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Introducing Coal-Water-Mix Fuels to the Philippines

Assessment of Project Feasibility

Volume II

Formulation of CWM Fuels from Philippine Coals

Report to the
National Economic and Development Authority
Republic of the Philippines
on behalf of the
National Power Corporation
Development Bank of the Philippines

Report by
Brookhaven National Laboratory
for
Office of Energy
U.S. Agency for International Development

April 1985

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Prepared by

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Section 1.0

SUMMARY AND CONCLUSIONS

The objective of the work described in this volume of the report was to establish the feasibility of preparing coal-water-mix fuels with acceptable characteristics for handling and combustion using Philippine coals. Five candidate coals were considered including samples from the Malangas, S. Cebu, Bislig, Bagacay, and Semirara areas.

The project was divided into four primary elements as follows:

1. Evaluation of the relative slurryability of the five candidate coals and establishing the technical issues which would permit selection of primary candidates for further evaluation.

2. Detailed studies on the formulation of coal-water-mix fuels using the primary candidates.

3. Coordination of, and collaboration with, the efforts of U.S. commercial coal-water-mix fuel manufacturers interested in participating.

4. For the fuels produced, laboratory and pilot scale evaluations were performed to determine their rheology, stability, flow properties in pipe loops, and furnace combustion characteristics.

Malangas coal is clearly the best in terms of slurry formulation, having a very low equilibrium moisture content. With this coal, slurries could be made with coal concentrations in the 65-70% (bone dry basis) range. This low equilibrium moisture content is consistent with the relative high rank of Malangas coal. Also consistent with its high rank, however, is a low volatiles content (19%). This may result in the requirement of support fuel for flame stability and somewhat lower carbon conversion efficiency than other coals.

The S. Cebu sample had a slightly higher equilibrium moisture content and a coal-water-mix fuel pepared with this coal would contain 60-65% coal. A major disadvantage of this coal is its low ash fusion temperature which has a significant impact on derating the capacity of a fuel oil boiler to be operated with a CWM fuel.

The remaining three coals are characterized by high equilibrium moisture contents consistent with their relatively low rank. Slurries made with these coals will contain 45-55% coal.

Based upon prospective coal loadings in CWM fuels prepared from Philippine coals, available information on Philippine coal resources, and on current coal production rates from the candidate coal deposits, the following two options were selected as the primary candidates for further evaluation.

1. A 100% Semirara coal-water-mix fuel.
2. A blend of 75% Semirara and 25% Malangas in the CWM fuel.

Formulation studies were performed at BNL for the two options, which considered the effects of:

- o coal concentration
- o coal particle size distribution
- o additives
- o methanol in place of water as a carrier
- o coal cleaning
- o coal blending

With the decision to use Semirara coal as the primary fuel source for the program, additional quantities were required. For this reason a second batch of Semirara coal was requested and shipped from the Philippines. The first and second sample differed somewhat in properties although the basic conclusions of the work are not strongly affected by these differences. The results of formulation studies lead to the following observations:

1. Fuels containing Semirara coal alone will contain about 50% coal on a bone-dry basis. Since this is a low rank coal it is hydrophilic in nature and as a result it is not practical to increase the dry coal concentration through the use of dispersant additives.

2. With the replacement of some of the water used to slurry the coal by methanol, the fuel mixture's coal concentration will not be significantly reduced. The primary advantages of alcohol use in the mix should be seen as those of increased fuel heating value and improved flame stability during combustion.

3. Benification tests on one batch of Semirara coal indicated good ash removal and Btu recovery with float and sink procedures in the high specific gravity range. Coal floated at this high specific gravity showed a reduced equilibrium moisture content which lead to an increase in dry coal loading potential (55% coal vs. 50% coal on a bone-dry basis). This result was, however, not repeated on the second batch of Semirara coal. This experience focuses attention on perspective Semirara coal variability and the relevance of its effects on the eventual supply of Semirara coal to a commercial fuel formulation facility. Further evaluation of the effects of Semirara coal variability on CWM fuels prepared from it should be part of the implementation of an eventual commercial scale project.

4. In the Western U.S., a 48% dry coal slurry is transported 273 miles through the Black Mesa pipeline under an essentially turbulent mode of flow. For a similar type of transportation mode, a Semirara slurry would contain 42% dry coal.

5. Coal-water-mix fuels formulated with the 75% Semirara/25% Malangas blend will have coal concentrations in the 56-59% range. The Malangas coal is naturally hydrophobic and as a result a dispersant is required to produce this fuel formulation. BNL used .1% sodiumlignosulfonate in the fuel formulation.

6. The Energy Research Center of the University of North Dakota performed drying tests on Semirara coal to reduce its inherent moisture content using its Hot Water Drying Process now under development. With this process, dry-coal loading potential is increased to 56% coal from 50% coal.

Based on these results, the following fuel formulation has been suggested as the fuel upon which work should be based toward the assessment of the project of introducing coal-water-mix fuels to the Philippines.

o Concentration	49.5% dry coal
o Particle size distribution	more than 75% less than 200 mesh more than 99% less than 50 mesh
o Additives	3% Ammonia 1000 ppm Formaldehyde
o Viscosity	875 cp at 100 sec^{-1}

The following commercial coal-water-mix fuel manufacturers participated in this program.

The Babcock and Wilcox Company
Coaliquid, Inc.
Methacoal Corp.
Atlantic Research Corp.
Foster-Wheeler Energy Corp. (with Carbogel, Inc.)
Morrison-Knudsen Company (with Snamprogetti, Inc. of Italy)
OXCE Fuel Company

Each of these companies was offered a 400 lb. sample of the Semirara coal and asked to prepare a 50 gallon slurry fuel sample for pipe loop and combustion tests at BNL. The first three companies on the above list responded to this request. Three of the remaining four companies submitted small (1 gallon or less) samples for rheology and concentration tests. Samples of the baseline formulation were prepared at BNL for comparison with these commercial fuels.

The coal-water-mix fuel samples submitted by the commercial groups ranged in concentration from 33 to 51% dry coal and viscosity ranged from 250 to 1850 centipoise. With one exception, all of the fuels were very close to 50% concentration. The 33% coal case was clearly an anomaly and appeared to result from unique institutional, rather than technical, problems.

The short time frame available for the work, combined with the limited amount of coal available and the voluntary nature of the collaboration of the commercial CWM fuel manufacturers, prevented each company from determining a fuel formulation that would be an optimum balance between performance and cost. Instead, properties of the baseline fuel formulation were used as a standard against which properties of the manufacturers' fuel samples were compared. The objectives are to show: (1) that the baseline fuel represents a conservative basis on which to assess the project and (2) that later emphasis on the services of commercial fuel manufactures to develop an optimum fuel formulation could only enhance the attractiveness of the project.

The three larger samples of CWM fuel were in storage approximately 1 month before arriving at BNL. One of these samples arrived with a hard packed coal layer on the bottom which was very difficult to resuspend. Another arrived with nearly uniform concentration from top to bottom in the shipping drum. This second fuel was later modified by its manufacturer, reducing viscosity to facilitate combustion tests. The modification greatly reduced this

fuel's stability. During the pilot tests performed at BNL, continuous agitation of the fuels was, in general, not required. Provision for agitation, in the storage tanks of a large commercial plant, or during storage while in transit is strongly recommended.

Piping loop tests were performed on two of the three large samples received. With one of these fuels excellent agreement between viscometer and pipe flow properties was found. For the second fuel, good agreement was observed at lower shear rates but some discrepancy was found at high shear rates. In general, the flow properties of these fuels are seen to be predictable. Further pipe loop tests with the final fuel formulation as selected for the ultimate large scale project use are clearly required.

For all fuel formulations, including the baseline fuel, simplified techniques for calculating pressure drops in piping systems were developed. Some of the fuels have rheological behavior which makes pressure drop calculation more complex and, in these cases, the suggested expressions are only approximate, valid over specified shear rate ranges. Illustrative calculations are performed for flow in a 1 inch, 10 inch and a 16 inch diameter pipeline.

Combustion tests were performed with the three large sample commercial fuels, as well as with the baseline fuel, at a 1 million Btu/hr scale. The key issue in these tests was seen as the conditions under which fuels with about 50% dry coal concentrations could be made to burn. Almost all of the recent U.S. experience with coal-water-mix fuels has been with 70% coal slurries.

Two of the commercial fuels, as well as the baseline fuel, were successfully burned with combustion air preheated to 500°F and without supplementary fuel support. Preheating was accomplished using a direct oil-fired heater which reduced the combustion air oxygen content from 21 to 19.2%. Oil co-firing with the coal-water-mix fuel was used for initial light off only. The burner system produced high air swirl for flame stability. This high swirl was achieved with a pressure drop across the burner of 10-12 inches of water. The third commercial fuel used in combustion tests contained greater than 50% methanol, and this fuel burned without preheated combustion air.

The results of the combustion tests indicate that coal-water-mix fuels made with Semirara coal can be burned in a reasonable manner. As a step in implementation of a commercial project, combustion trials on a large scale with the burner to be ultimately used is a requirement.

Section 2.0

INTRODUCTION

For a coal-water-mix fuel to be practical it must have the capability of being stored, handled, and burned in a routine manner. It is desirable for the fuel to have as high a useful heating value as possible. The properties of coal-water mix fuels relating to these considerations are determined by both the feed coal properties, and by chemical additives. This section reviews general slurry formulation considerations as well as the objectives of the work performed at BNL.

2.1 General Consideration in Coal Selection for Slurry Preparation

An ideal coal for coal-water-mix fuels would have the following features:

- low equilibrium moisture
- low ash content
- high heating value
- high ash fusion temperatures
- high volatiles content

Equilibrium moisture is a measure of a coals tendency to bind water. This bound water is unavailable to separate coal particles during fuel formulation in order to improve flow properties. For a given slurry viscosity, a coal with a higher equilibrium moisture will have a lower dry coal concentration. Coals which have been used for much of the recent U.S. coal-water-mix fuel work have had equilibrium moisture contents under 3%.

Low ash content is desirable for increased fuel heating value and reduced transport and ash disposal costs. High ash fusion temperatures reduce the boiler derating required because the likelihood of fouling by molten ash (slag) is reduced.

High volatiles content is needed to ensure flame stability. While this is true even with pulverized coal firing, it is more critical for coal-water-mix fuels where the evaporating water imposes an additional thermal drain on the flame in the critical early region of combustion. A minimum of 30-35% volatiles is generally considered reasonable for a coal to be used in coal-water-mix fuel formulation.

2.2 Loading, Stability and the Role of Additives

Additives are included in commercially produced slurries for the following purposes:

- dispersion
- prevention of biological degradation
- stability against sedimentation
- pH control

Coals can be broadly classified as hydrophilic or hydrophobic. High-rank coals tend to be hydrophobic while low-rank coals tend to be hydrophilic. Hydrophobic coals mixed into water tend to agglomerate, often bringing along air bubbles. If the hydrophobic coal is fine enough, the mixture will "settle" with a foamy coal layer on the top and a water layer on the bottom. This phenomenon is the basis of froth floatation coal cleaning. In coals of this type, a dispersant (or wetting agent) is needed to achieve high coal loadings in a coal-water-mix fuel.

Once dispersed, coal loading in a slurry can be improved further by carefully tailoring the particle size distribution. The objective is to optimize the efficiency of packing in the slurry. Some manufacturers have elected to achieve high coal loadings by using parallel course and fine grinding trains, followed by mixing of the two streams.

Biocide additives (e.g. formaldehyde) are used to prevent degradation of other additives and to ensure stability of slurry properties over time. One slurry manufacturer reported rupturing of sample containers in the laboratory during a formulation development due to biological activity.[1]

Additives which provide stability against sedimentation include natural gums (e.g. xanthan gum, gaur gum) and polymers (e.g. carboxymethylcellulose). With these additives a structure is imparted to the suspension evident by the presence of a yield stress and thixotrophic behavior. (The rheology of slurries is further discussed in section 4 of this report). Generally, stability additives have the undesirable property of increasing viscosity and hence pressure drops during flow in pipelines.

Proper control of pH optimizes the performance of the coal and stabilizer combination. Typical slurries range from pH 5 to pH 8.

2.3 Organization of the Report

The basic objective of the BNL work was to establish the feasibility of preparing coal-water-mix fuels, using Philippine coals, with acceptable characteristics for handling and combustion.

Initially, the work involved five coals which were considered to be candidates for this program. The properties of these coals and the results of comparative slurryability tests are discussed in section 3.0. As the project developed, a single coal (Semirara) was selected as the primary candidate with a second coal (Malangas) being considered for blending (up to 25% Malangas). The basis for this selection was partly technical and partially a result of a review of coal resources (Volume I of this study). The technical factors contributing to the selection can be found in Section 3.

Fuel formulation efforts included two basic approaches: 1) preparation of slurries by cooperating commercial organizations and 2) support studies performed at BNL.

The flow properties (rheology) of coal water mix fuels are significantly different from the familiar Newtonian behavior. The general nature of this behavior and the measurement techniques used are described in section 4. Support studies performed at BNL included examination of the effects on slurryability of:

- coal concentration
- additives
- particle size distribution
- use of methanol as a carrier
- coal cleaning
- coal blending

Results of these studies are described in section 5. The arrangements with the commercial groups and the basic properties of the fuels produced are discussed in section 6.

Two of the participating manufacturers submitted samples large enough to allow testing in a pipe loop circuit and combustion testing at a 1 million Btu/hr scale. Slurries were also prepared at BNL to supplement the available fuel supplies for testing. Experimental details and results of the loop tests are discussed in section 7. The combustion test results are discussed in section 8.

Section 3.0

PROPERTIES OF THE CANDIDATE COALS

On June 21, 1984 samples of five candidate coals arrived at Brookhaven from the Philippines. Basic analysis of these coal samples was provided by the USGS. Each of the coals was ground, size classified, and basic slurryability was evaluated. Results of these studies are discussed in this section along with the relative merits of the coals as CWM fuel feedstock.

3.1 Coal Analyses

The analyses of the received coal samples are summarized in Table 1. In Appendix I of this report the procedure used to obtain these samples at the mine is described. The Malangas coal is the only coal with a very low equilibrium moisture content and from this perspective might be expected to be an excellent CWM feedstock. In addition, the high fusion temperature of the Malangas ash would minimize fouling and slagging problems in boilers. A major disadvantage is its low volatiles content. Support fuel could be required for flame stability and carbon conversion efficiency could be lower (relative to

Table 1

COAL ANALYSES
(USGS, 1984)

	S. Cebu	Malangas	Bislig	Semirara	Bagacay
Proximate					
As Received:					
Ash %	4.43	15.18	14.49	8.86	11.78
Moisture %	9.93	1.94	18.92	25.66	33.11
Volatiles %	42.35	19.63	30.73	32.08	28.23
Fixed Carbon %	43.29	63.25	35.86	33.40	26.88
Higher Heating Value (Btu/lb)	12,212	12,909	8,209	8,209	6,662
Equilibrium					
Moisture %	5.57	2.10	18.96	25.96	32.27
Free Swelling Index	1.0	5.0	0	0	0
Hardgrove Grindability	46	101	43	42	47
Sulfur:					
Total	1.74	0.49	0.57	0.58	5.44
Sulfate	0.04	0.00	0.01	0.03	0.04
Pyritic	0.77	0.02	0.20	0.15	1.91
Organic	0.93	0.47	0.36	0.40	3.49
Ash Fusion- Temperature (°F) (reducing atmosphere)					
Initial Deformation	2050	2600	2530	2300	2250
Softening	2130	2680	2620	2370	2360
Fluid	2160	2720	2660	2410	2400

the other coals) in a given combustion situation. To illustrate further the differences in combustion characteristics, the TGA (Thermogravimetric Analysis) burning profiles for the coals are shown in Fig. 1. In this test a small sample of powdered coal is placed on a microbalance in a furnace. The temperature of the sample is increased from ambient at a controlled rate (12°C/min) in the presence of air, and the sample weight recorded. The temperature range over which the coal burns gives a measure of combustibility. A "good" coal would ignite and burn out at lower temperatures. As this figure shows, the Malangas coal sample ignites and burns out at a much higher temperature than the other coals.

The South Cebu sample has two positive features, low ash content and moderate to low equilibrium moisture. The ash fusion temperatures are, however, very low and fouling and slagging problems could be expected. This would result in severe derating on a converted oil designed boiler.

The remaining three coals have high equilibrium moisture contents which would limit dry coal concentration in a coal-water-mix fuel.

3.2 Relative Slurryability

Each of the coals was dry ground using a hammer mill and sieved into size fractions. From these fractions a standard "power plant" grind was assembled (size distributions are discussed in section 5.1) and slurries were produced. For each of the coals the effect of a dispersant (sodiumlignosulfonate) was explored. For all of these tests, viscosity was measured using a simple concentric cylinder viscometer (Brookfield Engineering Co.). While this viscometer is particularly useful for comparative measurements, its disadvantage is a limited shear rate range. The slurries at this point in the program were unstabilized and, in general, settling occured during viscosity measurements. For this reason only qualitative results are included in this section.

The Malangas coal had a relatively high viscosity without the dispersant and a very low viscosity after dispersant addition. The Bislig, Semirara and Bagacay coals all had high viscosity before addition of the dispersant. Viscosity increased slightly after the dispersant was introduced. The South Cebu coal had a moderately high viscosity without a dispersant and its viscosity dropped slightly after addition of the dispersant.

This behavior points out the relative hydrophobic/hydrophilic nature of these coals. The Malangas coal, being higher in rank, is naturally hydrophobic. As a result, it benefits significantly from the dispersant. The three lower-rank coals are hydrophilic and so the dispersant addition does not affect their state of aggregation. The dispersant was added as a dry powder and so increased the effective solids concentration in the slurry and this likely led to the slight viscosity increase.

Based upon all of the tests it may be concluded that the Malangas coal could be used to make a very highly loaded slurry (65-70% coal). The South Cebu coal would have a slightly lower coal loading. The three lower rank coals could be loaded in the 45-55% range, which is significantly lower than has been used in recent combustion tests with U.S. coal-water-mix fuel formulations.

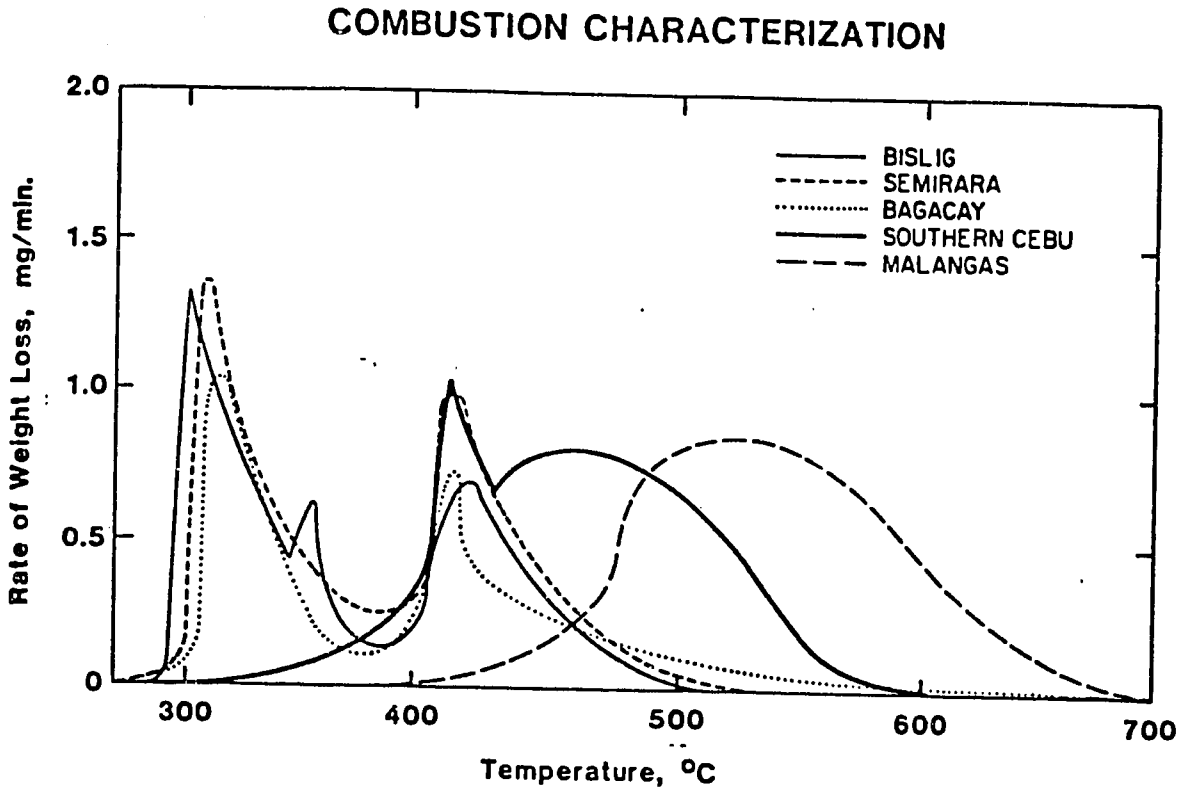

Figure 1. Coal TGA Profiles

3.3 Selection of Coals for Further Work

In a meeting held at BNL during August 1984, the results described in sections 3.1 and 3.2 were reviewed along with coal resource data. In attendance were representatives from all groups participating in the project. At that time a decision was made to aim subsequent work on two alternative feed coals 1) Semirara alone and 2) a blend composed of a maximum of 25% Malangas coal, the remainder Semirara coal. The large proven reserves of Semirara coal and the current mining production rate were key factors in the decision to emphasize this coal.

The resource base for the two coals (as known August 1984) are as follows:

	Resource Potential (MT)	Proven Reserves (MT)
Semirara		
Semirara Island	550,000,000	131,800,000
South Mindora	100,000,000	4,000,000
Malangas	45,000,000	18,600,000

The Malangas coal was included because of its excellent slurryability. At this point in the program a second batch of the Semirara coal was requested from the Philippines. This second sample arrived on September 27, 1984. As with the first shipment, this coal arrived in excellent condition, well-sealed in drums.

3.4 Detailed Analyses Malangas and Semirara Coals

With the selection of these two coals further analysis of properties was performed. The measured ash fusion temperatures are listed in Table 2 and the detailed ash analyses are listed in Table 3. The proximate and ultimate analysis, of both Semirara coal samples, (Batch 1 analysis repeated from Table 1 for convenience) are shown in Table 4. Inspection of these results indicates a significant difference between the two Semirara samples. Note, for example, the very low ash content of the second Semirara batch and the very high sodium content (12% vs. 2% in batch 1).

Ash fusion temperatures were also measured for a blend of 25% Malangas coal and 75% Semirara coal. Results, shown in Table 5, indicate that fusion temperatures for the blend fall between the two parent coals.

Table 2

ASH FUSION TEMPERATURES-MALANGAS AND SEMIRARA

	Malangas	Semirara (Batch 1)	Semirara (Batch 2)
Reducing (°F)			
Initial Deformation	2566	2101	1892
Softening	2672	2118	2051
Hemispherical	2673	2206	2138
Fluid	>2800	2300	2549
Oxidizing (°F)			
Initial Deformation	2640	2239	2200
Softening	2693	2332	2300
Hemispherical	2733	2422	2337
Fluid	>2800	2541	2553

Table 3

COAL ASH ANALYSIS

	Malangas	Semirara (Batch 1)	Semirara (Batch 2)
Silicon Dioxide	47.20%	48.56%	21.72%
Aluminum Oxide	29.94%	27.72%	18.80%
Iron Oxide	4.39%	4.58%	4.95%
Titanium Dioxide	1.20%	.98%	0.67%
Calcium Oxide	1.65%	1.79%	6.71%
Magnesium Oxide	1.[illegible]%	3.69%	10.94%
Sodium Oxide	.89%	2.15%	11.92%
Potassium Oxide	.49%	1.21%	1.95%
Lithium Oxide	440.2 ppm	155.5 ppm	293.9 ppm
Phosphorus Pentoxide	.67%	.24%	0.35%
Sulfates	8.27%	6.41%	22.13%
Loss on Ignition	84.75%	87.61%	--

Table 4

PROXIMATE AND ULTIMATE ANALYSES - SEMIRARA COAL

	Batch #1	Batch #2
Proximate (% as received)		
Moisture	25.66	29.12
Ash	8.86	4.27
Volatiles	32.08	32.43
Fixed Carbon	33.40	34.18
Sulfur	0.58	0.43
Btu/Lb	8209	8560
Ultimate (% dry)		
Carbon	64.50	68.39
Hydrogen	4.89	5.28
Nitrogen	1.22	1.11
Oxygen	16.69	18.58

Table 5

ASH FUSION TEMPERATURES - BLEND OF 75% SEMIRARA (BATCH 1) AND 25% MALANGAS

	Reducing	Oxidizing
Initial Deformation Temperature	2304	2417
Softening Temperature	2422	2468
Hemispherical Temperature	2462	2530
Fluid Temperature	2591	2599

Section 4.0

EVALUATION OF SLURRY RHEOLOGY

Simple Newtonian fluids are characterized by a shear stress/shear rate relationship of the form

$$\tau = \mu\dot{\gamma}$$

where τ = shear stress
μ = viscosity
$\dot{\gamma}$ = shear rate

A simple Newtonian fluid can be thought of as moving in very thin layers, one on another. The viscosity then is a measure of the amount of energy required to move one fluid layer past another fluid layer. The shear rate is a measure of the speed at which intermediate layers of fluid move with respect to each other. The viscosity of a simple Newtonian fluid is independent of both time and shear rate and is dependent only on temperature. Relationships for flow of conventional boiler fuel oils fit this characterization.

Inelastic non-Newtonian fluids are classified as time independent or time dependent. For time independent fluids, numerous models relating shear stress and shear rate are available including:

Power Law	$\tau = k\dot{\gamma}^n$	(pseudoplastic if $n<1$ dilatant if $n>1$)
Bingham Plastic	$\tau = \tau_o + k\dot{\gamma}$	
Yield Power Law (Herschel-Bulkley)	$\tau = \tau_o + k\dot{\gamma}^n$	

Time dependent behavior is broadly classified as thixotropic (shear thinning with time) or rheopectic (shear thickening).

The coal-water-mix fuels examined during this study were, in general, pseudoplastic and thixotropic. The time effects can be isolated in several ways. One common method is to use a viscometer with a variable shear rate; increasing the shear rate at a programmed rate from rest to a preset maximum. The shear rate is held at this maximum rate for a time (possibly zero time) and then returned at a set rate to zero. The difference between the shear stress measured during the increasing and decreasing shear rate periods is taken as a measure of thixotropy.

One alternative approach is to fix shear rate and record measured shear stress vs. time. While both techniques have been used during this program, and both are useful, more emphasis was placed on the second technique. Under a steady shear (typically 100 sec^{-1}) the measured shear stress generally decreases from an initial value to a steady state value (stable slurries only) within several minutes. After this value has been reached the shear rate is varied to generate a shear stress/shear rate relationship.

Two viscometers were used for rheology studies during this program. One was a relatively simple concentric cylinder viscometer with stepped speed control (Fann Instrument Corporation - Model 35A). With this viscometer only the second approach to evaluating time dependent effects could be used. Two cylinder gap sizes were used - 1.17 mm and 6.14 mm.

The second viscometer used was a Rheotron (Brabender OHG Duisberg) located at the Rheology Laboratory of the State University of New York at Stony Brook. All measurements were made by the Stony Brook staff. This viscometer has a very broad shear rate range (10^{-3} to ? x 10^{+4} nominal sec^{-1}), accurate temperature control, and continuous programmable shear rate variability.

In both viscometers a modified power law approach was used to calculate the shear rate corresponding to a given cylinder RPM. With this approach shear rate is calculated as

$$\dot{\gamma} = \frac{2N\omega}{1-S^{-2N}}$$

where ω = rotating cylinder angular velocity
S = ratio of outer cylinder radius to inner cylinder radius
N = slope of the log of the angular velocity plotted against the log of the torque/unit effective length of the inner cylinder

The value of N is determined at each rotating speed from a quadratic curve fit of the basic torque/rotating speed data. For fluids with a yield stress caution must be taken to ensure that the yield stress is exceeded everywhere in the gap when using the modified power-law method.

In general agreement between the two different gap sizes employed with the BNL viscometer and the viscometer used at Stony Brook (gap = 3mm) was very good. In some cases, however, there was a clear trend toward lower apparent viscosity with decreasing gap size. This difference becomes more pronounced as slurry concentration increased. To illustrate (an exaggerated example) a slurry was prepared which was much too thick to be considered for fuel use and its shear stress/shear rate relationship was measured using the BNL viscometer. Results for the two gap sizes are shown in figure 2. This type of behavior could be caused by the formulation of a slippage layer.

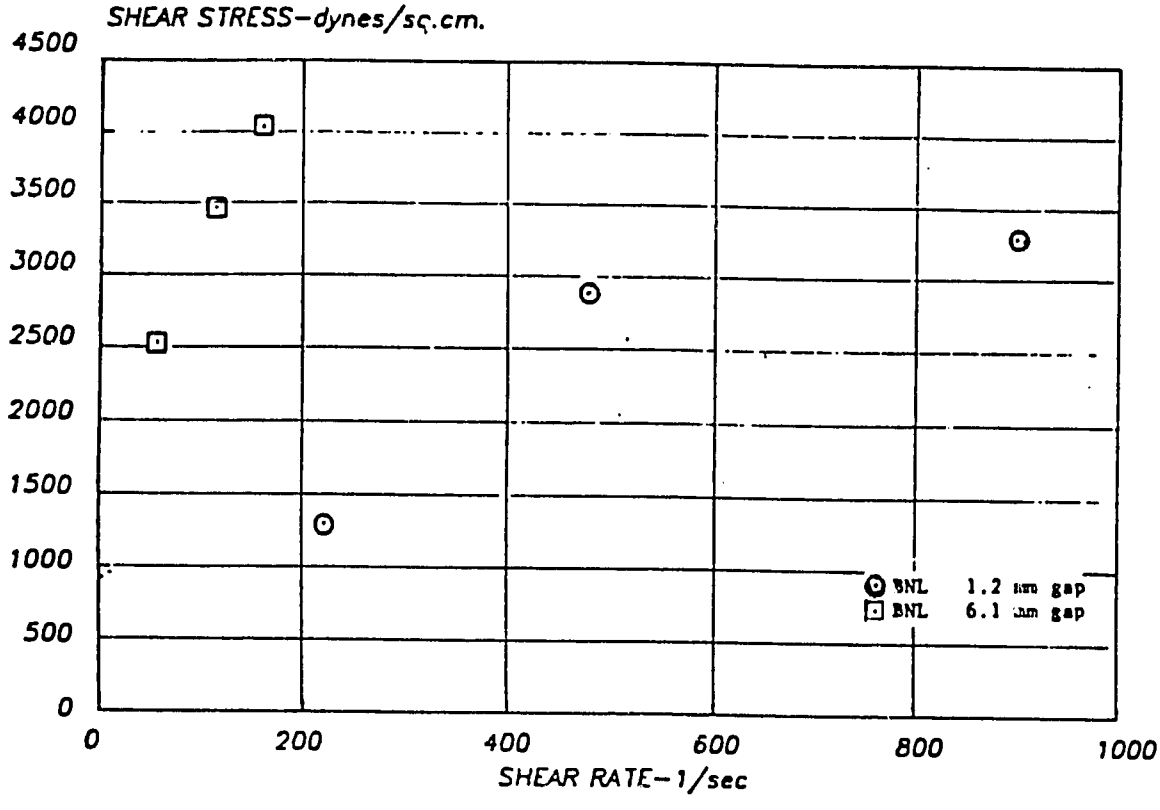

Figure 2. Example of Discrepancy Between Viscometer Gap Sizes

Section 5.0

BASIC SLURRY PREPARATION STUDIES

In this section results are described of studies performed at BNL on the formulation of CWM fuels using Semirara coal and Semirara/Malangas blends. These studies were performed to 1) establish the properties of the baseline fuel for use in the assessment of the project and 2) to provide a basis for comparing the properties of commercially prepared fuels.

5.1 Coal Particle Size Distribution for Slurry Preparation

Four size distributions used in this program are listed in Table 6. Distribution number 1 is typical of pulverized coal fired boiler practice. The second distribution is somewhat coarser and broader and is intended to simulate a grind which might be used in slurry manufacture. Size distribution number 3 is a simulation of the coarse grind slurry test fired in a cyclone boiler in the early 1960's. These tests were performed at the E.H. Werner station of the Jersey Central Power & Light Company in 1961. For these tests the slurry was concentrated to 70% coal. The interest in the present program was simply assessing the improvements in loading which could be achieved with such a size distribution. This slurry could only be burned directly in a cyclone furnace unless regrinding were done.

For the purpose of preparing slurries in larger quantities a drum of Semirara (batch 2) coal was sent to a contractor for grinding. The size distribution requested was number 1 in Table 6. The distribution received was number 4 in Table 6. This is significantly finer than desired but it did serve the purpose of providing fuel for testing.

In addition to the distributions listed in Table 6 a fifth size distribution was prepared to simulate that currently being used in the Black Mesa pipeline.[2] The specified size distribution is listed in Table 7.

Table 6

COAL SIZE DISTRIBUTIONS
% BY WEIGHT IN SIZE FRACTIONS

Size	Distribution Number 1	2	3	4
+8 Mesh	0	0	0	0
8 x 20	0	0	15.0	0
20 x 30	0	2.0	11.3	0
30 x 40	0	5.0	8.7	0
40 x 50	0	7.3	9.3	0.4
50 x 70	1.0	8.1	9.2	.7
70 x 100	4.0	15.1	6.3	1.6
100 x 140	11.0	4.9	2.7	4.4
140 x 200	14.0	7.4	3.3	5.1
200 x 270	17.0	7.1	3.4	8.8
-270	53.0	43.0	32.8	78.9

Table 7

SPECIFIED SIZE DISTRIBUTION FOR THE BLACK MESA PIPELINE
(Data from Ref. 2)

+14 mesh	0-2
-100 mesh	35-45
-325 mesh	18-20

5.2 Flow Properties of Semirara Slurries

Preliminary tests indicated that there was very little difference between slurries prepared using size distribution numbers 1 and 2 (Table 6). Most slurry formulation studies were performed using distribution number 2. A plot of apparent viscosity, at 100 1/sec, vs. coal concentration for Semirara Batch 1 is shown in Figure 3. Plots of shear stress vs. shear rate at two different coal concentrations are shown in Figure 4. No additives were included in this formulation.

Based upon this data and early discussions with slurry manufacturers a goal for concentration was set at 55%. It was assumed that with more development this could be the maximum practically attainable.

In attempting to increase slurry loading several surfactants were tested to evaluate their effect on viscosity at a constant slurry coal concentration. In addition to sodiumlignosulfonate this included Triton X-100, Aerosol OT-75 and Ammonium Bromide. In all cases no significant reduction in viscosity was observed. Tests were also done to evaluate the effect of changing pH. By adding acids (HCL) or bases (NaOH) to the water used for slurry preparation the pH of this water was varied from 4 to 10. Again, no significant effect on viscosity was observed.

The effect of drying the coal on slurry viscosity was evaluated using coal which was oven dried at 80°C. Slurry made with this coal had about 30% lower viscosity initially. Over a period of 24 hours the viscosity climbed back up to the undried value.

A sample of batch 1 of the Semirara coal was submitted to the University of North Dakota Research Center to evaluate potential benefits from Hot Water Drying. This process for irreversible reduction in the equilibrium moisture content of low rank coals has been under active development at the University for several years. The process involves exposing the slurry to high pressure and temperature conditions for a brief time period. Results of these tests, which are described in detail in Appendix II of this report, indicate that coal slurry concentration can be increased to 56.6%. An evaluation of the costs of the process in general is currently being performed by the University.

A plot of apparent viscosity, at 100 1/sec, vs. concentration for the Semirara Batch 2 with coal size distribution 4 (Table 6) is shown in Figure 5. Comparison of the overlap region of this curve with the data shown in Figure 3 indicates only small differences.

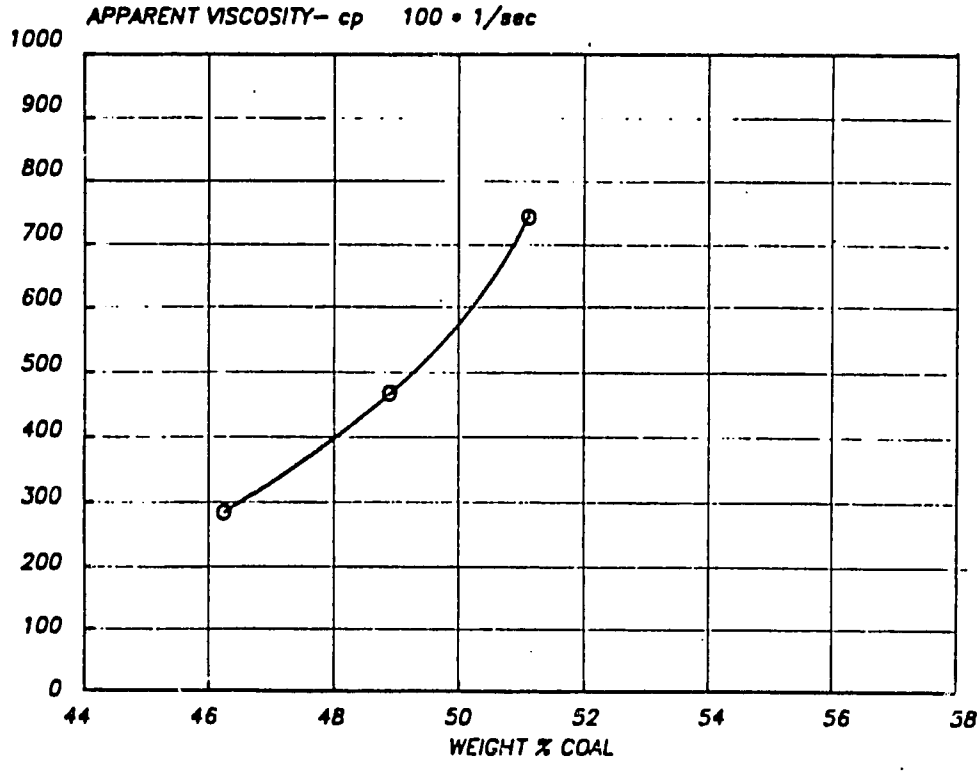

Figure 3. Apparent Viscosity vs. Slurry Coal Concentration Semirara Coal

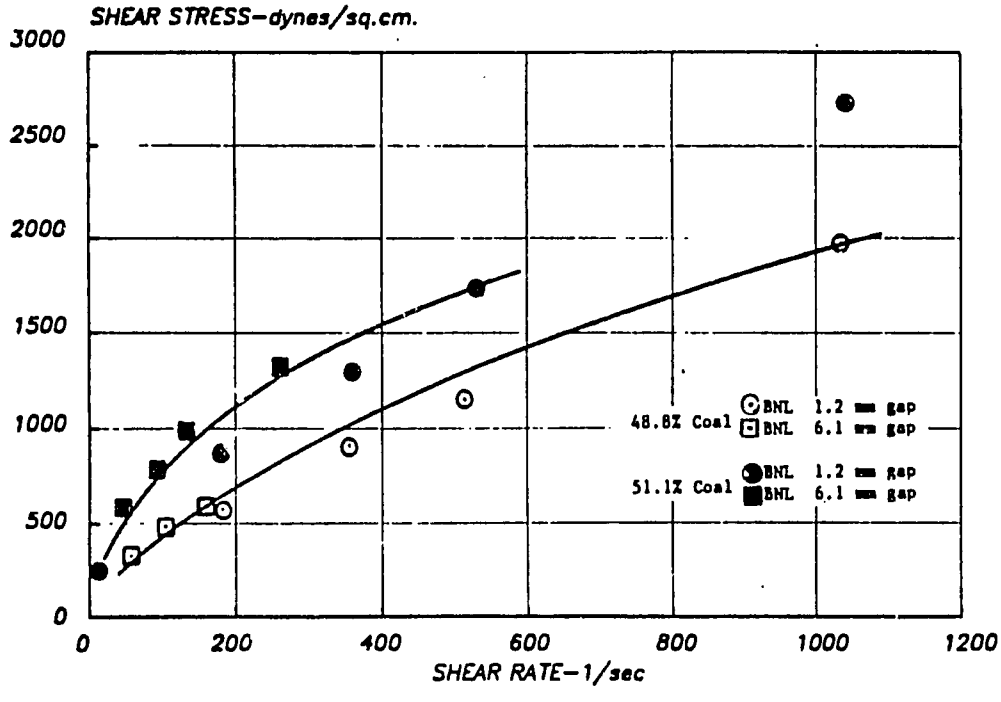

Figure 4. Flow Curves - Semirara Slurries

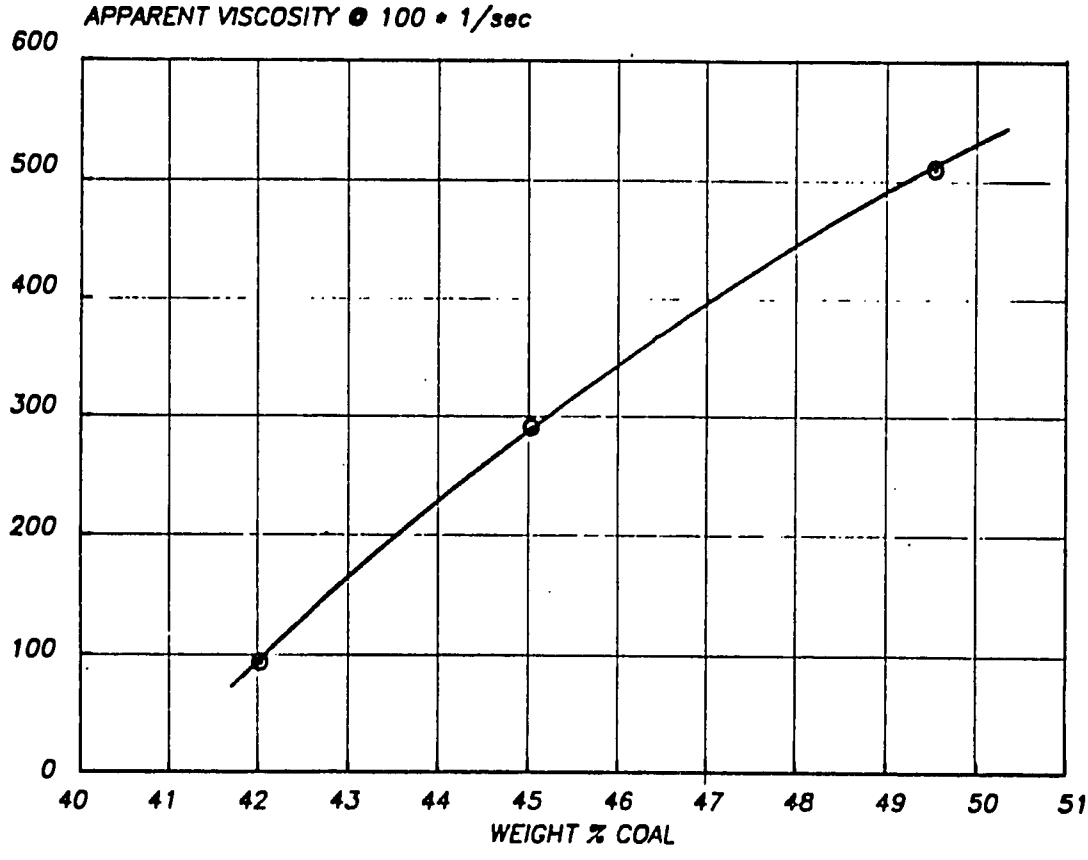

Figure 5. Apparent Viscosity vs. Concentration - Size Distribution No. 4 - Semirara Coal (Batch 2)

Tests were performed using the coarse size distribution listed in Table 7 to find the coal concentration at which coal might be transported in a mode similar to the Black Mesa slurry pipeline. This is a coarse slurry pipeline transporting a slurry which settles rapidly. To do this with the Semirara coal these tests showed that the concentration would be about 42%. This gives a viscosity of approximately 50 cp. Under these conditions the coal settles rapidly and for this reason detailed rheology is very difficult to determine. It is interesting to note however, that comparison with Figure 5 indicates a small difference in viscosity at 42% concentration between the very coarse and the very fine slurry. This suggests an option for pipeline transport which is turbulent flow of a fine coal dilute slurry followed by concentration at the use site.

Studies done using the coarse, broad size distribution listed as column number 3 in Table 6 showed that slurries in the 56% coal range could be achieved. As mentioned in section 5.1, however, this fuel could only be burned in a special furnace. As with the pipeline slurry described above, this slurry settles rapidly and rheological measurements are difficult.

All of the slurries described up to this point were unstabilized. In preparing fuel samples for combustion trials evaluations were made of the effects of several additives on stability against settling. This included xanthan gum (trade name - Kelzan, manufactured by the Kelco Division of Merck and Co., Inc.), corn starch (trade name - Hamaco 267, manufactured by the A.E. Staley Manufacturing Co.), carboxymethylcellulose (CMC), ammonia, and formaldehyde. For the purposes of preparing a fuel sufficiently stable for combustion trials it was found that a formulation containing 3% ammonia, 1000 ppm formaldehyde and using size distribution number 4 in Table 6 was adequate. The slurry produced had a viscosity of 875 cp at 100 1/sec and 49.5% coal. Its shear stress/shear rate relationship as measured at BNL is shown in Figure 6. The rheological data for this fuel mix could be reasonably well fit (least squares) with a power law expression of the form

$$\tau = 130.7\ \dot{\gamma}^{0.413}$$

where τ = shear stress, dynes/cm^2
$\dot{\gamma}$ = shear rate, 1/sec

This slurry formulation has been used as a baseline slurry for comparison with the commercially produced slurries (section 6).

5.3 Methanol as a Carrier

The advantages of incorporating methanol into the fuel-mix are seen as 1) increased fuel higher heating value and 2) flame stability. To illustrate, Figure 7 shows the higher heating value of a slurry containing 50% coal and 50% carrier. The carrier composition is varied from 100% water to 100% methanol.

To evaluate the effect of methanol addition to the carrier liquid viscosity tests were performed with specific formulations. Figure 8 shows the shear stress/shear rate relationship for a fuel containing 47.5% coal, 42.5% methanol and 10% water. This could represent a mixture of Semirara coal with industrial grade methanol. In Figure 9 is shown a mix with 50.3% coal, 8.2% methanol and 41.5% water. In this case the methanol could be considered as an additive, providing improved flame stability. From Figure 8 the apparent vis-

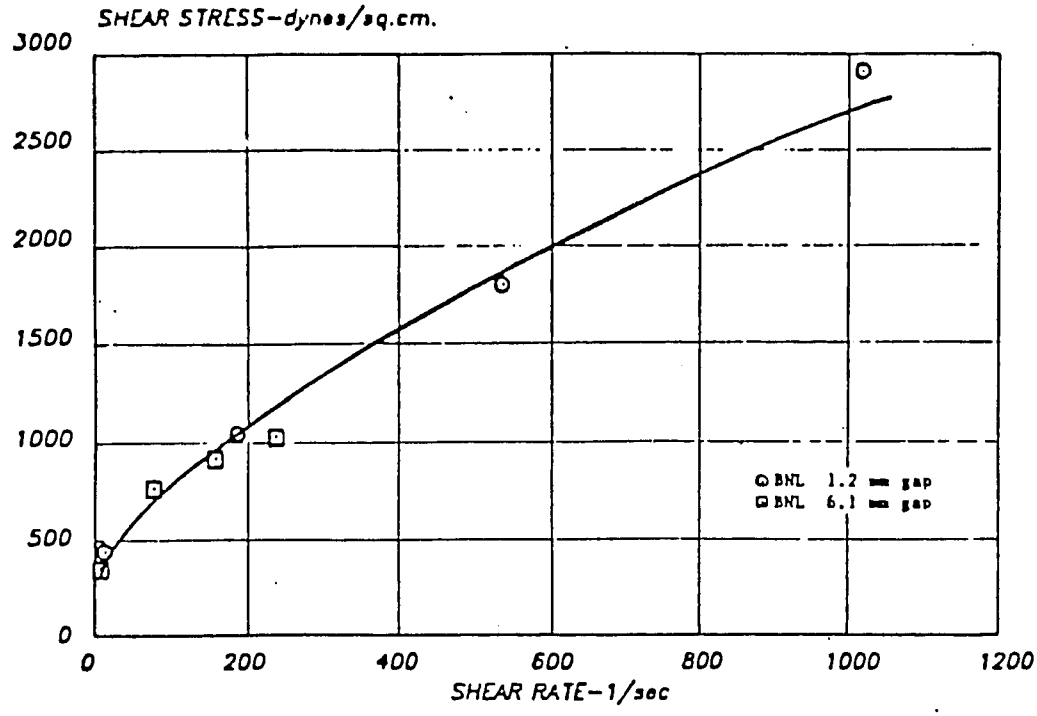

Figure 6. Flow Curve Data Baseline Fuel

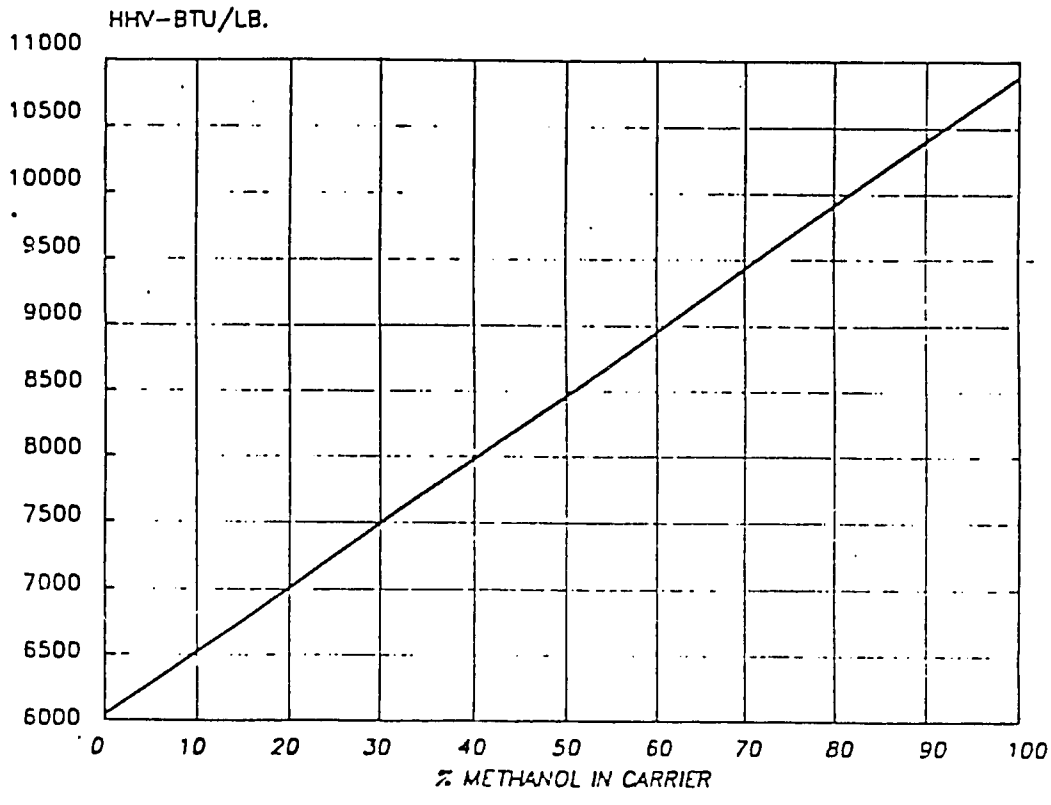

Figure 7. Effect of Methanol Addition on Fuel Higher Heating Value - Semirara Coal Concentration is 50% Carrier Liquid is Water Methanol Mix

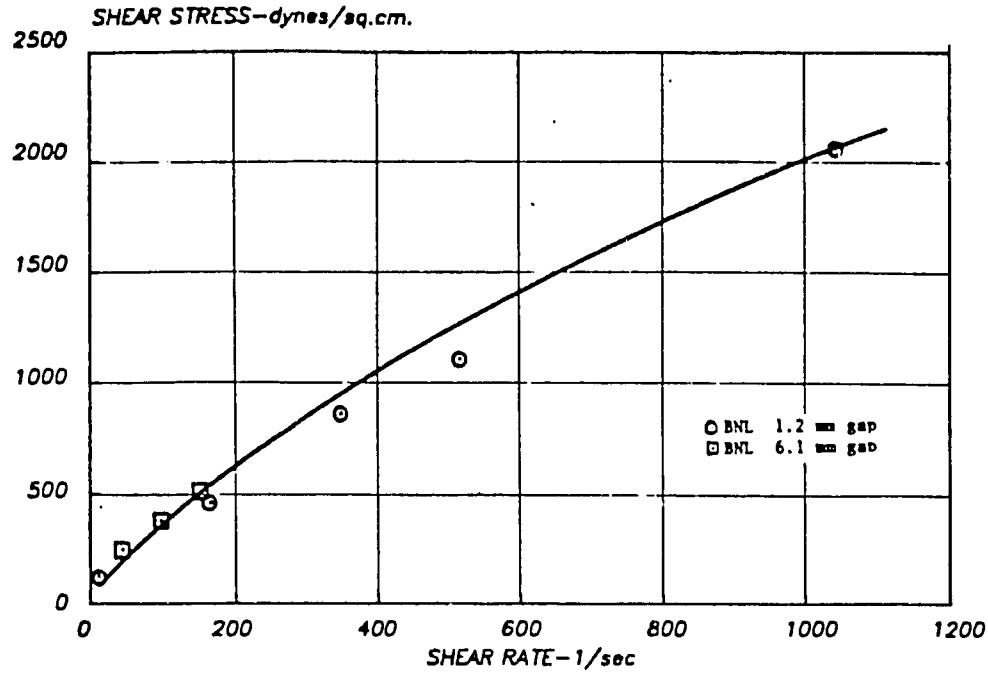

Figure 8. Flow Curve Semirara Fuel Mix Containing 47.5% Coal, 42.5% Methanol, 10% Water

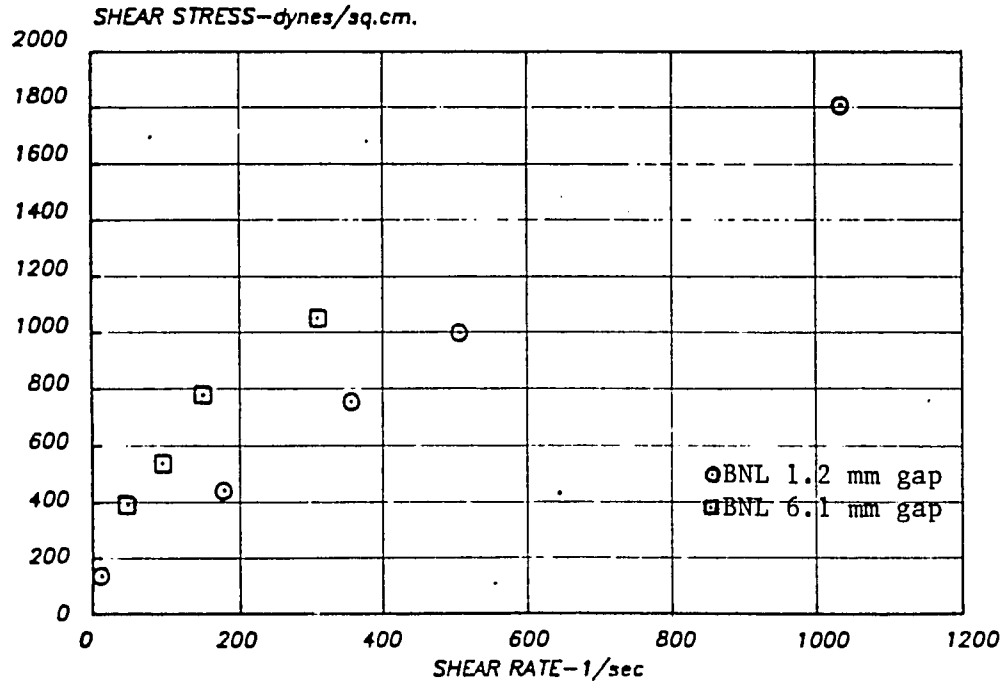

Figure 9. Flow Curve Data - Semirara Fuel Mix Containing 50.3% Coal, 8.2% Methanol, 41.5% Water

cosity of the 47.5% coal-mix at 100 1/sec is 350 cp. Comparison with Figure 3 shows that roughly the same coal loading limits can be expected with methanol as with only coal and water (i.e. from Fig. 3 the viscosity of a 47.5% mix with water only is also about 350cp at 100 1/sec).

5.4 Semirara/Malangas Blends

As discussed in section 3, one of the candidate fuels considered for this project was a blend of up to 25% Malangas and the remainder Semirara coal. Slurries made from this blend benefited significantly from the addition of a surfactant due to the hydrophobic nature of the Malangas coal. The slurries described in this section contained 0.1% (wt.% based on slurry) sodiumlignosulfonate. Tests showed that greater amounts of this dispersant did not significantly improve slurry properties.

Two points on a plot of apparent viscosity (100 1/sec) vs. coal concentration are shown in Figure 10. A comparison of the loading potential for the Semirara/Malangas blend with loading potential for the Semirara only fuel-mix can be made using a reference viscosity of 700 cp. From Fig. 10 a fuel made with a blend of 75% Semirara/25% Malangas (wt. % dry basis) has a viscosity of 700 cp at about 56% coal concentration. From Fig. 3 a fuel made with Semirara alone has this same viscosity at 51% coal, about 5% lower.

5.5 Effects of Coal Cleaning

Table 8 shows basic washability data on batch 1 of the Semirara coal. This separation was done using organic specific gravity media (trade name - Certigrav). The table indicates excellent ash removal and coal recovery at a fairly high specific gravity. Table 9 shows the effect of separation at s.g. 1.5 on ash content and equilibrium moisture as well as the ash composition and fusion temperatures of the floated coal. These results indicate small improvements in ash fusion temperatures. The reduction in equilibrium moisture content, however, suggests significant improvements in slurryability. Figure 11 shows apparent viscosity vs. concentration for the floated product. At a reference viscosity of 700 cp, this curve indicates a 6% increase in coal loading potential, compared to uncleaned Semirara (Fig. 3).

Measurements were also made of the concentration of selected ions in water separated from a Semirara slurry (uncleaned coal). The feed water for this slurry was laboratory distilled water. Results are shown in Table 10. The sodium level translates to over 100% of the sodium in batch 1 of Semirara coal (based on ash ultimate analysis). While >100% of the sodium is not possible, and coal variability is the expected reason, it is clear that a significant fraction of the sodium in Semirara batch 1 is water soluble.

The composition of another sample of Semirara batch 1, after simple water washing, is shown in Table 11. This water wash was performed by preparing a dilute slurry, removing the water using a vacuum filter, and finally flushing the filter cake thoroughly with a water spray.

In an actual washing plant, cleaning would effectively be a combination of specific gravity separation and water washing. To simulate this a sample of the Semirara coal was floated at 1.5 s.g. in the organic medium and then water washed. The measured fusion temperatures of the resulting cleaned coal are listed in Table 12. The ash fusion temperatures, under a reducing atmosphere, were increased almost 200°F as a result of this combined cleaning process.

Table 8

FLOAT AND SINK DATA[a]
SEMIRARA COAL 1/4" X 20 MESH

Specific Gravity	Individual Fraction Wt.%	Individual Fraction Ash %	Cumulative Float Wt.%	Cumulative Float Ash %	Cumulative Sink Wt.%	Cumulative Sink Ash %
Float 1.35	80.0	4.0	80.0	4.0	100.0	11.5
1.35 x 1.48	8.4	16.2	88.4	5.2	20.0	41.1
1.48 x 1.62	3.3	42.7	91.7	6.6	11.6	59.2
Sink 1.62	8.3	65.8	100.0	11.5	8.3	65.8

[a] All ash on a dry basis.

Table 9

SEMIRARA COAL (BATCH 1) FLOATED AT S.G. 1.5
COMPARISON WITH UNCLEANED COAL

	Feed Coal	Floated Coal
Ash Content (% Dry Basis)	11.2	5.2
Equilibrium Moisture (%)	23.0	16.5
Ash Composition		
Silicon Dioxide	48.56%	32.39%
Aluminum Oxide	27.72%	20.84%
Iron Oxide	4.58%	1.53%
Titanium Dioxide	.98%	0.85%
Calcium Oxide	1.79%	6.53%
Magnesium Oxide	3.69%	9.14%
Sodium Oxide	2.15%	0.75%
Potassium Oxide	1.21%	1.90%
Lithium Oxide	155.5 ppm	383.0 ppm
Phosphorus Pentoxide	.24%	0.45%
Sulfates	6.41%	20.56%

Ash Fusion Temperatures (°F)	Feed Coal Reducing	Feed Coal Oxidizing	Floated Coal Reducing	Floated Coal Oxidizing
Initial Deformation	2101	2239	2103	2367
Softening	2118	2332	2196	2492
Hemispherical	2206.	2422	2230	2430
Fluid	2300	2541	2756	2544

Table 10

ION CONCENTRATIONS IN SEMIRARA (BATCH 1)
SLURRY WATER PHASE

Calcium	230	µg/ml
Magnesium	259	µg/ml
Iron	<1	µg/ml
Sodium	3060	µg/ml
Potassium	41	µg/ml

Table 11

RESULTS OF TESTS ON WATER WASHED SEMIRARA COAL (BATCH 1)
COMPARISON WITH UNWASHED COAL

	Feed Coal		Washed Coal	
Ash Content: (Dry Basis)	11.2		12.3	
Ash Composition				
Silicon Dioxide	48.56%		45.61%	
Aluminum Oxide	27.72%		26.17%	
Iron Oxide	4.58%		5.60%	
Titanium Dioxide	.98%		0.93%	
Calcium Oxide	1.79%		4.19%	
Magnesium Oxide	3.69%		4.47%	
Sodium Oxide	2.15%		1.47%	
Potassium Oxide	1.21%		1.56%	
Lithium Oxide	155.5 ppm		178.1 ppm	
Phosphorus Pentoxide	.24%		0.12%	
Sulfates	6.41%		8.99%	
Ash Fusion Temperatures (°F)	Feed Coal Reducing	Feed Coal Oxidizing	Washed Coal Reducing	Washed Coal Oxidizing
Initial Deformation	2101	2239	2272	2355
Softening	2118	2332	2323	2381
Hemispherical	2206	2422	2335	2493
Fluid	2300	2541	2532	2666

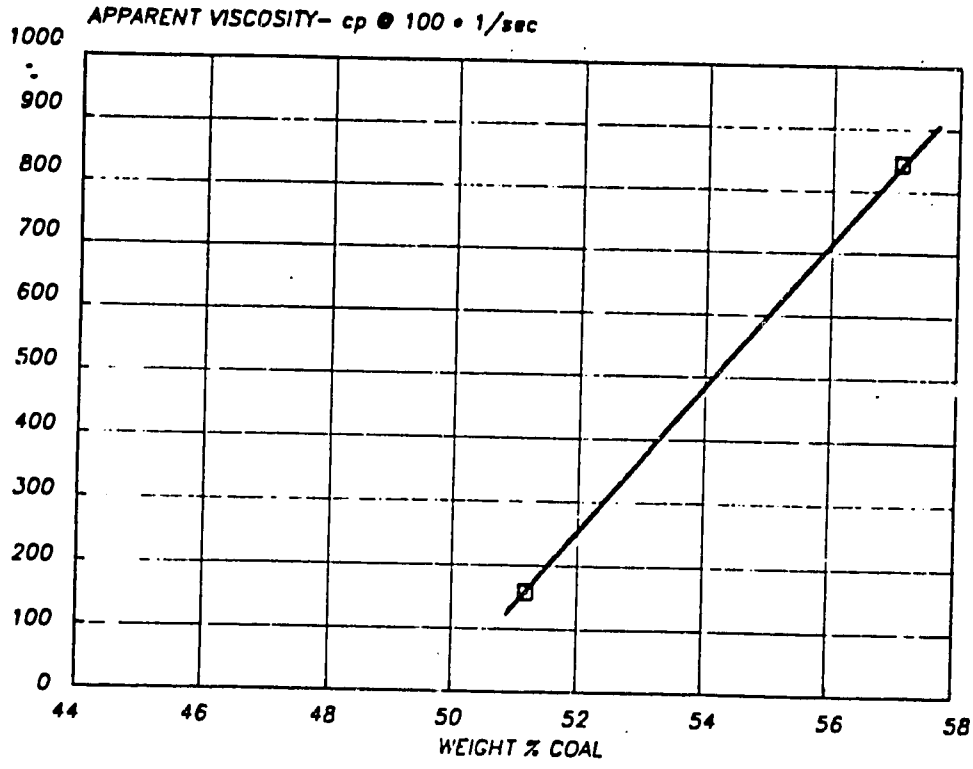

Figure 10. Apparent Viscosity vs. Concentration Blend of 75% Semirara Coal, 25% Malangas Coal

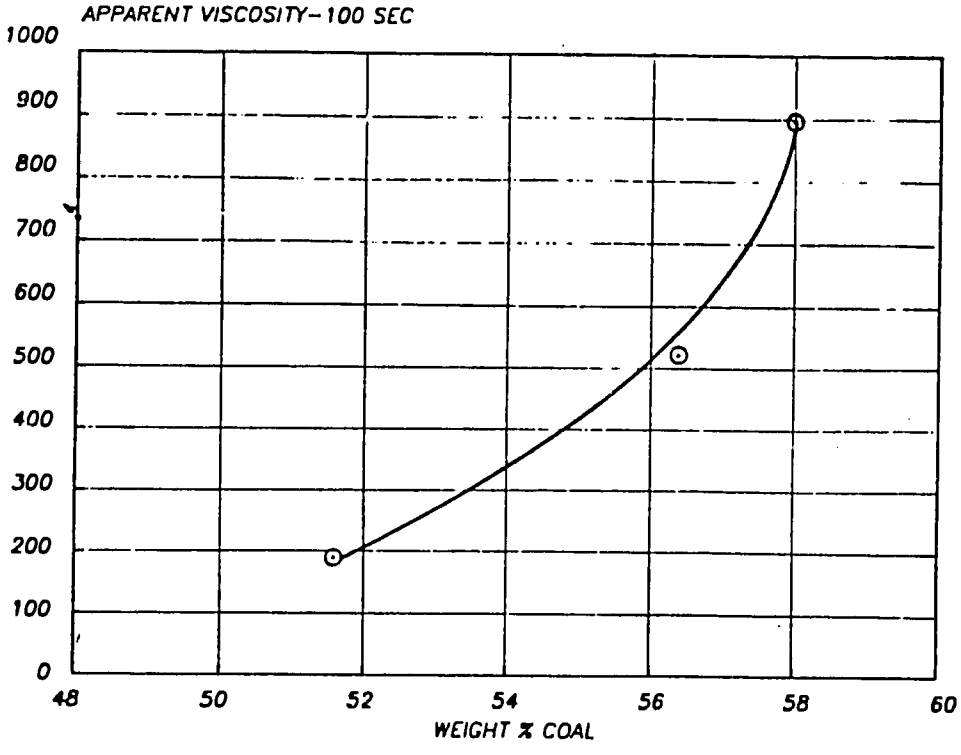

Figure 11. Apparent Viscosity vs. Concentration Semirara Coal (Batch 1) Float 1.55 s.g.

Table 12

ASH FUSION TEMPERATURES
SEMIRARA COAL (BATCH 1) FLOATED AT S.G. 1.48 AND THEN WATER WASHED

Ash Fusion Temperatures (°F)	Reducing	Oxidizing
Initial Deformation	2307	2298
Softening	2361	2350
Hemispherical	2372	2403
Fluid	2412	2485

These sink float tests were also repeated on batch 2 of the Semirara coal. In this case very poor ash rejection at s.g. 1.5 and only limited increase in coal loading potential was observed for the cleaned coal. The poor ash rejection might have been expected based upon the very low ash content of batch 2. This experience highlights the variability in coal samples included in this program.

Studies were also performed on the effects of an acid flush on coal loading potential. This was of interest because acids are known to remove from coal some minerals which hold water and contribute to the equilibrium moisture. The process involved preparation of a slurry in a 1N sulfuric acid solution followed by vacuum filter separation of the coal. This step was followed by a water flush and final air drying. The coal was reslurried and viscosities were measured at various concentrations. An increase in loading potential of about 5% was observed with both batch 1 and batch 2. Figure 12 shows the shear stress/shear rate relationship for a sample prepared from batch 2 of the Semirara coal. The viscosity of this sample at 100 sec^{-1} was 650 cp and the concentration was 55.7% coal.

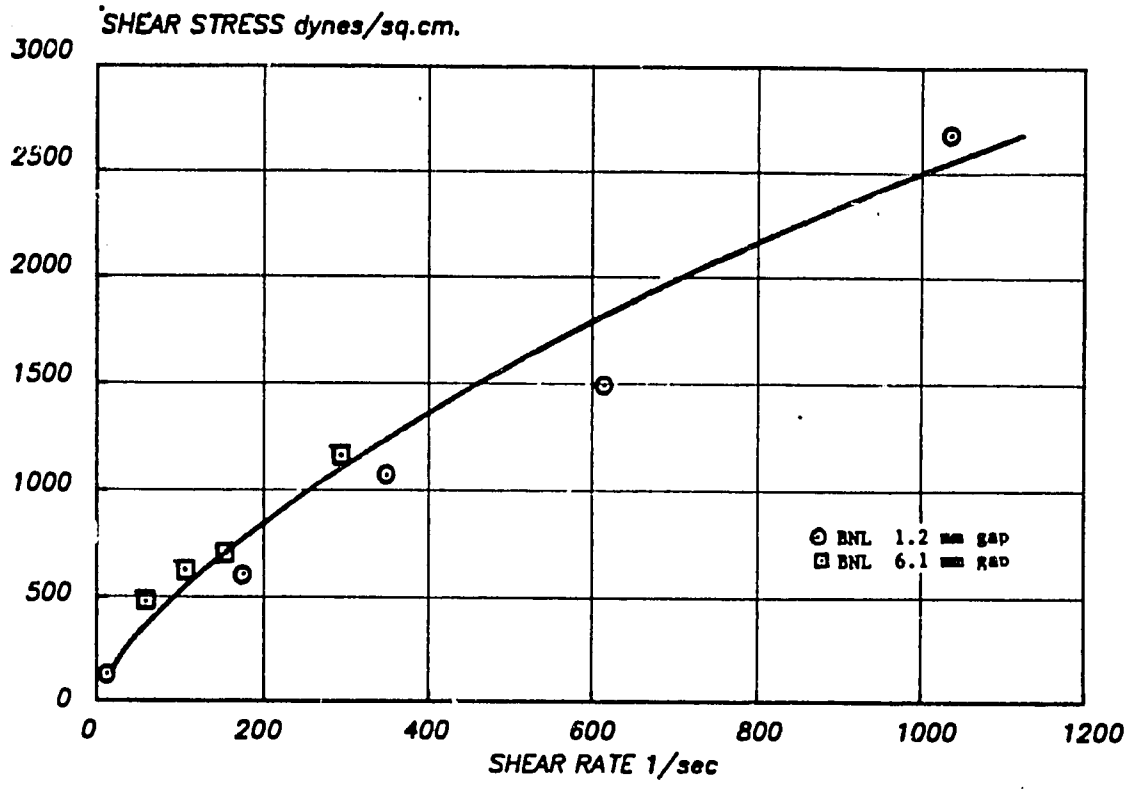

Figure 12. Flow Curve - Semirara (Batch 2) Washed - Sulfuric Acid 1 N 55.7% Coal

Section 6.0

PARTICIPATION OF SLURRY MANUFACTURERS

In the past five years a number of U.S. corporations have developed considerable expertise and capability in the commercial production of coal-water-mix fuels. Recognizing the importance of this capability for implementation of the project under consideration, the participation of these companies was invited. This section discusses the involvement and the important contributions of these groups. Participating companies included:

Atlantic Research Corp.
The Babcock and Wilcox Company
Coaliquid, Inc.
Foster Wheeler Energy Corporation (with Carbogel, Inc.)
Methacoal Corp.
Morrison-Knudsen Company Inc. (with Snamprogetti, Inc. of Italy)
OXCE Fuel Company

6.1 Participation Arrangements

Many of the companies listed above were first involved during a meeting held at BNL in August of 1984. In attendance were representatives of participating organizations from the U.S. and the Philippines. The commercial slurry manufacturers were invited to make brief presentations to the group on their interests and experiences with coal-water fuels. This information was factored into the project planning during that meeting.

Formal participation of these groups in the program was initiated on September 25, 1984 with a meeting in the U.S. AID offices in Washington D.C. In attendance were representatives from AID, BNL, Burns and Roe, Inc., Development Sciences, Inc., all of the above mentioned fuel manufacturers, and two additional manufacturers who later elected not to participate further. A presentation was made to the fuel producers which included a project overview, the results of all BNL tests to date, and a description of the planned BNL tests. Each group was asked to accept a sample of the Semirara coal and prepare a batch of coal-water-mix fuel sufficient to permit testing in the piping loop, atomization facility, and combustion facility at BNL. The original request was for 100 gallons of slurry from each corporation. Owing to the number of companies interested in participation and limited coal supply, this request was later reduced to 50 gallons.

The manufacturers were asked to use their judgement concerning the properties (viscosity, stability, etc.) of a useable slurry. The following specifications were offered to them as a suggestion.

Viscosity:	1500 cp max at 100 sec^{-1}
Size:	70% min - 200 mesh
	99% min - 50 mesh
Stability:	After 24 hours deviation at top and bottom less than 1%

Batch 2 of the Semirara coal, which was intended for this large sample slurry preparation, arrived at BNL from the Philippines on September 27, 1984. The number of companies offering to prepare the 50 gal sample was finally known by mid-October. The first larger sized coal samples were shipped from BNL on October 22, 1984.

During the September 25th meeting the potential use of up to 25% Malangas in the coal blend was discussed with the fuel manufacturers. Subsequently, several of the companies requested, and were sent, small (5 pound) samples of the Malangas coal for evaluation of this option in their laboratories.

6.2 Properties of the Delivered Fuels

Slurry samples, large enough for the planned loop, atomization, and combustion tests were received from the following three companies:

Coaliquid, Inc.
The Babcock and Wilcox Company
Methacoal Corp.

Of the remaining four participating companies, three submitted small samples (one gallon or less) and one was not satisfied with the fuel produced and elected not to submit any sample.

In the remainder of this report, slurry manufacturers names will be replaced by code letters. This is done to inhibit association of specific slurries with manufacturers. In discussions with the fuel companies, one comment on this program was expressed essentially by each of them independently. That is, there was insufficient time and sample quantity to prepare their best fuel formulation.

The basic properties of each of the commercial slurries as well as the baseline slurry (discussed in section 5.2) are listed in Table 13. Coal concentration was measured by simply oven-drying a small sample of the fuel mix. A discrepancy with one manufacturer concerning measurement techniques led to a brief investigation of the effect of oven temperature on measured coal concentration. Results for two slurries are shown in figure 13. A temperature of 50°C was adapted as the standard.

Each of the larger commercial fuel samples (A, C, D Table 13) was received at BNL about 30 days after shipment. Most of this time was spent in trucking company warehouse storage. The as-received concentration profiles in the shipping drums for these three fuels are shown in figures 14, 15 and 16. As these curves indicate, some settling has occured. The high concentration layer on the bottom of slurry A was particularly difficult to resuspend. This occurrence has been termed "hard pack". These concentration profiles point out the need for agitators in storage tanks even if these agitators are only for emergency backup.

A simple evaluation of the stability of samples B, C, and the baseline fuel was obtained in a one-week static settling test. At the end of the test fuel E showed no significant difference between top and bottom concentration, and fuel D had a 1 1/2% concentration difference. The baseline fuel had a 5% concentration difference although it was easily remixed to a uniform state.

Table 13
SUMMARY OF SEMIRARA CWM FUELS

MANUFACTURER	QUANTITY RECEIVED	COAL CONCENTRA-TION	VISCOSITY (CP)	PIPE LOOP	COMBUS-TION
A	25 GAL	50.1	250	X	X
B	PINT	47	500		
C	30 GAL	50.5	1850	X	
C^{1}*	30 GAL	50.5	850		X
D	50 GAL	33	480		X
E	GALLON	50.5	1400		
F	PINT	51	1000		
BASELINE	25 GAL	49.5	875		X

*This is slurry C modified by the manufacturer.

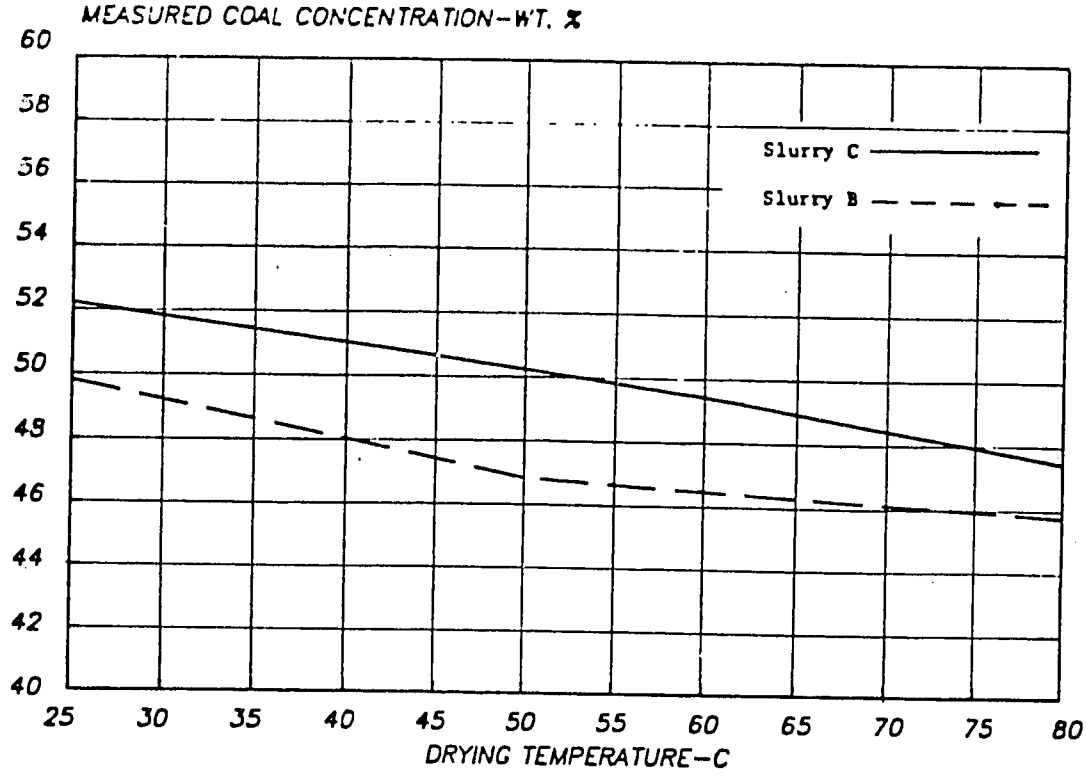

Figure 13. Effect of Oven Temperature on Measured Coal Concentration

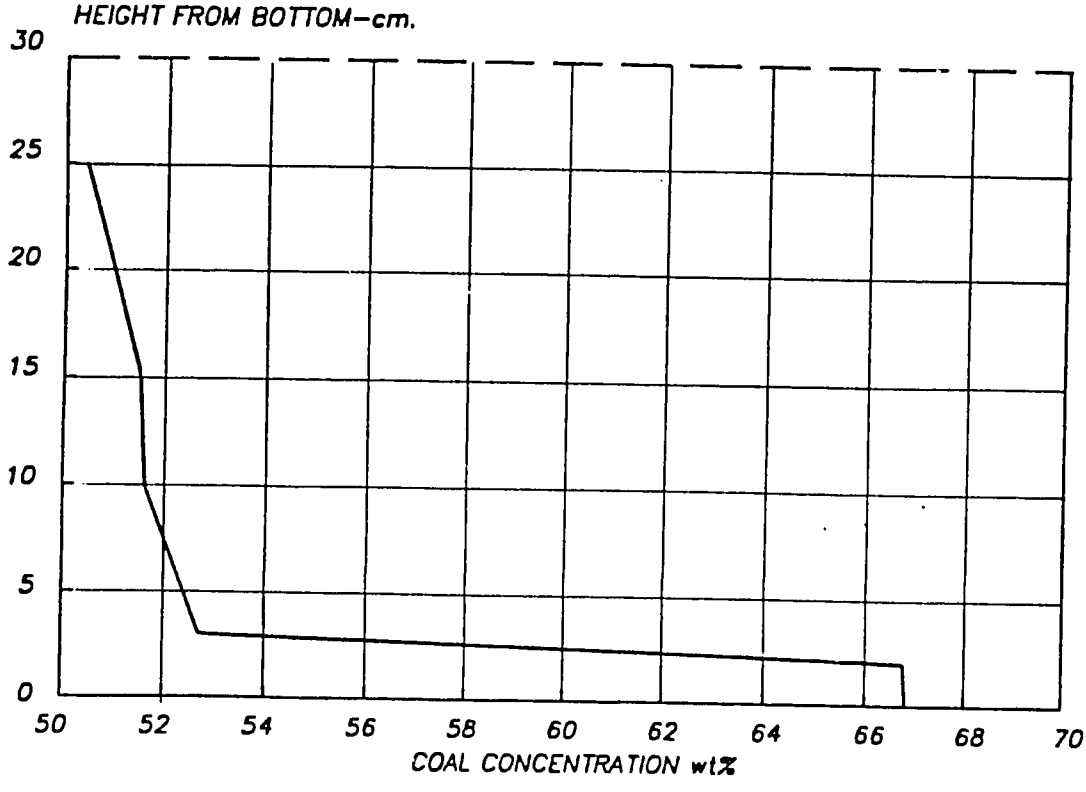

Figure 14. As-received Concentration Profiles - Fuel A

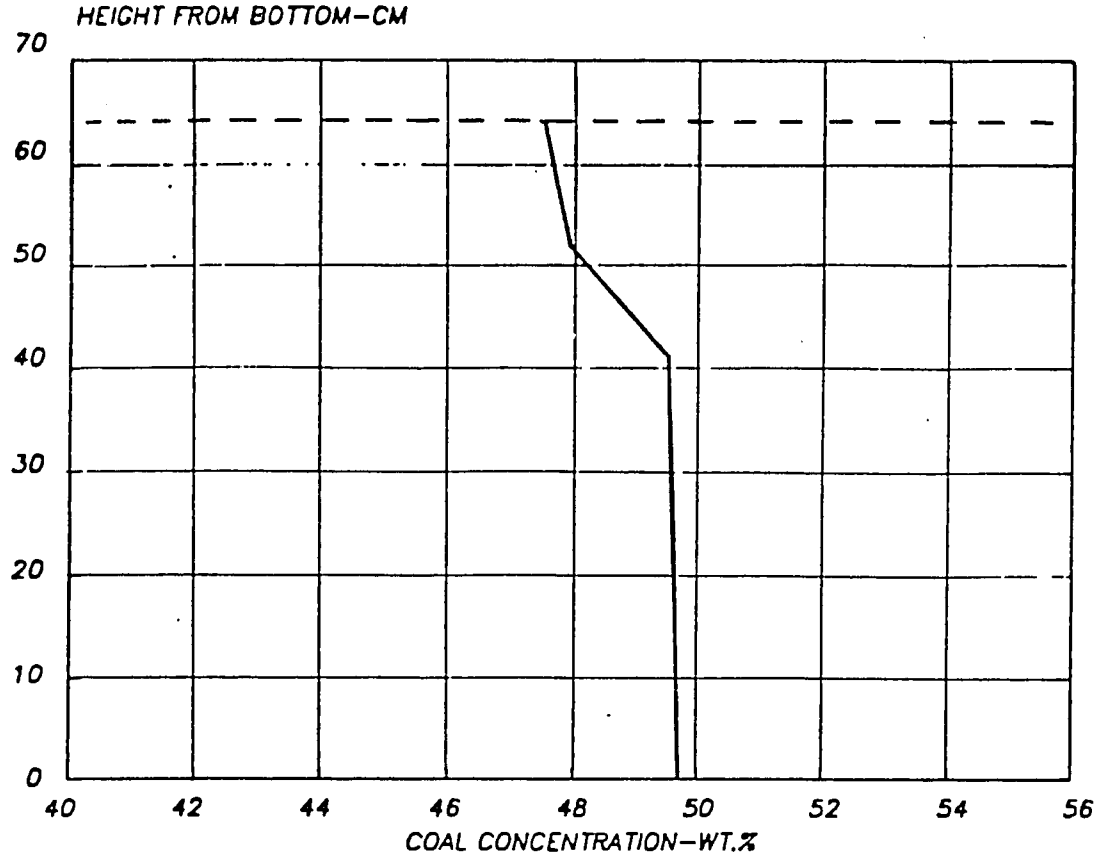

Figure 15. As-received Concentration Profiles - Fuel C

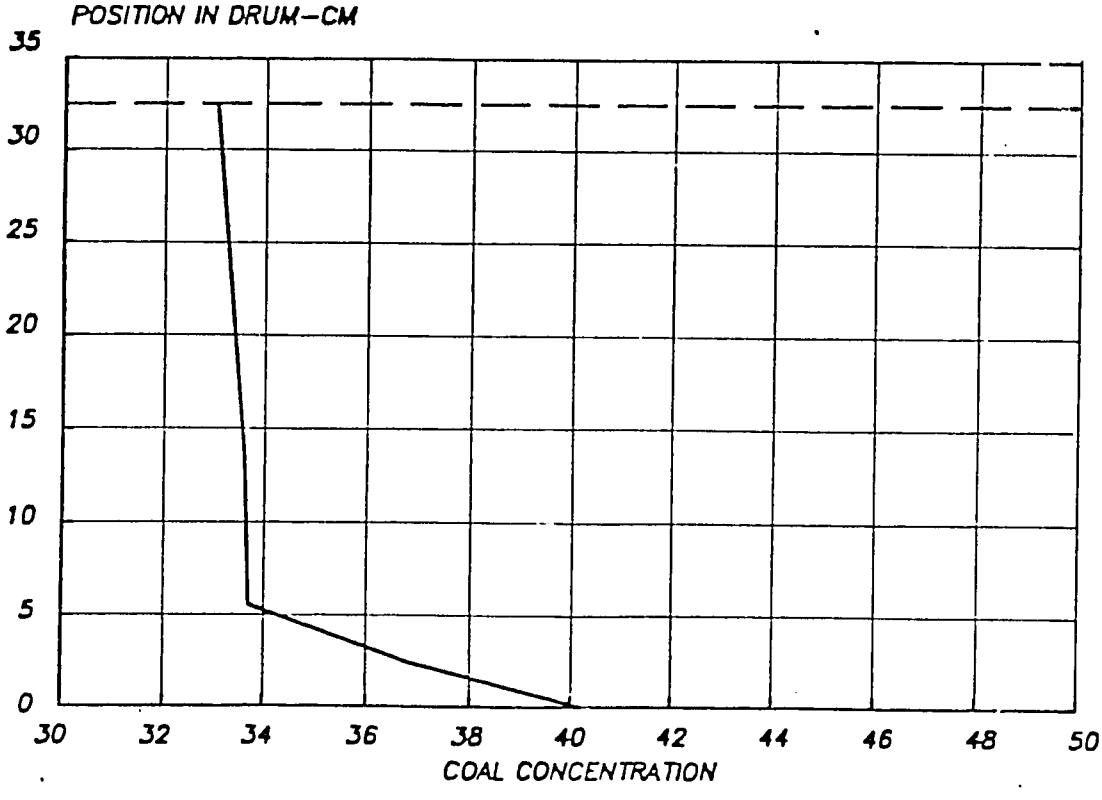

Figure 16. As-received Concentration Profiles - Fuel D

Figure 17 shows a flow curve for slurry A based upon data from both the BNL viscometer and the Stony Brook lab. Agreement can be seen to be good between the different gap sizes. This data for slurry A can be approximately fit with a Hershel-Bulkley model as

$$\tau = 32 + 1.92\,\dot{\gamma}^{.98}.$$

The flow curve for slurry B is shown in figure 18. As with slurry A good agreement between the different viscometer gap sizes can be noted. This data can be approximately fit with a Power Law expression:

$$\tau = 39.87\,\dot{\gamma}^{.596}.$$

Figure 19 shows greater detail for the shear rate range 0-70 sec^{-1} for fuel B (Stony Brook data only). Over this shear rate range the equation

$$\tau = 54.79\,\dot{\gamma}^{.497}$$

was found to best fit the data.

Figure 20 shows the shear stress/shear rate data for slurry C in the higher shear rate range. For this formulation there is a clear discrepancy between different gap sizes, particularly at the highest shear rate. As discussed in section 4.0, this discrepancy illustrates a departure from expected fluid behavior. The following expression fits the data obtained with the Stony Brook viscometer and is plotted on figure 20.

$$\tau = 97.6\,\dot{\gamma}^{.65}$$

Lower shear rate data for slurry fuel C is shown in figure 21, where less discrepancy between measurement systems can be seen. This data has been fit with the expression

$$\tau = 111.0\,\dot{\gamma}^{.61}.$$

During the course of combustion testing (Section 8) plugging problems were encountered with fuel mix C. The fuel manufacturer suggested two options; 1) enlarge the piping or 2) modify the slurry reducing viscosity at the expense of some stability. The second option was selected and a representative from the company came to BNL and added chemicals to the fuel. This modified slurry was significantly less viscous as shown in Table 13 (Fuel C^1). Shear stress vs. shear rate data taken only with the BNL viscometer is shown in figure 22. This data can be best fit by:

$$\tau = 19.2\,\dot{\gamma}^{.79}.$$

As discussed in section 8, combustion tests were successfully completed with this reformulated slurry. The plugging problems were completely eliminated. This modification to fuel C, however, essentially cancelled out all stability.

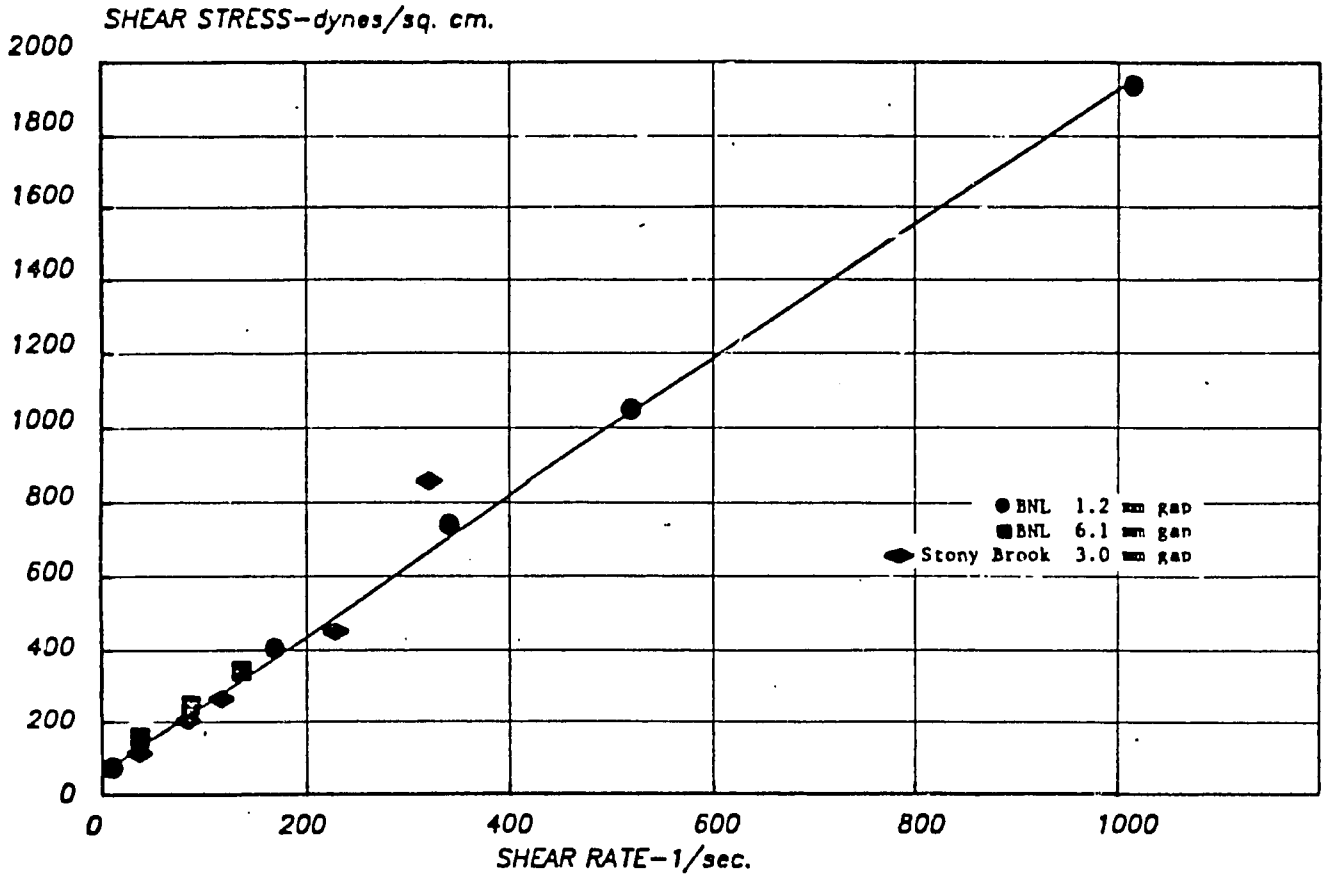

Figure 17. Flow Curve - Slurry A

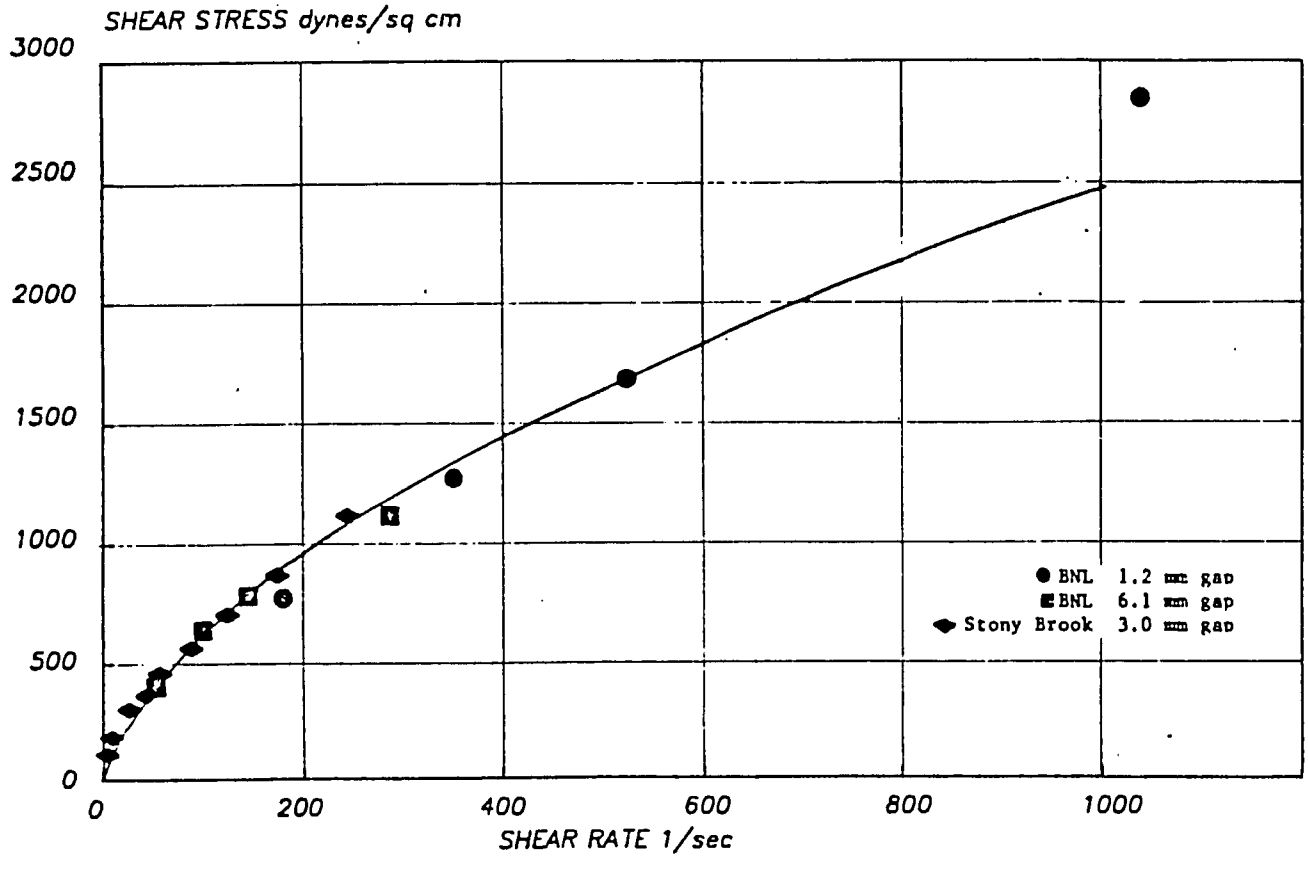

Figure 18. Flow Curve Slurry B

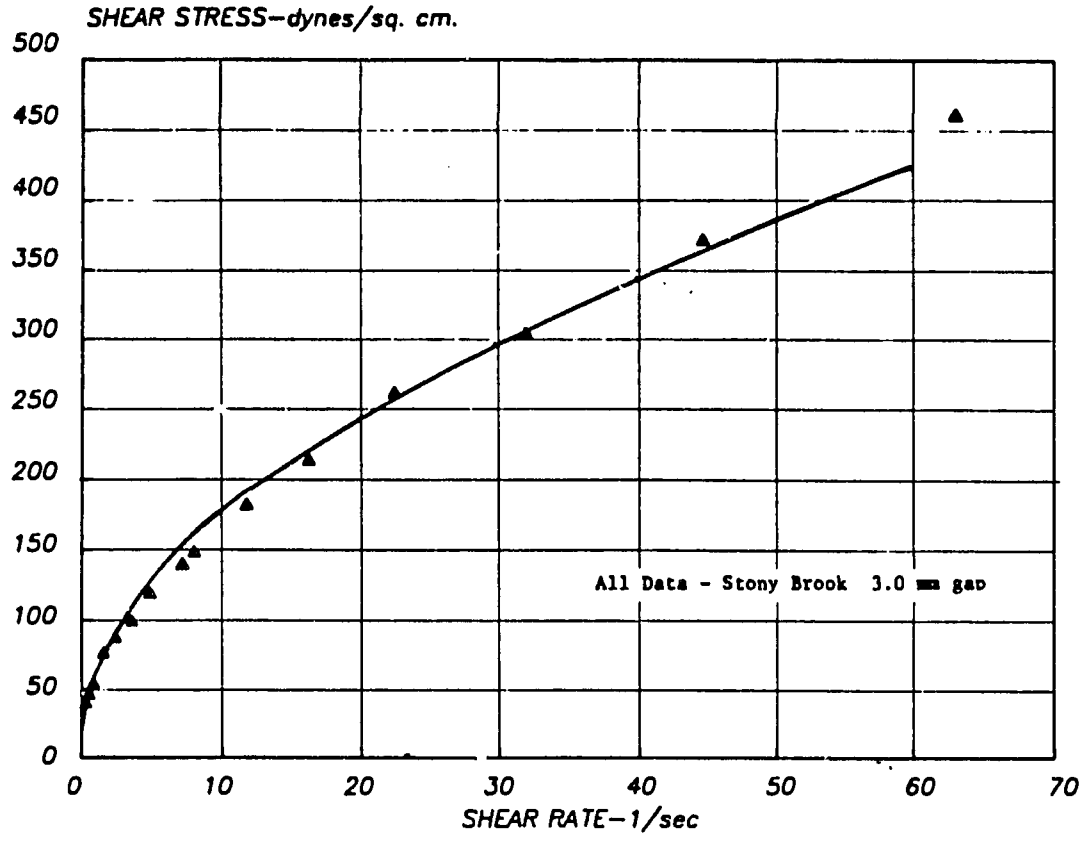

Figure 19. Flow Curve - Lower Shear Rate Range Slurry B

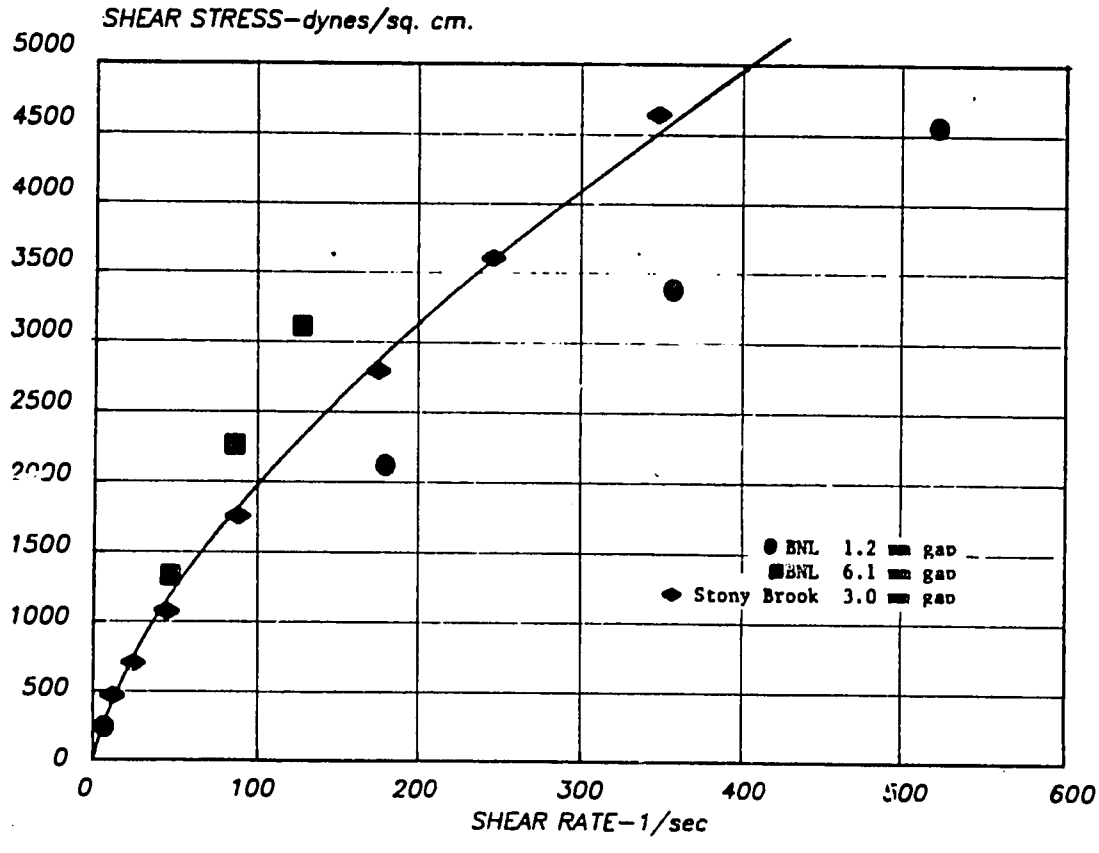

Figure 20. Flow Curve Slurry C

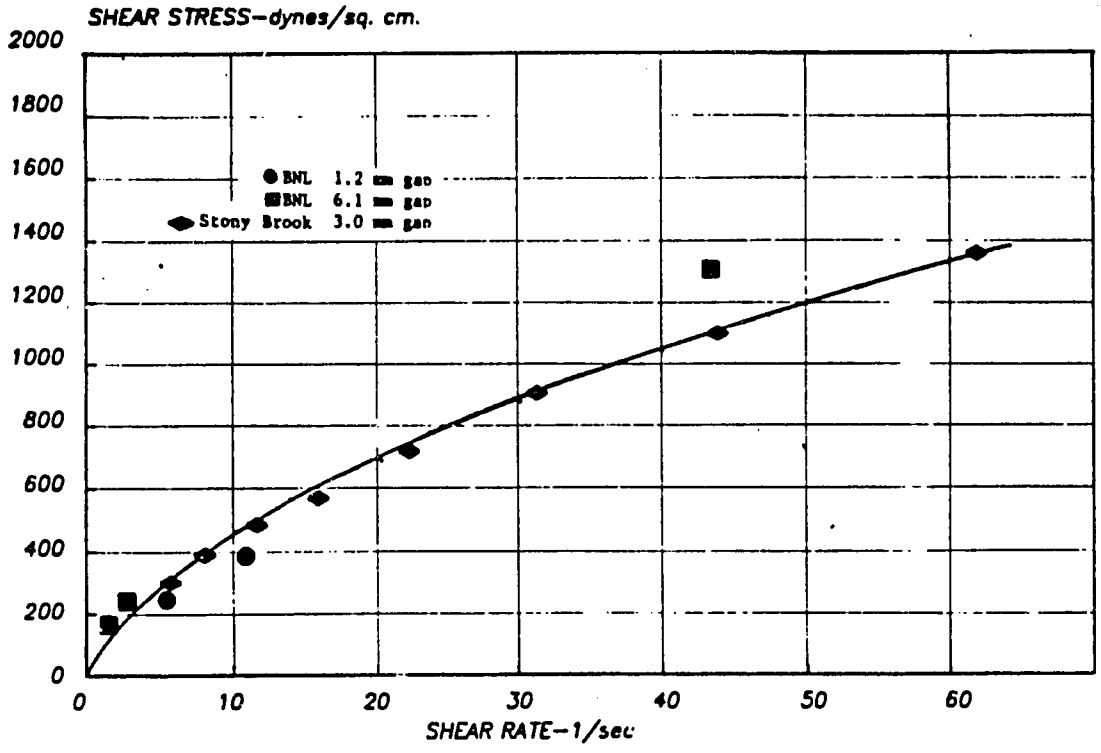

Figure 21. Flow Curve - Lower Shear Rate Range Slurry C

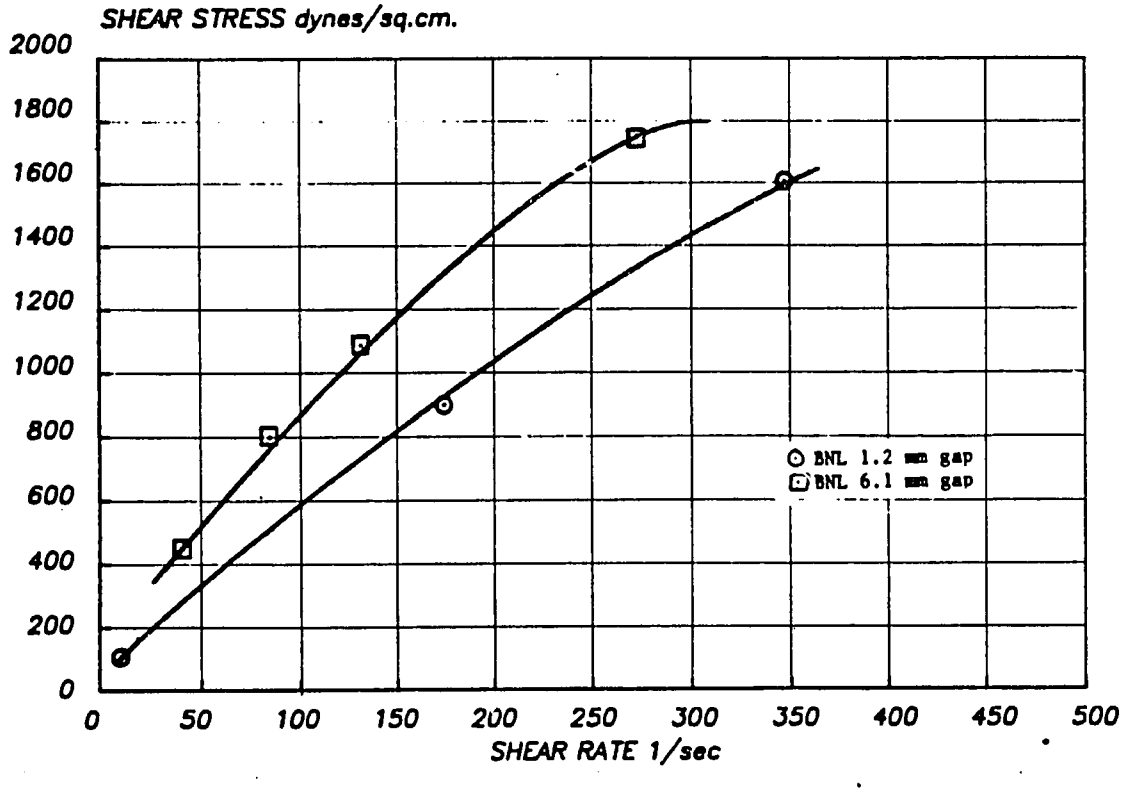

Figure 22. Flow Curve Data Fuel C1

Fuel mix D contained 33% coal, greater than 50% methanol, and the remainder water. This fuel was prepared under contract to the participating company and was not prepared to requested specifications. The tests which were performed at BNL, and reported in section 5.3, indicated that fuel mixtures containing methanol could be loaded to concentrations much higher than the 33% of fuel D. Shear stress vs. shear rate for fuel D is shown in figure 23.

This data can be fit with the expression:

$$\tau = 300 + 6.41\dot{\gamma}^{.711}$$

While this expression matches the data over the shear rate shown in figure 23, caution should be used in concluding that the fluid has a true yield stress of 300 dynes/cm^2. Limited lower shear rate data (not shown) indicates that the yield stress is certainly somewhat below 100 dynes/cm^2 and possibly much lower. This diminishing of the yield stress upon examination at very low shear rate is typical of many plastic fluids.

Figure 24 shows the flow curve for slurry E (BNL viscometer only). This data is represented by the equation

$$\tau = 25.4\dot{\gamma}^{.87}.$$

The flow curve for fuel F is shown in figure 25 (BNL viscometer only). A discrepancy between the two gap sizes used is obvious for this fuel. The data can be very roughly fit with a Bingham Plastic model as

$$\tau = 180 + 6\dot{\gamma} \quad .$$

A second sample was submitted by Manufacturer F towards the end of the program. This sample contained 51.7% coal and had a viscosity of approximately 800 cp. Only the first sample has been used for analysis in this study.

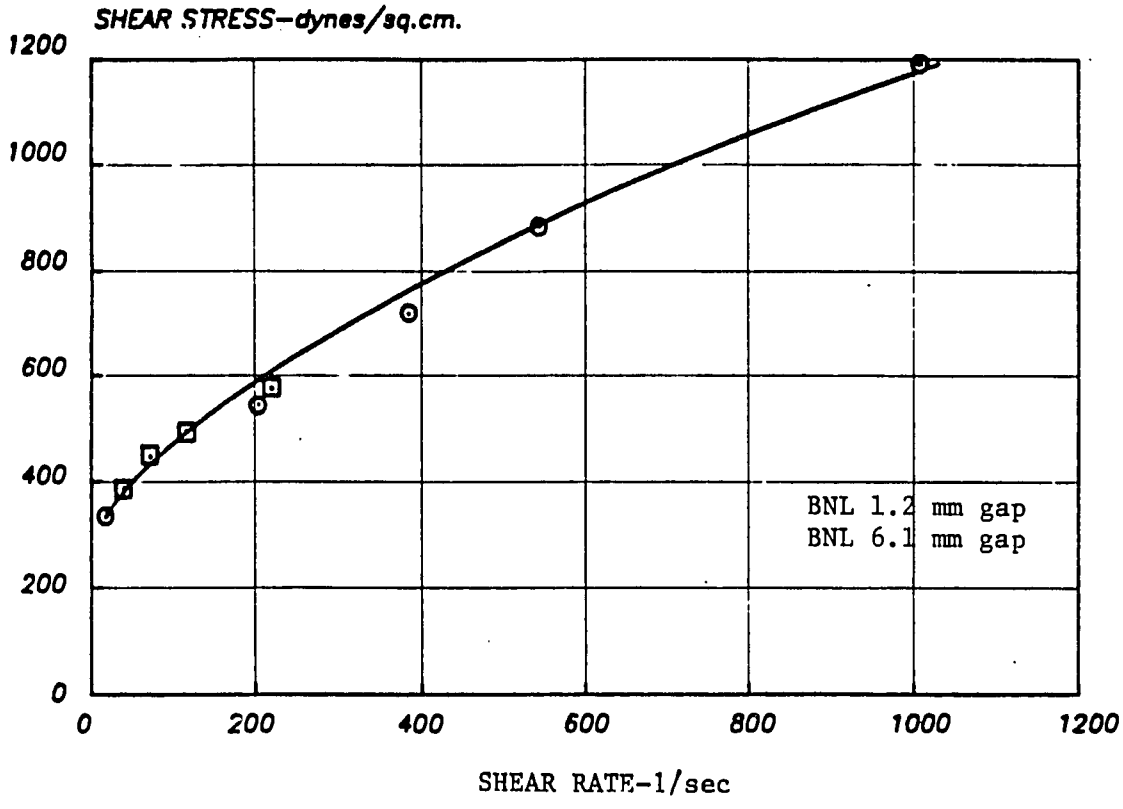

Figure 23. Flow Curve Data Fuel D

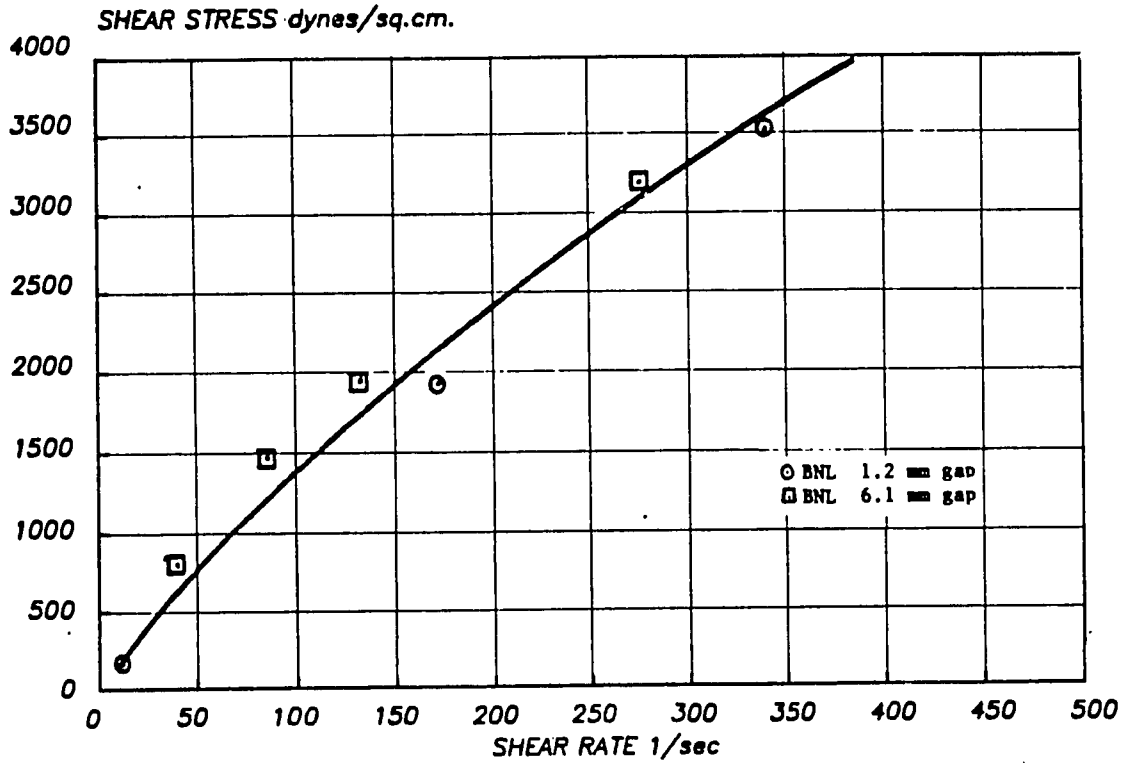

Figure 24. Flow Curve Data Fuel E

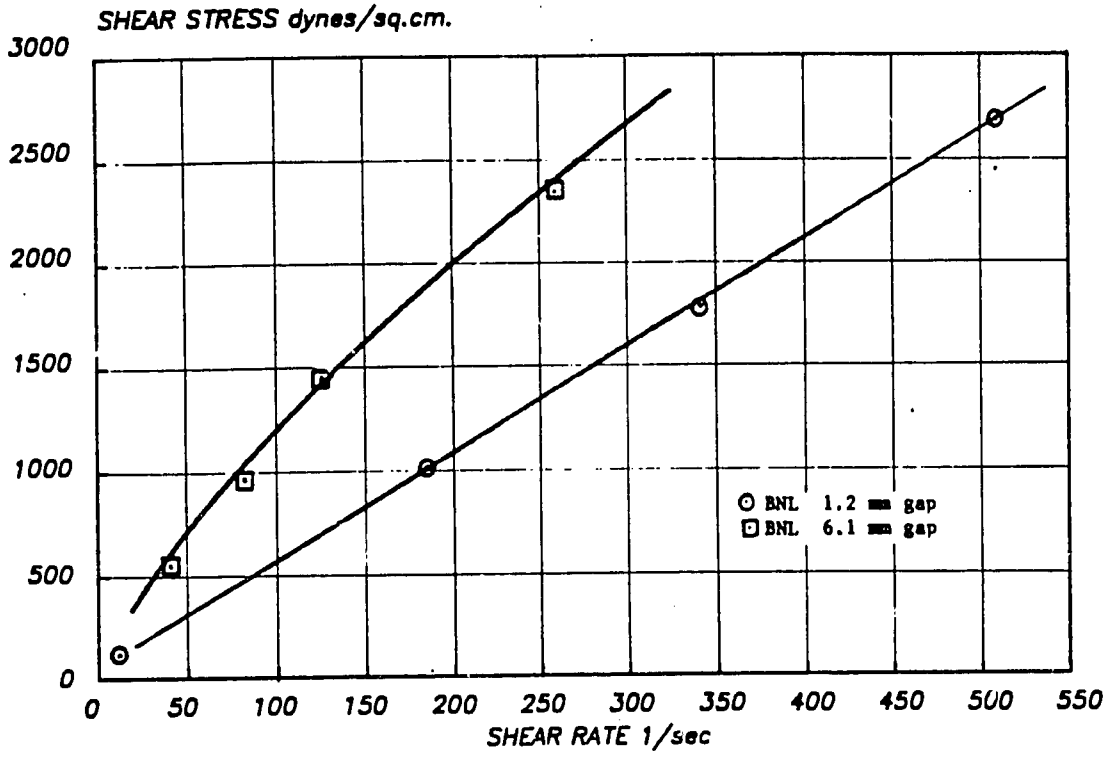

Figure 25. Flow Curve Data Fuel F

Section 7.0

PIPE LOOP TESTS AND PRESSURE DROP PREDICTION

Loop tests on the larger fuel samples were performed for two reasons: 1) to identify any handling problems which could affect implementation of a conversion project and 2) to determine the accuracy with which pressure drops can be predicted with viscometer data.

7.1 Experimental Details

The piping system used is illustrated in figure 26. The system consists of two loops sharing a common feed tank and flowmeter. The "small" loop has a 3 gallon/minute variable speed progressive cavity pump. The "big" loop is fed with a fixed speed 30 gallon/minute progressive cavity pump with flow control provided by recirculation to the 55 gallon storage tank (not shown fig. 26). The storage tank is jacketed and cooling is provided by a circulating water/glycol mix which rejects heat via a water to air heat pump unit. The loop is fitted with provisions for both air blowdown and water flushing.

The small loop has a 1" and a 1/2" nominal schedule 40 steel pipe, horizontal test section. The large loop has a 1" steel pipe and a 2" pvc pipe (id=1.905 inches) horizontal test section. Pressure drops across the 1" section of the small loop and the 2" section of the big loop are measured with a differential pressure transmitter using diaphram isolators. The pressure drop in the 1/2" section of the small loop and the 1" section of the big loop are measured with direct indicating strain guage pressure transducers mounted approximately 2" above the test point without diaphram isolators. These two sections also have midpoint pressure transducers (not shown in fig. 26). These midpoint taps were installed essentially to determine if the flow was fully developed. In general, it was found that the pressure drop across the first half of these sections was the same as the second half and total pressure drop was used in data reduction.

Table 14 lists the nominal shear rate ranges and average velocity ranges obtained in each test section during a typical run. Flow was strictly in the laminar regime for the fuels tested with Reynold's numbers ranging from 1 to 750.

Table 14

Loop Sections - Nominal Shear Rate and Velocity Ranges

	Shear Rate Range - sec^{-1}	Velocity Range ft/sec
Small (3 GPM) Loop		
1/2" section	190-415	1.2-2.7
1" section	20-85	.40-.95
Big (30 GPM) Loop		
1" section	200-1100	2.2-12.0
2" section	35-185	.7-3.6

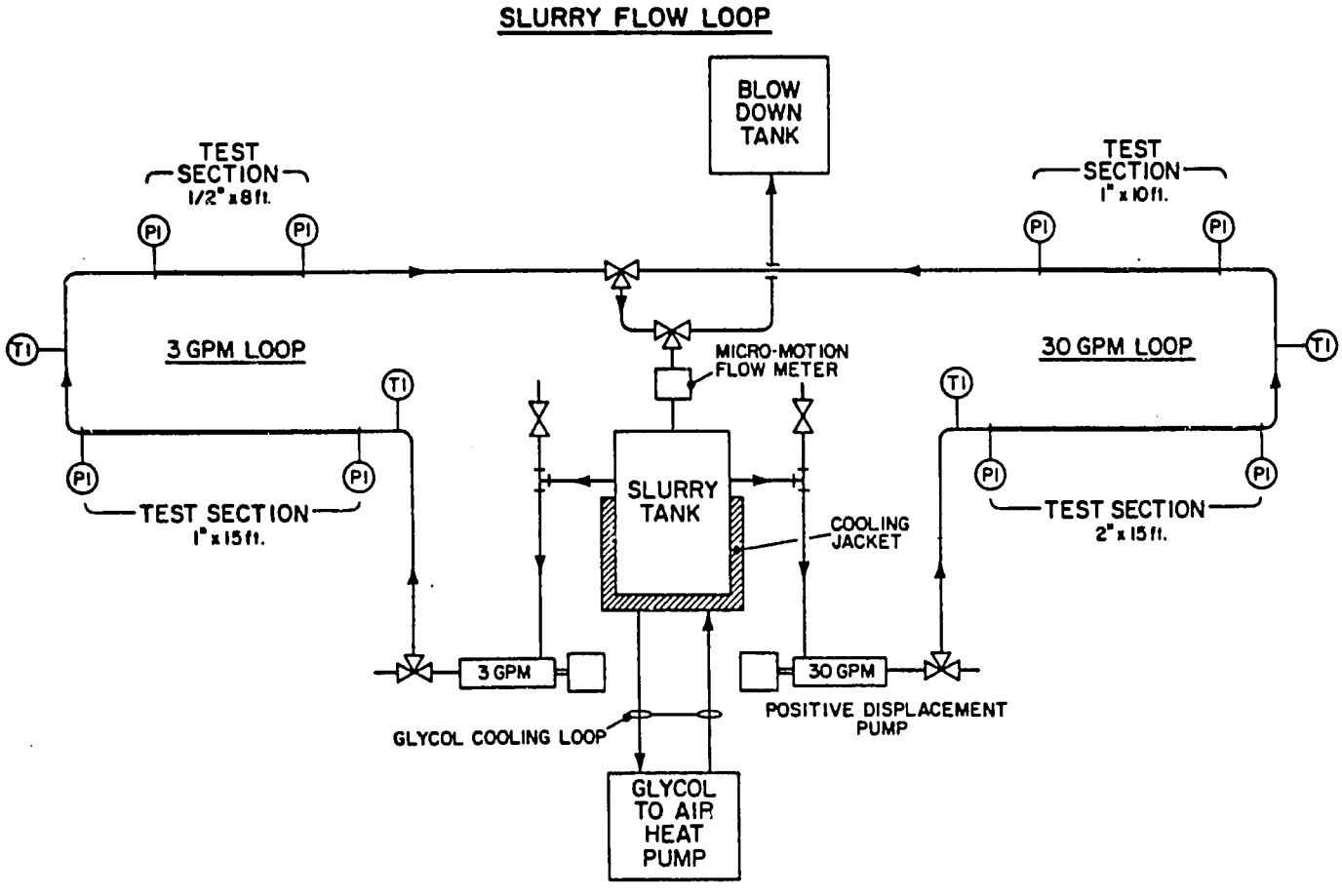
SLURRY FLOW LOOP
BLOW DOWN TANK
TEST SECTION
1/2" x 8 ft.
TEST SECTION
1" x 10 ft.
PI
TI
3 GPM LOOP
30 GPM LOOP
MICRO-MOTION FLOW METER
SLURRY TANK
COOLING JACKET
TEST SECTION
1" x 15 ft.
TEST SECTION
2" x 15 ft.
3 GPM
30 GPM
POSITIVE DISPLACEMENT PUMP
GLYCOL COOLING LOOP
GLYCOL TO AIR HEAT PUMP

Figure 26.

To put the data from the loop on an equivalent basis with the viscometer, it is necessary to determine wall shear stress and wall shear rate. The wall shear stress is simply related to pressure drop by

$$\tau_w = \frac{D \Delta P}{4L}$$

where τ_w = wall shear stress
ΔP = test section pressure drop
D = test section diameter
L = test section length

Note that ΔP and τ_w will have the same units.

Wall shear rate is calculated from a more complex expression, which has as its only assumption no slip at the pipe wall.

$$\dot{\gamma}_\omega = \frac{8v}{D}\left[\frac{3}{4} + \frac{1}{4N}\right] \quad \text{where } N = \frac{d\,\ell n\,(D\Delta P/4L)}{d\,\ell n\,(8v/D)}$$

v = velocity (ft/sec)

7.2 Loop Test Results

To ensure confidence with the data generated from the coal-water-mix fuels to be tested, "calibration" runs were performed with both a glycerine water mixture (93% glycerine) and a Semirara slurry prepared at BNL (46% coal). In both cases excellent agreement with viscometer data was obtained.

Figure 27 shows the results of the loop tests for fuel A along with viscometer data (BNL only) in a shear stress vs. shear rate format. As this figure shows, there is reasonably good agreement among measurements indicating that the flow properties of this fuel are "predictable".

The results in the same format for fuel C are shown in figure 28. This figure shows considerable discrepancy between viscometer and loop data at shear rates greater than 200 1/sec. Considering the discrepancy between different viscometer gap sizes, which was noted for this slurry (figure 20) this result might have been expected.

7.3 Suggested Methods for Prediction of Pressure Drops in Pipe Flows

The data for fuel A was shown in section 6.2 to be well fit with a Hershel-Bulkley model as

$$\tau = 32 + 1.92\ \dot{\gamma}^{.98}.$$

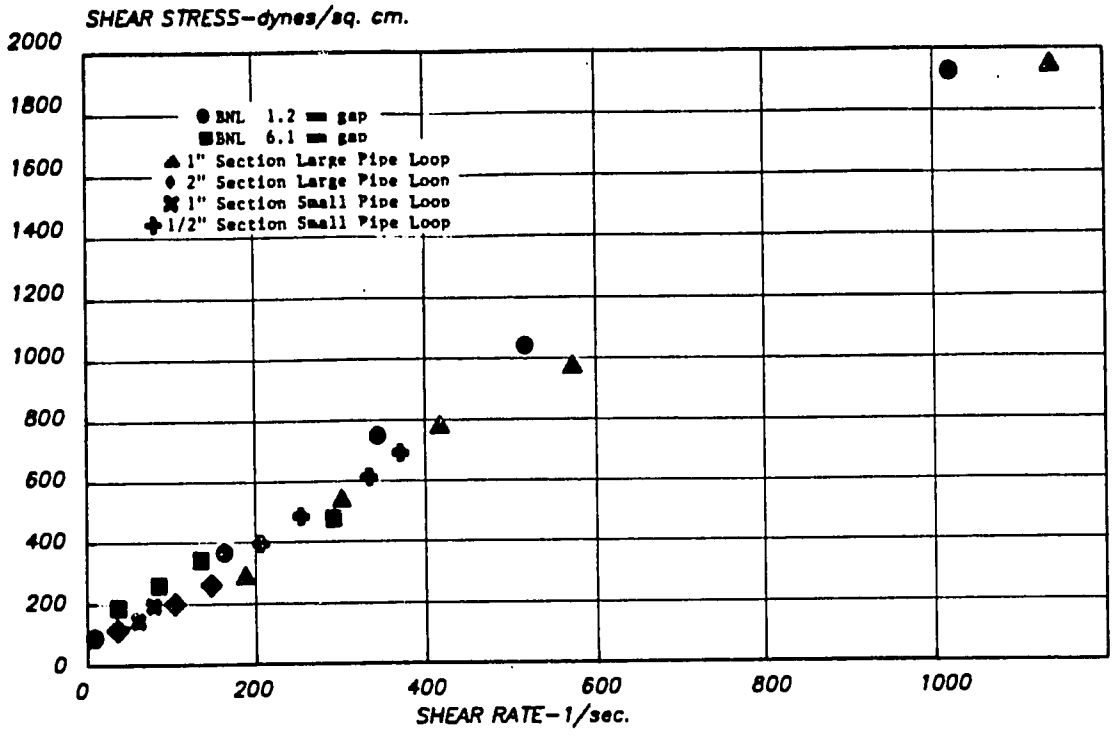

Figure 27. Comparison of Viscometer and Loop Flow Curves Fuel A

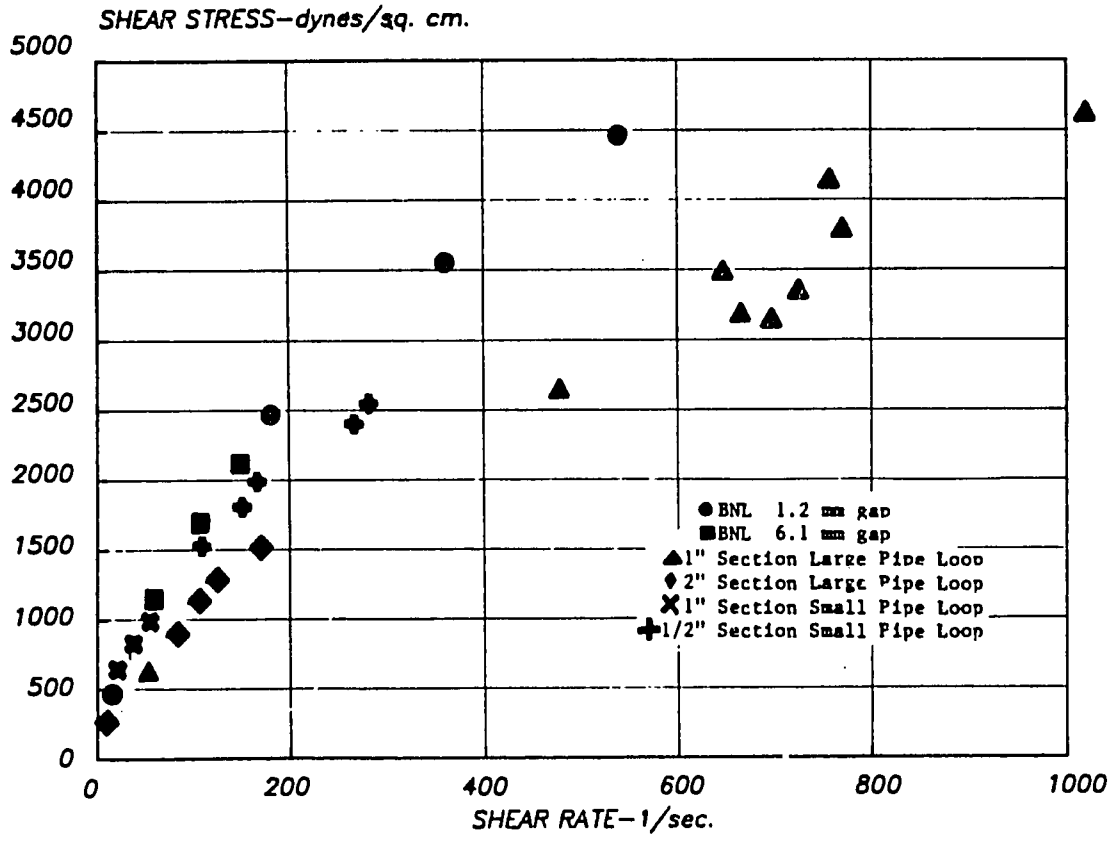

Figure 28. Comparison of Viscometer and Loop Flow Curves Fuel C

The pipe loop data confirms that this model will express the flow properties of fuel A. Utilization of this model in routine design, however, is not very convenient even if the flow index of .98 is approximated as 1.0 (Bingham Plastic Model). A convenient approach can be obtained, however, by using a generalized correlation of the pipe flow data (the results equivalent to an approximate Bingham Plastic approach). This is shown in figure 29. The data in this figure can be fit by the expression

$$\frac{D \Delta P}{4L} \times 1000 = .607 + .025 \left[\frac{8V}{D} \right]$$

D = Pipe diameter (ft)
L = Section length (ft)
V = Velocity - (ft/sec)
ΔP = Pressure Drop - (lb/in^2)

This expression is recommended for use at values of $\frac{8V}{D}$ greater than 20.

The data for fuels B, C, C[1] and E, as well as the baseline fuel, has been fit with a power law expression (commercial fuels section 6.2, baseline section 5.2). In this case there is a relatively simple expression for pressure drop[3]:

$$\frac{D \Delta P}{4L} = (1.45 \times 10^{-5}) \left[\left(\frac{3n + 1}{n} \right)^n K \left(\frac{2V}{D} \right)^n \right]$$

where: K - consistency (dynes sec^n/cm^2)
n = flow index (dimensionless)

These are defined by the general powerlaw expression

$$\tau = K \dot{\gamma}^n$$

The flow curve for fuel D in section 6.2 was shown to be fit by the expression:

$$\tau = 300 + 6.41 \dot{\gamma}^{.711}$$

The pressure drop calculation for this fuel is not as simple as for the other fuels. The process involves iterative solutions of the flow equation. Once this has been done for a specific fluid of this type, the results can be expressed in a single form, as follows.

$$\frac{D \Delta P}{4L} \times 1000 = 4.35 + (.292) \left[\frac{8V}{D} \right]^{.556}$$

approximation accurate to 3% for $17 < \frac{8V}{D} < 500$

For fuel F the following approximate expression can be used:

$$\frac{D \Delta P}{4L} = (1.45 \times 10^{-5}) \left[240 + 6 \left(\frac{8V}{D} \right) \right] \text{ for } \frac{8V}{D} > 30$$

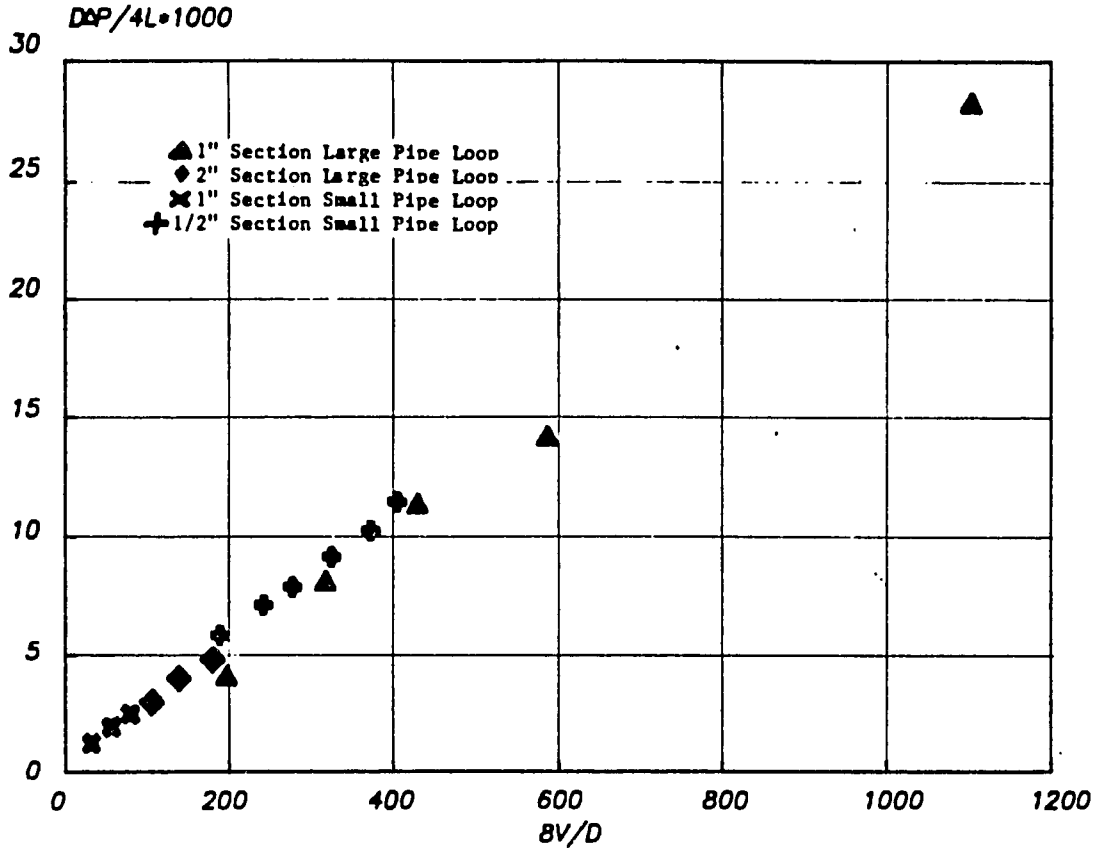

Figure 29. Fuel A Piping Loop Pressure Drop Relations

This approximation will provide accuracy better than 6% (excluding errors in the model equation) for wall shear stress greater than 360 dynes/cm^2. This corresponds approximately to values of 8V/D greater than 30. Considering the difference between the flow curves for the two gap sizes as shown in figure 25, a greater degree of accuracy at a cost of greater complexity would not seem justified.

These expressions apply only in laminar flow and to use them the generalized Reynold's number must be under 2100. This can be calculated as:

$$Re = .1077 \frac{\rho V^2}{\frac{D \Delta P}{4L}}$$

ρ = density - gms/cm^3

To illustrate, Table 15 shows pressure drops and generalized Reynolds number for a typical in plant pipe flow of 1 ft/sec in a 1" pipe. Nominal shear rate (8V/D) for this case is 460 sec^{-1}.

Tables 16 and 17 show the pressure drops and Reynold's numbers which could be expected in the two pipelines under consideration in this project (see Volume IV for further detail). For fuel D the approximate expression presented in this section was not used for these calculations but instead the exact expression was solved iteratively. In the 16 inch pipeline the Reynold's number for fuel A indicates turbulent flow and as a result the expression presented in this section should not be used. For this case a much more involved calculation procedure was followed which is not presented here.[4] One common approach used for Bingham Plastic fluids out of the laminar regime is to use the viscosity at very high shear rate (for fuel A 192 cp) and assume Newtonian flow. If this is done for the case of the 16" pipeline, a pressure drop of 31 psi/mile is calculated. Comparison with Table 17, (25 psi/mile for fuel A) shows that this approximation will give good results as a first approximation with fuel A. For all calculations shown in Tables 16 and 17 the error associated with the approximations to the models is under 2%.

Table 15

EXAMPLE FLOW CALCULATION 1" PIPE, 5 FT/SEC, NOMINAL SHEAR RATE 460 SEC^{-1}

Fuel	Pressure Drop psi/100 ft	Generalized Reynold's Number
A	55	270
B	110	120
C	380	40
C^1	170	80
D	62	224
E	360	40
F	209	70
Baseline	123	110

Table 16

EXAMPLE FLOW CALCULATIONS 10" PIPE, 5 FT/SEC, NOMINAL SHEAR RATE 48 SEC^{-1}

Fuel	Pressure Drop psi/Mile	Generalized Reynold's Number
A	46	1790
B	154	495
C	473	160
C^1	158	480
D	172	440
E	280	270
F	195	390
Baseline	269	280

Table 17

EXAMPLE FLOW CALCULATIONS 16" PIPE, 5 FT/SEC, NOMINAL SHEAR RATE 30 SEC^{-1}

Fuel	Pressure Drop psi/Mile	Generalized Reynold's Number
A	25	3600
B	76	630
C	222	220
C^1	68	700
D	99	480
E	116	410
F	97	500
Baseline	139	345

The expressions for fuels A and F will involve some error at lower values of 8V/D and for this reason lower shear rate limits have been recommended. For flows below these limits more complex design procedures should be used[3]. Caution should be used, however, in assuming that the shear-stress/shear-rate relationship derived from higher shear rate data is valid at very low shear rates.

Section 8.0

ATOMIZATION AND COMBUSTION STUDIES

Development work on coal-water-mix fuel in the U.S. and other countries has basically involved slurries with 65% or more coal. At the beginning of this program there was essentially no boiler combustion experience with CWM fuels with as low a coal content and heating value as the Semirara based blends discussed in previous sections. For this reason, an emphasis in this program was placed on determining the conditions under which this fuel could be made to burn. Detailed parametric combustion trials were, in general, impeded by the very limited amount of fuel available at this stage in the program.

8.1 Atomization Studies

Several fuel nozzle types were evaluated for use in the combustion trials. The primary issue influencing nozzle selection was plugging blockages in the fuel flow line. These are caused either by single oversized particles or, more often, by coal particle agglomerates. The nozzle used for most of the tests very simply consisted of a central fuel stream whch was atomized at the nozzle exit by a high pressure, swirling air blast. The advantage of this type of nozzle is an absence of sharp turns and small clearances in the fuel flow passages both of which tend to promote plugging. The expected disadvantage is a solid cone spray pattern with a narrow spray angle which could lead to a long flame.

Combustion tests were also performed using a commercial nozzle (Swirl Air Model - Delevan, Inc.) with a significantly wider spray angle. This nozzle has an impact plate at the front tip which is hit by the internally mixed fuel and air, forming a hollow cone spray angle. Combustion tests with this nozzle showed only small differences in flame pattern relative to the "straight" nozzle described above. This nozzle had a greater tendency to plug as a result of smaller clearances (2.6/32" minimum clearance as compared to 5/32" with straight nozzle) and for this reason the straight nozzle was used for most tests. With this nozzle the system was nearly free from plugs. Occasional plugs did occur when the fuel was too concentrated or when agglomerates were present in the fuel. These agglomerates can form from coal-water fuel which has dried on the supply container wall above the free surface. The dried coal agglomerates can be knocked off of the wall into the fuel leading to the difficulties.

These nozzles are not truly representative of nozzles expected to be used in a large boiler. Nozzles which are representative, however, are difficult to scale down to the size needed for these combustion tests. As system size shrinks and flow passages get smaller, the potential for plug formation necessitates different approaches.

Atomization tests were performed using the straight nozzle described above to provide an estimate of the drop sizes experienced in the combustion tests and to provide some comparison between fuel formulations.

A schematic of the BNL atomization test stand is shown in figure 30. Slurry is fed to the nozzle with a variable speed progressive cavity pump and through a Micro Motion flowmeter. An exhaust system pulls air from the room and through the tank opening to prevent a back flow of fine drops from biasing the drop size measurement. Drop size distribution measurements are made using a Malvern Laser Diffraction system.

Atomization tests were performed on two fuels, commercial mixes A and C^1 (section 6). Tests were not done with fuel D because of safety concerns associated with its high methanol content.

Figure 31 shows results of the atomization tests in the form of spray mean diameter plotted against atomizer fuel/air ratio. The curve in this figure represents water alone. Most of the combustion tests described in the following sections were performed at a fuel/air ratio of 4. Figure 31 shows that fuel-mix A atomizes much like water while mix C^1 produces significantly larger drops. Drop size is, in general, smaller than would be expected in a larger boiler.

8.2 Combustor System Description

Combustion tests were performed in a laboratory cast iron sectional boiler at a nominal firing rate of 1 million Btu/hr. A sketch of the boiler is shown in figure 32. This figure shows refractory liner sections as well as a refractory quarl which was installed for improved flame stability. The hot face of the refractory was measured during combustion tests to be approximately 2000°F which is significantly lower than the adiabatic flame temperature. Over the course of many combustion trials a significant ash deposit accumulated in the rear of the furnace. No slag was found on any surfaces during this program.

The burner used for these tests is illustrated in figure 33. It has an integral, direct contact, kerosene fired air heater which can produce air temperatures to 530°F. This is done at the expense of some of the oxygen in the combustion air. At 500°F, a typical operating temperature, oxygen content has been reduced from 21.0 to 19.2%.

The burner has a light oil nozzle adjacent to the slurry nozzle. The oil is pressure atomized and has a maximum heat input rate of 280,000 Btu/hr. Oil is used for warm-up, light off of the slurry fuel, and cofiring for flame stability when required.

The head of the burner has fixed axial flow swirl vanes to enhance recirculation and flame stability. The swirl is achieved by taking a relatively high pressure drop across the burner head (10-12 in. H_2O). Combustion air is supplied from a high pressure, high capacity air compressor. While certainly not practical in anything but a laboratory boiler, the compressor supply affords great flexibility in the design of burner head pressure drop.

Coal-water-fuel mix is supplied to burners by a variable speed progressive cavity pump. Fuel flow is monitored with a Micro Motion flowmeter. The combustion lab is equipped for measurement of gaseous and particulate emissions.

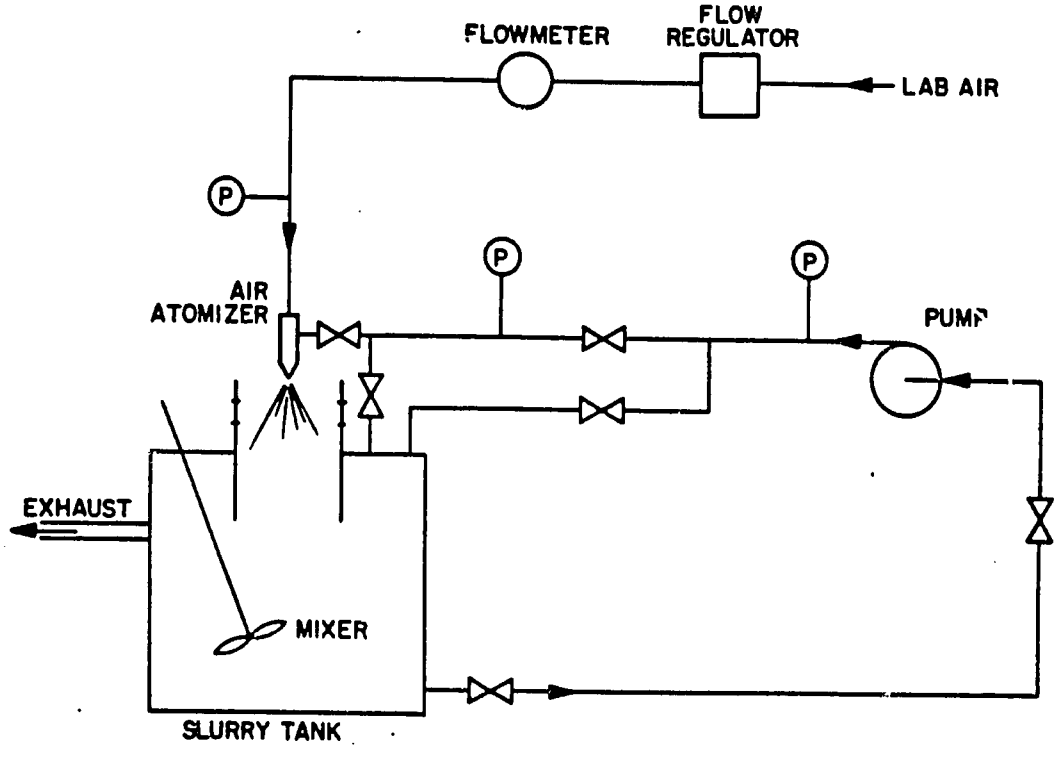

Figure 30. Atomization Test Stand

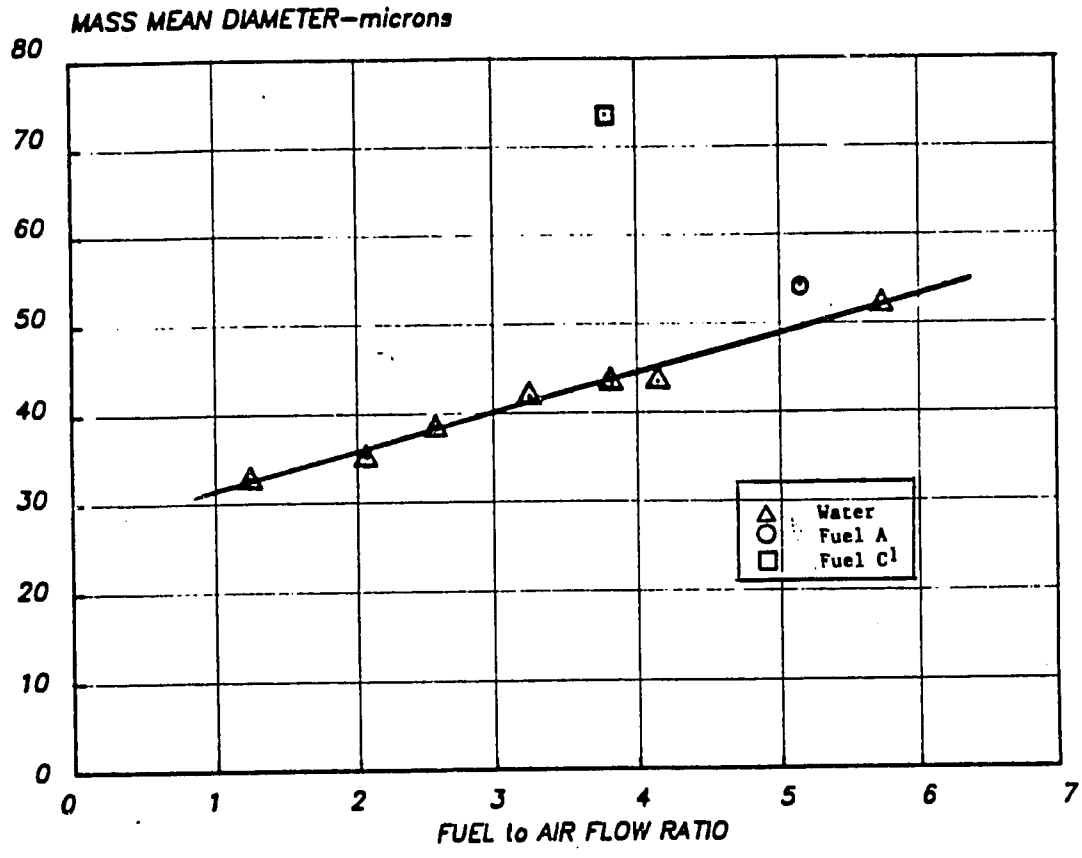

Figure 31. Atomization Test Results

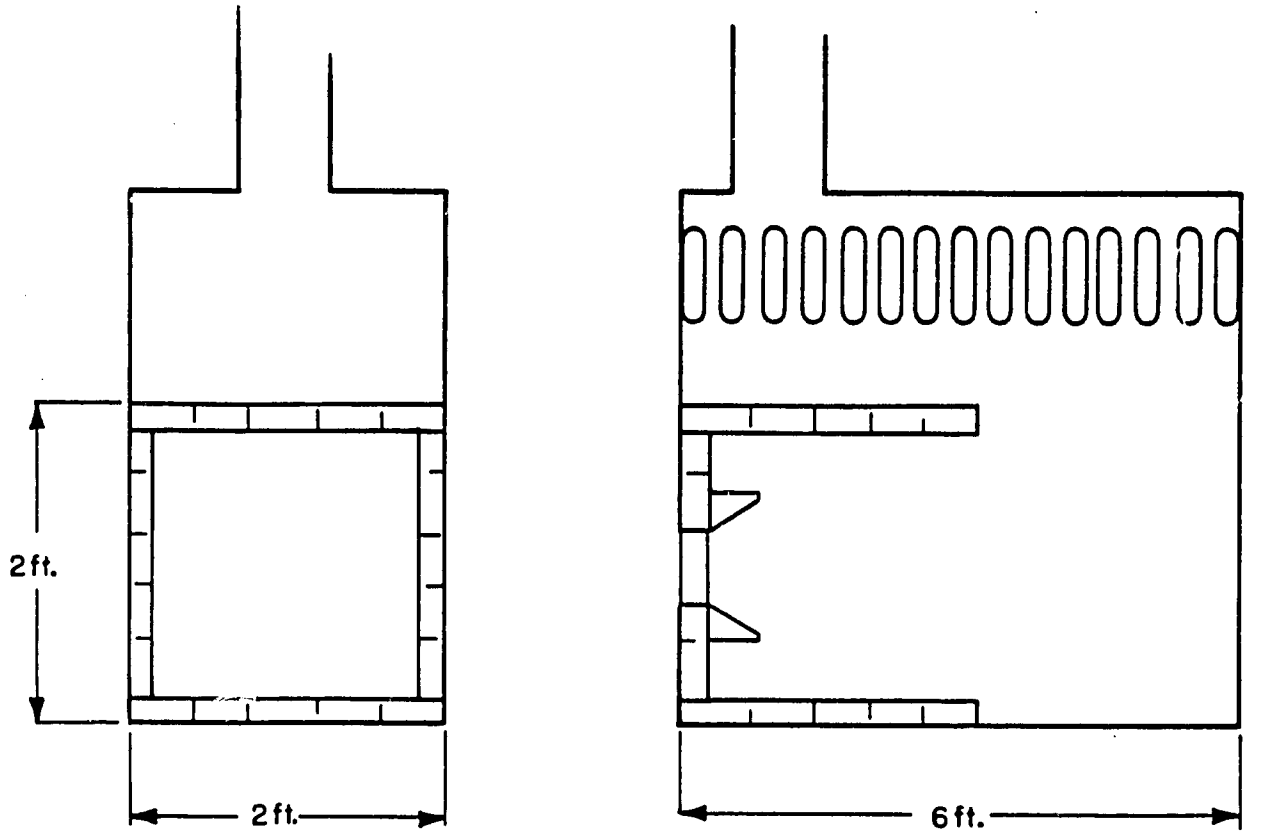

Figure 32. Combustion Chamber Arrangement

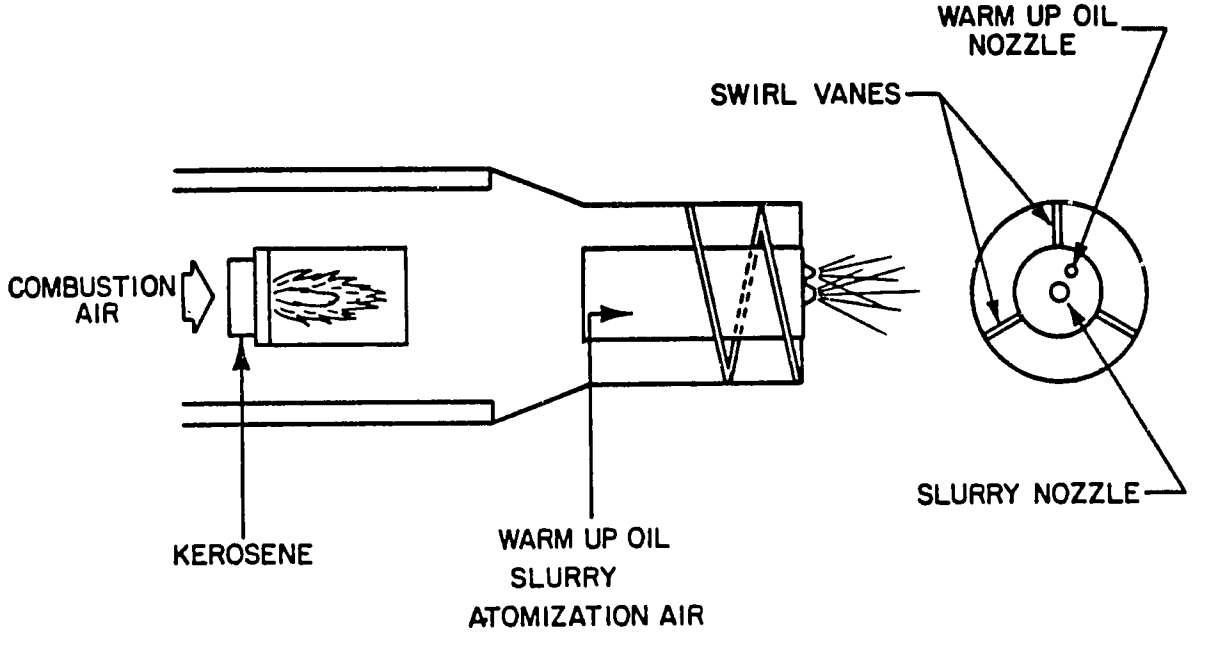

Figure 33. Coal-Water-Mix Fuels Burner

8.3 Combustion Test Results

As a routine during the combustion tests, water was passed through the slurry nozzle prior to introduction of the coal-water-mix. This was done to cool the slurry nozzle when operating with oil and/or air preheat and to prevent plug formation during slurry light off. Overheating of the oil nozzle was also of some concern particularly when the oil was off. During these times the oil line was continuously purged with compressed air.

As discussed in section 6, combustion tests with fuel C could not be performed due to atomizer and line plugging. The manufacturer modified the slurry (fuel C^1) reducing viscosity significantly. With this modification combustion trials were performed without difficulty.

The operating procedure for fuels A, C^1, and the baseline fuel was similar. Firing light oil without preheated combustion air, the boiler was warmed up for 30 min. The air preheater was then turned on and combustion air was warmed to 400°F. A warm up slurry was then injected beginning at a low flow rate with the oil on. This warm up slurry was made from an eastern U.S. bituminous coal (14,500 Btu/lb dry, 4% ash, 35% volatiles) and contained 46% coal on a dry basis. This fuel was used instead of the Semirara CWM simply to conserve the available quantity of the fuel samples. Cofiring the oil with the warm up fuel, the system was further warmed for 30 min. at a total heat input of approximately 1.1 million Btu/hr. During this time combustion air preheat temperature was increased to 500°F. Finally, pump suction was switched to the Semirara fuel sample to be tested and the oil was turned off. At this point the test began. Typical operating data is listed in Table 18. Each of the fuels A, C^1, and baseline behaved similarly in terms of flame appearance. In general, the flame was stable, although less well defined than an oil flame.

Table 18

TYPICAL OPERATING DATA
COMBUSTION TRIALS WITH FUELS A, C^1, AND BASELINE

Fuel Flow lbs/min	2.7
Atomizing Air Flow lbs/min	.53
Atomizing Air Pressure psi	45
ΔP Across Burner Head (inches water)	12
Flue Gas:	
O_2 (% dry)	2-3
CO (ppm)	1000
SO_2 (ppm)	300
Combustion Air Temp. °F	500°F
Boiler Stack Temp. °F	600°F

As discussed in section 6, fuel D contained a significant amount of alcohol. The combustion trials performed with this fuel were done without air preheat. After the 30 min. warm up on No. 2 oil, Fuel D was injected and lit directly. After a few minutes of cofiring, the No. 2 oil was turned off. Compared to the fuels containing only coal and water the flame pattern was much tighter and better defined. With the exception of combustion air temperature, operating data was the same as listed in Table 18.

Section 9.0

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2. Wasp, E. J., Aude, T. C., and Raymer, F. B., Terminal Facilities for Western Coal Slurry Pipelines, presented at the 1976 AIME Annual Meeting, Las Vegas, Nevada (Feb. 22-26, 1976).

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APPENDIX I

DESCRIPTION OF SAMPLING PROCEDURES AT THE COAL MINE

Economic Development Foundation

June 7, 1984

Mr. Tom Butcher
Brookhaven National Laboratory
UPTOWN, New York 11973
U. S. A.

Subject: Coding Instructions for Philippine Coal Samples

Dear Mr. Butcher:

The samples you received for the RP-CWM Project are from five (5) coal mining areas as follows:

1. Semirara - from active mine at Unong. Open-pit mine ± 12 meter seam

2. Southern Cebu - Luvimin mine. Underground ± 3.0 meter seam.

3. Bagacay, Samar - MMIC coal mine. Open-pit-mine ± 2.5 meter seam.

4. Malangas - PNOC mine in La Dicha. Underground ± 2.5 meter seam.

5. Bislig - PNOC mine. Underground ± 2.0 meter/seam, 4 seams (5, I, H & K).

All the coal mines are essentially single seam except for Bislig which has four (4) seams - 5, I, H & K.

The sampling procedures adopted are shown in Annex 1. These are essentially the ones recommended by Development Science Inc. (DSI) consultants, except that the bulk sample size was increased to 1500 kg instead of 150 lbs.

Samples from coal mines with single seam (Semirara, Southern Cebu, Bagacay, and Malangas) are about 40 kgs each. Some of these samples were divided into 2 portions (a & b) for convenience in handling. Both portions must be combined to reconstitute the sample thus, Sample 1(a) + Sample 1(b) Sample.

Main Office:
8th Floor, Bankmer Building, Ayala Avenue
Makati, Metro Manila Philippines/Tel. 883334, 895164, 882950, 882989, 883239, 883364

Management Training Services:
7th Floor, Combank Building, Ayala Avenue
Makati, Metro Manila, Philippines
Tel. 892237, 895168, 876202, 863189, 857264

MCC P.O. Box 370 Makati, Metro Manila/Cable: EDEFOINC

From Bislig, the weight of samples (4 each) from the various seams are as follows:

Seam 5	- 20 kgs
Seam I	- 10 kgs.
Seam H	- 10 kgs.
Seam K	- 10 kgs
TOTAL	50 kgs.

The quantities are based on the relative mine outputs from the various seams. The samples from each seam were packed separately so they may be analyzed and processed independently if desired.

A composite sample, however, can be prepared by reconstituting the samples in the same ratio of 2:1:1:1.

The coal sample list is shown in Annex 2. Each set of samples consists of individual samples from each of the sources, corresponding to the same code number (e.g. Sample 1 from Semirara, Sample 1 from Southern Cebu, etc.), as follows:

Sample Source	Approx. Wt. Kg.
o Semirara	40
o Southern Cebu	40
o Bagacay	40
o Malangas	40
o Bislig	
- Seam 5	20
- Seam I	10
- Seam H	10
- Seam K	10
TOTAL	210 kgs.

We hope that with the sample coding used, the tests and analyses can proceed smoothly.

Very truly yours,

PURITA M. FESTIN
Project Director

cc: Mr. Charles Bliss
Mr. Laurence Ervin

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BULK SAMPLING PROCEDURES FOR CWM TEST SAMPLES

I. Basis

It has been determined that about 1500 kg samples from five (5) mines will be prepared. Four (4) 40-kg samples will initially be sent by air freight to Brookhaven National Laboratory (BNL) which will later on distribute the samples to three test laboratories in the United States.

BNL will provide further instructions on the shipment of the remaining coal as soon as preliminary results on the samples are available. Indications are that BNL will specify the two coals for which the remaining samples are to be shipped.

II. Locations

To cover the range of coals likely to be considered for CWM use, it is suggested that samples from five active mining areas be taken as follows:

1. Semirara - Sample from active mine at Unong. Open-pit mine ± 12 meter seam.

2. Southern Cebu - Select Luvimin Mining Company mine. Underground mine ± 3.0 meter seam.

3. Samar - MMIC coal mine area near Bagacay - Developed coal seam - 2.5 meters thick. Open-pit mine.

4. Surigao - Bislig Area - PNOC mine, underground ± 2.0 meter seam.

5. Malangas - PNOC mine, underground ± 3.0 m.

III. Sampling Procedure

The idea is to sample a fresh coal face at each time, prepare that sample, and split out portions to end up with equal samples.

Take a sample of the entire coal seam from an active mining face or fresh new coal face at a location the mine manager or engineer believes would be representative of the present mining area. (No samples are to be taken from stock-piles, bins, or conveyor belts).

Take a channel cut of the entire seam from top to bottom as follows:

1. Clean up around the coal face and floor and lay down a collecting canvas or cloth (piece of canvas or material about 12' square).

2. Mark the channel to be cut from top to bottom with chalk or other marker. Then cut out this channel for the full height of the seam.

   With different seam heights, different widths of channel cut are taken to get approximately one (1) cu.m. of sample.

3. Chip out the sample by hand with hammer and chisel or pneumatic hammer, if available.

4. The sample will fall on the collecting cloth. From that, it should be put in a clean container or containers (barrel, box, etc.) and taken to an area where there is a flat, clean, concrete slab or other smooth, clean, uncontaminated surface. (Sweep surface absolutely clean with a broom).

5. At this location, the sample should be crushed so that every particle passes + 1/2". With these relatively small samples and coal, this can be done by hand on the flat surface by breaking up the lumps with a hammer. If there is a 1/2" screen available, the sample can be screened to be sure everything is 1/2". If not, the engineers judgment that everything is 1/2" is alright.

6. The sample is now mixed, coned and quartered, and split as follows:

   a) Pile up the sample in the shape of a cone (with square point shovels).

   b) Flatten out the pile with a shovel and mark into quarters.

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c) Form a new coned pile to one side by taking one quarter and placing it, then the opposite quarter, then the third quarter and then the fourth. Always place material on the top to form another coned pile. Be sure to take all the fine materials.

d) Repeat procedures (b) and (c) four times and end up with a coned pile.

e) Flatten the cone and mark into quarters. This will prepare four samples, each of about 375 kgs.

7. Place the first three (3) quarters (with shovels) into double plastic bags in a steel drum. Be sure all fines from each quarter go into the sample. Use a broom to sweep fines into the shovel and into the sample.

   Seal each plastic bag carefully (the inner one first, then the outer one). Close the drums and seal. Label samples as QTR2, QTR3, and QTR4 and store in a secure area at the mine site.

8. The remaining one quarter is now mixed, coned, quartered and split as in 6.

   a) With a small scoop or shovel, take a grab sample of about three pounds from the center of each quarter. Place these all together in a double plastic bag and seal.

      This provides the ± 10 lb. sample to remain in the Philippines for analysis.

   b) Form a new coned pile (as in 6c) with the remaining sample. Flatten it out and mark into quarters.

   c) Combine opposite quarters to come up with two (2) samples each of about 180 kgs.

   d) Place one of the samples into double plastic bags in steel drum (labeled QTR1-1) and store together with QTR2, QTR3 and QTR4.

9. The remaining 180 kg sample is now mixed, coned, quartered and split as in 6. This will prepare four (4) final samples, each of about 40-45 kgs to be sent to Brookhaven National Laboratory.

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NOTES:

1.. The sequence of coning and quartering is illustrated in Figure 1.

2.. All steps 1-9 should be completed as rapidly as possible all in one day or less. Be sure all samples are properly marked.

MATERIALS NEEDED:

- Mixing and collecting cloth 12' x 12' canvas
- Shovels, hammers, picks, broom
- Plastic bags - large, heavy duty, ± 100 pieces
- 1/2" screen
- Drums for sample shipping (55-gallon drums)

COAL SAMPLE LIST

1) Semirara (4 samples, approx. 40 kgs. each)

   Sample 1
   Sample 2
   Sample 3
   Sample 4

2) Southern Cebu (4 samples, approx. 40 kgs each)

   Sample 1
   Sample 2
   Sample 3
   Sample 4

3) Bagacay, Samar (4 samples, approx. 40 kgs. each)

   Sample 1
   Sample 2
   Sample 3
   Sample 4

4) Malangas (4 samples, approx. 40 kgs. each)

   Sample 1
   Sample 2
   Sample 3
   Sample 4

5) Rislig (16 samples)

   <u>Seam 5</u> (4 samples, approx. 20 kgs. each)

   Sample 1
   Sample 2
   Sample 3
   Sample 4

   <u>Seam I</u> (4 samples, approx. 10 kgs. each)

   Sample 1
   Sample 2
   Sample 3
   Sample 4

Seam H (4 samples, approx. 10 kgs. each)

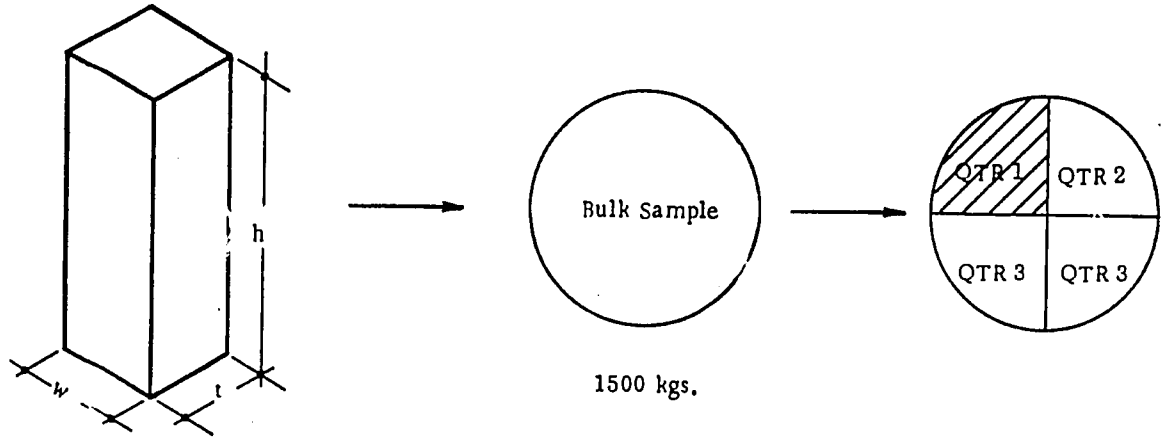
Sample 1
Sample 2
Sample 3
Sample 4

Seam K (4 samples, approx. 10 kgs. each)

Sample 1
Sample 2
Sample 3
Sample 4

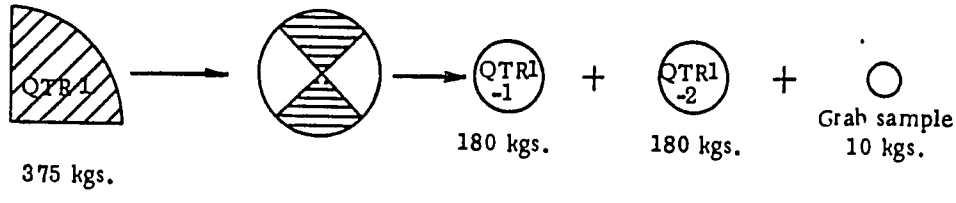
Figure 1

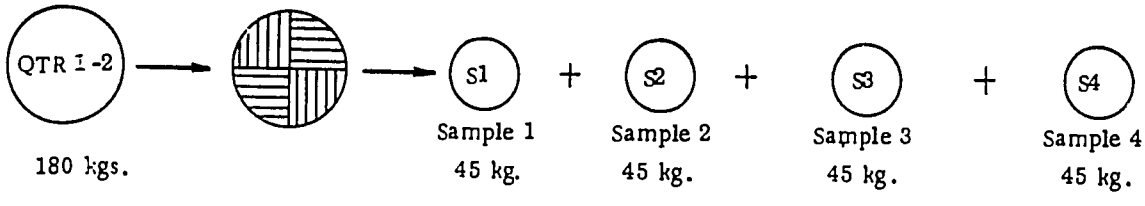
SAMPLE PREPARATIONS



Bulk Sample (Channel Cut)

h x w x t = 1.0 cu.m.





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APPENDIX II

LETTER REPORTS ON THE RESULTS OF HOT WATER DRYING PROCESS TESTS PERFORMED AT THE UNIVERSITY OF NORTH DAKOTA

Energy Research Center
University of North Dakota

October 23, 1984

Mr. Tom Butcher
Brookhaven National Labs
Building No. 120
Upton, NY 11973

Dear Mr. Butcher:

Enclosed you will find a dilute hot-water-dried coal slurry made from the Semirara coal you sent us, and also a rheogram for a concentrated portion of the product obtained using a Haake viscometer. The maximum pourable solids loading was 56.6 percent for this slurry (measured on a "bone-dry" solids basis).

The cold-charge autoclave test using your Phillipine coal was made on October 8. Equal quantities of coal and deionized water were charged to the autoclave. The autoclave and contents were brought to 330°C and maintained at temperature for 15 minutes. The maximum pressure reached during the test was 2135 psig. On October 9, the day following the test, 0.92 cubic feet of gas was vented from the autoclave, sampled, and analyzed. A copy of the gas analysis is included with the slurry sample. As you can see, it consists mostly of carbon dioxide. We have found this to be true for all the coals we have tested to date. A sample of the recovered product was filtered over a Buchner funnel, and the remaining unfiltered product is being sent to you. The filter cake and process water are being analyzed by our in-house labs.

As I mentioned on the phone, I will have the equilibrium moisture results next week and will send you a copy. The coal and wastewater analyses should be complete in about a month. After we receive these results, we will complete the material balance for this run and send you the results.

I would like to take this opportunity to invite you to visit us at the Energy Research Center. We are presently beginning the operation of a 200-pound-per-hour hot-water drying process development unit using a North Dakota lignite.

Sincerely,

Dana J. Maas

Dana J. Maas
Research Engineer
Coal-Water Slurry Preparation

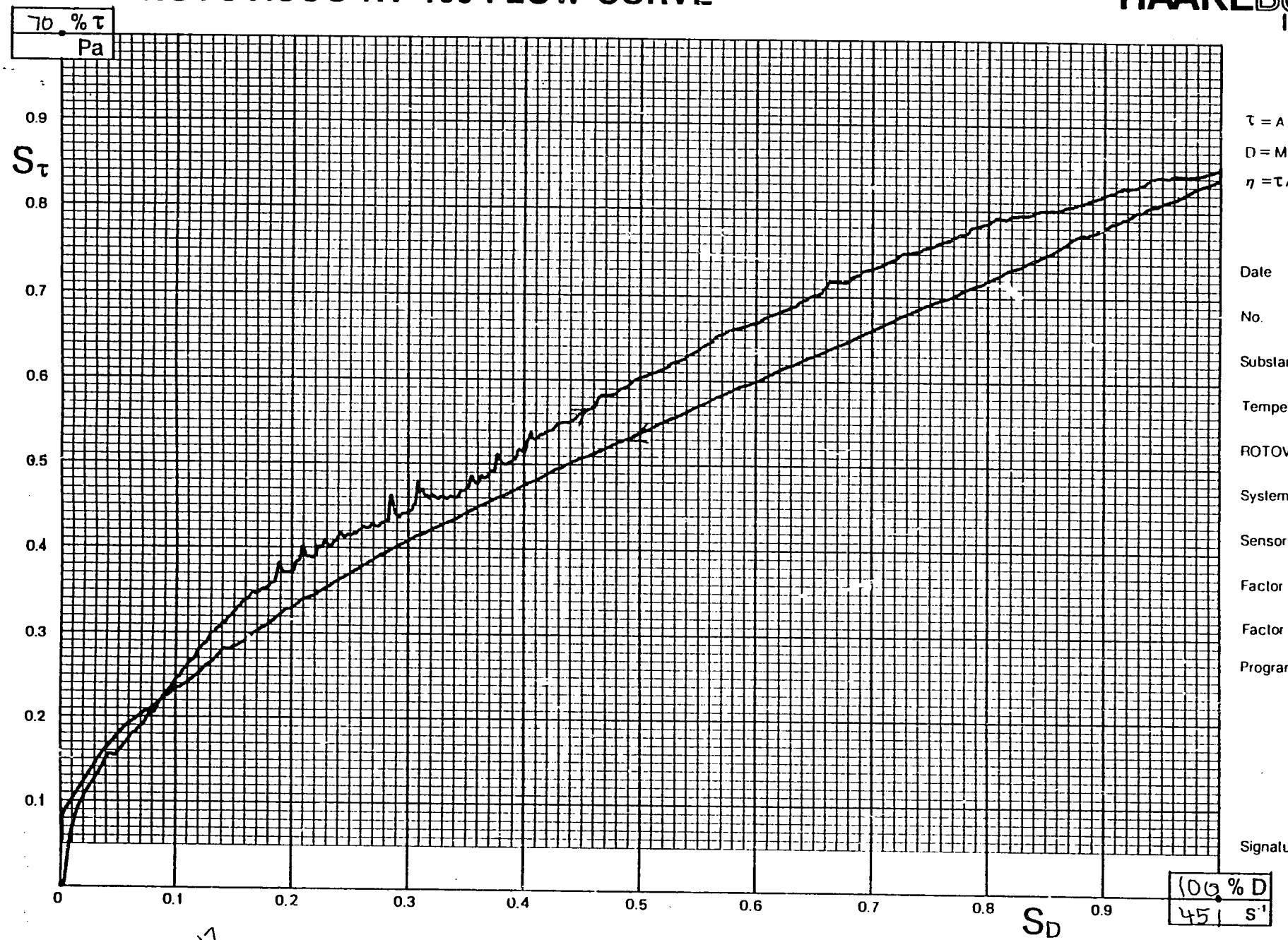
3 Enclosures

Box 8213, University Station / Grand Forks, North Dakota 58202 / (701) 777-5000

62

ROTOVISCO RV-100 FLOW CURVE

HAAKEBUCHLER Instruments Inc.



$\tau = A \cdot \%\tau \cdot S_\tau$ [Pa]

$D = M \cdot \%D \cdot S_D$ [s^{-1}]

$\eta = \tau / D$ [Pa·s]

330°C FINE

Date	10/25/84
No.	56.6%
Substance	~~Semi-cured~~ HWD
Temperature	276°C
ROTOVISCO RV	100
System	MS00
Sensor system	MV1
Factor A	3.74
Factor M	4.51
Program times:	
T_1	0.2
T_2	2.0
T_3	0.1
Signature	[illegible]

17
10:30

Order No 10-87-911

HAAKE BUCHLER INSTRUMENTS, INC.

244 Saddle River Road • P.O. Box 549 • Saddle Brook, N.J. 07662 • (201) 843-2320

RV-847A/982

COAL LIQUIDS LABORATORY

GAS ANALYSIS REPORT

SAMPLE IDENTIFICATION:
COLD CHARGE
HWD 28 #2

DATE: 10-10-84

NORMALIZED MOLE PER CENT

HYDROGEN	1.25
CARBON DIOXIDE	89.19
PROPANE	0.00
PROPYLENE	0.27
ISO-BUTANE	0.12
CARBONYL SULFIDE	0.00
N-BUTANE	0.02
HYDROGEN SULFIDE	0.87
1-BUTENE	0.40
T-2-BUTENE	0.01
ISO-PENTANE	0.01
C-2-BUTENE	0.00
PENTANE	0.00
ETHYLENE	0.10
ETHANE	0.26
OXYGEN	0.21
NITROGEN	2.92
METHANE	2.71
CARBON MONOXIDE	1.36
AMMONIA	0.30

NORMALIZED AIR-FREE MOLE PER CENT

HYDROGEN	1.29
CARBON DIOXIDE	92.07
PROPANE	0.00
PROPYLENE	0.28
ISO-BUTANE	0.12
CARBONYL SULFIDE	0.00
N-BUTANE	0.02
HYDROGEN SULFIDE	0.90
1-BUTENE	0.41
T-2-BUTENE	0.01
ISO-PENTANE	0.01
C-2-BUTENE	0.00
PENTANE	0.00
ETHYLENE	0.10
ETHANE	0.27
METHANE	2.80
CARBON MONOXIDE	1.40
AMMONIA	0.31

CALCULATED HEATING VALUE, BTU/SCF

SATURATED:	71.4
DRY:	72.6

CALCULATED SPECIFIC GRAVITY:	1.4653
CALCULATED AVERAGE MOLECULAR WEIGHT:	41.86

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Energy Research Center
University of North Dakota

December 12, 1984

Mr. Tom Butcher
Building NO. 120
Brookhaven National Labs
Upton, NY 11973

Dear Mr. Butcher:

This letter provides you the rest of the results of a hot-water autoclave test that was completed using Philippine coal. I think you will find the results interesting.

Run conditions were supplied in a previous letter, along with a dilute slurry sample from the run, gas analysis, and a rheogram. Since that time, the lab analyses of the filter cake and process water have been completed. The results of these analyses are attached, along with an "as-measured" and adjusted material balance for the run.

Several results of the test are noteworthy. The adjusted material balance indicated that about 11 percent of the moisture and ash-free (Maf) coal was converted to carbon dioxide. The sodium in the coal was reduced by 63 percent during hot-water drying, and the sulfur by 14 percent. The yield of Maf coal was 89 percent. The heating value of the product went up from 10,600 Btu per pound for the raw coal to 11,600 Btu per dry pound, accounting for 98 percent recovery of the original heat content. The equilibrium moisture of the product was 11.5 percent. These numbers are typical of the low-rank coals we have tested at this drying temperature, although the reduction in sodium is higher than we have observed for other coals.

Further tests to study the effects of temperature would be interesting, as would additional characterization studies of the dried coal product to determine functional group decomposition. If you have any questions about this test, please feel free to call me at (701) 777-5120.

Sincerely,

Dana J. Maas

Dana J. Maas
Research Engineer
Coal-Water Slurry Preparation

Enclosure

Box 8213, University Station / Grand Forks, North Dakota 58202 / (701) 777-5000

COAL ANALYSIS

		FEED COAL	FILTER CAKE
PROXIMATE	AR WGT	%	%
Moisture	------	10.80	20.10
Ash	------	11.92	8.30
V.M.	------	38.20	36.70
F.C.	------	37.70	35.40
ULTIMATE	DRY WGT	%	%
Carbon	------	52.54	64.52
Hydrogen	------	4.07	3.52
Nitrogen	------	1.08	1.04
Sulfur	------	2.96	1.77
Oxygen	------	17.42	12.88
ASH	WGT OF ASH	%	%
SiO2	------	42.60	34.20
Al2O3	------	20.10	22.90
Fe2O3	------	3.30	2.70
TiO2	------	1.40	1.50
P2O5	------	0.10	0.40
CaO	------	5.40	5.00
MgO	------	4.20	4.00
Na2O	------	2.70	1.10
K2O	------	1.70	1.90
SO3	------	5.90	6.90

Best Available Document

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RUN NO. [illegible] SCHWOZBREIGM

PRODUCT GAS ANALYSIS

		Mol%	wt%
H2	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
CH4	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
H2S	[illegible]	[illegible]	[illegible]
NH3	[illegible]	[illegible]	[illegible]

Sample	Start [illegible]	[illegible]
Mole	Weight [illegible]	[illegible]
Grams	[illegible]	[illegible]

Carbon	[illegible]
Hydrogen	[illegible]
Nitrogen	[illegible]
Sulfur	[illegible]
Oxygen	[illegible]

PROCESS WATER ANALYSIS mg/L

Br	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
Nitrate	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
Thio.	[illegible]	0.00
Phenol	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
Methanol	[illegible]	[illegible]
Ethanol	[illegible]	[illegible]
Acetone	[illegible]	[illegible]
Isopropanol	[illegible]	[illegible]
Acetonitrile	[illegible]	0.00
Propanol	[illegible]	0.00
MEK	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]
Ca	[illegible]	[illegible]
Fe	[illegible]	[illegible]
K	[illegible]	[illegible]
Mg	[illegible]	[illegible]
Na	[illegible]	[illegible]
Si	[illegible]	[illegible]
TOTAL CARBON	[illegible]	[illegible]
TOC	[illegible]	4110.00

Best Available Document

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RUN NO. ---- COHWD28SR33OM Driving Method: HOT WATER

MEASURED MATERIAL BALANCE

		IN	OUT	DIFF	YIELD-WT% of MAF Dry
C	----	0.00	0.05	0.05	0.00
[illegible]	----	0.00	0.02	0.02	0.00
[illegible]	----	0.00	0.02	0.02	0.00
[illegible]	----	0.00	0.15	0.15	0.02
CO2	----	0.00	0.20	0.20	0.02
[illegible]	----	0.00	0.20	0.20	0.02
[illegible]	----	0.00	0.17	0.17	0.02
[illegible]	----	0.00	0.17	0.17	0.02
CH4	----	0.00	0.48	0.48	0.07
H2S	----	0.00	0.48	0.48	0.07
NH3	----	0.00	0.18	0.18	0.02
Thio	----	0.00	0.00	0.00	0.00
Phenol	----	0.00	0.12	0.12	0.02
o-Cresol	----	0.00	0.01	0.01	0.00
m-Cresol	----	0.00	0.02	0.02	0.00
Methanol	----	0.00	0.34	0.34	0.05
Ethanol	----	0.00	0.00	0.00	0.00
Acetone	----	0.00	0.29	0.29	0.04
Isopropanol	----	0.00	0.02	0.02	0.00
Acetonitrile	----	0.00	0.00	0.00	0.00
Propanol	----	0.00	0.00	0.00	0.00
MEK	----	0.00	0.02	0.02	0.00
Water	----	1087.10	1064.41	-22.69	0.18
Oil	----	0.00	0.00	0.00	0.00
[illegible]	----	[illegible]	[illegible]	[illegible]	[illegible]
MAF Dry	----	715.15	[illegible]	[illegible]	[illegible]
TOTAL	----	1875.00	1876.45	[illegible]	

					% Recov.
Carbon	----	520.94	514.65	-6.29	98.79
Hydrogen	----	154.46	152.44	-2.02	98.69
Nitrogen	----	9.97	12.85	2.87	[illegible]
Sulfur	----	7.19	9.58	[illegible]	[illegible]
Si	----	22.28	20.27	-2.10	[illegible]
Al	----	14.98	15.38	[illegible]	[illegible]
Fe	----	4.58	4.75	0.18	[illegible]
Ti	----	0.95	0.91	-0.03	[illegible]
P	----	0.20	0.19	-0.02	[illegible]
Ca	----	5.18	5.59	0.41	[illegible]
Mg	----	2.59	[illegible]	[illegible]	[illegible]
Na	----	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	----	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	----	[illegible]	[illegible]	[illegible]	[illegible]
TOTAL	----	1875.00	1876.45	[illegible]	[illegible]

Tied H2O	----	113.57	291.02	177.44
Free H2O	----	947.50	774.38	-173.12

Coal Used: SEMIARA

Driving Temp. C: 320

Driving Press. Psig: 1878

Driving Res. Time: 15

Pct of Original in Dry Basis

S	74.58	Fe	80.02
Si	80.44	Ca	112.82
Al	89.30	Mg	98.73
Fe	103.89	Na	16.57
Ti	96.45	K	100.61

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[illegible] ANALYSIS REPORT

GF# 84-1859 LAB# 1245

DATE SAMPLED :10-09-84 DATE RECEIVED :10-09-84 DATE OF REPORT :10-31-84

SAMPLE DESCRIPTION :FC HWD SEMIRARA COAL 330C

SUBMITTER :D.MAAS SAMPLE RUN NUMBER :CWS-SR-028

AIR DRY LOSS=24.6

	AS RUN	AS RECD	MOIST FREE	MOIST,ASH FREE
PROXIMATE ANALYSIS				
MOISTURE	4.6	28.1	N/A	N/A
VOLATILE MATTER	35.4	26.7	37.1	42.9
FIXED CARBON (IND)	47.0	35.4	49.3	57.1
ASH	13.0	9.8	13.6	N/A
ULTIMATE ANALYSIS				
HYDROGEN	4.87	6.37	4.52	5.23
CARBON	63.46	47.85	66.52	76.99
NITROGEN	1.63	1.23	1.71	1.98
[illegible]	.73	.55	.77	.89
[illegible] (IND)	16.39	34.22	12.88	14.91
ASH	13.0	9.8	13.6	N/A
HEATING VALUE (BTU/LB)				
[illegible]	11070	8347	11604	13430
CALCULATED CALORIC VALUE	11051	8333		

AS RECD H NOT INCLUDING H FROM MOISTURE= 3.25
AS RECD O NOT INCLUDING O FROM MOISTURE= 9.27

SULFUR FORMS

SULFATE	N/A
PYRITIC	N/A
ORGANIC	N/A

COMMENTS:

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UND ENERGY RESEARCH CENTER
X-RAY FLUORESCENT ANALYSIS

SAMPLE NUMBER: GF-84-1859 REPETITION: 1

DATE ANALYZED: 11-13-84 RUN NUMBER: CWS-SR-028

DATE SAMPLED: 10-09-84

SAMPLE DESCRIPTION: CWS-SR-028 FC HWD SEMIRARA COAL 330C

SAMPLE SUBMITTER: D.MAAS

ANALYST: ALS

	%ELEMENTAL	% OF ASH	% AS OXIDES % OF ASH (NORM)	% AS OXIDES % SO3 FREE
SILICA,SIO2	19.9	42.6	42.7	46.9
ALUMINUM OXIDE,AL2O3	13.2	24.9	24.9	27.3
FERRIC OXIDE,FE2O3	4.7	6.7	6.7	7.4
TITANIUM OXIDE,TIO2	0.9	1.5	1.5	1.6
PHOSPH. PENTOXIDE,P2O5	0.2	0.4	0.4	0.4
CALCIUM OXIDE,CAO	5.7	7.9	8.0	8.8
MAGNESIUM OXIDE,MGO	2.4	4.0	4.0	4.4
SODIUM OXIDE,NA2O	0.8	1.1	1.1	1.2
POTASSIUM OXIDE,K2O	1.6	1.9	1.9	2.1
SULFUR TRIOXIDE,SO3	3.6	8.[illegible]	8.9	9.0
		[illegible]	100.1	100.1

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UNDERC COAL-ANALYSIS REPORT

GF# 84-1853 LAB# 1239

DATE SAMPLED :10-08-84 DATE RECEIVED :10-08-84 DATE OF REPORT :10-31-84

SAMPLE DESCRIPTION :RAW SENIRARA COAL

SUBMITTER :D.MAAS SAMPLE RUN NUMBER :CWS-SR-000

AIR DRY LOSS= 0.0

	AS RUN	AS RECD	MOIST FREE	MOIST,ASH FREE
PROXIMATE ANALYSIS				
MOISTURE	12.2	12.2	N/A	N/A
VOLATILE MATTER	38.2	38.2	43.5	50.3
FIXED CARBON (IND)	37.7	37.7	42.9	49.7
ASH	11.9	11.9	13.6	N/A
ULTIMATE ANALYSIS				
HYDROGEN	5.19	5.19	4.37	5.05
CARBON	54.98	54.98	62.62	72.48
NITROGEN	.96	.96	1.09	1.27
SULFUR	.79	.79	.90	1.04
OXYGEN (IND)	26.14	26.14	17.42	20.16
ASH	11.9	11.9	13.6	N/A
HEATING VALUE (BTU/LB)				
BTU	9322	9322	10617	12289
CALCULATED CALORIC VALUE	9559	9559		

AS RECD H NOT INCLUDING H FROM MOISTURE= 3.83
AS RECD O NOT INCLUDING O FROM MOISTURE=15.29

SULFUR FORMS

SULFATE	N/A
PYRITIC	N/A
ORGANIC	N/A

COMMENTS:

UND ENERGY RESEARCH CENTER

X-RAY FLUORESCENT ANALYSIS

SAMPLE NUMBER: GF-84-1853 REPETITION: 1

DATE ANALYZED: 10-27-84 RUN NUMBER: CWS-SR-000

DATE SAMPLED: 10-08-84

SAMPLE DESCRIPTION: CWS-SR-000 RAW SEMIRARA COAL

SAMPLE SUBMITTER: D.MAAS

ANALYST: ALS

	%ELEMENTAL	% AS OXIDES % OF ASH	% OF ASH (NORM)	% SO3 FREE
SILICA,SIO2	20.4	43.6	42.5	47.2
ALUMINUM OXIDE,AL2O3	13.7	25.8	25.1	27.9
FERRIC OXIDE,FE2O3	4.2	6.0	5.8	6.4
TITANIUM OXIDE,TIO2	0.9	1.4	1.4	1.6
PHOSPH. PENTOXIDE,P2O5	0.2	0.4	0.4	0.4
CALCIUM OXIDE,CAO	4.7	6.6	6.4	7.1
MAGNESIUM OXIDE,MGO	2.6	4.3	4.2	4.7
SODIUM OXIDE,NA2O	2.0	2.7	2.7	3.0
POTASSIUM OXIDE,K2O	1.5	1.8	1.7	1.9
SULFUR TRIOXIDE,SO3	4.1	10.1	9.9	0.0
TOTALS		102.7	100.1	100.2