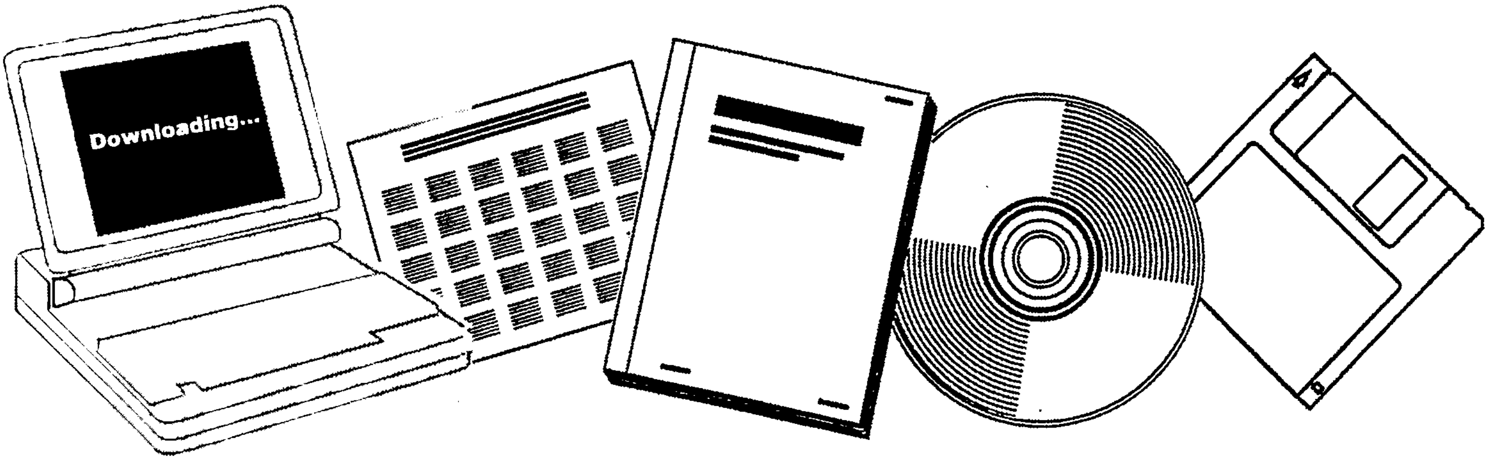
EXXON CATALYTIC COAL GASIFICATION PROCESS PREDEVELOPMENT PROGRAM. MONTHLY REPORT, DECEMBER, 1977

EXXON RESEARCH AND ENGINEERING CO.
BAYTOWN, TX

26 MAY 1978



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EXXON CATALYTIC COAL GASIFICATION PROCESS PREDEVELOPMENT PROGRAM

Monthly Report for the Period
December, 1977

N. C. Nahas--Project Manager

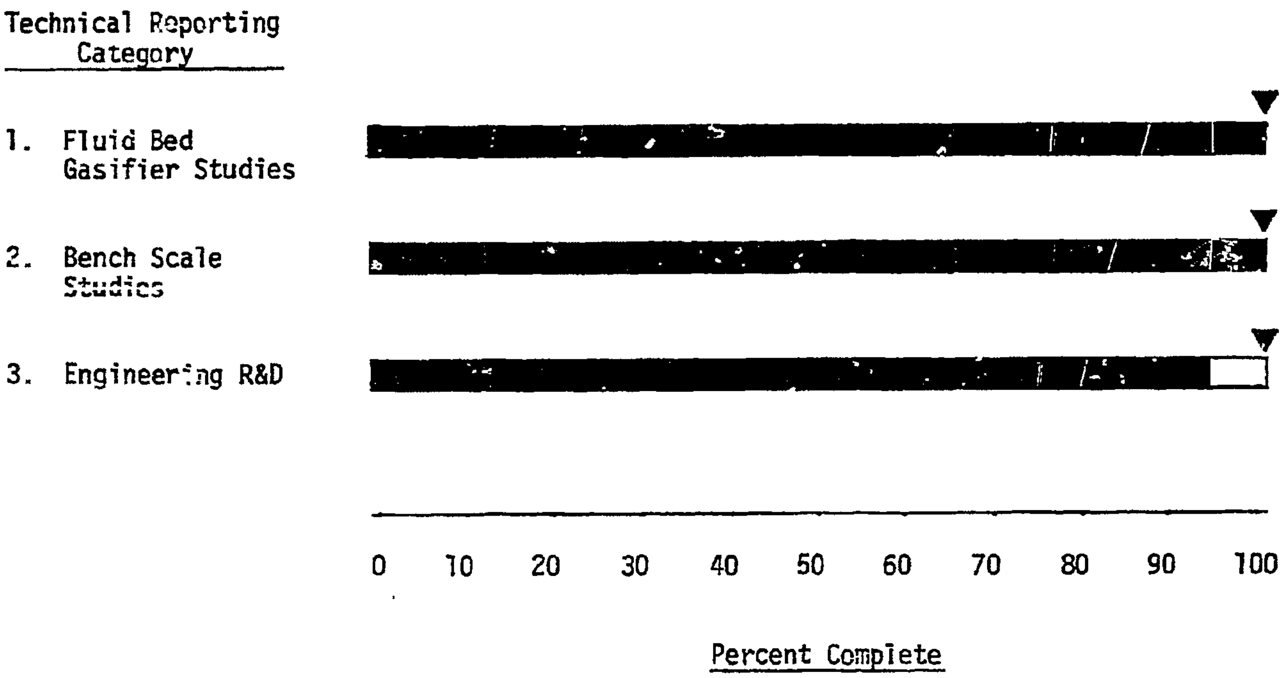
Exxon Research and Engineering Company
Baytown, Texas 77520

May 26, 1978

PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Contract No. E(49-18)-2369

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



Legend

Shaded area = Percent of activity actually completed

▼ = Percent of activity scheduled for completion

ABSTRACT

Solids analyses for all FBG yield periods have been completed. The off-line material balance computer program has been used to reconcile the data from 11 of the 18 FBG yield periods. Bench scale fixed bed gasification studies indicated that KOH is equivalent to K_2CO_3 as a gasification catalyst. However, K_2S shows lower catalytic activity than K_2CO_3. Also, Wyodak coal catalyzed with either KOH or K_2CO_3 shows the same reactivity as Illinois coal impregnated with these catalysts.

Work continued on the Catalytic Coal Gasification Commercial Plant Study Design. Process bases and heat and material balances have been completed. Current efforts are directed toward calculation of investment, operating and product gas costs.

A screening study was made of alternative methods of producing makeup KOH by electrolysis of KCl. This study showed that KOH could be produced in diaphragm cells or membrane cells at significantly lower cost than the current price of purchased KOH. Another screening study showed that the cost of potassium recovered by calcium hydroxide digestion is about 68 percent of the cost of purchased KOH. Further work will be required to better define the relative costs of recovered vs. manufactured KOH.

Studies to evaluate alternative acid gas removal processes were completed. An evaluation of conventional acid gas scrubbing techniques indicated that selective heavy glycol scrubbing is the preferred process for a catalytic gasification plant, closely followed by non-selective hot potassium carbonate scrubbing. An evaluation of cryogenic fractionation for acid gas removal showed that this technique cannot be carried out without CO_2 freezing out in the fractionator.

An evaluation was completed of the incentive for a two-stage gasification reactor with the upper stage being used for coal drying and the lower stage being used for gasification. The gas cost calculated for the two-stage gasifier is about one percent greater than for the base case gasifier. Thus, there is no incentive for further pursuit of this two-stage gasifier option.

DISCUSSION

FLUID BED GASIFIER STUDIES (REPORTING CATEGORY 1)

FBG Data Workup

Solids analyses for all FBG yield periods were finished during December. Completed yield period data are being reconciled by the off-line material balance computer program as they become available. Data from two more yield periods have been reconciled by this program with excellent results. This brings the total to date to 11. Results for all 18 FBG yield periods will be included in the final report of work completed under the Predevelopment Contract.

BENCH SCALE STUDIES (REPORTING CATEGORY 2)

Fixed Bed Gasification Studies

Data have been obtained in the fixed bed reactor on the catalytic gasification rates with steam of Illinois #6 coal impregnated with 16% K_2CO_3, 10% KOH and 12% K_2S by weight on coal. Data were also obtained on the gasification rate of Wyodak coal impregnated with 15% K_2CO_3 and 12% KOH by weight. The results of these tests indicate that KOH is equivalent on a molar basis to K_2CO_3 when impregnated on both Illinois and Wyodak coal. Also, the reactivity of Wyodak coal impregnated with either KOH or K_2CO_3 is essentially the same as Illinois coal impregnated with these catalysts. K_2S impregnated on Illinois coal yielded a lower reactivity than either KOH or K_2CO_3, which may be the result of the oxidation of K_2S to inactive sulfate by the pure steam atmosphere.

Figure 1 shows the gasification rates of Illinois coal impregnated with 16% K_2CO_3, 10% KOH (12% K_2CO_3 equivalent) and 12% K_2S (15% K_2CO_3 equivalent) as obtained in the fixed bed reactor at 1300°F and 500 psig. The plot represents conversion expressed as moles of carbon gasified per mole of steam fed versus relative steam residence time. Also shown on the plot are correlation lines for 12% and 16% K_2CO_3 which have been estimated from previous runs. The data obtained with 16% K_2CO_3 closely approximate the predicted reactivity of Illinois coal with this catalyst level. The 10% KOH loading yielded reactivities which are similar to those predicted for a 12% K_2CO_3 loading which represents equivalent moles of alkali per coal feed. The reactivity of Illinois coal impregnated with 12% K_2S (15% K_2CO_3 equivalent) yielded significantly lower reactivity than an equivalent amount of K_2CO_3. Two possibilities for this behavior exist: either K_2S is a poor catalyst or the pure steam atmosphere oxidized the K_2S to an inactive sulfate form. A thermodynamic analysis shows that the product gas composition should favor carbonate over sulfide and either are favored over sulfate, but at low steam conversions sulfate is favored. Future work should focus on the possible forms of K_2S in the gasifier under reducing conditions.

In Figure 2 the reactivities of Wyodak coal impregnated with 15% K_2CO_3 and 12% KOH are shown. As the plot indicates these loadings on Wyodak are essentially equivalent. The scatter in the KOH data is most likely due to uneven impregnation of the sample. A further conclusion is that 15% K_2CO_3 on Wyodak is essentially equivalent to 15% K_2CO_3 on Illinois.

Figure 1

CATALYTIC GASIFICATION OF ILLINOIS COAL

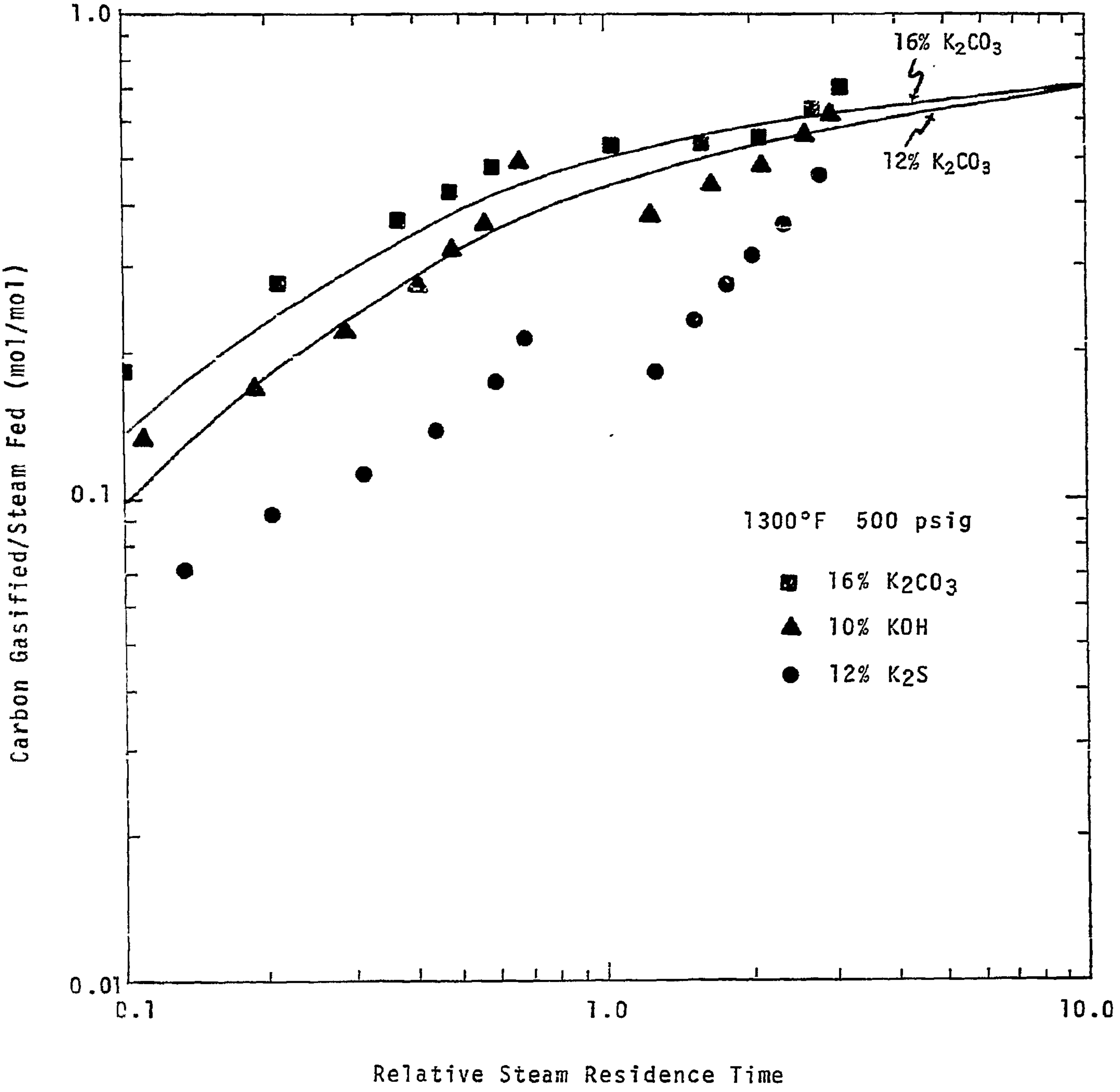


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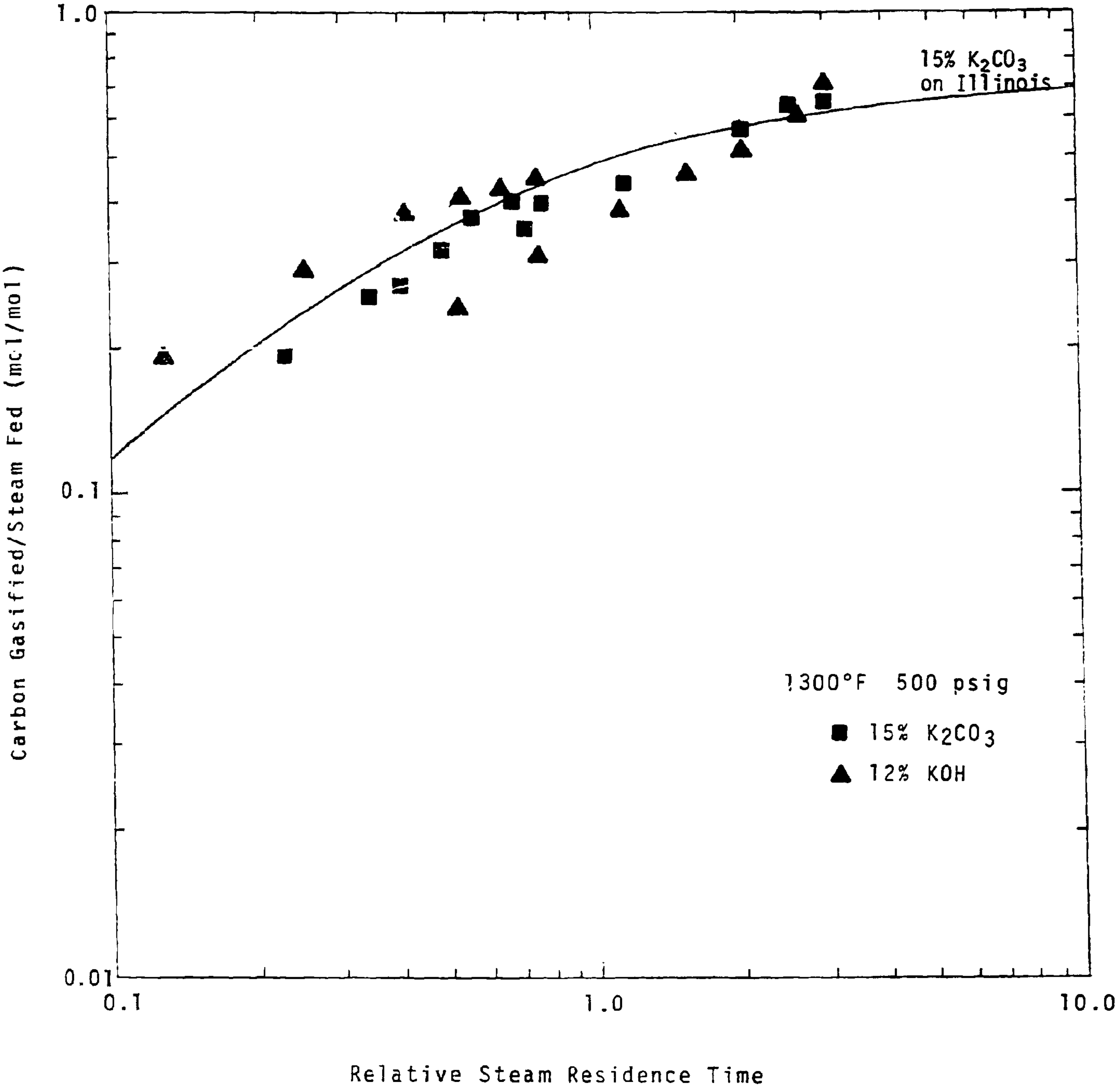
Figure 2

CATALYTIC GASIFICATION OF WYODAK



ENGINEERING RESEARCH AND DEVELOPMENT (REPORTING CATEGORY 3)

Catalytic Coal Gasification Commercial Plant Study Design

Work on the Catalytic Coal Gasification Commercial Plant Study Design neared completion in December. The Study Design reflects the current conception of a commercial plant producing approximately 250 MSCF/SD of SNG from Illinois coal. All necessary onsites, offsites, and utilities facilities are included in the Study Design. Estimates are being made of both investment and operating costs.

Process bases have been developed and heat and material balances have been completed for all onsites sections. Utilities balances have also been completed. Equipment sizes have been calculated and specifications lists prepared for all onsites, offsites, and utilities. The completed lists are providing the basis for the investment estimate for the Study Design.

The main steps remaining in the Study Design effort are: 1) completion and review of the investment estimate; 2) calculation of operating costs including coal, catalysts and chemicals, manpower, and utilities; 3) calculation of product SNG costs on utility and private investor financing bases; and 4) documentation of results in the final program report.

Commercial Sources of Potassium Catalysts: Analysis of Alternative Electrolysis Technologies

A rough screening study was completed of the alternative KCl electrolysis technologies for producing potassium catalyst for a commercial catalytic gasification plant. Vendor contacts were made to define investment and operating cost factors for use in this study. The results of this study, along with the previous catalyst manufacturing cost studies reported in the June and July 1977 Monthly Reports, indicate that KCl electrolysis is the most economical method for producing makeup potassium catalyst for catalytic gasification plants. The three electrolysis technologies evaluated were diaphragm, membrane and mercury cells at both 180 T/D and 470 T/D KOH product rate. The KOH makeup requirement for the Catalytic Coal Gasification Commercial Plant Study Design is about 190 T/D KOH.

The estimated relative catalyst costs of these alternatives are as follows:

	180 T/D KOH	470 T/D KOH
Purchased KOH Cost	---------100 (Base)---------	
Diaphragm Cells	76	56
Membrane Cells	75	62
Mercury Cells	67	54

These economics reflect an f.o.b. cost for 50% KOH solution produced in the diaphragm and mercury cells and 31% solution from the membrane cells. Capital charges are based on 100% equity funding and 10% constant dollar DCF return.

As indicated from these figures, potassium catalyst produced specifically for catalytic gasification plants may be significantly cheaper than purchasing KOH from the open market. The cost differences between purchased and manufactured KOH may be attributable to the differences in project basis: KOH for use in catalytic gasification is here assumed to be manufactured by a dedicated plant producing large quantities of relatively impure KOH (98-99% dry basis) as makeup catalyst; the current market reflects supplies of small quantities of high purity KOH ($99.6\%^{+}$ dry basis) to multiple users. Specifically, the electrolysis screening cases presented here are based on "white" grade KCl feedstock (rather than high purity "chemical" grade), unit train shipment of feed KCl, and use of electrolysis byproduct hydrogen for fuel in the SNG plant.

KOH Production from mercury cells is the most attractive technology economically. However, increasingly stringent mercury emissions regulations will likely obviate large scale use of this technology in the future. At the smaller 180 T/D KOH scale, the alternative diaphragm or membrane cell technologies are about equal in cost. Both produce KOH for catalytic gasification plants at about 75% of the cost of purchased KOH. Since membrane cells are at an earlier stage of development than diaphragm cells (pioneer plant vs. commercial), further improvements during development may make membrane cells the preferred technology in the long run.

Due to differing economies of scale, diaphragm cells are currently more economical than membrane cells at the larger 470 T/D KOH scale. However, the potential applicability of larger-size dedicated electrolysis plants is more limited. Such plants may be applicable in the context of a mature catalytic gasification industry or if secondary catalyst recovery is deleted.

Catalyst Recovery Studies: Screening Study of $Ca(OH)_2$ Digestion

A screening study has been completed to evaluate catalyst recovery using hot, aqueous $Ca(OH)_2$ digestion of the catalyst-containing char and fines solids withdrawn from the gasifiers. The objective was to compare the investment and operating costs for this case with costs for water washing alone to quantify the relative economics of the two alternatives.

Water washing alone has been shown to recover readily only about 70% of the potassium salts in the spent gasifier solids when starting with 15 wt.% K_2CO_3 equivalent on Illinois coal (dry basis). The remaining salts are tied up as water-insoluble complexes with the coal ash. $Ca(OH)_2$ digestion has been shown in laboratory experiments to solubilize most of the otherwise water-insoluble potassium. Then, countercurrent water washing can be used to recover 90% or more of the total potassium.

Based on "cash flow" scoping studies reported in the June 1977 Monthly Report and on recent laboratory results, $Ca(OH)_2$ digestion is the most attractive alternative of the processes which have been considered for recovering the water-insoluble catalyst.

A simplified process flow plan for the Base "Water Wash Only" Case is shown in Figure 3. Gasifier char solids from the char withdrawal system and gasifier fines from the tertiary cyclone catch are separately slurried with portions of the semi-rich catalyst solution from the second stage of catalyst recovery. The two slurries are then depressured into the first-stage water wash mixing drum, operating at 20 psia and 230°F. The mixing drum effluent slurry is pumped through the first-stage hydroclone separators. The overflow from the first stage hydroclones, which is the most concentrated potassium solution in the system, is fed into the rich catalyst solution holding drum. Makeup 30 wt.% KOH solution is also fed into this drum to replace the potassium not recovered in the washing sequence. The recovered catalyst solution is pumped to the catalyst addition facilities where it is reimpregnated on the feed coal to the gasifiers. The first-stage hydroclone underflow slurry is mixed with the third-stage hydroclone overflow solution in the second-stage mixing drum. The mixture is then pumped through the second-stage hydroclones. The semi-rich overflow from this stage is used to slurry the char and fines, and the underflow is fed into the third-stage mixing drum. This countercurrent water-washing sequence continues in a similar manner until the fifteenth stage, where clean makeup wash water is preheated and added to the system. The leached solids in the last stage underflow slurry are sent to offsites waste solids handling facilities. Catalyst solution from filtration of venturi fines slurry stripper bottoms enters catalyst recovery in the tenth stage, where the concentrations are similar.

A simplified process flow plan for the "$Ca(OH)_2$ Digestion with Water Wash" Case is shown in Figure 4. In this case, the gasifier char and fines feed slurries are depressured into a $Ca(OH)_2$ digestor, operating at 70 psia and 300°F. A portion of the semi-rich solution is fed in this case to a lime feed slurry drum and mixed with lime (CaO) solids and makeup catalyst (30 wt.% KOH solution). This lime slurry is then mixed and reacted with the char and fines slurries in the $Ca(OH)_2$ digestor, with a residence time of two hours. The Ca/total K ratio is maintained at 0.7 mol/mol. These digestion conditions have been shown in lab tests to solubilize at least 90% of the total potassium in the gasifier solids. The $Ca(OH)_2$ digestor effluent is pumped through the first-stage hydroclone separators. The first-stage hydroclone overflow, again the most concentrated potassium solution in the system, is depressurized into the rich catalyst solution holding drum and pumped on to the catalyst addition facilities. The downstream water washing sequence is similar to the Water Wash Only Case, except solids throughput is greater due to the presence of insoluble calcium salts, and one less stage of washing is required to obtain the target 98.5% recovery of solubilized potassium salts.

The investment costs considered in this screening evaluation fall into two main categories: costs for facilities directly related to recovery and handling of catalyst, i.e., costs for the catalyst recovery

Figure 3
CATALYTIC COAL GASIFICATION CATALYST RECOVERY SYSTEM
"WATER-WASH ONLY" CASE

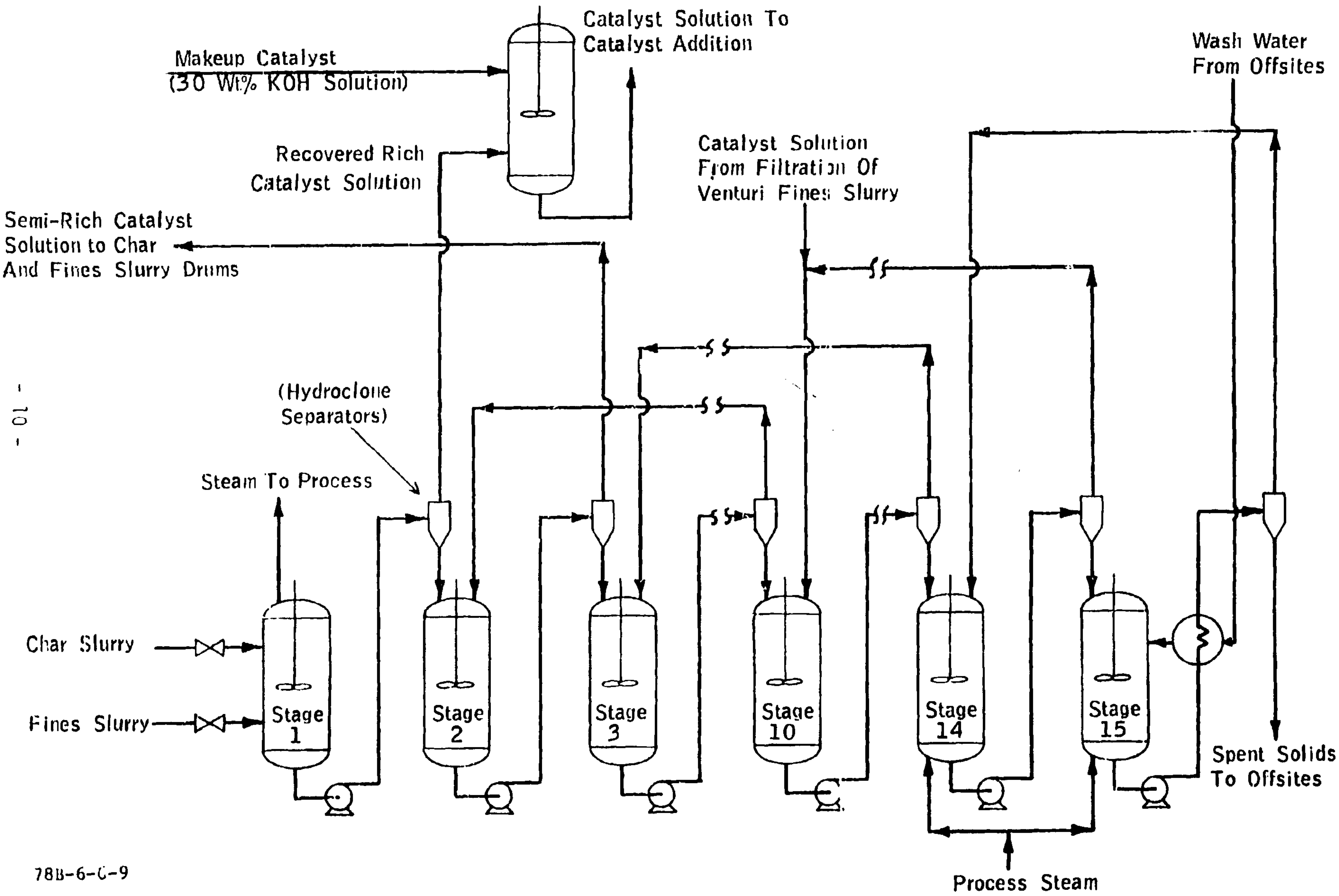
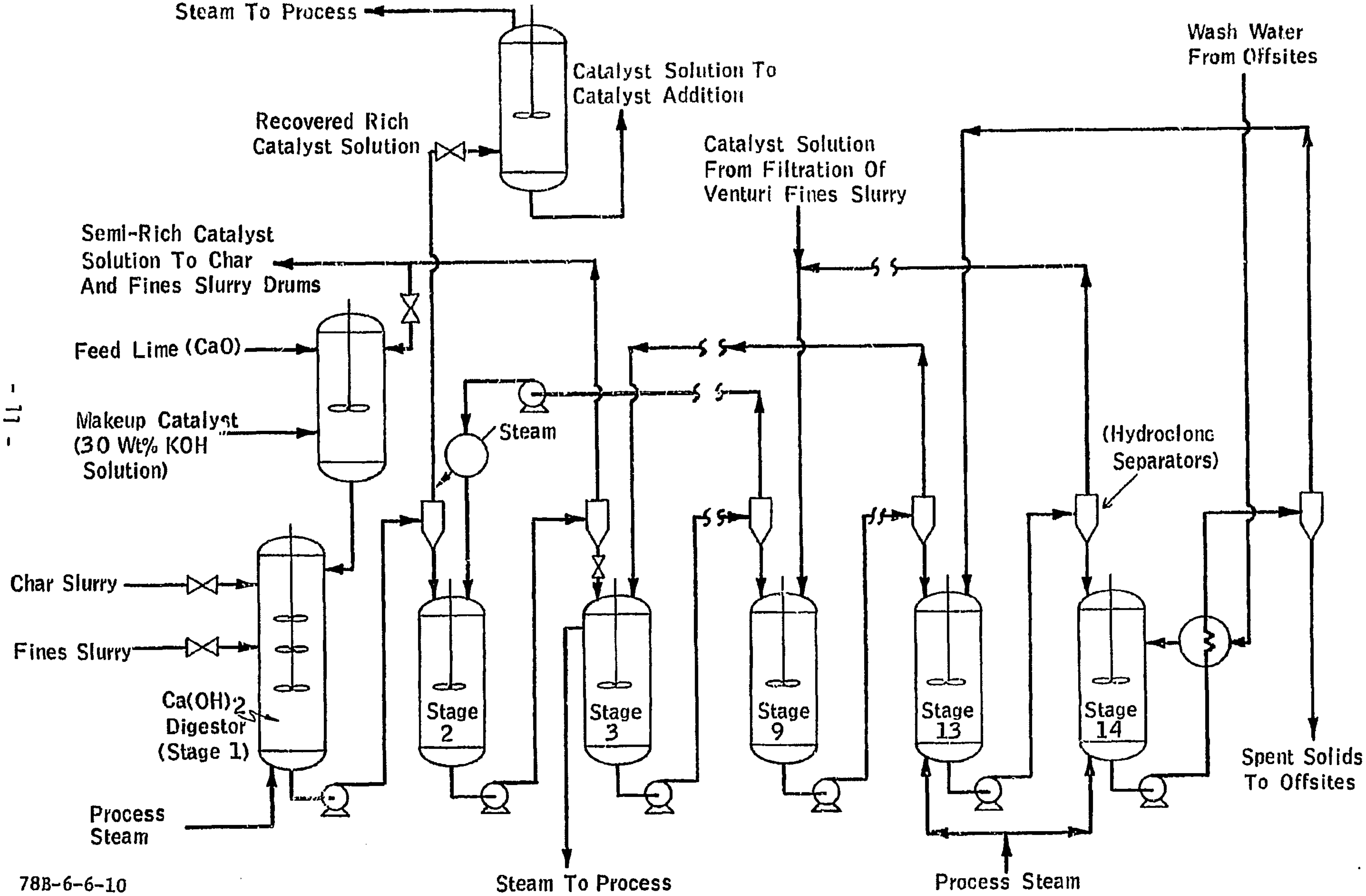


Figure 4

CATALYTIC COAL GASIFICATION CATALYST RECOVERY SYSTEM
"$Ca(OH)_2$ DIGESTION WITH WATER WASH" CASE



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system, associated raw materials handling, and facilities to supply required utilities; and second, costs for changes to other facilities in the gasification plant, i.e., coal drying/catalyst addition, preheat furnaces, waste solids handling, and flue gas desulfurization. The facilities for catalyst recovery and raw materials handling for $Ca(OH)_2$ Digestion with Water Wash were cost estimated based on individual equipment specifications. The facilities costs for catalyst recovery and raw materials handling for the Water Wash Only Case were prorated from these costs. Investment cost impacts for changes to other facilities in the gasification plant were prorated from earlier catalytic gasification studies.

Table 1 presents a breakdown of the incremental costs for catalyst recovery by $Ca(OH)_2$ Digestion with Water Wash versus the costs for Water Wash Only. The incremental costs have been divided by the incremental tons of KOH equivalent recovered by using $Ca(OH)_2$ digestion, and these costs per ton have in turn been expressed as percentages of the cost of purchased makeup KOH at the current market price. As Table 1 illustrates, the total cost of the incremental catalyst recovered by including $Ca(OH)_2$ digestion in the catalyst recovery system is 68% of the cost of an equivalent amount of purchased KOH. This potential advantage of over 30% justifies further development work on the $Ca(OH)_2$ digestion process.

Obviously, the incentive for $Ca(OH)_2$ digestion would be lower if makeup KOH could be manufactured at a cost substantially below the current market price. As reported earlier in this report, this may indeed be possible, since special factors associated with manufacturing KOH for use in catalytic gasification tend to lower the cost relative to the present market. In the next development phase, a more detailed study of catalyst manufacturing facilities will be needed to better define the costs of KOH for catalytic gasification. Results of future laboratory experiments on $Ca(OH)_2$ digestion and water wash will also help to firm up the relative economics of $Ca(OH)_2$ digestion.

Evaluation of Conventional Acid Gas Removal Processes

A screening quality evaluation of conventional acid gas (CO_2 and H_2S) scrubbing alternatives for use with catalytic gasification has been completed. The alternatives were limited to processes that are commercially demonstrated and have reasonable compatability with the catalytic gasification process. The three systems considered are selective scrubbing with a heavy glycol solvent, selective scrubbing with refrigerated methanol, and nonselective scrubbing with hot potassium carbonate.

The selective processes recover essentially all the H_2S from the gasifier effluent in an H_2S-rich stream concentrated enough to feed to a Claus sulfur recovery plant. Most of the CO_2 is recovered in a second stream and vented. The nonselective process recovers H_2S and CO_2 together in a single stream. The H_2S is then removed from this more dilute stream and converted to sulfur in a Stretford sulfur recovery unit. Since Stretford is more expensive than Claus, costs for conversion of the H_2S to by-product sulfur are significantly higher with the nonselective process.

Table I

SCREENING STUDY OF CATALYST RECOVERY USING $Ca(OH)_2$ DIGESTION
BREAKDOWN OF INCREMENTAL COSTS WITH RESPECT TO WATER WASH ONLY

Basis: Cost of Purchased KOH = 100

Cost Items	Incremental Costs Per Increment of KOH Equivalent Recovered
• Raw Materials	
- Lime, 97% CaO	39
• Utilities	
- Power	1
- Intermediate Pressure Steam(1)	(1)
• Coal for Dryer Fuel	(1)
• Fuel Gas	(1)
• Labor and Related Costs	4
• Investment-Related Costs	4
• Capital Charges(2)	23
Total Cost of Incremental Catalyst Recovered	68

Notes:

(1) Operating cost credit for intermediate pressure steam is based on using noncondensing steam turbine drivers to back out purchased power.

(2) Capital charges are based on 100% equity financing with 10% constant dollar DCF return.

The three systems were compared based on the processing conditions and acid gas removal requirements for the Catalytic Coal Gasification Commercial Plant Study Design. Non-confidential vendor design and cost information was used to evaluate the first two acid gas scrubbing alternatives. The vendor cost information was reviewed by ER&E and put on a consistent basis. An equipment list and cost estimate was prepared on the same basis by ER&E for hot potassium carbonate scrubbing.

A major problem in comparing these alternative acid gas removal processes is differences in the degree of engineering detail developed for each of the three cases. The cases were done by different engineering organizations, and this makes absolute comparison of the processes very difficult. The results of this evaluation must therefore be viewed as preliminary and a more thorough and consistent approach would be needed to arrive at definitive conclusions.

The relative investments and gas cost impacts of the three alternative processes are as follows:

	Selective Heavy Glycol Solvent	Selective Refrigerated Methanol	Non-Selective Hot Potassium Carbonate
Relative Investment			
Acid Gas removal	0.706	0.875	0.337
Sulfur Recovery	0.097	0.074	0.280
Associated Facilities	0.197	0.220	0.690
Total	1.000	1.169	1.307
Relative Gas Cost Impact			
Operating Costs	0.356	0.285	0.223
Capital Charges	0.602	0.708	0.783
Methane Losses	0.042	0.145	0.011
Total	1.000	1.138	1.017

For each case, these economics include the acid gas removal process and its associated facilities (e.g., feed pretreatment, sulfur recovery, final gas cleanup for feed to cryogenic methane recovery, and prorata utilities costs). Capital charges are based on 100% equity funding and 10% constant dollar DCF return.

Based on this preliminary analysis, selective heavy glycol scrubbing is the preferred acid gas removal process for a catalytic gasification SNG plant, but only by a small margin. This system has been included in the Commercial Plant Study Design. A non-selective hot potassium carbonate system would have only a 2% greater gas cost impact, and thus can be considered about breakeven with the heavy glycol system. The high associated facilities charge for this case is due to hot carbonate's high steam usage. The selective refrigerated methanol system is the least attractive as it increases the gas cost contribution due to acid gas removal by about 14%. One of the major debits for this process is a relatively high methane loss due to high methane solubility in the methanol solvent.

Evaluation of Cryogenic Fractionation for Acid Gas Removal

The objective of this study was to evaluate the technical feasibility of cryogenic fractionation for separating acid gases (CO_2 and H_2S) from the rest of the catalytic gasifier effluent gas, consisting principally of CH_4, CO and H_2. Conventional methods for removal of CO_2 and H_2S from gas streams generally utilize gas scrubbing with either a chemical or a physical solvent. Typically, acid gases are removed down to desired specification levels by scrubbing the feed gas with the solvent in an absorber, and the solvent is stripped and/or reboiled to separate the acid gases in a regenerator. The three acid gas removal processes reported above represent the conventional approach to acid gas removal.

Fractionation is thermodynamically more reversible than either chemical or physical absorption processes, and therefore may be attractive due to lower energy requirements. Additionally, since cryogenic methane recovery is required for CH_4 separation in the catalytic gasification SNG process, integration of the two cryogenic processing blocks could be particularly attractive.

In work done with Exxon funds prior to the current predevelopment phase, a proprietary fractionation scheme had been proposed for separating acid gases without CO_2 freeze-out. However, based on the present simulation studies of system process variables, it has been concluded that CO_2 freeze-out appears likely to occur in some part of the acid gas fractionation system throughout the range of tower operating conditions necessary to meet other system limitations. In the course of this study, several fractionation tower heat and material balances were developed using Exxon proprietary correlations of literature data on vapor-liquid equilibrium and CO_2 freeze-out in CH_4/CO_2 solutions.

The proposed acid gas fractionation system is severely constrained by phase behavior and process requirements. The operating pressure must be selected to avoid both CO_2 freeze-out and the CO_2 critical point. In addition, the overhead operating temperature must be kept low to maintain a low outlet CO_2 concentration to avoid excessive costs in the downstream molecular sieve final cleanup step preceeding the cryogenic methane recovery system. Furthermore, feed cooling is limited to temperatures above the CO_2 triple point to avoid CO_2 freeze-out in the feed cooler. The combination of these limitations appears to permit no feasible operating regime for the tower which could avoid CO_2 freeze-out.

The primary cause of CO_2 freeze-out in the acid gas fractionation scheme is that the fractionation between CH_4 and CO_2 is too good at the top of the tower. In this part of the tower where the temperature is low enough to allow CO_2 freeze-out, the volatility of methane relative to CO_2 is sufficiently high so that CO_2 is the primary component in the liquid phase. Since this liquid phase CO_2 concentration is above the limit of CO_2 solubility in CH_4 at these conditions, freeze-out of solid CO_2 would occur. No further engineering work is planned under the predevelopment contract on possible approaches to operation in the CO_2 freeze-out regime.

Evaluation of Two-stage Gasification Reactor with Upper Drying Stage

A screening-quality evaluation of a two-stage gasification reactor with the upper stage being used for coal drying and the lower stage being used for gasification has been completed. The objective of this study was to define the incentive for using the high level heat in the gasifier effluent for feed coal drying. The process bases and key process differences relative to the Base Case were presented in the October and November 1977 Monthly Reports.

The economic results for these cases are as follows:

	Base Case	Two-Stage Gasifier with Upper Drying Stage
Reactor System	One-Stage Gasifier	Two Stages: Upper for Drying Lower for Gasification
Relative Investment	1.000	1.000
Relative Gas Cost		
Coal	0.261	0.267
Gasification Catalyst	0.063	0.066
By-Product Credits	(0.057)	(0.059)
Operating Costs	0.235	0.240
Capital Charges	0.498	0.498
Total	1.000	1.012

Both cases are sized to produce 257 GBtu/SD SNG from Illinois coal. Capital charges are based on 100% equity funding and 10% constant dollar DCF return.

The investment relative to the Base Case remains the same. Use of an upper stage dryer permits a significant reduction in the duty of the catalyst addition/drying facilities in the coal preparation section. The gas-gas exchangers are eliminated because the incentive for their use is greatly reduced when the gasifier effluent high level heat is used for coal drying. Offsetting these savings is a more complicated two-stage reactor and incrementally larger recycle gas handling facilities.

The gas cost calculated for this two-stage gasifier case is about 1% greater than for the Base Case. There are two main reasons for this increase. First, total process coal and makeup catalyst requirements are about 6% higher for the same net SNG product rate due to increased consumption of methane as preheat furnace fuel. Second, utilities demands are greater due to larger overall processing requirements. At this time, there appears to be no incentive for further pursuit of this two-stage dryer/gasifier option.

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