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QUARTERLY TECHNICAL PROGRESS REPORT 11
APRIL - JUNE, 1995

ENGINEERING DEVELOPMENT OF ADVANCED PHYSICAL FINE COAL CLEANING FOR PREMIUM FUEL APPLICATIONS

Prepared for
U. S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, Pennsylvania 15236

By
Nick Moro, Gene L. Shields, Frank J. Smit, Mahesh C. Jha
Amax Research & Development Center
Golden, Colorado 80403-7499

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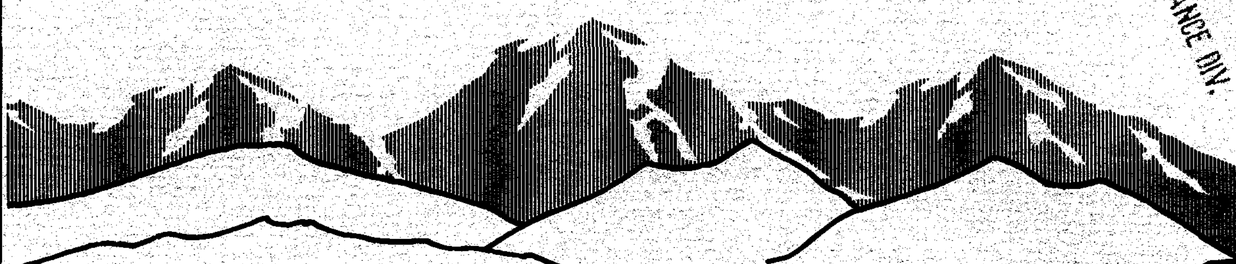

AMAX Research & Development Center
5950 McIntyre Street • Golden, Colorado 80403-7499
AMAX

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ABSTRACT

The primary goal of this project is the engineering development of two advanced physical fine coal cleaning processes, column flotation and selective agglomeration, for premium fuel applications. The project scope includes laboratory research and bench-scale testing on six coals to optimize these processes, followed by design, and construction of a 2-t/hr process development unit (PDU). The PDU will then be operated to generate 200 ton lots of each of three project coals, by each process. The project began in October, 1992 and is scheduled for completion by June, 1997.

During Quarter 11 (April - June, 1995), work continued on the Subtask 3.2 in-plant testing of the Microcel™ flotation column at the Lady Dunn Preparation Plant with the installation and calibration of a refurbished 30-inch diameter column. Several successful runs were also completed.

The final version of the Subtask 4.3 report discussing the formulation of coal-water slurry fuels (CWF) from advanced flotation products was issued. The evaluation of toxic trace element data for column flotation samples continued, with preliminary analysis indicating that reasonably good mass balances were achieved for most elements, and that significant reductions in the concentration of many elements were observed from raw coal, to flotation feed, to flotation product samples. Work on the detailed design of the PDU flotation unit under Task 5 was essentially completed during this reporting quarter.

A draft version of the Subtask 6.3 topical report discussing the selective agglomeration process optimization work was issued for comments. Significant progress was made on Subtask 6.5 selective agglomeration bench-scale testing. This included completion of construction and preliminary testing of all three project coals. Data from this work indicates that project ash specifications can be met for all coals evaluated, and that the bulk of the bridging liquid (heptane) can be removed from the product for recycle to the process.

The detailed design of the 2 t/hr selective agglomeration module progressed this quarter with the completion of several revisions of both the process flow, and the process piping and instrument diagrams.

Procurement of coal for PDU operation began with the purchase of 800 tons of Taggart coal. Construction of the 2 t/hr PDU continued through this reporting quarter and is currently approximately 60% complete.

EXECUTIVE SUMMARY

This project is a major step in the Department of Energy's program to show that ultra-clean coal-water slurry fuel (CWF) can be produced from selected coals and that this premium fuel will be a cost-effective replacement for oil and natural gas now fueling some of the industrial and utility boilers in the United States, as well as for advanced combustors currently under development.

The replacement of oil and gas with CWF can only be realized if retrofit costs are kept to a minimum and retrofit boiler emissions meet national goals for clean air. These concerns establish the specifications for maximum ash and sulfur levels and combustion properties of the CWF.

This multi-year cost-share contract started on October 1, 1992. This report discusses the technical progress made during the 11th quarter of the project from March 1 to June 30, 1995.

SPECIFIC OBJECTIVES OF PROJECT

The project has three major objectives:

- The primary objective is to develop the design base for prototype commercial advanced fine coal cleaning facilities capable of producing ultra-clean coals suitable for conversion to coal-water slurry fuel for premium fuel applications. The fine coal cleaning technologies are advanced column flotation and selective agglomeration.
- A secondary objective is to develop the design base for near-term application of these advanced fine coal cleaning technologies in new or existing coal preparation plants to efficiently process minus 28-mesh coal fines and convert them to marketable products in current market economics.
- A third objective is to determine the removal of toxic trace elements from coal by advance column flotation and selective agglomeration technologies.

APPROACH

The project team consists of Cyprus Amax Minerals Company through its subsidiaries Amax Research & Development Center (Amax R&D) and Cyprus Amax Coal Company (Midwest and Cannelton Divisions), Arcanum Corporation, Bechtel Corporation, Center for Applied Energy Research (CAER) of the University of Kentucky, Center for Coal and Mineral Processing (CCMP) of the Virginia Polytechnic Institute and State University, and The Industrial Company (TIC). Entech Global manages the project for Amax R&D and provides research and development services. Dr. Douglas Keller of Syracuse

University and Dr. John Dooher of Adelphi University are both consultants to the project.

The project effort has been divided into four phases which are further divided into eleven tasks, including coal selection, laboratory and bench-scale process optimization research, and design, construction, and operation of a 2 ton/hr process development unit (PDU). Tonnage quantities of the ultra-clean coals will be produced in the PDU for combustion testing by DOE. Near-term applications of advanced cleaning technologies to existing coal preparation plants is also being studied.

ACCOMPLISHMENTS DURING QUARTER

Activity continued during the April - June 1995 quarter on Phases I and II of the project, Tasks 3 through 8, as described below.

Task 3 Development of Near-Term Applications

A 1993 Bechtel engineering analysis evaluating potential column flotation and selective agglomeration applications found a column flotation application at the Lady Dunn Preparation Plant particularly attractive since the plant was being considered for a major capacity expansion. Because of the potential advantages of installing column flotation rather than mechanical flotation cells in the expanded fine coal cleaning circuit, Lady Dunn management was pleased to offer their plant as the study site for a near-term application of column flotation. The Microcel™ flotation column was selected for this study and the Center for Coal and Mineral Processing (CCMP) at Virginia Tech was assigned the responsibility for the on-site test work. During the previous reporting quarter, a new subtask 3.3 "Dewatering Studies" was added to the project. This work will also be performed by CCMP.

Subtask 3.2 Engineering Development

An existing 30-inch diameter Microcel™ test unit was refurbished and installed in the Lady Dunn plant for testing. The system has been calibrated and several successful runs were completed at quarter end. The initial performance met expectations, and once a few minor revisions are made to the equipment a plan for the parametric testing will be prepared. The main emphasis will be to obtain scale-up information for a full conversion to column flotation.

Subtask 3.3 Dewatering Studies

This new subtask was added during the previous reporting quarter. The work to be performed by Virginia Tech will aim at developing a novel dewatering process for clean coal fines. Three coals will be tested including the product from Subtask 3.2 near-term testing at the Lady Dunn plant. A test plan was prepared and submitted to DOE on April 11, 1995.

Task 4 Engineering Development of Froth Flotation

Task 4 is divided into five subtasks. Subtasks 4.1 Grinding, 4.2 Process Optimization Research, and 4.5 Conceptual Design of the PDU and Advanced Froth Flotation Module have been completed and were reported during previous quarters. There was activity on each of the two remaining subtasks during the eleventh quarter of this project.

Subtask 4.3 Advanced Flotation - CWF Formulation Studies

The final version of the Subtask 4.3 topical report was completed and issued to DOE and other project partners on May 23, 1995. The findings of this report indicate that four of the six project coals could be formulated into coal-water slurries with coal loadings varying from approximately 47% to 70%. The coarsest coal (Taggart) was loaded to the highest levels (68-70%) and the finest coal (Indiana VII) to the lowest levels (47-53%). Viscosities of these formulated slurries varied from several hundred to several thousand centipoise at a 100 Sec^{-1} shear rate depending on the coal type, loading level, and whether or not a stabilizer was used. It was generally found that slurry stabilities were poor unless a stabilizer was used, which invariably increased slurry viscosities dramatically. While a number of different approaches were evaluated in formulating these slurries, it was determined that the production of a bimodal coal particle size distribution (PSD) resulted in the best slurry properties. It was found that in order to generate the appropriate PSD, selective regrinding of a fine coal fraction was necessary for coarse coals like the Taggart. In contrast, for an already finely ground coal like the Indiana VII, it was necessary to generate a coarse coal fraction, prior to fine grinding, to achieve the appropriate bimodal PSD.

Subtask 4.4 Bench-Scale Testing and Process Scale-Up

While the test work for this subtask was completed in January, samples from selected bench-scale tests were retrieved from storage during this quarter for additional analyses, particularly sulfur and toxic trace elements. Flotation data from the 1-foot column testing was also organized and an initial draft of the subtask topical report initiated.

Toxic Trace Elements

The reduction in toxic trace element concentrations accomplished by bench-scale column flotation was studied by assaying the products from selected parametric tests. For this analysis, the distribution of the trace elements between the clean coal and waste product were determined. The toxic trace elements of interest are antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine.

The analyses were carried out by Huffman Laboratories since they reported consistent results and low detection limits on previously submitted samples. Huffman used perchloric acid decomposition and inductively coupled plasma (ICP) spectroscopy for most of the analyses. Mercury was determined by cold vapor spectroscopy, and chlorine by a total halides method.

The average of all of the mass balance closures was 102 percent, and the closures were good (+/-15 percent) for beryllium, chromium, cobalt, manganese, nickel and chlorine. The arsenic, mercury, lead, selenium, and cadmium balances however were off by more than 15 percent for some of the coals, and could not be determined for others. Repeat analyses are being obtained to resolve these discrepancies. Considering antimony, discrepancies are so widespread, it appears that there is a bias in the analytical procedure which will make repeat assaying of little value. Huffman suspects that there is a solubility problem at low concentrations and that it may not be feasible to quantify antimony concentrations below 1 ppm by the perchloric acid dissolution/ICP procedure.

The reductions in the various trace element concentrations accomplished during the column flotation tests were calculated on a heating value basis. While there are a few discrepancies in this data which are being resolved via repeat analyses, the concentrations of arsenic, beryllium, chromium, cobalt, manganese, mercury and selenium in the raw coal were clearly reduced by the combined conventional washing and advanced cleaning steps. While much of this reduction occurred at the mine preparation plant, column flotation reduced the concentrations of arsenic, chromium, manganese, and nickel remaining in the washed preparation plant product.

Task 5 Detailed Design of PDU and Advanced Flotation Module

Equipment Drawings

During this reporting quarter, equipment drawings were received for Clean Coal Conveyors 400-T-03 and 400-T-04. All drawings were "Approved for Construction" by Bechtel and Entech Global, Inc. engineers.

Detailed Steel and Grating Drawings

Drawings of the PDU's detailed steel and grating were reviewed and "Approved for Construction" by Bechtel and Entech Global, Inc. engineers.

Fine Grinding Mill Media Evaluations

Testing was performed during the quarter to determine the optimum grinding media for the Netzsch Fine Grinding Mill. Two media types were evaluated for use in the unit, steel and glass. Samples of Indiana VII coal were ground for a set period of time in a laboratory bead mill. Both glass and steel media (3 mm) were used. The ground coal samples were then subjected to laboratory cleaning via selective agglomeration and conventional froth flotation. Head samples, as well as the clean coal products were then analyzed for metal contamination.

The analysis revealed that significant chromium, iron, manganese, nickel, and silicon reduction occurs when cleaning the ground product. In addition, the overall concentration of these elements is significantly lower when using glass beads than when using steel media. As a result of this test work, the 3 mm glass media has been selected for use in the Netzsch fine grinding mill.

Task 6 Engineering Development of Selective Agglomeration

Task 6 is divided into six subtasks. Subtasks 6.1 Agglomerating Agent Selection, 6.2 Grinding Studies, and 6.6 Conceptual Design of the Selective Agglomeration PDU Module have been completed and were reported during previous quarters. There was activity on each of the three remaining subtasks during the eleventh quarter of this project.

Subtask 6.3 Process Optimization Research

A draft of the Subtask 6.3 topical report was submitted to DOE and project team partners on June 30, 1995 for review and comments. This report presents the laboratory optimization results for the continuous selective agglomeration process using pentane and heptane as reusable bridging liquids. The effects of varying feed rates/retention times and mixer speeds on performance were evaluated as well. The target ash specification of 2 lb ash/MBtu was met for each of the test coals when they were pre-ground to the required particle size distribution (PSD) for mineral liberation. Heating value recoveries generally exceeded 95 percent over a wide range of operating conditions.

Operation of a unitized high/low shear reactor was compared with operation of a conventional system with separate high- and low-shear mixing stages. Performance and energy consumptions of the two systems were found to be similar. The Taggart, Elkhorn No. 3, Sunnyside, and Winifrede coals were all found to agglomerate well. The Indiana VII coal, however, required the use of an activator (preferably asphalt) along with the bridging liquid to achieve inversion, while the subbituminous Dietz coal required acidification as well as an activator.

Comparison testing with diesel fuel and kerosene indicated that it would not be cost-effective to agglomerate with bridging liquids that could not be recovered from the product and reused.

Subtask 6.4 CWF Formulation Studies

Work completed on Subtask 6.4 during this reporting quarter involved preliminary evaluation of Subtask 6.5 selective agglomeration products for CWF formulation. To this end, steam stripped product samples of the Indiana VII and Sunnyside coals were filtered and prepared for slurry formulation. A base Sunnyside coal slurry, approximately 55% solids, was formulated from the as-received (no additional grinding) steam stripper product.

Subtask 6.5 Bench-Scale Testing and Process Scale-up

The Subtask 6.5 test work progressed significantly during this quarter. Construction of the 25 lb/hr bench-scale unit was completed along with start-up and shakedown testing of the system. A number of test conditions were evaluated utilizing three different coals ground to the sizes indicated below:

- Taggart coal - 62 mesh topsize
- Sunnyside coal - 100 mesh topsize
- Sunnyside coal - 150 mesh topsize
- Indiana VII coal - 325 mesh topsize

The bulk of the work completed to date was for the purpose of confirming the 2 t/hr PDU selective agglomeration module design. Parametric testing of those coals to be processed in the PDU will be completed during future testing.

A de-aromatized commercial grade heptane was used as the bridging liquid for all but one of the continuous agglomeration tests carried out, with a pure grade of heptane used for one 150-mesh topsize Sunnyside coal test.

Virtually all the unit operations in this bench-scale system were found to operate successfully with only minor difficulties encountered.

Taggart Coal - Start-up testing, utilizing Taggart coal ground to a 62-mesh topsize, was carried out primarily to determine system operability and to provide guidance in developing operating procedures. As expected, the Taggart coal results indicate that project ash specifications can be easily met while achieving very high product yields and Btu recoveries.

Generally, product ashes of less than 1 lb/MBtu were achieved at 98-99% yield and Btu recoveries approaching 100%. On a run-of mine basis, Btu recoveries were in the 93-

94% range. These product ash values, as well as performance indicators, utilize composited samples from froth skimming simulation, a procedure in which the material which floats in the tailings sample is included as part of the product. This procedure is used to simulate the PDU design, which will incorporate a froth skimming device to recover any coal lost through the screen. The effect of this unit operation is an increase in both yield and Btu recovery by as much as 2.5%, with minimal impact on product ash content due to the relatively small quantity of additional recovered material.

As part of testing completed for the Taggart coal, an evaluation of high-shear mixing requirements to achieve inversion was carried out. Two different high-shear impellers were evaluated during this testing, a 2.4-inch diameter impeller with 4 blades and a 3.6-inch diameter impeller with 6 mixing blades. This testing, completed to determine the minimum high-shear mixing requirements to achieve inversion indicated the following:

- As residence time in the high-shear vessel decreases, higher impeller tip speed must be used to maintain inversion and similar energy/coal ratios.
- As the slurry solids concentration increases, less power input is required to achieve inversion, since as the solids concentration increases more particle to particle contact occurs at similar energy inputs.
- The 2.4-inch impeller draws less power to achieve inversion than the 3.6-inch impeller.

Evaluation of the low-shear unit operation proved difficult due to plugging of the low shear discharge. It was determined through this initial start-up testing that when the low shear product approaches a critical size (4-6 mm in diameter) it ceases to flow through the 1-inch discharge port. The following general conclusions concerning the low-shear operation were made based on the initial Taggart coal test work:

- At heptane ratios required to achieve inversion during high shear (24-26% on a dry ash free coal basis), the low shear product grows to 4-6 mm in diameter at impeller tip speeds of 3-5 m/s.
- Higher impeller tip speeds (7-10 m/s) were needed to make agglomerates 2-3 mm in size that discharged easily from the low-shear vessel.
- Low shear discharge plugging is more pronounced at higher solids concentrations, particularly above 10%.

Product recovery on the vibrating screen proved adequate during typical operating conditions with the 62-mesh topsize Taggart coal. There was, however, some coal lost to the screen underflow (tailings) under all operating conditions. It was determined that both the amount of wash water used and the nature of the spray pattern utilized, influenced the amount of coal in the screen underflow. Higher water rates and stronger more forceful spray patterns both resulted in more lost coal. As such, finer spray pattern (misting) nozzles were obtained and utilized for all subsequent testing carried out with other coals.

Sunnyside Coal - During this reporting quarter, a total of 18 complete agglomeration test runs were carried out utilizing Sunnyside coal. Of these 18 test runs, 4 were carried out at a 100-mesh topsize grind, and the remaining 14 at a 150-mesh topsize grind. As for the Taggart coal, product ash values and performance indicators were based on the use of froth skimming simulation in which the material which floats in the tailings sample is included in the product.

For the four 100-mesh topsize Sunnyside coal test runs completed, the product ash specification of 2 lb ash/MBtu was met for 2 of them, with the other two considered borderline. As with the Taggart coal testing, agglomeration yields and Btu recoveries were very high, generally in the 96 and 98% ranges respectively. The yield reduction, from the 98-99% achieved with the Taggart coal, to 95-96% for the Sunnyside coal is expected since the Sunnyside feed coal ash content is approximately 5.5%, as compared to 2% for the Taggart coal.

All except one of the 150-mesh topsize Sunnyside tests carried out achieved the product ash specification of 2 lb/MBtu. The one test which did not achieve this specification utilized pure rather than commercial grade heptane and achieved the highest Btu recovery of all the tests. The bulk of these tests were completed at a 10% solids concentration with one test carried out at 7% solids and 3 tests at 13% solids.

Heptane dosage appeared to have a negligible effect on product ash content for all of these tests. It should be noted that only small ranges of heptane level can be evaluated to insure operability of the system, i.e., too little heptane prevents agglomerate formation while too much heptane results in an un-screenable oily mass.

While various operating conditions changed from test to test, in general, the lowest product ash and moisture contents are achieved when relatively large (2-4 mm) monosized agglomerates are produced during low shear. This indicates that a product of this size screens better, thereby rejecting more of the mineral matter bearing process water than smaller poorly formed agglomerates.

Indiana VII Coal - A total of 10 complete Indiana VII coal agglomeration tests were carried out this quarter. Using the same froth skimming simulation techniques as for the other coals, the product ash specification of 2 lb ash/MBtu was met at approximately 99% Btu recovery for three of the four tests that utilized a 48-mesh screen during agglomerate recovery. This indicates that the 325 mesh topsize grind used provides sufficient ash liberation.

However, the product ash specification was not met for any of the tests completed using the 100-mesh screen deck during agglomerate recovery. This indicates that this finer screen size does not provide drainage of the mineral matter bearing process water as well as the coarser 48-mesh screen. Other observations to be noted concerning agglomeration of the Indiana VII coal are as follows:

- The addition of asphalt at the rate of 7.5-15 lb per ton of coal is required to achieve inversion during high-shear agglomeration.
- Without the necessary asphalt, no growth will occur during low-shear, resulting in poor coal recovery during screening.
- Approximately 30-35% heptane (maf basis) is required to achieve high shear inversion and low-shear growth with the Indiana coal. This is more heptane than the 23-27% required for the Sunnyside and Taggart coals, as expected due to the finer grind.
- With the testing completed to date, there is no clear effect of solids concentration on product ash content.
- As noted for all testing to date, the product ash content appears most dependent on the agglomerate size distribution with relatively large (2-4 mm) monosize agglomerates having the lowest ash content.

Heptane Characterization - One of the most important aspects of evaluating steam stripping to recover heptane from agglomerated coal is characterization of both the heptane fed to the process and the heptane remaining on the stripped product.

During the course of this project, two types of heptane have been utilized. The first one is a de-aromatized commercial grade heptane which has a bulk cost of approximately $1.00 per gallon. The second is a pure grade of heptane which has a bulk cost of approximately $6.00 per gallon.

Characterization of these two heptane types was carried out by Phoenix Laboratories of Broomfield, Colorado. The results of these analyses, indicate that while the pure heptane is 99.2% n-heptane and 0.8% methylcyclohexane, the commercial grade of heptane contains around 25% n-heptane with the remaining material consisting of a dozen various hydrocarbons (primarily 2-methylhexane, 3-methylhexane, 2,3-dimethylpentane and methylcyclohexane).

In an effort to achieve reliable analysis of residual heptane in the stripped product, Huffman Laboratories of Golden, Colorado, was contacted. After discussions with Huffman, it was determined that given the many compounds present in this commercial grade of heptane, it would not be economically feasible to analyze for all of the components on a regular basis. It was therefore decided that standard practice during this project would be for Huffman Labs to determine the amount of n-heptane remaining in each sample. This value would then be converted to provide an estimate of the total solvent or bridging liquid remaining in any given sample. As a starting point a multiplier of "4" was used to convert from n-heptane to total bridging liquid remaining when the commercial grade was used. This assumes that n-heptane remained on the product in approximately the same ratio (25%) as it was present in the original material.

In order to allow for accurate conversion of n-heptane to total bridging liquid content for trace heptane analyses, i.e., to check the multiplier of "4", the distribution of the various hydrocarbon components remaining on different types of samples was determined by

Phoenix Labs. The following is a summary of observations made based on these residual hydrocarbon distribution results:

- When coal agglomerated with commercial grade heptane is thermally dried, n-heptane represents 7.9% of the total remaining hydrocarbons.
- When coal agglomerated with the commercial heptane is treated in the continuous 25 lb/hr steam stripper, n-heptane represented 29.9-33.0% of the total hydrocarbons remaining.
- When plain coal slurry (no heptane) is boiled for 15 minutes, 50-60 ppm of hydrocarbons on a dry coal basis were detected. These hydrocarbons consist primarily of benzene, toluene, ethylbenzene, and xylenes. Some heptane was also present along with the non-hydrocarbon compounds methyl butyrate and methyl isobutyrate.
- The stripping of pure heptane from coal resulted in a trace hydrocarbon distribution of which over 90% was n-heptane.
- Coal agglomerated with commercial heptane and then either boiled for 15 minutes, or boiled and then autoclaved at a higher temperature, had a trace hydrocarbon distribution in which 44-49% was n-heptane. Also present in these products were numerous other of the original hydrocarbon fractions.

Continuous Stripping Results - During this quarter, a variety of operating conditions were tested in the continuous steam stripping column to evaluate its heptane stripping capabilities. Due to problems in providing a consistent feed to the stripping operation, calculations of stripper operating conditions are based on stripper product flowrates and samples. In this manner more reliable operating conditions can be determined.

During preliminary testing, an estimate of the stripping column capacity determined that this unit operation could process coal at a rate approaching 20 lb/hr (at approximately 10% solids) without experiencing plugging problems. If the coal feed rate was set too high however, it was found that the stripper would plug with solids, indicating that there was insufficient steam available to break up the agglomerates (remove the bulk of the heptane), such that the coal could flow through the column and out the discharge.

In an effort to quantify the effects of various stripper operating parameters on product heptane levels, a number of tests were carried out utilizing Taggart, Sunnyside, and Indiana VII coals. For this work, the level of pall rings, the liquid level, and the steam rate were varied.

Single pass stripping of the 62-mesh topsize agglomerated Taggart coal achieved trace n-heptane concentrations in the range of 700-2000 ppm on a dry coal basis (about 2000-6000 ppm commercial heptane). Generally, there were no obvious trends relating residence time to product heptane concentration. Also, since residence time is determined by the liquid level in the column, there was no difference in performance between operating the column flooded (with a high liquid level) or in a continuous steam mode (low liquid level).

When carrying out continuous steam stripping of Sunnyside coal agglomerated with commercial heptane, n-heptane concentrations in the 1200-2700 ppm on a dry coal basis were achieved. Based on an estimated multiplier, this represents approximately 3500-8000 ppm of total commercial grade heptane on a dry coal basis. Similar ranges of trace n-heptane, 4700-5400 ppm on a dry coal basis, were determined for those three tests in which the pure grade of heptane was utilized.

For several of the Sunnyside stripper tests, stripper product was processed through the unit a second time providing longer residence times. The results of these tests indicate that while some additional reduction in heptane content is realized, substantial reductions do not occur with extended residence times.

Generally, these data indicate that slightly lower heptane levels are realized for the Taggart coal than the Sunnyside. One possible explanation for this is that the Sunnyside coal is ground finer (100- and 150-mesh) than the Taggart coal (62-mesh). This finer grind may provide more available surface area, and pores, to which the heptane adheres. This is confirmed by the fact that as a coal is ground finer, more heptane is required to achieve agglomeration.

Only two continuous stripping tests were carried out using agglomerated Indiana VII coal. As anticipated, the results for these two tests, one a single pass and the other a double pass through the stripper, indicate trace heptane levels similar to that achieved with the other two coals. Trace n-heptane detected for these two tests were about 2400 and 1800 ppm n-heptane respectively, equivalent to approximately 5000 and 7000 ppm of total hydrocarbons on a dry coal basis.

Batch Stripper Testing - In an effort to better quantify achievable trace bridging liquid concentrations remaining with a stripped product, a number of batch stripper tests were carried out. These batch test were found to provide a better indication of the effect of some parameters since more control over the tests were possible. Generally several different types of batch stripper tests were completed as indicated below:

- Boiling of recovered agglomerates in an open stirred beaker, on a hot plate, for a set period of time @ 94°C (the boiling point of water at Golden altitude)
- Thermal drying at 110°C for a set period of time
- Autoclave treatment at 115-1120°C for a set period of time

This initial batch work indicated that lower n-heptane levels are achieved as the residence time under stripping conditions increases. While this trend was expected, it was surprising that residence times of up to one hour were required to achieve trace n-heptane levels of approximately 1200 ppm on a dry coal basis, equivalent to about

3000 ppm of total hydrocarbons on a dry coal basis. This preliminary testing also indicated that thermal drying achieved much lower heptane levels.

In an effort to confirm these results, and to determine the removal characteristics of other bridging liquids, similar batch testing was completed using a pure heptane, commercial heptane, and pentane. Some tests were also completed with asphalt to evaluate its effect on heptane removal. Based on the results of this work the following conclusions were made:

- On a dry coal basis, thermal drying provides significantly lower trace solvent concentrations than boiling.
- Storage of the product for 2 days prior to stripping results in higher trace heptane concentrations than immediate stripping.
- The use of asphalt results in lower heptane concentrations when the product is boiled.
- Pentane can be removed to very low levels under all conditions tested.

Since significantly lower trace heptane contents are realized when thermal drying at 110°C over boiling at 94°C, batch stripper tests were carried out in which agglomerated products were boiled under pressure to maintain a slurry temperature in the range of 115-120°C for predetermined periods of time. To achieve these high temperatures, these tests were completed in a PARR Autoclave fixed with a 15 psi pressure relief valve. Heat was applied to the autoclave containing a fixed amount of slurry, and a continuous vapor discharge was released by the pressure relief valve to maintain the desired temperature. Based on results from these tests, it was determined that treatment in the autoclave at increased pressure and temperature, resulted in reduced heptane concentrations as compared to stripping at lower temperatures.

To reduce the amount of steam required during PDU operation, it is planned to carry out the steam stripping at approximately 25% solids. Therefore, a brief series of tests was completed to determine if solids concentration had any effect on the residual heptane level in a stripped product. It was found that stripping (or boiling) at 25% solids concentration results in negligibly higher levels of trace heptane concentration for both pure and commercial heptane tests than when the stripping is completed at 10% solids.

As part of this project, agglomeration circuit product will be formulated into highly loaded coal-water-fuels. As a first estimate to determine the fate of any heptane remaining on the coal after stripping, Sunnyside coal continuous stripper product was filtered and formulated into a slurry of approximately 55% solids. The heptane concentration of this slurry was monitored for two weeks and it was found that no decrease in heptane content occurred over time. This indicates that storage of highly loaded CWF slurries should not pose safety and environmental related risks.

Subtask 7.0 Selective Agglomeration - PDU Detailed Design

Three meetings were held with Arcanum and Bechtel personnel during this reporting period to discuss the detailed design of the PDU selective agglomeration module. Discussions focused on finalizing the PFD's and P&ID's so that the detailed design can be completed in a timely manner. The design has progressed considerably. Currently, only equipment sizing and layout remain to be completed along with a final review of the overall process control philosophy.

Grinding and Dewatering - It is not anticipated that any major changes in either the coal grinding or dewatering circuits will be required for the switchover from the flotation to agglomeration process. As such, the agglomeration process will use slurry ground by the installed equipment. This slurry will however be stored in one of two ground slurry storage tanks prior to being metered to the agglomeration process. This will insure that a homogeneous feed is available to the agglomeration circuit for extended periods of time.

Once processed, the agglomerated and steam stripped product will be dewatered in the same manner as during flotation circuit operation. Generally, the bulk of the product will be dewatered in the existing vacuum drum filter with two filter presses also available for product dewatering. Process tailings will be sent to the existing thickener from which they will be dewatered via a pair of tailings filter presses.

High Shear - High-shear agglomeration will be carried out in a circuit consisting of two high-shear reactors. These reactors will be sized to provide approximately 0.5 and 1 minute residence times respectively. Each high-shear agglomerator will be powered by variable speed drive units that can achieve up to 18 m/s impeller tip speeds. In this manner, high shear residence times from 0.5 to 1.5 minutes can be achieved by operating either unit individually, or both together in series.

Heptane will be metered to the high-shear vessel as required, anticipated not to exceed 40% of the coal rate. Currently it is anticipated that a pure grade of heptane will be used during PDU operation. However, the system will be capable of utilizing a commercial grade heptane also. Based on test results to date, the ability to add asphalt to high-shear will be provided for the Indiana VII coal. It is planned to use an anionic asphalt emulsion at a rate of 5-15 lb asphalt per ton of coal.

Low Shear - Low shear will be carried out in a single vessel divided into two sections via a horizontal baffle. Each section will provide approximately 2.5 minutes of residence time for a maximum of five minutes. The discharge piping will be arranged such that one or both sections can be utilized. The low shear operation will be powered by a single drive unit providing one impeller for each section of the vessel. The impellers will be of the radial-flow type and achievable tip speeds will be in the range of 2-8 m/s.

Agglomerate Recovery - The vibrating screen to be used to recover agglomerates from the low shear product will be a dewatering screen with a 48-mesh deck approximately 2-feet wide by 6-feet long. Sufficient spray nozzles will be provided to insure replacement of mineral matter bearing process water with fresh water.

The vibrating screen underflow (tailings) will then be processed through a froth skimmer. This skimmer will provide approximately 5-minutes of residence time for any carbonaceous material to float. If necessary, nitrogen will be bubbled through the skimmer to help the material float. A continuous paddle will then scrape the floating material to a launder, from where it will be combined with the screen product.

Steam Stripper - The combined screen and froth skimmer product will be diluted with hot water to approximately 25% solids. This feed will then be treated in the first stage steam stripper which will provide a 5-minute residence time at ambient pressure. The heat source for this stirred vessel will be the vapor product stream from the second stage stripper. This first-stage stripper product will then be pumped to the second-stage stripper which will provide about 10-minutes of residence time at 15-20 psi pressure and elevated temperatures. The product from this second stage stripper will then be cooled and sent to the dewatering circuit.

Condenser and Gravity Separator - Vapors from the first stage stripper will contain the recovered heptane and be condensed in a two-stage process. Initial cooling will be carried out by an air cooler, followed by a shell and tube condenser to provide the necessary sub-cooling. The condensed liquids will then be sent to a gravity separator from which both the heptane and the process water will be recycled to the process.

Task 8 PDU and Advanced Column Flotation Module

The majority of the Task 8 work effort was devoted to the construction of the Process Development Unit (PDU) and Advanced Flotation Module. TIC, The Industrial Company of Steamboat Springs, Colorado, continued the construction of the PDU during the quarter. All required permits were obtained from Jefferson County, Colorado. Currently, the flotation module is approximately 62% complete.

Entech Global personnel continued with the procurement of new capital equipment and instrumentation as well as the refurbishing of existing equipment. Efforts are also underway for the procurement of Taggart coal for use during startup and shakedown.

Subtask 8.1, Coal Selection and Procurement

Arrangements began for procuring the three feed coals needed for the PDU operation. Up to eight hundred tons of each coal will be needed when the requirements for the Task 9 selective agglomeration testing are included. To avoid a drastic change in the quality of the coal between loads, the required amount of each coal will be purchased at one time. The coals, selected on the basis of their performance in the bench-scale systems, will be shipped by rail and unloaded and stored in Denver. From there the coals will be trucked as needed to Ralston Development Corporation in Golden for crushing.

Taggart seam coal was picked as the Eastern coal for PDU testing. Eight hundred tons of this washed coal were purchased from the Steer Branch Mine in Virginia and has been received in Denver. The combined shipment analyzed 3.14 percent ash and 0.69 percent sulfur, and closely resembled the Taggart coal from the now closed Wentz Mine.

A survey indicated nine potential replacements for the coal from the Sunnyside mine which closed in 1994. Laboratory grinding/liberation tests showed that the Hiawatha seam coal from the Genwal Mine in Utah came closest to matching the properties of the Sunnyside coal. As such, present plans call for using the Hiawatha coal for PDU testing. It contains 0.5 percent sulfur and could be cleaned to less than 2.0 lb ash/MBtu.

Minnehaha Mine management confirmed that they will supply low sulfur Indiana VII coal for PDU testing as planned.

Subtask 8.2 PDU Construction

TIC Construction Activities - TIC, The Industrial Company of Steamboat Springs, Colorado, continued the construction efforts of the PDU Flotation Module. The PDU construction is on schedule for mechanical completion by July 31, 1995, with the overall project completion at 62% at the end of this reporting quarter.

TIC Request for Information - TIC and Amax R&D, Inc. have established a method to expedite and clarify unclear issues regarding the construction of the PDU. When an issue or question arises which requires a response by Amax R&D, Inc., TIC issues a written Request for Information (RFI) including the cost impact if any. Amax R&D then responds to the issue accordingly and authorizes the associated change order cost, if any. During the quarter, 56 RFI's were issued by TIC and acted upon by Amax.

Procurement of Permits for PDU Construction - A building permit was received from the Jefferson County Building Department on April 11, 1995. This allows for the structural erection of the PDU. A separate permit was also procured for all electrical work on May 5, 1995. This permit allows for all electrical construction at the PDU site.

Procurement of Capital Equipment - Entech Global personnel continued the procurement of capital equipment for use in the PDU. During the second quarter of 1995, RFQ packages for two equipment items were issued. Award packages, were also issued for three capital equipment items, while 25 equipment items were received.

Procurement of PDU Instrumentation - During the quarter, Entech Global, Inc. personnel issued RFQ's and awards for 51 PDU instrumentation items. Thirty items were received in June. The remaining items are expected during July.

Procurement of Control and Data Acquisition System (CDAS) - Honeywell, Inc. was selected during the quarter to supply and deliver a Control & Data Acquisition System for use in the PDU. The Controller, a Series 9000 unit is object oriented and would allow easy programming by Entech Global personnel. The control interface and database, a SCAN 3000 unit, will operate on a personal computer system using a SCO UNIX motif. Both units were received and installed during the quarter.

Control and Data Acquisition System (CDAS) Training - Because significant project savings can be realized, Entech Global will perform a considerable portion of the CDAS configuration and programming. As such, Entech Global personnel attended two formal training sessions at the Honeywell Automation College in Phoenix, AZ. These two training classes focused on programming the SCAN 3000 Data Acquisition Unit, and configuring the Series 9000 Controller.

Refurbishing of Existing Equipment - During this quarter, three of thirteen used equipment units have been refurbished. The remaining ten units will be reconditioned in July.

Assembly of Spare Parts List - A spare parts list has been assembled and short listed. Only wear items that are most likely to fail during the first year of operation will be purchased. Ordering of spare parts commenced during the quarter.

Transformer Installation - A Purchase Order was issued to Public Service Company of Colorado for installation of one 1500 kVA transformer as well as overhead service from McIntyre Street. A meeting with Mr. Leonard Ellis of Public Service Company on April 19, 1995 revealed that the transformer and overhead lines should be installed during the week of July 10, 1995.

Equipment Hold Points and Shop inspections - Inspection of three new capital equipment items was conducted during the quarter. Inspection of the Gardner-Denver air compressor was performed at the manufacturer's Sedalia, Missouri, assembly plant on April 21, 1995. Inspection of the Netzsch Fine Grinding Mill was conducted on April 26 & 27 at the Netzsch facility located in Exton, Pennsylvania. A rotational test and sound test were conducted on April 26 while a pressure/leak test and electrical interlock test were conducted April 27. Inspection of the Microcel™ column flotation unit was conducted on May 5, 1995 at the fabrication facility located in Salt Lake City, Utah. In all cases, the units met all specifications. As a result, a "Release for Shipment" was issued for each item.

On-Line Ash Analysis of Microcel™ Flotation Column - Tests conducted on coal slurry samples at Nalco Chemical Company's Richlands, Virginia, laboratory indicated that the ash probes work very well on tailings but not on clean coal. Accurate results were obtained at ash values greater than 40%. As a result, the use of such probes in the PDU is being re-evaluated.

Project Review Meeting - A project review meeting was conducted at the PDU site with Bechtel, TIC, and Entech Global personnel. A presentation was made by Entech Global and TIC personnel regarding the status and details of the PDU construction. Following a brief discussion, all attendees toured the PDU. Overall, the attendees were quite pleased with the progress of the PDU and Advanced Flotation Module construction.

INTRODUCTION AND BACKGROUND

The main purpose of this project is the engineering development of advanced column flotation and selective agglomeration technologies for premium fuel applications. Development of these technologies is an important step in the Department of Energy (DOE) program to show that an ultra-clean coal-water slurry fuel (CWF) can be produced from selected United States coals and that this fuel will be a cost-effective replacement for a portion of the oil and natural gas burned by electric utility and industrial boilers in this country, as well as for advanced combustors currently under development. Capturing even a relatively small fraction of the total utility and industrial oil-fired boiler fuel market would have a significant impact on domestic coal production and reduce national dependence on petroleum fuels. Significant potential export markets also exist in Europe and the Pacific Rim for cost-effective premium fuels prepared from ultra-clean coal.

The replacement of oil and natural gas with CWF can only be realized if retrofit costs and boiler derating are kept to a minimum. Also, retrofit boiler emissions must be compatible with national clean air goals. These concerns establish the specifications for the ash and sulfur levels and combustion properties of ultra-clean coal discussed below.

This multi-year cost-shared contract effort began on October 1, 1992, and is scheduled for completion by June 30, 1997. This report discusses the technical progress made during the eleventh quarter of the project, April 1 to June 30, 1995. Ten quarterly reports have been issued previously [1-10].

SPECIFIC OBJECTIVES OF THE PROJECT

The three major objectives of this project are discussed below.

The primary objective is to develop the design base for prototype commercial advanced fine coal cleaning facilities capable of producing ultra-clean coals suitable for conversion to stable, highly loaded coal-water slurry fuels. These slurry fuels should contain less than 2 lb ash/MBtu HHV (860 grams ash/gigajoule) but preferably less than 1 lb ash/MBtu HHV (430 grams ash/gigajoule), and less than 0.6 lb sulfur/MBtu HHV (258 grams sulfur/gigajoule). The advanced fine coal cleaning technologies to be employed are advanced column froth flotation and selective agglomeration. Operating conditions during the advanced cleaning processes should recover at least 80 percent of the heating value in run-of-mine source coals at an annualized cost of less than $2.50/MBtu ($2.37/gigajoule), including the mine mouth cost of the raw coal.

A secondary objective of the work is to develop a design base for near-term commercial applications of these advanced fine coal cleaning technologies. These applications should be suitable for integration into new or existing coal preparation plants for the purpose of economically and efficiently processing minus 28-mesh coal fines. The design base will also include the auxiliary systems required to yield a shippable, marketable product such as a dry clean coal product.

A third objective of the work is to determine the distribution of toxic trace elements between clean coal product and refuse during the cleaning of various coals by advanced froth flotation and selective agglomeration technologies. Eleven toxic trace elements have been targeted. They are antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, and selenium. The results will show the potential for removing these toxic trace elements from coal by advanced physical cleaning.

APPROACH

A team headed by Amax Research & Development Center (Amax R&D) was formed to accomplish the project objectives. Figure 1 shows the project organization chart. Entech Global, Inc. is managing the project for Amax R&D and also performing laboratory research and bench-scale testing. Entech Global will also be responsible for the operation and evaluation of the 2 t/hr process development unit (PDU). Cyprus Amax Coal Company is providing operating and business perspective, the site for the near-term testing, and some of the coals being used in the program. Bechtel Corporation is providing engineering and design capabilities, and the operating experience it gained while managing similar proof-of-concept projects for DOE. The Center for Applied Energy Research (CAER) at the University of Kentucky and the Center for Coal and Mineral Processing (CCMP) at the Virginia Polytechnic Institute and State University are providing research and operating experience in the column flotation area. Arcanum Corporation is providing similar experience in the selective agglomeration area, while Dr. Douglas Keller of Syracuse University is serving as a consultant in the area of selective agglomeration, and Dr. John Dooher of Adelphi University is serving as a consultant in the area of coal-water slurry formulation. Recently TIC was awarded a subcontract for construction of the PDU and R. Reynouard was retained as a consultant to help with electrical and instrumentation systems.

The overall engineering development effort has been divided into four phases with specific activities as discussed below. As shown in Table 1, Work Breakdown Structure, the four phases of the project have been further divided into tasks and subtasks, with specific objectives which may be inferred from their titles. Figure 2 shows the project schedule.

Phase I

Phase I encompasses preparation of a detailed Project Work Plan, selection and acquisition of the test coals, and laboratory and bench-scale testing. The laboratory and bench-scale work will determine the cleaning potential of the selected coals and establish design parameters and operating guidelines for a 2 t/hr PDU containing both advanced column flotation and selective agglomeration modules. A conceptual engineering design is being prepared for a fully integrated and instrumented 2 t/hr PDU incorporating the features determined from the laboratory and bench-scale studies. A generic approach is being utilized during laboratory studies to insure that the flotation and agglomeration systems selected for the PDU will meet the project objectives.

Additional activities to be completed during Phase I include:

- Production of ultra-clean coal test lots by bench-scale column flotation and selective agglomeration for end-use testing by the DOE or a designated contractor
- Determination of toxic trace element distribution during production of these test lots
- Evaluation of the rheological properties of slurry fuels prepared from ultra-clean coals
- Evaluation of methods for applying these advanced cleaning technologies to existing coal preparation plants in the near term

Phases II and III

Phases II and III cover the construction and operation of the 2 t/hr PDU. Phase II will investigate advanced column flotation while Phase III will evaluate selective agglomeration. Process performance will be optimized at the PDU-scale, and 200 ton lots of ultra-clean coal will be generated by each process for each of the three test coals. Toxic trace element distributions will also be determined during the production runs. The ultra-clean coals will be delivered to the DOE for end-use testing.

Phase IV

Phase IV activities will include decommissioning of the PDU, restoration of the host site, and preparation of the final project report.

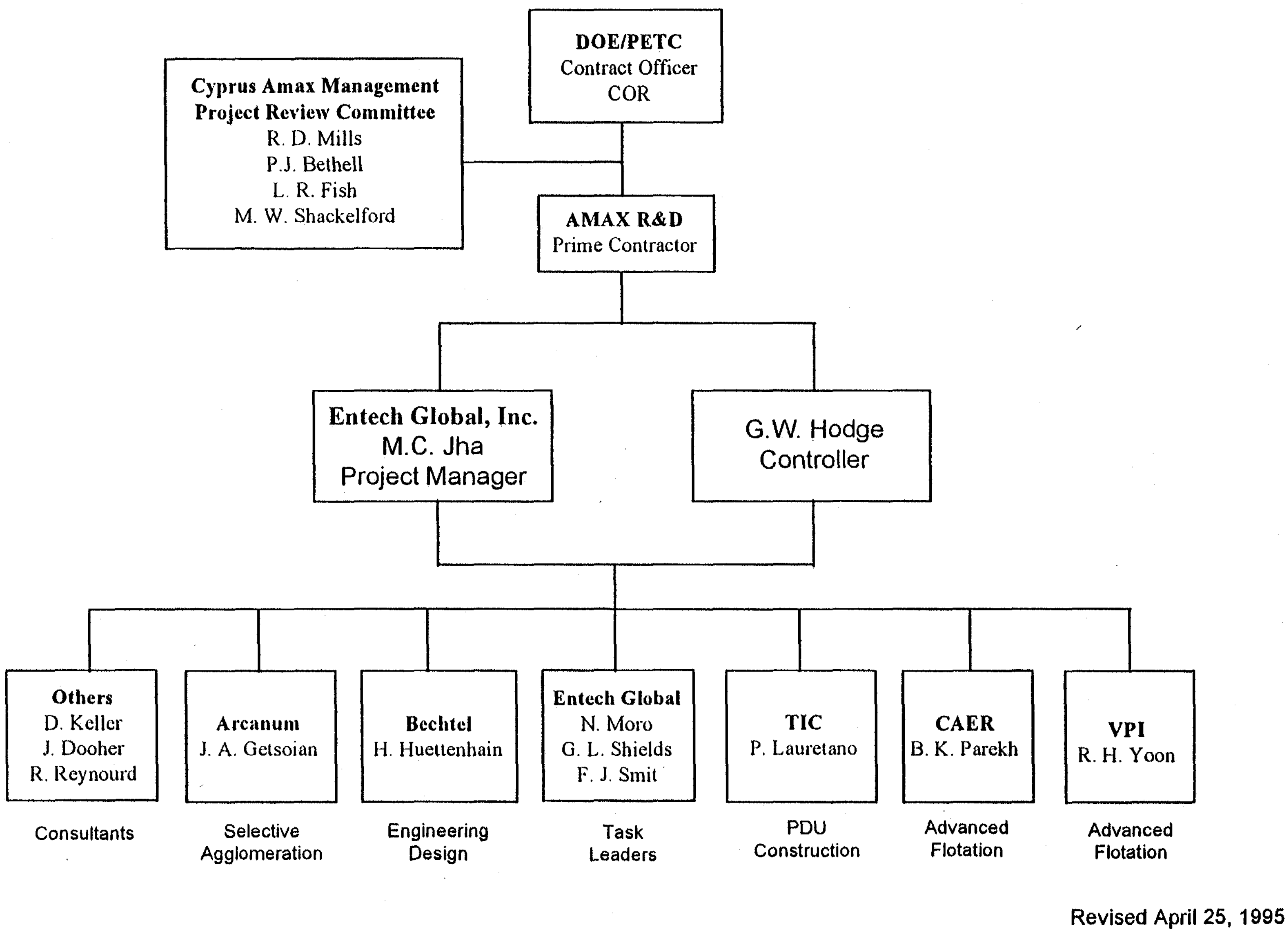


Figure 1. Project Management Organization Chart

Table 1. Outline of Work Breakdown Structure

	Phase I. Engineering Analysis and Laboratory and Bench-Scale R&D
Task 1.	Project Planning
Subtask 1.1.	Project Work Plan
Subtask 1.2.	Project Work Plan Revisions
Task 2.	Coal Selection and Procurement
Subtask 2.1.	Coal Selection
Subtask 2.2.	Coal Procurement, Precleaning and Storage
Task 3.	Development of Near-Term Applications
Subtask 3.1.	Engineering Analyses
Subtask 3.2.	Engineering Development
Subtask 3.3	Dewatering Studies
Task 4.	Engineering Development of Advanced Froth Flotation for Premium Fuels
Subtask 4.1.	Grinding
Subtask 4.2.	Process Optimization Research
Subtask 4.3.	CWF Formulation Studies
Subtask 4.4.	Bench-Scale Testing and Process Scale-up
Subtask 4.5.	Conceptual Design of the PDU and Advanced Froth Flotation Module
Task 5.	Detailed Engineering Design of the PDU and Advanced Flotation Module
Task 6.	Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels
Subtask 6.1.	Agglomeration Agent Selection
Subtask 6.2.	Grinding
Subtask 6.3.	Process Optimization Research
Subtask 6.4.	CWF Formulation Studies
Subtask 6.5.	Bench-Scale Testing and Process Scale-up
Subtask 6.6.	Conceptual Design of the Selective Agglomeration Module
Task 7.	Detailed Engineering Design of the Selective Agglomeration Module
	Phase II. PDU and Advanced Column Flotation Module Testing and Evaluation
Task 8.	PDU and Advanced Column Froth Flotation Module
Subtask 8.1.	Coal Selection and Procurement
Subtask 8.2.	Construction
Subtask 8.3.	PDU and Advanced Coal Cleaning Module Shakedown and Test Plan
Subtask 8.4.	PDU Operation and Clean Coal Production
Subtask 8.5.	Froth Flotation Topical Report
	Phase III. Selective Agglomeration Module Testing and Evaluation
Task 9.	Selective Agglomeration Module
Subtask 9.1.	Construction
Subtask 9.2.	Selective Agglomeration Module Shakedown and Test Plan
Subtask 9.3.	Selective Agglomeration Module Operation and Clean Coal Production
Subtask 9.4.	Selective Agglomeration Topical Report
	Phase IV. PDU Final Disposition
Task 10.	Disposition of the PDU
Task 11.	Project Final Report

Revised April 25, 1995

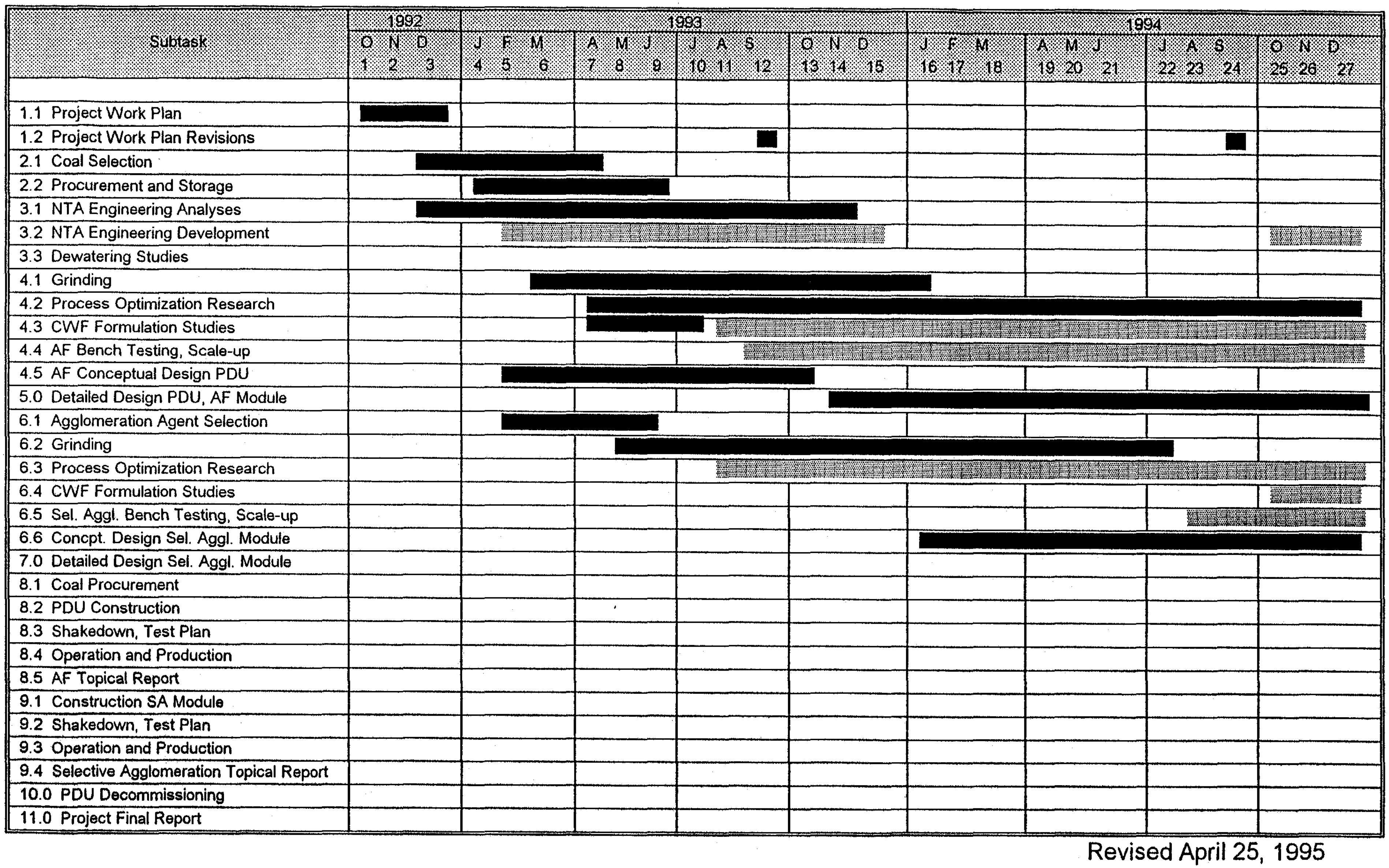
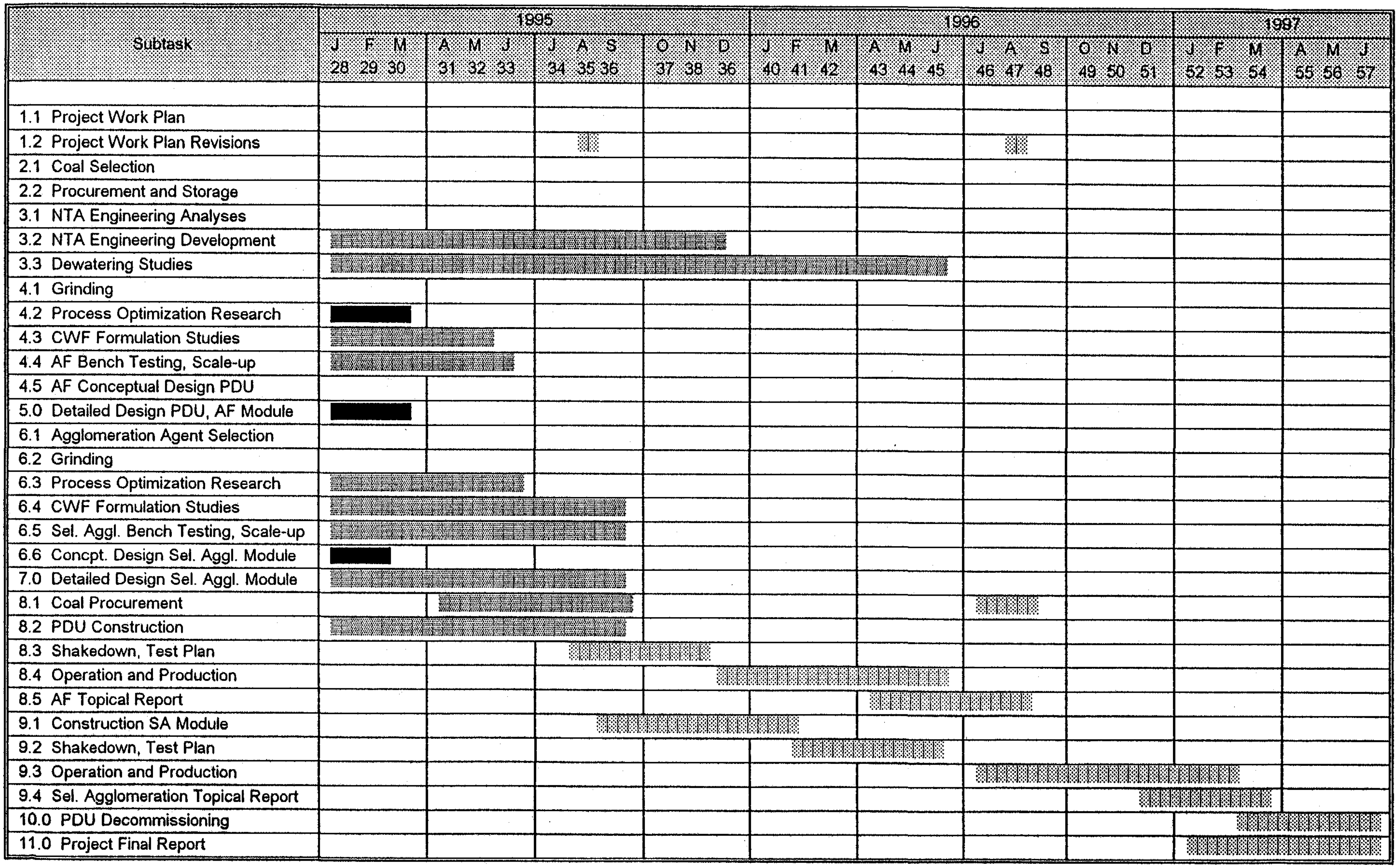


Figure 2. Project Schedule



Revised April, 25 1995

Figure 2. Project Schedule (Cont'd)

ACCOMPLISHMENTS DURING QUARTER

Work was carried out on Tasks 3 through 8 during the Eleventh Quarter (April 1 to June 30, 1995) reporting period. Good progress was made on these tasks as discussed below.

TASK 3 DEVELOPMENT OF NEAR-TERM APPLICATIONS

During 1993 Bechtel performed an engineering analysis evaluating potential applications for column flotation and selective agglomeration at three coal preparation plants operated by what is now Cyprus Amax Coal Company (11). Economic projections favored column flotation over selective agglomeration and an application at the Lady Dunn Preparation Plant was found to be particularly attractive since the plant was being considered for a major capacity expansion. Because of the potential advantages of installing column flotation rather than mechanical flotation cells in the expanded fine coal cleaning circuit, Lady Dunn management was pleased to offer their plant as the study site for a near-term application of column flotation. The Microcel™ flotation column was selected for this study and the Center for Coal and Mineral Processing (CCMP) at Virginia Tech was assigned the responsibility for the on-site column testing under Subtask 3.2 Engineering Development. During the previous reporting quarter, a new subtask 3.3 "Dewatering Studies" was added to the project. This work will also be performed by CCMP.

Subtask 3.2 Engineering Development

As discussed above and in the 10th Quarterly Report, the Lady Dunn Preparation Plant in West Virginia was selected as the host site for testing Microcel™ column flotation recovery of additional coal from minus 100-mesh fines cycloned from the raw coal. The clay content is quite high in this stream at the Lady Dunn Plant and, consequently, coal recovery in the existing mechanical cells is poor. For this reason the application is a good test of the near-term applicability of column flotation in many preparation plants. The Center for Coal and Mineral Processing (CCMP) at Virginia Tech is supervising the test work for the local plant management.

An existing 30-inch diameter Microcel™ test unit was refurbished and installed in the plant for the testing. The system has been calibrated and several successful runs were completed at quarter end. The initial performance met expectations, and once a few minor revisions are made to the equipment, a plan for the parametric testing will be prepared. The main emphasis will be to obtain scale-up information for a full conversion to column flotation.

Subtask 3.3 Dewatering Studies

This new subtask was added during the previous reporting quarter. The work to be performed by Virginia Tech will aim at developing a novel dewatering process for clean coal fines. Three coals will be tested including the product from near-term testing at the Lady Dunn plant (Subtask 3.2). A test plan was prepared and submitted to DOE on April 11, 1995.

TASK 4 ENGINEERING DEVELOPMENT OF ADVANCED FROTH FLOTATION

As described in the Subtask 2.1 report, Coal Selection Plan and Recommendations [12] and in previous quarterly reports [3, 4], six coals were identified as good candidate feedstocks for conversion into premium fuel and were selected for testing during Task 4. The six coals selected are described in Table 2.

The test coals are all washed bituminous coals except for the Dietz coal which is a subbituminous coal that is only crushed before marketing. Washing plant heating value recoveries were in the 89 to 94 percent range for the five bituminous coals. Thus, near 90 percent heating value recoveries are necessary during the advanced flotation step to meet the project goal of recovering 80 percent of the heating value from the raw coals.

Table 2. Test Coals Selected for Project

Coal	Mine	State	HGI	Ash, %	Sulfur, %
Taggart	Wentz	VA	52	2.07	0.62
Indiana VII	Minnehaha	IN	55	9.25	0.49
Sunnyside	Sunnyside	UT	54	5.11	0.63
Winifrede	Sandlick	WV	47	8.42	0.94
Elkhorn No. 3	Chapperal	KY	46	6.04	0.86
Dietz	Spring Creek	MT	41	4.98	0.33

Task 4 activity during this reporting quarter involved work in two areas, Subtask 4.3 Coal Water Fuel (CWF) Preparation, and Subtask 4.4 Bench-scale Testing and Process Scale-up. Results are discussed below.

Subtask 4.3 Advanced Flotation - CWF Formulation Studies

The final version of the Subtask 4.3 topical report [13] was completed and issued to DOE and other project partners on May 23, 1995. The findings of this report indicate that four of the six project coals could be formulated into coal-water slurries with coal loadings varying from approximately 47 to 70%. The coarsest coal (Taggart) was loaded to the highest levels (68-70%) and the finest coal (Indiana VII) to the lowest levels (47-53%). Viscosities of these formulated slurries varied from several hundred to several thousand centipoise at 100 Sec^{-1} depending on the coal type, loading level, and whether or not a stabilizer was used. It was generally found that slurry stabilities were poor unless a stabilizer was used, which invariably increased slurry viscosities dramatically.

While a number of different approaches were evaluated in formulating these slurries, it was determined that the production of a bimodal coal particle size distribution (PSD) resulted in the best slurry properties. It was found that in order to generate the appropriate PSD, selective regrinding of a fine coal fraction was necessary for coarse coals like the Taggart. In contrast, for an already finely ground coal like the Indiana VII, it was necessary to generate a coarse coal fraction, prior to fine grinding, to achieve the appropriate bimodal PSD.

Subtask 4.4 Bench-Scale Testing and Process Scale-Up

The flotation data from the 1-foot column testing were organized and an initial draft of the subtask topical report started. The test work was completed in January, but samples from selected bench-scale tests were retrieved from storage during the quarter for additional analyses, particularly for sulfur and toxic trace elements.

Toxic Trace Elements

The reduction in toxic trace element concentrations accomplished by bench-scale column flotation was studied by assaying the products from the selected parametric tests shown in Table 3. For this analysis, the distribution of the trace elements between the clean coal and waste product were determined. Test 32 was selected despite its low heating value recovery because it represented the best ash reduction achieved for the Winifrede coal.

Table 3. Parametric Tests Utilized for Trace Elements

Test ID	Coal	Nominal Top-Size	Flotation System	Residual Ash lb/mmBtu	Heating Value Rec. %
11	Elkhorn No. 3	62 mesh	Ken-Flote™	1.81	89.1
26	Elkhorn No. 3	200 mesh	Ken-Flote™	1.77	92.8
32	Winifrede	20 μm	Ken-Flote™	2.04	17.1
157	Taggart	62 mesh	Microcel™	1.00	99.5
205	Sunnyside	150 mesh	Microcel™	1.87	98.1
PSU	Indiana VII	325 mesh	Microcel™	2.03	79.9

The toxic trace elements of interest were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine. The analyses were carried out by Huffman Laboratories since they reported consistent results and low detection limits on a previous set of samples [10]. Huffman used perchloric acid decomposition and inductively coupled plasma (ICP) spectroscopy for most of the analyses. Mercury was determined by cold vapor spectroscopy, and chlorine by a total halides method.

The ranges for reported elemental concentrations and the mass balance closures are shown in Table 4. The average of all of the mass balance closures was 102%, and the closures were good (+/-15%) for beryllium, chromium, cobalt, manganese, nickel and chlorine. Mass balances could not be calculated for cadmium since it was detected only in the Elkhorn No. 3 and Indiana VII raw coals and the Indiana VII and Sunnyside fine refuse samples.

The arsenic and mercury balances were off by more than 15% for the Indiana VII separations, and the mercury balances also were off for the Winifrede and the fine Elkhorn No. 3 separations as well. Mercury was not detected in the Sunnyside clean coal so a balance could not be calculated for that particular coal. The selenium balances were off for the Taggart and the coarser Elkhorn No. 3 separations. Repeat analyses are being obtained to resolve the arsenic, mercury, lead and selenium mass balance discrepancies. For this reason analyses of individual samples will be reported at a later time.

The only satisfactory antimony mass balance was for Winifrede coal, a coal which did not separate well in the flotation column. Because the discrepancies are so widespread, it appears that there is a bias in the antimony analytical procedure which will make repeat assaying of little value. Huffman suspects that there is a solubility problem at low concentrations and that it may not be feasible to quantify antimony concentrations below 1 ppm by the perchloric acid dissolution/ICP procedure.

The reductions in the various trace element concentrations accomplished during the column flotation tests were calculated on a heating value basis. The range of reductions from the raw coal and from the washed coal flotation feed are shown in Table 5. There are a few discrepancies in the data which are being resolved by the repeat analyses, but the concentrations of arsenic, beryllium, chromium, cobalt, manganese, mercury and selenium in the raw coal were clearly reduced by the combined washing and advanced cleaning steps. Much of the reduction was accomplished by the preparation plant at the mine, but very definitely the column flotation step reduced the concentrations of arsenic, chromium, manganese, and nickel remaining in the washed coal from these seams.

Table 4. Trace Element Concentration Results

	Trace Element Concentration, ppm										Mass Balance Closure, percent		
	Raw Coal		Flotation Feed		Clean Coal		Fine Refuse		Calculated Flotation Feed				
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Average
Antimony (Sb)	0.03	1.2	0.04	1.4	0.07	2	0.17	0.81	0.08	1.7	98	190	137
Arsenic (As)	0.83	7.3	0.32	6.5	0.15	2.5	2.8	23	0.3	3.7	46	103	86
Beryllium (Be)	0.8	3.2	0.8	3.3	0.8	2.9	0.9	3.2	0.8	3.2	96	107	101
Cadmium (Cd)	< 0.1	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.3	< 0.1	0.15	n. d.	n. d.	n. d.
Chromium (Cr)	9.2	29	6.4	56	4.2	28	52	130	5.7	51	88	105	95
Cobalt (Co)	2.2	11	1.4	9.2	1.3	9.2	4.8	26	1.5	9.7	96	105	102
Lead (Pb)	2	12	< 1	6	2	10	3	10	1.9	6..3	78*	107*	98*
Manganese (Mn)	27	200	7	33	4	14	14	360	7.3	35	77	107	95
Mercury (Hg)	< 0.01	0.03	< 0.01	0.05	< 0.01	0.04	0.01	0.09	< 0.01	0.03	50**	158**	93**
Nickel (Ni)	1.7	30	7.7	38	3.3	36	23	240	7.0	39	87	110	98
Selenium (Se)	0.78	5.9	0.67	5.9	0.71	5.2	0.76	9.4	0.8	5.3	86	167	114
Chlorine (Cl)	38	1180	63	1200	68	1240	46	880	68	1186	98	108	102
											Grand Average:		**102**

"<" = Below indicated detection limit.
"n. d." = not determined
* For four of six tests
** For five of six tests

Table 5. Reduction in Toxic Trace Element Concentration, HHV Basis

	From Raw Coal, %			From Flotation Feed, %		
	Minimum	Maximum	Average	Minimum	Maximum	Average
Antimony (Sb)		no meaning			no meaning	
Arsenic (As)	75	84	80	11	76	46
Beryllium (Be)	13	59	39	-5	18	5
Cadmium (Cd)	n. d.	n. d.	n. d.	n. d.	n. d.	n. d.
Chromium (Cr)	20	87	63	35	53	44
Cobalt (Co)	37	74	53	-1	22	11
Lead (Pb)	-48	75	43	-134	28	-16
Manganese (Mn)	75	98	92	43	77	62
Mercury (Hg)	1	47	32	-87	63	13
Nickel (Ni)	-69	42	5	12	58	33
Selenium (Se)	31	76	49	-62	23	1
Chlorine (Cl)	-19	34	13	-8	1	-2

Notes: Negative sign indicates increase in element lb/mmBtu concentration
"n. d." = not determined

TASK 5 DETAILED DESIGN OF PDU AND ADVANCED FLOTATION MODULE

Clean Coal Conveyor Drawings (400-T-03 & 400-T-04)

The following drawings were received from Rocky Mountain Conveyor, Inc. and "Approved for Construction" by both Bechtel and Entech Global Engineers:

1. Drawing #1, REV 0, 57 inch x 24 foot conveyor;
2. Drawing #2, REV 0, 57 inch x 24 foot conveyor;

Detailed Steel and Grating Drawings

Drawings of the PDU's detailed steel and grating were reviewed and "Approved for Construction" by both Bechtel and Entech Global, Inc. engineers.

Fine Grinding Mill Media Evaluation

Because previous testing has shown that elemental metal concentrations in coal slurries may increase when ground with steel balls / beads, an evaluation was conducted to determine the effects of media type on slurry metal contamination. Two media types were evaluated - steel and glass. Samples of washed coal were ground for a set time period with each media type and sent to Huffman Laboratories for analysis.

These analyses show that significant chromium, iron, and manganese contamination of coal occurs when using steel beads. The test results are shown in Table 6.

Table 6. Grinding Media Evaluation - Contamination During grinding

Media	Head - N/A	Steel (3mm)	Steel (3mm)	Glass (3mm)	Glass (3mm)
Grind Time	N/A	15 min	60 min	15 min	60 min
Ash (%)	10.04	10.75	11.34	9.53	9.63
Chromium - ppm	29	32	60	28	28
Iron - ppm	3,770	8,520	13,000	3,740	4,000
Manganese - ppm	42	74	202	42	44
Nickel - ppm	63	66	65	55	54
SiO_2 (%)	5.63	5.93	5.88	5.62	5.60

The coal slurries ground in both steel and glass media were then subjected to advanced cleaning via froth flotation and selective agglomeration. The clean coal and tailings products were then analyzed to determine if there are any effects of media type on product quality or yield.

Lab analysis reveals that significant chromium, iron, manganese, nickel, and silicon reduction occurs when cleaning the ground product. In addition, the overall concentration of these elements is significantly lower when using glass beads as opposed to steel. The test results are shown in the Table 7.

Table 7. Grinding Media Contamination Removed During Cleaning

Media	Cleaning	Stream	Ash	Yield	Cr (ppm)	Fe (%)	Mn (ppm)	Ni (ppm)	Si (%)
Steel		Feed	10.75	100.00	60	1.30	202	65	5.88
Glass		Feed	9.63	100.00	28	0.40	44	54	5.60
Steel	Flotation	Clean	7.03	25.15	24	0.78	79	53	1.29
Steel	Flotation	Tails	12.00	74.85	18	0.66	76	41	1.98
Glass	Flotation	Clean	3.98	32.58	15	0.21	24	50	0.78
Glass	Flotation	Tails	12.36	67.42	16	0.36	37	44	2.78
Steel	Agglom	Clean	3.08	90.97	15	0.66	49	54	0.41
Steel	Agglom	Tails	88.04	9.03	40	3.95	570	66	22.20
Glass	Agglom	Clean	2.53	91.58	13	0.17	22	50	0.52
Glass	Agglom	Tails	86.85	8.42	27	2.54	220	54	23.80

As a result of this test work, 3-mm glass media was selected for use in the Netzsch fine grinding mill.

TASK 6 ENGINEERING DEVELOPMENT OF SELECTIVE AGGLOMERATION

Task 6 activity during this reporting quarter focused on the Subtask 6.3 Laboratory-scale Process Optimization Research, the Subtask 6.4 CWF Formulation Studies, and the Subtask 6.5 Bench-scale Testing and Process Scale-up.

Subtask 6.3 Process Optimization Research

A draft of the Subtask 6.3 topical report [14] was submitted to DOE and project team partners on June 30, 1995, for review and comments. The draft report presented the results of laboratory testing optimization of the continuous selective agglomeration process using pentane and heptane as reusable bridging liquids. The effects of varying feed rates/retention times and mixer speeds on performance were evaluated as well. The target ash specification of 2 lb ash/MBtu) was met for each of the test coals when they were pre-ground to the required particle size distribution (PSD) for mineral liberation. Heating value recoveries generally exceeded 95% over a wide range of operating conditions.

Operation of a unitized high/low shear reactor was compared with operation of a comparable system with separate high- and low-shear mixing stages. Performance and energy consumptions of the two systems were found to be similar. Pre-cooling the slurry was necessary when using pentane as the bridging liquid in order to avoid boiling in the unitized reactor. The amount of bridging liquid required for agglomeration ranged from 18-34% of the weight of coal depending upon the PSD of the slurry. Minus 325-mesh high volatile C Indiana VII slurries required the use of an activator (preferably asphalt) along with the bridging liquid, and minus 325-mesh subbituminous Dietz coal required acidification as well as an activator. Minus 62- to minus 150-mesh high volatile A Taggart, Elkhorn No. 3 and Sunnyside and minus 20-µm high volatile A Winifrede bituminous coal slurries all agglomerated well without an activator.

Comparison testing with diesel fuel and kerosene indicated that it would not be cost-effective to agglomerate with bridging liquids that would not be recovered from the product and reused.

Subtask 6.4 CWF Formulation Studies

Work completed on Subtask 6.4 during this reporting quarter involved preliminary evaluation of Subtask 6.5 selective agglomeration products for CWF formulation. To this end, steam stripped product samples of the Indiana VII and Sunnyside coals were filtered and prepared for slurry formulation. A base Sunnyside slurry, approximately 55% solids, was formulated from the as-received (no additional grinding) steam stripper product.

Subtask 6.5 Bench-Scale Testing and Process Scale-up

The Subtask 6.5 test work progressed significantly during this eleventh reporting quarter of the project. Construction of the 25 lb/hr bench-scale unit was completed along with start-up and shakedown testing of the system.

A number of test conditions were evaluated in the 25 lb/hr selective agglomeration test unit. This work was carried out utilizing three different coals ground to the sizes indicated below:

- Taggart coal - 62 mesh topsize
- Sunnyside coal - 100 mesh topsize
- Sunnyside coal - 150 mesh topsize
- Indiana VII coal - 325 mesh topsize

The bulk of the test work completed to date in this test unit was for the purpose of confirming the 2 t/hr PDU selective agglomeration module design. Parametric testing of those coals to be processed in the PDU will be completed during future testing.

A de-aromatized commercial grade heptane was used as the bridging liquid for all but one of the continuous agglomeration tests carried out this quarter. A pure grade of heptane was utilized for one 150-mesh topsize Sunnyside coal test.

Design and Construction

The design and construction of the 25 lb/hr selective agglomeration bench-scale unit was completed during the previous reporting quarter. Salient features of this system are shown in the block flow diagram, Figure 3.

Unit Operability

Feed System - The coal feed system to the agglomeration circuit appears to be operating well with no plugging problems experienced to date. It was found, however, that during typical testing, the coal slurry feed flowrate varied approximately 5% from the beginning to the end of a run. The net effect of this phenomenon is a slight increase in the heptane/coal ratio during the test.

High Shear - The high-shear reactor was found to work well over a wide range of feed conditions achieving inversion at various coal feed rates and impeller tip speeds.

Low Shear - Depending on the impeller tip speed, residence time, and heptane/coal ratio utilized, the low shear product varies in size from approximately 0.5 mm to 5 mm. It has been found that the agglomerate size can be controlled to some extent by the heptane dosage used, with higher dosages resulting in larger agglomerates. No other clear trends have been confirmed for this unit operation.

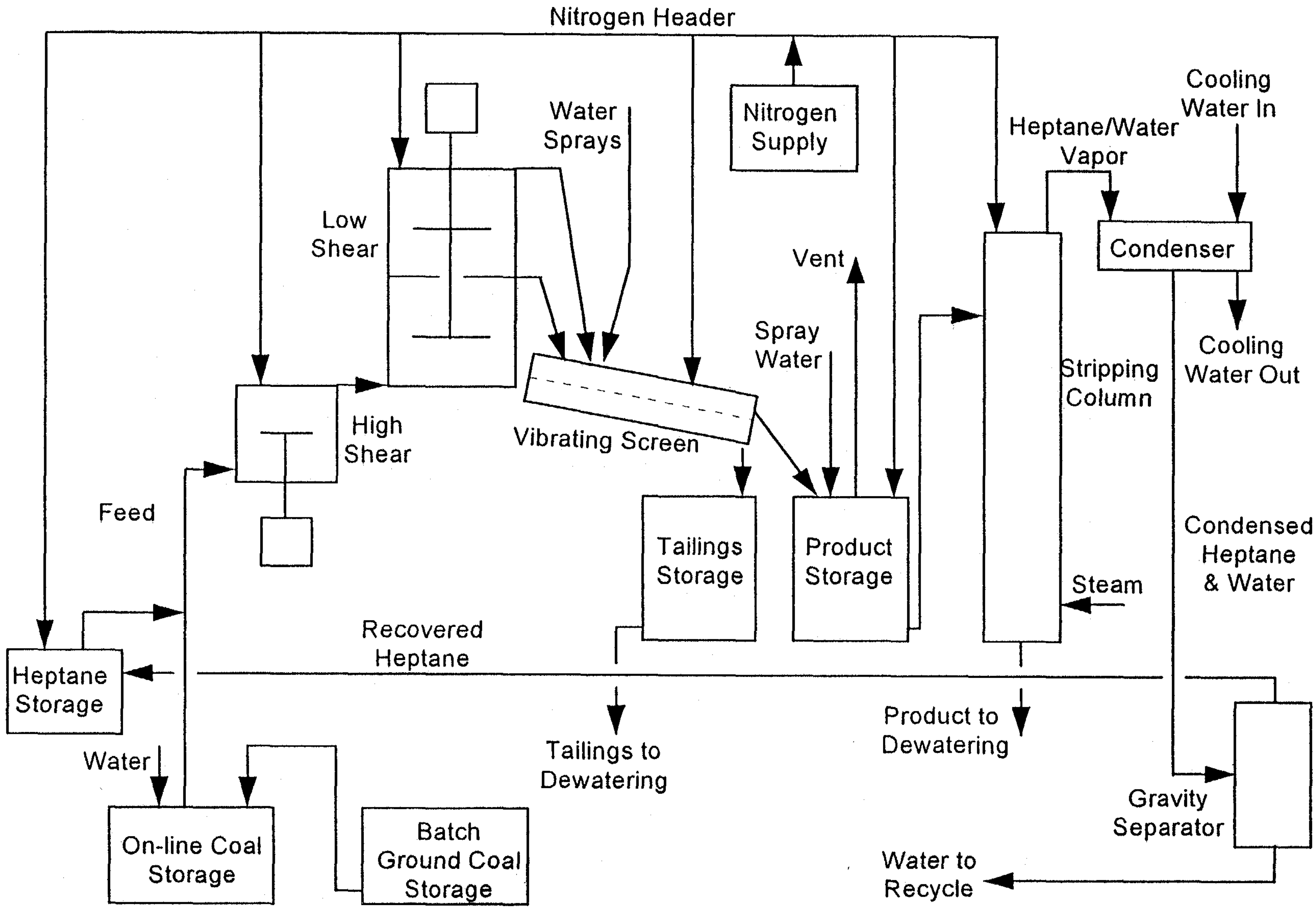


Figure 3. Bench-Scale Agglomeration Process Flow Diagram

Vibrating Screen - Operation of the vibrating screen for recovery of the low-shear product seems to be working well. Only minimal blinding of the 48-mesh screen deck has been observed to date, with more blinding occurring as the heptane dosage is increased. The screen is typically operated at an angle of approximately 38° and provides good agglomerate flow across the screen deck.

The screen product varies in moisture content depending on the size of the agglomerates. The smallest agglomerated products (<0.5 mm) generally contain about 35% coal 55% water, and 10% heptane. In contrast, the larger agglomerated products (2-4 mm) contain around 50% coal, 35% water, and 15% heptane. Generally, the larger agglomerated products contain less residual ash, due primarily to less carryover of mineral-matter bearing process water to the product, i.e., the larger well formed agglomerates drain their associated water easier.

Stripper Circuit - During the previous quarter, difficulty was encountered feeding agglomerated product to the steam stripping circuit. This problem was two-fold with both mixing and pumping proving difficult. The pumping problem was resolved by the use of a peristaltic pump utilizing a 3/8" diameter hose. This pump provides feed to the stripper without plugging since its small line size and relatively high flow velocities overcome the agglomerates tendency to float and plug the line. It was found that this peristaltic pump could provide feed flows as low as about 0.8 liters per minute.

Due to their buoyancy, heptane based agglomerates have proven very difficult to mix, with an increasing solids concentration gradient from bottom to top present under all mixing conditions evaluated. As such, solids concentrations of approximately 10-12% are the highest achieved for feed to the continuous stripper circuit. This is achieved by pulping agglomerates to approximately 25% solids and mixing them with a 4-Hp mixer in a 55-gallon drum.

With the resolution of these feed problems, operation of the steam stripper has gone smoothly. The stripping column was easily operated in one of two modes as desired. One mode of operation has been to maintain a liquid level in the flooded column to evaluate various stripping residence times. Conversely, a very low level is utilized to evaluate a continuous steam phase system, through which the feed slurry flows. For either of these scenarios, 5/8" stainless steel pall rings are used as column packing.

Condenser - Operation of the condenser, which condenses the evaporated heptane and its associated water vapor has proceeded smoothly. It appears that the condenser has sufficient capacity to achieve the goals easily. During typical operations to date, the stripper vapor product temperature averages about 203°F and the condensate temperature about 50°F. It has been found that under most stripper operating conditions evaluated, some coal is carried over to the condenser. This phenomenon typically does not cause any plugging problems and will be addressed by the use of a de-mister pad in the 2 t/h PDU system.

Gravity Separator - Once condensed, the water and heptane mixture enters the center of the gravity separator column. Operation of this separator has proceeded smoothly with the water/heptane interface easily observed. Utilizing a U-tube discharge of the water from the bottom of the separator, steady-state operation has been achieved in which consistent flows of both water from the bottom and heptane from the top can be observed. Preliminary analysis of this recovered heptane indicates that it is virtually identical in composition to the fresh heptane. Analysis of water samples taken from the separatory column indicate a total organic content of approximately 8 ppm, indicating minimal solubility of the heptane in water at the temperature of these condensed liquids.

Taggart Coal Continuous Agglomeration Results

Initial agglomeration system start-up was carried out utilizing the Taggart coal ground to a 62-mesh topsize. This Taggart coal work was carried out primarily to determine system operability and to provide guidance in developing operating procedures. As such, there has been no parametric testing carried out with the Taggart coal to date. Additional testing with the Taggart coal will be carried out later in the test program.

In many tests carried out with this coal, low-shear reactor plugging problems prevented the acquisition of the appropriate samples and completion of the tests as planned. A total of six complete test runs (where a full compliment of samples were obtained) were completed using this coal. The following conditions were held constant for these six tests, the results of which are shown in Table 8:

- Commercial grade heptane
- 4.8-inch (4-blade) low-shear impeller
- 48-mesh screen deck
- Screen inclination of 38°
- 2.0% feed coal ash

It should be noted that the product and tailings ash values, as well as performance calculations, utilized composited samples from froth skimming simulation. This procedure involves reporting the material which floats in the tailings sample (5 gallon) as part of the product. The effect of this procedure is seen in the difference between agglomeration yield with and without froth skimming. As this data indicates, both the yield and Btu recovery increase by as much as 2.5% when froth skimming is utilized. The effect of this increased yield on the final product ash content is minimal due to the relatively small quantity of additional recovered material.

As expected, these test results indicate that for this Taggart coal, product ash specifications can be easily met while achieving very high product yields and Btu recoveries. For the first four tests shown in Table 8, no wash water was used on the screen to help reduce the amount of coal lost to the tailings. Even under these conditions, product ash specifications were achieved, with the use of wash water providing additional reduction in product ash content.

Table 8. Taggart Coal Continuous Agglomeration Results

	System Configuration							Feed			
	High Shear			Low Shear			Screen	Solids		Heptane	
Test ID	Imp in	RT min	Tip m/s	RT min	Tip m/s	Vol H/F	H20 lb/hr	%	lb/hr	%, db	%, maf
T1	2.4	1.6	15.0	3.8	9.6	Half	0	10	22.7	26.0	26.5
T2	2.4	0.7	15.0	3.7	4.1	Full	0	10	49.2	24.4	24.8
T3	2.4	0.7	15.0	3.7	1.9	Full	0	10	49.2	26.2	26.8
T4	3.6	0.7	11.5	1.7	6.4	Half	none	5	25.1	23.5	24.0
T5	3.6	0.7	11.5	1.7	6.4	Half	med	5	25.1	23.5	24.0
T6	3.6	0.7	11.5	1.7	6.4	Half	full	5	25.1	23.5	24.0

			W/out Froth Skim			With Froth Skimming						
			Prod	Agg Perform		Product		Tails	Agg Perform		ROM Perform	
Test ID	% Sol	Size (mm)	% ash	Yield %	Btu Rec, %	% ash	#ash/ MBtu	% ash	Yield %	Btu Rec, %	Yield %	Btu Rec, %
T1	54.2	1-3	1.6	96.5	96.9	1.66	1.09	36.6	99.0	99.4	63.4	93.7
T2	49.2	1	1.5	99.1	99.7	1.47	0.97	71.9	99.3	99.9	63.5	94.2
T3	45.6	2-4	1.5	99.4	99.9	1.51	0.99	76.1	99.4	99.9	63.6	94.2
T4	47.9	NA	1.5	96.4	96.9	1.47	0.97	46.0	98.8	99.4	63.2	93.7
T5	37.6	NA	1.5	96.5	97.1	1.47	0.97	38.6	98.6	99.2	63.1	93.5
T6	38.2	NA	1.4	96.7	97.4	1.40	0.92	35.5	98.2	98.9	62.9	93.3

High Shear - *As part of testing completed for the Taggart coal, an evaluation of high-shear mixing requirements to achieve inversion was carried out.* Two different high-shear impellers were evaluated during this testing. One was a 2.4-inch diameter impeller with 4 blades, the other a 3.6-inch diameter impeller with 6 mixing blades. Testing was completed to determine the minimum high-shear mixing rpm required to achieve inversion under various coal feed rates and solids concentrations. Table 9 provides a brief summary of this data.

Several trends can be observed from this data:

- As residence time in the high-shear vessel decreases with increasing coal feed rate, the impeller tip speed must be increased to maintain inversion. This trend is expected since at shorter residence times, higher power input is required to maintain similar energy/coal ratios.
- As the slurry solids concentration increases, less power input (lower tip speed) is required to achieve inversion. This trend is also expected since

as the solids concentration increases, more particle to particle contact occurs at similar energy inputs.

- The 2.4-inch impeller draws less power to achieve inversion than the 3.6-inch impeller. This is attributed to the fact that the smaller impeller only has 4 blades while the larger impeller has 6 blades, thus requiring more power to achieve similar tip speeds.

Table 9. Taggart Coal - High Shear Requirements for Inversion

Impeller Diameter (in)	Solids Conc. (%)	Coal Rate (lb/hr)	Percent Heptane (dcb)	Residence Time (sec)	Tip Speed (m/s)	Power Draw* (amps)
2.4	10	25	24.5	88	7.2	1.3
		50	24.4	44	8.8	1.3
		92	24.5	24	11.2	1.5
	15	50	20.5	65	8.3	1.4
3.6	5	25	25.0	44	11.0	2.2
	10	25	24.2	87	7.7	1.8
		48	25.0	44	9.6	2.0
	18.4	24	25.3	160	7.2	1.7
		50	24.4	80	7.7	1.7

* Indicates relative power consumption only

Based on these test results, only minimal additional test work was completed using the 6-bladed 3.6-inch high-shear impeller.

Low Shear - Evaluation of the low-shear unit operation proved difficult through virtually all of the startup testing with the Taggart coal. In particular, the effect of low-shear residence time and impeller tip speed on product size was difficult to determine due to plugging of the low shear discharge. It was determined that when the low shear product approaches 4-6 mm in size, it ceases to flow through the 1-inch discharge port. This lack of flow is attributed to the fact that agglomerates 4-6 mm in diameter approach a critical size (1/4 to 1/6 of the discharge port diameter), preventing exit from the vessel.

One additional factor believed to contribute to the initial poor discharge from the low-shear vessel was that the overflow discharge arrangement withdrew from a stagnant section of the vessel, i.e., from the top surface, where floating material tends to accumulate. To remedy this problem, the discharge ports were lowered such that they were located below the liquid surface. An up-flowing discharge pipe was then used to maintain the desired level. While this modification did result in flow improvement, some discharge plugging still occurred.

The following general conclusions concerning the low-shear operation were made based on these initial Taggart coal test runs:

- At heptane dosages required to achieve inversion during high shear (24-26% on a dry ash free coal basis), the low shear product grows to large sizes (4-6 mm diameter) at the targeted impeller tip speeds of 3-5 m/s.
- Higher impeller tip speeds (7-10 m/s) were required to maintain agglomerates of 2-3 mm in size, i.e., agglomerates that discharged easily from the low-shear vessel. Apparently some breakage of the agglomerates occurs under these conditions.
- Low shear discharge plugging problems are more pronounced at higher solids concentrations (particularly above 10%). This trend is expected since at higher solids concentrations more particle to particle contact is expected for the same energy input. Also, at the same volumetric flowrate through the system, a higher solids loading increases the solids flow through the discharge port, which increases the chance for plugging.

Vibrating Screen - Product recovery on the vibrating screen proved to be adequate during typical operating conditions utilizing the 62-mesh topsize Taggart coal. There was however, some coal lost to the screen underflow (tailings) under all operating conditions. It was determined that both the amount of wash water used and the nature of the spray pattern utilized, influenced the amount of coal in the screen underflow. Higher water rates and stronger (more forceful) spray patterns both resulted in more lost coal. As such, finer spray pattern (misting) nozzles were obtained and utilized for all testing carried out with subsequent coals.

A froth skimming unit operation will be utilized in the PDU to recover coal from the screen tailings. This recovered coal will then be combined with the screen overflow (product) stream. To simulate this operation at the bench scale, tailings samples were routinely separated, with the floating material reported as clean coal and the settled solids considered the actual tailings. In this manner, more representative samples were composited to allow calculation of overall test performance.

Additional testing carried out to evaluate vibrating screen wash water requirements involved the batch screening of low-shear product samples with various wash water rates. This work was completed to determine if substantially greater quantities of wash water would decrease the product ash content. Results of these batch screen tests are shown in Table 10.

Table 10. Taggart Coal - Low-shear Product Rinsing

	Drain No Rinse	Spray Rinse	Soak Rinse
Ash content, %	1.57	1.51	1.51
	1.52	1.48	NA
	1.51	1.47	1.46
	1.47	1.43	1.43
	1.41	1.40	1.38

These tests were carried out by placing a 1-liter sample of low-shear product on an 8-inch diameter, 48-mesh test sieve. Samples of this product were then taken for ash analysis after each of the following:

- Allowing the water to drain from the bed (no rinsing)
- Rinsing the bed lightly with a spray bottle, using approximately 500 ml of water
- Soaking the bed with a gentle water stream for 2 minutes

As this data indicates, some product washing (as represented by 500 ml spray rinsing) was found to reduce product ash by approximately 0.04%. However, this data indicates that additional washing beyond a spray rinse did not reduce the product ash content substantially. For a low ash, relatively coarse coal like the Taggart, these results are not unexpected.

Sunnyside Coal Continuous Agglomeration Results

During this reporting quarter, a total of 18 complete agglomeration test runs were carried out utilizing Sunnyside coal. Of these 18 test runs, 4 were carried out at a 100-mesh topsize grind, and the remaining 14 at a 150-mesh topsize grind. Results for these complete tests, i.e., tests in which a full compliment of samples were obtained, are shown in Table 11. It should be noted that the following conditions were held constant for all of these Sunnyside coal tests:

- 2.4-inch (4-blade) high-shear impeller
- 4.8-inch (4-blade) low-shear impeller
- 48-mesh screen deck
- Screen inclination of 38°

It should also be noted that as for the Taggart coal results, the product and tailings ash values, as well as performance calculations, utilize composited samples from froth skimming simulation. This procedure involves reporting the material which floats in the tailings sample as part of the product. As with the Taggart coal, this additional material included as part of the product, represents a small portion of the total feed coal, generally less than 2%. As such, while 1-2% improvements in Btu Recovery are realized, virtually no increase in product ash is observed.

For the four 100-mesh topsize Sunnyside coal test runs completed, the product ash specification of 2 lb ash/MBtu was met for 2 of them, with the other two considered borderline. As with the Taggart coal testing reported above, agglomeration yields and Btu recoveries were very high, generally in the 96 and 98% ranges, respectively. The yield reduction, from the 98-99% achieved with the Taggart coal, to 95-96% for the Sunnyside coal is expected since the Sunnyside feed coal ash content is approximately 5.5%, as compared to 2% for the Taggart coal.

Table 11. Sunnyside Coal Continuous Agglomeration Results

		System Configuration				Feed				Product		W/out Froth Skim			With Froth Skimming						
	Top	High Shear		Low Shear						Product		Prod	Agg Perform		Product		Tails	Agg Perform		ROM Perfor	
	Size	RT	Tip	RT	Tip	Solids		Heptane		%	Size	%	Yield	Btu	%	#ash/	%	Yield	Btu	Yield	Btu
Run	Mesh	min	m/s	min	m/s	%	lb/hr	%, db	%, daf	Sol	(mm)	ash	%	Rec, %	ash	MBtu	ash	%	Rec, %	%	Rec, %
S1A1	100	1.5	14.0	3.5	8.0	10	25.2	24.8	26.2	50.0	1-2.5	2.88	94.2	96.6	2.89	2.01	57.1	96.0	98.4	76.0	88.7
S1A2	100	0.7	22.3	1.8	8.0	5	24.4	28.4	29.9	40.5	1	2.75	95.7	98.1	2.75	1.91	60.2	96.3	98.7	76.2	88.9
S1A3	100	1.1	19.1	2.5	6.1	7	24.5	26.4	27.7	49.4	2-2.5	2.58	94.9	97.4	2.59	1.79	55.4	95.8	98.3	75.9	88.6
S1A4	100	1.5	13.4	3.5	8.0	13	32.7	24.2	25.5	50.9	1-3	2.97	95.3	97.5	2.97	2.07	55.3	96.3	98.5	76.3	88.7
S2A5	150	1.5	12.1	3.5	8.0	10	24.5	26.3	27.9	47.4	.5-4	2.74	93.6	96.9	2.76	1.91	61.9	95.0	98.3	75.2	88.6
S2A6	150	1	16.0	2.4	6.1	7	25.1	26.5	28.0	51.5	2	2.53	95.0	98.2	2.54	1.75	64.1	95.5	98.6	75.6	88.8
S2A7	150	1.5	11.8	3.6	8.0	13	30.4	26.7	28.3	51.1	1	2.76	94.8	97.9	2.80	1.94	64.4	95.6	98.7	75.7	88.9
S2A8	150	1.4	13.1	3.4	8.0	10	25.4	25.1	26.5	41.2	.5-4	2.93	95.5	98.4	2.93	2.04	71.6	96.3	99.2	76.2	89.4
S2A9	150	1.4	18.0	7.3	4.8	10	26.1	22.3	23.6	33.0	.5	2.86	92.1	94.9	2.86	1.98	64.0	95.8	98.7	75.9	88.9
					3.2						.5-P										
S2A10	150	1.4	18.0	7.3	3.2	10	25.8	23.9	25.3	30.7	.5-P	2.76	92.9	95.8	2.77	1.92	64.7	95.8	98.7	75.9	89.0
S2A11	150	1.4	18.0	7.3	4.8	10	25.6	24.1	25.5	64.1	3-3.5	2.55	NA	NA	2.55	1.76	NA	NA	NA	NA	NA
S2A12	150	1.5	18.0	7.4	4.8	10	24.7	25.0	26.5	57.8	3	2.62	95.0	98.2	2.62	1.82	64.6	95.3	98.6	75.5	88.8
S2A13	150	0.7	20.4	3.8	4.8	10	48.9	25.9	27.3	50.0	.5-1.5	2.79	94.5	97.4	2.79	1.93	66.7	96.0	98.9	76.0	89.1
S2A14	150	1	19.1	4.8	4.8	10	37.8	25.1	26.5	52.0	1.5	2.54	95.7	98.9	2.54	1.76	71.2	95.9	99.1	75.9	89.3
S2A15	150	1.4	15.3	3.4	4.8	10	24.9	25.9	27.4	51.6	1.5-2	2.5	94.7	97.8	2.51	1.73	59.0	95.1	98.2	75.3	88.5
										38.7	.5	2.82	95.1	97.9	2.82	1.96	61.1	95.8	98.6	75.9	88.8
S2A16	150	1.4	14.7	3.4	8.0	13	32.9	24.7	26.0	50.5	1	2.42	95.2	98.1	2.42	1.67	61.9	95.6	98.5	75.7	88.8
S2A17	150	1.4	14.7	3.4	6.4	13	32.9	24.7	26.0	58.3	1.5	2.41	94.8	97.7	2.42	1.67	57.7	95.2	98.2	75.4	88.5
S2A18	150	1.5	15.3	7.3	4.8	10	24.5	25.6	27.0	56.6	1.5	2.37	94.5	97.7	2.38	1.64	59.4	95.0	98.2	75.2	88.5

Results for these four 100-mesh tests indicate that lower product ash contents were achieved when operating at 5 and 7% solids concentrations than when operating at 10 and 13% solids concentration. It should be noted, however, that other operating conditions varied also, in particular, for the two lower solids concentration tests, higher heptane ratios were utilized.

All except one of the 150-mesh topsize Sunnyside tests carried out achieved the product ash specification of 2 lb/MBtu as anticipated. The one test which did not achieve this specification (S2S8) utilized pure rather than commercial grade heptane and achieved the highest Btu recovery of all the tests. The bulk of these tests were completed at a 10% solids concentration with one test carried out at 7% solids and 3 tests at 13% solids.

While various operating conditions changed from test to test, it should be noted that the lowest product ash contents were achieved when relatively large (2-4 mm) monosized agglomerates were produced. This indicates that a product with these characteristics has a lower water content, rejecting more of the mineral matter bearing process water.

It should be noted that heptane dosage appeared to have a negligible effect on product ash content for all of these tests. However, it should also be noted that only small ranges of heptane/coal ratio can be evaluated to insure operability of the system, i.e., too little heptane prevents agglomerate formation while too much heptane results in an un-screenable oily mass.

Considering the last four Sunnyside agglomeration tests completed (S2A15 through S2A18), two of them (S2A16 and S2A17) were completed at 13% solids while the other two (S2A15 and S2A17) were carried out at 10% solids. The two tests carried out at 13% solids achieved very low product ash levels. This is attributed to the formation of well formed monosized agglomerates (1-1.5 mm) during low shear, which provide good drainage of mineral-matter bearing process water during screening.

This effect of agglomerate size versus ash content is evident for the results of Test S2A15. During this test, typical cyclic growth and reduction of agglomerates (as described later under low shear) occurred during low shear. As such, the product and tailings were sampled twice during this test, once when well formed 1.5 to 2.0 mm agglomerates were formed, and once when 0.5 mm agglomerates were produced. As expected, the ash content of the 0.5 mm agglomerates was higher (2.82%) than the 1.5-2 mm agglomerates (2.51%). Also as expected, the solids concentration of the well formed agglomerates was much higher (51.6%) than that of the smaller 0.5 mm agglomerates(38.7%).

The final Sunnyside coal test completed during this reporting period (S2A18), achieved the lowest product ash content to date, 2.38% (1.64 lb ash/MBtu).

High Shear - As part of the Sunnyside coal testing completed to date, an evaluation of high-shear mixing requirements to achieve inversion was carried out. As with the

Taggart coal, two different high-shear impellers were evaluated during this testing. One was a 2.4-inch diameter impeller with 4 blades, the other a 3.6-inch diameter impeller with 6 mixing blades.

Testing was completed to determine the minimum high-shear mixing rpm required to achieve inversion under various coal feed rates and solids concentrations. Table 12 provides a brief summary of this data, confirming the trends observed for the Taggart coal high-shear unit operation evaluation.

Table 12. Sunnyside Coal HS Requirements for Inversion

Impeller Diameter (in)	Solids Conc (%)	Coal Rate (lb/hr)	% Hept (maf)	Res Time (sec)	(rpm)	Tip Speed (m/s)	Power Draw* (amps)
Sunnyside (100 Mesh)							
2.4	5	25.0	28.3	44	7000	22.3	2.4
	7	24.9	26.5	62	6000	19.1	2.2
	10	25.0	26.2	87	3800	12.1	1.6
	10	24.8	26.5	88	4400	14.0	
	10	37.4	25.2	58	5000	16.0	
	10	37.4	26.5	58	5200	16.6	
	13	33.0	24.5	86	4200	13.4	1.6
	15	37.4	25.7	87	3800	12.1	1.5
	15	37.4	22.8	86	3800	12.1	1.5
3.6	10	25.0	25.6	87	3000	14.4	2.8
	10	20.2	24.7	108	2500	12.0	2.3
	15	25.5	25.5	127	2200	10.5	2.0
Sunnyside (150 Mesh)							
2.4	7	25.2	26.9	61	4800	15.3	1.8
	10	24.9	26.5	87	4300	13.7	1.7
	13	32.7	25.3	86	3600	11.5	1.4

* Meant to provide a relative power draw only

To help evaluate energy requirements during the high-shear unit operation, high shear inlet and outlet temperatures were monitored during testing. Table 13 presents high-shear operating conditions along with the recorded slurry temperature rise through the high-shear vessel.

Low Shear - As discussed for the Taggart coal, low shear testing to date has indicated that at the anticipated impeller tip speeds of 3-5 m/s, the product agglomerates grow to >4-6 mm in diameter, i.e., to the critical size which plugs the low shear discharge. As such, the low-shear mixer has been operated at 7-10 m/s tip speeds to prevent excessive growth. At these higher impeller tip speeds, the low shear growth typically proceeds in a cyclic manner as follows:

1. From start-up, approximately 20 minutes is required to form spherical 1-mm agglomerates in the low shear discharge.
2. During the next 20 minutes of operation, the product remains monosized as it grows to approximately 3-4 mm in diameter.
3. During the next 20 minutes, smaller agglomerates (<0.5-1.0 mm) start to appear and eventually constitute the majority of the product.
4. These small agglomerates then grow to 3-4 mm in size once again repeating the cycle.

Table 13. Slurry Temperature Rise Through High Shear

Test ID	% Sol	Coal lb/hr	% Hep maf	Res Time min	HS* rpm	Tip Speed* m/s	Δ T °F
S1A1	10.1	25.2	26.2	1.5	4400	14.0	4.5
S1A2	5.0	24.4	29.9	0.7	7000	22.3	6.1
S1A3	7.1	24.5	27.7	1.1	6000	19.1	6.5
S1A4	13.2	32.7	25.5	1.5	4200	13.4	4.9
S2A5	10.0	24.5	27.9	1.5	3800	12.1	3.5
S2A6	7.0	25.1	28.0	1.0	5000	16.0	4.5
S2A7	12.9	30.4	28.3	1.5	3700	11.8	3.2
S2A8	9.9	25.4	26.5	1.4	4100	13.1	3.6
S2A9	10.3	26.1	23.6	1.4	5650	18.0	7.5
S2A10	10.3	25.8	25.3	1.4	5650	18.0	7.4
S2A11	10.2	25.6	25.5	1.4	5650	18.0	7.4
S2A12	10.0	28.7	26.5	1.5	5650	18.0	7.5
S2A13	10.0	48.9	27.3	0.7	6400	20.4	5.1

* All tests utilized 2.4-inch diameter impeller

Under these operating conditions, the low-shear unit operation never achieves a steady-state discharge in terms of product size and consistency. It should be noted however, that throughout this growth/reduction cycle, the product remains screenable and can therefore be recovered. While this is an acceptable mode of operation, the production of a consistent monosized product is desirable to provide a lower ash, lower moisture product.

In an effort to reduce the occurrence of this cyclic growth/reduction cycle during low shear, lower heptane concentrations were tested. To achieve these conditions, the high-shear rpm was set at an 18 m/s tip speed, and the heptane level reduced such that inversion was just maintained. Operating under these conditions, the results shown in Table 14 were obtained. The following parameters were held constant for these tests:

- 150 mesh Sunnyside coal
- Approximately 26 lb/hr coal feed
- 10% solids concentration
- 7.3 minute low-shear residence time

Table 14. Low Shear Testing Results

Heptane % maf	LS Tip Speed m/s	Product Size mm	Product % Solids	Product % ash
23.6	4.8	0.5	33.0	2.86
23.6	3.2	0.5 - Powdery	NA	NA
25.4	3.2	0.5 - Powdery	30.7	2.76
25.4	4.8	3-4 - increasing	64.1	2.55

This data indicates that the higher (4.8 m/s) tip speed is required to produce well formed spherical agglomerates, with the lower tip speed (3.2 m/s) producing a powdery product not very well formed. This data also indicates that a slight increase in heptane dosage, from 23.6 to 25.4% on an ash free dry coal basis, has a great effect on product size. In particular, the product changed from a consistent 0.5 mm product (steady state) to a product that reached 3-4 mm in size and was continuing to grow.

The following general conclusions can be made from low-shear operation results to date:

- At typical heptane concentration operating levels, the low shear product grows to large sizes (4-6 mm diameter) at the originally anticipated impeller tip speeds of 3-5 m/s. However, lower heptane ratios (starvation levels) show promise in controlling low shear product size.
- At these typical heptane levels, high impeller tip speeds (7-10 m/s) are required to maintain agglomerates of a size (2-3 mm) that will discharge from the low-shear vessel. Under these conditions, a cyclic growth and size reduction of the low shear product is observed.
- Low shear discharge plugging problems are more pronounced at higher solids concentrations (particularly above 10%). This trend is expected since at higher solids concentrations more particle to particle contact is expected for the same energy input. Also, at the same volumetric flowrate through the system, a higher solids loading increases the solids flow through the discharge port, which increases the chance for plugging to occur.

Vibrating Screen - Additional testing to evaluate vibrating screen wash water requirements involved the batch screening of low-shear product samples with various wash water rates as was done for the Taggart coal. This work was carried out to determine if substantially greater quantities of wash water would decrease the product ash content. Results of these batch screen tests are shown in Table 15.

These tests were carried out by placing a 1-liter sample of low-shear product on an 8-inch 48-mesh test sieve. Samples of this product were then taken for ash analysis after each of the following:

- Allowing the water to drain from the bed (no rinsing)
- Rinsing the bed lightly with a spray bottle, using approximately 500 ml of water
- Soaking the bed with a gentle water stream for 2 minutes

As this data indicates, some product washing (as represented by 500 ml spray rinsing) was found to reduce the product ash by from approximately 0.01% to as much as 0.5%, and will therefore be utilized. However, it appears that additional washing beyond a spray rinse did not reduce the product ash content substantially. Therefore, a small quantity of wash water was used during typical vibrating screen operation.

It should be noted that the amount of wash water required will be dependent on the size and nature of the agglomerates formed. It is anticipated that when well-formed monosized agglomerates are generated, less wash water will be required since this material will drain well. However, when the agglomerated product proves to be poorly formed, the need for more wash water is anticipated since this product will not drain its associated mineral-matter bearing process water easily.

Table 15. Batch Low-shear Product Rinsing Results (% Ash)

Coal	Agglomerate Size (mm)	Drain No Rinse	Spray Rinse	Soak Rinse
Sunnyside (100 Mesh)	1.0-2.5	3.00	2.87	2.76
	1.0	2.83	2.60	2.49
	2.0-2.5	2.75	2.61	2.61
	1.0-3.0	2.93	2.91	2.82
Sunnyside (150 Mesh)	1.0-3.0	2.67	2.63	2.57
	2.0	2.63	2.54	2.51
	1.0	2.77	2.78	2.74
	0.5	3.01	2.82	2.70
	0.5-1.5 Sludge	2.55	2.49	2.46
	1.5	2.55	2.49	2.46

Indiana VII Coal Continuous Agglomeration Results

Indiana VII coal agglomeration results for complete 25 lb/hr unit tests, i.e., tests in which a full compliment of samples were obtained, are shown in Table 16. It should be noted that the following conditions were held constant for all of these tests:

- 325-mesh topsize grind
- 2.4-inch (4-blade) high-shear impeller
- 4.8-inch (4-blade) low-shear impeller
- Screen inclination of 38°

It should also be noted that as for the Sunnyside coal results, product and tailings ash values, as well as performance calculations, utilized composited samples from froth skimming simulation. This procedure involves reporting the material which floats in the tailings sample as part of the product.

As can be seen from the results in Table 16, the product ash specification of 2 lb ash/MBtu was met at approximately 99% Btu recovery for three of the four tests that utilized the 48 mesh screen. This indicates that the 325 mesh topsize grind utilized provides sufficient ash liberation.

Table 16. Indiana Coal 25 lb/hr Agglomeration Testing Results

Feed			Asph	High Shear		Low Shear		Screen	Product*			Tails*	Performance*	
Coal lb/hr	% Sol	Hept % maf	lb/ ton	Tip m/s	RT min	Tip m/s	RT min	Deck Mesh	% Ash	Size mm	lb Ash/ MBtu	% Ash	Yield %	Btu Rec %
24.1	9.9	34.0	10	18.0	1.5	8.0	3.5	48	2.72	3	1.94	84.8	91.4	99.3
23.5	9.9	34.7	10	18.0	1.5	4.8	7.6	48	2.95	1	2.10	89.1	92.1	99.7
24.5	9.8	33.4	7.5	18.0	1.4	4.8	7.3	48	2.65	3	1.92	79.0	90.7	98.5
23.8	10.0	34.3	10	18.0	1.5	4.8	7.6	48	2.72	1-1.5	1.95	82.3	91.1	99.0
23.8	9.9	34.0	10	18.0	1.5	4.8	7.6	100	2.86	1	2.04	79.7	90.9	98.6
23.6	9.9	32.4	15	13.4	1.5	4.8	7.7	100	2.86	1.5	2.01	88.1	91.9	99.6
25.0	7.0	31.1	15	14.7	1.0	4.8	5.1	100	3.54	<.5	2.53	88.6	92.7	99.7
24.8	6.9	33.0	15	14.7	1.0	4.8	5.1	100	3.26	0.5-1	2.33	89.5	92.5	99.8
16.7	6.9	33.7	10	18.0	1.5	4.8	7.5	100	2.94	0.5-3	2.10	82.0	91.5	99.0
33.4	12.7	30.0	10	17.9	1.4	4.8	9.0	100	3.17	0.5-1	2.26	89.0	92.5	99.7

* Results represents inclusion of simulated froth skimming separation

The product ash specification of 2 lb ash/MBtu was not met for any of the tests completed using the 100 mesh screen deck during agglomerate recovery. This indicates that this finer screen size does not provide drainage of the mineral matter bearing process water as well as the coarser 48-mesh screen. Other observations to be noted concerning agglomeration of the Indiana VII coal are as follows:

- The addition of asphalt at the rate of 7.5-15 lb per ton of coal is required to achieve inversion during high-shear agglomeration.
- Without the necessary asphalt, no growth will occur during the low-shear step resulting in poor coal recovery during screening.
- Approximately 30-35% heptane (maf basis) is required to achieve high shear inversion and low-shear growth with the Indiana coal. This is more heptane than the 23-27% required for the Sunnyside and Taggart coals, as expected due to the finer grind.

- With the testing completed to date, there is no clear effect of solids concentration on product ash content.
- As noted for all testing to date, the product ash content appears most dependent on the agglomerate size distribution with relatively large (1.5-3.0 mm) monosize agglomerates having the lowest ash content.

Low Shear - For the testing carried out on the Indiana VII coal during this reporting period, the low shear operation and product can be characterized by one of the following:

1. Growth in the low-shear vessel starts after about 20 minutes and continues indefinitely. Under these conditions, the increasing size of the low-shear product will eventually plug the low-shear discharge port. The anticipated explanation for this continued growth is the use of excess heptane and/or decreasing coal slurry feed rate combined with relatively low impeller tip speeds (3-5 m/s).
2. Growth in the low-shear vessel is of a cyclic nature in which spherical 1-mm agglomerates form, after which this monosized product grows to approximately 3-4 mm, followed by the formation of smaller agglomerates (<0.5-1.0 mm) which then grow to 3-4 mm in size repeating the cycle. This cyclic growth and reduction pattern occurs primarily when higher impeller tip speeds (6-8 m/s) are used. As such, it is believed that if lower tip speeds were used, growth would be continuous as in case 1 above, i.e., the higher tip speed limits the size of the agglomerates formed.
3. Growth in the low-shear vessel reaches steady-state conditions after approximately one hour of operation. The final size of these agglomerates is usually small, <0.5 - 1 mm in diameter. These conditions are generally achieved when "starvation" quantities of heptane are utilized in high shear. As discussed previously, it has been found that the size of the low-shear product is very sensitive to the amount of heptane utilized.

Heptane Characterization

One of the most important aspects of evaluating steam stripping to recover heptane from agglomerated coal is characterization of both the heptane fed to the process and the heptane remaining on the stripped product.

Feed Heptane - During the course of this project, two types of heptane have been utilized. The first one tested was a de-aromatized commercial grade heptane which has a bulk cost of approximately $1.00 per gallon. The second type of heptane utilized was a pure grade of heptane which has a bulk cost of approximately $6.00 per gallon.

Characterization of these two heptane types was carried out by Phoenix Laboratories of Broomfield, Colorado. The results of these analyses, two for the commercial grade and one for the pure grade, are shown in Table 17.

Table 17. Feed Heptane Analyses - Phoenix Labs

	Normalized Volume %		
	Commercial Grade		Pure
Compound	First	Second	Grade
Benzene	--*	--	--
2,4-Dimethylpentane	0.3	--	--
3,3-Dimethylpentane	3.3	2.2	--
2,3-Dimethylpentane	10.8	9.0	--
2-Methylhexane	18.4	19.9	--
1,3-Dimethylcyclopentane	2.7	2.1	--
3-Methylhexane	24.6	24.3	--
1,2-Dimethylcyclopentane	3.5	2.6	--
n-Heptane	25.3	20.9	99.2
Other Compounds**	NA***	5.6	--
Methylcyclohexane	6.9	6.3	0.8
Ethylcyclopentane	3.4	2.5	--
2,2,3,3-Tetramethylbutane	--	1.5	--
Toluene	0.5	0.2	--
2-Methylheptane	0.3	--	--
C8 Paraffin	--	2.8	--
1,3-Dimethylcyclohexane	--	--	--
Ethyl Benzene	--	--	--
Xylenes	--	--	--

* Not detected
** Primarily Methyl butyrate and Methyl isobutyrate
*** Not analyzed for

As can be seen from this data, the n-heptane content of the commercial grade heptane is in the 21 to 25% range, while the pure grade heptane contains greater than 99% n-heptane as anticipated. The differences between the two separate commercial grade analyses is attributed to different samples and different operators.

Trace Heptane - In an effort to achieve reliable analysis of residual bridging liquid remaining on the product after steam stripping, Huffman Laboratories of Golden, Colorado, was contacted. After discussions with Huffman, it was determined that given the many compounds present in this commercial grade of heptane, it would not be economically feasible to analyze for all of the components on a regular basis.

As such, it was decided that standard practice during this project would be for Huffman Labs to determine the amount of n-heptane remaining in each sample. However, since the commercial grade of heptane used contains only about 20-25% n-heptane, this value would then be converted to provide an estimate of the total solvent or bridging liquid remaining in any given sample. As a starting point a multiplier of "4" was used to convert from n-heptane to total bridging liquid remaining. This assumes that n-heptane remained on the product in approximately the same ratio (25%) as it was present in the original commercial grade of heptane.

In order to allow for accurate conversion of n-heptane to total bridging liquid content for residual heptane analyses, i.e., to check the multiplier of "4", the distribution of the various hydrocarbon components remaining on different types of samples needed to be determined. In an effort to quantify these distributions, additional analyses determining the distribution of the hydrocarbons present were carried out by Phoenix Labs. The samples submitted for these analyses covered a wide range of stripped product types as listed below:

- Coal slurry with no heptane added - boiled for 15 minutes
- Commercial heptane continuous (25 lb/hr) stripper product treated with:
  - Single pass through stripper
  - Two passes through stripper
  - Two passes through stripper and thermally dried
- Various batch agglomeration products boiled for 15 minutes:
  - Commercial heptane with and without asphalt
  - Pure heptane with and without heptane
- Batch agglomeration product (pure heptane) boiled in autoclave at 115 to 120°C for 10 minutes after an initial 5 minute boiling period
- Continuous stripper (25 lb/hr) product (commercial heptane) boiled in autoclave at 115 to 120°C for 10 minutes

Results for these hydrocarbon distribution analyses along with the feed heptane distribution analyses are shown in Table 18.

These results indicate that the originally assumed method of multiplying the n-heptane content by 4 to determine total bridging liquid remaining was conservative in the case of the slurry samples (boiled or steam stripped). The original heptane analysis indicates that 20.9% and 25.3% of the total hydrocarbons are n-heptane, while the stripped product analysis indicates that of the total hydrocarbons remaining, n-heptane represents 29.9-33.0% in the continuously stripped products, and 43-50% in the batch boiled or autoclaved products. As such a more appropriate multiplier would be in the range 2-3.

For the thermally dried product, the originally assumed multiplier of 4 was proven to be very low, since this analysis indicates that only 7.9% of the total hydrocarbons remaining are n-heptane. Therefore, the more appropriate multiplier would be 12.6.

Table 18. Hydrocarbon Distribution for Various Stripped Products

Compound	Commercial Heptane 1	Commercial Heptane 2	Pure Heptane	No Heptane Boiled 15 min Sunny	No Heptane Boiled 15 min Indiana	Sunnyside Coal Commercial Heptane Continuous Stripper Products One Pass	Two Passes	Two Passes & Thermal	Sunnyside Batch Prod Commercial Heptane Boiled 15 min Asphalt (lb/t) 0	10	Pure Heptane Batch Boiled 15 min Sunnyside Asphalt (lb/ton) 0	Sunnyside 10	Indiana 10	Autoclave @ 115-120 C for 10 min following: Comm Heptane Cont Stripping Ind	Comm Heptane Cont Stripping Sun	Pure Hept 5 min Boil Sun
Benzene	-*	-	-	12.4	12.5	1.0	0.8	0.1	0.5	0.6	1	0.5	0.3	0.5	0.4	0.3
2,4-Dimethylpentane	0.3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
3,3-Dimethylpentane	3.3	2.2	-	-	-	-	1.4	7.4	-	1.1	-	-	-	0.9	0.9	-
2.3-Dimethylpentane	10.8	9.0	-	-	-	-	-	19.8	-	-	-	-	-	2.9	-	-
2-Methylhexane	18.4	19.9	-	-	-	14.5	12.0	14.9	5.9	-	-	-	-	8.5	15.7	-
1,3-Dimethylcyclopentane	2.7	2.1	-	-	-	-	-	1.9	1.2	1.4	-	-	-	-	1	-
3-Methylhexane	24.6	24.3	-	-	-	27.3	27.0	2.0	14.2	21.3	-	-	-	20.9	19.4	0.7
1,2-Dimethylcyclopentane	3.5	2.6	-	-	-	1.3	-	4.6	1.9	2.2	-	-	-	1.2	2.5	-
n-heptane	25.3	20.9	99.2	-	13	29.9	33.0	7.9	49.2	45.7	91.7	93.3	96.3	46.5	43.6	95.7
Other Compounds**	NA***	5.6	-	29.9	42.1	12.3	12.7	20.2	2.2	5.2	2.2	3.4	1.5	1.5	1.6	1.4
Methylcyclohexane	6.9	6.3	0.8	-	-	6.7	6.2	13.6	10.7	9.9	-	-	1.1	6	5	1
Ethylcyclopentane	3.4	2.5	-	-	-	2.0	1.7	2.1	4.6	2.5	-	-	-	1.5	1.1	-
2,2,3,3-Tetramethylbutane	-	1.5	-	-	-	1.7	2.0	4.6	2	2.4	-	-	-	2.2	2.1	-
Toluene	0.5	0.2	-	25.2	24.6	2.4	1.7	0.4	2	2.4	2.3	1.1	0.7	1.2	1	0.7
2-Methylheptane	0.3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C8 Paraffin	-	2.8	-	-	-	-	-	-	4.5	4.7	-	-	-	4.8	5.5	-
1,3-Dimethylcyclohexane	-	-	-	-	-	-	-	-	-	-	-	-	-	1.2	-	-
Ethylbenzene	-	-	-	10.7	7.9	0.6	0.5	0.1	0.3	0.4	0.9	0.5	0.2	0.3	0.2	0.2
Xylenes	-	-	-	21.8	-	0.3	1.1	0.4	0.8	-	2	1.2	-	-	-	-
Total	100.0	99.9	100.0	100.0	100.1	100.0	100.1	100.0	100.0	99.8	100.1	100.0	100.1	100.1	100.0	100.0

* Not Detected

** Primarily Methyl butyrate and Methyl isobutyrate

*** Not Analyzed For

One important point to note is that this data indicates that the Benzene, Ethyl Benzene, and Xylenes, detected in the stripped product samples originate from the coal upon heating. As such, their presence in the stripped products cannot be avoided.

Upon analysis of these stripped samples, Phoenix classified some of the hydrocarbons present as "Other Compounds", and identified them primarily as Methyl butyrate and Methyl isobutyrate. While these compounds represent anywhere from 12-42% of the total hydrocarbon present, they are suspected to originate from the plastic lids used for the sample jars.

The following is a summary of observations made based on these trace hydrocarbon distribution results:

- n-heptane represents from 20.9 - 25.3 % of the original commercial grade heptane used
- The original pure grade heptane contains 99.2% n-heptane and 0.8% methylcyclohexane
- When coal agglomerated with commercial grade heptane is thermally dried, n-heptane represents 7.9% of the total remaining hydrocarbons
- When coal agglomerated with the commercial heptane was treated in the continuous 25 lb/hr steam stripper, n-heptane represented from 29.9 - 33.0 % of the total hydrocarbons remaining.
- When plain coal slurry (no heptane) is boiled for 15 minutes, 50-59 ppm of hydrocarbons on a dry coal basis were detected. These hydrocarbons consist primarily of benzene, toluene, ethylbenzene, and xylenes. Some heptane was also present along with the non-hydrocarbon compounds methyl butyrate and methyl isobutyrate.
- The stripping of pure heptane from coal resulted in a trace hydrocarbon distribution of which over 90% was n-heptane. Also consistently detected in these products were benzene, toluene, ethylbenzene and the non-hydrocarbon methylbutyrates.
- Coal agglomerated with commercial heptane and then either boiled for 15 minutes, or boiled and then autoclaved at a higher temperature, had a trace hydrocarbon distribution in which 44-49% was n-heptane. Numerous other hydrocarbons present in the original commercial heptane feed were also found in these products.

Continuous Steam Stripping Results

During this reporting quarter, a variety of operating conditions were tested in the continuous steam stripping column to evaluate its heptane stripping capabilities. Due to problems in providing a consistent feed to the stripping operation, calculations of stripper operating conditions are based on stripper product flowrates and samples. In this manner more reliable operating conditions can be determined.

Initial Testing - One preliminary stripper test was carried out to generate stripper product samples for heptane concentration determination. For this test the stripper feed rate was set to approximately 1 liter/min, at a solids concentration of 9.3%, or approximately 13 lb/hr of coal and 125 lb/hr of associated water. The stripper was then operated with a steam feed rate of 41 lb/hr, the maximum steam flow achievable from the available boiler. A 20-inch section of pall rings was utilized in the stripper, and the liquid level was maintained near the bottom of the column.

In this manner the stripper was operated with a continuous steam phase through which the coal flowed. During this test, the column product flowrate was cyclic, with little discharge under most conditions, followed by surges every few minutes. As such, stripper product samples were taken during both of these discharge conditions. Analytical results for these two product samples are shown in Table 19.

Table 19. Preliminary Stripper Results

	Typical Discharge	Surging Discharge
Solids Concentration, %	2.6	21.0
n-heptane concentration, ppm, sample basis	35.6	438
n-heptane concentration, ppm, dry coal basis	1369	2086

As can be seen from this data, there was a significant change in solids concentration between typical, and surging discharge conditions. However, the n-heptane remaining in the product was similar, ranging from 1369 to 2086 ppm n-heptane on a dry coal basis. It should be noted however, that this testing utilized the hydrotreated commercial grade of heptane, which as discussed previously contains various components. As such, it is estimated that the n-heptane reported in Table 19 would represent only about 30-50% of the total hydrocarbons remaining on the stripped product. Overall, while these analysis indicated that a significant quantity of heptane remains with the product, it was promising that the bulk of the heptane (>99%) was removed.

Stripper Capacity - During this preliminary testing phase an estimate of the capacity of the stripper column was also done. It was determined, that this unit operation could process a coal feed rate approaching 20 lb/hr without experiencing plugging problems. If the coal feed rate was set too high, it was found that the stripper would plug with solids, indicating that there was insufficient steam available to break up the agglomerates (remove the bulk of the heptane), such that the coal could flow through the column and out the discharge.

Additional Continuous Stripper Testing - In an effort to quantify the effects of various stripper operating parameters on product heptane levels, a number of additional tests were carried out. This work utilized Taggart, Sunnyside, and Indiana VII coals. For this work, the level of pall rings, the liquid level, and the steam rate were varied.

For data analysis purposes, the product flowrates and samples were used to determine feed rates. This was done since mixing the feed and pumping it to the stripping column proved difficult, and sometimes inconsistent for short periods of time. For this reason, and because the product samples and flowrates were taken over much longer time periods, the product analyses provided a more accurate determination of column throughput. Results for these tests are shown in Table 20.

It should be noted that heptane analytical results reported in Table 20 represent the n-heptane content remaining on the stripped products, not the total bridging liquid. As such, the actual amount of total bridging liquid remaining on any of the commercial heptane generated products would be 2-3 times greater than that reported. These numbers are reported in this manner since the most appropriate multiplier, to convert from n-heptane to total solvent had yet to be determined. However, for the three tests completed with the pure heptane, these values are approximately one and the same. It should also be noted that while considered very close to actual residence times, those residence times shown in this table represent relative values only.

Taggart Coal Results - As can be seen from these results, stripping of the 62-mesh topsize Taggart coal agglomerated product achieved trace n-heptane concentrations in the range of 700-2000 ppm on a dry coal basis, or approximately 2000-6000 ppm of total commercial heptane. The result for Test 9 is considered an anomaly. All of these results represent agglomerated product passed through the stripping column one time.

Generally, there are no obvious trends relating residence time to product heptane concentration. Also, since residence time is determined by the liquid level in the column, there is no difference in performance between operating the column flooded (with a high liquid level) or in a continuous steam mode (low liquid level).

It was found during this testing that the quantity of excess steam used (steam utilized beyond the theoretical requirement), could be tracked by the temperature of the exiting vapor stream. Theoretically, the boiling point of a heptane/water azeotrop at Golden's elevation is approximately 165°F, so under ideal conditions, only enough steam would be used to achieve this vapor temperature. However, it is believed that as the heptane content in the coal decreases, excess steam is required to remove additional heptane.

This data indicates that a significant reduction in the exiting vapor temperature, from 203 to 173°F does not result in an increased product heptane content. The steam to coal ratio, which varied from as high as 12.5 to as low as 1.7 for this Taggart coal testing also indicates that no benefit is realized from the use of large quantities of excess steam. As such, subsequent testing emphasized evaluation of steam/coal ratios in the lower range of those tested for the Taggart coal.

Table 20. Continuous Steam Stripping Column Results - All Coals

Run	Coal	Top Size Mesh	Hept Type	Steam Actual lb/hr	Ring Hght (in)	Liqu Lev (in)	Stripper Product Basis % Sol	Coal lb/hr	Water lb/hr	Prod. liter/ min	Resid. Time* min	n-heptane, ppm** sample basis	n-heptane, ppm** dry coal basis	Total Steam/ Coal	Total Steam/ Slurry	Stripper Temps (F) Feed	Vapor	Prod
4	Tag	62	Comm	41.2	20	20	2.3	3.3	140.6	1.1	5.6	16	699	12.51	0.29	NA	203	202
5	Tag	62	Comm	41.2	20	20	8.4	17.8	193.5	1.6	3.8	72	855	2.32	0.20	NA	203	202
6	Tag	62	Comm	41.2	6	21	4.5	6.6	141.2	1.1	5.7	52	1166	6.25	0.28	64	203	201
7	Tag	62	Comm	41.2	6	14	9.6	14.5	136.1	1.1	4.2	141	1469	2.85	0.27	67	203	203
8	Tag	62	Comm	41.2	6	6	4.7	7.0	141.5	1.1	2.5	63	1332	5.87	0.28	67	202	203
9	Tag	62	Comm	22.3	6	21	7.4	10.8	134.9	1.1	5.8	820	11111	2.07	0.15	62	200	200
12	Tag	62	Comm	41.2	6	21	8.2	15.6	173.8	1.4	4.5	157	1910	2.65	0.22	57	202	201
13	Tag	62	Comm	24.2	6	21	8.1	14.5	164.9	1.3	4.7	152	1877	1.67	0.13	60	193	198
14	Tag	62	Comm	23.7	6	21	7.1	13.3	175.9	1.4	4.4	113	1603	1.78	0.13	57	173	196
S1S1	Sun	100	Comm	41.2	20	20	11.6	16.7	127.3	1.1	5.7	144	1245	2.47	0.29	NA	203	205
S1S2	Sun	100	Comm	25.7	20	26	9.7	14.3	133.2	1.1	6.8	246	2547	1.80	0.17	NA	202	205
S1S3	Sun	100	Comm	25.7	20	15	9.8	14.3	131.7	1.1	4.5	234	2393	1.80	0.18	NA	201	202
S1S4	Sun	100	Comm	25.7	20	2	8.1	11.2	127.0	1.0	1.8	125	1547	2.30	0.19	NA	201	202
S2S5	Sun	150	Comm	25.7	20	27	6.3	10.7	158.6	1.3	6.1	168	2650	2.39	0.15	134	199	204
S2S6***	Sun	150	Comm	22.3	20	27	5.6	7.5	125.6	1.0	13.8	122	2165	2.97	0.17	130	202	204
S2S7***	Sun	150	Comm	22.3	20	0	5.3	9.0	161.4	1.3	7.2	98	1853	2.47	0.13	54	203	202
S2S8	Sun	150	Pure	41.2	20	23	9.3	13.9	135.6	1.1	6.1	442	4753	2.96	0.28	58	203	206
S2S9****	Sun	150	Pure	41.2	20	23	8.3	12.1	134.9	1.1	12.3	377	4570	3.39	0.28	127	204	206
S2S10****	Sun	150	Pure	41.2	20	1	8.3	11.7	129.9	1.1	7.6	443	5350	3.51	0.29	122	204	203
S2S11	Sun	150	Comm	41.2	20	15	6.2	9.6	145.4	1.2	4.2	155	2508	4.30	0.27	NA	203	205
I1S1	Ind	325	Comm	41.2	20	23	11.0	16.0	129.0	1.1	6.3	267	2418	2.57	0.28	NA	202	206
I1S2*****	Ind	325	Comm	41.2	20	23	10.7	17.5	146.2	1.2	11.9	191	1790	2.36	0.25	129	204	206

* Meant tot provide relative values only

** Represents only the n-heptane fraction of remaining bridging liquid

*** Feed = S2S5 Product

**** Feed = S2S8 Product

***** Feed = I1S1 Product

Sunnyside Coal Results - When carrying out continuous steam stripping of Sunnyside coal agglomerated with commercial heptane, n-heptane concentrations in the 1200-2700 ppm on a dry coal basis were achieved. Based on an estimated multiplier this represents approximately 3500-8000 ppm of total commercial grade heptane on a dry coal basis. Similar ranges of trace n-heptane, 4700-5400 ppm on a dry coal basis, were determined for those three tests in which the pure grade of heptane was utilized, since no multiplier is required.

For several of the Sunnyside stripper tests, stripper product was processed through the unit a second time, as indicated by the longer residence times. The results of these tests indicate that while some additional reduction in heptane content is realized, substantial reductions do not occur with extended residence times.

Generally, these data indicate that slightly lower heptane levels are realized for the Taggart coal than the Sunnyside. One possible explanation for this is that the Sunnyside coal is ground finer (100- and 150-mesh) than the Taggart coal (62-mesh). This finer grind may provide more available surface area, and pores, to which the heptane adheres. This is confirmed by the fact that as a coal is ground finer, more heptane is required to achieve agglomeration.

Indiana VII Coal Results - Only two continuous stripping tests were carried out using agglomerated Indiana VII coal. As anticipated, the results for these two tests, one a single pass and the other a double pass through the stripper, indicate performance, residual heptane levels, similar to that achieved with the other two coals. Residual n-heptane detected for these two tests were about 2400 and 1800 ppm, respectively, equivalent to approximately 7000 and 5000 ppm of total hydrocarbons on a dry coal basis.

Batch Stripper Testing

In an effort to better quantify the achievable trace bridging liquid concentrations remaining with a stripped agglomerated product, a number of batch stripper tests were carried out. These batch tests were found to provide a better indication of the effect of some parameters since more control over the tests were possible. Generally, several different types of batch stripper tests were completed as indicated below:

- Boiling of recovered agglomerates in an open stirred beaker, on a hot plate, for a set period of time @ 94°C (the boiling point of water at Golden altitude
- Thermal drying at 110°C for a set period of time
- Autoclave treatment at 115-1120°C for a set period of time

The results of the various batch stripper tests completed are summarized in the following sections of this report.

Initial Batch Testing - In an effort to better define achievable product heptane concentrations, batch stripper testing was carried out. For this work, samples of continuous stripper products and agglomerated products generated on a batch basis, were either boiled, thermally dried, or both, for set time periods. Table 21 presents some of the initial results which utilized product which had already been processed through the continuous stripper twice. This work used 150-mesh topsize Sunnyside agglomeration product.

Table 21. Preliminary Batch Stripping Test Results

	n-heptane, ppm	
Sample Description	sample	dcb
One pass through continuous stripper	168	2650
Two passes through continuous stripper	122	2165
Two pass product boiled @ 94°C for 30 minutes	100	1506
Two pass product boiled @ 94°C for 60 minutes	96	1184
60 min boil product thermally dried at 110°C for 2 hours	294	294

This data indicates that lower n-heptane concentrations are achieved as the residence time under stripping conditions increases. While this trend is expected, it is surprising that such long residence times (1 hour) are required to achieve the trace n-heptane levels of approximately 1200 ppm on a dry coal basis, equivalent to about 3000 ppm of total hydrocarbons on a dry coal basis..

As can also be seen from this data, thermal drying achieved much lower heptane levels. This is believed to be due to a combination of the longer residence time, the higher temperature, and the removal of virtually all water present.

In an effort to confirm these results, and to determine the removal characteristics of other bridging liquids, similar batch testing was completed using a pure heptane, commercial heptane, and pentane. Some tests were also completed with asphalt to evaluate its effect on heptane removal. Results for these tests are shown in Table 22. It should be noted that the commercial heptane analyses are for n-heptane only and as such are actually two to three times greater than indicated.

Based on the results shown in Table 22, the following conclusions were made:

- On a dry coal basis, thermal drying provides significantly lower trace solvent concentrations than boiling.
- Storage of the product for 2 days prior to stripping results in higher trace heptane concentrations than immediate stripping.
- The use of asphalt results in lower heptane concentrations when the product is boiled.
- Pentane can be removed to very low levels under all conditions tested.

Table 22. Batch Stripper Testing - Various Hydrocarbons

Solvent Type	Stripping Method	n-hept/pen, ppm sample	n-hept/pen, ppm dcb
Commercial Heptane*	Thermal**	147	147
	Boil***	40	887
	Store/Boil****	42	1254
Pure Heptane	Thermal	459	459
	Boil	75	2038
	Store/Boil	132	3687
Pure Heptane + Asphalt	Thermal	409	409
	Boil	24	792
Pentane	Thermal	2	2
	Boil	2	60
Pentane + asphalt	Boil	2	56

* Analysis for n-heptane only, total hydrocarbons about 2.5 times higher
** Screen product filtered and thermally dried @ 110°C for 2 hours
*** Screen product reslurried and boiled @ 94°C for 2 hours
**** Screen product stored as slurry for 48 hours and than processed

Asphalt Effect - To confirm the previous result indicating that the presence of asphalt may result in reduced product heptane concentrations, several tests were carried out. For this work, batch stripping tests, in which the agglomerated products were either boiled or thermally dried for two hours were completed with and without the use of asphalt during the agglomeration step. Results of these tests, along with previous data, are shown in Table 23.

As can be seen from these results, in every case except one, the presence of asphalt during agglomeration resulted in a lower trace bridging liquid content after stripping, whether done thermally or via boiling.

Pressure Effect - Since significantly lower trace heptane contents are realized when thermal drying at 110°C over boiling at 94°C, batch stripper tests were carried out in which agglomerated products were boiled under pressure to maintain a slurry temperature in the range of 115-120°C for predetermined periods of time. To achieve these high temperatures, these tests were completed in a PARR Autoclave fixed with a 15 psi pressure relief valve. Heat was applied to the autoclave containing a fixed amount of slurry, and as such, a continuous vapor discharge was released by the pressure relief valve to maintain the desired temperature. Results for these tests are shown in Table 24.

As can be seen from the data in Table 24, treatment in the autoclave at increased pressure and temperature resulted in reduced heptane concentrations as compared to the continuous stripper product.

Table 23. Batch Stripper Results - Asphalt Effects

Heptane Type	Asph lb/t	Coal	Stripping Method	n-heptane, ppm sample	n-heptane, ppm dcb
Commercial*	--	Indiana	Boil-2hrs**	31	1161
	8	Indiana	Boil-2hrs	7.6	262
	--	Indiana	Thermal***	220	220
	8	Indiana	Thermal	35	35
	--	Sunnyside	Boil-2hrs	33	1000
	8	Sunnyside	Boil-2hrs	118	4097
	--	Sunnyside	Boil-15min**	203	2509
	10	Sunnyside	Boil-15min	103	1533
Pure	--	Sunnyside	Boil-15min	390	5660
	10	Sunnyside	Boil-15min	284	4051
	--	Sunnyside	Thermal	459	459
	5	Sunnyside	Thermal	409	409
	--	Sunnyside	Boil-2hrs	75	2038
	5	Sunnyside	Boil-2hrs	24	792

* Analysis for n-heptane only, total hydrocarbons about 2.5 times higher
** Screen product reslurried and boiled @ 94°C for 2 hours
*** Screen product filtered and thermally dried @ 110°C for 2 hours

Table 24. Batch Stripper Results - Pressure Effects

Solvent	Asphalt lb/t	Coal	Autoclave** Ret Time (min)	n-heptane, ppm sample	n-heptane, ppm dcb
Commercial Heptane*	7.5	Ind	0**	208	1949
	7.5	Ind	10	82	804
	7.5	Ind	20	74	604
	7.5	Ind	30	29	334
	--	Sun	0***	155	2583
	--	Sun	10	61	893
	--	Sun	20	45	575
	--	Sun	30	55	589
Pure Heptane	--	Sun	0****	887	8153
	--	Sun	10	242	2995
	--	Sun	20	301	3053
	--	Sun	30	227	1903

* Analysis for n-heptane only, total hydrocarbons about 2.5 times higher
** Retention time in autoclave @ 115-120°C
*** Autoclave feed - Continuous stripping column product
**** Autoclave feed - Batch agglomeration product boil @ 94°C for 5 minutes

Storage Effect on Heptane Concentration - As part of this project, agglomeration circuit product will be formulated into highly loaded coal-water-fuels. As a first estimate to determine the fate of any heptane remaining on the coal after stripping, Sunnyside coal continuous stripper product was filtered and formulated into a slurry of approximately 55% solids. The heptane concentration of this slurry was monitored over a period of time. Table 25 presents the results of this work. It should be noted that the material used to generate the slurry was a combination of single and double pass continuous stripper product, originally agglomerated with pure heptane.

Table 25. CWF Heptane Concentration

Solvent	Days Stored*	n-hept ppm, samp	% Solids	n-hept ppm, dcb	Solvent ppm, dcb
Pure Heptane	0	3630	55.28	6567	6567
	2	4020	55.48	7246	7246
	7	4200	56.11	7485	7485
	14	3370	41.76	8070	8070

* Stored uncovered and mixed approximately 6 hours per day

As can be seen from this data, there appears to be a slight increase in heptane present in the slurry over time. While this is not possible, it is encouraging that the heptane level is not decreasing, indicating that storage of highly loaded CWF slurries should not pose safety and environmental related risks.

PDU Stripper Design

Based on the results presented above, the design of the 2 t/hr PDU stripping circuit calls for a two-stage process. In the first stage approximately 5-minutes of residence time will be provided for removal of the bulk of the bridging liquid (about 99%). During the second stage, 5-10 minutes of additional residence time will be provided, but at increased pressure and temperature (consistent with conditions tested in the autoclave). To reduce the amount of steam required, it is planned to perform these stripping steps at approximately 25% solids. As such, a brief series of test were carried out to determine if solids concentration had any effect on the reduction of heptane present in a stripped product. Results for these four test are shown in Table 26. It should be noted that batch agglomerated Sunnyside coal was used for these tests, and that each of these products was boiled at 94°C for 15 minutes to best simulate the anticipated PDU stripping circuit design.

As can be seen from this data, stripping (or boiling) at 25% solids concentration results in negligibly higher levels of trace heptane concentration for both pure and commercial heptane tests than when the stripping is completed at 10% solids.

TASK 7 SELECTIVE AGGLOMERATION - PDU DETAILED DESIGN

Three meetings were held with Arcanum and Bechtel personnel during this reporting period to discuss the detailed design of the PDU selective agglomeration module.

Discussions focused on finalizing the PFD's and P&ID's so that the detailed design can be completed in a timely manner.

Table 26. Batch Stripper - Solids Concentration Effect

Solvent*	% Solids**	n-heptane, ppm sample	n-heptane, ppm dcb
Commercial Heptane*	25	248	3225
	10	229	2884
Pure Heptane	25	544	7545
	10	563	7312

* Analysis for n-heptane, total hydrocarbons 2.5 times higher
** *Solids concentration of stripping procedure*

The design has progressed considerably. Currently, only equipment sizing and layout remain to be completed along with a final review of the overall process control philosophy. The following sections of this report discuss design features of the main unit operations.

Coal Grinding and Dewatering

It is not anticipated that any major changes in either the coal grinding or dewatering circuits will be required for the switchover from the flotation to agglomeration process module. As such, the agglomeration process will use slurry ground by the installed equipment. This slurry will however be stored in one of two ground slurry storage tanks prior to being metered to the agglomeration process. This will insure that a homogeneous feed is available to the agglomeration circuit for extended periods of time.

Once processed, the agglomerated and steam stripped product will be dewatered in the same manner as during flotation circuit operation. Generally, the bulk of the product will be dewatered in the existing vacuum drum filter (Westech), with two filter presses (Netzsch) also available for product dewatering. Process tailings will be sent to the existing thickener from which they will be dewatered via a pair of tailings filter presses (Shriver).

High Shear Vessel

High-shear agglomeration will be carried out in a circuit consisting of two high-shear reactors. These reactors will be sized to provide approximately 0.5 and 1 minute residence times respectively. These units will each consist of two sections utilizing 4-blade radial flow impellers. Each high-shear agglomerator will be powered by variable speed drive units that can achieve up to 18 m/s impeller tip speeds. In this manner, high shear residence times from 0.5 to 1.5 minutes can be achieved by operating either unit individually, or both together in series. Such a circuit was provided primarily to accommodate the Indiana VII coal which requires significantly longer residence times to achieve inversion during high shear. It should be noted that the smaller of these two units is an existing vessel from another DOE project.

Heptane will be metered to the high-shear circuit as required, currently anticipated not to exceed 40% of the coal rate. While it is anticipated that a pure grade of heptane will be used during PDU operation, the system will also be capable of utilizing a commercial grade heptane.

Based on test results to date, the ability to add asphalt to high-shear will be provided for the Indiana VII coal. It is currently planned to use an anionic asphalt emulsion metered to the process to provide 5-15 lb asphalt per ton of coal.

Low Shear Vessel

Low-shear agglomeration will be carried out in a single vessel divided into two sections via a horizontal baffle. Each section will provide approximately 2.5 minutes of residence time for a maximum of five minutes. The discharge piping will be arranged such that one or both sections can be utilized. The low shear operation will be powered by a single drive unit providing one impeller for each section of the vessel. The impellers will be of the radial-flow type and able to achieve tip speeds in the 2-8 m/s range.

Vibrating Screen and Froth Skimmer

The vibrating screen to be used to recover agglomerates from the low shear product will be a 48-mesh dewatering screen approximately 2-feet wide by 6-feet long. Sufficient spray nozzles will be provided to insure replacement of mineral matter bearing process water with fresh water.

The vibrating screen underflow (tailings) will then be processed through a froth skimmer. This skimmer will provide approximately 5-minutes of residence time for any carbonaceous material to float. If necessary, nitrogen will be bubbled through the skimmer to help the material float. A continuous paddle will then scrape the floating material to a launder from where it will be combined with the screen product.

Steam Stripper

The combined screen and froth skimmer product will be diluted with hot water to approximately 25% solids. This feed will then be treated in the first stage steam stripper which will provide a 5-minute residence time at ambient pressure and approximately 94°C. The heat source for this stirred vessel will be the vapor product (steam and heptane) from the second stage stripper. This first stage stripper has two primary functions. First it will remove the bulk of the heptane present (about 99%), and second, it will generate a handleable and pumpable product.

This first-stage stripper product will then be pumped to the second-stage stripper which will provide about 10-minutes of residence time at 15-20 psig pressure and 115-120°C. The product from this second stage stripper will then be cooled and sent to the dewatering circuit.

Condenser and Gravity Separator

Vapors from the first stage stripper will contain steam and recovered heptane and be condensed in a two-stage process. Initial cooling will be carried out by an air cooler, followed by a shell and tube condenser to provide the necessary sub-cooling.

The condensed liquids will then be sent to the gravity separator from which both the heptane and the process water will be recycled to the process.

TASK 8 PDU AND ADVANCED COLUMN FLOTATION MODULE

The majority of the Task 8 work effort was devoted to the construction of the PDU Flotation Module. TIC, The Industrial Company of Steamboat Springs, Colorado, continued the construction of the PDU during this reporting quarter. All required permits were obtained from Jefferson County, Colorado. Currently, the Flotation Module is approximately 62% complete. Entech Global personnel continued with the procurement of new capital equipment and instrumentation as well as the refurbishing of existing equipment.

Efforts are also underway for the procurement of Taggart coal for use during startup and shakedown.

Subtask 8.1 Coal Selection and Procurement

Since startup of the advanced flotation module is scheduled for August, arrangements began during this quarter for procuring the three coals needed for the operation of the PDU. The plan is to start the operation with an Eastern coal, continue on with a Western coal, and finish the flotation work with a more difficult to process Midwestern coal. Seven or eight hundred tons of each coal will be needed when the projected requirements for operation of the Task 9 selective agglomeration module are included. To avoid the danger of a drastic change in the quality of the mine production between loads, the entire requirement for each coal will be procured at one time.

Eastern Coal

Taggart seam coal was selected as the Eastern coal for the PDU operation because of its good performance in the bench-scale systems. Red River Coal Company has taken over the Taggart coal accounts served by Westmoreland Coal Company from the recently closed Wentz Mine. Red River is supplying Taggart coal from the Steer Branch Mine in Wise County near Norton, Virginia. Eight cars of washed coal from the Steer Branch Mine were ordered and four cars were shipped on June 26 and four cars on July 10. The first four cars have been received in Denver. The total shipment was for 799 tons. Red River provided the analysis shown in Table 27 for the two shipments.

Table 27. Analysis of Shipped Taggart Coal

	First Four Cars		Second Four Cars	
	As Rec'd	Dry	As Rec'd	Dry
Moisture, percent	5.83		5.84	
Ash, percent	3.01	3.20	2.90	3.08
Sulfur, percent	0.65	0.69	0.64	0.68
, lb/mmBtu	0.46	0.46	0.46	0.46
HHV, Btu/lb	14,039	14,908	14,028	14,898

Red River also provided a drum of Steer Branch raw coal for use when tracking carbon recovery and the reduction in toxic trace element concentrations during advanced cleaning.

Significant savings were made by utilizing rail haulage rather than truck haulage for the Taggart coal. Arrangements were made with the American Coal Company to unload the cars and store the coal at their yard in Denver. The Taggart coal will be trucked from there to Ralston Development Corporation in Golden as needed for crushing.

Western Coal

The first plans were to operate the PDU with Sunnyside seam coal from the Sunnyside Mine in Utah. However, that coal is no longer available due to mine closure so alternative bituminous coals from Utah, Colorado, and Wyoming were evaluated. A survey of Keystone entries and conversations with local coal brokers provided the following list of potential replacement coal sources which could meet coal quality requirements and also the project mine production and reserve objectives:

1. Andalex Mine, Utah
2. Genwal Mine, Utah
3. Bear Mine, Colorado
4. Somerset Mine, Colorado

5. Wet Elk Mine, Colorado
6. National King Mine, Colorado
7. Sanborn Creek Mine, Colorado
8. Powderhorn Mine, Colorado
9. Golden Eagle Mine, Colorado

All of the above are compliance coals that contain well under 0.6 lb sulfur per million Btu. Part of the production of the Andalex Mine is from the Sunnyside seam a few miles from the now-closed Sunnyside Mine, but the mine cannot segregate production from that seam to allow for separate shipment.

Heptane agglomeration liberation tests were carried out on samples of the nine coals listed above. This liberation testing was completed using minus 35-mesh pulverized coal, and minus 325-mesh attritor-ground slurry. A few follow-up tests were also carried out at a minus 100-mesh grind. A Sunnyside sample was also included in the set. Of the potential replacements, only the Genwal, National King, and Bear coals were sufficiently well liberated to meet the 2 lb ash/MBtu specification as shown in Table 28.

Table 28. Sunnyside Coal Replacement - Evaluation Results

Coal	Grind	Feed Coal % Ash	Residual Ash Percent	Residual Ash lb/MBtu	MAF Coal Recovery, %
Sunnyside	-48 mesh	5.32	3.42	2.39	97.6
Sunnyside	-325 mesh	5.32	2.06	1.42	99.4
Genwal	-100 mesh	8.43	2.65	1.83	98.9
Genwal	-325 mesh	8.43	2.10	1.45	98.5
King	-100 mesh	5.43	3.20	2.21	98.3
King	-325 mesh	5.43	2.22	1.52	99.2
Bear	-325 mesh	7.76	2.64	1.83	99.4

Of the coals evaluated, Genwal mine production, from the Hiawatha seam, comes closest to matching the Sunnyside coal used during Phase 1 of the program. Flotation amenability tests will be carried out before issuing a purchase order for the coal. National King may be a viable alternative to Genwal, but coal from that mine will be more expensive for PDU use since the mine is not served by a railroad.

Midwestern Coal

Minnehaha Mine management confirmed that they can supply the low sulfur Indiana VII coal required for the PDU. Comparison freight rates are being obtained for rail and truck haulage. It is of some concern that the shipment be completed before late November in order to avoid freezing problems.

Subtask 8.2 PDU Construction

TIC continued construction efforts during the second quarter of 1995. Overall, the PDU Flotation Module is 62% complete with one of the seven designated milestones at 100% completion and five greater than 55% complete. The PDU construction is on schedule for mechanical completion by July 31, 1995.

Entech Global personnel continued with the procurement of new capital equipment items and instrumentation for use in the PDU. Almost all equipment items have been received at the Amax site while approximately 50% of the instrumentation is still outstanding.

Reconditioning of existing equipment was also initiated during the quarter. Several equipment items have been reconditioned with the remainder scheduled for completion in July.

TIC Construction Activities

TIC - The Industrial Company continued the construction efforts of the PDU Flotation Module. The overall project completion (by milestone) is shown in Table 29.

Table 29. Construction Progress by Milestone

Milestone No.	Description	% Complete
1	Mobilization, Excavation, Concrete and Foundation Work	100%
2	Structural Steel Erection and Platework	85%
3	Equipment Installation	85%
4	Piping Installation	60%
5	Electrical and Instrumentation Installation	55%
6	Sheeting and Painting	65%
7	Testing, Startup, , and Demobilization	0%
Total		62%

The following construction activities were completed by TIC during the second quarter of 1995:

- Demolition / removal of structural steel from Area 200
- Demolition / removal of concrete from Area 100
- Drilled caissons for thickener. Bedrock was observed at a depth of 9-10 feet. Because the plan calls for all caissons to be 6 feet into bedrock, the overall depth of the caissons became 16 feet
- Drilled caissons for clarified water tank
- Drilled caissons for Microcel™ flotation column
- Drilled caissons for coal dump hopper

- Drilled caissons for coal storage bin
- Installed concrete for thickener caissons and floor
- Installed concrete for clarified water tank caissons and floor
- Installed concrete for Microcel™ flotation column caissons and floor
- Installed concrete for coal storage bin caissons and pad
- Installed concrete pad for tailings filter press feed sump (400-D-04)
- Installed concrete pad for clean coal sump (400-D-05)
- Installed concrete pad for slurry storage tanks (300-D-01 & 300-D-02)
- Installed concrete pad for primary and secondary ball mills (100-Y-01 & 100-Y-02)
- Installed concrete pad for coal dump hopper (100-D-01)
- Installed concrete pad for coal storage bin (100-D-02)
- Installed clean coal sump (400-D-05)
- Installed / assembled tailings thickener (400-D-01)
- Removed cluttered piping above tailings thickener bridge
- Removed siding from eastern side of building in Areas 100 and 400
- Removed siding from PDU Area 100 for equipment installation
- Removed siding & roof from PDU Area 200 for equipment installation
- Installed clean coal filter press feed pumps (400-G-04 & 400-G-05)
- Installed Netzsch clean coal filter #1 (400-Y-04)
- Installed Netzsch clean coal filter #2 (400-Y-05)
- Installed WesTech clean coal drum filter (400-Y-09)
- Saw cut floor in to accommodate Netzsch fine grinding mill (100-Y-03)
- Installed Netzsch fine grinding mill (100-Y-03)
- Moved primary and secondary ball mills to PDU site (100-Y-01 & 100-Y-02)
- Installed primary & secondary ball mills 100-Y-01 & 100-Y-02)
- Installed clarified water tank (400-D-02)
- Replaced siding from PDU Area 100
- Relocated existing Drais mill
- Installed structural steel for Area 200 penthouse
- Installed slurry storage tank A (300-D-01)
- Installed slurry storage tank B (300-D-02)
- Installed ground product agitator A (300-Y-01)
- Installed ground product agitator B (300-Y-02)
- Completed approximately 80% of process pipe fitting in Area 100
- Relocated Area 300 electrical conduit and process pipe

- Installed Sizetec fine sizing screens (100-Y-04A & 100-Y-04B)
- Installed thickener and thickener bridge (400-D-01)
- Installed primary conduit from proposed transformer site to MCC rooms 1, 2, and 3
- Installed primary ball mill discharge sump (100-D-03)
- Installed secondary ball mill discharge sump (100-D-04)
- Installed ground product sump (100-D-06)
- Installed fine grinding mill feed sump (100-D-07)
- Installed primary ball mill discharge pump (100-G-01)
- Installed secondary ball mill discharge pump (100-G-02)
- Installed ground product pump (100-G-04)
- Installed fine grinding mill feed pump (100-G-03)
- Installed CP-100 electrical panel in Area 100
- Installed drum filter feed pump (400-G-08)
- Installed fine grinding mill support steel
- Installed process pipe in Area 400
- Installed conduit to hand stations in Area 100
- installed conduit to hand stations in Area 400
- Installed overflow line on clean coal sump
- Installed make-up water line to thickener
- Installed structural steel in Area 200
- Installed isolation valve on thickener underflow line
- Installed power conduit to equipment items in Area 100
- Installed power conduit to equipment items in Area 200
- Installed power conduit to equipment items in Area 400
- Installed final tailings sump (200-D-03)
- Installed Microcel™ flotation column (200-Y-02)
- Installed fluid sensor on primary ball mill discharge sump
- Installed fluid sensor on secondary ball mill discharge sump
- Installed fluid sensor on fine grinding mill feed sump
- Installed discharge pipe on fine grinding mill feed pump (100-G-03)
- Removed existing slurry tanks from Area 400 to accommodate clean coal conveyors
- Installed cyclone header
- Installed classifying cyclones
- Installed feed and discharge piping to classifying cyclones (100-Y-07)
- Installed feed and discharge pipe to Netzsch fine grinding mill (100-Y-03)

- Installed feed and discharge pipe to Sizetec screens (100-Y-04 A & B)
- Connected & started Honeywell S9000 controller
- Connected & started Honeywell SCAN 3000 Data Acquisition System
- Erected coal storage shed
- Installed bypass line from grinding circuit to thickener
- Installed exterior junction box for power distribution / transformer
- Installed clean coal conveyor #1 (400-T-03)
- Installed clean coal conveyor #2 (400-T-04)
- Installed clarified water line to process
- Installed hand stations in Area 100
- Installed hand stations in Area 200
- Installed hand stations in Area 400
- Pulled power & control wire in Area 100
- Pulled power & control wire in Area 200
- Pulled power & control wire in Area 400
- Installed lighting fixtures in Area 200
- Installed ground product process pipe from Area 100 to Area 200
- Installed tailings pipe from Area 200 to Area 400
- Started to erect coal storage bin (100-D-02)
- Started to install CDAS panel in control room
- Installed flocculant mixing tank
- Installed cationic dilution tank
- Started to enclose Area 200 penthouse with siding

Construction activities are to be completed during the next quarter with mechanical completion of the PDU Flotation Module scheduled for July 31, 1995.

TIC Request For Information (RFI)

TIC and Amax R&D, Inc. have established a method to expedite and clarify unclear issues regarding the construction of the Process Development Unit. When an issue or question arises which requires a response by Amax R&D, TIC issues a written Request for Information (RFI), including cost implications if any. Amax R&D then responds to the issue accordingly, including approval of any cost associated with the change order. Table 30 below provides a list of RFI's issued during the quarter along with the reply dates and the name of the person who signed the authorization .

Table 30. Request For Informations Issued by TIC

RFI #	RFI Date	Subject	Reply	Signed
217-001	4/4/95	P.E. Stamped Drawings	4/4/95	G. Shields
217-002	4/4/95	Area 200 Demolition	4/4/95	G. Shields
217-003	4/4/95	Mechanical Equipment Schedule	4/13/95	G. Shields
217-004	4/5/95	CS-100-M-02 4-inch Drain	4/10/95	G. Shields
217-005	4/5/95	4-inch Void	4/10/95	S. Bajwa
217-006	4/5/95	Soil Compaction	4/10/95	S. Bajwa
217-007	4/10/95	Structural Detailing	4/11/95	S. Bajwa
217-008	4/12/95	Member Location - Plan @ El 125'-0"	4/18/95	S. Bajwa
217-009		DELETED		
217-010	4/14/95	Soils Report	4/17/95	G. Shields
217-011	4/14/95	Change Orders via RFI Approval	4/17/95	G. Shields
217-012	4/14/95	PE Approval -Caisson Drilling/Concrete	4/17/95	G. Shields
217-013	4/17/95	6-inch Movement of Microcel	4/17/95	G. Shields
217-014	4/17/95	Concrete Removal Additions	4/17/95	M. Jha
217-015	4/17/95	Area 100 Sump Tie-In	4/17/95	M. Jha
217-016	4/17/95	Small Footer Credit	4/17/95	G. Shields
217-017	4/18/95	T/C Elevation for Pads	4/18/95	S. Bajwa
217-018	4/19/95	Area 400 Beam Size (Moment / Bolt)	4/19/95	S. Bajwa
217-019	4/19/95	Area 200 Stairway	4/25/95	G. Shields
217-020	4/24/95	400-D-01 Catwalk Pipe Removal	4/24/95	G. Shields
217-021	5/1/95	Structural Steel Addition	5/1/95	M. Jha
217-022	5/1/95	Caisson Depth	5/1/95	M. Jha
217-023	5/1/95	Electrical Equipment Delivery	5/16/95	Reynouard
217-024	5/3/95	Removal of Pipe above Thickener	5/3/95	G. Shields
217-025	5/3/95	Alternate Method for Conv Mounting	5/3/95	G. Shields
217-026	5/3/95	Thickener Platform Fabrication	5/3/95	G. Shields
217-027	5/3/95	Thickener Platform Installation	5/3/95	G. Shields
217-028	5/8/95	PDU Instrumentation Status	5/15/95	G. Shields
217-029	5/8/95	Flow Meter Orientation	5/15/95	G. Shields
217-030	5/10/95	Fine Grinding Mill Concrete Failure	5/11/95	M. Jha
217-031	5/16/95	Area 300 Electrical Relocation	5/19/95	M. Jha
217-032	5/23/95	Structural Bolting Tensioning	5/23/95	S. Bajwa
217-033	5/24/95	Plug Valve Adjustment	5/24/95	G. Shields
217-034	5/25/95	Thick U'Flow Line Change	5/30/95	G. Shields
217-035	5/30/95	CC Pump Suction - Size Increase A	5/30/95	M. Jha
217-036	5/30/95	CC Pump Suction - Size Increase B	5/30/95	M. Jha
217-037	5/30/95	Thick O'Flow Valve Installation	6/7/95	G. Shields
217-038	5/30/95	Microcel™ Recirc Line Change - A	5/30/95	M. Jha
217-039	5/30/95	Microcel™ Recirc Line Change - B	5/30/95	M. Jha
217-040	6/7/95	Cyclone Header Cover	6/7/95	G. Shields
217-041	6/1/95	Cyclone Header Support Structure	6/2/95	G. Shields
217-042	6/1/95	Cyclone Header Support Structure - Installation	6/2/95	G. Shields
217-043	6/1/95	Cyclone Inlet & Overflow Piping	6/2/95	G. Shields
217-044	6/5/95	Cyclone Header Pressure indication	6/6/95	G. Shields
217-045	6/7/95	Primary & Secondary Mill Discharge Sump Modifications	6/7/95	G. Shields
217-046	6/7/95	Microcel Internals / Bolt vs. Flange	6/7/95	G. Shields
217-047	6/7/95	200-D-02 Leg Shortening	6/7/95	G. Shields
217-048	6/7/95	Lower Agitator Supports on 300-D-01 & 02	6/7/95	G. Shields
217-049	6/8/95	Wire Specification Change	6/9/95	G. Shields
217-050	6/8/95	Wire Length Reductions	6/9/95	G. Shields
217-051	6/8/95	Relocation of Slurry Tank	6/19/95	M. Jha
217-052	6/8/95	Raise Platform @ Elevation 120' - 0"	6/12/95	G. Shields
217-053	6/12/95	Sealing of Ball Mill Ends	6/19/95	M. Jha
217-054	6/13/95	Cyclone Piping Valve & Tee Credit	6/19/95	M. Jha
217-055	6/19/95	Cyclone Piping Changes	6/19/95	M. Jha
217-056	6/21/95	400-G-05 Pump Base Modification	6/21/95	G. Shields

Procurement of Permits for PDU Construction

A building permit was received from Jefferson County's Building Department on April 11, 1995. This allows for the structural erection of the Process Development Unit (PDU). TIC, the PDU construction subcontractor, was instrumental in procuring this permit.

A separate permit was procured for all electrical work on May 5, 1995. This permit allows for all electrical construction at the PDU site. Mr. Robert Reynouard, P.E., the electrical engineer retained to provide electrical consultation services, was very helpful in procuring this permit by approving and stamping all related electrical drawings.

Procurement of PDU Capital Equipment

Entech Global personnel continued to procure capital equipment for use in the PDU. During the second quarter of 1995, two Request for Quotation (RFQ) packages were issued. Award packages were issued for three capital equipment items while 25 equipment items were delivered. The status of each item, along with those items awarded and received during the quarter (hi-lighted in boxes), is listed in Table 31.

Delivery of the remaining reagent pump (200-G-04) is expected during the month of July.

Procurement of PDU Instrumentation

During the quarter, Entech Global, Inc. personnel issued RFQ's and awards for 51 PDU instrumentation items. Thirty items were received during the month of June. The status of each item, along with those items received (hi-lighted in boxes) is listed in Table 32.

Procurement of Control and Data Acquisition System (CDAS)

Honeywell, Inc. was selected during the quarter to supply and deliver a Control & Data Acquisition System for use in the PDU. The Controller, a Series 9000 unit, is object oriented and allows easy programming by Entech Global personnel. The control interface and database, a SCAN 3000 unit, will operate on a personal computer system using a SCO UNIX motif.

Both units were received and installed during the quarter. Mr. Charlie Richmond, a Honeywell Principal System Consultant, was on site from June 13 through June 17 to assist with the installation.

Table 31. Status of Capital Equipment Procurement

Equipment Number	Description	RFQ Date	Deadline	Award Date	Company	Delivered Price	Delivery
100-D-03	Pri. Ball Mill Disch. Sump	1/6/95	1/18/95	2/15/95	Metal-Craft	$2,835	5/2/95
100-D-04	Sec. Ball Mill Disch. Sump	1/6/95	1/18/95	2/15/95	Metal-Craft	$3,014	5/2/95
100-D-06	Ground Product Sump	1/6/95	1/18/95	2/15/95	Metal-Craft	$3,014	5/2/95
100-D-07	Fine Grind Mill Feed Sump	1/6/95	1/18/95	2/15/95	Metal-Craft	$2,151	5/2/95
100-D-09	Cyclone Distributor	4/1/95	4/12/95	4/12/95	Precision Mechanical, Inc.	$3,228	5/15/95
100-G-01	Pri. Ball Mill Disch. Pump	10/6/94	10/14/94	11/16/94	Quadna Pump Systems	$7,660	1/24/95
100-G-02	Sec. Ball Mill Disch. Pump	10/6/94	10/14/94	11/16/94	Quadna Pump Systems	$12,335	2/7/95
100-G-03	Fine Grind Mill Feed Pump	10/6/94	10/14/94	11/16/94	Quadna Pump Systems	$10,455	1/24/95
100-G-04	Ground Product Pump	10/6/94	11/16/94	12/19/94	Canmac Engineering Sales	$3,079	4/4/95
100-T-05	Vibrating Bin Bottom	10/21/94	11/4/94	11/29/94	Kinergy, Inc.	$7,336	3/16/95
100-T-06	Pri Ball Mill Screw Conv.	10/18/94	10/31/94	12/19/94	Rocky Mountain Conveyor	$6,324	3/3/95
100-Y-03	Fine Grinding Mill	9/15/94	9/29/94	11/10/94	Netzsch, Inc.	$292,169	5/2/95
100-Y-04	Vibrating Screens	10/12/94	10/21/94	11/10/94	Sizetec, Inc.	$43,229	1/23/95
100-Y-05	Ground Product Agitator	11/10/94	11/30/94	2/16/95	D.W. Daigler (Lightnin)	$1,307	3/14/95
100-Y-06	Fine Mill Feed Sump Agit.	11/10/94	11/30/94	2/16/95	D.W. Daigler (Lightnin)	$1,112	3/14/95
100-Y-07	Classifying Cyclones	11/11/94	11/23/94	12/19/94	APCOR (Krebs' Cyclones)	$2,658	3/9/95
200-C-01	Compressed Air Receiver	10/18/94	10/31/94	1/4/95	Colorado Compressor	$0	4/24/95
200-D-01	Column Feed Sump	1/6/95	1/18/95	2/15/95	Brewer Steel	$3,153	5/19/95
200-D-02	Final Tailings Sump	1/6/95	1/18/95	2/15/95	Brewer Steel	$3,405	6/6/95
200-D-03	Frother Drum						
200-D-04	Collector Drum						
200-G-01	Column Feed Pump	11/4/94	11/16/94	12/19/94	Canmac Engineering Sales	$5,908	5/11/95
200-G-02	Column Recirc Pump	11/29/94	12/9/94	12/19/94	Canmac Engineering Sales	$7,325	4/13/95
200-G-03	Final Tailings Pump	11/4/94	11/16/94	12/19/94	Canmac Engineering Sales	$5,908	4/13/95
200-G-04	Reagent Feeder - Frother	5/17/95	5/21/95	5/21/95	Nalco Chemical Company	$1,530	3 weeks
200-G-05	Reagent Feeder - Collector	5/17/95	5/21/95	5/21/95	Nalco Chemical Company	$1,530	6/26/95
200-G-06	Floor Sump Pump	11/14/94	11/23/94	12/19/94	Canmac Engineering Sales	$3,263	3/29/95
200-K-01	Air Compressor	10/18/94	10/31/94	1/4/95	Colorado Compressor	$16,953	4/24/95
200-Y-02	Microcel™ Flotation Column	10/21/94	11/4/94	12/1/94	Control International	$69,400	5/31/95
200-Y-03	In-Line Static Mixer	10/21/94	11/4/94	12/1/94	Control International	N/A	5/31/95
200-Y-04	Column Feed Agitator	11/10/94	11/23/94	2/16/95	D.W. Daigler (Lightnin)	$1,594	3/14/95
300-D-01	Slurry Storage Tank A	1/6/95	1/18/95	2/15/95	Metal-Craft	$3,836	5/2/95
300-D-01	Slurry Storage Tank B	1/6/95	1/18/95	2/15/95	Metal-Craft	$3,836	5/2/95
300-Y-01	Ground Product Agitator	2/1/95	2/2/95	2/16/95	D.W. Daigler (Lightnin)	$5,000	3/27/95
300-Y-02	Fine Grinding Mill Agitator	2/1/95	2/2/95	2/16/95	D.W. Daigler (Lightnin)	$5,000	3/27/95
400-D-01	Thickener	10/3/94	11/10/94	11/10/94	Enviro-Clear	$46,500	3/27/95
400-D-02	Thickener Overflow Sump	1/6/95	1/18/95	2/15/95	Metal-Craft	$5,691	5/2/95
400-D-04	Tailings Filter Feed Sump	1/6/95	1/18/95	2/15/95	Brewer Steel	$3,231	6/14/95
400-G-01	Filter Press Charge Pump	10/31/94	11/14/94	12/19/94	Quadna Pump Systems	$1,209	1/20/95
400-G-02	Filter Press Charge Pump	10/31/94	11/14/94	12/19/94	Quadna Pump Systems	$1,209	1/20/95
400-G-07	Clarified Water Pump	10/27/94	11/4/94	12/19/94	Canmac Engineering Sales	$3,835	5/11/95
400-G-11	Floor Sump Pump	10/31/94	11/14/94	12/19/94	Canmac Engineering Sales	$3,263	5/11/95
400-G-12	Cationic Metering Pump	2/28/95	3/6/95	3/6/95	Nalco Chemical Company	$1,550	3/16/95
400-G-13A	Anionic Metering Pump - A	2/28/95	3/6/95	3/6/95	Nalco Chemical Company	$1,550	3/16/95
400-G-13B	Anionic Metering Pump - B	2/28/95	3/6/95	3/6/95	Nalco Chemical Company	$1,550	3/16/95
400-T-03	Clean Coal Conveyor	1/19/95	2/3/95	2/28/95	Rocky Mountain Conveyor	$18,599	6/1/95
400-T-04	Clean Coal Conveyor	1/19/95	2/3/95	2/28/95	Rocky Mountain Conveyor	$18,599	6/1/95
400-Y-06	Thickener Mechanism	10/3/94	10/6/94	11/10/94	Enviro-Clear	N/A	3/16/95
400-Y-08	Filter Press Sump Agitator	11/4/94	11/10/94	2/16/95	D.W. Daigler (Lightnin)	$2,034	3/14/95

Table 32. Status of Instrumentation

TAG Number	Description	Company	Price	Delivery
DIT-203	Densitometer	Winn Marion	$ 5,065	
FIT-104	Flow Transmitter	PCI Sales	$ 1,845	6/29/95
FIT-105	Flow Transmitter	FAMCO	$ 963	
FIT-114	Flow Transmitter	FAMCO	$ 1,142	
FIT-116	Flow Transmitter	FAMCO	$ 963	
FIT-117	Flow Transmitter	FAMCO	$ 1,396	
FIT-202	Flow Transmitter	JMC Instruments	$ 2,544	6/28/95
FIT-204	Flow Transmitter	PCI Sales	$ 2,425	6/29/95
FIT-205	Flow Transmitter	PCI Sales	$ 2,230	6/29/95
FIT-206	Flow Transmitter	Power Controls	$ 630	
FI-207	Flow Gauge	PCI Sales	$ 1,570	6/29/95
FIT-209	Flow Transmitter	FAMCO	$ 1,278	
FIT-211	Flow Transmitter	JMC Instruments	$ 2,647	6/26/95
FIT-215	Flow Transmitter	Power Controls	$ 630	
LIT-101	Level Transmitter	DISCO	$ 1,061	6/16/95
LT-114	Level Transmitter	DISCO	$ 936	6/16/95
LT-118	Level Transmitter	DISCO	$ 936	6/16/95
LT-201	Level Transmitter	DISCO	$ 936	6/16/95
LT-213	Level Transmitter	DISCO	$ 936	6/16/95
LIT-401	Level Transmitter	DISCO	$ 936	6/16/95
LT-402	Level Transmitter	DISCO	$ 936	6/16/95
LT-411	Level Transmitter	DISCO	$ 936	6/16/95
LT-421	Level Transmitter	DISCO	$ 936	6/16/95
LT-422	Level Transmitter	DISCO	$ 936	6/16/95
PI-208	Pressure Gauge	JMC Instruments	$ 46	
PT-210	Press Transmitter	JMC Instruments	$ 608	
PI-214	Pressure Gauge	JMC Instruments	$ 106	
PIT-403	Press Transmitter	JMC Instruments	$ 806	
PI-414	Pressure Gauge	JMC Instruments	$ 106	
PI-423	Pressure Gauge	JMC Instruments	$ 106	
TI-110	Temp Gauge	JMC Instruments	$ 96	6/26/95
PCV-102	Valve - 2"	Winn Marion	$ 1,074	
FV-104	Valve - 2"	Rampart	$ 122	6/7/95
LV-114	Valve 1-1/2"	Winn Marion	$ 1,232	
LV-118	Valve - 1"	Winn Marion	$ 1,049	
LV-201	Valve - 3 way	Power Controls	$ 3,043	6/19/95
DV-203	Valve - 3"	Winn Marion	$ 2,007	
FV-206	Valve - 3"	Winn Marion	$ 1,966	
LV-402	Valve - 2"	Davis & Davis	$ 440	6/14/95
LV-411	Valve - 3 way	Power Controls	$ 3,043	6/19/95
PCV-412	Valve - 1"	Rampart	$ 68	6/7/95
PCV-415	Valve - 3/4"	Rampart	$ 68	6/7/95
FV-415	Valve - 3/4"	Davis & Davis	$ 165	6/14/95
FV-412A	Valve - 1"	Davis & Davis	$ 190	6/14/95
FV-412B	Valve - 1"	Davis & Davis	$ 190	6/14/95
FE-206	Flow Transmitter	Power Controls	$ 172	6/12/95
FE-215	Flow Transmitter	Power Controls	$ 227	6/12/95
PI-206	Press Indicator	JMC Instruments	$ 106	
FCV-209	Control Valve	Winn Marion	$ 1,049	
IT-SG1	Power Monitor	Power Equipment	$ 1,501	6/20/95
IT-121	Motor Monitor	Power Equipment	$ 806	6/20/95

Control and Data Acquisition System (CDAS) Training

Because significant project savings can be realized, Entech Global personnel will perform a considerable portion of the CDAS configuration and programming. As such, Mr. Gene L. Shields, Senior Project Engineer, attended two formal training sessions at the Honeywell Automation College in Phoenix, Arizona. The training conducted April 3 through April 7, focused on programming the SCAN 3000 Data Acquisition Unit. A second class, conducted May 8 through May 12, focused on configuring the Series 9000 Controller.

Refurbishing of Existing PDU Equipment

Reconditioning of existing process equipment items commenced during the quarter. The Samplers as well as the Primary and Secondary Ball Mills have been reconditioned as needed. The status of all remaining equipment to be reconditioned is shown in Table 33.

Table 33. Existing Equipment Items for Reconditioning

Equipment No.	Description	Schedule	Estimated Cost
100-D-01	Dump Hopper	July, 1995	$800
100-D-02	Feed Bin	July, 1995	$800
100-T-03	Elevating Conveyor	July, 1995	$300
100-T-04	Weight Belt Feeder	July, 1995	$3,800
100-Y-01	Primary Ball Mill	Complete	$ 1,225
100-Y-02	Secondary Ball Mill	Complete	$ 1,225
400-G-06	Filtrate Pump	July, 1995	$100
400-Y-01	Tailings Filter Press - A	July, 1995	$3,400
400-Y-02	Tailings Filter Press - B	July, 1995	$3,400
400-Y-04	Clean Coal Filter Press - A	July, 1995	$16,472
400-Y-05	Clean Coal Filter Press - B	July, 1995	$16,472
400-Y-09	Clean Coal Drum Filter	July, 1995	$1,500
N/A	Samplers	Complete	$2,000

Assembly of Spare Parts List

A spare parts list has been assembled and short listed. Only wear items that are most likely to fail during the first year of operation will be purchased. Ordering of spare parts commenced during the quarter.

Transformer Installation

A Purchase Order was issued to Public Service Company of Colorado for installation of one 1500 kVA transformer as well as overhead service from McIntyre Street. A meeting with Mr. Leonard Ellis of the Public Service Company on April 19, 1995, revealed that the transformer and overhead lines should be installed during the week of July 10, 1995.

Equipment Hold Points and Shop Inspections

Air Compressor (200-K-01) - Inspection of the Gardner-Denver air compressor was conducted at the manufacturer's Sedalia, Missouri, assembly plant on April 21, 1995. Inspection of the unit and associated performance test data revealed no flaws or ambiguities. The compressor was released for shipment and delivered April 24, 1995.

Fine Grinding Mill (100-Y-03) - Inspection of the Netzsch Fine Grinding Mill was conducted on April 26 & 27 at the Netzsch facility located in Exton, Pennsylvania. A rotational test and sound test were conducted on April 26 while a pressure/leak test and electrical interlock test were conducted April 27. Overall, the unit passed or exceeded all required test parameters. The unit was shipped April 28 and arrived at the PDU site the first week of May.

Microcel™ Flotation Column (200-Y-02) - Inspection of the Microcel™ was conducted on May 5, 1995, at the fabrication facility located in Salt Lake City, Utah. Visual inspection revealed that the unit passed or exceeded all required test parameters. The unit was received at the PDU site May 31, 1995.

On-Line Ash Analysis of Microcel™ Flotation Column

Tests conducted on coal slurry samples at Nalco Chemical Company's Richlands, Virginia laboratory indicate that the ash probes work very well on the flotation tailings but not on the clean coal. Accurate results were obtained only at ash values greater than 40%. As a result, Entech engineers are re-evaluating the use of such probes in the PDU.

Project Review Meeting

A project review meeting was conducted May 31 at the PDU site with Bechtel, TIC, and Entech Global personnel. Those in attendance included:

- S. Bajwa - Bechtel
- M.V. Chari - Bechtel
- H. Huettenhain - Bechtel

- M. Jha - Entech Global
- G. Shields - Entech Global
- F. Smit - Entech Global
- J. Miller - TIC
- P. Lauretano - TIC

A presentation was made by Entech Global and TIC personnel regarding the status and details of the PDU construction. Following a brief discussion, all attendees toured the PDU. Overall, the attendees were quite pleased with the progress of the Flotation Module construction.

<u>Miscellaneous Accomplishments</u>

The following miscellaneous accomplishments were made during the quarter:

- Received WesTech Drum Filter capacity information from John Smith at SEPCO. Mr. Smith said that the capacity of the filter when treating 28x0 clean coal should be about 2,500 lb/hr (dry) while that for 100x0 (60% minus 200 mesh) would be about 2,200 lb/hr (dry).
- Ordered and received 1,800 pounds of 3mm glass beads for use in the Netzsch fine grinding mill. Media will be transferred to the fine grinding mill during the PDU startup period.

PLANS FOR NEXT QUARTER

Following are the activities anticipated for continued work and/or completion during the April - June, 1995 quarterly reporting period:

- Subtask 3.2 - Near Term Applications testing will continue
- The Subtask 6.3 topical report, Process Optimization will be finalized after comments are received from various project team members
- Work will continue on Subtask 6.4, Selective Agglomeration - CWF Formulation Studies
- Efforts for the Subtask 6.5 work will focus on completing the Subtask 6.5 test work, and providing Bechtel with necessary engineering design data
- Work will continue on the Task 7 detailed design of the selective agglomeration portion of the 2 t/hr PDU
- Subtask 8.2 efforts will be directed toward the following:
  - Complete construction of PDU Flotation Module
  - Complete PDU Flotation Module startup activities
- Subtask 8.3 efforts will focus on:
  - Initiate parametric testing of Taggart seam coal in PDU Flotation Module
  - Complete test plan for PDU Flotation Module;

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11. Bechtel Corporation, "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Task 3. Development of Near-Term Applications, Subtask 3.1 Engineering Analysis Conceptual Designs and Cost Estimates", Report to the U. S. Department of Energy, November 5, 1993.

12. Smit, F. J., and Jha, M. C., "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 2.1 Coal Selection Plan and Recommendations", Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208, April 29, 1993.

13. Moro, N., Smit F. J., and Jha, M. C., "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 4.3 CWF Formulation Studies Topical Report", Amax R&D Report to the U. S. Department of Energy Contract DE-AC22-92PC92208, May 23, 1995.

14. Smit, F. J., and Jha, M. C., "Engineering Development of Advanced Physical Fine Coal Cleaning for Premium Fuel Applications, Subtask 6.3 Selective Agglomeration Process Optimization Research Draft Topical Report", Amax R&D Draft Report to the U. S. Department of Energy Contract DE-AC22-92PC92208, June 30, 1995.