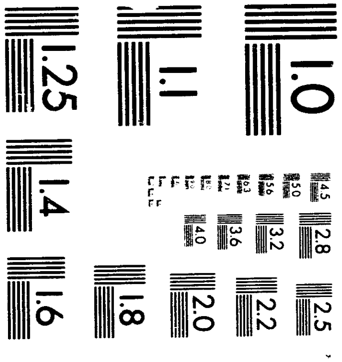
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Advanced Physical Fine Coal Cleaning
Spherical Agglomeration
U.S. DOE/PETC

Final Report

Contract No. DE-AC22-87PC79867
Bechtel Job. No. 19307

September 1990

Bechtel National, Inc.

MASTER

19307/1/c/Spherical/238
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Executive Summary

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ABSTRACT

The project included process development, engineering, construction, and operation of a 1/3 tph proof-of-concept (POC) spherical agglomeration test module. The POC tests demonstrated that physical cleaning of ultrafine coal by agglomeration using heptane can achieve:

- Pyritic sulfur reductions beyond that possible with conventional coal cleaning methods
- Coal ash contents below those which can be obtained by conventional coal cleaning methods at comparable energy recoveries
- Energy recoveries of 80 percent or greater measured against the raw coal energy content
- Complete recovery of the heptane bridging liquid from the agglomerates
- Production of agglomerates with 3/8-inch size and less than 30 percent moisture

Bechtel National, Inc. (BNI) installed the POC test module at Electric Power Research Institute's CQ Inc., near Homer City, Pennsylvania. Arcanum Corporation of Ann Arbor, Michigan, provided the agglomeration process development and bench-scale testing. The project began in September 1987 and the POC module was operated between November 1989 and March 1990 using coals from the Illinois No. 6, Upper Freeport, and Pittsburgh seams. Data evaluation and preparation of the final report was completed in September 1990. Test results met or exceeded all of the program objectives.

Nominal 3/8-inch size agglomerates with less than 20 percent moisture were produced. The clean coal ash content varied between 1.5 to 5.5 percent by weight (dry basis) depending on feed coal type. Ash reductions of the run-of-mine (ROM) coal were 77 to 83 percent. ROM pyritic sulfur reductions varied from 86 to 90 percent for the three test coals, equating to total sulfur reductions of 47 to 72 percent.

50:50 blend of Upper and Lower Freeport seam coals with an ash content of 12.1 percent and a sulfur dioxide emission potential of 2.8 lb/MMBtu was cleaned to 4.4 percent ash content and emission potential of 1.0 lb/MMBtu sulfur dioxide. The processing cost was estimated as $15.90 per ton (excluding cost of coal).

EXECUTIVE SUMMARY

This report describes proof-of-concept (POC) testing of ultrafine coal agglomeration with bridging liquid recovery performed by Bechtel National, Inc. (BNI) under U.S. Department of Energy (DOE) contract DE-AC22-87PC79867, Advanced Physical Fine Coal Cleaning Spherical Agglomeration. Work started in September 1987, and the operational testing was completed in March 1990. Data reduction and the final report were complete in September 1990. The program was sponsored by the DOE's Pittsburgh Energy Technology Center (PETC) and the Electric Power Research Institute (EPRI) under a joint agreement to promote research in promising coal cleaning technologies.

PROCESS DESCRIPTION

The POC agglomeration process is based on Spherical Agglomeration technology developed by Arcanum Corp., licensee of the process originators, the National Research Council of Canada. The process uses heptane to selectively agglomerate lyophilic organic coal materials from an aqueous slurry of ultrafine coal. A petroleum-based asphalt binder is added to assist in enlarging product agglomerates to handleable 1/4-inch to 3/8-inch spherical pellets. Hydrophilic inorganic ash-forming and pyritic sulfur mineral matter is rejected, leaving a virtually coal-free refuse. The agglomerated coal product is then stripped of the heptane bridging liquid by contact with steam.

Steam stripping is used to recover the heptane because heptane and steam form an azeotrope which has a boiling point (79.2°C) lower than either heptane or water alone (98°C and 100°C respectively). As long as the agglomerated coal contains one-fifth as much water as heptane, essentially all the heptane vaporizes before the mixture's temperature reaches the boiling point of water.

THE TEAM

Bechtel and Arcanum Corporation of Ann Arbor, Michigan teamed to engineer and operate the POC agglomeration process. Arcanum provided agglomeration process expertise and conducted the bench-scale tests to obtain design data for the POC test module engineering. Bechtel provided the project management, plant design, procurement, construction, and operation supervision. EPRI's CQ Inc., provided both the site and the operators of the POC test module. EXPORTech, Inc., of New Kensington, PA, performed the laboratory analysis work.

PROGRAM OBJECTIVES AND MAJOR TASKS

The primary project objective was to perform large-scale POC tests and evaluate the spherical agglomeration process for physical fine coal cleaning. Baseline performance and economic data was to be collected for evaluating the commercialization potential of the process. Specific process performance objectives included:

- Achieving significant (greater than 80 percent) pyritic sulfur reductions
- Producing a coal with an ash content of less than two percent
- Achieving an energy recovery of at least 80 percent based on run-of-mine (ROM) coal

The program included the following major tasks:

- Bench-scale tests with batch and continuous agglomeration units to establish base-line conditions for POC testing and provide engineering criteria
- Bench-scale tests of a selective grinding system for the POC plant to provide improved liberation of coal
- Engineering and installation of the 1/3 tph POC test module at EPRI's CQ Inc., at Homer City, Pennsylvania
- Operation of the POC test module and POC tests
- Conceptual designs and cost estimates for a commercial-scale agglomeration plant
- Data evaluation and report preparation

BENCH-SCALE TESTS

Bench-scale tests were performed to establish the process performance characteristics and develop the engineering data for design of a 1 tph test module. Bench-scale testing was conducted in a batch mode and in a semi-continuous mode. The tests confirmed that the agglomeration process could consistently meet the program objectives. The main factor found to affect agglomeration performance was the success of grinding to liberate pyrite and other ash-forming minerals from coal.

The bench-scale tests used samples of coals from the Pittsburgh, Illinois No. 6, and Upper Freeport seams. These coals are important to the U.S. utility industry and have high ash and sulfur contents. Each test coal was precleaned and ground in two stages to ultrafine size (50 percent of the particles passing 10 microns by weight, d_{50}). The analysis of the ROM and precleaned coals and the bench-scale test results are presented in Table 1.

The table shows that the agglomeration energy recovery levels were excellent (over 98 percent), and the pyritic sulfur reductions were 70 to 90 percent compared to the ROM coal. Ash reductions varied between 77 and 94 percent for the three coals. Although a 2 percent ash clean coal was not obtained, it was shown that this product quality could be achieved with coals having natural medium to low ash content.

Table 1

SUMMARY BENCH-SCALE TEST RESULTS
AVERAGE OF CONTINUOUS TESTING
(Dry Basis)

		Pittsburgh Coal	Illinois No. 6 Coal	Upper Freeport Coal
ROM Coal	Ash,%	39.16	15.71	57.31
	Total Sulfur,%	4.71	4.54	2.11
	Pyritic Sulfur,%	2.93	2.46	1.93
	Heating Value,Btu/lb	8528	11837	5947
Precleaning	Product (clean coal)			
	Ash,%	12.10	9.60	16.30
	Total Sulfur,%	4.37	3.17	2.18
	Pyritic Sulfur,%	2.02	0.96	1.62
	Heating Value,Btu/lb	12791	12705	12698
	Yield,wt.%	62.6	79.8	43.3
	Energy Recovery	93.9	85.7	92.5
	SO_2 Reduction,%[1]	38.1	34.9	51.6
	Pyritic SO_2 Reduction,%	54.0	63.6	60.7
Agglomeration	Clean Coal:			
	Ash,%	4.81	4.23	8.04
	Total Sulfur,%	3.38	3.05	1.58
	Pyritic Sulfur,%	1.06	0.71	0.85
	Heating Value,Btu/lb	13908	13541	14080
	Yield,wt.%	91.0	93.4	92.3
	Energy Recovery	98.9	99.6	99.8
Overall Results	ROM Basis:			
	Energy Recovery	92.9	85.3	91.8
	Ash Reduction,%	92.5	76.5	94.1
	SO_2 Reduction,%	56.0	41.3	68.4
	Pyritic SO_2 Reduction,%	77.8	74.8	81.4

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[1] All reductions are on a constant energy (lb/MMBtu) basis

Figure 1 shows the high-shear and low-shear reactors used in the bench-scale testing. Figure 2 shows the separation of the finished 1/4-inch pellets. The well-formed agglomerates are seen distinctly against a background of milky white water laden with mineral matter.

The importance of maximum liberation of coal from other minerals was apparent from the bench-scale test program. The tests showed that conventional two-stage grinding to ultrafine sizes did not adequately liberate all coal from non-coal minerals. Conventional grinding preferentially ground the softer coal to smaller particles but did poorly in grinding harder minerals. A "selective" grinding system was conceived to also grind the harder, and denser, pyritic and ash-forming minerals. The bench-scale test plan was revised to include the testing of the new grinding circuit at the facilities of AMAX corporation in Golden, Colorado. The tests validated the expected improvement in mineral liberation.

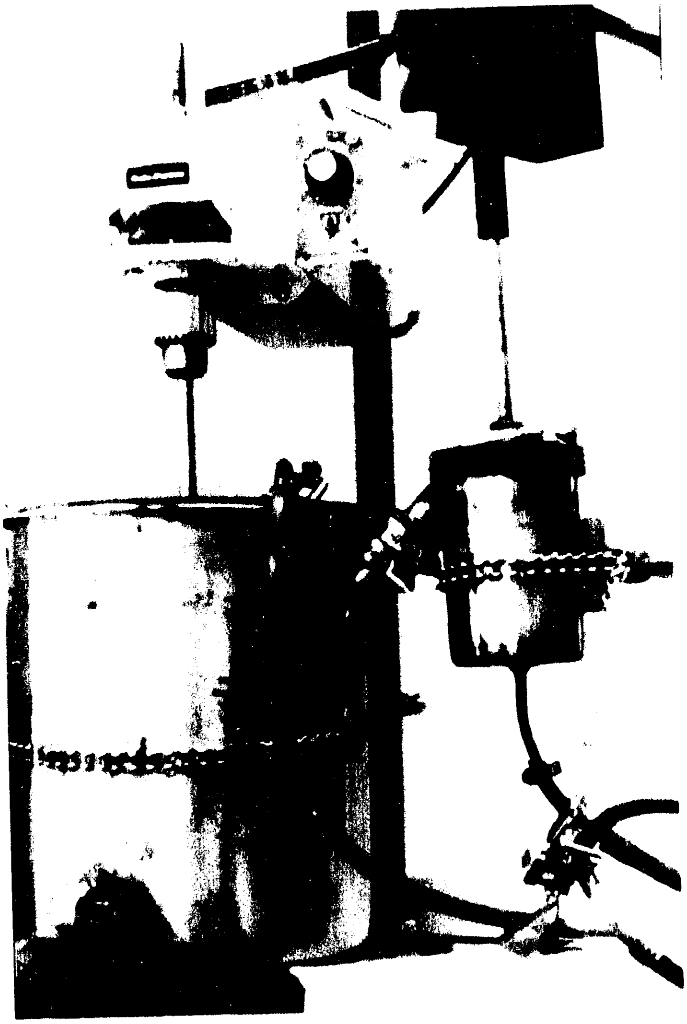

Figure 1 Bench-Scale High-Shear and Low-Shear Continuous Unit

Figure 2 Separation of Agglomerates from Mineral-Matter-Laden Water in Bench-Scale Test

THE TEST MODULE

Engineering and construction of the POC test module began June 1989 and was completed October 1990. The 1/3 tph POC test module installed at Homer City was smaller than the originally planned 1 tph unit. The project had an extensive health and safety system including a gas blanket, relief flare, ventilation, and fire protection systems.

A simplified POC process flow diagram is shown in Figure 3. This figure also shows the major subsystems of the plant.

Each feed coal for the process is received from the mine or coal preparation plant at the CQ Inc. site by truck, ground to 1/4-inch x 0, sampled, and then stored.

The POC process consists of four major operations. These are:

- Selective grinding to produce ultrafine feed slurry
- Agglomeration of the coal and separation of the clean coal from the mineral matter laden tailings
- Steam distillation and recovery of heptane from agglomerates
- Dewatering of the agglomerates for shipment and the tailings for disposal

Selective Grinding. The selective grinding system is designed to operate at a capacity of 1 tph. The 1/4-inch x 0 feed coal is slurried and ground in a ball mill to a 100 mesh x 0 size. The slurry is then sent to a solid bowl centrifuge which operates as a classifier to provide a separation at 20 microns (90 percent of the particles passing 20 microns by weight, d_{90}). The centrifuge cake consisting of the +20 micron material is reslurried and sent to the bead mill for further size reduction. The bead mill product is sent back to the centrifuge to close the recirculation loop. The centrifuge effluent, consisting of the -20 micron material, forms the feed to the agglomeration process. Water is added to produce feed slurries with 15 to 20 percent solids by weight.

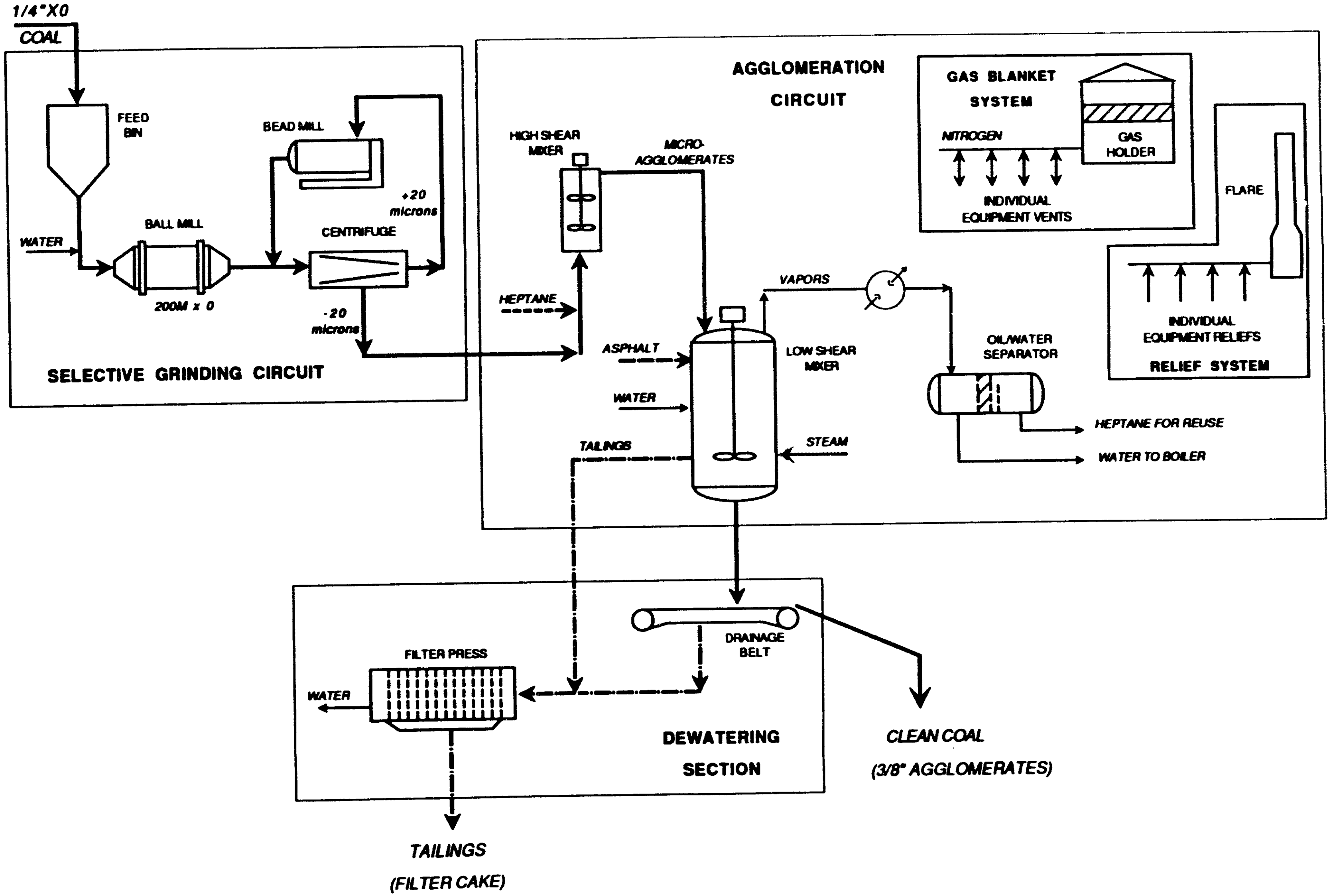

Figure 3 Simplified Flowsheet for Spherical Agglomeration Proof-of-Concept Plant

The ball mill, bead mill, and the centrifuge are shown as Figures 4 and 5 respectively.

Agglomeration. The agglomeration circuit processes the coal through the following steps:

- Microagglomerate formation
- Agglomerate formation
- Separation of agglomerates from mineral matter
- Heptane recovery
- Product dewatering and loadout

A high-shear reactor is used to mix heptane (15 to 30 percent by weight of coal) with the coal slurry from the selective grinding system. The high-shear reactor produces microagglomerates which, along with the water and mineral matter, overflows the high-shear reactor to fill the low-shear reactor.

When necessary, a small amount of asphalt dissolved in heptane is added to the high-shear reactor. This promotes the formation of microagglomerates in coals which are difficult to agglomerate (such as Illinois No. 6).

The microagglomerates formed in the high-shear reactor are further enlarged in the low-shear reactor by the addition, under gentle agitation, of 2 to 5 percent by weight of asphalt. After pellets of sufficient size and strength are formed, the agitation is stopped and the slurry is drained through a screen to remove the mineral matter laden tailings.

Heptane recovery uses 25 psig steam to heat the bed of agglomerates and strip the heptane from the solids. The vaporized heptane and steam are condensed and the heptane decanted for reuse.

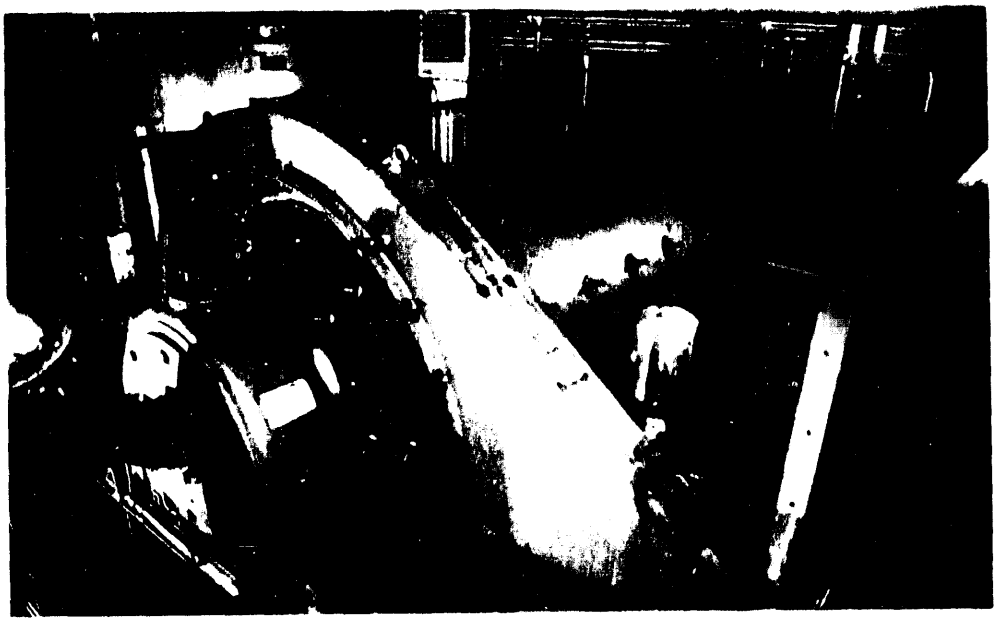

Figure 4 Selective Grinding Circuit Ball Mill

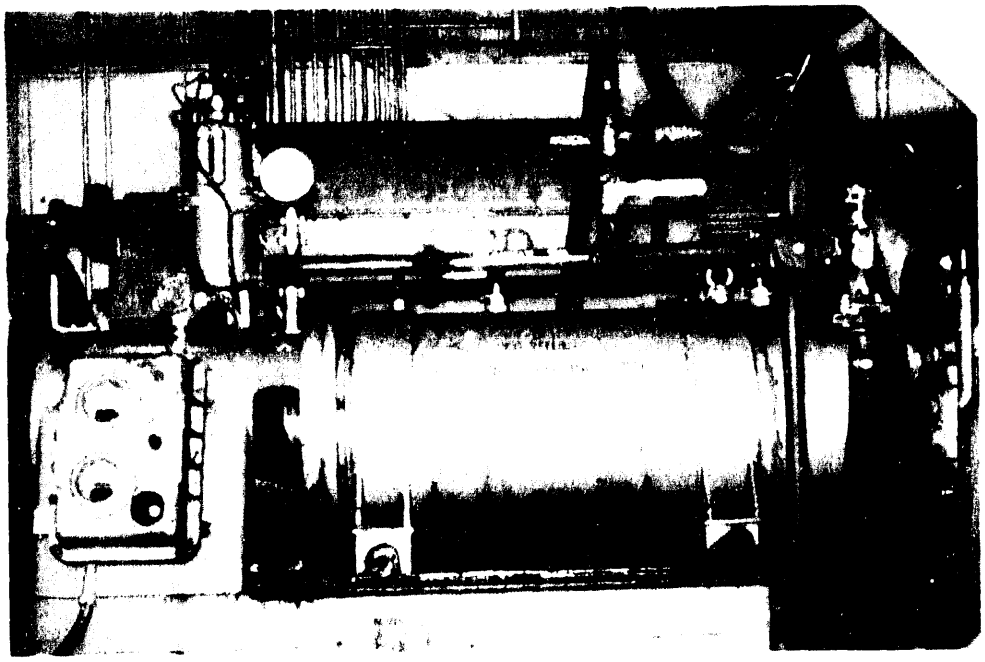

Figure 5 Selective Grinding Circuit Centrifuge and Stirred Bead Mill

Once heptane is recovered, the pellets are quenched with water and flushed from the reactor onto a drainage belt. Drained agglomerates are loaded into barrels for shipment to other DOE test programs. Tailings from the low-shear reactor and the drainage belt underflow are sent to a plate-and-frame filter press. The filter press dewaters the solids for disposal and recovers the water for reuse.

The high-shear and low-shear reactors used in the POC test module are shown in Figure 6.

Safety Systems. Heptane is highly combustible and its use required precautions – especially in a coal cleaning environment where large amounts of coal fines are present. Several safety systems are built into the POC test module:

- Nitrogen gas blanket for all heptane-containing vessels to keep the oxygen content in the tank atmospheres well below the lower explosive limit (LEL)
- A relief flare system to remove heptane vapors safely in the event that a process upset overpressurizes the gas blanket system
- A ventilation system using hydrocarbon detectors capable of sensing and dispersing buildups of heptane vapors in the building
- A fire protection system including flame detectors and deluge system

The design of the agglomeration section is patterned after proven petroleum plant engineering designs.

POC TEST RESULTS

The three coals tested in depth during the POC operation were:

- Illinois No. 6 seam coal from the Burning Star Mine, Perry County, Illinois
- Upper Freeport seam coal from the Helen Mine, Indiana County, Pennsylvania
- Pittsburgh coal from the Blacksville No. 2 Mine, Monongalia County, West Virginia

Taggart seam coal from the Wentz No. 1 preparation plant, Wise County, Virginia was also briefly tested. This coal and the Pittsburgh coal were precleaned at the mine.

A representative sample of agglomerates is shown in Figure 7. Agglomerates of a nominal 3/8-inch size with less than 20 percent moisture were produced from all the test coals.

It was noted during the preliminary runs with the selective grinding circuit that the solid bowl centrifuge was very effective in keeping the coarse and difficult-to-grind high ash and pyrite content particles from being fed to the agglomeration circuit. However this resulted in a build-up of these particles in the recirculation loop. This offered an opportunity to reject these particles before agglomeration without further grinding. A spiral separator was used to effectively remove these particles.

Table 2 presents a summary of the feed coal characteristics and the POC test products. Agglomeration had a 97 percent, or greater, energy recovery in all cases. The best results including all precleaning operations resulted in ROM energy recoveries from 84.4 to 95 percent. Ash reductions compared to the ROM were 77 to 83 percent with ash contents of the clean coal products varying from 1.5 to 5.5 percent (dry basis). ROM pyritic sulfur reductions varied from 86 to 90 percent for the three major test coals, resulting in total sulfur reductions of 47 to 72 percent.

The agglomeration process performance was compared to centrifugal float/sink tests of the same micronized coal feed. It was found that agglomeration separations and recoveries were better than those obtained using centrifugal float/sink methods.

The POC tests confirmed that particle top size and solids size distribution were the dominant factors affecting agglomeration process performance. Selective grinding of coal which had been precleaned using conventional methods produced the best clean coal

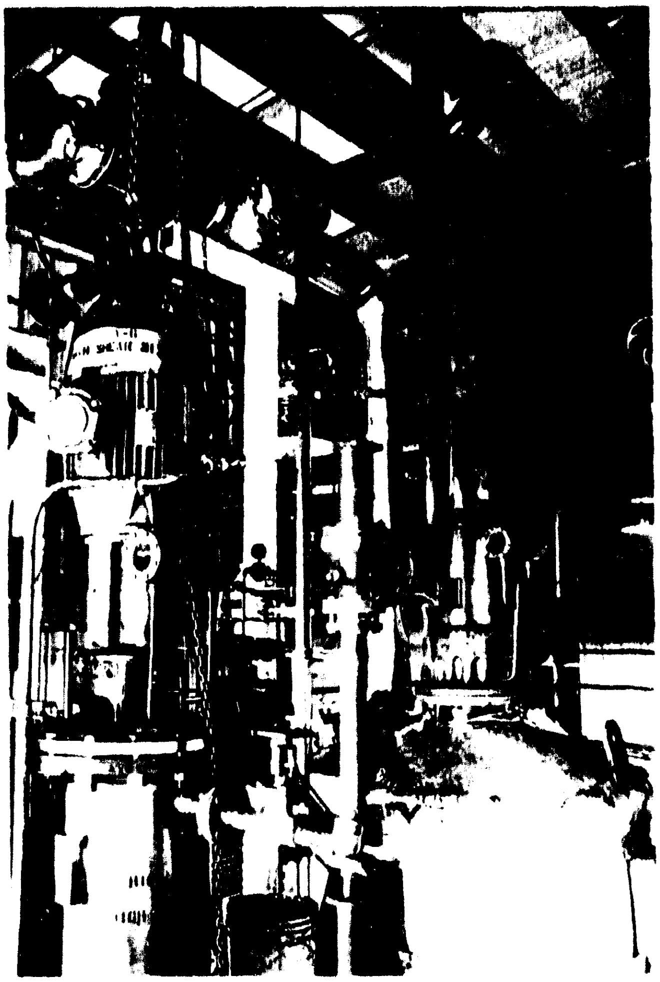

Figure 6 High-Shear and Low-Shear Reactors and Mixers (green) in Proof-of-Concept Test Plant

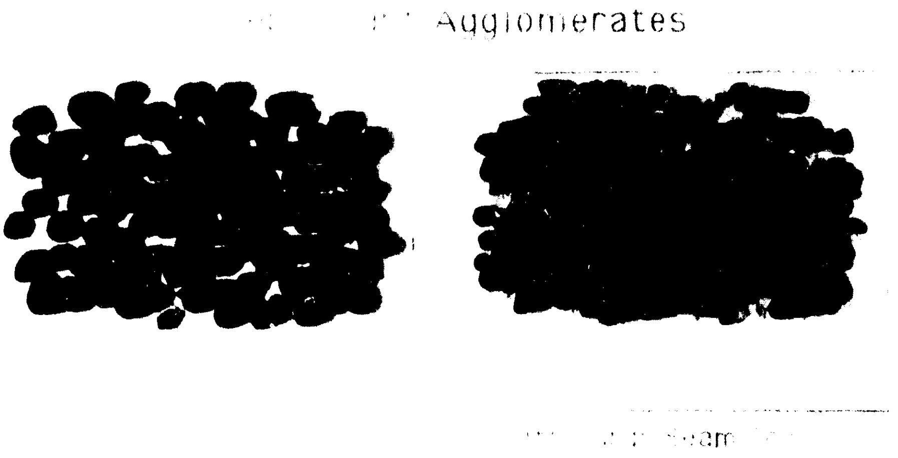

Figure 7 Agglomerates from Proof-of-Concept Plant

Table 2
SUMMARY POC TEST RESULTS
SELECTIVE GRIND WITH SPIRAL SEPARATOR
(Dry Basis)

		Pittsburgh Coal[2]	Illinois No. 6 Coal[3]	Upper Freeport Coal
ROM Coal	Ash,%	13.18	17.40	23.27
	Total Sulfur,%	4.71	4.03	3.74
	Pyritic Sulfur,%	2.88	1.81	2.87
	Heating Value,Btu/lb	13081	11708	11551
Precleaning	Product (clean coal)			
	Ash,%	9.43	15.16	17.81
	Total Sulfur,%	2.42	2.96	2.16
	Pyritic Sulfur,%	1.25	0.73	1.39
	Heating Value,Btu/lb	13635	12604	12547
	Yield,wt.%	90.0	91.0	87.3
	Energy Recovery	93.8	93.8	94.8
	SO_2 Reduction,%[2]	50.7	28.7	46.8
	Pyritic SO_2 Reduction,%	58.5	60.9	55.4
Agglomeration	Clean Coal (with binder):			
	Ash,%	3.40	3.54	5.48
	Total Sulfur,%	1.59	2.57	1.35
	Pyritic Sulfur,%	0.34	0.21	0.52
	Heating Value,Btu/lb	14793	14013	14080
	Energy Recovery	90.0	99.1	99.6
Overall Results	ROM Basis:			
	Energy Recovery	84.4	93.0	94.4
	Ash Reduction,%	76.9	83.0	81.7
	SO_2 Reduction,%	69.4	46.7	71.9
	Pyritic SO_2 Reduction,%	89.8	90.3	85.9

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[2] Precleaning of Pittsburgh Coal at preparation plant - results shown with energy recovery of selective grinding circuit grouped with agglomeration.

[3] Precleaning of Illinois and Uper Freeport coals by selective grinding circuit.

[4] All reductions are on a constant energy (lb/MMBtu) basis.

quality after agglomeration. Removal of larger pyritic sulfur particles by conventional cleaning improved the overall pyritic sulfur reduction. Petrographic analysis indicated significant rejection of pyrite crystals which were free or semi-locked in coal by the agglomeration process.

COMMERCIAL-SCALE PLANT

A 350 tph commercial plant conceptual design using the agglomeration process was prepared. The plant was designed to clean a 50:50 blend of Upper and Lower Freeport coal from the Hepburnia Coal Company's Clearfield County mine in Pennsylvania. This coal has an ash content of 12.1 percent with a sulfur dioxide emission potential of 2.8 lb/MMBtu. This coal is representative of an estimated 2-1/4 billion tons of recoverable Upper Freeport Seam coal and 1 billion tons of Lower Freeport Seam coal in Pennsylvania alone[2].

The commercial agglomeration plant simplified block flow diagram is shown in Figure 8. The plant uses conventional cleaning technology to produce a high quality clean coal, a middlings product, and reject rock from the coarse size fraction of the feed coal (1 1/4-inch x 14M). Natural coal fines and selectively ground middlings are sent to agglomeration.

The plant produces a product containing 4.4 percent ash with an SO_2 emission potential of 1.0 lb/MMBtu. The processing costs are estimated at $15.9 per ton added cost to coal (including operation and maintenance costs and capital charges).

CONCLUSIONS

A first-of-a-kind facility to agglomerate micronized coals with heptane as the bridging liquid was successfully constructed and operated. The POC module included a selective grinding circuit, agglomeration using a light hydrocarbon bridging liquid, and thermal

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[2] "The Reserve Base of Bituminous Coal and Anthracite for Underground Mining in the Eastern United States," USBM, IC-8655, 1974

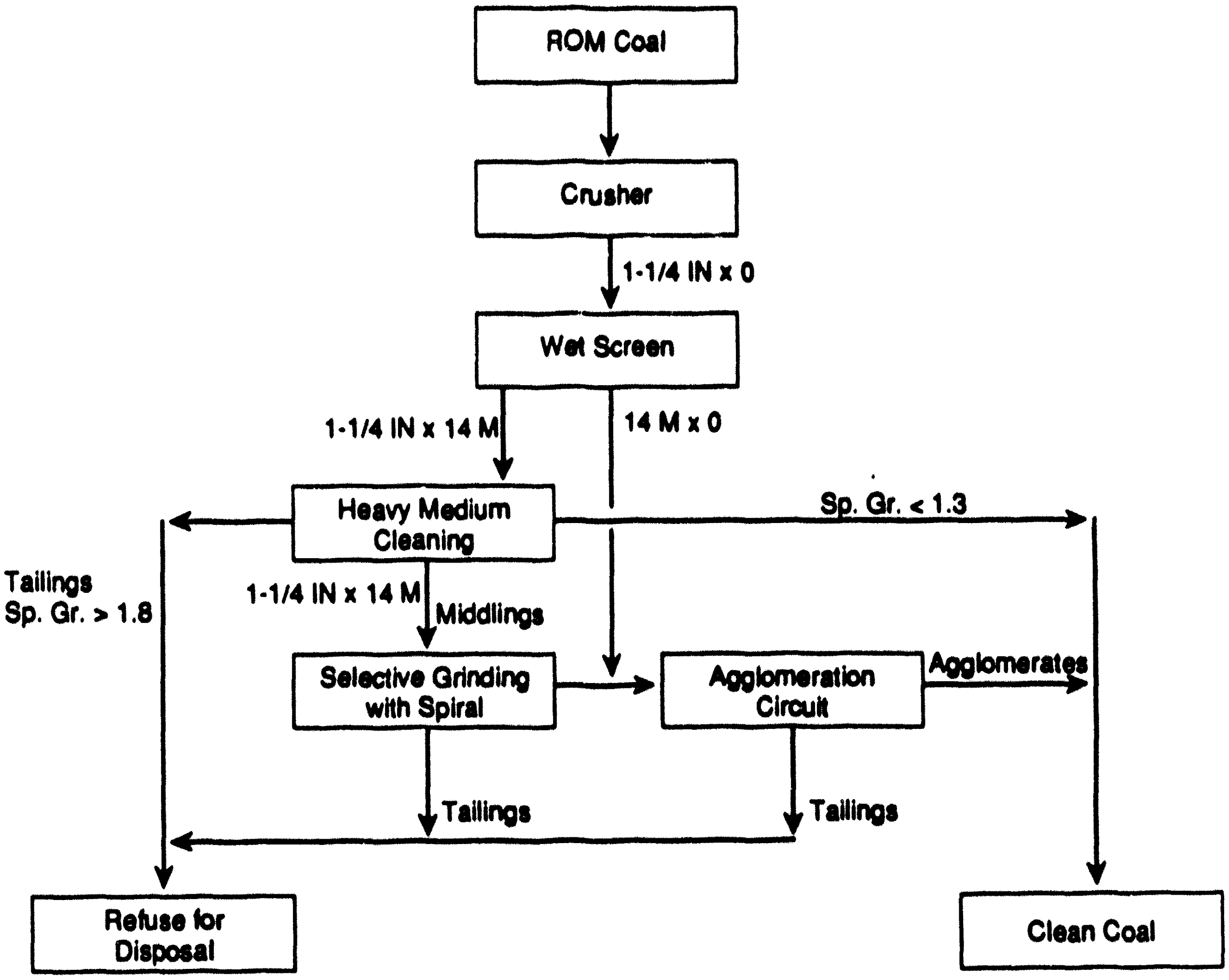

Figure 8 Commercial Agglomeration Cleaning Plant Simplified Block Flow Diagram

recovery of the heptane bridging liquid. The process rejected a high percentage of the liberated pyritic and ash-forming minerals, and produced a product that can be easily transported by conventional coal handling methods.

Further commercial development requires a demonstration plant. The plant design should include continuous operation of the heptane recovery system and the demonstration should be over a longer time period. Objectives include improving thermal efficiency, minimizing heptane consumption, confirming capital and operating costs and the physical characteristics of the agglomerates.

The selective grinding of coal requires further research to optimize process equipment design and plant operation. Test methods to quantify liberation of different coals also require development.

Section 1

INTRODUCTION

1.1 GENERAL

This report presents the results of work performed by Bechtel between November 1987 and September 1990 for the "Advanced Physical Fine Coal Cleaning-Spherical Agglomeration" program. The program was funded by the Pittsburgh Energy Technology Center of the U. S. Department of Energy (PETC/DOE) under Contract No. DE-AC22-87PC79867.

Coal-fired power plants currently emit about 17 million tons of sulfur dioxide per year. In August 1984, the U.S. Department of Energy (DOE) and the Electric Power Research Institute (EPRI) signed a project agreement to further their common objectives of reducing sulfur dioxide emissions from coal-fired power plants and improving power plant economics. Both DOE and EPRI recognized that advanced physical fine coal cleaning techniques could be developed to produce high quality fuels to cost-effectively meet these objectives. The project agreement was meant to serve as a vehicle for joint promotion of research in fine coal cleaning techniques.

The spherical agglomeration process for cleaning fine coal as offered by the Bechtel/Arcanum team was considered by DOE to be at the stage of development where further testing at a larger proof-of-concept (POC) scale was warranted. A contract was awarded to Bechtel in September 1987. This program was one of the research activities on which DOE and EPRI cooperated under their project agreement. A spherical agglomeration plant was designed, installed, and tested at EPRI's Coal Quality Development Center (CQ Inc.) at Homer City, Pennsylvania. Results of the tests and an evaluation of the process are presented in this report.

1.2 PURPOSE AND OBJECTIVES

The primary objective of this joint DOE/EPRI research project was to perform large-scale POC testing and evaluation of spherical agglomeration (an advanced physical fine coal cleaning process). The test work was intended to further establish baseline performance and economic data necessary for decision making regarding the commercialization potential of the process. Performance testing was to be done at a nominal 1-ton-per-hour scale of operation.

Specific performance objectives included the following targets:

- A significant pyritic sulfur reduction: The process should be capable of significantly reducing the pyritic sulfur content of any bituminous coal (28 mesh x 0 or finer) as compared to reduction achievable by state-of-the-art coal cleaning processes
- An ash content of not more than 2 weight percent (dry basis) in the clean coal
- For every 100 Btus fed to the plant in the form of ROM coal, not less than 80 to be recovered in the clean coal product
- As a measure of the easy handling and transportation characteristics, the clean coal and refuse products should have surface moisture contents of not greater than 30 weight percent

The POC test work was also meant to address the following:

- Chemically and physically characterize the clean coal in respect to subsequent use for combustion, gasification, and possibly, liquefaction
- both chemically and physically characterize the refuse (solids and liquids, if any) in respect to their disposal in an environmentally acceptable manner
- Provide process and engineering data for conceptual designs that would enable preparation of order-of-magnitude (plus or minus 30 percent) cost estimates

1.3 PROGRAM APPROACH

The team formed by Bechtel for the program included Arcanum Corporation of Ann Arbor, Michigan, an organization devoted to spherical agglomeration research.

The spherical agglomeration process used in the program has been researched by C.E. Capes of the National Research Counsel of Canada (NRCC) for several decades. Arcanum has further developed the process under a license from NRCC.

The project consisted of the following nine tasks:

- Task 1 - Project Planning
- Task 2 - Engineering of the Proof-of-Concept unit
- Task 3 - Procurement and Fabrication
- Task 4 - Installation and Shakedown
- Task 5 - Test Plan Formulation
- Task 6 - Bench-Scale Testing
- Task 7 - Test Plan Implementation, Process Operation
- Task 8 - Process Evaluation
- Task 9 - Equipment and Process Removal

Bechtel managed the project during all the phases and was primarily responsible for engineering, procurement, construction, startup, plant operation, formulation of the test plans, implementation of the POC test work, and data evaluation. The experience of NRCC and Arcanum was available throughout the project, specifically in the areas of process design, equipment designs and plant startup. In addition, the bench-scale tests, an essential element in the program, were performed at Arcanum's facilities and Arcanum personnel also took part in the startup and POC testing activities.

The bench-scale tests with the coal selected for the POC tests that were performed at Arcanum helped establish process design parameters for the plant and its vital components. They were also used to formulate the test plan and plant operating parameters.

Full details of the activities performed under the different project tasks are detailed in the report sections that follow.

Since the process used heptane, an inflammable liquid, health and safety related issues were paid particularly close attention in the design, training, and operation of the facility.

Union Electric, an EPRI member utility, furnished 200 tons of Illinois No.6 seam coal for the testing from the River King Mine No. 6. The Pittsburgh seam coal was provided by the New York State Electric and Gas Corporation (NYSEG) and Upper Freeport coal was provided by EPRI.

After successful completion of the plant startup activities, POC tests commenced in November 1989 and were completed in March 1990.

The laboratory analysis work was performed by EXPORTech of New Kensington, Pennsylvania.

1.4 REPORT ORGANIZATION

This volume contains 12 report sections. Section 2 provides background information on the spherical agglomeration technology and a brief description of the POC test module.

Section 3 describes the project planning, POC plant engineering, installation, and shakedown activities performed.

The bench-scale tests conducted at Arcanum are described in Section 4. Information on the test and sampling plans that were formulated is included in Section 5. Section 6 deals with the implementation of the test plan and process operation. The process evaluation criteria and definition of terms appear in Section 7. Section 8 presents the proof-of-concept test results and an evaluation of the process.

Product characterization forms the subject of Section 9. Performance evaluation of the selective grinding circuit is covered under Section 10. A conceptual design of a commercial plant appears under Section 11. Finally, Section 12 summarizes the "lessons learned" from the project.

Laboratory analysis (printouts) and daily plant operation logs are furnished in Appendices A and B, respectively. These appendices are in Volume 2 of the report.

Section 2

TECHNOLOGY DESCRIPTION

2.1 COAL AGGLOMERATION PROCESSES

The basic principles of oil or spherical agglomeration have been known for several years. The process was patented by Trent in 1922.[1] In this process, a mixture of fine coal, water, and a water immiscible liquid hydrocarbon is subjected to intense agitation. The hydrophobic coal particles selectively form agglomerates with the hydrocarbon serving as the bridge. The noncoal mineral matter including pyrite, being hydrophilic, remains in the water as dispersed discrete particles. The coal agglomerates are screened out as a clean coal product. Nonagglomerated ash-forming minerals, including pyrite, are separated as a tailings slurry for further dewatering and disposal.

Several variations of the original Trent process have been developed since 1922. Variations of the process differ in the type of agitation used to disperse the oil, wet the coal surface, and agglomerate the coal particles; the type and quantity of oil used; and in the separation and subsequent treatment of the products.[2] The Olifloc and Convertol processes of oil agglomeration have been used in Europe and the USA.[3] In the late '70s, a 10 tph Shell Pelletizing Separator was installed in Japan. Research coal agglomeration has been conducted in various parts of the world including the U.S., Canada, and India.

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[1] W.E. Trent, "Process for Purifying Materials," U.S. Patent 1,420,164, June 20, 1922

[2] A.W. Deurbrouk, R.Hucko, "Chemistry of Coal Utilization," Second Supplementary Vol.

[3] A.H. Brisse and W.L. McMorris, Jr., "Convertol Process," Min. Engineering, Feb. 1958

Smith, Puddington, and Capes developed the spherical agglomeration process based on their research at the National Research Council Of Canada (NRCC).[4,5]

While spherical agglomeration and related processes have been demonstrated and in some cases used commercially for bituminous coals, widespread application of these processes in the coal industry has been retarded by the prevailing economic conditions. The processes used petroleum products as the essential bridging liquid. The manyfold increases in the price of petroleum products in the '70s and '80s severely affected the economics of the process.

For example, a 50 tph oil agglomeration plant based on development work performed by Conoco Research Division and Consolidation Coal Company was operated from 1978 to 1980 in the United States. The product contained 11.3 percent fuel oil No.6. According to available information, the plant was shut down due to the unfavorable price conditions for oil and the prevailing selling price of coal.[6]

Extensive spherical agglomeration tests have been conducted by NRCC at a pilot plant with a capacity of 2 tph and, since 1985, with a prototype unit having a capacity of 10 to 15 tph. Based on NRCC scale-up data, commercial plants of 25 to 50 tph have been built in the northeastern United States, many for the recovery of saleable coal from coal preparation plant waste ponds.[7]

However, the oil remaining with the coal causes the product to emit a penetrating smell. This characteristic has been cited by coal

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[4] H.M. Smith and I.E. Puddington, "Spherical Agglomeration of Barium Salts," Canadian Journal Of Chemical Engineering, Vol. 38, 1960

[5] C. E. Capes, et al., "Application of Spherical Agglomeration to Coal Preparation," 7th International Coal Preparation Congress, Sydney, Australia, May 1976

[6] Engelleitner, W.H., "Developments in the Agglomeration of Fine Coal," Proceedings of Third USA-Korea Joint Workshop in Coal Utilization Technology, PA, October 5-7, 1986

[7] C.E. Capes, R.D. Coleman, J.D. Hazlett, et al., "The Recovery and Utilization of Fine Coal" 89th Annual General Meeting Of CIM, May 1987, Toronto, Canada

customers as a disadvantage and has contributed, along with the oil cost, to the difficulty of commercializing the technology.

2.2 SPHERICAL AGGLOMERATION WITH HEPTANE

The spherical agglomeration process as developed by Arcanum and the subject of POC testing under this program differs from all other agglomeration processes in two key elements. It uses a low boiling point light hydrocarbon, heptane, as the bridging liquid and includes steps to recover the same from the agglomerates for reuse. No bridging liquid remains with the product. Asphalt is used as a binder. The product does not smell and the asphalt provides sufficient strength for handling. The improvements have removed the most serious impediments to widespread commercial application of spherical agglomeration, namely, product smell and high cost of bridging liquid consumption.

The bridging liquid, heptane, has a boiling point of 200°F at atmospheric pressure and forms an azeotrope with water which makes steam stripping practical. Unlike many other chemicals suggested by other researchers for this duty, heptane is comparatively harmless. However, it is inflammable and it must be handled with care. The process also uses a small amount (2 to 3 percent) of asphalt for providing strength to the finished agglomerates after heptane recovery. The POC test work was designed to validate these developments at a nominal 1 tph scale.

2.3 POC TEST UNIT DESCRIPTION

The plant description highlights operations in the following areas:

- Selective grinding circuit
- Preparation of microagglomerates
- Low-shear reactor/stripper
- Heptane and asphalt handling
- Tailings filtration
- Boiler feed and process water system
- Vapor handling

A process block flow diagram and material balance are presented as Table 2-1 and Figure 2-1, respectively. (As-built P&IDs are included in the appendix.)

2.3.1 Selective Grinding Circuit

The precleaned and crushed (1/4-inch x 0) coal is stored in the feed bin (EX-2 on Figure 2-2). Load cells monitor the amount of coal in the bin. A vibrating bin bottom device facilitates the flow of coal from the bin. A weigh feeder (EX-3) located immediately below the feed bin delivers a preset rate of coal to a ball mill (EX-5). Water is added to the mill for wet grinding. The discharge end of the mill is equipped with a trommel screen to remove oversize tramp material from the ground coal slurry. The ball mill product slurry is diluted with water, combined with the fine grinding mill (EX-8) product slurry, and pumped to the centrifuge feed sump (D-13).

A solid bowl centrifuge (Y-6) classifies the feed slurry at about 20 microns. The centrate slurry (15-20 percent solids) made up of minus 20 micron particles is delivered to the primary sump (EX-11) as feed to the agglomeration circuit. Oversize material from the centrifuge collects in the fine grinding mill sump (EX-7) to feed the fine grinding mill (EX-8). Slurry product from the fine grinding mill is returned by gravity to the ball mill discharge sump (D-12).

The fine grinding mill (EX-8) consists of a stationary horizontal cylinder lined with abrasion-resistant material. It is fitted with a rotating agitator and can be filled with steel or ceramic beads. As the agitator turns, at about 700 rpm, the beads grind the coal to a very fine size. The product from the mill has a top size of 30 to 40 microns. A small amount of this slurry stream can be diverted to the tailings filtering station to serve as a filtering aid.

A sampler is provided to collect representative slurry samples ahead of the primary sump. The ground slurry is pumped to the slurry feed

Table 2-1

DESIGN PROCESS MATERIAL BALANCE

1-TON PER HOUR PLANT

Stream No.	Stream Name	Solids, lb/hr			Total Solids, lb/hr	Water, lb/hr	Heptane, lb/hr	Total Materials, lb/hr
		Coal MAF	Ash	Binder				
1	Feed Coal	1,598	282	-	1,880	120	-	2,000
2	Water to Grinding	-	-	-		10,533	-	10,533
3	Ground Coal Slurry	1,598	282	-	1,880	10,653	-	12,533
4	Heptane	-	-	-	-	-	519	519
5	HS Reactor Feed	1,598	282	-	1,880	10,653	519	13,052
6	Reactor Product	1,598	282	-	1,880	10,653	519	13,052
7	Binder Mix	-	-	85.9	85.9	-	201	287
8	Steam	-	-	-	-	3,169	-	3,169
9	Primary Tailings	47.9	234	2.6	284.5	9,693	-	9,978
10	Stripped Vapor	-	-	-	-	2,826	720	3,546
11	Stripped Agglom Slurry	1,550.1	48.0	83.3	1,681.4	1,303	-	2,984
12	Secondary Tailing	32.0	12.5	1.7	46.2	24,759	-	24,805
13	Recovered Water	-	-	-	-	37,410	-	37,410
14	Refuse Filter Cake	79.9	246.5	4.3	330.7	222	-	553
15	Dewatered Coal Product	1,518.1	35.5	81.6	1,635.2	408	-	2043
16	Recovered Heptane	-	-	-	-	-	720	720
17	Condensed Water	-	-	-	-	2,826	-	2,826
18	Spray #1	-	-	-	-	23,864	-	23,864
19	Spray #2	-	-	-	-	3,180	-	3,180
20	Makeup Water	-	-	-	-	510	-	510

(a) For steam location refer to Figure 2-1.

(b) Heptane remaining in the product is 0.01 lb/hr; not shown with one decimal calculations.

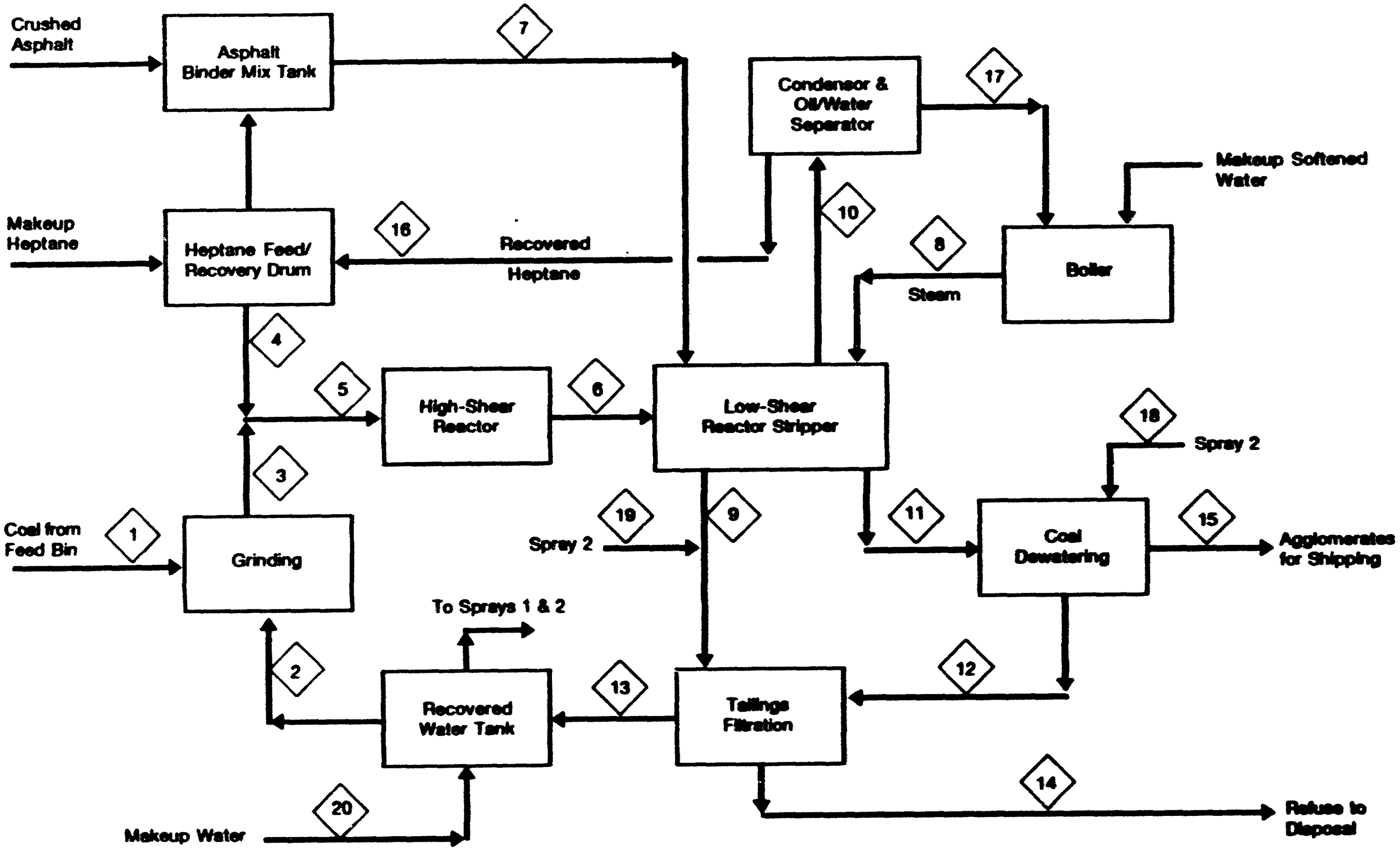

Figure 2-1 Spherical Agglomeration Block Flow Diagram

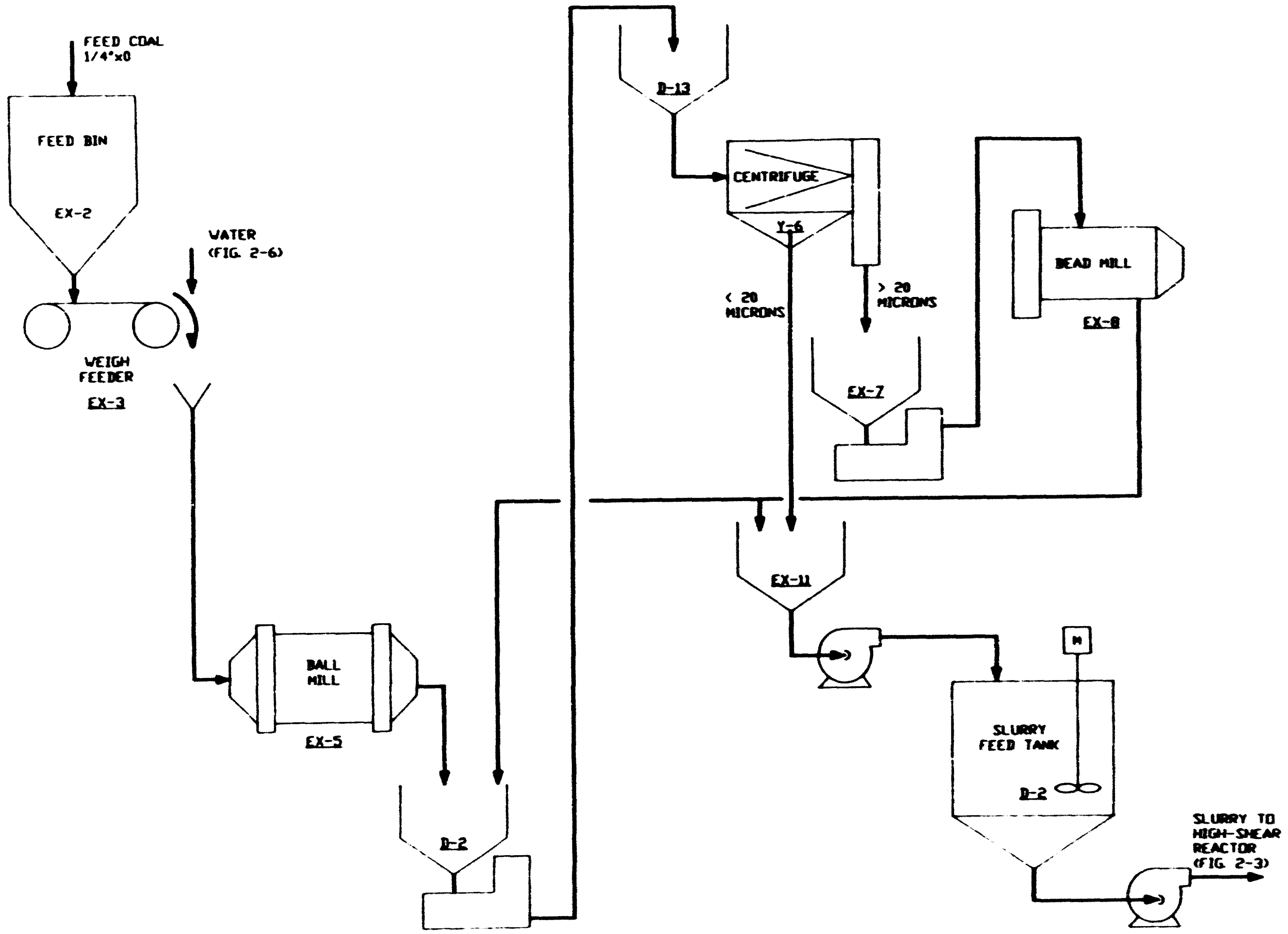

Figure 2-2 Grinding Circuit Simplified Process Flow Diagram

tank (D-2) by a centrifugal pump. A variable speed centrifugal pump delivers the slurry to the high shear reactor from the slurry feed tank.

2.3.2 Preparation of Microagglomerates

This step is illustrated in Figure 2-3. Selectively ground slurry is fed to the high-shear reactor (C-6). A measured quantity of heptane bridging liquid (25 to 30 weight percent of the coal) is added to the coal slurry prior to the reactor. An asphalt/heptane solution (binder) can also be added at the high-shear reactor if required as a conditioner for difficult to agglomerate coals such as Illinois No. 6 seam coal.

The high-shear reactor is a vertical cylindrical vessel fitted with a variable speed turbine agitator. Intense agitation causes particles of carbonaceous material to become wetted with heptane and coagulate into microagglomerates. These particles, being hydrophobic, adhere together with the heptane acting as a bridge between the particles. Ash-forming minerals are not wetted by heptane and stay in the water as finely dispersed particles. The microagglomerates at this stage are from 0.5 to 1 mm in diameter.

The slurry with microagglomerates overflows from the high-shear reactor into the low-shear reactor (LSR) thus initiating the filling cycle of the LSR.

The microagglomerates are too fragile for screening and steam stripping. Therefore, they are pelletized with a small amount of asphalt to a 3/8-inch size in the low-shear reactor/stripper.

2.3.3 Low-Shear Reactor/Stripper

The LSR (C-8) operates in a batch mode to accomplish its multiple functions. It is fitted with a single four-blade paddle mixer with a variable speed drive. The design batch functions and their sequence are shown in Figure 2-4. The agglomerate growth and heptane recovery device is shown in Figure 2-1.

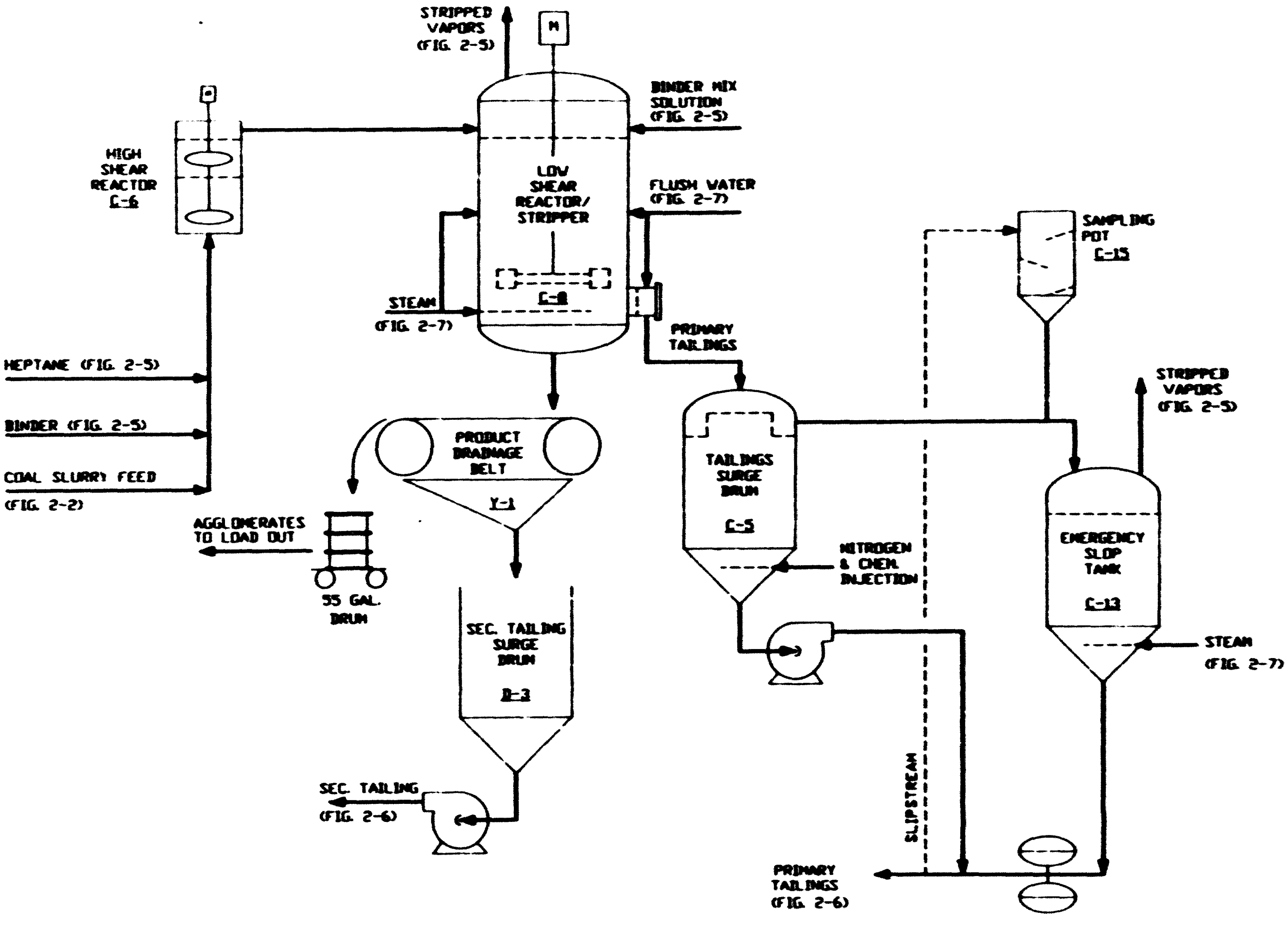

Figure 2-3 Agglomeration Circuit Simplified Process Flow Diagram

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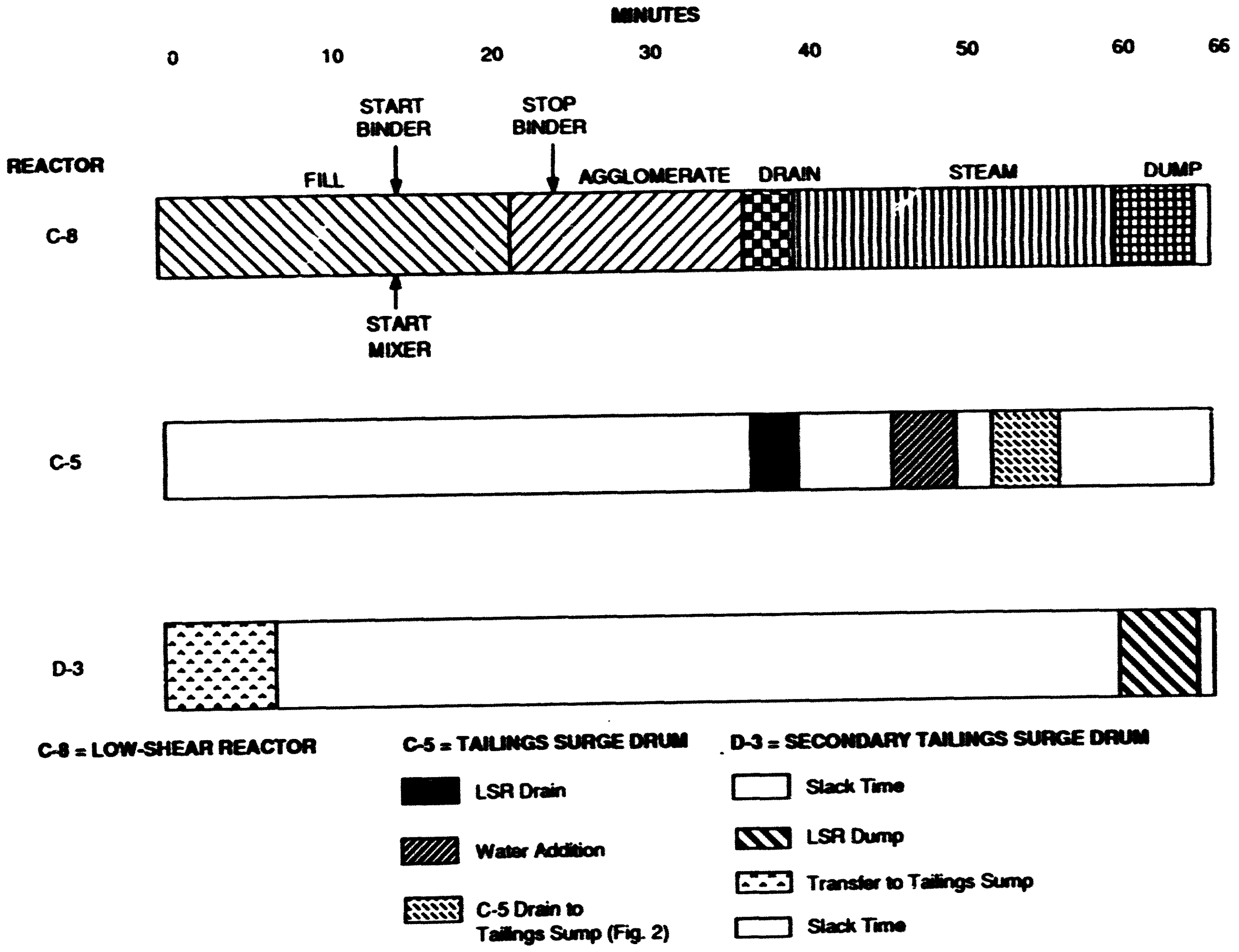


Figure 2-4 Low-Shear Reactor/Stripper Batch Sequence

Function	Duration (minutes)
Filling	22
Mixing/agglomerate growth	15
Tailings drainage	3
Heptane steamout	20
Reslurrying and dumping the reactor	5
Standby	1
Total	66

During the slurry filling cycle, the binder mix (asphalt dissolved in heptane) is also fed to the LSR. Upon reaching the predetermined level, the feed valve to the LSR is closed, the slurry flow to the high-shear reactor interrupted and the reactor operation stopped. After the LSR filling cycle is completed, its mixer speed is increased to a preprogrammed set point for the pelletizing step. At the end of the pelletizing step, the mixer is stopped and the primary tailings are drained from the vessel through a 50 mesh screen. While the agglomerates remain in the vessel, the drained water with suspended solids (primary tailings) is sluiced to the tailings surge drum (C-5).

During the filling and draining cycle the LSR vapor space is opened to a nitrogen blanketing header. Then, in preparation for steam stripping, the reactor's overhead three-way valve is lined up and opened to the stripper condenser (E-2 in Figure 2-5). A natural gas-fired package boiler is used to generate steam required for the heptane stripping operation.

Steam is first introduced into the top of the reactor where it sweeps nitrogen from the vessel and heats the vessel head to prevent heptane condensation on cold surfaces and refluxing. Steam is then sparged into the bed of agglomerates from the bottom of the reactor, stripping heptane from the agglomerates. Heptane vapor and steam from the reactor are then condensed in the stripper condenser (E-2 in Figure 2-5). Condensed heptane and water are separated by

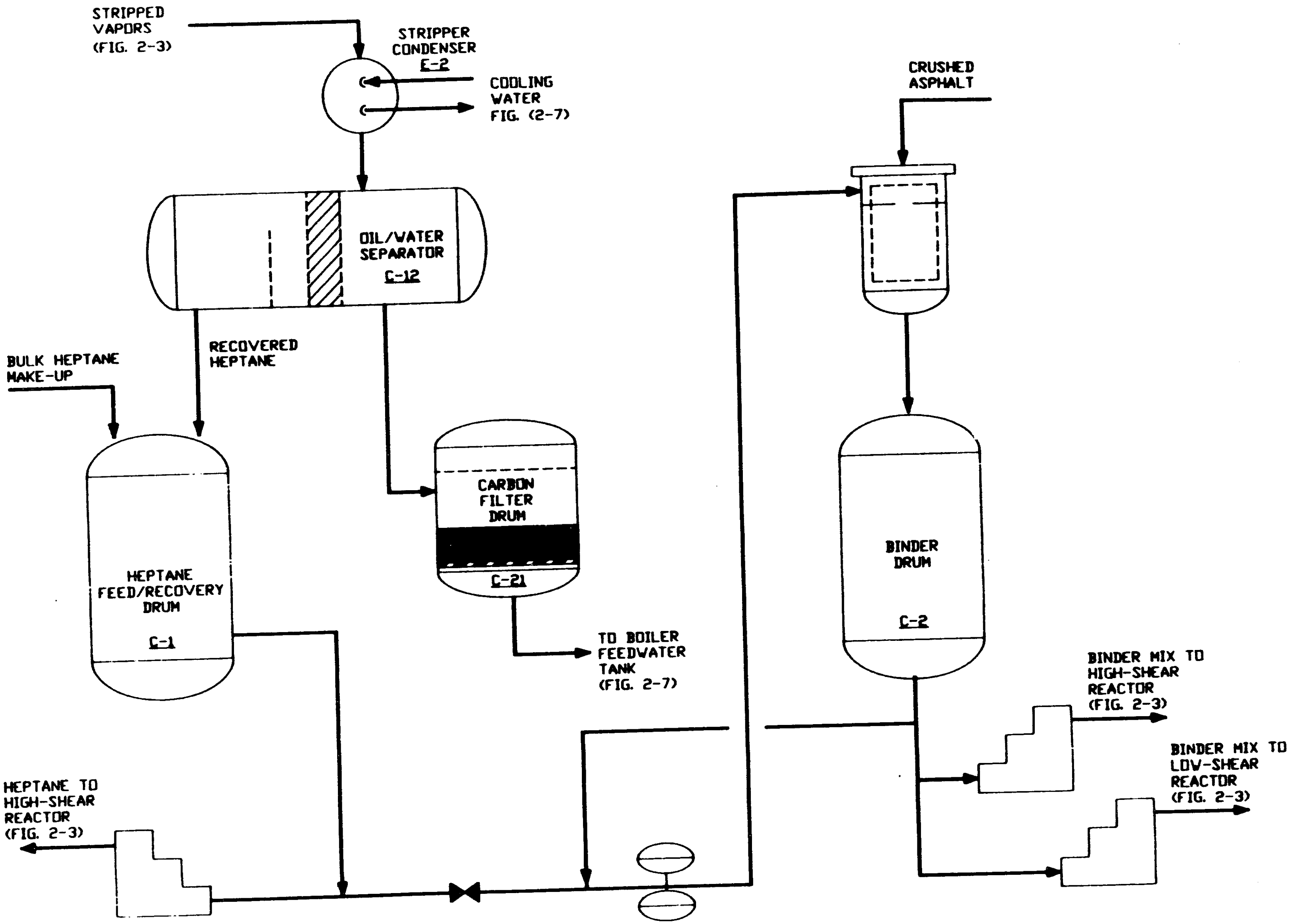


Figure 2-5 Heptane and Binder Handling Section Simplified Process Flow Diagram

gravity in the oil/water separator (C-12 in Figure 2-5). Heptane is returned to the heptane recovery drum (C-1). Recovered water is sent to the carbon filter drum (C-21) where traces of heptane, if any, are removed. Condensed water from C-21 is reused as boiler feed water.

After completion of the heptane stripping cycle, the overhead three-way valve is again lined up to the nitrogen blanketing system. The vessel is partially filled with quench water in order to cool the agglomerates. The bottom valve is opened, and the LSR content is discharged to the horizontal drainage belt (Y-1).

Water is used through sprays in the sides and bottom of the LSR vessel to assist discharge of the solids. The reactor mixer is also occasionally used at slow speed during the dumping step. The drainage belt dewaters the product coal agglomerates. Water sprays are employed to clean the belt. The drainage belt underflow is collected in the secondary tailings surge drum (D-3) and then pumped to the tailings filtering circuit.

The dewatered clean coal agglomerates are sampled and loaded into drums for shipment.

2.3.4 Heptane and Asphalt Handling

The heptane handling and asphalt binder preparation equipment is shown in Figure 2-5. It is located together in an area remote from the rest of the process area.

Heptane is delivered to the site on demand and transferred to the heptane feed/recovery drum (D-1). This drum holds the heptane requirement of the plant for use as the bridging liquid and as heptane/asphalt binder mixture. Heptane is added to the process by the heptane feed pump. The pump meters the heptane flow to the high-shear reactor (C-6).

To make a batch of binder mix, crushed asphalt is placed in a basket and lowered into the binder dissolution drum (C-16). The drum top is secured, and the vessel is purged with nitrogen to expel oxygen. A predetermined amount of heptane to provide the required binder/heptane concentration is added to the binder mix drum (C-2). This heptane is then circulated through the dissolution drum (C-16), and back to the binder drum (C-2) until all the asphalt is dissolved. A circulating pump is used to ensure that no asphalt solids settle out. The binder mix is fed to the process by metering pumps.

After completion of a batch of binder solution, the binder dissolution drum (C-16) is rinsed with heptane, isolated, and purged with nitrogen until no hydrocarbons are detected. The drum is then ready to be opened so that the next batch of binder can be made.

2.3.5 Tailings Filtration

The process has two tailings streams. The primary stream originates at the low-shear reactor (C-8) and the secondary stream at the drainage belt (Y-1). The drain and rinse water underflow from the belt is collected in the secondary tailings surge drum (D-3) shown in Figure 2-3. The slurry is sent to tailings sump (D-8) shown in Figure 2-6.

As shown in Figure 2-3, the primary tailings are collected in the tailings surge drum (C-5) where heptane-laden coal fines, if any, will float to be collected in the emergency slop tank (C-13). A small amount of nitrogen can be bubbled through the tailings surge drum (C-5) and a surfactant can be added to recover by flotation any heptane containing coal.

As shown in Figure 2-6, high-pressure piston pumps feed the tailings to filter press (Y-4), a plate-and-frame type filter. The dewatered filter cake is discharged directly into dumpsters. Another set of tailings sump (D-1) and filter press (Y-5) is provided to handle the overflow from the tailings sump (D-8).

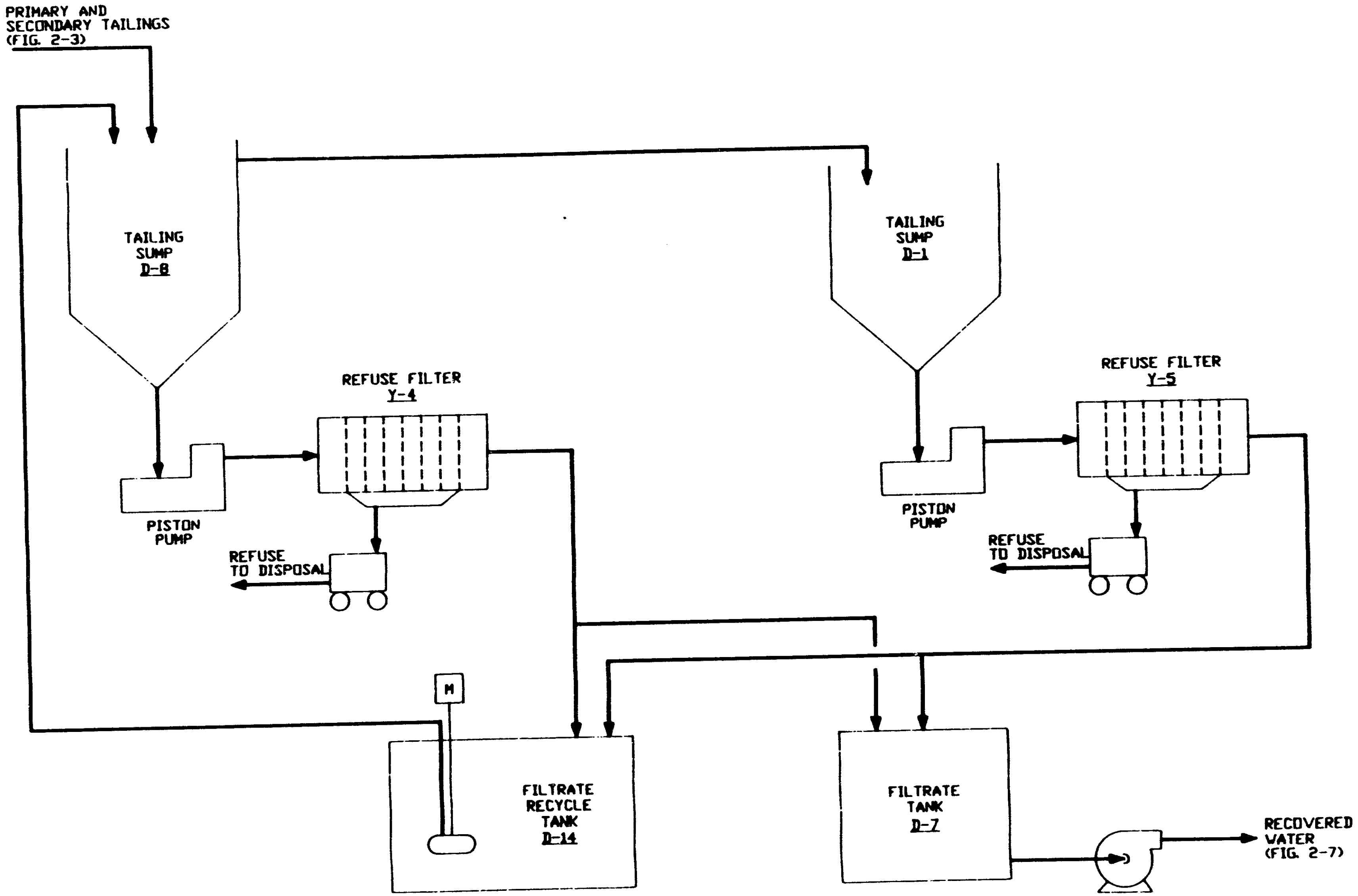


Figure 2-6 Tailing Dewatering Section Simplified Process Flow Diagram

By means of a three-way valve, the filtrate from either press can be diverted to either one of the two tanks (D-14 or D-7). If the filtrate contains excessive amounts of particulates, as during startup, it is sent to the filtrate recycle tank (D-14). The cloudy filtrate is returned via a pump to the tailings sump (D-8). Only clear filtrate is collected in the filtrate tank (D-7), and pumped to the water surge tank (D-4), as shown in Figure 2-7, for reuse.

2.3.6 Boiler Feed and Process Water System

Heptane-free condensate from the carbon filter drum (D-21 in Figure 2-5) is returned to the boiler feed water tank (D-15) as shown in Figure 2-7. Makeup boiler feed water comes from the CQDC advanced process building water softener.

Surge tank (D-4) provides water for coal grinding, spray washes, and displacement for the tailings surge drum (C-5), as well as flush water after steam stripping. If cooling is required, the recovered water is circulated through the flush water cooler (E-3).

2.3.7 Vapor Handling

A closed inert gas blanketing system is used to provide an oxygen-free atmosphere for all heptane handling systems and to prevent the uncontrolled escape of heptane vapors. The blanketing system is filled with nitrogen from a liquid nitrogen tank and maintained at pressure by bleeds from the liquid nitrogen tank. These facilities are illustrated in Figure 2-8.

Any vapor displacement in the system caused by temperature or liquid level changes is absorbed by a variable volume gas holder (D-10). The gas holder maintains the system at a positive pressure of 6 inches of water. The only gas that is normally vented from the system is surplus inventory caused by the small amount of nitrogen used for purging equipment during startups, shutdowns, and maintenance. Vented gas is sent to a flare (F-3) where any combustible vapors are burned.

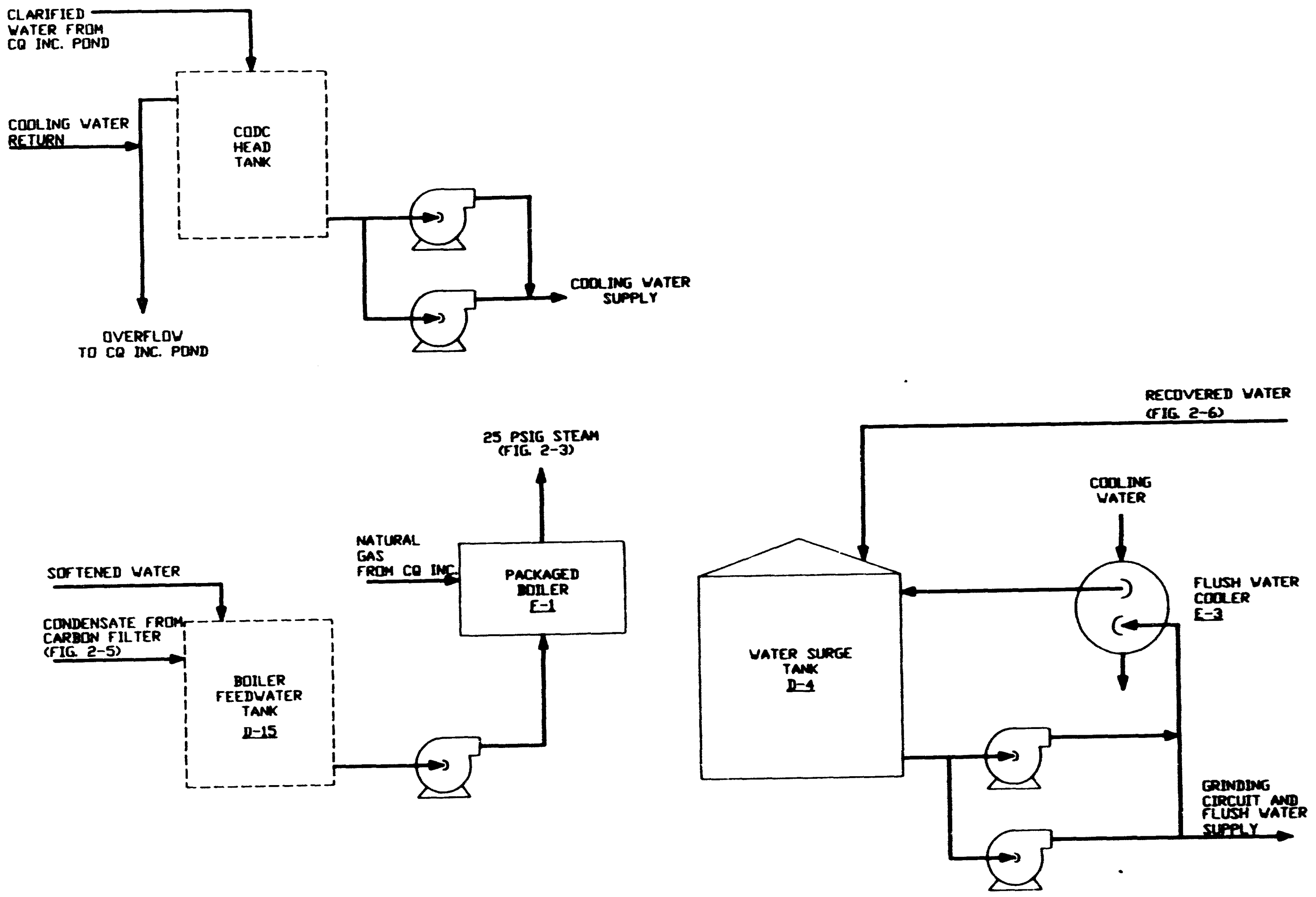


Figure 2-7 Recovered Water/Steam Simplified Process Flow Diagram

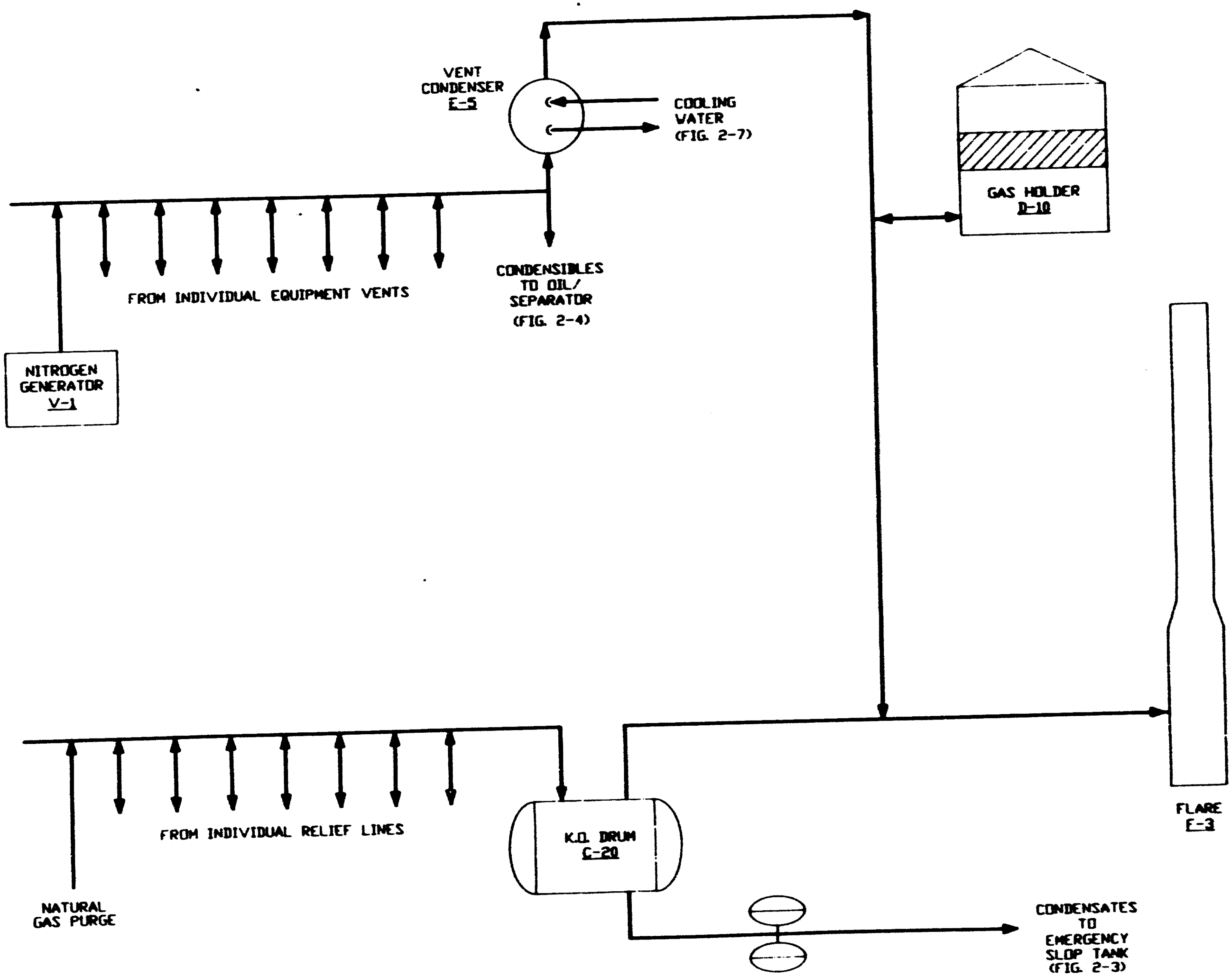


Figure 2-8 Gas Blanket and Relief System Simplified Process Flow Diagram

The closed vapor system has a condenser (E-5), which condenses and recovers the heptane vapors from the system. This minimizes losses of heptane in the normal venting operation and reduces the chance of heptane condensing in the gas holder. Condensate accumulating in the gas holder is collected and periodically sent to the oil/water separator (C-12).

Heptane-containing vessels are protected from overpressurization by a closed pressure relief system vented to the flare stack. Each heptane-containing vessel is provided with a spring-activated pressure relief valve which relieve the vessel vapors to the relief system in the event of overpressurization.

A relief knockout (K.O.) drum (G-20) upstream of the flare is provided to recover any condensibles present in the system. Collected liquid is then sent to the emergency slop tank (C-13).

In addition, hydrocarbon sensors are placed within the plant to detect heptane leaks. A positive hydrocarbon detection signal above a preset level that is below the lower explosive limit (LEL) triggers an alarm and directs the ventilation system to increase the rate of air changes in the building to dissipate the vapors. If the concentration of heptane vapor remains at alarm levels for a preset period of time, total plant shutdown procedures are initiated by the operators.

Section 3

PROJECT PLANNING, ENGINEERING, PROCUREMENT, INSTALLATION, AND SHAKEDOWN

3.1 PROJECT PLANNING

Immediately upon award of the contract in September 1987, Bechtel developed a project work plan and submitted it to DOE in October 1987. The work plan described the methods, control systems, and procedures to be used by Bechtel to perform and monitor the program activities. The plan, which was structured to reflect an integrated form of management, consisted of the following elements:

- Project management structure
- Management process
- Work breakdown structure
- Milestone schedule
- Cost plan
- Manpower plan
- Bench-scale test plan
- Engineering department procedures including quality assurance (QA) plan

The plan was used continuously during the program for monitoring the budget and schedule. DOE was kept informed through periodic reports.

3.2 ENGINEERING AND PROCUREMENT

Engineering and procurement activities were performed by Bechtel. Process and plant designs and equipment specifications were developed using bench-scale test findings and prior NRCC and Arcanum experience. Equipment procured under the earlier DOE Advanced Physical Fine Coal Cleaning Projects (Microbubble Flotation and Heavy Liquid Separation, Contract Nos. DE-AC22-85PC81205 and DE-

AC22-87PC79866, respectively) and available at the CQ Inc. site was incorporated into the designs to the maximum extent possible.

Details of the work performed and the engineering documents generated were reported in an interim report entitled "Advanced Fine Coal Cleaning-Spherical Agglomeration-Phase II Report," submitted to DOE in May 1989.

The report consisted of the following elements:

- o Plant description and operating procedures
- o Process flow diagram and material balance
- o Equipment list and specifications
- o Electrical system description
- o Description of instrumentation and controls, data acquisition, and logging systems
- o Utility requirements/anticipated usage

The following documents were appended to the report:

- o Appendix A - P&IDs and installation drawings
- o Appendix B - specifications and equipment data sheets
- o Appendix C - "what-if" safety review
- o Appendix E - permit applications and preparedness, prevention, and contingency plan (PPC)

The "what-if" review reflected a detailed scrutiny of the total design package and operating plans. It was performed by an independent, multidiscipline review team of experienced Bechtel engineers. Its purpose was to detect items that could cause serious hazard to operating personnel or others in the vicinity or cause serious damage to the process equipment or other facilities in the vicinity. The analysis was restricted to a review of safety matters from the design and operating points of view. The review recommendations were incorporated in the final designs and operating procedures.

The permit applications and PPC plan included applications that were required by the Commonwealth of Pennsylvania Department of

Environmental Resources, Bureau of Air Quality Control. Also included was the preparedness, prevention, and contingency plan (PPC) for the spherical agglomeration POC plant. The PCC constituted a safety plan for plant operations.

3.3 INSTALLATION AND SHAKEDOWN

Installation of the POC test unit began in June 1989. Lincoln Contracting & Equipment of Boswell, Pennsylvania performed the installation work under Bechtel's supervision.

Plant installation was completed by October 1989. At this time three technicians were provided by CQDC for the shakedown and operational phases of the program. Their first week under Bechtel's direction consisted of training and orientation.

Section 4

BENCH-SCALE TESTS

4.1 COAL SELECTION RATIONALE

This section summarizes the bench-scale tests which were performed to scale the agglomeration process to the proof-of-concept test module size. A more detailed bench-scale test report can be found in Appendix E, Bench-Scale Test Report. Detailed explanation of the test procedures can be found in Appendix F, Bench-Scale Test Procedures.

A review of several bituminous coals was performed early in the bench-scale test program to choose three coals for both bench-scale and POC testing levels.

The criteria used for the selection of coal seams were:

- Availability of extensive and extractable resources
- Importance of the coal seams as a present and future source of coal to the utility industry
- High ash and pyritic sulfur contents
- "Difficult to poor" cleanability to low ash/sulfur contents for conventional physical cleaning methods
- Amenability to cleaning by spherical agglomeration to low ash and sulfur levels with high energy recoveries

Coals with poor cleaning characteristics were included to establish practical limits to cleaning and achievable Btu recoveries at the test module.

Coal seams, naturally low in sulfur and/or ash, were precluded from the tests.

Based on the criteria listed, coals from the following sources were selected for bench-scale tests:

- Illinois No.6, Burning Star No.4 (Perry County, Illinois)
- Pittsburgh (Ohio No.8), North American Coal Co., No.6 (Belmont County, Ohio)
- Upper Freeport, Helen Mine (Indiana County, Pennsylvania)

4.2 SPHERICAL AGGLOMERATION TESTS

Batch and continuous mode agglomeration tests were conducted at Arcanum facilities in Ann Arbor, Michigan. The test results were reported in the Phase 1 Report dated October 1988. A brief summary is presented here.

Run-of-mine coals from the Pittsburgh, Illinois No.6 and Upper Freeport seams were precleaned at CQ Inc. in Homer City, Pennsylvania. The clean coal from the precleaning operation was ground and tested in the laboratory, using the spherical agglomeration process, first in a batch and later in a continuous mode. A total of 84 tests (including replicates) were completed in the batch mode; 21 tests were performed in the continuous mode.

4.2.1 Bench-Scale Test Objectives

The specific objectives for the bench-scale tests were:

- Evaluation of fine coal cleaning and pyrite rejection potential of the spherical agglomeration process
- Comparison of process performance with washability data generated for the feed coal at specific grind levels
- Screening of pyrite suppression additives and their dosages
- Evaluation of the grinding system at CQ Inc. (installed as part of the earlier DOE microbubble flotation project) for its effectiveness in ensuring required liberation of pyrite and ash-forming minerals
- Investigation of the "aging" effects of finely ground coals on agglomeration performance
- Determination of high shear reactor design and operating parameters

- Determination of appropriate feed pulp density
- Determination of low shear reactor design and operating parameters
- Identification of process variables and their testing ranges for the POC tests
- Verification of "steam stripping" of the final agglomerates and determination of process parameters for the operation
- Determination of residual heptane in the agglomerates after stripping
- Dewatering tests with the product using a screen

4.2.2 Summary of Bench-Scale Test Results

Analysis of the ROM coals and the precleaned coal (feed to agglomeration) are shown in Table 4-1. The table also includes performance indices for the precleaning operation.

Table 4-2 shows a summary of the spherical agglomeration tests results for all three coals. The significant process performance related findings, on a ROM coal basis, are as follows:

- Pyritic sulfur reduction on MMBtu basis ranged from 75 to 79 percent
- Energy recovery ranged from 85 to 92 percent
- Ash reduction on a MMBtu basis ranged from 77 to 94 percent

The data on a "ROM coal basis" reflect the combined performance using precleaning by conventional methods and spherical agglomeration.

Total sulfur dioxide reduction achieved for Pittsburgh and Illinois No. 6 seam coals at 57 and 42 percent, respectively, appears modest. This is due to the high organic sulfur content of these coals, equivalent to 3 pounds of sulfur dioxide per MMBtu. Organic sulfur in coal is not removable by physical coal cleaning methods. The agglomeration step itself showed near complete energy recovery. Pyrite reduction for both coals ranged from 70 to 87 percent.

Table 4-1

ROM COAL DATA AND PRECLEANING RESULTS

Seam **Mine** **County** **State**	**Pittsburgh** **(Ohio No.8)** **Powhatan No.6** **Belmont Ohio**	**Illinois No. 6** **Burning Star No. 4** **Perry Illinois**	**Upper Freeport** **Helen Indiana** **Pennsylvania**
Run-of-Mine Coal:			
Ash, %	39.16	15.71	57.31
Total sulfur, %	4.71	4.54	2.11
Pyritic sulfur, %	2.93	2.46	1.93
Heating value, Btu/lb	8,528	11,837	5,947
lb Ash/MMBtu	45.92	13.27	96.37
lb SO_2/MMBtu	11.05	7.67	7.10
lb Pyritic SO_2/MMBtu	6.87	4.16	6.49
Precleaned Coal to Agglomeration:			
Ash, %	12.10	9.60	16.30
Total sulfur, %	4.37	1.17	2.18
Pyritic sulfur, %	2.02	0.96	1.62
Heating value, Btu/lb	12,791	12,705	12,698
lb Ash/MMBtu	9.46	7.56	12.84
lb SO_2/MMBtu	6.83	4.99	3.43
lb Pyritic SO_2/MMBtu	3.16	1.51	2.55
Precleaning Refuse:			
Ash, %	84.49	39.90	88.68
Precleaning Results:			
Yield, %	62.6	79.8	43.3
Energy recovery, %	93.9	85.7	92.5
Ash reduction, % (1)	79.4	43.1	86.7
SO_2 reduction, %	38.1	34.9	51.6
Pyritic SO_2 reduction, %	54.0	63.6	60.7

(1) All reductions are on a constant energy (lb/MMBtu) basis

Table 4-2
BENCH-SCALE SPHERICAL AGGLOMERATION TEST RESULTS SUMMARY

	Ohio No. 8			Illinois No. 6			Upper Freeport		
	Batch Test Average (1) (Std Dev)	**Continuous Test Average (2) (Std Dev)**	**Batch Test Fine Grind (Typical)**	**Batch Test Average (3) (Std Dev)**	**Continuous Test Average (4) (Std Dev)**	**Batch Test Fine Grind (Typical)**	**Batch Test Average (5) (Std Dev)**	**Continuous Test Average (6) (Std Dev)**	**Batch Test Fine Grind (Typical)**
Grind Size (d_{50}):	12.1	12.1	3.7	9.8	9.8	3.7	4.2	4.2	2.9
Agglomerated Clean Coal:									
Ash, %	5.01 (0.19)	4.81 (0.40)	3.35	3.74 (0.28)	4.23 (0.11)	2.90	8.17 (0.89)	8.04 (0.26)	8.51
Total sulfur, %	3.78 (0.11)	3.38 (0.14)	3.48	2.72 (0.09)	3.05 (0.07)	2.56	1.58 (0.05)	1.58 (0.06)	1.40
Pyritic sulfur, %	1.41	1.06	0.86	0.39	0.71	0.36	0.87	0.85	0.38
Heating value, Btu/lb	13,877	13,908	14,133	13,617	13,541	13,748	14,058	14,080	14,002
lb Ash/MMBtu	3.61	3.46	2.37	2.75	3.12	2.11	5.81	5.71	6.08
lb SO_2/MMBtu	5.45	4.86	4.92	4.00	4.50	3.72	2.25	2.24	2.00
lb Pyritic SO_2/MMBtu	2.03	1.52	1.22	0.57	1.05	0.52	1.24	1.21	0.54
Precleaning Refuse:									
Ash, %	81.89	85.70	89.60	86.47	85.70	89.60	89.17	86.83	92.40
Bench-Scale Performance									
Yield, %	90.8	91.0	89.9	92.9	93.4	92.3	90.0	89.5	90.7
Energy recovery, %	98.5	98.9	99.3	99.6	99.6	99.8	99.6	99.3	99.9
Ash reduction, % (7)	61.8	63.4	74.9	63.7	58.7	72.1	54.7	55.5	52.7
SO_2 reduction, %	20.2	28.8	27.9	19.9	9.7	25.4	34.5	34.6	41.7
Pyritic SO_2 reduction, %	35.7	51.8	61.5	62.1	30.6	65.3	51.5	52.7	78.7
ROM Performance:									
Energy recovery, %	92.5	92.9	93.2	85.3	85.3	85.6	92.1	91.8	92.4
Ash reduction, %	92.1	92.5	94.8	79.3	76.5	84.1	94.0	94.1	93.7
SO_2 reduction, %	50.7	56.0	55.4	47.9	41.3	51.4	68.3	68.4	71.8
Pyritic SO_2 reduction, %	70.4	77.8	82.3	86.2	74.8	87.4	80.9	81.4	91.6

(1) Average of 23 tests
(2) Average of 8 tests
(3) Average of 18 tests
(4) Average of 6 tests
(5) Average of 12 tests
(6) Average of 7 tests
(7) All reductions are on a constant energy (lb/Btu) basis

The ash content of the clean coal ranged between 3.7 and 4.0 percent for Illinois No. 6 coal. With Pittsburgh coal, clean coal ash contents between 4.7 to 5.0 percent were achieved. The clean coal from the Upper Freeport seam coal, as expected, had a relatively high ash content of 8.0 percent (mean value for all tests). Ash in this coal was very finely disseminated: even grinding to 3 microns (50 percent passing) could not achieve significant liberation.

High ash refuse products, with ash contents in the range of 80 to 90 percent, were produced during all of the tests, indicating a high carbon (Btu) recovery.

Other significant findings from the tests are as follows:

- None of the pyrite depressants was found to be effective
- Aging of coal did not significantly affect process performance. Some of the samples tested were over 6 months old
- Grinding to liberate minerals was the most important variable for improving coal quality
- For Pittsburgh No. 8 and Illinois No.6 coals, a comparison of process performance of particles ground to 50 percent passing 10 microns and 50 percent passing 4 microns indicated that finer grinding did not significantly lower the product sulfur content. The improvement was in the range of 0.1 to 0.2 percent. A similar finding was noted with Upper Freeport seam coal which was tested at two grind levels of 4.3 and 2.9 microns (50 percent passing)
- A solid concentration of 15 percent (by weight) was most suitable for the grinds tested (50 weight percent passing 3 to 12 microns).
- Use of steam for stripping was effective and safe. Data on steam flow rate and quantity was determined. Steam stripping yielded a product with residual heptane content of 6 ppm as determined by gas chromatography. This heptane content is safe and acceptable by EPA standards.

The batch and continuous tests also helped fine-tune various aspects of the POC plant design. For example, based on the experiments, a special belt type filter was selected for dewatering the finished product. The agglomerates required gentle handling to prevent loss of fine coal with the secondary tailings during dewatering.

The tests also helped formulate POC test plant operating procedures and the test and sampling plans, particularly in the areas of benchmark process parameters, process variables to be tested, and their testing levels.

The petrographic analysis and agglomeration tests with the samples of ground coal received from the fine grinding circuit at CQ Inc. (installed as part of the earlier microbubble flotation project), indicated that the grinding system needed improvements to achieve better liberation of ash-forming minerals and pyrite. This led to the concept of selective grinding. The concept was verified on a pilot scale, as discussed below.

4.3 SELECTIVE GRINDING TESTS

Bench-scale tests verified the importance of effective liberation, or grinding, in achieving significant reductions of ash and pyritic sulfur. A selective grinding system was proposed to improve the process performance, and the bench-scale test program was modified to include its testing for scale-up to the 1 tph level.

4.3.1 Background

Performance and economics of physical coal-cleaning processes are strongly dependent upon the liberation of the impurities that have to be separated out from coal, namely, ash-forming minerals and pyrites. A high degree of pyrite rejection in the cleaning step, for example, depends on the effectiveness of the grinding system in transforming all coal-encased or -attached pyrite into discrete, coal-free particles. Coal-encased or coal-attached pyrite will be collected with the clean coal.

In theory, complete liberation can be achieved if the feed coal is ground to sub-micron levels. However, such grinding is neither practical nor economical.

4.3.2 Prior Experience in Fine Grinding

Bechtel participated in an earlier DOE-sponsored Advanced Physical Fine Coal Cleaning Project using microbubble flotation technology. During this program the coal feed to the process was ground to 10 microns (50 percent passing). The coal (1/4 inch x 0) was ground first in a wet ball mill to approximately 80 percent passing 100 mesh. The product from the ball mill was then passed through an attrition mill (bead mill) to obtain the flotation feed size. Size analysis, centrifugal float/sink, and petrographic analysis of the ground feed coal and the concentrates from flotation indicated the following:

- Even though the ground feed coal had the desired mean particle size of about 10 microns, there was a significant amount of material as large as 150 microns.
- The grinding system preferentially ground the soft low-ash components to extremely fine sizes, in the 3-4 micron range.
- Difficult-to-grind ash- and pyrite-rich components of the feed coal remained coarse, resulting in poor liberation.

The particles in the ground product exhibited a wide size range, from sub-micron to 150 microns, even though the mean size was within the desired range of 10 microns.

These observations led to a search for methods to improve liberation in the grinding operation, by avoiding overgrinding of the low-ash components and at the same time adequately grinding and thus liberating the high-ash and -pyrite coal particles. It was found that if a size classification step was introduced in the grinding system, it would permit repeated grinding of the hard, coarse particles until they reached the required final size. Also, by diverting soft particles of the desired size as soon as they were formed to the product stream, overgrinding could be eliminated. The particles in the ground product would then exhibit a narrow size spread in addition to better liberation. A closed-circuit selective grinding system could offer additional advantages of lower specific power consumption and higher capacities for the existing grinding equipment. The product size distribution, namely narrow and without excessive very fine particles, of slurry produced by selective grinding could also lead to easier product dewatering and reduced use of bridging liquid and binder for the spherical agglomeration process.

For classification at the 5-20 micron range required for selective grinding, none of the conventional sizing equipment used in the coal preparation industry such as screens, cyclones, spiral classifiers, and settling tanks were found suitable. After investigation Bechtel proposed using a solid-bowl centrifuge similar as applied for desliming in the kaolin and clay industries. Use of solid-bowl centrifuges in the coal industry has been limited to dewatering applications with maximum solids recovery.

4.3.3 Selective Grinding Tests

To verify the application of a solid-bowl centrifuge for the classification duty, a limited number of tests were conducted using the Bird Machine Company's pilot testing unit. This unit has a capacity of 10 gpm as compared to the 50 gpm required for the 1 ton-per-hour POC plant.

The tests were conducted in October 1988 at AMAX's grinding pilot plant in Golden, Colorado. The flow sheet used for the test, Figure 4-1, simulated the proposed POC plant grinding facilities. A ball mill was used for primary grinding and a bead (attrition) mill was used for the secondary. The solid bowl centrifuge was placed between the ball mill and the bead mill to classify the product from both mills. The coarse product, the cake, from the centrifuge was

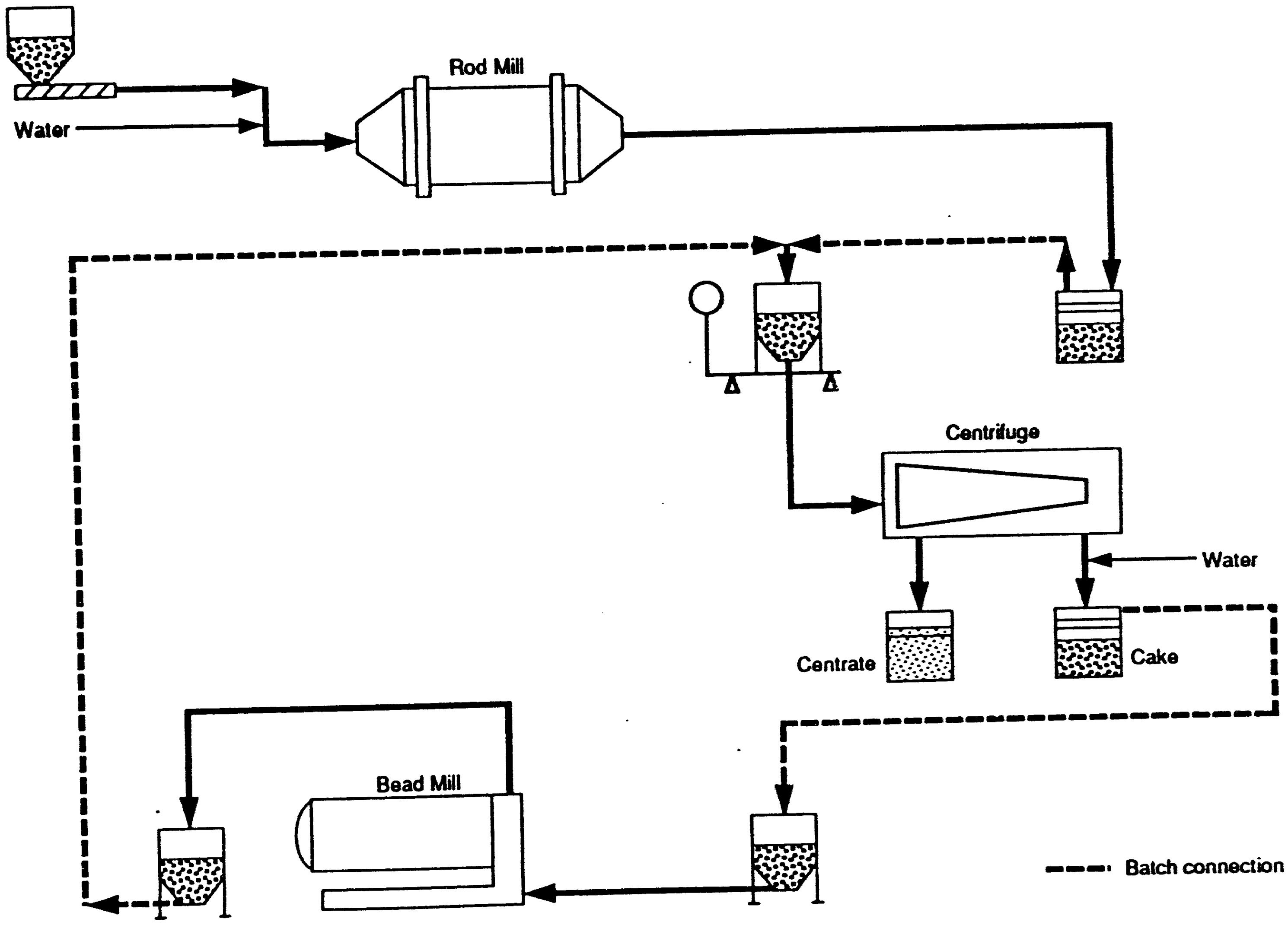


Figure 4-1 AMAX Grinding Flow Sheet

fed to the bead mill after dilution. The fine product (centrate from centrifuge) constituted the finished ground product.

For the tests only a limited quantity of feed (600 lb) was available; facilities to continuously operate the entire equipment train, namely of the ball mill, centrifuge, and the bead mill, were also limited. To simulate a continuous operation, each operation (ball milling, centrifuging, and bead mill grinding) was performed separately and the product collected over a period of time until adequate amounts were available as feed to the next unit in line. Since the selective grinding idea incorporates a recirculating stream, the operation was repeated to note the trends as the system reaches a steady state.

4.3.4 Selective Grinding Test Results

Analysis of laboratory data for the samples of the feed, the final product, and the feed to the centrifuge and attrition mill confirmed expectations. Complete details of the test equipment, procedures, laboratory analysis, and evaluation of the results were submitted in a report entitled "Selective Grinding Tests," dated November 1988.

The significant test findings are:

- The solid-bowl centrifuge was capable of satisfactorily classifying coal slurry in the 10-20 micron range.
- Harder, difficult to grind, high-ash and high-pyrite coal particles tended to concentrate in the centrifuge cake and thus could be reground repeatedly until they reached the required size.
- Over-grinding of low-ash components was avoided.
- The finish ground slurry exhibited a narrow size range, namely, 50 percent passing 10 microns and 90 percent passing 24 microns.
- The system could be operated to give a finished ground slurry with a solids concentration of 15 percent required for the agglomeration.

It was decided to incorporate the selective-grinding concept into the existing grinding system for the POC tests. A solid-bowl centrifuge was rented from Bird Machine Co. An additional sump and a pump for feeding the bead mill were ordered and installed.

Section 5

TEST AND SAMPLING PLAN

5.1 GENERAL

A test and sampling plan for the POC tests was prepared and was included in the report titled "Advanced Fine Coal Cleaning - Spherical Agglomeration - Phase II Report," dated May 1989.

Tests were planned for a 4-month period using the three coals selected during the bench-scale test program. The test matrix for each of the coals envisaged 39 tests with selected plant operating variables. All run-of-mine coals contained significant amounts of mine dilutions which could be economically eliminated by conventional coal cleaning technology. It was initially planned that the feed stock for the advanced technology POC plant would consist of coals precleaned at the CQ Inc. facilities.

The test and sampling plans also provided the following:

- POC test matrix and specifications for each test
- Listings of streams to be sampled
- Sample preparation and analytical requirements by test
- Process performance evaluation methodology
- Methodology for the preparation of material balance for each test run
- Test schedule
- ROM coal requirements
- Operator training

5.2 POC TEST MATRIX

A test matrix is an important element of any experimental plan and is developed to study systematically or quantify if possible, the effect of significant parameters (variables) on process performance (effects).

The first step in the development of a test matrix would be to identify the significant process variables that affect the performance indices. In coal cleaning, the relevant performance indices would be clean coal quality (sulfur, ash levels) and energy recovery.

The second step would be to select a range of meaningful values (levels) for each variable at which tests would be conducted.

The bench-scale tests evaluated effects of process parameters on the performance of the spherical agglomeration process. It was established during these tests that given the required amount of bridging liquid and an effective transfer of energy during the high and low shear mixing steps, the feed slurries of all the tested coals could be agglomerated satisfactorily into low ash/sulfur clean coal. The mineral matter in the feed stayed dispersed in the water as fine discrete particles. So long as the coal could be agglomerated, product quality and energy recoveries were largely the same over a wide range of values for the process variables tested. The single exception to this observation was the level of liberation achieved in the grinding step before agglomeration.

In addition to intensity of grinding, liberation is also dependent on the nature of the coal and the manner in which impurities are dispersed in the coal mass. When impurities are distributed throughout the coal in the form of extremely fine particles (say 3 microns), the entire coal has to be ground to sizes far below 3 microns for complete liberation. Limitations in grinding capability and exponential rise in energy and equipment costs limit the degree to which coal can be ground.

For a given grinding plant the amount of fine particles that could be achieved at a fixed throughput capacity depends on the hardness (grindability) of the coal. For example, the grinding system of the POC plant could produce a product with 50 percent passing 8 microns at 1 tph with either Pittsburgh or Illinois No. 6 coal. With softer Upper Freeport coal the system could produce a significantly finer product with 50 percent passing 4 microns at the same throughput rate. At the same time, in-spite of finer grinding, liberation was poorer with the Upper Freeport coal as compared to the other two coals. This is reflected in the high ash content (about 8 percent) of the Upper Freeport clean coal compared to 3 to 4 percent ash for the clean Illinois and Pittsburgh coals. The poor liberation with Upper Freeport coal is due to its impurities being dispersed in the coal mass as very fine particles.

The effectiveness of the grinding system was improved by using the selective grinding concept as discussed in Section 4.3.

As no major performance related effects were noted for the most candidate variables, the matrix was designed largely to address issues connected with scale-up of laboratory size operation to larger plants. The test matrix addressed issues such as size of the agglomerates, the appropriate amounts of bridging liquid and binder needs, input power requirements for agglomeration, losses of agglomerated coal with the tailings, the effectiveness of the steam stripping operation, and the operability of the plant.

Tables 5-1 and 5-2 show the test matrices that were formulated for the original test and sampling plan. Three coals were planned for testing.

A revised sampling plan was later issued reflecting a reduced effort testing program. Instead of testing coals from three seams over a period of 4 months, the program had to be changed to one coal and the testing period shortened to 6 weeks. The Illinois No. 6 coal was selected for testing.

Later, an extension to the operational phase of the project was granted during the testing of the Illinois coal. The extension also provided for the testing of the Pittsburgh and Upper Freeport coals.

Table 5-1

PITTSBURGH SEAM COAL
POC TEST MATRIX

Test No.	Feed Rate(a)	Particle Size(b)	Solid Conc.(c)	Heptane in HSR(d)	Asphalt Dosage(e)	Impeller Type (HS)(f)	Impeller Speed (HS)(g)	Impeller Type (LS)(h)	Remarks
High and Low Shear Reactor Tests									
P-01	2000	Fine	15	25	4	A	Low	A	
P-02	2000	Fine	15	25	4	A	High	B	
P-03	2000	Fine	15	25	4	B	Low	B	
P-04	2000	Fine	15	25	4	B	High	A	
P-05	2000	Fine	15	25	4	Selected	Selected	Selected	Replicate
Asphalt Tests									
P-06	2000	Fine	15	25	3	Selected	Selected	Selected	
P-07	2000	Fine	15	25	5	Selected	Selected	Selected	
P-08	2000	Fine	15	25	Selected	Selected	Selected	Selected	Replicate*
Heptane Tests									
P-09	2000	Fine	15	>25	Selected	Selected	Selected	Selected	Selected
P-10	2000	Fine	15	<25	Selected	Selected	Selected	Selected	Selected
P-11	2000	Fine	15	Selected	Selected	Selected	Selected	Selected	Selected

* If 3 or 5 percent asphalt is selected.

Table 5-1 (Cont'd)

Test No.	Feed Rate[a]	Particle Size[b]	Solid Conc.[c]	Heptane to HSR[d]	Asphalt Dosage[e]	Impeller Type (HS)[f]	Impeller Speed (HS)[g]	Impeller Type (LS)[h]	Remarks
Grind Size Tests									
P-12	2000	Medium	15	Selected	Selected	Selected	Selected	Selected	
P-13	2000	Medium	15	Selected	Selected	Selected	Selected	Selected	Replicate
P-14	2000	Coarse	15	Selected	Selected	Selected	Selected	Selected	
P-15	2000	Coarse	15	Selected	Selected	Selected	Selected	Selected	Replicate

(a) Feed rate in lb/hour of as-received coal. Use coal analysis to calculate dry basis feed.

(b) Fine grind refers to product from the grinding system using the solid bowl centrifuge for classification. Expected grind is 50 percent (by weight) passing 7 microns. The size may differ based on actual operation characteristics of the grinding system. Medium particle size refers to two-stage circuit grinding without the solid bowl centrifuge to produce a product coarser than the fine grind, with a size approximately 50 percent passing 11 microns. Coarse grind refers to product from the ball mill when neither the solid bowl centrifuge nor the fine grinding mill is used. In such an event a particle size of 50 percent passing 100 microns is expected.

(c) Solid concentration refers to percent solids by weight in the feed to the high shear reactor (HSR).

(d) Heptane to HSR, percent by weight of dry coal feed to the reactor. Tests at two additional levels are planned, one lower and the other higher than 25 percent.

(e) Asphalt dosages are given as weight percent of dry feed coal to the HSR. The values are derived from bench-scale tests. Additional tests at two levels, one lower and one higher, are planned.

(f),(g),(h) The first four tests use two types of impellers for the HSR and low shear reactor, and two speeds for the HSR.

Table 5-2

ILLINOIS NO. 6 AND UPPER FREEPORT SEAM COALS POC TEST MATRIX

Test No.	Feed Rate$^{(a)}$	Particle Size$^{(b)}$	Solid Conc.$^{(c)}$	Heptane to HSR$^{(d)}$	Asphalt Dosage$^{(e)}$	Impeller Type (HS)$^{(f)}$	Impeller Speed (HS)$^{(g)}$	Impeller Type (LS)$^{(h)}$	Remarks
High and Low Shear Reactor Tests									
I/U-01	2000	Fine	15	M	4	Selected	Selected	Selected	Setup test
I/U-02	2000	Fine	15	M	4	Selected	Selected	Selected	Setup test
Asphalt Tests									
I/U-03	2000	Fine	15	M	5	Selected	Selected	Selected	
I/U-04	2000	Fine	15	M	3	Selected	Selected	Selected	
I/U-05	2000	Fine	15	M	Selected	Selected	Selected	Selected	Replicate
Heptane Tests									
I/U-06	2000	Fine	15	-M	Selected	Selected	Selected	Selected	
I/U-07	2000	Fine	15	+M	Selected	Selected	Selected	Selected	
I/U-08	2000	Fine	15	Selected	Selected	Selected	Selected	Selected	Replicate
Grind Size Tests									
I/U-09	2000	Medium	15	Selected	Selected	Selected	Selected	Selected	
I/U-10	2000	Medium	15	Selected	Selected	Selected	Selected	Selected	Replicate
I/U-11	2000	Coarse	15	Selected	Selected	Selected	Selected	Selected	
I/U-12	2000	Coarse	15	Selected	Selected	Selected	Selected	Selected	Replicate

(a) Feed rate in lb/hour of as-received coal. Use feed coal analysis to calculate dry basis feed.

The test matrices for both additional coals were modified as a result of experience gained with testing the Illinois coal and are presented together as Table 5-3. Major changes included the addition of solids concentration as a variable and the testing of different coal feed preparation methods.

During the execution of the test matrix ton quantities of clean agglomerate were produced. The agglomerates were loaded into drums, inerted, and then shipped to other DOE test programs. The bulk of this coal was to be evaluated in a combustion test program. Tests run on the agglomerates by other DOE programs included liquefaction handling, grinding, and pneumatic transport.

Table 5-3

PITTSBURGH AND UPPER FREEPORT SEAM COALS
POC TEST MATRIX

Test No.	Grind Type	Solids Conc.	B.L. Conc.	Asphalt Conc.	Impeller Speed	Hi-Shear Res. Time	Remarks
1A	Selective	15	High	Medium	High	High	
1B	Selective	15	Medium	High	High	Medium	
2A	Sel w/Spiral	15	Medium	Low	High	Medium	
2B	Sel w/Spiral	15	Low	Medium	High	Low	
3A	2-Stage	15	Medium	Low	High	Medium	
3B	2-Stage	15	Low	Medium	High	Low	
4A	1-Stage	15	Medium	Low	High	Medium	
4B	1-Stage	15	Low	Medium	High	Low	
5A	Sel w/Spiral	15	Medium	Low	High	Medium	
5B	Sel w/Spiral	15	Low	Low	High	Low	
6A	Best	18	Medium	Best	High	Low	
6B	Best	18	Low	Best	High	Low	
7A	Best	15	Medium	Best	Variable	Variable	Constant Work Tests
7B	Best	15	Medium	Best	Variable	Variable	Constant Work Tests
8A	Best	20	Best	Best	Best	Best	
8B	Best	20	Best	Best	Best	Best	
9A	Best	Best	Best	Best	Best	Best	
9B	Best	Best	Best	Best	Best	Best	

Section 6

PROCESS OPERATION

6.1 OPERATION OVERVIEW

The POC plant operation began with the grinding circuit. Shakedown of the grinding circuit continued until the agglomeration circuit construction was completed. The first agglomerates were produced 1 month later and operations continued for 5 months. During that time coals from the Illinois No. 6, Upper Freeport, and Pittsburgh seams were tested. An additional coal from the Taggart seam located in Wise County, Virginia was also briefly tested.

In addition to producing test results from which the agglomeration process could be evaluated, bulk quantities of coal from each seam were produced for other DOE test programs. The total production of agglomerates (dry basis) during the POC operations were:

Coal Seam	Tons
Illinois No. 6	17.8
Upper Freeport	15.4
Pittsburgh	12.3
Taggart	1.4
Total	46.9

Other DOE test programs will evaluate combustion characteristics, handleability, liquefaction potential, and other characteristics of the clean coal.

6.2 REVIEW OF TEST MODULE PERFORMANCE

6.2.1 Heptane Recovery

The spherical agglomeration process economics depend to a large extent on the recovery of the heptane bridging liquid. The POC plant was designed to recover heptane. However, the nature of the batch design and test operation allowed heptane losses which would be unacceptable for a continuous operating plant. During the course of POC operation 8,440 lb of heptane was used to produce 46.9 tons of agglomerates. With heptane addition of approximately 30 percent to coal by weight, the heptane recovery from agglomerates would appear to be only 70 percent. In reality the major losses were through the gas blanket system. This system was designed to keep an inert blanket of nitrogen gas over each vessel containing heptane. A positive pressure of 6 inches of water was kept in the system to ensure that no oxygen would leak into the system. Even though a gas holder was used, occasionally nitrogen was bled gas from the gas blanket through the flare. Losses of heptane due to saturation in nitrogen which was bled out of the system could account for over 20 times the amount actually lost during the operation of the POC plant.

Some heptane losses were directly attributable to process upsets. Such occurrences were the result of heptane-laden microagglomerates being inadvertently pumped into the gas blanket system from the high-shear reactor. The microagglomerates in all cases had to be drained out of the system and spread out for air drying. A total of three upsets accounted for 300 lb of lost heptane or 3.6 percent of the total heptane lost in the POC plant.

The agglomerates and the primary tailings waste streams were tested for residual amounts of heptane after steam stripping. The results indicated that less than 0.2 percent of heptane was left in the coal or tailings after steaming. Agglomerates and tailings did not smell of heptane (odor threshold of 200 ppm) while incomplete test run produced products smelled of heptane, an indication of incomplete steaming.

It is apparent that a commercial operation will have to provide systems and operating procedures to recover the heptane more efficiently.

6.2.2 Water Recovery

The POC plant was designed with a closed water system. This system was separated from the rest of the CQ Inc. systems since the host site did not want to run the risk of contaminating the water quality for either the POC plant or their own processes.

The system used filter presses to recover solids from the POC plant refuse and grinding circuit overflows for disposal. Clear effluent was recirculated to a 10,000 gallon tank for use by the POC plant. Provisions were made to recirculate cloudy effluent back through the filter presses until it became clear.

The operation of this system was highly dependent on the type of coal used. Of the three coals tested, the Upper Freeport coal reject was the hardest to filter. The Pittsburgh and Illinois No. 6 rejects could be filtered with a clear effluent produced most of the time.

The Upper Freeport grinding circuit product was very difficult to filter as the very fine coal could not easily be recovered from the effluent without the use of flocculants. The Upper Freeport tailings presented another challenge as the +86 percent ash stream contained a large amount of clays which would quickly blind the filtering media of the filter press. Filtering these clays required a first coat of ground coal ball mill product - 100M x 0 coal.

Filtering a large amount of grinding circuit product for all coals was required since it took an hour or more to achieve steady state. During this startup phase all of the grinding circuit product was diverted to the filter press.

6.2.3 Steam Stripping Operation

Combining the steam stripping of heptane in the low-shear reactor represented a compromise between cost and function. Two months of operating time was spent with the pelletizing and screening steps learning about the stripping operation before heptane-free and firm agglomerates were produced.

The steaming operation, as designed, called for the addition of 3,000 lb/hour of 25 psig steam (100 percent quality) to be sparged directly into the bed of 3/8 inch size agglomerates. Since the system did not initially have a provision to control the steam flow rate as it entered the low-shear reactor batches of coal dust were produced instead of pellets. The destruction of the pellets was due to two factors: (1) the mechanical agitation caused by the steam sparging resulted in the "boiling" of the agglomerates and since this boiling action was limited to the vicinity of spargers the bed was unevenly heated; (2) the direct addition of steam into the drained bed of agglomerates resulted in a very rapid rise in temperature (initial temperature rate of increase of over 100°F/min). This rapid temperature rise caused the heptane in the agglomerates to be immediately vaporized. The result was an audible "pop" as the agglomerates were destroyed due to the immediate release of heptane vapor from the agglomerates.

During the initial test runs, agglomerates without excessive breakage could be produced only when steam flow rate was manually controlled at less than 600 lb/hr. At this rate, the steam stripping required over 2 hours.

A method was developed to steam the agglomerates in a bed of water with the result of reduced steaming time and retained integrity of the agglomerates. After draining the tailings from the agglomerates water was added back to just cover the agglomerates. This moderated the rate of temperature increase of the agglomerates, dampened the mechanical agitation of the steam, and provided a means for better heat transfer and more uniform agglomerate heating.

6.2.4 Binder Preparation and Use

The asphalt binder was dissolved in heptane and delivered to the microagglomerates in the low-shear reactor. This method of delivering asphalt in a liquid form with a metering pump enabled precise control of the dosage which was vital for successful agglomeration and heptane stripping.

However, the system had several drawbacks and was difficult to operate. Commercial designs will be able to avoid these shortcomings. For example, the binder solution was prepared by placing a weighed amount of crushed asphalt into the binder dissolution drum and submerging it in a measured amount of heptane. There were no instruments to show when the dissolution of asphalt into heptane was complete. Also, thin, uninsulated binder delivery piping (1/2 inch) plugged occasionally.

6.3 EVALUATION OF HIGH-SHEAR REACTOR PERFORMANCE

As part of the program, tests were performed on the high-shear reactor to confirm the vessel scale up from bench-scale and characterize its performance relative to the three feed coals.

The high-shear reactor (HSR) was scaled to produce a nominal 1 tph of Pittsburgh coal microagglomerates (35 gpm, 1 minute residence time, slurry at 15 percent solids, 20 percent ash, specific gravity 1.05) This design criterion recognizes that the three test coals varied dramatically during bench-scale testing with respect to their ease of microagglomeration - Pittsburgh coal falling in between the easy Upper Freeport coal and the very difficult Illinois No. 6 coal. The use of the intermediate rather than the most difficult coal as the design basis resulted in a lower throughput for the Illinois coal. Use of Illinois coal as the design basis would have resulted in a huge excess capacity when Upper Freeport coal was treated. Such excess capacity would, however, be useless due to capacity limitations downstream of the HSR.

6.3.1 Performance of Illinois No. 6 Coal

As expected, POC plant microagglomeration of Illinois No. 6 coal required the operation of the high-shear reactor at 100 percent rated turbine speed and at well below 1 tph. It was found by laboratory testing that the coal was less amenable to agglomeration than the material supplied for earlier bench-scale testing.

On the positive side, no difficulties were encountered in high-shear operation as feed solids concentrations were increased to approximately 20 percent, the upper limit which the grinding circuit could produce.

In spite of the increased solids concentration, but due to the higher residence time a throughput of only 0.5 tph was achieved in the high-shear reactor for Illinois No. 6 coal. As the high-shear reactor drive motor was not being loaded to capacity and as there was still some leeway in the impeller design to have increased power dissipation via impeller modification, approximately 50 percent additional throughput could likely have been achieved with the same motor and vessel configuration had the impeller been optimized for this coal.

6.3.2 Performance of Upper Freeport Coal

Of the coals used in both the bench-scale and POC plant, Upper Freeport seam coal was the easiest to microagglomerate. At 100 percent turbine speed, finely ground Freeport coal could readily be agglomerated with residence times of 50 seconds or less. Tests could not be conducted at lower residence times as the bridging liquid feed pump was run at 100 percent capacity at the 50 second level. Alternately, by increasing residence time the Freeport coal would successfully microagglomerate at turbine speeds as low as two-thirds of full rated speed. As with the Illinois coal, no difficulties were encountered at solids concentrations up to 20 percent. Thus the high-shear reactor was capable of processing 1.75 tph or more of Upper Freeport coal.

There was also some indication that ash and sulfur rejection were improved by decreasing residence time. The effect was not large, however, and a sufficient number of tests was not run to determine whether the effect was statistically significant.

6.3.3 Performance of Pittsburgh Coal

Pittsburgh seam coal could be microagglomerated in the high-shear reactor in 75 seconds residence time with 90 percent of full-rated turbine speed. No problems were encountered at solids content of up to 20 percent, leading to a throughput of 1.25 tph.

6.3.4 Low-Shear Growth Performance

In contrast to the high-shear microagglomeration process, which in addition to heptane dosage, residence time, and turbine speed is highly dependent on the feed coal properties (including particle size), the agglomerate growth in the low shear process is primarily dependent on the presence of good microagglomerates and the correct dosage of binder mix. Under such conditions, regardless of the feed coal type or its particle size, agglomerate growth to 6 mm could be easily achieved in 5 to 10 minutes.

6.3.5 Design Implications

The tests showed that cost and design criteria for the high-shear reactor is highly feedstock dependent. For the three coals the high-shear reactor capacity varied through a ratio of at least 3.5 to 1. However, the excellent agreement between the scale up predictions and actual performance of the high-shear system indicate that for a given coal, a system can be designed with a high degree of confidence using data generated by continuous bench-scale testing.

6.4 REVIEW OF CONTROLS AND INSTRUMENTATION

The objective of the instrumentation and control systems was to measure and record different functions of the coal grinding and agglomeration units, control the batch operation, notify operators of abnormal operating conditions, and react to fires and/or high hydrocarbon levels in the advanced process building. The design kept safety systems (fire, ventilation, and alarm) separate from the agglomeration unit control and data acquisition system. It was important to the project team that the success of the POC plant not be solely dependent on the operation of a computer control system.

6.4.1 Control and Data Acquisition System (CDAS)

The CDAS used both digital and analog inputs to monitor the operation of the plant including the batch operation of the low-shear reactor. The system was capable of both analog (4-20mA current loops) and digital (contact closure) control as described below.

The system used Texas Instruments Series 500 industrial control hardware. A system block diagram is shown in Figure 6-1. Analog and digital input/output (I/O) modules acted as the interface between the field wiring and the computer control system. The 530T Control processor, a ladder-logic programmable controller, was used to streamline the routing of information between the I/O modules and the Basic PID module. The Basic PID module was a Texas Instruments custom unit which allowed user programs to be run in conjunction with PID loop controls. The controller had two serial ports for connection to 'host' and 'slave' units to which monitors could be attached. Both monitors were operator interfaces to the process operation.

A Wyse monochrome ASCII monitor was used to observe all measured points in the process, verify the position of the valves at the low-shear reactor, and allow a limited control of the operation.

The heart of the system was the Wyse 286 high resolution computer. It provided the same information as the monochrome monitor (combined with graphics), acquired and stored data at specific points in the process at the appropriate times, and automatically sequenced the operation of the low-shear reactor.

It quickly became apparent, while running test versions of the software, that the 9600 baud serial line and the processing speed of the Wyse 286 could not give adequate response time (a single system scan would take 20 seconds or more). Because of this, operation of the low-shear reactor was controlled through the ASCII terminal with

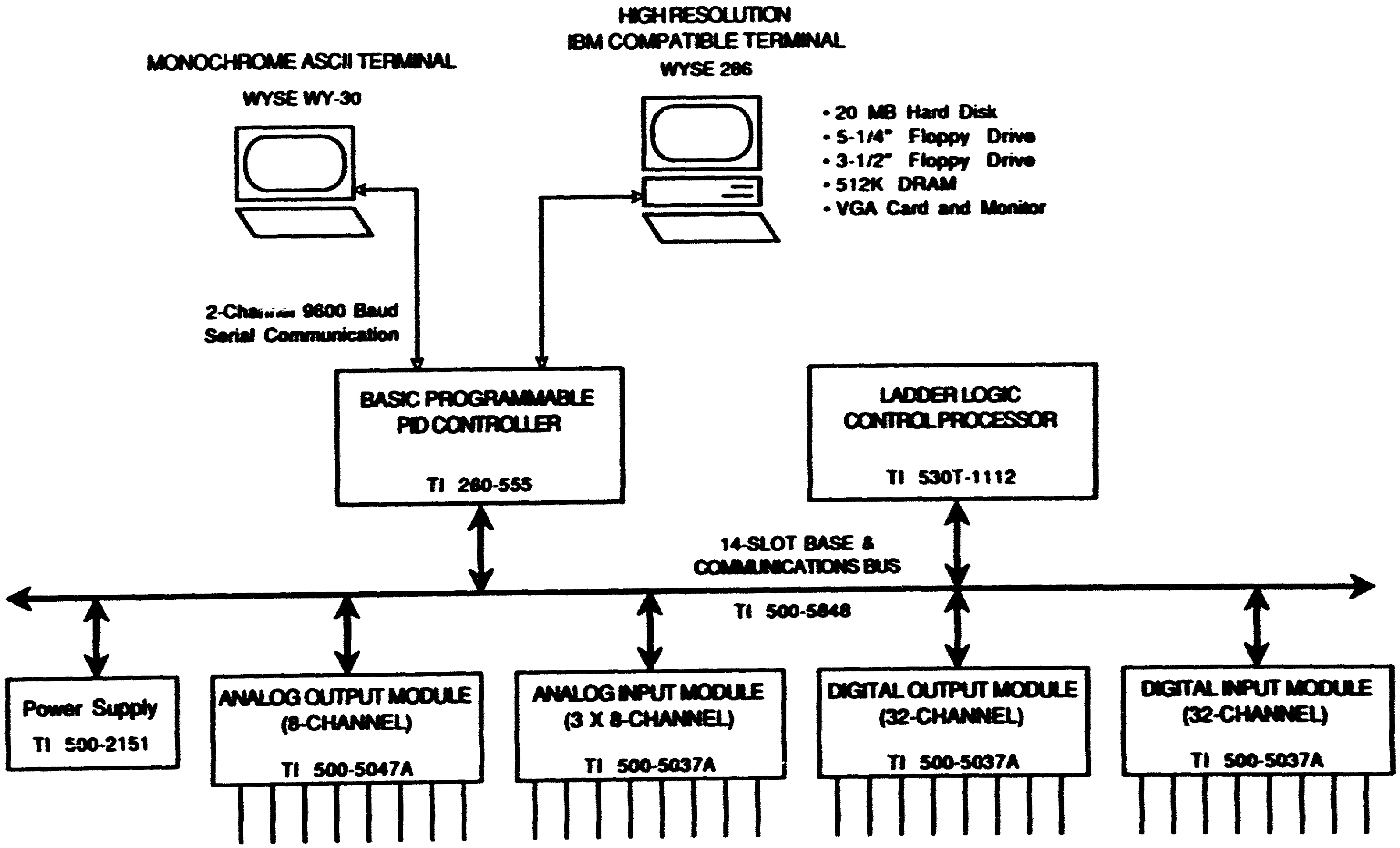


Figure 6-1 CDAS System Block Diagram

manual data acquisition through this same terminal (a single scan time of from 1 to 1.5 seconds).

The Wyse 286 personal computer was used to set up each agglomeration test run and to organize the results of test runs. This capability in the plant control room allowed for immediate evaluation of process results and determination of new process setpoints. The Wyse 286 was also fitted with a modem which allowed transfer of data between the field, home office, and others.

Individual CDAS Control Functions. The CDAS was designed to coordinate the startup, operation, and termination of each agglomeration run which involved opening and closing valves and starting pumps and mixers in the correct order. Actual POC operation used a mixture of automatic, remote, and local controls. A sequence of operation, showing how each step of the operation was carried out, is shown as Table 6-1.

The CDAS provided PID loop control of the slurry flow rate to the high-shear reactor. Other automatic control functions included the control of the interface level in the oil/water separator and the water level in the carbon filter drum. The CDAS also provided contact closures to the alarm panel to alert operators of low levels in the slurry feed tank and the primary tailings surge drum. When failures of individual control loops occurred they were all caused by a failure of instrumentation or other plant equipment.

CDAS Operation Notes. The CDAS monochrome monitor provided a 'window' for observing the status of the agglomeration plant. Many of the plant control functions were carried out from this terminal. In addition, problems in the plant could be detected using the information presented on the screen.

The PID control module custom BASIC program could be easily changed by a control systems engineer. It took a day to learn the system functions and approximately 8 days to operate it effectively. Using

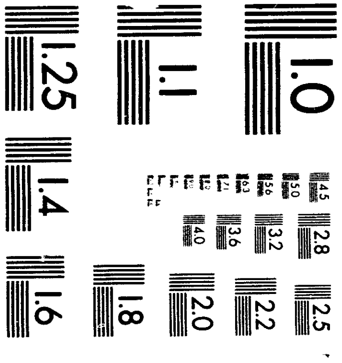
Table 6-1

SEQUENCE OF OPERATION SUMMARY FOR THE AGGLOMERATION POC PLANT

Sequence No.	Action	Type
Preoperation		
1.	Determine timing and set points for the run	286 *
2.	Set metering pump flow rates (G-3, G-5, G-25), put controls into auto position, open metering pump manual block valves	286/Local *
3.	Set low and high-shear reactor mixer speeds	286/CDAS *
4.	Set high-shear mixer flow rate (G-6)	286/CDAS
5.	Start high-shear mixer, check speed and power draw	Remote/CDAS *
6.	Open microagglomerate feed valve (UV-C08A)	CDAS
Operation		
7a.	Start slurry feed to agglomeration	Remote
7b.	Start metering pumps to high-shear reactor	Interlock *
7c.	Record start time and power draw (as required)	CDAS
	Upon set level in low-shear reactor:	
8a.	Start low-shear mixer	Remote
8b.	Open binder addition valve (UV-C08B)	CDAS
8c.	Start binder addition to C-08	Remote
8d.	Record time and power draw (as required)	CDAS
	Upon end of microagglomerate feed time:	

Legend

*286	– Advice from Wyse 286
*Local	– Action is at or near equipment or valve
*CDAS	– Action is accomplished with command through CDAS
*Remote	– Action is accomplished in control room by panel-mounted push buttons
*Interlock	– Action is hardwired interlocked


1.0
1.1
1.25
1.4
4.5
5.0
5.6
6.3
2.8
3.2
3.6
4.0
1.6
1.8
2.0
2.2
2.5

2 of 3

Table 6-1 (Cont'd)

Sequence No.	Action	Type
9a.	Stop slurry feed pump (G-6)	Remote *
9b.	Stop metering pump(s) to high-shear reactor, close block valves	Interlock *
10.	Stop high-shear mixer	Remote
11.	Close microagglomerate feed valve (UV-C08A)	CDAS
12.	Record time of action	
	Upon end of binder feed low shear reactor:	
13.	Stop binder pump to low-shear reactor	Remote
14.	Close the binder additional valve (UV-C08B)	CDAS
15.	Close the binder addition block valve	Local
16.	Record time of action	
	Upon end of agglomerate growth cycle:	
17.	Stop low-shear mixer (Y-8)	Remote
18.	Open primary tailings drain valve (UV-C08C)	Local
19.	Record time of action	
	Backflush of primary tailings screen (optional):	
20.	Open flush water block valve to the low-shear reactor screen	Local
21.	Close primary tailings drain valve	Local
22.	Open flush water valve for 30-60 seconds (UV-C08D)	CDAS
23.	Open primary tailings drain valve; repeat backflush sequence as necessary to obtain 6-10" residual water in low-shear reactor	Local

Legend

*286	– Advice from Wyse 286
*Local	– Action is at or near equipment or valve
*CDAS	– Action is accomplished with command through CDAS
*Remote	– Action is accomplished in control room by panel-mounted push buttons
*Interlock	– Action is hardwired interlocked

Table 6-1 (Cont'd)

Sequence No.	Action	Type
24.	Open flush water block valve	Local
25.	Close primary tailings drain valve to the low-shear reactor screen	Local
26.	Open flush water valve for 30-60 seconds (UV-C08D)	CDAS
27.	Close flush water block valve	Local
	Start agglomerate steam cycle:	
28.	Set low-shear reactor vapor vent line from gas blanket to heat exchanger (UV-C08H)	CDAS
29.	Record start of steaming time	
30.	Open top steam valve to achieve desired steam flow (UV-C08F)	Local
31.	When low-shear reactor overhead temperature +200°F open bottom steam valve (UV-C08G)	CDAS/Local
32.	Record agglomerate bed temperature and steam flow rate as needed	CDAS
	When agglomerate bed temperature +222°F and no heptane vapors overhead then stop steaming	
33a	Close bottom steam valve (UV-C08G)	Local
33b	Close top steam valve (UV-C08F)	Local
34.	Add 15-45 seconds of water through bottom of low shear reactor	CDAS/Local
35.	Set low-shear reactor vapor vent line to gas blanket (UV-C08H)	CDAS
	When agglomerate bed temperature <120°F then start dump cycle (add flush water as necessary to reduce bed temperature)	

Table 6-1 (Cont'd)

Sequence No.	Action	Type
36.	Start drainage belt with water and air sprays	Local
37.	With block valves closed, open flush water water valve (UV-C08D)	CDAS
38.	With instrument air off to bottom drain valve, start automatic cycling of valve (UV-C08E)	CDAS
39.	Dump low shear reactor using bottom drain valve, flush water and low-shear mixer as required to remove reactor contents	Local
40.	Record end of run time	

the CDAS functions to operate the POC plant was easy for engineers who had prior computer experience. The CQ Inc. plant operators were uncomfortable with the system. They would have preferred an all panel-mounted control system with "recipe" type instructions.

An improvement to the system is the use of the Wyse 286 as a data logger only, with no other function than to take trend information for only certain parts of the process. In hindsight, the system could have also worked better with an off-the-shelf PC program written specifically as an operator interface to plant control systems (e.g., Genesis or CIM-PAC software).

One of the major challenges in designing control systems for this type of POC plant is that the system not only *must* operate the process but also must provide information to safely run the plant and collect data for the POC test program. Also, for short duration demonstration programs the system is required to be fully operational immediately after the installation of all plant equipment. For commercial plants, the normal practice is to design and install the basic instruments and controls and then allow time for fine tuning the operation based on direct experience.

6.4.2 Process Alarm System

A separate, "hardwired" alarm system was also installed in the POC plant. This system consisted of switch contact inputs to an annunciator panel. The annunciator panel was mounted over the CDAS operator work station to alert the operators of plant conditions outside normal operation. When an alarm was activated, the system would sound an audible alarm and a flashing light would indicate the cause for the alarm. The operator could then acknowledge the alarm, assess the condition, and take the appropriate action(s).

6.4.3 Fire Protection System

The use of heptane required an automatic fire detection and extinguishing system consisting of a deluge-type sprinkler system with an aqueous film forming foam (AFFF) system. The system could be actuated manually or through flame detectors positioned throughout the plant. The system was designed and installed by Grinnel Fire Protection Company.

6.4.4 Ventilation System

The building ventilation system was designed to quickly dissipate concentrations of flammable vapors in the advanced process building. The system consisted of six hydrocarbon sensors positioned throughout the advanced process building, six roof ventilation supply fans, and two dual-speed wall exhaust fans. During normal operation the system provided a minimum of three air changes per hour in the advanced process building. If the hydrocarbon level reached 20 percent of the lower explosive limit (LEL) the system would automatically provide six air changes per hour to sweep the vapors out of the building. Detection of vapors above 60 percent of the LEL sounded an alarm and increased the number of air changes per hour to 12.

This system was found to be reliable. Four accidental heptane vapor releases caused actuation of the system at the 20 percent LEL level detections. The 60 percent level was never reached.

6.4.5 Gas Blanket System

The oxygen content of the vessels containing heptane was maintained below combustible limits by a nitrogen blanket system including a gas holder. Level control in the gas holder tank was maintained by a 'hardwired' level control system. Level gauge switches opened valves to add either nitrogen to the system or to purge excess gas through the flare. The oxygen content of the system was checked on a daily basis.

Frequent bleeding of nitrogen due to the intermittant plant operation resulted in the loss of heptane vapors through the flare. A continuously operating commercial plant would use refrigeration ahead of the flare to recover the heptane.

6.4.6 Instrumentation Notes

Measurement of flows, densities, levels, and other parameters of slurries and asphalt solutions provided a number of challenges. Some of the instruments performed well while others did not. Proper operation of instrumentation requires time to install, calibrate, and maintain. Information must be recorded, reduced, and analyzed. Efforts to obtain complete material balances for every flowstream for each test run were hindered by instrument related problems.

Magnetic Flow Meter. These instruments proved to be accurate and reliable and were easy to install, service, and calibrate. The agglomeration process depended on a steady flow rate of coal slurry into the high-shear reactor. During winter, the plant was subjected to freezing temperatures when the heating system failed on one occasion. The freezing slurry destroyed a meter. A spare meter was calibrated and installed within a day.

Turbine Flow Meters. Turbine flow meters provided highly accurate measurements of water streams. They were also prone to plugging if solids enter into the process stream. Turbine flow meters were used to measure flush water flow into the low-shear reactor and the water addition in to the grinding circuit.

Because of line size and flow rate considerations, the steam flow rate measurement was attempted by an insertion-type flow meter. Measurements were highly variable of up to 500 lb/hr within a second. The condition was alleviated by implementing a moving average filter on the signal through the CDAS. In addition to flow measurement, there were other operational difficulties with the steam sparging system. The agglomerates were very sensitive to steam sparging conditions and could not tolerate agitation or sudden heptane outgassing at high steam flow rates. The manual control valve could not be set for precise and steady steam flow. The entire steam sparging system must be designed to provide a controllable amount of steam.

The measurement of flush water to the low-shear reactor was useful only for indicating the moment of water addition. The measurement was not used to provide information for a water balance as originally intended.

Due to delivery problems, the turbine flow meter to monitor the total water addition to the grinding circuit was changed to a vortex shredder type flow meter which was available at the site. The two types of flow meters have comparable accuracies. The principal advantage of the vortex meter is its insensitivity to plugging. A disadvantage is the minimum flow requirement before the meter will indicate any flow. Since the flow rate was constant throughout the operation this was of no concern. The instrument was to provide flow rate data. However, manual valves at the rotameters provided a more direct and accurate control of the distribution of water throughout the grinding circuit. Instead the instrument was used to check the readings from the rotameters.

Rotameters. Rotameters proved to be a reliable and easy to use means of setting and monitoring the water addition to individual points in the grinding circuit. A disadvantage of these meters was the quite faint calibration line. The slightest buildup of solids in the meter made reading difficult and the instruments required cleaning at least once every 2 weeks.

Magnetic Level Gauges. Magnetic level gauges were used outside on process vessels. The gauges work with a magnetized float in a strongback which turned edge-magnetized wafers. A very clear indication of level could be obtained and interface levels could be detected.

This type of meter was best utilized at the emergency slop tank and the carbon filter drum. The emergency slop tank was often filled with slurries of coal and coal tailings that had to be steamed. Under these harsh conditions the level gauge performed well and provided a clear indication of the level in the vessel. Future use of this instrument should incorporate an additional magnetically actuated high level switch.

The magnetic level gauge was not accurate enough for use at the heptane feed tank. Instead, an armored glass gauge was added to give a direct indication of the level in the tank and measure the heptane inventory in the agglomeration system.

Differential Pressure Level Gauges. Differential pressure (D/P) level gauges were used with success. They worked well in the low-shear reactor, primary tailings surge drum, and the carbon filter drum.

Bubble Tube Level Transmitters. Bubble tube level transmitters were used successfully on the project. Bubble tubes were installed to monitor the level in open tanks such as the primary sump, the slurry feed tank, and the final tailings surge tank.

There were problems in ensuring a continuous supply of instrument air to the bubbler dip tube. One of the advantages of the bubble tube is that it is self cleaning: the air always bubbles out of the bottom of the dip tube, keeping the slurry out. Due to maintenance and operating cost considerations, CQ Inc. preferred not to keep their compressor operating on a continuous basis. It became standard practice to isolate the instrument air system from the rest of CQ Inc. systems at night and run a portable air compressor to provide the small quantity of air flow needed. On one occasion this compressor failed, resulting in plugged dip tubes.

Capacitance Level Probe. A capacitance level probe was used to measure the interface level between water and heptane in the oil/water separator. This instrument worked extremely well. The instrument was coupled through the CDAS to a valve which controlled the discharge of water from the separator, and therefore the interface level. The control valve, and the piping spool before it, would sometimes become clogged with slurry and require cleaning. A low level alarm was therefore wired between the CDAS and the alarm panel which activated when the transmitter indicated a danger of heptane draining out to the carbon filter drum.

Displacement Level Switches. Displacement level switches with ceramic floats were a cost effective means of providing level control and indicating alarm levels of sumps containing water and slurries. They were also very easy to install and performed without problems.

Temperature Transmitters. The temperature transmitters in the low-shear reactor agglomerate bed and vapor vent provided information about conditions in the reactor during the steam stripping step. The overhead vapor temperature was monitored to ensure that adequate steam was added to avoid recondensation of vapors (rain) inside the reactor. The agglomerate bed temperature was the most accurate indication of the progress of the steam stripping step. The bed temperature would rise to the boiling point of heptane rapidly and then slowly increase while the heptane was being stripped away from the agglomerates. When most of the heptane had been stripped the bed temperature would rise to the saturated steam temperature. When this temperature had been reached the operators knew that steaming was at, or near, completion. The temperature indicated in the subcooled liquid leg of the condenser was useful for ensuring a sufficient supply of cooling water.

Pressure Transmitters. Pressure transmitters were used to monitor the gas blanket system, nitrogen supply, cooling water, instrument air, and process water. Contacts were provided to the alarm system to alert the operators of any loss of pressure. Having the pressure indicators at a central location provided the operators with an easy means to monitor the operation.

A problem was encountered with the pressure switch which was to control the binder addition to the low-shear reactor. Binder addition was controlled by an automatic control valve which was programmed to open and close at the appropriate times. Since positive displacement metering pumps were used to pump the binder closing the valve caused an increase in the line pressure. This caused the pressure switch to shut off the metering pump. When the valve opened the pressure was relieved and the metering pump would restart. However, pressurizing the piping spool between the pump and the valve promoted asphalt plugging. It was found more reliable to add binder by timing the start-stop function at the metering pump.

Variable Speed Drives. While not considered instruments, the variable speed drives provided a direct and precise control of pumps and mixers. Their use contributed significantly to the smooth operation of the plant.

Nuclear Density Gauges. These instruments were used to monitor solids flow in the selective grinding circuit. Optimizing their use required more time than available during the project. The best use of these instruments was in monitoring fluctuations in grinding circuit conditions that merited closer investigation.

6.5 OPERATOR'S COMMENTS ON POC OPERATION

A discussion was held towards the end of the operation with the four CQ Inc. operators to evaluate the performance of the project from their point of view. Technicians were asked to provide their opinions on project objectives, communications, operator training, and safety. In any areas where they saw deficiencies, they were asked to offer solutions based on prior professional experience.

Well defined project objectives was a concern shared by all the operators. They expressed concern that despite all the work they had done with the equipment, they still felt that time was too short and they were not qualified to list this experience on their resumes.

After completion of testing, the least voiced concern of the operators was about the safety of the plant. This was in spite of that during the plant design stage there was a great amount of concern about the risks associated with using heptane in physical coal cleaning. The only safety concern was with the generation of coal dust during the off specification product dumping. This problem was taken care of by the use of respirators and operation of the building ventilation system at the maximum.

Section 7

PROCESS EVALUATION CRITERIA AND DEFINITIONS OF TERMS

Definitions of performance used to evaluate process performance are presented below together with sample calculations that use the data shown in Table 7-1.

7.1 YIELD OF CLEAN COAL

The yield refers to the weight percentage of the solids fed to a cleaning facility which is recovered as clean coal. The ash balance method was used to determine the yield. This method uses the ash contents of the refuse, feed, and clean coal (less binder).

The ash content of the feed, clean coal agglomerates, primary tailings, and secondary tailings was analytically determined. The ash content in the total tailings was determined from the ash contents and weight proportion of primary and secondary tailings. The clean coal ash content, less binder, was derived from the analysis as follows:

Clean Coal Ash (excluding binder)[%]=

$$\frac{100\ (\text{Agglomerate Ash }[\%]) - (\text{Binder }[\text{Wt }\%])\ (\text{Binder Ash}[\%])}{100 - (\text{Binder }[\text{Wt }\%])}$$

The yield was then calculated as follows:

$$\text{Yield} = \frac{(\text{Tailings Ash }[\%]) - (\text{Feed Ash }[\%])}{(\text{Refuse Ash }[\%]) - (\text{Clean Coal Ash }[\%])} \text{ X } 100\ [\%]$$

The sample calculation is described below.

The ash in the binder has been determined to be 0.2 percent. Taking the values for the test example (Table 7-1):

Table 7-1

SUMMARY TEST CONDITIONS AND RESULTS
PITTSBURGH SEAM COAL

Date: X/XY/89			Test No: X-0X		
A. POC Plant Feed:			G. Process Conditions:		
Ash	12.1	[%]	Feed Rate	—	[tph]
Total Sulfur	4.37	[%]	Med Particle	—	[mics]
Pyritic Sulfur	2.02	[%]	Heptane-HSR	—	[l%]
Heating Value	12791	[Btu/lb]	Asphalt Dosage	—	[%]
B. Clean Coal (excl. Binder):					
Ash	5.06	[%]	HSR Impeller Type	A/B	
Total Sulfur	xyzz	[%]	HSR Impeller	—	[RPM]
Pyritic Sulfur	xyzz	[%]	HST Power Draw	—	[kW]
Heating Value	13870	[Btu/lb]			
C. Clean Coal Agglomerates (incl. Binder)			LSR Impeller Type	A/B	
Ash	4.90	[%]	Steam Flow	—	[lb/m]
Total Sulfur	3.67	[%]	Steam Flow	—	[min]
Pyritic Sulfur	1.28	[%]	No. of Dumps	—	
Heating Value	14149	[Btu/lb]			
Asphalt (Binder)	3.3	[%]	Analysis ROM Coal		
D. Tailings			Ash	39.16	[%]
Ash	69.4	[%]	Total Sulfur	4.71	[%]
Total Sulfur	xyzz	[%]	Pyritic Sulfur	3.39	[%]
Pyritic Sulfur	xyzz	[%]	H.V.	8528	[Btu/lb]
Heating Value	xyzz	[Btu/lb]			
E. POC Plant Performance:			Precleaning		
Yield	89.1	[%]	Energy Recovery	3.74	[%]
Energy Recovery	96.6	[%]	Yield	62.5	[%]
Ash Reduction (a)	63.4	[%]	H.V. = 14646 - (153.3 x Ash %)		
SO_2 Reduction (a)	24.0	[%]			
Pyritic Sulfur Red. (a)	38.4	[%]	Ash in asphalt	0.2	[%]
Ash Removal (b)	56.4	[%]			
Sulfur Removal (b)	25.2	[%]	Analysis-Precleaned Coal		
Pyritic Sulfur Rem. (b)	—	[%]	Ash	12.1	[%]
Ash Reduction -(Alt) (c)	—	[%]	Total Sulfur	4.37	[%]
Sulfur Reduction -(Alt) (c)	—	[%]	Pyritic Sulfur	2.02	[%]
Pyritic Sulfur Red. -(Alt) (c)	—	[%]	H.V. 12791		[Btu/lb]
F. ROM Basis Performance					
Energy Recovery	90.5	[%]			
Ash Reduction (a)	92.4	[%]			
SO_2 Reduction (a)	52.8	[%]			
Pyritic Sulfur Red. (a)	77.3	[%]			
Ash Removal (b)	—	[%]			
Sulfur Removal (b)	56.7	[%]			
Pyritic Sulfur Rem. (b)	—	[%]			
Ash Reduction -(Alt) (c)	87.5	[%]			
Sulfur Reduction -(Alt) (c)	—	[%]			
Pyritic Sulfur Red. -(Alt) (c)	—	[%]			

(a) lb/MMBtu Basis
(b) ((100 X % in Feed)-(Yld % x % in Clean Coal))/(% in Feed)
(c) 100 x (% in Feed - % in Clean Coal)/(% in Feed)

Clean Coal Ash (excluding binder) =

$$\frac{100(5.78) - (3.3)(0.2)}{(100 - 3.3)} = 5.06\ [\%]$$

The ash contents in the refuse and feed were 69.4 and 12.1 percent, respectively. The yield was calculated as follows:

$$\text{Yield} = \frac{69.40 - 12.10}{69.40 - 5.06} \times 100 = 89.1\quad [\%]$$

7.2 ENERGY RECOVERY

Energy recoveries were calculated as follows:

a) $$\text{Test Energy Recovery} = \frac{\text{Yield} \times \text{HVc*}}{\text{HVf*}}\ [\%]$$

Where:

*HVc = Heating value of the clean coal from POC test (less binder) [Btu/lb]

*HVf = Heating value of the coal feed to the POC test [Btu/lb]

b) $$\text{ROM Energy Recovery} = \frac{(\text{Test Energy Recovery})(\text{Precleaning Energy Recovery})\ [\%]}{100}$$

Heating values were obtained either by analysis or by using a regression derived from several analysis of ash contents and heating values.

Precleaning energy recoveries were based on performance during the precleaning of ROM coal where applicable.

A sample calculation for POC plant performance is as follows:

HV = 14,646 - (153.3 x Ash%) [Btu/lb]

Feed HV = 14,646 - (153.3 x 12.10) = 12,791 [Btu/lb]

Clean Coal HV = 14,646 - (153.3 x 5.06) = 13,870 [Btu/lb]

$$\text{POC Plant Energy recovery} = 89.1 \times \frac{13,870}{12,791} = 96.6\ [\%]$$

The sample calculation for ROM basis performance is as follows:

Based on an energy recovery of 93.7 percent for the precleaning operation, the ROM basis energy recovery is:

$$\text{ROM Energy Recovery} = \frac{96.6 \times 93.7}{100} = 90.5 \ [\%]$$

7.3 ASH REDUCTION

Ash reduction is defined as the percentage decrease in ash content between the feed coal and the clean coal with the ash contents being measured on a constant energy basis (lb Ash/MMBtu):

$$\text{Ash reduction} = 100 \ \frac{(\text{ASHMf} - \text{ASHMca})}{\text{ASHMf}} \ [\%]$$

Where

ASHMf = Ash content in the feed [lb/MMBtu]

ASHMca = Ash content in the clean coal agglomerates [lb/MMBtu]

$$\text{Ash Content} = \frac{\text{As} \times 10{,}000}{\text{HVs}} \ [\text{lb/MMBtu}]$$

As = Ash content of the sample [%]

HVs = Heating value of the sample [Btu/lb]

Since the clean coal agglomerates contain a binder (asphalt), correction has to be made to the calculated HV of the agglomerates. This is done as follows:

$$\text{HVca} = \frac{(\text{HVb} \times \text{Bca}) + ((\text{HVc}) \times (100 - \text{Bca}))}{100} \ [\text{Btu/lb}]$$

Where:

HVca = Heating value of the clean coal agglomerates (including binder) [Btu/lb]

HVb = Heating value of the asphalt binder (22,000 Btu/lb) [Btu/lb]

HVc = Heating value of the clean coal (less binder) [Btu/lb]

Bca = Binder content of the clean coal agglomerates [%]

The sample calculation for POC plant performance is as follows:

$$\text{HVca} = \frac{(22{,}000)(3.3) + (13{,}870)(100-3.3)}{100} = 14{,}139 \ [\text{Btu/lb}]$$

$$\text{ASHMca} = \frac{4.90 \times 10{,}000}{14{,}139} = 3.47 \ [\text{lb/MMBtu}]$$

$$\text{ASHMf} = \frac{12.10 \times 10{,}000}{12{,}791} = 9.46 \ [\text{lb/MMBtu}]$$

$$\text{Ash reduction} = 100 \times \frac{9.46 - 3.47}{9.46} = 63.4 \ [\%]$$

The sample calculation for ROM basis performance is as follows:

$$\text{ASHMrom} = \frac{39.16 \times 10{,}000}{8{,}528} = 45.9 \ [\text{lb/MMBtu}]$$

$$\text{Ash Reduction} = \frac{45.9 - 3.47}{45.9} = 92.4 \ [\%]$$

7.4 SO_2 REDUCTION

The SO_2 reduction determination is similar to ash reduction described previously and is based on emissions of SO_2 per million Btu's of energy release.

$$SO_2 \text{ emission potential} = \frac{S \times 20{,}000}{HV} \ [\text{lb/MMBtu}]$$

Where:

S = Total sulfur content of sample [%]

HV = Heating value of sample [Btu/lb]

$$\text{Sulfur dioxide reduction} = \frac{100\ (SO_2f - SO_2\,ca)}{SO_2f} \ [\%]$$

Where:

SO_2f = SO_2 emission potential of the feed [lb/MMBtu]

SO_2ca = SO_2 emission potential of the clean coal agglomerates [lb/MMBtu]

The sample calculation for POC plant performance is as follows:

$$\text{SO}_2\text{ emission potential of the clean coal agglomerates} = \frac{3.67 \times 20{,}000}{14{,}139} = 5.19\ [\text{lb/MMBtu}]$$

$$\text{SO}_2\text{ emission potential of the feed} = \frac{4.37 \times 20{,}000}{12{,}791} = 6.83\ [\text{lb/MMBtu}]$$

$$\text{SO}_2\text{ reduction} = \frac{100\ (6.83 - 5.19)}{6.83} = 24.0\ [\%]$$

The sample calculation for ROM performance is as follows:

$$\text{SO}_2\text{ emission potential of the ROM coal} = \frac{4.71 \times 20{,}000}{8{,}528} = 11.0\ [\text{lb/MMBtu}]$$

$$\text{SO}_2\text{ reduction} = \frac{100\ (11.0 - 5.19)}{11.0} = 52.8\ [\%]$$

7.5 ASH REMOVAL, SULFUR REMOVAL, AND PYRITIC SULFUR REMOVAL

These performance categories are calculated using the methodology illustrated below for sulfur removal.

Sulfur removal is defined as the weight percentage of sulfur in the feed to the coal cleaning operation that is rejected with the refuse/tailings.

$$\text{Sulfur removal} = \frac{Sf - (\text{Yield, }\%100) \times Sca}{Sf} \times 100\ [\%]$$

Where:

Sf = Total sulfur content of the feed [%]

Sca = Total sulfur content of the clean coal agglomerates [%]

The sample calculation for POC plant performance is as follows:

$$\text{Sulfur removal} = \frac{4.37 - 0.891 \times 3.67}{4.37} \times 100 = 25.2\ [\%]$$

$$\text{Yield} = \frac{62.48 \times 89.1}{100} = 55.7\ [\%]$$

The sample calculation for ROM basis performance is as follows: The ROM basis yield is the product of the precleaning yield and the POC plant yield. Assuming a precleaning yield of 62.48 percent,

$$\text{Sulfur removal} = \frac{4.71 - (0.557 \times 3.67)}{4.71} \times 100 = 56.7\ [\%]$$

7.6 PYRITIC SULFUR REDUCTION

The calculation method that determines the pyritic sulfur reduction is as follows:

$$\text{Pyritic sulfur reduction} = \frac{100\ (\text{PSMf} - \text{PSMca})}{\text{PSMf}}\ [\%]$$

Where:

PSMca = Pyritic sulfur content in the clean coal agglomerates [lb/MMBtu]

PSMf = Pyritic sulfur content in the feed [lb/MMBtu]

The sample calculation for POC plant performance is as follows:

$$\text{PSMca} = \frac{1.28 \times 10{,}000}{14{,}149} = 0.90\ [\text{lb/MMBtu}]$$

$$\text{PSMf} = \frac{2.02 \times 10{,}000}{13{,}870} = 1.46\ [\text{lb/MMBtu}]$$

$$\text{Pyritic sulfur reduction} = \frac{100\ (1.46 - 0.90)}{1.46} = 38.4\ [\%]$$

The sample calculation for ROM basis performance is as follows:

$$\text{Pyritic sulfur in the ROM coal} = \frac{3.39 \times 10{,}000}{8{,}528} = 3.98\ [\text{lb/MMBtu}]$$

$$\text{Pyritic sulfur reduction} = \frac{100\ (3.98 - 0.90)}{3.98} = 77.3\ [\%]$$

7.7 ASH REDUCTION (ALT), SULFUR REDUCTION (ALT), PYRITIC SULFUR REDUCTION (ALT)

These performance indices are calculated using the same methodology as illustrated below for ash reduction:

Ash reduction (Alt)

Ash reduction is defined as follows:

$$\text{Ash reduction (Alt)} = \frac{100\ (\text{Af} - \text{Aca})}{\text{Af}}\ [\%]$$

Af = Ash content in the feed [%]

Aca = Ash content in the clean coal agglomerates [%]

The sample calculation for POC plant performance is as follows:

$$\text{Ash reduction (Alt)} = \frac{100\ (12.1 - 4.90)}{12.1} = 59.5\ [\%]$$

The sample calculation for ROM basis performance is as follows:

$$\text{Ash reduction (Alt)} = \frac{100\ (39.16 - 4.90)}{39.16} = 87.5\ [\%]$$

7.8 EFFICIENCY FACTOR

The Efficiency Factor is defined as follows:

$$\text{Efficiency Factor} = \frac{\text{Yield} \times \text{Ar}}{\text{Aca}}$$

where

Yield = See paragraph 7.1

Ar = Ash Content in the refuse [%]

Aca = Ash content in the clean coal agglomerates [%]

Section 8

PROCESS EVALUATION

8.1 SUMMARY TEST RESULTS

POC Test Results

The three coals tested in depth during the POC operation were:

- Illinois No. 6 seam coal from the Burning Star Mine, Perry County, Illinois
- Upper Freeport seam coal from the Helen Mine, Indiana County, Pennsylvania
- Pittsburgh coal from the Blacksville No. 2 Mine, Monongalia County, West Virginia

Taggart seam coal from the Wentz No. 1 preparation plant, Wise County, Virginia, was also briefly tested. This coal and the Pittsburgh coal were precleaned at the mine.

The POC tests demonstrated that the spherical agglomeration process achieved the major performance objectives of the program, namely, major reduction in pyritic sulfur, high process energy recovery and production of an easily handled product. All tests were characterized by energy recoveries of above 90 percent compared to the program target of 80 percent. On an equal energy content basis, one clean coal contained less than 14 percent of the pyritic sulfur found in the feed to the POC test unit. High energy recoveries were even noted for clean coal with ash contents as low as 3 percent and puritic sulfur content of 0.2 percent. It was observed during the earlier bench-scale tests that a target of 2 percent ash in the clean coal was not possible for the coals from the Illinois No.6, Upper Freeport, and Pittsburgh seams. However, a few tests with prewashed Taggart seam coal produced agglomerates with 1.5 percent ash at an energy recovery of 97 percent. A summary of the test results is shown in Table 8-1.

Table 8-1
PROOF-OF-CONCEPT TEST PERFORMANCE SUMMARY
(Dry Basis)

Coal:	**ILLINOIS**	**UPPER FREEPORT**	**PITTSBURGH(1)**	**TAGGART(1)**
GRIND TYPE:	**Selective W/Spiral**	**Selective W/Spiral**	**Selective W/Spiral**	**Selective**
GRIND SIZE (d90,µm):	18.0	11.6	15.6	17.5
(d50,µm):	7.4	3.7	5.7	7.9
POC Feed Coal				
Ash, %	17.40	23.27	9.43	3.57
Total Sulfur, %	4.03	3.74	2.42	0.72
Pyritic Sulfur, %	1.81	2.87	1.25	0.08
Heating Value, Btu/lb	11,708	11,551	13,600	15,109
Agglomerates (W/ Binder)				
Ash, %	3.54	5.48	3.40	1.47
Total Sulfur,%	2.57	1.35	1.59	0.64(2)
Pyritic Sulfur, %	0.21	0.52	0.34	0.04
Heating Value, Btu/lb	14,013	14,855	14,793	15,421
lb Ash/MMBtu	2.53	3.69	2.30	0.95
lb SO_2/MMBtu	3.67	1.82	2.15	0.83(2)
lb Pyritic SO_2/MMBtu	0.30	0.70	0.46	0.05
Agglomeration Refuse				
Ash, %	86.39	86.20	87.78	87.45
Sulfur, %	4.89	6.34	5.20	2.73
Performance				
Yield (overall)	78.2	74.1	83.1(3)	98.0(3)
Energy Recovery, %	93.0	94.7	90.0	99.8
Ash Reduction, %	83.0	81.7	66.7	59.7
SO_2 Reduction, %	46.7	71.9	39.3	
Pyritic SO_2 Reduction, %	90.3	85.9	74.9	
Efficiency Factor	1,910	1,166	2,146	5,830

(1) Taggart and Pittsburgh seam POC plant feeds were precleaned at the mine.

(2) Estimated.

(3) Yield based on feed to grinding circuit (spiral not used with Taggart coal).

As seen in the table, the refuse was practically free of combustible material at ash contents above 86 percent. The Efficiency Factor, an index of cleaning efficiency, was calculated at 1,910 for the Illinois seam coal, 1,166 for the Upper Freeport coal, and 2,146 for the Pittsburgh seam coal. A definition of 'Efficiency Factor' (EF) is given in Section 7. A large value for EF is indicative of a high yield of clean coal combined with a large reduction in the ash content. The value for Pittsburgh coal includes prewashing at the mine.

The POC tests proved that:

- The degree of mineral liberation obtained during the grinding step ahead of agglomeration was the dominant factor influencing clean coal quality
- Given an appropriate amount of bridging liquid (heptane), and binder (asphalt), and a sufficient high- and low-shear mixing (impeller design, speed, and retention time), fine coal could be agglomerated into transportable low ash and low pyrite clean coal agglomerates with negligible loss of combustibles during the process
- Performance results obtained during the bench-scale tests could be successfully duplicated and, in many cases, improved in the larger scale POC test unit

8.2 PYRITIC SULFUR REDUCTIONS

Reduction in pyritic sulfur content of coal was one of the prime objectives of the program. Reductions ranged from 75 percent for the Pittsburgh seam coal, to 85.9 percent for the Upper Freeport seam coal and 90.3 percent for the Illinois seam coal. The reported low value for the Pittsburgh coal corresponds to the low pyrite content of the feed which had been prewashed at the mine. In Figures 8-1, 8-2, and 8-3 the pyritic sulfur reductions are plotted against particle size for all three coals. They also identify the grinding circuit configuration used for the different data points.

While particle size is an important criteria, the method of grinding and configuration of the grinding circuit had an even greater

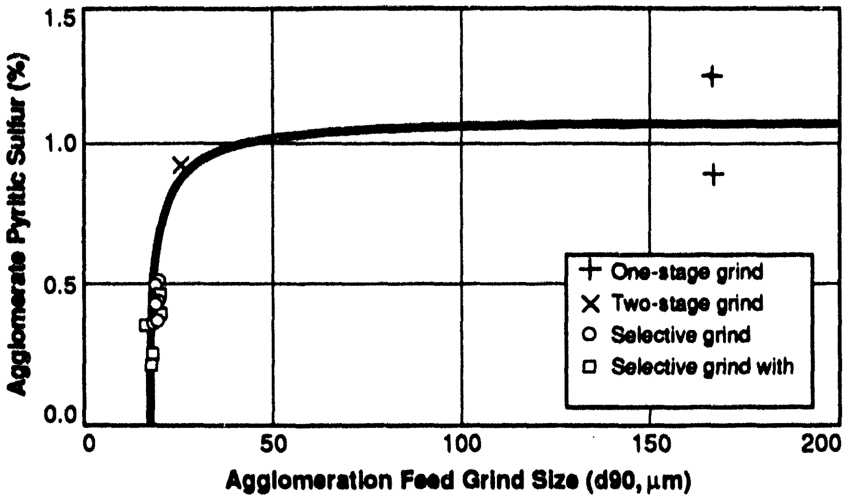


Figure 8-1 Illinois No. 6 Seam Coal — Agglomerate Pyritic Sulfur vs. Agglomeration Feed Grind Size

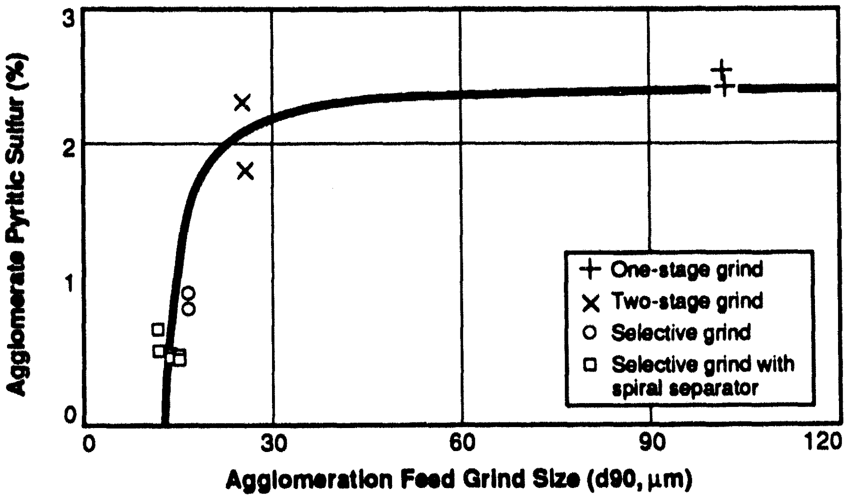


Figure 8-2 Upper Freeport Seam Coal — Agglomeration Pyritic Sulfur vs. Agglomeration Feed Grind Size

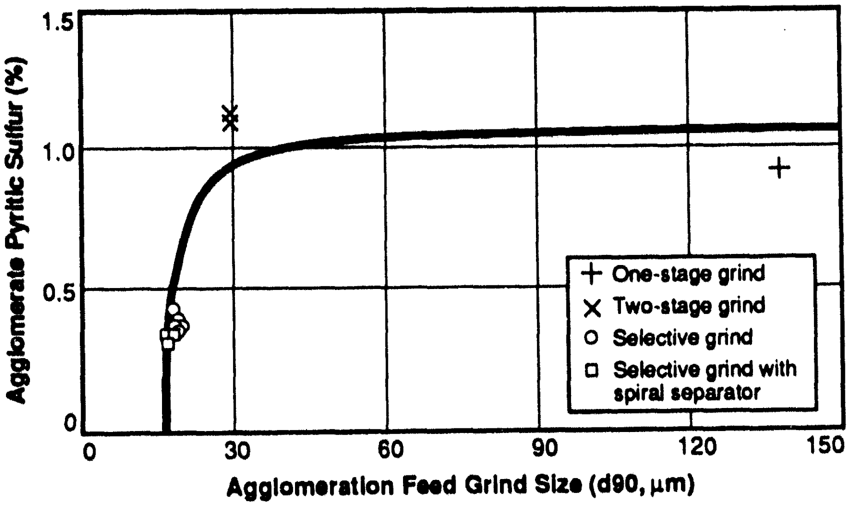


Figure 8-3 Pittsburgh Seam Coal — Agglomerate Pyritic Sulfur vs. Agglomeration Feed Grind Size

influence on process performance. The improvements achieved by the selective grinding system compared to two-stage open circuit grinding is dramatic, particularly when the modified selective grinding circuit (with spiral) was used. As discussed in detail in Section 10, the selective grinding circuit circulated the coarse, hard-to-grind, and high specific gravity pyrite and ash-rich particles repeatedly through the classifier and fine grinding mill until they were ground to extremely fine sizes. Particles that could not be ground to such sizes were indefinitely retained in the circulating loop and did not reach the agglomeration section. In the modified selective grinding configuration a spiral separator was used and such refractory material was recovered from the circulating loop as high ash and high sulfur tailings.

Using and open circuit, two-stage grinding configuration, these particles are agglomerated. The particles have sufficient coal surfaces to be recovered and contaminate the clean coal with pyrite. Rejection of this material by the spiral represented a loss of coal of less than 5 percent. From a pyrite reduction standpoint this loss seemed justified.

8.3 ASH REDUCTION

Ash reductions for all three coals were consistently high. Ash reductions were 66.7 percent for the Pittsburgh seam precleaned coal, 81.7 percent for the Upper Freeport seam ROM coal, and 83 percent for the Illinois ROM coal. Again, as with the pyritic sulfur reductions, higher reductions were experienced for the two ROM coals supplied to the POC plant.

Figures 8-4, 8-5, and 8-6 show a plot of clean coal agglomerate ash against particle size. They also identify the grinding circuit used for the different data points. As noted under Section 8.2, the tests using selective grinding could produce cleaner (lower ash and sulfur) agglomerates for a nominal decrease in the grain size of the agglomeration feed.

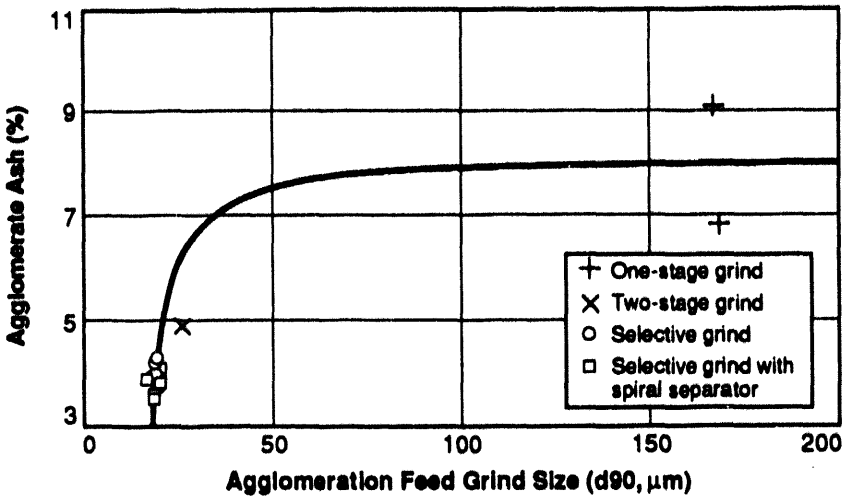


Figure 8-4 Illinois No. 6 Seam Coal — Agglomerate Ash vs. Agglomeration Feed Grind Size

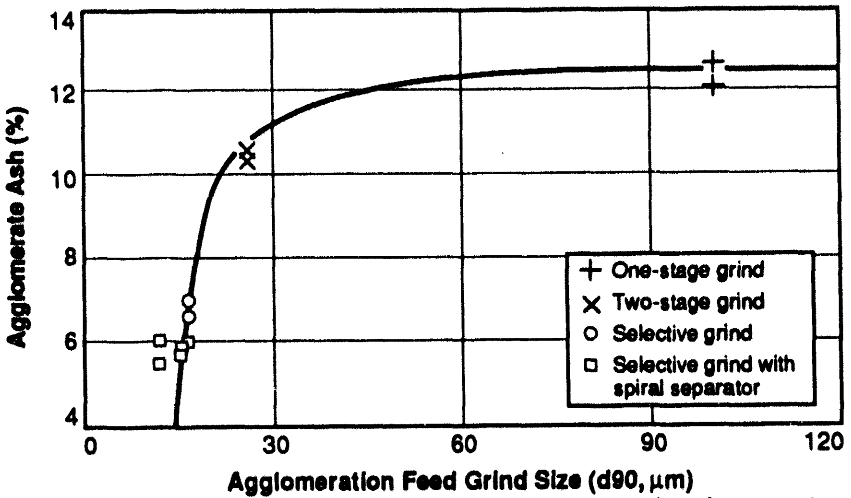


Figure 8-5 Upper Freeport Seam Coal — Agglomerate Ash vs. Agglomeration Feed Grind Size

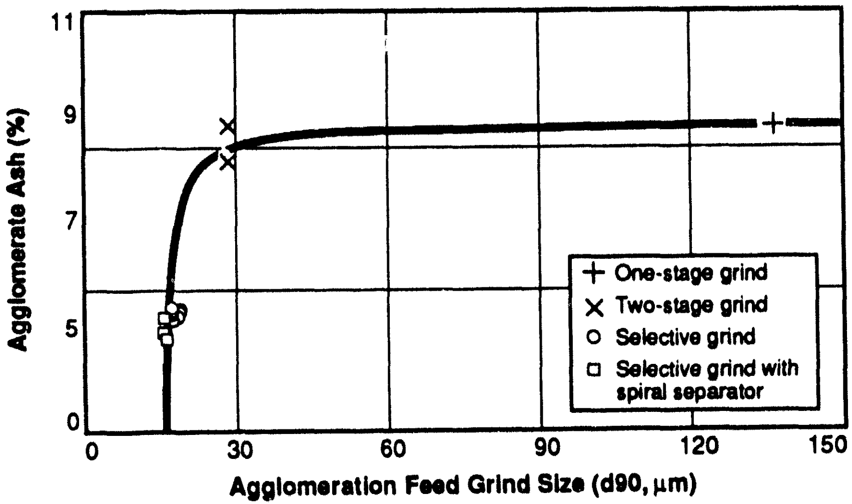


Figure 8-6 Pittsburgh Seam Coal — Agglomerate Ash vs. Agglomeration Feed Grind Size

8.4 PROCESS VARIABLES - EFFECTS ON PERFORMANCE

8.4.1 Illinois No. 6

Table 8-2 shows the test matrix used for the Illinois No. 6 seam coal. Identified below are the significant variables tested:

- Grinding circuit configuration
- Use of the spiral separator
- Solids concentration of the agglomeration feed slurry
- Residence time in the high shear reactor (HSR)
- Bridging liquid dosage to the HSR
- Speed of the HSR impeller
- Speed of the low shear reactor (LSR) impeller
- Agglomerate growth time
- Steaming rate and time
- Asphalt concentration in the agglomerates

The selection of process variables for the Illinois No. 6 coal was determined after reviewing the results of the bench-scale test program. The effects of these parameters were tested and evaluated in the POC unit and additional variables were included in the above list during operation. The effects of significant process variables on performance is seen in Table 8-3. This table also compares POC plant results with results obtained during the bench-scale tests.

The tests allowed for the identification of the most appropriate value for each of the variables. Detailed summaries of each test run can be found in Appendix A, "Individual Run Material Balances."

The Illinois No. 6 coal was relatively difficult to agglomerate compared with the other coals. As noted during bench-scale tests, the coal needed conditioning with a dosage of binder in the high-shear reactor.

As discussed in Section 8.3, the parameter that most affected the ash and sulfur reduction was the degree of liberation, measured by particle size. The importance of this parameter was recognized

Table 8-2
ILLINOIS No. 6 SEAM COAL
TEST MATRIX

Grinding Circuit					Agglomeration Circuit							
					HSR			LSR		Steaming		Asphalt
Serial No.	Run Number	Grind Type	Spiral Used	Slurry Solids(%)	Residence Time(Sec)	Bridging Liquid(1)	Impeller Speed(rpm)	Impeller Speed(rpm)	Growth Time(Min)	Rate(2) (Lb/Ton)	Time (mins)	Conc (%) in Agglomerates
1	30701	Selective	No	15.2	60	20.2	1800	113	10	ND	90	1.84
2	31201	Selective	No	14.6	84	36.1	1800	113	10	5545	42	2.73
3	32501	One Stage	No	13.4	90	25.2	1800	94	10	6125	75	2.53
4	32601	One Stage	No	12.9	90	21.5	1800	82	10	3033	90	7.35
5	34201	Selective	No	11.9	120	43.1	1800	82	15	6222	60	1.94
6	34601	Selective	No	13.4	120	37.1	1800	82	15	9268	140	1.74
7	34701	Selective	No	12.6	120	40.5	1800	82	13	7425	75	3.36
8	34802	Selective	No	14.6	180	35.2	1800	82	10	4740	66	2.22
9	34901	Selective	No	14.5	180	35.3	1800	82	10	2538	78	1.87
10	35301	Selective	Yes	14.0	180	33.1	1800	82	10	3270	80	2.98
11	35401	Selective	Yes	13.2	180	34.4	1800	82	10	2427	66	3.08
12	36801	Selective	Yes	13.3	180	35.0	1800	82	10	3502	92	3.27
13	36803	Selective	Yes	18.2	180	25.2	1800	82	10	1845	47	2.35
14	36902	Selective	Yes	18.2	180	26.4	1800	82	10	7022	72	1.75
15	36904	Selective	Yes	18.2	180	26.4	1800	82	10	ND	ND	2.38
16	37001	Selective	Yes	13.0	180	35.9	1800	82	10	5554	94	3.30
17	37003	Selective	Yes	13.0	180	35.9	1800	82	10	2606	70	3.32
18	37401	Two Stage	Yes	16.5	180	27.2	1800	82	10	3049	89	3.05

(1) Weight percent of dry coal feed

(2) Steam (25 psig) lb/ton of coal

ND Not Determined

Table 8-3
AGGLOMERATION PERFORMANCE SUMMARY
ILLINOIS No. 6 SEAM COAL
(Dry Basis)

Test Program	**Bench-Scale**	**Bench-Scale**	**POC**	**POC**	**POC**	**POC**	**POC**
Performance Data Origin	**Mean Value**	**Best Run**	**Run 32501**	**Run 37401**	**Mean Value**	**Mean Value**	**Best Run**
Grind Type	**Two-Stage**	**Lab Grind**	**Single-Stage**	**Two-Stage**	**Selective**	**Selective with Spiral**	**Selective with Spiril**
Grind Size (d90,µm):			165	25.7	18.9	18.6	18.0
(d50,µm):	9.8	3.7	40.0	9.4	7.5	7.4	7.4
Agglomerates (W/ Binder)							
Ash, %	3.74	2.90	6.80	4.91	3.98	3.77	3.54
Total Sulfur,%	2.72	2.56	3.09	3.39	2.80	2.70	2.57
Pyritic Sulfur, %	0.39	0.36	0.89	0.93	0.43	0.33	0.21
Heating Value, Btu/lb	13,617	13,748	13,475	13,755	13,906	13,964	14,013
lb Ash/MMBtu	2.75	2.11	5.05	3.57	2.86	2.70	2.53
lb SO2/MMBtu	4.00	3.72	4.59	4.93	4.03	3.87	3.67
lb Pyritic SO2/MMBtu	0.57	0.52	1.32	1.35	0.61	0.48	0.30
Overall Performance							
Energy Recovery, %	85.3	85.6	86.7	99.3	98.5	93.2	93.0
Ash Reduction, %	79.3	84.1	41.7	72.8	77.4	82.1	83.0
SO2 Reduction, %	47.9	51.4	11.3	25.1	25.3	45.0	46.7
Pyritic SO2 Reduction, %	86.2	87.4	31.1	48.1	64.4	84.8	90.3

during the bench-scale test program and resulted in the development of the selective grinding system. The effectiveness of this grinding system at liberating coal from mineral matter was first demonstrated during the POC testing of the Illinois No. 6 coal. The addition of a spiral separator into the recycle loop, as described in Section 10, allowed for the removal of concentrated minerals, which increased the effectiveness even more.

Different slurry solids concentrations were investigated and it was concluded that solids concentrations of 18 to 20 percent could be used without a noticeable decrease in process performance. Slurry viscosity characteristics of two-stage ground coal had limited the solids concentration in the feed to the high-shear reactor during bench-scale testing. This parameter was re-examined during the POC testing of the Illinois No. 6 coal, when selectively ground coal showed improved rheology characteristics. Rheology test results of micronized (-20μm) coal are presented in Appendix D.

Bridging liquid (heptane) addition ranges were established for specific particle size distributions, ash, and solids concentrations of the feed coal. Too little bridging liquid resulted in floc-like agglomerates, some of which could pass through the tailings screen. Too much bridging liquid resulted in large agglomerates that would cake into lumps requiring longer steaming and making the discharge of the final product from the low-shear reactor difficult.

Binder addition affected the growth cycle operation and the characteristics of the final agglomerate product. Too much binder resulted in large and hard agglomerates which hindered bridging liquid diffusion during steam stripping and caused clumping. Too little binder addition resulted in soft agglomerates which caused blinding of the primary tailings screen or passed through the screen. Additionally, the low binder agglomerates tended to disintegrate during the steaming and final product drainage cycles.

Three different combinations of high-shear reactor impellers were tested during the initial POC testing of Illinois No. 6 coal. The combination that gave the best microagglomerates was used throughout the rest of the test program. It was not feasible to test the effects of different impeller speeds or high-shear reactor residence times as originally intended with the Illinois No.6 seam coal since the maximum speed and residence time that could be obtained were required for agglomeration. Variation of the impeller speeds was tested with the other two coals.

8.4.2 Upper Freeport and Pittsburgh Coals

The Upper Freeport and Pittsburgh coals were tested in the same manner as the Illinois coal. The test matrix and result summaries for the Upper Freeport coal are presented in Tables 8-4 and 8-5, respectively. The test matrix and result summaries for the Pittsburgh coal are presented in Tables 8-6 and 8-7.

The results of testing the process variables with the two coals yielded no surprises compared to those obtained with the Illinois coal. Major differences were in the high-shear residence times and in steaming requirements.

The Upper Freeport (UF) coal was the easiest to agglomerate, producing successful agglomerates with the least specific mixing energy in the high-shear reactor. Lower energy requirements allowed increased throughput at the high-shear mixer to capacities at maximum impeller speed of 1.75 tph for UF coal and 1.25 tph for the Pittsburgh coal, compared to 0.5 tph for Illinois coal. A detailed treatment of high-shear reactor residence time and impeller speed requirements was included in Section 6.3.

Compared to the Illinois coal, a decreasing amount of steam was required to strip and recover the heptane bridging liquid from Pittsburgh and Freeport agglomerates. This reduction was due to experience gained by the operators to control the steam stripping step.

Table 8-4
UPPPER FREEPORT SEAM COAL
TEST MATRIX

Grinding Circuit					Agglomeration Circuit								
					HSR			LSR		Steaming		Asphalt
Serial No.	Run Number	Grind Type	Spiral Used	Slurry Solids(%)	Residence Time(Sec)	Bridging Liquid(1)	Impeller Speed(rpm)	Impeller Speed(rpm)	Growth Time(Min)	Rate(2) (Lb/Ton)	Time (mins)	Conc (%) in Agglomerates
1	40201	Selective	No	18.3	60	28.1	1800	100	10	1940	85	2.31
2	40401	Selective	No	18.2	60	23.3	1800	100	10	1241	64	3.45
3	40801	Two Stage	No	17.8	60	22.5	1800	100	20	1386	60	2.73
4	40802	Two Stage	No	17.8	60	21.2	1800	100	10	1823	77	2.64
5	41102	One Stage	No	16.1	60	8.5	1800	100	10	751	45	2.07
6	41201	One Stage	No	14.3	60	9.0	1800	100	10	ND	ND	2.66
7	42202	Selective	Yes	12.6	60	31.7	1800	100	10	2099	65	3.09
8	42203	Selective	Yes	12.6	60	26.6	1800	100	10	ND	ND	3.22
9	42304	Selective	Yes	13.3	60	35.9	1800	100	5	1659	55	2.40
10	42305	Selective	Yes	13.3	60	33.0	1800	100	10	2531	73	2.86
11	42401	Selective	Yes	13.3	60	26.0	1800	100	10	2276	75	3.19
12	42402	Selective	Yes	13.3	60	27.2	1800	100	10	1757	65	3.73
13	42403	Selective	Yes	13.3	60	25.9	1800	100	10	1750	64	3.32
14	42502	Selective	Yes	16.7	60	23.8	1800	100	10	ND	ND	2.45
15	43203	Selective	Yes	18.4	105	23.7	1300	100	10	927	60	1.39
16	43301	Selective	Yes	16.7	60	25.1	1300	100	10	1495	60	2.29
17	43302	Selective	Yes	16.7	60	21.2	1500	100	10	ND	ND	0.36
18	43303	Selective	Yes	16.7	60	22.0	1500	100	10	2162	65	2.11

(1) Weight percent of dry coal feed
(2) Steam (25 psig) lb/ton of coal
ND Not Determined

Table 8-5
AGGLOMERATION PERFORMANCE SUMMARY
UPPER FREEPORT SEAM COAL
(Dry Basis)

Test Program **Performance Data Origin** **Grind Type:**	**Bench-Scale** **Mean Value** **Two-Stage**	**Bench-Scale** **Best Run** **Lab Grind**	**POC** **Mean Value** **Single-Stage**	**POC** **Mean Value** **Two-Stage**	**POC** **Mean Value** **Selective**	**POC** **Mean Value** **Selective with Spiral**	**POC** **Best Run** **Selective with Spiral**
GRIND SIZE (d90,µm):			103	24.6	15.9	14.6	N.R.
(d50,µm):	4.2	2.9	25.9	7.9	5.5	5.1	N.R.
Agglomerates (W/ Binder)							
Ash, %	8.17	8.51	12.45	10.37	6.78	5.90	5.48
Total Sulfur,%	1.58	1.40	3.15	2.89	1.77	1.32	1.35
Pyritic Sulfur, %	0.87	0.38	2.39	2.05	0.88	0.52	0.52
Heating Value, Btu/lb	14,058	14,002	13,569	13,953	14,611	14,766	14,855
lb Ash/MMBtu	5.81	6.08	9.17	7.43	4.64	4.00	3.69
lb SO2/MMBtu	2.25	2.00	4.64	4.14	2.42	1.79	1.82
lb Pyritic SO2/MMBtu	1.24	0.54	3.52	2.93	1.20	0.70	0.70
Overall Performance							
Energy Recovery, %	92.1	92.4	97.8	99.1	99.1	88.9	94.7
Ash Reduction, %	94.0	93.7	51.7	64.7	73.6	79.6	81.7
SO2 Reduction, %	68.3	71.8	19.0	37.0	50.4	72.1	71.9
Pyritic SO2 Reduction, %	80.9	91.6	21.4	38.5	63.1	83.7	85.9

N.R. = Not Recorded

Table 8-6

PITTSBURGH SEAM COAL

TEST MATRIX

Grinding Circuit					Agglomeration Circuit							
					HSR			LSR		Steaming		Asphalt
Serial No.	Run Number	Grind Type	Spiral Used	Slurry Solids(%)	Residence Time(Sec)	Bridging Liquid(1)	Impeller Speed(rpm)	Impeller Speed(rpm)	Growth Time(Min)	Rate(2) (Lb/Ton)	Time (mins)	Conc (%) in Agglomerates
1	43901	Selective	Yes	13.2	120	23.7	1800	100	10	2615	85	2.37
2	43902	Selective	Yes	13.2	120	20.5	1800	100	10	1785	63	2.35
3	44001	Selective	Yes	13.2	120	30.0	1800	100	10	2710	67	1.71
4	44002	Two Stage	No	17.4	120	14.4	1800	100	10	1368	60	2.35
5	44301	Two Stage	No	17.4	120	27.7	1800	117	20	1440	60	2.34
6	44302	One Stage	No	16.0	120	18.5	1800	100	10	2175	70	2.18
7	44303	One Stage	No	16.0	120	7.8	1800	100	10	1285	45	2.10
8	44601	Selective	No	17.8	75	24.2	1800	106	5	1484	70	2.92
9	44602	Selective	No	17.8	84	22.3	1800	106	5	1458	70	2.39
10	44603	Selective	No	17.8	84	22.0	1800	106	5	1657	75	2.32
11	44703	Selective	No	19.1	84	27.0	1800	113	5	1234	60	2.56
12	44802	Selective	No	19.0	84	26.4	1800	143	5	1339	65	2.50
13	44803	Selective	No	19.0	84	26.6	1800	106	5	1962	75	2.08
14	45001	Selective	No	18.9	84	23.8	1800	106	5	1561	75	2.51
15	45101	Selective	No	17.3	84	20.2	1800	106	5	1357	60	2.73
16	45102	Selective	No	17.3	84	18.8	1800	106	15	1479	65	2.56
17	45201	Selective	No	17.3	84	26.6	1800	106	5	1573	70	3.87
18	45402	Selective	No	19.1	84	21.4	1800	106	5	1489	80	3.13

(1) Weight percent of dry coal feed
(2) Steam (25 psig) lb/ton of coal
ND Not Determined

Table 8-7
AGGLOMERATION PERFORMANCE SUMMARY
PITTSBURGH SEAM COAL
(Dry Basis)

Test Program **Performance Data Origin**	**Bench-Scale(1)** **Mean Value**	**Bench-Scale(1)** **Best Run**	**POC(2)** **Mean Value**	**POC(2)** **Mean Value**	**POC(2)** **Mean Value**	**POC(2)** **Mean Value**	**POC(2)** **Best Run**
GRIND TYPE:	**Two-Stage**	**Lab Grid**	**Single-Stage**	**Two-Stage**	**Selective**	**Selective with Spiral**	**Selective with Spiral**
GRIND SIZE (d90,µm):			135	28.5	17.5	15.6	15.6
(d50,µm):	12.1	3.7	26.4	10.0	6.4	5.7	5.7
Agglomerates (W/ Binder)							
Ash, %	5.01	3.35	6.37	6.06	3.81	3.44	3.40
Total Sulfur,%	3.78	3.48	2.33	2.42	1.86	1.63	1.59
Pyritic Sulfur, %	1.41	0.86	0.83	1.01	0.39	0.33	0.34
Heating Value, Btu/lb	13,877	14,133	14,266	14,324	14,735	14,795	14,793
lb Ash/MMBtu	3.61	2.37	4.46	4.23	2.59	2.33	2.30
lb SO2/MMBtu	5.45	4.92	3.26	3.38	2.53	2.20	2.15
lb Pyritic SO2/MMBtu	2.03	1.22	1.16	1.40	0.53	0.45	0.46
Overall Performance							
Energy Recovery, %	92.5	93.2	88.9	99.9	99.8	90.0	90.0
Ash Reduction, %	92.1	94.8	30.1	50.6	57.9	66.3	66.7
SO2 Reduction, %	50.7	55.4	4.0	15.2	19.0	37.9	39.3
Pyritic SO2 Reduction, %	70.4	82.3	41.9	28.2	51.6	75.6	74.9

(1) The bench-scale test program used Pittsburgh (Ohio No. 8) seam coal from Belmont County, Ohio.
(2) The POC test facility used prewashed Pittsburgh seam coal from Monongalia Co., WV.

8.5 FLOAT/SINK ANALYSIS

Float/sink analysis was used to assess the degree of liberation of pyrite and ash-forming minerals in the feed to agglomeration. Samples from all three coals were taken as the crushed 1/4 inch x 0 feed to the grinding circuit and also the micronized (-20μm) product from the selective grinding circuit. The 1/4 inch x 0 coal samples were screened at 28 mesh prior to the analysis. The +28 mesh material was analyzed using the static float/sink technique. The minus 28 mesh material and all micronized samples were analyzed using a centrifugal float/sink technique. The full results of this analysis are presented in Appendix B.

Agglomeration itself is likely the best measure of coal and pyrite liberation. Formation of microagglomerates in the high-shear reactor is not a static process, rather, microagglomerates are formed and destroyed quite rapidly in the high-shear mixing zone of the reactor. The repeated mechanical action of agglomeration and dispersion is probably a better method of breaking up unselective aggregates than chemical dispersants. This was concluded from the bench-scale agglomeration tests with different dispersants. Also, since agglomeration is a surface characteristic separation, it is less affected by changes in coal density.

8.5.1 Summary of Float/Sink Results

The float/sink analysis presented in Appendix B were combined into a composite analysis for each coal. A summary of the results for an equivalent "float," "middlings," and "sink" fraction of both 1/4 inch x 0 and selectively ground coals (d_{90} of 20μ) are presented in Tables 8-8 through 8-10.

A review of the differences between the 1/4 inch x 0 composite float/sink and the centrifugal float/sink of the selectively ground coal yields some interesting observations.

The most interesting observation is the apparent loss of "1.3 float" material (material with a density of less than 1.3 specific

Table 8-8
LIBERATION OF PYRITE AND ASH-FORMING MINERALS FROM ILLINOIS NO. 6 COAL USING CENTRIFUGAL FLOAT/SINK ANALYSIS

ILLINOIS NO. 6 COAL
Composite 1/4-inch x 0

Sp. Gr.	Wt %	Ash %	S %	Pyr S %
+1.3	35.58	3.21	2.57	0.38
1.3 x 1.8	53.41	12.05	3.13	1.12
-1.8	11.00	68.98	13.46	11.95

Cumulative Wt %	Cumulative Ash %	Cumulative Total S %	Cumulative Pyr S %
35.58	3.21	2.57	0.38
88.99	8.51	2.91	0.82
99.99	15.17	4.07	2.05

ILLINOIS NO. 6 COAL
Selectively Ground Coal

Sp. Gr.	Wt %	Ash %	S %	Pyr S %
+1.3	5.23	2.74	2.31	0.09
1.3 x 1.8	84.37	7.91	2.45	0.27
-1.8	10.40	69.13	6.91	4.54

Cumulative Wt %	Cumulative Ash %	Cumulative Total S %	Cumulative Pyr S %
5.23	2.74	2.31	0.09
89.60	7.61	2.44	0.26
100.00	14.01	2.91	0.70

Table 8-9

LIBERATION OF PYRITE AND ASH-FORMING MINERALS FROM UPPER FREEPORT COAL USING CENTRIFUGAL FLOAT/SINK ANALYSIS

UPPER FREEPORT COAL
Composite 1/4-inch x 0

Sp. Gr.	Wt %	Ash %	S %	Pyr S %
+1.3	35.63	3.04	0.97	0.34
1.3 x 1.8	44.02	15.97	2.22	1.62
-1.8	20.35	71.78	11.54	10.43

Cumulative Wt %	Cumulative Ash %	Cumulative Total S %	Cumulative Pyr S %
35.63	3.04	0.97	0.34
79.65	10.19	1.66	1.05
100.00	22.72	3.67	2.96

UPPER FREEPORT COAL
Selectively Ground Coal

Sp. Gr.	Wt %	Ash %	S %	Pyr S %
+1.3	13.79	2.21	0.86	0.08
1.3 x 1.8	65.21	7.85	1.23	0.31
-1.8	21.00	68.72	9.29	7.83

Cumulative Wt %	Cumulative Ash %	Cumulative Total S %	Cumulative Pyr S %
13.79	2.21	0.86	0.08
79.00	6.87	1.17	0.27
100.00	19.85	2.87	1.86

Table 8-10

LIBERATION OF PYRITE AND ASH-FORMING MINERALS FROM PITTSBURGH COAL USING CENTRIFUGAL FLOAT/SINK ANALYSIS

PITTSBURGH COAL
Composite 1/4-inch x 0

Sp. Gr.	Wt %	Ash %	S %	Pyr S %	Cumulative Wt %	Cumulative Ash %	Cumulative Total S %	Cumulative Pyr S %
+1.3	60.24	3.81	1.69	0.34	60.24	3.81	1.69	0.34
1.3 x 1.8	35.51	12.45	3.01	1.72	95.75	7.01	2.18	0.85
-1.8	4.25	67.33	9.34	8.35	100.00	9.58	2.48	1.17

PITTSBURGH COAL
Selectively Ground Coal

Sp. Gr.	Wt %	Ash %	S %	Pyr S %	Cumulative Wt %	Cumulative Ash %	Cumulative Total S %	Cumulative Pyr S %
+1.3	17.89	1.55	1.50	0.04	17.89	1.55	1.50	0.04
1.3 x 1.8	76.38	4.75	1.65	0.17	94.27	4.14	1.62	0.15
-1.8	5.73	67.92	10.12	7.89	100.00	7.80	2.11	0.59

gravity). The basic premise of physical coal cleaning to low levels of ash and pyritic sulfur content with high energy recoveries is that minerals must be liberated from the coal. This can be accomplished through intensive grinding of the coal. The major aim is to break into smaller sizes those particles of "middlings" which contain both coal and ash-forming minerals.

Comparison of the float/sink data of the coals before and after grinding actually shows a decrease of the 1.3 float fraction. This effect is especially noticeable for the Pittsburgh coal. Less than 18 percent of the material floated at 1.3 specific gravity after grinding, while over 60 percent of the material had floated at 1.3 specific gravity at the 1/4-inch x 0 composite.

Others have found similar results. J.T. Riley et al. from Western Kentucky University found that grinding of coal increases the density of the individual coal macerals[1]. Riley has characterized coals at various levels of grinding, down to 10µm (d_{50}) using centrifugal float/sink techniques. He has also measured the surface area of the coal before and after milling. Analysis of these results led Riley to the conclusion that there is a progressive collapse of the macropore structure of the individual coal macerals leading to an increase in coal density.

An alternate explanation for the loss of float material may be found in the centrifugal float/sink technique used to produce maceral isolation studies. E.J. Hippo of Southern Illinois University has extensive experience at separating individual macerals of coal using an ultra-high gravity centrifuge. Hippo grinds coal to 1µ size, demineralizes the micronized product with an acid wash, and then separates the remaining material using a centrifuge that produces a gradient of 16,000 g. This gravity gradient is much greater than that used by others for centrifugal float/sink analysis of coal (1,700 to 2,000 g). Hippo did not find a significant change in the

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[1] J.T. Riley, W.G. Lloyd, K.W. Kuehn, and D.L. Withers, "Coal Density Changes during Stirred-Ball Attritor Milling," Journal of Coal Quality, Vol.9, No.1, Jan-Mar, 1990, pp 12-17

density of coal macerals ground to micron-sized levels using his technique.

Alternate explanations for the apparent loss of 1.3 float material are advanced by F.J. Smit et al. in a study of micronized coals for DOE's Pittsburgh Energy Technology Center[2]. It was noted that there was no apparent liberation of clean coal in samples ground finer than 44 microns. Again, there was a marked loss of material floating at 1.3 gravity with decreasing grind size. It was theorized that this loss of material could be due to: 1) unselective aggregation of extremely fine particles, 2) liberation of exinite (spores and resins) from vitrinite, and 3) loss of unwetted voids as closed pores become exposed due to grinding which increases the particle density.

8.5.2 Comparison of Float/Sink Data with Agglomeration Results

The median agglomeration results were compared to the float/sink results. This was done to assess how close the agglomeration process results came to the bench mark results that could be achieved by specific gravity washabilitiy methods. This comparison is presented in spite of the controversy associated with centrifugal float/sink analysis for fine coal.

Figures 8-7 and 8-8 show the ash content to energy recovery data of agglomeration compared with centrifugal float/sink data for the Illinois No. 6 and Upper Freeport coals.

Figures 8-9 and 8-10 show the pyritic sulfur reduction to energy recovery data of agglomeration and centrifugal float/sink data for Illinois No. 6 and Upper Freeport seam coals.

8.6 PETROGRAPHIC ANALYSIS

Petrographic analysis was used to evaluate the level of pyrite liberation achieved by grinding. Secondary petrographic methods were

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[2] F.J. Smit, J.R. Odekirk, L.K. Baltich, "Ultra-Fine Coal Characterization," Final Report DOE/PC/72007-T14 to the U.S. Department of Energy, NTIS No. DE90011936, December 1988

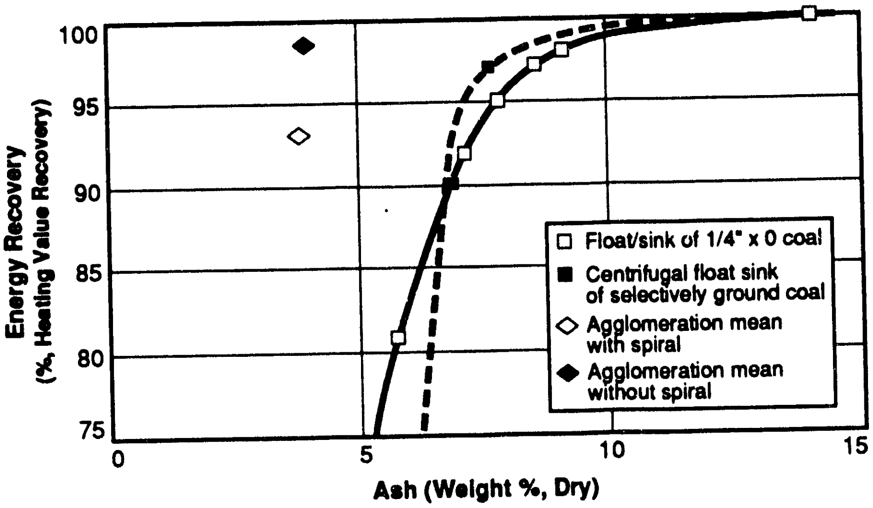

Figure 8-7 Illinois No. 6 Coal — Ash Content vs. Energy Recovery

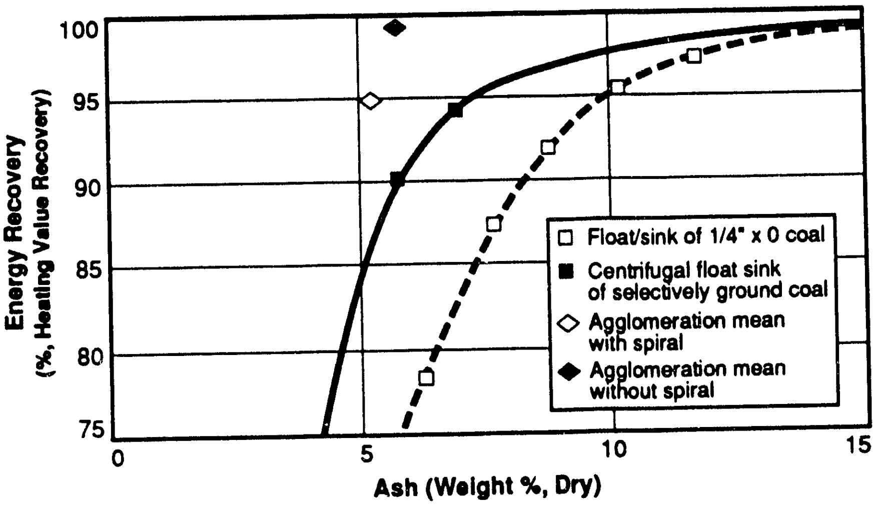

Figure 8-8 Upper Freeport Coal — Ash Content vs. Energy Recovery

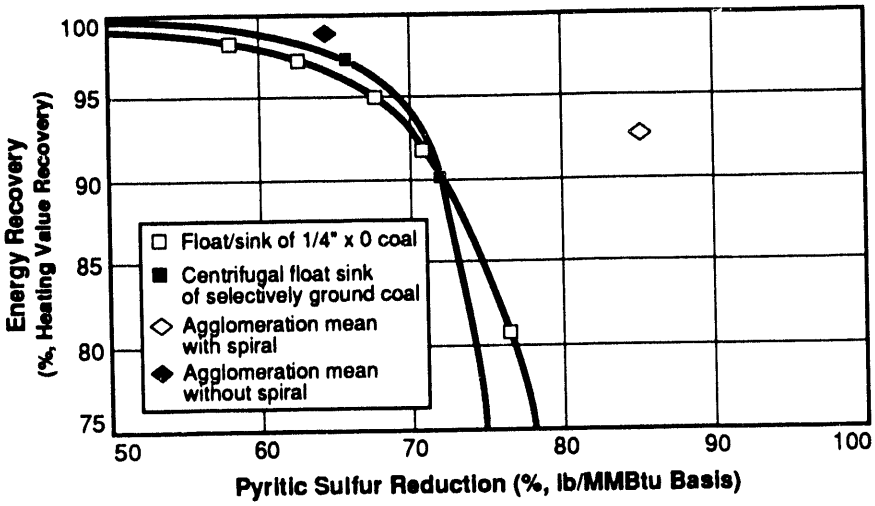

Figure 8-9 Illinois No. 6 Coal — Pyritic Sulfur Reduction vs. Energy Recovery

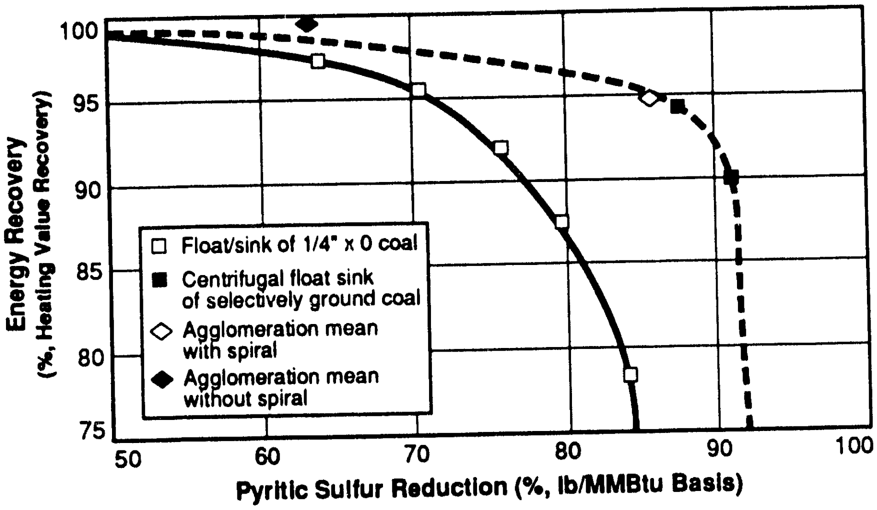

Figure 8-10 Upper Freeport Coal — Pyritic Sulfur Reduction vs. Energy Recovery

used to detect pyrite in the agglomerated clean coal. The petrography work was carried out by Mr. Ralph Grey of Process & Energy Management Corporation (P&EMC).

8.6.1 Petrographic Analysis of Illinois No. 6 Coal

Samples from the Illinois No. 6 coal were petrographically analyzed to measure and assign pyrite particles to the free, semi-locked, or locked category. The samples were collected when the grinding circuit was operated in the selective grinding mode. The samples were:

- Ball mill product
- Feed to agglomeration
- Agglomerates
- Fine grinding mill product

Each coal sample was mixed with a plastic mounting media, pressed into a cylindrical pellet, then ground and polished for microscopic observation. A total of 500 to 1000 pyrite particles per sample were measured. The results of the petrographic analysis are listed by frequency in Table 8-11; typical photomicrographs of the coal samples are shown in Figures 8-11 through 8-14.

Ball Mill Product. The photomicrographs presented in Figure 8-11 and Table 8-11 show a sample of the ball mill product. The figures indicate that the sample was largely made up of coarse particles. The coal was relatively high in mineral matter and pyritic sulfur. The predominant minerals were mostly clay with calcite, quartz, and pyrite. Pyrite occurred as free and semi-locked as indicated in Table 8-11 which shows the frequency distribution by size and association of pyrite particles as determined under the microscope. Some of the pyrite particles were relatively coarse, up to 150 microns in the longest dimension. However, the coarsest pyrite particle found during the count was 45 microns. The largest coal particle was about 200 microns in size.

Table 8-11

ILLINOIS NO. 6 COAL PETROGRAPHY
(Frequency Basis)

Sample No. Run No. Sample Name	**312201 35401 1-Stage Grind**			**312204 35401 Feed to Agglomeration**			**311302 35401 Agglomerates**			**312203 35401 Fine Grind Product**		
Mean Size Microns	**Free**	**Semi-Locked**	**Locked**	**Free**	**Semi-Locked**	**Locked**	**Free**	**Semi-Locked**	**Locked**	**Free**	**Semi-Locked**	**Locked**
1	26.2	21.5	6.8	10.6	41.2	19.9	7.2	40.9	24.8	16.9	23.6	6.1
3	10.5	9.7	3.3	4.4	13.9	4.0	2.6	11.4	5.1	14.4	12.7	1.1
5	4.4	4.7	1.6	0.7	3.3	0.7	0.7	3.9	1.6	5.3	7.0	0.8
7	2.8	1.7	0.8	--	1.1	0.2	--	0.9	0.2	3.4	3.4	0.4
9	0.8	0.7	0.5	--	--	--	--	0.5	0.2	1.7	0.6	--
11	0.4	0.7	0.1	--	--	--	--	--	--	0.4	0.4	--
13	0.2	0.7	--	--	--	--	--	--	--	0.2	0.4	0.2
15	0.3	0.4	--	--	--	--	--	--	--	0.2	--	--
17	0.1	0.2	0.2	--	--	--	--	--	--	0.2	--	--
19	0.2	--	--	--	--	--	--	--	--	0.2	--	--
21	--	--	0.1	--	--	--	--	--	--	--	0.2	0.2
23	0.1	--	--	--	--	--	--	--	--	--	--	--
25	--	--	--	--	--	--	--	--	--	--	--	--
27	--	--	--	--	--	--	--	--	--	--	--	--
29	--	--	--	--	--	--	--	--	--	--	--	--
31	0.1	--	--	--	--	--	--	--	--	--	--	--
33	--	--	--	--	--	--	--	--	--	--	--	--
35	--	--	--	--	--	--	--	--	--	--	--	--
37	--	--	--	--	--	--	--	--	--	--	--	--
39	0.1	--	0.1	--	--	--	--	--	--	--	--	--
41	--	--	--	--	--	--	--	--	--	--	--	--
43	--	--	--	--	--	--	--	--	--	--	--	--
45	--	--	--	--	--	--	--	--	--	--	--	--
47	--	0.1	--	--	--	--	--	--	--	--	--	--
Subtotal	46.2	40.3	13.5	15.7	59.5	24.8	10.5	57.6	31.9	42.9	48.3	8.8
Total			100.0			100.0			100.0			100.0

Figure 8-11 Photomicrograph of 1-Stage Grinding Product #312201, Illinois No. 6 Coal. Reflected Light in Oil, X 756

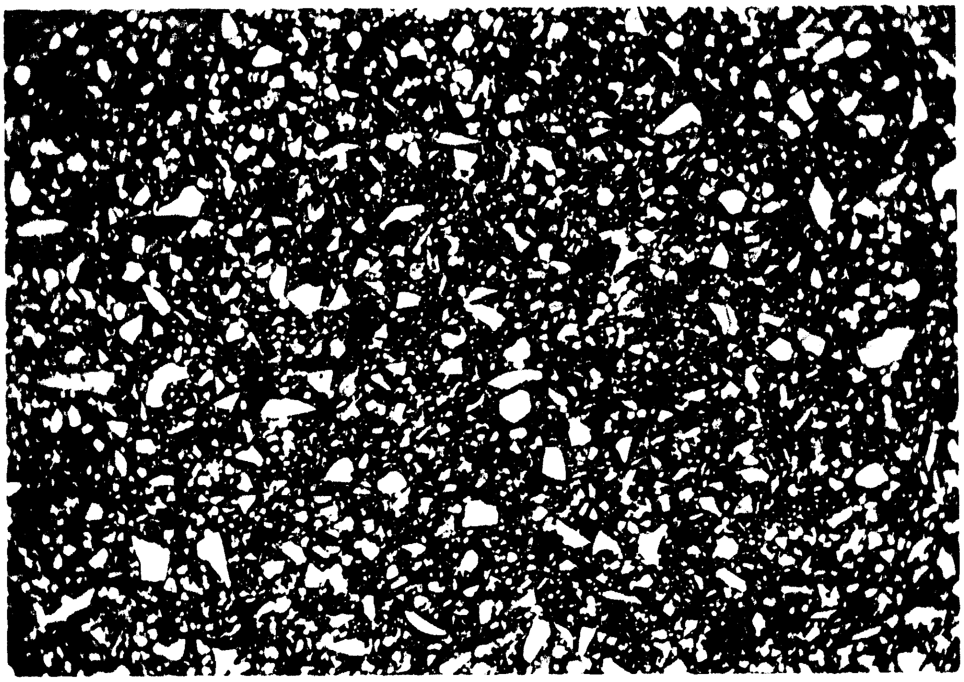

Figure 8-12 Photomicrograph of Agglomeration Feed #313802, Illinois No. 6 Coal. Reflected Light in Oil, X 315

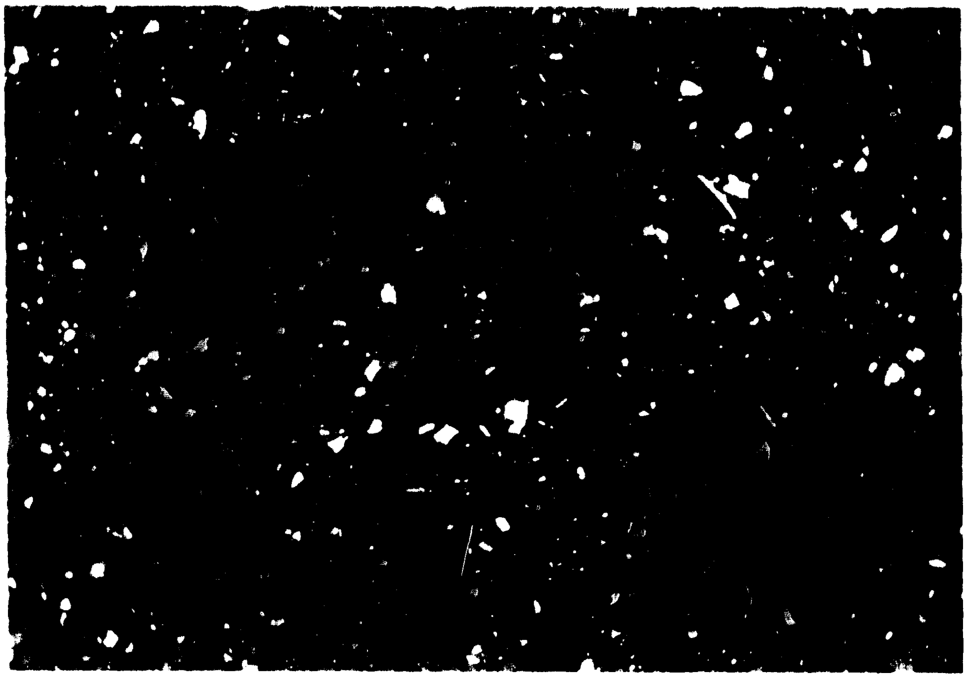

Figure 8-13 Photomicrograph of Agglomerates #312201, Illinois No. 6 Coal. Reflected Light in Oil, X 315

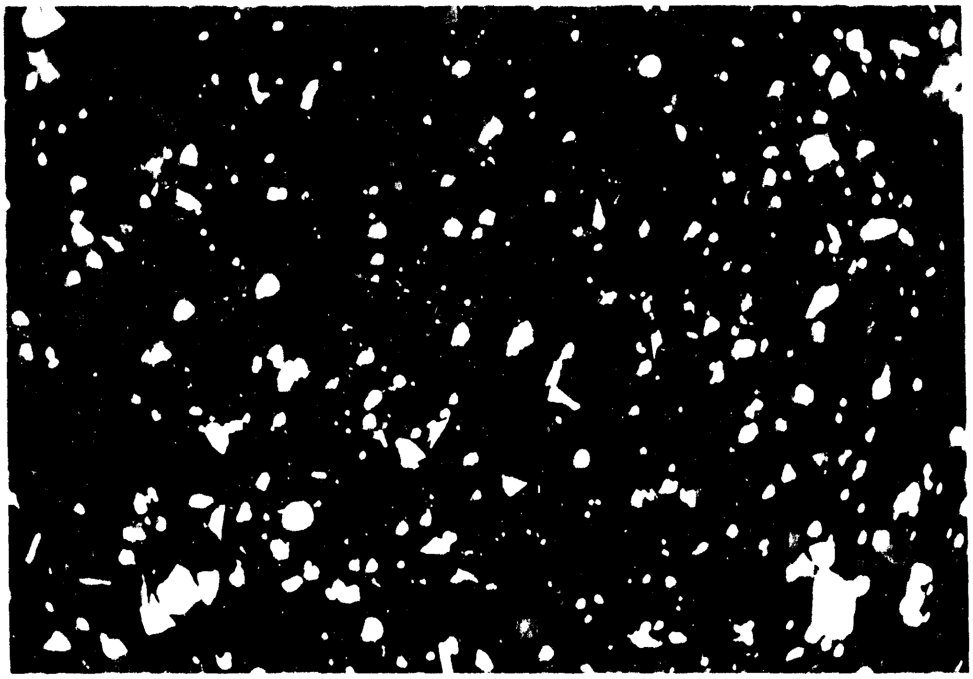

Figure 8-14 Photomicrograph of Finely Ground Recirculation Loop Material #312203, Illinois No. 6 Coal. Reflected Light in Oil, X 315

Feed to Agglomeration. Most of the pyrite was 7 microns or smaller, and was semi-locked or locked, as seen in Figure 8-12 and Table 8-11. Individual particles of coal were frequently irregular in shape. A relatively large amount of very fine clay was observed.

Agglomerates. The agglomerated coal had few pyrite and other mineral matter, as shown in Table 8-11 and Figure 8-13. The pyrite particles were all less than 10 microns in size and occurred most frequently in the semi-locked and locked categories.

Fine Grinding Mill Product. The product from the fine grinding mill included the grinding circuit recirculation material. The pyrite and mineral matter content of this material was higher than in any other process to stream analyzed. The petrographer noted that the sample was probably of a refuse or reject stream. Most of the pyrite was 21 microns or less and was concentrated in the semi-locked and free categories. A photomicrograph is shown in Figure 8-14 and pyrite occurrences are indicated in Table 8-11.

8.6.2 Petrographic Analysis of Upper Freeport Coal

Samples from the Upper Freeport seam coal were treated as the samples from Illinois No. 6 coal.

The results of the analysis are shown on Table 8-12 and in Figures 8-15 through 8-18.

Ball Mill Product. Figures 8-15 and 8-16 show photomicrographs of the ball mill product. The coal was of medium volatile bituminous rank, and contained a large amount of ash-forming minerals and pyrite. The predominant minerals were clay, pyrite, quartz, and calcite, with the pyrite mainly of the free and semi-locked categories. Some coal particles were up to 650 microns in the longest dimension and pyrite particles were as coarse as 240 microns. Most of the pyrite was in the 20-50 micron size range. The sample also contained bone coal, carbonaceous shale, and shale. Pyrite occurred as framboidal and (euhedral) crystals, as dense irregular masses, and even as framboidal colonies.

Table 8-12
UPPER FREEPORT COAL PETROGRAPHY
(Frequency Basis)

Sample No. Run No. Sample Name	330802 40402 1-Stage Grind			330804 40402 Fine Grind Product			330806 40402 Feed to Agglomeration		
Mean Size Microns	**Free**	**Semi-Locked**	**Locked**	**Free**	**Semi-Locked**	**Locked**	**Free**	**Semi-Locked**	**Locked**
1.0	40.0	20.4	11.5	29.3	14.4	2.6	32.7	41.8	8.3
3.0	8.9	5.2	3.8	24.0	6.7	0.9	6.9	6.6	0.8
5.0	4.3	3.0	1.6	12.4	2.2	0.1	1.5	1.2	0.2
7.0	2.0	2.0	0.8	5.1	0.5	--	--	--	--
9.0	0.7	0.5	0.4	0.8	0.1	--	--	--	--
11.0	0.2	0.5	0.2	0.3	--	--	--	--	--
13.0	0.3	0.3	0.2	0.3	--	--	--	--	--
15.0	0.1	0.2	--	0.1	--	--	--	--	--
17.0	0.1	--	--	0.1	--	--	--	--	--
19.0	0.1	0.1	--	--	--	--	--	--	--
21.0	0.3	0.3	0.1	--	--	--	--	--	--
23.0	--	--	--	--	--	--	--	--	--
25.0	0.1	--	--	--	--	--	--	--	--
27.0	--	0.1	--	--	--	--	--	--	--
29.0	0.1	0.1	0.1	--	--	--	--	--	--
31.0	--	--	--	--	--	--	--	--	--
33.0	0.1	--	--	--	--	--	--	--	--
35.0	0.1	0.1	--	--	--	--	--	--	--
39.0	--	--	--	0.1	--	--	--	--	--
41.0	--	--	--	--	--	--	--	--	--
43.0	--	--	--	--	--	--	--	--	--
45.0	--	0.1	--	--	--	--	--	--	--
Subtotal	57.4	32.9	18.7	72.5	23.9	3.6	41.1	49.6	9.3
Total			109.0			100.0			100.0

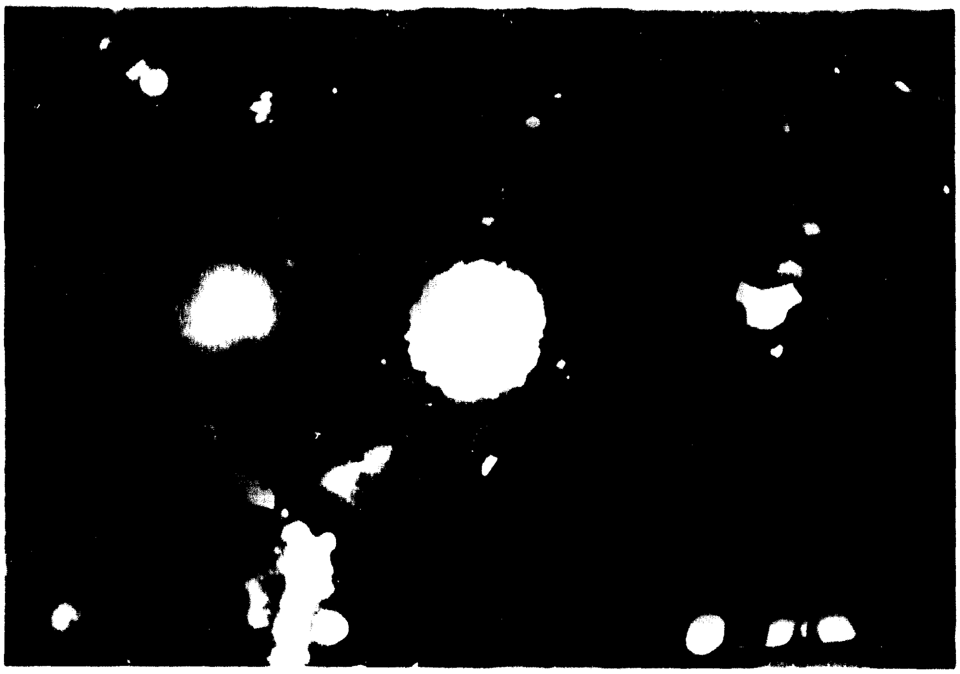

Figure 8-15 Photomicrograph of 1-Stage Grinding Product Showing Framboidal Pyrite #330802, Upper Freeport Coal. Reflected Light in Oil, X 756

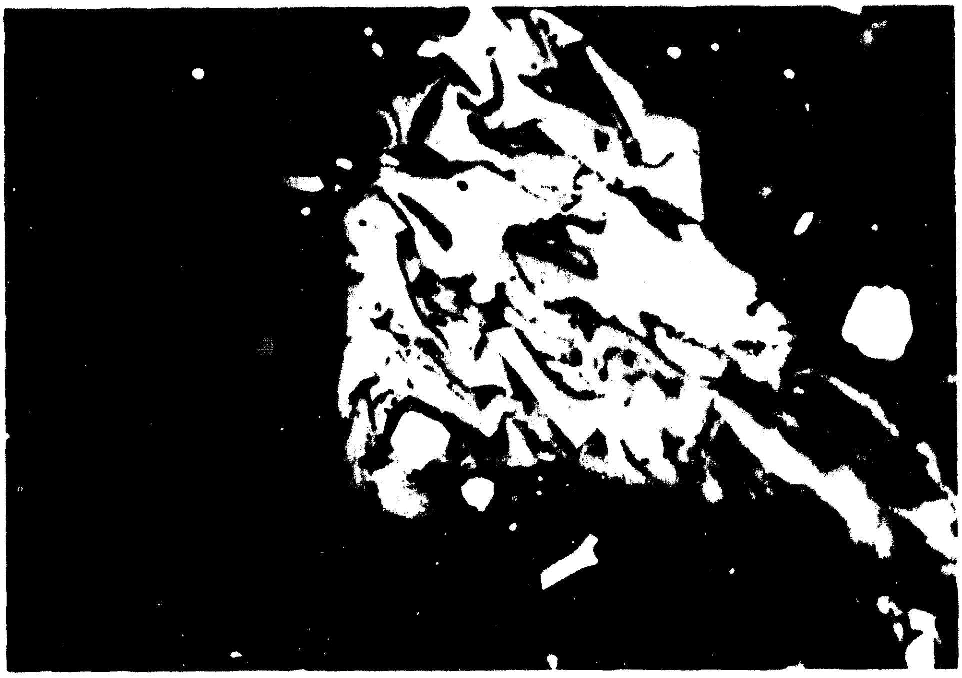

Figure 8-16 Photomicrograph of 1-Stage Grinding Product Showing Euhedral Pyrite #330802, Upper Freeport Coal. Reflected Light in Oil, X 756

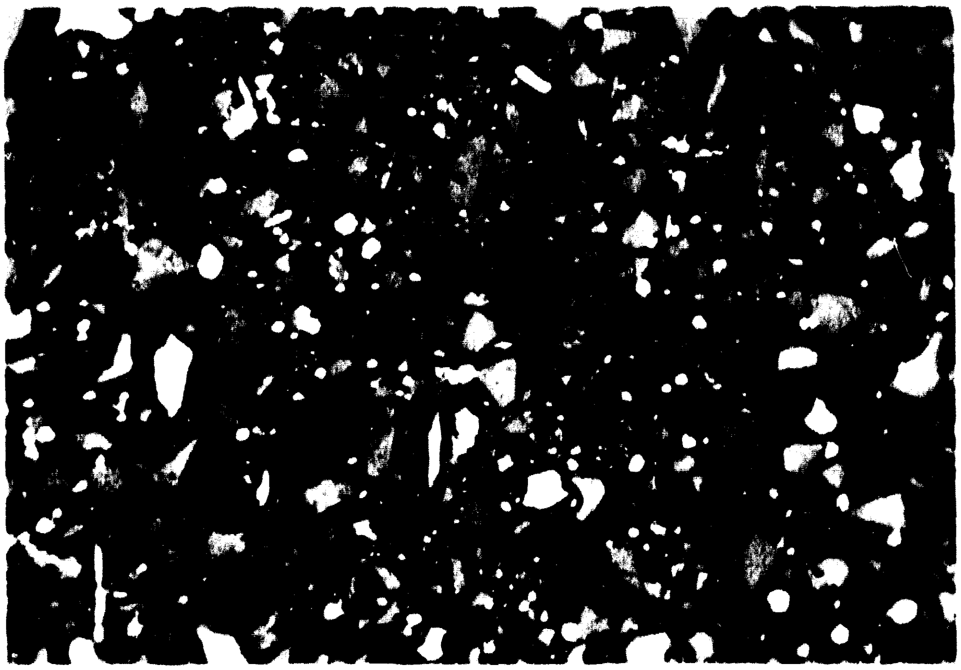

Figure 8-17 Photomicrograph of Feed to Agglomeration #330806, Upper Freeport Coal. Reflected Light in Oil, X 756

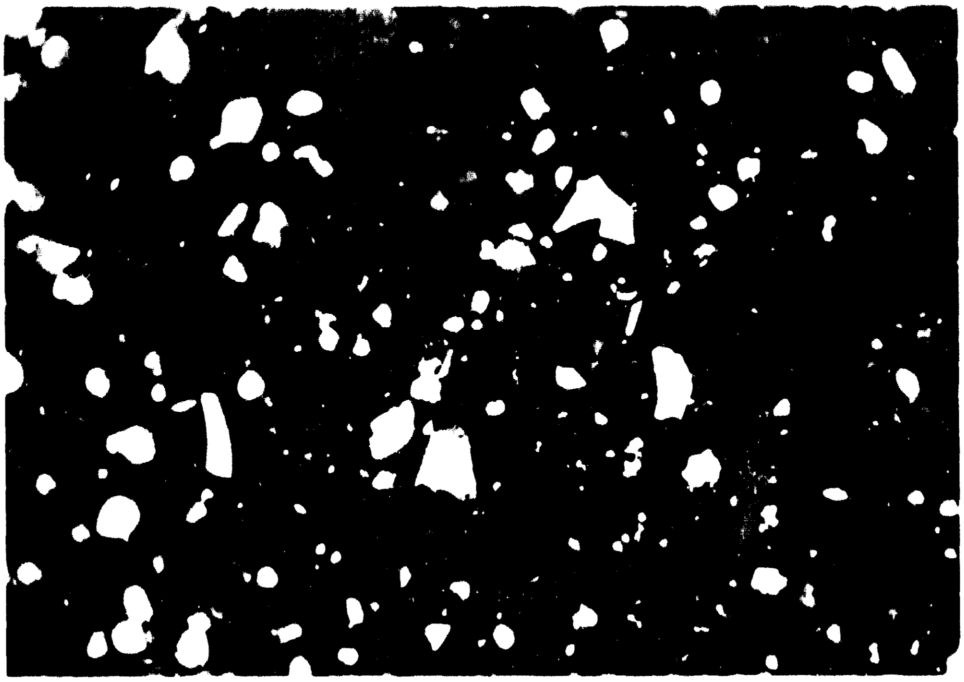

Figure 8-18 Photomicrograph of Fine Grinding Mill Product #330804, Upper Freeport Coal. Reflected Light in Oil, X 756

Feed to Agglomeration. The ground product had particles of up to 30 microns. Most of the particles were below 5 microns. The pyrite occurred mainly as free or semi-locked and was about 1 micron or less in size (Table 8-12 and Figure 8-17).

Fine Grinding Mill Product. The product included the recirculating material. It contained a few particles of up to 540 microns but most particles were smaller than 20 microns. The sample showed an abundance of pyrite and clay minerals as well as quartz and calcite, indicating the accumulation of minerals in the recycle loop of the grinding circuit. Most of the pyrite particles were smaller than 10 microns and were free and semi-locked, with a trace amount of locked pyrite (Table 8-12 and Figure 8-18).

8.6.3 Petrographic Analysis of Pittsburgh Coal

Only samples of the selectively ground feed to agglomeration, and of the agglomerates (or clean coal product) were examined. Six hundred pyrite particles were counted in each sample, sized, and categorized. The results of the analysis are listed in Table 8-13, and photomicrographs of the samples are shown in Figures 8-19 and 8-20.

The feed to agglomeration as well as the agglomerates displayed many of the same characteristics. The coal was of the high volatile bituminous rank with an abundance of vitrinite. Most of the pyrite was euhedral or fragments of framboids. Most particles were less than 10 microns with an abundance of fines of less than 5 microns. A few coal particles were up to 600 microns in the longest dimension but seldom exceeded 40 microns. Most of the pyrite ranged from 0.5 to 1 micron and was generally symmetrical in shape. The largest pyrite particles in the samples were about 9 microns.

The agglomeration feed had free pyrite and other ash-forming minerals (clay and quartz) as shown on Table 8-13 and in Figures 8-19 and 8-20. Most of the pyrite in the agglomerates was classified as either locked or semi-locked.

Table 8-13

PITTSBURGH COAL PETROGRAPHY

(Frequency Basis)

Sample No.	**339001**			**339002**		
Run No.	**45402**			**45402**		
Sample Name	**Feed to Agglomeration**			**Agglomerates**		
Mean Size (microns)	Free	Semi-Locked	Locked	Free	Semi-Locked	Locked
1.0	32.3	35.8	11.7	13.5	42.5	19.8
3.0	5.7	10.0	1.1	3.8	12.2	4.0
5.0	1.3	1.5	0.2	0.7	2.1	0.3
7.0	0.2	0.2	0.0	0.2	0.7	0.2
Subtotal	39.5	47.5	13.0	18.2	57.5	24.3
Total			100.0			100.0

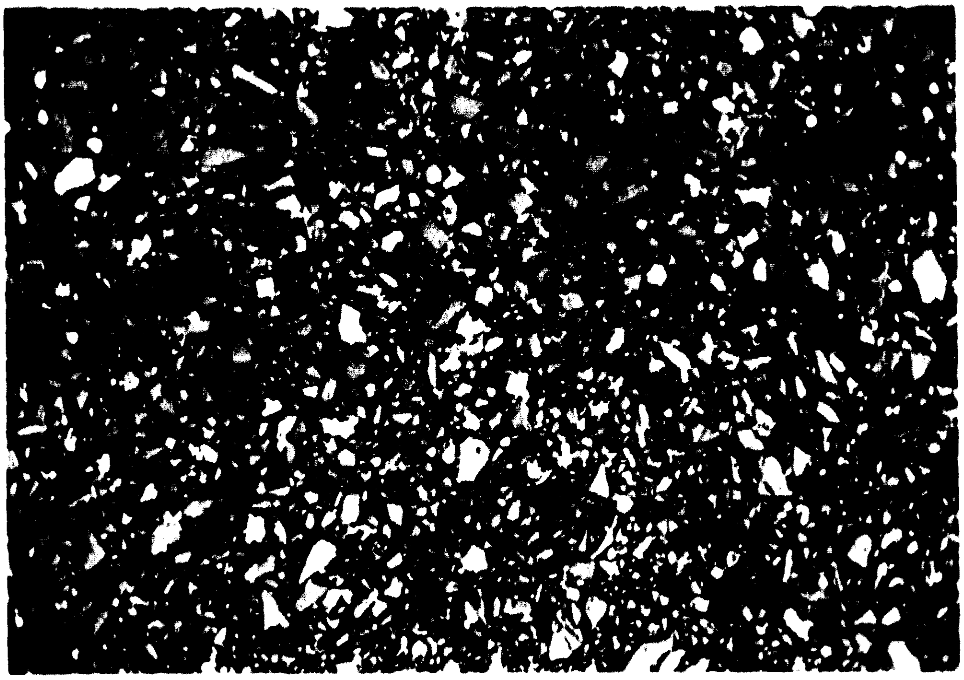

Figure 8-19 Photomicrograph of Agglomeration Feed #33901, Pittsburgh Coal. Reflected Light in Oil, X 315

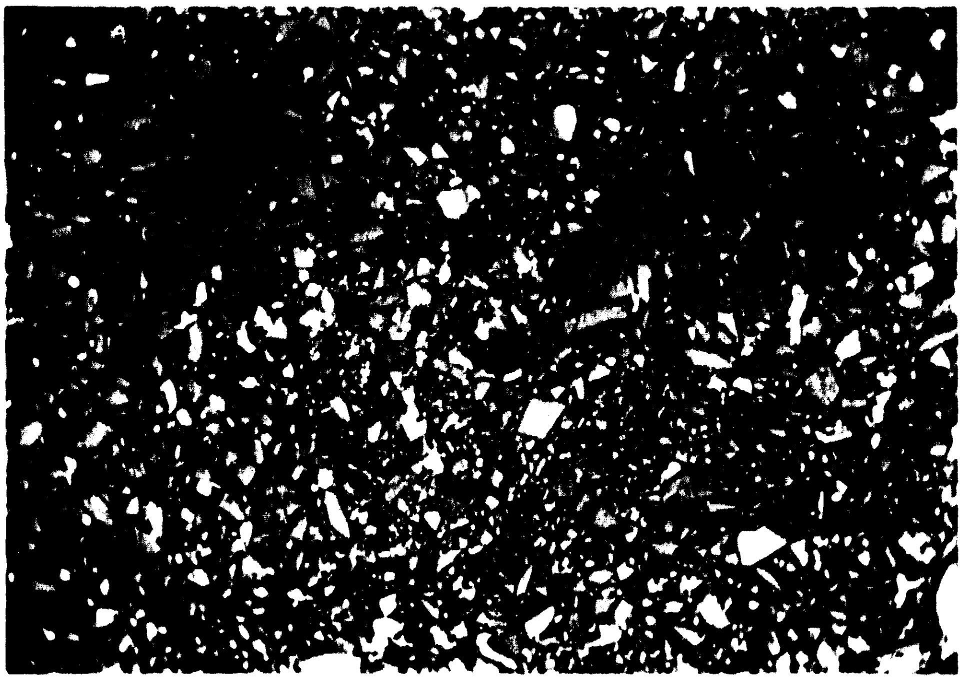

Figure 8-20 Photomicrograph off Agglomerates #339002, Pittsburgh Coal. Reflected Light in Oil, X 315

8.6.4 Determination of Pyrite Liberation and Pyrite in the Clean Coal Using Petrography

One of the uncertainties in the spherical agglomeration of coal using heptane was the amount of pyrite that could be rejected by the process. This concern was based on reports that pyrite is hydrophobic like coal and will agglomerate. The POC tests proved that spherical agglomeration can reject pyrite.

Using petrography it was possible to analyze "free," "semi-locked," or "locked" pyrite was agglomerated or rejected by the process. To use the petrographer's frequency count data for such an analysis, it is first necessary to convert the data to a weight basis. A report of pyrite occurrences can then be made by category for each sample on a weight basis. This report for agglomeration feeds and agglomerates is presented in Table 8-14. The pyrite occurrences in Table 8-14 clearly show that clean coal agglomerates contained less pyrite classified as free, or completely liberated from coal.

A further analysis of the different liberation categories of pyrite can be made by factoring in the amount of pyrite actually removed. This analysis is shown in Table 8-15, where the weight of pyritic sulfur for every 100 lb of feed coal was distributed among the three liberation categories. The same was done for the pyritic sulfur in the agglomerates (taking into account the yield of clean coal) and the removal of pyritic sulfur according to each pyrite liberation category was determined.

Table 8-15 indicates that most of the pyrite removed was from the free and semi-locked categories. Pyrite of the locked category remained largely with the coal. It is interesting to note that a significant amount of the removed pyrite was from the semi-locked category. The high ash content of the tailings would indicate that the method is rather subjective and should not be used for quantitative analysis.

Table 8-14

PYRITE OCCURRENCES BY CATEGORY

(By Weight)

Category	Free	Semi-Locked	Locked
Illinois No. 6(1)			
Feed to Agglomeration	11.2	71.7	17.1
Agglomerates	7.3	67.1	25.6
Upper Freeport(2)			
Feed to Agglomeration	48.9	44.5	6.6
Agglomerates	3.9	68.6	27.5
Pittsburgh(3)			
Feed to Agglomeration	39.9	53.7	6.4
Agglomerates	19.7	63.4	16.9

(1) From Run No. 35401

(2) From Run No. 40501

(3) From Run No. 45402

Table 8-15

PYRITE REDUCTION BY LIBERATION CATEGORY

(Basis – 100 lb of Coal Feed to Agglomeration)

CATEGORY	Free	Semi-Locked	Locked	Total
Illinois No. 6(1)				
Pyritic Sulfur in Feed, lb	0.082	0.523	0.125	0.730
Pyritic Sulfur in Agglomerates, lb	0.016	0.145	0.055	0.216
Pyrite Removed, %	12.8	73.6	13.6	100.0
Upper Freeport(2)				
Pyritic Sulfur in Feed, lb	1.051	0.957	0.142	2.150
Pyritic Sulfur in Agglomerates, lb	0.031	0.542	0.217	0.790
Pyrite Removed, %	75.0	30.5	-5.5	100.0
Pittsburgh(3)				
Pyritic Sulfur in Feed, lb	0.271	0.365	0.044	0.680
Pyritic Sulfur in Agglomerates, lb	0.069	0.223	0.060	0.352
Pyrite Removed, %	61.5	43.2	-4.7	100.0

(1) From Run No. 35401
Yield = 86.4%, Feed Pyritic Sulfur = 0.73%
Agglomerate Pyritic Sulfur = 0.25%

(2) From Run No. 40501
Yield = 82.3%, Feed Pyritic Sulfur = 2.15%
Agglomerate Pyritic Sulfur = 0.96%

(3) From Run No. 45402
Yield = 95.1%, Feed Pyritic Sulfur = 0.68%
Agglomerate Pyritic Sulfur = 0.37%

Section 9

COAL CHARACTERISTICS

9.1 DETAILED LABORATORY ANALYSIS OF AGGLOMERATION FEED, AGGLOMERATES, AND REFUSE

This section presents laboratory analysis conducted on the feed coal, POC test product and waste streams. Tables and figures are included at the end of this section.

9.1.1 Feed Coal Heating Value Relationships

Size analysis for the ROM coals tested at the POC plant are presented in Table 9-1.

Heating value relationships for the coals used in the POC test program are presented below. Linear regression was performed on laboratory ash and heating value data. Included with the best-fit line equations are R^2 values and the number of observations used for the regressions:

Illinois No. 6 Seam Coal

HV (Btu/Lb) = 14496 - 163.2 * Ash (%)
R2 = 0.996
26 Observations

Upper Freeport Seam Coal

HV (Btu/Lb) = 15797 - 182.5 * Ash (%)
R2 = 0.996
42 Observations

Pittsburgh Seam Coal

HV (Btu/Lb) = 15372 - 181.3 * Ash (%)
R2 = 0.997
23 Observations

Table 9-1
SIZE ANALYSIS FOR ROM ILLINOIS, UPPER FREEPORT, AND PITTSBURGH SEAM COAL

Size (mm)	Weight Percent	Percent Ash	Percent Sulfur	HV Btu/Lb	Weight Percent	Percent Ash	Percent Sulfur	H.V. Btu/Lb
Illinois No. 6								
6.3 x .600	74.52	14.18	4.18	12308	74.52	14.18	4.18	12308
.600 x .250	10.44	15.50	3.36	12135	84.96	14.34	4.08	12287
.250 x .150	4.59	17.56	3.27	11854	89.55	14.51	4.04	12265
.150 x .075	4.98	19.81	3.46	11455	94.53	14.79	4.01	12222
.075 x .045	1.70	22.39	3.71	11086	96.23	14.92	4.00	12202
minus 0.045	3.77	27.49	3.33	10299	100.00	15.39	3.98	12130
Upper Freeport Seam								
6.3 x .600	63.10	26.17	4.20	11140	63.10	26.17	4.20	11140
.600 x .250	17.38	17.74	3.18	12735	80.48	24.35	3.98	11484
.250 x .150	6.11	18.46	3.13	12570	86.59	23.93	3.92	11561
.150 x .075	6.49	19.04	2.97	12311	93.08	23.59	3.85	11613
.075 x .045	2.28	19.49	2.75	12257	95.36	23.49	3.83	11629
minus 0.045	4.64	19.93	2.40	12207	100.00	23.33	3.76	11656
Pittsburgh Seam								
plus 6.3	9.12	16.66	2.54	12580	9.12	16.66	2.54	12580
6.3 x .600	65.63	8.90	2.36	13915	74.75	9.85	2.38	13752
.600 x .250	10.75	8.55	2.39	13771	85.50	9.68	2.38	13754
.250 x .150	4.11	9.27	2.55	13618	89.61	9.66	2.39	13748
.150 x .075	4.66	9.98	2.71	13570	94.27	9.68	2.41	13739
.075 x .045	1.81	11.33	2.94	13313	96.08	9.71	2.42	13731
minus 0.045	3.92	12.67	2.58	13097	100.00	9.83	2.42	13707

9.1.2 Handleability

Agglomerates, up to 3/8-inch in size, were produced using asphalt as binder. The amount of asphalt binder addition was minimized to achieve just enough agglomerate strength for transport, decrease the loss of coal fines during shipping, and reduce the moisture content of the fine clean coal. Moisture contents of the clean coal agglomerates were consistently below 30 percent moisture and reached 20 percent.

Agglomerates from each of the coals produced at the POC facility were sent to Combustion Engineering (CE) in Windsor, Connecticut, for a DOE-sponsored combustion testing program. The most significant observation by CE during their tests with the Illinois No. 6 agglomerates was difficulty in feeding the agglomerates into their pilot-scale boiler. After pulverizing and drying in a bowl mill, fine coal was pneumatically conveyed with hot air into the boiler. Occasionally, pulverized agglomerates would stick to the burner feed tubes in the boiler. It is believed that deposits were caused by asphalt in the agglomerates which tended to melt and fuse to the hot tubes. It is not believed that the fineness of the agglomerated coal was the cause of the problem. The scale problem has not been observed with other ultrafine coals burned in the same facility. Scale deposits were sampled and analysis is pending[1]. It is unlikely that in commercial use only agglomerates would be fired. In a mixture with other clean coals the described behavior may completely disappear.

9.1.3 Grindability

Grindability indices of the test coals delivered to CQ Inc. are presented in Table 9-2. The Pittsburgh and Illinois coals show similar grindability characteristics. The Upper Freeport coal, with the highest as-received Hardgrove index (74), was noticeably easier to micronize than the other two coals.

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[1] Telephone conversation with Oscar Chow, Combustion Engineering, July 23, 1990

Table 9-2
ROM AND CLEAN COAL AGGLOMERATE PROXIMATE AND ULTIMATE ANALYSIS

	Illinois Nº 6 Seam Coal		Upper Freeport Seam Coal		Pittsburgh Seam Coal	
	ROM Parent	Agglomerates	ROM Parent	Agglomerates	ROM Parent	Agglomerates
Proximate Analysis						
Ash	15.14	3.63	23.26	5.52	9.62	3.80
Total Moisture	8.33	28.84	3.73	22.18	3.87	19.38
Volatile Matter	38.33	42.44	24.76	32.24	37.13	42.21
Fixed Carbon	47.56	53.93	51.98	62.24	53.13	53.99
Total Sulfur	3.65	2.56	3.77	1.35	2.63	1.89
Organic Sulfur	1.98	2.17	1.40	0.76	1.14	1.46
Pyritic Sulfur	1.57	0.27	2.33	0.52	1.46	0.37
Sulfate Sulfur	0.10	0.12	0.04	0.07	0.03	0.06
Ultimate Analysis						
Carbon	67.87	76.44	65.27	82.54	75.54	83.46
Hydrogen	4.82	5.16	3.82	5.24	5.02	5.11
Nitrogen	1.29	1.39	1.18	1.44	1.40	1.31
Chlorine	0.07	0.02	0.11	ND	0.05	ND
Sulfur	3.65	2.56	3.77	1.34	2.63	1.80
Ash	15.14	3.63	23.26	5.52	9.62	3.80
Oxygen (Dif)	7.16	10.80	2.70	3.92	5.79	4.52
Heating Value	12222	14281	11764	14845	13635	14843
Grindability Index	54	150+	74	NA	57	NA
Free Swelling Index	3.5	1.0	8.0	ND	7.5	ND

NA = Not Available at this time
ND = Not Determined

Grindability testing of agglomerates was not done during the POC test program. It was not expected that agglomerates would present any resistance to grinding. Tests conducted by Combustion Engineering on the Illinois No. 6 agglomerates confirmed this expectation[2].

9.1.4 Proximate and Ultimate Analysis

Proximate and ultimate analysis are presented as Table 9-2.

Proximate Analysis. On an ash-free basis, there was no significant change noted in the fixed carbon or volatile matter in the agglomerates when compared with the ROM coal. Organic sulfur remained in the product with reductions in pyritic sulfur.

Ultimate Analysis. The ultimate analysis shows unchanged levels of carbon, hydrogen, and nitrogen, for both agglomerates and ROM coal when examined on an ash-free basis. A significant increase in oxygen is noted for the Illinois coal. This is expected since the Illinois coal is lower in rank than the other two coals and that it is therefore more susceptible to oxidation, especially when grinding increases the surface area that can be subjected to oxidation. An independent ultimate analysis conducted by Combustion Engineering, on a pooled sample of Illinois coal, showed a similar rise in the oxygen level. Oxygen levels for the other two coals were unchanged between the ROM samples and the agglomerates.

Free Swelling Index. The free swelling index in the Illinois clean coal agglomerates dropped from 3.5 for the parent coal to 1 for the agglomerates. The other coals were not tested.

9.1.5 Heptane Concentration in the Agglomerates

Results of residual heptane analysis carried out during the bench-scale and the proof-of-concept (POC) test program are presented as Table 9-3. Heptane extraction during the bench-scale tests were

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[2] Telephone conversation with Oscar Chow, Combustion Engineering, July 23, 1990

Table 9-3

HEPTANE ANALYSIS IN BENCH-TEST AND POC TESTS

Coal	Run No.	Sample Origin	Sample Type	Steam Time	Extract Concentration	Sample Minimum Detection Limit	Sample Heptane Concentration
Illinois No. 6	I6C1	Bench Scale	Agglomerates	13	0.1 ppm	4.7 ppm	4.7 ppm
	I6C2	Bench Scale	Agglomerates	12	0.1 ppm	3.6 ppm	3.6 ppm
	I6C3	Bench Scale	Agglomerates	12	0.1 ppm	3.9 ppm	3.9 ppm
	I6C4	Bench Scale	Agglomerates	12	0.4 ppm	2.4 ppm	9.4 ppm
	I6C6	Bench Scale	Agglomerates	12	0.1 ppm	4.0 ppm	4.0 ppm
	39001	POC Facility	Agglomerates	83	0.01 %	0.18 %	0.18 %
	39001	POC Facility	Primary Tails	83	0.01 %	0.02 %	0.02 %
	39002	POC Facility	Agglomerates	95	0.01 %	0.18 %	0.18 %
	39002	POC Facility	Primary Tails	95	0.01 %	0.03 %	0.03 %
	39602	POC Facility	Agglomerates	60	0.01 %	0.19 %	0.38 %
	39702	POC Facility	Agglomerates	69	0.01 %	0.19 %	0.38 %
	39801	POC Facility	Agglomerates	ND	0.01 %	0.19 %	0.76 %
Upper Freeport	UC1	Bench Scale	Agglomerates	13	0.5 ppm	2.1 ppm	10.3 ppm
	UC2	Bench Scale	Agglomerates	13	0.2 ppm	2.8 ppm	5.7 ppm
	UC3	Bench Scale	Agglomerates	14	0.2 ppm	1.9 ppm	3.8 ppm
	UC4	Bench Scale	Agglomerates	16	0.2 ppm	2.6 ppm	5.1 ppm
	UC6	Bench Scale	Agglomerates	12	0.2 ppm	3.0 ppm	5.9 ppm
	UC7	Bench Scale	Agglomerates	12	0.1 ppm	3.3 ppm	3.3 ppm
Pittsburgh	P8C4	Bench Scale	Agglomerates	9	0.1 ppm	2.7 ppm	2.7 ppm
	P8C5	Bench Scale	Agglomerates	10	0.1 ppm	4.2 ppm	4.2 ppm
	P8C6	Bench Scale	Agglomerates	13	0.2 ppm	3.3 ppm	6.5 ppm
	P8C7	Bench Scale	Agglomerates	12	0.1 ppm	3.9 ppm	3.9 ppm
	P8C8	Bench Scale	Agglomerates	10	0.2 ppm	2.1 ppm	4.2 ppm
	44601	POC Facility	Agglomerates	70	0.01 %	0.22 %	<.22 %
	44601	POC Facility	Primary Tails	70	0.01 %	0.02 %	<.02 %
	44603	POC Facility	Agglomerates	75	0.01 %	0.31 %	<.31 %
	44603	POC Facility	Primary Tails	75	0.01 %	0.02 %	<.02 %
	45402	POC Facility	Agglomerates	80	0.01 %	0.32 %	<.32 %
	45402	POC Facility	Primary Tails	80	0.01 %	0.02 %	<.02 %

10,000 ppm = 1 %
Bench-Scale Extract MDL = 0.1 ppm
POC Extract MDL = 0.1 ppm

done with samples analyzed by gas chromatography (GC) by ENCOTEC Inc. of Ann Arbor, MI. POC test samples were extracted by Exportech with GC analysis performed by the University of Pittsburgh Applied Research Center (UPARC). Since the procedures used during the two project phases differed, they will be discussed separately below for comparison purposes.

Bench-Scale Program Procedure. Agglomerates were weighed into tared sintered glass extraction thimbles and placed in a Soxhelet reflux apparatus. Samples were refluxed with dry toluene for 4 to 16 hours until the condensed toluene passing through the sample no longer contained visible traces of asphalt. It was reasonable to assume that when all the asphalt had been visibly removed from the sample, all the heptane was also extracted into the toluene.

Toluene extracts were distilled to separate the asphalt from the toluene and the lighter hydrocarbons. Samples of toluene extracts were removed, placed in sealed containers, and sent to GC for analysis.

The toluene was analyzed for traces of heptane using GC with flame ionization detection (FID). Extracts were directly injected into the GC. Samples of heptane used in the bench-scale tests were shot onto the GC to determine the peaks corresponding to the isomers in the heptane sample. Extracts were shot and a summation of all the C7 peaks was reported. It was assumed in the analysis that the isomers of heptane all gave similar responses to the flame ionization detector.

Samples of agglomerates from the bench-scale tests had less than 11 ppm heptane.

POC Test Program Procedure. Samples of agglomerates and tailings were sealed after collection, labeled, and sent to the laboratory for extraction. A split of the agglomerate samples were taken and dried to determine the moisture content. Twenty grams of agglomerates and 200 grams of the primary tailings were extracted in toluene. Samples of agglomerates were placed in a thimble and refluxed with 500 ml of toluene in a Soxhelet extractor for 6-24 hours. The 200 ml samples of primary tails were added directly to the refluxing toluene. Agglomerate samples were refluxed for longer periods until the extract draining from the Soxhelet thimble was colorless (no longer contained asphalt). After extraction, samples were sent to UPARC for GC analysis.

The UPARC hydrocarbon analysis procedure used a GC with a flame ionization detector. Samples containing both heptane and asphalt were injected into the GC. The detection limits reported for the analysis of the extract was 100 ppm heptane. Since 20 gram samples were extracted into 500 ml of toluene, the sample preparation dilution factor for agglomerates was 20. This translated to heptane detection limits of 2,000 ppm on a weight basis to the original sample. Primary tails detection limits were lower at 200 ppm.

Three samples of agglomerates were reported by UPARC to have between 3,800 and 7,600 ppm heptane. This is significantly higher than the levels observed in the bench-scale test samples. Most other POC samples are reported at or below the detection limit. For those samples reported at the detection limit, little information can be gained. Since the heptane detection limit is high (about 3,000 ppm in many cases), all that can be said about these samples is that they contain less than 3,000 ppm heptane.

Procedures have yet to be developed for residual heptane determination. At UPARC, low grade Fisher analytical reagent toluene was used for the extractions without any blanks run to check for interferences. No coal samples were spiked with heptane to determine the extraction recovery performance. For the samples that were found to have high levels of heptane, no confirmation of the values was made by a different analytical technique. Operation of the POC plant had concluded before these deficiencies had been found.

Since the residual heptane data from POC test samples are suspect, the bench-scale test data are a better indicator of heptane concentrations in steamed agglomerates. Levels of heptane in the bench-scale test agglomerates averaged about 5 ppm.

9.1.6 Trace Element Analysis

Trace element analysis conducted on POC samples are tabulated in Table 9-4.

9.2 COMBUSTION CHARACTERISTICS OF CLEANED COAL

Combustion testing of agglomerates produced in the POC facility is currently (July 1990) being conducted by Combustion Engineering of Windsor, Connecticut. In the absence of actual data, approximations of ash slagging and fouling potential can be made using empirical indices and by examining combustion data from the same coals cleaned to similar mineral and ash compositions.

Ash behavior in coal-fired boilers can be classified into two major categories, slagging and fouling, defined below:

- Slagging is fused deposits or resolidified molten material that forms on furnace walls or surfaces exposed to radiant heat transfer or excessively high gas temperatures[3]. Critical ash characteristics affect the flow of molten slag from wet bottom furnaces. The most significant factors influencing slag deposits are viscosity at boiler operating temperature and deposit bonding strength.
- Fouling relates to bonded (sintered or cemented) ash deposits that form on convective heat transfer surfaces, such as superheater and reheater tubes and on furnace walls[4]. Fouling is predominantly the result of sodium oxide in the ash which vaporizes in the high temperature regions of the boiler and recondenses on the cooler convective heating surfaces later in the exhaust duct system.

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[3] J. G. Singer, editor, **Combustion Fossil Power Systems**, Combustion Engineering, Windsor Connecticut, 1981, pp 3-5, 3-6

[4] Ibid

Table 9-4
ROM, CLEAN COAL AGGLOMERATE, AND PRIMARY TAILS TRACE ELEMENT ANALYSIS

	Illinois No. 6 Coal			Upper Freeport Coal	Pittsburgh Seam Coal
	ROM Parent	**Agglomerates**	**Primary Tails**	**ROM Parent**	**ROM Parent**
Arsenic	36	53	54	26	4
Barium	84	24	435	595	980
Boron	130	180	120	105	9
Bromine	8	7	7	4	11
Cadmium	0.4	0.6	0.3	0.7	1
Chlorine	60	200	30	1,100	500
Chromium	30	80	120	160	76
Cobalt	22	12	20	42	62
Copper	44	41	45	138	160
Fluorine	107	180	210	93	14
Germanium	7	4	9	10	22
Lead	31	31	55	61	23
Lithium	910	580	610	840	380
Manganese	81	880	30	760	500
Mercury	0.04	0.02	0.04	0.04	0
Molybdenum	9	16	5	11	9
Nickel	39	60	49	79	220
Selenium		3	4	2	16
Sodium	0.55	13,250	2,294	2,220	4,900
Strontium	180	490	310	315	1,020
Sulfur	10,600	4,200	7,720	9,560	20,100
Vanadium	64	102	90	140	120
Zinc	190	215	310	320	180

Condensed, sticky, Na_2O will collect on superheater or reheater tubes and trap other flyash particles. If not removed by soot blowing, the deposits will clog passages and prevent the boiler from operating at full capacity.

9.2.1 Empirical Fouling and Slagging Indicators

Predictions of boiler behavior of coal ash slag and fouling have been successfully approximated using empirical equations developed from numerous combustion test data. Tables 9-5 and 9-6 list the indices used for the approximation of slagging and fouling deposit behavior with ranges indicating the degree of severity. A brief explanation of the individual indices follows.

Coal Ash Types. Predicting the behavior of coal ash can be made more accurate by dividing coal ashes into two separate types, "Eastern" and "Western." Coal ashes are categorized on the basis of the relative contents of CaO + MgO to Fe_2O_3. Eastern coals are those coals that have a higher incidence of iron oxide than combined calcium and magnesium oxides. Coals with more calcium and magnesium oxides than iron oxide are referred to as western coals. All coals used in this test program are of the eastern type and the following discussion and equations is restricted to that type.

Base-to-Acid Ratio. The base-to-acid (B/A) ratio of coals is defined as:

$$B/A = \frac{Fe_2O_3 + CaO + MgO + Na_2O + K_2O}{SiO_2 + Al_2O_3 + TiO_2}$$

As pure substances, the basic components (in the numerator of the above equation) of ash have low melting points, while the acidic components have higher melting points. From the B/A ratio the fusion point (melting point) of a particular coal ash sample can be roughly predicted. However, the ratio does not take into account that, when mixed together, combinations of acids and basic ash components will combine to yield salts with lower melting points than either of the two original components alone.

Table 9-5

COAL-FIRED BOILER SLAGGING INDICES
BITUMINOUS TYPE COAL ASH*

Index	Formula	Application	Fouling Tendency			
			Low	Medium	High	Severe
Base/Acid Ratio	$\frac{Fe_2+CaO+MgO+Na_2O+K_2O}{SiO_2+Al_2O_3+TiO_2}$	Forms the basis of many empirical indices used for approximating slagging and fouling	<.20	.20-.50	.50-1.0	>1.0
Ash Slagging Potential	(Base/Acid Ratio)* Percent Sulfur in Coal	Common empirical relationship used to predict slagging potential of a fuel	<.6	.6-2.0	2.0-2.6	>2.6
Percent Alkalis	% Ash*(Na_20 +0.659* K_20)/100	Predicts fouling potential based on total coal ash. Includes potassium oxide term	<.30	.45-.60		>.83
Critical Viscosity Temperature, T_{250}	Based on Base/Acid Ratio	Predicts temperature at which slag will still run freely (i.e., viscosity ≤250 poise)	<2600		>2600	

* Coal type determined from ratio of Fe203/(Ca0+Mg0). Coals with ratios greater than 1.0 are considered to have Eastern or bituminous type ash. Coals with ratios less than 1 are classified as having Western or lignite type ash.

Table 9-6

COAL-FIRED BOILER FOULING INDICES
BITUMINOUS TYPE COAL ASH*

Index	**Formula**	**Application**	**Fouling Tendency**			
			Low	**Medium**	**High**	**Severe**
Fouling index (R_f)	(Base/Acid Ratio)*Na_20	Empirical relationship predicting fouling potential of a fuel	<.20	.20-.50	.50-1.0	>1.0
Ash Sodium Oxide	% Na_20 in Ash	Predicts fouling potential of coal ash based on the content of sodium oxide	<.50	.50-1.0	1.0-2.5	>2.5
Percent Alkalis	% Ash*(Na_20 +0.659* K_20)/100	Predicts fouling potential based on total coal ash. Includes potassium oxide term	<.30	.30-.45	.45-.60	>.70

* Coal type determined from ratio of Fe203/(Ca0+Mg0). Coals with ratios greater than 1.0 are considered to have Eastern or bituminous type ash. Coals with ratios less than 1 are classified as having Western or lignite type ash.

Additionally, the base/acid ratio can be used to predict the viscosity of a particular coal ash slag. Since some of the basic components of ash are low melting solids which behave as fluxes, the viscosity (and fusion point) of ash slags is inversely related to B/A ratios.

The base/acid ratio has been used as a slagging indicator,however, the large number of parameters that influence its accuracy restrict its usefulness. The main utility of the base/acid ratio is as the basis of many other ashing and slagging indicators.

The slagging and fouling indicators found below have been proven useful from numerous laboratory and field observations.

Slagging Indicators. The ash slagging potential, (Rs), is defined as:

$$Rs = (\text{Base/Acid Ratio}) * \text{Coal Percent Sulfur}$$

The ash slagging potential (SP) is a common parameter used to predict the degree of slagging expected with a particular fuel. Using the base/acid ratio as its foundation, the SP designates as likely to cause severe fouling those coals with high sulfur contents or high (B/A) ratios.

Empirically, it has been found that coals with high sulfur contents and high B/A ratios cause severe fouling despite the lower viscosity commonly associated with higher B/A ratios. The usefulness of this relationship is explained in part by the fact that coals with high levels of sulfur have high levels of pyritic sulfur. Pyritic sulfur at boiler furnace conditions is readily converted to iron oxide. Increasing iron oxide content in the ash and coal ash slag viscosity are related. Decreased iron oxide also appears in the numerator of the B/A ratio.

Empirically, the silica percentage in ash has been related to the viscosity of coal ash slags. Just as higher levels of Fe_2O_3 have been found experimentally to decrease the viscosity, higher values for SiO_2 also result in higher slag viscosities.

The silica percentage is defined as:

$$Sp = \frac{SiO_2}{SiO_2 + Fe2O_3 + CaO + MgO}$$

Figure 9-1 shows the critical viscosity temperature by plotting the base/acid ratio on the nomograph. The thick black line in the figure represent the summation of numerous empirical data. The nomograph predicts the temperature at which the viscosity of the slag will be 250 poise. This point has been arbitrarily set as the maximum viscosity of a slag that will drain freely from a furnace bottom. Babcock and Wilcox suggests that the critical viscosity temperatures should be below 2600°F to maintain the combustion chamber at a reasonable operating temperature[5].

Fouling Indicators. The fouling Index, R_f, is defined as:

$$R_f = \text{(base/acid ratio)} * \text{percent Na2O in the ash}$$

The empirical fouling index uses the base/acid ratio as its basis with the contribution from sodium oxide being heavily weighted. High levels of fouling sodium oxide are reflected simultaneously in the numerator of the base/acid ratio and the percent sodium oxide in the ash.

The ash percent sodium oxide index examines solely the sodium oxide content of a coal ash sample to determine the fouling potential. Low fouling coals are those with less than 0.5 percent sodium oxide in their ash while coals with greater than 2.5 percent are considered to have severe fouling potential. Both the fouling index and the percent sodium oxide index ignore the quantity of ash in the

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[5] Steam/Its Generation and Use, 39th Edition, Babcock and Wilcox Inc., New York, NY, 1978, pp 15-4 to 15-6

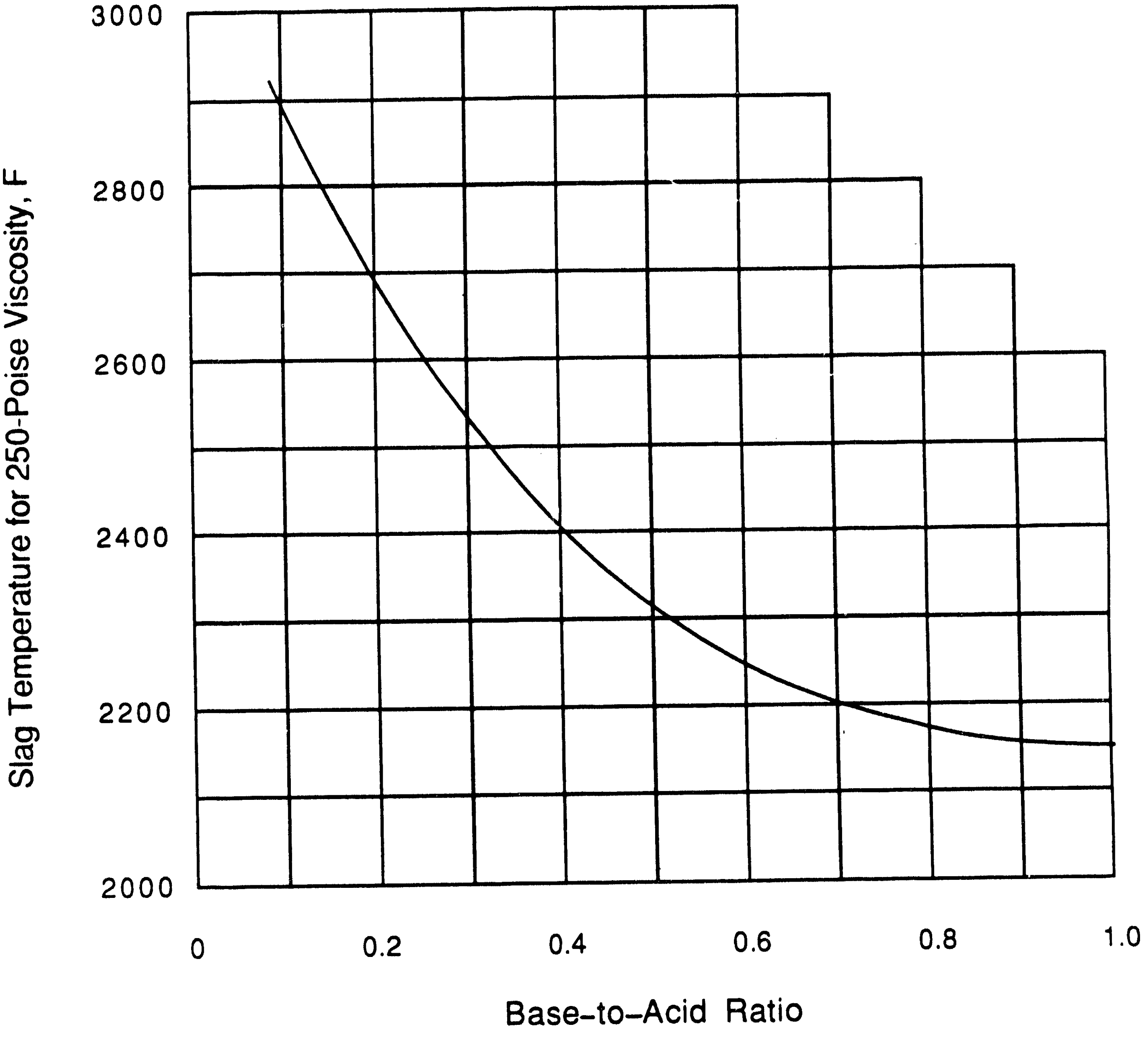

Figure 9-1 Temperature for 250-poise Viscosity vs Base-to-Acid Ratio

Reproduced From Steam/Its Generation and Use, 39th Edition, Babcock and Wilcox Inc., New York, NY, 1978, page 15-5 coal. While these indices are more reliable for coals with higher (15 to 20 percent) ash contents, they do predict the bonding strength of fouling deposits for coals with a wide range of ash contents. Higher concentrations of sodium oxide in fouling deposits are directly related to higher bonding strengths.

The percent alkalis index accounts for the contribution of potassium oxide (which may also sublime in their furnace and can cause fouling problems). This index also factors variation in coal quality giving credit to coals that have reduced ash contents. Since this index takes into account the actual amounts of ash generated, its usefulness can be best applied to approximate the frequency of sootblowing required of the convection tubes.

Percent alkalis is defined as:

$$\text{Percent Alkalis} = \text{Ash (Wt \%)} * (Na_2O + 0.659*K_2O)$$

9.2.2 Summary of Mineral Ash Data

Table 9-7 presents the raw data for the mineral ash compositions for ROM coal and clean coal for the Illinois No. 6, Upper Freeport, and Pittsburgh seam coals. Also included are ash fusion data. Values for the coals produced in the microbubble flotation project are included for comparison. A more through discussion of the combustion characteristics of clean coals is provided below.

9.2.3 Results and Discussion of the Empirical Indices

From examination of the empirical slagging and fouling indices it seems that similar slagging behavior exists for the clean coal as well as the ROM coals for each seam. However, the critical viscosity temperature for the Illinois No. 6 agglomerates as predicted by the nomograph is low compared to the medium severity predicted for the ROM coal. Such lower critical viscosity temperature should cause a reduction of deposit thickness on the waterwalls of the boiler for the clean coal. The lower critical

Table 9-7
MINERAL ASH AND ASH FUSION ANALYSIS - ROM COAL AND CLEAN COAL AGGLOMERATES

	Illinois Nº 6 Coal			Upper Freeport Coal			Pittsburgh Coal		
	ROM Parent	Agglomerates	MF Product	ROM Parent	Agglomerates	MF Product	ROM Parent	Agglomerates	MF Product
% Ash	15.1	3.6	4.2	9.6	5.5	5.5	23.3	3.8	3.3
% Sulfur	3.7	2.6	2.7	2.6	1.3	1.4	3.8	1.8	3.3
Si02	50.6	40.1	42.0	46.8	48.6	38.2	41.5	44.9	34.1
Al203	19.7	19.5	19.3	21.1	24.4	24.7	19.6	23.1	22.3
Ti02	2.0	2.9	2.2	0.9	1.0	1.8	0.9	1.0	1.8
Fe203	16.4	25.3	21.2	20.1	14.1	17.0	18.9	18.2	27.7
Ca0	4.1	3.6	3.7	3.1	4.7	3.1	7.1	4.9	4.6
Mg0	0.9	1.4	1.4	1.0	0.8	1.2	1.3	0.8	1.3
K20	2.1	2.7	2.3	2.7	2.1	2.5	3.5	1.9	1.6
Na20	0.8	1.8	2.3	0.3	0.6	7.0	0.7	0.6	2.2
S03	2.7	1.1	3.4	2.4	2.1	2.8	5.0	2.9	3.2
P205	0.2	0.3	0.1	0.4	0.2	0.2	0.6	0.3	0.2
Sr0	0.0	0.1	ND	0.0	0.1	ND	0.1	0.1	ND
Ba0	0.0	0.0	ND	0.1	0.0	ND	0.1	0.0	ND
Ash Fusion Analysis (°F)									
Reducing									
Initial Deform	2086	2053	2020	2090	ND	1900	2020	ND	1900
Softening	2287	2180	2180	2281	ND	2000	2169	ND	1980
Hemispherical	2388	2260	2230	2369	ND	2080	2243	ND	2020
Fluid	2510	2340	2280	2453	ND	2120	2360	ND	2119
Oxidizing									
Initial Deform	2315	2269	ND	2310	ND	2200	ND	ND	ND
Softening	2473	2380	ND	2425	ND	2328	ND	ND	ND
Hemispherical	2525	2440	ND	2459	ND	2400	ND	ND	ND
Fluid	2640	2515	ND	2570	ND	2459	ND	ND	ND

MF = Microbubble Floatation Product
ND = Not Determined

viscosity corresponds to the ash fusion analysis data appearing in Table 9-6. The other two slagging indicators, ash slagging index and silica percentage, are similar for the ROM and clean coals tested.

Based on the indicators, fouling deposits should be more difficult to remove when burning clean coal rather than ROM coal due to the higher levels of sodium in the clean coal product. For example, the fouling index and the percent sodium oxide indicators are high for the Illinois clean coal. An explanation can be found in that caustic soda (NaOH) was used to adjust the pH of the Illinois No.6 slurry feed. Since the process water system was closed, this sodium remained in the system for a long time. Additionally, makeup water from the CQ Inc. ponds has considerable levels of sodium from pH adjustments.

High sodium in process water, combined with grinding of the coal to increase the available surface area, exposes more ionic sites for sodium bondage. Lower rank coals, such as Illinois No. 6, have higher levels of reactive sites which can also be seen from the oxygen levels, which parallel the levels of sodium oxide in the ash for all three coals. The highest levels of sodium are found in the Illinois No. 6 clean coal agglomerates, which also experienced the greatest increase in oxidation.

The percent alkalis index for Illinois No. 6 was lower with the agglomerated coal at 0.13 (low) than for the ROM coal at 0.32 (medium). This trend corresponds to the lower ash levels in the clean coal compared to the ROM parent coal. It indicates that the clean coal product will require less sootblowing than the ROM coal.

9.2.4 Prediction of Combustion Results

From Table 9-7, it is evident that the Illinois No. 6 coal produced in the spherical agglomeration and microbubble advanced physical fine-coal cleaning projects have similar ash, sulfur, and mineral ash compositions. Extensive combustion tests have been conducted for the Illinois No. 6, Upper Freeport, and Pittsburgh seam coals performance results of coal produced in microbubble flotation process were reported by O. Chow et al.[6]

In general, the selective removal of the basic components in the ash resulted in the lowering of ash fusion temperatures. Combustion characteristics with near 100 percent carbon conversion efficiencies were seen with the microbubble flotation product (MFP). Similar conversion efficiencies are expected for the agglomerate product due to the particle size of the coal and its low levels of ash.

Higher waterwall heat transfer coefficients were noted with the MFP coal but removability of deposits remained the same. Deposits generated with the microbubble clean coal were much thinner than those from the parent coals, with a more rapid attainment of steady state heat transfer and a higher heat flux. The critical furnace temperature where deposits still remained removable remained the same for both coals. From examining the indices in Table 9-8, it can be expected that agglomerates will behave similarly, if not better. The ash slagging and critical viscosity indices are the same for both products with the critical viscosity being slightly lower for the agglomerates.

Convection tube deposits are expected to be similar for both clean coals. Agglomerates and the microbubble flotation product had considerably higher levels of sodium than was found in the parent coal. Both had very low levels of ash when compared with the parent ROM coals. Given these similarities, it is expected that the agglomerates will result in fouling deposits and high tube-to-deposit bonding strengths. As with the MFP, burning agglomerates should result in reduced soot blower requirements of the convection tubes compared to the ROM coals. Despite higher strengths, fouling deposits from the MFP (and deposits expected for the agglomerates) were found to still be within the range considered removable. Lower

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[6] O. K. Chow, "Performance Characteristics of Beneficiated Coal-Based Fuels," 15th International Conference on Coal and Slurry Technologies, April 23-26, 1990, pp 3, 8, 10, 14, 16

Table 9-8
MINERAL ASH ANALYSIS - EMPIRICAL INDICES FOR AGGLOMERATION FEED, AGGLOMERATES, AND MICROBUBBLE FLOTATION PRODUCT WITH RANGES OF ANTICIPATED SEVERITY

	Illinois Nº 6 Coal			Upper Freeport Coal			Pittsburgh Coal		
	ROM Parent	Agglomerates	MF Product	ROM Parent	Agglomerates	MF Product	ROM Parent	Agglomerates	MF Product
Base/Acid Ratio	0.33	0.56	0.49	0.39	0.30	0.48	0.51	0.38	0.64
Fe2O3/(CaO+MgO) Coal Type	3.26 Eastern	5.12 Eastern	4.16 Eastern	4.97 Eastern	2.57 Eastern	3.95 Eastern	2.27 Eastern	3.20 Eastern	4.69 Eastern
Slagging Indices									
Ash Slagging Index (Rs)	1.22 Medium	1.42 Medium	1.31 Medium	1.04 Medium	0.40 Low	0.67 Medium	1.91 Medium	0.69 Medium	2.12 High
Silica Percentage (%)	70.3 Medium	57.0 Medium	61.5 Medium	66.0 Medium	71.3 Medium	64.3 Medium	60.4 Medium	65.3 Medium	50.4 Medium
Critical Viscosity T250	2500 Medium	2280 Low	2320 Medium	2420 Medium	2425 Medium	2325 Medium	2300 Medium	2535 Medium	2240 Low
Fouling Indices									
Fouling Index (Rf)	0.25 Medium	1.00 High	1.12 Severe	0.12 Low	0.12 Low	3.33 Severe	0.33 Medium	0.23 Medium	1.41 Severe
Percent Na2O in Ash	0.75 Medium	1.79 High	2.30 High	0.30 Low	0.63 Medium	7.00 Severe	0.66 Medium	0.59 Medium	2.20 High
Percent Alkalies	0.32 Medium	0.13 Low	0.16 Low	0.20 Low	0.11 Low	0.48 High	0.69 High	0.07 Low	0.11 Low

sodium concentrations in the agglomeration products compared to MFP should result in an easier removable deposit. This also corresponds to the lower empirical fouling indicators for the agglomerates.

9.3 ENVIRONMENTAL EFFECTS OF WASTE STREAMS

Typical waste steams from the POC plant consisted of primary and secondary tails (86 percent mineral matter) filter press cake, boiler blowdown water, and flare stack combustion gasses. The primary tails stream represented the bulk of the waste generated from the process. Waste from the boiler blowdown consisted of hard water dissolved solids and was considered harmless. Flare exhaust gasses contained CO_2 and H_2O and excess nitrogen from the blanket system.

A complete waste stream analysis of the POC facility is currently being conducted by Radian Corporation for EPRI. Laboratory analysis is complete at the time of this writing (July 1990) and a report of the findings is pending.

9.3.1 Trace Element Analysis of Feed, Agglomerates, and Primary Tails

A complete set of trace element data is available for the Illinois No. 6 coal feed, agglomerates, and primary tails. For the Upper Freeport and Pittsburgh seam coals, only feed coal to the grinding circuit coal was analyzed for trace elements.

Examination of the Illinois No. 6 trace element analysis revealed that metals of environmental concern were not being concentrated in either the clean coal product or waste streams. The primary tails stream is nonhazardous since it consistently contained 85 percent or more environmentally harmless ash. Coal tailings are considered environmentally benign since they contain no significant levels of leachable heavy metals.

Suitable disposal methods for solid tailings were investigated by Radian Corporation under EPRI contract. Preliminary leaching analysis by Radian using the Toxic Characteristic Leaching Procedure showed leachate levels of silver, arsenic, barium, cadmium, chromium, mercury, lead, or selenium below the limits set in the latest Code of Federal Regulations (40 CFR Part 261, March 29, 1990). These results indicate that the primary and secondary tails can be disposed of in a "nonhazardous" landfill mixed with other solids to stabilize the material and avoid erosion or dustiness.

9.3.2 Heptane Concentration in the Refuse

Heptane concentrations in the refuse streams reported by the UPARC method are all below 300 ppm to solids (dry basis). No bench-scale refuse samples were analyzed for heptane.

Section 10

PERFORMANCE OF THE SELECTIVE GRINDING CIRCUIT

10.1 SELECTIVE GRINDING IN THE POC PLANT

The selective grinding system prepared the slurry feed for agglomeration and operated with a feed rate of 2,000 lb per hour of coal. Soon after startup it became clear that mineral matter was concentrating in the recirculation loop. A modification to the system was implemented with the installation of a spiral separator, which removed pyrite and other mineral matter.

10.2 BACKGROUND

The opportunity to remove ash-forming minerals from coal is best when the minerals and coal are liberated from each other. The degree of particle size reduction required to liberate the coal and mineral matter depends on the coal characteristics. The bituminous coals selected for the test program contained finely dispersed minerals and required extensive grinding to produce high yields of clean coal with low ash contents. This became apparent during the Microbubble Flotation project which Bechtel performed for DOE and EPRI (DOE Contract No. DE-AC22-85PC81205). Petrographic research conducted during that project indicated a significant amount of still unliberated mineral particles below 44 microns, which, since recovered with the clean coal, contributed to the ash and sulfur content of the product. The grinding system used at that time consisted of an open circuit ball mill receiving 1/4-inch x 0 coal followed by a bead mill. This system produced a quantity of larger particles which contained a relatively high amount of ash and pyrite to affect the clean coal quality.

It was concluded that an open circuit grinding system was not able to liberate these particles. It appeared that the softer coal was preferentially ground to very fine sizes while the harder mineral matter particles remained locked or semi-locked in coal at a coarser size.

These observations led to the testing and design of a closed grinding system using a solid bowl centrifuge as a classifier. The tests became part of the bench-scale test program. A description of the tests is presented in Section 4, Bench-Scale Tests.

10.3 SELECTIVE GRINDING SYSTEM

Figure 10-1 shows the selective grinding system as initially installed for the program. The 1/4-inch x 0 coal and water are fed to the ball mill.

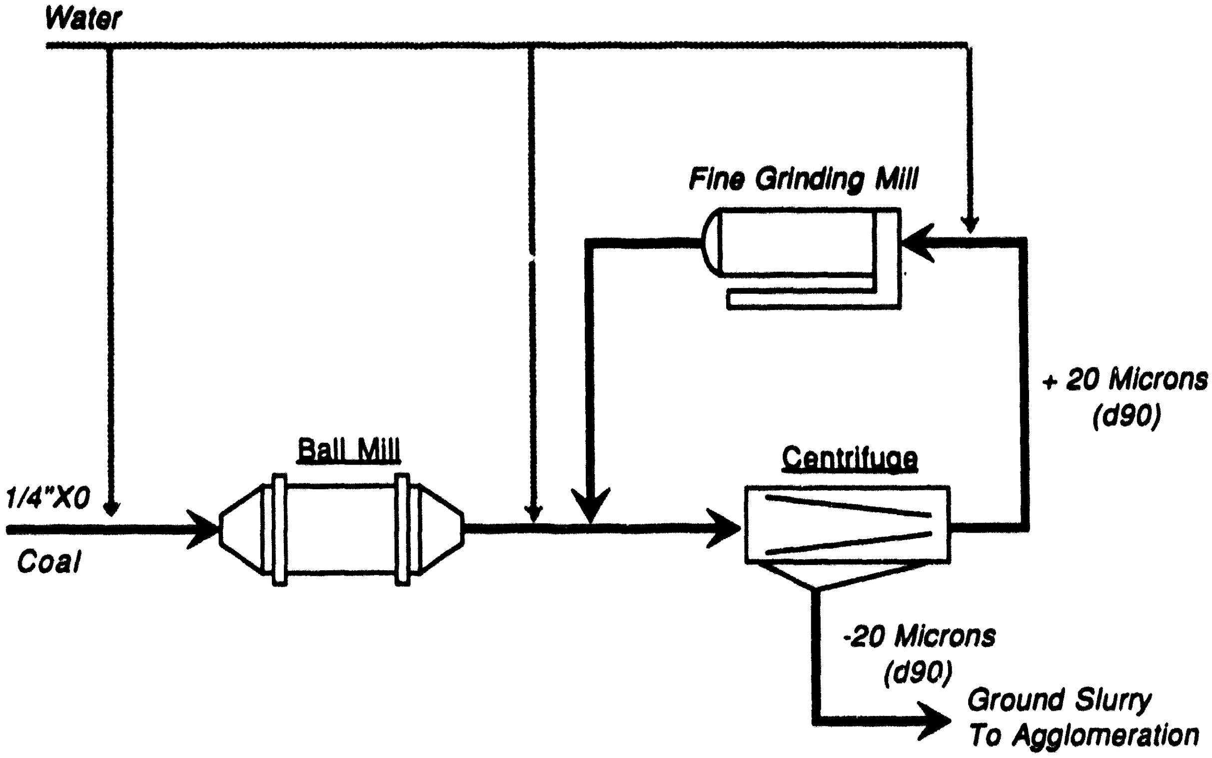

Figure 10-1 Selective Grinding Circuit Flowsheet

A typical ball mill product size distribution using the Illinois No. 6 coal is shown in Figure 10-2.

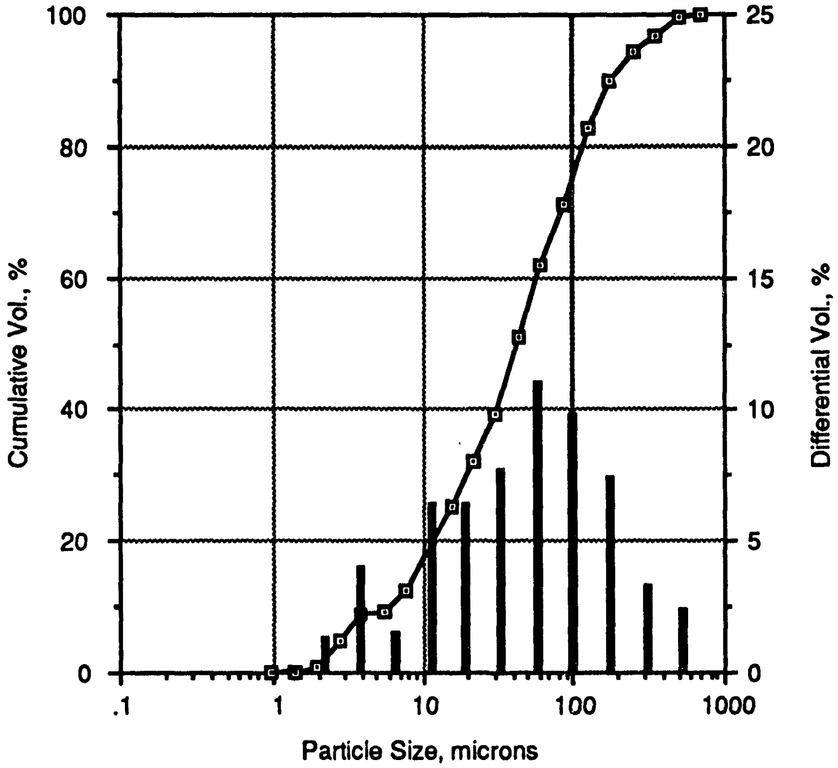

Figure 10-2 Ball Mill Product

Approximately 90 percent of the particles pass 176 microns (80 mesh) and 50 percent pass 44 microns (325 mesh). The ball mill product is collected in a sump which also receives the fine grinding mill product. Figure 10-3 shows the size distribution of the fine grinding mill product.

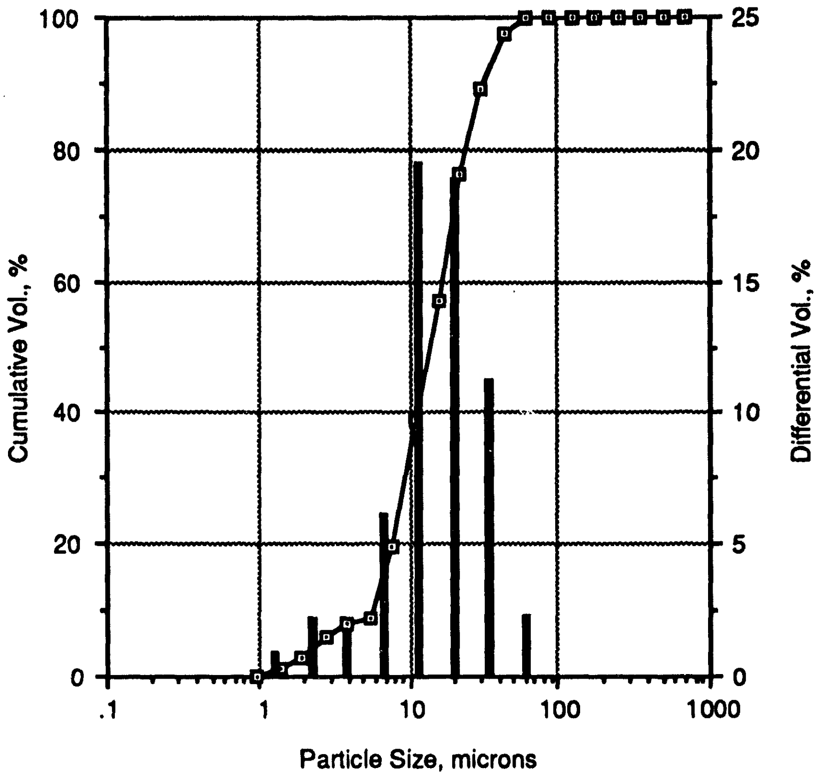

Figure 10-3 Fine Grinding Mill Product

Approximately 90 percent of the coal passes 25 microns and 50 percent passes 5 microns. Both products are mixed and diluted with water and pumped to a tank from where the slurry gravitates to a solid bowl centrifuge.

Figure 10-4 shows a typical size distribution of the centrifuge feed, which indicates that approximately 90 percent of the particles pass 30 microns and 50 percent pass 9 microns.

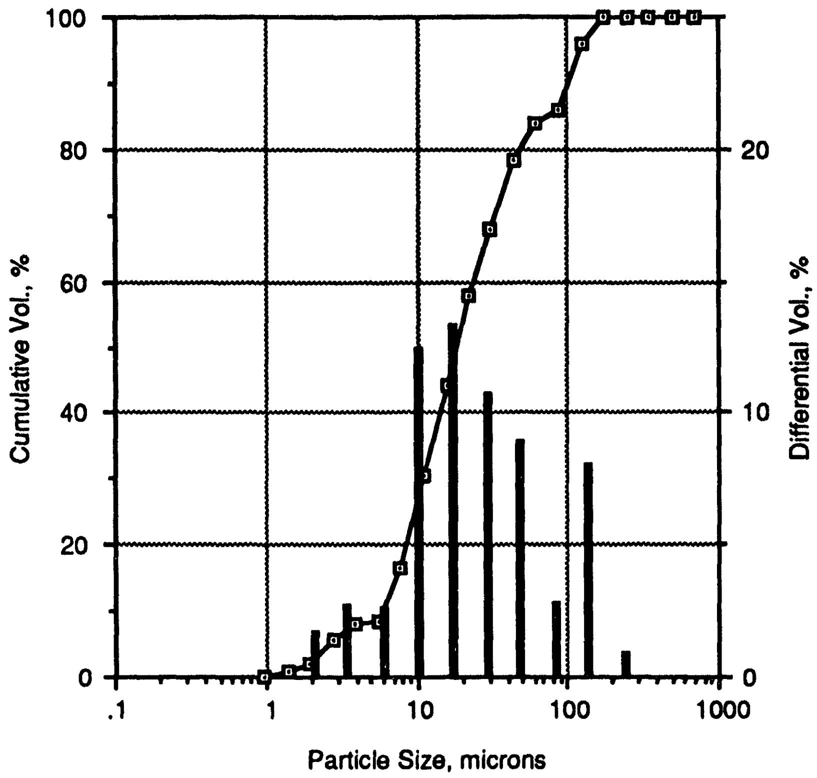

Figure 10-4 Centrifuge Feed

The centrifuge is operated as a classifier producing a cake of oversize material for further size reduction and a slurry effluent containing the particles of the required top size. The cake is diluted with water to the 35 percent solids and pumped to the fine grinding mill.

The centrifuge centrate is the final product of the grinding system which becomes the feed to the agglomeration process. Figure 10-5 shows the size distribution of this product.

Approximately 90 percent of the particles pass 14 microns and 50 percent pass 5.5 microns.

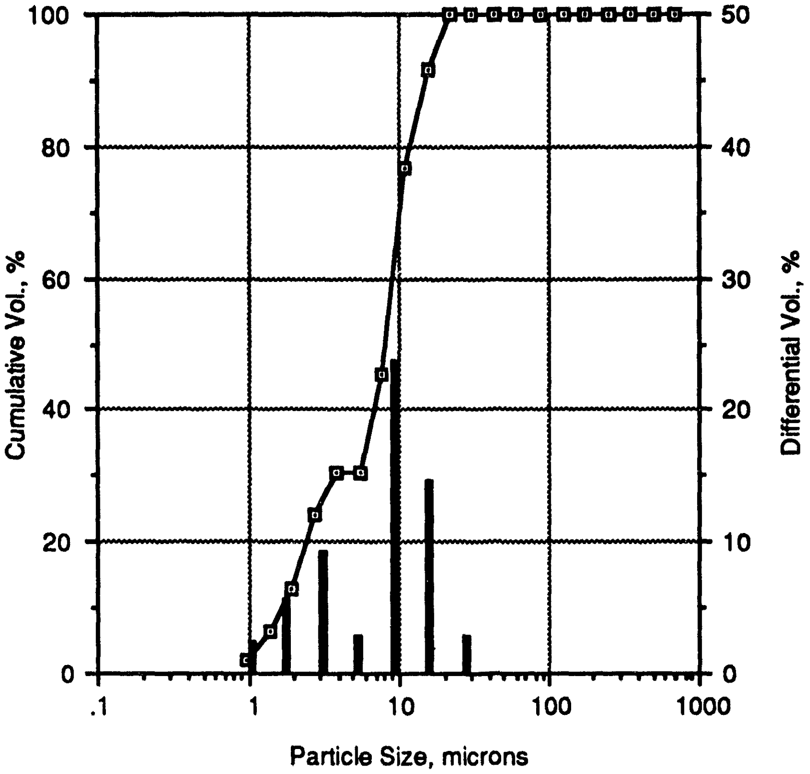

Figure 10-5 Final Product

Samples were regularly taken during the operation of the grinding circuit after the system had been balanced. The streams sampled include the ball mill product, centrifuge feed, fine grinding mill product and final product. The circuit was balanced when the final product, namely the centrifuge centrate, contained a solids content corresponding to the total coal and water being fed to the system. A further indication of a weight balanced system was the levelling out of the recirculation load. The ground product was discarded until these conditions were met. Typically the system was balanced within 1 hour from startup. Analysis of slurry around the grinding circuit are shown on the following page.

	Ash wt. %	Sulfur wt. %
Ball Mill Feed	15.06	4.05
Ball Mill Product	14.78	4.07
Centrifuge Feed	19.41	5.78
Fine Mill Product	21.43	6.55
Final Product	14.47	3.61

These results indicate that the final product was lower in ash and sulfur content than the ball mill feed even after several hours of operation under balanced conditions. Ash and sulfur minerals were apparently concentrated within the loop between the centrifuge and the fine grinding mill. This conclusion was confirmed by samples taken from the fine grinding mill product and final product streams during a shutdown period of the grinding system. This period started after the ball mill operation was stopped and the centrifuge-fine grinding mill loop continued to operate for an hour; during this hour the material in the recirculation loop was ground and reground by the fine grinding mill except for material that was allowed to leave the system as final product by the centrifuge. Samples taken at the end of this operation showed the following:

	Ash wt. %	Sulfur wt. %
Fine Mill Product	36.73	11.36
Final Product	17.69	4.57

The above data show a shift of ash and pyrite into the recirculating material stream. The final product during this period probably consisted of coal which could be loosened from the mineral-rich particles.

The data not only indicate that high ash particles resisted grinding but also that these particles were forced by the centrifuge into the cake for repeated grinding. This suggests that the centrifuge not only separates by particle size but also concentrates the higher density mineral-rich particles in the cake.

The petrographic analysis of the ground product from the three coals show that relative to coal, the pyrite particles were extremely small. While the composite coal was about 20 microns (90 percent passing) most of the pure pyrite was below 7 microns. This confirms that the centrifuge allowed the high density mineral-rich particles to leave the grinding circuit only when ground to a size below 7 microns whereas low ash and low density coal was classified at around 20 microns.

Most of the spherical agglomeration tests were performed using this selectively ground coal. This material showed lower ash and sulfur contents than the raw coal feed, as noted above. To compensate for this effect, a few agglomeration tests were performed using a feed prepared as follows: after operating the grinding system for several hours and filling the agglomeration feed sump approximately 3/4 full with ground coal, the feed to the ball mill and the ball mill itself were shut down. The remaining equipment of the grinding system, namely the centrifuge, fine grinding mill and pumps, continued operation. This allowed for the material in the centrifuge-fine grinding mill loop to be ground as many times as necessary to reach 20 microns. This material was then added to the normally ground feed in the spherical agglomeration feed sump. The resulting mixture was agglomerated. This test and a test with a "normally" selective ground feed are compared in the following table:

	Selective Grind (Normal)	**Selective Grind with Recirculation Loop Material Added**
Feed		
Ash, wt. %	13.19	16.48
Total Sulfur, wt. %	3.29	3.34
Pyritic Sulfur, wt. %	1.05	1.32
Clean Coal		
Ash, wt. %	4.30	5.52
Total Sulfur, wt. %	2.80	2.93
Pyritic Sulfur, wt. %	0.44	0.69
Refuse		
Ash, wt. %	71.04	87.90
Total Sulfur, wt. %	6.14	6.96
Yield, %	86.8	86.8
Energy Recovery, %	96.6	99.4
Ash Reduction, %	70.9	70.9
Pyrite Reduction, %	62.6	54.6

These results show interesting conclusions, namely:

- The higher ash content of the refuse for the test using more recirculation loop material seems to be the result of the addition of the finely ground, and therefore liberated, minerals.
- The higher ash content of the clean coal for the test using more recirculation loop material seems to be the result of an increase of particles which still contain coal and unliberated fine minerals.
- The concentration of high ash and high sulfur material in the grinding loop and its removal from the agglomeration feed results in a cleaner coal with lower ash and sulfur content.

10.4 MODIFIED SELECTIVE GRINDING SYSTEM

Figure 10-6 shows the flowsheet of the modified selective grinding system, which included a Humphrey-type spiral separator to process a slip stream of the grinding system loop material.

The spiral separator has proven to be an excellent equipment for removing pyrite and other heavy minerals from coal. The loop material, even though fine, was still an ideal feed for a spiral since it represented a narrow particle size range from which the ultra-fine material had been removed.

An alternate operation of the spiral separator was to feed all of the diluted loop material to the spiral separator, as shown in Figure 10-7.

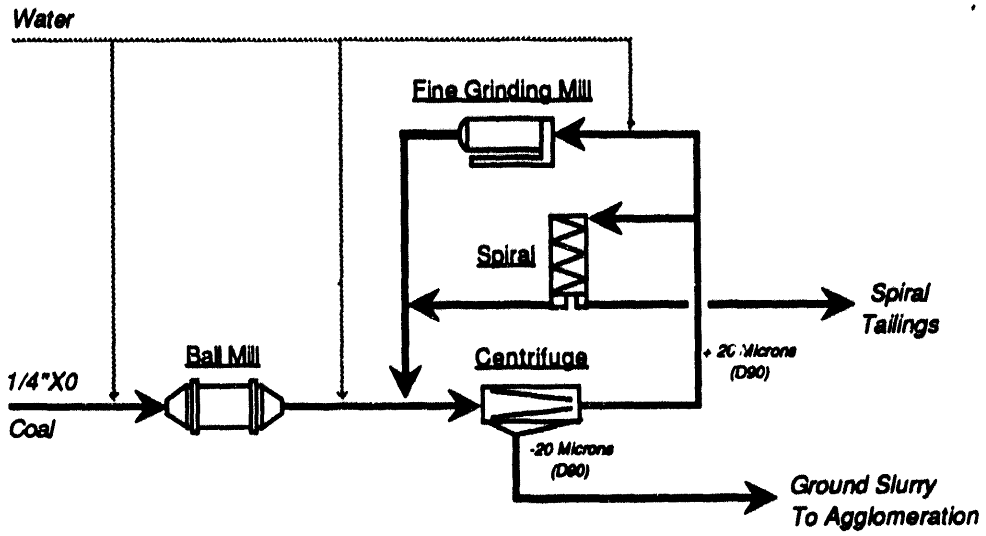

Figure 10-6 Modified Selective Grinding Circuit With Recirculation Loop Slipstream Feed to Spiral Separator

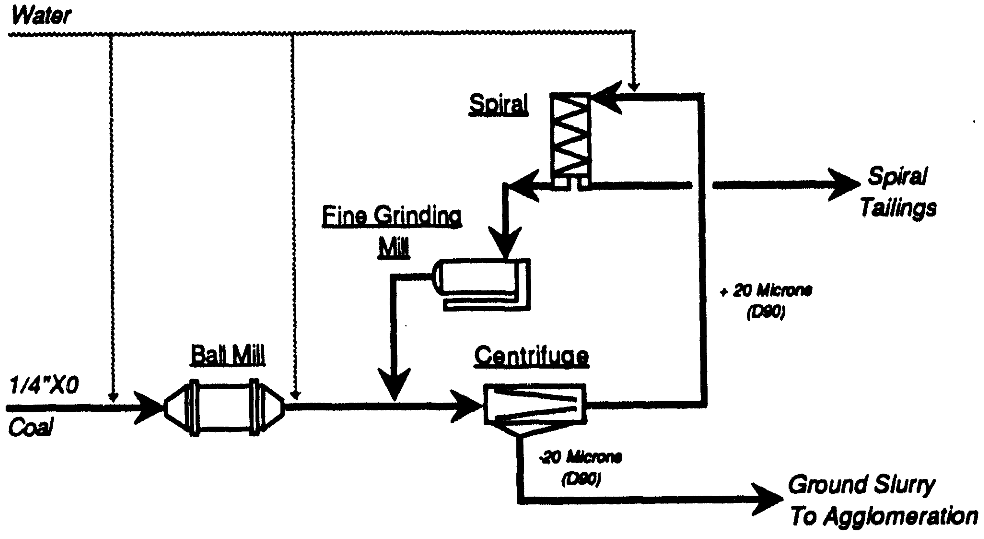

Figure 10-7 Modified Selective Grinding Circuit with Full Recirculation Loop Feed to Spiral Separator

This was not the optimum operation since the spiral separator requires a diluted feed which then resulted in a low solids concentration fine grinding mill feed. The fine grinding mill, however, provided the best size reduction with a thicker feed. Still, this configuration was used most of the time since an accurate splitting of the slurry stream and balancing of the water additions was not practical.

The modified selective grinding circuit provided a slurry feed to agglomeration that showed a marked improvement in the overall process results. Results from two spiral performance test runs are presented below:

	Selective Grind with Slip-Stream to Spiral (Illinois No. 6)	**Selective Grind with Full-Stream to Spiral (Illinois No. 6)**
Spiral Feed		
Ash, wt. %	13.00	16.16
Total Sulfur, wt. %	4.05	3.84
Pyritic Sulfur, wt. %	1.71	1.61
Spiral Clean Coal		
Ash, wt. %	12.81	14.35
Total Sulfur, wt. %	3.29	3.01
Pyritic Sulfur, wt. %	1.04	0.73
Spiral Refuse		
Ash, wt. %	51.53	44.14
Total Sulfur, wt. %	25.24	14.52
Yield, %	99.5	93.9
Energy Recovery, %	99.8	96.2
Ash Reduction, %	1.7	13.3
Pyrite Reduction, %	39.3	55.7

The operation of the spiral provided a much improved overall process performance with agglomeration for cleaning the Illinois No. 6 and Upper Freeport coals. Less improvement was noticed with the Pittsburgh coal since this coal had been precleaned at the mine. Agglomeration results before and after the introduction of the spiral are presented below:

	Selective Grind without Spiral (Mean Values)	**Selective Grind with Spiral (Mean Values)**
Illinois No. 6 Coal		
Ash, wt. %	3.93	3.77
Total Sulfur, wt. %	2.78	2.70
Energy Recovery, %	98.6	93.2
Sulfur Reduction, %	25.0	45.0
Pyrite Reduction, %	65.0	84.8
Upper Freeport Coal		
Ash, wt. %	7.55	5.90
Total Sulfur, wt. %	1.85	1.32
Energy Recovery, %	99.4	88.9
Sulfur Reduction, %	46.2	72.1
Pyrite Reduction, %	55.8	83.6

The above data show a marked increase in sulfur reduction using the spiral, almost double that of the sulfur reduction without the spiral. These results were achieved without an estimated adjustment of the grinding system and it is suggested that the energy recovery could have been improved under better operating conditions of the spiral separator. Further work in improving the efficient liberation of coal from mineral matter is needed.

Section 11

CONCEPTUAL DESIGN OF A COMMERCIAL PLANT

11.1 GENERAL

A conceptual design for a commercial plant using the spherical agglomeration technology is presented in this section. All tables and figures are included at the end of this section.

The design includes appropriate modifications to the design criteria used for the POC test unit to suit the commercial plant's scale of operation and economic objectives. For example, the POC test unit was designed to treat a variety of coals with a wide range of characteristics. The conceptual design is tailored for a specific coal taking into consideration its unique properties.

The conceptual design is based on coal from the Upper and Lower Freeport seams. These seams represent vast resources of high quality coking and steam coal in the Northern Appalachian region. They are extensively mined in Pennsylvania, Ohio, and West Virginia. There are an estimated 2-1/4 billion tons of recoverable Upper Freeport Seam coal and 1 billion tons of Lower Freeport Seam coal in Pennsylvania alone[1]. At several locations the two seams are mined together.

Coal from the Hepburnia Coal Company's Clearfield County mine in Pennsylvania has been selected as the design coal. Coal from the Hepburnia mine consists of approximately a 50:50 blend of the Upper and Lower Freeport seam coals.

Based on analytical data for the design coal, the following design criteria have been developed, which differ from that used for POC test module.

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[1] "The Reserve Base of Bituminous Coal and Anthracite for Underground Mining in the Eastern United States," USBM, IC-8655, 1974

11.1.1 Clean Coal Ash Content

Since very attractive energy recoveries were obtained during the POC tests for clean coal ash contents in the 4 to 5 percent range, the commercial plant has been designed for such clean coal ash contents.

11.1.2 Use of Heavy Medium Vessels

For POC testing ROM coal was ground to fine sizes in ball and bead mills and the ground product was processed by spherical agglomeration. An examination of the float/sink test data for the design coal indicates that 60 percent approximately of 4 percent ash clean coal, can be recovered by crushing to a top size of 1-1/4 inch and processing by conventional heavy medium (HM) vessels. It is also noted that a significant amount of rock and other impurities can also be removed efficiently using conventional HM cyclones. Thus grinding can be restricted to the difficult-to-clean middling fraction which comprises only 20 percent of the ROM feed coal.

The plant is designed to produce a clean coal made up of coarse coal (1-1/4 inch x 14M) from the HM vessels and agglomerates. This product will have good handling characteristics.

11.1.3 Continuous Heptane Stripper

Given the small capacity and the need for simplicity, a batch mode heptane stripping operation was adopted for the POC test unit. Considering the large capacity requirements of a commercial plant and the desire to reduce energy consumption during heptane recovery, a specially designed continuous heptane stripper is used.

11.2 DESIGN BASIS

Considering the deviations from the POC test design the basis used for the development of the conceptual design of the grassroot commercial plant is given below.

11.2.1 ROM Coal Quality

Coal from the Hepburnia Coal Company's Clearfield County mine in Pennsylvania was selected. Table 11-1 presents the properties of the ROM coal and the analysis of the size fractions of ROM coal when crushed to a nominal top size of 1-1/4 inch. The ROM coal has a sulfur dioxide emission potential of 2.8 lb/MMBtu. A high proportion of the coal sulfur (68 percent) is due to pyrites, making this coal a good candidate for physical cleaning. The rest of the sulfur in the coal is largely in the organic form.

Float/sink washability data for crushed ROM coal,(Table 11-2), has been used to develop a cleaning scheme for this coal and a material balance. Data is from the EPRI report "Coal Cleaning Test Facility Campaign Report Number 1: Freeport Seam Coal" (Interim Report, EPRI CS-3608, January 1985).

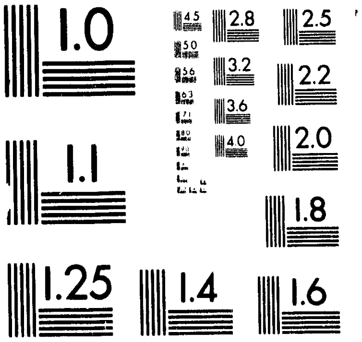
11.2.2 Hourly Rated Capacity

The plant has been sized to meet the requirements of a 700 MW power plant. The calculations as presented in Table 11-3 lead to an hourly capacity of 350 tons (dry basis).

11.2.3 Brief Description of the Cleaning Scheme

Figure 11-1 shows a simplified block diagram describing the cleaning scheme. The major elements are:

- o ROM coal is crushed to a nominal 1-1/4 inch top size.
- o The coal is wet screened. The 1-1/4 inch x 1/4 inch (coarse coal) and 1/4 inch x 14M (fine coal) fractions are cleaned in HM vessels (separators) at a specific gravity of approximately 1.3 to produce a clean coal with 3.8 to 5 percent ash and 0.8 percent total sulfur.
- o The sink material from the coarse coal HM vessel is crushed to a 1/4 inch top size and combined with the sinks from the fine coal vessels. The combined streams are then recleaned in HM cyclones after desliming. The HM cyclones are operated at a specific gravity of 1.8 to produce a high ash refuse. The


1.0
4.5
5.0
5.6
6.3
7.1
8.0
9.0
2.8
3.2
3.6
4.0
2.5
2.2
2.0
1.8
1.1
1.25
1.4
1.6

3 of 3

Table 11-1
FREEPORT COAL
ROM COAL CHARACTERISTICS

Analysis	**Quantity**
Ash, %	12.1
Total Sulfur, %	1.9
Pyritic Sulfur, %	1.25
Heating Value, Btu/Lb	13,639
Pyritic Sulfur, % of Total Sulfur	68
Total Sulfur, Lb SO_2/MMBtu	2.8
Total Moisture, "As Received," %	5.6

Size Analysis (Crushed to Nominal 1-1/4 Inch x 0)

Size	**Wt %**	**Ash %**	**Sulfur %**	**Heating Value Btu/Lb**
Plus 1/4 Inch	41.3	14.97	1.77	13,183
1/4 Inch x 14 M	33.7	10.31	2.00	13,928
14 M x 28 M	11.3	9.11	1.99	14,142
28 M x 60 M	6.7	8.49	1.89	14,202
60 M x 100 M	2.1	8.73	1.84	14,145
100 M x 200 M	1.9	9.70	2.24	14,005
Minus 200 M	3.0	15.06	2.03	12,934
Total	100.0	12.08	1.90	13,638

All analysis values reported are on a dry basis except "as received" moisture content

Table 11-2

FREEPORT COAL
ROM COAL CHARACTERISTICS

A. FLOAT/SINK OF PLUS 3/4 INCH FRACTION REPRESENTING 12.2% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	14.6	5.01	0.83	14,916	0.56	14.6	5.01	0.83	14,916	0.56
1.30	1.35	21.5	8.72	1.32	14,262	0.93	36.1	7.22	1.12	14,527	0.77
1.35	1.40	20.2	15.38	1.13	13,163	0.86	56.3	10.14	1.13	14,038	0.80
1.40	1.45	15.8	20.00	1.27	12,380	1.03	72.1	12.31	1.16	13,674	0.85
1.45	1.50	9.1	24.98	1.47	11,564	1.27	81.3	13.74	1.20	13,436	0.89
1.50	1.55	5.4	29.14	1.66	10,842	1.53	86.7	14.70	1.22	13,274	0.92
1.55	1.60	2.2	33.17	1.93	10,092	1.92	88.9	15.15	1.24	13,196	0.94
1.60	1.70	2.1	38.13	2.53	9,198	2.75	91.0	15.69	1.27	13,102	0.97
1.70	1.80	1.4	45.10	3.38	7,943	4.26	92.4	16.14	1.30	13,024	1.00
1.80		7.6	70.71	5.47	3,567	15.32	100.0	20.28	1.62	12,307	1.32

B. FLOAT/SINK OF 3/4 X 1/2 INCH FRACTION REPRESENTING 19.25% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	31.8	5.16	0.84	14,927	0.56	31.8	5.16	0.84	14,927	0.56
1.30	1.35	28.1	9.13	1.13	14,181	0.80	59.9	7.02	0.98	14,577	0.67
1.35	1.40	14.9	14.64	1.07	13,242	0.81	74.8	8.54	1.00	14,311	0.70
1.40	1.45	7.8	19.85	1.44	12,330	1.17	82.5	9.60	1.04	14,125	0.73
1.45	1.50	4.8	23.92	1.97	11,596	1.70	87.3	10.39	1.09	13,986	0.78
1.50	1.55	2.7	28.00	2.36	10,922	2.17	90.1	10.92	1.13	13,893	0.81
1.55	1.60	2.0	31.79	3.04	10,299	2.95	92.1	11.39	1.17	13,813	0.85
1.60	1.70	1.8	36.28	3.96	9,355	4.23	93.9	11.88	1.23	13,725	0.89
1.70	1.80	1.3	43.48	4.46	8,081	5.52	95.2	12.31	1.27	13,649	0.93
1.80		4.8	65.93	8.35	3,685	22.66	100.0	14.86	1.61	13,175	1.22

Table 11-2 (Cont'd)

C. FLOAT/SINK OF 1/2 INCH X 1/4 INCH FRACTION REPRESENTING 19.8% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	46.7	4.93	0.72	14,868	0.48	46.7	4.93	0.72	14,868	0.48
1.30	1.35	22.1	8.77	0.95	14,233	0.67	68.8	6.16	0.79	14,665	0.54
1.35	1.40	14.6	13.93	1.04	13,354	0.78	83.4	7.52	0.84	14,435	0.58
1.40	1.45	4.4	18.96	1.52	12,450	1.22	87.8	8.10	0.87	14,335	0.61
1.45	1.50	2.6	23.57	2.06	11,684	1.76	90.5	8.55	0.91	14,258	0.64
1.50	1.55	1.5	27.44	2.46	10,949	2.25	92.0	8.86	0.93	14,203	0.66
1.55	1.60	1.2	31.61	3.14	10.326	3.04	93.2	9.15	0.96	14,154	0.68
1.60	1.70	1.3	36.73	3.66	9,036	4.04	94.4	9.52	1.00	14,085	0.71
1.70	1.80	0.9	43.13	5.40	8,160	6.62	95.3	9.84	1.04	14,029	0.74
1.80		4.7	64.18	14.18	4,338	32.68	100.0	12.37	1.65	13,577	1.22

D. FLOAT/SINK OF 1/4 INCH X 1/8 INCH FRACTION REPRESENTING 15.6% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	54.9	4.18	0.87	15,000	0.58	54.9	4.18	0.87	15,000	0.58
1.30	1.35	24.8	8.69	0.97	14,213	0.68	79.7	5.59	0.90	14,755	0.61
1.35	1.40	6.9	13.92	1.20	13,297	0.90	86.6	6.25	0.92	14,639	0.63
1.40	1.45	3.5	17.76	1.73	12,600	1.38	90.1	6.70	0.95	14,559	0.66
1.45	1.50	1.6	22.98	2.17	11,756	1.84	91.7	6.98	0.98	14,511	0.67
1.50	1.55	1.1	26.69	2.91	11,078	2.62	92.7	7.21	1.00	14,471	0.69
1.55	1.60	0.8	29.74	3.56	10,530	3.38	93.6	7.41	1.02	14,437	0.71
1.60	1.70	1.0	34.63	5.41	9,635	5.61	94.5	7.69	1.07	14,387	0.74
1.70	1.80	0.7	40.69	5.78	8,523	6.78	95.2	7.92	1.10	14,346	0.77
1.80		4.8	60.74	18.42	4,648	39.63	100.0	10.45	1.93	13,880	1.39

Table 11-2 (Cont'd)

E. FLOAT/SINK OF 1/8 INCH X 28M FRACTION REPRESENTING 29.4% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	68.5	3.46	0.79	15,146	0.52	68.5	3.46	0.79	15,146	0.52
1.30	1.35	14.3	8.74	1.02	14,176	0.72	82.8	4.37	0.83	14,978	0.56
1.35	1.40	5.8	13.33	1.26	13,385	0.94	88.6	4.96	0.86	14,875	0.58
1.40	1.45	2.5	17.18	1.50	12,599	1.19	91.0	5.29	0.88	14,813	0.59
1.45	1.50	1.3	21.02	1.86	11,946	1.56	92.3	5.51	0.89	14,774	0.60
1.50	1.55	0.9	23.86	2.33	11,453	2.04	93.2	5.67	0.90	14,743	0.61
1.55	1.60	0.7	27.83	2.96	10,707	2.76	93.8	5.84	0.92	14,714	0.63
1.60	1.70	0.7	33.09	4.47	9,825	4.55	94.6	6.04	0.95	14,677	0.65
1.70	1.80	0.6	37.89	5.69	8,867	6.42	95.1	6.23	0.97	14,643	0.67
1.80		4.9	61.69	20.00	4,507	44.38	100.0	8.94	1.90	14,147	1.35

F. FLOAT/SINK OF 28M X 60M FRACTION REPRESENTING 6.65% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	72.3	2.74	0.74	15,258	0.48	72.3	2.74	0.74	15,258	0.48
1.30	1.35	11.0	8.60	1.06	14,167	0.75	83.3	3.51	0.78	15,114	0.52
1.35	1.40	5.3	12.98	1.33	13,386	0.99	88.6	4.08	0.81	15,011	0.54
1.40	1.45	2.1	17.18	1.55	12,578	1.23	90.6	4.37	0.83	14,956	0.55
1.45	1.50	1.4	19.95	1.64	12,054	1.36	92.0	4.60	0.84	14,913	0.56
1.50	1.55	1.1	23.46	1.89	11,407	1.66	93.1	4.82	0.85	14,873	0.57
1.55	1.60	0.6	25.38	2.08	11,068	1.88	93.6	4.95	0.86	14,850	0.58
1.60	1.70	0.9	31.21	2.55	10,058	2.53	94.6	5.21	0.88	14,802	0.59
1.70	1.80	0.6	35.04	3.95	9,354	4.22	95.1	5.38	0.90	14,770	0.61
1.80		4.9	65.02	17.09	3,959	43.17	100.0	8.29	1.68	14,244	1.18

Table 11-2 (Cont'd)

G. FLOAT/SINK OF 60M X 100M FRACTION REPRESENTING 2.12% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	69.5	2.35	0.74	15,248	0.48	69.5	2.35	0.74	15,248	0.48
1.30	1.35	11.7	7.56	1.02	14,308	0.72	81.2	3.10	0.78	15,112	0.51
1.35	1.40	5.7	11.96	1.25	13,421	0.93	86.9	3.68	0.81	15,001	0.54
1.40	1.45	2.5	15.93	1.48	12,686	1.17	89.4	4.03	0.83	14,936	0.55
1.45	1.50	1.6	18.77	1.63	12,147	1.34	91.0	4.28	0.84	14,888	0.57
1.50	1.55	1.4	21.97	1.74	11,585	1.50	92.3	4.54	0.85	14,839	0.58
1.55	1.60	0.7	23.99	1.81	11,214	1.62	93.0	4.69	0.86	14,812	0.58
1.60	1.70	1.1	29.96	2.24	10,180	2.20	94.1	4.98	0.88	14,759	0.59
1.70	1.80	0.6	37.73	2.75	8,785	3.13	94.7	5.18	0.89	14,722	0.60
1.80		5.3	66.15	17.27	3,673	47.03	100.0	8.41	1.76	14,137	1.24

H. FLOAT/SINK OF 100M X 200M FRACTION REPRESENTING 1.93% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	68.6	2.33	0.74	15,241	0.48	68.6	2.33	0.74	15,241	0.48
1.30	1.35	10.2	6.78	0.94	14,369	0.66	78.9	2.90	0.77	15,128	0.51
1.35	1.40	6.3	11.75	1.23	13,440	0.91	85.2	3.56	0.80	15,002	0.53
1.40	1.45	1.9	14.51	1.39	12,821	1.08	87.1	3.80	0.81	14,955	0.54
1.45	1.50	2.1	16.91	1.44	12,385	1.16	89.2	4.10	0.83	14,895	0.56
1.50	1.55	1.4	18.41	1.45	12,169	1.19	90.6	4.33	0.84	14,852	0.56
1.55	1.60	1.1	20.48	1.55	11,768	1.32	91.7	4.53	0.85	14,814	0.57
1.60	1.70	1.4	26.17	1.96	10,779	1.82	93.1	4.85	0.86	14,753	0.58
1.70	1.80	0.8	37.01	2.79	8,899	3.14	93.9	5.11	0.88	14,706	0.60
1.80		6.1	67.15	18.69	1,276	146.45	100.0	8.92	1.97	13,883	1.42

Table 11-2 (Cont'd)

I. COMPOSITE WASABILITY OF PLUS 200M MATERIAL REPRESENTING 97% OF THE TOTAL

Gravity		Direct Float					Cumulative Float				
Float	Sink	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu	WT %	Ash	Sulfur	Btu/Lb	Lbs S/MMBtu
	1.30	51.9	3.88	0.79	15,065	0.52	51.9	3.88	0.79	15,065	0.52
1.30	1.35	19.4	8.74	1.05	14,213	0.74	71.3	5.21	0.86	14,833	0.58
1.35	1.40	10.4	14.20	1.14	13,294	0.85	81.7	6.35	0.90	14,637	0.61
1.40	1.45	5.2	18.99	1.43	12,452	1.15	86.9	7.11	0.93	14,506	0.64
1.45	1.50	3.0	23.53	1.77	11,691	1.52	89.8	7.65	0.96	14,414	0.66
1.50	1.55	1.8	27.19	2.12	11,037	1.92	91.7	8.03	0.98	14,347	0.68
1.55	1.60	1.1	30.56	2.76	10,425	2.65	92.8	8.31	1.00	14,299	0.70
1.60	1.70	1.2	35.37	3.70	9,460	3.91	94.0	8.65	1.03	14,238	0.73
1.70	1.80	0.8	41.64	4.78	8,372	5.71	94.8	8.94	1.07	14,187	0.75
1.80		5.2	64.48	14.72	4,122	35.72	100.0	11.82	1.78	13,664	1.30

Table 11-3

SPHERICAL AGGLOMERATION - COMMERCIAL PLANT HOURLY CAPACITY

Category	Unit
Power plant rating, MW	750
Heat rate, Btu/kWh	9,600
Load factor, %	70
Heating value of fuel (Dry), Btu/lb	15,020
Yearly requirement of clean coal, MM tons	1.58
Operating days of cleaning plant, days/year	250
Scheduled operating hours, h/day	24
Availability of the cleaning plant, %	87
Required output of cleaning plant, tph	302
Btu recovery of cleaning plant, %	96.8
Heating value of ROM coal, Btu/lb	13,640
Required input capacity of cleaning plant, tph	344
Designed cleaning plant capacity, tph	**350**

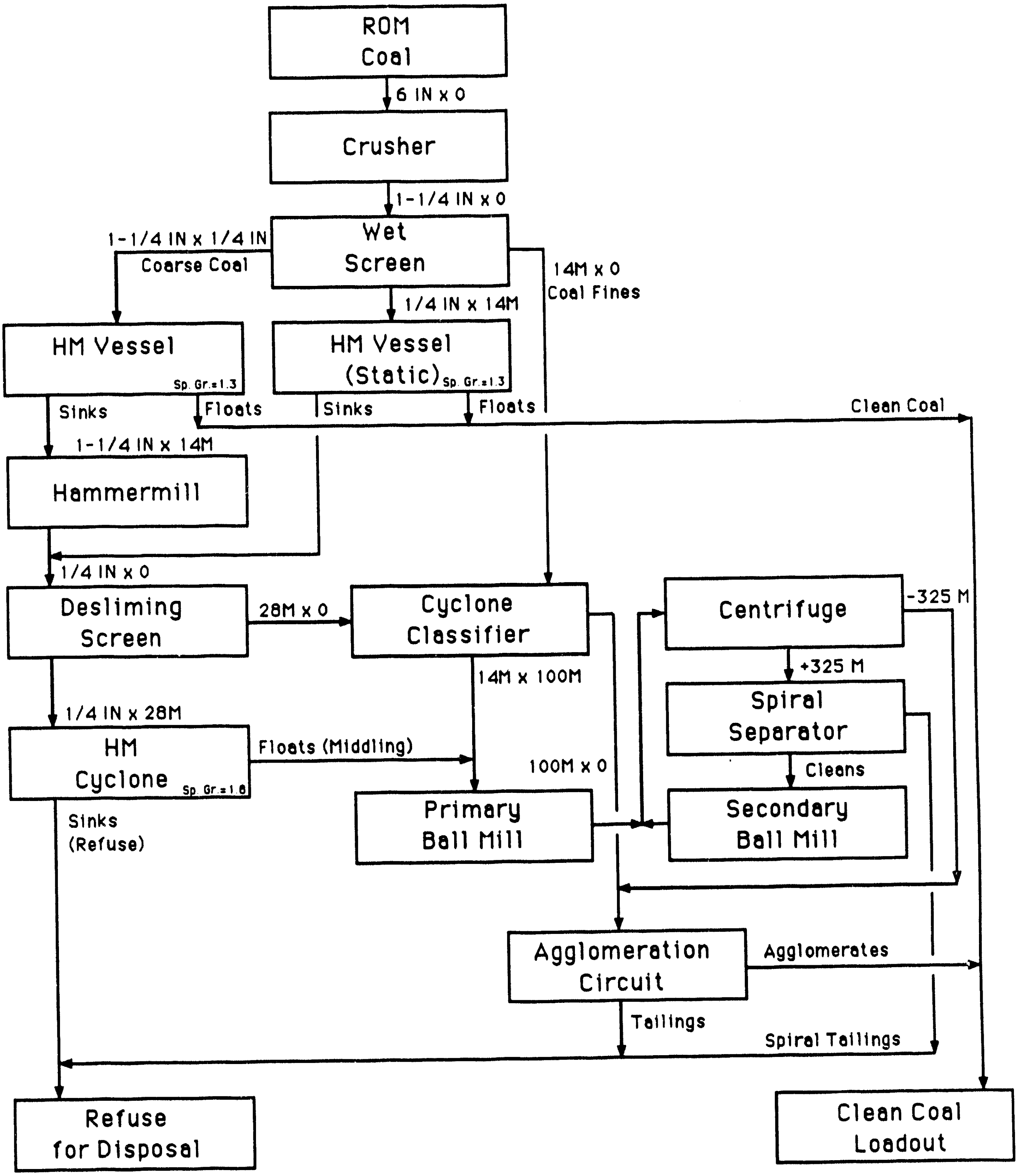


Figure 11-1 Commercial Cleaning Plant Block Flow Diagram

floats from the cyclones constitute a middling product.

- The middlings and the 14M x 0 size fraction (natural coal fines and fines produced from desliming screens) are sent to the selective grinding and agglomeration sections.
- The agglomerates, together with the clean coal from the HM vessels, are sent to the clean coal loadout.

A detailed description of the plant is provided in Section 11.3.

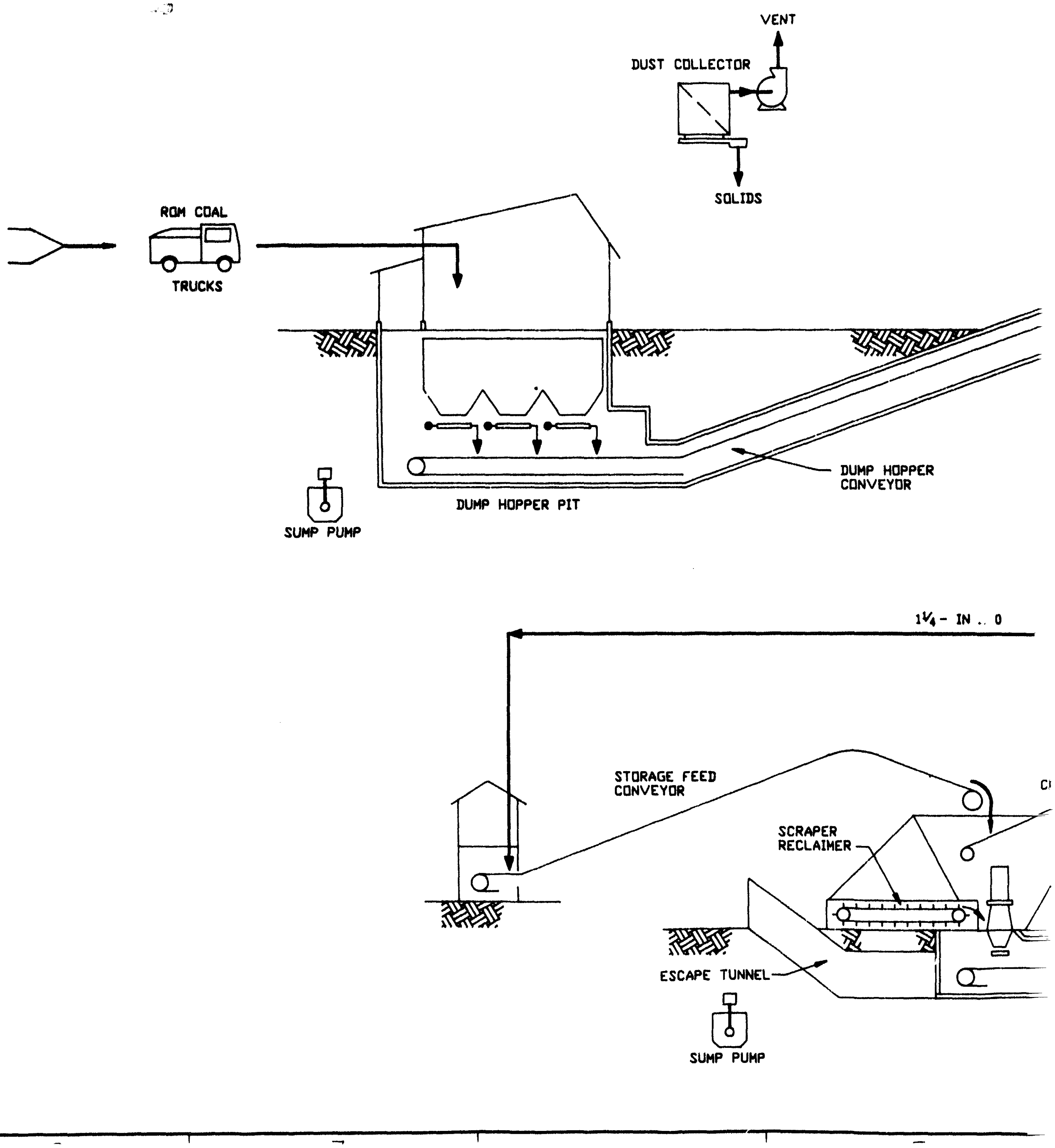
11.2.4 Plant Scope

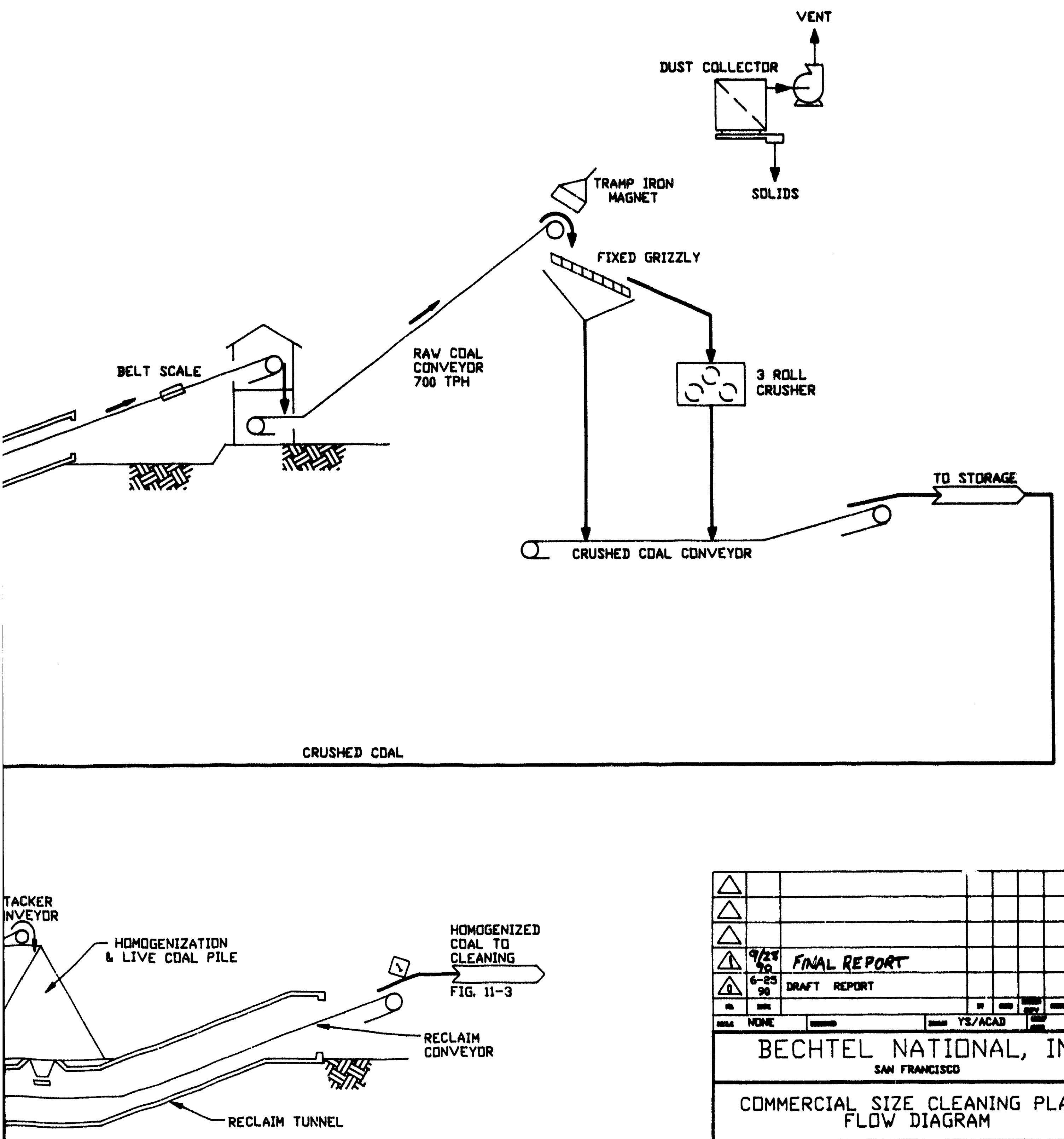
The grassroots mine-mouth plant includes facilities to receive coal, delivered in trucks, a crushing section to reduce the coal to a 1-1/4 inch top size, coal storage and homogenization facilities, a water clarification system, and equipment for loading out the clean coal. Coarse plant refuse is deposited in a landfill and fine tailings are pumped to a permitted collection pond.

11.3 PLANT DESCRIPTION

Flow diagrams for the conceptual plant are presented in Figures 11-2 through 11-4. Table 11-4 presents the material balance for the commercial plant. The anticipated clean coal analyses and plant performance indices are listed in Table 11-5. Major equipment is listed in Table 11-6. The plant is divided into the following sections:

- ROM coal receiving, crushing, and storage
- Conventional coal cleaning
- Magnetite recovery
- Selective grinding
- Spherical agglomeration
- Water clarification and refuse disposal
- Clean coal loadout


VENT
DUST COLLECTOR
SOLIDS
ROM COAL
TRUCKS
DUMP HOPPER CONVEYOR
DUMP HOPPER PIT
SUMP PUMP
1¼ - IN .. 0
STORAGE FEED CONVEYOR
SCRAPER RECLAIMER
ESCAPE TUNNEL
SUMP PUMP



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△0	6-25 90	DRAFT REPORT

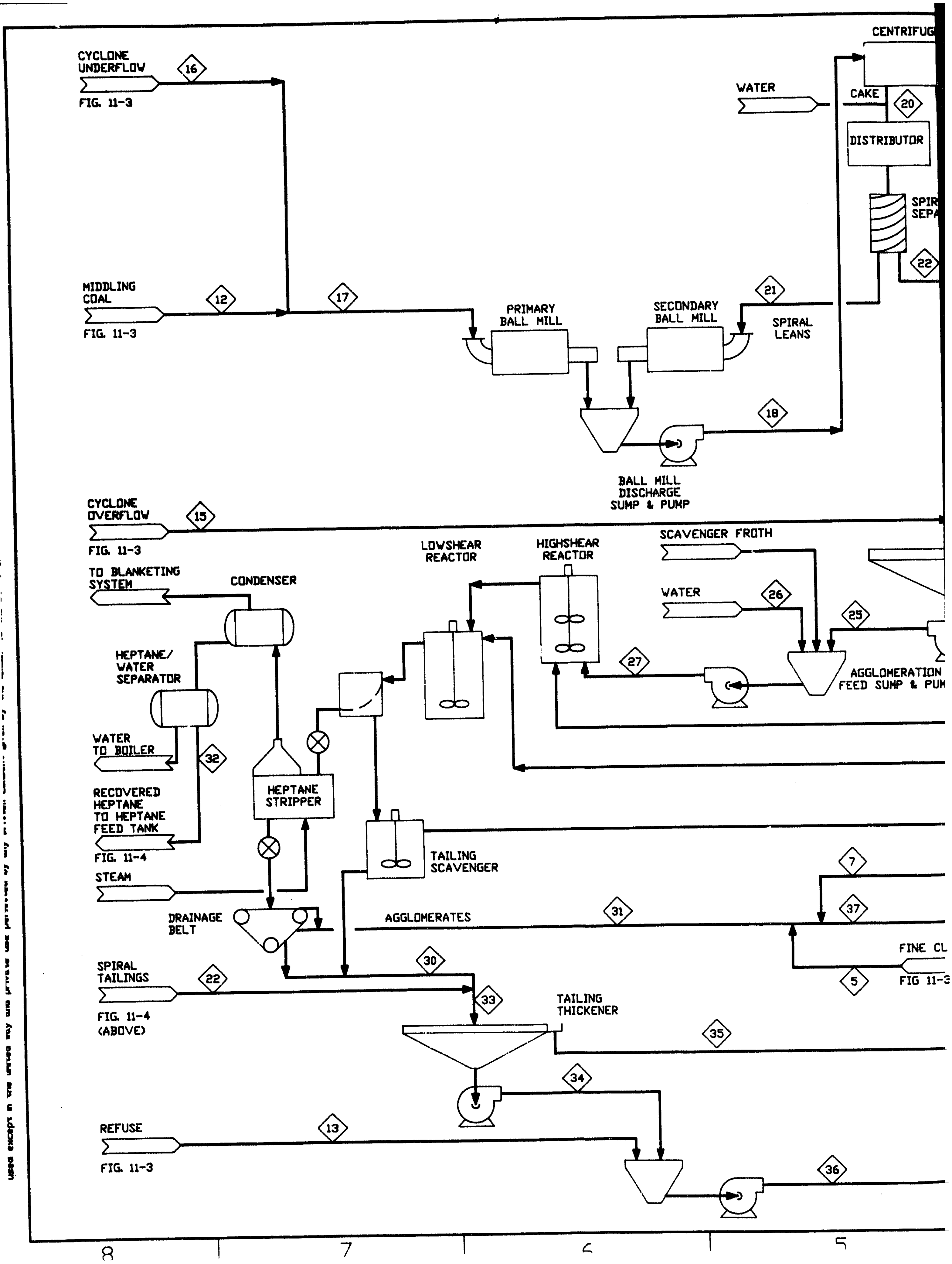
NONE YS/ACAD

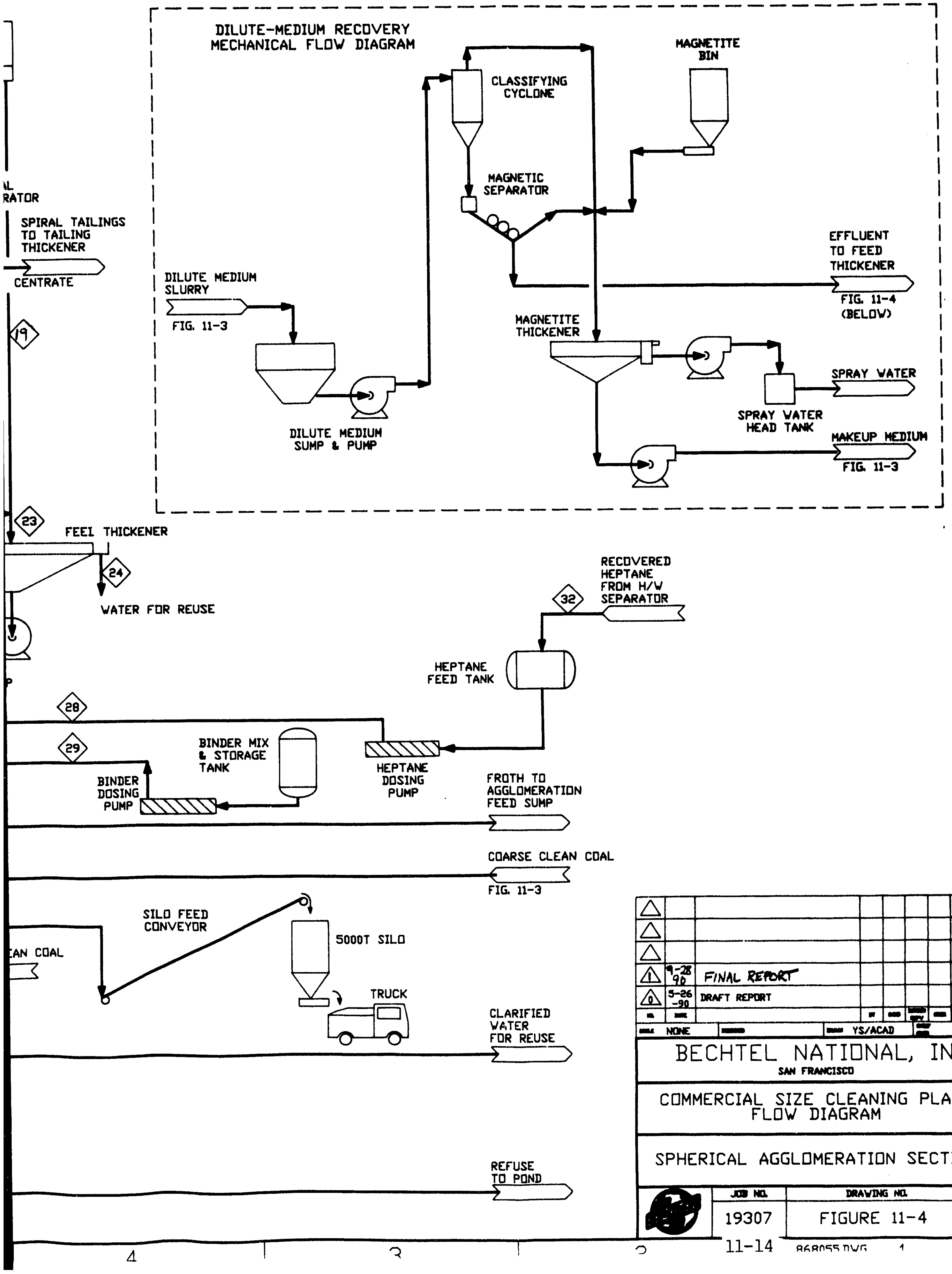
BECHTEL NATIONAL, INC.
SAN FRANCISCO

COMMERCIAL SIZE CLEANING PLANT
FLOW DIAGRAM

COAL RECEIVING & STORAGE SECTION

JOB NO.	DRAWING NO.	REV.
11-13	FIGURE 11-2	0


CYCLONE UNDERFLOW
16
FIG. 11-3
WATER
CENTRIFUG
CAKE
20
DISTRIBUTOR
SPIR
SEPA
22
MIDDLING COAL
12
FIG. 11-3
17
PRIMARY BALL MILL
SECONDARY BALL MILL
21
SPIRAL LEANS
18
BALL MILL DISCHARGE SUMP & PUMP
CYCLONE OVERFLOW
15
FIG. 11-3
SCAVENGER FROTH
LOWSHEAR REACTOR
HIGHSHEAR REACTOR
TO BLANKETING SYSTEM
CONDENSER
WATER
26
25
HEPTANE/ WATER SEPARATOR
27
AGGLOMERATION FEED SUMP & PUM
WATER TO BOILER
32
HEPTANE STRIPPER
RECOVERED HEPTANE TO HEPTANE FEED TANK
FIG. 11-4
TAILING SCAVENGER
7
STEAM
37
DRAINAGE BELT
AGGLOMERATES
31
FINE CL
FIG 11-3
5
SPIRAL TAILINGS
30
22
33
TAILING THICKENER
FIG. 11-4 (ABOVE)
35
34
REFUSE
13
FIG. 11-3
36
8
7
6
5



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△0	5-26-90	DRAFT REPORT

NONE YS/ACAD

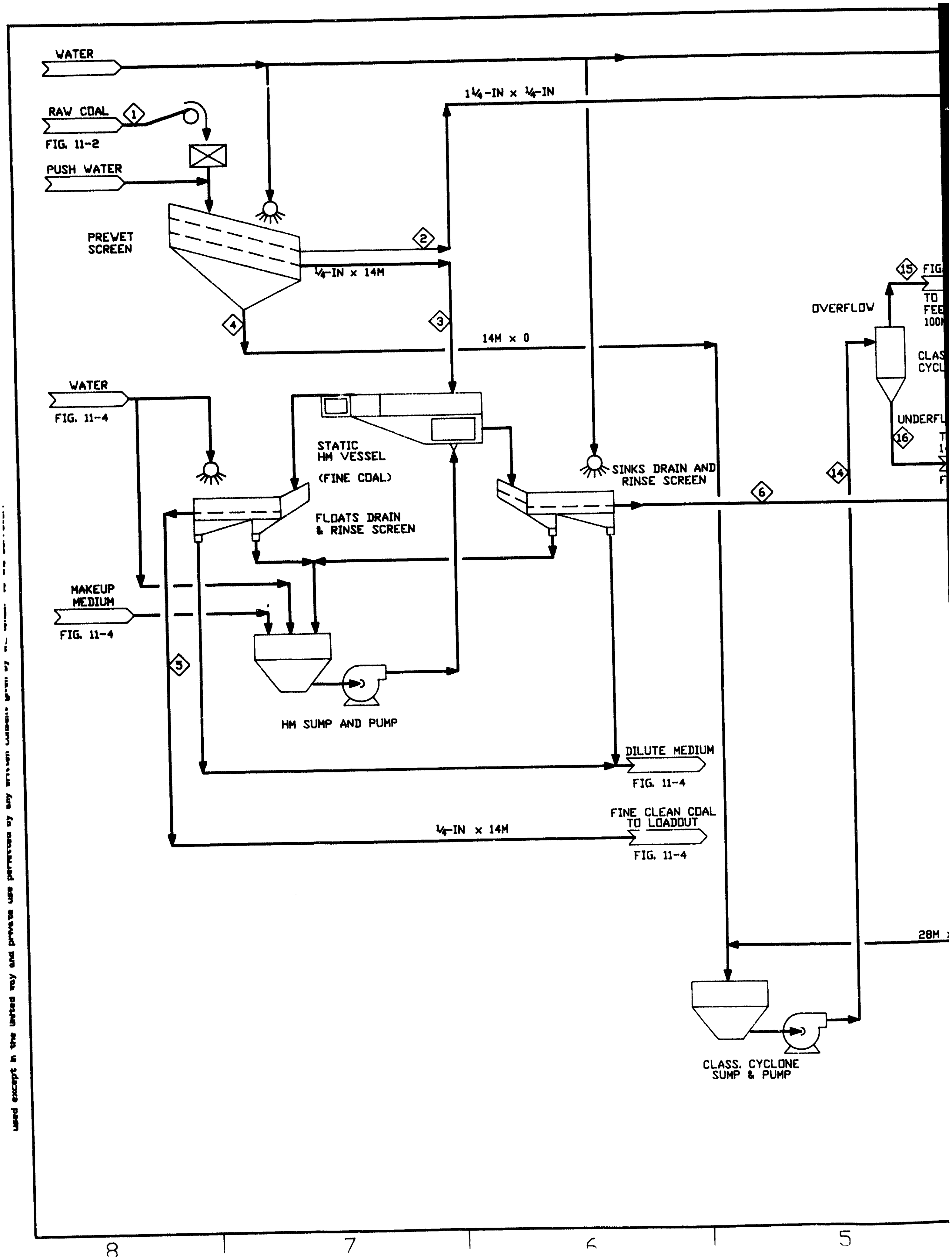
BECHTEL NATIONAL, INC.
SAN FRANCISCO

COMMERCIAL SIZE CLEANING PLANT
FLOW DIAGRAM

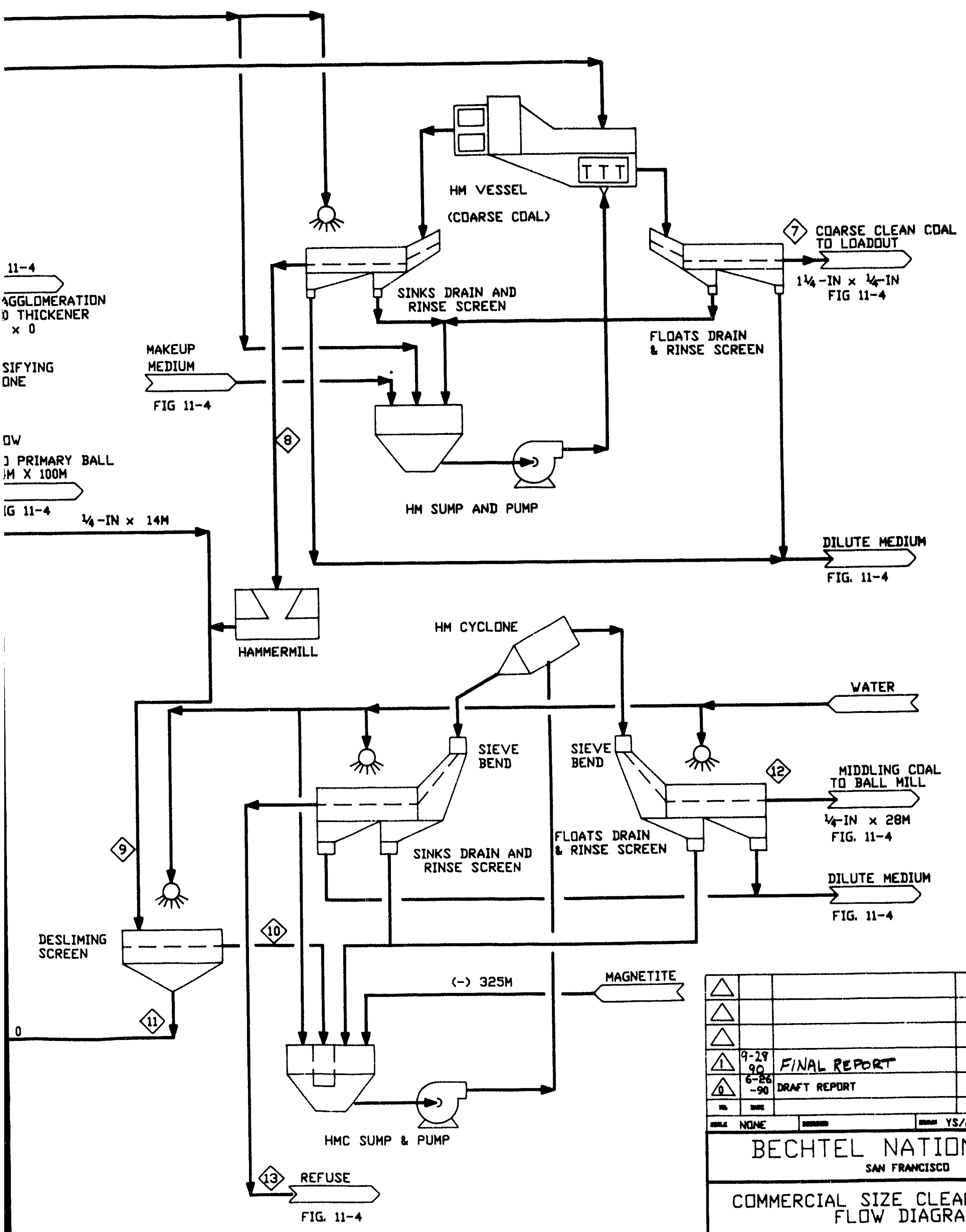
SPHERICAL AGGLOMERATION SECTION

JOB NO.	DRAWING NO.	REV.
19307	FIGURE 11-4	0

868055.DWG 1



WATER
RAW COAL
1
FIG. 11-2
PUSH WATER
PREWET SCREEN
2
1¼-IN x ¼-IN
¼-IN x 14M
3
4
14M x 0
15
FIG
TO
FEE
100M
OVERFLOW
CLAS
CYCL
UNDERFL
16
T
1
F
14
WATER
FIG. 11-4
STATIC HM VESSEL
(FINE COAL)
SINKS DRAIN AND RINSE SCREEN
6
FLOATS DRAIN & RINSE SCREEN
MAKEUP MEDIUM
FIG. 11-4
5
HM SUMP AND PUMP
DILUTE MEDIUM
FIG. 11-4
FINE CLEAN COAL TO LOADOUT
¼-IN x 14M
FIG. 11-4
28M
CLASS. CYCLONE SUMP & PUMP
8
7
6
5



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0	6-26-90	DRAFT REPORT

SCALE NONE
DRAWN YS/ACAD

BECHTEL NATIONAL, INC.
SAN FRANCISCO

COMMERCIAL SIZE CLEANING PLANT FLOW DIAGRAM

CONVENTIONAL CLEANING SECTION

JOB NO.	DRAWING NO.	REV.
19307	FIGURE 11-3	0

Table 11-4

SPHERICAL AGGLOMERATION - COMMERCIAL PLANT

MATERIAL BALANCE

FREEPORT SEAM COAL

STREAM NUMBER	1	2	3	4	5	6	7	8	9	10	11	12	13
	RAW COAL	**COARSE COAL**	**FINE COAL**	**FINE SLURRY**	**FINE COAL 3-6**	**FINE COAL 3-5**	**COARSE COAL 2-8**	**COARSE COAL 2-7**	**FEED TO DESLIMING SCREEN**	**DESLIMED COAL**	**DESLIMING SCREEN UNDERFLOW**	**MIDDLINGS**	**REFUSE**
SIZE (INCH OR MESH)	1-1/4 X 0	1-1/4 X 1/4	1/4 X 14M	14M X 0	1/4 X 14M	1/4 X14M	1-1/4 X 1/4	1-1/4 X 1/4	1/4 X 0	1/4 X 28M	28M X 0	1/4 X 28M	1/4 X 28M
SP. GR. FRACTION					1.3 FLOAT	1.3 SINK	1.3 FLOAT	1.3 SINK				1.8 FLOAT	1.8 SINK
• SOLIDS (TPH)	350	145	118	88	73	45	49	96	140	131	10	116	15
• SOLIDS (%)	92	96	88	10	88	88	96	96	92	86	10	86	86
• WATER (GPM)	122	24	64	3,150	40	24	8	16	49	85	344	75	10
• WT % ASH	12.1	15.0	10.3	9.7	3.8	21.1	5.0	20.1	20.4	20.4	20.1	14.6	65.0
• WT % SULFUR	1.90	1.77	2.00	1.97	0.82	3.94	0.80	2.27	2.80	2.84	2.27	1.84	10.46
• BTU CONTENT/LB	13,639	13,183	13,928	14,003	15,086	12,023	14,837	12,338	12,237	12,230	12,338	13,230	4,600
• LB SO2/MBTU	2.8	2.7	2.9	2.8	1.1	6.6	1.1	3.7	4.6	4.6	3.7	2.8	45.5

STREAM NUMBER	14	15	16	17	18	19	20	20A	21	22	23	24	25	26
	CYCLONE FEED	**CYCLONE OVERFLOW**	**CYCLONE UNDERFLOW**	**PRIMARY BALL MILL FEED**	**CENTRIFUGE FEED 17**	**GROUND PRODUCT**	**CENTRIFUGE CAKE**	**SPIRAL FEED**	**FEED TO SEC. MILL**	**SPIRAL TAILINGS**	**FEED THICKENER IN FLOW**	**THICKENER OVERFLOW**	**THICKENER UNDERFLOW**	**DILUTION WATER**
SIZE (INCH OR MESH)	14 M X 0	100M X 0	14M X 100M	1/4 X 100M	100M X 0	< 325M	14M X 325M	14M X 325M	> 325M	14M X 325M	< 325M			
SP. GR. FRACTION				16+12	17+21	18-20	18-19	18-19	1.8 FLOAT	1.8 SINK				
• SOLIDS (TPH)	97	58	39	154	293	139	154	154	139	15	197		197	
• SOLIDS (%)	10	7	30	59	29	18	35	30	28	35	12		30	
• WATER (GPM)	3,494	3,132	362	438	2,864	2,532	332	1,441	1,407	33	5,664	3,823	1,840	1,314
• WT % ASH	10.7	13.0	7.3	12.7	16.7	6.6	25.7	25.7	21.0	68.0	8.5		8.5	
• WT % SULFUR	2.00	1.80	2.30	1.95	3.16	1.00	5.10	5.10	4.50	10.50	1.24		1.24	
• BTU CONTENT/LB	13,839	13,349	14,574	13,568	12,908	14,518	11,458	11,458	12,174	4,725	14,173		14,173	
• LB SO2/MBTU	2.9	2.7	3.2	2.9	4.9	1.4	8.9	8.9	7.4	44.4	1.7		1.7	

STREAM NUMBER	27	28	29	30	31	32	33	34	35	36	37
	AGGLOMERATION FEED	**HEPTANE**	**BINDER MIX**	**AGGLOMERATION TAILINGS**	**AGGLOMERATES**	**RECOVERED HEPTANE**	**REFUSE THICKENER FEED**	**REFUSE THICKENER UNDER FLOW**	**REFUSE THICKENER OVERFLOW**	**TOTAL REFUSE**	**CLEAN COAL**
SIZE (INCH OR MESH)	< 325M			< 325M	3/8			< 325M		1/4 X 0	1-1/4 X 0
SP. GR. FRACTION											
• SOLIDS (TPH)	197			10	188		25	25		40	310
• SOLIDS (%)	20			1	85		3	30		40	87
• WATER (GPM)	3,155			3,022	132		3,056	233	2,822	243	181
• WT % ASH	8.5			86.8	4.5		75.2	75.2		71.3	4.4
• WT % SULFUR	1.24			11.84	0.70		11.01	11.01		10.80	0.74
• BTU CONTENT/LB	14,173			805	14,853		3,227	3,227		3,745	14,906
• LB SO2/MBTU	1.7			294.1	0.9		68.3	66.3		57.7	1.0
HEPTANE (TPH)		49	12			61					
BINDER (TPH)			5		5						

• Dry Basis

Table 11-5
SPHERICAL AGGLOMERATION - COMMERCIAL PLANT
PLANT PERFORMANCE
(Dry Basis)

COAL ANALYSIS	ROM COAL	CLEAN COAL
Ash, %	12.1	4.4
Total Sulfur, %	1.9	0.7
Pyritic Sulfur, %	1.3	0.1
Heating Value, Btu/lb	13,639	14,906
Sulfur Dioxide, Lb/MMBtu	2.8	1.0
Clean Coal HV, Btu/lb		15,020
Plant Yield, %	88.5	
Btu Recovery, %	96.8	
Sulfur Reduction, %*	64.1	
Pyrite Reduction, %*	93.0	

---

* MMBtu Basis

Table 11-6

SPHERICAL AGGLOMERATION - COMMERCIAL PLANT

MAJOR EQUIPMENT LIST

	I.D. NO.	Description	Qty	Unit	Capacity Each	Installed Total HP
Coal Receiving						
	110-1	Raw Coal Feeder	3	tph	250	15
	110-2	Dump Hopper Conveyor	1	tph	700	60
	110-3	Belt Scale	1	tph	700	0
	110-4	Sump Pump	2	gpm	100	10
	110-5	Dust Collector	1	cfm	7,000	25
	110-6	Ventilation Fan	2	cfm	10,000	6
ROM Coal Crushing						
	223-1	2-Stg. Triple Roll Crusher	2	tph	350	250
	223-2	Tramp Iron Magnet	1	tph	700	15
	223-3	Raw Coal Conveyor	1	tph	700	60
	223-4	Crushed Coal Conveyor	1	tph	700	50
	223-5	Dust Collector	1	cfm	5,500	15
Storage and Reclaim						
	350-1	Stacker/Reclaimer	1	tph	700/350	220
	350-2	Storage Feed Conveyor	1	tph	700	60
	350-3	Reclaim Conveyor	1	tph	350	30
	350-4	Reclaim Feeder	2	tph	350	10
	350-5	Sump Pump	1	gpm	150	10
	350-6	Run-off Water Pump	1	gpm	500	30
	350-7	Ventilation Fan	2	cfm	10,000	6
Conventional Coal Cleaning						
	520-1	Prewet Screen	2	tph	175	30
	520-2	Heavy Medium Vessel	1	tph	150	15
	520-3	Floats D&R Screen	1	tph	100	15
	520-4	Floats D&R Fixed Sieve	1	gpm	1,200	0
	520-5	Sinks D&R Screen	1	tph	100	15
	520-6	Sinks D&R Fixed Sieve	1	gpm	600	0
	520-7	HM Feed Pump	1	gpm	1,800	75
	520-8	Static Heavy Medium Vessel	1	tph	150	0
	520-9	Floats DR Screen	1	tph	100	15
	520-10	Floats D&R Fixed Sieve	1	gpm	1,200	0
	520-11	Sinks D&R Screen	1	tph	100	15
	520-12	Sinks D&R Fixed Screen	1	gpm	600	0
	520-13	HMC Feed Pump	1	gpm	1,800	75
	520-14	Classifying Cyclone	9	gpm	440	0
	520-15	Classifying Cyclone Pump	3	gpm	1,320	150
	520-16	Not Used	0	-	-	0
	520-17	Hammermill	1	tph	100	150
	530-2	Desliming Screen	2	tph	75	40
	530-3	HM Feed Pump	1	gpm	3,600	250
	530-4	Heavy Medium Cyclone	1	gpm	1,200	0
	530-5	Float D&R Sieve Bend	3	gpm	950	0
	530-6	Float D&R Screen	3	tph	50	45
	530-7	Sink D&R Sieve Bend	2	gpm	500	0
	530-8	Sink DR Screen	1	tph	50	15

Table 11-6 (Cont'd)

	I.D. NO.	**Description**	**Qty**	**Unit**	**Capacity Each**	**Installed Total HP**
Magnetite Recovery						
	580-1	Magnetic Separator	2	gpm	800	4
	580-2	Dilute Medium Pump	1	gpm	2,000	100
	580-3	Classifying Cyclone	2	gpm	1,000	0
	580-4	Thickener	1	gpm	1,500	5
	580-5	Thickener U'flow Pump	1	gpm	120	20
	580-6	Thickener O'flow Pump	1	gpm	1,000	75
	580-7	Bin Reclaim Feeder	1	tph	3	3
Grinding and Agglomeration						
	900-1	Primary Ball Mill	1	tph	160	1,800
	900-2	Bill Mill Discharge Sump	1	gal	7,000	0
	900-3	Ball Mill Discharge Pump	3	gpm	1,200	75
	900-4	Centrifuge	6	tph	50	1200
	900-5	Distributor- 12 way	3	gpm	1,200	0
	900-6	Spiral Separator(3 trough)	12	tph	13	0
	900-7	Secondary Ball Mill	3	tph	50	5,400
	900-8	Feed Thickener	1	gpm	6,000	25
	900-9	Not Used				
	900-10	Not Used				
	900-11	Underflow Pump	1	gpm	2,200	50
	900-12	Distributor- 8 way	1	gpm	2,200	15
	900-13	Agglomeration Feed Sump	8	gal	1,500	0
	900-14	Agglomeration Feed Pump	8	gpm	520	80
	900-15	Heptane Feed Tank	2	gal	35,000	0
	900-16	Heptane Dosing Pump	8	gpm	50	40
	900-17	Binder Mix & Storage Tanks	2	gal	9,000	0
	900-18	Binder Dosing Pump	8	gpm	13	5
	900-19	High Shear Reactor	16	gal	260	1,600
	900-20	Low Shear Reactor	24	gal	1,733	720
	900-21	Sieve Bends	8	gpm	500	0
	900-22	Tailings Scavenger	8	gpm	400	200
	900-23	Froth Pump	2	gpm	100	15
	900-24	Heptane Stripper	4	tph	50	40
	900-25	Condenser	4	tph	65	0
	900-26	Oil Water Separator	4	gal	1,000	0
	900-27	Drainage Belt	4	tph	50	80
	900-28	Boiler (evaporation capacity)	1	tph	64	50
Water Classification						
	710-1	Tailings Thickener	1	gpm	3,200	8
	710-2	Tailings Underflow Pump	1	gpm	250	5
	710-3	Clarified Water Pump	1	gpm	3,000	75
	700-4	Tunnel Sump Pump	2	gpm	100	10
	700-4	Flocculant Feed System	1	NA	NA	5
	700-5	Tailings Disposal Pump	1	gpm	280	15
Clean Coal Loadout						
	810-1	Bin Activator	1	NA	NA	10
	810-2	Vibrating Feeder	1	tph	600	5
	810-3	Storage Feed Conveyor	1	tph	350	75
	810-4	Storage Silo	1	tons	5,000	0
		TOTAL INSTALLED HP				**13,587**

NA = Not Applicable

I.D. No. As per Bechtel/EPRI Cost Model
except 900 Series

11.3.1 ROM Coal Receiving, Crushing, and Storage

As shown in Figure 11-2, the coal is received in trucks and unloaded at a rate of 700 tph. The coal is crushed to a nominal top size of 1-1/4 inch and sent to a 20,000-ton (live) capacity circular homogenization pile. The coal is reclaimed and fed to the cleaning plant at a rate of 350 tph. The storage capacity of the pile represents more than 2 days of continuous plant consumption.

11.3.2 Conventional Coal Cleaning

Figure 11-3 shows the conventional cleaning system using HM vessels and cyclones.

The raw coal (1-1/4 inch x 0) is wet screened on double deck screens to obtain three size fractions: 1-1/4 inch x 1/4 inch, 1/4 inch x 14M, and 14M x 0. The coarsest fraction is treated in conventional HM vessels. The 1/4 inch x 14M material is sent to a static bath HM vessel. All HM vessels operate at a specific gravity of 1.3.

For the smaller size fraction, a static bath HM vessel was chosen over HM cyclones, because unlike cyclones, the static vessel is able to separate coal at the specific gravity of the circulating medium. For such a low gravity separation, HM cyclones would have required using a circulating medium with a specific gravity lower than 1.3. At such low specific gravities, the medium is unstable.

A narrow size range of 1/4 inch by 14M has been selected for the static bath feed. This will facilitate efficient separation under the difficult operating conditions characterized by the low specific gravity split.

The floats from the coarse coal HM separators and the fine coal static bath separators are combined with the agglomerates and loaded as clean coal.

The sinks from the coarse coal HM separators are crushed to 1/4 inch in a hammermill, combined with the sinks from the static bath separators, and deslimed. HM cyclones, operating at a specific gravity of 1.8, are used to clean the combined and deslimed HM sinks from the vessels. Floats from the cyclones which constitute the middlings are sent to the selective grinding circuit. HM cyclone sinks are rinsed, dewatered, and discarded as part of the plant refuse.

The 28M x 0 fraction separated by the desliming screens and the natural 14M x 0 fraction from the raw coal are combined and pumped to a bank of classifying cyclones for separation at 100M. The cyclone overflow consisting of minus 100M fraction and most of the water is sent to a static thickener in the agglomeration circuit. The underflow from the cyclones, the plus 100M slurry, is sent to the primary ball mill in the selective grinding circuit.

11.3.3 Magnetite Recovery

The magnetite recovery circuit is shown in Figure 11-4.

A suspension of very fine (minus 325M) magnetite in water is used as the heavy media in the vessels and cyclones. The bulk of the heavy media is drained off the products at the drain and rinse screens. Additionally, water sprays are used to rinse the solids of adhering magnetite. Magnetite slurry recovered in the drain section of the screens is recycled through the vessels or cyclones. The dilute magnetite slurry (with some coal fines) recovered at the rinse section of the screens is classified in cyclones. Cyclone underflow is passed through multiple-drum magnetic separators. The magnetite rich slurry is then sent to a magnetite thickener. The magnetic separator effluent, containing coal fines and other non-magnetic material, is sent to the agglomeration feed thickener. The magnetite thickener provides concentrated magnetite slurry to the various HM circuits as needed. Magnetite thickener overflow is used as spray water on the rinsing screens. Heavy media losses are made up at the thickener by the addition of powdered magnetite from a storage bin.

11.3.4 Selective Grinding

The selective grinding circuit is shown in Figure 11-4. Selective grinding is used to reduce the size of feed to agglomeration to minus 325M and to reject coarse mineral matter and pyrite ahead of the agglomeration circuits. The effectiveness of the selective grinding circuit was clearly demonstrated in the POC test unit.

The combined floats from the HM cyclones and the underflow (14M x 100M) from the cyclone classifiers are ground to minus 100 M in the primary ball mill. The primary ball mill product is pumped to a bank of solid bowl centrifuges.

The use of solid bowl centrifuges is an essential element of the selective grinding concept. The feed to the centrifuges contains a mixture of particles of different sizes. Additionally, the mixture consists of material with a wide range of specific gravities. Due to their hardness, those particles rich in minerals and pyrite tend to occur in the coarser site fractions. Softer and low-ash coal is ground finer than its mineral laden counterpart and therefore occurs mostly in the smaller size fractions of the centrifuge feed. In the centrifuges, coarse coal and minerals are concentrated in the cake. Material ground finer than 325M overflows the centrifuge weirs forming the centrate slurry and feed to the agglomeration process.

A centrifuge does not sort particles based on size alone. Centrifuge performance is affected by particle densities as well. The centrifugal forces employed by the machine cause the high density ash and pyrite rich particles to be placed preferentially in the cake while coal particles of the same size report to the centrate. This density based separation further increases the concentration of mineral matter in the cake above what can be achieved by size-based sorting alone.

Downstream processing of the centrifuge cake in spiral separators removes most of the particles with minerals. Mineral matter and pyrite are separated as a refuse product. The clean coal from the spiral separators is fed to secondary grinding mills for intense grinding. The product from the secondary ball mills is combined with the primary ball mill product and again fed to the centrifuges.

The advantages derived from selective grinding are:

- Reduced load on the grinding mills and the agglomeration process leading to lower capital and operating costs
- Enhanced liberation of mineral matter from coal resulting in improved agglomerate quality
- Reduced overgrinding of low ash coal leading to further savings in grinding and agglomeration costs

11.3.5 Spherical Agglomeration

The spherical agglomeration circuit shown in Figure 11-4. The circuit is divided into the following sections:

- Preparation of microagglomerates
- Preparation of agglomerates
- Heptane recovery and agglomerate dewatering

Preparation of Microagglomerates. The first step in agglomeration is the preparation of microagglomerates in high-shear reactors followed by processing in low-shear reactors where microagglomerates are grown in size. The high- and low-shear reactors are arranged in eight parallel trains of 25 tph capacity each. Each train consists of two high-shear reactors in series followed by three low-shear reactors, also in series.

Feed to agglomeration consists of approximately 155 tph ground coal from the selective grinding circuit 58 tph the overflow from the cyclone classifiers. The combined slurry is concentrated in a static thickener. The thickener also acts as surge storage ahead of agglomeration.

The underflow from the feed thickener together with the froth from the tailings scavenger, 18 any, (is diluted to the required solids consist for agglomeration (20 percent solids by weight). Variable speed agglomeration feed pumps deliver slurry at a pre-set rate to the high-shear reactors. Heptane, the bridging liquid, is also pumped into the reactors in measured quantities by a dosing pump. At the high shear reactors, under conditions of intense shear, microagglomerates of coal are formed. While the coal matter in the feed slurry coalesce to form microagglomerates, the non-coal mineral matter remains in the slurry as discrete particles. The high shear reactors are connected to the low-shear reactors where the agglomerates are increased in size.

Preparation of Agglomerates. The microagglomerates are grown, in stages, in the low shear reactors by the addition of asphalt dissolved in heptane and under conditions of gentle agitation. Agglomerates of the required size overflow from the last low-shear reactors of each train over sieve bends. The underflow from the sieve bends is sent to tailings scavenger flotation units to reclaim any broken coal agglomerates. Froth from the scavengers units is returned to the agglomeration feed sump. Scavenger tailings are sent to the tailings thickener.

Heptane Recovery and Agglomerate Dewatering. Heptane is recovered from the agglomerates in a travelling slat-type steam stripper. Agglomerates are slowly drawn through a boiling water bath heated by steam. The heptane and steam azeotrope is condensed and heptane decanted from the water in an oil water separator. Condensed steam is recycled to the boiler.

Steamed agglomerates are removed from the heptane strippers, cooled and drained of water on drainage belts. The underflow from the drainage belts is directed to the tailings thickener.

To safeguard against risk of explosion, all vessels containing heptane are sealed and kept under a positive pressure of nitrogen by a blanketing system. This will eliminate possibility of heptane vapors and oxygen forming an explosive mixture. The nitrogen blanketing system includes a gas holder that controls the system pressure in addition to serving as a surge reservoir to accommodate changes in the fill volume in the tanks and vessels of the system. Should the gas holder become full for any reason, some of the nitrogen is vented though a flare. A refrigerated water-cooled condenser located between the flare and gas holder helps to recover most of the heptane in the gases before it is flared.

A comprehensive ventilation and fire protection system is also included to ensure safety at all times.

11.3.6 Water Clarification and Refuse Disposal

The following refuse streams are generated in the plant:

- o Coarse refuse from HM cyclones (1/4 inch x 28M)
- o Spiral tailings (100M x 0)
- o Agglomeration tailings from sieve bends and drainage belts (325M x 0)

The tailings from the spiral separators and the agglomeration circuit are collected into a tailings thickener and concentrated to a solids consistency of 30-35 percent. Clear water from the thickener overflow is recycled back into the plant. The underflow is pumped to a disposal pond. Course refuse is shipped to a landfill.

11.3.7 Clean Coal Loadout

Coarse clean coal from the heavy medium vessels, fine clean coal from the static heavy medium vessels, and the agglomerates are collected with a feed conveyer and delivered to a 5,000-ton silo. A vibrating feeder located below the silo is used to load the clean coal, for example, into trucks.

11.4 CAPITAL AND OPERATING COSTS

Table 11-7 shows a summary of the order-of-magnitude capital cost estimate for the commercial-scale plant. The plant is estimated to cost $66.6 million. Annual operating and maintenance (O&M) costs including capital charges are shown in Table 11-8. An annual processing cost of $26 million is estimated which corresponds to approximately $16 per ton. Cost of the ROM coal is not included in these costs. Annual O&M costs are summarized in Table 11-9.

Table 11-7

SPHERICAL AGGLOMERATION - COMMERCIAL PLANT

FINE COAL CLEANING WITH AGGLOMERATION

CAPITAL COST SUMMARY

(3rd Qtr 1990)

Category	Capital Costs ($ x 1000)
Plant and Equipment	18,600
Field Materials	12,000
Field Labor	11,300
Subcontract	2,600
Total Direct Field Cost	44,500
Distributables	9,600
Total Field Cost	54,100
Engineering & Home Office	3,800
Contingency	8,700
Total Installed Cost	66,600

Table 11-8

SPHERICAL AGGLOMERATION - COMMERCIAL PLANT

ANNUAL O & M COSTS

Feed Rate, tph (Dry Basis)	350
Annual Scheduled Operating Hours	6,000
Annual Effective hours	5,220
Availability	0.87
Annual Production, tons (Dry Basis)	1,644,000

			x $1000 Year	$/ton (Clean Coal)
A	**Fixed O & M Costs**			
1)	Operating and maintenance Labor	Including Admin.	2,000	1.22
2)	Maintenance Materials & Supplies	@ 6% of Total Direct Field Cost	2,670	1.62
	Total Fixed O & M Costs (A)		4,670	2.84
B	**Variable O & M Costs**			
3)	Magnetite		180	0.11
4)	Grinding Media		564	0.34
5)	Binder and Heptane		3,856	2.35
6)	Natural Gas		2,355	1.43
7)	Flocculants		157	0.10
8)	Operating supplies @ 2 % of Total Field Cost		1,082	0.66
9)	Electric Power		2,582	1.57
10)	Refuse Disposal		326	0.20
	Total Variable O & M Costs (B)		11,102	6.75
C	**Capital Charges @ 15% interest and 25 years**			
Factor		0.15		
	Capital Charges (C)		**10,303**	**6.27**
D	**Total O & M and Cap. Charges (A+B+C)**		**26,075**	**15.86**

Table 11-9
SPHERICAL AGGLOMERATION - COMMERCIAL PLANT
ANNUAL O & M COSTS - BASIS

ROM Coal		
Consumption	MM ton/year	1.83
Cost		Not Included
Operating & Maintenance Labor		
Number required	Each	40
Cost Including Administration	$/year	1,440
Magentite		
Consumption	ton/year	1,440
Cost	$/ton	1,000
Grinding Media		
Consumption	ton/year	550
Cost	$/ton	1,000
Refuse Disposal		
Annual Production (Dry Basis	ton/year	130,000
Cost	$/ton	2.5
Binder and Heptane		
Consumption	ton/year	25,800
Cost	$/ton	150
HV of Binder	Btu/lb	22,000
Natural Gas		
Consumption	MMBtu/year	785,000
Cost	$/MMBtu	3.0
Power		
Operating Hp	HP	13,000
Equivalent	kW	9.700
Load Factor	%	85
Operating hours	Hrs/year	5,220
Power Consumption	MWh/year	43,000
Cost	$/kWh	0.06s

Section 12

LESSONS LEARNED AND FUTURE TECHNOLOGY DEVELOPMENT

12.1 SPHERICAL AGGLOMERATION PROCESS FEATURES

The program represented the first successful application of a spherical agglomeration process for coal using a light hydrocarbon (heptane) bridging liquid at a scale of 1/3 tph. This section summarizes the lessons learned and highlights areas that need further work to develop the technology to a commercial scale.

12.2 PROCESS TECHNOLOGY CONSIDERATIONS

The lessons learned are as follows:

- Agglomeration with heptane rejects significant quantities of pyrite (over 80 percent) provided that the pyrite is liberated from the coal (refer to Process Evaluation, Sections 8.2 and 8.6).
- Agglomeration can reject pyrite from fine coal (minus 325M). This cleaning capability may be used to enhance conventional coarse coal cleaning processes (Process Evaluation, Sections 8.2 and 8.6).
- Agglomeration process refuse contains 85 to 88 percent ash. Energy recovery is from 98 to 99.9 percent (Process Evaluation, Sections 8.1 and 8.3).
- POC test results are consistent with the bench-scale batch test results, indicating that scale-up to larger scale plants is feasible (Process Evaluation, Section 8.1 and Tables 8-3, 8-5, and 8-7).
- Only a single stage of high-shear agglomeration was required to the meet process performance requirements. Originally, the design used two stages with a screen between the stages (Process Evaluation, Section 8).
- After steam stripping, less than 0.2 percent heptane remains in the final agglomerates (Coal Characteristics, Section 9).
- The 3/8 inch steam-stripped agglomerates were dewatered to less than 30 percent moisture on fixed screens (Process Operation, Section 6).

- Sodium may have accumulated in the recirculated water supply from a number of sources and concentrated in the agglomerates. Buildup of sodium in the larger-scale plant should be closely monitored (Coal Characteristics, Section 9).
- The liberation method, which ensures effective grinding of hard-to-grind and high ash/pyrite particles, was a major factor affecting clean coal quality and process performance (Process Evaluation, Sections 8.2 and 8.3).
- Petrographic analysis noted that pyrite was ground to 9 microns or less in selectively ground coal samples with a top size of 20 microns, demonstrating the effectiveness of selective grinding in the liberation of pyrite (Process Evaluation, Section 8.6).
- Agglomeration with heptane produced better separations than centrifugal float/sink of micronized coal possibly due to the multiple formation and dispersion of microagglomerates in the high-shear reactor (Process Evaluation, Section 8.5).

12.3 ENGINEERING

The lessons learned are as follows:

- High-shear reactor bench-scale test results were successfully used to scale equipment to the POC size.
- The POC high-shear reactor's range of performance was tested by agglomerating several coals (Evaluation of High-Shear Reactor Performance, Section 6.3).
- The low-shear reactor design was a compromise between budget and function. The reactor was multipurpose, including steam stripping of the heptane from the agglomerates (Review of Test Module Performance, Section 6.2).
- Steam stripping of heptane from the agglomerates was the best method for recovering heptane. As long as the agglomerates contain at least one-fifth as much water as heptane, essentially all of the heptane vaporizes before the pellets reach the boiling point of water. In addition, the product retains residual water which is desirable for safe handling and storage (Letter from J. Getsoian to H. Huettenhain, 3/6/87).

- Safety concerns and the batch steam stripping step, combined with the variable operating conditions made total heptane recovery difficult and POC unit stream monitoring and tracking of heptane impractical (Review of Test Module Performance, Section 6.2).
- The safety systems used for the POC plant proved to be sufficient. Systems for a commercial plant would require site- and plant-specific reappraisals (Process Operation, Sections 6 and 6.4).

12.4 FUTURE TECHNOLOGY DEVELOPMENT

The next recommended step to develop a commercial technology is a demonstration plant for continuous operation of the heptane recovery system over a long period of time (not less than 1 year). A major goal is to improve thermal efficiency, minimize heptane consumption, and reduce other operating costs.

Further development work for the selective grinding circuit is required; the relationships of grinding level and clean coal quality, and grinding circuit costs should be fully examined. There is little information about efficiently grinding coal to micron sizes for maximum pyrite and mineral matter separation. The use of a solid bowl centrifuge as classifier requires further study and optimization of performance.

A commercial coal cleaning plant to produce ultra clean coal will be most likely a combination of spherical agglomeration and conventional coal cleaning. The feed to agglomeration would be a combination of natural fines and selectively ground middlings. Coarse coal would be cleaned by conventional processes to produce high quality clean coal products and very high ash and sulfur refuse. Several coal cleaning plants exist which could be extended to incorporate agglomeration. A search for suitable sites should be conducted.

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