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~~DRAFT~~ TECHNICAL PROGRESS REPORT: April-June, 1996

Biochemical Removal of HAP Precursors from Coal
DE-AC22-95PC95155

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Executive Summary

Analyses were completed on residues from shake flask bioleaching tests with Pittsburgh No. 8 and Indiana No. 5 coal. A shake flask test with Rosebud coal (Montana subbituminous) is in progress. Column tests with Pittsburgh No. 8 coal (28 x 100 mesh) were completed. The rate of biodepyritization of Pittsburgh coal in columns was about 1.3% per day which was about 1/7 the rate of biodepyritization of -100 mesh coal in previous shake flask tests. Significant amounts of HAP precursors Ni, Mn, Co, Cd, and As were solubilized from Pittsburgh coal in columns within 3 weeks, when about 1/3 of the pyrite had been biooxidized. Lesser amounts of F, Be, Se detected in leach solutions. Little or no dissolution of Cr, Pb, Sb, Cl or Hg was detected after 3 weeks. Columns were sacrificed after 10 weeks of bioleaching. A plan for operation, sampling and analysis of coal from tests with the Idaho National Engineering Laboratory's coal slurry reactor was developed. Pittsburgh No. 8 coal was selected for these tests, and it was ground and shipped to INEL. A manuscript and poster were prepared for the PETC contractor's conference.

Concise Summary of Work Performed

Analytical methods were finalized and all analyses completed on shake flask tests with Indiana No. 5 and Pittsburgh No. 8 coal. A column leaching-rotating biological contactor (RBC) unit was used to bioleach pyrite and HAP precursors from Pittsburgh No. 8 coal. Shake flask tests with Rosebud subbituminous coal were begun. In connection with upcoming slurry column reactor tests, coal was prepared and shipped to INEL, and a detailed work plan was developed for operation and sampling for the tests. A manuscript and poster was prepared for presentation at the PETC contractor's conference.

Variances

There were no major variances to the work plan.

Open Items

There are currently no open items in the project.

Forecast for Upcoming Quarter

Test work will proceed on schedule in accordance with the following work. Shake flask tests on Rosebud coal will be completed and final set of shake flask tests with Kentucky No. 9 coal will begin. A column test with Indiana coal will be started. Testing will begin of HAP precursor removal in the slurry column reactor at the Idaho National Engineering Laboratory.

Technical and Scientific Results

1. Analytical Considerations

The problem of selenium analyses in coal was resolved. As reported last quarter, various modifications of Eschka coal oxidation were tested, but all showed low Se spike recoveries and poor results with NIST coal. However, hot aqua regia treatment (used in the EPA method 7471 mercury analysis) followed by hydride-ICP analysis, gave accurate quantitation of Se in coal. Duplicate analyses of NIST 1632b bituminous coal, certified to contain 1.29 ug/g of Se, showed 1.1 and 1.4 ug/g Se, or 85% to 109% of the true value, despite the fact that Se is present in NIST coal at only about twice our detection limits. Recovery of Se spiked into the aqua regia extracts of Pittsburgh, Indiana and Rosebud coals ranged from 87 to 93%.

Table 1 summarizes approximate detection limits for the inorganic HAP precursors in coal and the digestion method used prior to ICP spectroscopy. Mercury is determined by cold vapor atomic absorption spectrophotometry. These values may vary somewhat on a given day depending on ICP instrument sensitivity and precision. The methods were described more fully in the first and second quarter reports.

Table 1. Approximate Detection Limits of HAP Precursors in Coal (µg/g)

element	detection limit	digestion method
mercury	0.02	EPA 7471
arsenic	0.5	ASTM 4606
cadmium	0.2	ASTM 3683
chromium	0.5	ASTM 3683
selenium	0.5	EPA 7471
antimony	0.5	ASTM 4606
beryllium	0.2	ASTM 3683
cobalt	0.5	ASTM 3683
lead	2	ASTM 3683
manganese	0.2	ASTM 3683
nickel	0.5	ASTM 3683

2. Shake Flask Tests--Results of Pittsburgh No. 8 and Indiana No. 5 Coals (Task 2)

Most of the HAP precursor analyses from these tests were reported in Tables 1, 4, 5, and 8 in the second quarter report. However, some analyses, most notably Hg, Sb, and Se, were not complete at that time. Tables 2-5 below present final results for all HAP precursors in the shake flask tests with Pittsburgh and Indiana coals, along with ultimate, short proximate, and forms of sulfur analyses.

Results of Hg and Sb analyses showed no measurable bioleaching of these elements into solution with either Pittsburgh or Indiana coal. Mass balances for these elements were good in Indiana coal. Pittsburgh coal showed two values for Hg and three values for Sb that were outside the 80% to 120% range which we generally deem acceptable for mass balances. The two high mercury values were only slightly over the limit (125%) and are within (standard deviation 15%) the variance of the analytical method. The antimony content of Pittsburgh coal is at the detection limit, and so relatively high percent variation of replicate analyses can be expected.

Results of Se solution analyses showed measurable bioleaching of Se from Pittsburgh coal, corresponding to 17% Se removal (Table 3), although solids analyses (Table 2) did not give evidence for bioremoval of Se. However, it is likely that Se was bioleached since solution analyses are more sensitive, accurate, and precise than solids analyses, and since Se was present at only 2 to 3 times the detection limit in Pittsburgh coal. Also, the Se concentration in the starting coal may be low, given the mass balance results (Table 3) and the fact that the 28 x 100 mesh fraction from the same sample (discussed later in this report) contained a higher Se content (about 1.7 to 1.8 μg/g).

Table 2. Analysis of Pittsburgh 8 Coal: Shake Flask Test (-100 mesh, dry basis)

	Raw coal	Biotreated	Control	%loss-microbial	% loss-control
% carbon	67.95	70.35 70.49	69.43 69.50		
% hydrogen	4.59	4.53 4.51	4.52 4.49		
% nitrogen	1.38	1.37 1.36	1.33 1.33		
% sulfur	2.69	1.50 1.53	2.17 2.29	44 43	19 15
% ash	16.66	14.82 15.27	16.03 15.97	11 8	4 4
% oxygen (diff)	6.73	7.43 6.84	6.52 6.42		
Btu/lb	12120	12446 12433	12314 12307		
% pyritic sulfur	1.17	0.12 0.12	0.80 0.87	90 90	32 26
% sulfate sulfur	0.32	0.24 0.27	0.22 0.21		
% org. S (diff)	1.20	1.14 1.14	1.15 1.21		
HAPS, µg/g coal					
mercury	0.18 0.16	0.22 0.22	0.16 0.20	0	0
arsenic	8.9 8.4	4.9 4.4	7.6 8.8	43 49	12 0
cadmium	1.0 1.1	0.5 0.6	0.7 0.7	53 46	32 30
chromium	29.1 29.6	25.1 25.9	28.1 28.3	15 12	4 4
selenium	1.0 1.0	1.0 1.4	1.5 1.5	0	0
antimony	0.6 0.5	0.4 0.3	0.6 0.4	33 50	0 33
beryllium	<0.2 <0.2	<0.2 <0.2	<0.2 <0.2		
cobalt	7.6 7.3	4.7 4.7	5.2 5.3	37 37	30 29
lead	12.3 14.3	13.9 13.2	13.1 12.3	0 1	2 8
manganese	63.5 59.6	22.6 24.6	29.0 30.5	63 60	53 50
nickel	17.3 17.4	10.9 11.3	13.6 13.6	37 35	22 22
chlorine (%)	0.08	0.08 0.08	0.08 0.07	0 0	0 12
fluorine	120	109 133	151 138	9 0	0 0

Duplicate values are for duplicate flasks. Raw coal HAPs duplicates represent duplicate digestions.

Table 3. Mass Balances for Pittsburgh 8 Shake Flask Test*

	starting coal mg	solution mg	final coal mg	% recovery	% loss (by solution)
Hg cells	0.0051	<0.0001	0.0064	125	<2
cells	0.0051	<0.0001	0.0064	125	<2
control	0.0051	<0.0001	0.0048	94	<2
control	0.0051	<0.0001	0.0060	118	<2
arsenic	0.259	0.120	0.143	102	46
	0.260	0.111	0.127	92	43
	0.259	0.028	0.225	98	11
	0.259	0.004	0.262	103	2
cadmium	0.032	0.010	0.014	75	31
	0.032	0.010	0.017	84	31
	0.032	0.006	0.021	84	19
	0.032	0.006	0.022	88	19
chromium	0.882	0.035	0.729	87	4
	0.884	0.034	0.754	89	4
	0.882	0.019	0.833	97	2
	0.882	0.015	0.846	98	2
selenium	0.030	0.005	0.029	113	17
	0.030	0.005	0.041	153	17
	0.030	<0.001	0.044	147	0
	0.030	<0.001	0.045	150	0
antimony	0.018	<0.001	0.012	67	<6
	0.018	<0.001	0.009	50	<6
	0.018	<0.001	0.018	100	<6
	0.018	<0.001	0.012	67	<6
beryllium	<0.006	0.002	<0.006		
	<0.006	0.002	<0.006		
	<0.006	0.002	<0.006		
	<0.006	0.002	<0.006		
cobalt	0.225	0.063	0.135	88	28
	0.225	0.064	0.136	89	28
	0.225	0.056	0.153	93	25
	0.225	0.055	0.159	95	24
lead	0.399	0.025	0.404	108	6
	0.399	0.025	0.384	103	6
	0.399	0.016	0.388	101	4
	0.399	0.013	0.367	95	3
manganese	1.848	0.863	0.657	82	47
	1.853	0.874	0.715	86	47
	1.848	0.761	0.859	88	41
	1.848	0.737	0.911	89	40
nickel	0.523	0.153	0.317	90	29
	0.524	0.156	0.328	92	30
	0.523	0.120	0.403	100	23
	0.523	0.117	0.406	100	22
chlorine	24	2	23	104	8
	24	4	23	113	17
	24	0	24	100	0
	24	3	21	113	13
fluorine	3.60	0.83	3.17	111	23
	3.60	0.77	3.87	129	21
	3.60	0.51	4.47	138	14
	3.60	0.45	4.12	127	13

*Four values are shown for each HAP precursor. As shown for Hg above, the first two rows are for the two inoculated flasks, the last two rows are for control flasks

Table 4. Analysis of Indiana 5 coal: Shake Flask Test (-100 mesh, dry basis)

	Raw coal	Biotreated	Control	%loss-microbial	% loss-control
% carbon	70.67	72.05 72.27	70.28 70.69		
% hydrogen	4.21	4.76 4.89	4.66 4.82		
% nitrogen	1.44	1.50 1.49	1.45 1.44		
% sulfur	4.09	2.85 2.79	4.36 4.22	30 32	0 0
% ash	12.4	9.42 9.56	12.19 11.12	24 23	2 10
% oxygen (difference)	7.19	9.42 9.00	7.06 7.71		
Btu/lb	12519	12798 12826	12635 12677		
% pyritic sulfur	2.21	0.47 0.52	1.97 2.16	79 76	11 2
% sulfate sulfur	0.14	0.27 0.24	0.13 0.12		
% organic S (difference)	1.74	2.11 2.03	2.26 1.94		
HAPs, µg/g coal					
mercury	0.11 0.12	0.13 0.13	0.11 0.11	0 0	0 0
arsenic	5.5 5.7	2.9 3.0	5.3 7.3	48 46	5 0
cadmium	0.5 1.2	0.4 0.8	0.5 0.7	50 0	37 0
chromium	12.2 11.6	13.8 12.8	12.6 13.0	0 0	0 0
selenium	1.8 1.1	1.4 1.4	1.5 1.2	7 7	0 20
antimony	0.6 0.7	0.8 0.6	0.8 1.0	0 0	0 0
beryllium	0.8 0.8	0.8 0.7	0.9 0.7	6 12	0 17
cobalt	4.8 4.4	2.6 2.6	3.3 3.4	44 44	28 27
lead	9.7 9.4	9.1 9.6	10.7 9.2	6 0	0 2
manganese	39.1 43.1	14.8 19.0	17.3 21.0	64 54	58 49
nickel	16.0 15.6	10.8 10.9	15.6 14.7	32 31	1 7
chlorine (%)	0.01	0.01 0.01	0.00 0.01	0 0	100 0
fluorine	70	63 66	58 66	10 6	17 6

Table 5. Mass Balances–Indiana 5 Shake Flask Test

	starting coal mg	solution mg	final coal mg	% recovery	% loss (by solution)
mercury	0.0048	<0.0002	0.0047	98	<4
		<0.0002	0.0054	113	<4
		<0.0002	0.0049	102	<4
		<0.0002	0.0041	85	<4
arsenic	0.224	0.099	0.105	91	44
		0.091	0.112	91	41
		0.003	0.198	90	1
		0.003	0.264	119	1
cadmium	0.033	0.015	0.013	85	45
		0.016	0.027	130	48
		0.011	0.015	77	33
		0.011	0.022	100	33
chromium	0.476	0.021	0.499	109	4
		0.020	0.464	102	4
		0.012	0.472	102	3
		0.018	0.487	106	4
selenium	0.060	0.010	0.051	102	17
		0.009	0.052	102	15
		<0.001	0.056	93	0
		<0.001	0.043	72	0
antimony	0.028	<0.001	0.029	104	<4
		<0.001	0.025	86	<4
		<0.001	0.030	107	<4
		<0.001	0.037	132	<4
beryllium	0.033	0.003	0.028	94	9
		0.004	0.025	88	12
		0.003	0.033	109	9
		0.003	0.027	91	9
cobalt	0.190	0.087	0.093	95	46
		0.085	0.093	94	45
		0.063	0.123	98	33
		0.060	0.125	97	32
lead	0.384	0.038	0.330	96	10
		0.038	0.347	100	10
		<0.005	0.400	104	<1
		<0.005	0.345	90	<1
manganese	1.645	1.021	0.536	95	62
		0.981	0.689	102	60
		0.688	0.648	81	42
		1.138	0.786	117	69
nickel	0.633	0.246	0.393	101	39
		0.241	0.395	100	38
		0.110	0.583	109	17
		0.108	0.550	104	17
chlorine	4	<3	4	100	0
		3	4	175	75
		<3	0	100	0
		<3	4	100	0
fluorine	2.80	0.08	2.27	84	3
		0.08	2.38	88	3
		0.07	2.17	80	3
		0.07	2.47	91	3

3. Shake Flask Tests with Rosebud Coal (Task 2)

Shake flask tests were begun with Rosebud coal (-100 mesh). Initial coal analyses are shown in Table 6 below. A duplicate sample was spiked with 1.6 µg/g Hg, 5 µg/g of Cd and Se, and 20 µg/g As, Sb, Cr, Be, Co, Pb, Mn, and Ni and was carried through the digestion procedures. Spike recoveries ranged from 88% to 112%, with the exception of Sb which was 66%. The table below will be completed in the next quarterly report when the test and residue analyses are completed.

Table 6. Analysis of Rosebud Coal, Shake Flask Test (-100 mesh, dry basis)

	Raw coal	Biotreated	Control	%loss-microbial	% loss-control
% carbon	62.73				
% hydrogen	3.86				
% nitrogen	1.01				
% sulfur	1.74				
% ash	19.57				
% oxygen (difference)	11.09				
Btu/lb	10541				
% pyritic sulfur	0.86				
% sulfate sulfur	0.32				
% organic S (difference)	0.56				
HAPs, µg/g coal					
mercury	0.06 0.04				
arsenic	1.6				
cadmium	0.1				
chromium	4.4				
selenium	1.1				
antimony	0.5				
beryllium	0.15				
cobalt	0.9				
lead	10.8				
manganese	133				
nickel	2.2				
chlorine (%)	<0.01%				
fluorine	44				

Four flasks (250 ml Erlenmeyer) each received 35 g of Rosebud coal (Penn State coal bank sample DECS-10) and 140 ml of MKM solution at pH 1.8. After shaking for several hours, the pH rose to nearly 6.0. A total of 2.6 ml of additional 10N H_2SO_4 was added over the next two days to keep the pH at about 2. Two flasks received 3 ml of

2% thymol in methanol as a biocide. Two flasks were inoculated with a mixed population of bacteria growing on a pyrite-Pittsburgh coal-Indiana coal slurry in MKM at pH 1.8. The initial cell concentration in inoculated flasks was 2.1 x 10^7 cells per ml. The flasks were incubated at 22-26°C at 180 rpm. Samples were removed once or twice weekly to monitor progress of bioleaching by pH, redox potential, sulfate, total iron and ferrous iron. About 15% of the pyrite had been biooxidized at the end of the quarter and the first liquid sample was removed for analysis of HAP precursors. Plots of pyrite oxidation kinetics and HAP precursors in solution will appear in the next quarterly report.

4. Column Tests with Column-Rotating Biological Contactor (RBC) System (Task 3)

Microbiological leaching of HAP precursors from 28 x 100 mesh Pittsburgh No. 8 coal was tested in columns. At the end of the quarter, the test had just been completed, but results of HAP precursor analyses in final leach solutions and coal residues were not yet available. Table 7 lists components of the starting coal. Duplicate analyses of Mn showed poor precision, and reanalyses are in progress.

The column-RBC system was used as a small scale simulation of the potential effectiveness of heap bioleaching of pyrite and HAP precursors. Acidic, oxidizing (high ferric ion content) solutions created by microbial pyrite oxidation convert metal sulfides to soluble sulfates and may solubilize non metallic HAP precursors as well.

The column-RBC system consisted of six plastic columns of 300 ml capacity, 4 x 30 cm in size. Two columns were positioned above each of 3 plastic reservoirs. One hundred grams of 28 x 100 mesh Pittsburgh No. 8 coal was added to each column. Two hundred ml of double strength MKM mineral salts solution (pH 1.8) was poured through each column. After the solution dripped through each pair of columns, the initial weight of the 3 reservoirs was determined, and a mark was made on the side of the container so that distilled water could be added to correct for future evaporative losses. The columns retained about 35 ml of solution.

Two of the three reservoirs were inoculated with a mixed population of pyrite oxidizing bacteria grown on a pyrite-Pittsburgh coal slurry in MKM. The initial cell concentration in the reservoir solutions was 1.6 x 10^8 / ml. The third reservoir was not inoculated and received 5 ml of 2% thymol in methanol as a biocide. A six channel peristaltic pump was operated at a pump rate of 45 ml/hour to recirculate solution from the reservoirs through the columns. A gear motor was used to rotate (36 rpm) a series of 7 cm diameter plastic disks partially submerged in the reservoirs. This RBC was used to maintain sufficient aeration of the system to permit efficient iron biooxidation in reservoirs. Iron oxidizing bacteria in solution and in biofilms on the disks oxidized Fe^{2+} to Fe^{3+}. The ferric-rich solution was then be pumped to the top of the columns.

After two days of recirculation, the pH of the solutions had climbed to a value of about 3.0, and so 200 ul of 11N H_2SO_4 was added to bring the pH down to 2.0. Within a

Table 7. Column Test Analysis of Pittsburgh No. 8 Coal (28 x 100 mesh, dry basis)

	Raw coal	Biotreated	Control	%loss-microbial	% loss-control
% carbon	65.09				
% hydrogen	4.55				
% nitrogen	1.34				
% sulfur	2.34				
% ash	20.00				
% oxygen (difference)	6.68				
Btu/lb	11613				
% pyritic sulfur	1.11				
% sulfate sulfur	0.12				
% organic S (difference)	1.11				
HAPs, ug/g coal					
mercury	0.11 0.11 0.09				
arsenic	8.7 8.3				
cadmium	0.3 0.3				
chromium	26 29				
selenium	1.8 1.7				
antimony	1.0 0.4				
beryllium	0.8 0.8				
cobalt	6.6 7.0				
lead	16.7 17.6				
manganese[1]	49 142				
nickel	16.0 16.5				
chlorine (%)	0.07				
fluorine	120				

[1] reanalysis in progress

week, the leach solution in the reservoirs from the inoculated columns had turned brown, indicating the formation of ferric iron in solution, while the leach solution in the reservoir of the control columns remained clear for the duration of the test. At this time, the dissolved oxygen level in the inoculated reservoirs was 3 to 4 mg/l, compared to 6 mg/l (near saturation at this altitude) in the control reservoir.

The columns were operated in a continuous recirculation mode for 10 weeks. On three occasions during the test it was necessary to add more thymol to the control reservoir. The progress of bioleaching was determined by once or twice weekly sampling for pH, redox potential, sulfate, total iron and ferrous iron. In inoculated columns, the pH values gradually dropped to 1.7 to 1.8 as pyrite oxidation proceeded, the redox potential was generally above 500 mV (sce) and less than 5% of the iron in solution was ferrous. These measurements (along with solution sulfate analyses reported below) confirmed the activity of pyrite oxidizing bacteria. In contrast, the leach solution from control columns (treated with biocide to prevent pyrite biooxidation) remained at a pH value of 3 to 4, the redox potential remained generally below 400 mV (sce), and virtually all solution iron was in the ferrous form.

Samples of leach solution were removed for HAP precursor analysis after day 14 and day 23. These results (Table 8) showed that 24% or more of the Co, Ni, As, and Cd in the coal was is solution in inoculated columns after 23 days. Significant Mn was also solubilized, but additional analyses of the Mn content in the starting coal are required before percent removal can be calculated. These five elements are the same elements that were found to be significantly removed by bioleaching in the shake flask tests (previous quarterly report). Results of HAP precursor analysis of final solutions will be included in the next quarterly report.

When solution iron and sulfate measurements indicated microbial removal of about 70% of the pyrite in the coal, the test was concluded. At this point, the columns were allowed to drain, and the volume of leach solution was determined. The columns were rinsed with MKM until no orange color of ferric ions was visible, followed by a rinse with distilled water. The solids from each pair of columns were combined, dried, and weighed. Of the initial 200 grams, 195.95 g (98.0%) and 196.69 g (98.3%) of coal were recovered from the inoculated columns and 198.35 g (99.2%) was recovered from the control columns. The leach solutions and solid coal were analyzed for HAP precursors, but results were not complete at the end of the quarter.

The kinetics of pyrite oxidation as determined by solution iron and sulfate analyses are shown in Fig. 1. The kinetics over the first 5 weeks of leaching appear to be faster than over the final 5 weeks (Table 9). Rates shown below are based on a least squares linear regression calculation of pyrite biooxidation rates based on solution iron and sulfate concentrations. Little oxidation occurred in control columns. The rates shown are 14% (Fe) to 16% (sulfate) of the rates obtained with minus 100 mesh coal reported last quarter.

Table 8. % of Coal HAP Precursors in Leach Solutions[1] in Pittsburgh No. 8 Columns

	Day 14	Day 23	Final (day 70)
% pyrite biooxidation (based on solution SO_4 concentration)	17 (cells) 14 (cells) 3 (control)	35 34 6	
Arsenic	11 22 <1	28 24 <1	
Cadmium	30 30 10	58 58 12	
Selenium	4 2 <1	7 6 <1	
Beryllium	5 5 2	7 8 1	
Cobalt	22 23 20	32 33 17	
Nickel	19 28 17	28 52 18	
Fluorine	16 16 10	16 16 5	

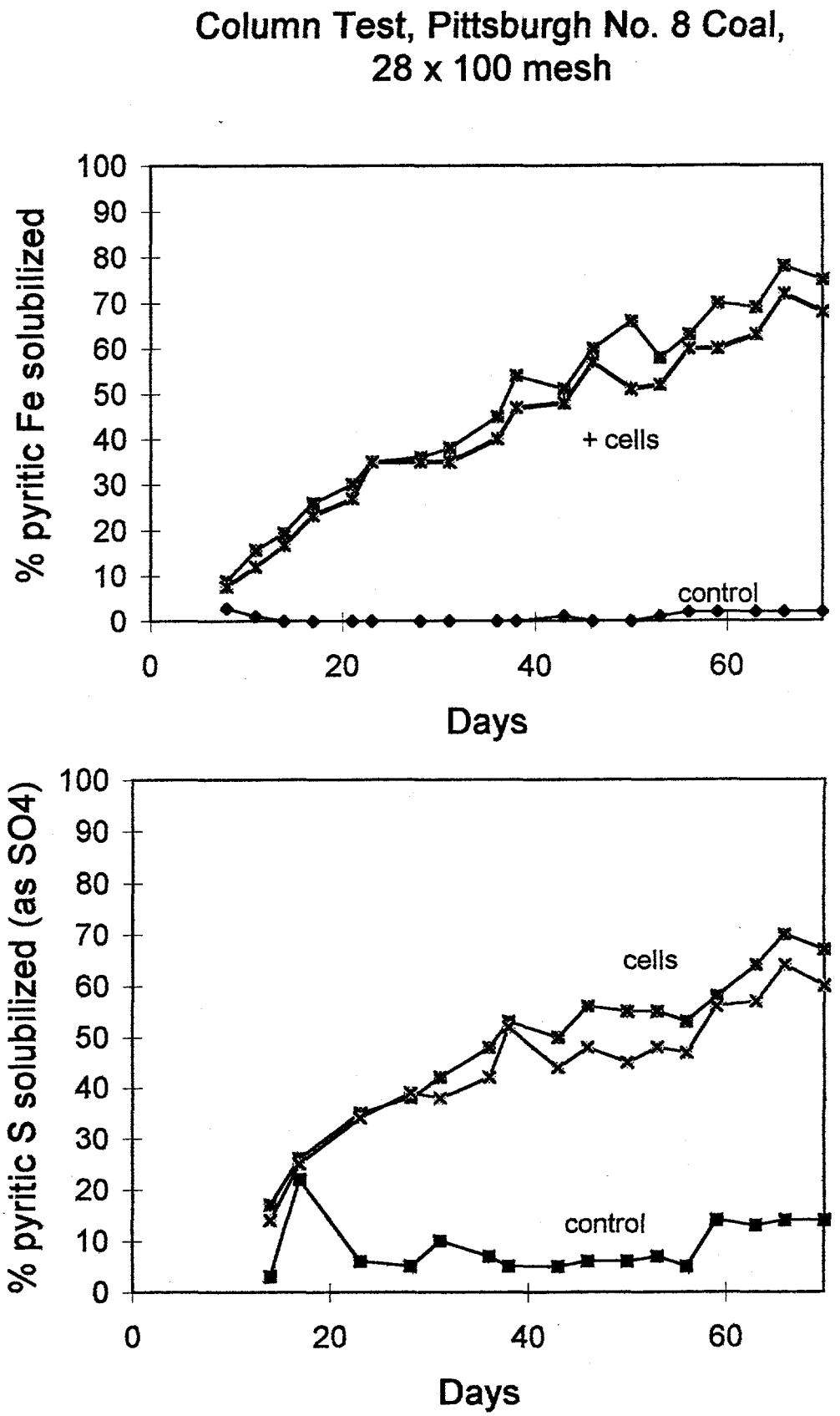
[1] Less than 1% of Sb, Hg, Cl, Cr, Pb was solubilized. Significant Mn was solubilized but starting concentration in coal not yet determined.

Table 9. Daily Rates of Pyrite Biooxidation (%) in Leach Columns[1]

column pair	Solution Fe Rate		Solution SO_4 Rate	
	Day 8-38	Day 8-70	Day 14-38	Day 14-70
1	1.2	1.1	1.3	0.8
2	1.2	1.0	1.3	0.7

[1]28 x 100 mesh Pittsburgh coal; based on solution Fe or SO_4 concentrations

Fig. 1. Kinetics of Pyrite Oxidation in Columns



4. INEL Slurry Column Test Work (Task 4)

A meeting was held in Idaho Falls with our subcontractor, Unifield Engineering, and with Dr. Karl Noah of INEL (the government furnished facility), to discuss sampling and operating protocols for the slurry column test work to be done in phase 1 of this project. The outcome of the meeting was the preparation of a detailed plan for testing, operational, and sampling procedures to be conducted during the upcoming slurry column tests.

Together with project monitor Dr. Mike Nowak, a Pittsburgh No. 8 coal was chosen for these tests, since others are working on HAP precursors in Pittsburgh coal and since INEL has experience with bio/physical processing of this coal in the slurry column reactor.

Pittsburgh No. 8 coal was ground at PETC's Coal Preparation Division, and five drums were shipped to INEL. Evaluation of the coal will begin in July.

5. Reporting Results (Task 5)

A manuscript describing research results was prepared and sent to PETC for inclusion in the proceedings of the contractor's conference held in July in Pittsburgh. A poster was also prepared for presentation at the conference.

Effects of Results on Future Work

The project is proceeding on schedule. Quantitation of all 13 inorganic HAP precursors in coal is proceeding with good mass balances, and so the additional shake flask test, column tests and INEL slurry column tests can proceed unimpeded by analytical considerations. The selection of a Pittsburgh No. 8 coal for slurry column tests at INEL was based in part on shake flask and column test results.

As the third quarter ended, three of the four planned shake flask tests were completed or in progress. The first of four column tests was concluding. Plans for operation of the INEL slurry column reactor and its sampling were completed and a coal was selected and shipped to INEL. Sampling protocols and analytical procedures are now fully developed and routine; the remaining phase 1 work should proceed according to plan.