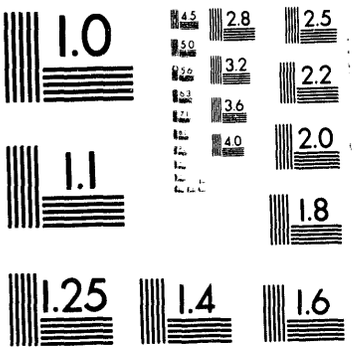
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1 of 1

DOE/PC/93521--T116

TECHNICAL REPORT
September 1 through November 30, 1993

Project Title: **PRODUCTION OF ILLINOIS BASE COMPLIANCE COAL USING ENHANCED GRAVITY SEPARATION**

DOE Grant Number: DE - FC22 - 92PC92521

ICCI Project Number: 93 - 1/5.1B - 1P

Principal Investigator: B. C. Paul
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Southern Illinois University

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Project Manager: K. Ho, Illinois Clean Coal Institute

ABSTRACT

Illinois Basin coal often contains a significant portion of finely dispersed pyrite. Most of the free pyrite particles exist in the fine size fractions, which are generally treated using froth flotation. An inherent problem of the froth flotation process is the inefficient treatment of middling particles containing a small amount of coal on their surface. On the other hand, gravity-based processes can effectively remove middling particles containing only a small amount of coal. Falcon Concentrators Inc. and Knelson Gold Concentrators Inc. have developed full-scale, enhanced gravity separators for the treatment of heavy minerals. This project will evaluate the potential of using these concentrators to de-ash and de-sulfurize Illinois coal fines, thus, producing coal products that meet the requirements for Phase I of the Clean Air Act. Since both continuous separators are commercially available, the results obtained in this investigation should be applicable to industrial operations.

Work in the first quarter has focused on equipment set-up and sample acquisition. The Falcon concentrator has been purchased and delivered to the Illinois Coal Development Park. Sample acquisition has been adversely effected by the on-going coal strike. Because the units being tested are commercial scale, 40 fifty-five gallon drums of slurry will need to be collected. Collection of such large samples requires labor at the preparation plant, which is in short supply during the strike. The cooperating partners are optimistic that the samples can be collected with little additional delay.

U.S. DOE Patent Clearance is **NOT** required prior to the publication of this document.

EXECUTIVE SUMMARY

Illinois Basin coals usually contain a significant portion finely dispersed pyrite. With Phase I of the Clean Air Act becoming effective in 1995, Illinois coal producers will need to produce a lower sulfur product to avoid loss of market. Physical coal cleaning can, in theory, remove pyritic sulfur from Illinois coals. In some cases, this will be sufficient in producing a marketable coal that meets the maximum Clean Air Act SO_2 emission limits.

At coarse coal particle sizes (+28 mesh), most of the pyrite is not sufficiently liberated from the coal matrix. Thus, physical cleaning in many current preparation plants cannot remove enough pyrite to ensure compliance. Therefore, to achieve effective pyrite rejection, the coal particle size must be reduced to a very fine size, typically less than 28 mesh. Froth flotation is the process most commonly used to treat this fine fraction. The process, however, has poor selectivity with respect to middling particles. For example, a particle covered by 90% pyrite and 10% coal may have enough coal surface area for bubble attachment and flotation. On the other hand, the same particle would be rejected easily by a gravity-based process due to the large density differences between coal and pyrite. Unfortunately, in the past, gravity-based separators have been ineffective at treating fine particles due to the low particle inertia.

One way of achieving a gravity-based separation on fine particles is to exert an enhanced gravity force on these particles using a centrifugal action. Both the Falcon Concentrators Incorporated and Knelson Gold Concentrators Incorporated have developed large commercial gravity separators for the gold and metal mining industries based on this principal. These units are capable of operating continuously with throughputs of up to 200 tph. Because manufactures already have units sufficiently large for the fine circuits at most Illinois preparation plants, there would be no delay in creating large commercial units from small laboratory proto-types.

The problem with applying these enhanced gravity separators to Illinois coal fines is that there is almost no test data for these units operating on coal. Falcon Concentrators Incorporated has run a few tests on Canadian coals, and the investigators at SIUC have run numerous batch tests on Illinois coal using laboratory batch concentrators. The SIUC results which led to this project were very promising. A sample of high sulfur waste fines discarded by one local preparation plant was cleaned to less than 2.5 lbs SO_2 per million BTU and would thus be classified as a compliance fuel.

The Falcon concentrator was originally designed to collect a few percent of the heaviest material from a feed stream. The

capacity is almost ideal for the removal of the pyrite fraction from Illinois coal. However, in addition, tests with the batch unit also indicate that high ash rejection can be achieved. Still other batch tests indicate that by changing only the percentage solids in the feed, the unit can be made to remove (deslime) fine clay particles. The reason most often given for not cleaning fine coal is that dewatering is too difficult or costly. Thus, the enhanced gravity concentrators show promise both in producing a compliance coal product and decreasing the dewatering difficulties associated with fine coal.

A second reason sometimes given for not cleaning fine coal is the high cost of grinding. The mining and standard crushing processes already used during the production cycle yields a significant portion of fine coal particles. As the Illinois coal industry moves from surface mines to underground high production longwall faces, the percentage of the coal product reporting to the fines circuit will probably increase to more than 10%. Discarding a few percent of the finest coal was considered acceptable to the industry in the past. However, the larger volumes of fines reporting to Illinois preparation plants in the future and the fact that the fine coal is the most readily cleanable fraction will make fine coal cleaning a more attractive alternative.

The current project will test the Falcon and Knelson continuous units for their ability to remove pyritic sulfur and ash-forming material from fine coal streams. The units will also be tested for their ability to remove toxic elements from Illinois coal fines. The specific objectives of the project are (1) to demonstrate the use of enhanced gravity concentrators for the efficient and economical treatment of fine coal, (2) to identify and optimize the critical operating parameters of the Falcon and Knelson centrifugal units, (3) to evaluate and compare the throughput capacity and scale-up factors of both units and (4) to determine the effect of the applied centrifugal field on the gravity cut point and process efficiencies.

To achieve the project objectives, the operating parameter values for the Falcon and Knelson concentrators will be tested over a large range of values to determine their effects on process performance and to establish their respective limiting values. The response variables to be evaluated will include total and pyritic sulfur rejection, ash rejection, BTU recovery, and separation efficiency. Selected tests will also be evaluated for toxic element removal. These tests will involve the treatment of a -28 mesh coal sample and a flotation concentrate from an Illinois Basin coal preparation plant.

The optimization of the operating parameters will involve conducting an experimental program whose data will be used to

develop empirical expressions describing the response variables as a function of the operating parameter values. The experimental program will be performed according to a Box-Behnken test design. The range of values for the operating parameters will be based on the findings of the exploratory experiments.

After completion of the experimental program, a commercial statistical analysis program known as DESIGN-EXPERT will be used to analyze the test data collected for each bowl length and to produce the empirical expressions. Using these expressions, the operating parameter values will be optimized based on the desired values of the response variables using an off-line optimization technique, such as the Simplex and Hooke-Jeeves methods. Once the optimum parameter values have been determined, additional experiments will be conducted to verify the calculated optimum conditions.

Using the optimum operating parameter values corresponding to the maximum separation efficiency, tests will be performed over a range of rotational speed to obtain the best possible recovery-grade curve achievable by the Falcon concentrator. These results will be compared to those obtained by release and washability analyses to determine the improvement in process performance over that achieved by flotation. A washability analyses of the samples collected from these tests will be conducted and partition curves constructed to determine the effect of the applied centrifugal field on the gravity cut point and process efficiencies. These test samples will also be analyzed for trace elements to determine the effectiveness of the centrifugal units for trace element reduction. These experiments will be conducted on both the Falcon and Knelson concentrators so that a comparison can be made to determine the most feasible unit for a given application.

During this reporting period, the Falcon concentrator was delivered and partially installed as initially indicated on the milestone chart. Sample collection is behind schedule due to the coal strike. The units being tested are commercial size and, thus, require a substantial quantity of samples. A total of 40 fifty-five gallon drum samples are needed for the tests in this project. To collect that much material requires the assistance of several preparation plant employees. During the strike, preparation plants are operating with skeleton crews, and at reduced capacity not generally representative of typical operating conditions. The industrial partners in the project have reaffirmed their commitments to help with sample collection as soon as normal operation is re-established. Contingency plans have been made to obtain samples from other Illinois preparation plants in the event that a strike settlement is not reached in the near future.

OBJECTIVES

The overall goal of this project is to provide the Illinois Basin coal industry with a means of reducing sulfur in the washed coal products they produce, in time to assist them with the phase I requirements of the Clean Air Act. To accomplish this goal the project is investigating enhanced gravity concentrators that have already been developed to the size required for preparation plant use (up to 200 tph), and that already have know capital, and operating costs, and whose space requirements are compatible with installation into existing preparation facilities. These enhanced gravity concentrators by Falcon Concentrators Incorporated and Knelson Concentrators Incorporated have not been tested and proven for coal. To demonstrate the application of these concentrators for the treatment of Illinois Basin coal fines, the project objectives are:

1. To demonstrate that continuous commercial size enhanced gravity concentrators can remove enough pyrite from Illinois coal fines to approach or exceed the Clean Air Act requirements of 2.5 lbs SO^2 per million BTU;

2. To determine the amount of ash rejection that can be achieved by enhanced gravity washing of Illinois coal fines;

3. To demonstrate the ability of commercial size enhanced gravity concentrators to deslime clay particles away from fine coal and thus reduce any dewatering problems;

4. To establish the operating parameter values for commercial size enhanced gravity concentrators used to clean coal. This will allow operators to use approximately correct initial settings when units are installed in the preparation plants, and to know which parameters to adjust to fine tune the unit to their needs.

5. To develop scale-up relations between laboratory batch tests and continuous full scale plant performance such as those now available for many column flotation systems.

These objectives are to be achieved through the following tasks:

Task 1: Acquire large samples of fines from the operating circuits at actual preparation plants.

Task 2: Characterize representative samples taken from the slurry for parameters such as percent solids, particle size distribution, proximate analysis, total and pyritic sulfur content, and BTU.

Task 3: Preliminary screening of operating conditions will be done to determine such things as effect of feed rate, time to steady state operation, and impact of feed recirculation. Information gained from the preliminary screening will be used to select the operating parameter settings for later parametric study.

Task 4: A Box-Behnken design parametric study will be conducted of unit operating conditions on response variables such as ash rejection, sulfur rejection, and separation efficiency.

Task 5: An empirical model will be developed based on the results of the parametric study. The model will be analyzed by techniques such as the Simplex or the Hook-Jeeves method to determine the optimum settings for each response variable. Physical tests will then be conducted to ensure that an actual optimum was achieved.

Task 6: Quarterly and Final Reports will be prepared.

INTRODUCTION AND BACKGROUND

Illinois Basin coals usually contain finely dispersed, sulfur bearing pyrite. With phase I of the Clean Air Act becoming effective in 1995, Illinois coal producers will need to produce a lower sulfur product to avoid loss of market. Physical coal cleaning can, in theory, remove pyritic sulfur from Illinois coals, in some cases bringing the resulting product into compliance with Clean Air Act sulfur dioxide emission limits.

At coarse coal sizes (+28 mesh), much of the fine pyrite remains attached to coal particles (is not liberated) and the physical cleaning in many current preparation plants cannot remove enough pyrite to ensure compliance. Fine coal (-28 mesh fraction) has much more of the pyrite physically liberated and distinct from the coal. Froth flotation is the process most commonly used on this fine fraction. Bubbles attach to the water repellent (hydrophobic) coal surfaces causing the particle so attached to float. The process, however, has poor selectivity with respect to middlings particles. A particle made of 90% pyrite and 10% coal would still have enough coal surface area for bubble attachment and flotation. A gravity-based process would easily recognize such a

particle as far to heavy to contain much coal and could reject the particle. Unfortunately, at fine sizes the coal particles have a large surface area relative to their mass. Surface forces exert a large amount of drag resistance to prevent the separation of heavy and light particles. With the normal acceleration of gravity, the differences in pull between the heavy and light particles are too small to separate the particles against the large surface based drag forces.

One way of using a gravity based separation at fine particle size is to create a strong artificial gravity by such methods as rapidly spinning a separation bowl. Both Falcon and Knelson have developed large commercial gravity separators for the gold and metal mining industries based on this principal. These units are capable of continuous operation and can handle throughputs of up to 200 tons per hour per unit. The units require no more headroom than a typical jig and occupy only around 100 square feet of floor space. The cost for a large unit is around $200,000. Integration of such units into existing preparation plant designs is easily within the technical and economic reach of Illinois basin coal producers. Because manufactures already have units as large as would be needed for fines circuits at most Illinois preparation plants, there would be no delay in creating large commercial units from small laboratory proto-types.

The problem with applying these enhanced gravity separators to Illinois coal fines is that there is almost no test data for these units operating on coal. Falcon has run a few tests on Canadian coals, and the investigators at SIUC have run numerous batch tests on Illinois coal using laboratory batch concentrators. The SIUC results that led to this project showed very promising results. A sample of high sulfur waste fines discarded by one local preparation plant was cleaned to less than 2.5 lbs SO_2 per million BTU and would thus be a compliance fuel.

The Falcon concentrator was originally designed to collect a few percent of the heaviest material from a feed stream. The capacity is almost ideal for removal of the pyrite fraction from Illinois coal, but tests with the batch unit also indicate that high ash rejection can be achieved. Still other batch tests indicate that by changing only the percentage solids in the feed, the unit can be made to remove (deslime) fine clay particles. The Falcon and Knelson concentrators may thus be able to produce both a compliance coal product and one that can be easily dewatered.

The current project will test continuous commercial units by Falcon and Knelson for their ability to remove pyritic sulfur from fine coal streams. The units will also be tested for their ability to de-ash coal fines.

EXPERIMENTAL PROCEDURE

In this project, the Falcon and Knelson concentrators are to be tested for their ability to clean pyritic sulfur and clay from Illinois coal fines. With Phase I SO_2 limits set to take effect in 1995, only technology that is already of commercial scale can be used to aid coal producers in meeting the immediate requirements of the act. The Falcon and Knelson concentrators were selected for testing because they are the only enhanced gravity units available in sizes needed by the coal industry. (The MGS and Kelsey Jig by Carpco can clean coal but at best handle only 5 to 10 tons per hour).

In this work, the critical process parameters for a Falcon C10 and similarly sized Knelson concentrator will be identified and optimized in terms of maximum separation efficiency, sulfur and ash rejection, and BTU recovery. Trace element analyses will be conducted on selected samples to determine the feasibility of the units to reduce toxic element contents in the clean coal product. Maximum throughput rates will be evaluated and scale-up calculations conducted.

Since the C10 unit has the ability to process up to 60 gallons per minute, a sample size of approximately 6 fifty-five gallon drums will be placed in a sump and mixed. A recirculation loop controlled by a centrifugal pump will be used to transport slurry from the bottom of the sump to its top. During the tests, the C10 unit will be fed from a split stream on the recirculation loop. The feed rate will be controlled using a variable speed pump. The C10 unit will be mounted above the feed sump so that the overflow (product) and underflow (tailings) streams can be easily fed back into the feed sump.

After allowing sufficient time for the process to reach steady-state, a timed-sample of the feed, product, and tailing streams will be collected in short incremental time periods for approximately 5 minutes. The samples will be analyzed for ash content, total sulfur content, pyritic sulfur content and solids content. In some cases, a size-by-size analysis of ash and sulfur content will be conducted on each product stream to evaluate the method of separation. Particle size distributions of the feed will also be conducted to determine the extent of particle size degradation occurring during the tests. The specific objectives of the proposed project will be accomplished in five distinct tasks which are described below.

Task 1: Sample Acquisition

Samples for this study will be collected from the Illinois coal preparation facilities of the industrial partners as listed below:

Rend Lake Preparation Plant - Consolidated Coal
Delta Mine Preparation Plant - Amax Coal Company

The coal at these facilities is mined from the Illinois No. 6 coal seam. The sample collected at the Rend Lake facility will be a - 28 mesh coal slurry sample whereas the sample from the Delta Preparation Plant will be a flotation concentrate (-100 mesh). A total of 1000 gallons will be collected for each sample.

Task 2: Sample Characterization

Sample characterization of the -28 mesh and flotation concentrate samples will be conducted on a five-gallon bucket of slurry obtained from one of their respective sample barrels. The sample will be collected by dumping a barrel of the slurry into a sump that is part of a closed-loop sump/pump circuit. The solids will be sufficiently mixed to insure dispersion using a mixer and a recirculation loop that is controlled by a pump. When complete dispersion is achieved, a 5-gallon sample will be collected in short time increments from a split stream in the recirculation line.

Each sample will be analyzed for their solids content, particle size distribution, proximate analysis, trace element, total and pyritic sulfur content, and BTU content.

Task 3: Exploratory Testing

In this task, tests will be conducted to evaluate the effectiveness of the Falcon and the Knelson concentrators for the de-sulfurization and de-ashing of -28 mesh coal fines and flotation product from Illinois coal preparation plants. In addition, the critical process variables will be identified and their effects on metallurgical performance quantified.

The process parameters that will be evaluated in this project include both operating and design parameters. The operating parameters consist of feed rate, feed solids content, bowl rotational speed, and underflow rate. Feed rate controls the retention time, solids content the extent of hindered settling, rotational speed the magnitude of the centrifugal force, and underflow rate the mass flow rate and solids content of the underflow. The sole design parameter that will be evaluated is bowl length, which determines bowl volume and, thus, particle retention time.

The effects of the operating parameter values on process response variables, such as sulfur rejection and separation efficiency, will be evaluated over a large range of values to determine their limitations. This will be accomplished by

varying one parameter while maintaining the others at constant values. The feed solids content will be varied using either a decantation method to increase the solids content or adding water to decrease the solids content. The testing procedure will be the same as previously described. These tests will be conducted for both the -28 mesh and flotation concentrate samples.

The remaining coal samples will be tested over a range of feed rate values to generate a recovery versus grade curve for each coal. The other parameters will be maintained at their appropriate values. These results will be used to evaluate the feasibility of the Falcon and Knelson concentrators to de-sulfurize and de-ash the Illinois coal fines.

Task 4: Evaluation of Process Variables

In this task, a test program utilizing the Box-Behnken experimental design method (Mason *et al.*, 1989) will be performed. The test data from this experimental program will be used in Task 5 to develop empirical relationships describing the response variables (i.e., ash and sulfur rejection, separation efficiency, etc.) as a function of the operating variables listed in Task 3. The tests in this task will be conducted on both the -28 mesh and flotation feed coal samples. The range of the parameter values used in the experimental design will be based on the findings in Task 3. This design method will require that each parameter be tested at a center value and two symmetrical peripheral values. Since there are four parameters with each requiring three levels, a total of 27 tests are required to complete the experimental matrix.

The experimental design described above will be used to evaluate the three different bowl lengths available. This will bring the number of tests in this task to 81.

The experimental procedure will be identical to the one previously described. Feed, product, and tailing samples will be collected for each test and analyzed for ash content, total and pyritic sulfur content, and solids content. The particle size distribution of the feed samples will be analyzed to determine the extent of the particle size degradation. If the degradation is considered extensive for a given test, the test will be run again with a new sample.

Task 5: Empirical Modeling and Optimization

After the completion of Task 4, a commercial statistical data analysis program known as DESIGN-EXPERT will be used to analyze the collected data. The overall objective will be to develop empirical relationships which describe the effect of the process parameters on the response variables. At a

minimum, the response variables will include ash rejection, total and pyritic sulfur rejection, combustible recovery, and separation efficiency. The data analysis program will be used to perform all required statistical computations and regression analyses. The process parameter values for each bowl length will be optimized by applying an off-line optimization technique, such as the Simplex or Hooke-Jeeves methods (Box *et al.*, 1978), to the predicted results obtained from each of the empirical expressions.

The effect of the different bowl lengths will be evaluated using the empirical expressions. The comparison will be based on metallurgical performance and throughput.

Using the optimized parameter values and bowl length determined in terms of maximum separation efficiency, exploratory tests will be conducted for both coal samples to verify the optimum parameter values. This will be achieved by varying one of the parameter values while maintaining the others at their respective optimum values. This will require approximately 10 tests.

Once the optimum has been verified, tests will be conducted at five different feed rates while holding the other parameters at their optimum values. This will generate the optimum recovery-grade curve which will be compared with those obtained by washability and release analyses. Based on these results, throughput capacity values will be estimated for a range of desired product grades.

A total of four additional tests will be conducted using different rotational speeds. A washability analyses will be conducted on the product and tailing samples obtained from these tests. Using this data, partition curves will be constructed which will indicate the effect of the applied centrifugal field on gravity cut-point.

Exploratory tests will also be performed on the Delta flotation concentrate using the optimum parameter values determined in this test. While using the optimum bowl length, the operating parameter values will be varied in an effort to determine their optimum values for treating the Delta sample. This procedure is expected to require approximately 10 tests.

Task 6: Reporting

Quarterly management and technical progress reports will be submitted throughout the duration of this project according to the guidelines specified by the Illinois Clean Coal Institute (ICCI).

RESULTS AND DISCUSSION

Efforts during this quarter have been limited to sample collection and equipment installation. No test results are available. Problems with the ongoing coal strike have slowed sample collection, since not all plants are at full operation, and those in operation have only skeleton crews and cannot assist with the large samples needed for this project. Most operators seem optimistic that the coal strike will soon be over and that sample collection will be completed shortly. Contingency plans have been prepared to obtain the coal samples from alternate sites in the Illinois basin in the event that the strike persists.

CONCLUSIONS AND RECOMMENDATIONS

No conclusions or recommendations different from those in the original proposal have been developed.

REFERENCES

Box, G., Hunter, W., and Hunter, S., 1978. "Statistics for Experimenters," (John Wiley and Sons, New York, New York) p. 653.

Mason, R., Gunst, R., and Hess, J., 1989. "Statistical Design and Analysis of Experiments," (John Wiley & Sons, New York, New York) p. 692.

PROJECT MANAGEMENT REPORT
September 1 through November 30, 1993

Project Title: Production of Illinois Base Compliance Coal Using Enhanced Gravity Separation

DOE Grant Number: DE-FC22-92PC92521 (Year 2)
ICCI Project Number: 93-1/5.1B-1P
Principal Investigator: Bradley Paul
Department of Mining Engineering
Southern Illinois University
at Carbondale
Project Manager: Ken Ho, Illinois Clean Coal
Institute

COMMENTS

The total project costs expended during this reporting period was $38,090 which is substantially lower than the projected amount of $47,345. The lower expenditures are due to the matching funds provided by the Department of Mining Engineering during this reporting period for graduate research assistant support. Also, there were no contractual service expenses since sample analyses were not conducted.

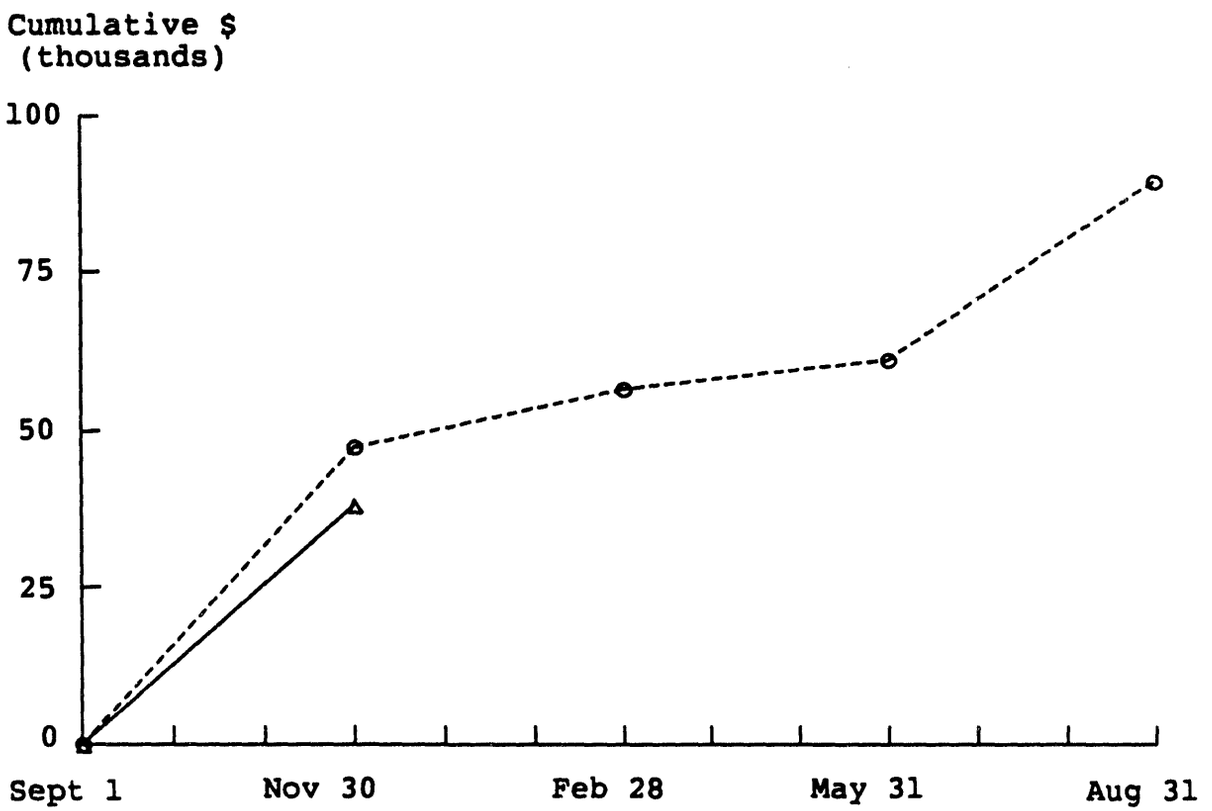
Projected and Estimated Expenditures by Quarter

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials & Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept. 1, 1993 to Nov. 30, 1993	Projected	2,908	0	2,000	333	34,800	3,000	4,304	47,345
	Estimated	0	0	2,430	0	32,200	0	3,460	38,090
Sept. 1, 1993 to Feb. 28, 1994	Projected	5,908	1,218	2,650	666	34,800	6,000	5,124	56,366
	Estimated								
Sept. 1, 1993 to May 31, 1994	Projected	6,158	1,218	3,300	1,000	34,800	9,000	5,548	61,024
	Estimated								
Sept. 1, 1993 to Aug. 31, 1994	Projected	24,041	4,298	4,600	1,500	34,800	12,000	8,124	89,363
	Estimated								

*Cumulative by Quarter

COSTS BY QUARTER

Production of Illinois Base Compliance Coal Using Enhanced Gravity Separation

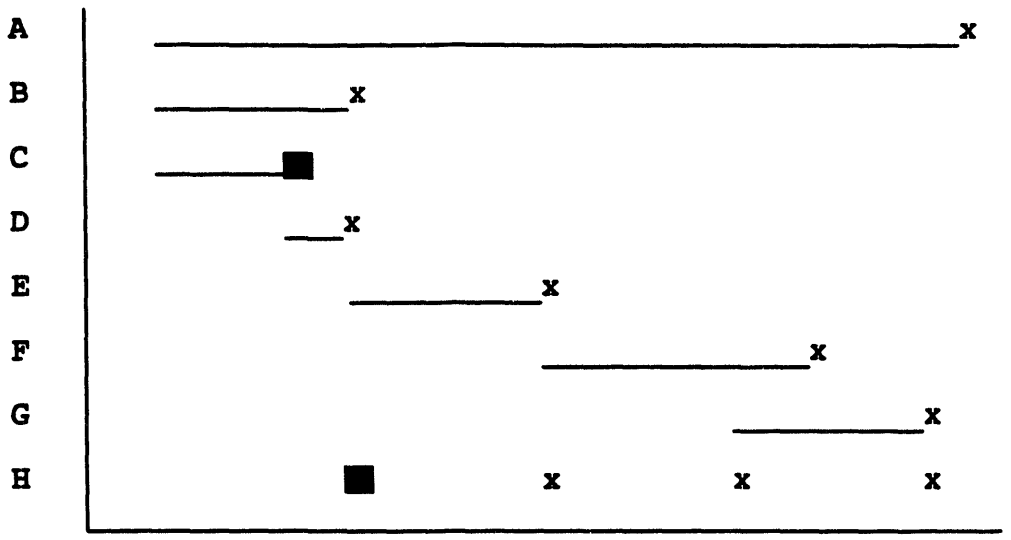


O = Projected Expenditures ---------

Δ = Estimated Actual Expenditures _____

Total Illinois Clean Coal Institute Award $89,363

SCHEDULE OF PROJECT MILESTONES



Milestones:

A. Research assistants employed.
B. Equipment ordered and received.
C. Sample acquisition completed (Task 1).
D. Sample characterization completed (Task 2).
E. Exploratory testing completed (Task 3).
F. Evaluation of process parameters completed (Task 4).
G. Empirical modeling and optimization completed (Task 5).
H. Reports written and submitted (Task 6).

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