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UTILIZATION OF COAL ASSOCIATED MINERALS

Final Report for Period September 1977—September 1980

By

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West Virginia University
Coal Research Bureau
Morgantown, West Virginia

U. S. DEPARTMENT OF ENERGY

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SECTION A - Summary and Conclusions

Under contract number DE-AS21-77ET10533 with the US-DOE several methods of utilizing coal associated by-products were examined for potential commercial use. Such use could transform a costly waste disposal situation into new materials for further use and could provide incentive for the adoption of new coal utilization processes. As a result of the research project, several utilization processes appear to have merit and are recommended for further study. Each process is discussed separately in the text of this report.

Common coal cleaning processes were also examined to determine the effect of such processes on the composition of by-products. Data obtained in this portion of the research effort is reported in the Appendix. Information of this type is required before utilization processes can be considered. A knowledge of the mineral composition of these materials is also required before even simple disposal methods can be considered.

SECTION B - Objective and Scope

The purpose of this research program is to examine the effects of coal mineral materials on coal waste by-products utilization and to investigate new and improved methods for the utilization of waste by-products from the cleaning, combustion and conversion processing of coal. The intermediate objectives include: (1) the examination of the effects of cleaning, gasification and combustion of coal mineral materials; and (2) the changes which occur in the coal wastes as a result of both form and distribution of mineral materials in feed coals in conjunction with the coal treatment effects resulting from coal cleaning or either gasification or combustion.

SECTION C - Discussion and Results

It is the purpose of this report to present research findings primarily dealing with the utilization of coal waste by-products.

Eight areas of investigation were considered. They include:

1. The production of aerated concrete utilizing fluidized bed ash.
2. The utilization of fluidized bed flyash as a partial replacement for Portland cement.
3. The utilization of coal refuse as a raw material for the production of lightweight aggregate for use in structural concrete.
4. The investigation of fluidized bed ash for suitability as a component of sand-lime brick.
5. The examination of flyash from a fluidized bed to determine its suitability as a source material for insulating wool fiber production.
6. The investigation of fluidized bed ash as suitable aggregate in fired structural products.
7. The comparison of thermal insulating properties of fired flyash based structural products with clay structural products.
8. The assesment of the potential use of fluidized bed ash as a strip mine soil amendment agent.

The results are discussed below.

1. The production of aerated concrete utilizing fluidized bed ash as a part of the calcareous component of the aerated concrete was investigated. Compressive strength values ranging from 225 to 630 pounds per square inch were obtained.

This area of utilization appears to have promising potential and will be thoroughly investigated under Contract No. DE-AT21-79MC11284, Task Order No. 29 which is currently an active contract being executed by the Coal Research Bureau of West Virginia University.

2. Fluidized bed flyash was examined as a partial replacement for Portland cement. It was observed that the compressive strength of the specimens decreased with increasing use of fluidized bed flyash and also that the compressive strength of the specimens decreased more rapidly with use of uncalcined fluidized bed flyash than with use of calcined fluidized bed flyash. The uncalcined fluidized bed flyash contains approximately 30 percent carbon and the carbon can be removed by calcining.

3. The utilization of coal refuse as a raw material for the production of lightweight aggregate for use in structural products resulted in lightweight aggregate blocks which were on the average of 15 percent lighter and when tested for compressive strength, out performed the limestone blocks by an average of 15 percent.

This area of utilization appears to have potential and additional work needs to be done to determine the optimum combination of forming pressure and firing temperature for the coal refuse.

4. Fluidized bed ash was investigated for suitability as a component of sand-lime brick. Laboratory-scale tests indicated that the bed material had substantial cementitious properties and may have utilization potential as a binder in the manufacture of structural products. It is recommended that further testing be conducted to firmly establish the parameters for using bed material as a binder in these structural products.

5. Fluidized bed flyash was examined to determine its suitability as a source material for insulating wool fiber production.

The results of these preliminary tests indicate that fluidized bed flyash wool has potential as a substitute for commercially marketed insulating materials. In order to more completely evaluate fluidized bed flyash wool as an insulating material, a more detailed study is required especially relating to costs of manufacture and optimization of process variables and an indepth study of the physical and chemical properties of the material.

6. The suitability of fluidized bed ash as an aggregate in fired structural products was investigated. The utilization potential of fluidized bed ash as an aggregate in fired structural products was found to be unfeasible because of cracks and fissures in the structural products. The cracks and fissures are caused by the free lime in the bed ash hydrating and expanding after being wetted during the mixing cycle.

7. The thermal insulating properties of fired flyash based structural products and clay structural products were compared. The comparison has shown that, based on thermal conductivity determinations, the use of flyash brick in place of clay brick would save 40 percent of the heat which would normally be lost through a wall.

8. The potential use of fluidized bed ash as a strip mine soil amendment agent was investigated. The investigation indicated that fluidized bed ash does have potential as an aid in strip mine soil revegetation and perhaps as a substitute for agricultural lime. There are also indications that fluidized bed ash has potential as a soil stabilization agent. Further work needs to be done over a period of several growing seasons in this area.

A detailed description of the research efforts for each of the areas is as follows.

COAL BY-PRODUCT UTILIZATION STUDIES

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Aerated Concrete

Samples of spent limestone bed ash and flyash from the carbon burn-off cell were obtained from the fluidized bed combustion facility at Rivesville, West Virginia. The samples were taken during a steady state run and to insure representativeness, the samples were taken at intervals over a four hour period. During this steady state run, the feed coal size was $-1/2$ inch $+ 1/4$ inch and the feed limestone size was $-6 + 16$ mesh. It is thought that representative samples of the combustion by-products were obtained during this steady state run. However, different operating conditions may produce combustion by-products having slightly different characteristics.

The chemical analyses of the spent limestone bed ash and flyash from the carbon burn-off cell are presented in Table 1.

Aerated concrete is a lightweight structural material which consists of small non-communicating cells entrained in a calcium silicate matrix.

Various methods may be used to form the non-communicating cells in aerated concrete. Some of the methods are chemical, entraining, micropore and prefoaming.

The non-communicating cells in the aerated concrete discussed in this report were formed by a chemical method. Aluminum powder, which reacts with calcium hydroxide and releases hydrogen gas, was used to form the non-communicating cells in the aerated concrete.

The calcareous component of the calcium silicate matrix may consist of lime or portland cement. Spent limestone bed ash from the fluidized bed combustor was used as a part of the calcareous component of the aerated concrete discussed in this report.

The siliceous component of the calcium silicate matrix may consist of flyash or sand. Both a Northern West Virginia flyash and a Southern West Virginia flyash were used as the siliceous component of the aerated concrete discussed in this report.

The formation of cementitious monocalcium silicate hydrate, designated mineralogically as tobermorite, imparts compressive strength to aerated concrete.

At the present time, aerated concrete is used more throughout Europe than in the United States. The rising costs of other construction materials may make aerated concrete a more economically competitive construction material in the United States in the future.

Acceptable density values for commercial aerated concrete are from between 20-30 pounds per cubic foot for use as insulation to between 80-100 pounds per cubic foot for structural purposes. Compressive strength ranges from 600-800 pounds per square inch at 40-50 pounds per cubic foot to 5,000 pounds per square inch at 80 pounds per cubic foot.

The aerated concrete discussed in this report was produced using the following procedure. The flyash, spent limestone bed ash, and portland cement were mixed together in a muller type mixer to intimately mix these materials together.

The material was then transferred into a planetary type mixer.

Aluminum powder, which reacts with calcium hydroxide and releases hydrogen gas, was then added to the material. The mixing in the planetary mixer was continued briefly after the aluminum powder had been added.

The aerated concrete was cast into molds and allowed to stand overnight and was then transferred into an autoclave.

The aerated concrete was autoclaved at 175 pounds per square inch for 20 hours.

When the aerated concrete was removed from the autoclave, it was dried to a constant weight. The bulk density of the aerated concrete was calculated and compressive strength tests were performed.

The composition and physical properties of the aerated concrete discussed in this report are presented in Table 2.

It can be noted from Table 2 that when using aluminum power at a level of 0.06 percent, bulk densities ranging from 43 to 49 pounds per cubic foot are obtained. When using the Northern West Virginia flyash, the bulk density is higher than when using the Southern West Virginia flyash.

Compressive strengths ranging from 255 to 632 pounds per square inch are obtained. There is a direct relation between bulk density and compressive strength with those batches which have the higher bulk densities also having the higher compressive strengths.

It can be noted from Table 1 that the fluidized bed flyash has a loss on ignition (LOI) of 36.7 percent. This high loss on ignition reduces the available silica to the point where it is not possible to utilize this flyash as the siliceous component of aerated concrete without beneficiation to remove excess carbon.

Conclusion

Although aerated concrete usage is more common in Europe than in the United States it should gain acceptance if a plentiful and low cost source of raw materials is available. Test results indicate that fluidized bed bottom ash can serve as a source of the calcareous component. Assuming an efficient process, eg. a carbon burn-off or recovery process in line,

the flyash could also be used as a source of the siliceous component. The manufacture of aerated concrete appears to be technologically sound, but acceptance will be dependent upon economic and legal factors.

TABLE 1
CHEMICAL ANALYSES OF FLUIDIZED BED COMBUSTION BY-PRODUCTS

	<u>Bed Ash</u>	<u>Flyash</u>
SiO_2	24.10	30.10
Al_2O_3	8.31	13.40
Fe_2O_3	2.12	9.07
TiO_2	0.34	0.46
CaO	37.20	38.70
MgO	1.52	1.24
Na	0.19	0.23
K	0.65	0.90
C	1.19	30.24
S	8.01	3.85
LOI	3.7	36.7

TABLE 2
COMPOSITION AND PHYSICAL PROPERTIES
OF AERATED CONCRETE

BATCH #	1	2	3	4	5	6	7	8	9
Northern West Virginia Flyash	74.94	72.44	69.94	—	—	—	—	—	—
Southern West Virginia Flyash	—	—	—	74.95	72.45	69.95	74.94	72.44	69.94
Spent Limestone Bed Ash	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
Portland Cement (Type I)	15.00	17.50	20.00	15.00	17.50	20.00	15.00	17.50	20.00
Aluminum Powder	0.06	0.06	0.06	0.05	0.05	0.05	0.06	0.06	0.06
Water	40.00	40.00	40.00	40.00	40.00	40.00	40.00	40.00	40.00
Bulk Density Lbs/Cu. Ft.	48	49	48	52	53	52	45	43	44
Compressive Strength Lbs./Sq. In.	448	475	632	425	563	547	277	255	333

Fluidized Bed Flyash in Portland Cement

A sample of flyash from the carbon burn-off cell was obtained from the fluidized bed combustor facility at Rivesville, West Virginia as previously discussed.

The chemical analyses of the flyash is presented in Table 3.

From Table 3 it can be noted that the carbon content is 30.24 percent which is larger than the carbon content of a typical flyash. A typical flyash will have a carbon content ranging from 0 to 2 percent.

This flyash was calcined in the laboratory at 800°C in an oxidizing atmosphere in order to reduce the carbon content by oxidation. The carbon content of the calcined flyash was 0.70 percent.

Cubes were made of various mixtures of portland cement and flyash in order to study the effect of the carbon in the flyash upon compressive strength when the flyash is used as a partial replacement for portland cement.

The compressive strength specimens were made in accordance with the American Society for Testing and Materials Designation C109 "Compressive Strength of Hydraulic Cement Mortars". This designation covers determination of the compressive strength of hydraulic cement mortars using 2-inch cube specimens.

Table 4 presents the composition and compressive strength of the specimens.

Flyash from the fluidized bed combustor process was used as a replacement for portland cement at levels of 10, 20 and 30 percent.

The flyash was used in both the uncalcined state (containing carbon) and the calcined state in which the carbon had been removed by oxidation.

Two different types of portland cement were used. Type I cement is a regular portland cement and Type III cement is a high early strength portland cement.

The specimens were formed and cured in accordance with ASTM methods and compressive strength was tested at times of 24 hours, 3 days, 7 days and 28 days.

The compressive strength data is shown in Table 4 and is also shown graphically in Figures 1 and 2.

Figure 1 shows the data when using Type I cement and Figure 2 shows the data when using Type III cement.

Figure 1 indicates that when using Type I cement stronger compressive strengths are achieved when using calcined flyash than when using uncalcined flyash.

Figure 2 indicates that when using Type III cement the compressive strength data is inconclusive.

Conclusion

There are indications that the carbon in the uncalcined flyash decreases compressive strength. The flyash can be utilized by calcining it in order to remove the carbon by oxidation.

Table 3

Chemical Analysis of Flyash From Fluidized
Bed Combustor Process

SiO ₂	30.10
Al ₂ O ₃	13.40
Fe ₂ O ₃	9.07
TiO ₂	0.46
CaO	38.70
MgO	1.24
Na	0.23
K	0.90
C	30.24
S	3.85
LOI	36.7

Table 4

Composition and Compressive Strength of Specimens

<u>Specimen</u>	<u>Portland Cement. Type</u>	<u>Flyash</u>	<u>24 Hour</u>	<u>3 Day</u>	<u>7 Day</u>	<u>28 Day</u>
1	I	No	2160	2540	2700	3560
2	I	10% Uncalcined	1150	2100	2680	2800
3	I	20% Uncalcined	930	1090	1830	2150
4	I	30% Uncalcined	380	860	1090	1670
5	I	10% Calcined	1130	1920	2700	3400
6	I	20% Calcined	780	1410	1900	2640
7	I	30% Calcined	340	670	800	1970
8	III	No	2500	3660	3850	4460
9	III	10% Uncalcined	2400	2970	3770	4320
10	III	20% Uncalcined	1460	1910	2160	3050
11	III	30% Uncalcined	840	1270	1490	2290
12	III	10% Calcined	1890	3110	3150	3630
13	III	20% Calcined	1160	1510	1580	3880
14	III	30% Calcined	920	1320	1650	3330

FIGURE I
COMPRESSIVE STRENGTH OF SPECIMENS
TYPE I CEMENT

FIGURE 2
COMPRESSIVE STRENGTH OF SPECIMENS
TYPE III CEMENT

Lightweight Aggregate for
Structural Concrete

The purpose of the described research effort was to investigate the potential of coal refuse as a raw material for the production of lightweight aggregate (LWA) for use in structural concrete.

Coal refuse samples were collected from commercial preparation plants mining three different major coal seams. A sample of refuse from the Illinois #6 seam was collected in central Illinois, refuse from the Pocahontas #3 seam was collected in southern West Virginia, and refuse from the Pittsburgh #8 seam was collected in eastern Ohio. The samples were taken from hoppers fed by the main refuse conveyor belt. Eight 55 gallon drums were collected, sealed, and returned to the facility from each location. The samples were crushed to 1" x 0 in a Gundlach roll crusher and riffled to fifty pound increments. This fraction was then crushed to 3/8" by 0 in a jaw crusher and split again to a ten pound sample. The samples were then pulverized to -100 mesh U.S.S. in a Holmes Mill (Figure 3) and stored for later use.

Figure 3 - Holmes Pulverizing Mill

A representative sample was submitted for chemical analysis. The results are shown in Table 5. The table shows that the Pocahontas #3 refuse has the highest ash; this is due, however, to the very low amount of sulfur rather than low carbon. The refuse with the lowest carbon is the Illinois #6 sample. This is supported by the low BTU value.

The laboratory procedure used for making Light Weight Aggregate from coal refuse consisted of four major steps. First the coal was pulverized to -100 mesh U.S.S. and pressed into pellets, the pellets were then fired and finally crushed into an angular aggregate. The test variables selected for comparison were forming pressure and firing temperature. The pellets were formed using a manual hydraulic press and a one inch diameter cylindrical pellet die pictured in Figure 4.

Figure 4 - Pellet and Die

PROXIMATE ANALYSIS

Sample	Moisture	Ash	Sulfur	BTU	V.M.	F.C.	%C
Illinois # 6	2.8	75.3	6.38	1,750	12.0	9.1	10.84
Pocahontas # 3	.8	73.1	.33	2,255	8.1	13.1	18.33
Pittsburgh # 8	2.2	72.3	5.26	2,275	14.4	11.1	14.66

SULFUR BREAKDOWN

Sample	Total S	Sulfate S	Pyritic S	Inorganic S	Organic S
Illinois # 6	6.38	.18	5.61	5.79	.59
Pocahontas # 3	0.33	.02	0.31	0.33	.00
Pittsburgh # 8	5.26	.06	4.55	4.61	.65

TABLE 5

PROXIMATE ANALYSIS AND SULFUR BREAKDOWN OF COAL REFJS"
FOR L.W.A.

Three-fourths inch high pellets were formed at two different pressures, 4,000 and 8,000 PSI. The pellets were fired in a Pereny Electric Kiln on a Nichrome wire mesh. (Figures 5, 6)

Figure 5 - Pellets on Nichrome Mesh

Figure 6 - Electric Kiln

The pellets were fired at two different temperatures. The first group of pellets was heated to 1000°F and maintained at that temperature for eight hours. The second group of pellets was fired at 2000°F. Each firing test contained pellets formed at both high and low pressures. No shrinkage was observed; however, fracture lines could be noticed in some of the fired pellets. The color of the pellets changed from gray-black to tan-red upon firing, most likely due to the presence of iron in the pellet (Figure 7).

Figure 7- Comparison before and after firing

The pellets were tested for compressive strength to determine which refuse would form a stronger aggregate and to ascertain the most effective combination of forming pressure and firing temperature. Compressive strength tests were conducted on a Wiedemann Baldwin Universal Tester.

(Figure 8)

Figure 8 - Compressive Strength Tester

Table 6 displays the compressive strengths of the three samples under the various forming conditions. The table shows that pellets made with the Illinois #6 refuse have the highest compressive strength and that the combination of 8,000 PSI forming pressure and 2000°F firing temperature yielded the highest compressive strength. These optimal parameters were employed in subsequent bulk production of aggregate for concrete cylinder testing.

Firing Temperature	Forming Pressure	Samp. No.	Illinois # 6	Pocahontas #3	Pittsburgh #8
1000°F	4000 PSI	#1	425	75	165
		#2	415	80	230
		Ave	420	78	198
	8000 PSI	#1	450	85	280
		#2	565	80	315
		Ave	508	83	298
2000°F	4000 PSI	#1	1200	295	695
		#2	1735	210	705
		Ave	1468	253	700
	8000 PSI	#1	1695	375	1000
		#2	2520	370	990
		Ave	2108	373	995

Table 6 - Comparison of Compressive Strength (PSI) Tests Between Coal Refuse Pellets Formed Under Varying Pressures and Fired Under Varying Temperatures.

A supplemental analysis was performed to determine the mineralogical characterization of each sample. This data explains factors causing color change, cementation and the performance characteristics of the samples being tested. Figures 9, 10 and 11 are accompanied by the microscopists report

and represent pellets produced from Illinois #6, Pocahontas #3 and Pittsburgh #8 refuse respectively. Fired pellets submitted for analysis by Scanning Electron Microscopy were slabbed and polished in order to reveal mineral grain relationships. One to two mm thick slabs were cut vertically from the center of each pellet. The slabs were halved, and each half was molded in a 1 1/4 inch epoxy pellet. The pellets were polished and cut into 5 x 5 x 1 mm slabs for examination. Photographs and maps were made on the polished surfaces.

Figure 9 - Illinois #6 Fired Refuse Pellet

Analysis

Of the fired portion, only a rim 250 microns thick was well polished. This rim may have been caused by complete firing, or epoxy may have impregnated this portion of the pellet. This rim was photographed to show mineral relationships discussed below.

Photograph I11-1: Shows the side and top of the pellet at 20X. The rim is evident, as is the poorly polished interior and the pores in the interior. Features along the extreme edge of the photo are air bubbles in the epoxy.

Photograph I11-2: Shows the rim at 500X. Some polished grain surfaces are evident in the matrix, and are angular quartz. Analysis of this portion of the pellet shows Si dominant, with Al and Fe. Minor amounts of K, Ca, Mg, and Ti were noted with trace amounts of Mn, Cr, Ni, and Cu. This is the location map for photos I11-3, 4 and 5.

Photograph I11-3: Backscatter electron image. The brighter the particle, the greater its atomic number. Numerous bright particles are evident. These are composed of Fe alone, and through optical petrography were identified as Fe_2O_3 , though some magnetite may be present. The polished quartz grains and the matrix are the same shade of gray.

Photograph I11-4: Elemental map of Fe showing hematite grains made evident in the preceding photo.

Photograph I11-5: Elemental map of Si in which the quartz grains show as concentrations of dots, and the matrix shows significant Si content. Analysis of the matrix shows a typical clay composition of Si, Al, and minor K. Details of the matrix surface shown in photos I11-2 & 3 do not indicate that significant melting of the clays occurred during firing.

Photograph I11-6: Shows a growth of calcium sulfate crystals within the pellet. These crystals probably formed during polishing from Ca and sulfate already in the pellet. Grains of Ca sulfate present during firing are also evident.

Summary of Analysis of Illinois #6 Fired Refuse Pellet

The cementing matrix of this pellet is compressed or partially fired clay of Si, Al, and K composition, and is most likely illite. The material shows no evidence of having melted to a glass. A number of different mineral grains are present within the clay matrix: (a) angular quartz showing no effects of the firing; (b) hematite (Fe_2O_3) grains of varied size resulting from the oxidation of pyrite; (c) calcium sulfate; (d) calcite, apparently unaffected by the firing; and other minerals typically contained in clay such as rutile (TiO_2).

Figure 10 - Pocahontas #3 Fired Refuse Pellet

Photograph Poca 1: Shows the side and top of the pellet at 20X. The well polished rim is evident, as is the interior of the pellet. The rim is cemented with epoxy.

Photograph Poca 2: Shows the rim at 500X. Three surfaces are evident in this photo: (a) polished mineral grain surfaces; (b) a light matrix; and (c) a dark matrix. The minerals with well polished surfaces are quartz, calcite, rutile, and hematite. The light matrix was composed of Si, Al, and K, and was clay. The dark matrix material is the cementing epoxy.

Photograph Poca 3: Backscatter electron image of the area shown in photo Poca 2. This photo accentuates the three materials discussed above. Polished surfaces of calcite, hematite, and rutile show brightest in this image, and the matrix appears medium gray. The epoxy is black in this image.

Photograph Poca 4: Elemental map of Si in which quartz grains show as concentrations of dots, and the matrix shows a significant Si content. Epoxy, calcite, and hematite are present in areas of this image that show few dots.

Photograph Poca 5: Another area of the rim of the pellet photographed at 200X. In this image a number of mineral grains can be seen with the tenuous clay matrix mixed with epoxy between.

Photograph Poca 6: Backscatter electron image of the area of photo Poca 5 showing the mineral grains, clay matrix, and epoxy more clearly.

Summary of Analysis of Pocahontas #3 Fired Refuse Pellet

The cementing matrix of this pellet was poorly compacted clay of Si, Al, and K composition. The matrix shows no evidence of melting, and appears to have lost volume during firing. The mineral grains within the clay matrix were predominantly quartz, but hematite, calcite, and rutile were noted.

Figure 11 - Pittsburgh #8 Fired Refuse Pellet

Analysis

Unlike the other two pellets the entire fired portion of this pellet polished well. Mineral relationships in the fired portion are discussed below.

Photograph Pitt 1: Shows one corner of the slabbed pellet at 20X. Epoxy is visible in the upper corner and left side of the photo. Numerous <200 micron pores are visible.

Photograph Pitt 2: Shows the edge of the pellet at 500X. Few grains are evident, but the irregular pores are visible.

Photograph Pitt 3: Backscatter electron image of the area in photo Pitt 2. The brightest mineral grains are hematite, and the dark matrix material at the top center of the image is epoxy. The intermediate gray material is matrix clay minerals (illite and kaolinite).

Photograph Pitt 4: Elemental map of Si showing few quartz grains. Dark areas are hematite, apatite, pores, and epoxy.

Photograph Pitt 5: Elemental map of Fe showing hematite grains resulting from the oxidation of pyrite.

Photograph Pitt 6: Elemental map of Ca showing the presence of an apatite ($\text{Ca}_5(\text{F}, \text{Cl}, \text{OH})\text{PO}_4)_3$ grain. Indentations in this grain are made by kaolinite which indicates that this apatite-kaolinite grain is an unaltered refuse particle.

Photograph Pitt 7: A backscatter electron image taken at 100X at the edge of the pellet showing pores in the pellet partially or completely filled by epoxy (light gray material). Examination of the complete slab of this pellet revealed that epoxy permeated the entire pellet indicating a system of fractures or pores throughout the pellet.

Summary of Analysis of Pittsburgh #8 Fired Refuse Pellet

The cementing matrix of clays in this pellet bound the mineral grains into a sturdy mass. However, the extensive network of pores, and or fractures, weakened this pellet severely. This permeability may have originated through shrinkage of the matrix clays as firing progressed. The mineral grains within the matrix were varied. Quartz was relatively rare, but hematite was common.

Comparisons of Pellet Analyses

All three pellets consisted of three phases: (1) mineral grains from

the refuse which acted as aggregate in the pellet, (2) cementing clay matrix which held and bound the aggregate mineral grains, and (3) pores, passages and fractures.

Two types of mineral grains were noted: (1) unaltered refuse material such as quartz, calcite, rutile, apatite, kaolinite, and some illite particles, (2) altered refuse material such as hematite and calcium sulfate.

The clay minerals present were examined after firing of the products to determine the effects of these minerals on the bonding mechanisms. In the most poorly bound pellet (Pocahontas #3) there was too little binding material (clays) to hold the mineral grains together, and this material appeared to shrink during firing leaving irregular gaps between the mineral grains as well as larger pores. In the Pittsburgh #8 refuse pellet the clay matrix material appeared to melt and cemented the mineral grains together, however, in the process the melting produced a network of pores and permeable passages. These openings weakened the pellet significantly. In the Carlinville refuse the clays appear not to have significantly melted, but neither did they shrink or develop pores. The pellet did have large openings, but very little permeability. Polishing characteristics and SEM examination showed that in this pellet the mineral grains are well bound, and the structure is not weakened by pores or fractures.

With the SEM analysis confirming the compressive strength test results, the Illinois #6 refuse was used to produce a quantity of aggregate sufficient for bulk testing of concrete mixes comparing concrete blocks made with Illinois #6 LWA to concrete blocks using limestone as an aggregate. 100 pellets were pressed from the refuse at a forming pressure of 8,000 PSI. The pellets were then fired for 8 hours at 200°F. After firing, the pellets were allowed to cool and then crushed in a three inch jaw crusher (Figure 12) to a 3/8 inch maximum size medium coarse aggregate. (Figure 13).

Figure 12 - 3" Jaw Crusher

Figure 13 - Illinois #6 Lightweight Aggregate

A small sample of one inch limestone was obtained and crushed to $3/8$ inch in the same fashion. A sieve analysis appearing in Table 7 shows the grading of the aggregates. The grading of the aggregates was similar both falling within the standards described in ASTM C-330 "Specifications of Light Weight Aggregate in Structural Concrete". The fines in the aggregate (-12 mesh) were removed and discarded from both samples leaving the remaining aggregate sized to $3/8$ inch by 12 mesh U.S.S.

A rectangular steel mold was used to form a 2 inch x 2 inch x 4 inch concrete test block. (Figure 14) Type 1 Portland cement was used with highway grade silica sand. The cement, sand and aggregate was mixed at a 1 to 1 to 2 ratio by volume. Each batch was prepared using the same volume of water. Each concrete mix was poured into the mold and placed on a vibrating table for 5 minutes to remove air and consolidate the mix.

Sieve Size	Illinois # 6	Limestone
5/16"	16%	16%
1/4"	30%	30%
7 m	40%	35%
12 m	6%	8%
-12 m	8%	11%

TABLE 7

COMPARISON OF AGGREGATE GRADING BETWEEN LWA AND LIMESTONE. %
RETAINED ON SCREEN

Figure 14 - Mold and Test Block

The blocks were allowed to set for 4 hours before they were stripped from the mold and placed in a humidity chamber to cure for 6 days. After curing, the blocks were weighed and tested for compressive strength. The compressive strength tests were conducted in the same manner as before using the same tester. The results shown in Table 8 indicate that the Illinois #6 refuse LWA is not only 15% lighter but also yielded a 15% higher compressive strength.

Conclusion

Preliminary results of the tests indicated that of the refuse tested, the Illinois #6 refuse was the best suited for light weight aggregate application. The Illinois #6 refuse contained the lowest carbon content of the tested refuse, a factor important in the firing process. Carbon burning during firing procedures increases pore space, resulting in a weakening of the structure, as was evident in the Pittsburgh #8 refuse. A disadvantage to the Illinois #6 refuse is the level of sulfur present. A high percentage of Pyritic sulfur could under certain conditions result in staining. The performance of the Illinois #6 refuse pellets when crushed was equal to that of limestone. When the fired refuse pellets were crushed under identical conditions as the limestone, they behaved in the same manner and yielded a well graded, properly sized angular aggregate. In the final performance test, LWA and limestone were used to form concrete test blocks to compare compressive strengths. The procedure for forming the test blocks was held constant. The LWA blocks were on the average 15% lighter and when tested for compressive strength, outperformed the limestone blocks by an average of 15%. Both the LWA and the limestone blocks demonstrated proper angular fracture as seen in Figure 15.

Figure 15 - Angular Fracture of Concrete Test Blocks

Concrete	Test #	Load in PSI	Weight
LWA	1	24,900	591
	2	28,100	587
	Ave.	26,500	589
Limestone	1	20,200	678
	2	24,950	681
	Ave.	22,575	680

TABLE 8

COMPRESSIVE STRENGTHS AND WEIGHTS OF TEST BLOCKS

SAND-LIME BRICK

The purpose of the research program was to examine the effects of coal mineral materials on coal waste by-product utilization and to investigate new and improved methods for the utilization of waste by-products from the combustion of coal. The research described deals specifically with the utilization of the "spent" limestone bed material, also called bottom ash, from the experimental fluidized combustion unit located at Rivesville in northern West Virginia. The goal of the work was to determine the structural properties of sand-lime brick produced from the spent-bed material.

Physical, Chemical, and Mineralogical Analyses

The spent bed material was submitted for size analyses by screening on 7/16 inch, Nos. 3 1/2, 7, 14, 25, 45, 80, 170, and 325 U.S.S. testing sieves. The results of the screen analysis are presented in Figure 16. As can be seen from the histogram, most of the spent-bed material (87.06%) was finer than No. 3 1/2 and coarser than No. 25 U. S. standard sieve sizes. The median particle size of the material was approximately No. 10 U.S.S. (1.7 mm).

A sample of the spent bed material was also submitted for chemical analysis to determine the amounts of Si, Al, Fe, Ti, Ca, and Mg (as oxides) and Na, K, S, and C on an elemental basis. The results of the chemical analysis are presented in Table 9.

Mineralogical analysis of the spent-bed material was accomplished by X-ray powder diffraction techniques. The data revealed that the material was, to a large extent, amorphous. A positive identification of hematite was made and, in addition, the presence of calcium-aluminum minerals was also indicated.

Table 9

Chemical Analysis of Fluidized Bed Process
Spent-Bed Material

<u>SiO₂</u>	<u>Al₂O₃</u>	<u>Fe₂O₃</u>	<u>TiO₂</u>	<u>CaO</u>	<u>MgO</u>	<u>Na</u>	<u>K</u>	<u>S</u>	<u>LOI</u>
24.10	8.31	2.12	0.34	37.20	1.52	0.19	0.65	8.01	3.7

Utilization Tests

The use of spent-bed material as a component of sand-lime brick was investigated in a series of laboratory-scale tests. The initial tests were both empirical and systematic in order to optimize parameters prior to standardized procedures. Photographs of the test procedures are shown in Figure 17.

The parameters investigated included % bottom ash material, % aggregate (silica sand), % water, bulk density of the final product, and compressive strength of the final products. Two additional variables were also examined during this preliminary research: effects of the relative coarseness of the bottom ash on the final product's compressive strength, and the effects of leaching the bottom-ash material on the final product's compressive strength.

The preliminary bench-scale work involved mixing variable proportions of bottom ash, aggregate, and water, forming the wet mix in a rectangular mold, and allowing the material to moist-cure for a 7-day period. The solid rectangular product measuring 102 mm x 51 mm x 40 mm was then weighed to calculate bulk density and finally subjected to controlled breakage to determine compressive strength. In Table 10, the parameters and results of each preliminary test are delineated.

As can be seen clearly in the results of the compressive strength tests listed in the table, the sample block made from pulverized, non-leached bottom ash was superior to all others. Based on these preliminary results, additional work was performed utilizing mixture #6 to examine the effects of prolonged

Table 10

Results of Preliminary Tests
Utilization of Fluidized Bed Bottom Ash in Unfired Structural Materials

Test Block #	Composition ¹			Bottom Ash Treatment			Results	
	% Bottom Ash	% Aggregate	% Water	Coarse ²	Fine ³	Leached ⁴	Bulk Density (grams/cm ³)	Compressive Strength (pounds/in ²)
1	100.0	0	20.0	yes	no	yes	1.79	352
2	100.0	0	20.0	yes	no	yes	1.71	228
3	100.0	0	25.0	yes	no	yes ⁵	1.60	366 $\frac{1}{2}$
4	75.0	25.0	25.0	no	yes	yes ⁵	1.69	796
5	50.0	50.0 ⁶	25.0	yes	yes	yes	1.76	454
6	75.0	25.0	25.0	no	yes	no	1.75	1501

1. Weight basis
2. Coarse Ground to - 14 mesh
3. Pulverized to - 60 mesh
4. Bottom ash leached in H₂O at 100°
5. Sample oven-dried at 80°C for 24 hrs.
6. Aggregate Composition = 50% Silica + 50% Coarse Bottom ash

curing.

Table 11

Results of Curing Tests
Utilization of Spent-Bed Material for Structural Products

Test No.	Curing Time	Bulk Density ¹	Compressive Strength ²
1	7 Days	114	630
2	14 Days	114	1564
3	21 Days	114	2392

¹Pounds Per Cubic Foot

²Pounds Per Square Inch

In Table 11, the results of the tests to determine the effects of curing time are reported. As can be seen by examining the data, the compressive strength of the material increased progressively as the length of the curing cycle was increased. The range of compressive strengths observed in the tests, if compared to conventional concrete brick, would be classified as low to moderate. It, therefore, appears that the spent-bed material is a potential binder for structural products, provided that either ample moist-curing time is allotted, or that steam curing is incorporated in the manufacturing process.

Summary

Although the spent-bed material from a fluidized-bed combustion unit in northern West Virginia did not yield sufficiently suitable test results to be considered as a possible aggregate substitute, further laboratory-scale tests did indicate that the spent-bed material had substantial cementitious properties and may have utilization potential as a binder in the manufacture of structural products. It is recommended that further testing be conducted to firmly establish the parameters for using spent-bed material as a binder in these structural products.

Figure 16

Histogram of the Size Distribution
of the Fluidized-Bed Combustion
Unit Spent-Bed Material

a

b

c

d

Figure 17

- a. Mixing pulverized fluidized-bed bottom ash with sand and water.
- b. Wet mixture in mold.
- c. Removing block from mold.
- d. Block placed in moist-curing chamber.

Insulating Wool

Increased interest in energy conservation in the United States has led to an increased demand for insulating material. One common insulating material in use today is wool fiber produced from blast-furnace slag or siliceous limestone. The Coal Research Bureau has been involved in research in past years designed to evaluate the use of coal ash from conventional power generation stations as the source material for insulating wool production. These studies have indicated that many coal ashes can be successfully converted into insulating wool fiber similar in appearance to commercial 'Rockwool'.¹ Based on these studies, limestone fluidized bed flyash (LFBFA) was examined to determine its suitability as a source material for insulating wool fiber production.

Physical property testing was performed on the LFBFA wool, and results compared with physical properties of wool fiber produced from pulverized flyash, commercial glass fiber wool ('Fiberglas')² and commercial mineral fiber wool ('Rockwool'). The results of these preliminary tests indicated LFBFA to be comparable to commercial products.

Production of Fiber

Under standard operating conditions, flyash from limestone fluidized bed combustion will contain little carbon. Since the sample used in this

¹ Rockwool is a product of Rockwool Industries of Denver, Colorado.

² Fiberglas is a trade name of Owens-Corning of Toledo, Ohio.

LFBFA Before and After Oxidation

Figure 18

study was obtained from a test facility where all conditions had not been optimized, it was necessary to beneficiate the sample prior to fiber production evaluation.

The limestone fluidized bed flyash collected for this study was analyzed and found to have a carbon content of 32 percent. Coal ashes with such high carbon contents are impossible to melt in the required reducing atmosphere at any reasonable temperature. A series of tests were conducted to determine a reasonable method of oxidizing the carbon in the flyash using available laboratory and pilot scale equipment. The details of this series of tests are not presented here because in commercial operation only low carbon ashes would be considered for use.

Laboratory Testing Furnace

Figure 19

It was determined that placing the ash in a cast iron container and exposing it to a temperature of 800°C for 24 hours under oxidizing conditions would produce an ash with a carbon content of 0.2 percent. This ash was used in subsequent testing. Figure 18 is a comparison of the flyash both before and after oxidation. The high carbon content of the flyash before oxidation is obvious from the much darker color of the untreated ash.

Mineral wool is made commercially by heating the source material until it becomes molten and sufficiently fluid for further processing. The choice of melting equipment used is determined in part by the size and nature of the raw materials and by the type of energy source or fuel to be used. The most common furnace used in commercial mineral wool production is the water-jacketed cupola type. Some manufacturers have used oil or gas fired reverberatory furnaces or submerged arc resistance furnaces. In the current testing program, a Detroit electric 10 lb.

capacity, 17 KVA, carbon arc rocking furnace (Figure 19) capable of being rotated on its horizontal axis was used. This type of furnace was utilized because the molten flyash can be poured directly from the hot furnace chamber as is done in industrial practices. During the heating of the flyash, the furnace was gently rocked to produce a homogenous melt by mixing the various components. Rocking also increased the efficiency of the furnace as the heat energy was more evenly distributed among the particles in the chamber. Temperature measurements were made using an optical pyrometer.

When the molten flyash became sufficiently fluid, it was poured in a narrow stream (1/4 to 1/2-inch in diameter) into the path of a "blowing-gas" stream. The gas was directed through a nozzle system located at approximately a right angle to the stream. In commercial practice superheated steam is often used as the blowing gas in order to extend the time period during which the molten particles cool after pouring. This practice can result in greater fiber production as compared to the amount formed when bottled compressed air is used. In the testing program, however, bottled air was used as there are fewer safety hazards for the operator who must work in close proximity to the blowing apparatus. In industrial practice, no workers are near the blowing apparatus.

The nozzle used in these tests was a flat-spray blow-off nozzle with a slot width of 0.023 inches. At 95 psig it delivered approximately 10 standard cubic feet per minute of compressed air. It was noted that this nozzle design is not as efficient for the production of mineral wool fibers as many currently used in the industry, but nozzle design efficiency was not considered within the scope of the testing program and nozzle choice was restricted to commercial off-the-shelf designs. The nozzle was hand held to maintain proper spatial alignment (air-jet perpendicular or at a slight upward acute angle) to the slag stream with the nozzle orifice

Blowing Wool

Figure 20

approximately 1/4 inch from the stream as shown in Figure 20.

The compressed air or blowing-gas has a shearing action upon the slag stream, breaking it into individual drops and propelling the drops into a collection area. As the drops of molten slag are propelled through the air, friction causes them to develop long "tails". These "tails" instantaneously solidify into insulating fibers and any remnants of the original drops are called "shot". If the pouring and blowing conditions have been properly controlled, almost all of each individual drop will be consumed in the formation of the fibers. If conditions are not optimum, i.e., air pressure too low or melt viscosity too high, shorter, coarser and more brittle fibers will be formed. In addition to these less desirable fibers, large shot particles will occur. When the melt becomes

Fiber Elongation

Figure 21

thin, fiber elongation does not occur and short interlocking fibers are produced resembling snow. Figure 21 is a photograph taken during a blowing operation using molten limestone fluidized bed flyash. In the left portion of the picture wool fibers may be seen floating to the floor.

Figure 22 is a photograph of a sample of the wool made from limestone fluidized bed flyash (LFBFA). This wool was produced at a temperature of 1375°C without the addition of any fluxing agents. In appearance, the wool is light gray in color, fine textured, with a moderate shot content.

Photomicrographic Analysis

Figure 23 contains ten scanning electron photomicrographs of five samples of insulating wool. Each sample is pictured at two magnifications as indicated by the micron scale on each. As can be seen from the photo-

LFBFA Wool

Figure 22

graphs, there is very little difference in structure apart from a moderate percentage of attached shot apparent in the laboratory produced samples. It is also apparent that the blown wool samples are all smaller in average particle diameter than the glass fiber sample which has an estimated 20 micron average particle diameter. Furthermore, it can be seen that the laboratory samples of blown wool, i.e., E. PFA, W. PFA, and LFBFA, are slightly finer than commercial 'Rockwool' (an estimated mean particle diameter of 10 versus 15 microns). Most significant is the similarity in structure in all samples pictured indicating that a similarity in physical insulating properties can be expected.

FIBERGLAS

FIBERGLAS

ROCKWOOL

Photomicrographs

Figure 23

LFBFA

LFBFA

E. PFA

E. PFA

W. PFA

W. PFA

Photomicrographs

Figure 23 (Continued)

Figure 24

Thermal Conductivity Test

The most important property of insulating fiber is its ability to prevent heat transfer. To effectively insulate, a material must prevent heat transfer by conduction and convection. In the case of insulating wool this is accomplished by trapping air (which has an extremely low thermal conductivity) in such a way that convection losses are minimized. If air is free to move, heat will be efficiently transferred through convection. In an effort to compare the relative insulating efficiencies of commercial 'Fiberglas', 'Rockwool' and laboratory blown insulating wool, a special test was devised. A thermal conductivity apparatus was modified according

Insulation Test

Figure 25

to the diagram, Figure 24.

For the test, a 4" x 4" x 2 1/2" piece was cut from a standard 'Fiberglas' batt and 'Rockwool' batt. For in-house produced mineral wools, i.e., those produced from limestone fluidized bed flyash (LFBFA) and pulverized flyashes from eastern and western United States sources, a 4" x 4" x 2 1/2" piece was cut with scissors from a bulk gathered sample. A 2 1/2" height was used because it best represented the actual thickness of the commercial batts of 'Rockwool' and 'Fiberglas'. The test samples were placed within the conductivity apparatus as indicated in Figure 25. A temperature of 500°F was maintained in the lower heater and the temperature of a metal plate above the heater in contact with the bottom of the sample was read and compared with the temperature of

Table 12
Insulation Properties

<u>2 1/2" Thickness Insulating Wool Type</u>	<u>Top Temperature °F</u>	<u>Bottom Plate Temperature °F</u>	<u>Temperature Difference °F</u>	<u>°F Improvement Over Air Gap</u>	<u>% Improvement Over Air Gap</u>
'Fiberglas'	198	512	314	37	13
'Rockwool'	196	512	316	39	14
LFBFA	196	512	316	39	14
W. PFA	206	512	306	29	10
Air Gap Control	235	512	277	0	0

the probe on top of the sample. Each test was run for a time sufficient to allow stabilization of temperature to occur, usually about 5 hours. As a control, the top probe was suspended in the chamber 2 1/2" above the heater plate to indicate the comparative insulating value of air. The results given in Table 12 show a similarity in insulating ability among samples tested. Convection losses are indicated by the smaller temperature difference for the air gap compared to the wool samples.

The results indicate no significant difference in insulating properties between commercial 'Fiberglas', 'Rockwool' and LFBFA wool. A lower insulation value (reported as a 29°F improvement over air gap) was recorded for wool produced from western pulverized flyash. Duplication of the test with different gathered samples of WPFA wool might be expected to produce results more consistent with the other wool samples. Since convection functions as the major heat transfer mechanism in this test, it is possible that a gap, hole, or space between discrete pieces of "gathered" wool in the test sample provided a convection path for air resulting in reduced insulating efficiency.

Corrosion Test

Preliminary testing was performed to determine the effects of close contact of mineral wool insulating fibers with metal surfaces and to indicate if any special corrosion problems exist with LFBFA. Certain applications may require contact with steel, zinc (as in galvanized pipe or electrical conduit), copper, lead, iron, aluminum and other metals. These metals are normally found exposed in plumbing and wiring systems within walls where insulation is likely to be used. Steel was chosen to represent the effects of wool contact with metal surfaces. For the test

Corrosion Samples

Figure 26

program, six one-inch wide strips were cut from a 22 gauge sheet of mild steel. The surface of each strip was treated by removing all grease and oil with trichlorethylene, pickling in dilute hydrochloric acid and grinding one side with 60 grit paper in a belt sander. These steps were performed to insure uniformity of initial corrosion (by pickling), and to present an identical surface (by grinding) for subsequent corrosion. Each strip was numbered as shown in Figure 26 and a piece of wool 4" x 3" x 2" thick was prepared for five of the strips. The sixth strip was left uncovered as a control. Table 13 indicates the source of each wool material and the weight of the test strips before and after 49 days exposure to 90-100 percent relative humidity at 70°F.

Corrosion Apparatus

Figure 27

Two stainless steel racks were used to sandwich the test specimens as indicated in Figure 27. After 49 days of exposure in the humidity chamber, the strips were removed from the chamber, the wool peeled from the corroded surfaces and the surfaces rinsed clean of any clinging fibers or loose corrosion with tap water and a soft brush. The strips were then dried, weighed and percent weight loss determined. The data presented in Table 13 indicates that aside from the control strip #6, strip #1 covered with LFBFA wool lost the least weight, 1.29 percent due to corrosion, while strip #5, covered with wool produced from Eastern pulverized flyash lost the most weight, 3.93 percent. The percent weight loss is seen to differ only slightly among the remaining strips, 2, 3 and 4. It is important to note that such high humidity is not likely to be encountered in normal use except in cases where the material is improperly placed or misapplied.

Table 13

Corrosion Test Weight Loss

<u>Source Material</u>	<u>#</u>	<u>Initial Weight</u>	<u>Final Weight</u>	<u>% Weight Loss</u>
LFBFA	1	23.3 gr.	23.0 gr.	1.29
'Rockwool'	2	22.9	22.1	3.49
'Fiberglas'	3	24.0	23.1	3.75
Western PFA	4	22.0	21.2	3.64
Eastern PFA	5	22.9	22.0	3.93
Control	6	22.2	22.0	0.90

Corroded Strips

Figure 28

As the photograph of the corroded strips, Figure 28, shows with the exception of strip number 6, the degree of corrosion was difficult to visually evaluate. In an attempt to objectively evaluate the degree of corrosion, the strips were shown to nine laboratory personnel who were asked to rank the strips from least corroded, 1, to most corroded, 6. The observers were not informed as to which strip corresponded to which wool sample. Results appear in Table 14.

Table 14

Visual Corrosion Evaluation

	<u>Least Corroded</u>		3	4	<u>Most Corroded</u>	
	1	2			5	6
Observer 1	1	3	2	5	6	4
2	6	3	4	1	5	2
3	6	1	3	5	4	2
4	6	1	3	2	5	4
5	6	1	3	2	5	4
6	6	4	3	1	2	5
7	6	1	3	4	5	2
8	6	1	3	5	2	4
9	6	4	1	2	3	5
Weight % Loss	<u>6</u>	<u>1</u>	<u>2</u>	<u>4</u>	<u>3</u>	<u>5</u>
Observer Average	<u>6</u>	<u>1</u>	<u>3</u>	<u>4</u>	<u>2</u>	<u>5</u>

Most observers identified the least corroded strip as no. 6, the uncovered control, and the most corroded strip as no. 5, the strip covered with wool produced from eastern pulverized flyash. The relatively small difference in weight loss recorded for strips 2, 3 and 4 in Table 13 relates to the difficulty in ranking the degree of corrosion for these samples as evidenced in Table 14. An observer average ranking shown in Table 14 calculated from the individual rankings correlates reasonably well with the weight loss data.

It is concluded from the data that only strip no. 1 covered with wool produced from limestone fluidized bed flyash showed significantly less corrosion than the other covered strips in the experiment including commercial 'Fiberglas' and 'Rockwool' and that no special corrosion problems are to be expected from insulating wools produced from pulverized flyash or limestone fluidized bed flyash.

Acid-Base Leaching

It was desired to determine if contacting insulating wool with water would produce undesirable acidic or basic solutions. A test was designed in which 10 gram samples of mineral wool and commercial wool were immersed in breakers containing 500 mls of distilled water for one week and pH measurements made at increasing time intervals as shown in Table 15.

As can be seen from the table, wool produced from limestone fluidized bed flyash (LFBFA) and commercial 'Rockwool' both caused the water to become slightly acidic initially while the water containing 'Fiberglas' wool was mildly basic. After a week in the water the pH had stabilized to slightly alkaline for all three samples. These results suggest no significant difference in aqueous acid-base reaction among the wools tested.

Table 15

pH Leaching Study

<u>Time Interval</u>	<u>LFBFA Wool</u>	<u>'Fiberglas' Wool</u>	<u>'Rockwool'</u>
1 Minute	6.5	8.8	6.4
2 Minutes	6.7	8.8	6.5
3 Minutes	7.0	8.9	6.7
4 Minutes	7.1	8.9	6.7
5 Minutes	7.2	8.9	6.7
2 Hours	9.0	8.8	7.3
7 Days	7.7	7.6	7.8

Flame Test

A flame test was performed in which three different samples of insulating wool fibers were held 6" above a Fisher laboratory burner for approximately one minute at a temperature of 1025°C. Exposed edges of the 'Fiberglas' sample melted more quickly than either 'Rockwool' or LFBFA wool which are produced at higher temperatures and benefit from a higher melting point. All three wools melted to some extent, however, none supported combustion.

Shot Content

The shot content of blown insulating wool is defined, in this report, to be the non-fibrous particles retained on a series of sieves after substantially all the fibers in a sample of insulating wool have been broken up and collected in a retaining pan. Samples weighing fifteen grams were collected from each of the insulating wools to be tested. Each sample

was screened through a 10, 30, and 50 mesh USS sieve and the fines collected in a bottom pan. The fibrous nature of the material initially required a light pressing of the wool sample against the screen surface of the top sieve in the nest, using the flat surface of a large rubber stopper. To further aid in the degradation of the insulating wool fibers during screening on a mechanical shaker, a no. 13 size rubber stopper was placed on each sieve. The samples were then mechanically screened until substantially all the fibers had been broken up and collected in the bottom pan (approximately 30 minutes). The particles retained on each screen were then collected and weighed. The shot content of each wool tested was calculated by summing the weights of all particles retained on the screens and then dividing by the original weight of the sample. This value was then multiplied by 100 to obtain a percentage. The percentage of shot for each insulating wool tested, along with the weight of the particles retained on each screen fraction appears in Table 16.

Table 16

Shot Content and Weights of Particles
on Each Sieve (Gr)

Wool Type	-10M x 30M	-30M x 50M	+50 Mesh	Shot Content %
'Rockwool'	0.2	1.0	1.2	16.0
Eastern PFA	0.6	2.1	3.2	39.3
Western PFA	0.4	1.7	2.6	31.3
LFBFA	0.7	2.1	3.6	42.7

As can be seen from the table, the shot content of insulating wool produced in the laboratory is significantly higher than the commercial

sample of 'Rockwool'. The higher shot content of the laboratory produced insulating wool is due largely to limitations in procedure, specifically the difficulty in maintaining constant stream temperature during the blowing operation and the use of 'cold' bottled compressed air to blow the wool. Under commercial conditions it should be possible to reduce the percentage of shot in blown insulating wools to values similar to the shot content of comparable commercial products.

Synopsis of Results

Blown insulating wool produced from limestone fluidized bed flyash and pulverized flyashes from the Eastern and Western United States coals are nearly identical in physical properties to commercial 'Rockwool'. Shot content of laboratory blown wool samples was higher than the commercially blown product, but insulating values were found to be nearly identical.

Drawn glass fiber insulation ('Fiberglas'), differs most obviously from blown wool fiber in the absence of shot due to the different method of manufacture. Also, 'Fiberglas' produced for home insulation is lower in bulk density than blown wool fiber through the insulating properties of each are comparable.

The results of these tests indicate that LFBFA has potential as a substitute for both 'Rockwool' & 'Fiberglas'. In order to more completely evaluate LFBFA wool as an insulating material, a more detailed study is required especially relating to costs of manufacture & optimization of process variables and an indepth study of the physical & chemical properties of the material.

Fired Structural Products

Samples of spent limestone bed ash and flyash from the carbon burn-off cell were obtained from the fluidized bed combustor facility at Rivesville, West Virginia.

The chemical analysis of the spent limestone bed ash and flyash from the carbon burn-off cell are presented in Table 17.

The median particle size of the spent limestone bed ash is around 10 mesh (1.7 MM) and the median particle size of the flyash from the carbon burn-off cell is approximately 200 mesh (0.08 MM).

In order to determine the suitability of the spent limestone bed ash as an aggregate in fired structural materials, test specimens in the form of a standard building brick were chosen for this study. The composition and physical properties of the flyash brick are presented in Tables 18 and 19. Control flyash brick were produced using compositions which had been determined previously during pilot scale operations conducted by the Coal Research Bureau. The ingredients were mixed in a mix-muller, formed in a hydraulic toggle press, dried in a humidity drier, and fired in a gas-fired shuttle kiln. Firing conditions were carefully monitored both by thermocouples and also pyrometric cones which indicate the combined effect of temperature and time sometimes called "heat work."

All of the flyash brick were tested in accordance with American Society for Testing and Materials designation C67 "Methods of Sampling and Testing Brick" and evaluated in accordance with ASTM designations C62 "Specifications for Building Brick" and C216 "Specifications for Facing Brick."

Two different flyashes were used in this study. These flyashes were selected as being representative of different major coal seams and are designated as Northern West Virginia flyash and Southern West Virginia flyash.

The spent limestone bed ash was crushed and screened through a 20 mesh sieve. This is the same sieve that the standard bottom slag was screened through. The minus 20 mesh bed ash was utilized in this study.

The composition and properties of the flyash brick are presented in Tables 18 and 19.

The standard bottom slag was replaced with bed ash at levels of 17.38, 27.38, and 37.38 percent when using the Northern West Virginia flyash and at levels of 11.24, 21.24, and 31.24 percent when using the Southern West Virginia flyash.

Upon completion of the drying cycle the flyash brick containing bed ash exhibited cracks and fissures. It is thought that the cracks and fissures in the flyash brick were caused by the free lime in the bed ash hydrating and expanding after being wetted during the mixing cycle.

Sodium hydroxide which is frequently beneficial in the flyash brick process was tried but did not reduce or eliminate the cracks and fissures.

A batch was wetted and left standing for ten (10) minutes in order to allow the free lime in the bed ash to hydrate and expand before being pressed but this did not reduce or eliminate the cracks and fissures.

The standard bottom slag was used with an equal percentage of bed ash in order to minimize the effects of the bed ash; however, cracks and fissures were still observed upon completion of the drying cycle.

From Table 17 it can be noted that the flyash has a loss on ignition (LOI) of 36.7 percent. Previous research, performed by the Coal Research

Bureau under Contract Number 14-01-0001-488 to the Office of Coal Research, has indicated that a flyash having a loss on ignition (LOI) in excess of 10-12 percent would require some special modifications, such as calcination, to eliminate the weak porous structure which commonly results from bulk weight loss after carbon burn-off in high carbon flyashes. Since calcining the flyash would be energy-intensive as well as uneconomical, research on utilizing flyash in fired structural materials was not investigated.

Conclusion

The utilization potential of spent limestone bed ash as an aggregate in fired structural products has been shown to be unfeasible because of cracks and fissures in the structural products. The cracks and fissures are caused by free lime in the bed ash hydrating and expanding after being wetted during the mixing cycle.

Table 17

CHEMICAL ANALYSES OF FLUIDIZED BED PROCESS BY-PRODUCTS

	<u>Bed Ash</u>	<u>Flyash</u>
SiO_2	24.10	30.10
Al_2O_3	8.31	13.40
Fe_2O_3	2.12	9.07
TiO_2	0.34	0.46
CaO	37.20	38.70
MgO	1.52	1.24
Na	0.19	0.23
K	0.65	0.90
S	8.01	3.85
LOI	3.7	36.7

Table 18

COMPOSITION AND PROPERTIES OF NORTHERN WEST VIRGINIA FLYASH BRICK

BATCH #	1	2	3	4	5	6	7	8	9
Northern West Virginia Flyash	69.62	79.62	69.62	59.62	79.62	69.62	59.62	69.62	69.62
Bottom Slag - 20 Mesh	27.38	-	-	-	-	-	-	-	13.69
Bed Ash - 20 Mesh	-	17.38	27.38	37.38	17.38	27.38	37.38	27.38	13.69
Silicate	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
Silicate Grade	47	47	47	47	47	47	47	47	47
Water	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
Sodium Hydroxide	-	-	-	-	1.0	1.0	1.0	-	-
Firing Rate, °F/Hr.	119	119	119	119	119	119	119	119	119
Final Firing Temp., °F.	2190	2190	2190	2190	2190	2190	2190	2190	2190
Cone	6	6	6	6	6	6	6	6	6
% Abs. 24 Hr. Soak	8.56	17.26	23.10	27.00	15.98	21.42	26.70	23.24	15.42
% Abs. 5 Hr. Boil	12.41	21.99	28.22	32.58	21.29	26.76	31.76	28.76	20.53
Saturation Coefficient	0.68	0.78	0.81	0.82	0.74	0.79	0.83	0.80	0.74
Apparent Porosity, %	23.55	36.15	42.60	46.55	35.35	41.24	45.90	43.04	34.41
Bulk Density, Lbs./Cu. Ft.	118.3	102.6	94.2	89.2	103.6	96.1	90.1	93.4	104.5
% Shrinkage	4.9	5.3	2.8	0.1	5.7	2.6	0.2	2.5	4.3
Unfired Compressive Strength, P.S.I.	984	82	25	63	60	49	99	89	49
Fired Compressive Strength, P.S.I.	7179	3526	1382	733	2801	1296	702	1204	2848
Suction, Gms. H ₂ O/30 Sq. In./Min.	104	172	257	334	156	242	303	248	164

Note: Batch 8 had a ten (10) minute interval between wetting and pressing.

Table 19

COMPOSITION AND PROPERTIES OF SOUTHERN WEST VIRGINIA FLYASH BRICK

BATCH #	1	2	3	4	5	6	7	8	9
Southern West Virginia Flyash	76.66	86.66	76.66	66.66	86.66	76.66	66.66	76.66	76.66
Bottom Slag - 20 Mesh	21.24	-	-	-	-	-	-	-	10.62
Bed Ash - 20 Mesh	-	11.24	21.24	31.24	11.24	21.24	31.24	21.24	10.62
Silicate	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1	2.1
Silicate Grade	47	47	47	47	47	47	47	47	47
Water	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6.0
Sodium Hydroxide	-	-	-	-	1.0	1.0	1.0	-	-
Firing Rate, °F./Hr.	119	119	119	119	119	119	119	119	119
Final Firing Temp., °F	2100	2100	2100	2100	2100	2100	2100	2100	2100
Cone	3	3	3	3	3	3	3	3	3
% Abs. 24 Hr. Soak	11.30	15.85	20.10	23.96	16.22	19.08	25.64	19.05	16.12
% Abs. 5 Hr. Boil	14.98	19.38	23.58	27.68	20.02	22.40	27.72	22.62	19.67
Saturation Coefficient	0.75	0.83	0.85	0.86	0.81	0.85	0.92	0.84	0.81
Apparent Porosity, %	26.58	32.08	36.99	41.31	32.43	35.55	40.95	35.54	32.18
Bulk Density, Lbs./Cu. Ft.	110.8	103.3	97.8	93.1	101.1	99.0	92.2	98.0	102.1
% Shrinkage	3.2	3.8	1.3	-1.4	3.1	0.7	-2.2	1.7	1.7
Unfired Compressive Strength, P.S.I.	304	66	40	55	96	50	55	41	93
Fired Compressive Strength, P.S.I.	7164	4363	1519	691	3806	1679	785	1760	2404
Suction, Gms. H ₂ O/30 Sq. In./Min.	103	126	178	268	146	185	292	184	163

Note: Batch 8 had a ten (10) minute interval between wetting and pressing.

Thermal Insulating Properties

The thermal insulating properties of fired flyash based structural products are discussed in this report. Thermal conductivities were determined using a comparative thermal conductivity instrument.

Four types of flyash structural products were chosen for this study.

A typical composition for a flyash brick will consist of approximately 72 percent flyash, 25 percent bottom slag, and 3 percent sodium silicate by weight.

Two standard flyash brick were used in this study. The flyashes used in the brick were selected as being representative of different major coal seams. The flyashes are designated as Northern West Virginia and Southern West Virginia.

Two lightweight flyash brick were used in this study. In the lightweight flyash brick a lightweight flyash aggregate is used in place of bottom slag. This results in a flyash brick which is lighter in weight than a standard flyash brick. Lightweight flyash brick are discussed in Coal Research Bureau Technical Report No. 153 "Lightweight Flyash Brick". A Northern West Virginia flyash and a Southern West Virginia flyash were used in the lightweight flyash brick.

For comparative purposes commercial clay brick were obtained from two different sources and were included in this study.

The thermal insulating properties of the fired flyash based structural products were determined by using a comparative thermal conductivity instrument.

The comparative thermal conductivity instrument is used for determining the thermal conductivity of solid materials by comparing this property between the sample material and a known reference material.¹

Thermal conductivity is usually determined by measuring the temperature difference between two points in a material when heat flows from one point in the direction of the other. The temperature difference (ΔT) is related to the amount of heat flow (q) by Fourier's heat conduction equation:

$$\Delta T = \frac{\Delta x}{k} \left[\frac{q}{A} \right]$$

where Δx is the distance between the two temperature measuring points, A is the area of the cross-section perpendicular to the direction of heat flow, and k is the coefficient of thermal conductivity.

It is quite obvious from the above equation that when the quantity q/A is kept the same for two materials, one of known thermal conductivity ($k_{\text{reference}}$) and one of unknown thermal conductivity (k_{sample}), the respective temperature drops are related as follows:

$$\left(\Delta T \frac{k}{\Delta x} \right)_{\text{sample}} = \left(\Delta T \frac{k}{\Delta x} \right)_{\text{reference}}$$

Thus, by measuring the temperature difference between two thermocouples a distance Δx apart in both reference material and test sample, the unknown thermal conductivity is:

$$k_{\text{sample}} = \left(k \frac{\Delta T}{\Delta x} \right)_{\text{reference}} \left(\frac{\Delta x}{\Delta T} \right)_{\text{sample}}$$

The condition of equal heat flux (or heat flow density, q/A) is obtained by placing a test sample and reference material of equal cross-section in intimate contact with each other and by then holding the two between a

heater and a heat sink.

To obtain most accurate results the sample is sandwiched between two identical reference materials. The thermal conductivity is now determined as follows:

$$k_{\text{sample}} = \frac{1}{2} \left(\frac{\Delta x}{\Delta T} \right)_{\text{sample}} \left[\left(k \frac{\Delta T}{\Delta x} \right)_{\text{top reference}} + \left(k \frac{\Delta T}{\Delta x} \right)_{\text{bottom reference}} \right]$$

Thermal conductivity was determined for the two commercial clay brick, two standard flyash brick and two lightweight flyash brick using the comparative thermal conductivity instrument. The mean temperature of the samples was kept as near to ambient temperature as possible and still be consistent with accurate determinations.

The properties of the brick used in the comparative thermal conductivity tests are presented in Table 20. It should be noted that from Table 20 the average thermal conductivity is 1.5 W/m . C for the commercial clay brick, 1.0 W/m . C for the standard flyash brick and 0.8 W/m . C for the lightweight flyash brick.

The lower thermal conductivity values of the flyash brick are the result of the lower bulk density of the flyash brick. The microstructure of flyash brick consists of sealed pores surrounded by touching flyash particles. This type of microstructure results in low thermal conductivity.

Since the thermal conductivity values of the flyash brick are lower than the clay brick, this indicates that 40 percent of the heat which would normally be lost through a clay brick wall would be saved by a flyash brick wall.

It is estimated that 10 quads of energy are consumed each year in the United States for residential heating and cooling.² The use of flyash brick

in place of clay brick would result in considerable energy savings.

In addition to energy savings, the use of flyash brick would increase the usage of flyash, a valuable recyclable by-product produced in electric utility boilers.

Conclusion

This study has shown that, based on thermal conductivity determinations, the use of flyash brick in place of clay brick would save 40 percent of the heat which would normally be lost through a wall.

References

1. Operating Manual for the Comparative Thermal Conductivity Instrument, Dynatech R/D Company, Cambridge, Massachusetts.
2. Reader's Digest, June, 1980, p. 73.

TABLE 20
PROPERTIES OF BRICK USED IN COMPARATIVE THERMAL CONDUCTIVITY TEST

	Clay Brick 1	Clay Brick 2	Northern West Virginia Flyash Brick	Southern West Virginia Flyash Brick	Northern West Virginia Lightweight Flyash Brick	Southern West Virginia Lightweight Flyash Brick
Thermal Conductivity W/m . C	1.46	1.60	1.17	0.78	0.91	0.74
% Abs. 24 Hr. Soak	3.41	3.01	8.73	11.53	11.37	11.95
% Abs. 5 Hr. Boil	3.73	3.68	12.39	15.27	16.65	17.00
Saturation Coefficient	0.91	0.82	0.70	0.76	0.68	0.70
Apparent Porosity, %	8.53	8.68	23.56	26.94	28.47	28.49
Bulk Density, Lbs./Cu. Ft.	142.6	147.2	118.6	110.1	106.7	104.6
Fired Compressive Strength, P.S.I.	7500	3572	6856	7164	4800	7467
Suction, Gms. H ₂ O/30 Sq. In./Min.	3	7	98	108	117	112

Strip Mine Soil Revegetation

Limestone fluidized bed bottom ash (LFBA) would appear to have some potential as a strip mine soil amendment agent because of its high calcium content (37 percent as CaO in the sample collected on this project) and because of its relatively fine particle size. Also LFBA contains various plant nutrients such as Mg, Na and K which could be of importance in the revegetation of strip mine soils.

A thirty gallon sample of strip mine soil was collected from an old abandoned strip mine in northern West Virginia. Although the mine has been inactive for over 10 years, there was little vegetation on the site. The soil sample was collected from an area of the mine in which no vegetation was growing. The pH of the soil as collected was determined to be 3.4. A test of the ability of LFBA to neutralize the acidity of this soil was made (Figure 29.) The results of this test indicated that approximately 10 percent by weight LFBA could produce a soil with a pH of 7 (neutral). Complete neutralization of the acidity of strip mine soil is of course not necessary, or in many cases even desirable, for revegetation. Based on these tests, a plant growth experiment was devised.

Fourteen clay flower pots of approximately 1 quart capacity were purchased. These pots were rinsed in distilled water, then soaked in distilled water for 48 hours and finally rinsed again in distilled water to leach out any water soluble materials within the clay. The pots were then allowed to air dry. Kentucky Fescue 31 and Medium Red Clover were selected as the test plants and seeds acquired. These plants were used in this study because both have application in strip mine revegetation,

the size of the mature plant is proportionate to the size of the flower pots and the seeds were readily available locally. The strip mine soil and the sample of LFBA were riffled and mixed so as to produce two samples each of the following proportions of LFBA to strip soil (by weight): 1 percent LFBA, 5 percent LFBA, 10 percent LFBA, and 20 percent LFBA. These samples and control samples of 100 percent LFBA, 100 percent strip mine soil, and 100 percent potting soil were placed in the clay pots so as to produce two series of seven pots with each pair of pots containing the same material. Accepted mixing and sample splitting techniques were used to assure that all pots contained representative aliquots of the parent sample. Each pot was planted with 25 seeds on a grid pattern to avoid variability in growth due to crowding. All pots were placed on a table on the roof of White Hall, W.V.U. as shown in Figure 30. The plants were watered with distilled water during dry periods and the test was monitored via a plant count, the results of which are shown in Table 21. Basically, most pots containing LFBA showed some improvement over the no growth condition of the strip soil, but fell much below the high growth rate of the potting soil. After approximately one month, plants began to die in all pots apparently due to nutrient deficiency. The test was terminated at 35 days and a new test series begun under the same parameters except for the addition of a fertilizer.

A liquid fertilizer was selected for use in the second test series because of ease of measurement and application. The fertilizer had a guaranteed analysis as follows: Total nitrogen (N) - 6 percent, available phosphoric acid (P_2O_5) - 13 percent, and soluble potash (K_2O) - 6 percent. The previously planted pots were returned to the laboratory where all

surviving plants were removed and the soil broken up and remixed. The 100 percent LFBA samples were found to have cemented into a single mass due solely to the effect of natural weathering conditions. This effect will be discussed later; however, the 100 percent LFBA samples were not used in the second growth test. After the pots were prepared, the seeds were planted as before and 1 ml of fertilizer in 100 mls of water added to each pot. The pots were returned to the roof and growth monitored by a plant count. The pots were fertilized at approximately three week intervals throughout the 56 day test. The results of this test are summarized in Table 22. Both plants did well in the potting soil control pots as was expected. Considering the clover first, the best growth of the experimental pots was obtained from the 5 percent LFBA pot and the second best from the 10 percent LFBA pot. No clover survived after 28 days in the other experimental pots or the 100 percent strip soil control pot. Generally clover survival was poor in all but the potting soil media. Fescue survival was much higher than that of clover. The best survival rate of any pot (including the potting soil) was obtained in the 10 percent LFBA media. The potting soil was second and the 20 percent LFBA a close third. It should be noted at this point that the fescue plants in the potting soil media were approximately 25 percent larger than were those in the 10 percent LFBA media.

As mentioned earlier, the media in the two pots containing 100 percent LFBA was found to have cemented into a single mass due solely to the effect of natural weathering conditions. The material in both pots was found to have a bulk density of 95 lb/ft^3 and a compressive strength of 267 lb/in^2 in one case and 224 lb/in^2 in the other. The unexpectedly high strength of the

LFBA after exposure to weathering suggests several possible applications of this material in the area of soil stabilization. For example, if this material were applied to a haul road, and perhaps wetted, would it serve in the same capacity as cement-treated soil mixtures? A second possibility could be the use of this material to control erosion on strip mine sites and coal refuse piles. A third possibility could be the use of LFBA as a water impervious lining in sediment ponds and diversion ditches. The potential advantage of this material would be that it could be placed where required and allowed to set up by simple exposure to the elements. Unfortunately, these possible applications of LFBA were not noted until too late in the subject contract to allow further investigation.

In order to estimate whether the use of LFBA in strip soil revegetation poses any pollution threat to either ground or surface water, the ASTM proposed method for leaching of waste materials was utilized (D.19.12.03), 2/1/78). Basically this procedure involves the shaking of a known weight of waste (350 grams of LFBA in this case) with either water or acetic acid in an amount equal to four times the weight of the waste for a period of 48 hours. Both distilled water and rain water were used in these tests to determine if the slight acidity of rain fall in Northern West Virginia could affect the leachability of the waste. After leaching, the liquid was removed by filtration and analyzed for the following cations by atomic adsorption: Ag, Al, Ba, Ca, Cd, Cr, Cu, Fe, Hg, Mn and Pb. The only cation of this group which was found to be present in the leachate was Ca. The Ca content of the acid leachate was 1,939 ppm and the Ca content of both the distilled water and the rain water leachates was 991 ppm. These results indicated that

LFBA poses little or no pollution threat to either ground or surface water and that the leaching affects of both distilled water and rainfall on LFBA are identical. Further detailed studies in this area would be necessary for a conclusive evaluation of the water pollution potential of LFBA.

These tests indicate that LFBA does have potential as an aid in strip mine soil revegetation and perhaps as a substitute for agricultural lime. There are also indications that LFBA has potential as a soil stabilization agent.

Table 21

Strip Mine Soil Revegetation Test

Days Since Planting	PLANT COUNT													
	Strip Soil		1 Percent LFBA		5 Percent LFBA		10 Percent LFBA		20 Percent LFBA		100 Percent LFBA		Potting Soil	
	C	F	C	F	C	F	C	F	C	F	C	F	C	F
1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0	0	0	8	0
3	0	0	0	0	0	0	0	0	0	0	0	0	21	0
5	0	0	0	0	2	0	0	0	0	0	0	0	21	6
6	0	0	0	0	4	1	0	0	0	0	0	0	21	9
7	0	0	0	0	4	2	0	0	0	0	0	0	21	11
14	0	0	0	9	1	8	0	10	1	3	6	6	21	19
21	0	0	0	5	0	9	0	8	0	3	3	7	20	19
28	0	0	0	3	0	6	0	5	0	0	1	7	17	18
35	0	0	0	2	0	5	0	4	0	0	1	4	14	11

C - Medium Red Clover
F - Kentucky Fescue 31

Table 22
Strip Soil Revegetation Test With Fertilizer

Days Since Planting	PLANT COUNT											
	Strip Soil		1 Percent LFBA		5 Percent LFBA		10 Percent LFBA		20 Percent LFBA		Potting Soil	
	C	F	C	F	C	F	C	F	C	F	C	F
7*	0	0	0	0	0	0	0	0	0	3	20	3
15	2	6	4	0	8	4	10	22	17	15	21	23
21	0	5	2	0	2	9	4	24	3	16	20	22
28*	0	11	2	0	2	9	3	21	4	21	20	22
35	0	7	0	0	2	8	3	23	0	20	18	20
42	0	9	0	0	2	9	3	23	0	21	20	21
52	0	6	0	0	2	9	2	24	0	20	16	20
56*	0	6	0	0	2	9	1	23	0	18	20	21

C - Medium Red Clover
F - Kentucky Fescue 31

*
Fertilizer applied.

FIGURE 29

STRIP SOIL pH VERSUS LFBA CONTENT

FIGURE 30

LFBA SOIL AMENDMENT TEST SITE

APPENDIX A

Collection of Samples

APPENDIX A
Collection of Samples

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District 3 Coal Preparation Plant
Northern West Virginia

Samples of coal and refuse were obtained from a district three preparation plant cleaning Pittsburgh seam coal. A flowsheet of the plant is included as Figure A-1.

All raw coal coming from the underground longwall operation is screened at 5 inches. The plus 5" is crushed to minus 5" and the resulting 5" x 0 raw coal goes to a 54" pad belt which feeds it onto a slope belt. This 48" belt has a lift of 807 feet and travels at the rate of 625 feet per minute averaging 1,500 tons of coal an hour. The slope belt feeds the coal into a 15,000 ton stockpile.

Under the stockpile are 26 vibrating feeders, any four of which will feed the plant at the rate of 1,200 tons per hour. The coal travels by conveyor from the feeders up into the plant.

The plant is divided into two sections, each having its own jigs, pumps, cyclones, Bird centrifuges, Deister tables and conveyors.

The 5" x 0 coal is fed directly into two Jeffrey Baum jigs. The jigs divide the coal into refuse, middlings, and a clean coal product. The refuse is screened at 3/16", and all above that is carried out to the refuse area. The middlings are screened at 1" and 3/16". The plus 1" is crushed to minus 1" and used in the drying plant for furnace fuel. The 1" x 3/16" is crushed to minus 3/16" and recirculated back through the jigs.

Clean coal coming from the jigs is screened at 1-1/4", 3/4", 3/8" and 3/16". The plus 1-1/4" can be loaded as 5" x 1-1/4" or can be crushed

to minus 1-1/4" and sent out to the loading tracks. The 1-1/4" x 3/4" can be loaded wet or can be combined with the 3/4" x 3/8" and sent to the McNally Vissac coarse coal dryers. The 3/4" x 3/8", when the 1-1/4" is loaded wet, is dried separately in the McNally Vissacs and can go separately to the loading tracks. The 3/8" x 3/16" goes to the flash dryers. The minus 3/16" from the refuse screens, from the middling screens and from the clean coal screens, together with the water from the jigs goes into sumps in the bottom of the plant. From there it is pumped at about 20 percent solids to thickening cyclones. The underflow from these cyclones (about 45 percent solids) goes on the Deister tables where the minus 3/16" is cleaned. The overflow from the cyclones is sent back to the jigs and is recirculated. The 3/16" x 0 refuse from the tables goes into a drag tank and then directly out to the refuse area. The 3/16" x 0 clean coal with its associated water goes to Bird centrifuges, from where it is sent with the 3/8" x 3/16" from the clean coal screens to the Raymond flash dryers.

There are five flash dryers evaporating the water from the 3/8" x 3/16" clean coal. Each flash dryer will evaporate 10 tons of water per hour and handle 100 tons per hour of dry solids.¹

An eight increment sample of raw coal was taken by stopping the 48" wide belt feeding the plant and shoveling a complete cross section approximately equal to the volume of a 55 gallon drum. A similar "belt cut" was repeated every fifteen minutes over a two hour period during the middle of the daylight shift. Total raw coal sample size was approximately 4500 lbs. A similar stop

¹

Plant description courtesy of coal company.

belt sampling technique was used for the washed coal but only four drums were required due to the smaller top size, i.e. $3/8 \times 0$. A bulk sample of coarse and fine refuse weighing approximately 4000 lb. was obtained from the refuse hopper at the end of the shift.

District 4 Coal Preparation Plant
Eastern Ohio

Samples were collected according to the terms of the subject contract from a preparation plant in District 4 (Ohio). Raw coal, clean coal, refuse and fine refuse slurry samples were collected during an eight hour dayshift period.

Eight 55 gallon drums of raw coal were removed from the run-of-mine slope belt feeding storage silos at the plant. The belt was sampled by a four (4) man crew. The belt was stopped for 60 seconds every 15 minutes, allowing the four men to shovel off a three foot cross section. The belt was stopped 10 times over a three hour period in order for the crew to fill the drums. A total of 4,000 lbs. of 4 x 0 run-of-mine coal were taken representing the raw coal feed in this manner.

Eight 55 gallon drums of refuse were obtained by filling the bucket of a front end loader from the main refuse hopper. The refuse was shoveled onto two tarps, coned, quartered and shoveled into the drums. Approximately 6,000 pounds of refuse was collected.

One 30 gallon drum of fine refuse slurry was collected through a 1-inch bleeder pipe from the main 6-inch slurry pipe feeding the slurry pond. The slurry was run at a slow rate allowing a layer of solids to fill up the drum approximately half way. The sample was estimated to be 50 percent solids by volume.

Two 55 gallon drums of 1-1/2" x 0" clean coal were collected by making six sampling increments. The samples were taken from the clean-coal-belt feeding-silo for a railroad car loader. The belt was stopped every fifteen minutes for 1 hour in order to collect a 1,000 pound sample of clean coal.

The samples were all sealed and labeled for delivery and shipped to the coal processing facility in Morgantown. When received in Morgantown the samples of coal and refuse were spread out to air dry and were processed in the normal manner. The slurry sample was sealed in a Nalgene sample vessel pending chemical analysis. A written description of the preparation plant is included in this text.

Description of the Pittsburgh No. 8 Seam Preparation Plant

Coal cleaning in the Pittsburgh No. 8 plant was accomplished by 3-product jig for coarse coal and classifying cyclones for fine coal.

Scalped 4" x 0" Run-of-mine coal was fed to the twin jig circuit where a 3-product separation was made. The clean coal product from the jig was dewatered and sized on double-deck screens. The screens separated the clean coal into 4" x 1 1/4" x 1/4", and 1/4" x 0 size fractions. The middlings from the jig circuit were dewatered and sized at 1/2" on a screen. Refuse from the jigs was also passed over a dewatering screen. The plus 28 mesh refuse was transferred to a refuse belt and the minus 28 M refuse was combined with the -1/2" material from the middling screen and sent to a spiral classifier. A 65 M separation was made by the refuse classifier; plus 65 M material was transferred to the refuse belt, and minus 65 M material was sent to a static thickener.

The plus 1/2" coal from the middling screen and the 4" x 1 1/2" coal from the clean coal screen were combined and sent to a ring crusher which reduced the top size of the coal to 1 1/2". The 1 1/2" x 0 product from the crusher was transferred to the clean coal belt.

The 1-1/4" x 1/4" material from the clean coal screens was dewatered in centrifuges and discharged onto the clean coal belt. Underflow from the clean coal screens (-1/4") was combined with water from the jig and was pumped to classifying cyclones which made a separation at 28 M. The 1/4" x 28 M underflow from the cyclones was dewatered with screens and centrifuges and discharged on the clean-coal belt. The overflow from the cyclone (-28 M), the underflow from the screens, and the effluent from the centrifuges were pumped to secondary cyclones where a separation at 200 M was made. The 28 M x 200 M underflow from the cyclones was dewatered by vacuum disc filters and discharged onto the clean coal belt. The minus 200 M overflow from the secondary cyclones was sent to the static thickener.

Fluidized Bed Combustor Facility
Rivesville, West Virginia

Samples of feed coal, feed limestone, flyash from the carbon burn-off cell and spent bed material were obtained from the limestone fluidized bed combustor facility in Rivesville, West Virginia.

Three 55-gallon drums of flyash from the carbon burn-off cell flyash silo (see sample point four, Figure A-2) were obtained by fabricating a special drum lid which was attached to a two inch drop pipe from the bottom of CBC ash silo. To insure representativeness, the sample was taken over a four hour period of a steady state run. It was not practical to obtain samples of precipitator ash (sample point five, Figure A-2) during this trip.

As illustrated in Figure A-2, fine ash, accounting for 20 percent of total flyash is recycled to the carbon burn-off cell from the number one and two precipitators at a designed 90 percent of input, leaving only two percent of the total flyash produced collected by precipitators 3, 4, 5, and 6 (proposed sample point 5). Therefore, the flyash sample obtained at sample point 4 represents 98 percent of total flyash waste from the process. Construction work on a separate fluidized bed waste silo prevented procurement of sample five.

A thirty gallon drum of feed coal $1\frac{1}{2}$ " x $\frac{1}{4}$ " was obtained from a surge bin directly feeding the combustion cells. An inspection plate on the top surface of the bin was removed to gain access to the bin interior where a bucket was passed across the feed chute opening to the bin periodically during feeding to obtain a representative sample. Approximately 350 pounds of feed coal were obtained in this manner. A thirty gallon drum of limestone was obtained by systematic shoveling of a conical pile of 6

x 16 mesh limestone within a storage silo. Approximately 600 pounds of limestone feed was obtained in this manner.

The spent bed material as illustrated in Figure A-2, is pumped pneumatically from the plant to a trailer truck. While still hot, this material was trucked to a disposal site approximately three miles away where a flexible four inch hose from the trailer truck was used to fill twelve 55-gallon drums. In all, nearly 8,500 pounds of spent bed material was obtained. Since this material contains a significant quantity of free lime, it readily absorbs water; therefore, the drums were sealed immediately on site with a standard drum gasket.

NOTE: Just a few hours after the Rivesville sample operation, the facility was forced to shut down due to a tragic fire which significantly damaged the plant. Fortunately, sufficient quantities of samples were obtained during a steady run to perform the intended research on the subject contract.

SCHMATIC FLOW DIAGRAM OF FLUID
BED COMBUSTOR

FIGURE A2

Solvent Refined Coal II Facility
Fort Lewis, Washington

A second sampling trip was made to the solvent refined coal facility in Fort Lewis, Washington. Arrangements were made with plant operation personnel to sample the feed coal weigh-feeder belt every fifteen minutes during an eight hour period. A full-stream belt cut was taken by diverting the entire flow of 1/2" x 0" coal off the end of the belt into the sample container for a 30 second time period. A total of 160 pounds of coal was collected in this manner.

Through consultation with the plant engineer a plant throughput time of eight hours was determined for the current operating conditions. This meant that the first portion of minerals from the sampled feed coal would appear in the waste material eight hours after the first coal sample was taken. Accordingly, eight hours after the first feed coal sample was taken, three-eighths of a 55-gallon steel drum of vacuum bottoms, a medium melting-point tar material, was removed from the waste belt. Again, to achieve the most representative sample possible, sample was removed from the entire belt width and diverted by a chute into the barrels. At the point of sampling the material was flexible but beginning to quickly solidify into brittle strips at an approximate temperature of 135°F. The material was sampled each half-hour over an eight hour period resulting in a total sample volume of six 55-gallon drums.

Immediately following sampling, each drum (including the feed coal) was sealed with butyl rubber, evacuated with a vacuum pump, and refilled to atmospheric pressure with nitrogen for subsequent shipping to the bulk sample processing facility in Westover, West Virginia. Samples were received

approximately three weeks later and processed for analytical characterization procedures with the remainder of sample stored in inert atmosphere for further utilization testing.

APPENDIX B

Characterization of Samples

APPENDIX B
Characterization of Samples

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DISTRICT 3 COAL PREPARATION PLANT

NORTHERN WEST VIRGINIA

DISTRICT 3 COAL PREPARATION PLANT
NORTHERN WEST VIRGINIA

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DISTRICT 3 COAL PREPARATION PLANT
NORTHERN WEST VIRGINIA

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TABLE B-1
CHEMICAL CHARACTERIZATION DATA*
(Pittsburgh Head Samples)

	Spectrographic Analysis**						Atomic*** Absorption		LTA	Btu
	<u>Si</u>	<u>Al</u>	<u>Fe</u>	<u>Ti</u>	<u>Ca</u>	<u>Mg</u>	<u>Na</u>	<u>K</u>		
Raw Coal	2.98	1.56	1.83	0.08	0.63	0.10	0.07	0.34	17.3	13,102
Raw Coal Composite ⁺	2.65	1.29	1.33	0.06	0.59	0.10	0.08	0.15	15.8	13,318
Clean Coal	1.66	1.07	1.20	0.05	0.40	0.06	0.07	0.08	10.1	14,020
Refuse	<u>14.77</u>	<u>6.00</u>	<u>9.18</u>	<u>0.29</u>	<u>4.77</u>	<u>0.52</u>	<u>0.31</u>	<u>0.52</u>	<u>83.2</u>	<u>4,403</u>

	Proximate Analysis				Sulfur Breakdown			
	<u>Moisture</u>	<u>Ash</u>	<u>Volatile Matter</u>	<u>Fixed Carbon</u>	<u>Sulfate S</u>	<u>Pyrite S</u>	<u>Organic S</u>	<u>Total S</u>
Raw Coal	1.0	13.9	36.5	50.4	0.04	1.57	1.40	3.01
Raw Coal Composite	0.9	12.9	37.9	49.2	0.01	1.50	1.50	3.01
Clean Coal	1.2	8.0	39.5	52.5	0.01	1.09	1.60	2.70
Refuse	<u>0.7</u>	<u>71.3</u>	<u>19.5</u>	<u>9.2</u>	<u>0.11</u>	<u>8.35</u>	<u>0.37</u>	<u>8.83</u>

*

Data reported on moisture free basis.

**

Percent element on a moisture free whole coal basis.

Percent element in the ash.

+

Recombined head sample pro-rated on weight percent of component gravity fractions.

TABLE B-2

RATIO OF ELEMENTAL CONCENTRATIONS IN HEAD SAMPLES

<u>Pittsburgh Head Samples</u>	<u>Si</u>	<u>Al</u>	<u>Fe</u>	<u>Ti</u>	<u>Ca</u>	<u>Mg</u>
Raw Coal/Refuse	0.20	0.26	0.20	0.27	0.13	0.19
Clean Coal/Refuse	0.11	0.18	0.13	0.17	0.08	0.12
<u>Clean Coal/Raw Coal</u>	<u>0.56</u>	<u>0.69</u>	<u>0.66</u>	<u>0.63</u>	<u>0.63</u>	<u>0.60</u>

Sulfur Forms

	<u>Sulfate S</u>	<u>Pyrite S</u>	<u>Organic S</u>	<u>Total S</u>
Raw Coal/Refuse	0.36	0.19	3.78	0.34
Clean Coal/Refuse	0.09	0.13	4.30	0.31
<u>Clean Coal/Raw Coal</u>	<u>0.25</u>	<u>0.69</u>	<u>1.10</u>	<u>0.89</u>

*

Based on Table 1 data.

TABLE B-3

Pittsburgh Seam Raw Coal

	<u>Spectrographic Analysis</u> *						<u>Atomic Absorption</u> **			
	<u>Si</u>	<u>Al</u>	<u>Fe</u>	<u>Ti</u>	<u>Ca</u>	<u>Mg</u>	<u>Na</u>	<u>K</u>	<u>BTU</u>	<u>LTA</u>
<u>+1" Coal</u>										
1.30 Float	1.11	0.70	0.76	0.04	0.22	0.04	0.48	0.95	14,431	7.8
1.40 Float	1.85	0.96	2.13	0.05	0.55	0.06	0.42	0.93	13,620	13.6
1.60 Float	3.62	1.16	2.00	0.09	2.73	0.65	0.31	1.30	11,806	26.0
1.80 Float	7.57	2.13	2.00	0.27	1.67	0.43	0.49	1.15	9,497	41.0
1.80 Sink	14.29	5.68	6.28	0.29	5.66	0.70	0.33	1.09	4,488	82.5
<u>1" x 1/4" Coal</u>										
1.30 Float	1.04	0.63	0.75	0.04	0.15	0.03	0.40	0.80	14,472	7.2
1.40 Float	2.16	0.87	1.70	0.05	0.28	0.06	0.96	1.01	13,465	14.8
1.60 Float	4.16	1.83	3.94	0.11	1.00	0.18	1.22	0.91	11,428	28.7
1.80 Float	6.55	1.91	5.52	0.17	2.48	0.49	1.08	1.28	9,396	42.4
1.80 Sink	15.24	5.88	6.19	0.35	4.82	0.44	1.22	1.10	4,197	84.6
<u>1/4" x 8 Mesh</u>										
1.30 Float	0.98	0.59	0.47	0.03	0.12	0.03	2.00	0.96	14,455	7.1
1.40 Float	3.84	1.66	3.23	0.10	0.79	0.11	1.32	1.39	12,156	23.7
1.60 Float	4.22	1.85	4.39	0.14	1.08	0.16	3.40	0.99	11,626	27.7
1.80 Float	5.90	2.11	6.09	0.14	0.32	0.32	2.56	0.94	9,969	39.0
1.80 Sink	14.37	5.37	9.08	0.35	4.32	0.47	0.90	1.11	4,216	84.4

*

Percent element on a moisture free whole coal basis (for each fraction).

**

Percent element in the ash.

TABLE B-3 (Continued)
Pittsburgh Seam Raw Coal

	<u>Spectrographic Analysis*</u>						<u>Atomic Absorption**</u>		<u>BTU</u>	<u>LTA</u>
	<u>Si</u>	<u>Al</u>	<u>Fe</u>	<u>Ti</u>	<u>Ca</u>	<u>Mg</u>	<u>Na</u>	<u>K</u>		
<u>8 x 28 Mesh</u>										
1.30 Float	0.87	0.63	0.44	0.03	0.13	0.03	0.34	1.00	14,566	6.1
1.40 Float	2.02	1.01	1.72	0.06	0.28	0.04	0.25	1.06	13,537	13.6
1.60 Float	3.82	1.68	3.84	0.09	0.86	0.13	0.69	0.89	12,182	24.3
1.80 Float	5.23	2.00	5.72	0.13	2.95	0.34	0.68	0.88	9,867	39.3
1.80 Sink	13.27	5.64	11.00	0.32	4.17	0.45	0.40	0.93	4,413	82.4
<u>28 x 100 Mesh</u>										
1.30 Float	1.03	0.70	0.46	0.03	0.22	0.04	0.87	1.14	14,509	7.0
1.40 Float	1.69	0.97	1.16	0.05	0.21	0.04	0.79	1.12	13,913	11.4
1.60 Float	2.83	1.47	2.47	0.08	0.53	0.09	0.68	1.12	12,658	19.9
1.80 Float	4.77	2.02	4.27	0.11	2.30	0.26	0.62	0.81	10,345	35.9
1.80 Sink	13.96	3.33	10.75	0.20	7.32	0.46	0.77	0.74	5,016	85.2
Minus 100 Mesh	3.98	1.63	2.25	0.09	1.53	0.16	0.91	1.04	11,901	24.8
<u>Pittsburgh Seam Refuse</u>										
(Pulverized)	14.77	6.00	9.18	0.29	4.77	0.52	0.43	0.74	4,403	83.2

*

Percent element on a moisture free whole coal basis (for each fraction).

**

Percent element in the ash.

TABLE B-4

ULTIMATE ANALYSIS
(PITTSBURGH SEAM - SIZED, GRAVITY FRACTIONS)

<u>Sample</u>	<u>C</u>	<u>H</u>	<u>O</u>	<u>N</u>	<u>S</u>
<u>+1" Coal</u>					
Float 1.3	79.70	5.28	4.17	1.51	2.45
1.3 x 1.4	74.69	4.97	3.60	1.25	3.72
1.4 x 1.6	66.86	4.38	0.51	1.09	3.80
1.6 x 1.8	52.64	3.35	1.21	0.81	3.10
Sink 1.8	17.72	1.24	2.39	0.27	7.55
<u>1" x 1/4" Coal</u>					
Float 1.3	78.39	5.35	7.34	0.65	2.22
1.3 x 1.4	72.54	4.94	5.06	0.54	3.92
1.4 x 1.6	62.42	4.13	2.53	0.41	5.62
1.6 x 1.8	51.59	3.16	1.38	0.04	6.64
Sink 1.8	15.64	1.25	3.44	0.10	6.49
<u>1/4" x 8 Mesh Coal</u>					
Float 1.3	78.41	5.28	7.36	0.76	2.15
1.3 x 1.4	62.29	4.42	4.99	0.52	4.72
1.4 x 1.6	62.60	4.15	3.49	0.49	5.85
1.6 x 1.8	54.06	3.28	0.96	0.20	6.89
Sink 1.8	15.78	1.19	1.11	0.10	8.67
<u>8 x 28 Mesh Coal</u>					
Float 1.3	78.09	5.29	9.37	0.48	2.04
1.34 x 1.4	72.19	4.86	7.51	0.34	3.43
1.4 x 1.6	65.46	4.46	3.40	0.48	5.49
1.6 x 1.8	54.03	3.10	0.45	0.75	6.82
Sink 1.8	15.83	1.21	0.29	0.24	9.92
<u>28 x 100 Mesh Coal</u>					
Float 1.3	78.80	5.19	5.95	1.78	2.11
1.3 x 1.4	76.03	4.99	4.09	1.30	2.76
1.4 x 1.6	70.31	4.47	2.71	1.24	4.09
1.6 x 1.8	58.70	3.26	0.34	0.71	5.99
Sink 1.8	17.25	0.94	2.30	0.26	11.13
<u>Minus 100 Mesh Coal</u>					
	64.36	4.14	5.29	0.24	3.60
<u>Raw Coal Head</u>					
	72.38	4.68	3.38	1.25	3.01

*Percent element on a moisture free whole coal basis (for each fraction).

TABLE B-5

FINAL LOW TEMPERATURE ASH (LTA) VALUES OF PITTSBURGH SEAM SAMPLES
(Including Other Comparable Mineral Matter Parameters)

<u>Sample</u>		<u>Ash</u>	<u>Parr M.M.</u>	<u>LTA</u>
Pittsburgh Seam Raw Coal +1"	1.30 Float	5.6%	7.4%	7.8%
Pittsburgh Seam Raw Coal +1"	1.40 Float	10.3	13.2	13.6
Pittsburgh Seam Raw Coal +1"	1.60 Float	22.2	26.1	26.0
Pittsburgh Seam Raw Coal +1"	1.80 Float	37.9	42.6	41.1
Pittsburgh Seam Raw Coal +1"	1.80 Sink	70.1	79.9	82.5
Pittsburgh Seam Raw Coal 1 x 1/4"	1.30 Float	5.4	7.0	7.2
Pittsburgh Seam Raw Coal 1 x 1/4"	1.40 Float	11.5	14.6	14.8
Pittsburgh Seam Raw Coal 1 x 1/4"	1.60 Float	24.2	29.2	28.7
Pittsburgh Seam Raw Coal 1 x 1/4"	1.80 Float	36.7	43.3	42.4
Pittsburgh Seam Raw Coal 1 x 1/4"	1.80 Sink	72.6	82.0	84.6
Pittsburgh Seam Raw Coal 1/4 x 8m	1.30 Float	5.3	6.9	7.1
Pittsburgh Seam Raw Coal 1/4 x 8m	1.40 Float	19.5	23.7	23.7
Pittsburgh Seam Raw Coal 1/4 x 8m	1.60 Float	22.9	28.0	27.7
Pittsburgh Seam Raw Coal 1/4 x 8m	1.80 Float	34.0	40.5	39.0
Pittsburgh Seam Raw Coal 1/4 x 8m	1.80 Sink	72.5	83.1	84.4
Pittsburgh Seam Raw Coal 8 x 28m	1.30 Float	4.1	5.6	6.1
Pittsburgh Seam Raw Coal 8 x 28m	1.40 Float	10.4	13.1	13.6
Pittsburgh Seam Raw Coal 8 x 28m	1.60 Float	19.7	24.3	24.3
Pittsburgh Seam Raw Coal 8 x 28m	1.80 Float	33.8	40.3	39.3
Pittsburgh Seam Raw Coal 8 x 28m	1.80 Sink	71.5	82.7	82.4
Pittsburgh Seam Raw Coal 28 x 100m	1.30 Float	5.1	6.7	7.0
Pittsburgh Seam Raw Coal 28 x 100m	1.40 Float	8.9	11.1	11.4
Pittsburgh Seam Raw Coal 28 x 100m	1.60 Float	16.2	19.8	19.9
Pittsburgh Seam Raw Coal 28 x 100m	1.80 Float	31.0	36.8	35.9
Pittsburgh Seam Raw Coal 28 x 100m	1.80 Sink	67.4	78.9	85.2
Pittsburgh Seam Raw Coal -100m screen fraction		21.2	24.9	24.8
Pittsburgh Seam Refuse Head Sample		71.3	81.9	83.2
Pittsburgh Seam Clean Coal Head		8.0	10.1	10.1
Pittsburgh Seam Raw Coal Head Sample		12.9	15.6	15.8
Pittsburgh Seam Raw Coal Head Sample		13.9	16.7	17.3

TABLE B-6

FLOAT AND SINK DATA (+ 1", PITTSBURGH SEAM HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
	(2) Wt %	(3) Ash %	(4) Ash Prod.	(5) Wt %	(6) Ash Prod.	(7) Ash %	(8) Wt %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	64.7	5.6	362.32	64.7	362.32	5.60	100.00	1617.51	16.18	2.44
1.3 x 1.4	13.8	10.3	142.14	78.5	504.46	6.43	35.30	1255.19	35.56	2.49
1.4 x 1.6	7.5	22.2	166.50	86.0	670.96	7.80	21.50	1113.05	51.77	2.60
1.6 x 1.8	1.3	37.9	49.27	87.3	720.23	8.25	14.00	946.55	67.61	2.62
Sink 1.8	12.8	70.1	897.28	100.1	1617.51	16.18	12.70	897.28	70.65	3.25

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FLOAT AND SINK DATA (1" x 1/4", PITTSBURGH SEAM HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
	(2) Wt %	(3) Ash %	(4) Ash Prod.	(5) Wt %	(6) Ash Prod.	(7) Ash %	(8) Wt %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	70.4	5.4	380.16	70.40	380.16	5.40	100.00	1318.63	13.19	2.22
1.3 x 1.4	15.8	11.5	181.70	86.20	561.86	6.52	29.60	938.47	31.71	2.53
1.4 x 1.6	4.1	24.2	99.22	90.30	661.08	7.32	13.80	756.77	54.84	2.67
1.6 x 1.8	1.3	36.7	47.71	91.60	708.79	7.74	9.70	657.55	67.79	2.73
Sink 1.8	8.4	72.6	609.84	100.00	1318.63	13.19	8.40	609.84	72.60	3.05

TABLE B-6 (Continued)

FLOAT AND SINK DATA (1/4" x 8 M, PITTSBURGH SEAM HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
	(2) Wt %	(3) Ash %	(4) Ash Prod.	(5) Wt %	(6) Ash Prod.	(7) Ash %	(8) Wt %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	83.2	5.3	440.96	83.20	440.96	5.30	100.00	1062.63	10.63	2.15
1.3 x 1.4	8.3	19.5	161.85	91.50	602.81	6.59	16.80	621.67	37.00	2.38
1.4 x 1.6	2.3	22.9	52.67	93.80	655.48	6.99	8.50	459.82	54.10	2.46
1.6 x 1.8	1.1	34.0	37.40	94.90	692.88	7.30	6.20	407.15	65.67	2.51
Sink 1.8	5.1	72.5	369.75	100.00	1062.63	10.63	5.10	369.75	72.50	2.82

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FLOAT AND SINK DATA (8 M x 28 M, PITTSBURGH SEAM HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
	(2) Wt %	(3) Ash %	(4) Ash Prod.	(5) Wt %	(6) Ash Prod.	(7) Ash %	(8) Wt %	(9) Ash Prod.	(10) Ash %	(11)
Float 1.3	78.6	4.1	322.26	78.6	322.26	4.10	100.00	950.85	9.51	2.40
1.3 x 1.4	10.6	10.4	110.24	89.2	432.50	4.85	21.40	628.59	29.37	2.52
1.4 x 1.6	4.1	19.7	80.77	93.3	513.27	5.50	10.80	518.35	48.00	2.65
1.6 x 1.8	1.1	33.8	37.18	94.4	550.45	5.83	6.70	437.58	65.31	2.70
Sink 1.8	5.6	71.5	400.40	100.00	950.85	9.51	5.60	400.40	71.50	3.10

TABLE B-6 (Continued)

FLOAT AND SINK DATA (28 M x 100 M, PITTSBURGH SEAM HEAD SAMPLE)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
	Wt %	Ash %	Ash Prod.	Wt %	Ash Prod.	Ash %	Wt %	Ash Prod.	Ash %	
Float 1.3	75.6	5.1	385.56	75.6	385.56	5.10	100.00	1038.03	10.38	2.11
1.3 x 1.4	10.5	8.9	93.45	86.1	479.01	5.56	24.4	652.47	26.74	2.19
1.4 x 1.6	6.1	16.2	98.82	92.2	577.83	6.27	13.9	559.02	40.22	2.32
1.6 x 1.8	1.8	31.0	55.80	94.0	633.63	6.74	7.8	460.20	59.00	2.39
Sink 1.8	6.0	67.4	404.40	100.0	1038.03	10.38	6.0	404.40	67.40	2.91

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FLOAT AND SINK DATA (+100 Mesh Composite)

Specific Gravity	Individual Fractions			Cumulative Float			Cumulative Sink			Cumulative Sulfur
	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
	Wt %	Ash %	Ash Prod.	Wt %	Ash Prod.	Ash %	Wt %	Ash Prod.	Ash %	
Float 1.3	73.9	5.2	384.3	73.9	384.3	5.2	99.8	1241.1	12.4	2.27
1.3 x 1.4	12.4	12.6	156.2	86.3	540.4	6.3	25.9	856.8	33.1	2.45
1.4 x 1.6	4.6	22.0	101.2	90.9	641.7	7.1	13.5	700.6	51.9	2.57
1.6 x 1.8	1.0	35.3	35.3	91.9	677.0	7.4	8.9	599.4	67.3	2.62
Sink 1.8	7.9	71.4	564.1	99.8	1241.1	12.4	7.9	564.1	71.4	3.03

TABLE B-7
FROTH FLOTATION RESULTS
-50 M Pittsburgh

<u>Sample Description</u>	<u>Reagents</u> ¹	<u>Yield</u>	<u>Moisture</u>	<u>Ash</u> ²	<u>Volatile</u> ² <u>Matter</u>	<u>Fixed</u> ² <u>Carbon</u>
Head Sample	-----	---	0.7	19.6	34.1	46.3
Flotation Product	MIBC-0.53	72%	0.4	11.3	34.9	53.9
Flotation Refuse	MIBC-0.53	28%	0.3	40.1	28.3	31.6
Flotation Product	MIBC-0.23	75%	0.5	8.6	35.6	55.8
Flotation Refuse	MIBC-0.23	25%	0.2	50.9	25.1	24.0
Flotation Product	MIBC-0.23 Pyrite Depressant	70%	1.0	9.3	33.7	57.0
Flotation Refuse	MIBC-0.23 Pyrite Depressant	30%	1.0	37.2	28.3	34.5
Flotation Product	MIBC-0.23 Wetting Agent	76%	0.9	9.9	36.0	54.1
Flotation Refuse	MIBC-0.23 Wetting Agent	24%	1.0	35.6	28.4	36.8

1. lb./ton

2. percentage of whole coal, dry basis

TABLE B-7 (Continued)
FROTH FLOTATION RESULTS
-50 M Pittsburgh

<u>Sample Description</u>	<u>Total² Sulfur</u>	<u>Sulfate² Sulfur</u>	<u>Pyritic² Sulfur</u>	<u>Inorganic² Sulfur</u>	<u>Organic² Sulfur</u>	<u>BTU</u>
Head Sample	3.26	----	----	----	----	-----
Flotation Product	3.14	0.02	1.60	1.62	1.52	13,350
Flotation Refuse	3.65	0.03	2.63	2.66	0.99	-----
Flotation Product	2.85	0.02	1.37	1.39	1.46	13,776
Flotation Refuse	4.45	0.05	3.78	3.83	0.62	-----
Flotation Product	2.95	0.05	1.31	1.36	1.59	13,359
Flotation Refuse	3.95	0.08	2.83	2.91	1.04	-----
Flotation Product	3.01	0.10	1.47	1.57	1.47	13,568
Flotation Refuse	3.78	0.11	2.67	2.78	1.00	8,845

2. percentage of whole coal, dry basis

TABLE B-8

PETROGRAPHIC ANALYSES OF PITTSBURGH COAL FLOAT-SINK FRACTIONS AND HEAD
SAMPLES PRESENTED AS VOLUME PERCENT OF THE WHOLE COAL

	+1"					1" x 1/4"					1/4 x 8 Mesh				
	1.3F	1.4F	1.6F	1.8F	1.8S	1.3F	1.4F	1.6F	1.8F	1.8S	1.3F	1.4F	1.6F	1.8F	1.8S
VITRINITE	78	66	54	27	6	80	64	48	32	8	79	66	52	31	8
Telinite	1	0	1	0	0	0	1	0	0	0	1	1	1	1	0
Collinite	77	66	53	27	6	80	63	47	31	7	78	65	51	30	8
Telocollinite	76	62	50	26	5	77	57	42	27	7	75	61	44	26	7
Desmocollinite	1	3	2	1	1	2	5	5	5	0	2	4	6	4	1
EXINITE	4	6	4	5	1	4	5	6	6	1	5	4	4	6	1
Sporinite	2	2	1	0	0	2	2	2	1	0	3	1	1	1	0
Cutinite	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Liptodetrinite	1	4	3	4	1	2	4	3	5	1	2	2	3	5	1
INERTINITE	13	16	16	22	2	10	17	21	24	5	12	15	23	27	6
Fusinite	1	1	3	3	0	1	1	4	8	1	1	2	4	9	2
Pyrofusinite	0	1	2	2	0	0	0	3	5	1	0	1	2	4	1
Degradofusinite	1	0	1	2	0	1	0	1	3	0	0	0	2	5	1
Semifusinite	4	6	3	3	0	4	7	7	4	0	3	6	9	4	1
Pyrosemifusinite	0	0	0	0	0	0	0	0	1	0	0	1	1	1	0
Degradosemifusinite	4	5	3	3	0	4	6	6	3	0	3	6	8	3	1
Macrinite	1	1	3	5	1	1	1	1	4	0	1	2	2	5	0
Micrinite	3	2	2	1	0	3	3	1	1	0	2	1	1	1	0
Inertodetrinite	4	5	5	9	1	3	6	8	8	3	3	4	6	7	3
MINERAL MATTER	5	12	26	46	91	6	13	26	37	86	4	15	21	36	86

TABLE B-8 (Continued)

**PETROGRAPHIC ANALYSES OF PITTSBURGH COAL FLOAT-SINK FRACTIONS AND HEAD
SAMPLES PRESENTED AS VOLUME PERCENT OF THE WHOLE COAL**

	8 x 28 Mesh					28 x 100 Mesh					-100 Mesh Screen Fraction	Raw Coal Head	Clean Coal Head	Refuse Head
	1.3F	1.4F	1.5F	1.8F	1.8S	1.3F	1.4F	1.6F	1.8F	1.8S				
VITRINITE	83	67	58	31	10	85	73	64	41	10	75	75	77	13
Telinite	2	1	0	0	0	1	0	0	0	0	1	0	1	0
Collinite	82	67	58	31	9	85	72	64	41	10	74	74	76	12
Telocollinite	78	59	55	28	9	81	66	58	38	10	72	70	71	12
Desmocollinite	3	7	3	3	1	3	7	5	3	0	2	5	4	1
EXINITE	3	3	2	2	1	3	3	4	3	0	1	3	4	0
Sporinite	2	2	0	1	0	1	2	1	0	0	0	1	2	0
Cutinite	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Liptodetrinite	1	1	2	2	0	1	2	3	2	0	1	2	2	0
INERTINITE	9	18	22	30	7	7	16	20	27	7	15	11	12	5
Fusinite	1	2	4	9	2	1	2	3	12	4	1	1	1	1
Pyrofusinite	0	1	2	6	1	0	1	2	8	3	1	1	1	1
Degradofusinite	0	1	3	5	2	1	1	2	4	2	0	0	1	0
Semifusinite	3	7	9	5	1	2	6	10	6	1	3	4	4	1
Pyrosemifusinite	0	1	1	1	0	0	1	1	1	0	0	0	0	1
Degradosemifusinite	3	7	8	4	1	2	5	9	5	1	3	4	4	1
Macrinite	0	2	2	6	1	1	1	1	4	0	0	0	0	0
Micrinite	3	1	1	0	0	2	2	1	1	0	2	1	2	0
Inertodetrinite	2	5	6	8	2	2	5	4	5	1	9	5	4	2
MINERAL MATTER	5	12	18	36	83	5	8	12	30	83	8	11	7	82

TABLE B-9

MACERALS PRESENT IN THE PITTSBURGH DISTRICT 7 COAL SAMPLES IN
AMOUNTS GREATER THAN 2 PERCENT BY VOLUME WITH
THEIR MINIMUM AND MAXIMUM OBSERVED VALUES

GROUP MACERAL			
Maceral		Minimum	Maximum
submaceral			
VITRINITE		6% (48%)*	85% (39%)*
Telinite		0%	2%
Collinite		6%	85%
telocollinite		5%	81%
desmocolinite		0%	7%
EXINITE		0% (0%)	6% (11%)
Sporinite		0%	3%
Liptodetrinite		0%	5%
INERTINITE		2% (7%)	30% (43%)
Fusinite		0%	9%
pyrofusinite		0%	8%
degradofusinite		0%	5%
Semifusinite		0%	9%
degradosemifusinite		0%	9%
Macrinite		0%	6%
Micrinite		0%	3%
Inertodetrinite		1%	9%
MINERAL MATTER		4%	91%

*

Values in parentheses are values of the maceral group recalculated on a mineral-matter-free basis.

TABLE B-10

PITTSBURGH COAL

<u>Sample Description</u>	(District 3) <u>Absorbance ($\log I_0/I$)</u>	<u>Percent Kaolinite</u>
+1", 1.30 Float	.166	22
1 x 1/4, 1.30 Float	.194	26
1/4 x 8 Mesh, 1.30 Float	.168	22
8 x 28 Mesh, 1.30 Float	.201	27
28 x 100 Mesh, 1.30 Float	.219	29 AVE = 25%
+1", 1.40 Float	.107	14
1 x 1/4, 1.40 Float	.161	21
1/4 x 8 Mesh, 1.40 Float	.125	16
8 x 28 Mesh, 1.40 Float	.185	24
28 x 100 Mesh, 1.40 Float	.184	24 AVE = 20%
+1", 1.60 Float	.066	9
1 x 1/4, 1.60 Float	.099	13
1/4 x 8 Mesh, 1.60 Float	.097	13
8 x 28 Mesh, 1.60 Float	.131	17
28 x 100 Mesh, 1.60 Float	.170	22 AVE = 15%
+1", 1.80 Float	.085	11
1 x 1/4, 1.80 Float	.083	11
1/4 x 8 Mesh, 1.80 Float	.073	10
8 x 28 Mesh, 1.80 Float	.078	10
28 x 100 Mesh, 1.80 Float	.101	13 AVE = 11%
+1", 1.80 Sink	.114	15
1 x 1/4, 1.80 Sink	.116	15
1/4 x 8 Mesh, 1.80 Sink	.038	12
8 x 28 Mesh, 1.80 Sink	.079	10
28 x 100 Mesh, 1.80 Sink	.060	8 AVE = 12%

MINERALS OF THE DISTRICT #3 PITTSBURGH COAL.
SYMBOLS INDICATE ANALYTICAL PROCEDURES AVAILABLE FOR EACH
MINERAL AND WHETHER THE PROCEDURE CAN BE USED FOR
QUANTITATIVE, SEMIQUANTITATIVE, OR QUALITATIVE MINERAL ANALYSIS.

	<i>X-ray Powder Diffraction</i>	<i>Infrared Spectroscopy</i>	<i>Normative Calculations</i>	<i>Optical Petrography</i>	<i>Scanning Electron Microscopy</i>	<i>Formulae</i>
ILLITE	S		Q	S	I	variable
KAOLINITE	Q	Q	Q	S	I	$Al_4(Si_4O_{10})(OH)_8$
QUARTZ	Q		Q	S	I	SiO_2
FELDSPARS	S				I	variable
MUSCOVITE				S	I	$KAl_4(AlSi_3O_{10})(OH)_2$
CARBONATES		Q		S		variable
CALCITE	Q	Q	S	I	I	$CaCO_3$
DOLOMITE	Q		S	I	I	$CaMg(CO_3)_2$
BASSANITE	S				I	$CaSO_4 \cdot \frac{1}{2}H_2O$
GYPSUM		I		I		$CaSO_4 \cdot 2H_2O$
IRON DISULFIDES	Q		Q	S	I	FeS_2
PYRITE				S		FeS_2
MARCASITE				S		FeS_2
APATITE	S				I	$Ca_5(F,Cl,OH)(PO_4)_3$
HEMATITE				S		Fe_2O_3
RUTILE				S	I	TiO_2

Q = Quantitative determinations ($\pm 10\%$)

S = Semiquantitative determinations ($\pm 10-30\%$)

I = Identification only possible

TABLE B-12

TYPICAL MINERAL SIZES IN THE DISTRICT #3 PITTSBURGH COAL SAMPLE

<u>Mineral</u>	<u>Crystallite Size</u>	<u>Mineral</u>	<u>Crystallite Size</u>
Illite	2u	Pyrite	20u
Kaolinite	10u	Marcasite	20u
Quartz	10u	Muscovite	50u
Calcite	500u	Hematite	1u
Dolomite	100u	Rutile	2u

TABLE B-13

OPTICAL CHARACTERISTICS OF MINERALS IN THE DISTRICT # 3
PITTSBURGH COAL SAMPLE

MINERAL

CHARACTERISTICS

ILLITE

Optical properties: *Because of interference from other grains of the aggregates anisotropy was not generally evident.*

Grain size and shape: *<1-2 μ tabular crystals in aggregates.*

Color: *dark brown to white.*

Polishing characteristics: *does not polish-leaves a depressed irregular surface.*

Staining properties: *ineffective at magnifications used.*

Difficult identifications: *when finely dispersed in coal, especially in layers with great diversity of components.*

Misidentifications: *with kaolinite-rare in low ash coals and common in high ash coals; with the maceral micrinite- only in 1.30 float fractions.*

KAOLINITE

Optical properties: *near-to-perfect match with index of refraction of immersion oil; anisotropy not evident in small aggregates of grains, but easily noted in kaolinite "worms".*

Grain size and shape: *rounded to irregular aggregates of interlocking grains.*

Color: *flat black, surface just visible.*

Polishing characteristics: *polishes poorly, leaves a raised, flat, rough surface.*

Staining properties: *ineffective at magnifications used.*

Difficult identifications: *when included dispersed or as grains in high illite particles, especially refuse or 1.80 sink grains; when present in fusinite or semi-fusinite cell cavities.*

Misidentifications: *with illite common in high illite fractions; with quartz common in all float-sink fractions.*

QUARTZ

Optical properties: *perfect match with index of refraction of immersion oil making grains visible only by internal reflections; anisotropy visible by internal reflections.*

Grain size and shape: *rounded, oval shaped singular grains 2-20 μ in size.*

Hardness: *very hard with great relief.*

Polishing characteristics: *polishes well with a raised smooth surface.*

Difficult identifications: *when present as <10 μ grains in clay-rich particles; when present in fusinite and semi-fusinite cavities.*

Misidentifications: *with kaolinite common in all fractions; Feldspars present, but not identified, in these coals were probably misidentified as quartz.*

TABLE B-13 (Continued)

OPTICAL CHARACTERISTICS OF MINERALS IN THE DISTRICT # 3
PITTSBURGH COAL SAMPLE

MINERAL

CHARACTERISTICS

CARBONATES

Optical properties: *anisotropic* - the index of refraction of the grains pass from below, through, then above the index of the immersion oil as the stage is turned; reflectivity equals that of vitrinite in these coals.

Grain size and shape: Interlocking aggregates of 10-100 μ crystals are common, as are isolated >500 μ crystals of calcite.

Color: *gray resembling vitrinite*.

Polishing characteristics: *polish well with smooth surfaces*.

Staining properties: *Calcite stains well in Alizarine Red in 0.2% HCl, but the 3% HCl pretreatment must be eliminated, and the treatment time must be shortened to 30 seconds. Dolomite stains well in Alizarine Red in 30% NaOH and boiled, and the 3% HCl pretreatment removes all calcite. Therefore calcite must be stained first.*

Difficult identifications: *when calcite forms the matrix within illite rich particles with closely spaced clays, quartz, and pyrite observation of the carbonate is difficult even with staining.*

Misidentifications: *without staining, the carbonate minerals present in these samples cannot be distinguished. The possible implications of calcite or dolomite misidentification with apatite has not been investigated at this writing. Misidentification with gypsum is possible, and careless observation can confuse carbonates for vitrinite.*

PYRITE

Optical properties: *Isotropic, but this is visible only on the larger grains; very high reflectivity.*

Grain size and shape: *crystals of 2 μ size form framboids, irregular to lenticular patches, and occur isolated in the coal; larger grains are irregular in shape and massive with interlocking crystals.*

Color: *brassy-yellow, metallic luster.*

Crystal habit: *cubic, octahedral, or pyritohedral in small crystals-<10 μ , and anhedral in larger masses.*

Hardness: *very hard.*

Polishing characteristics: *large grains polish well with raised smooth surfaces; small grains do not polish well, and have raised rounded crystal faces.*

Difficult identifications: *rare-small crystals may be confused with reflections from clay faces or small rutile grains.*

Misidentifications: *with marcasite possible on very small crystals-<6 μ .*

OPTICAL CHARACTERISTICS OF MINERALS IN THE DISTRICT # 3
PITTSBURGH COAL SAMPLE

MINERAL	CHARACTERISTICS
MARCASITE	<p>Optical properties: <i>Anisotropic showing colors ranging from blue to red; very high reflectivity.</i></p> <p>Grain size and shape: <i>radial growths reach 20-40μ with 10μ crystals; interlocking equidimensional crystals are 5-50μ in size.</i></p> <p>Color: <i>brassy-yellow, but whitish-yellow when compared to pyrite, metallic luster.</i></p> <p>Crystal habit: <i>generally occurs in radiating masses of crystals, either from a point or as overgrowths on a pyrite mass. Occasionally occurs in masses of interlocking equidimensional crystals.</i></p> <p>Hardness: <i>very hard; slightly less relief than on pyrite where the two occur together.</i></p> <p>Polishing characteristics: <i>polishes well with a raised smooth surface.</i></p> <p>Difficult identifications: <i>small crystals in a clay-rich matrix are difficult to identify from reflections from clay surfaces.</i></p> <p>Misidentifications: <i>with pyrite on very small crystals.</i></p>
MUSCOVITE	<p>Optical properties: <i>anisotropic, extinction parallel to the long axis of tabular grains, reflectance low, nearly equal to exinite in these samples.</i></p> <p>Grain size and shape: <i>tabular with long axis parallel to coal bedding; size ranges from 5μ to >500μ, but generally 20-40μ; striations parallel to long axis.</i></p> <p>Color: <i>dark gray with reddish-purple tint.</i></p> <p>Hardness: <i>>vitrinite.</i></p> <p>Polishing characteristics: <i>polishes well with a raised, smooth, sometimes striated surface.</i></p> <p>Difficult identifications: <i>when imbedded in clay-rich grains, especially if <10μ in size.</i></p> <p>Misidentifications: <i>commonly as kaolinite, rarely quartz, and can confuse this mineral with sporinite.</i></p>
HEMATITE	<p>Optical properties: <i>Isotropic, but exhibits blood-red color due to internal reflections under crossed polarized light.</i></p> <p>Grain size and shape: <i>greatly variable; sizes from 10 to >500μ, and occurs as irregular grains to fracture fillings.</i></p> <p>Color: <i>metallic gray with red edges in and around the grain.</i></p> <p>Hardness: <i>varies greatly.</i></p> <p>Polishing characteristics: <i>polishes well with a smooth surface.</i></p> <p>Misidentifications: <i>can confuse this mineral with vitrinite; some hematite is likely Fe-hydroxides or sulfates.</i></p>

TABLE B-13 (Continued)

OPTICAL CHARACTERISTICS OF MINERALS IN THE DISTRICT # 3
PITTSBURGH COAL SAMPLE

MINERAL

CHARACTERISTICS

RUTILE

Optical properties: *many internal reflections under cross polarized light.*

Grain size and shape: *small 2 μ grains in illite or coal.*

Color: *whitish-yellow; pearly luster.*

Difficult identifications: *when as <1 μ grains in a clay rich particles; when in coal as <1 μ grains, especially in layers with a high diversity of components.*

Misidentifications: *very small grains, <1 μ , can be confused with pyrite.*

TABLE B-14

MINERALS OF THE DISTRICT # 3 PITTSBURGH AS MEASURED BY XRPD, IR, NORMATIVE CALCULATIONS, AND OPTICAL PETROGRAPHY. XRPD ILLITE IS PRODUCED BY DIFFERENCE
CARBONATES WERE MEASURED AS A GROUP BY IR AND OPTICAL PETROGRAPHY, AND WERE SUMMED FROM CALCITE AND DOLOMITE MEASURED BY XRPD AND NORMATIVE CALCULATIONS.
XRPD AND NORMATIVE DETERMINATIONS OF PYRITE ALSO INCLUDE MARCASITE.

SAMPLE	ILLITE			KAOLINITE				QUARTZ			CARBONATES				CALCITE			DOLOMITE		PYRITE		
	17 ¹	32 ³	62 ⁴	23 ¹	24 ²	27 ³	14 ⁴	16 ¹	16 ³	12 ⁴	9 ¹	8 ²	7 ³	4 ⁴	4 ¹	6 ²	4 ³	5 ¹	3 ³	20 ¹	18 ³	6 ⁴
780037	17 ¹	32 ³	62 ⁴	23 ¹	24 ²	27 ³	14 ⁴	16 ¹	16 ³	12 ⁴	9 ¹	8 ²	7 ³	4 ⁴	4 ¹	6 ²	4 ³	5 ¹	3 ³	20 ¹	18 ³	6 ⁴
780038	15	31	62	15	9	17	6	17	16	16	8	13	9	4	3	10	7	5	2	27	30	11
780039	26	44	27	8	0	0	3	17	14	17	36	32	26	33	19	20	13	17	13	8	16	8
780040	48	39	44	6	3	3	5	24	23	40	16	21	10	6	8	15	5	8	5	1	10	5
780041	31	37	42	10	10	11	0	20	21	18	18	24	16	15	13	16	11	5	4	18	16	20
780012	22	27	64	29	32	27	21	17	17	7	8	8	5	0	4	6	3	4	2	17	14	7
780013	21	34	54	19	23	11	10	18	19	17	10	15	7	1	6	11	4	4	2	24	28	14
780014	22	31	42	11	6	12	4	19	17	26	7	15	8	7	2	11	5	5	3	28	27	18
780015	25	43	39	7	2	0	2	19	18	29	24	18	14	12	13	15	8	11	6	22	25	16
780016	29	37	48	10	11	12	0	21	22	24	16	21	13	15	12	13	10	4	3	14	14	11
780017	22	32	63	28	25	23	20	16	15	11	11	8	4	0	6	6	2	5	2	18	14	5
780018	22	47	54	20	13	9	11	18	16	18	13	15	8	8	9	10	5	4	2	24	25	8
780019	21	33	34	12	6	13	8	19	18	24	12	14	9	7	8	11	6	4	3	33	30	23
780020	20	32	28	7	0	7	5	18	19	32	20	20	4	12	16	15	0	4	4	28	28	19
780021	31	38	41	9	3	9	1	20	21	32	18	16	12	8	14	11	9	4	3	20	18	15
780023	14	34	65	28	34	36	20	16	15	8	11	8	5	0	6	6	3	5	2	24	12	5
780024	13	36	58	16	29	17	12	18	17	19	11	8	5	3	6	6	3	5	1	36	25	6
780025	11	30	48	11	15	15	12	18	21	19	13	15	8	6	9	10	6	4	3	43	31	12
780026	12	30	42	7	1	7	7	17	16	26	28	22	17	11	19	15	13	9	4	25	28	13
780027	25	31	45	7	1	13	0	18	19	23	21	17	12	11	17	11	9	4	3	24	22	15
780028	23	38	64	21	39	29	14	16	13	7	14	8	7	5	5	6	5	9	3	12	13	8
780029	30	38	75	25	29	20	13	17	14	7	10	11	4	0	5	6	3	5	2	13	19	3
780030	16	38	48	13	25	14	10	18	13	24	12	8	6	3	8	6	4	4	2	28	25	12
780031	0	27	36	9	7	10	8	15	16	31	23	20	14	9	15	14	11	8	4	23	24	13
780032	23	25	41	6	0	6	0	14	18	14	39	18	19	21	33	13	16	6	3	16	24	16
780022	26	35	58	10	7	11	1	18	19	15	10	19	14	12	2	13	1	8	3	8	16	12
780036	24	32	63	19	22	30	9	19	16	11	13	15	9	2	9	6	6	4	3	20	20	9
780043	17	82	54	12	20	0	8	17	5	11	11	14	8	8	7	9	6	4	3	28	17	11
780035	14	25	49	8	11	17	1	19	25	20	19	22	13	9	15	14	10	4	3	23	19	17

¹=X-ray powder diffraction ²=Infrared spectroscopy ³=Normative calculations ⁴=Optical petrography

TABLE B-15

STANDARDIZATION EQUATIONS FOR XRPD AND IR SPECTROSCOPY OF MINERALS IN THE DISTRICT # 3 PITTSBURGH COAL. R^2 VALUES SHOW GOOD FITS OF THE DATA TO THE STANDARD CURVES, EXCEPT FOR XRPD DETERMINED QUARTZ. DETECTABLE LIMITS AND ERRORS WERE INTERPOLATED FROM PLOTS OF THE VALUES.

Linear Regression Equation		R^2	Detectable Limit	Error*
KAOLINITE (wt.%)	= $1.07(\%nc^\dagger)+2$	0.62	6%	$\pm 10\%$
	= $268(Abs^\nabla)-20$	0.58	10%	$\pm 15\%$
QUARTZ (wt.%)	= $0.23(\%nc)+8$	0.34	10%	$\pm 6\%$
PYRITE (wt.%)	= $2.19(\%nc)-11$	0.72	10%	$\pm 10\%$
CARBONATES (wt.%)	= $163(Abs)+8$	0.77	8%	$\pm 10\%$
CALCITE (wt.%)	= $0.67(\%nc)+2$	0.74	5%	$\pm 10\%$
	= $252(Abs)+6$	0.75	6%	$\pm 8\%$
DOLOMITE (wt.%)	= $1.11(\%nc)+2$	0.83	5%	$\pm 3\%$
FELDSPARS [¶] (wt.%)	= $0.25(\%nc)$	?	?	?
APATITE [¶] (wt.%)	= $1.0(\%nc)$	?	?	?
BASSANITE [¶] (wt.%)	= $1.0(\%nc)$	?	?	?

* The error given is the range on either side of the regression equation containing 90% of the samples.

[†] %nc = Percent-of-net-counts.

[∇] Abs = Infrared absorbance.

[¶] Standardization equations for feldspars and bassanite were adopted and modified from Renton⁴.

? R^2 , detectable limit, and error are unknown for feldspars, apatite, and bassanite.

TABLE B-16

X-RAY POWDER DIFFRACTION QUALIFICATION AND QUANTIFICATION
LINES DESIGNATED BY "d" SPACING, AND INFRARED SPECTROSCOPY
RECIPROCAL WAVENUMBERS OF QUALITATIVE AND QUANTITATIVE
ADSORPTION BANDS OF MINERALS IN THE DISTRICT # 3 PITTSBURGH
COAL

<i>MINERAL</i>	<i>X-ray Powder Diffraction</i>	<i>Infrared Spectroscopy</i>
ILLITE	(10Å)	
KAOLINITE	[7.1Å]	[910 cm ⁻¹]
QUARTZ	[3.34Å]	
FELDSPARS	(3.2Å)	
CARBONATES [†]		[1430 cm ⁻¹]
CALCITE	[3.04Å]	[871 cm ⁻¹]
DOLOMITE	[2.90Å]	
BASSANITE	(3.00Å)	
GYPSUM	7.56Å	3540, 3400, 1680, 1620, 660, 594 cm ⁻¹
IRON DISULFIDES [‡]	[2.71Å]	
APATITE	(2.78Å)	

[] = peak used for quantification.

() = peak used for semiquantification.

Note: peaks with no enclosures used for qualification.

† = the carbonate group was measured by IR.

‡ = XRPD measured both pyrite and marcasite.

FIGURE B-1

FIGURE B-2

FIGURE B-3

FIGURE B-4

PITTSBURGH RAW COAL (CUMULATIVE LOGARITHMIC PLOT OF SCREEN ANALYSIS)

Figure B-5

-MACERAL FREQUENCY DISTRIBUTIONS^a OF THE PITTSBURGH COAL

^a FREQUENCY DISTRIBUTIONS OF MACERAL GROUPS, MACERALS, AND SUBMACERALS OCCURRING IN THE PITTSBURGH COAL FLOAT-SINK FRACTIONS AND HEAD SAMPLES.

DISTRICT 4 COAL PREPARATION PLANT

EASTERN OHIO

DISTRICT 4 COAL PREPARATION PLANT
EASTERN OHIO

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TABLE B-17

PITTSBURGH COAL
DISTRICT 4 COAL PREPARATION PLANT

	<u>Chemical Analysis</u>							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na	K
Run-of-Mine Coal	44.70	26.70	10.40	1.11	1.49	0.87	0.45	2.00
Clean Coal	38.50	25.90	26.30	0.90	2.03	0.71	0.39	1.15
Refuse	46.10	26.70	6.98	1.19	1.53	0.74	0.43	2.25

TABLE B-18

PITTSBURGH COAL
DISTRICT 4 COAL PREPARATION PLANT

	<u>Proximate Analyses</u>					
	Moisture	Ash	Sulfur	Btu	VM	FC
Run-of-Mine Coal	2.1	31.8	4.52	9,542	31.6	34.5
	0.0	32.5	4.62	9,747	32.3	35.2
Clean Coal	2.1	10.3	4.51	13,051	41.0	46.6
	0.0	10.5	4.61	13,332	41.9	47.6
Refuse	2.2	72.3	5.26	2,275	14.4	11.1
	0.0	73.9	5.38	2,331	14.8	11.3

TABLE B-19

PITTSBURGH COAL
DISTRICT 4 COAL PREPARATION PLANT

	<u>Sulfur Forms</u>				
	<u>Total Sulfur</u>	<u>Sulfate Sulfur</u>	<u>Pyritic Sulfur</u>	<u>Total Inorganic Sulfur</u>	<u>Organic Sulfur</u>
Run-of-Mine Coal	4.52	0.04	2.75	2.79	1.73
	4.62	0.04	2.81	2.85	1.77
Clean Coal	4.51	0.05	2.03	2.08	2.43
	4.61	0.05	2.07	2.12	2.49
Refuse	5.26	0.06	4.55	4.61	0.65
	5.38	0.06	4.65	4.71	0.67

TABLE B-20

PITTSBURGH COAL
DISTRICT 4 COAL PREPARATION PLANT

	<u>Ultimate Analysis</u>			
	<u>C %</u>	<u>H %</u>	<u>O %</u>	<u>N %</u>
Run-of-Mine Coal	51.84	3.93	4.78	0.94
	52.95	3.77	2.97	0.96
Clean Coal	70.59	4.96	6.37	1.17
	72.10	4.59	4.59	1.20
Refuse	14.66	1.51	3.79	0.28
	14.99	1.89	1.89	0.29

TABLE B-21

X-RAY POWDER DIFFRACTION ANALYSES OF DISTRICT 4
COAL PREPARATION PLANT SAMPLES

Sample	¹ ILLITE	² KAOLINITE	³ QUARTZ	⁴ CALCITE	⁵ PYRITE	⁶ FELDSPARS	⁷ PYRRHOTITE	⁸ HEMATITE	⁹ Undefined Ca-Al mineral(s)
Pittsburgh #8 Feed Coal	42%	8%	26%	3%	18%	P ¹⁰	—	—	—
Pittsburgh #8 Cleaned Coal	9%	12%	20%	6%	45%	P	—	—	—
Pittsburgh #8 Refuse	50%	7%	26%	4%	10%	P	—	—	—
Pittsburgh #8 Slurry Fines	24%	9%	19%	19%	25%	P	—	—	—

¹

Illite abundances were determined by difference and the error is unknown.

²Kaolinite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.³Quartz abundances are presented as weight percent of the low temperature ash and have an error of $\pm 6\%$.⁴Calcite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.⁵Pyrite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.⁶

Presence of Feldspar minerals in the low temperature ash is indicated by a "P".

⁷

Presence of Pyrrhotite in the low temperature ash is indicated by a "P".

⁸

Presence of Hematite in the material is indicated by a "P".

⁹

Presence of undefined Ca-Al minerals in the material is indicated by a "P".

¹⁰

A "P" indicated the presence of the mineral, but quantitative values were unobtainable.

FIGURE B-6

FIGURE B-8

FIGURE B-9

FLUIDIZED BED COMBUSTOR FACILITY

RIVESVILLE, WEST VIRGINIA

FLUIDIZED BED COMBUSTOR FACILITY
RIVESVILLE, WEST VIRGINIA

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TABLE B-22

FLUIDIZED BED COMBUSTOR FACILITY

	<u>Chemical Analyses</u>							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na	K
Feed Coal	45.50	25.90	21.80	0.89	1.62	0.80	0.26	1.75
Bed Ash	24.10	8.31	2.12	0.34	37.20	1.52	0.19	0.65
Fly Ash	30.10	13.40	9.07	0.46	38.70	1.24	0.23	0.90

TABLE B-23

FLUIDIZED BED COMBUSTOR FACILITY

	<u>Proximate Analyses</u>					
	Moisture	Ash	Sulfur	Btu	VH	FC
Feed Coal	1.9	16.3	3.41	12,216	31.1	50.7
	0.0	16.6	3.48	12,453	31.7	51.7
Bed Ash	Nil	96.3	8.01	-	3.0	0.7
		96.3	8.01	-	3.0	0.7
Fly Ash	0.3	62.5	3.82	-	7.8	28.9
	0.0	63.0	3.85	-	7.9	29.1

TABLE B-24

FLUIDIZED BED COMBUSTOR FACILITY

	<u>Sulfur Forms</u>				
	<u>Total Sulfur</u>	<u>Sulfate Sulfur</u>	<u>Pyritic Sulfur</u>	<u>Total Inorganic Sulfur</u>	<u>Organic Sulfur</u>
Feed Coal	3.41	0.27	1.99	2.26	1.15
	3.48	0.28	2.03	2.31	1.17
Bed Ash	8.01	7.91	0.10	8.01	Nil
	8.01	7.91	0.10	8.01	
Fly Ash	3.82	3.26	0.13	3.39	0.43
	3.85	3.29	0.13	3.42	0.43

TABLE B-25

FLUIDIZED BED COMBUSTOR FACILITY

	<u>Ultimate Analyses</u>			
	<u>C %</u>	<u>H %</u>	<u>O %</u>	<u>N %</u>
Feed Coal	67.34	4.59	5.21	1.25
	68.64	4.46	3.59	1.27
Bed Ash	1.19	0.05	2.44	0.02
	1.19	0.05	2.44	0.02
Fly Ash	30.00	0.56	1.59	0.73
	30.24	0.47	0.89	0.74

TABLE B-26

X-RAY POWDER DIFFRACTION ANALYSES OF FLUIDIZED BED
COMBUSTOR FACILITY SAMPLES

Sample	¹ ILLITE	² KAOLINITE	³ QUARTZ	⁴ CALCITE	⁵ PYRITE	⁶ FELDSPARS	⁷ PYRRHOTITE	⁸ HEMATITE	⁹ Undefined Ca-Al mineral(s)
Fluidized Bed Feed Coal	30%	17%	22%	4%	21%	P	--	--	--
Fluidized Bed Feed Limestone	--	--	--	P	--	--	--	--	--
Fluidized Bed Bottom Ash	--	--	--	--	--	--	--	P	P
Fluidized Bed Flyash	--	--	--	--	--	--	--	P	P
Fluidized Bed Flyash	--	--	--	--	--	--	--	P	P

¹
Illite abundances were determined by difference and the error is unknown.

²
Kaolinite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.

³
Quartz abundances are presented as weight percent of the low temperature ash and have an error of $\pm 6\%$.

⁴
Calcite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.

⁵
Pyrite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.

⁶
Presence of Feldspar minerals in the low temperature ash is indicated by a "P".

⁷
Presence of Pyrrhotite in the low temperature ash is indicated by a "P".

⁸
Presence of Hematite in the material is indicated by a "P".

⁹
Presence of undefined Ca-Al minerals in the material is indicated by a "P".

The Tyler Standard Screen Scale

Form No. L-4
Please mention above
when ordering

Cumulative Logarithmic Diagram of Screen Analysis on Sample of Red Ash

Name _____

Date _____

L-6

SCREEN SCALE RATIO 1.414															
Openings		Tyler Mesh	U. S. No.	Sample Weights	Per Cent	Per Cent Cumulative Weights	Sample Weights	Per Cent	Per Cent Cumulative Weights	Sample Weights	Per Cent	Per Cent Cumulative Weights	Sample Weights	Per Cent	Per Cent Cumulative Weights
Milli-meters	Inches														
11.20				716	10.1	1.3									
5.60				35	32.8	3.6									
2.80				7	88.9	11.6									
1.40				14	335.9	43.4									
.710				25	248.3	32.0									
.355				45	43.4	5.6									
.180				80	8.5	1.1									
.090				170	4.3	0.6									
.045				325	2.5	0.3									
				325	3.5	0.5									
Totals				774.4	100.0	100.0

The Tyler Standard Screen Scale

Form No. L-6
Please mention above
when ordering

Cumulative Logarithmic Diagram of Screen Analysis on Sample of Flyash

Name _____

Date _____

L-6

SCREEN SCALE RATIO 1.414															
Openings		Tyler Mesh	U. S. No.	Sample Weights	Per Cent	Per Cent Cumulative Weights	Sample Weights	Per Cent	Per Cent Cumulative Weights	Sample Weights	Per Cent	Per Cent Cumulative Weights			
Milli-meters	Inches														
.710		25		0.5	0.2	0.2									
.355		45		21.2	8.8	9.0									
.250		60		22.0	9.2	18.2									
.180		80		25.1	10.5	28.7									
.125		120		31.6	13.2	41.9									
.090		170		16.0	6.7	48.6									
.063		280		24.6	10.3	58.9									
.045		325		11.6	4.8	63.7									
Pass		-325		87.1	36.3	100.0									
Totals				238.7	100.0	100.0

FIGURE B-14

FIGURE B-15

FIGURE B-16

SOLVENT REFINED COAL II FACILITY

FORT LEWIS, WASHINGTON

SOLVENT REFINED COAL FACILITY
FORT LEWIS, WASHINGTON

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TABLE B-27

SOLVENT REFINED COAL II FACILITY

	<u>Chemical Analyses</u>							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na	K
Feed Coal	39.00	24.30	30.40	0.84	2.03	0.74	0.40	1.15
Vacuum Bottoms	41.10	20.80	37.10	0.99	2.50	0.80	0.43	1.44

TABLE B-28

SOLVENT REFINED COAL II FACILITY

	<u>Proximate Analyses</u>						
	Moisture	Ash	Sulfur	Btu	VM	FC	FSI
Feed Coal	2.6	9.8	4.31	12,990	41.2	46.4	-
	0.0	10.1	4.43	13,337	42.3	47.6	
Vacuum Bottoms	0.1	18.4	2.81	13,034	37.9	43.6	81.5
	0.0	18.4	2.81	13,047	37.9	43.7	81.6

TABLE B-29

SOLVENT REFINED COAL II FACILITY

	<u>Sulfur Forms</u>				
	<u>Total Sulfur</u>	<u>Sulfate Sulfur</u>	<u>Pyritic Sulfur</u>	<u>Total Inorganic Sulfur</u>	<u>Organic Sulfur</u>
Feed Coal	4.31	0.04	2.03	2.07	2.24
	4.43	0.04	2.08	2.12	2.31
Vacuum Bottoms	5.26	0.06	4.55	4.61	0.65
	5.38	0.06	4.65	4.71	0.67

TABLE B-30

SOLVENT REFINED COAL II FACILITY

	<u>Ultimate Analyses</u>			
	<u>C %</u>	<u>H %</u>	<u>O %</u>	<u>N %</u>
Feed Coal	70.93	5.22	6.00	1.14
	72.82	5.06	3.78	1.17
Vacuum Bottoms	14.66	1.51	3.79	0.28
	14.99	1.89	1.89	0.29

TABLE B-31

X-RAY POWDER DIFFRACTION ANALYSES OF SOLVENT
REFINED COAL II FACILITY SAMPLES

Sample	¹ ILLITE	² KAOLINITE	³ QUARTZ	⁴ CALCITE	⁵ PYRITE	⁶ FELDSPARS	⁷ PYRRHOTITE	⁸ HEMATITE	⁹ Undefined Ca-Al mineral(s)
SRC II Feed Coal	8%	16%	19%	6%	44%	p ¹⁰	--	--	--
SRC II Vacuum Bottoms	P	7%	17%	10%	0%	--	P	--	--
SRC II Vacuum Bottoms	P	15%	18%	9%	0%	--	P	--	--

¹

Illite abundances were determined by difference and the error is unknown.

²

Kaolinite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.

³

Quartz abundances are presented as weight percent of the low temperature ash and have an error of $\pm 6\%$.

⁴

Calcite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.

⁵

Pyrite abundances are presented as weight percent of the low temperature ash and have an error of $\pm 10\%$.

⁶

Presence of Feldspar minerals in the low temperature ash is indicated by a "P".

⁷

Presence of Pyrrhotite in the low temperature ash is indicated by a "P".

⁸

Presence of Hematite in the material is indicated by a "P".

⁹

Presence of undefined Ca-Al minerals in the material is indicated by a "P".

¹⁰

"--" indicates the mineral was absent from the sample.

FIGURE B-17

FIGURE B-18