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

Task 9. Selective Agglomeration Module Testing and Evaluation

Subtask 9.4 Selective Agglomeration Topical Report

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ABSTRACT

The primary goal of this project was the engineering development of two advanced physical fine coal cleaning processes, column flotation and selective agglomeration, for premium fuel applications. The project scope included laboratory research and bench-scale testing of both processes on six coals to optimize the processes, followed by the design, construction, and operation of a 2 t/hr process development unit (PDU). The project began in October, 1992, and is scheduled for completion by September 1997.

This report summarizes the findings of all the selective agglomeration (SA) test work performed with emphasis on the results of the PDU SA Module testing. Two light hydrocarbons, heptane and pentane, were tested as agglomerants in the laboratory research program which investigated two reactor design concepts: a conventional two-stage agglomeration circuit and a unitized reactor that combined the high- and low-shear operations in one vessel. The results were used to design and build a 25 lb/hr bench-scale unit with two-stage agglomeration. The unit also included a steam stripping and condensation circuit for recovery and recycle of heptane. It was tested on six coals to determine the optimum grind and other process conditions that resulted in the recovery of about 99% of the energy while producing low ash (1-2 lb/MBtu) products. The fineness of the grind was the most important variable with the D_{80} (80% passing size) varying in the 12 to 68 micron range. All the clean coals could be formulated into coal-water-slurry-fuels with acceptable properties.

The bench-scale results were used for the conceptual and detailed design of the PDU SA Module which was integrated with the existing grinding and dewatering circuits. The PDU was operated for about 9 months. During the first three months, the shakedown testing was performed to fine tune the operation and control of various equipment. This was followed by parametric testing, optimization/confirmatory testing, and finally a 72-hour round the clock production run for each of the three project coals (Hiawatha, Taggart, and Indiana VII).

The parametric testing results confirmed that the Taggart coal ground to a D_{80} of 30 microns could be cleaned to 1 lb ash/MBtu, whereas the Hiawatha and Indiana VII coals had to be ground to D_{80}s of 40 and 20 microns, respectively, to be cleaned to 2 lb ash/MBtu. The percent solids, residence time, shear intensity (impeller tip speed and energy input per unit volume), and heptane dosage were the main variables that affected successful operation (phase inversion or microagglomerate formation in the high-shear reactor and their growth to 2-3 mm in size during low shear). Downward inclination of the vibrating screen and adequate spray water helped produce the low ash products. Btu recoveries were consistently greater than 98%. Two-stage steam stripping achieved about 99% heptane recovery for recycle to the process. Residual hydrocarbon concentrations were in the 3000 to 5000 ppm range on a dry solids basis.

It was also found that the residual concentrations of several toxic trace elements were reduced substantially, over 25% on a heating value basis. The cleaning also reduced the ash fusion temperatures of the Indiana VII and Taggart coals.

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EXECUTIVE SUMMARY

This project is a major step in the Department of Energy's (DOE) 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-shared contract effort began on October 1, 1992, and is scheduled for completion by September 30, 1997. This report summarizes the findings of all the selective agglomeration test work completed during the course of this project, with the main emphasis on the results of the Subtask 9.3 Selective Agglomeration Operation and Clean Coal Production work. Also included in this report are brief summaries covering the Task 6 Selective Agglomeration Laboratory and Bench-Scale test work including Subtask 6.1 Agglomerating Agent Selection, Subtask 6.2 Grinding, Subtask 6.3 Process Optimization Research, Subtask 6.4 CWF Formulation Studies, and Subtask 6.5 Bench-Scale Testing and Process Scale-up.

SPECIFIC OBJECTIVES OF PROJECT

The three main objectives of the project are discussed below.

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

A secondary objective of the work was to develop a design base for near-term commercial applications of these advanced fine coal cleaning technologies. These applications were to 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 was also to include the auxiliary systems required to yield a shippable, marketable product such as a dry clean coal product.

A third objective of the work was 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. Twelve toxic trace elements were targeted. They were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium, and chlorine. The results of this work indicate the potential for removing these toxic trace elements from coal by advanced physical cleaning.

APPROACH

The project team consisted 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, and the Center for Coal and Mineral Processing (CCMP) of the Virginia Polytechnic Institute and State University. Entech Global, Inc. managed the project for Amax R&D and provided research and development services. Dr. Douglas Keller of Syracuse University and Dr. John Dooher of Adelphi University were both consultants to the project. TIC and Mech El, Inc., two Colorado-based companies, constructed the 2 t/hr process development unit (PDU).

The project effort was divided into four phases which were further divided into eleven tasks including coal selection, laboratory and bench-scale process optimization research and testing, along with design, construction, and operation of a 2 ton/hr PDU. Tonnage quantities of the ultra-clean coals were produced in the PDU for combustion testing. Near-term application of advanced cleaning technologies to existing coal preparation plants were also investigated.

SELECTIVE AGGLOMERATION PROCESS DESCRIPTION

Selective agglomeration is a coal cleaning process based on the differences in surface properties of the coal and its associated mineral impurities. Generally, coal particles are hydrophobic or repel water, while the mineral impurities associated with coal are hydrophilic or easily wetted by water. As such, when a hydrocarbon based agglomerant (agglomerating agent or bridging liquid), is added to a finely divided coal water slurry and agitated, the carbon containing coal particles are coated by the agglomerant while the mineral matter remains dispersed in the water phase.

The bridging liquid utilized for selective agglomeration can range from "heavy" organic liquids like fuel oil No. 2, to "light" hydrocarbons such as heptane. The particular type of agglomerant used depends on a number of factors, but is primarily influenced by the feed coal characteristics, process economics, and the required product quality. Depending on the type and quantity of bridging liquid used, the agglomerant is either allowed to remain with the product, or recovered and recycled back to the process.

Generally, when using heavier agglomerants such as fuel oil, the quantity used is minimized and allowed to remain with the product. If, however, a light hydrocarbon such as heptane is used, the quantity used is not so critical since it must be recovered from the product and recycled to the process for health, safety, environmental, and economic reasons.

Based on the results of Subtask 6.1 Agglomerating Agent Selection and Subtask 6.3 Process Optimization Research, heptane was selected for evaluation during subsequent Subtask 6.5 and Subtask 9.3 testing.

High-Shear Agglomeration

During high-shear agglomeration a mixture of water, coal, and heptane is vigorously agitated such that the heptane disperses and makes contact with the coal particles in the slurry. Throughout this agitation, hydrophobic coal particles are attracted to the heptane phase, while the hydrophilic mineral matter is repelled from the heptane and attracted to the water phase. Given the proper proportions, with continued mixing, the heptane coated coal particles coalesce to form microagglomerates, while the mineral impurities remain dispersed in the water phase (phase inversion). During this inversion, two distinct phases are formed, a coal/agglomerant phase (microagglomerates), and a water phase containing the dispersed mineral matter.

Low-shear Agglomeration

Following high-shear agglomeration, the microagglomerates are subjected to a low-shear agglomeration step. To promote agglomerate growth during low shear, the slurry is mixed at a shear rate significantly less than that used during high shear to promote agglomerate growth. For this project, the final process product was in the form of a highly-loaded slurry. As such, the formation of "large" agglomerates, say greater than 2 to 3 millimeters, with sufficient strength to withstand handling without degradation was not required. Therefore, the primary goal of the low-shear unit operation was to provide a product which could be easily recovered, washed, and dewatered on a screen.

Agglomerate Recovery

Once agglomerates are formed during low shear they must be physically recovered to the product. The goal of this unit operation is to achieve high product recovery and a good separation between the product agglomerates and the mineral-matter bearing process water, i.e., minimize coal losses to the process tailings (screen underflow) while minimizing the contamination of both the product with mineral matter, and the process tailings with heptane bearing carbonaceous material. Primary agglomerate recovery was carried out on a vibrating screen where the agglomerates were dewatered and reported to the screen oversize. Secondary agglomerate recovery, from the vibrating screen underflow, was achieved in a froth skimming device in which any floating carbonaceous material was recovered to the process product stream.

Heptane Recovery and Recycle

Once agglomerates are recovered by screening and froth skimming, the heptane must be removed from the product. Heptane recovery from the agglomerated product was accomplished by direct contact steam stripping in two stages. During this process, heat provided by steam evaporated an azeotropic mixture of heptane and water. From this vapor, the two liquids (heptane and water) were condensed, cooled, separated, and recycled to the process.

RESULTS AND DISCUSSION

The results of all the selective agglomeration test work completed throughout the course of this project are briefly discussed below.

Laboratory and Bench-Scale Work

Subtask 2.1 - Coal Selection

Successful completion of the project objectives by both the advanced flotation and selective agglomeration processes was dependent on the selection of suitable source coals. Due to the widely varying quality and economic factors of United States coals, many could not be considered as a feedstock for this project. Accordingly, guidelines were established to evaluate a number of candidate coals and select six coals for use in the project. Guidelines included in the contract Statement of Work suggested the following specifications for coal selection:

- Source Coal Properties
  - Organic sulfur should be less than 258g/GJ (dry basis), or approximately 0.88% for bituminous coals and 0.75% for low-rank coals
  - Ash minerals and pyrite must be sufficiently liberated by practical comminution methods
- Economic Factors - Coal Acquisition
  - Selected coals must be obtained from actively mined seams with reserves in excess of 300 million tons
  - Sufficient quantities must be available for purchase from the same source to meet the needs of the project
  - Market value of the coal should be less than \$1.18/GJ (\$1.25/MBtu) or approximately \$30/ton
- Economic Factors - Fuel Preparation

- Because variations in coal quality may affect the preparation of premium CWF, potential coals should have the following characteristics:
  1. Low ash content
  2. Low total sulfur content
  3. Low organic sulfur content
  4. Liberation of ash bearing minerals and pyrite at coarse sizes
  5. Low inherent moisture
  6. High Hardgrove grindability index
  7. High hydrophobicity

In addition to these parameters, geographic diversity was also considered with at least one coal from each US coal mining region (eastern, midwestern, and western). The initial screening of coals from the Keystone Coal Mining Directory and the Amax Database generated a list of 32.

These candidate coals were then subjected to various evaluations and rankings from which the following five bituminous (all of which had the characteristics required for successful production of premium fuels) and one low-rank coal were selected for testing during Phase I of the project:

- Taggart Coal - This was the highest ranking coal, which also performed very well in amenability testing.
- Sunnyside Coal - This coal compiled a very high score and performed very well in amenability testing.
- Indiana VII Coal - This coal contained less sulfur than most midwestern coals. Though it scored low, the coal was readily available for test work since the Minnehaha mine was owned by Amax Coal.
- Winifrede Coal - Winifrede coal is very typical of the coal produced in West Virginia. It was also readily available since the source mine was owned by Amax Coal.
- Elkhorn No. 3 Coal - This coal, which received a high score, is representative of the coal produced in eastern Kentucky.
- Dietz Coal - Dietz coal was recommended as the single low-rank selection. Though it compiled a low score, it responded better than other low-rank coals to amenability testing.

Subtask 6.1 - Agglomerating Agent Selection

The objective of Subtask 6.1 was to select the appropriate agglomerating agents to be used for testing under Subtask 6.3 Process Optimization Research and other subsequent work. It was determined that the heavy hydrocarbons would not be capable of meeting the project objectives for the following reasons:

- Their use would prevent the project low-ash target specification to be met
- Their presence in the final agglomerated product (since they would not be recovered and recycled to the process) would make coal-water-slurry fuel (CWF) formation difficult.

As such, only two “light “ hydrocarbons were selected during Subtask 6.1 based on the following general criteria:

- Potential carbon recovery and mineral-matter rejection
- Ease of agglomerant recovery for reuse
- Agglomerant availability and cost
- Health and environmental issues
- Effect of residual agglomerant on CWF formulation

Based on these criteria, the two “light” hydrocarbons with the highest rankings were n-heptane and n-pentane. As such, these two agglomerants were selected for evaluation during the Subtask 6.3 Process Optimization Research test work.

Subtask 6.2 - Grinding

During Subtask 6.2, initial selective agglomeration (SA) test work evaluated the grinding requirements necessary to achieve the project goal of 2 lb ash/MBtu for the selected project coals. The main objectives of this work were to:

- Determine the grind size required to achieve the mineral liberation needed to achieve the target CWF fuel specifications.
- Determine the grinding circuit configuration that best met the needs of the 2 t/hr process development unit (PDU) SA module, while allowing scale-up to a commercial premium fuel production plant.
- Determine design and operating parameters of PDU SA module grinding circuit.
- Prepare ground slurries for the Subtask 6.3 Selective Agglomeration Process Optimization Research and Subtask 6.4 CWF Formulation Studies subtasks.
- Determine the capacities of the available Amax R&D grinding equipment for the production of ground slurry feedstock for the Subtask 6.5 test work.

Laboratory agglomeration tests were carried out on the various ground products to quantify the liberation of ash and sulfur for each grind evaluated. It was determined, through liberation testing, that the grind size D_{80}s (80% passing sizes) shown below were required to insure the production of a clean coal containing less than 2 lb ash/MBtu (1 lb ash/MBtu in the case of the Taggart coal):

Test Coal	D_{80} (microns)
Taggart	45
Indiana VII	20
Sunnyside	45
Winifrede	11
Elkhorn No. 3	45
Dietz	20

Overall, a closed-circuit grinding configuration was found to be more efficient than an open-circuit configuration, in that it provided greater capacity for a given grind size. This benefit was most evident when grinding to very fine particle sizes, such as those needed for the Indiana VII, Winifrede, and Dietz coals.

Subtask 6.3 - Process Optimization Research

The main objectives of the Subtask 6.3 test work were to

- Optimize, by laboratory-scale research and testing, the selective agglomeration process to best meet the project clean coal quality and heating value recovery specifications.
- Compare the performance of the two “light” hydrocarbon agglomerating agents and recommend one for further testing at the bench-scale.
- Compare the performance of an innovative reactor design that combined the high- and low-shear mixing zones in a single unit, with the conventional agglomeration design in which the high-shear and low-shear unit operations are carried out separately.

This laboratory-scale testing was carried out at both Arcanum and Amax R&D. Dr. Keller of Syracuse University reviewed the work on an ongoing basis and aided in the interpretation of the results and recommended follow-up tests. Both n-pentane (C_5H_{12}) and n-heptane (C_7H_{16}) were employed as bridging liquids for much of the work.

The selective agglomeration process, as tested during this project, involved three primary unit operations, high-shear mixing, low-shear mixing, and screening. Subtask 6.3 testing focused on the high- and low-shear mixing steps via the following three types of laboratory-scale agglomeration tests:

1. Waring blender batch tests to quickly assess process inversion times, the behavior of differing coals, particle size distributions, bridging liquids, pretreatments, and activators, thereby providing a standard for gauging the performance of the continuous test apparatus.
2. Continuous testing in a single-stage unitized reactor system (combined high- and low-shear) that had the capacity to agglomerate about 50 grams/min (about 5 lb/hour) of coal.

3. Conventional two-stage agglomeration testing (separate high- and low-shear mixing steps) carried out at Arcanum in a test unit with a comparable capacity (about 50 grams/min or 5 lb/hour).

All five bituminous coals responded well to laboratory scale selective agglomeration with both pentane and heptane as the bridging liquids. Target residual ash and heating value recovery specifications were easily met for the Taggart, Elkhorn No. 3, Sunnyside, Indiana VII, and Winifrede coals ground to the fineness projected from the Subtask 6.2 liberation studies. The target sulfur specification was met when cleaning the Taggart, Sunnyside, and Indiana VII coals. The subbituminous Dietz coal did not respond well and required a considerable amount of asphalt activation and acidification before agglomeration, so agglomeration may not be a cost-effective method for cleaning that coal. As such, this coal was not included in subsequent testing.

It was especially noteworthy that over 98% of the heating value was recovered from four of the five bituminous coals and that recovery from even the very finely ground Winifrede coal exceeded 94% when achieving the desired ash rejection.

Pentane, pure heptane, commercial heptane, and dearomatized (hydrotreated) commercial heptane bridging liquids appeared to be equally capable of agglomerating the ground test coals while effectively rejecting ash minerals. The heptane to coal and pentane to coal bridging liquid ratios for good agglomeration ranged from 0.18 gram hydrocarbon per gram coal on up to 0.36 gram per gram coal. The more finely ground coals, such as the Winifrede, required more bridging liquid.

Agglomeration proceeded well in both of the continuous systems under a variety of operating conditions (percent solids, impeller speeds, feed rates, etc.). The heating value recovery fell sharply and agglomeration ceased when the capacity of the units were exceeded at high feed rates/short retention times. Changes in operating conditions had little impact upon the amount of residual ash left in the agglomerated clean coal.

The separation performances of the unitized reactor and the two-stage system were similar, but it appeared that the two-stage system required less high-shear mixing energy for agglomerating fine coal. Since the unitized reactor system did not seem to offer any power-saving advantages, the two-stage system was recommended for use in the bench-scale testing because its development was further along and more scale-up information was available from work performed by Arcanum and Bechtel under a prior DOE project. High-shear mixing energy consumptions in the two-stage system were in the 11.8 to 23.6 kwhr/ton range for minus 325 mesh coal.

Subtask 6.4 - CWF Formulation Studies

Following the completion of the Subtask 4.3 Coal Water Slurry Fuel (CWF) test work utilizing advanced flotation products, work began on Subtask 6.4 to investigate the formulation of CWF from selective agglomeration products. During this work, CWFs were formulated from five of the six project coals investigated during Phase I of the project, Taggart, Sunnyside, Elkhorn No. 3, Indiana VII, and Winifrede. Hiawatha coal was tested as well since it was to be utilized during the Subtask 6.5 bench-scale and Task 9 PDU operation test programs as a substitute for the Sunnyside coal. The subbituminous Dietz coal was not tested at all since it could not be cleaned by selective agglomeration sufficiently to meet the product ash specification goal.

A survey of past and present CWF combustion technology suggested that, for oil and gas retrofit applications, the slurry need not contain more than 60-62% coal (or approximately 8,800-9,300 Btu/lb) but should have a viscosity of less than 500 cP.

Subtask 6.4 testing focused on determining the reagent additions and particle size distributions (PSDs) required to meet these goals. Suitable CWF was prepared from the Taggart, Sunnyside, Elkhorn No. 3, and Hiawatha coals that had been ground to D_{80}s in the 34 to 67 micron range and cleaned by selective agglomeration to contain less than 2 lb ash/MBtu. The Indiana VII and Winifrede coals were found to be less desirable feedstocks since they required finer grinding for liberation of the ash minerals. This finer grinding, and in the case of Indiana VII coal the high inherent moisture content, resulted in very low slurry loadings (less than 52%).

It was found that between 10 and 20 lbs/ton coal of A-23M dispersant was required for the Taggart, Sunnyside, Elkhorn No. 3, and Hiawatha coal slurries to achieve 60-62% coal loadings at a viscosity of 500 cP. The solids loadings of 500 cP viscosity slurries, when no dispersant was used, were only in the 50 to 52% range. Generally, the CWFs prepared with dispersant were unstable and would need to be used soon after preparation, or agitated while stored. Their stability was improved by either omission of the dispersant or by adding Flocon 4800C xanthan gum as a stabilizer. In either case, there was a sacrifice in loading and in the case of the stabilizer addition, a significant extra cost for reagents along with an increase in slurry viscosity.

In some cases, it was found that particle size distribution manipulation to produce better packing of the particles in the slurry (bi-modal PSD), increased achievable slurry loadings. However, the improvements were usually meager compared to the higher capital and operating costs associated with the addition of sufficient grinding capacity to achieve these PSD manipulations in a commercial plant. Experimental results were generally consistent with predictions from a slurry properties model developed by Dr. John Dooher of Adelphi University.

Subtask 6.5 - Bench-Scale Testing

The continuous bench-scale testing carried out under Subtask 6.5 had three main objectives:

1. Design, construct, and operate a continuous selective agglomeration system of about 25 lb/hr capacity to demonstrate the feasibility of the process.

2. Optimize the selective agglomeration process conditions to minimize product ash contents, and reduce process costs.
3. Generate design data of sufficient reliability to insure successful scale-up of the process to the process development unit (PDU) 2 t/hr scale.

The bench-scale unit utilized during the Subtask 6.5 was of sufficient size to produce at least 25 lb/hr of agglomerated product (dry basis), and capable of processing all project coals. This testing utilized heptane as the agglomerant, or bridging liquid, which was recovered via steam stripping for recycle to the process.

Bench-Scale Unit Description - To simplify operation, coal grinding was carried out independently of agglomeration testing. Once finely ground to achieve the required liberation, the coal slurry was subjected to a high-shear unit operation in which intense mixing dispersed the bridging liquid (heptane) and provided sufficient heptane/coal and coal/coal contact to achieve a phase inversion and form what are termed “microagglomerates”. These microagglomerates were then subjected to additional mixing in a low-shear unit operation allowing the agglomerates to grow to a sufficient size for physical recovery by screening. Once formed, the agglomerates were dewatered, rinsed, and recovered on a vibrating screen.

Recovery of the heptane from the agglomerated product was achieved in two stages of steam stripping. In the first stage, reslurried agglomerates were steam stripped at ambient pressure boiling temperatures removing the bulk of the heptane. In the second stage, the slurry was subjected to additional steam stripping at elevated temperatures and pressures removing additional heptane. The recovered vapor (heptane and water) was then condensed, cooled, gravity separated, and recycled to the process.

Batch Agglomeration Testing - Batch agglomeration tests were performed on samples of various ground feedstocks to evaluate the liberation characteristics of each grind. Generally, slightly lower product ash levels were achieved in the continuous unit than during the batch tests. A list of grind sizes required to meet the project 2 lb ash/MBtu product specifications via batch testing for each coal is as follows:

- Winifrede Coal - D_{80} = 12 microns
- Elkhorn No. 3 Coal - D_{80} = 68 microns
- Taggart Coal - D_{80} = 15, 30, and 38 microns (1 lb ash/MBtu)
- Hiawatha Coal - D_{80} = 65 and 47 microns
- Indiana VII Coal - D_{80} = 20 microns

Continuous Agglomeration Testing - The Winifrede coal was ground to a D_{80} of 12 microns and continuous agglomeration tests carried out using both fresh commercial grade heptane and recycled heptane, i.e., heptane recovered from previous test work. The 2 lb ash/MBtu product specification was met in many of the tests completed, indicating that the 12 micron D_{80} grind provided sufficient mineral-matter liberation.

Results also indicated that very high Btu recoveries (>99%) were achieved with tailings ash values in the 47 to 89% range (most in the 78 to 89% range).

The Elkhorn No. 3 coal was ground to a D_{80} of 68 microns at which the 2 lb ash/MBtu product specification was met for all but one of the tests completed (1.7 to 1.9 lb/MBtu), indicating that the 100-mesh topsize grind provided sufficient mineral-matter liberation. Btu recoveries achieved were in the 88 to 98% range, with corresponding tailings ash values in the 25 to 65% range. These relatively low Btu recoveries and tailings ash values are attributed to oxidation of the Elkhorn No. 3 coal which had been stored for over two years prior to its use for this work.

The Sunnyside coal was ground to D_{80}s of 60 and 43 microns followed by the completion of high-shear evaluation and continuous agglomeration testing. The high-shear testing used both feedstocks and two different high-shear impellers to determine the minimum high-shear impeller tip speed required to achieve inversion at various coal feed rates and solids concentrations. Trends observed during this work included:

- As residence time in high shear decreases, impeller tip speed must be increased to maintain inversion.
- As solids concentration increases, lower impeller tip speeds are required to achieve inversion.

Some of the Sunnyside coal tests completed with each feedstock met the 2 lb/MBtu product ash specification at high Btu recoveries (>98%), indicating sufficient liberation. This testing also showed that lower product ash contents were achieved at solids concentrations of 5 and 7% than at 10 and 13%.

Taggart coal testing was carried out at feedstock D_{80}s of 91, 88, 65, and 33 microns. Results from this work showed that all four of the feedstocks met the 2 lb/MBtu product ash specification. However, only the finest grind tested (D_{80}=33 microns) was able to achieve the 1 lb/MBtu product ash specification. Btu recoveries were high (>96%) for all of the tests, with tailings ash values in the 32 to 83% range.

Continuous agglomeration testing utilizing the Indiana VII coal was carried out using feedstocks with D_{80}s of 22 and 26 microns. Results from this work showed that the product ash specification of 2 lb ash/MBtu was met, at approximately 99% Btu recovery, for the D_{80}=22 micron feedstock, indicating that this grind size provided sufficient liberation. The addition of 7.5 to 20 lb asphalt/ton of coal was required to achieve phase inversion during high-shear agglomeration.

The Hiawatha coal was tested at D_{80}s of 47 and 65 microns. Results of this testing indicated that the product ash specification of 2 lb ash/MBtu was met for both of these feedstocks. For the finer of these two grinds, Btu recoveries were all greater than 99% with tailings ash values in the 81 to 87% range. However, for the coarser grind, Btu recoveries were slightly lower, 96.6 to 99.6%, with tailings ash values in the 56 to 80% range.

Agglomeration Testing Conclusions - The results of the Subtask 6.5 agglomeration test work indicate that the product ash specification of 1 to 2 lb/MBtu, as well as the Btu recovery goal of at least 80% on a run-of-mine basis were met for all six of the coals tested. Of paramount importance in achieving these product ash levels was the size to which the coal was ground. The coarsest particle size distribution to which each coal was ground to achieve the project goals are summarized below along with the typical product ash and Btu recovery values attained when operating the system at optimized conditions:

	PSD Summary, Microns				Ash	Btu Recovery, %	
Coal	D_{20}	D_{50}	D_{80}	MMD	lb/MBtu	Agglomeration	Run-of-Mine
Taggart	5.9	16.2	32.8	23.0	0.95	99.1	93.5
Sunnyside	8.0	24.9	59.6	34.3	1.79	98.3	88.6
Indiana VII	4.2	10.4	21.9	14.5	1.95	99.0	89.6
Elkhorn No. 3	10.7	29.6	68.0	39.4	1.69	96.8	91.6
Winifrede	2.0	4.2	12.4	7.1	1.91	99.2	88.8
Hiawatha	11.5	32.9	65.2	40.9	1.85	99.6	89.7

It was found that the following guidelines should be followed to insure that consistent results are achieved:

1. Sufficient heptane must be used during high shear to achieve phase inversion, form microagglomerates, and allow agglomerate growth during low shear to the 2 to 3 mm size range. This requirement was found to vary between 25 and 60% heptane on a dry ash free coal basis.
2. Sufficient agitation intensity (10 to 18 m/s impeller tip speed) must be applied during high shear to achieve complete dispersion of the heptane and enough particle to particle contact to form microagglomerates.
3. Sufficient residence time must be provided in high shear to allow microagglomerates to form. Typically 30 to 60 seconds are necessary, depending primarily on coal fineness and rank.
4. The use of higher solids concentrations reduces high-shear energy requirements.
5. For lower rank and oxidized coals like the Indiana VII, the use of an agglomeration promoter like asphalt is required to achieve agglomeration.
6. The formation of consistent agglomerates in the 2 to 3 mm size range is paramount in achieving low product ashes. If agglomerates are too small, drainage of mineral-matter bearing process water will not occur. If agglomerates are too large, their handleability diminishes affecting downstream operations.
7. Low -shear Impeller tip speeds of about 5 m/s allowed the growth of well formed agglomerates of sufficient strength for vibrating screen recovery.

8. Low-shear residence times of 2 to 3 minutes are recommended since longer residence times make agglomerate growth difficult to control.
9. The design of the low-shear vessel should be such that the discharge is located at the same elevation as the impeller to insure continual low-shear discharge.
10. The vibrating screen used for agglomerate recovery must have sufficient forward linear motion to provide good transport of agglomerates across the screen deck.
11. The use of a froth skimmer to recover coal from the screen tailings was shown to result in Btu recovery increases on the order of 1 to 3%, depending on the coal and operating conditions used.

Stripper Testing - In an effort to better quantify the residual heptane concentrations remaining with a stripped agglomerated product, a number of batch stripper tests were carried out. Two types of heptane were used, a commercial grade heptane (21-25% n-heptane) and a pure grade heptane (>99% n-heptane). Batch stripping test results indicated that:

- Lower residual hydrocarbon concentrations were achieved as stripper residence time was increased.
- Thermal drying achieved much lower residual heptane levels than boiling.
- Steam stripping at elevated temperatures and pressures resulted in reduced residual hydrocarbon concentrations.
- Steam stripping at 25% solids concentration resulted in the same levels of residual heptane as when the stripping was carried out at 10% solids.
- Storage of agglomerated product prior to stripping resulted in higher residual hydrocarbon concentrations than immediate stripping.
- Under virtually all conditions tested, the presence of asphalt resulted in lower residual hydrocarbon concentrations.
- The residual heptane concentration of stored highly loaded CWF slurries did not decrease, indicating no safety and/or environmental related risks.

Continuous stripping test results indicated the following:

- Two stages of steam stripping achieved lower trace heptane concentrations than a single stage of steam stripping. This was due to the increased temperature used in the second stage.
- There were no obvious trends relating residence time to residual hydrocarbon concentrations.
- There was no difference in performance between operating the stripping column flooded (high liquid level) or in a continuous steam mode (low liquid level).
- A significant reduction in the exiting vapor temperature, indicating reduced steam consumption, did not result in higher residual hydrocarbon contents.

- Residual hydrocarbon concentrations were in the 2000 to 5000 ppm dry coal basis (dcb) range for the first-stage stripper products and in the 1000 to 3000 ppm dcb range for the second-stage stripper products.
- Regardless of whether a commercial or pure grade of heptane was used during agglomeration, total residual hydrocarbon concentrations achieved were similar.

Tailings Heptane Analysis - One set of agglomeration tailings samples (froth skimmer underflow) was analyzed for residual heptane content. These samples originated from an Elkhorn No. 3 coal agglomeration test utilizing commercial grade heptane. The ash content of this tailings sample was approximately 50%. Samples submitted included as produced 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. These results indicate that tailings disposal in conventional waste disposal sites should not be a problem.

Toxic Trace Elements Distribution - The reduction in toxic trace element (TTE) concentrations accomplished by selective agglomeration was studied by assaying the products from selected parametric bench-scale tests and calculating the distribution of the trace elements between the clean coal and tailings. The TTEs of interest were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine. The reductions in the various trace element concentrations were calculated on a heating value basis and generally, the concentrations of arsenic, beryllium, cobalt, lead, manganese, mercury, and selenium in the raw coals were clearly reduced by the combined conventional washing and advanced cleaning steps. Selective agglomeration reduced the concentrations of arsenic, chromium, manganese, and nickel remaining in the ground washed coals. Selective agglomeration had little impact upon the beryllium, cobalt, lead, mercury, and selenium concentrations, and it appears that at times the antimony and chlorine concentrations increased on a heating value basis. Overall, the residual amounts of the elements in the clean coals were found to be dependent upon the source coal.

Design and Construction of PDU SA Module

The design and construction of the process development unit (PDU) Selective Agglomeration (SA) Module is discussed in the following sections.

Subtask 6.6 - Conceptual Design of SA Module

The conceptual design of the PDU SA Module was a collaborative effort between Bechtel, Entech, and Arcanum. Flow diagrams and equipment selection are presented in the Subtask 6.6 Selective Agglomeration Module Conceptual Design Report. Based on the results obtained from the Subtask 6.3 Process Optimization Research and

Subtask 6.5 Bench-Scale Testing and Process Scale-up, the following conceptual design selections were made for the 2 t/hr PDU SA Module:

- Heptane, rather than pentane, was chosen as the agglomerant.
- A conventional two-stage agglomeration circuit (separate high- and low-shear unit operations) was chosen rather than the combined high- and low-shear unitized reactor tested during Subtask 6.3.
- It was determined that due to the long high-shear residence times required to achieve inversion with the Indiana VII coal (2 to 3 minutes), two stages of high shear would be installed
- It was determined that due to handling problems associated with the recovered agglomerates and residual heptane concentration considerations, two-stages of steam stripping would be utilized.

Task 7 - Detailed Design of SA Module

The detailed design of the PDU SA Module was performed by Bechtel Corporation of San Francisco, CA with support from Entech Global and Arcanum Corporation engineers. Details of this work can be found in the 3-volume Subtask 7.0 Detail Design of PDU and Selective Agglomeration Module Engineering Package.

All structural drawings as well as P&ID's were completed by Bechtel and issued for construction. Electrical drawings were issued by Control Technologies, Inc. Entech Global managed the procurement of all instrumentation as well as all new and refurbished capital equipment items used in the PDU SA Module.

Subtask 9.1 - Construction of SA Module

Construction of the PDU SA Module was carried out under Subtask 9.1. Request for Quotation (RFQ) packages were issued to four Colorado based construction companies during the last quarter of 1995. Entech Global and Bechtel personnel collaborated to decide issues regarding work scope and components of the RFQ. Following site inspection meetings for interested bidders, the subcontract for this work was awarded to Mech El, Inc. (MEI), of Aurora, Colorado. MEI mobilized onto the Amax R&D site on March 11, 1996 and was responsible for the installation of all process equipment, instrumentation, structural steel, concrete, process piping, power systems, and control systems. Control Technologies, Inc. of Lakewood, Colorado, was hired for development of the control and data acquisition system (DCS). Construction was completed during November 1996.

PDU Process and Plant Description

Area 100 - Raw Coal Handling

The three coals cleaned in the PDU SA module were normal commercial products of coal mines (minus 2-inch washed or run-of-mine coal). They were delivered in 100 ton rail cars to a coal yard located in north Denver, CO. The coal was then transported by truck to Ralston Development Company, located five miles north of the Amax R&D facility, where the coal was crushed to a 1/2-inch top size and stored. As needed, the coal was transported to the PDU site. A front end loader, receiving hopper, elevating conveyor, storage bin, vibratory feeder, weigh belt feeder, and screw conveyor were then used to feed the coal to the grinding circuit.

Area 100 - Grinding and Classification Circuits

The metered coal was fed to the primary ball mill for initial grinding. The primary mill product was pumped to the secondary ball mill for additional grinding. The secondary mill product was then classified by cyclones, with the oversize recycled for additional grinding in the secondary ball mill or a Netzsch fine grinding mill. The undersize was screened for topsize control before being fed to the agglomeration process (Area 300).

Area 300 - Selective Agglomeration Module

The main units of the selective agglomeration process plant comprising the Area 300 PDU SA module were as follows:

1. High shear agglomeration
2. Low shear agglomeration
3. Agglomerate recovery
4. Heptane Stripping Circuit
5. Condensate Recovery and Recycle
6. Tailings handling
7. Nitrogen blanketing system
8. Pressure relief system
9. Safety Features

High-Shear Agglomeration - Slurry from the grinding circuit was fed to either of two agitated slurry storage tanks from which it was fed to the agglomeration circuit by a variable speed centrifugal pump. The slurry feed rate was automatically controlled to maintain a constant volumetric flow with a nuclear density gauge providing solids concentration indications. This on-line density determination combined with the on-line slurry flowrate and other input variables, provided for the real time calculation of the dry ash free coal feed rate within the control system, which was then used to automatically maintain a constant heptane to coal ratio through the agglomeration circuit.

Coal slurry was fed to the high-shear agglomeration circuit which consisted of two high-shear reactors of 35 and 75 gallon capacity. The piping around these reactors allowed the use of either vessel individually, or both in series. Each of these high-shear vessels was fully baffled and divided into two mixing zones with a radial flow impeller centered in each zone. These impellers were powered by variable speed agitators that could achieve impeller tip speeds in the 14-18 m/s range.

Heptane was metered to the agglomeration process by a metering pump with heptane requirements generally ranging from 20 to 40% of the dry coal feed rate. When required, an agglomeration conditioner (asphalt) was fed into the agglomeration feed line via a gear pump. The asphalt conditioner (in the form of an asphalt emulsion) was only required for the Indiana VII coal, at a rate of 5 to 10 lb asphalt per ton of dry coal.

Low-shear Agglomeration - During low shear, the slurry was mixed at a shear rate significantly less than that used during high shear (impeller tip speeds of 3 to 5 m/s), to provide additional agglomerate growth. The low shear reactor was of 400 gallon capacity and divided into two mixing zones via a horizontal baffle. Discharge ports were arranged so that the low-shear vessel could be operated either full or at half its rated capacity. Centered vertically in each mixing zone was a radial flow impeller. The agitator was provided with a variable speed drive unit. This mixer could achieve impeller tip speeds up to 6.5 m/s.

Agglomerate Recovery - Once agglomerates were formed in low shear, they gravity flowed to the agglomerate recovery circuit which consisted of:

- A vibrating screen fitted with water sprays, from which the agglomerates reported to the overflow and the tailings to the underflow.
- A froth skimmer vessel which utilized a rotating paddle to skim any floating heptane bearing carbonaceous material from the screen tailings stream.

Heptane Stripping Circuit - The combined screen and froth skimmer products gravity flowed to the stripper feed sump from which they were pumped, along with the desired amount of dilution/reslurry water, to the first-stage stripper via a diaphragm pump, which was operated at a speed sufficient to keep the stripper feed tank empty. The agitated first-stage stripper was used to remove the bulk of the heptane and produce a handleable product (basically a coal water slurry). This stage of stripping was carried out at pressures in the 2 to 5 psi range, maintaining a temperature above the boiling point of the heptane/water mixture. The first-stage stripper product was then pumped, via a centrifugal pump, to the second-stage stripper where the residual heptane content was reduced further by stripping at elevated temperatures and corresponding pressures, typically in the 7 to 10 psi range.

The steam flow to the stripping circuit was countercurrent to the process slurry flow (steam entered stripper B first where it picked up a small amount of heptane vapor and then flowed to stripper A). Steam for the plant was generated in a trailer mounted 250 HP boiler fired by natural gas.

Vapor Condensation, Recovery, and Recycle - Heptane and water vapor exiting the stripping circuit were condensed in an air cooler, sub-cooled in a plateflow heat exchanger serviced by chilled water, gravity separated based on the differences in their specific gravities (1.0 for water and 0.7 for heptane), and then recycled to the process.

Tailings Handling - Final process tailings from the froth skimmer underflow piping gravity flowed to the tailings surge tank. The tailings were pumped out of area 300 via a fixed speed centrifugal pump with the proper flow maintained by a flow control valve. Under normal operating conditions, the tailings were pumped directly to the dewatering circuit. However, in the case of an upset condition resulting in contamination of the tailings stream with heptane, this flow was diverted for storage and subsequent steam stripping prior to disposal.

Chilled Water Cooling Circuit - In order to help dissipate heat from the system, a closed circuit chilled cooling water system was included in the design. This closed circuit included a 90 ton water chiller that serviced three different heat exchangers.

Nitrogen Blanketing System - Since heptane is a volatile and flammable compound, its use required a nitrogen blanket for health, safety, and environmental considerations. Under this system, all portions of the process in which heptane was present, were maintained under a positive pressure of 2 to 8 inches of water column with nitrogen gas. This insured that no air was drawn into the system allowing the formation of an explosive environment. This system included a variable volume gas holder that provided blanket gas surge capacity, maintained a relatively constant blanket gas pressure, and provided relief capabilities via a rupture disc. Located between the gas holder and the process connections was a gas blanket cooler for condensation of heptane vapors out of the gas blanket.

Pressure Relief System - In case of a major process upset, explosion, or fire inside any Area 300 process vessel, tank, or piping, the SA module was equipped with a pressure relief system. Activation of the pressure relief system was through the opening of any of the eight pressure relief valves installed on various process vessels. Once a pressure relief valve was opened, the relieved material (gas, liquid, or solids) flowed to the main relief header which was connected to a knockout drum. Downstream of the knock-out drum was a continuously operated flare designed to burn any relieved hydrocarbons.

Additional Safety Features - In addition to the gas blanket and pressure relief systems, the following other safety features were incorporated into the SA module:

- A fire protection system that included rate of rise heat detectors, audible alarms, visible alarms, manual pull stations, and automatic notification to the local fire department
- A continually operating, staged capacity ventilation system that provided a slight negative pressure to prevent the leakage of heptane vapors to other areas of the plant.
- Various hydrocarbon and oxygen detectors to detect the presence of oxygen in the gas blanket system, a deficiency of oxygen in the Area 300 atmosphere, and fugitive heptane vapors within and outside of Area 300.
- Various operating system alarms to detect upset conditions.
- A number of automatic interlocks programmed into the plant DCS control system.

Area 400 - Dewatering Circuit

The clean coal was dewatered in a circuit which utilized three filters. A WesTech vacuum drum filter was used as the primary product filtration unit. The remaining clean coal product, along with the filtrate from the drum filter was processed in two Netzsch plate and frame filter presses. Tailings from the SA module were sent to an Enviro-Clear thickener for initial dewatering. The thickened tailings were dewatered by two Eimco pressure filters while the clarified water was recycled to the process.

Subtask 9.2 - PDU SA Module Shakedown and Test Plan

Startup and shakedown of the PDU SA module was completed during the last quarter of 1996 and the first quarter of 1997 according to the Subtask 9.2 SA Shakedown and Test Plan. Though some minor operating difficulties were encountered, corrective actions resulted in a fully functional PDU SA Module. Physical and mechanical improvements resulted in the elimination of process bottlenecks which allowed the PDU SA Module to operate at steady-state conditions.

The main tasks carried out during start-up and shakedown testing included:

- Training a team of eight engineers, operators, and technicians who would insure safe effective operation of the PDU.
- Development of all required startup, operating, shutdown, and emergency procedures for the three plant Areas 100, 300, and 400.
- Start-up and shakedown testing of the SA Module by operating personnel, who insured that all equipment, instruments, process control loops, and safety features operated as designed.
- Development of the SA Module operations test plan which included parametric, optimization, and production testing of the three project coals (Hiawatha, Taggart, and Indiana VII).

Subtask 9.3 - PDU SA Module Operation and Clean Coal Production

The SA module was integrated with the existing PDU facility constructed during Subtask 8.2 and operated under Subtask 8.4. During operation of the SA module, the existing coal handling and grinding circuits (Plant Area 100) were used to produce ground coal slurry feed for the selective agglomeration process. Similarly, the existing product and tailings dewatering circuits (Plant Area 400) were also used. As such, the SA module (Plant Area 300) essentially replaced the Microcel™ flotation column (Plant Area 200), with the remainder of the plant remaining intact.

Test Coal Feedstock Characterization

Characterization of the three test coals used during Subtask 9.3 was completed by an outside laboratory. The results of these analyses are summarized below:

	Taggart		Indiana VII		Hiawatha	
	As-Recv'd	Bone Dry	As-Recv'd	Bone Dry	As-Recv'd	Bone Dry
Proximate, %:						
Ash	3.30	3.50	7.94	9.55	7.75	8.20
Volatile Matter	32.13	34.12	27.36	32.92	40.02	42.35
Fixed Carbon	58.73	62.38	47.81	57.53	46.72	49.45
Moisture	5.84		16.89		5.51	
Sulfur, %:						
Total	0.61	0.65	0.42	0.51	0.49	0,52
Pyrite	0.05	0.05	0.12	0.15	0.07	0.07
Sulfate	< 0.01	< 0.01	0.01	0.01	< 0.01	< 0.01
Organic	0.56	0.60	0.29	0.35	0.42	0.45
Ultimate, %:						
Carbon	80.30	85.28	62.40	75.39	72.93	77.18
Hydrogen	4.66	4.95	3.94	4.74	4.92	5.21
Nitrogen	1.38	1.47	1.40	1.68	1.37	1.45
Oxygen	3.91	4.15	6.75	8.13	7.03	7.44
HHV, Btu/lb	13,874	14,735	10,828	13,028	12,725	13,647
Equil. Moist., %	2.6		14.5		4.3	
Density, kg/m^3		1,260		1,360		1,275
HGI	49		54		44	
Coal Rank	hvA		hvC		hvA	
Prep Plant Yield, %		57.2		61.9		100.0
Prep Plant Btu Rec, %		84.9		90.5		100.0

Hiawatha Coal Parametric Testing Results

Parametric testing of the Hiawatha coal focused primarily on the evaluation of:

- High-shear agglomeration

- Low-shear agglomeration
- Vibrating screen
- Froth skimmer
- Steam stripping

A summary of parametric testing findings for the Hiawatha coal is as follows:

- While the SA module could be run at a 2 t/hr coal feed rate, a more stable operation was achieved at a slightly lower feed rate (3800 lb/hr).
- A feedstock grind with a D_{80} of approximately 40 microns was sufficiently fine to achieve the 2 lb ash/MBtu product target ash level for the Hiawatha coal.
- Overall plant Btu recoveries were very high, > 99%.
- There appeared to be no effect of high-shear energy input on product ash content as long as “good inversion” was maintained in high shear.
- Operation of the low-shear vessel half full provided sufficient residence time for agglomerate growth to a recoverable size.
- Operation of the low-shear vessel full resulted in an unstable (cyclic growth) pattern
- Lower product ash was achieved at higher screen spray water flow rates.
- Lower product ash was achieved when the screen was in the downhill orientation than when level, and when level as compared to uphill.
- There was no effect on plant performance when the nitrogen purge was used as compared to when it was not used in the froth skimmer.
- The froth skimmer design did not work well because of the presence of too much surface area for froth collection in the froth skimmer, the small amount of material floating in the skimmer, the possible readsorption of heptane from the heptane saturated nitrogen gas blanket onto the material that floats in the skimmer, and poor distribution of the nitrogen bubbles.
- No clear trends were obvious relating steam stripping residence times and operating temperatures to product residual heptane concentrations.
- No deleterious effects were observed when operating the stripping circuit at relatively high (20-25%) solids concentrations.
- Steam consumption was typically on the order of 1300 lb/ton coal.
- Product residual heptane concentrations were typically in the 2000 to 3000 ppm range on a dry solids basis.
- Tailings residual heptane concentrations were typically in the 1000 to 2000 ppm range on a dry solids basis.

Hiawatha Coal Production Run

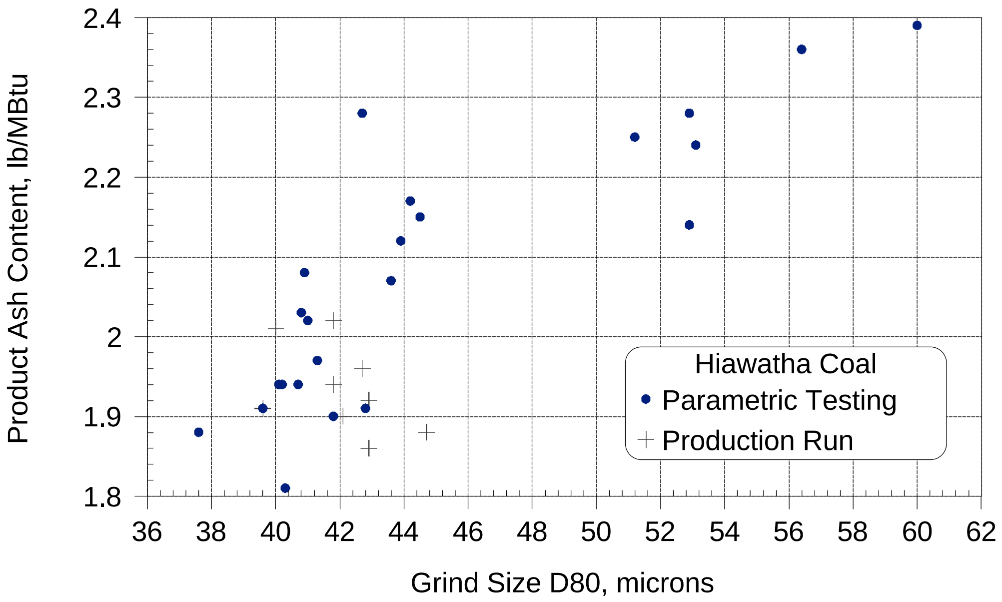
The Hiawatha coal production run was carried out during the week of April 14, 1997. The production run met the 2 lb ash/MBtu product specification for all of the sample periods evaluated. The following is a summary list of average production run operating conditions and results:

- Dry coal feed rate - 3839 lb/hr
- Plant feed grind D_{80} - 42.1 microns
- Plant feed solids concentration - 10.24%
- Plant feed ash content - 8.34%
- Heptane dosage utilized - 31.3% on a dry ash free coal basis
- Total agglomeration (high- and low-shear) energy input - 17 kwhr/ton feed coal (6.9 kwhr/1000 gallon slurry)
- Screen spray water rate - 500 gallons/ton product
- Steam consumption - 1380 lb/ton dry product (1.8 lb/gallon slurry stripped)
- Plant product ash content - 1.93 lb/MBtu (2.78%)
- Plant product residual heptane content - 2951 ppm on a dry coal basis
- Plant tailings ash content - 80.4%
- Plant tailings residual heptane content - 1470 ppm on a dry solids basis
- Plant yield - 92.8%
- Plant Btu recovery - 98.9%

During the production run, which was of 72 hours duration, there were two periods of downtime (15 hours total) due to the failure of the stripping circuit feed pump. No other operating problems were encountered.

Hiawatha Coal Testing Summary

In general, it was found throughout the Hiawatha coal testing, that the effects of most operating parameters had only small effects on the product ash content. However, the ground feedstock particle size distribution was found to have a significant effect on the product ash content. This relationship is illustrated in the following figure, which presents all of the Hiawatha coal testing complete plant test results in the form of plant product ash content as a function of the ground feedstock 80% passing (D_{80}) size, regardless of the plant operating conditions utilized. As can be seen from this data, while there is much scatter as a result of the parametric testing program, the relationship between the feedstock particle size distribution D_{80} and the plant product ash content (lb/MBtu) is clearly evident.



Product Ash Content vs Feedstock PSD D_{80} - Hiawatha Coal

Taggart Coal Parametric Testing Results

Parametric testing for the Taggart coal focused primarily on evaluation of the following:

- Grinding requirements
- High-shear agglomeration
- Low-shear agglomeration
- Steam stripping

A summary of parametric testing findings for the Taggart coal is as follows:

- The quality of the inversion exiting the high-shear circuit decreased as the high-shear impeller tip speed was reduced.
- Even under high-shear conditions resulting in very poor inversion, agglomerate growth in the low-shear vessel was still sufficient to afford good agglomerate and Btu recovery.
- The main effects of reducing high-shear energy input were:
  - A small decrease in product ash content
  - A decrease in tailings ash content
  - A small decrease in yield and Btu recovery
- At a coal feed rate of 3300 lb/hr, a feed grind size with a D_{80} of approximately 30 microns, and a 10 m/s high-shear impeller tip speed, the product ash target of 1 lb/MBtu was met at a tailings ash content of about 60%.
- There was no effect of low-shear solids concentration on product ash content.

- As the low-shear impeller tip speed was increased, a corresponding increase in product ash content was observed.
- Reductions in the steam stripping circuit solids concentration had no effect on the residual heptane concentration of the product, from either the first or second stage of steam stripping.
- Steam consumption was typically on the order of 1500 lb/ton coal.
- Product residual heptane concentrations were typically in the 4000 to 5000 ppm range on a dry solids basis.
- Tailings residual heptane concentrations were typically in the 3000 to 5000 ppm range on a dry solids basis.

Taggart Coal Production Run

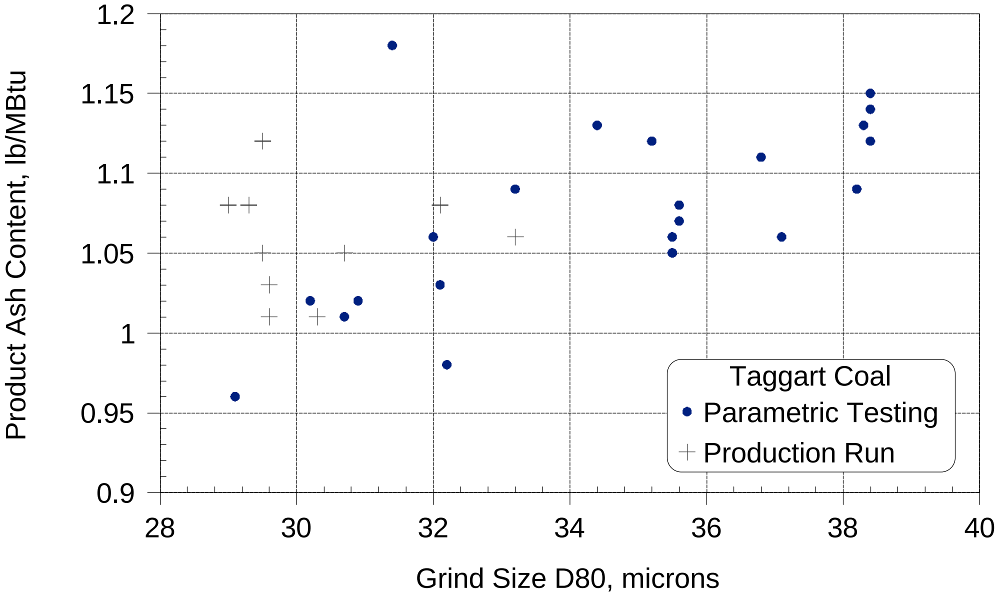
The Taggart coal production run was carried out during the week of May 19, 1997. The average product ash content was 1.06 lb/MBtu, slightly higher than the 1 lb/MBtu target. The range of product ash contents for the individual samples was from 1.01 to 1.12 lb/MBtu. The following is a summary list of average production run operating conditions and results:

- Dry coal feed rate - 3305 lb/hr
- Plant feed grind D_{80} - 30.3 microns
- Plant feed solids concentration - 10.02%
- Plant feed ash content - 3.64%
- Heptane dosage utilized - 39.2% on a dry ash free coal basis
- Total agglomeration (high- and low-shear) energy input - 16.1 kwhr/ton feed coal (6.2 kwhr/1000 gallon slurry)
- Screen spray water rate - 566 gallons/ton product
- Steam consumption - 1553 lb/ton dry product (1.8 lb/gallon slurry stripped)
- Plant product ash content - 1.06 lb/MBtu (1.59%)
- Plant product residual heptane content - 5115 ppm on a dry coal basis
- Plant tailings ash content - 63.0%
- Plant tailings residual heptane content - 4094 ppm on a dry solids basis
- Plant yield - 96.7%
- Plant Btu recovery - 99.2%

During the production run, which was of 72 hours duration, there was one period of downtime (2 hours) due to the failure of the stripping circuit feed pump. No other major operating problems were encountered.

Taggart Coal Testing Summary

In general, it was found throughout the Taggart coal testing, that the effects of most operating parameters had only small effects on the product ash content. However, the ground feedstock particle size distribution was found to have a significant effect on the product ash content. This relationship is illustrated in the following figure which presents all of the Taggart coal testing results, in the form of plant product ash content as a function of the ground feedstock 80% passing (D_{80}) size, regardless of the plant operating conditions utilized.



Product Ash Content vs Feedstock PSD D_{80} - Taggart Coal

As compared to the similar figure presented previously for the Hiawatha coal, the relationship between feedstock grind size and product ash content is not as clear for the Taggart coal. This is attributed to two factors:

- The narrow range of grind size D_{80}s tested for the Taggart coal (29 to 39 microns) as compared to the Hiawatha coal (37 to 60 microns).
- The effect of high-shear energy input on Btu recovery for the Taggart coal, which resulted in a range of product ash contents at similar grind sizes.

As such, while there is much scatter in the data for the above reasons, the relationship between the feedstock particle size distribution D_{80} and the plant product ash content (lb/MBtu) is still evident.

Indiana VII Coal Parametric Testing Results

Parametric testing for the Indiana VII coal focused on the following:

- Grinding requirements
- High-shear agglomeration
- Low-shear agglomeration
- Vibrating screen
- Steam stripping

A summary of parametric testing findings for the Indiana VII coal is as follows:

- To achieve consistent asphalt flows to the high-shear circuit, the emulsion first had to be screened at 28-mesh to remove the large particles and then diluted to approximately 2 to 3%.
- Increasing the asphalt dosage to high shear improved the quality of inversion achieved, increased Btu recovery, and increased the tailings ash content.
- There was a small effect of asphalt dosage on product ash content, with higher asphalt dosages resulting in slightly higher product ash values.
- Decreasing the high-shear tip speed (energy input) reduced the quality of inversion achieved and decreased the tailings ash content.
- There was a clear effect of high-shear energy input on product ash content, with lower energy resulting in higher product ash values.
- Increasing the asphalt dosage and decreasing high-shear energy input simultaneously resulted in a higher product ash content.
- To achieve the lowest product ash content at a given grind size, the asphalt dosage should be minimized and sufficient energy used to achieve the formation of good agglomerates.
- Increasing the high-shear solids concentration resulted in more particle to particle contact at similar energy inputs, and therefore a better quality inversion.
- Higher low-shear solids concentrations had no detrimental effect on product ash content.
- No difficulties were encountered when operating the low-shear vessel at increased solids concentrations.
- There was no significant increase in product ash content due to higher low-shear impeller tip speeds.
- Operation of low shear at reduced (3 m/s) impeller tip speeds resulted in poor agglomerate growth indicating that the lower tip speed did not supply sufficient energy for consistent agglomerate growth.
- Contrary to previous testing results, there was no observed difference in the product ash content as a function of agglomerate size
- Higher screen spray water flow rates resulted in a small reduction in product ash content.

- There was no significant reduction in the final plant product residual heptane content at increased temperatures. However, the temperature increase resulted in a lower first stage stripping product residual heptane content.
- Product residual heptane concentrations were in the 3000 to 5000 ppm range on a dry solids basis.
- Tailings residual heptane concentrations were in the 300 to 1000 ppm range on a dry solids basis.
- A feedstock grind size D_{80} of approximately 20 microns was required to achieve the product ash target of 2 lb/MBtu.
- Btu recoveries were consistently greater than 99% with yields from 90 to 92%.
- Tailings ash contents were consistently in the 85 to 92% range.

Indiana VII Coal Production Run

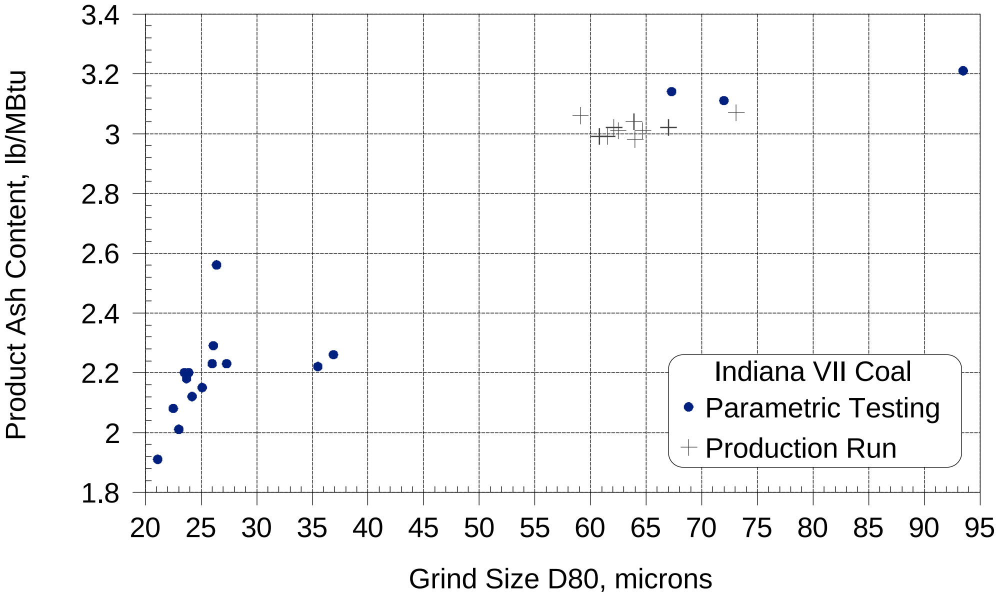
The Indiana VII coal production run was carried out during the week of July 28, 1997. For this production run, the product ash specification of 2 lb/MBtu was not targeted. Rather, the grind was coarsened and the feed rate reduced to allow continuous operation for the duration of the run. In particular, these goal modifications were made to accommodate the dewatering circuit which was not able to dewater the 20 micron D_{80} grind required to meet the 2 lb ash/MBtu product specification. As such, the average production run product ash content was 3.02 lb/MBtu. The range of product ash contents for the individual setpoints were from 2.98 to 3.08 lb/MBtu. The following is a summary list of average production run conditions and results:

- Dry coal feed rate - 3491 lb/hr
- Plant feed grind D_{80} - 63.9 microns
- Plant feed solids concentration - 12.48%
- Plant feed ash content - 9.8%
- Heptane concentration utilized - 34.8% on a dry ash free coal basis
- Asphalt concentration utilized - 5.4 lb/ton coal
- Total agglomeration (high and low shear) energy input - 36.6 kwhr/ton feed coal (17.9 kwhr/1000 gallon slurry)
- Screen spray water rate - 549 gallons/ton product
- Steam consumption - 1778 lb/ton dry product (2.1 lb/gallon slurry stripped)
- Plant product ash content - 3.02 lb/MBtu (4.19%)
- Plant product residual heptane content - 3967 ppm on a dry coal basis
- Plant Tailings ash content - 91.0%
- Plant tailings residual heptane content - 472 ppm on a dry solids basis
- Plant yield - 93.5%
- Plant Btu recovery - 100%

During the production run, which was of 72 hours duration, there were two periods of downtime (7 hours total) due to the failure of one tailings filter and one control valve. No other major operating problems were encountered.

Indiana VII Coal Testing Summary

In general, it was found throughout the Indiana VII coal testing, that the effects of most operating parameters had only small effects on the product ash content. However, the ground feedstock particle size distribution was found to have a significant effect on the product ash content. This relationship is illustrated in the following figure, which presents all of the Indiana VII coal complete plant testing results, in the form of plant product ash content as a function of the ground feedstock 80% passing (D_{80}) size, regardless of the plant operating conditions utilized.



Product Ash Content vs Feedstock PSD D_{80} - Indiana VII Coal

While there is some scatter in this data as a result of the parametric testing program, i.e., the completion of testing at a variety of plant operating conditions, the relationship between the feedstock particle size distribution D_{80} and the plant product ash content (lb/MBtu) is clearly evident.

Clean Coal Ash Properties

Samples of the feed coal and the clean coal from the extended production PDU runs with the Taggart, Indiana VII, and Hiawatha coals were submitted to Hazen Research Inc., of Golden, CO for determination of ash chemistry and fusion properties. It was found that the PDU selective agglomeration consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals and a small increase in the fusion temperatures of the ash in the Hiawatha coal. For example, the reducing atmosphere ash softening (spherical) temperatures were as follows before and after agglomeration cleaning in the PDU:

	Before	After
Taggart	2552°F	2396°F
Indiana VII	2479°F	2362°F
Hiawatha	2145°F	2181°F

Toxic Trace Elements Distribution

Huffman Laboratories analyzed crushed feed coal, ground agglomeration feed coal, clean coal and fine refuse samples from PDU runs on the Taggart, Indiana VII, and Hiawatha coals for toxic trace elements. The particular elements of interest were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine.

The same variations in trace element concentrations from coal to coal were seen for these samples as were seen for the set of samples from the bench-scale testing and from the PDU flotation. There were substantial reductions, over 25% on a heating value basis, in the residual concentrations of arsenic and manganese from the amounts in all three as-received test coals. The reduction in the concentrations of mercury and chlorine varied from coal to coal. The PDU agglomeration did not appear to have reduced the concentration of antimony, beryllium, cadmium, chromium, cobalt, nickel and selenium in any of these coals on a heating value basis.

The residual concentrations of all twelve trace elements in the Taggart and Indiana VII clean coals were especially lower than their concentrations in the their respective ROM parent coals on a heating value basis. On the other hand, only the arsenic and manganese concentrations were substantial reduced from the amounts in the as-received Hiawatha coal even though the latter coal had not been washed at the mine before marketing.

Lessons Learned

Based on the test work and operation of the PDU SA Module, the following general lessons were learned:

- Feed coal should be stored in a silo for protection from the elements. Coal left uncovered results in material handling problems due to freezing or sticking at transfer points. Also, surface oxidation of exposed coal may adversely affect agglomeration.

- Sumps should be designed with enough capacity that small changes in volume do not produce large fluctuations in level readings.
- Proposed ball mill charges should be reviewed for proper loading and ball size. PDU ball mills were initially improperly charged resulting in inefficient grinding and premature ball wear.
- Ball mill discharge magnets should be used for the removal of degraded grinding media.
- Multi-stage cycloning, instead of cycloning backed by top-size screen control, would allow for higher solids concentrations in the agglomeration feed. This would improve economics in both the grinding and agglomeration areas.
- All agitated tanks should be baffled to avoid vortexing, pump cavitation, and inaccurate level readings.
- Production of a ground feedstock with consistent solids concentration and size consist is important for producing agglomerates of consistent size. It was found that both of these parameters ultimately effect agglomerate growth and size.
- Production of consistently sized agglomerates from the low-shear unit operation is important for product ash and handling considerations.
- Low-shear reactors should provide only one mixing zone per vessel. The use of dual mixing zones results in difficult to control agglomerate growth.
- The separation of agglomerates from tailings via a vibrating screen should be performed in a downhill orientation to reduce agglomerate bed depth and product ash content.
- Froth skimming of carbonaceous material from the screen underflow should be carried out in a column-style vessel with the recovered material recycled to the high-shear unit operation.
- Recovered agglomerates should not be stored in an agitated tank prior to the steam stripping circuit due to their buoyancy and possible additional growth.
- Agglomerates should be fed to the stripping circuit via a diaphragm pump.
- Feed to the second stage of steam stripping should be via a positive displacement pump rather than a centrifugal pump to avoid high velocity flow reversal.
- The steam stripping circuit should include provisions for the removal of coal fines, carried within the vapor stream, prior to the gravity separation unit operation.
- During steam stripping, the process and instrument design must assure that the various pressure control loops required do not interact to produce operating instabilities.
- The scale-up methodology developed by the project team for the design of coal agglomeration agitation equipment is robust and reliable.
- Dewatering equipment should be designed specifically for its intended use to avoid low filtering capacity and unscheduled downtime.

- No deleterious effects were observed on the selective agglomeration process due to the use of recycled process water.

Conclusions

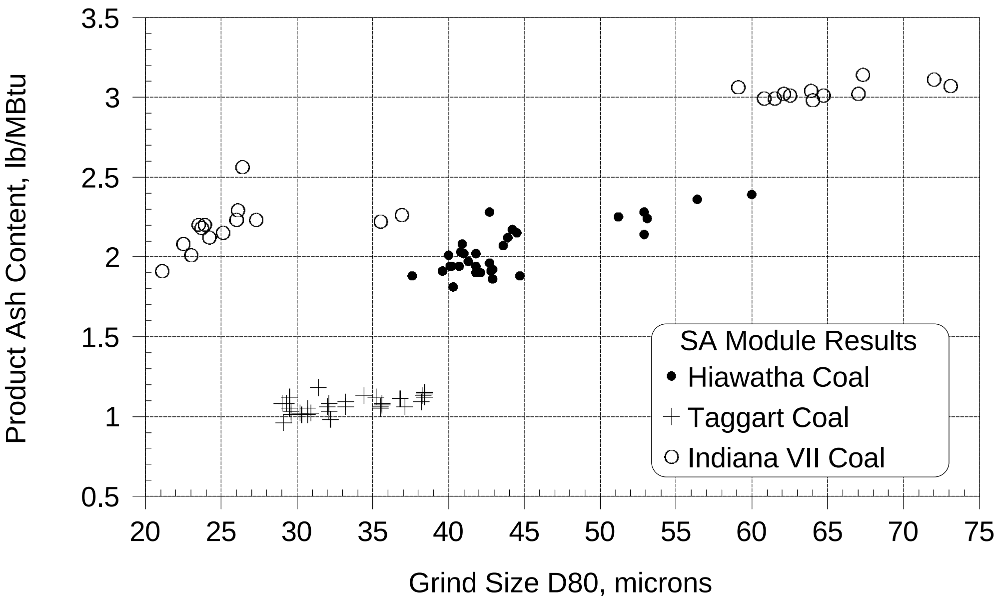
Program Success

The work and results related to this project should be considered successful. The 2 t/hr selective agglomeration module was operated from November, 1996 through July, 1997 processing over 800 tons of the Taggart, Indiana VII, and Hiawatha coals. Parametric testing was performed on each test coal followed by optimization test work and a round-the-clock production run. A substantial amount of each coal's clean product was transported to Penn State University for combustion testing. Overall, the Taggart coal was cleaned to produce a 1 lb ash/MBtu product while the Indiana VII and Hiawatha coals were cleaned to produce a 2 lb ash/MBtu product. Not only were the project goals achieved, the process equipment performed well in terms of reliability and control. A commercial plant cost study performed by Bechtel estimated the cost of production for premium quality coal water slurry fuel to be $2.42/MBtu which met the overall project goal.

Operation and Performance of the SA Module

The operation and performance of the SA Module was very successful. The well instrumented plant proved relatively simple to operate and maintain and was easily capable of producing premium quality fuel. Overall, the SA Module was able to reach steady-state conditions within approximately one hour and maintain production levels with little variance, assuming a consistent quality feedstock was used. Extended production runs indicated that the SA Module was a dependable and cost effective means of cleaning coal to high quality levels.

The following figure presents the SA module testing results for all three coals in the form of product ash content in lb/MBtu vs feedstock 80% passing size (D_{80}) in microns.



Selective Agglomeration Module Testing Results Summary

A summary of the PDU SA Module performance for the Taggart and Hiawatha production runs, and for an Indiana VII coal test in which the product ash target was met (product ash target was not a goal of Indiana VII coal production run) is as follows:

Coal	PSD D_{80}, microns	Ash, lb/MBtu	Sulfur, lb/MBtu	Yield, %	Btu Recovery, %
Taggart	30	1.06	0.67	96.7	>99
Indiana VII	20	1.91	0.35	91.3	>99
Hiawatha	42	1.93	0.4	92.8	98.9

Important Process Variables

Testing of the three coals in the PDU SA Module indicated that several process variables were important to proper operation. The most important variables and their effects on performance are discussed below:

- Feedstock PSD - The grind size of the slurry feedstock was found to have the greatest impact on product ash contents. In addition, it was found that a consistent feedstock PSD was important in the production of consistently sized agglomerates.
- High-shear agglomeration should be performed at a high solids concentration to minimize high-shear energy requirements. The practical limit for this solids loading, from an agglomeration view point is on the order of 15 to 20% solids. However, this limit is really determined by the grinding circuit capabilities.

- High-shear impeller tip speeds on the order of 10 to 15 m/s are required to insure the occurrence of phase inversion and subsequent agglomerate growth in low shear.
- High-shear residence time requirements are coal dependent but were typically found to be between 30 seconds for the Taggart coal and 120 seconds for the Indiana VII coal.
- High-shear energy requirements ranged from approximately 10 to 15 kwhr/ton coal for the Taggart and Hiawatha coals, to as high as 30 to 35 kwhr/ton for the Indiana VII coal.
- Low-shear agglomeration is best carried out in a single stage providing a residence time of about 2 to 3 minutes allowing agglomerate growth to 2 to 3 mm in size.
- The best compromise between low-shear growth control and product ash content was achieved at solids concentrations in the 7 to 10% range.
- Steam stripping should be performed in two stages. In the first stage, the bulk of the heptane is removed to produce a handleable product while in the second stage elevated temperatures are used to remove additional hydrocarbons.

Clean Coal Ash Properties

It was found that selective agglomeration consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals and a small increase in the fusion temperatures of the ash in the Hiawatha coal.

Toxic Trace Elements Distribution

The same variations in trace element concentrations from coal to coal were seen for coal samples cleaned in the PDU SA Module as were seen for the set of samples from the bench-scale testing and from the PDU Flotation Module. There were substantial reductions, over 25 percent on a heating value basis, in the residual concentrations of arsenic and manganese from the amounts in all three as-received test coals. The reduction in the concentrations of mercury and chlorine varied from coal to coal. Agglomeration did not appear to have reduced the concentration of antimony, beryllium, cadmium, chromium, cobalt, nickel and selenium in any these coals on a heating value basis.

The residual concentrations of all twelve trace elements in the Taggart and Indiana VII clean coals were especially lower than the concentrations in their respective ROM parent coals on a heating value basis. On the other hand, only the arsenic and manganese concentrations were substantially reduced from the amounts in the as-received Hiawatha coal even though the latter coal had not been washed at the mine before marketing.

Recommendations

Commercial Plant Design

The design of any commercial SA plant should be based on sound scale-up data. This data should be obtained from the operation of a plant that utilizes a single train of the largest practical agglomeration equipment that can be fabricated, estimated to be in the 20 to 25 t/hr range.

The maintenance of selective agglomeration equipment should also be considered thoroughly for a commercial plant design. In particular, the shaft seals for the agglomeration unit operations require significant attention and should be readily accessible.

In addition, design engineers should be mindful of the process control scheme developed for the selective agglomeration process. Because many different parameters affect the performance of the process, careful control of these parameters is necessary for consistent product yield and quality. In particular, the production of a consistent ground feedstock (both size and solids concentration) is considered critical. Beyond the feedstock control, proper metering of heptane and asphalt is required to maintain consistent reagent to coal ratios. In addition, good dilution water flow controls are important. As a result, instrumentation and control equipment are vital and highly recommended.

Future R&D Work

Each year, hundreds of thousands of recoverable tons of fine coal are lost to refuse disposal. This may be the result of poor performance in an existing preparation plant or even the lack of an economical fine coal cleaning process itself. It is recommended that the selective agglomeration process be investigated further for the recovery of these coal fines, rather than for the processing of an entire plant feedstock, as was done during the course of this project. This scenario would benefit the economics of the selective agglomeration process, particularly given the ability of the process to achieve very high energy recoveries under almost all possible operating conditions.

INTRODUCTION

The main goal of this project was 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's program to show that an ultra-clean coal-water slurry fuel (CWF) can be produced from selected United States coals and that this fuel could 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 as discussed below.

This multi-year cost-shared contract effort began on October 1, 1992, and is scheduled for completion by September 30, 1997. This report summarizes the findings of all the selective agglomeration test work completed during the course of this project, with the main emphasis on the results of the Subtask 9.3 Selective Agglomeration Operation and Clean Coal Production test work. Also included in this report, however, are brief summaries covering the Task 6 Selective Agglomeration Laboratory and Bench-Scale test work including Subtask 6.1 Agglomerating Agent Selection, Subtask 6.2 Grinding, Subtask 6.3 Process Optimization Research, Subtask 6.4 CWF Formulation Studies, and Subtask 6.5 Bench-Scale Testing and Process Scale-up.

SPECIFIC OBJECTIVES OF THE PROJECT

The three main objectives of this project are discussed below.

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

A secondary objective of the work was to develop a design base for near-term commercial applications of these advanced fine coal cleaning technologies. These applications were to 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 was also to include the auxiliary systems required to yield a shippable, marketable product such as a dry clean coal product.

A third objective of the work was 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. Twelve toxic trace elements were targeted. They were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium, and chlorine. The results of this work indicate 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. managed the project for Amax R&D (now part of Cyprus Amax Minerals Company) and also performed laboratory research and bench-scale testing. Entech Global was also responsible for the operation and evaluation of the 2 t/hr process development unit (PDU). Cyprus Amax Coal Company provided operating and business perspective, the site for the near-term testing, and some of the coals used in the program. Bechtel Corporation provided 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 provided research and operating experience in the column flotation area. Arcanum Corporation provided similar experience in the selective agglomeration area. Dr. Douglas Keller of Syracuse University served as a consultant in the area of selective agglomeration and Dr. John Dooher of Adelphi University served as a consultant in the area of coal-water-slurry formulation. Robert Reynouard of Control Technology, Inc. was retained as a consultant to help with electrical and instrumentation systems in the PDU. The Industrial Company (TIC) and Mech EL Contracting, Inc. (MEI) constructed the Advanced Flotation and Selective Agglomeration Modules of the PDU, respectively.

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

Phase I

Phase I encompassed 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 during Phase I included:

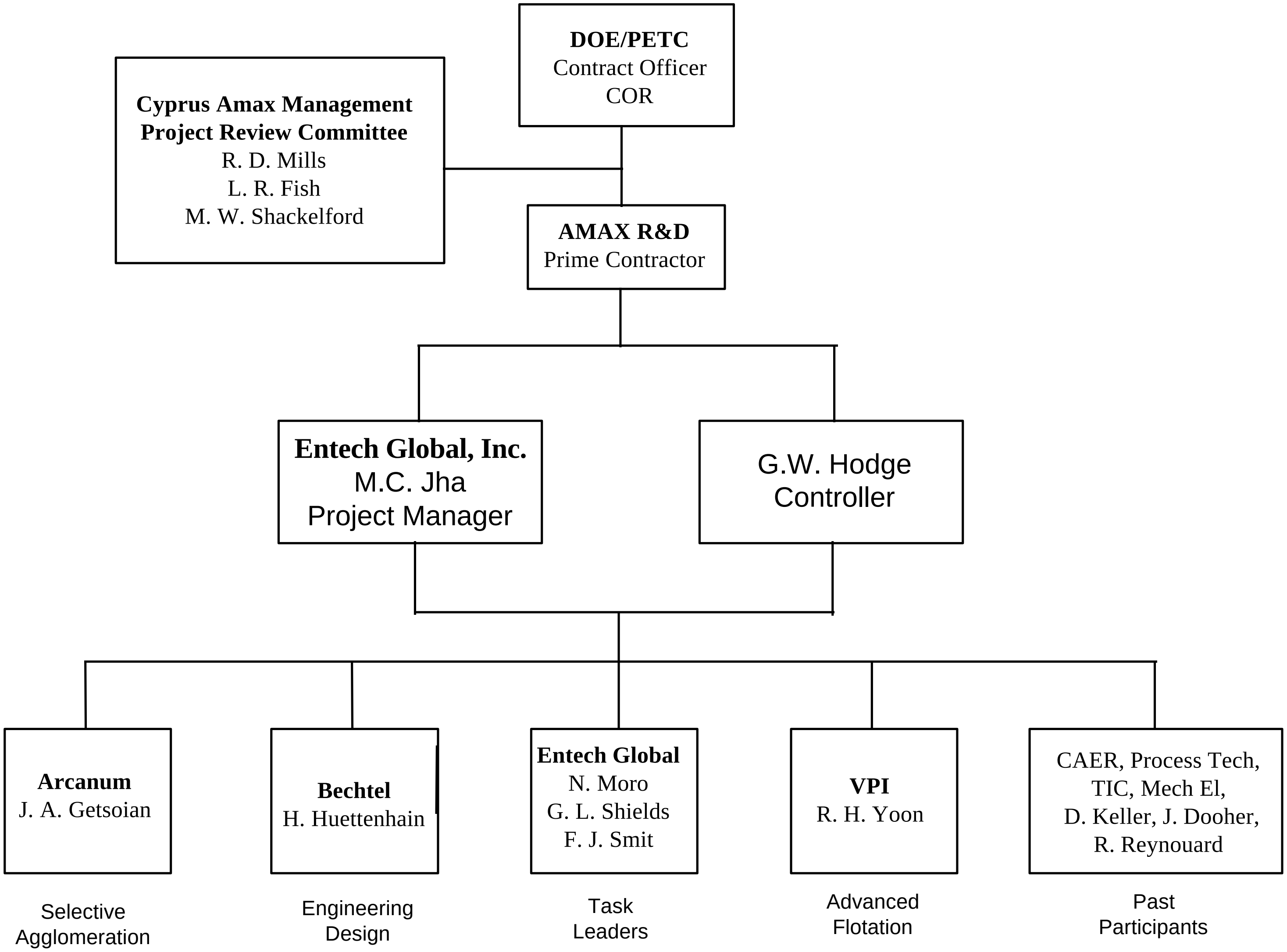
- Production of ultra-clean coal test lots by bench-scale column flotation and selective agglomeration for end-use testing
- Determination of toxic trace element distribution during production of these test lots
- Evaluation of the rheological properties of slurry fuels prepared from ultra-clean coals
- 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 covered the construction and operation of the 2 t/hr PDU. Phase II was for advanced column flotation while Phase III was for selective agglomeration. Process performance was optimized at the PDU-scale, and tonnage quantities of ultra-clean coal were produced by each process for each of the three test coals. The toxic trace element distribution was also determined during the production runs. The ultra-clean coals were delivered to a DOE designated contractor (Penn State) for end-use testing.

Phase IV

Phase IV activities included decommissioning of the PDU, restoration of the host site, and preparation of the final project report, which includes a conceptual design and cost estimate for commercial plants based on the two technologies.



Revised April 23, 1997

Figure 1. Project Management Organization Chart

{PRIVATE }Table 1. Outline of Work Breakdown Structure

Phase I. Engineering Analysis and Laboratory and Bench-Scale R&D

Task 1.	Project Planning
Subtask 1.1.	Project Work Plan
Subtask 1.2.	Project Work Plan Revisions
Task 2.	Coal Selection and Procurement
Subtask 2.1.	Coal Selection
Subtask 2.2.	Coal Procurement, Precleaning and Storage
Task 3.	Development of Near-Term Applications
Subtask 3.1.	Engineering Analyses
Subtask 3.2.	Engineering Development
Subtask 3.3	Dewatering Studies
Task 4.	Engineering Development of Advanced Froth Flotation for Premium Fuels
Subtask 4.1.	Grinding
Subtask 4.2.	Process Optimization Research
Subtask 4.3.	CWF Formulation Studies
Subtask 4.4.	Bench-Scale Testing and Process Scale-up
Subtask 4.5.	Conceptual Design of the PDU and Advanced Froth Flotation Module
Task 5.	Detailed Engineering Design of the PDU and Advanced Flotation Module
Task 6.	Selective Agglomeration Laboratory Research and Engineering Development for Premium Fuels
Subtask 6.1.	Agglomeration Agent Selection
Subtask 6.2.	Grinding
Subtask 6.3.	Process Optimization Research
Subtask 6.4.	CWF Formulation Studies
Subtask 6.5.	Bench-Scale Testing and Process Scale-up
Subtask 6.6.	Conceptual Design of the Selective Agglomeration Module
Task 7.	Detailed Engineering Design of the Selective Agglomeration Module

Phase II. PDU and Advanced Column Flotation Module Testing and Evaluation

Task 8.	PDU and Advanced Column Froth Flotation Module
Subtask 8.1.	Coal Selection and Procurement
Subtask 8.2.	Construction
Subtask 8.3.	PDU and Advanced Coal Cleaning Module Shakedown and Test Plan
Subtask 8.4.	PDU Operation and Clean Coal Production
Subtask 8.5.	Froth Flotation Topical Report

Phase III. Selective Agglomeration Module Testing and Evaluation

Task 9.	Selective Agglomeration Module
Subtask 9.1.	Construction
Subtask 9.2.	Selective Agglomeration Module Shakedown and Test Plan
Subtask 9.3.	Selective Agglomeration Module Operation and Clean Coal Production
Subtask 9.4.	Selective Agglomeration Topical Report

Phase IV. PDU Final Disposition

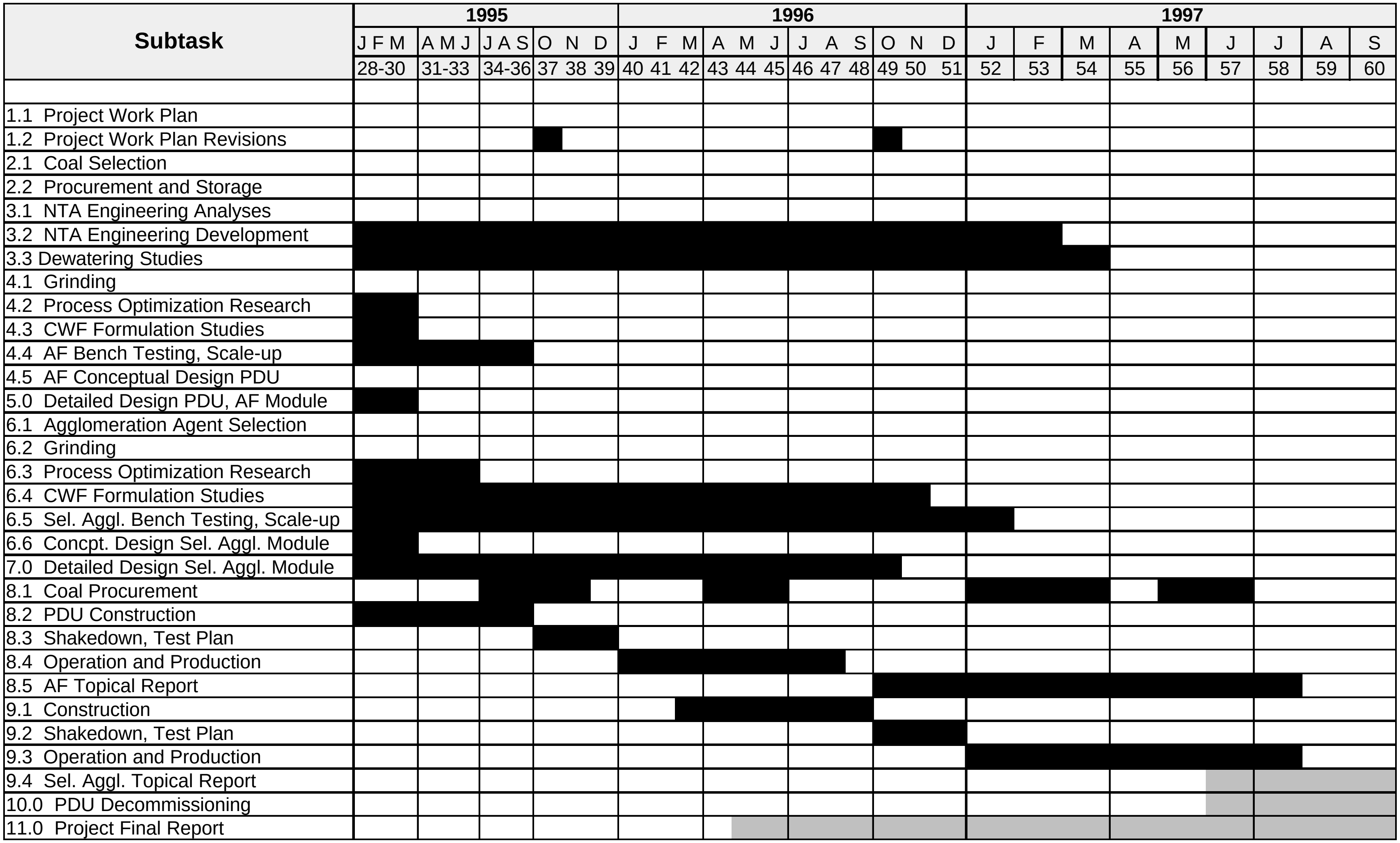
Task 10.	Disposition of the PDU
Task 11.	Project Final Report

Revised April 25, 1995

Subtask	1992			1993												1994											
	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F	M	A	M	J	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 Concpt. 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																											
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 August 25, 1997

Figure 2. Project Schedule



Subtask	1995				1996				1997								
	J F M	A M J	J A S	O N D	J F M	A M J	J A S	O N D	J	F	M	A	M	J	J	A	S
	28-30	31-33	34-36	37 38 39	40 41 42	43 44 45	46 47 48	49 50 51	52	53	54	55	56	57	58	59	60
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 Concpt. 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					■	■	■										
9.2 Shakedown, Test Plan								■									
9.3 Operation and Production									■	■	■	■	■	■	■		
9.4 Sel. Aggl. Topical Report														▨	▨	▨	▨
10.0 PDU Decommissioning														▨	▨	▨	▨
11.0 Project Final Report						▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨	▨

Revised August 25, 1997

Figure 2. Project Schedule (Cont'd)

SELECTIVE AGGLOMERATION PROCESS DESCRIPTION

Selective agglomeration is a coal cleaning process based on the differences in surface properties of the coal and its associated mineral impurities. Generally, coal particles are hydrophobic or repel water, while the mineral impurities associated with coal are hydrophilic or easily wetted by water. As such, when a hydrocarbon based agglomerating agent (agglomerant or bridging liquid), is added to a finely divided coal water slurry and agitated, the carbon containing coal particles are coated by the agglomerant while the mineral matter remains dispersed in the water phase.

The bridging liquid utilized during selective agglomeration can range from "heavy" organic liquids like fuel oil No. 2, to "light" hydrocarbons such as heptane. The particular type of agglomerant used depends on a number of factors, but is primarily influenced by the feed coal characteristics, process economics, and the required product quality [1]. Depending on the type and quantity of bridging liquid used, the agglomerant is either allowed to remain with the product, or recovered and recycled back to the process. Generally, when using heavier agglomerants such as fuel oil, the quantity used is minimized and allowed to remain with the product. If, however, a light hydrocarbon such as heptane is used, the quantity used is not so critical since it must be recovered from the product and recycled to the process for health, safety, environmental, and economic reasons.

Based on the results of Subtask 6.1 Agglomerant Agent Selection [1] and Subtask 6.3 Process Optimization Research [2], heptane was selected for evaluation during subsequent Subtask 6.5 and Subtask 9.3 testing.

HIGH-SHEAR AGGLOMERATION

During high-shear agglomeration a mixture of water, coal, and heptane is vigorously agitated, at impeller tip speeds in the 10 to 18 m/s range, such that the heptane disperses and makes contact with the coal particles in the slurry. Throughout this agitation, hydrophobic coal particles are attracted to the heptane phase, while the hydrophilic mineral matter is repelled from the heptane and attracted to the water phase. Given the proper proportions, with continued mixing, the heptane coated coal particles coalesce to form microagglomerates, while the mineral impurities remain dispersed in the water phase.

It is important that the high-shear unit operation provide mechanical agitation of sufficient intensity and duration to insure the formation of these microagglomerates, also described as a “phase inversion.” During this inversion, two distinct phases are formed, a coal/agglomerant phase (microagglomerates), and a water phase containing the dispersed mineral matter.

The shear rate used during high shear must be sufficiently high to guarantee heptane dispersion to a very small droplet size. Once the heptane is dispersed, the high-shear residence time and total power input must then be sufficient to insure microagglomerate formation, i.e., provide enough energy for sufficient heptane to coal and particle to particle contacts.

It should be noted that in some cases, for aged (oxidized) or lower rank coals, an aid may be required to enhance agglomeration. Typically this agent would be asphalt, which has been shown to enhance agglomeration kinetics when present during the high-shear unit operation [2].

LOW-SHEAR AGGLOMERATION

Following high-shear agglomeration, the microagglomerates are subjected to a low-shear agglomeration step. During low shear, the slurry is mixed at a shear rate significantly less than that used during high shear, typically at impeller tip speeds in the 3 to 5 m/s range, to promote agglomerate growth. If an agglomerated product of a particular size and/or strength is required, the low-shear heptane dosage, shear rate, solids concentration, and/or residence time may be tailored to generate the appropriate product.

For this project, the final process product was in the form of a highly-loaded slurry. As such, the formation of "large" agglomerates, say greater than 2 to 3 millimeters, with sufficient strength to withstand handling without degradation was not required. Therefore, the primary goal of the low-shear unit operation was to provide a product which could be easily recovered, washed, and dewatered on a screen.

Ideally, product agglomerate size should be just large enough to insure product recovery without the incorporation of tailings into the product, i.e., the screen size used should be only marginally larger than the topsize of the feed coal. This scenario ensures low power consumption during low shear. In practice, however, it is generally found that the larger the product agglomerates are, the easier it is to reject the tailings mineral matter during screening. This is due to the presence of larger voids within the agglomerate bed as agglomerate size increases. Subsequently, better drainage of the mineral-matter bearing process water occurs. Since agglomerate growth is carried out under low shear rates, the additional power consumption required to generate slightly larger agglomerates is small.

AGGLOMERATE RECOVERY

Once agglomerates are formed during low shear they must be physically recovered to the product. The goal of this unit operation is to achieve high product recovery and a good separation between the product agglomerates and the mineral-matter bearing process water, i.e., minimize coal losses to the process tailings (screen underflow) while minimizing the contamination of both the product with mineral matter, and the process tailings with heptane bearing carbonaceous material.

Primary agglomerate recovery was carried out on a vibrating screen where the agglomerates were dewatered, reporting to the screen oversize. The screen was fitted with water sprays for product rinsing. The tailings or mineral-matter bearing process water reported to the screen underflow.

Secondary agglomerate recovery, from the vibrating screen underflow, was achieved in a froth skimming device in which any floating carbonaceous material was recovered to the process product stream. If required to insure that all heptane bearing material was recovered, dispersion of nitrogen into the froth skimmer can be used to help float any additional carbonaceous material.

HEPTANE RECOVERY AND RECYCLE

Once agglomerates are recovered by screening and froth skimming, the heptane must be removed from the product. Heptane recovery from the agglomerated product was accomplished by direct contact steam stripping in two stages. During this process, heat provided by steam evaporated an azeotropic mixture of heptane and water. From this vapor, the two liquids (heptane and water) were condensed, cooled, separated, and recycled to the process.

RESULTS AND DISCUSSION

This portion of the selective agglomeration topical report presents the results and discussion of all the selective agglomeration test work completed throughout the course of this project. The first section presents a brief summary of the laboratory and bench-scale test work including:

- Subtask 2.1 - Coal Selection
- Subtask 6.1 - Agglomerating Agent Selection
- Subtask 6.2 - Grinding
- Subtask 6.3 - Process Optimization Research
- Subtask 6.4 - CWF Formulation Studies
- Subtask 6.5 - Bench-Scale Testing

This is followed by a summary of the Task 9 Process Development Unit (PDU) Selective Agglomeration (SA) Module 2 t/hr pilot-scale work. This section covers the following:

- Subtask 9.1 - Construction
- Subtask 9.2 - SA Module Shakedown and Test Plan
- Subtask 9.3 - SA Module Operation and Clean Coal Production

LABORATORY AND BENCH-SCALE WORK

The following sections of this report present brief summaries of the laboratory and bench-scale selective agglomeration test work carried out during this project.

Subtask 2.1 - Coal Selection

Successful completion of the project objectives by both the froth flotation and the selective agglomeration processes was dependent on the selection of suitable source coals. Due to the widely varying quality and economic factors of United States coals, many could not be considered as a feedstock for this project. Accordingly, guidelines were established to evaluate a number of candidate coals, and select six coals for use in the project. Overall, five bituminous coals and one low-rank coal were to be selected. Details of the selection procedure are provided in the Subtask 2.1 topical report [3] and only a brief summary is presented here. Guidelines included in the contract Statement of Work suggested the following specifications for coal selection:

- Source Coal Properties
    - Organic sulfur should be less than 258g/GJ (dry basis), or approximately 0.88% for bituminous coals and 0.75% for low-rank coals

    - Ash minerals and pyrite must be sufficiently liberated by practical comminution methods
- Economic Factors - Coal Acquisition
    - Selected coals must be obtained from actively mined seams with reserves in excess of 300 million tons
    - Sufficient quantities must be available for purchase from the same source to meet the needs of the project
    - The cost of the coal should be less than \$1.18/GJ (\$1.25/MBtu) or approximately \$30/ton
- Economic Factors - Fuel Preparation
    - Because variations in coal quality may affect the preparation of premium CWF, potential coals should have the following characteristics:
        1. Low ash content
        2. Low total sulfur content
        3. Low organic sulfur content
        4. Liberation of ash bearing minerals and pyrite at coarse sizes
        5. Low inherent moisture
        6. High Hardgrove Grindability Index
        7. High hydrophobicity

In addition to these parameters, geographic diversity was also considered with at least one coal from each US coal mining region (eastern, midwestern, and western). The initial screening of coals from the Keystone Coal Mining Directory and the Amax Database generated a list of 32 candidate coals, which after preliminary evaluation was narrowed down to the 17 coals listed in Table 2.

These candidate coals were then subjected to the following evaluations:

- Proximate Analysis - Moisture, Ash, Volatile Matter, and Fixed Carbon
- Sulfur Forms
- Heating Value - Btu/lb
- Equilibrium Moisture
- Hardgrove Grindability
- Coarse Coal Liberation - float/sink at SG of 1.6 and 1.9 on 100Mx0 sample
- Fine Coal Liberation - float/sink at SG of 1.6 and 1.9 on 325Mx0 sample
- Supplemental Amenability Testing - Flotation and Agglomeration testing on 20µm x 0 sample

A coal selection matrix was then established for ranking each coal according to the previously mentioned parameters and test evaluations. The selection matrix is presented in detail in the Subtask 2.1 Topical Report [3]. They are also included in a published paper [4].

Table 2. Candidate Coals for Preparation of Premium Fuels

Coal Seam	State	County	Mine	Operator
Upper Freeport	PA	Indiana	Helen	Helen Mining
Stockton / Mercer	WV	Kanawa	130 Mine	Amax - Cannelton
Winifrede	WV	Boone	Sandlick	Amax - Cannelton
Taggart	VA	Wise	Wentz	Westmoreland
Hazard 4A / 5A	KY	Knott	KY Prince	Roaring Creek
Elkhorn No. 3	KY	Pike	Chapperal	Costain
No. 2 Gas	WV	Wyoming	N/A	N/A
No. 2 Gas	WV	Boone	N/A	N/A
Indiana VII	IN	Sullivan	Minnehaha	Amax - Midwest
Illinois No. 5	IL	Wabash	Wabash	Amax - Midwest
Maxwell	CO	Las Animas	Golden Eagle	Basin Resources
O'Conner	UT	Carbon	Skyline	Utah Fuels
Sunnyside	UT	Carbon	Sunnyside	Sunnyside
Wyodak	WY	Campbell	Belle Ayr	Amax - West
Dietz	MT	Big Horn	Spring Creek	Nerco
Rosebud	MT	Rosebud	Rosebud	Western Energy
Lower Smith	WY	Campbell	Eagle Butte	Amax - West

As a result of this evaluation, the following five bituminous (all of which had the characteristics required for successful production of premium fuels) and one low-rank coal were selected for testing during Phase I of the project:

- Taggart Coal - This was the highest ranking coal, which also performed very well in amenability testing.
- Sunnyside Coal - This coal compiled a very high score and performed very well in amenability testing.
- Indiana VII Coal - This coal contained less sulfur than most midwestern coals. Though it scored low, the coal was readily available for test work since the Minnehaha mine was owned by Amax Coal.
- Winifrede Coal - Winifrede coal is very typical of the coal produced in West Virginia. It was also readily available since the source mine was owned by Amax Coal.
- Elkhorn No. 3 Coal - This coal, which received a high score, is representative of the coal produced in eastern Kentucky.
- Dietz Coal - Dietz coal was recommended as the single low-rank selection. Though it compiled a low score, it responded better than other low-rank coals to amenability testing.

These six test coals were then used for agglomerating agent selection (Subtask 6.1), laboratory mineral liberation studies (Subtask 6.2), and laboratory selective agglomeration process optimization research (Subtask 6.3).

Subtask 6.1 - Agglomerating Agent Selection

The objective of Subtask 6.1 was to select the appropriate agglomerating agents to be used for testing under Subtask 6.3 Process Optimization Research, and other subsequent Subtasks. Detailed information covering this work can be found in the Subtask 6.1 Agglomerating Agent Selection Topical Report [1].

Of the four agglomerants to be selected, at least two were to be "light" hydrocarbons which would require a recovery system for agglomerant reuse. As such, the other two agglomerants could be "heavy" hydrocarbons which would be less expensive and consequently remain with the product.

It was determined, however, that the heavy hydrocarbons would not be capable of meeting the project objectives for the following reasons:

- Their use would prevent the project low-ash target specification to be met
- Their presence in the final agglomerated product (since they would not be recovered and recycled to the process) would make coal-water-slurry fuel (CWF) formulation difficult.

As such, it was determined that only two "light " hydrocarbons would be selected during Subtask 6.1. These selections would be based on the following general criteria:

- Potential carbon recovery and mineral-matter rejection
- Ease of agglomerant recovery for reuse
- Agglomerant availability and cost
- Health and environmental issues
- Effect of residual agglomerant on CWF formulation

The "light" hydrocarbons, along with their class and formula, evaluated during the Subtask 6.1 selection process included:

- n-Pentane, Paraffin (C_5H_{12})
- Cyclohexane, Cyclo paraffin (C_6H_{12})
- n-Hexane, Paraffin (C_6H_{14})
- n-Heptane, Paraffin (C_7H_{16})
- n-Octane, Paraffin (C_8H_{18})
- n-Nonane, Paraffin ($C_{10}H_{22}$)
- Toluene, Aromatic (C_7H_8)

- p-Xylene, Aromatic (C_8H_{10})

The various selection criteria, utilized to develop a point-based ranking system for the evaluation of the "light' hydrocarbons listed above included:

- Ash rejection
- Pyrite rejection
- Toxicity
- Flash point
- Cost
- Purge loss rate
- Water solubility
- Azeotrope composition
- Azeotrope boiling point
- Viscosity

Based on these criteria, the two "light" hydrocarbons with the highest rankings were n-heptane and n-pentane, scoring 79 and 76 points, respectively, out of a maximum possible 100 points. As such, these two agglomerants, or bridging liquids, were selected for evaluation during the Subtask 6.3 Process Optimization Research test work.

Subtask 6.2 - Grinding

During initial selective agglomeration (SA) test work, an evaluation of the grinding requirements (liberation) necessary to achieve the project goal of 2 lb ash/MBtu for the selected project coals was carried out under Subtask 6.2. Detailed information covering this work can be found in the Subtask 6.2 Grinding topical report [5]. The main objectives of Subtask 6.2 were to

- Determine the grind size required to achieve the mineral liberation needed to achieve the target CWF fuel specifications.
- Determine the grinding circuit configuration that best met the needs of the 2 t/hr process development unit (PDU) selective agglomeration (SA) module, while allowing scale-up to a commercial premium fuel production plant.
- Determine the design and operating parameters of the PDU SA module grinding circuit.
- Prepare ground slurries for the Subtask 6.3 Selective Agglomeration Process Optimization Research and Subtask 6.4 CWF Formulation Studies subtasks.
- Determine the capacities of the available Amax R&D grinding equipment as they pertain to the production of ground slurry feedstock for Subtask 6.5 Bench-scale Testing and Process Scale-up.

The six project coals selected during Subtask 2.1 were generally stage-ground in the pilot-plant's 4-foot x 4-foot ball mill and a 40-liter stirred-ball mill to determine unit capacities and provide ground material for evaluation of pyrite and ash mineral liberation. Open-circuit, closed-circuit, and selective grinding configurations were compared during this investigation, and grinding rates and particle size distributions (PSD) recorded for subsequent use during the 2 t/hr PDU grinding circuit design.

Laboratory agglomeration tests were carried out on the various ground products to quantify the liberation of ash and sulfur for each grind evaluated. It was determined, through liberation testing, that the D_{80}s (80% passing sizes) shown in Table 3 were required to insure the production of a clean coal containing less than 2 lb ash/MBtu (1 lb ash/MBtu in the case of the Taggart coal).

Table 3. Grind Sizes Required for Sufficient Liberation of Test Coals

Test Coal	D_{80} (microns)
Taggart	45
Indiana VII	20
Sunnyside	45
Winifrede	11
Elkhorn No. 3	45
Dietz	20

Overall, a closed-circuit grinding configuration was found to be more efficient than an open-circuit configuration, in that it provided greater capacity for a given grind size. This benefit was most evident when grinding to very fine particle sizes, such as those needed for the Indiana VII, Winifrede, and Dietz coals. The size classification method found to work best in these closed-circuit grinding configuration evaluations was dependent on the target product size. A solid bowl centrifuge was used for size separations less than 30 microns while a SWECO vibrating screen was used for size separations greater than 30 microns.

It was found that pyrite tended to accumulate in the circulating load of oversize coal from the classifying centrifuge, As such, one test was completed with the Indiana VII coal in which a spiral classifier was used to treat the circulating load to remove pyrite directly from the grinding circuit. While this was found to reject 25% of the pyrite in the Indiana VII coal, the overall impact on the ground feedstock sulfur content was minimal due to the small amount of pyrite present. Since none of the other test coals contained significantly more pyrite than the Indiana VII, no additional work was carried out utilizing this selective grinding configuration.

Other Subtask 6.2 findings of interest were that:

- Grinding to a D_{80} of 20 microns or coarser could be accomplished as efficiently in the 4-foot x 4-foot ball mill as in the 40-liter stirred-ball mill.

- Crushing to a 1/4-inch topsize (rather than a 1/2-inch topsize) prior to grinding had little impact on the circuit capacity
- The circuit capacity was doubled by utilization of a closed-circuit grinding operation, as compared to an open-circuit operation.

Based on the complete set of continuous pilot-scale grinding tests completed under Subtask 6.2, Table 4 presents estimated grinding power requirements for size reduction of the six project coals to their anticipated required target grind sizes.

Table 4. Estimated Grinding Energy Requirements for Test Coals

Test Coal	HGI	Target D_{80} (microns)	kW / tph	hp / stph
Taggart	52	.45	96	116
Indiana VII	55	20	153	185
Sunnyside	54	45	91	110
Winifrede	47	11	341	413
Elkhorn #3	46	45	118	143
Dietz	41	20	203	247

The data in Table 4 indicates that grinding power requirements varied greatly from coal to coal. These differences are attributed primarily to the varying target sizes (D_{80}s) required for mineral and pyrite liberation. The differing Hardgrove Grindability Indices (HGI) of the test coals were of lesser importance.

The grinding performance and capacity of the existing Amax R&D 4-foot x 4-foot ball mill and stirred-ball mill were considered to be adequate for the completion of the Subtask 6.5 Bench-scale Testing and Process Scale-up work.

Subtask 6.3 - Process Optimization Research

The main objectives of the Subtask 6.3 test work were to

- Optimize, by laboratory-scale research and testing, the selective agglomeration process to best meet the project clean coal quality and heating value recovery specifications
- Compare the performance of the two “light” hydrocarbon agglomerating agents and recommend one for further testing at the bench-scale
- Compare the performance of an innovative reactor design that combined the high- and low-shear mixing zones in a single unit, with the conventional agglomeration design in which the high-shear and low-shear unit operations are carried out separately.

This laboratory-scale testing was carried out at both Arcanum and Amax R&D. Dr. Keller of Syracuse University reviewed the work on an ongoing basis and aided in the interpretation of the results and recommended follow-up tests. Detailed results for this test work can be found in the Subtask 6.3 Process Optimization Research Topical Report [2].

Both n-pentane (C_5H_{12}) and n-heptane (C_7H_{16}) were employed as bridging liquids for most of the work as recommended by the Subtask 6.1 Agglomerating Agent Selection Topical Report [1]. Usage cost was an important reason for favoring these light volatile hydrocarbons over heavier hydrocarbons such as fuel oil and kerosene since the light hydrocarbons could be conveniently stripped from the product and reused. The main difference between n-pentane and n-heptane was their boiling points, 97º and 209º F, respectively.

The selective agglomeration process, as tested during this project involved three primary unit operations, high-shear mixing, low-shear mixing, and screening. Subtask 6.3 testing focused on the high- and low-shear mixing steps via the following three types of laboratory-scale agglomeration tests:

1. Waring blender batch tests to quickly assess phase inversion times, the behavior of differing coals, particle size distributions, bridging liquids, pretreatments, and activators thereby providing a standard for gauging the performance of the continuous test apparatus.
2. Continuous testing in a single-stage unitized reactor system (combined high- and low-shear) that had the capacity to agglomerate about 50 grams/min (about 5 lb/hour) of coal.
3. Conventional two-stage agglomeration testing (separate high- and low-shear mixing steps) carried out at Arcanum in a test unit with a similar capacity (about 50 grams/min or 5 lb/hour) as the single-stage unitized reactor.

Unitized Reactor Agglomeration Testing

The basic procedure used to optimize laboratory agglomeration of the test coals was to first perform a batch test on a slurry and then perform a series of parametric tests at varying flowrates and rotor speeds in the unitized reactor. Further optimization testing was then based on the results of the initial batch test and these parametric tests. This matrix basically defined the effects of retention time and mixing intensity on agglomeration. Pentane bridging liquid, without any activator, was used for the baseline comparisons. Activator additions were found to be essential, though, for agglomerating the Indiana VII and Dietz coals.

The effects of operating parameters, using pentane as the agglomerant, were best shown by results for the four high volatile A bituminous coals (Elkhorn No. 3, Taggart, Winifrede, and Sunnyside) since these coals all agglomerated quite rapidly. The effects of changing operating conditions were less clear with the Indiana VII and Dietz coals because of the need for an activator to assist agglomeration.

The parametric testing provided quantitative data on the relationship between feed rate and rotor speed on the performance of the unitized agglomerator. The best region for recovery (>90% Btu recovery, usually >98%) appeared to be between 3200 and 4800 rpm and when feeding between 200 ml and 800 ml of slurry per minute. Sufficient agglomeration for capture on the screen failed to occur outside the preferred range. Within the preferred operating range, a regression analysis suggested that coarser particle size distributions, slower feed rates and faster rotor speeds favored increased Btu recovery by small amounts. The 2 lb/MBtu ash specification (1 lb/MBtu for the Taggart coal) was met in almost every instance where there was sufficient agglomeration to capture the clean coal on the 100-mesh sieve.

During initial testing, it was found that excessive amounts of pentane formed sticky globs or clusters of agglomerated coal in the unitized reactor. On the other hand if insufficient pentane were added, the agglomerates (if any formed at all) would pass through the screen completely. Satisfactory operation occurred only in a narrow range of pentane additions and was generally a function of PSD as illustrated in Table 5.

Table 5. Pentane Requirements for Acceptable Agglomeration

Coal	Feedstock D_{80}, microns	Pentane Ratio, g/g coal
Taggart	103	0.18
Sunnyside	50	0.21 - 0.26
Elkhorn No. 3	45	0.25 - 0.31
Indiana VII	22	0.26 - 0.32
Winifrede	11	0.29 - 0.34

There was no clear picture to suggest any effect of the pentane ratio on ash rejection. Regression studies on the data for the Sunnyside, Elkhorn No. 3, and Winifrede coals indicated that lower percent solids, lower feed rates and slower rotor speeds resulted in less residual ash in the clean coal.

As testing progressed, the n-pentane was replaced with “pure” grade n-heptane for testing with the Taggart, Sunnyside, Elkhorn No. 3, and Sunnyside coals. Approximately the same amount of heptane was needed for agglomeration as was needed of the pentane, as shown in Table 6.

Table 6. Heptane Requirements for Acceptable Agglomeration

Coal	Feedstock D_{80}, microns	Heptane Ratio, g/g coal
Taggart	103	0.21
Sunnyside	50	0.22 - 0.26
Elkhorn No. 3	45	0.24 - 0.32
Indiana VII	22	0.28 - 0.36

The Indiana VII coal did not agglomerate well with pentane in the unitized reactor unless a considerable amount (30 to 60 lb/t dry coal) of 2-ethylhexanol activator was added. It worked best to inject the ethylhexanol directly into the bridging liquid before the liquid mixed with the coal slurry in the high-shear mixing zone. Acidification of the Indiana VII slurry had little if any effect upon the amount of activator required for agglomeration.

The use of asphalt was also investigated as an activator for agglomeration of the Indiana VII coal with pentane. Only 15 lb asphalt/ton coal was needed for agglomeration instead of the 30 to 60 lb of ethylhexanol. Furthermore, the pellets were drier and lower in ash when asphalt was used in place of the ethylhexanol. The improvement in ash rejection appeared to be related to the smaller amount of moisture remaining in the pellets prepared with asphalt.

A similar comparison was made between ethylhexanol and asphalt activation when agglomerating Indiana VII coal with heptane bridging liquid. These tests show significant superiority for the asphalt. The capacity of the agglomeration system was doubled, and the activator dosage was less than 20 lb/t compared to the 27 lb/t or more required with ethylhexanol. Clean coal ash contents were comparable at comparable heating value recoveries. The pellets formed with asphalt activator also were drier and appeared to be stronger.

Sulfur analyses were obtained for the clean coals from selected agglomeration tests. The less than 0.6 lb sulfur per million Btu goal was met for the Taggart, Indiana VII, and Sunnyside coals. There was a discernible sulfur reduction on a lb/MBtu basis only in the case of the Indiana VII coal. The sulfur in all of the feed coals was predominately in the organic form with very little pyrite present so the poor sulfur rejection was expected.

Increasing the percent solids in the coal slurry feeding agglomeration would have a beneficial impact upon operating and capital costs since smaller volumetric flow rates allow use of smaller mixers and vessels. At the time the maximum pulp density of the feed slurry was set at about 15 percent solids by grinding-circuit requirements, but installation of a thickener ahead of the high-shear agglomeration was considered for the PDU in order to produce a thicker slurry for agglomeration. To be cost-effective, though, any thickener would have to be small and require the addition of polymeric flocculants to the slurry in order to avoid overflowing coal from the tank. Comparison tests were made in the unitized reactor to determine whether such polymeric flocculants would effect agglomeration. It was found that slightly more coal was left in the refuse when the flocculants were added to the slurry before agglomeration. The loss in clean coal recovery was particularly noticeable at the higher feed rates, but the reduction in coal recovery was not accompanied by any reduction in the amount of ash remaining in the clean coal.

Work during liberation studies under Subtask 6.2 indicated that the Dietz subbituminous coal required special treatment before and during high- and low-shear mixing in order to achieve satisfactory agglomeration. Specifically, pre-acidification of the slurry to pH 3 was required as was the addition of a considerable amount of dissolved asphalt in the bridging liquid. It was also confirmed during initial testing for Subtask 6.3 that longer periods of high-shear mixing were required for inversion (up to 10 minutes compared to a minute or less with bituminous coals). The low pH was necessary during the oiling step; the separation was not as good when pH 3 acidified slurry was filtered and repulped before agglomeration. Heptane bridging liquid seemed to perform as well as or better than pentane, kerosene, and diesel fuel.

Additional batch agglomeration tests were carried out on the Dietz coal to define conditions for good ash rejection. The kind of acid employed had little impact upon agglomeration, although inversion may have been delayed somewhat as pH increased. At least 1% asphalt, dissolved in the heptane, was needed for good ash rejection. Performance deteriorated rapidly when the asphalt concentration was reduced below 1% (about 8 lb/ton dry coal).

The use of chelating agents, specifically EDTA (ethylene diamine tetraacetic acid) and sodium metaphosphate, was also examined,. The EDTA and metaphosphate additions were less effective than acidification for initiating agglomeration.

Two-Stage Agglomeration Testing

Two-stage continuous agglomeration tests were performed at Arcanum using the Indiana VII and Winifrede coals. The results showed that the minus 325-mesh Indiana VII and Winifrede coals could be agglomerated to less than 2 lb ash/MBtu with very high heating value recovery. A small amount of asphalt, about 0.5% by weight of the clean product, was required to achieve agglomeration of the Indiana VII coal at practical rates. Winifrede coal agglomerated adequately with less than 0.25% asphalt and may not have needed any at all. High-shear energy and shear rate values were obtained during this work for use during design of the bench-scale and PDU test units.

Results Summary

Conditions were found where each of the test coals responded well to selective agglomeration. The preferred operating conditions for the unitized reactor, and the responses of the project coals, are summarized in Table 7.

Table 7. Unitized Reactor Selective Agglomeration Testing Results Summary

	Taggart	Indiana VII	Sunnyside	Elkhorn No. 3	Winifrede	Dietz
Clean Coal:						
Ash, lb/MBtu	0.99	1.62	1.80	1.83	1.75	1.9-2.5
Btu Recovery, %	99.8	98.6	99.4	99.4	94.2	93-98
Feed Slurry:						
Solids, %	10-13	10	7-10	7-13	7	7
Heptane Ratio, g/g	0.21	0.28	0.22	0.25	0.35	0.5
Asphalt, lb/t	none	16	none	none	none	25-80
Acidity	none	none	none	none	none	pH 3-4
High-Shear Mixing:						
Tip Speed, m/s	15.5	15.5	15.5	17.5	15.5	15.5
Retention, minutes	0.10	0.16	0.10	0.10	0.11	0.19
Low-Shear Mixing:						
Tip Speed, m/s	11.8	11.8	11.8	13.3	11.8	11.8
Retention, minutes	0.34	0.58	0.29	0.34	0.39	0.56
Estimated Mixing Energy:						
Unit Reactor, kWh/t	14-18	30	16-23	19-35	30	45

Target residual ash and heating value recoveries were met in each case except possibly for the Dietz coal. It was especially noteworthy that over 98% of the heating value was recovered from four of the five bituminous coals and that recovery from even the very finely ground Winifrede coal exceeded 94% when achieving the desired ash rejection. These heating values recoveries and ash rejections were confirmed by the two-stage agglomeration work on two of the coals.

Conclusions

All five bituminous coals responded well to laboratory scale selective agglomeration with both pentane and heptane as the bridging liquids. Target residual ash and heating value recovery specifications were easily met for the Taggart, Elkhorn No. 3, Sunnyside, Indiana VII, and Winifrede coals ground to the fineness projected from the Subtask 6.2 liberation studies. The target sulfur specification was met when cleaning the Taggart, Sunnyside, and Indiana VII coals. The subbituminous Dietz coal did not respond well and required a considerable amount of asphalt activation and acidification before agglomeration, so agglomeration may not be a cost-effective method for cleaning that coal. As such, no further work was performed on the Dietz coal.

Pentane, pure heptane, commercial heptane, and dearomatized (hydrotreated) commercial heptane bridging liquids appeared to be equally capable of agglomerating the ground test coals while effectively rejecting ash minerals. Because of its low boiling temperature, the coal slurries required precooling when pentane was used as the bridging liquid. Dearomatized commercial heptane was the preferred bridging liquid because of its low cost and mild odor and was therefore recommended for use during Subtask 6.5 testing.

The heptane to coal and pentane to coal bridging liquid ratios for good agglomeration ranged from 0.18 grams hydrocarbon per gram coal on up to 0.36 grams per gram coal. The more finely ground slurries, such as the Winifrede slurry, required more bridging liquid. Indiana VII slurry also required about 16 lb/t asphalt along with the bridging liquid in order to activate agglomeration.

Agglomeration proceeded well in both of the continuous systems under a variety of operating conditions (percent solids, impeller speeds, feed rates, etc.). The heating value recovery fell sharply and agglomeration ceased when the capacity of the units were exceeded at high feed rates/short retention times. Changes in operating conditions had little impact upon the amount of residual ash left in the agglomerated clean coal.

The separation performances of the unitized reactor and the two-stage system were similar, but it appeared that the two-stage system required less high-shear mixing energy for agglomerating fine coal. Since the unitized reactor system did not seem to offer any power-saving advantages, the two-stage system was recommended for use in the bench-scale testing because its development was further along and more scale-up information was available, based on the experience of Arcanum and Bechtel gained under a prior DOE project. High-shear mixing energy consumptions in the two-stage system were in the 11.8 to 23.6 kwhr/ton range for minus 325 mesh coal.

Subtask 6.4 - CWF Formulation Studies

Following the completion of the Subtask 4.3 Coal Water Slurry Fuel (CWF) test work utilizing advanced flotation products, work began on Subtask 6.4 to investigate the formulation of CWF from selective agglomeration products. Details of this work can be found in the Subtask 6.4 CWF Formulation Studies Topical Report [6].

During this work, CWFs were formulated from five of the six project coals investigated during Phase I of the project, Taggart, Sunnyside, Elkhorn No. 3, Indiana VII, and Winifrede. The Hiawatha coal (replacement for Sunnyside) was tested as well since it was to be utilized during the Subtask 6.5 bench-scale and Task 9 PDU operation test programs. The subbituminous Dietz project coal was not tested at all since it could not be cleaned by selective agglomeration to meet the product ash specification goal.

A survey of past and present CWF combustion technology suggested that, for oil and gas retrofit applications, the slurry need not contain more than 60-62% coal (or approximately 8,800-9,300 Btu/lb) but should have a viscosity of less than 500 cP.

Subtask 6.4 testing focused on determining the reagent additions and particle size distributions (PSDs) required to meet these goals. Suitable CWF was prepared from the Taggart, Sunnyside, Elkhorn No. 3, and Hiawatha coals that had been ground to D_{80}s in the 34 to 67 micron range and cleaned by selective agglomeration to contain less than 2 lb ash/MBtu. The Indiana VII and Winifrede coals were found to be less desirable feedstocks since they required finer grinding for liberation of the ash minerals. This finer grinding, and in the case of Indiana VII coal the high inherent moisture content, resulted in very low slurry loadings (less than 52%).

It was found that between 10 and 20 lb/ton coal of A-23M dispersant was required for the Taggart, Sunnyside, Elkhorn No. 3, and Hiawatha coal slurries to achieve 60-62% coal loadings at a viscosity of 500 cP. The solids loadings of 500 cP viscosity slurries, when no dispersant was used, were only in the 50 to 52% range.

Generally, the CWFs prepared with dispersant were unstable and would need to be used soon after preparation, or agitated while stored. Their stability was improved by either omission of the dispersant or by adding Flocon 4800C xanthan gum as a stabilizer. In either case, there was a sacrifice in loading and in the case of the stabilizer addition, a significant extra cost for reagents along with an increase in slurry viscosity.

In some cases, it was found that particle size distribution manipulation to produce better packing of the particles in the slurry (bi-modal PSD), increased achievable slurry loadings. However, the improvements were usually meager compared to the higher capital and operating costs associated with the addition of sufficient grinding capacity to achieve these PSD manipulations in a commercial plant.

Experimental results were generally consistent with predictions from a slurry properties model developed by Dr. John Dooher of Adelphi University.

Subtask 6.5 - Bench-Scale Testing

Following the completion of the Subtask 6.3 Process optimization Research test work, the selective agglomeration effort shifted to the continuous bench-scale testing carried out under Subtask 6.5, which had three main objectives:

1. Design, construct, and operate a continuous selective agglomeration system of about 25 lb/hr capacity to demonstrate the feasibility of the process.
2. Optimize the selective agglomeration process conditions to minimize product ash contents, and reduce process costs.
3. Generate design data of sufficient reliability to insure successful scale-up of the process to the 2 t/hr process development unit (PDU) scale.

These objectives were achieved through bench-scale testing on coals selected during Task 2 [3]. The basis for this work stemmed from the results obtained during Subtask 6.3 Process Optimization Research testing. Details of this work can be found in the Subtask 6.5 Bench-Scale Testing and Process Scale-up Topical Report [7].

The bench-scale unit utilized during the Subtask 6.5 was of sufficient size to produce at least 25 lb/hr of agglomerated product (dry basis), and capable of processing all project coals. This testing utilized heptane as the agglomerant, or bridging liquid, which was recovered via steam stripping for recycle to the process.

To simplify operation, coal grinding was carried out independently of agglomeration testing. Once finely ground to achieve the required liberation, the coal slurry was subjected to a high-shear unit operation in which intense mixing dispersed the bridging liquid (heptane) and provided sufficient heptane/coal and coal/coal contact to achieve a phase inversion and form what are termed “microagglomerates”. These microagglomerates were then subjected to additional mixing in a low-shear unit operation allowing the agglomerates to grow to a sufficient size for physical recovery by screening. Once formed, the agglomerates were dewatered, rinsed, and recovered on a vibrating screen. The mineral impurities remained dispersed in the water (screen underflow).

Recovery of the heptane from the agglomerated product was achieved in two stages of steam stripping. In the first stage, the reslurried agglomerates were steam stripped at ambient pressure boiling temperatures removing the bulk of the heptane. In the second stage, the slurry was subjected to additional steam stripping at elevated temperatures and pressures removing additional heptane. The recovered vapor (heptane and water) was then condensed, cooled, separated into separate heptane and water streams by gravity settling, and recycled for reuse in the process.

Bench-Scale Unit Description

The bench-scale unit was designed to produce about 50 lb/hr of product on a dry basis. This design insured that a processing rate of 25 lb/hr could be achieved for coals requiring longer residence times. While the test unit was capable of evaluating the agglomeration process continuously, the grinding, steam stripping, and final product dewatering steps were normally carried out separately. As such, the test unit included storage for both the ground feedstock and the recovered product.

The selective agglomeration bench-scale unit consisted of the following unit operations:

- Coal Grinding and Feed System - Coals were ground to the appropriate size in a 4-foot x 4-foot ball mill and a Drais stirred-ball mill. Some coals were also ground in the 2 t/hr Process Development Unit (PDU). Ground slurry was then fed to the process from an agitated 55-gallon feed drum.
- High-Shear Agglomeration - The high-shear unit operation was designed to provide complete dispersion of the heptane agglomerant, sufficient heptane/coal contact to coat the coal with heptane, and sufficient particle to particle contact, insuring the formation of microagglomerates (phase inversion). To meet these objectives for all project coals, the high-shear vessel design was based on the requirements for the Indiana VII coal, the coal needing the longest residence time (2 to 3 minutes). Heptane was added prior to high shear. When

necessary to promote agglomeration, asphalt in the form of an emulsion was also added.

- Low-Shear Agglomeration - Based on previous experience, the low-shear vessel was designed to provide a residence time of 5 minutes, insuring agglomerate growth to sufficient size. This vessel, through the application of a centrally located horizontal baffle, provided two separate low-shear mixing zones.
- Agglomerate Recovery - Primary agglomerate recovery was carried out on a 10-inch x 16-inch vibrating screen with adjustable inclination, a 48- or 100-mesh screen deck, and water sprays. The tailings (screen underflow) discharged into a froth skimming column designed to recover any carbonaceous heptane bearing material from the tailings into the product.
- Heptane Removal - Heptane was removed from the agglomerated product by direct contact steam stripping in one or two stages, where both heptane and water were evaporated simultaneously. During initial testing, a single stage of steam stripping was used. This stripping vessel was a 4-inch diameter column approximately 52 inches tall. A portion of this column was filled with packing (5/8-inch stainless steel Pall rings). For later testing, the stripping circuit was modified to include a new stripping vessel installed prior to the above described column. This allowed the bulk of the heptane to be removed in the new first-stage stripper followed by additional heptane removal in the column, which was modified to operate at elevated pressures and temperatures.
- Heptane Recovery - The exiting vapor stream from the steam stripping circuit was condensed in a tube coil submersed in a water bath. Once condensed and cooled, this heptane/water mixture was separated by gravity in a column where the heptane overflowed the top and the water was removed from the bottom.
- Product and Tailings Dewatering - Due to the small scale of the test unit, a continuous integrated dewatering system was not provided. Therefore, bulk quantities of both product and tailings were dewatered by existing equipment.

Test Coals and Grinding Requirements

Prior to the start of Subtask 6.5 testing, it was determined that the low-rank Dietz coal would not be evaluated due to the combination of the long high-shear residence time, high asphalt dosage, and low pH required to achieve agglomeration. As such, bench-scale testing focused on the five remaining project coals as well as the Hiawatha coal which was chosen as a replacement for the no longer available Sunnyside coal. These six coals are shown in Table 8 along with their ranks and sources.

Table 8. Subtask 6.5 Test Coals

Coals Seam	Rank	State	Source Mine
Taggart	hvA Bituminous	Virginia	Wentz
Indiana VII	hvC Bituminous	Indiana	Minnehaha
Sunnyside	hvA Bituminous	Utah	Sunnyside
Winifrede	hvA Bituminous	West Virginia	Sandlick
Elkhorn No. 3	hvA Bituminous	Kentucky	Chapperal
Hiawatha	hvA Bituminous	Utah	Crandall Creek

Table 9 summarizes all of the particle size distributions (PSDs) evaluated during Subtask 6.5 continuous testing by showing 20, 50, and 80 percent passing sizes (D_{20}, D_{50}, and D_{80}), as well as the mass mean diameter (MMD) for each feedstock. Also shown is an indication as to whether each PSD provided sufficient mineral-matter liberation to meet the project ash target of 2 lb/MBtu (1 lb/MBtu for the Taggart coal).

Table 9. Subtask 6.5 Feed Particle Size Distributions

	Microns				
Coal	D_{20}	D_{50}	D_{80}	MMD	Ash Target Met
Taggart	8.9	28.1	90.8	50.7	No
	8.8	26.8	64.9	36.9	No
	12.6	38.0	74.7	45.7	No
	5.9	16.2	32.8	23.0	Yes
Sunnyside	8.0	24.9	59.6	34.3	Yes
	6.5	19.5	42.9	25.1	Yes
Indiana VII	4.2	12.0	24.1	14.6	Yes
	4.2	10.8	26.0	16.4	No
Elkhorn No. 3	10.7	29.6	68.0	39.4	Yes
Winifrede	2.0	4.2	12.4	7.1	Yes
Hiawatha	11.5	32.9	65.2	40.9	Yes
	8.1	22.5	46.6	27.4	Yes

Batch Agglomeration Testing

Batch agglomeration tests were performed on samples of various ground feedstocks to evaluate the liberation characteristics of each grind. Generally, slightly lower product ash levels were achieved in the continuous unit than during the batch tests. A list of grind sizes required to meet the project 2 lb ash/MBtu product specifications via batch testing for each coal is as follows:

- Winifrede Coal - D_{80} = 12 microns
- Elkhorn No. 3 Coal - D_{80} = 68 microns
- Taggart Coal - D_{80} = 15, 30, and 38 microns (1 lb ash/MBtu)
- Hiawatha Coal - D_{80} = 65 and 47 microns

- Indiana VII Coal - D_{80} = 20 microns

Continuous Agglomeration Testing

Winifrede Coal Results - The Winifrede coal was ground to a D_{80} of 12 microns and 16 continuous agglomeration tests were carried out using both fresh commercial grade heptane and recycled commercial grade heptane (recovered from previous test work). Following is a list of main operating condition ranges tested for the Winifrede coal:

- Feedstock D_{80} - 12 microns
- Coal feed rate - 12 to 32 lb/hr
- Feed slurry solids concentration - 7 and 10%
- Heptane dosage - 52 to 62% on a dry ash free coal basis
- Asphalt addition level - 5 lb/ton coal
- High-shear impeller tip speed - 15 to 20 m/s
- High-shear residence time - 1 to 2 minutes
- Low-shear impeller tip speed - 5 to 10 m/s
- Low-shear residence time - 3 to 7 minutes

The 2 lb ash/MBtu product specification was met in many of the tests completed, indicating that the 12 micron D_{80} grind provided sufficient mineral-matter liberation, confirming the batch testing results. Results also indicated that very high Btu recoveries (>99%) were achieved with tailings ash values in the 47 to 89% range, with most in the 78 to 89% range.

Elkhorn No. 3 Coal Results - The Elkhorn No. 3 coal was ground to a D_{80} of 68 microns and 32 agglomeration test runs were completed with this coal covering the following main operating condition ranges:

- Feedstock D_{80} - 68 microns
- Coal feed rate - 17 to 33 lb/hr
- Feed slurry solids concentration - 7, 10, and 13%
- Heptane dosage - 23 to 35% on a dry ash free coal basis
- High-shear impeller tip speed - 12 to 18 m/s
- High-shear residence time - 1 to 2 minutes
- Low-shear impeller tip speed - 4.8 and 8 m/s
- Low-shear residence time - 3.4 to 9.4 minutes

The 2 lb ash/MBtu product specification was met for all but one of the tests completed (1.7 to 1.9 lb/MBtu), indicating that the 100-mesh topsize grind provided sufficient mineral-matter liberation. Btu recoveries achieved were in the 88 to 98% range, with corresponding tailings ash values in the 25 to 65% range. These relatively low Btu recoveries and tailings ash values are attributed to oxidation of the Elkhorn No. 3 coal which had been stored for over two years prior to its use for this work.

Sunnyside Coal Results - The Sunnyside coal was ground to D_{80}s of 60 and 43 microns followed by the completion of high-shear evaluation and continuous agglomeration testing.

The high-shear testing utilized both the 100- and 150-mesh topsize feedstocks and two different high-shear impellers and determined the minimum high-shear impeller tip speed required to achieve inversion at various coal feed rates and solids concentrations. Trends observed during this work included:

- As residence time in high shear decreases, impeller tip speed must be increased to maintain inversion.
- As solids concentration increases, lower impeller tip speeds are required to achieve inversion.
- The 2.4-inch diameter impeller draws less power to achieve inversion than the 3.6-inch impeller.

A total of 18 tests were completed with the Sunnyside coal utilizing both feedstocks. Following is a list of main operating variable ranges tested:

- Feedstock D_{80} - 60 and 43 microns (100- and 150-mesh topsizes)
- Coal feed rate - 24 to 49 lb/hr
- Feed slurry solids concentration - 5, 7, 10, and 13%
- Heptane dosage - 25 to 30% on a dry ash free coal basis
- High-shear impeller tip speed - 12 to 22 m/s
- High-shear residence time - 0.7 to 1.5 minutes
- Low-shear configuration - Half full and full
- Low-shear residence time - 1.8 to 7.3 minutes

Two of the four 100-mesh topsize tests achieved the 2 lb/MBtu product ash specification at high Btu recoveries (>98%). This testing showed that lower product ash contents were achieved at solids concentrations of 5 and 7% than at 10 and 13%. All but one of the 150-mesh topsize tests achieved the product ash specification of 2 lb/MBtu, indicating that both grinds were sufficiently fine to meet the project goals.

Taggart Coal Results - Taggart coal testing was carried out at D_{80}s of 91, 88, 65, and 33 microns. A total of 29 tests were completed with the various Taggart coal feedstocks. The following is a list of operating variable ranges tested:

- Feedstock D_{80} - 33, 65, 88, and 91 microns
- Coal feed rate - 17.5 to 50.5 lb/hr

- Feed slurry solids concentration - 5, 7, 10, and 13%
- Heptane dosage - 22 to 35% on a dry ash free coal basis
- High-shear impeller tip speed - 6.4 to 15.0 m/s
- High-shear residence time - 0.5 to 1.5 minutes
- Low-shear impeller tip speed - 3 to 8 m/s
- Low-shear residence time - 1.7 to 7.3 minutes

Results from this work showed that all four of the feedstocks met the 2 lb/MBtu product ash specification. However, only the finest grind tested (D_{80} = 33 microns) was able to achieve the 1 lb/MBtu product ash specification. Btu recoveries were high (>96%) for all of the tests, with tailings ash values in the 32 to 83% range.

In comparing the two middle size grinds, 65 and 88 micron D_{80}s, it is interesting to note that the coarser of these two feedstocks resulted in lower product ash values. This trend was unexpected since typically, the finer the grind, the greater the mineral matter liberation. However, when comparing these two particular grinds, it should be noted that the coarser grind was produced in the PDU, where the bulk of the material being reground was the cyclone underflow stream. Conversely, the finer grind was produced in the 4-foot x 4-foot ball mill operated in closed circuit with a 100-mesh screen, where the regrind material is the screen overflow stream. While the cyclone separation in the PDU grinding circuit is based primarily on size, the material specific gravity also affects the separation, with the underflow stream typically heavier and higher in ash. As such, selective regrinding of the higher ash material occurred, which in this case, resulted in a more liberated product as compared to the finer grind obtained from the 4-foot x 4-foot ball mill.

Indiana VII Coal Results - Continuous agglomeration testing utilizing the Indiana VII coal was carried out using feedstocks with D_{80}s of 22 and 26 microns A total of 20 agglomeration circuit tests were completed using these two feedstocks. The following main operating variable ranges were tested:

- Feedstock D_{80} - 22 and 26 microns
- Coal feed rate - 12.3 to 33.4 lb/hr
- Feed slurry solids concentration - 7, 10, and 13%
- Heptane dosage - 30.0 to 35% on a dry ash free coal basis
- Asphalt dosage - 7.5 to 20 lb per ton feed coal
- High-shear impeller tip speed - 13.4 to 18.0 m/s
- High-shear residence time - 1 to 2 minutes
- Low-shear impeller tip speed - 3 to and 8 m/s
- Low-shear residence time - 3.4 to 7.6 minutes

Results from this work showed that the product ash specification of 2 lb ash/MBtu was met, at approximately 99% Btu recovery, for the D_{80} = 22 micron feedstock, indicating that this grind size provided sufficient mineral-matter liberation. None of the tests carried out utilizing the PDU ground feedstock (D_{80} = 26 microns) met the 2 lb/MBtu product ash specification.

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

- The addition of 7.5 to 20 lb asphalt/ton of coal was required to achieve phase inversion during high-shear agglomeration.
- Without this asphalt addition, no growth occurred during low shear resulting in no coal recovery.

Hiawatha Coal Results - The Hiawatha coal was tested at D_{80}s of 47 and 65 microns. A total of 27 tests were completed to evaluate Hiawatha coal covering the following range of main operating variables:

- Feedstock D_{80} - 47 and 65 microns
- Coal feed rate - 17.5 to 75.8 lb/hr
- Feed slurry solids concentration - 7, 10, and 13%
- Heptane dosage - 21.6 to 28.5% on a dry ash free coal basis
- High-shear impeller tip speed - 6.7 to 18.0 m/s
- High-shear residence time - 0.5 to 1.9 minutes
- Low-shear impeller tip speed - 3.0 to and 8.0 m/s
- Low-shear residence time - 1.5 to 7.2 minutes

Results of this testing indicated that the product ash specification of 2 lb ash/MBtu was met for both of these feedstocks. For the finer of these two grinds, Btu recoveries were all greater than 99% with tailings ash values in the 81 to 87% range. However, for the coarser grind, Btu recoveries were slightly lower, 96.6 to 99.6% with tailings ash values in the 56 to 80% range. This drop in Btu recovery for the coarser grind is attributed to the presence of more unliberated mineral-matter particles, which reported to the tailings stream along with their associated carbon content.

Agglomeration Testing Conclusions

The results of the Subtask 6.5 agglomeration test work indicate that the product ash specification of 1 to 2 lb/MBtu, as well as the Btu recovery goal of at least 80% on a run-of-mine basis were met for all six of the coals tested. Of paramount importance in achieving these product ash levels was the size to which the coal is ground. As for any physical coal cleaning process, if sufficient mineral-matter liberation is not achieved, the desired product grade can not be attained, except at the expense of significant Btu losses to the tailings stream. The coarsest particle size distribution to which each coal was ground to achieve the project goals are summarized in Table 10. Included in Table 10 are typical product ash and Btu recovery values attained when operating at optimized conditions for the grind sizes shown.

Table 10. Bench-Scale Agglomeration Results Summary

	PSD Summary, Microns				Ash	Btu Recovery, %	
Coal	D_{20}	D_{50}	D_{80}	MMD	lb/MBtu	Agglomeration	Run-of-Mine
Taggart	5.9	16.2	32.8	23.0	0.95	99.1	93.5
Sunnyside	8.0	24.9	59.6	34.3	1.79	98.3	88.6
Indiana VII	4.2	10.4	21.9	14.5	1.95	99.0	89.6
Elkhorn No. 3	10.7	29.6	68.0	39.4	1.69	96.8	91.6
Winifrede	2.0	4.2	12.4	7.1	1.91	99.2	88.8
Hiawatha	11.5	32.9	65.2	40.9	1.85	99.6	89.7

Operating conditions required to achieve these typical agglomeration results were found to be various, i.e., several combinations of residence times, energy inputs, and heptane levels could yield similar results.

It was found that the following guidelines should be followed to insure that consistent results are achieved:

1. Sufficient heptane must be used during high shear to form microagglomerates and achieve phase inversion, i.e., the formation of two distinct phases - heptane coated coal and mineral matter bearing process water. Typically, the heptane required increases with decreasing particle size and decreasing coal rank.
2. Sufficient energy (agitation intensity or impeller tip speed) must be applied during high shear to achieve complete dispersion of the heptane and enough particle to particle contact to form microagglomerates. Typically, to achieve these objectives, impeller tip speeds in the range of 10 to 18 m/s are required.
3. Sufficient residence time must be provided in high shear to allow microagglomerates to form. This is a function of the agitation intensity applied, i.e., higher tip speeds will reduce residence time requirements. Typically, on the order of 30 to 60 seconds are necessary, depending primarily on coal fineness and rank.
4. The use of higher, up to 13%, solids concentration during high shear reduces energy input requirements. While these higher solids loadings also resulted in higher product ash levels, this is attributed to the low shear operation, i.e., operation of high shear at high solids followed by dilution for low shear is recommended.

5. For lower rank and oxidized coals like the Indiana VII, the use of an agglomeration promoter like asphalt may be required to achieve phase inversion during high shear.
6. Sufficient heptane must be provided to allow agglomerate growth during low shear to the 2 to 3 mm size range. Over the course of the Subtask 6.5 testing this was found to vary between 25 and 60% heptane on a dry ash free coal basis, depending primarily on the coal particle size distribution and rank.
7. The formation of consistent agglomerates in the 2 to 3 mm size range is paramount in achieving low product ashes. If agglomerates are too small, drainage of mineral-matter bearing process water will not occur. If agglomerates are too large, their handleability diminishes affecting downstream operations.
8. Impeller tip speed (agitation intensity) during low shear typically needs to be about 5 m/s to allow the growth of well formed agglomerates of sufficient strength for vibrating screen recovery. If agitation is too mild, weak agglomerates with high water contents are formed while if agitation is too intense, agglomerate growth will not occur. Low-shear operation appears to be independent of the coal rank and only slightly affected by particle size with finer feedstocks generally forming stronger better formed agglomerates.
9. Residence time in low shear was found to have little effect on agglomerate growth, since ultimately, agglomerate formation is controlled by heptane dosage and low-shear agitation intensity. However, residence times no greater than 2 to 3 minutes are recommended since longer residence times make agglomerate growth very difficult to control.
10. The design of the low-shear vessel should be such that the discharge is located at the same elevation as the impeller. This will insure that continual low-shear discharge occurs under all potential operating conditions.
11. The use of higher, 13%, solids concentrations in low shear results in higher product ash levels than if operated at lower solids concentrations. High solids loadings during low shear also makes agglomerate growth difficult to control.
12. The vibrating screen used for agglomerate recovery must have sufficient forward linear motion to provide good transport of agglomerates across the screen deck. If good agglomerate movement is not achieved, the agglomerate bed depth increases reducing the drainage of associated mineral-matter bearing process water.
13. Screen spray water is required to rinse the mineral matter associated with the process water to the tailings stream. The amount of rinse water necessary has not been quantified, but is believed to be relatively low.
14. The use of a froth skimmer to recover coal from the screen tailings was shown to result in Btu recovery increases on the order of 1 to 3%, depending on the coal and operating conditions used. Application of a froth skimmer also helps reduce tailings heptane contamination. However, the usefulness of a froth skimmer is ultimately determined by the amount of coal present in the screen

underflow with potential sources of coal contamination in the screen underflow being coal not agglomerated during low shear, and agglomerate degradation during screening. Therefore, process optimization should focus on minimizing the introduction of coal to the screen tailings rather than reliance on a froth skimmer. If utilized, a froth skimmer must be of the appropriate design to insure the formation of a removable froth layer.

Overall, the selective agglomeration process was found to be very robust in that it either worked well or didn’t work at all. Therefore, as long as the coal grind provides sufficient mineral-matter liberation and a consistent low-shear product of the appropriate size is produced, the desired product grade along with consistently high Btu recoveries will be achieved.

Batch Stripper Testing

In an effort to better quantify the residual heptane concentrations remaining with a stripped agglomerated product, a number of batch stripper tests were carried out. These batch tests were found to provide a good indication of some parameter effects on residual heptane concentrations.

During the course of Subtask 6.5 testing, two types of heptane were used. The first type tested was a commercial grade heptane with a bulk cost of about $1.00 per gallon. The second type tested was a pure grade heptane with a bulk cost of about $6.00 per gallon. Characterization of these two heptane types was carried out with the results indicating that the n-heptane content of the commercial grade heptane was in the 21 to 25% range, while the pure grade heptane contained greater than 99% n-heptane.

In an effort to achieve reliable analysis of residual heptane on steam stripped products, it was determined that given the many compounds present in the commercial grade heptane, it was not economical to analyze for all components regularly. As such, it was decided that the samples would be analyzed for n-heptane only, and the amount of total residual hydrocarbon calculated, based on the percentage of total residual hydrocarbons that was n-heptane. To better define residual hydrocarbon (n-heptane) concentrations samples of product agglomerated with commercial heptane, pure heptane, and pentane were stripped in various ways including through the continuous stripper either once or twice, boiled for set periods of time, and thermally dried. The following is a summary of the initial batch stripping test results:

- Lower residual hydrocarbon concentrations were achieved as the residence time under stripping conditions was increased.
- Thermal drying achieved much lower residual heptane levels than boiling. This is believed to be due to a combination of the longer residence times used, the higher temperatures used, and the removal of virtually all water present.
- Storage of the product for 2 days prior to stripping resulted in higher residual hydrocarbon concentrations than immediate stripping.

Other conclusions made from the batch stripping test work included:

- Under virtually every set of conditions tested, the presence of asphalt in the stripper feed material resulted in lower residual hydrocarbon concentrations. This was found to hold true regardless of whether the stripping was done by boiling or thermally, and for both commercial and pure grades of heptane.
- Steam stripping at elevated temperatures and pressures resulted in reduced residual hydrocarbon concentrations.
- The residual heptane concentration of stored highly loaded CWF slurries formulated from agglomerated products does not decrease, indicating no safety and/or environmental related risks.
- Steam stripping (or boiling) at 25% solids concentration resulted in virtually the same levels of residual heptane as when the stripping was carried out at 10% solids concentration.

Continuous Stripper Testing

Single-Stage Stripper Results - In an effort to quantify the effects of various stripper operating parameters on product heptane levels, a number of single-stage stripper tests were carried out. This work utilized Sunnyside, Taggart, and Indiana VII coals.

Sunnyside Coal - Sunnyside coal tests indicated residual hydrocarbon concentrations of 3500 to 8000 ppm dcb when the commercial grade heptane was used. Similar ranges of trace n-heptane, 4700 to 5400 ppm dcb, were determined for those tests in which the pure grade of heptane was utilized.

Taggart Coal - Stripping of the 62-mesh topsize Taggart coal agglomerated product achieved residual total hydrocarbon concentrations in the range of 2000 to 6000 ppm dcb. There were no obvious trends relating residence time to product heptane concentration. It was also determined that there was no difference in performance between operating the column flooded (high liquid level) or in a continuous steam mode (low liquid level). This data also indicated that a significant reduction in the exiting vapor temperature, from 95 to 78.3ºC did not result in an increased product heptane content. The steam to coal ratio, which varied from as high as 12.5 to as low as 1.7 for the Taggart coal testing, indicated that no benefit was realized from the use of large quantities of excess steam.

Indiana VII Coal - Residual n-heptane concentrations for the two tests completed with the Indiana VII coal were about 2400 and 1800 ppm of n-heptane, respectively, equivalent to approximately 7000 and 5000 ppm dcb of total residual hydrocarbons.

Two-Stage Stripper Testing - Following are the two-stage stripping circuit results.

Elkhorn No. 3 Coal - Three different tests were completed using the modified two-stage stripping circuit with Elkhorn No. 3 coal. These results indicated that two stages of steam stripping achieved lower trace heptane concentrations than a single stage of steam stripping. This was due to the increased temperature in the second stage.

Hiawatha Coal - One continuous steam stripping test with agglomerated Hiawatha coal was completed. The results of this test indicated that the Stripper 1 and Stripper 2 products contained approximately 4300 and 3000 ppm (0.43 and 0.3%) of heptane on a dry coal basis, respectively.

Indiana VII Coal - One continuous steam stripping test utilizing the Indiana VII coal was carried out. The results of this test indicated that the Stripper 1 and Stripper 2 products contained approximately 5500 and 1800 ppm (0.55 and 0.18%) of heptane on a dry coal basis, respectively.

Stripper Testing Conclusions

In general, steam stripping to remove heptane from agglomerated products is a straight forward operation. The steam is applied directly to the reslurried agglomerates to evaporate heptane along with water, and the ratio of heptane to water in the exiting vapor phase from the stripping circuit is maximized to insure that steam consumption is kept as low as possible.

It was determined that due to the advantage of carrying out steam stripping at elevated pressures and temperatures (lower residual heptane concentrations), a two-stage system should be used. In this scenario, the first-stage stripper was agitated to keep the buoyant agglomerates dispersed throughout the vessel, and operated at ambient pressure to facilitate pumping of the difficult to handle agglomerate feed into this vessel. During this first-stage stripping, the bulk of the heptane was removed disintegrating the agglomerates and providing a handleable first-stage product slurry that could be easily pumped into the second-stage stripper which was operated at elevated pressure and temperature to remove small amounts of additional heptane. The second stage stripper was of a plug flow packed column design to provide greater mass transfer efficiency while insuring minimal back-mixing and good steam distribution across the column’s entire cross section. A counter-current steam flow was used with the fresh steam feeding the second stripper, and its vapor product feeding the first stripper. The first-stage stripper vapor product was then the feed to the condenser and gravity separation heptane recovery column.

Continuous two-stage stripper testing was carried out at operating temperatures of approximately 92°C and 115°C in the first- and second-stage strippers, respectively. Residence times in these strippers were 5 and 10 minutes for the first and second stages, respectively. Under these conditions, residual hydrocarbon concentrations on the order of 2000 to 6000 ppm (0.2 to 0.6%) and 1000 to 3000 ppm (0.1 to 0.3%) on a dry coal basis were achieved in the intermediate and final products, respectively. These residual concentrations appeared to be independent of the coal tested and the type of heptane used, pure grade or commercial grade.

The effect of various steam stripping operating variables on the residual heptane content of the stripped products is summarized as follows:

- When steam stripping heptane from agglomerated products at ambient boiling temperatures, no benefit is gained by providing residence times greater than approximately five minutes.
- Steam stripping at elevated temperatures, as achieved by increased operating pressures, results in lower residual heptane concentrations.
- Increasing the solids concentration at which steam stripping is carried out has no detrimental effect on residual heptane concentrations.
- The presence of asphalt (used as an activator during agglomeration) results in lower residual hydrocarbon concentrations under otherwise similar stripping conditions.
- Regardless of whether a commercial or pure grade of heptane is utilized during agglomeration, total residual hydrocarbon concentrations achieved are similar.

It was found that the only major problem encountered during the continuous stripping testing was feeding the circuit. First, it was very difficult to mix the reslurried agglomerates due to their tendency to float in the feed tank. As such, continuous feeds solids concentrations never exceeded 10 to 15%, compared to the original target feed solids concentration of 25%. This resulted in higher than anticipated steam consumption due to the extra water that needed to be heated. Second, plugging of the stripper circuit feed lines occurred consistently even at the low feed solids concentrations used. It was anticipated that both of these problems would be overcome at larger-scale operations by the use of a diaphragm pump.

Beyond feeding the stripping circuit, no other major operational difficulties were encountered during the stripper testing during Subtask 6.5. Vapor condensation and liquid cooling were achieved in a tube coil submersed in a water bath serviced by utility water. Complete condensation was consistently achieved with minimal carryover of coal from the stripping circuit. Separation of the condensed water and heptane was easily accomplished in a gravity separator column with the heptane overflowing from the top and the water exiting the bottom. This separation was complete with only minimal solubility (<10 ppm) of heptane into the water phase.

Tailings Heptane Analysis

For the design of a tailings disposal system, at both PDU and commercial scales, it is important to know the heptane content of the selective agglomeration process tailings. As such, one set of agglomeration tailings samples (froth skimmer underflow) was analyzed for residual heptane content. These samples originated from an Elkhorn No. 3 coal agglomeration test utilizing commercial grade heptane. The ash content of this tailings sample was approximately 50%. Samples submitted included as produced 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. These results indicate that tailings disposal in conventional waste disposal sites should not be a problem.

Toxic Trace Elements Distribution

The reduction in toxic trace element (TTE) concentrations accomplished by selective agglomeration was studied by assaying the products from selected parametric bench-scale tests and calculating the distribution of the trace elements between the clean coal and tailings. The TTEs of interest were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine. In tracking the TTEs through the selective agglomeration process, the average of all of the mass balance closures was 104%, and the closures were generally within 20% of the amount reported in the agglomeration circuit feed.

The reductions in the various trace element concentrations accomplished during the selective agglomeration tests were calculated on a heating value basis and generally, the concentrations of arsenic, beryllium, cobalt, lead, manganese, mercury, and selenium in the raw coals were clearly reduced by the combined conventional washing and advanced cleaning steps. Much of the reduction, however, was accomplished during washing at the mine-site preparation plant.

Very definitely, selective agglomeration reduced the concentrations of arsenic, chromium, manganese, and nickel remaining in the ground washed coals. It appears that chromium and nickel may have been introduced into the coal slurries during grinding. Selective agglomeration had little impact upon the beryllium, cobalt, lead, mercury, and selenium concentrations, and it appears at times that the antimony and chlorine concentrations increased on a heating value basis. Overall, the residual amounts of the elements in the clean coals were found to be dependent upon the source coal. For example, there was about four times as much antimony in the Indiana VII clean coal as found in the other four clean coals and there was a third as much or less arsenic in the Sunnyside clean coal as in the other clean coals.

In comparing the toxic trace element reductions achieved by the selective agglomeration and advanced flotation processes, both resulted in about the same amounts of the trace elements in the final product.

Reductions from the concentrations found in the ROM parent coals were generally greater than the reductions from the as-received test coals, with substantial reductions, 25 to 90% on a heating value basis, in the concentrations of arsenic, beryllium, chromium, cobalt, lead, manganese, mercury, and selenium. On the other hand, there was little or no reduction, less than 25%, in the amount of antimony and the reduction of nickel and chlorine varied from coal to coal.

DESIGN AND CONSTRUCTION OF PDU SA MODULE

The design and construction of the process development unit (PDU) Selective Agglomeration (SA) Module is briefly discussed in the following sections of this report.

Subtask 6.6 - Conceptual Design of SA Module

The conceptual design of the PDU SA Module was a collaborative effort between Bechtel, Entech, and Arcanum. Flow diagrams and equipment selection are presented in the Bechtel Subtask 6.6 Selective Agglomeration Module Conceptual Design Report [8]. Important issues are summarized below.

Based on the results obtained from the Subtask 6.3 Process Optimization Research and Subtask 6.5 Bench-Scale Testing and Process Scale-up, the following conceptual design selections were made for the 2 t/hr PDU SA Module:

- Heptane, rather than pentane, was chosen as the agglomerant.
- A conventional two-stage agglomeration circuit (separate high- and low-shear unit operations) was chosen rather than the combined high- and low-shear unitized reactor tested during Subtask 6.3.
- It was determined that due to the long high-shear residence times required to achieve inversion with the Indiana VII coal (2 to 3 minutes), two stages of high shear would be installed with the flexibility to use either, or both, vessels.
- It was determined that due to handling problems associated with the recovered agglomerates and residual heptane concentration considerations, two-stages of steam stripping would be utilized.

Task 7 - Detailed Design of SA Module

The detailed design of the PDU SA Module was performed by Bechtel with support from Entech Global and Arcanum engineers. Details of this work can be found in the 3-volume Subtask 7.0 Detail Design of PDU and Selective Agglomeration Module Engineering Package [9].

All structural drawings as well as P&ID's were completed by Bechtel and issued for construction. Electrical drawings were issued by Control Technologies, Inc.

Entech Global managed the procurement of all instrumentation as well as all new and refurbished capital equipment items used in the PDU SA Module.

Even though the plant area (Area 300) designated for the SA Module was of limited space, it was determined that sufficient room was available. As such, several larger equipment items were designed for outdoor installation. The only building modifications made to Area 300 were as follows:

- Installation of a raised removable roof section above the agglomeration vessels/agitators to insure sufficient head space for equipment servicing.
- Installation of a floor sump and pump.
- Strengthening of several steel structural members and the installation of additional members to support the installation of equipment on the roof.
- Modification of the air handling/ventilation system to meet code requirements for an explosion proof area.
- Sealing of all wall and roof penetrations to meet area electrical classification requirements.

To insure safe operation of the SA Module during Subtasks 9.2 and 9.3, the following safety systems were included in the detailed design:

- A nitrogen blanket system, serviced by a liquid nitrogen supply w/evaporator and a variable volume gas holder. This gas blanket system was designed to blanket all pieces of equipment in which heptane was present, to insure that no explosive oxygen/heptane mixtures were formed in the plant.
- A hydrocarbon and oxygen sensing system was also included in the design. This system consisted of several hydrocarbon detectors within Area 300 to detect the presence of heptane vapors in the plant atmosphere. The oxygen detectors were used for two different purposes, to detect a lack of oxygen in the general plant atmosphere and to detect the presence of oxygen on the nitrogen blanket system.
- An emergency relief system was also included in the plant design. This system consisted of pressure relief valves on any equipment in which a significant quantity of heptane was anticipated, a relief system piping network, a knock out drum to catch any liquid relieved from the system, and an emergency flare to burn any relieved vapors/liquids.
- Due to the use of heptane, a volatile and explosive hydrocarbon, in the PDU SA Module, Area 300 was classified electrically as an explosion proof area. As such, all motors and instruments within the area had to be explosion proof or intrinsically safe.
- Due to the presence of heptane and nitrogen in the SA Module, the Area 300 ventilation system was designed to insure the sufficient turnover of the fresh air supply.

Subtask 9.1 - Construction of SA Module

Construction of the PDU SA Module was carried out under Subtask 9.1. Request for Quotation (RFQ) packages were issued for the SA Module construction subcontract during the last quarter of 1995. Entech Global and Bechtel personnel collaborated to decide issues regarding work scope and components of the RFQ. Final copies of the RFQ which included project drawings were sent to the following construction companies:

1. The Industrial Company of Steamboat Springs, Colorado
2. Read Industrial Corporation of Wheatridge, Colorado
3. Western Industrial Contractors of Denver, Colorado
4. Mech El, Inc. of Aurora, Colorado

A site inspection and meeting for interested bidders was held on January 3, 1996. All four construction companies attended this meeting. The majority of this meeting was used to clarify questions and issues regarding the RFQ package. Three of the construction companies submitted a bid estimate for construction of PDU SA Module on January 15, 1996. After careful evaluation, the subcontract for the construction of the PDU SA Module of the PDU was awarded to Mech El, Inc. (MEI), of Aurora, Colorado.

MEI mobilized onto the Amax R&D site on March 11, 1996 and was responsible for the installation of all process equipment, instrumentation, structural steel, concrete, process piping, power systems, and control systems related to the operation of the PDU SA Module.

Control Technologies, Inc. was also hired for development of the control and data acquisition system (DCS) for the SA Module Construction was completed during November 1996.

PDU PROCESS AND PLANT DESCRIPTION

This section of the report describes the PDU selective agglomeration (SA) process and plant (Area 300). It should be noted that two other areas of the plant which were utilized along with the SA module, coal handling and grinding (Area 100) and coal dewatering (Area 400), are also discussed here.

The PDU SA module was a pilot scale (2 t/hr) advanced physical coal cleaning plant which utilized selective agglomeration to remove unwanted mineral matter and its related impurities, such as sulfur and select trace elements, from Run-of-mine (ROM) or washed coal.

The liberation of impurities from coal particles was an important consideration when utilizing selective agglomeration to separate coal and its associated mineral matter. This is because if even small coal surfaces were present on a particle consisting mostly of mineral matter (ash), the particle would agglomerate due to the affinity of the carbonaceous material for the bridging liquids utilized. As such, fine grinding of agglomeration feed was required to insure adequate liberation of mineral matter from the coal so that product quality specifications could be met.

Once finely ground to achieve the required liberation, the coal slurry was subjected to a series of unit operations comprising the selective agglomeration process as studied during the course of this project. The coal slurry was first sent to a high-shear unit operation in which intense mixing dispersed the bridging liquid (heptane) and provided sufficient heptane/coal and coal/coal contact to achieve a phase inversion and form what are termed "microagglomerates." These microagglomerates were then subjected to additional mixing, at a substantially lower shear rate than in the high-shear vessel, allowing the growth of agglomerates to a recoverable (screenable) size, with the fine mineral particles remaining dispersed in the water.

Once these large agglomerates were formed, they were washed, dewatered, and recovered on a vibrating screen. At this point in the process, the critical separation of mineral matter from carbonaceous material was complete. However, for the process to be economic, the bridging liquid heptane (which is volatile, and produces explosive and environmentally unacceptable vapor), had to be recovered from the product and recycled to the process. This heptane recovery was achieved via steam stripping in which steam was applied to the agglomerated product in a two stage process. Once removed from the coal product, the heptane vapor, along with the associated steam was condensed. The condensate was then cooled prior to gravity separation of the heptane and water, both of which were recycled to the process.

The following sections describe the advanced coal cleaning PDU and SA module. Specifically, the following areas of the plant are described:

- Area 100 - Raw Coal Handling
- Area 100 - Grinding and Classification Circuits
- Area 300 - Selective Agglomeration (SA) Circuit
- Area 400 - Dewatering Circuit

Area 100 - Raw Coal Handling

The three coals which were cleaned in the PDU SA module were normal commercial products of coal mines (minus 2-inch washed or run-of-mine coal). They were delivered in 100 ton rail cars to a coal yard located in north Denver, CO. Here, the coal was unloaded and stored until needed at the PDU site. The coal was then transported by truck to Ralston Development Company, located approximately five miles north of the Amax R&D facility. Here the coal was crushed to a 1/2-inch top size and stored in covered bunkers. As needed, the coal was transported by truck to the PDU site for storage in a covered bunker.

A front end loader was used to dump the coal into a receiving hopper from which a vibratory feeder discharged the coal onto an elevating belt conveyor which transported the material to a 15 ton capacity feed bin. A vibrating bin activator, located at the base of the storage bin, minimized plugging while delivering the material onto a weigh belt feeder which metered the coal to the grinding circuit, as shown in Figure 3.

Area 100 - Grinding and Classification Circuits

The grinding and classification circuits were very important to the proper operation of the PDU SA module. Because most of the undesired mineral matter associated with coal is actually disseminated throughout individual coal particles, the mineral matter must first be released or liberated from the coal before any separation can take place. This liberation was achieved by progressively reducing the particle size of the coal. The PDU SA module used two ball mills in series, followed by a fine grinding bead mill.

Figure 3 shows the flow scheme for the Area 100 grinding and classification circuit. The coal, fed at a constant rate from the weigh belt feeder, was dumped into a screw conveyor which transported it to the primary ball mill for grinding. Clarified recycled process water was added to the coal prior to its entrance to the ball mill. The primary ball mill was charged with equal weight distributions of 2-inch, 1-1/2-inch, and 1-inch steel balls.

The ground coal slurry exiting the primary ball mill entered the primary ball mill discharge sump from which it was pumped to the secondary ball mill with a progressive cavity pump. In the secondary ball mill, the slurry was further ground in an effort to achieve adequate liberation of the unwanted minerals. The secondary ball mill was charged with equal weight distributions of 1-1/2-inch, 1-inch, and 1/2-inch steel balls.

At this point, the ground slurry particle size was evaluated by a bank of classifying cyclones, two of which were 3-inch diameter while the remaining four were 2-inch diameter. The slurry exiting the secondary ball mill was pumped to these cyclones by a progressive cavity pump. The 3-inch diameter cyclones were designed to size the Taggart and Hiawatha coals to 80% passing 45 microns while the 2-inch cyclones were designed to size the Indiana VII coal to 80% passing 20 microns. The fines exited through the top of the cyclones (vortex finder) while the coarse material exited through the bottom of the units (apex). Because the optimum solids concentration of the cyclone feed stream was about 20%, additional water was added to the cyclone feed as required.

The coarse cyclone underflow, considered larger than the required top size, was then pumped, via a progressive cavity pump, to either a horizontal fine grinding bead mill or the secondary ball mill for regrinding. This reground product was then combined with the secondary ball mill product and sent to the classifying cyclones.

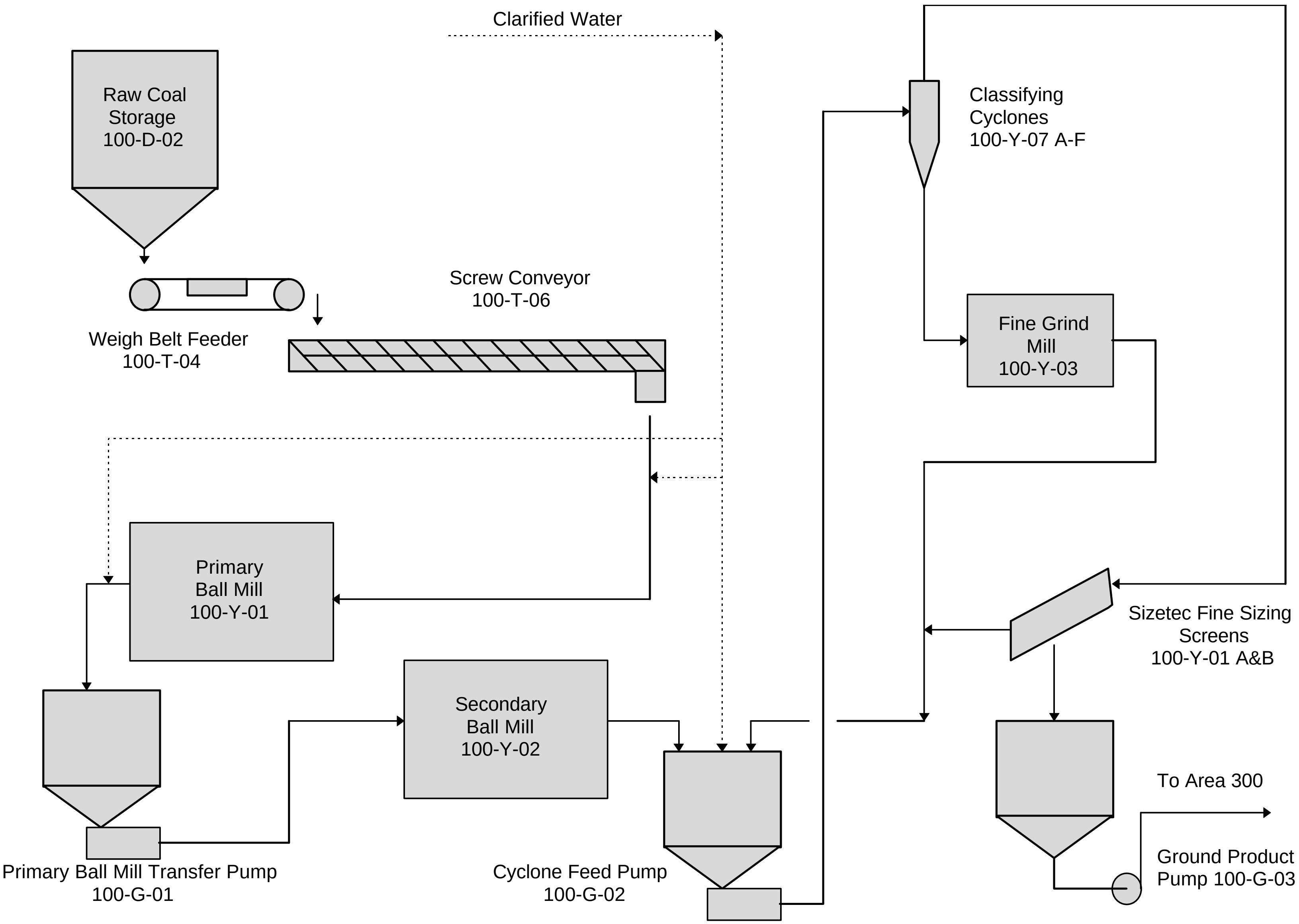


Figure 3. PDU Area 100 - Grinding/Classification Circuit

To guarantee that the particle top size constraint was maintained, the cyclone overflow stream was sent to a pair of high frequency fine sizing screens. The screens assured that all oversize material was removed. The oversized screen overflow product was combined with the classifying cyclone underflow stream and sent to the fine grinding mill or the secondary ball mill for regrinding. The fine material, which passed through the sizing screens flowed by gravity to the ground product sump from where it was pumped to the agglomeration circuit feed tanks by means of a centrifugal pump. With this grinding system, the maximum recommended operating solids concentration for the cyclones plus the wash water requirement for the screens, combined to fix the maximum solids concentration available for testing in the SA module.

Area 300 - Selective Agglomeration Module

The main units of the selective agglomeration process plant comprising the Area 300 PDU SA module are listed below and described on the following pages:

1. High shear agglomeration
2. Low shear agglomeration
3. Agglomerate recovery
4. Heptane Stripping Circuit
5. Condensate Recovery and Recycle
6. Tailings handling
7. Nitrogen blanketing system
8. Relief system
9. Safety Features

High-Shear Agglomeration

During high-shear agglomeration, a mixture of water, coal, and heptane was mechanically agitated such that the heptane dispersed, making contact with all particles in the slurry. Throughout this agitation, hydrophobic coal particles were attracted to the heptane phase, while the hydrophilic mineral matter was repelled from the heptane and attracted to the water phase. With continued mixing, the heptane coated coal particles coalesced to form microagglomerates (phase inversion), while the mineral impurities remain dispersed in the water phase. The following paragraphs describe in detail the unit operations required to complete high-shear agglomeration.

Feed Slurry Storage and Delivery - The SA module feed slurry storage and delivery circuit is shown in Figure 4 along with the high- and low-shear agglomeration unit operation. As this figure depicts, slurry from the grinding circuit was fed to either of two slurry storage tanks (300-D-01 and 300-D-02). Both storage tanks were fitted with fixed speed mixers (300-Y-01 and 300-Y-02) that drove a single axial flow impeller to insure consistent slurry composition. Each of these tanks was fully baffled and had an effective storage capacity of about 4000 gallons.

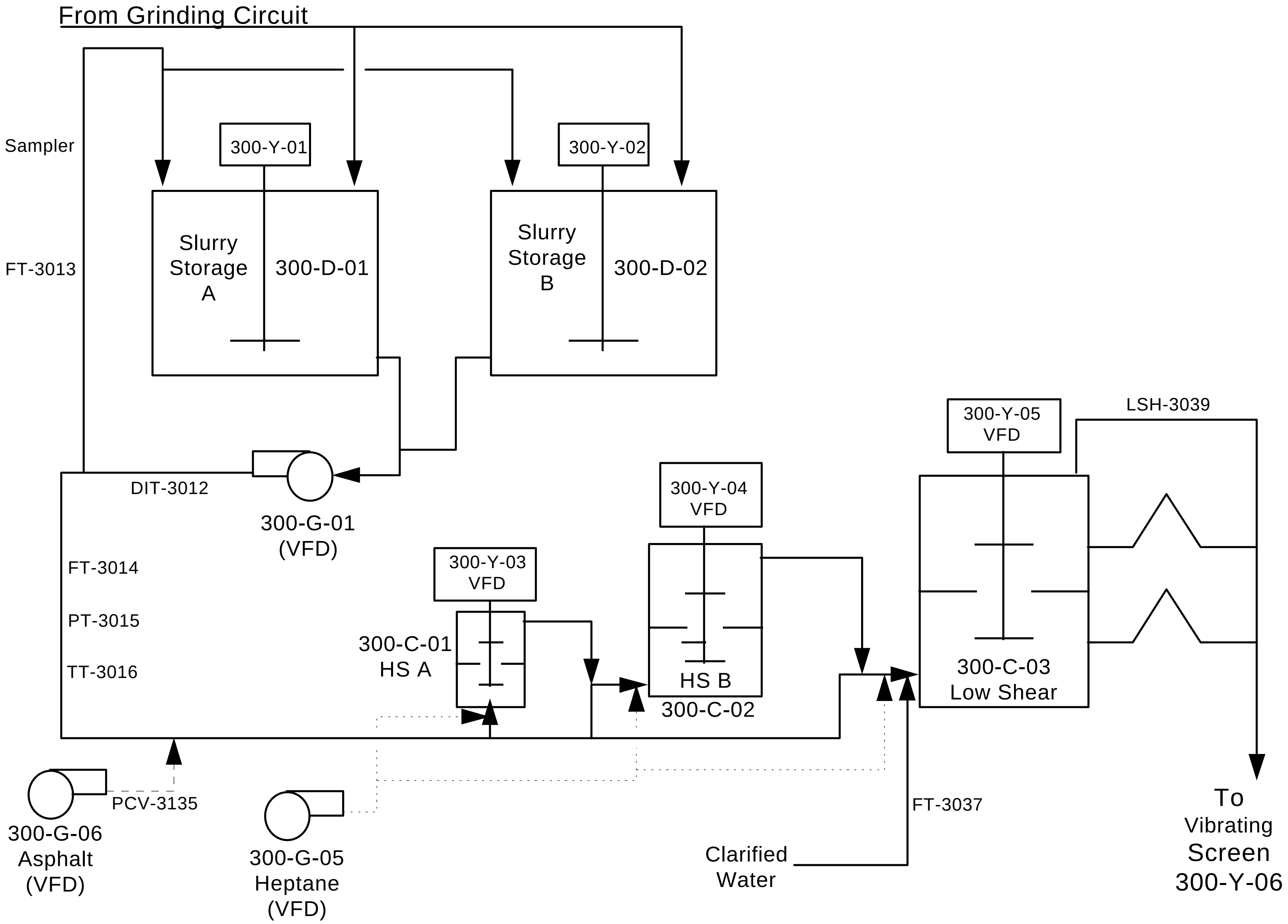


Figure 4. PDU Area 300 - Feed, High Shear, and Low Shear Circuits

Coal was fed to the agglomeration circuit from either of these storage tanks by a variable speed centrifugal pump (300-G-01). The coal slurry feed rate was automatically controlled to maintain a constant volumetric slurry flow to the process based on a magnetic flowmeter (FT-3014) reading. Located directly on the discharge side of this pump was a nuclear density gauge (DIT-3012) providing a solids concentration determination. This on-line density determination combined with the on-line slurry flowrate and other input variables, provided a dry ash free coal feed rate which was then used to control the heptane flow rate in order to automatically maintain a constant heptane to coal ratio.

Downstream of the density meter was a recycle line to the feed storage tanks fitted with a flowmeter (FT-3013) and a sampler. Prior to daily startup, this recycle stream was used to confirm that the solids concentration of the ground slurry feed was in the anticipated range. During normal operation, this feed recycle stream was continuous allowing on-line feed samples to be obtained.

There was also a pressure indicator (PI-3015) located in the agglomeration feed line. This indicator provided a high pressure alarm warning upon any process piping plugging. A temperature transmitter (TT-3016) was also located in the agglomeration circuit feed line.

High-Shear Agglomeration - Coal slurry from the feed system was fed to the high-shear agglomeration circuit. The two high-shear reactors were of 35 gallon (300-C-01) and 75 gallon (300-C-02) capacity and are shown in Figure 4. The piping around these high-shear reactors allowed the use of either vessel individually, or both in series.

Coal slurry entered each high-shear reactor at the bottom of the vessel and discharged from the top. This arrangement assured that the vessels remained full. View ports were provided at the discharge of each high-shear vessel allowing visual inspection to insure that microagglomerates were formed (inversion occurred). Each of these high-shear vessels was fully baffled and divided into two mixing zones with a radial flow impeller centered in each zone. These impellers were powered by variable speed drive mixers (300-Y-03 and 300-Y-04 for 300-C-01 and 300-C-02, respectively) that could achieve impeller tip speeds in the 14-18 m/s range.

Reagent Delivery - Heptane was metered to the agglomeration process by a metering pump (300-G-05). The heptane flow from this pump was controlled by the stroke setting, with actual flows determined by a flowmeter in the heptane delivery line.

When required, an agglomeration conditioner (asphalt) was fed into the agglomeration feed line via a gear pump (300-G-06). The pressure of the asphalt feed was regulated via an orifice union fitting (PCV-3135) to prevent the application of too much pressure to the agglomeration circuit.

Low-shear Agglomeration

Following high shear agglomeration, the microagglomerates were subjected to a low-shear agglomeration step. During low shear, the slurry was mixed at a shear rate significantly less than that used during high shear, typically at impeller tip speeds in the 3 to 5 m/s range, to provide additional agglomerate growth.

For this project, the final process product was in the form of a highly-loaded slurry. As such, the formation of "large" agglomerates, say greater than 2-3 millimeters, with sufficient strength to withstand handling without degradation was not required. Therefore, the primary goal of the low shear agglomeration unit operation was to provide a product which could be easily recovered and dewatered on a screen.

The low shear reactor (300-C-03) is shown in Figure 4 along with the high-shear and slurry feed circuits. This reactor was of 400 gallon capacity and divided into two mixing zones via a horizontal baffle. Centered vertically in each mixing zone was a radial flow impeller driven by a variable speed mixer (300-Y-05). This mixer could achieve impeller tip speeds up to 6.5 m/s.

Discharge ports were arranged so that the low-shear vessel could be operated either full or at half its rated capacity. Discharge from the low-shear vessel was by gravity overflow to the vibrating screen for agglomerate recovery. An additional port was provided in the low shear vessel cover and fitted with a flow switch (LSH-3039) to provide an indication of normal discharge port plugging.

To allow control of the low-shear solids concentration, a provision was made for the addition of dilution water to the low shear vessel. This clarified water addition to the low shear was monitored by flowmeter FT-3037.

Agglomerate Recovery

Once agglomerates were formed in low shear they had to be recovered to the product. The primary goal of this unit operation was to achieve high energy recovery, i.e., minimize coal losses to the process tailings. The second objective of product recovery was to provide a good separation between the product agglomerates and the mineral matter bearing process water. The agglomerate recovery circuit is shown in Figure 5 along with the stripper feed, stripper slurry, and stripper vapor stream circuits.

Vibrating Screen - The screen used for primary agglomerate recovery in the SA module was a 2-foot wide by 6-foot long vibrating dewatering screen (300-Y-06) supplied by Sizetech, Inc. This screen operated with a high-frequency low amplitude linear forward motion. The motion of this screen was such that the agglomerates were moved toward the discharge end of the screen. The screen deck pitch was adjustable to three positions, 6 degrees uphill, level, and 6 degrees downhill. Two spray bars, with two sprays nozzles each, were fitted on the screen. The screen spray water was set based on the output of flowmeter FT-3046. The screen opening size was 48 mesh to allow easy passage of mineral matter while the smallest agglomerates were retained.

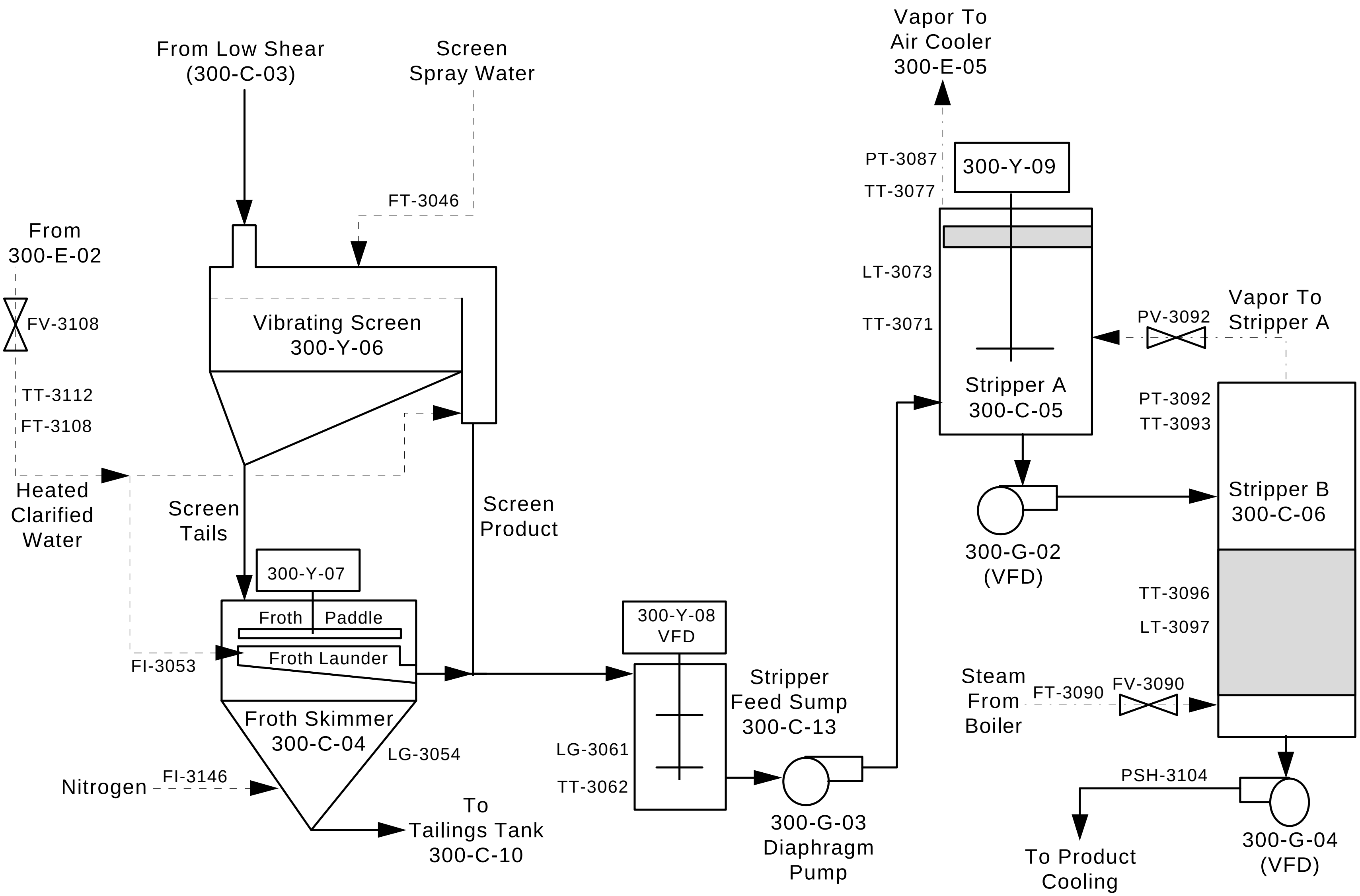


Figure 5. PDU Area 300 - Agglomerate Recovery and Steam Stripping Circuits

Froth Skimmer - Following screening of agglomerates, the screen underflow was processed to recover any coal lost to this stream. This unit operation was carried out in a froth skimmer (300-C-04). Since coated with heptane, coal in the screen underflow floated to the surface where it was skimmed off and combined with the screen overflow product. The froth skimmer used for this was a four-foot diameter tank with a cone shaped bottom. The height of the discharge piping was adjustable to maintain a liquid level in the froth skimmer approximately 1/2-inch below the internal discharge launder. A rotating paddle (300-Y-07) operated at about 15 rpm scraped the froth into the launder. The froth skimmer was also equipped with a sight glass LG-3054 for confirmation of the liquid operating level

The skimmed froth was flushed from the internal launder with pre-heated clarified water and combined with the vibrating screen overflow. This heated push/dilution water was split as needed between the froth skimmer launder and the screen overflow discharge chute. The field flow indicator FI-3053 was used to set up these water flows. When needed to help achieve complete recovery/flotation of all heptane bearing material, nitrogen was dispersed into the bottom of the froth skimmer tank. The nitrogen flowrate was monitored by the field flow indicator FI-3146.

Heptane Stripping Circuit - Slurry Stream

The SA Module steam stripping circuit utilized a two-stage steam stripping process to remove heptane from the recovered agglomerates. The steam flow in this circuit was countercurrent to the process slurry flow (steam entered stripper B first where it picked up a small amount of heptane vapor and then flowed to stripper A). The slurry stream processing of this stripping circuit is shown in Figure 5. The first stage stripper (stripper A or 300-C-05), was used to remove the bulk of the heptane from the agglomerates. Typically, the heptane content of the recovered agglomerates was reduced from the 20-40% heptane (dry coal basis) required to achieve agglomeration, to approximately 1% heptane (dry coal basis). While reducing the heptane content drastically in stripper A, a much more handleable product (basically a coal water slurry) was produced. This stage of stripping was carried out at a pressure in the 2 to 5 psi range, maintaining a temperature above the boiling point of the heptane/water mixture, so that the fresh agglomerates were stripped of the bulk of their heptane virtually instantaneously.

During the second stage of stripping, carried out in stripper B (300-C-06), the residual heptane content was reduced further by stripping at elevated temperatures and corresponding pressures, typically in the 7 to 10 psi range.

Stripping Circuit Feed - The stripping circuit feed sump (300-C-13) had a capacity of about 90 gallons and was fitted with a 10 hp variable speed mixer (300-Y-08) with two axial flow impellers. The combined agglomerated product and heated dilution water were transferred to the first-stage stripper via a diaphragm pump which was operated at a speed sufficient to keep the stripper feed tank empty. The stripper feed mixer was not used in this scenario. Initially a variable speed centrifugal pump, combined with a level transmitter and flowmeter to maintain either a constant feed tank level or a constant flowrate, was used for this task but was found to be incapable of consistently pumping the difficult to handle agglomerates. As such, the stripping circuit feed tank and its associated agitator turned out be unnecessary to the final operating method of the plant.

First Stage Stripper - The level in stripper A was held constant by a variable speed centrifugal pump 300-G-02 (stripper B feed pump) based on the output of the stripper A differential pressure level transmitter LT-3073.

This first stage stripping vessel (300-C-05) is described as follows:

- Diameter of 4-foot 6-inches and approximately 8 feet tall (including a dished top and bottom) for a total volume of about 850 gallons.
- Fitted with a fixed speed (125 rpm) agitator with one 36-inch diameter 3-bladed axial flow impeller located approximately 3-feet off the vessel bottom.
- Contained two steam delivery sparging pipe loops located near the bottom of the vessel, one each for main and auxiliary steam flows, as well as a 6-inch thick demister pad (located about 2-feet from the top of the vessel) to reduce the entrainment of solids in the exiting vapor stream.
- Feed entered on the side, approximately 2-feet from the bottom of the vessel, and the product was withdrawn from the bottom of the vessel.

Given the location of the impeller and demister pads in stripper A, the actual acceptable fluctuation in operating level in this vessel was very small, on the order of 1 to 2 feet. At a level considered full, virtually level with the bottom of the demister pad, the effective volume of stripper A was about 600 gallons. At the lowest operating level used, which corresponded to complete coverage of the impeller, the effective volume of stripper A was approximately 400 gallons.

The temperature of the slurry in stripper A was monitored by TT-3071, while the exiting vapor pressure and temperature were recorded by PT-3087 and TT-3077, respectively. Stripper A was also fitted with a pressure relief valve PSV-3067.

Second Stage Stripper - In a similar manner as for stripper A, the level in stripper B was maintained to a constant level by the variable speed Moyno pump 300-G-04 (stripper B discharge pump) based on the output of the stripper B differential pressure level transmitter LT-3097. The stripper B discharge pump 300-G-04 was fitted with a high pressure switch (PSH-3104) to warn of any downstream plugging conditions.

This second stage stripping vessel (300-C-06) is described as follows:

- Diameter of 4-foot and approximately 12 feet tall (including a dished top and bottom) for a total volume of about 850 gallons.

- Fitted with a steam delivery sparging pipe loop located near the vessel bottom.
- Partially filled with 3/4-inch stainless steel Pall rings to help prevent back mixing of the slurry as it flowed down through the vessel.

Feed entered stripper B near the top of the vessel (above the Pall ring level) and discharged at the vessel bottom. The slurry temperature in stripper B was monitored by TT-3096, while the exiting vapor temperature was recorded by TT-3093. Stripper B was also fitted with a pressure transmitter (PT-3092) and a pressure relief valve (PSV-3095). Given the feed entry point to 300-C-06, and considering this as the maximum operating level, the maximum effective operating volume of this vessel was on the order of 650 gallons.

Product Cooling - Once material was pumped from stripper B, it was cooled by two plateflow heat exchangers in series (300-E-02 and 300-E-03). The product cooling circuit is shown in Figure 6 along with the clarified water distribution system and the utility water cooling circuit.

Cooling through the first exchanger 300-E-02 was achieved with clarified water which was subsequently used to dilute the stripping circuit feed. The clarified water flowrate through, and the temperatures of the clarified water in and out of 300-E-02 were monitored by instruments FT-3108, TT-3107, and TT-3112, respectively. Similarly, the slurry flowrate through, and the temperatures of the slurry in and out of 300-E-02 were monitored by instruments FT-3102, TT-3096, and TT-3105, respectively. The flowrate of the clarified water was maintained by control valve FV-3108 based on the target stripper feed solids concentration.

Cooling in the second heat exchanger 300-E-03 was achieved with utility water, which once used, was discharged to the sewer. The cumulative cooling water flowrate to the sewer was monitored by the field flow totalizer FQ-6025. Target conditions for this cooler were to produce a final product slurry temperature of about 80 °F at a utility cooling water flow rate of approximately 60 gpm. The utility cooling water flowrate through, and the temperatures of the cooling water in and out of 300-E-03 were monitored by instruments FT-3115, TT-6013, and TT-3111, respectively. Similarly, the slurry flowrate through, and the temperatures of the slurry in and out of 300-E-03 were monitored by instruments FT-3102, TT-3105, and TT-3110, respectively.

Clarified Water Circuit - Clarified water was supplied via the clarified water pump 400-G-07 and distributed to both the grinding circuit (Area 100) and the SA Module (Area 300). The total clarified water flow to Area 100 was maintained at a fixed rate by control valve FV-206 based on the output of flowmeter FT-104. As shown in Figure 6, the distribution of clarified water in Area 300 was as follows:

- To the vibrating screen (300-Y-06) as spray water through flowmeter FT-3046
- To the low-shear vessel (300-C-03) as dilution water via flowmeter FT-3037
- To the tailings surge tank (300-C-10) as flush water

- To the emergency slop tank (300-C-12) as flush/cooling water
- To product cooler 300-E-02 as cooling/dilution water

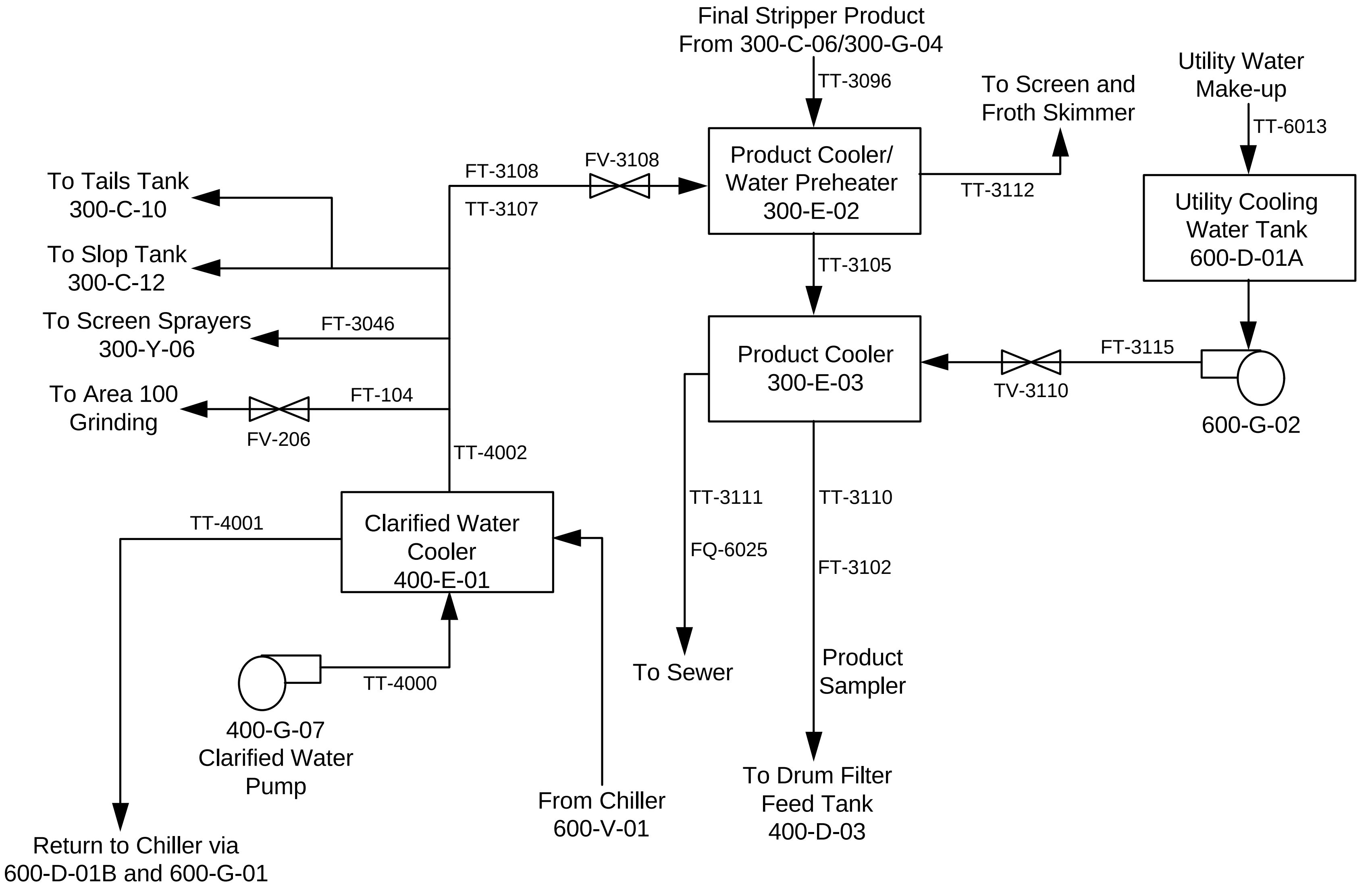


Figure 6. PDU Area 300 - Product Cooling and Clarified Water Circuits

Heptane Stripping Circuit - Vapor Stream

The steam stripping process utilized a steam flow countercurrent to the process slurry. As such, the steam flowed into stripper B first, and then on to stripper A, from which the exiting vapor was condensed, cooled, and recovered.

Steam Feed - Steam for the plant was generated in a trailer mounted 250 HP boiler (300-F-01) fired by natural gas. The steam capacity of this boiler was 6500 lb/hr at an operating pressure of 25 psi. This boiler was self modulating to maintain the desired steam pressure set point. The steam distribution system is shown in Figure 7.

The main steam flow to the process was controlled by a flow control valve (FV-3090). This valve could be operated in three different modes:

1. In manual mode based on a percent open setting.
2. In auto at a target steam lb/hr flowrate monitored by flowmeter FT-3090.
3. In auto to maintain a target temperature (TT-3071) in the stripper A vessel.

In addition to the main steam flow to stripper B, the steam delivery system was also capable of supplying steam to the following plant locations:

- Stripper A, used as an auxiliary steam flow during startup conditions.
- The emergency slop tank (300-C-12), used to strip heptane contaminated process streams such as plant tailings in a batch mode,
- Two steam hand stations, used as required to unplug process piping and heat outdoor process equipment.

The Area 300 steam distribution system was also fitted with two steam traps to remove condensate and a number of manual valves to isolate steam flows as required.

Stripper B Pressure Control - The second stage stripper was generally operated at pressures in the 7 to 10 psi range. Regardless of what control mode was utilized for the main steam flow to stripper B, the vapor discharge stream from this vessel was based on the maintenance of a target pressure in stripper B. This portion of the process is shown in Figure 5. The pressure in stripper B was maintained by control valve PV-3092, whose operation was based on the output of pressure transmitter (PT-3092) located in the head space of stripper B. Under this scenario, any excess steam delivered to stripper B, i.e., steam in addition to that required to raise the slurry temperature coming out of stripper A to the stripper B target temperature, was released by PV-3092 and allowed to flow to stripper A where it was applied to the incoming coal agglomerates.

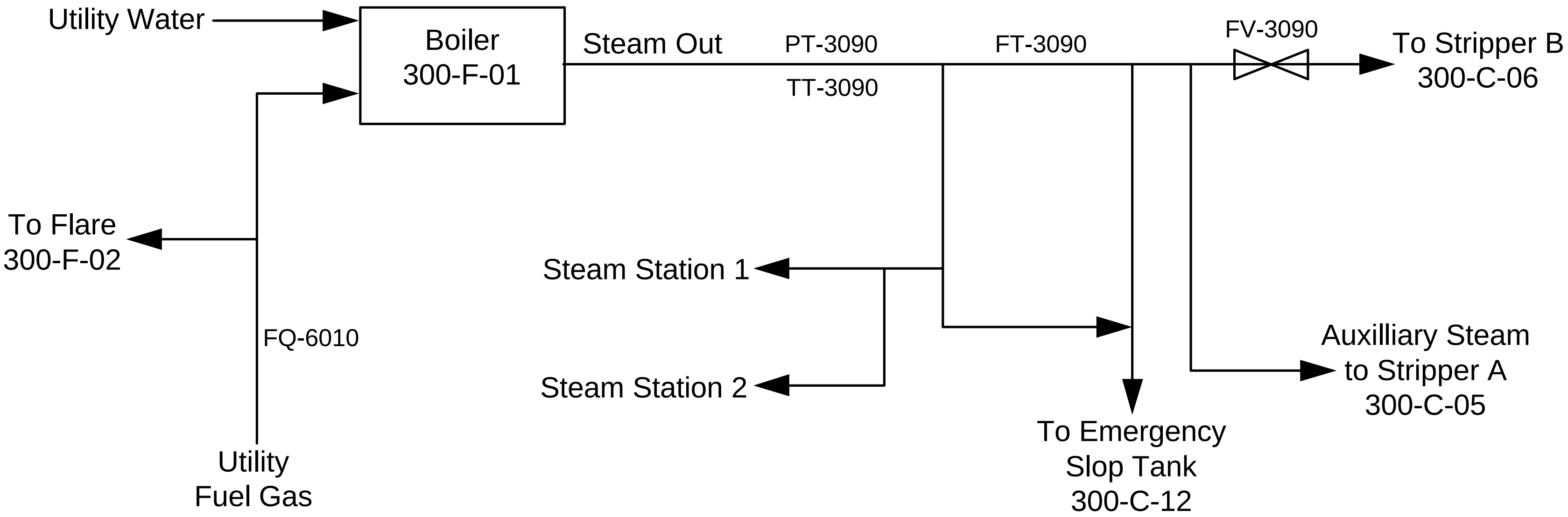


Figure 7. PDU Area 300 - Steam Distribution Circuit

Stripper A Pressure Control - As steam entered stripper A from striper B, it was utilized to maintain a target temperature (TT-3071) of about 200°F in Stripper A. With stripper A at this temperature, the incoming agglomerates were virtually instantly stripped of the bulk of the heptane they contained, resulting in a handleable coal slurry. This section of the process is shown in Figure 5.

In theory, from a heptane removal standpoint, stripper A could have been operated at ambient pressure. However, to help provide surge control and insure uniform residence time in the air cooler, a back pressure of at least 1 psi was maintained from the air cooler condensate discharge point back into stripper A. This back pressure was maintained by a flow control valve (PV-3087) located downstream of the air cooler, which was driven by the output of pressure transmitter (PT-3087) located in the stripper A vapor discharge piping.

Vapor Condensation - As heptane and water were evaporated from the stripping circuit feed in stripper A, this vapor was condensed, cooled, and separated for recycle to the process. This section of the SA Module is shown in Figure 8.

Condensation was achieved in an air cooler (300-E-05) located on the roof of the Area 300 building. This air cooler operated like a radiator, with the vapor/condensate flowing through a series of tubes which were cooled by ambient air via two fans. In order to provide surge control and insure uniform residence time in the air cooler, a back pressure of 2 to 5 psi was maintained from the air cooler condensate discharge point back into stripper A. The temperature of the condensed liquids was monitored by TT-3082 located between the air cooler and control valve PV-3087.

Condensate Cooling - A plateflow heat exchanger (300-E-01), shown in Figure 8, was provided to cool the condensate stream to approximately 80ºF. To provide this required cooling, 300-E-01 was serviced by cooling water at 50ºF and a flow rate of about 10 gpm. This cooling water was drawn from, and returned to, a closed loop chilled water circuit. The cooling water flowrate was set manually and monitored by flowmeter FT-3084.

The temperature of the cooled condensate was monitored by TT-3086, while the temperature of the cooling water in and out of 300-E-01 was recorded by TT-6022 and TT-3085, respectively.

Gravity Separator - Once cooled, the condensed heptane and water gravity flowed to the gravity separator (300-C-07), shown in Figure 8, where these co-condensed immiscible liquids were then allowed to separate based on the differences in their specific gravity (1.0 for water and 0.7 for heptane). The gravity separator utilized a level probe (LT-3121) to detect the location of the heptane/water interface. The output from LT-3121 was used to control the automatic valve LV-3121 which allowed water to drain from the feed side of the internal weir. In this manner the heptane/water interface was maintained at a level between the top of the weir and a set minimum level. This prevented both the carry over of water to the heptane side of the weir, and the discharge of heptane through LV-3121.

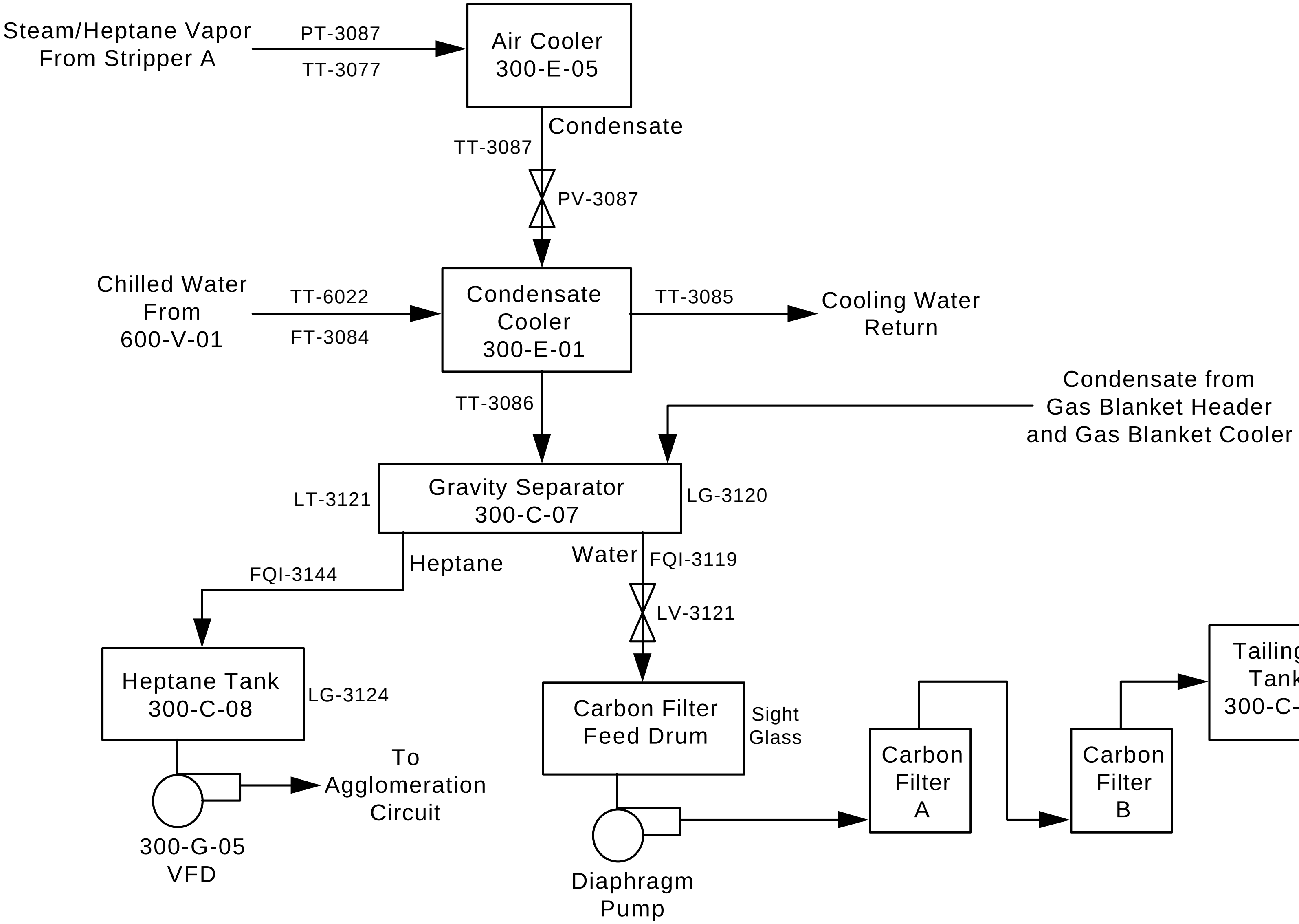


Figure 8. PDU Area 300 - Vapor Condensation, Cooling, and Recycle Circuits

Due to the intermittent nature of this control scheme, the recovered heptane and water flows were monitored on a cumulative basis over a given time period by flow totalizers FQI-3144 and FQI 3119, respectively. The gravity separator was also fitted with a sight glass (LG-3120) for confirmation of the level in the separator and a pressure relief valve (PSV-3122).

Heptane Storage - Once the heptane and water were separated in the gravity separator, the heptane flowed by gravity back to the heptane storage tank (300-C-08) as shown in Figure 8. This tank was of 4-foot 6-inch diameter and about 7 feet tall including a dished top and bottom. The effective operating volume of this tank was approximately 550 gallons, based on the elevation of the heptane return point located almost two-feet below the top of the vessel. The tank was fitted with a level gauge/transmitter (LT-3124) so that the level of heptane in the tank could be tracked. This level gauge also provided high and low level alarm setpoints. In addition, this tank was fitted with a thermocouple (TT-3128) to monitor the heptane temperature, and a pressure relief valve (PSV-3127).

Carbon Filters - Water from the gravity separator was returned to the process tailings tank after treatment in two carbon drum filters as shown in Figure 8. With the solubility of heptane in water in the 50 ppm range, these filters removed heptane from the recovered water stream. Also, in case of malfunction of the gravity separator, they prevented the introduction of large quantities of heptane to the process tailings circuit. The recovered water was drained through LV-3121 into the carbon filter feed tank (a 55-gallon drum). From this feed drum, the water was pumped through two 55-gallon carbon drum filters in series prior to discharge to the tailings surge tank (300-C-10).

Tailings Handling

The tailings handling circuit is shown in Figure 9. Final process tailings from the froth skimmer (300-C-04) underflow piping gravity flowed to the tailings surge tank 300-C-10. This tank was of 4-foot diameter, with a cylindrical section about 3-feet high, a dome shaped top, and a sloped flat bottom. The total capacity of 300-C-10 was on the order of 400 gallons with a typical operating capacity of about 250 gallons. Tailings from 300-C-10 were pumped out of area 300 via a fixed speed centrifugal pump (300-G-07). A level gauge/transmitter (LT-3149) located on the tailings surge drum was used to maintain a constant level in the vessel by controlling the flow control valve (LV-3149) on the tailings pump discharge piping.

A magnetic flowmeter (FT-3055) was located on the tailings discharge line to monitor the tailings flowrate. Under normal operating conditions, i.e., when the tailings were free of heptane contamination, the process tailings were pumped out of Area 300 to the Area 300 tailings tank (200-D-02) for disposal.

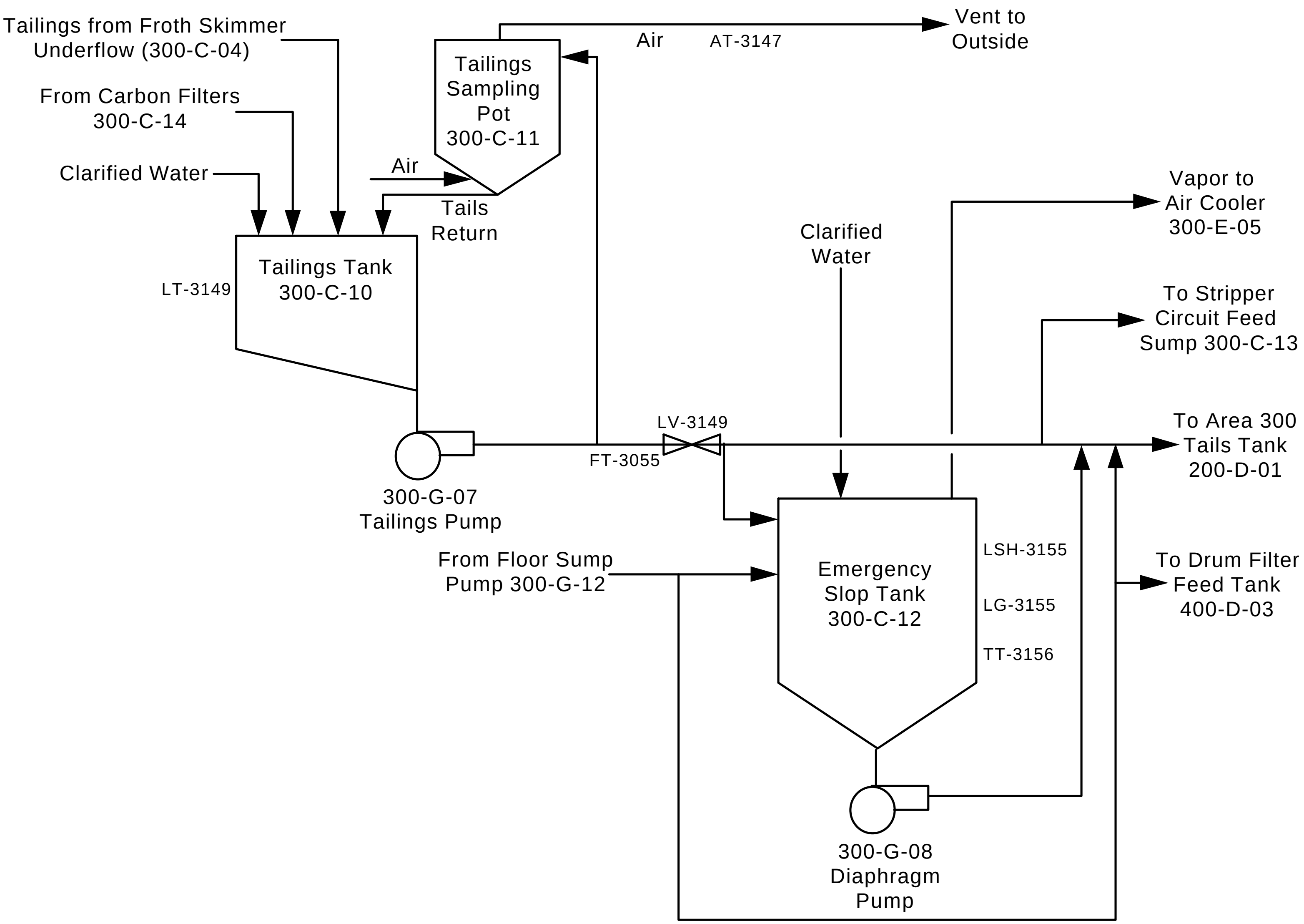


Figure 9. PDU Area 300 - Process Tailings Circuit

Tailings Sampling Pot - In the case of an upset condition in the agglomeration/screening/froth skimming circuit that resulted in contamination of the tailings stream with heptane, this flow was diverted for steam stripping treatment prior to disposal. Checking of the tailings stream for heptane contamination was carried out continuously by the diversion of a recycle tailings stream to the tailings sampling pot (300-C-11) as shown in Figure 9.

This tailings sampling pot had dimensions of 1-1/2 feet by 1-1/2 feet by 2-3/4 feet high and was fitted with angled baffles so that the tailings stream entering the top of this vessel cascaded to the bottom from which it discharged back to the tailings surge drum 300-C-10 by gravity flow. During this cascade flow through 300-C-11, the tailings stream flow was countercurrent to a continuous air purge stream flowing into the bottom of the vessel and out the top.

The contact time between the tailings and air purge stream was sufficient for any heptane present to evaporate into the air stream. The exiting air stream was then checked for heptane concentration by hydrocarbon detector AT-3147 before venting outside. When this alarm triggered, the tailings flow was diverted to the emergency slop tank until the process upset condition causing the tailings contamination with heptane was corrected. Another hydrocarbon detector (AT-3205), with the same alarm configuration, was located directly above the downstream tailings sump located outside of Area 300.

Emergency Slop Tank - The emergency slop tank (300-C-12), shown in Figure 9, was a vessel installed in Area 300 to handle any material contaminated with heptane such that it could not be discharged from Area 300 for typical dewatering and disposal procedures. Sources of material sent to the emergency slop tank included:

- Any process tailings stream contaminated with heptane
- Any accidental process spill to the floor of Area 300 contaminated with heptane
- Any deliberate process spill to the floor of Area 300 contaminated with heptane, i.e., as generated during the emptying of a vessel for service
- Any agglomerated coal drained from the agglomeration circuit reactors
- Any Area 300 wash down material contaminated with heptane

Details of the emergency slop tank (300-C-12) were as follows:

- Dimensions of 8-foot diameter, 5-foot cylindrical section, 3-1/2-foot tall cone bottom, and a dome top
- Capacity of approximately 2500 gallons
- Provided sufficient storage for 15 to 20 minutes of typical process tailings flow
- Sight glass LG-3155
- High level switch (LSH-3155)
- Steam inlet sparging loop

- 8-inch diameter view port
- Pressure relief valve (PSV-3154)
- Thermocouple (TT-3156)
- Clarified water addition point at the top of the vessel
- Capability to accept feed from tailings pump 300-G-07, floor sump pump 300-G-12, and from high and low shear agglomeration vessel drains

Once any material was present in the emergency slop tank it required steam stripping treatment prior to disposal. This steam stripping was typically carried out by pumping the material to the normal steam stripping circuit described previously.

Chilled Water Cooling Circuit

During typical SA module operation, heat input to the process consisted primarily of about 2000 to 4000 lb/hr of steam input along with the heat generated by the various process mixers and the grinding circuit. In order to help dissipate heat out of the system, a closed-circuit chilled cooling water system was included to supplement dissipation of heat by the air cooler during condensation of the evaporated heptane and water and by the discharge of final product cooling water to the sewer. This chilled cooling water circuit is shown in Figure 10.

The design of the chilled cooling water circuit called for the water to be chilled to 50°F and to return at 60°F. Chilling of this cooling water was carried out in water chiller (600-V-01) installed outside of Area 300. This closed circuit chilled cooling water circuit serviced three different heat exchangers in the process as follows:

- Approximately 25 gpm of chilled water to cool the gas blanket in the gas blanket cooler 300-E-04.
- Approximately 10 gpm of chilled water to cool the condensed heptane and water from the stripping circuit in heat exchanger 300-E-01.
- Approximately 175 gpm of chilled water to cool all of the clarified water utilized in both Areas 100 (grinding) and 300 (selective agglomeration) in the clarified water cooler 400-E-01.

As such, through these various cooling capabilities, the overall temperature of the PDU process and water streams were maintained at acceptable levels.

Nitrogen Blanketing System

Since heptane is a volatile and explosive compound, its use required a nitrogen blanket for health, safety, and environmental considerations. Under this system, all portions of the process in which heptane was present, were maintained under a positive pressure of 2 to 8 inches of water column with nitrogen gas. This insured that no air was drawn into the system allowing the formation of an explosive environment.

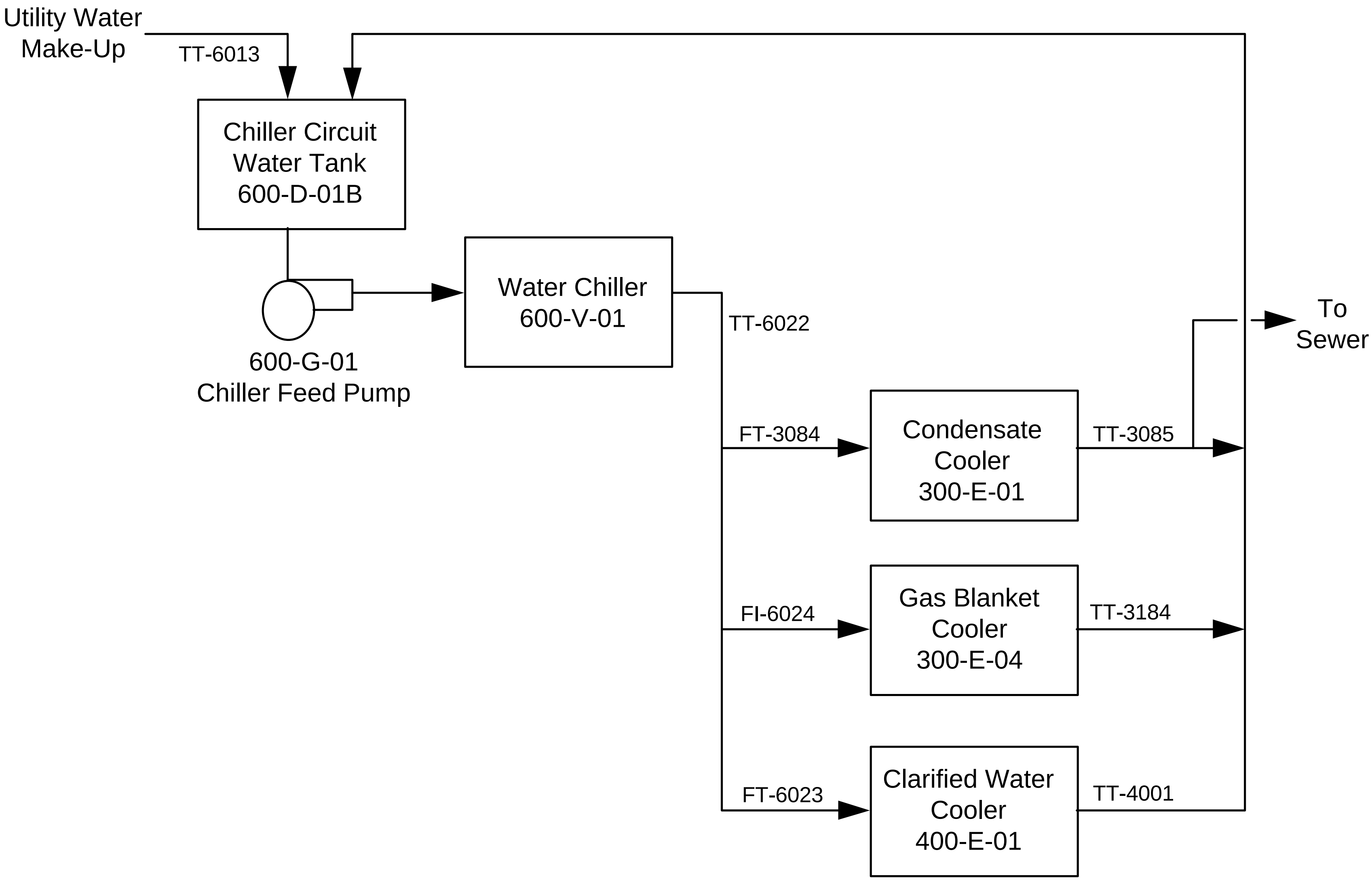


Figure 10. PDU Area 300 - Chilled Cooling Water Circuit

During implementation of the gas blanket system, all air was purged from the process and gas blanket until oxygen levels were below the explosive limit. After that, the oxygen content in the gas blanket system was continually monitored. The nitrogen blanketing system is depicted in Figure 11.

Nitrogen Supply - Nitrogen storage for use in the gas blanket system was in the liquid form in a 900 gallon storage tank. (300-V-01). Additional nitrogen was ordered as needed based on the tank's gauge reading. The discharge of the nitrogen tank was fitted with a 1000 SCFH ambient air evaporator to supply nitrogen gas to the process. The nitrogen gas supply was regulated into the nitrogen header at a pressure of between 8 and 15 psi. The nitrogen header was fitted with flowmeter FT-3175 for monitoring of nitrogen consumption. The header also contained a low pressure switch (PSL-3176) for indication of unexpected nitrogen pressure loss.

Gas Holder - A gas holder (300-D-04) was installed between the nitrogen header and the gas blanket header into the process. The purpose of the gas holder was three-fold. First, since the gas holder was a variable volume vessel, it provided surge capacity for the nitrogen blanket to the process, allowing the reuse of nitrogen, thereby reducing nitrogen consumption. Second, the gas holder maintained a relatively constant blanket gas pressure on the process by use of a fixed mechanical weight. Third, the gas holder provided relief capabilities via a rupture disc (set to relieve at 15-inches of water column pressure) to prevent excess gas blanket pressure in case of a failure of the associated control valves. The gas holder (300-D-04) was a 12-foot diameter by 10-feet high tank with variable volume capabilities. The internals of the tank included a 6000 lb plate attached to a bladder which was sealed around the circumference at the tank's top as shown in Figure 11. As the volume in the gas blanket system changed, due to increasing or decreasing levels or temperatures in process vessels and tanks, the plate floated up and down to compensate for the volume change.

When the gas blanket pressure reached a pre-determined maximum level, typically 6 to 10 inches of water, a control valve (LV-3187) was manually opened to release gas from the incoming gas blanket header to the relief system. Similarly, when the gas blanket pressure reached a predetermined minimum pressure, typically 1 to 2 inches of water, a control valve (LV-3188) was manually opened allowing fresh makeup nitrogen into the incoming gas blanket header from the nitrogen header. In this manner, a gas blanket pressure in the 1 to 10 inches of water column range was maintained.

Gas Blanket Cooler - Located between the gas holder and the gas blanket connections to the process was the gas blanket cooler (300-E-04), a shell and tube heat exchanger. The purpose of this exchanger was to condense any heptane vapors out of the gas blanket as the gas moved from the process to the gas holder. The blanket gas traveled through the shell side of the gas blanket cooler, while chilled cooling water from the water chiller (600-V-01) was circulated through the heat exchanger tubes. Typical chilled water flow to 300-E-04 was on the order of 25 gpm. Condensed heptane from this gas blanket cooler flowed by gravity to the gravity separator (300-C-07).

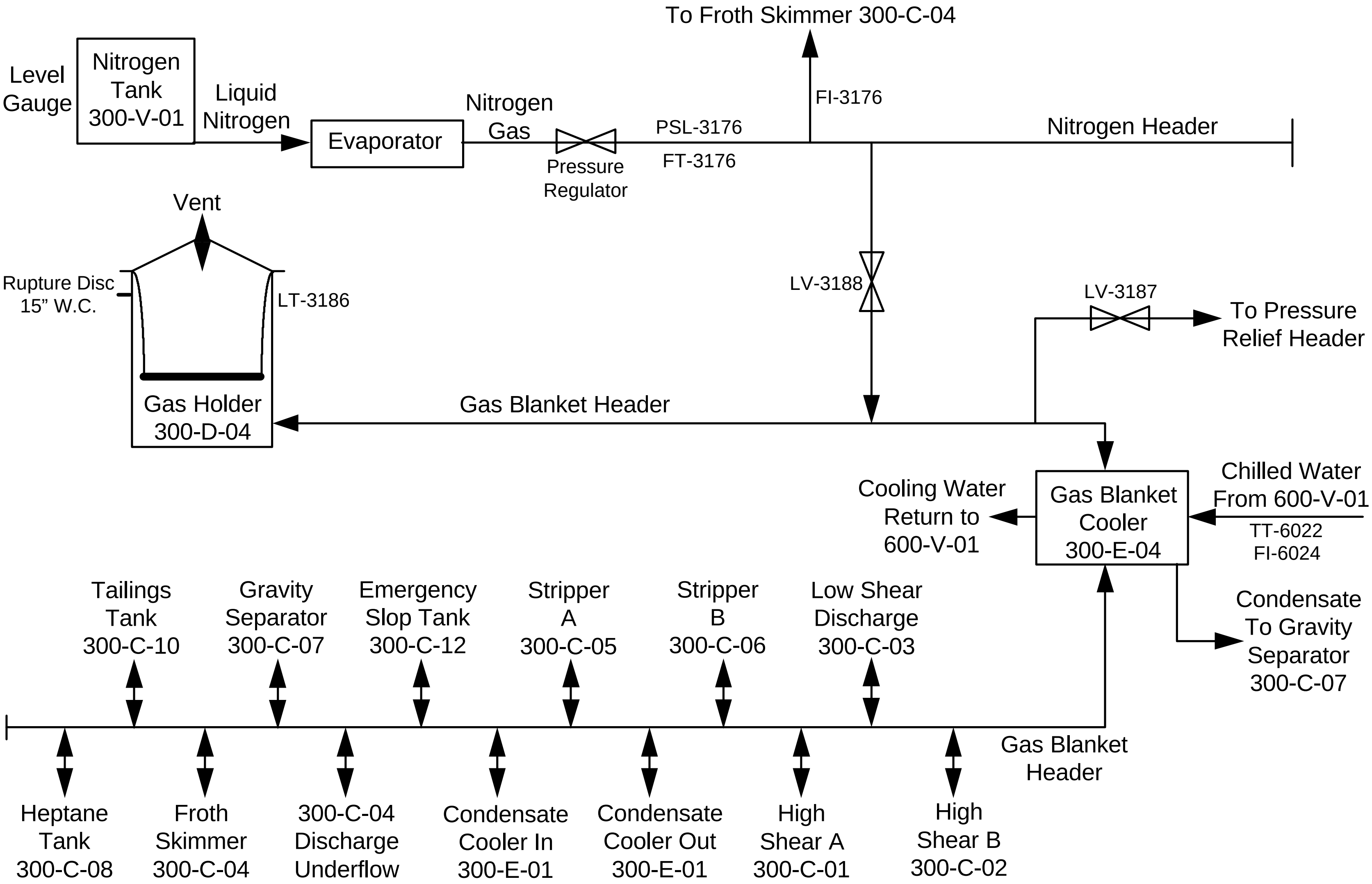


Figure 11. PDU Area 300 - Nitrogen and Gas Blanket System

Pressure Relief System

In case of a major process upset, explosion, or fire inside any Area 300 process vessel, tank, or piping, the SA module was equipped with a pressure relief system. Activation of the pressure relief system was through the opening of any of the eight pressure relief valves installed on various process vessels. Once a pressure relief valve was opened, the relieved material (gas, liquid, or solids) flowed to the main relief header which was connected to the knockout drum (300-C-15). Downstream of the knock-out drum was a flare (300-F-01), designed to burn any relieved hydrocarbons. The pressure relief system is shown in Figure 12.

As with the gas blanket system, prior to its implementation, the relief system was purged of all oxygen to prevent the formation of an explosive atmosphere within the relief system itself.

Pressure Safety Valves - The SA module was fitted with a total of ten pressure safety valves. These valves were of varying size and set to relieve at various pressures based on the anticipated vessel conditions and relief flows. In general, opening of any given pressure safety valve was activated by excessive pressure. This excessive pressure could be due to either a plugged process line or an explosion within the system. A list of the eight installed pressure safety valves which relieved to the knock-out drum and flare, along with their location, size, and pressure setpoints are as follows:

1. PSV-3021, high shear A (300-C-01), 1-1/2” x 2”, 30 psi
2. PSV-3029, high shear B (300-C-02), 1-1/2” x 2-1/2”, 20 psi
3. PSV-3039, low shear (300-C-03) overflow piping, 2” x 3”, 15 psi
4. PSV-3067, stripper A (300-C-05), 1-1/2” x 3”, 25 psi
5. PSV-3095, stripper B (300-C-06), 1-1/2” x 2-1/2”, 25 psi
6. PSV-3122, gravity separator (300-C-07, 1-1/2” x 3”, 15 psi
7. PSV-3127, heptane storage tank (300-C-08), 3” x 4”, 15 psi
8. PSV - 3154, emergency slop tank (300-C-12), 3” x 4”, 25 psi

In addition, there were two other pressure safety valves, PSV-3103 and PSV 3109, located on the discharge process piping of product slurry cooling heat exchangers 300-E-02 and 300-E-03, respectively. Since there was no heptane at the location of these valves, they did not relieve to the relief header, but rather to the ground locally.

Knock-out Drum - All of the above listed pressure safety valves were connected to an 8-inch diameter header which ran outside to the knockout drum (300-C-15). The primary purpose of the knockout drum was to remove solids and liquid form the relief stream prior to its entering the flare. The knockout drum was fitted with a high level switch (LSH-3179) to indicate that it was full, and a drain to remove accumulated liquids and solids.

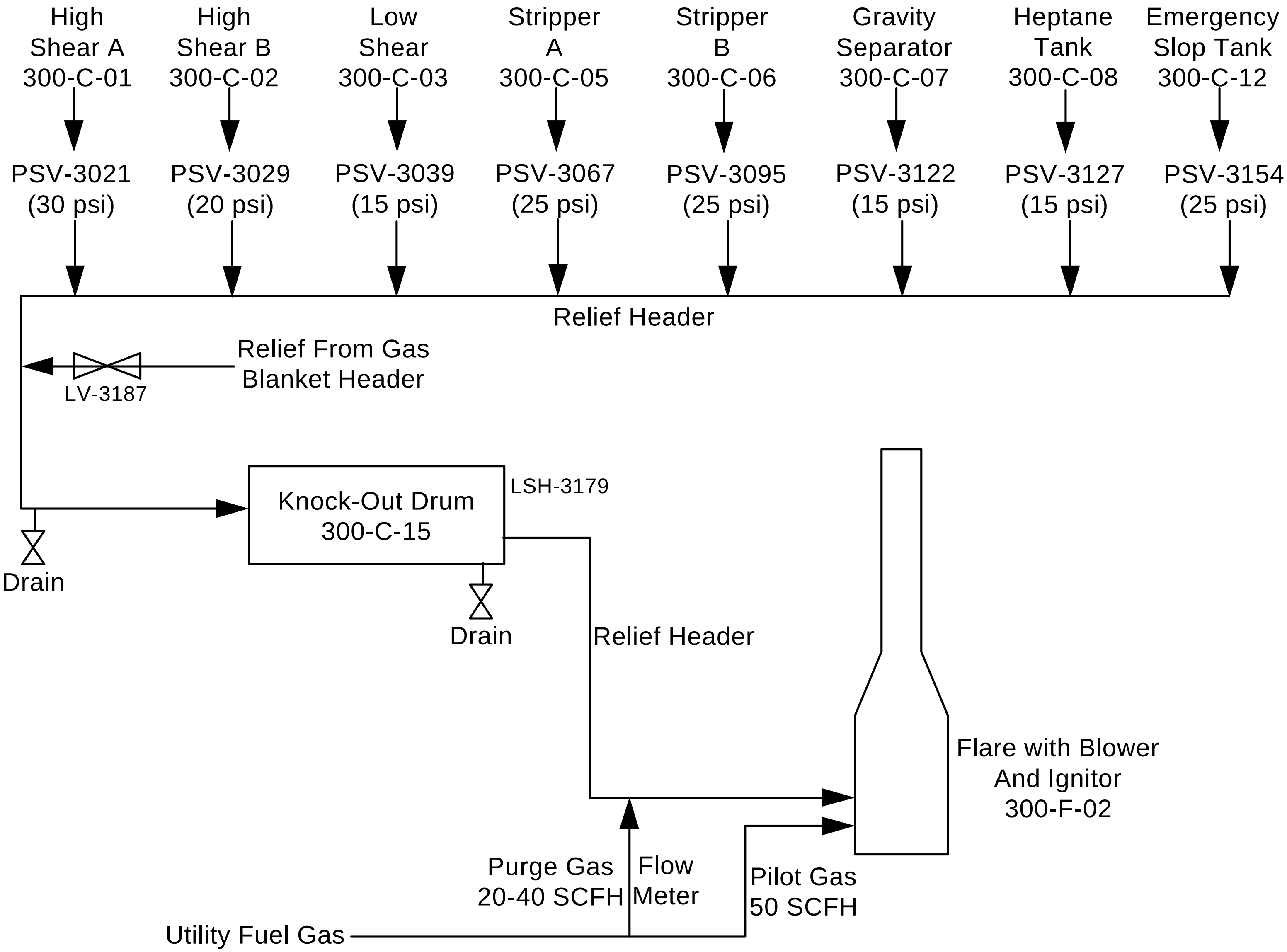


Figure 12. PDU Area 300 - Pressure Relief System

Flare - Once the relief stream passed through the knock-out drum, it entered the flare (300-F-01). This flare was 25-feet high and located 50-feet from the edge of the Area 300 building. The flare was designed to burn any relieved heptane vapors from the process.

This flare had a continuous natural gas fired pilot flame which burned at all times. Since there was no pilot monitor to insure that the pilot was lit, the flare was equipped with a continuous flare ignitor that fired for 2 seconds every 20 seconds. With this arrangement, if the pilot light blew out, it was automatically relit.

In addition, the flare was equipped with a velocity seal which required a continual purge of at least 20 SCFH of natural gas to prevent the diffusion of air into the flare, preventing the formation of an explosive atmosphere within the relief system piping and the flare itself.

Safety Features

In addition to the gas blanket and pressure relief systems discussed above, a number of other safety features were incorporated in to the SA module to insure safe operation of the plant.

Fire Protection - Area 300 of the SA module was equipped with a fire detection system that consists of the following:

- Rate of rise heat detectors
- Audible alarms
- Visible alarms
- Manual pull stations at all Area 300 exits
- Automatic notification system to the local fire department

Ventilation System - Area 300 was equipped with a continually operating ventilation system that provided a slight negative pressure. This ventilation system consisted of a two speed air handling unit that provided an incoming air flow rate of 1700 and 3000 SCFM at its low and high operating speeds, respectively. This unit had the capability to provide as received, heated, or cooled air to Area 300.

In conjunction with this air handling unit, a separate exhaust fan operated independently. The flows generated by this exhaust fan were 2000 and 3600 SCFM at its low and high operating speeds, respectively, providing a negative pressure inside the Area 300 building.

Normal operation of both the air inlet and exhaust units were at low speed. Control of these units was also tied in with alarms based on the hydrocarbon analyzers located throughout Area 300. If an area hydrocarbon or oxygen analyzer alarmed, the ventilation system automatically switched to high speed operation. When the alarm cleared, the ventilation system automatically returned to low speed. It should also be noted that the ventilation system was not connected to the emergency shutdown controls, so when the plant was shut down in an emergency situation, the ventilation system continued to operate.

Hydrocarbon and Oxygen Detectors - As part of the plant safety features, the SA module process was equipped with a number of analyzers, set up to detect the presence of heptane in the Area 300 atmosphere, the presence of oxygen in the gas blanket system, and a deficiency of oxygen in the Area 300 atmosphere. Hydrocarbon detectors were installed to detect fugitive heptane vapors within and outside of Area 300 at a number of locations. Heptane is heavier than air and therefore would tend to concentrate in the lower level of the plant. Following is a list of these detector tag numbers along with their locations:

- AT-3201 - At grade near the floor sump
- AT-3202 - At grade in the northeast corner of Area 300
- AT-3203 - At grade in the center of the room
- AT-3204 - In the air exhaust duct on the roof
- AT-3205 - Above the Area 300 Tails Sump 200-D-02 outside of Area 300

Operation of the first four of these detectors (all except AT-3205) was as follows based on a low explosion limit (LEL) of 1% heptane in air:

- When 10% of the LEL was reached, a slow intermittent audible alarm sounded and the ventilation system shifted to its high speed.
- When 20% of the LEL was reached, the audible alarm became continuous and continued until acknowledged by the operator.
- When 30% of the LEL was reached, a rapid audible alarm sounded. If this alarm was not acknowledge by the operator or the alarm condition reversed, the entire plant (with the exception of the ventilation system and gas blanket controls) was shut down in two minutes.

Operation of hydrocarbon detector AT-3205, located directly above the Area 300 tails sump outside of Area 300, was the same as for the previously discussed detector AT-3147 that analyzed for heptane contamination in the Area 300 process tailings. If heptane was detected, an intermittent and then continuous audible alarm sounded at two heptane concentration levels. Upon triggering of this alarm, the tailings flow was diverted to the emergency slop tank until the process upset condition causing the tailings contamination with heptane was corrected.

One oxygen detector was installed near the high point of Area 300 roof to detect a deficiency of oxygen in the atmosphere caused by a buildup of nitrogen in Area 300. This detector was designated as AT-3205. The interlocks associated with this detector's alarm conditions were as follows:

- At 19.5% O_2, a constant audible alarm sounded and the ventilation system shifted to high speed.
- At 16% O_2, a rapid audible alarm sounded and an automatic emergency shutdown commenced.

One oxygen detector was installed to detect a buildup of oxygen in the gas blanket system and was designated as AT-3186. A buildup of oxygen in the gas blanket system could have occurred due to continuous expulsion of air dissolved in the coal water slurry feed to the plant, oxygen present in the incoming steam, or a loss of gas blanket pressure. The interlocks associated with this detector's alarm conditions were as follows:

- At 4% O_2, a slow audible alarm sounded.
- At 8% O_2, a constant audible alarm sounded.

Alarms - In addition to those alarms associated with the hydrocarbon and oxygen detectors discussed above, this section of the report discusses those alarms associated with normal operation of Area 300. Table 11 presents a list of alarms programmed into the DCS system which indicated a failure in either a piece of equipment or in a DCS control loop.

Included in Table 11 are:

- The instrument associated with the alarm.
- The type of instrument and the units involved.
- A description of the process stream.
- The low and high level alarm points indicated as either an actual measurement, or if preceded by a "+" or "-" as a differential from the operating setpoint.

Other alarms associated with the Area 300 operation that do not relate to particular setpoints are as follows:

1. LSH-3155 indicated a high level in the emergency slop tank 300-C-12. When this alarm triggered, filling of the slop tank was manually stopped.
2. LSH-3039 indicated a liquid level in the low shear overflow piping i.e., a plugged low shear discharge port. When this alarm triggered, the plug was cleared immediately, the alternate low shear discharge port opened, or the agglomeration feed stopped.
3. LSH 3179 Indicated a high liquid level in the knockout drum. When this alarm triggered, the liquid was drained from the knockout drum and the reason for its presence determined.
4. XSH-3181 indicated a pilot flame ignitor fault which was caused by either low voltage in the pilot ignitor spark or the pilot controller switch being in the off position.

Table 11. PDU Area 300 DCS System Alarms

Instrument	Type	Description	Low	High
FT-3014	Flow - gpm	Agglomeration Feed	-5	+5
FT-3046	Flow - gpm	Screen Spray	5	--
FT-3065	Flow - gpm	Stripper A Feed	-5	+5
FT-3090	Flow - lb/hr	Steam Main	1500	5500
FT-3115	Flow - gpm	Cooling Water to Sewer	--	65
FT-6023	Flow - gpm	Chilled Cooling Water to 400-E-01	100	--
LIT-3124	Level - %	Heptane Storage Tank	25	75
LIT-3149	Level - %	Tailings Surge Tank	25	75
LT-3001	Level - %	Slurry Storage Tank North	15	90
LT-3002	Level - %	Slurry Storage tank South	15	90
LT-3059	Level - %	Stripper Feed Tank	25	75
LT-3073	Level - %	Stripper A	50	85
LT-3097	Level - %	Stripper B	25	85
LT-3121	Level - %	Gravity Separator	25	75
LT-3186	Level - %	Gas Holder	40	60
PSH-3104	Press. - psi	300-G-04 Discharge	--	80
PSL-3176	Press. - psi	Nitrogen Header	10	--
PT-3015	Press. - psi	Agglomeration Feed	--	40
PT-3087	Press. - psi	Stripper A	0.5	5
PT-3090	Press. - psi	Main Steam Supply	19	25
PT-3092	Press. - psi	Stripper B	--	25
TT-3071	Temp - ºF	Stripper A Slurry	--	230
TT-3077	Temp - ºF	Stripper A vapor	--	230
TT-3093	Temp - ºF	Stripper B Slurry	--	265
TT-3096	Temp - ºF	Stripper B Vapor	--	265
TT-3111	Temp - ºF	Cooling Water to Sewer	--	110
TT-3110	Temp - ºF	Final Product Temperature	--	120
TT-6022	Temp - ºF	Chilled Water	--	65

Control System Interlocks - A number of automatic interlocks were programmed into the DCS control system for operation of the PDU SA module as follows:

1. The heptane pump 300-G-05 would not start if it was in auto mode, unless the agglomeration feed pump 300-G-01 was running.
2. When the heptane pump 300-G-05 was in auto mode, it stopped when the agglomeration feed pump 300-G-01 stopped.
3. When the agglomeration feed pump 300-G-01 started, the high shear circuit gas blanket isolation valve LV-3008 closed, and when 300-G-01 stopped, LV-3008 opened.
4. When the tailings pump 300-G-07 stopped, its discharge control valve LV-3149 closed and when 300-G-07 started, LV-3149 opened.
5. When stripper B feed pump 300-G-02 started, its discharge automatic isolation valve UV-3074 opened, and when 300-G-02 stopped, UV-3074 closed.

6. When the final slurry pump (300-G-04) discharge high pressure switch PSH-3104 alarmed, pump 300-G-04 switched to manual mode, the current stripper B level became the new control loop setpoint, and the pump was not allowed to increase in speed. Pump 300-G-04 then returned to auto mode when the PSH-3104 alarm condition cleared, utilizing the current stripper B level as the new control loop setpoint.
7. The water chiller 600-V-01 would not start unless flow was confirmed by its discharge flow switch.

Area 400 - Dewatering Circuit

The dewatering circuit was very important to the continuous operation of the PDU SA module. Dewatering, or solid-liquid separation, produced dry (relative) filter cake and water (filtrate). The filter cake was the product of the plant and the water (filtrate) was used over and over again in the process. Each product, coal and tailings, was dewatered separately to ensure that no cross contamination occurred. A flowsheet of each area is shown separately in Figures 13 and 14, respectively.

The clean coal was dewatered in a circuit which utilized three filters. A WesTech vacuum drum filter (400-Y-09) was used as the primary filtration unit while two Netzsch pressure filter presses (400-Y-04 and 400-Y-05) filtered the remaining clean coal product. Clean coal which entered the drum filter feed sump (400-D-03) was stored for a short period of time before being pumped to the units. A diaphragm pump (400-G-08) was used for transferring the clean coal slurry to the WesTech filter. The unit, which produced a cake continuously at a rate of approximately 2,000 lb/hr (dry solids), discharged the product into a supersack for storage or disposal. The remaining clean coal product, along with the filtrate from the drum filter was dewatered by the two Netzsch plate and frame filter presses.

The coal which was stored in the Netzsch filter feed sump could be pumped to either filter by the Netzsch piston pumps (400-G-04 and 400-G-05). The filter cake produced by these units was discharged onto dedicated conveyor belts (400-T-03 and 400-T-04) and into supersacks for storage or disposal. The filtrate from both units was collected in a common filtrate sump (400-D-06) from where a vertical sump pump (400-G-06) transferred it to the thickener (400-D-01) for treatment.

Tailings from the SA module were sent to an Enviro-Clear thickener (400-D-01) for initial dewatering. Cationic and anionic polymers were added to the tailings stream to accelerate the particle settling rate to approximately 12 inch/minute. The thickened solids fell to the bottom of the thickener tank and formed slurry of approximately 20 to 30% solids. The clarified water overflowed the top of the unit where it was collected in the clarified water sump (400-D-02) and reused in the process.

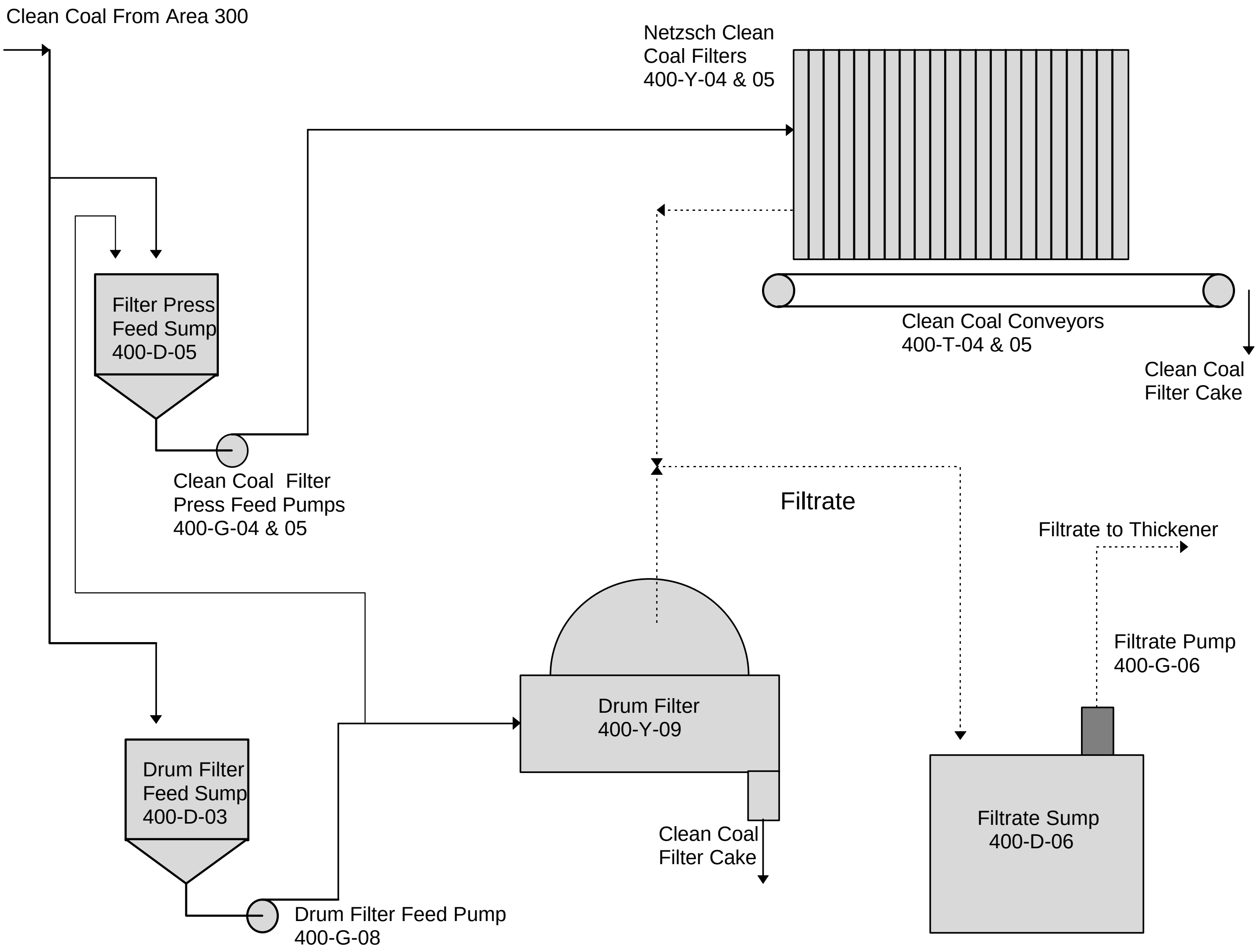


Figure 13. PDU Area 400 - Clean Coal Dewatering Circuit

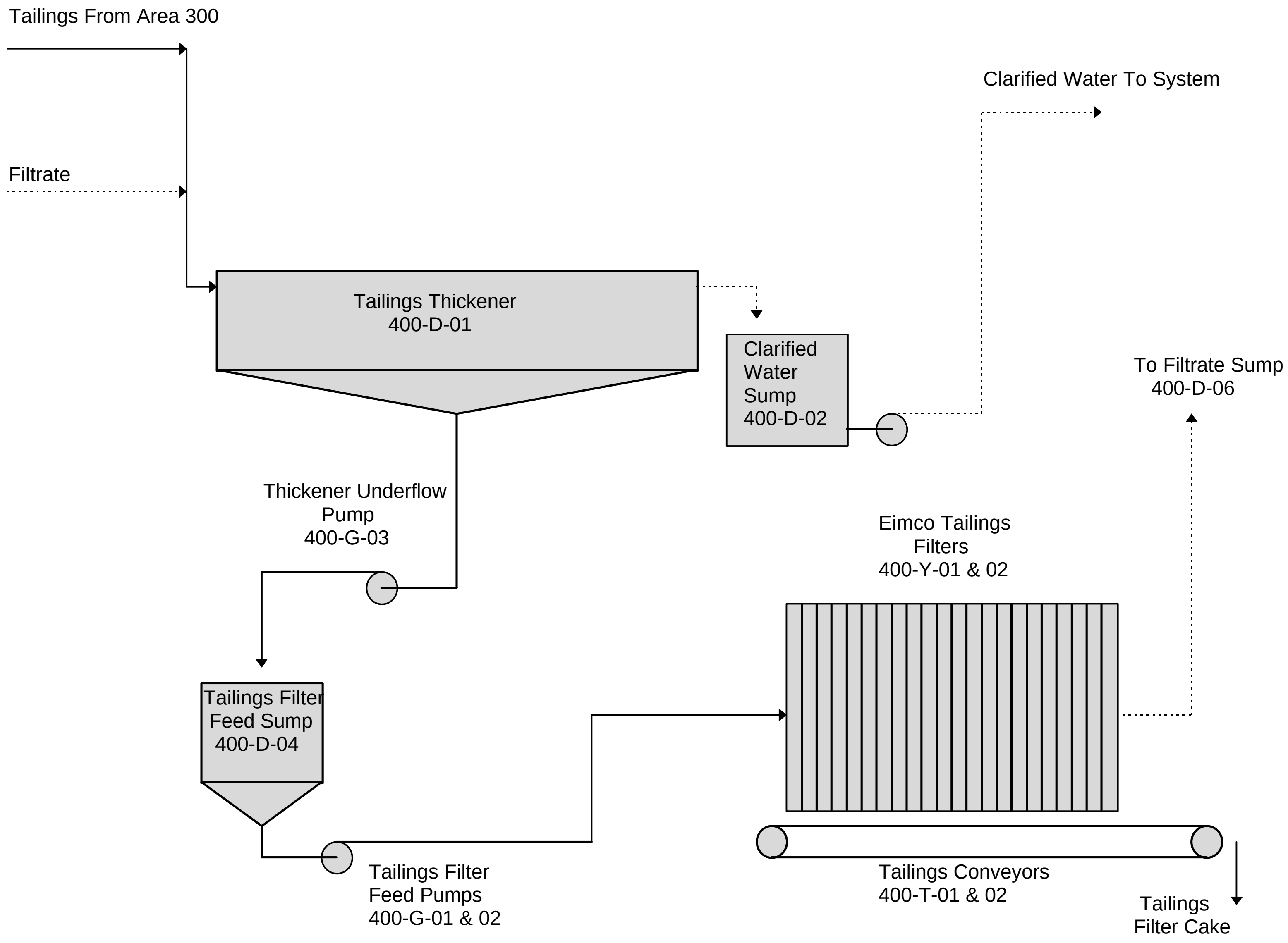


Figure 14. PDU Area 400 - Tailings Dewatering Circuit

The thickener underflow pump (400-G-03) transported the thickened tailings to the tailings filter press feed sump (400-D-04). Here, two air operated diaphragm pumps (400-G-01 and 400-G-02) transferred the material, under pressure, to two Eimco pressure filters (400-Y-01 and 400-Y-02). The filter cake was manually discharged from each unit for disposal in supersacks. The tailings filtrate was either combined with the clean coal filtrate before being sent to the thickener (400-D-01), or sent directly to the clarified water tank.

TASK 9.2 - PDU SA MODULE SHAKEDOWN AND TEST PLAN

Startup and shakedown of the PDU SA module was completed during the last quarter of 1996 and first quarter of 1997 according to the Subtask 9.2 SA Module Shakedown and Testing Plan [10]. Though some minor operating difficulties were encountered, corrective actions resulted in a fully functional PDU SA Module. Physical and mechanical improvements resulted in the elimination of process bottlenecks which allowed the PDU SA Module to operate at steady-state conditions.

PDU SA Module Operating Personnel

The PDU SA module required many different crafts and skills to properly operate and maintain the equipment. Since research and development was the main thrust behind this project, technicians who possessed strong operating, maintenance, and analytical skills were utilized.

The staffing schedule shown in Table 12 was utilized during Subtask 9.2 and 9.3 operations.

Table 12. PDU and SA Module Staffing Schedule

Staff Position	Primary Responsibility
Operator - Area 100	Monitor/operate grinding and classification circuits
Operator - Area 300	Monitor/operate SA module
Operator - Area 400	Monitor/operate dewatering circuit
Control Room Operator	Monitor PDU status, collect and summarize test data
Laboratory Technicians	Prepare samples and perform sample analyses
Project Engineer	Supervise operation of PDU SA module
Process Engineer	Design test plan, evaluate results, and prepare reports

This team of eight engineers, operators, and technicians assured safe effective operation of the PDU and completion of the test program, including production of 100 ton lots of clean coal. Details of the duties performed by the PDU personnel are included in the Subtask 9.2 test plan [10].

PDU SA Module Procedure Development

In order to provide guidelines for the safe and proper startup of the PDU SA module, a number of operating procedures were developed. These procedures were followed, and revised as necessary, during the start-up and shakedown testing of the SA Module. Due to the presence of the Area 300 slurry feed storage tanks and the Area 400 filter feed storage tanks, the startup procedures for the three plant Areas 100, 300, and 400 could be carried out independently and were therefore developed in this manner. Details of the following operating procedures can be found in the Subtask 9.2 Start-Up and Shakedown Test Plan [10]:

- Area 100 Operating Procedures
    - Area 100 Pre-Start Procedure
    - Area 100 Startup Procedure
    - Area 100 Steady-State Operating Procedure
    - Area 100 Shutdown Procedure
- Area 300 Nitrogen Purging Procedures
    - Blanket System Purge Procedure
    - Process System Purge Procedure
    - Relief System Purge Procedure
- Area 300 Operating Procedures
    - Area 300 Pre-Start Procedures
    - Area 300 Initial Coal/Heptane Startup Procedure
    - Area 300 Startup Procedure
    - Area 300 Steady-State Operating Procedures
    - Area 300 Shutdown Procedure
- Area 400 Operating Procedures
    - Area 400 Pre-Start Procedure
    - Area 400 Start-up Procedure
    - Area 400 Steady-State Operating Procedure
    - Area 400 Shutdown Procedure
- Control Room Steady-State Operating Procedure
- Area 300 Emergency Shutdown Procedures
    - Loss of Instrument Air Emergency Shutdown Procedure
    - Loss of Power Emergency Shutdown Procedure
    - Fire Emergency Shutdown Procedure
    - Heptane Leak Emergency Shutdown Procedure

PDU SA Module Equipment Start-Up and Shakedown

The following list of checks, where appropriate, were completed for each piece of PDU SA Module equipment prior to plant start-up:

1. Check for proper physical installation of unit and associated piping.
2. Check for proper electrical installation of unit.
3. Check for any air, water, or lubricant leaks.
4. Check shaft alignment.
5. Check for proper lubrication of motor, bearings, gearboxes, and seals.
6. Check level of hydraulic fluid
7. Check drive belt alignment and tension.
8. Check for proper rotation of all motors.
9. Check operation of empty unit when applicable.
10. Check operation of unit with water when applicable.
11. Check operability of any associated low and high level sensors.
12. Check operability of any associated low and high pressure sensors.
13. Check operating range of empty unit when applicable.
14. Check operating range of full unit.
15. Check operation of variable frequency drive.
16. Charge grinding mills with media as required.
17. Calibrate unit as required.

The following check list was completed during shakedown testing for each piece of the PDU SA Module instrumentation:

1. Confirmation of communication between instrument and the DCS system.
2. Confirmation of 4-20 milliamp signal to and from instrument where applicable.
3. Calibrate instrument.

Shakedown testing of all the Proportional Integral Derivative (PID) loop controls found in the PDU, as listed below, involved confirmation that the control loop was operating properly tuned to some degree:

- Area 100 Control Loops
  - Total Area 100 clarified water flow (FT-104) via flow control valve FV-206
  - Classifying cyclone dilution water flow (FIT-114) via control valve FV-114
  - Ground product sump level (LT-118) via pump 100-G-04 VFD
  - Ground product dilution water flow (FT-117) via flow control valve FV-118
- Area 300 Control Loops
  - Slurry storage feed sump level (LT-201) via pump 200-G-01 VFD

  - Agglomeration feed flow (FT-3014) via pump 300-G-01 VFD
  - Heptane pump (300-G-05) speed via nuclear density gauge DIT-3012 percent solids and agglomeration feed flow (FT-3014) gpm outputs
  - Heated clarified dilution water flow (FT-3108) via flow control valve FV-3108
  - Stripper A level (LT-3073) via pump 300-G-02 VFD
  - Stripper A pressure (PT-3087) via pressure control valve PV-3087
  - Stripper B level (LT-3097) via pump 300-G-04 VFD
  - Stripper B pressure (PT-3092) via pressure control valve PV-3092
  - Steam mass flow to process (FT-3090) via flow control valve FV-3090
  - Stripper A slurry temperature (TT-3071) via steam flow control valve FV-3090
  - Utility cooling water to 300-E-03 flow (FT-3115) via flow control valve TV-3110
  - Gravity separator interface level (LT-3121) via flow control valve LV-3121
  - Tailings surge drum level (LT-3149) via flow control valve LV-3149
  - Gas holder plate level (LT-3186) via flow control valves LV-3187 and LV-3188
  - Area 300 tailings sump level (LT-213) via pump 200-G-03 VFD
- Area 400 Control Loops
  - Clarified water sump level (LT-402) via flow control valve LV-402
  - Tailings filter feed sump level (LT-411) via flow control valve LV-411

PDU SA Module Test Plan

The PDU SA Module Test Plan [10] was completed and submitted to DOE for review and approval on December 31, 1996.

The primary goal of the plan was to develop a testing procedure that would insure that the project objectives were met. This included parametric, optimization, and production testing of the PDU SA module with each of the three project coals (Hiawatha, Taggart, and Indiana VII), in that order. The testing was designed to identify the operating parameters which would enable the selective agglomeration process to recover at least 80 percent of the energy contained in the Run-Of-Mine (ROM) coal while producing a clean coal product with less than 0.6 lb sulfur/MBtu and less than 1-2 lb ash/MBtu.

The testing philosophy developed in the test plan was broken down into the following general categories, which would be carried out for each project coal:

- Agglomeration circuit unit operations evaluation
- Agglomerate recovery circuit evaluation
- Steam stripping circuit evaluation

Table 13 presents an outline of the parametric testing planned for evaluation of the agglomeration circuit.

Table 13. Parametric Agglomeration Circuit Testing

Task Description	Days	Set Points/Day	Set Points
Heptane/Coal Ratio for High Shear Inversion			
Fixed solids concentration			
Fixed impeller tip speed			
Residence Time (3)			
Heptane Level (3)	1	9	9
Minimize High Shear Energy			
Solids concentration (2)			
Residence time (3)			
Impeller tip speed (2)	2	6	12
Low Shear Evaluation			
Fixed high shear conditions			
Low shear solids concentration (2)			
Low shear residence time (2)			
Low shear impeller tip speed (2)	2	4	8
Summary	5	--	29
Repeat Days of Testing and Setpoints	1	4	4
Total Days of Testing and Setpoints	**6**	**--**	**33**

Table 14 presents an outline of the parametric testing planned for evaluation of the agglomerate recovery circuit.

Table 14. Parametric Agglomerate Recovery Circuit Testing

Task Description	Days	Set Points/Day	Set Points
Screen Parameter Evaluation Consistent low shear discharge Screen deck inclination (3) Screen spray flow rate (3)	3	3	9
Froth Skimmer Parameter Evaluation Consistent low shear discharge Consistent screen operating conditions Froth skimmer liquid operating level (2) Froth skimmer nitrogen purge rate (2)	1	4	4
Summary	4	--	13
Repeat Days of Testing and Setpoints	1	3	3
Total Days of Testing and Setpoints	**5**	**--**	**16**

Table 15 presents an outline of the parametric testing planned for evaluation of the steam stripping circuit.

Table 15. Parametric Stripping Circuit Testing

Task Description	Days	Set Points/Day	Set Points
Stripping Circuit Feed % Solids Evaluation			
Consistent agglomerate recovery product			
Fixed stripper A operating parameters			
Fixed stripper B operating parameters			
Feed solids concentration (3)	1	3	3
Stripper A Operating Parameters Evaluation			
Fixed feed solids concentration			
Fixed stripper B operating parameters			
Stripper A residence time (2)			
Stripper A operating temperature (2)			
Steam/coal ratio (2)	2	4	8
Stripper B Operating Parameters Evaluation			
Fixed feed solids concentration			
Fixed stripper A operating parameters			
Stripper B residence time (3)			
Stripper B operating temperature (3)	3	3	9
Summary	6	--	20
Repeat Days of Testing and Setpoints	1	3	3
Total Days of Testing and Setpoints	**7**	**--**	**23**

Following completion of parametric testing of each coal, and prior to each production run, at least one day of optimization/confirmation testing was carried out. This testing was used to reevaluate those operating parameters revealed as most critical during the parametric testing, and to confirm that the production run would provide the results anticipated.

After the optimization/confirmation testing, the plant was operated around the clock at the optimized conditions for the clean coal production run.

TASK 9.3 - PDU SA MODULE OPERATION AND CLEAN COAL PRODUCTION

Phase III of this project involved the construction and operation of a 2 t/hr selective agglomeration (SA) PDU module. This SA module was integrated with the existing PDU facility constructed during Subtask 8.2 and operated under Subtask 8.4.

During operation of the SA module, the existing coal handling and grinding circuits (Plant Area 100) were used to produce ground coal slurry feed for the selective agglomeration process. Similarly, the existing product and tailings dewatering circuits (Plant Area 400) were also used. As such, the SA module (Plant Area 300) essentially replaced the Microcel™ flotation column (Plant Area 200), with the remainder of the plant remaining intact.

Just like the advanced flotation PDU, selective agglomeration process performance was optimized at the 2 t/hr scale, and bulk lots of ultra-clean coal were produced for each of the three test coals. Toxic trace element distributions were also determined during the production runs. The ultra-clean coals were delivered to a DOE designated contractor, Penn State, for end-use testing.

Test Coal Feedstock Characterization

Characterization of three test coals used during the Subtask 9.3 operations were completed by an outside laboratory. Table 16 presents the results of this analyses. Included in Table 16 are the preparation plant yield and Btu recovery values achieved at the mine-site preparation plant for the Taggart and Indiana VII coals. The Hiawatha coal was not washed at the mine site.

Table 16. Properties of PDU Coals

	Taggart		Indiana VII		Hiawatha	
	As-Received	Bone Dry	As-Received	Bone Dry	As-Received	Bone Dry
Proximate, %:						
Ash	3.30	3.50	7.94	9.55	7.75	8.20
Volatile Matter	32.13	34.12	27.36	32.92	40.02	42.35
Fixed Carbon	58.73	62.38	47.81	57.53	46.72	49.45
Moisture	5.84		16.89		5.51	
Sulfur, %:						
Total	0.61	0.65	0.42	0.51	0.49	0,52
Pyrite	0.05	0.05	0.12	0.15	0.07	0.07
Sulfate	< 0.01	< 0.01	0.01	0.01	< 0.01	< 0.01
Organic	0.56	0.60	0.29	0.35	0.42	0.45
Ultimate, %:						
Carbon	80.30	85.28	62.40	75.39	72.93	77.18
Hydrogen	4.66	4.95	3.94	4.74	4.92	5.21
Nitrogen	1.38	1.47	1.40	1.68	1.37	1.45
Oxygen	3.91	4.15	6.75	8.13	7.03	7.44
HHV, Btu/lb	13,874	14,735	10,828	13,028	12,725	13,647
Equil. Moist., %	2.6		14.5		4.3	
Density, kg/m^3		1,260		1,360		1,275
HGI	49		54		44	
Coal Rank	hvA		hvC		hvA	
Preparation Plant Yield, %		57.2		61.9		100.0
Preparation Plant Btu Rec, %		84.9		90.5		100.0

Hiawatha Coal Parametric Testing Results

Initial testing of the PDU SA Module, including start-up and shakedown testing, was carried out with the Hiawatha coal. Following completion of the start-up and shakedown testing, work began on the parametric testing. Parametric testing for the Hiawatha coal focused primarily on evaluation of the following unit operations:

- High-shear agglomeration
- Low-shear agglomeration
- Vibrating screen
- Froth skimmer
- Steam stripping

Results for all of the Hiawatha coal PDU selective agglomeration module start-up and parametric testing are presented in Appendix A along with the results for the Hiawatha coal production run.

Start-up Testing

Prior to and during the high-shear evaluation testing, two complete plant test runs were completed to confirm continuous operation of the entire selective agglomeration module.

The following operating conditions were held relatively constant for both of these tests:

- Feed solids concentration - 10%
- High-shear circuit configuration and impeller tip speed - HS A only @ 18m/s
- Low-shear vessel configuration and impeller tip speed - Half full @ 5m/s
- Stripping circuit feed solids concentration - 23%
- Stripper A operating temperature - 205°F
- Stripper B operating temperature - 229°F

Table 17 presents the pertinent operating conditions and results for these two tests. Both of these tests were successful runs confirming continuous operation of all plant equipment.

High-Shear Circuit Testing

The purpose of the high-shear evaluation testing was to determine the minimum shear rate required to achieved inversion, as well as the appropriate amount of total high-shear energy input required.

Table 17. Start-Up Testing Conditions and Results - Hiawatha Coal

Test ID	HS-1	H-4
Grind D80, microns	60.7	56.1
Dry Coal Feed Rate, lb/hr	3100	4352
Heptane %, maf coal basis	24.7	28.4
High-Shear Residence Time, sec	39	28
Low-Shear Solids Concentration, %	7.28	8.15
Low-Shear Residence Time, sec	140	111
Low-Shear Agglomerate Size, mm	.5-3	0-1
Screen Spray Rate, gal/t coal	89	229
Steam Feed, lb/gal slurry	2.24	1.7
Stripper A Residence Time, min	16.5	11.9
Stripper B Residence Time, min	23.6	16.2
Product Ash, lb/MBtu	2.31	2.51
Product Heptane, ppm dcb	1381	2019
Tails Ash, %	83.6	82.3
Yield, %	93.0	93.2
Btu Recovery, %	99.7	99.6

In order to expedite the high-shear evaluation testing, complete plant samples and operating conditions were not taken and/or recorded for this testing. In lieu of complete plant tests, evaluation terminated at the low-shear vessel discharge. As such, 1-liter low-shear product samples were taken and processed using a low-shear rinse procedure which involved a 2 minute rinsing of the low-shear sample contents on a 48-mesh screen in the laboratory. Following this rinsing, both the product (retained on screen) and the tailings (screen underflow) were analyzed for ash and a separation performance calculated. In general, this low-shear rinse procedure was carried out in duplicate, and an average of the two sets of results used for evaluation.

Six test runs (H-1, H-2, H-3, H-5, H-6, H-7), covering 19 operating setpoints were carried out to evaluate the high-shear unit operation for the Hiawatha coal.

Minimum Shear Rate Determination - Determination of the minimum shear rate requirements to maintain inversion at the high-shear circuit discharge was evaluated over 5 different test runs (H-1, H-2, H-3, H-5, and H-7) evaluating 14 sets of high-shear operating conditions with three repeats.

In the first three tests (H-1, H-2, and H-3), three different high-shear configurations, high-shear A and B vessels in series, high-shear B vessel only, and high-shear A vessel only were evaluated. For each of these configurations, and with all other operating conditions held relatively constant, the high-shear impeller tip speeds were incrementally decreased until inversion was lost at the high-shear circuit discharge.

While most of the operating conditions for this testing were held constant, the grind size did vary, becoming finer as the testing progressed. Approximate coal feed rates of both 3400 and 4200 lb/hr were used for this work.

For each of the test points, an evaluation of the inversion quality was made at the high-shear circuit discharge point. The inversion quality was ranked from very good (VG) to very poor (VP).

Table 18 presents selected operating conditions at which inversion was just maintained at the high-shear circuit discharge.

Table 18. High-Shear Conditions Required to Maintain Inversion - Hiawatha Coal

			High-Shear Energy Input	
Grind D_{80}, microns	High-Shear Residence Time, sec	Impeller Tip Speed, m/s	kwhr/Ton Feed Coal	kwhr/1000 Gallon Slurry
61.3	36	11.0	4.2	1.8
61.3	99	7.0	4.5	1.9
66.7	63	9.1	9.1	3.9
63.2	36	10.6	3.7	1.6
40.0	27	15.0	8.1	3.2
40.6	33	11.2	4.5	1.7

Based on this data, the following was concluded:

- Given sufficient residence time (99 seconds), a high-shear impeller tip speed of 7.0 m/s will maintain inversion for the Hiawatha coal.
- A total high-shear energy input between 3.7 and 9.1 kwhr/ton feed coal was required to maintain inversion at the various conditions tested.
- A total high-shear energy input between 1.6 and 3.9 kwhr/1000 gallon slurry was required to maintain inversion at the various conditions tested.

Additional evaluation of this data set also indicated that in order to maintain what was classified as “good” inversion, the following high-shear conditions were required:

- A total high-shear energy input in the 8 - 15 kwhr/ton coal range
- A total high-shear energy input in the 3 - 7 kwhr/1000 gallon slurry range
- High-shear Impeller tip speeds in the 11 - 15 m/s range

Total Energy Requirement Evaluation - To further evaluate the high-shear unit operation, one additional test series (H-6) was completed to determine the total energy required to maintain “good” inversion at the high-shear circuit discharge. For these tests, the following operating conditions were held constant while the coal feed rate was incrementally increased until the inversion was considered “very poor”:

- HS A only configuration
- 11.1 m/s impeller tip speed
- 10% solids concentration
- Grind D_{80} of approximately 40 microns

For each coal feed rate tested, the quality of inversion at the high-shear circuit discharge was recorded and low shear samples taken for laboratory rinse testing evaluation. Pertinent results for this test work are shown in Table 19.

Table 19. Minimum HS Energy Requirements for Inversion - Hiawatha Coal

Coal Feed Rate, lb/hr	High-Shear Residence Time, sec	High-Shear Energy Input kwhr/Ton Feed Coal	High-Shear Energy Input kwhr/1000 Gallon Slurry	Inversion Quality
1586	79	9.1	4.0	Very Good
2389	53	6.1	2.7	Good
2946	40	4.9	2.0	Fair
3447	34	4.2	1.7	Poor
3689	32	4.0	1.6	Very poor

Based on the results of these tests, it was found that a minimum energy input of 6.1 kwhr/ton of feed coal was required to maintain "good" inversion. This data differs slightly from the results of the previous test work which indicated that a range of 8 to 15 kwhr/ton of feed coal was required to maintain good inversion.

High-Shear Energy Input Effects on Product Ash - Throughout this high-shear evaluation testing, low-shear product samples were processed via the laboratory rinse procedure to determine the effect of high-shear energy input on product ash content. From this work, it was determined that while in general, there was a slight increase in product ash content as the quality of inversion decreased, the effects were insignificant considering the varying feedstock and agglomerate sizes.

Low-Shear Circuit Testing

One test run (H-8), covering 8 setpoints, was completed to evaluate the low-shear unit operation. For this test, the following feed and high-shear operating conditions were utilized:

- 3800 lb/hr dry coal feed rate
- 9.1 to 10.5% feed slurry and high-shear solids concentration
- Heptane concentration as required to produce agglomerates in the 2 mm size range
- High-shear A only configuration
- 17.8 m/s high-shear impeller tip speed

- High-shear energy input of approximately 13 kwhr/ton feed coal (5.3 kwhr/1000 gallons of high shear throughput).

Since no significant effect of high-shear energy input on product ash was observed during the previous testing, the relatively high high-shear energy input of 13 kwhr/ton coal was utilized to insure that a very good inversion would be continually maintained throughout the low-shear testing.

Eight separate evaluations of the low shear unit operation were completed covering the following conditions:

- Low-shear solids concentrations of approximately 7 to 8% and 9.5 to 10.5%
- 3 and 5.2 m/s low-shear impeller tip speeds
- Low-shear vessel operated half full providing 1.8 to 2.4 minutes residence time
- Low-shear vessel operated full providing 4.5 to 6 minutes residence time
- Low-shear energy inputs of approximately 1.35, 2.6, and 7.8 kwhr/ton feed coal (0.45, 0.9, and 3 kwhr/1000 gallon throughput)

Pertinent results for these eight operating setpoints are shown in Table 20.

Table 20. Low-Shear Evaluation Results - Hiawatha Coal

D80, microns	LS Sol Conc, %	LS Imp Tip Speed, m/s	LS Res Time, Seconds	LS Energy, kwhr/1000 gal	LS Prod Ash, %	LS Prod Size, mm
			Low-Shear Vessel Half Full			
42.0	7.12	5.2	112	0.8	2.93	1-3
39.3	7.04	3.0	111	0.4	3.02	1-3
40.1	9.49	5.2	146	1.0	3.20	0.5-1.5
46.0	9.72	3.0	146	0.5	3.49	1-3
			Low-Shear Vessel Full			
46.3	7.77	5.2	274	2.6	3.15	C* 1-3
47.1	8.16	3.0	289	0.8	3.32	C* 0.5-1.5
50.8	10.47	5.2	365	3.3	3.13	C* 1-3
50.2	10.34	3.0	370	1.0	3.33	C* 0.5-1

* "C" indicates a cycling, or unstable, low shear operation

It should be noted that, unfortunately, the grind size increased for the second set of low-shear testing (vessel operated full), as compared to the first set of low-shear testing (vessel operated half full). As such, direct comparisons between operation of the low-shear vessel full and half-full cannot be made. However, based on the low-shear evaluation data available, and operating experience, the following conclusions were drawn from this data:

- When operating the low-shear vessel half full:

  - Agglomerates of consistent size could be produced under all conditions tested.
  - Lower product ash was achieved at lower solids concentration
  - Lower product ash was achieved at higher low-shear impeller tip speeds
- When operating the low-shear vessel full:
  - Low-shear operation was unstable resulting in cyclic agglomerate growth under all conditions tested, i.e., agglomerates would cycle from a minimum size (.5 mm) to a maximum size (3-5 mm) and back, repeatedly
  - The agglomerate size range during cyclic growth depended on the heptane level utilized, i.e., a higher heptane dosage produced larger agglomerates at the maximum growth point
  - The cycle time from large agglomerates to small agglomerates decreased as the solids concentration was increased
  - Lower product ash was achieved at higher low-shear impeller tip speeds
  - No effect of solids concentration was seen on the product ash content

Based on this data, it was decided that the unstable growth pattern observed when the low-shear vessel was operated full was unacceptable, and as such was not tested further. Therefore, all subsequent test work with the Hiawatha coal utilized the following low-shear operating conditions:

- Lower solids concentration
- Higher impeller tip speed
- Half full operation

Vibrating Screen Testing

Vibrating screen evaluation was carried out to investigate the effects of the following vibrating screen operating parameters on the final plant product ash content:

- Screen spray water flowrate
- Screen Inclination: uphill (U), level (L), or downhill (D)

For the vibrating screen evaluation testing, full plant testing was carried out and a full compliment of plant samples were taken for analysis. During this work, the following operating conditions, upstream of the vibrating screen were held constant to the degree that they did not have an impact on final product ash content:

- High-shear solids concentration - approximately 10%
- High-shear energy input - 5.5 to 11.4 kwhr/1000 gallons
- Heptane concentration - as appropriate to achieve 2 mm agglomerate size
- Low-shear solids concentration - 7.3 to 8.6%

- Low-shear vessel - half full
- Low-shear energy input - 0.8 to 0.9 kwhr/1000 gallons

Unfortunately, due to changes in ground feedstock particle size distributions during this testing, not all data can be compared directly. As such, Table 21 presents the pertinent results from selected vibrating screen evaluation tests as a function of the feedstock grind size D_{80}. Included in Table 21 is the agglomerate bed depth on the screen deck, as measured during operation.

Table 21. Vibrating Screen Evaluation Results - Hiawatha Coal

D80, microns	Screen Inclination	Bed Depth, inches	Spray gal/ton prod	LS Rinse Ash, %	Product Ash, %	Δ % Ash Prod - LS Rinse
Screen Spray Water Effect						
42.7	Uphill	4	243	2.80	3.24	0.44
43.9	Uphill	3-1/2	**508**	2.71	**3.03**	**0.32**
52.9	Level	2-1/2	271	3.00	3.24	0.24
51.2	Level	1-3/4	253	3.03	3.21	0.18
52.9	Level	1-7/8	**510**	2.97	**3.05**	**0.08**
53.1	Level	1-7/8	**505**	2.96*	**3.19***	**0.23***
44.2	Level	2-1/8	257	2.87	3.09	0.22
40.9	Level	2	**531**	2.94	**2.97**	**0.03**
43.6	Downhill	5/8	241	2.80	2.95	0.15
40.2	Downhill	5/8	**481**	2.73	**2.78**	**0.05**
39.6	Downhill	5/8	**511**	2.64	**2.73**	**0.09**
Screen Inclination Effect						
High Spray Rate						
56.4	Uphill	3	531	3.11	3.31	0.20
52.9	**Level**	1-7/8	510	2.97	**3.05**	**0.08**
53.1	**Level**	1-7/8	505	2.96*	**3.19***	**0.23***
43.9	Uphill	3-1/2	508	2.71	3.03	0.32
44.5	Uphill	3-1/2	510	2.73	3.07	0.34
40.9	Level	2	531	2.94	2.97	0.03
40.2	**Downhill**	5/8	481	2.73	**2.78**	**0.05**
39.6	**Downhill**	5/8	511	2.64	**2.73**	**0.09**
Low Spray Rate						
42.7	Uphill	4	243	2.80	3.24	0.44
44.2	Level	2-1/8	257	2.87	3.09	0.22
43.6	**Downhill**	5/8	241	2.80	**2.95**	**0.15**

* Considered an anomaly

Also shown in Table 21 is the difference between the plant final product ash content and the ash content achieved via the laboratory low-shear rinse procedure, which is considered to represent the best performance achievable from the 2 t/hr screen.

Spray Water Effects - As can be seen from the data in Table 21, in every case except for the one test, considered an anomaly, a lower product ash, and lower differential ash value between the plant product and the laboratory low-shear rinsing analysis, was achieved at the higher screen spray rate, than at the lower screen spray rate. This trend held constant regardless of the grind size and the screen inclination tested.

Screen Inclination Effects - From the data in Table 21, it is seen that in general, lower product ashes, and differential ashes, were achieved when the screen was level as compared to uphill, and when downhill as compared to level. This trend is attributed primarily to the lower screen bed depth achieved, due to less agglomerate retention time on the screen, as the orientation moves towards downhill. The lower bed depth is believed to allow better drainage of mineral-matter bearing process water. One additional advantage of the lower screen retention time (and bed depth) is that less degradation of the agglomerates occurs during screening.

As such, all additional testing carried out with the Taggart and Indiana VII coals, utilized the downhill screen inclination and the high screen spray water flow rates.

Froth Skimmer Testing

Evaluation testing of the froth skimmer was carried out during Tests H-13-A and H-13-B. The only froth skimmer variable evaluated was the use of the nitrogen, sparged into the froth skimmer, to help float any heptane bearing carbonaceous material in the vibrating screen underflow stream. For this work, all upstream plant conditions were held constant and nitrogen flow to the froth skimmer was used during Test H-13-A, followed by test H-13-B, identical except that the nitrogen flow to the froth skimmer was turned off.

Results from this work indicated that there was no effect on plant performance when the nitrogen purge was used as compared to when it was not used. In particular, there was no effect (not attributable to the small change in grind size) on the ash content of the following plant streams:

- Froth skimmer product
- Froth skimmer tailings
- Final plant tailings
- Final plant product

It was found throughout the Hiawatha coal testing that the froth skimmer, as installed in the PDU SA Module, was mostly ineffective in removing carbonaceous material from the plant tailings stream. At best, it is estimated that about 1% additional yield of the plant product was achieved from the froth skimmer. The froth skimmer operating problems are believed to be due to the following:

- The presence of too much surface area for froth collection in the froth skimmer. This factor, combined with the small amount of material floating in the skimmer, prevented the buildup of a sufficient froth layer for removal.
- The possible readsorption of heptane from the heptane saturated nitrogen gas blanket onto the material that floats in the skimmer. This resulted in the formation of a stiff cake-like layer, which could not be removed by the rotating scraper paddle.
- Poor distribution of the nitrogen bubbles throughout the froth skimmer.

Based on these observations during testing, the use of nitrogen in the froth skimmer was not tested further. Recommendations for an alternative froth skimmer design are as follows:

- The froth skimmer should be of a column design. This will allow for improved nitrogen dispersion across the full cross-sectional area and result in a higher solids/surface area ratio, allowing a thicker froth layer to form.
- The nitrogen that is used to blanket the skimmer should be heptane lean. The most effective scenario to achieve this would be to disperse heptane lean nitrogen, originating from directly downstream of the chilled knock-out cooler, through the skimmer. This gas would then pass back into the blanket system, upstream of the cooler, providing constant recirculation.
- In lieu of the above changes, a constant skimmer overflow (including some tailings) must be recycled to the high shear circuit.

Steam Stripper Testing

Concurrent with the screen testing, evaluation of the stripping circuit was also carried out. Four different sets of steam stripping circuit conditions were evaluated as follows:

- Low stripper A and B temperature, low stripper B residence time
- Low stripper A and B temperature, high stripper B residence time
- High stripper A and B temperature, low stripper B residence time
- High stripper A and B temperature, high stripper B residence time

Pertinent results for these test are shown in Table 22. As can be seen from this data, no clear trends were obvious due to either the effect of increased temperature and/or residence time. This is believed to be due primarily to the anticipated scatter in the heptane analytical results.

It should be noted, however, that relatively low residual heptane concentrations (approximately 2000 ppm, 0.2%, on a dry coal basis) were consistently achieved.

Furthermore, the lowest residual heptane concentrations achieved (1057 and 1194 ppm on a dry coal basis), resulted from operation at increased residence times. Also, based on previous batch and bench-scale testing results, a decrease in residual heptane concentration results from operation at higher temperatures.

As such, for the remainder of the Hiawatha coal testing, the stripping circuit was operated utilizing high residence times and high pressures/temperatures. Additional investigation into the effects of these stripping circuit variables was carried out during evaluation of the Taggart and Indiana VII coals, to be discussed later.

Table 22. Stripping Circuit Evaluation - Hiawatha Coal

Stripper A						Stripper B				
Sol Conc (%)	Oper Press (psi)	Slurry Temp (F)	Vapor Temp (F)	Res Time (min)	Hept, dcb (ppm)	Oper Press (psi)	Slurry Temp (F)	Vapor Temp (F)	Res Time (min)	Hept, dcb (ppm)
Residence Time Effects										
Low Temperature										
24.0	1.3	204	152	14.0	3149	5.0	232	230	**9.2**	**1544**
31.1	1.0	200	150	18.9	6916	5.0	222	231	**13.4**	**2242**
23.6	1.4	205	152	13.0	5041	4.8	230	231	**20.5**	**1194**
30.3	0.9	200	152	17.6	6077	5.0	230	231	**22.1**	**2135**
High Temperature										
21.2	4.8	212	158	12.6	4434	13.0	246	244	**9.2**	**1953**
28.5	4.2	210	159	16.6	5133	10.0	240	238	**12.3**	**1861**
28.3	4.1	210	159	16.5	5192	10.0	240	239	**12.7**	**1718**
29.0	4.3	210	161	16.9	6295	10.0	236	241	**22.5**	**2184**
27.3	4.5	210	161	15.7	4132	10.0	236	241	**20.9**	**1057**
Temperature Effect										
Low Residence Time										
24.0	1.3	204	**152**	14.0	3149	5.0	232	**230**	9.2	**1544**
31.1	1.0	200	**150**	18.9	6916	5.0	222	**231**	13.4	**2242**
21.2	4.8	212	**158**	12.6	4434	13.0	246	**244**	9.2	**1953**
28.5	4.2	210	**159**	16.6	5133	10.0	240	**238**	12.3	**1861**
28.3	4.1	210	**159**	16.5	5192	10.0	240	**239**	12.7	**1718**
High Residence Time										
23.6	1.4	205	**152**	13.0	5041	4.8	230	**231**	20.5	**1194**
30.3	0.9	200	**152**	17.6	6077	5.0	230	**231**	22.1	**2135**
29.0	4.3	210	**161**	16.9	6295	10.0	236	**241**	22.5	**2184**
27.3	4.5	210	**161**	15.7	4132	10.0	236	**241**	20.9	**1057**

One unexpected effect that was seen in this data was that regardless of what operating conditions were tested, lower stripping circuit feed solids concentrations resulted in lower residual heptane levels. Since this solids concentration effect was not observed during previous batch and bench-scale testing, however, and the economic penalty for operating at the lower solids concentration is substantial due to the additional water which must be heated throughout the stripping circuit, this effect was investigated separately during testing of subsequent project coals.

Additional comments concerning the stripper circuit testing for the Hiawatha coal follow:

- Operation of stripper B at levels greater than approximately 60% under high pressure conditions, resulted in an unstable operation. This was attributed to the carryover of coal in the vapor stream exiting stripper A.
- The operating pressures/temperatures of strippers A and B could not be varied totally independently due to the limited capabilities of the stripper B feed pump. If the differential pressure between the two vessels exceeded approximately 5 psi, this pump did not work.
- Since there was very little heptane in the stripper B vapor discharge, the stripper B slurry and vapor temperatures were almost identical.
- Since the stripper A vapor discharge was heavily loaded with heptane, the vapor temperature was significantly lower (150-160°F) than the slurry temperature (200-210°F).
- This low stripper A vapor discharge temperature indicated the presence of a heptane/water azeotrope, whose boiling point decreased as the heptane/water ratio increased. This was confirmed by the condensate return stream heptane/water ratio which was typically in the 2 to 3 range on a volumetric basis.

Optimization and Confirmation Testing

Prior to the start of the Hiawatha coal production run, two additional series of tests were carried out. These involved high-shear circuit (impeller tip speed) testing as well as confirmation testing at the anticipated production run operating conditions.

High-Shear Evaluation - Following completion of the initial Hiawatha coal high-shear circuit testing, it was decided to operate the plant utilizing high-shear vessel B only operating at its maximum tip speed (14 m/s) for the duration of the parametric testing. The reasoning for this was as follows:

- Problems with the high-shear A agitator seal forced the use of high-shear vessel B
- Even though inversion could be achieved with less high-shear energy input, since there was no effect on the product ash content due to high-shear energy input, this mode of operation would not be detrimental to the plant product ash content.

- This mode of operation would insure consistent high-quality inversion at the high-shear circuit discharge throughout the remainder of the parametric testing.

Therefore, one test run was completed (H-14-A) to evaluate the effect of a lower high-shear energy input on the selective agglomeration plant performance. For this test a high-shear impeller tip speed of 11 m/s was used, as compared to the 14 m/s tip speed tested for the bulk of the previous Hiawatha coal parametric testing. This resulted in a reduction of high-shear energy input by almost half, from approximately 28 kwhr/ton feed coal (11.2 kwhr/1000 gallon slurry) to 16.1 kwhr/ton feed coal (6.2 kwhr/1000 gallon slurry).

The results from this test indicated no detrimental effect on either product quality or plant Btu recovery as a result of this high-shear energy reduction. As such, this lower high-shear tip speed was chosen for use during the production run.

Confirmation Testing - Prior to the start of the production run, one test run (H-15) was completed at constant operating conditions, in which three separate sample sets were obtained. This was carried out to confirm that the conditions chosen for the production run would consistently provide a product which met the target ash content of 2 lb/MBtu. The results of this test did confirm acceptable plant performance over an extended time period, and as such, identical conditions were used for the Hiawatha coal production run.

Hiawatha Coal Production Run

The Hiawatha coal production run was carried out during the week of April 14, 1997. The operating conditions and results for the production run are shown in Appendix A, along with the previously discussed parametric testing results.

Production Run Results

As can be seen from the data in Appendix A, the production run met the 2 lb ash/MBtu product specification for all of the sample periods evaluated (H-P-1 through H-P-12). The following is a summary list of average production run conditions and results:

- Dry coal feed rate - 3839 lb/hr
- Plant feed grind D80 - 42.1 microns
- Plant feed solids concentration - 10.24%
- Plant feed ash content - 8.34%
- Heptane dosage utilized - 31.3% on a dry ash free coal basis
- Total agglomeration (high- and low-shear) energy input - 17 kwhr/ton feed coal (6.9 kwhr/1000 gallon slurry)
- Screen spray water rate - 500 gallons/ton product

- Steam consumption - 1380 lb/ton dry product (1.8 lb/gallon slurry stripped)
- Plant product ash content - 1.93 lb/MBtu (2.78%)
- Plant product residual heptane content - 2951 ppm on a dry coal basis
- Plant Tailings ash content - 80.4%
- Plant tailings residual heptane content - 1470 ppm on a dry solids basis
- Plant yield - 92.8%
- Plant Btu recovery - 98.9%

The following represents the operating schedule for the Hiawatha coal production run:

- Run start: 6:00 April 14
- Shutdown due to stripping circuit pump failure: 8:00 April 15 (26 hours run time)
- Run restart: 8:30 April 15 (1/2 hour down time)
- Shutdown due to stripping circuit pump failure: 19:30 April 15 (11 hours run time)
- Run restart: 10:00 April 16 (14-1/2 hours down time)
- Run end: 6:00 April 17 (20 hours run time)
- **Total approximate run time: 57 hours**
- **Total approximate down time: 15 hours**
- **Total run duration: 72 hours**

Both periods of down time during the production run were due to problems with the stripper feed diaphragm pump. This pump was rebuilt during the down time and was found to have a broken diaphragm caused by a wooden wedge lodged within the pump.

Production Run Grinding Circuit Analysis

Samples of the grinding circuit process streams were taken during the production run and analyzed for solids concentration, ash content, and particle size distribution (PSD). Figure 15 presents the grinding circuit utilized during the Hiawatha coal production run. Included in Figure 15 are the dry coal mass flow, the particle size distribution (PSD) D_{80}, and the ash content for the pertinent grinding circuit streams.

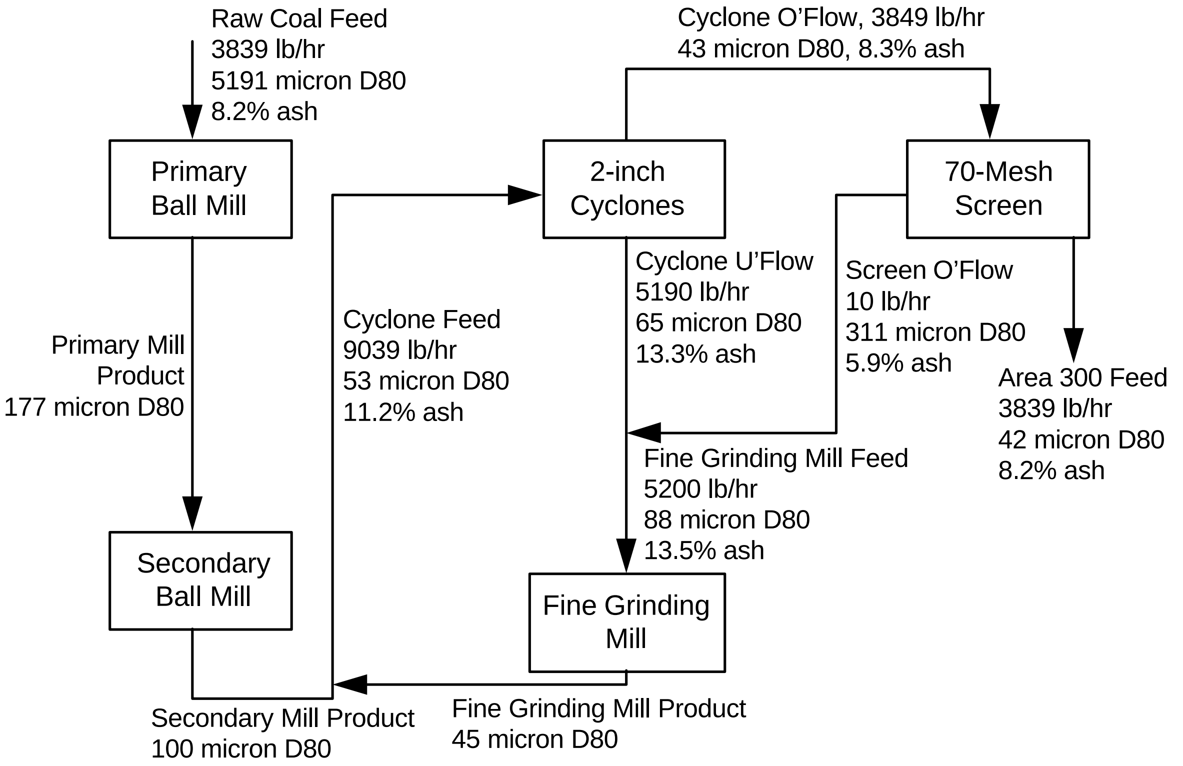


Figure 15. Hiawatha Coal Production Run Grinding Summary

Production Run Feed, Product, and Tailings Characterization

Composite samples of the crushed coal, ground feed slurry, product slurry, and tailings streams from the Hiawatha production run were submitted for complete analyses. Table 23 presents the analytical results for these composite production run samples.

Table 23. Hiawatha Coal Production Run Feed, Clean Coal, and Tailings Analyses

	Crushed Coal*	Feed Slurry*	Clean Coal*	Tailings*
Proximate, %:				
Ash	7.47	7.85	2.73	78.72
Volatile Matter	42.57	42.74	43.54	18.20
Fixed Carbon	49.96	49.41	53.73	3.08
Total	100.00	100.00	100.00	100.00
Forms of Sulfur, %:				
Total	0.52	0.51	0.50	0.77
Pyrite	0.07	0.09	0.03	0.83
Sulfate	< 0.01	< 0.01	< 0.01	0.02
Organic	0.45	0.42	0.47	< 0.01
HHV, Btu/lb	13,470	13,399	14,302	875
Ultimate, %:				
Carbon	77.48		82.35	
Hydrogen	5.23		5.51	
Nitrogen	1.46		1.56	
Sulfur	0.52		0.50	
Ash	7.47		2.73	
Oxygen	7.84		7.35	
Total	100.00		100.00	

* Bone-dry basis

Hiawatha Coal Testing Summary

This section of the report summarizes the PDU SA Module testing for the Hiawatha coal. In particular it presents the relationship between feedstock grind size and plant product ash content and a summary list of observations and conclusions.

Particle Size Distribution vs Product Ash Content Relationship

In general, it was found throughout the Hiawatha coal testing, that the effects of most operating parameters had only small effects on the product ash content. However, the ground feedstock particle size distribution was found to have a significant effect on the product ash content. This relationship is illustrated in Figure 16 which presents all of the Hiawatha coal testing results (complete plant tests), in the form of plant product ash content as a function of the ground feedstock 80% passing (D_{80}) size, regardless of the plant operating conditions utilized.

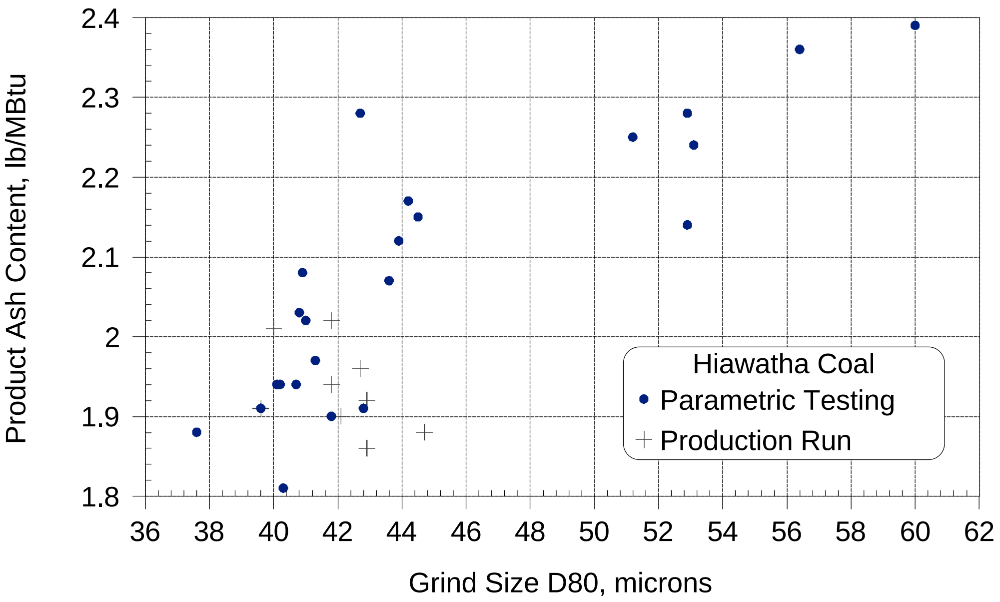


Figure 16. Product Ash Content vs Feedstock PSD D_{80} - Hiawatha Coal

As can be seen in Figure 16, there is much scatter in the data as a result of the parametric testing program, i.e., the completion of testing at a variety of plant operating conditions. However, the relationship between the feedstock particle size distribution D_{80} and the plant product ash content (lb/MBtu) is clearly evident.

Observations and Conclusions

The following is a summary list of observations and conclusions based on the Hiawatha coal PDU SA Module testing:

- While the SA module could be run at a 2 t/hr coal feed rate, a more stable operation was achieved at a slightly lower feed rate (3800 lb/hr).
- A feedstock grind with a D_{80} of approximately 40 microns was sufficiently fine to achieve the 2 lb ash/MBtu product target ash level for the Hiawatha coal.
- Overall plant Btu recoveries were very high, > 99%.
- There appeared to be no effect of high shear energy input on product ash content as long as “good inversion” was maintained in high shear.
- Operation of the low shear vessel half full provided sufficient residence time for agglomerate growth to a recoverable size.
- Operation of the low shear vessel full resulted in an unstable cyclic growth pattern
- Lower product ash was achieved at higher screen spray water flow rates.
- Lower product ash was achieved when the screen was in the downhill orientation than when level, and when level as compared to uphill.

- There was no effect on plant performance when the nitrogen purge was used as compared to when it was not used in the froth skimmer.
- The froth skimmer design did not work well because of the presence of too much surface area for froth collection in the froth skimmer, the small amount of material floating in the skimmer, the possible readsorption of heptane from the heptane saturated nitrogen gas blanket onto the material that floats in the skimmer, and poor distribution of the nitrogen bubbles.
- No clear trends were obvious relating steam stripping residence times and operating temperatures to product residual heptane concentrations.
- No deleterious effects were observed when operating the stripping circuit at relatively high (20-25%) solids concentrations.
- Steam consumption was typically on he order of 1300 lb/ton coal.
- Product residual heptane concentrations were typically in the 2000 to 3000 ppm range on a dry solids basis.
- Tailings residual heptane concentrations were typically in the 1000 to 2000 ppm range on a dry solids basis.

Taggart Coal Parametric Testing Results

Based on the parametric testing results of the Hiawatha coal, it was determined that the following operating conditions would not be evaluated for the Taggart coal:

- Low (<10%) high-shear solids concentration since these were found to provide no reduction in product ash content while resulting in higher high-shear energy input requirements.
- Low-shear vessel operated full since this was found to make agglomerate growth difficult to control and the half-full configuration provided sufficient residence time for agglomerate growth to the 2 to 3 mm size range.
- Vibrating screen in uphill or level orientation since downhill orientation was shown to reduce both agglomerate bed depth and product ash content.
- Froth skimmer with the use of the nitrogen sparger.

As such, the parametric testing for the Taggart coal focused primarily on evaluation of the following:

- High-shear agglomeration
- Low-shear agglomeration
- Steam stripping
- Grinding requirements

Results for all of the Taggart coal PDU SA Module parametric testing for the Taggart coal are presented in Appendix B.

High-Shear Circuit Testing

High-Shear Impeller Tip Speed Evaluation - As for the Hiawatha coal, initial parametric testing of the Taggart coal focused on the determination of the minimum tip speed requirements to maintain inversion at the high-shear circuit discharge. During this high-shear evaluation testing, the impeller tip speed was incrementally reduced to determine the point at which inversion at the high-shear discharge was lost. In addition, for each high-shear energy input level (impeller tip speed), low-shear samples were taken and subjected to the laboratory rinsing procedure to help determine any potential effects of high-shear operating parameters on the product ash content.

Pertinent results from this work are shown in Table 24.

Table 24. Initial High-Shear Tip Speed Evaluation - Taggart Coal

	Grind	Tip Speed	Kwhr per		Inversion	Prod	Tails	Yield	Btu Rec
Test	D_{80}	m/s	ton coal	1000 gal	Quality	ash, %	ash, %	%	%
High Shear B Evaluation									
T-1-1	35.5	14.0	26.7	11.1	V. Good	1.59	76.5	97.1	99.3
T-1-2	35.5	11.5	16.2	6.7	Fair	1.58	72.3	96.9	99.1
T-1-3	35.5	9.0	8.3	3.4	Poor	1.60	68.6	96.7	99.0
High Shear A Evaluation									
T-2-1	38.4	17.4	12.0	5.2	V. Good	1.73	81.3	97.7	99.5
T-2-2	38.4	15.1	8.6	3.7	Good	1.69	78.5	97.5	99.5
T-2-3	37.1	11.0	3.6	1.8	V. Poor	1.60	63.1	96.8	98.8
T-6-1	34.4	13.0	6.5	2.8	V. Good	1.70	73.1	97.4	99.3
T-6	35.6	11.0	3.9	1.7	Good	1.63	68.6	97.2	99.1

As expected, the quality of the inversion exiting the high-shear circuit decreased as the high-shear impeller tip speed was reduced. This was found to hold true for all three sets of tests shown in Table 24. However, it was found that even under high-shear conditions resulting in very poor inversion, agglomerate growth in the low-shear vessel was still sufficient to afford good agglomerate (and therefore Btu) recovery.

The main effects of reducing high-shear energy input were:

- A small decrease in product ash content
- A decrease in tailings ash content
- A small decrease in yield and Btu recovery

Due to an increasingly worse high-shear A shaft seal leak, one additional series of tests was carried out utilizing high-shear B, in which the coal feed rate was held constant and the impeller tip speed reduced. This work was completed to determine at what energy input (impeller tip speed), the product ash target of 1 lb/MBtu could be met at a feed grind size with a D_{80} of approximately 30 microns. Pertinent results for these tests are shown in Table 25.

Table 25. High-Shear B Tip Speed Evaluation - Taggart Coal

		High-Shear Operating Conditions							Tails	Btu
	Grind	Solids	Tip Spd	Kwhr per		Inver.	Product Ash		Ash	Rec
Test	D80	%	m/s	ton coal	1000 gal	Quality	%	lb/MBtu	%	%
T-12	30.2	10.1	11.0	16.3	6.7	Good	1.54	1.02	63.5	98.6
T-13	29.1	10.4	9.5	10.8	4.5	Poor	1.45	0.96	55.6	98.1
T-14	31.4	11.1	8.5	7.3	3.3	V. Poor	1.77	1.18	62.0	99.0

As can be seen from the data in Table 25, as the high-shear impeller tip speed was decreased from 11.0 to 9.5 m/s, the product ash content, tailings ash content, and Btu recovery all decreased as expected. However, in test T-14, where the tip speed was decreased to 8.5 m/s, the reverse effect was seen. While the T-14 results are considered an anomaly, they may be partially due to the increased high-shear solids concentration, which is known to reduce high-shear energy requirements.

Due to the limited supply of Taggart coal, the results from these three tests were used to select operating conditions for the Taggart coal production run. It was decided to operate the production run at conditions similar to those shown in Appendix B for tests T-12 and T-13, except that a high-shear impeller tip speed of 10.0 m/s would be used. This was anticipated to yield a product ash content of approximately 1 lb/MBtu.

High-Shear A Energy Input Evaluation - A series of tests was carried out to determine the effect of decreasing high-shear energy input, at a constant impeller tip speed (11 m/s), on product ash content. To accomplish this, the coal feed rate to the plant was incrementally increased with all other operating conditions, including the heptane/coal ratio, remaining constant. Evaluation of these tests was based on low-shear product samples which were rinsed in the lab. It should be noted that the changing coal throughput rate also effected the energy input into low shear. Pertinent results for these tests are shown in Table 26.

Table 26. High-Shear A Energy Input Evaluation - Taggart Coal

						LS Rinse Basis		Btu
	Grind	Coal Feed	Kwhr per		Inversion	Ash (%)		Rec
Test	D80	lb/hr	ton coal	1000 gal	Quality	Prod	Tails	%
T-10-1	32.0	2553	5.8	2.4	Fair	1.60	69.0	99.1
T-10-2	32.1	3003	4.8	2.0	Poor	1.55	59.9	98.6
T-10-3	32.2	3517	4.1	1.7	V. Poor	1.47	50.7	97.8
T-10-4	30.7	3765	3.8	1.6	None	1.53	45.0	97.3

As can be seen from this data, as the plant coal throughput was increased, decreasing high-shear energy input on a coal and slurry basis, the inversion quality achieved from high-shear decreased as expected. As a result of decreasing quality of inversion, the tailings ash decreased along with the Btu recovery. These trends were due to increasing degrees of “incomplete agglomeration” resulting in more coal losses to the tailings as energy input on a coal basis was decreased. A similar trend of decreasing product ash content was also observed when the high-shear energy was decreased. The high product ash content for Test T-10-4 (1.53%) is considered an anomaly, however, especially since this test had a slightly finer feed coal grind size. As such, it was determined that reductions in high-shear energy input could be used to achieve small reductions in product ash content with corresponding small reductions in Btu recovery.

Low-Shear Circuit Testing

Low-Shear Solids Concentration Effects - Tests T-7 and T-8 were carried out to evaluate the effect of low-shear solids concentration on product ash content. While attempts were made to maintain all other plant conditions at similar set points, problems with the weigh belt feeder resulted in a finer grind size for test T8. Results for these test are shown in Table 27.

Table 27. Low-Shear Parameter Effects - Taggart Coal

			Low Shear		LS Rinse		Full Plant	
	Grind	%	Imp Tip	Res Time	Ash (%)		Ash (%)	
Test	D80	Solids	Speed (m/s)	Seconds	Prod	Tails	Prod	Tails
T-7	35.6	8.4	5.2	131	1.63	67.6	1.63	69.7
T-8	33.2	7.1	5.2	116	1.63	64.6	1.64	67.2
T-9	35.2	8.6	6.6	131	1.72	72.2	1.68	75.3

As can be seen from this data, no effect on product ash was observed when decreasing the low-shear solids concentration from 8.4 to 7.1% in Tests T-7 and T-8, respectively. In fact, since T-8 had a slightly finer grind, the reduced solids concentration was definitely not advantageous.

Low-Shear Impeller Tip Speed Effect - Also shown in Table 27 are the results of Test T-9, which evaluated the effect of a higher low-shear impeller tip speed. When comparing the effect of increasing the low-shear impeller tip speed from 5.2 m/s in test T-7, to 6.6 m/s in test T-9, an increase in product ash is observed. As such, no further testing was done at the higher low-shear impeller tip speed for the Taggart coal.

Steam Stripper Testing

Stripping Circuit Solids Concentration Effect - Table 28 presents results for two tests performed at different stripping circuit solids concentrations (T-8 and T-9). As can be seen from this data, reducing the steam stripping circuit solids concentration had no effect on the residual heptane concentration of the product, from either the first or second stage of steam stripping. As such, subsequent testing was carried out at the higher solids concentration to reduce steam requirements.

Table 28. Stripper Solids Concentration Effects - Taggart Coal

	First Stage Steam Stripper (A)		Second Stage Steam Stripper (B)	
Test	% Solids	Residual heptane, ppm dcb	% Solids	Residual heptane, ppm dcb
T-8	29.6	9953	28.7	4716
T-9	21.2	9762	20.7	5082

Grinding Requirements Testing

Additional parametric testing carried out with the Taggart coal focused on decreasing the plant feed rate such that a finer grind could be achieved and the product ash target of 1 lb/MBtu met. Results of these tests indicated that a grind with a D_{80} of approximately 30 microns was required to achieve the 1 lb ash/MBtu target product grade. At this grind size, it was found that a tailings ash content in the 60% range was needed to insure that the product grade was met. It was found that at a dry coal feed rate of approximately 3300 lb/hr and a high-shear solids concentration of about 10%, the desired product ash could be achieved at a high-shear impeller tip speed in the 10 m/s range. As such, these conditions were chosen as the target operating parameters for the production run.

Figure 17 shows the results of all the Taggart coal parametric testing as product grade in lb ash/MBtu vs Grind size D_{80} in microns. As can be seen from this data, while there is scatter in the data attributed to the various operating conditions tested, the general trend of decreasing product ash content with decreasing grind size D_{80} is evident.

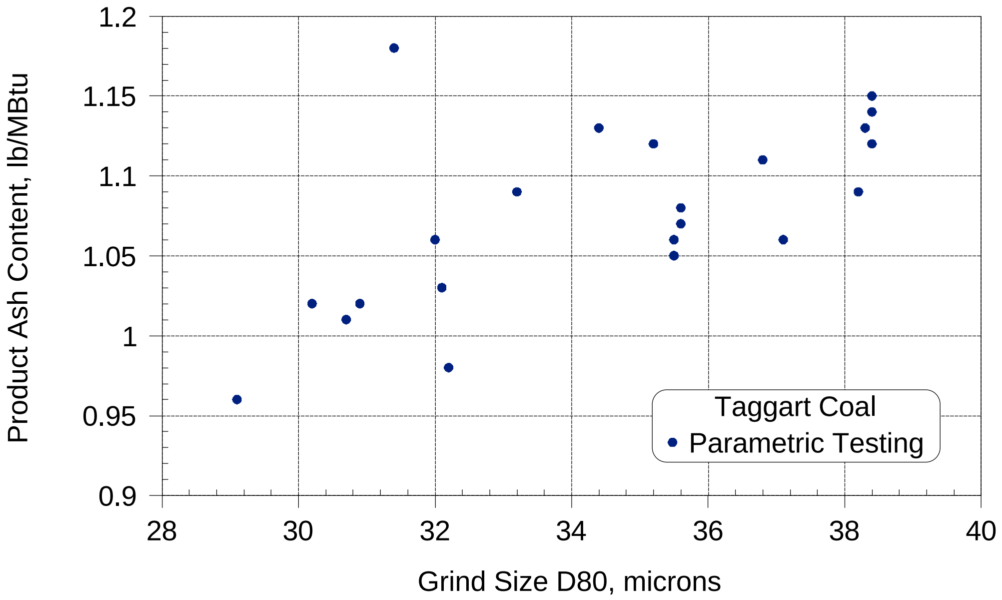


Figure 17. Taggart Coal Parametric Testing Results

Taggart Coal Production Run

The Taggart coal production run was carried out during the week of May 19, 1997. John Getsoian from Arcanum was on site during the production run to provide operations support and videotape the PDU operation. Individual set point and average operating conditions and results for the production run are shown in Appendix B.

Production Run Results

As can be seen from this data, the average product ash content was 1.06 lb/MBtu, slightly higher than the 1 lb/MBtu target. The range of product ash contents for the individual samples was from 1.01 to 1.12 lb/MBtu. The following is a summary list of average production run operating conditions and results:

- Dry coal feed rate - 3305 lb/hr
- Plant feed grind D_{80} - 30.3 microns
- Plant feed solids concentration - 10.02%
- Plant feed ash content - 3.64%
- Heptane dosage utilized - 39.2% on a dry ash free coal basis
- Total agglomeration (high- and low-shear) energy input - 16.1 kwhr/ton feed coal (6.2 kwhr/1000 gallon slurry)
- Screen spray water rate - 566 gallons/ton product
- Steam consumption - 1553 lb/ton dry product (1.8 lb/gallon slurry stripped)
- Plant product ash content - 1.06 lb/MBtu (1.59%)
- Plant product residual heptane content - 5115 ppm on a dry coal basis
- Plant tailings ash content - 63.0%
- Plant tailings residual heptane content - 4094 ppm on a dry solids basis
- Plant yield - 96.7%
- Plant Btu recovery - 99.2%

The following represents an approximate summary of the operating schedule for the Taggart coal production run:

- Run start: 7:15 May 19
- Shutdown due to stripping circuit feed pump failure: 20:15 May 19 (13 hours run time)
- Run restart: 22:15 May 19 (2 hours down time)
- Run end: 7:15 May 22 (57 hours run time)
- **Total approximate run time: 70 hours**
- **Total approximate down time: 2 hours**

- **Total run duration: 72 hours**

The two hours of down time during the production run was due to worn diaphragms in the stripping circuit feed pump. This problem was expected since replacement diaphragms of the correct material (viton) had been ordered and were to be installed prior to the production run. However, the wrong diaphragms were shipped, and as such, the production run was started on schedule, but with the old diaphragms in place. The new viton diaphragms arrived the day the production run was started and were installed when the pump failed.

Other problems encountered with equipment during the production run, none of which forced a plant shutdown, were as follows:

- Shortly after start up, the grinding circuit cyclones were found to be plugged. These were unplugged on line, allowing operation to continue.
- Failure of the control valve that maintained the correct operating level in the gravity separator. This was overcome by manually controlling the water discharge from the gravity separator for the duration of the production run.
- The fine grinding mill tripped out resulting in the reduction of feed to the Area 300 feed storage tank for approximately 30 minutes. The grinding circuit circulating load was rerouted to the secondary ball mill until the fine grinding mill could be restarted.

Production Run Grinding Circuit Analysis

Samples of the grinding circuit process streams were taken during the production run and analyzed for solids concentration, ash content, and particle size distribution (PSD). Figure 18 presents the grinding circuit utilized during the Taggart coal production run. Included in Figure 18 are the dry coal mass flow, the particle size distribution (PSD) D_{80}, and the ash content for pertinent grinding circuit streams.

Production Run Feed, Product, and Tailings Characterization

Composite samples of the crushed coal, ground feed slurry, product slurry, and tailings streams from the Taggart coal production run were submitted for complete analyses. Table 29 presents the analytical results for these composite production run samples.

Taggart Coal Testing Summary

This section of the report summarizes the PDU SA Module testing for the Taggart coal. In particular it presents the relationship between feedstock grind size and plant product ash content and a summary list of observations and conclusions.

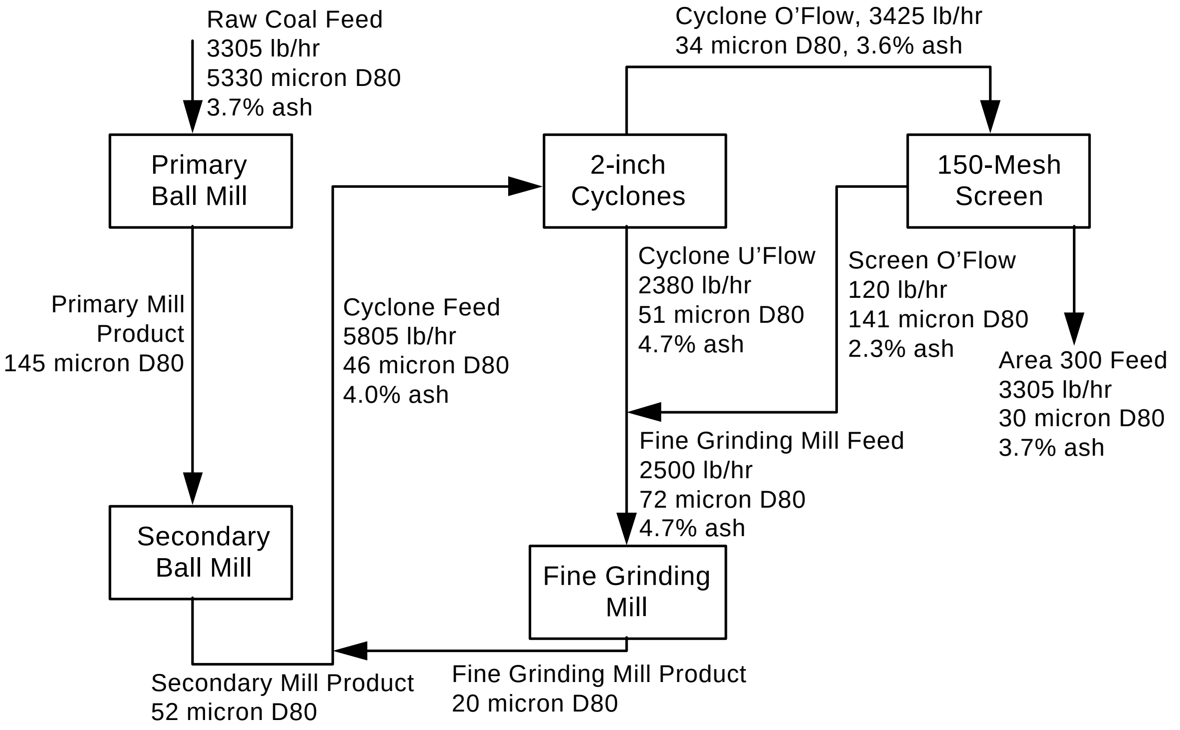


Figure 18. Taggart Coal Production Run Grinding Circuit Summary

Table 29. Taggart Coal Production Run Feed, Clean Coal, and Tailings Analyses

	Crushed Coal*	Feed Slurry*	Clean Coal*	Tailings*
Proximate, %:				
Ash	3.50	3.70	1.64	63.20
Volatile Matter	34.12	34.34	35.09	18.55
Fixed Carbon	62.38	61.96	63.27	18.25
Total	100.00	100.00	100.00	100.00
Forms of Sulfur, %:				
Total	0.65	0.64	0.63	0.81
Pyrite	0.05	0.06	0.04	0.78
Sulfate	< 0.01	< 0.01	< 0.01	0.12
Organic	0.60	0.58	0.59	< 0.01
HHV, Btu/lb	14,735	14,688	15,072	4,260
Ultimate, %:				
Carbon	85.28		87.62	
Hydrogen	4.95		5.14	
Nitrogen	1.47		1.53	
Sulfur	0.65		0.63	
Ash	3.50		1.64	
Oxygen	4.15		3.44	
Total	100.00		100.00	

* Bone-dry basis

Particle Size Distribution vs Product Ash Content Relationship

In general, it was found throughout the Taggart coal testing, that the effects of most operating parameters had only small effects on the product ash content. However, the ground feedstock particle size distribution was found to have a significant effect on the product ash content. This relationship is illustrated in Figure 19 which presents all of the Taggart coal testing results, in the form of plant product ash content as a function of the ground feedstock 80% passing (D_{80}) size, regardless of the plant operating conditions utilized.

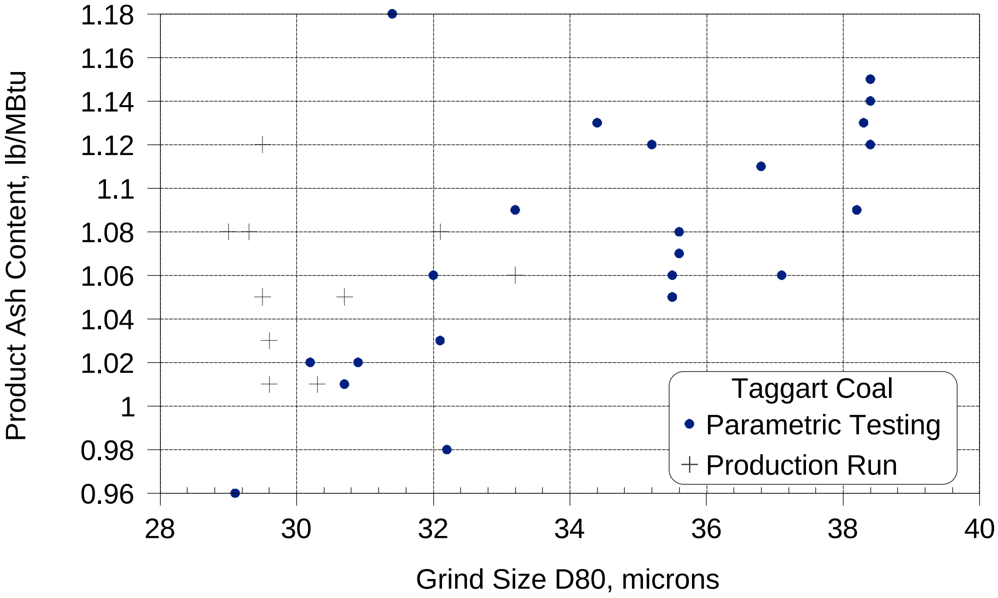


Figure 19. Product Ash Content vs Feedstock PSD D_{80} - Taggart Coal

As compared to the similar figure presented previously for the Hiawatha coal, the relationship between feedstock grind size and product ash content is not as clear for the Taggart coal. This is attributed to two factors:

- The narrow range of grind size D_{80}s tested for the Taggart coal (29 to 39 microns) as compared to the Hiawatha coal (37 to 60 microns).
- The effect of high-shear energy input on Btu recovery for the Taggart coal, which resulted in a range of product ash contents at similar grind sizes.

While there is much scatter in the data for the above reasons, the relationship between the feedstock particle size distribution D_{80} and the plant product ash content (lb/MBtu) is still evident in Figure 19.

Observations and Conclusions

The following is a summary list of observations and conclusions based on the Taggart coal PDU SA Module testing:

- The quality of the inversion exiting the high-shear circuit decreased as the high-shear impeller tip speed was reduced.
- Even under high-shear conditions resulting in very poor inversion, agglomerate growth in the low-shear vessel was still sufficient to afford good agglomerate (and therefore Btu) recovery.
- The main effects of reducing high-shear energy input were:
  - A small decrease in product ash content
  - A decrease in tailings ash content
  - A small decrease in yield and Btu recovery
- At a coal feed rate of 3300 lb/hr, a feed grind size with a D_{80} of about 30 microns, and a 10 m/s high-shear impeller tip speed, the product ash target of 1 lb/MBtu was met at a tailings ash content of approximately 60%.
- There was no effect of low-shear solids concentration on product ash.
- As the low-shear impeller tip speed was increased, a corresponding increase in product ash content was observed.
- Reductions in the steam stripping circuit solids concentration had no effect on the residual heptane concentration of the product, from either the first or second stage of steam stripping.
- Steam consumption was typically on the order of 1500 lb/ton coal.
- Product residual heptane concentrations were typically in the 4000 to 5000 ppm range on a dry solids basis.
- Tailings residual heptane concentrations were typically in the 3000 to 5000 ppm range on a dry solids basis.

Indiana VII Coal Parametric Testing Results

Based on the parametric testing results of the Hiawatha and Taggart coals, it was determined that the following operating conditions would not be evaluated for the Indiana VII coal:

- Low (<10%) high-shear solids concentration
- Low-shear vessel operated full since this was found to make agglomerate growth difficult to control and the half-full configuration provided sufficient residence time for agglomerate growth to the 2 to 3 mm size range.
- Vibrating screen in uphill or level orientation since downhill orientation was shown to reduce both agglomerate bed depth and product ash content.

- Froth skimmer with the use of the nitrogen sparger.

As such, parametric testing for the Indiana VII coal focused primarily on the following:

- High-shear agglomeration
- Low-shear agglomeration
- Vibrating screen wash water flow rate
- Steam stripping
- Grinding requirements

Results for all of the PDU SA Module parametric testing for the Indiana VII coal are presented in Appendix C.

Start-Up Test

Since the Indiana VII coal is a very difficult to agglomerate coal, typically requiring long (2 to 3 minutes) high-shear residence times, asphalt in the form of an emulsion was used to promote inversion during high shear. When received, this asphalt emulsion was approximately 55% solids, i.e., 55% asphalt with the remainder being water. Before its use, this emulsion was diluted to a solids concentrations in the 3 to 10% range. This allowed the emulsion to be metered to the process at the target dosage of 5 to 15 lb asphalt per dry ton of feed coal. This asphalt was delivered to the process (the high-shear circuit feed line) via a centrifugal gear pump.

During the initial Indiana VII coal start-up test (I-1) an asphalt dosage of 9.5 lb/ton coal was used and high-shears A and B operated in series at their maximum tip speeds of 17.4 and 14 m/s, respectively. At these operating conditions, only marginal phase inversion (a rating of 2 on a 1 to 10 scale) was achieved. This poor inversion at these relatively high asphalt and energy levels was attributed to the low solids concentration (6.96%) used during high shear. This low solids concentration, due to a faulty weight belt feeder metering coal to the grinding circuit, resulted in a less efficient high-shear unit operation due to less particle to particle contact than would be achieved at a higher solids loading. This low solids loading also reduced the available high-shear residence time due to the high volumetric flowrate required to maintain the target dry coal throughput rate.

It was also found during this start-up test, that the asphalt emulsion contained large pieces of what appeared to be tar. These large particles bound up the asphalt pump causing the asphalt pump to stop pumping on several occasions. During these periods of no asphalt flow, inversion was lost completely resulting in the loss of coal to the tailings stream. As such, the tailings ash content for this start-up test was only 73% while the product ash content was 2.2 lb/MBtu and the Btu recovery 97.9%.

To resolve this asphalt delivery problem, the asphalt emulsion was screened on a 28-mesh sieve prior to its use to remove the large particles. While this reduced the pump plugging problem, it was also determined that when the asphalt delivery pump was operated below about 20% speed, the pump continually stalled. As such, the asphalt emulsion had to be diluted to 2 to 3% solids for consistent asphalt flows to be maintained with the pump being used.

High-Shear Circuit Testing

After the initial start-up test, subsequent testing with the Indiana VII Coal was carried out to determine the asphalt dosage and high-shear energy input required to achieve inversion at the high-shear circuit discharge. During this testing, additional operating and feedstock parameters known to effect the high-shear unit operation, namely feedstock PSD and high-shear solids concentration, were also varied. As such, data presented here for high-shear energy and asphalt dosage effects on inversion, are independent of these variables.

Asphalt Effect - Table 30 presents 4 pairs of test results, using low-shear product samples subjected to the laboratory low-shear rinse procedure, to illustrate the effect of asphalt dosage on product ash content and inversion quality with all other variables held relatively constant.

Table 30. Asphalt Effects on Inversion and Product Ash - Indiana VII Coal

Test	Grind D80	% solids	Asphalt lb/ton	HS Tip Speed m/s	High Shear Kwhr per ton coal	High Shear Kwhr per 1000 gal	Inversion Quality (1-10)	LS Rinse Basis Ash (%) Prod	LS Rinse Basis Ash (%) Tails
I-2	23.9	8.80	**8.5**	14.0	49.0	17.4	**4**	**3.14**	**85.2**
I-3-1	23.3	8.83	**11.3**	14.0	48.9	17.4	**6**	**3.15**	**88.8**
I-6	26.0	11.34	**8.5**	14.0	38.8	17.6	**2**	**2.96**	**84.1**
I-7-1	26.2	11.75	**13.3**	14.0	37.4	17.5	**5**	**3.07**	**92.2**
I-9-3	20.0	12.40	**4.8**	14.0	46.8	23.1	**2**	**2.65**	**86.2**
I-9-2	21.1	12.42	**7.7**	14.0	46.7	23.0	**6**	**2.76**	**91.3**
I-10-3	22.8	12.11	**4.9**	14.0	40.3	19.4	**3**	**2.95**	**85.2**
I-10-2	22.5	12.28	**6.8**	14.0	39.7	19.3	**5**	**2.93**	**89.1**

As expected, it can be seen from this data that in every case, increasing the asphalt dosage improved the quality of inversion achieved. This effect can also be seen in the tailings ash values which consistently increased with higher asphalt dosages. It was found that when the lower asphalt dosages were used, resulting in lower tailings ash values, more unagglomerated filmy material was observed in the low-shear samples.

It can also be seen from the data in Table 30, that there appears to be a small effect of asphalt dosage on product ash content, with higher asphalt dosages resulting in slightly higher product ash values for 2 of the 4 pairs of results presented. However, for the other two pairs there was no effect at all.

High-Shear Energy Effect - Table 31 presents 2 pairs of test results, using low-shear product samples screened in the lab, to illustrate the effect of high-shear energy input on product ash content and inversion quality, with all other variables held constant.

Table 31. High-Shear Energy Effects - Indiana VII Coal

				HS Tip	High Shear		Inversion	LS Rinse Basis	
	Grind	%	Asphalt	Speed	Kwhr per		Quality	Ash (%)	
Test	D80	solids	lb/ton	m/s	ton coal	1000 gal	(1-10)	Prod	Tails
I-3-1	23.3	8.83	11.3	**14.0**	48.9	17.4	**6**	**3.15**	**88.8**
I-3-2	23.3	8.83	11.3	**11.0**	26.6	9.4	**2**	**3.23**	**83.8**
I-7-1	26.2	11.75	133	**14.0**	37.4	17.5	**5**	**3.07**	**92.2**
I-7-2	26.2	11.75	13.3	**12.0**	27.3	12.7	**3**	**3.23**	**90.4**

As expected, it can be seen from this data that for both cases, decreasing the high-shear tip speed (energy input) reduced the quality of inversion achieved. This effect can also be seen in the tailings ash values which consistently decreased with lower energy input indicating incomplete agglomeration, as observed during testing by the presence of more unagglomerated filmy material in the low-shear samples.

It can also be seen from this data, that there is a clear effect of high-shear energy input on product ash content, with lower energy resulting in higher product ash values for both pairs of results presented. This is due to the production of better formed agglomerates at the higher energy levels, resulting in better screening, i.e., improved drainage of associated mineral-matter bearing process water.

Combined Asphalt Dosage and High-Shear Energy Effect - Table 32 presents 4 pairs of test results, using low-shear product samples screened and rinsed in the laboratory, to illustrate the combined effect of simultaneously increasing asphalt dosage and decreasing high-shear energy input on product ash content, with all other variables held relatively constant.

Table 32. Combined Asphalt & High-Shear Energy Effects - Indiana VII Coal

Test	Grind D80	% solids	Asphalt lb/ton	HS Tip Speed m/s	High Shear Kwhr per		Inversion Quality (1-10)	LS Rinse Basis Ash (%)	
					ton coal	1000 gal		Prod	Tails
I-2	23.9	8.80	**8.5**	**14.0**	49.0	17.4	4	**3.14**	85.2
I-3-1	23.3	8.83	**11.3**	**11.0**	26.6	9.4	2	**3.23**	83.8
I-4	26.1	8.50	**11.8**	**--**	59.7	20.4	9	**3.19**	90.1
I-5	26.4	8.80	**18.9**	**--**	38.0	13.4	5	**3.30**	88.2
I-6	26.0	11.34	**8.5**	**14.0**	38.8	17.6	2	**3.07**	92.2
I-7-1	26.2	11.75	**13.3**	**12.0**	27.3	12.7	3	**3.23**	90.4
I-9-3	20.0	12.40	**4.8**	**14.0**	46.8	23.1	2	**2.65**	86.2
I-9-4	20.1	12.61	**9.5**	**12.0**	34.2	17.0	1	**2.81**	89.1

As can be seen from this data, when the asphalt dosage was increased and the high-shear energy decreased simultaneously, a higher product ash content resulted. This data combined with the results presented in the previous two tables indicate that if the goal of the process is to achieve the lowest product ash content at a given grind size, the asphalt dosage should be minimized and sufficient energy used to achieve the formation of good agglomerates.

Solids Concentration Effect - No testing was performed specifically to evaluate the effect of high-shear solids concentration on the energy and/or asphalt required to achieve inversion. However, a comparison of tests I-2, I-3-1, and I-8-1, as shown in Table 33 illustrates this effect to some degree.

Table 33. High-Shear Solids Concentration Effect on Inversion - Indiana VII Coal

Test	Grind D80	% solids	Asphalt lb/ton	HS Tip Speed m/s	High Shear Kwhr per		Inversion Quality (1-10)
					ton coal	1000 gal	
I-2	23.9	**8.80**	8.5	14.0	49.0	17.4	**4**
I-3-1	23.3	**8.83**	11.3	14.0	48.9	17.4	**6**
I-8-1	23.7	**12.40**	9.7	14.0	46.4	22.8	**8**

It can be seen from this data that Test I-8-1, which utilized an asphalt dosage in the same range as the previous tests, achieved better inversion quality at the same high-shear tip speed and similar energy inputs. This is attributed to the increased solids concentration in high shear which results in more particle to particle contact at similar energy inputs.

Low-Shear Circuit Testing

One series of tests (I-8-1, I-8-2, and I-8-3) was carried out to evaluate the effect of low-shear solids concentration and tip speed on the operability of the low-shear vessel and the product ash content.

Solids Concentration Effect - Unfortunately during the completion of this test series, the grind size was increasing, and as such, a direct comparison of low-shear sample rinse product ash contents as a function of these two variables can not be made. It should be noted, however, that even though the grind size increased from Test I-8-1 to I-8-2 (D_{80} of 23.7 to 25.1 microns) and the solids concentration was increased from 7.7 to 12.7%, no significant increase in product ash content (3.11 to 3.16%) was observed. This indicates that higher solids loadings during low shear does not have a detrimental effect on product ash content for the Indiana VII coal.

Considering the operability of the low-shear vessel at the higher solids concentration, no difficulties were encountered. This was somewhat surprising since previous testing of the low-shear unit operation at high solids concentration resulted in very difficult to control agglomerate growth. It is possible that the use of asphalt during high shear to achieve inversion, and its subsequent presence during low shear, may make agglomerate growth more controllable, i.e., less sensitive to the heptane dosage utilized.

Low-Shear Tip Speed Effect - In comparing Tests I-8-1 and I-8-3, when the low-shear tip speed was increased from 5 to 6.5 m/s during Test I-8-3, and the grind was coarser (D_{80} = 27.5 microns as compared to 23.7 microns during Test I-8-1), only a relatively small increase in product ash content (3.11 to 3.22%) was observed. This indicates no significant increase in product ash content due to the higher low-shear impeller tip speed.

One additional test (I-10-1) was completed to evaluate the effect of utilizing a lower low-shear impeller tip speed. It was found that the 3 m/s tip speed tested resulted in poor agglomerate growth with the agglomerates appearing overdosed with heptane even though they were not. During this test, some of the agglomerates grew very large (6 mm) while much of the coal remained in the microagglomerate form, i.e., no growth. This observation was confirmed by the relatively low (81.5%) tailings ash value achieved during rinsing of the low-shear sample in the lab. This low low-shear impeller tip speed test was performed on three different occasions with similar results, indicating that the 3 m/s tip speed does not supply sufficient energy for consistent agglomerate growth, i.e., sufficient mixing to achieve thorough particle to particle contact in the low-shear vessel.

Agglomerate Size Effect - Two tests (I-10-3 and I-10-4) were carried out to evaluate the effect of agglomerate size on product ash content. For these tests, all conditions were held constant and the low-shear product sampled at two different times, once when the agglomerates were about 2 mm in size, and once when the agglomerates were about 0.5 mm in size. Results of these two tests, using low-shear samples rinsed in the lab, indicated virtually no difference in product ash content. This result is surprising since during previous testing (particularly during Subtask 6.5, bench-scale testing) larger agglomerates consistently resulted in lower product ash values.

Vibrating Screen Testing

Two tests (I-9-1 and I-9-2) were performed to evaluate the effect of screen spray water flowrate on product ash content. Both of these tests were full plant tests in which a full compliment of selective agglomeration module samples were taken. However, due to continuing problems with the weigh belt feeder, the feedstock grind size decreased between the two tests from a D_{80} of 23.0 microns for Test I-9-1 (low screen spray rate) to a D_{80} of 21.1 microns for Test I-9-2 (high screen spray rate).

Results form these tests indicate a decrease in the final plant product ash content of 0.13% from 2.84 to 2.71%. However, when comparing the low-shear samples rinsed in the lab, a decrease of only 0.05% from 2.81 to 2.76% was observed. As such, it is possible that the higher screen spray rate resulted in some product ash content reduction not attributable to the finer grind size.

Steam Stripper Circuit Testing

One pair of tests (I-9-1 and I-9-2) was carried out to evaluate the effect of stripper operating temperature on the product residual heptane content. For the first test, the steam strippers were operated at pressures of approximately 1 and 5 psi in strippers A and B, respectively. For the second test, the stripper operating pressures were increased to approximately 6 and 10 psi, respectively. Stripper operating temperatures and residual heptane concentrations for these two test are shown in Table 34.

Table 34. Stripper Temperature Effects - Indiana VII Coal

	First Stage Steam Stripper (A)		Second Stage Steam Stripper (B)	
Test	ºF	Residual heptane, ppm dcb	ºF	Residual heptane, ppm dcb
T-9-1	199	13487	231	5152
T-9-2	210	11771	240	4917

As can be seen from this data, there appears to be no significant reduction in the final plant product residual heptane concentration as a result of the increased stripping temperatures. However, the temperature increase resulted in a reduction of the first stage stripping product residual heptane content.

During virtually all of the Indiana VII coal testing completed, residual heptane concentrations were in the 5000 ppm range on a dry coal basis (dcb). However, for one test (I-5), in which a high asphalt dosage (approximately 19 lb/ton) was used, a higher residual heptane concentration (7300 ppm dcb) was found. It was noted during this test that the material in the strippers was very foamy and difficult to pump. As such, this higher residual heptane content was attributed to poor operational control rather than the presence of greater amounts of asphalt. Tailings residual heptane contents for the Indiana VII coal testing were in the 300 to 1000 ppm range on a dry solids basis.

Grinding Requirements

Throughout much of the Indiana VII coal testing, PSDs with D_{80}s in the 20 to 26 micron range were utilized. During this testing, product ash values ranged from a high of 3.58% (2.56 lb/MBtu) to a low of 2.71% (1.92 lb/MBtu). In addition, one laboratory rinsed low shear sample achieved a product ash content of 2.65% (1.88 lb/MBtu). These grinds were achieved by operating the grinding circuit in closed circuit with 2-inch cyclones and 200-mesh screens.

As such, the project product ash content goal of 2 lb/MBtu was met for only the finest grinds evaluated (D_{80}s of 20 to 21 microns during tests I-9-2, I-9-3, and I-9-4). Attempts to repeat these results during the I-10 series of tests were unfortunately spoiled by coarser PSDs (D_{80}s in the 22 to 24 micron range), due to the malfunctioning weigh belt feeder.

Due to the difficulty in producing the 20 micron grind and the difficulty encountered filtering the product at those fine sizes, additional testing specifically targeting the 2 lb/MBtu product ash goal was not carried out. As such, additional PDU SA Module testing with the Indiana VII coal focused on operating at various feed rates and grinding circuit configurations to determine an operating scenario that would provide a feedstock coarse enough to be filtered continuously during the production run, at both a reasonable feed rate and product ash content.

During this work, dry coal feed rates in the 2500 to 4300 lb/hr range were evaluated. Grinding configurations tested used 70-mesh and 100-mesh screens, 2-inch and 3-inch cyclones, and recycling to either the Netzsch fine grinding mill or the secondary ball mill. From this work, it was determined that if the feed rate was greater than 3600 to 3800 lb/hr, the available filtering capacity was exceeded due to the high flowrate of clean coal. Likewise it was found that if the feed rate was lower than this range, the filtering capacity was exceeded due to fineness of the clean coal product. It was also found that in order to allow for continuous filtering, the regrinding would be carried out in the secondary ball mill rather than the Netzsch fine grinding mill. In this scenario, less fines were generated, increasing the filtering capacity.

As such, it was planned that the production run grinding would be carried out at a 3500 to 3800 lb/hr coal feed rate and utilize the 3-inch cyclones, 100-mesh sizing screens, and recycling to the secondary ball mill. It was anticipated that this would provide a clean coal product with an ash content of about 3 lb/MBtu.

Yield and Btu Recovery - Throughout the Indiana VII coal testing, relatively high (85 to 92%) tailings ash contents were consistently achieved. For the few tests in which the tailings ash values were below this range, insufficient asphalt and/or energy was used. Given these high tailings ash values, it is not unexpected that Btu recoveries were consistently greater than 99% with yields in the 90 to 92% range.

However, based on the calculation procedure used to determine Btu recovery (product Btu content divided by the feed Btu content and adjusted for yield), the Btu recoveries were consistently greater than 100%. This is attributed to the very high (>90%) tailings ash values achieved, which undermined the ash balance method utilized. While the use of a correlated correction method, such as the Parr method, would correct the Btu recovery values to below 100%, it was not applied since virtually all of the Btu recovery values would still be greater than 99%.

Indiana VII Coal Production Run

The Indiana VII coal production run was carried out during the week of July 28, 1997. Individual setpoint and average operating conditions and results for the production run are shown in Appendix C along with the previously discussed parametric testing results. As seen from this data, the average production run product ash content was 3.02 lb/MBtu. The range of product ash contents for the individual setpoints were from 2.98 to 3.08 lb/MBtu.

The following is a summary list of average production run conditions and results:

- Dry coal feed rate - 3491 lb/hr
- Plant feed grind D_{80} - 63.9 microns
- Plant feed solids concentration - 12.48%
- Plant feed ash content - 9.8%
- Heptane concentration utilized - 34.8% on a dry ash free coal basis
- Asphalt concentration utilized - 5.4 lb/ton coal
- Total agglomeration (high and low shear) energy input - 36.6 kwhr/ton feed coal (17.9 kwhr/1000 gallon slurry)
- Screen spray water rate - 549 gallons/ton product
- Steam consumption - 1778 lb/ton dry product (2.1 lb/gallon slurry stripped)
- Plant product ash content - 3.02 lb/MBtu (4.19%)
- Plant product residual heptane content - 3967 ppm on a dry coal basis
- Plant tailings ash content - 91.0%
- Plant tailings residual heptane content - 472 ppm on a dry solids basis
- Plant yield - 93.5%
- Plant Btu recovery - 100%

The following represents an approximate summary of the operating schedule for the Indiana VII coal production run:

- Run start: 6:00, July 29
- Shutdown due to filter failure: 12:30 July 30 (30-1/2 hours run time)
- Run restart: 17:30 July 30 (5 hours down time)
- Shutdown due to control valve failure: 4:00 July 31 (10-1/2 hours run time)
- Run restart: 6:00 July 31 (2 hours down time)
- Run end: 6:00 August 1 (24 hours run time)
- **Total approximate run time: 65 hours**
- **Total approximate down time: 7 hours**
- **Total run duration: 72 hours**

The first down time period (5 hours) was due to a torn filter cloth in the EIMCO filter press used to filter the drum filter filtrate. As a result of this torn cloth, the thickener filled with coal, overloading the filtering circuit. As such, the plant was shut down to allow the filtering operation to catch up and the filter cloth to be replaced.

The second down time period (2 hours) was due to the failure of the control valve which maintains pressure on the first stage steam stripper. The failure was caused by excess water in the instrument air line due to a faulty air dryer. No other major problems were encountered during the Indiana VII Coal production run.

Production Run Grinding Circuit Analysis

Samples of the grinding circuit process streams were taken during the production run and analyzed for solids concentration, ash content, and particle size distribution (PSD). Figure 20 presents the grinding circuit utilized during the Indiana VII coal production run. Included in Figure 20 are the dry coal mass flow, the particle size distribution (PSD) D_{80}, and the ash content for pertinent grinding circuit streams.

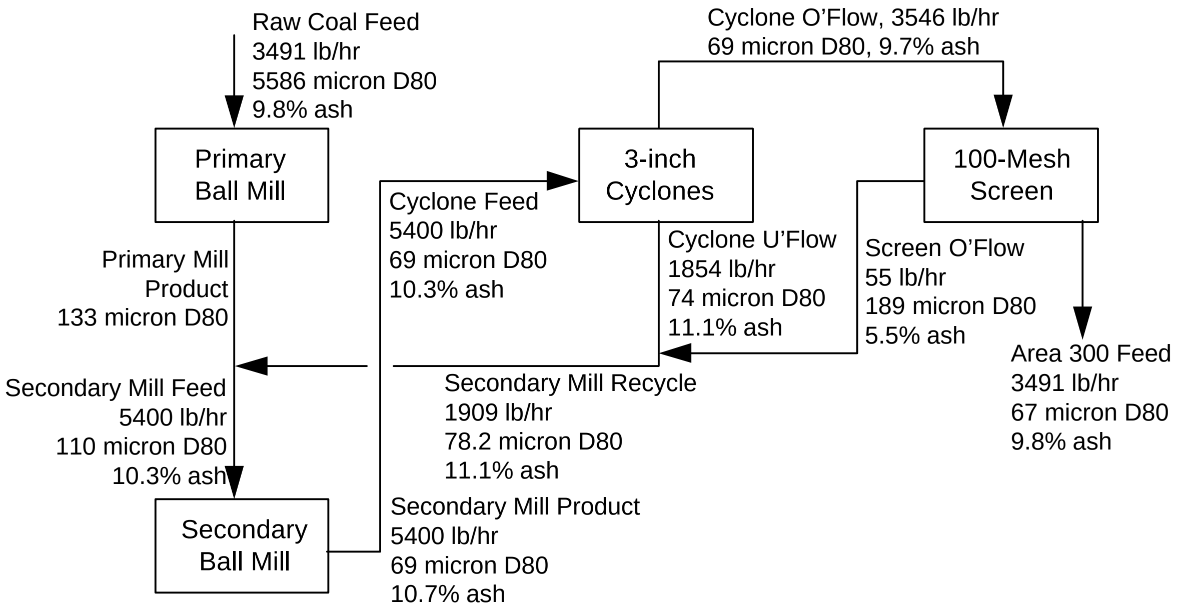


Figure 20. Indiana VII Coal Production Run Grinding Circuit Summary

Production Run Feed, Product, and Tailings Characterization

Composite samples of the crushed coal, ground feed slurry, product slurry, and tailings streams from the Indiana VII Coal production run were submitted for complete analyses. Table 35 presents the analytical results for these production run samples.

Indiana VII Coal Testing Summary

This section of the report summarizes the PDU SA Module testing for the Indiana VII coal. In particular it presents the relationship between feedstock grind size and plant product ash content and a summary list of observations and conclusions.

Particle Size Distribution vs Product Ash Content Relationship

In general, it was found throughout the Indiana VII coal testing, that most operating parameters had only small effects on the product ash content. However, the ground feedstock particle size distribution was found to have a significant effect on the product ash content. This relationship is illustrated in Figure 21 which presents all of the Indiana VII coal complete plant testing results, in the form of plant product ash content as a function of the ground feedstock 80% passing (D_{80}) size, regardless of the plant operating conditions utilized.

Table 35. Indiana VII Production Run Feed, Clean Coal, and Tailings Analyses

	Crushed Coal*	Feed Slurry*	Clean Coal*	Tailings*
Proximate, %:				
Ash	9.55	9.88	4.27	88.94
Volatile Matter	32.92	33.06	35.12	9.09
Fixed Carbon	57.53	57.06	60.61	1.97
Total	100.00	100.00	100.00	100.00
Forms of Sulfur, %:				
Total	0.51	0.46	0.43	1.08
Pyrite	0.15	0.17	0.07	1.17
Sulfate	0.01	< 0.01	< 0.01	0.01
Organic	0.35	0.29	0.36	< 0.01
HHV, Btu/lb	13,028	12,945	13,836	281
Ultimate, %:				
Carbon	75.39		81.24	
Hydrogen	4.74		4.82	
Nitrogen	1.68		1.79	
Sulfur	0.51		0.43	
Ash	9.55		4.27	
Oxygen	8.13		7.45	
Total	100.00		100.00	

* Bone-dry basis

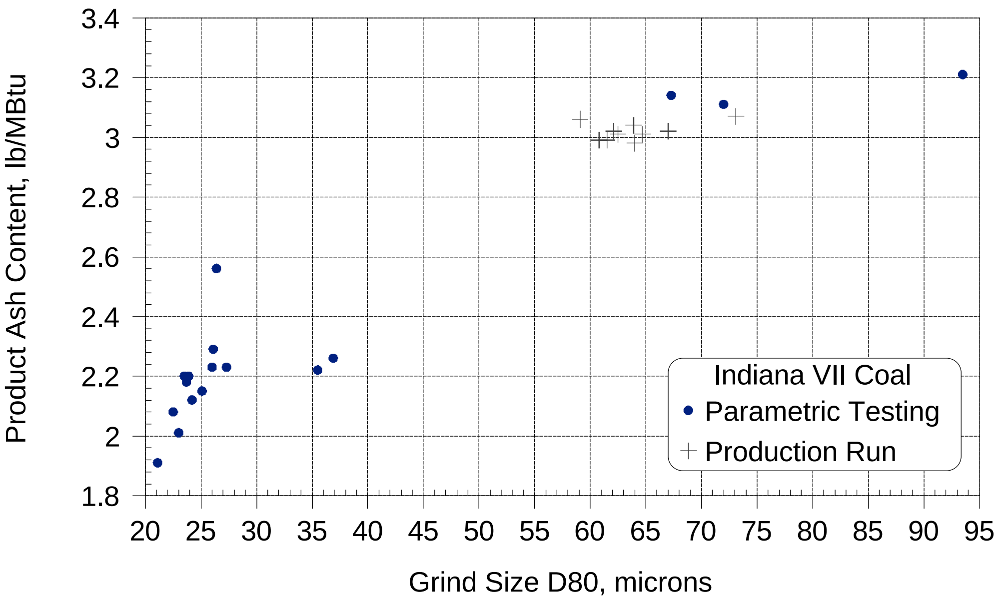


Figure 21. Product Ash Content vs Feedstock PSD D_{80} - Indiana VII Coal

Observations and Conclusions

The following is a list of observations and conclusions based on the Indiana VII coal PDU SA Module testing:

- To achieve consistent asphalt flows to the high-shear circuit, the emulsion first had to be screened at 28-mesh to remove the large particles and then diluted to approximately 2 to 3%.
- Increasing the asphalt dosage to high shear improved the quality of inversion achieved, increased Btu recovery, and increased the tailings ash content.
- There was a small effect of asphalt dosage on product ash content, with higher asphalt dosages resulting in slightly higher product ash values.
- Decreasing the high-shear tip speed (energy input) reduced the quality of inversion achieved and decreased the tailings ash content.
- There is a clear effect of high-shear energy input on product ash content, with lower energy resulting in higher product ash values.
- Increasing the asphalt dosage and decreasing high-shear energy input simultaneously, resulted in a higher product ash content.
- To achieve the lowest product ash content at a given grind size, the asphalt dosage should be minimized and sufficient energy used to achieve the formation of good agglomerates.
- Increasing the high-shear solids concentration resulted in more particle to particle contact at similar energy inputs and a better quality inversion.
- Higher low-shear solids concentrations had no detrimental effect on product ash content.
- No difficulties were encountered when operating the low-shear vessel at increased solids concentrations.
- There was no significant increase in product ash content due to higher low-shear impeller tip speeds.
- Operation of low shear at reduced (3 m/s) impeller tip speeds resulted in poor agglomerate growth indicating that the lower tip speed did not supply sufficient energy for consistent agglomerate growth.
- Contrary to previous testing results, there was no observed difference in the product ash content as a function of agglomerate size
- Higher screen spray water flow rates resulted in a small reduction in product ash content.
- There was no significant reduction in the final plant product residual heptane concentration at increased stripping temperatures. However, the temperature increase resulted in a reduction of the first stage stripping product residual heptane content.
- Product residual heptane concentrations were in the 3000 to 5000 ppm range on a dry solids basis.

- Tailings residual heptane concentrations were in the 300 to 1000 ppm range on a dry solids basis.
- A feedstock grind size D_{80} of approximately 20 microns was required to achieve the product ash target of 2 lb/MBtu.
- Btu recoveries were consistently greater than 99% with yields in the 90 to 92% range.
- Tailings ash contents were consistently in the 85 to 92% range.

Clean Coal Ash Properties

Hazen Research Inc., of Golden, CO determined the ash chemistry and fusion properties of the feed and clean coal samples from the extended production PDU runs on the Taggart, Indiana VII, and Hiawatha coals. It was found that PDU selective agglomeration cleaning consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals and a small increase in the fusion temperature of the ash in the Hiawatha coal. The softening (spherical) temperatures are compared in Figure 22 to illustrate the difference caused by the cleaning. The complete set of fusion temperatures are listed in Table 36. It should be noted that the shipment of Indiana VII coal cleaned by selective agglomeration seemed to have a more siliceous ash than previous shipments. This may be the reason that the fusion temperatures of this ash were not affected as much by the agglomeration cleaning as was the ash in the Indiana VII coal cleaned by flotation and reported in the Subtask 8.5 Topical Report [11].

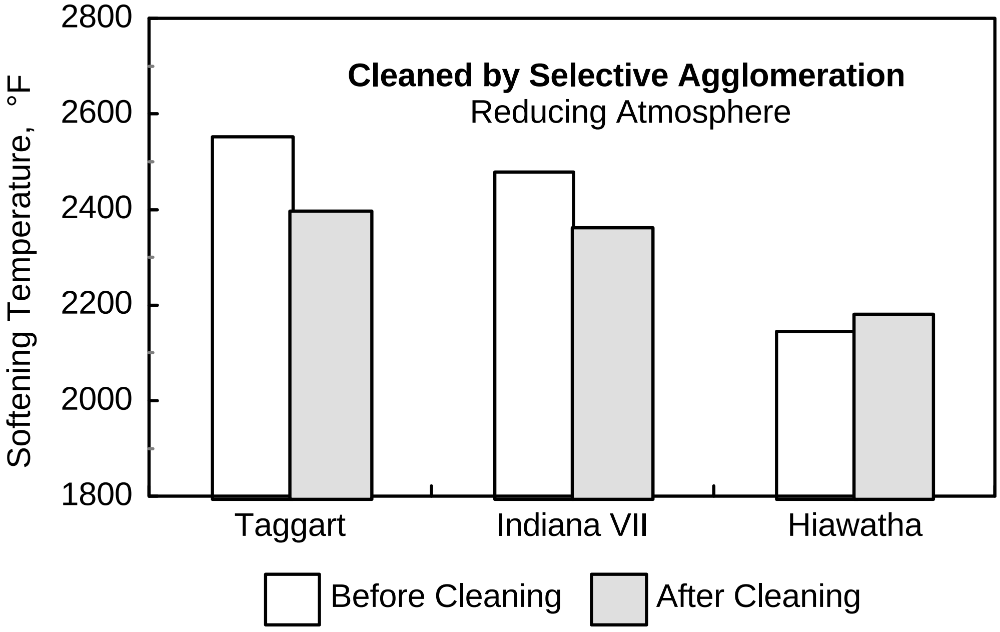


Figure 22. Softening Temperatures of Ash in Test Coals

The ash compositions of the coals are presented in Table 37 along with slag viscosity calculations and assessments of the slagging and fouling characteristics of the ash. The calculated viscosities agree with the fusion temperature measurements. Except for titania, and perhaps the phosphorus in the case of the Hiawatha coal, the concentrations of the ash constituents were significantly reduced on a heating value (lb/MBtu) basis by agglomeration in the PDU. These ash constituent concentration reduction data are presented in Tables 38, 39, and 40 for the Taggart, Indiana VII, and Hiawatha coals, respectively.

Toxic Trace Elements Distribution

Samples of the crushed feed coal, ground agglomeration feed coal, clean coal, and fine refuse from the extended production PDU runs with the Taggart and Hiawatha coals were submitted to Huffman Laboratories, of Golden, CO for determination of the concentrations of twelve toxic trace elements. Similar samples from parametric test I-9-2 on Indiana VII coal were also submitted for these analyses. Samples for test I-9-2 were used because the target ash specification of less than 2 lb/MBtu was met during that test but was not met during the production run on the Indiana VII coal. The toxic trace elements were antimony, arsenic, beryllium, cadmium, chromium, cobalt, lead, manganese, mercury, nickel, selenium and chlorine. The perchloric acid dissolution/atomic absorption, total halides, and cold-vapor spectroscopy methods used to analyze these samples were the same as the methods used to analyze the samples from the bench-scale testing [7,12,13].

The analytical results for the clean coals, as-received test coals, and the run-of-mine (ROM) coals are presented in Table 41. The analyses of the PDU feed and clean coal products are also compared in Figures 23, 24, 25, and 26. The concentrations of mercury and selenium are of particular interest, and are shown in Figures 25 and 26. The concentrations of these two elements in the Taggart, Indiana VII, and Hiawatha coals were little changed by the advanced fine coal cleaning.

Table 36. Fusion Temperatures (°F) of Ash Before and After Agglomeration

	Taggart Coal		Indiana VII Coal		Hiawatha Coal	
	Before Cleaning	After Cleaning	Before Cleaning	After Cleaning	Before Cleaning	After Cleaning
Oxidizing Atmosphere:						
Initial	2570	2485	2540	2482	2170	2290
Softening	2657	2618	2583	2541	2230	2306
Hemispherical	2695	2630	2600	2560	2300	2319
Fluid	2710	2680	2625	2590	2445	2333
Reducing Atmosphere:						
Initial	2286	2236	2300	2270	2084	2120
Softening	2552	2396	2479	2362	2145	2181
Hemispherical	2600	2475	2489	2400	2255	2195
Fluid	2664	2600	2500	2455	2346	2220

Table 37. Ash Chemistry of Test Coals Cleaned by Selective Agglomeration

	Taggart Coal			Indiana VII Coal			Hiawatha Coal		
	Before Cleaning	After Cleaning	Reduction Percent*	Before Cleaning	After Cleaning	Reduction Percent*	Before Cleaning	After Cleaning	Reduction Percent*
Ash Constituent, %:									
SiO_2	47.83	46.24	56	59.51	58.96	58	51.18	38.54	74
Al_2O_3	26.97	28.81	51	26.20	24.68	60	17.25	21.87	56
TiO_2	1.10	1.92	20	1.06	2.20	12	0.95	2.22	20
Fe_2O_3	10.10	14.12	36	4.38	6.56	36	5.24	7.29	52
CaO	1.60	2.05	41	2.31	1.63	70	11.60	8.49	75
MgO	0.72	0.60	62	0.57	0.67	50	1.02	0.78	74
Na_2O	1.21	0.81	69	0.67	0.71	55	2.48	4.30	40
K_2O	2.96	2.27	65	2.92	3.20	54	0.64	0.56	70
P_2O_5	0.23	0.29	42	0.13	0.19	38	0.51	1.03	30
SO_3	1.24	1.52	44	3.91	1.34	86	9.16	10.20	62
Ash Viscosity Calculations:									
Base Content, %	17.94	20.50		11.11	12.95		23.22	25.48	
Acid Content, %	82.06	79.50		88.89	87.05		76.78	74.52	
Dolomite Ratio	13.98	13.35		26.54	18.01		60.15	43.28	
Base/Acid Ratio	0.22	0.26		0.13	0.15		0.30	0.34	
Silica/Alumina Ratio	1.77	1.60		2.27	2.39		2.97	1.76	
T(cv), °F	2800	2675		2689	2600		2455	2395	
T250 Temp, °F	2677	2603		> 2800	> 2800		2541	2480	
Equiv Silica, %	79.87	73.39		89.13	86.94		74.13	69.95	
Viscosity at 2600 °F, P	740.95	261.13		> 1000	> 1000		294.90	149.01	
Ash Type	High Rank	High Rank		High Rank	High Rank		Lignite	Lignite	
Slagging/Fouling Characteristics:									
Slagging Type	Low	Low		Low	Low		Medium	Medium	
Fouling Type	Medium	Medium		Low	Low		Low	Medium	

* Percentage reduction calculated on a heating value (lb/MBtu) basis

Table 38. Ash Constituent Concentration Reductions - Taggart Coal

	Concentration in Clean Coal lb/MBtu	Reduction from Concentration in As- Received Coal, %
Ash	1.06	54
Sulfur, total	0.45	5
Sulfur, pyrite	0.04	22
Sulfur, sulfate	< 0.01	> 50
SiO_2	0.488	56
Al_2O_3	0.304	51
TiO_2	0.020	20
Fe_2O_3	0.149	36
CaO	0.022	41
MgO	0.006	62
Na_2O	0.009	69
K_2O	0.024	65
P_2O_5	0.003	42
SO_3	0.016	44

Notes: Production run heating value recovery = 99.2 percent.
Reductions are on a heating value basis.

Table 39. Ash Constituent Concentration Reductions - Indiana VII Coal

	Concentration in Clean Coal lb/MBtu	Reduction from Concentration in As- Received Coal, %
Ash	3.08	58
Sulfur, total	0.31	21
Sulfur, pyrite	0.03	69
Sulfur, sulfate	0.015	6
SiO_2	1.175	58
Al_2O_3	0.492	60
TiO_2	0.044	12
Fe_2O_3	0.131	36
CaO	0.032	70
MgO	0.013	50
Na_2O	0.014	55
K_2O	0.064	54
P_2O_5	0.004	38
SO_3	0.027	86

Notes: Production run heating value recovery = 99+percent.
Reductions are on a heating value basis.

Table 40. Ash Constituent Concentration Reductions - Hiawatha Coal

	Concentration in Clean Coal lb/MBtu	Reduction from Concentration in As- Received Coal, %
Ash	1.96	66
Sulfur, total	0.36	9
Sulfur, pyrite	0.04	60
Sulfur, sulfate	< 0.01	> 7
SiO_2	0.757	74
Al_2O_3	0.430	56
TiO_2	0.044	20
Fe_2O_3	0.143	52
CaO	0.167	75
MgO	0.015	74
Na_2O	0.084	40
K_2O	0.011	70
P_2O_5	0.020	30
SO_3	0.200	62

Notes: Production run heating value recovery = 98.9 percent.
Reductions are on a heating value basis.

Table 41. Toxic Trace Elements in Coals

	Analyses, % or ppm			Reduction on Heating Value Basis*, %	
	Clean Coal	As-Rec'd Test Coal	ROM Coal	from As-Rec'd Test Coal	from ROM Coal
Taggart:					
Ash, %	1.59	3.48	34.70	55	97
S(tot), %	0.67	0.70	0.46	6	0
S(pyr), %	0.06	0.07	0.02	16	neg
Sb, ppm	0.8	0.8	0.17	7	neg
As, ppm	2.3	3.7	2.5	40	37
Be, ppm	2.0	2	2.0	2	31
Cd, ppm	< 0.1	< 0.1	0.1	ind	> 31
Cr, ppm	7	6	30	neg	83
Co, ppm	8.8	8.7	12	1	49
Pb, ppm	3	4	38	27	95
Mn, ppm	4.0	7.0	110	44	97
Hg, ppm	0.01	0.02	0.03	51	77
Ni, ppm	11	11	11	2	31
Se, ppm	1.36	1.52	1.39	11	32
Cl, ppm	140	192	177	29	45
Indiana VII:					
Ash, %	2.76	9.45	38.10	73	96
S(tot), %	0.40	0.54	0.77	31	69
S(pyr), %	0.04	0.10	0.51	63	95
Sb, ppm	2.4	1.1	1.2	neg	neg
As, ppm	0.62	1.27	4.1	54	91
Be, ppm	2.3	2.3	2.3	7	42
Cd, ppm	< 0.1	< 0.1	0.1	ind	> 39
Cr, ppm	13	14	22	9	64
Co, ppm	9.5	9.4	11	5	49
Pb, ppm	7	6	14	17	70
Mn, ppm	8	28	150	72	97
Hg, ppm	< 0.01	< 0.01	0.02	ind	> 70
Ni, ppm	53	50	30	neg	neg
Se, ppm	0.51	0.45	0.78	neg	61
Cl, ppm	42	41	38	2	34
Hiawatha**:					
Ash, %	2.81	7.52		65	
S(tot), %	0.52	0.56		13	
S(pyr), %	0.07	0.11		40	
Sb, ppm	0.1	0.09		neg	
As, ppm	0.5	0.7		41	
Be, ppm	0.3	0.3		6	
Cd, ppm	< 0.1	< 0.1		ind	
Cr, ppm	9	4.8		neg	
Co, ppm	0.9	0.8		neg	
Pb, ppm	< 2	< 2		ind	
Mn, ppm	3.0	8		65	
Hg, ppm	0.01	0.01		6	
Ni, ppm	3	1		neg	
Se, ppm	1.07	1.1		10	
Cl, ppm	216	268		24	

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* neg = negative number; ind = could not be calculated

** Hiawatha ROM coal will be the same as the as-received test coal

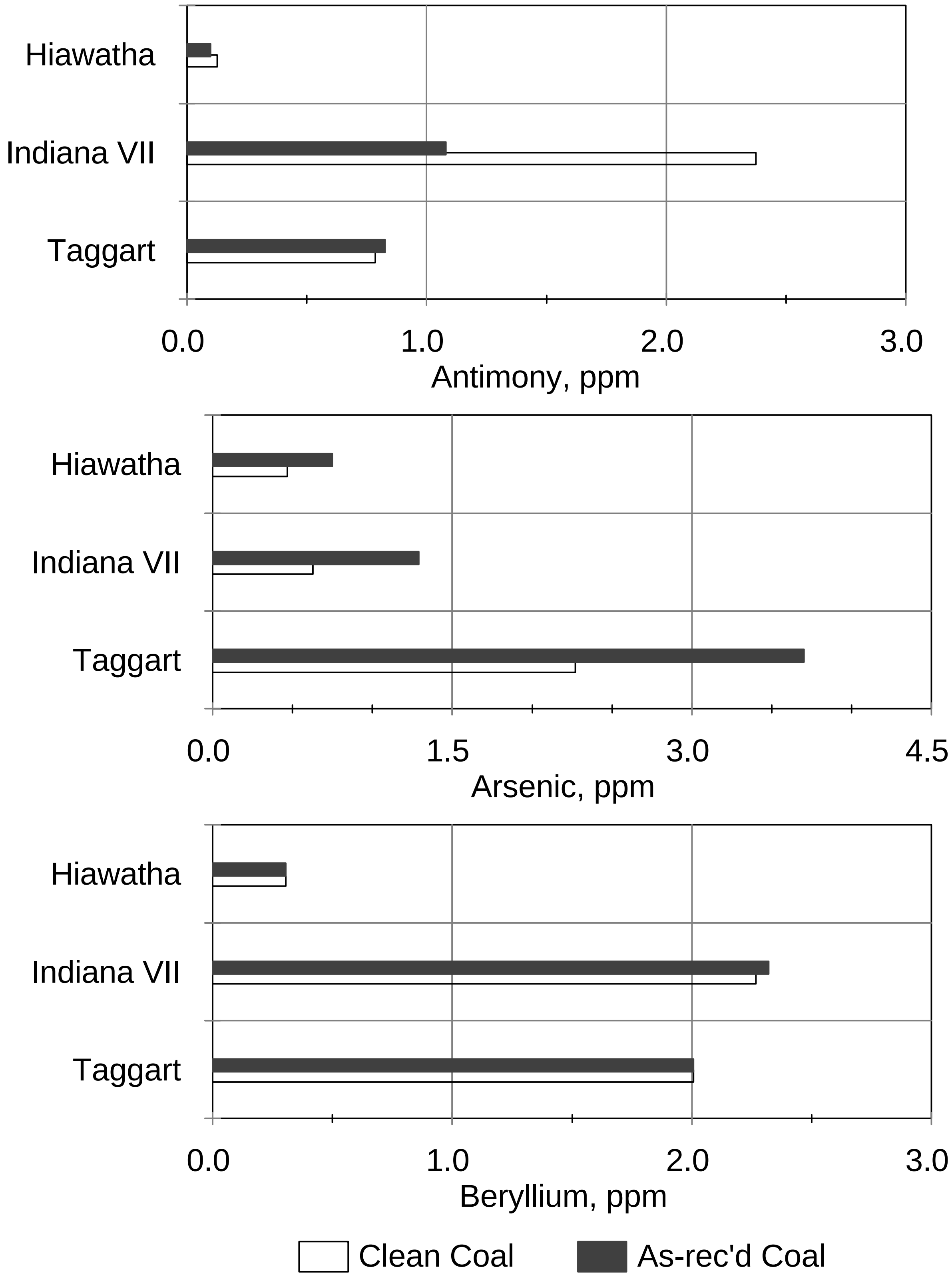

Figure 23. Antimony, Arsenic and Beryllium Analyses

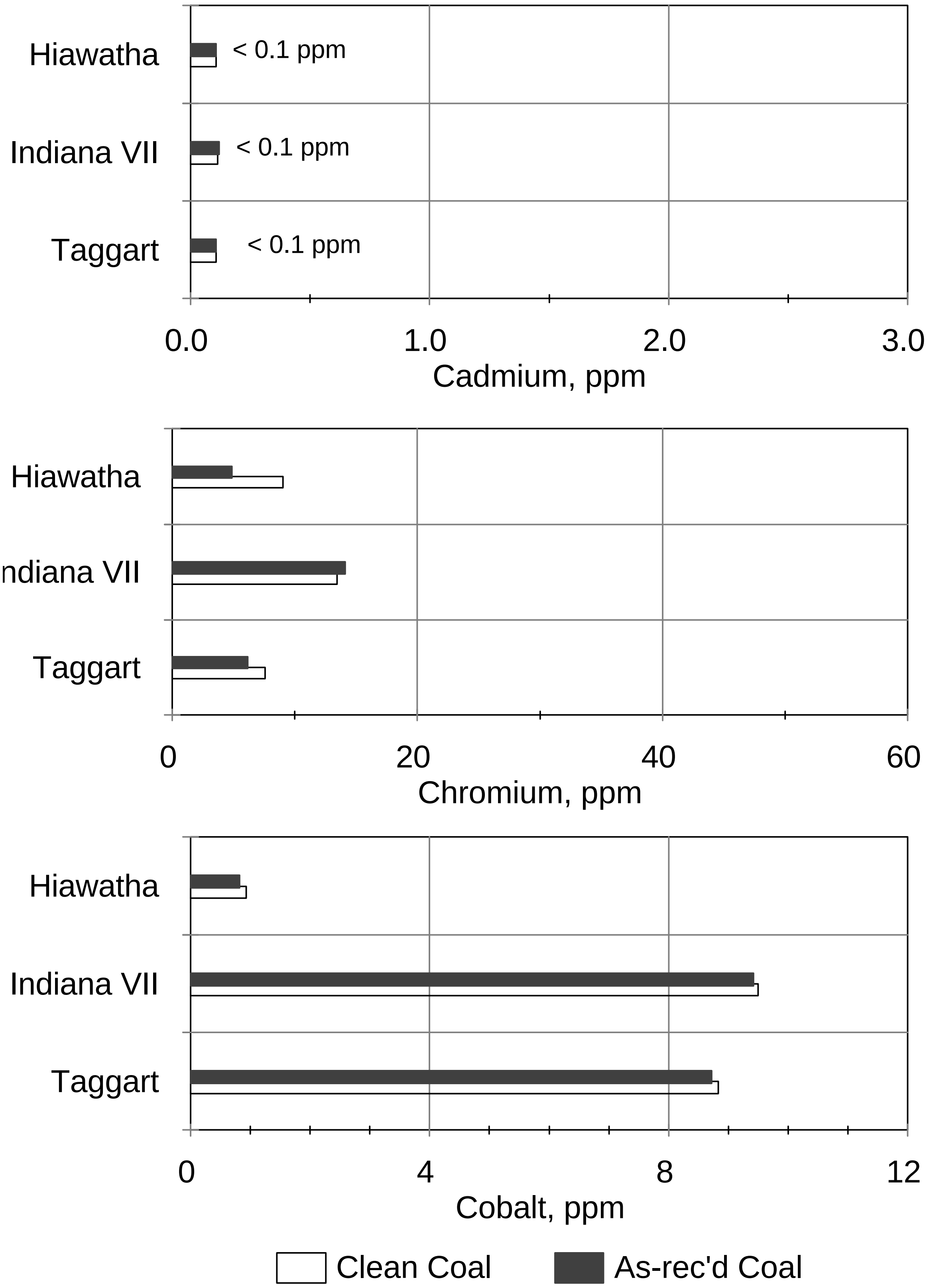

Figure 24. Cadmium, Chromium and Cobalt Analyses

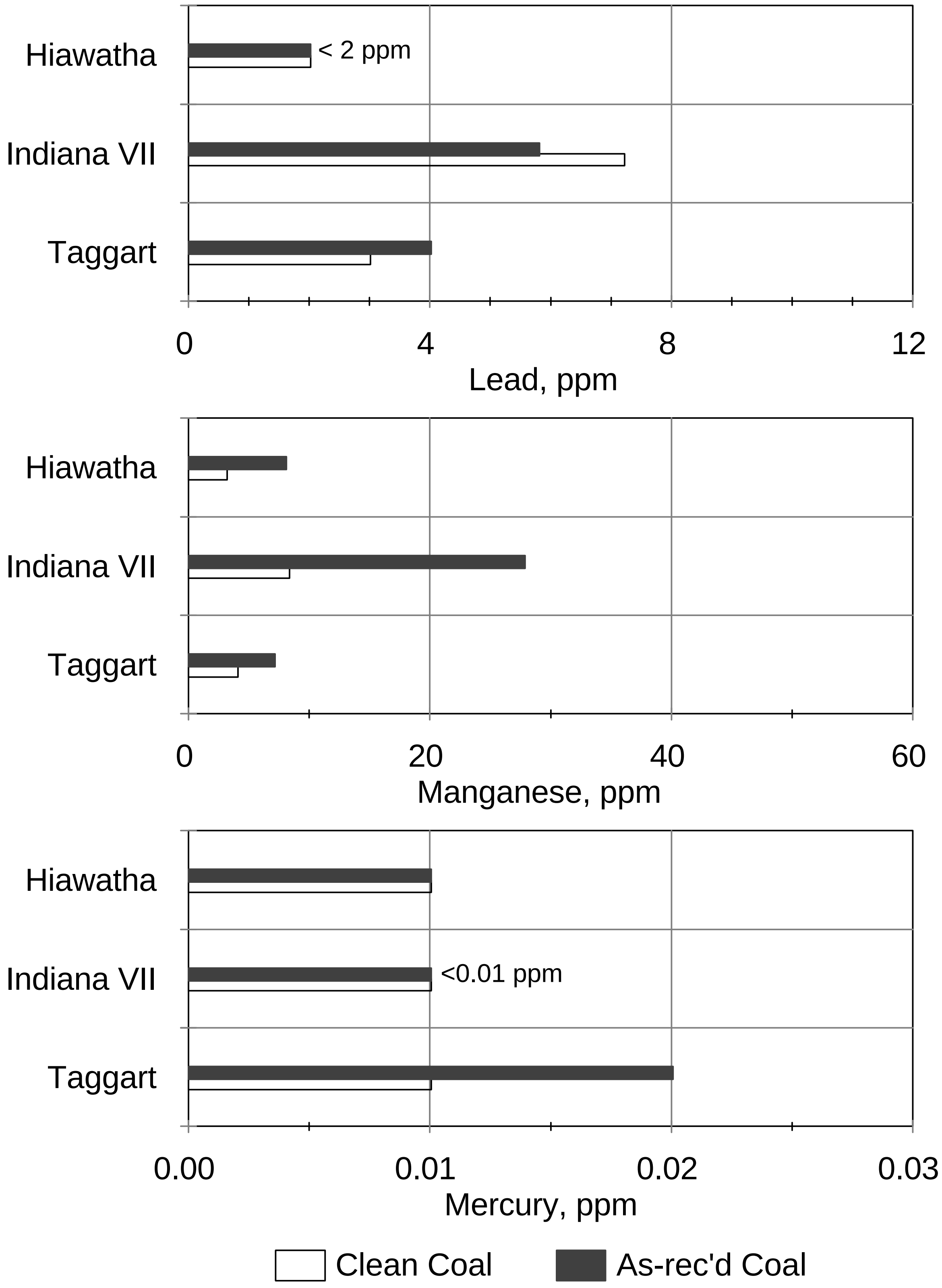

Figure 25. Lead, Manganese and Mercury Analyses

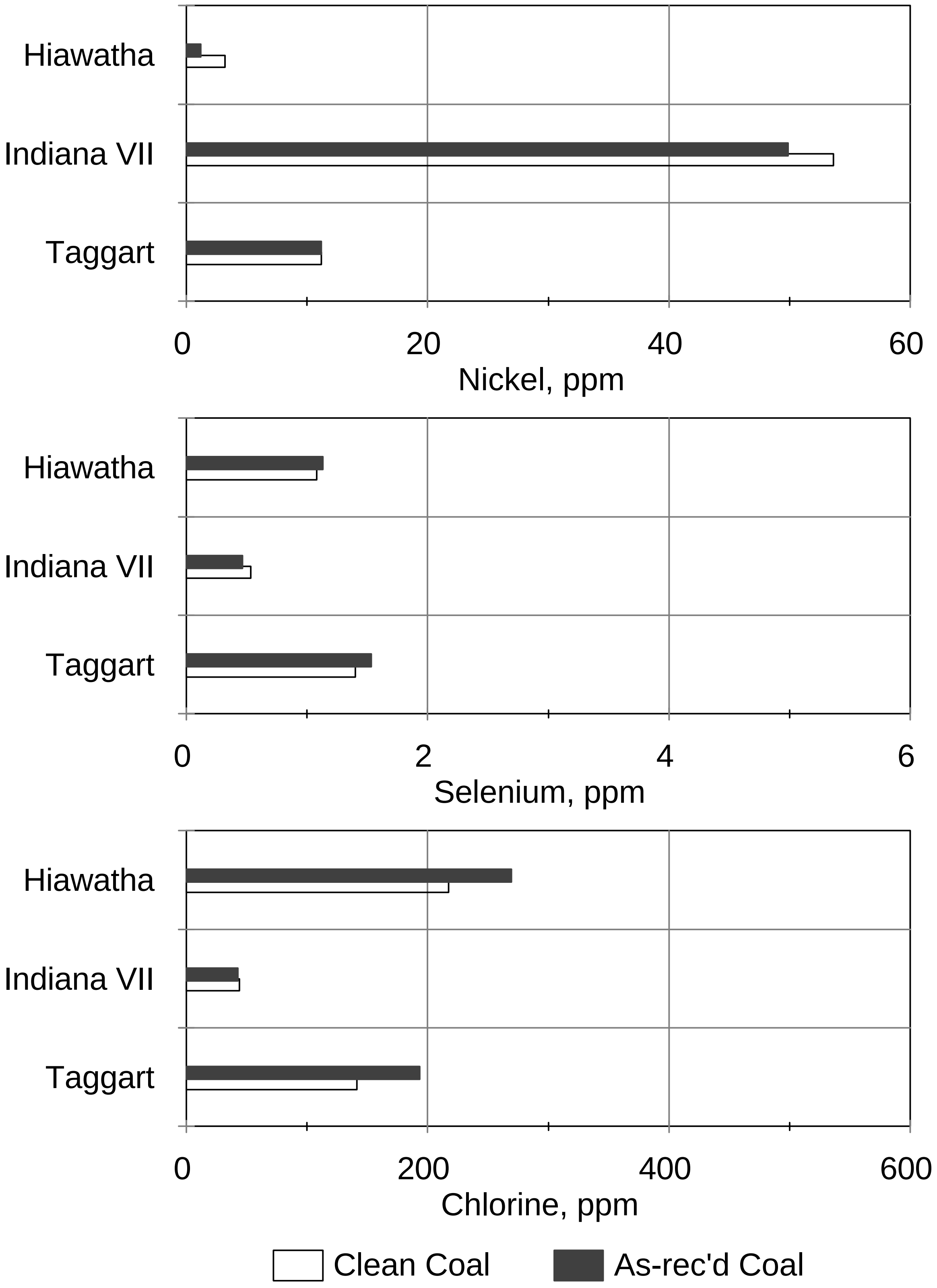

Figure 26. Nickel, Selenium and Chlorine Analyses

The variations in trace element concentrations from coal to coal seen for these samples were similar to the variations seen for the set of samples from the bench-scale testing [7,12]. As listed in Table 41, there were substantial reductions, over 25 percent on a heating value basis, in the residual concentrations of arsenic and manganese for all three as-received test coals. The reduction in the concentrations of mercury and chlorine varied from coal to coal. The PDU agglomeration did not appear to have reduced the concentration of antimony beryllium, cadmium, chromium, cobalt, nickel and selenium in any these coals on a heating value basis.

The residual concentrations of all twelve trace elements in the Taggart and Indiana VII clean coals were especially lower than their concentrations in the two ROM parent coals. On the other hand, only the arsenic and manganese were substantially reduced from the amounts in the as-received Hiawatha coal even though the latter coal had not been washed at the mine before marketing.

The agglomeration feed and clean coal analyses are reported in Tables 42, 43, and 44 along with the ROM coal, as-received coal, and fine refuse analyses for the Taggart, Indiana VII, and Hiawatha coals, respectively.

Table 42. Toxic Trace Element Analyses - Taggart Coal Production Run

	ROM Coal	As-Recv’d Coal	Agglom. Feed	Clean Coal	Fine Refuse	Calc’d Feed	Mass Balance Closure, %
Ash, %	34.70	3.48	3.44	1.59	62.47	3.60	105
Sulfur, tot, %	0.46	0.70	0.68	0.67	0.88	0.68	100
Sulfur, pyr, %	0.02	0.07	0.07	0.06	0.47	0.07	105
Sulfur, sulf, %	0.01	0.02	0.02	< 0.01	0.13	< 0.01	21
HHV, Btu/lb	9,936	14,735	14,688	15,072	4,260	14,859	101
Antimony, ppm	0.17	0.8	0.7	0.8	0.7	0.78	109
Arsenic, ppm	2.47	3.7	3.5	2.3	30	3.19	91
Beryllium, ppm	2.0	2	2.1	2.0	2.5	2.02	96
Cadmium, ppm	0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	ind.
Chromium, ppm	30	6	14	7	174	12.90	92
Cobalt, ppm	12	8.7	9.3	8.8	22	9.24	99
Lead, ppm	38	4	5	3	34	4.02	80
Manganese, ppm	110	7.0	13.0	4.0	229	11.43	88
Mercury, ppm	0.03	0.02	0.02	0.01	0.08	0.01	62
Nickel, ppm	11	11	13	11	50	12.29	95
Selenium, ppm	1.39	1.52	1.48	1.38	4.15	1.47	99
Chlorine, ppm	177	192	152	140	115	139	92

Notes: Production run heating value recovery = 99.2 percent.
Production run yield = 96.7 percent.
Analyses are on a dry coal basis (Huffman Laboratories).

Table 43. Toxic Trace Element Analyses - Indiana VII Coal Test I-9-2

	ROM Coal	As-Recv'd Coal	Agglom. Feed	Clean Coal	Fine Refuse	Calc'd Feed	Mass Balance Closure, %
Ash, %	38.10	9.45	10.12	2.76	90.00	10.35	102
Sulfur, tot, %	0.77	0.54	0.43	0.40	0.76	0.43	101
Sulfur, pyr, %	0.51	0.10	0.12	0.04	0.63	0.09	75
Sulfur, sulf, %	0.03	0.02	0.01	0.02	0.02	0.02	200
HHV, Btu/lb	8,382	13,028	12,945	13,836	281	13,698	106
Antimony, ppm	1.2	1.08	1.17	2.37	0.22	2.18	186
Arsenic, ppm	4.1	1.27	1.16	0.62	5.40	1.03	89
Beryllium, ppm	2.3	2.3	2.2	2.3	1.8	2.22	99
Cadmium, ppm	0.1	< 0.1	0.2	< 0.1	1.6	< 0.23	ind.
Chromium, ppm	22	14	23	13	83	19.41	83
Cobalt, ppm	11	9.4	9.2	9.5	12	9.69	106
Lead, ppm	14	6	9	7	48	10.79	118
Manganese, ppm	150	28	39	8	392	41.56	107
Mercury, ppm	0.02	< 0.01	< 0.01	< 0.01	0.05	< 0.01	ind.
Nickel, ppm	30	50	50	53	37	52.08	104
Selenium, ppm	0.78	0.45	0.54	0.51	0.30	0.50	92
Chlorine, ppm	38	41	40	42	23	41	102

Notes: Test I-9-2 heating value recovery = 99+ percent.
Test I-9-2 yield = 91.3 percent.
Analyses are on a dry coal basis (Huffman Laboratories).

Mass balances of the trace elements are also shown in these tables. Mass balance closures were usually between 80 and 120 percent of amount indicated by the agglomeration feed analyses. This degree of agreement is probably as good as can be expected considering the precision of the analyses (usually to only one or two significant figures at best). The as-received coal and the agglomeration feed analyses are on different cut samples taken before and after grinding, respectively.

Table 44. Toxic Trace Element Analyses - Hiawatha Coal Production Run

	ROM Coal	As-Recv'd Coal	Agglom. Feed	Clean Coal	Fine Refuse	Calc'd Feed	Mass Balance Closure, %
Ash, %	7.52	7.52	8.00	2.81	78.62	8.27	103
Sulfur, tot, %	0.56	0.56	0.54	0.52	0.83	0.54	100
Sulfur, pyr, %	0.11	0.11	0.12	0.07	0.71	0.12	97
Sulfur, sulf, %	0.01	0.01	< 0.01	< 0.01	0.03	< 0.01	
HHV, Btu/lb	13,470	13,470	13,399	14,302	875	14,056	105
Antimony, ppm	0.09	0.09	0.1	0.1	0.1	0.12	121
Arsenic, ppm	0.7	0.7	0.7	0.5	5	0.78	102
Beryllium, ppm	0.3	0.3	0.3	0.3	0.4	0.31	102
Cadmium, ppm	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.10	ind.
Chromium, ppm	4.8	4.8	20	9	158	19.64	98
Cobalt, ppm	0.8	0.8	0.9	0.9	4	1.14	127
Lead, ppm	< 2	< 2	< 2	< 2	< 20	< 3.30	ind.
Manganese, ppm	8	8	13	3.0	144	13.15	101
Mercury, ppm	0.01	0.01	0.01	0.01	0.04	0.01	122
Nickel, ppm	1	1	6	3	43	5.88	96
Selenium, ppm	1.1	1.1	1.07	1.07	1.54	1.10	103
Chlorine, ppm	268	268	191	216	19	202	106

Notes: Production run heating value recovery = 98.9 percent.
Production run yield = 92.8 percent.
Analyses are on a dry coal basis (Huffman Laboratories).

LESSONS LEARNED

Based on the test work and operation of the PDU Flotation Module, the following general lessons were learned:

- Feed coal should be stored in a silo for protection from the elements. Coal left uncovered results in material handling problems due to freezing or sticking at transfer points. Also, surface oxidation of exposed coal may adversely affect agglomeration.
- Sumps should be designed with enough capacity that small changes in volume do not produce large fluctuations in level readings.
- Proposed ball mill charges should be reviewed for proper loading and ball size. PDU ball mills were initially improperly charged resulting in inefficient grinding and premature ball wear.
- Ball mill discharge magnets should be used for the removal of degraded grinding media.
- Multi-stage cycloning, instead of cycloning backed by top-size screen control, would allow for higher solids concentrations in the agglomeration feed. This would improve economics in both the grinding and agglomeration areas.

- All agitated tanks should be baffled to avoid vortexing, pump cavitation, and inaccurate level readings.
- Production of a ground feedstock with consistent solids concentration and size consist is important for producing agglomerates of consistent size. It was found that both of these parameters ultimately effect agglomerate growth and size.
- Production of consistently sized agglomerates from the low-shear unit operation is important for product ash and handling considerations.
- Low-shear reactors should provide only one mixing zone per vessel. The use of dual mixing zones results in difficult to control agglomerate growth.
- The separation of agglomerates from tailings via a vibrating screen should be performed in a downhill orientation to reduce agglomerate bed depth and product ash content.
- Froth skimming of carbonaceous material from the screen underflow should be carried out in a column-style vessel with the recovered material recycled to the high-shear unit operation.
- Recovered agglomerates should not be stored in an agitated tank prior to the steam stripping circuit due to their buoyancy and possible additional growth.
- Agglomerates should be fed to the stripping circuit via a diaphragm pump.
- Feed to the second stage of steam stripping should be via a positive displacement pump rather than a centrifugal pump to avoid high velocity flow reversal.
- The steam stripping circuit should include provisions for the removal of coal fines, carried within the vapor stream, prior to the gravity separation unit operation.
- During steam stripping, the process and instrument design must assure that the various pressure control loops required do not interact to produce operating instabilities.
- The scale-up methodology developed by the project team for the design of coal agglomeration agitation equipment is robust and reliable.
- Dewatering equipment should be designed specifically for its intended use to avoid low filtering capacity and unscheduled downtime.
- No deleterious effects were observed on the selective agglomeration process due to the use of recycled process water.

CONCLUSIONS AND RECOMMENDATIONS

The worked completed during this projected has provided considerable insight into the scale-up, design, operation, and performance of a heptane-based selective agglomeration process, and its related unit operations, as well as the need for further research in this area. A summary of relevant conclusions and recommendations is presented below.

CONCLUSIONS

Program Success

The work and results related to this project should be considered highly successful. The 2 t/hr selective agglomeration module was operated from November, 1996 through July, 1997 processing over 800 tons of the Taggart, Indiana VII, and Hiawatha coals. Parametric testing was performed on each test coal followed by optimization test work and a round-the-clock production run. A substantial amount of each coal's clean product was transported to Penn State University for combustion testing. Overall, the Taggart coal was cleaned to produce a 1 lb ash/MBtu product while the Indiana VII and Hiawatha coals were cleaned to produce a 2 lb ash/MBtu product. Not only were the project goals achieved, the process equipment performed well in terms of reliability and control. A commercial plant cost study performed by Bechtel [14], estimated the cost of production for premium quality coal water slurry fuel to be $2.42/MBtu which met the overall project goal.

Operation and Performance of the SA Module

The operation and performance of the SA Module was very successful. The well instrumented plant proved relatively simple to operate and maintain and was easily capable of producing premium quality fuel. Overall, the SA Module was able to reach steady-state conditions within approximately one hour and maintain production levels with little variance, assuming a consistent quality feedstock was used. Extended production runs indicated that selective agglomeration was a dependable means of cleaning coal to high quality levels at very high energy recoveries.

Figure 27 presents the SA module testing results for all three coals in the form of product ash content in lb/MBtu vs feedstock 80% passing size (D_{80}) in microns.

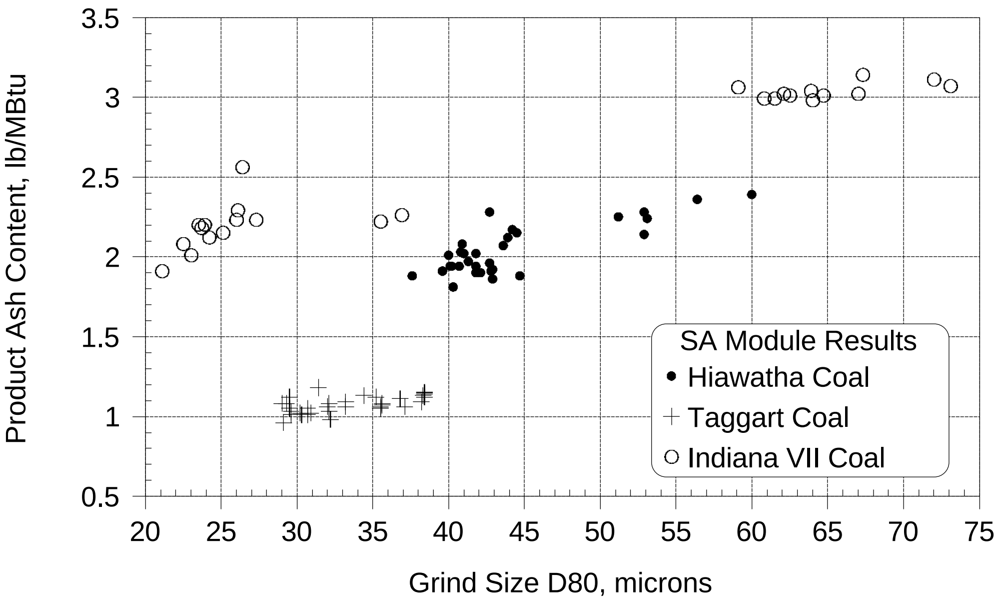

Figure 27. Selective Agglomeration Module Testing Results Summary

Table 45 presents a summary of the PDU SA Module performance for the Taggart and Hiawatha production runs, and for an Indiana VII coal test in which the product ash target was met (product ash target was not a goal of Indiana VII coal production run).

Table 45. PDU SA Module Performance Summary

Coal	PSD D_{80}, microns	Ash, lb/MBtu	Sulfur, lb/MBtu	Yield, %	Btu Recovery, %
Taggart	30	1.06	0.67	96.7	>99
Indiana VII	20	1.91	0.35	91.3	>99
Hiawatha	42	1.93	0.4	92.8	98.9

Important Process Variables

Testing of the three coals in the PDU SA Module indicated that several process variables were important to proper operation. The most important variables and their effects on performance are discussed below:

- Feedstock PSD - The grind size of the slurry feedstock was found to have the greatest impact on product ash contents. In addition, it was found that a consistent feedstock PSD was important in the production of consistently sized agglomerates.
- High-shear agglomeration should be performed at a high solids concentration to minimize high-shear energy requirements. The practical limit for this solids loading, from an agglomeration view point is on the order of 15 to 20% solids. However, this limit is really determined by the grinding circuit capabilities.

- High-shear impeller tip speeds on the order of 10 to 15 m/s are required to insure the occurrence of phase inversion and subsequent agglomerate growth in low shear.
- High-shear residence time requirements are coal dependent but were typically found to be between 30 seconds for the Taggart coal and 120 seconds for the Indiana VII coal.
- High-shear energy requirements ranged from approximately 10 to 15 kwhr/ton coal for the Taggart and Hiawatha coals, to as high as 30 to 35 kwhr/ton for the Indiana VII coal.
- Low-shear agglomeration is best carried out in a single stage providing a residence time of about 2 to 3 minutes allowing agglomerate growth to 2 to 3 mm in size.
- The best compromise between low-shear growth control and product ash content was achieved at solids concentrations in the 7 to 10% range.
- Steam stripping should be performed in two stages. In the first stage, the bulk of the heptane is removed to produce a handleable product while in the second stage elevated temperatures are used to remove additional hydrocarbons.

Clean Coal Ash Properties

It was found that selective agglomeration consistently increased the base/acid ratio of the ash and decreased the silica/alumina ratio. The overall results were declines in the reducing atmosphere fusion temperatures of the ash in the Taggart and Indiana VII coals and a small increase in the fusion temperature of the ash in the Hiawatha coal.

Toxic Trace Elements Distribution

The same variations in trace element concentrations from coal to coal were seen for coal samples cleaned in the PDU SA Module as were seen for the set of samples from the bench-scale testing and from the PDU Flotation Module. There were substantial reductions, over 25 percent on a heating value basis, in the residual concentrations of arsenic and manganese from the amounts in all three as-received test coals. The reduction in the concentrations of mercury and chlorine varied from coal to coal. Agglomeration did not appear to have reduced the concentration of antimony, beryllium, cadmium, chromium, cobalt, nickel and selenium in any these coals on a heating value basis.

The residual concentrations of all twelve trace elements in the Taggart and Indiana VII clean coals were especially lower than the concentrations in their respective ROM parent coals on a heating value basis. On the other hand, only the arsenic and manganese concentrations were substantially reduced from the amounts in the as-received Hiawatha coal even though the latter coal had not been washed at the mine before marketing.

RECOMMENDATIONS

Commercial Plant Design

The design of any commercial SA plant should be based on sound scale-up data. This data should ultimately be obtained from the operation of a plant that utilizes a single train of the largest practical agglomeration equipment that can be fabricated, estimated to be in the 20 to 25 t/hr range.

The maintenance of selective agglomeration equipment should also be considered thoroughly for a commercial plant design. In particular, the shaft seals for the agglomeration unit operations require significant attention and should be readily accessible.

In addition, design engineers should be mindful of the process control scheme developed for the selective agglomeration process. Because many different parameters affect the performance of the process, careful control of these parameters is necessary for consistent product yield and quality. In particular, the production of a consistent ground feedstock (both size and solids concentration) is considered critical. Beyond the feedstock control, proper metering of heptane and asphalt is required to maintain consistent reagent to coal ratios. In addition, good dilution water flow controls are important. As a result, instrumentation and control equipment are vital and highly recommended.

Future R&D Work

Each year, hundreds of thousands of recoverable tons of fine coal are lost to refuse disposal. This may be the result of poor performance in an existing preparation plant or even the lack of an economical fine coal cleaning process itself. It is recommended that the selective agglomeration process be investigated further for the recovery of these coal fines, rather than for the processing of an entire plant feedstock, as was done during the course of this project. This scenario would benefit the economics of the agglomeration process, particularly given the ability of the process to achieve very high energy recoveries under almost all possible operating conditions.

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APPENDIX A

HIAWATHA COAL AGGLOMERATION RESULTS

Hiawatha Coal - PDU 2 t/hr Agglomeration Test Conditions and Results

						High Shear Agglomeration							Low Shear Agglomeration					Low Shear Rinse Basis					Vibrating Screen					Steam Stripper A				Steam Stripper B				Plant Product			Plant Tails		Performan.	
	Agglomeration Feed				Hept	Imp Tip		Res	kwhr/		Invers'n			Imp	Res	kwhr/		Agg				Btu		Bed	Spry	Steam		Res	Temp		Hept	Res	Temp		Hept		Ash			Hept		Btu
	Grind	Sol	Coal	Ash	maf	m/s		Time	ton	1000	Quality		Sol	Tip	Time	ton	1000	Size	% Ash		Yield	Rec	Deck	Dpth	gal/t	lb/t	lb/	Time	Slur	Vap	ppm	Time	Slur	Vap	dcb	Sol		lb/	Ash	ppm	Yield	Rec
Run	D80	%	lb/hr	%	%	A	B	sec	coal	g slur	A	B	%	m/s	sec	coal	g slur	mm	Prod	Tails	%	(%)	Incl	(in)	Prd	coal	gal	min	F	F	dcb	min	F	F	ppm	%	%	MBtu	%	dcb	%	(%)
Complete Plant Test																																										
HS-1	60.7	10.1	3100	8.9	24.7	18.0	--	39	--	--	VG	--	7.3	5.2	140	--	--	.5-3	3.08	--	--	--	U	--	96	2181	2.6	19.4	205	159	2976	25.8	227	228	1395	22.4	3.29	2.31	83.6	2763	93.0	100.3
Minimum Impeller Tip Speed Determination																																										
H-1-A	61.3	10.1	3337	8.4	25.5	14.0	14.0	99	39.0	16.3	G	VG	7.3	5.2	130	3.4	1.0	1	3.13	83.2	93.4	99.7	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-1-B	61.3	10.4	3445	8.5	24.7	11.0	11.1	99	20.0	8.6	M	VG	7.5	5.2	130	3.2	1.0	.5-1	3.12	91.9	94.0	100.4	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-1-C	61.3	10.3	3404	8.4	24.7	7.0	7.0	99	5.7	2.4	N	M	7.5	5.2	131	3.2	1.0	1-3	3.16	81.9	93.4	99.6	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-2-A	66.7	10.4	3449	8.3	25.0	--	14.0	63	28.8	12.3	--	VG	7.6	5.2	131	3.2	1.0	.5	3.09	87.9	93.9	100.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-2-B	66.7	10.5	3481	8.2	23.7	--	11.1	63	15.7	6.8	--	G	7.7	5.2	132	3.1	1.0	.5-1	3.04	86.0	93.7	99.9	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-2-C	66.7	10.5	3463	8.4	24.2	--	9.1	63	9.1	3.9	--	M	7.7	5.2	132	3.2	1.0	1-2	3.09	74.9	92.7	98.9	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-3-A	63.2	10.7	3550	9.9	24.0	18.0	--	36	14.3	6.3	VG	--	7.8	5.2	131	3.1	1.0	.5-2	3.49	90.4	92.6	100.3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-3-B	63.2	10.6	3487	10.0	25.6	14.1	--	36	8.0	3.5	G	--	7.6	5.2	129	3.1	1.0	1-3	3.33	82.2	91.6	99.5	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-3-C	63.2	10.5	3458	9.6	24.6	10.6	--	36	3.7	1.6	M	--	7.5	5.2	129	3.2	1.0	1-3	3.25	63.7	89.4	96.8	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Complete Plant Test																																										
H-4	56.1	10.2	4352	8.9	28.4	18.0	--	28	11.4	4.8	VG	--	8.1	5.2	111	2.4	0.8	0-1	3.10	--	--	--	U	5.00	246	1764	1.7	11.5	205	143	4588	13.1	231	233	2040	20.8	3.56	2.52	82.3	694	93.2	99.0
Minimum Impeller Tip Speed Determination																					0.0	0.0																				
H-5-A	54.4	9.8	4305	9.1	28.3	17.4	--	27	11.4	4.6	G	--	8.1	5.2	111	--	--	0.5	3.26	76.8	92.1	99.0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-5-B	40.0	9.5	4151	9.2	27.2	15.0	--	27	8.1	3.2	F	--	7.9	5.2	114	2.5	0.8	0.5	3.44	75.9	92.1	98.9	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Minimum Energy Requirement Evaluation																																										
H-6-A	42.0	10.6	1586	8.5	25.3	11.1	--	79	9.1	4.0	VG	--	7.1	5.2	266	6.5	1.9	0-2	2.83	75.2	92.1	98.8	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-6-B	42.0	10.6	2389	8.5	23.8	11.1	--	53	6.1	2.7	G	--	8.0	5.2	199	4.4	1.4	1-2	2.83	74.9	92.1	98.8	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-6-C	38.9	9.8	2946	9.2	26.7	11.1	--	40	4.9	2.0	F	--	7.4	5.2	150	3.5	1.1	.5-2	2.84	64.9	89.8	97.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-6-D	38.9	9.8	3447	9.2	26.3	11.1	--	34	4.2	1.7	P	--	7.8	5.2	135	3.1	1.0	0-1	2.86	55.2	87.9	95.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-6-E	38.9	9.8	3689	9.2	27.5	11.1	--	32	4.0	1.6	VP	--	7.9	5.2	127	2.9	0.9	0-1	2.91	61.5	89.3	96.5	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Minimum Impeller Tip Speed Determination																																										
H-7-A	40.6	9.2	3434	9.0	28.5	17.9	--	32	14.8	5.6	VG	--	7.4	5.2	128	3.1	1.0	0-2	2.79	70.7	90.9	98.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-7-B	40.6	9.2	3294	9.0	29.5	14.1	--	33	8.4	3.1	G	--	7.3	5.2	132	3.2	1.0	0-2	2.86	63.4	89.8	96.9	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-7-C	40.6	9.2	3247	9.0	29.4	11.2	--	33	4.5	1.7	M	--	7.3	5.2	133	3.2	1.0	0-2	2.97	65.5	90.4	97.3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Low Shear Evaluation																																										
H-8-A	42.0	9.2	3770	9.3	29.3	17.6	--	29	13.2	5.0	VG	--	7.1	5.2	112	2.8	0.8	1-3	2.93	70.5	90.6	98.0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-8-B	39.3	9.1	3759	9.2	30.2	17.6	--	29	13.3	5.0	VG	--	7.0	3.0	111	1.4	0.4	1-3	3.02	71.5	91.0	98.2	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-8-C	40.1	9.5	3851	9.4	29.0	17.7	--	29	13.0	5.1	VG	--	9.5	5.2	146	2.6	1.0	.5-1.5	3.20	77.5	91.6	99.0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-8-D	46.0	9.7	3946	9.4	28.1	17.4	--	29	12.7	5.1	VG	--	9.7	3.0	146	1.3	0.5	1-3	3.49	77.6	92.1	99.0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Low Shear Evaluation																																										
H-8-E	46.3	9.4	3791	9.2	25.8	17.8	--	29	13.1	5.1	VG	--	7.8	5.2	274	8.0	2.6	1-3	3.15	81.9	92.4	99.5	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-8-F	47.1	10.0	3784	9.2	26.0	17.8	--	31	13.5	5.5	VG	--	8.2	3.0	289	2.4	0.8	.5-1.5	3.32	76.1	92.0	98.9	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-8-G	50.8	10.5	3836	9.0	24.7	18.0	--	32	12.8	5.5	VG	--	10.5	5.2	365	7.6	3.3	1-3	3.13	86.0	92.9	99.9	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
H-8-H	50.2	10.3	3735	9.1	25.5	18.0	--	33	13.6	5.8	VG	--	10.3	3.0	370	2.4	1.0	.5-1	3.33	81.0	92.5	99.4	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

Hiawatha Coal - PDU 2 t/hr Agglomeration Test Conditions and Results (Cont'd)

	Agglomeration Feed					High Shear Agglomeration							Low Shear Agglomeration						Low Shear Rinse Basis				Vibrating Screen					Steam Stripper A				Steam Stripper B				Plant Product			Plant Tails		Performan.	
Run	Grind D80	Sol %	Coal lb/hr	Ash %	Hept maf %	Imp Tip m/s A	B	Res Time sec	kwhr/ ton coal	1000 g slur	Invers'n Quality A	B	Sol %	Imp Tip m/s	Res Time sec	kwhr/ ton coal	1000 g slur	Agg Size mm	% Ash Prod	Tails	Yield %	Btu Rec (%)	Deck Incl	Bed Dpth (in)	Spry gal/t Prd	Steam lb/t coal	lb/ gal	Res Time min	Temp Slur F	Vap F	Hept ppm dcb	Res Time min	Temp Slur F	Vap F	Hept dcb ppm	Sol %	Ash %	lb/ MBtu	Ash %	Hept ppm dcb	Yield %	Btu Rec (%)
Uphill Screen Evaluation																																										
H-9-A	60.0	10.3	3853	8.9	26.9	17.9	--	32	13.1	5.5	VG	--	8.2	5.2	126	2.6	0.9	0-2	3.11	81.5	92.6	99.8	U	2.50	252	2322	2.4	13.5	204	157	--	18.6	228	229	1371	22.0	3.38	2.39	80.4	1329	92.8	99.2
H-9-B	56.4	10.4	3707	8.9	26.4	17.9	--	33	13.7	5.8	VG	--	7.8	5.2	125	2.7	0.9	0-3	3.11	89.2	93.2	100.5	U	3.00	531	2584	2.4	12.6	203	157	3288	18.6	228	229	1604	20.7	3.31	2.36	68.6	3464	91.4	96.7
Level Screen Evaluation																																										
H-9-C	52.9	10.1	3592	9.2	27.9	17.9	--	33	14.1	5.8	VG	--	7.7	5.2	127	2.9	0.9	.5-3	3.00	77.9	91.7	99.2	L	2.50	271	2599	2.2	12.3	201	153	2587	20.8	229	230	1905	21.0	3.24	2.28	81.2	2491	92.4	99.6
Screen/Stripper Evaluation																																										
H-10-A	44.2	10.1	3802	9.1	29.7	17.9	--	31	13.3	5.5	VG	--	7.3	5.2	114	2.7	0.8	.5-2.5	2.87	80.7	92.0	99.3	L	.5-1.7	257	2336	2.3	13.0	204	152	3149	9.2	232	230	1544	22.2	3.09	2.17	80.0	580	92.1	99.3
H-10-B	40.9	9.8	3680	9.4	29.9	17.9	--	32	13.8	5.5	VG	--	7.8	5.2	126	2.8	0.9	.5-2	2.94	89.6	92.5	100.2	L	2.5-1.5	531	2081	1.9	12.2	212	158	4434	9.2	246	244	1953	20.0	2.97	2.08	85.5	601	92.2	99.8
H-10-C	42.7	10.7	3999	9.4	28.8	--	13.5	55	23.7	10.4	--	VG	8.4	5.2	125	2.6	0.9	.5-1.5	2.80	83.0	91.8	99.6	U	5.5-2.5	243	2198	2.3	13.1	205	152	5041	21.2	230	231	1194	23.1	3.24	2.28	85.5	2326	92.6	99.8
Uphill Screen and Stripper Evaluation																																										
H-11-A	41.0	9.8	3692	9.2	30.0	--	14.0		27.5	11.1	--	VG	8.0	5.2	129	2.8	0.9	.5-2.5	2.67	78.6	91.4	99.1	U	3-2	265	1587	2.2	18.5	200	150	6916	13.3	222	231	2242	29.7	2.89	2.02	79.8	0	91.9	99.2
H-11-B	44.5	9.7	3829	9.1	29.5	--	14.0	52	26.5	10.5	--	VG	8.5	5.2	131	2.7	0.9	.5-2	2.73	79.0	91.6	99.1	U	5-2	510	1723	2.1	16.0	210	159	5133	12.2	240	238	1861	27.2	3.07	2.15	80.4	0	92.2	99.3
H-11-BR	43.9	9.7	3840	9.0	29.1	--	14.0	52	26.4	10.5	--	VG	8.6	5.2	133	2.7	0.9	.5-1.5	2.71	83.1	92.1	99.6	U	5-2	508	1753	2.2	16.7	210	159	5192	12.6	240	239	1718	27.3	3.03	2.12	81.2	0	92.3	99.4
Level Screen and Stripper Evaluation																																										
H-11-C	51.2	9.7	3813	9.1	29.3	--	14.0	53	27.0	10.8	--	VG	8.0	5.2	124	2.7	0.9	.5-2.5	3.03	85.6	92.6	99.9	L	.5-1.2	253	1648	2.3	18.3	200	152	6077	21.6	230	231	2135	29.2	3.21	2.25	90.0	364	93.2	100.3
H-11-D	52.9	10.3	3859	9.2	28.8	--	14.0	55	26.2	11.1	--	VG	8.3	5.2	128	2.7	0.9	.5-2	2.97	80.1	91.9	99.3	L	1.75-1	510	1720	2.1	15.7	210	161	6295	21.1	236	241	2184	29.1	3.05	2.14	75.5	0	91.5	98.7
H-11-DR	53.1	10.3	3872	9.1	28.5	--	14.0	55	26.3	11.1	--	VG	8.4	5.2	129	2.7	0.9	.5-2	2.96	80.7	92.1	99.4	L	1.75-1	505	1739	2.0	14.9	210	161	4132	19.5	236	241	1057	26.9	3.19	2.24	77.1	0	92.0	99.0
Downhill Screen and Stripper Evaluation																																										
H-11-E	43.6	11.0	4006	8.2	30.3	--	14.0	56	25.4	11.4	--	VG	8.4	5.2	124	2.6	0.9	.5-2.5	2.80	82.1	93.2	98.7	D	.75-.5	241	1455	2.3	19.1	210	160	--	21.4	237	238	2656	30.6	2.95	2.07	82.2	0	93.3	98.5
H-11-F	40.2	10.7	4029	8.5	30.5	--	14.0	55	25.2	11.1	--	VG	8.6	5.2	126	2.6	0.9	.5-2.5	2.73	81.8	92.7	98.9	D	.75-.5	482	1643	2.1	15.5	210	163	--	19.8	240	241	2544	28.4	2.78	1.94	81.8	0	92.8	99.3
Downhill Screen, High Shear, and Low Shear Evaluation																																										
H-12-A	39.6	10.2	3810	8.5	31.7	--	14.0	55	26.7	11.1	--	VG	8.1	5.2	125	2.7	0.9	1-3	2.64	80.9	92.5	100.1	D	.75-.5	511	1476	2.0	16.8	210	155	10241	21.1	238	241	2712	27.3	2.73	1.91	80.3	0	92.5	99.8
H-12-B	40.8	10.2	3827	9.0	31.1	--	14.0	55	26.5	11.0	--	VG	8.1	6.5	126	3.8	1.3	.5-2.5	2.77	82.6	92.2	99.1	D	.5-0.2	505	1501	2.0	16.1	210	158	--	20.1	238	241	3068	27.3	2.89	2.03	90.8	751	93.0	99.3
H-12-C	40.1	10.2	3821	8.7	30.9	--	11.0	55	14.3	6.0	--	F	8.1	5.2	125	2.7	0.9	.5-3.5	2.80	85.3	92.8	99.1	D	.75-.5	511	1329	1.8	16.8	210	155	--	20.5	238	241	3250	29.3	2.74	1.94	78.6	0	92.1	97.3
Froth Skimmer Evaluation																																										
H-13-A	37.6	9.5	3571	8.5	31.9	--	14.0	55	28.7	11.2	--	VG	7.5	5.2	125	2.9	0.9	.5-2.5	2.79	86.6	93.2	99.6	D	0.50	546	1379	1.8	17.4	210	152	11251	21.7	240	240	3446	26.6	2.66	1.88	78.5	--	92.4	97.8
H-13-B	40.7	9.9	3704	8.3	31.3	--	14.0	55	27.6	11.2	--	VG	7.8	5.2	125	2.7	0.9	.5-2.5	2.74	80.4	92.9	98.4	D	0.38	522	1402	1.8	16.4	210	152	--	21.4	239	241	3146	28.2	2.79	1.94	83.2	--	93.2	99.2
High Shear Evaluation																																										
H-14-A	42.8	9.5	3551	8.4	31.1	--	11.1	55	16.1	6.2	--	F	7.4	5.2	124	2.9	0.9	.5-3	2.76	71.8	91.9	96.4	D	0.25	547	1496	1.9	16.8	210	152	--	20.7	239	239	3392	25.3	2.74	1.91	79.6	--	92.7	97.5
Confirmation Testing																																										
H-15-A	41.8	9.9	3714	8.1	33.5	--	11.0	55	14.8	6.0	--	G	7.1	5.2	113	2.8	0.8	--	2.69	70.5	92.0	96.1	D	0.50	524	1105	1.3	15.1	211	148	--	15.9	235	240	2970	25.5	2.73	1.90	75.1	--	92.6	97.0
H-15-B	40.3	9.5	3551	8.4	32.7	--	11.0	55	15.5	6.0	--	G	6.8	5.2	113	2.9	0.8	--	2.70	69.5	91.5	98.1	D	0.50	551	956	1.1	15.3	210	148	--	16.4	240	240	3824	26.1	2.60	1.81	74.5	--	92.0	98.8
H-15-C	41.3	10.0	3769	8.0	32.9	--	11.0	55	14.6	6.0	--	G	7.2	5.2	113	2.8	0.8	--	2.60	75.4	92.6	99.1	D	0.50	510	897	1.1	15.0	210	148	--	16.0	238	240	3519	26.0	2.82	1.97	83.5	--	93.6	100.0
Production Run																																										
H-P-1	42.1	9.7	3620	8.4	31.4	--	11.0	55	15.3	6.1	--	G	7.3	5.2	120	2.9	0.9	.5-2.5	2.69	76.2	92.3	98.7	D	0.50	537	1533	2.0	16.5	210	152	--	17.7	239	240	2633	26.9	2.73	1.90	78.2	2661	92.5	99.5
H-P-2	42.7	10.6	3996	8.2	31.7	--	11.0	55	13.9	6.0	--	G	8.0	5.2	118	2.6	0.9	.5-1.5	2.67	79.9	92.9	98.0	D	0.50	484	1385	1.9	15.6	210	154	8052	17.2	239	240	2835	27.8	2.81	1.96	80.2	1120	93.0	98.2
H-P-3	42.9	10.5	3949	8.1	31.9	--	11.0	55	14.1	6.0	--	G	7.9	5.2	118	2.6	0.9	.5-3	2.63	77.6	92.7	98.0	D	0.50	491	1335	1.9	16.5	210	154	--	17.3	239	240	3213	29.4	2.69	1.86	77.9	1130	92.9	98.7
H-P-4	40.0	10.3	3850	8.1	31.3	--	11.0	55	14.2	5.9	--	G	7.7	5.2	118	2.7	0.9	.75-3	2.57	75.4	92.5	97.3	D	0.50	502	1434	1.9	15.7	209	152	--	17.9	238	240	3011	26.9	2.87	2.01	79.3	1597	93.2	97.6
H-P-5	41.8	10.5	3923	8.0	30.5	--	11.0	55	13.9	5.9	--	G	7.9	5.2	119	2.7	0.9	.5-2	2.71	74.5	92.7	98.2	D	0.50	491	1298	1.9	16.8	210	152	8756	17.0	239	240	2929	26.3	2.91	2.02	81.1	1079	93.5	99.6
H-P-6	41.8	10.4	3893	8.2	31.9	--	11.0	55	14.0	6.0	--	G	7.8	5.2	119	2.7	0.9	1-3	2.68	82.3	93.1	99.1	D	0.50	494	1494	1.8	15.0	209	155	--	16.9	238	240	2841	27.4	2.78	1.94	86.9	1024	93.6	99.4
H-P-10	39.6	9.9	3719	9.1	31.2	--	11.0	55	14.8	6.0	--	G	7.5	5.2	119	2.8	0.9	1-3.5	2.77	59.1	88.8	95.7	D	0.50	528	1295	1.6	15.9	210	152	--	17.7	239	240	3369	26.7	2.74	1.91	78.4	1547	91.6	98.7
H-P-11	42.9	10.2	3836	8.8	30.9	--	11.0	55	14.3	6.0	--	G	7.6	5.2	118	2.7	0.8	.5-3	2.66	81.2	92.2	99.3	D	0.50	508	1300	1.7	15.8	210	154	6091	16.9	239	240	2789	27.0	2.75	1.92	81.5	1406	92.3	99.2
H-P-12	44.7	10.1	3768	8.4	30.8	--	11.0	55	14.6	6.0	--	G	7.5	5.2	118	2.8	0.8	1-3.5	2.59	77.6	92.3	98.4	D	0.50	516	1348	1.7	15.8	210	148	--	16.6	239	240	2934	27.1	2.70	1.88	80.2	1664	92.7	99.0
Production Run Average																																										
	42.1	10.2	3839	8.3	31.3	--	11.0	55	14.3	6.0	--	G	7.7	5.2	119	2.7	0.9	.75-2.5	2.66	76.0	92.3	98.2	D	0.50	505	1380	1.8	15.9	210	153	7633	17.3	239	240	2951	27.3	2.78	1.93	80.4	1470	92.8	98.9

APPENDIX B

TAGGART COAL AGGLOMERATION RESULTS

Taggart Coal - PDU 2 t/hr Agglomeration Test Conditions and Results

	Agglomeration Feed					High Shear Agglomerration							Low Shear Agglomeration					Low Shear Rinse Basis					Vib Screen*				Steam Stripper A				Steam Stripper B				Plant Product			Plant Tails		Perform.	
Run	Grind D80	Sol %	Coal lb/hr	Ash %	Hept maf %	Imp Tip m/s A	Imp Tip m/s B	Res Time sec	kwhr/ Ton Coal	kwhr/ 1000 g slur	Invers'n Quality A	Invers'n Quality B	Sol %	Imp Tip m/s	Res Time sec	kwhr/ Ton Coal	kwhr/ 1000 g slur	Agg Size mm	% Ash Prod	% Ash Tails	Yield %	Btu Rec %	Bed Dpth in	Spry gal/t Prd	Steam lb/t coal	Steam lb/ gal	Res Time min	Temp Slur F	Temp Vap F	Hept ppm dcb	Res Time min	Temp Slur F	Temp Vap F	Hept ppm dcb	Sol %	Ash %	Ash lb/ MBtu	Ash %	Hept dcb ppm	Yield %	Btu Rec (%)
High Shear Impeller B Tip Speed Evaluation																																									
T-1-1	35.5	10.2	3816	3.8	31.7	--	14.0	55	26.7	11.1	--	VG	8.1	5.2	126	2.9	1.0	.5-1.5	1.59	76.5	97.1	99.3	--	--	--	2.2	17.8	210	148	--	13.8	240	236	--	--	--	--	--	--	--	--
T-1-2	35.5	10.2	3814	3.8	33.6	--	11.5	55	16.2	6.7	--	F	7.9	5.2	123	2.8	0.9	.5-1.5	1.58	72.3	96.9	99.1	--	--	--	2.2	17.3	210	149	--	15.3	240	235	--	--	--	--	--	--	--	--
T-1-3	35.5	10.2	3816	3.8	36.0	--	9.0	54	8.3	3.4	--	P	7.7	5.2	119	2.8	0.9	.5-1.5	1.60	68.6	96.7	99.0	--	--	--	2.1	15.8	209	151	--	16.7	240	230	--	--	--	--	--	--	--	--
High Shear A Impeller Tip Speed Evaluation																																									
T-2-1	38.4	10.6	4038	3.6	33.4	17.4	--	31	12.0	5.2	VG	--	8.2	5.2	120	2.6	0.9	.5-1.5	1.73	81.3	97.7	99.6	--	--	--	2.1	15.5	210	154	--	16.4	240	235	--	--	--	--	--	--	--	--
T-2-2	38.4	10.6	3996	3.6	35.7	15.1	--	31	8.6	3.7	G	--	8.1	5.2	119	2.6	0.9	.5-1.5	1.69	78.5	97.5	99.5	--	--	--	2.1	15.0	210	154	--	17.1	240	235	--	--	--	--	--	--	--	--
T-2-3	37.1	10.8	4051	3.5	33.3	11.0	--	31	3.6	1.6	P	--	8.0	5.2	116	2.6	0.9	.5-3.5	1.60	63.1	96.8	98.8	--	--	--	1.7	14.3	210	151	--	17.9	240	229	--	--	--	--	--	--	--	--
High Shear A Full Plant Test																																									
T-3	38.4	10.7	4024	3.5	35.5	15.1	--	31	8.6	3.7	G	--	8.2	5.2	119	2.6	0.9	.5-2.5	1.72	79.2	97.7	102.1	1.00	457	1621	2.1	15.5	210	154	7679	16.9	240	233	4060	29.0	1.79	1.25	79.6	1243	97.8	97.8
High Shear A Full Plant Tests																																									
T-4-1	38.2	10.7	4033	3.6	35.8	15.1	--	31	8.6	3.7	G	--	8.0	5.2	116	2.6	0.8	.5-3	1.64	76.2	97.4	99.4	--	--	--	1.9	15.0	210	151	--	17.4	240	224	--	--	--	--	--	--	--	--
T-4-2	38.3	10.7	4007	3.5	36.2	15.1	--	31	8.7	3.7	G	--	7.9	5.2	116	2.6	0.8	.5-3	1.69	77.9	97.7	99.4	0.75	460	1439	1.8	14.9	210	151	9300	17.6	240	216	4268	29.8	1.70	1.13	76.5	1512	97.7	99.3
Reduced Feed Rate - PSD Evaluation																																									
T-5	36.8	11.1	3670	3.6	36.6	11.0	--	35	3.9	1.7	P	--	8.2	5.2	131	2.9	1.0	1-4	1.65	71.4	97.2	99.5	--	445	1507	2.0	17.3	211	155	9313	17.7	240	212	4108	29.8	1.67	1.11	70.4	1438	97.2	99.4
High Shear A Tip Speed/Energy Evaluation																																									
T-6	35.6	11.2	3697	3.5	37.8	11.0	--	35	3.9	1.7	G	--	8.3	5.2	131	2.8	1.0	.5-3.5	1.63	68.6	97.2	99.1	1.25	439	1421	1.8	16.5	210	161	--	18.8	240	219	--	29.7	1.61	1.07	70.5	--	97.3	99.2
T-6-1	34.4	10.8	3560	3.6	39.2	13.0	--	35	6.5	2.8	VG	--	8.0	5.2	132	2.9	0.9	--	1.70	73.1	97.4	99.3	--	--	--	1.7	15.1	210	161	--	18.4	240	216	--	--	--	--	--	--	--	--
Low Shear % Solids, Low Shear Tip speed/Energy, and Stripper % Solids Evaluation																																									
T7	35.6	11.3	3754	3.5	38.0	11.0	--	35	3.8	1.8	G	--	8.4	5.2	131	2.8	1.0	.5-3.5	1.63	67.6	97.2	99.1	1.00	438	1473	2.0	17.5	210	157	11184	18.7	241	224	4983	29.6	1.63	1.08	69.7	3148	97.2	99.2
T8	33.2	11.0	3621	3.6	38.7	11.0	--	35	3.9	1.7	G	--	7.1	5.2	116	2.9	0.8	.5-3.5	1.63	64.6	96.8	98.9	2.25	446	1585	2.1	17.3	210	160	9953	17.6	241	223	4716	28.7	1.64	1.09	67.2	3683	97.0	99.0
T9	35.2	11.6	3830	3.5	38.5	11.0	--	35	3.7	1.7	G	--	8.6	6.6	131	3.8	1.3	.5-3	1.72	72.2	97.5	99.3	0.75	428	1927	1.8	11.1	208	157	9762	12.2	241	231	5082	20.7	1.68	1.12	75.3	2773	97.5	99.4
High Shear A Energy Evaluation																																									
T-10-1	32.0	10.2	2553	3.5	36.8	11.0	--	47	5.8	2.4	F	--	7.5	5.2	173	4.0	1.2	.5-3.5	1.60	69.0	97.1	99.1	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
T-10-2	32.1	10.1	3003	3.6	36.6	11.0	--	39	4.8	2.0	P	--	7.4	5.2	146	3.4	1.0	.5-5	1.55	59.9	96.5	98.6	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
T-10-3	32.2	10.1	3517	3.6	37.1	11.0	--	33	4.1	1.7	VP	--	7.5	5.2	125	3.0	0.9	.5-3.5	1.47	50.7	95.6	97.8	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
T-10-4	30.7	10.0	3765	3.6	37.5	11.0	--	31	3.8	1.6	N	--	7.4	5.2	116	2.8	0.8	.5-3.5	1.53	45.0	95.2	97.3	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
T-11	30.9	10.0	3312	3.7	35.1	11.0	--	35	4.5	1.8	?	--	7.4	5.2	132	3.1	0.9	.5-4	1.59	51.6	95.7	97.9	1.00	658	1659	2.0	17.6	210	160	6760	18.7	242	226	4775	26.6	1.53	1.02	58.6	5121	96.1	98.4
High SHear B Energy Evaluation																																									
T-12	30.2	10.1	3345	3.9	36.6	--	11.0	62	16.3	6.7	--	G	7.3	5.2	129	3.2	0.9	.5-2.5	1.53	64.8	96.2	98.6	0.75	501	1608	2.2	19.5	210	156	10374	20.7	241	224	3888	28.6	1.54	1.02	63.5	4740	96.2	98.6
T-13	29.1	10.4	3436	3.7	39.4	--	9.5	62	10.8	4.5	--	P	7.9	5.2	134	3.0	1.0	.5-2.5	1.54	55.7	96.0	98.2	1.00	489	1623	1.9	16.5	210	159	11179	19.3	241	225	5278	27.5	1.45	0.96	55.6	5583	95.9	98.1
T-14	31.4	11.1	3659	3.4	40.1	--	8.5	61	7.3	3.3	--	VP	8.2	5.2	131	2.9	1.0	.5-3	1.64	61.6	97.1	98.9	1.00	453	1591	2.2	18.0	210	159	--	18.9	241	227	--	28.5	1.77	1.18	62.0	--	97.3	99.0
Production Run																																									
T-P-1	33.2	10.3	3416	3.6	38.2	--	10.0	62	12.5	5.2	--	G	7.7	5.2	132	3.1	1.0	.75-2.5	1.59	63.7	96.7	98.9	0.75	547	1551	1.9	17.7	210	154	--	19.4	242	226	4623	28.8	1.60	1.06	62.4	5401	96.7	99.1
T-P-3	29.3	10.2	3372	3.8	39.5	--	10.0	62	12.6	5.2	--	G	7.6	5.2	132	3.1	1.0	1-3	1.67	67.5	96.8	100.5	0.75	540	1556	1.9	17.6	210	154	--	18.9	242	222	5579	27.8	1.61	1.08	64.8	4562	96.6	99.5
T-P-4	29.5	10.1	3339	3.8	36.8	--	10.0	62	12.7	5.2	--	G	7.5	5.2	132	3.2	1.0	.75-3	1.61	66.3	96.7	100.2	0.75	563	1608	1.9	17.3	210	154	11633	18.2	242	227	4532	27.0	1.58	1.05	65.5	3569	96.6	100.2
T-P-5	30.7	10.2	3361	3.7	38.3	--	10.0	62	12.7	5.2	--	G	7.6	5.2	132	3.1	1.0	.5-2.5	1.54	63.8	96.5	99.4	0.75	563	1556	1.8	17.0	210	157	--	18.0	242	226	5489	26.8	1.58	1.05	60.0	4126	96.3	98.9
T-P-6	29.6	10.0	3300	3.4	39.0	--	10.0	62	12.9	5.2	--	G	7.4	5.2	132	3.2	1.0	1-3	1.52	67.5	97.1	99.7	0.75	569	1498	1.8	17.4	210	157	--	18.8	242	220	5424	26.8	1.55	1.03	65.3	3858	97.1	99.3
T-P-7	30.3	9.6	3179	3.4	40.3	--	10.0	62	13.4	5.2	--	G	7.1	5.2	132	3.3	1.0	1-3	1.57	65.1	97.1	99.7	0.75	592	1567	1.8	17.2	210	152	12314	19.0	242	219	5065	26.0	1.52	1.01	64.4	3537	96.9	99.5
T-P-8	29.6	9.7	3199	3.5	40.1	--	10.0	62	13.3	5.2	--	G	7.2	5.2	132	3.3	1.0	.5-3	1.56	67.6	97.0	99.3	0.75	589	1579	1.8	17.2	210	154	--	18.3	242	224	5431	25.6	1.53	1.01	61.2	3379	96.7	99.2
T-P-9	29.0	10.1	3321	3.6	39.8	--	10.0	62	12.8	5.2	--	G	7.5	5.2	132	3.2	1.0	1-3	1.59	69.4	97.0	99.7	0.75	567	1512	1.8	16.9	210	157	--	18.1	242	222	5158	26.4	1.62	1.08	64.5	3728	96.8	99.0
T-P-10	29.5	10.1	3340	3.9	40.5	--	10.0	62	12.7	5.2	--	G	7.5	5.2	131	3.1	1.0	1-3	1.55	65.6	96.4	99.3	0.75	554	1558	1.9	17.1	210	155	12274	18.0	242	220	4875	26.9	1.69	1.12	62.5	4670	96.4	99.2
T-P-11	32.1	9.8	3226	3.7	39.5	--	10.0	62	13.2	5.2	--	G	7.3	5.2	132	3.3	1.0	1-3	1.67	65.4	96.9	99.1	0.75	577	1550	1.8	17.1	210	154	--	18.4	242	222	4976	26.2	1.61	1.08	59.3	4112	96.5	98.3
Production Run Average																																									
	30.3	10.0	3305	3.6	39.2	--	10.0	62	12.9	5.2	--	G	7.4	5.2	132	3.2	1.0	1-3	1.58	66.2	96.8	99.6	0.75	566	1553	1.8	17.2	210	155	12074	18.5	242	223	5115	26.8	1.59	1.06	63.0	4094	96.7	99.2

* Downhill Screen Orientation

APPENDIX C

INDIANA VII COAL AGGLOMERATION RESULTS

Indiana VII Coal - PDU 2 t/hr Agglomeration Test Conditions and Results

	Agglomeration Feed						High Shear Agglomeration							Low Shear Agglomeration						Low Shear Rinse Basis				Vib Screen*				Steam Stripper A				Steam Stripper B				Plant Product			Plant Tails		Perform.	
Run	Grind D80	Sol %	Coal lb/hr	Ash %	Hept maf %	Asph lb/t	Imp Tip m/s A	Imp Tip m/s B	Res Time sec	kwhr/ Ton Coal	kwhr/ 1000 g slur	Invers'n Quality A	Invers'n Quality B	Sol %	Imp Tip m/s	Res Time sec	kwhr/ Ton Coal	kwhr/ 1000 g slur	Agg Size mm	% Ash Prod	% Ash Tails	Yield %	Btu Rec %	Bed Dpth in	Spry gal/t Prd	Steam lb/t coal	Steam lb/ gal	Res Time min	Temp Slur F	Temp Vap F	Hept ppm dcb	Res Time min	Temp Slur F	Temp Vap F	Hept dcb ppm	Sol %	Ash %	Ash lb/ MBtu	Ash %	Hept dcb ppm	Yield %	Btu Rec %
Indiana VII Coal Start-Up Test																																										
I-1	23.5	7.0	2760	9.8	43.4	9.5	17.4	14.0	81	--	--	0	2	5.5	5.2	118	--	--	--	3.04	86.1	91.9	100.2	--	632	1931	1.7	16.9	211	156	--	15.5	240	227	--	20.7	3.08	2.20	73.0	--	90.4	97.9
High Shear Energy and Asphalt Dosage Evaluation																																										
I-2	23.9	8.8	2551	10.0	42.4	8.5	14.0	14.0	110	49.0	17.4	0	4	6.4	5.2	147	4.1	1.1	2.00	3.14	85.2	91.6	99.9	0.50	588	1684	1.8	21.4	210	155	13767	22.3	241	205	4633	24.1	3.10	2.20	90.4	339	92.1	100.7
I-3-1	23.3	8.8	2564	10.0	41.7	11.3	14.0	14.0	110	48.9	17.4	0	6	6.3	5.2	146	4.0	1.0	1.50	3.15	88.8	92.0	100.4	--	--	--	--	--	--	155	--	--	--	215	--	--	--	--	--	--	--	--
I-3-2	23.3	8.8	2556	10.0	43.2	11.3	11.0	11.0	110	26.6	9.4	0	2	6.3	5.2	145	4.2	1.1	2.00	3.23	83.8	91.6	99.9	--	--	--	--	--	--	155	--	--	--	215	--	--	--	--	--	--	--	--
High Shear Energy and Asphalt Dosage Evaluation																																										
I-4	26.1	8.5	2455	10.2	41.3	11.8	17.4	14.0	111	59.7	20.4	0	9	6.1	5.2	147	4.2	1.1	1.75	3.19	90.1	91.9	100.7	0.50	606	1795	1.8	20.5	210	160	12328	20.8	241	219	4355	25.1	3.21	2.29	89.5	686	91.9	100.5
I-5	26.4	8.8	2547	9.7	39.3	18.9	--	14.0	70	38.0	13.4	--	5	6.4	5.2	148	4.1	1.1	2.00	3.30	88.2	92.5	100.2	--	576	1696	1.9	25.0	209	159	13155	34.6	241	216	7307	23.3	3.58	2.56	89.1	369	92.9	100.2
High Shear Energy and Asphalt Dosage Evaluation																																										
I-6	26.0	11.3	2360	10.1	40.3	8.5	--	14.0	97	38.8	17.6	--	2	7.5	5.2	187	4.4	1.3	2.00	2.96	84.1	91.2	100.4	0.50	547	1633	1.8	24.1	210	157	13445	25.0	241	220	4647	24.1	3.11	2.23	86.2	1006	91.6	100.1
I-7-1	26.2	11.8	2447	10.2	38.0	13.3	--	14.0	96	37.4	17.5	--	5	7.8	5.2	187	4.2	1.4	1.75	3.07	92.2	92.0	100.8	--	--	--	--	--	--	158	--	--	--	218	--	--	--	--	--	--	--	--
I-7-2	26.2	11.8	2450	10.2	38.9	13.3	--	12.0	96	27.3	12.7	--	3	7.8	5.2	187	4.2	1.3	1.75	3.23	90.4	92.0	100.6	--	--	--	--	--	--	160	--	--	--	217	--	--	--	--	--	--	--	--
Low Shear Solids Concentration and Tip Speed Evaluation																																										
I-8-1	23.7	12.4	2584	10.2	39.2	9.7	14.0	14.0	151	46.4	22.8	0	8	7.7	5.2	176	4.0	1.3	1.50	3.11	90.7	91.9	100.6	0.50	504	1606	1.8	22.2	209	155	--	24.1	240	216	--	26.6	3.07	2.18	90.1	--	91.8	100.6
I-8-2	25.1	12.7	2661	10.0	37.1	9.4	14.0	14.0	150	45.1	22.8	0	8	12.7	5.2	280	3.7	1.9	1.50	3.16	93.1	92.4	100.6	0.75	492	1503	1.8	23.0	209	155	--	24.9	240	214	--	28.5	3.02	2.15	90.5	--	92.1	100.4
I-8-3	27.5	12.9	2702	10.1	37.4	9.3	14.0	14.0	151	44.4	22.8	0	8	8.2	6.6	179	5.2	1.7	1.50	3.22	94.4	92.5	101.0	--	--	--	--	--	--	154	--	--	--	223	--	--	--	--	--	--	--	--
Stripper Temperature, Screen Spray, and High Shear Evaluation																																										
I-9-1	23.0	13.4	2815	10.2	40.2	7.1	14.0	14.0	149	43.1	22.8	0	6	8.0	5.2	167	3.6	1.2	2.00	2.81	90.5	91.6	100.8	0.75	468	1556	1.8	20.5	199	154	13487	15.9	231	209	5152	27.3	2.84	2.01	90.0	310	91.6	100.7
I-9-2	21.1	12.4	2595	10.3	40.9	7.7	14.0	14.0	150	46.7	23.0	0	6	7.7	5.2	175	4.0	1.3	1.50	2.76	91.3	91.5	100.4	0.75	841	1811	1.9	20.8	210	160	11771	17.5	240	222	4917	23.6	2.71	1.91	89.8	279	91.3	100.6
I-9-3	20.0	12.4	2590	10.3	42.3	4.8	14.0	14.0	151	46.8	23.1	0	2	7.7	5.2	174	4.0	1.2	2.00	2.65	86.2	90.9	100.2	--	--	--	--	--	--	160	--	--	--	220	--	--	--	--	--	--	--	--
I-9-4	20.1	12.6	2634	10.1	40.1	9.5	12.1	12.0	150	34.2	17.0	0	1	7.7	5.2	172	3.9	1.2	1.00	2.81	89.1	91.5	100.5	--	--	--	--	--	--	160	--	--	--	223	--	--	--	--	--	--	--	--
Low Shear and Asphalt Effect Evaluation																																										
I-10-1	24.8	14.3	2990	10.1	37.8	6.7	14.0	14.0	151	34.1	19.4	0	5	8.5	3.0	168	1.6	0.6	0-6	2.91	81.5	90.9	99.6	--	--	--	--	--	--	--	--	--	--	236	--	--	--	--	--	--	--	--
I-10-2	22.5	12.3	2567	10.1	41.9	6.8	14.0	14.0	150	39.7	19.3	0	5	7.6	5.2	174	4.0	1.2	1.75	2.93	89.1	91.7	100.5	--	513	1800	2.1	23.0	211	158	0	19.2	242	221	0	25.3	2.93	2.08	90.3	0	91.8	100.7
I-10-3	22.8	12.1	2528	10.1	43.9	4.9	14.0	14.0	151	40.3	19.4	0	3	7.5	5.2	174	4.1	1.2	2.00	2.95	85.2	91.3	100.0	--	--	--	--	--	--	--	--	--	--	221	--	--	--	--	--	--	--	--
I-10-4	23.7	12.1	2530	10.1	43.9	4.9	14.0	14.0	151	40.2	19.4	0	3	7.5	5.2	174	4.1	1.2	0.50	2.98	88.2	91.7	100.4	--	--	--	--	--	--	--	--	--	--	221	--	--	--	--	--	--	--	--

* Downhill Screen Orientation

Indiana VII Coal - PDU 2 t/hr Agglomeration Test Conditions and Results (Cont’d)

							High Shear Agglomeration							Low Shear Agglomeration					Low Shear Rinse Basis					Vib Screen*				Steam Stripper A				Steam Stripper B				Plant Product			Plant Tails		Perform.	
	Agglomeration Feed				Hept		Imp Tip		Res	kwhr/		Invers'n			Imp	Res	kwhr/		Agg				Btu	Bed	Spry	Steam		Res	Temp		Hept	Res	Temp		Hept		Ash			Hept		Btu
	Grind	Sol	Coal	Ash	maf	Asph	m/s		Time	Ton	1000	Quality		Sol	Tip	Time	Ton	1000	Size	% Ash		Yield	Rec	Dpth	gal/t	lb/t	lb/	Time	Slur	Vap	ppm	Time	Slur	Vap	dcb	Sol		lb/	Ash	dcb	Yield	Rec
Run	D80	%	lb/hr	%	%	lb/t	A	B	sec	Coal	g slur	A	B	%	m/s	sec	Coal	g slur	mm	Prod	Tails	%	%	in	Prd	coal	gal	min	F	F	dcb	min	F	F	ppm	%	%	MBtu	%	ppm	%	%
PSD Evaluation																																										
I-11-1	24.2	13.8	2658	9.9	40.5	6.6	14.0	14.0	163	38.3	20.9	0	6	7.9	5.2	175	3.9	1.2	2.00	2.98	91.6	92.2	100.7	0.75	476	1676	2.1	23.7	210	160	--	20.3	241	214	--	26.4	2.98	2.12	92.7	--	92.3	100.8
I-11-2	27.3	13.8	3195	9.9	39.8	6.6	14.0	14.0	135	31.9	17.4	0	4	8.5	5.2	156	3.2	1.1	2.00	3.20	90.7	92.3	100.6	1.25	495	1647	2.1	19.3	210	160	11933	14.2	241	214	4964	27.4	3.14	2.23	90.9	471	92.3	101.0
PSD Evaluation																																										
I-12-1	35.5	13.6	3540	9.4	37.2	9.7	14.0	14.0	121	34.8	18.8	0	9	8.4	5.2	141	2.9	1.0	2.00	3.14	91.4	92.9	100.6	1.00	485	1528	1.8	16.8	210	162	10463	14.6	241	215	4275	28.3	3.12	2.22	91.0	385	92.8	100.6
I-12-2	37.0	13.6	3531	9.5	39.1	4.8	14.0	14.0	122	34.9	18.9	0	2	8.5	5.2	142	3.0	1.0	2.00	2.96	83.0	91.8	99.8	--	--	--	--	--	--	--	--	--	--	215	--	--	--	--	--	--	--	--
PSD Evaluation																																										
I-13-1	36.9	13.4	3923	9.3	36.7	9.8	14.0	14.0	108	32.0	17.1	0	8	8.7	5.2	132	2.6	0.9	2.25	3.25	91.2	93.2	100.6	1.25	499	1533	2.1	17.6	212	163	10166	11.5	240	220	4153	28.2	3.17	2.26	91.4	586	93.1	100.6
I-13-2	37.2	13.2	3881	9.3	37.1	5.6	14.0	14.0	109	32.4	17.3	0	2	8.7	5.2	132	2.7	0.9	3.00	3.12	86.6	92.6	100.2	--	--	--	--	--	--	--	--	--	--	220	--	--	--	--	--	--	--	--
PSD Evaluation																																										
I-14	35.0	12.9	4340	9.5	37.3	9.9	14.0	14.0	94	29.0	14.9	0	3	8.8	5.2	120	2.4	0.9	2.00	3.42	90.5	93.0	100.5	--	--	--	--	--	--	--	--	--	--	220	--	--	--	--	--	--	--	--
PSD Evaluation																																										
I-15	93.5	13.5	4223	9.4	33.0	9.5	14.0	14.0	101	30.4	16.4	5	10	9.4	5.2	131	2.5	1.0	1.75	4.45	91.4	94.3	100.5	1.25	482	1500	1.9	15.0	207	161	6346	14.7	236	225	2886	28.4	4.44	3.21	89.6	1462	94.2	100.4
I-16	80.8	13.4	4301	9.7	34.2	4.7	14.0	14.0	100	29.8	16.2	0	7	9.4	5.2	129	2.4	0.9	1.50	4.64	92.0	94.2	100.5	--	--	--	--	--	--	--	--	--	--	226	--	--	--	--	--	--	--	--
Confirmation Testing																																										
I-17-1	72.0	14.2	4183	10.0	35.3	4.6	14.0	14.0	108	30.7	17.5	0	8	9.7	5.2	138	2.5	1.0	1.50	4.28	90.0	93.4	100.4	--	468	1750	2.3	15.4	207	160	--	12.2	236	232	--	27.7	4.30	3.11	89.9	--	93.4	100.4
I-17-2	67.3	14.2	4166	10.0	35.8	4.6	14.0	14.0	108	30.8	17.5	0	8	9.7	5.2	138	2.5	1.0	1.50	4.37	89.5	93.4	100.4	--	470	1573	1.8	12.8	207	161	--	13.5	237	232	--	28.4	4.35	3.14	89.0	--	93.4	100.4
Production Run																																										
I-P-1	64.7	12.4	3638	9.6	35.5	5.4	14.0	14.0	109	34.1	17.1	0	8	8.6	5.2	140	2.8	1.0	1.50	4.14	90.5	93.7	100.5	1.00	538	1752	2.0	15.9	208	154	--	14.1	237	230	3848	26.3	4.17	3.01	91.7	844	93.8	100.6
I-P-2	67.0	12.4	3630	9.9	36.1	5.4	14.0	14.0	109	34.2	17.1	0	8	8.6	5.2	140	2.8	1.0	2.00	4.23	89.8	93.3	99.6	1.00	540	1704	2.1	16.9	208	156	6927	13.0	237	219	4235	27.6	4.19	3.02	91.9	764	93.5	99.8
I-P-3	59.1	13.3	3833	9.8	35.3	5.1	14.0	14.0	110	30.6	16.3	0	8	9.2	5.2	141	2.7	1.0	2.00	4.20	89.8	93.5	99.8	1.00	505	1749	2.0	15.0	208	156	--	12.3	237	227	4024	26.3	4.23	3.06	87.4	865	93.3	99.4
I-P-4	73.1	12.3	3476	10.0	36.2	5.6	14.0	14.0	112	33.7	16.6	0	8	8.4	5.2	143	3.0	1.0	2.00	4.30	89.5	93.3	99.6	1.00	548	1839	2.1	16.5	208	157	6569	14.1	237	229	4196	25.9	4.27	3.07	91.4	292	93.4	100.2
I-P-6	62.5	12.1	3494	9.9	35.4	5.6	14.0	14.0	110	33.5	16.3	0	8	8.4	5.2	142	3.0	1.0	1.00	4.24	90.0	93.4	99.9	1.00	561	1805	2.1	16.7	208	159	6877	13.1	234	226	3567	26.2	4.18	3.01	90.7	361	93.4	100.2
I-P-7	60.8	12.3	3217	9.9	30.6	5.7	14.0	14.0	123	35.5	17.7	0	8	8.2	5.2	151	3.2	1.1	2.50	4.14	90.3	93.4	99.4	1.00	588	1851	2.1	17.0	209	160	--	13.8	235	227	4161	26.0	4.16	2.99	91.2	307	93.5	100.0
I-P-8	63.9	12.5	3336	10.0	33.3	5.5	14.0	14.0	120	34.3	17.3	0	8	8.3	5.2	148	3.1	1.1	1.00	4.29	90.1	93.3	99.9	1.00	568	1735	2.1	18.3	208	160	6552	15.3	237	230	3823	26.1	4.21	3.04	91.4	348	93.3	100.0
I-P-9	62.1	12.9	3588	9.9	34.4	5.1	14.0	14.0	115	31.8	16.6	0	8	8.9	5.2	147	2.9	1.1	2.50	4.34	90.3	93.5	99.9	1.00	526	1697	2.1	17.3	208	161	--	15.0	237	230	3876	26.6	4.17	3.02	91.1	277	93.4	99.6
I-P-10	61.5	12.3	3387	9.7	35.7	5.4	14.0	14.0	116	33.7	16.7	0	8	8.6	5.2	150	3.1	1.1	1.50	4.16	90.6	93.6	100.0	1.00	553	1792	2.1	17.1	208	162	6761	13.3	237	228	4080	26.6	4.14	2.99	91.0	317	93.6	100.0
I-P-11	64.0	12.2	3311	9.7	35.0	5.5	14.0	14.0	118	34.5	17.0	0	8	8.5	5.2	152	3.1	1.1	1.00	4.25	90.3	93.6	99.6	1.00	562	1857	2.1	16.8	208	161	--	12.8	237	229	3861	25.4	4.14	2.98	92.2	344	93.6	99.8
Production Run Average																																										
	63.9	12.5	3491	9.8	34.8	5.4	14.0	14.0	114	33.6	16.9	0	8	8.6	5.2	145	3.0	1.0	1.70	4.23	90.1	93.5	99.8	1.00	549	1778	2.1	16.7	208	159	6737	13.7	236	227	3967	26.3	4.19	3.02	91.0	472	93.5	99.9

* Downhill Screen Orientation