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Project Title: Combined Chemical and Microbiological Removal of Organic Sulfur from Coal DE-FG22-90PC90176

Principal Investigator: Leo A. Raphaelian, Institute of Gas Technology

Project Manager: Daniel D. Banerjee, CRSC

ABSTRACT

The objective of this work is to investigate techniques for chemically converting the sulfur containing organic compounds in coal to compounds that can be treated microbiologically to remove the organically bound sulfur.

The goal is to achieve an economically feasible mild chemical oxidation of the organic sulfur in a representative Illinois Basin coal by converting the sulfur to sulfoxides and sulfones; the carbon sulfur bond in the sulfoxides and sulfones would then be broken microbiologically and the sulfur removed from the coal as a sulfate.

The following oxidants were tried under a variety of conditions: hydrogen peroxide, potassium permanganate, sodium periodate, sodium perborate, and potassium persulfate. The bacteria used was IGTS8, a culture developed at IGT to remove sulfur from fossil energy products.

It was found that the reduction of total sulfur in IBC-107 by chemical oxidation was 6.2 to 18.1% and of oxidized coal by microbial desulfurization 9.6 to 22.4%. The overall reduction, combined chemical and microbial treatment, was 22.2 to 27.2%. Differences in the removal of sulfur by the combination treatments with varying oxidants were very small.

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EXECUTIVE SUMMARY

The objective of this work was to investigate techniques for chemically converting the sulfur containing organic compounds in coal to compounds that can be treated microbiologically to remove the organically bound sulfur.

The goal was to achieve an economically feasible mild chemical oxidation of the organic sulfur in a representative Illinois Basin coal by converting the sulfur to sulfoxides and sulfones; the carbon sulfur bond in the sulfoxides and sulfones would then be broken microbiologically and the sulfur removed from the coal as a sulfate.

The oxidants used in this study were hydrogen peroxide, potassium permanganate, sodium periodate, sodium perborate, and potassium persulfate. All oxidants were reacted with the coal, IBC-107, at room temperature. In addition, hydrogen peroxide was reacted at elevated temperatures, with and without the catalyst, acetic acid. The bacteria used was IGTS8, a culture developed at IGT to remove sulfur from fossil energy products. A variety of blanks were run to determine the changes in sulfur content due to chemical treatment alone and microbiological treatment alone. A total of 91 samples were analyzed for total sulfur. Those samples that gave promising results were analyzed for sulfur-by-type, elemental carbon, hydrogen, and nitrogen (ultimate analysis), moisture, and heating value.

Three sets of experiments were done. The first two sets were exploratory runs with hydrogen peroxide, potassium permanganate, and sodium periodate. Since hydrogen peroxide was the most promising oxidant in the first two sets of experiments, four different experimental conditions with hydrogen peroxide as the oxidant were used in the third set of experiments along with two new oxidants, sodium perborate and potassium persulfate.

With potassium permanganate, manganous dioxide was formed and could not be separated from the coal. Thus, inconsistent results were obtained and further study of this oxidant was abandoned.

Also, the removal of bacteria or of products associated with the microbial desulfurization from the coal was not complete in certain cases. This led to incorrect analytical results. However, a procedure was found to correct for these inaccuracies and total sulfur and other analytical data could be obtained.

It was found that the reduction of total sulfur in IBC-107 coal by chemical oxidation was 6.2 to 18.1% and of oxidized coal by microbial desulfurization 9.6 to 22.4%. The overall reduction of sulfur, chemical and microbial desulfurization, was 22.2 to 27.2%.

OBJECTIVES

The objective of this work is to investigate techniques for chemically converting the sulfur containing organic compounds in coal to compounds that can be treated microbiologically to remove the organically bound sulfur.

The goal is to achieve an economically feasible mild chemical oxidation of the organic sulfur in a representative Illinois Basin coal by converting the sulfur to sulfoxides and sulfones; the carbon sulfur bond in the sulfoxides and sulfones would then be broken microbiologically and the sulfur removed from the coal as a sulfate.

INTRODUCTION AND BACKGROUND

Of the sulfur in Illinois Basin coals, 30% or more is organic sulfur, that is, bound in organic molecules such as alkyl or aryl sulfides, mercaptans, disulfides, thiophenes, and sulfonic acids. Since the organic sulfur is bonded to carbon, chemical removal is difficult without destroying some of the organic compounds present in the coal and lowering the BTU or fuel value. Likewise, no one specific physical, chemical, or microbial treatment of all sulfur species in coal, namely, pyritic, organic, elemental sulfur, etc., has successfully reduced the total sulfur sufficiently without excessive destruction of the coal. However, by making use of the strong points of each treatment technique, stepwise use of a combination of these techniques might lead to enhanced removal of sulfur.

In this program, a stepwise approach for removal of organic sulfur will be used: The organically bound sulfur compounds in coal will be modified chemically to make them more susceptible to attack by microorganisms. Then, the microorganisms will be used to break the carbon-sulfur bond and remove the sulfur from the organic molecules converting the organic sulfur to inorganic sulfur that can be easily washed away from the coal. One important aspect of this approach will be to restrict the chemical conversion. Only mild chemical oxidation will be used to give rise to a product that can be microbiologically converted to a coal that is free of sulfur and high in fuel value.

This combination approach also leads to favorable process economics: The chemical conversion of organosulfur compounds to products that are amenable to microbial desulfurization can be done efficiently with relatively inexpensive materials at low temperatures. The critical step of selectively breaking the carbon-sulfur bond without excessive decomposition is left to relatively slow microbiological techniques.

EXPERIMENTAL PROCEDURES

In the first set of experiments, three oxidants were reacted in an aqueous solution for three hours at room temperature with the coal, IBC-107, ground to -200 mesh. The three oxidants were hydrogen peroxide, potassium permanganate, and sodium periodate. After three hours, the mixture of coal and oxidant in solution was filtered, washed five times with 100mL of deionized water, and dried in an oven overnight at 105°C. A chemical blank was also run in which no oxidant was added. The reactants used in the four experiments were as follows:

1) 15g coal in 7.5mL of 30% hydrogen peroxide and 192.5mL deionized water.
2) 15g coal in 10.8g potassium permanganate in 200mL deionized water.
3) 40g coal in 41g sodium periodate in 200mL deionized water.
4) 15g coal in 200mL deionized water.

In each case, the oxidant solution was added to the coal over a period of approximately five minutes.

Each of these four samples of coal, hydrogen peroxide treated, potassium permanganate treated, sodium periodate treated, and no oxidant, were split into three groups: 1) samples that were subjected to microbiological treatment with bacteria, 2) samples that were subjected to the biological medium with no bacteria added, and 3) samples that were not treated microbiologically at all.

Three more samples were prepared: They included a chemical blank in which there was only microbiological treatment, a microbiological blank in which there was only chemical treatment, and a chemical and microbiological blank which was untreated coal.

Microbial desulfurization was performed with bacteria (IGTS8) known to be effective in removing sulfur from coal. Two grams of the coal sample was placed in 2L of sulfur-deficient bacterial growth medium, inoculated with the

IGTS8 bacteria, and incubated at 30°C for 2-3 weeks*. When this incubation was completed, the coal was recovered by centrifugation, washed free of bacteria by differential centrifugation, and dried at 105°C. Media for the growth of the microorganisms (BSM medium) consisted of 4g of dipotassium hydrogen phosphate, 4g of disodium hydrogen phosphate, 2g of ammonium chloride, 0.2g of magnesium chloride hexahydrate, 0.001g of calcium chloride dihydrate, and 0.001g of ferric chloride hexahydrate per liter of distilled water.

Glycerol, at a concentration of 20mM, was added to BSM to serve as a carbon and energy source for microbial growth. All experiments were performed using 2g samples of coal, placed in 2L of sterile BSM in 4L Erlenmeyer flasks. Samples were inoculated with Rhodococcus rhodochrous IGTS8 at an initial cell density of 10^5 cells/mL. All 14 flasks were then incubated at 30°C with shaking for 27 days. Microbial growth was monitored frequently by removing 1mL samples, centrifuging at 14,000 x gravity for 2 minutes, and measuring the size of the bacterial pellet. The incubation period was terminated when maximum growth, as determined by three consecutive days with no measurable increase in microbial cell density, had taken place.

Harvesting of microbially-treated coal samples was performed by centrifuging at 10,000 x gravity for 10 minutes to obtain pellets which consisted of a mixture of coal and bacteria. These pellets were suspended in sterile water and centrifuged from 500 to 3000 x gravity such that coal pelleted and bacterial cells remained suspended. The supernatant containing bacteria was removed, the coal pellet suspended in distilled water, and the process was repeated until no more bacterial cells could be recovered in the supernatant. The coal pellet was then suspended in 50mL of distilled water, placed in a boiling water bath for 10 to 15 minutes, and centrifuged at about 1000 x gravity for 2 minutes. The supernatant, which contained bacterial

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*In tests late in 1990, it was found that the best biodesulfurization of coal, that is, healthy microbial growth (approximately 10^8 cells/mL) at the expense of sulfur in coal, is achieved in very diluted solutions of seven liters or more of microbial growth medium per gram of coal. Due to limitations in equipment, it was decided that it would be best to run an initial screen of oxidant/microbiological experiments with fourteen fermenters at a more concentrated solution of one gram of coal per liter of microbial growth medium. To prepare for the second phase of experiments that would follow the initial screen, sixteen fermenters with a capacity of twenty liters each were built while the initial experiments were being carried out (Appendix I).

cells and lysed bacterial cells, was removed. This wash step was repeated until there was no further removal of bacteria. Then, the coal pellet was suspended in 50mL of 0.1M NaOH, placed in a boiling water bath for 10 to 15 minutes, and subjected to differential centrifugation. This alkali washing procedure was repeated until a clear supernatant was achieved. At this point, the coal sample was deemed to be free of biomass and dried to a constant weight at 105°C.

The microbiological blank samples were subjected to an abbreviated washing procedure since they were free of biomass. The coal was harvested by centrifugation at 10,000 x gravity for 10 minutes. The pellet was suspended in 50mL of distilled water, centrifuged at 1000 x gravity for 2 minutes, and the supernatant removed; this process was repeated until a clear supernatant was produced. The coal was then suspended in 50mL of distilled water, placed in a boiling water bath for 10 to 15 minutes, and centrifuged at 1000 x gravity for 2 minutes. This process was repeated until a clear supernatant was produced. The coal was then dried at 105°C to a constant weight.

A second set of samples was prepared in which a shorter reaction time (1 hour) was used. Certain experiments from the first set of samples were repeated for measurement of repeatability. Table 1 is a listing and status of samples in the first and second set of experiments.

A third set of samples was prepared by adding the oxidant to the coal over a period of 30 minutes. The catalyst was then added over a period of 5 minutes. The following oxidations were done.

Oxidant	Catalyst	(Hours)	Temperature
Hydrogen peroxide (H_2O_2)	-	72	RT
Hydrogen peroxide (H_2O_2)	-	24	50°C
Hydrogen peroxide (H_2O_2)	Acetic Acid	24	RT
Hydrogen peroxide (H_2O_2)	Acetic Acid	24	50°C
Sodium perborate ($NaBO_3 \cdot 4H_2O$)	-	24	RT
Potassium peroxydisulfate ($K_2S_2O_8$)	-	24	RT

RESULTS AND DISCUSSION

In initial experiments, a number of blanks were run to determine the variation of results in the analysis of organic sulfur due to the oxidation process and the microbiological process. Thus, there were twelve untreated coal samples (Table 1: 57-66) that were analyzed for total sulfur and sulfur-by-type and five that were analyzed for CHN and BTU to determine the variance in the analysis of the coal samples, ten samples treated with sodium periodate (Table 1: 31-40) that were analyzed to determine the variance in the chemical treatment, and five microbiologically treated samples (Table 1: 48-52) that were analyzed to determine the variance in the microbiological treatment. Table 2 is a tabulation of the analysis data of the first two sets of data and Table 3 is a summary of the data in Table 2. From Table 3, the following data can be used to see the variation in the analysis of data and variation due to the experiment:

Type of Variation	Sample	Analysis	Samples	Mean	Deviation
Analytical	Coal itself	Total Sulfur (%)	10	3.53	0.113
Analytical	Coal itself	Elemental carbon (%)	10	65.34	0.392
Analytical	Coal itself	Btu (Btu/lb)	10	11806.4	63.46
Experimental/analytical	$NaIO_4$ treated	Total sulfur (%)	10	3.53	0.061
Experimental/analytical	Microbial treated	Total sulfur (%)	4	3.00	0.191
Experimental/analytical	Microbial treated	Elemental carbon (%)	4	65.48	1.288
Experimental/analytical	Microbial treated	Btu (Btu/lb)	4	11838.3	168.34

It can be seen that the variation due to analytical is small in the analysis of total sulfur, elemental carbon, and Btu. However, the experimental variation in the microbial treated sample is somewhat higher, particularly in elemental carbon. Once the variances in the experimental procedures had been established, reductions in sulfur content in the coal due to chemical oxidation and/or microbiological treatment could be evaluated.

The chemical oxidation only effects were determined by comparing the following samples:

For hydrogen peroxide,
- Samples 5-7 versus 57-66
- Samples 10-11 versus 67-68
- Samples 1-2 versus 67-68

For potassium permanganate,
- Samples 18-20 versus 57-66
- Samples 23-24 versus 67-68
- Samples 14-15 versus 67-68

For sodium periodate,
Samples 31-40 versus 57-66
Samples 43-44 versus 67-68
Samples 27-28 versus 67-68

The microbiological only effects were determined by comparing the following samples:

Samples 48-52 versus 57-66 and 47
Samples 54 versus 67-68 and 53

The effectiveness of the combination treatments were determined by comparing the combination treatments with the untreated coal in the following samples:

For H_2O_2/IGTS8 treatment,
Samples 9 versus 57-66 and 8
Samples 13 versus 67-68 and 12
Samples 3-4 versus 67-68

For $KMnO_4$/IGTS8 treatment,
Samples 18-20 versus 57-66 and 21
Samples 26 versus 67-68 and 25
Samples 16-17 versus 67-68

For $NaIO_4$/IGTS8 treatment,
Samples 31-40 versus 57-66 and 41
Samples 46 versus 67-68 and 45
Samples 29-30 versus 67-68

A summary of the results from sample sets 1 and 2 of the oxidation/microbial treatment is shown in Table 3. From these data, it can be seen that the hydrogen peroxide and the hydrogen peroxide/microbial treatments were effective in removing sulfur from coal. With potassium permanganate, no assessment could be made due to contamination of the product by manganese dioxide. Sodium periodate appeared to have no effect on the product.

With oxidation and microbial treatment, several processes can occur. Oxidation can lead to degradation of the molecules in the coal, oxidation of alcohol or phenol groups, addition of oxygen to the sulfur atoms (the desired reaction), etc. If all sulfur atoms in the molecules in the coal added one oxygen, the elemental carbon composition would drop from 65.34 to 64.41% or about 0.93%, assuming no other processes had taken place; with the addition of two oxygens to sulfur, the drop would be from 65.34 to 63.50% or about 1.84%. Of course, changes in elemental carbon composition can be due to other factors, as was the case with potassium permanganate oxidation of coal forming manganese dioxide.

Under the elemental carbon column in Table 3, it can be seen that the percent carbon with the treatments, H_2O_2(3 hr), H_2O_2(3 hr)/microbial, and

$NaIO_4$(3 hr), is higher whereas the percent carbon with the treatments, $NaIO_4$(3 hr)/microbial and microbial, is approximately the same as the standard, the coal itself. (The potassium permanganate values are not considered here.) Thus, there is no direct evidence that any of these treatments has led to the addition of oxygen to the sulfur atoms in the coal.

From Table 3, it can be seen that there was a drastic reduction in total sulfur with potassium permanganate treatment. However, there are also similar reductions in elemental carbon and BTU. It appears that manganese dioxide, formed during reaction of coal with potassium permanganate, coprecipitates with the coal and acts as a diluent. Thus, it is difficult to determine whether the sulfur is reduced because there is no obvious procedure for compensating for the manganese dioxide. There are similar problems in analyzing the data in the dual treatment, potassium permanganate and microbial treatment. Since the separation of the manganese dioxide from coal would be difficult, it would appear that potassium permanganate would not be a good oxidant for oxidizing sulfur.

Since the major contribution of total sulfur in IBC-107 coal is the organic sulfur which is derived from subtraction of sulfide, sulfate, and pyritic sulfur from total sulfur, one can consider just the total sulfur initially. Table 4 is a tabulation of percent reductions of total sulfur in the IBC-107 coal with chemical, microbial, and combination chemical/microbial treatments.

Table 4. Percent Reduction in Total Sulfur in IBC-107 Coal

Chemical Treatment	Without Microbial Treatment		With Microbial Treatment	
	1st Set	2nd Set	1st Set	2nd Set
Hydrogen periodate, H_2O_2, 1 hour	-	5.5(2)	-	15.8(2)
Hydrogen periodate, H_2O_2, 3 hours	5.7(3)	5.0(2)	11.9(1)	13.9(1)
Sodium periodate, $NaIO_4$, 1 hour	-	6.6(2)	-	15.8(2)
Sodium periodate, $NaIO_4$, 3 hours	0.0(10)	9.7(2)	12.8(1)	20.2(1)
None	-	-	15.0(4)	13.9(1)

NOTE: Number in parentheses () is number of samples.

With hydrogen peroxide, there is approximately a 5% reduction in total sulfur with chemical treatment and a 14% reduction with the combination treatment. There appears to be no difference between one or three hour treatments with hydrogen peroxide. In the case of sodium periodate, the situation is more complicated. In the first set of experiments, the sodium periodate appears to have no influence on the efficiency of the removal of sulfur from the coal. With the second set of samples, there is a distinct reduction in total sulfur with chemical treatment alone and a further decrease due to microbial treatment. Also, the 3 hour treatment with sodium periodate appears to be 50% more effective than the 1 hour treatment. Thus, possibly longer reaction times with sodium periodate might lead to further decreases in total sulfur. The highest reduction in total sulfur (20.2%) was achieved with the combination treatment, 3 hour sodium periodate/microbial.

Data on the third set of samples is presented in Tables 5 and 6. It can be seen that there is a decrease in total sulfur with each chemical treatment and a further decrease with microbial treatment. However, the microbial treatment is accompanied with decreases in elemental carbon. This indicates that some of the microbial material is being left behind, possibly trapped in or adsorbed on the surface of the coal. For example, in sample 74, which had only microbial treatment, the total sulfur is 2.30% compared to 3.81% for untreated coal (samples 88-91). This appears to be a substantial decrease in the total sulfur in coal; however, the CHN analysis is carbon 62.60%, hydrogen 5.95%, and hydrogen 4.04% for the treated sample (74) and carbon 66.53%, hydrogen 4.62%, and nitrogen 1.04% for the untreated coal (samples 88-91). This indicates contamination, probably with bacteria which typically have a composition of carbon 50%, hydrogen 6%, and nitrogen 12%. Contamination with bacteria would decrease the carbon content and increase the nitrogen content, which is precisely what happens in a number of the microbial treatment samples. In fact, one can obtain a reasonably good fit by assuming bacterial contamination and calculating the percent contamination.

An easier technique for looking at the samples that are contaminated is to plot sulfur content versus the carbon content and then extrapolate back to the original content of carbon in coal. By this technique, the sulfur content in the microbial treatment samples can be determined. This is done in Figure 1. The results are tabulated in Table 7.

Table 7. Percent Reduction of Total Sulfur in IBC-107 Coal by Chemical/Microbial Treatment

	Treatment			Reduction in Total Sulfur (%)		
Oxidant	Time(hr)	Temperature	Catalyst	Chemical	Microbial	Chemical/ Microbial
H_2O_2	72	RT	-	15.8	10.7	24.8
H_2O_2	24	50°C	-	12.8	13.9	24.9
H_2O_2	72	RT	HOAc	18.1	9.6	26.0
H_2O_2	24	50°C	HOAc	12.9	16.2	27.0
$NaBO_3$	24	RT	-	10.7	12.9	22.2
$K_2S_2O_8$	24	RT	-	6.2	22.4	27.2
-	-	-	-	-	18.4	-

It can be seen that the reduction in total sulfur in IBC-107 coal by chemical oxidation was 6.2 to 18.1% and of oxidized coal by microbial desulfurization was 9.6 to 22.4%. The overall reduction in sulfur by chemical and microbial desulfurization was 22.2 to 27.2%.

CONCLUSIONS AND RECOMMENDATIONS

In the combination treatment, chemical oxidation and microbial desulfurization, for removal of sulfur from coal, it was found that the total sulfur in IBC-107 coal was reduced 6.2 to 18.1% with chemical oxidation depending upon the oxidant and conditions used. Removal of sulfur in the oxidized coal by IGTS8, a culture developed at IGT, resulted in a further reduction of 9.6 to 22.4%. The overall reduction of sulfur by chemical and microbial desulfurization was 22.2 to 27.2%.

Problems in the combined oxidation-microbial process that require study include 1) the enhancement of the conversion of the alkyl and aryl sulfur containing compounds in coal to sulfoxides and sulfones, 2) the development of an analytical technique for measuring the oxidation, 3) the development of microbial cultures capable of specifically attacking the carbon-sulfur bond in sulfoxides and sulfones, and 4) the development of a procedure for removing bacteria from the coal.

Although the reductions in total sulfur were modest in this initial study, it appears that the concept of a combined approach, chemical and microbiological, is attractive and deserves further study.

Table 1. Status of Samples in First and Second Sets of Experiments

Sample Number	Chemical Treatment Oxidant	Chemical Treatment Time (hr)	Microbial Treatment	Status Set 1 Chemical	Status Set 1 Microbial	Status Set 1 Analysis	Status Set 2 Chemical	Status Set 2 Microbial	Status Set 2 Analysis
1	H_2O_2	1	-	-	-	-	X	-	X
2	H_2O_2	1	-	-	-	-	X	-	X
3	H_2O_2	1	IGTS8	-	-	-	X	X	X
4	H_2O_2	1	IGTS8	-	-	-	X	X	X
5	H_2O_2	3	-	X	-	X	-	-	X
6	H_2O_2	3	-	X	-	X	-	-	X
7	H_2O_2	3	-	X	-	X	-	-	-
8	H_2O_2	3	Media only	X	X	X	-	-	-
9	H_2O_2	3	IGTS8	X	X	X	-	-	-
10	H_2O_2	3	-	-	-	-	X	-	X
11	H_2O_2	3	-	-	-	-	X	-	X
12	H_2O_2	3	Media only	-	-	-	X	X	X
13	H_2O_2	3	IGTS8	-	-		X	X	X
14	$KMnO_4$	1	-	-	-	-	X	-	X
15	$KMnO_4$	1	-	-	-	-	X	-	X
16	$KMnO_4$	1	IGTS8	-	-	-	X	X	X
17	$KMnO_4$	1	IGTS8	-	-	-	X	X	X
18	$KMnO_4$	3	-	X	-	X	-	-	X
19	$KMnO_4$	3	-	X	-	X	-	-	X
20	$KMnO_4$	3	-	X	-	X	-	-	-
21	$KMnO_4$	3	Media only	X	X	X	-	-	-
22	$KMnO_4$	3	IGTS8	X	X	X	-	-	-
23	$KMnO_4$	3	-	-	-	-	X	-	X
24	$KMnO_4$	3	-	-	-	-	X	-	X
25	$KMnO_4$	3	Media only	-	-	-	X	X	X
26	$KMnO_4$	3	IGTS8	-	-	-	X	X	X
27	$NaIO_4$	1	-	-	-	-	X	-	X
28	$NaIO_4$	1	-	-	-	-	X	-	X
29	$NaIO_4$	1	IGTS8	-	-	-	X	X	X
30	$NaIO_4$	1	IGTS8	-	-	-	X	X	X
31	$NaIO_4$	3	-	X	-	X	-	-	X
32	$NaIO_4$	3	-	X	-	X	-	-	X
33	$NaIO_4$	3	-	X	-	X	-	-	-
34	$NaIO_4$	3	-	X	-	X	-	-	-
35	$NaIO_4$	3	-	X	-	X	-	-	-
36	$NaIO_4$	3	-	X	-	X	-	-	-
37	$NaIO_4$	3	-	X	-	X	-	-	-
38	$NaIO_4$	3	-	X	-	X	-	-	-
39	$NaIO_4$	3	-	X	-	X	-	-	-
40	$NaIO_4$	3	-	X	-	X	-	-	-
41	$NaIO_4$	3	Media only	X	X	X	-	-	-
42	$NaIO_4$	3	IGTS8	X	X	X	-	-	-
43	$NaIO_4$	3	-	-	-	-	X	-	X
44	$NaIO_4$	3	-	-	-	-	X	-	X
45	$NaIO_4$	3	Media only	-	-	-	X	X	X
46	$NaIO_4$	3	IGTS8	-	-	-	X	X	X
47	None	-	Media only	-	X	X	-	-	-
48	None	-	IGTS8	-	X	X	-	-	-
49	None	-	IGTS8	-	X	X	-	-	-
50	None	-	IGTS8	-	X	X	-	-	-
51	None	-	IGTS8	-	X	X	-	-	-
52	None	-	IGTS8	-	L	-	-	-	-
53	None	-	Media only	-	-	-	-	X	X
54	None	-	IGTS8	-	-	-	-	X	X
55	Solvent only	-	Media only	X	-	X	-	-	-
56	Solvent only	-	IGTS8	X	-	X	-	-	-
57	Coal only	-	-	-	-	X	-	-	X
58	Coal only	-	-	-	-	X	-	-	X
59	Coal only	-	-	-	-	X	-	-	-
60	Coal only	-	-	-	-	X	-	-	-
61	Coal only	-	-	-	-	X	-	-	-
62	Coal only	-	-	-	-	X	-	-	-
63	Coal only	-	-	-	-	X	-	-	-
64	Coal only	-	-	-	-	X	-	-	-
65	Coal only	-	-	-	-	X	-	-	-
66	Coal only	-	-	-	-	X	-	-	-
67	Coal only	-	-	-	-	-	-	-	X
68	Coal only	-	-	-	-	-	-	-	X

X Work completed
L Part of sample lost during workup

Table 2. Analytical Data on Oxidation and Microbiological Treatments for Removing Sulfur from Coal; Sample Sets 1 and 2

Sample Number	Oxidant	Time (hr.)	Microbial Treatment	Total Sulfur	Sulfide	Sulfate	Pyritic Sulfur	Organic Sulfur	C	H	N	Ratio Total S/C	Ratio Org S/C	BTU
1	H_2O_2	1		3.59										
2	H_2O_2	1		3.59										
3	H_2O_2	1	IGTS8	3.32										
4	H_2O_2	1	IGTS8	3.10										
5	H_2O_2	3		3.25	<0.012	0.16	0.33	2.76	67.12	4.90	1.26	0.048	0.041	12060.0
6	H_2O_2	3		3.42	0.010	0.13	0.31	2.97	66.74	4.79	1.22	0.051	0.045	11954.3
7	H_2O_2	3		3.35	<0.020	0.15	0.33	2.87	67.12	4.87	1.30	0.050	0.043	12098.9
8	H_2O_2	3	Media only	1.92	0.012	0.05	0.23	1.63	48.17	3.82	1.45	0.040	0.034	7563.2
9	H_2O_2	3	IGTS8	3.11	0.018	<0.06	0.25	2.84	66.94	5.00	1.74	0.046	0.042	12064.0
10	H_2O_2	3		3.70										
11	H_2O_2	3		3.69										
12	H_2O_2	3	Media only	3.30										
13	H_2O_2	3	IGTS8	3.28										
14	$KMnO_4$	1												
15	$KMnO_4$	1												
16	$KMnO_4$	1	IGTS8											
17	$KMnO_4$	1	IGTS8											
18	$KMnO_4$	3		1.96	0.028	0.41	0.21	1.31	38.92	3.19	0.70	0.050	0.034	6835.5
19	$KMnO_4$	3		1.76	0.030	0.43	0.30	1.00	39.30	3.23	0.77	0.048	0.025	6805.6
20	$KMnO_4$	3		1.98	0.029	0.45	0.47	1.03	39.17	3.20	0.71	0.051	0.026	6835.9
21	$KMnO_4$	3	Media only	0.92	0.015	0.20	0.43	0.28	24.41	2.23	1.06	0.038	0.012	4212.2
22	$KMnO_4$	3	IGTS8	2.24	0.011	0.05	0.18	2.00	46.41	3.75	1.09	0.048	0.043	8296.2
23	$KMnO_4$	3												
24	$KMnO_4$	3												
25	$KMnO_4$	3	Media only											
26	$KMnO_4$	3	IGTS8											
27	$NaIO_4$	1		3.57										
28	$NaIO_4$	1		3.55										
29	$NaIO_4$	1	IGTS8	3.13										
30	$NaIO_4$	1	IGTS8	3.30										
31	$NaIO_4$	3		3.54	<0.016	0.14	0.43	2.97	66.36	4.75	1.18	0.053	0.045	11867.4
32	$NaIO_4$	3		3.54	<0.011	0.13	0.42	2.99	66.02	4.75	1.24	0.054	0.045	11910.8
33	$NaIO_4$	3		3.55										
34	$NaIO_4$	3		3.58										
35	$NaIO_4$	3		3.55										
36	$NaIO_4$	3		3.53										
37	$NaIO_4$	3		3.58										
38	$NaIO_4$	3		3.40										
39	$NaIO_4$	3		3.58										
40	$NaIO_4$	3		3.44										
41	$NaIO_4$	3	Media only	2.15	0.012	0.08	0.28	1.78	49.57	3.78	1.30	0.043	0.036	8986.5
42	$NaIO_4$	3	IGTS8	3.08					65.77	4.95	1.69	0.047		11806.6
43	$NaIO_4$	3		3.59										
44	$NaIO_4$	3		3.61										
45	$NaIO_4$	3	Media only	3.15										
46	$NaIO_4$	3	IGTS8	3.04										
47	None		Media only	1.62	0.018	0.05	0.15	1.40	36.92	3.31	1.62	0.044	0.038	6670.3
48	None		IGTS8	2.82	0.020	0.08	0.50	2.22	64.09	5.29	3.02	0.044	0.035	11727.4
49	None		IGTS8	3.02	0.014	0.05	0.19	2.76	66.06	5.20	1.98	0.046	0.042	11788.7
50	None		IGTS8	3.26	<0.010	0.07	0.37	2.82	66.97	4.95	1.64	0.048	0.042	12087.9
51	None		IGTS8	2.91	<0.019	<0.07	0.41	2.50	64.78	5.23	2.54	0.045	0.039	11749.1
52	None		IGTS8	a										
53	None		Media only	3.00										
54	None		IGTS8	3.28										
55	Solvent only		Media only	1.74	0.010	0.05	0.38	1.30	39.43	3.31	1.25	0.044	0.033	6956.1
56	Solvent only		IGTS8	2.85					65.52	5.27	2.21	0.044		11794.9
57	Coal only			3.42					65.71	4.90	1.15	0.052		11840.6
58	Coal only			3.41					65.03	4.90	1.16	0.053		11788.2
59	Coal only			3.34					65.35	4.90	1.16	0.051		11806.9
60	Coal only			3.68					64.57	4.88	1.10	0.057		11686.1
61	Coal only			3.56					65.87	4.93	1.13	0.054		11918.5
62	Coal only			3.59	0.017	0.28	0.37	2.92	65.29	4.89	1.12	0.055	0.045	11803.0
63	Coal only			3.65	0.022	0.34	0.53	2.76	65.38	4.85	1.18	0.056	0.042	11815.2
64	Coal only			3.45	0.014	0.34	0.11	2.99	65.79	4.81	1.10	0.052	0.045	11871.5
65	Coal only			3.56	0.013	0.28	0.24	3.03	65.34	4.90	1.16	0.054	0.046	11778.8
66	Coal only			3.59	0.012	0.29	0.49	2.80	65.08	4.97	1.19	0.055	0.043	11755.3
67	Coal only			3.77										
68	Coal only			3.85										

[a]Part of sample lost during workup.

Table 3. Summary of Data on Oxidation/Microbial Treatment of Coal in Sample Sets 1 and 2

			Total Sulfur			Organic Sulfur			Elemental Carbon			Btu		
Sample	Sample Set	Sample Numbers	Mean	Standard Deviation	Level of Significance	Mean	Standard Deviation	Level of Significance	Mean	Standard Deviation	Levelof Significance	Mean	Standard Deviation	Level of Significance
Coal itself	1	57-66	3.53(10)	0.113	-	2.90(5)	0.117	-	65.34(10)	0.392	-	11806.4(10)	63.46	-
Coal itself	2	67-68	3.81(2)	-	-	-	-	-	-	-	-	-	-	-
H_2O_2 (1 hr)	2	1-2	3.59(2)	-	-	-	-	-	-	-	-	-	-	-
H_2O_2 (1 hr)/Microbial	2	3-4	3.21(2)	-	-	-	-	-	-	-	-	-	-	-
H_2O_2 (3 hr)	1	5-7	3.34(3)	0.085	0.025	2.86(3)	0.110	-	66.99(3)	0.219	0.001	12037.7(3)	74.83	0.01
H_2O_2 (3 hr)/Microbial	1	9	3.11(1)	-	NT	2.78(1)	-	NT	66.94(1)	-	NT	12064.0(1)	-	NT
H_2O_2 (3 hr)	2	10-11	3.69(2)	-	NT	-	-	-	-	-	-	-	-	-
H_2O_2 (3 hr)/Microbial	2	13	3.28(1)	-	NT	-	-	-	-	-	-	-	-	-
$KMnO_4$ (1 hr)	2	14-15	W	-	-	-	-	-	-	-	-	-	-	-
$KMnO_4$ (1 hr)/Microbial	2	16-17	W	-	-	-	-	-	-	-	-	-	-	-
$KMnO_4$ (3 hr)	1	18-20	1.90(3)	0.122	NT	1.11(3)	0.171	NT	39.13(3)	0.193	NT	6825.7(3)	17.38	NT
$KMnO_4$ (3 hr)/Microbial	1	22	2.24(1)	-	NT	2.00(1)	-	NT	46.41(1)	-	NT	8296.2(1)	-	NT
$KMnO_4$ (3 hr)	2	23-24	W	-	-	-	-	-	-	-	-	-	-	-
$KMnO_4$ (3 hr)/Microbial	2	26	W	-	-	-	-	-	-	-	-	-	-	-
$NaIO_4$ (1 hr)	2	27-28	3.56(2)	-	NT	-	-	-	-	-	-	-	-	-
$NaIO_4$ (1 hr)/Microbial	2	29-30	3.21(2)	-	NT	-	-	-	-	-	-	-	-	-
$NaIO_4$ (3 hr)	1	31-40	3.53(10)	0.061	-	2.98(2)	-	-	66.19(2)	[illegible].240	NT	11889.1(2)	30.69	NT
$NaIO_4$ (3 hr)/Microbial	1	42	3.08(1)	-	NT		-	-	65.77(1)	-	-	11806.6(1)	-	-
$NaIO_4$ (3 hr)	2	43-44	3.44(2)	-	NT	-	-	-	-	-	-	-	-	-
$NaIO_4$ (3 hr)/Microbial	2	46	3.04(1)	-	NT	-	-	-	-	-	-	-	-	-
Microbial	1	48-51	3.00(4)	0.191	0.005	2.56(4)	0.274	0.05	65.48(4)	1.288	-	11838.3(4)	168.34	-
Microbial	2	54	3.28(1)	-	-	-	-	-	-	-	-	-	-	-
H_2O_2 (3 hr)/Media only	1	9	1.92(1)	-	NT	1.63(1)	-	NT	48.17(1)	-	NT	7563.2(1)	-	NT
$KMnO_4$ (3 hr)/Media only	1	22	0.92(1)	-	NT	0.28(1)	-	NT	24.41(1)	-	NT	4212.2(1)	-	NT
$NaIO_4$ (3 hr)/Media only	1	41	2.15(1)	-	NT	1.78(1)	-	NT	49.57(1)	-	NT	8986.5(1)	-	NT
Media only	1	47	1.62(1)	-	NT	1.40(1)	-	NT	36.92(1)	-	NT	6670.3(1)	-	NT

NT Significance of results not tested due to insufficient number of samples or due to samples associated with manganese dioxide contamination.

W Withdrawn; samples not analyzed because they are associated with manganese dioxide contamination.

Table 5. Analytical Data on the Oxidation and Microbiological Treatments for Removing Sulfur from Coal

Sample Number	Sample Set	Chemical Treatment: Oxidant	Catalyst	Reaction Time (hr)	Temperature (°C)	Microbial Treatment	Total Sulfur (%): Untreated Coal	Treated Coal	Per Cent Reduction	Sulfur by Type: Sulfide	Sulfate	Pyritic Sulfur	Organic Sulfur	Elemental Analysis: C	H	N	Ratio S/C	Heating Value Btu/lb
1	1	H_2O_2	–	3	RT	–	3.53	3.25	7.8	<0.012	0.16	0.33	2.76	67.12	4.90	1.26	0.048	12060.0
2	1	H_2O_2	–	3	RT	–	3.53	3.42	2.9	0.010	0.13	0.31	2.97	66.74	4.79	1.22	0.051	11954.3
3	1	H_2O_2	–	3	RT	–	3.53	3.35	5.0	<0.020	0.15	0.33	2.87	67.12	4.87	1.30	0.050	12098.9
4*	2	H_2O_2	–	3	RT	–	3.81	3.46	9.2	–	–	–	–	–	–	–	–	–
5*	2	H_2O_2	–	3	RT	–	3.81	3.38	11.3	–	–	–	–	–	–	–	–	–
6	2	H_2O_2	–	3	RT	–	3.81	3.30	13.4	–	–	–	–	–	–	–	–	–
7	2	H_2O_2	–	3	RT	–	3.81	3.32	12.9	–	–	–	–	–	–	–	–	–
8	1	H_2O_2	–	3	RT	medium only	3.53	1.92	45.5	0.012	0.05	0.23	1.63	48.17	3.82	1.45	0.040	7563.2
9	2	H_2O_2	–	3	RT	medium only	3.81	3.10	18.6	–	–	–	–	–	–	–	–	–
10	1	H_2O_2	–	3	RT	IGTS8	3.53	3.11	11.8	0.018	<0.06	0.25	2.84	66.94	5.00	1.74	0.046	12064.0
11	2	H_2O_2	–	3	RT	IGTS8	3.81	3.28	13.9	–	–	–	–	–	–	–	–	–
12	2	H_2O_2	–	1	RT	–	3.81	3.59	5.8	–	–	–	–	–	–	–	–	–
13	2	H_2O_2	–	1	RT	–	3.81	3.59	5.8	–	–	–	–	–	–	–	–	–
14	2	H_2O_2	–	1	RT	IGTS8	3.81	3.70	2.9	–	–	–	–	–	–	–	–	–
15	2	H_2O_2	–	1	RT	IGTS8	3.81	3.69	3.2	–	–	–	–	–	–	–	–	–
16	3	H_2O_2	–	72	RT	–	3.89	3.25	16.5	–	–	–	–	66.62	4.64	1.21	–	11891.9
17	3	H_2O_2	–	72	RT	–	3.89	3.30	15.2	–	–	–	–	66.87	4.64	1.28	–	–
18	3	H_2O_2	–	72	RT	IGTS8	3.89	2.48	36.2	–	–	–	–	64.68	5.46	2.12	–	11976.5
19	3	H_2O_2	–	72	RT	IGTS8	3.89	2.70	30.6	–	–	–	–	65.04	5.09	2.03	–	–
20	3	H_2O_2	–	24	50	–	3.89	3.36	13.6	–	–	–	–	67.14	4.74	1.32	–	11820.4
21	3	H_2O_2	–	24	50	–	3.89	3.42	12.1	–	–	–	–	67.11	4.67	1.16	–	–
22	3	H_2O_2	–	24	50	IGTS8	3.89	2.89	25.7	–	–	–	–	65.85	5.08	1.67	–	11828.9
23	3	H_2O_2	–	24	50	IGTS8	3.89	2.73	29.8	–	–	–	–	66.14	5.16	1.54	–	–
24	3	H_2O_2	HOAc	72	RT	–	3.89	3.17	18.5	–	–	–	–	66.78	4.71	1.25	–	11908.6
25	3	H_2O_2	HOAc	72	RT	–	3.89	3.20	17.7	–	–	–	–	67.08	4.67	1.16	–	–
26	3	H_2O_2	HOAc	72	RT	IGTS8	3.89	2.90	25.5	–	–	–	–	66.59	4.86	1.46	–	11885.6
27	3	H_2O_2	HOAc	72	RT	IGTS8	3.89	2.63	32.4	–	–	–	–	65.20	5.33	2.39	–	–
28	3	H_2O_2	HOAc	24	50	–	3.89	3.38	13.1	–	–	–	–	66.66	4.61	1.20	–	11799.0
29	3	H_2O_2	HOAc	24	50	–	3.89	3.40	12.6	–	–	–	–	66.54	4.56	1.25	–	–
30	3	H_2O_2	HOAc	24	50	IGTS8	3.89	2.40	38.3	–	–	–	–	52.93	4.40	1.95	–	9464.8
31	3	H_2O_2	HOAc	24	50	IGTS8	3.89	2.76	29.0	–	–	–	–	64.14	4.99	2.42	–	–

32	1	$KMnO_4$	-	3	RT	-	3.53	1.96	44.4	0.028	0.41	0.21	1.31	38.92	3.19	0.70	0.050	6835.5
33	1	$KMnO_4$	-	3	RT	-	3.53	1.76	50.1	0.030	0.43	0.30	1.00	39.30	3.23	0.77	0.048	6805.6
34	1	$KMnO_4$	-	3	RT	-	3.53	1.98	43.8	0.029	0.45	0.47	1.03	39.17	3.20	0.71	0.051	6835.9
35	1	$KMnO_4$	-	3	RT	medium only	3.53	0.92	73.9	0.015	0.20	0.43	0.28	24.41	2.23	1.06	0.038	4212.2
36	1	$KMnO_4$	-	3	RT	IGTS8	3.53	2.24	36.5	0.011	0.05	0.18	2.00	46.41	3.75	1.09	0.048	8296.2
37	1	$NaIO_4$	-	3	RT	-	3.53	3.54	0.0	<0.016	0.14	0.43	2.97	66.36	4.75	1.18	0.053	11867.4
38	1	$NaIO_4$	-	3	RT	-	3.53	3.54	0.0	<0.011	0.13	0.42	2.99	66.02	4.75	1.24	0.054	11910.8
39	1	$NaIO_4$	-	3	RT	-	3.53	3.55	0.0	-	-	-	-	-	-	-	-	-
40	1	$NaIO_4$	-	3	RT	-	3.53	3.58	0.0	-	-	-	-	-	-	-	-	-
41	1	$NaIO_4$	-	3	RT	-	3.53	3.55	0.0	-	-	-	-	-	-	-	-	-
42	1	$NaIO_4$	-	3	RT	-	3.53	3.53	0.0	-	-	-	-	-	-	-	-	-
43	1	$NaIO_4$	-	3	RT	-	3.53	3.58	0.0	-	-	-	-	-	-	-	-	-
44	1	$NaIO_4$	-	3	RT	-	3.53	3.40	3.5	-	-	-	-	-	-	-	-	-
45	1	$NaIO_4$	-	3	RT	-	3.53	3.58	0.0	-	-	-	-	-	-	-	-	-
46	1	$NaIO_4$	-	3	RT	-	3.53	3.44	2.4	-	-	-	-	-	-	-	-	-
47	1	$NaIO_4$	-	3	RT	medium only	3.53	2.15	39.0	0.012	0.08	0.28	1.78	49.57	3.78	1.30	0.043	8986.5
48	1	$NaIO_4$	-	3	RT	IGTS8	3.53	3.08	12.6	-	-	-	-	65.77	4.95	1.69	0.047	11806.6
49*	2	$NaIO_4$	-	3	RT	-	3.81	3.59	5.8	-	-	-	-	-	-	-	-	-
50*	2	$NaIO_4$	-	3	RT	-	3.81	3.61	5.2	-	-	-	-	-	-	-	-	-
51	2	$NaIO_4$	-	3	RT	-	3.81	3.15	17.3	-	-	-	-	-	-	-	-	-
52	2	$NaIO_4$	-	3	RT	-	3.81	3.13	17.8	-	-	-	-	-	-	-	-	-
53	2	$NaIO_4$	-	3	RT	medium only	3.81	3.30	13.4	-	-	-	-	-	-	-	-	-
54	2	$NaIO_4$	-	3	RT	IGTS8	3.81	3.04	20.2	-	-	-	-	-	-	-	-	-
55	2	$NaIO_4$	-	1	RT	-	3.81	3.57	6.3	-	-	-	-	-	-	-	-	-
56	2	$NaIO_4$	-	1	RT	-	3.81	3.55	6.8	-	-	-	-	-	-	-	-	-
57	2	$NaIO_4$	-	1	RT	IGTS8	3.81	3.46	9.2	-	-	-	-	-	-	-	-	-
58	2	$NaIO_4$	-	1	RT	IGTS8	3.81	3.43	10.0	-	-	-	-	-	-	-	-	-
59	3	$NaBO_3$	-	24	RT	-	3.89	3.49	10.3	-	-	-	-	65.30	4.53	1.15	-	11629.4
60	3	$NaBO_3$	-	24	RT	-	3.89	3.42	12.1	-	-	-	-	65.39	4.58	1.18	-	-
61	3	$NaBO_3$	-	24	RT	IGTS8	3.89	2.95	24.2	-	-	-	-	66.04	5.04	1.70	-	11920.1
62	3	$NaBO_3$	-	24	RT	IGTS8	3.89	2.65	31.9	-	-	-	-	64.10	5.27	2.89	-	-
63	3	$K_2S_2O_8$	-	24	RT	-	3.89	3.69	5.1	-	-	-	-	67.13	4.59	1.18	-	11944.4
64	3	$K_2S_2O_8$	-	24	RT	-	3.89	3.61	7.2	-	-	-	-	67.26	4.61	1.14	-	-
65	3	$K_2S_2O_8$	-	24	RT	IGTS8	3.89	2.52	33.9	-	-	-	-	864.92	5.56	2.62	-	11888.5
66	3	$K_2S_2O_8$	-	24	RT	IGTS8	3.89	3.01	22.6	-	-	-	-	67.34	4.88	1.40	-	-
67	1	-	-	-	-	medium only	3.53	1.62	54.0	0.018	0.05	0.15	1.40	36.92	3.31	1.62	0.044	6670.3
68	2	-	-	-	-	medium only	3.81	3.00	21.6	-	-	-	-	-	-	-	-	-

69	1	–	–	–	–	IGTS8	3.53	2.82	20.0	0.020	0.08	0.50	2.22	64.09	5.29	3.02	0.044	11727.4
70	1	–	–	–	–	IGTS8	3.53	3.02	14.3	0.014	0.05	0.19	2.76	66.06	5.20	1.98	0.046	11788.7
71	1	–	–	–	–	IGTS8	3.53	3.26	7.5	<0.010	0.07	0.37	2.82	66.97	4.95	1.64	0.048	12087.9
72	1	–	–	–	–	IGTS8	3.53	2.91	17.4	<0.019	<0.07	0.41	2.50	64.78	5.23	2.54	0.045	11749.1
73	2	–	–	–	–	IGTS8	3.81	3.28	13.9	–	–	–	–	–	–	–	–	–
74	3	–	–	–	–	IGTS8	3.89	2.30	40.9	–	–	–	–	62.60	5.95	4.04	–	–
75	3	–	–	–	–	IGTS8	3.89	3.16	18.8	–	–	–	–	66.40	5.01	1.82	–	–
76	1	–	–	–	–	–	–	3.59	–	0.017	0.28	0.37	2.92	65.29	4.89	1.12	0.055	11803.0
77	1	–	–	–	–	–	–	3.65	–	0.022	0.34	0.53	2.76	65.38	4.85	1.18	0.056	11815.2
78	1	–	–	–	–	–	–	3.45	–	0.014	0.34	0.11	2.99	65.79	4.81	1.10	0.052	11871.5
79	1	–	–	–	–	–	–	3.56	–	0.013	0.28	0.24	3.03	65.34	4.90	1.16	0.054	11778.8
80	1	–	–	–	–	–	–	3.59	–	0.012	0.29	0.49	2.80	65.08	4.97	1.19	0.055	11755.3
81	1	–	–	–	–	–	–	3.42	–	–	–	–	–	65.71	4.90	1.15	0.052	11840.6
82	1	–	–	–	–	–	–	3.41	–	–	–	–	–	65.03	4.90	1.16	0.053	11788.2
83	1	–	–	–	–	–	–	3.34	–	–	–	–	–	65.35	4.90	1.16	0.051	11806.9
84	1	–	–	–	–	–	–	3.68	–	–	–	–	–	64.57	4.88	1.10	0.057	11686.1
85	1	–	–	–	–	–	–	3.56	–	–	–	–	–	65.87	4.93	1.13	0.054	11918.5
86	2	–	–	–	–	–	–	3.77	–	–	–	–	–	–	–	–	–	–
87	2	–	–	–	–	–	–	3.85	–	–	–	–	–	–	–	–	–	–
88	3	–	–	–	–	–	–	3.84	–	–	–	–	–	66.29	4.56	0.94	–	11868.5
89	3	–	–	–	–	–	–	3.93	–	–	–	–	–	66.46	4.66	1.07	–	–
90	3	–	–	–	–	–	–	3.95	–	–	–	–	–	66.64	4.62	1.05	–	11972.7
91	3	–	–	–	–	–	–	3.84	–	–	–	–	–	66.72	4.63	1.10	–	–

*Repeat analyses of samples from sample set 1.

Table 6. Total Sulfur Data on Sample Set 3

Sample	Treatment					Total	Elemental	
Number	Oxidant	Time (hr)	Temp.	Catalyst	Microbial	Sulfur (%)	Carbon (%)	Btu
16	H_2O_2	72	RT	-	-	3.25	66.62	11891.9
17	H_2O_2	72	RT	-	-	3.30	66.87	-
18	H_2O_2	72	RT	-	IGTS8	2.48	64.68	11976.5
19	H_2O_2	72	RT	-	IGTS8	2.70	65.04	-
20	H_2O_2	24	50°	-	-	3.36	67.14	11820.4
21	H_2O_2	24	50°	-	-	3.42	67.11	-
22	H_2O_2	24	50°	-	IGTS8	2.89	65.85	11828.9
23	H_2O_2	24	50°	-	IGTS8	2.73	66.14	-
24	H_2O_2	72	RT	HOAc	-	3.17	66.78	11908.6
25	H_2O_2	72	RT	HOAc	-	3.20	67.08	-
26	H_2O_2	72	RT	HOAc	IGTS8	2.90	66.59	11885.6
27	H_2O_2	72	RT	HOAc	IGTS8	2.63	65.20	-
28	H_2O_2	24	50°	HOAc	-	3.38	66.66	11799.0
29	H_2O_2	24	50°	HOAc	-	3.40	66.54	-
30	H_2O_2	24	50°	HOAc	IGTS8	2.40	52.93	9464.8
31	H_2O_2	24	50°	HOAc	IGTS8	2.76	64.14	-
59	$NaBO_3$	24	RT	-	-	3.49	65.30	11629.4
60	$NaBO_3$	24	RT	-	-	3.42	65.39	-
61	$NaBO_3$	24	RT	-	IGTS8	2.95	66.04	11920.1
62	$NaBO_3$	24	RT	-	IGTS8	2.65	64.10	-
63	$K_2S_2O_8$	24	RT	-	-	3.69	67.13	11944.4
64	$K_2S_2O_8$	24	RT	-	-	3.61	67.26	-
65	$K_2S_2O_8$	24	RT	-	IGTS8	2.52	64.92	11888.5
66	$K_2S_2O_8$	24	RT	-	IGTS8	3.01	67.34	-
74	-	-	-	-	IGTS8	2.30	62.60	
75	-	-	-	-	IGTS8	3.16	66.40	-
88	-	-	-	-	-	3.93	66.29	11868.5
89	-	-	-	-	-	3.84	66.46	-
90	-	-	-	-	-	3.95	66.64	11972.7
91	-	-	-	-	-	3.84	66.72	-

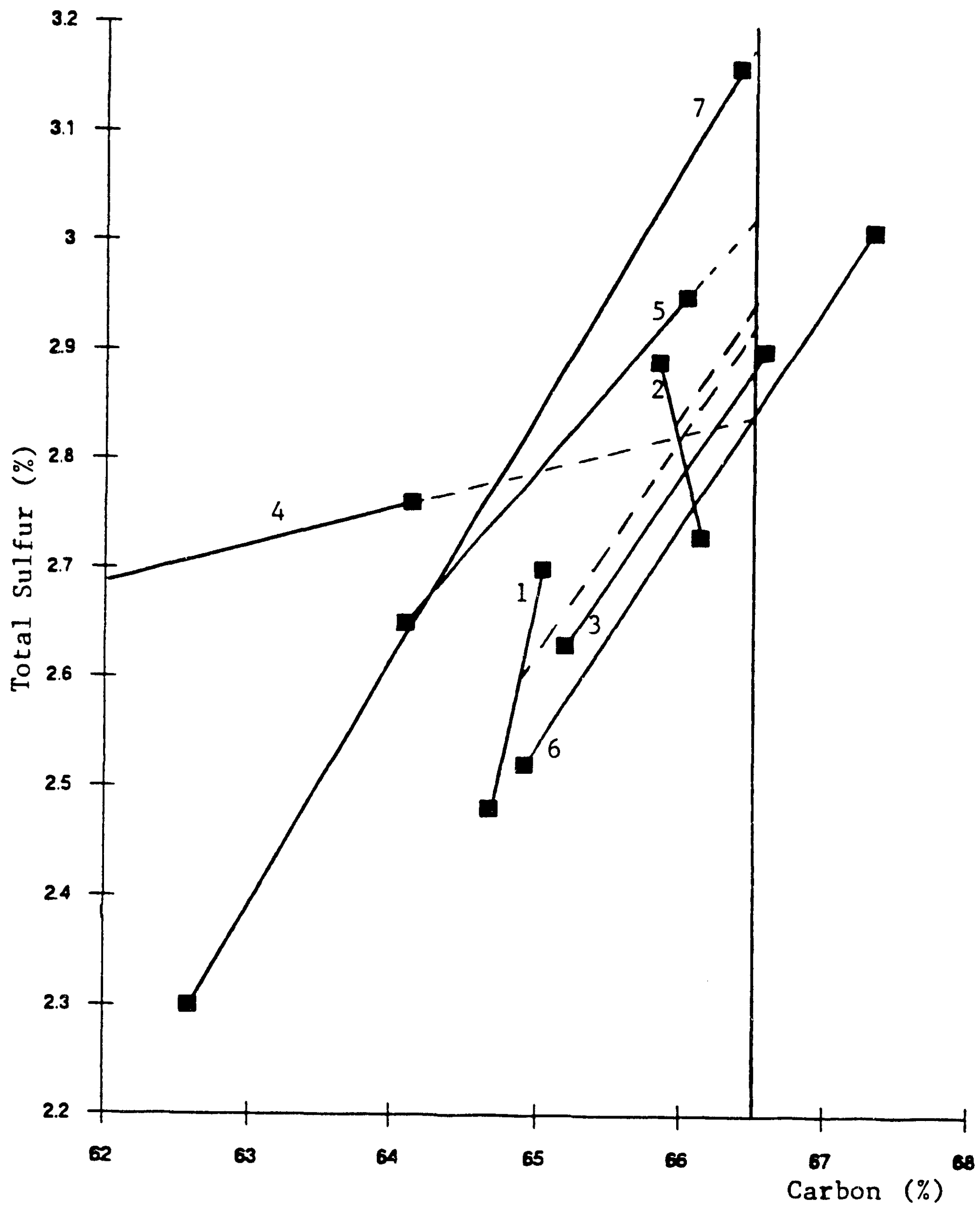


Figure 1. Plot of Total Sulfur versus Carbon for Various Treatments:

1. Hydrogen peroxide, 72 hours at room temperature, plus microbial.
2. Hydrogen peroxide, 24 hours at 50°C, plus microbial.
3. Hydrogen peroxide, 72 hours at room temperature with acetic acid, plus microbial.
4. Hydrogen peroxide, 24 hours at 50°C with acetic acid, plus microbial.
5. Sodium perborate, 24 hours at room temperature, plus microbial.
6. Potassium persulfate, 24 hours at room temperature, plus microbial.
7. No chemical treatment; microbial only.

Appendix I

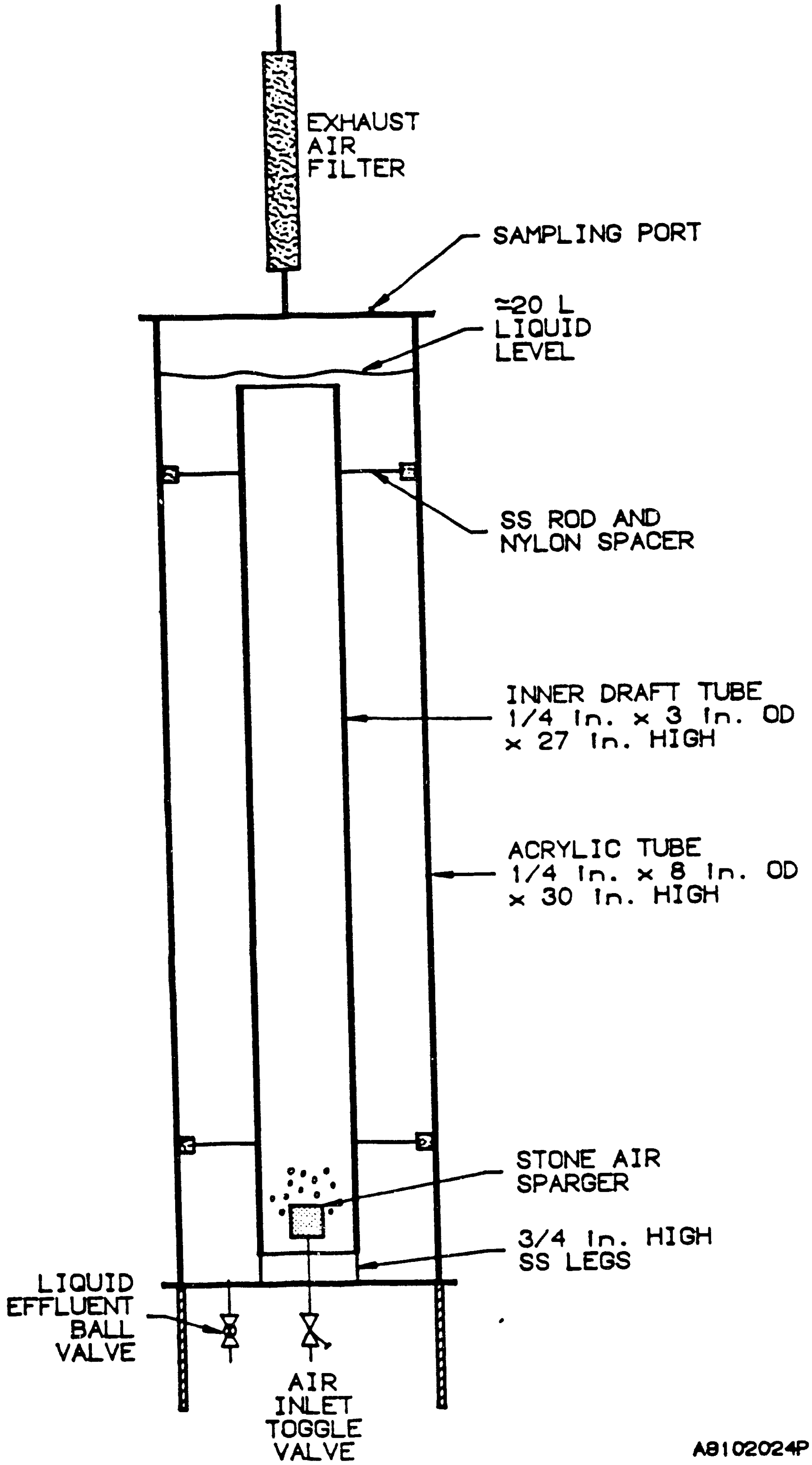
Bioreactor Design and Operation

The objective of this work was to design and operate a reactor for the biological removal of organic sulfur from chemically pretreated coal.

A draft tube bubble column reactor was the design of choice because of the following advantages: 1) Less maintenance due to the absences of moving parts; 2) Solids can be handled without plugging problems; 3) Low operating cost; and, 4) Slow reactions can be carried out due to high liquid residence times.

Sixteen reactors were fabricated from acrylic tubing (Figure 1). The outer tube is 30in high x 8in OD while the inner draft tube is 27in high x 30in OD. Air is supplied from the bottom of the reactor through an air diffusion stone located in the center of the draft tube at a flow rate of about 200mL/min. The air is exhausted through a filter at the top of the reactor before being vented. Sample ports are at the top and bottom of the reactor and the head plate is removable to facilitate reactor entry. The total liquid volume of the bioreactor is 20 liters.

The reactors will be operated in batch mode for two weeks after which time the coal and cells will be harvested, the reactor cleaned, and recharged with fresh media and chemically pretreated coal. Mixing of the reactor contents is provided by the air lift action in the draft tube. These reactors provide an economical means of treating the large volumes of media (20 liters) required for the biological treatment of organic sulfur in chemically pretreated coal.



Schematic of Bioreactor Used for Coal Desulfurization

PROJECT MANAGEMENT REPORT
September 1, 1990 through August 31, 1991

Project Title: Combined Chemical and Microbiological Removal of Organic Sulfur form Coal

Principal Investigator: Leo A. Raphaelian, Institute of Gas Technology

Project Manager: Daniel D. Banerjee, CRSC

(This project is funded by the U. S. Department of Energy as part of its cost-shared program with Illinois Department of Energy and Natural Resources.)

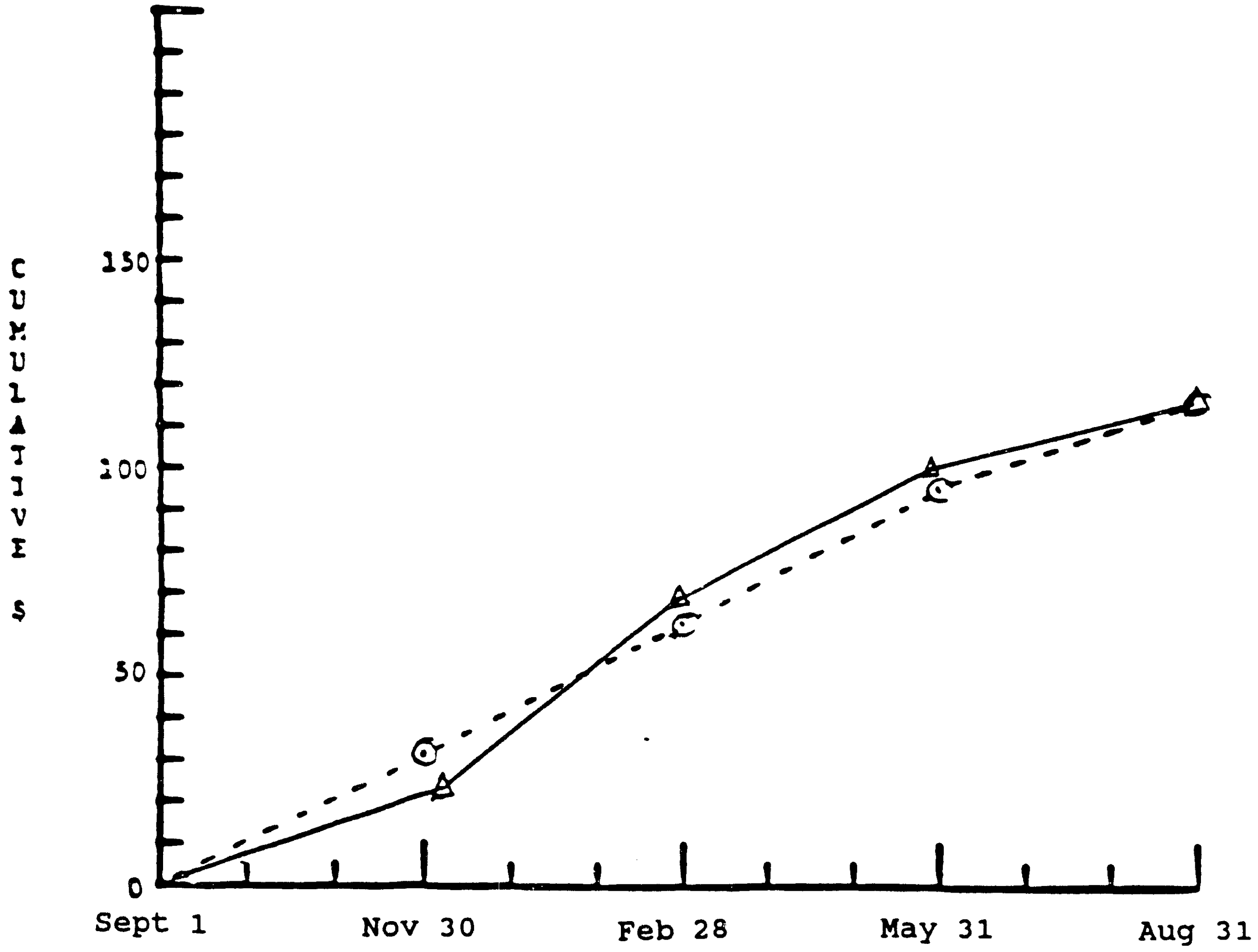
EXPENDITURES - EXHIBIT B

Projected and Estimated Actual Expenditures by Quarter

Quarter*	Types of Cost	Direct Labor	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sep. 1, 1990 to Nov. 30, 1990	Projected	10,641	1,000				19,463	31,104
	Actual	5,789.55	1,875.72				10,856.78	18,522.06
Dec. 1, 1990 to Feb. 28, 1991	Projected	21,282	2,000	426			38,926	62,634
	Actual	21,225.89	6,355.85	172.66			39,730.43	67,484.83
Mar. 1, 1991 to May 31, 1991	Projected	31,923	3,000	426			58,389	93,738
	Actual	31,752.16	8,848.86	172.66			59,277.03	100,050.71
May 1, 1991 to Aug. 31, 1991	Projected	35,662	12,000	1,000			67,179	115,841
	Estimated	35,962	9,296	605			69,978	115,841

COSTS BY QUARTER - EXHIBIT C

Combined Chemical and Microbiological
Removal of Organic Sulfur from Coal



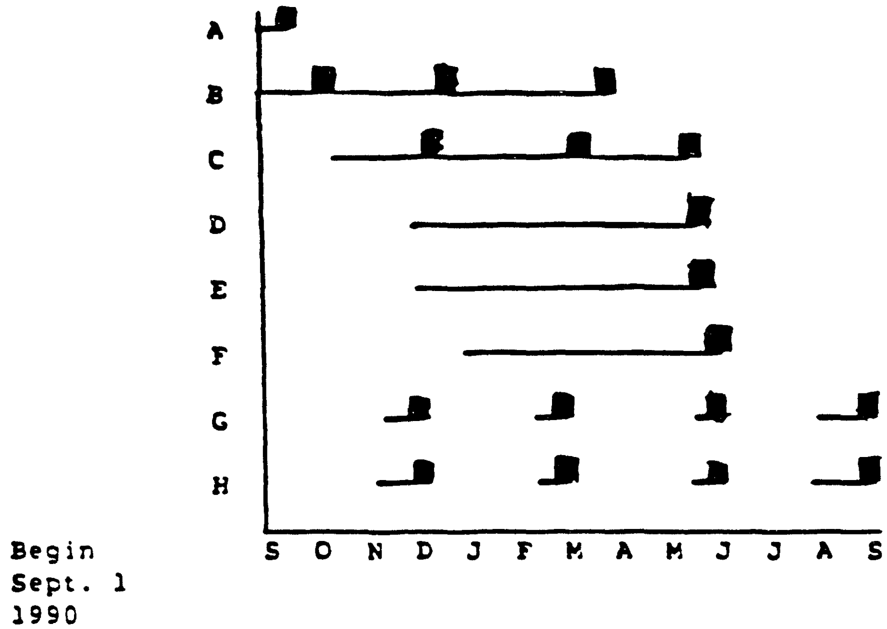
Months and Quarters

O = Projected Expenditures ____________

Δ = Actual Expenditures $115,841

Total CRSC Award $115,841

SCHEDULE OF PROJECT MILESTONES



A. Preparation of coal sample for chemical/microbiological treatment.

B. Chemical oxidation of coal samples.

C. Microbiological treatment of chemically treated coal samples.

D. Chemical analysis of treated coal samples.

E. Determination of fuel value and volatility.

F. Data reduction.

G. Preparation of technical reports.

H. Preparation of project management reports.

END

DATE FILMED

01/29/92