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**APPLICATION OF THE SULF-X PROCESS TO COAL CONVERSION
AND UTILIZATION**

Phase II. Final Report

**By
E. Shapiro
H. C. Bramer
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Work Performed Under Contract No. AC01-78ET11273

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APPLICATION OF THE SULF-X PROCESS
TO COAL CONVERSION AND UTILIZATION

PHASE II FINAL REPORT

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Prepared for the
DEPARTMENT OF ENERGY
OFFICE OF COAL UTILIZATION SYSTEMS
UNDER CONTRACT DE-AC01-78ET11273

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ABSTRACT

Pittsburgh Environmental and Energy Systems, Inc. contracted with the Department of Energy to demonstrate the efficacy of an iron sulfide flue gas treatment system (FGT) for removing sulfur dioxide (SO_2) and nitrogen oxides (NO_x) and to correlate process variables to system performance. Laboratory and bench-scale testing was conducted with the SULF-X process, using both synthesized gas and actual flue gas from a coal-fired furnace. Laboratory tests resulted in 95% SO_2 removal and up to 95% NO_x removal. The bench-scale system demonstrated similar SO_2 removal efficiencies, but achieved only 39% NO_x removal due to relatively high oxygen concentrations in the flue gas and insufficient liquid-gas interfacial area within the absorber. Elemental sulfur was recovered during the regeneration steps. Total capital investment for the SULF-X system was estimated to be \$91 to \$103 per kilowatt (electric), compared to \$90/kw for sodium solution scrubbing, \$78 to \$83/kw for magnesia slurry scrubbing and \$74/kw for limestone slurry scrubbing. Annual operating costs for the SULF-X system were estimated to be 5.44 to 6.90 mills per kilowatt-hour, compared to 4.96 to 5.22 for sodium, 3.68 to 3.99 for magnesia and 3.73 to 4.25 for limestone.

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SUMMARY

The SULF-X Process is an application of iron sulfide chemistry for the removal of sulfur dioxide (SO_2) and nitrogen oxides (NO_x) from combustion flue gases. In the laboratory, virtually 100% SO_2 removal and 70 to 80% nitric oxide removal were demonstrated repeatedly as part of Phase I of the Department of Energy Contract ET-78-C-01-1779. The results from the Phase I project were presented in an earlier report.

Batch bench-scale tests were originally conducted utilizing actual flue gas and regenerated FeS for up to twelve recycles. The scope of work was expanded and batch laboratory tests utilizing synthetic flue gas were conducted to determine performance for variations in FeS type, FeS concentration, SO_2 concentration, oxygen presence and ferrous ion concentration. The batch bench-scale tests were repeated for an additional five recycles. Steady-state tests were conducted on the bench apparatus using continuous feed and blowdown to maintain a constant system pH. Spent slurry was regenerated and reused to maintain a four-week run. The system was shut down overnight and on weekends.

Laboratory tests resulted in 95% SO_2 removal and up to 95% NO_x removal. Bench tests demonstrated similar SO_2 removal efficiencies, but obtained a maximum of only 39% NO_x removal. Major technical difficulties in the process, as determined in the bench and steady-state tests, were caused by both procedural and mechanical inadequacies that resulted in relatively poor NO_x removal and lower solids reactivity due to oxidation during regeneration.

The economics of the SULF-X process were estimated and compared to desulfurization processes. Total capital investment for the SULF-X system was estimated to be \$91 to \$103 per kilowatt, compared to \$90/kw for sodium solution scrubbing, \$78 to \$83/kw for magnesia slurry scrubbing and \$74/kw for limestone slurry scrubbing. Annual operating costs for the SULF-X system were estimated to be 5.44 to 6.90 mills per kilowatt-hour, compared to 4.96 to 5.22 for sodium, 3.68 to 3.99 for magnesia and 3.73 to 4.25 for limestone. All estimates were performed on a consistent basis, utilizing the procedures, techniques, data base and alternate system flowsheets described in a TVA study of advanced desulfurization processes prepared for EPA. The cost ranges for the capital and operating estimates are the result of a secondary evaluation that considered the use of coal for the SULF-X primary regeneration operations and resulting differences in the design basis.

INTRODUCTION

This final report addresses the objectives as stated in Department of Energy Contract No. DE-AC01-781123 (formerly ET-78-C-01-2779), Phase II, and is intended to fulfill the requirements of that contract phase.

Prior to completing the original scope of work under Phase II of the contract, PENSYS proposed to expand the scope of work to include steady-state testing with the bench-scale unit and to use more comprehensive analytical techniques to better define the system chemistry.

On September 18, 1979, a contract extension was issued by DOE. Laboratory and 20-cfm, bench-scale testing was conducted on the SULF-X Process at Westinghouse Advanced Energy Systems Division facilities. Test operations and on-line data gathering were performed by PENSYS; off-line sample analysis and data reduction were performed by Westinghouse. Results of this effort are reported in the Addendum.

This project is a continuing demonstration effort because of the successful results previously performed under the bench-scale feasibility tests identified in Phase I and IA.⁽¹⁾ The success of these initial efforts was followed by an expanded undertaking (Phase II) designed to evaluate SO_2/NO chemisorption with fresh ferrous sulfide (FeS) and with regenerated/recycled iron sulfide using a closed-cycle, bench-scale system on actual coal combustion gases.

The body of this report relates specifically to Phase II activities and discusses the equipment, mechanics and flow of the test system. The report presents operating and testing procedures and the results of these efforts. Finally, a discussion of the results with appropriate conclusions and recommendations is presented.

DESCRIPTION OF TEST SYSTEM DESIGN

Design Rationale

The design of the Phase II, 20 CFM test system is based on information acquired during Phase I of the Department of Energy (DOE) Contract ET-78-C-01-2779 as well as from previous laboratory and field testing of the SULF-X Process.⁽¹⁾⁽²⁾⁽³⁾

Phase II of this contract identified that Pittsburgh Environmental and Energy Systems, Inc. (PENSYS) would provide the personnel, apparatus, materials, and other items necessary to set up and check out the test system equipment for the performance of SO₂/NO chemisorption with fresh and regenerated/recycled iron sulfide.

The test system is comprised of the following four primary unit operations and is illustrated in Figure 1:

- absorption
- spent solids dewatering
- solids regeneration
- slurry preparation.

Figure 1. SULF-X Flow Diagram (conceptual)

In general, the test system was designed with the following considerations:

1. Equipment and instruments would be selected from readily available conventional industrial items.
2. Gravity flows would be established wherever feasible.
3. Process areas would relate to the experience gained in other similar environmental processes.
4. Storage of fresh and spent reagents would be based on a six-hour operating cycle.
5. SO_2 and NO_x concentrations could be based on typical operating values.

The 20 CFM pilot plant was designed and constructed according to the flow sheet "Bench Scale Test System" (Appendix Figure 1A). The system consisted of a domestic stoker as a combustion source to generate the flue gas, a 6-in. diameter absorber and recirculating pump, a compressor, six process tanks, a slurry feed pump, a spent slurry blowdown pump, a filter and filter pump, a dryer/regenerator/ball mill, and the necessary instrumentation to analyze the inlet and discharge gases (refer to Appendix for equipment and instrument listing).

For the absorption step, a scrubber design was chosen that had the following demonstrated characteristics:

- relatively maintenance free operation
- constant absorbing efficiency over a wide range of operating rates from 20 to 110% of rated gas flow
- low pressure drop per unit of efficiency
- low liquid recirculation rates, as low as 4 gpm/1000 CFM gas
- relatively high efficiency mass transfer
- proven size scalability

For the spent solids dewatering step, gravity settling tanks were included upstream from the filtering unit in order to reduce the volume of slurry that would need dewatering. A pressure filter consisting of a single container housing a filter cloth of predetermined porosity was chosen because of the following features:

- capacity to produce cake with minimal water retention
- simple design and construction
- ease of operation/maintenance.

For the regeneration step, an electric kiln with a removable steel container was chosen because of the following reasons:

- availability
- ease of operation/maintenance
- integral temperature controls
- adaptability for both drying and regenerating modes.

For the slurry preparation step, a ball mill was selected to break up agglomerates formed during the regeneration step and to produce a finely divided material with greater surface area for better reactivity. The unit was designed so the vessel from the previous regenerating step could also be utilized for the milling operation. The milled solids were suspended in the recycled supernate, and the resulting slurry was used for the subsequent cycle.

Equipment and Operation Descriptions

An Axeman Anderson Anthratube furnace, rated at 130,000 BTU per hour, was the combustion source. The furnace is a water wall overfeed domestic stoker normally used for hot water heating of homes when burning a pea-sized anthracite coal. A coal-feed hopper insured a continuous supply of coal to the stoker. An adjoining boiler was used as a heat sink to dissipate the heat in the hot water generated by the furnace during the test period.

The absorber is a venturi Flexitray unit 6-in. diameter by 15 ft. 10 in. high constructed of 316 stainless steel manufactured by the Koch Engineering Company. It contains five reaction trays, each containing a single venturi, and a final wash water tray, also with a single venturi. A 2.5-in. liquid level was maintained on each tray by overflow baffles that controlled the flow of liquid feeding the down comers that carried the liquid from tray to tray. The absorber is equipped with a side inlet and a top discharge for the gas to enter and leave the system. The scrubbing liquid entered on the top tray and cascaded from tray to tray, from the bottom of the unit to a 50-gallon reservoir. The minimum liquid flow rate for this unit is one gallon per minute in order to maintain the 2.5-in. liquid depth on each tray.

A Lapp Pulsafeeder (positive displacement) diaphragm metering pump, with a range of 0 to 423 gph, pumped the slurry from the reservoir tank to the fifth tray of the scrubber. The slurry flowed to the reservoir by gravity, and the process was continuously repeated.

A Gast vacuum pump, rated at 25 CFM, was used to induce flow through the absorber. The pump is a positive displacement compressor that also serves as a flow control device. The unit operated at a constant speed, and the pressure drop across the compressor was adjusted to maintain the desired flow according to the pressure versus flow chart.

The six process tanks are constructed of fiberglass and equipped with hinged covers. A 165-gallon tank, equipped with an Eastern Manufacturing Company 1/4-hp mixer, was used as the slurry or makedown tank. Two, 165-gallon tanks served as decant and filter feed tanks. A 165-gallon tank served as a filtrate holding tank. A 100-gallon tank was used as a filtrate recycle tank, and a 65-gallon tank equipped with a 1/4 hp mixer served as the absorber reservoir tank.

A felt bag filter, manufactured by GAF Corporation and fitted with a 10 micron filter bag, was installed to dewater the slurry and prepare the solids for drying and regeneration. The settled material from the decant tank was fed by gravity or pumped to the filter, and the filtrate was discharged into the filtrate holding tank. The unit is rated at 30 gpm at 75 psi.

Two positive displacement chemical feed pumps, manufactured by American Lewa Company, were used. One served as the makeup slurry feed pump to the recycle tank and was controlled by the pH controller that monitors the pH of the slurry in the recycle tank. The second pump served as the spent slurry blowdown pump and discharged spent slurry from the recycle tank into the decant tank for settling and separation.

The filter feed pump, manufactured by the Teel Manufacturing Company, pumped the concentrated spent slurry from the decant tanks to the filter at the rate of 15 gallons per minute at 80 inches water head. A filtrate transfer pump pumped the filtrate to the slurry makedown tank as required.

A small ceramic electric Skutt kiln with pyrometer and variable temperature controls served as the heat source for drying and regeneration. Drying and regeneration took place in the same unit. A drying/regeneration chamber equipped with a stirrer was fabricated from 12-in. diameter by 15-in. long, 304 stainless steel pipe and was designed to process 16 pounds of dry material per batch.

Two sulfur condensers, 15-in. diameter and 15-in. high, were fabricated from 316 stainless steel. One collected the elemental sulfur in dry form. The second served as a water trap and a seal to prevent oxygen from entering the system and as a device to maintain a positive pressure in the unit during pruging in the drying and regeneration cycles.

The regenerator was designed to serve also as a ball mill to minimize the handling and transfer of the oxidizable material. A Jar Mill drive was used to rotate the mill and 1/4-in. carbon steel balls were used to do the grinding.

These units were assembled according to the flow sheet equipment arrangements in Appendix Figures 1A, 2A and 3A. Upon completion of the installation, the system was filled with water to flush out all foreign matter and to check for leaks and the operation of the equipment. Preliminary testing of the equipment was then begun.

PRELIMINARY TESTING

After the initial check of the system for leaks and equipment operation, 50 gallons of a 1% caustic soda solution were circulated through the absorber and connecting pipe lines to flush the system and to observe its operational characteristics. During this period, flue gas from the stoker was also circulated through the absorber and the sampling systems were tested for performance and accuracy. A 50 gallon slurry of 1% FeS precipitated from the reaction between ferrous sulfate and sodium sulfide was then circulated through the system to further evaluate equipment and instrumentation performance with slurry as the scrubbing medium.

Absorber

The slurry recycle, slurry feed and blowdown pumps were calibrated during the caustic wash test using a flow through the absorber of one gallon per minute. The gas rate was maintained at approximately 20 CFM. After a short period of time, the recycle solution began accumulating fly ash and soot, indicating that the flue gas was being scrubbed by the recirculating solution within the absorber.

The gas flow through the absorber then began to decrease. The cartridge filters before and after the compressor were found to be plugged with moisture and particulate carryover from the absorber. The filters were replaced and a sand filter was installed between the absorber and the compressor to remove the bulk of the particulates and moisture. Periodic, but less frequent, changes of all filters were still required. The slurry feed pump and blowdown pumps were also affected by the particulates in the system. The problem was solved by installing both different style check valves in the pumps and strainers in the pump suction lines.

Filters

Spent slurry from a previous study conducted by PENSYS was used to evaluate the filter. This slurry passed through the 10 micron bag without developing a filter cake. Pulverized coal and pyrite were tested as filter aides without success.

The material either passed directly through the filter or developed such a dense cake that the flow through the filter ceased. Different filter bags were tested, including a one-micron wool felt bag, with essentially the same results. A laboratory Buchner funnel with vacuum filtration proved to be the most effective in dewatering this slurry. This filtering technique was used throughout the test program and generally produced a filter cake containing 20 to 30% moisture. However, this method was extremely time-consuming and resulted in lengthy and irregular delays that eventually affected all system functions.

Drying

The filter cake was manually transferred to the regenerator/dryer, and several tests were performed to determine the optimum drying time. A drying time of three hours at 150°C gave the most consistent results. A 2.5-liter-per-minute nitrogen purge was used to maintain an inert atmosphere within the dryer. All drying operations were performed in this manner. The subsequent regeneration step was performed in the same unit but at a higher temperature and, for some tests, with purge gases of different compositions.

Regeneration

To evaluate the performance of the regenerator and associated sulfur condensers, four pounds of pyrite were charged into the container and heated at 750°C. A 2.5-liter-per-minute nitrogen purge through the container was maintained during the entire heating cycle. Sulfur fumes were seen in the sight glass located in the discharge line between the regenerator and the dry condenser, indicating sulfur was being driven off from the pyrite. However, some problems were encountered. The regenerator and sulfur condenser gaskets leaked, the sight glass was too fragile, and the sulfur fumes condensed prematurely in the discharge line, creating a blockage and preventing any further flow of sulfur vapor and purge gas. The sight glass was replaced with a section of steel pipe, new gaskets were secured, and the line wrapped with electric heat tape to prevent the solidification of sulfur prior to the condenser. Several more tests were conducted to develop system reliability and familiarity.

Eight pounds of pyrite were charged into the regenerator, maintained at 750°C for three hours under a nitrogen purge, and then allowed to cool in place. One and one-half pounds of sulfur were collected in the sulfur trap indicating almost complete conversion of the pyrites to iron sulfide (FeS). This reagent was mixed with water and tested with the absorption equipment for activity toward sulfur dioxide; it was reactive. The regeneration with fresh pyrite was repeated, maintaining the regenerator temperature at 750°C for four hours to determine the effect of increasing the heating time. The $1\frac{1}{2}$ pounds of sulfur recovered indicated that the additional hour had no effect on the amount of sulfur dissociated from the pyrite nor on the FeS reactivity. During subsequent tests, the regenerator was maintained at 750°C for three hours. The total regeneration cycle was ten hours; three hours to reach 750°C , three hours at 750°C and four hours to cool down.

Ball Mill

The regenerator container was designed to serve as a ball mill. Upon completion of the regeneration cycle, the container was weighed and water added to the material in the regenerator to form a 50%-by-weight slurry. The top of the container and stirrer were removed and 50 pounds of $\frac{1}{4}$ -in. steel milling balls were added. The unit was then sealed and placed on the drive rollers and allowed to rotate overnight for 15 hours. Subsequently, a brief investigation indicated that slightly less time was required to yield a suitably fine particle size, so a timer was installed and a 12-hour grinding cycle was established. All milling operations during this project were conducted in this manner. Upon completion of the milling, the filtrate from the previous run was used to wash out the ball mill and prepare the feed slurry for the succeeding run.

TEST RESULTS

During the bench-scale tests conducted in Phase I of this contract and the initial tests in the current phase, PENSYS used an iron sulfide reagent that had been precipitated from the chemical reaction between ferrous sulfate and sodium sulfide. However, after the initial cycle, ground pyrites became the primary source of the reagent. The pyrites had the following compositions:

Fe	=	45.28%
S	=	52.48%
SiO ₂	=	0.88%
Other	=	1.35%

Two different types of Kentucky and Ohio bituminous coal, already at the site, were available for use in the pilot plant stoker:

<u>Kentucky Coal</u>	<u>Ohio Coal</u>
12,265 BTU/lb.	10,702 BTU/lb.
3.89% sulfur	3.37% sulfur
14.23 ash	23.34% ash

The coals were screened to $-\frac{1}{2}$ -in. by $+\frac{1}{4}$ -in. pea size. Both coals performed equally well throughout the testing period.

During the preliminary check-out of the equipment, a procedure was established whereby reagent generated from pyrite was mixed in a slurry and recirculated in the absorption column until exhausted at a pH of 5. The spent slurry was filtered, dried and regenerated. The time required to perform each unit process permitted duplicate runs to be made, thereby allowing a cross check on reproducibility. One test series served as a control, while conditions in the second series were varied to investigate correlations between process variables.

Four cycles had been performed, two on each series, when PENSYS determined that material losses during regeneration and filtering steps were becoming excessive. The decision was made to abandon all tests to date and start over using the experience gained on the first four cycles to minimize material losses from the system. A test schedule (Appendix Table 2A) was established to maximize the effort and establish a routine for performing the work.

Even though the key components in the flue gas were continually monitored, there was no specific instrumentation available to control their concentrations. The furnace which was the source of the flue gas failed to operate uniformly thus causing the concentrations of SO_2 , NO_x and O_2 in the flue gas to fluctuate beyond desirable limits. The average O_2 concentration was also high (13%) throughout the study. The severity of the fluctuations is illustrated in Figure 2, which is a reduced copy of a segment of chart paper containing operating data. Every effort was made to minimize changes in the fuel source, but coal is not homogenous, and some variations in the flue gas were directly attributable to the coal. Although the cycling of the gas concentrations did not appear to adversely affect the removal efficiency of the test system, it did mask system changes and hinder the subsequent quantitative evaluation of the effect of flue gas composition on the system chemistry.

The significant test data for the absorption and regeneration runs are presented in Tables 1, 2, 3 and 4. Additional test data are presented in Tables 3A and 4A in the Appendix. Series A and its duplicate, Series B, were each initially generated from eight pounds of pyrite. The resultant generated material was used to prepare a 50% slurry that was milled for 12 hours, suspended in 50 gallons of fresh water in the recycle tank, and exposed to flue gases in the absorber until the pH of the slurry reached five. The spent slurry was allowed to settle, the settled material was filtered and the filter cake was transferred to the dryer. Two ounces of activated carbon, which was intended reductant, and 2.5 pounds of fresh pyrite were added to the filter cake. The pyrite was added to compensate for both handling and dissolution losses. The mixture was then dried, regenerated and resuspended in the filtrate from the previous absorption run. The initial absorption, A1W, was followed by regeneration, A1R, and the resultant slurry was feed for A2W absorption. This cycle designation continued throughout the length of the testing program.

There were 12 cycles each of Series A and B for a total of 26 tests in all, including the initial cycle with caustic soda solution. The original starting material, pyrite, was used and regenerated twelve times. The capacity of the material varied somewhat, and the methods to improve the capacity were, in general, successful to the point that the final capacity after 12 reuses was equal to or greater than the original capacity of the material.

Figure 2 - Data Recorded with Multipoint Recorder

Legend

1. Inlet SO₂ Concentration: scale 0-3000 ppm_v
2. Outlet SO₂ Concentration: scale 0-3000 ppm_v
3. Inlet NO₂ Concentration: scale 0-1000 ppm_v
4. Outlet NO₂ Concentration: scale 0-1000 ppm_v
5. Inlet O₂ Concentration: scale 0-25% vol.
6. Outlet O₂ Concentration: scale 0-25% vol.
7. Slurry pH (non-linear scale)

TABLE 1

Absorption Runs
A Series

Sample No.	A1	A2	A3	A4	A5	A6
Date	<u>5/23/79</u>	<u>6/01/79</u>	<u>6/07/79</u>	<u>6/13/79</u>	<u>6/19/79</u>	<u>6/15/79</u>
Wt. Dry Material (lbs)	6.50	8.00	9.31	8.69	9.28	9.56
Vol. of Slurry, initial (gal)	50	53	51	49	56	50
end (gal)	53	51	49	54	55	55
Wt. % Solids	1.40	1.86	2.21	1.09	1.99	2.09
Initial pH	7.0	6.5	6.1	6.0	5.9	5.9
Final pH	5.0	5.0	5.0	5.0	4.5	4.8
Recycle Rate (gpm)	1	1	1	1	1	1
Length of Run (hrs)	2.10	4.00	3.32	4.42	3.45	5.00
Flue Gas Rate (scfm)	13.1	11.9	12.3	10.4	11.2	10.9
TSCF to Absorber	1,647	1,863	2,444	2,768	2,314	3,258
SO ₂ Avg. Inlet Conc. (ppm _v)	1,187	1,183	988	1,048	924	1,054
Outlet Conc. (ppm _v)	30	30	30	30	30	30
SO ₂ Removal %	97.5	97.5	96.9	97.1	96.7	97.1
SO ₂ Removed (ft ³)	1.96	3.39	2.41	2.90	2.14	3.43
SO ₂ Removed (gm·mols)	2.30	3.98	2.84	3.40	2.51	4.03
NO _x Inlet Conc. (ppm _v)	152	157	169	196	184	194
NO _x Outlet Conc. (ppm _v)	150	162	161	167	143	152
NO _x Removal (%)	1.8	0	4.7	14.9	22.0	21.6
L/G ($\frac{\text{gpm}}{1000 \text{ SCFM}}$)	77	84	81	96	89	92-184
Utilization, $\frac{\text{SO}_2}{\text{FeS}}$ (molar)*	0.069	0.096	0.059	0.076	0.052	0.082

* assuming "dry material" is 100% FeS

TABLE 1 (Cont.)

Absorption Runs
A Series

Sample No.	A7	A8	A9	A10	A11	A12
Date	<u>6/29/79</u>	<u>7/09/79</u>	<u>7/13/79</u>	<u>7/19/79</u>	<u>7/25/79</u>	<u>8/01/79</u>
Wt. dry material (lbs)	10.16	10.44	10.44	9.13	9.84	10.13
Vol. of Slurry, initial (gal)	55	53	25	26	38	36
end (gal)	53	60	25	38	36	34
Wt. % Solids	2.22	2.09	5.49	3.13	3.48	3.37
Initial pH	5.4	5.6	5.4	4.9	5.5	5.8
Final pH	4.5	4.5	4.5	4.5	4.5	4.5
Recycle Rate (gpm)	1-2	1-2	2	2	2	2
Length of Run (hrs)	3.87	4.37	1.75	0.63	2.33	4.00
Flue Gas Rate (scfm)	9.6	10.3	15.2	16.5	12.9	13.2
TSCF to Absorber	2,221	2,701	1,600	628	1,800	3,142
SO ₂ Avg. Inlet Conc. (ppm _v)	1,016	918	967	479	857	902
Outlet Conc. (ppm _v)	30	30	30	30	30	30
SO ₂ Removal %	97.0	96.7	96.9	93.7	96.5	96.7
SO ₂ Removed (ft ³)	2.28	2.48	1.55	0.30	1.54	2.84
SO ₂ Removed (gm·mols)	2.65	2.92	1.82	0.35	1.81	3.33
NO _x Inlet Conc. (ppm _v)	216	195	172	79	167	167
NO _x Outlet Conc. (ppm _v)	176	161	119	73	121	141
NO _x Removal (%)	18.5	17.6	30.7	6.9	27.5	15.8
L/G ($\frac{\text{gpm}}{1000 \text{ scfm}}$)	104-208	194	132	121	155	152
Utilization $\frac{\text{SO}_2}{\text{FeS}}$ (molar)*	0.051	0.054	0.031	0.007	0.036	0.064

*assuming "dry material" is 100% FeS

TABLE 2
Absorption Runs
B Series

Sample No.	B1	B2	B3	B4	B5	B6
Date	<u>5/25/79</u>	<u>6/05/79</u>	<u>6/11/79</u>	<u>6/15/79</u>	<u>6/21/79</u>	<u>6/27/79</u>
Wt. dry material (lbs)	6.50	6.95	8.10	9.56	9.41	10.56
Vol. of Slurry, initial (gal)	50	49	47	51	50	48
end (gal)	49	47	51	50	48	47
Wt. % Solids	1.66	1.76	2.22	2.29	2.26	2.64
Initial pH	7.2	6.1	6.0	5.0	5.0	5.0
Final pH	5.0	5.0	5.0	4.5	4.5	4.5
Recycle Rate (gpm)	1	1	1	1	1	1-2
Length of Run (hrs)	3.20	4.47	4.42	3.25	5.65	4.95
16 Flue Gas Rate (scfm)	13.4	10.6	11.4	11.5	10.1	10.7
TSCF to Absorber	2,576	2,839	3,026	2,261	3,448	3,200
SO ₂ Avg. Inlet Conc. (ppm _v)	1,052	1,153	875	1,011	916	951
Outlet Conc. (ppm _v)	30	30	30	30	30	30
SO ₂ Removal (%)	97.2	97.4	96.6	97.0	96.7	96.8
SO ₂ Removed (ft ³)	2.71	3.27	2.65	2.29	3.16	3.04
SO ₂ Removed (gm·mols)	3.18	3.84	3.11	2.68	3.71	3.57
NO _x Inlet Conc. (ppm _v)	164	229	163	194	187	174
NO _x Outlet Conc. (ppm _v)	163	208	145	168	152	137
NO _x Removal (%)	0.6	9.4	11.1	13.6	18.5	21.6
L/G ($\frac{\text{gpm}}{1000 \text{ scfm}}$)	75	94	88	86	98	928-186
Utilization $\frac{\text{SO}_2}{\text{FeS}}$ (molar)*	0.095	0.107	0.074	0.054	0.076	0.066

*assuming "dry material" is 100% FeS

TABLE 2 (Cont.)

Absorption Runs
B Series

Sample No.	B7	B8	B9	B10	B11	B12-1	B12-2
Date	<u>7/23/79</u>	<u>7/11/79</u>	<u>7/17/79</u>	<u>7/23/79</u>	<u>7/30/79</u>	<u>8/03/79</u>	<u>8/03/79</u>
Wt. dry material (lbs)	11.44	11.25	11.03	12.15	12.88	5.83	5.83
Vol. of Slurry, initial (gal)	54	51	49	47	45	45	45
end (gal)	54	49	47	45	43	44	43
Wt. % Solids	2.54	2.68	2.64	3.10	3.43	1.55	1.55
Initial pH	5.3	5.0	5.0	5.6	5.8	5.5	7.0
Final pH	4.5	4.5	4.5	4.5	4.5	4.5	4.5
Recycle Rate (gpm)	1.5	2	2	2	2	2	2
Length of Run (hrs)	0.50	1.22	0.68	2.17	4.00	2.42	7.32
Flue Gas Rate (scfm)	12.9	8.3	16.0	14.9	14.9	15.1	13.1
TSCF to Absorber	389	61	659	1,941	3,375	2,193	5,789
SO ₂ Avg. Inlet Conc. (ppm _v)	643	723	407	856	1,059	794	1,137
Outlet Conc. (ppm _v)	30	30	30	30	30	30	30
SO ₂ Removal (%)	95.3	95.8	92.6	96.5	97.2	96.2	97.4
SO ₂ Removed (ft ³)	0.25	0.44	0.26	1.66	3.57	1.74	6.58
SO ₂ Removed (gm·mols)	0.29	0.52	0.32	1.95	4.2	2.05	7.73
NO _x Inlet Conc. (ppm _v)	147	150	94	146	192	162	204
NO _x Outlet Conc. (ppm _v)	116	92	109	108	162	135	179
NO _x Removal (%)	22.4	38.7	-	26.3	15.9	16.8	12.2
L/G ($\frac{\text{gpm}}{1000 \text{ scfm}}$)	-	238	124	134	142	132	152
Utilization, $\frac{\text{SO}_2}{\text{FeS}}$ (molar)*	0.005	0.009	0.006	0.031	0.063	0.058	0.257

*assuming "dry material" is 100% FeS

TABLE 3

Regenerator-Material Balance
A Series

Sample No.	A	A1	A2	A3	A4	A5
Date	<u>5/21/79</u>	<u>5/31/79</u>	<u>6/06/79</u>	<u>6/11/79</u>	<u>6/17/79</u>	<u>6/21/79</u>
Wt. Filter Cake (lbs)	-	7.69	10.31	11.06	12.22	12.75
% Moisture	-	26.1	31.8	27.4	34.2	31.8
Water (lbs)	-	2.01	3.28	3.03	4.18	4.06
Dry Cake (lbs)	-	5.68	7.03	8.03	8.04	8.69
% Fe	-	57.5	55.3	51.1	51.7	52.7
% S	-	41.2	42.2	46.9	38.4	32.3
Makeup FeS ₂ (lbs)	8.0	2.5	2.4	1.0	1.0	1.0
% Fe	45.3	45.3	45.3	45.3	45.3	45.3
% S	52.5	52.5	52.5	52.5	52.5	52.5
18 Regenerated FeS (lbs)	6.50	8.00	9.31	8.69	9.28	9.56
% Fe	59.4	56.2	57.3	54.5	49.6	54.2
% S	39.2	42.4	41.4	33.5	32.0	33.8
To Regenerator:						
Fe, Cake	-	3.266	3.888	4.103	4.157	4.580
Fe, Makeup	3.624	1.133	1.078	0.453	0.453	0.453
Fe, Vessel Walls	0.163	0.124	0.241	0.137	0.163	0.124
Fe Total (lbs)	<u>3.787</u>	<u>4.523</u>	<u>4.207</u>	<u>4.693</u>	<u>4.773</u>	<u>5.157</u>
S, Cake	-	2.340	3.044	3.766	3.087	2.807
S, Makeup	4.200	1.313	1.250	0.525	0.525	0.525
S, Total (lbs)	<u>4.200</u>	<u>3.653</u>	<u>4.294</u>	<u>4.291</u>	<u>3.612</u>	<u>3.322</u>
From Regenerator:						
Fe Discharge (lbs)	3.861	4.496	5.335	4.734	4.603	5.182
S, Discharge	2.548	3.392	3.854	2.910	2.970	3.231
S, Dry Trap	1.289	0.314	0.012	0.012	0.003	0.004
S, Wet Trap	0.025	0.029	0.044	0.007	0.006	0.005
S, Vent Gas	0.003	0.026	0.140	0.001	0.001	0.001
S Total (lbs)	<u>3.865</u>	<u>3.761</u>	<u>4.050</u>	<u>2.930</u>	<u>2.980</u>	<u>3.241</u>

TABLE 3 (Cont.)

Regenerator-Material Balance
A Series

Sample No.	A6	A7	A8	A9	A10	A11
Date	<u>6/27/79</u>	<u>7/03/79</u>	<u>7/11/79</u>	<u>7/17/79</u>	<u>7/23/79</u>	<u>7/27/79</u>
Wt. Filter Cake (lbs)	13.00	13.75	14.75	14.31	14.25	14.19
% Moisture	29.5	31.2	30.8	32.2	38.2	25.9
Water (lbs)	3.84	4.29	4.55	4.61	5.45	3.68
Dry Cake (lbs)	9.16	9.46	10.20	9.70	8.80	10.51
% Fe	52.6	52.6	52.2	50.3	48.2	47.8
% S	33.7	30.6	32.3	28.7	27.0	26.4
Makeup FeS ₂ (lbs)	1.0	1.0	0	0	1.0	1.0
% Fe	45.3	45.3	-	-	45.3	45.3
% S	52.5	52.5	-	-	52.5	52.5
Regenerated FeS (lbs)	10.16	10.44	10.44	9.13	9.84	10.13
% Fe	53.8	52.2	51.4	49.5	47.8	47.8
% S	30.8	32.1	31.1	29.3	25.9	26.6
To Regenerator:						
Fe, Cake	4.818	4.976	5.324	4.520	4.242	4.372
Fe, Makeup	0.453	0.453	0	0	0.453	0.453
Fe, Vessel Walls	0.163	0	0.026	0	0	0
Fe Total (lbs)	<u>5.434</u>	<u>5.429</u>	<u>5.350</u>	<u>4.520</u>	<u>4.695</u>	<u>4.825</u>
S, Cake	3.087	2.895	3.295	2.784	2.376	2.775
S, Makeup	0.525	0.525	0	0	0.525	0.525
S Total (lbs)	<u>3.612</u>	<u>3.420</u>	<u>3.295</u>	<u>2.784</u>	<u>2.901</u>	<u>3.300</u>
From Regenerator:						
Fe Discharge (lbs)	5.464	5.448	5.365	4.517	4.705	4.840
S, Discharge	3.128	3.350	3.246	2.674	2.549	2.693
S, Dry Trap	0.004	0.007	0.006	0.006	0.004	0.004
S, Wet Trap	0.005	0.005	0.006	0.001	0.005	0.006
S, Vent Gas	0.002	0.002	0.000	0.001	0.004	0.004
S Total (lbs)	<u>3.139</u>	<u>3.364</u>	<u>3.258</u>	<u>2.682</u>	<u>2.562</u>	<u>2.707</u>

TABLE 4

Regenerator-Material Balance
B Series

Sample No.	B	B1	B2	B3	B4	B5
Date	<u>5/24/79</u>	<u>6/01/79</u>	<u>6/08/79</u>	<u>6/13/79</u>	<u>6/19/79</u>	<u>6/25/79</u>
Wt. Filter Cake	-	7.19	8.81	12.41	11.94	13.81
Water (lbs)	-	2.81	3.22	4.25	3.55	4.1
% Moisture	-	38.9	36.5	34.2	29.7	29.7
Dry Cake (lbs)	-	4.41	5.59	8.16	8.39	9.71
% Fe	-	55.8	55.3	55.1	50.6	51.4
% S	-	42.5	43.1	33.0	33.6	31.9
Makeup FeS ₂ (lbs)	8.0	2.4	2.5	1.0	1.0	1.0
% Fe	45.3	45.3	45.3	45.3	45.3	45.3
% S	52.5	52.5	52.5	52.5	52.5	52.5
Regenerated FeS (lbs)	6.50	6.94	8.09	9.56	9.41	10.56
% Fe	58.2	55.5	55.6	53.5	52.3	52.7
% S	40.4	42.8	36.6	37.0	32.8	31.0
To Regenerator:						
Fe, Cake	-	2.461	3.091	4.496	4.245	4.991
Fe, Makeup	3.624	1.105	1.133	0.453	0.453	0.453
Fe, Vessel Walls	0.202	0.286	0.163	0.163	0.221	0.124
Fe Total (lbs)	<u>3.826</u>	<u>3.852</u>	<u>4.337</u>	<u>5.112</u>	<u>4.919</u>	<u>5.568</u>
S, Cake	-	1.874	2.409	2.693	2.819	3.097
S, Makeup	4.20	1.881	1.313	0.525	0.525	0.525
S, Total (lbs)	<u>4.20</u>	<u>3.155</u>	<u>3.722</u>	<u>3.218</u>	<u>3.344</u>	<u>3.622</u>
From Regenerator:						
Fe Discharge (lbs)	3.783	3.852	4.498	5.115	4.921	5.565
S, Discharge	2.626	2.970	2.961	3.537	3.086	3.274
S, Dry Trap	1.339	0.295	0.142	0.004	0.004	0.005
S, Wet Trap	0.011	0.128	0.013	0.009	0.006	0.008
S, Vent Gas	0.015	0.026	0.037	0.000	0.010	0.002
S, Total (lbs)	<u>3.991</u>	<u>3.419</u>	<u>3.153</u>	<u>3.550</u>	<u>3.116</u>	<u>3.289</u>

TABLE 4 (Cont.)

Regenerator-Material Balance
B Series

Sample No.	B6	B7	B8	B9	B10	B11
Date	<u>6/29/79</u>	<u>7/09/79</u>	<u>7/13/79</u>	<u>7/19/79</u>	<u>7/29/79</u>	<u>8/02/79</u>
Wt. Filter Cake	15.50	19.06	16.19	15.94	17.47	18.00
Water (lbs)	4.88	8.88	4.97	4.69	4.86	7.28
% Moisture	31.5	46.6	30.7	29.4	27.8	40.4
Dry Cake (lbs)	10.62	10.18	11.22	11.25	12.61	10.72
% Fe	51.3	49.8	48.9	49.1	47.8	47.8
% S	30.1	27.8	28.3	29.4	28.6	26.1
Makeup FeS ₂ (lbs)	1.0	1.0	0	0.75	0.75	0.75/0.75
% Fe	45.3	45.3	-	64.4	45.3	64.4/45.3
% S	52.5	52.5	-	0	52.5	0/52.5
21 Regenerated FeS (lbs)	11.44	11.25	11.03	12.16	12.88	11.66
% Fe	52.6	49.8	49.7	49.4	49.9	51.0
% S	30.8	29.3	30.6	27.4	27.2	28.6
To Regenerator:						
Fe, Cake	5.448	5.070	5.487	5.524	6.028	5.124
Fe, Makeup	0.453	0.453	0	0.483	0.340	0.823
Fe, Vessel Walls	0.078	0.078	0	0	0.059	-
Fe Total (lbs)	<u>5.979</u>	<u>5.601</u>	<u>5.487</u>	<u>6.007</u>	<u>6.427</u>	<u>5.947</u>
S, Cake	3.197	2.830	3.175	3.308	3.606	2.798
S, Makeup	0.525	0.525	0	0	0.394	0.394
S Total (lbs)	<u>3.722</u>	<u>3.355</u>	<u>3.175</u>	<u>3.308</u>	<u>4.000</u>	<u>3.192</u>
From Regenerator:						
Fe Discharge (lbs)	6.017	5.603	5.482	6.007	6.427	5.947
S, Discharge	3.524	3.296	3.375	3.332	3.503	3.335
S, Dry Trap	0.005	0.028	0.009	0.006	0.006	0.004
S, Wet Trap	0.011	0.059	0.046	0.029	0.020	0.008
S, Vent Gas	0.001	0.007	0.001	0.001	0.009	0.012
S Total (lbs)	<u>3.541</u>	<u>3.390</u>	<u>3.431</u>	<u>3.368</u>	<u>3.538</u>	<u>3.359</u>

DISCUSSION OF TEST RESULTS

Reaction Rates of SO₂ and NO_x with FeS

a. SO₂ Reaction with FeS

The first important mechanism is the absorption of sulfur dioxide gas into the aqueous medium. During this study, the measured average SO₂ absorption rates per cycle were 1.70 to 4.22 x 10⁻⁵ gm·moles per liter per second. Upon absorption sulfurous acid is formed and, above pH 1.9, dissociates to H⁺ and HSO₃⁻ ions.⁽⁴⁾ In fact, at the pH range of 4 to 5, HSO₃⁻ is the dominant sulfite species. The reaction rates for the dissociation of sulfurous acid and the association of bisulfite are extremely fast, on the order of 10⁻⁴ to 10⁻⁶ seconds.⁽⁴⁾

In addition to the sulfur dioxide absorption process, there is also the dissolution of oxygen from the flue gas. The amount of oxygen in solution is important since it plays a role in determining the kinetic path taken by the slurry.⁽⁵⁾ Probably neither of these solvation processes are rate controlling in the system.

After dissolution, one of the important subsequent reactions is the oxidation of the bisulfite ion in the following manner:

Rand and Gale⁽⁶⁾ found that their reaction rates were well correlated to the sulfite concentration. At 20°C, for a pH range of 6 to 8, an initial dissolved oxygen of 7.7 mg/l, and a sulfite concentration between 4 to 18x10⁻⁵ moles/liter, the rates were in the range of 1.2 to 6.7x10⁻⁶ moles/liter/second. The reaction is obviously relevant to the optimization of the process since the oxygen undoubtedly competes with the ferrous sulfide for the available sulfite. This study did not specifically investigate the reaction rates for sulfite-bisulfite oxidation.

There are three relevant heterogeneous solid-liquid reactions to the sulfur dioxide-iron sulfide system:

1. Hydrolysis of FeS: $\text{FeS} + 2\text{H}^+ \rightarrow \text{Fe}^{2+} + \text{H}_2\text{S}$
(with subsequent oxidation of the H_2S)
2. FeS- SO_2 reaction: $3\text{FeS} + 2\text{H}^+ + 2\text{HSO}_3^- \rightarrow 2\text{FeS}_2 + \text{Fe}^{2+} + \text{SO}_4^{2-} + 2\text{H}_2\text{O}$
(where the reaction is simplified by using FeS and FeS_2)
3. Oxidation of the FeS: $\text{FeS} + 2\text{O}_2 \rightarrow \text{Fe}^{2+} + \text{SO}_4^{2-}$

There are some preliminary data on Reaction 1, the hydrolysis of FeS. One study used mackinawite (Fe_9S_8),⁽⁷⁾ and the other study used troilite (FeS).⁽⁸⁾ Both studies were conducted in a pH range of 3 to 7 and yielded the following rate law for hydrolysis:

$$\frac{dC}{dt} \text{Fe} = k_1 (\text{H}^+) \frac{A}{V}$$

where $\frac{A}{V}$ is the specific area of solids.

The rate constant k_1 was evaluated in both studies at 25°C:

$$k_1 = 2.7 \times 10^{-6} \text{ meter/sec (troilite)}$$

$$k_1 = 40.0 \times 10^{-6} \text{ meter/sec (mackinawite)}$$

The measured difference in the rate constants for the troilite and mackinawite are consistent with past data that indicate that iron sulfide reactivity is inversely proportional to its sulfur content. The rate constant k_1 for pyrrhotite, the compound generated by the thermal dissociation of pyrite used in this study, was not measured. However, based on the relative rates for the troilite and mackinawite, its hydrolysis rate constant is estimated at approximately 1.8×10^{-7} meters per second.

The rate of iron dissolution for a number of tests was compared to the aforementioned rate law for hydrolysis. Although the rate of iron dissolution is influenced by all three reactions and, therefore, should be affected by factors other than just the hydrogen ion concentration and the $\frac{A}{V}$ term, a relatively consistent rate constant was measured. The average rate constant for the whole system was

$$k_{\text{total}} = 9.9 \times 10^{-6} \text{ meter/sec.}$$

This value is approximately 55 times larger than that which would be expected by simple hydrolysis of the pyrrhotite. Reactions 2 and/or 3 must, therefore, play a significant role in the dissolution of iron.

Reaction 2 is the central reaction in the SULF-X Process. The most detailed work on the reaction rates of the FeS-SO₂ reaction has been carried out by Kunda and Rudyk using pyrrhotite.⁽⁹⁾ The main problem with their work is that they used highly acidic solutions (pH < 1). Nonetheless, there are some important observations which can be made from their raw data. The data suggest that the kinetics of Reaction 2 is increased by using more pure pyrrhotite, increasing the wt % of FeS in the slurry, increasing P_{SO₂} and increasing temperature. Of these variables, only the temperature of the slurry was intentionally changed during the study. Increasing the slurry temperature improved reagent utilization although the NO_x absorption rate decreased.

The rate of oxidation of FeS by dissolved oxygen in Reaction 3 has not been accurately measured. The reaction

is favored overwhelmingly to the right thermodynamically ($K_{\text{eq}} = 10^{172}$; Naumov et. al., 1974),⁽¹⁰⁾ yet kinetic data are definitely lacking. This reaction may have a significant effect on the efficiency of the FeS, depending on the O₂ content of the solution.

Investigators⁽⁹⁾ have attempted to study the kinetics of the FeS-SO₂ systems by following the rate of formation of dissolved iron. However, the results obtained during this study indicate that the pH endpoint of an absorption run has a significant influence on the average rate of iron dissolution. Whereas the rate of iron dissolution, as measured by the increase in ferrous ion concentration, averaged 5×10^{-4} gm·moles/second for runs terminated at pH 5.0, the overall rate decreased to 9×10^{-5} gm·moles/second for runs terminated at pH 4.5. This would indicate that the oxidation rate of the ferrous ion, and therefore its loss from solution, increases at lower pH values.

b. NO_x absorption with FeS

It was observed during earlier studies that nitric oxide was effectively absorbed by an iron sulfide slurry.⁽³⁾ Visual examination of the used slurry indicated a brown discoloration in some of the samples. This was probably due to the formation of nitroso compounds that were not completely reduced. However, an increased concentration of nitrogen was detected in the test gas from the absorber, thereby indicating the total or partial reduction of the nitroso compounds to nitrogen.

The rate at which nitrogen was absorbed was found to be directly proportional to the ferrous ion concentration in solution. Figure 3 is a graph of the nitric oxide removal and ferrous ion concentration relationship. Curves are given for three temperature levels. The data used to develop these curves were obtained from earlier laboratory studies.⁽³⁾ The data plotted along with the curves were obtained during the 20 cfm tests. The recent findings are consistent with earlier results and indicate that the relatively low nitric oxide removal rates are largely a result of relatively low ferrous ion concentrations in the slurry.

Elemental Balances

Slurry samples were taken immediately before and after each test from the recirculation tank. Results are presented in Tables 5, 5X, 6 and 6X. The recirculation pump and mixer were operating during sampling to maintain homogenous conditions. The fresh spent slurries for each test were analyzed for iron and sulfur in both the dissolved and solid forms. The quantity ΔFe_{total} is the calculated difference between the total iron in the fresh slurry and the total iron in the spent slurry. The relative percent difference was calculated from the ΔFe_{total} value divided by the average total iron from the fresh and spent slurries:

$$Fe_{total} = Fe_{soluble} + Fe_{insoluble}$$

$$Fe_{total} = Fe_{total \text{ in spent slurry}} - Fe_{total \text{ in fresh slurry}}$$

$$\text{Relative \%} = \left(\frac{Fe_{total}}{\frac{Fe_{total \text{ in fresh slurry}} + Fe_{total \text{ in spent slurry}}}{2}} \right) \times 100\%$$

A similar procedure was used for the calculation of the quantities of sulfur. The quantity of sulfur removed from the flue gas was added to the sulfur in the fresh slurry for the material balance calculations:

$$S_{total \text{ in fresh slurry}} = S_{soluble} + S_{insoluble} + S_{from SO_2}$$

$$S_{total \text{ in spent slurry}} = S_{soluble} + S_{insoluble}$$

The ΔFe total in A1 through A12 averages +1.4% and averages 2.0% in B1 to B12. The material balance was based on analyses of the slurry, and the total amounts of Fe were calculated from the analysis and volume in the recycle tank. With a 50-gallon tank, a change in volume of one gallon due to evaporation, dilution or sampling would account for a difference of 2% in the total amount of Fe as calculated in the material balance.

The ΔS_{total} in tests A1 to A12 has an average relative value of -12.7 gms or -0.7% and an average is -197.4 gms or -6.2% in tests B1 through B12.

The general loss of sulfur (ΔS_{total}) cannot be explained by the generation of H_2S . For example, in test B5, ΔS_{total} is gm'moles which is equivalent to 140 gms of H_2S or 99.2 liters at 1 atmosphere and 70°F . This would amount to an average concentration of 1,016 ppm in the $3,448 \text{ ft}^3$ (97,619 liters) total gas flow through the system. Periodic checks of the outlet gas failed to detect any H_2S gas.

The slurries were analyzed for insoluble sulfur in the forms of $\text{SO}_4^{=}$ and $\text{SO}_3^{=}$. Other soluble species, such as thiosulfate, may have caused interference in the analyses or gone undetected in the spent slurry. In some of the tests, particularly those after A6 in which the heater was installed in the recirculation tank, colloidal sulfur had formed producing a cloudy supernate, and a thin layer of sulfur was noticed on top of the settled iron sulfide. This colloidal sulfur may have remained with the filtrate and not been detected in analysis.

Although great care was used to prevent oxidation of the solids during vacuum drying and weighing, finely ground FeS will oxidize rapidly and sulfur may have been lost from the solids by oxidation to SO_2 .

FeS Capacity for SO_2

The capacity of the iron sulfide for sulfur dioxide, calculated on the molar ratio of absorbed SO_2 to insoluble solids (insoluble solids assumed to be 100% FeS) is shown in Figure 4 for each of the absorption runs in Test Series A and B.

Tables 3 and 4, Regeneration - Material Balance, indicate that the composition of the regenerated solids contained increasing amounts of inert matter. As a result, the iron sulfide capacities presented in Figure 4 do not really reflect the capacity of the iron sulfide but rather, the capacity of the total insoluble solids, some of which are inert matter with no activity toward SO_2 . In a study by Kunda and Rudyk,⁽⁸⁾ iron sulfide reagent was prepared by thermal decomposition of pyrite at 1300°F in a neutral atmosphere. The resulting sulfur/iron molar ratio was approximately 1.15. The initial reagent preparation during PENSYS study produced similar results. Subsequent regenerations at first produced iron sulfide with sulfur in excess of the 1.15 ratio, but then, by the fourth cycle, the sulfur content was lower than the 1.15 ratio. Normally, that would be an indication that the more reactive troilite, FeS , was being generated. However, it appears that the excess iron was not chemically bound with the sulfur but with some of the inerts that were present. Since the chemical analysis did not differentiate between iron compounds or sulfur compounds, there was no way to accurately determine the percentage of iron sulfide in the solids. However, a procedure was used to estimate the percentage of chemically bound iron and sulfur in the solids, and then that estimated value was used to recalculate the capacity of the iron sulfide for sulfur dioxide. Those recalculated values were then plotted per cycle for A and B Series and are presented in Figure 5.

Energy Balance - Regeneration

There are two process operations in the SULF-X system that require energy in the form of heat — drying of the spent solids and reactivation (regeneration) of these solids to a reusable iron sulfide compound. A description of the drying/regenerating equipment was presented earlier.

The energy required to dry the spent solids after the filtering or centrifuging step is primarily a function of the amount of moisture retained in the dewatered cake. The determination of how much energy is needed to evaporate a known quantity of water is a straightforward calculation. The determination of the relative influence on this value by other parameters, such as particle size of the solids and the concentration and nature of the dissolved solids, was not part of this investigative effort and should be addressed in future studies.

The amount of energy needed to heat the dry solids to 750°C and then to maintain that temperature for 3 hours was measured. For sample run A6R, it took 3 hours and 6 minutes to heat the furnace to 750°C. For the next 3 hours, the thermostatic controller kept the furnace on 71% of the time. The total energy expended for the total 6-hour operation was 92,990 BTU. However, a significant portion of the energy used went into heating both the furnace and retort. In addition, there were continuous radiative, conductive and convective external heat losses to the surroundings and internal heat losses to the purge gas.

These miscellaneous heat losses are a function of test equipment design and operating procedures and, therefore, not a true indication of the net energy required to regenerate the solids and vaporize the elemental sulfur. In order to correct for these heat losses, a blank test was conducted without the spent solids but with all other controllable conditions kept the same as those for sample run A6R. The difference in energy consumed between these two runs represents the net energy needed for the regeneration process.

Both runs were begun at room temperature with the furnace controller set at 750°C. The type of purge gas used for each run was the same as were the flow rates. The furnace remained on continuously until 750°C was attained and then began cycling on and off until the end of each run. Compared to the data for sample run A6R, it took the empty system 3 hours and 16 minutes to reach 750°C and then the furnace was on for 70.3% of the time for the next 3 hours.

Although a comparison of the energies consumed during run A6R and the blank run indicates an exothermic reaction for the overall cycle, a review of the values for the heating up phase and for the 750°C regenerating phase indicates apparent exothermic and endothermic reactions, respectively.

If the following insoluble solids are formed when ferrous sulfide reacts with sulfur dioxide and oxygen

then, theoretically, it should take 367.5 BTU/lb to regenerate the product solids to ferrous sulfide. Since there were 10.16 pounds of regenerated solids from A6R, the next amount of energy needed to regenerate this batch would have been 3,734 BTU. However, based on the absorption data, the reaction did not go to completion according to the above reaction. Adjusting the theoretical value to the measured degree of reaction indicates that 162.7 BTU/lb, or a total of 1,653 BTU, should have been absorbed by the solids during regeneration.

During the heating up phase, the miscellaneous oxidation of sulfur and carbon may be a significant source of heat. The coal, which was added to the spent solids as a reductant, contained both sulfur and oxygen. Atmospheric oxygen may also have been present within the bed of solids. There is evidence that at temperatures below 750°C, both sulfur dioxide and carbon dioxide were generated within the retort undoubtedly accounting for the generation of heat.

Subsequently, during the 750°C phase, a net total of 373 BTU were absorbed by the solids, which is equivalent to approximately 37 BTU/lb. This amount is significantly less than that predicted by thermodynamic methods. However, these values are a small percentage of the total, and caution must be exercised with interpreting their significance. Not only was the mass of the regeneration equipment too large compared to that of the spent solids, but the failure to exclude free oxygen from the system undoubtedly contributed to much of the exothermic reactions.

Flue Gas Impurities

The concentration of particulates in the flue gas from the furnace to the absorber was periodically checked by measuring the weight of solids accumulated within the filter in the inlet sample gas line preceding the instrumentation. The average concentration of these solids was approximately 0.013 grams/cu. ft. of flue gas. Subsequent investigations of these solids revealed that 65% by weight were soluble in ethyl alcohol. Unlike fly ash released by pulverized coal boilers, the particulates from the stoker furnace appear to consist primarily of tars and condensed volatiles produced by incomplete combustion of the coal.

A comparison of "acid insolubles" data presented in Tables 5 and 6 indicates that there is an inconsistent increase in these solids from one cycle to the next. In general, after compensation for slurry volume changes, the amount of acid insolubles increased during the absorption cycle. This increase was undoubtedly caused by the particulates in the flue gas transferring to the slurry within the absorber. The increase, based on the total volume of flue gas passed through the absorber for each cycle, ranged from 0 to 0.081 grams/cu. ft. with a repeated value of approximately 0.05 grams/cu. ft. There were significant fluctuations in the increase of acid insolubles between cycles caused by the inability to closely control the composition of the flue gases being generated.

There was also contribution of metals, probably as the corresponding sulfides, from the walls of the regenerator retort. This was evidenced by both the loss in tare weight of the retort, as presented in Table 7 and the presence of chromium within the solids, as shown in Figure 6. The walls of the retort would be the only measurable source of chromium.

The precise rate of accumulation of inert materials within the SULF-X system could not be measured due to the combined effect of physical losses during either the dewatering and solids transfer operations and the thermally induced, but not necessarily complete, loss of carbonaceous compounds during the regeneration cycle. There was no measurable adverse effect of these impurities on the ability of the system to absorb sulfur dioxide. The removal efficiency of nitrogen oxides decreased toward the latter cycles but this is believed to be caused by the decrease of ferrous ion in the slurry and not because of the impurities.

FIGURE 4

UTILIZATION VALUE
BASED ON INSOLUBLE SOLIDS WEIGHT
(ASSUMING SOLIDS ARE
100% IRON SULFIDE)
 SO_2/FeS MOLAR RATIO

A SERIES
B SERIES

FIGURE 5

UTILIZATION VALUE
BASED ON
CALCULATED IRON SULFIDE COMPOSITION
 SO_2/FeS MOLAR RATIO

FIGURE 6

ELEMENTAL COMPOSITION
OF
THERMALLY REGENERATED IRON SULFIDES
CONTAMINATED WITH ELEMENTS
FROM
REGENERATOR VESSEL

CURSOR: 6.340KEV =2650

TABLE 5
Iron-Sulfur Balance
A Series

Sample No.	A1	A2	A3	A4	A5	A6
Date	<u>5/23/79</u>	<u>6/01/79</u>	<u>6/07/79</u>	<u>6/13/79</u>	<u>6/19/79</u>	<u>6/25/79</u>
Feed Slurry:						
Fe, insol	8,320	10,450	12,660	11,170	9,970	-
Fe, sol	19	700	2,250	2,600	4,740	-
Fe total (ppm _w)	<u>8,339</u>	<u>11,150</u>	<u>14,910</u>	<u>13,770</u>	<u>14,720</u>	<u>-</u>
S, insol	5,490	7,890	9,150	6,870	6,430	-
S, sol	190	580	1,970	2,420	2,880	-
S, total (ppm _w)	<u>5,680</u>	<u>8,470</u>	<u>11,120</u>	<u>9,290</u>	<u>9,310</u>	<u>-</u>
TDS	360	1,970	6,590	8,980	8,420	-
TSS	14,000	18,600	22,100	20,500	20,100	-
Tot. Solids (ppm _w)	<u>14,360</u>	<u>20,570</u>	<u>28,690</u>	<u>29,480</u>	<u>28,520</u>	<u>-</u>
HCl Insol (ppm _w)	-	-	-	476	2,050	-
Spent Slurry:						
Fe, insol	6,670	9,950	10,880	9,620	10,220	11,150
Fe, sol	1,250	1,600	4,500	3,800	5,500	3,750
Fe, total (ppm _w)	<u>7,920</u>	<u>11,550</u>	<u>15,380</u>	<u>13,420</u>	<u>15,720</u>	<u>14,900</u>
S, insol	4,780	7,790	9,990	7,140	6,270	7,140
S, sol	750	1,250	2,740	3,220	3,050	5,300
S, total (ppm _w)	<u>5,530</u>	<u>9,040</u>	<u>12,730</u>	<u>10,360</u>	<u>9,320</u>	<u>12,440</u>
TDS	3,180	4,650	8,770	11,320	9,390	11,430
TSS	11,600	18,000	21,300	18,600	19,400	21,200
Tot. Solids (ppm _w)	<u>14,780</u>	<u>22,650</u>	<u>30,070</u>	<u>29,920</u>	<u>28,790</u>	<u>32,630</u>
HCl insol (ppm _w)	-	-	-	519	1,668	2,099
Feed Slurry S as SO ₃	9.6	342	979	1,133	1,126	-
Spent Slurry S as SO ₃	446	1,021	1,289	1,258	1,380	1,480

TABLE 5 (Cont.)

Iron-Sulfur Balance
A Series

Sample No.	A7	A8	A9	A10	A11	A12
Date	<u>6/29/79</u>	<u>7/09/79</u>	<u>7/13/79</u>	<u>7/19/79</u>	<u>7/25/79</u>	<u>8/01/79</u>
Feed Slurry:						
Fe, insol	11,840	11,800	24,980	24,260	10,540	13,670
Fe, sol	4,100	5,500	5,700	8,300	6,900	8,400
Fe total (ppm _w)	<u>15,940</u>	<u>17,300</u>	<u>30,680</u>	<u>32,560</u>	<u>17,440</u>	<u>22,070</u>
S, insol	6,780	7,250	15,110	14,357	5,710	7,610
S, sol	3,390	5,040	6,370	7,170	1,470	1,500
S total (ppm _w)	<u>11,270</u>	<u>12,290</u>	<u>21,480</u>	<u>21,527</u>	<u>7,180</u>	<u>9,110</u>
TDS	12,840	14,640	17,640	31,160	39,440	39,200
TSS	22,000	22,600	48,600	49,000	22,060	28,600
Tot. Solids (ppm _w)	<u>34,840</u>	<u>37,240</u>	<u>66,240</u>	<u>80,160</u>	<u>61,500</u>	<u>67,800</u>
HCl insol (ppm _w)	2,288	1,821	3,402	4,606	3,750	4,290
Spent Slurry:						
Fe, insol	11,520	10,650	23,440	16,970	11,950	14,010
Fe, sol	4,600	4,500	7,500	5,600	7,400	9,350
Fe total (ppm _w)	<u>16,120</u>	<u>15,150</u>	<u>30,940</u>	<u>22,570</u>	<u>19,350</u>	<u>23,360</u>
S, insol	6,700	6,590	13,370	9,500	6,600	8,260
S, sol	4,900	4,750	6,540	5,520	1,510	1,700
S total (ppm _w)	<u>11,600</u>	<u>11,340</u>	<u>19,910</u>	<u>15,020</u>	<u>8,110</u>	<u>9,960</u>
TDS	15,540	15,730	28,000	21,840	38,800	43,750
TSS	21,900	20,400	46,600	35,200	25,000	30,600
Tot. Solids (ppm _w)	<u>37,440</u>	<u>36,130</u>	<u>74,600</u>	<u>57,040</u>	<u>63,800</u>	<u>74,350</u>
HCl insol (ppm _w)	2,278	2,013	4,474	4,259	3,750	5,202
Feed Slurry S as SO ₃	1,539	1,544	1,404	1,220	-	-
Spent Slurry S as SO ₃	1,553	1,285	982	632	-	-

TABLE 5 X

Iron-Sulfur Balance
A Series

Sample No.	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10	A11	A12
Date	<u>5/23/79</u>	<u>6/01/79</u>	<u>6/07/79</u>	<u>6/13/79</u>	<u>6/19/79</u>	<u>6/25/79</u>	<u>6/29/79</u>	<u>7/09/79</u>	<u>7/13/79</u>	<u>7/19/79</u>	<u>7/25/79</u>	<u>8/01/79</u>
Feed Slurry:												
Fe, insol	28.2	37.5	43.8	37.1	37.8	-	44.1	42.4	42.3	42.7	27.1	33.4
Fe, sol	0.1	2.5	7.8	8.6	18.0	-	15.3	19.8	9.7	14.6	17.8	20.5
Fe total (gm·mols)	<u>28.3</u>	<u>40.0</u>	<u>51.6</u>	<u>45.7</u>	<u>45.8</u>	<u>-</u>	<u>59.4</u>	<u>62.2</u>	<u>52.0</u>	<u>57.3</u>	<u>44.9</u>	<u>53.9</u>
S, insol	32.4	49.4	55.1	39.7	42.5	-	44.0	45.4	44.6	44.1	25.6	32.3
S, sol	1.1	3.6	11.9	14.0	19.0	-	22.0	31.5	18.8	22.0	6.6	6.4
S, absorbed	2.3	4.0	2.8	3.4	2.5	-	2.7	2.9	1.8	0.4	1.8	3.3
S total (gm·mols)	<u>35.8</u>	<u>57.0</u>	<u>69.8</u>	<u>57.1</u>	<u>64.0</u>	<u>-</u>	<u>68.7</u>	<u>79.8</u>	<u>65.2</u>	<u>66.5</u>	<u>34.0</u>	<u>42.0</u>
37												
S/Fe (molar)	1.19	1.32	1.30	1.18	1.10	-	1.23	1.24	1.22	1.15	0.72	0.72
Spent Slurry:												
Fe, insol	24.0	34.4	36.1	35.2	38.1	41.6	41.4	43.3	39.7	43.7	29.2	32.3
Fe, sol	4.5	5.5	14.9	13.9	20.5	14.0	16.5	18.3	12.7	14.4	18.1	21.5
Fe total (gm·mols)	<u>28.5</u>	<u>39.9</u>	<u>51.0</u>	<u>49.1</u>	<u>58.6</u>	<u>55.6</u>	<u>57.9</u>	<u>61.6</u>	<u>52.4</u>	<u>58.1</u>	<u>47.3</u>	<u>53.8</u>
S, insol	29.9	46.9	57.8	45.5	40.7	46.4	41.9	46.7	39.5	42.6	28.1	33.2
S, sol	4.7	7.5	15.9	20.5	19.8	34.4	30.7	33.6	19.3	24.8	6.4	6.8
S total (gm·mols)	<u>34.6</u>	<u>54.4</u>	<u>73.7</u>	<u>66.0</u>	<u>60.5</u>	<u>80.8</u>	<u>72.6</u>	<u>80.3</u>	<u>58.8</u>	<u>67.4</u>	<u>34.5</u>	<u>40.0</u>
S/Fe (molar)	1.22	1.36	1.44	1.34	1.03	1.45	1.25	1.30	1.19	1.16	0.79	0.74

TABLE 6

Iron-Sulfur Balance
B Series

Sample No.	B1	B2	B3	B4	B5	B6
Date	<u>5/25/79</u>	<u>6/05/79</u>	<u>6/11/79</u>	<u>6/15/79</u>	<u>6/21/79</u>	<u>6/27/79</u>
Feed Slurry:						
Fe, insol	9,660	9,770	12,350	12,410	14,120	14,760
Fe, sol	10	900	3,250	3,000	3,850	4,450
Fe total (ppm _w)	<u>9,670</u>	<u>10,670</u>	<u>15,600</u>	<u>15,410</u>	<u>17,970</u>	<u>19,210</u>
S, insol	6,710	7,530	8,130	8,580	8,860	8,680
S, sol	160	1,060	2,500	3,250	4,640	5,610
S total (ppm _w)	<u>6,870</u>	<u>8,590</u>	<u>10,630</u>	<u>11,830</u>	<u>13,500</u>	<u>14,290</u>
TDS	350	3,090	10,140	12,640	14,110	14,320
TSS	16,600	17,600	22,220	23,200	17,000	28,000
Total Solids (ppm _w)	<u>16,950</u>	<u>20,690</u>	<u>32,360</u>	<u>35,840</u>	<u>41,110</u>	<u>42,320</u>
HCl, insol (ppm _w)	-	-	962	1,020	1,593	3,789
Spent Slurry:						
Fe, insol	7,810	9,290	11,070	11,440	13,670	14,470
Fe, sol	1,150	2,000	3,350	3,950	4,950	4,950
Fe total (ppm _w)	<u>8,960</u>	<u>11,290</u>	<u>14,420</u>	<u>15,390</u>	<u>18,620</u>	<u>19,420</u>
S, insol	5,940	7,240	6,630	7,590	8,490	8,490
S, sol	530	1,560	3,400	4,530	5,500	6,650
S total (ppm _w)	<u>6,480</u>	<u>8,800</u>	<u>10,030</u>	<u>12,120</u>	<u>13,990</u>	<u>15,140</u>
TDS	3,280	6,610	13,240	12,980	16,240	14,920
TSS	14,000	16,800	10,090	22,600	26,600	28,200
Total Solids (ppm _w)	<u>17,280</u>	<u>23,410</u>	<u>23,330</u>	<u>35,580</u>	<u>42,840</u>	<u>43,120</u>
HCl, insol (ppm _w)	-	-	1,770	1,989	2,633	2,933
Feed S as SO ₃	9.6	600	1,301	1,541	1,679	1,582
Spent S as SO ₃	619	1,032	1,358	1,709	1,564	1,469

TABLE 6 (Cont.)

Iron-Sulfur Balance
B Series

Sample No.	B7	B8	B9	B10	B11	B12-1	B12-2
Date	<u>7/03/79</u>	<u>7/11/79</u>	<u>7/17/79</u>	<u>7/23/79</u>	<u>7/30/79</u>	<u>8/03/79</u>	<u>8/03/79</u>
Feed Slurry:							
Fe, insol	15,830	12,900	14,260	15,860	17,140	9,720	9,640
Fe, sol	30,800	31,000	30,000	30,000	28,350	27,300	3
Fe total (ppm _w)	<u>46,630</u>	<u>43,900</u>	<u>44,260</u>	<u>45,860</u>	<u>45,490</u>	<u>37,020</u>	<u>9,943</u>
S, insol	9,270	7,590	8,780	8,800	9,340	5,800	5,590
S, sol	24,300	20,500	19,200	19,500	20,500	18,100	84
S total (ppm _w)	<u>33,570</u>	<u>28,090</u>	<u>27,980</u>	<u>28,300</u>	<u>29,840</u>	<u>23,900</u>	<u>5,674</u>
TDS	120,300	103,000	99,800	98,500	97,500	95,100	500
TSS	30,100	25,900	28,700	32,100	34,360	20,000	19,200
Total Solids (ppm _w)	<u>150,400</u>	<u>128,900</u>	<u>128,500</u>	<u>130,600</u>	<u>131,860</u>	<u>115,100</u>	<u>19,700</u>
HCl, insol (ppm _w)	3,492	2,305	2,985	4,943	3,780	2,000	1,632
Spent Slurry:							
Fe, insol	11,950	12,270	13,750	15,340	16,360	9,540	7,520
Fe, sol	34,000	31,000	30,300	30,100	28,550	26,850	2,000
Fe total (ppm _w)	<u>45,950</u>	<u>43,270</u>	<u>44,050</u>	<u>45,440</u>	<u>44,910</u>	<u>36,390</u>	<u>9,520</u>
S, insol	6,670	7,100	8,230	9,180	9,510	5,740	3,890
S, sol	23,000	21,000	19,800	20,500	20,900	18,500	2,400
S total (ppm _w)	<u>29,670</u>	<u>28,100</u>	<u>28,030</u>	<u>29,680</u>	<u>30,410</u>	<u>24,240</u>	<u>6,290</u>
TDS	109,000	102,000	102,000	102,000	101,700	100,560	8,100
TSS	24,000	25,100	28,000	32,100	36,440	20,000	15,700
Total Solids (ppm _w)	<u>133,000</u>	<u>127,100</u>	<u>130,000</u>	<u>134,100</u>	<u>138,140</u>	<u>120,560</u>	<u>23,800</u>
HCl, insol (ppm _w)	1,284	1,581	2,856	4,815	6,195	2,000	1,884
Feed S as SO ₃	1,285	1,307	1,631	1,782	-	-	-
Spent S as SO ₃	1,588	1,350	1,782	1,804	-	-	-

TABLE 6X

Iron-Sulfur Balance
B Series

Sample No.	B1	B2	B3	B4	B5	B6
Date	<u>5/25/79</u>	<u>6/05/79</u>	<u>6/11/79</u>	<u>6/15/79</u>	<u>6/21/79</u>	<u>6/27/79</u>
Feed Slurry:						
Fe, insol	32.7	32.4	39.3	42.9	47.8	48.0
Fe, sol	<u>0.0</u>	<u>3.0</u>	<u>10.4</u>	<u>10.4</u>	<u>13.0</u>	<u>14.5</u>
Fe total (gm·mols)	32.7	35.4	49.7	53.3	60.8	62.5
S, insol	39.6	43.6	45.1	51.7	52.3	49.2
S, sol	0.9	6.1	13.9	19.6	27.4	31.8
S, absorbed	<u>3.2</u>	<u>3.8</u>	<u>3.1</u>	<u>2.7</u>	<u>3.7</u>	<u>3.6</u>
S total (gm·mols)	43.7	53.5	62.1	74.0	83.4	84.6
S/Fe (molar)	1.24	1.40	1.19	1.34	1.31	1.30
Spent Slurry:						
Fe, insol	25.9	29.6	38.3	38.8	44.5	46.1
Fe, sol	<u>3.8</u>	<u>6.4</u>	<u>11.6</u>	<u>13.4</u>	<u>16.1</u>	<u>15.8</u>
Fe total (gm·mols)	29.7	36.0	49.9	52.2	60.6	61.9
S, insol	34.4	40.2	39.9	44.8	48.1	47.1
S, sol	<u>3.1</u>	<u>8.7</u>	<u>20.5</u>	<u>26.7</u>	<u>31.2</u>	<u>36.9</u>
S total (gm·mols)	37.5	48.9	60.4	71.5	79.3	84.0
S/Fe (molar)	1.26	1.36	1.21	1.37	1.31	1.36

TABLE 6X (Cont.)

Iron-Sulfur Balance
B Series

Sample No.	B7	B8	B9	B10	B11	B12-1	B12-2
Date	<u>7/03/79</u>	<u>7/11/79</u>	<u>7/17/79</u>	<u>7/23/79</u>	<u>7/30/79</u>	<u>8/03/79</u>	<u>8/03/79</u>
Feed Slurry:							
Fe, insol	57.9	44.6	47.4	50.5	52.3	29.6	29.4
Fe, sol	113.7	107.1	99.6	95.6	86.5	83.3	0.0
Fe total (gm·mols)	<u>170.6</u>	<u>151.7</u>	<u>147.0</u>	<u>146.1</u>	<u>138.8</u>	<u>112.9</u>	<u>29.4</u>
S, insol	59.1	45.7	50.8	48.8	49.6	30.8	29.7
S, sol	154.9	123.4	111.1	108.2	108.9	96.2	0.4
S, absorbed	0.3	0.5	0.3	2.0	4.2	2.1	7.7
S total (gm·mols)	<u>214.3</u>	<u>169.6</u>	<u>162.2</u>	<u>159.0</u>	<u>162.7</u>	<u>129.1</u>	<u>37.8</u>
S/Fe (molar)	1.25	1.11	1.10	1.08	1.14	1.12	1.03
Spent Slurry:							
Fe, insol	43.7	40.7	43.8	46.8	47.7	28.4	21.9
Fe, sol	124.4	102.9	96.5	91.8	83.2	80.1	5.8
Fe total (gm·mols)	<u>168.1</u>	<u>143.6</u>	<u>140.3</u>	<u>138.6</u>	<u>130.9</u>	<u>108.5</u>	<u>27.7</u>
S, insol	42.5	41.1	45.7	48.8	48.3	29.8	19.7
S, sol	146.6	121.5	109.9	108.9	106.1	96.1	12.2
S total (gm·mols)	<u>189.1</u>	<u>162.6</u>	<u>155.6</u>	<u>157.7</u>	<u>154.4</u>	<u>125.9</u>	<u>31.9</u>
S/Fe (molar)	1.12	1.13	1.11	1.14	1.18	1.16	1.15

TABLE 7

Miscellaneous Data

Regen. Sample No.	A	B	A1	B1	A2	E2	A3	B3	A4
Date	5/21/79	5/23/79	5/30/79	6/01/79	6/05/79	6/07/79	7/11/79	6/13/79	6/15/79
Initial Tare Vessel (lbs)	64.25	64.0	63.69	63.50	63.03	62.69	62.44	62.22	61.97
Final Tare Vessel (lbs)	64.0	63.69	63.50	63.06	62.69	62.44	62.22	61.97	61.72
Difference, loss (lbs)	0.25	0.31	0.19	0.44	0.37	0.25	0.22	0.25	0.25
Weight Wet Cake (lbs)	0	0	7.69	7.19	10.31	8.81	11.06	12.41	12.22
Weight Reductant (oz)	0	0	2	2	2	2	2	2	2
Makeup FeS ₂ (lbs)	8.0	8.0	2.50	2.44	2.38	2.56	1.0	1.0	1.0
42 Regen. FeS Dry Wt. (lbs)	.50	6.50	8.0	6.94	9.31	8.09	8.69	9.56	9.28
Milling Time (hrs)	12	12	12	12	12	12	12	12	12
pH Ball Mill Slurry	8.4	8.9	8.3	8.3	-	-	8.6	8.25	8.15
pH Filtrate/Supernate	7.2	7.2	4.45	4.97	-	4.5	4.3	4.55	4.10
pH Resultant Slurry	6.9	7.45	6.50	7.13	6.13	6.0	6.02	5.55	5.85

Table 7 (Cont.)

Miscellaneous Data

Regen. Sample No.	B4	A5	B5	A6	B6	A7	B7	A8	B8
Date	<u>6/19/79</u>	<u>6/21/79</u>	<u>6/25/79</u>	<u>6/27/79</u>	<u>6/29/79</u>	<u>7/13/79</u>	<u>7/09/79</u>	<u>7/11/79</u>	<u>7/13/79</u>
Initial Tare Vessel (lbs)	61.72	61.38	61.19	63.25	63.0	62.88	63.0	62.88	62.84
Final Tare Vessel (lbs)	<u>61.38</u>	<u>61.19</u>	<u>-</u>	<u>63.0</u>	<u>62.88</u>	<u>63.0</u>	<u>62.88</u>	<u>62.84</u>	<u>62.88</u>
Difference, loss (lbs)	0.34	0.19	-	0.25	0.12	+	0.12	0.04	+
Weight Wet Cake (lbs)	11.94	12.75	13.81	13.0	15.50	13.75	19.06	14.75	16.19
Weight Reductant (oz)	2	2	2	2	2	2	2	2	2
Makeup FeS ₂ (lbs)	1.0	1.0	1.0	1.03	1.0	1.0	1.0	0	0
Regen. FeS Dry Wt. (lbs)	9.41	9.56	10.56	10.16	11.44	10.44	11.25	10.44	11.03
Milling Time (hrs)	12	12	12	12	12	12	12	12	12
pH Ball Mill Slurry	7.85	8.35	8.4	8.6	8.45	8.36	9.05	8.8	8.05
pH Filtrate/Supernate	3.47	3.9	3.5	3.4	3.1	3.2	3.6	3.12	4.34
pH Resultant Slurry	5.61	5.9	5.45	5.43	5.33	5.55	5.02	5.35	5.28

TABLE 7 (Cont.)

Miscellaneous Data

Regen. Sample No.	A9	B9	A10	B10	A11	B11
Date	<u>7/19/79</u>	<u>7/19/79</u>	<u>7/23/79</u>	<u>7/30/79</u>	<u>7/31/79</u>	<u>8/02/79</u>
Initial Tare Vessel (lbs)	62.88	62.81	62.88	62.97	62.88	63.16
Final Tare Vessel (lbs)	<u>62.88</u>	<u>62.88</u>	<u>62.97</u>	<u>62.88</u>	<u>63.16</u>	<u>63.69</u>
Difference, loss (lbs)	0	+	+	0.09	+	+
Weight Wet Cake (lbs)	14.31	15.94	14.25	17.47	14.19	17.94
Weight Reductant (oz)	2	2	2	2	2	2
Makeup FeS ₂ (lbs)	0	< .75 IOD .75 FeS ₂	1	< .75 IOD .75 FeS ₂	1	< .5 IOD .5 FeS ₂
Regen. FeS Dry Wt. (lbs)	9.13	12.16	9.84	12.88	10.13	11.6
Milling Time (hrs)	12	12	12	12	12	12
pH Ball Mill Slurry	7.56	10.15	8.7	10.6	8.6	10.3
pH Filtrate/Supernate	4.05	4.4	3.45	3.7	3.2	3.5
pH Resultant Slurry	4.89	5.6	5.50	5.8	5.78	5.54

OBSERVATIONS AND RECOMMENDATIONS

The operation of the 20-cfm test system, with some modifications proved adequate for the present study. However, there was insufficient time to increase the performance of each of the unit operations that comprised the test system. Because the system was operated in a closed-loop, totally recycled mode, problems in one unit operation impacted on each subsequent operation, making performance evaluation of the system difficult.

The following recommendations are presented in the hope of improving the test system to provide a more consistent operation and more reproducible data. Many problems appeared to be a direct result of the small size of the system components.

The combustion unit generating the flue gas performed inconsistently. A major problem associated with the unit was that it was unable to maintain a constant SO_2 concentration in the flue gas. Because the SO_2 , and also the CO and NO_x , concentrations in the flue gas were uncontrollable, it was impossible to determine the effects of various concentrations of these gases on reagent activity. Better control should be maintained over those gases in future studies. It is doubtful that this fine control can be maintained using a small domestic heating unit, like the one used in this study, because units of this type do not have adequate controls to optimize the various gaseous concentrations in the flue gas.

The flow through the absorber was a side stream taken from the main flue gas stream that was to be controlled by a positive displacement vacuum pump located on the discharge side of the absorber. Pressure drop across the pump was to be correlated with a pump performance curve indicating flow for that pressure drop. The vacuum pump was equipped with cartridge filters up- and down-stream from the unit that continually plugged with particulate and moisture carryover from the absorber. This resulted in misleading gas flow measurements.

There were no significant problems detected in the performance of the absorber. However, there was no way to sample either the gas or liquid between the various trays so that individual tray performance could be evaluated. The optimum number of trays required to consistently remove prescribed levels of SO_2 and NO_x could not be determined. It is recommended that studies be conducted to determine the optimum number of trays required to remove both SO_2 and NO_x .

During the testing, the initial and spent slurries were checked periodically for magnetic response. There was slight to moderate magnetic response in almost every case. In those amples where the reagent was regenerated using a CO in argon or nitrogen atmosphere, there was no magnetic response. The type of purge gas used during regeneration appears to have an effect on the magnetic response of the regenerated matter. Further study of the effect of various purge gases could be useful.

Problems in regenerator and ancillary equipment operation may have had an adverse impact on the recycled reagent. The regenerator vessel was equipped with a stirrer that, because of its design, became inoperative when the unit exceeded 600° operating temperature. As a result, the material was not agitated while in the regenerator. In future studies, the vessel design should be changed so that maximum heat transfer and homogenous conditions can be obtained throughout the material.

A liquid sampling train was installed (refer to Figure 4A) to trap the vent gases from the regenerator in an effort to identify and account for all sulfur materials. The installation was necessary because in initial runs, the sulfur in the gas had plugged up sampling lines to the continuous analyzers. After appropriate modifications, the final two cycles were run with the off-gas samples being piped to the continuous analyzers used on the absorption runs. The sulfur losses in the vent gases proved to be minimal; however, not all the sulfur in the system could be accounted for. There may be some form of non-condensable sulfur leaving the system in the vent gas that was not detected by the instrumentation being used. The latter problem points up the need for better sampling techniques and more comprehensive analytical procedures.

In conclusion, the original test procedure had to be abandoned due to the inability to identify the effects caused by various parameter changes, some intentional and some uncontrollable. A change in one part of the system affected the entire system and the effects of the change became compounded with each recycle. Future investigative projects should be structured to expand studies of each of the unit operations within the process.

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APPENDIX

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Figure 1A

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Figure 3A

Figure 4A

TABLE 1A

EQUIPMENT AND INSTRUMENT LIST

DOE Phase II

<u>Qty</u>	<u>Item</u>
1	GAS, GAFLO Model RBS felt bag filter, 10 micron bag, 30-40°C service mild steel.
1	Koch MVFT scrubber, 6" diameter by 15'0" high, 316 SS construction.
1	Model #727CM39 vacuum pump, Thomas Industries Power-Air Division.
1	Model #104CA14 vacuum pump, Thomas Industries Power-Air Division.
1	Makedown tank, 165-gallon round bottom fiberglass tanks 32" diameter by 50" high, with skirt equipped with hinged bolt-down cover constructed of standard resin.
1	Filtrate holding tank - same as above.
2	Decant settling tanks - same as above.
1	Scrubber recycle tank, 65-gallon round bottom fiberglass, 24" diameter by 36" high, with skirt equipped with hinged bolt-down cover constructed of standard resin.
1	Filtrate recycle tank, 100-gallon fiberglass tank, 30" diameter by 51" high, with skirt equipped with hinged bolt-down cover constructed of standard resin.
1	Model M130 Axeman-Anderson anthratube furnace with jacket and agitator.
1	Absorber recycle tank mixer, Eastern Model H-1, $\frac{1}{4}$ HP, 1750 rpm, single propeller with stabilizer, 316 SS propeller and shaft, 5/8" diameter by 30" long.
1	Makedown tank mixer, Eastern Model PH-1, $\frac{1}{4}$ HP, 1750 rpm dual propeller with stabilizer, 316SS propeller and shaft, 1" diameter by 46" long.
1	Makeup feed pump, American Lewa Model HK-1 plunger metering pump with 25MM K101 displacement plunger pump head, complete with $\frac{1}{2}$ HP, 3450 rpm, 230V, 60 cycle.
1	Filter feed pump, Teel Model #3P606, 3 HP, 3600 rpm, 230V, 60 cycle motor, TEFC motor, 15 gpm at 80" head.
1	Spent slurry blowdown pump - same as above.
1	Filtrate transfer pump, Teel ball bearing centrifugal pump Model 1P797, 1/3 HP, 1725 rpm, 115V, 60-cycle, TEFC motor, 14 gpm at 10' head.
1	Gast vacuum pump, Model No. 3040 - V 16 complete with base, V belt drive, and a 1 $\frac{1}{2}$ HP, 1800 rpm open drip-proof motor, 3-phase, 60 cycle - 230/460V.
1	Skutt kiln #183 with pyrometer and variable control.

TABLE 1 A (Cont.)

EQUIPMENT AND INSTRUMENT LIST

DOE Phase II

<u>Qty</u>	<u>Item</u>
1	Jar Mill Model 764AV complete with $\frac{1}{2}$ HP, 115V, 60-cycle single phase TEFC motor.
1	3" figure 484 bronze water jet exhauster to handle 20 ACFM of air at 20" WC requiring 73 gpm of 40 psig, 80°F water.
1	Gusher centrifugal pump, 1 $\frac{1}{2}$ " by 1 $\frac{1}{2}$ " 9 SEII, complete with 10 HP, 480V, 3-phase, 60-cycle, open drip-proof motor rated at 60 gpm of water at 80°F and 150' head.
2	Deckman Model 715 Process O ₂ analyzer with dual range, 0 to 5% and 0 to 15% O ₂ Part No. 19040.
1	Blue M utility oven, Part No. SW17Ta.
1	Model IR702 CO/CO ₂ analyzer with CO range, 0 to 1% and 0 to 3% - CO, range 0 to 6% and 0 to 20%.
1	Furnace, Lindberg lab box type with electronic controller $\pm 5^{\circ}$ C.
1	H ₂ S to SO ₂ converter, Thermoelectron Model 340 Standard.
1	NO/NO _x Chemiluminescent Analyzer, TECO Model 10A - Rack version with vacuum ^x pump, shock tray, sample pump, activated charcoal ozone absorber, accumulator, tubing and filterings.

TABLE 2A

DOE Phase II Test Schedule

Legend

A = Initial Sample

1, 2, 3, etc. = Cycle No.

R = Regeneration

W = Absorption

<u>Day</u>	<u>Date</u>	<u>Generate and Grind</u>	<u>Absorb</u>	<u>Filter and Dry</u>
T	5/22	A		
W	5/23		A1W	
T	5/24	B		
F	5/25		B1W	
W	5/30			A1
T	5/31	A1R		
F	6/01		A2W	B1
M	6/04	B1R		
T	6/05		B2W	A2
W	6/06	A2R		B2
T	6/07		A3W	B2
F	6/08	B2R		A3
M	6/11		B3W	
T	6/12	A3R		
W	6/13		A4W	B3
T	6/14	B3R		
F	6/15		B4W	A4
M	6/18	A4R		
T	6/19		A5W	B4
W	6/20	B4R		
T	6/21		B5W	A5
F	6/22	A5R		
M	6/25		A6W	B5
T	6/26	B5R		
W	6/27		B6W	A6
T	6/28	A6R		
F	6/29		A7W	B6
M	7/02	B6R		
T	7/03		B7W	A7
W	7/04	(Holiday)		
T	7/05	A7R		
F	7/06		A8W	B7

TABLE 2A (Cont.)

DOE Phase II Test Schedule

<u>Day</u>	<u>Date</u>	<u>Generate & Grind</u>	<u>Filter & Dry</u>	
M	7/09	B7R		
T	7/10		B8W	A8
W	7/11	A8R		
T	7/12		A9W	B8
F	7/13	B8R		
M	7/16		B9W	A9
T	7/17	A9R		
W	7/18		A10W	D9
T	7/19	B9R		
F	7/20		B10W	A10
M	7/23	A10R		
T	7/24		A11W	B10
W	7/25	B10R		
T	7/26		B11W	A11
F	7/27	A11R		
M	7/30		B11W	A11
T	7/31	A11R		
W	8/01		A12W	B11
T	8/02	B11R		
F	8/03		B12W	A12

TABLE 3A

Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In.</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>A-1-W</u>							
1220-1235	6.46	900	30	95.7	173.6	150	13.6
1235-1250	6.00	1594	30	98.1	208.1	198.8	4.5
1250-1305	5.88	915	30	96.7	106.9	101.9	4.7
1305-1320	5.85	1080	45	95.8	142.5	138.1	3.1
1320-1325*	5.78	1014	300	70.4	148.3	93.3	37.1
1335-1350	5.70	1350	75	94.4	154.3	195.0	-
1350-1403	5.45	1665	60	95.5	183.0	201.0	-
1425-1440	5.42	1110	30	97.3	120.0	126.3	-
1440-1458	5.11	1133	45	96.0	134.0	141.5	-
*Recycle pump not operating							
<u>A-2-W</u>							
1215-1230	6.01	622	15	97.6	146.9	150.8	-
1230-1245	5.88	1208	15	98.8	156.1	172.2	-
1245-1300	5.68	1466	15	99.0	168.9	182.2	-
1300-1315	5.41	1969	15	99.2	201.1	140.0	-
1315-1330	5.39	990	30	97.0	153.6	155.7	-
1330-1345	5.48	754	45	94.0	101.7	95.0	6.6
1345-1400	5.50	945	45	95.2	133.9	128.3	4.2
1400-1415	5.42	1298	45	96.5	154.4	150.0	2.8
1415-1430	5.44	742	45	93.9	75.6	76.9	-
1430-1445	5.40	1324	45	96.6	189.4	179.4	5.3
1445-1550	5.30	1238	45	96.4	161.7	157.8	2.4
1500-1515	5.35	1001	45	95.5	162.1	165.0	-
1515-1530	5.12	1594	45	97.2	220.0	240.0	-
1620-1635	5.34	1192	15	98.7	172.9	175.7	-
1635-1650	5.27	911	15	98.4	96.1	98.9	-
1650-1705	5.14	1759	15	99.1	212.8	227.8	-

TABLE 3A (Cont.)

Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In.</u> ²	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u> ^x	<u>ppm NO_x Out</u> ^x	<u>% NO_x Rem</u> ^x
<u>A-3-W</u>							
1020-1035	6.01	889	15	98.3	155	149.4	3.6
1035-1050	5.58	1185	15	98.7	197.5	182.5	7.6
1050-1120	5.40	731	10	98.6	161.3	178.8	-
1120-1136	5.22	1328	15	98.9	195.0	183.9	5.7
1330-1345	5.38	799	30	96.2	161.9	146.9	9.3
1345-1400	5.31	1084	30	97.2	201.9	180.0	10.8
1400-1415	5.26	896	30	96.6	173.3	143.3	17.3
1415-1430	5.23	814	30	96.3	141.7	123.9	12.6
1430-1445	5.16	1076	30	97.2	200.6	173.9	13.3
1445-1500	5.09	968	30	96.9	170.6	175.6	-
1500-1515	5.06	769	30	96.1	125.6	125.0	.5
1515-1533	5.02	1103	30	97.3	201.4	206.8	-
<u>A-4-W</u>							
1045-1100	5.74	613	15	97.6	100	90	10.0
1100-1115	5.52	1063	15	98.6	201.3	183.8	8.7
1115-1130	5.38	1564	35	97.8	293.1	279.4	4.7
1130-1145	5.25	1330	35	97.4	245.8	220.8	10.2
1145-1200	5.25	700	35	95.0	159.4	136.1	14.6
1200-1215	5.35	848	35	95.9	159.4	124.4	22.0
1215-1230	5.31	890	35	96.1	200.0	158.1	21.0
1230-1245	5.20	1204	35	97.1	260.0	193.3	25.7
1245-1300	5.20	717	35	95.1	140	130	7.1
1300-1315	5.28	890	35	96.1	153.3	150.6	1.8
1315-1330	5.26	1065	35	96.7	180.6	147.8	18.2
1330-1345	5.21	1103	35	96.8	155	151.1	2.5
1345-1400	5.19	1031	35	96.6	136.7	126.7	7.3
1400-1415	5.22	807	30	96.3	120.6	100.6	16.6
1415-1430	5.18	1443	60	95.8	248.9	200.0	19.6
1430-1445	5.12	1380	60	95.6	255	203.1	20.4
1445-1500	5.08	1178	60	94.9	208.8	181.3	13.2
1500-1510	5.03	1577	60	96.2	314	228	27.4

TABLE 3A (Cont.)

Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>A-5-W</u>							
1005-1020	5.61	717	30	95.8	179	129	28.2
1020-1035	5.35	1010	30	97.0	228	164	27.7
1035-1050	5.27	900	45	95.0	151	117	22.8
1050-1105	5.19	1072	30	97.2	191	169	11.8
1105-1120	5.08	1013	30	97.0	161	142	11.7
1120-1135	5.12	911	30	96.7	158	149	5.6
1135-1150	5.26	506	30	94.1	91	67	26.2
1150-1205	5.27	767	60	92.2	176	132	24.9
1205-1220	5.24	832	60	92.8	129	99	23.2
1220-1235	5.22	777	60	92.3	152	142	6.1
1235-1250	5.15	963	50	94.8	188	167	11.0
1250-1305	5.09	1230	50	95.9	237	214	9.6
1305-1320	5.08	1003	35	96.5	227	167	26.6
1320-1332	5.03	1384	75	94.6	308	150	51.4

TABLE 3A (Cont.)
Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>#A-6-W</u>							
1120-1135	5.57	1011	15	98.5	210	177.1	15.7
1135-1150	5.22	1393	15	98.9	250.7	212.8	15.1
1150-1205	4.96	1303	15	98.6	235.0	20.4	12.2
1205-1220	4.77	1071	15	98.6	220.7	182.1	17.5
1220-1235	4.74	715	45	93.7	137.1	80.7	41.1
1235-1250	4.74	819	45	94.5	162.8	108.6	33.3
1250-1305	4.65	1054	45	95.7	202.1	169.3	16.2
1305-1320	4.62	994	30	97.0	179.3	163.6	8.8
1420-1335	4.76	870	30	96.6	157.1	131.4	16.4
1335-1350	5.01	797	30	96.2	172.9	110	36.4
1350-1405	5.02	844	60	92.9	202.1	108.6	46.3
1405-1420	4.90	1059	60	94.3	207.1	142.9	31
-							
1500-1515	4.85	990	5	99.5	163.6	125	23.6
1515-1530	4.69	1256	10	99.2	189.3	166.4	12.1
1530-1545	4.60	1363	10	99.9	257.9	196.4	23.8
1545-1600	4.60	1089	35	96.8	237.1	170.7	28.0
1600-1615	4.62	1110	35	96.8	239.3	173.6	27.5
1615-1630	4.58	1063	15	98.6	156.4	152.1	2.7
1630-1645	4.68	1085	5	99.5	143.6	133.6	7.0
1645-1700	4.80	1033	40	96.1	153.6	131.4	14.5

TABLE 3A (Cont.)

Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>A-7-W</u>							
1000-1015	5.29	711	10	98.6	159.3	128.6	19.3
1015-1030	5.07	861	10	98.8	188.1	162.5	13.6
1030-1045	4.86	1421	10	99.3	260.6	226.6	12.9
1045-1100	4.75	1211	15	98.8	253.6	195.0	23.1
1100-1115	4.73	1041	15	98.6	211.4	145	31.4
1115-1130	4.71	1204	15	98.8	266.3	169.4	36.4
-							
1200-1215	4.94	668	10	98.5	153.1	141.3	7.7
1215-1230	4.86	724	10	98.6	131.9	130	1.4
1230-1245	4.82	947	10	98.9	173.8	166.9	4.0
1245-1300	4.81	874	45	94.8	213.6	152.1	28.8
1300-1315	4.76	969	30	96.9	237.5	185.0	22.1
1315-1330	4.74	1001	30	97.0	199.3	133.6	33.0
1330-1345	4.73	1059	15	98.6	205	153.8	25
1345-1400	4.65	1329	15	98.9	274.3	253.6	7.5
1400-1415	4.57	1305	10	99.2	256.3	223.1	13
1415-1422	4.48	1418	10	99.3	273.3	243.3	11.0

TABLE 3A (Cont.)

Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>A-8-W</u>							
1000-1015	5.46	516	10	98.1	128.3	102.5	20.0
1015-1030	5.22	956	10	99.0	211.4	184.2	12.8
1030-1045	5.04	1127	15	98.7	247.1	202.8	17.9
1045-1100	5.04	780	20	97.4	170	133.6	21.4
1100-1115	5.05	737	10	97.3	155.7	130.7	16.0
1115-1130	4.94	887	30	96.6	206.4	187.8	9.0
1130-1145	4.86	890	30	96.6	196.3	148.6	23.2
1145-1200	4.87	777	45	94.2	213.9	163.9	23.4
1300-1315	4.92	621	10	98.4	127.9	122.1	4.5
1315-1330	4.78	900	10	98.9	210.7	170.7	19.0
1330-1345	4.69	1136	15	98.7	250.7	182.9	27.0
1345-1400	4.71	905	30	96.7	167.9	143.6	14.5
1400-1415	4.72	1029	30	97.1	206.4	163.6	20.7
1415-1430	4.67	994	20	98.0	176.4	141.4	19.8
1430-1445	4.62	1316	25	98.1	239.3	190.7	20.3
1445-1500	4.55	986	45	95.4	177.9	152.9	14.0
1500-1517	4.50	1245	45	99.6	232.1	209.3	9.8
<u>A-9-W</u>							
1025-1040	5.21	532	10	98.1	148.1	80.6	45.6
1040-1055	5.02	947	10	98.9	168.6	115.0	31.8
1055-1110	4.91	1282	10	99.2	200.0	151.3	24.3
1110-1125	4.83	1157	20	98.3	186.4	129.3	30.6
1125-1140	4.75	1028	15	98.5	166.4	111.4	33.0
1140-1155	4.65	1020	15	98.5	168.8	119.4	29.7
1155-1210	4.55	825	50	93.9	166.4	127.9	23.1

TABLE 3A (Cont.)

Test Data (Reduced)
Series A

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>A-10-W</u>							
1122-1145	4.67	562	5	99.1	106.8	96.8	9.4
1145-1200	4.51	349	5	98.6	50.7	50.0	1.4
<u>A-11-W</u>							
1045-1100	5.33	823	5	99.4	171.4	105	38.7
1100-1115	5.15	831	5	99.4	175.7	99.3	43.5
1115-1130	5.00	1170	5	99.6	213.6	140.7	34.1
1130-1145	4.90	1234	15	98.8	212.1	156.4	26.3
1148-1200	4.78	1110	15	98.6	205	143.3	30.1
1200-1215	4.68	849	15	98.2	167.9	127.9	23.8
1215-1230	4.61	883	30	96.6	150.7	137.9	8.5
1230-1245	4.57	650	30	95.3	107.1	97.9	8.6
1245-1300	4.56	647	30	95.3	130	97.1	25.3
1300-1308	4.52	652	30	95.3	137.5	106.3	22.7
<u>A-12-W</u>							
1040-1055	5.37	570	0	100	108.8	81.9	24.7
1055-1110	5.12	1132	0	100	198.7	183.1	7.9
1110-1125	5.03	754	0	100	121.3	100.6	17.0
1125-1140	5.01	557	20	96.4	153.1	109.4	28.6
1140-1155	4.94	892	20	97.8	161.9	149.4	7.7
1155-1210	4.81	1275	15	98.8	222.5	204.4	8.1
1210-1225	4.74	1102	10	99.1	217.5	170.6	21.6
1225-1240	4.76	1023	30	97.1	207.5	155.0	25.3
1250-1305	4.73	973	15	98.4	179.3	168.6	6.0
1305-1320	4.64	1211	15	98.8	233.6	219.3	6.1
1320-1336	4.57	1298	10	99.2	268.8	193.1	28.2
1425-1440	4.66	611	15	97.5	125.0	101.3	19.0
1440-1455	4.59	820	15	98.2	125.6	121.9	3.0
1455-1510	4.54	855	5	99.4	142.5	132.5	7.0
1510-1525	4.52	780	0	100	129.4	100.0	22.7
1525-1538	4.51	613	30	95.1	82.5	65	21.2

TABLE 4A

Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg.</u> <u>pH</u>	ppm <u>SO₂</u> <u>In</u>	ppm <u>SO₂</u> <u>Out</u>	% <u>SO₂</u> <u>Rem</u>	ppm <u>NO_x</u> <u>In</u>	ppm <u>NO_x</u> <u>Out</u>	% <u>NO_x</u> <u>Rem</u>
<u>B-1-W</u>							
1035-1050	6.55	1185	0	100	240	254	-
1050-1105	6.14	1324	0	100	224	229	-
1105-1120	5.92	1065	15	98.6	172.5	176.2	-
1120-1135	5.81	926	15	98.4	120	125	-
1135-1150	5.71	934	15	98.4	148	159.6	-
1150-1205	5.57	668	15	97.8	101.9	92.5	9.2
1205-1220	5.46	999	10	99.0	138	144	-
1220-1235	5.43	911	10	98.9	121.9	125.8	-
1235-1250	5.36	1001	10	99.0	153	136	10.7
1250-1305	5.30	844	10	98.8	118.8	125.6	-
1305-1320	5.22	1129	10	99.1	183.8	168.1	8.5
1320-1327	5.07	1286	10	99.9	222.5	198.3	10.9
-							
1440-1455	5.38	1372	10	99.3	210	207	1.4
1455-1500	5.07	1272	10	99.2	183	175	4.4

TABLE 4A (Cont.)

Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-2-W</u>							
1030-1045	5.92	1222	10	99.2	187.5	194.4	-
1045-1100	5.84	701	10	98.6	116.1	114.4	1.4
1100-1115	5.86	724	10	98.6	176.9	131.9	25.4
1115-1130	5.74	911	15	98.4	217.8	185	15.0
1130-1145	5.62	1148	15	98.7	247.5	230	7.1
1145-1200	5.53	1178	15	98.7	268	240	9.4
1200-1215	5.38	1286	15	98.8	248.1	213.8	13.8
1215-1230	5.24	1492	15	99.0	373.3	297.8	20.2
1230-1245	5.26	1256	15	98.8	268.6	223.6	16.7
1245-1300	5.34	1148	15	98.7	279.4	215.6	22.8
1300-1315	5.27	1320	15	98.9	254.4	226.7	10.9
1315-1330	5.13	1155	20	98.3	217.8	189.4	15.3
1330-1345	5.07	1286	30	97.7	193.8	213.1	-
1345-1400	5.03	1444	60	95.8	256.7	238.9	6.9
1510-1525	5.23	821	5	99.4	157.9	155.0	1.8
1525-1540	5.15	1162	5	99.5	179.4	176.9	1.4
1540-1555	5.12	1275	5	99.6	220.5	216.1	2.0
1555-1608	5.05	1526	5	99.7	262.1	277.8	-

TABLE 4A (Cont.)

Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-3-W</u>							
945-1000	5.66	1088	10	99.1	274.2	244.2	10.9
1000-1010	5.51	1170	100	91.4	212	212	0.0
1010-1025	5.39	1016	10	99.0	203.1	164.3	19.1
1025-1040	5.25	1039	10	99.0	188.9	160.6	15.0
1040-1055	5.23	806	10	98.8	138.9	131.7	5.2
1055-1110	5.32	712	10	98.6	129.4	96.2	25.6
1110-1125	5.37	848	15	98.2	131.9	120.6	8.5
1125-1140	5.23	1174	10	98.3	246.7	216.7	12.2
1140-1155	5.22	1072	10	98.1	196.2	174.4	11.1
1155-1210	5.34	754	30	96.0	151.7	98.3	35.2
1220-1345	5.30	428	5	98.8	73.9	64.4	12.8
1345-1400	5.18	622	5	99.2	185.5	177.2	4.5
1400-1415	5.15	772	5	99.4	130	118.8	8.6
1415-1430	5.17	844	5	99.4	146.1	132.8	9.1
1430-1445	5.10	990	5	99.5	141.9	140.0	1.3
1445-1500	5.06	776	5	99.4	139.4	136.2	2.2
1500-1515	5.07	844	5	99.4	131.9	131.7	0.0
1515-1530	5.04	1005	5	99.5	165.6	156.9	5.3
<u>B-4-W</u>							
950-1005	5.07	1249	5	99.6	231	202	12.4
1005-1020	4.87	1031	5	99.5	179	154	14.3
1020-1035	4.79	1087	5	99.5	202	182	9.9
1035-1050	4.66	1309	5	99.6	213	202	5.2
1050-1105	4.61	956	5	99.5	183	149	18.5
1105-1120	4.59	1010	10	99.0	210	175	16.7
1120-1135	4.70	713	10	98.6	112	106	5.9
1135-1150	4.84	982	10	99.0	172	154	10.9
1150-1205	4.86	829	10	98.8	176	136	22.4
1205-1220	4.83	1230	10	99.2	246	204	12.0
1220-1235	4.58	922	15	98.4	139	112	19.0
1235-1245	4.58	1580	15	99.0	262	239	8.6

TABLE 4A (Cont.)

Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-5-W</u>							
(6-21-79)							
1055-1110	5.63	281	5	98.2	68.8	26.5	26.5
1110-1125	5.37	859	5	99.4	204.4	15.5	24.2
1125-1140	5.23	867	5	99.4	135.6	126.1	7.0
1140-1155	5.23	788	10	98.7	132.2	111.1	16.0
1155-1210	5.26	510	10	98.0	103.3	79.4	23.1
1210-1255	5.18	878	10	98.9	207.8	158.9	23.5
1255-1240	5.16	641	15	97.6	125	115.6	7.5
1240-1255	5.23	653	15	97.7	143.9	120	16.6
1255-1310	5.23	567	15	97.3	145.6	103.3	29.1
-							
1335-1343	5.23	570	15	97.4	130	99	23.8
<u>B-5-W</u>							
(6-22-79)							
840-855	5.13	1058	5	99.5	152.8	133.9	12.4
855-910	4.94	1033	5	99.5	213.9	181.1	15.3
910-915	4.76	1470	5	99.6	282.2	249.4	11.6
925-940	4.72	926	5	99.4	213.3	165.6	22.4
940-955	4.75	947	5	99.5	218.8	153.1	30.0
955-1010	4.69	1162	10	99.1	245.6	170.6	30.5
1010-1025	4.64	1241	10	99.1	232.5	198.8	14.5
1025-1040	4.64	1227	10	99.2	210.6	195.6	7.1
1040-1055	4.64	1069	10	99.1	156.1	137.8	11.7
1055-1105	4.57	1795	10	99.4	308.3	233.3	24.3
-							
1145-1200	4.90	626	10	98.4	119.4	91.7	23.2
1200-1215	4.76	1282	15	98.8	260.6	209.4	19.6
1215-1230	4.60	1219	15	98.8	237.2	189.4	20.2
1230-1236	4.52	1260	15	98.8	237.5	228.8	3.7

TABLE 4A (Cont.)

Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-6-W</u>							
1015-1030	5.24	832	0	100	155	120	22.6
1030-1045	4.99	1076	10	99.1	183.8	162.5	11.6
1045-1100	4.90	1035	10	99.0	179.4	148.1	17.4
1100-1115	4.89	1149	10	99.1	231.3	168.1	27.3
1115-1130	4.86	772	10	98.7	141.3	105.6	25.3
1130-1145	4.77	1298	15	98.8	220	161.3	26.7
1145-1200	4.70	1076	30	97.2	191.3	160.6	16.0
1200-1215	4.71	1088	30	97.2	195	161.9	17.0
1215-1230	4.66	968	15	98.4	165.7	137.9	16.8
1230-1245	4.59	973	30	96.9	199.4	142.5	28.5
1245-1300	4.63	851	30	96.5	158.1	115	27.3
1300-1315	4.69	921	30	96.7	192.5	123.1	36.1
1415-1430	5.02	827	25	97.0	156.4	130	16.9
1430-1445	4.57	1161	25	97.3	197.1	172.1	12.7
1445-1500	4.59	765	20	97.4	112.5	100	11.1
1500-1515	4.57	806	-10	95.0	149.3	111.4	25.4
1515-1530	4.52	784	40	94.9	152.9	116.4	23.9
1530-1545	4.53	711	30	95.8	145	106.3	26.7
1545-1600	4.55	930	30	96.8	181.3	149.4	17.6
1600-1612	4.50	945	25	97.4	173.3	138.3	20.2
<u>B-7-W</u>							
(7-3-79)							
1045-1100	5.27	821	10	98.8	161.4	146.7	12.8
(7-5-79)							
1000-1015	4.54	454	10	97.8	134.3	91.4	31.9

TABLE 4A (Cont.)

Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-8-W</u>							
1045-1100	4.80	531	5	99.0	116	64.3	44.4
1100-1115	4.75	677	5	99.3	134	80	40.1
1115-1130	4.66	1001	5	99.5	199	128	35.8
1130-1145	4.58	716	10	98.6	147	85	42.2
1145-1158	4.50	735	10	98.6	148	97	34.3
<u>B-9-W</u>							
1023-1238	4.98	321	5	98.4	80	71.4	10.8
1038-1053	4.75	401	5	98.8	86.9	116.3	-33.8
1053-1104	4.57	540	5	99.1	116	138	-18.9
<u>B-10-W</u>							
1030-1045	5.22	544	0	100	102.1	70.0	31.4
1045-1100	5.01	885	0	100	146.4	109.3	25.4
1100-1115	4.89	814	20	97.5	140.0	100.7	28.0
1115-1130	4.80	1024	20	98.0	190.0	130.7	31.2
1130-1145	4.75	660	15	97.7	90.0	70.7	21.4
1145-1200	4.66	1234	15	98.8	228.6	185.7	18.7
1200-1245	4.61	945	40	95.2	135.0	100.7	25.4
1215-1230	4.57	639	35	94.5	112.9	78.6	30.4
1230-1240	4.50	1074	30	97.2	170.0	123.3	27.5

TABLE 4A (Cont.)
Test Data (Reduced
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-11-W</u>							
1130-1145	5.56	611	0	100	145.0	105.7	27.1
1145-1200	5.33	911	0	100	185.6	138.8	25.2
1200-1215	5.14	1350	5	99.6	248.1	220.6	11.1
1215-1230	5.02	1462	5	99.6	236.1	217.8	7.8
1230-1245	4.89	1485	5	99.7	203.8	221.3	21.7
1245-1300	4.89	1292	5	99.6	276.9	217.5	21.5
1300-1315	4.99	945	5	99.5	167.8	142.2	15.2
1315-1330	5.00	956	10	99.0	153.3	125.0	18.5
-							
1350-1405	4.92	747	15	98.0	148.8	122.5	17.7
1405-1420	4.82	1136	15	98.7	208.1	166.3	20.1
1420-1435	4.77	1170	15	98.7	201.4	188.6	6.4
1435-1450	4.72	1340	15	98.9	222.8	216.7	2.7
1450-1505	4.70	953	20	97.9	131.9	117.5	10.9
1505-1520	4.67	610	30	95.1	78.8	66.9	15.1
1520-1535	4.58	1129	30	97.3	207	161.7	22.2
1535-1550	4.51	975	30	96.9	171.4	159.3	7.1

TABLE 4A (Cont.)
Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-12-W-1</u>							
915-930	5.32	405	5	98.7	97.9	63.6	35.1
930-945	5.09	574	5	99.1	109.4	81.1	25.9
945-1000	4.88	825	5	99.4	190.7	167.1	12.4
1000-1015	4.69	1093	5	99.5	196.9	178.1	9.5
1015-1030	4.57	1020	5	99.5	224.4	175.6	21.7
1030-1045	4.58	733	5	99.3	145.6	115.6	20.6
1045-1100	4.58	836	5	99.4	150.6	141.9	5.8
1100-1115	4.56	788	5	99.4	143.1	136.3	4.8
1115-1130	4.56	758	5	99.3	170.0	123.1	27.6
1130-1145	4.51	1025	5	99.5	194.0	168.0	13.4

TABLE 4A (Cont.)
Test Data (Reduced)
Series B

<u>Time</u>	<u>Avg. pH</u>	<u>ppm SO₂ In</u>	<u>ppm SO₂ Out</u>	<u>% SO₂ Rem</u>	<u>ppm NO_x In</u>	<u>ppm NO_x Out</u>	<u>% NO_x Rem</u>
<u>B-12-W-2</u>							
1315-1330	6.77	424	5	98.8	95.8	90.8	5.2
1330-1345	6.30	1144	5	99.5	223.8	208.8	6.7
1345-1400	6.10	1061	5	99.5	203.1	165.0	18.8
1400-1415	6.00	753	5	99.3	154.4	121.3	21.5
1415-1430	5.88	1243	5	99.6	256.3	253.1	1.2
1430-1445	5.79	1084	5	99.5	190.0	188.1	1.0
1445-1500	5.71	1009	5	99.5	220.6	185.0	16.1
1500-1515	5.65	787	10	98.7	184.8	122.5	17.6
-							
1517-1530	5.51	990	10	99.0	183.6	177.9	3.1
1530-1545	5.40	1230	10	99.2	230	227.8	1.0
1545-1600	5.33	1030	15	98.5	226.3	170.0	24.7
1600-1615	5.33	1010	15	98.5	219.4	160.6	26.8
-							
1619-1630	5.16	1240	15	98.8	298.0	260.0	12.8
1630-1645	5.09	1294	15	98.8	251.9	225.7	10.4
1645-1700	5.10	1215	15	98.7	181.3	183.8	-
1700-1715	5.05	1483	15	99.0	252.8	255.6	-
1715-1730	4.90	1856	15	99.2	348.6	292.1	16.2
1730-1745	4.82	1365	15	98.9	261.2	213.8	18.2
1745-1800	4.83	950	30	96.8	196.7	141.7	27.6
1800-1815	4.83	1084	60	94.5	176.4	177.9	-
1815-1830	4.81	1027	30	97.1	161.1	154.4	4.1
1830-1845	4.78	1343	30	97.8	181.3	183.1	-
1845-1900	4.73	1268	30	97.6	196.3	157.5	19.8
1900-1915	4.68	1298	30	97.7	207.5	148.8	28.3
1915-1930	4.61	1028	30	97.1	169.4	132.5	21.8
1930-1945	4.57	1273	30	97.6	225.6	181.9	19.4
1945-2000	4.55	1125	30	97.3	168.8	168.8	0
2000-2015	4.53	1144	30	97.4	190.6	166.3	12.8
2015-2030	4.52	950	30	96.8	126.9	118.8	6.4
2030-2040	4.51	1115	30	97.3	142	145	-

ADDENDUM

**APPLICATION OF THE SULF-X PROCESS
TO COAL CONVERSION AND UTILIZATION**

**PHASE II EXTENSION
FINAL REPORT**

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DEPARTMENT OF ENERGY
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ABSTRACT

This report presents the results obtained during the program extension to Phase II of DOE Contract No. DE-AC01-781123 (formerly ET-78-C-01-2779). Laboratory and 20-cfm, bench-scale testing was conducted on the SULF-X process at Westinghouse Advanced Energy Systems Division facilities. Test operations and on-line data gathering was performed by PENSYS; off-line sample analysis and data reduction was performed by Westinghouse.

Batch laboratory tests utilizing synthetic flue gas were conducted to determine performance for variations in FeS type, FeS concentration, SO₂ concentration, oxygen presence, and ferrous ion concentration. Batch bench-scale tests were conducted utilizing actual flue gas and regenerated FeS for up to five recycles. Steady-state tests were conducted on the bench apparatus using continuous feed and blowdown to maintain a constant system pH. Spent slurry was regenerated and reused to maintain a four-week run. The system was shut down over night and on weekends.

Laboratory tests resulted in 95% SO₂ removal and up to 95% NO_x removal. Bench tests demonstrated similar SO₂ removal efficiencies, but obtained a maximum of only 26% NO_x removal. Major technical difficulties in the process, as determined in the bench and steady-state tests, were caused by both procedural and mechanical inadequacies that resulted in relatively poor NO_x removal, oxidation during regeneration, and lower reactivity after regeneration.

Total capital investment for the SULF-X system was estimated to be \$91 to \$103 per kilowatt (electric), compared to \$90/kw for sodium solution scrubbing, \$78 to \$83/kw for magnesia slurry scrubbing, and \$74/kw for limestone slurry scrubbing. Annual operating costs for the SULF-X system were estimated to be 5.44 to 6.90 mills per kilowatt-hours, compared to 4.96 to 5.22 for sodium, 3.68 to 3.99 for magnesia, and 3.73 to 4.25 for limestone. All estimates were performed on a consistent basis, utilizing the procedures, techniques, data base, and alternate system flowsheets described in a TVA study of advanced desulfurization processes prepared for EPA.

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SUMMARY

Batch laboratory tests utilizing synthetic flue gas were conducted to determine performance for variations in FeS type, FeS concentration, SO₂ concentration, oxygen presence, and ferrous ion concentration. Batch bench-scale tests were conducted utilizing actual flue gas and regenerated FeS for up to five recycles. Steady-state tests were conducted on the bench apparatus using continuous feed and blowdown to maintain a constant system pH. Spent slurry was regenerated and reused to maintain a four-week run. The system was shut down over night and on weekends.

Laboratory tests resulted in 95% SO₂ and up to 95% NO_x removal. Bench tests demonstrated similar SO₂ removal efficiencies, but obtained a maximum of only 26% NO_x removal. Major technical difficulties in the process, as determined in the bench and steady-state tests, were caused by both procedural and mechanical inadequacies that resulted in relatively poor NO_x removal, oxidation during regeneration, and lower reactivity after regeneration.

However, after recognizing that further testing and development could provide data more appropriate to the needs of a commercial plant, the economics of the SULF-X process, based on idealized performance, were estimated and compared to the desulfurization processes.

Total capital investment for the SULF-X system was estimated to be \$91 to \$103 per kilowatt, compared to \$90/kw for sodium solution scrubbing, \$78 to \$83/kw for magnesia slurry scrubbing, and \$74/kw for limestone slurry scrubbing. Annual operating costs for the SULF-X system were estimated to be 5.44 to 6.90 mills per kilowatt-hours, compared to 4.96 to 5.22 for sodium, 3.68 to 3.99 for magnesia and 3.73 to 4.25 for limestone. All estimates were performed on a consistent basis, utilizing the procedures, techniques, data base and alternate system flowsheets described in a TVA study of advanced desulfurization processes prepared for EPA.⁽¹⁾ The cost ranges for the capital and operating estimates are the result of a secondary evaluation that considered the use of coal for the SULF-X primary regeneration operations and related differences in the design basis.

INTRODUCTION

This report represents the results obtained during the program extension to Phase II of DOE Contract No. DE-AC01-781123 (formerly ET-78-C-01-2779).

Prior to completing the original scope of work under Phase II of the Contract, PENSYS proposed to expand the scope of work to include steady-state testing with the bench-scale unit and to use the more comprehensive analytical techniques to better define the system chemistry.

On September 18, 1979, a contract extension was issued by DOE. Laboratory and 20-cfm, bench-scale testing was conducted on the SULF-X process at Westinghouse Advanced Energy Systems Division facilities. Test operations and on-line data gathering was performed by PENSYS; off-line sample analysis and data reduction was performed by Westinghouse.

The SULF-X test program under DOE Contract No. ET-78-C-01-2779 was divided into the following five major tasks:

- TASK 1 - Performance Characterization - Scrubber Operation
- TASK 2 - Performance Characterization - Regeneration Operation
- TASK 3 - Scrubber Characterization with Regenerated FeS
- TASK 4 - Closed-Cycle (Steady-State Operation)
- TASK 5 - Preliminary Economic Analysis

The purpose of the Westinghouse sub-contract GO No. XAL-72431 was to provide engineering assistance, off-line analytical chemical evaluation of test samples and assistance in data reduction and analysis for Tasks 1 through 4. This sub-contract also included performance for Task 5 in its entirety.

TEST PROGRAM

Test plans were prepared for Task 1A (Lab Scale), Task 1B and 2 (20 cfm Bench Scale) and Task 3 (Bench Scale with Regeneration). The original test plans are presented in Tables 1, 2, and 3.

All process performance testing for the various tasks were performed by PENSYS. A schematic diagram of the laboratory test apparatus utilized for Task 1A is shown in Figure 1. The 20 CFM Bench Scale Facility is shown schematically in Figure 2.

On-line performance (SO_2 removal, pH, etc.) was measured by PENSYS as indicated by the above diagrams. Off-line analyses were performed by Westinghouse at various test laboratories. The testing laboratories and analytical tests conducted are shown in Table 4.

TABLE 1
TASK 1A (LAB SCALE)

<u>Run No.</u>	<u>FeS Type</u>	<u>FeS</u> (gms/l)	<u>SO₂</u> (ppm)	<u>O₂</u> (%)
1-1	-	0	4000	-
1-2	Precipitated	14	4000	-
1-3	Precipitated	2	4000	-
1-4	Precipitated	2	500	-
1-5	Precipitated	2	500	5
1-6	Regenerated*	14	4000	-
1-7	Regenerated*	14	500	-
1-8	Regenerated*	14	500	5

<u>Run No.</u>	<u>FeS Type</u>	<u>Fe⁺⁺</u> (gms/l)	<u>FeS</u> (gms/l)	<u>SO₂</u> (ppm)	<u>O₂</u> (%)	<u>NO</u>
1-9	Regenerated*	0	14	4000	5	500
1-10	Regenerated*	10	14	4000	5	500
1-11	Regenerated*	80	14	4000	5	500
1-12	Precipitated	80	2	4000	5	500

*Actually thermally decomposed pyrite. Grinding time in ball mill equals 12 hours.

TABLE 2
TASK 1B and 2 (20 cfm SCALE)

<u>Run No.</u>	<u>FeS Type</u>	<u>FeS</u> (gms/l)	<u>Fe⁺⁺</u> (gms/l)	<u>Regeneration</u> (Task 2)
2-2a	Precipitated	14	0	w/N ₂
2-2a	Precipitated	14	80	w/N ₂
2-2b	-	-	-	w/N ₂ and CO
2-3	Regenerated*	14	0	w/N ₂
2-4	Regenerated*	14	80	w/N ₂

TABLE 3
TASK 3 (20 cfm - BATCH)

<u>Run No.</u>	<u>FeS Type</u>	<u>Regeneration</u>
3-1	Regenerated from 2-3	w/N ₂
3-2	Regenerated from 3-1	w/N ₂
3-3	Regenerated from 3-2	w/N ₂
3-4	Regenerated from 3-3	w/N ₂
3-5	Regenerated from 2-4	w/N ₂ (and CO)
3-6	Regenerated from 3-5	w/N ₂ (and CO)
3-7	Regenerated from 3-6	w/N ₂ (and CO)
3-8	Regenerated from 3-7	w/N ₂ (and CO)

*Actually thermally decomposed pyrite. Grinding time in Ball Mill equals 12 hours.

FIGURE 1

LABORATORY 1-LITER REACTOR STUDIES SCHEMATIC DIAGRAM OF PROCESS FLOWS & MEASUREMENTS

TABLE 4
ANALYTICAL TESTS

<u>Testing Laboratory</u>	<u>Analyses</u>
Advanced Energy Systems Div.	Scanning Electron Microscope S and Fe Mapping X-Ray Fluorescence
Advanced Reactors Div.	Ion Chromatography Atomic Absorption % Solids and Density Scanning Electron Microscope X-Ray Fluorescence Mass Spectrometry
Research and Development Center	Electron Spectroscopy for Chemical Analysis
P.E.C. Laboratories	Wet Chemical Analyses

TASK 1A - LABORATORY-SCALE TESTS

As indicated in the test plan for this task, SO_2 and NO removal tests were run for various combinations of operating parameters. Test variables included FeS type, FeS concentration, SO_2 concentration, oxygen concentration, NO_x concentration, and ferrous ion concentration. All tests were conducted by bubbling bottled gases with an argon carrier through a fritted disk in a 1-liter beaker.

PERFORMANCE RESULTS

The actual test conditions are presented in Table 5. Sulfur dioxide and NO_x removal efficiencies for the twelve Task 1A tests are given in Table 6. Test 1-1, using water only with no FeS, had minimal SO_2 removal before the pH of the solution was lowered below the test end-point of 4.5. Tests 1-2, 1-3 and 1-4 all resulted in measurable levels of H_2S in the scrubbed gas. These tests all employed precipitated FeS with no oxygen present in the inlet gas. Tests 1-5 through 1-12 all had undetectable levels of SO_2 and H_2S in the exit gas stream. These results appear to indicate that H_2S formation will not occur when either oxygen is present in the gas stream, or regenerated, rather than precipitated, FeS is used as the reagent. The formation and subsequent oxidation of the H_2S is consistent with the hydrolysis reactions described by Lasaga.⁽²⁾ However, the lack of detectable H_2S for Runs 1-6 and 1-7, which were conducted without oxygen in the inlet test gas, may be the result of catalysis by impurities in the regenerated FeS.⁽³⁾

The effect of the various test variables on FeS utilization (moles of SO_2 removal per mole of initial FeS) is shown in Table 7 and 8. Precipitated FeS shows much higher utilization ratios than regenerated FeS (i.e., roasted pyrites) in all cases. This difference in performance is attributed to two major factors — impurities in the roasted pyrites and particle size, with the latter probably more significant.

The system was run to determine the effect of iron sulfide composition and particle size on the reactivity toward sulfur dioxide and nitrogen oxides. The runs were conducted to predetermined pH end points as well as for specific time intervals. This was planned so as to be able to ascertain the reaction kinetics of the system.

The relationship between milling time and iron sulfide capacity (utilization) to remove sulfur dioxide at the prescribed test conditions was developed and is presented in Table 9.

The iron sulfide capacity as a function of the reciprocal of the average particle diameter is presented in Figure 3.

Figure 4 depicts the pH of the laboratory test system during the last three (3) minutes of four runs. For each of the runs, the controllable test conditions were almost identical with the exception of the iron sulfide particle size ranges. The reagent came from the same source but was milled for periods of 3, 6, 9, and 12 hours as labelled on the graph. The same amount of iron sulfide was used in each of the tests. Zero time represents the end-point of each of the runs.

At the conclusion of the runs, the sulfur dioxide was turned off and the system continually purged with inert gas (argon or nitrogen). The pH of the slurry was monitored during and after the tests. These pH curves are plotted on the same graph and demonstrate that the rate of pH recovery, due to iron sulfide hydrolysis, is in proportion to the amount of unreacted iron sulfide which, in turn, is a function of the iron sulfide particle size.

Impurities in the roasted pyrites were detected by both ESCA and X-ray fluorescence (SRF). Major impurities are silicon, chromium, other metals, and iron oxides. The effect of impurities is to lower the mass of FeS for a given total weight of reactant, thus lowering the utilization ratio per unit weight of total reactant. Perhaps more important than impurities, however, is the effect of particle size. Previous tests by PENSYS have demonstrated the effect of increasing utilization by increasing grinding time of the roasted pyrites. Since precipitated FeS has a very fine particle size, it would be expected to have a higher utilization ratio than the coarser regenerated FeS. The expected difference in particle size between the two types of FeS was verified by the use of a scanning electron microscope.

Higher utilization was also observed for 2 g/l FeS concentration versus 14 g/l and 500 ppm SO₂ versus 4000 ppm. The most apparent explanation for these results is the "batch" operating conditions. Tests were conducted from an initial high pH to a predetermined pH end-point of 4.5. Since no fresh reactant was added to the system to replace the amount reacted, the pH decreased throughout the test for all operating conditions. The higher SO₂ concentration (4000 ppm) caused a more rapid pH drop than the lower concentration, resulting in more unreacted FeS at pH 4.5 and a lower utilization ratio. Likewise, the higher initial FeS concentration (14 g/l) resulted in more unreacted FeS and a lower utilization ratio. It should be expected that dynamic tests (e.g., continuous FeS feed and constant pH) will result in significantly higher utilization ratios.

The effect of oxygen on utilization was indeterminate. No discernible effect was observed with precipitated FeS, yet a significant increase was measured with regenerated reagent. A possible explanation is that the presence of oxygen promotes the formation of the thiosulfate ion and the hydroxyl ion,

which would tend to extend the length of run by raising the pH.

The data indicate a direct correlation between Fe⁺⁺ concentration and NO_x removal.⁽⁴⁾ However, the utilization ratio (with respect to SO₂) decreased with increasing ferrous ion levels. This is attributable to lower initial pH values caused by sulfuric acid impurities in the ferrous sulfate and the acidic nature of FeSO₄. It appears from these tests that the presence of high concentrations of ferrous ions may result in lower SO₂ utilization ratios, but most likely the magnitude of this effect will be less than that shown in Table 8.

ANALYSIS RESULTS

In order to measure the physical properties and chemical composition of the fresh and spent slurries from each performance test, off-line analyses were performed. ESCA, Electron Spectroscopy for Chemical Analysis, was conducted at the Westinghouse Research Laboratories. Samples of fresh FeS, fresh pyrites, roasted pyrites, and spent scrubber slurry were analyzed. The test procedures and detailed results are given in Appendix A. Most significant results were the following:

- Significant quantities of $\text{SO}_4^{=}$ were detected, except in the roasted pyrites sample.
- S/Fe ratio was less than theoretical and a large deficiency of S was found in the roasted pyrite sample.
- Iron oxide was observed.

The ESCA tests were not able to distinguish between the oxygen atoms present as FeSO_4 , FeSO_3 , iron oxides, water, and oxygen-containing species. This limited the usefulness of the ESCA results, so there were no ESCA tests run on subsequent samples.

Spent slurry samples from Tests 1-4, 1-5, 1-7, and 1-11 were also analyzed at the Westinghouse Advanced Reactors Division Analytical Laboratory. Ferrous ion concentration was measured by colormetric analysis, sulfite and sulfate were measured with an ion chromatograph, specific gravity and percent suspended solids were measured, impurities were determined by spectrographic analysis, solid phase chemical composition was measured by X-ray fluorescence, and the solids physical appearance was observed with a scanning electron microscope. Results of the slurry analyses are given in Appendix B; the XRF results are presented in Appendix C; and the SEM results are given in Appendix D.

Mass balances for the four tests for which detailed analyses were performed are given in Table 10. Reasonable balances were obtained on both iron and sulfur for all tests with the exception of the sulfur balance for Test 1-5. Significant observations from these tests results are:

- More than 1 mole of Fe^{++} was created for each mole of SO_2 removed.
- Total sulfur into the slurry can be accounted for by SO_3 and SO_4 (i.e., no thiosulfate)
- Significant sulfite (SO_3) levels were detected.

The solids analyses by XRF showed some unexpected results. Total iron and sulfur was less than 70% by weight for all samples, including fresh pyrites and reagent grade FeS. These percentages were determined by comparing XRF spectra against known standards prepared from stable metal oxides and elemental sulfur. The results of the calibration runs are given in Appendix C. Although some error may be expected, it is highly unlikely that more than a few percent of the 30% of impurities is attributable to analysis error. Therefore, the samples are presumed to consist of the indicated compounds with the remainder being light elements most probably oxygen and hydrogen. It should be noted that oxidation of FeS to FeSO_4 causes an increase in molecular weight from 88 to 152. Further hydration of FeSO_4 to $\text{FeSO}_4 \cdot 7 \text{H}_2\text{O}$ causes the molecular weight to increase to 278. Therefore, a small amount of oxidation can lead to a large weight gain. For this reason, all drying and sample preparation was conducted under an inert atmosphere. The oxidation percentage measured is presumed to be representative of the original sample.

The reagent grade FeS and the slurry samples from Tests 1-4 and 1-5, both of which used precipitated FeS, were found to be relatively free of impurities, with the exception of the aforementioned light elements. However, fresh pyrites was found to contain 1.7% silicon and 0.6% titanium, along with other trace impurities. Sample 1-7, which utilized roasted pyrites, contained silicon from the original pyrites along with alloying elements from the regeneration vessel. These included 1.2% chromium and 0.6% nickel.

The overall sulfur-to-iron atomic ratio in roasted pyrites was found to be 1.01. It is probable that the actual ratio in iron sulfide crystals is somewhat higher,⁽³⁾ but the presence of iron oxides reduces the overall ratio. Precipitated FeS is assumed to have a S to Fe molar ratio of 1.0. Atomic ratios for the spent slurry samples were as follows:

TABLE 11

Sulfur to Iron Molar Ratios

<u>Test</u>	<u>S/Fe</u>
1-4	.85
1-5	1.24
1-7	.93
1-11	.90

Tests 1-4, 1-7, and 1-11 show a decrease in overall solids S/Fe ratio, while test 1-5 shows an increase. Test 1-5 utilized precipitated FeS with a low SO₂ concentration and had 5% oxygen in the feed gas. This test also resulted in the highest utilization ratio. However, it should also be noted that the slurry sample showed a high sulfate concentration and there was a large sulfur imbalance. The significance of these results is not fully understood at this time. In summary, the XRF results appeared to indicate the following:

- Significant oxidation exists in all samples.
- Fresh pyrites contains impurities, mostly silicon (or sand).
- Roasted pyrites is contaminated with alloying elements from the regeneration vessel.
- No discernible increase in sulfur content of the solids is detected. In fact, a decrease in sulfur content appears to be indicated.

Scanning electron microscope (SEM) analysis was conducted on slurry samples at both the AESD and ARD laboratories. SEM photographs and S and Fe maps are included in Appendix D. The SEM results were mixed, and appear to be very dependent on the sample preparation methods. Samples studied at AESD showed the fresh pyrites and roasted pyrites (before grinding to consist of 15-25 micron particles).

The roasted pyrites after milling appeared the same both before and after contact with the flue gas. In each case, it appeared as clumps of fine particles, 1 micron or less in diameter. However, samples of roasted pyrites studied at the ARD laboratory appeared as large (100 to 200 micron) particles with a porous coal-like structure. In no samples could individual compounds be observed as discrete crystals. Sulfur and iron maps indicated homogeneous distribution. One sample of spent slurry from the steady-state tests did show a sulfur-rich region and an iron-rich flat-surfaced flake, but the identification of single FeS particles, FeSO₄ crystals, or iron oxide crystals was not accomplished.

TABLE 5

Lab Test Conditions

Task 1-A

Run No.	Type	FeS		Fe ⁺⁺ g/l	Inlet Gas						Time Min
		Weight Grams	Moles		Flow l/min	SO ₂ ppm	SO ₂ Total Moles	NO Avg. ppm	NO Total Moles	%O ₂	
1-1(BS91)	distilled water, 1 liter			-	2.00	4475	.0126	-	-	0	.17
1-2(BS95)	precipitated	14.0 g	.159	-	2.00	4475	.0554	-	-	0	149
1-3(BS98)	precipitated	2.0 g	.0228	-	2.12	4475	.0138	-	-	0	35
1-4(BS100)	precipitated	2.0	.0228	-	2.12	560	.0218	-	-	0	443
1-5(BS101)	precipitated	2.0	.0028	-	2.00	672	.0175	-	-	5	313
1-6(BS89)	regenerated	15.51	.176	-	2.00	4480	.0138	-	-	0	37
1-7(BS90)	regenerated	15.213	.173	-	2.00	544	.0135	-	-	0	300
1-8(BS102)	regenerated	14.207	.162	-	2.00	548	.0317	-	-	5	720
1-9(BS103)	regenerated	14.85	.169	0	2.00	1930	.00865	385	.00173	5	54
1-10(BS104)	regenerated	16.247	.185	10	2.00	1890	.00267	382	.00054	5	17
1-11(BS105)	regenerated	15.617	.178	80	2.00	2150	.00054	450	.00011	5	3
1-12(BS106)	precipitated	2.0	.0028	80	2.00	1990	.00230	425	.00049	5	14

TABLE 5 - Cont.

Lab Test Conditions

Task 1-A

Run No.	Outlet Gas				Absorbed		% Removal		Moles SO ₂ Mole FeS ₂	pH	
	SO ₂ Moles	H ₂ S Moles	NO Avg ppm	NO Total Mcles	SO ₂ Moles	NO _x Moles	SO ₂	NO _x		Initial	Final
1-1 (BS91)	-	-	-	-	.00006	-	-	-	-	7.25	4.5
1-2 (BS95)	-	.00387	-	-	.0515	-	-	-	.324	8.55	4.50
1-3 (BS98)	-	.00191	-	-	.0119	-	-	-	.521	7.74	4.50
1-4 (BS100)	-	.00481	-	-	.0170	-	-	-	.746	7.56	4.50
1-5 (BS101)	-	-	-	-	.0175	-	-	-	.769	7.50	4.50
1-6 (BS89)	-	-	-	-	.0138	-	-	-	.078	7.68	4.50
1-7 (BS90)	-	-	-	-	.0135	-	-	-	.078	7.72	4.50
1-8 (BS102)	-	-	-	-	.0327	-	-	-	.202	7.35	4.69
1-9 (BS103)	0	0	345	.00155	.00865	.00018	100	10.4	.0512	7.15	4.50
1-10 (BS104)	0	0	250	.00135	.00267	.00019	100	34.5	.0144	5.81	4.48
1-11 (BS105)	0	0	84	.00002	.00054	.00009	100	81.1	.00303	5.03	4.40
1-12 (BS106)	0	0	23	.000026	.00230	.00046	100	94.6	.101	5.67	4.48

TABLE 6

Lab Tests - Task 1-A

Removal Efficiency

<u>Test</u>	<u>% SO₂</u>	<u>% NO_x</u>	<u>H₂S (ppm)</u>	<u>Fe⁺⁺ (g/l)</u>
1-1	0.5	-	-	-
1-2	93	-	275	-
1-3	86	-	800	-
1-4	78	-	150	-
1-5	100	-	-	-
1-6	100	-	-	-
1-7	100	-	-	-
1-8	100	-	-	-
1-9	100	10	-	0
1-10	100	35	-	10
1-11	100	81	-	80
1-12	100	95	-	80

TABLE 7

Lab Tests - Task 1-A

Effects of Test Variables on Utilization Values

<u>Run No.</u>	<u>FeS Type</u>	<u>FeS (g/l)</u>	<u>SO₂ (ppm)</u>	<u>% O₂</u>	<u>Test Variable</u>	<u>moles SO₂</u> <u>mole FeS</u>
1-1	--	14	4000	0	Precipitated FeS	.324
1-6					Roasted Pyrites	.078
1-3	Precip.	-	4000	0	2 g/l FeS	.521
1-1					14 g/l FeS	.324
1-3	Precip.	2	-	0	4000 ppm SO ₂	.521
1-4					500 ppm SO ₂	.746
1-6	Pyrites	14	-	0	4000 ppm SO ₂	.078
1-7					500 ppm SO ₂	.078
1-4	Precip.	2	500	-	0% O ₂	.746
1-5					5% O ₂	.769
1-7	Pyrites	14	500	-	0% O ₂	.078
1-8					5% O ₂	.202

TABLE 8

Lab Tests - Task 1-A
Performance vs Fe⁺⁺ Concentration

14 g/l FeS
2000 ppm SO₂
500 ppm NO_x
5% O₂

	<u>Fe⁺⁺ g/l</u>	<u>moles SO₂</u> <u>mole FeS₂</u>	<u>% NO_x Removal</u>
Roasted Pyrites	0	.051	10
	10	.014	35
	80	.003	81
Precipitated	80	.101	95

TABLE 9

Lab Tests

Effect of FeS Particle Size (Milling Time) on Utilization Values

<u>Exp. #</u>	<u>Milling Time (hr)</u>	<u>FeS Wt (g)</u>	<u>FeS Moles</u>	<u>Initial pH</u>	<u>Time To pH 4.5 (min)</u>	<u>SO₂ ppm</u>	<u>SO₂ Moles/Min</u>	<u>SO₂ Moles</u>	<u>Moles SO₂ / Mole FeS</u>
Regenerated pyrite									
BS 7	3 hr	14.271	0.162	7.10	3.25	4535	.000379	.00123	.0076
BS 6	6 hr	13.152	0.152	7.56	13.5	4535	.000379	.00512	.0341
BS 5	9 hr	12.280	0.140	7.59	33.5	4535	.000379	.00853	.0609
BS 8	12 hr	15.216	0.173	8.04	39.5	4535	.000379	.01497	.0865
BS 10	12 hr	13.923	0.158	8.04	38.0	4535	.000379	.01440	.0911
BS 13	12 hr	12.104	0.138	7.20	62.0	2267	.000190	.01175	.0853
Regenerated pyrite + FeSO ₄									
BS 9	3 hr	11.094	0.126	8.77	7.25	4535	.000379	.00274	.0218
BS 11	12 hr	12.526	0.142	8.85	15.25	4535	.000379	.00578	.0407
BS 12	24 hr	11.745	0.134	8.85	20.0	4535	.000379	.00758	.0567
Pyrite + IOD									
BS 16	3 hr	11.403	0.130	10.22	22.0	4535	.000379	.00830	.0643
BS 14	6 hr	10.898	0.124	10.00	27.0	4535	.000379	.0102	.0825
BS 15	9 hr	13.288	0.151	10.06	46.0	4535	.000379	.0174	.1153
BS 17	12 hr	13.533	0.154	10.07	53.0	4535	.000379	.0201	.1305
BS 18	24 hr	13.758	0.156	10.13	78.0	4535	.000379	.0296	.1889
A 12R									
BS 20	3 hr	10.547	0.120	7.38	4.0	4535	.000379	.00152	.0126
BS 21	6 hr	10.844	0.123	7.55	8.75	4535	.000379	.00332	.0269
BS 22	6 hr	10.670	0.121	7.50	18.5	2055	.000172	.00318	.0262
BS 23	6 hr	10.395	0.118	7.45	103.0	423	.000035	.00364	.0303
BS 25	9 hr	10.360	0.118	7.58	10.25	4535	.000379	.00388	.0330
BS 24	12 hr	10.675	0.121	7.45	14.0	4535	.000379	.00531	.0437
BS 27	12 hr	21.591	0.246	8.00	47.0	4535	.000379	.0102	.0417
BS 28	24 hr	10.983	0.125	7.85	19.75	4535	.000379	.00748	.0599

TABLE 9 - Cont.

Lab Tests

Effect of FeS Particle Size (Milling Time) on Utilization Values

<u>Exp. #</u>	<u>Milling Time (hr)</u>	<u>FeS Wt (g)</u>	<u>FeS Moles</u>	<u>Initial pH</u>	<u>Time To pH 4.5 (min)</u>	<u>SO₂ ppm</u>	<u>SO₂ Moles/Min</u>	<u>SO₂ Moles</u>	<u>Moles SO₂ Mole FeS</u>
BS 34	3 hr	11.051	0.126	9.40	11.0	4535	.000379	.00417	.0332
BS 33	6 hr	10.807	0.123	9.30	17.0	4535	.000379	.00644	.0524
BS 36	9 hr	11.075	0.126	9.50	23.0	4535	.000379	.00872	.0692
BS 32	12 hr	11.810	0.134	9.40	32.5	4535	.000379	.01232	.0917
BS 35	24 hr	12.344	0.140	9.77	49.75	4535	.000379	.01885	.1342
B 12 R 2									
BS 38	0 hr	13.128	0.149	8.96	1.08	4535	.000379	.00079	.0053
BS 56	3 hr	13.140	0.149	9.65	15.17	4535	.000379	.00575	.0384
BS 39	6 hr	12.966	0.148	9.40	25.0	4535	.000379	.00948	.0643
BS 37	9 hr	13.479	0.153	9.45	36.0	4535	.000379	.0136	.0887
BS 57	12 hr	13.089	0.149	9.73	37.0	4535	.000379	.0140	.0942
BS 58	24 hr	14.165	0.161	10.08	64.0	4535	.000379	.0242	.1505
Pyrite 850/CO									
BS 41	3 hr	14.129	0.161	8.52	10.0	4535	.000379	.00379	.0235
BS 44	3 hr	13.950	0.159	8.40	8.0	4535	.000379	.00303	.0190
BS 42	6 hr	14.233	0.162	8.57	12.0	4535	.000379	.00455	.0281
BS 43	9 hr	14.764	0.168	8.30	18.0	4535	.000379	.00766	.0456
BS 45	12 hr	14.213	0.162	8.29	29.0	4535	.000379	.01099	.0679
BS 46	24 hr	14.965	0.170	8.81	40.0	4535	.000379	.01516	.0890
BS 47	24 hr	14.791	0.168	8.24	41.0	4535	.000379	.01553	.0923

Table 9 - Cont.

Lab Tests

Effect of FeS Particle Size (Milling Time) on Utilization Values

<u>Exp. #</u>	<u>Milling Time (hr)</u>	<u>FeS Wt (g)</u>	<u>FeS Moles</u>	<u>Initial pH</u>	<u>Time To pH 4.5 (min)</u>	<u>SO₂ ppm</u>	<u>SO₂ Moles/Min</u>	<u>SO₂ Moles</u>	<u>Moles SO₂ Mole FeS</u>
Pyrite 850/N ₂									
BS 48	3 hr	13.970	0.159	7.89	8.0	4535	.000379	.00303	.0191
BS 49	6 hr	15.003	0.171	-	14.0	4535	.000379	.00531	.0311
BS 50	6 hr	14.979	0.170	8.06	14.5	4535	.000379	.00548	.0322
BS 51	9 hr	14.956	0.170	8.19	20.75	4535	.000379	.00786	.0462
BS 54	12 hr	15.090	0.172	8.45	23.0	4535	.000379	.00872	.0508
BS 52	24 hr	15.745	0.179	8.60	34.75	4535	.000379	.01317	.0735
Great Western FeS									
BS 53	3 hr	12.214	0.139	7.83	13.75	4535	.000379	.00521	.0375
BS 55	6 hr	11.900	0.135	7.45	20.25	4535	.000379	.00767	.0568
BS 62	9 hr	12.225	0.139	7.03	22.75	4535	.000379	.00862	.0620
BS 65	12 hr	11.605	0.132	7.07	31.17	4535	.000379	.01181	.0895
BS 61	24 hr	12.940	0.147	7.75	43.5	4535	.000379	.01648	.1120

Figure 4
pH CURVES
DURING AND AFTER TESTS
FOR IRON SULFIDE
MILLED 3, 6, 9 AND 12 HOURS

TABLE 10

Mass Balances - Task 1A

Sulfur

Item	Units	Test 1 - 4		Test 1 - 5		Test 1 - 6		Test 1 - 11	
		In	Out	In	Out	In	Out	In	Out
Volume	l	1.0		1.0		1.0		1.0	
Susp. Solids	g/l	2		2		14		14	
Sulfur as:									
SO ₂	gm	0.698	0	0	0	0	0	0	0
H ₂ S	gm	0	0.154	0	0	0	0	0	0
Total Gaseous S	gm	0.698	0.154	0.560	0	0.441	0	0.017	0
Sulfur as:									
SO ₄ "	gm	0.729	1.40	.729	3.30	0	0.013	45.81	51
SO ₃ "	gm	0	0.42	0	0.10	0	0.046	0	0.21
S ₂ O ₃ "	gm	0	-	0	-	0	0.605*	0	-
Total Soluble S	gm	0.729	1.82	0.729	3.40	0	0.664	45.81	51.21
Insoluble Sulfur	% wt.	-	21.3	-	23.7	23.9	24.3	-	-
Total Insoluble S	gm	.723	.20	.723	.100	3.64	3.47	2.80	2.92
Total S	gm	2.150	2.174	2.150	3.50	4.081	4.134	49.63	54.13
Balance	%		+1		+63		+1		+9

* Calculated

TABLE 10 - Cont.

Mass Balances - Task 1A

Iron

Item	Units	Test 1 - 4		Test 1 - 5		Test 1 - 6		Test 1 - 11	
		In	Out	In	Out	In	Out	In	Out
Iron as:									
Fe ⁺⁺	gm	0	1.06	0	1.25	0	0.58	80.17	81.14
Total Soluble Fe	gm	0	1.06	0	1.25	0	0.58	80.17	81.14
Insoluble Fe	% wt.	-	43.9	-	33.5	41.3	45.9	-	-
Total Insoluble Fe	gm	1.277	0.499	1.277	0.146	6.28	6.54	6.55	6.89
Total Fe	gm	1.277	1.508	1.277	1.40	6.28	7.12	86.72	88.03
Balance	%		+18		+10		+13		+1

TASKS 1 B AND 2 - SCRUBBER CHARACTERIZATION WITH 20 CFM BENCH SCALE

PERFORMANCE TESTS

In order to accelerate the test program, Tests 2-1 and 2-3 (without Fe^{++}) were deleted. The results of Tests 2-2 and 2-4 are shown in Table 12 along with the laboratory scale results for the same operating conditions. For both precipitated FeS and roasted pyrites, the following differences between the 20 cfm bench scale (50-gallon recirculation tank) and the laboratory scale (1 liter beaker) were observed:

- Higher utilization of FeS for the bench scale.
- Nearly 100% SO_2 for both cases.
- Lower NO_x removal for the bench scale.

The better FeS utilization in the bench scale can be attributed to the batch operation and the use of the predetermined pH end-point. The larger recirculation tank prevents rapid depression of the pH and allows for more reaction time. In the lab tests, the pH was lowered below the end-point in a matter of minutes, primarily due to the low initial pH experienced with high Fe^{++} concentrations.

The lower NO_x removal with the bench-scale scrubber may be due to the relatively low gas velocity, less than 2 feet per second, through the Koch tray scrubber. Apparently, the gas velocity was insufficient to create the turbulent conditions necessary to produce a large liquid-gas interface;⁽⁵⁾ a situation that went undetected throughout the project. Due to the poor mass transfer characteristics of NO_x , a large liquid-gas interfacial area is needed to offset the low transfer rate per unit area.

OFF-LINE ANALYSES

ESCA and IR analyses are summarized in Table 13 and SEM/XRF results (AESD Laboratory) are given in Table 14. Further details on the analyses are given in Appendices A, C and D. Slurry and XRF results (ARD Laboratory) are summarized in Table 15; details are given in Appendices B and C.

The following observations can be made from those analytical results:

- There is a small increase in Fe^{++} concentration and a larger decrease in $\text{SO}_4^{=}$ concentration between the fresh and spent slurry samples.
- No clear indication was found of sulfur enrichment of the solid phase in the spent slurry.
- Regeneration without a reducing agent caused a dramatic decrease in S/Fe ratio, probably due to oxidation.
- Regeneration of spent precipitated FeS with CO present resulted in an S/Fe ratio of 0.69, a 22% decrease from the spent slurry composition.
- ARD analysis of roasted pyrites showed a S/Fe ratio of approximately 1.0, compared to much lower ratio detected by ESCA and AESD analysis.
- Regenerated spent slurry from tests with roasted pyrites had a S/Fe ratio of 0.8, roughly a 20% decrease from the initial roasted pyrites.

TABLE 12

Test Results
Lab Scale vs 20 CFM Bench

14 g/l FeS

80 g/l Fe⁺⁺2000 ppm SO₂ (Lab)1000 ppm SO₂ (Bench)5% O₂

<u>Test Apparatus</u>	<u>FeS Type</u>	<u>moles SO₂</u> <u>mole FeS</u>	<u>% SO₂</u> <u>Removal</u>	<u>% NO_x</u> <u>Removal</u>
Lab	Roasted Pyr.	.003	100	81
Bench	Roasted Pyr.	.030	98	14
Lab	Precipitated	.101	100	95
Bench	Precipitated	.288	100	26

TABLE 13

Task 2

Regeneration

ESCA and IR Analysis

	Species Identified		<u>S/Fe Ratio</u>
	<u>ESCA</u>	<u>Infrared</u>	
Pyrites	FeS_x , FeSO_4	FeS_2 , FeSO_4	1.75
Roasted Pyrites	FeS_x , Iron Oxide	FeS	0.30

TABLE 14

Task 2

XRF Analyses (AESD Laboratory)

	<u>Elements Identified</u>	<u>S/Fe Ratio</u>
Pryites	Fe, S (major)	1.64
	Si, Al (minor)	
Roasted Pyrites	Fe, S (major)	0.55 - 0.91
Precipitated FeS (fresh)	Fe, S	0.97
Precipitated FeS (spent)	Fe, S	0.70
Regenerated		
---with CO	Fe, S (major)	0.32
	Si, Al, Cr, Ti, Ni (minor)	
--- without CO	Fe, S	0.16

TABLE 15

Tasks 1B and 2

Sample Analyses (ARD Laboratory)

<u>No.</u>	<u>Sample Description</u>	<u>Slurry Analyses (g/l)</u>			<u>X-Ray Fluorescence</u>		
		<u>Fe</u>	<u>SO₄</u>	<u>SO₃</u>	<u>% Fe</u>	<u>% S</u>	<u>S/Fe (Atomic)</u>
2-2F	Fresh Precipitated FeS	83	84	0.4	44.0	21.7	.86
2-2S	Spent Slurry	89	67	0.3	43.1	21.7	.88
2-2AR	Regenerated w/N ₂	0	14	0.3	72.3	15.8	.38
2-2BR	Regenerated w/N ₂ and CO	0	< 0.1	0	43.9	17.3	.69
2-4NG	Roasted Pyrites-Not Ground				48.3	25.1	.91
2-4G	Roasted Pyrites - Milled				41.3	23.9	1.01
2-4F	Fresh Slurry	84	59	0.3			
2-4S	Spent Slurry	87	48	0.3			
2-4RNG	Regenerated-Not Ground				47.5	22.5	.83
2-4RG	Regenerated - Milled				47.1	21.2	.79

TASK 3 - 20 CFM SCALE SCRUBBER CHARACTERIZATION WITH FIVE RECYCLES

Based on earlier tests results, it was decided to limit Task 3 to five recycles, using precipitated FeS as the starting material. Therefore, the regenerated spent solids from test 2-2 was used as the sorbent for test 3-1, which became the second recycle.

PERFORMANCE RESULTS

The performance results of Task 3 are shown in Table 16. The large decrease in FeS utilization for Cycle 2 was due to the fact that the regenerated solids were only milled for a short period of time. Subsequent tests all employed regenerated solids that were ground for 12 hours. This demonstrates the necessity of milling even for material that was originally precipitated FeS. Taking into account the abnormality for Cycle 2, the utilization still decreased approximately 25% for each recycle. Several explanations are possible for the decrease including the following:

- Gradual growth in average particle size.
- Oxidation during scrubbing or regeneration.
- Lowered reactivity caused by "dead-burning" during regeneration.

The growth in particle size could be caused by either sintering during regeneration or a gradual increase in average particle size from the original small precipitated particle size to the equilibrium size from the ball mill.

Studies conducted by Huuska, et al,⁽⁶⁾ indicated an autocatalytic reaction between iron sulfide and iron sulfate above 440°C resulting in the evolution of sulfur dioxide and the corresponding formation of iron oxides. The spent solids from the absorber were dewatered with a centrifuge prior to transfer to the dryer/regenerator but still retained approximately 30% by weight of the scrubbing liquor. The scrubbing liquor contained varying amounts of iron sulfate that undoubtedly reacted, in the afore-described manner, to generate iron oxides and lower the S/Fe ratio.

Sulfur dioxide removal from the five recycles was approximately 95%, while NO_x removal varied between 12% and 26%. As discussed earlier, the poor NO_x removal is attributed to below optimum gas flow velocity.

OFF-LINE ANALYSES

Data sheets for off-line analyses of the soluble species and XRF analysis of the solids are given in Appendices B and C. The results for the five recycles are summarized in Table 17. After the third regeneration, the spent slurry composition stabilized with 70-75 g/l Fe^{++} and 45 g/l $\text{SO}_4^{=}$. Analyses of regenerated solids showed a S/Fe ratio of approximately 0.55, which remained constant after the first regeneration. These results imply that oxidation of FeS during regeneration either reaches an equilibrium or is not important after the first regeneration. Oxidation of FeSO_4 may be occurring, however, and was indicated by mass balances around the regenerator.

TABLE 16

Task 3

Effect of Regeneration on Absorption

Initial Charge: 14 g/l FeS (Precip.)
80 g/l Fe⁺⁺

<u>Cycle</u>	<u>Moles SO₂</u> <u>Mole FeS</u>	<u>% No_x</u>	<u>% SO₂</u>
1	.288	26	100
2	.060	-	94
3	.173	12	95
4	.123	24	94
5	.082	-	95

Initial Charge: 14 g/l FeS (Roasted Pyrites)

1	.030	14	98
2	.030	-	91

TABLE 17

Task 3 Analysis Results

No.	Sample Description	Slurry Analysis (g/l)			X-Ray Fluorescence		
		Fe ⁺⁺	SO ₄ ⁼	SO ₃ ⁼	% Fe	% S	S/Fe (Atomic)
2-2F	Fresh Slurry	83	84	0.4	44	22	.88
2-2S	1st Spent Slurry	89	67	0.3			
2-2R	1st Regeneration				58	17	.51
3-1S	2nd Spent Slurry	80	41	0.4			
3-1R	2nd Regeneration				53	17	.56
3-2S	3rd Spent Slurry	82	51	0.2			
3-2R	3rd Regeneration				57	17	.52
3-3R	4th Spent Slurry	73	46	0.2			
3-3R	4th Regeneration				50	16	.56
3-4S	5th Spent Slurry	67	44	0.2	48	20	.73
3-4R	5th Regeneration				-	-	
3-5S	6th Spent Slurry	78	46	0.2			

TASK 4 - STEADY-STATE TESTING

"Steady-state" tests were run by supplying fresh reagent (FeS) to the recirculation tank to maintain a constant pH level. Spent slurry was removed by overflow to maintain a constant recirculation tank level. Several batches of precipitated FeS were prepared as starting material. One batch was used to charge the recirculation tank initially, and the pH was lowered to the set-point of pH 5 by contacting it with flue gas. Once pH 5 was reached, fresh FeS from a second batch was added as make-up. A third batch of fresh FeS was used as make-up until the first regenerated spent slurry was prepared. From this point on, regenerated FeS was used as make-up.

Samples of the fresh precipitated FeS and sixteen samples of slurry from the recirculation tank were analyzed for soluble iron and sulfur compounds. XRF analysis was performed on selected slurry samples and several regenerated solids samples. Data sheets are contained in the Appendices, while a summary of the results is given in Table 18. Calculation of the FeS utilization ratios for the steady-state tests are shown on Table 19.

The following observations can be made from these results:

- Utilization ratio with fresh precipitated FeS is approximately 0.6.
- Utilization ratio with one-time regenerated FeS (3 hours at 750°C) is 0.2.
- Regeneration for only 1 hour at 750°C results in a utilization ratio of only 0.1.
- Regeneration results in approximately a 20% decrease in S/Fe ratio, but subsequent regenerations do not effect this ratio. This confirms Task 3 results.
- Very little FeSO_4 build-up was observed during testing, implying that the FeSO_4 is being oxidized to insoluble oxides during regeneration.

TABLE 18
Steady-State Test Results
Off-Line Analysis

No.	Description	Date Sampled	Slurry Analysis (g/l)			% Fe	% S	S/Fe (Atomic)
			Fe ⁺⁺	SO ₄ ⁼	SC ₃ ⁼			
4-1F	Fresh Precipitated FeS	1/23	0	3	.05	42.0	21.2	.88
RT-1	Recirculation Tank	1/25	7	1	.05	38.5	20.6	.94
RT-2	Recirculation Tank	1/25	5	1	.05	40.3	24.0	1.04
RT-3	Recirculation Tank	1/28	5	1	.05	42.9	20.2	.82
RT-4	Recirculation Tank	1/28	7	1	.05			
RT-5	Recirculation Tank	-						
RT-6	Recirculation Tank	-						
RT-7	Recirculation Tank	1/30	6	1	.16			
RT-8	Recirculation Tank	2/1	5	1	.42	39.0	19.2	.86
RT-9	Recirculation Tank	2/4	6	.8	.16			
4-1 RNG	First Regeneration	2/6				46.0	10.5	.78
RT-10	Recirculation Tank	2/6	6	.9	.16			
RT-11	Recirculation Tank	2/8	6	1	.16	45.1	20.0	.78
RT-12	Recirculation Tank	2/11	6	2	.16	49.8	21.7	.76
4-2 RNG	Second Regeneration	2/11				44.8	20.4	.80
RT-13	Recirculation Tank	2/12	6	1.3	.13			
RT-14	Recirculation Tank	2/13	7	1.3	.13	50.6	21.7	.75
4-3 RNG	Third Regen.-Not Ground	2/12				44.1	19.4	.77
4-3 RG	Third Regen.-Milled	2/14				45.7	19.2	.74
4-4 RNG	Fourth Regeneration	2/14				45.8	19.5	.75
RT-15	Recirculation Tank	2/15	7	1.3	.13	48.4	10.6	.75
RT-16	Recirculation Tank	2/19	7	1.4	.13	47.1	20.0	.74

TABLE 19
Steady-State Tests
Utilization Ratios

<u>Testing Period</u>	<u>Make-Up Reagent</u>	<u>Utilization ($\frac{\text{Moles SO}_2}{\text{Mole FeS}}$)</u>
1/25 to 1/25	Fresh Precipitated	0.59
1/25 to 1/29	Fresh Precipitated	0.65
1/29 to 1/31	Fresh Precipitated	0.69
1/31 to 2/04	Fresh Precipitated	0.55
2/04 to 2/06	Regenerated (3 Hrs. at 750° C)	0.25
2/06 to 2/10	Regenerated (1 Hr. at 750° C)	0.10
2/10 to 1/12	Fresh Precipitated (1 Hr. at 750° C)	0.11
2/10 to 2/14	Regenerated (3 Hrs. at 750° C)	0.17
2/14 to 2/19	Regenerated (3 Hrs. at 750° C)	0.19

MISCELLANEOUS TEST RESULTS

THIOSULFATE TESTS

Initial tests for thiosulfate in the spent slurry samples were negative. However, it was later found that the ion chromatograph was very insensitive to thiosulfate and much stronger concentration ratios were required. Subsequent tests showed thiosulfate concentrations in the steady-state recirculation tank samples to be approximately equal to the sulfate concentration (based on mass of soluble sulfur). This implies the following concentrations of soluble sulfur species:

$\text{SO}_4^{=}$	4000 mg/l
$\text{SO}_3^{=}$	400 mg/l
$\text{S}_2\text{O}_3^{=}$	2300 mg/l

The presence of thiosulfate explains the electrical imbalance between soluble iron and sulfate/sulfite. The above concentration more nearly balances the ionic charge of the iron, the difference probably being due to errors in exact thiosulfate concentration and other minor anions, mostly chloride. Further verification of thiosulfate presence was provided by independent tests conducted by the Penn Environmental Consultants, Inc. (PEC). These tests were run by wet chemical means and the results are given in Appendix E. In general, the iron, sulfate, and thiosulfate results gave very good agreement with the ARD test results. The PEC values for sulfite were much higher than the ARD results. The reason for the differences was not resolved.

ULTIMATE ANALYSIS OF COAL

An ultimate analysis was conducted on the coal used for the steady-state tests and is shown in Table 20. The coal was found to contain 5.14% sulfur and 17.04% ash on a dry basis. Total moisture as received was 2.87%.

TABLE 20
Ultimate Analyses of Coal

	<u>As Rec. (Wt. %)</u>	<u>Dry (Wt. %)</u>
Carbon	65.39	67.32
Hydrogen	4.70	4.51
Oxygen	6.97	4.56
Nitrogen	1.40	1.44
Sulfur	4.99	5.14
Ash	16.55	17.04

Total moisture as rec. = 2.87% by Wt.

The above analysis was done by ASTM methods as follows:

- 1) Carbon & Hydrogen - ASTM D-3178-73
- 2) Oxygen - By difference
- 3) Nitrogen - ASTM D-3179-73
- 4) Sulfur - ASTM D-3177-75
- 5) Ash - ASTM D-3174-73
- 6) Total Moisture - ASTM D-3173-73

PROPOSED REACTION MECHANISM

It was not the intent of this test program to measure reaction kinetics or reaction mechanisms. However, several clues can be derived from the data as to the overall reaction products and several of the intermediate steps. Several of these observations have been noted above and include the following:

- One mole of Fe^{++} is produced per mole of SO_2 absorbed.
- Utilization ratios (moles of SO_2 absorbed per mole FeS) of greater than 0.75 have been observed, implying that a 1:1 ratio may be theoretically possible.
- Sulfate, sulfite, and thiosulfate have all been found as reaction products. No thiosulfate was found in tests run down to a low pH (4.5), but sulfite concentrations increased.
- Sulfur-rich iron sulfides were not detected from overall S/Fe measurements, but their presence could have been masked by high iron oxide concentrations.

Based on these observations, a theory was developed as to the SO_2 removal reaction mechanism. The overall reaction and proposed mechanism are given in Figure 5.

FIGURE 5

Proposed Reaction Mechanism

Overall Reaction:

Mechanism:

Equal molar production of thiosulfate, sulfite, sulfate and 2 moles FeS per mole SO₂ gives:

PROCESS FLOWSHEETS AND MATERIAL BALANCES

Task 5 was conducted solely by Westinghouse and consisted of an economic analysis of a commercial-sized SULF-X system applied to a utility boiler. Before conducting the economic analysis, it was necessary to prepare a detailed process flowsheet and material balance. A new process flowsheet was prepared and is shown in Figure 6. The terminal points for the SULF-X process are the electrostatic precipitator outlet and the stack inlet. Main features of the design are a quencher/TCA absorber, separate suspended solids and dissolved solids regeneration trains, and a wet milling system.

Details on the performance (as well as cost) of certain process components (e.g., fluid bed calciner, TCA absorber, etc.) were determined by reference to a TVA report prepared for EPA and entitled "Detailed Cost Estimates for Advanced Effluent Desulfurization Processes."⁽¹⁾ Performance of other components was estimated from engineering experience, handbooks, or manufacturers' brochures.

The basis for the material balances was a new 500 MWe coal-fired boiler burning 3.5% sulfur coal. The flue gas quantities and mass of SO_2 entering the system were taken from the TVA report to be comparable to the other processes analyzed by TVA. A material balance was prepared based on our best estimate of full-sized plant performance as indicated by the laboratory data. The bench-scale data were not used as a basis for the full-sized plant projections because of the recognized procedural and mechanical problems that could not be rectified before completion of the test program. The design parameters used for the material balance are given in Table 21. The system is designed for SO_2 and NO_x removal only.

Other simplifying and favorable assumptions utilized included high sulfur purity, ideal calciner performance (i.e., 100% yield and 100% stoichiometric feed), no FeS losses due to mechanical means or oxidation (other than oxidation due to reaction with FeSO_4) and no FeSO_4 production caused by NO_x reduction. In addition, it was assumed that FeS and SO_2 react according to the simplified overall reaction, Equation (1), producing only FeSO_4 and elemental sulfur as products.

Material balance sheets are given in Table 22. Stream numbers refer to those shown in Figure 6. Calculations and work sheets are presented in Appendix F.

TABLE 21

DESIGN PARAMETERS FOR MATERIAL BALANCES

	<u>Design Parameters</u>
Utilization Ratio	
$\frac{\text{Moles SO}_2}{\text{Mole Fe}}$	0.50
Oxidation in Indirect Rotary Kiln (Kiln I)	10%
Oxidation in Fluid Bed Calciner (Kiln II)	0%
% Solids in Recirc. Tank	40 g/l FeS
Fe ⁺⁺ Concentration	80 g/l
Fe ₃ O ₄ /FeS in Spent Slurry	25%
Exhaust from Calciner	Recycle
Sulfur Purity	Pure S
Calciner Yield	100% FeS
Calciner Stoichiometry	100%
Mechanical Losses	0
FeS Oxidation	0
FeSO ₄ from NO _x	0
Particulate Removal	0

TABLE 22

Material Balance
Stream

	1	2	3	4	5	6	7	8	9	10
<u>Description</u>	<u>Quencher Recirc. (x4)</u>	<u>Absorber Recirc. (x4)</u>	<u>Spent Slurry</u>	<u>Thickener Underflow</u>	<u>Cent. Return</u>	<u>Cent. Cake</u>	<u>Blow Down</u>	<u>Feed To Dryer</u>	<u>Dried Solids</u>	<u>Pyrite Make-Up</u>
Rate, M Lbs/Hr										
FeS			29.9	29.9		29.9	1.3	28.7	28.7	
S ⁰			10.9	10.9		10.9	0.5	10.4	10.4	
Fe ₃ O ₄			7.5	7.5		7.5	0.3	7.2	7.2	
FeS ₂										2.5
FeSO ₄ · 7 H ₂ O									9.4	
FeSO ₄ · H ₂ O										
FeSO ₄										
Fe ⁺⁺			59.8	7.5	4.3	3.2	0.1	3.1		
SO ₄ ⁼			102.5	12.9	7.4	5.5	0.2	5.3		
H ₂ O			733.0	92.2	52.7	9.5	1.6	37.9		
SO ₂										
S (g)										
H ₂ O (g)										
Fuel Gas, MSCF/Hr										
Other Gases										
Total	200	14,250	943.6	160.9	64.4	66.5	4.0	92.6	55.7	2.5
Spec. Grav.	1.25	1.25	1.25	1.55			1.93			
W/O Solids	5.1	5.1	5.1	30	0	50	50	50	100	100
GPM	320	22,800	1,509.7	208			4.1			
°F	127	127	127					60	400	60

TABLE 22 - Cont.

Material Balance										
Stream										
Description	11	12	13	14	15	16	17	18	19	20
Rate, M Lbs/Hr	Calcined Solids	Thickener Overflow	Feed to Cooler	Feed to Cryst.	Slurry Out Cryst.	Centrate Return	Recycle to Recirc.	Feed to Dryer	Dried Crystals	Prod. From Kiln
FeS	30.4									29.2
S ^o										
Fe ₃ O ₄	7.7									
FeS ₂										
FeSO ₄ · 7 H ₂ O					86.9			86.9		
FeSO ₄ · H ₂ O	7.5								56.4	
FeSO ₄										
Fe ⁺⁺		56.1	51.9	51.9	34.4	33.3	38.0	1.1		
SO ₄ ⁼		97.0	88.9	88.9	58.9	57.1	65.2	1.8		
H ₂ O		693.5	635.8	635.8	596.3	577.9	635.7	18.4		
SO ₂										
S (g)										
H ₂ O (g)										
Fuel Gas, MSCF/Hr										
Other Gases										
Total	45.6	846.5	776.6	776.6	776.5	658.3	738.9	108.2	56.4	29.2
Spec. Grav.			1.197	1.197			1.174			
W/O Solids	100	0	0	0		0	0			
GPM			1,297.6	1,297.6			1259			
°F	150	60	60	50	32			60	400	

TABLE 22 - Cont.

Material Balance											
Stream											
	21	22	23	24	25	26	27	28	29	30	31
Description	Milled Slurry	Feed Slurry	Dryer I Exhaust	Dryer II Exhaust	Fuel to Kiln	Kiln Exhaust	Fuel to Kiln I	Nitrogen Purite	Kiln I Comb. Gas	Kiln I Exhaust Gas	Sulfur to Storage Tank
Rate, M Lbs/ Hr											
FeS	59.6	59.6									
S ⁰											
Fe ₃ O ₄	7.7	7.7									
FeS ₂											
FeSO ₄ · 7 H ₂ O											
FeSO ₄ · H ₂ O											
FeSO ₄											
Fe ⁺⁺	2.8	2.8									
SO ₄ ⁼	4.7	4.7									
H ₂ O	93.5	420.8									
SO ₂											
S (g)										11.1	10.9
H ₂ O (g)			36.9	51.8		18.8	99.0				
Fuel Gas, MSCF/Hr					652		92.4				
Other Gases						42.5		9.7	700.0	9.7	
Total	168.3	495.6	36.9	51.8		61.3		9.7		20.8	
Spec. Grav.		1.11									
W/O Solids	40	14									
GPM		893									
°F	60	60						60			

TABLE 22 - Cont.
Material Balance
Stream

Description	32	33	34	35	36	37
	<u>SO₂</u> <u>to</u> <u>Quencher</u>	<u>Boiler</u> <u>Flue</u> <u>Gas</u>	<u>Gases</u> <u>to</u> <u>Quencher</u>	<u>Scrubber</u> <u>Exhaust</u> <u>Gas</u>	<u>Reheated</u> <u>Gas</u>	<u>Fuel Gas</u> <u>to</u> <u>Dryer I</u>
Rate, M Lbs/Hr.						
FeS						
S ⁰						
Fe ₃ O ₄						
FeS ₂						
FeSO ₄ · 7 H ₂ O						
FeSO ₄ · H ₂ O						
FeSO ₄						
Fe ⁺⁺						
SO ₄ ⁼						
H ₂ O						
SO ₂	0.4	23.8	24.2	2.4	2.4	
S (g)						
H ₂ O (g)		230	230	460	460	
Fuel Gas, MSCF/Hr.						162
Other Gases		4594				
Total		4848				
Spec. Grav.						
W.O Solids						
GPM						
°F	125	175	60			

TABLE 22 - Cont.

<u>Description</u>	Material Balance Stream				
	38 Fuel Gas to <u>Dryer II</u>	39 Make Up Water to <u>MPT</u>	40 Make Up Water to <u>SFT</u>	41 Steam From Waste Heat <u>Boiler</u>	42 Steam to <u>Boiler</u>
Rate, M Lbs/Hr					
FeS					
S ^o					
Fe ₃ O ₄					
FeS ₂					
FeSO ₄ · 7 H ₂ O					
FeSO ₄ · H ₂ O					
FeSO ₄					
Fe ⁺⁺					
SO ₄					
H ₂ O		93.5	226.8		
SO ₂					
S (g)					
H ₂ O (g)				40.8	17.6
Fuel Gas, MSCF/Hr.	259				
Other Gases					
Total	93.5	226.8			
Total		93.5	226.8		
Spec. Grav.		1.0	1.0		
W.O Solids					
GPM		187	353		
°F	60	60	60	470	470

ECONOMIC ANALYSIS

In order to determine the commercial viability of the SULF-X process, an economic analysis of a commercial-sized system was performed. However, a single-point estimate of a flue gas desulfurization process has limited usefulness due to the wide range of estimates caused by differences in scope of work, energy costs, plant location, environmental regulations, economic assumptions, date of estimate, etc. Therefore, it was decided to estimate the SULF-X process on a basis similar to that used for the TVA estimates. The SULF-X process could then be compared to three competing processes estimated by TVA:

1. Wet limestone slurry scrubbing
2. Magnesia slurry scrubbing with regeneration of sulfuric acid
3. Sodium solution scrubbing (i.e., Wellman-Lord process) with reduction to elemental sulfur

Capital costs and energy costs for the three TVA estimates were updated to a 1979 basis to make all systems comparable. A summary of the cost factors assumed for the study is given in Tables 23 and 23-X.

Capital cost estimates for the SULF-X process were prepared by dividing the system into the following major subsystems:

Area 1	-	Raw Materials Handling
Area 2	-	Feed Preparation
Area 3	-	SO ₂ Scrubbing
Area 4	-	Flue Gas Reheat
Area 5	-	Flue Gas Handling (Fan)
Area 6	-	Slurry Dewatering
Area 7	-	Sulfate Crystallization
Area 8	-	Solids Drying and Calcination
Area 9	-	Sulfur Recovery
Area 10	-	FeSO ₄ Drying and Calcination
Area 11	-	Services

Each subsystem was further subdivided into its major components. The type and capacity of each major component was determined and cost estimates made. Where possible, capital cost estimates and scaling factors were taken from the TVA report. Other cost estimates were derived from handbook or vendor data. The cost estimate work sheets, including component descriptions and estimate sources, are given in Appendix G.

After equipment costs (FOB) were calculated for each subsystem, direct investment costs were determined by applying installation factors. Separate installation factors were applied to each subsystem and were determined by comparing scope and equipment type with the TVA estimates.

Finally, total capital investment was calculated by applying the following additional factors to the direct investment:

Construction Facilities	=	5%
Engineering and Supervision	=	27%
Contingencies	=	10%
Allowance for Start-up	=	13.7%
Interest During Construction	=	15%

The capital cost summary is given in Table 24.

Operating costs were determined by first estimating energy requirements and raw material and by-product capacities. These values were derived from the flow rates given in Table 22 and the energy requirements summarized in Table 25. A comparison of energy requirements with the other three FGD processes is given in Table 26.

A secondary economic evaluation was conducted by PENSYS in order to consider available alternates to system design. It has always been SULF-X design philosophy to maximize the use of coal as the primary energy source for the regeneration phase. Accordingly, part of the system was redesigned and its overall capital and operating costs re-estimated. The basic system design and estimating methodology remained the same except for a 25% process contingency that was applied to the capital cost estimate for the regeneration and ancillary equipment. The operating costs were recalculated to reflect the utilization of coal instead of the other fossil fuels (calculations are in Appendix F).

In addition, waste disposal costs were included in the limestone system estimate, the capital cost for the magnesia slurry system was re-estimated, the unit cost for steam was recalculated at \$2.50/MM Btu, and the "by-product credits" for sulfur and heat were increased to \$90/ton and \$2.00/MM Btu, respectively. All these changes are presented in tables and figures located immediately after the tables and figures containing the respective original values.

The cost comparisons between the various processes are summarized in Tables 27 and 27-X and are graphically depicted in Figures 7 and 7-X. The capital and operating costs of the SULF-X process are higher than the other three (3) processes as was expected since SULF-X has the added capability of removing nitrogen oxides (95% in the laboratory). Once its full potential has been achieved through continued development, future comparisons should only be made with other emerging flue gas treatment (SO_2/NO_x) processes.

TABLE 23

Cost Basis for FGD System Comparison

Cost Basis - Mid-1979

C.E. Plant Cost Index = 237.5

Annual Charge on Capital = 0.18

Operating and Maintenance = 10% of Direct Investment

Energy Cost Basis

Steam	\$1.50/MM Btu
Electricity	\$0.03/Kwhr
No. 6 Fuel Oil	\$0.75/Gal.
Natural Gas	\$3.00/MCF
Reducing Gas - Med. Btu	\$5.00/MM Btu

Raw Material Cost Basis

Limestone	\$6/Ton
Soda Ash	\$70/Ton
Pyrites	\$20/Ton
MgO	\$200/Ton

By-Product Credits

Sulfur	\$60/Ton
Sulfuric Acid - 98%	\$30/Ton
Sodium Sulfate	\$30/Ton
Heat	\$1.25/MM Btu

TABLE 23-X

Cost Basis for FGD System Comparison (Revalued)

Cost Basis - Mid-1979

C. E. Plant Cost Index = 237.5

Annual Charge on Capital = 0.18

Operating and Maintenance = 10% of Direct Investment

Energy Cost Basis

Steam	\$ 2.50/MM Btu
Electricity	\$ 0.03/Kwhr
No. 6 Fuel Oil	\$ 0.75/Gal.
Natural Gas	\$ 3.00/MCF
Reducing Gas - Med. Btu	\$ 5.00/MM Btu
Coal	\$35.00/ton

Raw Material Cost Basis

Limestone	\$ 6/Ton
Soda Ash	\$ 70/Ton
Pyrites	\$ 20/Ton
MgO	\$200/Ton

By-Product Credits

Sulfur	\$90/Ton
Sulfuric Acid - 98%	\$30/Ton
Sodium Sulfate	\$30/Ton
Heat	\$ 2/MM Btu

TABLE 24

Capital Cost Summary (Thousands of 1979 Dollars)

Area	1	2	3	4	5	6
<u>Description</u>	<u>Raw Material Handling</u>	<u>Feed Preparation</u>	<u>SO₂ Scrubbing</u>	<u>Reheat</u>	<u>Gas Handling</u>	<u>Slurry Dewatering</u>
Equipment Cost (FOB)	119.1	672.0	3517.0	535.4	408.7	549.5
Install. Factor	2.10	2.35	2.37	1.36	3.00	2.10
Direct Invest.	250.1	1579.2	8335.3	728.1	1226.1	1154.0
Construction Facilities (5%)						
Total Direct Investment						
Eng. and Super. (27%)						
Contingencies (10%)						
Allowance for Start-Up (13.7%)						
Interest During Construction (15%)						
Total Capital Investment						

TABLE 24 - Cont.

Capital Cost Summary (Thousands of 1979 Dollars)

	7	8	9	10	11	
	<u>Sulfate</u>	<u>Solids</u>	<u>Sulfur</u>	<u>FeSO₄</u>	<u>Services</u>	<u>Total</u>
	<u>Crystallization</u>	<u>Drying and</u>	<u>Recovery</u>	<u>Drying and</u>		
		<u>Calcination</u>		<u>Calcination</u>		
Equipment						
Cost (FOB)	1375.1	2356.7	167.8	2295.3	139.8	12,136
Install.						
Factor	2.00	2.05	2.00	1.64	9.00	2.16
Direct Invest	2750.2	4831.2	335.6	3764.3	1258.2	26,167
Construction						
Facilities (5%)						1,308
Total Direct						
Investment						24,475
Eng. and Super.						7,418
Contingencies						2,747
Allowance For						
Start-Up						3,764
Interest During						
Construction						4,121
Total Capital						
Investment						45,525

TABLE 25

Energy Requirements - SULF-X

Fuel:

Fluid Bed Dryer I	$45.7 \times 10^6 \frac{\text{Btu}}{\text{hr}}$
Indirect Rotary Calciners	86.7×10^6 "
Fluid Bed Dryer II	73.1×10^6 "
Fluid Bed Calciner (Kiln II)	185×10^6 "

Steam:

Flue Gas Reheater	$7.04 \times 10^4 \frac{\text{lb}}{\text{hr}}$
Sulfur Condenser (Credit)	(2.7×10^4) "
Waste Heat Boiler (Credit)	(4.1×10^4) "

Electricity:

Fans	4 x 1275 HP
Absorber Recirc. Pump	10 x 600 "
Recycle Pump	2 x 500 "
Refrigeration Pump	2 x 400 "
Other Pumps	331 "
Agitators and Mixer	366 "
Centrifuge I	250 "
Centrifuge II	2 x 550 "
Ball Mill	2 x 675 "
Indirect Rotary Calciners (Kiln I)	7 x 25 "
Fluid Bed Dryer I	560 "
Fluid Bed Dryer II	785 "
Fluid Bed Calciner (Kiln II)	690 "
Miscellaneous	100 "
Total	18,067 HP

TABLE 26

Energy Requirements

	<u>Limestone</u>	<u>MgO</u>	<u>Sodium</u>	<u>SULF-X</u>
Steam				
lbs/hr	70,400	75,700	308.400	70,400
10^6 Btu/hr	63.4	68.1	277.6	63.4
Natural Gas				
SCFM				1540
10^6 Btu/hr				86.7
Reducing Gas				
SCFM				
10^6 Btu/hr			92	185
Fuel Oil				
gal/hr		765		792
10^6 Btu/hr		114.8		118.8
Heat Credit				
lbs/hr				68,000
10^6 Btu/hr		(14.4)	(11.8)	(84.5)
Electricity				
MW	11.2	10.2	10.6	13.9
10^6 Btu/hr	38.2	34.8	36.2	47.4
Total Energy				
10^6 Btu/hr	101.6	198.7	394	416.8

TABLE 27

Cost Summary - 1979 Basis

	<u>Limestone</u>	<u>Mgo</u>	<u>Sodium</u>	<u>SULF-X</u>
<u>Capital Costs</u>				
Direct Investment (10^6 \$)	23.1	23.5	27.1	27.5
Total Capital Invest. (10^6 \$)	37.1	38.9	44.9	45.5
\$/kW	74	78	90	91
<u>Operating Costs (mills/kW hr)</u>				
Depreciating Capital	1.91	2.00	2.31	2.34
Oper. and Maint.	0.66	0.67	0.77	0.79
Raw Materials	0.30	0.06	0.19	0.05
Energy Costs	0.86	1.96	2.39	4.58
Steam	0.19	0.20	0.83	0.19
Electric	0.67	0.61	0.64	0.83
Fuel Oil		1.15		1.19
Nat. Gas				0.52
Red. Gas			0.92	1.85
By-Product Credits	0	(1.01)	(0.70)	(0.86)
Sulfur			0.56	0.65
Sulfuric Acid		0.97		
Sodium Sulfate			0.11	
Heat		0.04	0.03	0.21
TOTAL (mills/kW hr)	3.73	3.68	4.96	6.90

TABLE 27-X

Cost Summary - 1979 Basis (Revalued)

	<u>Limestone</u>	<u>Mgo</u>	<u>Sodium</u>	<u>SULF-X</u>
<u>CAPITAL COSTS</u>				
Direct Investment (10^6 \$)	23.1	25.1	27.1	31.1
Total Capital Invest. (10^6 \$)	37.1	41.6	44.9	51.6
\$/kW	74	83	90	103
<u>OPERATING COSTS (mills/kW hr)</u>				
Depreciating Capital	1.91	2.14 ^b	2.31	2.65
Oper. and Main.	0.66	0.72	0.77	0.89
Raw Materials	0.30	0.06	0.19	0.05
Energy Costs	0.99	2.10	1.95	3.17
Steam	0.32	0.34	1.39	0.32
Electric	0.67	0.61	0.64	0.83
Fuel Oil		1.15		
Nat. Gas				
Red. Gas			0.92	2.02
Waste Disposal Cost	0.39 ^a			
By-Product Credits	0	(1.03)	(1.00)	(1.32)
Sulfur			0.84 ^c	0.98
Sulfuric Acid		0.97		
Sodium Sulfate			0.11	
Heat		0.06	0.05	0.34
TOTAL (mills/kW hr)	4.25	3.99	5.22	5.44

^a Assumes a waste disposal cost of approximately 10 percent of total annual cost.

^b Based on relationship between capital costs of magnesia slurry and sodium (Wellman-Lord) system assumed in EPRI report (P.6-99)

^c Based on a 22 percent discount from the current sulfur price of \$127/metric ton.

CONCLUSIONS

1. The SULF-X process is capable of removing $> 95\%$ of SO_2 from flue gas in both laboratory and bench-scale equipment.
2. Up to 95% NO_x removal is achievable under laboratory conditions, but bench-scale scrubbing only achieved $\leq 26\%$ during testing. NO_x removal is dependent on high soluble iron concentrations.
3. Sulfates, sulfites, and thiosulfates are all products of the SO_2 removal reaction. Thiosulfates are favored at pH of 5 and higher.
4. One mole of soluble iron is produced per mole of sulfur dioxide absorbed. Regeneration of soluble iron requires crystallization and chemical reduction or precipitation.
5. Regeneration reduces the absorption capability of precipitated FeS and requires particle size reduction (e.g., wet ball milling) before reuse.
6. Regeneration of FeSO_4 in the presence of FeS results in the production of iron oxides and the loss of available reagent.⁽⁶⁾
7. Assuming idealized regeneration, SULF-X process capital costs and energy requirements (Btu-basis) are comparable to the sodium (Wellman-Lord) process.
8. SULF-X energy costs and annual operating costs are approximately 2 mils per kilowatt-hour, or 39%, higher than the sodium system. Operating costs are over 3 mils per kilowatt-hour, or 85%, higher than the limestone or magnesia scrubbing systems.
9. Major technical problem areas that must be investigated in order to approach the idealized flow sheet conditions are sulfite/thiosulfate formation, and FeSO_4 self-oxidation during regeneration.
10. High energy costs per unit of heating value result from reducing gas requirements and liquid or gaseous dryer and calciner fuel requirements. Fuel costs for drying and regenerating could be lowered by using steam and coal, respectively.

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**APPLICATION OF THE SULF-X PROCESS
TO COAL CONVERSION AND UTILIZATION**

PHASE II EXTENSION FINAL REPORT

**Work Performed Under Contract DE-AC01-781123
(Formerly Contract ET-78-C-01-2779)**

APPENDIX

ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS

ESCA, Electron Spectroscopy for Chemical Analysis, was performed at Westinghouse Research Laboratories on several samples, including fresh FeS, fresh pyrite, roasted pyrite, and spent scrubber slurry. In order to determine the best testing method, several variations were made to the test procedure. Identical samples were prepared under an inert atmosphere (helium glove box) and under exposure to air. Surprisingly, the sample prepared in air showed a smaller percentage of oxidized sulfur (37 percent versus 46 percent) and a lower percentage of iron oxides. A subsequent second analysis of the sample exposed to air (this time tested under helium) showed a higher percentage of those results has been determined to date, but all further testing was conducted in the glove box to give comparable results.

Many of the early tests were conducted using a magnesium anode. The spectra from these tests had good resolution, but was very difficult to interpret. An aluminum anode was tried and gave a much cleaner spectra, but with poorer resolution. As a result, only a qualitative description of the presence of iron oxides could be obtained. All test results reported herein were obtained with the aluminum anode.

Table 1 gives the measured peak heights for six samples: pure FeS, pyrites (FeS_2), reagent grade FeSO_4 , roasted pyrite, spent slurry sample BS79, and spent slurry sample 1-6. All peak heights are measured relative to an internal standard (i.e. the Fe-3p peak was arbitrarily taken to have a value of 100). The Fe-2p peak is representative of the surface composition, while the Fe-3p peak is more of a bulk measurement. The carbon values shown are assumed to be due to interference from the cathode.

The results are summarized in Table 2, which gives sulfate-to-sulfide, total S-to-total Fe, and oxygen-to-iron ratios. It should be noted that these are ratios of "peak heights", and are not directly convertible to atomic ratios. For a true atomic ratio the sensitivity of the instrument to the various electron energy levels and the area under the peak must be considered.

TABLE 1: ESCA TEST RESULTS (AT ANODE)

Peak Due To	Peak Heights					
	FeS	Pyrites	FeSO ₄	Roasted Pyrites	BS79	Test 1-6
Fe-2p	186	260	213	222	213	233
Fe-3p	100	100	100	100	100	100
C-1s	200	230	174	206	153	286
O-1s	467	653	957	533	693	640
S-2p						
Sulfate	62	130	243	---	100 (SO ₃ ⁻)	52 (SO ₃ ⁻)
Sulfide	90	327	---	67	57	42
N	---	33	17	---	---	---
Si	---	16	---	28	13	22
Ca	---	---	---	22	---	---

TABLE 2: PEAK HEIGHT RATIOS (A1 ANODE)

	$\text{SO}_4^{=}: \text{S}^{=}$	$\text{S}/\text{Fe}(2p)$	$\text{O}/\text{Fe}(2p)$	Comments
FeS	41:59	0.82	2.51	
Pyrites	28:72	1.75	2.51	
Roasted Pyrites	0:100	0.30	2.40	Iron Oxide Observed
FeSO_4	100:0	1.14	4.49	Sulfate Could represent $\text{Fe}_2(\text{SO}_4)_3$
BS79	54:36 ($\text{SO}_3^{=}: \text{S}^{=}$)	0.74	3.25	Retested after previous exposure to air
Test 1-6	55:45 ($\text{SO}_3^{=}: \text{S}^{=}$)	0.40	2.75	Iron Oxide Observed

In comparing the FeS and FeS₂ samples, it can be seen that the measured S/Fe ratio for the pyrites sample is approximately double that for the FeS sample. This is in accordance with expectations. The degree of sulfur oxidation (% SO₂) and the O/Fe ratio are less useful. ESCA is a surface measurement, and the composition of the first few molecular layers is not necessarily representative of the bulk composition. Since any oxidation would occur on the surface, ESCA results tell little about the overall composition. Further obscuring the oxygen measurements is the ability of iron sulfate to retain water of hydration. ESCA measurements are not able to distinguish between the oxygen present as FeSO₄, FeSO₃, iron oxides, water, and other oxygen-containing species.

Concentrating our attention on the S/Fe ratio, we see that the FeSO₄ has slightly greater than a one-to-one ratio, while the roasted pyrites and spent slurry samples (BS79 and 1-6) are deficient in sulfur. Sample BS79 is the one that was first exposed to air and then re-tested under helium. The initial test results on this sample under helium, without air exposure, were similar to those for sample 1-6. Therefore, the listed results for BS79 will not be considered in the subsequent discussion.

The only difference between roasted pyrites and spent slurry (test 1-6) was the exposure to flue gas and the resultant absorption of SO₂. Although both samples have a very low S/Fe ratio on the surface, we do see an increase between the fresh slurry (roasted pyrites) and the spent slurry (test 1-6) of 0.30 to 0.40. Also, we qualitatively measure the presence of iron oxides in both of these samples. The sulfur oxides in the spent slurry samples are SO₃⁼, while those measured in the fresh slurry are SO₄⁼.

In order to obtain further analytical data on these samples, an infrared spectrometry was used. The results are shown in Table 3. It should be noted that IR gives a bulk composition, whereas ESCA is a surface measurement. As might be expected, the pyrites sample shows the presence of FeS₂, while the fresh and spent slurry samples show FeS. No iron oxide bands could be identified in the samples. The identification of iron oxide bands would have established the presence of these compounds; however, the absence of such bands does not necessarily preclude their presence.

TABLE 3: INFRARED SPECTRA

<u>Sample</u>	<u>Frequencies Observed</u>	<u>Interpretation</u>
Pyrites	417, 375, 348, 1150	$\text{FeS}_2 + \text{SO}_4^{-2}$
Roasted Pyrites	620, 520, 375	FeS
BS79	620, 520, 375	FeS
Fe_2O_3	560, 470, 310	
Fe_3O_4	570, 370	

To summarize the ESCA results, we can make the following conclusions:

- 1) The Al anode gives a much cleaner spectra, but poorer resolution, than the Mg anode.
- 2) The analysis is dependent on particle size and the method of sample preparation.
- 3) Air oxidation results in sulfates, whereas aqueous solutions yield sulfites.
- 4) There is a depletion in S/Fe ratio in going from pyrites to roasted pyrites, and an increase after contact with the flue gas.
- 5) Iron oxides may represent a significant component of roasted pyrites and the spent slurry samples.

DATA Sheet

	P11	P1	P11
Fe 2p	186 (surface peaks)	260	213
Fe 3p	100 (more h/k)	100	100 — internal std.
C 1s	200	230	174
O 1s	467	553	957
S 2p	62 — sulfate 90 — sulfide	130 227	243
N	—	33	17
Si	—	16	—
	PH BE	PI BE	PI BE
Fe 2p	222 710.2	213 714.5 709.5	233
Fe 3p	100 55.8	100 55.1	100
C 1s	206 284.6	153 284.6	286
O 1s	533 530.6 529.2	693 531.0	640 531.0 529.0
S 2p	—	100 167.1 57 161.6	52 42
Ca	22	—	—
Si	28	155	—

WRL Comments

1. All results given under electron spectra
but resolution is poor - only a qualitative
description is obtained about the oxide

2. BS 79 was under three different environments

SO_4^{2-} or SO_3^{2-} : S^{2-}

oxide

a. opened in glove box

46 : 54

sulfate

b. opened in air

37 : 63

small

c. (b) opened in glove box at a later date

64 : 36

None

3. Analysis depends on

a. particle size

b. how the sample was prepared

4. Air oxidation gives sulfates

water solutions go show sulfites

FeS_2 observed only in Pyrite sample

APPENDIX B

SLURRY ANALYSIS DATA SHEETS

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TABLE 3-1: TEST NUMBER CROSS-REFERENCE

<u>Test No.</u>	<u>Sample No.</u>	<u>Sample Description</u>
1 - 4	1 - 4	Spent Slurry
1 - 5	1 - 5	" "
1 - 7	1 - 7	" "
1 - 11	1 - 11	" "
2 - 2	2 - 2 F	Fresh Slurry
	2 - 2 S	Spent Slurry
	2 - 2 aR	Regen. Solids - a
	2 - 2 bR	" " - b
2 - 4	2 - 4 NG	Roasted Pyrites (not ground)
	2 - 4 G	Roasted Pyrites (milled)
	2 - 4 F	Fresh Slurry
	2 - 4 S	Spent Slurry
	2 - 4 RNG	Regen. Solids (not ground)
	2 - 4 G	" " (milled)
3 - 1	3 - 1 S	Spent Slurry
	3 - 1 RNG	Regen. Solids (not ground)
3 - 2	3 - 2 S	Spent Slurry
	3 - 2 RNG	Regen. Solids (not ground)
3 - 3	3 - 3 S	Spent Slurry
	3 - 3 RNG	Regen. Solids (not ground)
	3 - 3 RG	" " (milled)
3 - 4	3 - 4 F	Fresh Slurry
	3 - 4 S	Spent Slurry
3 - 5	3 - 5 S	Spent Slurry
4 - 1	4 - 1 F	Fresh Slurry
	4 - 1 R	Regen. Solids (not ground)
4 - 2	4 - 2 RNG	" " (" ")
4 - 3	4 - 3 RNG	" " (" ")
	4 - 3 RG	" " (milled)
4 - 4	4 - 4 RNG	" " (not ground)

TABLE B-1: TEST NUMBER CROSS-REFERENCE (cont.)

<u>Test No.</u>	<u>Sample No.</u>	<u>Sample Description</u>
4 - 1 thru 4 - 4 (Steady-State Tests)	RT - 1	Recirc. Tank - 1/25
	RT - 2	" " - 1/25
	RT - 3	" " - 1/28
	RT - 4	" " - 1/28
	RT - 5	" " - -
	RT - 6	" " - -
	RT - 7	" " - 1/30
	RT - 8	" " - 2/1
	RT - 9	" " - 2/4
	RT - 10	" " - 2/6
	RT - 11	" " - 2/8
	RT - 12	" " - 2/11
	RT - 13	" " - 2/12
	RT - 14	" " - 2/13
	RT - 15	" " - 2/15
	RT - 16	" " - 2/19

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg, Fe/cc

Soluble SO_3 as Mg, S/cc

SO_4 as Mg, S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

%

%

%

%

%

AL # ID

70354

1-4

424.7

172.3

252.4

1.0010

1.06

.019

.42

.012

.11

.044

3.5553

0.261.2

3.2891

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID

80-255

1-5

421.9

166.6

255.3

9982

1.25

.022

10

.003

13.3

.103

3630

1121 1091

5.55

25.09

IRON SULFIDE SLURRY ANALYSIS

AL # ID 86-423A

1-7

Gross Bottle Wt.

256.4

-Tared Bottle Wt.

172.3

Grams: Total Sample (Liquid, Salts, Residue)

84.1

Solution Specific Gravity grams/cc

0.9977

Solution: Soluble Fe as Mg Fe/cc

0.58

.010

Soluble SO₃ as Mg S/cc

0.015

.001

SO₄ as Mg S/cc

0.013

.0004

Residue: Wet Residue (Liq. + Salt)

1.8742

Dry Residue (Salt)

1.2009

Liquid in Residue

0.6733

X-ray

% S

% Fe

(Impurities)

%

%

%

%

%

%

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

• Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities)

%

%

%

%

%

%

Al. # ID

10-0257

1-11

428.6

166.6

312.

1.1924

8111

1.449

.021

.007

.51

1.591

5.4165

11.0728

1.3377

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID

22F 80-259

828.1

259.4

618.7

1.2269

8.262

0.36

8.4 ?

118.1559

56.8216

56.6898

61.4661

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt:

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

%

%

%

%

%

AL # ID

2-2-S 80-0200

874.7

257.4

615.3

1.2342

14.30299

1.2342

89.00

1.38

6.30

1430299

55.5801

87.1498

11.7 7/55

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fd/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

Al. # ID

2-2 AB

43.0

16.4

26.6

1.01

0.27

13.8

27.2694

19.5227

8.3467

IRON SULFIDE SLURRY ANALYSIS

AL # ID			
2-2-55			
Gross Bottle Wt.	32.7		
-Tared Bottle Wt.			
Grams: Total Sample (Liquid, Salts, Residue)			
Solution Specific Gravity grams/cc			
Solution: Soluble Fe as Mg Fe/cc	2.01		
Soluble SO ₃ as Mg S/cc	0.002		
SO ₄ as Mg S/cc	0.055		
Residue: Wet Residue (Liq. + Salt)	23.1255		
Dry Residue (Salt)	15.7850		
Liquid in Residue	7.8903		
X-ray % S			
% Fe			
(Impurities) %			
%			
%			
%			
%			
%			
%			

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO₃ as Mg S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID

2-4-F 82.0265

869.5

257.9

611.6

1.2280

84.09

1.502

0.32

.010

59

1.877

319.537

15.6239

16.9298

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) %

%

%

%

%

%

AL # ID

245-80-0260

86.30

258.5

609.5

1.2013

86.56

1.546

5.20

.009

1.8

1.520

29.1939

13.7317

15.4742

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

• Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID

3-1580-0269

914.0

306.6

607.4

1.2173

2-nd det/l

20.15

1.431

0.37

.012

11

1.281

139.1895

66.2624

72.9271

IRON SULFIDE SLURRY ANALYSIS

AL # 10
#0-411
32A

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

87.18

1.468

160

50.5

1.578

172

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

116.3568

37.2857

72.9711

X-ray % S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

173

100

AL # ID 80-412
3-3-X

73.15

0.16

2158

47,5327

12.6294

34,9043

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

80-415
AL # ID

3-4-F

175.9

172.9

302.3

1.1734

76.85

0.160

43.9

723.3

385.2

338.37

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) %

%

%

%

%

%

AL # ID 70-411

3-4-5

471.2

171.8

300.0

1.1965

66.612

0.110

113.9

55.118

157.7117

39.3733

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SC_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID

35S 80-0270

858.9

259.6

599.3

1.1908

77.20

0.17

4.6

12.0590

8.6251

3.3739

IRON SULFIDE SLURRY ANALYSIS

AL # ID 70-415			
4-1-F			
Gross Bottle Wt.	424.2		
-Tared Bottle Wt.	172.3		
Grams: Total Sample (Liquid, Salts, Residue)	251.9		
Solution Specific Gravity grams/cc	0.9968		
Solution: Soluble Fe as Mg Fe/cc	1.01		
Soluble SO ₃ as Mg S/cc	0.15		
SO ₄ as Mg S/cc	2.11		
Residue: Wet Residue (Liq. + Salt)	4.8774		
Dry Residue (Salt)	0.8349		
Liquid in Residue	4.0425		
X-ray % S			
% Fe			
(Impurities) %			
%			
%			
%			
%			
%			
%			

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO₃ as Mg S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID 8-416

RT-1

424.7

171.7

253.0

1.6020

6.66

.119

0.046

.001

0.979

.031

21.065

2.1066

18.9584

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO₃ as Mg S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) %

%

%

%

%

%

AL # ID 80-417

RT-2

1424.8

171.6

253.2

1.0129

4.89

0.046

0.912

38.449

2.8442

35.6049

IRON SULFIDE SLURRY ANALYSIS

AL # ID 80-46
RT- 3

Gross Bottle Wt.

407.7

-Tared Bottle Wt.

171.5

Grams: Total Sample (Liquid, Salts, Residue)

256.2

Solution Specific Gravity grams/cc

1.01266

Solution: Soluble Fe as Mg Fe/cc

4.97

Soluble SO_3 as Mg S/cc

0.0415

SO_4 as Mg S/cc

0.979

Residue: Wet Residue (Liq. + Salt)

244.9666

Dry Residue (Salt)

2.8595

Liquid in Residue

22.1031

X-ray % S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO₃ as Mg S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID 80-419

RT- 4

9.45/cc

7.49

.134

.046

.001

1.046

.035

38.7213

2.5835

35.7577

LYS

Waxed Bottle Mt.

Solution Specific Gravity grams/cc

Soluble SO_3 as H_2S S/cc

SO₄ as Mg S/cc

Dry Residue (Salt)

Liquid in Residue .

X-ray

% S.

% Fe

(Impurities) _____ %

८३

%

%

01
10

%

AL # ID

80-512

CT-7

426.11

1.0156

5.60

0.16

o. 98

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

- Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as liq S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray	% S
-------	-----

% Fe

(Impurities) _____ %

5

%

%

%

%

183

AL # ID			
81-513			
R78	1.28.1		
	172.1		
	256		
	1.0159		
	11.60		
	.42		
	.90		
	14.0607		
	2.0540		
	12.0067		

ALL 87615

१३

[illegible]

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities)

%

%

%

%

%

%

AL # ID

80-415

Rt 10

4.284

1.0169

6.4

0.16

0.856

IRON SULFIDE SLURRY ANALYSIS

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO₃ as Mg S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities) _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

Al. # ID

80-517

RT-12

958.8

171.4

587.4

1.0130

5.78

0.16

1.87

75831

36094

39732

IRON SULFIDE SLURRY ANALYSIS

IND. #2115

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Fe as Mg S

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

{Impurities} _____ %

_____ %

_____ %

_____ %

_____ %

_____ %

AL # ID

80-517

PT-13

804.7

1.0116

6.17

0.16

1.22

h. - 1.40

0.04

IRON SULFIDE SLURRY ANALYSIS

ASL - 8000

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO₃ as Mg S/cc

SO₄ as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray % S

% Fe

(Impurities) %

%

%

%

%

%

%

AL # ID

80-571

RT 14

10.116

295.3

505.7

1.0120

1.51

0.12

1.28

15.4381

7.1773

7.260%

IRON SULFIDE SLURRY ANALYSIS

8630

Gross Bottle Wt.

-Tared Bottle Wt.

Grams: Total Sample (Liquid, Salts, Residue)

Solution Specific Gravity grams/cc

Solution: Soluble Fe as Mg Fe/cc

Soluble SO_3 as Mg S/cc

SO_4 as Mg S/cc

Residue: Wet Residue (Liq. + Salt)

Dry Residue (Salt)

Liquid in Residue

X-ray

% S

% Fe

(Impurities)

%

%

%

%

%

%

AL # ID

80 576

RT 16

8059

297.3

508.6

1.0204

6.67

0.16

1.44

14.0189

6.4850

7.5339

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APPENDIX C

X-RAY FLUORESCENCE DATA SHEETS

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TABLE C-1: TEST NUMBER CROSS-REFERENCE

<u>Test No.</u>	<u>Sample No.</u>	<u>Sample Description</u>
1 - 4	1 - 4	Spent Slurry
1 - 5	1 - 5	" "
1 - 7	1 - 7	" "
1 - 11	1 - 11	" "
2 - 2	2 - 2 F	Fresh Slurry
	2 - 2 S	Spent Slurry
	2 - 2 aR	Regen. Solids - a
	2 - 2 bR	" " - b
2 - 4	2 - 4 NG	Roasted Pyrites (not ground)
	2 - 4 G	Roasted Pyrites (milled)
	2 - 4 F	Fresh Slurry
	2 - 4 S	Spent Slurry
	2 - 4 RNG	Regen. Solids (not ground)
	2 - 4 G	" " (milled)
3 - 1	3 - 1 S	Spent Slurry
	3 - 1 RNG	Regen. Solids (not ground)
3 - 2	3 - 2 S	Spent Slurry
	3 - 2 RNG	Regen. Solids (not ground)
3 - 3	3 - 3 S	Spent Slurry
	3 - 3 RNG	Regen. Solids (not ground)
	3 - 3 RG	" " (milled)
3 - 4	3 - 4 F	Fresh Slurry
	3 - 4 S	Spent Slurry
3 - 5	3 - 5 S	Spent Slurry
4 - 1	4 - 1 F	Fresh Slurry
	4 - 1 R	Regen. Solids (not ground)
4 - 2	4 - 2 RNG	" " (" ")
4 - 3	4 - 3 RNG	" " (" ")
	4 - 3 RG	" " (milled)
4 - 4	4 - 4 RNG	" " (not ground)

TABLE C-1: TEST NUMBER CROSS-REFERENCE (cont.)

<u>Test No.</u>	<u>Sample No.</u>	<u>Sample Description</u>
4 - 1 thru 4 - 4 (Steady-State Tests)	RT - 1	Recirc. Tank - 1/25
	RT - 2	" " - 1/25
	RT - 3	" " - 1/28
	RT - 4	" " - 1/28
	RT - 5	" " - -
	RT - 6	" " - -
	RT - 7	" " - 1/30
	RT - 8	" " - 2/1
	RT - 9	" " - 2/4
	RT - 10	" " - 2/6
	RT - 11	" " - 2/8
	RT - 12	" " - 2/11
	RT - 13	" " - 2/12
	RT - 14	" " - 2/13
	RT - 15	" " - 2/15
	RT - 16	" " - 2/19

The X-ray analysis program for Iron Sulfide residue has been completed.

The enclosed data shows the calibration and standard recovery.

Preparation of the standards used stable metal oxides and elemental sulfur.

SAMPLE BY SAMPLE REPORT

*****RESULTS*****

SAMPLE # 1 (STANDARD)

TIME : 200 SEC.

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.665	0.490	0.296 ± 0.584
S	9.549	1.951	20.400	20.544 ± 0.255
K	0.005	1.280	0.120	0.173 ± 0.011
CA	3.724	1.811	0.340	0.346 ± 0.025
TI	6.784	2.866	0.260	0.284 ± 0.015
CR	14.359	4.531	0.420	0.419 ± 0.010
NI	2.336	10.492	0.310	0.350 ± 0.038
FE	2329.195	13.300	1.000	50.759 ± 0.159
NI	16.326	4.262	0.220	0.270 ± 0.014
NO	8.582	1.658	0.250	0.228 ± 0.005

TOTAL CONCENTRATION OF SAMPLE # 1 :

LISTED = 23.010

CALCULATED= 73.769

NO CONCENTRATION VALUE WAS GIVEN FOR ELEMENT(S): FE

SAMPLE # 2 (STANDARD)

TIME : 200 SEC.

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	2.130	0.170	0.403 ± 0.400
S	13.881	2.299	24.400	24.638 ± 0.295
K	0.478	1.607	0.440	0.528 ± 0.107
CA	2.654	1.826	0.200	0.189 ± 0.023
TI	12.926	3.674	0.680	0.464 ± 0.021
CR	24.535	5.335	0.680	0.489 ± 0.012
MN	0.471	2.989	0.140	0.156 ± 0.020
FE	2376.829	13.367	48.400	48.537 ± 0.186
NI	27.701	10.782	0.700	0.701 ± 0.020
NO	19.638	2.237	0.520	0.450 ± 0.007

TOTAL CONCENTRATION OF SAMPLE # 2 :

LISTED = 76.750

CALCULATED= 77.154

SAMPLE # 3 (STANDARD)

TIME : 200 SEC.

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.826	0.400	0.403 ± 0.555
S	19.862	1.963	32.700	31.811 ± 0.364
K	0.357	1.093	0.330	0.286 ± 0.047
CA	8.441	2.144	0.940	0.957 ± 0.033
TI	9.227	2.713	0.520	0.504 ± 0.020
CR	42.113	5.407	1.310	1.321 ± 0.017
MN	9.319	17.448	0.980	0.965 ± 0.045
FE	1988.951	12.342	40.200	40.115 ± 0.173
NI	24.544	10.774	0.210	0.667 ± 0.020
NO	4.421	1.739	0.100	0.144 ± 0.004

TOTAL CONCENTRATION OF SAMPLE # 3 :

LISTED = 78.520

CALCULATED= 77.381

SAMPLE # 4 (STANDARD)

TIME : 200 SEC.

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.022	1.873	0.300	0.511 ± 0.564
S	27.191	2.559	42.800	41.798 ± 0.453
K	0.492	1.363	0.890	0.945 ± 0.161
CA	1.957	1.423	0.100	0.103 ± 0.020
TI	14.595	3.040	0.870	0.882 ± 0.025
CR	57.954	6.131	1.870	1.888 ± 0.021
MN	3.412	13.944	0.430	0.370 ± 0.032
FE	1762.124	11.042	33.900	34.011 ± 0.178
NI	15.725	5.464	0.240	0.221 ± 0.014
MO	29.509	3.056	0.630	0.648 ± 0.008

TOTAL CONCENTRATION OF SAMPLE # 4 :

LISTED = 82.030

CALCULATED= 81.379

SAMPLE # 5 (STANDARD)

TIME : 200 SEC.

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.865	1.000	0.403 ± 0.561
S	31.963	2.502	47.500	48.412 ± 0.501
K	0.363	1.177	1.080	1.041 ± 0.281
CA	7.560	1.900	0.810	0.811 ± 0.030
TI	3.144	2.071	0.070	0.055 ± 0.011
CR	52.193	5.907	1.690	1.706 ± 0.020
MN	10.406	17.909	0.840	0.863 ± 0.038
FE	1622.926	10.154	1.000	30.886 ± 0.175
NI	38.856	17.514	1.090	1.111 ± 0.024
MO	24.476	2.904	0.510	0.547 ± 0.008

TOTAL CONCENTRATION OF SAMPLE # 5 :

CALCULATED= 86.085

NO CONCENTRATION VALUE WAS GIVEN FOR ELEMENT(S): FE

SAMPLE # 6 (STANDARD)

TIME : 200 SEC.

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	2.136	0.760	0.603 ± 0.601
S	15.497	2.308	25.300	26.296 ± 0.310
K	0.358	1.437	0.470	0.576 ± 0.146
CA	5.105	2.010	0.490	0.494 ± 0.027
TI	7.334	2.891	0.310	0.331 ± 0.017
CR	29.342	5.553	0.820	0.820 ± 0.013
NN	4.289	11.084	0.480	0.472 ± 0.036
FE	2350.749	13.925	47.200	47.133 ± 0.191
NI	21.480	7.092	0.460	0.450 ± 0.017
MO	9.352	1.958	0.250	0.243 ± 0.005

TOTAL CONCENTRATION OF SAMPLE # 6 :

LISTED = 76.540

CALCULATED= 77.418

SAMPLE # 7 (UNKNOWN)

Fisher FeS

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	2.095	-	0.603 ± 0.595
S	14.214	2.141	-	24.619 ± 0.388
K	0.800	1.500	-	1.647 ± 0.261
CA	1.795	2.070	-	0.075 ± 0.021
TI	4.588	2.897	-	0.076 ± 0.010
CR	0.713	3.472	-	0.001 ± 0.005
NR	- 0.079	7.749	-	0.087 ± 0.024
FE	2420.009	13.821	-	49.789 ± 0.193
NI	14.318	3.765	-	0.166 ± 0.013
MO	1.988	1.587	-	0.095 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 7 :

SAMPLE # 11 (UNKNOWN) AL 8571

[1-4]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	1.733	315.267	-	0.396 ± 0.045
S	1546.519	295.481	-	21.277 ± 0.017
K	5.467	197.333	-	0.119 ± 0.001
CA	132.745	243.225	-	0.012 ± 0.002
TI	430.370	424.430	-	0.341 ± 0.002
CR	120.433	542.567	-	< 0.019 ± 0.000
MU	11.722	1178.425	-	0.074 ± 0.003
FE	223778.5	2152.500	-	43.849 ± 0.009
NI	1011.432	545.024	-	0.088 ± 0.001
BO	29.032	298.968	-	0.071 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 11 :
CALCULATED 44.222

201

SAMPLE # 12 (UNKNOWN) AL 8571

[1-5]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	235.532	-	< 0.342 ± 0.039
S	1497.370	221.430	-	23.742 ± 0.022
K	0.000	144.000	-	0.114 ± 0.000
CA	157.137	191.862	-	0.042 ± 0.002
TI	498.421	318.519	-	0.455 ± 0.005
CR	152.967	449.133	-	< 0.006 ± 0.000
MU	14.849	839.212	-	0.061 ± 0.004
FE	273989.7	1468.333	-	33.510 ± 0.006
NI	474.707	337.436	-	0.025 ± 0.001
BO	38.441	121.559	-	0.067 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 12 :
CALCULATED 58.571

SAMPLE # 13 (UNKNOWN) *AL 8571 [Fe S 99%]*

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	342.367	-	< 0.342 ± 0.047
S	2240.370	385.630	-	23.331 ± 0.018
K	25.332	190.667	-	0.165 ± 0.003
CA	182.745	367.255	-	0.001 ± 0.002
TI	772.706	466.296	-	0.259 ± 0.002
CR	0.000	637.333	-	< 0.039 ± 0.000
NI	0.000	1303.333	-	0.084 ± 0.002
FE	477272.2	2365.803	-	46.445 ± 0.011
NI	1056.289	510.821	-	0.094 ± 0.001
NO	169.656	257.344	-	0.077 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 13 :
CALCULATED= 76.760

SAMPLE # 14 (UNKNOWN) *AL 8571 [Fe Pyrite]*

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	58.400	305.600	-	1.690 ± 0.046
S	3922.323	394.667	-	36.326 ± 0.029
K	95.667	259.333	-	0.307 ± 0.004
CA	279.397	289.608	-	0.087 ± 0.002
TI	696.444	390.556	-	0.560 ± 0.003
CR	0.000	576.467	-	< 0.039 ± 0.000
NI	0.000	973.697	-	0.081 ± 0.002
FE	337602.7	1838.333	-	34.443 ± 0.010
NI	833.820	425.026	-	0.052 ± 0.001
NO	459.817	352.183	-	0.102 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 14 :
CALCULATED= 73.612

SAMPLE # 15 (UNKNOWN) *AL 8571 [11-17]*

27-11

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	28.733	377.267	-	0.241 ± 0.050
S	2453.815	401.185	-	24.398 ± 0.018
K	47.000	372.000	-	0.266 ± 0.006
CA	174.194	374.804	-	< 0.013 ± 0.002
TI	841.889	551.111	-	0.001 ± 0.001
CR	7768.767	1130.233	-	1.123 ± 0.001
MN	57.948	2331.333	-	0.109 ± 0.002
FE	451683.0	2785.833	-	47.488 ± 0.012
NI	4341.477	2498.103	-	0.723 ± 0.001
MO	620.226	314.774	-	0.123 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 15 :
CALCULATED= 75.164

SAMPLE # 16 (UNKNOWN) AL 8571 [2-2F]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	314.200	-	< 0.342 ± 0.045
S	1647.630	318.370	-	21.696 ± 0.018
K	0.000	212.647	-	0.114 ± 0.001
CA	112.447	307.353	-	< 0.038 ± 0.002
TI	2137.185	499.815	-	2.445 ± 0.004
CR	118.123	597.847	-	< 0.018 ± 0.000
MN	340.539	1582.041	-	0.348 ± 0.003
FE	285188.1	2223.333	-	44.027 ± 0.009
NI	951.348	500.590	-	0.076 ± 0.001
MO	77.376	412.624	-	0.070 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 16 :
CALCULATED= 69.262

SAMPLE # 17 (UNKNOWN) AL 8571 [2-4G]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
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[2-5]

SI	14.733	314.267	-	0.714 ± 0.045
S	1981.593	295.407	-	23.923 ± 0.020
K	45.000	234.000	-	0.157 ± 0.002
CA	231.510	295.490	-	0.167 ± 0.002
TI	741.859	411.111	-	0.276 ± 0.002
CR	4405.467	805.333	-	0.789 ± 0.001
MI	94.958	1483.574	-	0.161 ± 0.003
FF	245280.8	2148.333	-	41.287 ± 0.009
NI	3050.206	1688.974	-	0.486 ± 0.001
MO	526.366	248.634	-	0.114 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 17 :
CALCULATED= 69.975

SAMPLE # 18 (UNKNOWN)

AL8571 [2-4RG]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	362.867	-	< 0.342 ± 0.048
S	1742.926	378.074	-	21.244 ± 0.017
K	45.467	295.333	-	0.178 ± 0.003
CA	172.431	371.549	-	0.003 ± 0.002
TI	10367.48	1543.519	-	5.403 ± 0.005
CR	4558.267	849.733	-	0.718 ± 0.001
MI	172.048	1944.061	-	0.192 ± 0.003
FF	419148.5	2532.500	-	47.120 ± 0.010
NI	3168.504	1744.718	-	0.505 ± 0.001
MO	462.817	310.183	-	0.108 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 18 :
CALCULATED= 75.812

SAMPLE # 19 (UNKNOWN)

AL8571 [2-4NG]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	414.600	-	< 0.342 ± 0.051
S	2993.852	440.148	-	25.127 ± 0.018
K	720.667	321.333	-	1.162 ± 0.010

TI	995.815	495.185	-	0.151 ± 0.001
CR	2424.900	821.100	-	0.309 ± 0.001
NN	- 72.284	1795.273	-	0.058 ± 0.002
FF	471533.9	2747.500	-	48.254 ± 0.013
NI	2024.274	1199.333	-	0.277 ± 0.001
MO	640.495	337.505	-	0.125 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 19 :
CALCULATED= 75.979

SAMPLE # 20 (UNKNOWN) *AL 8571 [2-4ENG]*

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	33.200	353.800	-	1.306 ± 0.049
S	2111.232	345.778	-	22.533 ± 0.017
K	112.667	293.333	-	0.341 ± 0.004
CA	153.941	417.059	-	< 0.027 ± 0.002
TI	10715.67	1628.333	-	4.987 ± 0.005
CR	4592.733	941.247	-	0.493 ± 0.001
NN	301.055	1944.727	-	0.244 ± 0.002
FE	434798.3	2546.333	-	47.521 ± 0.011
NI	3269.544	1787.333	-	0.522 ± 0.001
MO	431.624	329.376	-	0.105 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 20 :
CALCULATED= 78.239

SAMPLE # 21 (UNKNOWN) *AL 8571 [3-45]*

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	8.467	318.533	-	0.556 ± 0.045
S	1445.333	306.667	-	20.015 ± 0.015
K	30.333	242.667	-	0.149 ± 0.002
CA	270.549	297.451	-	0.093 ± 0.002
TI	4559.593	917.407	-	2.888 ± 0.004
CR	1595.867	738.133	-	0.233 ± 0.001
NN	896.477	1943.515	-	0.490 ± 0.003

NI	1409.747	638.615	-	- 0.145 ± 0.001
NO	221.548	371.452	-	0.084 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 21 :
CALCULATED= 72.458

SAMPLE # 22 (UNKNOWN) *AL 8571 [2-25]*

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	51.667	300.333	-	1.646 ± 0.046
S	1617.481	328.519	-	21.705 ± 0.018
K	0.000	220.667	-	0.114 ± 0.001
CA	175.809	311.471	-	0.024 ± 0.002
TI	3004.852	713.148	-	2.726 ± 0.005
CR	138.167	557.833	-	< 0.014 ± 0.000
NN	366.656	1647.030	-	0.390 ± 0.004
FE	374212.2	2144.167	-	43.082 ± 0.009
NI	924.241	466.487	-	0.071 ± 0.001
NO	162.684	438.366	-	0.079 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 22 :
CALCULATED= 69.830

SAMPLE # 23 (UNKNOWN) *AL 8599 [RT-1]*

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	16.467	266.533	-	0.755 ± 0.042
S	1189.741	225.259	-	20.558 ± 0.019
K	210.000	276.000	-	0.194 ± 0.002
CA	212.922	291.078	-	0.099 ± 0.002
TI	2374.593	522.407	-	3.146 ± 0.066
CR	121.333	498.667	-	< 0.015 ± 0.000
NN	2.715	1043.576	-	0.087 ± 0.004
FE	317209.3	1780.833	-	38.481 ± 0.007
NI	858.478	358.974	-	0.061 ± 0.001
NO	113.215	296.785	-	0.076 ± 0.000

CALCULATED= 63.463

SAMPLE # 24 (UNKNOWN) AL 8599 [3-4F]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	33.200	232.800	-	1.180 ± 0.040
S	612.259	264.741	-	16.727 ± 0.014
K	0.000	228.667	-	0.114 ± 0.000
CA	190.529	241.471	-	0.077 ± 0.002
TI	7494.889	1046.111	-	8.049 ± 0.008
CR	2758.847	458.123	-	0.516 ± 0.001
MN	499.000	1633.758	-	0.721 ± 0.006
FE	335854.0	1822.500	-	41.806 ± 0.006
NI	1987.912	913.590	-	0.284 ± 0.001
MO	200.753	216.247	-	0.082 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 24 :
CALCULATED= 69.556

SAMPLE # 25 (UNKNOWN) AL 8571 2-2BR

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	258.067	-	< 0.342 ± 0.040
S	756.593	279.407	-	17.291 ± 0.014
K	43.667	245.333	-	0.133 ± 0.001
CA	238.039	306.961	-	0.113 ± 0.002
TI	4066.815	1025.185	-	4.998 ± 0.006
CR	3676.467	798.533	-	0.667 ± 0.001
MN	357.115	1815.091	-	0.470 ± 0.005
FE	359351.3	1998.333	-	43.887 ± 0.007
NI	2902.408	1411.128	-	0.463 ± 0.001
MO	167.086	199.914	-	0.079 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 25 :
CALCULATED= 68.443

SAMPLE # 26 (UNKNOWN) *AL 8599* [3-3RNG]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	11.333	302.667	-	0.428 ± 0.044
S	574.407	324.593	-	15.799 ± 0.012
K	37.333	300.667	-	0.140 ± 0.001
CA	273.529	341.471	-	0.118 ± 0.002
TI	11784.15	1596.852	-	4.722 ± 0.006
CR	4064.100	368.900	-	0.478 ± 0.001
BN	523.446	2071.151	-	0.506 ± 0.004
FE	415525.4	3301.667	-	50.313 ± 0.008
NI	2056.378	913.933	-	0.293 ± 0.001
MO	215.312	241.688	-	0.086 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 26 :
CALCULATED= 75.280

208

SAMPLE # 27 (UNKNOWN) *AL 8571* [2-2AR]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	184.133	-	< 0.342 ± 0.056
S	1152.512	195.481	-	15.848 ± 0.008
K	23.333	388.667	-	0.326 ± 0.019
CA	402.333	506.667	-	0.063 ± 0.001
TI	13463.04	2197.963	-	1.682 ± 0.002
CR	2545.300	1135.700	-	0.297 ± 0.001
MN	955.519	2386.848	-	0.258 ± 0.001
FE	450770.5	3708.333	-	72.336 ± 0.016
NI	2182.548	1090.744	-	0.301 ± 0.001
MO	285.129	320.871	-	0.091 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 27 :
CALCULATED= 91.512

SAMPLE # 28 (UNKNOWN) *AL 8599* [RT-2]

[R, 2]

ELEMENT	INTENSITY	INTENSITY	CONCENTRATION	CONCENTRATION
SI	22.200	213.800	-	0.903 ± 0.045
S	1933.593	329.407	-	23.987 ± 0.020
K	199.667	257.333	-	0.277 ± 0.003
CA	207.725	276.275	-	0.155 ± 0.002
TI	2457.111	563.889	-	2.518 ± 0.005
CR	122.633	539.367	-	< 0.017 ± 0.000
NI	- 11.532	1139.697	-	0.073 ± 0.003
FE	332937.9	1946.833	-	40.251 ± 0.009
NI	882.824	478.872	-	0.043 ± 0.001
NO	42.860	361.140	-	0.067 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 28 :
CALCULATED= 68.279

SAMPLE # 29 (UNKNOWN) AL 8599 [3-3RG]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	7.067	290.933	-	0.521 ± 0.043
S	499.630	268.370	-	15.609 ± 0.012
K	11.667	261.332	-	0.120 ± 0.001
CA	210.275	318.725	-	0.071 ± 0.002
TI	10493.74	1534.259	-	7.072 ± 0.006
CR	3847.333	827.667	-	0.669 ± 0.001
NI	613.201	1919.515	-	0.659 ± 0.004
FE	392307.9	2245.833	-	48.085 ± 0.007
NI	1743.318	883.615	-	0.233 ± 0.001
NO	190.796	239.204	-	0.081 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 29 :
CALCULATED= 73.121

SAMPLE # 30 (UNKNOWN) AL 8599 [3-1RNG]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	13.200	327.800	-	0.675 ± 0.046

K	31.000	278.000	-	0.15 ± 0.002
Ca	211.098	389.902	-	0.034 ± 0.002
Ti	10592.07	1512.943	-	5.017 ± 0.003
Cr	2846.933	884.067	-	0.423 ± 0.001
Mn	583.742	2031.091	-	0.432 ± 0.002
Fe	455252.9	2511.667	-	53.317 ± 0.009
Ni	2356.012	1202.385	-	0.346 ± 0.001
Mo	136.452	265.548	-	0.076 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 30 :
CALCULATED= 77.443

SAMPLE # 31 (UNKNOWN) *AL-8599* [RT-3]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	104.406	255.600	-	2.977 ± 0.046
S	1294.815	325.185	-	20.200 ± 0.017
K	203.233	298.667	-	0.244 ± 0.002
Ca	319.255	282.745	-	0.177 ± 0.002
Ti	2236.407	592.593	-	2.112 ± 0.006
Cr	153.000	584.000	-	< 0.010 ± 0.000
Mn	88.382	1134.364	-	0.167 ± 0.002
Fe	344103.8	2095.833	-	42.933 ± 0.008
Ni	899.274	460.923	-	0.067 ± 0.001
Mo	119.022	357.978	-	0.074 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 31 :
CALCULATED= 68.943

SAMPLE # 32 (UNKNOWN) *AL-8599* [3-2 RING]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	21.000	360.000	-	0.872 ± 0.049
S	974.252	364.741	-	16.763 ± 0.011
K	31.333	356.667	-	0.172 ± 0.004
Ca	226.314	415.686	-	0.030 ± 0.002
Ti	13744.19	1996.815	-	4.559 ± 0.005

NN	729.387	2311.091	-	0.428 ± 0.002
FE	493494.4	2902.500	-	57.060 ± 0.010
NI	2781.414	1438.231	-	0.427 ± 0.001
NO	197.978	286.022	-	0.082 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 32 :
CALCULATED= 81.013

SAMPLE # 33 (UNKNOWN)

AL 8571 [1-7]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	16.600	340.400	-	0.761 ± 0.047
S	2447.704	374.294	-	24.273 ± 0.018
K	84.667	271.333	-	0.306 ± 0.004
CA	316.745	297.255	-	0.100 ± 0.002
TI	930.148	506.852	-	0.078 ± 0.001
CS	8097.967	1074.033	-	1.246 ± 0.001
NN	120.542	2210.970	-	0.144 ± 0.002
FE	427476.5	2550.823	-	45.871 ± 0.011
NI	3827.563	1970.231	-	0.628 ± 0.001
NO	651.720	288.280	-	0.126 ± 0.000

TOTAL CONCENTRATION OF SAMPLE # 33 :
CALCULATED= 73.538

SAMPLE # 34 (UNKNOWN)

AL 8599 [4-1F]

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	302.067	-	0.342 ± 0.044
S	1459.444	315.556	-	21.214 ± 0.018
K	0.000	206.000	-	0.114 ± 0.001
CA	479.059	277.941	-	0.335 ± 0.002
TI	1563.741	459.259	-	1.449 ± 0.004
CR	83.567	569.433	-	< 0.024 ± 0.000
NN	222.632	1250.303	-	0.294 ± 0.003
FE	358898.1	1994.167	-	41.992 ± 0.008
NI	886.414	356.282	-	0.065 ± 0.001

TOTAL CONCENTRATION OF SAMPLE # 34 :
CALCULATED- 65.859

DO YOU WISH TO OUTPUT THE ORIGINAL INPUT DATA? NO

DESCRIPTION OF THE CORRECTION PROTOCOL AND RUN CONDITIONS

DATE: 2-15-80

MICROAMPS: 2
KILOVOLTS: 40
ENERGY SCALE: 0-20
FILTER: NO 200SEC
ANODE: H VAC

CONCENTRATION UNIT: %
ANALYSIS TIME: 200 SECONDS

PEAK AND BACKGROUND REGIONS

TYPE	#	CHANNEL
BACKGROUND	1	80
START REGION	1	85
END REGION	1	88
BACKGROUND	2	90
BACKGROUND	3	104
START REGION	2	113
END REGION	2	116
BACKGROUND	4	121
BACKGROUND	5	160
START REGION	3	164
END REGION	3	167
BACKGROUND	6	171
BACKGROUND	7	176
START REGION	4	182

74-8-38
8615

SAMPLE # 8 (UNKNOWN) RT-11

		BACKGROUND	STANDARD	CALCULATED
ELEMENT	INTENSITY	INTENSITY	CONCENTRATION	CONCENTRATION
SI	0.000	1.598	-	0.403 ± 0.520
S	8.314	1.516	-	19.969 ± 0.264
K	0.493	1.307	-	0.220 ± 0.038
CA	1.150	1.510	-	0.023 ± 0.021
TJ	17.679	3.584	-	1.043 ± 0.025
CP	9.775	3.185	-	0.321 ± 0.011
CU	0.585	7.068	-	0.070 ± 0.034
FE	1792.511	9.792	-	45.067 ± 0.137
NI	3.134	4.031	-	0.227 ± 0.012
CO	0.995	1.595	-	0.075 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 8 :
CALCULATED 67.589

213

SAMPLE # 9 (UNKNOWN) 4-4 RNG

		BACKGROUND	STANDARD	CALCULATED
ELEMENT	INTENSITY	INTENSITY	CONCENTRATION	CONCENTRATION
SI	0.010	1.550	-	0.543 ± 0.513
S	7.980	1.445	-	19.545 ± 0.262
K	0.417	1.223	-	0.220 ± 0.023
CA	1.094	1.509	-	0.014 ± 0.021
TJ	15.487	3.248	-	0.903 ± 0.023
CP	7.402	3.098	-	0.264 ± 0.010
NI	0.002	6.798	-	0.098 ± 0.036
FE	1304.825	9.992	-	45.838 ± 0.139
PT	2.747	3.823	-	0.209 ± 0.012
CO	0.742	1.558	-	0.070 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 9 :
CALCULATED 67.724

SAMPLE # 10 (QUANTUM) 4-1R

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.081	1.594	-	0.272 ± 0.525
S	8.884	1.606	-	20.456 ± 0.269
K	1.092	1.403	-	0.220 ± 0.061
CO	1.105	1.665	-	0.014 ± 0.021
TI	16.516	3.534	-	0.917 ± 0.023
CR	4.447	3.073	-	0.154 ± 0.009
MO	0.443	4.282	-	0.158 ± 0.034
FE	1821.310	10.494	-	45.951 ± 0.146
NI	1.397	3.593	-	0.134 ± 0.011
CU	0.865	1.590	-	0.073 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 10 :
CALCULATED 69.889

214

SAMPLE # 11 (QUANTUM) RT-15

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.145	1.695	-	0.074 ± 0.548
S	9.303	1.772	-	20.610 ± 0.269
K	0.478	1.387	-	0.220 ± 0.028
CO	1.610	1.625	-	0.037 ± 0.021
TI	17.620	4.025	-	1.029 ± 0.025
CR	13.635	3.930	-	0.450 ± 0.011
MO	0.443	8.515	-	0.152 ± 0.035
FE	2044.932	11.879	-	48.424 ± 0.164
NI	4.034	5.301	-	0.295 ± 0.014
CU	1.061	1.699	-	0.077 ± 0.003

TOTAL CALCULATED OF SAMPLE # 11 :
CALCULATED 71.222

4.3 R

WAVELENGTH	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
43	0.129	1.121	-	0.222 ± 0.505
45	11.39	1.506	-	19.207 ± 0.257
47	0.132	1.113	-	0.228 ± 0.029
49	1.352	1.388	-	0.065 ± 0.021
51	14.128	3.282	-	0.832 ± 0.023
53	2.120	3.065	-	0.294 ± 0.010
55	0.532	4.718	-	0.174 ± 0.038
57	1800.575	10.117	-	45.678 ± 0.135
59	2.974	3.729	-	0.221 ± 0.012
60	0.779	1.426	-	0.071 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 12 :
CALCULATED- 44.921

215

RT-14

WAVELENGTH	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
43	0.100	1.916	-	0.403 ± 0.549
45	10.212	1.937	-	21.740 ± 0.276
47	0.710	1.426	-	0.228 ± 0.044
49	1.102	2.048	-	0.001 ± 0.021
51	12.639	4.231	-	1.028 ± 0.024
53	14.041	4.434	-	0.480 ± 0.011
55	0.703	9.302	-	0.176 ± 0.032
57	2257.504	13.267	-	50.582 ± 0.190
59	5.050	6.100	-	0.314 ± 0.015
60	1.430	1.655	-	0.084 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 13 :
CALCULATED- 75.285

RT. 8

SAMPLE # 14 (UNKNOWN)

RT- 8

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.078	1.212	-	0.281 ± 0.460
S	7.024	1.296	-	19.158 ± 0.265
K	0.427	1.253	-	0.220 ± 0.031
CA	1.361	1.401	-	0.061 ± 0.022
TI	15.396	3.259	-	0.255 ± 0.024
CR	1.241	2.234	-	0.039 ± 0.003
NI	0.092	4.489	-	0.033 ± 0.033
FE	1383.871	7.354	-	39.048 ± 0.101
NI	0.337	2.298	-	0.082 ± 0.003
NO	0.641	1.859	-	0.068 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 14 :
CALCULATED= 59.996

216

SAMPLE # 15 (UNKNOWN)

RT- 12

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.741	-	0.603 ± 0.542
S	10.706	1.899	-	21.744 ± 0.277
K	0.292	1.283	-	0.221 ± 0.018
CA	1.061	1.719	-	0.003 ± 0.020
TI	15.238	3.522	-	0.756 ± 0.021
CR	6.877	3.677	-	0.200 ± 0.009
NI	0.441	7.982	-	0.148 ± 0.030
FE	2202.247	12.550	-	49.811 ± 0.185
NI	2.225	4.265	-	0.174 ± 0.012
NO	1.037	1.488	-	0.076 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 15 :
CALCULATED= 73.732

SAMPLE # 16 (UNKNOWN)

4-2 R. 3

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.618	-	0.403 ± 0.522
S	7.661	1.594	-	19.378 ± 0.263
K	0.380	1.150	-	0.220 ± 0.042
Ca	1.442	1.618	-	0.070 ± 0.023
TI	19.894	4.081	-	1.372 ± 0.030
CR	21.445	3.765	-	0.840 ± 0.015
NI	0.525	8.155	-	0.185 ± 0.043
FE	1296.017	9.992	-	44.133 ± 0.129
MO	8.154	6.147	-	0.496 ± 0.017
CO	1.610	1.740	-	0.083 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 16 :
CALCULATED= 67.379

217

SAMPLE # 17 (UNKNOWN)

RT-16

ELEMENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.758	-	0.403 ± 0.545
S	8.556	1.739	-	20.001 ± 0.266
K	0.527	1.373	-	0.220 ± 0.030
Ca	1.278	1.577	-	0.037 ± 0.021
TI	17.212	3.853	-	1.029 ± 0.025
CR	12.231	3.603	-	0.426 ± 0.011
NI	0.145	7.839	-	0.118 ± 0.037
FE	1927.069	10.971	-	47.101 ± 0.150
MO	3.622	5.138	-	0.252 ± 0.014
CO	1.089	1.591	-	0.077 ± 0.003

TOTAL CONCENTRATION OF SAMPLE # 17 :
CALCULATED= 69.863

SAMPLE # 18 (UNKNOWN)

U-2 R

SAMPLE # 18 (UNKNOWN)

4-2 R

ELEMENT	IDENTITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.541	-	0.603 ± 0.510
S	8.748	1.497	-	20.447 ± 0.269
K	0.067	0.903	-	0.220 ± 0.004
CA	0.926	1.164	-	0.019 ± 0.018
TI	8.662	2.208	-	0.421 ± 0.017
IT	4.054	2.834	-	0.137 ± 0.008
PR	0.559	4.374	-	0.176 ± 0.035
Fe	1794.560	9.754	-	44.798 ± 0.139
BI	1.229	3.186	-	0.131 ± 0.010
PO	0.529	1.171	-	0.066 ± 0.002

TOTAL CONCENTRATION OF SAMPLE # 18 :
CALCULATED 66.289

218

DO YOU WISH TO OUTPUT THE ORIGINAL INPUT DATA? NO

DESCRIPTION OF THE CORRECTION PROTOCOL AND RUN CONDITIONS

DATE: 2-1-90

NOISE: 0
COUNTS: 40
PEAK SCALE: 0-20
FILTER: NO PROCESS
SLOPE: 0.000

CALCULATION TIME: 1
TOTAL RUN TIME: 200 SECONDS

PEAK AND BACKGROUND REGIONS

std FeSF

FLUORENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	2.126	0.760	0.603 ± 0.701
S	15.132	2.308	25.300	26.435 ± 0.321
E	0.338	1.437	0.470	0.565 ± 0.125
F	5.105	2.010	0.490	0.494 ± 0.027
	7.334	2.291	0.310	0.322 ± 0.017
CF	29.542	5.553	0.820	0.831 ± 0.013
F	4.289	14.084	0.480	0.474 ± 0.025
S	2279.023	13.225	47.200	47.200 ± 0.221
III	7.998	7.092	0.660	0.424 ± 0.015
IV	9.512	1.958	0.250	0.243 ± 0.005

TOTAL CONCENTRATION OF SAMPLE # 6 :
 CALCULATED = 76.540
 CALCULATED = 77.592

219

SAMPLE # 7 (UNKNOWN) FeSF2 std FeSF

FLUORENT	INTENSITY	BACKGROUND INTENSITY	STANDARD CONCENTRATION	CALCULATED CONCENTRATION
SI	0.000	1.899	-	0.603 ± 0.566
S	14.242	2.203	-	25.295 ± 0.312
E	0.252	1.343	-	0.365 ± 0.020
F	4.347	2.133	-	0.425 ± 0.027
F	7.188	2.957	-	0.324 ± 0.017
CF	28.556	5.399	-	0.827 ± 0.014
III	5.005	13.660	-	0.543 ± 0.038
FE	2240.209	13.242	-	47.582 ± 0.210
III	10.505	7.820	-	0.554 ± 0.017
IV	9.024	1.776	-	0.237 ± 0.005

TOTAL CONCENTRATION OF SAMPLE # 7 :
 CALCULATED = 76.686

SAMPLE # 8 (UNKNOWN) RT-11

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APPENDIX D

SCANNING ELECTRON MICROSCOPE ANALYSIS

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SCANNING ELECTRON MICROSCOPE ANALYSIS

Advanced Energy Systems Division, SEM/EDAX

Tests conducted by the Materials Laboratory of the Advanced Energy Systems Division consisted of scanning electron microscope analysis (SEM), element mapping with EDAX, and quantitative analysis by X-ray fluorescence (XRF). Samples of pyrites, roasted pyrites (unmilled), roasted pyrites (after 12 hours in the ball mill), and spent slurry (Test 1-6) were analyzed.

The SEM photographs for the four samples are shown in Figures 1 and 2 for magnification multiples of 250 and 1500 times, respectively. There does not appear to be any significant change in particle size between the original pyrites and the roasted pyrites (before milling), both being approximately 15-25 microns in diameter. The roasted pyrites particles have a somewhat glassy surface appearance and more rounded edges than the original pyrites, apparently caused by the heating of the sample. There does not appear to be any fusing caused by the roasting process.

Comparison of the photographs of roasted pyrites (12 hour milling) and spent slurry (after contact with flue gas) shows little physical difference. Both appear to be clumps of fine particles, 1 micron in diameter or less. The milled sample was the result of subjecting the roasted pyrites to 12 hours of grinding in a small wet ball mill. Considering the reduction in particle size and the irregular appearance of the resultant particles, the sample surface area appears to have been increased by at least a factor of 10^3 .

Pyrites

Roasted Pyrites (Not Milled)

Figure 1. SEM Photographs (250 X) (Sheet 1 of 2)

Roasted Pyrites (12 Hour Milling)

Spent Slurry (Test 1-6)

Figure 1. SEM Photographs (250 X) (Sheet 2 of 2)

Pyrites

Roasted Pyrites (Not Milled)

Figure 2. SEM Photographs (1500 X) (Sheet 1 of 2)

Roasted Pyrites (12 Hour Milling)

Spent Slurry (Test 1-6)

Figure 2. SEM Photographs (1500 X) (Sheet 2 of 2)

Each of the four samples were further analyzed by superimposing images representing S and Fe on the photographs to show areas of enrichment or depletion. These sulfur and iron maps (not shown) showed a relatively homogeneous distribution of both elements. No particles could be identified that were enriched or depleted in either S or Fe.

The samples were analyzed quantitatively for S and Fe by use of an X-ray fluorescence spectrometer. The results are shown in Figure 3. The difference in S/Fe ratio between the roasted pyrites (not milled) and the milled pyrites is probably due more to differences in the roasting processes than to changes caused by grinding. The unmilled sample was the result of roasting pyrites mixed with carbon in a N_2 and CO atmosphere. The milled sample was not taken from the same regeneration batch, but was from an earlier roasting which was done without either C or CO present. This roasting was also done some months before the sample was analyzed, and oxidation could have occurred in the interim period.

In either case, the roasted samples show a decrease in S/Fe ratio as a result of thermal decomposition of the pyrites. This was further verified by the recovery of sulfur in the off-gas of the roasting vessel. Considering the roasted pyrites after milling as the fresh slurry, the increase in S/Fe ratio as a result of exposure to SO_2 -containing gases was from 0.55 to 1.06. As reported earlier, the ESCA results for these two samples (i.e. surface composition) were S/Fe ratios of 0.30 and 0.40. Therefore, both analyses indicate a sulfur enrichment of the particles as a result of SO_2 removal. However, it is not possible at this time to assign values to the S/Fe atomic ratio of the iron sulfide (i.e. value of X in FeS_x for the samples before and after SO_2 exposure).

Advanced Reactors Division Laboratory, SEM Photographs

Three samples were analyzed by SEM at the ARD laboratory on February 14, 1980. The three samples and the SEM results were the following:

- 1) Sample 2-4 S - Spent Slurry from test 2-4 employing roasted pyrites. Sample appeared as 100 - 200 micron diameter particles. The particles had a homogeneous, coral-like

Pyrites
S/Fe = 1.64

Roasted Pyrites (Not Milled)
S/Fe = 0.91

Roasted Pyrites (12 Hour Milling)
S/Fe = 0.55

Spent Slurry (Test 1-6)
S/Fe = 1.06

Figure 3. XRF Spectra

appearance. The ratio of sulfur-to-iron peak heights was approximately 0.9. A sulfur trace across a particle is shown in Figure 4. An iron map is shown in Figure 5.

- 2) Sample 2-4 RNG - Regenerated solids from test 2-4 before milling. Physical appearance was similar to 2-4S. The S/Fe ratio was 0.6. An SEM photograph of sample 2-4 RNG is shown in Figure 6 at 50 x magnification. Sulfur and iron maps are shown in Figures 7 and 8.
- 3) Sample 2-4 RG - Regenerated solids from test 2-4 after milling. Physical appearance was very similar to the other two samples. S/Fe ratio was 0.5.

Four additional samples were analyzed on February 22, 1980 with the following results:

- 1) Sample 4-1 F - Freshly precipitated FeS. Appeared as 200-250 micron diameter flat plates with five holes in the surface. Overall S/Fe ratio was 0.77, but the porous regions showed S/Fe = 0.22.
- 2) Sample RT-1 - Spent slurry from the recirculation tank. Appeared as 150-300 micron flat plates attached to a porous structure. S/Fe of flat plates was 0.16, while S/Fe of porous region was 0.39. A sulfur-rich region was located (see Figures 9, 10, and 11) with S/Fe of 0.74. An SEM photograph of a portion of a particle is shown in Figure 9, while Figures 10 and 11 are sulfur and iron maps, respectively.
- 3) Sample 3-3 RNG - Regenerated solids from test 3-3 before milling. Appeared as a mixture of fine 20 micron particles and one large particle with a very fine structure. S/Fe of fine particles was 0.38, while S/Fe of large particle was 0.51.
- 4) Sample 3-3 RG - Regenerated solids from test 3-3 after milling. Sample consisted of a single large particle with S/Fe = 0.38.

Figure 4. Sulfur trace across Sample 2-4S

Figure 5. Iron trace across Sample 2-4S

Figure 6. SEM Photograph (50 X) of Sample 2-4 RNG

Figure 7. Sulfur map for Sample 2-4 RNG (50 X)

Figure 8. Iron map for Sample 2-4 RNG (50 X)

Figure 9. SEM Photograph of Sample RT-1 (129 X)

Figure 10. Sulfur map of Sample RT-1 (129 X)

Figure 11. Iron map of Sample RT-1 (129 X)

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APPENDIX E

PENN ENVIRONMENTAL CONSULTANTS, INC.
DATA SHEETS

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DATE AND TIME		Pittsburgh Environmental & Emergency Systems, Inc.		B.P.E.C. PROJECT NO.	
1-31-80		Recycle Tank 1/30/80		353-9000	
E. CHEMIST		F. TYPE OF SAMPLE		G. SAMPLE NO.	
REM				5253	

[illegible]

PENN ENVIRONMENTAL CONSULTANTS, INC.
FORT PITT PROFESSIONAL BUILDING
1517 WOODRUFF STREET
PITTSBURGH, PA. 15220
412-381-1133

LAB ANALYSIS REPORT

CLIENT NAME: PITTSBURGH ENVIRONMENTAL & ENERGY SYSTEMS
ADDRESS: 67 OLD CLAIRTON ROAD
PITTSBURGH, PA 15226

PEC PROJECT NO: 0353-00
PEC JOB NUMBER: 9176
PEC SAMPLE NO: 52629

REPORT DATE: 02.25.80

ATTENTION: MR ED SHAPIRO

DATE RECEIVED: 02.18.80

SAMPLE IDENTIFICATION: SPENT RECYCLE TANK 2-15

PARAMETER	SYMBOL	RESULTS	UNITS
pH	pH	4.0	
SUSPENDED SOLIDS @ 105 C	S.S.	13363	MG/L
SULFATE ()	SO ₄	5200	MG/L
TOTAL SULFIDE BY NITROGEN GENERATION ()	S—, N ₂	< 02	MG/L
SULFITE ()	SO ₃	3700	MG/L
THIOSULFATE ()	S ₂ O ₃	9000	MG/L
TOTAL IRON ()	FE TOT	39.7	MG/L
DISSOLVED IRON	FE DISS	6430	MG/L
SULFUR, TOTAL	T. SULF.	20.72	%
ACID DIGESTION	ACID DIG.		

COMMENTS:

Reviewed and Approved by.

PENN ENVIRONMENTAL CONSULTANTS, INC.
FORT PITT PROFESSIONAL BUILDING
1517 WOODRUFF STREET
PITTSBURGH, PA. 15220
412-381-1133

LAB ANALYSIS REPORT

CLIENT NAME: PITTSBURGH ENVIRONMENTAL & ENERGY SYSTEMS
ADDRESS: 67 OLD CLAIRTON ROAD
PITTSBURGH, PA. 15236

PEC PROJECT NO: 0353-00
PEC JOB NUMBER: 9183
PEC SAMPLE NO: 52667

REPORT DATE: 02.25.80

ATTENTION: MR. ED SHAPIRO

DATE RECEIVED: 02.19.80

SAMPLE IDENTIFICATION: FeS SLURRY AFTER SO₂ ABSORPTION

PARAMETER	SYMBOL	RESULTS	UNITS
PH	PH	4.0	
SUSPENDED SOLIDS @ 10 °C	S.S.	12220	MG/L
SULFATE	SO ₄	5200	MG/L
TOTAL SULFIDE BY NITROGEN GENERATION	S ²⁻ , N ₂	< 0.2	MG/L
SULFITE	SO ₃	3600	MG/L
THIOSULFATE	S ₂ O ₃	11200	MG/L
TOTAL IRON	FE TOT	39.5 % <i>total iron</i>	MG/L
DISSOLVED IRON	FE DISS	6360	MG/L
SULFUR, TOTAL	T. SULF.	21.50	%
ACID DIGESTION	ACID DIG		

COMMENTS

Reviewed and Approved by:

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