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Project Title: Recovery of Coal Fines from Preparation Plant Effluents

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ABSTRACT

The objectives of this project were to test and demonstrate the feasibility of recovering coal fines that are currently disposed of with coal preparation plant effluent streams and producing a fine clean coal product that can be blended with the plant coarse clean coal. This recovery was effected by means of Michigan Technological University's static tube flotation process, which was successfully demonstrated on a number of raw coals to reject 85% of the pyritic sulfur and recover 90% of the combustible matter. Under this project, the process parameters for the technology were modified for this application in order to recover a low-ash, low-sulfur clean coal that is, at a minimum, compatible with the quality of the clean coal currently produced by the preparation plant.

The preliminary phase of the work under this project consisted of collecting effluent samples from four plants operating in Southern Illinois and characterizing them with respect to particle size distribution, sink-float testing, mineralogical and chemical composition, and studies on wettability by film flotation. The sink-float testing was undertaken on the +230 mesh portion of the effluent since the -230 mesh slimes, with an ash content of about 70%, were essentially clays. These tests provided an estimate of the liberation of the coal from the mineral matter in the effluent samples.

Static tube flotation tests conducted at Michigan Technological University on the four preparation plant effluent samples achieved excellent results. A test conducted using effluent Sample A resulted in a clean coal with 7.5% ash and 1.1% total sulfur at 97.7% Btu recovery. Another test on the same sample aimed at lowering the ash further analyzed at 3.0% ash and 0.92% total sulfur but at a lower Btu recovery. Test results on the other three samples confirmed that the static tube can be used to recover fine coal containing low ash and sulfur with high recovery of carbonaceous material from plant waste streams. In addition, the quality of the recovered coal can be changed by controlling the process operating parameters. The static tube test results were compared with the washability analyses of the effluent samples and with results from conventional froth flotation tests. The performance of the static tube closely approached the theoretical limit of separation indicated by the washability tests.

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EXECUTIVE SUMMARY

Owing to the nature of Illinois basin coals, cleaning of fine coal has not been economically attractive. Coal preparation plants typically clean only the coarse coal and dispose of all material finer than 28 mesh in size in tailings ponds. In a few plants, however, some of the coarser fines (+ 100 mesh) are recovered by size classification but without cleaning and the remaining material is discarded.

The practice of disposing of fines in tailings ponds has been followed to a larger extent in plants processing mid-continent coals than in those processing Appalachian coals because the former are more hydrophilic in nature and require high dosages of collectors for conventional flotation. According to a recent study conducted by the Illinois State Geological Society (ISGS), coal cleaning generates about 20 million tons of refuse per year in the State of Illinois, of which 4-6 million tons is recoverable fine coal. Each year, plant effluents containing recoverable fines are disposed of in ponds covering 300 acres of land to a depth of 15 feet. In addition to this ongoing disposal, Illinois had 120 abandoned slurry ponds which may pose environmental problems and require costly remediation in the future.

The CRSC funded a project to test the suitability of the MTU static tube flotation process to recover a low-ash, low-sulfur fine coal product from preparation plant effluents which would be suitable for blending with the existing plant product. The static tube flotation process differs from both column and conventional flotation in that it uses a packed tube to ensure intimate particle-bubble contact. This design eliminates the problem of vertical mixing encountered with column flotation and that of the difficulty of recovering very fine coal particles encountered with conventional flotation.

Samples of the effluent streams were collected from four coal preparation plants operating in the Southern Illinois region. The following characterization tests were performed on the samples:

- o Particle size analysis (sieve and sub sieve)
- o Ash, total sulfur, pyritic sulfur, Btu analysis of + 230 mesh and -230 mesh fractions
- o Sink-float testing
- o Film flotation on the 45 x 60-mesh fraction
- o Conventional flotation on the 45 x 60-mesh fraction
- o Zeta potential
- o Mineralogical composition

- o Froth flotation testing

Sieve analysis of the +230-mesh fraction of the effluent samples indicated that Samples B and D have an similar size distribution. Sample C is considerably coarser in size and needed to be comminuted prior to conducting flotation tests. The minus 230-mesh content of Sample A is almost identical to that of Sample C, but it contains very little +14-mesh material. This analysis indicated that all of the samples would achieve additional liberation of mineral matter following further comminution.

Film flotation tests conducted on the 45 x 60-mesh fraction of the four effluent samples indicated that at all values of surface tension Sample D has the least amount of lyophobic material. This indicates that Sample D may require new coal surfaces to be generated and/or surface-active agents to be added during flotation. Sample A has the highest amount of lyophobic material. In general, the relative lyophobicity of the four effluent samples tested is, in increasing order: A, B, C and D.

Conventional flotation tests were conducted on the 45 x 60-mesh fraction of the four effluent samples for correlation with the film flotation test results. Sample D was determined to be very slow-floating; these results were consistent with the film flotation tests. Detailed flotation testing was also conducted on Sample A, which was ground to 75% passing 200 mesh and tested along with a number of frothers, promoters, and pyrite depressants. This resulted in an improvement in the flotation performance of this coal. For example, a 10.6% ash product containing 1.25% pyritic sulfur was produced during a standard flotation test using a pyrite depressant. This performance was marginally better than that of a two-stage flotation test without a depressant. The weight recovery in these two tests was 33% and 38% respectively.

Sink-float tests were conducted on the +230-mesh fraction of the effluent samples on the assumption that the minus 230-mesh material essentially consists of mineral matter and that the high clay content of this fraction would hinder sink-float separation. It was therefore treated as slimes and excluded from the sink-float tests. However, static tube flotation testing and some froth flotation tests were conducted using the entire effluent sample.

The washability test results indicated that while the effluent samples have some common characteristics, they are different from one another in nature. Analysis of the washability data provided an excellent picture of the cleanability of the four effluent samples. These results indicated the following:

- o Sample A contains a low ash (3.3%) and low total sulfur (0.93%) material at 1.3 specific gravity and allows high overall pyritic sulfur rejection. However, this sample yielded less recoverable coal than did the other samples.
- o Sample B contains low ash (4%) and medium sulfur (2.72%) recoverable coal at 1.3 specific gravity due to its inherent high total sulfur content. The organic sulfur (by difference) for this coal is very high, as is typical of Illinois No. 6 coals, but the pyritic sulfur

is in a reasonably liberated form. High weight recovery is possible from this effluent.

- o Sample C yields a low amount of recoverable coal with an ash content of 5.05%. The pyritic sulfur rejection is not very high primarily due to the coarse nature of the material. This sample needed to be comminuted prior to further testing to improve liberation.
- o Sample D yields a coal with a higher ash content (5.21%) than the other samples at comparable specific gravities of separation. While its total sulfur is high, it will allow good pyritic sulfur rejection during cleaning.

Initial static tube tests were conducted to establish operating parameters and reagent dosages. This was followed by extensive tests on each effluent sample following grinding in an attempt to reduce the topsize to 85% passing 200 mesh. It was considered useful to grind the samples in order to generate new surfaces and eliminate any +30-mesh material. This is especially beneficial in the case of Sample C, which contained 51% of the material in the +30-mesh size.

The following table presents a summary of the static tube flotation test results alongside the froth flotation test results for purposes of comparison. As this table indicates, the static tube flotation test results for all of the samples were excellent. When compared to the results of froth flotation tests the static tube performance is superior. The froth flotation test was conducted after grinding the effluent slurry to 75% passing 200 mesh.

COMPARISON OF STATIC TUBE PERFORMANCE WITH FROTH FLOTATION

<u>Source</u>	<u>Procedure</u>	<u>Product Analysis</u>			<u>Performance</u>		
		<u>Ash%</u>	<u>TotS%</u>	<u>PyS%</u>	<u>%Btu Rec</u>	<u>%PyS Rej</u>	<u>Eff Ind</u>
Sample A	Static Tube	7.5	1.06	0.51	97.7	73.6	71.3
	Froth Flotation	12.7	3.10		92.5		
Sample B	Static Tube	8.5	3.15	1.2	91.2	67.6	58.8
	Froth Flotation	19.9	3.16		86.3		
Sample C	Static Tube	9.8	3.12	1.30	95.3	61.1	56.4
	Froth Flotation	13.1	2.78		85.8		
Sample D	Static Tube	11.0	3.32	1.46	94.0	74.4	68.4
	Froth Flotation	19.9	2.84		85.3		

Based on the experimental work conducted it may be concluded that:

- o The plant effluents contain a considerable amount of coal that is recoverable at low ash and sulfur values.
- o Each effluent sample has certain unique characteristics that are attributable to the plant operating conditions.
- o One of the plant effluents contains some coarse material (+30 mesh) that needs to be ground, although in two cases over 70% of the material is minus 230 mesh in size.
- o The static tube process has achieved high Btu recoveries and pyritic sulfur rejection for all of the samples tested. At 90% Btu recovery, pyritic sulfur rejection varied between 68 and 78%. Higher rejection levels are achievable with a slight drop in Btu recovery.
- o It is possible to obtain a low-ash product (<5%) for three of the effluent samples. The lowest ash obtained for the fourth sample was 7.7% at a Btu recovery of 87%.
- o The static tube process has demonstrated the capability to provide a range of product ash values for each sample. This range, as would be expected, is coal-specific.
- o The collector dosages used in the process are in the 1.6-6 lb/ton range. Reagent optimization is expected to reduce the reagent requirements, and will be carried out in Phase II.
- o The static tube process achieved a superior performance to froth flotation when using identical grind sizes.

OBJECTIVES

The objectives of this project were to test and demonstrate the feasibility of recovering the coal fines that are currently disposed of with coal preparation plant effluent streams. The quality of the recovered coal should be such that it can be blended with the plant coarse clean coal. In addition, coal (or Btu) recovery should be sufficiently high that the process economics would encourage commercial application of this technology. This recovery was effected by means of Michigan Technological University's static tube flotation process, which had been successfully demonstrated on a number of raw coals to reject 85% of the pyritic sulfur and recover 90% of the combustible matter. Under this project, the process parameters for the technology were modified for this application in order to recover a low-ash, low-sulfur clean coal. Once the applicability of the process for the recovery of coal had been demonstrated at the bench scale for one effluent sample, three other effluent samples were tested successfully to demonstrate its applicability to a variety of effluents. Having achieved the Phase I objectives, the need to advance the process to the pilot scale, undertake product dewatering, and evaluate the product handling and utilization characteristics becomes apparent. These issues will be addressed in Phase II of the project.

INTRODUCTION AND BACKGROUND

The project, entitled "Recovery of Coal Fines from Preparation Plant Effluents," was undertaken jointly by Praxis Engineers, Inc., the Illinois State Geological Survey (ISGS), and Michigan Technological University (MTU), with Praxis acting as the prime contractor to the CRSC. The work distribution was based on the expertise of the three organizations: ISGS conducted the field sampling and basic characterization work; MTU provided its static tube flotation process and test facilities; and Praxis provided its expertise in process engineering, use of pyrite depressants, data management, and overall project coordination, management, and reporting.

This project addressed the recovery of coal fines from preparation plant effluent streams using the MTU static tube flotation process. It was anticipated that if clean coal could be recovered from plant effluent streams efficiently and economically, the value of the recovered coal would meet the costs of the recovery process. It would also partially offset the costs of dewatering the tailings and disposing of them as solid waste, as opposed to the current practice of ponding. Demonstration of the process as a low-cost method of recovering coal fines from effluents would also indicate that the technology could be applied to existing ponds as a means of reclamation.

Conventional froth flotation, which is typically used to clean coal at 28 mesh or finer, is not effective in terms of its selectivity in rejecting pyrite and its ability to treat very fine particles below 400 mesh in size. Major difficulties can be attributed to particle heterocoagulation induced by the strong surface forces of fine particles and the lack of froth drainage, which is an inherent problem in the design of nearly all flotation machines. The use of wash water in conventional flotation cells has resulted in lower ash, but the improvement in concentrate grade has often been accompanied by a drop in recovery

because of the limited froth bed height. Column flotation cells have been applied in many instances to help alleviate these difficulties, but they suffer from intrinsic problems of vertical mixing in the column that complicate the scaleup effort.

The static tube process overcomes the problems associated with conventional flotation methods by combining a concept of controlled slurry dispersion with innovative design of a very efficient flotation device. As a consequence, the range of coal particle sizes for effective flotation has been extended down to a few microns. Also, the effective volume of the machine is the major factor for scale-up considerations provided the unit cell geometry of packing elements does not change significantly.

The static tube process, shown schematically in Figure 1 (not to scale), incorporates a packed-bed column design to provide small flow passages for intimate particle-bubble contact. The packing used in the column is a stack of corrugated plates, as shown in the exploded view. The packing elements are arranged in blocks positioned at right angles to each other. These stacked corrugated plates obviate the need for a sparger, since they break any bubbles into small sizes. Also, the capillary effects between the plates enable an almost unlimited height of froth to be supported. The device has a pulp inlet at an intermediate location, a wash water inlet near the top, and an inlet that introduces compressed air into the bottom of the cell.

As air passes upward through the flow passages, it automatically breaks into fine bubbles which carry the coal particles into a froth compartment in the upper portion of the column, where the clean product is discharged through an outlet. Wash water descending through the same flow passages induces entrained mineral particles to separate from the clean product. By adjusting the machine operating parameters it is possible to obtain various degrees of separation.

EXPERIMENTAL PROCEDURES

In order to develop sound experimental procedures, a brief project work plan was developed by Praxis with input from MTU and ISGS, and a copy was submitted to CRSC. The work plan provided details on each task including the minimum quantities of slurry samples to be collected and the sampling and preparation procedure. A project communication procedure was also developed.

The project experimental procedure is summarized below:

- o All four effluent samples were collected and thickened using a uniform procedure.
- o Characterization work (film flotation and froth flotation) was done using a standard procedure.
- o Prior to conducting static tube flotation tests the samples were ground to a similar topsize to eliminate the effect of grind size (85% passing 200 mesh).

- o Flotation testing was done on one coal using a standard procedure, and a number of repeat tests were also conducted.
- o Some of the analytical work was repeated at commercial laboratories for confirmatory purposes.
- o Mass balances were calculated to reconcile the quality of the characterization and static tube testing samples for internal consistency.

TECHNICAL WORK COMPLETED

This section summarizes the experimental results obtained during the project.

Project Work Plan (Task 1)

Upon award of the contract, a project work plan was developed by Praxis, with input from ISGS and MTU. This document focused on the specific details of the tasks to be executed, which included the following:

- o Sample procurement and preparation
- o Characterization of test samples
- o MTU static tube flotation testing
- o Process evaluation criteria or efficiency indices to be applied
- o Project reports and other deliverables

Sample Procurement and Preparation (Task 2)

The following coal preparation plants were visited during the week beginning 22 October 1990 in order to collect samples of the plant effluents:

- o Turris Coal Company, Elkhart mine plant
- o Arch of Illinois, Captain mine plant
- o Peabody Coal Company, Randolph preparation plant
- o Consolidated Coal Company, Sesser preparation plant

The managements of all of the preparation plants listed above assisted in the sampling effort undertaken by ISGS. As a result, the sample collection work was completed in approximately two weeks rather than the three originally anticipated. In addition, samples were obtained from all four plants instead of three as originally proposed.

Three to five 55-gallons drums of effluent slurry were collected from each preparation plant during steady-state operation. Each plant sample was thickened by settling and decantation in the field to consolidate the sample following the detailed sampling procedure given in the work plan. For particle size analysis, about 5 gallons of material representative of each effluent sample was collected from the main sample and placed in plastic buckets. The main samples were shipped to MTU for static tube testing.

Upon receipt of the slurry samples at MTU, the drums were opened one at a time. Using a stirrer and pump, representative samples of the slurry were pulled from the drum and placed in 5-gallon buckets for testing. The sample remaining in the drum was sealed and stored as a reserve sample.

The effluent samples have been assigned codes (A, B, C, and D) in order to preserve the confidentiality of the mines participating in the study.

Characterization of Test Samples (Task 3)

The coal preparation plant waste (effluent) stream samples were characterized in order to obtain a clear understanding of their properties.

In run-of-mine coals, clays and other impurities tend to concentrate in the fines fraction. Therefore, the mineral matter content in the coal below a certain size tends to be very high and makes it difficult to achieve a good separation. For the characterization test work conducted under this project, both the whole sample and two fractions obtained by screening were used, i.e., +230 mesh US (63 microns) and -230 mesh. The screen size was selected based on our prior experience. For the static tube flotation tests, however, this size needed to be selected individually for each effluent sample.

Characterization tests conducted using the +230-mesh fraction consisted of washability analysis at 1.3, 1.4, 1.5, and 1.65 specific gravity and froth flotation. Film flotation and standard flotation tests were conducted using the 45 x 60 mesh fraction. Zeta potential tests were also conducted. The -230 mesh effluent samples were analyzed for mineralogical composition. The results of these tests are discussed in subsequent sections of this report.

Particle Size Analysis. The effluent samples were sieved using the wet method. The +230-mesh fraction was dried and a mechanically split sample was used for dry sieve analysis.

Table 1 presents a summary of the proportion of slimes in the four effluent samples and indicates their quality. Samples A and C have a similar slimes content of approximately 35-40%, while Samples B and D each contain over 79% slimes. For all samples, the ash content of the -230 mesh slimes is more than 65%, with the exception of Sample C for which the desliming size to obtain an ash content of 65% is 400 mesh. In general, slimes finer than 230 mesh tend to have little commercial value due to their high mineral matter content and can be discarded.

**TABLE 1
COMPOSITION OF THE FOUR EFFLUENT SAMPLES**

	Sample A			Sample B			Sample C			Sample D		
	+230M	-230M	Total	+230M	-230M	Total	+230M	-230M	Total	+230M	-230M	Total
Wt %	60.5	39.5	100.0	26.8	73.2	100.0	65.3	34.7	100.0	28.2	71.8	100.0
Ash %	39.2	65.5	49.6	18.1	67.4	54.2	37.0	58.0	44.3	29.3	73.2	60.8
TS %	2.56	0.64	1.80	3.74	1.91	2.40	5.63	1.79	4.30	5.64	1.96	3.00
PS %	2.17	0.61	1.56	1.86	1.31	1.46	4.24	1.12	3.16	4.17	1.57	2.30
Btu/lb	7,973	4,516	6,607	11,080	3,974	5,878	8,311	5,376	7,293	9,326	3,245	4,960

Size analysis of the +230 mesh (+63 micron) material was carried out by dry-screening for 10 minutes on a deck of sieves using a rctotap. The particle size distribution of the -230 mesh (-63 micron) slimes was determined by means of a Microtrac II analyzer. These distributions were composited to derive the overall size distribution of the effluent samples. These results are presented in Table 2 and plotted in Figure 2.

**TABLE 2
PARTICLE SIZE DISTRIBUTION FOR THE EFFLUENT SAMPLES**

Size (μ)	Sample A		Sample B		Sample C		Sample D	
	Wt, %	% Pass	Wt, %	% Pass	Wt, %	% Pass	Wt, %	% Pass
4760.0		100.0		100.0		100.0		100.0
1410.0	0.3	99.7	0.2	99.8	41.3	58.7	0.9	99.1
595.0	6.5	93.2	0.6	99.2	9.3	49.4	3.0	96.1
500.0	5.7	87.5	0.3	98.9	1.2	48.2	2.1	94.0
354.0	11.9	75.6	0.9	98.0	1.4	46.8	0.9	93.1
250.0	10.8	64.8	2.0	96.1	1.3	45.5	2.7	90.4
125.0	12.1	52.7	8.9	87.2	2.6	42.9	8.3	82.1
63.0	13.2	39.5	14.0	73.2	8.2	34.7	10.3	71.8
44.0	3.7	35.8	4.8	68.4	2.7	32.0	3.9	67.9
31.0	3.2	32.7	4.4	64.0	2.8	29.1	4.2	63.7
22.0	3.8	28.8	5.3	58.7	3.2	25.9	5.0	58.7
16.0	3.9	25.0	5.9	52.9	3.6	22.3	6.1	52.6
11.0	3.4	21.5	6.2	46.7	3.8	18.5	6.9	45.7
7.8	3.9	17.7	7.2	39.6	3.6	14.9	7.5	38.1
5.5	3.8	13.8	8.6	31.0	3.5	11.5	8.3	29.9
3.9	3.5	10.4	8.4	22.6	3.3	8.1	8.0	21.9
2.8	4.0	6.4	9.2	13.3	3.4	4.8	9.0	12.9
1.9	3.7	2.7	8.1	5.3	2.9	1.8	7.8	5.0
1.4	1.5	1.2	3.1	2.2	1.1	0.7	2.9	2.1
0.9	0.9	0.2	1.8	0.4	0.6	0.1	1.7	0.4
0.0	0.2	0.0	0.4	0.0	0.1	0.0	0.4	0.0

The results indicate that Samples B and D are very fine in nature and have a very similar size distribution, with over 90% of the material below 60 mesh. Sample A is considerably

coarser than Samples B and D and contains a larger amount of material in the middle size range. Sample C contains over 51% material in the coarse range (+30 mesh) and is not suitable for flotation in this form. This sample therefore needed to be comminuted prior to conducting flotation tests.

It is also evident that some desliming (say at 230 mesh) can be done in order to reduce the volume of pulp for processing without any significant loss of coal.

Washability by Sink-Float Analysis. Sink-float separation at a given specific gravity is typically carried out to determine the quality and quantity of the product that may be recovered if the processing device has been set to separate the feed at that gravity setting. Since the presence of clayey fines can interfere with sink-float separation, the samples were deslimed at 230 mesh (63 micron) and the tests were conducted only on the +230 mesh fraction of the dried effluent sample. A centrifuge was used to accelerate the separation of the sink and float phases during these tests. These tests were carried out using a procedure similar to that developed by Praxis for a DOE project. This procedure is summarized below:

- o Adjust the specific gravity of the polytungstate solution (heavy medium) with a calibrated hydrometer.
- o Add a small quantity of coal to the solution in the centrifugal tube.
- o Stir the solids to liberate trapped material.
- o Centrifuge the mixture for 20 minutes or until a clear separation of the float and sink material is achieved.
- o Remove and wash the float material to remove traces of the heavy medium.
- o Filter the heavy medium to recover the sink material and wash the filtrate to remove any adhering heavy medium material.
- o Dry, weigh, and process the float and sink materials for analysis.

Btu, ash, total sulfur, and pyritic sulfur analyses were performed on all of the gravity fractions using ASTM standard procedures, as specified in ASTM D 2015, D 3174, D 3177, and D 2492, respectively. The results of these tests are presented in Tables 3 through 6.

Figure 3 shows the cumulative weight percent floated versus the specific gravity of the test liquid for the four effluent samples. As expected, there is a gradual increase in the weight of the float with increasing specific gravity. The sink-float results show that about 42% of the material in Samples A and C contains particles heavier than 1.6 specific gravity, which represents high-ash shale. Sample B contains a significant amount of liberated (low-ash) coal matter, as is evident in the high amount of 1.30 float material

TABLE 3
RESULTS OF SINK-FLOAT TESTS FOR EFFLUENT SAMPLE A
(+230 Mesh Size Fraction)

ELEMENTARY DATA							COMPUTED DATA: CUMULATIVE								
SPEC GRAV	(%)	(%)	(%)	(%)		# SO2/	(%)	(%)	(%)	(%)		(%)	# SO2/	%PS	
SINK FLOAT	WT%	ASH	TS	PS	BTU	M BTU	WT%	ASH	TS	PS	BTU	BTU REC	M BTU	REJ	
	1.30	39.60	3.33	0.93	0.32	13,688	1.36	39.60	3.33	0.93	0.32	13,688	67.99	1.36	94.17
1.30	1.40	12.88	9.29	1.22	0.65	12,746	1.91	52.48	4.79	1.00	0.40	13,457	88.58	1.49	90.31
1.40	1.50	3.66	23.83	1.59	1.11	10,566	3.01	56.14	6.03	1.04	0.45	13,268	93.43	1.57	88.44
1.50	1.60	1.83	52.23	1.45	1.15	6,236	4.65	57.97	7.49	1.05	0.47	13,046	94.86	1.61	87.47
1.60		42.04	83.03	4.63	4.52	977	94.78	100.01	39.25	2.56	2.17	7,973	100.01	6.41	0.00

TABLE 4
RESULTS OF SINK-FLOAT TESTS FOR EFFLUENT SAMPLE B
(+230 Mesh Size Fraction)

ELEMENTARY DATA							COMPUTED DATA: CUMULATIVE								
SPEC GRAV	(%)	(%)	(%)	(%)		# SO2/	(%)	(%)	(%)	(%)		(%)	# SO2/	%PS	
SINK FLOAT	WT%	ASH	TS	PS	BTU	M BTU	WT%	ASH	TS	PS	BTU	BTU REC	M BTU	REJ	
	1.30	60.88	3.98	2.72	0.34	13,472	4.04	60.88	3.98	2.72	0.34	13,472	74.02	4.04	88.86
1.30	1.40	15.53	14.74	3.16	1.36	11,860	5.33	76.41	6.17	2.81	0.55	13,144	90.65	4.27	77.49
1.40	1.50	4.76	27.91	3.57	2.19	9,780	7.30	81.17	7.44	2.85	0.64	12,947	94.85	4.41	71.87
1.50	1.60	1.85	38.43	4.79	3.94	7,937	12.07	83.02	8.13	2.90	0.72	12,835	96.17	4.51	67.95
1.60		16.99	66.66	7.88	7.43	2,501	63.01	100.01	18.08	3.74	1.86	11,080	100.01	6.76	0.00

TABLE 5
RESULTS OF SINK-FLOAT TESTS FOR EFFLUENT SAMPLE C
(+230 Mesh Size Fraction)

ELEMENTARY DATA							COMPUTED DATA: CUMULATIVE								
SPEC GRAV	(%)	(%)	(%)	(%)		# SO2/	(%)	(%)	(%)	(%)		(%)	# SO2/	%PS	
SINK FLOAT	WT%	ASH	TS	PS	BTU	M BTU	WT%	ASH	TS	PS	BTU	BTU REC	M BTU	REJ	
	1.30	29.51	5.05	2.65	0.55	13,374	3.96	29.51	5.05	2.65	0.55	13,374	47.49	3.96	96.18
1.30	1.40	17.45	12.46	3.03	1.10	12,254	4.95	46.96	7.80	2.79	0.75	12,958	73.21	4.31	91.65
1.40	1.50	3.67	24.18	5.00	3.33	10,339	9.67	50.63	8.99	2.95	0.94	12,768	77.78	4.62	88.77
1.50	1.60	4.20	31.36	5.72	4.21	9,257	12.36	54.83	10.70	3.16	1.19	12,499	82.46	5.06	84.61
1.60		45.16	68.99	8.63	7.95	3,227	53.49	99.99	37.03	5.63	4.24	8,311	99.99	13.55	0.01

(61%). The other samples have about 30-40% material in the 1.30 gravity fraction. The ash content of the 1.3 fraction is also low for these samples, varying between 3 and 5%.

Figure 4 is a plot of ash versus Btu recovery for the four effluent samples and indicates a high degree of ash liberation for Samples A and B. These two effluent samples would yield a product with an ash content of 5-6% at 90% Btu recovery. At the same Btu

TABLE 6
RESULTS OF SINK-FLOAT TESTS FOR EFFLUENT SAMPLE D
(+ 230 Mesh Size Fraction)

ELEMENTARY DATA							COMPUTED DATA: CUMULATIVE								
SPEC GRAV	(%)	(%)	(%)	(%)	# S02/	(%)	(%)	(%)	(%)	(%)	# S02/	%PS			
SINK FLOAT	WT%	ASH	TS	PS	BTU	M BTU	WT%	ASH	TS	PS	BTU	BTU REC	M BTU	REJ	
	1.30	36.53	5.21	2.37	0.40	13,307	3.56	36.53	5.21	2.37	0.40	13,307	52.12	3.56	96.49
1.30	1.40	24.68	13.18	3.02	1.21	12,121	4.98	61.21	8.42	2.63	0.73	12,829	84.20	4.10	89.33
1.40	1.50	6.15	20.08	3.97	2.31	11,014	7.21	67.36	9.49	2.75	0.87	12,663	91.46	4.35	85.92
1.50	1.60	3.84	56.41	10.93	10.02	4,698	46.53	71.20	12.02	3.20	1.36	12,234	93.39	5.22	76.69
1.60		28.79	72.14	11.68	11.10	2,137	109.31	99.99	29.33	5.64	4.17	9,326	99.99	12.09	0.01

recovery level, Sample D would yield a product with an ash content of about 8%. Ash liberation for Sample C is relatively poor, with an ash content of about 10% and a maximum Btu recovery of about 80%.

Figure 5 is a plot of pyrite rejection versus Btu recovery. Sample A has the highest liberation of pyrites, with 38% rejection at 90% Btu recovery. At about 90% Btu recovery, pyrite rejection for Sample D would be about 87%, and that of Sample B will be slightly lower at about 77%. At the same Btu recovery, Sample C will have a pyrite rejection of about 50%.

The washability data analysis of the effluent streams provided an excellent picture of the cleanability of the four effluent samples. These results indicated the following:

- o Sample A contains a low ash (3.3%) and low total sulfur (0.93%) material at 1.3 specific gravity and allows a high pyritic sulfur rejection. This sample contains a smaller quantity of recoverable coal than the other samples but the Btu recovery, which is a measure of the recovery efficiency, is high.
- o Sample B contains the highest quantity of coal containing low ash (4%) and medium sulfur (2.72%) recoverable at 1.3 specific gravity. Recovery at 1.6 specific gravity is 83%, with 8% ash and 2.9% total sulfur. The organic sulfur content (~2%, calculated by difference) of this coal is very high, as is typical of Illinois No. 6 coal, but the pyritic sulfur is in a reasonably liberated form. High weight recovery is possible from this effluent at low ash levels.
- o The recoverable coal in Sample C is low in quantity but high in quality. Pyritic sulfur rejection is not very high primarily due to the coarse nature of the material. This sample needs to be comminuted prior to further testing to improve liberation.
- o Sample D yielded a coal with a slightly higher ash content than the other samples at comparable specific gravities of separation.

While its total sulfur content is high, good pyritic sulfur rejection is possible during cleaning.

Film Flotation Tests. Coal floats on water because it is hydrophobic, i.e., it is not wetted by water. Coal is, however, wetted by other hydrophobic liquids such as alcohol. Coal can be transferred to the aqueous phase by altering the surface properties of the water, particularly its surface tension, by the addition of alcohols or surfactants to the water. The amount of coal transferred to the aqueous phase as a function of the surface tension of the aqueous phase is a measure of its hydrophobicity; film flotation studies quantify the hydrophobicity of coal.

The wetting characteristics of the effluent samples were assessed using the film flotation technique developed by Fuerstenau and colleagues¹. The 45 x 60 mesh size fractions of the effluent samples were used for these tests. In this procedure, a known quantity of coal is added to mixtures of ethanol and water in varying proportions. By varying the proportions of ethanol and water in the mixture it is possible to control the surface tension of the mixture. The weight of the coal that is transferred to the aqueous phase and that of the coal that floats is measured. The weight percent of the float is plotted against the surface tension of the ethanol-water mixtures to obtain the critical wetting tension diagrams.

Table 7 presents a summary of the test results. The critical wetting tension diagrams obtained for the four samples tested, shown in Figure 6, are generally similar. However, Samples C and D are more hydrophilic (easily wettable) than Samples A and B; the amount of coal transferred to the aqueous phase is 7 and 12% for Samples A and B respectively and 15 and 19% for sample C and D respectively. In general, the hydrophobicity of the four samples tested, in decreasing order, is A, B, C and D. These results are consistent with the standard flotation test results presented later in this report. Since the floatability of a coal is a function of its hydrophobicity, this information was very useful during subsequent flotation testing.

Standard Flotation Tests. A standard test developed at the University of California, Berkeley², for a Department of Energy project was used as a model for the procedure with a few exceptions that are identified in this section. These tests were run on the 45 x 60 mesh size fraction of the effluent samples. For each flotation test, about 120 g of effluent solids was used as the flotation feed. Water was added to the cell and a pulping time of 4 minutes was used. Following pulping, 100 μ l (1.32 lb/T) of dodecane was added as a collector and a conditioning time of 2 minutes was allowed. In addition, 100 μ l (1.32 lb/T) of frother (MIBC) was used and a 3-minute conditioning time was allowed. Other frothers (identified as A, B, C, D, E, and F) were used at a dosage of 100

¹Fuerstenau, D. W., and M. C. Williams. 1987. "Characterization of the Lyophobicity of Particles by Film Flotation." Colloids and Surfaces, 22: 87-91.

²Fuerstenau, D. W., V. Choudhry et al. 1990. Coal Surface Control for Fine Coal Flotation. Annual Report No. 1 for DOE Project No. DE-AC22-88PC88878. Chapter 6: Development of a Standard Flotation Test. University of California, Berkeley.

TABLE 7
FILM FLOTATION TEST RESULTS FOR EFFLUENT SAMPLES
(45 X 60 Mesh Size Fraction)

Surface Tension (mN/m)	Weight Percent Floated			
	Sample A	Sample B	Sample C	Sample D
72.0	92.9	88.0	84.0	79.8
57.5	90.7	61.9	65.9	45.3
51.0	60.0	49.5	37.8	37.1
38.8	10.1	18.0	8.4	14.0
34.0	1.0	6.1	3.1	5.3
30.0	0.0	5.0	2.0	3.2
27.0	0.0	2.0	1.0	1.1
22.5	0.0	0.0	0.0	0.0

μ l (about 1.32 lb/T). Natural air flow by suction was allowed at an impeller speed of 1200 rpm for these tests. The coal recovery from the 45 x 60 mesh fraction for the four effluent samples was:

Sample A	120.17 g	Sample B	71.92 g
Sample C	30.95 g	Sample D	7.82 g

Thus, coal recovery for the four effluent samples, in decreasing order, is A, B, C and D, which is consistent with the results obtained from the film flotation tests.

Zeta Potential. The surface charge of the effluent samples was measured with a zeta meter. The effluent samples were conditioned in distilled water at various pH levels for 24 hours before measuring the surface charge. The surface charge values reported are averages of at least twenty measurements.

The surface charge values of the four effluent samples are very similar, as is evident from Figure 7 which presents a plot of the surface charge values at different pH values. Also plotted in the figure for purposes of comparison are the results for a sample of -200 mesh raw Illinois No. 6 coal in 2×10^{-3} KNO₃, obtained from UC Berkeley's DOE project on "Coal Surface Control For Advanced Fine Coal Flotation." The two sets of results are very similar. However, the zeta potential drop is less drastic for the subject project than for the DOE project.

Coal surface charge measurement is useful since it enables the selection of collectors for selective flotation at various pH operating ranges. As the four effluent samples exhibited similar zeta potentials, it was determined that the same frother and collector could be used to process the four effluent samples.

Mineralogical Composition. The mineralogical composition of the -230 mesh effluent samples was determined as part of the sample characterization work. Each sample was run in three modes: an ethylene-solvated oriented aggregate, a heated (about 300°C)

oriented aggregate, and a random powder pack of the whole sample. The peaks obtained from these modes were analyzed by a deconvolution program that determines the position and area of these peaks. The computed peak areas were then used to determine the weight percentage of the minerals present in the samples, using a procedure (Method III in Table 1) outlined by Hughes and Warren³. Precision for the measurement of the peak area is $\pm 2.4\%$ based on ten repeat peak area measurements.

Table 8 presents the mineralogical composition of the four effluent samples. From the table it can be concluded that the mineralogy of the four effluent samples is essentially identical. The percentage of the total clay minerals varies from 68% for Sample A to 79% for Sample D. The predominating non-clay mineral in the four effluent samples is quartz and its content varies between 16 and 21%.

TABLE 8
MINERALOGICAL COMPOSITION OF THE FOUR EFFLUENT SAMPLES

Sample	% Qtz	% KFeld	% Plag	% Calc	% Pyr	% CM	% Exp	% Ill	% Kao	% Chl	% I in I/S
A	16	0.6	1	2	0.0	80	77	18	5	0	75
B	19	0.0	3	2	0.0	76	68	23	6	2	82
C	21	0.0	2	1	1.9	73	76	19	3	2	78
D	16	0.3	1	2	1.6	79	79	16	4	1	58

Qtz	-	Quartz	KFeld	-	Potassium feldspar
Plag	-	Plagioclase	Calc	-	Calcite
Pyr	-	Pyrite	CM	-	Total clay mineral
Exp	-	Expandable clays	Ill	-	Illite
Kao	-	Kaolinite	Chl	-	Chlorite
I in I/S	-	Illite in the mixed layered clay mineral illite/smectite			

Froth Flotation Studies Detailed froth flotation tests were conducted on one of the effluent samples in order to:

- o Test the applicability of froth flotation to treat the effluents for coal recovery
- o Test the effectiveness of a pyrite depressant in treating effluent streams

³ Hughes, R. E. and R. L. Warren. 1989. "Evaluation of the Economic Usefulness of Earth Materials by X-Ray Diffraction." In Proceedings of the 23rd Forum on the Geology of Industrial Minerals, eds. Hughes and Bradbury, Illinois State Geological Survey. IMN102.

- o Generate data for comparison with the performance of the static tube flotation test results.

A deslimed (+230 mesh) fraction of Sample A was used for this study following comminution to about 80% passing 200 mesh. Grinding was accomplished using ISGS's Akbar mill. The grind size distribution is given in Figure 8.

The procedure used for these tests is similar to that outlined in the section on standard flotation with a few exceptions that are identified in this section. Commercial frothers (identified as A, B, C, D, E, and F) were used at a dosage of 100 μ l (1.32 lb/T). Oxone was used as a pyrite depressant in one of the tests at a dosage of 0.125 g (2.0 lb/T). The results of these tests are given in Table 9.

TABLE 9
RESULTS OF STANDARD FROTH FLOTATION TESTING
Using Deslimed +230 Mesh Material From Sample A
Comminuted to 75% Passing 200 Mesh

Flotation Conditions		Product Analysis				% Rejection	
Collector	Frother	Yield	CMR	Ash%	TotS%	Ash	Tot S
100 μ l	Std Test	41.9	64.1	9.9	1.29	89.9	80.6
100 μ l	A-100 μ l	38.5	58.8	10.1	1.34	90.5	81.4
100 μ l	B-100 μ l	59.5	86.3	14.6	1.84	78.9	60.6
100 μ l	C-100 μ l	46.1	68.7	12.2	1.45	86.3	75.9
100 μ l	D-100 μ l	55.8	81.6	14.0	1.74	81.0	65.1
100 μ l	E-100 μ l	44.1	65.4	12.8	1.54	86.3	75.5
100 μ l	F-100 μ l	47.2	69.9	12.9	1.50	85.2	74.6
100 μ l	Oxone	33.3	50.5	10.6	1.25	91.4	85.0
100 μ l	D-Reclean	38.3	59.7	8.3	1.55	92.3	78.6

Froth flotation tests were also conducted on the four effluent samples at test conditions similar to those used for the static tube in order to evaluate the performance of the static tube. The results of these tests are summarized in Table 10.

These results indicate that though a high yield is obtained from froth flotation the product quality is poor since it has high ash and sulfur contents. These results are discussed in greater detail in a later section of this report.

MTU Static Tube Flotation Testing (Task 4)

The static tube equipment at MTU was commissioned and made ready for use in this project, and all of the effluent samples collected from the preparation plants were tested for recovery of coal fines. The design features and the stages involved in the testing process are as follows:

TABLE 10
FROTH FLOTATION RESULTS FOR THE FOUR EFFLUENT SAMPLES

Sample	Feed (Calc)		Product Analysis			Tails	Performance		
	% Ash	% Tot S	% Yield	% Ash	% Tot S	% Ash	% CMR	% Ash Rej	% TS Rej
A	35.5	2.58	68.3	12.7	3.10	84.6	92.5	75.5	17.7
B	52.7	2.31	50.9	19.9	3.16	86.8	86.3	80.8	30.1
C	34.8	2.69	64.4	13.1	2.78	74.0	85.8	75.7	33.5
D	56.9	2.31	45.9	19.9	2.84	88.3	85.3	84.0	43.4

- o The first stage of conditioning allows use of a pyrite depressant.
- o Collector and frother are introduced in the second stage of conditioning.
- o Conditioning at both stages is carried out in a loop, thus ensuring full conditioning of the feed with almost no bypass of feed from the conditioner to the next stage.
- o The MTU flotation process, consisting of a packed static tube, creates intimate particle-bubble contact, thus preventing the problem of vertical mixing that is commonly encountered with column flotation.
- o The mechanical design of the MTU cell is considerably simplified by the elimination of the air sparger, as air can be introduced through a tube.
- o The feed is introduced in the center of the cell, and the air is introduced at the bottom. This design allows countercurrent flow, resulting in a high degree of selectivity, as shown by the test results.
- o The quality of the product is controlled by manipulating the froth-pulp interface.

The general range for the operating parameters used in the static tube system is:

Static tube size:	4.6 cm ID x 244 cm tall
Feed rate:	16 g/min (or 1 g/min/cm ²)
Pulp density:	8% solids
Reagent range:	Diesel (4-6 lb/T) + pine oil (2-3 lb/T)
Air:	6-600 ml/min/cm ²
Wash water:	2-10 ml/min/cm ²
Pulp level:	50-100 cm from the bottom

The products at the concentrate and tailings outlets were sampled after steady-state operation was reached. The machine operating variables were then changed and the system was allowed to equilibrate. A portion of the grade-recovery curve was thus established in a test series. A slight change in the reagent dosage (e.g., the frother) can shift the data point along the grade-recovery curve, depending on the requirements of a specific system. The test conditions used are summarized in Table 11.

TABLE 11
OPERATING CONDITIONS OF THE LABORATORY STATIC TUBE FLOTATION TESTS
Plant Effluent Samples

Sample Source	Test No.	Grind		Reagent, lb/T		Air l/min	Water ml/min	Pulp Level cm
		Time, min	%-200M	Diesel	Pine Oil			
A	1	5	81.7	3.2	1.8	2.1	110	64
	2			4.4	2.4	2.1	110	51
	3			4.4	2.4	2.1	110	55
	4			4.4	1.6	3.2	98	76
	5			6.6	3.6	3.2	98	76
	6			3.2	1.8	6.0	48	89
B	1	1	84.8	4.2	2.8	2.1	110	51
	2			3.2	4.0	2.1	110	51
	3			4.2	2.8	0.9	135	51
	4			2.0	2.4	3.2	135	64
	5			1.6	6.4	2.1	160	51
	6			3.2	9.4	3.2	160	51
	7			2.0	9.4	4.7	110	83
	8			3.2	9.4	4.7	85	102
C	1	10	93.3	6.0	4.0	4.7	135	89
	2			6.0	4.0	3.2	135	102
	3			6.0	4.0	7.6	60	102
D	1	5	94.8	5.2	3.6	1.2	110	64
	2			5.2	3.6	1.2	160	51
	3			5.2	3.6	1.2	110	51
	4			5.2	3.6	2.6	110	70
	5			5.2	3.6	3.2	110	89

The results of the tests conducted on the four effluent samples using the static tube are summarized in Tables 12 through 15. Figure 9 is a plot of Btu recovery versus weight percent ash of the product obtained from the static tube for the effluent streams. As expected, the best results were obtained with Sample A, followed by Samples B and C, which is consistent with the sink-float analyses and froth and film flotation test results. Sample D yielded high Btu recoveries but at a slightly higher ash content.

TABLE 12
BENCH-SCALE TEST RESULTS USING THE MTU PROCESS
Effluent Sample A

Feed (Calculated)			Product Analysis				Tails	Performance		
%	%	%	%	%	%	%	%	% Ash	%PyS	%Btu
Ash	Tot S	PyS	Yield	Ash	Tot S	PyS	Ash	Rej	Rej	Rec
46.5	1.42	1.07	37.3	2.66	0.91	0.30	72.5	97.9	89.1	66.8
44.9	1.40	1.03	39.9	2.99	0.92	0.31	72.8	97.3	87.8	69.5
43.9	1.41	1.04	42.8	3.77	0.97	0.36	73.9	96.3	85.2	76.1
43.0	1.49	1.09	57.0	6.51	1.13	0.50	91.4	91.4	74.1	99.6
44.5	1.46	1.11	57.8	7.53	1.06	0.51	92.9	90.4	73.6	97.7
42.2	1.39	1.04	60.1	9.45	1.00	0.46	92.9	87.7	74.0	99.8

TABLE 13
BENCH-SCALE TEST RESULTS USING THE MTU PROCESS
Effluent Sample B

Feed (Calculated)			Product Analysis				Tails	Performance		
%	%	%	%	%	%	%	%	% Ash	%PyS	%Btu
Ash	Tot S	PyS	Yield	Ash	Tot S	PyS	Ash	Rej	Rej	Rec
51.7	3.43	1.82	40.7	4.88	2.98	1.00	83.8	96.2	74.6	82.4
51.6	3.44	1.83	41.2	5.67	2.99	1.05	83.8	95.5	76.4	82.5
53.8	3.49	1.88	42.7	6.32	3.07	1.11	89.2	95.0	74.6	83.8
50.3	3.49	1.91	48.3	8.54	3.15	1.27	89.3	91.8	67.6	91.2
50.1	3.55	1.97	49.0	8.74	3.28	1.41	89.8	91.5	64.8	93.9
50.0	3.56	1.94	49.4	8.87	3.34	1.36	90.1	91.2	65.5	94.5
48.8	3.55	1.93	51.8	10.38	3.32	1.40	90.1	89.0	62.2	96.6
47.7	3.57	2.00	55.1	12.86	3.36	1.58	90.5	85.2	56.8	99.4

Data Evaluation (Task 5)

Washability tests were conducted to provide information regarding the quantity and quality of the coal matter in the effluent samples. The ash-Btu recovery and pyrite-Btu recovery curves indicate that additional coal/pyrite and coal/ash liberation can be achieved by reducing the particle size, particularly for Samples C and D. The effluent samples were therefore comminuted to a target grind of 85% passing 200 mesh. While this grind size is commonly used for research, the actual grind will need to be selected for each sample depending on the quality requirements for the recovered coal.

Washability tests for the effluent samples were conducted using the as-received +230-mesh fraction. These test results serve as a guideline for the standard flotation and static tube flotation tests which were conducted on the effluent samples after grinding.

TABLE 14
BENCH-SCALE TEST RESULTS USING THE MTU PROCESS
Effluent Sample C

<u>Feed (Calculated)</u>			<u>Product Analysis</u>				<u>Tails</u>	<u>Performance</u>		
<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>% Ash</u>	<u>%PyS</u>	<u>%Btu</u>
<u>Ash</u>	<u>Tot S</u>	<u>PyS</u>	<u>Yield</u>	<u>Ash</u>	<u>Tot S</u>	<u>PyS</u>	<u>Ash</u>	<u>Rej</u>	<u>Rej</u>	<u>Rec</u>
51.9	2.56	1.68	43.7	4.39	2.86	0.91	88.7	96.3	76.4	88.8
48.2	2.50	1.66	49.0	5.18	3.03	0.99	89.6	94.8	70.7	91.0
50.0	2.55	1.68	50.2	9.78	3.12	1.30	92.3	91.9	61.1	95.3

TABLE 15
BENCH-SCALE TEST RESULTS USING THE MTU PROCESS
Effluent Sample D

<u>Feed (Calculated)</u>			<u>Product Analysis</u>				<u>Tails</u>	<u>Performance</u>		
<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>%</u>	<u>% Ash</u>	<u>%PyS</u>	<u>%Btu</u>
<u>Ash</u>	<u>Tot S</u>	<u>PyS</u>	<u>Yield</u>	<u>Ash</u>	<u>Tot S</u>	<u>PyS</u>	<u>Ash</u>	<u>Rej</u>	<u>Rej</u>	<u>Rec</u>
61.8	3.68	2.31	35.4	7.69	3.20	1.50	91.4	95.6	76.9	86.9
60.9	3.69	2.29	36.6	7.79	3.19	1.35	91.6	95.4	77.9	89.7
59.9	3.70	2.28	38.3	8.89	3.30	1.41	91.5	94.3	75.8	90.5
59.5	3.81	2.28	39.9	11.02	3.32	1.46	91.6	92.6	74.4	94.0
59.3	3.70	2.31	41.9	13.83	3.37	1.60	92.2	90.3	71.2	97.2

TABLE 16
QUALITY OF 1.60 FLOAT FOR EFFLUENT SAMPLES
(+230 Mesh Size Fraction)

<u>Effluent</u> <u>Sample</u>	<u>Feed (Calculated)</u>				<u>1.60 Float</u>				
	<u>Ash %</u>	<u>TS %</u>	<u>PS %</u>	<u>Btu/lb</u>	<u>% Yield</u>	<u>Ash %</u>	<u>TS %</u>	<u>PS %</u>	<u>Btu/lb</u>
A	39.2	2.56	2.17	7,973	58.0	7.5	1.05	0.47	13,046
B	18.1	3.74	1.86	11,080	83.0	8.1	2.90	0.72	12,835
C	37.0	5.63	4.24	8,311	54.8	10.7	3.16	1.19	12,499
D	29.3	5.64	4.17	9,326	71.2	12.0	3.20	1.36	12,234

Typically, the floats at 1.6 specific gravity provide an indication of the quality achievable for the clean coal product. The 1.6 float data for the +230-mesh fraction of each effluent sample are presented in Table 16. These values were reconstituted for the entire 4 mesh x 0 effluent sample and are presented in Table 17. In this calculation, the -230 mesh slime fraction was treated as the sink at 1.6 specific gravity since its ash content is in the same range as the 1.6 sinks from the sink-float tests.

TABLE 17
RECONSTITUTED QUALITY OF 1.60 FLOATS

Effluent Sample	1.60 Floats					Performance			
	Yield %	Ash %	TS %	PS %	Btu/lb	Btu Rec	Ash Rej	TS Rej	PS Rej
A	35.1	7.5	1.05	0.47	13,046	69.3	94.7	79.5	89.4
B	22.3	8.1	2.90	0.72	12,835	48.6	96.7	73.2	89.1
C	35.8	10.7	3.16	1.19	12,499	61.4	91.4	73.7	86.5
D	20.1	12.0	3.20	1.36	12,234	49.5	96.0	78.6	88.1

Note: -230 mesh material added to 1.60 sink fraction

The reconstituted float at 1.6 specific gravity provides a realistic value for the yield and Btu recovery for the entire, uncrushed sample. As stated above, this separation performance can be further improved if the samples are comminuted.

The static tube flotation performance was compared with that of the froth flotation tests as well as the plant clean coal quality requirements. The typical clean coal ash content at the plants that were sampled is in the 8-10% range.

Table 18 provides a summary of the results, with the best overall performance measured by the efficiency index as defined below:

$$\text{Efficiency index} = \text{Btu recovery \%} + \text{pyritic sulfur rejection \%} \text{ minus } 100$$

TABLE 18
COMPARISON OF STATIC TUBE PERFORMANCE WITH FROTH FLOTATION

Source	Procedure	Product Analysis			Performance		
		Ash%	TotS%	PyS%	%Btu Rec	%PyS Rej	Eff Ind
Sample A	Static Tube	7.5	1.06	0.51	97.7	73.6	71.3
	Froth Flotation	12.7	3.10		92.5		
Sample B	Static Tube	8.5	3.15	1.2	91.2	67.6	58.8
	Froth Flotation	19.9	3.16		86.3		
Sample C	Static Tube	9.8	3.12	1.30	95.3	61.1	56.4
	Froth Flotation	13.1	2.78		85.8		
Sample D	Static Tube	11.0	3.32	1.46	94.0	74.4	68.4
	Froth Flotation	19.9	2.84		85.3		

The efficiency index is a measure of process performance, defined as maximum Btu recovery at the highest pyritic sulfur rejection. A clean coal product containing lower ash or sulfur is easily achieved, as indicated in Tables 12 to 15, but at a slightly lower efficiency.

The Btu recovery for all of the samples tested was high, ranging between 89 and 99%, and the pyritic sulfur rejection ranged between 66 and 76%. Sample A, which had the highest liberation potential in the washability tests, had the highest efficiency at 73.7, at a high Btu recovery. Sample B had a high Btu recovery but slightly lower pyritic sulfur rejection. Additional liberation was achieved in the case of Sample C by grinding, which resulted in a higher Btu recovery at a lower product ash and sulfur. Sample D static tube performance was identical to that indicated by the coarse washability. The ash content of the clean coal for this sample, which is in the 8-12% range for the washability tests, can be lowered further during static tube processing by additional size reduction.

CONCLUSIONS AND RECOMMENDATIONS

A substantial amount of work was completed during the fourth quarter of the project pertaining to sample characterization and static tube testing on all four effluent samples. The excellent performance of the static tube in recovering coal from the effluent samples is indicated by the high weight and Btu recoveries achieved at high pyritic sulfur rejection levels. The process has also demonstrated the capability of providing a clean coal product at either 8-10% ash, which is compatible with the plant clean coals, or a low ash product (<5%) that may be used to improve the overall plant clean coal quality. The reagent dosages required by the process are comparable to those used for slow-floating coals from the Southern Illinois region. Reagent optimization, which will be carried out in Phase II, is expected to reduce reagent dosages. All of the effluent samples contain some coarse material which needs to be comminuted to achieve better liberation.

The results of the experimental work provide an excellent basis for consideration of advancing the study to the continuous pilot-scale level, and treatment of:

- o Plant effluent streams
- o Material obtained from abandoned tailings ponds
- o The entire 28 mesh x 0 fines fraction at the preparation plant

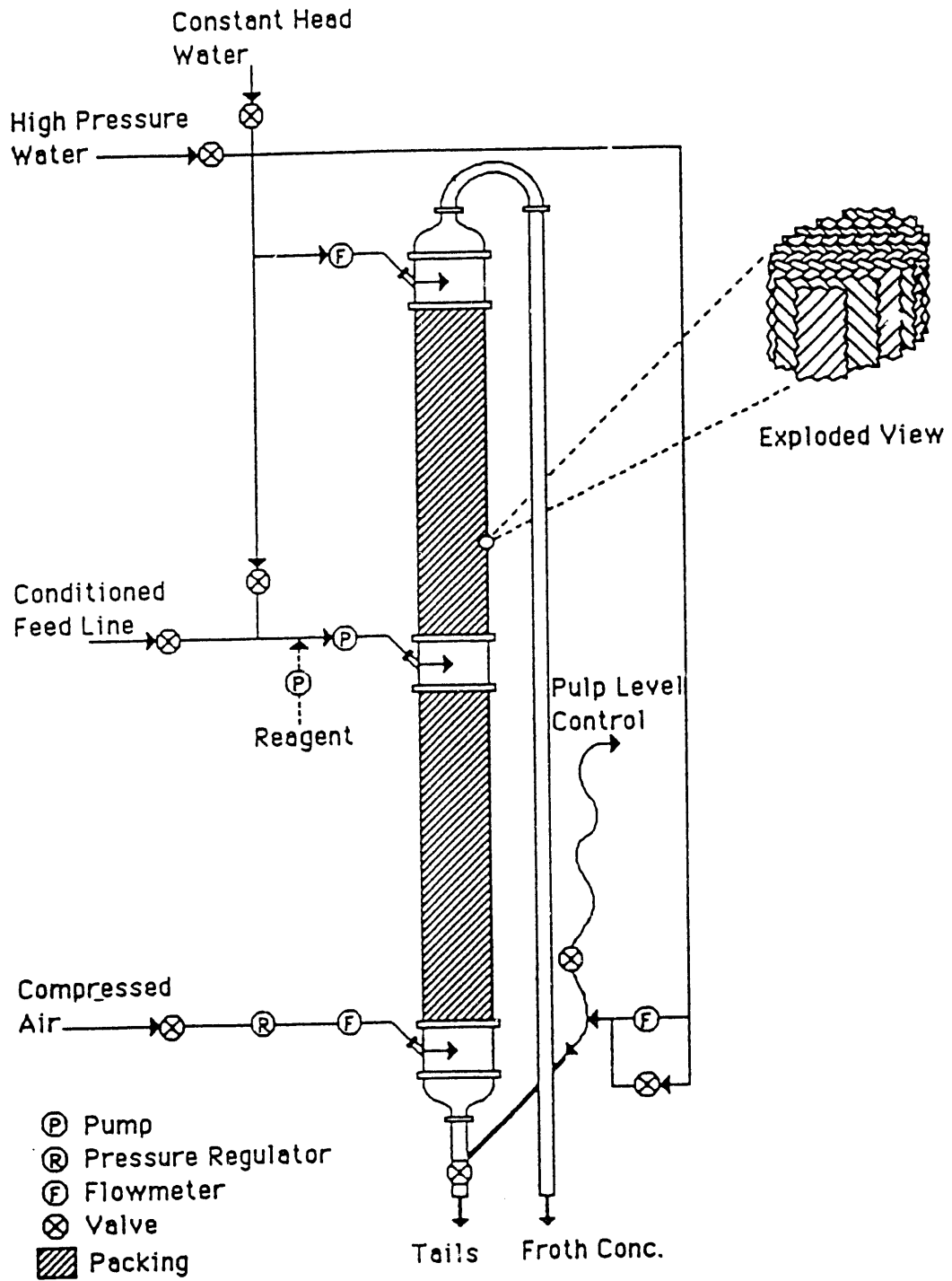


Figure 1: A Schematic Diagram of the Static Tube Flotation Machine

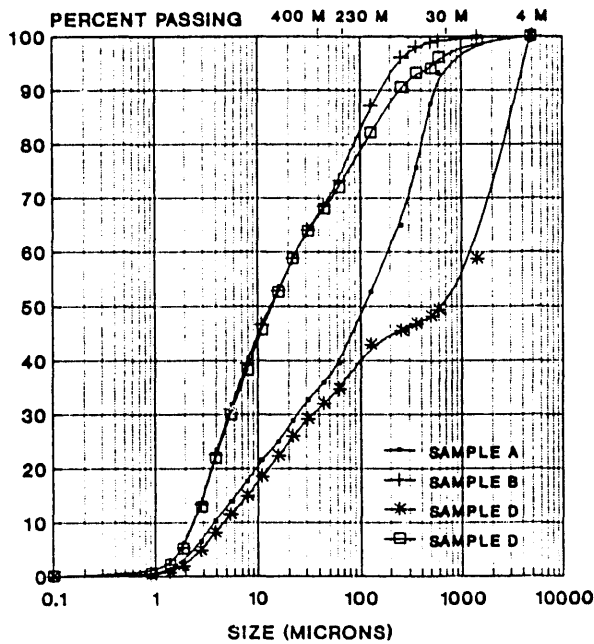


Figure 2: Particle size distribution for the four effluent samples

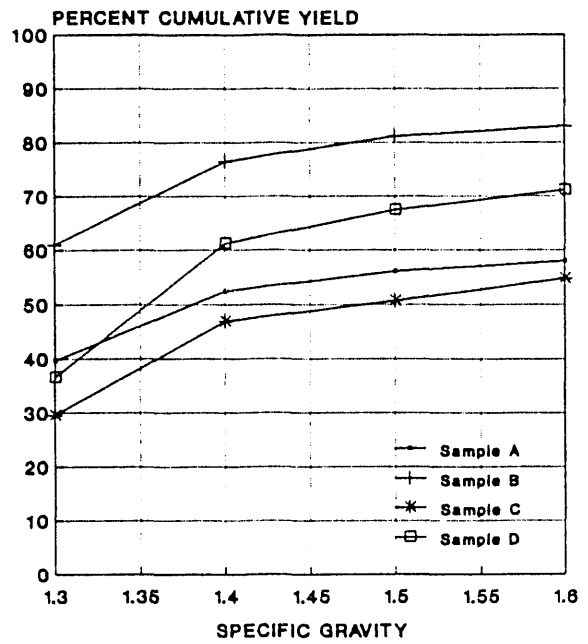


Figure 3: Cumulative weight percent (yield) float at various specific gravities for +230 M fraction of the four effluent samples.

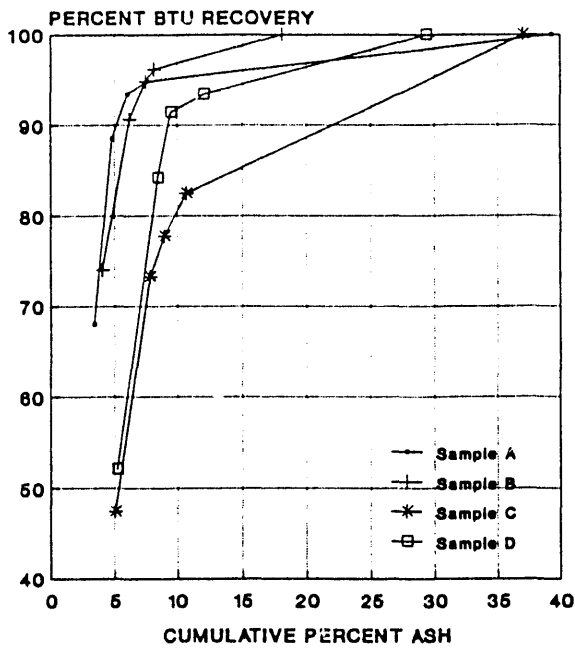


Figure 4: Btu recovery of the cumulative float product as a function of ash content for +230 M fraction of the four effluent samples

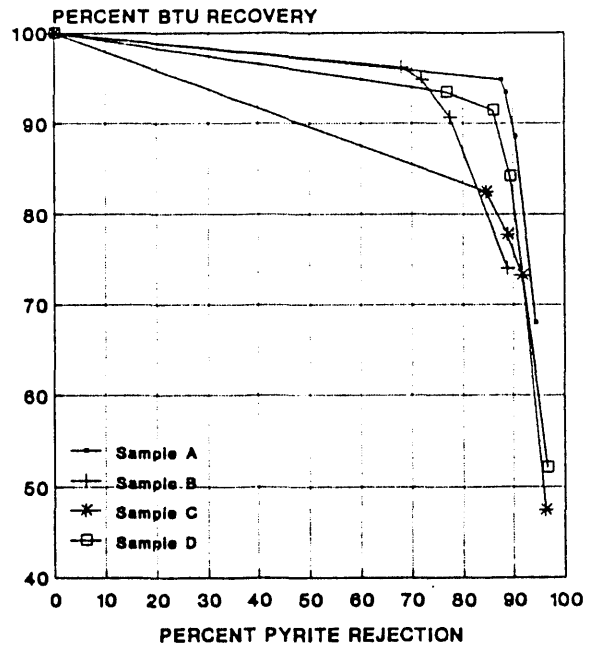


Figure 5: Btu recovery of the cumulative float product as a function of pyritic sulfur rejection for +230 M fraction of the four effluent samples

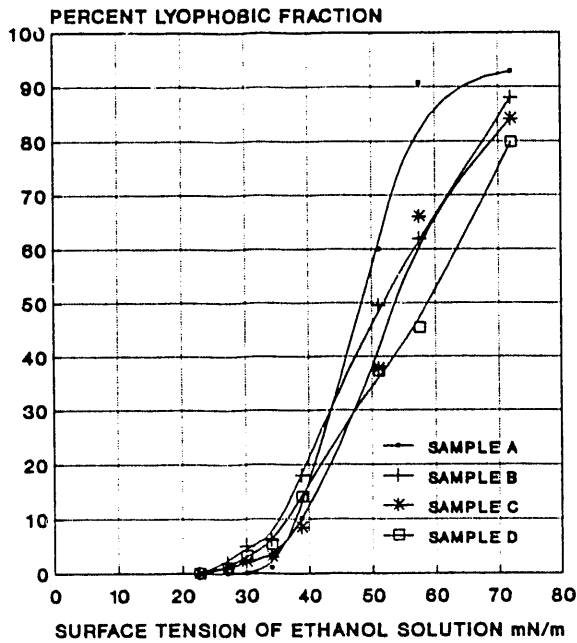


Figure 6: Partition curve for the film flotation of 45 x 80 mesh size fraction of the four effluent streams

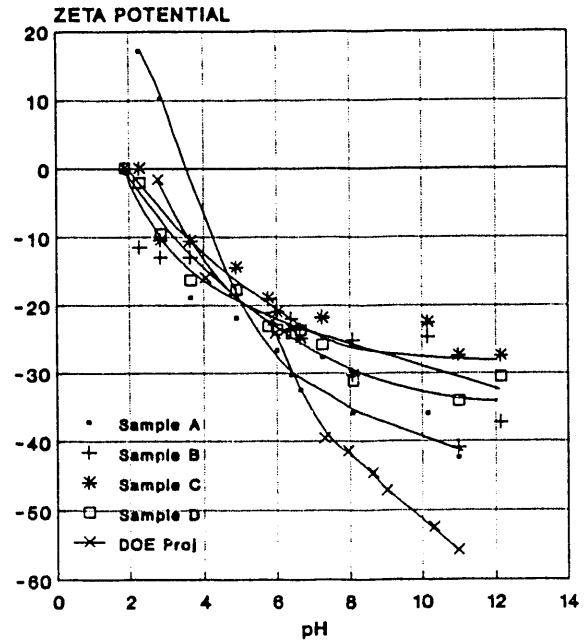


Figure 7: Zeta potential for the effluent samples as a function of pH

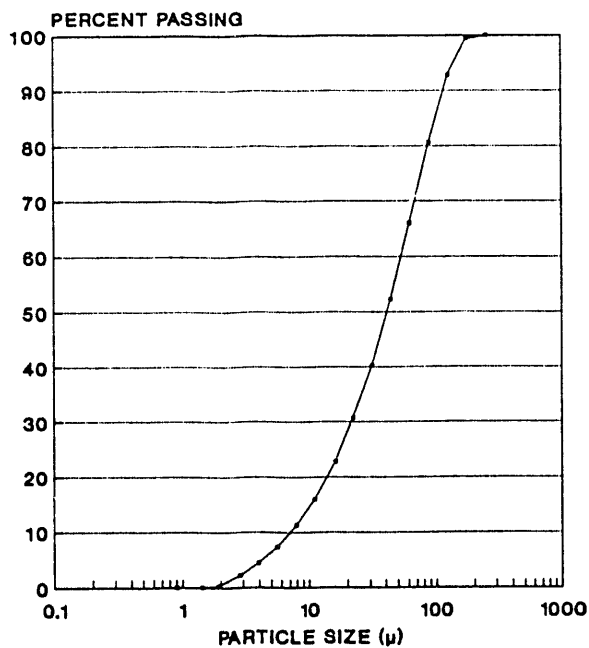


Figure 8: Grindsize distribution of the froth flotation feed for Sample A

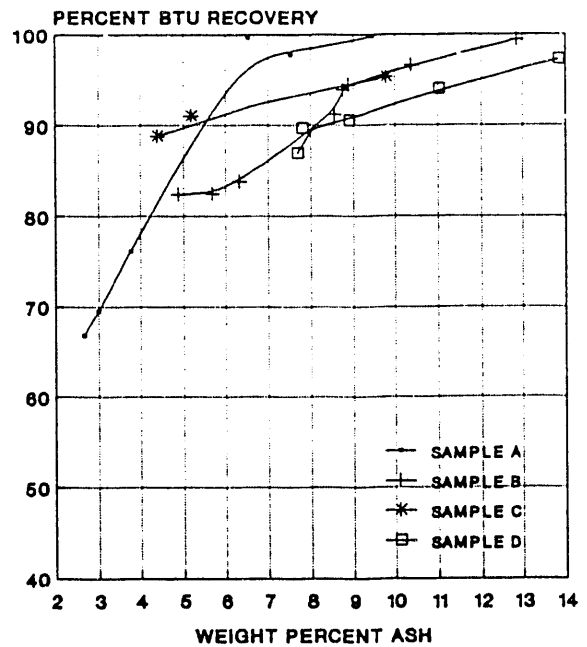


Figure 9: Btu recovery of the static tube product as a function of ash content for the four effluent samples

PROJECT MANAGEMENT REPORT
June 1 through August 31, 1991

Project Title: Recovery of Coal Fines from Preparation Plant Effluents
Principal Investigator: Vas Choudhry, Praxis Engineers, Inc.
Other Investigators: Latif Khan, Illinois State Geological Survey, and
David Yang, Michigan Technological University
Project Monitor: Daniel D. Banerjee, CRSC

COMMENTS

The project has been successfully completed as scheduled within the allotted funds. All project goals have been met. Excellent progress was made during the third and fourth quarters, which compensated for the slow start of the project in the first quarter.

All expenditures incurred on the project have been submitted by the two subcontractors, Illinois State Geological Survey (ISGS) and Michigan Technological University (MTU), and are included in this quarterly report. These invoices have been finalized and are being sent to CRSC for payment.

Project Expenditures

Projected and estimated expenditures are summarized in Table 1. As may be seen in this table, the cumulative costs for the four quarters are essentially within the budgeted amounts for most of the categories. However, the costs in the labor category were slightly higher than the budgeted amount due to the report writing effort. The travel and indirect costs were kept lower than the budgeted amount thus keeping the overall expenditure for the project within the budget. Table 2 presents a summary of the costs by various categories for each quarter. Figure 1 presents the projected expenditures by the quarter in comparison with the actual estimated expenditures on a cumulative basis. As may be seen, the project has been completed within the allotted funds.

Project Schedule

Figure 2 presents the project progress marked on the original project schedule. The planned progress has essentially been achieved in all tasks, and all project requirements have been met as indicated by the major milestones.

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

Table 1
Projected and Estimated Expenditures by Quarter

Quarter*	Types of Cost	Direct Labor	Materials & Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Cost	Total
Sept. 1, 1990 to Nov 30, 1990	Projected	\$6,049	\$375	\$2,100	\$0	\$1,200	\$8,052	\$17,776
	Estimated	\$4,799	\$251	\$1,076	\$0	\$6	\$5,455	\$11,587
Sept. 1, 1990 to Feb 28, 1991	Projected	\$22,598	\$1,200	\$3,900	\$2,146	\$2,257	\$21,552	\$53,653
	Estimated	\$9,987	\$251	\$1,280	\$0	\$127	\$11,917	\$23,562
Sept. 1, 1990 to May 31, 1991	Projected	\$39,148	\$1,650	\$3,900	\$2,146	\$2,757	\$34,351	\$83,952
	Estimated	\$30,171	\$782	\$1,748	\$2,360	\$3,031	\$27,036	\$65,128
Sept. 1, 1990 to Aug 31, 1991	Projected	\$45,197	\$1,650	\$5,100	\$2,146	\$3,257	\$42,118	\$99,468
	Estimated	\$47,665	\$782	\$3,700	\$2,360	\$4,854	\$40,518	\$99,878

* Cumulative by Quarter

Note: This table was revised to include the subcontractors' costs by each category rather than as a single item under ODC as was done in the final proposal

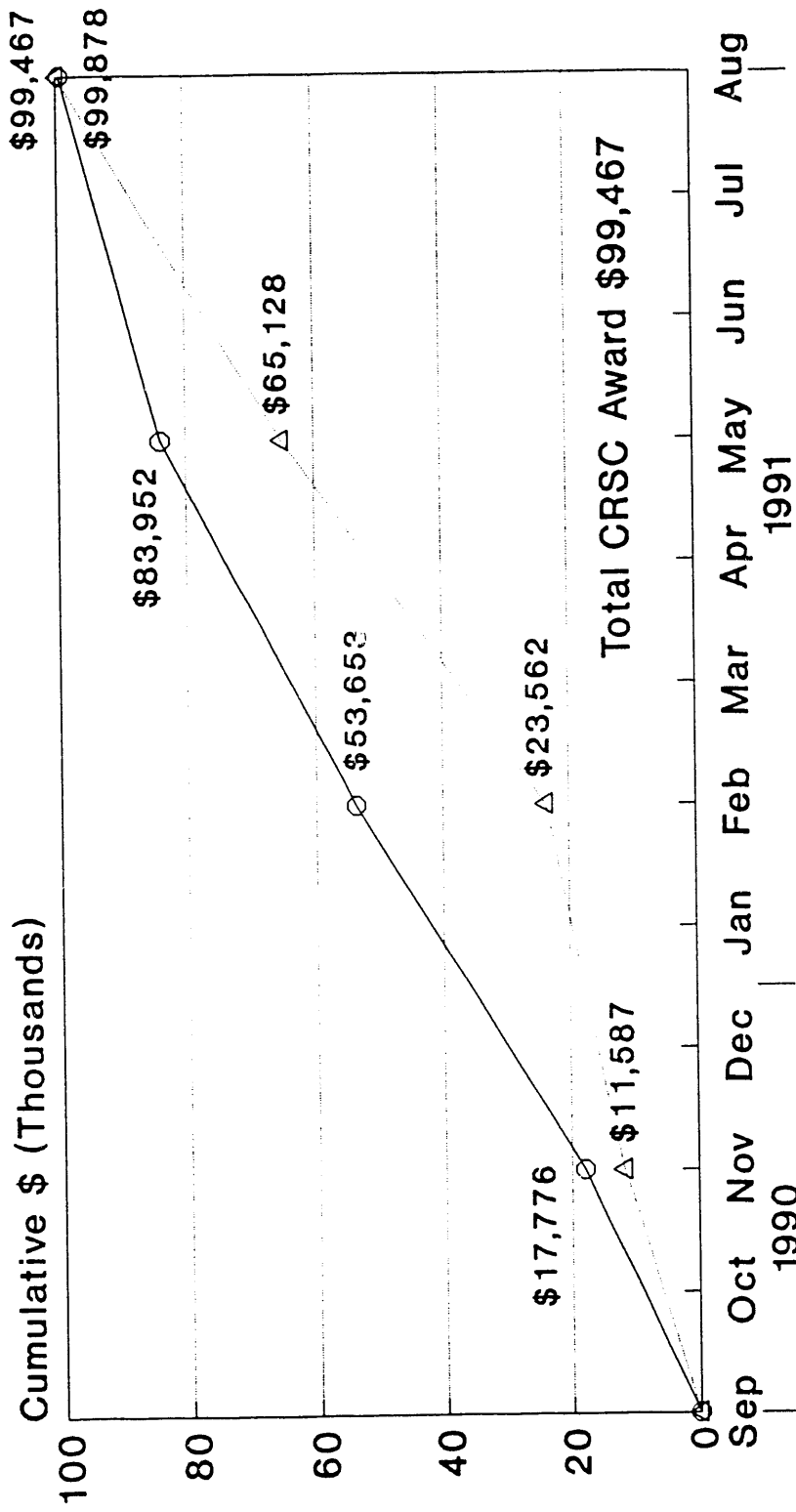
Table 2

PROJECT EXPENDITURES BY QUARTER

Category	Quarter 1		Quarter 2		Quarter 3		Quarter 4		Project Total
	Direct	Cumulative	Direct	Cumulative	Direct	Cumulative	Direct	Cumulative	
1A Direct Labor	\$4,116	\$4,116	\$4,309	\$8,425	\$16,376	\$24,801	\$15,378	\$40,180	\$40,180
1B Benefits	\$682	\$682	\$879	\$1,562	\$3,808	\$5,369	\$2,115	\$7,485	\$7,485
1 Total Labor	\$4,799	\$4,799	\$5,188	\$9,987	\$20,184	\$30,171	\$17,494	\$47,665	\$47,665
2 Materials & Supplies	\$251	\$251	\$0	\$251	\$531	\$782	\$0	\$782	\$782
3 Travel	\$1,076	\$1,076	\$204	\$1,280	\$468	\$1,748	\$1,952	\$3,700	\$3,700
4&5 Other Direct Costs	\$6	\$6	\$122	\$127	\$2,904	\$3,031	\$1,823	\$4,854	\$4,854
6 Equipment	\$0	\$0	\$0	\$0	\$2,360	\$2,360	\$0	\$2,360	\$2,360
7 Total Direct Costs	\$6,131	\$6,131	\$5,514	\$11,644	\$26,447	\$38,092	\$21,269	\$59,360	\$59,360
14 Overhead	\$3,996	\$3,996	\$4,955	\$8,952	\$7,424	\$16,375	\$7,899	\$24,274	\$24,274
9 Total Indirect Costs	\$5,454	\$5,454	\$6,463	\$11,917	\$15,119	\$27,036	\$13,481	\$40,518	\$40,518
10 Total Costs (7 + 9)	\$11,585	\$11,585	\$11,976	\$23,562	\$41,566	\$65,128	\$34,750	\$99,878	\$99,878

COSTS BY QUARTER

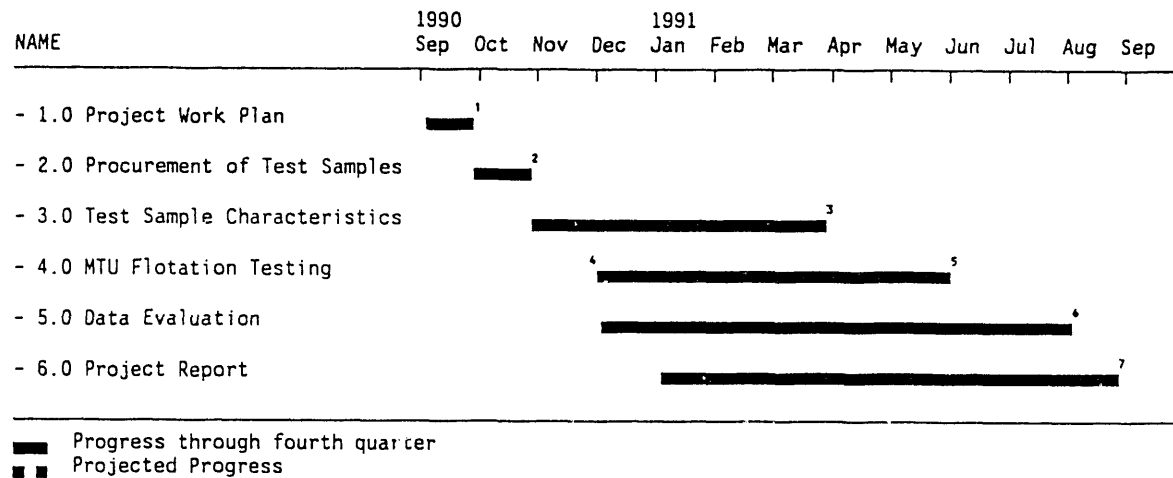
Recovery of Coal From Effluent Streams



Months and Quarters

○ Projected △ Estimated

Figure 1



List of Major Milestones

- 1 Work plan submitted
- 2 Sample Collection completed
- 3 Characterization completed
- 4 Flotation testing started
- 5 Flotation testing completed
- 6 Data Evaluation completed
- 7 Final report issued

Figure 2: Project Schedule

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

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