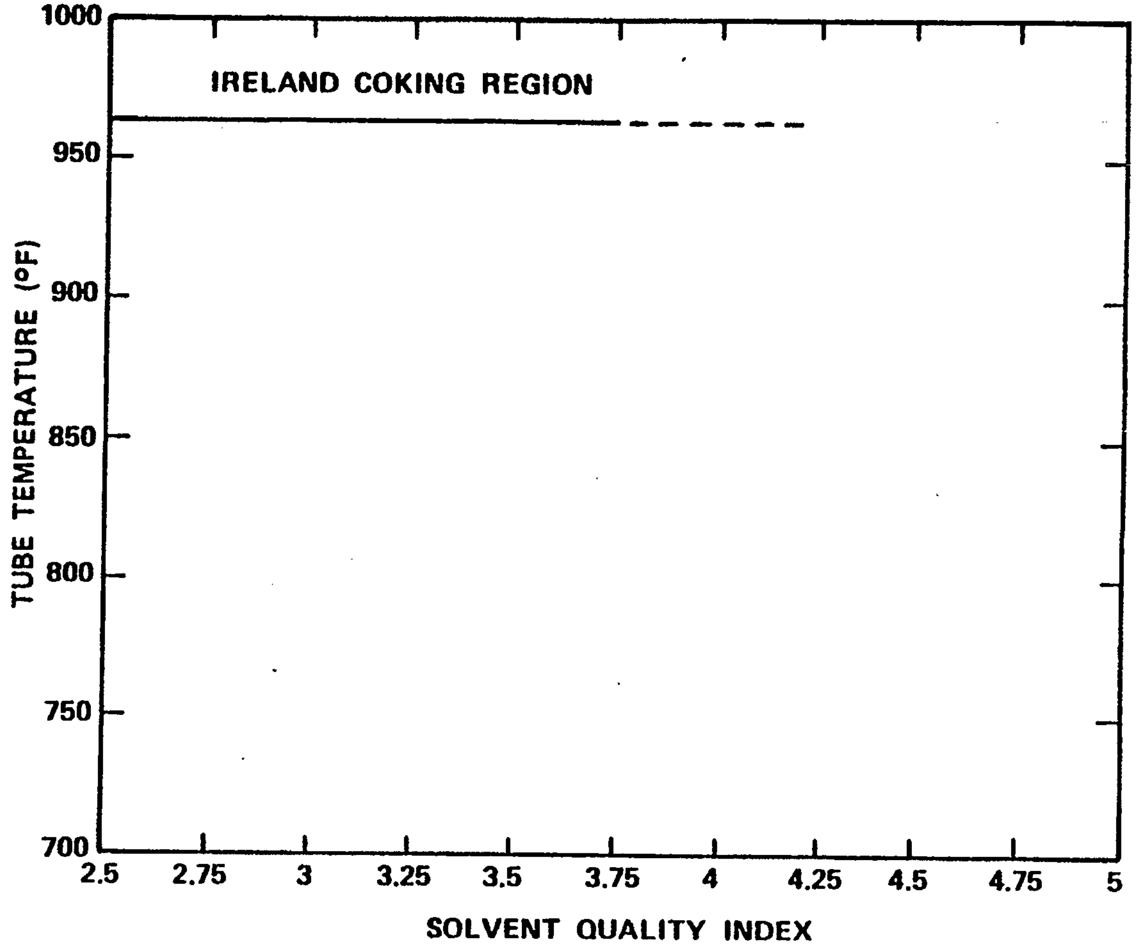


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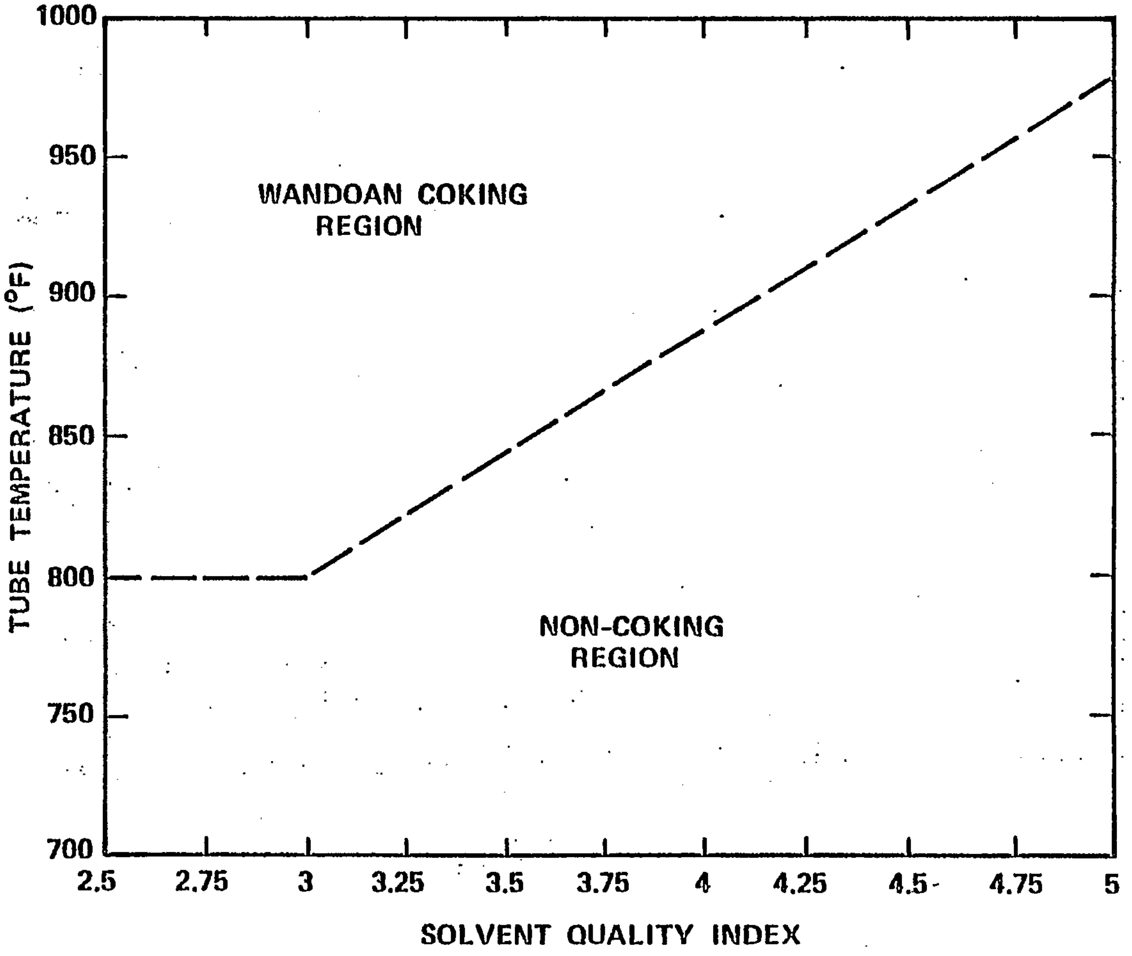
FIGURE 6-7

WANDOAN COKING MAP



- Burning Star Coal

  Burning Star coal slurries showed the least coking tendency of any coal studied. Only one condition studied showed any coke formation (975°F, 2.6 SQI). Therefore, it appears that Burning Star slurries only begin to coke at film temperatures of 975°F or higher and a low SQI ($\leq$ 2.6).

- Wandoan Coal

  The last coal tested in the flow unit was Wandoan coal (Figure 6-7). Although the tests showed considerble scatter (coking and non-coking points intermixed), the results are qualitatively similar to Wyodak coal in that coking was observed as low as 800°F.

  From these studies, the relative coking tendency of each coal in the bottoms recycle mode has been established as: Wyodak $\cong$ Wandoan > Martin Lake > Monterey > Ireland > Burning Star.

  The flow unit tests have been a cost effective and successful method of determining the relative coking tendencies of coal slurries and have provided valuable guidance and understanding during ECLP slurry preheater operations. This tool will play a role in the EDS licensing program to qualitatively assess slurry preheater coking risk for non-program coals.

6.4 EDS Bottoms Characterization

6.4.1 Introduction

Characterization of physical and chemical properties of EDS bottoms are required to provide a basis for bottoms utilization. Technical issues involved in each bottoms processing option can be better addressed with a complete understanding of bottoms structure and properties. Also, a detailed characterization and understanding is needed to establish a relationship between the bottoms properties and liquefaction conditions. This understanding, coupled with a knowledge of the coal structure, would help in understanding the coal liquefaction mechanism and thus impact on process improvements.

This characterization is carried out first by the inspection of pilot conditions, which can be correlated with bottoms properties, and then by systematic laboratory analyses. These analyses address both the chemical and physical natures of the bottoms, with measurements being both quantitative and qualitative.

6.4.2 Major Considerations

This report reviews bottoms characterization studies and documents current understanding of bottoms properties. Table 6-7 contains references for more detailed information. The analytical techniques used for bottoms characterization, the effect of liquefaction conditions on bottoms properties, and the chemical interactions which alter bottoms viscosity were studied and the major conclusions are summarized below.

- Bottoms Recycle Effects

  Recycling the vacuum bottoms to the liquefaction reactor in the EDS process produced improvements in at least three major areas as reported in the EDS Annual Technical Progress Report, July 1, 1980-June 30, 1981 (FE-2893-74).

Table 6-7

EDS Bottoms Characterization Studies

Reference	Topic
1980 Annual (FE-2893-53)	• Discussion of the viscosity and storage stability of once-through bottoms • Analyses of Illinois No. 6 (Monterey) bottoms by mass spectrometry and FTIR
3Q81 (FE-2893-78)	• Differences between Monterey once-through and recycle bottoms
4Q81 (FE-2893-83)	• Characterization of Wyodak recycle bottoms • Comparison of ECLP and CLPP bottoms for Wyodak bottoms • Correlation of solubility characteristics with overall conversion
1981 Annual (FE-2893-74)	• DSC analyses of Wyodak bottoms demonstrates thermal differences between once-through and recycle bottoms • Thermal stability of once-through versus recycle bottoms • Analyses of Wyodak bottoms by mass spectrometry
3Q82 (FE-2893-99)	• Characterization of Texas lignite (Martin Lake) recycle bottoms • Comparison of hydrogen to carbon and heteroatom to carbon ratios • Regressive reactions of the bottoms • Results of bottoms combustability by TGA

These improvements include a lighter product slate, lower bottoms viscosity and bottoms with improved thermal stability. Much of this improvement is a result of the longer reaction time, which allows for additional bond breaking and reduction of molecular size.

When the liquefaction bottoms samples from CLPP operations on Illinois No. 6 (Monterey mine), Wyoming (Wyodak), and Texas (Big Brown) lignite coals were analyzed, substantial differences were noted for the once-through and the recycle mode. One obvious difference was the higher ash content of the recycle bottoms resulting from the overall increase in conversion. The recycled bottoms have improved solubility, lower heteroatom contents and lower viscosities. The lower viscosity probably results from the reduction of aromatic sidechains and oxygen functionalities.

The viscosity of the recycled bottoms also is more shear rate-dependent (shear thinning) and less temperature-dependent than once-through bottoms. The decrease in temperature dependency is important because the recycle bottoms have less tendency for degradation during upsets or extended residence times.

- Elemental Analyses

Elemental analyses is one method used for comparing properties of the bottoms. Graphs presenting the H/C ratios and heteroatom ratios can be found in the EDS Quarterly Technical Progress Report July 1-September 30, 1982 (FE-2893-99). A low H/C ratio is characteristic of bottoms that are high in aromatics and very condensed. A high H/C ratio of approximately 2.0 is indicative of long sidechain aliphatics. The bottoms generated from ECLP had H/C ratios that ranged from 0.7 to 1.0, generally lower than the parent feed coal. The atomic H/C ratio in the bottoms typically decreases with increasing temperature and residence time, but increases with increasing hydrogen partial pressure.

Many of the physical characteristics of the bottoms are also affected by high atomic ratios of heteroatoms in the sample. These include oxygen-, nitrogen-, and sulfur-to-carbon atomic ratios. High heteroatomic ratios would be typical of polar bottoms which have a high melting range and a high viscosity. The O/C and S/C atomic ratios in bottoms are substantially lower than in the feed coal. Furthermore, these ratios decrease with increasing liquefaction severity. It is notable that the N/C ratio of the bottoms is very close to the ratio existing in the parent coal. This condition would indicate that the nitrogen is incorporated into the aromatic ring structure, which would add to the polarity of the bottoms.

- Solubility

Solubility of the bottoms is generally characterized by the extractability of four fractions. Oils, which generally consist of the 1000°F$^-$ material, can be separated from the bottoms by cyclohexane extraction. The asphaltene fraction of the bottoms is toluene or benzene soluble, but cyclohexane insoluble. This bottoms fraction is low melting and has a relatively low viscosity. The preasphaltenes consist of pyridine solubles but toluene insolubles. This fraction has a higher melting point and is considerably more viscous as compared to the asphaltenes. The pyridine insolubles are characterized by their high ash levels and highly crosslinked organics. The pyridine insolubles can remain unmolten in the bottoms during normal bottoms processing and contributes to high bottoms viscosity.

The October 1-December 31, 1981 EDS Quarterly Technical Progress Report reports the asphaltenes and preasphaltenes as being directly proportional to the amount of conversion. However, the scatter in the data suggest that operational factors also influence the solubility character.

- Regressive Reactions

Regressive reactions occur in the bottoms to different degrees, and are dependent upon the severity of the temperature, coal type (chemical functionalities present), unit residence time and the liquefaction conditions. The regressive reaction of asphaltenes to preasphaltenes and pyridine insolubles was investigated by analyzing the solubilities of atmospheric (or S-2 separator) bottoms and vacuum bottoms obtained from CLPP and ECLP. There were 64 of these sample sets (consisting of the atmospheric and vacuum bottoms) taken from CLPP and 9 taken from ECLP. Analyses on these samples show that the atmospheric bottoms asphaltenes are deconverted to preasphaltenes or pyridine insolubles in the vacuum bottoms. Analyses indicated a deconversion of 15.5±12.2% for CLPP and a 21.9±13.7% for ECLP.

It is suspected that the vacuum tower temperature and residence time are the primary factors in the amount of regressive reaction. These correlations, however, have been difficult to make without better data on the thermal history of the bottoms.

- Viscosities

When the bottoms viscosity was measured with different levels of solids (ash and pyridine insolubles), effects were observed in temperature and shear dependency. As the solids level increases there is less change in the viscosity resulting from variations in temperature. This non-Newtonian behavior of the bottoms is thixotropic, a condition where the viscosity decreases with increasing shear rate. Bottoms with lower levels of solids also show a much higher shear dependency at 600°F.

- Thermal Characterization

Thermal characterization of the bottoms has been conducted in three areas. Thermogravimetric (TGA) analyses provide information on the weight lost as vacuum bottoms are heated at a specified rate over a predetermined temperature range. The Differential Scanning Calorimetry (DSC) provides information on phase transitions of the bottoms over different temperature ranges and in different gaseous environments. The Thermomechanical Analysis (TMA) is typically used to obtain the melting range of the bottoms. With the collective use of these tools it has been possible to gain an understanding of the physical changes occurring in the bottoms at process temperatures.

TGA's were conducted on the weekly composites generated from ECLP. The results indicated that there is little difference in the thermal decomposition of the bottoms. However, when each of the solubility fractions were separated from the vacuum bottoms and analyzed, the thermal fractions were found to be in consistent ratios for each of the solubility fractions. The feed coals and the unit size had no apparent effect on the ratios of the thermal fractions for each soluble fraction. The asphaltene fractions contained from 50 to 55% volatiles and 45 to 50% fixed carbon. These volatiles were evolved over a range of 150 to 600°C. By comparison, the preasphaltenes had 20 to 25% volatiles and 75 to 80% fixed carbon. The pyridine insolubles maintained a volatile to fixed carbon ratio of 2:3. Because the ratio of the solids (ash and fixed carbon)-to-volatiles is a major factor in determining bottoms viscosity, the TGA can give an approximation of the bottoms viscosity range.

The DSC is used to detect both physical and chemical changes in the bottoms such as melting. Samples of the thermograms produced from DSC are reported in the July 1-June 30, 1981 EDS Annual Technical Progress Report (FE-2893-84). An exothermic peak that is characteristic of once-through bottoms can be observed above 140°C in the DSC thermogram. It is believed that this exothermic transition is the result of chemical condensation of the sample. When the sample was heated to 250°C and cooled back to 50°C for three cycles, the exotherm decreased with each cycle. This would indicate that the functionalities involved in the condensation reactions are slowly being consumed by the reaction. Experiences with heat soaking the bottoms over 24 hours at 600°F demonstrate the thermal instability of once-through bottoms and agree with the DSC findings.

Analyses by TMA show that the bottoms melt over a wide temperature range, normally between 160 and 300°C. By placing loads on the TMA probe, correlations can be made between the probe penetration and the pumpability of the bottoms at a given temperature.

- Bottoms Combustion

A study was conducted to determine the feasibility of providing process heat to the feed slurry or for steam generation by burning the vacuum bottoms. It was reported in the July 1-September 30, 1982 EDS Quarterly Technical Progress Report (FE-2893-99) that the bottoms burned at a slower rate and had a higher ignition temperature than the parent coal. Tests made on all of the coals processed in ECLP indicate the same results. The difficulty in burning the bottoms is proportional to the increases in conversion which result in high percentages of asphaltenes. It should be noted, however, that the laboratory tests were conducted at very low heating rates compared to the actual heating rates in a commercial furnace.

Fouling studies show that both the recycle and once-through bottoms had fouling rates that were close to that of the parent coal. This was supported in the tests made by Combustion Engineering where an Illinois once-through (ECLP) sample proved to be slightly less pone to fouling than would have been expected from the parent coal.

Sulfur emissions in the Solid Fuels Evaluation Unit (SFEU) suggest that controls to meet the 90% reduction criterion will be required, but SO_2 emissions will not approach the absolute limit. The data also suggest that NO_x reduction practices will be required.

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

1. PROJECT MANAGEMENT

This task was completed in 1980 and has been summarized in the Construction of EDS Coal Liquefaction Pilot Plant Final Report (FE-2893-66).

2. ENGINEERING AND PROCUREMENT

This task was completed in 1980 and has been summarized in the Construction of EDS Coal Liquefaction Pilot Plant Final Report (FE-2893-66).

3. CONSTRUCTION

This task was completed in 1980 and has been summarized in the Construction of EDS Coal Liquefaction Pilot Plant Final Report (FE-2893-66).

4. PREPARATION FOR OPERATIONS

This task was completed in 1980 and has been summarized in the Construction of EDS Coal Liquefaction Pilot Plant Final Report (FE-2893-66).

5. OPERATIONS

5.1 ECLP Simplified Flow Plan

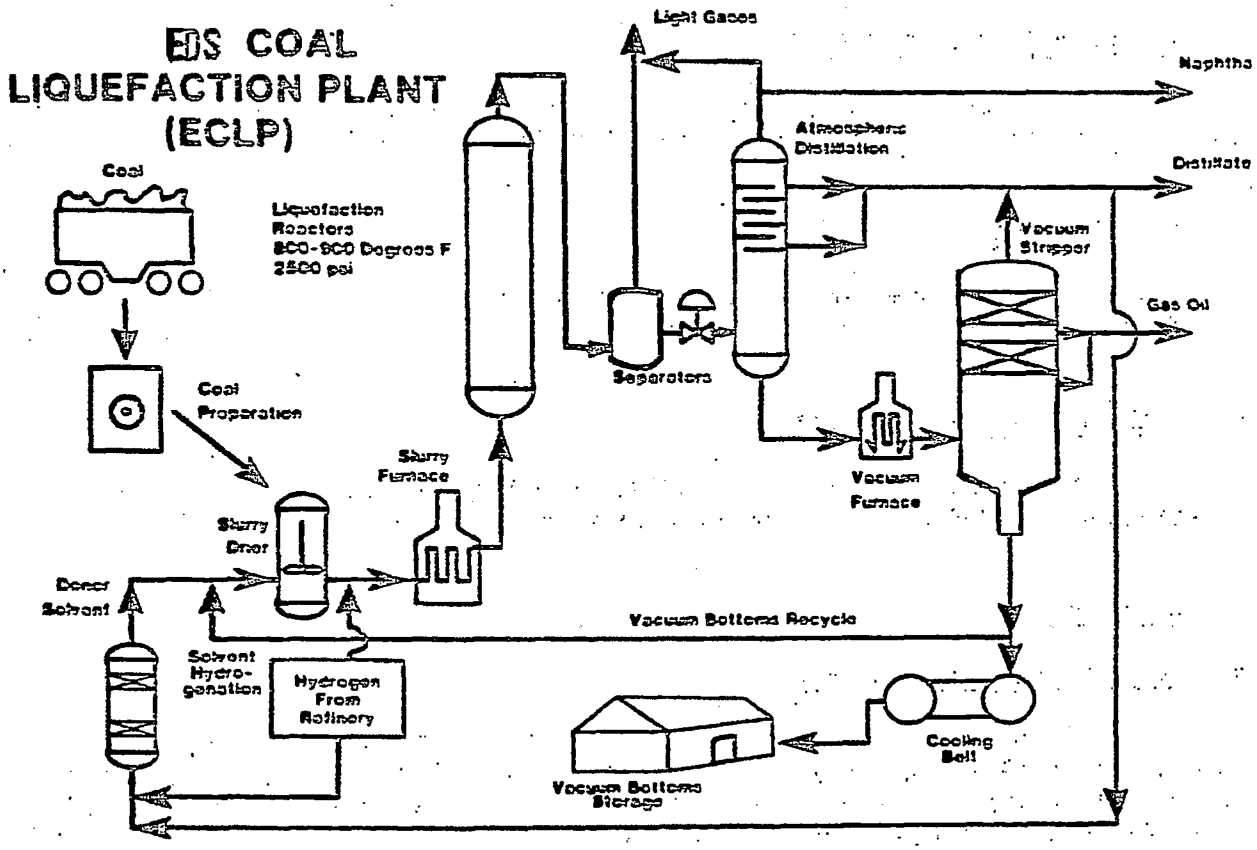
A simplified flow plan of the ECLP plant is shown in Figure 5-1. A more detailed flow plan is included in Appendix A, Figure A-1. The plant had four major sections -- coal receipt/preparation, coal liquefaction, product distillation, and solvent hydrogenation.

Mined coal was transported to the plant by rail in a 30-car unit train. Coal was stored in a silo of 5,000-ton capacity and was prepared by crushing and drying in either a gas-swept mill or an impact mill plus slurry drier. The prepared coal was slurried with hot, hydrogenated recycle solvent in the slurry drier and was pumped in two stages to a pressure of about 2500 pounds per square inch. Hydrogen-rich recycle gas was added to the slurry and the mixture was then heated to 780°F to 810°F in a commercial-type furnace using vertical serpentine coils. The mixture passed through a series of two to four vertical liquefaction reactors which provided nominal residence times of 40 to 90 minutes. Exotherms from the reaction increased the average liquefaction temperature to about 830°F to 850°F.

After liquefaction, the three-phase mixture passed into a separator drum. The vapors from the separator were sent through hot and cold separators for additional condensation. The slurry from the separator was sent through a single-stage pressure letdown valve and fed, after mixing with the hot and

FIGURE 5-1

ECLP SIMPLIFIED FLOW PLAN



cold separator liquids, to an atmospheric tower. The slurry was fractionated into a naphtha stream, two distillate streams, and a bottoms stream containing the original coal ash. These atmospheric bottoms were sent through a preheat furnace into a vacuum tower where light and heavy vacuum gas oil streams were stripped from the vacuum bottoms stream. The distillate sidestreams from the atmospheric tower and the overhead from the vacuum tower were combined, hydrotreated to replenish donor hydrogen, and returned to the slurry drier as recycle solvent. A slipstream of net solvent was directed to product tankage. The gas oil sidestreams from the vacuum tower were at various times taken as product, recycled to the slurry drier, or recycled to the solvent hydrotreater. The vacuum bottoms contained hydrocarbons boiling above 1000^{O}F, plus the original ash in the coal and any unconverted coal. During bottoms recycle operations, a portion of the bottoms was recycled to the slurry drier and fed to the liquefaction reactors. The remaining portion of the bottoms was solidifed on a cooling belt and stored in a building onsite for further upgrading tests.

5.2 Summary of ECLP Operations

5.2.1 ECLP Start-up and Shakedown

Mechanical completion of ECLP was achieved on March 21, 1980 with the turnover of the Liquefaction Distillation Unit (LDU) to Exxon Company, U.S.A. Following mechanical completion, all plant equipment was checked out for proper operation, pressure tested and run in. Also, the Solvent Hydrogenation Unit (SHU) catalyst was activated and a supply of creosote oil was hydrogenated for a start-up solvent.

Initial coal operations were achieved on June 24, 1980. During the three and a half month shakedown period that followed, Illinois No. 6 coal from the Monterey No. 1 mine was fed to the plant for a total of 1245 hours in eight individual runs. A detailed tabulation of each run is included in Appendix B. As shown in Figure 5-2, the coal-in percentage for this time was 51 percent. This period consisted of shaking down the plant, learning how to run it, detecting problems and potential problem areas, and developing changes to correct any deficiencies found. Some of the problems encountered during the shakedown were the short packing life of the slurry reciprocating pumps, the plugging of process piping due to the solidification of heavy coal liquids, slurry piping erosion, which resulted in failure of an elbow in the vacuum furnace transfer line, and plugging of the slurry feed heat exchanger (E-102) due to saltation of coal solids.

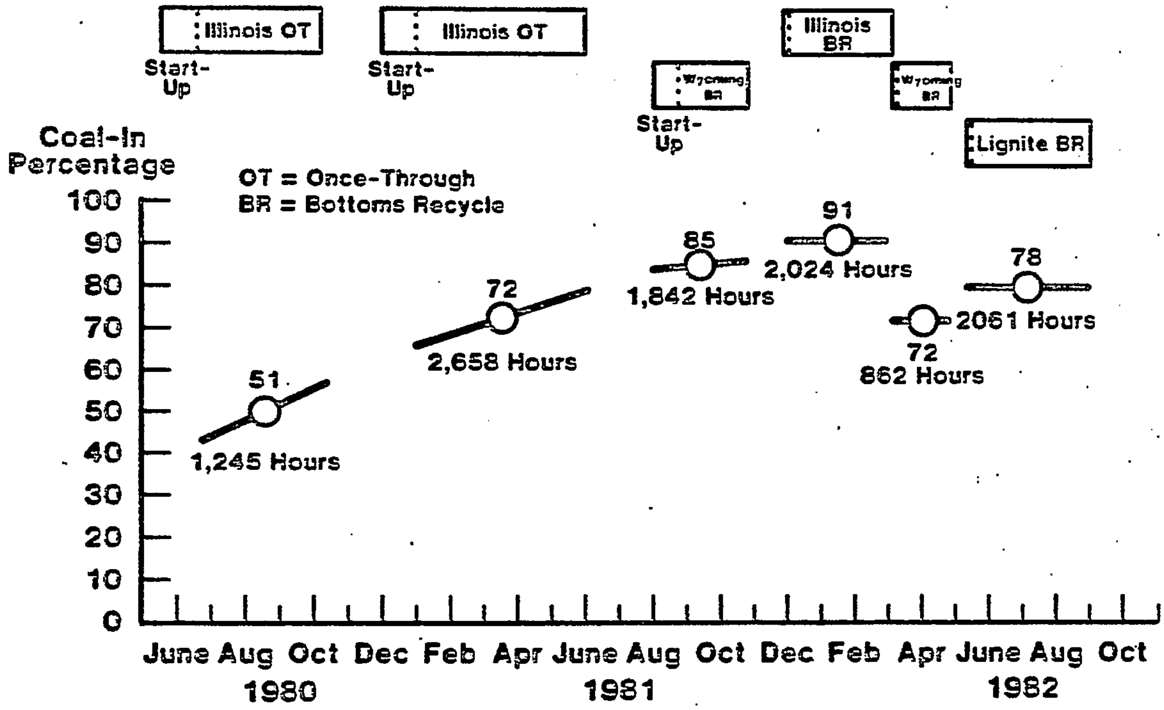
5.2.2 First Turnaround - October, 1980

In October 1980, a turnaround was conducted to allow equipment inspection to determine the effect of operation on plant internals and to make modifications to remedy the problems detected during shakedown. The major findings of this turnaround were:

- Plugging and disruption of the distributor trays in the two liquefaction reactors which were used during shakedown. The trays were removed for the next operating period.

Figure 5-2

ECLP OPERATING HISTORY



- Slight erosion/corrosion in the upper section of the atmospheric tower (T-201).
- Erosion in the inlet area of the vacuum tower (T-204).
- Erosion of the transfer line from the vacuum furnace (F-201) to T-204. It was replaced with a refractory-lined line and elbow.
- Erosion of the cyclones in the gas-swept mill.

Additional turnaround information is contained in the July 1, 1980 - June 30, 1981 EDS Annual Technical Progress Report (FE-2893-74).

5.2.3 Illinois Coal Testing Operation

Following completion of the turnaround at the end of November 1980, start-up activities were commenced. Coal-in operations resumed on December 30, 1980 beginning a five-month testing program. During both the shakedown and testing periods, the operation was in a once-through mode, that is, without the recycle of vacuum bottoms to the liquefaction section of the plant.

During the testing operation, the coal-in factor was improved to 72 percent. A total of 2658 hours of operations on Illinois No. 6 coal from the Monterey No. 1 mine were achieved in nine individual runs (Fig. 5-2 plus details in Appendix B).

In general, operations during this period were hampered by low coal conversion, poor solvent quality and hot spot formation in the slurry preheat furnaces. Other major learnings were:

- Corrosion in the upper section of the atmospheric tower shell resulted in its holing-through near Tray No. 23. Temporary stainless steel slab latches were welded on the shell between four trays.
- Wear-life of the main slurry letdown valve (L-106CV) was excellent. A life of 128 days was achieved with one valve.
- A rotary-valve coal feeder to the slurry drier gave much better service than the original gate-lock feeder.

Additional information concerning this operation is contained in the July 1, 1980 - June 30, 1981 EDS Annual Technical Progress Report, FE-2893-74.

5.2.4 Second ECLP Turnaround - June 1981

The second ECLP turnaround occurred during June 1981 to quantify further the effects of the Illinois coal operation on plant equipment. The major findings of this turnaround were:

- Major erosion of some flooded tees in the slurry pumparound piping. All tees of this type were replaced with long-radius elbows.
- Coke deposits in the atmospheric tower feed preheat exchanger (E-105).

- Hard coke-like deposits in the liquefaction reactor effluent separator (D-103).

- Severe spalling of the atmospheric tower feed mix chamber (MC-203).

- Fouling of the stripping baffles in the vacuum tower (T-204).

- Severe erosion at the tangential inlet to the vacuum tower (T-204).

- Ammonium chloride deposition in the solvent hydrogenation cold separator condenser (E-302).

Additionally, part of the upper section of the atmospheric tower was replaced with a 321 stainless-steel-lined section in a five-tray zone. The tangential inlet section of the vacuum tower (T-204) was lined with alumina bricks for better erosion resistance. Redesigned stuffing boxes were installed on the atmospheric bottoms pump (P-204A) to extend packing life. Redesigned distributor trays were installed in the first two liquefaction reactors (R-101A/B). R-101C and D were placed in the operating reactor train and were left without trays.

More detailed information about this turnaround is contained in the July 1, 1980 - June 30, 1981 EDS Annual Technical Progress Report, FE-2893-74.

Following completion of this turnaround, the installation of the facilities to recycle a portion of the vacuum bottoms stream to liquefaction was completed. All subsequent ECLP operations were carried out in this bottoms recycle mode.

5.2.5 First Wyoming Coal Operation

On July 31, 1981, operations began in a bottoms recycle mode. Operations continued through August 7 when a switch was made to a Wyoming subbituminous coal from the Wyodak mine. During this operating period, 1842 hours of coal-in operation were completed in two runs (Figure 5-2 plus details in Appendix B). The coal-in factor was improved to 85 percent.

Highlights of the first Wyoming coal bottoms recycle operation were:

- Completion of Run 18 which lasted for 57.9 days. This was the longest run during ECLP operations.

- Successful commissioning of the vacuum bottoms recycle facilities.

- Reduction of the solvent hydrogenation unit catalyst volume to improve solvent quality.

- Completion of radioactive tracer tests to determine gas holdups in the liquefaction reactors.

- Improvement in solvent quality by removing additional light-boiling saturates from the solvent by distillation.

- Improved packing life of the atmospheric bottoms pump (P-204A).

As in the previous operation, low coal conversion, formation of hot spots in the slurry preheat furnace, and poor solvent quality were encountered during this operation. Some improvement in solvent quality was made because of the above changes in operations.

Additional information on this operation is contained in the July 1,-September 30, 1981 and the October 1-December 31, 1981 EDS Quarterly Technical Progress Reports, FE-2893-78 and FE-2893-83, respectively.

5.2.6 Third ECLP Turnaround - November, 1981

Following a completion of the Wyoming coal bottoms recycle operations in October 1981, a turnaround inspection was conducted. During this turnaround, an equipment inspection was completed, the slurry preheat furnace was modified into a two-zone (high-flux/low flux) design to lower the slurry film temperatures, and redesigned stuffing boxes were installed on the slurry feed pump (P-102B). The major findings during the equipment inspections were:

- The long-radius elbows in the slurry pumparound piping were in good condition. Minor erosion was noted downstream of weld intrusions.
- A minor accumulation of solids was left in the liquefaction reactors (R-101A/B/C/D) after draining. Only minimal plugging has occurred on the R-101 A/B distributor trays.
- Deposits of soft solids were noted in the liquefaction reactor effluent separator (D-103).
- No erosion or corrosion was detected in the atmospheric tower (T-201).
- Minor erosion had occurred in the transfer line from the vacuum furnace to the vacuum tower (T-204).
- Significant thinning of the chimneys on the vacuum overflash and heavy vacuum gas oil trays in the vacuum tower (T-204) had occurred. The chimneys were sleeve-lined to repair them.

Additional details of this turnaround are contained in the October 1-December 31, 1981, EDS Quarterly Technical Progress Report (FE-2893-83).

5.2.7 Illinois Coal Bottoms Recycle Operation

Following completion of the turnaround, a three-month Illinois No. 6 (Monterey No. 1 mine) bituminous coal bottoms recycle operation was started on November 28, 1981. During the operating period, 2024 hours of coal-in operation were achieved in four runs (Figure 5-2 plus details in Appendix B). Two of these runs were terminated because of extremely cold weather in January, 1982. The coal-in factor was 91 percent.

Highlights of the Illinois coal bottoms recycle operations were:

- Attainment of design points for a commercial-sized plant at solvent-to-coal-to-bottoms (S/C/B) ratios of 2.3/1/0.5 and 1.6/1/0.5.
- Trouble-free operation of the modified slurry preheat furnace. There were no indications of hot spot or coke formation at S/C/B ratios as low as 1.6/1/0.5.
- Demonstration of a coal conversion level of 60 percent $1000^{\circ}F^{-}$ on a dry and ash-free coal basis.
- Improved solvent quality. Higher donor hydrogen, lower saturates and higher $600^{\circ}F+$ contents were obtained.
- Installation and operation of slurry drier screw feeders (CO-101, 102) to inject the feed coal below the slurry drier liquid level.
- Installation and operation of the redesigned, vertical slurry heat exchangers (E-102A/B). Plugging of these exchangers continued but was caused by oversize coal rather than saltation.
- Installation and operation of the modified slurry letdown valve (L-106B-CV). The valve was streamlined to improve its wear life during bottoms recycle operations.

Further information on this operating period is contained in the October 1-December 31, 1981 and January 1-March 31, 1982, EDS Quarterly Technical Progress Reports, FE-2893-83 and FE-2893-89, respectively.

5.2.8 Second Wyoming Coal Bottoms Recycle Operation

On March 1, 1982, the on-site supply of Illinois coal was used up and operations continued on Wyoming subbituminous coal from the Wyodak mine in a bottoms recycle mode. There was no coal outage between these operations. During this Wyoming coal operating period, 862 hours of coal-in operations were achieved in six runs (Figure 5-2 plus details in Appendix B). Mechanical problems, not specifically related to coal type, dropped the coal-in factor to 72 percent during this operation.

Highlights of the second Wyoming coal bottoms recycle operations were:

- Attainment of a design point for a commercial-sized plant at a solvent-to-coal-to-bottoms (S/C/B) ratio of 2.0/1/0.5.
- Trouble-free operation of the modified slurry preheat furnace (F-102B) with no indications of hot-spot formation at a S/C/B ratio of 2.0/1/0.5. Operations at lower S/C/B ratios did result in some increases in tube metal temperatures.
- Demonstration of a coal conversion of 66 percent $1000^{\circ}F^{-}$ on a dry and ash-free coal basis.

- Production of high-quality donor solvent.

- Completion of a record run on the atmospheric bottoms pump (P-204A) with redesigned stuffing boxes. During this run, a packing life of 88 days (79 days on slurry) was achieved.

- Successful withdrawal of solids from the liquefaction reactors using the high-pressure letdown system.

- Installation and operation of the slurry drier vapor disengagement drum (D-122). This drum was designed to allow water vapor disengagement from the feed slurry before it entered the suction of the slurry feed booster pumps (P-101 A/B).

- Installation of smaller diameter tubes in the outlet row of the modified slurry preheat furnace (F-102A).

Additional information about this operation is contained in the January 1-March 31, 1982 EDS Quarterly Technical Progress Report (FE-2893-89) and in the July 1 1981 - June 30, 1982 EDS Annual Technical Progress Report, FE-2893-93.

5.2.9 Fourth ECLP Turnaround

The second Wyoming coal operation was discontinued on April 19, 1982, when a hole was eroded in the shell of the atmospheric tower feed preheat exchanger (E-105). A mini-turnaround was conducted to:

- Repair the atmospheric tower feed preheat exchanger (E-105).

- Inspect the vacuum tower (T-204).

- Clean the effluent separator (D-103) and hot separator (D-104) drums.

- Skim a catalyst restriction from the first solvent hydrogenation reactor (R-301).

Turnaround inspection results were:

- Extensive, hard, coke-like deposits in the effluent separator (D-103).

- Hard, coke-like deposits in the atmospheric tower feed preheater exchanger (E-105) shell.

- Misplaced chimney hats in the vacuum tower trays for light and heavy vacuum gas oils.

- Moderate erosion of the refractory-lined transfer line from the vacuum furnace to the vacuum tower (T-204).

Additional turnaround information is contained in the July 1, 1981 - June 30, 1982 EDS Annual Technical Progress Report, FE-2893-93.

5.2.10 Texas Lignite Bottoms Recycle Operations

Following completion of the turnaround, operations were resumed on May 2, 1982, with some Wyoming coal remaining from the previous operation. With operations continuing, a switch was made on May 12, 1982 to a Texas lignite from the Martin Lake mine. During this operating period, 2061 hours of coal-in operations were achieved in six separate runs (Figure 5-2 plus details in Appendix B). The overall coal-in factor was 78 percent.

Highlights of the Texas lignite bottoms recycle operation were:

- Attainment of a design point for a commercial-sized plant at a solvent-to-coal-to-bottoms (S/C/B) ratio of 1.6/1/0.5.
- Completion of 28 days of operation at a S/C/B ratio of 1.6/1/0.5 with no indication of hot spot formation in the modified slurry preheat furnace (F-102A) with smaller diameter tubes in the last half of the outlet row. Lower S/C/B ratios (down to 1.2/1/0.5) were tested for shorter time periods. Also, higher slurry film temperatures were demonstrated at a 2.0/1/0.8 S/C/B ratio.
- Demonstration of a coal conversion level of 66 percent $1000^{\circ}F^{-}$ on a dry and ash-free coal basis.
- Production of high-quality donor solvent. Although the $600^{\circ}F^{-}$ content of the lignite solvent was slightly higher than the two previous operations, the donor hydrogen and saturates contents were similar. The lighter solvent was caused by lower vacuum tower severity necessitated by the high lignite bottoms viscosity.
- Demonstration of the EDS process with feed coal ash contents of 9 to 20 weight percent (a maximum of 23 weight percent).
- Slurry drying of high-moisture lignite.
- Control of liquefaction reactor solids buildup by high-pressure letdown solids withdrawal.
- Further improvement in the packing life of the slurry reciprocating pumps.

Additional details of this Texas lignite bottoms recycle operation are contained in the July 1, 1981 - June 30, 1982 EDS Annual Technical Progress Report, FE-2893-93, and in the July 1- September 30, 1982 EDS Quarterly Technical Progress Report, FE-2893-99.

5.2.11 Final ECLP Inspection

ECLP operations were terminated on August 20, 1982 to begin the final ECLP inspection. During this inspection, all major pieces of equipment were opened or disassembled for inspection. Notable inspection results were:

- Severe erosion of the casings and impellers of the gas-swept mill venturi scrubber circulation (P-1101) and blowdown (P-1510A/B) pumps.
- Severe fouling of the distributor trays in the lead liquefaction reactors (R-101A/B). Some hard, coke-like deposits were found in all liquefaction reactors.
- Severe plugging of R-101D. Drilling and hydroblasting were required to remove the plug. The plug may have resulted from operational difficulties during the final shutdown.
- Hard coke-like deposits in one-half of the bottom head of the liquefaction reactor effluent separator (D-103). The upper cartridge-tray section was clean.
- Heavy wear of the main slurry letdown valve (L-106ACV) plug tip and seat. Both were made of tungsten carbide with a titanium diboride coating.
- Moderate erosion of the atmospheric tower (T-201) inlet transfer line.
- Moderate corrosion of the carbon steel shell of the atmospheric tower (T-201) below the stainless steel-lined section (Tray No. 19).
- Cracking of some stainless steel hardware in the area of Tray Nos. 20 to 26 in the atmospheric tower (T-201).
- Moderate erosion of the refractory-lined vacuum tower (T-204) transfer line and flash zone ceramic brick.
- Moderate erosion of the carbon steel chimneys on the vacuum tower (T-204) overflash and heavy vacuum gas oil trays.
- Moderate corrosion of the carbon steel trays and internals in the upper section of the solvent fractionator (Tray Nos. 27 to 33).

Additional details of the results of this inspection are contained in the July 1-June 30, 1982 EDS Quarterly Technical Progress Report, FE-2893-99.

5.3 ECLP Equipment Performance

This section presents a brief summary of the performance of the major ECLP equipment during total operations.

5.3.1 Drums

- Slurry Drier (D101) — There were initial operating problems with D-101 caused by the inadvertent setting of its mixer speed at 28 rpm, which did not suspend all of the coal. At mixer speeds of 68 or 84 rpm, there were no problems.

  During slurry drying operations, problems were encountered with the slurry feed booster pumps (P-101) because of entrained water vapor bubbles in the pump suction. An additional drum (D-122) was placed in the pump suction to allow vapor disengagement. This drum allowed higher feed rates of wet coal but did not completely eliminate pumping problems during slurry drying. However, sufficient data for design of a commercial-size slurry drier were obtained.

Liquefaction Reactor Effluent Separator (D-103) -- During Operations, there was a general difficulty in detecting the precise liquid level in D-103. Nuclear level detectors (continuous level) and point source alarm level) were used to detect level but a head-type meter was used to control the level. Overflow of D-103 into the hot separator drum (D-104) was caused both by inaccuracies in determining level and by sudden increases in gas rates which "burped" solids from the liquefaction reactors into D-103.

During turnaround inspections, hard coke-like deposits were generally found in the bottom of D-103 due to high drum temperatures (above 800°F). Similar deposits were found in the upper cartridge section when the overhead wash system was not used. After wash oil, addition to the top of D-103 was commenced in May 1982, fouling and coking in this cartridge section were minimal.

- Liquefaction Hot Separator Drum (D-104) -- This horizontal drum trapped loose reactor solids which were carried over from D-103 overflows during upsets. These solids fouled the displacement-type level detector and severely eroded the hot separator letdown valve (L-108CV).

- Solvent Hydrogenation Unit Feed Filter (D-308) -- Pressure drop increases across D-308 occurred on several occasions during ECLP operations. The cause of these increases was solids carry-over into liquefaction product solvent streams. At times, normal pressure drops could be restored by backflushing D-308 to slop tankage with solvent. If this was not successful, catalyst bed replacement in the drum was necessary.

5.3.2 Towers

- Atmospheric Tower (T-201) -- The upper, carbon-steel section of T-201 corroded through the tower wall during the Illinois coal once-through testing period. Replacement of the affected section with a stainless steel-lined section controlled the corrosion. Similar corrosion was found in the atmospheric Tower No. 1 sidestream stripper (T-202), but to a lesser extent.

  During operations, overflow of the T-201 bottoms into the atmospheric Tower No. 2 sidestream caused pump mechanical seal failures and solvent hydrogenation unit feed filter (D-308) plugging.

  During the final inspection, moderate erosion was detected in the T-201 inlet transfer line.

- Vacuum Tower (T-204) -- Erosion of the T-204 inlet transfer line initially was severe. The line was replaced with a fiber-reinforced, refractory-lined one during the first turnaround. This new line controlled the erosion but did not totally eliminate it. Erosion of the thermowells in this transfer line, especially those near the tower, was very severe. Similar erosion occurred in the T-204 flashzone area and was controlled by lining this area with ceramic bricks.

Corrosion of the carbon-steel chimneys occurred on the trays for the vacuum overflash and heavy vacuum gas oil drawoffs. Sleeve lining with stainless steel was used to extend their lives.

- Solvent Fractionator (T-303) — During the final inspection, corrosion similar to that in T-201 was found in the upper section of T-303 and in the solvent fractionator No. 1 sidestream stripper (T-304).

5.3.3 Slurry Pumps

- Slurry Feed Booster Pumps (P-101) — The P-101 pumps generally performed well. The single mechanical seals used in these pumps were adequate. Erosion in these pumps generally occurred in the cutwater area. Erosion in other areas was highly specific to pump design.

- Slurry Feed Pumps (P-102) — Although the P-102 pumps performed well generally, early operations were hampered by short packing life and pumping problems due to large coal particles. Packing life was greatly improved through the use of different packing schemes (type, material, arrangement, etc.) and redesigned stuffing boxes. Also, coal preparation operations were adjusted to produce coal with no more than one weight percent larger than eight mesh.

  A pulsation dampener, which used recycle gas injection, dampened the pump discharge pulsations much more effectively than the original liquid-filled dampener.

- Atmospheric Bottoms Pumps (P-204) — These vertical reciprocating pumps also performed well, in general, during ECLP operations. Although early packing life was very short, it was improved by using alternate packing schemes and redesigning the stuffing box. Packing life was improved from one to two days up to 131^{+} days at the end of operations.

  Pump performance was reduced, at times, by refractory particles which had spalled from upstream equipment. In-line filters were installed to keep this material out of the P-204 suction.

- Atmospheric Bottoms Test Pump (P-216) — Although the operating time of this pump, which was in a recycle test loop, was very limited due to its system design deficiencies, its performance was adequate to generate the necessary data.

- Vacuum Bottoms Pump (P-210) — The performance of this screw pump was satisfactory, in general, but poor at times; however, it was difficult to determine its pumping rate due to continuous use of an unmetered recycle from discharge to suction. During the 1982 ECLP operation, performance deteriorated because of wear of the screw flights and casing.

A clean, high-pressure flush oil supply was critical to achieving long packing life in this pump. Leaking of the light vacuum gas oil through the packing into the pumpage affected solvent balance and solvent quality. After leakage of bottoms into the packing began, packing life was very short.

- Vacuum Bottoms Recycle Pumps (P-222) -- These centrifugal pumps performed well but were generally operated at low speed due to low flows and heads required. Their double mechanical seals and flush oil supply system worked well.

5.3.4 Reactors

- Liquefaction Reactors (R-101) -- The R-101 reactors had a lower liquid holdup than the smaller EDS pilot plants (CLPP and RCLU). This lower holdup was confirmed by pressure drop and radioactive tracer testing. It resulted in lower coal conversions than had been achieved in the smaller pilot plants at the same "nominal residence time."(1) Acceptable conversions were eventually obtained by using all four reactors in series and operating them at higher temperatures.

  Temporary loss of flow in the reactors caused the bed of reactor solids to slump and partially plug the distributor trays in the reactor. This plugging resulted in the disruption of the inlet distributor trays or the formation of hard, coke-like deposits. Redesigned supports with greater strength were needed to keep the trays in place if plugging occurred. Performance in the two foot diameter ECLP reactors was not affected by the absence or presence of the distributor trays.

  The original systems for withdrawing liquefaction reactor solids were not operable chiefly due to flow control difficulties and plugging of piping. The cycling-valve system was modified to allow its operation as a high-pressure letdown system. This system was used to successfully remove solids from all four reactors during the ECLP Texas lignite operations.

- Solvent Hydrogenation Reactor (R-301) -- The only operating problem associated with R-301 was periodic pressure drop increases across its catalyst bed. These increases were caused both by carry-over of liquefaction reactor solids into clean solvent streams and the deposition of a heavy, organic, iron-rich phase from the solvent or from tank scale. R-301 catalyst bed skimmings or replacements were performed in August 1980, and during the turnarounds of October 1980, June 1981, and April 1982.

5.3.5 Valves

- Slurry Block Valves -- Through conduit gate valves worked well during ECLP operations. These valves needed to be flushed during each cycle to remove coal solids from the body cavity.

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(1) Defined as reactor volume divided by slurry volumetric rate at 60°F.

Plug valves also performed well. Regular lubrication was required to maintain performance. Some stem system failures occurred when proper lubrication was not provided.

- Slurry Letdown Valve -- The original design slurry letdown valve, a sweep-flow angle valve with tungsten carbide internals, had a long life (128 days) during once-through operations.

  The valve was redesigned to handle the higher ash slurries during bottoms recycle operations. A life of 79^+ days was obtained during the Illinois and Wyoming coal bottoms recycle operations.

  Valve wear accelerated during the high-ash Texas lignite operations. The valve life of 24 days was adequate to avoid operational problems. There was no apparent benefit in using a titanium boride coating over the base tungsten carbide.

5.3.6 Miscellaneous Equipment

- Coal Feed System -- The original gatelock feeder (GL-101) for the slurry drier gave many operational problems due to its mechanical complexity and unreliability. A replacement rotary valve (RV-101) gave much better service but did allow gas blow-by except at very low slurry drier pressures. This resulted in carry-over of coal fines into the slurry drier overhead system.

  Both of these problems were solved by using screw feeders (CO-101/102) to feed the coal into the slurry drier below the slurry level. Flush solvent injection was required at critical locations on these feeders to keep the coal moving into the slurry drier.

- Slurry Feed Heat Exchangers (E-102A/B -- The original E-102 design was a horizontal, multipass exchanger. Although the tubes were designed for velocities above the saltation velocity, saltation occurred in the channel heads where there were much lower velocities. These exchangers were completely redesigned to vertical down-flow, single-pass ones. This eliminated the saltation problem.

  Trash material and oversize coal particles did plug these exchangers on occasion. Back flushing or mechanical cleaning were required to remove the plugging material.

- Flushing Systems -- The flushing systems at ECLP worked well, especially in emergency situations. The biggest problem in this area was the carry-over of solids into clean solvent streams and then into the flushing system. These solids caused packing and seal failures and erosion in equipment which was not designed for slurries. Another problem in the vacuum bottoms area was that the 400°F flush oil was too cold to adequately flush high-melt-point bottoms.

- Blowdown System — The blowdown system at ECLP worked well as long as the blowdown piping was flushed after use. Erosion of the piping did occur when high-pressure equipment containing slurry was discharged into the low-pressure blowdown system.

  Rerunning of the slop solvent from the blowdown system was a general consideration during routine operations. Slop was rerun into the slurry drier (D-101). The need to maintain high coal concentrations in D-101 for testing purposes generally allowed only a small amount of slop to be rerun.

- Coal Crushing

  - Impact Mill (CR-1102) — CR-1102 operations were very limited because of problems in slurry drying. Special care was taken during the start-up of the mill to keep oversize coal out of the slurry drier system where it could clog the slurry heat exchangers. Wear and breakage of its internals was high during the high-ash Texas lignite operations due to the presence of petrified wood and rocks.

  - Gas-Swept Mill (CR-1101) — CR-1101 was used to crush coal during most of ECLP operations and operated well. Some erosion and corrosion occurred in the overhead cyclone and venturi scrubber system but was easily controlled.

- Slurry Flowmeter — Venturi meters were used almost exclusively for slurry flow measurements. Although affected by the non-Newtonian slurry, their accuracy was adequate for control purposes. A reliable high-pressure flush oil supply was necessary to prevent impulse line plugging and ensure continual operation.

- Vacuum Bottoms Solidification Belt (CO-1901) — Operating problems with CO-1901 were chiefly caused by poor distribution of vacuum bottoms across the width of the belt. This was caused by an inadequate bottoms distributor design which allowed bottoms to solidify at the ends of the distributor. The resulting sheet of bottoms on the belt was thicker than necessary and more difficult to completely solidify. Additional water was sprayed directly onto the bottoms sheet to aid in solidification.

  Control of fugitive fume emissions from the belt was also difficult at times because the venturi scrubbing system was not always adequate to keep a negative pressure inside the belt enclosure. The problem was compounded by the need to open the enclosure to observe or correct bottoms distribution, and by southerly winds which created a "wind tunnel" inside the enclosure. Emissions were minimized by installing internal curtains to break the wind and minimizing opening of the enclosure.

5.4 ECLP Safety and Occupational Health Programs

During the Preparation for Operation Phase of the ECLP project, a 22-point Safety and Occupational Health Program was instituted. The components of this program were:

- Safety Policy Statement
- Plant Safety Steering Committee
- Special Task Committee on Employees
- Accident/Disability Reporting and Investigation
- Safety Performance Indices
- Safe Operation Committee
- Emergency Organization Plans
- Risk Management
- OSHA Compliance
- Safety Awareness
- Basic Safety Training
- Equipment Identification
- Task Books
- Operating Procedures
- Interactions with Maintenance and Other Contractors
- Housekeeping
- Buildings Safety Plans
- Equipment Designs
- Safety and Operability Audits
- General Inspection Program
- Equipment Inspection Program
- Carcinogenic Materials Protection Program

Additional information on these components is contained in the Appendix. A separate report, ECLP Industrial Hygiene - II, FE-2893-119, summarizes the results of the Occupational Health Program.

Table 5-1 briefly summarizes the ECLP safety performance during ECLP operations.

5.5 ECLP Operating Data

Table 5-2 briefly summarizes yield test data from operations during the Illinois coal bottoms recycle, the second Wyoming coal bottoms recycle and the Texas lignite bottoms recycle operating periods.

TABLE 5-1

ECLP SAFETY SUMMARY

1980

	EXXON			CONTRACTORS	
	Process	Others	Total	Mechanical	Instrument/ Electrical
On-Job Hours	99,821	180,055	279,876	558,556	36,514
Off-Job Hours	179,677	324,197	503,874	558,600	39,492
AII or RII[1]	2.38 (AII)	1.8 (AII)	2.04 (AII)	2.5 (AII)	0 (RII)
Target AII or RII	1.0 (AII)	0 (AII)	1.0 (AII)	4.0 (AII)	4.0 (RII)
On-Job Disabling Injuries	0	0	0	1	0

1981

	EXXON			CONTRACTORS	
	Process	Others	Total	Mechanical	Instrument/ Electrical
On-Job Hours	109,200	175,310	284,510	456,398	28,565
Off-Job Hours	181,920	315,619	497,539	664,711	37,880
AII or RII	2.75 (AII)	0.63 (AII)	1.45 (AII)	2.2 (RII)	0 (RII)
Target AII or RII	1.2 (AII)	NA	1.0 (AII)	3.0 (RII)	3.0 (RII)
On-Job Disabling Injuries	0	0	0	1	0

1982

	EXXON			CONTRACTORS	
	Process	Others	Total	Mechanical	Instrument/ Electrical
On-Job Hours	72,254	104,106	176,270	196,733	16,332
Off-Job Hours	120,425	187,272	307,697	NA	NA
AII or RII	0 (AII)	2.1 (AII)	1.3 (AII)	7.1 (RII)	0 (RII)
Target AII or RII	1.7 (AII)	0.9 (AII)	1.3 (AII)	2.5 (RII)	2.5 (RII)
On-Job Disabling Injuries	0	0	0	2	0

---

[1] AII - All Injury Index; RII - Recordable Injury Index

TABLE 5-2

ECLP Operating Data

Coal	Illinois No. 6	Wyoming	Texas
Type	Bituminous	Subbituminous	Lignite
Mine	Monterey No. 1	Wyodak	Martin Lake
Yield Test Date	1/28/82	3/22/82	6/23/82
Liquefaction Temperature, °F	846	855	845
Liquefaction Pressure, psig	2520	2493	2512
Liquefaction Hydrogen Rate lbs/100 lbs dry coal	4.7	6.9	5.3
Nominal Liquefaction Residence Time, Minutes	81	85	88
Solvent-to-coal Ratio	1.6	2.0	1.55
Recycle Bottoms-to-coal Ratio	0.6	0.5	0.5
Yields, lbs/100 lbs dry ash-free coal			
H_2	-4.8	-4.9	-4.5
$C_1 - C_3$	11.0	10.4	10.3
C_4 - 400°F	23.3	25.0	23.9
400-1000°F	14.8	15.7	15.4
$1000°F^+$	39.2	34.0	33.8

Additional and more detailed information is contained in Appendix C, Tables C-1 through C-7.

Table 5-3 summarizes the ovarall consumption of coal and production of coal liquids for the different ECLP operating periods. The "yields" calculable from these numbers will not be the same as the above yield pattern due to coal and liquid losses in the plant and to operations at different conditions.

TABLE 5-3

ECLP Production Summary

	Illinois Coal Once-Through	Illinois Coal Bottoms Recycle	Wyoming Coal Bottoms Recycle	Texas Lignite Bottoms Recycle
Coal Processed, tons	36255	13717	21894	19422
Naphtha Produced, barrels	17919	11780	14963	11763
Solvent Produced, barrels	-514	4727	3910	5079
Heavy Vacuum Gas Oil Produced, barrels	5096	0	0	0
Vacuum Bottoms Produced, tons	14868	4947	6965	4887

5.6 ECLP Product Shipments

Tables 5-4 to 5-7 summarize the disposal of naphtha, solvent, heavy vacuum gas oil (HVGO) and vacuum bottoms produced at ECLP. Additionally, 3474 barrels of HVGO were sent to the Exxon Baytown Refinery for blending with an asphalt stream; 21974 barrels of naphtha were sent to the Exxon Baytown Chemical Plant for furnace fuel and 30214 barrels of naphtha were sent to the Exxon Baytown Refinery for blending with catalytic reformer feed as a test.

6.0 ECLP Environmental Assessment

An environmental assessment of ECLP was completed in 1977 and has been documented in Environmental Assessment of the Exxon Coal Liquefaction Pilot Plant, Baytown, Texas, prepared by the United States Energy Research and Development Administration, September, 1977.

ECLP TERMINATION OF OPERATION

1.0 Dismantling

Following completion of the final ECLP turnaround, all toxic materials were removed from the plant and surplus material and equipment were disposed of. The plant was then turned over to a contractor for dismantling.

TABLE 5-4

SUMMARY OF SAMPLE SHIPMENTS OF ILLINOIS COAL ONCE-THROUGH PRODUCTS

Samples Shipped	Naphtha (Barrels)	Solvent (Barrels)	HVGO (Barrels)	Vacuum Bottoms (Tons)	Other (1) (Barrels)
Project	6.25	207.0	54.0	233.0	304.0*
EPRI	0.0	686.0	315.0	0.0	0.0
DOE	22.0	22.0	22.0	0.0	615.0**
Exxon	0.1	0.1	20.0	0.2	0.1***
Phillips	60.0	60.0	60.0	6.0	0.0
Anaconda	13.0	13.0	13.0	2.0	0.0
RAG	46.0	16.0	1.0	0.0	0.0
ENI	94.0	200.0	96.0	4.0	0.0
JCLD	0.0	500.0	225.0	0.0	0.0
TOTAL	241.35	1704.1	806.0	245.2	919.1

---

(1) Samples included under "Other":

* 69 Barrels of 600°F+ solvent for testing as a feed to a partial oxidation gasification process; 32 gallons of various streams for material testing; 220 barrels of fuel oil for PNA emission testing; 500 gallons of 350-430°F oil for engine tests.

** 275 gallons of T-303 No. 1 sidestream, 330 gallons of T-303 bottoms, 600 barrels of 750°F$^-$ solvent.

*** Five gallons of 750°F$^-$ solvent.

TABLE 5-5

SUMMARY OF SAMPLE SHIPMENTS OF ILLINOIS COAL BOTTOMS RECYCLE PRODUCTS

Samples Shipped	Naphtha (Barrels)	Solvent (Barrels)	HVGO (Barrels)	Vacuum Bottoms (Tons)	Other (1) (Barrels)
Project	246.5	542.0	0.1	306.4	6.0*
EPRI	0.0	4648.0(2)	0.0	160.0	0.0
DOE	0.0	4.4	0.0	0.0	0.0
JCLD	0.0	0.0	0.0	20.0	0.0
Exxon	15.0	40.0	0.0	0.0	0.0
Anaconda	0.0	65.0	0.0	0.0	0.0
TOTAL	261.5	5299.4	0.1	486.4	6.0

---

(1) Samples included under "Other":

* 220 gallons of hydrotreated creosote oil, one gallon of sour water from D-105, one gallon of solvent from Tray 23 of Fractionator T-201, nine gallons of LVGO, and six gallons of HAGO.

(2) Includes some Wyoming solvent.

TABLE 5-6

SUMMARY OF SAMPLE SHIPMENTS OF WYOMING COAL BOTTOMS RECYCLE PRODUCTS

Samples Shipped	Naphtha (Barrels)	Solvent (Barrels)	HVGO (Barrels)	Vacuum Bottoms (Tons)	Other (1) (Barrels)
Project	20.0	187.0	1.3	136.0	3.0*
JCLD	0.0	0.0	0.0	30 lbs.	0.0
EPRI	0.0	1976.0	0.0	0.0	0.0
ENI	13.0	26.0	0.02	0.2	0.0
Anaconda	6.5	6.5	0.0	0.8	0.0
Phillips	6.6	6.5	0.0	8.3	0.0
DOE	0.0	13.0	0.0	0.0	0.0
RAG	1.3	1.3	0.0	0.0	0.0
TOTAL	47.4	2216.3	1.32	145.3	3.0

---

(1) Samples included under "Other":

* One gallon of solvent from Tray 23 of Fractionator T-201, six gallons of LVGO, 2.6 barrels of hydrotreated creosote oil, 10 gallons of slop oil.

TABLE 5-7

SUMMARY OF SAMPLE SHIPMENTS OF TEXAS LIGNITE BOTTOMS RECYCLE PRODUCTS

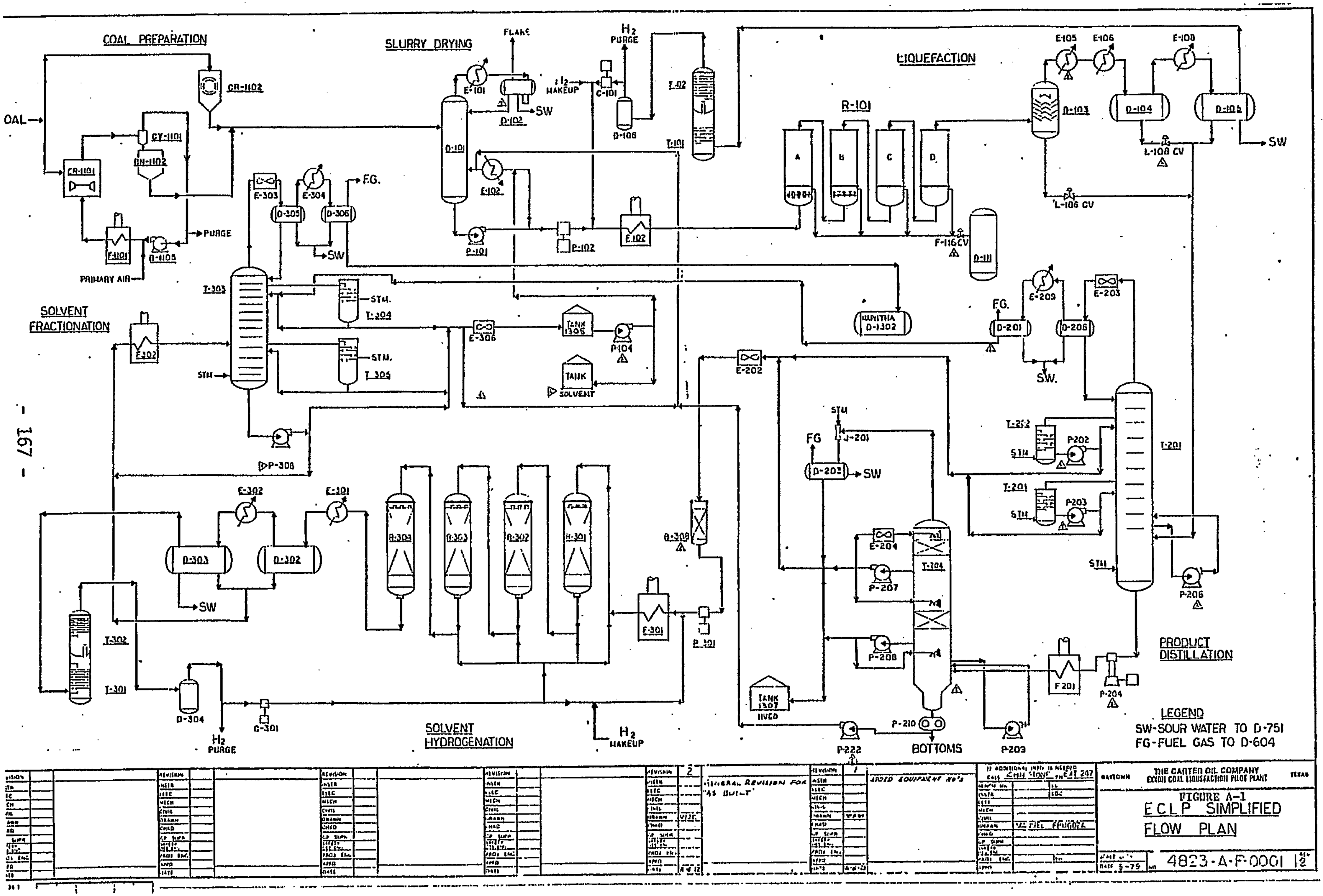
Samples Shipped	Naphtha (Barrels)	Solvent (Barrels)	HVGO (Barrels)	Vacuum Bottoms (Tons)	Other (1) (Barrels)
Project	13.1	172.4	0.0	32.0	5.0*
DOE	0.0	235.7	0.0	0.0	0.0
EPRI	0.0	5079.0(2)	0.0	0.0	0.0
RAG	1.3	1.3	0.0	0.0	0.0
Phillips	170.2	276.3	0.2	7.5	0.0
Anaconda	6.5	6.5	0.0	0.0	0.0
ENI	13.0	25.0	0.2	0.2	0.0
TOTAL	202.8	5796.2	0.4	39.7	5.0

---

(1) Samples included under "Other":

* 220 gallons of hydrotreated creosote oil.

(2) Also includes some solvent produced from Illinois and Wyoming coals and small amount of hydrotreated creosote.



LEGEND

SW-SOUR WATER TO D-751
FG-FUEL GAS TO D-604

FIGURE A-1
ECLP SIMPLIFIED
FLOW PLAN

APPENDIX B

This Appendix presents detailed tabulations of time on coal and causes for coal outages for each of the ECLP operating periods.

TABLE B-1

ECLP OPERATING HISTORY

ILLINOIS ONCE-THROUGH SHAKEDOWN

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative			Remarks
						Coal-In Hours	Total Hours	% Coal-In	
1	In	6/24/80	2135	-	-	-	-	-	-
	Out	6/30/80	0530	128	128	128	128	100	P-204, P-102, T-204 plugging.
2	In	7/10/80	1600	250	-	128	378	34	
	Out	7/31/80	2100	509	509	637	887	72	T-204 transfer line erosion.
3	In	8/15/80	1400	353	-	637	1,240	51	Includes 120 hours for hurricane plant shutdown; P-204 and P-210 plugging.
	Out	8/16/80	2200	32	32	669	1,272	53	
4	In	8/24/80	1600	186	-	669	1,458	46	
	Out	8/30/80	0600	134	134	803	1,592	50	FE-1104 failure.
5	In	9/9/80	0100	235	-	803	1,827	44	FE-1104 failure and P-210 problem.
	Out	9/9/80	2200	21	-	824	1,848	45	
	In	9/10/80	0600	8	-	824	1,856	44	
	Out	9/12/80	1400	56	77	880	1,912	46	P-210 motor loss.
6	In	9/16/80	2200	104	-	880	2,016	44	
	Out	9/20/80	0300	77	77	957	2,093	46	HVGO plugging.
7	In	9/24/80	1600	109	-	957	2,202	43	
	Out	9/25/80	0900	17	-	974	2,219	44	Instrument tubing failure.
	In	9/25/80	1300	4	-	974	2,223	44	C-701 recycle valve, Sandvik stopping, P-101 plug.
	Out	10/2/80	0100	156	173	1,130	2,379	47	
8	In	10/6/80	0900	104	-	1,130	2,483	46	
	Out	10/11/80	0400	115	115	1,245	2,598	48	D-103 carryover.

TABLE B-2

ECLP OPERATING HISTORY

ILLINOIS ONCE-THROUGH (TEST)

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative			Remarks
						Coal-In Hours	Total Hours	% Coal-In	
9	In	12/30/80	1000	-					
	Out	12/31/80	1500	29	29	29	29	100	Sandvik belt trip-out.
10	In	1/3/81	0100	58		29	87	33	
	Out	1/6/81	0600	77		106	164	65	RV-1101 failure.
	In	1/6/81	1300	7		106	171	62	
	Out	1/12/81	1800	149	226	255	320	80	P-102 block valve failure.
11	In	1/17/81	1900	121		255	441	58	
	Out	1/30/81	2400	317	317	572	758	75	F-102B hot spots.
12	In	2/2/81	1900	67		572	825	69	
	Out	2/4/81	2000	49	49	621	874	71	F-102A hot spots.
13	In	2/15/81	1000	254		621	1,128	55	
	Out	2/15/81	1600	6	6	627	1,134	55	P-204 suction refractory plug.
14	In	2/19/81	1200	92		627	1,226	51	
	Out	2/22/81	0400	64		691	1,290	54	Sandvik belt gear box failure.
	In	2/22/81	1200	8		691	1,298	53	
	Out	3/22/81	0200	662		1,353	1,960	69	F-102B hot spot.
	In	3/22/81	1000	8		1,353	1,968	68	
	Out	3/22/81	2200	12	738	1,365	1,980	69	Sandvik belt cracking.
15	In	4/1/81	1000	228		1,365	2,208	62	
	Out	4/17/81	2400	398	398	1,763	2,606	68	Hole in T-201 shell.

TABLE B-2 (Continued)

ECLP OPERATING HISTORY

ILLINOIS ONCE-THROUGH (TEST)--CONTINUED

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative			Remarks
						Coal-In Hours	Total Hours	% Coal-In	
16	In	4/24/81	1700	161		1,763	2,767	64	
	Out	4/26/81	0800	39	39	1,802	2,806	64	P-204 suction refractory plug.
17	In	4/28/81	0300	43		1,802	2,849	63	
	Out	5/8/81	1800	255		2,057	3,104	66	P-102 packing.
	In	5/8/81	2000	2		2,057	3,106	66	
	Out	6/2/81	2100	601	856	2,658	3,707	72	End of operation (planned).

TABLE B-3

ECLP OPERATING HISTORY

WYOMING COAL, BUTTOMS RECYCLE

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative			Remarks
						Coal-In Hours	Total Hours	% Coal-In	
Illinois									
18I	In	7/31/81	1500	-	168	168	168	100	Continuing--swung to Wyoming.
Wyoming									
18W	In	8/7/81	1500	-		168	168	100	Swung to Wyoming.
	Out	8/21/81	0800	329		497	497	100	FE-1104 plugging.
	In	8/21/81	1400	6		497	503	99	P-210 packing--actually out
	Out	8/27/81	0700	137		634	640	99	0700, 0800, 1100.
	In	8/27/81	1000	3		634	643	99	P-210 packing--actually out
	Out	8/29/81	1400	52		686	695	99	1400, 1500, 2200, 2300.
	In	8/29/81	1800	4		686	699	98	P-210 packing--actually out
	Out	8/30/81	1500	21		707	720	98	1500, 1900-2400.
	In	8/30/81	2100	6		707	726	97	P-210 packing-- actually out
	Out	8/31/81	0000	3		710	729	97	0000-0400, 1400, 1500.
	In	8/31/81	0600	6		710	735	97	RV-101 plugging--actually out
	Out	9/2/81	1400	56		766	791	97	1400-2300.
	In	9/2/81	2300	9		766	800	96	T-204 shed plug--due to
	Out	9/28/81	2200	623	1,321	1,389	1,423	98	solids withdrawal.
19	In	10/11/81	0100	291		1,389	1,714	81	F-102 swing/backflow of solids.
	Out	10/29/81	2200	453	453	1,842	2,167	85	F-102 swing/backflow of solids.
NOTE:	Excluding Illinois Portion:					1,674	1,999	84	

TABLE B-4

ECLP OPERATING HISTORY

ILLINOIS COAL, BOTTOMS RECYCLE

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative Coal-In Hours	Cumulative Total Hours	Cumulative % Coal-In	Remarks
20	In	11/28/81	1600	-					
	Out	12/2/81	2100	101		101	101	100	P-204 plugging.
	In	12/3/81	0500	8		101	109	93	
	Out	12/18/81	1600	371		472	480	98	H_2 loss from Baytown Refinery.
	In	12/18/81	2100	5		472	485	97	
	Out	1/11/82	0400	559	1,031	1,031	1,044	99	Freeze-ups--15°F.
21	In	1/15/82	1600	108		1,031	1,152	89	
	Out	1/16/82	1200	20	20	1,051	1,172	90	Freeze--voluntary.
22	In	1/17/82	1000	22		1,051	1,194	88	
	Out	1/29/82	0300	281	281	1,332	1,475	90	Plug between MC-101 and E-105.
23	In	1/31/82	0400	49		1,332	1,524	87	Terminated Illinois run (planned).
	Out	2/28/82	2400	692	692	2,024	2,216	91	

TABLE B-5

ECLP OPERATING HISTORY

WYOMING COAL, BOTTOMS RECYCLE

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative			Remarks
						Coal-In Hours	Total Hours	% Coal-In	
24	In	2/28/82	2400	-					
	Out	3/1/82	0600	6		6	6	100	Plugged barometric leg.
	In	3/1/82	0700	1		6	7	86	P-101/102 coal chunks plug impact.
	Out	3/3/82	1900	60		66	67	99	
	In	3/3/82	2300	4		66	71	93	
	Out	3/5/82	0700	32	98	98	103	95	Plug valves at P-102 failed.
25	In	3/8/82	1300	78		98	181	54	
	Out	3/10/82	0700	42	42	140	223	63	P-210 packing--replaced pump.
26	In	3/11/82	1100	28		140	251	56	
	Out	3/23/82	1400	291	291	431	542	80	Seal weld leak--L-108 bypass.
27	In	3/26/82	1200	70		431	612	70	
	Out	4/8/82	1600	316		747	928	80	Plugged screw feeder RV-101.
	In	4/9/82	1300	21		747	949	79	
	Out	4/9/82	1400	1	317	748	950	79	Plugged barometric leg.
28	In	4/10/82	1530	26		748	976	77	P-102 valve plug and crankcase failure.
	Out	4/11/82	0100	9	9	757	985	77	
29	In	4/15/82	0100	96		757	1,081	70	
	Out	4/19/82	1000	105	105	862	1,186	73	E-105 hole.
	Out	4/19/82	2400	14		862	1,200	72	Start turnaround.

TABLE B-6

ECLP OPERATING HISTROY

TEXAS LIGNITE COAL, BOTTOMS RECYCLE

Run	In/ Out	Date	Time	Hours	Run Coal-In Hours	Cumulative			Remarks
						Coal-In Hours	Total Hours	% Coal-In	
Wyoming									
30W	In	5/2/82	2200	-	218	218	218	100	Continuing--swung to lignite.
Lignite									
30L	In	5/12/82	0000	-		218	218	100	1.6/1.0/0.5 (5/14--1200).
	Out	5/22/82	1300	253	253	471	471	100	Cable failure to Substation 4.
31	In	5/31/82	1300	216		471	687	68	
	Out	5/31/82	2300	10		481	697	69	T-204 upset due D-108.
	In	6/1/82	1300	14		481	711	68	
	Out	6/3/82	0900	43	53	524	754	69	FE-1104 failure--E-102 plug.
32	In	6/4/82	1300	29		524	783	67	
	Out	6/6/82	0200	37	37	561	820	68	Liquefaction exotherm.
33	In	6/10/82	0200	96		561	916	61	1.6/1.0/0.5 (6/12--1700).
	Out	7/12/82	1400	780		1,341	1,696	79	Install screen--impact
	In	7/12/82	1700	3		1,341	1,699	79	Slurry drying.
	Out	7/18/82	1800	145	925	1,486	1,844	80	Explosion at CXI.
34	In	7/19/82	1700	23		1,486	1,867	80	1.2/1.0/0.4 (7/29--1000).
	Out	8/1/82	1400	309	309	1,795	2,176	82	Sandvik belt (CO-1901) failure aborted restart.
35	In	8/9/82	1000	188		1,795	2,364	76	
	Out	8/20/82	1200	266	266	2,061	2,630	78	THE END (PLANNED)
NOTE:	Excluding Wyoming Portion					1,843	2,412	76	
	TOTAL (EXCLUDING TURNAROUND)					10,692	14,518	74	
	TOTAL					10,692	18,878	57	

APPENDIX C

ECLP Tabulated Yield Period Data

This Appendix provides tabulated yield test data for ECLP during bottoms recycle operation on Illinois coal, Wyoming coal (second run) and Texas lignite

TABLE C-1

ECLP Yield Test Data

Feed Coal Analyses

Coal Type	Illinois Coal	Wyoming Coal	Texas Lignite
Composition, wt% feed coal			
● Moisture***	1.77	4.70	7.10
● Ash	10.08	9.77	13.33
● Dry ash-free coal*	88.15	85.53	79.57
Elements of dry coal, wt%			
● Carbon	69.44	67.07	63.99
● Hydrogen	4.95	4.80	4.63
● Oxygen*	9.62	17.90	16.70
● Nitrogen	1.11	0.92	1.32
● Sulfur	4.95	0.76	1.71
● Ash**	9.93	8.55	11.65
Ash Elements, wt% ash			
● P_2O_5	0.36	1.01	0.37
● SiO_2	49.00	34.29	40.47
● Fe_2O_3	20.47	7.79	7.40
● Al_2O_3	16.91	16.41	11.15
● TiO_2	1.00	1.11	1.19
● CaO	4.63	16.88	15.34
● MgO	0.95	4.33	3.98
● K_2O	2.12	0.55	0.32
● NaO	1.42	1.05	0.93
● SO_3	3.14	16.57	18.85

All properties normalized

*By difference
**Does not include SO_3
***After drying in gas-swept mill

TABLE C-2

ECLP Yield Test Data

Vacuum Bottoms Analyses

Coal Type	Illinois Coal	Wyoming Coal	Texas Lignite
Composition, wt% bottoms			
• Ash	21.53	22.22	28.15
• Ash-free bottoms	78.47	77.78	71.85
Elements of bottoms, wt%			
• Carbon	68.55	68.99	63.21
• Hydrogen	4.15	3.94	3.73
• Oxygen*	2.61	4.97	5.05
• Nitrogen	1.20	0.89	1.28
• Sulfur	2.66	0.51	0.94
• Ash**	20.83	20.70	25.79
Ash Elements, wt% ash			
• P_2O_5	0.43	1.03	0.41
• SiO_2	47.99	41.68	46.98
• Fe_2O_3	21.13	6.08	7.24
• Al_2O_3	16.42	19.11	13.28
• TiO_2	0.96	1.22	1.40
• CaO	5.50	17.68	16.43
• MgO	0.94	4.51	4.43
• K_2O	2.05	0.64	0.39
• NaO	1.31	1.18	1.06
• SO_3	3.27	6.87	8.38
Viscosity @ 550°F, 21 sec^{-1}, poise	19.48	28.53	19.37
1000°F^- content, wt% (by microlube distillation)	6.1	5.6	10.6

All properties normalized

*By difference
**Does not include any SO_3

TABLE C-3

ECLP Yield Test Data

Naphtha Analyses

Coal Type	Illinois Coal	Wyoming Coal	Texas Lignite
Composition, wt%			
● Carbon	85.40	85.34	84.93
● Hydrogen	12.70	12.65	12.97
● Oxygen*	1.55	1.81	1.79
● Nitrogen	0.13	0.15	0.18
● Sulfur	0.22	0.05	0.13
Specific gravity @ 60°F	0.8217	0.8204	0.8050
Distillation**			
IBP, °F	112	90	93
5 LV %, °F	168	150	148
10 LV %, °F	192	176	178
20 LV %, °F	224	214	214
30 LV %, °F	253	244	243
40 LV %, °F	279	273	267
50 LV %, °F	307	302	291
60 LV %, °F	330	328	314
70 LV %, °F	349	350	332
80 LV %, °F	371	373	354
90 LV %, °F	390	394	379
95 LV %, °F	407	413	395
FBP, °F	435	444	425

*By difference

**D-86 data corrected to a true boiling point basis

TABLE C-4

ECLP Yield Test Data

Solvent Analyses

Coal Type	Illinois Coal	Wyoming Coal	Texas Lignite
Composition, wt%			
• Carbon	89.36	89.72	89.05
• Hydrogen	9.59	9.62	10.22
• Oxygen*	0.62	0.47	0.57
• Nitrogen	0.24	0.19	0.16
• Sulfur	0.19	0.003	0.004
Donor Hydrogen, wt%	1.68xRV	1.72xRV	1.91xRV
Specific gravity at 60°F	0.9928	0.9807	0.9549
Distillation**			
IBP, °F	322	380	368
5 LV%, °F	415	438	418
10 LV%, °F	433	453	437
20 LV%, °F	462	479	461
30 LV%, °F	492	507	488
40 LV%, °F	531	538	509
50 LV%, °F	572	571	535
60 LV%, °F	616	613	576
70 LV%, °F	669	659	625
80 LV%, °F	731	712	699
90 LV%, °F	826	810	813
95 LV%, °F	897	872	909
FBP, °F	979	948	1002

*By Difference
**D-1160 data corrected to a true boiling point basis.

TABLE C-5

ECLP Yield Test Data

Sour Water Analyses

Coal Type	Illinois Coal		Wyoming Coal		Texas Lignite	
Contaminants in water make*, ppm	LDU	SHU	LDU	SHU	LDU	SHU
H_2S	8569	10105	4069	1817	5557	38675
NH_3	3690	5178	2833	5081	2964	6275
CO_2	5100	11700	18600	800	27900	6400
Contaminants in other process water streams, ppm						
H_2S	----		40		10	
NH_3	----		--		41	
CO_2	----		30		2100	
Atmospheric Fractionator						
• Reflux Drum						
H_2S	1254		90		250	
NH_3	1326		56		1338	
CO_2	200		20		2300	
• Distillate Drum						
H_2S	13000		5060		4700	
NH_3	4114		2807		1569	
CO_2	1300		7200		10400	
Vacuum Fractionator						
H_2S	595		290		180	
NH_3	88		1808		68	
CO_2	100		900		2000	
Solvent Hydro Fractionator						
• Reflux Drum						
H_2S	9680		----		----	
NH_3	6358		----		----	
CO_2	340		----		----	
• Distillate Drum						
H_2S	11400		1930		6890	
NH_3	7290		2871		3554	
CO_2	400		200		3800	

*High-pressure cold separators

TABLE C-6

ECLP Yield Test Data

Feed/Product Rates

Coal Type	1/28/82 Illinois Coal		3/22/82 Wyoming Coal		6/23/82 Texas Lignite	
Feed Rates, lb/hr	Raw[+]	DRS[++]	Raw[+]	DRS[++]	Raw[+]	DRS[++]
• Coal[***]	12517	11882	11185	11710	13034	12745
• Make up hydrogen gas	1172	1248	787	717	752	762
TOTAL	13689	13130	11972	12427	13786	13507
Product Rates, lb/hr						
• Purge Gas						
- LDU	UNM	790	538	523	733	664
- SHU	286	299	67	67	124	117
• Fuel Gas						
- LDU	1287	1339	1206	1260	1496	1515
- SHU	182	184	100	96	151	151
• Acid Gas[*]	269	263	506	452	457	441
• Sour Water [**]						
- LDU	1098	1308	1616	1830	1924	2193
- SHU	UNM	279	UNM	344	UNM	230
• Naphtha	2030	2043	2292	2376	1683	2014
• Solvent	950	1148	973	1094	833	1031
• Bottoms	5904	5607	4599	4744	5750	5491
• Process Drum Level Changes	~(130)	(130)	(368)	(368)	~(340)	(340)
TOTAL	~12945	13130	11873	12427	13041	13507
Balance Closure (Products/Feed) x 100	94.6	100.0	99.2	100.0	94.6	100.0

[+] As measured rates

[++] As corrected by the Data Reconciliation System computer program

[*] That fraction of the acid gas which was dissolved in water, assumed to be 58%

[**] Process Water rates discounted

[***] As received from the Gas-Swept Mill Drier.

UNM = Unmeasured. DRS values used in summing totals in Raw column.

TABLE C-7

ECLP Yield Test Data

Operating Conditions and Yields

Coal Type	Illinois Coal	Wyoming Coal	Texas Lignite
LDU Operating Conditions			
Average Liquefaction Temperature, °F	846	855	845
Pressure, psig	2520	2493	2512
Nominal Residence time, minutes	81	85	88
Treat Gas			
• Rate, KSCFD	3277	3897	3463
• H_2 purity, mole %	80	86	84
Solvent-to-Coal Ratio	1.6	2.0	1.55
Bottoms-to-Coal Ratio	0.6	0.5	0.5
Solvent Quality Index	3.41	4.37	3.75
SHU Operating Conditions			
EIT**, °F	112+RV	93+RV	99+RV
Pressure, psig	1845	1798	1827
Weight Hourly Space Velocity, hour^{-1}	1.66xRV	1.96xRV	1.58xRV
Treat gas			
• Rate, SCF of H_2/Bbl of feed solvent	0.77xRV	0.74xRV	0.85xRV
• Gas purity, mole % H_2	81	98	93
Yields, wt % DAF Coal*	1/28/82	3/22/82	6/23/82
H_2	-4.8	-4.9	-4.5
H_2O	10.1	12.9	12.0
H_2S	4.3	0.6	1.5
NH_3	0.5	0.6	0.8
CO	0.3	0.6	0.9
CO_2	1.3	5.1	5.9
CH_4	4.0	4.0	4.1
C_2	3.2	3.5	3.3
C_3	3.8	2.9	2.9
C_4	1.6	1.5	1.5
C_5/400 °F	21.7	23.5	22.4
400/700 °F	8.8	10.3	7.9
700/1000 °F	6.0	5.4	7.5
1000 °F$^+$	39.2	34.0	33.8
Naphtha	22.2	24.8	22.7
Solvent	10.3	11.1	9.2

*Does not include SO_3

**Equivalent Isothermal Temperature

LIST OF ABBREVIATIONS

API	American Petroleum Institute
APS	Atmospheric Pipe Still
ART	Actual (Slurry) Residence Time
ASTM	American Society for Testing and Materials
BARD	Baytown Research and Development Division (Baytown, TX)
B/CD	Barrels per Calendar Day
B/SD	Barrels Per Stream Day
BTX	Benzene Toluene Xylenes
CCLU	Continuous Coking Liquefaction Unit
C-E	Combustion Engineering, Inc.
CLPP	Coal Liquefaction Pilot Plant (One Ton Per Day Unit)
Co/Mo	Cobalt/Molybdenum
COD	Chemical Oxygen Demand
COT	Coal Outlet Temperature
CSCU	Continuous Stirred Coking Unit
DAF	Dry, Ash-Free
DBM	Design Basis Memorandum
DC	Direct Combustion
DCF	Discounted Cash Flow
DEA	Diethanolamine
DMMF	Dry Mineral Matter-Free
DOE	Department of Energy
DRS	Data Reconciliation System
DSC	Differential Scanning Calorimetry
ECLP	EDS Coal Liquefaction Pilot Plant (250 Tons Per Day)

LIST OF ABBREVIATIONS (Continued)

EDS-FPU	EDS - FLEXICOKING Prototype-Unit
EEPM	Exxon Engineering Project Management
EETD	Exxon Engineering Technology Department (Florham Park, NJ)
EOR	End-of-Run
EPA	Environmental Protection Agency
EPRI	Electric Power Research Institute
ERDL	Exxon Research and Development Laboratories (Baton Rouge, LA)
ER&E	Exxon Research and Engineering Company
FBG	Fluid Bed Gasifier
FBU	Fluidized Bed Unit
FCBU	Fluid Coker Bogging Unit
FSBF	Full Scale Burner Facility
FTIR	Fourier Transform Infra Red
GC	Gas Chromatography
GPM	Gallons Per Minute
GSM	Gas Swept Mill
HB	Hybrid Boiler
HBG	High Btu Gas
HCO	Hydrogenated Cresote Oil
HVGO	Heavy Vacuum Gas Oil
IBG	Intermediate Btu Gas
IKG	Integrated Coking/Gasification Pilot Plant
k	Thousand
LBG	Low Btu Gas
LDU	Liquefaction/Distillation Unit

LIST OF ABBREVIATIONS (Continued)

LHV	Lower Heating Value
LP	Linear Program
LPG	Liquefied Petroleum Gas
LSCU	Large Stirred Coker Unit
LSFO	Low Sulfur Fuel Oil
M	Million
MBG	Moving-Bed Coal Gasification
MPSS	Multi-Pass Spent Solvent
NDIR	Non-Dispersive Infrared
NDT	Non-Destructive Testing
Ni/Mo	Nickel Molybdenum
NPSH	Net Positive Suction Head
NRT	Nominal Residence Time
OTCLU	Once-Through Coal Liquefaction Unit
PACT	Powered Activated Carbon Treatment
PAM	Process Alternatives LP Model
PDA	Process Development Allowance
P&ID	Piping and Instrument Diagram
PNA	Polynuclear Aromatics
POX	Partial Oxidation
PRD	Product Research Division (Linden, NJ)
psig	pounds per square inch, gauge
RCLU-1	Recycle Coal Liquefaction Unit with Nominal Coal Feed Rates of 75 Pounds-Per-Day

LIST OF ABBREVIATIONS (Continued)

RCLU-2	Recycle Coal Liquefaction Unit with Nominal Coal Feed Rates of 75 Pounds-Per-Day
RCO	Raw Creosote Oil
RISP	Required Initial Selling Price
RV	Reference Value - Code to prevent disclosure of non-program data.
SASS	Source Assessment Sampling System
S/C	Solvent/Coal
S/C/B	Solvent/Coal/Bottoms
SCF	Standard Cubic Feet
SDU	Study Design Update
SEM	Scanning Electron Microscope
SFEU	Solid Fuels Evaluation Unit
SFU	Small Fluidized Bed Unit (for coke gasification)
SHU	Solvent Hydrogenation Unit
SOR	Start-of-Run
SQI	Solvent Quality Index
TDS	Total Dissolved Solids
T/D	Tons Per Day
TEC	Total Erected Cost
TGA	Thermal Gravimetric Analysis
TIR	Temperature Increase Required
TMA	Thermomechanical Analysis
TOC	Total Organic Carbon
T/SD	Tons Per Stream Day
UOP, Inc	Union Oil Products, Inc.

LIST OF ABBREVIATIONS (Continued)

VB	Vacuum Bottoms
VGO	Vacuum Gas Oil
VLE	Vapor Liquid Equilibrium
VPS	Vacuum Pipe Still
V/Hr/V	Volumetric Hourly Space Velocity, ft^3 feed per hour per ft^3 of reactor volume.
WWT	Wastewater Treatment
YP	Yield Period

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