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EXXON CATALYTIC COAL GASIFICATION PROCESS DEVELOPMENT PROGRAM

MASTER

QUARTERLY TECHNICAL PROGRESS REPORT
FOR THE PERIOD
January 1 - March 31, 1979

EXXON RESEARCH AND ENGINEERING COMPANY

Baytown, Texas 77520

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY UNDER
Contract No. ET-78-C-01-2777

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Quarterly Technical Progress Report
For the Period
January 1 - March 31, 1979

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Exxon Research and Engineering Company
Baytown, Texas 77520

May, 1979

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ABSTRACT

This report covers the activities for the Exxon Catalytic Coal Gasification Development Program during the quarter January 1 - March 31, 1979. This work is being performed by the Exxon Research and Engineering Company (ER&E) and is supported by the United States Department of Energy under Contract No. ET-78-C-01-2777.

The highlights of this report are summarized below:

- Bench Scale Research and Development

Initial results from the one-gallon digester show total potassium recoveries of over 90% with less than one hour residence time as compared with two hours used in the Catalytic Coal Gasification Study Design.

Preliminary runs have been made using the single tank leaching unit which show that large amounts of digested char (>25%) are carried out of leaching tanks operating at reasonable superficial velocities. Water washed char did not carry over to this extent.

Char combustion screening studies were completed. Results show that bulk density of the char does increasing during combustion and both sulfur and potassium may be quantitatively retained on the char under selected combustion conditions.

Studies were initiated to determine the effects of variable pH and potassium ion concentration on the amount of catalyst remaining on the char.

Construction of a bench apparatus to study reactions of product and recycle gas in furnace and heat exchanger tubes was completed and checkout of the apparatus was begun. The computer program to analyze the data from the unit was written and debugged.

- Process Development Unit Operations

A Startup and Initial Operation Schedule, a Checkout Test Plan, and an Initial Startup Plan were developed for the Process Development Unit (PDU). The PDU will be started up in a sequential manner, with the gasification system being started up on a once-through basis first. The gas separation system will be started up next, followed by the catalyst recovery system. A three-day Operator Training School was completed. Coal was successfully circulated through the coal handling system. The automatic coal feed system is undergoing initial testing, and fluidization tests are being performed on the coal that has been fed to the gasifier.

The programmable controller, which handles valve sequencing, alarming, and other miscellaneous functions on the PDU, was programmed and checkout was completed on the coal feed, gas feed, and filter systems.

Char production operations in the Fluid Bed Gasifier (FBG) were completed. Sufficient char was made for PDU startup as well as for catalyst recovery process variable experiments and bench scale kinetic studies. The degree of oxidation of the coal is believed to be the major reason for the difference in char density observed during the present FBG operations and those under the predevelopment program (Contract No. E(49-18)-2369).

Work continued on defining the cause of the breakdown of char and lime during digestion in the prototype catalyst recovery unit. It was concluded that both the lime and char particles are fragile and will break down to fines if handled roughly. Removal of the potassium from the char by water washing does not cause the char particles to disintegrate. Modifications were made to the digester to provide better mixing than before and to minimize particle breakdown.

The preferred processing sequence for catalyst recovery in the PDU has been identified. It consists of two stages of mixing tanks and solid-liquid separation devices in series. This system will be capable of operating in a water wash mode or a digestion followed by water wash mode. In the digestion mode a 90% catalyst recovery should be achieved for recycle back to the catalyst addition unit. The high recycle rate will allow the detection of buildup of trace compounds in the catalyst recycle loop.

- Data Acquisition and Correlations

A series of experiments were carried out in the cold model to evaluate the performance of the solids feed and the fines return systems of the PDU. Recommendations were made regarding purge locations and gas rates through them. Modifications have been made to the cyclone and the intersection block of the PDU as a result of these studies.

The off-line data reconciliation program for the PDU has been developed, debugged, and tested using a set of simulated data. This program provides a tool for obtaining consistent and reliable data from PDU operations.

- Advanced Study of the Exxon Catalytic Coal Gasification Process

A replacement gas chromatograph system has been purchased for the fixed-bed bench scale gasification unit. Delivery of the replacement unit will be in June. Kinetic data will be obtained in a mini-fluid bed gasification unit until the installation of the new gas chromatograph.

Bench scale tests confirmed that the change in catalyst from K_2CO_3 to KOH was not responsible for the differences in fluidized bed densities between the present and the predevelopment operations of the FBG. Tests also showed that uniform catalyst impregnation of particles larger than 20 mesh eliminated their tendency to agglomerate. Work was initiated to determine a sensitive measure of the oxidation level of the coal.

- Engineering Research and Development

Work was completed on a revised offsites facilities definition and cost estimate to update the CCG Commercial Plant Study Design prepared during the predevelopment program. This update includes a more detailed study of water treatment and reuse options and a flue gas desulfurization (FGDS) study design for a lime scrubbing system. For a pioneer commercial plant feeding Illinois No. 6 coal and producing 257 billion Btu/SD SNG, the updated investment estimate is 1,530 M$ and the updated gas cost is 6.18 $/MBtu. These economics are on a January, 1978 cost basis, and reflect 100% equity financing and a 15% current dollar DCF return. The investment is down 7% and the gas cost is down 4% from the Predevelopment Program Study Design. Revised cost estimating tools for materials handling equipment and the use of lime scrubbing for FGDS were the main factors leading to cost reductions.

The Coal Devolatilization Impact Study has been completed. Results indicate gasifier volume savings can be achieved if devolatilization yields are higher than those assumed for the CCG Study Design. Gasifier volume savings can also be achieved by feeding the coal higher in the gasifier bed. Significant debits would be incurred if higher feedpoints resulted in the production of tars or hydrocarbons heavier than CH_4 in the gasifier effluent.

A laboratory guidance study has been made to estimate the economic impact of evaporating dilute catalyst solutions from catalyst recovery to concentrations suitable for direct addition to the gasifier feed coal. The incremental gas cost for concentrating a 10% (wt) KOH solution is about 0.12-0.19 $/MBtu, only 2-3% of the CCG Study Design gas cost. The use of dilute catalyst solutions will reduce the number of stages required for catalyst recovery. Studies will be conducted later in the program to find the optimum balance between evaporation costs and catalyst recovery investment.

Work continued on the engineering process improvement study to evaluate an integral steam reformer to provide heat input for the gasifier. Initial evaluations indicate incentives for operating at high reformer coil outlet temperature of 1500°F and high steam conversion of 49%.

A brief incentive study of an alternative two-stage gasification concept was completed during the first quarter. Process economics indicate an incentive for staged gasification, but additional laboratory data are required to develop a better estimate of the incentive.

Systems modeling continued with the objective of developing material and energy balance tools which will reduce the effort required to do engineering studies under the present contract. A material balance model for the catalyst recovery system was completed. Work is continuing on the development of a material and energy balance model for the CCG reactor system.

Activities continued in the engineering technology programs:

- A detailed summary of the proposed materials evaluation program for the PDU was completed. The eight corrosion racks to be installed in the PDU were fabricated and assembled.
- Experimental data needs were defined for vapor-liquid equilibria in sour water/catalyst systems.
- Important physical and thermodynamic properties of catalyst solutions were identified and a literature search is underway.

- Evaluation of alternatives for solid-liquid separations in catalyst recovery is continuing. Laboratory filtration equipment is under construction and a second laboratory unit has been designed to quantify the attritability of char particles.

- A program has been developed to obtain and characterize liquid and solid waste samples from the PDU to identify potential effluent quality problems in a commercial plant.

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LABORATORY PROCESS RESEARCH AND DEVELOPMENT

1. Bench Scale Research and Development (Reporting Category C01)

1.1 Catalyst Recovery Studies

Construction of three bench units described in the October-December, 1978 Quarterly Report has been completed and startup has begun. These units are a one-gallon char-lime digester, a char leaching system, and a fluidized bed char combustion reactor. Process variable data from the digestion and leaching systems has been obtained, and screening studies have been completed on the char combustion reactor.

Digestion

Construction of the one-gallon char-lime digester described in the October-December, 1978 Quarterly Report was completed in February. This unit can heat its slurry contents from 150°F (below digestion reaction temperatures) to 300°F in 3 minutes and to 400°F in approximately 20 minutes. The reactor will cool down from 400°F to 150°F in 15 minutes. Reactor temperature and residence time can therefore be measured more accurately than was possible in the bigger 100 gallon prototype catalyst recovery unit. The digester is also designed to investigate effects of agitation and lime particle size on potassium recovery from char.

Table 1.1-1 presents initial process variable results from this unit. The following preliminary observations may be made based on these data:

- A potassium recovery of 92% is possible at a digestion temperature of 325°F, 15 minutes residence time, and Ca/K molar ratio of 1.06 with continuous agitation as shown by Run A.
- Digestion at 400°F with no agitation yields total potassium recoveries of 95% and 96% (Runs C and D). Therefore, agitation may not be necessary for potassium solubilization.

Figure 1.1-1 is a particle size distribution of the char/lime solids before and after digestion for Run C. The plots show that the weight of particles smaller than 10 microns increases from 25% to 43% during digestion. These fine particles are presently being analyzed to determine the factors accounting for the increase in fine particles during digestion.

Leaching

Single tank leaching runs were made to generate fundamental information to be used in estimating the performance of the six-stage leaching unit discussed in the October-December, 1978 Quarterly Report. Data from the six-stage unit will be used to assist in designing leaching tanks for the PDU catalyst recovery system. Figure 1.1-2 is a diagram of the single tank leaching unit. Figures 1.1-3 through 1.1-5 present the results of runs made in this unit.

Table 1.1-1

Digestion Process Variable Results

Run	Ca/K	Reactor Temperature, °F	Reactor Residence Time, min.	Agitation	Lime Mesh Size	Overall K^+ Recovery, % (wt)
A	1.06	325°	15	225 RPM*	20 x 50	92.0
B	1.06	325°	30	225 RPM*	20 x 50	93.6
C	1.03	400°	30	No Agitation	-325	95.0
D	1.05	400°	60	No Agitation	20 x 50	96.0

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* During time of reaction

FIGURE 1.1-1

PARTICLE SIZE DISTRIBUTION OF RUN C BEFORE AND AFTER DIGESTION

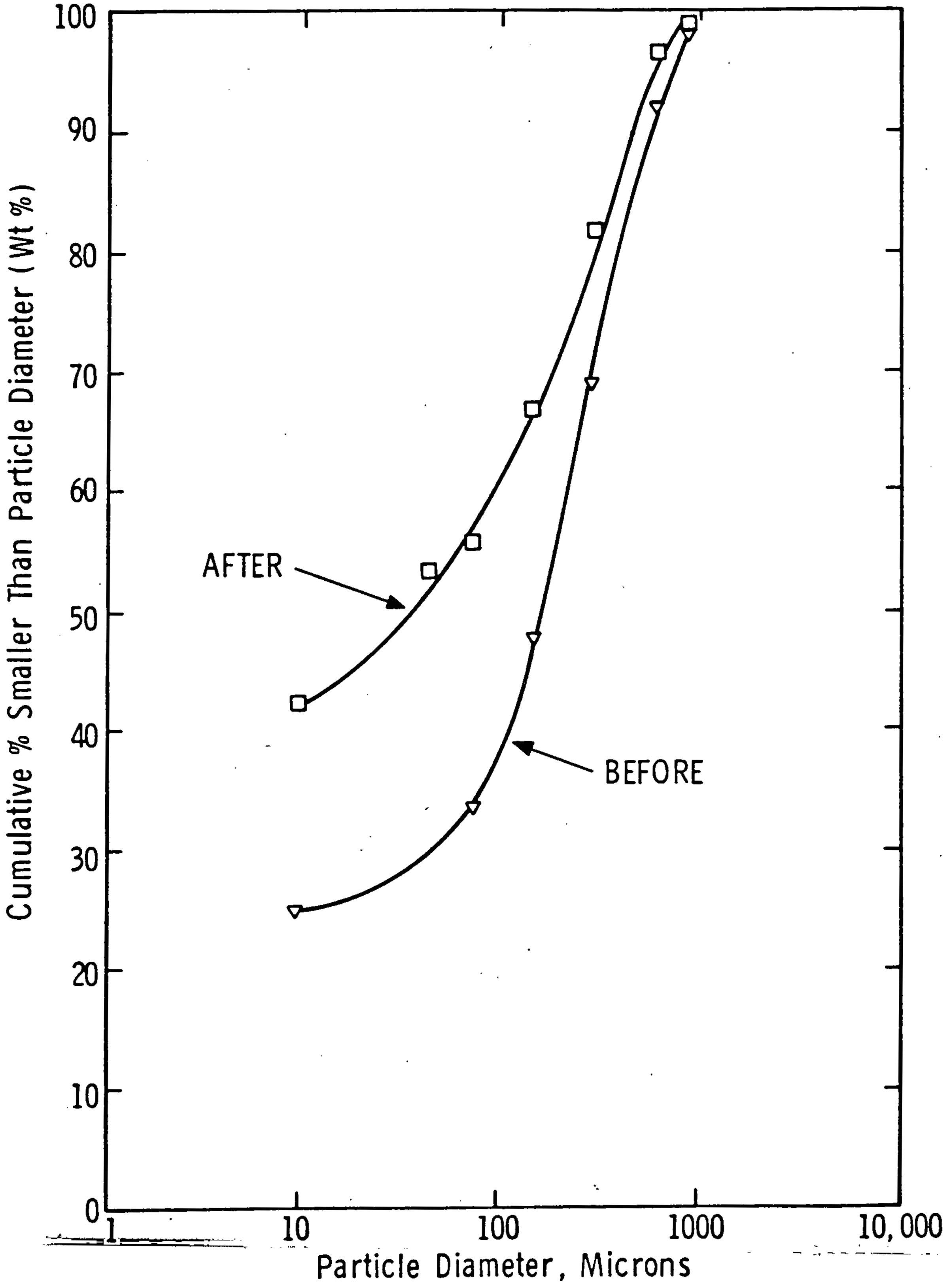
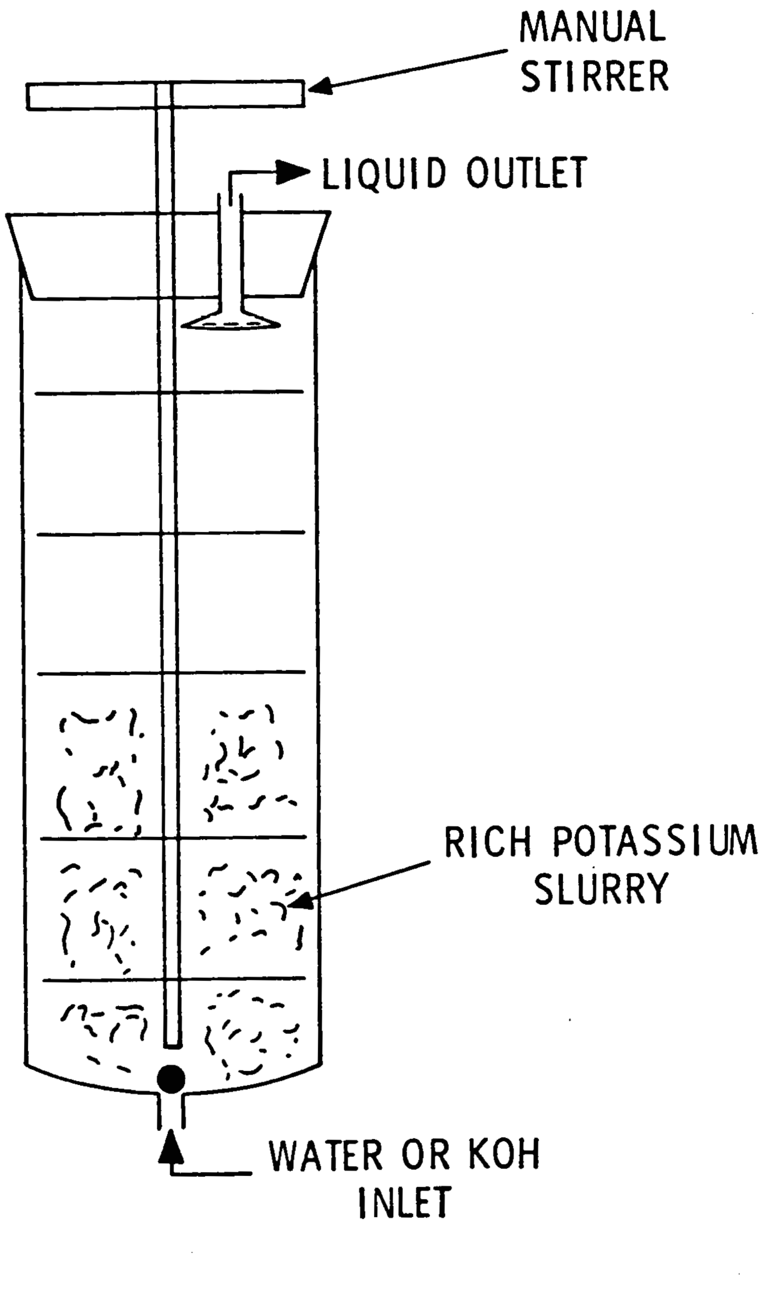


FIGURE 1.1-2

SINGLE TANK LEACHING UNIT



The material used to generate Figures 1.1-3 and 1.1-4 was char-lime solids from digestion runs. These figures show the solids carryover increased with increased potassium concentration in the leaching liquid. Increasing solids concentration in the slurry between 8-15% solids appears to have a minor effect on solids carryover. Figure 1.1-5, a run with undigested char, shows a great reduction in the char carryover at comparable superficial velocities. This could be because digestion produces smaller particle size material which overflows to a greater extent than the larger undigested char particles. A second possible explanation for the reduced char carryover for the runs in Figure 1.1-5 is that water was used for leaching.

Combustion

Fluidized bed char combustion screening studies were completed in February. These studies were undertaken to determine the feasibility of burning char prior to leaching to enhance catalyst recovery by increasing the char bulk density.

Table 1.1-2 presents representative data which show that the char bulk density does increase during combustion. Combustion was at a temperature below 1800°F in all cases. As can be seen from the table, the bulk density of the char particles increased 0.3-0.4 g/cc during combustion.

Table 1.1-2

Char Bulk Density (g/cc)

Before Combustion	After Combustion
0.43	0.85
0.47	0.78
0.50	0.90
0.60	0.96

Sulfur and potassium retention on combusted char has also been determined. Table 1.1-3 is a summary of the results. Sulfur is retained on combusted char at temperatures as high as 1600°F. These results may indicate that flue gas desulfurization would not be needed for a char burner. Potassium retention on Illinois No. 6 combusted char was found to be dependent on initial combustion temperature. When initial char combustion occurs at temperatures less than 1270°F, over 90% of the potassium on char is retained for most runs. However, when initial combustion occurs at 1450°F or higher, only 60-70% of the potassium present on the char is retained on the combusted char.

FIGURE 1.1-3

LEACHING TANK CARRYOVER AT 8% SOLIDS LOADING

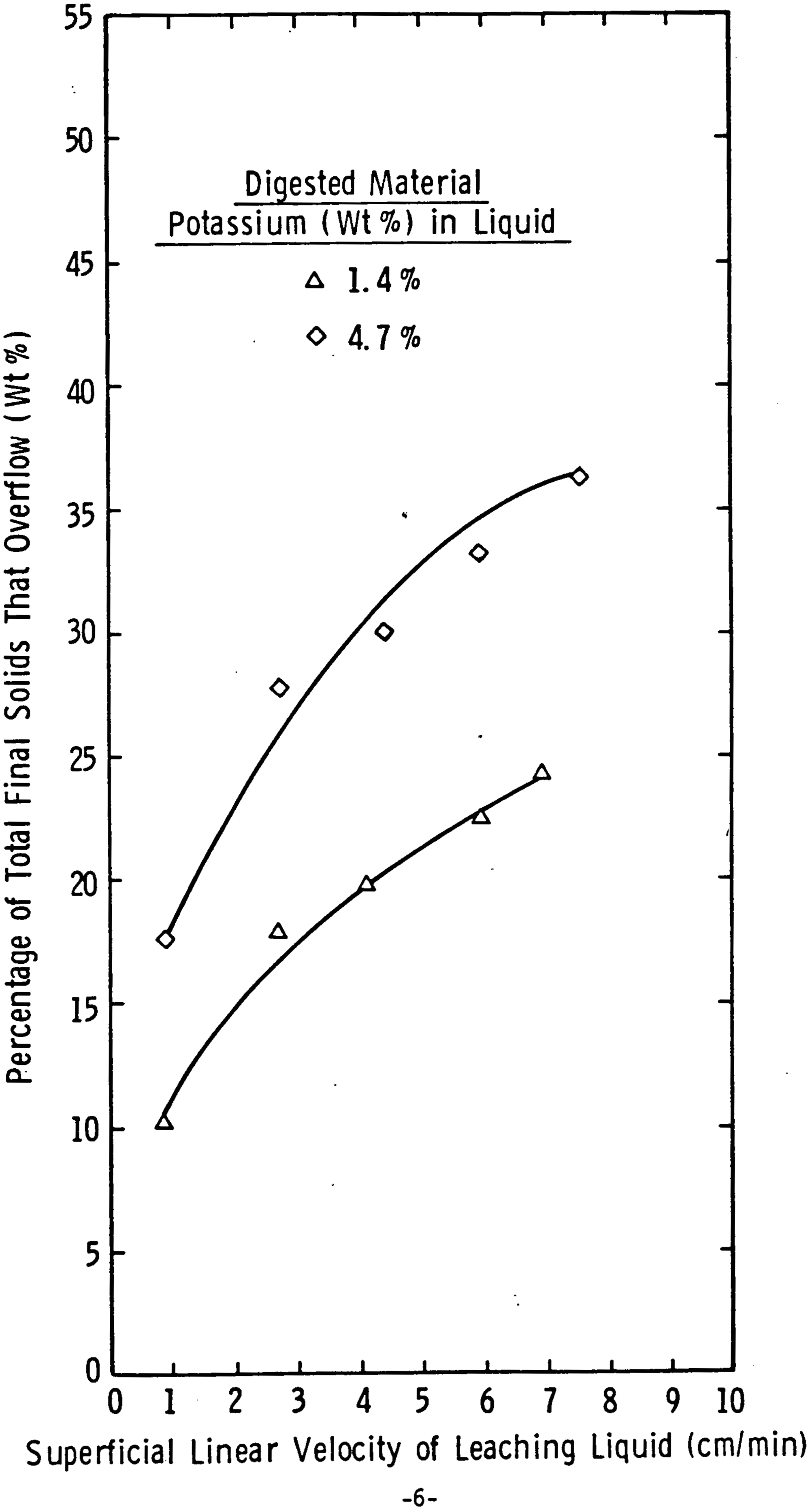


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FIGURE 1.1-4

LEACHING TANK CARRYOVER AT 15% SOLIDS LOADING

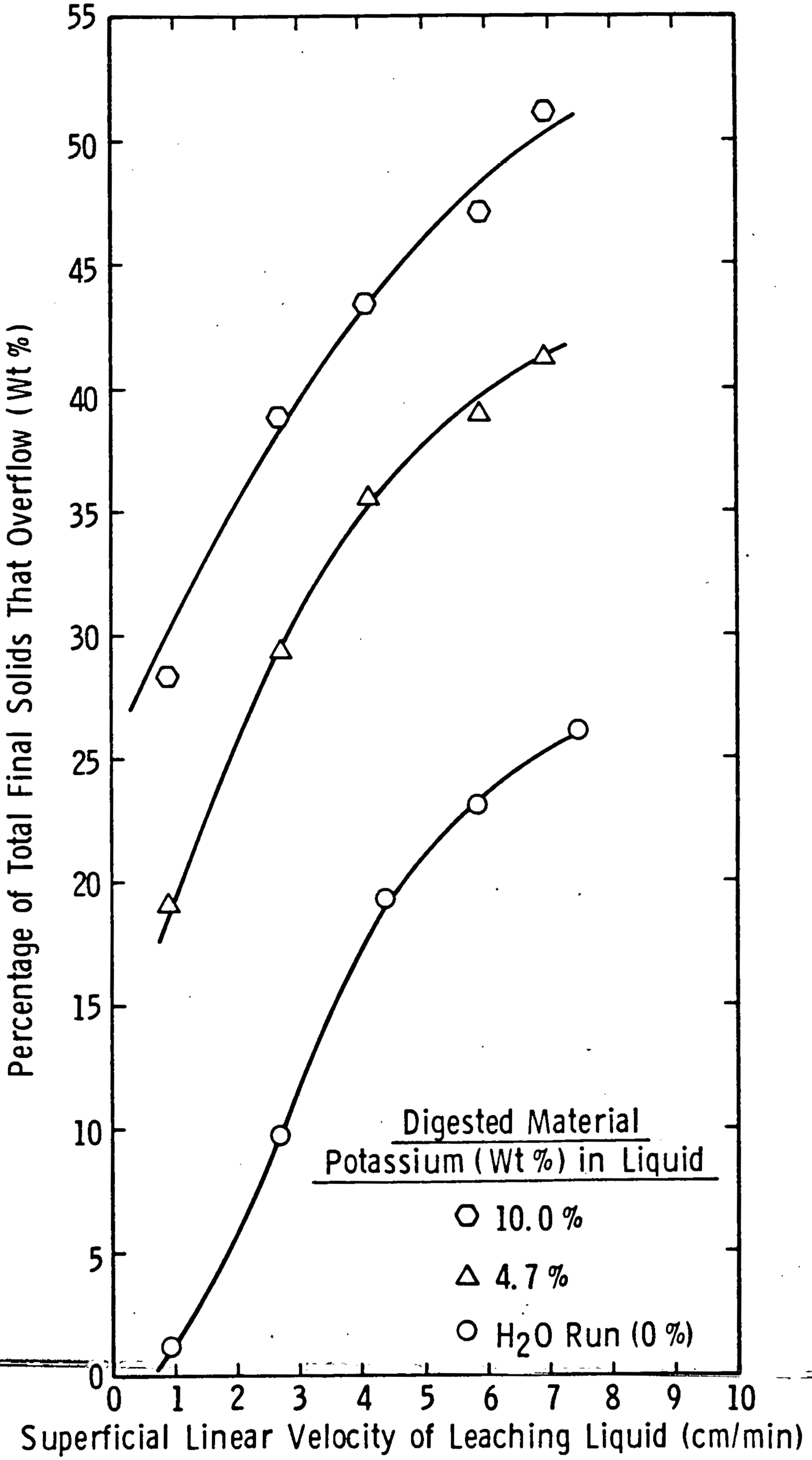


FIGURE 1.1-5

LEACHING TANK CARRYOVER AT 10 % SOLIDS LOADING

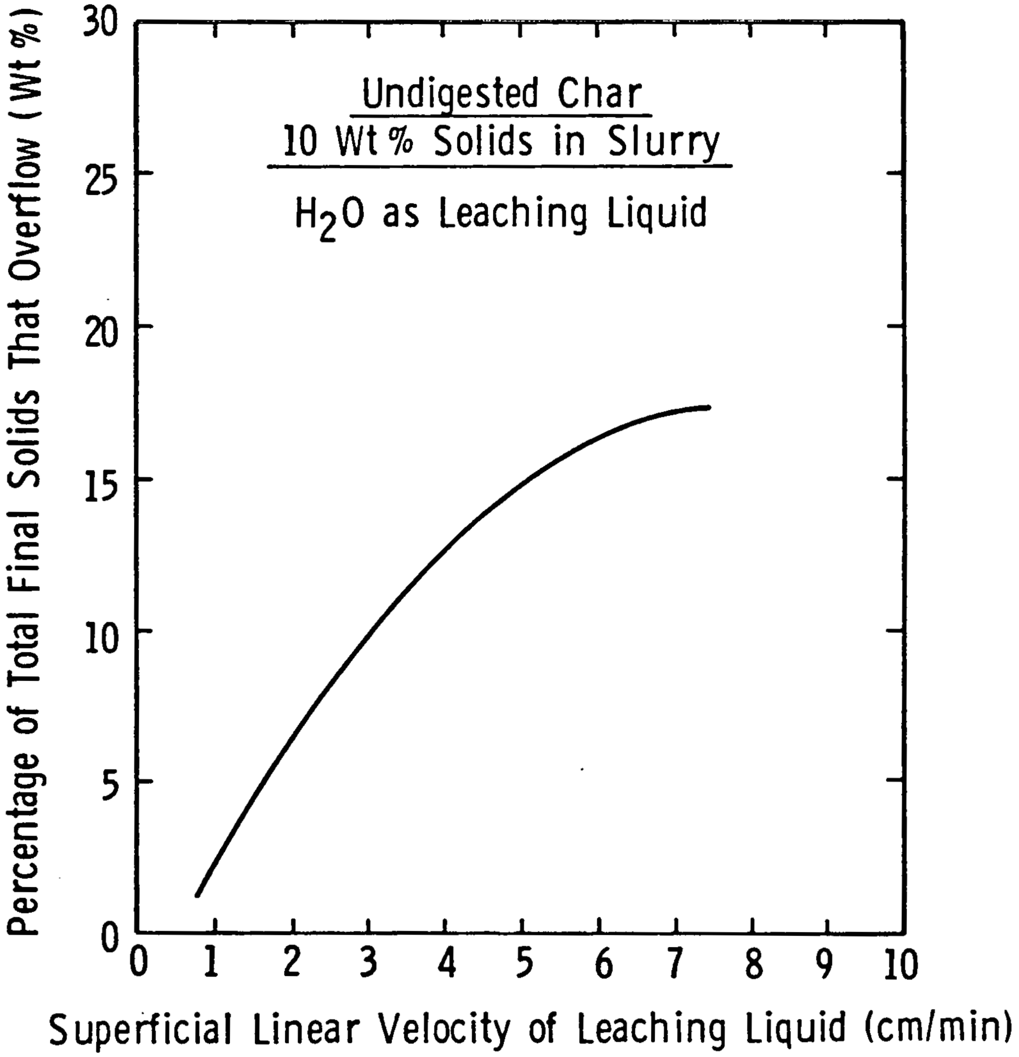


Table 1.1-3

Sulfur and Potassium Retention During Char Combustion

Initial Temp. (°F)	Final Temp. (°F)	% (wt) Sulfur Retained	% (wt) Potassium Retained
1250	1550	100	99
1250	1550	100	91
1250	1600	90	88
1250	1760	96	99
1270	1550	100	95
1450	1500	100	63
1500	1500	100	69

Future work will focus on the following areas:

- Completion of the single tank leaching studies of digested char-lime slurries.
- Single tank leaching studies using undigested char.
- Additional digestion runs to investigate the theory that digestion reactions produce fine particles.

1.2 Catalyst/Char Equilibrium Studies

The proposed PDU catalyst recovery procedure uses a batch counter-current multiple-contact leaching system (October-December, 1978 Quarterly Report). In this system, the solids produced by hydrothermal digestion of spent gasifier char are subjected to contacting with aqueous leaching solutions of varying catalyst (potassium ion) concentrations.

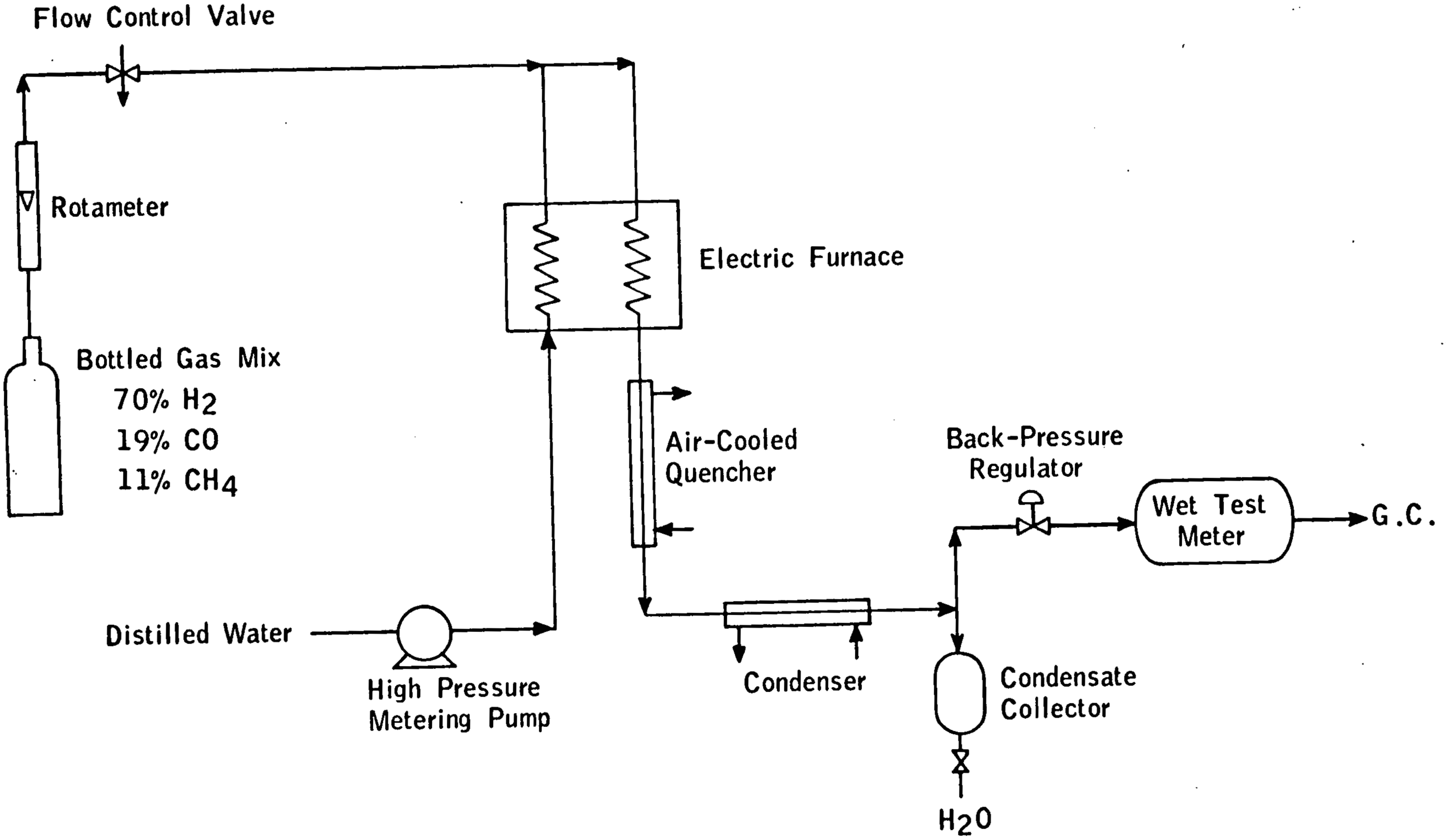
Bench scale studies were recently initiated to determine the effects of variable pH and potassium ion concentration on the amount of catalyst remaining on the char. The equilibrium concentration of potassium on digested char, undigested char, and fines will be determined as a function of potassium concentration in solutions of constant pH and as a function of the pH of solutions of constant potassium concentration. Data will be obtained at room temperature and at the solution boiling temperature. This fundamental information will be used in both the PDU and the commercial CCG catalyst recovery system process definition.

1.3 Gas Phase Reactions Study

Construction of the Gas Phase Reactions Unit (Figure 1.3-1) was completed during January. The apparatus was modified to include a rapid quench of the

FIGURE 1.3-1

REVISED GAS PHASE REACTIONS UNIT



reactor product gas to prevent possible equilibration of the gas at temperatures lower than that of the furnace reaction zone. A feed gas rotameter was also added to doublecheck for a change in the number of moles of gas due to reaction in the furnace. The computer program to analyze the data from the experimental apparatus was also written and debugged. This program calculates the equilibrium composition of the furnace feed gas if shift alone or both shift and methanation reactions were to occur, and expresses the actual gas composition as a percentage approach to these limits.

The pipe for the reactor was received and the reactor fabricated during January. The reactor and its internals are shown in Figure 1.3-2. The pipe is 36" long, with the middle 18" encased in the furnace. The remainder is exposed to allow for radiative cooling of the flanges, which are limited to 1175°F at 525 psig. A mullite (ceramic) liner prevents contact of the gas with the reactor walls.

Water for process steam is metered through a 1/8" tube which rises inside the reactor to within the heated section, then doubles back to the bottom of the reactor. A sintered metal filter disperses the gas at the tube end. This configuration should allow total boiloff, while the 1/16" mullite beads packed in the bottom half of the reactor should distribute the steam across the reactor cross-section and provide a large heat transfer area to bring the steam to reactor temperature.

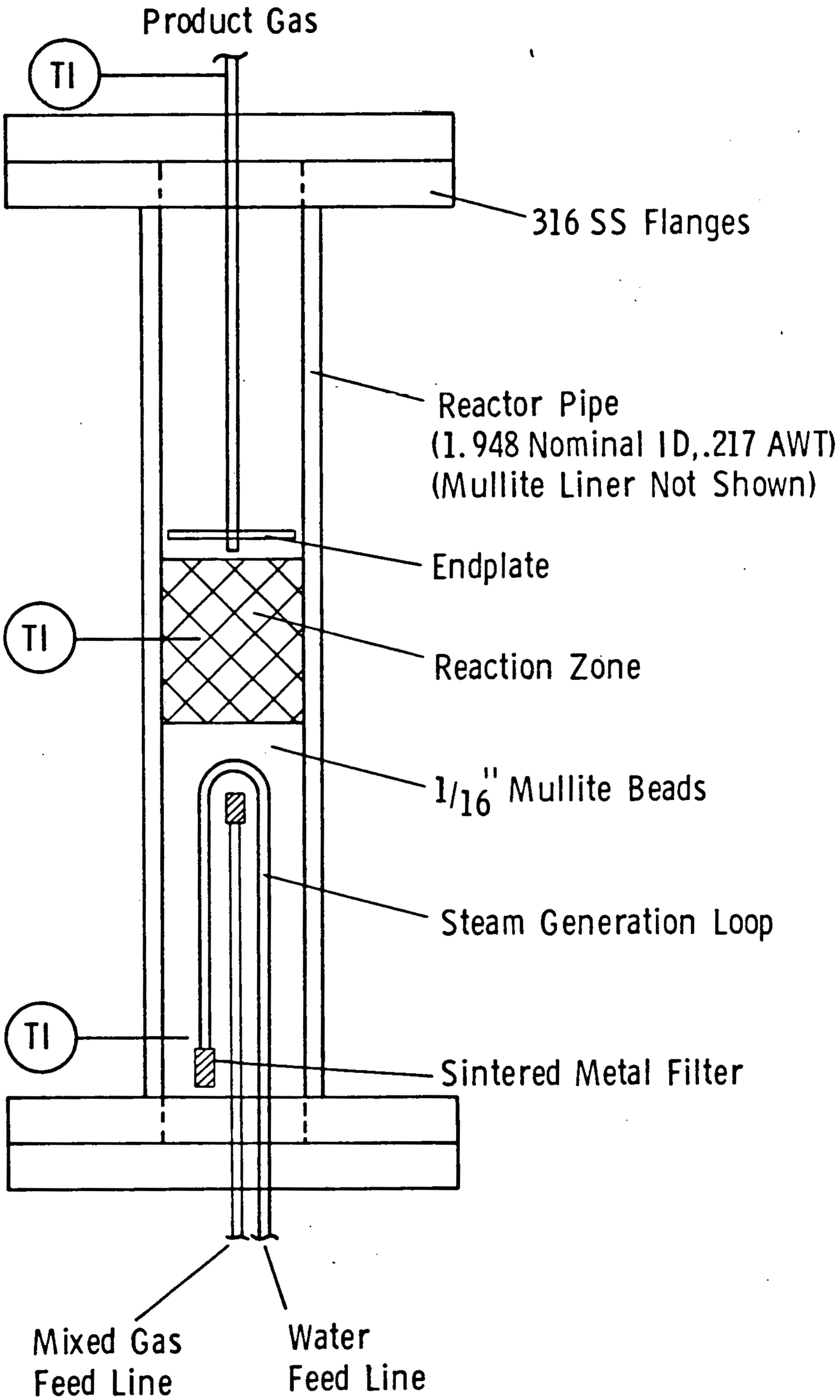
The bottled $H_2/CO/CH_4$ gas mixture enters the reactor through a 1/4" tube ending in a sintered metal filter about four inches below the reaction zone. This allows space for the gas and steam to mix while minimizing exposure to the potentially catalytic steam coil. The reaction zone is a four inch section in the center of the furnace where temperature control is most accurate. This zone will be filled with mullite beads or with alloy wire mesh to test the catalytic effects of different alloys. Immediately above the reaction zone is an endplate to minimize mixing in the dead space above the reaction zone and to position the 1/8" product gas line which leads to the quencher.

Initial attempts to heat the reactor revealed that a maximum temperature of 1400°F (instead of the 1600°F required for the study) could be attained in the reaction zone. This was attributed to excessive heat loss from the reactor flanges which had been left uninsulated. The reactor installation was modified by adding more insulation and by installing a small heater on the bottom extension of the reactor, but there was no significant increase in reaction zone temperature. A different electric furnace was then installed and resulted in dependable rapid heatup of the reaction zone to the required temperature.

With the equipment now operational, experimental runs will begin. The shift reaction will be studied as a function of temperature and amount of metal in contact with the gas stream.

FIGURE 1.3-2

RECYCLE FURNACE EXPERIMENTAL REACTOR



LABORATORY PROCESS RESEARCH AND DEVELOPMENT

2. Process Development Unit Operations
(Reporting Category C02)

2.1 PDU Startup and Initial Operations

Startup and Initial Operation Schedule

Figure 2.1-1 presents a time line for construction, startup, and initial operations during 1979.

The gasification section will be completed and started up in a once-through operation before construction of the gas separation section is completed. This will allow smooth operation of the gasifier to be achieved before the acid gas removal, molecular sieve cleanup, and cryogenic distillation systems are started up. Recycle gas to the gasifier will be simulated by blending hydrogen and carbon monoxide from trailers during this period.

The catalyst recovery system will be constructed while the gas separation system is coming on line.

By use of this stepwise startup procedure of the entire PDU system, the most efficient use may be made of the technical, operating, and construction personnel. It has the added benefit of debugging and establishing operation of one system at a time so that reliable operations may be built up sequentially during the startup period.

Checkout Test Plan

Unit checkout of the PDU has begun. The Checkout Test Plan is presented in Table 2.1-1. Step 1, hydrostatic testing, has been completed for all vessels, including the gasifier.

Initial Startup Plan

Initial startup operations began with full shift coverage after a three day Operator Training School. Mechanical construction is being concentrated on the gasification section and operators are checking equipment as it becomes available.

The steps and procedures for the PDU Initial Startup are outlined in Table 2.1-2. These steps are being integrated with the Checkout Test Plan wherever possible in order to complete the startup of the gasification section as soon as possible. Item 1, gas flow testing, has been started concurrently with gas pressure testing (Item 2 of the Checkout Test Plan) and is essentially complete.

Item 2, cold solids circulation, has begun. Coal has been transported by automatic control from the Coal Preparation Unit (CPU) approximately 600

FIGURE 2.1-1

STARTUP AND INITIAL OPERATION SCHEDULE FOR PDU

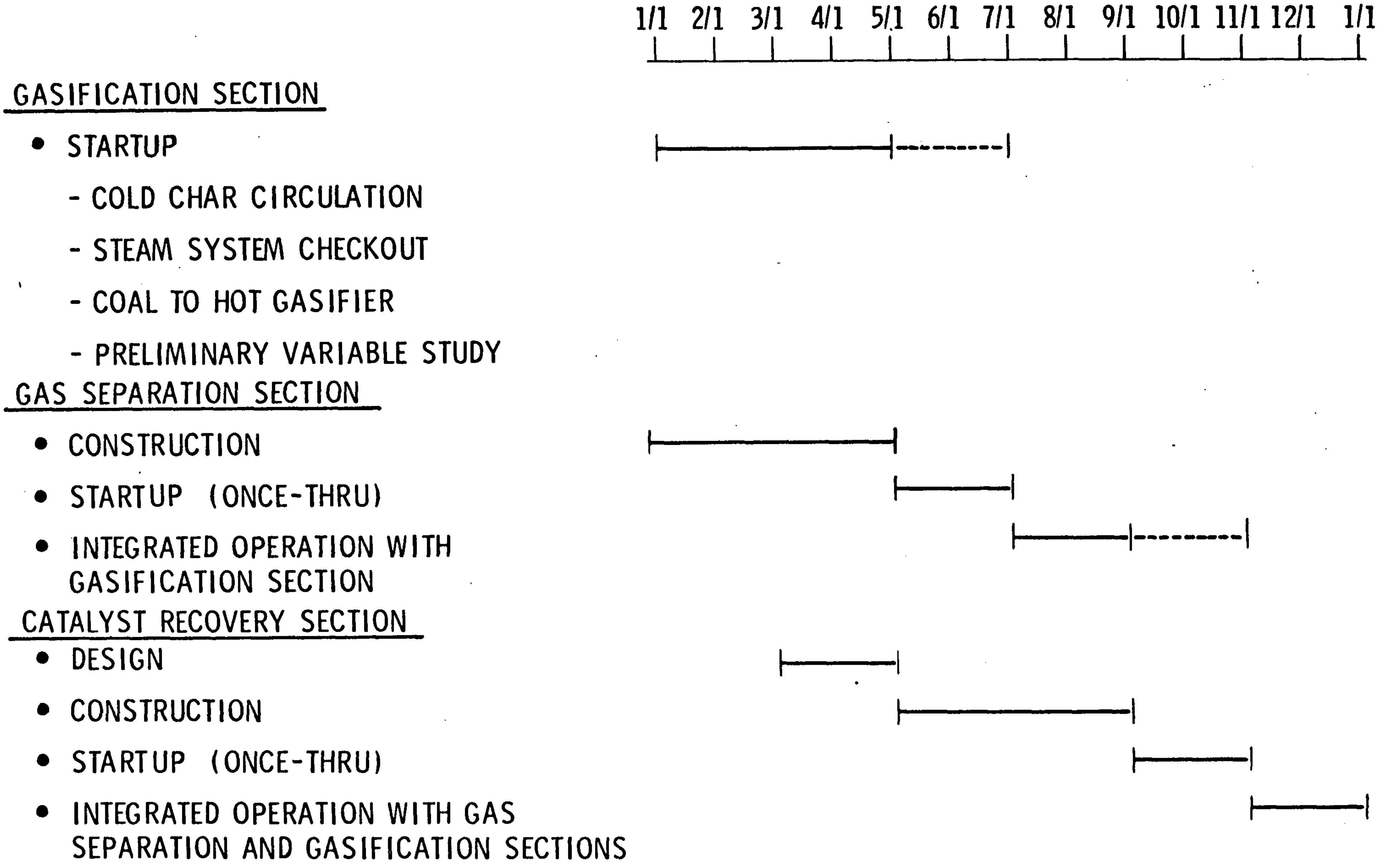


Table 2.1-1

Checkout Test Plan for the PDU Gasification Section

Summary of Steps	Criteria for Satisfactory Performance
1. Hydrostatic Testing - All systems liquid filled with water. Hydrostatic Test Pressure = $(1\text{-}1/2) \times (\text{Design Pressure at Design Temperature}) \times \dfrac{(\text{Allowable Stress for Material at Ambient Temperature})}{(\text{Allowable Stress for Material at Design Temperature})}$	No ruptures, cracks, or leaks.
2. Gas Pressure Testing - Similar to above. All pipe and tubing joints to be tested with liquid leak detector. Flanges to be wrapped with tape and a pinhole tested with leak detector.	No leaks.
3. Commissioning and Checkout of Equipment a. Programmable Controller "Burn-in" of electronics to eliminate initial failures. Check each circuit internally on CRT for proper programming action by forcing inputs to simulate signals from the field, and observe action of output on CRT. Check each system for proper integrated function resulting from individual circuits.	Inspection by qualified technical person to determine proper functioning of all systems.
b. Checkout of Block Valves All block valves must be checked visually in the field for proper open/close operation, forced from the programmable controller. In sequenced double block valves, verify tandem operation of both valves to be sure of proper wiring of actuation signal through limit switches of the other valve.	Verification of operation in field as forced from programmable controller.
c. Checkout of Alarm Systems Verify from alarm sensors in the field or other source, through all associated circuits, to the alarm in the control room. Verify analog alarms from analog controllers, through current to contact closure devices to alarm. Check alarms in both the alarm annunciators and graphic display, including redundancy in graphic display lights when specified.	Verification of complete circuit function for each individual alarm.
d. Graphic Display Verify approximately 100 status lights (in addition to the alarm lights) indicating open block valves, motors and pumps, pressure switches and other miscellaneous status lights.	Verification from source to each light.
e. Analog Controllers Check all wiring on loop diagrams against installed wiring to be sure the sensor, current to contact closure device, and 3-pen recorder are properly looped on the input signal, and the output signal operates the final control device. Set initial tuning constants as specified by instrument engineer.	Visual wiring check. Observe operation of final control device by manually adjusting the output at the controller.
f. Temperature Controllers Check for proper range and type. Verify output wiring of temperature controllers (TIC) and temperature limit switches (TL) through power relays, heater breakers, power controllers and field wiring to heaters by individual voltage checks at heaters. Check analog action of TIC's and on/off action of TL's.	Individual voltage checks in field. Later, as heaters are turned on, check controller thermocouples and other skin couples on analog multiplexer as heaters warm up.

Table 2.1-2

PDU Initial Startup

1. Pressurize the Gasification System with Nitrogen

Check all flow, pressure and ΔP controllers with flowing nitrogen. Start up and check operation of compressors. Pressure and flow control must be smooth and controllers must be tuned for stable operation at design conditions of the unit.

2. Cold Solids Circulation with Nitrogen

Demonstrate ability to handle solids through dense-phase solids transporters, lockhoppers, feed lines, fluidized bed reactor, char withdrawal systems, and fines filters. This is to be done with char solids, using nitrogen only, at pressures up to 3500 kPa at ambient temperature. Includes automatic operation of lockhopper swing and fill systems, feed lockpots, char withdrawal, and fines filters automatic swing and emptying systems.

3. Heatup of Reactor System

Initial conditioning of heaters at low temperatures (below 80°C) may proceed during Step 2 above. When completed, the reactor should be depressured for safety while the heaters are brought up to design temperatures. The TL's and TIC's should be stepped up in 50°C increments during this period to avoid sudden overheating of the radiant ceramic heaters. Final tuning of the controllers should be done at design temperatures. Each heater must respond to process upsets and changes in set point without cycling. Tuning should be optimized to provide stable, slow response, rather than borderline on cycling.

4. Solids Circulation with Nitrogen at Design Temperature

This is the final checkout of the unit prior to introducing gasification streams. All control, alarm, and sensing equipment should be functioning reliably at this point. Operators should be familiar and confident with the operation of the unit and able to handle upsets in a safe manner. All automatic sequencing equipment should be functioning with override capability easily available where specified. All problems of instrument or mechanical nature should be solved during this relatively safe period of unit operation.

5. Startup of Stream Generation System

During Steps 2, 3, and 4 above, the steam generation system may be started and debugged. This is done off-line without feeding steam to the gasifier. The vaporizer may be operated at design temperature and pressure while the steam is going to the condenser. This permits tuning the vaporizer controls off-line. Smooth, reliable operation must be achieved with no detectable cycling of liquid level or pressure.

6. Feed Coal, Steam, and Syngas to the Gasifier

This completes the initial startup of the gasification section. Syngas is used once-through which allows the gasification section to be checked out without the necessity of running the MEA absorbers, molecular sieves, or cryogenic distillation equipment. This begins the shakedown operation and variables study period with simulated syngas recycle. Preliminary material balance data should be available, and data logging by the computer should be debugged by this time.

feet to the storage hopper on the PDU, LH-3. The rotary feeder, the Catalyst Addition Unit (CAU), and the transporter that lifts the catalyzed coal from the ground level to the surge hopper on the 12th floor, LH-1, have all been operated successfully.

The automatic coal feed system is currently undergoing initial startup tests using nitrogen as the transporting fluid. Fluidization tests are also being performed on the coal that has been fed to the gasifier. These tests are necessary to help determine bed fluidization characteristics, set pressure tap flow rates, and realistically test downstream portions of the unit such as the automatic fines filter system and the gasifier char withdrawal valves. All tests have been conducted at ambient temperature using nitrogen as the transporting fluid.

Programmable Controller

The PDU uses a programmable controller to handle valve sequencing, alarming, and other miscellaneous functions in the following systems:

- Catalyst Addition Unit
- Coal Feed System
- Char Withdrawal System
- Gas and Steam Feed System
- Filter/Scrubber System

As shown in Figure 2.1-2, the programmable controller takes inputs from the field, simulates relay circuits from the programmed logic, and activates the appropriate outputs in the field. Changing the logic changes the relay action without costly hardware changes. The programmable controller uses relay language and requires very little maintenance or operator training.

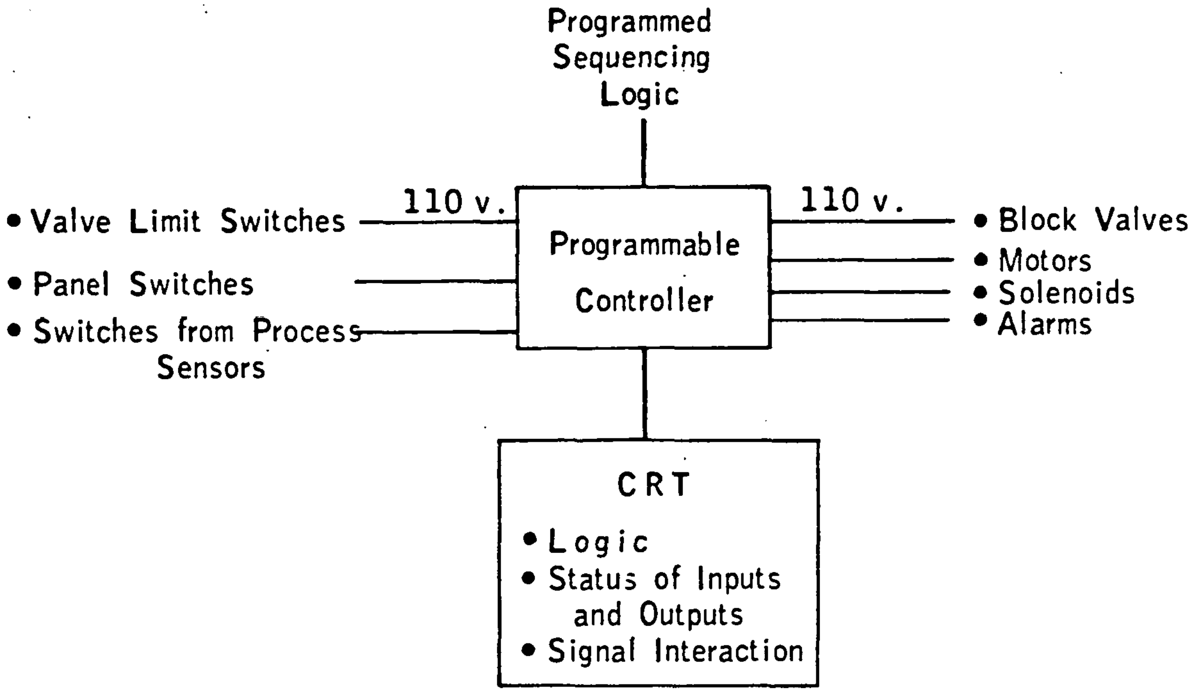
The general functions of the programmable controller on the PDU are:

- Sequencing double block valves
- Switching parallel trains on and off line; regenerating the off-line train
- Constraining override action
- Emergency shutdowns of individual systems
- Automating iterative batch processes (lockpots)

Input and output signals were assigned for existing systems. Programmed logic was checked during cold operations for the coal feed, gas feed, and filter systems.

FIGURE 2.1-2

PROGRAMMABLE CONTROLLER



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2.2 Char Production in Fluid Bed Gasifier (FBG)

Approximately 3000 lbs of char have been produced in the FBG from Illinois No. 6 coal both for PDU startup and for catalyst recovery studies. These recovery studies are being carried out in bench scale and prototype char digesters and leaching tanks to obtain design and operability data for a PDU catalyst recovery unit.

Table 2.2-1 summarizes results of the recent FBG operations. The char density initially was low compared to densities observed during predevelopment contract studies. Several differences in the present operation and that of the predevelopment contract were identified and investigated. These differences are listed below:

Table 2.2-1

Recent FBG Operations

Char Properties	Initial	Present	Predevelopment Period
Fluidized bed density, lb/ft^3	7	11	11-20
Bulk density, lb/ft^3	15	21	22-28

Differences in Operation Possibly Affecting Bed Density

- New batch of coal
- Different mesh size ranges in coal
- KOH vs K_2CO_3 catalyst
- Possible differences in air exposure

The first operations, starting in October, 1978, used coal feed which was ground to -8 mesh and contained approximately 35 wt.% of particles finer than 100 mesh. This was a departure from prior operations in 1977 using -8 to +100 mesh size coal. It was determined that inclusion of the fines in the feed coal caused the bed to contain a large portion of fines which could lead to a low fluidized bed density.

A new grind of -8 to +100 mesh was made using a fresh batch of coal. When this coal was fed to the unit, severe feedline and reactor plugging occurred immediately. It was found in laboratory devolatilization tests that the larger particles, approximately 8 to 16 mesh, were swelling and becoming plastic. Particles less than 20 mesh did not swell detectably on devolatilization. It was determined that the final screen on the Catalyst Addition Unit (CAU) had been changed to 6 mesh rather than the 8 mesh used in the predevelopment operations. The 8 mesh screen had rejected a significant portion of the 8 to 16 mesh particles during the predevelopment runs so that the severe plugging did not occur at that time. A screen was then installed on the CAU to reject all particles larger than 16 mesh. The operation of the FBG on the new coal was immediately very good with no plugging or bridging in the gasifier. However, the bed density was still low. This showed that neither fines in the feed nor the inclusion of 8 to 16 mesh particles were the primary cause of low char density.

Another difference from the predevelopment operations was the use of KOH catalyst in place of K_2CO_3. To test this as a possible cause of low density, K_2CO_3 was applied to the coal. No measurable difference in char density was found.

The catalyzed coal was then deliberately air-exposed in both the CAU at normal conditions (350°F max.) and in barrels prior to loading in the FBG. This resulted in higher bed densities and is now thought to be the principal difference between recent and prior operations.

The FBG operated during the month of February with air exposed feed in order to produce an inventory of high density, high conversion char for catalyst recovery studies. FBG operations have now been concluded.

In summary, several questions have been raised in this work and are not yet completely answered. The coal-catalyst interactions of soak time, catalyst concentration in the solution, and particle size as well as the effect of oxidation need further investigation. A bench scale program is underway to help define these interactions.

2.3 Pilot Plant Catalyst Recovery Studies

Prototype Catalyst Recovery Unit Operations

The Prototype Catalyst Recovery Unit, shown in Figure 2.3-1, consists of a digester, leaching tank, and circulating pump. Char and lime feed are added to KOH solution in the digester and the solids are kept suspended by an agitator and a circulating pump. After digestion, the slurry is pumped by the circulating pump into the leaching tank. The solids are then leached with either a hot KOH solution or water.

As discussed in the October-December, 1978 Quarterly Report, runs with the prototype unit have shown a large solids carryover in the solution leaving the leaching tank. In addition, the particle size distribution of the solids in the overflow from the leaching tank (Figure 2.3-2) shows that approximately 80% (wt) of the solids are less than 10 microns in diameter. Figure 2.3-2 also shows that fine particles are separated from the solids in the leaching tank and are carried out of the system since the digested solids charged to the leaching tank have a particle size distribution between the overflow solids and those remaining in the tank. These observations led to a program to identify the source of the large number of fines which comprised the bulk of the overflow from the leaching tank.

Figure 2.3-3 is a plot of a typical particle size distribution for the char feed mixed with the lime (all -325 mesh) fed to the digester and the solids after digestion with both the agitator and the pump operating. The figure shows that, although 26% of the solids fed to the digester are less than 10 microns, after digestion 61% of them are. From this result, it is clear that many small particles are being made in the digestion step. These particles may come from two sources; either they are made by the digestion process itself, or they are a result of particle breakdown from agitation of the digestion mixture. To reduce particle breakdown, a new centrifugal pump designed to minimize particle breakdown was installed. This pump moves the fluid and particles by creating a vortex with a recessed impeller. Particles in the fluid should not contact the impeller and therefore avoid the resulting high stresses that would break them down. Figure 2.3-4 shows a plot of the char/lime particle size distribution after digestion with the agitator and the new circulating pump in constant operation. In addition, the lime feed is larger, -20, +50 mesh cut, so all particles are initially greater than 10 microns. It can be seen that the particles are broken down until about 70%

FIGURE 2.3-1
SINGLE STAGE CATALYST RECOVERY PILOT PLANT

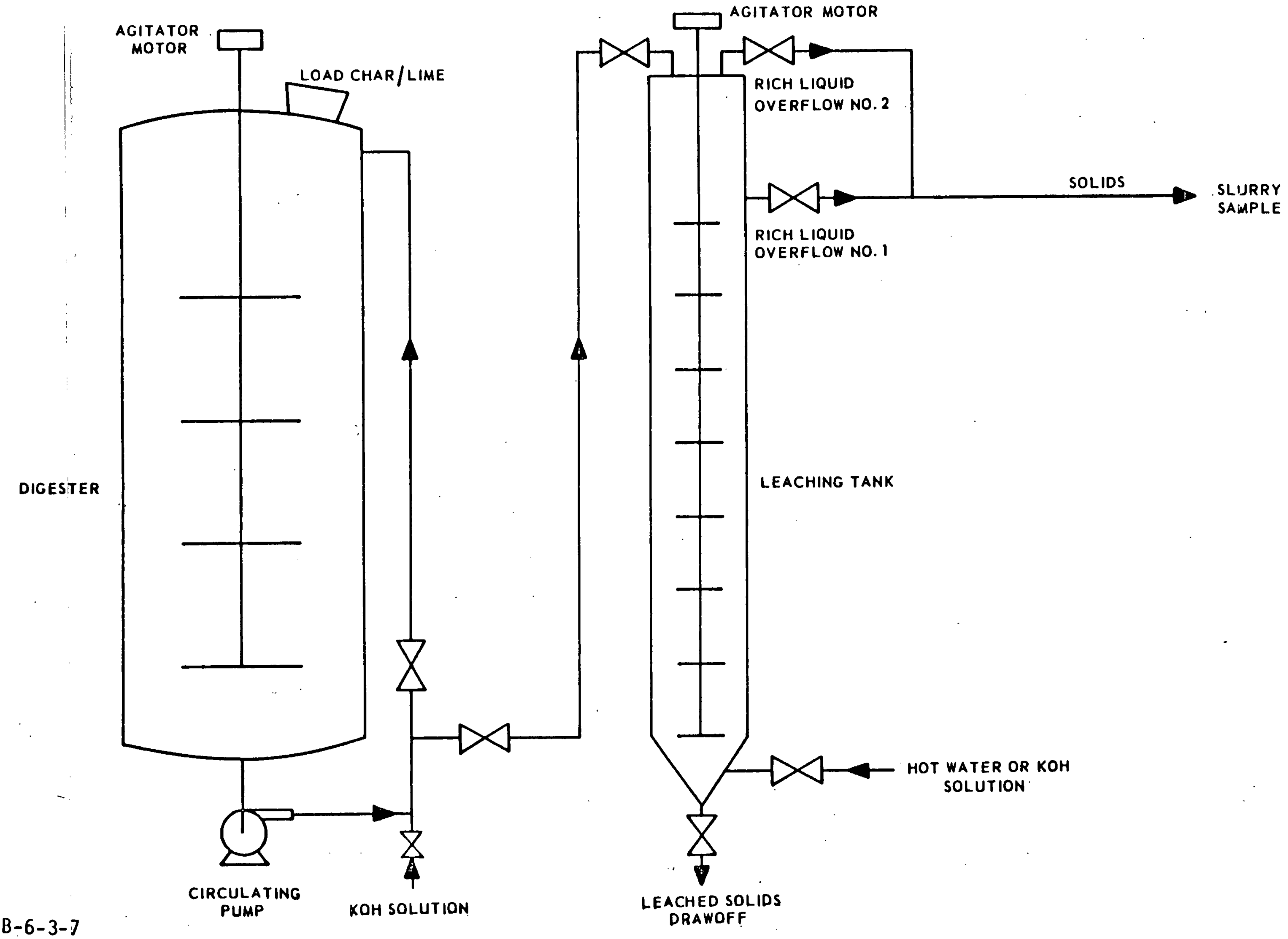

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FIGURE 2.3-2

LEACHING TANK SOLIDS PARTICLE SIZE DISTRIBUTION

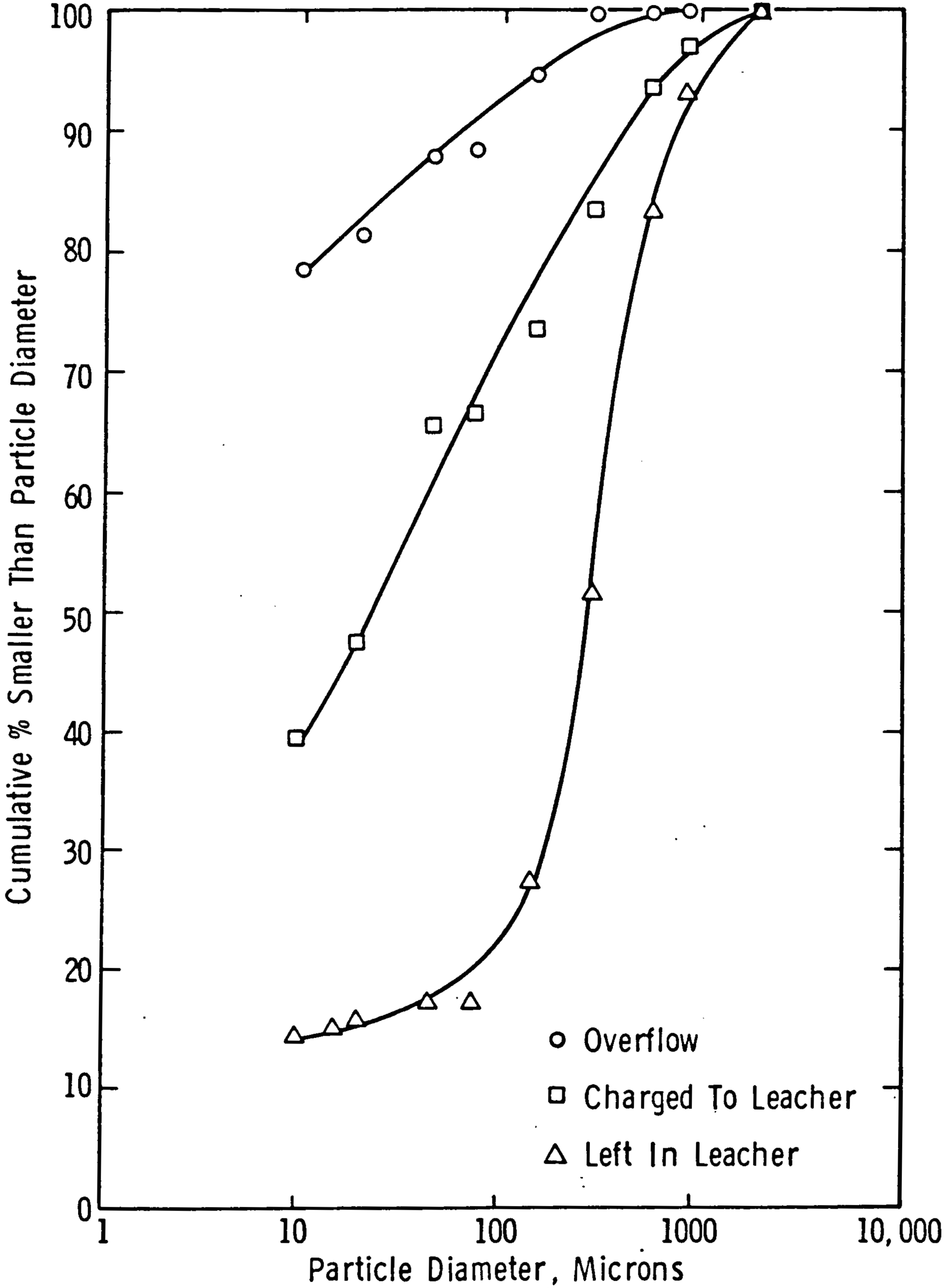

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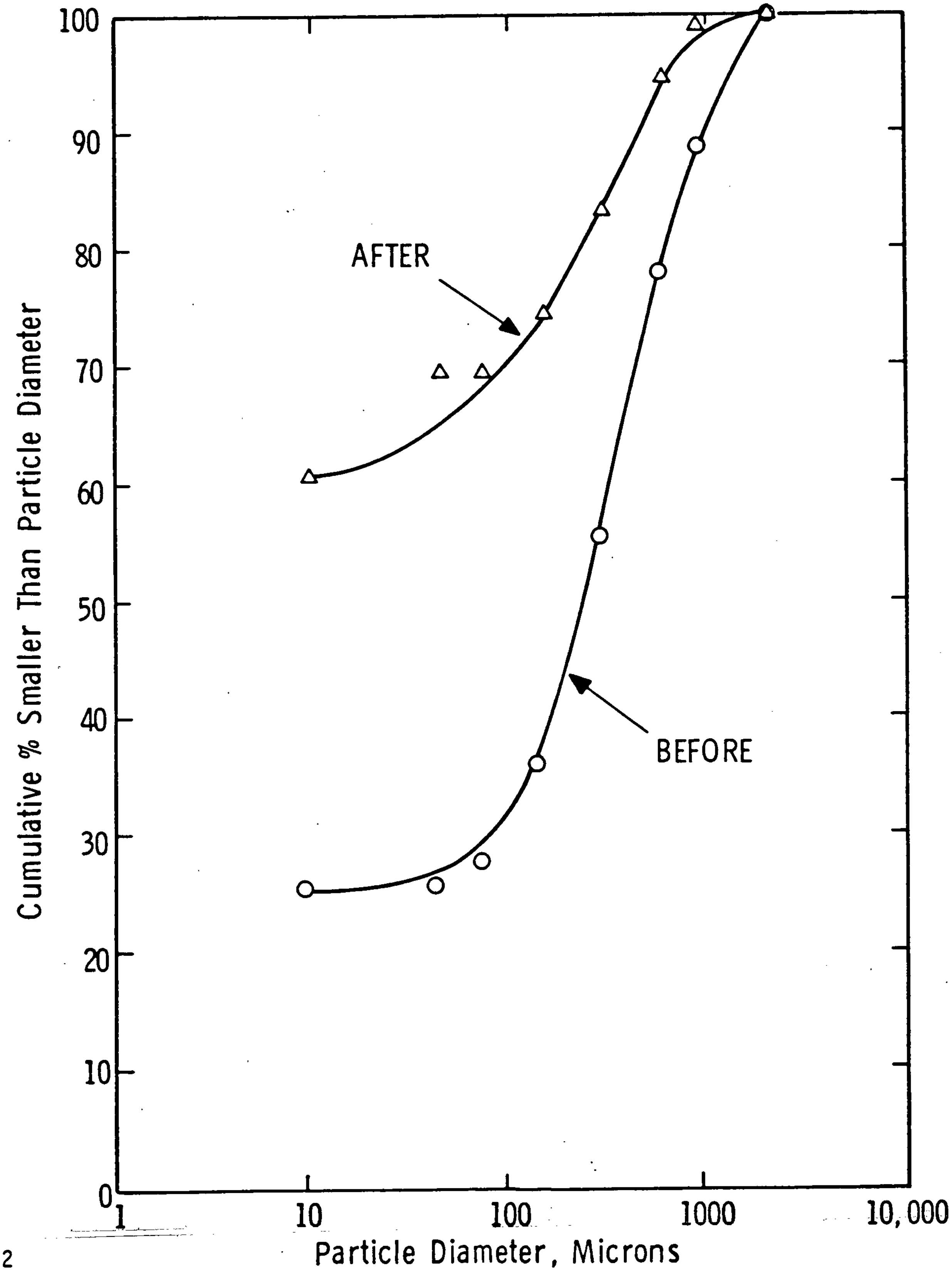
FIGURE 2.3-3

PARTICLE SIZE DISTRIBUTION OF SOLIDS BEFORE AND AFTER DIGESTION USING ORIGINAL PUMP



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are less than 10 microns. A comparison of the particle size distributions after digestion of Figures 2.3-3 and 2.3-4 shows that the new pump results in more breakdown of particles than the original pump. Figure 2.3-4 also shows the particle size distribution after digestion with the new pump running only when necessary to keep the solids suspended (minimum pumping). This shows that less than 25% of the solids is less than 10 microns with the minimum amount of pumping, while approximately 70% is less than 10 microns while using a pump continuously. Figure 2.3-4 also shows the particle size distribution of the solids before and after digestion but without pumping. Only the agitator, turning at 140 RPM, is providing mixing. This case represents a practical minimum of particle breakdown with this equipment. This shows that only 15% of the particles is less than 10 microns compared with 60 to 70% using a circulating pump operating continuously and about 25% with the minimum amount of pumping. Thus, it appears that the circulating pump is causing extensive particle breakdown.

Some form of energy input to the digester is necessary, however, to prevent the solids from settling. The other readily available mean for suspending the solids is using an agitator. However, the agitator presently in the digester does not provide enough mixing to suspend the solids. The minimum pumping runs in Figure 2.3-4 cannot be duplicated because the lime and char settle and plug the discharge line. This indicates that better mixing is needed than can be supplied by the existing agitator.

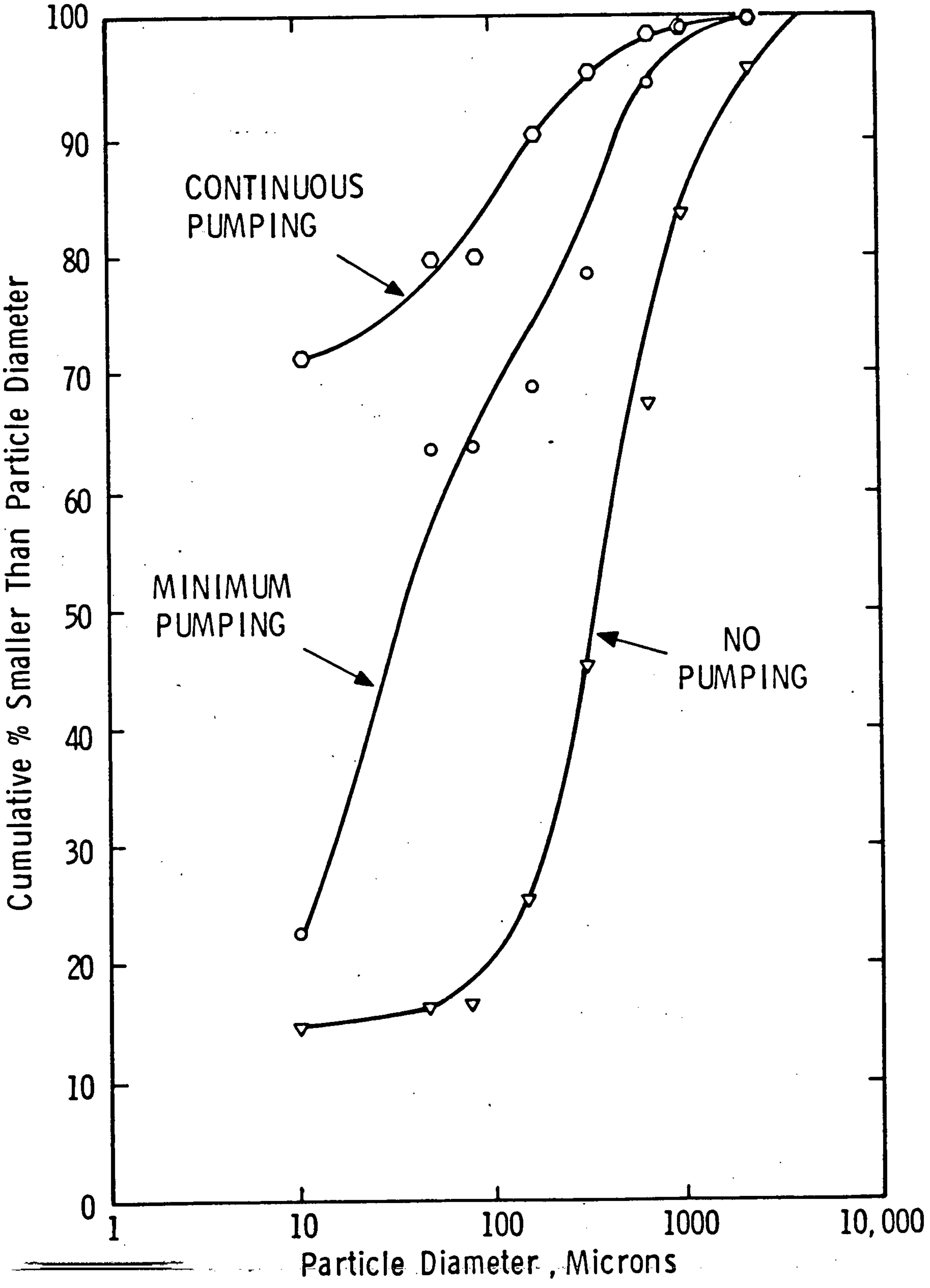
To prevent the solids from settling while minimizing particle breakdown due to agitation, the digester design has been improved to provide better agitation by impeller alone. As stated above, the prototype digester had an impeller turning at 140 RPM and a circulating pump to keep the solids in suspension. Although four baffles are located in the vessel to improve mixing, an internal coil which is used for heating and cooling minimizes their effect by creating dead zones and allows solids to settle to the bottom of the vessel, which results in plugging the discharge line. Increased agitation will keep the solids suspended, but will break down the particles.

The first step in the modification of the digester was to replace the old baffles with new baffles which have a gap between the vessel wall and the baffle in order to eliminate dead zones. The next step was to replace the existing impeller with an impeller better designed to suspend the solids. A variable speed drive was also installed. The final step in the digester modification consisted of removing the heating/cooling coil. Quick heatup, as described in the July-September, 1978 Quarterly Report, is actually less necessary than a well mixed reactor from an operability point of view. Electrical heaters on the outside of the digester will provide the necessary heat input. Cooling will be provided by a small coil located entirely in the vapor space above the liquid so that the coil will not disturb the fluid circulation.

Several bench scale experiments were performed to further define why a large quantity of small particles is being generated in digestion. As mentioned previously, fine lime particles contribute to the fines problem. An experiment was run to investigate the breakdown of the lime particles. Lime with a size distribution of -20 + 100 mesh was placed in a beaker with water

FIGURE 2.3-4

PARTICLE SIZE DISTRIBUTION OF SOLIDS AFTER DIGESTION USING NEW PUMP



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and stirred at approximately 120 RPM for two hours. The mixing done in this experiment is quite mild, as indicated by the fact that the stirrer tip velocity was about 0.4 ft/sec while the digester agitator tip velocity is 9.9 ft/sec. Figure 2.3-5 shows the results of this experiment. Although 20% of the lime was initially less than 10 microns, approximately 70% was less than 10 microns after such relatively mild agitation.

A bench scale experiment was performed to determine particle breakdown of the char due to the removal of potassium. Char was placed in water, left overnight, and gently filtered with a large amount of water. Potassium recovery in this experiment was comparable to that obtained in previous water wash experiments using large amounts of water. A particle size distribution of the char before and after washing/filtering is shown in Figure 2.3-6. The distribution shows a very small increase in the amounts of fine particles. This means that removal of the potassium from the char does not itself cause a significant particle breakdown.

The lime breakdown experiment was repeated using the washed/filtered char to determine char particle breakdown. Figure 2.3-7 shows the results of this experiment. Initially, nearly all of the char is greater than 10 microns. After the experiment, about 50% is less than 10 microns. Comparing the after mixing curves of both Figure 2.3-5 and Figure 2.3-7 shows that while lime is apparently more fragile than char, char particles are fragile enough so that great care is needed to avoid excessive agitation whenever possible.

In summary, experiments in this quarter show excessive particle breakdown does occur in the digester when using a pump. In addition, both the char and lime are fragile and break down relatively easily. Finally, removal of the potassium from the char by water washing does not cause the char particles to break down.

Future work will focus on the modified digester to see if the changes mentioned earlier have reduced particle breakdown.

2.4 PDU Catalyst Recovery Design

Work has begun on the design of the catalyst recovery system for the PDU. This system must be able to meet several criteria. First it must demonstrate recycle of 90% of the total potassium fed to the gasifiers. This large recycle rate should allow any potential buildup problems to be identified at an early date. Secondly, the system must be flexible enough to allow operation in both a water wash only and a digestion followed by water wash mode and also be able to handle fine char particles. Finally, it is desirable to have a system with an uncomplicated flow plan to ensure ease of operation.

Figure 2.4-1 shows the processing scheme that was identified as the preferred catalyst recovery system for the PDU at the beginning of the current contract. It consists of a lime digestion step followed by a counter current water wash using leaching tanks. These tanks act as an upflow fixed bed leacher to wash the digested char with progressively weaker solutions of KOH. The leaching solution is pumped into the bottom of the tank and flows

FIGURE 2.3-5

BREAKDOWN OF COARSE LIME PARTICLES

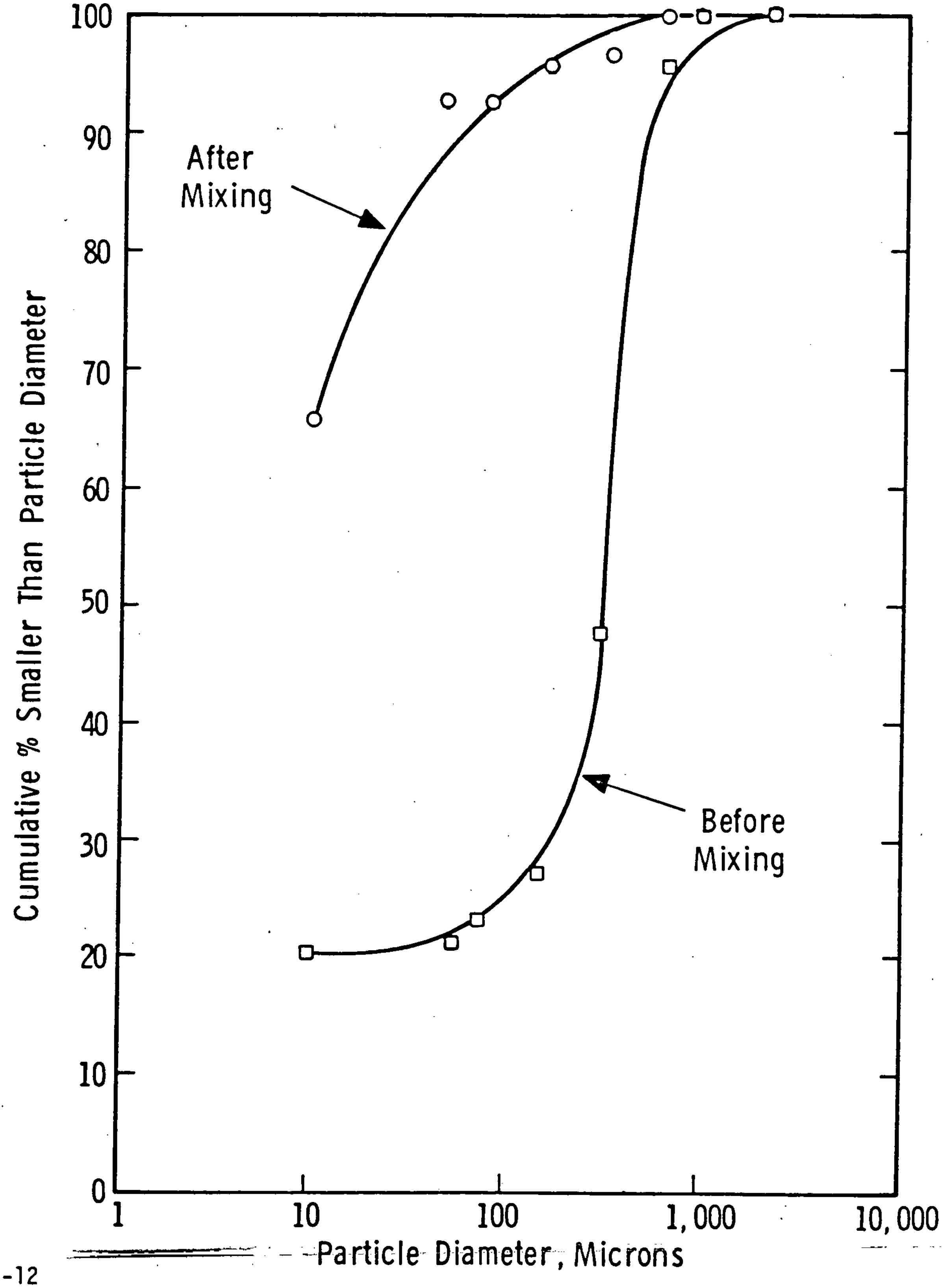


FIGURE 2.3-6

PARTICLE SIZE DISTRIBUTION OF CHAR BEFORE AND AFTER WASHING/FILTERING

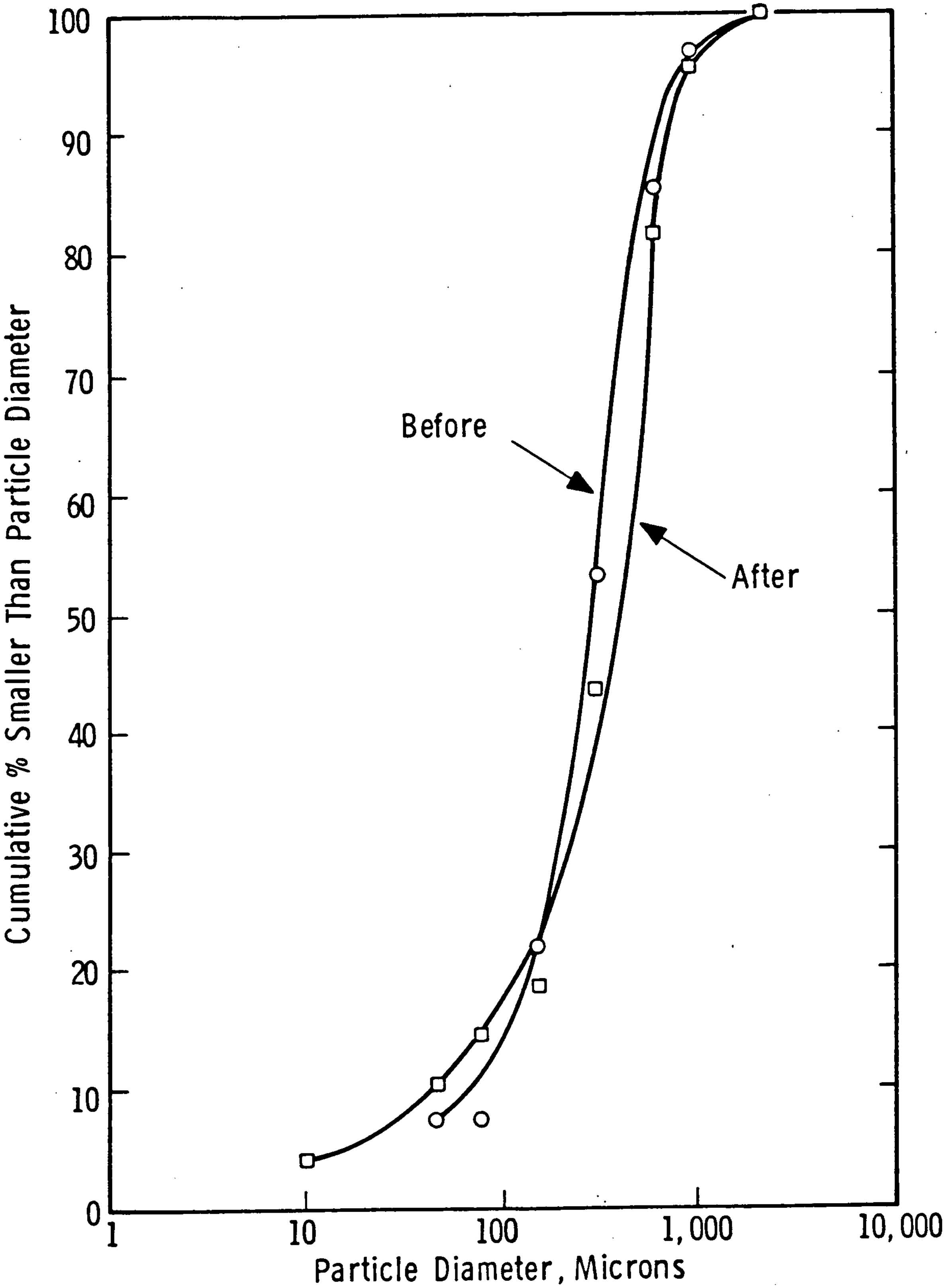


FIGURE 2.3-7

BREAKDOWN OF WASHED/FILTERED CHAR

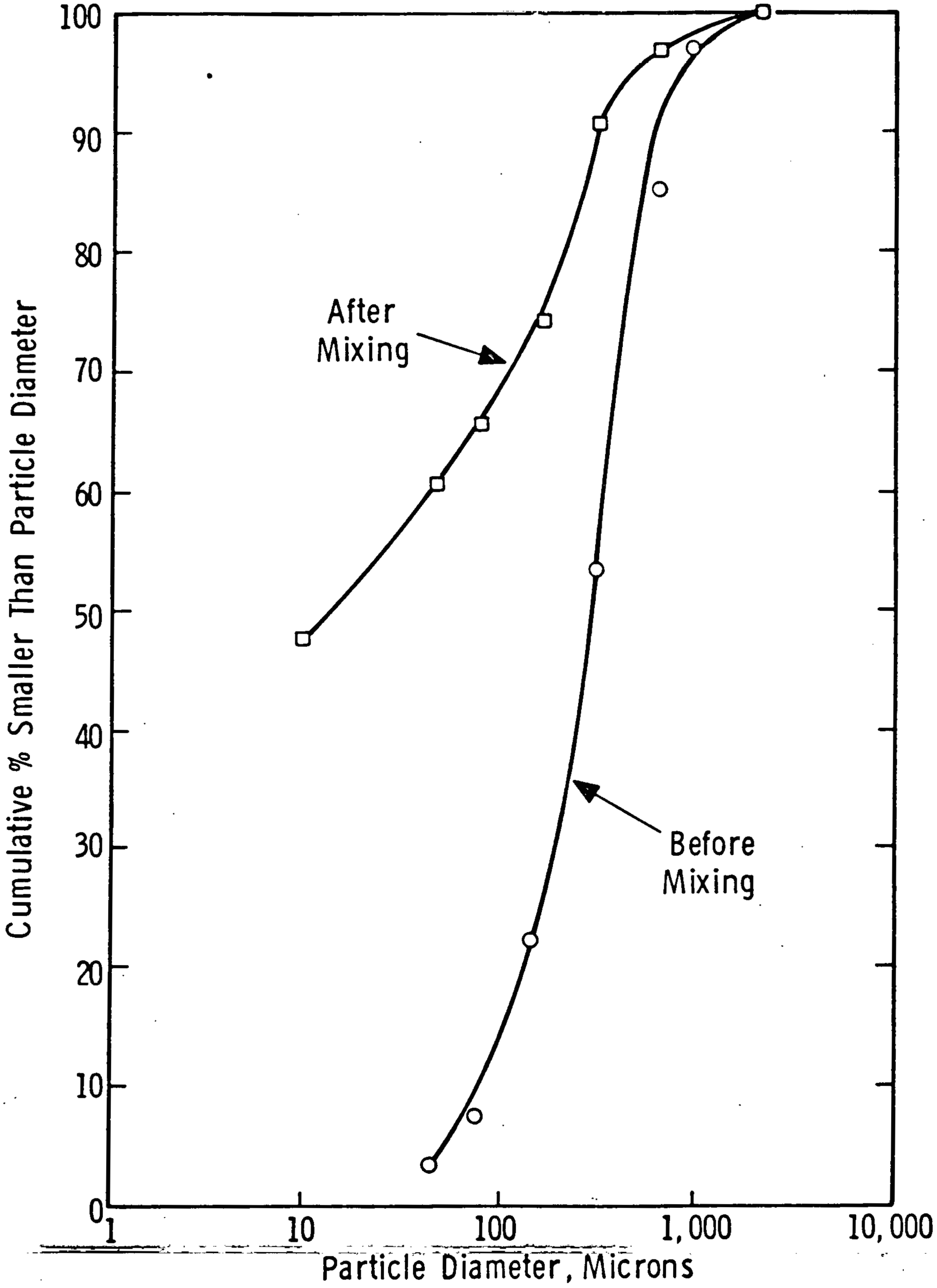
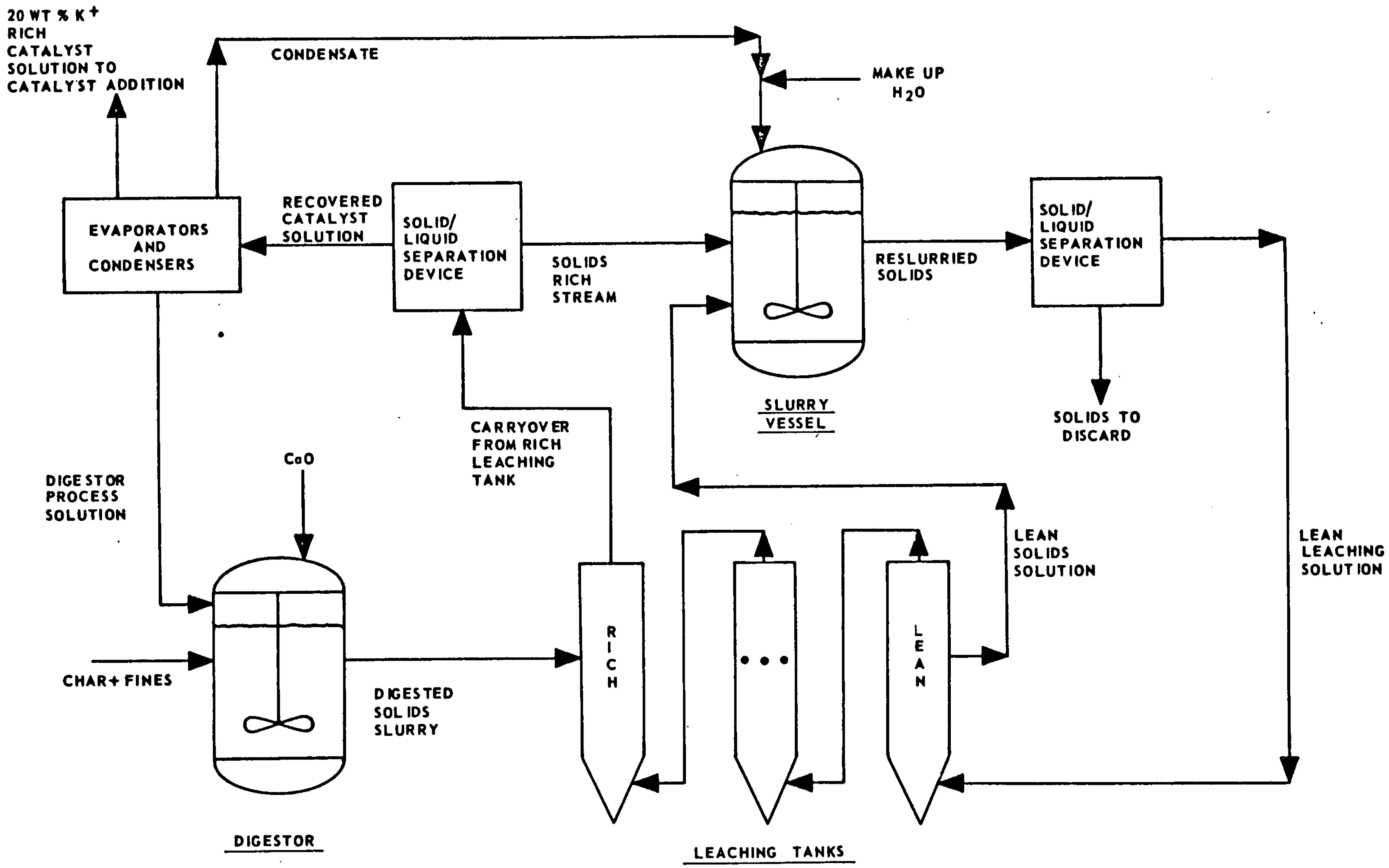


FIGURE 2.4-1

LEACHING TANKS IN SERIES



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upwards through the bed of suspended char particles. The upward velocity of the liquid keeps the solids suspended, while the settling velocity of the particles prevents them from being carried out of the tank. Any solids that do not have a sufficient settling velocity to remain in the leaching system will be carried out in the overflow from the rich leaching tank. These solids are captured by a positive solid/liquid separation device such as a filter or centrifuge, then reslurried with water in a final washing step. The rich liquor from the separation device is the recovered catalyst solution which will be concentrated to 20% (wt) K^+ in an evaporator before being sent to catalyst addition. Spent solids leave the system as a dry cake.

As mentioned earlier, this was the preferred PDU catalyst recovery system at the beginning of the current contract. To examine the operability of this concept, a full PDU scale digester and leaching tank were constructed and operated during 1977 and 1978. This prototype equipment yielded important material balance and particle size distribution data as well as providing information on leaching tank performance.

The first step in the design of the PDU catalyst recovery system is to perform a material balance around the proposed flow plan. The material balance determines whether a given configuration will meet the goal of 90% catalyst recycle. In addition, it gives information concerning relative flow rates, equipment size, evaporation requirements, and make-up rates. This information can help determine whether the system is feasible for PDU.

Table 2.4-1 lists the important basis items used in the PDU catalyst recovery material balance. Many of the items, such as the solids loading to the digester and the expected particle size distribution, are based on the performance of the FBG during the predevelopment contract. The performance of the digester and leaching tanks are based on the data gathered in the prototype and bench scale units. The basis listed in Table 2.4-1 was used only to obtain approximate overall potassium recoveries and stream rates, not for specific equipment design. Actual design will be based on maximum or minimum basis valves to allow flexibility in unit operation.

From an operability standpoint, the most important basis item is the particle size distribution expected in the slurry feed to the water wash section. This will determine the ease with which solid-liquid separations can be accomplished. For these material balances it was assumed that no particle degradation occurred anywhere outside of the gasifier; that is, no degradation of char and overhead fines was assumed to occur in the char slurry pots, the slurry let down valves, or in the digester itself. Any lime added to the digester in excess of that needed to free insoluble potassium in the hydrothermal reactions is assumed to go to fine precipitates of $CaCO_3$ or $Ca(OH)_2$. These assumptions on particle degradation are deliberately optimistic. Any configuration which is unsuccessful in recovering the desired amount of potassium from this relatively coarse particle size distribution will not be acceptable as the actual PDU system.

Unfortunately, as Table 2.4-1 shows, even this optimistic particle size distribution is too fine to be treated successfully in leaching tanks. In the prototype leaching tank it was not possible to retain particles smaller

Table 2.4-1

Basis Item		Technical Backup/Comments
● Solids loading to digester		Based on performance of FBG solids removal system.
- 25 lbs/hr bottoms char		
- 10 lbs/hr overhead fines		
- 12 lbs/hr CaO		In digestion case only. Results in a Ca/K ratio of 1.0.
● Catalyst forms in feed to digester		
- Catalyst loading of 24% (wt) on char and fines fed to digester		Based on analysis of FBG char and fines
- Other soluble forms	% Total K^+	
K_2CO_3	59.55	
KOH	6.77	
Sulfur forms	3.68	
Total water soluble	70.00	
- Water Insoluble forms	% Total K^+	
$KAlSiO_4$	25.50	
$KFeS_2$	4.50	
Total water insoluble	30.00	
● Digester Performance		
- 98% of total K^+ assumed soluble after digestion for one hour at 300°F		Based on bench and prototype work.
- No particle breakdown assumed in digester		This assumption is optimistic and will be modified before the final design of the catalyst recovery system is completed. Past digestion runs have shown severe particle degradation. However, attempts to modify the digester to reduce this degredation are being made.
● Leaching tank performance		
- Solids carryover		
+ Assume all particles less than 75 μ are carried out in rich leaching tank carryover		Based on performance of bench and prototype scale scale leaching tanks
+ 15% of bottoms char is less than 75 μ		Consistent with particle sizes obtained in FBG.
+ Assume all CaO not needed to react with mineral matter is carried out of rich leaching tank		Consistent with theory that unused lime forms fine $CaCO_3$ and $Ca(OH)_2$ precipitates.
+ Assume all gasifier overhead fines caught in secondary cyclone are carried out of rich leaching tank		Based on particle size distributions on overhead fines obtained in FBG
● Performance of solid/liquid separation devices		
+ Assume solids rich stream contains 30% (wt) solids		Based on optimistic guess at best performance of filters or centrifuges
+ Assume solids lean stream contains no solids		

than 75 μ in diameter. Yet 15% of the PDU bottoms char and all of the cyclone fines are expected to be smaller than 75 μ. In addition, most of the lime that does not react in the digester should be present as a fine precipitate. The result is that approximately 50% of the solids fed to the leaching tanks should be fine enough to be carried out the top of the first stage.

This means that half of the solids in the system will see only one stage of water washing. Potassium recoveries of 90% would be possible only at very dilute recovered catalyst solution concentrations. In addition, any further particle degradation in the digester or char slurry valves would render this system inoperable. Because of these unexpected problems with fines, it was decided not to use leaching tanks as the catalyst recovery system for the PDU. Instead, a search was begun for a simple system that would be capable of handling fine particles.

Two such systems were proposed:

- Digestion followed by one stage water wash.
- Digestion followed by counter current water wash using mixing tanks and separators.

Both systems rely upon an effective solid/liquid separation device to separate the washed char from the leaching solution. At this time it is not known what type of device will be used to perform this service. However, several possible choices including filters, centrifuges, and hydroclones are being investigated.

Digestion Followed by One Stage Water Wash

Figure 2.4-2 shows the flow plan for this configuration. The digested slurry is diluted in a large slurry tank to such a low catalyst concentration that the solids discard stream from the subsequent separation device has only a small fraction of the total potassium. The recovered catalyst solution is then concentrated in an evaporator and sent to catalyst addition.

This system is probably the simplest that could be devised. Only one solid-liquid separation is required. However, recovered catalyst solution concentrations are of necessity very low. To achieve 90% recovery of the total potassium the concentration would have to be approximately 0.5% (wt) K^+. This results in an excessive evaporation duty in order to achieve the 20% (wt) K^+ solution needed in catalyst addition. One possible method of decreasing this duty is to use additional stages of separators and mixing tanks.

Digestion Followed by a Counter Current Water Wash Using Mixing Tanks and Separators

Figure 2.4-3 gives the flow plan for this configuration. At this time, it is the preferred configuration for the PDU catalyst recovery system. Each stage of the counter current wash consists of a mixing tank followed by a

FIGURE 2.4-2

ONE STAGE RECOVERY

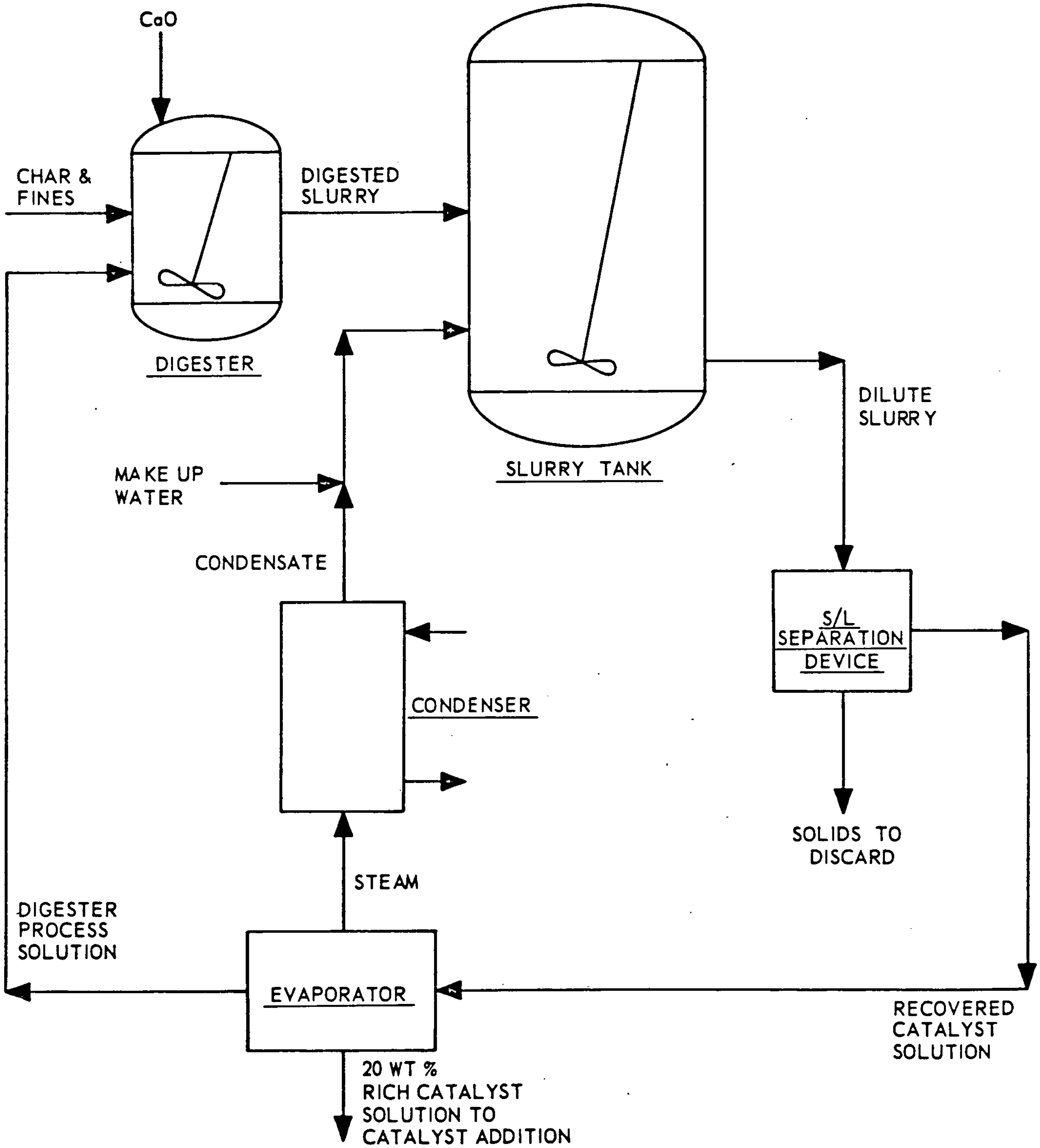

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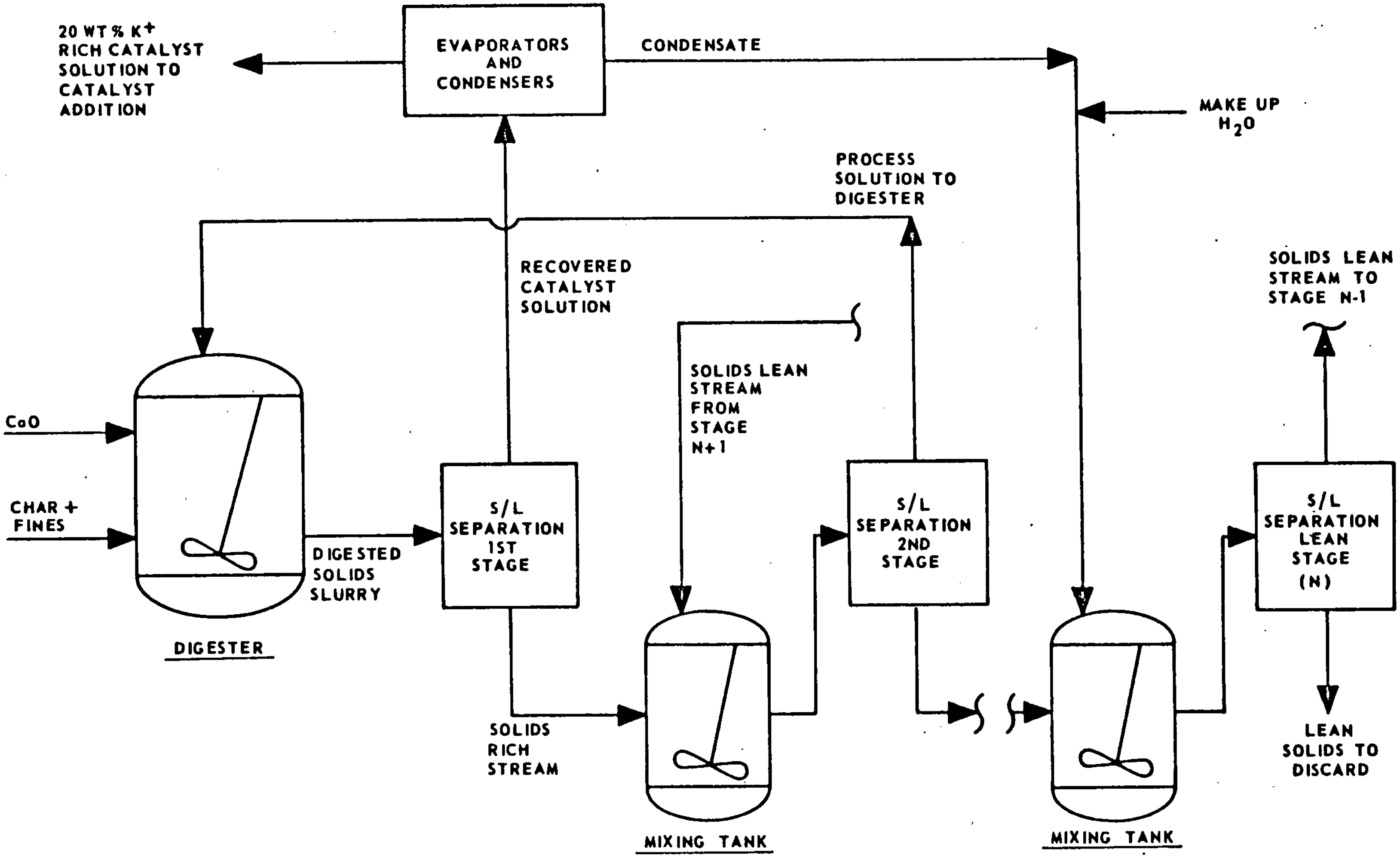
FIGURE 2.4-3

SEPARATORS AND MIXING TANKS IN SERIES



solid-liquid separation device. Thus the digester serves as the mixing tank for the first stage. The solids slurry leaving the digester is fed to a solid-liquid separation device. The solids lean stream from this device is the recovered catalyst solution which is sent to an evaporation step to be concentrated to a 20% (wt) K^+ solution. The solids rich stream from the separator is sent to the mixing tank for stage 2 where it is slurried with the solids lean stream from the separator on stage 3. This counter current processing continues until the final stage where the water condensed in the evaporator is added to the mixing tank along with any makeup water required. This configuration is identical to the one used in the 1977 CCG study design.

Table 2.4-2 presents the results of the material balances performed on this system. Recovered catalyst solution concentrations of 1, 4, and 10% (wt) K^+ were examined. For a concentration of 1% (wt) K^+, the desired recovery can be obtained in only two stages.

For a concentration of 4% (wt) K^+, five stages are required. However, the evaporation costs are greatly reduced over those for the 1% (wt) cases. To obtain a recovered catalyst solution concentration of 10% (wt) K^+, sixteen stages would be required.

While it is important to obtain high potassium concentration in the recovered catalyst solution for the commercial plant to save on evaporation costs, it is not essential to do so in the PDU. The goal for the PDU is to demonstrate catalyst chemistry at a recovery level typical of that projected for commercial operation. This goal can be met while recovering dilute solutions. Additional data on the impact of higher concentrations on performance of the solid-liquid separation device will be obtained off-line.

Future work will be directed at obtaining data necessary to design these separators and at obtaining a detailed material balance around this system.

Table 2.4-2

SUMMARY OF MATERIAL BALANCE RESULTS FOR SEPARATORS AND MIXING TANKS IN SERIES*

Case	Recovered Catalyst Solution Concentration, % (wt) K^+	Number of Stages	Recovery, % of Total K^+	Required Water Evaporation Rate to Obtain 20% (wt) K^+ Concentration, lb/hr	Makeup Water, lb/hr
1	1	2	93.41	767	122
2	1	3	95.15	781	122
3	1	4	95.40	783	123
4	1	5	95.43	783	123
5	4	5	91.18	162	122
6	10	16	90.23	41	122

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*All cases assume 30% (wt) solids in solids rich streams.

3. Data Acquisition and Correlations
(Reporting Category C03)

3.1 Cold Model Studies

The cold model of the process development unit (PDU) has been used in experiments designed to evaluate the performance of the solids feeding and fines return systems of the PDU. A diagram of the model is shown in Figure 3.1-1. The dimensions of the cold model are the same as the PDU except that the model gasifier is 14 feet in height compared to the 85 foot PDU. This height difference should not affect solids flow studies in the model.

PDU Solids Feed System

In the PDU, coal is fed to the reactor in a cyclic manner from a lockpot with a volume of 0.1 ft^3. First the lockpot is filled from above. The contents of the lockpot then flow through a vertical line into a 45° feed line and finally into the reactor. The coal feed rate is controlled by the frequency of the feed cycle. Figure 3.1-2 is a diagram of the feed system of the cold model. Dimensions of the model feed system are similar to those of the PDU except that the length of the 3/4" feed line is much longer in the PDU.

Cold Model Feed System Studies

The first experiments conducted were to determine whether solids from the reactor could be kept from backing up into the feed line. The effects of bed height above the feed point, superficial gas velocity in the reactor and gas purge rate to the feed line were examined.

The distance that the solids backed up from the reactor into the feed line was measured for reactor bed heights of 2, 3-1/2, 5, 6-1/2, and 8 feet above the feed point. The superficial gas velocity in the reactor was 0.45 ft/sec for each case. Higher bed heights forced solids farther up the feed line when there was no gas purge; however, a low flow of gas purged to the feed line from a tap located at the upper end of the 45° section of the line effectively eliminated the problem for all the bed height studies. Figure 3.1-3 shows the distance the solids backed up from the reactor as a function of bed height above the feed point and purge rate to the feed line.

A second set of experiments was carried out with a decrease in the reactor superficial velocity from 0.45 ft/sec to 0.11 ft/sec. The decreased superficial velocity reduced the solids backup in the feed line. The problem could be controlled in these cases by maintaining a low gas purge rate to the feed line as before. Figure 3.1-4 shows the results of experiments for two reactor superficial gas velocities with a bed height above the feed point of 8 feet.

The results of these experiments indicate that the problem of solids moving from the reactor into the feed line can be controlled by maintaining a

FIGURE 3.1-1

COLD MODEL OF GASIFICATION REACTOR SECTION OF PDU

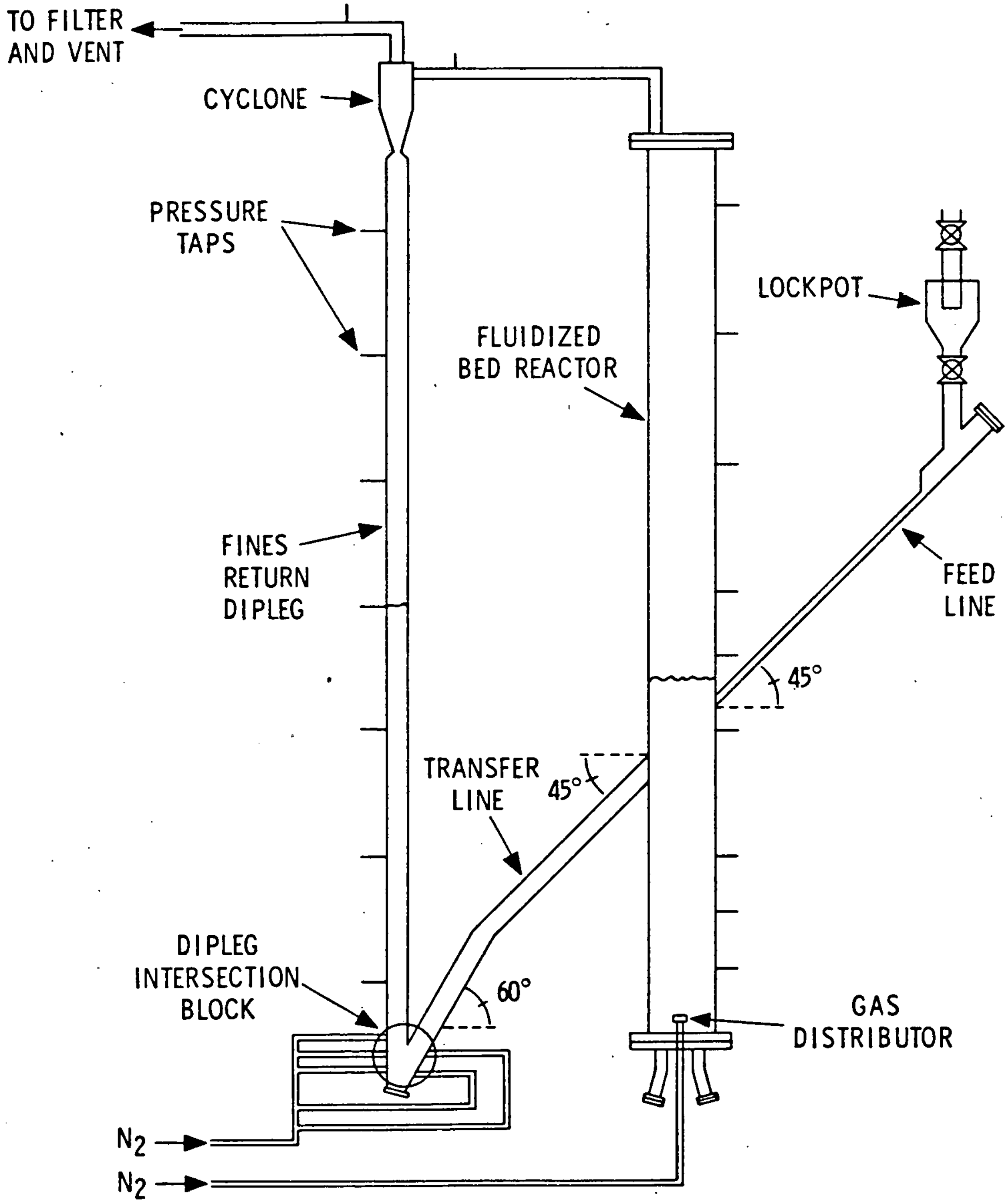

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FIGURE 3.1-2

SOLIDS FEED SYSTEM FOR COLD MODEL

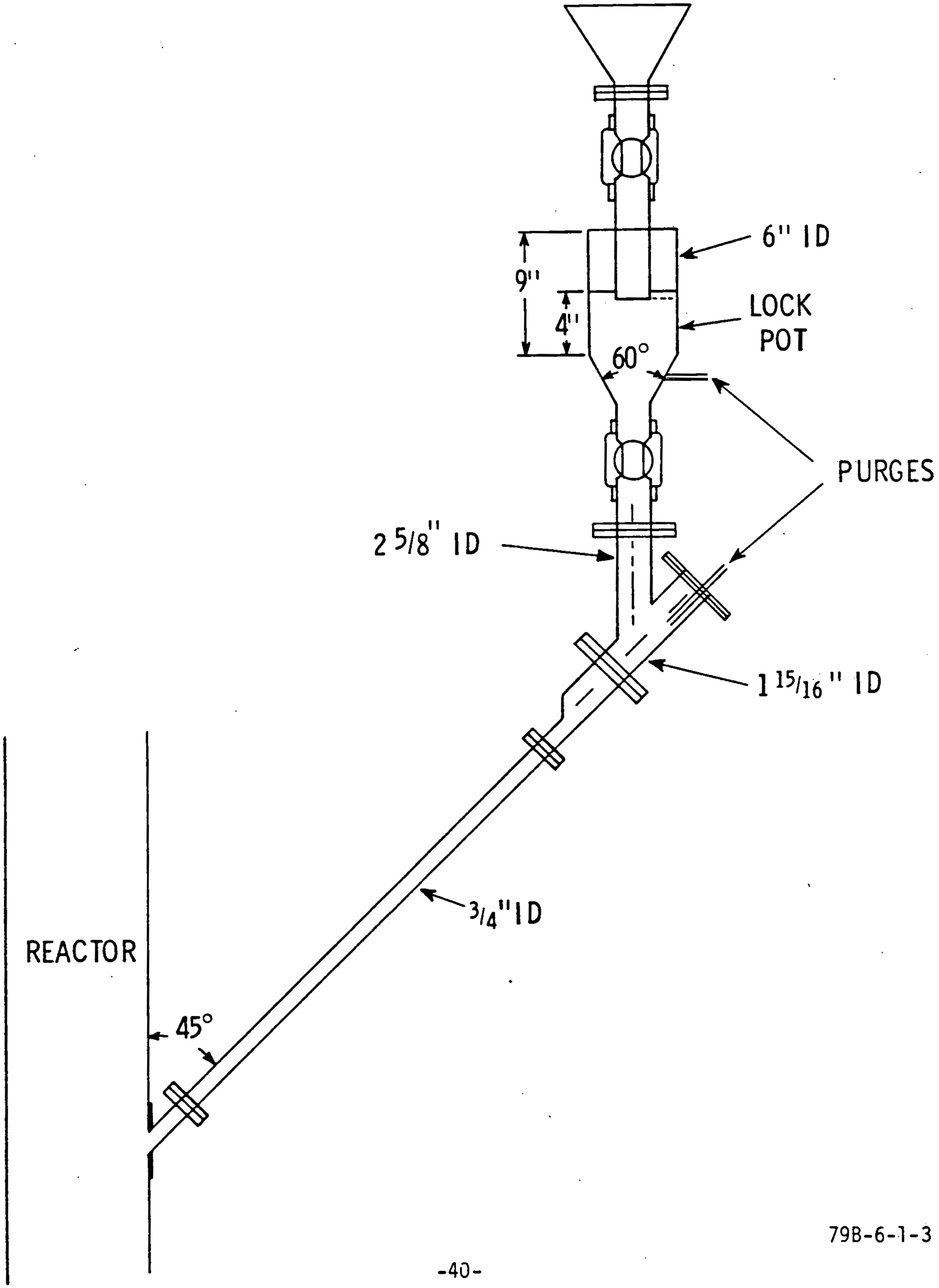

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FIGURE 3.1-3

PURGE GAS REQUIREMENTS TO PREVENT SOLIDS FROM BACKING UP INTO FEED LINE

EFFECT OF BED HEIGHT

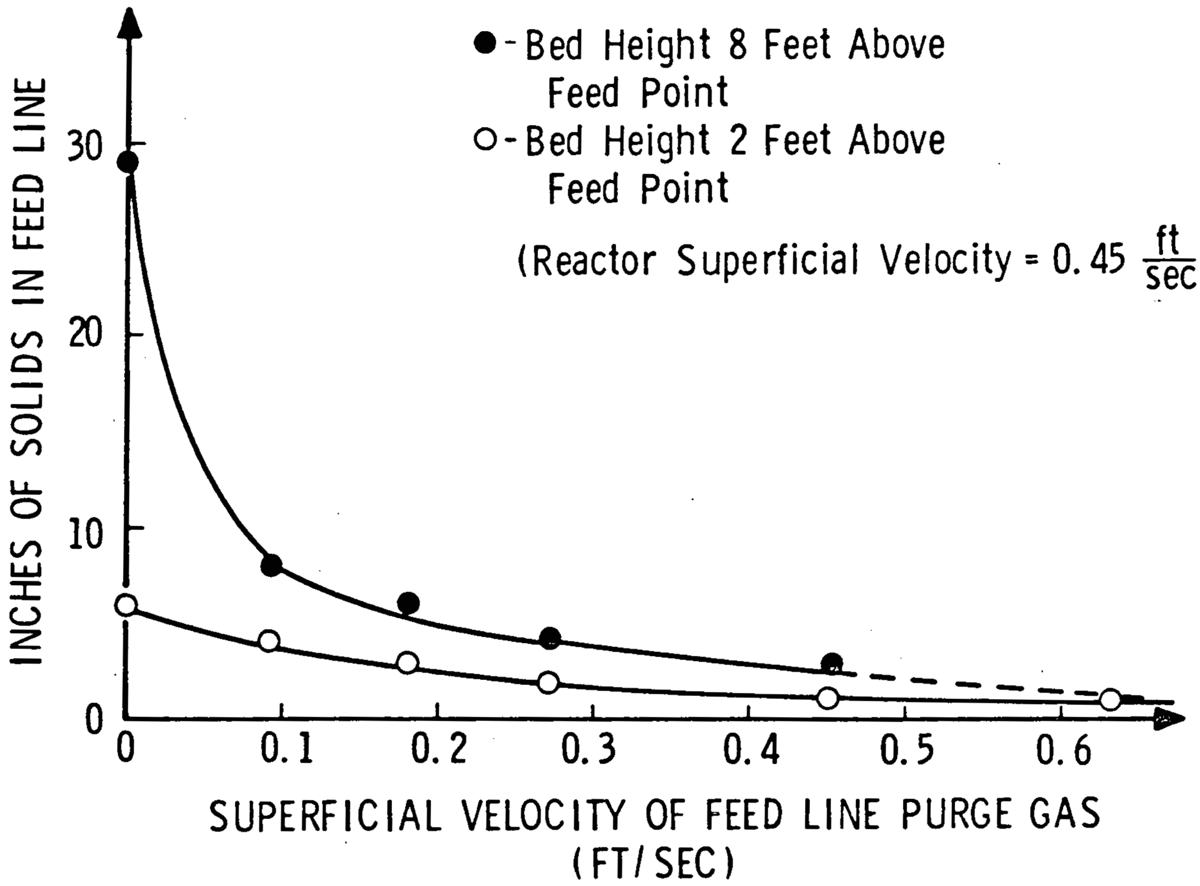
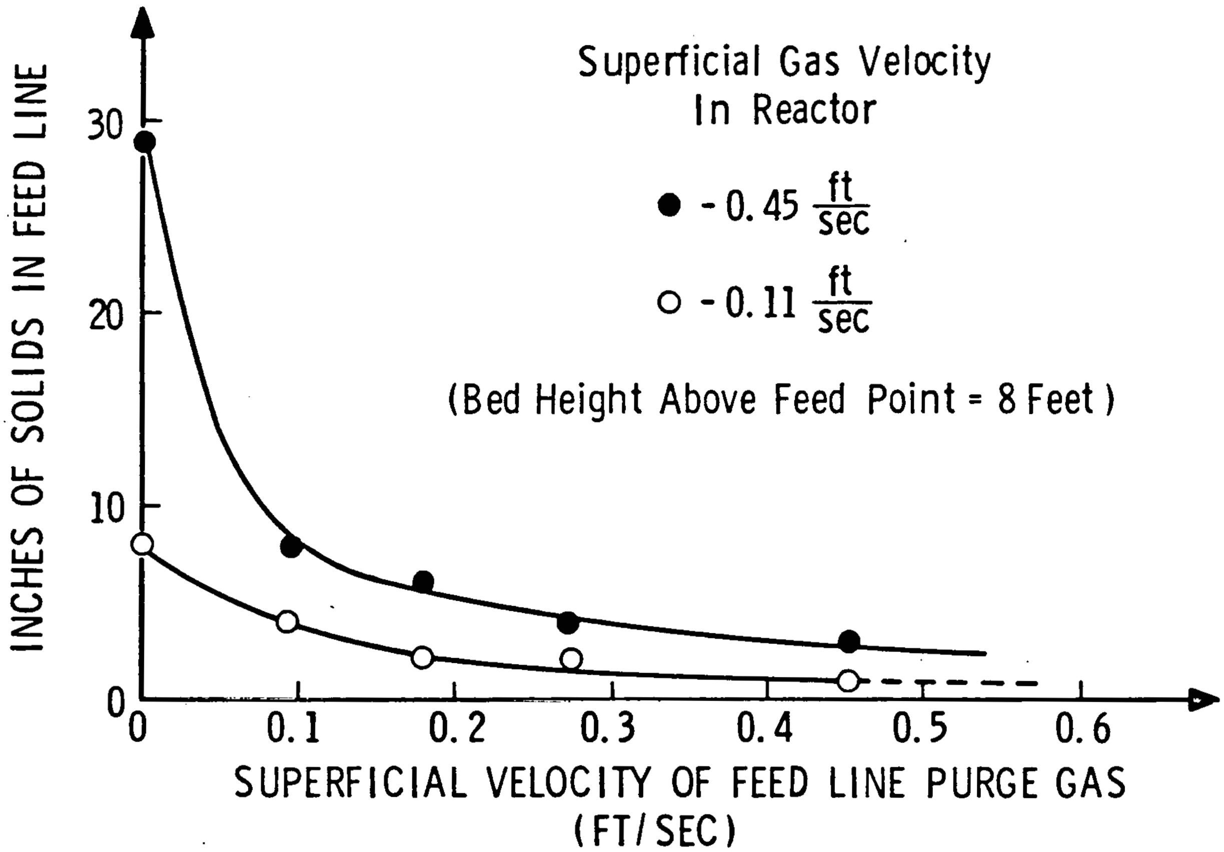


FIGURE 3.1-4

PURGE GAS REQUIREMENTS TO PREVENT SOLIDS FROM BACKING UP INTO FEED LINE

EFFECT OF SUPERFICIAL VELOCITY IN THE REACTOR



gas purge so that the superficial gas velocity through the 3/4" line is at least 0.2-0.3 ft/sec.

Lockpot Operation

As mentioned earlier, solids feed rate is controlled by the frequency of the feed cycle. A catalyzed coal feed rate of 115 lbs/hr (the PDU design basis) would require one complete feed cycle every 140 seconds if the lockpot filled and emptied completely during the cycle. Experiments were carried out to determine how to operate the feed system in order to achieve the necessary cycle time. Initial experiments were designed to determine the length of time to empty the lockpot under different operating conditions.

The lockpot would not empty when the bottom valve was opened unless there was a gas purge directly to the lockpot of about 8 ACFH. At this low purge rate the lockpot drained erratically and occasionally would not empty completely. When the purge rate to the lockpot was increased above 8 ACFH, not only did the time required to empty the lockpot decrease but also the reproducability of duplicate runs improved because the lockpot drained more smoothly. Purge location was very important in these experiments. A gas purge to the feed line below the lockpot was not as effective as a direct purge to the lockpot. Figure 3.1-5 shows how an increase in gas purge results in a decrease in the time required to empty the lockpot.

As shown in Figure 3.1-2, after the solids leave the lockpot, they travel through the feed line and into the reactor. The first part of the line is vertical with an inside diameter of 2-5/8". It then goes through a 45" bend and into an eccentric reducer where the line is reduced to 3/4" ID.

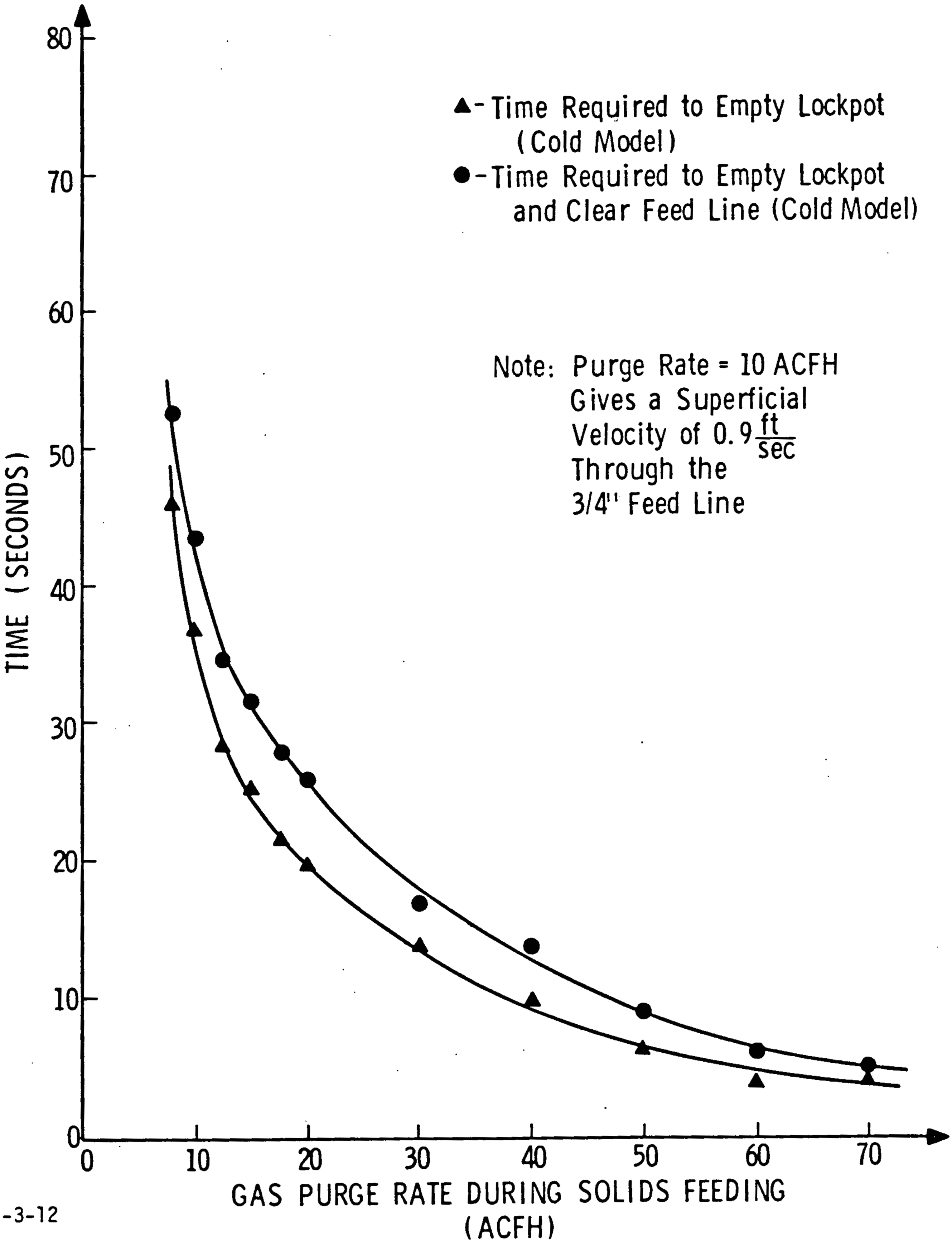
Experiments were conducted in the cold model to determine how fast the solids would move through the feed line and into an actively fluidized bed. The feed line on the Plexiglas model is six feet long, which is considerably shorter than that of the PDU. The longer feed line in the PDU should not have a significantly higher resistance to solids flow than the feed line in the model because most of the resistance to solids flow results from bends and constrictions in the line and the resistance of solids flow into the fluidized bed. These effects are present in both the cold model and the PDU.

It has already been shown that solids will back up from the fluidized bed into the feed line unless a small gas purge is maintained. When feeding solids into the reactor, a higher purge rate of at least 12 ACFH was needed. This is more than the minimum purge required to empty the lockpot. If the purge rate was below 12 ACFH, the solids did not move into the reactor from the 3/4" section of the feed line as fast as they drained from the lockpot and so the level of solids in the feed line rose. Frequently this resulted in compaction and bridging of solids which caused the feed line to plug.

At purge rates in the range of 12 to 18 ACFH, the solids moved through the feed line and into the reactor in spurts. Above 18 ACFH there was enough gas moving with the solids to keep the material from compacting and maintain smooth solids flow. Figure 3.1-5 shows the time required to empty the lockpot and to clear the feed line for a range of gas purge rates from 10 to 70 ACFH. Higher purge rates gave greater solids mass flow rates into the reactor.

FIGURE 3.1-5

PURGE GAS REQUIREMENTS FOR FEEDING SOLIDS INTO THE PDU



79B-6-3-12

Recommendations for PDU Operation

The results from the cold model have indicated that it should be possible to achieve smooth operations and the required coal feed rates to the PDU by supplying gas purges to the feed system. When the lockpot is being filled or the bottom lockpot valve is closed, purge gas must enter directly into the feed line below the lockpot at a rate of at least 2.5 ACFH (0.25 ft/sec) to keep solids from moving from the reactor up into the feed line. When the bottom lockpot valve is opened to feed solids, a gas purge directly into the lockpot in the range of 20-60 ACFH is needed to drain solids from the pot. Once the solids are out of the lockpot, a gas purge is required to feed the solids into the fluidized bed. This gas can be supplied through the lockpot purge if the bottom lockpot valve remains open.

PDU Fines Return System

As demonstrated by past operations of fluid bed catalytic coal gasification pilot plants, solid particles are entrained in the gas stream leaving the reactor. These particles are generally less than 50 microns in diameter and have a higher carbon content than char in the fluidized bed. The difference in the carbon content of the two types of char can be attributed to relatively low residence times for the smaller particles which leave the reactor more quickly than larger particles. The fine char carried overhead in the gas stream comes from two sources. Part of it is char from fine feed coal particles, while the rest is the product of attrition of larger particles in the fluidized bed. This fine, high carbon char should be returned to the reactor for further gasification to achieve a higher overall carbon conversion and higher process efficiency.

On the PDU, the system to return the fine char to the reactor consists of a cyclone, dipleg, intersection block and a transfer line as shown in Figure 3.1-1. The cyclone and dipleg are not inside the reactor due to its relatively small diameter. The fact that the cyclone and dipleg are external to the reactor results in a special design for the dipleg return which is characteristic of smaller fluidized bed units. At the bottom of the dipleg is an intersection block from which a transfer line leads back to the reactor. The transfer line begins at an angle 60° from the horizontal, goes through a 15° bend and enters the reactor at 45° from the horizontal.

The design of the fines return system is such that the rate of fines return to the bed should be controlled by pressure balance. If the solids in the dipleg, intersection block, transfer line, and reactor are properly fluidized, the system should behave like a manometer. As fine char falls into the dipleg from the cyclone, the level of solids rises in the dipleg, causing an increase in static pressure at the bottom of the dipleg. If this pressure is greater than that at the point at which the transfer line enters the reactor, then the fines should move from the dipleg into the reactor.

Cold Model Fines Return System Studies

The cold model is equipped with a fines return system like the one previously described. Internal dimensions of the model are nearly identical to those of the PDU except that the length of the dipleg is approximately 14 feet compared to the 70 foot dipleg on the PDU. Initial experiments on the model were designed to investigate solids flow behavior in the dipleg and transfer line.

Dipleg Operation

The fines in the dipleg should be fluidized slightly above minimum fluidization if they are to flow smoothly through the intersection block and into the transfer line. Too little purge gas in the cold model resulted in solids slumping, compacting, and bridging in the dipleg, causing solids flow to stop. Once this occurred, it was difficult to reestablish a fluidized state in the dipleg. Sudden increases in gas flow caused plugs of solids to move up the dipleg like a piston. This behavior was accompanied by an increase in pressure drop which was characteristic of flow through a packed bed. The most successful procedure for refluidizing compacted solids was to slowly increase and decrease the gas flow to the dipleg. This resulted in a smooth transition from a packed to a fluidized bed. Excess gas flowing up the dipleg led to slugging in the bed of fines.

The gas flowing through the dipleg must pass through the base of the cyclone and out the top with the gas from the reactor. The original cyclone design called for a throat diameter of 13/16", as shown in Figure 3.1-6. This would mean that the superficial gas velocity of the dipleg purge gas would be ten times greater through the cyclone throat than through the 2-5/8" ID dipleg. Experiments were carried out to determine whether cyclone performance was affected by the dipleg purge gas passing through the cyclone.

Dipleg purge rates above 3 ACFH resulted in cyclone plugging. Beginning at the throat of the cyclone, the polypropylene powder clung to the walls of the cyclone cone and accumulated there until it plugged completely. The cyclone did not plug when the dipleg purge rate was below about 3 ACFH. These results indicate that gas flowing up through the cyclone does affect cyclone performance. The total purge gas rate to the dipleg should be kept to a minimum during operation of the PDU to avoid high superficial gas velocities at the cyclone throat which would interfere with cyclone performance.

A change was made in the cyclone design for the PDU based on these experiments. The throat diameter was increased from 13/16" to 1-1/8", reducing the gas superficial velocity by nearly one-half in the throat of the cyclone. This should reduce the frequency of cyclone plugging.

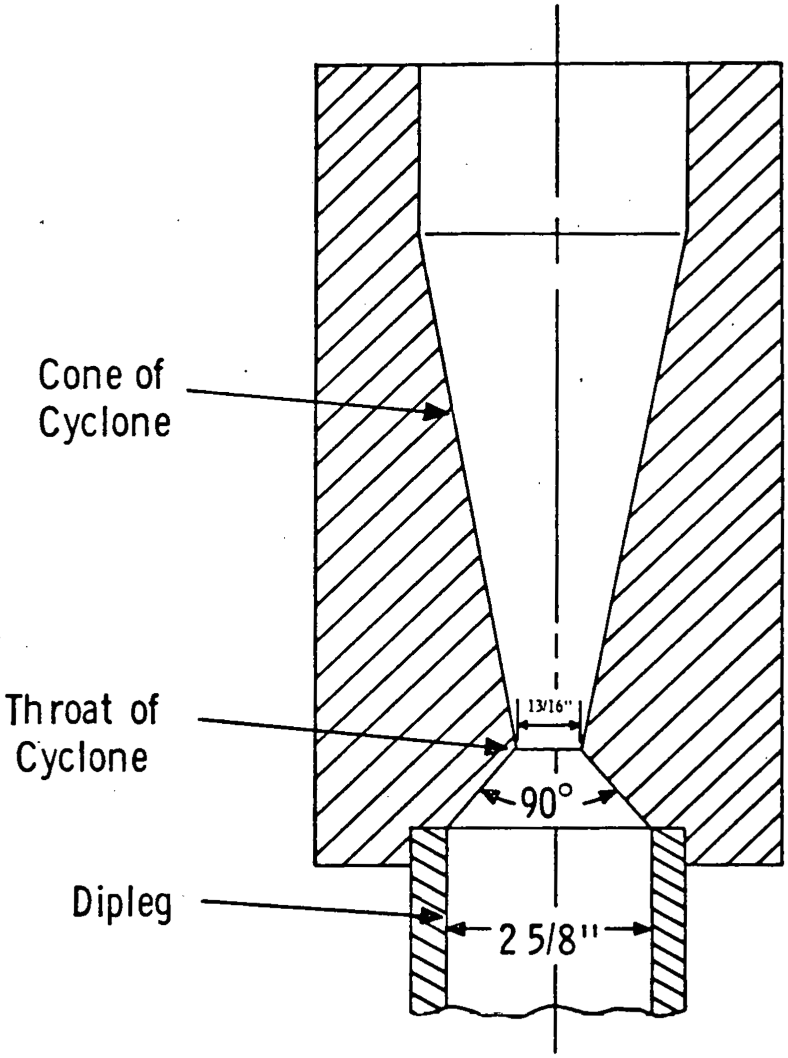
Transfer Line Operation

Solids must travel up the inclined transfer line to return to the reactor from the dipleg. Gas must be fed into the transfer line to keep the particles moving in order that they will flow back into the reactor. Gas was supplied to the transfer line at various rates and the behavior of the solids in the inclined tube was observed.

Gas superficial velocities below about 0.20 ft/sec in the transfer line resulted in stagnant solids along the entire length of the line. As the superficial gas velocity was increased, solids activity increased along the top of the transfer line while solids in the bottom of the line remained

FIGURE 3.1-6

ORIGINAL CYCLONE DESIGN - BOTTOM OF CYCLONE



stationary. Solids in top half of the 60° section of the line began to slug at a superficial gas velocity of about 0.3 ft/sec. Slugs broke up at the angle between the 60° and 45° sections and solids in the 45° section were motionless. Gas velocities of about 1-2 ft/sec were required to eliminate zones of stagnant solids along the bottom of the transfer line. At these gas velocities, the solids slugged up the line and then flowed back down the bottom of the line. Generally, the solids activity in the 60° part of the transfer line was greater than that in the 45° part of the line.

Intersection Block Studies

Recent experiments on the cold model were designed to determine how to control dipleg and transfer line fluidization simultaneously by varying purge gas rates and locations. The purge gas can enter the system at any of five locations in the intersection block. A diagram of the intersection block with the purge locations numbered 1 through 5 is shown in Figure 3.1-7. Based on the experiments described above, most of the gas entering the fines return system at the intersection block should travel up the transfer line. High gas flow rates are required in the sloping line to eliminate zones of stagnant solids. Purge gas flow traveling up the vertical dipleg should be kept to a low value to avoid interference with cyclone performance but should be enough to keep the solids in the dipleg fluidized.

Each of the intersection block purges is equipped with a sliding tube that can be moved into the intersection block as indicated in Figure 3.1-7. Sliding the tube into the intersection block to different positions results in different gas flow patterns.

Purge location #3 gave the best control of flow up either the dipleg or the transfer line but not to both simultaneously. When the tube was extended beyond the entrance to the dipleg, most of the gas went into the transfer line and there was little solids motion in the dipleg. When the tube was retracted to the wall (as shown in Figure 3.1-7), most of the purge gas flowed up the dipleg. Purge location #2 produced gas flow patterns similar to location #3 but control was not as good. Most of the purge gas flowed up the transfer line in the most extended tube position, but there was intermittent slugging in the dipleg which did not occur when purge location #3 was used. Purges #1 and #4 supplied purge gas only to the vertical dipleg at all tube extensions. Purge #5 gave little control of flow up the transfer line. Most of the purge gas flowed up the dipleg when the tube was extended to greater than 1/3 of the maximum extension into the intersection block.

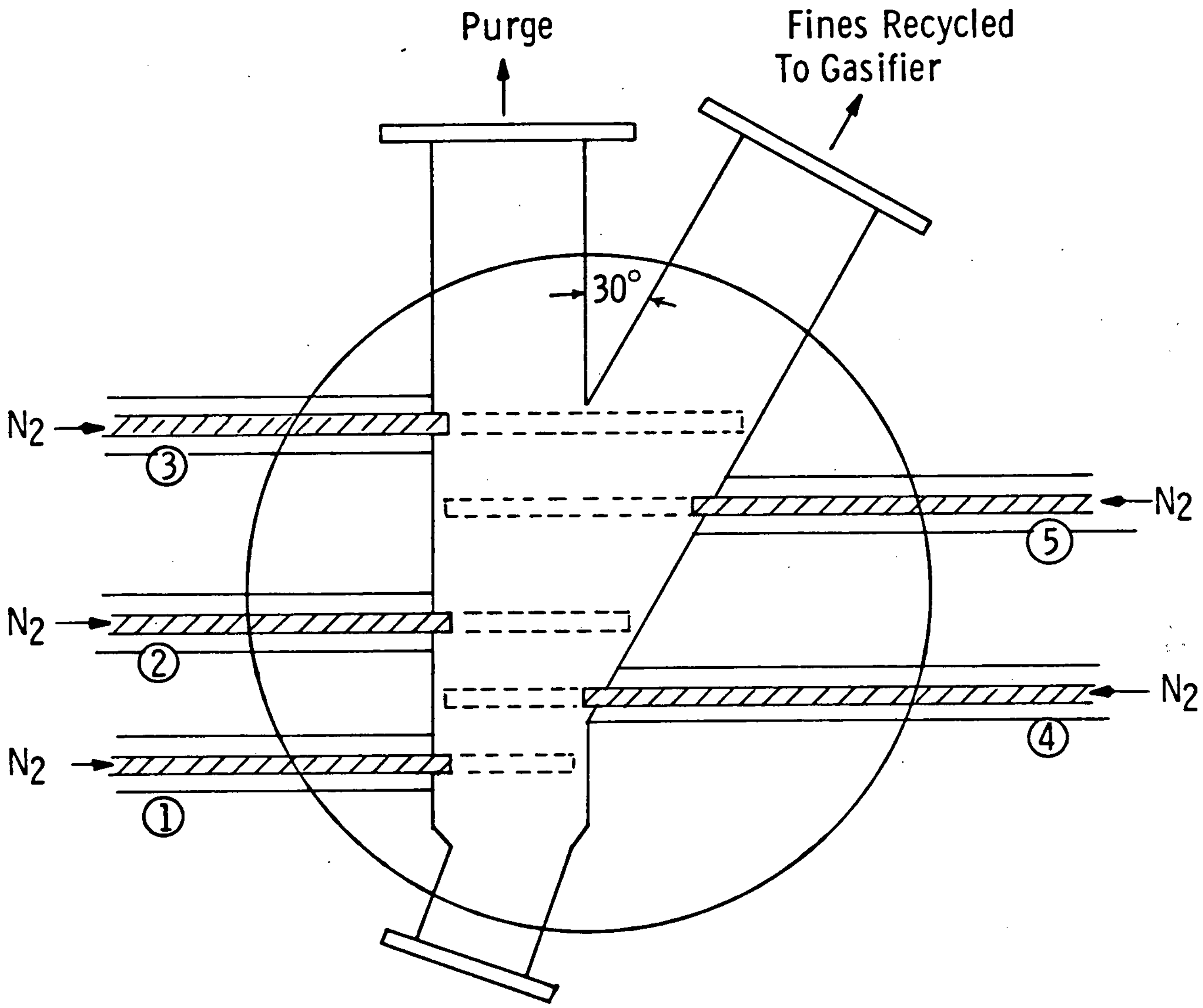
These results indicate that purge location is important in controlling fluidization of the fines return system. A purge directly into the base of the transfer line is required to supply high gas flow rates to the transfer line while allowing negligible amounts of gas into the dipleg. Required flow to the dipleg can be supplied from other purge locations in the intersection block.

On the basis of the above work, the PDU intersection block has been modified to provide purge locations which should control flow of purge gas to the dipleg and intersection block.

FIGURE 3.1-7

DIPLEG INTERSECTION BLOCK

Cold Model Version



Future work on the cold model of the PDU will include more experimentation on the fines return system to evaluate response of the system to unit pressure upsets.

3.2 Off-line Data Reduction and Reconciliation

The off-line data reconciliation program for the PDU has been developed, debugged, and tested using a set of simulated data. This program provides a tool for obtaining consistent and reliable data from PDU operations. To perform this analysis, the program accepts raw operations data from different sections of the PDU, processes the data, and then generates detailed stream reports for use in correlations, commercial plant study design, and simulation studies. The program was written so that calculations of different models of PDU operations such as once-through or recycle can be carried out with the same program. The program can also be used to reconcile operations data from other catalytic gasification pilot plants. The operation of the data reconciliation program is summarized as follows.

Input Data

Two types of input data, reconcilable and nonreconciliable, must be submitted to execute this program. Reconcilable data are gas and solid flow rates and compositions which will be adjusted to satisfy the material balance constraints. Nonreconciliable data are the stream and unit temperatures and pressures.

Reconciliation Algorithm

The reconciliation algorithm is an iterative procedure which makes minimal adjustments to the process data to satisfy the constraints. In each iteration, a new set of reconciled data is determined through the use of redundant data and the knowledge of the reliabilities of instrumentation. The iteration procedure is continued until a set of self-consistent values is obtained.

Reporting of Data

A complete listing of the measured and reconciled values of all reconcilable variables will be reported. The report also lists the reliabilities associated with the measured values and the percent changes between the measured and reconciled values. This report aids the users in detecting erroneous measured data.

The reconciled data will be used in the following calculations:

- Gasification and methanation reaction rates
- Reaction equilibrium constants
- Gasifier fluid bed properties
- Solid entrainment
- Carbon and steam conversions

The results will be put in a detailed report, together with the following information:

- Unit conditions and performance
- Unit material balances
- Gas and solid stream compositions
- Solid particle size distributions
- Catalyst distribution

4. Advanced Study of the Exxon Catalytic Coal Gasification Process (Reporting Category C04)

4.1 Kinetics of Gasification and Devolatilization

A laboratory program is in progress to expand the kinetic data base for gasification of Illinois No. 6 coal. Kinetic data will be obtained in the 1200-1350°F temperature range. Experimental studies are being made in a bench scale fixed bed gasification unit. Details of this unit can be found in the July-September, 1978 Quarterly Report.

As reported in the October-December, 1978 Quarterly Report, a series of shakedown runs was made at 1300°F and 500 psig using Illinois No. 6 char with a catalyst loading of 20% (wt) potassium carbonate on dry coal. The purpose of these runs was to check the operability of the unit before initiating the temperature study. During the data workup for these runs, a problem with the gas analyses was discovered. The sum of the unnormalized compositions of the individual product gases was significantly less than 100%.

Two possible causes of this low total were:

(1) incorrect calibration gas analysis, and

(2) nonlinear response of the gas chromatograph with respect to gas composition.

Samples of product gas were collected during subsequent runs and analyzed on a mass spectrometer as well as on several other gas chromatographs on site. A comparison of the results from the on-line gas chromatograph with those from the other systems indicated a nonlinearity in the on-line analysis with respect to hydrogen concentration. This nonlinearity was confirmed through the analysis of gas samples of known hydrogen composition on the on-line unit.

Although the incorporation of a nonlinear hydrogen response factor into the data workup procedure resulted in unnormalized product gas analyses totaling essentially 100%, the final results indicated a much lower gasification rate than that obtained during the predevelopment phase of catalytic gasification research (Contract No. E(49-18)-2369). As a result of these observations, the entire gas chromatograph system was again checked for gas leaks. Several leaks were found throughout the gas chromatograph sampling system as well as a malfunctioning thermal conductivity detector. The entire gas chromatograph sampling and detection system was then rebuilt. A new thermal conductivity detector as well as new automatic switching valves were installed in the unit. New chromatographic columns were also installed in accordance with the gas chromatograph manufacturer's specifications. The stability of the gas chromatograph's response, unfortunately, was not significantly increased following the replacement of the items mentioned above.

In addition, various efforts to service the unit by the manufacturer have not been successful.

In view of the recurring problems with the on-line gas chromatograph as well as the lack of success by the manufacturer in servicing the instrument, a new gas chromatograph system has been purchased for the fixed bed unit. The experimental program using the high pressure fixed bed unit will resume following the delivery and installation of the new chromatograph.

In the interim, kinetic data will be obtained in a mini-fluid bed gasification unit. A schematic of this unit is shown in Figure 4.1-1. The reactor portion of the unit consists of a 1/4" I.D. quartz U-tube inside a hot steel block. Water is fed to the U-tube using a small syringe pump and is vaporized in the reactor. Ceramic beads are placed in the inlet leg of the U-tube to enhance the vaporization process and help disperse the flow. Argon carrier gas is also fed to the unit in order to provide good fluidization of the char sample. The exit gases from the reactor flow into an oxidizer where all carbon species are converted to carbon dioxide. After condensing any unreacted steam, the gas stream is bubbled through a sodium hydroxide solution where the amount of total carbon converted is automatically monitored using the change in conductivity of the solution. Initial studies will be made using Illinois No. 6 char produced by the Fluid Bed Gasifier (FBG) earlier this year.

4.2 Catalyst Reactions with Coal and Ash

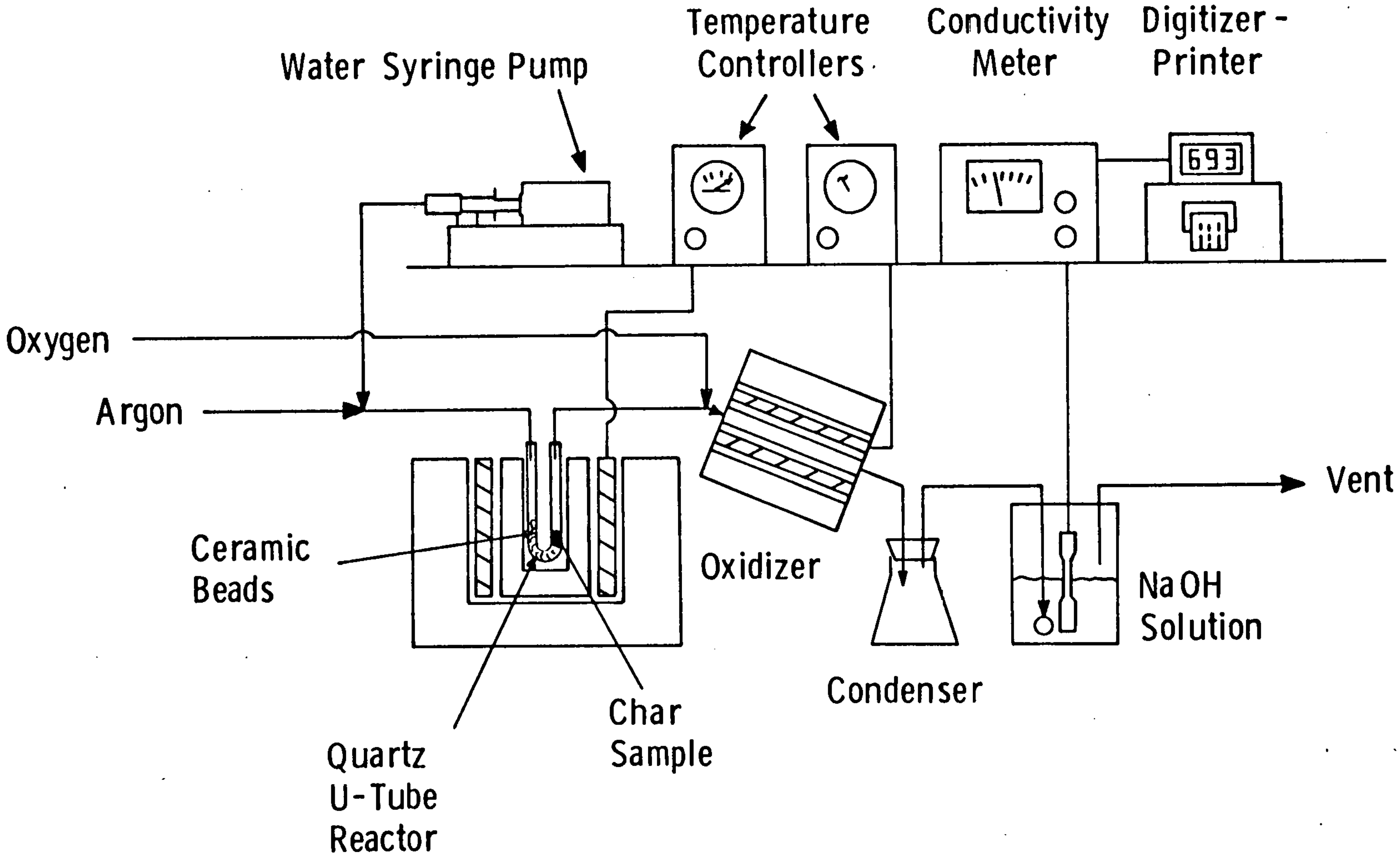
As reported in the October-December, 1978 Quarterly Report, the Fluid Bed Gasifier (FBG) had been operated successfully during the predevelopment contract (1977) on a feedstock of potassium carbonate (K_2CO_3) catalyzed Illinois No. 6 coal. During the last quarter of 1978, operation with a new carload of Illinois No. 6 coal and with potassium hydroxide (KOH) as the catalyst was accompanied by some initial operability problems as well as a lower fluidized bed density than was experienced in the predevelopment work. Operations were improved by removing the large (+16 mesh) particles from the feed coal and fluidized bed density was increased by exposing the catalyzed coal to air prior to loading in the FBG.

Bench scale studies were initiated to address questions involving the effect of variables in catalyst impregnation on both agglomeration and the bulk density of the devolatilized coal. Areas investigated were:

- FBG Operations Support
    + Analyses of raw coals
    + Catalyst impregnation of raw coal sieve fractions
    + Effect of changing catalyst on bulk density
- Test for Measure of Oxidation of Coal

FIGURE 4.1-1

SCHEMATIC OF MINI-FLUID BED REACTOR UNIT



FBG Operations Support

Analyses of raw coals

Samples of coal from both the predevelopment (1977) and 1978 operations were submitted for microscope analysis. Petrographic analysis showed the two samples to have similar maceral compositions and mineral matter content. The major difference between the predevelopment and the current supply is that the older coal has a higher reflectance. This may be due to either different types of vitrinite or a higher oxidation level of the older coal. Since it is uncommon for samples from the same mine to have different types of vitrinite, the higher reflectance suggests that the older coal is more highly oxidized. This difference in oxidation level is confirmed by the elemental analysis shown in Table 4.2-1 which shows that the predevelopment coal has a significantly higher oxygen content than the 1978 coal.

Table 4.2-1

Elemental Analysis of Untreated FBG Feed Coals

- Analyses on dry coal basis

	Predevelopment Coal (1977)	1978 Coal
Moisture	2.53	2.77
SO_3 Free Ash	9.96	10.28
C-H Residue	10.55	9.40
Volatile Matter	40.70	43.47
Btu/lb	12117	12280
C	67.51	69.32
H	4.88	4.94
N	1.10	1.09
O	12.09	9.73
S	4.29	4.24
$S_{pyritic}$	1.05	1.66
$S_{sulfate}$	0.09	0.03
$S_{organic}$	3.15	2.59
SO_3 Free Ash Elements		
P_2O_5	0.37	0.22
SiO_2	52.75	50.30
Fe_2O_3	17.15	20.22
Al_2O_3	19.79	18.60
TiO_3	1.04	0.96
CaO	3.92	4.18
MgO	1.24	1.09
K_2O	2.48	2.43
Na_2O	1.25	1.31

The difference in oxidation level agrees with the observed increase in bed density using catalyzed coal which was air-exposed prior to loading in the FBG, but the result is inconclusive since the oxidation level of the older coal could have changed during storage. The effect of oxidation level on char density and agglomeration is therefore being studied on samples whose history can be better defined. It is essential, therefore, to have a sensitive measure of the level of oxidation of coal samples.

Catalyst impregnation of raw coal sieve fractions

Previously reported analytical data (October-December, 1978 Quarterly Report) shows that the large (+20 mesh) particles in the 1978 FBG feed coal had a lower catalyst loading than the remainder of the feed. Laboratory chars prepared from these particles showed a high degree of agglomeration. The study described below was performed to address the question of whether or not the low catalyst loading was the cause of agglomeration of the large particles during devolatilization.

A sample of uncatalyzed FBG feedstock was divided into sieve fractions. Portions of the individual fractions were then treated with either KOH or K_2CO_3 catalyst. The laboratory procedure for catalyst impregnation simulated that used in the Catalyst Addition Unit (CAU) of the FBG. In this procedure, the coal was mixed with a 30% (wt) catalyst solution in the appropriate quantity to result in a final catalyst loading on the coal equivalent to 15% (wt) K_2CO_3. Analysis of the sieve fractions treated in this manner showed that each fraction had the same catalyst loading.

The mixture was then dried under nitrogen and the treated coal samples were charred in the laboratory. The chars were examined for agglomeration and their loose bulk densities measured. The results are shown in Tables 4.2-2 and 4.2-3 below.

Table 4.2-2

KOH Catalyzed FBG Feedstock

Sieve Cut	Loose Bulk Density of Char (g/cc)	Agglomeration
+20	.51	No
-20 + 50	.52	No
-50 + 100	.56	No
-100	.52	No

Table 4.2-3

K_2CO_3 Catalyzed FBG Feedstock

Sieve Cut	Loose Bulk Density of Char (g/cc)	Agglomeration
+20	.58	No
-20 + 50	.53	No
-50 + 100	.55	No
-100	.55	No

The lack of agglomeration, particularly for the +20 mesh particles, suggests that uniform catalyst impregnation would allow this sieve size to be included in the reactor feed. In addition, the observed loose bulk densities have virtually the same value for chars from coal of all particle sizes and for equivalent loadings of both KOH and K_2CO_3 catalysts.

Effect of changing catalysts on bulk density

At equimolar K^+ loadings, KOH and K_2CO_3 are known to be equivalent gasification catalysts. The possible effect of changing the catalyst on the FBG fluidized bed density was investigated by determining the loose bulk densities of coals and their chars catalyzed by either K_2CO_3 or KOH.

Samples of raw coal from the current grind and the predevelopment grind were impregnated with either KOH or K_2CO_3 and dried under vacuum. Catalyst loadings equivalent to 15% (wt) K_2CO_3 were used for all cases. The loose bulk densities of the raw and catalyzed coals are presented in Table 4.2-4. Also included are bulk densities of chars produced in a laboratory muffle furnace from both the laboratory prepared catalyzed coals and FBG feed coals prepared by the Catalyst Addition Unit (CAU) operated in conjunction with the FBG. All chars were produced by heating at 1300°F under nitrogen in the muffle furnace for three minutes.

Table 4.2-4

Bench Scale Loose Bulk Densities (g/cc)

	Predevelopment Coal		Current Coal	
Sample	Coal	Char	Coal	Char
Untreated	.66	--	.63	--
K_2CO_3 treated, laboratory	.76	.56	.80	.60,.60
KOH treated, laboratory	72	.58	.68	.55,.55
K_2CO_3 treated, CAU sampled, 6/22/77	.81,.79	.64	--	--
KOH treated, CAU sampled 11/25/78	--	--	.79,.76	.64

The data show that catalyst impregnation increases the bulk density of the coal as expected. The densities of the raw coals, both untreated and catalyzed, are the same for both the predevelopment and the current coals. The densities of the chars are also not significanly different for different catalysts or different carloads of coal. The densities of the coals and chars produced by catalyst impregnation on the pilot plant CAU are slightly higher than the densities of the laboratory prepared samples. This may be due to variation of the catalyst loading on the CAU treated samples. Overall, the loose bulk densities of these samples support the conclusion that changing the catalyst is not directly responsible for the lowering of the FBG bed density.

The observed value of the bulk density of the devolatilized coal (0.55 g/cc) is higher than that reported for the FBG reactor bottom char at low conversion (0.28 g/cc), suggesting that muffle furnace char may not be directly comparable to reactor char.

Test for a Measure of Oxidation of Coal

The agglomeration properties of coal during devolatilization and the density of the gasifier bed appear to depend on both the presence of catalyst and the extent of oxidation of the coals. Therefore, a sensitive measure of the oxidation level of coal samples is being sought.

The literature available on coal oxidation indicates that a number of qualitative and semi-quantitative procedures are available. These include elemental analysis for oxygen by neutron activation, infrared spectroscopy, nuclear magnetic resonance spectroscopy, free swelling index, solubility and pH measurements, reaction with organic dyes, microscopic reflectance, composition of gases produced during devolatilization, reactivity under liquefaction conditions, and thermal content analysis. Most of the above are known to be qualitative or insufficiently sensitive to small changes in oxidation level.

Two methods were selected for the initial studies. These are neutron activation analysis for oxygen and thermal content analysis.

Samples have been prepared from unoxidized Illinois No. 6 coal samples by exposing them to air under varying conditions. These samples have been submitted for analysis and the results will be described in a future report.

5. Engineering Research and Development
(Reporting Category C20)

Engineering research and development studies are being carried out under the Catalytic Coal Gasification (CCG) Process Development Contract in conjunction with the laboratory bench-scale research and process development unit (PDU) operations. This work includes both engineering and cost studies to evaluate process improvements and to guide the continuing laboratory programs, and engineering technology programs to develop fundamental process and equipment technology to support the laboratory and engineering efforts. The overall objective of the engineering work is to define the conceptual commercial CCG process at the end of the contract period.

The engineering research and development work under the CCG Process Development Contract is divided into four major subtasks:

Cost Reduction and Laboratory Guidance Studies
Systems Modeling
Process Definition
Engineering Technology Studies

During the period covered by this report, the engineering efforts focused on the first, second, and fourth sub-tasks. Work on the Process Definition is not scheduled until January, 1980.

5.1 Cost Reduction and Laboratory Guidance Studies

CCG Commercial Plant Study Design - Offsites Revision

A Catalytic Coal Gasification Commercial Plant Study Design was prepared during the latter part of the CCG Process Predevelopment Program which was completed in January, 1978 under Contract No. E(49-18)-2369. The results of the "CCG Study Design" are documented in the Final Project Report for that contract (FE-2369-24). This was a detailed study involving substantial engineering efforts on material and energy balances, equipment specifications, and investment cost estimating.

Offsites facilities (including materials handling, utilities, and general offsites) constituted 40% of the total plant direct and indirect investment cost for the CCG Study Design. Although considerable effort was involved in specifying the offsites facilities for the Study Design, for the most part these areas were studied in less engineering depth and specified in less detail than the onsites process sections. Because the onsites and offsites design work proceeded at the same time, some inconsistencies developed between the final onsites utilities demands and the estimated demands used in specifying the utilities sections. Also, the process wastewater rate used in sizing the wastewater treating facilities was underestimated. A preliminary plant layout was used in specifying common on-site facilities and off-site piping for utilities distribution and for industrial sewers. A final look at the plant layout indicated that these requirements were probably overestimated.

In view of these factors, a revised offsites facilities definition and cost estimate was prepared to firm up the CCG Study Design in this important area. The revised Study Design will serve as the "base case" for screening studies to evaluate new data, process improvements, and optimum process conditions under the present Process Development Contract. As a result of the offsites revision, the accuracy of such screening studies will be improved.

Most of the changes in this offsites revision were simply adjustments to equipment sizes to correct for inconsistencies between the initial and final utilities demands and plant layout requirements. However, more extensive changes were made in two sections. First, in the wastewater treating section, more detailed consideration was given to water quality and reuse options to better define treating needs and further reduce plant makeup and effluent water rates. Second, the flue gas desulfurization (FGDS) process was changed from a regenerative system using sodium carbonate to a once-through system using lime scrubbing. This change allowed integration of lime scrubbing offsites with other CCG plant offsites. For example, lime receipt for FGDS was integrated with lime receipt for onsite catalyst recovery, which uses lime as feed to $Ca(OH)_2$ digestion. Common absorbers were utilized to handle flue gas from the offsite boilers, the feed coal dryers, and the catalyst addition dryers, all of which are coal fired. In addition to these integration advantages, the technology and costs for lime (and limestone) scrubbing are better defined today than for regenerative FGDS.

In general, the revised Study Design was prepared using the same approaches as the earlier Predevelopment Program Study Design. Except for the change in the FGDS process described above, the project basis is the same. The onsites process bases and material and energy balances are also unchanged. Utilities balances were updated to reflect the final onsites demands and the demands of the revised offsites facilities. Equipment lists for the revised offsites were developed by engineers specializing in offsites design. Direct equipment costs were estimated using the same techniques and cost bases used for Exxon's commercial projects. Indirect costs were estimated based on recent experience with large projects. Contingencies were included in the total investment estimate, also based on Exxon practices for actual projects.

The revised investment for the CCG Study Design is presented in Table 5.1-1. (This updates Table 4.8-1 of the Predevelopment Report FE-2369-24.) The total investment is 1,530 M$ for the pioneer commercial plant feeding Illinois No. 6 coal and producing 257 billion Btu per stream day of SNG (substitute natural gas). This is for a January, 1978 cost level at an Eastern Illinois location.

The revised Study Design investment is 110 M$ less than the investment estimated during the Predevelopment Program, a reduction of over 7%. The investment changes are broken down by plant section in Table 5.1-2, starting with the Predevelopment Program Study Design investment of 1,640 M$. The key factors which have contributed to the overall investment change are:

TABLE 5.1-1

CATALYTIC COAL GASIFICATION
COMMERCIAL PLANT STUDY DESIGN

INVESTMENT FOR PIONEER PLANT

Basis:
- January, 1978 Instant Plant
- Eastern Illinois Location
- 257 Billion Btu/Stream Day SNG (HHV Basis)

	Investment Breakdown	
Plant Section	Million $	% (1)
ONSITES		
Coal Drying	27	2
Catalyst Addition	18	2
Reactor System	197	18
Product Gas Cooling and Scrubbing	86	8
Sour Water Stripping and Ammonia Recovery	20	2
Acid Gas Removal and Sulfur Recovery	161	15
Methane Recovery System	44	4
Refrigeration	31	3
Catalyst Recovery	39	3
Common Onsite Facilities	55	5
ONSITES SUBTOTAL	678	62
MATERIALS HANDLING		
Coal Handling and Storage	19	
Coke/Char Handling	5	
Chemicals Handling and Storage	20	
By-Products Storage and Shipping	3	
Waste Solids Handling and Disposal	27	
MATERIALS HANDLING SUBTOTAL	74	7
UTILITIES		
Raw Water/BFW Treating	29	
Steam Generation and Distribution	120	
Cooling Water	9	
Electric Power Distribution	23	
Miscellaneous Utilities	5	
Flue Gas Desulfurization (2)	51	
UTILITIES SUBTOTAL	237	22
GENERAL OFFSITES		
Wastewater Treating	48	
Safety and Fire Protection	13	
Site Preparation	6	
Miscellaneous Offsites	34	
GENERAL OFFSITES SUBTOTAL	101	9
TOTAL DIRECT AND INDIRECT COSTS	1,090	100
PROCESS DEVELOPMENT ALLOWANCE (25% of Onsites Direct & Indirect Costs)	169	
PROJECT CONTINGENCY (25% of Total Direct & Indirect Costs)	271	
TOTAL ERECTED COST	1,530	

Notes:

(1) Percentage breakdown of investment is based on total direct and indirect costs excluding process development allowance and project contingency.

(2) Includes desulfurization for flue gases from steam generation (coal-fired boilers) and from coal drying and catalyst addition.

TABLE 5.1-2

CCG STUDY DESIGN
SUMMARY OF INVESTMENT CHANGES

	Investment Million $	
● TOTAL ERECTED COST FOR PREDEVELOPMENT PROGRAM STUDY DESIGN		1,640
● CHANGES IN TOTAL ERECTED COST		
ONSITES		
Coal Drying	(11)	
Catalyst Addition	(6)	
Common Onsite Facilities	(8)	
Other Sections	2	
MATERIALS HANDLING		
Coal Handling and Storage	(33)	
Chemicals Handling and Storage	(7)	
Other Sections	(3)	
UTILITIES		
Steam Generation and Distribution	3	
Flue Gas Desulfurization	(16)	
Other Sections	(10)	
GENERAL OFFSITES		
Wastewater Treating	7	
Other Sections	-	
TOTAL DIRECT AND INDIRECT COSTS		(82)
PROCESS DEVELOPMENT ALLOWANCE		(6)
PROJECT CONTINGENCY		(22)
● TOTAL ERECTED COST FOR REVISED STUDY DESIGN		1,530

- Costs are substantially lower in materials handling sections (including coal drying and catalyst addition, which are grouped with the onsites). The lower investments stem in part from modest reductions in facilities requirements made as part of the offsites revisions. For example, the electrostatic precipitators used to remove fines from flue gases produced in the coal dryers and the catalyst addition dryers were deleted. Fines removal from these flue gases is now accomplished by venturi scrubbers located in the flue gas desulfurization section upstream of the lime absorbers. Also, surge coal storage silos were reduced in size. However, the major factor which lowered the estimated investment in these sections is improvements in the methods and cost bases used in cost estimating materials handling equipment, such as silos, conveyors, and associated structures and foundations. Exxon's commercial experience with materials handling equipment was quite limited when the Predevelopment Program investment estimate was prepared in late 1977, and cost estimating tools were not well developed. Experience since that date, including the Exxon Coal Liquefaction Pilot Plant now under construction, has led to improved estimating approaches. Applying these new tools shows that the cost estimates for silos and conveyors were too high in the earlier Study Design. Costs for common on-site facilities (pipe racks, utility headers, roads, sewers, lighting, etc.) are reduced based on the final plant layout.

- Steam generation and distribution has slightly increased in cost. This is due primarily to an upward revision of coal-fired boiler cost bases, also resulting from learning experience since the previous estimate was completed over a year ago. Boiler capacity is actually down 8%, due mainly to lower steam demands for lime FGDS.

- The flue gas desulfurization facilities costs are down as a result of the change from regenerative FGDS to lime scrubbing. The investment shown for FGDS is especially low because lime receipt and handling is shared with the onsites catalyst recovery system. The investment for the shared lime facilities is included under chemicals handling and storage. Even so, the cost for the latter section is lower because of the new cost estimating approaches for silos and conveyors.

- The investment for wastewater treating is up because of the increase in process wastewater rate and in facilities for reuse. As a result of more detailed study of water reuse options, the estimated average raw water makeup rate for the CCG Study Design has been reduced from 7,300 gpm to 5,600 gpm.

- The percentage add-ons for process development allowance and project contingency are down in proportion to the reductions in onsites and total plant direct and indirect costs.

Thus, overall, the estimated investment for the CCG Study Design is reduced from 1,640 M$ to 1,530 M$.

Consistent with this revised investment, the cost of SNG produced from Illinois coal in a pioneer CCG plant is now estimated to be about 6.18 \$/MBtu on a 1978 basis, as shown in Table 5.1-3. (This updates Table 4.9-2 of the

TABLE 5.1-3

CATALYTIC COAL GASIFICATION
COMMERCIAL PLANT STUDY DESIGN

COST OF SNG FROM PIONEER PLANT WITH 100% EQUITY FINANCING

Basis:
- January, 1978 Instant Plant, Eastern Illinois Location
- 257 Billion Btu/Stream Day SNG (HHV Basis)
- 90% Capacity Factor
- 100% Equity Financing
- 15% Current Dollar DCF Return
- Escalation Rates:
  - Operating Costs and By-Product Revenues at 5%/Year
  - SNG Revenues at 6%/Year
- Total Erected Cost of 1,530 M$ (From Table 5.1-1)

SNG Cost Components	Requirements (At Full Capacity)	Unit Costs (1978)	SNG Cost Breakdown $/Million Btu (1978)
● Illinois No. 6 Coal (Cleaned)			
- To Gasifiers	14,490 ST/SD (2)	20 $/ST	1.128
- To Coal Dryer Fuel	710 ST/SD	20 $/ST	0.055
- To Offsite Boiler Fuel	2,960 ST/SD	20 $/ST	0.230
Subtotal	18,160 ST/SD		1.413
● Major Chemicals			
- KOH Solution (30 wt%)	189 ST/SD (Contained)	300 $/ST	0.221
- Lime (97% CaO) to Catalyst Recovery	1,005 ST/SD	39 $/ST	0.153
- Lime (97% CaO) to FGDS	272 ST/SD	39 $/ST	0.041
Subtotal			0.415
● Other Operating Costs			
- Purchased Electric Power	147 MW	2.5 ¢/kWh	0.343
- Raw Water	5,600 gpm	15 ¢/k gal	0.005
- Other Catalysts and Chemicals	Many Items	4.7 M$/yr	0.056
- Wages and Benefits	980 Men	21 k$/man/yr	0.244
- Salaries and Benefits	260 Men	25 k$/man/yr	0.077
- Labor Overheads and Supplies	20% of Wages, Salaries, and Benefits		0.064
- Materials and Overheads	3.3% of Total Erected Cost/Year		0.598
- Ash Disposal	8,400 ST/SD (Wet)	1 $/ST	0.033
Subtotal			1.420
● By-Product Revenues			
- Ammonia (20 wt%)	231 ST/SD (Contained)	160 $/ST	(0.144)
- Sulfur	324 LT/SD (2)	25 $/LT	(0.031)
Subtotal			(0.175)
● Capital Charges	Per Above Basis		3.104
TOTAL SUBSTITUTE NATURAL GAS COST (RISP) (3)			6.177
CALL			6.18

Notes:

(1) $k = 10^3$, $M = 10^6$, $G = 10^9$.

(2) ST/SD = short tons/stream day (i.e., one day's operation at full plant capacity). LT = long tons.

(3) Required initial selling price in first year of plant operation (1978).

Predevelopment Report.) This gas cost is a required initial selling price based on 100% equity financing with a 15% current dollar DCF return. It was assumed that SNG product revenues will escalate at 6% per year and that operating costs and by-product revenues will escalate at 5% per year. On a financing basis of 70% debt/30% equity with 9% interest on debt, the initial gas cost is 4.65 $/MBtu. This cost is also based on the same DCF return on the equity and the same escalation assumptions. The complete economic basis for these gas costs is documented in the Predevelopment Report.

The revised SNG cost in the 100% equity case is 0.24 $/MBtu less than the gas cost calculated during the Predevelopment Program. The changes in the SNG cost can be summarized as follows:

	SNG Cost, $/MBtu		
SNG Cost Component	Predevelopment Study Design	Revised Study Design	Net Change
Coal	1.40	1.41	0.01
Major Chemicals	0.37	0.41	0.04
Other Operating Costs			
- Utilities	0.35	0.35	-
- Labor and Related	0.40	0.39	(0.01)
- Materials and Overheads	0.64	0.60	(0.04)
- Other	0.10	0.09	(0.01)
By-Product Revenues	(0.19)	(0.18)	0.01
Capital Charges	3.35	3.11	(0.24)
Total	6.42	6.23	(0.19)

Lower capital charges associated with the drop in investment are the main factor contributing to the reduction in gas cost. This is partially offset by the added cost of purchasing lime (included under "major chemicals") for the lime scrubbing process now used for flue gas desulfurization.

Despite the 8% reduction in offsite boiler capacity mentioned earlier, the coal to boiler fuel is up about 3% in the revised Study Design. This is the reason for the small increase in coal cost shown above. The increase in boiler fuel is a reflection of a change in the approach used to estimate average requirements for all plant utilities. As described in the Predevelopment Report, the total design capacities for CCG Study Design utilities systems included: (1) normal requirements calculated from the onsite and offsite equipment lists; (2) intermittent requirements also calculated from the equipment lists; (3) allowances for estimated increases in utilities loads as facilities definition improves during project development; and (4) an additional allowance for reserve capacity in source facilities for startup and emergency needs. (Source facilities include offsite boilers, BFW treating, cooling tower, etc.) This approach is consistent with Exxon practices for commercial projects; the allowances for items (3) and (4) are based on Exxon's experience for a broad range of commercial process plants. For the Predevelopment Program Study Design, average plant utilities requirements for operating costs were based on the calculated normal requirements plus the average intermittent requirements. For the revised CCG Study Design, the allowances for estimated increases in utilities loads during project development (item (3)) were also included in the average utilities requirements for operating costs. This is consistent with the experience showing that such increases do occur, on average, in actual projects. Adding these allowances in the revised Study Design has increased operating costs only for coal fuel purchased to generate steam in the offsite boilers. Utilities savings resulting from the use of lime FGDS, more complete utilization of available steam in noncondensing steam turbine drivers, and increased reuse of wastewaters have offset these additional allowances for the other utilities. Thus there has been no net change in the electric power requirements (147 MW) and a substantial reduction in the raw water makeup rate (as noted earlier).

As discussed in the Predevelopment Report cited earlier, estimates of coal gasification costs can vary widely depending on the philosophy used to set the process and offsites bases, the detail of the equipment design, and the approach to the investment estimate. In addition, the method of financing, plant size, coal type, and the maturity of the technology can have significant impacts on SNG costs. The time frame for which costs are presented is also an important factor. Thus, caution must be used when comparing these economics with published estimates for other coal gasification processes. A consistent comparison of CCG with state-of-the-art gasification technology has been made by Exxon Research and Engineering Company, and it has been concluded that significant incentive exists for development of the catalytic coal gasification process.

Coal Devolatilization Impact Study

The objective of this study was to investigate the impact of uncertainties in the amount of carbon devolatilized on gasifier volume requirements and to investigate the potential risks and benefits of alternative coal feed injection points along the height of the gasifier bed.

When feed coal is injected into the fluidized bed catalytic gasifier, it is rapidly heated and devolatilized into gas phase species (such as CO, CO_2, H_2, CH_4, etc.) and hydrocarbon liquids. It is important to know the amount of feed coal devolatilized in the gasifier since this affects the amount of carbon to be gasified and influences the kinetics of the gasification reactions. The carbon rich char remaining after devolatilization must be gasified. Sufficient residence time must be provided in the gasifier to convert the remaining carbon to gaseous products. Thus it is important to know what fraction of carbon is devolatilized versus what fraction must be gasified. The second important impact of devolatilization is the inhibiting effect of the devolatilization products on the reaction rate for gasifying the remaining carbon. Thus it is important to know the amount of feed coal devolatilized and the composition of the devolatilization products.

Another important consideration with respect to devolatilization is the location of the point where feed coal is injected into the fluid bed. If the coal is injected near the bottom of the bed, reaction of the devolatilization products as they flow through the bed results in essentially no hydrocarbons heavier than methane in the gasifier effluent. This permits the recovery of high level heat from the gasifier overhead since fouling of heat exchangers from heavy hydrocarbons should not occur. However, it also results in the largest inhibition effect of devolatilization products on gasification rate since these products are present over almost the entire length of the bed. If the feed coal is injected near the top of the bed, high direct methane yields from devolatilization may increase the product gas methane content above equilibrium levels. The increase would be due to insufficient residence time to reform the devolatilized methane back to equilibrium levels. The higher direct methane yield would result in lower recycle gas rates. However, there would be the risk of heavy hydrocarbons in the gasifier overhead with resultant fouling of heat exchange surfaces because of insufficient residence time to convert them to light gaseous products. In the CCG Study Design, the feed coal was injected into the bottom of the fluid bed to assure the absence of heavy hydrocarbons in the gasifier effluent.

The Coal Devolatilization Impact Study was carried out in two parts. First, the impacts of changing the amount and composition of devolatilization products and the feed point location were evaluated in a series of nine cases. These cases included three different devolatilization yields:

- Base Case - 17% Carbon Devolatilized (CCG Study Design)
- Intermediate Case - 28% Carbon Devolatilized
- Maximum Case - 36% Carbon Devolatilized

Data on the devolatilization yield for catalyzed Illinois coal at 1300°F and 500 psia in a gasification atmosphere is limited. The range of yields assumed for this study probably brackets that which would actually be obtained in a catalytic gasifier.

Three different feed points were evaluated for each devolatilization yield:

- Bottom -- 2 feet above the bottom of the bed (CCG Study Design Base Case)
- Middle -- 25-30 feet below surface of fluid bed (FBG experience)
- Top -- 5-10 feet below surface of fluid bed (high feed point incentive case)

Each of these cases was simulated using the CCG reactor model and the same gasifier coal feed as in the CCG Study Design. The model was used to determine gasifier volume and the approach to methanation equilibrium. The results of these nine cases are summarized in Table 5.1-4.

As shown in the table, with the coal feed point near the bottom of the gasifier, the predicted gasifier volume for 90% carbon conversion is reduced by 10-18% with the higher coal devolatilization yields. The approach to methanation equilibrium is unaffected for these cases as shown below:

Devolatilization Model	Relative Gasifier Volume	Approach to Methanation Equilibrium, °F
Base	100	4
Intermediate	89	4
Maximum	82	4

TABLE 5.1-4

CATALYTIC COAL GASIFICATION
COAL DEVOLATILIZATION IMPACT STUDY

CCG Gasifier Reactor Model Simulation Results

Devolatilization Yields	Coal Feed Point	Height of Bed Above Feed, Ft.	Relative Gasifier Volume	Approach to (1) Methanation Equilibrium, °F
Base Case - 17% C Devol.	Bottom	86	100	4
"	Middle	28	88	5
"	Top	8	88	11
Intermediate Case - 28% C Devol.	Bottom	77	89 (100)	4
"	Middle	28	73 (82)	3
"	Top	9	67 (75)	-3
Maximum Case - 36% C Devol.	Bottom	70	82 (100)	4
"	Middle	28	63 (77)	0
"	Top	7	54 (66)	-31

Note:

(1) A positive value indicates a deficiency of CH_4 relative to equilibrium, while a negative value indicates an excess of CH_4 relative to equilibrium.

Also shown in Table 5.1-4 is the effect of changes in the location of the coal feed point. With the base case devolatilization model, predicted gasifier volume can be reduced by 10-15% by moving the coal feed point up from the bottom of the gasifier bed as shown below:

Coal Feed Point	Relative Gasifier Volume	Approach to Methanation Equilibrium, °F
Bottom	100	4
Middle	88	5
Top	85	11

Feeding the gasifier in the middle of the bed can save about 12% of the gasifier volume with little effect on the approach to methanation equilibrium. The higher feed location showed little savings in volume over the middle feed case while showing a poorer approach to methanation equilibrium. The higher feed location also has a greater risk of tar production as discussed below. Operation of the FBG during the CCG Predevelopment Program with the feed 28 feet below the surface of the bed showed no tars or overhead fouling.

Additionally, it appears from the information in Table 5.1-4 that the methane content of the gasifier overhead cannot be increased above equilibrium for a system which avoids tar breakthrough in the gasifier effluent. The only case showing significant methane concentrations above equilibrium was that with very high devolatilization yield and a feed point location very near the top of the bed. It is unlikely that the devolatilization yield will be this high and it is also unlikely that the coal can be reliably fed 5-10 feet below the top of the bed without fouling of the overhead heat recovery system. For the other eight cases presented in Table 5.1-4, the methane content of the gasifier overhead is near the equilibrium amount.

In the second part of this study, the potential risks and benefits of different coal feed points were investigated. As stated previously, the potential benefit of a higher coal feed point is a gasifier volume reduction of 10-15%. The risk of heavy hydrocarbon breakthrough was quantified by running heat and material balances for a case without a high level heat recovery system. For this case, the gasifier effluent was quenched with water prior to low level heat recovery. For this case, the amount of SNG fired in the preheat furnace doubles, and the offsite boilers increase in size by 30%. This reflects the absence of the gas-gas exchangers and high pressure waste boilers. These effects decrease plant thermal efficiency by about 10%. This illustrates the significant risk of tar in the gasifier overhead.

The conclusions drawn from this study are summarized below:

- The amount of feed coal devolatilization has a significant impact on gasifier volume requirements. The actual level of devolatilization for a potassium catalyzed feed coal at 1300°F and 500 psia in a gasification atmosphere is uncertain. Since the kinetic model used for the CCG Study Design was conservative (low yields), better data could reduce gasifier volume requirements.

- Fouling the high level heat recovery system by tar breakthrough would result in a serious economic debit to the process and must be avoided.

- Moving the coal feed point higher up in the fluidized bed could reduce the gasifier volume by 10-15%. Tests in the PDU must be run to confirm this savings and demonstrate that no tar breakthrough occurs by raising the coal feed point.

- It appears unlikely that the methane content of the gasifier overhead can be increased above equilibrium without the risk of tar breakthrough and poor approaches to methanation equilibrium.

Cryogenic Acid Gas Removal Incentive Study

As discussed in previous technical progress reports, a study is underway to evaluate the economic incentives associated with using a cryogenic fractionation scheme for acid gas removal in the Catalytic Coal Gasification process. In this study, CO_2 and H_2S in the cooled gasifier effluent are removed using cryogenic distillation instead of physical absorption as specified in the CCG Commercial Plant Study Design developed in 1977 under the CCG Predevelopment Program. It is believed that a cryogenic acid gas removal process may have lower investment and lower energy requirements than a physical absorption system. It is also believed that there may be benefits from the integration of cryogenic acid gas removal with the cryogenic methane recovery section of the CCG process.

The objective of this study is to identify the economic incentive and data needs for further research on this concept. This work includes the definition of the process flow scheme, detailed heat and material balances, design of the required equipment, and the development of investment and operating costs for this plant section. Process economics will be developed and compared with those of the conventional acid gas removal process used in the CCG Study Design to identify the economic incentive for cryogenic acid gas removal.

Previous work done under the CCG Predevelopment Contract led to the conclusion that carbon dioxide (CO_2) freeze-out would occur in some part of the acid gas fractionation system over the entire range of possible tower operating conditions. For this study, it has been assumed that the freeze-out problem can be handled in a simple manner within the system. Further work to determine the actual effect of CO_2 freeze-out would be undertaken if the economics for this alternative acid gas removal scheme were favorable.

The proposed cryogenic acid gas removal flow scheme incorporates two distillation towers. In the first, the Acid Gas Fractionator (AGF), CO_2 and H_2S are separated from an overhead H_2, CO and CH_4 stream. The overhead stream is then fed to cryogenic methane recovery. The bottoms CO_2 and H_2S stream from the Acid Gas Fractionator is fed to the second tower, the Acid Gas Splitter (AGS), where the overhead is essentially pure CO_2 and the bottoms is a 80/20 mixture of CO_2/H_2S. This bottoms stream is then sent to sulfur recovery.

As discussed in previous reports, various process conditions, flow schemes, and heat integration/refrigeration options were investigated. The alternatives were compared on the basis of minimizing total system horsepower requirements. This is believed to be the major investment and operating cost parameter in cryogenic systems.

During this quarter, work was completed on the heat integration/refrigeration scheme for the process. The final scheme, as shown in Figure 5.1-1, consists of the following:

- An external three-level cascade refrigeration system utilizing methane, ethylene and propylene refrigerants provides both the Acid Gas Fractionator condenser duty (-172°F process temperature) and a portion of the feed cooling duty.
- A single heat pump loop of propylene refrigerant accomplishes both the condenser and reboiler duty for the Acid Gas Splitter.
- The overhead stream from the Methane Recovery Tower (MRT) is used to subcool the methane bottoms product. This bottoms stream is then adiabatically expanded to provide the refrigeration requirements for the condenser (-240°F process temperature) and feed cooling of the MRT.
- The remainder of the Acid Gas Fractionator feed cooling is accomplished by feed/effluent heat exchange with the methane product, recycle gas, and CO_2 vent gas.

Work was also completed on the specification of equipment for the Methane Recovery, Refrigeration and Acid Gas Splitting Sections. Equipment specifications are currently being prepared for the Acid Gas Fractionation Section. Costs for the Methane Recovery and Refrigeration sections will be prorated from the corresponding sections of the CCG Study Design. The cost estimate for the Acid Gas Splitting section is currently being prepared, and work on the Acid Gas Fractionation section will begin as soon as equipment specifications are complete.

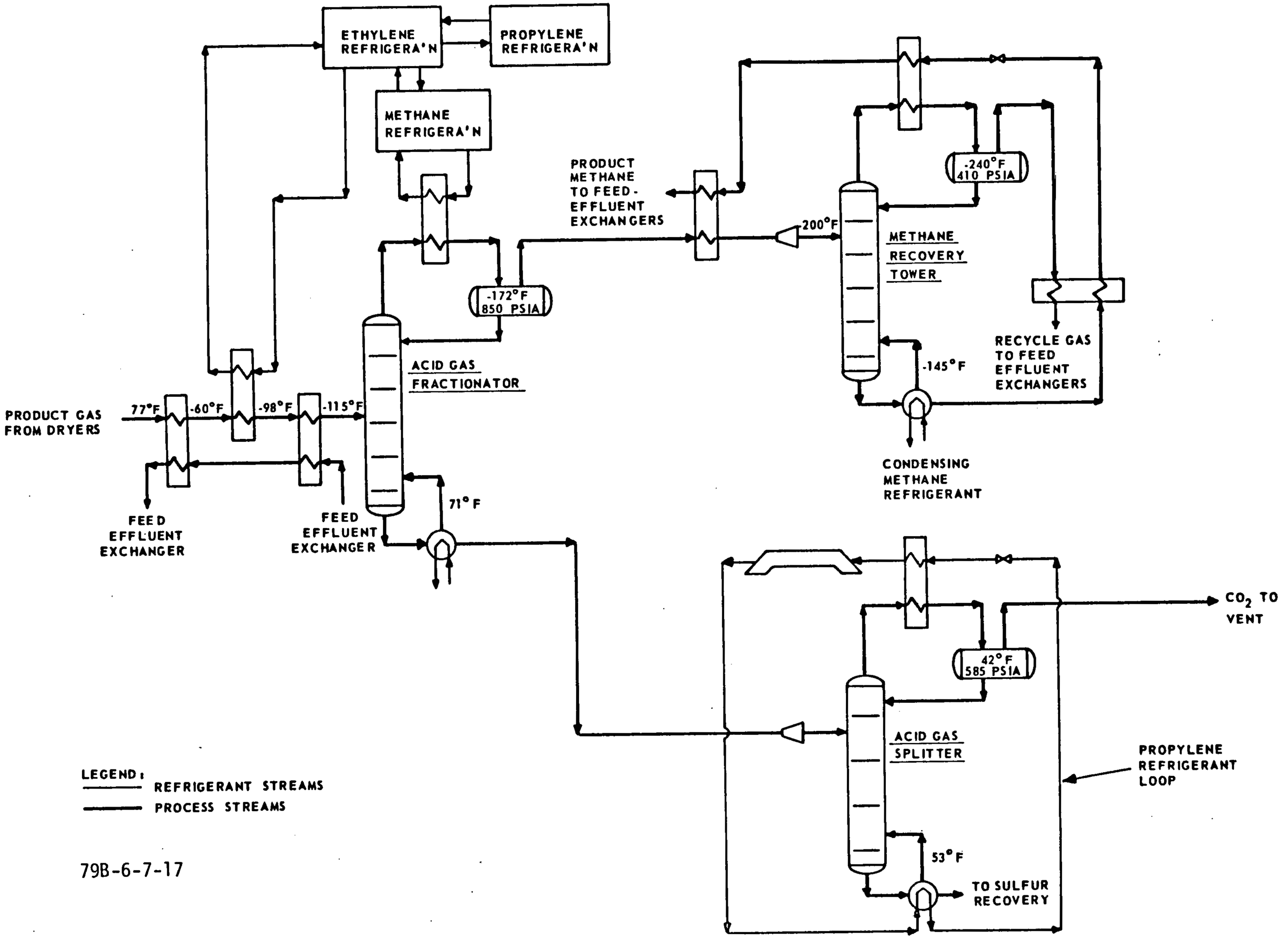
When cost estimating is completed, process economics will be developed for comparison with the base case CCG Study Design. Completion of the study is expected during the second quarter of 1979.

Evaporation of Catalyst Solutions

A laboratory guidance study has been made to estimate the economic impact of evaporating dilute catalyst solutions from catalyst recovery to concentrations which are suitable for direct addition to the gasifier feed coal. These estimates of evaporation costs will be used to help assess technical and economic tradeoffs in the catalyst recovery section. As recovered solution concentration is reduced below the level in the CCG Study Design, fewer washing stages are required to achieve the same overall recovery. Also, the solid-liquid separations are easier in dilute solutions, due to lower viscosities, and in the case of separations based on gravitational forces (e.g., settlers, centrifuges), due to larger particle-solution

FIGURE 5.1-1

CRYOGENIC ACID GAS REMOVAL REFRIGERATION AND HEAT INTEGRATION FLOW SCHEME



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density differences. The potential cost savings for dilute solutions must be weighed against the added costs to concentrate the recovered solution to the same level used in the Study Design.

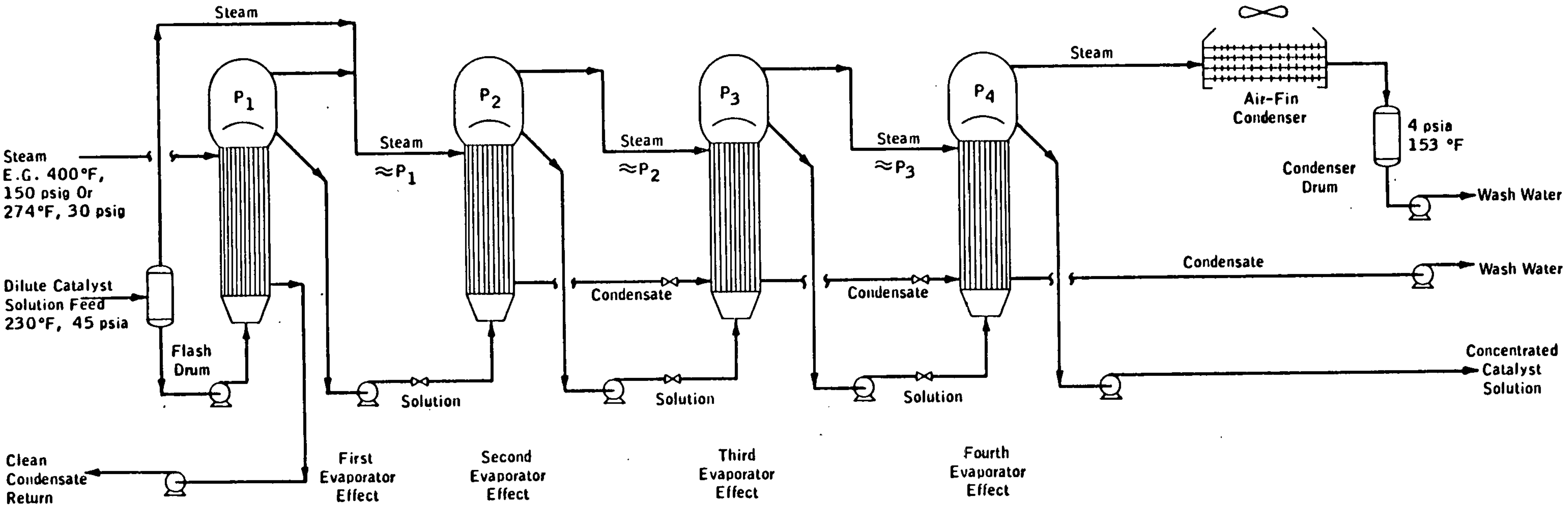
In order to estimate the costs for evaporation, a series of screening studies were carried out. Figure 5.1-2 shows the general process flowsheet for these screening studies. It includes a conventional, multiple-effect evaporator for concentrating the catalyst solution and an air-fin condenser for recovering the evaporated water for recycle to catalyst recovery. The multiple-effect evaporator uses process steam in the first effect to concentrate the catalyst solution. Vapor raised in the first effect is condensed in the second effect to further concentrate the remaining solution. The vapor from the second effect is then condensed in the third effect, and so on. The vapor from the last effect is condensed in the air-fin condenser. To operate the evaporator in this manner, the solution pressure in each effect is maintained lower than the pressure in the preceding effect. The pressure in the last effect was set at 4.5 psia. This pressure is typical of multiple-effect evaporators and was selected because it gave the lowest combined evaporator-condenser area for representative cases.

The process basis for the current studies was set based on the CCG Study Design. The catalyst feed rate to the evaporator is the same as the catalyst rate from catalyst recovery in the Study Design (equivalent to 122.8 klb/hr of KOH). Two catalyst solution concentrations, 5 and 10% (wt), were considered as feeds to the evaporator system. The concentrated product from the evaporator is a 32.2% (wt) KOH catalyst solution, which is the same concentration as the recovered catalyst solution fed directly to the catalyst addition/entrained drying system in the Study Design. Steam to concentrate the solution in the evaporator is potentially available from two sources. Low pressure steam (e.g., 10-30 psig) can be produced from onsite waste heat, and higher pressure steam (e.g., 150 psig) can be produced by letting down high pressure steam from offsite boilers across non-condensing steam turbine drivers.

To estimate the economic impact of concentrating the dilute catalyst solutions, heat and material balances were made for each catalyst solution feed (5 and 10% (wt) KOH) with each steam source and with a variable number of effects in the evaporator. Based on these balances, both onsite and offsite equipment was sized and utility demands were determined. The number of parallel evaporation trains was set to maintain individual evaporator area below 32,000 ft^2 (the approximate maximum commercial size today). The incremental investment and operating costs were estimated based on comparable equipment and operating costs for the CCG Study Design. The incremental impact on the gas cost was then estimated using the CCG Study Design economic basis (100% equity financing, 15% DCF return on investment, January 1978 cost level, East Illinois location). By minimizing these incremental gas costs, the approximate optimum number of effects for each feed concentration at each steam pressure were selected. The first two lines in Table 5.1-5 summarize these optimum cases assuming no limit to the availability of 30 psig steam. The range of incremental gas costs shown in the table reflects the sensitivity of the gas cost to uncertainties in the evaporator costs and the inclusion of a 25% process development allowance.

Figure 5.1 - 2

FORWARD FEEDING MULTIPLE-EFFECT EVAPORATOR



Note: (1) Pressures Are Cascaded So That $P_1 > P_2 > P_3 > P_4 = 4.5$ psia

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TABLE 5.1 - 5

INCREMENTAL GAS COST FOR CONCENTRATING DILUTE CATALYST SOLUTIONS BY EVAPORATION

Process Basis

• Catalyst Feed:	122.8 k lb/hr KOH (dry basis)
• Feed Concentration:	5 wt% KOH or 10 wt% KOH
• Product Concentration:	32.2 wt% KOH
• Available Steam:	Offsite boiler steam at 150 psig and/or onsite waste heat steam at 10-30 psig

Economic Basis

- CCG Study Design producing 257 GBtu/SD SNG
- 100% equity financing/15% DCF return

	5% KOH Feed		10% KOH Feed	
Evaporator Steam Basis	Evaporator Effects @ Steam Pressure, Psig	Incremental Gas Cost, \$/MBtu SNG	Evaporator Effects @ Steam Pressure, Psig	Incremental Gas Cost, \$/MBtu SNG
• All Offsite Boiler Steam	5 @ 150	0.37-0.47	5 @ 150	0.17-0.22
• "Unlimited" Waste Heat Steam	2 @ 30	0.25-0.33	2 @ 30	0.11-0.14
• "Limited" Waste Heat Steam (Limit set by 1977 CCG Study Design)	4 @ 30 3 @ 10 5 @ 150	0.32-0.46	4 @ 30	0.12-0.16

A comparison of the results for the high-pressure (offsite boiler) steam and unlimited low-pressure steam cases shows that for both feed concentrations, the impact on gas cost is minimized by utilizing the onsite waste heat to raise the required low-pressure steam. However, evaluation of the Study Design heat balance indicates that there is not sufficient waste heat available to raise the low-pressure steam required to operate the evaporator at the optimum conditions. With this constraint, either more effects must be added to the evaporator to make it more thermally efficient or high-pressure steam must be used to fill the deficit. If more effects are added to the evaporator, less steam is required, but the incremental gas cost will increase due to higher investment charges. If only a few effects are added, the incremental gas cost increases above the optimum, but is still less than that for all high-pressure steam. The last line in Table 5.1-5 summarized the rough optimum cases using the low-pressure steam estimated to be available based on the CCG Study Design heat balance.

In the case of 5% KOH feed with limited steam, all available 30 psig steam is used in a four-effect evaporator to concentrate about 40% of the total feed. Additionally, the low-level waste heat which remains after raising the 30 psig steam is used to raise 10 psig steam. The 10 psig steam is used in a three-effect evaporator to concentrate about 30% of the feed. The remaining feed (30%) is concentrated in a five-effect evaporator with high-pressure steam. (The incremental gas cost of using all 10 psig steam is greater than the incremental gas cost of using a combination of 10 psig and 30 psig steam.) In the case of 10% (wt) KOH feed, the solution can be evaporated to 32.2% (wt) entirely with 30 psig steam in a four-effect evaporator.

The impacts of evaporating dilute catalyst solutions on the overall CCG process efficiency and gas cost are much less if the solution from catalyst recovery is 10% (wt) KOH rather than 5% (wt). For 10% (wt) KOH solution, no supplemental offsite steam is required, and thus the impact on process efficiency is slight. The 10% (wt) KOH case also shows a clear economic incentive over the 5% (wt) case. The incremental gas cost for concentrating the 10% (wt) solution to 32.2% (wt) is 0.12-0.19 \$/MBtu, only 2-3% of the CCG Study Design gas cost of 6.18 \$/MBtu. However, evaporating more dilute solutions could have a slightly greater cost impact. The incremental gas cost for concentrating the 5% (wt) solution is 0.32-0.46 \$/MBtu.

Integral Steam Reformer Heat Input Study

A key feature of the Catalytic Coal Gasification process is the recycle of CO and H_2 to the gasifier. This forces the net products of gasification to be only CH_4 and CO_2 along with smaller amounts of H_2S and NH_3. Using this approach, the overall chemistry can be represented as follows:

$$\text{Coal} + H_2O \rightarrow CH_4 + CO_2 \qquad \Delta H = 0$$

Thus, coal is converted to methane in a single reaction step which is approximately thermally neutral. A small amount of heat input is required to preheat the feed coal, recycle gas, and steam to reaction temperature, to account for catalyst reactions, and to provide for gasifier heat losses.

In the 1977 CCG Study Design, this heat input was supplied by heating the steam and recycle gas in a furnace to 1540°F. This preheat is sufficient to provide for the heat input requirements listed above. The preheat furnace design temperature was set at 1575°F to allow for operating flexibility and control. A schematic flow plan for this system is shown in Figure 5.1-3.

In previous Exxon funded work, the concept of using a steam reformer for heat input was identified. In this concept, a small amount of methane is reformed to make additional CO and H_2 for feed to the gasifier. This CO and H_2 forms methane in the gasifier, thus providing both chemical and sensible heat input. The use of a reformer provides greater flexibility than the base case heat input scheme which uses only sensible heat for heat input. The reformer could be either a small reformer operating in parallel with the preheat furnace, or the reformer could replace the preheat furnace by reforming methane already present in the recycle gas. This last alternative, called an Integral Steam Reformer, was shown by previous rough screening studies to be lower in cost than a parallel reformer but was an economic standoff with the base case utilizing a preheat furnace.

A study was initiated during February to consider the Integral Steam Reformer in greater depth using the CCG Study Design basis. A schematic flow plan for this system is also shown in Figure 5.1-3. Work is underway to evaluate several alternative processing conditions including a range of steam reformer coil outlet temperatures and steam conversions. The CCG reactor system material and energy balance model has been modified to incorporate the steam reforming process option and initial simulations have been carried out. Initial study results for reformer coil outlet temperature and steam conversion are summarized below.

- Reformer Coil Outlet Temperature - Steam reformer coil outlet temperatures (COT) from 1400°F to 1500°F have been evaluated at the base case coil outlet pressure of 520 psia. A comparison of the cases is shown below:

Basis: 14,490 ST/SD Coal feed to gasifier
Gasifier operating conditions of 1275°F, 500 psia.

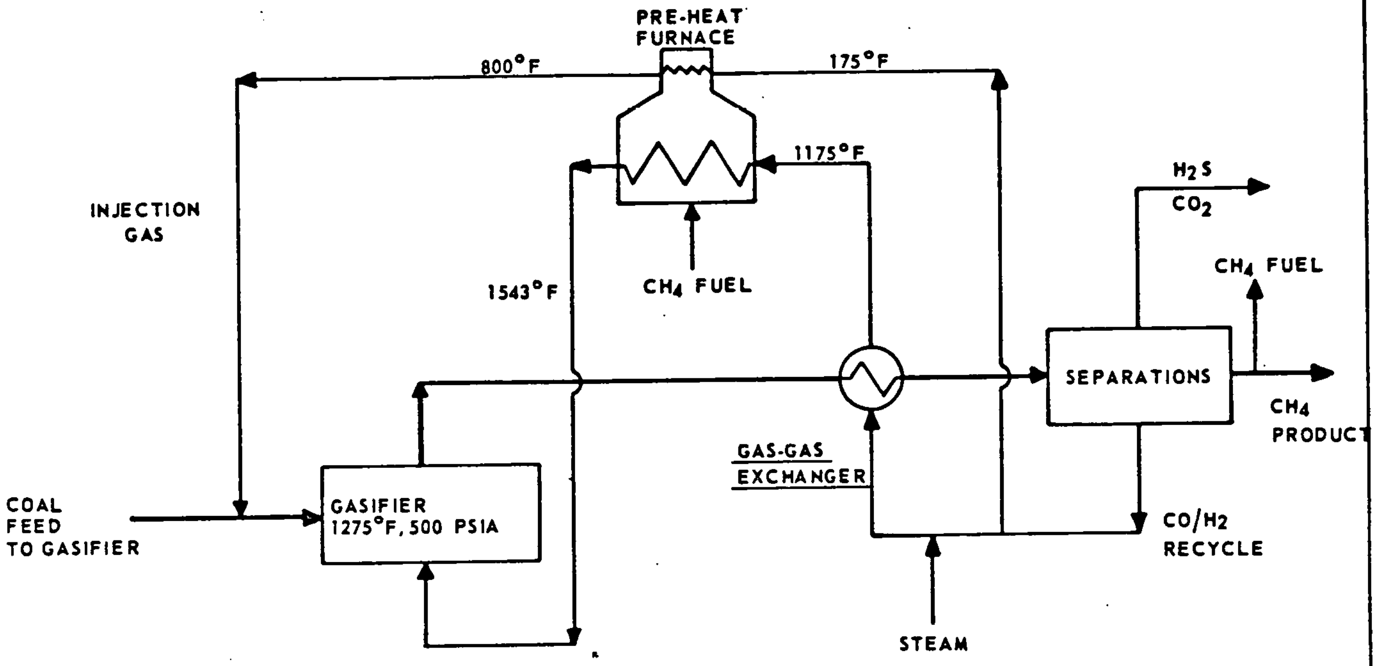
Coil Outlet Temperature	1400°F	1500°F	% Change
Recycle Gas Rate, lb moles/hr	66,300	52,800	-20.0
Raw Gasifier Product Rate, lb moles/hr	181,600	152,800	-16.0
Acid Gas Removal Feed, lb moles/hr	127,000	105,800	-17.0
Overall Steam Conversion, %	39	42	+8.0
Offsite Steam Required, lb moles/hr	64,400	55,700	-14.0
Relative Gasifier Volume	100	95.4	-5.0
Reformer Furnace Duty, $\frac{\text{MBtu}}{\text{Hr}}$	680	630	-7.0
Net Methane Product, $\frac{\text{GBtu}}{\text{SD}}$	252.1	254.8	+1.0

The credits for higher temperature include reduced gas flow rates, reduced steam requirements, reduced furnace duty, etc. The debit for the higher temperature will be a higher furnace investment. It is believed that the credits of higher reformer outlet temperature offset the debits.

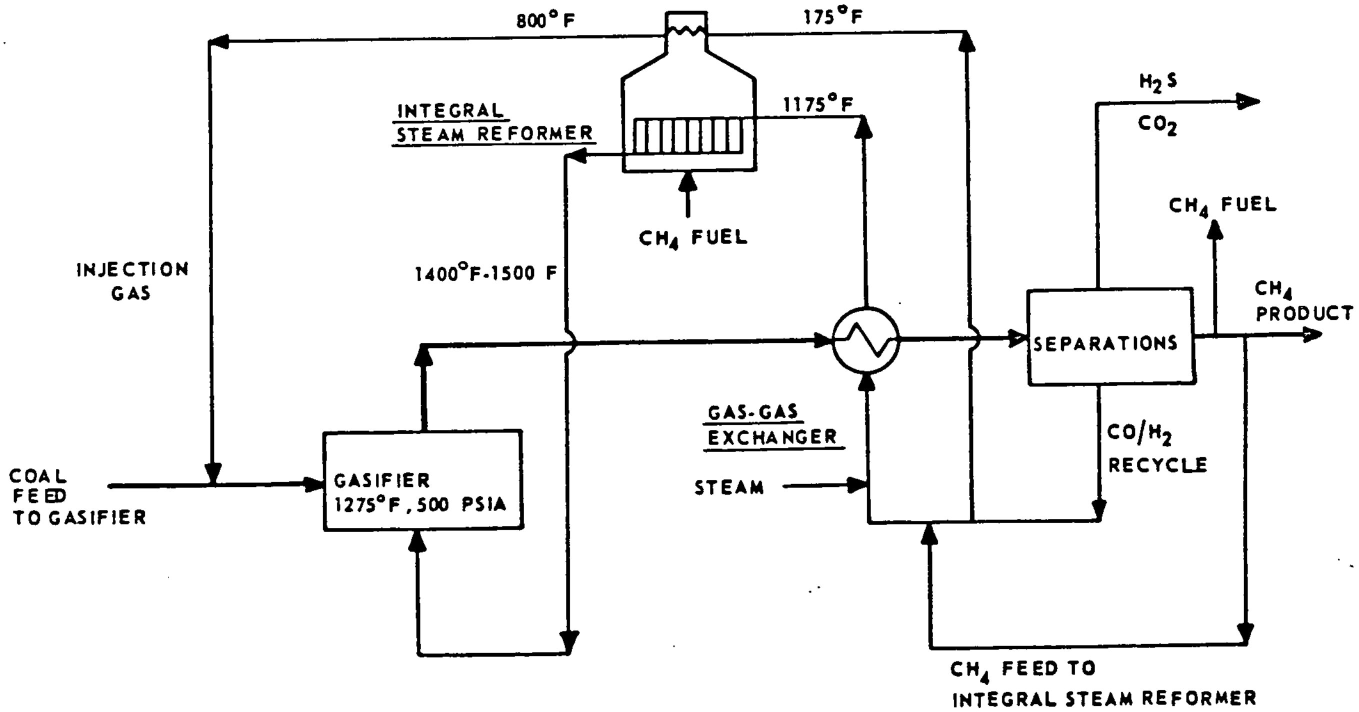
FIGURE 5.1-3

INTEGRAL STEAM REFORMING HEAT INPUT STUDIES - SIMPLIFIED SCHEMATIC FLOW PLAN

CCG STUDY DESIGN BASE CASE



INTEGRAL STEAM REFORMING CASE



- Steam Conversion - A range of overall steam conversions from 41 to 50% was evaluated. These results are shown below:

  Basis: 14,490 ST/SD Coal feed to gasifier,
  Gasifier operating conditions of 1275°F, 500 psia, and
  steam reformer coil outlet temperature of 1450°F.

Overall Steam Conversion, %	41	47	50
Recycle Gas Rate, lb moles/hr	59,200	53,700	51,300
Gasifier Product Rate, lb moles/hr	163,200	148,000	141,400
Acid Gas Removal Feed, lb moles/hr	113,500	109,600	108,100
Offsite Steam Required, lb moles/hr	58,600	48,400	44,000
Reformer Furnace Duty, MBtu/hr	650	630	620
Relative Gasifier Volume	100	130	160
Net Methane Product, GBtu/SD	253.7	254.0	254.1

The credits for higher steam conversion include reduced gas flow rates, reduced steam requirements, reduced furnace duty, etc. The debit for the higher steam conversion will be higher gasifier investment. It is believed that the 47% steam conversion case represents the optimum balance.

Other process options for the Integral Steam Reforming Study are currently under evaluation. The gasifier product gas stream downstream of H_2S removal (containing CH_4, CO, H_2 and CO_2) is being evaluated as an alternative steam reformer fuel in place of methane used in the base case. Also, this same stream is being considered as supplemental feed to the reformer for gasifier heat input control in place of methane product. These options may offer investment and operating cost savings by reducing the feed rate to the CO_2 removal and cryogenic methane separation sections of the CCG process and by increasing the nitrogen purge rate from the recycle gas loop. Following selection of the preferred Integral Steam Reformer process conditions, equipment design, investments, and economics will be developed for comparison to the CCG Base Case.

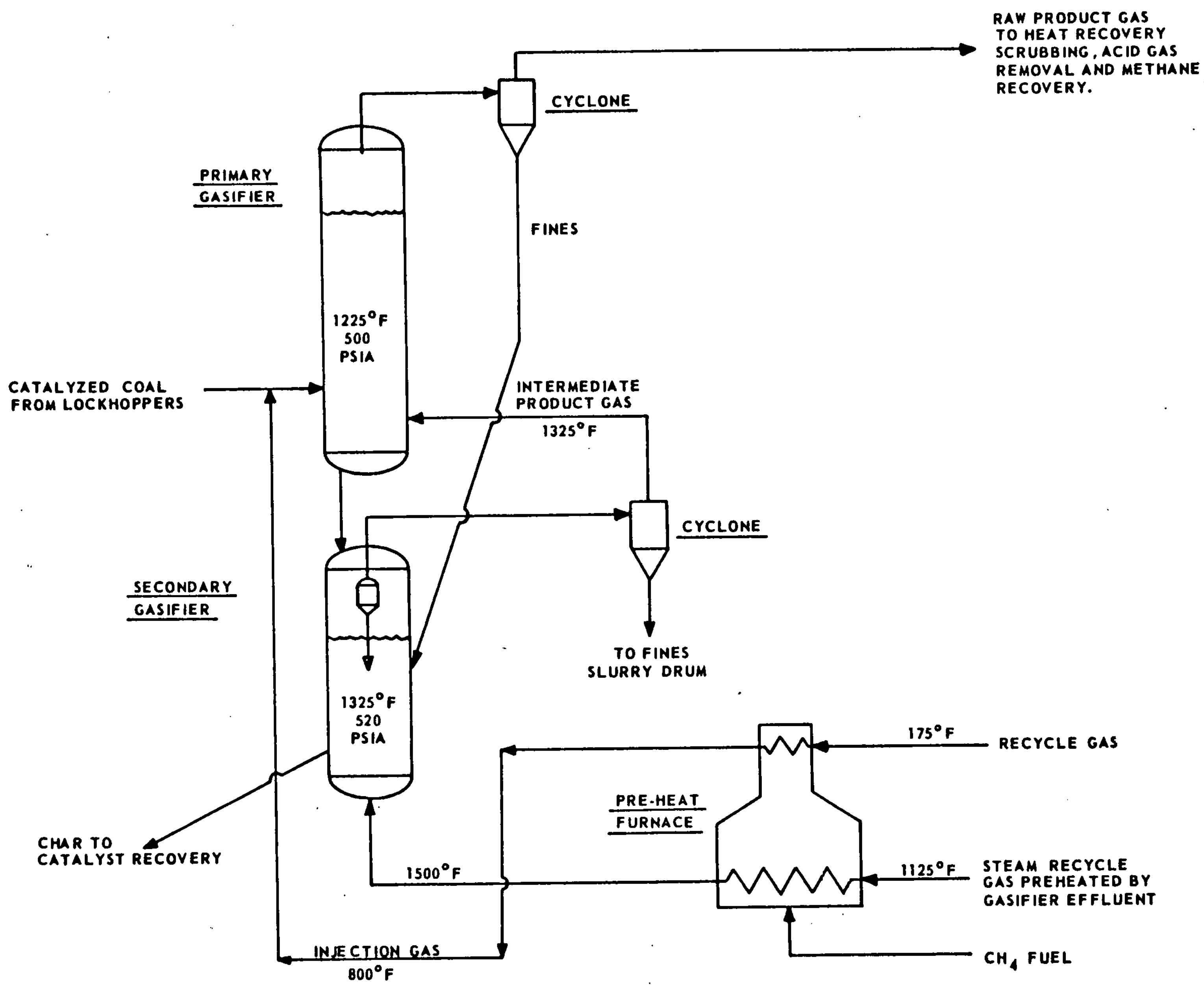
Two Stage Gasifier Incentive Study

In the Catalytic Coal Gasification Study Design, a simple fluidized bed gasifier with one gasification stage was used to achieve a target carbon conversion of 90%. A previous study done during the Predevelopment Phase of research investigated the use of a second gasification stage to increase overall carbon conversion to 95%. In this study, fines and char withdrawn from the first gasification stage were fed to the second gasification stage. The primary gasifier was operated in parallel at the same temperature. Steam and recycle gas from the preheat furnace were fed in parallel to each gasification stage. This process configuration showed only a small gas cost savings of about 0.6% relative to the single stage base case.

A brief incentive study of an alterative two-stage gasification concept was completed during the first quarter. The two-stage gasifier process configuration selected for this study is illustrated in Figure 5.1-4. In this scheme, coal is fed to the first stage gasifier which operates at low

FIGURE 5.1-4

SIMPLIFIED FLOW PLAN FOR TWO STAGE GASIFICATION



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temperature (1225°F). The coal is fluidized and gasified by product gas from the second stage gasifier. A carbon conversion of 80% is achieved in this first stage. The char and fines from the first stage are withdrawn and fed to the second-stage gasifier. This operates at a higher temperature (1325°F) to achieve high carbon conversions. Steam and recycle gas from the preheat furnace are fed to the secondary gasifier to achieve an overall carbon conversion of 95% for the two gasifier stages.

This concept differs from that evaluated in the predevelopment research phase in that the two gasification stages are operated in series with respect to steam and recycle gas flow. This permits operating the gasifiers at different temperatures. Reduced recycle gas rates are achieved by operating the upper stage at a lower temperature (1225°F) and high carbon conversions are obtained by operating the bottom stage at a higher temperature (1325°F).

A summary of the process basis and heat and material balance is provided in Table 5.1-6. The two-stage gasifier case was evaluated on the basis of the same coal feed rate to gasification as the CCG Study Design. Total gasifier steam required increased by 10% while the recycle gas rate decreased by 12%. Due to the lower temperature in the first-stage reactor, the preheat furnace coil outlet temperature decreased from 1543 to 1500°F. The net SNG product rate increased to 271.3 GBtu/SD (up 5.6%) while the overall plant efficiency increased by 3.1%.

Rough screening economics were developed for this two-stage gasification scheme. As shown in Table 5.1-7, total investments are up by 5.1% over the base case. This is a slightly smaller percentage increase than the increase in plant SNG output (5.6% increase). The most significant investment increase is associated with a larger first stage gasifier volume required for the lower reactor temperature (1225°F) than the base case and for the addition of the separate second stage gasifier. Also, steam generation investments are increased due to the increased steam requirements for this case.

Process economics are presented in Table 5.1-8. The total gas cost with two-stage gasification is 2.3% less than the Study Design cost. Savings are achieved in coal, catalyst, and operating costs. Thus, based on these results, there appears to be an incentive for staged gasification. However, additional research and supporting engineering studies would be required to develop a better estimate of the incentive for two-stage gasification. Additional data are required to firm up reaction kinetics at the lower gasifier temperature of 1225°F and at carbon conversions over 90%. The current data base at these conditions is limited since the Fluid Bed Gasifier (FBG) runs made during the predevelopment research phase were generally at temperatures of 1300°F and carbon conversions of 80-90%. Additional data are also required to allow better prediction of the rate of fines entrained from the primary gasifier and the ability of the two stage system to retain and gasify the fines. Data on lower gasification temperatures, higher carbon conversions and fines generation will be obtained as part of the current Process Development Program. These data can then be used for a more definitive estimate of the incentive for a two-stage gasification system.

Table 5.1-6

INCENTIVE STUDY FOR TWO-STAGE GASIFICATION

Reactor System	Base Case "Primary" Gasifier Only	Two-Stage Gasification Primary and Secondary Gasifiers
Free Carbon Conversion		
Primary Gasifier	90%	80%
Overall	90%	95%
Conditions:		
Primary Gasifiers	1275°F/500 psia	1225°F/500 psia
Secondary Gasifier	-	1325°F/520 psia
Key Stream Rates:		
Coal Feed to Gasifier, ST/SD (1)	14,490	14,490
Coal to Boilers, ST/SD	2,840	3,030
Coal to Dryer Fuel, ST/SD	710	710
Total Coal, ST/SD	18,040	18,230
Total Gasifier Steam, MPH	86,008	94,970
Total Recycle Rate, MPH	57,525	50,696
Preheat Furnace Coil Outlet Temperature, °F	1,543	1,500
Net SNG Product Rate, GBtu/SD	257.0	271.3
Utilities Requirements:		
Electric Power, MW	147	151
Raw Water, GPM	7,300	7,300
Overall Thermal Efficiency (2)	62.6	65.7

Notes:

(1) Two-stage gasification evaluated on the basis of constant coal feed rate to gasification.

(2) Thermal efficiency includes purchased electric power (evaluated at a power plant heat rate of 8,950 Btu/KWH) and by-products.

Table 5.1-7

Two Stage Gasification Incentive Study Relative Investment Breakdown

Basis: Base Case Total Investment = 100

	Base Case	Two-Stage Gasification
Onsites		
Coal Drying/Catalyst Addition	4.7	4.7
Reactor System	15.2	19.0
Product Gas Cooling/Scrubbing	6.5	6.2
Sour H_2O Stripping/NH_3 Recovery	1.5	1.6
Acid Gas Removal/Sulfur Recovery	12.0	12.0
Methane Recovery	3.3	3.3
Refrigeration	2.3	2.4
Catalyst Recovery	3.0	2.9
Common Facilities	4.8	4.9
Onsites Subtotal	53.3	57.0
Offsites		
Utilities	19.8	20.5
Materials Handling	8.9	9.0
General Offsites	7.2	7.2
Offsites Subtotal	35.9	36.7
Process Development Allowance (25% of Onsite Direct & Indirect Cost)	10.8	11.4
Total Plant TEC	100.0	105.1

Table 5.1-8

Two-Stage Gasification Incentive Study
Summary of Relative Gas Cost

Basis: Base Case Total Gas Cost = 100

Gas Cost Components	Base Case	Two Stage Gasification
Coal to Gasifiers	17.6	16.6
Coal to Dryer Fuel	0.9	0.8
Coal to Offsite Boilers	3.4	3.5
Subtotal	21.9	20.9
Major Chemicals		
KOH Solution (30 wt %)	3.4	3.2
Lime (97% CaO)	2.4	2.2
Subtotal	5.8	5.4
Other Operating Costs		
Purchased Electric Power	5.3	5.2
Raw Water	0.1	0.1
Other Catalysts & Chemicals	1.1	1.0
Wages and Benefits	4.0	3.9
Salaries and Benefits	1.3	1.2
Labor Related Operating Costs	1.0	1.0
Investment Related Op. Costs	10.0	9.8
Ash Disposal	0.4	0.4
Subtotal	23.2	22.6
By-Products Credits	(2.9)	(2.9)
Capital Charges (1)	52.0	51.7
Relative Gas Cost, %/MBtu	100.0	97.7
Gas Cost Savings, %	-	2.3

Note:

(1) Capital charges based on 100% equity financing with 15% DCF return.

5.2 Systems Modeling

Systems modeling work is being carried out as part of the CCG Process Development Program to develop material and energy balance tools which will reduce the engineering effort required to do screening studies and process definition studies. A material balance model for the catalyst recovery system was completed during the first quarter. Work is continuing on the development of a material and energy balance model for the CCG reactor system.

Catalyst Recovery Material Balance Model

Catalyst recovery as incorporated in the CCG Commercial Plant Study Design involves "digestion" of gasifier char and fines with $Ca(OH)_2$ to solubilize most of the catalytic potassium salts, followed by multi-stage countercurrent leaching with water to remove the soluble catalyst from the gasifier and calcium solids. Material balances for this system have required extensive stage-by-stage hand calculations, as well as some simplifying assumptions. The new catalyst recovery material balance model has been developed to perform rigorous stage-by-stage calculations taking into account the solid-liquid separation efficiencies for individual stages. This computer model will be used shortly in screening studies to determine preferred process configurations, operating conditions, and solid-liquid separation methods for the catalyst recovery system.

Figure 5.2-1 represents one stage in the countercurrent leaching sequence. Each stage involves mixing of solids from a richer (more concentrated) stage with solution from a leaner (less concentrated) stage, followed by solid-liquid separation to produce a richer solution stream and a leaner solids stream. The catalyst recovery model is capable of handling these four streams as well as an internal stream representing the feed to the solid/liquid separation device and a net side feed stream. The latter stream would be used to represent any special feeds or products that may be involved. One example is $Ca(OH)_2$ digestion, where calcium solids are added and water is consumed in chemical reactions. Provision is also made within the model to reflect adsorption of soluble potassium salts on the solids.

The model is capable of calculating any one of the following three parameters, with the remaining two specified by the user:

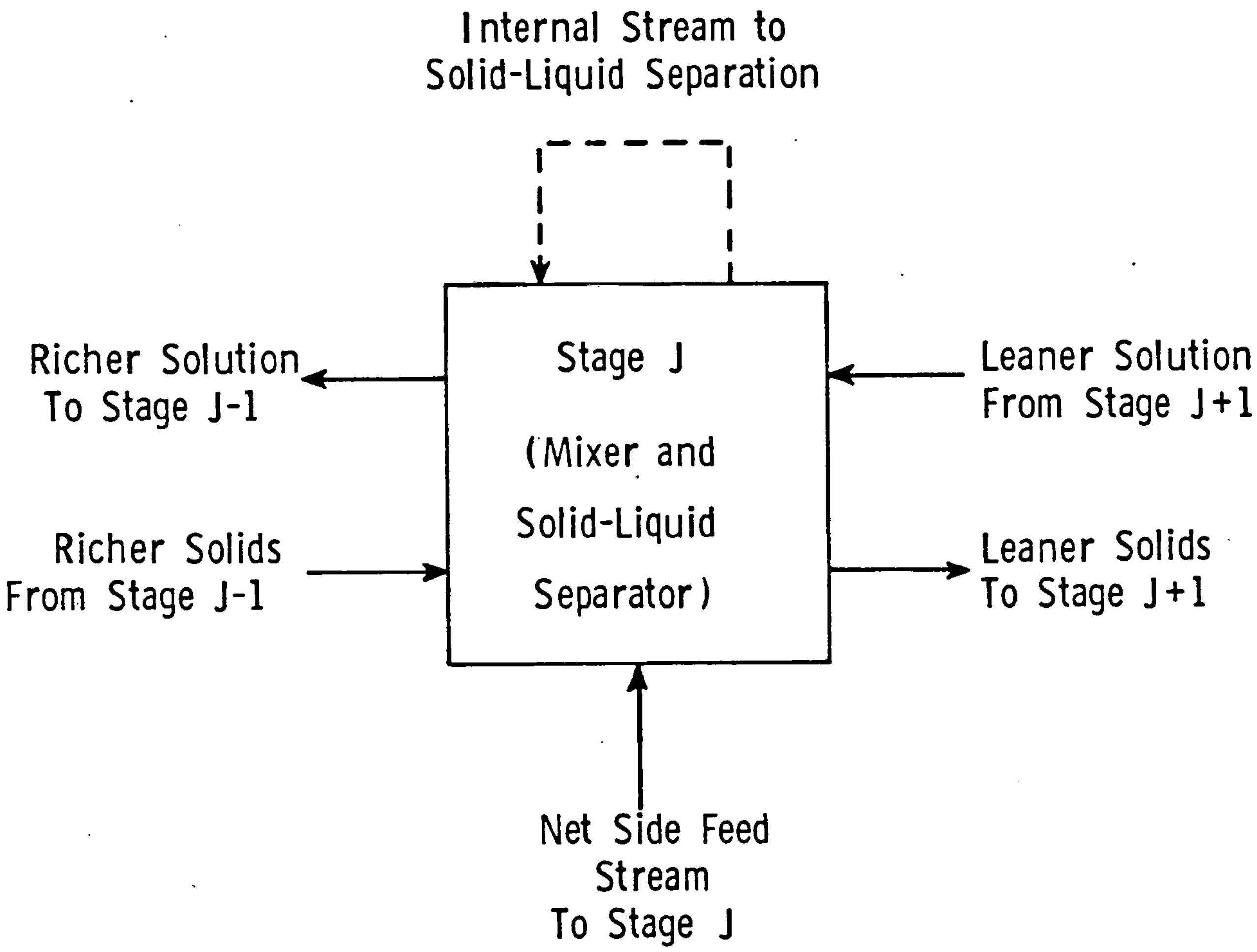
- Overall catalyst recovery
- Recovered "rich" solution concentration
- Number of washing (leaching) stages.

Varying rich solution concentration is equivalent to varying wash water rate. Other required inputs are the rich solids feed to the first (richest) stage as well as the solid-liquid separation performances and side feed streams for all stages.

The material balance routines in the model were validated by duplicating the catalyst recovery material balance for the CCG Study Design. The material balances for soluble catalyst salts, water, and insoluble solids all closed to

Figure 5.2-1

CATALYST RECOVERY MATERIAL BALANCE MODEL: STAGE CONFIGURATION AND STREAMS



within ±0.01%. Numerous test cases were run to validate various material balance convergence and output options. After minor modifications to improve the convergence methods, all cases converged satisfactorily. Computer costs per run were very low. The model is now available for use in engineering and laboratory studies of the catalyst recovery system.

CCG Reactor System Material and Energy Balance Model

Development of the updated CCG reactor system model to material and energy balance catalytic gasifiers and the associated recycle gas loop continued during the first quarter of 1979. Updated computer tools are needed to allow greater efficiency, flexibility, and consistency in carrying out the laboratory guidance studies planned under the current program.

During the period, definitive plans covering capabilities, options, input/output, and basic program logic were developed for the overall model and the individual model blocks. Three main blocks are being programmed to model the heart of the commercial process, the gasifier itself:

- The first block models the CCG gasifier solids material and energy balance. This block feeds coal and catalyst and produces "reacting coal" (the portion of the solids feed which is gasified) and spent solids (unreacted char, ash, and catalyst). Solids stream enthalpies, including the effects of catalyst-coal reactions, are also calculated.
- The second block feeds the "reacting coal" and the various gaseous feed streams and produces an effluent gas at specified shift and methanation equilibria.
- The third block carries out the overall gasifier energy balance. The model has the flexibility to energy-balance other related reactor systems, such as steam reformers.

This functional breakdown of the gasifier model into three independent blocks will facilitate modeling of flowsheets or gasifier configurations different from the base case in future lab guidance and process improvement studies.

The programming of these three model blocks was well along by the end of the quarter. The first two blocks were programmed and debugged in preparation for validation with the commercial base case (the Predevelopment Program Study Design). However, modeling of catalyst-coal reactions has been deferred until the second quarter. The third block was programmed and debugging was underway. Upon completion of this work these three blocks will be incorporated within the framework provided by "COPE", Exxon's proprietary process network simulation program. The overall COPE simulation will model the material balances for the cleanup and separations steps downstream of the gasifier, and converge the overall material balance and gasifier energy balance.

Work during the second quarter of 1979 will include programming of catalyst-coal reaction effects, validation of the overall model using the CCG Study Design and other selected cases, and documentation of the model. In addition, an optional block will be programmed to incorporate the gasifier kinetics/contacting model as updated during the CCG Predevelopment Program. This feature will allow calculation of the gasifier bed size as well as the material and energy balance.

5.3 Engineering Technology Studies

As part of the CCG Process Development Program, a coordinated set of engineering technology programs is being conducted to develop fundamental process and equipment technology to support the overall laboratory and engineering process development effort. As of March, work was underway on five of these programs, as described below. Additional programs will be initiated later in 1979.

Evaluation of Construction Materials for Catalytic Gasification

The major objective of this engineering technology program is to assemble a data base on materials performance for those plant sections which have materials considerations unique to catalytic gasification. The main focus of the program will be in-situ materials evaluation in the CCG PDU at Baytown, Texas.

A detailed summary of the proposed materials evaluation program for the PDU has been prepared. This summary describes the location, test type, and materials for each corrosion probe and rack. In addition, tentative recommendations have been made on inspection and stream sampling. Finally, a program has been outlined for failure analysis of selected components and for systematic destructive examination of critical tubing sections.

During the first quarter of 1979, all eight corrosion racks to be installed in the PDU were fabricated, assembled, and shipped to Baytown. The racks and test materials are summarized in Table 5.3-1. The two corrosion probes are also listed. Fabrication of a second set of racks and specimens will begin in the second quarter.

A baseline non-destructive testing (NDT) survey of the PDU is underway. The recorded baseline data will be compared to NDT readings obtained during PDU operation to monitor metal loss on a periodic basis.

An agreement-in-principal was obtained from DOE for materials screening tests for the CCG process at the Bureau of Mines' Tuscaloosa Metallurgy Research Center. To institute and discuss these proposed tests, a joint meeting with DOE and Tuscaloosa was held in March. Tests under discussion would involve exposure of selected metal and refractory specimens to simulated CCG process gasifier conditions. The specific purpose is to look for evidence of molten salt/slag attack, which might be caused by the presence of KOH or another potassium salt gasification catalyst. Accelerated refractory attack by alkalis has been documented by Tuscaloosa and others at high temperatures (e.g., 1800°F), but has never been explored at CCG reactor temperatures (ca. 1300°F). Definitive planning of an R&D program at Tuscaloosa will take place in the second quarter.

Vapor-Liquid Equilibria in Sour Water/Catalyst Systems

An engineering technology program is underway with the objective of developing a vapor-liquid equilibrium model applicable to the sour water

TABLE 5.3-1

CATALYTIC COAL GASIFICATION
PDU CORROSION RACKS AND PROBES

Test Site	Location	Type of Test	Coupon Type	Test Materials
1	Gasifier - dense phase	Rack	Refractory Cylinders	Med. wt. castable with and without fiber reinforcement
2	Gasifier - dense phase	Rack	Metal Cylinders	HK-40, 304 SS, 309 SS
3	Gasifier - dense phase	Rack	Metal discs	HK-40, 310 SS, 304 SS, 309 SS, 304 SS Alonized
4	Sour water accumulator	Rack	Metal cylinders	Carbon steel, 304 SS, 316 SS, Alloy 20, Monel 400, Titanium
5	Sour water accumulator	Probe	Wire element	Carbon steel
6	Sour water stripper	Rack	Metal Cylinders	Carbon steel, 304 SS, 316 SS
7	Sour water stripper	Rack	Metal cylinders	Alloy 20, Monel 400, Titanium
8	Char slurry pot	Rack	Metal U-bends	Carbon steel, 316 SS, Inconel 600, Inconel 625, Alleghany Ludlum 29-4
9	Char digester	Rack	Metal U-bends	Carbon steel, 316 SS, Monel 400, Inconel 600, Alleghany Ludlum 29-4
10	Char digester	Probe	Wire element	Carbon steel

systems in the CCG Process. The systems for which the model would be used include the gasifier product gas wet scrubbers and condensate drums and the sour water stripping facilities.

Work this quarter has concentrated on developing an experimental program to obtain vapor-liquid equilibrium data to support the design of equipment for processing sour water streams. A literature search has identified available experimental data on the volatility of ammonia, carbon dioxide, and hydrogen sulfide in aqueous solutions, including solutions containing catalytic potassium compounds. Preliminary screening of the quaternary data (ammonia-carbon dioxide-hydrogen sulfide-water) has shown that they are of poor quality above 140°F. Accurate high-temperature quaternary data must, therefore, be obtained. Additional data on the volatility of ammonia, carbon dioxide, and hydrogen sulfide in aqueous solutions containing potassium compounds are also needed.

A preliminary experimental program has been formulated for obtaining this data, and a laboratory has been asked to provide a cost quotation. If the cost is acceptable, a contract will be prepared to carry out this experimental work during the current year.

Physical and Thermodynamic Properties of Catalyst Recovery Solutions

Physical and thermodynamic properties of catalyst-containing streams are needed to design the equipment in the catalyst recovery section of the CCG Process. A brief review of the CCG Study Design identified the most important properties as density, viscosity, boiling point, and heat capacity of aqueous solutions containing up to 30 weight percent of potassium compounds. Temperatures of greatest interest range from 60 to 300°F. A literature search for properties of aqueous solutions containing potassium hydroxide or potassium carbonate has been initiated, and pertinent articles are being collected and evaluated. Methods must be developed to extend the available data to the higher temperatures of interest and to solutions containing several potassium compounds. Future efforts will be directed towards developing such methods.

Slurry Rheology and Solid-Liquid Separations for Catalyst Recovery

The objectives of this program are to identify and evaluate alternatives for solid-liquid separations in catalyst recovery and to investigate the rheological properties of char plus catalyst solution slurries. Work is underway to evaluate alternative solid-liquid separation devices. Laboratory filtration equipment is under construction and will be used to measure filtration rates under a variety of operating conditions.

In the CCG Process, an aqueous slurry of gasifier char and fines is contacted with lime in a digester vessel. The soluble potassium is then counter-currently leached from the solids. The catalyst recovery depends on the solid-liquid separation efficiency between leaching stages. This efficiency is strongly dependent on the particle size distribution. Preliminary lab data indicate that the particles are fragile and break up during processing. Analyses of digested slurries show substantial amounts of solids below 5 μm.

The procedures used in the digestion experiments which produced these samples may have led to excessive particle attrition, which may be avoidable in a commercial system. On the other hand, if these early data prove representative, separations based on centrifugal or gravitational forces may not be economically feasible, and filtration or agglomeration techniques may be necessary. It can already be concluded that particle attritability, in addition to initial particle size, will be a key factor in establishing the suitability of alternative devices.

With this in view, a laboratory unit has been designed to quantify the attritability of char particles as a function of liquid shear rate and potassium content. The device consists of concentric cylinders with the inner cylinder rotating at speeds up to 4000 rpm. This arrangement will provide a well-defined annular shear field for these future attrition studies.

Preliminary filtration data from three batch experiments were analyzed to assess the feasibility of using non-precoated filtration for catalyst recovery. These data were obtained in 1978 under a separate Exxon-funded program. Slurries containing 20% (wt) char and potassium ion concentrations of either 8.5 or 17% (wt) were fed to a laboratory-scale vacuum filter. The results of the data analysis were as follows:

Preliminary Batch Filter Data Evaluation

Sample	Cake Resistance cm/gm	Filtration Rate gal/hr-ft^2 Initial	Filtration Rate gal/hr-ft^2 After Ten Minutes
Sample 1 20% (wt) unwashed char in 17% (wt) K^+ solution	6.5×10^{10}	3.3	0.9
Sample 2 20% (wt) unwashed char in 8.5% (wt) K^+ solution	6.3×10^{10}	4.9	0.6
Sample 3 20% (wt) washed char in 17% (wt) K^+ solution	9.2×10^{10}	4.0	0.8

For all three samples, cake resistances were high relative to the 10^9 cm/gm level typical of commercial systems. Initial filtration rates calculated from the data ranged from 3.3 to 4.9 gal/hr-ft^2. These rates, fairly low for commercial use, fall off to less than 0.9 gal/hr-ft^2 after ten minutes.

Based on this preliminary data evaluation, non-precoated filtration does not appear attractive for catalyst recovery. However, additional data are needed with representative slurries of known particle sizes using different media to confirm this conclusion. Laboratory filtration equipment was designed and is being constructed to help obtain this information.

During the next quarter, attrition studies and laboratory filtration tests will be initiated. Particle attrition will be determined as a function of the shear field and potassium content; filtration characteristics of the slurries will be evaluated as a function of particle size and potassium content. In addition, a general review of solid-liquid separation techniques used in leaching services such as traveling belt filters will continue.

Environmental Control: Water and Solids Effluents

The objective of this program is to generate the data needed for a quantitative assessment of the environmental impact of the CCG Process. The focus of this program will be to characterize wastewaters, spent solids, and solids slurries produced in the CCG PDU. The work during the first quarter included identification of potential liquid and solid waste streams and components, compilation of analytical and preservation techniques for laboratory samples, and a visit to the PDU site.

The PDU sampling program will include liquid waste samples from four locations and solid waste samples from two or three sources. Each of these samples will be tested extensively to determine what contaminants will be present and, of those, which might present effluent quality problems in a commercial plant. As a starting point, solids samples from ongoing $Ca(OH)_2$ digestion experiments will be subjected to preliminary analyses.