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JULY - SEPTEMBER, 1995

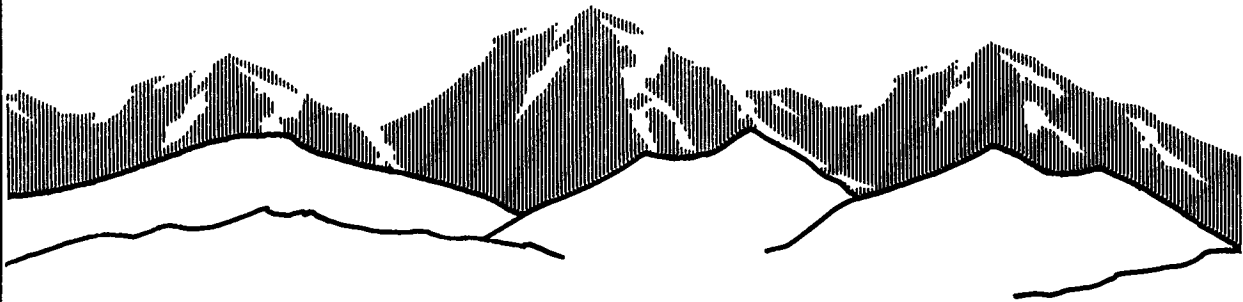
**ENGINEERING DEVELOPMENT OF ADVANCED PHYSICAL  
FINE COAL CLEANING FOR PREMIUM FUEL APPLICATIONS**

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  
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DOE Contract No. DE-AC22-92PC92208  
Amax R&D Project No. 91455

October 31, 1995



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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 and operation of a 2-t/hr process development unit . The project began in October, 1992, and is scheduled for completion by June, 1997.

During Quarter 12 (July - September 1995), work continued on the Subtask 3.2 in-plant testing of the Microcel™ flotation column at Lady Dunn. Testing produced clean coal meeting the 7 to 9% product ash target from both minus 48- and minus 100-mesh streams, with combustible recoveries of 65 to 80%. Twelve drums of clean coal were shipped to PETC for centrifuge dewatering tests using their GranuFlow process. Subtask 3.3 testing, investigating a novel Hydrophobic Dewatering (HD) process, began this quarter. Work included work plan development, collection of coal samples, identification of suitable hydrocarbon substances, and design of the batch HD test unit.

Under Subtask 4.4, additional toxic trace element analysis of column flotation samples finalized the data set. Data analysis indicates that reasonably good mass balances were achieved for most elements. Significant reductions of many element concentrations were observed from raw coal, to flotation feed, to flotation product. The detailed design of the PDU flotation unit, Task 5, was completed this quarter with the submission of the final design package.

The final Subtask 6.3 Selective Agglomeration Process Optimization topical report was issued this quarter. Preliminary Subtask 6.4 work investigating coal-water-fuel slurry formulation indicated that selective agglomeration products formulate slurries with lower viscosities than advanced flotation products. Work continued on Subtask 6.5 agglomeration bench-scale testing. Results indicate that a 2 lb ash/MBtu product could be produced at a 100-mesh topsize with the Elkhorn No. 3 coal. Results with the new Taggart coal indicated that a 100-mesh grind would be required to achieve the desired 1 lb/MBtu product ash specification. In addition, construction and initial testing of a 2-stage steam stripping circuit for heptane recovery was completed.

The detailed design of the 2 t/hr selective agglomeration module neared completion this quarter with the completion of additional revisions of both the process flow, and the process piping and instrument diagrams. In addition, work started on the preparation of the bid package for construction of the selective agglomeration module. Procurement of coal for PDU operation continued, and the Subtask 8.1 topical report was issued. The three coals selected for PDU operation are Taggart coal from Wise County, West Virginia, Indiana VII coal from Sullivan County, Indiana, and Hiawatha coal from Emery County, Utah. Construction of the 2 t/hr PDU and advanced flotation module was completed this quarter and startup and shakedown testing began.

## **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 12th quarter of the project from July 1 to September 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 Doohar 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 July - September 1995 quarter on Phases I and II of the project. Work was carried out under 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. In addition, subtask 3.3, investigating a novel dewatering process for advanced flotation products will also be performed by CCMP.

#### **Subtask 3.2 Engineering Development**

Performance data were obtained from the 30-inch Microcel™ column installed in the Lady Dunn Preparation Plant during the last reporting quarter. The purpose of this work is to test the near-term application of advanced column flotation technology for recovering clean coal from the fines generated in a coal washing plant. A high-quality product meeting the plant requirement of 7 to 9 percent ash was produced during initial testing on minus 48-mesh and minus 100-mesh streams. Combustible recoveries were in the 65 to 80 percent range.

Twelve drums of the clean coal froth were collected and shipped to the Pittsburgh Energy Technology Center (PETC) for centrifuge dewatering tests using their GranuFlow process. Lady Dunn plant management also began making arrangements for an in-plant test of a horizontal belt vacuum filtration unit.

### **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 hydrophobic dewatering (HD) process for clean coal fines. In this process a hydrophobic substance is added to a coal-water slurry to displace water from the coal surface. The hydrophobic substance is then recovered for recycle to the process. Three coals will be tested including the product from near-term testing at the Lady Dunn plant (Subtask 3.2). Subtask 3.3 was further divided into five additional subtasks as follows:

- Subtask 3.3.1 - Project Planning
- Subtask 3.3.2 - Identification of Hydrophobic Substances
- Subtask 3.3.3 - Process Development
- Subtask 3.3.4 - Design/Operation of Continuous Bench-scale Unit
- Subtask 3.3.5 - Economic Analysis

Work was carried out on Subtasks 3.3.1, 3.3.2, and 3.3.3 during this quarter.

**Subtask 3.3.1, Project Planning** - During this reporting quarter a detailed work plan was prepared and submitted to DOE. This plan contained a description of the test plan, experimental procedures, analytical methods, and reporting guidelines for implementation and completion of the project.

**Subtask 3.3.2, Identification of Hydrophobic Substances** - To identify suitable hydrophobic substances that can readily displace water from coal, contact angle measurements were conducted during this reporting quarter (3.3.2.1). Thermodynamically, when the contact angle, defined as the angle between the liquid and coal surface as measured through the liquid phase, becomes greater than 90°, the displacement of water by the hydrophobic substance on the coal surface becomes spontaneous. As the contact angle further increases, the better the hydrophobic substance performs as a dewatering agent.

To evaluate the dewatering potential of the coals proposed, average water contact angles were determined. Based on the low water contact angle of the Indiana VII coal, due to its low rank, hydrocarbon contact angle determinations were limited to the Sunnyside and Elkhorn No. 3 coals.

Subsequently, contact angle measurements were carried out on Elkhorn No. 3, and Sunnyside coals using pentane, hexane, heptane, octane, and decane paraffinic hydrocarbons. Results indicated that as the hydrocarbon chain length increases, the contact angle decreases. As such, pentane is potentially the best dewatering agent tested. During the next quarter, two hydrocarbons of shorter chain length, butane and propane, will be evaluated in a high pressure contact angle cell.

**Subtask 3.3.3, Process Development** - Samples of the Indiana VII and Sunnyside coals were obtained from AMAX R&D during this quarter as part of Subtask 3.3.3.1, Coal Sample Acquisition, Preparation and Characterization. Under Subtask 3.3.3.2, a batch dewatering unit was designed to test the HD process. The design incorporates the following:

- A high pressure cell to keep the hydrocarbons in a liquid form
- A variable speed mixer to provide adequate mixing
- A transparent window for visual observation
- A pressurized sampling vessel
- Continuous monitoring of temperature, pressure, and mixer speed

Once this unit is constructed by PARR Instruments, it will be tested under Subtask 3.3.3.3. Recovery and regeneration of the hydrophobic substance will be tested separately under Subtask 3.3.3.4.

#### **Task 4 Engineering Development of Froth Flotation**

Task 4 is divided into five subtasks. Subtasks 4.1 Grinding, 4.2 Process Optimization Research, 4.3 Coal-Water-Fuel Formulation Studies, 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 the remaining subtask, 4.4, during the twelfth quarter of this project.

#### **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 the previous quarter for additional analyses, particularly sulfur and toxic trace elements. The discrepancies noted last quarter regarding the toxic trace element analyses were resolved by repeating the antimony, lead and mercury analyses. It was found that a more rigorous dissolution and dilution procedure improved the consistency of the antimony results. With the revised analyses, the mass balances generally closed within 20 percent. The concentrations of arsenic, beryllium, chromium, cobalt, lead, manganese, mercury and selenium in the raw coals were clearly reduced by the combined washing and advanced cleaning steps. Also very definitely, column flotation reduced the concentrations of arsenic, chromium, manganese, mercury, and nickel remaining in the washed coal from the preparation plants.

It appears that the antimony concentration, and in certain cases, the nickel, selenium and chlorine concentrations as well, increased on a heating value basis. Such results could indicate a preferential association of these particular elements with a highly floatable component of the carbonaceous portion of the coal. The amounts of cadmium

in the coals and flotation products were usually too small to be detected so the reduction in cadmium concentration accomplished by flotation could not be quantified.

A draft of the Subtask 4.4 topical report containing the bench-scale flotation work and the toxic trace element reduction data was nearing completion at quarter's end.

### **Task 5 Detailed Design of PDU and Advanced Flotation Module**

The final detailed engineering design package for the PDU and the advanced flotation module was submitted to DOE during August. This package represents the final engineering design utilized for construction of the PDU. This engineering package was divided into three volumes as follows:

- Design Package
- Construction Specifications
- Material Requisitions

With the issuing of this design package, the Task 5 work is complete.

### **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 reporting quarter.

#### **Subtask 6.3 Process Optimization Research**

The final version of the Subtask 6.3 Topical Report was submitted to DOE and project team partners on September 28, 1995. 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 (non-recovery type agglomerating agents) indicated that neither quality or cost goals could be met.

#### **Subtask 6.4 CWF Formulation Studies**

The primary objective of Subtask 6.4 is to evaluate the formulation of coal-water-fuel (CWF) slurries from selective agglomeration products. The slurry feedstock used for this work was generated during Subtask 6.5, selective agglomeration bench-scale testing.

While much of this test work will evaluate the effect of various parameters on slurry quality, there are two other objectives for the Subtask 6.4 work. First, this test work will provide a comparison between similar slurries formulated from flotation and agglomeration products. This information will provide some insight into whether one process generates a product inherently more amenable to highly-loaded slurry formulation than the other process. Second, the Subtask 6.4 work will attempt to determine slurry quality guidelines for commercial production. To this end, determinations of required slurry coal loadings, stabilities, and viscosities will be carried out.

The Subtask 6.4 testing began in earnest during this twelfth quarterly reporting period. The initial work involved particle size distributions (PSD) characterization of Subtask 6.5 steam stripping circuit products, i.e., following heptane removal from the recovered agglomerates. PSD characterization was completed for the following coals:

- Taggart 1 (62-mesh topsize) - The Taggart coal utilized for all of the Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 start-up testing.
- Taggart 2 (62-mesh topsize) - The replacement Taggart coal which will be utilized for the remainder of project testing. While this coal comes from the same seam as the Taggart 1 coal, it is from a different mine.
- Sunnyside (150-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 testing. This coal has since been replaced with the Hiawatha coal.
- Elkhorn No. 3 (100-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.
- Indiana VII (325-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.

Utilizing these Subtask 6.5 products, testing was done to characterize the formulation of slurries from "as-received" agglomeration circuit products. Testing was also carried out to provide a comparison between the formulation of slurries from advanced flotation products and from selective agglomeration products. This was accomplished by repeating the formulation of various slurries carried out during Subtask 4.3 testing.

It should be noted that the 62-mesh Taggart 1 agglomeration product will be used only to provide a comparison of slurry formulation characteristics with the Subtask 4.3 results. The replacement Taggart 2 coal will then be used for the bulk of the Subtask 6.4 test work. Similarly, the Sunnyside agglomeration product will also be utilized only to provide a comparison with previous results, since it has been replaced with the Hiawatha coal (to be used for the bulk of the Subtask 6.4 test work).

**Taggart 1 Coal** - Slurries were formulated using three different types of 62-mesh topsize Taggart 1 coal selective agglomeration product feedstock as follows:

- As-received agglomeration product utilizing 0.5% of A-23 dispersant. Coal loadings for these slurries were in the range of 61 to 67% by weight. These results provide a baseline with which to gauge the quality of other slurries formulated from this coal.
- A blend of 70% as-received material and 30% material reground for 30 minutes in the attritor mill utilizing 0.5% A-23 dispersant. This blend is referred to as the 70/30 regrind blend, and is utilized to generate more fines to improve slurry particle packing characteristics. Coal loadings for these slurries ranged from 55 to 68% by weight. Two slurry formulations were also repeated using Flocon for stabilization.
- A blend of 42.9% +325M material, 27.1% -325M material, and 27.1% -325M material reground for 30 minutes in the attritor mill utilizing 0.5% of A-23 dispersant. This blend is referred to as the 325 mesh regrind blend, and was originally formulated during Subtask 4.3 testing to generate a bimodal PSD. Coal loadings for these slurries were in the range 60 to 70% by weight. Two slurry formulations were also repeated using Flocon for stabilization.

Results from this work indicate that slurries formulated from the 70/30 regrind blend achieved lower viscosities at similar coal loadings than the slurries formulated from the 100% as-received agglomeration product. This confirms the trend observed during Subtask 4.3 testing. Similarly, slurries formulated from the 325-mesh regrind blend provided additional viscosity reduction at similar coal loadings. This additional viscosity reduction also confirms Subtask 4.3 results, and is due to increased particle packing within the slurry as a result of PSD manipulation. Also, as expected, the use of Flocon to improve stability as discussed below results in increased slurry viscosity at the same slurry coal loadings for both the 70/30 blend and the 325-mesh regrind slurry formulations.

In comparing these Subtask 6.4 results to those achieved during Subtask 4.3 testing, similar trends are observed in terms of reduced slurry viscosity with PSD manipulation. The data also indicates that lower viscosities, at similar coal loadings, are achieved with the selective agglomeration circuit product than with the column flotation product. It is believed that this may be due to a number reasons including:



- The presence of aggregates of particles in the steam stripped agglomeration circuit product, effectively coarsening the PSD.
- An effect of the steam stripping process on the surface properties of the coal.
- The presence of residual heptane in the agglomeration circuit product.

All of the slurries formulated with this Taggart 1 coal, except those that utilized Flocon, had poor stabilities with a rating of "1" after overnight storage. While this rating of "1" indicates that at least 25% of the slurry volume was occupied by a very hard pack sediment, for all of these slurries, 60-90% was very hard packed. When Flocon was used, however, stabilities were in the "3" to "4" range after overnight storage, indicating that only about 5-10% of the slurry formed a very hard packed sediment.

**Sunnyside Coal** - Slurries were formulated using two different types of 150-mesh topsize Sunnyside coal agglomeration product feedstock as follows:

- As-received agglomeration product with 1% A-23 dispersant. Coal loadings for these slurries were in the 56 to 62% range by weight. Two slurry formulations were also repeated using Flocon.
- A blend of 70% as-received material and 30% material reground for 30 minutes in the attritor mill utilizing 0.5% A-23 dispersant. This blend is referred to as the 70/30 regrind blend. Coal loadings for these slurries were in the range of 59 to 65% by weight. Two of these slurry formulations were also repeated using Flocon for stabilization.

As expected based on previous experience, slurries formulated from the 70/30 regrind blend achieved lower viscosities at similar coal loadings than slurries using 100% as-received agglomeration product. Also, the use of Flocon to improve stability resulted in increased slurry viscosity over slurries formulated at the same coal loadings that did not use Flocon.

In comparison to Subtask 4.3 results, this data indicates that the quality of slurries formulated from flotation products (both as received and the 70/30 regrind blend) are similar to that achieved with the 100% as-received agglomeration product. However, slurries formulated from the 70/30 regrind blend of agglomeration product achieved lower viscosities, at similar coal loadings, than slurries formulated from both as received and 70/30 regrind blend flotation product feedstocks.

All of the slurries formulated with the Sunnyside coal, except those that utilized Flocon, had poor stabilities with a rating of "1" after overnight storage. When Flocon was used, however, stabilities were rated in the "3" to "4" range after overnight storage.

**Indiana VII Coal** - Indiana VII coal slurries were formulated from as-received agglomeration product utilizing 1% and 2% A-23 dispersant levels. Coal loadings for these slurries were in the range 49 to 53% by weight. These results provide a baseline

with which to gauge the quality of other slurries formulated from this coal and also provide a comparison with slurries formulated during Subtask 4.3 testing.

Results from this work indicate that all slurries made with this Indiana VII coal followed the same coal loading vs slurry viscosity trend, regardless of the quantity of A-23 dispersant utilized. It should be noted that no regrind blends were formulated with this coal due to the fineness (325-mesh topsize) of this agglomeration product. Future testing may investigate the formulation of coarsened slurry feedstock material, if appropriate.

Comparing these results to the Subtask 4.3 work, it is found that like the other two coals evaluated this quarter, slurries formulated from flotation product (both with 1% and 2% A-23) had higher slurry viscosities at similar coal loadings than those slurries formulated from the agglomeration product. As with the two previous coals, all of the Indiana VII coal slurries had poor stabilities. However, no Flocon was utilized in the formulation of these Indiana VII coal slurries.

### **Subtask 6.5 Bench-Scale Testing and Process Scale-up**

The Subtask 6.5 test work progressed significantly during this reporting quarter. During the previous reporting quarter, construction and shakedown testing of the 25 lb/hr bench-scale unit was completed. In addition, evaluation of the Taggart 1 and Sunnyside coals was carried out. These two coals have since been replaced with the Taggart 2 and Hiawatha coals, respectively, and as such will not be tested further in the bench-scale agglomeration unit. In addition, testing during the last quarter evaluated the agglomeration characteristics of the Indiana VII coal. This work indicated that product ash specifications could be met at the selected 325-mesh topsize grind.

A number of test conditions were evaluated in the 25 lb/hr selective agglomeration test unit during this quarter, utilizing the Elkhorn No. 3 coal ground to a 100-mesh topsize and the Taggart 2 coal ground to a 62-mesh topsize. A de-aromatized commercial grade heptane and a pure grade of heptane were used.

Evaluation of steam stripping recovery of heptane from agglomerated products continued. In an effort to confirm the 2 t/hr PDU steam stripping circuit design, the bench-scale unit was modified to allow testing of a two-stage stripping circuit.

**Elkhorn No. 3 Coal Testing** - A total of 32 agglomeration circuit test runs were completed this reporting period to evaluate the performance of the continuous agglomeration unit for the 100-mesh topsize grind Elkhorn No. 3 coal. The coal was closed-circuit ground to a 100-mesh topsize in the 4' x 4' ball mill. This grind provided a coal particle size distribution (PSD) similar to that utilized during the Subtask 4.3 advanced flotation test work.

The following conditions were held constant for all of the tests:

- 100-mesh topsize grind
- 2.4-inch (4-blade) high-shear impeller
- 4.8-inch (4-blade) low-shear impeller
- Commercial grade heptane except for one test which used pure heptane
- Screen spray water flow rate

The following variables were changed during this testing:

- High-shear impeller tip speed
- High-shear residence time
- Low-shear impeller tip speed
- Low-shear residence time
- Heptane concentration
- Solids concentration
- Screen inclination
- Heptane type (pure or commercial)

The 2 lb ash/MBtu product specification was met for all but one of the tests completed. This indicates that the 100-mesh topsize grind provides sufficient mineral-matter liberation. As such, no other grinds were evaluated. Agglomeration Btu recoveries achieved were in the range of 88 to 99%, equivalent to approximately 83 to 94% on a run of mine (ROM) basis. As such, sufficient energy recovery was achieved for all of the tests completed to meet the project goal of 80% energy recovery on a ROM basis.

While these tests achieved the project product ash and Btu recovery specifications, it proved difficult to achieve consistently high Btu recoveries, which is generally easy for the selective agglomeration process. This is indicated by the tailings ash values for this Elkhorn No. 3 coal test work which ranged from 24.8% to a high of 73.2% (mostly in the 35 to 55% range). These relatively low tailings ash values are attributed to aging of the Elkhorn coal which had been stored for over two years prior to its use for this testing. To overcome this effect, asphalt was utilized to enhance agglomeration in several tests. With the use of asphalt, Btu recoveries of 98.7 to 99.1% were achieved, with associated tailings ash values ranging from 66.9 to 73.2%. In contrast, when asphalt was not used to enhance agglomeration, the highest Btu recoveries achieved were in the 96 to 98% range.

Primary to achieving high Btu recoveries without the use of asphalt was the total energy input to the combined high- and low-shear unit operations, with higher energy inputs achieving higher Btu recoveries. As such, when higher impeller tip speeds and longer agglomeration residence times were utilized, higher Btu recoveries were generally observed.

It was found that tests utilizing a 13% solids concentrations achieved higher energy recoveries than similar tests carried out at 10 and 7% solids concentration. This confirms previous results indicating that agglomeration carried out at a higher solids concentration requires less energy than at a lower solids concentration. This effect is due to the occurrence of more particle to particle contact at the higher solids loadings.

One other important factor in achieving high Btu recoveries was the utilization of sufficient heptane. Most of the initial tests used heptane dosages in the range of 24 to 29% on a dry ash free (daf) coal basis. Of these tests, only the one with the highest overall energy input achieved a Btu recovery greater than 97%. In contrast, for the remainder of the Elkhorn tests which utilized heptane dosages of 30 to 35% daf, most of the tests achieved Btu recoveries greater than 97%.

The drainage of mineral-matter bearing process water is dependent primarily on the size of the agglomerates, with better formed agglomerates achieving lower product ash contents due to better drainage. This confirms the trend observed during previous testing with other coals. In some cases a very poorly formed agglomerated product was discharged from the low shear reactor. For these tests, manual assistance was often required to maintain the flow of agglomerates across the screen. This was the case even when the screen inclination was increased to improve agglomerate flow. This phenomenon was found to be more prominent at lower agglomeration energy input levels, i.e., lower high- and low-shear impeller tip speeds.

The size of the agglomerates formed generally determines the product ash content, with larger agglomerates achieving lower product ash values, due primarily to better drainage of mineral-matter bearing process water. No other parameters evaluated had a noticeable effect on product ash content, except indirectly where they ultimately affected agglomerate size and strength.

Based on these results, it is apparent that both product ash content and Btu recovery specifications can be met with the 100-mesh topsize grind for the Elkhorn coal. The best results for agglomeration of this coal were achieved when sufficient bridging liquid and energy were utilized to insure the production of well formed agglomerates from low shear. When this was the case, the agglomerates screened well, resulting in product ash values of 2.5 to 2.7%, equivalent to approximately 1.7 to 1.9 lb ash/MBtu.

As such, with the completion of the test work reported here, continuous agglomeration testing of the Elkhorn No. 3 coal is considered complete.

**Taggart 2 Coal Testing** - Preliminary work was carried out during this reporting quarter utilizing the replacement Taggart coal (Taggart 2). Based on previous work with this coal, a closed circuit 62-mesh topsize grind was completed in the 4' x 4' ball mill. Five continuous agglomeration tests were completed with this coal.

The primary variables changed during the completion of the five agglomeration tests were:

- Coal feed rate/residence time
- High-shear impeller tip speed
- Low-shear residence time
- Heptane concentration

Just like Taggart 1 coal, the Taggart 2 coal was easy to agglomerate. This was confirmed by tests in which a high-shear impeller tip speed of 6.4 m/s proved sufficient to achieve acceptable inversion, as compared to other coals requiring impeller tip speeds in the range of 15-18 m/s.

Also, as determined during start-up testing with the Taggart 1 coal, difficulties were encountered at the low-shear discharge point. This lack of flow from the low-shear vessel is attributed to the extreme buoyancy of the agglomerated Taggart coal product. While changes to the low-shear discharge arrangement were made during start-up testing, with some improvement realized, additional modifications may be necessary during future Taggart coal testing.

This low-shear discharge plugging was a problem for all Taggart 2 tests completed except when a 50 lb/hr coal feed rate was used. Under these operating conditions, the higher volumetric flowrate proved sufficient to prevent plugging of the low-shear discharge. One other point to note from this work is that the lowest product ash content and the highest product solids concentration were achieved when the largest agglomerated product (up to 4 mm) was produced. This confirms a general trend observed through the bulk of all testing to date.

Results from these tests indicate that while the 62-mesh topsize grind achieved the 2 lb ash/MBtu product specification, it did not provide sufficient mineral-matter liberation to achieve the desired product ash specification of 1 lb ash/MBtu. As such, additional test work with this coal will be completed utilizing a 100-mesh topsize grind.

**Winifrede Coal Testing** - Work began on grinding the Winifrede coal for evaluation in the continuous bench-scale agglomeration unit. As of the end of this reporting quarter, the Winifrede coal had been ground in the 4' x 4' ball mill followed by a single pass through the Drais Stirred Ball Mill.

This ground Winifrede coal had a mass mean diameter of approximately 13 microns, with about 80% passing 20 microns. This ground product was evaluated in batch agglomeration testing to determine additional liberation requirements. Results indicated that the 2 lb ash/MBtu product specification could not be met. As such, additional passes through the Drais Mill will be carried out until sufficient liberation is achieved.

**Continuous Steam Stripper Testing** - During the previous quarter, batch stripping tests were carried out to evaluate the effect of elevated temperatures on steam stripping recovery of heptane from agglomerated products. Results from this test work

indicated that increased temperature resulted in reduced heptane concentrations as compared to the continuous stripper product, generated at boiling point (94°C). As such, a two-stage continuous stripping circuit was constructed to test these results.

Work was completed during this reporting period on the design and construction of the modified two-stage continuous steam stripping circuit. This circuit utilizes two stages of stripping as is planned for use in the 2 t/hr PDU selective agglomeration module. The design criteria for this unit was to allow the processing of 25 lb/hr of coal through a two-stage stripping circuit at 20-25% solids concentration. This unit also utilizes a counter-current steam flow in which the second stage stripper vapor product is utilized as the steam feed to the first stage stripper.

This modified stripping circuit is fed with a peristaltic pump from a newly constructed feed tank. A new first-stage stripping vessel was also built to provide approximately 5-minutes residence time at typical feed rates. This vessel was fitted with an agitator and designed to be operated at ambient pressure, and therefore operates in the temperature range of about 92-94°C. During this first stage of stripping, the bulk of the heptane is removed, and a more handleable stream produced.

The product from this first-stage stripper is pumped to the second-stage stripper by a variable speed Moyno pump. The speed of this pump is manually controlled to maintain a constant level in the first-stage stripper, visible in a sight tube. The second stage steam stripping vessel is the previously utilized stripping column (4-inches in diameter), lengthened to provide a residence time of approximately 10 minutes. This unit was modified to allow its operation at a positive pressure of 15-20 psig. As such, the operating temperature in this second-stage stripper can be maintained in the 115-120°C range. The operating level of this second-stage stripper is maintained at a relatively constant level via manual operation of the product discharge valve. The desired operating pressure and temperature are maintained in this vessel by manual operation of the vapor discharge control valve. As before, this vessel was partially filled with 5/8-inch pall rings to insure steam distribution across the column's cross-sectional area.

Three tests were completed using this modified two-stage stripping circuit during this reporting period. All three of these tests utilized Elkhorn No. 3 coal agglomerated product from the continuous agglomeration circuit. The first test completed used agglomerated Elkhorn coal that had been in storage for several days. For the other two continuous tests, the agglomeration circuit screen product was fed directly to the stripping circuit feed tank. In this manner, the entire agglomeration/stripping unit was run together. While the feedstock for the first two tests had been agglomerated with commercial grade heptane, the last test carried out utilized feedstock agglomerated with pure heptane.

The results of these two-stage stripping tests indicate that two stages of steam stripping, as tested here, achieved lower trace heptane concentrations than a single stage of steam stripping. This is believed to be due primarily to the increased

temperature in the second stage. Additional testing will be carried out with other project goals to confirm the results.

**Tailings Heptane Analyses** - Samples of agglomeration tailings (froth skimmer underflow) were sent for trace heptane analyses. These samples originated from an Elkhorn coal agglomeration test in which approximately 50% tailings ash was achieved utilizing commercial grade heptane. Samples submitted included as received tailings, tailings filter cake, tailings filtrate, and tailings samples that had been boiled for 5, 10, and 20 minutes. Less than 10 ppm of n-heptane was detected in all of the tailings samples, except for the filter cake, which contained 380 ppm n-heptane, at 67% solids, or 567 ppm n-heptane on a dry solids basis. There was less than 1 ppm of n-heptane detected in the tailings filtrate.

### **Task 7 PDU Selective Agglomeration Module Detailed Design**

Several 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 and the construction bid package can be completed in a timely manner. The following sections of this Executive Summary 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 switch over from the flotation to agglomeration process module. As such, the agglomeration process will use slurry ground by the installed equipment.

The ground product will then be sent to what is currently the Microcel™ flotation column feed tank. The product stream from this tank is monitored by a density meter, whose determination is utilized to dilute the out flowing slurry to a target solids concentration. The product from this feed tank will feed one of two ground slurry storage tanks. From these storage tanks, the slurry will be 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. 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. Each high-shear agglomerator will be powered by variable speed drive units that can achieve up to 18 m/s impeller tip speeds..

Heptane will be metered to the agglomeration process as required, currently anticipated not to exceed 40% of the coal feed rate. The ability will be provided to add bridging liquid to both high shear vessels and the low shear vessel. 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. The ability to add asphalt to the high-shear circuit will also be provided for evaluation of 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 6-bladed impeller for each section of the vessel. The impellers will be of the radial-flow type and the drive unit of sufficient Hp to achieve tip speeds in the 3-5 m/s range. Under some operating conditions, primarily to control excessive agglomerate growth, it is desirable to achieve low-shear impeller tip speeds of greater than 5 m/s. To accomplish this without the incorporation of a significantly larger drive unit, the low-shear impellers will have four hinged blades, so that when run in the opposite rotation direction, much less power will be drawn.

**Agglomerate Recovery** - 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 3-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 115-120°C and 15-20 psig pressure.



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.

**Design Status** - As of the end of this reporting quarter, the design has been virtually finalized. By the end of October, it is anticipated that the P&ID's, will be finalized. Also within this same time frame, material data sheets should be completed for all major pieces of equipment and all of the required instrumentation. It is currently anticipated that the construction bid package will be completed by the end of November. As such, purchasing will begin during November with a construction start date scheduled for mid to late January, 1996.

### **Task 8 PDU and Advanced Column Flotation Module**

Work completed as part of Task 8 this reporting quarter involved coal selection and procurement under Subtask 8.1, construction of the PDU and advanced flotation module under Subtask 8.2, and shakedown testing under Subtask 8.3

#### **Subtask 8.1 Coal Selection and Procurement**

The Subtask 8.1 PDU Coal Selection Recommendations Topical Report was issued August 17, 1995. The recommended coals and source mines were as follows:

- Taggart Coal
  - Steer Branch Mine
  - Red River Mining Company
  - Wise County, Virginia
- Indiana VII Coal
  - Minnehaha Mine
  - Cyprus Amax Coal Company
  - Sullivan County, Indiana
- Hiawatha Coal
  - Crandall Creek Mine
  - Genwal Mining Company
  - Emery County, Utah

The Hiawatha coal replaces the Sunnyside seam coal from the Sunnyside Mine in Utah which is no longer available. The liberation and cleaning properties of the Hiawatha coal closely resemble the properties of the Sunnyside coal tested during Phase I.

The coals will be delivered in 100-ton rail cars to a coal yard in north Denver and trucked as needed to Ralston Development Company near Amax R&D for further crushing and temporary storage in covered bunkers. The Taggart coal has been received. A preliminary Denver cell flotation test indicated that the coal can be cleaned to less than 1.0 lb ash per million Btu. The initial laboratory liberation testing also indicated that this lot of Taggart coal may require somewhat finer grinding than the lot received in 1993 for the Phase I testing.

### **Subtask 8.2 PDU Construction**

As scheduled, TIC achieved mechanical completion of the PDU flotation module on July 31, 1995. Electrical, as well as any safety related change order work, was completed during the month of August. Overall, construction of the PDU flotation module is 100% complete. Entech Global personnel completed the procurement of capital equipment as well as all process instrumentation.

**TIC Construction Activities** - TIC, The Industrial Company of Steamboat Springs, Colorado, achieved both mechanical and electrical completion of the PDU flotation module during the quarter. Mechanical completion was accomplished in July while electrical completion was accomplished in August.

**TIC Request for Information (RFI)** - TIC and Amax R&D, Inc. have established a method to expedite and clarify issues regarding the construction of the PDU. When an issue or unclear question arises which requires a response by Amax R&D, TIC issues a written Request for Information (RFI). The RFI states the nature of the question and also any associated cost implications. Amax R&D then responds to the issue accordingly. During the quarter, 60 RFI's were issued by TIC and acted upon by Amax.

**Receipt of County Approval** - During the month of August, 1995, official inspectors from Jefferson County, Colorado, issued overall approval of the structural/construction work as well as electrical work. As a result of this final approval/closure, no further inspections will be necessary.

**Procurement of PDU Capital Equipment** - Entech Global, Inc. personnel completed the procurement of capital equipment for use in the PDU. During the third quarter of 1995, no Request for Quotation (RFQ) packages or Award Packages were issued. One equipment item was delivered during the quarter.

**Procurement of PDU Instrumentation** - Entech Global, Inc. personnel completed procurement of PDU instrumentation. Twenty one items were received during the

quarter. Overall, 51 instrumentation items were received and installed from nine different vendors.

**Installation and Configuration of the Control and Data Acquisition System** - Configuration of the Honeywell Series 9000 controller and SCAN 3000 Data Acquisition Unit was completed during the quarter. The control screens and graphics, which were completed by Entech Global personnel, are operational. The configuration of the Series 9000 controller was completed by Mr. Bob Reynouard of Control Technologies, Inc. (a consultant), with assistance from Honeywell and Entech Global. Mr. Reynouard and Entech Global personnel will perform some general screen "cleanup" tasks during the upcoming quarter as well as complete the installation of a printer.

**Refurbishing of Existing PDU Equipment** - Reconditioning of all existing process equipment items was completed during the quarter. Overall, 12 equipment items have been reconditioned with nine items completed during the quarter.

**Transformer Installation** - Transformer installation efforts commenced July 11, 1995. Public Service of Colorado installed a pole line from McIntyre Street along Amax's northern property line to a point east of the pilot plant. From there, underground service runs from the pole line to the new 1500 kVA transformer located near the eastern access doors of PDU area 200. The new transformer, which was received July 17, was energized and placed into service on July 25. All PDU equipment is now serviced by the new power distribution system.

### **Subtask 8.3 PDU Advanced Coal Cleaning Module Shakedown and Test Plan**

PDU flotation module shakedown and evaluation commenced during the quarter. Any problems, mechanical or electrical, were corrected accordingly by TIC or Entech personnel. The remaining efforts dealt with assembly of the PDU flotation module test plan.

**PDU Flotation Module Shakedown** - Efforts related to the PDU module shakedown and test plan commenced during the quarter. All process equipment items were checked for proper rotation (where applicable) and operated individually. All equipment items, except for the Techweigh scale, passed the shakedown test and are considered ready for operation.

Some additional problems (mostly electrical) were discovered during initial shakedown efforts. As a result, a TIC electrician was retained through the latter part of September to complete the associated tasks.

**Equipment Repairs** - During the initial PDU shakedown, operators noticed an irregular noise generated from the secondary ball mill speed reducer. Inspection of the unit by qualified reducer representatives indicated that the unit is in a failure mode. Continued use of the unit may result in total failure and unwanted downtime of the PDU. As a

result, Reliance/Dodge will supply a new speed reducer for replacement during the upcoming quarter. A local machinist/millwright company will perform all related work.

**Test Plan** - A project review meeting was conducted at the PDU site with DOE, Bechtel, VPI, and Entech Global personnel. A presentation was made by Entech Global personnel regarding the status of PDU construction as well as the proposed test matrix/schedule for the PDU flotation module. A tour of the PDU pilot plant followed the presentation.

## 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 twelfth quarter of the project, July 1 to September 30, 1995. Eleven quarterly reports have been issued previously [1-11].

### 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. The Industrial Company (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 revised 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 determined the cleaning potential of the selected coals and established design parameters and operating guidelines for a 2 t/hr PDU containing both advanced column flotation and selective agglomeration modules. A conceptual engineering design was prepared for a fully integrated and instrumented 2 t/hr PDU incorporating the features determined from the laboratory and bench-scale studies.

Additional activities to be completed during Phase I include:

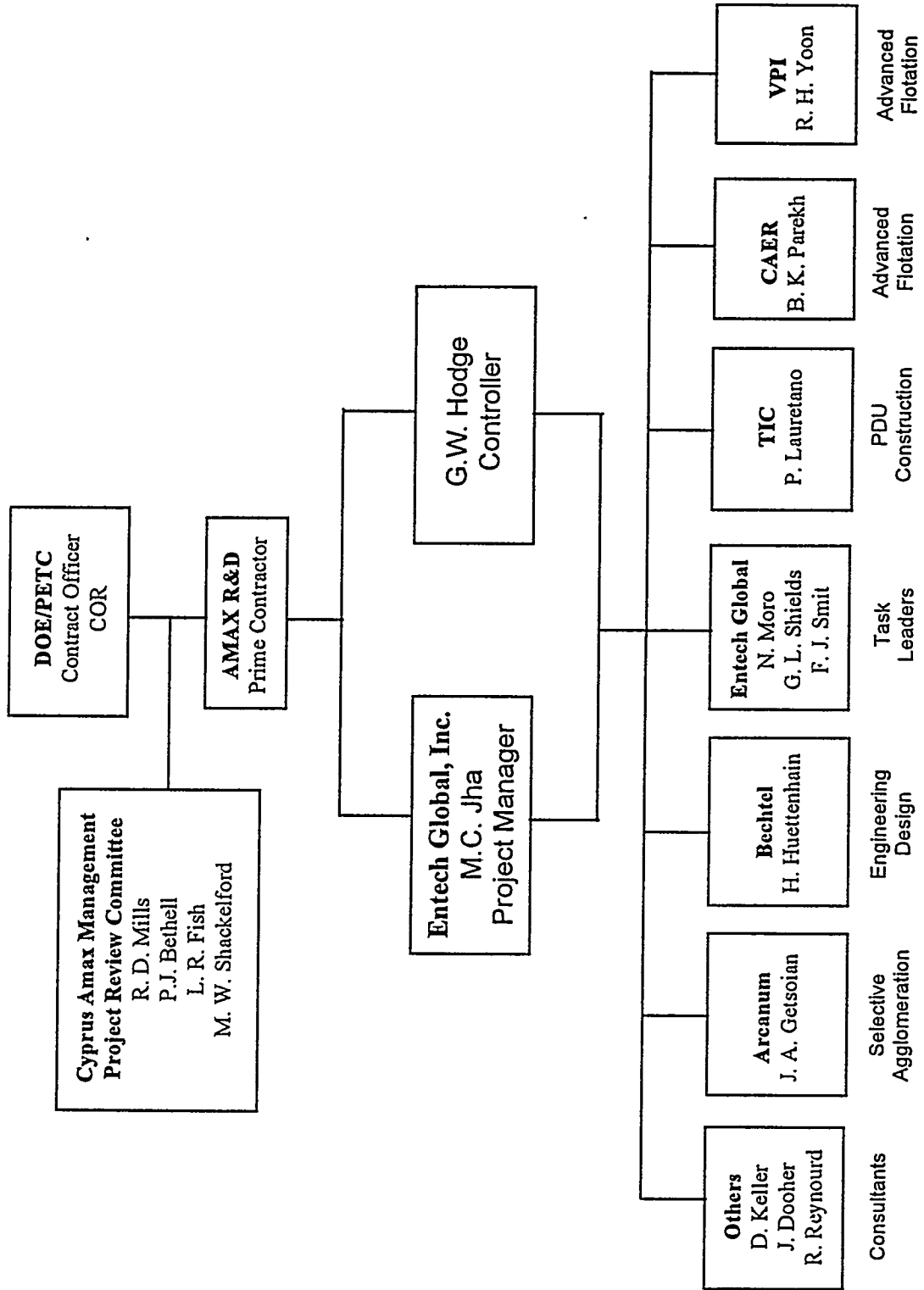
- Production of ultra-clean coal test lots by bench-scale column flotation and selective agglomeration for end-use testing by the DOE or a designated contractor
- Determination of toxic trace element distribution during production of these test lots
- Evaluation of the rheological properties of slurry 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 is for advanced column flotation while Phase III is for 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.



Revised April 25, 1995

Figure 1. Project Management Organization Chart



**Table 1. Outline of Work Breakdown Structure**

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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
<u>Phase II. PDU and Advanced Column Flotation Module Testing and Evaluation</u>	
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
<u>Phase III. Selective Agglomeration Module Testing and Evaluation</u>	
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
<u>Phase IV. PDU Final Disposition</u>	
Task 10.	Disposition of the PDU
Task 11.	Project Final Report

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Revised April 25, 1995

Subtask	1992			1993							1994																
	O	N	D	J	A	M	J	J	A	S	O	N	D	J	A	S	J	A	S	O	N	D					
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27
1.1 Project Work Plan																											
1.2 Project Work Plan Revisions																											
2.1 Coal Selection																											
2.2 Procurement and Storage																											
3.1 NTA Engineering Analyses																											
3.2 NTA Engineering Development																											
3.3 Dewatering Studies																											
4.1 Grinding																											
4.2 Process Optimization Research																											
4.3 CWF Formulation Studies																											
4.4 AF Bench Testing, Scale-up																											
4.5 AF Conceptual Design PDU																											
5.0 Detailed Design PDU, AF Module																											
6.1 Agglomeration Agent Selection																											
6.2 Grinding																											
6.3 Process Optimization Research																											
6.4 CWF Formulation Studies																											
6.5 Sel. Aggl. Bench Testing, Scale-up																											
6.6 Concept. Design Sel. Aggl. Module																											
7.0 Detailed Design Sel. Aggl. Module																											
8.1 Coal Procurement																											
8.2 PDU Construction																											
8.3 Shakedown, Test Plan																											
8.4 Operation and Production																											
8.5 AF Topical Report																											
9.1 Construction SA Module																											
9.2 Shakedown, Test Plan																											
9.3 Operation and Production																											
9.4 Selective Agglomeration Topical Report																											
10.0 PDU Decommissioning																											
11.0 Project Final Report																											

Revised October 16, 1995

Figure 2. Project Schedule

Subtask	1995							1996							1997																
	J	F	M	A	M	J	J	O	N	D	J	F	M	A	M	J	J	F	M	A	M	J	J	F	M	A	M	J			
	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	
1.1 Project Work Plan																															
1.2 Project Work Plan Revisions																															
2.1 Coal Selection																															
2.2 Procurement and Storage																															
3.1 NTA Engineering Analyses																															
3.2 NTA Engineering Development																															
3.3 Dewatering Studies																															
4.1 Grinding																															
4.2 Process Optimization Research																															
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5.0 Detailed Design PDU, AF Module																															
6.1 Agglomeration Agent Selection																															
6.2 Grinding																															
6.3 Process Optimization Research																															
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6.5 Sel. Aggl. Bench Testing, Scale-up																															
6.6 Concept. Design Sel. Aggl. Module																															
7.0 Detailed Design Sel. Aggl. Module																															
8.1 Coal Procurement																															
8.2 PDU Construction																															
8.3 Shakedown, Test Plan																															
8.4 Operation and Production																															
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9.3 Operation and Production																															
9.4 Sel. Agglomeration Topical Report																															
10.0 PDU Decommissioning																															
11.0 Project Final Report																															

Revised October 16, 1995

Figure 2. Project Schedule (Cont'd)

## ACCOMPLISHMENTS DURING QUARTER

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

### TASK 3 DEVELOPMENT OF NEAR-TERM APPLICATIONS

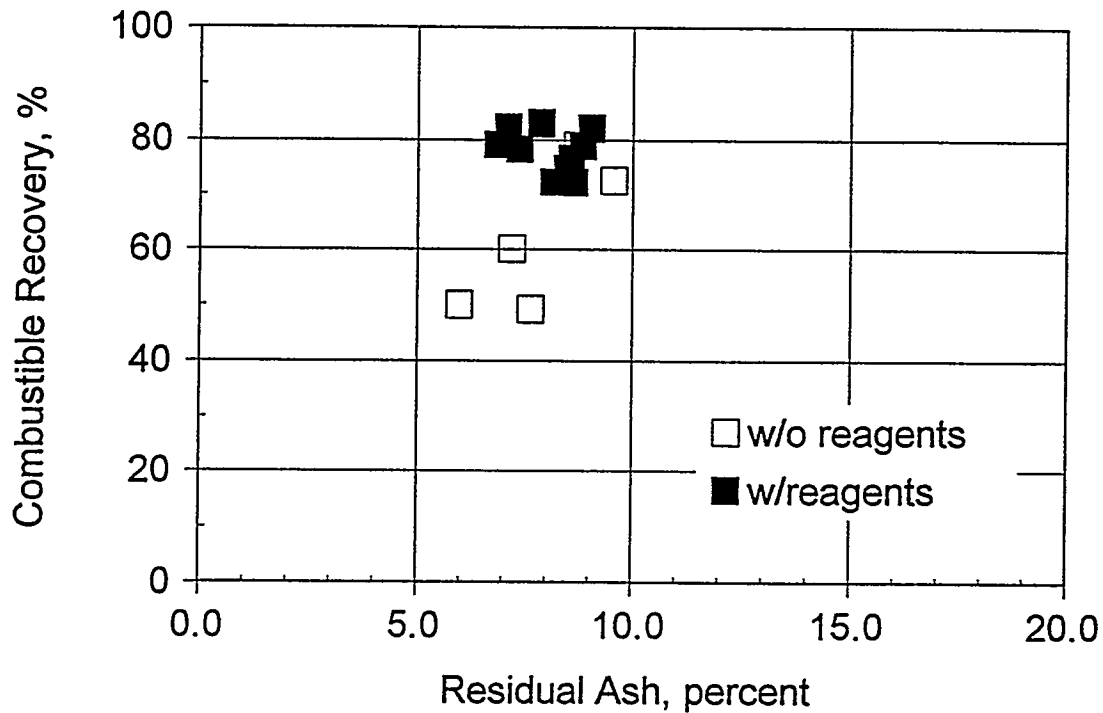
During 1993, Bechtel performed an engineering analysis evaluating potential applications for column flotation and selective agglomeration at three coal preparation plants operated by what is now Cyprus Amax Coal Company [12]. Economic projections favored column flotation over selective agglomeration and an application at the Lady Dunn Preparation Plant (Cannelton Coal Company) 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 in previous quarterly reports [10, 11], the Lady Dunn Preparation Plant in West Virginia is the host site for testing Microcel™ column flotation recovery of the clean coal in the fines screened and cycloned from the raw coal. The clay content is quite high in this stream at the Lady Dunn Plant, and as a result, clean coal recovery with the existing mechanical cells is poor. For this reason the Lady Dunn 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. The main emphasis of the work so far has been to obtain scale-up information for a full plant conversion to column flotation.

An existing 30-inch diameter Microcel™ test unit was refurbished and installed in the plant for testing during the previous quarter. It was first piped to receive minus 100-mesh desliming cyclone overflow. The cyclone overflow contained between 4.4 and 7.8 percent solids, and the solids generally contained 33 to 46 percent ash. Feed rates between 60 and 115 gpm slurry were investigated which produced between 800 and 1600 lb/hr of clean coal. Variations in the frother and diesel fuel dosages were also

studied during the initial testing. A grade-recovery plot for these tests is presented in Figure 3.



**Figure 3. Microcel™ Results - Lady Dunn Cyclone Overflow**

As can be seen in Figure 3, a high-quality product (7 to 9 percent ash) meeting Cannelton requirements was produced at a good recovery of combustible matter (65 to 80 percent).

A goal of Cannelton management, though, is to clean a coarser stream (minus 48-mesh or even minus 28-mesh) by flotation. Encouraging results were obtained during preliminary tests when a 48-mesh by 100-mesh fraction was added to flotation feed slurry. There was not enough of the 48 x 100-mesh fraction available though to really test Microcel™ flotation of the coarser coal particles so the column was being re-piped at quarter end to receive minus 0.75-mm fines screened directly from the raw coal.

Consideration is being given to dewatering the clean coal product from the Microcel™ flotation. It was agreed at a project review meeting in August that centrifuging and a horizontal belt filter will be evaluated for the this application. In this regard, twelve drums of clean coal slurry were collected from the 30-inch Microcel™ system at the end of the quarter and shipped to DOE/PETC. The column was receiving minus 100-mesh cyclone overflow at the time the clean coal froth was being collected. The Coal Preparation Research group at PETC will conduct centrifuge dewatering tests on the slurry using the GranuFlow process. An asphalt emulsion from Venezuela called Orimulsion is added to the centrifuge feed during the GranuFlow process in order to improve the properties of the dewatered product. Baseline tests will be performed

without the additive. Cannelton management is also making arrangements for an on-site test of a 13-sq ft continuous horizontal belt vacuum filter.

### **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 hydrophobic dewatering (HD) process for clean coal fines. In this process a hydrophobic substance is added to a coal-water slurry to displace water from the coal surface. The hydrophobic substance is then recovered for recycle to the process. Three coals will be tested including the product from near-term testing at the Lady Dunn plant (Subtask 3.2). Subtask 3.3 was further divided into five additional subtasks as follows:

- Subtask 3.3.1 - Project Planning
- Subtask 3.3.2 - Identification of Hydrophobic Substances
- Subtask 3.3.3 - Process Development
- Subtask 3.3.4 - Design/Operation of Continuous Bench-scale Unit
- Subtask 3.3.5 - Economic Analysis

Work was carried out on Subtasks 3.3.1, 3.3.2, and 3.3.3 as discussed below.

#### **Subtask 3.3.1 - Project Planning**

During this reporting quarter a detailed work plan was prepared and submitted to DOE. This plan contained a description of the test plan, experimental procedures, analytical methods, and reporting guidelines for implementation and completion of the project.

#### **Subtask 3.3.2 - Identification of Hydrophobic Substances**

To identify suitable hydrophobic substances that can readily displace water from coal, contact angle measurements were conducted during this reporting quarter (3.3.2.1). Thermodynamically, when the contact angle, defined as the angle between the liquid and coal surface as measured through the liquid phase, becomes greater than 90°, the displacement of water by the hydrophobic substance on the coal surface becomes spontaneous. And, as the contact angle further increases, the better the hydrophobic substance performs as a dewatering agent.

While a number of different contact angle measurement methods are available, this project will primarily utilize the "sessile drop method", in which a hydrophobic droplet is placed over a polished coal surface and the angle subtended by the liquid at the three phase contact is measured. To evaluate the dewatering potential of the coals proposed, average water contact angles were determined with the results as follows:

- Indiana VII - 29°
- Elkhorn No. 3 - 62°
- Sunnyside - 54°

Based on the low water contact angle of the Indiana VII coal, due to its low rank, hydrocarbon contact angle determinations were limited to the Sunnyside and Elkhorn No. 3 coals.

Subsequently, contact angle measurements were carried out on Elkhorn No. 3, and Sunnyside coals using pentane, hexane, heptane, octane, and decane paraffinic hydrocarbons. Results indicated that as the hydrocarbon chain length increases, the contact angle decreases. As such, pentane is potentially the best dewatering agent tested. During the next quarter, two hydrocarbons of shorter chain length, butane and propane, will be evaluated in a high pressure contact angle cell.

Under Subtask 3.3.2.2, the three most suitable hydrocarbons will be used to determine the free energy of displacement for one of the coals to be utilized during the process development work.

### **Subtask 3.3.3 - Process Development**

Samples of the Indiana VII and Sunnyside coals were obtained from AMAX R&D during this quarter as part of Subtask 3.3.3.1, Coal Sample Acquisition, Preparation and Characterization.

Under Subtask 3.3.3.2, a batch dewatering unit was designed to test this HD process. The design incorporates the following:

- A high pressure cell to keep the hydrocarbons in a liquid form
- A variable speed mixer to provide adequate mixing
- A transparent window for visual observation
- A pressurized sampling vessel
- Continuous monitoring of temperature, pressure, and mixer speed

Once this unit is constructed by PARR Instruments, it will be tested under Subtask 3.3.3.3. Recovery and regeneration of the hydrophobic substance will be tested separately under Subtask 3.3.3.4.

## **TASK 4 ENGINEERING DEVELOPMENT OF ADVANCED FROTH FLOTATION**

As described in the Subtask 2.1 report, Coal Selection Plan and Recommendations [13] 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**

<u>Coal</u>	<u>Mine</u>	<u>State</u>	<u>HGI</u>	<u>Ash, %</u>	<u>Sulfur, %</u>
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 under Subtask 4.4, Bench-scale Testing and Process Scale-up. Results are discussed below.

#### **Subtask 4.4 Bench-Scale Testing and Process Scale-Up**

Work continued on the Subtask 4.4 Topical Report during this reporting quarter, with the report nearing completion at quarter end. In addition, the analysis of samples from selected bench-scale tests continued. This analytical work focused on sulfur and toxic trace elements analyses.

#### **Toxic Trace Elements**

The evaluation of reduction in toxic trace element concentrations accomplished by bench-scale column flotation was initiated last quarter by assaying the products from selected parametric tests. From these analyses, the distribution of the trace elements between the clean coal product and the waste refuse were determined.

For the most part, the discrepancies noted last quarter for antimony, lead, and mercury analyses were resolved by repeat analyses. In particular, it was found that a more rigorous sample dissolution and dilution procedure improved the consistency of the antimony results. A revised listing of the ranges for reported elemental concentrations and the mass balance closures are shown in Table 3. The revised average of all mass balance closures was 98 percent, with the mass balance closures generally within 20 percent. Good mass balance closures were difficult to achieve for mercury since the amount present in most samples was very close to the detection limit (0.01 ppm or 10 ppb). Mass balances were not calculated for cadmium since it was detected only in the Elkhorn No. 3 and Indiana VII raw coal and the Indiana VII and Sunnyside fine refuse.



**Table 3. Trace Element Concentration Results**

	Trace Element Concentration, ppm												
	Raw Coal		Flotation Feed		Clean Coal		Fine Refuse		Calculated Flotation Feed		Mass Balance Closure, percent		
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Average	
Antimony (Sb)	0.06	1.1	0.14	2.1	0.14	2.7	0.22	1.0	0.15	2.3	104	122	111
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)	3	15	2	8	2	6	6	13	2.0	7.8	85*	117*	99*
Manganese (Mn)	27	200	7	33	4	14	14	360	7.3	35	77	107	95
Mercury (Hg)	0.03	0.06	<0.01	0.04	<0.01	0.02	0.03	0.08	<0.01	0.03	71*	84*	79*
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
													<b>Grand Average: 98</b>

"<" = Below indicated detection limit.

"n. d." = not determined

\* For four of six tests

The reductions in the various trace element concentrations accomplished during the column flotation tests were calculated on a heating value basis using the revised analyses. The newly calculated ranges of the trace-element reductions from the raw coal and from the washed coal flotation feed are shown in Table 4. As reported last quarter, the concentrations of arsenic, beryllium, chromium, cobalt, lead, manganese, mercury, and selenium in the raw coals 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 the column flotation step very definitely further reduced the concentrations of arsenic, chromium, manganese, mercury, and nickel remaining in the washed coal from these seams.

It appears that the antimony concentration, and in certain cases, the nickel, selenium and chlorine concentrations as well, increased on a heating value basis. Such results could indicate a preferential association of these particular elements with a highly floatable component of the carbonaceous portion of the coal.

**Table 4. Reduction in Toxic Trace Element Concentration, HHV Basis**

	From Raw Coal, %			From Flotation Feed, %		
	Minimum	Maximum	Average	Minimum	Maximum	Average
Antimony (Sb)	-103	-46	-64	-79	3	-23
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)	54	76	67	4	31	21
Manganese (Mn)	75	98	92	43	77	62
Mercury (Hg)	40	75	68	36	53	41
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**

The detailed engineering design package for the PDU and the advanced flotation module was submitted to DOE during August [14]. This package represents the final engineering design utilized for construction of the PDU. This engineering package was divided into three volumes, and further subdivided, as follows:

- Volume I - Design Package
  - PFD's
  - P&ID's
  - Equipment, Motor, and Instrument List

- Civil and Structural Design Drawings
- Electrical Design Drawings
- Volume II - Construction Specifications
  - Scope of Work
  - Instrument and Controls Basic Materials and Methods
  - Electrical Basic Materials and methods
  - Structural Steel and Miscellaneous Steel
  - Cast-in-Place Concrete Reinforcement and Grouting
  - Piping, Valves, and Fittings
  - Field Pipe Installation
  - Hydrotesting
- Volume III - Material Requisitions
  - Individual sections for various groups of equipment

With the issuing of this design package, the Task 5 work is complete.

## **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**

The final version of the Subtask 6.3 Topical Report [15] was submitted to DOE and project team partners on September 28, 1995. This 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 product ash specification of 2 lb/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.

The amount of bridging liquid required for agglomeration ranged from 18 to 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- $\mu$ m high volatile A Winifrede bituminous coal slurries all agglomerated well without an activator.

Comparison testing with diesel fuel and kerosene (non-recovery type agglomerating agents) indicated that neither quality (1 to 2 lb ash/MBtu at 80% energy recovery) nor cost (\$2.50/MBtu) goals could possibly be met.

#### **Subtask 6.4 Coal-Water-Fuel Formulation Studies**

The primary objective of Subtask 6.4 is to evaluate the formulation of coal-water-fuel (CWF) slurries from selective agglomeration products. The slurry feedstock, i.e., selective agglomeration products, used for this work are generated during Subtask 6.5 (selective agglomeration bench-scale testing and process scale-up).

While much of this test work will evaluate the effect of various parameters on slurry quality, there are two other objectives for the Subtask 6.4 work. First, this test work will provide a comparison between similar slurries formulated from flotation and agglomeration products. This information will provide some insight into whether one process generates a product inherently more amenable to highly-loaded slurry formulation than the other process. Second, the Subtask 6.4 work will attempt to determine slurry quality guidelines for commercial production. To this end, determinations of required slurry coal loadings, stabilities, and viscosities will be carried out.

The Subtask 6.4 testing began in earnest during this twelfth quarterly reporting period. The initial work involved particle size distributions (PSD) characterization of Subtask 6.5 products. This PSD characterization work was carried out on the final process product, i.e., product from the steam stripping circuit used to remove heptane from the recovered agglomerates. PSD characterization was completed for the following coals:

- Taggart 1 (62-mesh topsize) - The Taggart coal utilized for all of the Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 start-up testing.
- Taggart 2 (62-mesh topsize) - The replacement Taggart coal which will be utilized for the remainder of project testing. While this coal comes from the same seam as the Taggart 1 coal, it is from a different mine.
- Sunnyside (150-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and the initial Subtask 6.5 testing. This coal has since been replaced with the Hiawatha coal.
- Elkhorn No. 3 (100-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.
- Indiana VII (325-mesh topsize) - This coal was used during Subtask 4.3 and 4.4 testing, and is also being used for Subtask 6.5 testing.

Typical agglomeration circuit product PSD's for these coals are shown in Table 5. It should be noted, however, that based on Subtask 6.5 testing results, the Taggart 2 coal will require a finer (100-mesh topsize) grind to achieve the 1 lb/MBtu or less target

product ash specification set for this coal. As such, no further slurry formulation work will be carried out with this 62-mesh topsize Taggart 2 coal agglomeration product.

**Table 5. Selective Agglomeration Product Particle Size Distributions**

<u>Microns</u>	<u>Taggart 1</u>	<u>Taggart 2</u>	<u>Sunnyside</u>	<u>Elkhorn No. 3</u>	<u>Indiana VII</u>
	<u>62 mesh</u>	<u>62 mesh</u>	<u>150 mesh</u>	<u>100 mesh</u>	<u>325 mesh</u>
	<u>Cumulative Percent Passing</u>				
600	100.00	100.00	100.00	100.00	100.00
300	99.93	100.00	100.00	100.00	100.00
212	99.57	99.55	100.00	100.00	100.00
150	96.58	96.24	100.00	100.00	100.00
106	88.62	87.66	100.00	96.85	99.83
75	75.76	76.52	97.30	85.91	99.16
53	63.39	66.14	90.14	75.55	97.73
45	57.06	61.02	82.34	64.95	95.28
38	54.15	57.93	78.48	60.64	94.10
30	46.24	51.86	70.37	51.62	87.21
20	33.18	39.51	48.93	35.55	74.22
15	26.62	31.96	39.86	28.56	60.74
10	18.41	23.70	28.64	20.09	46.32
8	15.29	20.00	24.91	16.31	38.53
6	12.40	15.94	19.78	12.45	30.90
4	8.50	10.90	13.63	8.17	21.62
3	6.02	8.17	9.81	5.68	15.96
2	3.55	4.99	6.04	3.27	9.92
1	<u>0.83</u>	<u>1.21</u>	<u>1.38</u>	<u>0.67</u>	<u>2.75</u>
<b>MMD*</b>	49.70	47.25	24.86	37.92	15.34

\* Mass Mean Diameter = Sum of size interval midpoint (microns) times weight fraction of particles in that interval (percent)

Utilizing Subtask 6.5 products with the PSD's shown in Table 5, testing was carried out to characterize the formulation of slurries from "as-received" agglomeration circuit products. Testing was also carried out to provide a comparison between the formulation of slurries from advanced flotation products (Subtask 4.3) and from selective agglomeration products. This was accomplished by repeating the formulation of various slurries carried out during Subtask 4.3 testing.

It should be noted that the 62-mesh Taggart 1 agglomeration product will be used only to provide a comparison of slurry formulation characteristics with the Subtask 4.3 results. The new 100-mesh topsize Taggart 2 agglomeration product will then be used for the bulk of the Subtask 6.4 test work. Similarly, the Sunnyside agglomeration product will also be utilized only to provide a comparison with previous results, since it has been replaced with the Hiawatha coal (to be used for the bulk of the Subtask 6.4 test work).

The results of this work for the coals tested to date (Taggart 1, Sunnyside, and Indiana VII) are presented below.

### **Taggart 1 Coal CWF Formulation Results**

Slurries were formulated using three different types of 62-mesh topsize Taggart 1 coal selective agglomeration product feedstock as follows:

- As-received agglomeration product utilizing 0.5% of A-23 dispersant. Coal loadings for these slurries were in the range of approximately 61 to 67% by weight. These results provide a baseline with which to gauge the quality of other slurries formulated from this coal, and also provide a comparison with slurries formulated during Subtask 4.3 testing.
- A blend of 70% as-received material and 30% material reground for 30 minutes in the attritor mill utilizing 0.5% of A-23 dispersant. This blend is referred to as the 70/30 regrind blend, and is utilized to generate more fines to improve particle packing characteristics within the slurry. Coal loadings for these slurries were in the range of 55 to 68% by weight. Two slurry formulations were also repeated using Flocon for stabilization. These slurries provide a comparison with similar slurries formulated during Subtask 4.3 testing.
- A blend of 42.9% +325M material, 27.1% -325M material, and 27.1% -325M material reground for 30 minutes in the attritor mill utilizing 0.5% of A-23 dispersant. This blend is referred to as the 325 mesh regrind blend, and was originally formulated during Subtask 4.3 testing to generate a more bimodal PSD. Coal loadings for these slurries were in the range of approximately 60 to 70% by weight. Two slurry formulations were also repeated using Flocon for stabilization. These slurries were also formulated to provide a comparison with similar slurries formulated during Subtask 4.3 testing.

The results for all of these slurries are presented in Figure 4, which shows slurry coal loading vs slurry viscosity.

As can be seen from the data in Figure 4, slurries formulated from the 70/30 regrind blend achieved lower viscosities at similar coal loadings than the slurries formulated from the 100% as-received agglomeration product. This confirms the trend observed in the Subtask 4.3. Similarly, slurries formulated from the 325-mesh regrind blend provided additional viscosity reduction at similar coal loadings. This additional reduction in slurry viscosity also confirms Subtask 4.3 results, and is due to increased particle packing within the slurry as a result of PSD manipulations of this type.

Also, as expected, the use of Flocon (to improve slurry stability as discussed below) results in increased slurry viscosity at the same slurry coal loadings for both the 70/30 blend and the 325-mesh regrind formulations.

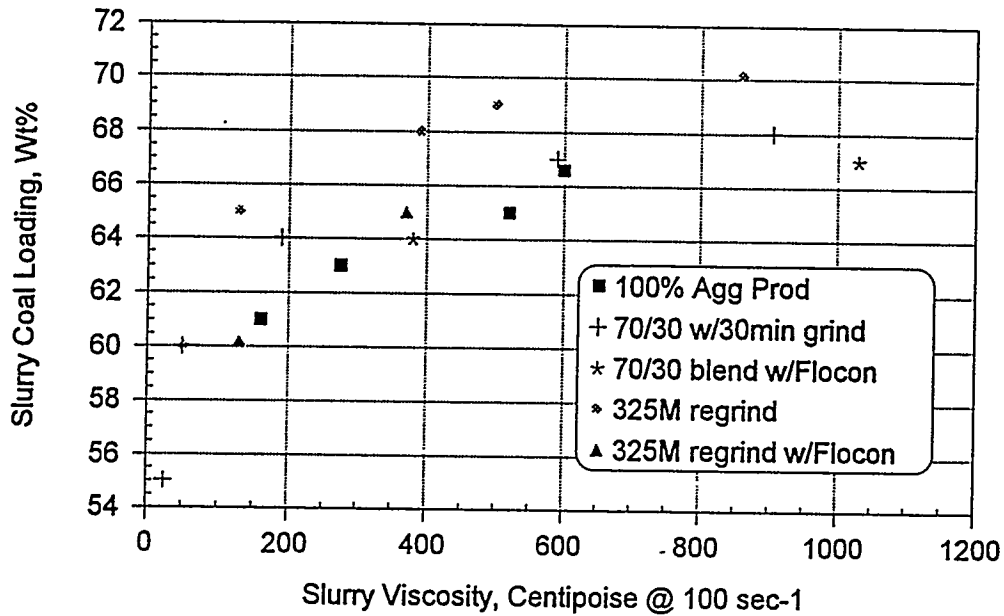


Figure 4. Taggart 1 - Agglomeration Product Slurry Results

Figure 5 provides a comparison of similar slurries formulated with both selective agglomeration and flotation products.

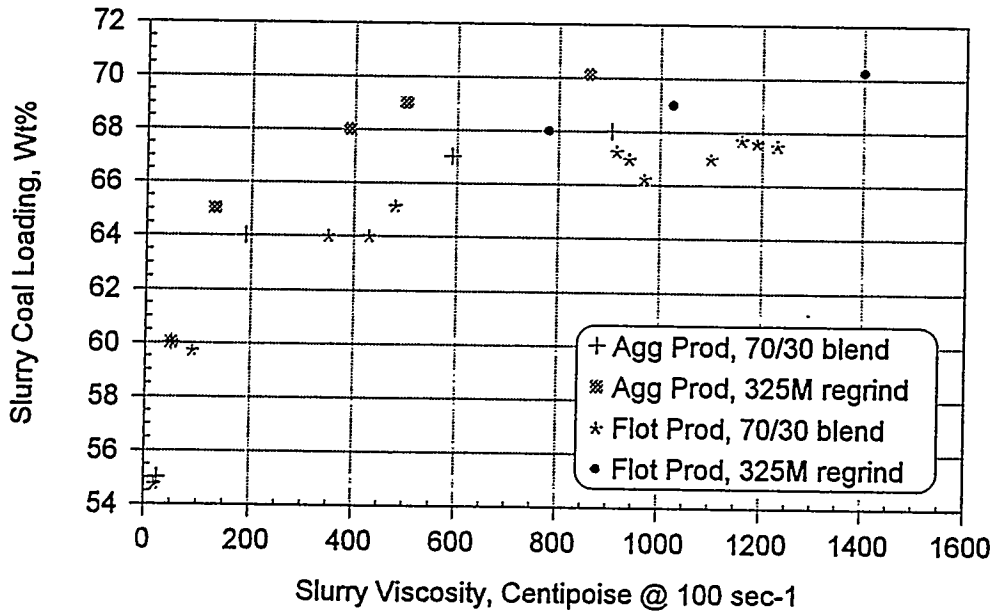


Figure 5. Taggart 1 - Agglomeration & Flotation Product Slurry Results

While the trends observed as a result of PSD manipulation may be similar, it appears that lower viscosities, at similar coal loadings, are achieved with the selective agglomeration circuit product than with the column flotation product. It is believed that this may be due to a number reasons including:

- The presence of aggregates of particles in the steam stripped agglomeration circuit product, effectively coarsening the PSD.
- An effect of the steam stripping process on the surface properties of the coal.
- The presence of residual heptane in the agglomeration circuit product.

All of the slurries formulated with this Taggart 1 coal, except those that utilized Flocon, had poor stabilities with a rating of "1" after overnight storage. While this rating of "1" indicates that at least 25% of the slurry volume was occupied by a very hard pack sediment, for all of these slurries, 60-90% was very hard packed. When Flocon was used, however, stabilities were in the "3" to "4" range after overnight storage, indicating that only about 5-10% of the slurry formed a very hard packed sediment.

### **Sunnyside Coal CWF Formulation Results**

Slurries were formulated using two different types of 150-mesh topsize Sunnyside coal agglomeration product feedstock as follows:

- As-received agglomeration product utilizing 1.0% of A-23 dispersant. Coal loadings for these slurries were in the range of approximately 56 to 62% by weight. Two slurry formulations were also repeated using Flocon. These results provide a baseline with which to gauge the quality of other slurries formulated from this coal, and also provide a comparison with slurries formulated during Subtask 4.3 testing.
- A blend of 70% as-received material and 30% material reground for 30 minutes in the attritor mill utilizing 0.5% of A-23 dispersant. As for the Taggart 1 coal, this blend is referred to as the 70/30 regrind blend. Coal loadings for these slurries were in the range of 59 to 65% by weight. Two of these slurry formulations were also repeated using Flocon for stabilization. These slurries provide a comparison with similar slurries formulated during Subtask 4.3 testing.

The results for all of these slurries are shown in Figure 6, which shows slurry coal loading vs slurry viscosity. As expected based on previous experience (Subtask 4.3), slurries formulated from the 70/30 regrind blend achieved lower viscosities at similar coal loadings than the slurries formulated from the 100% as-received agglomeration product. This trend of decreasing slurry viscosity at similar coal loadings is expected since PSD manipulations of this type increase particle packing within the slurry. Also, the use of Flocon to improve slurry stability resulted in increased slurry viscosity over slurries formulated at the same coal loadings that did not use Flocon.

Figure 7 provides a comparison of slurries formulated with selective agglomeration and flotation products for the 150-mesh topsize Sunnyside coal agglomeration product. This data indicates that the quality of slurries formulated from flotation products (both



as received and the 70/30 regrind blend) were similar to that achieved with the 100% as-received agglomeration product. However, slurries formulated from the 70/30 regrind blend of agglomeration product achieved lower viscosities, at similar coal loadings, than the slurries formulated from both flotation product feedstocks.

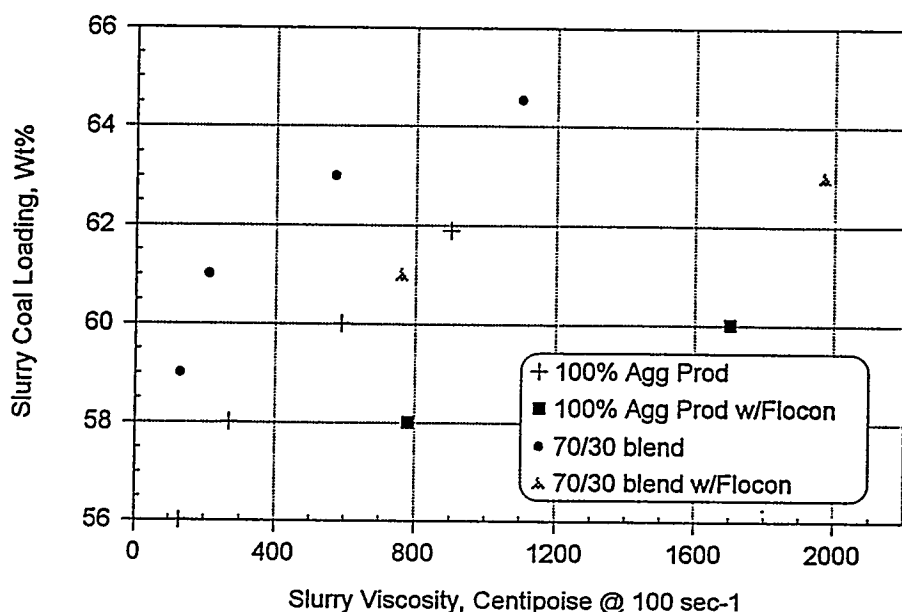


Figure 6. Sunnyside - Agglomeration Product Slurry Results

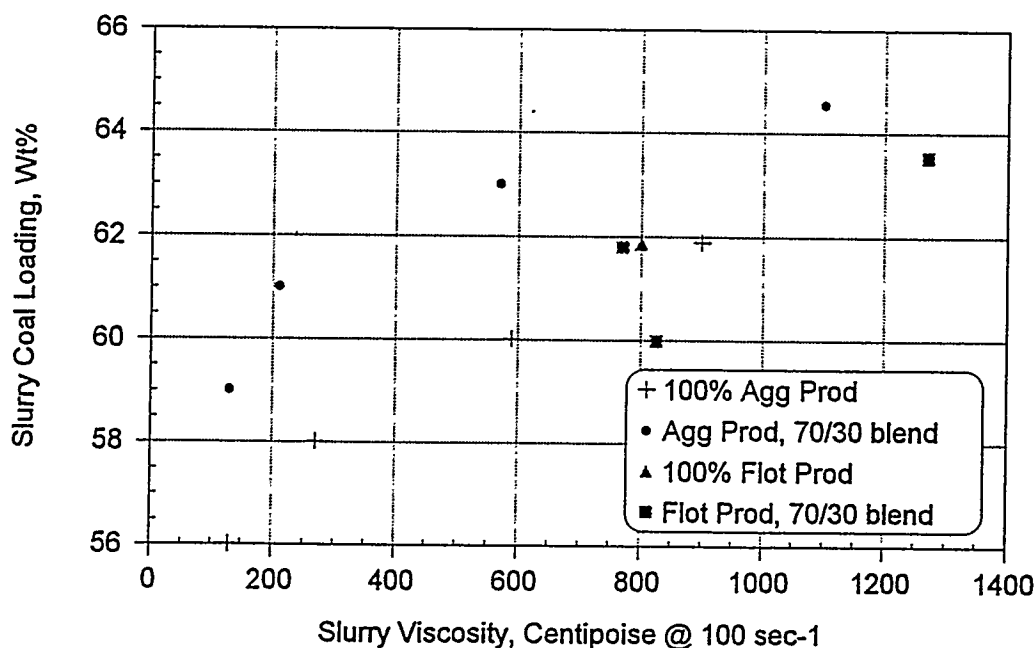


Figure 7. Sunnyside - Agglomeration and Flotation Product Slurry Results

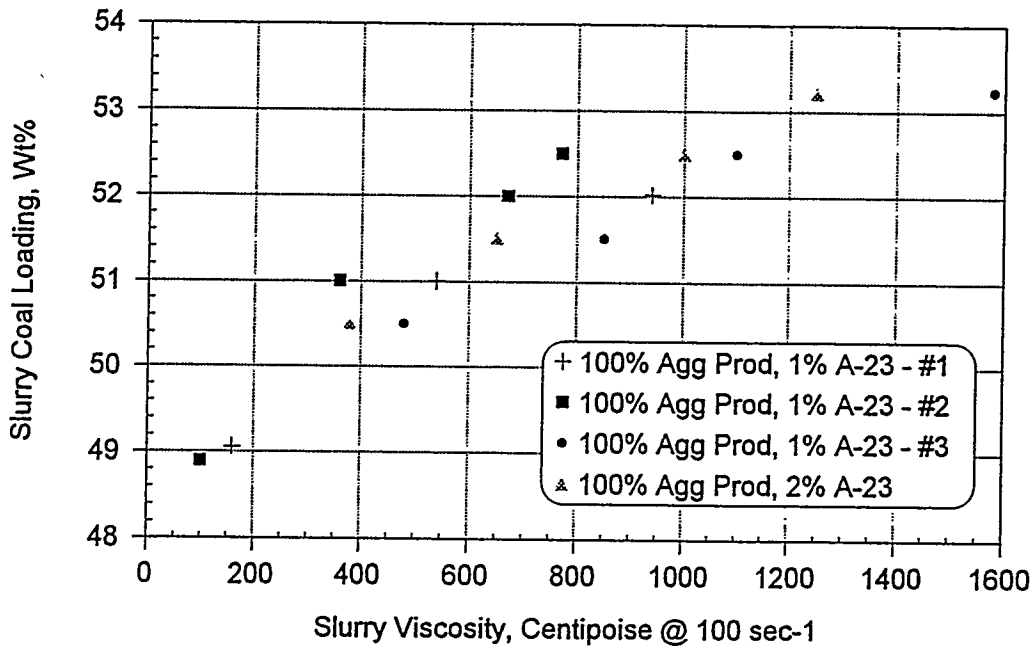
As with the Taggart 1 coal slurries, all of the slurries formulated with the Sunnyside coal, except those that utilized Flocon, had poor stabilities with a rating of "1" after

overnight storage. When Flocon was used, however, stabilities were rated in the "3" to "4" range after overnight storage.

### Indiana VII Coal CWF Formulation Results

Indiana VII coal slurries were formulated from as-received agglomeration product utilizing 1.0% and 2.0% A-23 dispersant levels. Three different feedstocks were utilized for this work, all of which had similar PSD's. Coal loadings for these slurries were in the range of approximately 49 to 53% by weight. These results provide a baseline with which to gauge the quality of other slurries formulated from this coal and also provide a comparison with slurries formulated during Subtask 4.3 testing.

The results for all of these slurries are presented in Figure 8, which shows slurry coal loading vs slurry viscosity.

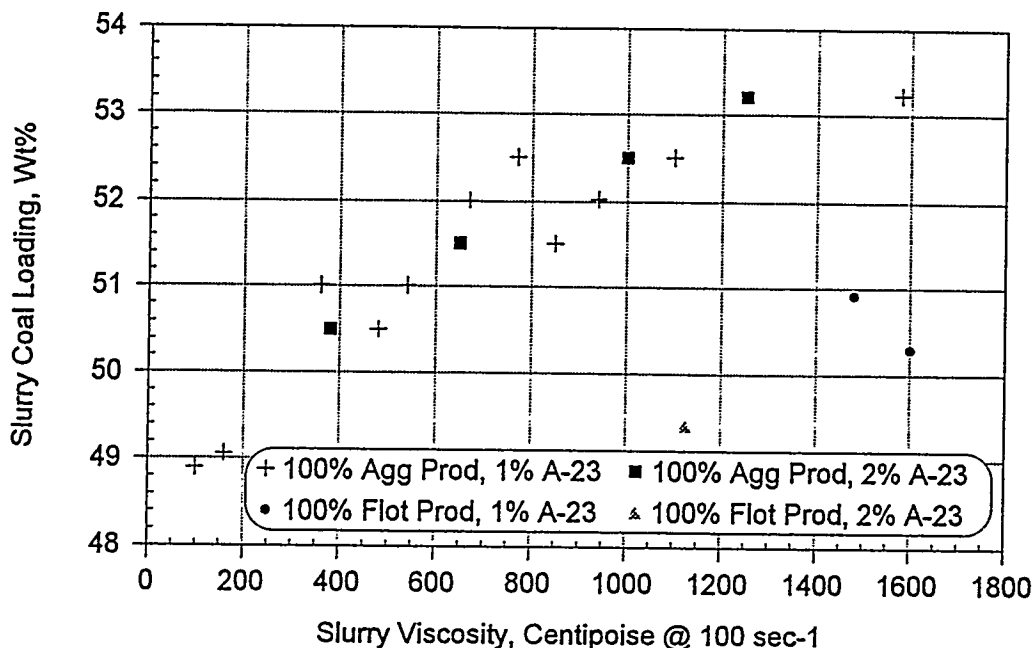


**Figure 8. Indiana VII - Agglomeration Product Slurry Results**

As can be seen from this data, all slurries made with Indiana VII coal followed the same coal loading vs slurry viscosity trend, regardless of the quantity of A-23 dispersant utilized. It should be noted that no regrind blends were formulated with this coal due to the fineness (325-mesh topsize) of this agglomeration product. Future testing may investigate the formulation of coarsened slurry feedstock material, if appropriate.

Figure 9 provides a comparison of slurries formulated with selective agglomeration and flotation products for the 325-mesh topsize Indiana VII coal. This data indicates that the quality of slurries formulated from flotation product (both with 1% and 2% A-23) had higher slurry viscosities at similar coal loadings than those slurries formulated from the

agglomeration product. This confirms the general trend of improved slurry characteristics for agglomeration products observed for the Taggart 1 and Sunnyside coals.



**Figure 9. Indiana VII - Agglomeration and Flotation Product Slurry Results**

As with the two previous coals, all of the slurries formulated with the Indiana VII coal had poor stabilities with a rating of "1" after overnight storage. No Flocon was utilized in the formulation of these Indiana VII coal slurries.

**Subtask 6.5 Bench-Scale Testing and Process Scale-up**

The Subtask 6.5 test work progressed significantly during this reporting quarter. During the previous reporting quarter, construction and shakedown testing of the 25 lb/hr bench-scale unit was completed. In addition, evaluation of the Taggart 1 and Sunnyside coals was carried out. It should be noted, that these two coals have since been replaced with the Taggart 2 and Hiawatha coals, respectively, and will not be tested further in the bench-scale agglomeration unit. In addition, testing during the last quarter evaluated the agglomeration characteristics of the Indiana VII coal. While this work indicated that product ash specifications could be met at the selected 325-mesh topsize grind, additional testing with the Indiana VII coal will be carried out later in this test program.

A number of test conditions were evaluated in the 25 lb/hr selective agglomeration test unit during this quarter. This work was carried out utilizing the Elkhorn No. 3 coal ground to a 100-mesh topsize and the Taggart 2 coal ground to a 62-mesh topsize.

A de-aromatized commercial grade heptane was used as the bridging liquid for the bulk of the continuous agglomeration testing carried out this quarter. In addition, a pure grade of heptane was utilized for some of the test work.

Evaluation of steam stripping recovery of heptane from agglomerated products continued during this quarter. In an effort to confirm the 2 t/hr PDU steam stripping circuit design, the bench-scale unit was modified to allow testing of a two stage stripping circuit.

**Elkhorn No. 3 Coal Testing**

Continuous agglomeration testing utilizing the Elkhorn No. 3 coal was carried out during this reporting quarter. For this work, the Elkhorn coal was closed-circuit ground to a 100-mesh topsize in the 4' x 4' ball mill. This grind provided a coal particle size distribution (PSD) similar to that utilized during the Subtask 4.3 advanced flotation test work. This 100-mesh topsize Elkhorn No. 3 feed coal PSD, along with the agglomeration circuit product PSD is shown in Table 6.

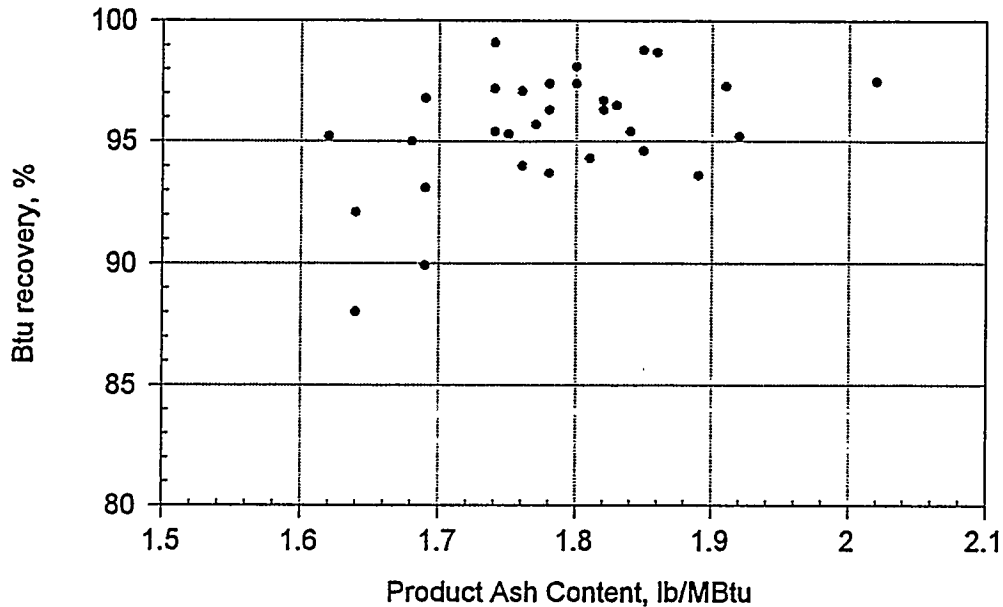
**Table 6. Elkhorn No. 3 - Feed and Product PSD's**

<u>Microns</u>	<u>Feed</u> <u>Cumulative Percent</u>	<u>Product</u> <u>Percent Passing</u>
150	100.00	100.00
106	96.86	96.85
75	84.34	85.91
53	70.64	75.55
45	63.73	64.95
38	59.58	60.64
30	50.63	51.62
20	35.84	35.55
15	27.47	28.56
10	18.80	20.09
8	15.78	16.31
6	12.11	12.45
4	8.07	8.17
3	5.73	5.68
2	3.21	3.27
1	0.44	0.67
<b>MMD*</b>	39.43	37.92

\* Mass Mean Diameter = Sum of size interval midpoint (microns) times weight fraction of particles in that interval (percent)

A total of 32 agglomeration circuit test runs were completed this reporting period to evaluate the performance of the continuous agglomeration unit for the 100-mesh

topsize grind Elkhorn No. 3 coal. Operating conditions and results for all of these test runs are shown in Appendix A. A plot of Btu recovery vs product ash content in lb/MBtu, for all of this data, is shown in Figure 10.



**Figure 10. Elkhorn Agglomeration - Product Ash vs Btu Recovery**

The following conditions were held constant for all of these tests:

- 100-mesh topsize grind
- 2.4-inch (4-blade) high-shear impeller
- 4.8-inch (4-blade) low-shear impeller
- Commercial grade heptane except for Test E1A32 which used pure heptane
- Screen spray water flow rate

The following variables were changed during this testing:

- High-shear impeller tip speed
- High-shear residence time
- Low-shear impeller tip speed
- Low-shear residence time
- Heptane concentration
- Solids concentration
- Screen inclination
- Heptane type (pure or commercial)

**Froth Skimming Effects** - It should be noted that 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 data in Appendix A, the utilization of froth skimming resulted in yield and Btu recovery increases in the ranges of approximately 0.2-4.0% and 0.4-5.0%, respectively. It should be noted that those tests with lower Btu recoveries generally benefited more from the froth skimming operation. This trend is expected since lost energy is generally in the form of unagglomerated coal, which would be expected to float during froth skimming.

**Liberation Requirements** - As can be seen from the data in Appendix A, and Figure 10, the 2 lb ash/MBtu product specification was met for all but one of the tests completed. This indicates that the 100-mesh topsize grind provides sufficient mineral-matter liberation. As such, no other grinds were evaluated for the Elkhorn No. 3 coal.

**Energy Recovery** - Agglomeration yields for the Elkhorn coal ranged from approximately 85 to 96%. On a run-of mine (ROM) basis, yields were in the 44-50% range due to the original raw coal feed ash of approximately 45%, which resulted in a raw coal precleaning yield of only 51.6%. Agglomeration Btu recoveries achieved were in the range of 88 to 99%, equivalent to approximately 83 to 94% on a ROM basis. As such, sufficient energy recovery was achieved for all of the tests completed to meet the project goals of 80% energy recovery on a ROM basis.

While these tests achieved the project product ash and Btu recovery specifications, it proved difficult to achieve consistently high Btu recoveries, which is generally easy for the selective agglomeration process. This is indicated by the tailings ash values for this Elkhorn No. 3 coal test work which ranged from 24.8% to a high of 73.2% (mostly in the 35 to 55% range).

These relatively low tailings ash values are attributed to aging of the Elkhorn coal which had been stored for over two years prior to its use for this testing. To overcome this effect, asphalt was utilized to enhance agglomeration in three of the tests, E1A29 through E1A31. With the use of asphalt, Btu recoveries of 98.7 to 99.1% were achieved, with associated tailings ash values ranging from 66.9 to 73.2%.

When asphalt was not used to enhance agglomeration, the highest Btu recoveries achieved were in the 96 to 98% range. These higher levels of Btu recovery were only achieved, however, when a combination of various operating conditions were utilized as discussed below.

**Energy Input** - Primary to achieving high Btu recoveries was the total energy input to the combined high- and low-shear unit operations, with higher energy inputs achieving higher Btu recoveries. As such, when higher impeller tip speeds and longer agglomeration residence times were utilized, higher Btu recoveries were generally

observed. As mentioned above, this need for high energy inputs is attributed to the aging of the coal, i.e., oxidation of the coal surface. As such, the energy input capability of the continuous agglomeration system was pushed to its limit for processing this aged Elkhorn No. 3, coal.

**Solids Concentration Effect** - It was found that tests utilizing higher solids concentrations (13%), E1A27 and E1A28, achieved higher energy recoveries than similar tests carried out at 10 and 7% solids concentration. This confirms previous results indicating that agglomeration carried out at a higher solids concentration requires less energy than at a lower solids concentration. This effect is due to the occurrence of more particle to particle contact, given the same energy input, at the higher solids loadings.

**Heptane Concentration Effect** - The other important factor in achieving high Btu recoveries was the utilization of sufficient heptane. As can be seen from the data in Appendix A, the first 16 agglomeration tests used heptane dosages in the range of 24 to 29% on a dry ash free coal basis. Out of these tests, only one test (E1A8, with the highest overall energy input) achieved a Btu recovery greater than 97%. In contrast, for the remainder of the Elkhorn tests which utilized heptane dosages of primarily 30 to 35% daf, most achieved Btu recoveries greater than 97%. In those latter tests that didn't achieve higher Btu recoveries, lower impeller tip speeds, particularly during low shear, had been used.

**Agglomerate recovery** - In many of the tests, the agglomerates screened as a bed, rather than individual flowing agglomerates. As a result, the drainage of mineral-matter bearing process water was dependent primarily on the size of the agglomerates, with better formed agglomerates achieving lower product ash contents due to better drainage. This confirms the trend observed during previous testing with other coals

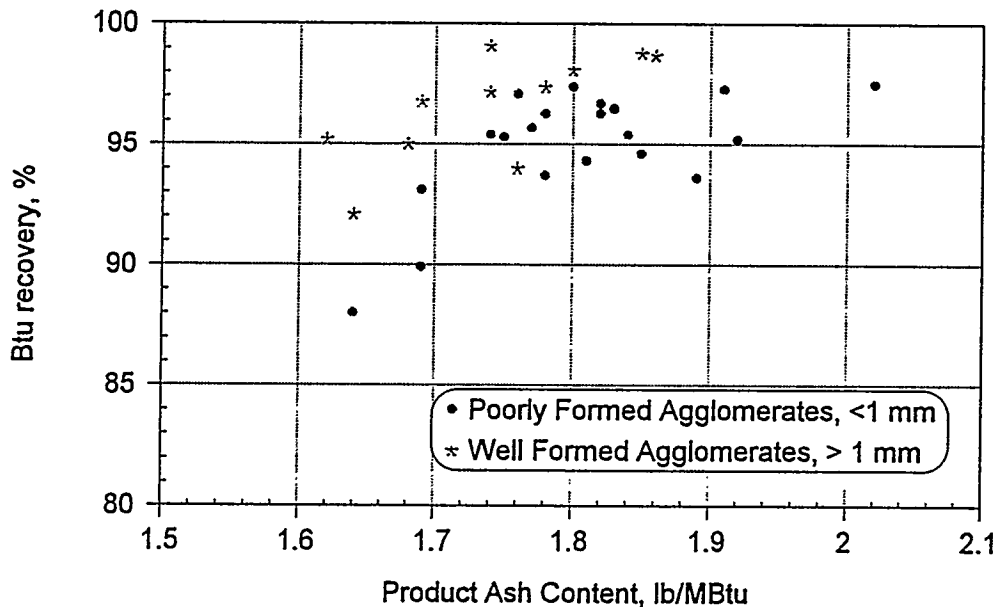
In some cases a very poorly formed agglomerated product was discharged from the low shear reactor. This product was defined as "slop", as indicated by the identifying "s" in the Product Size column of Appendix A. For these tests, manual assistance was often required to maintain the flow of agglomerates across the screen. This was the case even when the screen inclination was increased to improve agglomerate flow. This phenomena was found to be more prominent at lower agglomeration energy input levels, i.e., lower high- and low-shear impeller tip speeds. This trend is expected since the higher shear rates generally form better agglomerates, as confirmed by the energy recovery/power input trend discussed above.

As experienced previously with the testing of other coals, when well formed agglomerates were produced, the agglomerates screened well without manual assistance. It should also be noted that the production of well formed agglomerates resulted in higher screen product solids concentrations, as expected due to better drainage of associated water.

**Product ash content** - As discussed above, the size of the agglomerates formed generally determines the product ash content, with larger agglomerates achieving lower product ash values, due primarily to better drainage of mineral-matter bearing process water. No other parameters evaluated had a noticeable effect on product ash content, except indirectly where they ultimately affect the agglomerate size and strength.

**Summary** - Based on these results, it is apparent that both product ash content and Btu recovery specifications can be met with the 100-mesh topsize grind for the Elkhorn coal. The best results for agglomeration of this coal were achieved when sufficient bridging liquid and energy were utilized to insure the production of well formed agglomerates from low shear. When this was the case, the agglomerates screened well, resulting in a product ash values of 2.5 to 2.7%, equivalent to approximately 1.7 to 1.9 lb ash/MBtu.

This effect of agglomeration performance as a function of agglomerate formation is illustrated in Figure 11, which plots all the data as a function of agglomerate size.



**Figure 11. Elkhorn Coal, Btu Recovery vs Ash Content - Agglomerate Size**

As such, with the completion of the test work reported here, continuous agglomeration testing of the Elkhorn No. 3 coal is considered complete.

**Taggart 2 Coal Testing**

Preliminary work was carried out during this reporting quarter utilizing the replacement Taggart coal (Taggart 2). Based on previous work with this coal, a closed circuit 62-mesh topsize grind was completed in the 4' x 4' ball mill. Five continuous agglomeration tests were completed with this coal. Results for these tests are presented in Appendix B.



As can be seen from these continuous agglomeration results, it appears that while the 62-mesh topsize grind achieved the 2 lb ash/MBtu product specification, it did not provide sufficient mineral-matter liberation to achieve the desired product ash specification of 1 lb ash/MBtu. As such, additional test work with this coal will be completed utilizing a 100-mesh topsize grind.

The following conditions were held constant for all of these tests:

- 62-mesh topsize grind
- 2.4-inch (4-blade) high-shear impeller
- 4.8-inch (4-blade) low-shear impeller

As for all previous testing, product and tailings ash values, as well as performance calculations, utilized composited samples from froth skimming simulation.

The primary variables changed during the completion of these five agglomeration tests were:

- Coal feed rate/residence time
- High-shear impeller tip speed
- Low-shear residence time
- Heptane concentration

As with the Taggart 1 coal, this Taggart 2 coal proved very easy to agglomerate as indicated by Test T2A1 in which a high-shear impeller tip speed of 6.4 m/s proved sufficient to achieve acceptable inversion, as compared to other coals requiring impeller tip speeds in the range of 15-18 m/s.

It should also be noted that, as determined during start-up testing with the Taggart 1 coal, many difficulties were encountered at the low-shear discharge point. This lack of flow from the low-shear vessel is attributed to the extreme buoyancy of the agglomerated Taggart coal product. While changes to the low-shear discharge arrangement were made during start-up testing, with some improvement realized, it seems apparent that additional modifications may be necessary to allow the completion of the Taggart coal testing.

This low-shear discharge plugging was a problem for all Taggart 2 tests completed except for test T2A3 in which a 50 lb/hr coal feed rate was used. For this test, the higher volumetric flowrate was sufficient to prevent plugging of the low-shear discharge. One other point to note from this data, is that the lowest product ash content and the highest product solids concentration were achieved in Test T2A4, in which the largest agglomerated product (up to 4 mm) was achieved. This confirms a general trend observed through the bulk of all testing to date.

Taggart 2 coal testing in the continuous 25 lb/hr agglomeration unit will continue during the next reporting quarter utilizing a 100-mesh topsize grind.

### **Winifrede Coal Testing**

Work began on grinding the Winifrede coal for evaluation in the continuous bench-scale agglomeration unit. As of the end of this reporting quarter, the Winifrede coal had been ground in the 4' x 4' ball mill followed by a single pass through the Drais Stirred Ball Mill.

This ground Winifrede coal had a mass mean diameter of approximately 13 microns, with about 80% passing 20 microns. This ground product was evaluated in batch agglomeration testing to determine additional liberation requirements. Results indicated that the 2 lb ash/MBtu product specification could not be met. As such, additional passes through the Drais Mill will be carried out until sufficient liberation is achieved.

### **Continuous Steam Stripper Testing**

During the previous quarter, batch stripping tests were carried out to evaluate the effect of elevated temperatures on steam stripping recovery of heptane from agglomerated products. To achieve higher than ambient pressure boiling 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, as reported in the previous quarterly report are shown in Table 7.

As can be seen from the data in Table 7, treatment in the autoclave at increased temperature and pressure resulted in reduced heptane concentrations as compared to the continuous stripper product, generated at ambient pressure, i.e., a boiling point of 94°C. As such, a two-stage continuous stripping circuit was constructed to test these results.

**Two-Stage Steam Stripper Design** - Work was completed during this reporting period on the design and construction of a modified continuous steam stripping circuit. This circuit utilizes two stages of stripping as is planned for use in the 2 t/hr PDU selective agglomeration module. The design criteria for this unit was to allow the processing of 25 lb/hr of coal through a two-stage stripping circuit at 20-25% solids concentration. This unit also utilizes a counter-current steam flow in which the second stage stripper vapor product is utilized as the steam feed to the first stage stripper.

This modified stripping circuit is fed with a peristaltic pump from a newly constructed feed tank. This feed tank has a capacity of approximately 15 gallons, and is fitted with a 4 Hp air mixer to insure good mixing, and as such, consistent feed to the stripping

circuit. The newly constructed, agitated, first-stage stripping vessel is 8-inches in diameter and 24-inches tall. With a normal operating level of about 6 to 8-inches, this unit provides approximately 5-minutes residence time at a feed rate of 0.8 to 1.0 liter per minute. This first-stage stripping is carried out at ambient pressure, and therefore operates in the temperature range of about 92-94°C. The excess head space, approximately 18-inches, was provided to reduce the amount of coal carryover to the vapor condenser and the gravity separator.

**Table 7. Batch Stripper Results - Pressure Effects**

<u>Solvent</u>	<u>Coal</u>	<u>Autoclave**</u>	<u>n-heptane, ppm</u>	
		<u>Ret Time (min)</u>	<u>sample</u>	<u>dcB</u>
Commercial Heptane*	Indiana VII	0**	208	1949
	Indiana VII	10	82	804
	Indiana VII	20	74	604
	Indiana VII	30	29	334
	Sunnyside	0***	155	2583
	Sunnyside	10	61	893
	Sunnyside	20	45	575
	Sunnyside	30	55	589
Pure Heptane	Sunnyside	0****	887	8153
	Sunnyside	10	242	2995
	Sunnyside	20	301	3053
	Sunnyside	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

The feed to this first-stage stripper enters the vessel on the side of the tank, about 2-inches from the bottom. The product is withdrawn from the bottom of the vessel. During this stage of stripping, the bulk of the heptane is removed, and a more handleable stream produced. The product from this first-stage stripper is pumped to the second-stage stripper by a variable speed Moyno pump. The speed of this pump is manually controlled to maintain a constant level in the first-stage stripper, visible in a sight tube.

The stripped first-stage product is then processed through a second stage of steam stripping. This second stage stripping vessel is the previously utilized stripping column (4-inches in diameter), lengthened to increase its residence time to approximately 10 minutes. This unit was also modified to allow its operation at a positive pressure of 15-20 psig. As such, the operating temperature in this second stage stripper can be maintained in the 115-120°C range.

As done previously, the feed enters this second-stage stripper in the upper portion of the column, and is discharged from the bottom via a U-tube arrangement. The

operating level of this second-stage stripper is maintained (to provide 10-minutes residence time) at a relatively constant level via manual operation of the product discharge valve. The desired operating pressure and temperature are maintained in this vessel by manual operation of the vapor discharge control valve. As before, this vessel was partially filled with 5/8-inch pall rings to insure steam distribution across the column's entire cross-sectional area.

**Two-Stage Steam Stripper Operation** - Three different tests were completed using this modified two-stage stripping circuit during this reporting period. All three of these tests utilized Elkhorn No. 3 coal agglomerated product from the continuous agglomeration circuit. For the first test (E1S2), Elkhorn coal agglomerated with commercial heptane was utilized as the feedstock. It should be noted that this feedstock had been agglomerated several days prior to being fed to the steam stripping circuit. For the other two continuous tests, the agglomeration circuit screen product was fed directly to the stripping circuit feed tank. In this manner, the entire agglomeration/stripping unit was run together. Test E1S3 utilized commercial heptane and test E1S4 utilized pure heptane.

Results for all three of these two-stage stripping tests are shown in Table 8. It should be noted that the primary heptane analyses are from Huffman Laboratories, while the heptane analyses in parenthesis are from Analytica (formerly Phoenix Labs).

**Table 8. Two-Stage Stripper Testing Results**

Test ID	Hept Type	Prod Coal lb/hr	Stripper 1				Stripper 2			
			Temp °C	RT min	Prod % sol	n-hept ppm, dcb	Temp °C	RT min	Prod % sol	n-hept ppm, dcb
E1S2	Comm	37.5	93	5.5	20.6	1650(823)**	117	10	22.6	1175(598)
E1S3	Comm	30.0	92	4.5	18.9	505(868)	115	8	20.1	384(350)
E1S4*	Pure	29.0	92	5.0	17.5	1250(3066)	117	9	19.0	1031(1128)

\*\* Results in parenthesis from Analytica

Based on these results, it appears that two stages of steam stripping as tested here, achieved lower trace heptane concentrations than a single stage of steam stripping. This is believed to be due primarily to the increased temperature in the second stage. Additional testing will be carried out with other project coals to confirm the results presented here.

**Tailings Heptane Analyses**

Samples of agglomeration tailings (froth skimmer underflow) were sent for trace heptane analyses. These samples originated from an Elkhorn coal agglomeration test in which approximately 50% tailings ash was achieved, utilizing commercial grade heptane. Samples submitted included as received tailings, tailings filter cake, tailings

filtrate, and tailings samples that had been boiled for 5, 10, and 20 minutes. Less than 10 ppm of n-heptane was detected in all of the tailings samples, except for the filter cake, which contained 380 ppm n-heptane, at 67% solids, or 567 ppm n-heptane on a dry solids basis. There was less than 1 ppm of n-heptane detected in the tailings filtrate.

## **TASK 7 PDU SELECTIVE AGGLOMERATION - MODULE DETAILED DESIGN**

Several 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 and the construction bid package can be completed in a timely manner.

### **Design Overview**

The following sections of this report discuss design features of the unit operations. A block flow diagram of the PDU selective agglomeration module is shown in Figure 12.

### **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 switch over from the flotation to agglomeration process module. As such, the agglomeration process will use slurry ground by the installed equipment.

The ground product will then be sent to what is currently the Microcel™ flotation column feed tank. The product stream from this tank is monitored by a density meter, whose determination is utilized to dilute the out flowing slurry to a target solids concentration. The product from this feed tank will feed one of two ground slurry storage tanks. From these storage tanks, the slurry will be 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).

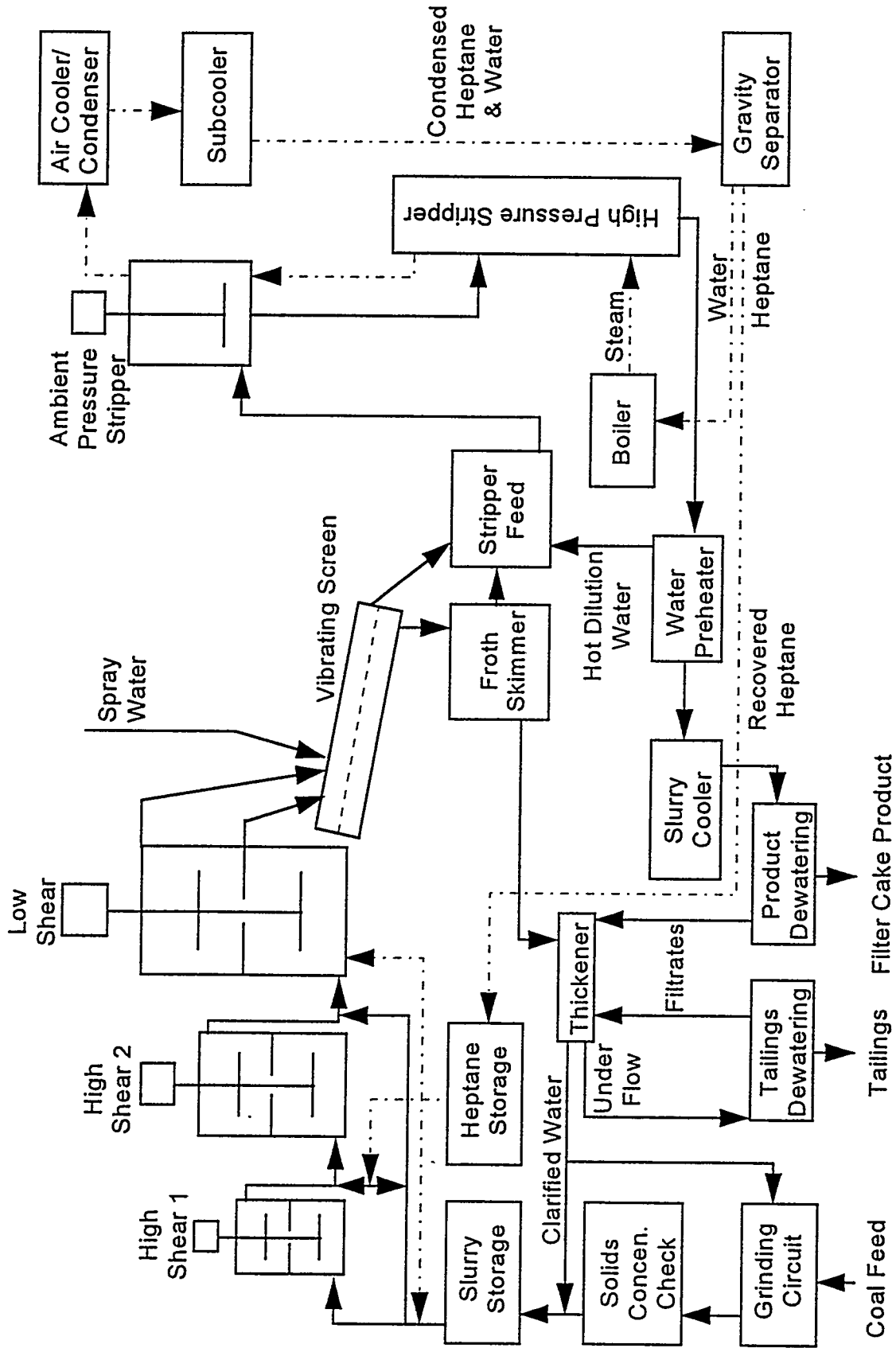


Figure 12. PDU Selective Agglomeration Module Block Diagram

### **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 a single radial flow impeller in each section. 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 agglomeration process as required, currently anticipated not to exceed 40% of the coal feed rate. The ability will be provided to add bridging liquid to either high shear vessel and the low shear vessel. 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.

The ability to add asphalt to the high-shear circuit will also be provided for evaluation of 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 6-bladed impeller for each section of the vessel.

The impellers will be of the radial-flow type and the drive unit of sufficient Hp to achieve tip speeds in the 3-5 m/s range. Under some operating conditions, primarily to control excessive agglomerate growth, it is desirable to achieve low-shear impeller tip speeds of greater than 5 m/s. To accomplish this without the incorporation of a significantly larger drive unit, the low-shear impellers will have four hinged blades, so that when run in the opposite rotation direction, much less power will be drawn.

### **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 3-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.

### **Design Status**

As of the end of this reporting quarter, the design has been virtually finalized. By the end of October, it is anticipated that the P&ID's will be finalized. Also within this same time frame, material data sheets should be completed for all major pieces of equipment and all of the required instrumentation.

It is currently anticipated that the construction bid package will be completed by the end of November. As such, purchasing will begin during November with a construction start date scheduled for mid to late January, 1996.



## **TASK 8 PDU AND ADVANCED COLUMN FLOTATION MODULE**

The Task 8 work completed this reporting quarter focused on Subtask 8.1 Coal Selection and Procurement, Subtask 8.2 PDU and Advanced Flotation module Construction, and Subtask 8.3 PDU Module Shakedown and Test Plan, as discussed in the following sections of this report.

### **Subtask 8.1 Coal Selection and Procurement**

The Subtask 8.1 PDU Coal Selection Recommendations Topical Report [16] was issued to DOE and project partners on August 17, 1995. The recommended coals and source mines were as follows:

- Taggart Coal
  - Steer Branch Mine
  - Red River Mining Company
  - Wise County, Virginia
- Indiana VII Coal
  - Minnehaha Mine
  - Cyprus Amax Coal Company
  - Sullivan County, Indiana
- Hiawatha Coal
  - Crandall Creek Mine
  - Genwal Mining Company
  - Emery County, Utah

The Hiawatha coal replaces Sunnyside seam coal from the Sunnyside Mine in Utah which is no longer available. As described in the Topical Report [16], the liberation and cleaning properties of the Hiawatha coal closely resemble the properties of the Sunnyside coal tested during Phase I. Properties of the three coals recommended for use in the PDU are summarized below:

- Taggart Coal:
  - 2" x 0 washed coal
  - 5 - 7 % moisture
  - 2.8% dry ash
  - 0.72% max sulfur (<0.51 lb/MBtu)
  - >14,000 Btu/lb as received
  - >34% Volatile Matter
  - HGI 52
- Indiana VII Coal:
  - 2" x 0 washed coal

- 15% moisture
  - 9% dry ash
  - 0.7% max sulfur (<0.52 lb/MBtu)
  - 13,400 Btu/lb as received
  - 35% Volatile Matter
  - HGI 55
- Hiawatha Coal:
    - 2" x 0 crushed coal
    - 5% moisture
    - 9% dry ash
    - 0.5% sulfur (0.4 lb/MBtu)
    - 13,300 Btu/lb as received
    - 45% Volatile Matter
    - HGI 50

The coals will be delivered in 100-ton rail cars to a coal yard in north Denver and trucked as needed to Ralston Development Company near Amax R&D for further crushing and temporary storage in covered bunkers. The Taggart coal has been received. A preliminary Denver cell flotation test indicated that the coal can be cleaned to less than 1.0 lb ash per million Btu. The initial laboratory liberation testing also indicated that this lot of Taggart coal may require somewhat finer grinding than the lot received in 1993 for the Phase I testing.

### **Subtask 8.2 PDU Construction**

As scheduled, TIC achieved mechanical completion of the PDU flotation module on July 31, 1995. Electrical, as well as any safety related change order work, was completed during the month of August. Overall, construction of the PDU flotation module is 100% complete.

Entech Global personnel completed the procurement of capital equipment as well as all process instrumentation.

### **TIC Construction Activities**

During the quarter, TIC - The Industrial Company, achieved mechanical and electrical completion of the PDU flotation module. The overall project completion (by milestone) is shown in Table 9.

**Table 9. Construction Progress by Milestone**

<u>Milestone No.</u>	<u>Description</u>	<u>% Complete</u>
1	Mobilization, Excavation, Concrete and Foundation Work	100%
2	Structural Steel Erection and Platework	100%
3	Equipment Installation	100%
4	Piping Installation	100%
5	Electrical and Instrumentation Installation	100%
6	Sheeting and Painting	100%
7	Testing, Startup, and Demobilization	100%
Total		100%

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

- Installed dump hopper (100-D-01)
- Installed elevating conveyor (100-T-03)
- Installed conduit in PDU Area 100
- Installed control wire to PDU Area 100
- Completed siding of Area 200 penthouse
- Completed installation of process piping
- Installed belting on elevating conveyor (100-T-03)
- Completed installation of all process instrumentation
- Installed vibratory feeder (100-T-02)
- Installed ventilation fans in Area 100
- Installed vibrating bin activator (100-T-05)
- Installed electrical switchgear
- Installed size 6 starter for Netzsch fine grinding mill
- Installed CDAS panel in control room
- Relocated WesTech drum filter to accommodate discharge chute
- Installed discharge chute on WesTech drum filter
- Terminated control wiring in CDAS panel
- Installed weigh belt feeder (100-T-04)
- Installed spray water lines in Microcel™
- Installed anionic lines to thickener
- Installed cationic lines to thickener
- Installed 2 inch air line to Microcel™
- Installed Area 200 steps
- Installed Microcel launder sprays

- Installed frother pump (200-G-04)
- Installed frother line to Microcel™
- Installed collector pump (200-G-05)
- Installed collector line to Microcel™
- Checked rotation of all motors
- Checked v-belt alignment of all motors
- Filled clarified water tank with water
- Checked operation of primary ball mill
- Checked operation of secondary ball mill. Loud noise generated from speed reducer. May be pinion gear or seal bearing. Machine shop to investigate.
- Pressure tested all process pipe
- Installed calibration tubes on all chemical metering pumps
- Installed 1 inch air control valve on Microcel™
- Installed Sisetec screen sprays
- Installed new impeller on WesTech drum filter
- Installed samplers where designated
- Repaired PDU paging system
- Connected power to MCC #4
- Wired Techweigh scale (#100-T-04)
- Wired size 6 starter for fine grinding mill
- Troubleshooting of Sisetec screen motor & motor block
- Replaced motor on Sisetec screen
- Installed / wired Microcel™ pressure / level indicators
- Installed / wired Microcel™ level control valve
- Installed / wired Microcel™ feed flow meter
- Installed / wired Microcel™ recirculation flow meter
- Installed Area 200 access catwalk
- Labeled process piping
- Painted dump hopper 100-D-01
- Installed safety ballards around perimeter of new transformer
- Removed 100-D-02 handrail for ball / drum access
- Installed ball mill fill pipe
- Installed 70 mesh screen cloths on Sisetec screens
- Painted Area 200 walkway
- Started demobilization

### TIC Requests For Information

TIC and Amax R&D, Inc. have established a method to expedite and clarify issues regarding the construction of the Process Development Unit. When an issue or unclear question arises which requires a response by Amax R&D, TIC issues a written Request for Information (RFI). The RFI states the nature of the question and also any associated cost implications. Amax R&D then responds to the issue accordingly. Table 10 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 10. TIC Request For Information Items**

<u>RFI #</u>	<u>Date</u>	<u>Subject</u>	<u>Reply</u>	<u>Signed</u>
217-057	7/8/95	Bin Activator Support	7/10/95	M. Jha
217-058	7/8/95	Feed Bin Access Ladder	7/10/95	G. Shields
217-059	7/8/95	Rise Box in Drain Pipe	7/10/95	G. Shields
217-060	7/8/95	Relocation of Stairs	7/10/95	M. Jha
217-061	7/8/95	Area 200 Access Platform	7/10/95	G. Shields
217-062	7/8/95	Microcel Platform Access	7/10/95	G. Shields
217-063	7/8/95	Area 200 Handrail	7/10/95	G. Shields
217-064	7/8/95	Relocation of 3 VFD Power Conduits	7/10/95	G. Shields
217-065	7/8/95	Elimination of Common Pipe Header	7/10/95	M. Jha
217-066	7/10/95	Bad Motor on Sisetec Screen	7/18/95	G. Shields
217-067	7/16/95	Delivery of Remaining Electrical Equipment	7/18/95	B. Reynouard
217-068	7/17/95	Mechanical Completion	7/18/95	G. Shields
217-069	7/18/95	Air & Electric to Samplers	7/24/95	G. Shields
217-070	7/18/95	3 inch Line from 400-G-10 to 400-D-04	7/18/95	M. Jha
217-071	7/18/95	Additional Cationic Mixer	7/18/95	G. Shields
217-072	7/18/95	Purchase of 4 Rotameters for Sisetec Screen Sprays	7/18/95	G. Shields
217-073	7/24/95	Drum Filter Chute Work	7/25/95	G. Shields
217-074	7/18/95	Installation of 4 Microcel Spray Lines	7/18/95	M. Jha
217-075	7/18/95	Air Line Change from 1 inch to 2 inch - Credit	7/18/95	G. Shields
217-076	7/18/95	Air Line Change from 1 inch to 2 inch - Credit	7/18/95	M. Jha
217-077	7/18/95	7/28/95 Electrical Shutdown	7/18/95	G. Shields
217-078	7/24/95	Relocation of 100-T-06	7/25/95	G. Shields
217-079	7/25/95	Cooling Water on 400-G-04 and 05	7/25/95	G. Shields
217-080	7/25/95	Netzsch Filter Air Blow Valves	7/25/95	M. Jha
217-081	7/25/95	Wrong Wire Specification for 100-T-03	7/25/95	G. Shields
217-082	7/25/95	Switchgear DCS Signal	7/25/95	G. Shields
217-083	7/25/95	100-Y-03 Additional Wiring	7/25/95	G. Shields
217-084	7/25/95	Cationic Tubing to Microcel Tailings Sump	7/25/95	G. Shields
217-085	7/26/95	Drum Filter Seal Water Piping	7/26/95	G. Shields
217-086	7/26/95	Drum Filter Muffler Piping	7/26/95	G. Shields
217-087	7/26/95	Netzsch Fine Grinding Mill Cooling Water	7/26/95	G. Shields
217-089	7/26/95	Weigh Scale Wire	7/26/95	G. Shields
217-090	7/26/95	Installation of Transmitter IT-121	7/26/95	G. Shields
217-091	8/7/95	Additional Instrument Air not on P&ID's	8/7/95	M. Jha
217-092	8/10/95	Netzsch Filter Added Piping	8/25/95	G. Shields
217-093	8/10/95	City Water to Microcel	8/10/95	G. Shields
217-094	8/10/95	Added 400-D-03 Piping	8/25/95	G. Shields
217-095	8/15/95	400-D-05 Bypass	8/25/95	G. Shields

**Table 10 (Cont'd). TIC Request For Information Items**

<u>RFI #</u>	<u>Date</u>	<u>Subject</u>	<u>Reply</u>	<u>Signed</u>
217-096	8/16/95	400-D-08 Bypass	8/25/95	G. Shields
217-097	8/16/95	400-Y-01 & 02 Additional Valves	8/25/95	G. Shields
217-098	8/16/95	Installation of 80 Replacement Microcel Flange Bolts	8/25/95	G. Shields
217-101	8/28/95	Additional Mixer Installation - Cationic	8/25/95	G. Shields
217-102	8/28/95	Air Piping - Labor	8/28/95	G. Shields
217-103	8/28/95	Relocation of Beam Under 100-T-06 - Labor	8/28/95	M. Jha
217-104	8/28/95	Sizetec Motor Troubleshooting - Labor	8/28/95	G. Shields
217-105	8/28/95	100-D-02 Handrail - Labor	8/28/95	M. Jha
217-106	8/28/95	Handrail at Elevation 128' - 0" - Labor	8/28/95	G. Shields
217-107	8/28/95	100' - 0" Catwalk Installation - Labor	8/28/95	M. Jha
217-108	8/28/95	Drum Filter Chute Installation - Labor	8/28/95	M. Jha
217-109	8/28/95	200-Y-02 Kickplate	8/28/95	G. Shields
217-110	8/28/95	Microcel Flange Bolt Installation - Labor	8/28/95	G. Shields
217-111	8/28/95	Handrail and Guard on Thickener Mechanism - Labor	8/28/95	M. Jha
217-112	8/28/95	Install Netzsch Blowout Sump - Labor	8/28/95	G. Shields
217-113	8/28/95	Ball Mill Loading Pipe - Labor	8/28/95	G. Shields
217-114	8/28/95	Installation of AMAX Lightning Deterrent System	8/29/95	G. Shields
217-115	8/28/95	Wrong Wire Specification for 100-T-03	8/29/95	M. Jha
217-116	9/8/95	Calibration / Troubleshoot Instruments & Valves	9/8/95	G. Shields
217-117	9/8/95	Troubleshoot Microcel Level & Flowmeters	9/8/95	G. Shields
217-118	9/15/95	Electrical Troubleshooting 9/19 - 9/20	9/15/95	G. Shields
217-119	9/15/95	Electrical Troubleshooting 9/21 - 9/22	9/15/95	G. Shields

**Receipt of County Approval**

During the month of August, 1995, official inspectors from Jefferson County, Colorado, issued overall approval of the structural/construction work as well as electrical work. As a result of this final approval/closure, no further inspections will be necessary.

**Procurement of PDU Capital Equipment**

Entech Global, Inc. personnel completed the procurement of capital equipment for use in the PDU. During the third quarter of 1995, no Request for Quotation (RFQ) packages or Award Packages were issued. One equipment item was delivered during the quarter. The status of each capital item, along with those items awarded and received during the quarter (hi-lighted in boxes) is listed in Table 11.

**Table 11. Status of Capital Equipment Procurement**

<u>Equip. Number</u>	<u>Description</u>	<u>RFQ Date</u>	<u>RFQ Deadline</u>	<u>Award Date</u>	<u>Company</u>	<u>Delivered Price</u>	<u>Delivery Date</u>
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

**Table 11 (Cont'd). Status of Capital Equipment Procurement**

<u>Equip. Number</u>	<u>Description</u>	<u>RFQ Date</u>	<u>RFQ Deadline</u>	<u>Award Date</u>	<u>Company</u>	<u>Delivered Price</u>	<u>Delivery Date</u>
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 (Kreb's 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-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	7/21/95
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

**Procurement of PDU Instrumentation**

Entech Global, Inc. personnel completed procurement of PDU instrumentation. Twenty one items were received during the quarter. The status of each item, along with those items received during this reporting quarter (hi-lighted in boxes) is listed in Table 12.

**Table 12. Status of Instrumentation**

<u>TAG #</u>	<u>Description</u>	<u>Company</u>	<u>Price</u>	<u>Delivery</u>
DIT-203	Densitometer	Winn Marion	\$ 5,065	7/7/95
FIT-104	Flow Transmitter	PCI Sales	\$ 1,845	6/29/95
FIT-105	Flow Transmitter	FAMCO	\$ 963	7/3/95
FIT-114	Flow Transmitter	FAMCO	\$ 1,142	7/3/95
FIT-116	Flow Transmitter	FAMCO	\$ 963	7/3/95
FIT-117	Flow Transmitter	FAMCO	\$ 1,396	7/3/95
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	7/6/95
FI-207	Flow Gauge	PCI Sales	\$ 1,570	6/29/95
FIT-209	Flow Transmitter	FAMCO	\$ 1,278	7/3/95
FIT-211	Flow Transmitter	JMC Instruments	\$ 2,647	6/26/95
FIT-215	Flow Transmitter	Power Controls	\$ 630	7/6/95
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	7/3/95
PT-210	Press Transmitter	JMC Instruments	\$ 608	7/3/95
PI-214	Pressure Gauge	JMC Instruments	\$ 106	7/3/95
PIT-403	Press Transmitter	JMC Instruments	\$ 806	7/3/95
PI-414	Pressure Gauge	JMC Instruments	\$ 106	7/3/95
PI-423	Pressure Gauge	JMC Instruments	\$ 106	7/3/95
TI-110	Temp Gauge	JMC Instruments	\$ 96	6/26/95
PCV-102	Valve - 2"	Winn Marion	\$ 1,074	7/11/95
FV-104	Valve - 2"	Rampart	\$ 122	6/7/95
LV-114	Valve 1-1/2"	Winn Marion	\$ 1,232	7/11/95
LV-118	Valve - 1"	Winn Marion	\$ 1,049	7/11/95
LV-201	Valve - 3 way	Power Controls	\$ 3,043	6/19/95
DV-203	Valve - 3"	Winn Marion	\$ 2,007	7/11/95
FV-206	Valve - 3"	Winn Marion	\$ 1,966	7/11/95
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



**Table 12 (Cont'd). Status of Instrumentation**

<u>TAG #</u>	<u>Description</u>	<u>Company</u>	<u>Price</u>	<u>Delivery</u>
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	7/3/95
FCV-209	Control Valve	Winn Marion	\$ 1,049	7/11/95
IT-SG1	Power Monitor	Power Equipment	\$ 1,501	6/20/95
IT-121	Motor Monitor	Power Equipment	\$ 806	6/20/95

**Installation and Configuration of Control and Data Acquisition System**

Configuration of the Honeywell Series 9000 controller and SCAN 3000 Data Acquisition Unit was completed during the quarter. The control screens and graphics, which were completed by Entech Global personnel, are operational. The configuration of the Series 9000 controller, was completed by Mr. Bob Reynouard of Control Technologies, Inc. (a consultant), with assistance from Honeywell and Entech Global. Mr. Charlie Richmond, a Honeywell Principal System Consultant, was on site from July 24 through July 28, August 28 through August 31, and September 5 through September 8 to support the configuration efforts. Mr. Reynouard and Entech Global personnel will perform some general screen "cleanup" tasks during the upcoming quarter as well as complete the installation of a printer.

**Refurbishing of Existing PDU Equipment**

Reconditioning of existing process equipment items was completed during the quarter. The status of all reconditioned equipment is shown in Table 13.

**Transformer Installation**

Transformer installation efforts commenced July 11, 1995. Public Service of Colorado installed a pole line from McIntyre Street along Amax's northern property line to a point east of the pilot plant. From there, underground service runs from the pole line to the new 1500 kVA transformer located near the eastern access doors of PDU area 200. The new transformer, which was received July 17, was energized and placed into service on July 25. All PDU equipment is now serviced by the new power distribution system.

**Table 13. Existing Equipment Items for Reconditioning**

<u>Equip #</u>	<u>Description</u>	<u>Schedule</u>	<u>Price</u>
100-D-01	Dump Hopper	Complete	\$800
100-T-03	Elevating Conveyor	Complete	\$300
100-T-04	Weight Belt Feeder	Complete	\$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	Complete	\$100
400-Y-01	Tailings Filter Press - 1	Complete	\$3,400
400-Y-02	Tailings Filter Press - 2	Complete	\$3,400
400-Y-04	Clean Coal Filter Press - 1	Complete	\$16,472
400-Y-05	Clean Coal Filter Press - 2	Complete	\$16,472
400-Y-09	Clean Coal Drum Filter	Complete	\$1,500
N/A	Samplers	Complete	\$2,000

### **Subtask 8.3 PDU and Advanced Flotation Module Shakedown and Test Plan**

The majority of all efforts were associated with individual equipment shakedown and evaluation. Any problems, mechanical or electrical, were corrected accordingly by TIC or Entech personnel. The remaining efforts dealt with assembly of the PDU flotation module test plan.

#### **PDU Flotation Module Shakedown**

Efforts related to the PDU module shakedown and test plan, commenced during the quarter. All process equipment items were checked for proper rotation (where applicable) and operated individually. All equipment items, except for the Techweigh scale, passed the shakedown test and are considered ready for operation.

Some additional problems (mostly electrical) were discovered during initial shakedown efforts. As a result, a TIC electrician was retained through the latter part of September to complete the associated tasks. The following shakedown related activities were completed during the third quarter of 1995:

- Pressure tested pipe lines between Netzsch piston pumps and Netzsch filters
- Installed calibration tubes on chemical metering pumps
- Stroked control valves via transmatron and CDAS system
- Filled entire flotation module with water
- Repaired leaks in Microcel™ flanges
- Checked operation of vibrating feeder 100-T-02 - ok

- Checked operation of elevating conveyor 100-T-03 - ok
- Checked operation of bin activator 100-T-05 - ok
- Checked operation of screw conveyor 100-T-06 - ok
- Checked operation of primary ball mill 100-Y-01 - ok
- Checked operation of primary ball mill discharge pump 100-G-01 - ok
- Checked operation of secondary ball mill 100-Y-02 - ok
- Checked operation of secondary ball mill discharge pump 100-G-02 - ok
- Checked operation of fine grinding mill feed pump 100-G-03 - ok
- Checked operation of ground product pump 100-G-04 - ok
- Checked operation of Microcel™ feed pump 200-G-01 - ok
- Checked operation of Microcel™ recirculation pump 200-G-02 - ok
- Checked operation of Microcel™ tailings pump 200-G-03 - ok
- Checked operation of clarified water pump 400-G-07 - ok
- Checked operation of drum filter feed pump 400-G-08 - ok
- Checked operation of clean coal conveyor 400-T-03 - ok
- Checked operation of clean coal conveyor 400-T-04 - ok
- Checked operation of sump pump 400-G-11 - ok
- Calibrated Microcel™ air control valve
- Calibrated Microcel™ wash water valve
- Installed air booster on Microcel™ density control valve
- Increased slope of Sizattec screen overflow line
- Installed push water line in Sizattec screen oversize discharge chutes
- Calibrated Microcel™ level control valve. Due to valve sticking, Red Valve Corporation supplied a new electro-pneumatic positioner which also sticks. Replaced with I/P converter and booster.
- Corrected Sizattec screen motor rotation
- Checked rotation of Netzsch fine grinding mill
- Filled Netzsch fine grinding mill with 3-mm glass media
- Repaired primary ball mill discharge pump after unit locked up with grinding media. Balls were removed and pump operating normal
- Lined primary and secondary ball mill trommel discharge chutes with new fine screen
- Installed a variable frequency drive (VFD) on ground product pump (100-G-04). The unit was installed in order to better control the level of the ground product sump (100-D-06) and help stabilize the solids concentration of slurry feeding the Microcel™
- Re-installed lightning deterrent system on roof of PDU pilot plant

- Installed baffles in Netzsch fine grinding mill feed sump (100-D-07) to control / stop splashing
- Installed baffles in tailings filter press feed sump (400-D-04) to control / stop splashing
- Reversed control wiring between Netzsch piston pumps (400-G-04 & 400-G-05)
- Relocated Microcel™ level indicators / sensors as per Control International, Inc.
- Performed water balance of PDU area 200 on September 5 and 6. Experienced some surging problems with the Microcel™ tailings. Discovered sticking problem with level control valve
- Troubleshoot and corrected operating problem with Drais attritor mill
- Worked on automation of frother pump (200-G-04). Discovered that control board was bad. Replaced entire controller with a Baldor unit
- Worked on automation of collector pump (200-G-05)
- Troubleshoot and corrected operating problem with automatic clean coal samplers. Found that solenoid activators were not working properly. Replaced malfunctioning solenoids with new units from spare samplers
- Initiated setup and calibration of Techweigh weigh belt feeder (100-T-04). Found some compatibility problems with scale (obsolete) and updated controller. Technical support from vendor continuing with positive results
- Installed new starter contactors on primary ball mill (100-Y-01) and secondary ball mill (100-Y-02). Previous units overloaded which required replacement of each unit
- Calibrated nuclear density gauge (DIC-203)
- Troubleshoot operating problem with WesTech drum filter blower. WesTech representative John Smith found that the blower fins and shaft are bent. He recommended that the entire unit be replaced
- Received six (6) loads of crushed Taggart coal from Ralston Development Company (approximately 80-85 tons) on September 12
- Performed water balance of PDU areas 100 and 200 on September 14. Overall, system balanced very well. All sump levels were controlled very tightly
- Operated PDU flotation module on September 21 at a reduced feed rate (Taggart coal at 1000 lb/hr estimate). Found that clean coal pipe exiting Microcel™ is too small and should be increased from 3 inches to 8 inches
- Replaced approximately 60 percent of 3 inch Microcel™ clean coal pipe with 8 inch PVC
- Installed throttle valve on discharge line of drum filter feed pump (400-G-08)

- Resized sheaves on ground product pump (100-G-04) to alleviate sporadic full sump conditions associated with the ground product sump (100-D-06)
- Installed recirculation line in discharge of cyclone feed pump (100-G-02) to better stabilize the solids concentration of slurry feeding the Microcel™

All Proportional Integral Derivative (PID) Loops were configured and tested during the quarter. Specifically, the following loops were evaluated and considered operational:

- Area 100 - Cyclone sump level control
- Area 100 - Ground product sump level control
- Area 200 - Microcel™ feed density control
- Area 200 - Microcel™ feed sump level control
- Area 200 - Microcel™ level control
- Area 200 - Microcel™ tailings level control
- Area 200 - Microcel™ air delivery control
- Area 200 - Microcel™ wash water control
- Area 200 - Microcel™ recirculation flow rate control

The Techweigh weigh belt feeder exhibited some operational difficulties during the quarter. Efforts to calibrate the unit were unsuccessful due to improper compatibility of scale/controller parameters. Because the weigh belt feeder and its associated technology is somewhat obsolete, technical support from Technetics personnel is painstakingly slow. Continued progress has been made, however, with calibration scheduled for completion early next quarter.

Shakedown of the PDU flotation module is slated for completion during the upcoming quarter.

### **Equipment Repairs**

During the initial PDU shakedown, operators noticed an irregular noise generated from the secondary ball mill speed reducer. Inspection of the unit by qualified reducer representatives indicated that the unit is in a failure mode. Continued use of the unit may result in total failure and unwanted downtime of the PDU. As a result, Reliance/Dodge will supply a new speed reducer for replacement during the upcoming quarter. A local machinist/millwright company will perform all related installation work.

### **Test Plan**

The test plan is currently under preparation. It was discussed at a Project Review Meeting on September 21, 1995 at Amax R&D. Those in attendance included:

- S. Bajwa - Bechtel
- M.V. Chari - Bechtel
- H. Huettenhain - Bechtel
- T. Feeley - DOE
- D. Phillips - VPI
- M. Jha - Entech
- N. Moro - Entech
- F. Smit - Entech
- G. Shields - Entech

Based on this discussion, the flotation test plan matrix was restructured by adding a constant spray water ratio point to each high and low feed rate point and reducing the collector dosage range for the Taggart coal.

## PLANS FOR NEXT QUARTER

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

- Subtask 3.2 - Near Term Applications testing will continue as follows
  - Obtain performance data for the 30-inch Microcel™ column when cleaning minus 0.75 mm coal fines at the Lady Dunn Preparation Plant.
  - Perform solid-bowl and screen-bowl centrifuge dewatering tests on the Lady Dunn clean coal froth using the GranuFlow process.
  - Set-up equipment and begin in-plant horizontal belt vacuum filter tests at Lady Dunn to dewater clean coal froth.
  - Explore feasibility of briquetting the fine coal from the Lady Dunn plant in order to produce a higher-valued product for near-term sale.
- Subtask 3.3 - Dewatering Studies will continue with the evaluation of additional hydrocarbons via contact angle measurements. The construction and assembly of a batch dewatering test unit will also be carried out.
- A draft of the Subtask 4.4 Topical Report will be distributed to DOE and project partners for review and comment.
- Work will continue on Subtask 6.4, Selective Agglomeration CWF Formulation Studies.
- Efforts for the Subtask 6.5 work will continue with the evaluation of the Winifrede and Taggart 2 coals in the 25 lb/hr continuous agglomeration unit.
- Work will be completed on the Task 7 detailed design of the selective agglomeration portion of the 2 t/hr PDU.
- Under Subtask 8.1, four hundred tons of compliance-grade Indiana VII coal will be purchased from the Minnehaha Mine for delivery in mid-November.
- Subtask 8.3 efforts will be directed toward the following:
  - Complete and submit test plan
  - Complete shakedown of PDU flotation module.
- Subtask 8.4 efforts will be directed toward the following:
  - Complete parametric testing of Taggart coal
  - Commence parametric testing of Indiana VII coal.

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# ***APPENDIX A***

## **Elkhorn No. 3 Coal Agglomeration Results**

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# ***APPENDIX B***

## **Taggart 2 Coal Agglomeration Results**

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