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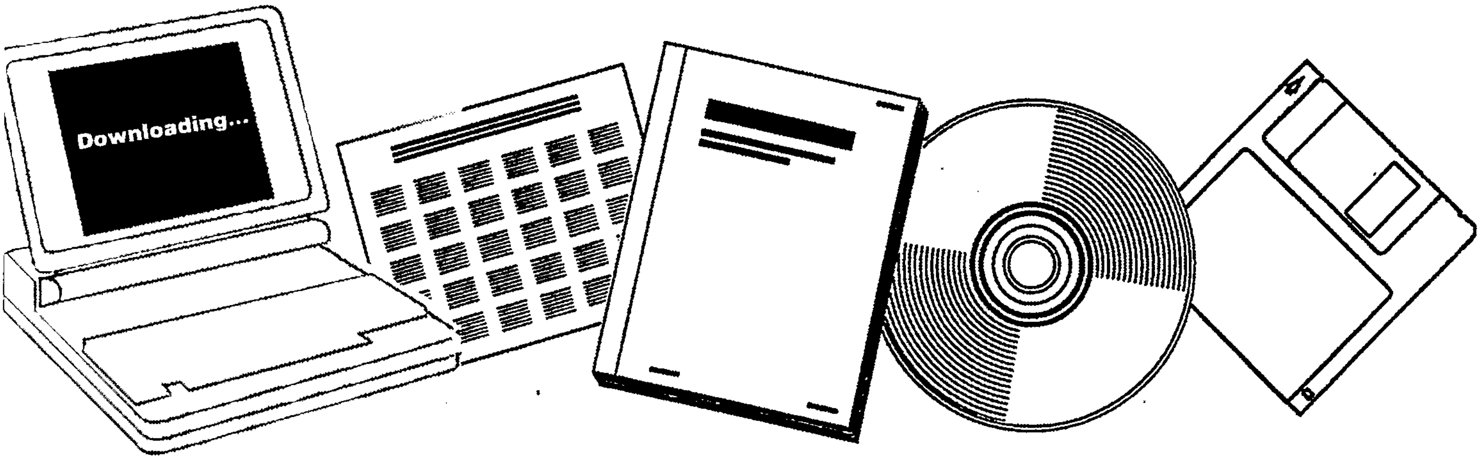
EXXON CATALYTIC COAL GASIFICATION PROCESS DEVELOPMENT PROGRAM. QUARTERLY TECHNICAL PROGRESS REPORT, OCTOBER 1-DECEMBER 31, 1978

EXXON RESEARCH AND ENGINEERING CO.
BAYTOWN, TX

FEB 1979



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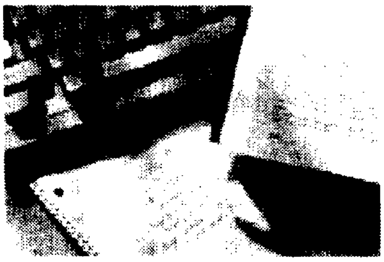

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EXXON CATALYTIC COAL GASIFICATION PROCESS DEVELOPMENT PROGRAM

Quarterly Technical Progress Report
for the period
October 1 - December 31, 1978

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Exxon Research and Engineering Company
Baytown, Texas 77520

February, 1979

PREPARED FOR THE UNITED STATES
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ABSTRACT

This report covers the activities for the Exxon Catalytic Coal Gasification Development Program during the quarter October 1 - December 31, 1978. 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

Construction of three bench scale units to study catalyst recovery was begun. These units include a one-gallon stirred tank for char digestion studies, a series of leaching tanks for char-liquid separation studies, and a fluidized bed reactor for char combustion studies. Construction of the leaching tanks and the char combustion reactor was completed. Preliminary experiments in the char combustor showed that combustion increases both char density and particle size. Directionally, this effect should improve solids-liquid separation in the catalyst leaching operation.

Construction of a bench unit to study cryogenic separation of CO_2 from product gas was also begun. The unit is a batch crystallizer that will permit measurement of solid CO_2 crystal growth rate and nucleation rate as a function of crystallization conditions. Completion is planned for April 1979.

In addition, the construction of a bench apparatus to study reactions of product and recycle gas in furnace and heat exchanger tubes was started. This unit will be used primarily to study the kinetics of the water-gas-shift reaction in the preheat furnace tubes. Completion is planned for the end of January.

- Process Development Unit Operations

The 1/4 ton/day Fluid Bed Gasifier was operated to produce char for the Process Development Unit (PDU) startup and catalyst recovery studies. However, the char density was lower than expected based on operations in the predevelopment program. The reason for this difference is being investigated.

Construction of a single stage catalyst recovery pilot plant was completed. This unit was built to test a proposed catalyst recovery system for the Process Development Unit (PDU). Preliminary results show that many fine particles having a diameter of less than 10 microns were formed in the digester, and therefore, modifications in the digester design and operation are underway to minimize fines formation. The leaching tank gave good solids-liquid contacting with well-mixed tank characteristics.

- Data Acquisition and Correlations

The design of the on-line data acquisition system for the PDU was completed. The system is designed to monitor unit temperatures, flow rates,

pressures, gas analyses, and weights and is capable of monitoring 600 process variables. A computer progam has been written to use this data to calculate an on-line material balance and identify possible operating problems such as faulty instrument readings or process leaks.

The design of the off-line data reconciliation program for the Process Development Unit was set. This computer program will use on-line data collected from the unit as well as analyses of various solids streams to give material balanced yield period information.

A Plexiglas model of the PDU gasification reactor has been built to help troubleshoot any solids flow problems in PDU operation. This cold model has virtually the same dimensions as the PDU gasifier except that it is 1/6 of the height.

- Advanced Study of the Exxon Catalytic Coal Gasification Process

A fixed-bed bench scale gasification unit has been recommissioned to better define catalytic gasification kinetics. A series of shakedown runs were made but material balance was not achieved. Troubleshooting of the unit is underway.

Bench scale agglomeration tests showed that Fluid Bed Gasifier (FBG) feed coal particles larger than 20 mesh agglomerate when heated to 1300°F. Removal of these +20 mesh particles from the FBG feed greatly improved the operability of the unit. Analyses showed that potassium loading on the large particles was less than for small ones, and this low loading could cause the larger particles to agglomerate.

- Engineering Research and Developement

Work continued on a revised offsites facilities definition and cost estimate to update the CCG Commercial Plant Study Design prepared during the predevelopment program (Contract No. E(49-18)-2369). This update will include a more detailed study of water treatment and reuse options as well as a flue gas desulfurization study design for a lime scrubbing system.

Work continued on the study to determine the incentives and data needs for a cryogenic separation system to remove acid gases from the product gas. The process basis and heat and material balance has been completed. The heat integration and refrigeration schemes are nearing completion.

Systems modeling work was initiated to develop material and energy balance tools which will reduce the effort required to do engineering studies under the present contract. Two models are being prepared: a material balance model for catalyst recovery and a material and energy balance model for the CCG reactor system.

Several engineering technology programs began during this quarter. Activities underway by year end include a materials evaluation program, a sour water/catalyst vapor-liquid equilibria program, a slurry rheology/solid-liquid separations program, and two environmental control programs.

TABLE OF CONTENTS

Page

ABSTRACT i

1. BENCH SCALE RESEARCH AND DEVELOPMENT 1
   - 1.1 Catalyst Recovery Studies
   - 1.2 Cryogenic Acid Gas Removal
   - 1.3 Gas Phase Reactions Study

2. PROCESS DEVELOPMENT UNIT OPERATIONS 13
   - 2.1 Char Production for PDU Startup and Catalyst Recovery Operations
   - 2.2 Pilot Plant Catalyst Recovery Studies

3. DATA ACQUISITION AND CORRELATIONS 22
   - 3.1 On-Line Data Acquisition
   - 3.2 Off-Line Data Reduction and Reconciliation
   - 3.3 Cold Model Studies

4. ADVANCED STUDY OF THE EXXON CATALYTIC COAL GASIFICATION PROCESS 34
   - 4.1 Kinetics of Gasification and Devolatilization
   - 4.2 Catalyst Reactions with Coal and Ash

5. ENGINEERING RESEARCH AND DEVELOPMENT 38
   - 5.1 Cost Reduction and Laboratory Guidance Studies
   - 5.2 Systems Modeling
   - 5.3 Engineering Technology Studies

LABORATORY PROCESS RESEARCH AND DEVELOPMENT

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

1.1 Catalyst Recovery Studies

Design and construction of three bench scale catalyst recovery units began in October and continued throughout the remainder of the year. These units are: (1) a fluidized char combustion reactor; (2) a one-gallon digester; and (3) a series of leaching tanks. The leaching tanks and the char combustion reactor have been completed while construction of the digester is continuing. These units will be used to study various options in catalyst recovery.

Bench Scale Fluid Char Combustor

Pilot scale catalyst recovery work has shown that separation of digested solids (char and lime) from a potassium solution is troublesome due to both the effective particle density being 0.2-0.3 g/cc greater than the associated liquid and approximately 1/3 by weight of the char-lime particles being smaller than 10 microns (July-September, 1978 Quarterly Report). Both these properties cause digested solids to settle slowly. An option to enhance the settling behavior of the char particles is to burn the char in a fluidized bed, thereby increasing both the particle density and diameter.

Figure 1.1-1 is a diagram of the fluidized bed reactor used to study the char combustion option in catalyst recovery. This unit operates as follows. Nitrogen and air enter the annular space between the inner and outer tubes and flow downward where the gas is preheated by the furnace. Nitrogen also flows continually down the char fines conveying system. Both gas streams fluidize the solid particles inside the inner tube. Initially, a load of 50-100 gms of coarse char (20 by 50 mesh) is charged to the reactor. The gas stream (2-5% O_2) burns the char slowly so that the char does not overheat and cake. The superficial velocity of the gas is between 0.5-1.5 ft/sec with a combustion temperature between 1100-1800°F.

After the coarse char bed is well combusted (black color vanished), the gas composition is changed to 15-20% O_2 at the same total flow rate. At this point char fines (-325 mesh) is conveyed into the bottom of the bed where it burns in the oxygen rich atmosphere.

It has been found that combustion increases both the char particle density and the diameter. Figure 1.1-2 is a particle size distribution of undigested char before and after combustion. As is shown, many of the char particles in the size range 10-100 μ do agglomerate. Table 1.1-1 shows that particle density does increase during combustion. Both these results favor improved solid-liquid separation.

FIGURE 1.1-1

FLUIDIZED CHAR-LIMESTONE COMBUSTER

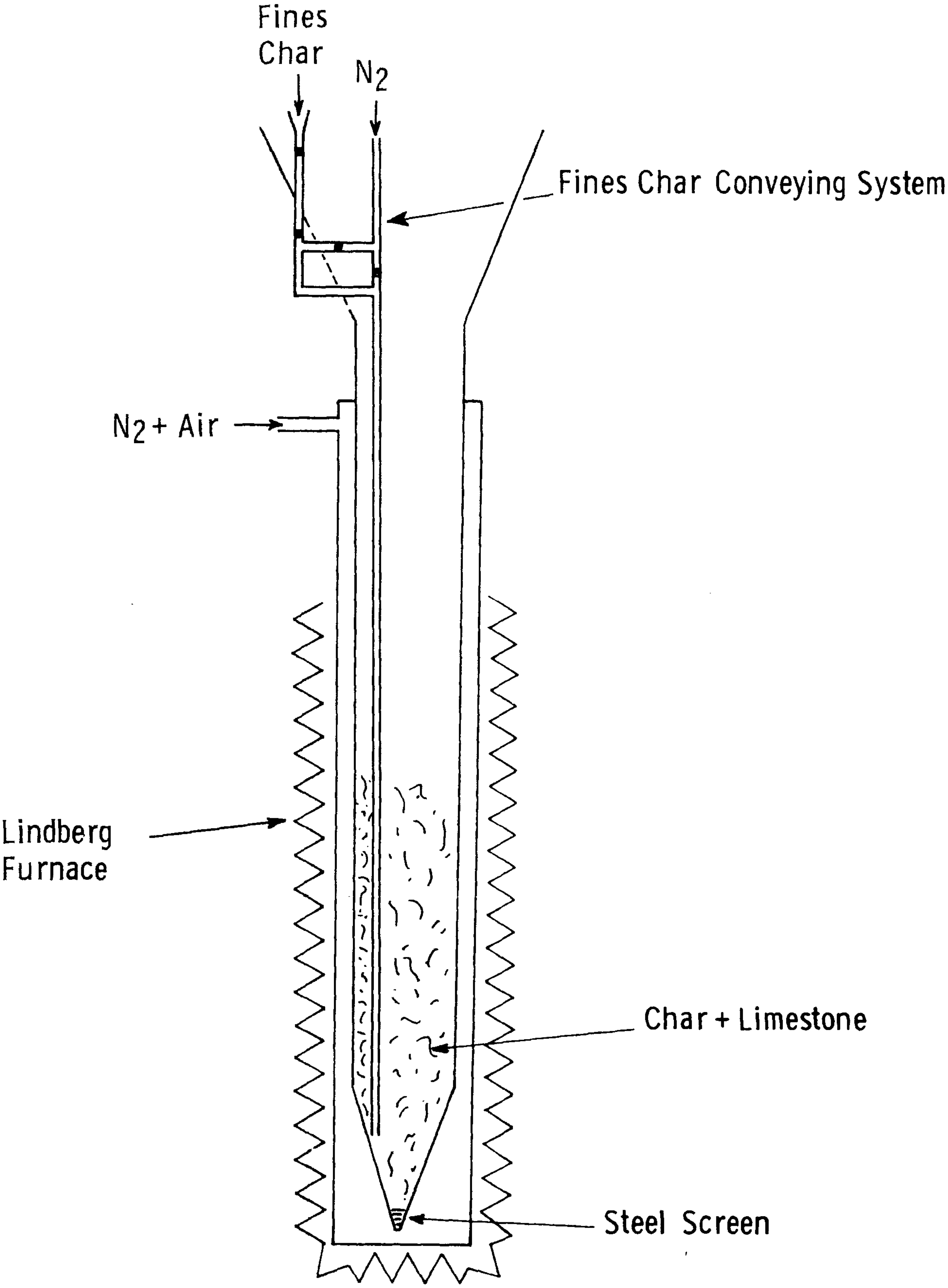


FIGURE 1.1-2

COMBUSTION INCREASES PARTICLE SIZE OF CHAR

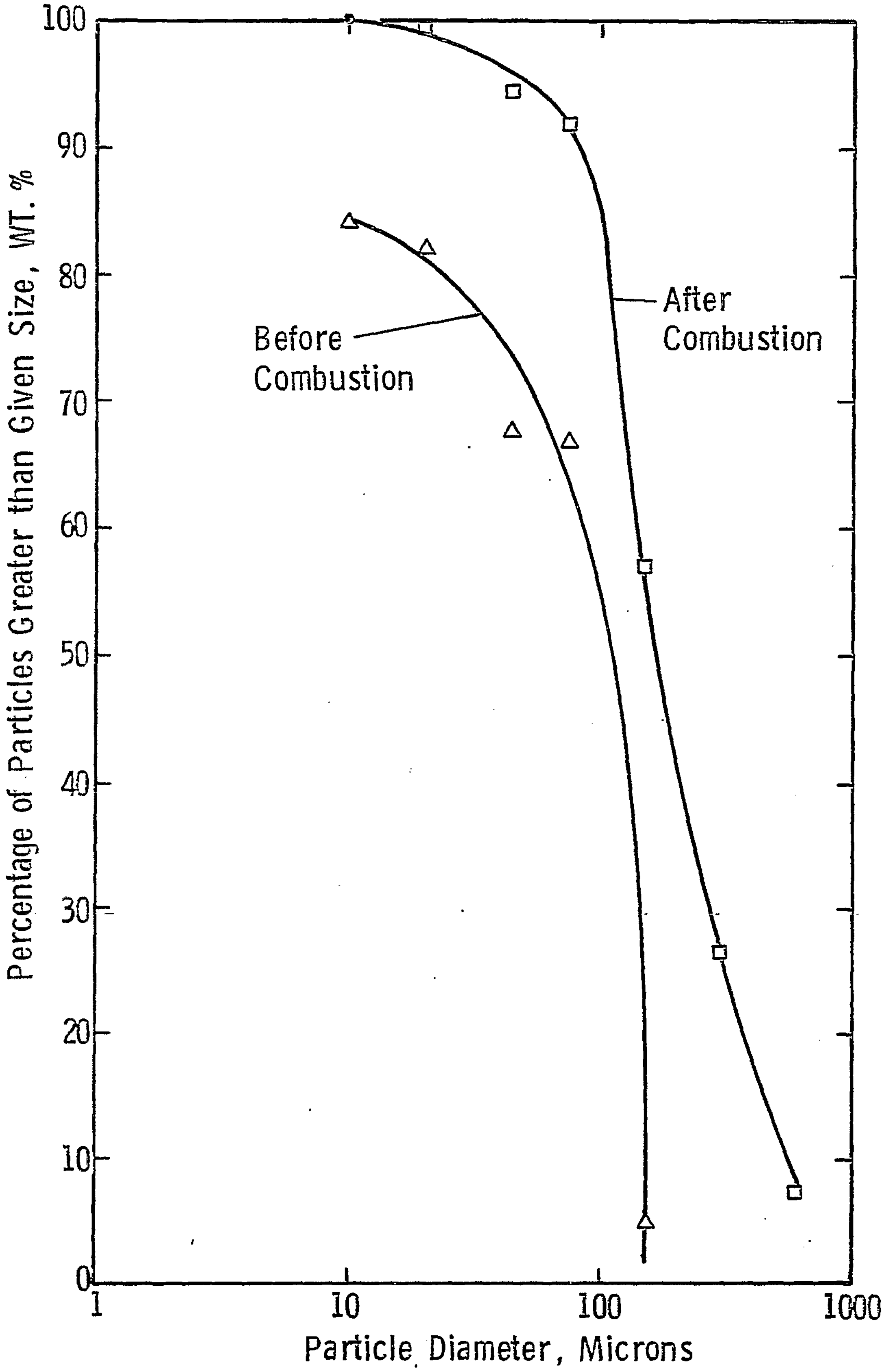


Table 1.1-1

Change in Bulk Density of Char During Fluidized Combustion

Run	Bulk Density Before Combustion g/cc	Bulk Density After Combustion g/cc
42-1	0.24	0.53
42-2	0.24	0.59
45-1	0.60	0.86
45-2	0.60	0.96
46-2	0.60	0.85

Limestone ($CaCO_3$) has also been added to the combustor with the char and was found to calcine to CaO which is a starting material for digestion. Thus, use of some of the heating value of char to produce CaO from relatively inexpensive limestone is possible.

Bench Scale Digester

A one-gallon batch digester diagrammed in Figure 1.1-3 is being constructed to study the char digestion process on a small scale. This digester will be used to simulate the 100-gallon pilot unit discussed in the July-September 1978 Quarterly Report and is designed to heat or cool the reactor contents within ten minutes. This rapid temperature change option makes it possible to determine process temperature and residence time more accurately than was possible in the tubing bombs used during the predevelopment contract. A variable speed impeller will be used to agitate the contents of the digester. This unit will be used to:

- study the interaction of char digestion and combustion
- study the effect of large lime particles on digestion
- investigate the effect of agitation on particle attrition and catalyst recovery
- manufacture slurry for solid-liquid separation studies
- determine the feasibility of a continuous, rather than batch, digester

Bench Scale Leaching Tanks

The small leaching unit shown in Figure 1.1-4 will be used to leach potassium from Illinois No. 6 char-lime solids and from combusted solids. This unit will permit small scale study of the solid-liquid separation process. Water will be passed through the tanks to produce a potassium rich solution.

FIGURE 1.1-3

ONE GALLON RAPID HEATUP-COOLOFF DIGESTER

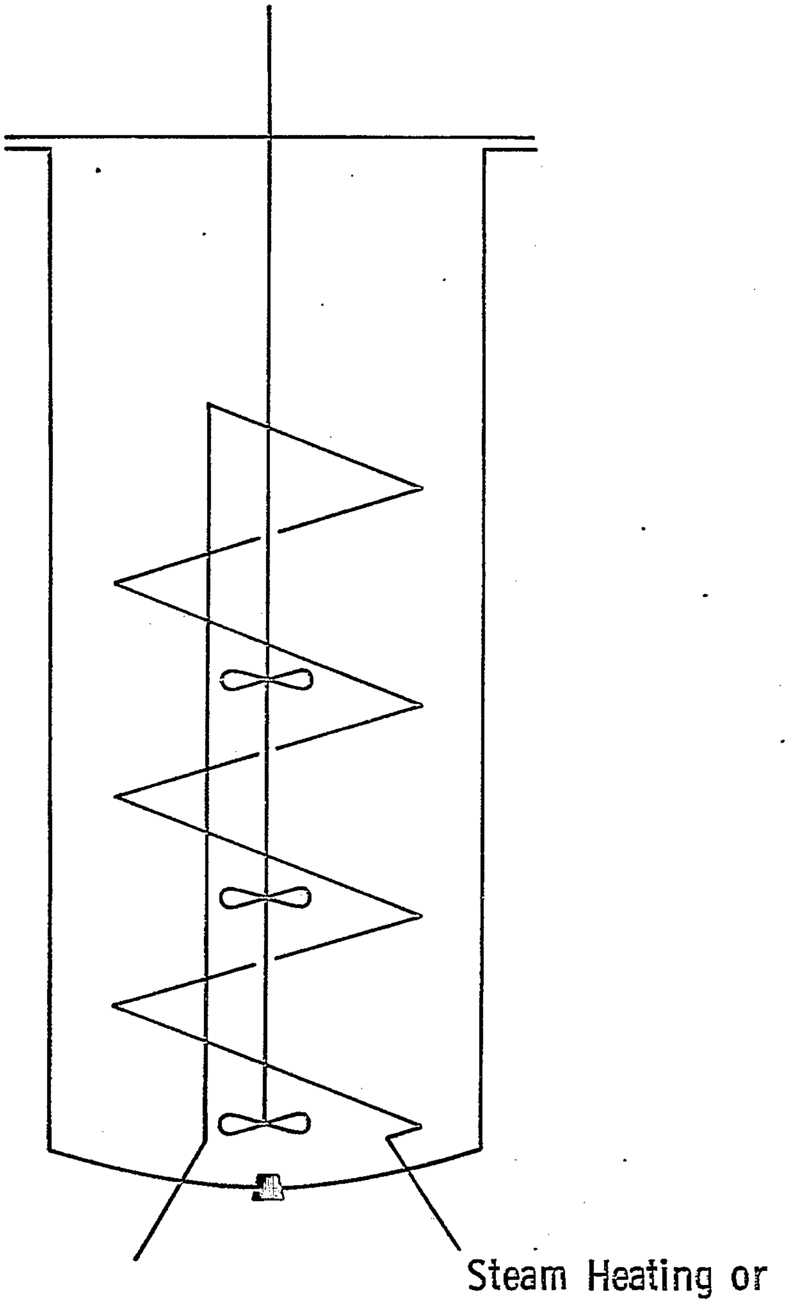
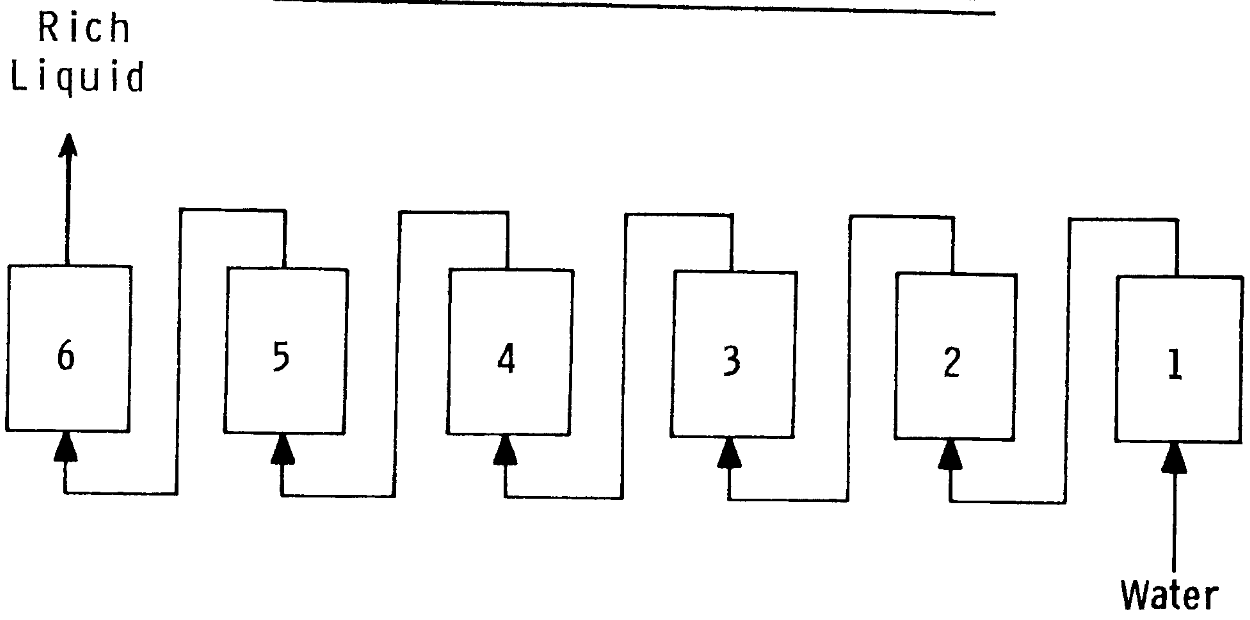


FIGURE 1.1-4

SERIES OF LEACHING TANKS



In this system, solids will remain in a single leaching tank throughout the leaching process. This will reduce particle agitation which causes particle attrition. During a leaching run, the fines content of the liquid overflow from the tanks will be used as a measure of the solid-liquid separation efficiency. This series of leaching tanks will be used to study:

- the effect of potassium concentration on both solid-liquid separation and potassium recovery
- the effect of coarse lime particles from the digestion process on the leaching operation
- the effect of leaching conditions on potassium recovery
- the interaction of char combustion, char digestion, and potassium leaching operations.

1.2 Cryogenic Acid Gas Removal

Background

Existing commercial acid gas removal processes operate by contacting the gas stream with a circulating liquid into which the acid gases are absorbed or reacted. The acid gases are stripped from the liquid in a separate regeneration step, and the liquid returns to the absorber to pick up more acid gases. The Exxon Catalytic Coal Gasification study design uses a heavy glycol as the circulating liquid. This acid gas removal step contributes about 15% to the final SNG cost. Since cryogenic distillation is used as the next processing step to separate H_2 and CO from CH_4, a cryogenic acid gas removal process could be economically attractive.

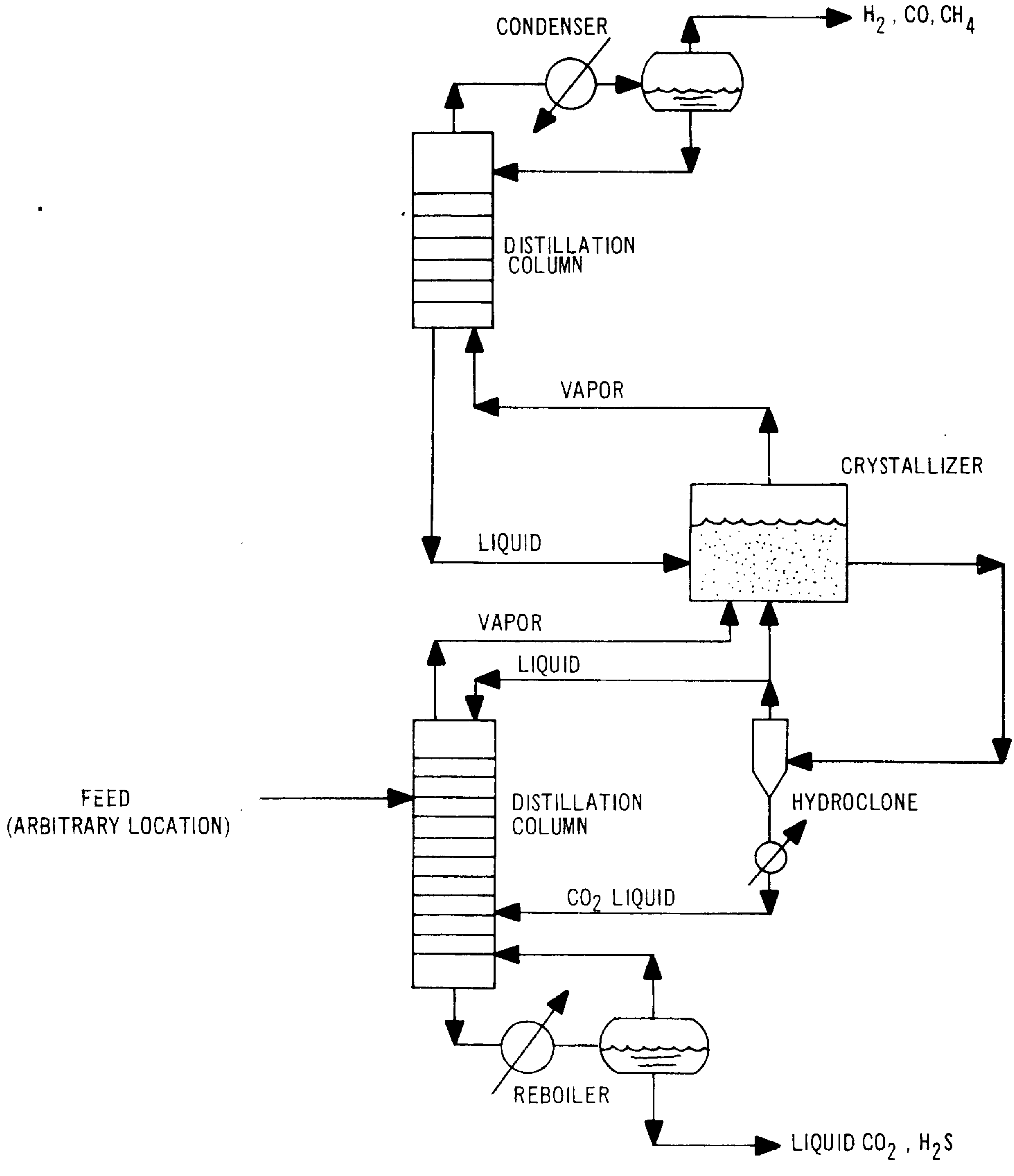
Previous Exxon work has identified CO_2 freezeout as a problem in cryogenic distillation of CO_2 and H_2S from H_2-CO-CH_4 streams. On cooling, CO_2 vapor will freeze solid rather than condense to a liquid at CO_2 partial pressures below the triple point pressure at 75 psia. The maximum operating pressure of an acid gas distillation column is limited by the CO_2 critical pressure of 1070 psia, so the overhead CO_2 concentration can only be reduced to 7% (75 psia/1070 psia) without the possibility of freezeout. However, separation to about 150 ppm is necessary to prevent freezeout in the methane recovery tower.

Separation by Crystallization

Rather than fighting CO_2 freezeout, it is possible to use it to advantage. Figure 1.2-1 illustrates a conceptual process in which a crystallizer has replaced the portion of a distillation column in which CO_2 would normally freeze out. The solid, liquid, and gas phases equilibrate at conditions controlled to give the desired particle size distribution of the CO_2 crystals formed. The system operates at 800-1000 psia with a crystallizer temperature of about -120°F. Slurry from the crystallizer is fed to a hydroclone to generate a thicker slurry of CO_2 crystals in CO_2-CH_4 liquid and a clear overhead stream. Some of this clear liquid is returned to the crystallizer to

FIGURE 1.2-1

CRYSTALLIZATION PROCESS FOR CRYOGENIC ACID GAS REMOVAL



control the slurry density. The remainder is fed to the lower half of the distillation column to have the methane stripped out of it. The CO_2 slurry is melted and returned to the lower part of the column on a stage where the liquid composition approximates that of the melted slurry. Other process configurations using crystallizers may also be developed.

Experimental

Although there is extensive literature about crystallizer design and crystallization parameters for various chemical systems, there is no data available for the CO_2-CH_4-CO-H_2 system. The work to be performed under the contract will initially attempt to derive growth rate and nucleation rate data for the crystallization of CO_2 from binary CO_2-CH_4 mixtures. The effect of CO and H_2 will be studied later. With this information, larger crystallizers can be designed using established techniques.

The experimental apparatus for this study is under construction with completion scheduled for March 1979. The unit is schematically presented in Figure 1.2-2. The crystallizer will be operated batchwise by filling the precooled crystallizer (about four liters capacity) with liquid CH_4 and CO_2 in the approximate proportion desired. Methane rich vapor will then be flashed off to lower the liquid temperature and concentrate the CO_2 in solution. When the desired supersaturation is reached, the liquid mixture will be analyzed with an on-line gas chromatograph.

Crystallization will not occur in mixtures of moderate supersaturation until a seed crystal is introduced. Seed crystals will be formed by freezing some of the mixture onto a small coil through which liquid nitrogen vapor at -320°F is flowed. Alternately, a dry ice seed crystal of known dimensions may be added to the liquid mixture through a pressure tight lock (not shown). After seeding, the bulk liquid will crystallize until the supersaturation is exhausted. During freezeout a temperature history of the liquid will be recorded which will be the source of growth and nucleation rate data. Both initial subcooling and composition will be varied to determine the dependence of the growth and nucleation rates on them.

After the system has crystallized, the slurry will be agitated and photographed to determine the size and number of CO_2 crystals. This size distribution data will be used to corroborate the growth and nucleation rate data and to predict the operation of solid-liquid separation equipment such as hydroclones.

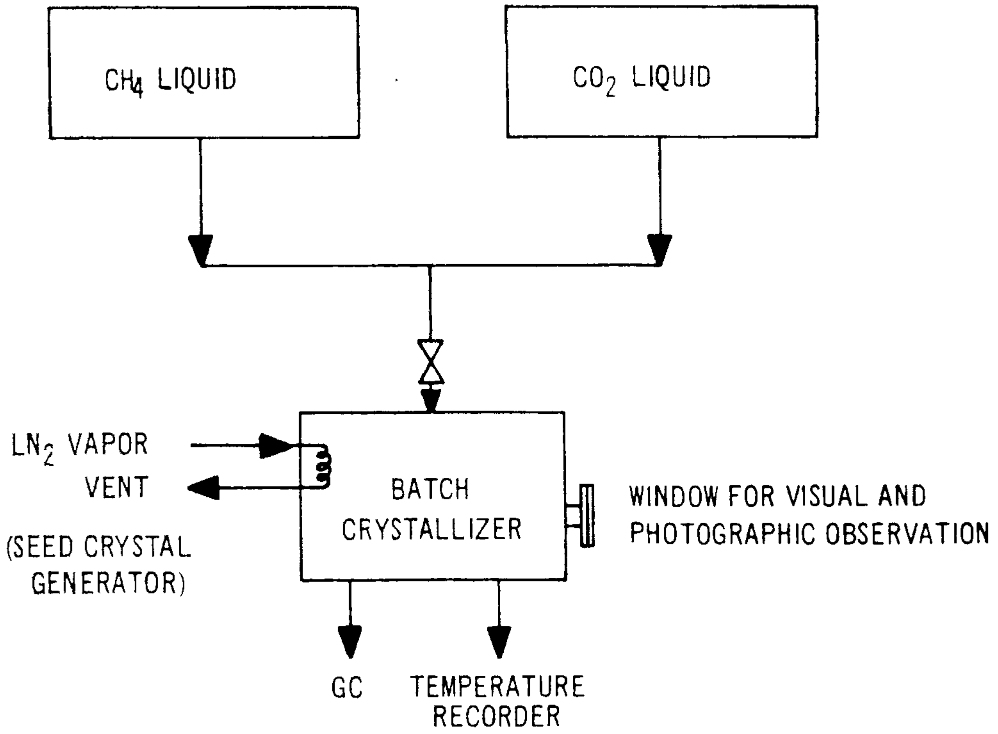
1.3 Gas Phase Reactions Study

Background

In the Exxon Catalytic Coal Gasification Process study design, a gas recycle stream of H_2, CO and some CH_4 is preheated with the steam necessary for gasification to around 1575°F to provide heat input to the gasification reactor. This heat input comes from two sources: sensible temperature difference and chemically stored energy. At the reactor temperature of

FIGURE 1.2-2

SCHEMATIC EXPERIMENTAL CRYSTALLIZATION APPARATUS



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1300°F, the recycle mixture is not at equilibrium with respect to the mildly exothermic water-gas shift reaction

$$H_2O + CO \rightleftharpoons H_2 + CO_2 \qquad \Delta H \approx -8 \text{ kcal/gmole}$$

so when the gases are equilibrated in the gasifier, the shift reaction provides an additional heat input. If, however, the shift reaction occurs in the recycle preheat furnace, that heat of reaction is not released in the gasifier and must be replaced by a larger sensible heat contribution. This in turn requires a higher coil outlet temperature from the furnace which will result in increased furnace costs.

Engineering evaluations have shown that a reformer may be cost competitive with a furnace if a coil outlet temperature higher than ~1575°F is required from the furnace. A reformer uses the endothermic reforming reaction

$$CH_4 + H_2O \rightleftharpoons 3H_2 + CO \qquad \Delta H \approx 50 \text{ kcal/gmole}$$

to add energy to the recycle stream at lower temperatures, in the opposite manner from which the exothermic shift reaction necessitates a higher coil outlet temperature. Investigation of the extent of the shift and reforming reactions in the recycle stream at commercial conditions is therefore necessary to choose and design the best recycle stream heat input option.

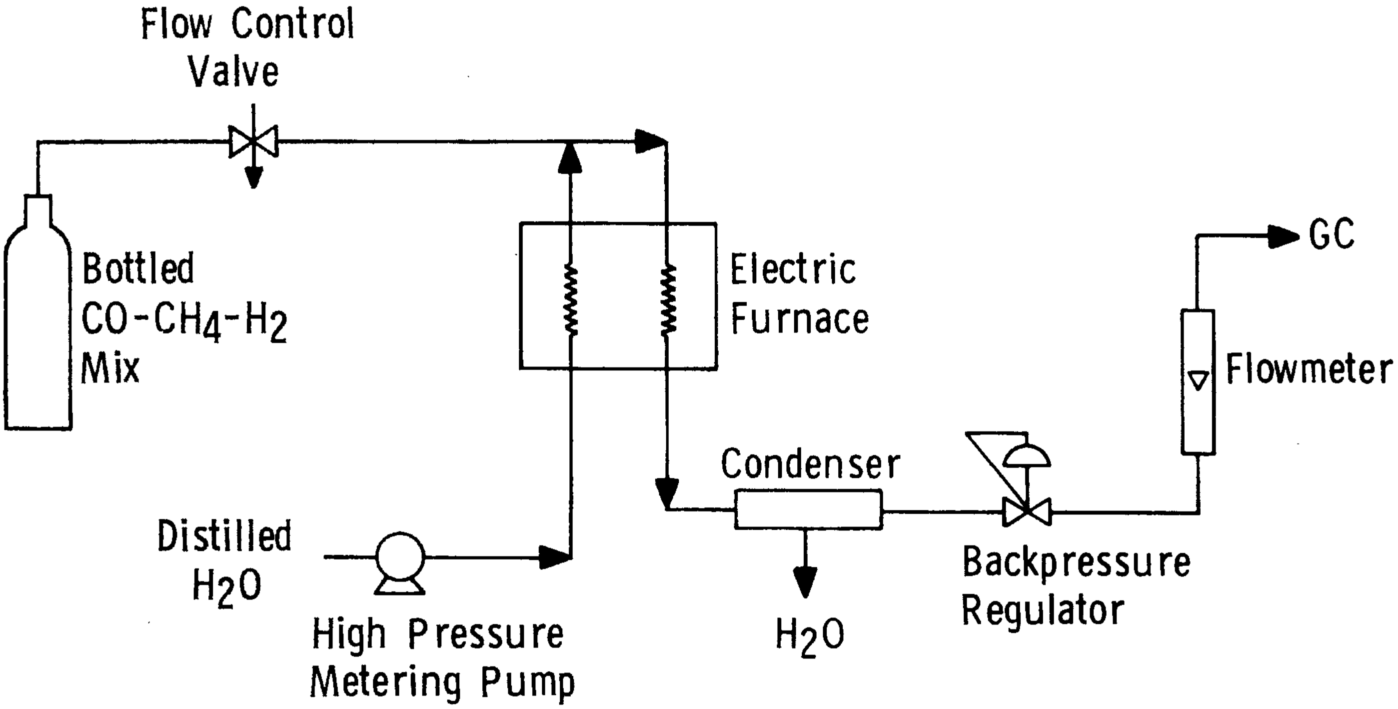
Experimental

The apparatus for this study is shown in Figure 1.3-1. A bottled mix of H_2, CO and CH_4 in the proportions of the recycle gas will be combined with steam generated by pumping a metered amount of water at pressure through an electric furnace. This mixture will then pass through the furnace at conditions representative of the commercial recycle gas furnace. A condenser will remove unreacted steam from the furnace effluent and a gas chromatograph will analyze the product gas mixture for any change in composition.

The initial variables to be studied are temperature and the possible catalytic effect of different metals in contact with the hot gas stream. Catalysis of the shift and reforming reactions by the furnace tube walls will be studied by filling the experimental furnace with various alloy wire meshes, using a range of stainless steels and nickel alloys. Startup and shakedown of the unit will begin in late January 1979.

FIGURE 1.3-1

EXPERIMENTAL APPARATUS FOR RECYCLE FURNACE REACTION INVESTIGATION



2. Process Development Unit Operations (Reporting Category C02)

2.1 Char Production for PDU Startup and Catalyst Recovery Operations

The Fluid Bed Gasifier (FBG) was operated during the quarter with the objective of producing an inventory of high conversion, steady-state char. The FBG, which gasifies 1/4 ton of catalyzed Illinois coal per day, was used during the predevelopment contract work on the Exxon Catalytic Coal Gasification Process.

Fifteen hundred pounds of char are needed initially to load the Process Development Unit (PDU) which is to operate under the current contract. A fluidized bed of catalyzed char is preferred for startup of a catalytic gasifier in order to dilute the feed coal to ensure that any liquid devolatilization products from the coal are destroyed. Sufficient char is needed during startup operations so that the solids handling system on the PDU can be tested. These include the lockhoppers and coal feed system, the gasifier cyclone and char return leg, the gasifier filters, and the char withdrawal system. These char requirements for the PDU were fulfilled during the quarter.

Another 1000 pounds of char are needed for use in the PDU prototype catalyst recovery unit. This unit consists of a digestion vessel for the reaction of char with calcium hydroxide ($Ca(OH)_2$) and also another vessel to act as one stage of a proposed multistage countercurrent water leaching apparatus. Studies in these units will determine equipment operability and define the process variables needed for design of the catalyst recovery section of the PDU. Additionally, small amounts of char at various conversion levels will also be needed for kinetic studies in bench scale reactors. These chars are scheduled to be produced in January and the first part of February.

FBG char production operations during the quarter were generally good. However, the fluidized char bed density was lower than expected compared to Illinois coal operations during the predevelopment contract. This was due primarily to swelling of char particles as indicated by direct observation and by a decrease in char bulk density compared to the earlier char. Possible causes for this difference in density are being intensely investigated. Changes in operating conditions compared to the last operating period include:

(1) greater range in feed coal size including more -8/+16 mesh and more -325 mesh particles

(2) KOH catalyst in place of K_2CO_3

(3) different train load of Illinois coal

(4) more effective nitrogen blanketing in catalyst addition and coal drying system.

To date the first two changes have been investigated. The -8/+16 mesh particles were difficult to feed through the FBG's 1/2-inch feed line and removing them improved operability. The char density was unaffected however.

Returning to a more dilute potassium carbonate (K_2CO_3) catalyst solution for impregnating the coal also did not measurably improve the fluidized bed density.

Numerous coal samples have been submitted for laboratory analysis to investigate possible differences between the present coal and that used earlier. These tests include elemental and ash element analysis, free swelling index, equilibrium moisture, and sulfate sulfur analysis. Results will be available in January. To ensure that operational differences have not been responsible for lower density char, higher steam and lower coal feed rates will be investigated.

Regarding the last change, some air along with the nitrogen blanket will be allowed to enter the catalyzed coal drying system since oxygen is known to help prevent swelling in agglomerating coals. Current engineering design predicts a small amount of oxygen in the flue gas used to dry catalyzed coal commercially. Therefore, the inclusion of air will permit the system to operate more in line with projected commercial conditions. This test is also scheduled for January.

2.2 Pilot Plant Catalyst Recovery Studies

The basic concept of catalyst recovery in the Catalytic Coal Gasification Process is a digestion step to free potassium chemically bonded to the coal mineral matter followed by a multistage countercurrent leaching. This concept is illustrated in Figure 2.2-1. Previous preliminary pilot plant and bench unit data (July-September, 1978 Quarterly Report) shows that potassium recoveries of 90%+ can be achieved with this operation.

This concept is the basis of a system proposed for the PDU catalyst recovery unit as shown in Figure 2.2-2. The system is a batch countercurrent contact system and consists of a number of batch-contact units arranged in a circle or row called an extraction battery. The key feature of this system is that solids remain stationary in each tank but are subjected to a multiple number of contacts with extracts of diminishing concentration. Fresh solvent contacts the most nearly exhausted solids (Tank 1) while concentrated solution leaves the system in contact with fresh solids (Tank N-1). Countercurrent operation of the battery is obtained by advancing the position of the tanks in the cycle. Since handling of the char-lime solids tends to break the solids into finer particles and thereby aggravates the solids-liquid separation problem, this system will subject the solids to the required number of contacts and yet minimize handling of the char-lime particles.

Pilot Plant Design

To verify the feasibility of the proposed PDU leaching system and to collect data for the design, a single stage pilot scale system was built. Figure 2.2-3 is a diagram of the system. The system consist of a digester and one leaching tank. In the digester, char and lime are reacted with a KOH solution to solubilize potassium chemically bonded with the coal mineral matter. The leaching tank is used to wash solubilized potassium from char-

FIGURE 2.2-1

DIGESTION AND MULTISTAGE COUNTERCURRENT LEACHING

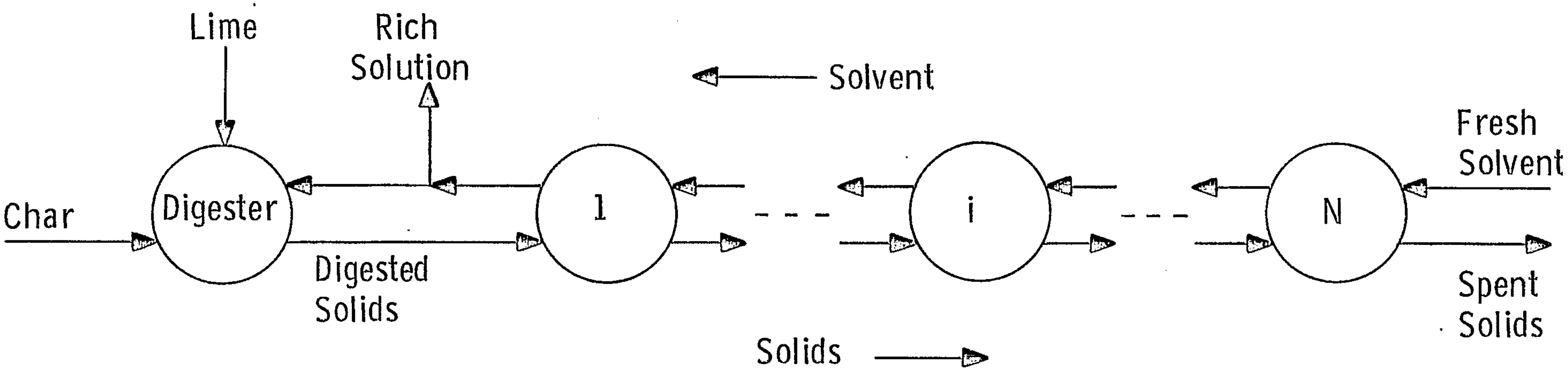


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FIGURE 2.2-2

BATCH COUNTERCURRENT MULTIPLE-CONTACT LEACHING SYSTEM

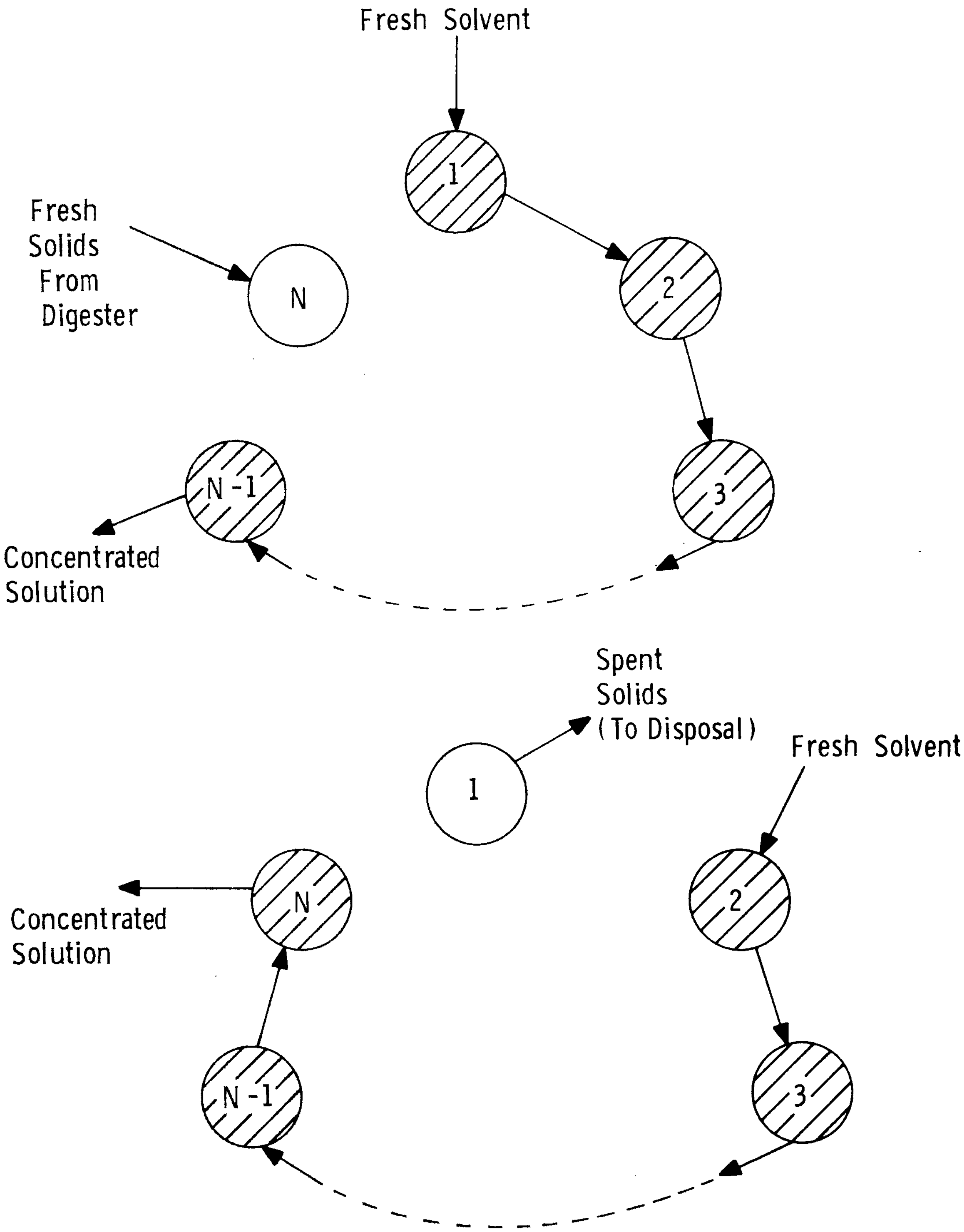
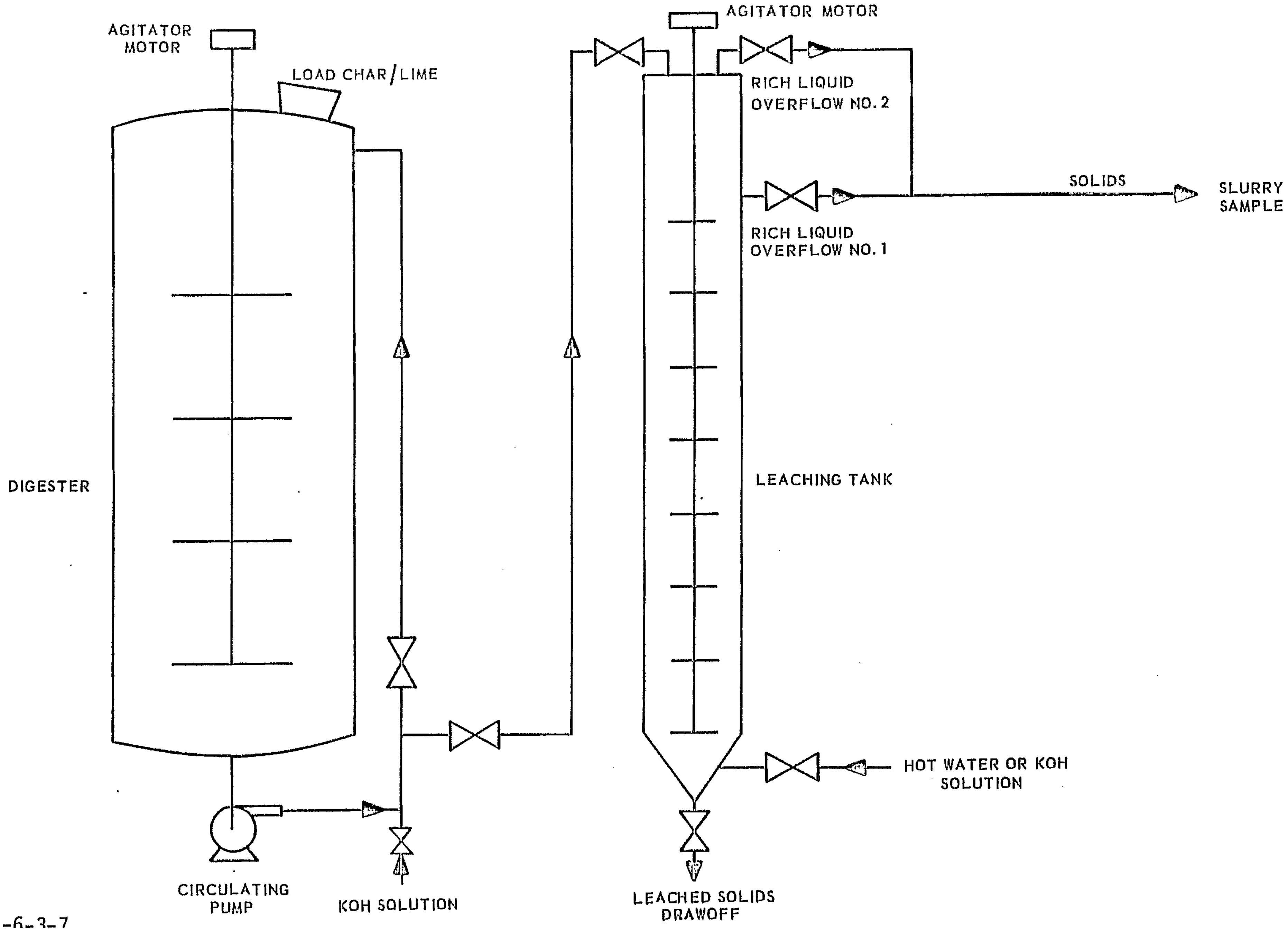


FIGURE 2.2-3
SINGLE STAGE CATALYST RECOVERY PILOT PLANT



lime solids. The PDU unit will have a series of leaching tanks to achieve countercurrent operation.

Initial runs of this digestion and leaching system have been made to eliminate operational problems and gather preliminary data. Seal failures on the circulating pump have been a problem and this is attributed to erosive wear of the seal faces by the char particles. A new pump which minimizes slurry contact with the seal faces has been ordered. As an additional benefit, the new pump minimizes particle contact with moving surfaces which should reduce particle degradation.

Pilot Plant Operations

A limited amount of operating experience has been obtained on the unit so far. It has been learned that a small flow of liquid must always be maintained to the leaching tank in order to prevent the solids from settling into and plugging the leached solids drawoff. Also, a change has been implemented in the preparation for leaching runs. Previously enough char and lime was digested for two leaching runs and half the resulting slurry was sent to the leaching tank for each leaching run. However, analysis of the data led to the conclusion that the solids in the slurry sent to the leaching tank were not necessarily half the amount charged to the digester. This is because of settling of the solids on the relatively flat bottom of the digester. Now, only enough solids for one leaching run are digested at a time which results in a more accurate solids material balance.

It is desirable for the leaching tank to operate as a continuous stirred tank in order to achieve good solids-liquid contacting. For a stirred tank, the concentration of potassium in the water solution leaving the tank decreases with time as shown in the equation below

$$\%K^+ = (\%K^+)_{\mathrm{initial}} \exp(-Ft/V)$$

where F is the flow rate of solvent, t is the time, and V is the tank volume. Thus a semi-log plot of $\%K^+$ versus time should be a straight line with slope of -F/V. Figure 2.2-4 is typical of the preliminary data from the runs made to date. The plot is linear with a slope of $-0.57\ \mathrm{hr}^{-1}$ which is close to the calculated value of $-0.50\ \mathrm{hr}^{-1}$. This indicates that the leaching tank operates as a well stirred tank.

An excessive carryover of solids from the leaching tank was observed in the leaching runs completed this quarter. The unexpectedly large carryover is due to large amounts of extremely small particles. Figure 2.2-5 shows a plot of the particle size distribution of the char fed to the digester and the solids after digestion. Before digestion, none of the char particles are less than 10 microns; after digestion with the circulating pump running, over 60% of the solids were less than 10 microns. It is believed that the circulating pump, which is run continuously during digestion, is causing the breakdown of the particles.

FIGURE 2.2-4

LEACHING TANK PERFORMANCE

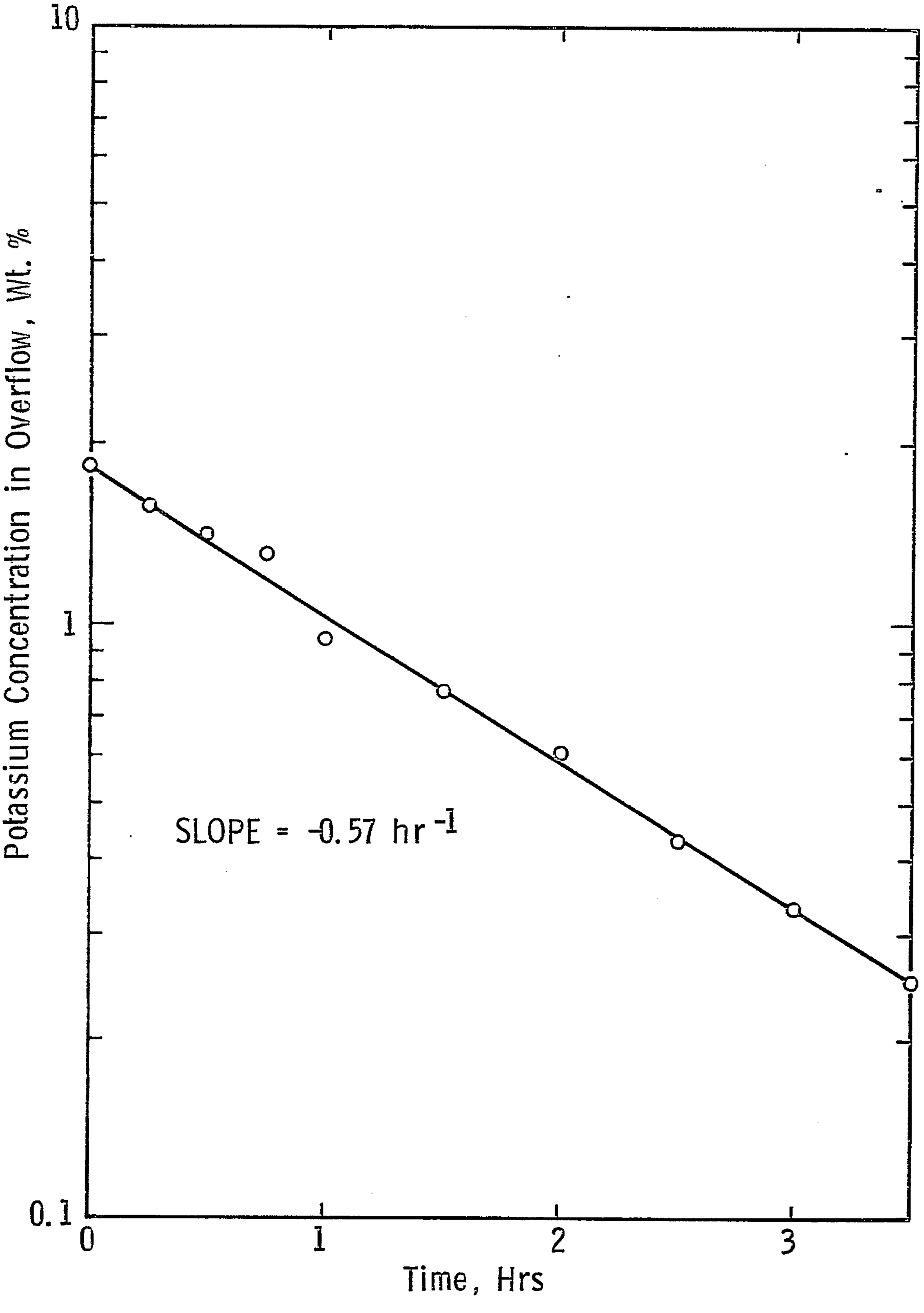
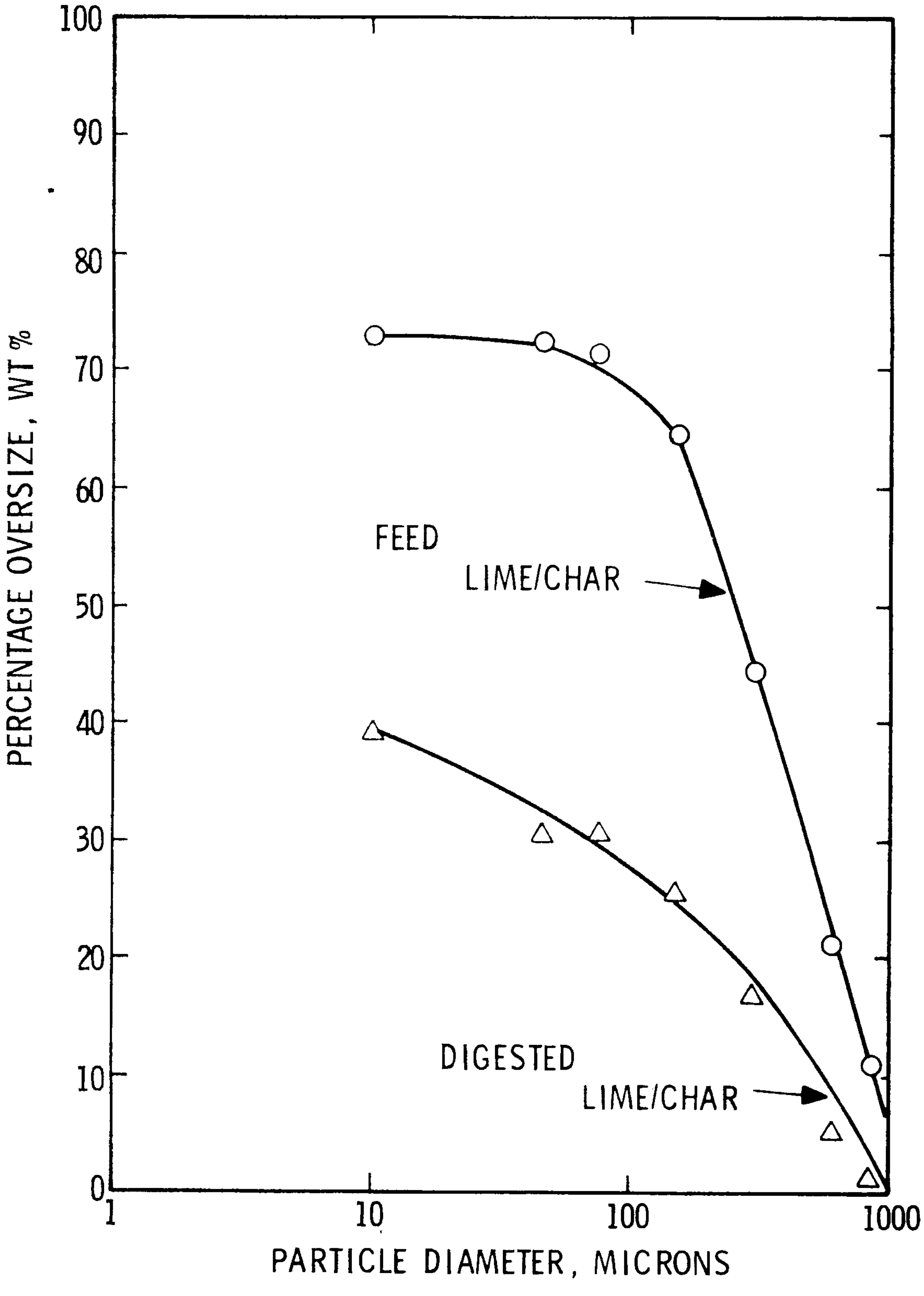


FIGURE 2.2-5

PARTICLE DEGRADATION DURING DIGESTION



Several runs were attempted without the circulating pump running but with the agitator to provide mixing. In these cases, lime tended to settle out of the solution and fill the bottom of the digester. Based on these runs, it was concluded that the circulating pump is necessary to keep the lime suspended. As mentioned previously, a new pump has been ordered which minimized particle contact, thus reducing particle degradation. To further cut down particle degradation, the new pump will not be run continuously during digestion, but only when necessary to keep the lime in suspension.

3. Data Acquisition and Correlations Reporting Category C03)

3.1 On-line Data Acquisition

The main purpose of the on-line data acquisition and reduction system is to monitor the PDU pilot plant operation and to provide means for the evaluation of the operating data. The design of the system shown schematically in Figure 3.1-1 is complete. The analog signals from sensors on the unit, such as pressure transmitters, weigh cells, and thermocouples are converted to digital form in the analog/digital converter. This data is then transferred to the memory core in the central process unit (CPU) of a TI-960 minicomputer. The memory core contains software programs necessary for the alarming, logging, and operator interface functions for the Process Development Unit (PDU). Substantial modifications have been made to the Standard Texas Instruments Process Automation Monitor/Disc(PAM/D) operating system. The modifications will improve the performance and reliability of the system, notably in the areas of table management, message storage and interrupt servicing. Data reduction is accomplished through application of software programs. The reduced data are stored on the disc for future displays on cathode ray tubes (CRT) or printers, and for storage on magnetic tapes.

The system provides several interrelated functions discussed briefly as follows:

Routine Data Processing and Acquistion

The routine data processing includes scanning of all digital and process data variables at intervals ranging from once every 20 seconds to once every hour and converting digital and analog data to engineering units. The types and approximate number of process variables are tabulated below.

Type of Measurement	Number of Measurement Points
Temperatures	400
Flows	30
Pressures	60
Gas Analyses	100
Weights	10
TOTAL	600

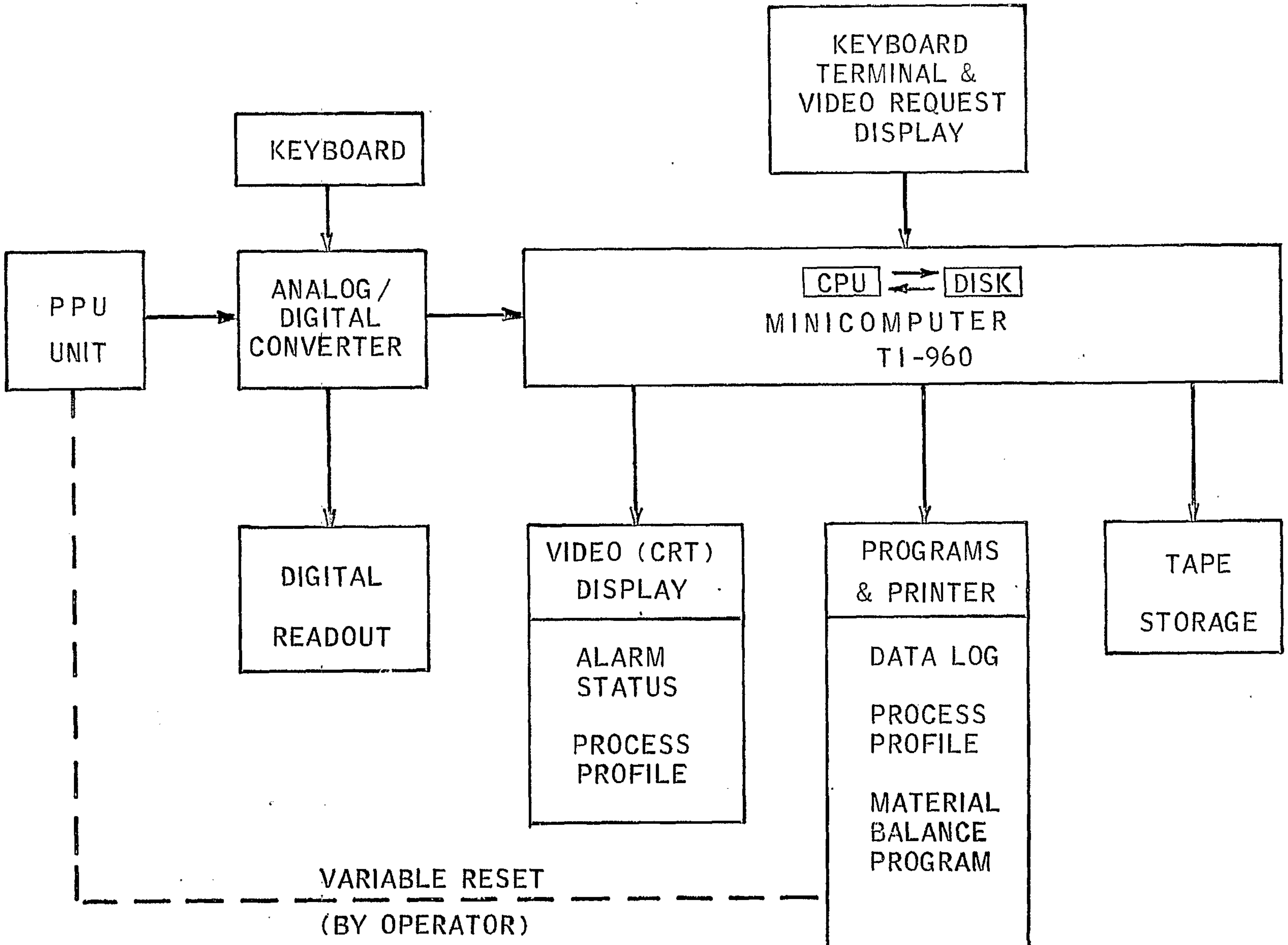
During unit operation, the values of all process variables will be instant=ly available to the operators in the form of a digital readout accessed by a keyboard in the control room. The computer has also been programmed to provide operation profiles displayed on the operator request CRT screens.

Alarm Processing and Checking

The system is capable of determining if the process variables go above or below their maximum or minimum allowed values. Variable alarms result in a

FIGURE 3.1-1

PDU ON-LINE DATA ACQUISITION SYSTEM



printed message displayed on alarm CRT and printers. For most variable alarms, the system also updates the variable's status. The displays on alarm CRT's are updated once a minute with current alarm information.

Data Logging

Three different log formats are available. An hourly log consists of all the values for a shift through the last hour for each variable. A period log consists of averages for a specified period for each variable. A demand log consists of the current value and previous hour average, maximum, and minimum for each variable. Both the demand and period average logs can be requested as desired.

On-line Data Evaluation

On-line data evaluation is accomplished through application of a material balance program stored in the memory core of the computer. This program input consists of 44 automatic computer measurements such as temperatures, pressures, flows, etc. Four material balances (overall, hydrogen, oxygen and syngas balances) as well as average unit conditions are computed and printed in hard copy. This program not only provides guidance on conditions required to achieve a desired conversion but also aids in locating operating problems. An example of the output is shown in Table 3.1-1 using simulated data as input.

The required software programs have been tested and will be implemented as part of PDU operations.

3.2 Off-line Data Reduction and Reconciliation

The primary purpose of the off-line data reduction and reconciliation is to provide consistent and reliable data for use in correlations, commercial plant study design, and kinetic model development. For an integrated catalytic gasification pilot plant, more than 500 process measurements including gas and solids analyses will be collected. Much of the data describing plant operations have some inaccuracies due to random instrumentation errors. Furthermore, some data points may be in error as a result of faulty or incorrectly calibrated meters. As a result, raw operations data may not exactly satisfy material balance constraints. Use of these inconsistent and erroneous data for feasibility studies and decision making may lead to incorrect conclusions. To resolve the inconsistencies in the pilot plant data, a data reconciliation technique is used. Data reconciliation consists of adjusting the measured operations data based on the estimated tolerances assigned to each variable. That is, the most reliable data will be changed least and the least reliable data the most in order to satisfy the material balance constraints. In this way, the random instrumentation errors will be corrected, unmeasured quantities will be determined, and faulty measurements will be isolated and flagged for correction.

The mathematical formulation of the data reconciliation problem consists of:

Table 3.1-1

```
PDU ON-LINE GASIFIER MATERIAL BALANCE

       ENGLISH UNIT SYSTEM  (Simulated data)

 GASIFIER MATERIAL BALANCE ****************************************************************
 INPUT:                (LBS/HR)              OUTPUT:                   (LBS/HR)
   COAL+CATALYST       123.46                  PRODUCT GAS              191.34
   STEAM               179.01                  PRODUCT WATER            114.75  PRES(A-I)
   SYN GAS              47.47                  CHAR ENTRAINED             6.00  HR    PSI
                                               CHAR WITHDRAWN            29.98  LAST 6.1
 TOTAL                 349.94                TOTAL                      342.07  /1ST 6.1
 CLOSURE: (OUTPUT+ACCUM/INPUT) = 97.7 %        ACCUMULATION               0.0

 SYN GAS BALANCE ***********************************************************
 INPUT:                  (SCFH)              OUTPUT:                    (SCFH)
   GASIFIER SYN GAS      2115.0                H2+CO IN PRODUCT GAS 1995.0
 CLOSURE: OUTPUT/INPUT =    94.3 %

 ****** HYDROGEN BALANCE (SCFH H2) ************ OXYGEN BALANCE (SCFH O2) ******
 INPUT:             OUTPUT:                   INPUT:           OUTPUT
   COAL+CAT1106.3   PRODUCT GAS 3880.5          COAL+CAT 176.6   PRODUCT GAS 1064.4
   STEAM   3769.0   PG WATER    2416.0          STEAM   1884.5   PG WATER    1360.2
   SYN GAS 1586.2   CHAR ENTRND    7.9          SYN GAS  264.4   CHAR ENTRND    0.0
                    CHAR WTHDRN    4.4                           CHAR WTHDRN    0.0
   TOTAL   6461.5   TOTAL       6308.8          TOTAL   2325.4   TOTAL       2424.6
 CLOSURE: (OUT/IN) =  97.6 %                  CLOSURE: (OUT/IN) = 104.3 %

 UNIT CONDITIONS *********************** ACTUAL UNIT ***** TARGET ***********
                                          CONDITIONS      CONDITIONS
   GASIFIER TEMPERATURE           (DEG F)   1302.8           ----
   GASIFIER PRESSURE               (PSIA)    515.1           ----

   CARBON CONVERSION (GC ANALYSIS)    (%)     81.1           ----

   STEAM  CONVERSION (H2O BALANCE)    (%)     37.4           43.1
   STEAM  CONVERSION (H2  BALANCE)    (%)     33.5            "
   STEAM  CONVERSION (O2  BALANCE)    (%)     34.7            "

   TOTAL CH4 MADE   (SCF CH4/LB C IN FEED)    15.5           10.9
   CH4 IN DRY N2 FREE PRODUCT GAS  (MOL %)    28.9           30.3

 UNIT CONTROL VARIABLES ******************************************  NEW *****
                                                                  SET POINT
   STEAM FEED RATE                (LBS/HR)    179.0          152.7          165.8

   SYN GAS FEED RATE            (SCFH) MOL % 2115.0 MOL %  1642.7  MOL % 1878.8
        H2 FEED RATE            (SCFH) 75.00 1586.2  77.71 1276.6  76.18 1431.4
        CO FEED RATE            (SCFH) 25.00  528.7  22.29  366.2  23.82  447.5

 EQUILIBRIUM CONSTANT ************ EQUILI. *** TARGET **** ACTUAL **** EQUILI **
                                   CONSTANT   CONDITIONS  CONDITIONS  TEMPERATURE
   GRAPHITE+H2O: C + H2O=CO + H2    1.8474      1.5778      1.4979      1281.0
   SHIFT       : CO+ H2O=CO2+ H2    1.5157      1.5157      1.2991      1368.9
   METHANATION : CO2+3H2=H2O+CH4    0.0665      0.0665      0.0578      1311.7
   OVERALL     : 2C+2H2O=CO2+CH4    0.3440      0.2509      0.1686
```

$$\text{minimize:} \quad f(R) = \sum_i \frac{(Mi - Ri)^2}{\sigma i^2} \qquad i = 1, \ldots, NVAR \quad j = 1, \ldots, NC0N$$

$$\text{subject to:} \quad E_j(R) = 0$$

where: Mi = Measured value of variable i
Ri = Reconciled value of i
σi = Standard deviation of the ith measurement
E_j = Set of nonlinear equations representing the physical relationships among the variables
NVAR = Number of variables
NC0N = Number of constraints

Standard deviation is defined in terms of reliability for each measured variable as follows:

$$\sigma_i = M_i \cdot rel_i/200$$

Reliabilty (rel_i) is an estimate of the quality of the individual data points based upon the user's experience. For example, a reliability of 10% implies that if a measuring device is functioning properly, it will measure to within ±10% of the true value 95% of the time (i.e., two standard deviations). Thus, a small numerical value for reliability indicates the measured value is of high quality.

The objective function (f) represents the sum of the deviations of the reconciled variables from the measurement values. These deviations are weighted by the user's estimate of the reliability of the measurements. During the iterative minimization of the objective function, the algorithm attempts to keep the reconciled values for the reliable measurements close to the measured values. The constraints which describe the physical relationships of the process variables (such as material balances) must be satisfied during the minimization of the objective function.

For the integrated Catalytic Gasification Process Development Unit (PDU), 150 variables are defined which are involved in the material balances. Among these are the input and output volumetric flows, gas streams compositions and the gasifier solids analyses. Table 3.2-2 lists these variables and their reliabilities. The constraints are elemental and material balances of each section of the PDU (gasification, acid gas removal, and cryogenic distillation). The constraints for the catalyst recovery section have not been defined at this time. Forty-six constraints will be used and are listed in Table 3.2-3. The basic structure and the flow chart of the computer program have been worked out. Program development is underway and will be finished prior to the initial startup of the PDU.

3.3 Cold Model Studies

A cold model of the process development unit (PDU) for the Exxon Catalytic Coal Gasification Process was constructed to assist in troubleshooting solids

Table 3.2-2

Process Variables and Their Reliabilities

Reconciled Variable	Unit	Reliability, %
Total Primary Gasifier Steam	lb/hr	5
Char Withdrawn	lb/hr	5
H in Char Carryover	wt.%	5
H in Solids Samples	wt.%	5
H in Coal + Catalyst Feed	wt.%	5
H in Gasifier Starting GMC	wt.%	15
H in Gasifier Ending GMC	wt.%	15
A.S. K_2O in Coal + Catalyst Feed		5
O in Coal + Catalyst Feed	wt.%	15
CO_2 in Total Product Gas	mol%	5
CH_4 in Total Product Gas	mol%	1
CO in Total Product Gas	mol%	20
H_2 in Total Product Gas	mol%	5
N_2 in Total Product Gas	mol%	1
H_2S in Total Product Gas	mol%	20
Carbon in Solids Samples	wt.%	5
A.S. K_2O in Char Carryover	wt.%	5
A.S. K_2O in Solids Samples	wt.%	5
Carbon in Gasifier Starting GMC	wt.%	15
Carbon in Gasifier Ending GMC	wt.%	15
A.S. K_2O in Gasifier Starting GMC	wt.%	15
A.S. K_2O in Gasifier Ending GMC	wt.%	15
ASTM Ash in Char Carryover	wt.%	10
ASTM Ash in Solids Samples	wt.%	10
ASTM Ash in Feed Coal + Catalyst	wt.%	10
C/H Residue in Feed Coal + Catalyst	wt.%	10
C/H Residue in Char Carryover	wt.%	10
ASTM Ash in Gasifier Starting GMC	wt.%	15
ASTM Ash in Gasifier Ending GMC	wt.%	15
C/H Residue in Solids Samples	wt.%	10
C/H Residue in Prim. Gasifier Start GMC	wt.%	15
C/H Residue in Prim. Gasifier Ending GMC	wt.%	15
SO_3-Free ASTM Ash in Feed Coal + Catalyst	wt.%	10
SO_3-Free ASTM Ash in Char Carryover	wt.%	10
SO_3-Free ASTM Ash in Solids Samples	wt.%	10
SO_3-Free ASTM Ash in Starting GMC	wt.%	15
SO_3-Free ASTM Ash in Ending GMC	wt.%	15
SO_3-Free C/H Res. in Feed Coal + Catalyst	wt.%	10
SO_3-Free C/H Res. in Char Carryover	wt.%	10
SO_3-Free C/H Res. in Solids Samples	wt.%	10
SO_3-Free C/H Res. in Starting GMC	wt.%	15
SO_3-Free C/H Res. in Ending GMC	wt.%	15
Coal and Catalyst Feed	lb/hr	5
Char Entrained	lb/hr	5
Carbon in Feed Coal + Catalyst	wt.%	5
Carbon in Char Carryover	wt.%	5
Gasifier Syn Gas Flow Rate	CFH	5
Gasifier Product Gas Flow Rate	CFH	10
Product Gas Knockout Water	lb/hr	5
S in Gasifier Starting GMC	wt.%	20

Table 3.2-2 (Cont.)

Reconciled Variable	Unit	Reliability, %
S in Gasifier Ending GMC	wt.%	20
S in Coal + Catalyst Feed	wt.%	15
S in Solids Sample	wt.%	15
S in Char Carryover	wt.%	15
S in PGKO	wt.%	15
SIO_2 in Ash-SO_3 in Starting GMC	wt.%	20
SIO_2 in Ash-SO_3 in Ending GMC	wt.%	20
SIO_2 in Ash-SO_3 in Coal + Cat Feed	wt.%	15
SIO_2 in Ash-SO_3 in Solid Samples	wt.%	15
SIO_2 in Ash-SO_3 in Char Carryover	wt.%	15
Fe_2O_3 in Ash-SO_3 in Starting GMC	wt.%	20
Fe_2O_3 in Ash-SO_3 in Ending GMC	wt.%	20
Fe_2O_3 in Ash-SO_3 in Coal + Cat Feed	wt.%	15
Fe_2O_3 in Ash-SO_3 in Solids Samples	wt.%	15
Fe_2O_3 in Ash-SO_3 in Char Carryover	wt.%	15
Al_2O_3 in Ash-SO_3 in Starting GMC	wt.%	20
Al_2O_3 in Ash-SO_3 in Ending GMC	wt.%	20
Al_2O_3 in Ash-SO_3 in Coal + Cat Feed	wt.%	15
Al_2O_3 in Ash-SO_3 in Solid Samples	wt.%	15
Al_2O_3 in Ash-SO_3 in Char Carryover	wt.%	15
CaO in Ash-SO_3 in Starting GMC	wt.%	25
CaO in Ash-SO_3 in Ending GMC	wt.%	25
CaO in Ash-SO_3 in Coal + Cat Feed	wt.%	20
CaO in Ash-SO_3 in Solid Samples	wt.%	20
CaO in Ash-SO_3 in Char Carryover	wt.%	20
Chlorine in Starting GMC	wt.%	40
Chlorine in Ending GMC	wt.%	40
Chlorine in Coal + Catalyst feed	wt.%	35
Chlorine in Char Withdrawn	wt.%	35
Chlorine in Entrained Char	wt.%	35
A.S. Na_2O in Gase. Starting GMC	wt.%	25
A.S. Na_2O in Gase. Ending GMC	wt.%	25
A.S. Na_2O in Coal + Catalyst Feed	wt.%	20
A.S. Na_2O in Solid Samples	wt.%	20
A.S. Na_2O in Char Carryover	wt.%	20
MgO in Ash-SO_3 in Starting GMC	wt.%	25
MgO in Ash-SO_3 in Ending GMC	wt.%	25
MgO in Ash-SO_3 in Coal + Cat Feed	wt.%	20
MgO in Ash-SO_3 in Solid Samples	wt.%	20
MgO in Ash-SO_3 in Char Carryover	wt.%	20
TiO_2 in Ash-SO_3 in Starting GMC	wt.%	30
TiO_2 in Ash-SO_3 in Ending GMC	wt.%	30
TiO_2 in Ash-SO_3 in Coal + Cat Feed	wt.%	25
TiO_2 in Ash-SO_3 in Solid Samples	wt.%	25
TiO_2 in Ash-SO_3 in Char Carryover	wt.%	25
P_2O_5 in Ash-SO_3 in Starting GMC	wt.%	40
P_2O_5 in Ash-SO_3 in Ending GMC	wt.%	40
P_2O_5 in Ash-SO_3 in Coal + Cat feed	wt.%	35
P_2O_5 in Ash-SO_3 in Solid Samples	wt.%	35
P_2O_5 in Ash-SO_3 in Char Carryover	wt.%	35
N in Starting GMC	wt.%	15
N in Ending GMC	wt.%	15

Table 3.2-2 (Cont.)

Reconciled Variable	Unit	Reliability, %
N in Coal + Cat Feed	wt.%	10
N in Solid Samples	wt.%	10
N in Char Carryover	wt.%	10
O in Starting GMC	wt.%	20
O in Ending GMC	wt.%	20
O in Solids Samples	wt.%	15
O in Char Carryover	wt.%	15
Starting Differential Pressure (B-A)	psi	5
Starting Differential Pressure (C-B)	psi	5
Starting Differential Pressure (D-C)	psi	5
Starting Differential Pressure (E-D)	psi	5
Starting Differential Pressure (F-E)	psi	5
Starting Differential Pressure (G-F)	psi	5
Starting Differential Pressure (H-G)	psi	5
Starting Differential Pressure (I-H)	psi	5
Starting Differential Pressure (I-A)	psi	5
Ending Differential Pressure (B-A)	psi	5
Ending Differential Pressure (C-B)	psi	5
Ending Differential Pressure (D-C)	psi	5
Ending Differential Pressure (E-D)	psi	5
Ending Differential Pressure (F-E)	psi	5
Ending Differential Pressure (G-F)	psi	5
Ending Differential Pressure (H-G)	psi	5
Ending Differential Pressure (I-H)	psi	5
Ending Differential Pressure (I-A)	psi	5
H_2 in Gasifier Syngas	mol%	5
CO in Gasifier Syngas	mol%	20
NH_3 in PGKO Water	wt.%	20
Acid Gas Flow Rate	CFH	10
Gas from Mol Sieves Flow Rate	CFH	10
SNG Product Gas Flow Rate	CFH	5
Syngas Output Flow Rate	CFH	10
H_2 in Gas from Mol Sieves	mol%	5
H_2 in Substitute Natural Gas		5
H_2 in Syngas Output	mol%	5
CO in Gas from Mol Sieves	mol%	20
CO in Sub. Natural Gas	mol%	20
CO in Syngas Output	mol%	20
CH_4 in Gas from Mol Sieves	mol%	1
CH_4 in Sub. Natural Gas	mol%	1
CH_4 in Syngas Output	mol%	1
CO_2 in Acid Gas	mol%	5
H_2S in Acid Gas	mol%	10
N_2 in Gas from Mole Sieves	mol%	1
N_2 in Syngas Output	mol%	1
CH_4 in Gasifier Syngas	mol%	1
N_2 in Gasifier Syngas	mol%	1
Purge Gas Flow Rate	CFH	10

Table 3.2-3

Constraints Used in Reconciling PDU Operations Data

Constraint No.	Constraint*
1	Gasifier Hydrogen Balance
2	Gasifier Carbon Balance
3	Gasifier Oxygen Balance
4	Gasifier Sulfur Balance
5	Gasifier Nitrogen Balance
6	Gasifier Acid Soluble Potassium Balance
7	Gasifier SO_3-Free Ash Balance
8	Gasifier Ash Balance
9	Gasifier C/H Residue Balance
10	Gasifier SiO_2 Balance
11	Gasifier Fe_2O_3 Balance
12	Gasifier Al_2O_3 Balance
13	Gasifier CaO Balance
14	Gasifier MgO Balance
15	Gasifier TiO_2 Balance
16	Gasifier P_2O_5 Balance
17	Gasifier Acid Soluble Sodium Balance
18	Gasifier Chlorine Balance
19	Sum of All Product Gas Components
20	Sum of All Makeup (or Recycle) Syngas Components
21	Sum of Entrained Char Components
22	Sum of Coal + Catalyst Feed Components
23	Sum of Withdrawn Char Components
24	Sum of Starting Mid Char Components
25	Sum of Ending Mid Char Components
26	Sum of Ash Components in Entrained Char
27	Sum of Ash Components in Coal + Catalyst Feed
28	Sum of Ash Components in Withdrawn Char
29	Sum of Ash Components in Starting Mid Char
30	Sum of Ash Components in Ending Mid Char
31	Sum of Starting Gasifier Differential Pressures/ Total Differential Pressure
32	Sum of Ending Gasifier Differential Pressures/ Total Differential Pressure
33	MEA Absorber H_2 Balance
34	MEA Absorber CO Balance
35	MEA Absorber CH_4 Balance
36	MEA Absorber CO_2 Balance
37	MEA Absorber H_2S Balance
38	MEA Absorber N_2 Balance
39	Sum of Gas Components from Acid Gas Regeneration
40	Sum of Gas Components from Molecular Sieves
41	Cryogenic Fractionator H_2 Balance
42	Cryogenic Fractionator CO Balance
43	Cryogenic Fractionator CH_4 Balance
44	Cryogenic Fractionator N_2 Balance
45	Sum of Gas Components in SNG Product
46	Sum of Gas Components in SNG Gas Output

flow problems that may arise in the PDU. Throughout the startup and operation of the PDU, the transparent Plexiglas model should prove valuable in providing visual understanding of many of the solids flow problem areas which may be encountered. A diagram of the cold model is shown in Figure 3.3-1. The unit consists of a fluidized bed reactor, a cyclone, a fines return system, and solids feeding equipment. A solids withdrawal system may be added in the future.

Most dimensions of the cold model are the same as the PDU except that the model gasifier is 14 feet in height versus the 80 feet of the PDU. This height difference should not affect any of the solids transfer studies planned. The inside diameter of the model reactor is 9-1/2 inches compared to 9-7/8 inches for the pilot unit reactor. The inside diameter of the model dipleg is 2-5/8 inches which is identical to that of the PDU.

Polypropylene powder is the particulate solid used in the model. The particle density of the polypropylene is 44 lb/ft^3 (0.70 g/cc) and the surface volume mean particle diameter is about 230 microns. These properties, as well as the shape factor for polypropylene, are similar to those of gasified char. In addition, the negligible attrition of the polypropylene makes it a particularly good solid substitute for char.

The model has been through preliminary operations during which instruments were calibrated and we became familiar with the general performance of the unit. This work indicated areas that will require detailed experimentation.

Some of the studies planned include:

- cyclone operation for recycling fines
- fines return dipleg operability and control of gas purged to the intersection block
- performance evaluation of solids feeding equipment
- pressure tap design

The first experiments will involve the solids feeding system shown in Figure 3.3-2. The model equipment is similar to that of the PDU except for the length of the vertical line from the lockpot to the 45° feed line and the length of the 3/4-inch feed line, both of which are shorter in the model.

During a solids feeding cycle, the lockpot is first filled from a large lockhopper, then the contents of the lockpot are emptied into the 45° feed line and flow into the reactor. Successful solids feeding will depend on proper valve sequencing, gas purge rate to the system, and purge location. Performance of the equipment will be evaluated with respect to these operating variables and to reactor conditions including bed height and superficial gas velocity. These experiments should help to identify operating conditions necessary for reliable performance of the solids feed system.

The Plexiglas model will be maintained in operating condition during startup of the gasification section of the PDU so that problems encountered may be examined in the model.

FIGURE 3.3-1

COLD MODEL OF GASIFICATION REACTOR

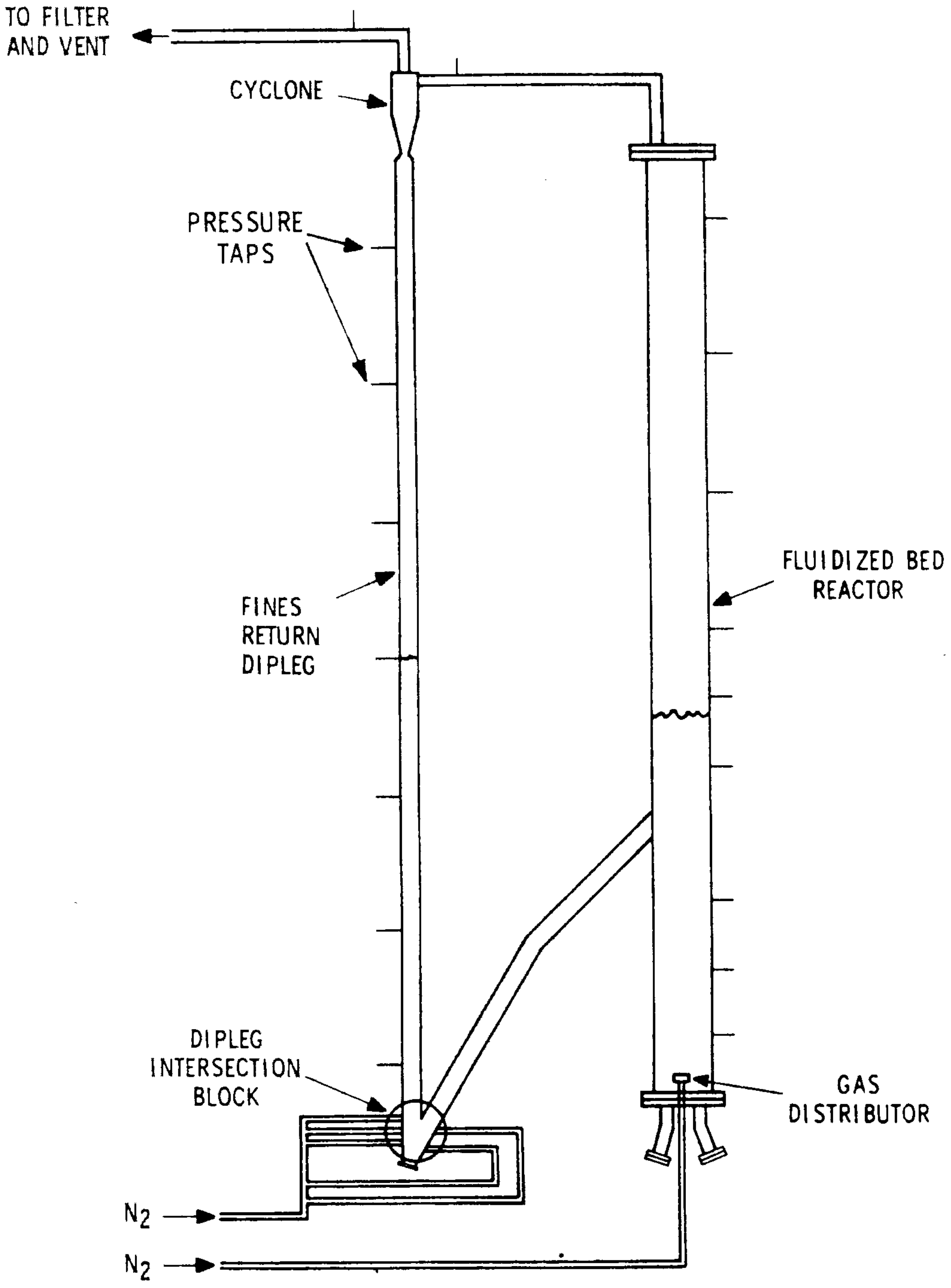
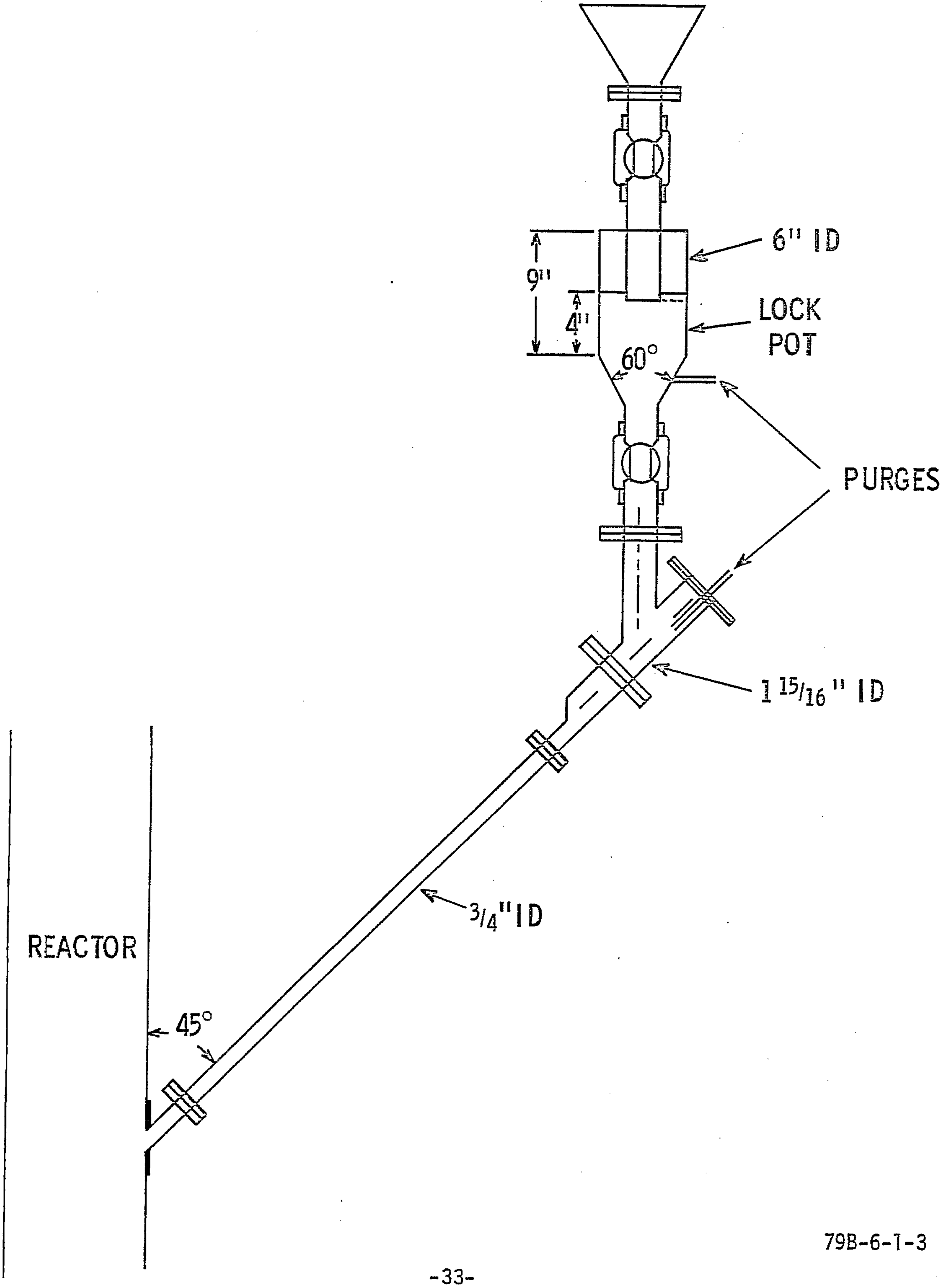


FIGURE 3.3-2

SOLIDS FEED SYSTEM FOR COLD MODEL



79B-6-1-3

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

4.1 Kinetics of Gasification and Devolatilization

The kinetics of char gasification have previously been investigated during the predevelopment phase of catalytic gasification research. Kinetic data was obtained in a bench scale fixed bed reactor at 1300°F with catalyst loadings of 10 and 20 wt.% potassium carbonate on dry Illinois coal. Some data was also obtained at 1200°F. The coal was devolatilized under an inert atmosphere before loading in the fixed bed reactor.

A laboratory program has been initiated 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.

The fixed bed unit has been recommissioned for use in the current program. Numerous instrumentation problems have been identified and corrected. These problems included a leaking gas chromatograph sampling valve system as well as several faulty temperature and pressure indicators.

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 causes of this low total are:

(1) incorrect calibration gas analysis, and

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

Troubleshooting of this problem is currently in progress. Samples of the product gas are being analyzed in a mass spectrometer as well as in other gas chromatograph systems on site to assist in finding the cause of the low gas analyses.

4.2 Catalyst Reactions with Coal and Ash

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 coal. Current operation in this quarter with a new carload of Illinois coal and with potassium hydroxide (KOH) as the catalyst was accompanied by some initial operability difficulties as well as a lower fluidized bed density than was experienced in the predevelopment work. The bench scale work described below identified a solution to the operability problem which was successfully used in the pilot plant.

The particle size distributions of the coals used in the predevelopment work (1977) and in recent work (1978) are shown in Table 4.2-1. The K_2CO_3 catalyzed coal used in predevelopment operations did not contain as many large particles (+20 mesh) as that currently used.

Table 4.2-1

Sieve Analysis of FBG Feed Coals

Coal Description	Mesh Size	Weight % of Sample	% K_2O Acid Soluble
K_2CO_3 treated	+20	5	7.4
FBG feed from 1977	-20 + 60	59	8.0
Predevelopment	-60 + 100	21	9.1
Operation	-100	15	12.5
KOH treated	+20	28	10.0
FBG feed from	-20 + 60	53	11.5
1978 Operation	-60 + 100	15	12.9
	-100	4	13.4

The +20 mesh particles account for 5% of the weight of the predevelopment feed coal and 28% of the recent feed coal. For both coals the catalyst loading of these large particles is low, as shown by the total amount of K^+ present, analyzed for as acid soluble K_2O. Since the active catalyst inhibits swelling and agglomeration during devolatilization, the low catalyst loading on the large +20 mesh particles was thought to account for the poor operability of the FBG when feeding a coal with a relatively large fraction of such particles.

Samples of both the 1977 and 1978 feed coals and the large particles (+20 mesh) only from the 1978 feed were charred in the laboratory at 1300°F and atmospheric pressure under a nitrogen atmosphere. The results are pictured in Figure 4.2-1. The 1977 feed did not agglomerate. The 1978 feed did agglomerate with the agglomerates containing most of the large particles initially present. The sample containing only +20 mesh particles agglomerated severely.

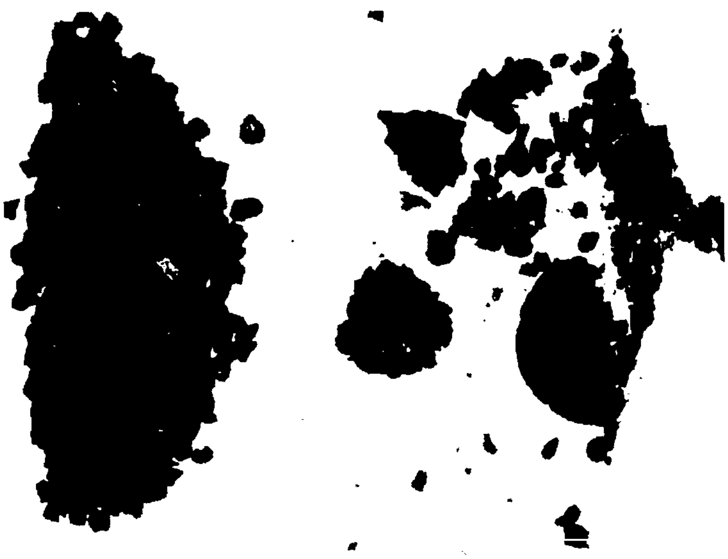
FBG operations were greatly improved when the +20 mesh particles were removed from the feed coal, although the bed density was still lower than what was observed in predevelopment work.

Figure 4.2-1
AGGLOMERATION TESTS

PREDEVELOPMENT FBG

FEED COAL

RECENT FBG FEED COAL



+20 MESH FROM RECENT

FBG FEED COAL

Further bench work is in progress to determine if increased contact time of the coal with the catalyst solution before drying will improve catalyst impregnation on the larger coal particles. Studies to determine the effect of oxidation as well as the change in catalyst from potassium carbonate to potassium hydroxide are planned to see what impact these variables have on char bulk density.

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 sub-tasks:

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 two sub-tasks. Work was also initiated on Engineering Technology Studies. 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

Work continued during the fourth quarter on a revised facilities definition and cost estimate for the offsites sections of the CCG Commercial Plant Study Design. This detailed engineering study design was prepared during the CCG Predevelopment Program. The revised study design will serve as the "base case" for evaluating new data, process improvements, and optimum process conditions under the current Process Development Program.

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, these areas were for the most part 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. A preliminary plant layout was used in specifying piping for utilities distribution and for industrial sewers. A final look at the onsites layout indicated that these piping requirements were probably overestimated. Also, the process wastewater rate used in sizing the wastewater treating facilities was underestimated.

In view of these factors, a revised offsites facilities definition and cost estimate is being prepared to firm up the CCG Study Design in this important area. As a result of this offsites revision, the accuracy of screening studies which use the Study Design as a "base case" will be improved. Most of the changes in this offsites revision are a matter of adjusting equipment sizes to correct for inconsistencies between the initial and final utilities demands and plant layout requirements. However, more extensive changes are being made in two sections. First, in the wastewater treating section, more detailed consideration is being given to water quality and reuse options to better define treatment needs and further reduce plant makeup and effluent water rates. Second, the flue gas desulfurization (FGDS) process is being changed from a regenerative system using sodium carbonate to a once-through system using lime scrubbing. This change will allow integration of the lime scrubbing offsites with other CCG plant offsites. For example, lime receipt for FGDS can be integrated with lime receipt for onsite catalyst recovery, which uses lime as feed to $Ca(OH)_2$ digestion. Common absorbers can be utilized to handle the flue gases from the coal-fired boilers and the coal-fired feed coal dryers and catalyst addition dryers. In addition to these integration advantages, the technology and costs for lime (and limestone) scrubbing are better defined today than for regenerative FGDS.

Equipment lists for the revised CCG Study Design offsites were completed and reviewed early in the fourth quarter. The investment cost estimate was underway by year end after a delay due to other estimating work. Updated utilities lists reflecting the revised offsites facilities are nearing completion. These will serve as part of the basis for updated economics to be developed during the first quarter 1979.

Cryogenic Acid Gas Removal Incentive Study

As discussed in the July-September, 1978 Quarterly Report, 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 will be 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 a simpler, lower investment process configuration than a process using physical absorption and may also have lower energy requirements. It is also believed that there may be benefits for 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 involves the definition of the process flow scheme, detailed heat and material balances, design of the required equipment, and 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.

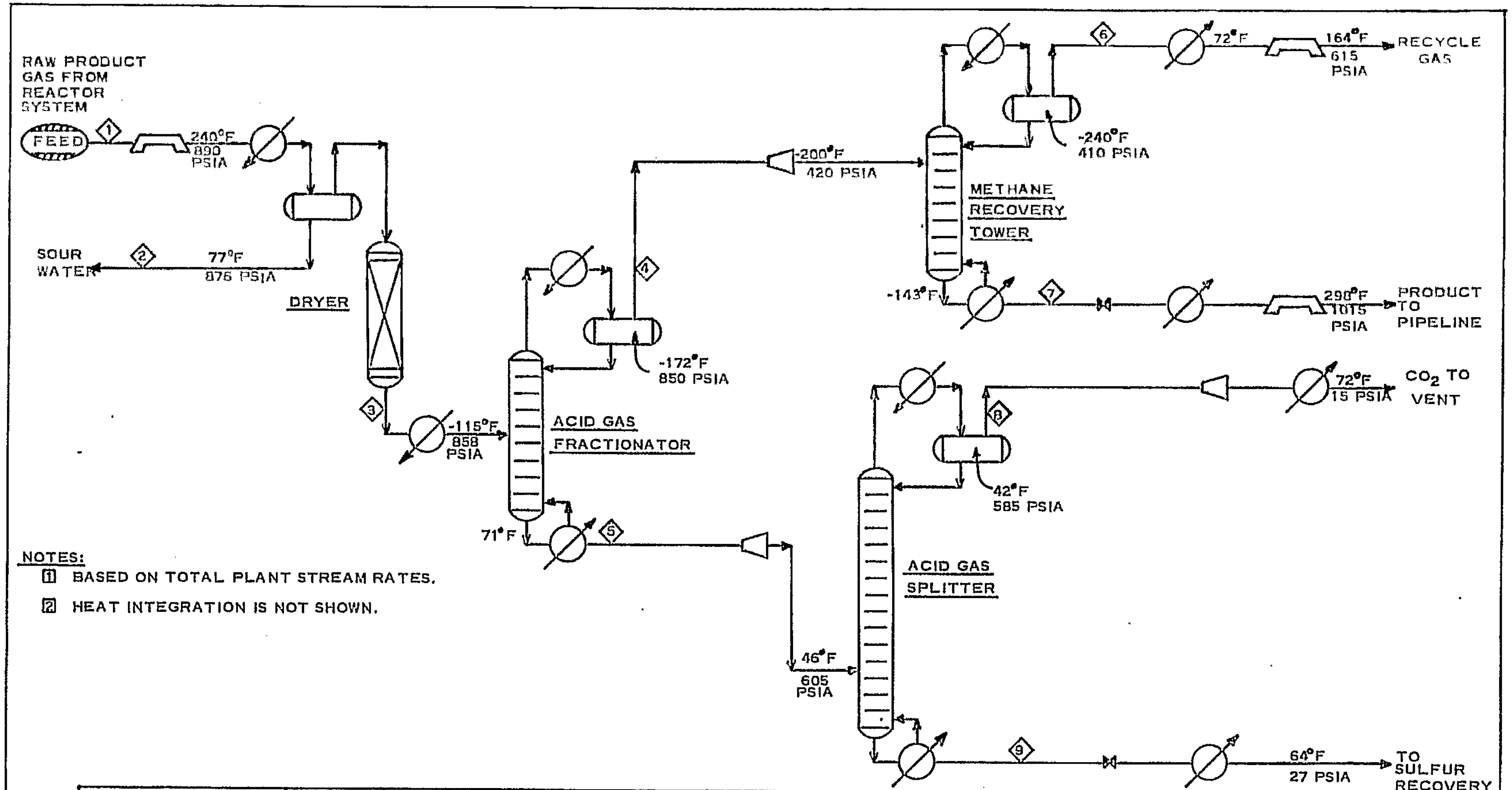
Previous work done under the CCG Predevelopment Contract led to the conclusion that carbon dioxide (CO_2) freeze-out will occur in some part of the acid gas fractionation system throughout the range of possible tower operating conditions. For this study, it has been assumed that the freeze-out problem can be handled in a simple manner within the system. Further work to determine the actual effect of CO_2 freeze-out would then 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). A flowplan showing process operating conditions and major equipment is presented in Figure 5.1-1.

The process bases have now been set for the AGF, AGS and the Methane Recovery Tower (MRT). In the Acid Gas Fractionator an overhead CO_2 concentration of 150 vppm was specified to eliminate the need for molecular sieve adsorption for CO_2 removal upstream of methane recovery. The AGF bottoms specification was set to limit methane losses to 0.1% of the methane fed to the tower. Alternative AGF operating pressures were evaluated. High pressure is desirable to increase tower operating temperatures which minimizes refrigeration requirements and also is believed to minimize the impact of CO_2 freeze-out. Pressures of 1000 psia and 850 psia were evaluated and the lower pressure level provided about a 7% savings in feed/product compression and refrigeration power requirements. AGF feed temperatures between -60°F and -140°F were evaluated and the minimum power requirements were obtained at a feed temperature of about -115°F. This produced a 17% savings versus a feed temperature of -60°F.

The Acid Gas Splitter separates a CO_2 overhead product from an 80% CO_2/20% H_2S bottoms product. After energy and refrigeration recovery, the CO_2 stream is vented to the atmosphere. An overhead H_2S concentration of 10 vppm in the CO_2 vent stream has been specified, consistent with the CCG Study Design base case. The H_2S containing bottoms product is fed to a Claus Plant for sulfur recovery. The AGS operating conditions selected enable use of a heat pump loop with propylene refrigerant for both the condenser and reboiler duties. The specification of a lower CO_2 level in the AGS bottoms would require additional stages and/or reboiler duty and would raise the bottoms temperature. This would significantly increase power requirements in the propylene heat pump loop. Cost savings in the Claus plant would be unlikely to offset these debits.

In the Methane Recovery Tower, a CO/H_2 overhead stream is separated from the CH_4 bottoms product. The tower specifications are consistent with the Study Design base case (0.1% CO in product methane, 10% CH_4 in the



NOTES:

1. BASED ON TOTAL PLANT STREAM RATES.
2. HEAT INTEGRATION IS NOT SHOWN.

STREAM NUMBER	1	2	3	4	5	6	7	8	9
PRESSURE (PSIA)	450	876	866	850	865	410	420	585	606
TEMPERATURE (°F)	100	77	77	-172	72	-240	-143	43	53
FLOWRATES, MPH(1)									
CO	10510		10510	10510		10480	30		
CO_2	21633		21633	13	21620		13	18106	3514
H_2	37671		37671	37671		37671			
H_2O	233	175							
CH_4	35657		35657	35635	22	5750	29885	22	
C_2H_6	5		5		5			4	1
H_2S	878		878		878				878
COS	14		14		14				14
N_2	3585		3585	3585		3585			
TOTAL	110,186	175	109,953	87414	22539	57486	29928	18132	4407

Figure 5.1-1
SIMPLIFIED FLOW PLAN (2)
CRYOGENIC ACID GAS REMOVAL
AND METHANE RECOVERY

recycle gas). The MRT feed from the AGF overhead is cooled and flashed to 420 psia (the same as in the Study Design). Tower feed temperatures from -198°F to -240°F were evaluated, and -200°F was chosen as the basis. At this condition, expanding the bottoms product provides the entire MRT condenser and feed cooling duty. This stream is also used to help cool the AGF feed.

The process basis and heat and material balance have been completed. Work is nearing completion on the heat integration/refrigeration scheme and is expected to be completed in January, 1979. Work will then begin on equipment specification, cost estimating, and economics.

Coal Devolatilization Impact Study

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 stems from the inhibiting effects 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 up 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 also 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 objective of this study is to investigate the impact of uncertainties in the amount of carbon devolatilized on gasifier volume requirements and to investigate the potential risks/benefits of alternative coal feed injection points along the height of the bed. This will provide guidance to laboratory studies which are planned to obtain more definitive estimates of coal devolatilization yields as well as data on residence time required to convert hydrocarbon liquids to light gases. This laboratory work will lead to an improved prediction of devolatilization in the gasifier model.

During December gasifier kinetic model runs were initiated to study the impact of different levels of devolatilization on gasifier volume requirements.

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. Two models are being prepared: a material balance model for the catalyst recovery section, and a material and energy balance model for the CCG reactor system.

Catalyst Recovery Material Balance Model

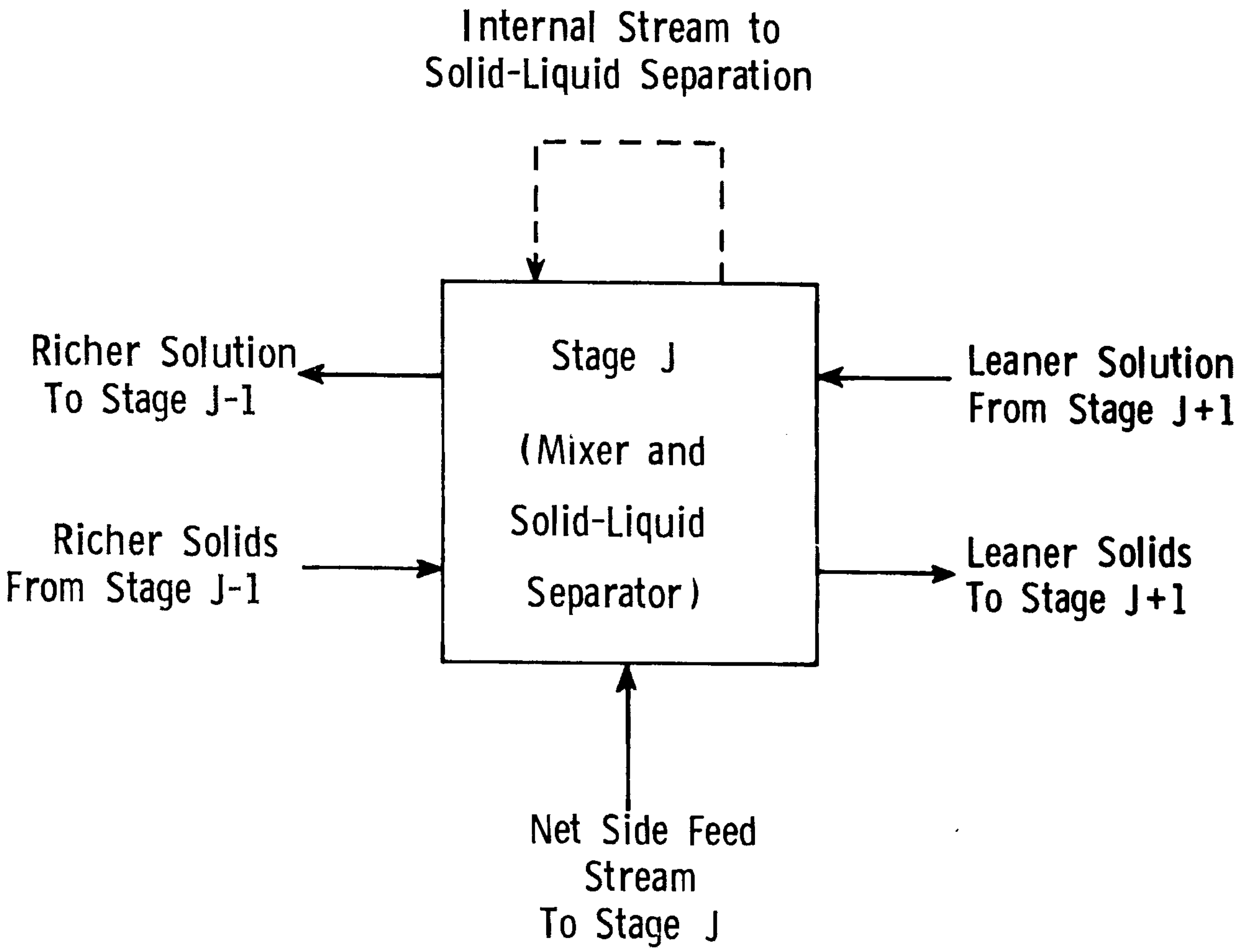
Catalyst recovery as incorporated in the CCG Commercial Plant Study Design involves "digestion" of gasifier char and fines with $Ca(OH)_2$ to solubilize most of the catalytic potassium salts, followed by multi-stage countercurrent leaching with water to remove the soluble catalyst from the gasifier solids and calcium. Material balances for this system have required extensive stage-by-stage hand calculations, as well as some simplifying assumptions. The catalyst recovery material balance model now being developed will perform rigorous stage-by-stage calculation taking into account the solid-liquid separation efficiencies for individual stages. This computer model will be used 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 user of the model must specify the number of stages, but then will be able to choose one of two convergence methods. The user can either specify the recovered solution concentration and have the model calculate overall catalyst recovery, or he can specify the overall recovery and have the model calculate recovered solution concentration. Varying recovered solution concentration is equivalent to varying wash water rate. Other

Figure 5.2-1

CATALYST RECOVERY MATERIAL BALANCE MODEL:
STAGE CONFIGURATION AND STREAMS



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.

During the final quarter of 1978, the catalyst recovery model was programmed and preliminary documentation was prepared. The model is now in the debugging phase and will be tested for validity in the first quarter of 1979. Part of the initial testing will include using the program to duplicate the results of the CCG Study Design.

CCG Reactor System Material and Energy Balance Model

A second systems modeling effort began in December to develop updated material and energy balance tools for the CCG reactor system. The catalytic gasification reactors and the associated recycle gas loop and preheat furnaces are key parts of the commercial process flowsheet. Five of the eight process blocks in the CCG Commercial Plant Study Design are directly involved. They include the Reactor, the Product Gas Cooling and Scrubbing, the Acid Gas Removal, the Methane Recovery, and the Refrigeration sections. These sections carry out the gasification step and the cleanup and separation of the raw gasifier product gases.

A "first pass" reactor system material and energy balance model was used in preparing the CCG Commercial Plant Study Design. Although this model is accurate and proved satisfactory for use in the Study Design effort, the gasifier material balance routine is not specifically intended for coal gasification and the energy balance calculations are complex and cumbersome. In applying this first-pass model, extensive hand calculations are necessary to set up "pseudo streams" for the material balance and develop solids enthalpy terms for the overall energy balance. Improved reactor system material and energy balance techniques and computational tools are desirable to allow the laboratory guidance studies planned under the current program to be carried out efficiently and consistently.

The updated CCG reactor system model will be incorporated within the framework provided by Exxon's proprietary process network simulation program, known as "COPE". The overall model is expected to consist of the following elements:

- A block to model the CCG gasifier itself, feeding coal and catalyst and producing a product gas at specified shift and methanation equilibria along with spent solids (char, ash, and catalyst). This block will also calculate enthalpies for the solids streams.

- A block to carry out the gasifier energy balance.

- An optional block 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.

- An overall COPE process simulation to join together the above blocks, model the material balances for the cleanup and separations steps downstream of the gasifier, and converge the overall material balance and gasifier/preheat furnace energy balances.

In December, a preliminary outline was developed defining the proposed capabilities of the updated CCG reactor system material and energy balance model. Definitive plans for the updated model and the individual model blocks will be developed in January. Work on this model is expected to be completed in the second quarter of 1979.

5.3 Engineering Technology Studies

As part of the CCG Process Development Program, a coordinated set of engineering technology programs will be conducted to develop fundamental process and equipment technology to support the overall laboratory and engineering process development effort. Work began on several of these programs during the fourth quarter of 1978, as described below. Additional programs will be initiated 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. During the fourth quarter of 1978, two steps were taken: (1) the broad outlines were defined for an in-situ materials evaluation program to be conducted in the CCG PDU at Baytown, Texas; and (2) detailed design was initiated for individual test components.

The PDU materials evaluation program will consist of five elements:

- Corrosion racks
- Corrosion probes
- Non-destructive testing (NDT) and inspection
- Failure analysis
- Stream sampling

The immediate objective of the program is to have corrosion racks and probes ready for corrosion testing and monitoring during early PDU operations.

Table 5.3-1 presents a summary of pertinent data on corrosion racks and probes. Locations of eight corrosion racks and two corrosion probe test sites have been selected. The detailed designs of corrosion racks and specimens has been completed. The information needed to purchase, install, and operate the two corrosion probes has been developed. In addition, the details are being provided for nozzle/coupling requirements for proper installation of the eight corrosion racks. Fabrication of these corrosion racks has been initiated at Florham Park. Completion and shipment of the racks to Baytown is targeted for early February.

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

Work has been started in defining NDT/inspection, failure analysis, and stream sampling requirements. A list of recommended inspection items and points will be prepared. This facet of the program is important because size and accessability limitations will require much reliance on NDT/inspection techniques.

Failure analyses will be used to improve the understanding of materials behavior in the PDU. Potential areas where "preventive analysis" will be performed on a selected basis will be identified. In these areas, samples will be systematically removed from equipment to establish if any failure mechanism has been initiated. In areas such as the gasifier overhead tubing and the preheater gas tubing, this approach will forewarn of problems and potential failures.

Interpretation of corrosion and inspection results necessitates a thorough knowledge of stream constituents. Therefore stream sampling requirements are being developed to aid in understanding chemical reactions which could produce corrosion and/or plugging problems. The inspection and stream sampling programs are expected to be finalized in the first quarter of 1979.

Other Engineering Technology Studies

Initial planning work on four other engineering technology studies was in progress by year end 1978:

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

- Slurry Rheology and Solid-Liquid Separations for Catalyst Recovery

  The key 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.

- Environmental Control: Water and Solids Effluents

  The objective of this program and the Environmental Control: Atmospheric Emissions program is to generate the data needed for a quantitative assessment of the environmental impact of the CCG process. The focus of this first program will be to characterize wastewaters, spent solids, and solids slurries produced in the CCG PDU.

- Environmental Control: Atmospheric Emissions

  This second environmental control program will be directed toward identification of potential atmospheric emissions sources and the quantification of these emissions through testing in the PDU.

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