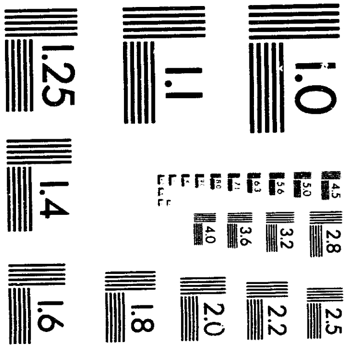
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FUNDAMENTAL STUDY OF ASH FORMATION AND DEPOSITION: EFFECT OF REDUCING STOICHIOMETRY

Quarterly Report No. 3
for the Period
1 October 1993 - 31 December 1993

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SECTION 1

EXECUTIVE SUMMARY

1. EXECUTIVE SUMMARY

The technical objectives of this project are:

a: To identify the partitioning of inorganic coal constituents among vapor, submicron fume, and fly ash products generated during the combustion of pulverized coal under a variety of combustion conditions. Fuel lean and fuel rich combustion conditions will be considered.

b: To identify and quantify the fundamental processes by which the transformations of minerals and organically-associated inorganic species occurs. Emphasis will be placed on identifying any changes that occur as a result of combustion under sub-stoichiometric combustion conditions.

c: To incorporate the effects of combustion stoichiometry into an Engineering Model for Ash Formation based upon the understanding developed in (a) and (b). When completed, this model will predict the particle size and chemical composition distributions of ash formed during the combustion of pulverized coal under a broad range of conditions.

A description of the work plan for accomplishing these objectives is presented in Section 2 of this report.

The work discussed in this report highlights the accomplishments of the third quarter of this 2-year project. This includes characterization of the program coals by PSI PowerServe, CCSEM and Mössbauer analysis of the four program coals by the University of Kentucky, completion of reactor preparation and characterization by the Massachusetts Institute of Technology, a summary of equipment modifications being undertaken by the University of Arizona, and a description of the results of initial experiments designed to measure ash particle stickiness under conditions of overall reducing stoichiometry (incomplete combustion) by PSI PowerServe.

Specifically, in Section 3 of this report we report ultimate, proximate, and ash chemical analyses for the four coals selected for this project: a run-of-mine Pittsburgh #8 bituminous coal, a washed Pittsburgh #8 bituminous coal (representative of a commercial Pittsburgh #8) obtained from DOE/PETC, a physically beneficiated Pittsburgh #8 bituminous coal, and a Black Thunder sub-bituminous coal from the Powder River Basin. CCSEM analyses identifying the included and excluded mineral content of the Black Thunder and run-of-mine Pittsburgh #8 bituminous coals are also discussed in this Section.

In Section 4, the results of CCSEM and Mössbauer characterization of the four program coals are reported by the University of Kentucky. Quartz and aluminosilicates were the principal minerals present in the Black Thunder coal, whereas quartz, potassium aluminosilicate (illite), and other silicates comprised a significant percentage of the mineral matter in the run-of-mine Pittsburgh #8 coal. Pyrite was the dominant mineral in the washed Pittsburgh #8, whereas pyrite and silicates dominated the beneficiated Pittsburgh #8. Mössbauer spectroscopy indicated that the majority of the iron was in the pyrite phase for all coals except the beneficiated product, in which partially or fully oxidized states of iron were in the majority. Mineral size distributions measured by CCSEM were finest for the beneficiated coal; the washed DOE Pittsburgh #8 had the largest mineral size distribution.

Section 5 describes the results of calibration measurements for the gas and particle feed systems as reported by MIT. This recalibration was deemed necessary because the reactor had not been operated in approximately 2 years. Recalibration is now complete and experimental work has begun as described in Section 5.

In Section 6, recent equipment modifications at the University of Arizona are discussed. A brief description of experimental plans for the following quarter is also provided.

In Section 7, the completion of temperature profiling of the PSI PowerServe reactor facility is described. The results of initial combustion experiments focusing on ash deposition as a function of char particle burnout are also presented. Because the molten intermediate products produced from the oxidation of pyrite have a high tendency to deposit, it was hypothesized that prolonging the duration of reducing combustion conditions might increase the amount of pyrite depositing. A countering effect, however, may be provided by the presence of carbon in partially burned samples. This is seen in the results presented in Section 7. Under conditions of inertial impaction, a transition was observed from low sticking at high carbon content, to high sticking at moderate carbon content, to lower sticking efficiency as complete burnout is approached. Carbon effects were believed secondary to the effect of residence time (burnout) on pyrite conversion, however; this will be explored in more detail in the coming quarter.

SECTION 2

OVERVIEW OF PROGRAM TASKS

2. OVERVIEW OF PROGRAM TASKS

This project is designed to examine the effects of combustion stoichiometry on the fundamental aspects of ash formation and ash deposit initiation. Emphasis will be placed on reducing stoichiometries associated with low-NOx combustion, although a range of oxidant/fuel ratios will be considered. Previous work has demonstrated that ash formation depends strongly upon coal mineralogy, including mineral type, size, amount, and the presence of organically associated inorganic species. Combustion temperature and the oxidation state of iron will also play a significant role. As these latter items will vary with changes in stoichiometry, research to determine the net effect on deposition is required.

To achieve these goals, a research program with the following technical objectives will be pursued:

(1) identify the partitioning of inorganic coal constituents among vapor, submicron fume, and fly ash products generated from the combustion of pulverized coal under a variety of combustion stoichiometries. Fuel-lean and fuel-rich combustion conditions will be considered.

(2) identify and quantify the fundamental processes by which the transformation of minerals and organically-associated inorganic species occurs. Identify any differences from standard pulverized coal combustion conditions.

(3) modify, to incorporate the effects of combustion stoichiometry and based on the understanding developed in (1) and (2) above, an Engineering Model for Ash Formation. The previously developed model is capable of predicting the size and chemical composition distributions of the final ash products under standard pulverized coal combustion conditions of 20% excess air. These modifications will extend the model to include phenomena that may be dominant under a broad range of stoichiometries.

Experiments, sample analyses, and modeling will be conducted at several facilities as part of this program. Detailed coal and ash sample analysis using Mössbauer spectroscopy, X-ray absorption fine structure spectroscopy (XAFS), and computer controlled scanning electron microscopy will be carried out at the University of Kentucky (UKy). Small-scale drop tube combustion tests using size and density classified coal samples and possibly synthetic char samples will be carried out at the Massachusetts Institute of Technology (MIT) to determine the extent of mineral coalescence and inorganic vaporization as a function of combustion stoichiometry. Combustion experiments utilizing utility grind coals will be conducted at PSI to examine the effects of stoichiometry on mineral interactions. Deposition experiments using ash generated from combustion experiments and using pure minerals will also be conducted to investigate deposit initiation as a function of combustion conditions. The Engineering Model for Ash Formation (EMAF) will be modified to include effects of combustion stoichiometry as part of this effort. Self-sustained pilot scale combustion experiments will be conducted in the University of Arizona (UA) 100,000 Btu/h facility to address issues of scaling in combustion processes. The interaction of iron with aluminosilicates as a function of changing combustion conditions will be the focus of this effort. Modeling of the iron-aluminosilicate interaction process will be conducted as part of the UA study. Finally, interaction with an integrated program led by the utility PowerGen will be used to address issues of deposit formation at full scale for comparison with the bench and laboratory scale results of this program. A work breakdown structure containing a brief description of each task follows. The relationship among the participants is illustrated schematically in Figure 2-1.

Task 1 - Program Planning, Management, Reporting, and Peer Review

This task, to be performed by PSI PowerServe, consists of: (1) preparing and annually updating a Program Plan; (2) coordinating, managing, and integrating the subcontracts and project results; (3) preparing project monthly reports; (4) integrating and preparing project quarterly reports; (5) integrating and preparing the program Final Report; (6) conducting annual peer review and project coordination meetings with the project Principal Investigators, either as a separate meeting or in conjunction with a technical conference, and including when appropriate the Principal Investigators of other DOE-supported ash formation research programs identified by the DOE Project Manager; and (8) acquiring and distributing coals to all of the project Principal Investigators.

Task 2 - Fundamental Study of Ash Formation and Deposit Initiation Under Reducing Conditions (PSI PowerServe)

PSI will study the effect of combustion stoichiometry on mineral matter and inorganic species transformations with an emphasis on sub-stoichiometric combustion. This will occur through a series of experiments to be conducted in a well-characterized laboratory flow reactor on a minimum of two coals. Coal blends may also be examined if of benefit to the program and of mutual interest to PSI and DOE/PETC. One or more of the coals studied in this task may be coals previously studied at PSI under DOE contract number DE-AC22-86PC90751 to permit a direct comparison with results obtained under oxidizing conditions. Model mineral compounds will also be examined as necessary to identify the importance of the oxidation state of iron in determining mineral coalescence and ash deposition. Selection of any coal samples will be coordinated with the DOE/PETC program manager.

PSI will also conduct limited experiments to determine the effect of pyrite weathering on the evolution of an iron oxide fume. These experiments will be coordinated with a similar study at Sandia National Laboratories.

PSI will also conduct in-situ combustion experiments in conjunction with the University of Kentucky and Brookhaven National Laboratories using an in-situ combustion reactor at beamline X19-A of the National Synchrotron Light Source.

Activities under this task will include collection and examination of ash samples at varying combustion temperatures and/or oxidant concentrations. Ash samples will be collected using extractive sampling. In-situ deposit collection techniques will be used to measure ash particle collection efficiencies as a function of temperature and stoichiometric ratio. Extracted ash and deposit samples will be characterized as necessary to determine particle size and chemical composition distributions as well as the chemical state of key components such as iron and calcium.

Task 3 - PowerGen Collaboration (PSI)

In order to understand the effects of combustion scale on ash formation and deposition under reducing conditions, PSI will interact with a government and industrial program currently underway in Great Britain. Led by the utility PowerGen, this program includes experiments at scales ranging from fundamental bench reactors to operating power plants. As part of a collaborative effort, PSI hopes to conduct laboratory experiments either at PSI, at BNL on the in-situ XAFS combustor, or in both reactors under a range of combustion conditions using a coal provided by PowerGen. If deemed appropriate by PSI, UA, and the DOE/PETC Program Manager, UA may also conduct combustion experiments with one of these coals. These experiments will permit direct comparison of results

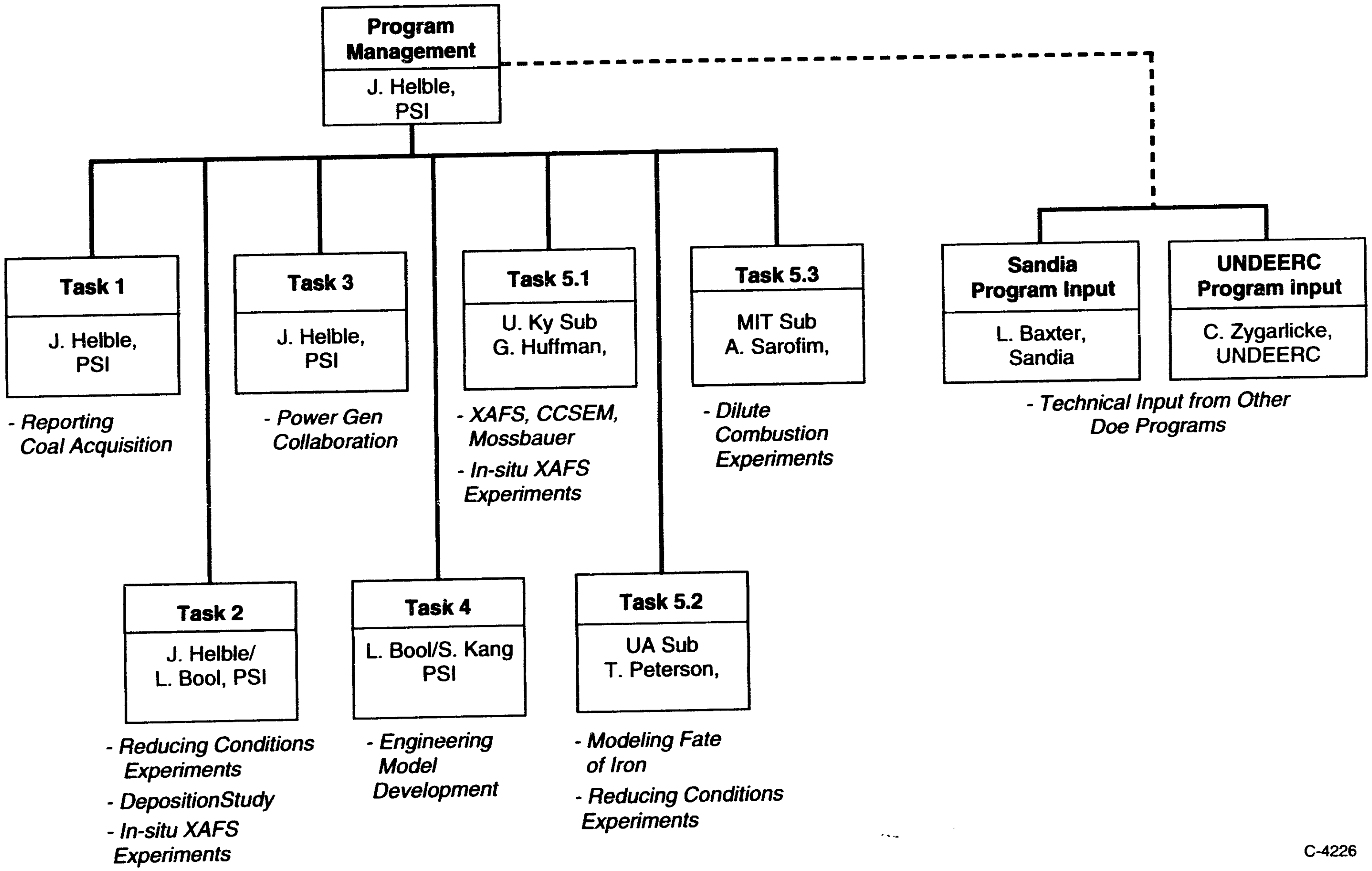


Figure 2-1. Program Structure

obtained in this project with those obtained at full scale in the PowerGen program. This interchange will also add to the database used in the modifications of the Engineering Model for Ash Formation described under Task 4.

Task 4 - Engineering Model for Ash Formation (PSI)

The results obtained by the organizations participating in this project will be applied by PSI to revise, test, and validate the Engineering Model for Ash Formation (EMAF) developed previously. The work on this subtask includes analyzing the experimental results and identifying data and technical information related to extending EMAF. Specific areas of interest include the formation of ash under reducing stoichiometries, the formation (particularly the chemical composition) of the submicron ash fume, and the influence of char fragmentation on ash formation. As part of this task, PSI will also coordinate modeling activities to be conducted by the MIT and UA subcontractors.

Task 5.1 - Advanced Techniques for Coal and Ash Characterization Including In-situ XAFS Measurements (UKy)

The University of Kentucky (UKy) will apply advanced analytical techniques such as computer-controlled scanning electron microscopy (CCSEM), Mössbauer spectroscopy, and X-ray absorption fine structure spectroscopy (XAFS) to characterize coal and ash samples from this program as needed by the individual Principal Investigators. If agreed upon by PSI, UKy, and the DOE Program Manager, UKy will also analyze selected coal and/or ash samples obtained from the PowerGen program in Great Britain.

As part of this task, UKy will conduct dynamic in-situ XAFS experiments at the Brookhaven National Laboratories (BNL) National Synchrotron Light Source (NSLS) with PSI and BNL using an in-situ drop tube furnace previously constructed by PSI. Static in-situ measurements at elevated temperatures will also be conducted using a cell previously built by UKy. The in-situ measurements will be used to investigate phenomena such as the transformation of pyrite under various conditions, the formation of iron aluminosilicate compositions as a function of combustion conditions, the formation of calcium aluminosilicate ash compositions as a function of combustion conditions, and the composition and oxidation state of key components in sticky, depositing ash particles as a function of combustion conditions.

Task 5.2 - Pulverized Coal Combustion Studies Under Reducing Conditions in a Continuous, Self-sustained Laboratory Scale Reactor (UA)

UA will conduct experiments to assess the interaction of iron and aluminosilicate minerals under reducing combustion conditions. UA will also examine the amount and chemical composition of the submicron fume generated under reducing conditions. This study will be conducted in the UA self-sustained 100,000 Btu/hr combustion facility. UA will conduct this study using at least one of the coals studied by PSI and MIT under this program, and including at least one coal studied previously under oxidizing conditions. As part of the experimental effort, UA will conduct Auger analyses for samples provided by other Principal Investigators on an as-needed basis. UA will also conduct Mössbauer analysis on selected ash samples generated at UA.

Based upon the results generated in this program, UA will modify the previously developed model for iron aluminosilicate formation to incorporate additional coals and/or combustion conditions.

Task 5.3 - Fundamental Study of Mineral Interactions and Ash Vaporization Under Reducing Conditions (MIT)

MIT will conduct drop tube experiments to study the fundamental aspects of ash formation under sub-stoichiometric conditions using narrowly sized and density classified coal samples and/or synthetic char samples. Size segregated chemical composition measurements will be conducted to assess the release of inorganic species to the vapor phase under sub-stoichiometric conditions. MIT will also conduct fundamental coalescence experiments under reducing conditions to elucidate the mechanism and rate of interaction of key mineral pairs. MIT will assist in the EMAF model testing and validation as described under Task 4.

SECTION 3

COAL SELECTION AND ACQUISITION
(PSI PowerServe)

3. COAL SELECTION AND ACQUISITION

Four U.S. coals will be studied during this program: 1) Black Thunder sub-bituminous coal from the Powder River Basin, 2) a run-of-mine Pittsburgh #8 bituminous coal from the Appalachian Basin, 3) a washed commercial version of a Pittsburgh #8 coal, and 4) a physically beneficiated product produced from Pittsburgh #8 coal. One additional coal - Silverdale bituminous coal - may be obtained from Great Britain as part of a collaborative effort with a program led by the utility Powergen (Task 3). The preliminary analysis of this final coal will be reported in the next Quarterly Report.

The Black Thunder sub-bituminous coal was obtained from the University of North Dakota Energy and Environmental Research Center (UNDEERC). Ultimate and proximate analyses for this coal are reported in Table 3-1. Ash chemical analysis is reported in Table 3-2. The results of chemical fractionation of the major inorganic species contained within Black Thunder are presented in Table 3-3. A majority of the calcium, magnesium, and sodium within the coal were extractable, suggesting that these elements are principally organically associated. Silicon, aluminum, and potassium were relatively insoluble, suggesting that these elements are principally mineral bound. Iron demonstrated a high acid-soluble fraction, suggesting that a portion of this element may be present as iron carbonate or sulfate rather than as a sulfide (pyrite) or oxide.

Table 3-1. Ultimate and Proximate Analysis of Black Thunder Sub-bituminous Coal

	As-received	Dry
Proximate (wt %)		
Moisture	24.30	-
Volatile Matter	35.89	47.42
Fixed Carbon	35.32	46.64
Ash	4.49	5.94
Ultimate (wt %)		
C	52.84	69.83
H	7.04	5.73
N	0.70	0.92
O (ind)	34.54	17.07
S	0.39	0.51
Ash	4.49	5.94
Calculated HHV, Btu/lb	9,620	

Table 3-2. Ash Chemical Analysis - Black Thunder Sub-bituminous Coal (wt% of ash)

SiO_2	32.57
Al_2O_3	16.81
Fe_2O_3	5.69
CaO	22.09
MgO	4.79
Na_2O	0.93
K_2O	0.15
TiO_2	1.11
P_2O_5	1.17
SO_3	14.69

Table 3-3. Chemical Fractionation of Black Thunder Sub-bituminous Coal (wt%, normalized to zero Si loss)

Element	Initial Conc. in coal (ppm)	Removed by H_2O	Removed by NH_4OAc	Removed by HCl	Remaining (insoluble)
Si	7750	0	0	0	100
Al	4530	0	0	20	80
Fe	2020	8	0	70	22
Ti	340	0	0	19	81
P	261	3	0	87	10
Ca	8030	1	58	39	2
Mg	1470	0	72	22	5
Na	350	22	64	10	4
K	62	15	0	0	85

The mineralogy of this coal was analyzed by CCSEM at the University of Kentucky as part of Task 5.1 of this program. Detailed results are reported in Section 4 of this report. CCSEM analysis of this coal was also performed by UNDEERC on a larger sample of Black Thunder coal. Although not acquired on the specific sample of coal used in this program, the UNDEERC results are of relevance because:

(1) Comparison with the UK results provides an indication of the potential variability in mineralogy between different samples of the same coal.

(2) The UNDEERC CCSEM results can be used to help interpret the results of UNDEERC chemical fractionation analysis of a sample of Black Thunder.

(3) UNDEERC has also provided included/excluded analysis of the Black Thunder coal. These results are only relevant to this project if the specific sample under study is similar to the larger sample analyzed at UNDEERC (i.e. item (1) above).

CCSEM analysis of the mineral matter in the "bulk" sample of Black Thunder coal as reported by UNDEERC is presented in Table 3-4. Weight percents were calculated by assuming that the cross-sectional area fraction of each mineral class was equivalent to the volume fraction, and then multiplying by the appropriate mineral density. Aluminosilicates and quartz were found to be the primary mineral phases, with pyrite concentrations relatively low. Some of the iron was identified as being in oxide or carbonate phases. Because much of the iron was acid-extracted during chemical fractionation (Table 3-3), it is likely that iron carbonate is present. The category labeled "iron oxide" in the UNDEERC CCSEM (and iron-rich in the UK CCSEM reported in Section 4) is therefore believed to be iron carbonate. A calcium aluminum phosphorus phase was also reported.

Table 3-4. CCSEM Mineral Analysis of Black Thunder Coal

Mineral	Area Percent* of Mineral Matter	Weight Percent of Mineral Matter
Quartz	25.6	24.1
Iron Oxide/Carbonate	2.4	4.6
Kaolinite	30.9	29.1
Aluminosilicate	4.9	4.6
Montmorillonite	8.0	7.1
Pyrite	2.7	4.7
Ca Al Phosphate	9.9	9.8
Unknown	7.3	7.0

* area percent = volume percent for cross-sectioned sample

These CCSEM results are similar to those reported in Section 4 by the University of Kentucky. A simplified comparison of the two analyses is presented in Table 3-5. Detailed comparison on a compositional basis is complicated by the use of different mineral categorization schemes by the two analytical groups, primarily for the aluminosilicates, as seen in the K-Al-Si ternary mineral diagrams in Figures 3-1 and 3-2. Mineral size distributions can, however, be directly compared. As seen in Figure 3-3, distributions were similar although not identical. Relative standard

Table 3-5. Comparison of Black Thunder CCSEM Mineral Data - Project Sample (UK) and Bulk Sample (UNDEERC) (results expressed in weight percent)

Mineral	Project Sample (UK)	Bulk Sample (UNDEERC)
Quartz	26.6	24.1
Iron Oxide / Carbonate	3.0	4.6
Kaolinite	14.4	29.1
Aluminosilicate	-	4.6
Montmorillonite	3.9	7.1
Misc. Silicates	23.8	0.5
Pyrite	1.9	4.7
Ca Al P	6.2	9.8
(Kaol + Al-sil + Misc. Sil)	38.2	34.2

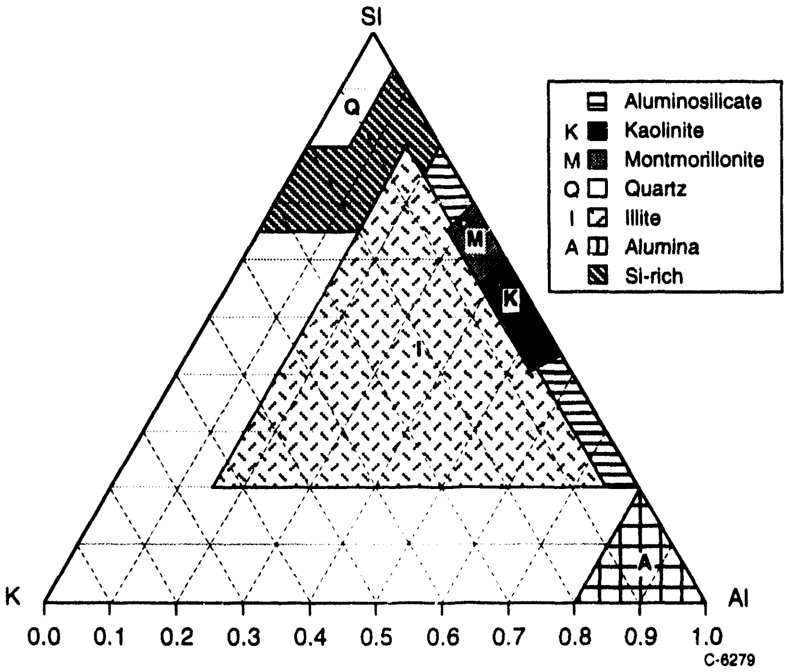


Figure 3-1. Mineral definitions for K-Al-Si mineral particles; UNDEERC/MTI classification.

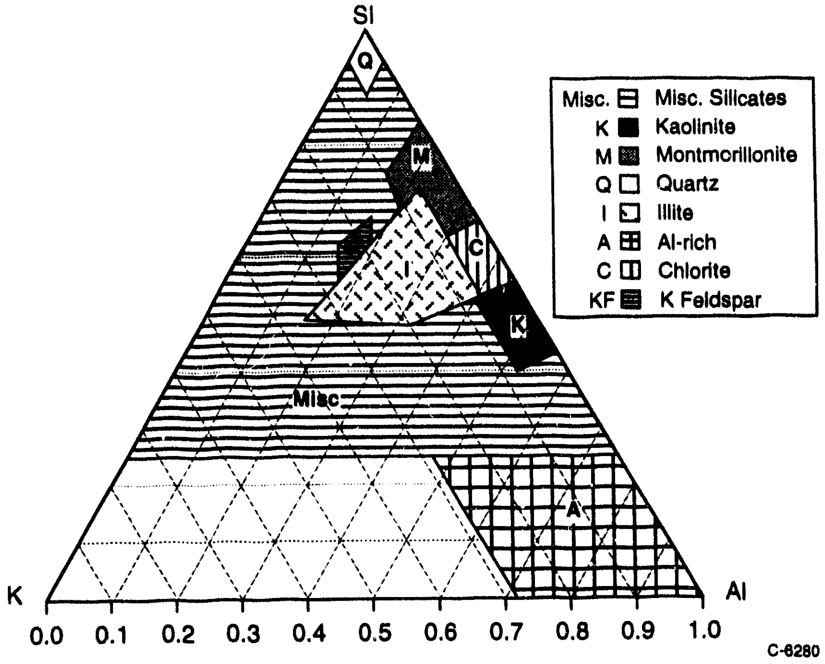


Figure 3-2. Mineral definitions for K-Al-Si mineral particles; University of Kentucky classification.

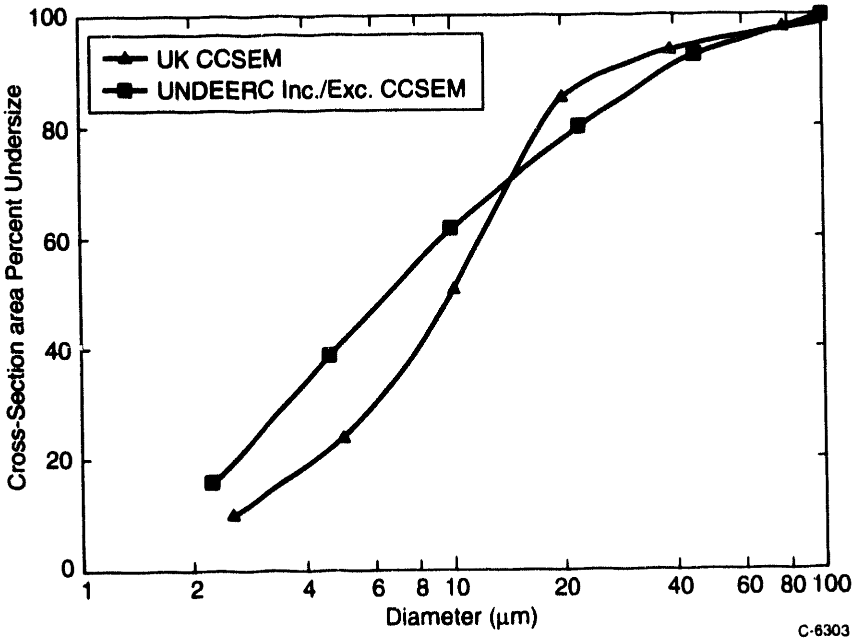


Figure 3-3. Mineral size distributions for Black Thunder coal by CCSEM analysis.

deviations of mineral weight fractions between laboratories of 20% have been reported from round-robin analyses (Casuccio et al., 1990); similar or greater uncertainty is expected with mineral size distribution measurements. It is therefore impossible to determine whether the differences observed in Figure 3-3 are the results of sample variation or simply the use of different laboratories. The overall similarity of mineral size and composition, however, suggests that the sample being studied in this program *is* similar to the larger sample examined separately at UNDEERC. Chemical fractionation data and included/excluded CCSEM data obtained on the larger sample therefore provide some indication of the mineralogy of our particular coal.

From the UNDEERC CCSEM and chemical fractionation analyses, therefore, an approximate measure of the division between mineral bound inorganic species and organically associated inorganic species can be derived. As shown in Table 3-6, CCSEM analysis indicates that mineral matter comprises 2.5 wt% of the Black Thunder coal. Since the ultimate/proximate analysis of the coal indicated a total ash content of 4.5 wt%, the organically associated content of this coal is 2 wt%. Chemical fractionation results compare favorably. If the elemental concentrations reported in Table 3-3 are converted to oxide concentrations, a total ash content of 4.3 wt% (sulfur-free basis) is obtained. This compares with a sulfur-free ash concentration of 3.85% from ultimate/proximate and ash chemical analyses. The fraction of mineral matter in the coal, based upon chemical fractionation analysis, was found to be 2.5% if only the insoluble fraction was considered, and 2.7% if the HCl-extractable iron was included in the mineral total. From these analyses, we conclude that approximately 60% of the inorganic species are mineral bound, and 40% are organically associated in the Black Thunder coal.

Table 3-6. Distribution of Inorganic Species Between Mineral and Organically Associated Phases - Black Thunder coal

Analysis	Wt% Ash	Wt% Ash (S-free)	Wt% Minerals	Wt% Organ. Assoc.
ult/prox/ash	4.5	3.85	-	-
CCSEM*	-	-	2.6	1.9
chem. fract.+	-	4.3	2.5-2.7	1.6-1.8

* percent organically associated by difference (from total percent ash)

\+ on a sulfur-free basis (sulfur not measured during chemical fractionation)

A mineral size distribution measured by CCSEM for the Black Thunder coal is given in Table 3-7. On an area percentage basis, greater than 60% of the minerals were less than 10 μm in diameter. Quartz, kaolinite, and other aluminosilicates were concentrated in these smaller particle sizes. Much of the pyrite was greater than 22 μm in size. Included/excluded CCSEM analysis of this coal is presented in Table 3-8. Approximately 43% of the mineral matter was found to be excluded from coal particles by this analysis. Chemical analysis of the major species described in Tables 3-7 and 3-8 is presented in Table 3-9. Complete summaries of the CCSEM data shown in Tables 3-7 through 3-9 are presented in Appendix A of this report.

Table 3-7. Mineral Size Distribution Analysis for Black Thunder Coal by CCSEM (basis: % of mineral area)

Mineral	1 - 2.2 μm	2.2 - 4.6 μm	4.6 - 10 μm	10 - 22 μm	22 - 46 μm	46 - 100 μm
Quartz	18	15	28	14.5	21	3.5
Iron Oxide/Carb.	9.5	13	29.5	20	28	0
Kaolinite	13	29	22	17	13	6
Aluminosilicate	5	20	10	53	2	10
Montmorillonite	6	28	9	30	16	11
Pyrite	11	0	2	47	12	29
Ca Al Phosphate	16	31	40.5	9.5	2	0
Unknown	43	14	17	7	12	7
Total	16.0	23.2	22.9	17.5	13.5	7.0

Table 3-8. Excluded Mineral Analysis of Black Thunder Coal by CCSEM

Mineral	% of individual mineral area in each size bin that is excluded						Total % of mineral excluded
	1 - 2.2 μm	2.2 - 4.6 μm	4.6 - 10 μm	10 - 22 μm	22 - 46 μm	46 - 100 μm	
Quartz	28	37	41	37	56	100	43
Iron Ox./Car.	31	30	87	0	80	0	55
Kaolinite	50	66	31	23	36	30	43
Aluminosil.	30	88	63	30	100	100	53
Montmorill.	0	27	38	92	86	100	63
Pyrite	78	0	0	69	70	100	78
Ca Al P	18	39	11	0	0	0	19
Unknown	22	22	28	0	44	100	29
Total	31	46	34	37	52	74	43

Table 3-9. Average Mineral Composition for Black Thunder Coal by CCSEM (only elements present in ≥ 0.5 atom percent quantities reported) (values may not sum to 100% because of round-off error)

Mineral	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Fe	Ba
Quartz				95		1	1		1	1	1	
Iron Ox/Car.	1	1	2	3	1	1	1	1	2	1	88	1
Kaolinite			45	48		1	1	1	1	1	1	1
Aluminosil.	1		35	56	1	1	1	1	1	1	1	1
Montmorill.			31	58		2	1	2	2	1	1	1
Pyrite			1	1		23			1	1	73	
Ca Al P	1	1	40		21		3	1	25	3	2	3
Unknown	1	2	19	18	16	7	4	4	14	4	8	4

A run-of-mine Pittsburgh #8 coal, representative of the parent of a beneficiated coal being studied here and in other DOE programs, was also obtained for study. This particular coal was provided by Kaiser Engineers, and henceforth will be referred to as either "run-of-mine (Kaiser) Pittsburgh #8" or simply "Kaiser Pittsburgh #8" to distinguish it from the other program coals. Ultimate and proximate analyses of this coal are reported in Table 3-10. Bulk ash chemical analysis of this coal is reported in Table 3-11. A coal particle size distribution, measured with a Malvern Instruments particle sizer, is reported in Table 3-12.

Table 3-10. Ultimate and Proximate Analysis of Run-of-Mine (Kaiser) Pittsburgh #8 Bituminous Coal

Proximate (wt %)	As-received	Dry
Moisture	2.00	-
Volatile Matter	30.76	31.40
Fixed Carbon	40.09	40.88
Ash	27.15	27.72
Ultimate (wt %)		
C	57.16	58.35
H	4.00	3.86
N	0.98	1.00
O (ind)	8.43	6.76
S	2.28	2.32
Ash	27.15	27.72
Calculated HHV, Btu/lb	10,040	

Table 3-11. Ash Chemical Analysis of Run-of-Mine (Kaiser) Pittsburgh #8 Bituminous Coal (wt% of ash)

SiO_2	54.82
Al_2O_3	23.06
Fe_2O_3	10.52
CaO	3.48
MgO	2.26
Na_2O	0.50
K_2O	1.74
TiO_2	0.87
P_2O_5	0.13
SO_3	2.63

Table 3-12. Coal Particle Size Distribution - Run-of-Mine (Kaiser) Pittsburgh #8

Size Microns	% Under	% in band	Size Microns	% Under	% in band	
118.4	100.0	0.0	11.1	57.1	4.5	
102.1	100.0	0.1	9.6	52.6	4.8	
88.1	99.9	0.3	8.3	47.8	5.6	
76.0	99.6	0.6	7.2	42.2	6.1	
65.6	99.0	1.0	6.2	36.1	6.2	
56.6	98.0	1.5	5.3	29.9	5.8	
48.8	96.6	2.0	4.6	24.1	5.4	
42.1	94.6	2.7	4.0	18.8	5.3	
36.3	91.9	3.4	3.4	13.4	5.0	D(v,0.5)=8.9 μm
31.3	88.5	3.9	3.0	8.5	4.0	D(v,0.9)=33.3 μm
27.0	84.7	4.0	2.6	4.5	2.6	D(v,0.1)=3.1 μm
23.3	80.7	3.9	2.2	1.9	1.0	D(4,3)=13.4 μm
20.1	76.8	4.2	1.9	0.9	0.4	D(3,2) = 7.0 μm
17.4	72.6	5.1	1.6	0.5	0.1	
15.0	67.5	5.4	1.4	0.4	0.2	
12.9	62.2	5.0	1.2	0.2		

Coal minerals were measured by CCSEM analysis at the University of Kentucky (UK), using a small sample of coal provided by PSI. Results are presented in Section 4 of this report. A portion of the PSI sample was also shipped to a commercial analytical laboratory, MTI, for included/excluded analysis. In Figure 3-4, mineral particle size distributions determined by CCSEM at MTI and UK are shown to be similar, as would be expected for two samples of the same coal. It should be noted that the sample was analyzed twice by MTI, once as standard CCSEM and once as included/excluded CCSEM. The size distribution obtained from the standard CCSEM analysis at MTI was markedly different from the included/excluded CCSEM and the UK CCSEM (Figure 3-3); potential causes of this difference are being examined. Mineral composition data for all three analyses were somewhat similar (Table 3-13), but again suffer from differences in categorization schemes. Included/excluded mineral analyses for the run-of-mine (Kaiser) Pittsburgh #8 are shown in Table 3-14, with the corresponding mineral distributions provided in Table 3-15. The complete included/excluded CCSEM analysis obtained at MTI for this coal is given in Appendix B.

A second sample of Pittsburgh #8 coal with lower ash content was obtained through the DOE Pittsburgh Energy Technology Center. This coal, representative of a commercial washed Pittsburgh #8, will be referred to as DOE Pittsburgh #8. Ultimate, proximate, and ash analyses for this coal are provided in Tables 3-16 and 3-17. A coal particle size distribution, measured with a Malvern Instruments particle sizer, is given in Table 3-18. The coal psd is similar to that of the run-of-mine (Kaiser) Pittsburgh #8 discussed previously (Table 3-12). CCSEM analysis for this coal was provided by the University of Kentucky as part of Task 5.1 and is discussed in Section 4 of this report.

For the final domestic coal being studied in this program, a beneficiated Pittsburgh #8 coal was selected. The fuel is a Southern Company agglomerate product being studied as part of the DOE-funded ABB/Combustion Engineering program "Combustion Characterization of Beneficiated Coal-based Fuels." Ultimate and proximate analyses for the specific sample being studied in this program are compared with ABB/CE analyses of the "bulk" sample and a smaller sample tested

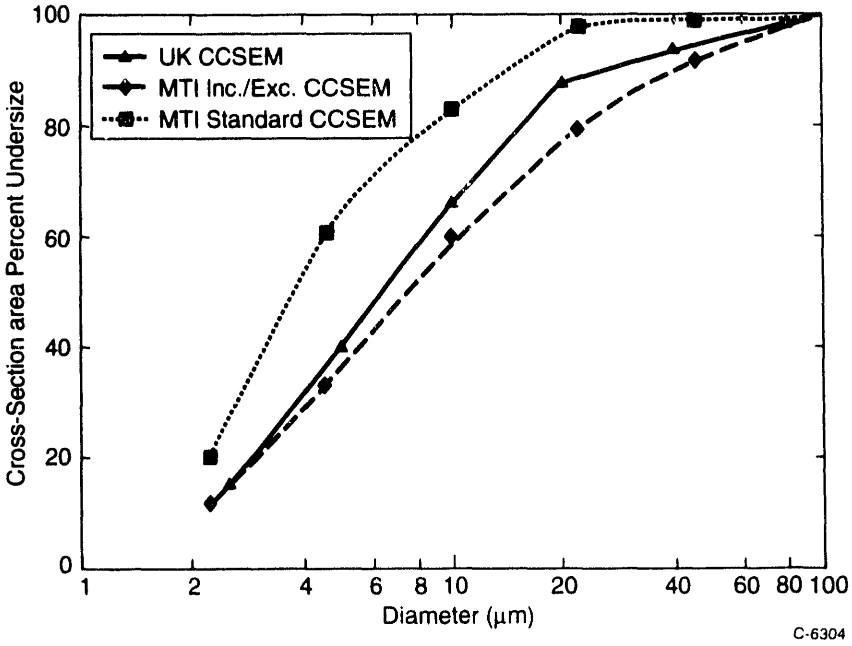


Figure 3-4. Mineral size distributions for run-of-mine Pittsburgh #8 by CCSEM analysis.

Table 3-13. Comparison of (Kaiser) Pittsburgh #8 CCSEM Mineral Data - UK and MTI (results expressed in weight percent

Mineral	UK	MTI (inc/exc analysis)	MTI (std analysis)
Quartz	21.7	16.3	18.7
Kaolinite	6.4	12.0	11.0
Montmorillonite	0.2	3.9	4.8
K-Al Silicate*	22.0	27.4	25.3
Pyrite	11.9	11.7	8.3
Misc. Silicates	22.7	10.0	11.0
(Kaol+Misc. Sil)	29.1	22.0	22.0
* "illite," with more restrictive chemical definition, in UK categorization			

Table 3-14. Excluded Mineral Analysis for Run-of-Mine (Kaiser) Pittsburgh #8 Coal by CCSEM (data in this table are ZAF corrected).

Mineral	% of individual mineral area in each size bin that is excluded						Total % of mineral excluded
	1 - 2.2 μm	2.2 - 4.6 μm	4.6 - 10 μm	10 - 22 μm	22 - 46 μm	46 - 100 μm	
Quartz	77	90	87	78	94	100	87
Kaolinite	59	68	67	65	90	100	69
Montmorill.	86	88	62	100	96	100	84
K-Al Silicate	79	85	71	84	86	100	84
Pyrite	74	63	47	91	87	100	77
Misc. Sil.	81	85	84	86	94	100	86
Unknown	79	93	69	88	90	100	85
Total	79	84	78	85	89	100	83

Table 3-15. Mineral Size Distribution Analysis (UNDEERC; part of included/excluded analysis) for (Kaiser) Pittsburgh #8 Coal by CCSEM (Basis: % of mineral area)

Mineral	1 - 2.2 μm	2.2 - 4.6 μm	4.6 - 10 μm	10 - 22 μm	22 - 46 μm	46 - 100 μm
Quartz	12.4	27.4	31.4	12.2	9.9	6.6
Kaolinite	13.4	24.2	25.7	23.2	10.5	3.0
Montmorill.	16.9	21.5	27.6	17.0	12.9	4.1
K-Al Silicate	10.2	25.8	31.9	18.6	10.2	3.3
Pyrite	5.5	20.2	19.2	13.1	24.0	18.0
Misc. Silicates	13.2	14.2	23.6	30.2	13.2	10.6
Unknown	16.5	19.3	20.8	22.7	10.8	9.8
Total	11.9	22.4	26.6	19.8	12.2	7.1

Table 3-16. Ultimate and Proximate Analysis of DOE Pittsburgh #8 Bituminous Coal

Proximate (wt %)	As-received	Dry
Moisture	1.40	-
Volatile Matter	36.85	37.36
Fixed Carbon	54.27	55.06
Ash	7.48	7.58
Ultimate (wt %)		
C	77.05	78.11
H	4.79	4.70
N	1.39	1.41
O (ind)	7.40	6.27
S	1.89	1.92
Ash	7.48	7.58
Calculated HHV, Btu/lb	13,560	

Table 3-17. Ash Chemical Analysis of DOE Pittsburgh #8 Bituminous Coal (wt% of ash)

SiO_2	46.81
Al_2O_3	25.03
Fe_2O_3	14.76
CaO	4.80
MgO	1.71
Na_2O	0.94
K_2O	1.02
TiO_2	0.72
P_2O_5	0.19
SO_3	4.02

Table 3-18. Coal Particle Size Distribution - Washed DOE Pittsburgh #8

Size Microns	Under	% in band	Size Microns	Under	% in band	
118.4	100.0	0.2	11.1	47.7	5.1	
102.1	99.8	0.5	9.6	42.6	5.5	
88.1	99.4	0.7	8.3	37.1	6.3	
76.0	98.6	1.0	7.2	30.8	6.1	
65.6	97.6	1.1	6.2	24.8	5.0	
56.6	96.5	1.3	5.3	19.8	3.9	
48.8	95.2	1.6	4.6	15.9	3.3	
42.1	93.6	2.3	4.0	12.7	3.3	
36.3	91.3	3.2	3.4	9.4	3.3	D(v,0.5)=11.1 μm
31.3	88.1	4.1	3.0	6.1	2.8	D(v,0.9)=34.0 μm
27.0	84.0	4.8	2.6	3.3	1.2	D(v,0.1)=3.5 μm
23.3	79.3	5.4	2.2	2.0	0.9	D(4,3)=15.3 μm
20.1	73.8	6.2	1.9	1.1	0.4	D(3,2) = 8.5 μm
17.4	61.7	7.2	1.6	0.7	0.2	
15.0	60.4	6.9	1.4	0.5	0.2	
12.9	53.5	5.8	1.2	0.3		

previously at PSI in Table 3-19. All analyses are fairly similar, with the only major difference being a high moisture content (32.5%) reported in the bulk ABB/CE sample on an as-received basis (see Quarterly Report Number 2, this project). Independent analysis of the current PSI sample in the PSI laboratory using temperatures, sample masses, atmospheric compositions, and times specified in ASTM standard procedure number D-3173 yielded moisture values of 2.0%, verifying the low moisture content of the samples being used in this program. The chemical analysis of the bulk ash in this coal, as measured by ABB/CE, is reported in Table 3-20. CCSEM analysis on our specific sample (i.e., the PSI sample) was provided by UK as part of Task 5.1 and is reported in Section 4 of this report.

Table 3-19. Ultimate and Proximate Analysis of Beneficiated Pittsburgh #8 Bituminous Coal

	This sample		ABB/CE analysis	
Proximate (wt %)	As-received	Dry	Dry (1992 PSI sample)	Dry (ABB/CE sample)
Moisture	2.00	-	-	-
Volatile Matter	36.37	37.13	39.4	35.7
Fixed Carbon	56.42	57.55	55.7	59.1
Ash	5.21	5.32	4.9	5.2
Ultimate (wt %)				
C	77.22	78.83	79.9	80.6
H	5.51	5.39	5.3	5.0
N	1.37	1.40	1.5	1.4
O (ind)*	8.98*	7.33*	6.7	6.1
S	1.71	1.74	1.7	1.8
Ash	5.21	5.32	4.9	5.1
Calculated HHV, Btu/lb	13,900	14,200	14,400	14,450

Table 3-20. Ash Chemical Analysis of Beneficiated Pittsburgh #8 Bituminous Coal (wt% of ash)

Compound	1992 PSI sample	ABB/CE sample
SiO_2	49.2	42.7
Al_2O_3	24.8	25.2
Fe_2O_3	19.0	20.8
CaO	2.4	2.8
MgO	1.2	1.0
Na_2O	0.6	0.1
K_2O	1.2	1.3
TiO_2	1.1	1.4
P_2O_5	0.5	0.4
SO_3	2.4	2.6

SECTION 4

TASK 5.1

ADVANCED TECHNIQUES FOR COAL AND ASH CHARACTERIZATION

(University of Kentucky)

4. ADVANCED TECHNIQUES FOR COAL AND ASH CHARACTERIZATION

During this quarter, we received the following four coal samples for characterization using Mössbauer spectroscopy and Computer controlled Scanning Electron Microscopy (CCSEM):

1. Pittsburgh #8 parent coal (Kaiser)
2. Pittsburgh #8 washed to reduce ash content (DOE/PETC)
3. Beneficiated Pittsburgh #8 coal (agglomerated by Southern Company Services)
4. Black Thunder coal (UNDEERC).

Ultimate, proximate, and ash analysis data for these coals (discussed in detail in Section 3 of this report) are summarized in Table 4-1.

CCSEM Analysis

Standard CCSEM samples were made by mixing coal with C-4 epoxy and pressing the mixture as a pellet. These pellets were polished and carbon coated to carry out CCSEM analysis. In the case of Black Thunder coal, moisture in the coal caused excessive bubbles and cracks in the pellet. Therefore, this coal was heated at 70°C for 12 hr and then was mixed with epoxy to make a sample pellet. All coals were analyzed at three different magnifications, totaling 1200 mineral particles per coal. Tables 4-2 through 4-5 show the results from these analyses. Mineral size distributions for the four coals are shown in Figure 4-1.

Mössbauer Analysis

Standard Mössbauer analysis was carried out on these coals to detect the distribution of forms of iron. Table 4-6 and Figures 4-2 through 4-5 show the results from Mössbauer analysis. Black Thunder had only a weak pyrite quadrupole doublet in the Mössbauer spectrum. Although examination of the spectrum suggested asymmetry (indicative of additional phases), only a single doublet, consistent with pyrite, was fit. Recall that CCSEM and chemical fractionation analyses indicated that pyrite and iron carbonate phases were present. Pittsburgh #8 coal (Kaiser run-of-mine and DOE) showed illite, siderite, and jarosite peaks. Though there is significant difference in the ash content of these coals (the washed DOE sample has 72% less ash than the run-of-mine Kaiser sample), the pyrite content is reduced by only 15%. Thus, the washing treatment to reduce ash seems to be removing the clay partings of the seam. The pyrite in the coal matrix is relatively unaffected.

We were not able to assign a specific phase to the major quadrupole doublet in the Mössbauer spectrum of SCS beneficiated Pittsburgh #8 coal. Since the coal has been treated, we were expecting some oxidation of the pyrite and the quadrupole doublet could be due to FeOOH. However, a low temperature (14 K) Mössbauer spectrum did not show any splitting of this quadrupole doublet into a six-line magnetic spectrum. A new Mössbauer spectrum acquired after washing the coal with cold water showed a substantial decrease in this quadrupole doublet. From these observations, we can conclude that this doublet could be due to a water soluble ferric sulfate phase. As shown in the figure, after washing, we can easily differentiate the contribution to the Mössbauer spectrum from both the insoluble jarosite and pyrite. Using these parameters, we could refit the original spectrum (before cold water wash) with pyrite, jarosite, and sulfate peaks. Weight percent pyrite in both samples (calculated using the area under the curve) also agree well with each other. We plan to carry out a forms-of-sulfur XANES analysis during our upcoming XAFS run at NSLS (April 1994) to confirm formation of sulfate from pyrite.

Table 4-1. Proximate, Ultimate, and Ash Analysis on Coals

	Pittsburgh #8 (Kaiser)		Pittsburgh #8 (DOE)		Pittsburgh #8 (SCS Cleaned)		Black Thunder (UNDEERC)	
	As received	Moisture free	As-received	Moisture free	As-received	Moisture free	As-received	Moisture free
Proximate Analysis								
Moisture	2.00	-	1.40	-	2.00	-	24.30	-
Volatile Matter	30.76	31.40	36.85	37.36	36.37	37.13	35.89	47.42
Fixed Carbon	40.09	40.88	54.27	55.06	56.42	57.55	35.32	46.64
Ash	27.15	27.72	7.48	7.58	5.21	5.32	4.49	5.94
Ultimate Analysis								
C	57.16	58.35	77.05	78.11	77.22	78.83	52.84	69.83
H	4.00	3.86	4.79	4.70	5.51	5.39	7.04	5.73
N	0.98	1.00	1.39	1.41	1.37	1.40	0.70	0.92
O (ind)	8.43	6.76	7.40	6.27	8.98	7.33	34.54	17.07
S	2.28	2.32	1.89	1.92	1.71	1.74	0.39	0.51
Btu/lb	10,040	--	13,560	--	13,900	--	9,620	--
Ash Analysis								
SiO_2	54.82		46.81		42.7*		32.57	
Al_2O_3	23.06		25.03		25.2		16.81	
Fe_2O_3	10.52		14.76		20.8		5.69	
CaO	3.48		4.80		2.8		22.09	
MgO	2.26		1.71		1.0		4.79	
Na_2O	0.50		0.94		0.1		0.93	
K_2O	1.74		1.02		1.3		0.15	
TiO_2	0.87		0.72		1.4		1.11	
P_2O_5	0.13		0.19		0.4		1.17	
SO_3	2.63		4.02		2.6		14.69	

*Analysis of ABB/CE bulk sample.

Table 4-2. CCSEM Analysis Results for Black Thunder Coal

```
CCSEM File:1113R6.cma
RUN        6  DATE 25-FEB-94 SUMMARY    132    TOTAL  1199

                                                  AVERAGE SPECIES COMPOSITION

   #  MINERAL SPECIES      Na   Mg   Al   Si   P    S   Cl    K   Ca   Ti   Fe     Weight %

 331  Quartz               0.   0.   0.  99.  0.   0.   0.   0.   1.   0.   0.        26.6
 260  Kaolinite            0.   0.  47.  52.  0.   0.   0.   0.   0.   0.   0.        14.4
  10  Illite               0.   0.  31.  60.  0.   1.   0.   6.   1.   0.   1.         2.4
  48  Montmorillonite      0.   0.  22.  65.  0.   2.   0.   0.   8.   1.   0.         3.9
 256  Misc. Silicates      0.   0.  21.  68.  0.   3.   0.   1.   4.   2.   0.        23.8
  27  Pyrite               0.   0.   0.   0.  0.  67.   0.   0.   0.   0.  33.         1.9
   3  Misc. sulf.          0.   0.   0.   3.  0.  55.   0.   2.   6.   9.  23.         0.1
  48  Misc. Phosphate ***  0.   0.  34.   0. 29.   0.   0.   0.  35.   1.   1.         6.2
  13  Fe-rich              0.   0.   0.   0.  0.   0.   0.   0.   0.   0.  99.         3.0
   9  Calcite              0.   0.   0.   1.  0.   0.   0.   0.  99.   0.   0.         0.2
   1  Mixed Carbonate      0.  13.   0.   0.  0.   0.   0.   0.  87.   0.   0.         0.0
   9  Ti oxide             0.   0.   0.   0.  0.   1.   0.   0.   0.  99.   0.         1.4
   6  Quartz-Sulfate       0.   0.   1.  58.  0.  26.   0.   0.  15.   0.   0.         1.0
   1  Sil-sulf             0.   0.  13.  32.  0.  38.   0.   0.  17.   0.   0.         0.0
   2  Silicate-Pyrite      0.   0.  22.  33.  0.  24.   0.   0.   0.   0.  21.         0.0
 135  Misc. Mixed          0.   0.  14.  54.  2.  11.   0.   2.  14.   1.   1.        15.2
1159  GRAND TOTALS         0.   0.  18.  63.  2.   4.   0.   1.   6.   2.   4.       100.0

*** Crandallite

                                  WEIGHT DISTRIBUTION

                                          Size Ranges (Microns)

  MINERAL SPECIES  WT. %      0.0      2.5      5.0      10.      20.      40.      80.
                              2.5      5.0     10.0      20.      40.      80.     500.


 Quartz             26.6       9.      13.      23.      39.      12.       4.       0.
 Kaolinite          14.4      23.      17.      24.      21.      12.       2.       0.
 Misc. Silicates    23.8       9.      11.      24.      48.       4.       5.       0.
 Misc. Mixed        15.2       2.       6.      42.      47.       4.       0.       0.
 MINOR MINERALS     20.1      11.      24.      24.      20.       6.       7.       9.
----------------------------------------------------------------------------------------
 GRAND TOTALS      100.0      10.      14.      26.      36.       8.       4.       2.
```

Table 4-3. CCSEM Analysis Results for Pittsburgh #8 (Kaiser) Coal

CCSEM File:1120r2.cma (repolished)
RUN 2 DATE 28-FEB-94 SUMMARY 108 TOTAL 1199

AVERAGE SPECIES COMPOSITION

#	MINERAL SPECIES	Na	Mg	Al	Si	P	S	Cl	K	Ca	Ti	Fe	Weight %
209	Quartz	0.	0.	0.	98.	0.	0.	0.	1.	0.	0.	0.	21.7
59	Kaolinite	0.	0.	47.	50.	0.	1.	0.	1.	0.	0.	0.	6.4
262	Illite	0.	0.	27.	54.	0.	2.	0.	14.	0.	1.	3.	22.0
14	K-Feldspar	0.	0.	18.	53.	0.	2.	0.	19.	0.	0.	8.	0.7
1	Chlorite	0.	0.	21.	33.	0.	0.	0.	2.	0.	2.	42.	0.1
1	Montmorillonite	9.	0.	15.	76.	0.	0.	0.	0.	0.	0.	0.	0.2
290	Misc. Silicates	0.	0.	17.	63.	0.	3.	0.	8.	1.	1.	6.	22.7
101	Pyrite	0.	0.	0.	0.	0.	65.	0.	0.	0.	0.	35.	11.9
6	Ferrous Sulfate	0.	0.	0.	3.	0.	48.	0.	1.	0.	0.	47.	1.1
10	Gypsum	0.	0.	0.	1.	0.	47.	0.	0.	52.	0.	0.	0.1
28	Misc. sulf.	0.	0.	1.	6.	0.	51.	0.	0.	7.	0.	34.	2.3
2	Apatite	0.	0.	2.	0.	29.	0.	0.	0.	70.	0.	0.	0.1
1	Fe-rich	0.	0.	0.	0.	0.	3.	0.	0.	0.	0.	97.	0.0
41	Calcite	0.	2.	0.	1.	0.	0.	0.	0.	97.	0.	0.	2.2
45	Mixed Carbonate	0.	6.	1.	8.	0.	0.	0.	1.	82.	0.	1.	3.6
2	Ti-rich	0.	0.	7.	16.	0.	0.	0.	5.	0.	70.	2.	0.3
17	Quartz-Sulfate	0.	0.	3.	53.	0.	35.	1.	0.	3.	0.	0.	0.3
4	Quartz-Pyrite	0.	0.	6.	33.	0.	36.	0.	1.	0.	0.	24.	0.4
20	Sil-sulf	0.	0.	23.	42.	0.	30.	0.	3.	0.	0.	1.	0.8
8	Silicate-Pyrite	0.	0.	16.	37.	0.	26.	0.	4.	0.	0.	17.	0.3
51	Misc. Mixed	1.	1.	13.	42.	1.	6.	0.	7.	24.	0.	3.	2.8
1173	GRAND TOTALS	0.	0.	14.	54.	0.	11.	0.	5.	6.	1.	8.	100.0

WEIGHT DISTRIBUTION

Size Ranges (Microns)

MINERAL SPECIES	WT. %	0.0 2.5	2.5 5.0	5.0 10.0	10. 20.	20. 40.	40. 80.	80. 500.
Quartz	21.7	9.	26.	27.	31.	2.	4.	1.
Illite	22.0	23.	36.	23.	11.	5.	3.	0.
Misc. Silicates	22.7	19.	29.	30.	10.	6.	4.	2.
Pyrite	11.9	0.	0.	26.	43.	15.	15.	0.
MINOR MINERALS	21.7	17.	23.	23.	24.	8.	3.	2.
GRAND TOTALS	100.0	15.	25.	26.	22.	6.	5.	1.

Table 4-4. CCSEM Analysis Results for Pittsburgh #8 (DOE) Coal

```
CCSEM File:1114r3.cma (repolished)
RUN       3  DATE 26-FEB-94 SUMMARY   136   TOTAL  1199

                                             AVERAGE SPECIES COMPOSITION

   #  MINERAL SPECIES      Na  Mg  Al  Si  P   S   Cl  K   Ca  Ti  Fe    Weight %

 106  Quartz               0.  0.  0. 98.  0.  0.  0.  0.  0.  0.  0.        9.1
  57  Kaolinite            0.  0. 45. 52.  0.  1.  0.  1.  1.  0.  0.        4.0
 106  Illite               0.  0. 29. 53.  0.  2.  0. 12.  1.  0.  2.        9.0
   1  K-Feldspar           0.  0. 17. 63.  0.  0.  0. 20.  0.  0.  0.        0.0
   9  Chlorite             0.  0. 20. 31.  0.  1.  0.  0.  1.  0. 47.        1.9
   5  Montmorillonite      6.  0. 19. 61.  0.  6.  0.  2.  4.  0.  1.        0.6
 207  Misc. Silicates      0.  0. 25. 64.  0.  3.  0.  3.  2.  0.  2.       19.2
   9  Elem. Sulfur         0.  0.  0.  3.  1. 84.  0.  0.  3.  0.  5.        1.0
 273  Pyrite               0.  0.  0.  0.  0. 64.  0.  0.  0.  0. 36.       27.2
  44  Misc. sulf.          0.  0.  1.  8.  0. 71.  1.  1.  1.  2. 11.        3.7
   1  Apatite              0.  0.  0.  0. 26.  3.  3.  0. 68.  0.  0.        0.0
  25  Fe-rich              0.  0.  0.  0.  0.  1.  0.  0.  0.  0. 99.        4.7
  30  Calcite              0.  3.  0.  0.  0.  0.  0.  0. 96.  0.  0.        2.8
  39  Mixed Carbonate      0.  9.  0.  0.  1.  3.  0.  0. 78.  0.  9.        6.2
   6  Ti oxide             0.  0.  0.  2.  0.  0.  0.  0.  0. 98.  0.        1.7
  39  Quartz-Sulfate       0.  0.  2. 36.  0. 59.  0.  1.  0.  1.  0.        1.1
   2  Quartz-Pyrite        0.  0.  5. 25.  0. 45.  0.  2.  0.  0. 23.        0.2
  37  Sil-sulf             0.  0. 18. 35.  0. 38.  0.  6.  0.  0.  0.        2.5
   8  Silicate-Pyrite      0.  0. 23. 31.  0. 31.  0.  1.  1.  0. 13.        0.9
  66  Misc. Mixed          0.  0. 18. 35.  1. 19.  1.  3. 15.  2.  4.        4.1
1070  GRAND TOTALS         0.  1. 11. 33.  0. 25.  0.  2.  9.  2. 17.      100.0


                              WEIGHT DISTRIBUTION

                                        Size Ranges (Microns)

  MINERAL SPECIES  WT. %     0.0     2.5     5.0     10.     20.     40.     80.
                             2.5     5.0    10.0     20.     40.     80.    500.


Quartz               9.1     15.      7.     35.     34.     10.      0.      0.
Illite               9.0     24.      0.     43.     21.      1.      1.     10.
Misc. Silicates     19.2     24.     12.     15.     33.      3.      5.      8.
Pyrite              27.2     10.     19.     20.      9.     27.     15.      0.
MINOR MINERALS      35.5     12.     12.     21.     35.      5.      9.      5.
--------------------------------------------------------------------------------
GRAND TOTALS       100.0     15.     12.     23.     26.     11.      8.      4.
```

Table 4-5. CCSEM Analysis Results for Beneficiated Pittsburgh #8 (SCS Cleaned) Coal

```
CCSEM File:1121r3.cma (repolished)
RUN        3  DATE 27-FEB-94 SUMMARY    104    TOTAL  1199

                                        AVERAGE SPECIES COMPOSITION

   #  MINERAL SPECIES       Na   Mg   Al   Si   P    S   Cl    K   Ca   Ti   Fe     Weight %

  64  Quartz                0.   0.   0.  98.  0.   1.  0.   0.   0.   0.   0.          6.0
 153  Kaolinite             0.   0.  46.  52.  0.   1.  0.   1.   0.   0.   0.          7.1
  57  Illite                0.   0.  26.  52.  0.   2.  0.  16.   0.   0.   3.         10.2
 225  Misc. Silicates       0.   0.  30.  56.  0.   7.  0.   2.   0.   0.   4.         18.1
   2  Elem. Sulfur          0.   0.   0.   9.  0.  82.  0.   0.   0.   0.   9.          0.1
 167  Pyrite                0.   0.   0.   0.  0.  66.  0.   0.   0.   0.  34.         21.4
   4  Ferrous Sulfate       0.   0.   0.   4.  0.  48.  0.   0.   0.   0.  47.          0.3
   1  Jarosite              0.   0.   0.   6.  0.  42.  0.  10.   0.   0.  42.          0.1
  89  Gypsum                0.   0.   0.   0.  0.  46.  0.   0.  54.   0.   0.          3.8
   1  Chalcopyrite          0.   0.   0.   0.  0.  30.  0.   0.   0.   0.  62.          0.1
   2  Sphalerite            0.   0.   0.   5.  0.  61.  0.   0.   0.   0.  13.          0.5
  81  Misc. sulf.           0.   0.   2.   6.  0.  50.  0.   2.  12.   0.  26.          9.6
   4  Fe-rich               0.   0.   0.   0.  0.   2.  0.   0.   0.   0.  97.          1.3
   5  Mixed Carbonate       0.   0.   0.   5.  0.  12.  0.   0.   0.   0.  84.          0.3
  13  Quartz-Sulfate        0.   0.   7.  42.  0.  40.  0.   6.   0.   0.   4.          2.8
  11  Quartz-Pyrite         0.   0.   0.  24.  0.  28.  0.   1.   0.   0.  43.          1.8
  50  Sil-sulf              0.   0.  17.  34.  0.  38.  0.   1.   0.   1.   2.          3.7
  44  Silicate-Pyrite       0.   0.  19.  32.  0.  29.  0.   0.   0.   0.  18.          5.2
  70  Misc. Mixed           0.   0.   9.  18.  0.  33.  2.   2.   1.   1.  30.          7.6
1045  GRAND TOTALS          0.   0.  14.  32.  0.  30.  0.   3.   3.   0.  17.        100.0


                               WEIGHT DISTRIBUTION

                                          Size Ranges (Microns)


 MINERAL SPECIES  WT. %       0.0      2.5      5.0      10.      20.      40.      80.
                              2.5      5.0     10.0      20.      40.      80.     500.


Kaolinite           7.1        1.       7.      60.      24.       8.       0.       0.
Illite             10.2        3.      91.       5.       0.       1.       0.       0.
Misc. Silicates    18.1       32.      34.      26.       7.       2.       0.       0.
Pyrite             21.4        4.      23.      23.      27.       3.      20.       0.
Misc. sulf.         9.6        4.      24.      34.      34.       1.       4.       0.
Misc. Mixed         7.6       19.      52.      12.      16.       0.       0.       0.
MINOR MINERALS     26.0        8.      35.      28.      21.       2.       6.       0.
-----------------------------------------------------------------------------------------
GRAND TOTALS      100.0       11.      36.      26.      19.       2.       6.       0.
```

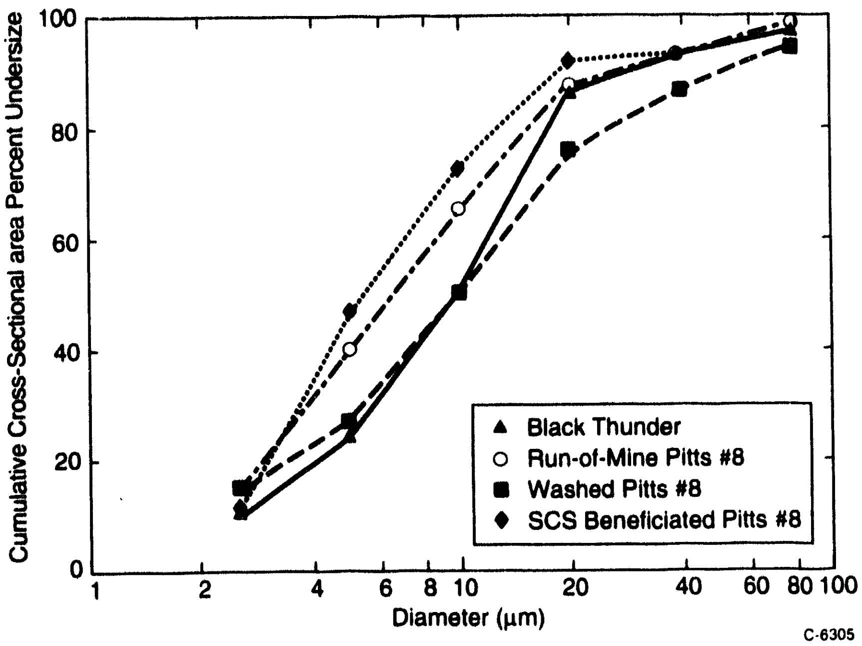


Figure 4-1. CCSEM mineral size distributions (UK analysis)

Table 4-6. Mössbauer Analysis on Coals (% of Fe)

Mössbauer File	Sample	Pyrite	Illite (Clay)	Siderite	Jarosite	Ferric Sulfate (?)	Wt% Pyrite
MK1695	Pittsburgh #8 (Kaiser)	75	11		14		0.86
MK1698	Pittsburgh #8 (DOE)	90	7	3			0.73
MK1697	Pittsburgh #8 (SCS)	34	7		22	37	0.23
MS1736	Pittsburgh #8 (SCS) H_2O Washed	59			41		0.27
MK1699	Black Thunder	100*					0.071

*Weak signal; CCSEM and chemical fractionation indicated approximately 50 to 60% iron as carbonate (Section 3).

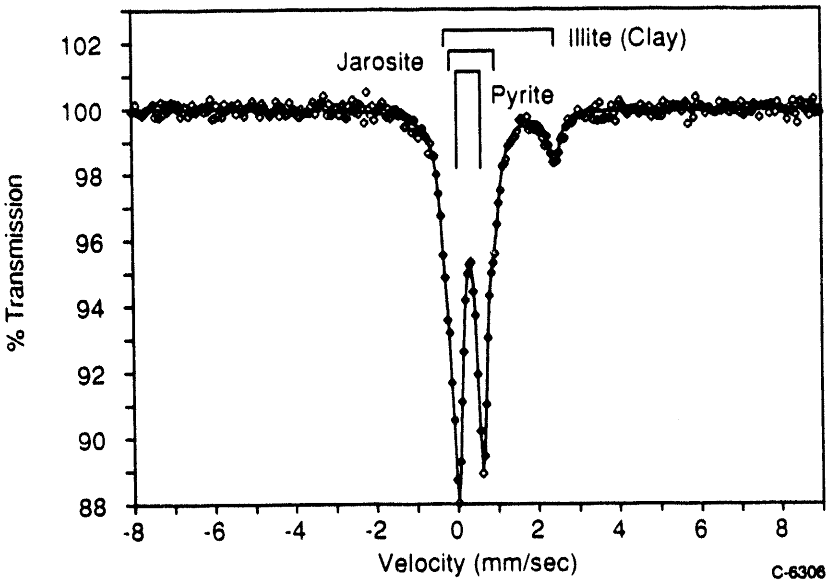


Figure 4-2. Pittsburgh #8 (Kaiser) parent coal (CFFLS #1120, MK1695)

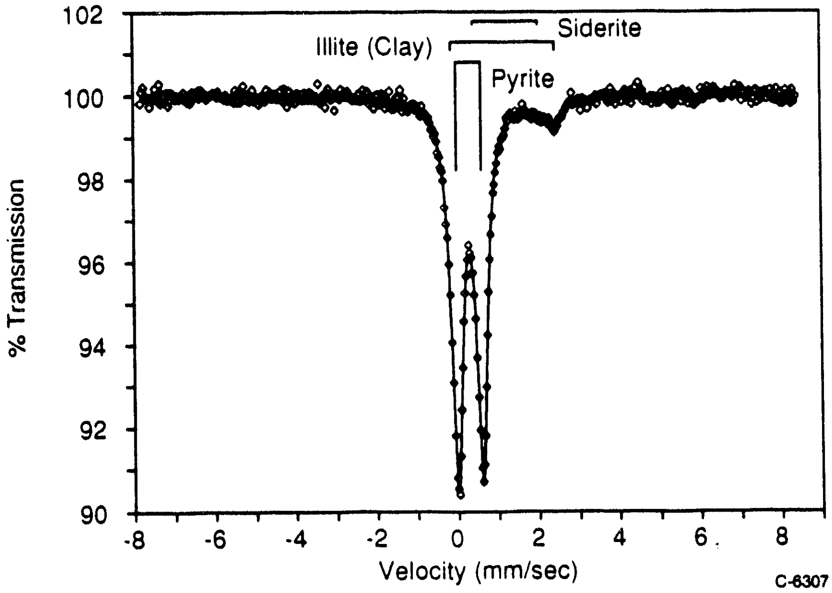


Figure 4-3. Pittsburgh #8 (DOE) coal (CFFLS #1114, MK1698)

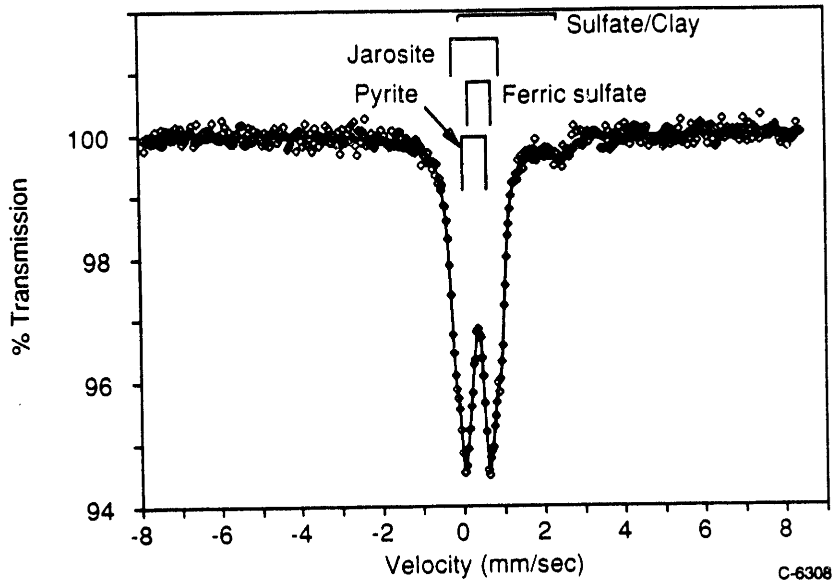


Figure 4-4. Beneficiated (SCS) Pittsburgh #8 (CFFLS #1121, MK1697)

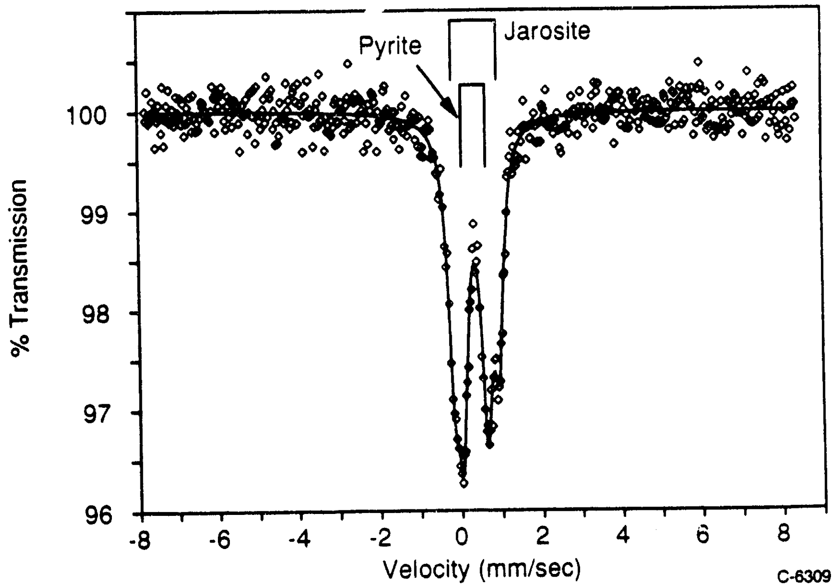


Figure 4-5. Cold Water Washed Pittsburgh #8 (SCS), (CFFLS #1121, MK1736)

SECTION 5

TASK 5.2

PULVERIZED COAL COMBUSTION STUDIES UNDER REDUCING CONDITIONS IN A CONTINUOUS, SELF-SUSTAINED LABORATORY SCALE REACTOR

(University of Arizona)

5. PULVERIZED COAL COMBUSTION STUDIES UNDER REDUCING CONDITIONS IN A CONTINUOUS, SELF-SUSTAINED LABORATORY SCALE REACTOR

During the last quarter, the following tasks were accomplished:

1. Purchase of the computer system and installation of the SMPS software on the DMPS particle sizing system. This will allow for much more rapid particle size analysis (albeit without chemical speciation) of samples extracted from combustion experiments.

2. Study of the coal analyses of the new Pittsburgh #8 coals (DOE washed, Kaiser parent, and Kaiser (SCS) beneficiated) and the Black Thunder coal to determine the priority for subsequent coal experiments. Selection will be based on which coals will best expand the range of coal ash and Fe content beyond the ranges of those coals previously studied. Ash and Fe content are important parameters in our current modeling efforts.

3. Reconstruction of the "drop tube" aerosol reactor. This work is still ongoing, including the replacement of the quartz furnace tube and complete redesign and reconstruction of the feeder system.

4. Replacement of a significant portion of the laboratory self-sustained-furnace gas sampling lines, air and fuel plumbing lines, and safety interrupts. This work is being done largely be another student on a separately funded project, but the delay will obviously affect the progress of this program as well. After modifications are complete, the feed, sampling, and analysis system of the self-sustained combustor should be more reliable.

Future Plans

As soon as the furnace is operational, base case runs on the Black Thunder Coal will be performed. This coal is a relatively low ash content coal and as such should be easier to burn than the higher ash coals. Following these initial experiments, experiments using the Kaiser run-of-mine Pittsburgh #8 parent and SCS beneficiated coals will also be conducted. The intention is to obtain data comparing combustion behavior of a coal in both the beneficiated and unbeneficiated state. There is some concern about the exact characteristics of these coals, however. First, the beneficiated sample was not prepared directly from the same batch of coal as the parent sample. Second, while the ash content has been reduced significantly (27% down to 5%), the iron content, *as a fraction of the total ash content*, has actually increased. It seems that the beneficiation has preferentially removed Si rather than Fe. Additionally, more controlled experiments, against which we can compare model results, will be conducted in a drop-tube aerosol reactor. In both cases, total filter and size-segregated samples will be collected and analyzed, predominately by Mössbauer, which focuses on Fe speciation.

The potential use of a Silverdale coal from the UK is also be considered. The attractive aspect of this particular coal is the fact that this is a high pyrite content coal where the pyrite is likely to be more finely disseminated than in a typical eastern U.S. coal. This particular characteristic will complement the previous coals studied.

SECTION 6

TASK 5.3

FUNDAMENTAL STUDY OF MINERAL INTERACTIONS AND ASH VAPORIZATION UNDER REDUCING CONDITIONS

(M.I.T.)

6. FUNDAMENTAL STUDY OF MINERAL INTERACTIONS AND ASH VAPORIZATION UNDER REDUCING CONDITIONS

During this quarter the strategy of the research program has been refined and the experimental studies initiated. The drop-tube furnace has been refurbished. Tests with the Black Thunder coal showed that the coal underwent devolatilization, ignition, and partial combustion when the stoichiometry was less than 1. In the future, these experiments will be supplemented with single particle oxidation experiments to quantitatively study the details of the evolution of the fly ash in the final stage of burnout.

6.1 Introduction

In the previous progress report a review on the factors governing the size distribution, chemical composition distribution, and deposition potential of coal combustion derived ash was presented. Although major progress has been made in characterizing the mineral matter in coals and its transformation, several areas in need of improvements in mechanisms and the models for predicting ash particle size and composition were proposed. The objective of this study is therefore to develop an improved understanding of coal ash and deposit formation under realistic coal combustion conditions, especially under reducing conditions relevant to combustion modification for NO_x control.

To accomplish this objective, a coal combustion condition which approximates realistic reducing conditions should be created in our experiment. Low-NO_x burners use a concentrated coal/air mixture in the combustion zone of primary-air burner (the stoichiometric ratio of fuel/air is greater than for standard burners). The combustion temperature of the concentrated coal dust is lower than that of the ordinary burners. The concentrated coal dust is therefore under reducing stoichiometry during devolatilization and ignition.

In the experimental apparatus used in this laboratory by previous researchers, a dilute coal stream with a large excess of air or oxygen in the annulus was injected along the axis. In those efforts, the laboratory experiments were designed to determine the fundamental mechanisms of mineral matter transformation during coal combustion under fuel-lean conditions and not to simulate the real combustor.

As stated in the last report, the mineral matter transformation depends upon many factors. In general, the submicron ash is determined by vaporization, nucleation and heterogeneous condensation, and the residual fly ash is determined by ash coalescence and char burnout. If we know the mineral distribution within pulverized coal and the changes of the coal structure during combustion, we can predict its behavior given the physico-chemical properties of the mineral inclusions. At this stage we can relate the final results to the combination of single factors, which can also enable us to understand the mechanisms and predict the mineral transformation under a wide range of conditions.

Nevertheless, coal mineral transformation during combustion is a complicated physicochemical process which needs to be studied under well-defined conditions. For example, combustion temperature is dependent upon the coal/carbon physical properties, the environment under which it burns, and the ratio of CO/CO_2.[2-3] Unfortunately, one parameter is difficult to change while keeping other parameters constant.

Therefore, the objective of this research is to distinguish the effects of stoichiometry and other variables on the mineral transformation and how they affect the process and the difficulty of separating one variable from the others may be kept in mind. This is possible in the laboratory where the temperature can be adjusted independently of the fuel/air ratio by electrically heating the walls.

6.2 Approach

As discussed above, mineral transformation during combustion is a complicated physicochemical process. The process cannot be studied by merely changing the oxygen concentration. In this study, a drop-tube furnace and single particle experiments will be used. In use of the furnace, a well-characterized sample will be prepared for quantitative study. The single particle experiments will be used to follow the details of the evolution of the fly ash, but will not be necessarily representative, which will enable us to distinguish variables.

6.3 Progress

The diagram of the drop-tube furnace is shown in Figure 6-1. This furnace had not been used for about 2 years and needed to be recalibrated.

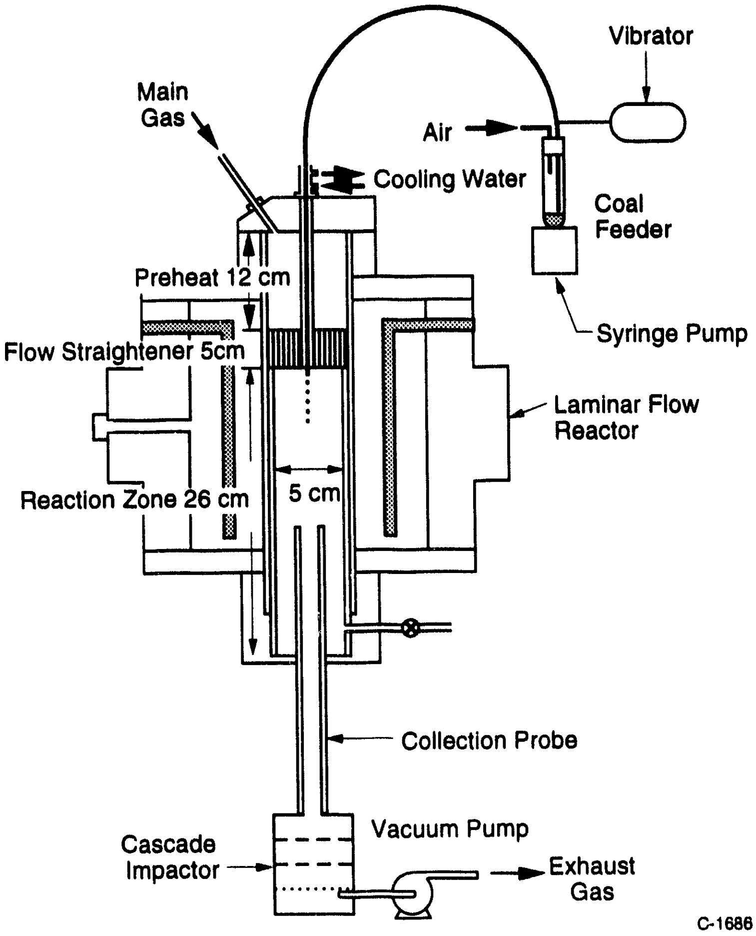
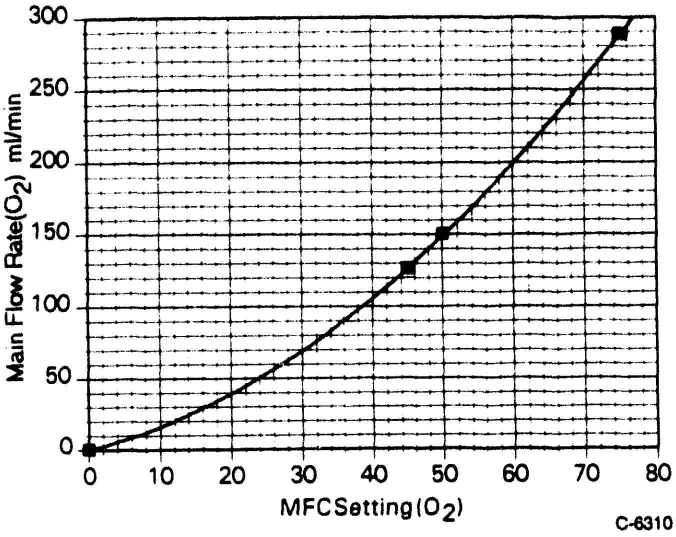


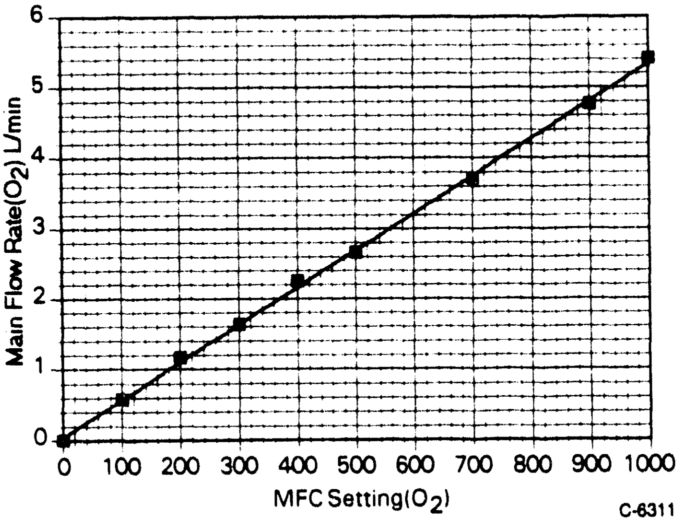
Figure 6-1. MIT drop-tube furnace.

6.3.1 Furnace Testing

Accessories such as connectors, pipes, and most flow meters were changed. The collection probe was replaced by a new design. The furnace was exhaustively tested for leaks. The new flow meters were calibrated. When the flow rate was low (less than 5 ml/min), bulb method was applied. When the flow rate was high (greater than 6 ml/min), a wet standard test meter was used. The calibration curves for each flow meter are given in Figures 6-2 through 6-13.



(a)



(b)

Figure 6-2. Calibration of MFC for O_2

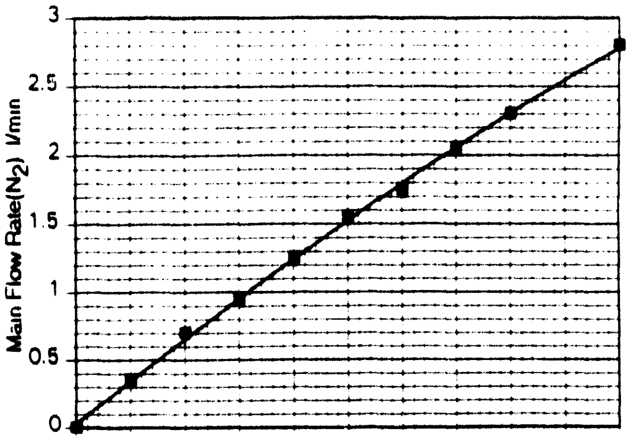


Figure 6-3. Calibration of MFC for N_2

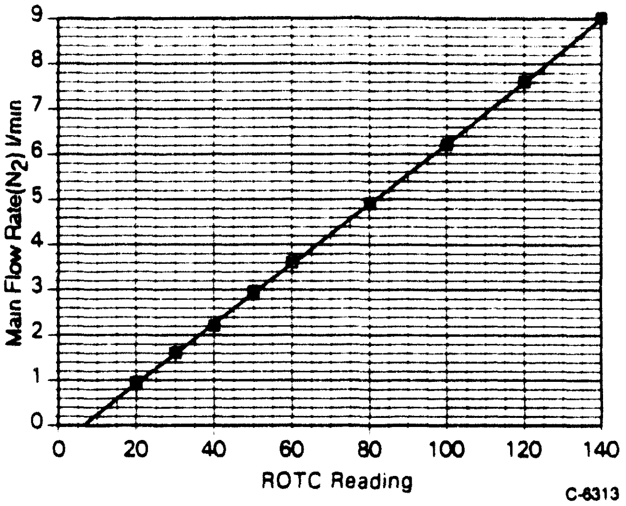


Figure 6-4. Calibration of rotameter for main N2

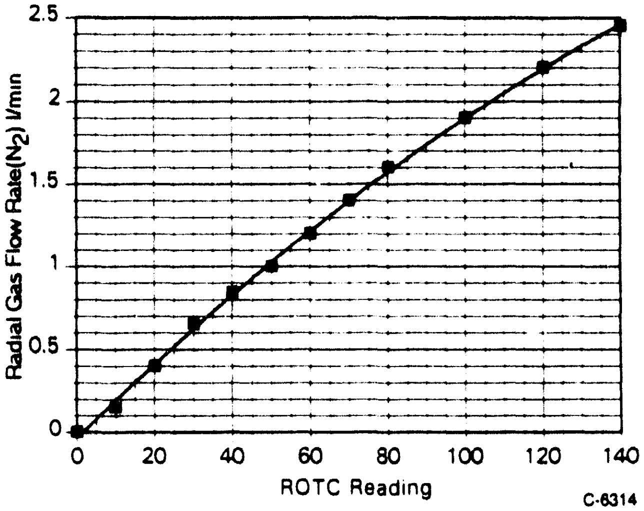


Figure 6-5. Calibration of rotameter for radial gas (N_2).

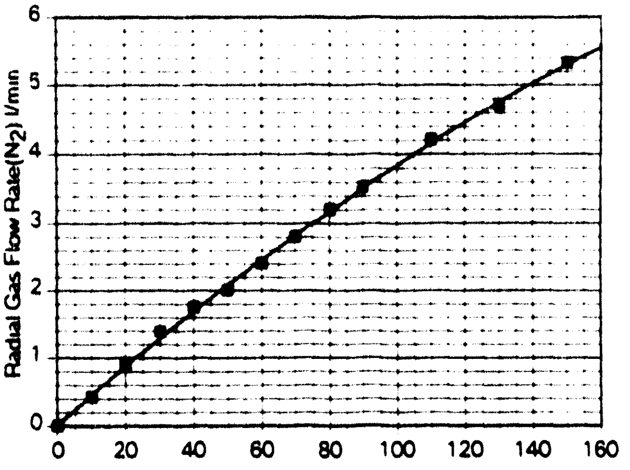


Figure 6-6. Calibration of rotameter for radial gas (N_2) - stainless ball.

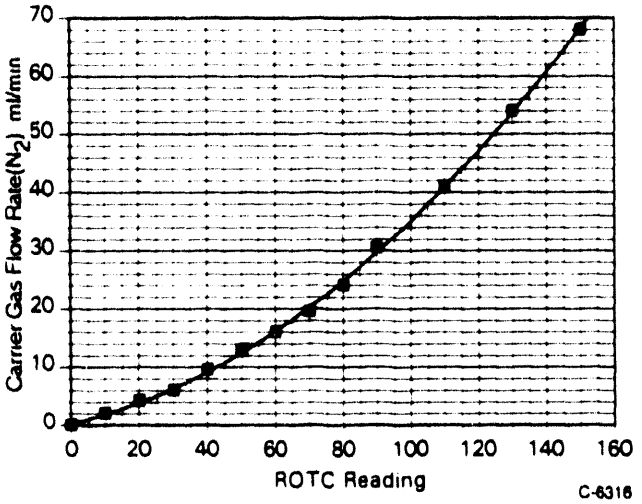


Figure 6-7. Calibration of rotameter for carrier gas (N_2).

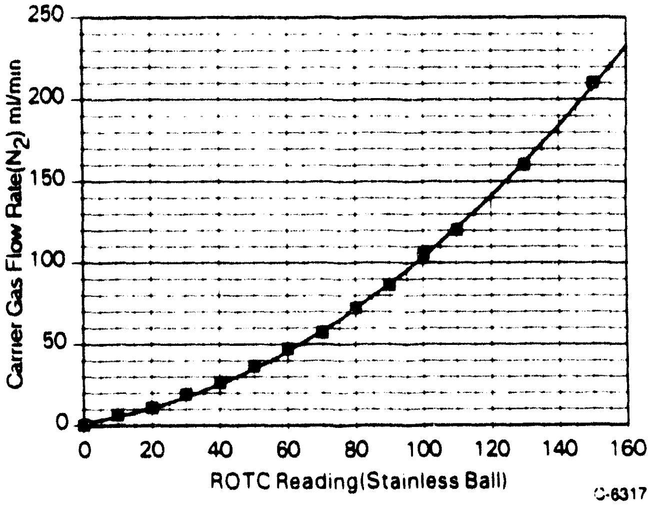


Figure 6-8. Calibration of rotameter for carrier gas (N_2) - stainless ball.

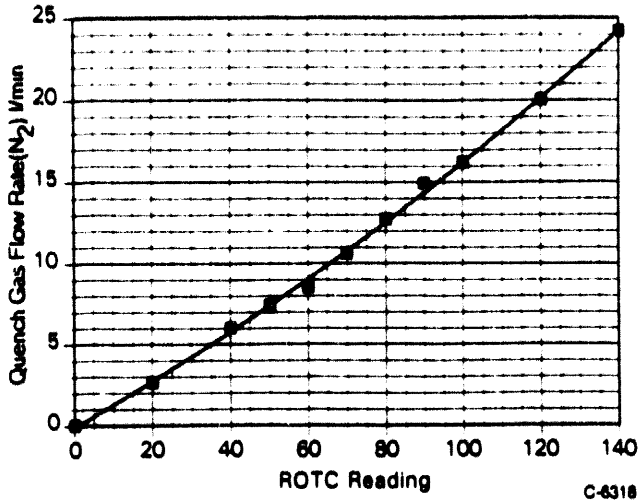


Figure 6-9. Calibration of rotameter for quench gas (N_2).

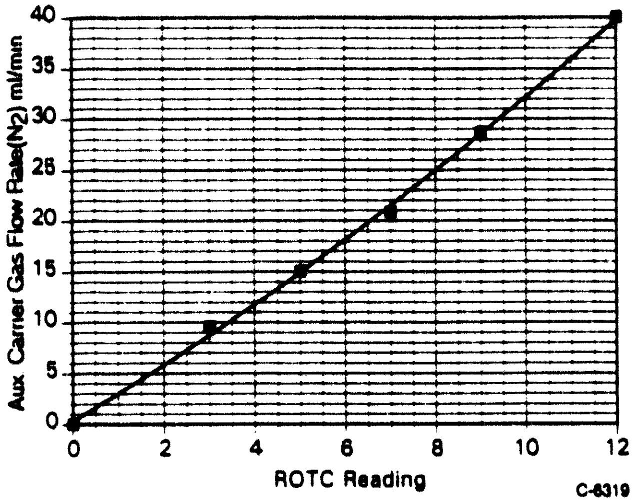


Figure 6-10. Calibration of rotameter for auxiliary carrier gas (N_2).

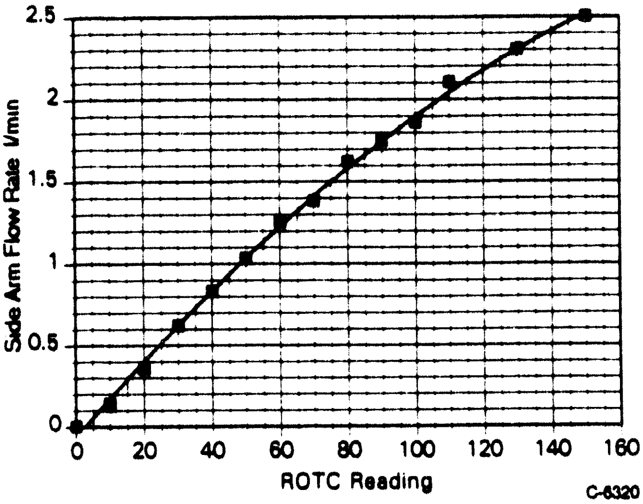


Figure 6-11. Calibration of rotameter for side arm (air).

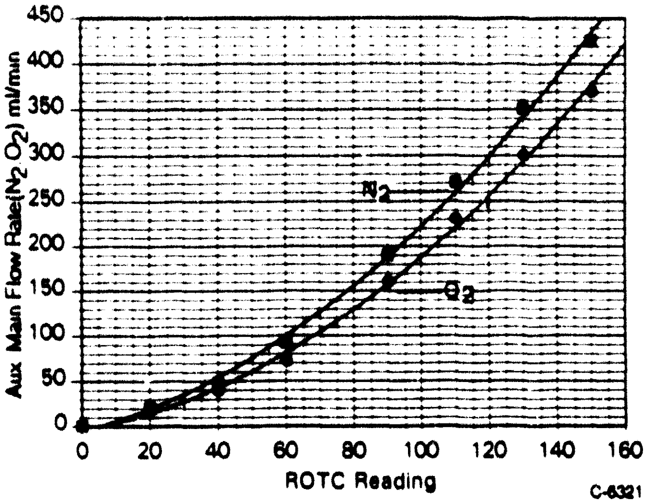


Figure 6-12. Calibration of rotameter for auxiliary gas.

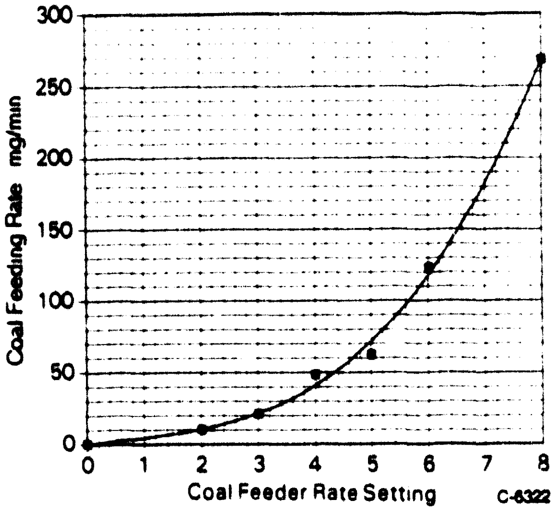


Figure 6-13. Calibration of coal feeder.

6.3.2 Coal Preparation

The coal samples to be used were prepared by dry sieving. First, about 0.5 kg of coal was put on the upper sieve (number 120 or 125 μm). Then it was put on the mill together with the lower sieve (number 140, or 105 μm) and the covers. After the mill had run for about half an hour, the coals on the upper sieve and on the lower cover were removed. Then the set of sieves were again put on the mill. The coals were separated for about another hour to prepare 106/125 μm size fractions.

6.3.3 Experiment

Before running a combustion experiment, the recovery efficiency of particles from the furnace was carefully calibrated. The Black Thunder coal with size between 106 and 125 microns was fed into the furnace at ambient temperatures. The probe, preseparator and cascade impactor described elsewhere[4-6] were used to collect the coals. Several operations were performed. A typical result was as follows:

The coal fed in 1.34977 g
The coal collected 1.19185 g
to give a recovery of 1.19185/1.34977 = 88.3%.

As mentioned in Ref. 5, good recovery is near 90%.

Operation Condition. Because of feeder limitations, we can only use about 1 gram coal for each run. The total amount of oxygen of each run at stoichiometry = 1.0 will be 1568 ml (note: here the Black Thunder coal was used). If the time for a run is 10 min, the flow rate will be 157 ml/min. In the test, about 25 l/min quench gas, 4 l/min radial gas, and about 1 l/min main nitrogen and 157 ml/min main oxygen were used.

6.3.4 Results and Discussion

Table 6-2 summarizes the results obtained from our initial experiment with Black Thunder coal. Each operation condition was repeated four times. From the samples collected on the cascade impactor stage substrates, we could see that when the stoichiometry was not less than 1, the collected samples were white and there was no material deposited on the preseparator and stage 0, which indicated that the coal burned completely. When the stoichiometry was less than 1, significant amounts of char and ash were collected on the preseparator and stage 0. This showed that the coal underwent devolatilization, ignition and partial combustion. These samples will be analyzed to determine the distribution and chemical composition of the inorganic constituents on each stage.

6.4 Future Work

The immediate tasks will be to determine the fate of the mineral matter during fuel rich combustion. The questions to be addressed are how mineral matter escapes with the volatiles and what is the form of the mineral matter in the char leaving the fuel-rich combustor as a function of char burnout. The four program coals will be studied. Particular attention will be paid to the fate of sodium and iron in the partially burned char, since this has a major impact on both vaporization and coalescence of mineral inclusions.

SECTION 7

TASK 2

FUNDAMENTAL STUDY OF ASH FORMATION AND DEPOSIT INITIATION UNDER REDUCING CONDITIONS

(PSI PowerServe)

7. FUNDAMENTAL STUDY OF ASH FORMATION AND DEPOSIT INITIATION UNDER REDUCING CONDITIONS

7.1 Iron Oxidation State in Study Glasses

In order to better understand the effect of reducing conditions on ash deposition characteristics, a number of experiments are planned to measure the stickiness of glassy iron-containing ash particles as a function of iron oxidation state. The iron oxidation state has a significant effect on viscosity (Figure 7-1), a key parameter in determining ash particle stickiness. Iron-containing glasses will be used as model ash compounds. These glasses will be exposed to various oxidizing environments and their stickiness measured with deposition probes. Early last year PSI ordered, and received, the two glasses to be used as model compounds. These glasses, produced by Mo-Sci Corp., were specified to contain approximately 20% iron (see Table 7-1 for specifications), all of which was to be in the Fe^{2+} state.

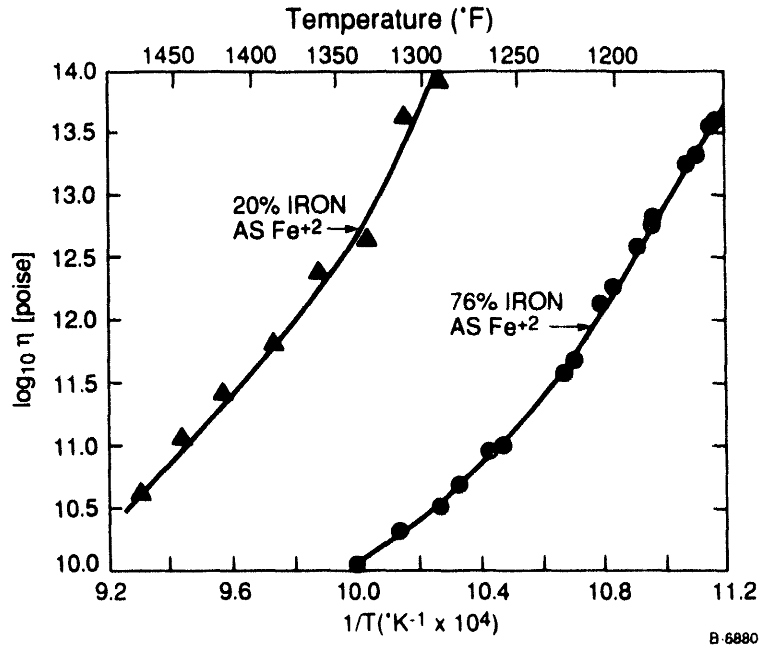


Figure 7-1. Iron oxidation state effects on ash particle viscosity.

Table 7-1. Composition Specifications for Iron Containing Glass

Oxide	Weight %
SiO_2	51
Al_2O_3	21
FeO	20
K_2O	8

To verify that only Fe^{2+} was present, samples of these glasses were analyzed by Mössbauer spectroscopy and by (Fe^{2+}) titration. The initial Mössbauer analysis indicated that, in addition to the expected Fe^{2+}, both glasses contained an appreciable amount of metallic iron. The FeO titration, however, indicated the glasses contained approximately 19.54% and 19.49% FeO by weight, the target amount. When a second Mössbauer analysis also indicated that metallic iron was present, it was thought that the glasses might contain more iron than the target 20%, some of which could be as metallic iron. Therefore, samples of the glasses were sent to a commercial laboratory for total iron analysis by Atomic Absorption (AA). The total iron analysis (18.64% and 19.95%) agreed with both the manufacturing specifications and the FeO titration. Therefore, for the purpose of the planned stickiness experiments, all of the iron is assumed to be present initially as Fe^{2+}.

7.2 Temperature Mapping of PSI Entrained Flow Reactor

Many of the experiments planned for this program will use reactor configurations, and operating conditions, not previously explored at PSI. These new configurations include increased gas flow in the reactor, and insertion of an injection probe. New operating conditions include deeply reducing combustion stoichiometries (stoichiometric ratio 0.6). As the effect of these new configurations/operating conditions on gas temperatures in the reactor was unclear, efforts were made in the last quarter to characterize the reactor under many of these configurations/operating conditions. Temperature measurements were made with two different probes and indicated that the gas flowrate in the reactor has a relatively small effect on the gas temperature. The temperature measurements also showed that the furnace temperature setpoint, location of the N_2 quench probe, and the location of the coal injection probe significantly affect the gas temperature.

7.2.1 Description of Temperature Probes

For these temperature measurements two different types of probes were used; a modified particle injection probe, and a suction pyrometer. Both probes used type R thermocouples. In addition, the thermocouple beads were shielded to minimize the effect of wall radiation on the temperature readings.

The modified injection probe consisted of a particle injection probe, to be used in future experiments, with a thermocouple inserted down the center. This probe was used to measure gas temperatures from the top of the reactor downwards (see Figure 7-2). The thermocouple bead was shielded by an open platinum foil cylinder suspended from the ceramic thermocouple protector as shown in Figure 7-3. By using the platinum foil cylinder, gas was allowed to flow around the bead, but radiation from the wall was minimized. The injection probe was inserted in one of three locations, corresponding to approximately 30.5, 58.4, and 86.4 cm from the top of the reactor (the same locations used in subsequent experiments, see Subsection 7.3). At each location the thermocouple was extended in small increments to a maximum of 23.5 cm from the end of the probe, with temperature measurements recorded at each position. In order to quantify the quenching effect of the injection probe, the probe was insulated with 1/8 in. thick Inswool-HP ceramic fiber paper. The probe was then fully inserted in the reactor, and the temperature measurements repeated.

The gas temperatures in the lower half of the reactor were also measured using a suction pyrometer. The pyrometer was inserted in increments upwards from the bottom of the reactor, with temperature measurements taken at each location. The optimum gas sampling rate, 5.7 slpm, was determined by slowly increasing the draw into the pyrometer and noting the temperature. At gas sampling rates lower than the optimum, the relatively stagnant gas around the bead is heated by

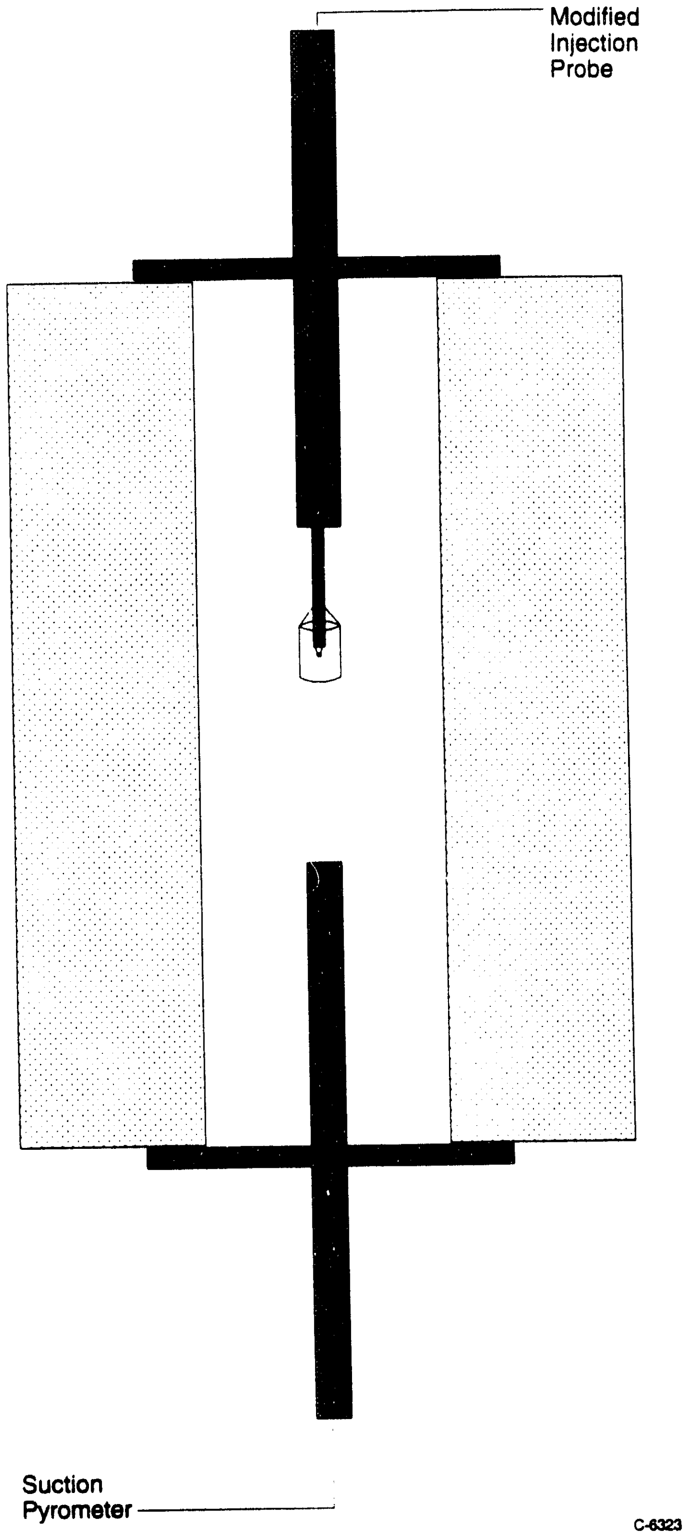


Figure 7-2. Schematic diagram of furance showing insertion of thermocouple probes.

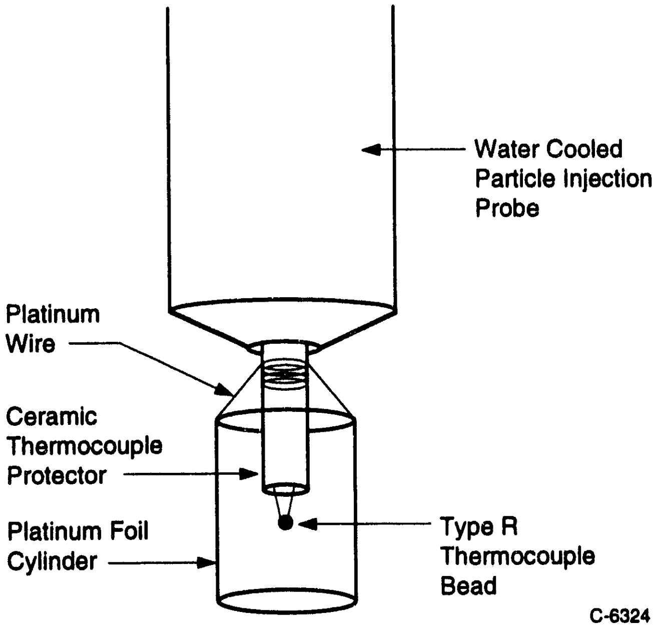


Figure 7-3. Schematic diagram of radiation-shielded thermocouple used for temperature profiling of PSI reactor.

radiation/conduction to temperatures higher than the true gas temperatures. For higher than optimum sampling rates, gas from other locations in the reactor are drawn into the probe. Therefore the temperature measurement is an average of the larger volume sampled, rather than the temperature of the gas at the thermocouple location. As seen from Figure 7-4, a plateau was reached at sampling rates near the optimum. In addition, this optimum flow was found to be constant for all three reactor flow rates.

7.2.2 Temperature Profiles

Effect of Furnace Setpoint and Gas Flowrate:

The first two experiments were designed to determine the effect of the furnace setpoint and gas flowrate on the reactor temperature profile. These experiments used furnace setpoints of 1300° and 1500° C and gas flowrates of 1, 2.5, and 4 SCFM (28.3, 70.8, and 113.2 slpm). For these experiments the N_2 quenched sampling probe was also inserted approximately 25 cm into the bottom of the reactor (approximately 117 cm from the top of the reactor). The temperature profiles, Figures 7-5 and 7-6, were measured using only the modified injection probe described above. The effect of gas flowrate was relatively small, but non-negligible. As expected, increasing the furnace setpoint caused a corresponding increase in the gas temperature. The peak gas temperatures were approximately 50 and 100° C less than the setpoint for the lowest and the highest flowrates, respectively. Another important point to note is the effect of the N_2 quenched probe. This probe, and the fully inserted injection probe, caused a significant drop in the gas temperature in the lower part of the furnace. This effect was explored in the next set of measurements.

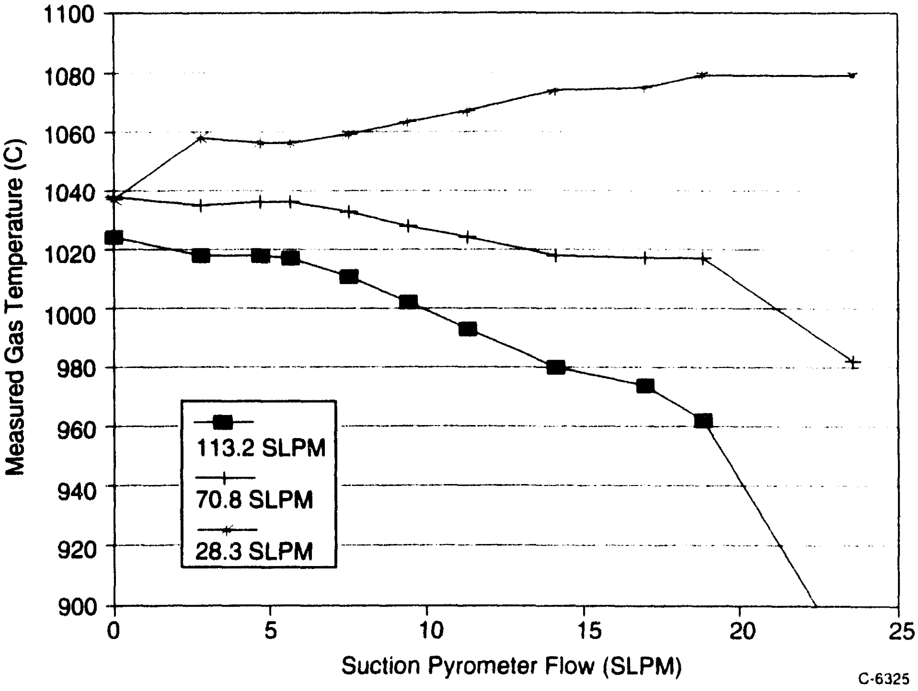


Figure 7-4. Suction pyrometer temperature measurements for various furnace flowrates.

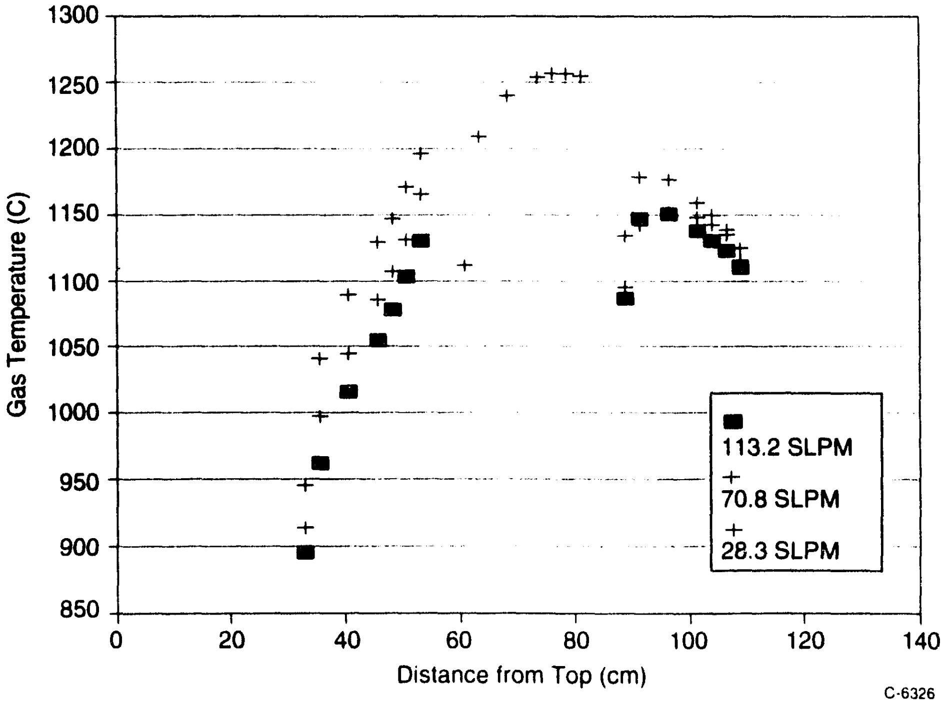


Figure 7-5. PSI drop tube temperature profile (setpoint: 1300°C with quenched probe)

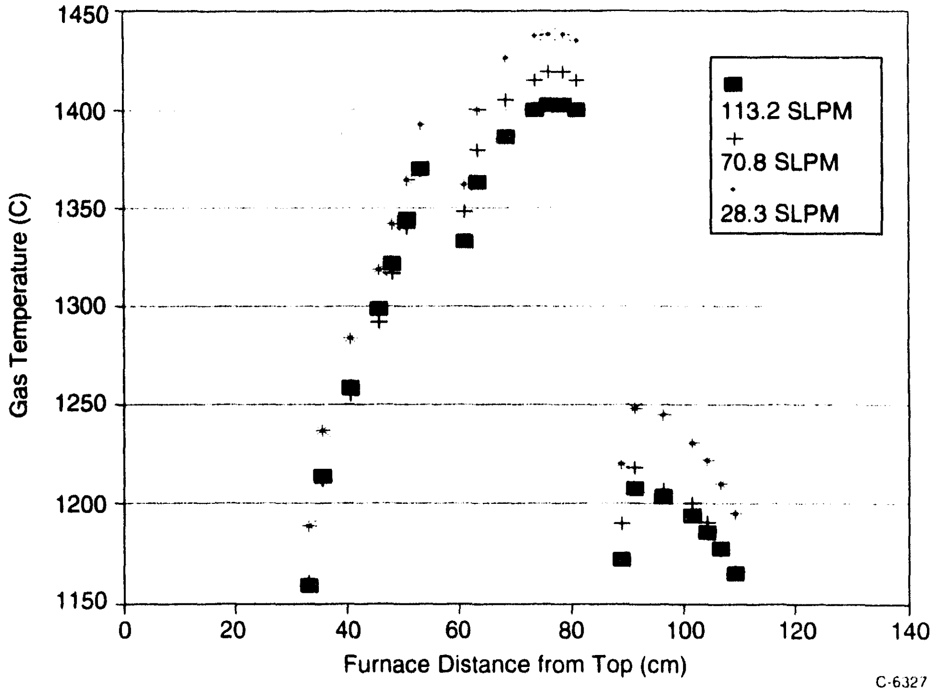


Figure 7-6. PSI drop tube temperature profile (setpoint: 1500°C with quenched probe).

Effect of N_2 Quenched Probe and Fully Inserted Injection Probe:

The effect of both the N_2 quenched probe and the fully inserted injection probe can be seen in Figure 7-7. The furnace temperature profile in the absence of both probes can be seen in Figure 7-8. In this case the peak temperature in the lower section of the reactor is approximately 50°C below the setpoint. When the injection probe was fully inserted, the peak temperature decreased by approximately 150° C. Addition of the N_2 quenched probe caused another 100°C decrease. However, when the N_2 quenched probe was removed and the injection probe insulated with a ceramic fiber insulation, the lower-reactor temperatures are very similar to those obtained in the absence of both probes.

The data from these measurements suggest that the gas temperature profile will be fairly similar for experiments at different gas flowrates. However, for those experiments where the injection probe is to be fully inserted, the probe must be insulated to avoid significant decreases in reactor gas temperatures. When the probe is insulated, or only partially inserted, its effect on the temperature profile is small.

7.3 Short Residence Time Experiments

One of the important questions to be answered in this work is the effect of sub-stoichiometric combustion conditions on ash deposition behavior. One of the effects of substoichiometric conditions is the presence of residual carbon and unoxidized, or only partially oxidized, pyrite in the ash. In the past quarter several experiments were performed to explore the effect of these components on ash

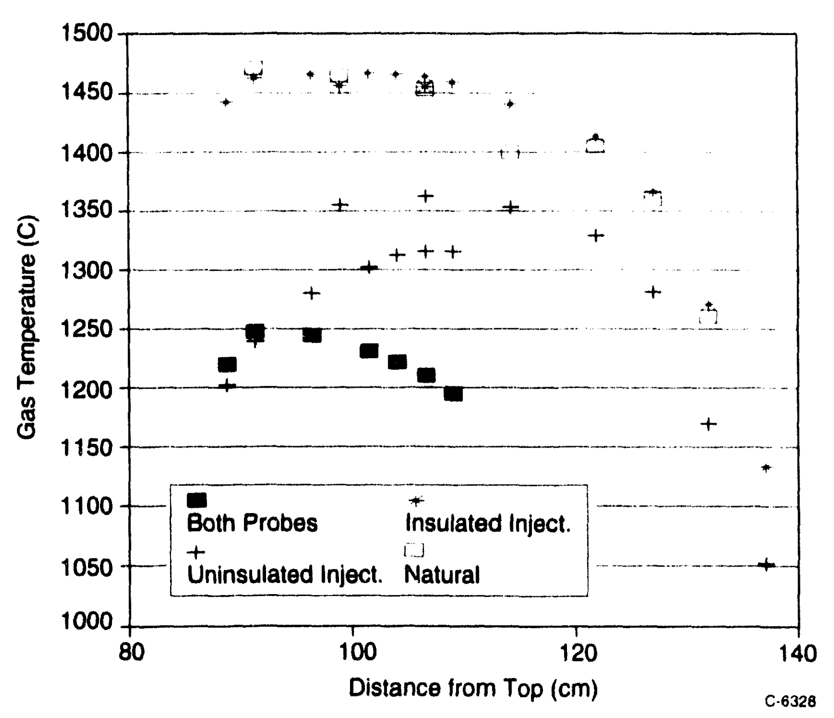


Figure 7-7. PSI drop tube temperature profile (effect of quench and injection probes).

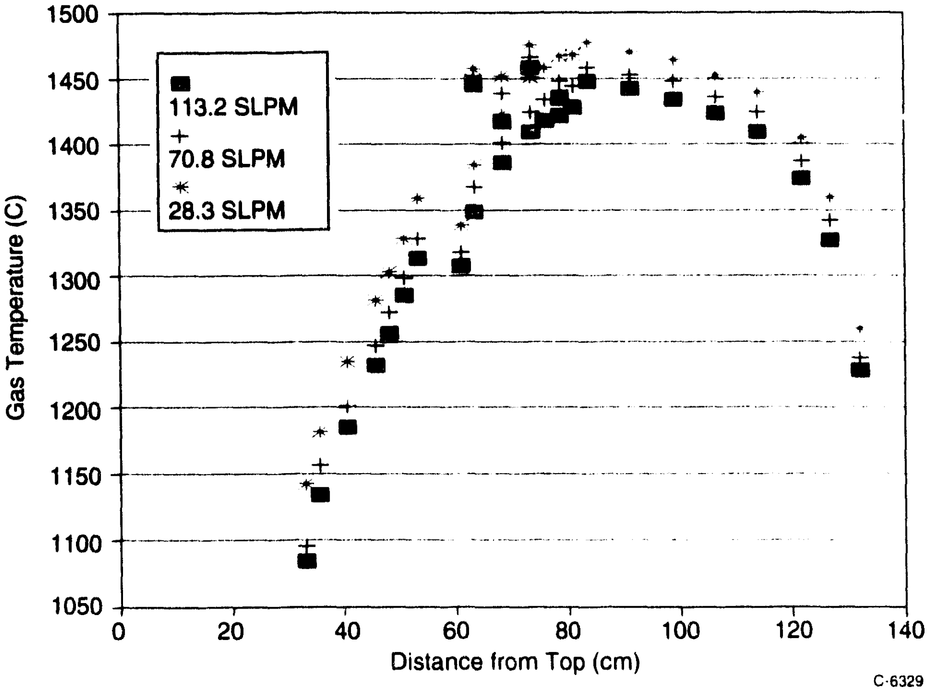


Figure 7-8. PSI drop tube temperature profile (setpoint: 1500°C, no probes).

stickiness. These experiments focused on the stickiness of ash at short residence times. Although the experiments were all carried out under fuel lean conditions, the presence of carbon and partially oxidized pyrite in the ash at short residence times is similar to that obtained by combustion of the coal under reducing conditions.

For these experiments the coal, washed Pittsburgh No. 8 (obtained from DOE/PETC), was fed at 0.6 g/min into the reactor, using the injection probe. In order to control the fractional conversion of pyrite, and the amount of carbon in the ash, the residence time was varied between 0.25 and 2.9 seconds by varying injection probe location and gas flowrates. Table 7-2 outlines the conditions used to achieve each residence time. As seen in the table, the furnace setpoint was reduced for the lower flowrate. This was to ensure that the gas temperature in the test section of the deposition probe, discussed later, remained constant (as measured with a thermocouple) between the two different gas flowrates.

Table 7-2. Reactor Conditions and Configurations for Short Residence Time Experiments

Setpoint (°C)	Flow (slpm)	Injection Location (cm from top)	Residence Time (seconds)
1500	113.2	86.4	0.25
		58.4	0.40
		30.5	0.55
		0.0	0.72
1300	28.3	86.4	0.99
		58.4	1.60
		30.5	2.21
		0.0	2.87

At each of the residence times outlined in Table 7-2, ash deposition tests, similar to those described elsewhere[7] were performed. The particle velocities at the point of impaction were kept constant at 1 and 5 m/s, although for the high gas flowrate experiments only 5 m/s samples could be collected. The gas temperature at the deposition probe surface was also kept constant at approximately 1300°C. Ash samples were also collected using the nitrogen quenched probe. These samples were then analyzed for carbon content to determine the fractional burnout at the point (residence time) of deposition. Subsequent experiments are also currently being performed to collect ash samples under the same conditions for iron analysis, by Mössbauer spectroscopy. When complete, these samples will be sent to University of Kentucky for analysis.

The data from these experiments can be seen in Figure 7-9. In this figure both the fraction of *fixed* carbon remaining and the ash collection efficiency are plotted as a function of time. The *fixed* carbon remaining was calculated using the proximate analysis for this coal and assuming that devolatilization was complete. This parameter was used to determine the fractional char burnout. From the plot we can see that the collection efficiency, described in detail elsewhere,[7] increased dramatically between 0.25 and 0.400 seconds, with a maximum occurring at approximately 1 second. The collection efficiency continues to increase substantially after burnout is essentially complete (0.55 second). These data, and the data in Figure 7-10 suggest that, although high carbon contents (greater than 70%) in the ash may decrease ash stickiness, carbon plays at most a secondary role in

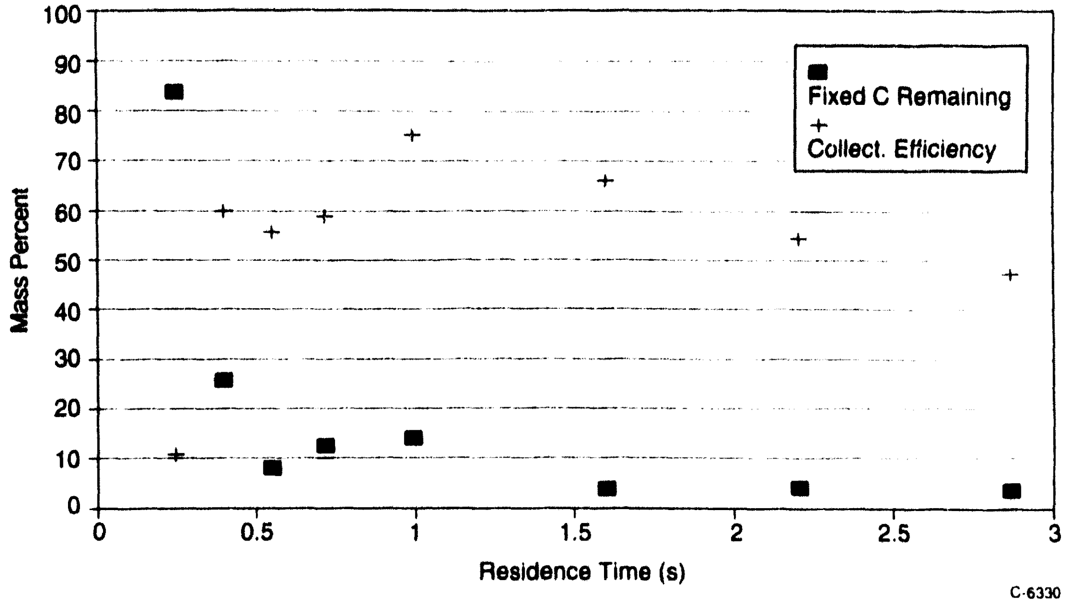


Figure 7-9. Burnout and stickiness comparison (washed Pittsburgh #8 (DOE)).

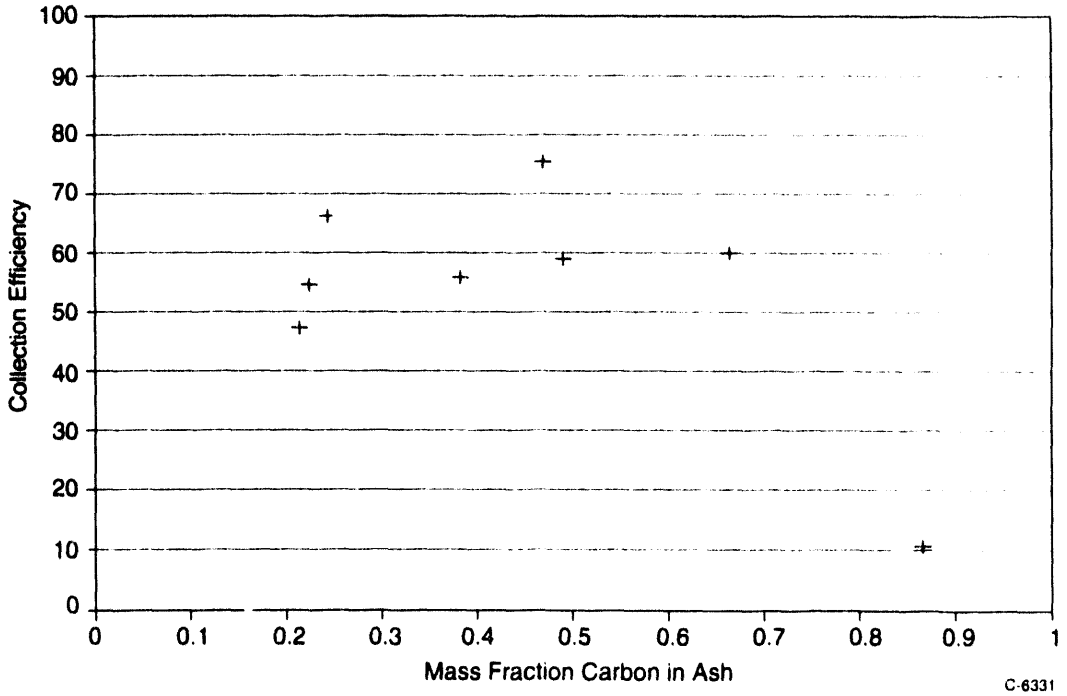


Figure 7-10. Collection efficiency versus carbon content (washed Pittsburgh #8 (DOE)).

ash stickiness. Pyrite oxidation, on the other hand, may cause the observed increase in stickiness due to the prolonged melt phase present during oxidation.[8] The subsequent reduction of ash stickiness seen in Figure 7-9 may be caused by the oxidation of iron in glassy ash particles (see Subsection 7.1). As previously mentioned, additional experiments are underway to collect data on pyrite and iron-glass oxidation under these conditions. By adding the fractional conversion of pyrite to the plot in Figure 7-9, it should be possible to determine the importance of un-oxidized iron on ash stickiness. This analysis is expected to be completed in the next quarter.

SECTION 8

REFERENCES

8. REFERENCES

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APPENDIX A

BLACK THUNDER SUB-BITUMINOUS COAL INCLUDED/EXCLUDED CCSEM DATA (UNDEERC)

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UMMARY OF CCSEM RESULTS: PROG VERSION 3BF  7/21/90
AMPLE DESCRIPTION ---> Black Thunder Coal-From EERC Blend Project
UBMITTER          ---> Katrinak
CC # AND FUND #   ---> 42586132
UN DATE AND TIME  --->  11  3 1993  15:28

ERCENT EPOXY USED                            =         67.8
OTAL MINERAL AREA ANALYZED AT  800.0 MAG     =       1652.3
ORMALIZED AREA ANALYZED AT  800.0 MAG        =     194259.7
INERAL AREA ANALYZED  240.0 MAG              =      18159.4
ORMALIZED AREA ANALYZED  240.0 MAG           =     200047.1
OTAL MINERAL AREA ANALYZED AT   50.0 MAG     =     101628.7
UMBER OF FRAMES AT  800.0 MAG                =           99
UMBER OF FRAMES AT  240.0 MAG                =           99
UMBER OF FRAMES AT   50.0 MAG                =           47
OTAL MINERAL AREA ON A COAL BASIS            =        1.314  ∴ coal is 2.6 wt %
OTAL MINERAL WGHT % ON A COAL BASIS          =        2.606        minerals
OTAL NUMBER OF POINTS ANALYZED               =          960
UMBER OF POINTS UNDER THRESHOLD              =            9

EIGHT PERCENT ON A MINERAL BASIS
                       1.0      2.2      4.6     10.0     22.0     46.0
                        TO       TO       TO       TO       TO       TO    TOTALS
                       2.2      4.6     10.0     22.0     46.0    100.0
-------------------------------------------------------------------------------
UARTZ                  4.3      3.7      6.8      3.5      5.0       .8      24.1
RON OXIDE               .4       .6      1.4       .9      1.3       .0       4.6
ERICLASE                .0       .0       .0       .0       .0       .0        .0
UTILE                   .2       .0       .0       .8       .0       .0       1.0
LUMINA                  .0       .0       .2       .0       .0       .0        .3
ALCITE                  .0       .1       .1       .0       .2       .4        .9
OLOMITE                 .0       .0       .0       .0       .0       .6        .7
NKERITE                 .0       .0       .0       .0       .0       .0        .0
AOLINITE               3.9      8.4      6.2      4.8      3.8      1.9      29.1
ONTMORILLONITE          .4      2.0       .7      2.1      1.1       .8       7.1
 AL-SILICATE            .2       .4       .1       .0       .0       .0        .7
E AL-SILICATE           .0       .3       .1       .0       .0       .0        .4
A AL-SILICATE           .1       .4       .1       .0       .1       .0        .8
A AL-SILICATE           .0       .0       .0       .0       .0       .0        .0
LUMINOSILICATE          .2       .9       .5      2.5       .1       .5       4.6
IXED AL-SILICA          .1       .1       .3       .0       .0       .0        .5
E SILICATE              .0       .0       .0       .0       .0       .0        .0
A SILICATE              .0       .0       .0       .0       .0       .0        .0
A ALUMINATE             .0       .0       .0       .0       .0       .0        .0
YRITE                   .5       .0       .1      2.2       .6      1.4       4.7
YRRHOTITE               .1       .0       .0       .0       .0       .0        .1
XIDIZED PYRRHO          .1       .0       .0       .0       .2       .0        .2
YPSUM                   .0       .2       .0       .0       .0       .0        .2
ARITE                   .1       .2       .4       .0       .0       .0        .6
PATITE                  .0       .0       .0       .0       .0       .0        .0
A AL-P                 1.6      3.1      4.0       .9       .2       .0       9.8
CL                      .0       .0       .0       .0       .0       .0        .0
YPSUM/BARITE            .1       .0       .0       .0       .0       .0        .1
YPSUM/AL-SILIC          .0       .0       .0       .0       .0       .0        .0
SI-RICH                 .3       .7       .5       .0       .2       .4       2.1
CA-RICH                 .0       .0       .0       .0       .1       .0        .1
CA-SI RICH              .0       .1       .0       .0       .0       .0        .1
UNKNOWN                3.0      1.0      1.2       .5       .8       .5       7.0
-------------------------------------------------------------------------------
 TOTALS               15.9     22.3     22.7     18.3     13.6      7.3     100.0
AGE    1  Black Thunder Coal-From EERC Blend Project
```

AREA PERCENT MINERAL BASIS

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOTA
QUARTZ	4.5	3.9	7.2	3.7	5.3	.9	25
IRON OXIDE	.2	.3	.7	.5	.7	.0	2
PERICLASE	.0	.0	.0	.0	.0	.0	
RUTILE	.1	.0	.0	.4	.0	.0	
ALUMINA	.0	.0	.2	.0	.0	.0	
CALCITE	.1	.1	.1	.0	.2	.4	
DOLOMITE	.0	.0	.0	.0	.0	.6	
ANKERITE	.0	.0	.0	.0	.0	.0	
KAOLINITE	4.1	9.0	6.6	5.1	4.1	2.0	30
MONTMORILLONITE	.5	2.2	.8	2.4	1.2	.9	8
K AL-SILICATE	.2	.5	.2	.0	.0	.0	
FE AL-SILICATE	.0	.3	.1	.0	.0	.0	
CA AL-SILICATE	.1	.4	.1	.0	.1	.0	
NA AL-SILICATE	.0	.0	.0	.0	.0	.0	
ALUMINOSILICATE	.2	1.0	.5	2.6	.1	.5	4
MIXED AL-SILICA	.1	.1	.3	.0	.0	.0	
FE SILICATE	.0	.0	.0	.0	.0	.0	
CA SILICATE	.0	.0	.0	.0	.0	.0	
CA ALUMINATE	.0	.0	.0	.0	.0	.0	
PYRITE	.3	.0	.0	1.3	.3	.8	2
PYRRHOTITE	.1	.0	.0	.0	.0	.0	
OXIDIZED PYRRHO	.1	.0	.0	.0	.1	.0	
GYPSUM	.0	.2	.0	.0	.0	.0	
BARITE	.0	.1	.2	.0	.0	.0	
APATITE	.0	.0	.0	.0	.0	.0	
CA AL-P	1.6	3.1	4.0	1.0	.2	.0	9
KCL	.0	.0	.0	.0	.0	.0	
GYPSUM/BARITE	.0	.0	.0	.0	.0	.0	
GYPSUM/AL-SILIC	.1	.0	.0	.0	.0	.0	
SI-RICH	.4	.7	.5	.0	.2	.4	2
CA-RICH	.0	.0	.0	.0	.1	.0	
CA-SI RICH	.0	.1	.0	.0	.0	.0	
UNKNOWN	3.1	1.0	1.3	.5	.9	.5	7
TOTALS	16.0	23.2	22.9	17.5	13.5	7.0	100

ISTRIBUTION BY % OF EACH MINERAL PHASE

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOTALS
JARTZ	17.7	15.3	28.1	14.5	20.9	3.5	100.0
RON OXIDE	9.5	13.4	29.5	19.9	27.8	.0	100.0
ERICLASE	.0	.0	.0	.0	.0	.0	.0
JTILE	20.8	.0	.0	79.2	.0	.0	100.0
LUMINA	15.8	.0	84.2	.0	.0	.0	100.0
ALCITE	5.8	14.1	14.6	.0	19.8	45.7	100.0
OLOMITE	5.4	.0	6.0	.0	.0	88.6	100.0
NKERITE	.0	.0	.0	.0	.0	.0	.0
AOLINITE	13.4	29.0	21.4	16.6	13.1	6.4	100.0
ONTMORILLONITE	6.1	27.8	9.4	29.9	15.6	11.2	100.0
AL-SILICATE	20.8	59.1	20.1	.0	.0	.0	100.0
E AL-SILICATE	5.9	74.5	19.6	.0	.0	.0	100.0
A AL-SILICATE	15.5	51.5	16.7	.0	16.4	.0	100.0
A AL-SILICATE	100.0	.0	.0	.0	.0	.0	100.0
LUMINOSILICATE	4.7	19.9	10.1	53.0	2.1	10.2	100.0
IXED AL-SILICA	21.7	26.3	51.9	.0	.0	.0	100.0
E SILICATE	.0	.0	.0	.0	.0	.0	.0
A SILICATE	.0	.0	.0	.0	.0	.0	.0
A ALUMINATE	.0	.0	.0	.0	.0	.0	.0
YRITE	10.8	.0	1.7	47.0	11.8	28.8	100.0
YRRHOTITE	100.0	.0	.0	.0	.0	.0	100.0
XIDIZED PYRRHO	38.6	.0	.0	.0	61.4	.0	100.0
YPSUM	8.8	75.9	15.3	.0	.0	.0	100.0
ARITE	10.2	28.3	61.5	.0	.0	.0	100.0
PATITE	.0	.0	.0	.0	.0	.0	.0
A AL-P	16.4	31.3	40.5	9.6	2.2	.0	100.0
CL	.0	.0	.0	.0	.0	.0	.0
YPSUM/BARITE	100.0	.0	.0	.0	.0	.0	100.0
YPSUM/AL-SILIC	100.0	.0	.0	.0	.0	.0	100.0
I-RICH	16.2	34.0	21.9	.0	7.5	20.4	100.0
A-RICH	.0	.0	.0	.0	100.0	.0	100.0
A-SI RICH	.0	100.0	.0	.0	.0	.0	100.0
NKNOWN	42.7	14.3	17.2	7.2	11.7	6.9	100.0
TOTALS	16.0	23.2	22.9	17.5	13.5	7.0	100.0

NUMBER OF PARTICLES IN EACH SIZE RANGE

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOTA
QUARTZ	111.0	26.0	94.0	11.0	36.0	1.0	279.
IRON OXIDE	7.0	2.0	8.0	1.0	4.0	.0	22.
PERICLASE	.0	.0	.0	.0	.0	.0	.
RUTILE	3.0	.0	.0	1.0	.0	.0	4.
ALUMINA	1.0	.0	1.0	.0	.0	.0	2.
CALCITE	2.0	1.0	2.0	.0	2.0	1.0	8.
DOLOMITE	1.0	.0	1.0	.0	.0	1.0	3.
ANKERITE	.0	.0	.0	.0	.0	.0	.
KAOLINITE	92.0	48.0	83.0	18.0	25.0	3.0	269.
MONTMORILLONITE	11.0	13.0	11.0	7.0	8.0	1.0	51.
K AL-SILICATE	3.0	2.0	3.0	.0	.0	.0	8.
FE AL-SILICATE	1.0	1.0	1.0	.0	.0	.0	3.
CA AL-SILICATE	4.0	3.0	1.0	.0	1.0	.0	9.
NA AL-SILICATE	1.0	.0	.0	.0	.0	.0	1.
ALUMINOSILICATE	5.0	4.0	5.0	7.0	1.0	1.0	23.
MIXED AL-SILICA	2.0	1.0	2.0	.0	.0	.0	5.
FE SILICATE	.0	.0	.0	.0	.0	.0	.
CA SILICATE	.0	.0	.0	.0	.0	.0	.
CA ALUMINATE	.0	.0	.0	.0	.0	.0	.
PYRITE	9.0	.0	1.0	3.0	2.0	1.0	16.
PYRRHOTITE	2.0	.0	.0	.0	.0	.0	2
OXIDIZED PYRRHO	2.0	.0	.0	.0	1.0	.0	3
GYPSUM	1.0	1.0	1.0	.0	.0	.0	3
BARITE	1.0	1.0	4.0	.0	.0	.0	6
APATITE	.0	.0	.0	.0	.0	.0	
CA AL-P	34.0	19.0	53.0	3.0	2.0	.0	111
KCL	.0	.0	.0	.0	.0	.0	
GYPSUM/BARITE	2.0	.0	.0	.0	.0	.0	2
GYPSUM/AL-SILIC	2.0	.0	.0	.0	.0	.0	2
SI-RICH	10.0	4.0	7.0	.0	1.0	1.0	23
CA-RICH	.0	.0	.0	.0	1.0	.0	1
CA-SI RICH	.0	1.0	.0	.0	.0	.0	1
UNKNOWN	72.0	7.0	16.0	2.0	5.0	1.0	103
TOTALS	379.0	134.0	294.0	53.0	89.0	11.0	960

PAGE 9 Black Thunder Coal-From EERC Blend Project

DISTRIBUTION OF MINERAL PHASES - FREQUENCY PERCENT

TOTAL NUMBER OF POINTS = 960.0

% QUARTZ = 29.1

```
AMPLE DESCRIPTION ---> Black Thunder Coal-From EERC Blend Project
UBMITTER          ---> Katrinak
CC # AND FUND #   ---> 42586132
UN DATE AND TIME  --->  11  3 1993  15:28

UMMARY OF PARAMETERS

ERCENT EPOXY USED                                  =       67.8
OTAL MINERAL AREA ANALYZED AT  800.0 MAG           =     1652.3
ORMALIZED AREA ANALYZED AT  800.0 MAG              =   194259.7
INERAL AREA ANALYZED  240.0 MAG                    =    18159.4
ORMALIZED AREA ANALYZED  240.0 MAG                 =   200047.1
OTAL MINERAL AREA ANALYZED AT   50.0 MAG           =   101628.7
UMBER OF FRAMES AT  800.0 MAG                      =         99
UMBER OF FRAMES AT  240.0 MAG                      =         99
UMBER OF FRAMES AT   50.0 MAG                      =         47
OTAL MINERAL WGHT % ON A COAL BASIS                =      2.606
OTAL NUMBER OF POINTS ANALYZED                     =        960
UMBER OF POINTS UNDER THRESHOLD                    =          9

EIGHT PERCENT ON A MINERAL BASIS
                    1.0     2.2     4.6    10.0    22.0    46.0
                     TO      TO      TO      TO      TO      TO  TOTALS
                    2.2     4.6    10.0    22.0    46.0   100.0          % EXCLUDED
-----------------------------------------------------------------------------------
UARTZ               4.3     3.7     6.8     3.5     5.0      .8    24.1    42.6   10.27
RON OXIDE            .4      .6     1.4      .9     1.3      .0     4.6    54.8   2.52 ✓
ERICLASE             .0      .0      .0      .0      .0      .0      .0      .0
UTILE                .2      .0      .0      .8      .0      .0     1.0    17.2   .17
LUMINA               .0      .0      .2      .0      .0      .0      .3    15.8   .05
ALCITE               .0      .1      .1      .0      .2      .4      .9    90.2   .81
OLOMITE              .0      .0      .0      .0      .0      .6      .7   100.0   .70
NKERITE              .0      .0      .0      .0      .0      .0      .0      .0
AOLINITE            3.9     8.4     6.2     4.8     3.8     1.9    29.1    42.8   12.46
ONTMORILLONITE       .4     2.0      .7     2.1     1.1      .8     7.1    63.3   4.49
 AL-SILICATE         .2      .4      .1      .0      .0      .0      .7    27.1   0.19
E AL-SILICATE        .0      .3      .1      .0      .0      .0      .4      .0
A AL-SILICATE        .1      .4      .1      .0      .1      .0      .8    22.9   0.18
A AL-SILICATE        .0      .0      .0      .0      .0      .0      .0      .0
LUMINOSILICATE       .2      .9      .5     2.5      .1      .5     4.6    53.2   2.45
IXED AL-SILICA       .1      .1      .3      .0      .0      .0      .5    51.9   0.26
E SILICATE           .0      .0      .0      .0      .0      .0      .0      .0
A SILICATE           .0      .0      .0      .0      .0      .0      .0      .0
A ALUMINATE          .0      .0      .0      .0      .0      .0      .0      .0
YRITE                .5      .0      .1     2.2      .6     1.4     4.7    77.9   3.66
YRRHOTITE            .1      .0      .0      .0      .0      .0      .1    46.3   0.05
XIDIZED PYRRHO       .1      .0      .0      .0      .2      .0      .2    15.3   0.03
YPSUM                .0      .2      .0      .0      .0      .0      .2    84.7   0.17
ARITE                .1      .2      .4      .0      .0      .0      .6    40.4   0.24
PATITE               .0      .0      .0      .0      .0      .0      .0      .0
A AL-P              1.6     3.1     4.0      .9      .2      .0     9.8    19.3   1.89
CL                   .0      .0      .0      .0      .0      .0      .0      .0
YPSUM/BARITE         .1      .0      .0      .0      .0      .0      .1    46.4   0.05
YPSUM/AL-SILIC       .0      .0      .0      .0      .0      .0      .0      .0
I-RICH               .3      .7      .5      .0      .2      .4     2.1     4.7   0.10
A-RICH               .0      .0      .0      .0      .1      .0      .1   100.0   0.10
A-SI RICH            .0      .1      .0      .0      .0      .0      .1   100.0   0.10
NKNOWN              3.0     1.0     1.2      .5      .8      .5     7.0    29.1   2.04
-----------------------------------------------------------------------------------
 TOTALS            15.9    22.3    22.7    18.3    13.6     7.3   100.0           42.98

SAMPLE DESCRIPTION ---> Black Thunder Coal-From EERC Blend Project
```

```
SUBMITTER              ---> Katrinak
ICC # AND FUND #       ---> 42586132
RUN DATE AND TIME      --->  11  3 1993  15:28


Percent excluded as a function of particle
size and phase.


                      1.0    2.2    4.6   10.0   22.0   46.0
                       TO     TO     TO     TO     TO     TO
                      2.2    4.6   10.0   22.0   46.0  100.0
--------------------------------------------------------------
QUARTZ               27.5   37.3   40.7   37.3   56.1  100.0
IRON OXIDE           31.2   30.3   86.7     .0   79.9     .0
PERICLASE              .0     .0     .0     .0     .0     .0
RUTILE               82.7     .0     .0     .0     .0     .0
ALUMINA             100.0     .0     .0     .0     .0     .0
CALCITE             100.0  100.0  100.0     .0   50.2  100.0
DOLOMITE            100.0     .0  100.0     .0     .0  100.0
ANKERITE               .0     .0     .0     .0     .0     .0
KAOLINITE            49.7   66.0   31.0   22.5   36.0   29.8
MONTMORILLONITE        .0   27.4   38.3   91.5   86.4  100.0
K AL-SILICATE        33.6     .0  100.0     .0     .0     .0
FE AL-SILICATE         .0     .0     .0     .0     .0     .0
CA AL-SILICATE       41.9     .0     .0     .0  100.0     .0
NA AL-SILICATE         .0     .0     .0     .0     .0     .0
ALUMINOSILICATE      29.9   87.6   62.5   29.6  100.0  100.0
MIXED AL-SILICA        .0     .0  100.0     .0     .0     .0
FE SILICATE            .0     .0     .0     .0     .0     .0
CA SILICATE            .0     .0     .0     .0     .0     .0
CA ALUMINATE           .0     .0     .0     .0     .0     .0
PYRITE               78.2     .0     .0   69.0   70.2  100.0
PYRRHOTITE           46.3     .0     .0     .0     .0     .0
OXIDIZED PYRRHO      39.5     .0     .0     .0     .0     .0
GYPSUM              100.0  100.0     .0     .0     .0     .0
BARITE                 .0     .0   65.7     .0     .0     .0
APATITE                .0     .0     .0     .0     .0     .0
CA AL-P              17.8   38.8   10.5     .0     .0     .0
KCL                    .0     .0     .0     .0     .0     .0
GYPSUM/BARITE        46.4     .0     .0     .0     .0     .0
GYPSUM/AL-SILIC        .0     .0     .0     .0     .0     .0
SI-RICH                .0     .0   21.4     .0     .0     .0
CA-RICH                .0     .0     .0     .0  100.0     .0
CA-SI RICH             .0  100.0     .0     .0     .0     .0
UNKNOWN              21.5   22.1   27.7     .0   43.5  100.0




SAMPLE DESCRIPTION ---> Black Thunder Coal-From EERC Blend Project
SUBMITTER          ---> Katrinak
ICC # AND FUND #   ---> 42586132
```

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 DATE AND TIME  --->  11  3 1993  15:28

erage phase composition.
ercent Relative X-ray Intensity)

                 SI   AL   FE   TI    P   CA   MG   NA    K    S   BA   CL
---------------------------------------------------------------------------
ARTZ           95.4   .1   .5   .5   .0   .8   .3   .2   .4   .6   .4   .6
ON OXIDE        2.6  1.5 87.5   .8   .8  1.7   .8   .5  1.2   .9   .7  1.0
RICLASE          .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
TILE             .3   .3   .3 49.6   .3  1.0   .5   .3  1.3   .8 44.9   .8
UMINA            .0 95.0   .5  1.5   .0  1.0   .0   .5   .5   .0   .5   .5
LCITE           1.1   .6  1.3   .6   .1 92.5   .3   .3   .3  1.5   .6   .9
LOMITE          4.3   .0  1.0   .7   .3 65.6 23.8   .3  1.3  1.3   .3  1.0
KERITE           .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
OLINITE        48.3 45.0   .7   .8   .2  1.0   .1   .4   .8  1.0   .8   .9
NTMORILLONITE  57.9 31.1  1.4  1.0   .2  1.6   .3   .4  2.0  2.2   .7  1.1
 AL-SILICATE   49.3 31.9  1.7   .4   .1  1.7   .0  1.0 10.6   .9  1.7   .6
 AL-SILICATE   41.2 25.2 19.3  1.3   .0  4.3  1.0  2.3   .3  2.3  1.3  1.3
A AL-SILICATE  46.2 35.1  1.2  1.2   .8  8.4   .7   .6  1.7  1.7  1.2  1.3
A AL-SILICATE  72.3 18.8   .0   .0   .0  2.0   .0  5.9   .0   .0   .0  1.0
UMINOSILICATE  56.3 34.6  1.3  1.2   .7  1.2   .1   .5  1.0   .9  1.1  1.1
IXED AL-SILICA 44.1 32.8  2.2  3.6   .4  3.4  1.4  1.0  3.4  3.0  2.4  2.6
 SILICATE        .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
A SILICATE       .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
A ALUMINATE      .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
YRITE            .9   .6 22.8   .6   .3   .6   .4   .3   .3 73.0   .3   .1
YRRHOTITE       2.0  1.5 36.4  1.5  1.0  1.5   .5  1.0  4.0 47.5  1.5  1.5
XIDIZED PYRRHO  2.3  1.3 79.1  4.3  1.0  1.0   .7  1.3   .3  6.3  1.3  1.0
YPSUM            .7  1.3   .7  1.3   .7 41.1   .7   .0  1.3 52.3   .0   .0
ARITE           2.7  1.2  1.0 26.5   .3  2.5   .2   .3  1.0 34.5 29.8   .0
PATITE           .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
A AL-P           .1 40.3  1.9  2.7 21.1 25.3   .5   .7  1.4   .3  3.0  2.8
CL               .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
YPSUM/BARITE    9.5  3.0  1.5 26.4   .0  7.5   .0  1.0   .0 30.3 20.9   .0
YPSUM/AL-SILIC 37.0 37.0  2.5   .0  1.0  8.5  1.0   .0  2.5  6.5  3.0  1.0
I-RICH         74.0 12.4  1.0  1.6   .3  1.8   .3  1.8  1.4  2.8  1.3  1.2
A-RICH          9.0  3.0  2.0  1.0  1.0 77.0  3.0   .0   .0  1.0   .0  3.0
A-SI RICH      30.0   .0   .0   .0   .0 61.0  3.0   .0   .0  6.0   .0   .0
NKNOWN         18.2 19.0  7.9  4.1 15.7 13.9  1.7  1.0  3.8  6.6  3.7  4.4
```

APPENDIX B

RUN-OF-MINE (KAISER) PITTSBURGH #8 BITUMINOUS COAL INCLUDED/EXCLUDED CCSEM DATA

(MTI)

SAMPLE DESCRIPTION ---> MTI 93-101 ABB-CE (KAISER) Pittsburgh #8 Parent Coal
SUBMITTER ---> Jones
ICC # AND FUND # ---> 48636179
RUN DATE AND TIME ---> 12 14 1993 11:58

SUMMARY OF PARAMETERS

PERCENT EPOXY USED = 55.4
TOTAL MINERAL AREA ANALYZED AT 800.0 MAG = 3674.9
NORMALIZED AREA ANALYZED AT 800.0 MAG = 1516835.0
MINERAL AREA ANALYZED 240.0 MAG = 53046.3
NORMALIZED AREA ANALYZED 240.0 MAG = 2051502.0
TOTAL MINERAL AREA ANALYZED AT 50.0 MAG = 853427.8
NUMBER OF FRAMES AT 800.0 MAG = 27
NUMBER OF FRAMES AT 240.0 MAG = 27
NUMBER OF FRAMES AT 50.0 MAG = 45
TOTAL MINERAL WGHT % ON A COAL BASIS = 16.355
TOTAL NUMBER OF POINTS ANALYZED = 2780
NUMBER OF POINTS UNDER THRESHOLD = 13

WEIGHT PERCENT ON A MINERAL BASIS

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOTALS	% EXCLUDED	
QUARTZ	2.0	4.5	5.1	2.0	1.6	1.1	16.3	87.1	14.20
IRON OXIDE	.0	.0	.2	.0	.1	.2	.5	100.0	0.50
PERICLASE	.0	.0	.0	.0	.0	.0	.0	.0	
RUTILE	.1	.2	.1	.3	.0	.0	.6	72.7	0.44
ALUMINA	.0	.0	.0	.0	.0	.0	.0	.0	
CALCITE	.2	.3	.3	.5	.4	.6	2.3	98.1	2.26
DOLOMITE	.4	.5	.5	.8	.2	.6	2.9	98.7	2.86
ANKERITE	.0	.0	.0	.0	.0	.0	.0	.0	
KAOLINITE	1.6	2.9	3.1	2.8	1.3	.4	12.0	68.9	8.27
MONTMORILLONITE	.7	.8	1.1	.7	.5	.2	3.9	83.8	3.27
K AL-SILICATE	2.8	7.1	8.7	5.1	2.8	.9	27.4	83.7	22.93
FE AL-SILICATE	.2	.6	.7	.8	.2	.0	2.4	89.3	2.14
CA AL-SILICATE	.1	.0	.0	.2	.1	.0	.3	40.2	0.12
NA AL-SILICATE	.0	.0	.0	.0	.0	.0	.0	100.0	
ALUMINOSILICATE	.1	.2	.4	.6	.2	.1	1.6	93.0	1.49
MIXED AL-SILICA	.2	.1	.3	.3	.1	.0	1.1	79.7	0.88
FE SILICATE	.0	.0	.0	.0	.0	.0	.0	.0	
CA SILICATE	.1	.0	.0	.3	.1	.1	.6	96.2	0.58
CA ALUMINATE	.0	.0	.0	.0	.0	.0	.0	.0	
PYRITE	.6	2.4	2.3	1.5	2.8	2.1	11.7	76.5	8.95
PYRRHOTITE	.1	.0	.0	.0	.0	.0	.2	100.0	0.20
OXIDIZED PYRRHO	.0	.0	.0	.2	.2	.0	.5	100.0	0.50
GYPSUM	.1	.2	.3	.4	.5	.2	1.7	99.5	1.69
BARITE	.0	.0	.0	.0	.0	.0	.0	.0	
APATITE	.0	.0	.0	.0	.0	.0	.1	100.0	0.10
CA AL-P	.0	.0	.0	.0	.0	.0	.0	.0	
KCL	.0	.0	.0	.0	.0	.0	.0	.0	
GYPSUM/BARITE	.0	.0	.0	.0	.0	.0	.0	.0	
GYPSUM/AL-SILIC	.2	.1	.1	.1	.0	.0	.5	60.5	0.30
SI-RICH	.2	.4	.7	.7	.6	.2	2.9	87.2	2.53
CA-RICH	.2	.0	.1	.1	.1	.0	.5	96.4	0.48
CA-SI RICH	.0	.0	.0	.0	.0	.1	.1	100.0	0.10
UNKNOWN	1.6	1.9	2.1	2.2	1.1	1.0	9.9	84.9	8.41
TOTALS	11.6	22.1	26.0	19.5	12.9	7.8	100.0		83.2 Inclu in Misc [illegible]

SAMPLE DESCRIPTION ---> MTI 93-101 ABB-CE (KAISER) Pittsburgh #8 Parent Coa
SUBMITTER ---> Jones
ICC # AND FUND # ---> 48636179
RUN DATE AND TIME ---> 12 14 1993 11:58

Percent excluded as a function of particle size and phase.

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0
QUARTZ	77.0	89.6	87.3	78.4	94.3	100.0
IRON OXIDE	100.0	.0	100.0	.0	100.0	100.0
PERICLASE	.0	.0	.0	.0	.0	.0
RUTILE	100.0	.0	100.0	100.0	.0	.0
ALUMINA	.0	.0	.0	.0	.0	.0
CALCITE	86.8	100.0	95.5	100.0	100.0	100.0
DOLOMITE	97.4	100.0	94.4	100.0	100.0	100.0
ANKERITE	.0	.0	.0	.0	.0	.0
KAOLINITE	59.4	67.6	66.7	64.6	89.6	100.0
MONTMORILLONITE	85.8	87.6	61.6	100.0	95.8	100.0
K AL-SILICATE	78.8	85.3	81.3	83.8	86.2	100.0
FE AL-SILICATE	75.8	90.8	93.9	86.1	93.0	100.0
CA AL-SILICATE	100.0	.0	100.0	.0	82.7	.0
NA AL-SILICATE	.0	.0	.0	.0	100.0	.0
ALUMINOSILICATE	100.0	72.8	91.0	100.0	89.9	100.0
MIXED AL-SILICA	88.6	100.0	66.4	75.1	85.5	.0
FE SILICATE	.0	.0	.0	.0	.0	.0
CA SILICATE	57.9	.0	100.0	100.0	100.0	100.0
CA ALUMINATE	.0	.0	.0	.0	.0	.0
PYRITE	73.8	63.1	46.7	91.2	86.7	100.0
PYRRHOTITE	100.0	.0	.0	.0	100.0	.0
OXIDIZED PYRRHO	100.0	.0	100.0	100.0	100.0	.0
GYPSUM	100.0	100.0	100.0	100.0	98.3	100.0
BARITE	.0	.0	.0	.0	.0	.0
APATITE	100.0	.0	100.0	.0	100.0	.0
CA AL-P	.0	.0	.0	.0	.0	.0
KCL	.0	.0	.0	.0	.0	.0
GYPSUM/BARITE	.0	.0	.0	.0	.0	.0
GYPSUM/AL-SILIC	63.0	33.7	62.5	100.0	47.0	.0
SI-RICH	75.4	91.1	75.7	91.1	94.5	100.0
CA-RICH	88.1	100.0	100.0	100.0	100.0	100.0
CA-SI RICH	.0	.0	.0	.0	100.0	100.0
UNKNOWN	79.0	92.8	68.9	87.9	90.2	100.0

```
SAMPLE DESCRIPTION ---> MTI 93-101 ABB-CE (KAISER) Pittsburgh #8 Parent Coal
SUBMITTER          ---> Jones
ICC # AND FUND #   ---> 48636179
RUN DATE AND TIME  --->  12 14 1993   11:58

Average phase composition.
(Percent Relative X-ray Intensity)

                     SI   AL   FE   TI    P   CA   MG   NA    K    S   BA   CL
--------------------------------------------------------------------------------
QUARTZ             95.1   .5   .6   .5   .0   .5   .2   .2   .8   .6   .5   .4
IRON OXIDE          2.1  1.5 87.7   .8   .6   .1   .6   .6  2.1  2.0  1.4   .4
PERICLASE            .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
RUTILE              3.5  2.0   .8 46.3   .8   .2   .2   .2  1.0   .2 44.1   .8
ALUMINA              .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
CALCITE             1.6  1.3   .9   .6   .5 90.7   .9   .3   .6  1.1   .7   .8
DOLOMITE            3.6   .6  1.8   .8   .7 72.7 15.9   .0   .9  1.2   .8  1.0
ANKERITE             .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
KAOLINITE          49.8 41.8  1.3   .9   .1   .7   .1   .3  2.1  1.3   .7   .8
MONTMORILLONITE    57.0 31.3  1.8  1.2   .1  1.0   .2   .3  3.5  1.8   .9   .8
K AL-SILICATE      52.6 29.8  2.0  1.0   .1  1.0   .3   .3 10.0  1.3   .9   .7
FE AL-SILICATE     45.1 26.9 15.9  1.2   .1  1.1  4.0   .1  2.8  1.4   .9   .4
CA AL-SILICATE     44.2 26.6  1.2  2.1  1.1 16.4   .5   .9  3.5  2.0  1.1   .4
NA AL-SILICATE     74.0 19.0   .0   .0   .0   .0   .0  7.0   .0   .0   .0   .0
ALUMINOSILICATE    65.2 25.0  1.4   .9   .1   .6   .2   .3  3.5  1.2  1.1   .6
MIXED AL-SILICA    51.1 26.4  6.2  1.7   .0  1.7  1.0   .3  7.5  2.0  1.4   .6
FE SILICATE          .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
CA SILICATE        47.0  1.1  1.5   .4   .3 42.3  3.9   .4   .9  1.4   .5   .3
CA ALUMINATE         .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
PYRITE              1.0   .8 22.2   .5   .3   .4   .2   .1   .4 73.6   .4   .0
PYRRHOTITE          2.6  1.8 40.1  1.3   .9  2.0   .9  1.0  5.0 44.4   .1   .0
OXIDIZED PYRRHO     1.4  1.0 74.0  2.3   .5  1.6   .3   .5  1.1 15.4   .8  1.1
GYPSUM              1.4  1.0   .9   .5   .6 43.8   .3   .3   .5 50.2   .6   .0
BARITE               .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
APATITE              .0  3.3  1.8   .3 24.5 66.0   .8   .5  2.3   .0   .5   .3
CA AL-P              .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
KCL                  .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
GYPSUM/BARITE        .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0   .0
GYPSUM/AL-SILIC    35.2 17.8  2.7  1.6   .4 18.5   .6   .4  3.6 17.9   .7   .6
SI-RICH            73.1 13.8  1.9   .9   .1  1.0   .2   .8  4.9  1.3  1.0   .9
CA-RICH            11.6  2.7  2.8   .9  1.2 72.0  2.9   .5  1.9  2.3   .8   .5
CA-SI RICH         43.9 10.1  2.5  2.0   .0 37.4   .0  1.5  1.5   .5   .5   .0
UNKNOWN            44.9 13.8  6.9  2.8  1.2 11.7  1.5  1.7  5.2  6.7  2.7  1.0
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PAGE 1 MTI 93-101 ABB-CE (KAISER) Pittsburgh #8 Parent Coal

AREA IN EACH SIZE RANGE

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOT
QUARTZ	230.8	508.9	6221.0	2423.9	76198.9	50571.5	13615
IRON OXIDE	1.4	.0	101.2	.0	1791.6	5839.6	773
PERICLASE	.0	.0	.0	.0	.0	.0	
RUTILE	4.1	9.6	47.8	179.3	.0	.0	24
ALUMINA	.0	.0	.0	.0	.0	.0	
CALCITE	24.9	27.7	384.9	540.6	18944.6	28043.6	4796
DOLOMITE	43.3	50.7	546.9	871.5	9000.2	26048.0	3656
ANKERITE	.0	.0	.0	.0	.0	.0	
KAOLINITE	183.6	331.4	3759.3	3393.0	59475.0	16965.8	8410
MONTMORILLONITE	80.0	101.9	1393.6	860.6	25167.0	7965.6	3556
K AL-SILICATE	324.6	820.9	10849.8	6327.9	134037.9	43882.3	19624
FE AL-SILICATE	19.9	60.8	75[illegible].5	882.8	8110.7	1938.8	1176
CA AL-SILICATE	6.0	.0	24.2	200.5	2557.1	.0	278
NA AL-SILICATE	.0	.0	.0	.0	729.7	.0	72
ALUMINOSILICATE	12.5	20.4	516.4	705.4	10688.8	2437.2	1438
MIXED AL-SILICA	28.0	15.5	373.3	375.1	4287.7	.0	507
FE SILICATE	.0	.0	.0	.0	.0	.0	
CA SILICATE	6.0	.0	26.2	348.7	5589.4	4466.5	1043
CA ALUMINATE	.0	.0	.0	.0	.0	.0	
PYRITE	39.2	143.8	1458.6	991.2	70411.9	52933.0	12597
PYRRHOTITE	8.6	.0	.0	.0	1232.3	.0	124
OXIDIZED PYRRHO	2.8	.0	18.4	141.9	4527.5	.0	469
GYPSUM	15.3	23.0	337.6	530.4	23749.6	12144.0	3679
BARITE	.0	.0	.0	.0	.0	.0	
APATITE	2.0	.0	44.9	.0	1919.9	.0	196
CA AL-P	.0	.0	.0	.0	.0	.0	
KCL	.0	.0	.0	.0	.0	.0	
GYPSUM/BARITE	.0	.0	.0	.0	.0	.0	
GYPSUM/AL-SILIC	19.6	12.7	149.1	83.6	1301.7	.0	15
SI-RICH	24.4	49.5	891.0	908.3	28110.9	7801.6	377
CA-RICH	17.9	5.2	68.6	163.1	3307.8	2334.2	58
CA-SI RICH	.0	.0	.0	.0	1242.8	3335.1	45
UNKNOWN	183.6	214.6	2462.6	2690.3	49259.3	45078.8	998
TOTALS	1278.2	2396.7	30428.2	22618.2	541642.2	311785.6	9101

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DISTRIBUTION BY % OF EACH MINERAL PHASE

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOTALS
QUARTZ	12.4	27.4	31.4	12.2	9.9	6.6	100.0
IRON OXIDE	4.8	.0	32.3	.0	14.8	48.1	100.0
PERICLASE	.0	.0	.0	.0	.0	.0	.0
RUTILE	11.8	27.3	12.8	48.0	.0	.0	100.0
ALUMINA	.0	.0	.0	.0	.0	.0	.0
CALCITE	9.8	10.9	14.2	20.0	18.1	26.8	100.0
DOLOMITE	13.9	16.3	16.4	26.2	7.0	20.2	100.0
ANKERITE	.0	.0	.0	.0	.0	.0	.0
KAOLINITE	13.4	24.2	25.7	23.2	10.5	3.0	100.0
MONTMORILLONITE	16.9	21.5	27.6	17.0	12.9	4.1	100.0
K AL-SILICATE	10.2	25.8	31.9	18.6	10.2	3.3	100.0
FE AL-SILICATE	7.7	23.5	27.3	32.0	7.6	1.8	100.0
CA AL-SILICATE	18.0	.0	6.8	56.6	18.6	.0	100.0
NA AL-SILICATE	.0	.0	.0	.0	100.0	.0	100.0
ALUMINOSILICATE	7.0	11.4	27.0	36.9	14.4	3.3	100.0
MIXED AL-SILICA	22.6	12.5	28.2	28.4	8.4	.0	100.0
FE SILICATE	.0	.0	.0	.0	.0	.0	.0
CA SILICATE	9.1	.0	3.7	49.9	20.7	16.5	100.0
CA ALUMINATE	.0	.0	.0	.0	.0	.0	.0
PYRITE	5.5	20.2	19.2	13.1	24.0	18.0	100.0
PYRRHOTITE	74.2	.0	.0	.0	25.8	.0	100.0
OXIDIZED PYRRHO	9.6	.0	6.0	46.2	38.1	.0	100.0
GYPSUM	7.4	11.1	15.3	24.0	27.8	14.2	100.0
BARITE	.0	.0	.0	.0	.0	.0	.0
APATITE	18.0	.0	38.9	.0	43.0	.0	100.0
CA AL-P	.0	.0	.0	.0	.0	.0	.0
KCL	.0	.0	.0	.0	.0	.0	.0
GYPSUM/BARITE	.0	.0	.0	.0	.0	.0	.0
GYPSUM/AL-SILIC	34.2	22.2	24.4	13.7	5.5	.0	100.0
SI-RICH	7.4	15.0	25.3	25.8	20.7	5.7	100.0
CA-RICH	30.5	8.9	11.0	26.1	13.7	9.7	100.0
CA-SI RICH	.0	.0	.0	.0	27.1	72.9	100.0
UNKNOWN	16.5	19.3	20.8	22.7	10.8	9.8	100.0
TOTALS	11.9	22.4	26.6	19.8	12.2	7.1	100.0

PAGE 8 MTI 93-101 ABB-CE (KAISER) Pittsburgh #8 Parent Coal

NUMBER OF PARTICLES IN EACH SIZE RANGE

	1.0 TO 2.2	2.2 TO 4.6	4.6 TO 10.0	10.0 TO 22.0	22.0 TO 46.0	46.0 TO 100.0	TOT
QUARTZ	108.0	67.0	182.0	18.0	109.0	16.0	50
IRON OXIDE	1.0	.0	2.0	.0	4.0	1.0	
PERICLASE	.0	.0	.0	.0	.0	.0	
RUTILE	3.0	1.0	1.0	1.0	.0	.0	
ALUMINA	.0	.0	.0	.0	.0	.0	
CALCITE	13.0	4.0	17.0	3.0	25.0	7.0	6
DOLOMITE	22.0	7.0	17.0	5.0	10.0	8.0	6
ANKERITE	.0	.0	.0	.0	.0	.0	
KAOLINITE	81.0	41.0	112.0	21.0	74.0	5.0	33
MONTMORILLONITE	39.0	13.0	36.0	5.0	30.0	3.0	12
K AL-SILICATE	151.0	97.0	309.0	48.0	181.0	16.0	80
FE AL-SILICATE	10.0	6.0	20.0	6.0	13.0	1.0	5
CA AL-SILICATE	3.0	.0	1.0	1.0	3.0	.0	
NA AL-SILICATE	.0	.0	.0	.0	1.0	.0	
ALUMINOSILICATE	5.0	3.0	13.0	3.0	14.0	1.0	3
MIXED AL-SILICA	17.0	3.0	13.0	3.0	6.0	.0	4
FE SILICATE	.0	.0	.0	.0	.0	.0	
CA SILICATE	2.0	.0	1.0	1.0	6.0	1.0	1
CA ALUMINATE	.0	.0	.0	.0	.0	.0	
PYRITE	22.0	17.0	34.0	7.0	97.0	17.0	19
PYRRHOTITE	6.0	.0	.0	.0	2.0	.0	
OXIDIZED PYRRHO	1.0	.0	1.0	1.0	5.0	.0	
GYPSUM	7.0	2.0	9.0	4.0	30.0	4.0	5
BARITE	.0	.0	.0	.0	.0	.0	
APATITE	1.0	.0	1.0	.0	2.0	.0	
CA AL-P	.0	.0	.0	.0	.0	.0	
KCL	.0	.0	.0	.0	.0	.0	
GYPSUM/BARITE	.0	.0	.0	.0	.0	.0	
GYPSUM/AL-SILIC	9.0	3.0	4.0	1.0	2.0	.0	1
SI-RICH	12.0	6.0	24.0	8.0	41.0	3.0	9
CA-RICH	9.0	1.0	3.0	1.0	6.0	1.0	2
CA-SI RICH	.0	.0	.0	.0	1.0	1.0	
UNKNOWN	101.0	27.0	74.0	16.0	74.0	11.0	30
TOTALS	623.0	298.0	874.0	153.0	736.0	96.0	278

DATE
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