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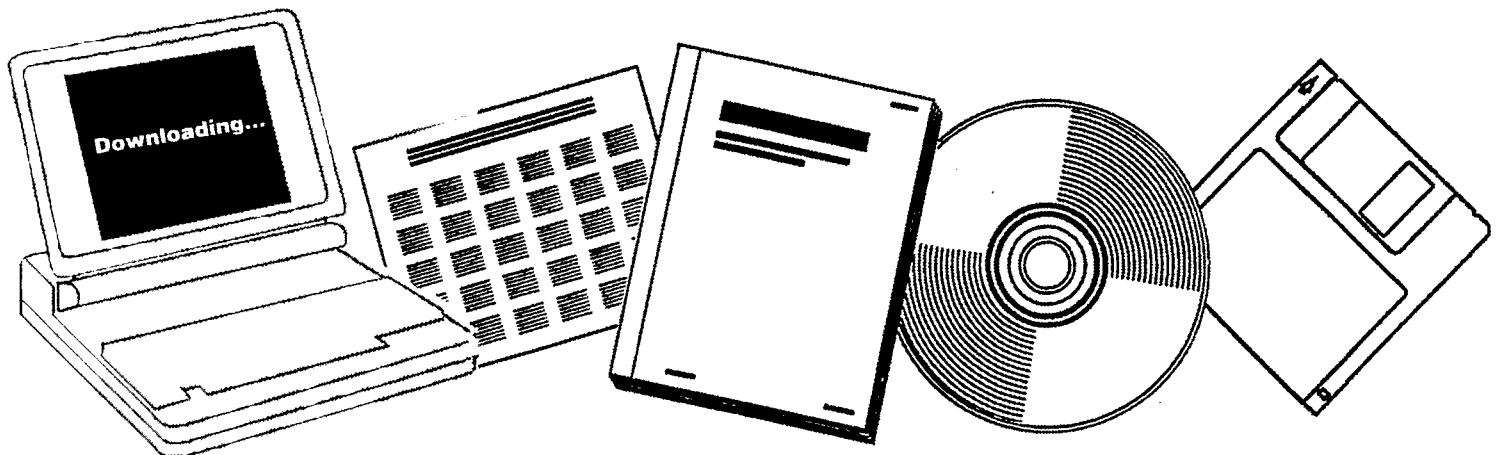
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**EXXON CATALYTIC COAL GASIFICATION PROCESS:
PREDEVELOPMENT PROGRAM. MONTHLY REPORT,
JULY 1977**

**EXXON RESEARCH AND ENGINEERING CO.
BAYTOWN, TX**

26 AUG 1977



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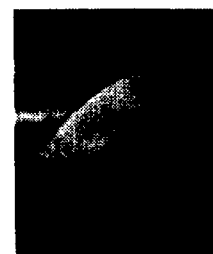
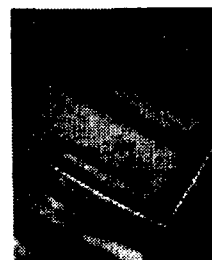
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EXXON CATALYTIC COAL GASIFICATION PROCESS
PREDEVELOPMENT PROGRAM

Monthly Report for the Period
July, 1977

T. Kalina - Project Manager

EXXON RESEARCH AND ENGINEERING COMPANY
Baytown, Texas 77520

August 26, 1977

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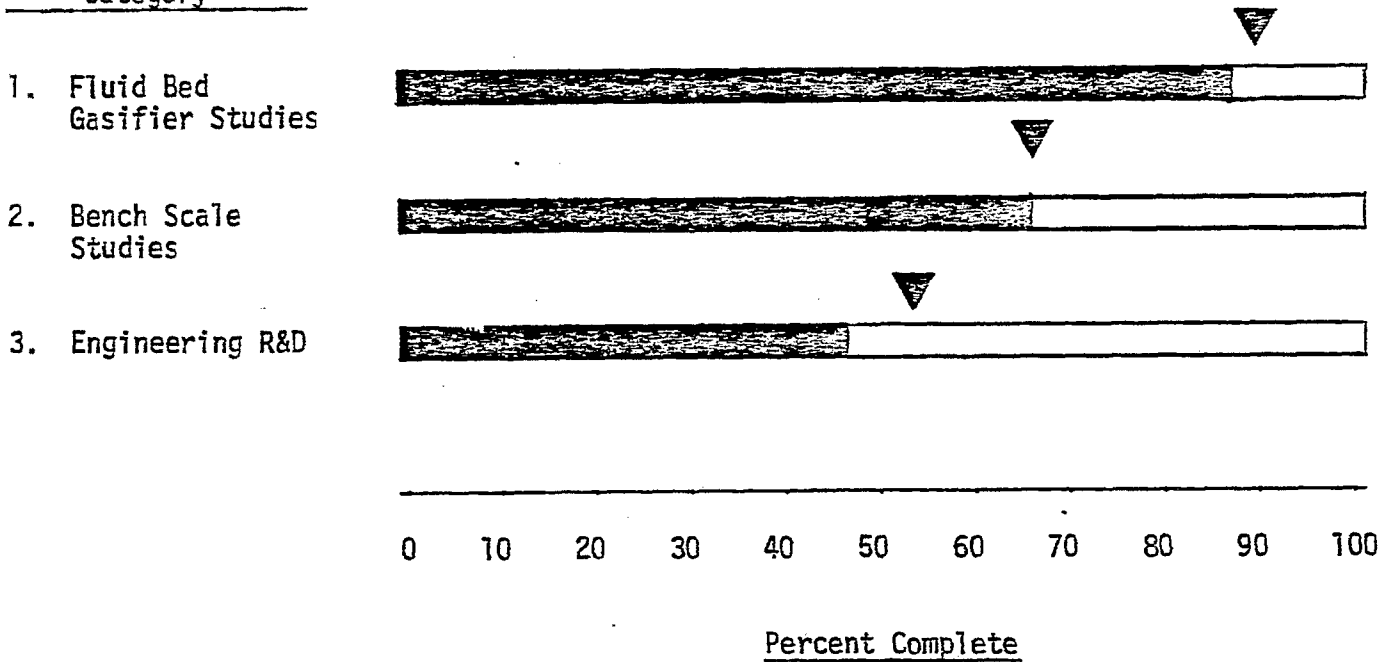
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SUMMARY OF PROGRESS THROUGH July 31, 1977 FOR PREDEVELOPMENT
WORK ON EXXON CATALYTIC COAL GASIFICATION PROCESS

Technical Reporting
Category



Legend

Shaded area = Percent of activity actually completed

▼ = Percent of activity scheduled for completion

Abstract

During the month of July, operation of the catalyst recovery unit (CRU) continued and water soluble potassium was recovered and recycled to the catalyst addition unit (CAU). The recovery of water soluble potassium increased to 94.0%. Approximately 324 hours of material balanced operations were logged during the month with excellent closures. The longest continuous run, which began in June and extended into July, lasted over 290 hours. Carbon and steam conversions remained essentially the same as for the June material balance periods. Construction of a digestion unit for secondary catalyst recovery was completed during July.

Bench scale studies of catalyst recovery via water washing were continued in July. Data were obtained which confirm the discovery in June that exposure of char to air adversely affects catalyst recovery. The effect of residence time on catalyst recovery by water-washing was also investigated during July. Over the range of conditions investigated, recovery of water-soluble catalyst is independent of residence time.

During July, work continued on the use of $\text{Ca}(\text{OH})_2$ digestion to recover water-insoluble potassium from gasifier ash/char residue. Recoveries of water-insoluble potassium from unwashed char decreased with a decrease in liquid/char ratio. A simulation of digestion using solutions of recovered catalyst was conducted. Recoveries from these recycle runs are in the same range as recoveries obtained in runs made under the same conditions with fresh 15% KOH solution.

Work has continued on the development of the process basis for the Catalytic Gasification Study Design. The study design will reflect the current concept of a commercial catalytic gasification plant producing approximately 250 MSCF/SD from Illinois coal. Preparation of this study design will involve the major share of the engineering effort under the Predevelopment contract during the latter part of 1977. A screening quality evaluation of the Engel-Precht process for manufacturing potassium catalyst for the catalytic gasification process has been completed. This screening study indicates that the total estimated cost of KHCO_3 catalyst from an Engel-Precht plant is about 94% of the cost of purchased KOH. Based on "cash flow" scoping studies reported in the June Monthly Report, catalyst recovery via hydrothermal treatment with $\text{Ca}(\text{OH})_2$ offers the potential for substantial savings relative to purchased KOH at the market price. The "minimum cost" of the incremental potassium recovered via this route was estimated to be 36-54% of the cost of purchased KOH, depending on $\text{Ca}(\text{OH})_2$ requirement.

DISCUSSION

FLUID BED GASIFIER STUDIES (REPORTING CATEGORY 1)

Catalyst Recovery Unit Operation

During the month of July, operation of the catalyst recovery unit (CRU) continued and water soluble potassium was recovered and recycled to the catalyst addition unit (CAU). As reported in the June Monthly Report, operations conducted during the period 6/27 through 7/8 recovered 90.2 percent of the water soluble catalyst. The results of this operation are summarized in Figure I.

It was subsequently found (see discussion under Reporting Category 2 of this report) that the procedures used to protect the char from air exposure were inadequate and that air exposure reduced recovery. Hence, char handling procedures were instituted. The N₂ blanketed can of char taken off the gasifier was placed in N₂ filled bags to further protect the char. As an apparent consequence, the recovery of water soluble potassium increased to 94.0% for this second period of operation. Figure 2 gives details of the overall balance for this period.

As reported in June, lime was added to the feed water because there was evidence that some potassium bound to the char at ion exchange sites could be released by calcium ions. When CaO was removed from the water feed during the third operation period, potassium recovery dropped to 89.6% (see Figure 3). This result is consistent with the hypothesis that potassium is tied to char by ion exchange, and that calcium aids in its removal.

In summary, these results represent an improvement over the June operation. Increased water rate, introduction of lime into the feed water, and improved procedures for blanketing the char were the factors which apparently account for this improvement.

FBG Operations

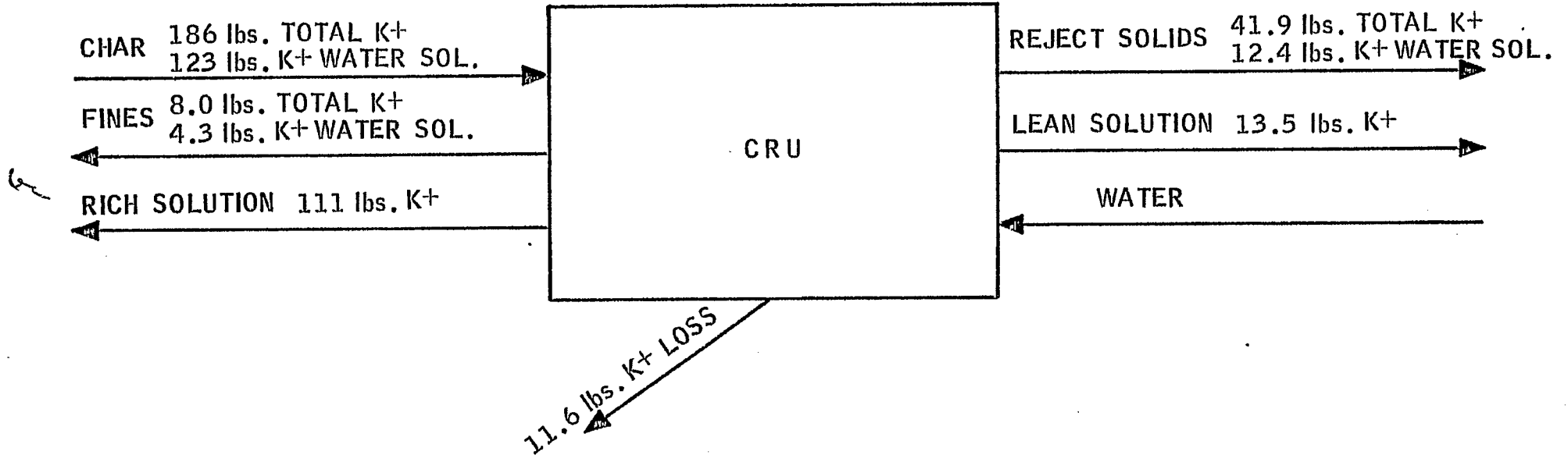
In July FBG operations continued with coal feed impregnated with recycle catalyst solution. Near the end of the month these operations were terminated. During the remainder of the month the unit was again run using coal feed impregnated with fresh potassium carbonate solution. Higher coal and steam feed flow rates were employed in order to produce an inventory of gasified char. This char is to be used for continuing work on various catalyst recovery techniques.

Approximately 324 hours of material balanced operations were logged during the month with excellent closures. The longest continuous run, which began in June and extended into July, lasted over 290 hours. The unit service factor for the month was 70 percent. This is not as high as

FIGURE 1

CATALYST RECOVERY OPERATIONS

PERIOD 1 - OPERATION WITH LIME AND UNBLANKETED CHAR



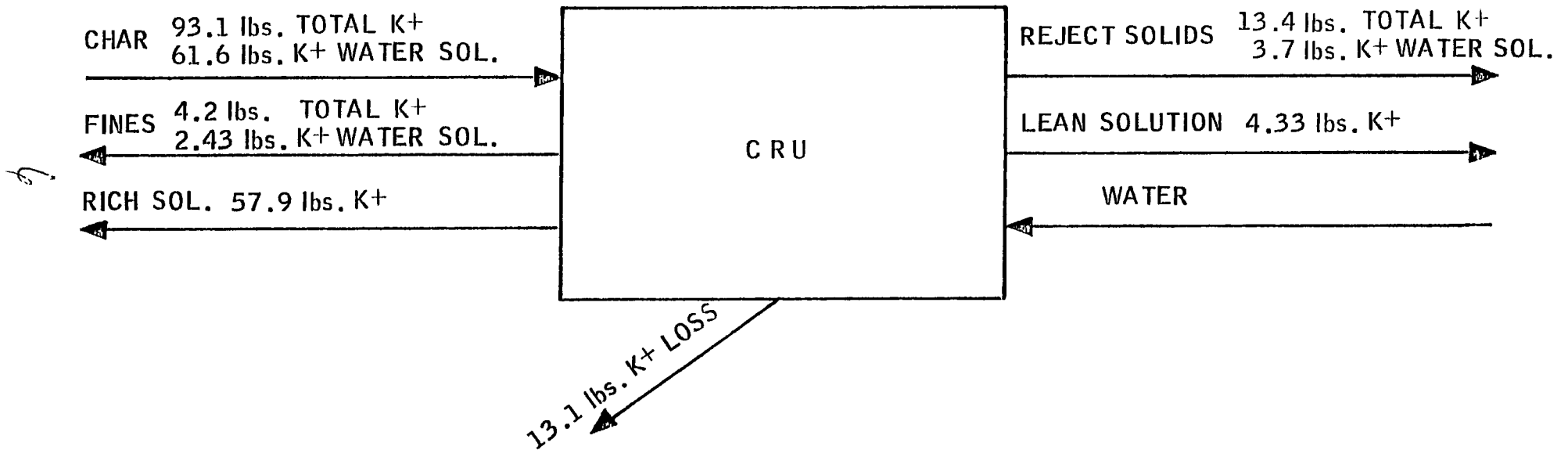
RECOVERY WATER SOLUBLE K+ = $111/123 \times 100\% = 90.2\%$

K+ CLOSURE OUT/IN $\times 100\% = 93.8\%$

FIGURE 2

CATALYST RECOVERY OPERATIONS

PERIOD 2 - OPERATION WITH LIME AND BLANKETED CHAR



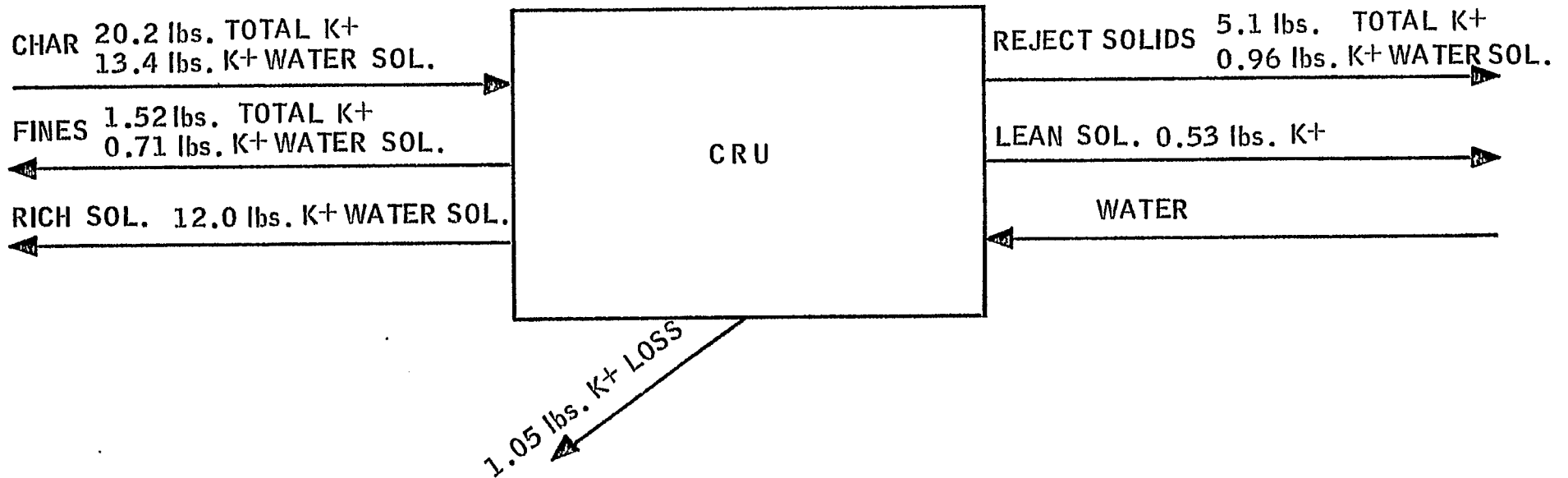
RECOVERY WATER SOLUBLE K+ = $57.9 / 61.6 \times 100\% = 94.0\%$

K+ CLOSURE OUT/IN $\times 100\% = 85.9\%$

FIGURE 3

CATALYST RECOVERY OPERATIONS

PERIOD 3 - OPERATION WITHOUT LIME AND BLANKETED CHAR



RECOVERY WATER SOLUBLE K+ = $12/13.4 \times 100\% = 89.6\%$

K+ CLOSURE OUT/IN = 94.8%

last month's recycle operations primarily because several minor mechanical and computer problems were encountered. Data from six material balance periods are summarized in Table I. Periods 41 thru 45 are for recycle operations. Carbon and steam conversions remained essentially the same as those for the June material balance periods. Material balance period 46 utilized fresh catalyst solution and high feed flow rates.

Catalyst Recovery by Char Digestion

Construction of a digestion unit for secondary catalyst recovery was completed during July. The digester, a pressure vessel with a capacity of approximately 100 gallons, will operate at 400°F psi and 250 psi. It is equipped with an impeller for agitation, and an internal steam/water coil for rapid heatup and cool down during startup and shutdown. Electrical resistance heaters are used to supply additional heat. The slurry product from the digester is fed to a batch vacuum filter where liquids are separated from the solids. The resulting solution and solids will be weighed and analyzed for their potassium content. Bench scale work previously reported has shown that the water insoluble potassium catalyst on the char can be recovered by reaction with a $\text{Ca}(\text{OH})_2$. A series of runs is being planned to determine the effects on a small pilot scale of processing variables such as temperature, calcium level, solution/char ratio, and recycle solution composition. Initial results from this work are expected to be available in August.

Sour Water Analysis

During June and July additional samples of sour water from the Fluid Bed Gasifier (FBG) were collected and analyzed for several contaminants, including ammonia, carbon dioxide, chlorides, cyanides, phenols, sulfides, and total organic carbon. The results of these analyses are shown in Table II. Three of the samples are associated with designated material balance periods. The other samples were collected during periods of good operation between designated material balance periods.

Though the FBG was operating well, problems did develop with the wet gas scrubber during this period. The product gas passes first through bag filters and then through the wet gas scrubber. Early in June the wrappings on the bag filters were changed and it was subsequently learned that spaces in the wrappings had allowed gasifier fines to enter the scrubber and mix with the sour water.

Carbon fines from other gasification processes have been shown to act as activated carbon by absorbing organic contaminants from wastewater. It is possible that the fines in the scrubber may have reduced the levels of some contaminants in the scrubber water. The phenol level was very low during this time period, and the total organic carbon level in the filtered scrubber water apparently decreased. However, the chemical oxygen demand remained relatively constant.

TABLE II
FLUID BED GASIFIER SCRUBBER WATER ANALYSIS

Concentration (ppm)

<u>Material Balance Period</u>	<u>pH</u>	<u>Ammonia</u>	<u>Alkalinity As Na₂CO₃</u>	<u>Carbon Dioxide</u>	<u>Chlorides</u>	<u>Free Cyanides</u>	<u>Thio- Cyanate</u>	<u>Phenols</u>	<u>Sulfides</u>	<u>Thio- Sulfate</u>	<u>Total Organic Carbon</u>	<u>Chemical Oxygen Demand</u>
33	8.2	14,900	24,800	40,900	17	2	4	0.4	271	3	6780	950
35	8.4	16,900	43,100	47,200	<1	1	1	0.2	54	38	40	1130
37	8.7	16,800	37,600	17,700	96	1	3	0.6	61	31	194	1665
44/45*	8.5	12,900	27,100	43,600	178	1	5	1.9	120	18	52	1474
45/46*	8.2	10,200	31,700	55,700	10	1	2	1.1	57	42	36	1281

* In these cases sampling was done during steady operation between the material balance periods indicated.

Several of these samples were extracted with chloroform and the organic residue analyzed by mass spectroscopy. The residues contained a greater percentage of inert material (probably due to the fines present), but were otherwise similar to previous samples in that they contained mostly compounds with 2 or 3 rings.

TABLE II
 FLUID BED GASIFIER SCRUBBER WATER ANALYSIS

Concentration (ppm)

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45/46*	8.2	10,200	31,700	55,700	10	1	2	1.1	57	42	36	1281

* In these cases sampling was done during steady operation between the material balance periods indicated.

BENCH SCALE STUDIES (REPORTING CATEGORY 2)

Catalyst Recovery Via Water Washing

Bench scale studies of catalyst recovery via water washing were continued in July. Data were obtained which confirm the discovery in June that exposure of char to air adversely affects catalyst recovery. Also, as an input to the Catalytic Gasification Study Design, the effect of residence time on water-soluble catalyst recovery was investigated.

Figure 4 shows all the data obtained to date on the effect of air exposure on potassium recovery, including the data reported in June. These data were obtained by serial washing of FBG char with portions of distilled water. Fresh distilled water was used for each wash (i.e., a cross-current extraction), and the water/char ratio was 3/1 on a weight basis. The newly obtained data for a 24 hour air exposure are most significant since both the location of the recovery curve and its shape are intermediate between the 30 minute and the 3 month curves. This effectively confirms the observation that air exposure reduces recovery, and indicates that the reaction responsible for reduced recovery does not rapidly reach completion, but continues to occur for a period of several hours after initial exposure, if not longer. Experiments to determine the mechanism responsible for these reduced recoveries will be initiated in August.

The effect of residence time on catalyst recovery by water-washing was also investigated during July. Again, a cross-current extraction was used, with a 4/1 water/char ratio being used for the first wash, and ratios of 3/1 being used for subsequent washes. Inerted char was used in all runs, and the residence times were varied from 5 minutes to 2 hours. The results of these experiments are shown in Figure 5. Over the range of conditions investigated, recovery of water-soluble catalyst is independent of residence time. This result implies that mixing vessels in the catalyst recovery section of the Catalytic Gasification Study Design can be sized to provide good operability, without having to provide extended residence time.

Recovery of Water-Insoluble Catalyst

During July work continued on the use of $\text{Ca}(\text{OH})_2$ digestion to recover water-insoluble potassium from gasifier ash/char residue. As indicated last month, a commercial countercurrent catalyst recovery system would use a $\text{KOH}/\text{K}_2\text{CO}_3$ solution from downstream washing steps as the source of liquid for the digestion step. Therefore, most of the experiments conducted this month employed a 15% KOH solution. Furthermore, since the ultimate recovery of total potassium present is the critical parameter in determining the required makeup, experimental results will be reported as percent of total potassium removed by the digestion. The ash/char

Figure 4

AIR EXPOSURE REDUCES RECOVERY

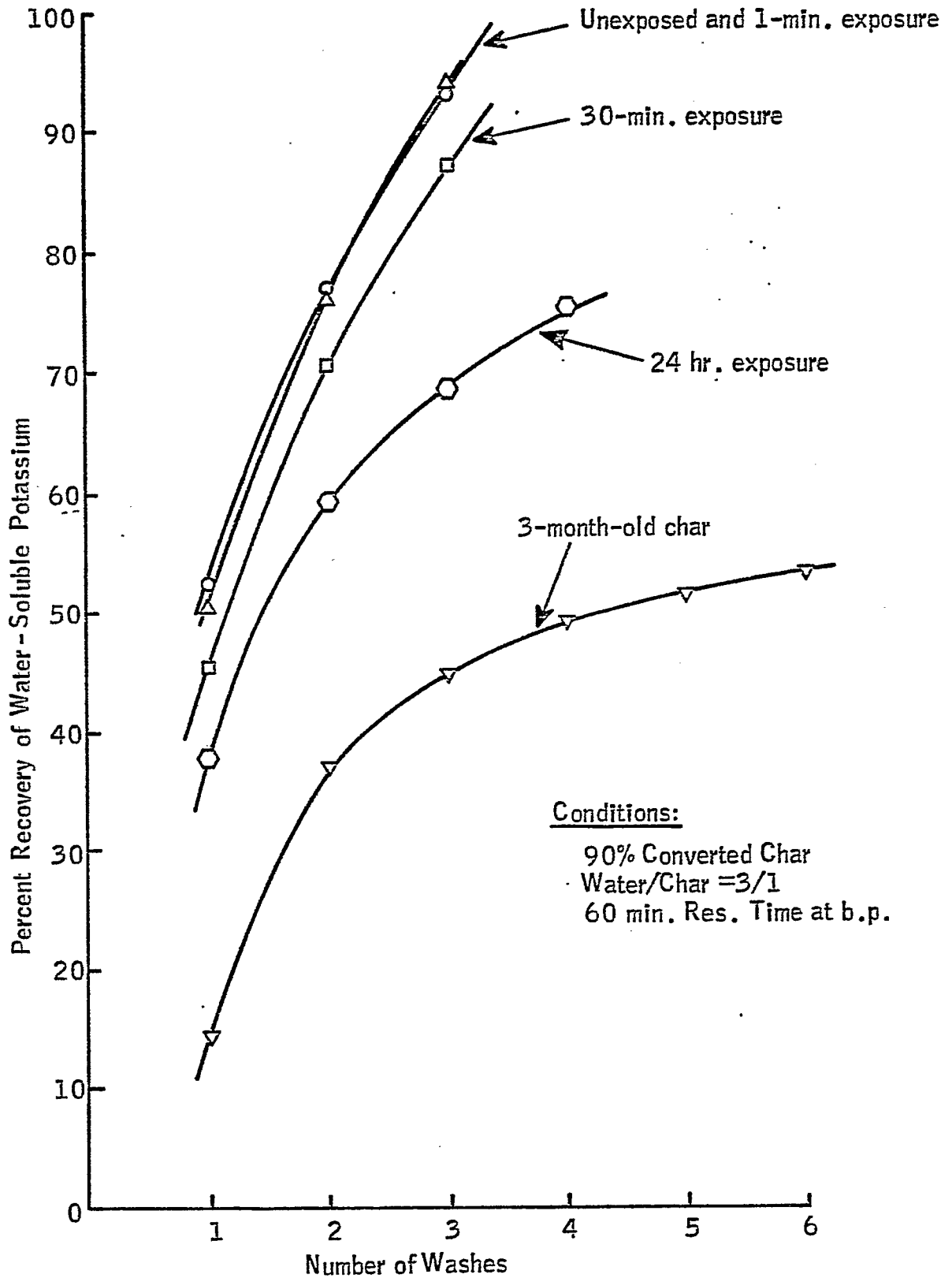
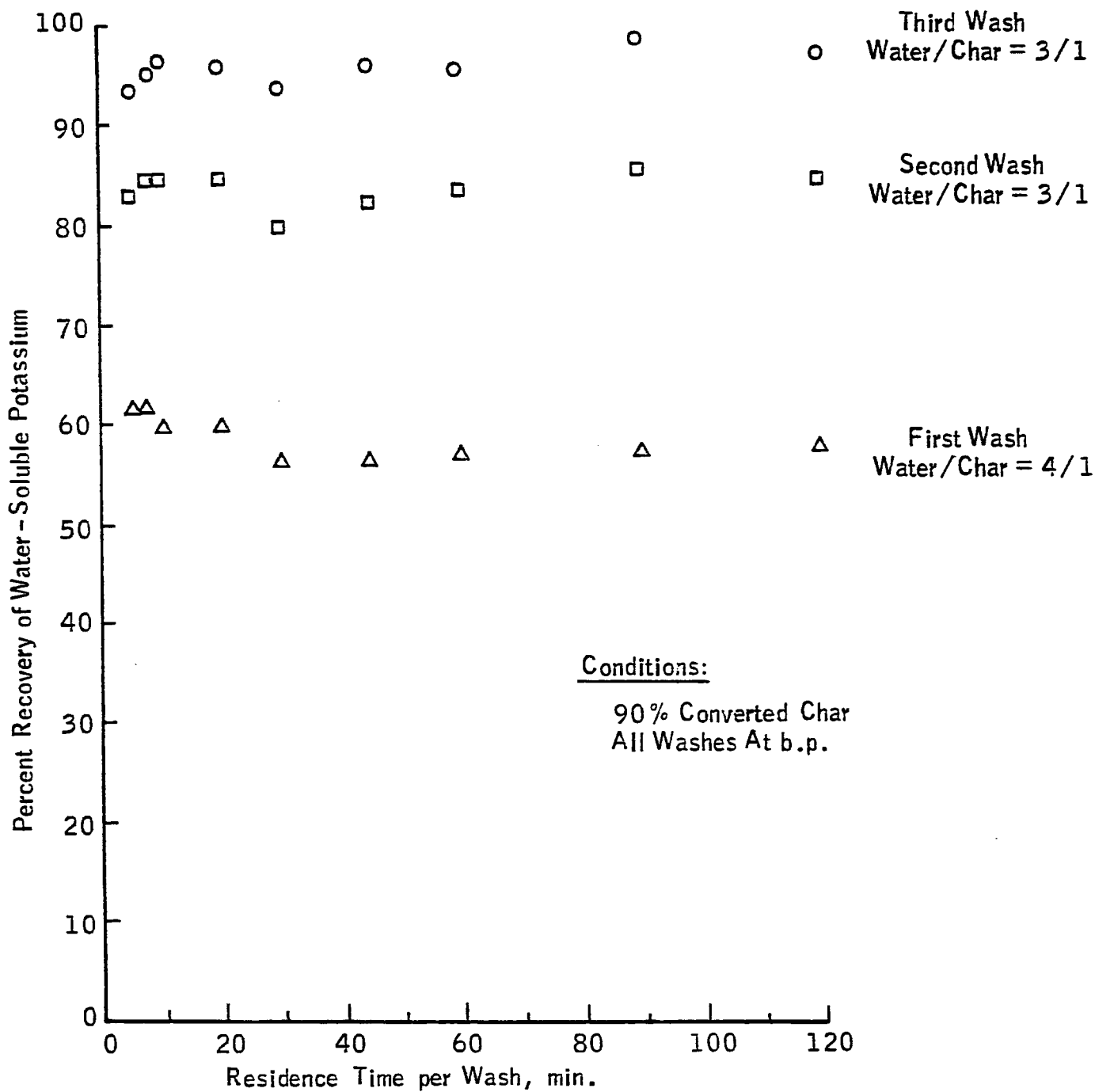


Figure 5

EFFECT OF CONTACT TIME ON RECOVERY WITH
LOW WATER/CHAR RATIO



residue used in all this work was from FBG gasification of Illinois coal treated with 15% K_2CO_3 .

In most of the experiments completed thus far, a liquid:char weight ratio of 10:1 was used. However, in a commercial catalyst recovery system there is an incentive to operate the digestion step with a more concentrated slurry. Therefore, several bench scale runs were made with a 15% KOH solution using a liquid:char ratio of 4:1.

The runs were made using various amounts of $Ca(OH)_2$ at 400°F for four hours. Recoveries of water-insoluble potassium from unwashed char decreased with a decrease in liquid:char ratio as shown in Figure 6. The calcium to potassium mole ratio shown on the X-axis is based on the amount of calcium loaded and the total potassium present on the undigested char fed. The removals/recoveries are based on analyses of the char before and after digestion. The recoveries from these runs may be compared to the recoveries for the runs made with a 15% KOH solution using a liquid:char ratio of 10:1 which were reported in June. These data are also shown on Figure 6 for comparison. Several factors could contribute to this decrease in recoveries with a decrease in liquid:char ratio, such as solubility limits or less thorough mixing.

A simulation of digestion using solutions with the actual composition expected from downstream washing steps was conducted. Portions of the solutions resulting from runs made with 15% KOH were concentrated and used instead of 15% KOH for the first recycle runs. The recycle solutions were made up to contain an amount of potassium equivalent to the amount which would be found in an equal volume of 15% KOH. The solutions resulting from runs were subsequently reused through two or three cycles as shown by the flow diagram presented in Figure 7. The data from duplicate runs conducted with 4:1 liquid:char ratios are shown below:

	<u>Ca/KT</u>	<u>Cycle No</u>	<u>% Removal Total K</u>
Sample 1	0.62	0	89%
	0.68	1	88%
	0.62	2	85%
	0.62	3	85%
Sample 2	0.61	0	88%
	0.65	1	87%
	0.61	2	85%

Recoveries from these recycle runs are in the same range as recoveries obtained in runs made under the same conditions with fresh 15% KOH solution. These results indicate that the results obtained with fresh 15% KOH solution are representative of what should be achieved with a recycle solution in a commercial countercurrent process.

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FIGURE 6
EFFECT OF SLURRY CONCENTRATION
ON POTASSIUM RECOVERY

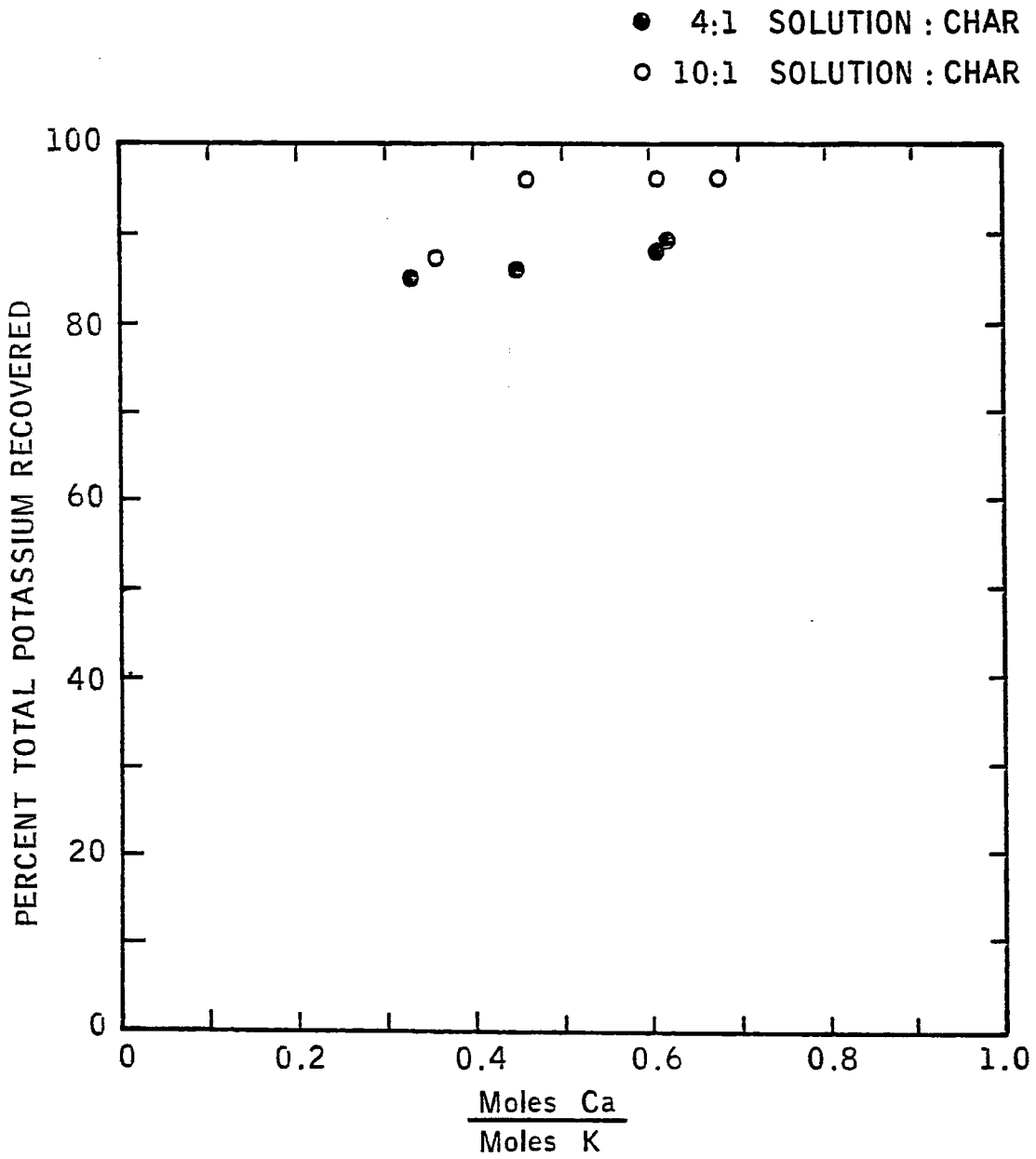
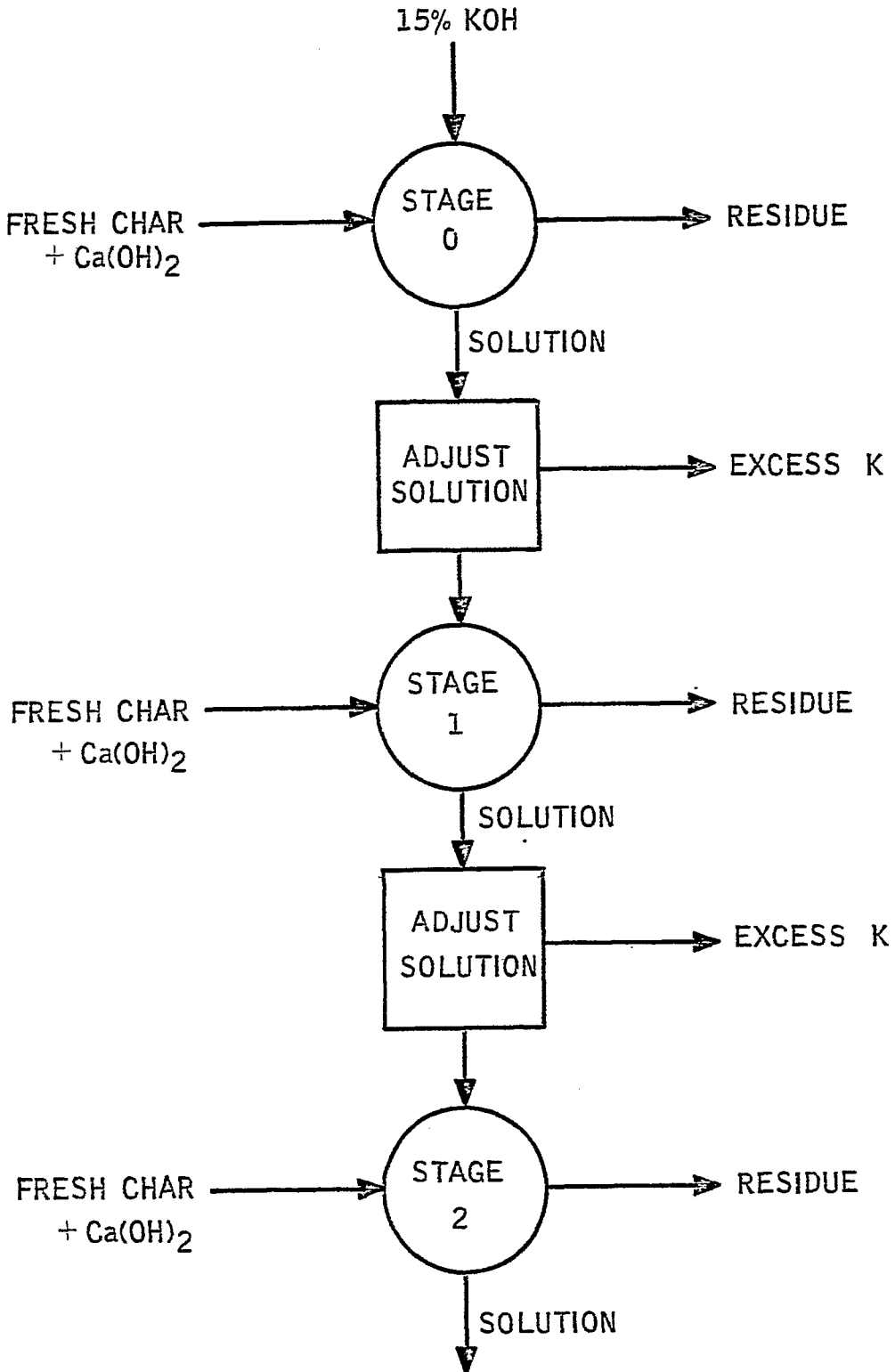


FIGURE 7

SIMULATION OF DIGESTION WITH DOWNSTREAM
SOLUTION COMPOSITIONS



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ENGINEERING RESEARCH AND DEVELOPMENT (REPORTING CATEGORY 3)

Catalytic Gasification Study Design

Work has continued on the development of the process basis for the Catalytic Gasification Study Design. The study design will reflect the current concept of a commercial catalytic gasification plant producing approximately 250 MSCF/SD of SNG from Illinois coal. Estimates will be made of both investment and operating costs. Preparation of this study design will involve the major share of the engineering effort under the Predevelopment contract during the latter part of 1977.

Efforts have focused on beginning studies needed to set the process bases for the catalytic gasifier and recycle synthesis gas loop. Gasifier pressure for the new study design will be 500 psia, the same pressure as in the previous "Base Case." Drawing upon the results of engineering studies to evaluate the commercial impacts of alternative operating conditions (described in the April, 1977 Monthly Report), the gasifier temperature will be reduced from 1300°F to 1275°F to take partial advantage of savings indicated for lower temperature operation. Although still lower temperatures could lead to additional savings, designing for a temperature below 1275°F at this time is judged to involve too great an extrapolation from the current data base obtained in Fluid Bed Gasifier (FBG) operations. Most FBG material balance periods have been carried out at about 1300°F.

In order to provide key inputs to the gasifier heat balance, studies by the Exxon Engineering Technology Department have been initiated in the areas of coal feeding methods and gasifier lining design. Data from FBG material balance periods are under review to develop bases for gasifier solids carryover and gasifier char withdrawal rates and properties. These bases will be used in designing solids recovery equipment for the gasifier effluent gas stream, and in designing the catalyst recovery system. Data on properties of the "mid-char" from the FBG fluid bed will also be reviewed to serve as a basis for gasifier volume calculations using the catalytic gasifier reactor model.

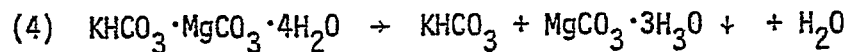
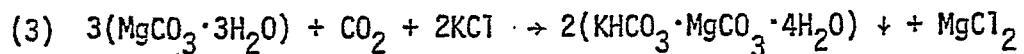
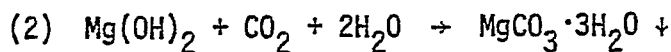
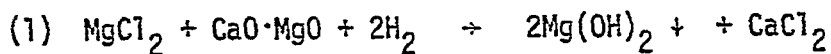
Commercial Sources of Potassium Catalyst--Engel-Precht Process Screening Evaluation

A screening-quality evaluation of the Engel-Precht process for manufacturing potassium catalyst for the catalytic gasification process has been completed. Based on the "cash flow" scoping studies reported in the June Monthly Report, the Engel-Precht process appeared to be the most economically attractive of the alternative chemical processes which were considered.

Historically, the Engel-Precht process was used in Germany to manufacture potassium carbonate and bicarbonate commercially as a batch operation from about 1900 to 1938. In about 1938, the Formate process

(K₂SO₄ to KHCO₂) superceded the Engel-Precht process. In the last 20 years, Israel and Saskatchewan have had renewed interest in the Engel-Precht process to convert their KCl deposits to K₂CO₃ or KHCO₃ in a continuous operation. All the process basis information used in developing this study was taken from the open literature. Significant uncertainties exist as to the quality and consistency of the available data. Based on the literature, no commercial Engel-Precht plants, continuous or batch, are presently in operation.

The Engel-Precht process involves four reaction steps, as follows: follows:



Because reaction (4) requires careful control of reaction conditions to produce reusable MgCO₃·3H₂O, only a dilute KHCO₃ solution can be made directly (about 9 wt% maximum). Steam-heated evaporators have been included in the screening case to concentrate the product solution to 25 wt% for feed to the catalyst addition facilities.

Investment costs for this Engel-Precht screening evaluation fall into three main categories: costs of materials handling and processing equipment to carry out the Engel-Precht reactions, costs of facilities required to supply needed utilities (principally the steam for the evaporators), and net costs of modifying the catalytic gasification plant to receive catalyst makeup as 25% KHCO₃ solution. The Engel-Precht process facilities were cost-estimated based on individual equipment specifications, and the remaining facilities were prorated from the catalytic gasification "Base Case."

The breakdown of the product KHCO₃ cost for the Engel-Precht process screening evaluation is shown in Table III. Costs are shown as percentages of the cost of KOH solution purchased on the open market (on a potassium-equivalent basis). This is consistent with the basis used in presenting the results of the "cash flow" analyses in the June Monthly Report. As shown in Table III, this screening study indicates that the total estimated cost of KHCO₃ catalyst from an Engel-Precht plant is about 94% of the cost of purchased KOH. This 6% advantage appears to be insufficient to justify further development work on the Engel-Precht process as a catalyst source for catalytic gasification. However, a final comparison must await completion of ongoing studies to better define the

TABLE III

ENGEL-PRECHT PROCESS SCREENING EVALUATION
BREAKDOWN OF RELATIVE PRODUCT CATALYST COST

Basis: Cost of Purchased KOH = 100 (Note 1)

● Raw Materials:	
- KCl (Agricultural Grade)	32
- CaO·MgO (Calcined Dolomite)	6
● Utilities:	
- Coal Fuel	4
- Raw Water	Nil
- Cooling Water	Nil
- Electric Power	5
- Low Pressure Steam (Note 2)	(3)
● Byproduct Sulfuric Acid	(1)
● Labor and Related Costs	5
● Investment-Related Costs	7
● Capital Charges (Note 3)	<u>39</u>
Total Product Catalyst Cost	<u>94</u>
	—

Notes:

- (1) On a potassium-equivalent basis.
- (2) Operating cost credit for low pressure steam based on using non-condensing steam turbine drivers to back out purchased power.
- (3) Capital charges based on 100% equity financing with 10% DCF return.

cost of KCl electrolysis to produce KOH.

Evaluation of Catalyst Recovery Alternatives--Screening
Study of Hydrothermal Treating

Based on "cash flow" scoping studies reported in the June Monthly Report, catalyst recovery via hydrothermal treatment with $\text{Ca}(\text{OH})_2$ offers the potential for substantial savings relative to purchased KOH at the market price. The "minimum cost" of the incremental potassium recovered via this route was estimated to be 36-54% of the cost of purchased KOH, depending on the $\text{Ca}(\text{OH})_2$ requirement. This evaluation was based on bench-scale experiments. Although investment charges for hydrothermal treatment were not included in the "Minimum cost," the added investment required for these facilities is expected to be small.

A screening study is now in progress for catalyst recovery via hydrothermal treatment which will define the facilities and qualify the investment required. Bases are being set reflecting current laboratory data. Investments and operating costs for this case will be compared with costs for water washing only to select the preferred catalyst recovery system for inclusion in the new study design described above.

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