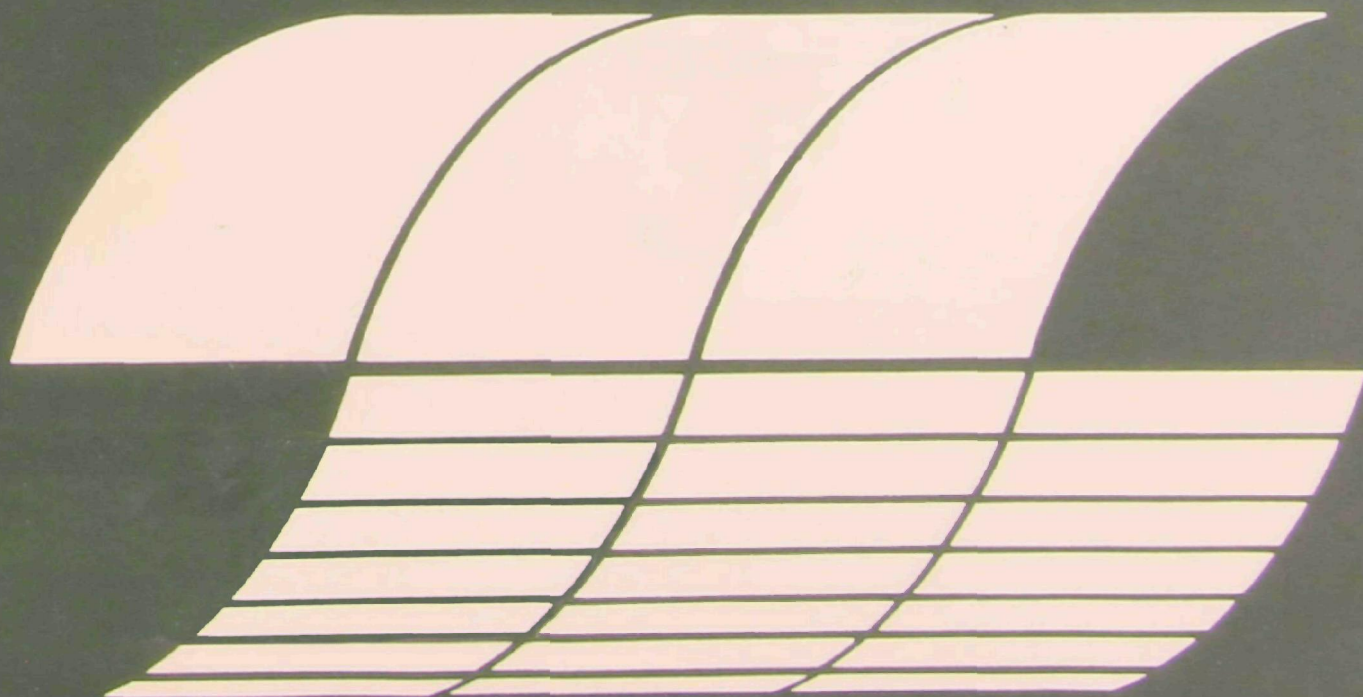




Assessment of Coal Cleaning Technology: An Evaluation of Chemical Coal Cleaning Processes

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Assessment of Coal Cleaning Technology: An Evaluation of Chemical Coal Cleaning Processes

by

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ABSTRACT

This Task report is the result of a nine month study on one phase of a three-year contract commissioned by the Fuel Process Branch of the Industrial Environmental Research Laboratory of the U.S. Environmental Protection Agency at Research Triangle Park, North Carolina. The primary objective of the total program is to perform a comprehensive collection and evaluation of physical and chemical coal cleaning technology for the removal of sulfur from coal.

This specific Task report covers the technical and economic evaluation of major U.S. chemical coal cleaning processes.

A variety of chemical coal cleaning processes are under development which will remove a majority of pyritic sulfur from the coal with acceptable heating value recovery i.e. 95 percent BTU recovery. Some of these processes are also capable of removing organic sulfur from the coal, which is not possible with the physical coal cleaning processes. Chemical coal cleaning processes can remove as much as 95 to 99 percent of pyritic sulfur and up to about 40 percent of the organic sulfur from the run-of-mine coal. This removal efficiency could result in total sulfur reductions in U.S. coals in the range of 53 to 77 percent.

This report presents available technical and economic information on major chemical coal cleaning processes identified during the study. Information on each process is provided in a format to identify:

- Process details,
- Developmental status,
- Technical evaluation, including process potential for sulfur removal, sulfur by-products, process advantages and disadvantages, environmental aspects, research and development needs, and
- Process economics.

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SECTION 1

INTRODUCTION

A major concern for preserving the quality of the environment resulted in Congress passing the Air Quality Act of 1963 which initiated a concerted effort by Federal, State and local Governments to clean up the quality of the Nation's air. This act placed special emphasis on the problem of sulfur oxide emissions from the combustion of coal and oil in stationary plants.

The U.S. Environmental Protection Agency under the Clean Air Act of 1970 and the Clean Air Act Amendments of 1977 is charged with the promulgation of standards and the implementation of state and federal plans for the reduction of sulfur dioxide emissions.

In 1974, sulfur oxide emissions from coal combustion were in excess of 18.6 million metric tons (20.5 million tons). With the projected increase in the use of coal as a major energy source, improved methods are needed for the control of this pollutant emission. Industrial coal consumption is projected to quadruple from 61.7 million metric tons (68 million tons) in 1975 to 251 million metric tons (277 million tons) in 1985. Utility coal consumption is projected to increase from 366 million metric tons (404 million tons) to 707 million metric tons (779 million tons).¹ The success of the National Energy Plan depends heavily on the adequacy of pollutant emission control technology.

The possible solutions to this problem are the following processes which are capable of attaining sulfur dioxide reductions:

- Flue gas desulfurization - removes SO₂ from coal combustion flue gases;
- Physical coal cleaning - removes pyritic sulfur from coal prior to combustion;

- Chemical coal cleaning - removes pyritic and organic sulfur from coal prior to combustion;
- Synthetic fuel production - conversion of coal into clean burning gaseous or liquid fuels; and
- Fluidized bed combustion - burning coal in the presence of additives that will remove sulfur as a mineral residue.

Several chemical coal cleaning processes are under development that claim removal of substantial quantities of organic sulfur as well as greater than 90 percent reduction of pyritic sulfur. If these processes are found to be feasible on a commercial scale, they could have a significant impact on coal utilization. It has been estimated that chemical processes which can remove as much as 95 to 99 percent of pyritic sulfur and up to 40 percent of organic sulfur from raw coal could achieve total sulfur reductions in U.S. coals in the range of 53 to 77 percent.

Recognizing the importance of chemical coal cleaning processes as a potential sulfur dioxide pollutant control option, the Energy Assessment and Control Division of the Industrial Environmental Research Laboratory of EPA contracted with Versar to study the technical and economic feasibility of the chemical coal cleaning processes. This study is one task of a major study titled "Coal Cleaning Technology Development".

PROJECT OBJECTIVES

The objective of this study was to survey the field of chemical coal cleaning processes to identify active and inactive processes and perform a critical evaluation of competing processes. The purposes of this evaluation were fourfold:

- To provide updated information on technical and economic viability of these processes and identify their developmental stage;
- To examine their performance characteristics and environmental aspects;
- To develop quantifiable technical and economic parameters for purposes of process comparison; and
- To identify specific research and development needs for processes showing a potential for substantial reduction of sulfur in coals.

PROJECT METHODOLOGY

The conduct of the project may be described in terms of the following four work phases:

- data acquisition;
- develop data base;
- process and cost information analysis; and
- process and cost comparison.

Data Acquisition

The data needed for this study were obtained by four different methods. First, by reviewing published information in the technical literature, patents and government documents made available by EPA. These references are cited throughout this report and listed in Section 5. The second method involved using data compiled by EPA during a previous study on chemical coal cleaning. The third method involved telephone and mail contact with the developer of each process to obtain detailed process and economic information. The fourth method of data acquisition was by interviewing process developers and making site visits to process laboratories or pilot plants.

The data needed for this report were assembled and compiled in the time frame May through September, 1977. The chemical coal cleaning is, however, a dynamic field and several of the processes are under further investigation and development. It is likely that the on-going effort could have a significant impact on some processes and could have resulted in process designs that are superior to the ones described and discussed in this report. This report, therefore, may not include the latest thinking of some of the developers on their process flow diagram and design specifications.

Develop Data Base

At the onset of the project it was recognized that an organized data base on the chemical coal cleaning processes was essential to formulate meaningful conclusions regarding the performance of various processes, their developmental stage and their economics. Therefore, an initial objective of this phase was to prepare a "Process Information Form" to

serve as a check list for obtaining and recording important technical and economic data. Emphasis was placed on completing the form, to the extent possible, from available in-house information prior to contacting the process developers or knowledgeable personnel on each process for missing information. The use of these forms prevented the inadvertent omission of important process details during phone contacts and personal interviews. The completed forms, also, served as the principal source of data for process evaluation work.

Process and Cost Information Analysis

Subsequent to data base compilation, the major tasks involved in data analysis were:

- To review the collected data on individual processes for adequacy and probable accuracy;
- To develop or compile tabular and graphical representations of available data on each process in a format to allow the formulation of meaningful conclusions; and
- To identify critical process parameters and prepare a list of criteria for process and cost evaluation work.

Engineering judgement was used in selecting or developing schematic flow sheets for processes on which adequate experimental data are unavailable. The process economics prepared for these processes are based on preliminary conceptual processing schemes and, as such, are engineering estimates. The process operating conditions, the process chemistry, the levels of removal of mineral and organic sulfurs, the heating value and yield recovery information are based on Versar's evaluation of the individual developer's claims. These are reported in appropriate sections of this report.

Where cost information was supplied by a developer, these costs were utilized, to the extent possible, as the basis of the cost information in this report. However, these costs were modified to allow the evaluation of the various processes on a comparable basis. Cross-checks were made whenever information was available from different sources.

Cost Basis--

Interest costs and equity financing charges--Capital investments involve the expenditure of money which must be financed either on borrowed money or from internal equity. Estimates for this study have been based on 10 percent cost of capital, representing a composite number for interest paid or return on investment required.

Time index for costs--All cost estimates are based on First Quarter 1977 prices and, when necessary, have been adjusted to this basis using the Chemical Engineering or the Marshall and Stevens Cost Indices.

Useful service life--The useful service life of process equipment varies depending on the nature of the equipment and process involved, its usage pattern, maintenance care and numerous other factors. Individual companies have their own service life values based on actual experience and use these internal values for amortization. Another source of service life information, less relevant than company experience, is the Internal Revenue Service guidelines. A useful service life of 20 years was used for all equipment in this study.

Capital costs--Capital costs are defined for the purposes of this report as all front-end loaded, out-of-pocket expenditures for the provision of the coal cleaning facilities. These costs include land, equipment construction and installation, buildings, services and engineering costs.

When capital costs were known for a specific coal cleaning technology, cost adjustment to the typical plant size was made using an exponential factor of 0.65.

Contingencies--A contingency allowance of 20 percent is added to installed capital cost in all estimates, with the exception of TRW's. A lower contingency allowance (10 percent) was used for the latter process since it is at a more advanced stage of development and adequate process data were available to develop the economics of this process with a greater degree of confidence.

Capital equipment amortization--It is assumed that regardless of tax and depreciation considerations, a plant operator would probably finance and amortize a chemical coal cleaning plant by means of an equal-payment, self-liquidating loan or its equivalent. If the loan is payable with equal installments, the amount due per period per dollar of loan as a function of the loan period and interest rate is given by:

$$R = \frac{i (1+i)^n}{(1+i)^n - 1}$$

where R = capital recovery per period per dollar invested

i = interest rate per period expressed as a decimal

n = number of periods in the amortization schedule

As mentioned above, all annual capital recovery costs were calculated based upon a 20-year lifetime and a 10 percent interest rate. The capital recovery factor is then

$$R = \frac{(0.1)(1.1)^{20}}{(1.1)^{20} - 1} = 0.1175$$

Operating expenses--Annual costs of operating a coal cleaning facility include labor, supervision, labor additive and support costs; maintenance cost, taxes and insurance costs; power, water and steam costs; raw coal and chemical costs and refuse disposal cost.

● Labor, supervision and labor additive and support costs--Where not provided by the process developer, the following costs were used:

Direct Labor (DL)	\$14,400/man year
Supervision (SL)	\$19,200/man year
Labor Additives (LA)	@ 30 percent (DL + SL)
Services & Support	@ 20 percent (DL + SL + LA)

● Maintenance cost--Where not provided by the process developer, maintenance is taken as 5 percent of total invested capital.

● Taxes and insurance costs--Taxes and insurance are taken as 2 and 1 percent, respectively, of the total invested capital.

● Power, energy and utilities costs--Where not provided by the process developer, costs for power, energy and utilities were taken as:

Electric power, mil/kwh	25
Cooling water, \$/1,000 liters (\$/1,000 gal)	0.013(0.05)
Process water, \$/1,000 liters (\$/1,000 gal)	0.066(0.25)
Steam (110 to 220 psia), \$/1,000 kg (\$/1,000 lb)	8.81(4.0)

For the IGT process, since the process is at a very early stage of development and adequate process data were unavailable to estimate the electric power and water requirements, these costs were taken as 5 percent of the raw coal cost.

Where appropriate, product coal or raw coal has been used to supply in-process fuel needs or for generating steam for in-process use.

● Raw coal and chemical costs--The cost of one ton raw coal input to each cleaning plant was taken as \$27.6 per metric ton (\$25/ton). Chemical costs are, however, variable for each individual process. Where the total cost of the chemicals was not provided by the process developer, individual costs listed below were used to estimate this cost.

Lignin sulfonate binder, \$/kg(\$/lb)	0.13(0.06)
Lime, \$/kkg(\$/ton)	38.5(35.0)
Iron carbonyl, \$/kg(\$/lb)	0.222(0.101)
Liquid ammonia, \$/kkg(\$/ton)	143(130)
Chlorine, \$/kkg(\$/ton)	38.6(35.0)
Oxygen, \$/kkg(\$/ton)	27.6(25.0)
Nitrogen dioxide, \$/kkg(\$/ton)	220(200)
Caustic soda, \$/kkg(\$/ton)	176(160)

For the IGT process, these costs were taken as 5 percent of the raw coal cost.

● Cost for solid waste disposal--For those processes which generate quantifiable amounts of solid waste, the cost for disposal was assumed to be \$1.1 per metric ton (\$1.0/ton).

Process and Cost Comparison

Sulfur removal level, heating value recovery potential, capital investment and operating cost comparisons were made between competing processes. These are discussed in Section 4.

SECTION 2

SUMMARY

Chemical coal cleaning processes are still under development. None of these processes have been tested in units larger than eight metric tons per day and only one process even at that size. Consequently, performance and cost comparisons are relatively uncertain at this time. The chemical coal cleaning processes vary substantially in their approach, because of the large number of possible reaction mechanisms and chemicals which can be used to effect removal of sulfur and other reactive impurities in coal. Most chemical processes remove over 90 percent of the pyritic sulfur and several remove up to 40 percent of the organic sulfur as well.

Twenty-nine chemical coal cleaning processes were identified during this technology overview study. Eleven U.S. developed processes are classified as major processes; seven U.S. and Canadian processes are classified as minor processes due to their early stage of development or inactive status. The remaining eleven U.S., Japanese and Australian processes are judged to deserve no further consideration. These processes are listed in tables later in this section. Other processes of importance may exist, but have not been identified in the extensive search conducted in this study.

The eleven major chemical coal cleaning processes exhibit a great deal of diversity with respect to such variables as:

- type of coal successfully desulfurized
- degree of coal crushing and grinding prior to chemical processing
- state of process development
- process chemistry
- major process steps
- kinds and amounts of sulfur removed
- prospects for technical and economic success

Table 1 shows a listing of the major processes and briefly summarizes some of the above factors. The first four processes listed (Magnex, Syracuse, TRW, and Ledgemont) will remove pyritic sulfur only; the remaining seven processes (ERDA, GE, Battelle, JPL, IGT, KVB, and ARCO) claim to remove most of the pyritic sulfur and varying amounts of organic sulfur. Also, the first two processes are unique in that the coal is chemically pretreated, then sulfur separation is subsequently achieved by mechanical means. The remaining nine processes are more typically chemical in that sulfur compounds in the coal are chemically attacked and converted. A capsule summary of each major process follows.

MAGNEX PROCESS

In this process, dry, pulverized (minus 14 mesh) coal is pretreated with iron penta-carbonyl to render the mineral components of the coal magnetic. Separation of coal from pyrite and other mineral elements is then accomplished magnetically. The process has been proven on a pilot plant scale using the carbonyl on a once through basis. The cost of the Magnex process critically depends on the recycle of iron-carbonyl. It is claimed that iron-carbonyl can be produced on-site from carbon monoxide released in the process. However, the continuous recycle of carbon monoxide to produce low-cost iron-carbonyl requires demonstration. If success is not achieved, the process may prove economically infeasible. Approximately 40 coals, mostly of Appalachian origin, have been evaluated on a laboratory scale. These coals are rich in pyritic sulfur and are thus applicable to this process. For the most part, the process will produce coals which meet State regulations for sulfur dioxide emissions of $4.3 \text{ kg SO}_2/10^6 \text{ kg cal}$ ($2.4 \text{ lb SO}_2/10^6 \text{ BTU}$).

SYRACUSE PROCESS

Coal of about 3.8 cm ($1\frac{1}{2}$ ") top size is chemically comminuted by exposure to moist ammonia vapor at intermediate pressure. After removing the ammonia, conventional physical coal cleaning then effects a separation of coal from pyrite-rich ash. Generally, 50-70% of pyritic sulfur has been removed from Appalachian and Eastern Interior coals, producing coals which meet state regulations for sulfur dioxide emission. Construction of a 36 metric tons

TABLE 1. SUMMARY OF MAJOR CHEMICAL COAL CLEANING PROCESSES

PROCESS & SPONSOR	METHOD	TYPE SULFUR REMOVED	STAGE OF DEVELOPMENT	PROBLEMS	ANNUAL OPERATING COST \$/TON CLEAN COAL INCLUDING COST OF COAL*
"MAGNEX", HAZEN RESEARCH INC., GOLDEN COLORADO	DRY PULVERIZED COAL TREATED WITH FE (CO) ₅ CAUSES PYRITE TO BECOME MAGNETIC. MAGNETIC MATERIALS REMOVED MAGNETICALLY	UP TO 90% PYRITIC	BENCH & 91 KG/HR (200 LB/HR) PILOT PLANT OPERATED	DISPOSAL OF S-CONTAIN- ING SOLID RESIDUES. CONTINUOUS RECYCLE OF CO TO PRODUCE FE (CO) ₅ REQUIRES DEMONSTRATION	40.7
"SYRACUSE" SYRACUSE RESEARCH CORP., SYRACUSE, N.Y.	COAL IS COMMINUTED BY EXPOSURE TO NH ₃ VAPOR; CONVENTIONAL PHYSICAL CLEANING SEPARATES COAL/ASH	50-70% PYRITIC	BENCH SCALE	DISPOSAL OF SULFUR CONTAINING RESIDUES.	37.0
"MEYERS", TRW, INC. REDONDO BEACH, CAL.	OXIDATIVE LEACHING USING FE ₂ (SO ₄) ₃ + OXYGEN IN WATER	90-95% PYRITIC	8 METRIC TON/DAY PDU FOR REACTION SYSTEM, LAB OR BENCH SCALE FOR OTHER PROCESS STEPS.	DISPOSAL OF ACIDIC FESO ₄ & CASO ₄ , SULFUR EXTRACTION STEP REQUIRES DEMONSTRATION	43.4
"LOL" KENNECOTT COPPER CO. LEDGEMONT, MASS.	OXIDATIVE LEACHING USING O ₂ AND WATER @ MODERATE TEMP. AND PRESSURE	90-95% PYRITIC	BENCH SCALE	DISPOSAL OF GYPSUM SLUDGE, ACID CORROSION OF REACTORS	46.9

* RAW COAL COST IS INCLUDED AT \$25/TON.

TABLE 1. SUMMARY OF MAJOR CHEMICAL COAL CLEANING PROCESSES

PROCESS & SPONSOR	METHOD	TYPE SULFUR REMOVED	STAGE OF DEVELOPMENT	PROBLEMS	ANNUAL OPERATING COST \$/TON CLEAN COAL INCLUDING COST OF COAL*
"ERDA" (PERC) BRUCETON, PA.	AIR OXIDATION & WATER LEACHING @ HIGH TEMPERATURE AND PRESSURE	~95% PYRITIC; UP TO 40% ORGANIC	BENCH SCALE 11 KG/DAY (25 LB/DAY) CONTINUOUS UNIT UNDER CONSTRUCTION	GYPSUM SLUDGE DISPOSAL ACID CORROSION AT HIGH TEMPERATURES	51.6
"GE" GENERAL ELECTRIC CO., VALLEY FORGE, PA.	MICROWAVE TREATMENT OF COAL PERMEATED WITH NaOH SOLUTION CONVERTS SULFUR FORMS TO SOLUBLE SULFIDES	~75% TOTAL S	BENCH SCALE	PROCESS CONDITIONS NOT ESTABLISHED CAUSTIC REGENERATION PROCESS NOT ESTABLISHED.	41.8
"BATTELLE" LABORATORIES COLUMBUS, OHIO	MIXED ALKALI LEACHING	~95% PYRITIC; ~25-50% ORGANIC	9 KG/HR (20 LB/HR) MINI PILOT PLANT AND BENCH SCALE	CLOSED LOOP REGENERATION PROCESS UNPROVEN. RESIDUAL SODIUM IN COAL	55.9
"JPL" JET PROPULSION LABORATORY PASADENA, CAL.	CHLORINOLYSIS IN ORGANIC SOLVENT	~90% PYRITIC; UP TO 70% ORGANIC	LAB SCALE BUT PROCEEDING TO BENCH AND MINI PILOT PLANT	ENVIRONMENTAL PROBLEMS; CONVERSION OF HCL TO CL ₂ NOT ESTABLISHED	46.0
"IGT" INSTITUTE OF GAS TECHNOLOGY CHICAGO, ILL.	OXIDATIVE PRETREATMENT FOLLOWED BY HYDRODESULFURIZATION AT 800°C	~95% PYRITIC; UP TO 85% ORGANIC	LAB AND BENCH	LOW BTU YIELD (<55%). CHANGE OF COAL MATRIX	65.8

*RAW COAL COST IS INCLUDED AT \$25/TON.

TABLE 1. SUMMARY OF MAJOR CHEMICAL COAL CLEANING PROCESSES

PROCESS & SPONSOR	METHOD	TYPE SULFUR REMOVED	STAGE OF DEVELOPMENT	PROBLEMS	ANNUAL OPERATING COST \$/TON CLEAN COAL INCLUDING COST OF COAL*
"KVB" KVB, INC. TUSTIN, CAL.	SULFUR IS OXIDIZED IN NO ₂ -CONTAINING ATMOSPHERE. SULFATES ARE WASHED OUT.	~95% PYRITIC; TO 40% ORGANIC	LABORATORY	WASTE & POSSIBLY HEAVY METALS DISPOSAL, POSSIBLE EXPLOSION HAZARD VIA DRY OXIDATION.	47.5
"ARCO" ATLANTIC RICHFIELD COMPANY HARVEY, ILL.	TWO STAGE CHEMICAL OXIDATION PROCEDURE	~95% PYRITIC; SOME ORGANIC	CONTINUOUS 0.45 KG/HR (1 LB/HR) BENCH SCALE UNIT	UNKNOWN	46-58 (ESTIMATES)

*RAW COAL COST IS INCLUDED AT \$25/TON.

(40 tons per day) pilot plant is contemplated. No major technical problems have been reported for this process other than potential problems involving scale-up to pilot plant size.

MEYERS PROCESS

This process is the most advanced of the chemical coal cleaning processes, with an 9 metric ton per day Reaction Test Unit (RTU) in operation. The process removes 80-99% of the pyritic sulfur from nominally 14 mesh top size coal. The process uses an aqueous solution of ferric sulfate and sulfuric acid to effect a chemical leaching at moderate temperatures and pressures, but at rather long holding periods (8-13 hours). Thirty-two different coals have been tested: twenty-three from the Appalachian Basin; six from the Interior Basin; one from Western Interior Basin and two western coals. The Meyers process is more applicable to coals rich in pyritic sulfur, thus about one-third of Appalachian coal could be treated to sulfur contents of 0.6 to 0.9 percent to meet the sulfur dioxide emission requirements of current EPA NSPS. Process by-products are elemental sulfur, gypsum from waste water treatment, and a mixture of ferric and ferrous sulfate, with the latter presenting a disposal problem.

LEDGEMONT PROCESS

The Ledge-mont oxygen leaching process is based on the aqueous oxidation of pyritic sulfur in coal at moderately high temperatures and pressures. The process has been shown to remove more than 90% of the pyritic sulfur in coals of widely differing ranks, including lignite, bituminous coals, and anthracite, in bench-scale tests. However, little, if any, organic sulfur is removed by the process. The process became inactive in 1975 during divestiture of Peabody Coal Company by Kennecott Copper Co. Although not as well developed as the Meyers process, the Ledge-mont process is judged to be competitive in cost and sulfur removal effectiveness. The principal engineering problem in this process is the presence of corrosive dilute sulfuric acid, which may pose difficulties in construction material selection and in choosing means for pressure letdown. The process also has a potential environmental problem associated with the disposal of lime-gypsum-ferric hydroxide sludge which may contain heavy metals.

ERDA PROCESS

The ERDA air and steam leaching process is similar to the Ledgesmont oxygen/water process except that the process employs higher temperature and pressure to effect the removal of organic sulfur and uses air instead of oxygen. This process can remove more than 90% of the pyritic sulfur and up to 40% of organic sulfur in coals starting with minus 200 mesh coal. Coals tested on a laboratory scale include Appalachian, Eastern Interior and Western. The developer's claim is that using this process, an estimated 45 percent of the mines in the eastern United States could produce environmentally acceptable boiler fuel in accordance with current EPA standards for new installations. Effort to date is on a bench scale, but a mini-pilot plant is expected to start up soon. The problems associated with this process are engineering in nature. The major one is associated with the selection of materials for the unit construction. Severe corrosion problems can be expected in this process as the process generates dilute sulfuric acid which is highly corrosive at the operating temperatures and pressures.

G.E. PROCESS

Ground coal (40 to 100 mesh) is wetted with sodium hydroxide solution and subjected to brief (~30 sec.) irradiation with microwave energy in an inert atmosphere. After two such treatments, as much as 75-99% of the total sulfur is converted to sodium sulfide or polysulfide which can be removed by washing. No significant coal degradation occurs. That portion of the process which recovers the sulfur values and regenerates the NaOH is not proven. Work to date is in 100 gram quantities, but scale-up to 1 kg quantities is presently in progress. The process appears to attack both pyritic and organic sulfur, possibly at about the same rate. Appalachian and Eastern Interior coals having wide ranges of organic and pyritic sulfur contents have been tested with about equivalent success.

BATTELLE PROCESS

In this process, 70 percent minus 200 mesh coal is treated with aqueous sodium and calcium hydroxides at elevated temperatures and pressures, which removes nearly all pyritic sulfur and 25-50% of organic sulfur. Test work

on a bench and pre-pilot level on Appalachian and Eastern Interior coals has resulted in products which meet current EPA NSPS for sulfur dioxide emissions. The conceptualized process, using lime-carbon dioxide regeneration of the spent leachant, removes sulfur as hydrogen sulfides which is converted to elemental sulfur using a Stretford process. In addition to being a costly process, there are two major technical problems:

- The feasibility of the closed-loop caustic regeneration feature in a continuous process is as yet undemonstrated; and
- The products may contain excessive sodium residues, causing low melting slags and making the coal unusable in conventional dry-bottom furnaces.

JPL PROCESS

This process uses chlorine gas as an oxidizing agent in a solution containing trichlorethane to convert both pyritic and organic forms of sulfur in coal to sulfuric acid. Since removal of sulfur can approach the 75% level, without significant loss of coal or energy content, products should generally meet current EPA NSPS for sulfur dioxide emissions. To date the process has been tested on a laboratory scale only, on two Eastern Interior coals, however the effort will progress to bench-scale and pre-pilot plant scale in the near future. The project is supported by the Bureau of Mines. There are some potential environmental problems with the process. The trichloroethane solvent is listed by EPA as a priority pollutant in terms of environmental effects. A major drawback is in the need to recycle by-product hydrochloric acid for conversion to chlorine. At a chlorine consumption rate of 250 kg per metric ton of coal, the incorporation of a Kel-Chlor unit in the JPL system will add approximately \$10/metric ton of coal. This may be a difficult economic problem for the JPL process to surmount.

IGT PROCESS

This process employs essentially atmospheric pressure and high temperatures [about 400°C (750°F) for pretreatment and 815°C (1500°F) for hydro-desulfurization] to accomplish desulfurization of coal. These high temperatures cause considerable coal loss due to oxidation, hydrocarbon

volatilization and coal gasification, with subsequent loss of heating value. Experimental results have indicated an average energy recovery potential of 60% for this process. The treated product is essentially a carbon char with 80-90% of total sulfur removed. Most of the experimental work to date has been accomplished with four selected bituminous coals with a size of plus 40 mesh. Present effort is on a bench-scale level. The net energy recovery potential of the system and the change in the coal matrix by the process have been identified as possible severe problems for the IGT process. The process must be developed to a stage where the process off-gas can be satisfactorily utilized for its energy and hydrogen content. If this cannot be technically and economically accomplished, the process will prove to be inefficient and too costly for commercialization.

KVB PROCESS

This process is based upon selective oxidation of the sulfur constituents of the coal. In this process, dry coarsely ground coal (plus 20 mesh) is heated in the presence of nitrogen oxide gases for the removal of a portion of the coal sulfur as gaseous sulfur dioxide. The remaining reacted, non-gaseous sulfur compounds in coal are removed by water or caustic washing. The process has progressed through laboratory scale, but was discontinued recently for lack of support. Laboratory experiments with five different bituminous coals indicate that the process has desulfurization potential of up to 63 percent of sulfur with basic dry oxidation and water washing treatment and up to 89 percent with dry oxidation followed by caustic and water washing. The washing steps also reduce the ash content of the coal.

In cases where dry oxidation alone could remove sufficient sulfur to meet the sulfur dioxide emission standards, this technology may provide a very simple and inexpensive system. Potential problem areas for this system are:

- oxygen concentration requirements in the treat gas exceed the explosion limits for coal dust, and thus the application of this process may be hazardous.
- Nitrogen uptake by the coal structure will increase NO_x emission.

ARCO PROCESS

Little information is available on this process. It is presently in the pre-pilot plant stage of development and is alleged to remove both pyritic and organic sulfur. The process was wholly funded internally until recently, when EPRI financed a study on six coals in which there was a wide distribution of pyrite particle size. Energy yield for the process is alleged to be 90-95%, and ash content can be reduced by as much as 50%.

SUMMARY OF MINOR AND MISCELLANEOUS PROCESSES

Tables 2 and 3 summarize process information on the minor and miscellaneous chemical coal cleaning processes.

TABLE 2 . PROCESS INFORMATION SUMMARY OF MINOR CHEMICAL COAL CLEANING PROCESSES

Process	Method	Type S Removed	Stage of Development	Problems, Comments	Economics
Colo. Sch. of Mines Res. Inst.	Selective ferrofluid wetting of pulverized coal constituents followed by magnetic separation	Pyritic	Starting lab work	EPRI funded	No data
Jolevil	Unknown	Unknown	Allegedly active & being marketed	Unknown	Unknown
U. of Houston	Hydrothermal alkaline leaching	Pyritic & some organic	Lab scale	Alleges improvement on Battelle process. Internally funded	No data
Ohio State U.	Microbiological oxidation	Pyritic	Lab scale	7+ day process. Internally funded	No data
NRC (Canada)	Oil agglomeration of very fine coal particles leaving rejected pyrites in water slurry	Pyritic	Active	May be especially applicable in recovery of fines	\$2/ton applied to fines recovery
	Microbiological oxidation of pyrite particles to increase hydrophobic properties	Pyritic	Active on lab scale	May make oil agglomeration more efficient	---
W. Ill. U.	Hydrosulfurization in plasma arc	Both	Active, at low level	Seeking funding	No data
Texaco	H ₂ O ₂ oxidation during pipeline transport	Pyritic	Inactive	---	No data

TABLE 2. (continued)

Process	Method	Type S Removed	Stage of Development	Problems, Comments	Economics
U. of Fla.	Gas oxidation/reduction at very high temperature	Both	Inactive	Poor yield; no data since 1975	No data
Methonics, Inc.	Wet hydrogenation	Both	Inactive	Company probably no longer exists	No data
Rare Earth Industries	Rare earths recycled as S-getters during SRC liquefaction	Both	Inactive	Company probably no longer exists	No data
MIT	Catalytic desulfurization of petroleum fractions	Not Given	Active	Not applicable to coals	No data
Gulf & Western	Coal liquefaction via graft polymerization	Not Given	Inactive	Changes coal matrix. Prior ERDA funding now discontinued	No data
New South Wales	H ₂ O ₂ oxidation	Not Given	Discontinued	Method is analytical, not meant to be coal cleaning	No data

TABLE 3. PROCESS INFORMATION SUMMARY OF MISCELLANEOUS CHEMICAL COAL CLEANING PROCESSES

Process	Method	Type S Removed	Stage of Development	Problems, Comments	Economics
U.S. Steel	Fused NaOH @ high temperature	Both	Inactive	Excess Na in product; coal matrix affected	No data
Chemico	Wet oxidation using air @ high temperature and pressure	Both	Inactive	—	No data
ERDA (Laramie)	Leaching using H_2SO_4 or $H_2SO_4 + H_2O_2$	Pyritic	Inactive—prev. on lab basis only	Inactive since 1975	No data
Rutgers	Microbiological oxidation of organic S	Organic	Inactive	Recently discontinued; negative results	No data
Dynatech	Microbiological oxidation	Pyritic	Inactive	Inactive	~\$4/ton (company data)
Kyoto Univ. (Japan)	Cl_2/O_2 wet oxidation	Probably both	Unknown	No answer to our letter of inquiry	No data
Kellogg	High temperature and pressure leaching in KOH solution w/ Fe_2O_3 catalyst	Both	Discontinued	Poor yield; coal matrix altered	No data

SECTION 3

EVALUATION OF CHEMICAL COAL CLEANING PROCESSES

Coal has traditionally been cleaned by sizing and specific gravity separation to reduce the quantity of ash forming mineral constituents, which include pyritic sulfur. However, these physical coal cleaning techniques are only capable of reducing the pyritic sulfur content of the coal, often with a considerable loss of heating value due to a large quantity of fine coal in the refuse from the plant.

A variety of chemical coal cleaning processes are under development which will remove a majority of pyritic sulfur from the coal with acceptable heating value recovery, i.e. 95 percent BTU recovery. Some of these processes are also capable of removing organic sulfur from the coal, which is not possible with the physical coal cleaning processes.

This section presents available technical and economic information of eleven major chemical coal cleaning processes identified during a nine month study. A detailed evaluation is included on each process in a format that identifies:

- Process details;
- Developmental status;
- Technical evaluation, including process potential for sulfur removal, sulfur by-products, process advantages and disadvantages, environmental aspects, research and development needs; and
- Process economics.

The first four processes discussed are capable of reducing only the amount of pyritic sulfur in the feed coal, while the next seven processes are capable of reducing both pyritic and organic sulfur.

TRW MEYERS' CHEMICAL COAL CLEANING PROCESS

Process Description

The Meyers' process, developed at TRW, is a chemical leaching process using ferric sulfate and sulfuric acid solution to remove pyritic sulfur from coal. The leaching takes place at temperatures ranging from 50° to 130°C (120°-270°F); pressures from 1 to 10 atmospheres (15-150 psia) with a residence time of 1 to 16 hours. Process development and optimization studies conducted to date have included a number of alternative processing methods.

Some of the variations which have been tested and considered are:

- Air vs. oxygen for regeneration
- Coal top sizes from 0.64 cm (¼ inch) to 100 mesh
- Leaching and regeneration in the same vessel and in separate vessels
- Removal of generated elemental sulfur by vaporization or solvent extraction.

Current development work is directed toward elemental sulfur recovery by acetone extraction. This system appears to be promising and may prove to be economical. However, since the technical and economic feasibility of this modification has not yet been proven, Versar, with TRW's concurrence, elected to assess their most promising process for fine coals (top size of 8 mesh or finer). This system includes the removal of elemental sulfur with superheated steam. The flow sheet for this preferred system is shown in Figure 1. The diagram includes the four distinct sections² of the process which are described below.

Reaction Circuit—

Crushed coal, with a nominal top size of 14 mesh, is mixed with hot recycled iron sulfate leachant. The mixing is performed in a continuous reactor with about 15 minutes residence time. The wetted coal, having undergone about 10 percent pyrite extraction in the mixer, is introduced into the reaction vessel at about 80 psig and about 102°C (215°F). In this step, about 83 percent of the pyrite reaction takes place under conditions

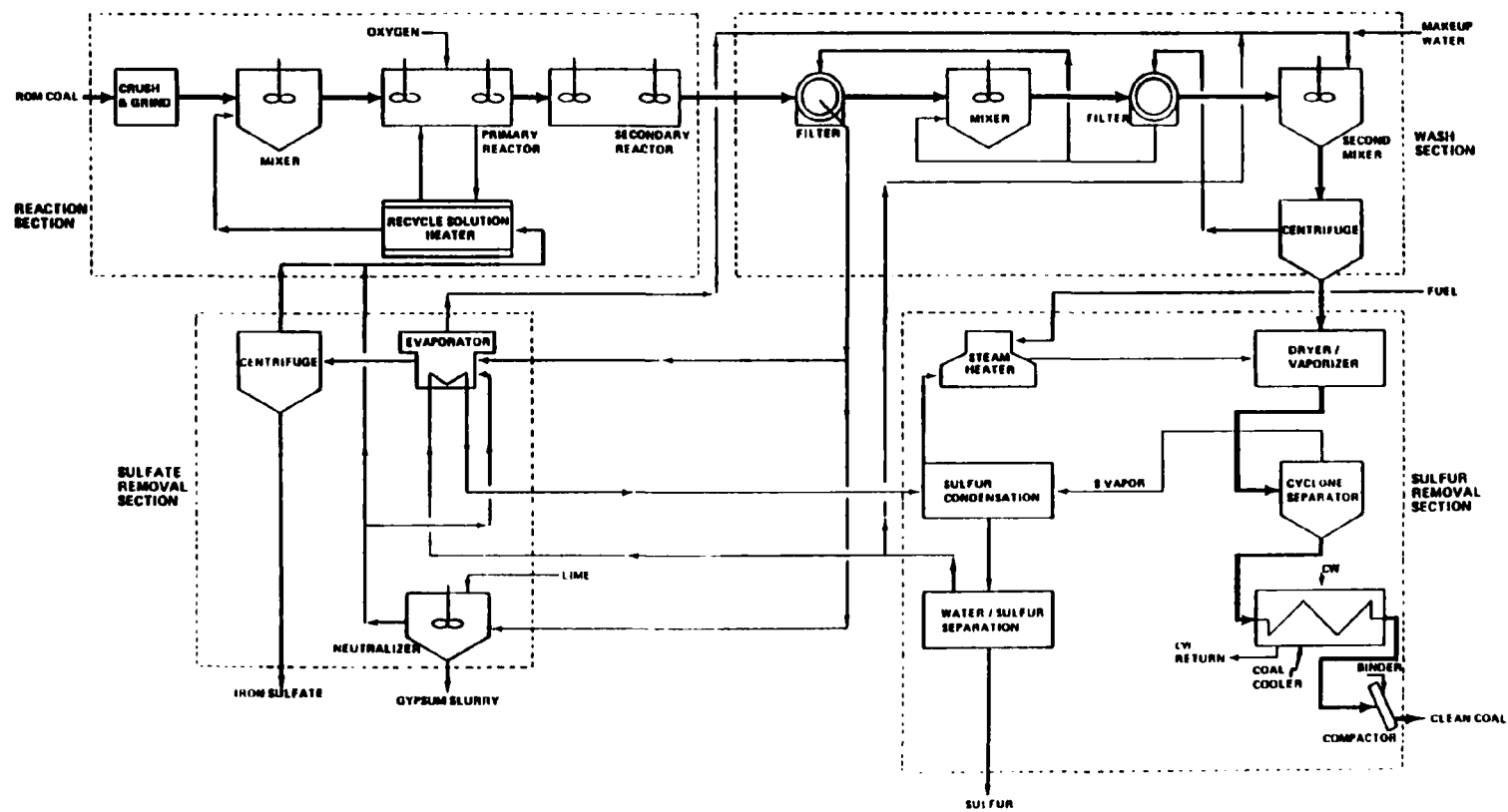


FIGURE 1 TRW (MEYER'S) PROCESS FLOW SHEET

of 5.4 atm. (80 psi) and 118°C (245°F), with varying residence time for different coals. Oxygen from an oxygen plant, which is an integral part of the coal cleaning plant, is simultaneously added to regenerate the leachate. The slurry then moves to a secondary reactor where the reaction continues to about 95% completion.

Wash Circuit—

The iron sulfate leachate is removed from the fine coal in a series of countercurrent washing and separation steps. The slurry from the secondary reactor is filtered and washed with water. Both the filtrate and the wash water are sent to the sulfate removal circuit. The filter cake is reslurried, filtered a second time and then reslurried with recovered clear water and finally dewatered in a centrifuge.

Sulfate Removal Circuit—

The prime function of this circuit is to concentrate the leachate for recycle. The filtrate and the wash water from the first stage filter are fed to a triple effect evaporator which recovers most of the wash water. The by-product iron sulfate crystals which are found in the third evaporation stage are removed from the concentrated leachate and stored or sent to disposal. The remaining wash water from the first filter is partially neutralized with lime to precipitate a gypsum by-product. The partially neutralized wash water is combined with the dilute leachate from the centrifuge and recycled to the process as leach solution.

The fuel requirement of this circuit is equal to a few percent of the product coal. Make-up water is needed to replace water of crystallization and water vaporization losses due to vacuum filters and vacuum evaporator.

Sulfur Removal Circuit—

Wet coal from the centrifuge is flash-dried by high temperature steam which vaporizes both the water and the sulfur. The dry coal is separated from the hot vapors in a cyclone and cooled to give the clean product. The hot vapor from the cyclone is scrubbed with large quantities of recycled hot water from the evaporator. The gas and liquid phases from the gas cooler are separated in a cyclone. The liquid stream from the cyclone which contains water and sulfur is phase separated in a vessel. The gas phase

consisting of saturated steam is compressed, reheated and recycled to the drier.

It is recognized that the processing steps and equipment needed for recovering sulfur from fine or suspended coal sizes would be different from those required for coarser material. The process developer's claim is that coarse coal can be treated in non-pressurized reaction vessels and would employ support equipment which is significantly lower in cost than that necessary for the fine coal system. However, since the coarse coal processing has not been studied to an extent permitting the assessment of its technical feasibility, Versar elected to limit this assessment of the Meyers' process as applied to the fine coal.

Status of the Process

TRW has conducted extensive bench-scale testing of the major treatment units for the Meyers' process.² More than 45 different coals have been tested, and over 100 complete material balances on the process have been calculated and tabulated. The initial bench scale program was directed toward generating critical process data for the chemical removal of pyritic sulfur. This program was aimed at optimizing the leaching and regeneration steps, evaluating analytical techniques and studying other process improvements. From these data the chemistry and rate expressions for the various processing steps have been determined. Additionally, the applicability of the Meyers' process to a variety of coals has been established during a survey program. In this latter study, the process was compared to physical cleaning for thirty-five different coals.^{3,4} It is the developer's claim that in all but two cases the Meyers' process was superior.

In addition to the work under various EPA contracts, TRW has funded independent evaluations of the process by Battelle Columbus Laboratories and Stanford Research Institute. EPA also has funded independent studies done by Exxon, Dow Midland, Dow-Texas and AEC (Oak Ridge). An evaluation of the process has also been done by the University of Michigan for the Electric Power Research Institute. As a result of these extensive studies of the Meyers' process, this chemical coal cleaning process is probably the best characterized process of all the chemical coal cleaning technologies currently underway.

Developmental efforts for this process began in 1969. The bench-scale testing effort generated the data necessary for the design of the eight metric ton/day Reactor Test Unit (RTU). The erection of this unit at the Capistrano Test site was completed in early 1977. With EPA's sponsorship, the RTU started up in June, 1977.

Currently, TRW efforts are directed toward:

- Bench-scale investigations in support of the RTU program on improved techniques for sulfur by-product recovery and on the identification and evaluation of process modifications with potential for reducing processing costs; and
- Testing the RTU. The unit has been run with coal slurry and plans are to introduce the leachate in the circuit in the near future.

The RTU is designed to handle coal less than 0.32 cm (1/8 inch) in size and variable test parameters of temperature, pressure, residence time and oxygen concentration. Limited ability to filter and wash the coal to remove the spent leachate is also included. This unit does not have the capability to remove the elemental sulfur produced by the leaching reaction or to handle coal particle sizes greater than 0.32 cm (1/8 inch).

The first ten months of operation of the RTU will be dedicated to treatment of two types of coal from the Martinka mine. It has been established that this coal will not meet the current NSPS SO₂ emission standards by physical coal cleaning techniques. The specific samples have been selected in cooperation with American Electric Power Service Corp. (AEP), which has elected to participate in this program for cleaning the Martinka mine coal to an acceptable fuel.

The selected coals will be treated in the RTU for the purposes of removing the pyritic sulfur. The treated coal will be washed and filtered to remove the iron salts leaving a wet filter cake (17 to 28 percent moisture by weight) containing some elemental sulfur. The product coal from this operation will be sent to various equipment suppliers to dry the coal and recover the elemental sulfur.

Extensive investigations are projected to optimize this process technically and economically. Some of the studies projected are:

- Pelletizing the powdered product coal by compaction, without binder, to sizes greater than 0.95 cm (3/8 inch) to permit shipping in open hopper cars.
- Determine the effects of desulfurized coal on combustion and performance characteristics of utility boilers.
- Determine the effects of desulfurized coal on performance characteristics of electrostatic precipitators employed to remove particulates from the boiler flue gas.

Technical Evaluation of the Process

This process has been extensively studied and is currently on an eight metric ton/day pilot plant stage. Thus, an assessment of its industrial potential is possible at this time. Only pyritic sulfur is removed by this process. As such, the process is more applicable to coals rich in pyritic sulfur. These coals are found in the Appalachian region of the United States which now supplies about 60 percent of the current U.S. production. An estimated one third of Appalachian production can be treated to a level permitting the burning of the product in conformance with the new source sulfur dioxide emission standards. Some Interior Basin coal can also be treated by this process to meet the new sulfur dioxide emission guidelines.

A Meyers' treatment plant can be either located at a centralized processing site or at a power plant site. If the treatment plant is located at a large power plant site, steam and power requirements might be purchased. This could result in some cost savings. Furthermore, the Meyers' processing plant can operate steadily with shutdowns only for required or scheduled normal maintenance. Thus, the plant would only have to be designed to furnish sufficient coal for the power plant's average load factor, which is, in general, 60 percent of the full name plate capacity. Additionally, capital and operating costs for such a plant would be even more favorable if the process were integrated with coal-fired power generating facilities which would already have included adequate raw coal handling, crushing, pulverizing and fine coal handling facilities. In some instances, when the treatment plant is added to a plant with a very large coal demand, it is possible that the entire operating cost of the system can be absorbed by the power plant due to improved product yield.

Another option for the Meyers' processing plant, which is potentially attractive, is a combination physical and chemical cleaning operation. In this case, the run-of-mine coarse coal containing high ash and high pyritic sulfur would be fed to a physical cleaning plant to reduce the ash content of the coal by about 75 percent. The ash discard consisting of about 15 percent of the ROM coal will contain primarily ash and 10 to 15 percent pyritic sulfur. The low ash coal can then be fed to a gravity separation system. The heavy fraction from the float/sink system, consisting of 40 to 50 percent of the total coal, will be used as feed to the Meyers' process. This latter fraction, containing high concentration of pyritic sulfur, will be reduced to 14 mesh top size and fed to a fine coal Meyers' circuit to yield a product with a very low sulfur content. The desulfurized sample may then be recombined with the float fraction giving an overall yield of about 80 percent on the run-of-mine coal feed. Thus, the combined treated product containing 10-20 percent of the total sulfur of the ROM coal will meet the NSPS standards of sulfur dioxide emission while only processing a fraction of the total coal through the Meyers' process.

Potential for Sulfur Removal—

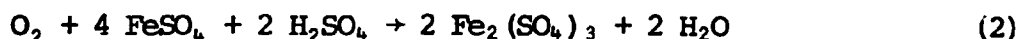
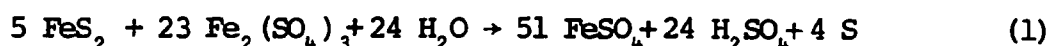
Only pyritic sulfur is removed by this process. A survey program (EPA Contract No. 68-02-0627) has established that this process is able to remove 80-99 percent of the pyritic sulfur (23 to 75 percent of the total sulfur from 23 Appalachian Basin Coals and 91-99 percent of pyritic sulfur (43 to 55 percent of total sulfur) from the six Eastern Interior Basin Coals. Tests with Western coals showed 92 percent removal of the pyritic sulfur (65 percent of total sulfur) from a single Western Interior Basin Coal, and 83-90 percent removal of the pyritic sulfur (25-30 percent of total sulfur) from the two Western coals. Two other Western coals (from Edna and Belle Ayr mines) were also investigated, however, since these coals contain very low pyritic sulfur (0.14 - 0.22 wt%), the results of these tests are inconclusive. Under the same program, tests conducted on float-sink have indicated that conventional coal cleaning at 1.9 specific gravity could reduce only two of the coals tested to a sulfur content as low as that obtained by the Meyers' process.

The results of these investigations are presented in Table 4.^{3,4} Most coals, ground to 100 mesh x 0, were found to give the maximum pyrite removal (90-99 percent). However, several of the coals required 150 and some 200 mesh size reduction to achieve ultimate amounts of pyrite removal. The size reduction also resulted in an increase in the rate of pyrite removal so that, in most cases, the reaction time was reduced considerably.

These studies also provided data which allowed formulation of expressions for pyritic sulfur removal. The kinetic equation developed for lower Kittanning coal is given in Appendix I. Using this equation, the removal of pyritic sulfur was measured as a function of time at 102 °C (215°F) for 18 Appalachian and 3 Eastern Interior region coals. The results presented in Table I-1, Appendix I, indicate that significant pyrite removal rate differences do exist between various coals.

Sulfur By-Products—

The by-products of the Meyers' process are elemental sulfur, a mixture of ferrous and ferric sulfate and calcium sulfate (gypsum) from the wastewater treatment. The by-product chemistry of the process is represented by the treating step, Equation 1, and the solution, regeneration step, Equation 2.²



Once the coal enters the Meyers' reaction vessels, it is not exposed to the atmosphere again until all reactions and washings are completed and the coal is cooled to a point permitting no further emissions of volatile matter. One possible sulfur dioxide (SO₂) emissions source is the scrubber vent gas.

Most of the sulfuric acid (H₂SO₄) is recycled, although some is lost at the filter wash and will be limed and disposed of with solid waste.

Benefit Analysis—

The major benefit associated with the Meyers' process is the removal of pyritic sulfur from pyrite rich coals (primarily Appalachian coals) to a sulfur level consistent with the current standards for sulfur emissions from power plants and industrial sources. TRW investigations indicate

TABLE 4. MEYERS' PROCESS - SUMMARY OF PYRITIC SULFUR REMOVAL RESULTS (100-200 MICRON TOP-SIZE COAL)

MINE	SEAM	STATE	% TOTAL SULFUR W/W IN COAL ^a		MEYER'S PROCESS PYRITE CONVERSION % W/W	MEYER'S PROCESS TOTAL SULFUR DECREASE % W/W	% SULFUR IN COAL ^Δ AFTER FLOAT-SINK
			INITIAL	AFTER MEYER'S PROCESS CURRENT RESULTS			
APPALACHIAN COALS							
KOPPERSTONE NO. 2	CAMPBELL CREEK	W. VIRGINIA	0.9	0.6	92	33	0.8
HARRIS NOS. 1 & 2	EAGLE & NO. 2 GAS	W. VIRGINIA	1.0	0.8	94	23	0.9
WARWICK	SEWICKLEY	PENNSYLVANIA	1.4	0.6	92	54	1.0
MARION	UPPER FREEPORT	PENNSYLVANIA	1.4	0.7	96	50	1.2
MATHIES	PITTSBURGH	PENNSYLVANIA	1.5	0.9	95+	36	1.7
ISABELLA	PITTSBURGH	PENNSYLVANIA	1.6	0.7	96	54	1.5
LUCAS	MIDDLE KITTANNING	PENNSYLVANIA	1.8	0.6	94+	64	0.7
JANE	LOWER FREEPORT	PENNSYLVANIA	1.8	0.7	91	63	0.8
MARTINKA	LOWER KITTANNING	W. VIRGINIA	2.0	0.6	92	70	0.8
NORTH RIVER	CORONA	ALABAMA	2.1	0.9	91	55	2.2
HUMPHREY NO. 7	PITTSBURGH	W. VIRGINIA	2.6	1.5	91	42	1.9
NO. 1	MASON	E. KENTUCKY	3.1	1.6	90	48	2.3
BIRD NO. 3	LOWER KITTANNING	PENNSYLVANIA	3.1	0.8	96+	75	1.5
WILLIAMS	PITTSBURGH	W. VIRGINIA	3.5	1.4	96+	50	2.3
SHOEMAKER	PITTSBURGH	W. VIRGINIA	3.5	1.7	80	51	3.6
MEIGS	CLARION 4A	OHIO	3.7	1.9	93	48	2.8
FOX	LOWER KITTANNING	PENNSYLVANIA	3.8	1.6	89	57	2.0
DEAN	DEAN	TENNESSEE	4.1	2.1	94+	49	3.0
POWATTAN NO. 4	PITTSBURGH NO. 8	OHIO	4.1	1.9	85	53	3.3
ROBINSON RUN	PITTSBURGH	W. VIRGINIA	4.4	2.2	97+	50	3.0
DELMONT	UPPER FREEPORT	PENNSYLVANIA	4.9	0.8	96+	80	2.1
MUSKINGHAM	MEIGS CREEK	OHIO	6.1	3.2	94+	47	4.4
EGYPT VALLEY #21	PITTSBURGH NO. 8	OHIO	6.6	2.7	89	59	4.6
EASTERN INTERIOR COALS							
ORIENT NO. 6	HERRIN NO. 6	ILLINOIS	1.7	0.9	96+	44	1.4
EAGLE NO. 2	ILLINOIS NO. 5	ILLINOIS	4.3	2.0	94	54	2.9
STAR	NO. 9	W. KENTUCKY	4.3	2.5	91+	43	3.0
HOMESTEAD	NO. 11	W. KENTUCKY	4.5	1.7	93	47	3.2
CAMP NOS. 1&2	NO. 9 (W.KY.)	W. KENTUCKY	4.5	2.0	89	55	2.9
KEN	NO. 9	W. KENTUCKY	4.8	2.8	91	42	3.5
WESTERN COALS							
NAVAJO	NOS. 6,7,8	N. MEXICO	0.8	0.6	90	25	---
COLSTRIP	ROSEBUD	MONTANA	1.0	0.6	83	30	---
WESTERN INTERIOR COALS							
WELDON NO. 11	DES MOINES NO. 1	IOWA	6.4	2.2	92	65	3.9

^a DRY, MOISTURE-FREE BASIS^Δ 1.90 FLOAT MATERIAL, 14 MESH X 0, IS DEFINED HERE AS THE LIMIT OF CONVENTIONAL COAL CLEANING⁺ RIN AT 150 X 0

that samples from coal mines in Montana through Iowa, Illinois, Ohio, Pennsylvania, West Virginia and Kentucky, representing a wide range of U.S. production, have been desulfurized to meet these standards. Physical cleaning of these coals has, in general, been unable to accomplish similar results without significant coal reject losses. Based on studies conducted by University of Michigan and Exxon, the net heat energy recovery for the Meyers' process is 87 to 92 percent.

It has been concluded that with Appalachian coals, little or no reaction of the reagents with the coal matrix occurs. Thus, it is expected that the Meyers' processing of the coals will not change the fluidity of the slag which could cause fouling of heat-receiving surfaces. Additionally, the caking properties of coal will be improved by removal of a portion of the coal ash by the leachate.

It is also expected that the combustion properties of the coal will remain unchanged. However, it is anticipated that the Meyers' processing may affect fly-ash resistivity, by reducing the efficiency of electrostatic precipitators (ESP) used for dust control purposes. The addition of conditioners, in small quantities, may be required in order to improve this efficiency. However, it is claimed that there is still a reasonable chance that the ESP's may operate in a normal fashion due to the presence of small quantities of iron sulfate in the coal which may decompose to produce sulfur trioxide necessary for conditioning. Studies on the combustion characteristics of the treated coal and related effects on ESP's are in the planning stage.

A study has been conducted with respect to the trace elements extracted and the degree of extraction achieved by this process. Fifty coal samples have been analyzed in duplicate or triplicate for 18 trace elements which are of interest to the Environmental Protection Agency. The samples included 20 "as received", 20 Meyers' process, and 10 float sink treated coal samples. The conclusions drawn from the study follow:"

- As, Cd, Mn, Ni, Pb, and Zn are removed to a significantly greater extent by the Meyers' process,

- F and Li are partitioned to the refuse a greater amount by physical separation procedures,
- Ag and Cu are removed with a slight preference for float-sink separation, and
- Cr and V are removed by both processes with equal success.

The data on six elements, B, Be, Hg, Sb, Se, and Sn yielded negative or inconclusive results. Figure 2 depicts the trace element removal data.⁹

No analysis has been conducted to determine the nitrogen content of the treated product. However, it is anticipated that the nitrogen content of the feed coal will be either unaffected by this process or slightly increased due to nitrogen used for coal blanketing.

The Meyers' process is a more efficient pyritic sulfur removal method than high gravity physical cleaning. However, it is obviously more complicated and therefore more expensive. Although the process is chemically efficient, it has drawbacks: (1) the long residence time, (2) the difficulty of washing the iron sulfate out of the coal, (3) the need for an extraction to remove elemental sulfur, (4) the process is limited in application due to removal of the pyritic sulfur only, and (5) the generation of 2.65 weight percent iron sulfate waste for each one percent by weight pyritic sulfur removed which needs to be treated prior to disposal. The elemental sulfur and gypsum by-products from the Meyers' process are in a relatively compact form and are manageable (0.40 and 0.54 weight percent, respectively, on coal feed, per one percent pyritic sulfur removed).

Environmental Aspects—

The major environmental problem associated with this process is the disposal of a large quantity of iron sulfate by-product which is acidic and highly corrosive. Treatment of this waste and the recovery of sulfuric acid may be required to provide an environmentally sound solid waste material for disposal. Additionally, the ferric sulfate dissolves a small amount of coal ash. Since the ash is rejected from the process with the iron sulfate, this solid waste will contain some traces of heavy metal salts. The quantity of trace metals rejected will depend upon the coal, as will the nature of sludges and the ratio of coal to waste.

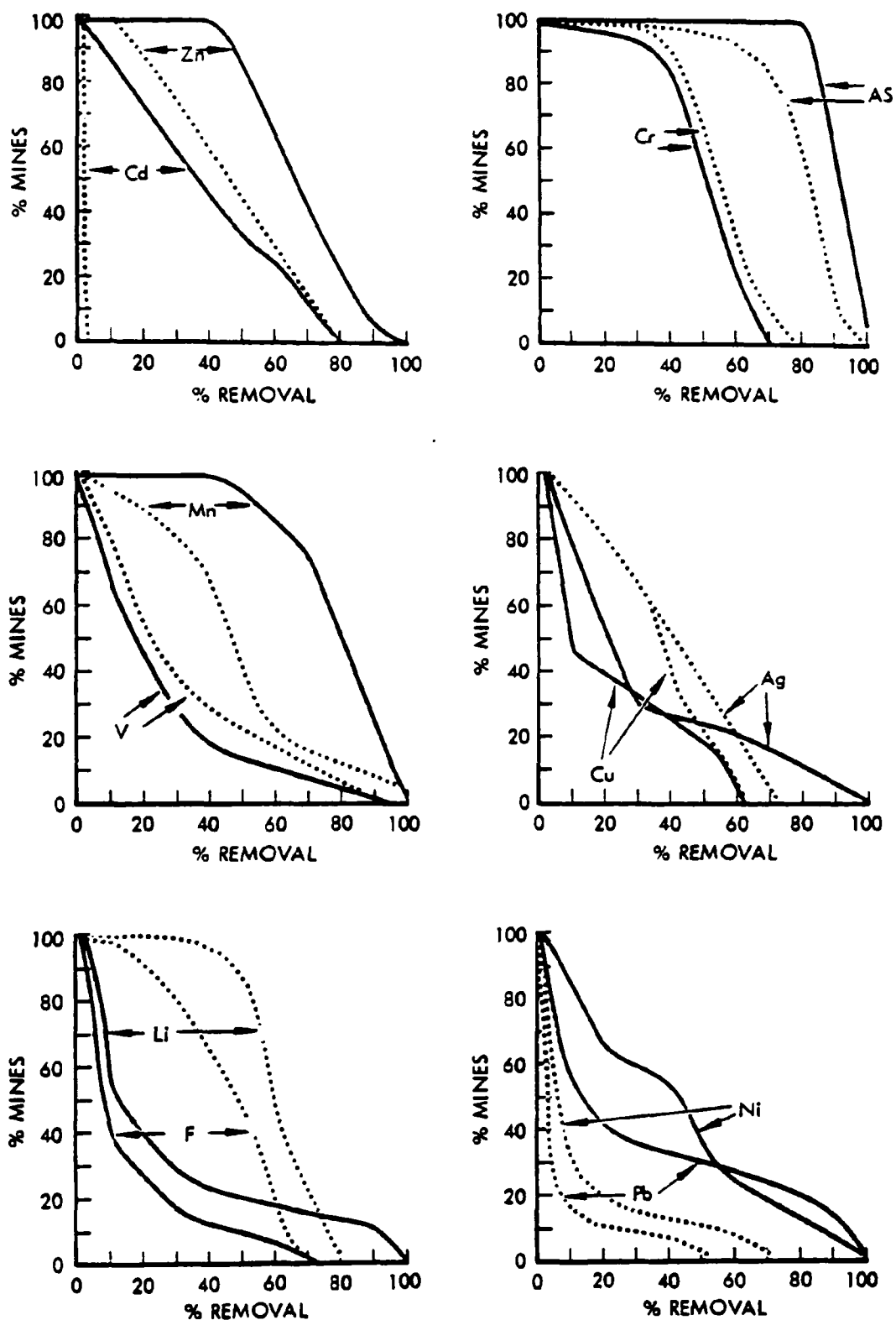


FIGURE 2 MEYERS' PROCESS VS. PHYSICAL COAL CLEANING: TRACE ELEMENT REMOVAL DATA

The iron sulfate waste is dewatered prior to disposal. However, it should be rendered essentially insoluble to yield an environmentally safe material. Several techniques have been suggested to accomplish this:

- Conversion of ferrous sulfate to basic iron sulfate;
- Roasting to iron ores and producing a concentrated stream of SO_2 ; and
- Direct treatment with lime to produce calcium sulfate and iron oxides which are both relatively insoluble.

The elemental liquid sulfur which is removed during the coal drying stage may be cast into blocks and stockpiled or sold where a market exists. The gypsum by-products can be dewatered and disposed of by standard acceptable practices.

The only water that leaves a Meyers' process plant is low pressure steam that is vented to the atmosphere and is environmentally acceptable.

One possible sulfur dioxide emission source from this process is from the vent gas scrubber which is incorporated in this system for the removal of traces of acid mist. This emission is expected to be primarily oxygen containing about ten percent SO_2 and organics.

Problem Areas—

The disposal of by-product generated by the Meyers' process is the main problem area for this process. Handling of this material has to be determined for each commercial plant since their quantity depends upon the coal feed. The quality and potential saleability of by-products are unknowns.

The sulfur recovery system, by superheated steam, is yet unproven. Water and sulfur vapor discharge from the gas/solid cyclone separator without some coal fines is unlikely. Any coal fine carry over from this operation will hinder the subsequent water/sulfur phase separation operations. Furthermore, the by-products, hot water and sulfur, will be contaminated with coal.

R&D Efforts and Needs—

Specific research efforts and needs for the Meyers' process are summarized below:

- A new and promising Meyers' system combines the coal drying and sulfur recovery operations. Additional experimental work, both in bench and pilot scale is required to properly optimize and demonstrate this recovery circuit.
- Bench-scale testing should continue in support of the RTU for identification of process improvements necessary for overall process optimization. It is recommended that this investigations be primarily aimed to improve the techniques of elemental sulfur removal. Additionally, since it is recognized that a large percentage of the total equipment cost is due to the reactor/regeneration circuit, any process improvement in this section will affect the process cost favorably.
- Studies should be conducted to define the combustion behavior of the treated coal and to evaluate the pollutant emissions from the burning of the product coal.
- The effect of the treated product on the operational efficiency of electrostatic precipitators must be evaluated.
- The characteristics, resource recovery and treatment alternatives of the iron sulfate waste material must be investigated to provide a more manageable material for disposal.
- The feasibility of coarse coal processing in unpressurized systems should be further investigated to permit a better assessment of sulfur removal potential and leaching residence time of the coarse coal system.

Process Economics

A variety of organizations have made cost evaluations of the various Meyers' process options.^{5,6,7 and 8} The range of operating costs on an annual basis estimated by various organizations is \$13 to \$19 per metric ton of clean coal. In most cases, these costs were developed based on lowering the sulfur level of a given coal to a level meeting the current NSPS sulfur dioxide emission standard of 2.16 kg. per million kg cal

(1.2 pounds per million BTU). In one case (the Bechtel study⁸) costs were based on an arbitrary 90 percent removal of pyritic sulfur. In 1975, the economics of various process options were analyzed in detail by TRW for EPA under Contract No. 68-02-1336.²

The economic estimates presented herein are based on a plant which processes 300 metric tons/hr (7,200 metric tons/day) [330 tons/hr (8,000 tons/day)] coal. The coal, which is assumed to have 3.2 percent pyritic sulfur and 2 percent moisture, is processed to remove 95 percent of the pyritic sulfur. This information is based on the most recent (1975) TRW flow scheme and economic evaluation for the fine coal processing system; however, it assumes a grass roots plant which includes off-site facilities such as grinding and handling, product compacting, office buildings, rail facilities, etc.

The detailed flow sheet for the battery limit plant is given in Appendix I. The corresponding mass balance and stream properties are also given in Appendix I. The mass balance shown in Appendix I represents only one of the three trains required for processing 300 metric tons/hr (330 tons/hr) coal. TRW has determined that a 100 metric tons/hr (110 tons/hr) operation is about the maximum size for a single train based on available commercial equipment. It has been assumed that the plant will operate 24 hours per day and 8,000 hours per year.

A summary pertinent to the coal balance, based on TRW generated mass balance, is given in Table 5. The other raw materials, utilities and waste streams have been expressed as a function of the product coal, less moisture, in Table 6. The ash loss is taken as the initial pyritic content less the pyrite and iron and sulfur containing residual salts left from the reacted pyrite, without correcting for the oxygen component of the sulfates.

The major equipment for each battery limit process train is given in Appendix I. A summary of economics for the TRW process is given in Table 7. Details on the installed capital costs for this process are given in Table 8. Details on the corresponding estimated annualized operating costs are presented in Table 9. No credits have been given to any by-product (elemental sulfur and gypsum). The unit operating costs shown are based on a coal yield

TABLE 5. MEYERS' PROCESS COAL BALANCE

	One Process Train-		Three Process Trains	
	<u>Metric tons (tons) per hr.</u>		<u>Metric tons (tons) per yr.</u>	
Coal feed				
Coal (MPF)	85.26	(94.00)	2,046,192	(2,256,000)
Pyrite (FeS_2)	<u>5.44</u>	<u>(6.00)*</u>	<u>130,608</u>	<u>(144,000)</u>
Sub-total	90.70	(100.00)	2,176,800	(2,400,000)
Fuel ^Δ				
Coal (MPF)	3.63	(4.00)	87,072	(96,000)
Iron and sulfur compounds	<u>.01</u>	<u>(0.01)</u>	<u>218</u>	<u>(240)</u>
Sub-total	3.64	(4.01)	87,290	(96,240)
Product [†]				
Coal (MPF)	81.63	(90.0)	1,965,120	(2,160,000)
Iron and sulfur compounds	<u>0.26</u>	<u>(0.29)</u>	<u>6,313</u>	<u>(6,960)</u>
Sub-total	81.89	(90.29)	1,965,433	(2,166,960)
Ash loss, by difference				
Iron and sulfur compounds	5.17	(5.70)	124,077	(136,800)
Product				
Coal, dry basis	81.89	(90.29)	1,965,433	(2,166,960)
Binder	1.22	(1.35)	29,387	(32,400)
Moisture	<u>3.28</u>	<u>(3.61)^α</u>	<u>78,582</u>	<u>(86,640)</u>
Total	86.39	(95.25)	2,073,402	(2,286,000)

* Equivalent to 3.2 weight percent pyritic sulfur

Δ Product coal used as fuel

† Net product without binder

α Assumes 4 percent equilibrium moisture

TABLE 6. Meyers' Process Raw Materials, Utilities and Waste Streams Balance

	<u>Units</u>	<u>Hourly units per process train</u>	<u>Unit Ratio</u>
Product coal, dry basis	metric tons (Tons)	81.9 (90.3)	1.0
Coal received, dry basis	metric tons (Tons)	90.7 (100.0)	1.107
Ash loss	metric tons (Tons)	5.2 (5.7)	0.063
Oxygen, 99.5%	metric tons (Tons)	3.5 (3.9)	0.043
Binder	metric tons (Tons)	1.3 (1.4)	0.015
Fuel coal, dry basis	metric tons (Tons)	3.6 (4.0)	0.044
Power	kw	8,400	93.0
Water*	liters	2,180,000	26,620
Iron sulfate wastes	metric tons (Tons)	7.3 (8.1) ^Δ	0.090
Sulfur by-product	metric tons (Tons)	1.2 (1.3) [†]	0.014
Gypsum	metric tons (Tons)	1.45 (1.6)	0.018
Lime, dry basis	metric tons (Tons)	0.45 (0.5)	0.006

* Includes 36,000 l/min cooling water and 420 l/min process water

Δ Includes 0.9 metric tons/hr water

† Includes 0.1 metric tons/hr coal

of 90 percent and a heating value yield of 94 percent, as estimated by TRW. The single largest cost item is the purchased coal used as feed to the chemical processing plant. It is TRW's claims that based on the current conceptual process designs, a broad spectrum of Eastern coals can be upgraded to meet the current NSPS SO₂ emission standards at about the same costs.

TABLE 7. SUMMARY OF ECONOMICS FOR THE MEYERS' CHEMICAL
COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Gross roots plant installation

90% weight yield, 94% heating value recovery

Installed Capital Cost: \$109,100,000

Annual Operating Costs
on Clean Coal Basis:

\$37,243,000 process cost, excluding coal cost

\$103,243,000 process cost, including coal cost*

\$17.28/metric ton (\$15.67/ton), excluding coal cost

\$47.90/metric ton (\$43.45/ton), including coal cost*

\$2.42/10⁶ kg cal (\$0.61/10⁶ BTU), excluding coal cost

\$6.71/10⁶ kg cal (\$1.69/10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 8. INSTALLED CAPITAL COST ESTIMATE FOR THE MEYERS'
CHEMICAL COAL CLEANING PROCESS

	<u>\$1977 (1st Quarter)</u>
Coal handling and preparation	8,830,000
Desulfurization process costs *	
Reaction section	20,800,000
Wash section	7,460,000
Sulfur removal section	9,600,000
Sulfate removal section	5,550,000
Compacting and product handling	5,120,000
Building and miscellaneous ^Δ	700,000
Utilities (off-sites) [†]	28,330,000
Site development and general ^α	<u>4,530,000</u>
Subtotal	90,920,000
Engineering design @ 10%	9,090,000
Contingency @ 10%	<u>9,090,000</u>
Total Installed Plant Capital (TPC)	109,100,000

* Based on TRW's most recent estimate (1975)

Δ Includes control rooms, plant laboratory, administration building, maintenance shop and stockrooms and stores.

† Off-sites include the following facilities:

- steam generation
- water supply
- process water and potable water
- fire protection
- cooling water
- oxygen-nitrogen plant
- instrumentation

^α Includes railroad facilities for incoming and outgoing cars and loading and unloading facilities for raw materials and loading facilities for by-products waste sulfates.

TABLE 9. ESTIMATED ANNUAL OPERATING COSTS FOR THE
MEYERS' CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	\$12,820,000
Taxes @ 2% TPC	2,180,000
Insurance @ 1% TPC	1,090,000
Labor (direct, indirect, additives, support)	2,322,000
General and administrative @ 1.5% TPC	1,640,000
Maintenance and supplies @ 5% TPC	5,460,000
Utilities:	
Electric power	5,040,000
Water	724,000
Steam & Fuel*	—
Chemicals:	
Binder	4,272,000
Lime	420,000
Waste Disposal	1,275,000
Total Annual Processing Cost	37,243,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$103,243,000

* Heating requirement of the process has been estimated at 3×10^6 kg cal/hr (291×10^6 BTU hr); it is assumed that 11 metric tons/hr (12 tons/hr) of clean coal will be adequate to provide in process heating needs.

LEDGEMONT CHEMICAL COAL CLEANING PROCESS

Process Description

The Ledgemont oxygen leaching process is based upon the aqueous oxidation of pyritic sulfur in coal at elevated temperatures and pressures using a stream of oxygen as the oxidant. The process has been developed by the Ledgemont Laboratory of the Kennecott Copper Corporation. The process was patented in 1976.¹⁰

There has been no R&D effort by Ledgemont on the process since 1975. Based on a series of tests run prior to 1975, the Ledgemont process claims to remove 90% of the pyritic sulfur from a wide variety of bituminous coals with essentially zero organic sulfur removal. The product is suitable for combustion in standard utility boilers, but will meet EPA NSPS for sulfur dioxide emissions only if the organic sulfur level in the coal is 0.7-0.8% or less.

The Ledgemont process as conceptualized, consists of five principal steps:

Coal Preparation—

The raw coal is crushed and ground to a suitable particle size for maximum leaching efficiency. The ground coal goes directly to a slurry tank for mixing with water. Alternatively, the ROM coal may be subjected to physical coal cleaning to remove pyrite and ash, before introduction into the process.

Oxidation Treatment—

The coal slurry is then fed to leaching reactors where essentially all of the pyritic sulfur is oxidized to soluble sulfates and insoluble iron oxide under suitable conditions of temperature, pressures, slurry density, oxygen dispersion, mixing and residence time. The proposed initial reaction is as follows:

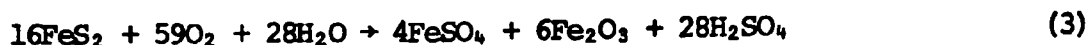


When the process operates at the preferred temperature and pressure [between 50° and 150°C (120° and 300°F), 20 to 25 atm (300 to 350 psig) oxygen pressure], it is claimed that 75 percent of the iron sulfate formed in

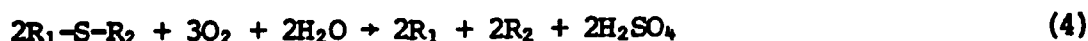
reaction (1) converts to iron oxide, as in equation (2), below:



Accordingly, the overall desulfurization reaction would be:



Some organic sulfur may also be removed by the following reaction:



The Ledgeмонт Laboratory has found that organic sulfur removed in the aqueous oxidation process is highly variable, and depending on the feed coal used, has ranged from 0-20% removal. The inert iron oxide formed in reaction (2) would be removed with the product coal.

Fuel Separation—

The desulfurized coal slurry is partially dewatered and filtered. The filter cake is then water washed.

Drying and Agglomeration—

The washed coal is sent to a suitable drier where water is evaporated leaving a clean, dry solid fuel. This material is then compacted to a suitable pellet size for shipment to a power plant.

Wastewater Treatment—

The acid water overflow from the thickening, filtration and washing steps are sent to a wastewater treatment facility where lime is added, neutralizing the dilute sulfuric acid stream and precipitating any solubilized ash, according to the following reaction:



The mixture of iron hydroxide and gypsum is thickened and filtered with the water overflow being recycled to the leaching process. The gypsum sludge is disposed of in a landfill. Analyses of representative coals which have been treated by the Ledgeмонт process are given in Appendix II.

Table 10 presents Ledgeмонт's current best estimates of key parameters which would be involved in the process design of a continuous system.¹⁹

TABLE 10. Typical Values of Key Parameters in the Conceptual
Ledgeмонт Oxygen Leaching Process for Bituminous Coal ¹²

Operating Factor: 333 days per year
Overall Yield (avg. coal): 97-98%
Net yield after fuel uses: ~90%
Net heating value yield (avg. coal): 93-95%
Pyritic sulfur removal: 90%
Organic Sulfur Removal: 0-20%

<u>Chemical Process</u>	<u>Parameter</u>	<u>Typical Value</u>
Coal preparation	Mesh size	80% -100 mesh
Coal desulfurization	Coal/water in feed	0.2/l
	Reaction time	2 hours
	Temperature	130° C (266° F)
	Oxygen pressure	20 atm. (300 psig)
	Oxygen consumption per metric ton coal feed	0.138 metric ton (0.125 ton)*
Treated coal/water separation system	Thickening:	
	Thickening area required	1 m ² /TPD (11 sq ft/TPD)
	Underflow solid concentration	43% solids
	Filtration:	
	Filtration rate	23 kg/hr/.09 m ² (50 lb/hr/sq ft)
	Percent solids in fuel cake discharge	66%
	Wash water/dry solids	.46/l
Wastewater treatment	Lime addition rate	0.25 T/T coal feed ϕ

* The oxygen demand includes the following:

	<u>metric ton O₂/metric ton coal</u>
O ₂ for pyrite reaction	0.035 [†]
O ₂ for Fe ²⁺ → Fe ³⁺	0.0019
O ₂ uptake by coal	0.054
O ₂ to form CO ₂	0.031
O ₂ to form CO	0.0014
O ₂ lost to flashing	0.0019
Total	0.1252

[†] Based on 2% pyritic sulfur in the coal. The amount of O₂ used in organic sulfur oxidation is unknown.

ϕ This is approximately 8 times the stoichiometric requirement for neutralization.

The process energy efficiency is estimated to be 83-85%. The bulk of the process energy use would be in treated coal drying and in oxygen plant operation. Oxidation of the coal results in conversion of carbon to carbon dioxide and carbon monoxide as well as trace amounts of higher hydrocarbons. Approximately 5-7% of the heating value of the coal is estimated to be lost at the process operating conditions.

Based on the published Ledgemont process information and recent contacts with the Ledgemont Laboratory, a schematic flow diagram for a 7,200 metric tons (8,000 tons) per day coal processing plant is shown in Figure 3. The process removes little or no organic sulfur and 90% of the pyritic sulfur (starting with 2% pyritic sulfur in the raw coal feed).

Status of the Process

The Ledgemont Laboratory of the Kennecott Copper Corporation began work on a process for coal desulfurization in 1970. The R&D effort was carried out in partnership with the Peabody Coal Company - then a wholly owned Kennecott subsidiary. The joint effort culminated in the Ledgemont flow-sheet, the basic features of which have been demonstrated at the bench and semi-pilot scale levels. It is claimed that each step of the process has a complete experimental study to determine the operating range of process variables. Complete reports setting forth the experimental work, process specifications and process economics have been prepared. The entire developmental effort has been internally funded throughout - to the extent of approximately two million dollars.

In 1975, the FTC ordered the divestiture of Peabody Coal by Kennecott, and this resulted in halting further development work on the Ledgemont process. Plans for installing a 1/2 metric ton per day pilot scale desulfurization operation were scrapped and no further R&D work is planned. Kennecott is currently exploring the possibilities of licensing the Ledgemont process.

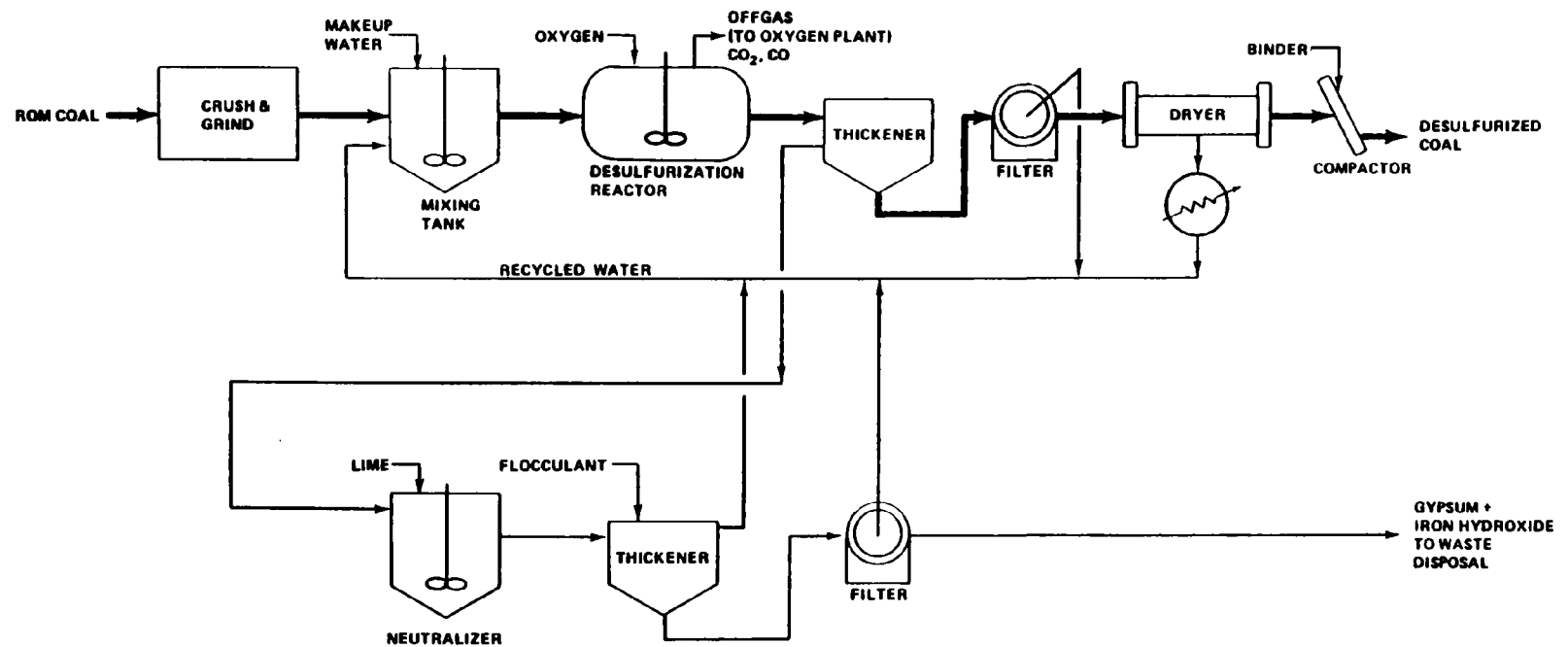


FIGURE 3 LEDGEMONT OXYGEN LEACHING PROCESS FLOW SHEET

Technical Evaluation of the Process

The Ledgemont Laboratory has made available an in-house report containing all of the information made public to date on the process. In addition, the Bechtel Corporation has made a technical and economic study of the Ledgemont process.⁸ A study of this information plus direct contacts with Ledgemont personnel has permitted the following assessment of the process to be made.¹²

Potential for Sulfur Removal—

The Ledgemont process has been shown to remove more than 90% of the pyritic sulfur in coals of widely differing ranks including lignite, high volatile B bituminous, and semi-anthracite, in bench-scale autoclave equipment. Reaction conditions have been standardized at 130° - 132°C (265°-270°F), 20 atm (300 psig) oxygen pressure and two hours residence time. Several bituminous coals including Illinois #6, Ohio #6, and Kentucky, have been treated in "semi-pilot scale" equipment with consistent removal of 90% of the pyritic sulfur. The data on these coals is tabulated in Appendix II. Little, if any, organic sulfur is removed by the process (from 0-20%, depending on coal treated) and there is no credit taken in the conceptual process for this type of sulfur removal.

Sulfur By-Products—

The Ledgemont process produces a gypsum by-product which is unsaleable since it is contaminated with ferric hydroxide.

Benefit Analysis—

The main benefit associated with the process is the demonstrated removal of 90% or better of the pyritic sulfur from a wide variety of coals. Other advantages of this process when compared to processes with a similar leaching mechanism, i.e., ferric sulfate leaching as in the TRW Process, include:

- No elemental sulfur is formed. This eliminates the difficult and expensive sulfur extraction step required after ferric sulfate leaching;
- No regeneration of the leach solution is required;
- Considerably less washing is required because of the lower levels of sulfate ions in the Ledgemont leach solution as compared with TRW

ferric sulfate leach solutions. This has the further effect of reducing the volume of the neutralization tanks; and

- The reaction time in aqueous oxygen leaching is less than one quarter of that required for ferric sulfate leaching. For example, only two hours is required to remove greater than 90% of the pyritic sulfur from Illinois #6 coal, whereas 8-10 hours are required for the average coal in the TRW ferric sulfate process.

The above advantages all have a significant effect on process economics.

Environmental Aspects—

The Ledgeмонт process has one potentially serious environmental problem - disposal of approximately 0.3 metric ton of lime-gypsum-ferric hydroxide sludge per metric ton of coal fed to the process. This sludge is bulky and requires substantial land storage space, and, although it has not yet been determined, the sludge may contain trace elements such as heavy metals which could pose a threat of uncontrolled leaching into ground water.

Problem Areas—

The principal problem areas in this process appear to be associated with the presence of high temperature, 120°C (250°F) dilute sulfuric acid at elevated pressures, 25 atm (350 psia). At these temperatures, the dilute acid (a few percent) is highly corrosive. The presence of this material poses problems in material selection and in choosing means for pressure letdown.

A significant effort will be required to find a suitable material for lining those pieces of equipment which will be exposed to corrosive acid. This would include all equipment involved in the coal desulfurization step from feed-effluent heat exchangers through flash gas scrubber, pumps, heaters, reactors, pressure letdown devices, and treated coal slurry flush tanks.

A possible cladding material suggested for lining the Ledgeмонт reactors is a 60-40 tantalum-niobium alloy, which costs \$154 per kilogram (\$70 per pound) and is as costly as silver. Any further optimization study by Ledgeмонт should include consideration of the lining materials problem.

The second major engineering problem associated with the corrosivity of sulfuric acid is a means to accomplish pressure letdown. Normal valving will probably not withstand the erosion; ceramic equipment may be required.

A possible problem may occur if coal loss through oxidation in production-scale equipment at the elevated temperature and pressures involved is greater than anticipated.

R&D Needs—

In terms of an aqueous coal oxidation process under active development, the Ledgemont process has been superseded in part by the ERDA oxydesulfurization process. The ERDA process is based on a similar oxidative leaching mechanism but claims to effect substantial organic sulfur removal in addition to 90-100% pyritic sulfur removal. The Ledgemont process may effect significant organic sulfur removal at pressures higher than those studied to date.

Process Economics

The Ledgemont Laboratory¹¹ has provided capital and operating costs based on a 7,200 metric tons (8,000 tons) per day coal processing plant, values of key parameters as shown in Table 10 and the process conceptual flow sheet (Figure 3).

A summary of economics of the Ledgemont process is given in Table 11. Details on capital and annual operating costs are presented in Tables 12 and 13, respectively. These costs are presented as received, except that Versar has added a 20% contingency factor to the depreciable portion of the capital investment. Operating cost components are shown both as dollars per metric tons (dollars per ton) of product coal and as dollars per million Kg cal (dollars/10⁶ BTU) heating value. It should be noted that no by-product credit is taken for the 3,600 metric ton (4,000 ton) per day of nitrogen which would be co-generated in the oxygen preparation plant.

TABLE 11. SUMMARY OF ECONOMICS FOR THE LEDGEMONT
CHEMICAL COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Gross roots plant installation

90% weight yield, 94% heating value recovery

Installed Capital Cost: \$114,020,000

Annual Operating Costs
on Clean Coal Basis:

\$45,300,000 process cost, excluding coal cost

\$111,300,000 process cost, including coal cost*

\$21.02/metric ton (\$19.07/ton), excluding coal cost

\$51.64/metric ton (\$46.85/ton), including coal cost*

\$2.94/10⁶ kg cal (\$0.74/10⁶ BTU), excluding coal cost

\$7.23/10⁶ kg cal (\$1.82/10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 12. INSTALLED CAPITAL COST ESTIMATE FOR THE
LEDGEMONT CHEMICAL COAL CLEANING PROCESS² ⁸

	<u>\$ 1977</u>
Coal handling and preparation*	\$14,400,000
Desulfurization process costs	
Reaction equipment	19,700,000
Liquid/solid separation	11,100,000
Neutralization	8,100,000
Drying	4,600,000
Compacting and product handling ^Δ	5,120,000
Building and miscellaneous [†]	--
Utilities (off-sites) ^α	22,400,000
Site development and general	<u>2,300,000</u>
Subtotal	87,700,000
Engineering design @ 10%	8,770,000
Contingency @ 20%	<u>17,530,000</u>
Total Installed Plant Capital (TPC)	\$114,020,000

* Crushing raw coal to -100 mesh and includes site development

Δ Versar estimate

† Included in coal preparation and handling cost

α Includes oxygen plant

TABLE 13. ESTIMATED ANNUAL OPERATING COSTS FOR THE LEDGEMONT
CHEMICAL COAL CLEANING PROCESS

	<u>\$</u>
Amortization 20 years @ 10% interest (factor = 0.1175)	13,400,000
Taxes @ 2% TPC	2,300,000
Insurance @ 1% TPC	1,100,000
Labor (direct, indirect, includes G&A)	1,600,000
General and administrative (included above)	—
Maintenance and supplies	7,300,000
Utilities:	
Electric power	7,000,000
Water	100,000
Steam	3,500,000
Chemicals:	8,200,000
Oxygen	
Lime	
Flocculant	
Binder	
Waste Disposal	800,000
Total Annual Processing Cost	<u>\$ 45,300,000</u>
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	<u>\$111,300,000</u>

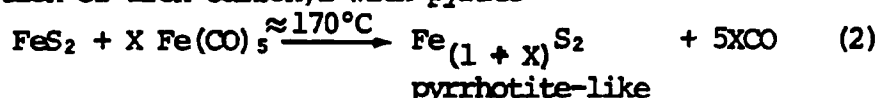
MAGNEXSM CHEMICAL COAL CLEANING PROCESS

The MagnexSM process is a coal beneficiation process which utilizes vapors of iron pentacarbonyl [Fe(CO)₅] to render the mineral components of the coal magnetic. It has been experimentally demonstrated that free iron resulting from decomposition of the pentacarbonyl selectively deposits on or reacts with the surface of pyrite and other ash forming mineral elements to form magnetic materials. Microscopic observations and chemical analyses suggest that for pyrite the magnetic material is a coating of a pyrrhotite-like mineral, while for ash the magnetic material is metallic iron. It has also been demonstrated that the pentacarbonyl does not deposit iron on the surface of coal particles. Reactions suggested for this process are:¹⁴

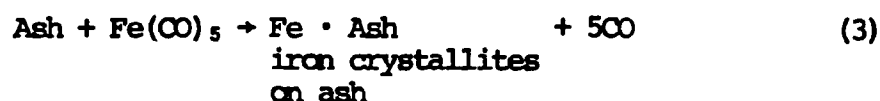
- Iron carbonyl decomposition



- Reaction of iron carbonyl with pyrite



- Reaction of iron carbonyl with ash-forming minerals



Process Description

The process involves four major steps:

- crushing and grinding
- heating and pretreatment
- carbonyl treatment, and cooling
- magnetic separation.

Figure 4 presents a flow diagram for the MagnexSM process as described by the process developer, Hazen Research, Inc., of Golden, Colorado.

Run-of-mine (ROM) coal is crushed to minus 14 mesh and then fed to the thermal pretreating unit where it is heated to about 170°C (365°F) in the presence of steam. The steam and thermal treatment conditions the coal to improve the selectivity of the magnetic coating (increase yield and reduce sulfur content of the coal).

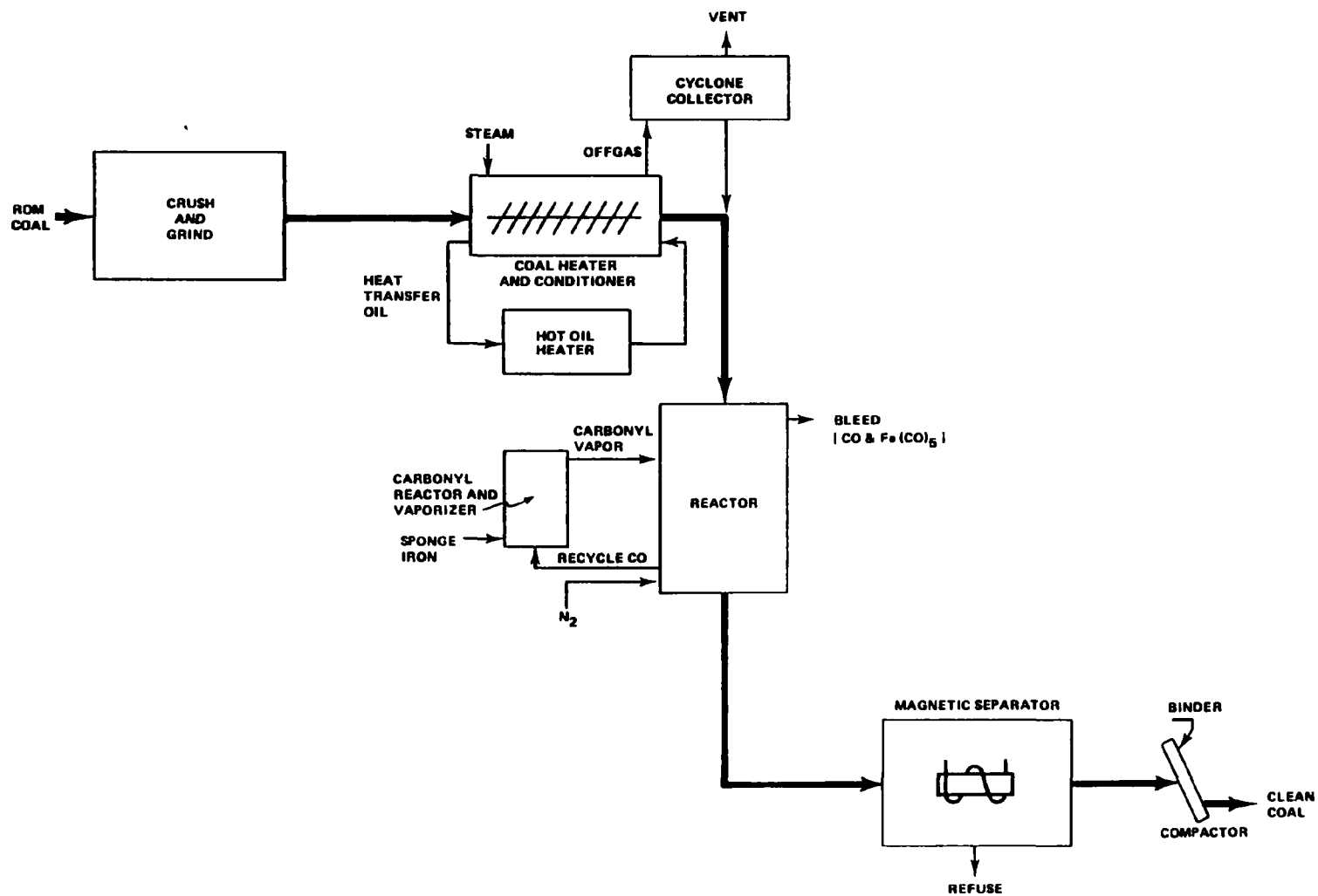


FIGURE 4 MAGNEX PROCESS FLOW SHEET

The heated coal is then gravity fed to the iron pentacarbonyl reaction vessel where it is subjected to the treatment vapors at atmospheric pressure for a residence time of thirty minutes to one hour. The reactor is insulated and maintains the sensible heat of the coal.

The carbonyl treated coal is conveyed to the magnetic separation section. The treated coal passes across three induced magnetic rolls in series. The first roll removes the strongly magnetic minerals and the second and third rolls remove the weakly magnetic minerals. Several commercially available magnetic separators have been evaluated under funding by EPRI. The report will be released in 1978.

After passing through the magnetic separator, the clean coal is conveyed into a storage bin. Some clean coal from the storage may be returned to the CO burner for in-process use; the remaining will be conveyed to the compactor unit. The pelletized coal will be then conveyed to the product storage for subsequent shipment.

The process consumes 1 to 20 kilograms of iron pentacarbonyl per metric ton of coal (2-40 lb/ton), depending on the feed coal; and generates 0.6 to 13.0 kilograms (1.4 to 28.6 lb) of gaseous carbon monoxide (CO) for recycle.

"In the 1977 pilot plant, the CO-rich gas was not recycled to iron carbonyl generation. Rather, it was discharged through a hypochlorite scrubber to remove traces of iron carbonyl". Since the major operating cost for this process is associated with the consumption of the iron pentacarbonyl, it is planned to react the CO-rich gas with iron to produce iron carbonyl on-site. Even with a projected CO recirculation system, a bleed stream may be discharged from the reactor.

Status of the Process

The MagnexSM process has been under development for 30 months. For the first 18 months, the process has been investigated on a laboratory scale, using initially 75 gram samples and later one kilogram samples, on a batch

scale basis. To date about 40 coals, mostly Appalachian in origin, have been tested.¹⁴ The major emphasis of the laboratory work has been on the chemistry of the process. During this study efforts were directed to determine the effects of process variables such as reactor temperature, iron carbonyl requirements and reaction residence time.

"On February 17, 1976, United States Patent #3,938,966 was issued to Hazen Research, Inc. The MagnexSM process is owned by NEDLOG TECHNOLOGY GROUP. NEGLOG plans to continue process development and initiate design, construction and operation of a 54 metric tons (60 tons) per hour demonstration plant. The MagnexSM pilot plant schematics are given in Appendix III.

Start-up operation for the pilot plant was in November, 1976. The coal selected for the pilot plant evaluation was from the Allegheny group of Pennsylvania. This coal was run in the pilot plant during the first quarter of 1977 and was upgraded to meet the current new source sulfur dioxide emission standard of 2.2 kg per million Kg cal (1.2 lb SO₂ per million BTU). Washability studies of this coal had indicated that conventional gravity cleaning would not significantly reduce the sulfur content of the feed coal.

Under funding from the Electric Power Research Institute, a study on the mechanical aspects of magnetic separation of carbonyl treated coal has been conducted. The result of this study is scheduled to be published in the spring of 1978 (EPRI RP-980-1).

At the present, various coal samples are being evaluated in the laboratory stage and research and developmental work is proceeding in the area of iron carbonyl generation.

Technical Evaluation of the Process

The MagnexSM process removes only pyritic sulfur and therefore, it is more applicable to coals rich in pyritic sulfur, which are found in the Appalachian region. The process also reduces the ash content of the coal.

It is claimed that fine coal crushing is not necessary to enable the MagnexSM process to find a wide application in pyrite-rich coal desulfuriza-

tion. The Bureau of Mines prediction curves which correlate pyrite particle size with pyrite sulfur removal do not allow accurate prediction of sulfur reduction for a given coal by the MagnexSM process. These curves are only applicable to gravity coal cleaning techniques. It has been reported that in one test the average pyrite particle size of the minus 14 mesh coal sample was 15 micron. Removal of pyritic sulfur from this sample by the Magnex process was approximately 80 percent; while a 30 percent sulfur removal was predicted for this coal using the Bureau of Mines prediction curves.

Limited published information is available on MagnexSM process test results. A report covering the applicability of this process for desulfurization of coals surveyed may be issued in the future. However, available information is discussed below.

Potential for Sulfur Removal—

Laboratory experiments conducted by Hazen indicate that the MagnexSM process can remove enough sulfur and ash from many Appalachian coals to produce compliance coals. However, significant iron carbonyl consumption rate differences do exist between pyrite removal in various coals (2-40 lb per ton of coal processed).

In a test with coal from the lower Freeport Seam, ash was reduced from 27 to 9 percent and pyritic sulfur from 2.1 to 0.3 percent with a coal product yield at 73 percent. Table 14 presents results from this test.¹⁵ These results were obtained with iron carbonyl addition rate of 2 kilograms per metric ton (4 lb per ton) of coal. In a similar test with a different sample of the same coal, using an iron carbonyl addition rate of 31 kilograms per metric ton (62 lb per ton) of coal, ash was reduced from 27 to 5 percent and pyritic sulfur from 2.1 to 0.2 percent with a product yield of 46 percent. These results indicate that better product quality can be achieved with this process with greater amounts of iron carbonyl addition, but at the expense of the product yield.

TABLE 14. SULFUR AND ASH REMOVAL FROM LOWER FREEPORT
SEAM COAL BY THE MAGNEXSM PROCESS

Results:

<u>Analyses, dry</u>	<u>Clean Coal</u>	<u>Refuse</u>	<u>Calculated Feed</u>
Yield, wt. %	72.7	27.3	—
Ash, %	9.4	73.8	27.0
Pyritic Sulfur, %	0.33	6.88	2.12
Total Sulfur, %	0.88	7.28	2.63
Calorific Value, BTU/lb	13,970	2,997	10,974

Distribution, %

Weight	73	27
Ash	25	75
Pyritic Sulfur	11	89
Heating Value	93	7

Conditions:

Temperature	190-195°C
Time	1 hour
Feed Size	14 by 150-mesh
Iron Carbonyl	4 lb/ton (0.2%)

Table 15 presents a set of results with a Pittsburgh seam coal. In this test ash was reduced from 17.0 to 10.2 percent and pyritic sulfur from 1.6 to 0.56 with a coal product yield of 87.1 percent. These results were achieved with iron carbonyl addition rate of 32 lb/ton. This Pittsburgh seam coal is a metallurgical grade coal and is presently cleaned by heavy media washing for the coarse coal and Deister tables for the fines. In Table 16, results of the conventional gravity cleaning are compared to two test results obtained while processing this feed in the MagnexSM system. As shown, the coal products obtained with carbonyl treatments are superior in terms of sulfur content to that obtained by conventional cleaning techniques.

During the first quarter of 1977 a coal feed from the Allegheny Group of Pennsylvania was evaluated on the MagnexSM pilot plant. Table 17 presents the analysis of the feed coal. Two shipments of this coal were received from the same mine and seam. The ash content of the first shipment was considerably lower than the second (12.7 vs. 18.3 percent); however, the sulfur content of both shipments was the same (0.71 percent inorganic and 0.56 percent organic sulfur). Washability curves presenting specific gravity versus yield, cumulative percent ash float and ash sink, and plus or minus 0.10 specific gravity distribution curve of the ROM pilot feed are given in Figure 5. This plot indicates that at a specific gravity of 1.5 (where 10 percent of the raw coal feed lies within ± 0.10 specific gravity curve) theoretical perfect sink/float cleaning would yield 87.7 percent clean coal containing 9.5 percent ash and 1.13 percent sulfur. While significant ash reduction can be achieved at that specific gravity by sink/float techniques, the resulting coal will not meet the current new source emission standard of 2.2 kg SO₂ per million Kg cal (1.2 lb SO₂ per million BTU).

The results of the laboratory MagnexSM evaluation of the pilot plant feed are presented in Table 18. These data indicate that at 170°C (338°F) and 20 kg of iron carbonyl per metric ton (40 lb/ton) of coal, the clean coal yield was 81 percent with product sulfur content equivalent to 1.82 kg SO₂ per million Kg cal (1.01 lb SO₂ per million BTU).

TABLE 15. SULFUR AND ASH REMOVAL FROM PITTSBURGH SEAM
COAL BY THE MAGNEXSM PROCESS

Results:

	<u>Clean Coal</u>	<u>Refuse</u>	<u>Calculated Feed</u>
<u>Analyses, dry</u>			
Yield, wt. %	87.1	12.9	100.0
Ash, %	10.2	63.1	17.0
Pyritic sulfur, %	0.56	8.65	1.60
Total sulfur, %	1.33	8.88	2.30
Calorific value, BTU/lb	13,655	4,697	12,499
<u>Distribution, %</u>			
Weight	87	13	
Ash	52	48	
Pyritic sulfur	30	70	
Heating value	95	5	
Conditions:			
Temperature	170°C		
Time	1 hour		
Feed size	14-mesh by zero		
Iron carbonyl	32 lb/ton (1.6)		

TABLE 16. SULFUR AND ASH REMOVAL FROM PITTSBURGH SEAM COAL
BY THE MAGNEXSM PROCESS VS. CONVENTIONAL GRAVITY
SEPARATIONS

	<u>Clean Coal</u>		
	<u>Yield, wt. %</u>	<u>Ash, %</u>	<u>Pyritic Sulfur, %</u>
Carbonyl, treatment A ^Δ	87.1	10.2	0.56
Conventional processing	82.0	8.4	0.93
Carbonyl, treatment B [†]	75.3	8.3	0.49
Feed coal, average	—	17.7	1.64

^Δ Carbonyl addition rate 32 lb/ton.

[†] Carbonyl addition rate is greater than 32 lb/ton; however, the exact level is unknown.

SM
TABLE 17. ANALYSIS OF MAGNEX PROCESS PILOT PLANT FEED COAL

Sample Number ^Δ	11089	10442
Ash, wt. %	18.29	12.7
Total sulfur, wt. %	1.27	1.27
Organic sulfur, wt. %	0.56	0.58
Inorganic sulfur,† wt. %	0.71	0.70
Calorific value, BTU/lb	11,980	12,903
Emission, lb SO ₂ /10 ⁶ BTU	2.12	1.97

Δ Two shipments of coal were received. Although they were from the same mine and seam, the ash content was significantly higher in 11089.

† Inorganic sulfur = pyritic + sulfate.

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TABLE 18. SUMMARY OF LABORATORY EVALUATION OF MAGNEX PROCESS PILOT PLANT FEED COAL*

	Units	Test Numbers		
		A	B	C
Carbonyl treatment				
Temperature	°C	170	170	170
Dosage	lb/ton	2.5	10	40
Clean coal				
Yield	%	96.4	86.4	81.0
Ash	%	11.6	11.8	10.7
Total sulfur	%	1.08	0.89	0.66
Inorganic sulfur	%	0.34	0.24	0.09
Heating value	BTU/lb	12,992	12,964	13,160
Emission	lb SO ₂ /10 ⁶ BTU	1.66	1.38	1.01

* Feed coal was 10442, minus 14-mesh, 1.27% total sulfur, 0.71% inorganic sulfur, 12.7% ash, 12,736 BTU/lb.

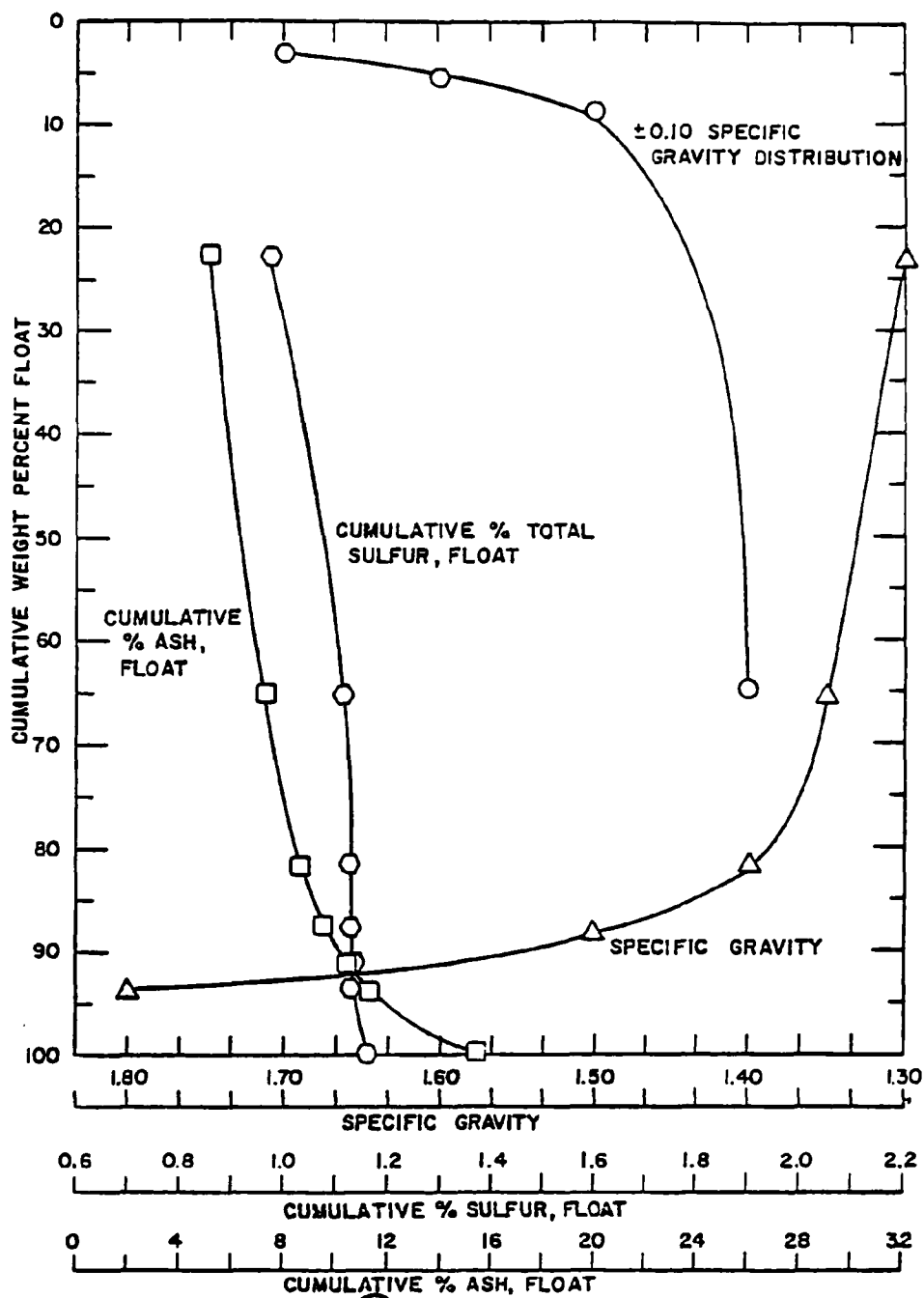


FIGURE 5 MAGNEX PROCESS WASHABILITY PLOT FOR A 6 INCH X 100 MESH COAL

Figure 6 is the graphical representation of the laboratory data with superimposed pilot plant test data shown by asterisk.¹⁶ In two pilot plant runs, using 75 and 10 kg (15 and 20 lbs.) of iron carbonyl per ton of coal, the clean coal yields were significantly higher (7.9 and 3.6 percentage points, respectively) than the results obtained from the laboratory runs. The sulfur dioxide to BTU ratios for the pilot tests were close to that predicted by the laboratory runs. Pilot plant results indicated that for coal used in this evaluation 10 kg per metric ton (20 lb per ton) of iron carbonyl was adequate to yield a product to meet the current new source SO₂ standard.

Sulfur By-Products—

There are no sulfur by-products generated by the MagnexSM process. The process is a totally dry method of sulfur removal and the waste is a dry mineral refuse.

Benefit Analysis—

The main benefit associated with the MagnexSM process is that it is a totally dry process and has no coal washing and dewatering problems. The process utilizes moderate temperature and residence time and atmospheric pressures. Furthermore, the MagnexSM process achieves good ash removal, higher pyritic sulfur removal and higher yields when compared to a conventional gravity separation. The net heating value recovery of the system is estimated to be 76 percent, assuming clean product coal is used to generate steam and burn the CO released in the process.⁸ In the projected MagnexSM system, where plans are to recirculate the CO for the production of iron carbonyl, the net heating value yield could be as much as 80 percent. This is because only a small bleed stream will be incinerated or scrubbed in the alternative system.

The process is, however, restricted to the removal of mineral sulfur and ash and also requires rather extensive monitoring because of the use of highly toxic iron pentacarbonyl and the generation of carbon monoxide.

No analysis has been conducted to determine the nitrogen or the trace metals content of the treated product. However, it is anticipated that the MagnexSM process will remove some of the trace metals in the coal while reducing the total ash content of the coal feed.

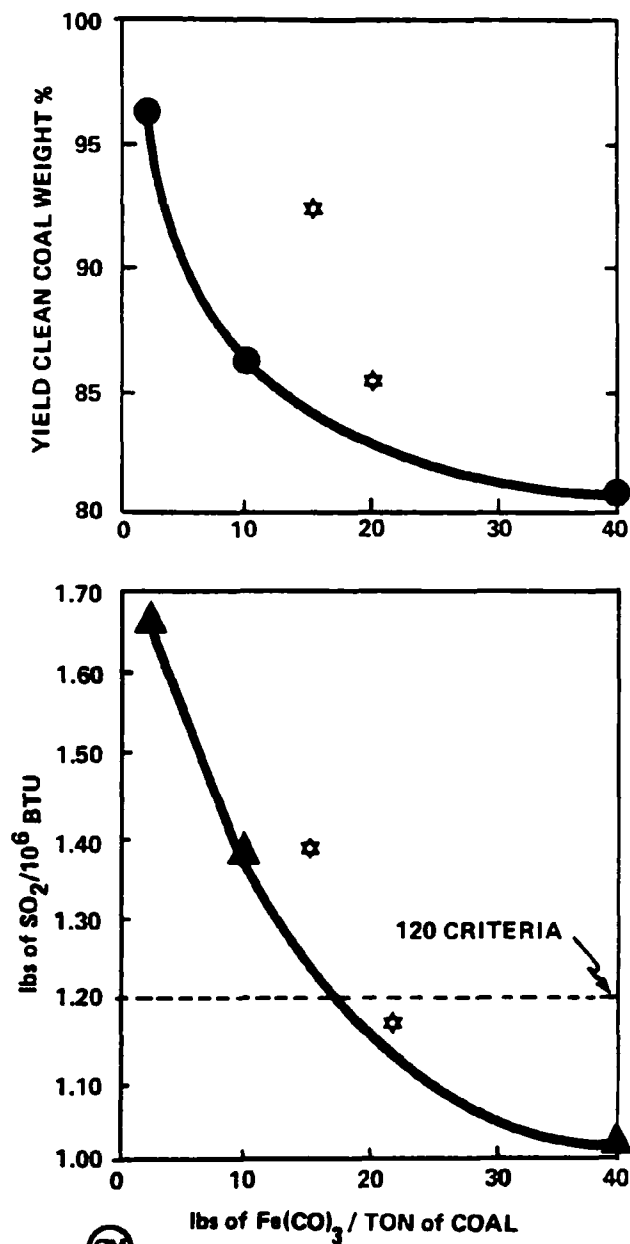


FIGURE 6 SM MAGNEX PROCESS EFFICIENCY COMPARISON OF LABORATORY AND PILOT PLANT DATA

Environmental Aspects--

The treat-gas stream used in this process consists of iron pentacarbonyl and carbon monoxide. Both of these gases are toxic and thus extensive safety measures should be taken to isolate and contain these hazardous materials. There are several other industries in the U.S. which currently use toxic materials. For example, toxic nickel carbonyl is used in nickel powder manufacturing. Since the hazard of nickel carbonyl is recognized, safety precautions have been instituted at these plants to render their operations environmentally safe. Similar control and safety measures could be instituted at MagnexSM plants.

Extensive use of lock-hoppers will be made to isolate the toxic compounds. The bleed gas from the reactor would be incinerated or scrubbed. Toxic gas alarm systems will be utilized as a warning measure in cases of unavoidable gas emissions. The use of proper ventilation system coupled with adequate air emission controls will minimize the adverse environmental effects from MagnexSM facilities.

In the vicinity of the plant, coal handling, crushing, grinding and conveying operations will be enclosed to provide dust control. Use of cyclones and baghouses for solids recovery and particulate emission control will be adequate.

There will be no waterborne waste generated by the MagnexSM plant. However, the dry refuse generated by this facility will contain heavy metals, sulfur compounds, and will be enriched in iron content. This waste will have essentially the same characteristics as the refuse material generated by physical coal cleaning plants. However, it will be in a totally dry and relatively compact form and as such it will be more manageable.

Problem Areas--

The major problem area for this process is to develop and demonstrate the production of low-cost iron pentacarbonyl using the CO generated from the decomposition of the treat gas. The use of impure CO in iron carbonyl manufacture is highly questionable.

Crushing coal to much less than 14 mesh top size may be necessary to enable the MagnexSM process to be applicable to coals containing a widespread pyrite particle size. Should the results of further investigations necessitate fine coal crushing for pyrite liberation, the commercially available magnetic separators may be inadequate for fine size magnetic mineral separation. Further developmental and demonstration work would then be necessary in the area of fine particle magnetic separation from the carbonyl treated coal feed.

R&D Efforts and Needs—

The specific research efforts and needs for this process are:

- demonstrate the process of on-site iron carbonyl manufacture from the recycle CO.
- design, assemble and operate a demonstration plant [54 metric tons (60 tons) per hour plant] incorporating and integrating the iron carbonyl manufacturing from recycle CO.
- demonstrate what size consist various coals must be crushed to, for pyrite liberation, prior to iron carbonyl treatment.
- study physical, chemical and combustion characteristics of the treated product in order to define its combustion behavior and to evaluate the pollutant emissions from the burning of the treated material.

Process Economics

The MagnexSM process is a totally dry process and therefore the capital costs associated with equipment installation are relatively low.

The economic estimates presented herein are based on a plant which processes 300 metric tons (330 tons) per hour, 7,200 metric tons (8,000 tons) per day of coal. The coal is assumed to have 0.70 weight percent organic sulfur and 1.22 weight percent pyritic sulfur which is processed to meet the current new source sulfur dioxide emission standard of 2.2 kg SO₂/million Kg cal (1.2 lb SO₂/million BTU). It is also assumed that the coal is treated with 10 kg of iron carbonyl per metric ton (20 lb/ton).

A summary of economics for the Magnex process is given in Table 19. Details on the capital costs are presented in Table 20. The capital estimate for the desulfurization circuit is based on preliminary estimates reported by Hazen. The total capital estimate assumes a grass roots plant which includes off-site facilities such as coal crushing and handling, product compacting, office buildings, rail facilities, etc. The estimated annual operating costs are presented in Table 21. It is assumed that iron carbonyl can be manufactured on-site at a cost of \$0.22 per kilogram (\$0.10 per lb). This is the price of iron carbonyl projected by Hazen. The current vendor quotes of iron carbonyl range up to \$3.3 per kilogram (\$1.50 per pound).⁸ Therefore, the economic feasibility of this process is dependent upon the developer's success in producing low cost iron carbonyl on-site.

TABLE 19. SUMMARY OF ECONOMICS FOR THE MAGNEXSM
CHEMICAL COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Grass roots plant installation

79.4% weight yield, 80% heating value recovery

Installed Capital Cost: \$37,815,000

Annual Operating Costs

on Clean Coal Basis: \$19,238,000 process cost, excluding coal cost
 \$85,238,000 process cost, including coal cost*
 \$10.12/metric ton (\$9.18/ton), excluding coal cost
 \$44.84 /metric ton (\$40.67 /ton), including coal cost*
 \$1.47 /10⁶ kg cal (\$0.37 /10⁶ BTU), excluding coal cost
 \$6.52 /10⁶ kg cal (\$1.64 /10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 20. INSTALLED CAPITAL COST ESTIMATE FOR THE MAGNEX SM
CHEMICAL COAL CLEANING PROCESS

	<u>\$ 1977 (1st Quarter)</u>
Coal handling and preparation*	\$ 6,000,000
Desulfurization process costs ^Δ	
Carbonyl treatment and generation	5,250,000
Magnetic separation	11,925,000
Heating	2,000,000
Compacting and product handling [†]	5,120,000
Building and miscellaneous ^α	700,000
Utilities (off-sites) ^Φ	3,565,000
Site development and general ^δ	<u>4,525,000</u>
Subtotal	29,085,000
Engineering design @ 10%	2,910,000
Contingency @ 20%	<u>5,820,000</u>
Total Installed Plant Capital (TPC)	\$37,815,000

* Versar estimate based on crushing raw coal to -14 mesh

Δ Hazen estimate

† Versar estimate

α Includes administration building, maintenance shop, stockrooms and stores

Φ Versar estimate includes the following facilities:

- low pressure steam generation
- water treatment for boiler make-up
- water supply
- process and potable water
- fire protection
- instrumentation
- electrical

δ Includes railroad facilities for incoming and outgoing cars and loading and unloading facilities for raw materials and loading facilities for refuse material

TABLE 21. ESTIMATED ANNUAL OPERATING COSTS FOR THE MAGNEXSM
CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	4,444,000
Taxes @ 2% TPC	757,000
Insurance @ 1% TPC	378,000
Labor (direct, indirect, additives, support)	219,000
General and administrative @ 1.5% TPC	567,000
Maintenance and supplies @ 5% TPC	1,891,000
Utilities:*	1,400,000
Electric power	
Water	
Steam & fuel [†]	
Chemicals:	
Iron carbonyl ^Δ	5,333,000
Binder	3,811,000
Waste Disposal,	438,000
Total Annual Processing Cost	19,238,000
Raw coal 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$85,238,000

* Excluding CO incineration and steam generation.

[†] It has been assumed that 11.8 metric tons/hr (13 tons/hr) product coal will be used to provide in-process needs (CO incineration and steam generation).¹

^Δ Hazen estimate; operating cost of iron pentacarbonyl manufacturing on-site.

SYRACUSE RESEARCH CHEMICAL COAL COMMINUTION PROCESS

The Syracuse Research Corporation has developed a process for the chemical fracturing or comminuting of coal, which is an alternative to mechanical crushing and fine grinding. The process is a precursor to the removal of pyritic sulfur and ash-forming components of coal by physical coal cleaning methods. Since the process is chemical in nature and it does remove pyritic sulfur when combined with a physical coal cleaning process, it has been included in this study of chemical coal cleaning processes.

Chemical comminution is a process that involves the exposure of the coal to certain low molecular weight chemicals that are relatively inexpensive and recoverable (usually ammonia gas or a concentrated aqueous ammonia solution). "The chemical disrupts the natural bonding forces acting across the internal boundaries of the coal structure where the ash and pyritic sulfur deposits are located. An apparent breakage of natural bonds occurs along these boundaries, thus exposing the ash and pyrite for follow-on separation. No significant dissolution of the coal occurs, nor is there any apparent reaction between the non-coal constituents and the comminuting chemical."¹⁷

"Since no mechanical breaking is involved in the chemical comminution approach, the size distribution of the comminuted (fractured) coal is governed by the internal fault system, the chemical employed, and the process operating parameters. The size distribution of the pyrite and other mineral constituents in the coal is solely dependent upon the characteristics and history of the coal being treated."¹⁷

Process Description

A conceptual flow sheet for the Syracuse process is presented in Figure 7. The starting material is raw coal which has been sized to 3.8 cm (1½ in) x 100 mesh. The minus 100 mesh coal is separated and shipped directly to the physical cleaning plant. The 3.8 cm x 100 mesh coal is weighed and charged to a batch reactor. In a typical cycle, the reactor is then closed and evacuated by a rotary seal pump for removal of air. The reactor is then pressurized with ammonia vapor to about 9 atm (120 psig). In a full scale operation this would be accomplished in two steps, first to 5 atm (60 psig) by equalizing ammonia pressure with another batch reactor (operated in

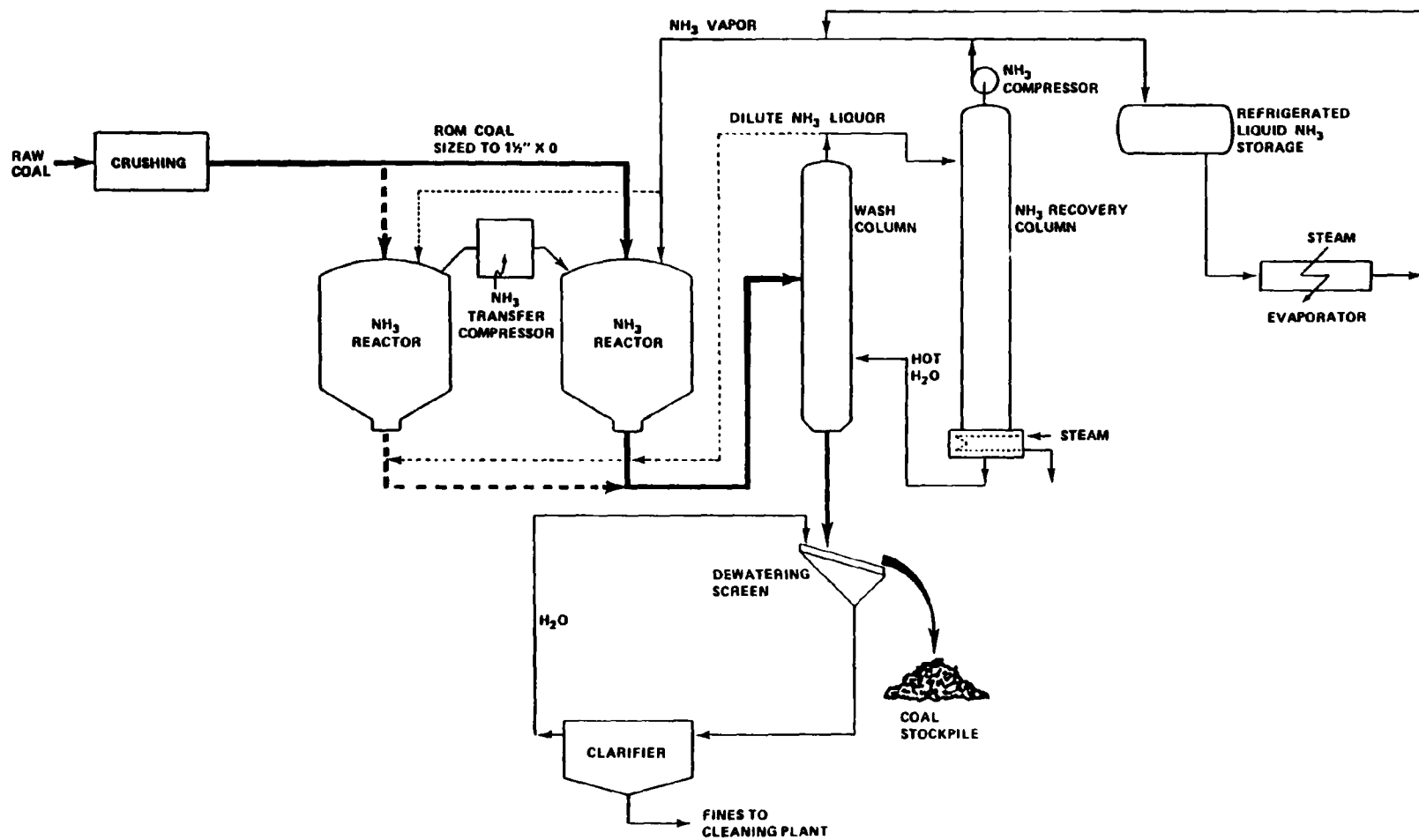


FIGURE 7 SYRACUSE COAL COMMUNITION PROCESS FLOW SHEET

parallel and just completing its' reaction cycle), and then to 9 atm (120 psig), using ammonia from either the ammonia compressor or from an evaporator which draws from a liquified ammonia storage tank. The reactor is held at 9 atm (120 psig) pressure for 120 minutes. During the reaction period, the temperature in the reactor rises 50°C to 65°C above the ambient temperature due to heat of solution of ammonia absorbed by moisture in the coal. The coal is comminuted to about 1 cm (3/8") top size.

At the end of the reaction cycle, the reactor is depressurized to 0.14 atm (2 psia) by first equalizing with another reactor which is charged with fresh coal, and then exhausting with a transfer compressor. These steps minimize loss of ammonia in coal. By this time, the temperature of the coal has dropped to about 27°C (80°F). The vacuum is then released in the reactor, and the coal is conveyed directly to a slurry mix tank prior to washing. The cycle of a batch is suggested as follows:

<u>Operation</u>	<u>Time (Min)</u>
Charging	30
Evacuation	30
Equalizing to 5 atm (60 psig)	30
Pressurizing and holding at 9 atm (120 psig)	120
Equalizing to 5 atm (60 psig)	30
Depressurizing to 1.1 atm (2 psig)	30
Release vacuum and discharge	30
Idle time	as required
TOTAL	<u>300 plus idle time</u>

All vent gases are collected through a rotary seal pump and scrubbed. The scrubber effluent is added to coal slurry.

Comminuted coal is slurried with a recycle stream pumped from the ammonia wash column. This recycle stream contains minus 30 mesh coal of 15-20% solids, plus 5-10% dissolved ammonia. A 35% solids slurry is formed with the comminuted coal and is pumped to the midpoint of the wash column.

As the coal sinks in this column it is washed free of ammonia with hot water. Coal containing about 20% moisture settles to the bottom of the column and is periodically discharged by a rotary valve to a dewatering screen.

The coal on the dewatering screen is washed to remove all minus 28 mesh fines and discharged to a stockpile, where it can then be sent to a cleaning plant. The minus 28 mesh fines from the dewatering screen leaves as a 20% slurry, and are sent to a clarifier. The fines are recovered as a 40% sludge, which is sent to the cleaning plant. The clarifier overflow water is recycled to product washing.

The ammonia recovery column is equipped with a feed preheater, a reflux condensor, and dome-cap trays. The column operates at one atmosphere pressure, nominally and the reboiler is heated by 2.7 atm (25 psig) steam. Ammonia is released from the incoming ammonia solution, and ammonia vapor containing about 2% moisture is cooled to 30°C (90°F) as it leaves the column. This vapor is compressed to 9.5 atm (125 psig) by the recycle compressor, and the vapor ammonia is either recycled immediately to a reactor, or is condensed and stored in a tank.

As has been stated above, all products from the chemical comminution step would be sent to a conventional coal cleaning or washing plant for separation of beneficiated coal from pyrite and ash-enriched refuse. A proposed operation of this type is illustrated in the flow sheet given in Figure 8. This flow sheet is proposed by the Syracuse Research Corporation.¹⁷

Status of the Process

In 1971 Syracuse Research Corporation initiated development of a program aimed at the removal of pyritic sulfur and ash-forming substances from coal. The results of this effort have been patented in the United States and in a number of foreign countries. During a portion of the project, effort was supported by the Energy Research and Development Administration, and a final report was published.¹⁸

All work to date has been performed on a laboratory or bench scale at the facilities of Syracuse Research. The largest tests have been with 23 kg (50 lb) batches of coal, which were run in large, specially constructed steel "bombs".

The diagram illustrates the coal washing process flow:

- Mine** → **Breaker** → **Stock Pile** (3 in. \pm 0)
- Stock Pile** → **Raw Coal Feed** (1250 TPH @ 8% Moist)
- Raw Coal Feed** → **Mill** → **Chemical Commminution** (1 1/2 in. \pm 0)
- Chemical Commminution** → **3/8 in. \pm 100 Mesh Comminuted Coal** (1050 TPH) → **Puddling Tank** → **Hydrocyclones** → **650 TPH** → **Screens** (\pm 1/4 in.) → **Mill** → **Chemical Commminution**
- Chemical Commminution** → **100 Mesh \pm 0 Screenings** (125 TPH) → **Flotation Cells** → **50 TPH** → **Thickener** → **Recycled Water to Plant or Washer**
- Chemical Commminution** → **400 TPH** → **Dewatering Screens** → **Covertfugal Dryers** → **Loading Bin** → **To Railroad Cars**
- Chemical Commminution** → **100 TPH** → **Classifier** → **Fines** → **Thickener** → **Refuse to Waste** (175 TPH)
- Chemical Commminution** → **550 TPH** → **Covertfugal Dryers** → **Filtrate Recycled**

All Weights are Dry Solids Unless Noted.

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Proof of the "cleanability" of the chemically comminuted coal product has been limited to development of laboratory washability data, followed by complete sulfur and ash analyses of the various fractions, and development of cumulative percent sulfur and percent ash contents versus percent coal recovery curves. It appears that no chemically comminuted coal has yet been subjected to separation in a coal washing plant, or even on coal washing pilot plant equipment.

In 1977 marketing of the process was undertaken by Catalytic, Inc. of Philadelphia, Pennsylvania and a complete report of the process and process economics was prepared.¹⁷

Exploratory efforts by Catalytic, Inc. to build and operate a pilot plant at a suitable location include negotiations for a site at Homer City, Pennsylvania or at TVA.¹⁹

Catalytic performed a study, at EPRI's request, comparing chemical comminution with mechanical crushing, both followed by heavy medium separation facilities for the Homer City application.

Technical Evaluation of the Process

Potential for Sulfur Removal—

As stated previously, chemical comminution by itself does not remove sulfur from coal. However, chemical fracturing exposes unwanted mineral matter in coal so that it may be more readily and efficiently removed in the following cleaning operation. After chemical comminution, both the mineral matter and the coal itself are claimed to have a larger particle size than mechanically fractured coal, when seeking the same pyritic sulfur or ash liberation rate.

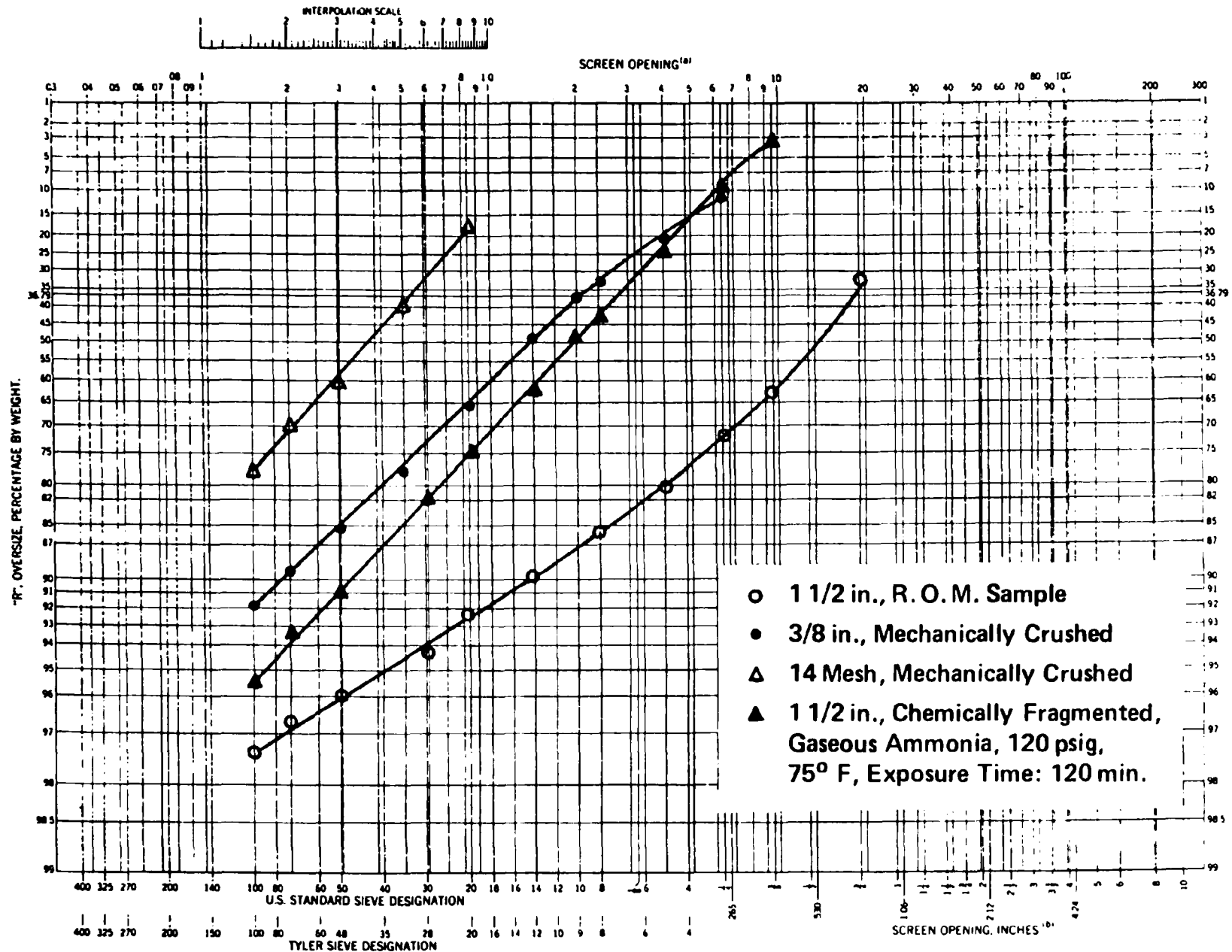
Since coal which has been chemically comminuted liberates pyritic sulfur more readily than mechanically fractured coal of the same size consist, the user can employ higher sulfur coals as feed stock to achieve a given sulfur level in the cleaned product. Conversely, for a given level of sulfur, chemical comminution will generally yield increased coal product.

In order to illustrate these claims, Figures 9, 10 and 11 are graphical presentations of washability studies completed on Illinois No. 6 (Franklin County) coal. Analyses of this coal and other coals discussed in this section are given in Appendix II.

In Figure 9, size consists are plotted for the following top sizes: 3.8 cm (1½ in) ROM; 1 cm (3/8 in) mechanically crushed; 14 mesh mechanically crushed; and 3.8 cm (1½ in) Syracuse process product (exposed for 120 min. to NH₃ gas @ 9 atm (120 psig) and 24°C (75°F)). The Syracuse process product contains 4.5% of minus 100 mesh fines, whereas the 1 cm (3/8 in) and 14 mesh mechanically crushed samples contain 8% and 22%, respectively, of minus 100 mesh fines. Since the initial 3.8 cm (1½ in) ROM contains 2.5% of fines, this means that chemical comminution has resulted in only a small additional amount of fines in the coal, while mechanical crushing results in larger amounts of fines. Generally, the minus 100 mesh fines are separated prior to heavy media coal separation and are either lagooned or are subjected to more intensive beneficiation such as flotation. Thus, according to the data of Figure 9, for Illinois No. 6 coal, the use of chemical comminution results in greater yields of coal for physical beneficiation than do the mechanically crushed coals.

In Figure 10, the washability data has been plotted to show percent cumulative ash in the four samples of Illinois No. 6 coal versus recovery of plus 100 mesh coal. This data indicates that, at any given recovery level, the 14 mesh mechanically crushed coal contains less ash than the chemically crushed coal and consequently is somewhat superior to the chemically comminuted coals in terms of ash rejection.

In Figure 11, the washability data is plotted to illustrate percent cumulative sulfur versus recovery. In this comparison, the chemically comminuted coal is clearly superior to the other three samples. For example, at a 90% recovery of plus 100 mesh coal, sulfur content would be 1.3%, for the Syracuse product, 1.48% for 1 cm (3/8 in) mechanically crushed coal, 1.44% for 14 mesh mechanically crushed coal and 1.51% for 3.8 cm (1½ in) ROM sample, respectively. For a selected sulfur value of 1.40%, weight yield recoveries would be 96%, for the Syracuse product, 78% for 14 mesh mechanically crushed coal, 70% for 1 cm (3/8 in) mechanical crushed coal, and 49% for 3.8 cm (1½ in) ROM sample.



**FIGURE 9 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: SIZE CONSIST COMPARISON
USING ILLINOIS NO. 6 COAL**

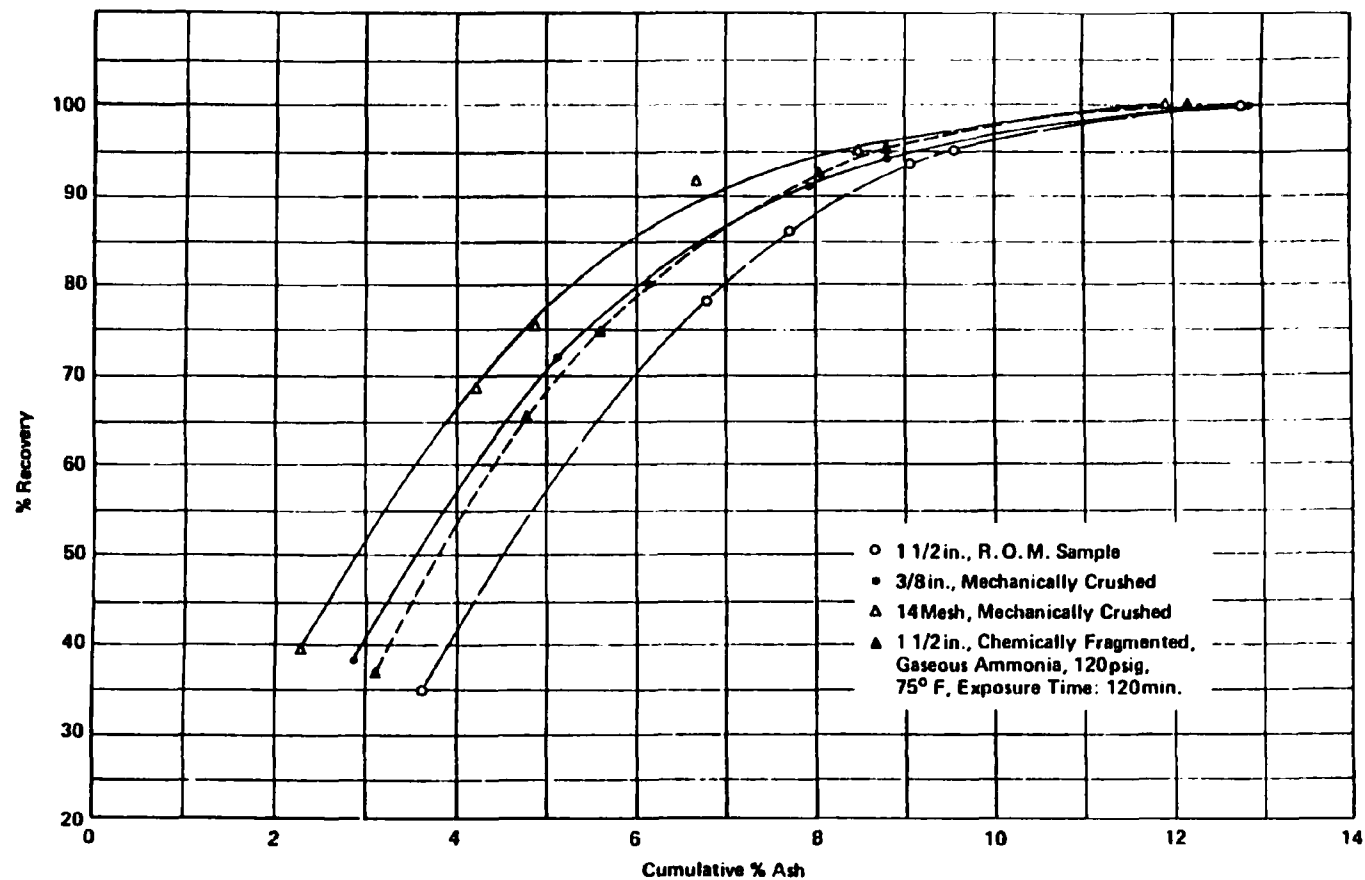


FIGURE 10 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: PERCENT ASH VS. PERCENT RECOVERY OF ILLINOIS NO. 6 COAL

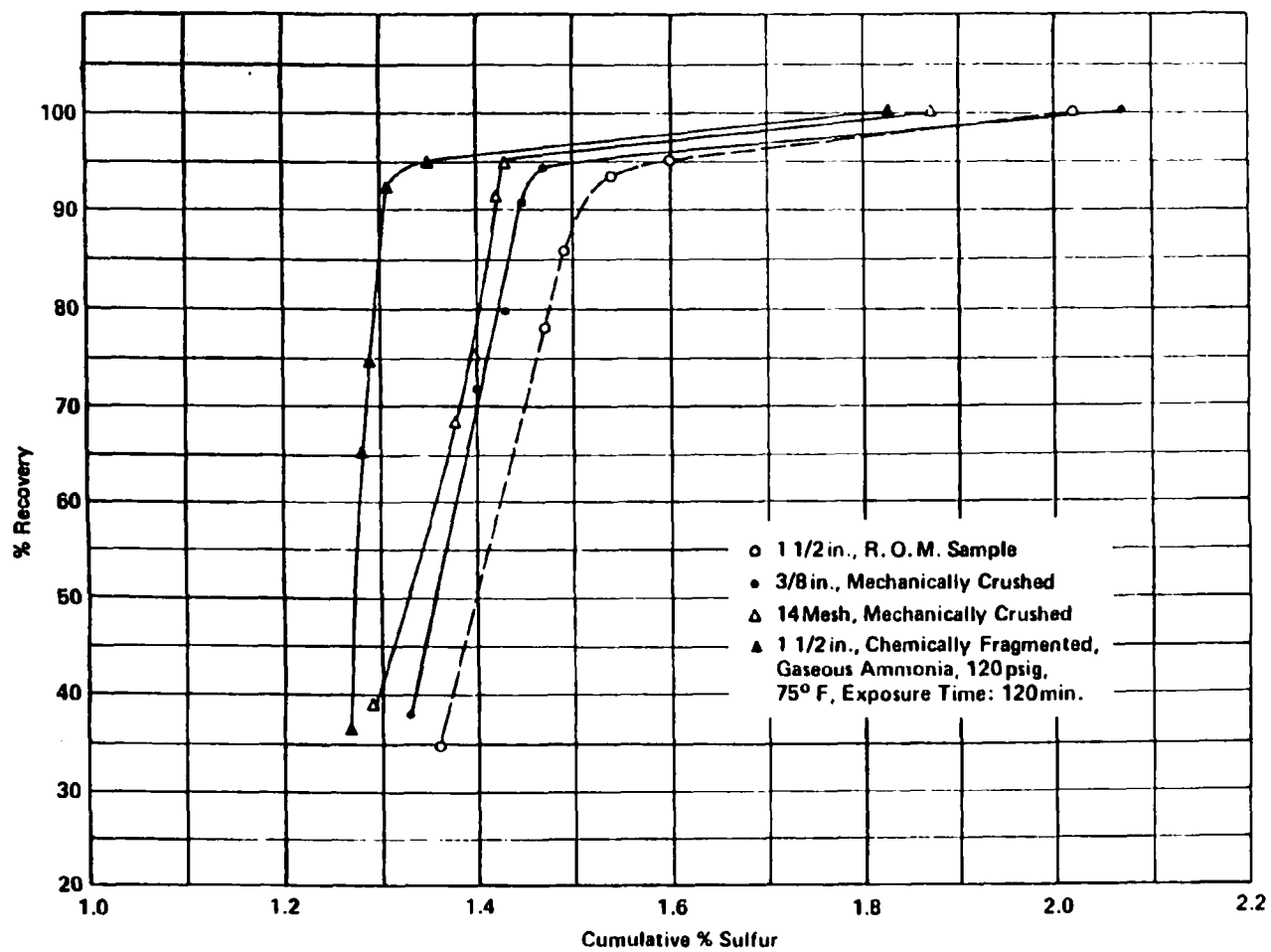


FIGURE 11 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: PERCENT SULFUR VS. PERCENT RECOVERY OF ILLINOIS NO. 6 COAL

Figures 10 and 11 are based on recoveries of plus 100 mesh coal only. When these recoveries are adjusted for the rejected minus 100 mesh fines, the absolute recovery levels of all four coal samples will be lowered. However, as shown in Table 22, for any given percent sulfur value the product recovery potential is greater for the chemically comminuted coal.¹⁷

The above example based on Illinois No. 6 coal illustrates a favorable application of the Syracuse chemical comminution process, in that product recoveries from the Syracuse process are superior to recoveries from ROM or mechanically crushed coals at any sulfur level. However, for optimum results on some coals the residence time at full pressure, 9 atm (120 psig), may extend beyond 2 hours, to 3 or possibly 4 hours. For other coals however, 30 minutes is a sufficient residence time. Furthermore, the process is not superior to mechanical crushing on some coals.

As an example where the process is not superior to mechanical crushing the data pertaining to an Upper Freeport (Westmoreland County, Pennsylvania) coal is reproduced in Table 23 and Appendix III. Coal recovery of fines-free Syracuse process product is poorer than one or both of the mechanically crushed products at lower sulfur values. Specifically, at a sulfur value of 0.9% the Syracuse product recovery is 77% versus 87% and 72% respectively for the 14 mesh and 1 cm (3/8 in) mechanically crushed coals. However, as shown in Table 23, on an overall yield recovery basis the Syracuse process is slightly superior to either mechanically crushed coal product.¹⁸ At a 1.3% sulfur content, the Syracuse process product is also inferior on a fines-free basis, but on an overall basis it approximates the recovery of the 1 cm (3/8 in) mechanically crushed coal. Thus, technically, there is little advantage of using the chemical comminution instead of mechanical crushing on the Upper Freeport coal.

The effect of gaseous ammonia exposure time on sulfur washability curves is illustrated in Figures 12 and 13. These show size consist and sulfur washability on samples of Pittsburgh seam coal (Green County) which has been treated as follows:

- ROM;
- minus 1 cm (3/8 in), mechanically crushed;
- minus 14 mesh, mechanically crushed;
- chemically comminuted, 2 hrs @ 9 atm (120 psig) & 75°F;
- chemically comminuted, 4 hrs @ 9 atm (120 psig) & 75°F.

TABLE 22. PRODUCT RECOVERY OF FOUR SAMPLES OF TREATED ILLINOIS NO. 6 COAL AT 1.4% SULFUR

<u>Sample Top Size</u>	<u>Percent Minus 100 mesh fines</u>	<u>Percent Recovery, fines free basis</u>	<u>Overall Recovery</u>
3.8 cm (1½ in) ROM Coal	2.5	50	49
1 cm (3/8 in) Mechanically Crushed	8	70	64
14 Mesh, Mechanically Crushed	22	78	61
3.8 cm (1½ in) Chemically Comminuted	5.5	96	91

TABLE 23. PRODUCT RECOVERY OF FOUR SAMPLES OF TREATED UPPER FREEPORT COAL AT 0.9% and 1.3% SULFUR

<u>Sample Top Size</u>	<u>Percent Minus 100 mesh fines</u>	<u>Percent Recovery, fines free</u>		<u>Overall Recovery</u>	
		<u>0.9%S</u>	<u>1.3%S</u>	<u>0.9%S</u>	<u>1.3%S</u>
3.8 cm (1½ in) ROM	2.8	37	72	36	70
1 cm (minus 3/8 in), Mechanically Crushed	9	72	92	65.5	84
14m, Mechanically Crushed	19	87	94	70.5	76
3.8 cm (1 1/2 in.) Chemically Comminuted	5	77	88	73	83.5

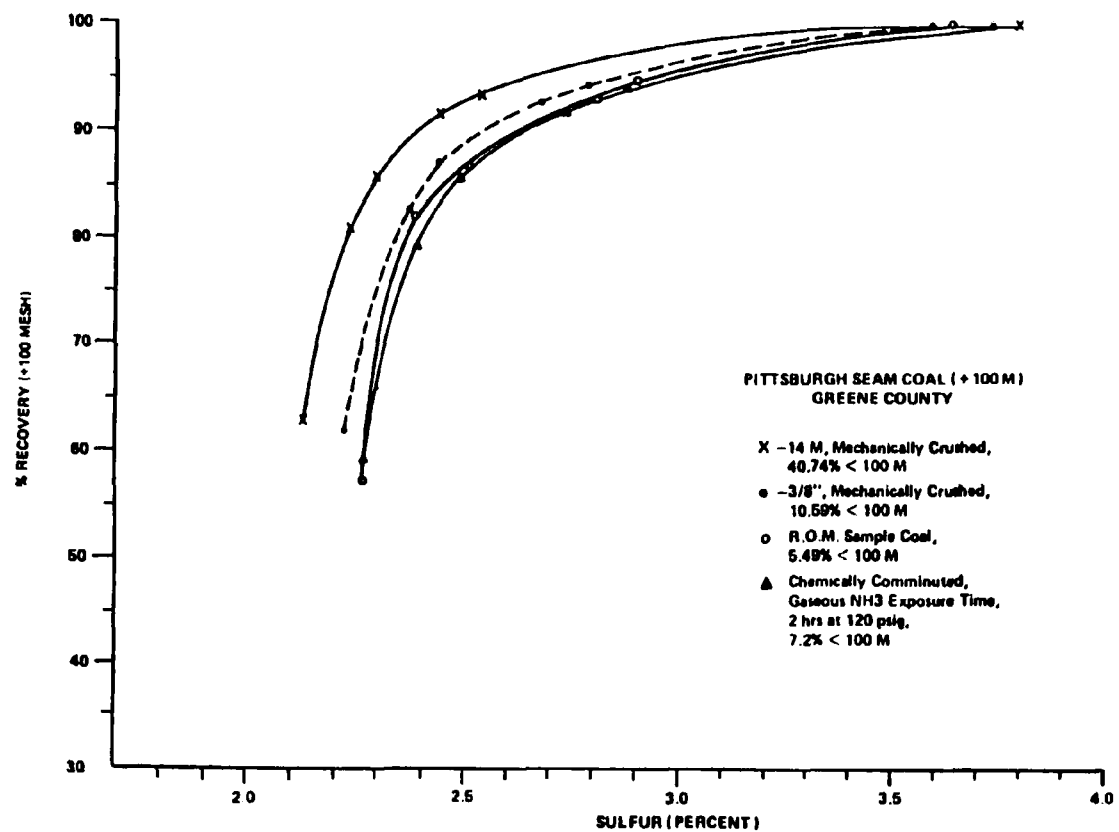


FIGURE 12 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: SULFUR WASHABILITY CURVES FOR PITTSBURGH COAL (TWO HOURS)

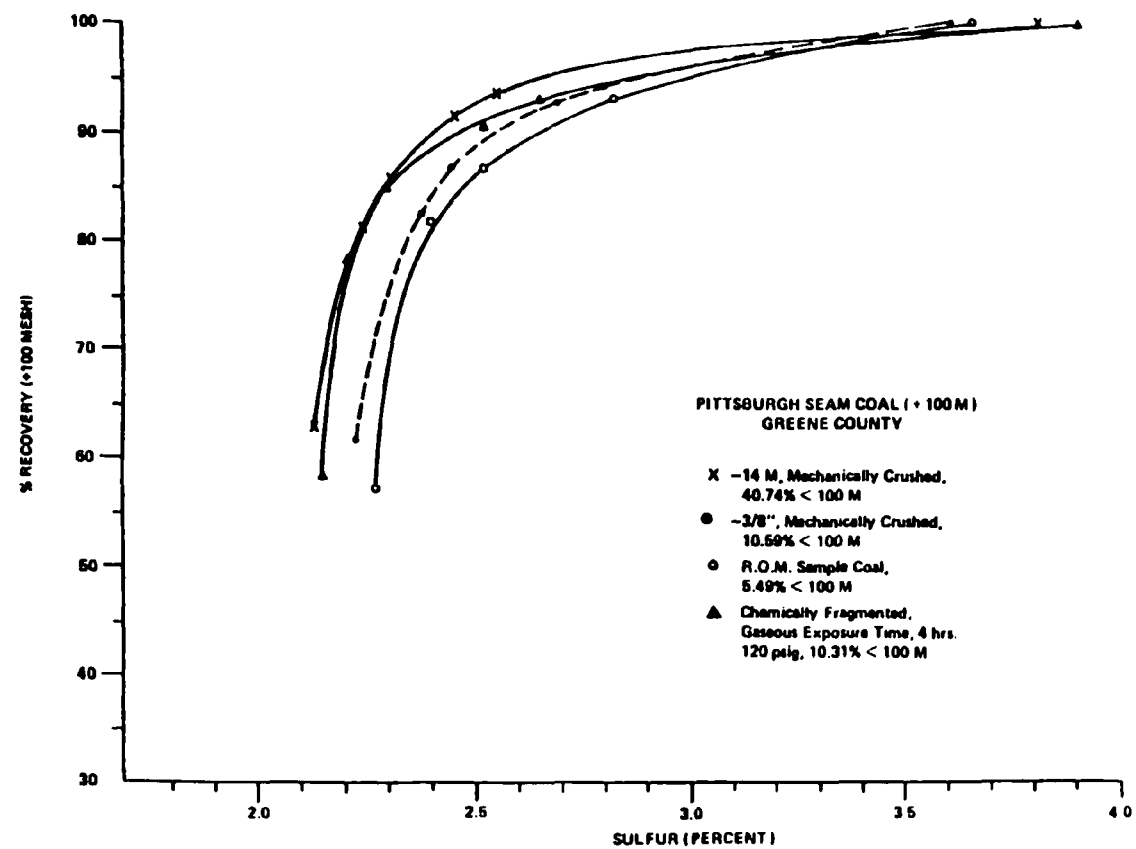


FIGURE 13 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: SULFUR WASHABILITY CURVES FOR PITTSBURGH COAL (FOUR HOURS)

As shown in Figure 12, the sulfur washability curve for the plus 100 mesh portion of the 2 hr. chemically comminuted coal overlaps the curve for the ROM coal, and the minus 14 mesh coal demonstrates the best washability. In Figure 13, the plus 100 mesh portion of the 4 hr. chemically comminuted coal is comparable to the minus 14 mesh mechanically crushed coal at the lower sulfur levels and to the minus 1 cm. (3/8 in) mechanically crushed coal at the higher sulfur levels. However, when these coals are compared on an "as is" basis, the severe losses (about 40%) to fines of the minus 14 mesh coal alters the comparative results. This is shown in the data of Table 24. On an overall basis, the recovery of minus 14 mesh coal is

TABLE 24. PRODUCT RECOVERY OF FIVE SAMPLES OF TREATED PITTSBURGH SEAM COAL (GREEN COUNTY) AT 2.5% AND 2.3% SULFUR

<u>Sample</u>	<u>% Minus 100 Mesh Fines</u>	<u>% Recovery Fines Free At 2.5%S</u>	<u>% Overall Recovery At 2.5%S</u>	<u>% Recovery Fines Free At 2.3%S</u>	<u>% Overall Recovery At 2.3%S</u>
3.8 cm (1½ in) ROM	5	86	82	67½	64
Minus 1 cm (minus 3/8 in) Mechanically Crushed	10	88	79	75	67.5
Minus 14 m, Mechanically Crushed	40	92.5	55.5	85	51
2 hr. Syracuse Process	7	85	79	66	61
4 hr. Syracuse Process	9	90	82	85	77

poor at a 2.5% sulfur level, and the remaining samples demonstrate approximately the same recovery. At 2.3% sulfur, the 4 hr chemically comminuted sample is clearly best, and the 2 hr chemically comminuted sample is superior only to the minus 14 mesh sample. It is therefore quite probable that optimum plant operating conditions for different coals will be different, and optimum conditions will have to be established by laboratory tests. In this respect, the chemical comminution process is no different than most other chemical coal cleaning processes.

As previously mentioned, the potential for removal of pyritic sulfur from ROM mechanically crushed coal, or chemically comminuted coal has been assessed to date only by laboratory washability data. This laboratory technique yields optimal results which are rarely duplicated in full-scale coal cleaning plants. Therefore, the washability comparisons made with respect to sulfur removal or product recovery, between chemically comminuted coal and mechanically crushed coals may be altered in plant operation.

Based on available data, it is anticipated that the Syracuse chemical comminution process followed by conventional physical coal cleaning, will remove 50 to 70 percent of pyritic sulfur in coals, with product recoveries of 90 to 60 weight percent. The coals used in laboratory studies contained high organic sulfur. Therefore, even removal of 100% of pyritic sulfur would not bring these coals into compliance with current EPA NSPS for SO₂ emissions. It is also concluded that the Syracuse chemical comminution process, followed by conventional physical coal cleaning, will bring some coals into compliance range if the organic sulfur level is sufficiently low.

Sulfur By-Products—

Chemical comminution in itself does not result in removal of sulfur from coal or in any chemical change in the sulfur. However, it does liberate pyrite and other mineral impurities from coal, so that a more efficient separation of pyrite and other minerals may be achieved in a subsequent physical coal cleaning step.

As a result, pyrites are concentrated and discarded in the refuse from the physical coal cleaning plant. There are no other sulfur by-products.

Environmental Aspects—

The chemical comminution process, per se, appears to possess no undesirable environmental aspects. Ammonia gas and resulting ammonium hydroxide are utilized or operated on in a completely closed system, so that fire or explosion hazards, or escape of concentrated vapors to the worker operating areas should be only a small possibility. In the event of a process stream leaking to the environment, there should be sufficient provision of seal pumps or compressors to minimize large losses. Small

losses can be safely allowed to dissipate to the environment with no adverse environmental effects.

After physical coal cleaning of chemically comminuted coal, the pyrite-rich refuse must be disposed of in the same manner as other sulfur-rich refuses.

Benefit Analysis—

The Syracuse chemical comminution process in combination with a conventional physical coal cleaning process, offers a means to remove up to 90% of the pyritic sulfur in some coals, with improved coal heating-values, and significantly decreased quantities of minus 100 mesh fines, as compared to mechanical crushing processes in combination with the same conventional physical coal beneficiation processes. This process also reduces the ash content of the coal, however not to the same extent as mechanical crushing followed by physical cleaning.

Problem Areas—

The chemical comminution process appears to be fully developed, without any major problems at this point. However, it should be mentioned that both the pilot plant and commercial plant designs are based on a reactor residence time of 120 minutes even though available experimental data show that some coals require as much as 4 hours (240 minutes) for good comminution. For coals requiring a longer residence time than 120 min., it is obvious that plant throughput rate and operating economics will be adversely affected.

R&D Efforts and Needs—

A report on the processing of 16 coals was due to be issued in 1977-78.²⁰ It should increase knowledge regarding the spectrum of coals which can be successfully and feasibly chemically comminuted. The data from this report may also clarify any necessity for providing reactor residence times greater than 120 min.

No additional laboratory research effort is recommended at this time. However, construction and operation of a pilot plant would contribute greatly to confirmation of plant design parameters and process operating costs.

Process Economics

A conceptual plant design for a commercial chemical comminution process was developed by Catalytic, Inc.¹⁷ to simulate that part of coal cleaning it would supplant, i.e., mechanical grinding. Versar has utilized the Catalytic, Inc. cost data and have modified and supplemented it as necessary.

Syracuse process cost estimates have been prepared for a grass-roots plant which includes:

- raw coal receiving, storage, coarse crushing and handling facilities, chemical comminution, coarse beneficiation, and drying, compaction, and shipping facilities;
- the Catalytic estimate for chemical comminution was based on a 27,000 metric ton/day (30,000 ton/day) plant. Capital costs for the 7,200 metric tons (8,000 tons) per day comminution plant were scaled down using the 0.6 exponential factor. Operating costs were, for the most part, scaled down linearly;
- amortization of all capital was calculated on the basis of 20 years capital recovery at 10% interest cost;
- drying and coal handling capital costs were based on data available from a Dow Chemical Co. cost document,⁵ scaling down with a 0.65 exponential factor, and adjusting for the relative cost indices of 1st quarter 1977 versus 1975. It has been assumed that the coarse beneficiated coal will require no compaction;
- operating and maintenance costs for coarse beneficiation were obtained (without breakdown) from an unpublished 1977 Gibbs & Hill study; and
- the coarse beneficiation process alleges an 80% weight yield and a 95% heating value recovery from the feed coal. No weight or heating value loss is charged against the chemical comminution step.

A summary of economics for chemical comminution plus physical beneficiation is presented in Table 25. Details on the capital costs and operating costs are given in Tables 26 and 27, respectively.

Using the cost estimation techniques and assumptions described above, the cost of chemical comminution plus physical beneficiation is \$6.36 per

metric ton (\$5.77/ton) of clean coal, or \$0.78/10⁶kg cal (\$0.20/10⁶BTU) excluding coal costs. Assuming a coal cost of \$27.6/metric ton (\$25/ton), these costs become \$40.82/metric ton (\$37.02/ton) of clean coal or \$5.03/10⁶kg cal (\$1.27/10⁶BTU).

TABLE 25. SUMMARY OF ECONOMICS FOR THE SYRACUSE RESEARCH
CHEMICAL COMMINUTION PROCESS PLUS COARSE COAL
BENEFICIATION

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Grass roots plant installation

80% weight yield, 95% heating value recovery

Installed Capital Cost: \$48,960,000

Annual Operating Costs

on Clean Coal Basis: \$12,190,000 process cost, excluding coal cost
 \$78,190,000 process cost, including coal cost*
 \$6.36 /metric ton (\$5.77 /ton), excluding coal cost
 \$40.82 /metric ton (\$37.02 /ton), including coal cost*
 \$0.78 /10⁶ kg cal (\$0.20 /10⁶ BTU), excluding coal cost
 \$5.03 /10⁶ kg cal (\$1.27 /10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 26. INSTALLED CAPITAL COST ESTIMATE FOR THE SYRACUSE
RESEARCH CHEMICAL COMMINUTION PROCESS PLUS COARSE
COAL BENEFICIATION*

Coal	<u>\$ 1977</u>
Coal handling and preparation	\$ 5,080,000
Desulfurization process costs	
Chemical comminution ^Δ	15,877,000
Coarse beneficiation (cleaning)	8,215,000
Drying and conveying	4,430,000
Product handling [†]	4,060,000
Building and miscellaneous	---
Utilities (off-sites)	---
Site development and general	---
Subtotal	<u>37,662,000</u>
Engineering design @ 10%	3,766,000
Contingency @ 20%	<u>7,532,000</u>
Total Installed Plant Capital (TPC)	\$48,960,000

* Including incoming coal handling facilities, comminution process facilities, physical coal cleaning facilities, refuse disposal, product drying, storage and handling.

Δ Catalytic, Inc. estimates includes power substation, cooling tower, air compressor, site development, building and fire protection.

† Assumes the coarse beneficiated coal can be handled and shipped without compaction.

TABLE 27. ESTIMATED ANNUAL OPERATING COSTS FOR THE SYRACUSE
RESEARCH CHEMICAL COMMINUTION PROCESS PLUS COARSE
COAL BENEFICIATION

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	\$ 5,750,000
Taxes @ 2% TPC	980,000
Insurance @ 1% TPC	490,000
Labor* (direct, indirect, additives, support)	620,000
General and administrative (included in labor)	---
Maintenance and supplies ^Δ	1,910,000
Operating and maintenance costs for physical coal cleaning	1,260,000
Utilities: [†]	
Electric power	560,000
Water	80,000
Steam	400,000
Chemicals:	
Liquid ammonia	140,000
Waste Disposal [†]	---
Total Annual Processing Cost	12,190,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAN ANNUAL COST	\$78,190,000

* Catalytic Inc. estimate.

^Δ Costs for chemical comminution process, only. These costs were taken as 5 percent of total comminution plant cost.

[†] Chemical comminution process, only.

ERDA CHEMICAL COAL CLEANING PROCESS

The ERDA air/steam leaching process is similar to the Ledgemont oxygen/water process, except that the process employs higher temperature and pressure to affect organic sulfur removal and uses air instead of oxygen. A coal desulfurization process very similar to the ERDA process is also described in a U.S. patent 3,824,084 assigned to the Chemical Construction Corporation.

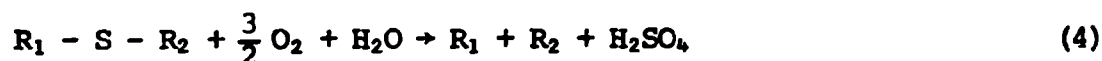
In the ERDA chemical coal cleaning process the pyritic sulfur is first oxidized to soluble sulfates. It is claimed that when the process operates at the preferred temperature and pressure of 150°C (302°F) and 34 atm (500 psia), essentially all the soluble sulfate is oxidized to insoluble iron oxide and sulfuric acid. Details on the pyrite removal reactions are given below.



The resulting stoichiometric reaction for pyrite removal is



The organic sulfur leaching chemistry is not well known. It is the developers belief that the major portion (>50 percent) of the organic sulfur in coal is of the dibenzothiophene (DBT) type which is inert to air at relatively high pressure and temperature. However, the remaining fraction of organosulfurs are not DBT-like and can react with air and steam to produce sulfuric acid.²¹ The suggested organic sulfur removal reaction is as follows



Process Description

In the ERDA air/steam oxidative desulfurization process the coal slurry is heated in the presence of compressed air at temperatures of 150°C to 200°C (300°-400°F), pressures 34 to 102 atm (500 to 1500 psia), and residence time of 1 hour or less. At these operating conditions, it is claimed that essentially all the mineral sulfur and approximately 40 percent of the organic sulfur is removed as sulfuric acid. The ERDA process has been conceptualized by Bechtel⁸.

A detailed flow diagram for this process including mass balance and stream properties, as developed by Bechtel is given in Appendix V.

A simplified flow diagram of the process as developed by Bechtel, is shown in Figure 14. Pulverized coal is mixed with water in the slurry mixing tank. The coal slurry is pumped to feed-effluent exchanges where the feed is heated with recovered heat from the reacted product. The feed is further heated in the flash gas quench tower by direct contact with desulfurization reaction off-gas, recycled from the product slurry flash tank. The feed slurry at operating temperature and pressure is passed through a series of reaction vessels where the sulfur in coal is oxidized in presence of compressed air. The product slurry is next flashed into product slurry tank and subsequently thickened, filtered and dried prior to compacting. A portion of the clean coal is burned to provide heat for drying.

The coal thickener overflow is combined with the filtrate from the coal filter and sent to lime treatment for neutralization of sulfuric acid and ferrous sulfate. The sulfuric acid in this stream is converted to gypsum and the ferrous sulfate to gypsum and ferrous hydroxide. These reaction products are sent to gypsum sludge thickener and subsequently filtered. The filter cake from this operation constitutes the solid waste from this process. The thickener overflow and the filtrate constitute the recycle water, which is sent to the slurry mixing tank.

Status of the Process

The ERDA chemical coal cleaning process was conceived approximately seven years ago by Dr. Friedman at the Bureau of Mines and the process is currently under study at ERDA's Pittsburgh Energy Research Center (PERC). Initial experiments on the air steam oxydesulfurization of coal were carried out using a batch, stirred autoclave system with 35 gram coal samples. This apparatus was modified to allow continuous air flow through the stirred reactor while the coal-water slurry remained as a batch reactant.

The current effort at PERC, centers on completing the installation of a 25 kg/day fully continuous unit. The unit was expected to be available for start-up testing in late 1977.² The system consists of a slurry feeder, slurry

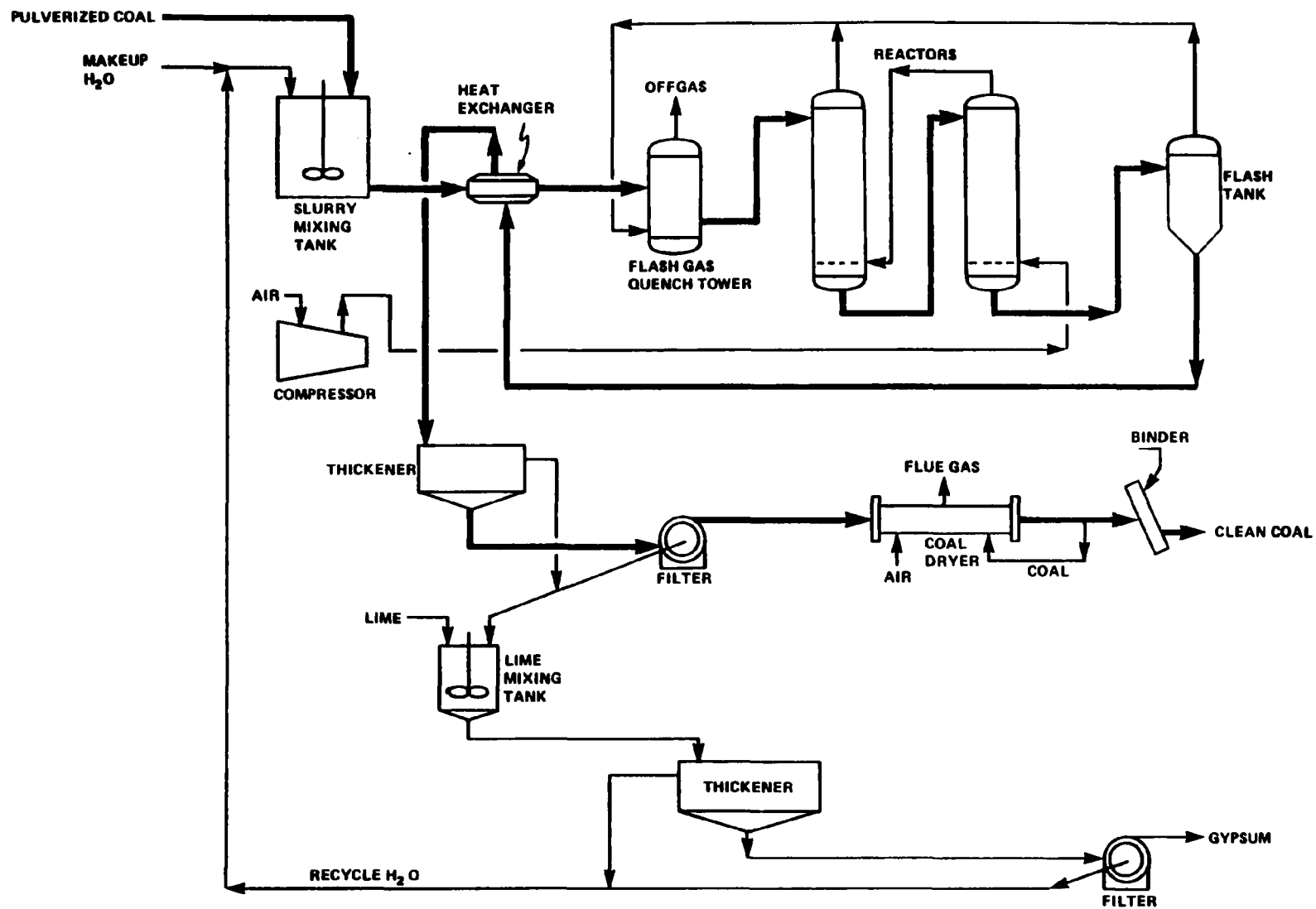


FIGURE 14 ERDA PROCESS FLOW SHEET

pre-heater, air preheater, a single Monel pressure vessel capable of operating at up to 69 atm (1,000 psig), two parallel pressure let-down tanks and a product recovery tank. This system is designed to obtain data on reaction rates and develop information on process engineering and economic evaluation. It is hoped that operating data will be available within 9 months so that a decision can be made regarding the design, construction, and operation of a larger continuously operated process development unit (PDU). There is a possibility that a large, private engineering group may assume the PDU effort, with support from ERDA.

Technical Evaluation of the Process

Technical evaluation presented here in is based upon published information and discussion with ERDA researchers, as well as the Bechtel⁸ conceptualization of this process and their prepared economic evaluation.

Potential for Sulfur Removal—

The developer's claim is that using this process, an estimated 45 percent of the mines in the eastern United States could produce environmentally acceptable boiler fuel in accordance with current EPA standards for new installations.²³ Available data from batch operations indicate that at mild temperatures of 150° to 160°C (300°-320°F) the ERDA air/steam oxydesulfurization process can remove more than 90 percent of the pyritic sulfur in coals. Table 28²³ presents pyrite removal information from several representative coals. The process is also claimed to remove up to 40 percent of coal's organic sulfur if the reaction temperature is raised to 180-200°C (360-400°F), this latter information is shown in Table 29.²³ Table 30²³ indicates that at low operating temperatures of 150 to 160°C (300-320°F) several high sulfur content coals, such as coals from Iowa and Indiana (Lovilia #4 and Minshall seams, respectively), can be significantly reduced in sulfur content by this process. Higher temperatures and pressures will be required to reduce the sulfur contents of these coals further.

The coal preparation requirements of this process are not known at this time. Minus 200 mesh ROM coal has been used in most runs, but a few runs using

TABLE 28. PYRITE REMOVAL FROM REPRESENTATIVE COALS USING THE ERDA PROCESS

<u>Seam</u>	<u>State</u>	<u>Temp, °C</u>	<u>Pyritic sulfur, wt. %</u>	
			<u>Untreated</u>	<u>Treated</u>
Illinois No. 5	Illinois	150	0.9	0.1
Minshall	Indiana	150	4.2	0.2
Lovilia No. 4	Iowa	150	4.0	0.3
Pittsburgh	Ohio	160	2.8	0.2
Lower Freeport	Pennsylvania	160	2.4	0.1
Brookville	Pennsylvania	180	3.1	0.1

TABLE 29. ORGANIC SULFUR REMOVAL FROM REPRESENTATIVE COALS USING THE ERDA PROCESS

<u>Seam</u>	<u>State</u>	<u>Temp, °C</u>	<u>Organic sulfur, wt. %</u>	
			<u>Untreated</u>	<u>Treated</u>
Bevier	Kansas	150	2.0	1.6
Mammoth*	Montana	150	0.5	0.4
Wyoming No. 9*	Wyoming	150	1.1	0.8
Pittsburgh	Ohio	180	1.5	0.8
Lower Freeport	Pennsylvania	180	1.0	0.8
Illinois No. 6	Illinois	200	2.3	1.3
Minshall	Indiana	200	1.5	1.2

* Subbituminous

TABLE 30. ERDA PROCESS OXYDESULFURIZATION OF REPRESENTATIVE COALS

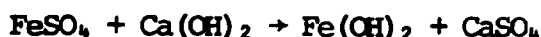
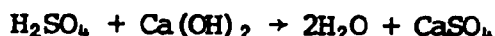
<u>Seam</u>	<u>State</u>	<u>Temp, °C</u>	<u>Total sulfur, wt. %</u>		<u>Sulfur, lb/10⁶ BTU</u>	
			<u>Untreated</u>	<u>Treated</u>	<u>Untreated</u>	<u>Treated</u>
Minshall	Indiana	150	5.7	2.0	4.99	1.81
Illinois No. 5	Illinois	150	3.3	2.0	2.64	1.75
Lovilia No. 4	Iowa	150	5.9	1.4	5.38	1.42
Mammoth*	Montana	150	1.1	0.6	0.91	0.52
Pittsburgh	Pennsylvania	150	1.3	0.8	0.92	0.60
Wyoming No. 9*	Wyoming	150	1.8	0.9	1.41	0.78
Pittsburgh	Ohio	160	3.0	1.4	2.34	1.15
Upper Freeport	Pennsylvania	160	2.1	0.9	1.89	0.80

* Subbituminous

minus 14 mesh coal are claimed to produce comparable results. Due to physical sizing limitations in the mini-pilot plant minus 200 mesh coal will be processed.

Sulfur By-Products—

The by-products from this process are dilute sulfuric acid and probably some unhydrolyzed ferrous and ferric sulfate. These are treated with lime according to the following equations.



The gypsum (CaSO_4) and ferrous hydroxide can be disposed of as filter cake. The filtrate from this operation can be recycled to the slurry mixing tank.

Benefit Analysis—

The main benefit associated with the ERDA air/steam oxydesulfurization process is the developer's claim for both mineral and organic sulfur removal. The process utilizes a relatively simple technique and inexpensive reagents for coal desulfurization. Additionally it requires no extraction or washing techniques for the removal of sulfur by-products.

It is also claimed that in the ERDA chemical coal cleaning process, sulfur is removed without incurring excessive oxidation of coal. The heat energy recovery of the system is said to be better than 90 percent. Ash is decreased only to the extent of sulfur removed. Consequently most coals have shown very slight decrease in heat content after this treatment. The ERDA air/steam process also destroys caking properties of coals, and thus the process can be also utilized as a pretreatment step for coal gasification.

Nitrogen content of the treated product has been determined and there is no change upon treatment. No analysis has been conducted to determine the trace elements.

Environmental Aspects--

There are no serious air emission problems anticipated with this process. The offgas from the reaction section will be scrubbed and condensed prior to

venting. In the vicinity of the plant, coal handling, crushing, grinding and conveying operations will be enclosed, to provide dust control. There should be essentially no waterborne waste generated by this system, provided the plant is designed to operate as a close loop system. The water balance in the system is claimed to be very good, with minimal make-up water requirement.

A potentially serious environmental problem associated with this process is the disposal of gypsum and ferrous hydroxide solid waste. This filter cake, approximately 0.1 metric ton per metric ton of coal will contain some trace metals and should be disposed of in an environmentally safe manner.

Problem Areas—

The problems associated with this process are engineering in nature. The major one appears to be associated with the selection of materials for the unit construction. The process generates dilute sulfuric acid which is highly corrosive at the process operating conditions.

A significant effort must be directed toward process optimization studies in order to select an economical and suitable material for lining the vessels and equipment in contact with the acid. This includes all equipment in the reaction section, feed preheaters, off-gas flash gas tank and scrubber system, and pressure let down equipment.

The cladding material selected by Bechtel for the economic assessment of this process is a 60-40 tantalum-niobium alloy at a thickness of approximately 2 mm (5/64 inch)⁶. The cost per square foot of the lining material is reported to be three times as much as the cost of the 7.62 cm (3-inch) thick carbon steel shell material selected for the high pressure reactor vessels.

The second major engineering problem is to select a means for pressure let-down to avoid erosion problems that may occur due to exposure of hardware to corrosive acid. Normal valving will erode in acid atmosphere. Therefore, other systems must be considered for pressure let-down.

R&D Efforts and Needs—

Specific research efforts and needs for this process are:

- Conduct bench scale tests to determine the coal preparation requirements for this process. The feasibility of coarse coal

(minus 14 mesh) processing should be further investigated to permit better assessment of sulfur removal potential and residence time requirement for the coarse coal system.

- Conduct pilot plant level technical effort to verify process data generated during the batch reactor operations and to establish accurate heat and material balance information for process economics evaluation and process development unit (PDU) design.
- Design, engineer, construct, and test a PDU having a throughput capacity of at least one metric ton/day. The PDU should integrate all major processing sections, including coal feed preparation and product compacting.
- Conduct studies to define the combustion behavior of the treated coal and to evaluate the pollutant emissions from the burning of the product coal.
- The effect of the treated product on the operational efficiency of electrostatic precipitators must be evaluated.

Process Economics

The economic estimates presented herein are based on Bechtel's conceptual design. However, the Bechtel cost estimate was modified by Versar to allow the evaluation of the ERDA process on a comparable basis with other processes included in this report. The estimates are based on a plant processing 300 metric tons (330 tons) per hour of pulverized coal (80 percent finer than 200 mesh). It has been assumed that the plant will operate 24 hours per day and 330 days per year basis.

The detailed flow sheet for the battery limit plant is given in Appendix V.

A summary of economics for the ERDA process is given in Table 31. Details on the installed capital cost for this process are given in Table 32, and the corresponding estimated annual operating costs are presented in Table 33. The unit operating costs shown are based on a net coal yield of 90 percent and a heating value yield of 94 percent. It has been assumed that the coal will be upgraded to meet the current NSPS sulfur dioxide emission standards.

TABLE 31. SUMMARY OF ECONOMICS FOR THE ERDA CHEMICAL
COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Gross roots plant installation

90% weight yield, 94% heating value recovery

Installed Capital Cost: \$166,810,000

Annual Operating Costs
on Clean Coal Basis:

\$56,595,000 process cost, excluding coal cost

\$122,595,000 process cost, including coal cost*

\$26.26/metric ton (\$23.82/ton), excluding coal cost

\$56.89/metric ton (\$51.60/ton), including coal cost*

\$3.69/10⁶ kg cal (\$0.92/10⁶ BTU), excluding coal cost

\$7.98/10⁶ kg cal (\$2.00/10⁶ BTU), including coal cost

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 32. INSTALLED CAPITAL COST ESTIMATE FOR THE ERDA
CHEMICAL COAL CLEANING PROCESS

	<u>\$ 1977</u>
Coal handling and preparation*	\$ 18,000,000
Desulfurization process costs ^Δ	100,000,000
Compacting and product handling [†]	5,120,000
Building and miscellaneous ^α	700,000
Utilities (off-sites) ^Δ	—
Site development and general ^Φ	<u>4,500,000</u>
Subtotal	\$128,320,000
Engineering design @ 10%	12,830,000
Contingency @ 20%	<u>25,660,000</u>
Total Installed Plant Capital (TPC)	\$166,810,000

* Versar estimate based on coal crushing and grinding to 80 percent
-200 mesh

Δ Bechtel estimate adjusted to 1st quarter 1977 price using CE plant
cost index. Includes off-sites.

† Versar estimate

α Versar estimate; includes administrative building, the maintenance shop,
stockrooms and stores

Φ Versar estimate; includes railroad facilities for incoming and outgoing
cars and loading and unloading facilities for raw materials and loading
facilities for by-product waste material

TABLE 33. ESTIMATED ANNUAL OPERATING COSTS FOR THE ERDA
CHEMICAL COAL CLEANING PROCESS

	<u>\$</u>
Amortization 20 years @ 10% interest (factor = 0.1175)	19,600,000
Taxes @ 2% TPC	3,336,000
Insurance @ 1% TPC	1,668,000
Labor (direct, indirect, additives & support)	753,000
General and administrative @ 1.5% TPC	2,502,000
Maintenance and supplies @ 5% TPC	8,340,000
Utilities:*	12,744,000
Electric power	--
Water	480,000
Steam	--
Chemicals:	
Lime	2,660,000
Binder	4,272,000
Waste Disposal	240,000
Total Annual Processing Cost	<u>56,595,000</u>
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	<u>\$122,595,000</u>

* Total excluding product drying and water. It has been assumed that 25.8 metric tons/hr (28.5 tons/hr) product coal will be used for in-process drying needs.

GENERAL ELECTRIC CHEMICAL COAL CLEANING PROCESS

The General Electric microwave process for chemically cleaning coal consists of the following steps:

- Crushed and ground coal (40 to 100 mesh) is wetted with a sodium hydroxide solution, then subjected to a brief (≤ 30 sec) irradiation with microwave energy in an inert gas atmosphere. Both pyritic and organic forms of sulfur react with the sodium hydroxide to form soluble sodium sulfide (Na_2S) and polysulfides (Na_2S_x) during irradiation.
- The coal is washed to remove the partially spent caustic and the sodium sulfides, then it is again wetted with caustic solution, and subjected to microwave radiation for an equivalent period.
- The coal is again washed to remove the partially spent caustic and the soluble sulfides, it is then dried and compacted.

The uniqueness of microwave treatment lies in the fact that the sodium hydroxide and the sulfur species in the coal can be heated more rapidly and efficiently than coal itself. Thus the reaction between sodium hydroxide and sulfur occurs in such a short time and with such low bulk temperatures that an insignificant amount of coal degradation occurs. As a result, the heating value of the coal is either unchanged or is slightly enhanced.

A number of bituminous coals having total sulfur contents from 1 to 6%, and having either predominately pyritic sulfur or organic sulfur contents, have been tested with total sulfur removals of 70 to 99%. Thus, the process does address itself to both of the two major forms of sulfur in coal. For most coals, two microwave irradiation treatments with fresh caustic are necessary. However, for the few coals with relatively low total sulfur content, a single treatment may be adequate to reduce the sulfur to a sufficiently low level to meet EPA NSPS standards for SO_2 emissions. Single treatments are generally 30-70% effective in total sulfur removal.

Process Description

In the absence of a flow sheet from G.E., a schematic flow sheet (Figure 15) of the desulfurization steps of the process has been proposed and discussed with G.E. project personnel. They agree with its principal features, which are as follows:

- 40 mesh top-size coal is slurried with a 20% solution of sodium hydroxide so that the coal is thoroughly wetted by the caustic.
- The moist coal is then subjected to microwave radiation for seconds. During this brief time, 30-70% of the total sulfur in the coal is converted to sodium sulfide (Na_2S) or polysulfide (Na_2S_x), and some of the water is evaporated.
- The coal is then slurried in water to dissolve and remove the sodium sulfides, dewatered, and then resaturated with about the same concentration and amount of caustic as previously.
- After a second exposure to microwave energy, the desulfurized coal is again washed free of sulfides and excess caustic, and is dewatered and dried to the extent required for on-site use, or is dried and compacted prior to shipping. Depending on the coal itself, and certain operating factors, 70% of the total sulfur in the coal will have been removed.

A schematic flow sheet has been proposed for the sulfur recovery process steps, which is also shown in Figure 15. This is necessary for an adequate conceptualization of the entire G.E. process and for process cost estimation. It is G.E.'s present intent to process wash waters containing sulfur by carbonating these liquors to produce hydrogen sulfide gas (H_2S), and then recover elemental sulfur via the Claus Process. The sodium carbonate, which also results from the carbonation step, would be treated with lime to regenerate soluble sodium hydroxide and insoluble calcium carbonate. The latter is then kilned to produce the CO_2 and lime (CaO), which are both recycled and reused. This regeneration process is almost identical to the one being considered by the Battelle Institute as a part of their chemical coal cleaning process.

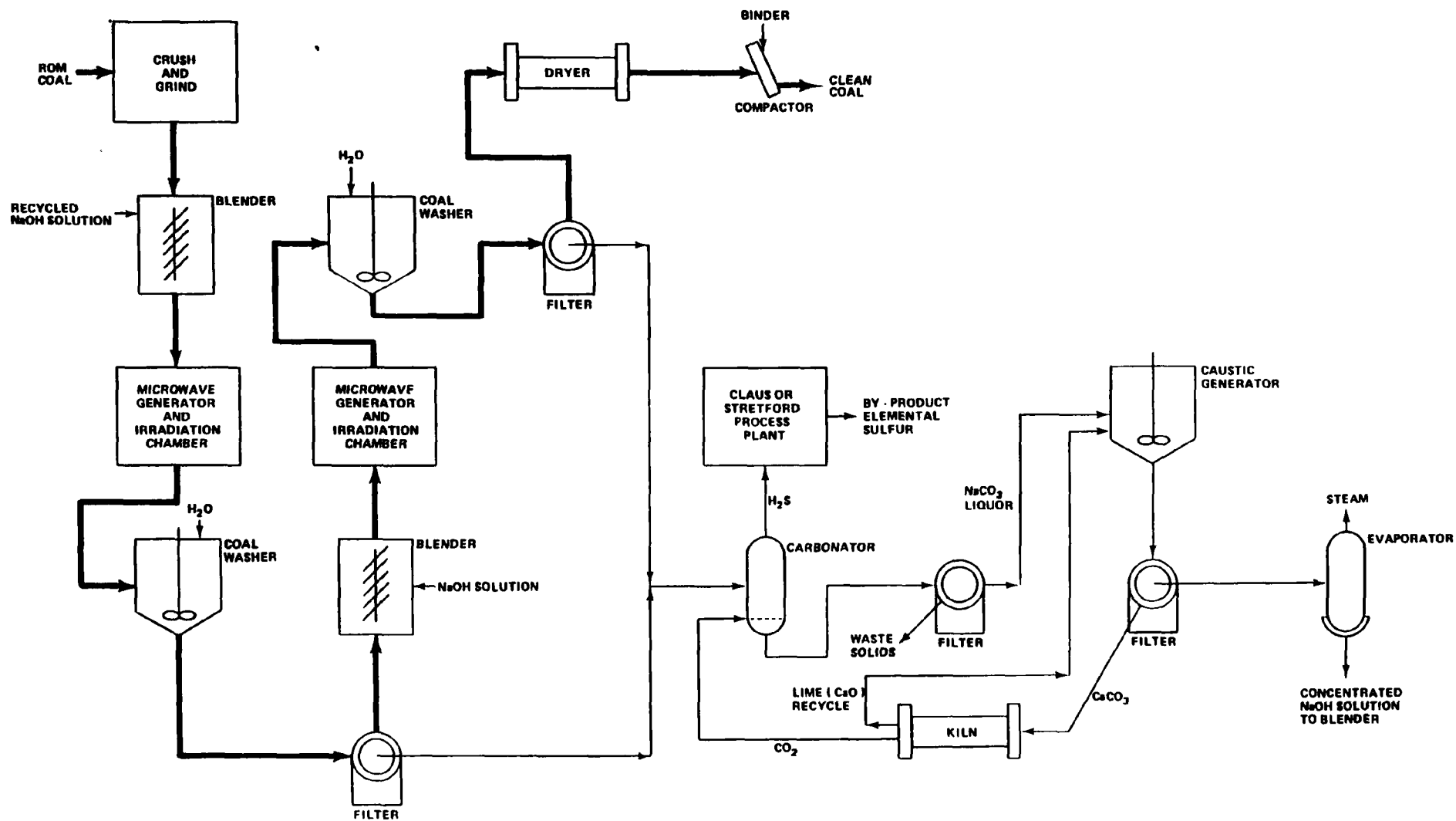


FIGURE 15 GENERAL ELECTRIC MICROWAVE PROCESS FLOW SHEET

The regeneration process at first glance appears simple and compact, however it may prove energy intensive due to:

- evaporative heat required to concentrate solids in the several filtrate streams.
- heat input to the kiln.

It will, therefore, be necessary to use minimum quantities of water and sodium hydroxide reactant in order to conserve heat energy in the subsequent sodium hydroxide regeneration steps.

Status Of The Process

All work to date has been done on a laboratory scale with small (10-100g) quantities of coal subjected to microwave radiation from a 1 KW, 2.4 GHz or a 2.5 KW, 8.35 GHz generator. The coal is first impregnated with a 20% solution of sodium hydroxide (NaOH), and sufficient caustic solution is retained on the coal after dewatering so that about 16 parts of NaOH are present per 100 parts of coal at time of treatment. Batch tests have been made on a number of coals in which the coals were irradiated once or twice for varying periods of time. However, exposure periods exceeding 30 seconds rarely gained further benefits.

Coals tested are obtained from the Fuel Sciences Department of Pennsylvania State University. As shown in Table 34, these coals provide a sulfur spectrum ranging from low organic-high inorganic to high organic-low inorganic sulfur.²⁴ These are all bituminous coals, with heating values of 6,200-7,500 kg cal/kg (11,300-13,400 BTU/lb) and a size consist of -40 to +100 mesh.

A 12 KW microwave generator has been requisitioned and will be in operation by the end of 1977. At that time, test runs will be made on quantities of coal up to 1 kilogram. These tests will also be made in conjunction with a pressure chamber which will allow microwave irradiation under pressures of 7.8 atm. (100 psig) with various gases. The principal functions of the inert gases are to retain any evaporated water as water vapor, to exclude oxygen from the working atmosphere, to minimize formation of undesired oxysulfur reaction products and to eliminate possibility of fire in case an electrical discharge occurs in the reaction zone.

TABLE 34.

ANALYSES OF COAL SAMPLES USED IN THE EVALUATION OF THE G.E. PROCESS

<u>Coal #</u>	<u>Geographic Origin</u>	<u>Pyritic</u>	<u>Sulfur Content, %</u>	
			<u>Organic</u>	<u>Sulfate</u>
PSOC-26	Illinois #6 Seam	4.23	2.08	0.35
PSOC-252	Illinois #5 Seam	2.82	1.84	0.06
PSOC-255	Lower Kittanning Seam from Pa.	4.49	0.78	0.03
PSOC-257	Upper Freeport Seam from Pa.	1.06	0.56	—
PSOC-294	Pittsburgh Seam from Pa.	2.27	0.34	0.01
PSOC-320	Pittsburgh Seam from Pa.	0.45	0.64	0.07
PSOC-353	Clarion Seam from Pa.	4.65	1.21	0.07
PSOC-272	Kentucky Seam #9	0.03	3.80	0.06
PSOC-273	Kentucky Seam #11	0.2-0.3	4.49	0.14

Total sulfur (combustible to SO_2) removals of 75% have been achieved for most bituminous coals provided that two sequential treatments are given. However, much remains to be done in terms of economic optimization of the process.

Technical Evaluation of the Process

Potential for Sulfur Removal—

A substantial removal of sulfur from bituminous coal appears technically feasible with this process, providing that microwave treatment of the coal is accomplished in two steps (See Figure 16).²⁴ It should be noted that when this figure was initially drawn by G.E., all analytical data indicated that 95-100% removal of sulfur could be achieved as a result of the two step treatment. Since that time, additional analytical techniques have been utilized and are yielding conflicting data. For example, on untreated coals the Leco and the Eschka methods show nearly identical sulfur analyses. On G.E. process treated coals, the Eschka (barium sulfate precipitation) method shows considerably more residual sulfur in the coal than does the Leco (combustion) method. Two conclusions are possible:

- The G.E. process does remove 75% or more of total S from coal, but not necessarily 95-100% in a 2-step process as was previously claimed.
- Since the sulfur which is not removed does not show up in a Leco combustion-type analysis, it may end up in the ash and thus may still not result in SO_2 emissions. Further effort to resolve this matter is in progress.

A one-step treatment is effective to the extent of 30-70% sulfur removal, as shown in Figure 17²⁵ and Table 35,²⁴ depending on the coal itself and other processing factors. Sulfur removal in subbituminous coal, anthracite, or lignite has not yet been attempted.

Sulfur By-Products—

The only projected by-product from the G.E. process will be elemental sulfur. This will be obtained by carbonation of the intermediate by-products, sodium sulfide and sodium polysulfide, to form gaseous hydrogen sulfide (H_2S). Hydrogen sulfide can then be reacted to form elemental sulfur via the

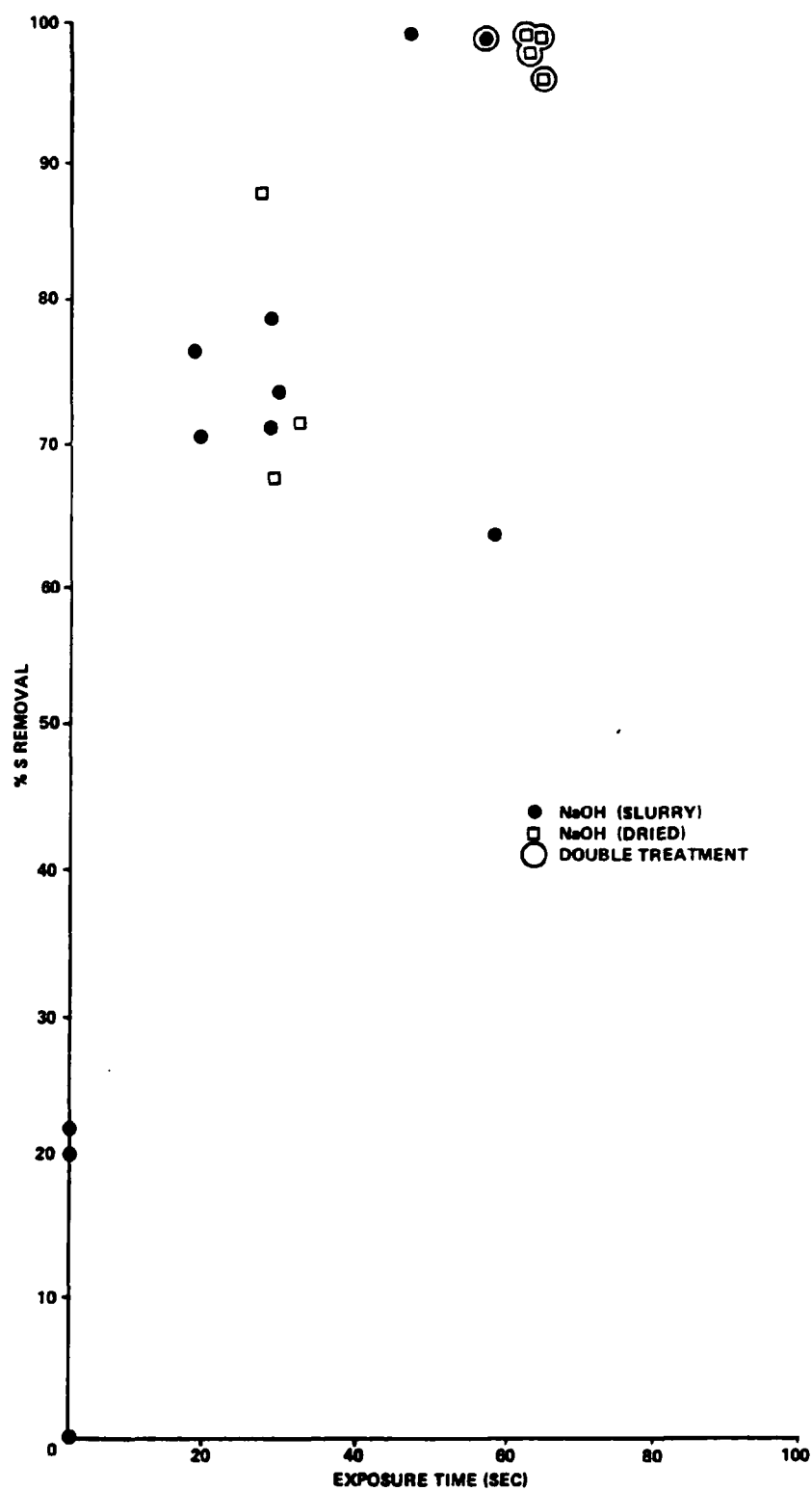


FIGURE 16 G.E. PROCESS: PERCENT SULFUR REMOVAL VS. EXPOSURE TIME
MULTIPLE EXPOSURE

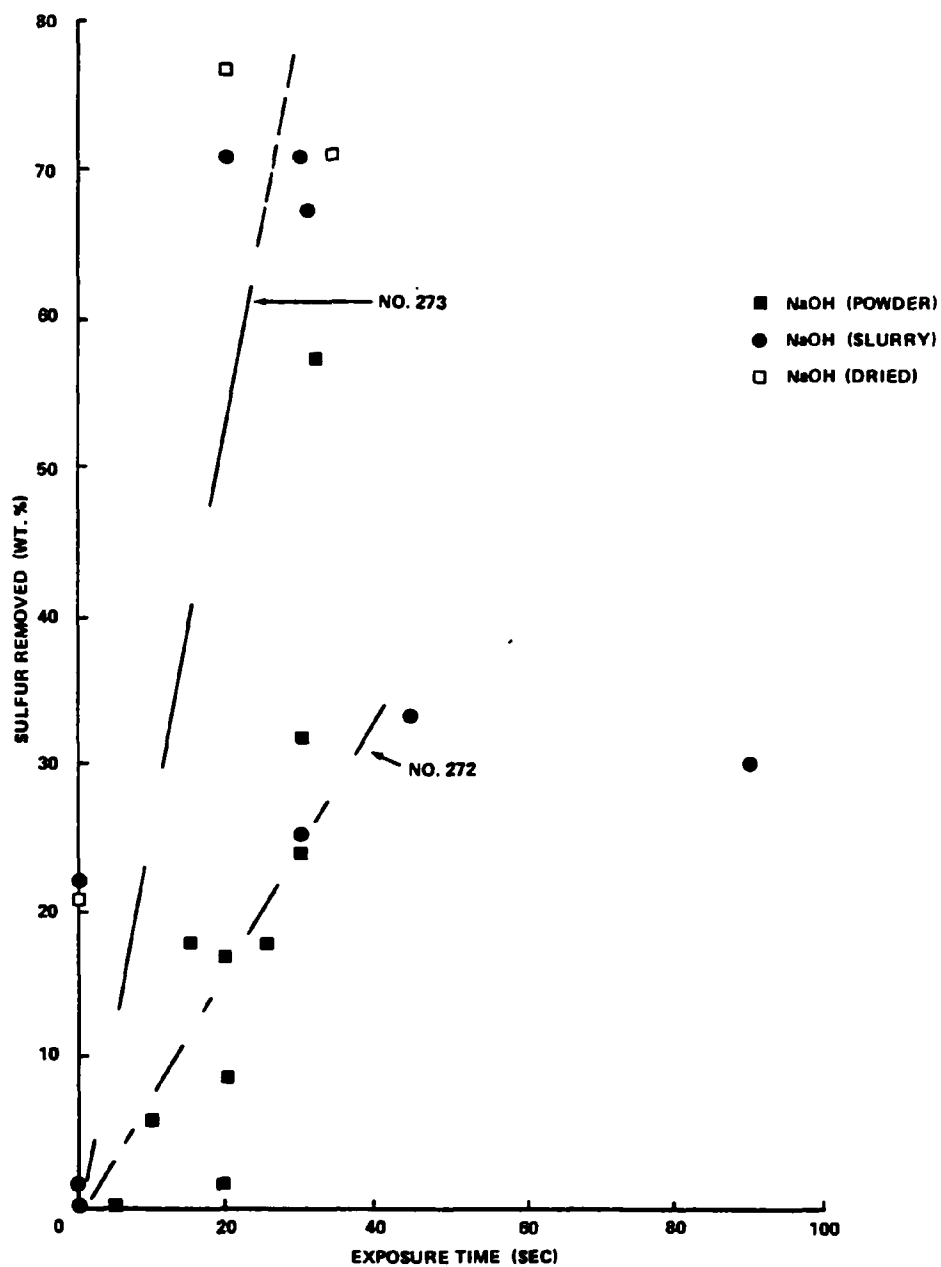


FIGURE 17 G.E. PROCESS: PERCENT SULFUR REMOVAL VS. EXPOSURE TIME
SINGLE EXPOSURE

TABLE 35. ANALYSES FOR RAW AND G.E. PROCESS TREATED COALS

	%S	%N	%C	%H	%O ₂	% Ash	% H ₂ O	% V.M.	% FC
PSOC 294 Pittsburgh-mostly pyritic	2.02	1.21	62.65	4.24	10.08	19.94	0.7	34.74	45.32
Single Treatment, 30 sec.	1.29	1.24	59.21	3.88	12.36	22.02	-	-	-
Double Treatment	0.4	1.09	53.87	2.22	14.06	29.24	3.0	24.7	46.10
PSOC 273 Kentucky #11 organic	4.18	1.33	60.19	4.80	18.62	12.88			
Dbl. Treatment 30 sec.ea.	< 0.1	1.15	63.34	4.39	22.14	8.98			
#5 Coal Clarion Co. Penna, mostly pyritic	2.37	1.53	78.82	5.69	9.12	2.47			
Single Treatment 30 sec.	0.88	1.49	75.51	5.17	11.44	5.53			

Claus or Stretford process. Other by-products attributable to imperfections in the caustic recovery and the Claus or Stretford process areas are possible but are presently unknown.

Benefit Analysis—

The G.E. chemical coal cleaning process possesses some excellent potential benefits, as follows:

- On the small scale thus far tested, the process appears highly efficient in removing sulfur from bituminous coal, regardless whether the sulfur is pyritic or organic.
- The coal matrix is only slightly affected by the process, and weight and heating value yields of product based on feed coal appear to be high but little data is currently available.

Environmental Aspects—

Few environmental problems of a special nature are apparent. Two process steps will require built-in design safeguards to prevent their becoming safety or environmental problems, as follows:

- Carbonation of the spent aqueous stream containing sulfides or polysulfides will result in the generation of highly toxic hydrogen sulfide gas. Since this gas is valuable and will be further processed to elemental sulfur, a properly designed enclosed reaction system should minimize the problems from this unit.
- The high intensity microwave generators which will be used, must be completely shielded. If adequate shielding is not provided, other microwave transmissions (TV, radio, telephone microwave transmitters) will be affected. In addition, humans can be affected adversely by exposure to microwaves, which can produce cataracts in the eyes.

No analyses of toxic trace elements are available at this time.

Problem Areas—

There are potential problems which can be recognized on the basis of its early state of development. Some of these potential problems are:

- Scaling up the process, particularly from batch-wise microwave treatments to large scale continuous mode operation, may prove difficult.
- The sodium hydroxide regeneration step may prove more costly and energy intensive than now estimated, particularly if the proportion of caustic to coal is high.
- If sodium is retained in the coal to a significant extent ($>0.5\%$), the ash resulting from subsequent coal combustion may be in the form of slag and thus the coal will not be usable in most boilers.

R&D Efforts and Needs--

As mentioned above, the G.E. process is at a very early stage of development. Consequently, there are a number of areas which need research and development before a comprehensive technical and economical evaluation can be completed. Some of these are as follows:

- The present scale of batch size for irradiator (10-100g) needs to be increased.
- An optimum microwave frequency for coal processing needs to be selected, concurrently with such parameters as NaOH/coal ratio, NaOH solution strength, pressure, presence or absence of inert gases, irradiation field strength and irradiation exposure time.
- Optimization of a single treatment step could lead to elimination of the present two step irradiation process, resulting in important economic benefits.
- The size consist of the coal should be varied in the experimental program. In general, the larger the size consist, the cheaper the coal preparation costs and the more storeable and shippable the product would be. In addition, other coals such as sub-bituminous, anthracite and lignite should be tested.

- More complete weight balances, and analyses of trace elements are needed to evaluate the process.
- Since there has been no research effort on by-product recovery (through evolution of H_2S gas), and sodium hydroxide recovery, these process steps should be thoroughly investigated. If sulfur recovery is not essentially complete, recycling of the impure caustic soda may result in reduced efficiency of sulfur removal in the coal.
- In the coal treatment steps and in the subsequent sodium hydroxide regenerations system, there are a number of dewaterings and filtrations (such as separation of calcium carbonate from sodium hydroxide). Special emphasis should be placed on investigating these operations, since an inefficient filtration could prove to be a process bottleneck and require very expensive equipment.
- Process research studies should attempt to minimize sodium hydroxide and water use, as the economics and the net energy yield of the process will be very adversely affected by excessive use and the resultant recovery of these materials.
- Any sodium build-up in coal (as ash) should be noted as this would be detrimental to combustion in some boilers.

Process Economics

Capital costs and operating costs were developed by Day & Zimmerman Co., Philadelphia, Pa., for G.E. for the coal needs of a 500 MW coal-burning power plant [requiring the use of about 4,500 metric tons (5,000 tons) per day of 5,550 kg cal/kg (10,000 BTU/lb) coal].

The G.E. cost estimate was based on certain assumptions which are sufficient for G.E.'s purposes, but which do not make it comparable with other process cost estimates included in this report. The G.E. last estimates were altered as follows:

- Production capability was increased from about 4,500 metric ton (5,000 tons) per day of 5,550 kg cal/kg (10,000 BTU/lb) coal to 7,200 metric tons, (8,000 tons) per day of 6,800 kg cal/kg (12,300 BTU/lb) coal.

- Capital costs were increased to the larger capacity using the 0.6 exponential factor, except microwave reactor costs, which were extrapolated linearly. In addition, capital costs for product compaction were added, and a 20% contingency was applied to the total.
- Operating costs, other than capital amortization, taxes and insurance, were increased linearly. The capital cost was amortized over a period of 20 years at 10% interest per year.
- An operating factor of 70% was initially used by G.E. on the basis of coupling their desulfurization process to a utilities steam plant. However, by uncoupling, a 90.4% operating factor is possible and is being assumed.
- Weight yield and BTU yield are assumed at 96% although no supporting data are available.

The new estimates for the G.E. process are reasonably comparable with estimates available for other processes and are quite comparable for a specific set of estimates recently prepared by Bechtel Corp.⁸ for six other chemical coal cleaning processes. The new capital estimate is \$102,000,000 and the new operating costs are \$39,820,000 per year. This data is summarized in Table 36. Details on the capital costs and operating costs are given in Tables 37 and 38, respectively.

TABLE 36. SUMMARY OF ECONOMICS FOR THE GENERAL ELECTRIC
CHEMICAL COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Grass roots plant installation

96% weight yield, 96% heating value recovery

Installed Capital Cost: \$102,000,000

Annual Operating Costs

on Clean Coal Basis: \$39,820,000 process cost, excluding coal cost
 \$105,820,000 process cost, including coal cost*
 \$17.33/metric ton (\$15.72/ton), excluding coal cost
 \$46.05/metric ton (\$41.77/ton), including coal cost*
 \$2.54/10⁶ kg cal (\$0.64/10⁶ BTU), excluding coal cost
 \$6.72/10⁶ kg cal (\$1.69/10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 37. INSTALLED CAPITAL COST ESTIMATE FOR THE GENERAL
ELECTRIC CHEMICAL COAL CLEANING PROCESS*

	<u>\$ 1977</u>
Coal handling and preparation ^Δ	\$ 11,284,000
Desulfurization process costs	
microwave reactors	29,200,000
Product washing & recovering ^φ	19,616,000
Claus process	5,627,000
Evaporators	4,426,000
Compacting and product handling [†]	8,309,000
Building and miscellaneous	—
Utilities (off-sites)	—
Site development and general	—
Subtotal	<u>\$ 78,462,000</u>
Engineering design @ 10%	7,846,000
Contingency @ 20%	<u>15,692,000</u>
Total Installed Plant Capital (TPC)	\$102,000,000

* Capital costs scaled up from estimates made by Day & Zimmerman Co. on a smaller installation. All costs are on a fully installed basis.

^Δ Includes storage, sludge pond, railroad sidings.

^φ Includes treated coal washing tanks, thickeners, surge tanks, vacuum filters and pumps.

[†] Versar estimate, includes product drying prior to compaction and handling.

TABLE 38. ESTIMATED ANNUAL OPERATING COSTS FOR THE GENERAL
ELECTRIC CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	11,980,000
Taxes @ 2% TPC	2,040,000
Insurance @ 1% TPC	1,020,000
Labor (direct and indirect) [†]	1,830,000
General and administrative (included in labor)	--
Maintenance and supplies [†]	5,310,000
Utilities:	
Electric power [†]	3,100,000
Water *	100,000
Steam [†]	4,070,000
Chemicals: [†]	
Sodium hydroxide	4,640,000
Lime	1,220,000
Binder*	4,510,000
Waste Disposal	--
Total Annual Processing Cost	\$ 39,820,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$105,820,000

[†] Information supplied by the process developer.

* Estimated by Versar.

BATTELLE CHEMICAL COAL CLEANING PROCESS

The Battelle hydrothermal coal process (BHCP) is based upon hydrothermal alkali leaching of mineral and organic sulfur compounds from coal. The process presently proposed by Battelle employs sodium and calcium hydroxides as a mixed leachant and operates under conditions of elevated temperatures and pressures. The desulfurized coal, after filtration and washing to separate the spent leachant, is dried and compacted for use in coal-fired utility boilers. At the present stage of development, the process must be considered as partially conceptual.

The BHCP desulfurization step has been tested on a series of raw bituminous coals and has been shown to extract essentially all of the pyritic sulfur and 25 to 50% of the organic sulfur starting with a range of total sulfur content of 2.4 to 4.6 percent. The product is a solid fuel which meets the current new source standard of a maximum of 2.16 kilograms of sulfur dioxide emission per million kg cal (1.2 lbs/10⁶ BTU) with certain coals.

Process Description

The proposed process consists of five principal steps:

Coal Preparation—

The raw coal is crushed and ground to suitable particle size, generally 70 percent minus 200 mesh. The coal then goes directly to a slurry tank for mixing with the leachant. Alternatively, the coal can be first physically beneficiated to remove some ash and pyritic sulfur before introduction into the slurry tank.

Hydrothermal Treatment—

The coal slurry is pumped into a reactor where it is heated to temperatures in the range of 200° to 340°C (400° to 650°F) and subjected to a pressure in the range of 18 to 170 atm (250 to 2,500 psig) to extract sulfur and dissolve a portion of the ash from the coal. Residence time is approximately 10 minutes. It is essential that this operation and the following one be carried out in an oxygen-free atmosphere to minimize the formation of oxysulfur compounds which prevent the quantitative recovery of sodium hydroxide from the spent leachant.

The recommended leachant for the process is a mixture of 8 to 10 percent sodium hydroxide (NaOH) solution in a 3 percent calcium hydroxide (Ca(OH)₂) slurry. Concentrations of these components of the leachant will vary depending on coal properties.

Fuel Separation—

The desulfurized coal is separated from the leachate by means of filtration and water washing. The leachate is then concentrated before regeneration.

Drying and Agglomeration—

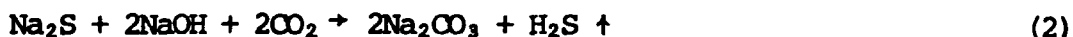
Water is evaporated from the coal in a drier, leaving dry, clean, solid fuel. This material is then compacted to a suitable pellet size for shipment to the user.

Leachant Regeneration—

A chemical regeneration step using carbon dioxide is used to remove sulfur from the leachate as hydrogen sulfide. This gas is then converted to elemental sulfur by either the Claus or Stretford process.

Table 39 presents Battelle's current best estimates of some key parameters which would be involved in a continuous process if the BHCP is based on lime-carbon dioxide regeneration of the spent leachant.²⁶

A simplified schematic flow diagram of the conceptualized BHCP based on a plant processing 300 metric tons (333 tons) per hour [7,200 metric tons (8,000 tons) per day] of coal feed is shown in Figure 18. The key chemical reactions in the process are shown below:



The schematic incorporates raw coal grinding, and treated coal drying and compaction steps, not included in the latest Battelle process flow sheet. Battelle proposes the production of treated coal as a wet material which is stored in silos prior to shipment to the utility. If located at power plant site the utility would be responsible for grinding

TABLE 39. TYPICAL VALUES OF KEY PARAMETERS IN
BATTELLE HYDROTHERMAL COAL PROCESS

Overall yield (average coal): 90-95%

Overall heating value yield (average coal): within ± 3 -10% of original heating value

<u>Unit Process</u>	<u>Parameter</u>	<u>Typical Value</u>
Coal Preparation	mesh size	70% minus 200 mesh
Coal Desulfurization	water/coal in feed	2
	NaOH/coal in feed	0.16
	CaOH/coal in feed	0.05
	reaction time	10 minutes
	temperature	275°C (525°F)
	pressure	50-55 atm. (700-800 psig)
Treated Coal/Water Separation System*	filtration/washing:	9 stages in series (optimum)
	percent solids in final cake discharge	45% (optimum)
	wash water/dry solids	1.5 (optimum)
	OR	
	centrifuging/washing:	3 stages in series (optimum)
	percent solids in final cake discharge	55% (optimum)
	wash water/dry solids	1.25 (optimum)
	total sodium in treated coal	0.25-1.96 wt.% (MAF) Δ
	total calcium in treated coal	8.0-9.2 wt.% (MAF) Δ
Sulfide Stripping	temperature	50-80°C (120-180°F)
	CO ₂ concentration in stripping gas	20-100%
	pH of stripped liquor	8

* Results of a computer simulation study

+ Battelle has used a value of 2.0 for costing purposes, using a combination of filtration and centrifuging to accomplish solid/liquid separation in the cost study.

Δ Range of values (moisture and ash-free basis) determined from hydro-thermal treatment of Westland and Martinka coals, EPA Contract No. 68-02-2119.

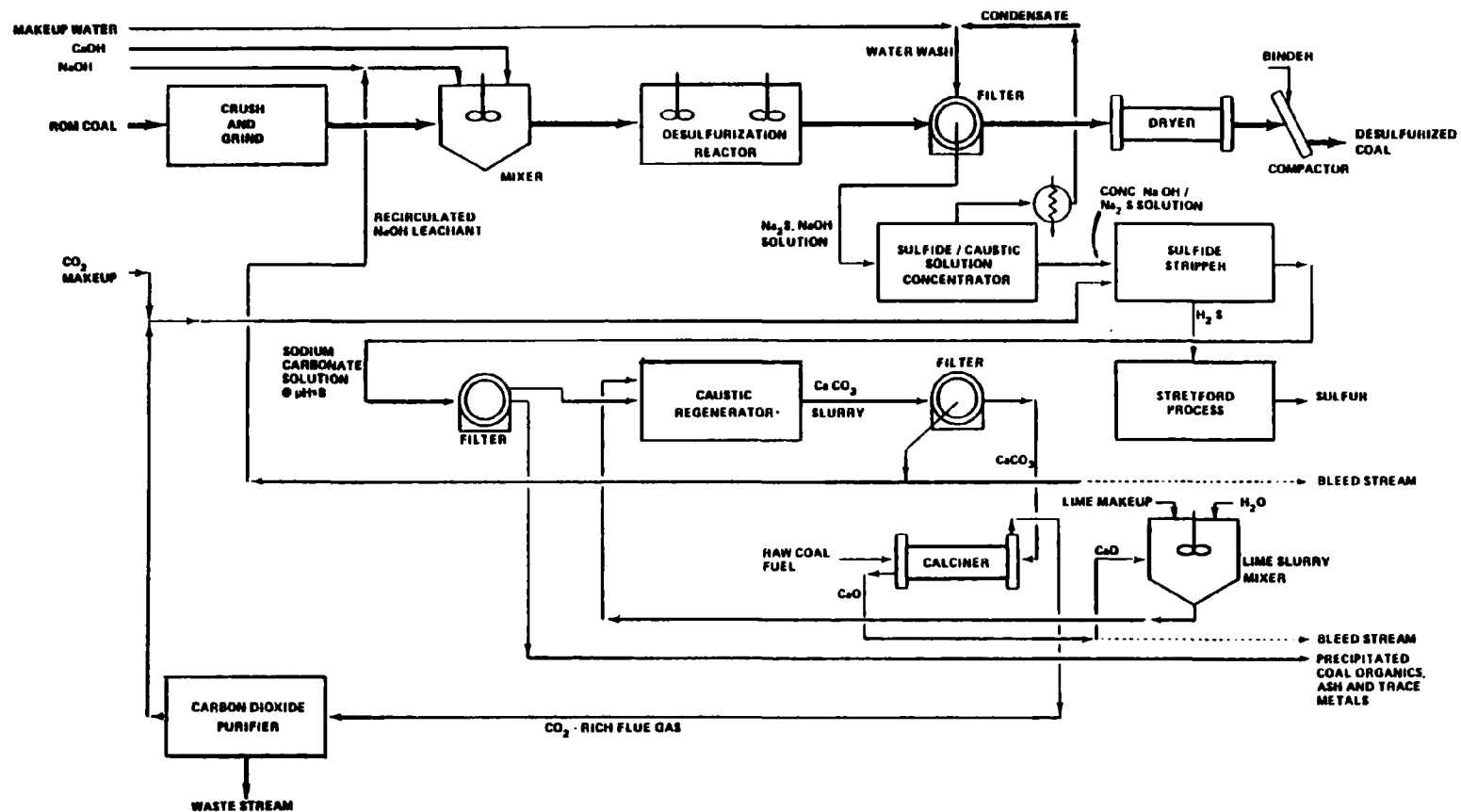


FIGURE 18 BATTELLE HYDROTHERMAL PROCESS FLOW SHEET

the raw coal and drying the treated coal. Battelle has included a charge to the BHCP for the cost of drying in their latest cost estimate. However, to make the cost estimate comparable to the other processes being considered in this study, i.e., for a plant not necessarily located adjacent to a power plant, the drying of the minus 200 mesh coal followed by a compaction (briquetting) step are included in the flow sheet and cost estimate.

The steps involved in leachant regeneration represent Battelle's latest thinking on this phase of the process - there is no performance data available as yet on continuous closed loop operation. The following Battelle-supplied information has been used in deriving the overall material balance shown in Figure 19.

- Five percent of the feed coal (including varying amounts of ash, coal and trace metals) is assumed to be dissolved in the desulfurization step. The dissolved material is precipitated as a result of pH change, and leaves the process as filter cake. Battelle has indicated that on an overall basis, between 5 and 10% of the average coal is solubilized. In the sulfide stripping operation, carbonation of the sodium sulfide-bearing caustic solution, results in lowering the pH to approximately 8 at which point most of the dissolved coal, ash and trace heavy metals precipitate from solution.
- An intermediate material balance (shown in Figure 19)²⁶ around the raw coal slurry preparation, desulfurization, solid-liquid separation and evaporation operations has been supplied by Battelle based on the values of the parameters shown in Table 39. The material balance shown in Figure 19 is based on a computer model of the process and conservatively assumes 100% and 0% removal of pyritic and organic sulfur, respectively, from the coal. On this basis, the product coal has 0.81% total sulfur (MAF) equivalent to $2.3 \text{ kg}/10^6 \text{ kg cal}$ ($1.3 \text{ lb}/10^6 \text{ BTU}$) of sulfur dioxide emissions (based on a $6,900 \text{ kg cal/kg}$ or $12,500 \text{ BTU/lb coal}$). Battelle makes a further assumption, believed to be conservative, that the

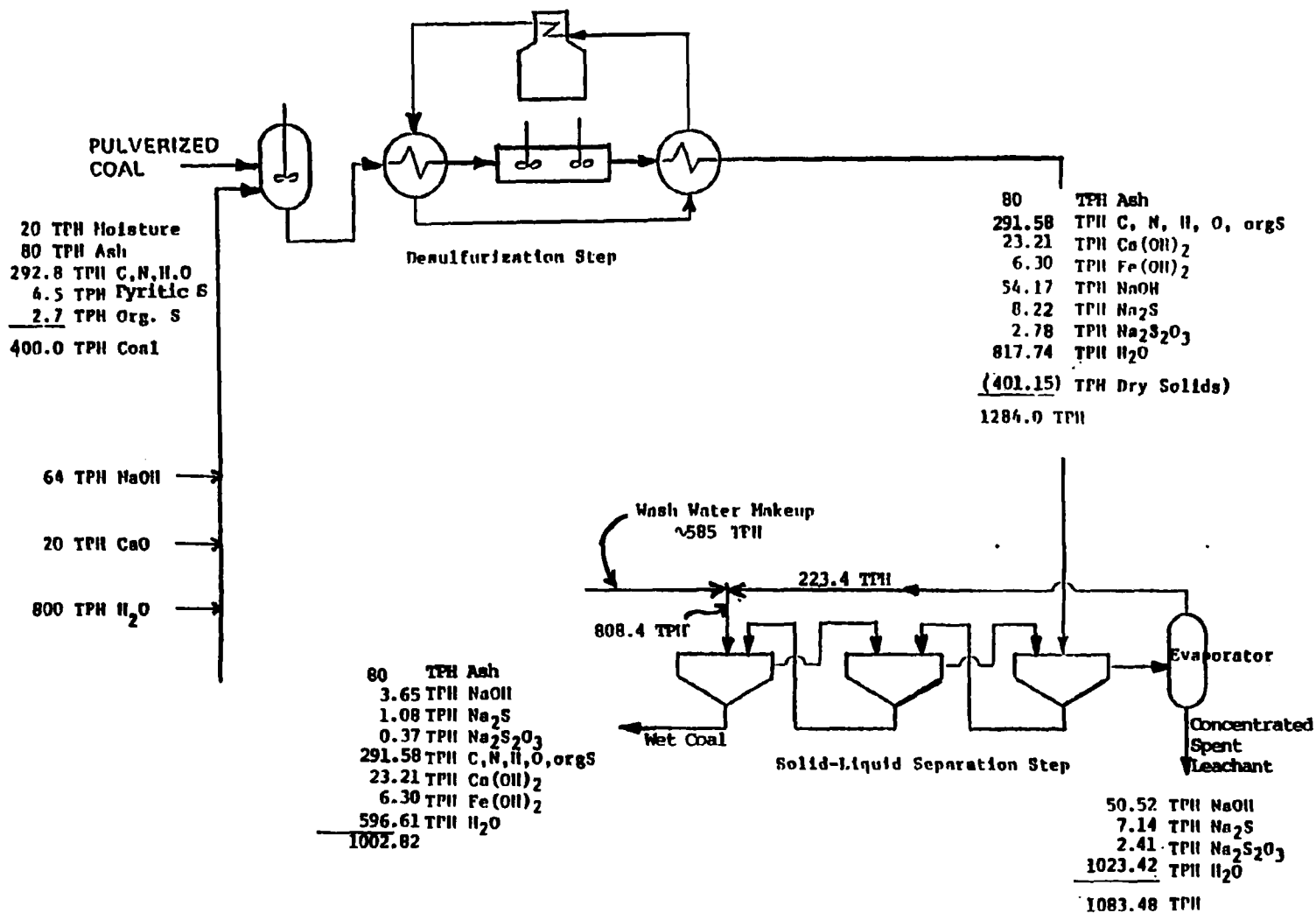


FIGURE 19 BATTELLE HYDROTHERMAL PROCESS: MATERIAL BALANCE FOR REACTOR AND SOLID / LIQUID SEPARATION SECTIONS

calcium in the product coal will capture at least another 10% of the sulfur (by a "getting" action) during combustion thereby reducing the net sulfur dioxide emission of this coal to 2.12 kg SO₂/10⁶ kg cal (1.17 lb SO₂/10⁶ BTU), which is below the EPA NSPS value. By-product sulfur would be generated at the rate of 2.9 metric tons (3.2 tons) per hour based on: (a) the above information, (b) an assumed 100% conversion of the sodium sulfide to hydrogen sulfide (H₂S) and (c) theoretically complete conversion of the H₂S to S in the Stretford process.

- All of the lime input to the process would remain with the treated coal. The excess of lime is intended to reduce sodium entrapment by the coal. The lime make-up rate, on a once-through basis is 14.5 metric tons (16 tons) per hour.
- Based on the Battelle intermediate material balance given in Figure 19, assuming 100% conversion of the sodium carbonate to sodium hydroxide and no leachant purge required, recycle NaOH would be supplied at the rate of 48.5 metric tons (53.5 tons) per hour, including fresh material at the rate of 3.8 metric tons (4.2 tons) per hour.

Battelle has supplied estimates of the heat and power consumption in the major process steps shown in Figure 19. Table 40 is a tabulation of the estimated values of the heat and power requirements of the BHCP.²⁶

It can be noted from Table 40, that limestone calcination and drying of the coal, make up 39% and 47%, respectively, of the heat consumption of the process. These two process steps together, in effect, use about 24% of the input coal heating value. There would be some economy in process heat requirements, if hot flue gas from the calcination operation can be used in the wet coal drying process.

Status of the Process

The original Battelle hydrothermal coal process has been under development at the Columbus Laboratories since 1960 under Battelle sponsorship. The desulfurization step has been carried through pre-pilot level (continuous

TABLE 40. ESTIMATED HEAT AND POWER CONSUMPTION OF THE
BATTELLE HYDROTHERMAL COAL PROCESS

Heat Consumption [based on 360 metric tons (400 tons) per hour of coal feed]

<u>Process Step(s)</u>	<u>Heat Consumption 10^6 kg cal/hr (10^6 BTU/hr)</u>
Desulfurization	33 (131)
Evaporation of water	47 (187)
Leachant regeneration	20 (78)
Limestone calcination	272 (1,081)
Drying of coal*	330 (1,308)
TOTAL	702 (2,785)

Thermal Efficiency of Process [based on 6,900 kg cal/kg (12,500 BTU/lb) of feed coal]

Coal heat input = $2,520 \times 10^6$ kg cal/hr ($10,000 \times 10^6$ BTU/hr)

Process heat consumption = 702×10^6 kg cal/hr ($2,785 \times 10^6$ BTU/hr)

Heat loss (solubilized coal) = 50×10^6 kg cal/hr (200×10^6 BTU/hr)**

Thermal efficiency (T.E.) = 70%

<u>Power Consumption</u>	<u>Power Consumption kwh/metric ton (kwh/ton) coal feed</u>
Desulfurization	6.8 (6.2)
Solid/liquid separation and washing	6.2 (5.6)
Calcination	18.4 (16.7)
Sulfur recovery plant (Stretford Process)	21.2 (19.2)
Off-sites	1.3 (1.2)
TOTAL	53.9 (48.9)

* Based on drying the filtered, washed coal with an initial 45% moisture to 5% moisture.

** Based on Battelle value of 5% loss of coal due to solubilization by leachant and a Versar assumption of 1,000 kg cal/hr (4,000 BTU/hr) for average heat content of this material.

bench-scale) laboratory investigations. In this effort, sulfur extraction from approximately twenty different eastern and midwestern bituminous coals have been studied. Battelle has published pyritic sulfur extraction data on 6 coals, organic sulfur extraction data on 6 coals, and overall sulfur reduction data on 6 coals.²⁷ In all of these studies, the SO₂ emission on the BHCP treated coals was equal to or less than the EPA-NSPS of 2.16 kg/10⁶ kg cal (1.2 lb/10⁶ BTU) for coal-fired steam generators.

Liquid/solid separation and regeneration of spent leachant are being studied in bench-scale equipment in an attempt to:

- establish definitive information as to whether the process can operate in closed-loop fashion; and
- improve the economic viability of the process by reducing the cost of these two high cost segments.

The EPA has funded a third area of interest in the BHCP: a combustion study on BHCP treated coals (Contract No. 68-02-2119). This study was a laboratory scale evaluation of BHCP treated coal combustion characteristics. This work was completed and reported in "Study of the Battelle Hydrothermal Treatment of Coal Process", to IERL, RTP, in November of 1976.

With respect to regeneration of spent leachant, experimental efforts have concentrated on screening the use of zinc and iron compounds as possible regenerants for spent leachant from the coal desulfurization step. Results so far have not indicated significant process viability for either of these two heavy metals as alkali regenerants. In the case of zinc, there are indications of residual zinc build-up in the coal as well as environmental problems expected when zinc sulfide is roasted to regenerate the zinc oxide. In the case of iron oxides or hydroxides as possible regenerants, there has been no notable success to date.

To date, no experimental work has been attempted on optimization of the solid, liquid separation treatment of the slurry from the desulfurization step. A computer model has been developed in order to optimize (on paper) the relationships between the parameters involved, including the method of

separation (filtration, centrifugation or thickening), the number of separation/washing stages involved, the wash water/dry solids ratio, the percent of water in the underflow coal and the amount of entrained sodium in the coal. These parameters have all been related to the cost contribution per ton of coal product. This study has shown that 9 countercurrent filtration/washing stages at an overall wash water/dry solids ratio of 1.5 with a final solids level of 45% in the underflow (filter cake), gave the lowest operating cost contribution per metric ton of product, i.e., \$10.50/metric ton (\$9.50/ton). At a cost contribution of \$10.50 metric ton (\$9.50/ton) with nine filtration/washing stages and 45% solids in the underflow, the lowest entrained sodium level was determined to be 0.0018, i.e., about 1.8 kg entrained sodium per metric ton of dry solid (3.6 lbs/ton).

Using a value of 0.005 metric ton of bound sodium in the treated coal per metric ton of dry solid, the total sodium input to the process (as 73% NaOH) would be about 0.016 metric ton per metric ton of dry product coal, i.e., 16 kg/metric ton (32 lb/ton). With caustic at \$176/metric ton (\$160/ton), the sodium input represents about 27% of the total cost contribution of the solid/liquid separation portion of the process. This caustic input value is still subject to experimental verification.

In the preliminary combustion studies with two BHCP treated coals under Contract No. 68-02-2119, the combustion characteristics of these coals were determined in two test facilities at Battelle, a one-half kg/hr (one lb/hour) laboratory-scale furnace and a 10-40 kg (20-80 lb) per hour multi-fuel furnace facility. Tests in both units were conducted with dry, pulverized BHCP treated coal. The results of these tests indicated that the treated coals would meet the present U.S. EPA-NSPS for sulfur dioxide emissions and that combustion of these coals proceeded as well or better than the corresponding raw coals.²⁶

The BHCP appears to have a significant effect on the trace elements levels of the treated coals. Table 41, compares the concentrations of twelve trace elements in raw coals and in the leached product for three Ohio coals.²⁷ Based on these results, there would be less trace metals emissions to be expected from combustion of BHCP coals as compared to raw coals. Varying quantities of the leached trace elements would be expected to precipitate

TABLE 41. TRACE ELEMENT REDUCTION IN COALS TREATED
BY THE BATTELLE HYDROTHERMAL PROCESS

Metal	Concentration, ppm		Percent Reduction
	Raw Coal	Leached Product	
Lithium	15	3	80
Beryllium	10	3	70
Boron	75	4	95
Phosphorus	400	80	80
Chlorine	20	2	90
Potassium	5000	200	96
Vanadium	40	2	95
Arsenic	25	2	92
Molybdenum	20	5	75
Barium	25	4	84
Lead	20	5	75
Thorium	3	0.5	83

* Average value for 3 Ohio coals: CN719-Seam 6, HN658-Seam 6A, and Jackson-Seam 4. Analyses were conducted by Battelle.

with the solubilized coal in the sulfide stripping operation and then be removed in the filter cake in the subsequent filtration operation.

Landfilling of this material could present some environmental problems.

A potentially serious problem indicated by the preliminary combustion studies is the potential for slagging and fouling of furnaces due to the high alkali content of the BHCP coals. Battelle has determined that the critical level for sodium in utility coals is 0.5%. It appears that sodium levels above 0.5% will make these coals unusable in dry-bottom furnaces due to lowered ash melting temperature and resulting slag-forming tendencies. Wet-bottom furnaces (slag-tapping type) may be adaptable to the high sodium coals although the possibility of fouling of heat transfer surfaces due to the formation of slag can occur in either type of furnace. Further combustion studies are needed to investigate the potential severity of this problem in prototype boiler units.

It should be noted that the function of the calcium hydroxide in the mixed leachant is to displace the sodium which can combine with the coal during the hydrothermal treatment. By the use of mixed leachant, Battelle hopes to keep the ultimate sodium level in the treated coal to 0.5% or below. However, a complicating factor is the presence of high ash levels in certain feed coals which seems to prevent the calcium oxide functioning as a sodium replacement in the treated coal. An example of this is the treatment of Martinka coal with the mixed leachant. This coal had a sodium level, after treatment, of 1.96 weight percent, which is believed to be due to the high ash content (20%) of the raw coal. The treatment of a lower ash coal (10%) with mixed leachant resulted in residual sodium levels of as low as 0.25 weight percent (Westland coal). Based on these results, Battelle is suggesting the necessity of monitoring all incoming coals to a BHCP plant so that suitable coal blending can be carried out to prevent high ash levels in the coal feed to the process.

Technical Evaluation of the Process

The BHCP is one of the few chemical coal cleaning processes that has made significant advances to a point permitting at least partial engineering evaluation. Based on the information available, a technical evaluation of the process follows.

Potential for Sulfur Removal--

The ability of the process to remove sulfur is shown in the table below.²⁷

PYRITIC SULFUR EXTRACTION BY THE BHCP

Source of Coal			Percent Pyritic Sulfur*		Extraction Efficiency, Percent
Mine	Seam	State	Raw Coal	BHCP Coal	
CN719	6	Ohio	4.0	0.1	99
Belmont	8	Ohio	1.6	0.1	92
NE41	9	Ohio	4.0	0.1	99
Ken	14	Ky.	2.1	0.2	92
Beach Bottom	8	Pa.	1.7	0.1	95
Eagle 1	5	Ill.	1.5	0.2	87

*Moisture and ash free basis. Coal samples were supplied from the various mines. Analyses were conducted by Battelle on raw and hydrothermally treated coals.

Ninety percent or greater pyritic sulfur removal has been demonstrated on a variety of bituminous coals from Ohio, Pennsylvania, Illinois and Kentucky. It is believed that pyritic sulfur can be essentially completely removed (95%) from any bituminous coal using the BHCP.

It is believed that the BHCP is capable of removing 25-50% of organic sulfur from a wide variety of coals. The table below²⁷ presents typical organic sulfur extraction data from the BHCP.

EXTRACTION OF ORGANIC SULFUR BY THE BHCP

Source of Coal			Percent Organic Sulfur*		Extraction Efficiency, Percent
Mine	Seam	State	Raw Coal	BHCP Coal	
Sunny Hill	6	Ohio	1.1	0.6	41
Martinka #1	Lower Kittanning	W. Va.	0.7	0.5	24
Westland	8	Pa.	0.8	0.5	38
Beach Bottom	8	W. Va.	1.0	0.7	30
Reign #1	4A	Ohio	2.3	1.1	52

*Moisture and ash free basis coal samples were supplied from the various mines. All analyses were conducted by Battelle on raw and hydrothermally treated coals.

Experiments have been conducted also on a semicontinuous bench-scale to confirm the results of laboratory batch experiments. The equipment has a capacity of about 9 kilograms (20 pounds) of coal per hour and all of the basic steps of the desulfurization process are included. Table 42 presents results of the continuous bench-scale experiments on two coals, Martinka and Renton - a West Virginia and a Pennsylvania coal respectively, along with similar data from laboratory batch operations.²⁹ These experiments were not necessarily run under the same conditions, but it is shows a comparison of desulfurized in the batch, bench-scale work compared to similar conditions in a continuous operation. The operation, however, has not yet employed recycled, regenerated reactants, so that the influence on leaching due to buildup of contaminants in the system is unknown.

Sulfur By-Products--

In the conceptualized BHCP using lime-carbon dioxide regeneration of the spent leachant, sulfur is removed from the process as hydrogen sulfide which is then converted to elemental sulfur using either the Claus or Stretford process (currently, Battelle prefers the Stretford process).

Benefit Analysis--

The main benefit associated with the BHCP, is the demonstrated removal of essentially all of the pyritic sulfur and a substantial portion (up to 50%) of the organic sulfur from a wide variety of bituminous coals. Additional benefits claimed for the process include the substantial removal of trace metals from the coal.

The process may produce a coal having substantial problems in the coal combustion process due to the increased sodium level in the BHCP treated coal. Coals with high sodium content increase the slagging tendency of the ash and create ash removal problems in dry bottom boiler. Additionally, high sodium levels in coal cause fouling of heat transfer surfaces in all types of boilers.

TABLE 42. CONTINUOUS BENCH-SCALE RESULTS FOR THE BATTELLE PROCESS

Coal Source		Sulfur Analysis, wt %		SO ₂ Equivalent, kg/10 ⁶ kg cal (lb/10 ⁶ BTU)	
		Raw	BHCP	Raw	BHCP
Mine	Seam	Coal	Coal	Coal	Coal
<u>1. Laboratory Scale</u>					
Martinka No. 1	Lower Kittanning (W. Va.)	1.07	0.39	3.87 (2.15)	1.57 (0.87)
Renton	Upper Freeport (Pa.)	1.32	0.52	4.36 (2.42)	1.66 (0.92)
<u>2. Continuous Bench-Scale Studies</u>					
Martinka No. 1	Lower Kittanning (W. Va.)	2.77	0.76	7.20 (4.00)	1.89 (1.05)
Renton	Upper Freeport (Pa.)	1.20	0.60	4.32 (2.40)	1.42 (0.79)

Environmental Aspects—

The BHCP is claimed to be essentially free of environmental problems due to the "closed-loop" feature of the process. However, this assertion is open to question due to the following factors:

- The feasibility of the closed-loop feature in a continuous process is as yet undemonstrated. In limited batch-type evaluation of the carbon dioxide/lime regeneration process for the mixed leachant (four complete recycles of the regenerated mixed leachant were carried out), there is a tendency for oxysulfur compound build-up which inhibits the desulfurization ability of the recycled mixed leachant. A fairly sizable purge stream may have to be discharged from the system for disposal. This stream would contain some dissolved organics and trace metals from the hydrothermally treated coal. Additionally, pH adjustment of this stream prior to disposal would create large quantities of dissolved salts. Disposal of this stream could therefore pose environmental problems.
- In the processing scheme proposed by Battelle, the ash solubilized by the hydrothermal treatment would precipitate as a result of the carbonation of the spent leachant (in the sulfide stripping step). The filtered ash would contain some precipitated metals and insoluble inorganics and could pose environmental problems if placed in ordinary landfills.
- Elemental sulfur recovery from the sulfide stripping operation will be accomplished by treatment of the hydrogen sulfide in either a Claus or Stretford process. Tail-gas from the Claus or Stretford process will require scrubbing for sulfur dioxide or hydrogen sulfide removal, respectively.

- Conveying of the minus 200 mesh dry treated coal to either a briquetting operation or intermediate storage, may create particulate emissions problems (and possible spontaneous combustion problems due to the pyrophoric nature of this material). Use of baghouses, water sprays and cyclones may be necessary for recovery of the sub-micron-size solids before venting the gases to the atmosphere.
- In the closed-loop calcination of the precipitated calcium carbonate to regenerate calcium oxide, the possibility of impurity buildup in the lime, i.e., heavy metals and ash components from the coal, could require periodic purge of this material. Disposal of the purged material could pose environmental problems.

Problem Areas—

The two overriding problem areas in the BHCP are:

- Demonstration of a technically and economically feasible closed-loop process by which the alkaline leachant may be regenerated and recycled, has not yet been achieved.
- A value of 0.5 weight percent or less residual sodium in the treated coal, in order to prevent slagging and fouling tendencies of the ash during firing in utility furnaces, has not been achieved for some coals.

Severe corrosion problems may occur in the desulfurization reaction since alkalis at high temperatures $>250^{\circ}\text{C}$ (482°F) in the presence of water are notorious for initiating stress corrosion failures of materials. Battelle has found only one material (Inconel 671 alloy), which has shown the possibility of being able to withstand the desulfurization reaction conditions without undergoing relatively rapid failure. However, this material has not been evaluated in any long-term production cycle under actual reaction conditions (as part of a prototype vessel). Inconel 671 reactors will be extremely costly.

Preliminary indications are that filtration of the mixed leachant treated coal slurry is extremely slow, even at relatively coarse mesh sizes (up to minus 20 mesh).

Nothing is really known at this time about the influence of the buildup of contaminants in the leaching ability of the recycled mixed leachant. Contaminants can interfere with the reactions involved in the calcium oxide-carbon dioxide regeneration system thereby preventing efficient recovery of the leachant. Laboratory-scale investigations have only been able to affect 84% sulfur removal from the spent leachant using carbonation-liming, even though Battelle believes that essentially 100% removal can be achieved.²⁹ It should be noted that the lower result was achieved in an oxygen-free atmosphere is essential in order to obtain complete sulfur removal from the spent leachant, i.e., avoid the formation of oxysulfur compounds which are not reactive in the carbonation step.

Coal loss due to solubility by the alkali leachant at elevated temperatures, may be more severe in actual practice than is now anticipated.

R&D Efforts and Needs—

Based on a discussion with Battelle personnel and examination of Battelle reports available to Versar on the BHCP, the following R&D efforts and needs have been identified.

- Determine the conditions for sulfur removal during the leaching step to enable optimization of: residence time; temperature; particle size of coal; water to coal ratio; sulfur removal; leachant concentration; leachant alternatives; and coal loss due to solubilization by the leachant.
- Develop the best technology for separating the treated coal from the spent leachant and for washing the coal free of sodium and sulfur after treatment.

- Study the effect of coal mesh size on water retention of mixed leachant-treated coal.
- Determine the best trade-off between reaction conditions, reaction systems, and post-treatment for consistently keeping the residual sodium content in the treated coal to 0.5 percent or lower.
- Identify and demonstrate the best technology to regenerate the spent leachant in a closed loop process.
- Determine in prototype reaction equipment the best materials of construction for the critical steps of the process.
- Continue the studies on the effect of residual sodium levels in treated coal on the slagging and fouling tendencies in boilers.
- Determine the fate of trace metals extracted from raw coal by the BHCP, including possibilities for recovering these materials as a highly enriched stream.
- Apply the BHCP technique to other types of coals including sub-bituminous, lignite and bituminous coals with a high ratio of organic to pyritic sulfur (55-60% organic, 45-50% pyritic), in order to determine the applicability of the process to as wide a variety of coals as possible.

No attempt has been made to prioritize the list presented above. However, when completed, the present Battelle laboratory studies (EPA Contract No. 68-02-2187) on spent leachant regeneration and solid-liquid separation techniques should provide a definitive answer on the viability of the BHCP as a closed-loop process.

Process Economics

Battelle has revised and updated an earlier cost estimate of their conceptualized BHCP. The current estimate reflects the results of bench-scale experiments carried out in order to bracket the range of variables involved and at least establish the most likely methods to be used in the closed-loop process.²⁹

A summary of BHCP economics is presented in Table 43. Details on the capital costs and operating costs are given in Tables 44 and 45 respectively. Versar has included the following modifications of the Battelle estimate:

- An estimate of the cost of coal preparation including handling, grinding and storage facilities.
- An estimate of the cost of product coal compaction, handling and storage.
- Proration of the Battelle plant size of 363 metric tons (400 tons) per hour to 300 metric tons (333 tons) per hour. The latter capacity is equivalent to 7,256 metric tons (8,000 tons) per day. A 0.6 exponential factor was used to adjust plant size.
- A 20% contingency factor was added to the Battelle capital cost.
- Variable operating costs were prorated based on the 7,256 metric ton (8,000 tons) per day coal processing rate.

The auxiliaries and offsites are not shown in the conceptual process flow diagram (Figure 18), but are included in the Battelle cost estimate. The major item is silo storage for 20 days' production. Other significant offsites included in the design are a steam plant and cooling towers. An allowance has also been made for site preparation, buildings (offices, maintenance shop, laboratory, change house, etc.), electrical distribution, and offsite piping.

The Bechtel Corporation has also prepared a cost estimate for BHCP⁸ using the same basic flowsheet and with the two plant capacities being roughly equivalent. Battelle uses 360 metric tons (400 tons) per hour of raw coal feed with a 90% operating factor, and Bechtel uses 300 metric tons (333 tons) per hour of raw coal feed with a 100% operating factor. The Bechtel process scheme features a more elaborate system of process heat recovery and heat utilization than does the Battelle process and also omits the cost of an evaporation system to concentrate the diluted spent caustic leachant from the filtration/washing step. The Bechtel process flowsheet includes

receiving, storage and grinding of the raw coal, plus compaction of the dried treated minus 200 mesh coal to permit handling and transportation to a utility not located adjacent to the coal treatment plant.

A major difference in the approach used in the two operating cost estimates, is the use of cleaned coal by Bechtel to provide heat input to the calcium carbonate calcination operation, thereby reducing the net coal yield from the process. Battelle uses raw coal as fuel in the calcium carbonate calcination step since they believe that the sulfur dioxide generated would be absorbed by the lime formed in the process. However, no estimate is provided by Battelle of the purge rate required from the regenerated lime stream, in order to control calcium sulfate build-up.

An analysis of the Bechtel data indicates that the annual operating costs developed by Battelle and Bechtel for the BHCP are quite comparable even though the Battelle capital cost is approximately 60% greater than that of Bechtel. None of the major operating cost components show any large differences between the two estimates. Both estimates reflect the energy-intensive nature of the process, showing 25-35% of the operating cost being accounted for by fuel cost. Caustic soda makeup cost could go significantly higher in actual practice if the mixed-leachant approach fails to minimize sodium entrapment by the treated coal. Another unknown factor which could add significantly to the BHCP operating cost, would be the need for spent leachant purge, if this were required due to impurity buildup in closed-loop operation. Battelle estimates that a 10 percent purge would require an additional 0.016 metric ton of NaOH/metric ton of coal processed, adding \$2.82/metric ton (\$2.56/ton) of input coal processed [\$3.48/metric ton (\$3.16/ton) of equivalent heating value product coal]. Additionally, spent leachant purge will probably incur appreciable disposal costs.

TABLE 43. SUMMARY OF ECONOMICS FOR THE BATTELLE
CHEMICAL COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Gross roots plant installation

95% weight yield, 88% heating value recovery

Installed Capital Cost: \$168,630,000

Annual Operating Costs

on Clean Coal Basis: \$74,203,000 process cost, excluding coal cost
\$140,203,000 process cost, including coal cost*
\$32.61/metric ton (\$29.58/ton), excluding coal cost
\$61.63/metric ton (\$55.90/ton), including coal cost*
\$5.15/10⁶ kg cal (\$1.30/10⁶ BTU), excluding coal cost
\$9.74/10⁶ kg cal (\$2.45/10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 44. INSTALLED CAPITAL COST ESTIMATE FOR THE BATTELLE
CHEMICAL COAL CLEANING PROCESS

	<u>\$ 1977</u>
Coal handling and preparation*	\$ 16,600,000
Desulfurization process costs ^Δ	108,000,000
Compacting and product handling*	5,120,000
Building and miscellaneous [†]	---
Utilities (off-sites) [†]	---
Site development and general [†]	---
Subtotal	<u>\$129,720,000</u>
Engineering design @ 10%	12,970,000
Contingency @ 20%	<u>25,940,000</u>
Total Installed Plant Capital (TPC)	\$168,630,000

* Versar estimate.

Δ Battelle estimate for a grass roots plant including all off-site requirements, scaled to 7,200 metric tons (8,000 tons) per day plant size.

† Included in the grass roots plant cost under desulfurization process cost.

TABLE 45. ESTIMATED ANNUAL OPERATING COSTS FOR THE BATTELLE
CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	\$ 19,814,000
Taxes @ 2% TPC	3,370,000
Insurance @ 1% TPC	1,680,000
Labor (direct, supervisory and additives)	2,100,000
General and administrative @ 1.5% TPC	2,530,000
Maintenance and supplies	10,000,000
Utilities:	
Electric power	3,800,000
Water	300,000
Steam and fuel*	19,000,000
Chemicals:	
Caustic soda	3,900,000
Lime	3,200,000
Binder	4,509,000
Waste Disposal	--
Total Annual Processing Cost	\$ 74,203,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$140,203,000

* Additional raw coal is purchased to provide fuel needs for calcium carbonate calcining and drying. It has been assumed that the sulfur dioxide generated would be absorbed by the lime formed in the process.

JPL CHEMICAL COAL CLEANING PROCESS

The Jet Propulsion Laboratory (JPL), California Institute of Technology, at Pasadena, California, is developing a chemical coal cleaning process which attacks both pyritic and organic sulfur compounds in coal, and allegedly results in the removal of up to 75% of the total sulfur in coal.³⁰ Both types of sulfur are attacked during a low temperature coal chlorinolysis step, followed by hydrolysis and dechlorination.

Process Description

A flow diagram based on the JPL process is shown in Figure 20.³¹ Chlorine gas is sparged into a suspension of moist, pulverized coal (minus 100 to minus 200 mesh) in methyl chloroform (1,1,1-trichloroethane) at 74°C (165°F) and atmospheric pressure for 1 to 4 hours. The suspension consists of approximately 1 part coal to two parts solvent. Chlorine (Cl_2) usage is 3 to 3.5 moles of chlorine per mole sulfur, or about 250 kg Cl_2 per metric ton (500 lbs/ton) of coal. Moisture is added to the feed coal to the extent of 30-50% by weight.

After chlorination the coal slurry is distilled for solvent recovery, and the solvent is recycled for reuse in the chlorinolysis step. The chlorinated coal is then hydrolyzed with water at 50-70°C (120-150°F) for 2 hours, and then filtered and washed. The coal filter cake is simultaneously dried and dechlorinated by heating at 300-500°C (570-930°F) with superheated steam (or possibly a vacuum) for about 1 hour.

There are a number of by-product streams which are as follows:

- Vented gas from the chlorinolysis reactors contains unreacted chlorine (Cl_2) and by-product hydrogen chloride (HCl). The gas is cooled to condense Cl_2 , which is recycled, and the relatively non-condensable HCl gas is piped to a Kel-Chlor process unit which converts the HCl to Cl_2 .
- Vapors from the solvent evaporation step are cooled to permit condensation and recycling of the methyl chloroform. The HCl gas is piped to a Kel-Chlor unit for conversion.
- Filtrates and wash water from the filtration of hydrolyzed coal contain hydrochloric acid and sulfuric acid. The HCl is driven off in a stripper and

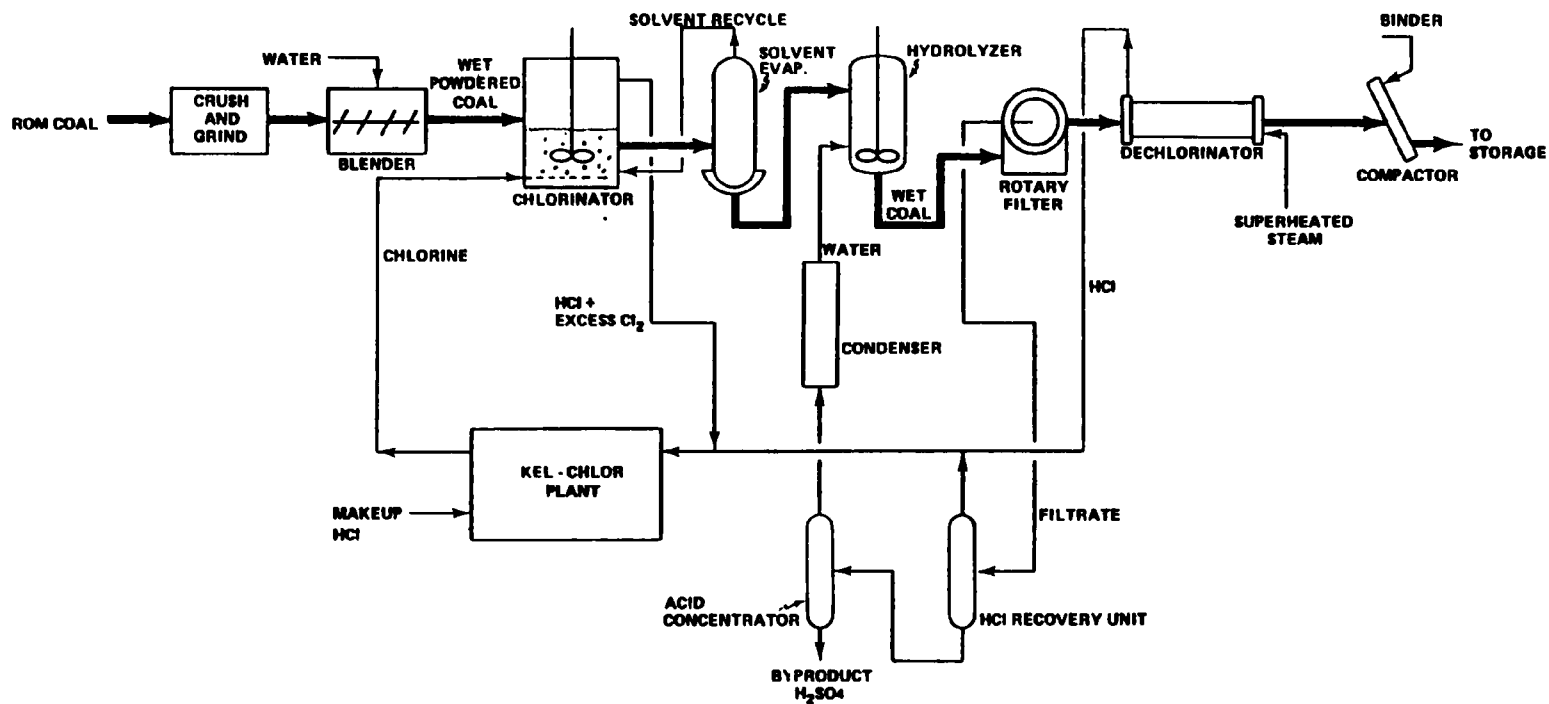
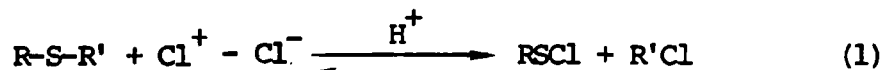


FIGURE 20 JPL PROCESS FLOW SHEET

recycled to a Kel-Chlor unit. The residual dilute sulfuric acid is concentrated to a saleable 91% sulfuric acid.

● Superheated steam exhausting from the dechlorination will also contain HCl gas which must be condensed as hydrochloric acid and recycled to a Kel-Chlor unit for chlorine recovery.

The chemistry of this process is somewhat complex, but is hypothesized as follows:³⁰

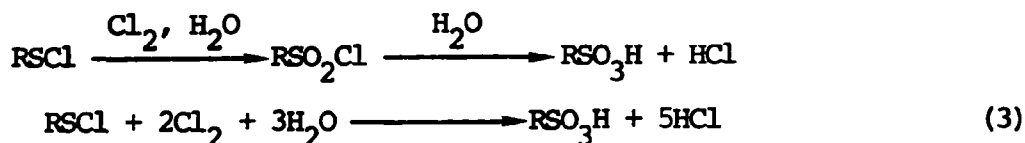


where R and R' represent hydrocarbon groups, and S stands for sulfur.

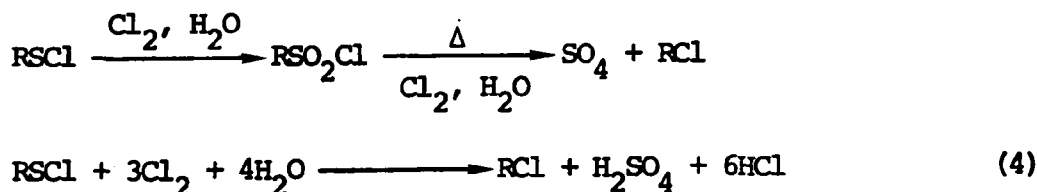
S-S Bond (Electrophilic cleavage) REACTION -



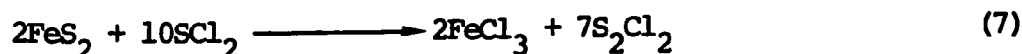
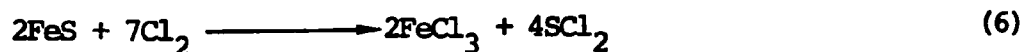
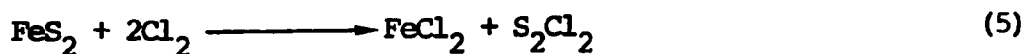
Sulfonyl chloride is oxidized to sulfonate or sulfate according to the following reactions:



or



pyritic sulfur reactions are summarized as follows:

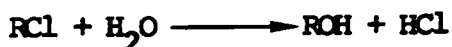




"These reactions are exothermic in nature and occur favorably at moderate temperature. The overall chlorine requirement for conversion of organic sulfur to sulfonate sulfur and sulfate sulfur are approximately 3 moles of Cl_2 and 4 moles of Cl_2 respectively per mole of organic sulfur and 3.5 moles of Cl_2 per mole of inorganic sulfur." ³⁰

"In the presence of water and at a temperature (i.e., $> 50^\circ\text{C}$) higher than room temperature, the S_2Cl_2 formed from FeS_2 chlorination is readily converted to HCl and H_2SO_4 . At room temperature, without the presence of adequate moisture content, this reaction is slow and S_2Cl_2 may react with organic compounds to form organo-sulfur compounds. On the other hand in an organic solvent, at a slightly elevated temperature, the rate of chlorination of coal is slower than in aqueous media at room temperature. Reaction in an organic solvent gives a greater degree of structural loosening of coal and consequently may remove more organic sulfur with a lesser degree of chlorination. Structural loosening of coal by the action of the organic solvent will make chlorine more accessible to sulfur compounds. High chlorine solubility in an organic solvent may also be advantageous for desulfurization. Moreover, an organic solvent may dissolve some of the organo-sulfur compounds. Chlorination of the coal matrix is mainly a substitution reaction and hydrogen chloride is evolved as a product. If coal is chlorinated under mild conditions the chlorine can be completely removed as hydrogen chloride by heating at $300\text{--}500^\circ\text{C}$. Chlorination at high temperature and pressure results in coal which is difficult to dechlorinate."

Chlorinated coal is hydrolyzed to give hydrochloric acid according to the following reaction.



where R represents a hydrocarbon group in coal.

The sulfur converted to sulfates or sulfonate is water soluble and is leachable by water washing at 60°C (140°F) with retention times up to 2 hours in a stirred reactor.

Dechlorination--

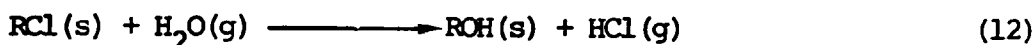
After hydrolysis, the coal is dechlorinated by heating in steam or an inert gas atmosphere. This can be accomplished easily because of chlorination at low temperature. The possible reactions during dechlorination are:

in an inert gas atmosphere:



and

in steam atmosphere:



This reaction is endothermic and proceeds favorably at a moderately high temperature [300-500°C (570-930°F)].

According to the literature, steam will assist pyritic sulfur removal. Dechlorination in a steam atmosphere proceeds by substitution of -Cl in chlorinated coal by -OH and possibly -H groups from H₂O. "No loss of heating value is experienced for the processed coal when dechlorinated in a steam atmosphere."³⁰

Status of the Process

As of mid-July, 1977, effort on this process was on a laboratory scale batch operation using 100 g. coal samples. It was expected at that time that larger scale (1 kg) batch runs would be initiated in the near future, and at a still later date, a 1 kg/hour mini-pilot plant would be constructed and operated.

The early stages of the process research work were supported by the National Aeronautics and Space Administration (NASA) under Contract No. NAS 7-100. Recently the project obtained support from the Bureau of Mines for a period of approximately 16 months. The new contract requires that specific coals be evaluated under the sponsored program. The nine coals selected are given in Table 46.

TABLE 46. PROPERTIES OF NINE SELECTED COALS FOR THE JPL
PROCESS EXPERIMENTS*

<u>ERDA PSOC NO.</u>	<u>SEAM, COUNTY & STATE</u>	<u>RANK</u>	<u>SULPHUR FORMS</u>		<u>TOTAL</u>
			<u>ORGANIC</u>	<u>PYRITIC</u>	
108	Pittsburgh, Washington, Pennsylvania	HVA (Bit.)	1.07	2.06	3.13
219	Kentucky #4, Hopkins, Kentucky	HVA	1.08	1.40	2.56
190	Illinois #6, Knox, Illinois	HVA	1.90	1.05	3.05
276	Ohio #8, Harrison, Ohio	HVA	1.73	1.34	3.07
026	Illinois #6, Saline, Illinois	HVC	2.08	4.23	6.66
342	Clarion, Jefferson, Pennsylvania	HVA	1.39	5.01	6.55
240A1	Big D, Lewis, Washington	Subbit.B	1.75	1.60	3.36
097	Seam 80, Carbon, Wyoming	Subbit.A	0.84	0.38	1.23
086	Zap, Mercer, N. Dakota	Lignite	0.63	0.56	1.22

* This table was obtained from JPL

Technical Evaluation of Process

Potential for Sulfur Removal—

The process claims a 97-98% weight recovery of input coal, with about a 2% loss in heating value, and 70-75% removal of total sulfur. Two high sulfur coals have been examined carefully for sulfur removal. The Illinois No. 5 high volatile bituminous coal from Hillsboro mine had 4.77% total sulfur content. The other high volatile bituminous coal was a Kentucky No. 9 coal from Hamilton, Kentucky. Proximate analyses of these two coals are given in Appendix VI .

Experimental data obtained with Illinois No. 5 (Hillsboro) coal is presented below.

JPL PROCESS: PRELIMINARY CHLORINOLYSIS DATA FOR ILLINOIS NO. 5 COAL DESULFURIZATION *

Sulfur Form	Raw Coal (% Sulfur)φ	Treated Coal (% Sulfur)φ	Sulfur Removal (%)
Pyritic	1.89	0.43	77 [†]
Organic	2.38	0.72	70
Sulfate	0.50	0.35	100 ^Δ
Total	4.77	1.50	76

* (Chlorination - stirred reactor, 74°C(165°F), 1 atm (14.8 psig), 1 hour, powdered coal 100-150 mesh with 50% water, methyl chloroform to coal 2/1; hydrolysis and water wash - stirred reactor, 60°C(140°F), 2 hours, excess water).

φ Analyses by Galbraith Laboratories, Inc., Knoxville, Tennessee

Δ Additional water washing should remove 100% of sulfate

† Up to 90% pyritic sulfur removal has been achieved in other conditions

The overall sulfur removal is 76% with a reduction from 4.77% to 1.50%. Results of experiments with this coal indicate that removals up to 70% organic sulfur, 90% pyritic sulfur and 76% total sulfur have been achieved.

The kinetic data for chlorination and desulfurization of minus 100 mesh, Illinois No. 5 coal are presented in Figure 21.³¹ The initial rate of chlorination is very fast. The chlorine content in coal is 23% in half an hour and then slowly increases to 26% within the next one and a half hours. Within the initial half an hour period most of the pyritic sulfur and a portion of organic sulfur are converted to sulfate sulfur. In the next one and a half hour period pyritic and organic sulfurs are slowly converted to sulfate sulfur. Based on the sulfur balance, the gain in sulfate sulfur is equal to the combined reduction of pyritic and organic sulfurs. The above reactions extend to the hydrolysis period. The overall sulfate compounds produced either directly or indirectly through sulfonate are removed from coal in the hydrolysis step as indicated by the analysis of hydrolysis solution.

Experimental data obtained from a run on minus 200 mesh Kentucky No. 9 (Hamilton, Ky.) coal is given below.

PRELIMINARY CHLORINOLYSIS DATA FOR THE JPL DESULFURIZATION
PROCESS ON BITUMINOUS COAL (HAMILTON, KENTUCKY)*

Sulfur Form	Raw Coal (% Sulfur) Δ	Treated Coal (% Sulfur) Δ	Sulfur Removal (%)
Pyritic	0.08	0.03	62.5
Organic	2.67	1.16	56.5
Sulfate	0.15	0.29	100 [†]
Total	2.90	1.48	59.0

* Chlorination - stirred reactor, 74°C(165°F), 1 atm (14.8 psig), up to 4 hours, minus 200 mesh coal with 30% water, methyl chloroform to coal 2/1; hydrolysis and water wash - stirred reactor, 60°C (140°F), 2 hours, excess water.

Δ Analyses by Galbraith Laboratories, Knoxville, Tennessee

[†] 100% sulfate removal by added water wash.

The sulfur content of this coal is predominately organic (>90%). About 57% of the organic sulfur, and 59% of the total sulfur, are removed.

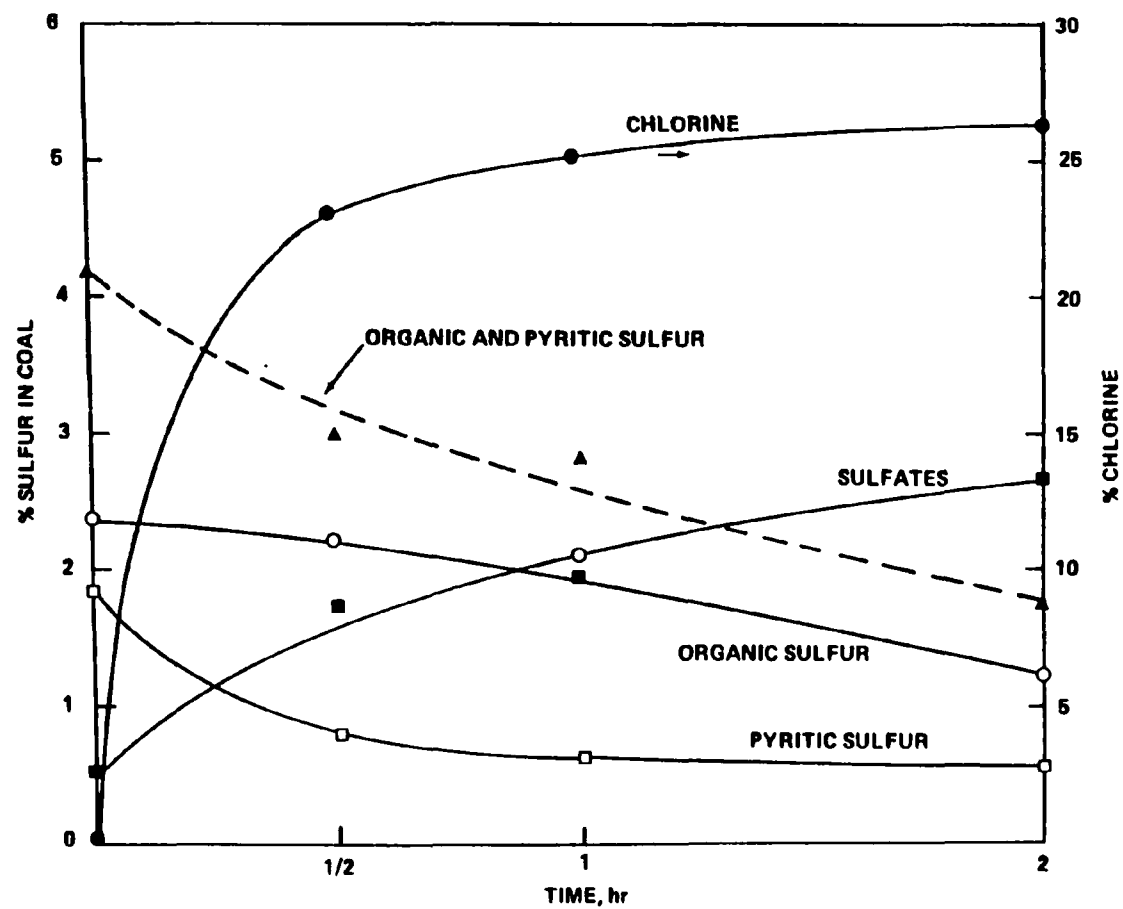


FIGURE 21 JPL PROCESS: PERCENT SULFUR AND CHLORINE IN COAL VS. TIME OF CHLORINATION

The data on the above two coals is the only detailed experimental results available at this time. Based on these results and discussions with JPL project personnel, it is concluded that the removal of pyritic sulfur by the JPL process is somewhat more complete than removal of organic sulfur. Consequently, if a high percentage of total sulfur removal is desired, the coal should be rich in pyritic sulfur rather than in organic sulfur. Neither product from the two above experiments will meet EPA-NSPS SO₂ emissions of 2.16 kg/10⁶ kg cal (1.2 lb SO₂/10⁶ BTU) when burned. A more accurate assessment of the sulfur removing potential of this process must therefore await results from the 9 coals to be tested under the Bureau of Mines contract.

Sulfur By-Products—

All sulfur removed from coal by the JPL process is converted to sulfate ion (SO₄⁼), and as presently conceived, this sulfur species will be retained in aqueous solution as sulfuric acid, concentrated to about 91% and sold. However, analysis of the JPL process scheme indicates that trace metals extracted from the coal will exit the system with the by-product acid. Therefore, it is doubtful that the impure sulfuric acid by-product can be sold without some prior clean-up.

Environmental Aspects—

There appear to be several severe potential environmental problems associated with this process. The hydrocarbon solvent used for the chlorinolysis reaction is 1,1,1-trichloroethane which has been listed by the EPA as a priority pollutant. Most of the substances on the list of priority pollutants are suspected carcinogens. The release of even small quantities of this material to the environment will probably be prohibited from a new source processing plant. Vent gases from the chlorinolysis reactors contain chlorine and by-product hydrogen chloride. Although these will presumably be sent to the Kel-Chlor process unit there is a potential for release of gases from this process unit. Filtrate from the hydrolysis unit will contain hydrochloric acid, sulfuric acid and probably chlorinated hydrocarbons and organic sulfonates. This filtrate will be concentrated in a sulfuric acid concentration step which will probably require a bleed stream to remove impurities from the concentrated sulfuric acid product. The disposal

of this bleed stream will present some environmental problems. Also there may be some environmental problems associated with the operation of the Kel-Chlor Chlorine recovery process.

Benefit Analysis—

The JPL chemical coal cleaning process provides a number of advantages which include: a significant degree of sulfur removal, including organic sulfur removal; removal of a number of trace metals contained in the original coal which would otherwise result in undesirable emissions to the atmosphere; and a product coal that is useful for direct combustion or for gasification operations since the treated coal is alleged to be non-caking and non-swelling.

Data relating to the degree of removal of trace metals in JPL process-treated-coal is given in the following Table.³¹

JPL PROCESS RESULTS ON TRACE METAL REMOVAL FROM COAL*

Elements	Original Coal ^Δ (ppm)	Treated Coal ^Δ (ppm)
As	2	1
Hg	0.6	<0.5
Ti	476	460
Pb	18	4
Va	15	<1
P	736	126
Se	<1	<1
Li	9	3
Be	3	1
Ba	38	30

* A high sulfur bituminous coal with 11% ash, from Hillsboro, Illinois chlorinated at 74°C (165°F) and atmospheric pressure for 1 hour, followed by aqueous leaching at 60°C (140°F) and atmospheric pressure for 2 hours.

^Δ Chemical analyses were conducted by the Galbraith Lab., Inc., Knoxville, Tennessee.

Although the JPL process is based on chlorination and dechlorination of coal, the product coal contains no more than, or even less chlorine than the elemental chlorine in the raw coal. Data demonstrating this is given in the following table.

JPL PROCESS PRELIMINARY DECHLORINATION DATA (HILLSBORO, ILLINOIS BITUMINOUS COAL)

Coal	Dechlorination			Elemental Chlorine (wt.%)
	Temp °C (°F)	Time (Hrs)	Atmosphere	
Raw	-	-	-	0.14
Chlorinated coal (after hydrolysis)	-	-	-	11.0
Dechlorinated coal	450 (840)	1	Steam	0.064
Dechlorinated coal	500 (930)	1	Vacuum	0.15-0.30
Dechlorinated coal	550 (1,020)	1	Vacuum	0.06

The dechlorination can be accomplished by either superheated steam treatment or by imposing a vacuum. The former step appears to be the one of choice, and it serves an important secondary function of drying the coal.

Problem areas—There are a number of real and potential problem areas which can adversely affect the technical and economic feasibility of the process. Some of these problem areas are as follows:

- Chlorine, hydrogen chloride (gas), hydrochloric acid, and sulfuric acid (dilute and concentrated) are all utilized or produced by the JPL process. All are highly acidic and corrosive in nature and will require special, and therefore expensive, materials of construction. In several steps of the process two of these chemicals coexist. For example, the vent gases from the chlorination step and from the subsequent distillation step will probably

contain hydrogen chloride and chlorine, plus some methyl chloroform solvent vapor. The wastewater from the coal filtration step after hydrolysis, contains hydrochloric acid, sulfuric acid plus chlorinated hydrocarbons and organic sulfonates. The exhaust steam from the dechlorination step [carried out at 300° - 500°C (570° - 930°F)], contains hydrogen chloride, and may present a very severe corrosion problem.

- The JPL process as presently conceived recovers all sulfur removed from the coal as sulfuric acid. It appears that all trace metals removed from the coal will remain with this acid, and tend to make this by-product relatively unmarketable.
- A key factor in the economic feasibility of the JPL process is the cost of chlorine. JPL has estimated the usage of chlorine to be about 250 kg/metric ton (500 lbs/ton) of coal (dry basis). The exact requirement for chlorine will largely depend on the sulfur content of a given coal. Purchase of chlorine for usage on a once through basis is out of the question, since this commodity presently sells for \$150-\$165/metric ton (\$135-\$150/ton). At these prices, the cost of chlorine alone would be about \$39/metric ton (\$35/ton) of feed coal.

Furthermore, there would be equivalent large quantities of by-product hydrogen chloride to be stored and disposed of. There are two major processes for conversion of hydrogen chloride to chlorine. The "Uhde" process converts hydrochloric acid to chlorine by an electrolytic process, which is electrical energy intensive. The "Kel-Chlor" process of Pullman Kellogg Div. of Pullman, Inc. is believed to be less energy intensive and it is used by DuPont in one commercial installation on the Gulf Coast of Texas. JPL contemplates incorporating a Kel-Chlor unit in their system and has included a "Kel-Chlor" unit in their rough flow-sheet.

The cost of producing chlorine by the "Kel-Chlor" process varies with a number of factors, including:

- whether the hydrogen chloride is available as a dry gas (preferable) or as a dilute solution.
- the purity of the delivered chlorine (oxygen being the main impurity likely).
- whether gaseous or liquified chlorine product is required.

Not all of the conditions imposed on a "Kel-Chlor" facility by the JPL process can be defined at this time. Nevertheless, a rough figure of \$38.60/metric ton (\$35/ton) of "Kel-Chlor" chlorine has been used in costing the JPL process. This price of \$38.60/metric ton of chlorine includes all manufacturing costs, fixed costs including depreciation and interest, and assume a source of hydrogen chloride at no cost.³² At a rate of 250 kg Cl₂/metric ton (500 lb/ton) of coal this will add about \$10/metric ton (\$9/ton) of coal before any other cost item is considered.

R&D Efforts and Needs—

The process research program at JPL appears well-gearred to the needs of the process. Many of the basic parameters of the process appear to be well established. The present contractual requirement to test sulfur removal on nine additional coals, including two sub-bituminous coals and one lignite is highly desirable. This work will determine the applicability to the process to the various coal reserves in the U.S. There are, however, a few important areas of research and development which require further investigation. These are:

- The trace metals removed from the coal will probably be difficult to concentrate and dispose of. At present, it appears that these elements will end up in the concentrated sulfuric acid, and their presence may render the acid unmarketable. Research effort is therefore necessary to determine the extent of acid contamination and its marketability. Effort may be required to find means for removing the trace element from the acid or from its' precursor, the coal filtrate. As a last resort it may be necessary to lime the acid by-product and produce a gypsum which would be dewatered

and land-disposed. This would result in additional costs not presently considered.

- A cursory investigation of the "Kel-Chlor" process shows that product chlorine can be furnished in gaseous form, at various pressures, or as a liquid under pressure. If the chlorine is furnished as a gas without prior pressurization and distillation it will be less costly but will contain 7-8%, by volume, of oxygen. Feeding an impure chlorine as this to the chlorinolysis reactor may have unpredictable consequences. Therefore, the technical and economic alternatives concerning the purity of the recycled chlorine, will probably require experimental verification.
- Most of the JPL effort has been based on finely divided coal (minus 100 to minus 200 mesh feed). If the process is nearly as efficient on a larger coal particles, say minus 14 or minus 28 mesh, additional benefits would be derived from decreased coal preparation costs. Kinetic studies on coals of various size consists are desirable therefore to establish minimum coal preparation requirements.
- Due to the highly corrosive nature of hydrochloric acid, sulfuric acid, chlorine, hydrogen chloride, and various mixtures of these reagents, it is strongly recommended that both engineering and laboratory effort be directed toward selection of materials of construction for equipment used in each process step, and for the connecting piping, controls and instrumentation.

Process Economics

Capital and annual operating costs have been developed for the JPL chemical coal cleaning process, and are presented in Tables 47, 48 and 49. Both sets of cost data are based, in part, on cost data furnished by JPL. However, changes have been made in the JPL data to produce cost data which is comparable to that produced for other chemical coal cleaning processes studied. Changes made are as follows

- At the present time JPL is using minus 100 to minus 200 mesh coal as feed to their process. Accordingly, capital estimates for receiving ROM coal, crushing, grinding and handling minus 200 mesh coal have been used. In time, if a coal feed stock of plus 100 mesh, or minus 14 mesh, can be efficiently utilized by the process, these costs can be greatly decreased.
- The JPL capital investment costs do not include product coal compaction costs. Some additional capital and operating costs have therefore been added.
- JPL depreciates their plant over a period of 15 years, but does not include the cost of capital. A capital recovery factor based on a 20-year plant life, paying 10% for the cost for the capital has been used.
- The capital and particularly the operating costs for a "Kel-Chlor" plant are greater than the costs used by JPL. Accordingly, recent data from Pullman-Kellogg has been used.³²
- Costs associated with the use of superheated steam to dechlorinate and dry coal have been underestimated by JPL since the steam usage is not known. However, since the TRW process uses almost 500 kg of superheated steam per metric ton (1,000 lbs/ton) of coal to sublimate free elemental sulfur from its product, the same value of superheated steam consumption for the JPL process has been used. Assuming that product coal will be used to generate this steam, and further assuming an 85% combustion efficiency and a 6,800 kg cal/kg (12,300 BTU/lb) coal, it is estimated that 470 metric ton (520 tons) per day of product will be consumed internally for this purpose. Further, assuming a 2% loss in coal heating value due to processing, the thermal (BTU) efficiency of the process is approximately 91%.

TABLE 47. SUMMARY OF ECONOMICS FOR THE JPL CHEMICAL
COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Grass roots plant installation

91% weight yield, 91% heating value recovery

Installed Capital Cost: \$103,200,000

Annual Operating Costs

on Clean Coal Basis: \$44,410,000 process cost, excluding coal cost
\$110,410,000 process cost, including coal cost*
\$20.38/metric ton (\$18.49/ton), excluding coal cost
\$50.67/metric ton (\$45.97/ton), including coal cost*
\$2.97/10⁶ kg cal (\$0.75/10⁶ BTU), excluding coal cost
\$7.40/10⁶ kg cal (\$1.86/10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 48. INSTALLED CAPITAL COST ESTIMATE FOR THE JPL
CHEMICAL COAL CLEANING PROCESS

	<u>\$ 1977</u>
Coal handling and preparation*	\$ 16,600,000
Desulfurization process costs ^Δ	12,290,000
Compacting and product handling*	5,120,000
Building and miscellaneous ^Δ	—
Utilities (off-sites) ^Δ	
Kel-Chlor [†]	45,375,000
Site development and general ^Δ	
Subtotal	<u>\$ 79,385,000</u>
Engineering design @ 10%	7,938,000
Contingency @ 20%	<u>15,877,000</u>
Total Installed Plant Capital (TPC)	\$103,200,000

* Versar estimate, installed cost

Δ JPL grass roots estimate including site development

† Battery limits plus grass roots requirements not otherwise furnished;
based on Pullman-Kellogg estimate

TABLE 49. ESTIMATED ANNUAL OPERATING COSTS FOR THE JPL
CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)*	\$ 7,274,000
Taxes @ 2% TPC ^Δ	884,000
Insurance @ 1% TPC ^Δ	442,000
Labor (direct and indirect)	2,200,000
General and administrative @ 1.5% TPC	1,500,000
Maintenance and supplies @ 5% TPC ^Δ	2,210,000
Utilities:	
Electric power	1,200,000
Water [†]	100,000
Steam [†]	—
Chemicals:	
Chlorine ^α	23,000,000
Miscellaneous chemicals	1,300,000
Binder	4,300,000
Waste Disposal	—
Total Annual Processing Cost	\$ 44,410,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$110,410,000

* 30% of Kel-Chlor facilities; 100% others.

^Δ Excluding Kel-Chlor.

[†] 470 metric ton/day product coal will be consumed internally to generate steam.

^α Kel-Chlor process, including chemicals, depreciation, utilities, labor, maintenance and interest on construction costs of 70% of capital.

INSTITUTE OF GAS TECHNOLOGY (IGT) CHEMICAL COAL CLEANING PROCESS

The IGT flash desulfurization process is based upon chemical and thermal treatment of coal. In this process, sulfur is removed from the coal by a hydrogen treatment under the proper conditions of temperature, heat-up rate, residence time, coal size, hydrogen partial pressure and treatment gas composition.

An oxidative pretreatment is included in this system to prevent caking and also to increase the sulfur removal in the subsequent hydrotreating step. Both pyritic and organic sulfur are removed by the combination of these treatments. The treated product is a solid fuel (possibly char) which presumably may be burned without a need for flue gas scrubbing.

This report contains a conceptualized process design and process economics based upon IGT data. Subsequent to our cut-off date for data input, IGT has developed its own conceptualized process design that includes the effects of many factors derived from IGT's general background in coal conversion. The IGT-developed process efficiencies and costs are significantly better than those reported here, based upon the earlier IGT report specific to this program. The following discussion, therefore, does not include IGT's latest thinking on the process design; it should be regarded as preliminary and subject to significant process efficiency improvements and downward product cost modification.

Process Description

The process employs essentially atmospheric pressure and high temperatures [about 400°C (750°F) for pretreatment and 800°C (1500°F) for hydrosulfurization] to enhance the desulfurization of the coals. These high temperatures cause considerable coal loss due to oxidation, hydrocarbon volatilization and coal gasification, with subsequent loss of heating value. Batch reactor tests have indicated an average product recovery potential of 60 weight percent based on the feed.

Experiments have been conducted with several coals in both laboratory and bench scale batch hardware to test IGT concepts and to determine the pretreatment and hydrodesulfurization operating conditions. Adequate experimental data on heat and material balances are not yet available to conceptualize a process design. It is, however, anticipated that the process will employ the following equipment or processing steps:

- Fluidized bed reactors will be used for both pretreatment and hydrodesulfurization stages;
- Air will be used as the source of oxygen;
- Off-gases from the hydrodesulfurization, provided they contain hydrogen partial pressure, would be compressed and recycled to the hydrogenation reactor to provide the necessary hydrogen for desulfurization of coal.
- Hydrogen make-up may be necessary to maintain hydrogen partial pressure.
- The exothermic pretreatment reaction would provide a portion of the heat necessary for the endothermic hydrodesulfurization reactions.
- The sulfide and sulfate sulfur would be removed from the hydrodesulfurized product by either chemical or mechanical means. This step will be necessary when the coal char product from the processing of certain coals contains residual sulfur levels exceeding the allowable limits.
- The hydrogen sulfide/carbon dioxide gases recovered from the hydrodesulfurizer off-gas will be treated in a Claus plant to produce elemental sulfur.
- Purification of the off-gas from the hydrodesulfurizer system will be necessary prior to recycle.

- Off-gas clean-up from the pretreater will be necessary prior to venting the gases to the atmosphere.

Versar has provided a suggested process flow sheet which integrates the IGT concepts and is shown in Figure 22. This flow sheet has been provided to permit the development of process economics on a consistent basis with other processes.

Status of the Process

The IGT process is in an early stage of development. An extensive bench scale and pilot level technical effort is needed before an integrated process design is conceptualized. The program, sponsored by EPA, is now directed toward testing in a 25 cm (10-inch) continuous fluidized-bed unit, which is sized for coal feeds of 10 to 45 kilograms (25 to 100 pounds) per hour.

Two pretreatment runs of about seven hours each have been made in this 25 cm (10-inch) unit. A beneficiated Illinois No. 6 coal, which was crushed to minus 14 mesh and contained 2.43 weight percent of total sulfur, was used as feed. The objectives of these runs were to test the operating conditions over a sustained period of time and to produce pretreated material for subsequent hydrodesulfurization evaluations. The pretreatment runs have been successful and they have confirmed most of the results of corresponding batch tests. These runs indicated that a temperature of 400°C (750°F), a residence time of 30 minutes, an actual gas velocity of 0.3 meter (one foot) per second in the bed and 0.616 cubic meter of oxygen per kilogram [one standard cubic foot (SCF) per pound] of coal is adequate to pretreat the coal when the unit is fed at a rate of about 23 kilograms (50 pounds) per hour. However, material and heat balance information generated on one of these runs, contradicts conclusions derived from the batch runs. The analyses of data indicated very low quantities of light hydrocarbon in the off-gases [90 kg cal/cu m (10 BTU/SCF)] and a very high solids recovery around the pretreatment unit (97.7 wt%). Thus only 2.3 wt % of the coal was consumed in off-gases and water as compared to the expected 8 to 12 percent. Information from a single run is not adequate

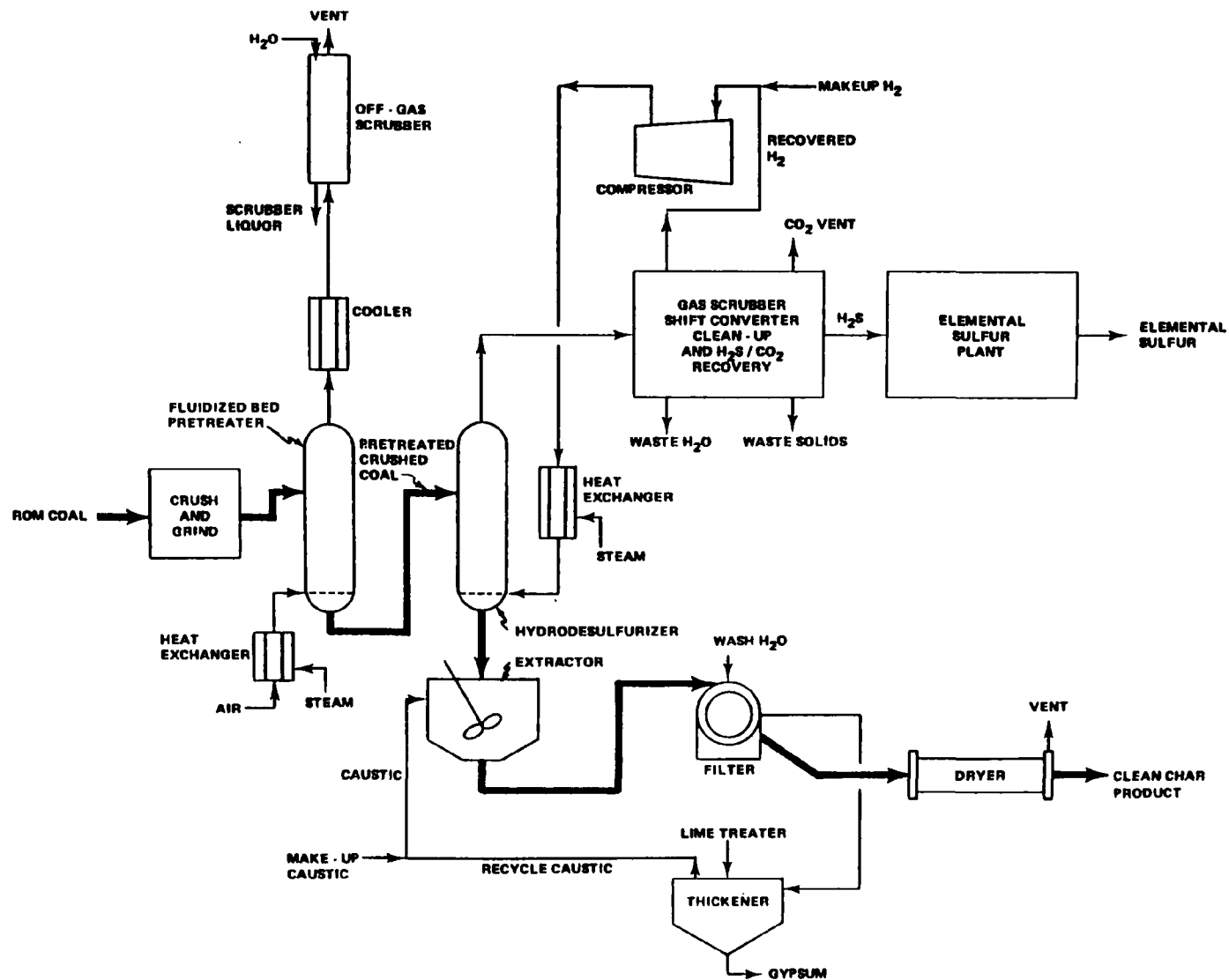


FIGURE 22 IGT PROCESS FLOW SHEET

to draw definitive conclusions, however, if these data are confirmed in the Pilot Demonstration Unit (PDU), then no excess heat would be available from the pretreatment stage for either steam generation or on-site consumption.

The data from the larger unit will be used to establish the necessary energy and material balance information for the design of an integrated system and for an accurate economic evaluation of the process.

Supportive runs are being continued in the batch reactor to determine the effects of nitrogen, carbon monoxide, water vapor and hydrogen sulfide concentrations in the treat gas on the hydrodesulfurization operation. Additionally, crushing tests on a run-of-mine, Illinois No. 6 coal are being conducted to determine the crusher conditions to minimize fines in coal preparation and to define the coal preparation requirements for the process.

IGT estimates that this process could be ready for commercialization in four or five years after the successful operation of a pilot demonstration unit.

Technical Evaluation of the Process

This process is currently at the bench scale level, thus, a definitive assessment of its industrial potential is not possible at this time. However, available information is summarized in the following subsections.

Potential for Sulfur Removal—

Laboratory and bench scale experiments conducted thus far indicate that the IGT process can remove 83 to 89 percent of the total sulfur from four bituminous feed coals. The process removes both pyritic and organic sulfur. In most cases, enough sulfur is removed so that the treated product could be burned in conformance with current EPA new source performance standards for SO₂ emissions.

A preliminary evaluation of the desulfurization potential of four selected bituminous coals was conducted in a laboratory device (thermobalance) with

2 to 6 gram coal samples. Pyritic, organic and total sulfur removal rates obtained from these investigations are reported in Table 50.³³ Detailed laboratory information reported by IGT is included in Appendix VII. Samples for the above thermobalance tests were +40 mesh pretreated coal. The feed was placed in the sample basket and then lowered into the treating zone. A heating rate of 2.8°C (5°F) per minute was used up to the terminal temperature of 815°C (1500°F). Soaking time at the terminal temperature was 30 minutes for each test.

Table 50 indicates that for the Western Kentucky No. 9 coal, in addition to 98 percent pyritic sulfur removal, 88 percent of organic sulfur removal was also achieved. Sufficient total sulfur removal was realized in this test so that SO₂ emissions from combustion of the treated product would be 0.76 kg/10⁶ kg cal (0.42 lb/10⁶ BTU).

The sulfur reduction obtained for the Pittsburgh seam coal from the West Virginia mine was 98 percent pyritic and 83 percent organic sulfur. The reduction in total sulfur content, accounting for sulfide/sulfate compounds, was 83 percent, with sufficient sulfur removed to comply with current EPA new source performance standard of 2.16 kg/10⁶ kg cal (1.2 lb/10⁶ BTU) of SO₂.

Results for the Pittsburgh seam coal from the Pennsylvania mine indicate that in addition to all of the pyritic sulfur, 77 percent of the organic sulfur was also removed. This coal having a lower initial total sulfur and relatively low initial organic sulfur content also yielded a product with acceptable SO₂ emission value.

The sulfur reduction obtained for a beneficiated Illinois No. 6 coal was 98 percent pyritic and 82 percent organic sulfur. This sulfur reduction was such that SO₂ emissions from combustion of the treated product would be below the current new source SO₂ standards.

The results of all thermobalance tests conducted with the above mentioned four feeds are superimposed in Figure 23.³⁴ These experiments were conducted using varying heat-up rates of 2.8 to 11°C (5 to 20°F) per minute up to temperatures of 538° to 815°C (1000° to 1500°F) and soaking times at the terminal temperature from a few minutes to 5.5 hours. The plot indicates that all coals behaved similarly and that higher temperatures (about 815°C) are needed to achieve adequate hydrodesulfurization.

TABLE 50. IGT PROCESS THERMOBALANCE SULFUR REMOVAL RESULTS

<u>Source of Coal</u>	<u>Raw Coal Characteristics</u>		<u>Sulfur Removal Efficiency, Weight Percent</u>		
	<u>Feed Type</u>	<u>Sulfur* Content wt.% of Feed (dry basis)</u>	<u>Pyritic^Δ</u>	<u>Organic[†]</u>	<u>Total</u>
Western Ky #9	ROM	3.03	97.8	88.5	89.4
Pittsburgh Seam From W. Virginia	Highly Caking	2.41	98.4	83.1	83.0
Pittsburgh Seam From Pa. Mine	High Ash Content	1.01	100.0	77.1	78.1
Illinois #6	Beneficiated	2.28	98.0	82.0	87.7

NOTES:

Experimental Conditions Were: At 1500°F terminal temperature, 5°F heat-up rates and 30 mins. soaking time.

* Sulfur content of +40 mesh material.

^Δ The pyritic sulfur removal during pretreatment ranges from 38% to 51%.

[†] The organic sulfur removal during pretreatment ranges from 0% to 10%.

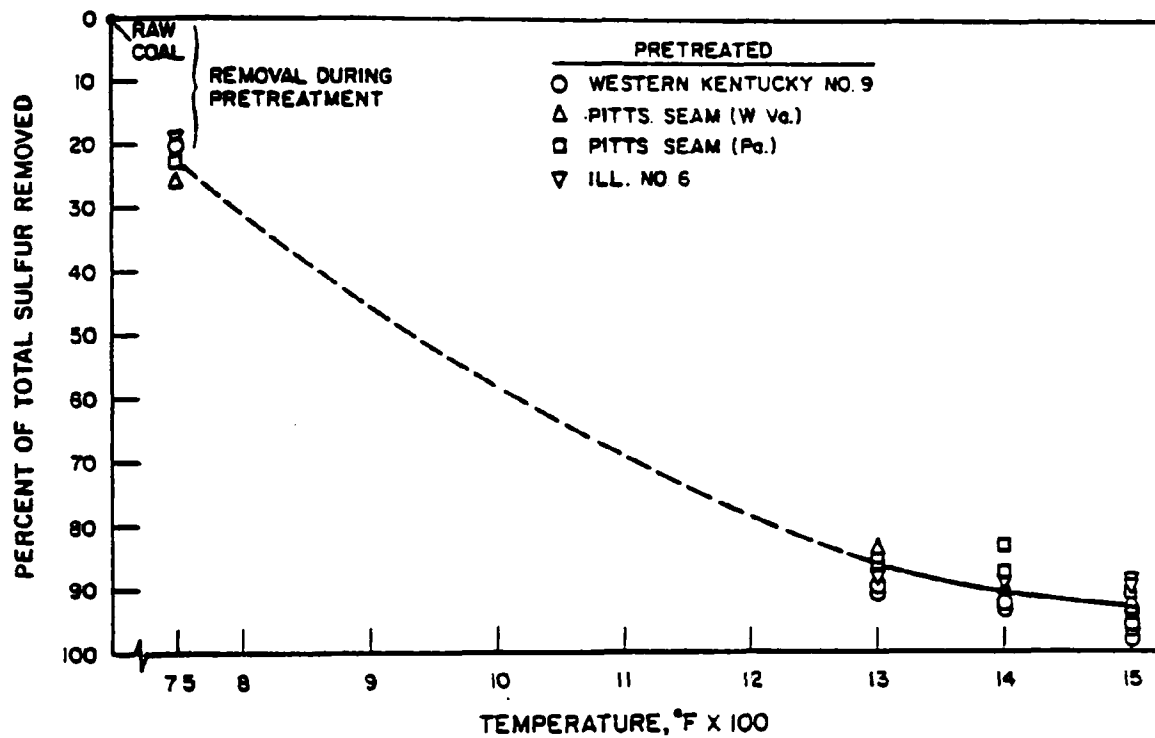


FIGURE 23 IGT PROCESS: PERCENT SULFUR REMOVAL VS. TEMPERATURE

Batch reactor evaluations have been made using the Western Kentucky No.9 and Illinois No. 6 coals to verify the thermobalance test results. This reactor operates as a fluidized bed, similar to the mode of operation anticipated for the commercial plant. The unit is capable of handling larger samples (75 to 150 grams); therefore, a more complete characterization of the treated product was achieved. Table 51⁸⁴ presents results of two batch reactor tests conducted at conditions identical to the thermobalance experiments presented in Table 50. Results are in good agreement with the thermobalance investigations. The treated coal from these two evaluations would produce SO₂ emissions under 1.8 kg/10⁶ kg cal (1.0 lb/10⁶ BTU). More detailed information reported by IGT on these two runs is included in Appendix VII.

A series of thermobalance tests has also been performed on the pretreated Western Kentucky No. 9 coal using rapid heat-up rates to determine the effect of the holding time on sulfur removal. The final temperature was 816°C (1500° F) with residence times of 15, 30, 60 and 90 minutes. The results are presented in Figure 24. These tests indicate decreasing total sulfur with increased soaking time. Figure 24 also indicates that the terminal temperature is primarily controlling the pyritic sulfur reduction, while the organic sulfur removal is dependent upon the soaking time. Other similar tests have indicated that the pyritic sulfur is primarily removed by hydrogenation, while the organic sulfur removal depends on the heating rate. At rapid heat-up conditions, the organic sulfur removal is improved.

Other supportive runs with a mixture of gases have been made to provide background information necessary for the design and operation of an integrated system. Batch reactor runs using mixtures of hydrogen (H₂) carbon monoxide (CO) and water vapor (H₂O) have indicated that CO levels in hydrogen up to 37.5 weight percent are not detrimental to the removal of sulfur. Similar runs with a mixture of H₂ and Nitrogen (N₂) gases have indicated that the H₂ concentration in the treat gas must be at least 50 weight percent to bring the organic sulfur level down considerably. Batch reactor runs are planned with a mixture of one percent hydrogen sulfide (H₂S) in hydrogen to determine the effect of H₂S concentration on the desulfurization operation.

TABLE 51. IGT PROCESS TYPICAL BATCH REACTOR RUNS WITH SPECIFIED FEEDSTOCKS

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Run No.	<u>RB-76-3</u>			<u>BR-76-34</u>		
Coal Type	<u>Run-of Mine W. Ky. #9</u>			<u>Washed Ill. #6</u>		
Sample	<u>Pretreated</u>			<u>Pretreated</u>		
	<u>Feed</u>	<u>Coal</u>	<u>Product</u>	<u>Feed</u>	<u>Coal</u>	<u>Product</u>
Terminal Temperature, °F		750	1,500		750	1,500
Heat-Up Rate, °F/min.			5			5
Soaking Time, min.		30	30		30	30
Sulfur, wt.%						
Sulfide	0.02	0.04	0.12	0.01	0.01	0.05
Sulfate	0.60	0.10	0.00	0.13	0.04	0.05
Pyritic	1.06	1.54	0.02	0.84	0.65	0.03
Organic	<u>1.82</u>	<u>1.38</u>	<u>0.37</u>	<u>1.50</u>	<u>1.52</u>	<u>0.47</u>
Total	3.50*	3.06*	0.51	2.48*	2.22*	0.60
Total Sulfur Removal, wt.%	-	20.6	90.9	-	19.1	84.7
Yield, wt. % (From Feed Coal)		90.8%	62.2%		90.4%	62.9%
Heating Value, BTU/lb.	12,454	11,809	11,967	13,022	12,915	12,793
Sulfur Emission, lb/10 ⁶ BTU	5.62	5.18	0.85	3.76	3.44	0.94

*Calculated for +40 Mesh fraction.

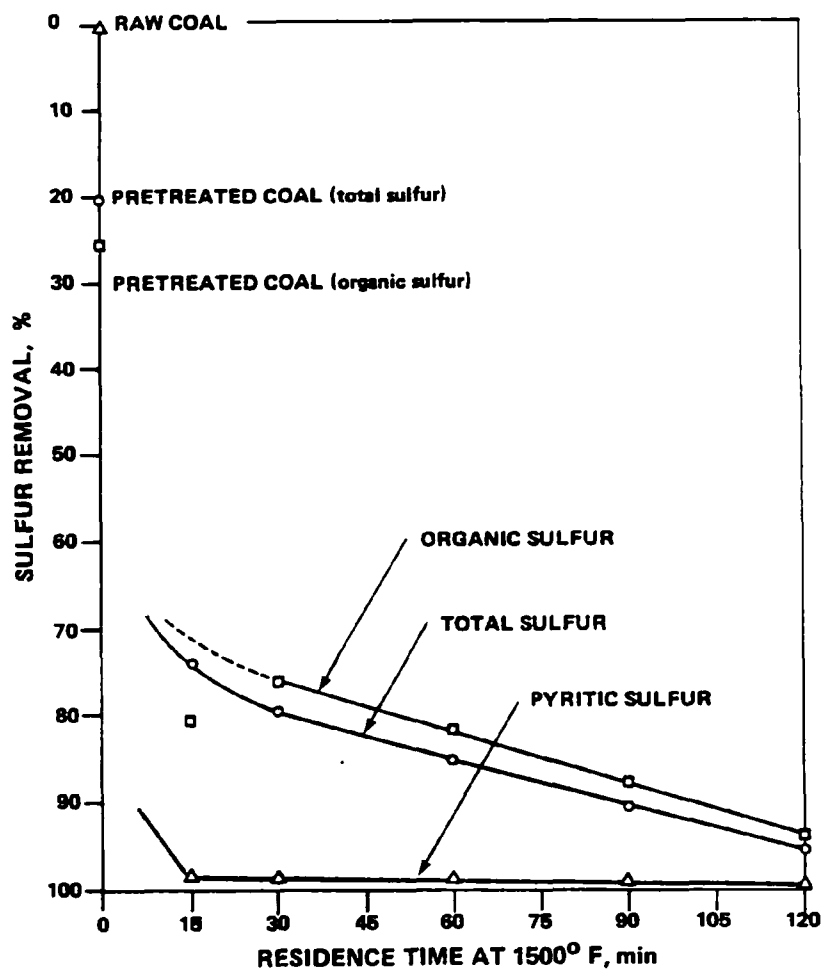


FIGURE 24 IGT PROCESS: EFFECT OF HOLDING TIME ON SULFUR REMOVAL

A supply of run-of-mine Illinois No. 6 coal has been obtained. This coal is considerably different from the previous beneficiated Illinois No. 6 feed. It is much higher in moisture, ash and sulfur content and the fixed carbon is lower. With its low heating value and high sulfur content this coal would be a good feed candidate to demonstrate the desulfurization potential of the process in the 25.4 cm (10-inch) unit.

Sulfur By-Products—

In the IGT process the sulfur components of the coal are converted primarily into sulfur oxides and hydrogen sulfides. Gaseous sulfur dioxide and sulfur trioxide generated will most probably be removed in an off-gas scrubber system. Sulfur removed as gaseous hydrogen sulfide will be subsequently converted to elemental sulfur in an ancillary process.

IGT has suggested the removal of any remaining sulfide and sulfate from the treated product by chemical or mechanical means. This after treatment could also precipitate other sulfur compounds. For example if caustic is used as the extracting medium, its regeneration would result in the precipitation of gypsum (CaSO_4).

Benefit Analysis—

The main benefit associated with the IGT process is the removal of both pyritic and organic sulfur from coals to an extent permitting the burning of the treated product in conformance with EPA's current NSPS for SO_2 emissions. If this claim is verified in an integrated pilot system, this process could represent a potential major technology for the control of sulfur oxide emissions from combustion of coals, primarily from coals which contain large quantities of organic sulfur.

The nitrogen content of the product fuel from the IGT hydrodesulfurizer is about half of the content of that of the raw coal based upon results of batch reactor tests. A reduction in the nitrogen content of the treated fuel may help to reduce the NO_x emissions during combustions. No analysis has been yet conducted to determine the trace metals content of the treated product. However, it is anticipated that the IGT high temperature desulfurization process, by incorporating a gas purification system, might provide a method for the control of mercury and other trace metals.

The process, however, has a low net yield (approximately 60 percent), with a heating value of about 5 percent lower than that of the feed stock. The net energy yield is calculated to be 57 percent. The net energy yield of the process may be even lower than that estimated above due to the extreme operating temperatures [816°C (1500°F)] of the hydrodesulfurization unit. Additionally, the IGT process changes the matrix of the coal. The treated product is a solid fuel (possibly a char) with its volatile matter reduced significantly. Thus modified combustion equipment may be required for the utilization of the treated product.

Environmental Aspects—

It would be unrealistic to assume that there will not be some unavoidable adverse environmental effects from this process.

Gaseous emissions from the integrated system will primarily include water vapor, carbon dioxide, and nitrogen, along with quantities of sulfur and nitrogen oxides. There will also be some solid by-products such as sulfur and gypsum which may be sold or disposed of. Pollution controls will be needed to ensure that the process meets emissions standards in a practical manner with all relevant U.S. coals.

Since the IGT process is not developed to a level to permit the discussion of specific details of all its environmental emissions, discussed below are only major anticipated pollutants and some general means for their control.

Sulfur dioxide—Since coal pretreatment is used to avoid the caking of coals in the subsequent hydrodesulfurizer, substantial sulfur dioxide evolution will take place. The effluent stack gases will carry as much as 25 to 30 percent of the coal's sulfur. This will amount to over 181 metric tons (200 tons) per day sulfur dioxide emission when charging high-sulfur coal (5-6 wt% total sulfur in coal) at a feed rate of 7200 metric tons (8000 tons) per day. The coal pretreating temperature is anticipated to be 400°C (750°F). The off-gas from such pretreat operations will need to be scrubbed. The investment for SO₂ removal from a 180 metric tons/day scrubber could be over 8 million dollars.

Process heat needs, not recoverable from the pretreatment system, can be supplied by burning a portion of the product char. If the treated material is

not adequately desulfurized, stack gas scrubbing for SO_2 removal will be required. An alternative source is the production of low heating value (low-BTU) gas from the treated product by one of the several commercial processes. Such gas products can be then used for firing process heaters. However, investment for this alternative route may be much greater.

Another alternative which may be adaptable for firing the product chars containing high residual sulfur is a fluidized-bed combustor using lime. In this case, lime addition may adsorb the residual sulfur and avoid the need for stack-gas scrubbing.

Hydrogen sulfide—An acid-gas scrubbing process will be most likely used to remove carbon dioxide (CO_2) and hydrogen sulfide (H_2S) from the hydrodesulfurizer effluent gas system. The H_2S removed in this step will represent the major sulfur content of the feed coal. It is anticipated that IGT will select the Claus process approach to recover elemental sulfur from this stream. If so, stack-gas scrubbing will also be required for the clean-up of the Claus plant tail gas.

Trace metals—The mercury in the coal tends to be concentrated in the pyrite, although a substantial fraction may be organically bonded. The pre-treatment step of the IGT process at 400°C (750°F) may release as much as 1/3 of the coal's mercury into the pretreater flue gas stream. Another mercury emission source would be the hydrodesulfurizer. However, this mercury can be removed within the gas purification system, primarily in an activated carbon tower. This mercury will be retained on the active carbon. Disposal of the spent carbon by proper burial should protect against any contamination by the mercury. Thus, the IGT process may also provide a route for control of the mercury that would be mainly emitted to the atmosphere when burning the same coal without treatment.

No beryllium or vanadium emissions should occur from the IGT reaction system; these trace metals are expected to be found in the treated product. Some volatilization of cadmium compounds can take place; but any cadmium volatilized from the hydrodesulfurization system may be picked up by the gas purification system.

Particulates— In the vicinity of the plant, coal handling, crushing, grinding and conveying operations may need to be enclosed to provide dust control. These controls should help to meet plant emission restrictions in particulates. Where ground coal is pneumatically conveyed, use of bag house or several cyclones for solid recovery may be adequate before venting the gases to the atmosphere.

Waste waters—Since this process is in an early stage of development, very little is known regarding process waste waters. However, the potential air pollutants in the coal are normally converted to water soluble salts and thus the process waters may contain high concentrations of dissolved solids, hydrogen sulfide, ammonia, phenol, benzene and dissolved oils. Concentration of these contaminants will be dependent on the quantity of the discharge waters and on the design of the various scrubbers selected for the integrated system.

Problem Areas—

It is as yet premature to define potential problem areas for this process precisely, since the IGT concepts are not at a developmental stage where an integrated system may be conceptualized. The net energy recovery potential of the system and the change in the coal matrix by the process have been identified as possibly severe problems for the IGT process.

R&D Efforts and Needs—

Specific immediate research efforts and needs for this process are:

- Prove the concept on larger continuous equipment to assess the process viability and establish heat and material balance information.
- Establish the process engineering of an integrated system to estimate the process economics.
- Design, assemble and operate a small pilot plant incorporating and integrating the major segments of process.
- Study physical, chemical and combustion characteristics of the treated product in order to define its combustion behavior and to evaluate the pollutant emissions from the burning of the treated material.

Conclusions derived from the above recommended studies will indicate whether this process warrants further optimization and demonstration studies.

Process Economics

Heat and material balance information have not been established for the IGT process; therefore, economic factors have not been determined by IGT. However, a preliminary rough economic evaluation was developed by Versar for this process using: (1) Figure 22 as the accepted flow sheet for this process and (2) the economics developed for the Lurgi process as the basis for the estimate.

It is Versar's contention that since the IGT chemical coal cleaning process is a gasification method for removing sulfur it will use many of the unit operations employed by the Lurgi's system. Adjustment of Lurgi process economics for known differences will yield a rough estimate for the IGT process.

A summary of economics for the IGT process is given in Table 52. It can be seen from Table 52 that the sulfur removal cost is very high due to low yield and low BTU recovery. Details on the capital costs are given in Table 53. The total differences between the Lurgi and the IGT process result from (1) lower reactor cost for IGT due to lower operating pressures; (2) lower gas treatment and purification cost due to lower gas volume and lower operating pressures; (3) elimination of the methanation and oxygen manufacturing operations and (4) addition of extraction, filtration, thickening, product drying and compacting operations. The estimated operating costs of the IGT process are presented in Table 54.

TABLE 52. SUMMARY OF ECONOMICS FOR THE IGT CHEMICAL
COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Gross roots plant installation

60% weight yield, 57% heating value recovery

Installed Capital Cost: \$134,620,000

Annual Operating Costs

on Clean Coal Basis: \$38,277,000 process cost, excluding coal cost
 \$104,277,000 process cost, including coal cost*
 \$26.64/metric ton (\$24.16/ton), excluding coal cost
 \$72.57/metric ton (\$65.83/ton), including coal cost*
 \$4.09/10⁶ kg cal (\$1.03/10⁶ BTU), excluding coal cost
 \$11.16/10⁶ kg cal (\$2.81/10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 53. INSTALLED CHEMICAL COST ESTIMATE FOR THE IGT
CHEMICAL COAL CLEANING PROCESS

	<u>\$ 1977*</u>
Coal handling and preparation	\$ 7,020,000
Desulfurization process costs	
Gasification	10,800,000
Gas cooling	1,980,000
Shift conversion	4,140,000
Gas purification	16,470,000
Sulfur recovery ^Δ	4,950,000
Compression	1,800,000
Extraction	720,000
Water pollution control	7,900,000
Compacting and product handling [†]	10,890,000
Building and miscellaneous ^Σ	700,000
Utilities (off-sites) ^α	29,000,000
Site development and general ^φ	<u>7,200,000</u>
Subtotal	\$103,570,000
Engineering design @ 10%	10,350,000
Contingency @ 20%	<u>20,700,000</u>
Total Installed Plant Capital (TPC)	\$134,620,000

* Lurgi process cost estimates published in April 1973 were used as basis. These estimates were prepared by the Synthetic Gas-Coal Task Force appointed by the National Gas Survey of the Federal Power Commission. Cost adjustment to a 7,200 metric ton (8,000 ton) per day plant was made using an exponential factor of 0.6. Cost estimates were further adjusted to the first quarter 1977 prices using the Marshall and Stevens Cost Indices

Δ Data supplied by General Electric

† Includes filtration, thickening, drying and compacting

Σ Includes administrative building, maintenance shop, stockrooms and stores.

α Includes steam plant, in-plant electric power, distribution cooling tower, boiler feed water treatment, instrument and plant air, fuel gas distribution, communications and water pollution control.

φ Includes site preparation, rail facilities, fire protection, safety system, chemical and by-product storage.

TABLE 54. ESTIMATED ANNUAL OPERATING COSTS FOR THE IGT
CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	15,800,000
Taxes @ 2% TPC	2,700,000
Insurance @ 1% TPC	1,350,000
Labor (direct and indirect)	3,075,000
General and administrative @ 1.5% TPC	2,020,000
Maintenance and supplies @ 5% TPC	6,732,000
Utilities:	3,300,000
Electric power	
Water	
Steam & fuel*	—
Chemicals and Catalyst	3,300,000
Waste Disposal	—
Total Annual Processing Cost	\$ 38,277,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$104,277,000

* It has been assumed that 9.07 metric ton/hr (10 tons/hr) product coal will be used to generate steam for in-process needs.

KVB CHEMICAL COAL CLEANING PROCESS

The KVB coal desulfurization process is based upon selective oxidation of the sulfur constituents of the coal. In this process, dry coarsely ground coal (+28 mesh) is heated in the presence of nitrogen oxide gases for the removal of a portion of the coal sulfur as gaseous sulfur dioxide (SO_2). The remaining reacted sulfur in the coal is claimed to be in the form of inorganic sulfates, sulfites or is included in an organic radical. These non-gaseous sulfur compounds are removed from the pretreated coal by subsequent washing with water or heated caustic solution followed by water wash.

The active oxidizing agent is believed to be NO_2 . The process, however, uses a gas mixture containing oxygen (0.5 to 20 percent O_2 by volume), nitrogen monoxide (0.25 to 10 percent NO by volume), nitrogen dioxide (0.25 to 10 percent NO_2 by volume) and nitrogen (N_2) the remainder.

The process can be operated either on a batch or continuous basis as desired. There are no data available, as yet, to indicate which system is more economical. For a continuous operation, the reaction may be carried out at 120°C (250°F) 2.4 atm (35 psia) for 1/2 to 1 hour period. The mechanism of oxidation is still unknown. Details of process chemistry, as explained by KVB, are given below.³⁵

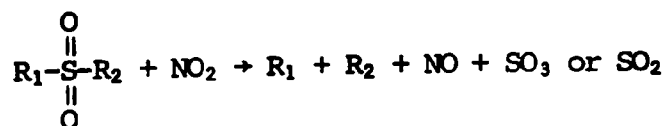
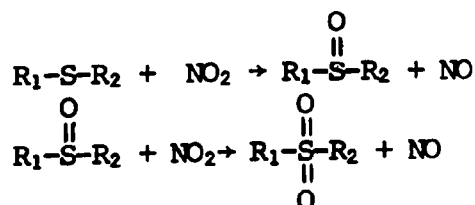
Oxidant generation



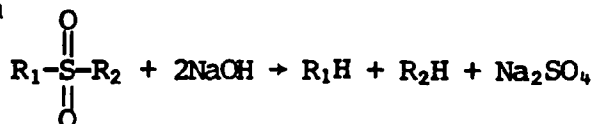
Pyrite oxidation



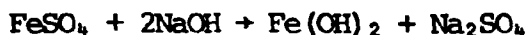
Organic sulfur oxidation reactions



Extraction of sulfur from
an organic radical



Removal of iron sulfates
in the extractor



Caustic regeneration



Process Description

Laboratory experiments have been conducted with several coals on 50 gram samples in a 2.54 centimeter (one-inch) diameter batch reactor to test the sulfur removal potential of the process. The process has been conceptualized both by KVB³⁶ and Bechtel⁸. The KVB design incorporates a somewhat more optimistic water and caustic extraction operation than the flow scheme suggested by Bechtel. In this section, the flow diagram developed by Bechtel will be used since it incorporates standard processing equipment in conceptualizing the process.

A simplified flow diagram of the process is shown in Figure 25⁸. Dry coal from the preparation section is pneumatically conveyed to a gas/solid cyclone where it is separated from its conveying gas (nitrogen). Then it is gravity fed into a fluidized bed reactor. The reactant gas is introduced through the bottom of the reactor through a distributor. The reaction gases leave the reactor, passing through a two-stage cyclone separator which removes the fine coal particles from the gas.

The treated coal from the reactor is next reacted with caustic solution to remove additional sulfur (organic sulfur) and also convert the ferrous sulfate to ferrous hydroxide and soluble sodium sulfate. The coal slurry from the extractor is filtered and water washed on the filter. The product coal is then dried prior to compacting. The process also incorporates treatment of the various effluents from the system.

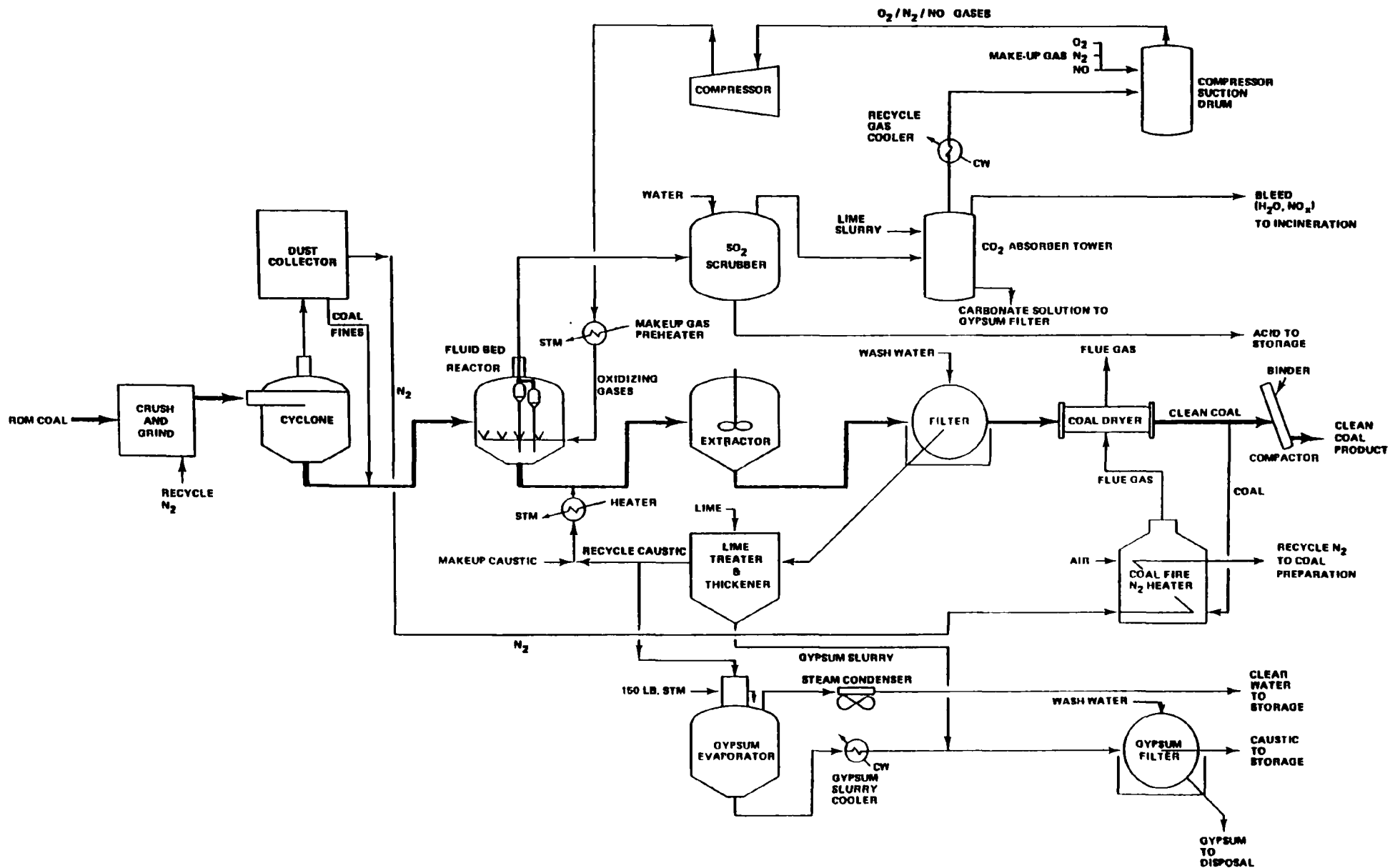


FIGURE 25 KVB PROCESS FLOW DIAGRAM

Nitrogen, (the transporting gas) from the cyclone is passed through a dust collector for the recovery of fine coal particles and is then discharged via a blower into a coal-fired heater prior to recycling this gas to the coal preparation and conveying section.

Off-gas from the reactor is scrubbed with water to remove sulfur oxides and nitrogen oxide gases. The acid product from the scrubber containing sulfurous, sulfuric and also nitric acid is cooled prior to storage. The treated gas from the water scrubber is subsequently reacted with calcium hydroxide to remove carbon dioxide as calcium carbonate sludge. The purified gas from the CO₂ remover is cooled to condense water vapor. A fraction of the gas leaving the purifier is vented to prevent a buildup of inert gas in the gas stream. By venting a portion of the gas and providing makeup gas, the required gas proportion can be maintained. The recycle gas is then combined with makeup NO₂ and O₂ to form the treat-gas. The treat-gas is compressed and recycled to the reactor.

The filtrate from the coal filter is treated with lime to regenerate caustic and form gypsum. The sludge from the lime treatment tank is concentrated in a thickener. The underflow of the thickener containing a large fraction of the gypsum is filtered to recover the caustic solution. The thickener overflow is divided into two streams. One portion is recycled to the extractor and the other is sent to an evaporator for further removal of gypsum in order to prevent gypsum buildup in the system. The steam generated in the evaporator is condensed and used as wash water for the filter cake. The gypsum slurry is cooled and set to the gypsum filter. Gypsum constitutes the solid waste from this process.

Status of the Process

The process has been tested batchwise in the laboratory, using 50 gram coal samples. KVB owns all rights to the process as of April 1977 and has funded all the work thus far. U.S. Patent No. 3,909,211 was issued on September 30, 1975³⁷ and the filing of foreign patents in major coal producing countries is in progress.

The KVB laboratory test work on their chemical coal cleaning process is presently inactive. Plans are to develop and commercially license the process to coal producers and users. Funding is being actively sought at this time to speed up the developmental schedule in view of the current energy shortage.

Technical Evaluation of the Process

This process is in its early stages of development and thus, it is difficult to make an accurate assessment of its industrial potential. However, depending on the amount of desulfurization required, the extraction and washing steps may or may not be required. It should be mentioned that in cases where dry oxidation only could remove sufficient sulfur to meet the sulfur dioxide emission standards, this technology could provide a very simple and inexpensive system. Thus, there may be a potential for this process for application to some coals, primarily metallurgical grade coals, where partial removal of sulfur could be very beneficial.

Potential for Sulfur Removal—

Laboratory experiments conducted on 50 gram samples in a batch reactor, with five different coals, indicate that the process has desulfurization potential of up to 63 percent of sulfur with basic dry oxidation plus water washing treatment and up to 89 percent with dry oxidation followed by caustic treatment and water washing. Table 55³⁶ presents the results of the laboratory studies. The results indicate that higher desulfurization is achieved when the treat-gas contains 10 percent by volume of nitric oxide.

The washing step removes iron and loosely bound inorganic material which reduces the ash content of the coal. KVB claims a 95+ percent ash removal with their system, however, there are no published experimental results to substantiate this claim.

Sulfur By-Products—

In the KVB process all the pyritic sulfur is converted to either sulfites or sulfates. No elemental sulfur is produced by this process.

TABLE 55. COAL DESULFURIZATION DATA USING THE KVB PROCESS

Coal Sample Identification	Size Mesh	Oxidation 200°F			Feed Sulfur Level		Sulfur Level After Oxidation		Sulfur Level After Water Wash		Sulfur Level After 10% NaOH Wash & water wash	
		Time Hrs.†	NO in Air % Vol.	Gas Flow l/min.	Total	Organic	Total S	% Sulfur Removed	Total S	% Sulfur Removed	Total S	% Sulfur Removed‡
Lower Kittanning	-14to+28	-	-	-	4.3	0.7	-	-	-	-	4.5 [§]	0
	-14to+28	3	5	.42	4.3	0.7	3.3	23	2.4	43	2.1	51
	-14to+28	3	10	.44	4.3	0.7	-	-	1.6	63	0.5	89
	-14to+28	1.5	10	.44	4.3	0.7	-	-	-	-	1.4	67
	-80to+100	3	5	.42	4.3	0.7	-	-	-	-	2.9	32
Illinois #5	-14to+28	1.5	10	.42	3.0	1.9	-	-	-	-	2.5	17
	-14to+28	3	10	.44	3.0	1.9	-	-	2.0	33	1.0	67
	-14to+28	3	5	.42	3.0	1.0	-	-	1.9	37	1.2	59
K-16914 ^Δ	-14to+28	3.5	10	.44	6.7	1.16	4.2	37	3.1	54	3.2	52
K-14702 ^Δ	-14to+28	3.0	5	.42	5.3	1.3	4.3	19	3.0	43	3.1	41
	-14to+28	3.0	10	.44	5.3	1.3	2.7	49	2.5	53	-	-
K-16394 ^Δ	-14to+28	3.0	5	.42	3.2	1.9	2.5	22	-	-	-	-
	-14to+28	3.0	10	.44	3.2	1.9	2.0	38	-	-	-	-

† No oxidation, wash only.

Δ U.S. Bureau of Mines Designation.

† It is claimed that recent tests achieved the same results in 10 minutes using a rotary reactor.

‡ The samples were dried at 250°F before analysis.

Sulfur is removed from the coal as sulfur oxides, in the gas stream, or as soluble sulfates by caustic and water wash. Sulfur leaves the process as sulfurous and sulfuric acid, which may be commercially saleable, and as calcium salts which must be disposed.

Benefit Analysis—

The main benefit associated with the KVB process is the developer's claim that the process removes all three forms of sulfur in the coal (pyritic, sulfate and organic sulfur). This means that it may have general applicability and greater capability to handle the variations in sulfur distribution in the process feed than some other processes.

Additionally, the process is claimed to require relatively coarsely ground coal (+28 mesh). This characteristic would facilitate the feed coal and product coal handling operations. However, all tests have been made on very closely sized fractions. This chemical coal cleaning process is also claimed to reduce a major portion of the ash content of the feed coal.

No analysis has yet been conducted to determine the nitrogen or the trace metals content of the treated product. However, it is anticipated that the KVB desulfurization process will remove some of the trace metals in the coal while reducing the total ash content of the coal.

The process utilizes moderate temperatures, pressures and residence time and has a high coal yield (up to 87 percent) with essentially complete recovery of carbon and hydrogen values in the coal feed. The net heating value recovery of the system is estimated to be about 91 percent.

Environmental Aspects—

Gaseous emissions from the integrated system will primarily include water vapor, carbon dioxide, nitrogen and may contain some small quantities of sulfur and nitrogen oxides. Since the basic process is a dry oxidation system, there will be some dust problems; however, adherence to good engineering practices should keep these to a minimum. In the reactor system, the coal fines in the reaction gases will be recovered because these gases will be passed through a two-stage, internal cyclone separator prior to leaving the reactor. Both the feed coal and the treated product would be stored in lock hoppers and introduced to the reactor or removed from the system

through air lock rotary valves. The lock hoppers will be continuously purged with nitrogen to prevent the formation of an explosive dust. The vent from each lock hopper may be piped to the lime treatment tank, located in the caustic regeneration section for the removal of traces of nitrogen dioxide and sulfur oxides before venting these gases to the atmosphere. In the vicinity of the chemical coal cleaning plant, coal handling, crushing, grinding and conveying facilities may need to be equipped with dust control equipment.

The process generates solid waste consisting primarily of gypsum with calcium carbonate and coal ash. This waste material must be handled in an environmentally safe manner since it will contain some trace metals.

Essentially no waterborne waste will be generated by this system assuming the plant can be designed to operate as a closed loop system. Caustic solution would be regenerated and recycled to the extractor and all water condensate from the process can be utilized as wash water in the process. The process has a by-product acid stream, originating from the off-gas scrubber, which may have a market value, or may present a disposal problem.

Problem Areas—

The KVB process is still at its early stages of development; thus, it is premature to precisely define problem areas for this system. It should be mentioned, however, that the oxygen concentration requirements in the treat-gas exceed the explosion limits for coal dust, and thus the operation of this process may be hazardous. Furthermore, nitrogen uptake by the coal structure will increase NO_x emission and therefore may limit the marketability of the product.

R&D Efforts and Needs—

Specific research efforts and needs for this process are:

- A development program is required to determine the effects of process variables on the sulfur content of the product coal;

- Extensive bench-scale and pilot level technical effort is necessary to establish accurate heat and material balance information and assess the process economics;
- Since the process can be carried out using a continuous reactor or with batch reactors, data should be generated to determine which system is more suitable for various coal feeds.
- It is claimed that a batch reactor system may permit the reaction and extraction operation to be conducted in the same vessel. This possibility should be investigated since it may prove to be very economical.
- The basic chemistry of the process should be studied to develop a better understanding of NO_2 (active gas) oxidation of coal as applied to the removal of the various forms of sulfur. This study should define the rate of the active gas formation, and the degree of sulfur and ash removal as a function of the processing variables such as:
 - treat-gas to coal ratio
 - caustic or water to coal ratio
 - caustic concentration
 - reactor residence time;
- The caustic extracted coals may have a high potential to slag in boiler furnaces. Therefore, the physical, chemical and combustion characteristics of the treated product should be studied; and
- Studies should be conducted to determine the optimum method of removal of oxidized sulfur forms from the treated dry coal.

Process Economics

The cost estimates developed for this process should be considered preliminary since adequate process engineering information is unavailable at this time for the development of an accurate and optimum process flow sheet. The economic estimates presented herein are based on a plant operating on 7,200 metric tons (8,000 tons) per day of coal throughput. These economics are based on the Bechtel's flow scheme⁸ for the chemical treatment plant; however, it incorporates Versar's estimates for the annual operating costs based on discussions with KVB.

The flow sheet for this treatment plant, as developed by Bechtel, with corresponding mass balance and stream properties is given in Appendix VIII. A summary pertinent to the coal balance is given in Table 56. The other raw materials, utilities and the waste stream have been expressed as a function of the product coal less moisture in Table 57.

A summary of economics of the KVB process is given in Table 58. Details on the estimated installed capital costs for this process, including caustic extraction, are given in Table 59. However, if the basic dry oxidation process alone would be sufficient for sulfur removal, this process may prove to be a relatively low cost method of coal desulfurization.

The estimated annual operating costs are presented in Table 60. The unit operating costs shown are based on a clean coal yield of 85.3 percent (dry basis) and a heating value recovery of 91 percent. The single largest cost item, other than purchased coal used as feed, is the steam requirement projected for this process. The evaporation system, for the control of gypsum build-up in the recycle water, is a costly item in the process economics. It has been assumed that only one-third of the product coal is less than 28 mesh and these fines are compacted prior to shipment.

TABLE 56. KVB PROCESS COAL BALANCE

Coal Feed	<u>Kg /100 Kg coal feed</u>	
Coal *	72.6	
Ash	22.7	
Sulfur	1.9	
Moisture	<u>2.8</u>	
Subtotal	100.0	
Fuel ^Δ	<u>KVB Estimate</u>	<u>Bechtel Estimate</u>
Coal *	6.23	6.23
Ash	0.09 ^α	1.69 ^φ
Sulfur	0.04	0.04
Moisture	<u>0.09</u>	<u>0.09</u>
	6.45	8.05
Product [†]		
Coal *	66.77	66.77
Ash	0.91	18.11
Sulfur	0.46	0.46
Moisture	<u>0.91</u>	<u>0.91</u>
	69.05	86.25
Ash Loss by difference	21.7 ^α	2.9 ^φ
Product [‡]		
Coal dry basis	68.14	85.34
Binder	1.02	1.28
Moisture	<u>2.72</u>	<u>3.41</u>
	71.88	90.03

NOTES:

* Moisture, sulfur and ash free basis.

Δ Product coal used as fuel in the system

† Net product without binder

^α Ash reduction estimated by KVB for their system. There are, however, no experimental data to support this claim^φ Ash reduction estimated by Bechtel for this system[‡] Treated product with binder and 4 percent moisture.

TABLE 57. KVB PROCESS RAW MATERIALS, UTILITIES AND WASTE STREAMS BALANCE

	<u>Units</u>	<u>Basis: 100 metric Tons coal Received</u>	<u>Unit Ratio</u>
Product Coal without binder, dry basis	Metric Tons	85.34	1.0
Coal received, dry basis	Metric Tons	97.2	1.139
Fuel coal, dry basis	Metric Tons	7.96	0.093
Ash loss	Metric Tons	2.9	0.034
Oxygen	Metric Tons	2.8	0.033
Nitrogen dioxide	Metric Tons	0.3	0.351
Water	Metric Tons	67	0.785
Caustic (100%)	Metric Tons	0.5	0.006
Lime (100%)	Metric Tons	2.5	0.029
Steam (150 psia)	Metric Tons	75.75	0.888
Power	Kw	2004*	23.5
Binder	Metric Tons	1.28	0.015
Gypsum waste (63% solid)	Metric Tons	5.0	0.059

Note: * Power requirement based on 100 metric Tons/hr feed

TABLE 58. SUMMARY OF ECONOMICS FOR THE KVB CHEMICAL
COAL CLEANING PROCESS

Basis: 7,200 metric tons (8,000 tons) per day of 6,800 kg cal/kg
(12,300 BTU/lb) coal

90.4% operating factor (330 days/yr)

Capital amortized for 20 years @ 10% interest

Grass roots plant installation

85.3% weight yield, 91% heating value recovery

Installed Capital Cost: \$65,940,000

Annual Operating Costs

on Clean Coal Basis: \$41,059,000 process cost, excluding coal cost
\$107,059,000 process cost, including coal cost*
\$20.10 /metric ton (\$18.23 /ton), excluding coal cost
\$52.40 /metric ton (\$47.54 /ton), including coal cost*
\$2.75 /10⁶ kg cal (\$0.69 /10⁶ BTU), excluding coal cost
\$7.22 /10⁶ kg cal (\$1.81 /10⁶ BTU), including coal cost*

* Coal costed at \$27.60/metric ton (\$25/ton)

TABLE 59. INSTALLED CAPITAL COST ESTIMATE FOR THE KVB
CHEMICAL COAL CLEANING PROCESS

	<u>\$ 1977</u>
Coal handling and preparation*	\$ 6,000,000
Desulfurization process costs ^Δ	35,100,000
Compacting and product handling [†]	4,400,000
Building and miscellaneous ^α	700,000
Utilities (off-sites)	---
Site development and general ^φ	<u>4,525,000</u>
Subtotal	\$50,725,000
Engineering design @ 10%	5,070,000
Contingency @ 20%	<u>10,145,000</u>
Total Installed Plant Capital (TPC)	\$65,940,000

* Versar estimate based on crushing and sizing the coal to +28 mesh

Δ Bechtel estimate adjusted to 1st quarter 1977 price using CE plant cost index; includes off-sites.

† Versar estimate; assumes only one-third of the product coal is less than 28 mesh and these fines are compacted prior to shipment.

α Includes administrative buildings, the maintenance shop, stockrooms and stores.

φ Includes railroad facilities for incoming and outgoing cars and loading and unloading facilities for raw materials and loading facilities for by-product waste material.

TABLE 60. ESTIMATED ANNUAL OPERATING COSTS FOR THE KVB
CHEMICAL COAL CLEANING PROCESS

	\$
Amortization 20 years @ 10% interest (factor = 0.1175)	\$ 7,748,000
Taxes @ 2% TPC	1,319,000
Insurance @ 1% TPC	659,000
Labor (direct, indirect additives and support)	440,000
General and administrative @ 1.5% TPC	989,000
Maintenance and supplies @ 5% TPC	3,297,000
Utilities:	
Electric power	1,200,000
Water	71,000
Steam*	16,000,000
Chemicals:	
Oxygen	1,848,000
Nitrogen dioxide	1,584,000
Caustic	2,112,000
Lime Δ	2,310,000
Binder	1,350,000
Waste Disposal	132,000
Total Annual Processing Cost	41,059,000
Raw coal, 2.39×10^6 metric tons (2.64×10^6 tons)	66,000,000
TOTAL ANNUAL COST	\$107,059,000

* Assumes purchased steam (150 psia) @ \$8.81/1,000 kg (\$4/1,000 lb).

Δ Assumes one-third of the product will require compaction.

ATLANTIC RICHFIELD COMPANY CHEMICAL COAL CLEANING PROCESS

Process Description

The Atlantic Richfield Company (ARCO) is developing a chemical coal cleaning process at Harvey, Illinois, which removes both pyritic and organic sulfur compounds and ash from coal. The process requires the use of either a recoverable or a non-recoverable reaction promoter.

Very little has been published about the process, no flow sheet is available, and ARCO has not permitted an on-site inspection.

Status of the Process

Process development work has largely proceeded on the basis of data generated from batch bench scale experiments. However, a 0.45 kg (1-pound) per hour continuous reactor system was recently built and is currently being used to provide additional data.

Until recently ARCO has financed this experimental program without external assistance. The Electric Power Research Institute, Palo Alto, California (EPRI) has financed a study on the continuous reactor system on five coals in which there is a wide distribution of pyrite particle size. This study is now complete and a final report is expected to issue in 1978. The EPRI contract has been extended to demonstrate in the continuous pilot plant, low cost process options which ARCO has developed.

Technical Evaluation of the Process

Potential for Sulfur Removal--

The five coals selected by EPRI and tested in the ARCO process are:

- Lower Kittanning, Martinka #1
- Illinois #6, Burning Star #2
- Pittsburgh #8, Montour #4
- Western Kentucky #9/14, Colonial
- Sewickley, Green County, Pennsylvania (beneficiated)

The coals were selected to meet the following criteria:

- Mean pyrite crystallite chord size for the five coals should cover a wide range.

- Pyrite and organic sulfur content should cover a wide range.
- Reduction of sulfur content to the NSPS compliance level; i.e., to 1.1 g/M cal. (0.6 lbs./M BTU), should be attainable by removal of pyritic sulfur in the case of at least one coal.
- The coals should be from producing mines on seams with substantial reserves.

Depending on the coal treated, overall reduction of sulfur was up to 98% for pyritic sulfur, up to 20% for organic sulfur, and 66-72% for total sulfur. Overall reduction of iron was up to 96% and of ash up to 78%. The BTU yield of the process is estimated at 90-98%. Ash content of the product is frequently reduced by 50%, compared to feed coal, and the process weight yield is about 95%, depending on ash removal.

Sulfur By-Products—

ARCO process by-products consist of gypsum plus an iron-containing by-product. No other data is available.

Environmental Aspects—

The process allegedly has no off-gases. Disposition of trace metals is unknown, but there is a possibility that some could be commingled with the by-product gypsum. No other data is available.

Problem Areas—

Although the process appears adequate to meet NSPS for Eastern coals with low organic sulfur, process improvements are required before high organic sulfur coals will meet NSPS.

R&D Efforts and Needs—

R&D efforts and needs are not known at this time.

Process Economics

Little was learned about ARCO process capital requirements and operating costs. Various process variations have been estimated at \$17 to \$31 per ton of product. The low cost options are now receiving EPRI development support. Steam requirements are costed at \$5.72/1,000 kg (\$2.00/1,000 lb.), regardless of pressure. Solids disposal costs of \$11/metric ton (\$10/ton) of dry solids are used.

MISCELLANEOUS CHEMICAL COAL CLEANING PROCESSES

University of Houston Process

Development of this process is proceeding under the direction of Dr. Attar of the Chemical Engineering Department of the University of Houston. In a telephone interview in July, 1977, Dr. Attar stated that the work involves bench-scale development of a modified version of the Battelle process.⁴⁰ The significant difference between the two processes is claimed to be a modified leaching process (proprietary at this time) which results in much lower residual sodium in the coal than the Battelle leaching conditions yield.

The University of Houston process claims to remove essentially all of the pyritic sulfur and better than 40% of the organic sulfur (It is believed that this process is removing at least the mercaptan and aliphatic organic sulfur forms). This project is studying methods to regenerate an additive to the leachant which apparently represses the bonding of sodium to the coal. Experiments have been on 10 gram samples (using Illinois #6 coal) to this point, but larger samples of 454 grams (1 lb) will be tried. The effort is being funded internally.

National Research Council (NRC), Canada Process

The National Research Council of Canada (NRC) is actively developing and optimizing a process involving the agglomeration of the carbonaceous constituents of finely divided coals. Different oils are used as collector liquids, while the inorganic constituents remain in aqueous suspension and are rejected.^{41,42} Oil agglomeration relies on differences in the surface properties of coal and inorganic minerals to effect separation, as does froth flotation. However, flotation is effective primarily in the 45 to 200 mesh range, whereas agglomeration is claimed to have no lower limit on particle size, and can treat particles up to 3 mm (1/8 inch) in diameter.⁴³

Coal agglomeration is generally considered to be a physical coal cleaning process as opposed to a chemical coal cleaning process.

However, in the late 1960's and early 1970's NRC performed some exploratory research utilizing selected bacteria to effect surface oxidation of metal sulfides in ores and pyrite in coal to render the surfaces more hydrophilic. Selective agglomeration (or flotation) is then even more effective. After dropping this effort for several years, it was reactivated in 1977. The research is being performed at the University of Waterloo, Ontario, Canada, by Dr. Kempton.

Jolevil Process

This process was developed for Jolevil Associates, Inc., Hoover, Alabama, by the Southern Research Institute, Birmingham, Alabama. The process is considered proprietary (patent applied for) with only sketchy details available.⁴⁴ Jolevil management will release no specific details. The basic principle involved appears to be wet oxidation of pyritic sulfur in coal using air at 10-14 atm. (150-200 psi) and temperatures up to 120°C (250°F). The process is claimed to remove most of the pyritic sulfur and does not affect organic sulfur. Indications are that the process could be used in a coal slurry pipeline application. There is no cost data available.

Ohio State University Process

Development of a microbiological process for coal desulfurization is under the direction of Dr. Patrick R. Dugan, of the Microbiology Department at the Ohio State University, Columbus, Ohio. The experimental effort is currently in the laboratory stage and is privately funded. The work to date is scheduled to be published.⁴⁵

The study has been conducted for approximately 8 months with a pulverized coal blend supplied by a local utility. The total sulfur content of this coal is 4.6% with about 3.1% pyritic sulfur. The coal has been screened and used in two mesh size ranges as well as the "as received" material. Microbiological treatment of these fractions has resulted in sulfur reductions as tabulated

RESULTS OF MICROBIOLOGICAL TREATMENT OF UTILITY
COAL FOR SULFUR REMOVAL

<u>Mesh Size Range</u>	<u>Initial Sulfur Percentage</u>		<u>Final Sulfur Percentage</u>	
	<u>Total</u>	<u>Pyritic</u>	<u>Total</u>	<u>Pyritic</u>
as received	4.6	3.1	2.0	0.1
100 - 200 mesh	4.1	2.9	1.8	0.1
-200 mesh	5.4	2.9	2.0	0.1

Treatment time was reduced from 20 days in the initial tests to around 7 days in tests currently being run. The microbiological treatment is effective in removing better than 96% of the pyritic sulfur, but appears to have little or no effect on organic sulfur. (There is about 20% reduction of organic sulfur in the -200 mesh fraction; this reduction, however, is probably within the experimental error of the sulfur determinations.)

Dr. Dugan is currently assessing the results of the work to date and will decide on the future direction of the effort.

Western Illinois University Process

Dr. M. Venugopalan of the Department of Chemistry of Western Illinois University, Macomb, Illinois, has conceived a process for coal gasification and desulfurization utilizing a plasma jet, and has constructed a laboratory unit. A few experiments have been carried out to date with some indication of success. For example, during an 8-hour run, the total sulfur content of an essentially dry Illinois #6 coal (plus 6 mesh) was reduced from 2.1% to 1.5%.⁴⁶ Argon was initially used as the inert plasma gas, but the equipment has been operated on hydrogen gas as well. Runs in a one meter long tube are carried out with about a 100 g sample of coal using 60-100 watts of electrical power and achieving temperatures of about 1,200°C. Off-gases are analyzed for methane, ethane and hydrogen sulfide. Further experiments are planned utilizing finer coal particles, and varying other parameters in order to obtain more supportive data. With argon, the methane off-gas is derived entirely from the coal, but with hydrogen plasma, the coal will probably not lose very much hydrogen. No other data is available at this time.

Texaco Process

This process was conceptualized for treating a pipelined coal slurry in-situ for pyritic sulfur reduction. A pyrite oxidant such as hydrogen peroxide would be added to the slurry, upstream of the dewatering plant in order to accomplish pyritic sulfur reduction. A process patent was issued to Texaco, Inc. (November 23, 1976), but the idea has been shelved for the present.

U.S. Steel Process

This process was developed by Dr. P. X. Mascantonio at the U.S. Steel Research Laboratories, Monroeville, Pennsylvania. The process is based on treatment of coal with a molten mixture of alkali hydroxides at elevated temperatures and atmosphere pressure. The process, as conceptualized in U.S. Patent 3,166,483, involves separation of the mixture of fused hydroxides and desulfurized coal by decanting, with the molten hydroxide mixture recycled to the coal processing step. The desulfurized coal is washed free of residual alkali which is recycled to the coal processing step in a molten form.⁴⁸ The aqueous wash solution is concentrated to the anhydrous state, melted and recycled.

The U.S. Steel process was shown to remove significant amounts of sulfur from the coals tested. However, the physical properties of the treated product underwent drastic changes, making the material unusable for combustion in utility boilers. Additionally, high residual sodium levels would be expected in the treated product, which would create severe slagging and fouling problems in conventional boilers. The research program on this approach was discontinued for these reasons.⁴⁹

Kellogg Process

This process was developed by the M.W. Kellogg Company, Houston, Texas. The process was abandoned as unworkable in 1972 after some preliminary experimental work was attempted and a report issued to IERL/RTP/EPA in June 1972.⁵⁰ The basis of the work performed was to remove sulfur from coal using an iron oxide catalyst in the presence of hydrogen at elevated temperatures and pressures. Appreciable desulfurization resulted from this treatment (about

85% total sulfur removal), but the treated coal yield was only 58%. No process economics are available, but the poor yield would appear to make the process unattractive. Also it appears that the coal matrix might be adversely affected by this treatment.

Chemical Construction Corporation (Chemico) Process

The Chemico process consists primarily of the reduction of the pyritic sulfur content of coal by reaction with water and air at elevated temperatures and pressures. Experimentation has consisted of limited bench scale treatment of ground coal in several hundred gram quantities with water and air. Appreciable pyritic sulfur reduction in the treated coal was claimed.

Chemico obtained a conceptual process patent on July 16, 1974. No process developed work has been carried out since the patent was issued and no cost estimates have been published.⁵¹

The Chemico process is quite similar in concept to the Ledgemont (Kennecott) and ERDA oxydesulfurization processes.

University of Florida Process

Experimental work was performed under an ERDA grant (No. 801296) at the Chemical Engineering Department of the University of Florida, Gainesville, Florida, during 1974 to determine the effectiveness of various gases, at elevated temperatures and atmospheric pressure, for the desulfurization of coal. Three gases were used (air, steam and hydrogen) in attempts to desulfurize 10 high-volatile bituminous coals. Only hydrogen was reported to be effective in reducing the total sulfur to meet the current EPA sulfur dioxide emission standard.

The results achieved with hydrogen closely corroborate the results currently being obtained by the IGT hydrodesulfurization process. The University of Florida work showed that maximum total sulfur reduction was achieved by hydrogen treatment and temperatures around 480°C (900°F). At this temperature, 86% of the total sulfur was removed, including 94% of the inorganic and 76% of the organic sulfur. An oxidation pretreatment was performed in the Florida work (typically for 10 minutes at 150°C (300°F))

in order to reduce coal caking properties. Hydrogenation without oxidative pretreatment took 15 times as long to remove the same amount of sulfur as compared to the hydrodesulfurization of pretreated coal. This result also parallels the IGT observations. Weight loss of the hydrogen treated coal at 480°C (900°F) was approximately 40%, a result which the IGT work has also duplicated.

No further work has been carried out at the University of Florida since these experimental runs were completed and no further work is contemplated.

Laramie Process

The Laramie process developed at ERDA's Laramie, Wyoming, Energy Research Center, consists of a coal treatment step only - reacting ground coal at ambient temperatures and pressures with a mixture of sulfuric acid and hydrogen peroxide. Five different bituminous coals have been treated. The mild oxidation treatment results in partial pyritic sulfur and ash extraction into the aqueous phase but does not affect organic sulfur. Studies to date have only been at the laboratory level. The work on this approach has been suspended for some time, due to lack of personnel, facilities, and the need to carry on other more pressing research efforts. There is no information available regarding the process flow scheme or process economics.

Dynatech Process

The principle of aerobic microbial leaching of pyritic sulfur from coal formed the basis for a proposal submitted to IERL/RTP/EPA in July, 1975, by the Dynatech Company of Cambridge, Massachusetts. There apparently has been no experimental work carried out by Dynatech to evaluate the approach and no action was taken by EPA on the proposal.

Kyoto Process

Laboratory-scale experimentation was carried out in Japan at Kyoto University in 1969 in attempts to remove both inorganic and organic sulfur by reaction with chlorine or hydrogen peroxide at ambient temperatures and pressures.⁵² No attempts were made to characterize off-gases or define the chemical reactions. Indications were that while most of the inorganic sulfur and some organic sulfur removal was affected, processing times were slow

and a large amount of reagent was necessary. As far as is known, there has not been any follow-up process development work or cost studies performed.

Methonics Process

The intention of Methonics, Inc. was to develop a process based on hydrogen attack of the organic sulfur portion of coal, after initial dissolution in a solvent and removal of the pyritic sulfur by filtration. In this process, the product would no longer be in a solid form. Professor Wiser of the University of Utah College of Mines and Mineral Industries indicated that no experimental work was ever performed to develop the Methonics idea.

Rare Earth's Process

This process was proposed by Rare Earth Industries, Inc., Orlando, Florida, and is entirely conceptual in nature. The process is claimed to remove organic sulfur from liquefied coal in the solvent refined coal (SRC) process, using rare earths as scavengers for the sulfur. This sulfur removal process was considered outside the scope of the present Versar study since the coal no longer is in the solid form.

MIT Process

The Massachusetts Institute of Technology, Cambridge, Massachusetts, (MIT), Chemical Engineering Department, is concerned with developing improved methods of removing sulfur and nitrogen contaminants in liquid fuels by catalytic hydrogenation treatment. For coal to be treated by this method, it would be necessary to subject the coal to liquefaction prior to processing it. The MIT process, therefore, cannot be considered as a chemical coal cleaning process since the product is no longer a coal-like material.^{53,54}

Rutgers University Process

This process developed by Rutgers University, New Brunswick, New Jersey, featured microbiological treatment of coal in an attempt to reduce organic sulfur levels. Treated coal samples submitted to the Bureau of Mines for analysis showed no reduction of organic sulfur level. No other information is available on this process, and it is believed inactive.

The Gulf & Western Process

The Gulf & Western Advanced Development and Engineering Center, Swarthmore, Pennsylvania, has been involved in a program to develop a coal desulfurization process. The program objectives as recently reported included:⁵⁵

- Exploring the utility of graft polymerization for solubilizing coal to remove pyritic sulfur and ash.
- Chemically and physically characterizing grafted coal specimens with respect to composition, molecular weight and other properties.

This program was ERDA funded until the spring of 1977. The process consisted of an attempt to liquefy coal by a graft polymerization technique using a free radical mechanism. Approximately 30% of the coal was Liquefied using benzene at 70°C (160°F). The sulfur level in the solubilized extract was 0.7% as compared to 2% in the original coal. At the present time ERDA is no longer funding the project. The process could not be justified from an economics standpoint.

Colorado School of Mines Research Institute (CSMRI) Process

The Electric Power Research Institute (EPRI) is financing a technical program at CSMRI in Golden, Colorado, which will evaluate the physical beneficiation of coals through the use of magnetic fluids.⁵⁶ Commercially available magnetic fluids, and some non commercial fluids, will be obtained and tested with specific coals, starting with high-ash western subbituminous grades. The work will proceed from laboratory studies through pilot-plant stage, if justified. Economic as well as technical evaluation will continue throughout the term of the project.

This project is concerned with stable colloidal suspensions of submicron size ferro - or ferrimagnetic particles in a carrier such as water or kerosene, with a dispersing agent. Application of a magnetic field to such fluids can levitate or float materials which are much more dense than the fluid itself. Thus, if ash in a coal/ash mixture has a greater affinity for a magnetic fluid than coal, it may be separated magnetically from the coal. It is hoped that pyritic sulfur will be selectively removed with the ash.

SECTION 4

PROCESS AND COST COMPARISON

This section presents a comparison of technical and economic results obtained from the assessment of major chemical coal cleaning processes as described and discussed in Section 3 of this report. The analysis and conclusions presented herein are based on process claims made by individual developers, research reports and published information.

Most processes included in this report are not at an adequate developmental stage to permit the preparation of a precise engineering process flow sheet for capital and operating cost evaluation. Thus, the process economics presented for most processes are best engineering estimates, based upon the information available.

SULFUR REMOVAL AND HEATING VALUE RECOVERY POTENTIAL

A comparison of process performance and costs can best be accomplished by looking at each process on a common coal feed. This basis allows the comparison of the following parameters process by process:

- Weight yield of clean coal product based upon a feed coal rate (moisture free basis) of 7,110 metric tons (7,840 tons) per day [7,200 metric tons (8,000 tons) per day of 2 percent moisture coal];
- Weight percent sulfur in the clean coal product based upon the sulfur removal efficiency of the process;
- Heating value yield of the process based upon a feed coal value of 6,800 kg cal/kg (12,300 BTU/lb) and net heating value yield in percent; and
- Costs -
 - total capital costs for the process,
 - total annual processing costs,
 - annual costs per metric ton of clean coal, including coal costs and excluding coal costs, and

annual costs per heating value unit, including coal costs and excluding coal costs.

This comparison data is shown in Table 61, arranged according to categories of processes.

The common coal feed selected is a bituminous coal from the Pittsburgh seam, which cannot readily be cleaned by conventional washing techniques to meet the current new source performance standards for sulfur dioxide emission. This coal does have an organic sulfur content low enough (0.7 weight percent) so that complete removal of pyritic sulfur would result in a product which will meet current NSPS for sulfur dioxide emission.

The percent removal of pyritic and organic sulfur assigned to each process is based on data supplied by individual developers. The table indicates a range of SO₂ emission levels for the clean coal products of 1.5 to 3.8 kg/10⁶ kg cal (0.8 to 2.1 lb/10⁶ BTU). The calculated sulfur levels for processes which remove both types of sulfur are lower than the 2.2 kg/10⁶ kg cal (1.2 lb/10⁶ BTU) NSPS for sulfur dioxide emission. Of the four processes which remove pyritic sulfur only, two (TRW and Ledgeмонт) will produce slightly higher sulfur levels than that required to meet the current NSPS; however, within the levels of accuracies involved they also might be considered to be in compliance. The remaining two processes [Magnex and Syracuse] would produce coal which would be in compliance only with a standard of 4.3 kg/10⁶ kg cal (2.4 lb/10⁶ BTU) for sulfur dioxide emission.

Processes which remove pyritic sulfur alone are primarily applicable to coals rich in pyritic sulfur, so that efficient removal of pyritic sulfur could bring these coals into compliance. Processes which remove both types of sulfur are primarily applicable to coals which cannot be adequately treated by pyritic removal processes. All chemical coal cleaning processes are more selective and efficient than conventional coal cleaning techniques and it is very likely that each process will eventually find an area of application.

As shown in Table 61, the heating value yields estimated for these processes are generally greater than 90 percent with a range from a low 57 percent for the IGT process to a high of 96 percent for the GE process.

TABLE 61. PROCESS PERFORMANCE AND COST COMPARISON FOR MAJOR CHEMICAL COAL CLEANING PROCESSES

		PROCESSES WHICH REMOVE PYRITIC SULFUR ONLY				PROCESSES WHICH REMOVE PYRITIC AND ORGANIC SULFUR						
	FEED [†]	TRW	LOL	MAGNEX SM	SYRACUSE ⁺ PHYSICAL CLEANING	ERDA	GE	BATTELLE	JPL	IGT	KVB	ARCO
NET COAL YIELD, METRIC TONS PER DAY (TONS/DAY) ^Δ	7,110 (7,840)	6,400 (7,056)	6,400 (7,056)	5,645 (6,225)	5,645 (6,225)	6,400 (7,056)	6,826 (7,526)	6,755 (7,448)	6,470 (7,135)	4,270 (4,704)	6,070 (6,690)	6,400 (7,056)
WEIGHT % SULFUR IN THE PRODUCT	1.93	0.83	0.83	0.97	1.50	0.65	0.50	0.65	0.6	0.55	0.61	0.69
HEATING VALUE, KG CAL/KG (BTU/LB)	6,800 (12,300)	7,100 (12,835)	7,100 (12,835)	6,900 (12,400)	8,100 (14,600)	7,100 (12,835)	6,800 (12,300)	6,300 (11,350)	6,800 (12,300)	6,500 (11,685)	7,300 (13,120)	6,900 (12,400)
KG SO ₂ /MM KG CAL (LB SO ₂ /MM BTU)	5.6(3.1)	2.3(1.3)	2.3(1.3)	2.8(1.6)	3.8(2.1)	1.6(0.9)	1.5(0.8)	2.2(1.2)	1.8(1.0)	1.6(0.9)	1.6(0.9)	2.0(1.1)
PERCENT NET BTU YIELD	--	94	94	80	95	94	96	88	91	57	91	91
COSTS												
CAPITAL (\$MM)	--	109	114.0	37.8	49.0	166.8	102.0	168.6	103.2	134.6	65.9	--
ANNUAL PROCESSING (\$MM)	--	37.2	45.3	19.2	12.2	56.6	39.8	74.2	44.4	38.3	41.0	58.7
\$/ANNUAL METRIC TON	--	17.2	21.0	10.1	6.4	26.3	17.3	32.6	20.4	26.6	20.1	27.5
(\$/ANNUAL TON) OF CLEAN COAL, EXCLUDING COAL COST	--	(15.6)	(19.1)	(9.2)	(5.8)	(23.8)	(15.7)	(29.6)	(18.5)	(24.2)	(18.2)	(25)
\$/ANNUAL METRIC TON	--	47.9	51.6	44.8	40.8	56.9	46.0	61.6	50.7	72.6	52.4	--
(\$/ANNUAL TON) OF CLEAN COAL, INCLUDING COAL COST ^Δ	--	(43.4)	(46.9)	(40.7)	(37.0)	(51.6)	(41.8)	(55.9)	(46.0)	(65.8)	(47.5)	--
\$/MM KG CAL (\$/MM BTU), EXCLUDING COAL COST	--	2.42 (0.61)	2.94 (0.74)	1.47 (0.37)	0.78 (0.20)	3.69 (0.92)	2.54 (0.64)	5.15 (1.30)	2.97 (0.75)	4.09 (1.03)	2.75 (0.69)	--
\$/MM KG CAL (\$/MM BTU), INCLUDING COAL COST ^Δ	--	6.71 (1.69)	7.23 (1.82)	6.52 (1.64)	5.03 (1.27)	7.98 (2.00)	6.72 (1.69)	9.74 (2.45)	7.40 (1.86)	11.2 (2.81)	7.22 (1.81)	--

* ALL VALUES REPORTED ARE ON A MOISTURE FREE BASIS.

† THE COAL SELECTED IS A PITTSBURGH SEAM COAL FROM PENNSYLVANIA WHICH CONTAINS 1.22 WEIGHT PERCENT PYRITIC, 0.01 PERCENT SULFATE AND 0.70 PERCENT ORGANIC SULFUR. IT IS ASSUMED THAT THIS COAL HAS A HEATING VALUE OF 6,800 KG CAL/KG (12,300 BTU/LB).

Δ ASSUMES COAL FEED @ \$27.6/METRIC TON (\$25/TON).

All heating value yields listed in Table 61 reflect both the coal loss due to processing and the coal used to provide in-process heating needs. However, with the exception of the IGT process, the actual coal loss due to processing is claimed to be small. For most processes, the major heating value loss is due to the use of clean coal for in-process heating.

It is believed that the high yield estimated for the GE process may not adequately reflect the heat requirements that may be needed to regenerate the caustic reagent employed in the process. This process is in its early stage of development and as such, the energy requirements for the process cannot be properly assessed at this time. It is possible, that in the final analysis, the heating value recovery from this process will be more in line with other chemical coal cleaning processes.

COST COMPARISON FOR MAJOR CHEMICAL COAL CLEANING PROCESSES

Estimates of capital and annual operating costs for each major chemical coal cleaning process are also given in Table 61. These estimates are based on an assumed plant throughput capacity of 7,200 metric tons (8,000 tons) per day, equivalent to a 750 M.W. electric power plant. The total annual operating costs for each process, including and excluding cost of the raw coal, have been expressed also in terms of dollars per metric ton and dollars per million kg cal heat content in the coal.

The capital cost estimate prepared by each process developer was used as the basis of the cost estimates in this report. In some cases, these costs were modified to allow the evaluation of the various processes on a comparable basis. The estimated capital costs assume a grass roots operation including costs for coal crushing, grinding, product compacting and feed and product handling. The capital costs also include land acquisition and site development, off-site facilities, and engineering and design costs. A contingency allowance of 20 percent has been included in all estimates, with the exception of TRW's. A lower contingency allowance (10 percent) was used for the TRW process since it is at a more advanced stage of development and adequate process data is available to develop the economics of this process with a greater degree of confidence.

Annual operating costs are based on a 24-hour workday, 90.4 percent service factor (330 days per year) basis. The capital cost is amortized over a period of 20 years at 10 percent interest per year. Where adequate information was available, the utilities and chemical consumptions are based upon actual process demand. The operating labor costs reflect wage rates for the Pittsburgh, Pennsylvania, area. The estimates for the maintenance and supplies, general and administrative, taxes and insurance are taken as 5, 1.5, 2 and 1 percent on total installed plant capital cost (TPC), respectively.

Capital Cost Comparisons

In general, pyritic sulfur removal processes require the least amount of capital investment. However, these processes have limited sulfur removal efficiencies.

Among processes that remove both organic and pyritic sulfur, the KVB process appears to have the lowest capital investment, since it is a partially dry process requiring lower investment for the dry reaction section. The high capital cost of the Battelle process is due to the processing steps associated with reagent regeneration.

The high capital cost of the ERDA process is due to costly equipment associated with the handling of dilute sulfuric acid at elevated temperatures and pressures. At the process operating conditions the dilute acid is highly corrosive and it poses problems in terms of selection of construction material for equipment and devices which are exposed to the corrosive atmosphere.

Very little is known about the ARCO process details and process chemistry. Therefore, a capital cost estimate was not developed for that process.

Operating Cost Comparisons

Table 62 presents a summary of operating cost elements for each process. The ranges of annual operating costs, including raw coal cost, in terms of \$/metric ton and \$/10⁶ kg cal are \$43.40 to \$72.40 and \$5.38 to \$11.20, respectively, Pyritic sulfur removal processes using chemical

pretreatment are the least expensive of all processes listed in Table 62. Operating cost for the Magnex process depends primarily on the cost of iron pentacarbonyl manufacturing. In the estimate presented in Table 61, an operating cost of \$0.22/kg for the iron carbonyl manufacturing was used, as projected by the developer. The current vendor quotes for iron carbonyl range up to \$3.30/kg. At a consumption rate of 10 kg/metric ton of coal, each \$0.20 cost increase per kilogram of iron carbonyl manufactured would increase the annual operating cost of this process by about 27 percent.

Between the two processes which remove pyritic sulfur by leaching, the TRW process appears to be slightly less costly. In the Ledgeмонт process the fixed charges associated with the higher capital investment have an adverse impact on the annual operating costs. Additionally, the TRW process has a much higher probability of technical success since it is currently active at a PDU stage. The Ledgeмонт process, tested only at a mini-pilot plant level, is currently inactive.

The most expensive processes, in terms of energy output, are the IGT process followed closely by the Battelle process. Laboratory data available at this time, indicate a very low BTU recovery for the IGT process. The Battelle process is adversely impacted by the fixed charges associated with the high capital investment and by the costs associated with chemicals consumption and reagent regeneration operations.

The least expensive process capable of removing pyritic and organic sulfur is the GE process followed closely by the JPL and KVB processes. The GE estimate is based, however, on early laboratory data and it is quite possible that the projected costs will prove somewhat inaccurate in the long run. The basic process utilizes a caustic reagent in coal pretreatment and the costs associated with caustic consumption and caustic regeneration are questionable at this time. The JPL process estimates are also preliminary since investigations on this process have been initiated recently. More definitive cost information on this process will be available in 1978

TABLE 62 OPERATING COST COMPARISONS FOR MAJOR CHEMICAL COAL CLEANING PROCESSES

COST ELEMENT	MEYERS PROCESS		LEDGEMONT PROCESS		MAGNEX ^{CS} PROCESS		SYRACUSE PROCESS		ERDA PROCESS		G.E. PROCESS		BATTELLE PROCESS		JPL PROCESS		IGT PROCESS		KYB PROCESS	
	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT	COST (\$1000)	\$/TON PRODUCT
LABOR & G. & A.	3,952	1.67	1,600	0.67	786	0.38	620	0.31	3,255	1.36	1,830	0.72	4,630	1.85	3,700	1.54	5,095	3.22	1,429	0.63
AMORTIZATION (20 YRS)	12,820	5.39	13,400	5.04	4,444	2.12	5,750	2.72	19,600	8.25	11,900	4.73	19,814	7.90	7,274	3.03	15,800	9.97	7,748	3.44
TAXES & INSURANCE	3,270	1.38	3,400	1.44	1,135	0.54	1,470	0.69	5,004	2.11	3,060	1.21	5,000	2.00	1,326	0.55	4,050	2.58	1,978	0.88
MAINTENANCE & SUPPLIES	5,460	2.29	7,300	3.07	1,891	0.90	3,170	1.50	8,340	3.51	5,310	2.10	10,000	3.99	2,210	0.92	6,732	4.24	3,297	1.46
UTILITIES	5,764	2.43	10,600	4.46	1,400	0.67	140	0.06	13,224	5.57	7,270	2.87	23,100	9.21	1,300	0.54	3,300	2.08	12,271	7.67
CHEMICALS	4,692	1.97	8,200	3.45	9,144	4.36	4,220	1.99	6,932	2.92	10,370	4.09	11,609	4.63	28,600	11.91	5,300	2.48	9,204	4.09
WASTE DISPOSAL	1,275	0.54	800	0.34	438	0.21	-	-	240	0.10	-	-	-	-	-	-	-	-	131	0.06
ANNUAL PROCESSING COST	37,243	15.67	45,300	19.07	19,238	9.18	12,190	5.77	56,595	23.82	39,820	15.72	74,213	29.53	44,410	18.44	38,277	24.17	41,059	18.23
RAW COAL	66,000	27.78	66,000	27.78	66,000	31.49	66,000	31.25	66,000	27.78	66,000	26.05	66,000	26.37	66,000	27.48	66,000	41.66	66,000	29.31
TOTAL ANNUAL COST	103,243	43.45	111,300	46.85	85,238	40.67	78,190	37.02	122,595	51.60	105,820	41.77	140,213	55.90	110,410	45.97	104,277	65.83	107,059	47.54

when more process information and accurate material and heat balance information becomes available. The annual costs reported for the KVB process are also preliminary since the process is at its early stages of development and accurate conceptualization of the process for purposes of economic evaluation is not possible at this time. The main advantage of the KVB process is the simplicity of the first stage dry oxidation process. If the dry oxidation process can be successfully demonstrated using coarse coals, this process would be an inexpensive technology for beneficiation of coals where partial removal of sulfur would substantially upgrade the coal.

Among the processes capable of removing pyritic and organic sulfur the ERDA process has one of the highest probabilities of technical success. The process is currently active and most technologies employed in this system have been already tested in other systems such as Ledgeмонт and TRW. The process is attractive because it is claimed to remove both types of sulfur and uses air as a major reagent. Furthermore, the sulfur by-product from this process is claimed to be dilute sulfuric acid, rather than iron sulfate, which greatly simplifies the coal washing operations. The process is somewhat expensive due to high operating temperature and pressure requirements and the corrosive nature of dilute acid present in this system. The dilute sulfuric acid at the operating conditions of the ERDA process will require the use of expensive construction material and consequently a higher capital investment cost.

Table 63 presents a cost effectiveness summary derived from information presented in Table 61. Costs are presented in terms of dollars per percent of sulfur removed from coal regardless of the quality of the treated product. However, column 7 of the table shows whether the product would comply with the current EPA's NSPS for SO₂ emissions. The processes are then rated based upon the cost effectiveness of sulfur removal. The subjective probability of success assigned to each process shown in column 8 of this table is based on integration of several factors such as:

TABLE 63. COST EFFECTIVENESS AND OTHER COMPARISONS OF CHEMICAL COAL CLEANING PROCESSES*

Process	Type of Sulfur Removed	Percent Sulfur in Product *	Percent Sulfur Removed (%)†	Process Cost (\$/metric ton incl. cost of coal ¢)	Cost Effectiveness of S removal, \$/% S removed	Cost Effectiveness Ranking	Meets EPA NSPS*	Probability of Success (based on available info.)	Time Frame for Commercial Availability (Years)α
Magnex Ⓢ	P ^Δ	.97	0.96	44.8	46.6	2	No	85%	2-3
Syracuse & Physical Cleaning	P	1.50 ^β	0.43	40.8	94.9	4	No	70%	2-3
TRW	P	.83	1.10	47.9	43.5	1	No	90%	<3
LOL [§]	P	.83	1.10	51.6	46.9	3	No	50%	4-5
ERDA	(P&O) [†]	.65	1.28	56.9	44.5	4	Yes	70%	5
GE	(P&O)	.50	1.43	46.0	32.2	1	Yes	60%	5
Battelle	(P&O)	.65	1.28	61.6	48.1	5	Yes	35%	4-5
JPL	(P&O)	.60	1.33	50.7	38.1	2	Yes	55%	5
IGT	(P&O)	.55	1.38	72.6	52.6	6	Yes	20%	5
KVB	(P&O)	.68	1.25	52.4	41.9	3	Yes	10% [§]	5
ARCO	(P&O)	.69	1.24	— ^Λ	— ^Λ	—	Yes	— ^Λ	— ^Λ

NOTES: * Based on Pittsburgh seam coal from Pennsylvania which contains 1.22 weight percent pyritic, 0.01 percent sulfate and 0.70 percent organic sulfur.

Δ P = pyritic sulfur.

† (P&O) = pyritic and organic sulfur.

α Time frame assumes continuing effort or renewed effort starting immediately.

Λ Information available is insufficient to make educated guesses.

§ Processes not currently active, partially accounting for low probability of success.

β 80 percent yield of product assumed in cleaning plant.

- available experimental data;
- our understanding of the status of the process;
- known product quality deficiencies;
- known process problems; and
- the degree and quality of effort assigned to the individual program.

In conclusion, all chemical coal cleaning processes discussed in this section offer a possibility of converting coal into clean fuel. Each process has an area of application. However, processes that remove both pyritic and organic sulfur will have a greater impact in coal utilization. If chemical coal cleaning is to be used as an approach for greater utilization of coal as an environmentally acceptable fuel, the pyritic and organic sulfur removal processes should be given the most emphasis and support.

SECTION 5

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SECTION 6

GLOSSARY

ash: The solid mineral residue left after incineration in the presence of oxygen.

autoclave: A chamber, usually of cylindrical shape, provided with a door or gate at one end which can be securely closed during operation. It is built heavily enough to accomodate pressures of considerable magnitude. It is used to effect chemical reactions requiring high temperature and pressure.

beneficiation: A process used to upgrade coal by removing unwanted impurities.

cladding material: A metal which is bonded to another metal by being rolled together at suitable pressure and temperature.

cyclone: A piece of equipment using centrifugal force to separate materials by size or density.

electrostatic precipitator: Consists of a source of high voltage current, an electrode system, an enclosure to provide a collection zone and a system for removing precipitated dust. Dust particle are electrically charged by means of ionization of the carrier gas and transported by the electric field to collecting electrodes. The particles are then neutralized on the collecting surfaces and removed for disposal.

endothermic: A process or change that takes place with absorption of heat and requires high temperature for initiation and maintenance.

exothermic: A process or chemical reaction which is accompanied by evolution of heat.

extractor: Any mechanical device or chemical substance which will allow the release of one substance from another.

filtrate: Liquid passing through a filter.

fluidized bed reactor: A reactor in which finely divided solids are caused to behave like fluids due to their suspension in a moving gas or liquid stream.

gas quench tower: A large tower or drum in which a cool liquid is used to lower the temperature of a hot gas.

incineration: The consumption of material by burning.

leaching: The process of extraction of a soluble component from a mixture with an insoluble component, by percolation of the mixture with a solvent.

magnetic fluid: A fluid which is appreciably attracted by a magnet.

magnetic separation: Removal of magnetic material from the coal as it passes through a magnetic field placed close to the stream of particles.

microwave: Any electromagnetic radiation having a wavelength in the approximate range from one millimeter to one meter.

mineral sulfur: Sulfurs that are inorganic in nature (sulfates and pyrites).

organic sulfur: Sulfur bound in an organic matrix.

pelletize: To form into a solid or densely packed ball or mass.

pyrite: Iron disulfide, FeS_2

pyrrhotite: Magnetic pyrites, FeS . A natural iron sulfide. Frequently has a deficiency in iron. May contain small amounts of nickel, cobalt, manganese and copper.

sensible heat: The perceptible or measurable effect of energy (heat) on a substance.

slurry: A watery suspension of solid materials.

APPENDIX I

TRW MEYERS' PROCESS

KINETIC EXPRESSIONS FOR PYRITIC SULFUR REMOVAL BY THE MEYERS' PROCESS

The survey program (EPA Contract No. 68-02-1647) provided adequate data which allowed formulation of expressions for pyritic sulfur removal. Using these data, for lower Kittanning coal, the sulfur removal is expressed by TRW as:

$$r_L = - \frac{dW_p}{dt} = K_L W_p^2 Y^2 = \text{wt of pyrite removed/100 wts of coal per hr.} \quad (1)$$

W_p = wt% pyrite in coal

Y = ferric iron-to-total iron weight ratio in leacher

$K_L = A_L \exp (- E_L/RT)$, A_L and E_L are constants for each coal and particle size at least over most of the reaction range.

This Kinetic equation (Equation 1) can be simplified by holding the reagent concentration relatively constant and thus can be expressed as

$$r_L = - \frac{dW_p}{dt} = K_o W_p^2 \quad (2)$$

where K_o is a function of temperature, reagent concentration, coal type, and particle size.

By integrating equation (2), the fraction of pyrite removed as a function of time can be shown as equation (3)

$$- \int_{W_F}^{W_F} \frac{dW_p}{W_p^2} = K_o \int_{t_o}^{t_F} dt$$

$$\int_{W_p}^{W_F} \frac{1}{W} = K_o t_F$$

$$\frac{1}{W_F} - \frac{1}{W_P} = K o t_F$$

$$\frac{W_P - W_F}{W_F W_P} = K o t_F$$

$$\frac{W_P - W_F}{W_F} = K o W_P t_F$$

where $W_P - W_F = F$, fraction of pyrite removed, Equation (2) can be expressed as

$$\frac{F}{1-F} = K o W_P t_F \quad (3)$$

Using this equation the removal of pyritic sulfur was measured as a fraction of time at 100°C for 18 Appalachian and 3 Eastern Interior region coals.⁴ The results are presented in Table I-1 which indicates that significant removal rate differences do exist between pyrite in various coals.

In Table I-1 the initial weight percent of pyritic sulfur Sp^o is substituted for W_P and equation (3) is rearranged to equation (4)

$$1/(Sp^o t_F) \propto K = \text{Actual rate constant} \quad (4)$$

Using this equation and assuming 80 percent sulfur removal as basis for comparison the values of $\frac{1}{Sp^o t_{80\%}}$ was calculated for the eighteen Appalachian coals. Since extensive engineering and experimental work has been performed with Martinka coal (as shown in column 7 of Table I-1) the $Sp^o t_{80\%}$ of this coal was set equal to one and was used as the basis of comparison. The reduced data on (column 7 of Table I-1 indicates that the Muskingum coal reacts much slower (by a factor of about 30) when compared to the Kopperston No. 2 coal.

TABLE I-1. MEYERS' PROCESS: RELATIVE RATE CONSTANTS FOR PYRITIC SULFUR REMOVAL

Coal Mine	Seam	Top Size, Microns	Sp ⁰	t _{80%} , hrs	Relative Rate Constants	
					$\frac{1}{Sp^0 t_{80\%}}$	Relative* Rate
Kopperston No. 2	Campbell Creek	149	0.47	2.0	1.1	16
Harris Nos. 1&2	Eagle & No.2 Gas	149	0.49	2.3	0.89	13
Marion	Upper Freeport	149	0.90	3.0	0.37	5.3
Lucas	Middle Kittanning	100	1.42	3.25	0.22	3.1
Shoemaker	Pittsburgh	149	2.19	2.9	0.16	2.3
Williams	Pittsburgh	100	2.23	3.0	0.15	2.1
Ken	No. 9	149	2.85	2.5	0.14	2.0
North River	Corona	149	1.42	5.0	0.14	2.0
Star	No. 9	100	2.66	3.0	0.13	1.9
Mathies	Pittsburgh	100	1.05	9.0	0.11	1.6
Prwhattan No. 4	Pittsburgh No. 8	75	2.75	4.0	0.091	1.3
Homestead	No. 11	149	3.11	3.5	0.092	1.3
Fox	Lower Kittanning	75	3.09	4.5	0.072	1.0
Isabella	Pittsburgh	149	1.07	13.0	0.072	1.0
Martinka	Lower Kittanning	149	1.42	10.0	0.070	1.0
Meigs	Clarion 4A	149	2.19	8.5	0.054	0.77
Bird No. 3	Lower Kittanning	100	2.87	8.0	0.044	0.62
Dean	Dean	100	2.62	10.2	0.037	0.53
Muskingum	Meigs Creek No. 9	149	3.65	8.0	0.034	0.49

NOTE: * $1/Sp^0 t_{80\%}$ relative to value for Martinka Mine.

MEYERS' PROCESS: CONCEPTUAL DESIGN FOR COMMERCIAL SCALE

Process engineering studies and trade-offs produced a baseline flow diagram for a commercial scale plant. The flow sheet, which is divided into its four major sections is given in Figure I-1. The corresponding mass balance and stream properties are given in Table I-2. The baseline plant size was chosen equal to 100 tons of dry coal feed per hour equivalent to about 250 MW power plant feed. This size is about the maximum size for a single train based on available commercial equipment.

Feed and Mixer--Crushed coal, nominally 14 mesh top size, is feed from feed hopper A-1. The coal is assumed to have 3.2 percent pyritic sulfur and 10 percent moisture on a dry basis; thus, the total solids feed rate is 110 tons per hour (TPH) at room temperature, assumed to be 77°F. The coal feed, stream 1, is brought to the mix tank, T-1, by conveyor, C-1, and introduced through the rotary feed valve, RV-1. Recycled leach solution, stream 4, at its boiling point (215°F) is introduced to the first mixer stage after first passing through the gas scrubber SP-1. Steam, streams 2 and 3, is needed to raise the feed coal from 77°F to the 215°F mixer temperature. Approximately 5.6 TPH of atmospheric pressure steam is required to heat the coal while 6.5 TPH is available from the flash drum, T-2. It is possible that the steam would actually be added to the enclosed conveyor to provide heated coal with an effective 15.6 percent moisture content. The excess 0.9 TPH would be vented through SP-1 along any flash steam formed in stream 4.

The mixer vessel T-1 was sized for three stages of mixing at 0.25 hours per stage. Under the design constraint that the vessel is 75 percent full, the cost model used for vessel sizing found a field fabricated vessel 18.7 feet in diameter by 32.9 feet long has minimum cost. The selected vessel size (18 x 36) gives three stages each about 12 feet long and 12.6 feet deep with slightly less than 15,000 gallons in each stage. Any foam generated during coal wetting will be broken down and the entrapped air will be scrubbed in SP-1 by the returning leach solution. The actual air flow through SP-1 is very low and will probably not exceed the air in the bulk coal (50 cubic feet per minute).

A-1	C-1	RV-1	T-1	M-1A/C	SP-1	T-2	R-1	M-2A/E	K-1	V-1	R-2
FEED	FEED	ROTARY	MIX	MIX	SCRUBBER-	FLASH	PRIMARY	PRIMARY	PRIMARY	KNOCK-OUT	SECONDARY
HOPPER	CONVEYOR	VALVE	TANK	TANK	MIST	DRUM	REACTOR	REACTOR	RECYCLE	DRUM	REACTOR
				MIXERS	ELIMINATOR			MIXERS	COMPRESSOR		

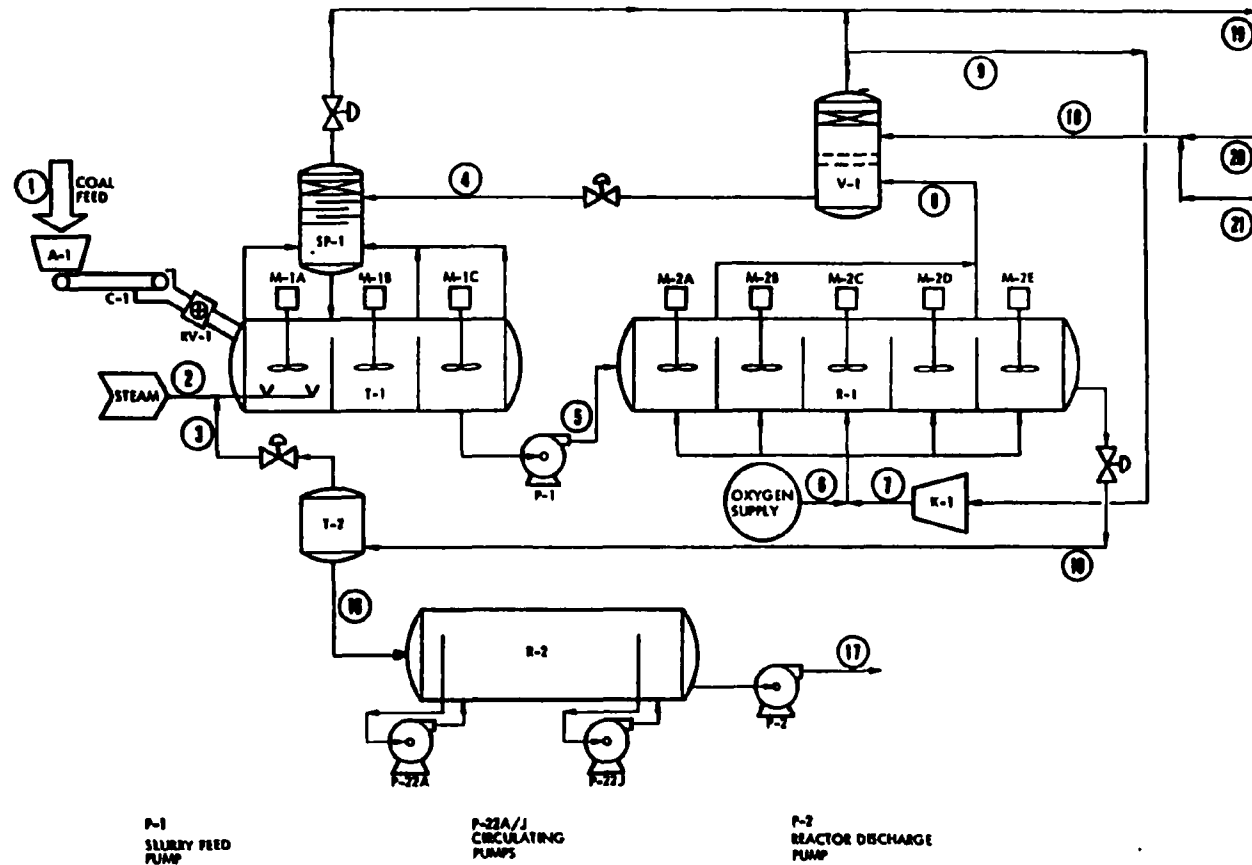


FIGURE 1-1 MEYERS' PROCESS FLOW SHEET FOR FINE COAL

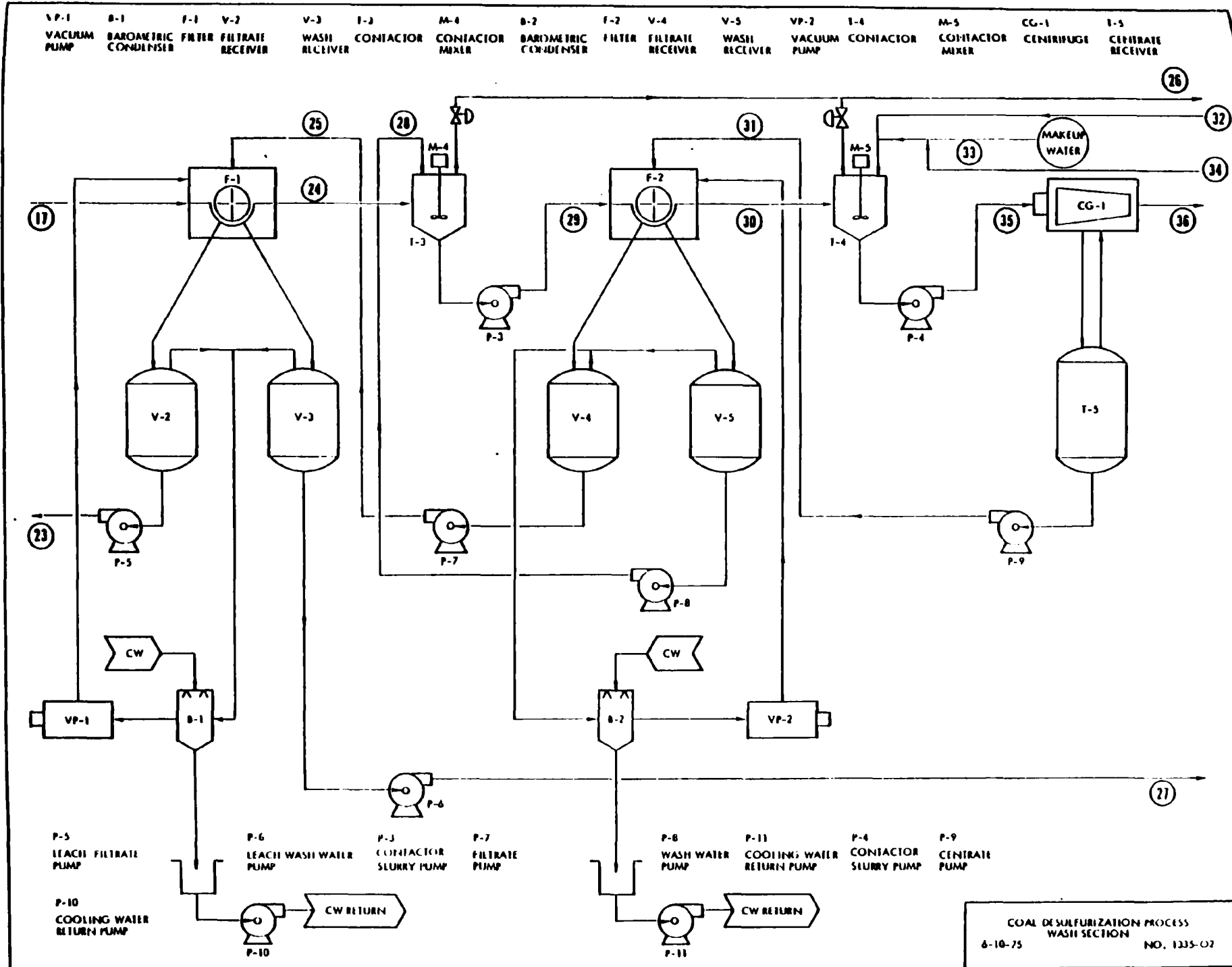


Figure I-1. (Continued)

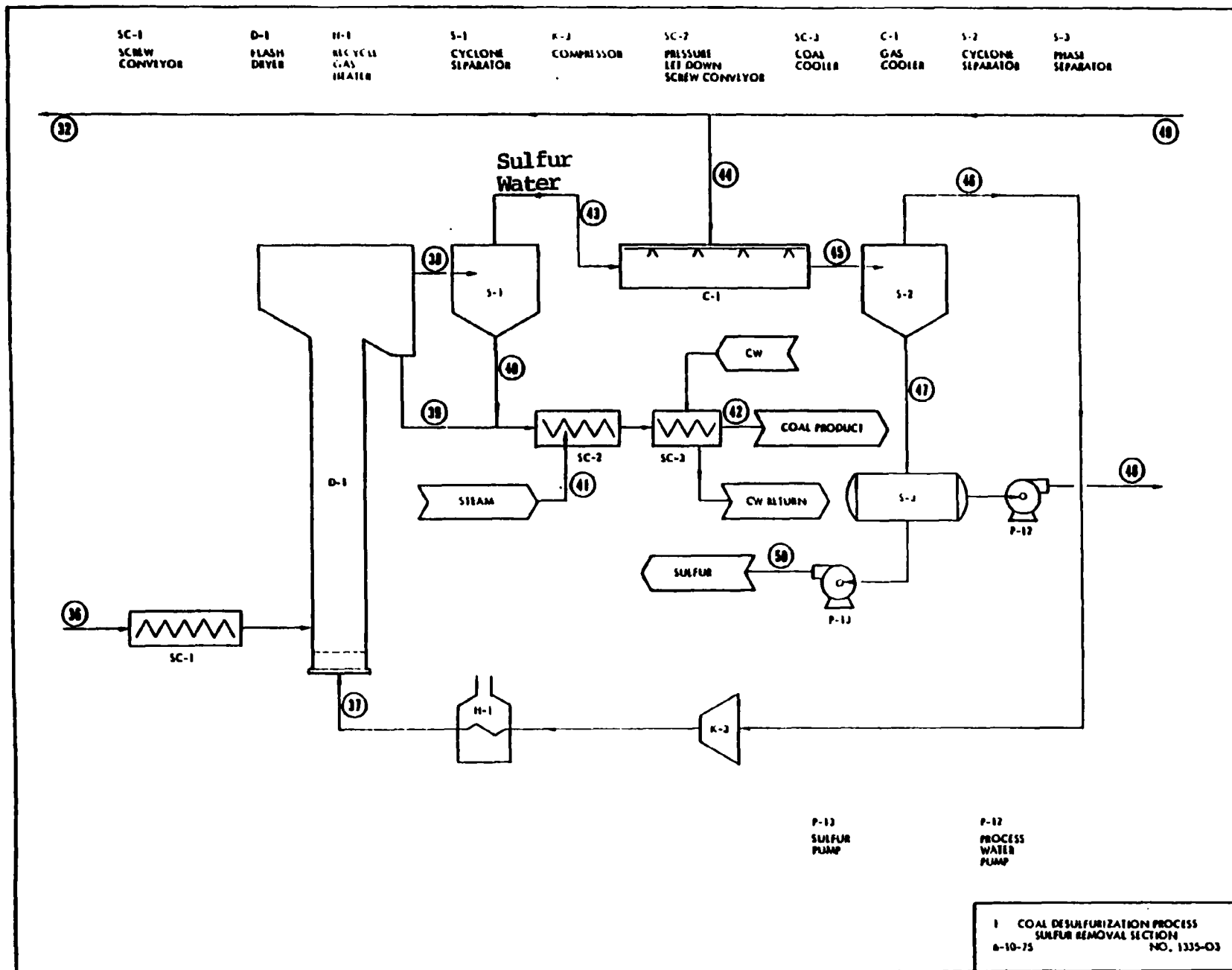


Figure I-1. (Continued)

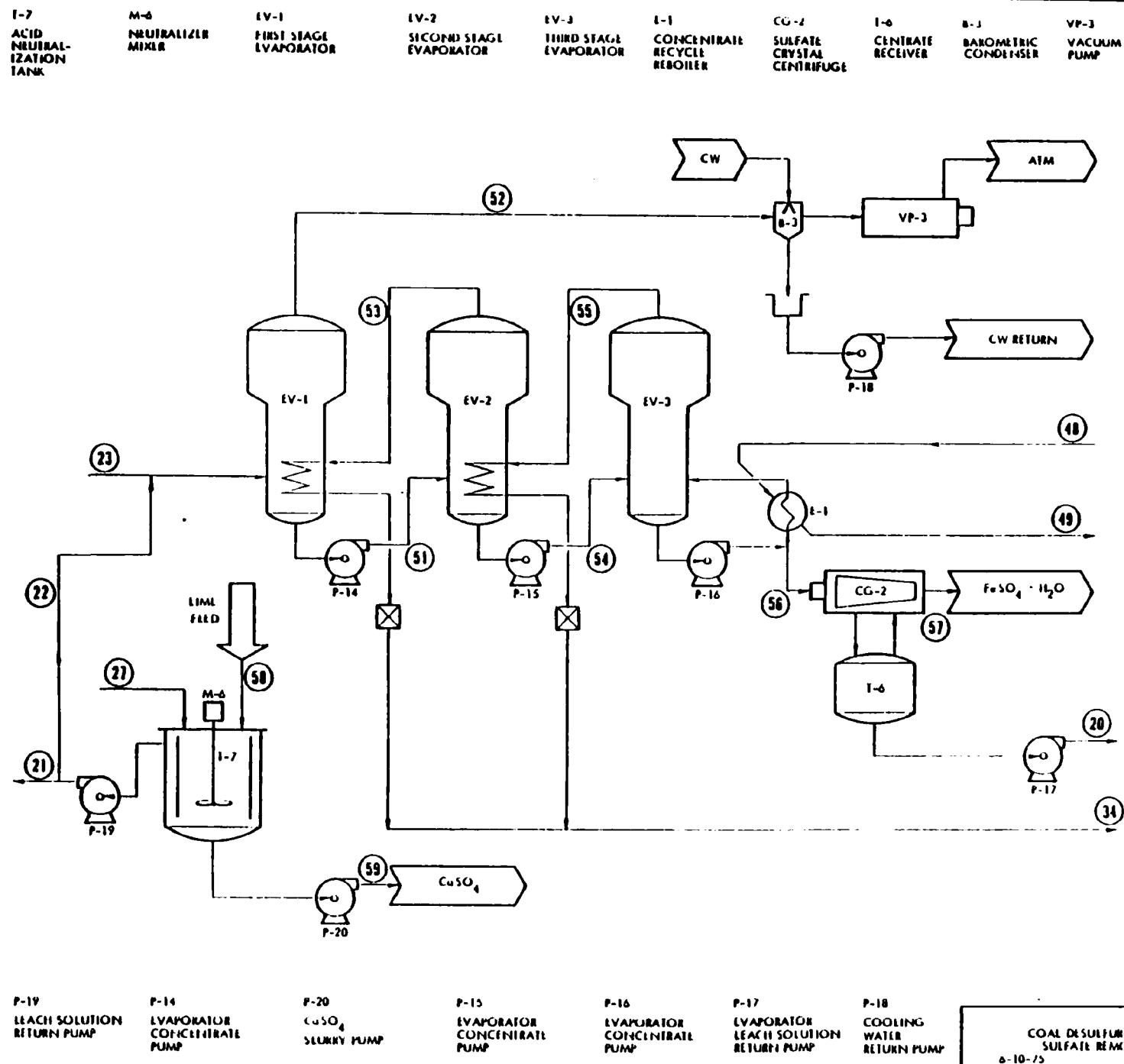


Figure I-1. (Continued)

TABLE I-2. MEYERS' PROCESS MASS BALANCE FOR FINE COAL
(Stream Flows in Tons Per Hour)

	COAL FEED	MAKEUP STEAM	FLASH STEAM	FEED SOLN.	R-1 FEED	O ₂ MAKEUP	RECYCLE GAS	R-1 GAS	COMPRESSOR FEED	R-1 EXIT	R-2 FEED	R-2 EXIT
	1	2	3	4	5	6	7	8	9	10	16	17
Water	10.0	-.9*	6.5	144.1	159.2		1.5	14.0	1.5	147.4	140.9	140.7
FeSO ₄				3.9	14.4					5.8	5.8	11.1
Fe ₂ (SO ₄) ₃				30.6	18.2					37.0	37.0	30.6
H ₂ SO ₄				5.7	8.8					5.0	5.0	6.6
Pyrite	6.0				5.2					.7	.7	.3
Sulfur					.2					1.1	1.1	1.2
Coal	94.0				94.0					94.0	94.0	94.0
Oxygen						3.9	13.1	13.4	13.1			
Inert						Tr	.8	.8	.8			
Total, TPH	110.0	-0.9	6.5	184.4	300.0	3.9	15.3	28.2	15.3	291.0	284.5	284.5
T, °F	77	215	215	215	215	77	264	250	177	250	215	215
P, Psig	0	.9	.9	0	28.8	53.8	53.8	28.8	28.8	28.8	0	0
gpm	-	-	-	614	968	-	-	-	-	907	873	874
ρ, lb/ft ³	50.0	-	-	74.8	77.3	-	-	-	-	80.0	81.3	81.2
Fe, %	-	-	-	5.4	5.2	-	-	-	-	6.3	6.6	6.7
Y	-	-	-	.86	.49	-	-	-	-	.83	.83	.68
SO ₄ /Fe	-	-	-	1.75	1.73	-	-	-	-	1.64	1.64	1.64

* Excess steam to vent

TABLE I-2. (CONTINUED)

	Return Soln.	Vent O ₂	Crystallizer Centrate	Neutralizer Return	Neutralizer to Crystallizer	Filtrate to Crystallizer	Cake F-1	Wash F-1	Contactor Vents	To Neutralizer	Cont actor Feed T-3	F-2 Feed Slurry
	18	19	20	21	22	23	24	25	26	27	28	29
Water	131.7	Tr	50.3	81.4	54.3	105.2	43.1	143.1	Tr	135.5	146.3	189.4
FeSO ₄	3.9		2.2	1.7	1.1	8.3	1.0	1.0		2.8	.3	1.3
Fe ₂ (SO ₄) ₃	30.6		26.0	4.6	3.1	22.9	3.0	3.0		7.7	.9	3.9
H ₂ SO ₄	5.7		5.2	.5	0.3	4.9	.6	.6		1.7	.2	.8
Pyrite							.3					.3
Sulfur							1.2					1.2
Coal							94.0					94.0
235 Oxygen		.4										
Inert		Tr										
Total, TPI:	171.9	0.4	83.7	88.2	58.8	141.3	143.2	147.7	0.0	147.7	147.7	290.9
T, °F	127	177	200	160	160	160	160	160	77	160	160	160
P, Psig	28.8	0	30.0	15.0	15.0	10.0	0	10.0	0	5.0	5.0	15.0
gpm	548	-	221	335	224	443	-	589	-	561	601	1065
ρ, lb/ft ³	78.2	-	95.4	65.6	65.6	79.6	-	62.5	-	65.7	61.3	68.1

TABLE I-2. (CONTINUED)

	<u>Cake F-2</u>	<u>Wash F-2</u>	<u>Dryer Water Return</u>	<u>Makeup Water</u>	<u>Evaporator Return</u>	<u>Centrifuge Feed Slurry</u>	<u>Cake Centrifuge</u>	<u>Dryer Gas</u>	<u>Dryer Output</u>	<u>Dryer Coarse Cut</u>	<u>Cyclone Solids</u>	<u>Purge Steam</u>	<u>Coal Product</u>	<u>Cyclone Gas</u>	<u>Cooler Water Feed</u>
	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44
Water	47.2	147.2	14.3	27.1	72.9	161.5	14.3	258.9	273.2	Tr	Tr	0.1	0.1	273.2	1137.4
FeSO ₄	.1	.1				.1	Tr		Tr	Tr	Tr		Tr		
Fe ₂ (SO ₄) ₃	.3	.3				.3	Tr		Tr	Tr	Tr		Tr		
H ₂ SO ₄	.1	.1				.1	Tr		Tr	Tr	Tr		Tr		
Pyrite	.3					.3	0.3		0.1	0.2	0.1		0.3		
Sulfur	1.2					1.2	1.2	Tr	1.2	Tr	Tr		Tr	1.2	
Coal	94.0					94.0	94.0	Tr	41.4	52.6	41.3		93.9	.1	
Oxygen															
Inert															
Total, TPH	143.2	147.7	14.3	27.1	72.9	257.5	109.8	258.9	315.9	52.8	41.4	0.1	94.3	274.5	1137.4
T, °F	160	160	215	77	180	180	180	650	450	450	450	300	150	450	215
P, Psig	0	10.0	30.0	30.0	30.0	15.0	0	20.0	18.0	18.0	18.0	18.0	0	17.8	30.0
gpm	-	607	60	108	473	942	-	-	-	-	-	-	-	-	4745
ρ, lb/ft ³	-	60.7	59.8	62.3	60.2	68.2	-	-	-	-	50.0	-	50.0	-	59.8

TABLE I-2. (CONTINUED)

	Cooler Effluent	Separator Gas Effluent	Separator Liquid	Crystallizer Reboiler Feed	Water Return	Sulfur Product	Liq. From EV-1	Steam to Vacuum	Steam From EV-2	Liq. From EV-2	Steam From EV-3	Centrifuge Feed	Sulfate Product	Lime	Calcium Sulfate
	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59
Water	1410.6	258.9	1151.7	1151.7	1151.7		124.1	35.4	35.2	88.9	37.7	51.2	0.9	0.4	
FeSO ₄							9.4			9.4		9.4	7.2		
Fe ₂ (SO ₄) ₃							26.0			26.0		26.0			
H ₂ SO ₄							5.2			5.2		5.2			
Pyrite															
Sulfur	1.2	Tr	1.2			1.2									
Coal	.1	Tr	.1			0.1									
Oxygen															
Inert															
Lime														0.5	
Gypsum															1.6
Total, TPH	1411.9	258.9	1153.0	1151.7	1151.7	1.3	164.7	35.4	35.2	129.5	37.7	91.8	8.1	0.9	1.6
T, °F	250	250	250	250	215	215	120	115	150	155	207	210	200	77	77
P, Psig	15.0	15.0	15.0	40.0	30.0	25.0	5.0	(1.5 P _{sia})	(3.7 P _{sia})	5.0	(13 P _{sia})	5.0	0	0	5.0
gpm	-	-	4851	4851	4802	2.9	516			376		230	-	-	2.8
ρ, lb/ft ³	-	-	59.2	59.2	59.8	112.0	79.6	-	-	85.9	-	99.7	-	-	144.8

Primary Reactor—The fully wetted and deaerated coal slurry from the mixer is pumped by slurry pump P-1 (stream 5) into the first stage of the primary reactor R-1. Both removal of pyrite and oxidation of ferrous to ferric iron sulfate occur in this reactor. A five stage reactor was selected since the cost model showed the minimum cost field fabricated vessel had length to diameter ratios near five. Under the design constraint that the reactor must have five stages and operates about 85 percent full, the cost model found a reactor 25.9 feet in diameter by 127.7 feet long operated at 15 psi of oxygen was minimum cost. The selected vessel size (26 x 125) gives five stages each about 25 feet long by 23 feet deep and holding about 80,000 gallons of slurry. At the residence time of 1.5 hours per stage, a temperature of 250°F and an oxygen partial pressure of 15 psi, the pyrite is reduced to 88 percent of the original level and the leach solution is regenerated to a Y (ferric iron to total iron ratio) of 0.83 in the primary reactor.

Oxygen Loop—Excess oxygen saturated with steam and containing an equilibrium level of inert gas (mainly argon) leaves the primary reactor in stream 8. The gas is contacted with returning leach solution, stream 18, in a knock-out drum, vessel V-1. The leach solution is warmed to 215°F (stream 4) by condensing steam from the oxygen stream. The gaseous effluent, which was assumed to leave V-1 50°F warmer than the feed leach solution is split to give a small vent stream 19 and a recycle oxygen stream 9. The vent rate is selected to maintain the inert gas at the design level; namely 5 percent on a dry basis. The recycle oxygen is compressed by K-1 to the reactor feed pressure. Makeup oxygen, stream 6, is added to balance the oxygen used for regeneration in R-1 and that vented to remove inerts.

Assuming 15 psia oxygen pressure the gas pressures in reactor R-1 at 250°F are as follows:

Oxygen	15.0 psia
Inert Gas	.8 psia
Steam	<u>27.7 psia</u>
	43.5 psia (28.8 psig)

Since the recycle gas must also overcome the liquid head in the reactor (about 13 psi), the control valve/injector drop (about 10 psi) and other line losses, the recycle compressor was sized to provide a 25 psi pressure increase. For the baseline case this results in a 300 horsepower compressor operating at a 1.58 compression ratio and a compressor outlet pressure of 53.8 psig.

Flash Steam—The heat of reaction and regeneration is accommodated in three ways: temperatures of the recycled oxygen and the feed slurry are raised in R-1, heat is lost from the insulated walls of the mixer and reactors, and water is evaporated from the solutions. Part of the steam (13.4 TPH) is removed from the recycle oxygen to provide an isothermal primary reactor R-1 at 250°F and part of the steam (6.5 TPH) is removed by flash drum T-2 in dropping and slurry temperature and pressure from reactor R-1 (250°F) to reactor R-2 (215°F). The heat is almost entirely utilized in heating the feed coal and the recycle leach solution.

Secondary Reactor—The secondary reactor, R-2, is operated near the atmospheric boiling point with a residence time of 36 hours. During this time, additional pyrite is removed from the coal to provide an overall pyrite removal of 95 percent while the Y of the solution is decreased to a value of 0.68 in the reactor effluent. The low value of Y is desired to provide sufficient ferrous sulfate for removal as the by-product iron form. The cost model found the minimum cost reactor was 27.9 feet in diameter by 465.9 feet long. The final equipment list and costing used three field fabricated vessels each 28 feet in diameter and 160 feet long. The reactors contain no internal stages, but have circulating pumps to avoid large vertical concentration gradients from occurring in the solution. The slurry from the secondary reactor, stream 17, is pumped by P-2 to the first filter, F-1.

Coal Washing—Bench-scale experience with removal of the sulfate leach solution from coal shows that the solution may be treated as consisting of two types. Surface solution is readily removed by flushing with water or may be readily displaced by a more dilute wash solution. Solution in the pores of the coal particles requires a definite residence time to reach

equilibrium with the bulk or surface liquid. The coal washing section, therefore, consists of filtration, washing on the filter, equilibration with dilute solution, a second filtration and wash, equilibration with wash water and finally dewatering in a centrifuge.

First Filter--Coal slurry from the secondary reactor, stream 17, containing approximately 33 percent solids is fed to a 12 foot diameter by 24 foot long rotary vacuum filter, F-1. The filtrate from vacuum receiver V-2, stream 23, is pumped, P-5, to the sulfate removal section. Dilute wash solution from the second filter, stream 25, is used to wash the filter cake and displace the surface solution on the coal particles. This sulfate rich wash solution, stream 27, is pumped, P-6, from the vacuum receiver V-3 to the sulfate removal section. Vacuum is provided by a 3,000 standard cubic feet per minute (SCFM) vacuum pump, VP-1, which is vented, stream 26, back to the enclosed filter F-1. The vapors and gases removed from the vacuum receivers, V-2 and V-3, pass through a barometric condenser, B-1, before entering the vacuum pump. In B-1 most of the flash steam is condensed and enters the cooling water loop where it is pumped to the cooling water tower by P-10.

First Stage Repulping--The washed filter cake from the first filter, stream 24, and dilute wash water from the second filter are gravity fed through a closed chute to a stirred tank, T-3. This 40,000 gallon tank is operated about three-fourths full to give an average residence time of 30 minutes to equilibrate pore solution with the bulk liquid. The slurry, stream 29, is pumped, P-3, to the second stage filter. Any gases introduced with the cake are vented to the scrubbing system, stream 26.

Second Filter--The partially washed slurry, stream 29, containing approximately 33 percent solids, is filtered and washed on a second filter of the same size and type as the first filter. Filtrate, stream 25, is pumped, P-7, from the vacuum receiver, V-4, to the first filter wash. Wash water for the second filter, stream 31, is obtained from the centrate receiver. The partially spent wash water is pumped, P-8, from the vacuum receiver V-5 to the first stage contractor. Vacuum is provided by vacuum pump VP-2 operating through the barometric condenser B-2.

Second Stage Repulping--The washed filter cake from the second filter, stream 30, is contacted with water in a 40,000 gallon stirred tank, T-4. The wash water, streams 32, 34, and 33 is obtained from the dryer, the evaporators, and makeup, respectively.

Dewatering--The slurry from the second contractor, stream 35, is pumped P-4, to the dewatering centrifuge, CG-1. The slurry with approximately 33 percent solids is separated in the 36 inch diameter by 90 inch long solid bowl centrifuge to provide a dewatered coal is expected to have about 15 percent moisture. The centrate from receiver T-5 is pumped, P-9, to provide the wash, stream 31, for the second filter.

Drying--Coal from the centrifuge, stream 36, is fed to a flash dryer, D-1, by a screw feeder, SC-1. In this dryer concept the coal is heated to about 450°F by superheated steam, stream 37, and carried upward to the enlarged top area of the dryer. The larger particles are removed from the dryer, stream 39, while the fine particles and gas, stream 38, are fed to a cyclone, S-1. During the drying in D-1 sulfur is also vaporized from the coal and is present along with water vapor in the cyclone effluent gas stream 43. The fine coal from the cyclone, stream 40, and coarse coal, stream 39, are let down to atmospheric pressure by screw conveyor SC-2 which is back purged with a small quantity of steam to prevent the sulfur containing gas in the cyclone from leaving the system with the coal. The coal, stream 42, is then transported and cooled to product storage temperature by the screw conveyor, SC-3 which rejects heat either to cooling water or to the atmosphere.

Sulfur Removal--The cyclone effluent gas, stream 43, at about 450°F is cooled by a large spray of water, stream 44, in gas cooler C-1. The water is obtained from return stream 49 from the sulfate removal section. The gas and liquid, stream 45, cooled to 250°F is separated in cyclone S-2 to give vapor stream 46 and liquid stream 47. The liquid stream 47 contains the water fed to the gas cooler, stream 44, the water vaporized from the coal in the dryer, and the sulfur vaporized from the coal. The liquid is phase separated in vessel S-3. The liquid sulfur by-product, stream 50, is pumped, P-13, to storage while the hot water, stream 48, is pumped, P-12, to the sulfate removal section.

Steam Circulation—Saturated steam at 250°F from the cyclone, stream 46, is compressed by K-3, reheated by H-1, and fed to the dryer as stream 37. Compression is accomplished by two 3500 HP series compressors which make up the 10 psi pressure drop around the gas circulation loop. The heater provides nearly 100 million BTU per hour (MM BTU/hr) to the steam to supply the heat required to heat the dryer feed, stream 36, to 450°F and vaporize the water and sulfur. Slightly more than 80 MM BTU/hr are rejected to the hot water loop, stream 48, for use in the sulfate removal section while about 15 MM BTU/hr are lost from the equipment and lines or rejected as sensible heat in the hot coal and liquid sulfur. The circulating water is kept in balance by returning a portion of the water, stream 32, to the wash section equal to the water vaporized from the feed coal, stream 36.

Neutralization—Sulfate rich wash solution from the wash section, stream 27, is fed to a stirred tank, T-7, and a lime slurry, stream 58, is added to neutralize part of the sulfuric acid. The tank is sized for about 10 minutes of residence time and has a baffled settling zone. Gypsum slurry stream 59 is withdrawn for disposal and the partially neutralized liquid is removed by pump P-19. A portion of the liquid, stream 21, is returned to the reactor section while the remainder, stream 22, is combined with the filtrate, stream 23, as feed to the triple effect evaporators.

Evaporation—Evaporator EV-1 is operated at partial vacuum (about 0.1 atmospheres) and uses condensing steam from the second evaporator, stream 53, to evaporate water, stream 52, in the first evaporator. The evaporated water is condensed in the barometric condenser, B-3, and any residual gas is removed by vacuum pump VP-3. The partially concentrated leach solution, stream 51, is pumped, P-14, to the second evaporator, EV-2. The second evaporator operates at about 155°F and 0.2 atmospheres using steam from the third evaporator, stream 55, to evaporate the water, stream 53. The two condensate streams from the reboilers of the first and second evaporators (streams 53 and 55) are combined, stream 34, to provide clean wash water for the wash section. The leach solution from the second evaporator, stream 54, which has been concentrated to 8.3 percent iron, is at a temperature where the solubility of ferrous sulfate is a maximum and is a solids free solution. This stream is feed to the third evaporator, EV-3,

which is operated at atmospheric pressure and at the normal boiling point of the solution. Heat to vaporize water is provided to the reboiler, E-1, by the hot water loop from the wash section (streams 48 and 49). The overhead steam, stream 55, is used in the second evaporator as previously described. The leach solution in EV-3 is concentrated to a total iron concentration of nearly 12 percent which exceeds the solubility of ferrous sulfate. Thus, crystalline ferrous sulfate forms in EV-3 and a portion of the slurry, stream 56, is fed to a centrifuge CG-2 to separate the crystals, stream 57, from the concentrated leach solution, stream 20. The concentrated leach solution is pumped, P-17, to the reactor section.

Solubilities--Since the solubility of ferrous sulfate in the presence of ferric sulfate, sulfuric acid and trace ions is not yet fully defined, the baseline process flows may require some adjustment when pilot scale data have been evaluated. Nevertheless, the planned mode of operation which takes advantage of the reported solubility characteristics of ferrous sulfate in aqueous solution should be applicable. Below about 150°F, the equilibrium crystalline phase is $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ which has an increasing solubility with temperature. It reaches a maximum solubility of nearly 60 grams of FeSO_4 (anhydrous basis) per 100 grams of water. Above about 150°F the equilibrium solid phase is $\text{FeSO}_4 \cdot \text{H}_2\text{O}$ which has a decreasing solubility in water with increasing temperature. Both the first and second stages of evaporation are below the saturation limits and are expected to remain solids free. Only the final stage operates as a crystallizer and produces crystalline ferrous sulfate both from a decreased solubility at the higher temperature and from an increased concentration because of evaporation.

TABLE I-3. MEYERS' PROCESS COAL DESULFURIZATION PROCESS EQUIPMENT LIST

REACTOR SECTION \$3.26 MM FOB, \$6.36 MM INSTALLED*

1	A-1	Ground Coal Feed Hopper - 5,000 ft ³
2	C-1	Feed Conveyor - 20 in Wide x 20 ft, 5 hp, 200 ft/min
3	K-1	Oxygen Recycle Compressor - 300 hp, 1.6 Compression Ratio
4	M-1A/C	Mix Tank Mixers (3) - 15 hp, Stainless Steel (SS)
5	M-2A/E	Primary Reactor Mixers (5) - 200 hp, SS
6	P-1	Slurry Feed Pump - 1,000 gpm, 60 psi, 50 hp, SS
7	P-2	Reactor Discharge Pump - 875 gpm, 5 psi, 3.5 hp, SS
8	P-22A/J	Circulation Pumps (12) - 1,000 gpm, 5 psi, 4.0 hp, SS
9	R-1	Primary Reactor - 26 ft 0 x 125 ft, Carbon Steel (CS) with SS clad, 30 psig
10	R-2	Secondary Reactor (3) - 28 ft 0 x 165 ft, SS, 0 psig
11	RV-1	Rotary Valve - .5 hp, 18 in x 18 in , 20 RPM
12	SP-1	Scrubber-Mist Eliminator - 3 ft 0 x 10 ft, SS, 0 psig, Baffles, Demister Pad
13	T-1	Mix Tank - 18 ft 0 x 36 ft, SS, 0 psig
14	T-2	Flash Drum - 5 ft 0 x 10 ft, SS, 5 psig
15	V-1	Knock-Out Drum - 5 ft 0 x 25 ft, SS, 30 psig, 15 ft Packing, Demister Pad

*Installed costs for each process section were derived through the application of the appropriate Guthrie factor⁶ to the FOB cost of individual pieces of equipment.

TABLE I-3. (CONTINUED)

WASH SECTION \$1.16 MM FOB, \$2.28 MM INSTALLED

1	B-1	Barometric Condenser - SS, Condensation Rate = 13 ton/hr
2	B-2	Barometric Condenser - SS, Condensation Rate = 2.5 ton/hr
3	CG-1	Centrifuge (4) - 36 in 0 x 90 in Solid Bowl, SS, 150 hp
4	F-1	Rotary Vacuum Filter - 12 ft 0 x 24 ft Drum, 912 ft ² , SS, 8 hp
5	F-2	Rotary Vacuum Filter - 12 ft 0 x 24 ft Drum, 912 ft ² , SS, 8 hp
6	M-4	Contacto Mixer, 35 hp, SS
7	M-5	Contacto Mixer, 35 hp, SS
8	P-3	Contacto Slurry Pump - 1,065 gpm, 15 psi, 15 hp, SS
9	P-4	Contacto Slurry Pump - 950 gpm, 15 psi, 10 hp, SS
10	P-5	Leach Filtrate Pump - 450 gpm, 10 psi, 3.5 hp, SS
11	P-6	Leach Wash Water Pump - 560 gpm, 5 psi, 2.5 hp, SS
12	P-7	Filtrate Pump - 590 gpm, 10 psi, 5 hp, SS
13	P-8	Wash Water Pump - 560 gpm, 5 psi, 2.5 hp, SS
14	P-9	Centrate Pump (4) - 150 gpm, 10 psi, 1 hp, SS
15	P-10	Cooling Water Return Pump - 1,200 gpm, 5 psi, 5 hp
16	P-11	Cooling Water Return Pump - 200 gpm, 5 psi, 1 hp, CS
17	T-3	Contacto - 40,000 gal, 0 psig, SS
18	T-4	Contacto - 40,000 gal, 0 psig, SS
19	T-5	Centrate Receiver (4) - 650 gal, 0 psig, SS
20	V-2	Filtrate Receiver - 2,000 gal, Vac, SS
21	V-3	Wash Receiver - 2,500 gal, Vac, SS
22	V-4	Filtrate Receiver - 2,500 gal, Vac, SS
23	V-5	Wash Receiver - 2,500 gal, Vac, SS
24	VP-1	Vacuum Pump - 3,000 SCFM, 200 hp, CS
25	VP-2	Vacuum Pump - 3,000 SCFM, 200 hp, CS

TABLE I-3. (CONTINUED)

SULFUR REMOVAL SECTION \$1.42 MM FOB, \$2.94 MM INSTALLED

1	C-1	Gas Cooler - 7 ft 0 x 100 ft, Water Sprays, SS, 15 psig
2	D-1	Flash Dryer - 11 ft 0 x 65 ft Drying Section, 22 ft 0 x 20 ft De-entrainment Section, SS, 20 psig
3	H-1	Recycle Gas Heater - 97 MM Btu/Hr, Radiant Section = 6,000 ft ² , Convective Section = 12,000 ft ² , SS Tubes
4	K-3	Compressor (2) - 1.15 Compression Ratio, 3,500 hp
5	P-12	Process Water Pump - 4,850 gpm, 40 psi, 150 hp, CS
6	P-13	Sulfur Pump - 3 gpm, 25 psi, 0.5 hp, SS
7	S-1	Cyclone Separator - SS, 15 psig, 120,000 ACFM Capacity
8	S-2	Cyclone Separator - SS, 15 psig, 107,000 ACFM Capacity
9	S-3	Phase Separator - 50,000 gal, 15 psig, SS
10	SC-1	Screw Conveyor - 20 ft x 14 in 0, 2 hp, SS
11	SC-2	Pressure Let Down Screw Conveyor - 20 ft x 14 in 0, 2 hp, CS
12	SC-3	Coal Cooler - Screw Type, 20 ft x 14 in 0, Cooled Shell, CS, 2 hp

TABLE I-3. (CONTINUED)

SULFATE REMOVAL SECTION \$0.97 MM FOB, \$1.68 MM INSTALLED

247	1	B-3	Barometric Condenser - SS, Condensation Rate = 35.4 ton/hr
	2	CG-2	Sulfate Crystal Centrifuge - 36 in 0 x 72 in Solid Bowl, SS, 125 hp
	3	E-1	Concentrate Recycle Reboiler - 10,000 ft ² , SS/SS
	4	EV-1	First Stage Evaporator - Evaporation Rate = 35 ton/hr, 1.5 psia, SS
	5	EV-2	Second Stage Evaporator - Evaporation Rate = 35 ton/hr, 3.7 psia, SS
	6	EV-3	Third Stage Evaporator - Evaporation Rate = 38 ton/hr, 13 psia, SS
	7	M-6	Neutralizer Mixer - 5 hp, SS
	8	P-14	Evaporator Concentrate Pump - 520 gpm, 5 psi, 2.0 hp, SS
	9	P-15	Evaporator Concentrate Pump - 380 gpm, 5 psi, 1.5 hp, SS
	10	P-16	Evaporator Concentrate Pump - 1,380 gpm, 5 psi, 5.0 hp, SS
	11	P-17	Leach Solution Return Pump - 220 gpm, 30 psi, 5.0 hp, SS
	12	P-18	Cooling Water Return Pump - 8,000 gpm, 5 psi, 30 hp, CS
	13	P-19	Leach Solution Return Pump - 560 gpm, 30 psi, 10 hp, SS
	14	P-20	Calcium Sulfate Slurry Pump - 3 gpm, 5 psi, 0.5 hp, SS
	15	T-6	Centrate Receiver - 900 gal, SS, 0 psig
	16	T-7	Neutralizer Tank - 7,500 gal, SS, 0 psig
	17	VP-3	Vacuum Pump - 700 CFM, 50 hp

TOTAL ESTIMATED CAPITAL \$6.81 MM FOB, \$13.26 MM INSTALLED

APPENDIX II

LEDGEMONT PROCESS

TABLE II-1 REPRESENTATIVE ANALYSES OF ILLINOIS
#6 AND KENTUCKY COALS

<u>Coal Source</u>	<u>Illinois #6 (ROM Coal)</u>	<u>Kentucky (Washed Coal)</u>
Ultimate Analyses (dry basis):		
Carbon	62.78	71.4
Hydrogen	4.31	5.08
Nitrogen	1.03	1.68
Sulfur (total)	4.85	3.55
Sulfur (pyritic)	2.0	N/A
Oxygen	7.13	9.67
Ash	19.90	8.00
Heating value kg cal/kg (BTU/lb)	6,180 (11,140)	7,395 (13,325)
Moisture content (ROM)	13.88	9.32

APPENDIX III

MAGNEX PROCESS

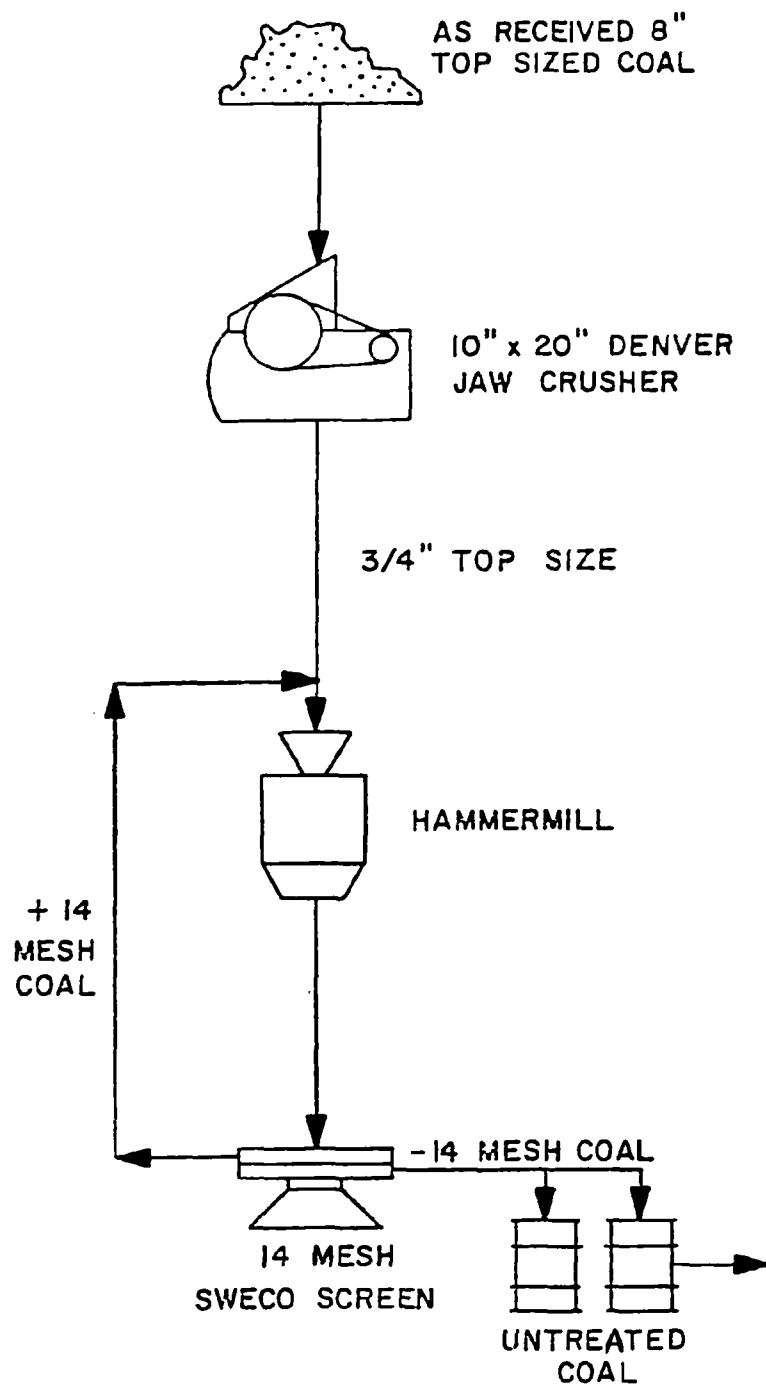


FIGURE III - 1 MAGNEX PILOT PLANT SCHEMATIC, CRUSHING STEP

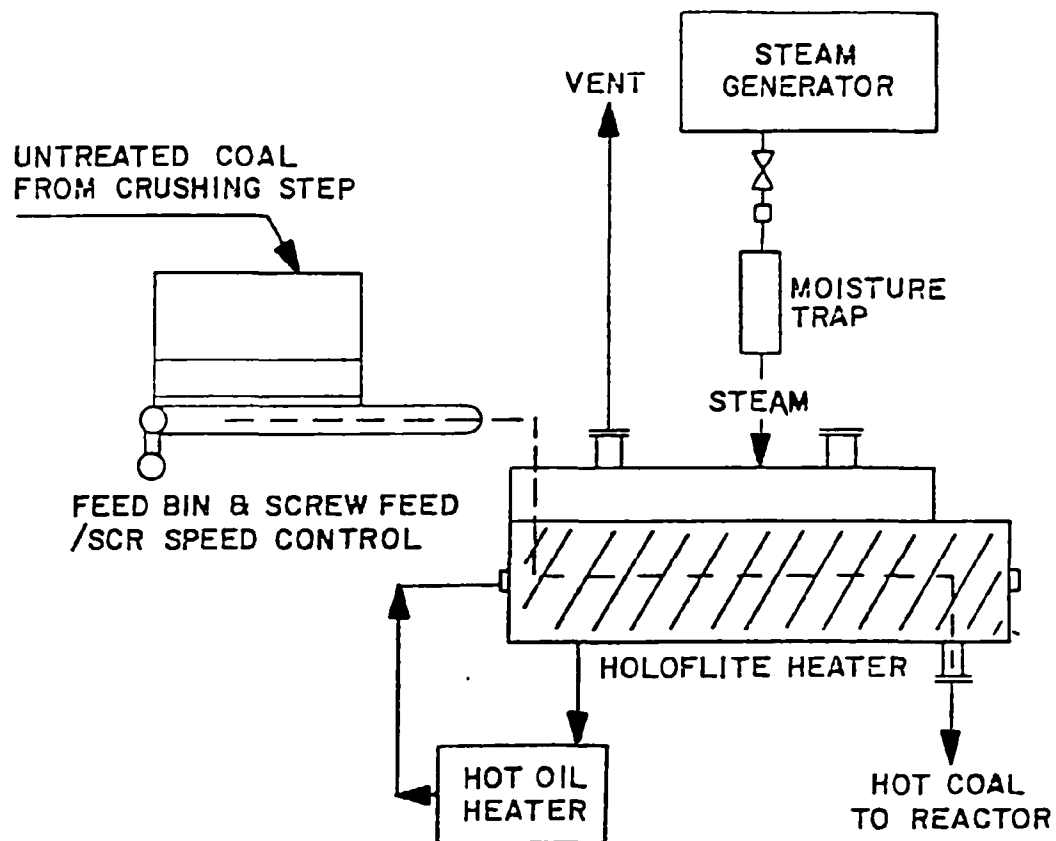


FIGURE III - 2 MAGNEX PILOT PLANT SCHEMATIC, HEATING STEP

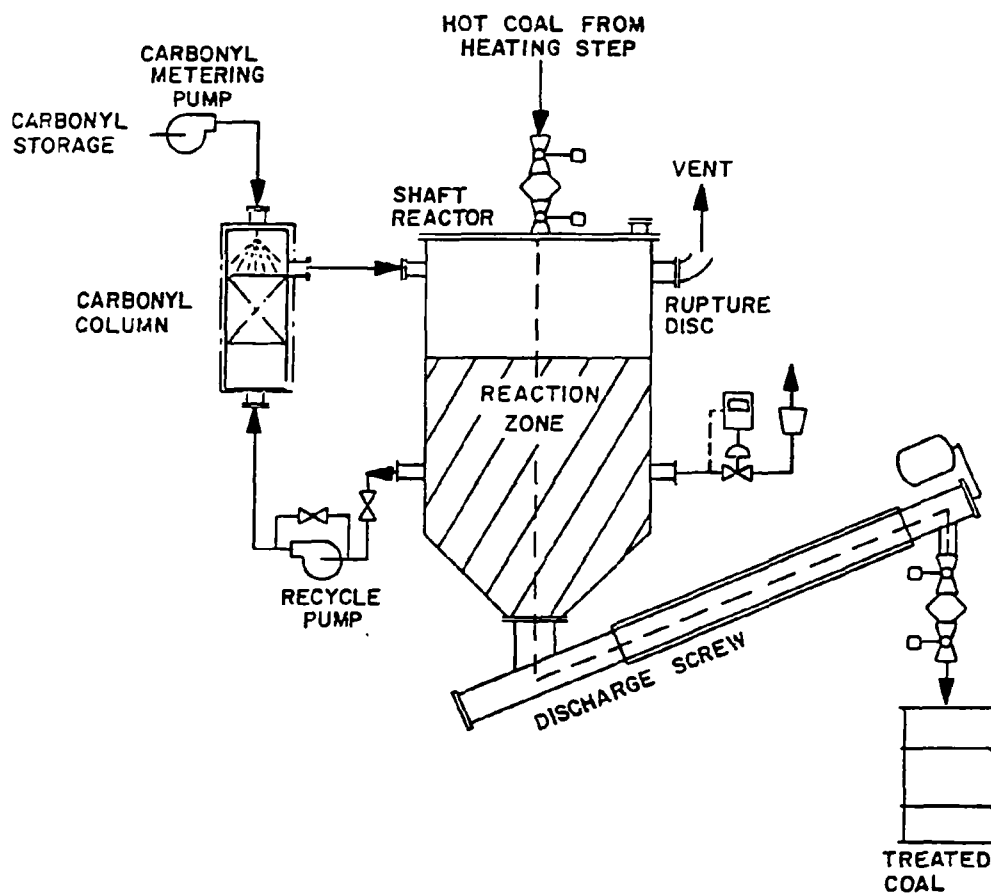


FIGURE III-3 MAGNEX PILOT PLANT SCHEMATIC, CARBONYL TREATMENT STEP

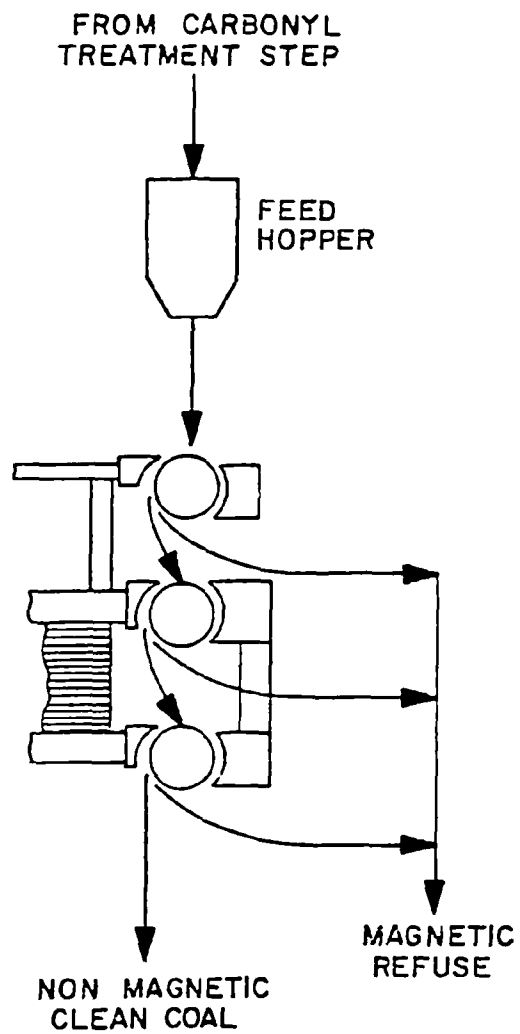


FIGURE III -4 MAGNEX PILOT PLANT SCHEMATIC, MAGNETIC SEPARATION STEP

APPENDIX IV

SYRACUSE PROCESS

TABLE IV-1 ANALYSES OF UPPER FREEPORT SEAM, PITTSBURGH SEAM, BLENDED (UPPER AND LOWER FREEPORT SEAM) COAL, AND ILLINOIS NUMBER 6 SEAM COAL SAMPLES.¹⁸

	Weight Percent (except BTU)							
	Upper Freeport Seam Coal ^{*Δ}		Pittsburgh Seam Coal ^{*Φ}		Blended (Upper Freeport and and Lower Freeport Seam) Coal Sample		Illinois Number 6 Seam Coal [*]	
	Air Dried	Dry	Air Dried	Dry	Air Dried	Dry	Air Dried	Dry
Moisture	1.01	xx	1.06	xx	0.97	xx	5.29	xx
Volatile	26.33	26.60	37.21	37.61	24.43	24.67	33.77	35.58
Ash	17.98	18.16	10.81	10.93	21.91	22.12	11.86	12.52
Fixed C	55.52	55.99	50.55	51.12	52.69	53.21	49.15	51.90
Sulfur	2.76	2.79	3.77	3.81	2.71	2.74	2.02	2.13
British Thermal Units per pound	12397	12523	13369	13512	11784	11899	11913	12579
Sulphate sulfur	0.15	0.15	0.05	0.05	0.07	0.07	0.07	0.07
Pyritic sulfur	2.20	2.22	2.09	2.11	2.11	2.13	1.04	1.10
Organic sulfur	0.41	0.42	1.63	1.65	0.53	0.54	0.91	0.96
Carbon	69.61	70.32	73.09	73.87	65.49	66.13	66.31	70.01
Hydrogen	4.27	4.31	4.94	4.99	4.07	4.11	4.45	4.70
Nitrogen	0.99	1.00	1.30	1.31	1.20	1.21	1.44	1.52
Oxygen	3.38	3.42	5.01	5.06	3.65	3.69	8.63	9.12

* Coal was air dried.

Δ R.O.M. coal as received from mine had 5.37% moisture.

Φ R.O.M. coal as received from mine had 2.89% moisture.

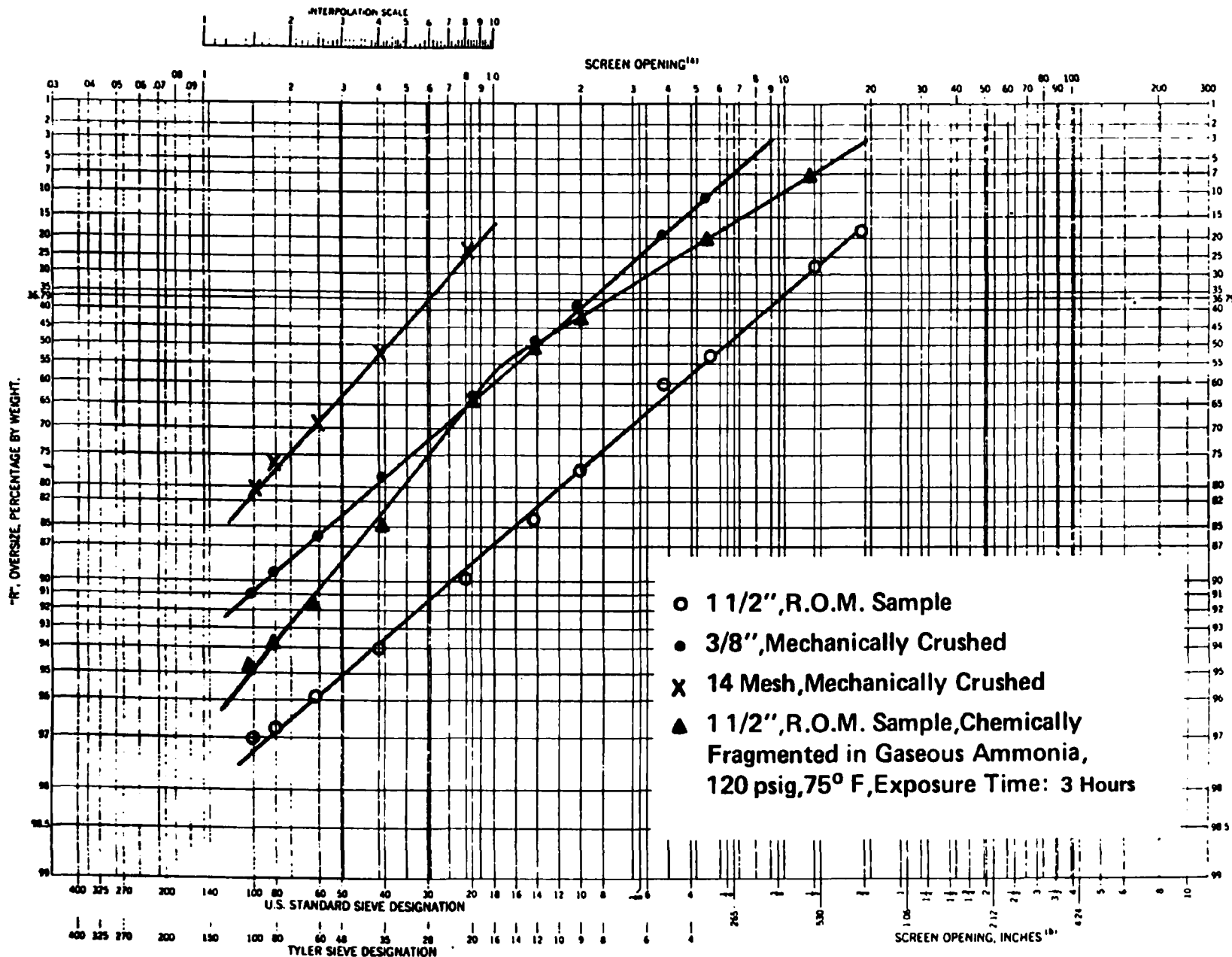


FIGURE IV - 1 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: SIZE CONSIST OF UPPER FREEPORT COAL

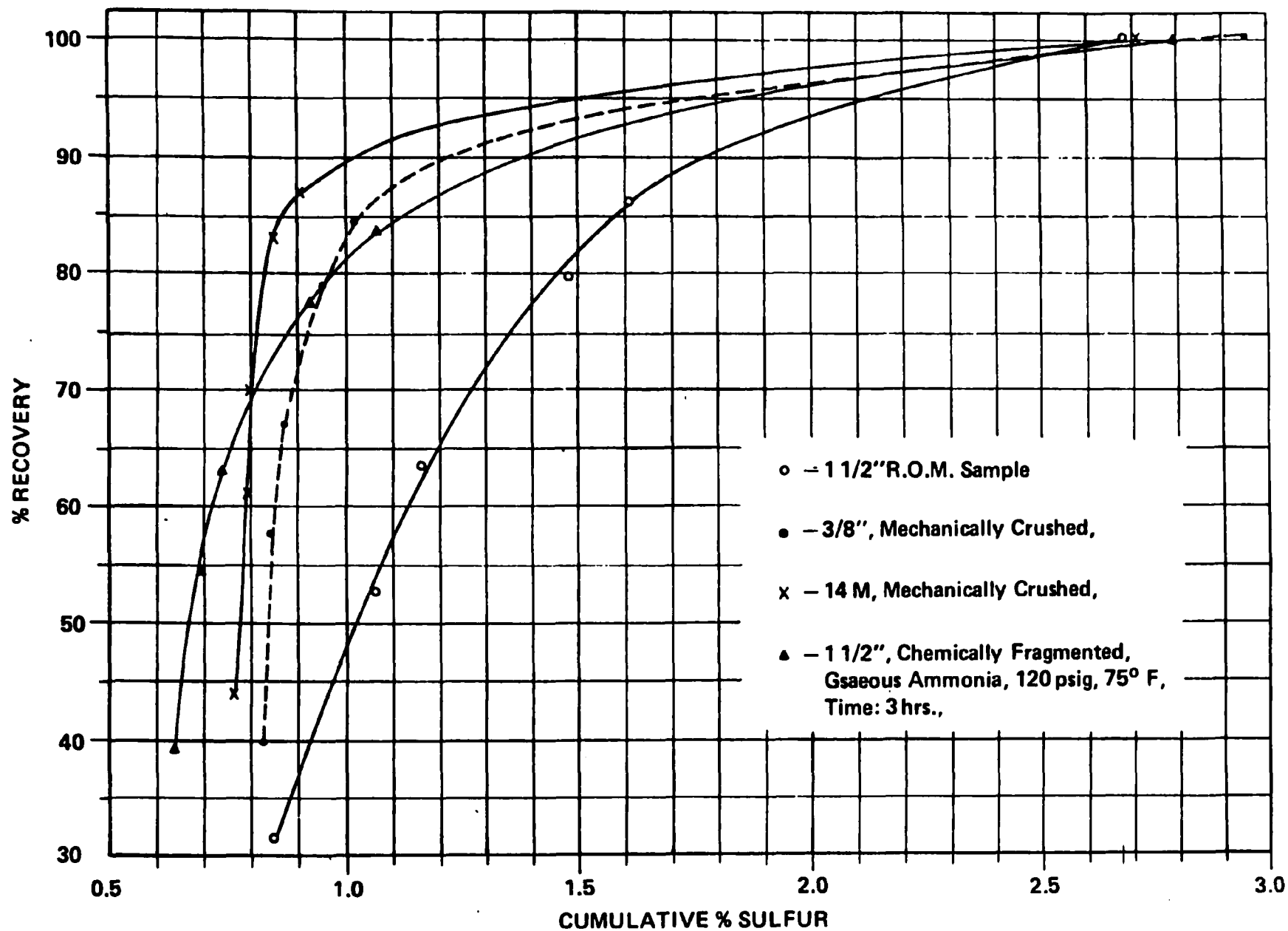


FIGURE IV - 2 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: PERCENT SULFUR VS. PERCENT RECOVERY OF UPPER FREEPORT COAL

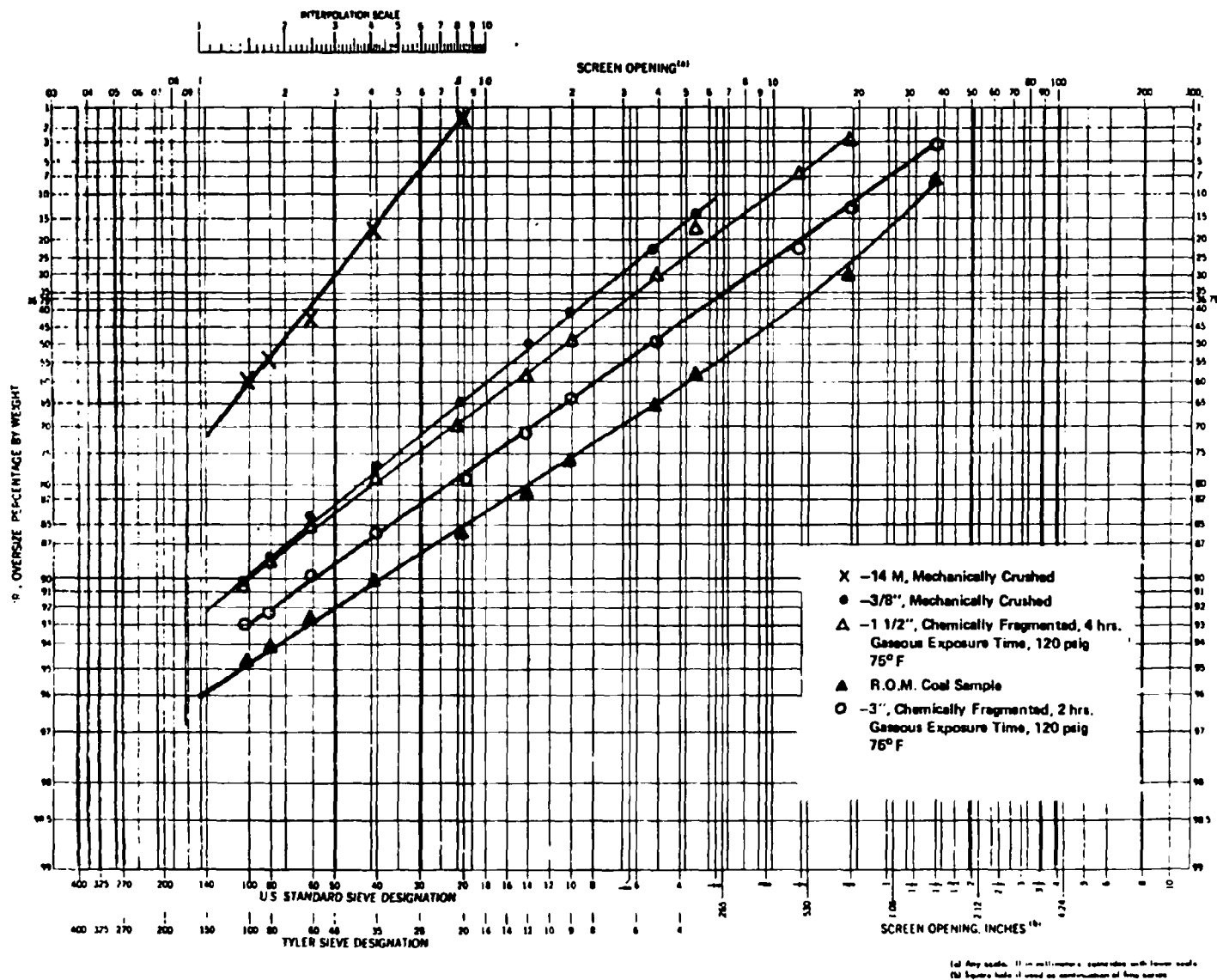
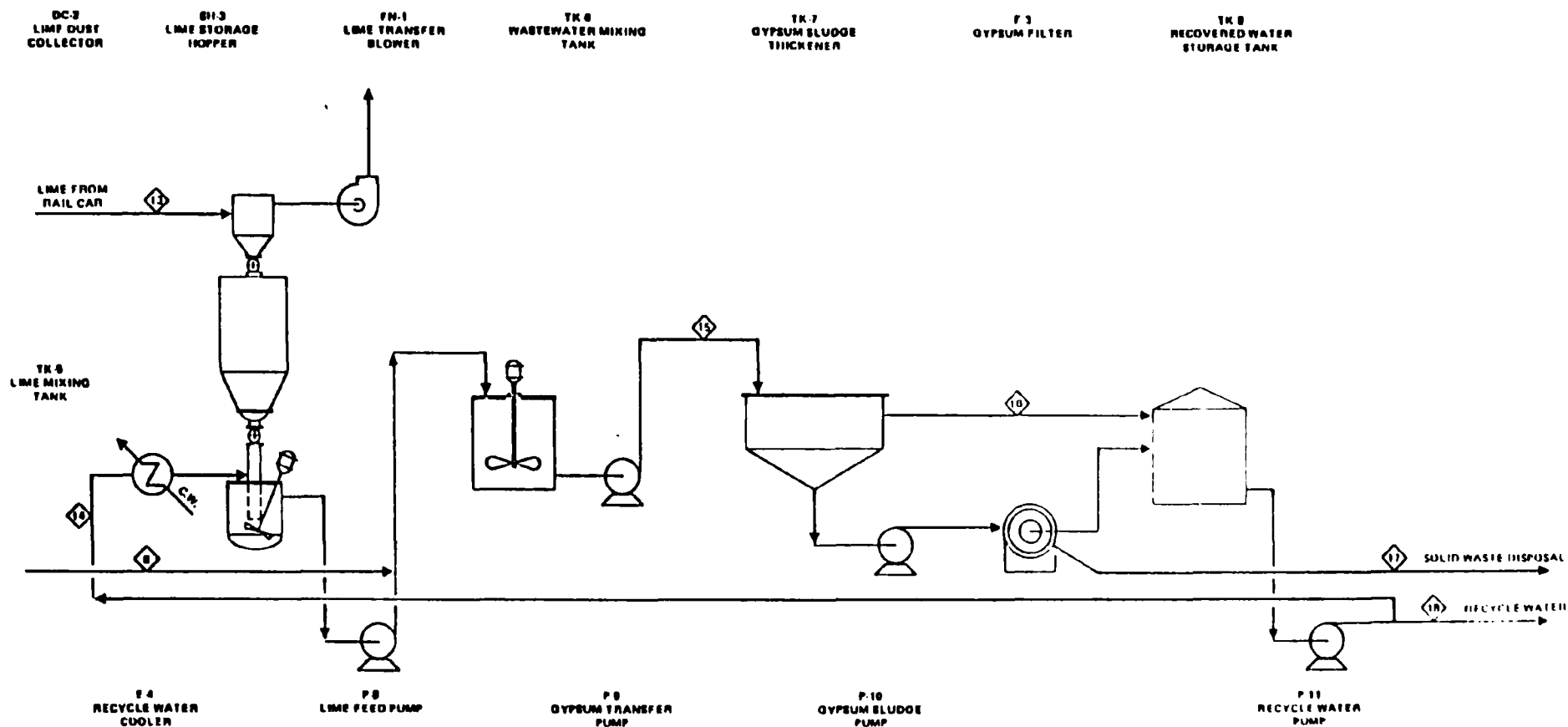


FIGURE IV - 3 SYRACUSE PROCESS VS. MECHANICAL CRUSHING: SIZE CONSIST OF VARIOUS PITTSBURGH COALS

APPENDIX V

ERDA PROCESS

FIGURE V - 1 ERDA PROCESS FLOW SHEET SUGGESTED BY BECHTEL



STREAM NUMBER	8	13	14	15	16	17	18
STREAM NAME	WASTE TO LIME TREATMENT	LIME FROM PAIL CAN	WATER FOR LIME MIXING	GYPSUM SLUDGE TO THICKENER	DECANTED WATER	SOLID WASTE DISPOSAL	RECYCLE WATER
TEMPERATURE, °F	160	80	183	183	183	183	183
PRESSURE, PSIA	63	11.0	87	60	15	15	60
GAS PHASE, FT ³ /MIN	-	27,000	-	-	-	-	-
SOLID PHASE, LBS/HR	-	174,100	-	36,000	-	36,000	-
LIQUID PHASE, LBS/HR	394,000	-	175,000	552,000	489,000	24,000	353,000
TOTAL, LBS/HR	394,000	287,100	175,000	588,000	489,000	(30,000)	353,000

Figure V-1 (Continued)

APPENDIX VI

JPL PROCESS

TABLE VI-1. JPL PROCESS: PROXIMATE ANALYSIS DATA OF TWO TESTED COALS³¹

<u>Coal</u>	<u>Fixed Carbon</u>	<u>Volatile Matter</u>	<u>Moisture</u>	<u>Ash</u>
Illinois No. 5	42.74%	36.35%	9.88%	11.03%
Kentucky No. 9	52.45%	35.0%	4.49%	8.06%

APPENDIX VII

IGT PROCESS

TABLE VII-1. IGT PROCESS: ANALYSIS OF WESTERN KENTUCKY
NO. 9 COAL USED IN BATCH REACTOR RUNS

	<u>Coal</u>	<u>Pretreated Coal</u>	<u>+40 Mesh Pretreated Coal</u>
Proximate Analysis , wt.%			
Moisture	5.8	0.8	1.6
Volatile Matter	36.3	27.7	26.7
Ash	10.6	11.2	14.1
Fixed Carbon	<u>47.3</u>	<u>60.3</u>	<u>57.6</u>
Total	100.0	100.0	100.0
Ultimate Analysis , wt.%			
Ash	11.24	11.25	14.31
Carbon	70.00	71.40	67.80
Hydrogen	4.54	4.06	3.84
Sulfur	3.74	3.16	3.23
Nitrogen	1.53	1.64	1.48
Oxygen	<u>8.95</u>	<u>8.49</u>	<u>9.34</u>
Total	100.00	100.00	100.00

TABLE VII-2. IGT PROCESS: THERMOBALANCE TEST RUN DATA, PRETREATED WESTERN KENTUCKY #9 COAL

<u>Run No.</u>	<u>Feed Coal*</u>	<u>Pretreated Coal</u>	<u>TB-76-8</u>	
	<u>W. Ky. No. 9</u>		<u>Pret. W. Ky. No. 9</u>	
Coal				
Heating Rate, °F/min			5	
Terminal Temperature, °F		750	1,500	
Holding Time, min		30	30	
Sulfur, wt %			<u>Feed</u>	<u>Residue</u>
Sulfide	0.02	0.01	0.01	0.19
Sulfate	0.52	0.10	0.10	0.00
Pyritic	0.92	1.25	1.25	0.03
Organic	1.57	1.29	1.29	0.28
Total	3.03	2.65	2.65	0.50
Weight, g				
Initial	100.00		2.2871	
Treated		90.84		1.5766
Weight Loss, %				
Total, wt		9.16		31.07
Coal, wt		9.16		31.07
Reduced Data				
Weight, lb		90.84	90.84	62.62
Sulfur Weight, lb				
Sulfide	0.02	0.01	0.01	0.12
Sulfate	0.52	0.09	0.09	0.00
Pyritic	0.92	1.13	1.13	0.02
Organic	1.57	1.17	1.17	0.18
Total	3.03	2.40	2.40	0.32
Sulfur Content, %				
Sulfide	0.02	0.01		
Sulfate	0.52	0.10		
Pyritic	0.92	1.25		0.03
Organic	1.57	1.29		0.28
Total	3.03	2.65		0.31
Sulfur Removal, wt %				
From Feed				87.1
From Coal		20.5		89.8

*Calculated for +40 mesh fraction.

TABLE VII-3. IGT PROCESS: THERMOBALANCE RUN DATA, WESTERN KENTUCKY
NO. 9 COAL (RAPID HEAT-UP RATE)

	Feed Coal*	Pretreated Coal*	TB-76-24 0:1		TB-76-45 0:1		TB-76-46 0:1		TB-76-47 0:1		TB-76-48 0:1	
Lime/Coal Feed Ratio												
Coal	W.Ky.No.9		Pretreated W.Ky.No.9									
Heating Rate, °F/min			Rapid Heating		Rapid Heating		Rapid Heating		Rapid Heating		Rapid Heating	
Terminal Temperature, °F		750	1500		1500		1500		1500		1500	
Holding Time, min		30	120		90		60		30		15	
			Feed	Residue	Feed	Residue	Feed	Residue	Feed	Residue	Feed	Residue
Sulfur, wt %												
Sulfide	0.02	0.01	0.01	0.13	0.01	0.23	0.01	0.23	0.01	0.41	0.01	0.50
Sulfate	0.52	0.10	0.10	0.00	0.10	0.00	0.10	0.01	0.10	0.01	0.10	0.02
Pyritic	0.92	1.25	1.25	0.02	1.25	0.02	1.25	0.02	1.25	0.02	1.25	0.00
Organic	1.57	1.29	1.29	0.14	1.29	0.32	1.29	0.46	1.29	0.57	1.29	0.68
Total	3.03	2.65	2.65	0.29	2.65	0.57	2.65	0.72	2.65	1.01	2.65	1.20
Weight, g												
Initial	100		2.5892		2.9610		2.8700		2.7170		2.5688	
Treated		90.84		1.7646		2.0686		2.0121		1.9329		1.8493
Weight Loss, %												
Total Weight		9.16		31.85		30.14		29.89		28.86		28.00
Coal Weight		9.16		31.85		30.14		29.89		28.86		28.00
Reduced Data												
Weight, lb		90.84	90.84	61.91	90.84	63.46	90.84	63.69	90.84	64.62	90.84	65.40
Sulfur Weight, lb												
Sulfide	0.02	0.01	0.01	0.08	0.01	0.15	0.01	0.15	0.01	0.26	0.01	0.33
Sulfate	0.52	0.09	0.09	0.00	0.09	0.00	0.09	0.01	0.09	0.01	0.09	0.01
Pyritic	0.92	1.13	1.13	0.01	1.13	0.01	1.13	0.01	1.13	0.01	1.13	0.00
Organic	1.57	1.17	1.17	0.09	1.17	0.20	1.17	0.29	1.17	0.37	1.17	0.44
Total	3.03	2.40	2.40	0.18	2.40	0.36	2.40	0.46	2.40	0.65	2.40	0.78
Sulfur Content, %												
Sulfide	0.02	0.01	0.01		0.01		0.01		0.01		0.01	
Sulfate	0.52	0.10	0.10		0.10		0.10		0.10		0.10	
Pyritic	0.92	1.25	1.25	0.02	1.25	0.02	1.25	0.02	1.25	0.02	1.25	0.00
Organic	1.57	1.29	1.29	0.14	1.29	0.32	1.29	0.46	1.29	0.57	1.29	0.68
Total	3.03	2.65	2.65	0.16	2.65	0.34	2.65	0.48	2.65	0.59	2.65	0.68
Sulfur Removal, wt %												
From Feed				95.8		91.3		87.5		84.2		81.7
From Coal		20.5		96.7		93.1		90.1		87.5		85.5

*Calculated for +40 mesh.

TABLE VII-4. IGT PROCESS: THERMOBALANCE RUN DATA, ILLINOIS NO. 6 COAL

<u>Run No.</u>	<u>Feed Coal*</u>	<u>Pretreated Coal</u>	<u>TB-76-42</u>	
Heating Rate, °F/min			5	
Terminal Temperature, °F		750	1,500	
Holding Time, min		30	30	
Sulfur, wt %			<u>Feed</u>	<u>Residue</u>
Sulfide	0.00	0.01	0.01	0.04
Sulfate	0.01	0.01	0.01	0.00
Pyritic	0.99	0.63	0.63	0.03
Organic	1.28	1.39	1.39	0.37
Total	2.28	2.04	2.04	0.44
Weight, g				
Initial	100		2.4040	
Treated		90.4		1.6678
Weight Loss, %				
Total Weight		9.6		30.65
Coal Weight		9.6		30.65
Reduced Data				
Weight, lb	100.00		90.4	62.69
Sulfur Weight, lb				
Sulfide	0.00	0.01	0.01	0.03
Sulfate	0.01	0.01	0.01	0.00
Pyritic	0.99	0.57	0.57	0.02
Organic	1.28	1.26	1.26	0.23
Total	2.28	1.85	1.85	0.28
Sulfur Content %				
Sulfide	0.00	0.01	0.01	
Sulfate	0.01	0.01	0.01	
Pyritic	0.99	0.63	0.63	0.03
Organic	1.28	1.39	1.39	0.37
Total	2.28	2.04	2.04	0.40
Sulfur Removal, wt %				
From Freed				86.5
From Coal		18.9		89.0

*Calculated for +40 mesh fraction.

TABLE VII-5. IGT PROCESS: BATCH REACTOR TEST RUN DATA FOR PRETREATED WESTERN KENTUCKY NO. 9 COAL

Run No.	Feed Coal*	+40 Mesh Pretreated Coal	BR-76-3	
Lime/Coal Feed Ratio			0:1	
Coal	W. Ky. No. 9			
Heating Rate, °F/min			5	
Terminal Temperature, °F		750	1,500	
Holding Time, min		30	30	
Sulfur, wt %			Feed	Residue
Sulfide	0.02	0.04	0.04	0.12
Sulfate	0.60	0.10	0.10	0.00
Pyritic	1.06	1.54	1.54	0.02
Organic	1.82	1.38	1.38	0.37
Total	3.50	3.06	3.06	0.51
Weight, g				
Initial	100.00		75.0	
Treated		90.84		51.7
Weight Loss, %				
Total Weight		9.16		31.1
Coal Weight		9.16		31.1
Reduced Data				
Weight, lb			90.84	62.62
Sulfur Weight, lb				
Sulfide	0.02	0.04	0.04	0.08
Sulfate	0.60	0.09	0.09	0.00
Pyritic	1.06	1.40	1.40	0.01
Organic	1.82	1.25	1.25	0.23
Total	3.50	2.78	2.78	0.32
Sulfur Content %				
Sulfide	0.02	0.04	0.04	
Sulfate	0.60	0.10	0.10	
Pyritic	1.06	1.54	1.54	0.02
Organic	1.82	1.38	1.38	0.37
Total	3.50	3.06	3.06	0.39
Sulfur Removal, wt %				
From Feed		20.6		91.4
From Coal				93.1
Heat Value, BTU/lb	12,454	11,809		11,967

*Calculated for 40 mesh fraction.

TABLE VII-6. ILLINOIS NO. 6 ANALYSES

	<u>Coal</u>	<u>Pretreated Coal</u>	<u>+40 Mesh Pretreated Coal</u>
Proximate Analysis , wt. %			
Moisture	1.3	0.4	1.6
Volatile Matter	37.3	28.5	27.6
Ash	8.5	9.4	8.8
Fixed Carbon	<u>52.9</u>	<u>61.7</u>	<u>62.0</u>
Total	100.0	100.0	100.0
Ultimate Analysis (dry) ,wt. %			
Ash	8.63	9.42	8.98
Carbon	74.50	75.20	74.80
Hydrogen	4.91	4.30	4.29
Sulfur	2.77	2.16	2.34
Nitrogen	1.49	1.66	1.55
Oxygen	<u>7.70</u>	<u>7.26</u>	<u>8.04</u>
Total	100.00	100.00	100.00

TABLE VII-7. IGT PROCESS: BATCH REACTOR TESTS-PRETREATED ILLINOIS NO. 6 (-10+40 MESH) COAL

<u>Run No.</u>	<u>Feed Coal*</u>	<u>Pretreated Coal</u>	<u>BR-76-34</u>	
Lime/Coal Feed Ratio			0:1	
Heating Rate, °F/min			5	
Terminal Temperature, °F		750	1,500	
Holding Time, min		30	30	
Sulfur, wt %			<u>Feed</u>	<u>Residue</u>
Sulfide	0.01	0.01	0.01	0.05
Sulfate	0.13	0.04	0.04	0.05
Pyritic	0.84	0.65	0.65	0.03
Organic	1.50	1.52	1.52	0.47
Total	2.48	2.22	2.22	0.60
Weight, g				
Initial	100.00		75.0	
Treated		90.84		52.2
Weight Loss, %				
Total Weight		9.6		30.4
Coal Weight		9.6		30.4
Reduced Data				
Weight, lb	100.00	90.4	90.4	62.9
Sulfur Weight, lb				
Sulfide	0.01	0.01	0.01	0.03
Sulfate	0.13	0.04	0.04	0.03
Pyritic	0.84	0.59	0.59	0.02
Organic	1.50	1.37	1.37	0.30
Total	2.48	2.01	2.01	0.38
Sulfur Content %				
Sulfide	0.01	0.01	0.01	
Sulfate	0.13	0.04	0.04	
Pyritic	0.84	0.65	0.65	0.03
Organic	1.50	1.52	1.52	0.47
Total	2.48	2.22	2.22	0.50
Sulfur Removal, wt %				
From Feed				84.1
From Coal		18.9		87.1
Heat Value, BTU/lb	13,022	13,069		12,793

*Calculated for +40 mesh fraction.

TABLE VII-8. PITTSBURGH SEAM, WEST VIRGINIA ANALYSES

	<u>Coal</u>	<u>Pretreated Coal</u>	<u>+40 Mesh Pretreated Coal</u>
Proximate Analysis, wt. %			
Moisture	2.2	0.3	0.7
Volatile Matter	35.9	25.5	26.6
Ash	10.6	13.0	9.8
Fixed Carbon	<u>51.3</u>	<u>61.2</u>	<u>62.9</u>
Total	100.0	100.0	100.0
Ultimate Analysis, wt %			
Ash	10.87	13.07	9.83
Carbon	73.40	71.5	74.80
Hydrogen	4.87	3.93	4.20
Sulfur	2.77	2.36	2.16
Nitrogen	1.37	1.42	1.47
Oxygen	<u>6.72</u>	<u>7.72</u>	<u>7.54</u>
Total	100.0	100.0	100.00

TABLE VII-9. IGT PROCESS: THERMOBALANCE RUN DATA, PRETREATED PITTSBURGH SEAM, WEST VIRGINIA COAL

Run No.	Feed Coal*	Pretreatment*	TB-76-18	
Lime/Coal Feed Ratio			0:1	
Coal	Pitt. Seam, W. Va.			
Heating Rate, °F/min			5	
Terminal Temperature, °F		750	1,500	
Holding Time, min		60	30	
Sulfur, wt %			Feed	Residue
Sulfide	0.00	0.00	0.00	0.29
Sulfate	0.42	0.27	0.27	0.00
Pyritic	0.63	0.37	0.37	0.01
Organic	1.36	1.47	1.47	0.39
Total	2.41	2.11	2.11	0.69
Weight, g				
Initial	100.00		2.4268	
Treated		83.5		1.7168
Weight Loss, %				
Total Weight		16.5		29.26
Coal Weight		16.5		29.26
Reduced Data				
Weight, lb		83.5	83.50	59.10
Sulfur Weight, lb				
Sulfide	0.00	0.00	0.00	0.17
Sulfate	0.42	0.23	0.23	0.00
Pyritic	0.63	0.31	0.31	0.01
Organic	1.36	1.23	1.23	0.23
Total	2.41	1.77	1.77	0.41
Sulfur Content %				
Sulfide	0.00	0.00	0.00	
Sulfate	0.42	0.27	0.27	
Pyritic	0.63	0.37	0.37	0.01
Organic	1.36	1.47	1.47	0.39
Total	2.41	2.11	2.11	0.40
Sulfur Removal, wt %				
From Feed				86.4
From Coal				90.0

*Calculated for 40 mesh fraction.

TABLE VII-10. ANALYSES OF PITTSBURGH SEAM COAL (Pennsylvania Mine)

	<u>Coal</u>	<u>Pretreated Coal</u>	<u>+40 Mesh Pretreated Coal</u>
Proximate Analysis, wt. %			
Moisture	3.0	0.3	0.9
Volatile Matter	26.0	19.7	21.2
Ash	33.3	35.3	33.7
Fixed Carbon	<u>37.7</u>	<u>44.7</u>	<u>44.2</u>
Total	100.0	100.0	100.0
Ultimate Analysis, wt. %			
Ash	34.34	35.41	33.99
Carbon	52.50	52.10	54.10
Hydrogen	3.54	2.94	3.26
Sulfur	1.35	1.23	1.11
Nitrogen	1.08	1.13	1.10
Oxygen	<u>7.19</u>	<u>7.19</u>	<u>6.44</u>
Total	100.00	100.00	100.00

TABLE VII-11. IGT PROCESS: THERMOBALANCE RUN DATA, PITTSBURGH SEAM COAL (PENNSYLVANIA MINE)

<u>Run No.</u>	<u>Feed Coal*</u>	<u>Pretreated Coal*</u>	<u>TB-76-32</u>	
Lime/Coal Feed Ratio			0:1	
Heating Rate, °F/min			5	
Terminal Temperature, °F		750	1,500	
Holding Time, min		30	30	
Sulfur, wt %			<u>Feed</u>	<u>Residue</u>
Sulfide	0.01	0.00	0.00	0.21
Sulfate	0.44	0.34	0.34	0.00
Pyritic	0.21	0.16	0.16	0.00
Organic	0.35	0.41	0.41	0.12
Total	1.01	0.91	0.91	0.33
Weight, g				
Initial	100.00		3.3107	
Treated		86.34		2.5341
Weight Loss, %				
Total Weight		13.66		23.46
Coal Weight		13.66		23.46
Reduced Data				
Weight, lb			86.34	66.08
Sulfur Weight, lb				
Sulfide	0.01	0.00	0.00	0.14
Sulfate	0.44	0.29	0.28	0.00
Pyritic	0.21	0.14	0.13	0.00
Organic	0.35	0.35	0.36	0.08
Total	1.01	0.78	0.77	0.22
Sulfur Content %				
Sulfide	0.01	0.00	0.00	0.00
Sulfate	0.44	0.34	0.34	0.00
Pyritic	0.21	0.16	0.16	0.00
Organic	0.35	0.41	0.41	0.12
Total	1.01	0.91	0.91	0.12
Sulfur Removal, wt %				
From Feed				89.6
From Coal		22.8		92.1

*Calculated for 40 mesh fraction.

TABLE VII-12. ILLINOIS NO. 6 ANALYSES (Hillsboro Mine)

Proximate Analysis, wt %

Moisture	12.02
Ash	22.83
Volatile Matter	30.18
Fixed Carbon	34.97

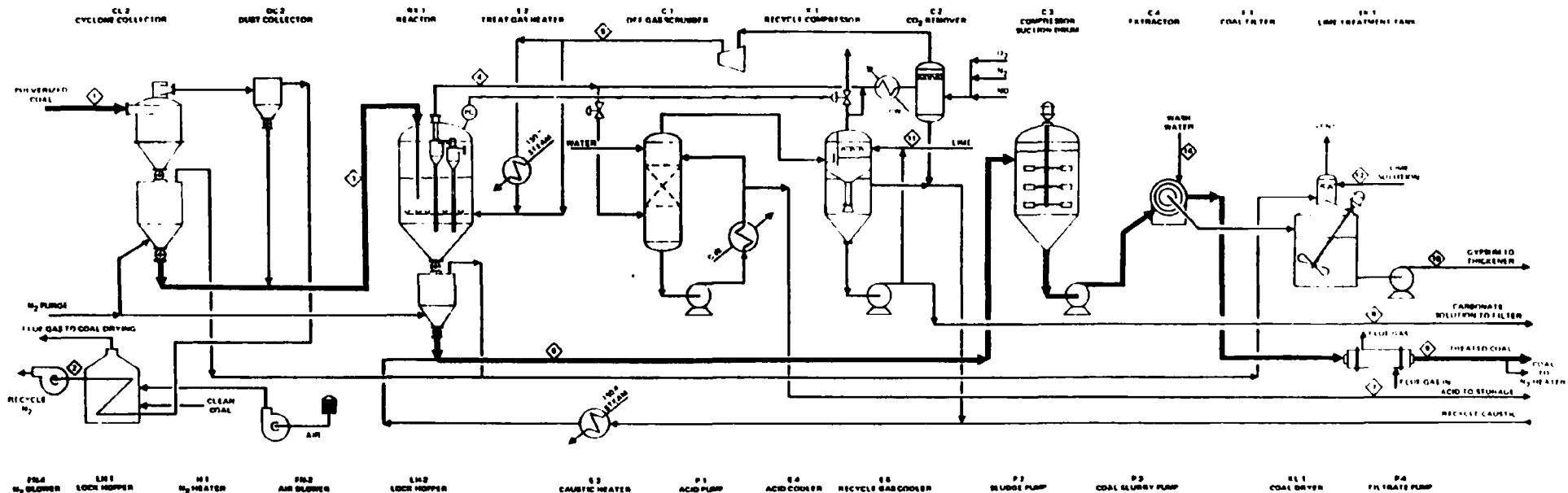
Ultimate Analysis, wt % (dry)

Ash	25.95
Carbon	57.07
Hydrogen	4.01
Sulfur	5.06
Nitrogen	0.98
Oxygen	6.82

Heating Value, BTU/lb	10,198
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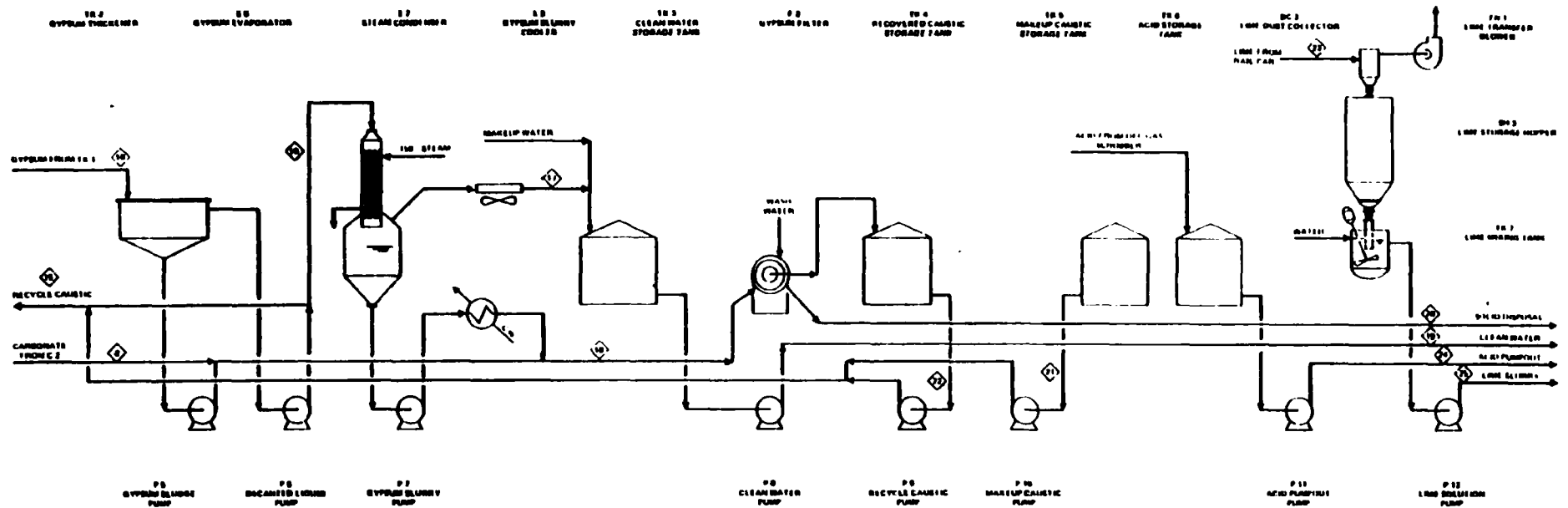
APPENDIX VIII

KVB PROCESS



STREAM NAME	1	2	3	4	5	6	7	8	9	10	11	12	13	14
STREAM NAME	PULVERIZED COAL	RECYCLE N ₂	COAL TO RE ACTION	OFF GAS FROM REACTION	TREAT GAS TO REACTION	COAL SLURRY	HHV ₂ + H ₂ O ₂ TO STORAGE	SOLID TO FILTER	TREATED COAL	GYPSUM SLURRY TO THICKENER	LIME SLURRY TO C 2	LIME SLURRY TO TR 1	90% CAUSTIC MANIP	WASH WATER TO FILTER
TEMPERATURE °F	250	250	250	250	250	250	150	130	-	150	130	120	50	115
POUNDS PER HOUR	10.4	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
SCHEMATIC FLOW	312,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000	340,000
LIQUID PHASE - LBS/HR	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000	667,000
TOTAL LBS/HR	1,007,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000	1,110,000

FIGURE VIII-1 KVB PROCESS FLOW SHEET SUGGESTED BY BECHTEL



Stream Name	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID
Stream Name	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID	Stream ID
TEMPERATURE, °F	120	103	100	103	100	100	110	100	100	100	100	100	100
PRESSURE, PSIA	75	35	65	65	15	35	15	15	15	15	15	15	15
SOLID PHASE, LBS/HR	0.000	21.700	1.000	1.000	0.000	21.700	0.000	21.700	1.000	1.000	1.000	1.000	1.000
LIQUID PHASE, LBS/HR	10.000	1.000	1.000	1.000	0.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
TOTAL, LBS/HR	10.000	22.700	2.000	2.000	0.000	22.700	1.000	22.700	2.000	2.000	2.000	2.000	2.000

Figure VIII-1. (Continued)

TECHNICAL REPORT DATA
(Please read instructions on the reverse before completing)

1. REPORT NO. EPA-600/7-78-173a	2.	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE Assessment of Coal Cleaning Technology: An Evaluation of Chemical Coal Cleaning Processes		5. REPORT DATE August 1978
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) G. Y. Contos, I. F. Frankel, and L. C. McCandless		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Versar, Inc. 6621 Electronic Drive Springfield, Virginia 22151		10. PROGRAM ELEMENT NO. EHE623A
		11. CONTRACT/GRANT NO. 68-02-2199
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		14. SPONSORING AGENCY CODE EPA/600/13

15. SUPPLEMENTARY NOTES **IERL-RTP project officer is James D. Kilgroe, Mail Drop 61, 919/541-2851.**

16. ABSTRACT **The report assembles and assesses technical and economic information on chemical coal cleaning processes. Sufficient data was located to evaluate 11 processes in detail. It was found that chemical coal cleaning processes can remove up to 99% of the pyritic sulfur and 40% of the organic sulfur, resulting in total sulfur removals of 53 to 77%. This performance can be achieved with heat value recoveries of 57 to 96%. Processes which remove only pyritic sulfur were generally judged to have the highest probabilities of success. Of techniques which remove both pyritic and organic sulfur, the ERDA and the GE microwave processes were judged to have the highest probabilities of success.**

17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution Coal Desulfurization Coal Preparation Pyrite Microwaves	Pollution Control Stationary Sources Chemical Coal Cleaning Pyritic Sulfur Organic Sulfur	13B 07C 08G, 21D 05C 07A, 07D 08I 20N
18. DISTRIBUTION STATEMENT Unlimited	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 295
	20. SECURITY CLASS (This page) Unclassified	22. PRICE