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GASIFIER SLAG AND FEEDSTOCK CHARACTERIZATION

Final Technical Report

By
M. S. Lancet
D. E. Havekotte
F. A. Sim
R. Sivasubramanian

March 1983

Work Performed Under Contract No. AC05-78OR03054

International Coal Refining Company
Allentown, Pennsylvania

Technical Information Center
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PROGRAM OBJECTIVES

In the 6,000-ton-per-day (tpd) SRC-I Demonstration Plant, 1,600 tpd of Kerr-McGee ash concentrate supplemented with Kentucky #9 coal is to be gasified to generate makeup hydrogen. KMAC has not been commercially gasified, and data on the gasification characteristics of KMAC or similar materials do not exist. The objective of this program, conducted by Conoco Coal Development Company, was to generate needed information for the design of gasifiers using coal and KMAC as feed materials. Ten samples comprising four coals and six KMACs were characterized in this program. The criteria used in sample selection were:

- ° to test a variety of coals and KMACs,
- ° to select some coals that had been liquefied in the ICRC CPDU and at Wilsonville,
- ° to determine differences between coal and its derivative KMAC,
- ° to determine the effect of Na_2CO_3 addition on KMAC properties, and
- ° to test some samples in this program that had also been tested at GKT.

The program was divided into two tasks. In the first task, detailed physical, chemical, and petrographic analyses were performed on all samples. In addition, CO_2 -carbon reactivities of chars produced during the pressurized coking at simulated gasifier conditions were determined for all 10 samples. In task 2, the slag viscosity/temperature relationship for the partially reduced ash from each material tested was ascertained.

The attached final report from Conoco describes the experiments in detail and summarizes their conclusions. This report represents the satisfactory completion of this program at Conoco.

ONGOING WORK IN COAL, ASH CONCENTRATE, AND SLAG CHARACTERIZATION

ICRC has compared data generated by GKT on some of the same samples used in this program. ICRC has also used correlations from the literature to predict slag viscosity as a function of temperature based on the elemental analysis of ash. These predictions have been compared to the viscosity data generated by Conoco and report number DOE/OR/03054-57 entitled "Subcontracted R&D Task Summary Report: Coal, KMAC, and Slag Physical Properties" authored by R. Sivasubramanian of ICRC and dated September 1983 has been issued summarizing the subject physical properties.

In addition to the summary report identified above, heat capacity and enthalpy data on coal, ash concentrate, and slag has been generated and is presented in report number DOE/OR/03054-73 entitled "Subcontracted R&D Final Report: Gasifier Slag and Feedstock Physical Properties" authored for ICRC by Messrs. K. C. Mills, F. H. Putland, and M. J. Richardson of The National Physical Laboratory, London, England and dated September 1983.

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Section 1

SUMMARY

This is a report of work by Conoco Coal Development Company to determine the properties of coals and residual carbon from coal liquefaction as gasifier feedstocks. This work was funded by subcontract to the International Coal Refining Company (ICRC), a joint venture of Air Products and Chemicals, Inc., and Wheelabrator-Frye, as part of their prime contract with the U.S. Department of Energy to design, build and operate the SRC-I demonstration plant. The purpose of this subcontract work was to establish the gasification reactivities and ash and slag properties of several Midwestern coals and Kerr-McGee ash concentrates which would be available to the SRC-I demo plant as gasifier feedstocks to produce process hydrogen. The ash concentrates are high ash by-products of the Kerr-McGee critical solvent deashing (CSD) process which is a leading candidate for solids removal at the SRC-I demo plant. The Kerr-McGee process has been developed and tested for several years at the Wilsonville, Alabama, SRC-I pilot plant, from which the samples used in our work were obtained.

Four Midwestern coals, three from Kentucky and one from Illinois, and six Kerr-McGee ash concentrate samples produced at the SRC-I pilot plant in Wilsonville, Alabama, during the liquefaction of several Midwestern coals were characterized in this work. In addition to complete chemical analysis, a petrographic analysis was performed on each coal sample. CO_2 -carbon reactivities of chars produced during the pressurized coking at simulated gasifier conditions of all ten materials were determined. These reactivities were measured under pressure (300 psig) at 800°C, and at 950°C and 1 atmosphere. The results of the atmospheric high temperature reactivity determinations suggest that all materials tested should be sufficiently reactive for use in a Koppers-Totzek or Texaco slagging gasifier. The pressurized, 800°C reactivities are fairly low for every material tested. This is consistent with the reactivities of Eastern and Midcontinent bituminous coals and limits

the desirability of using these coals and Kerr-McGee ash concentrates as feedstocks to lower temperature, non-slugging gasifiers. The low value of the pressurized CO_2 -carbon reactivity, when coupled with the low fusion temperatures of the ashes from all of these materials, virtually precludes their economic useage in a conventional (dry-bottom) Lurgi gasifier.

The slag viscosity-temperature relationship was determined for the partially reduced (iron present as FeO) ash from each material tested. A total of fifteen viscosity measurements, including duplicates, were made. All but one ash, that of the FSM-15 (Kentucky 14) coal, form well-behaved slags which remain Newtonian liquids until cooled to just above the freezing point. The FSM-15 ash demonstrates a significant "shortness" or steepening of the viscosity curve at temperatures nearing the melting point of the ash. The remainder of the ashes are glassy in nature through most of their liquid phases. The frozen slag from each material is quite homogeneous with no detectable differences in chemical composition or physical appearance. Scanning electron micrographs revealed no elemental segregation in the slags.

One sample of Kerr-McGee ash was contaminated with sodium. The added sodium is almost surely sodium carbonate which is added at Wilsonville during liquefaction to control chloride release. Most of this sodium is volatilized during the determination of slag viscosities. The gasification of materials with such high levels of added sodium by high temperature (i.e. slugging) processes will probably require some means of sodium capture to avoid the formation of deposits in associated downstream facilities. Alternatively, this may preclude the use of Na_2CO_3 to control chloride corrosion in the SRC-I process if the residual carbon is to be gasified.

A working correlation of slag viscosity, temperature and chemical composition was unobtainable because of the limited data available. It is believed, however, that such a correlation can be derived as more slags are added to the data base.

Section 2

PHYSICAL AND CHEMICAL ANALYSIS OF TESTED MATERIALS

Ten materials were examined in this program. These included six samples of Kerr-McGee ash concentrates produced in the SRC pilot plant at Wilsonville, Alabama, during the liquefaction of various coals, and four Midwestern bituminous coals. The chemical analyses of the six ash concentrates are given in Table 2-1. There is quite a large variation in the ash content of these materials, from about 22.5% in the KM-211 to a high of nearly 50% in the KM-143 sample. The fixed carbon values also show considerable variation with a spread of about 10% between the high of ~70% and the low of ~61% on an MAF basis. The elemental composition (ultimate analysis, MAF basis) of these materials is much more uniform with the major difference between samples being the sulfur content which goes from ~2.9% in KM-236 to ~8.3% in KM-143. The large majority of the sulfur in these ash concentrates is organic sulfur (Table 2-1).

Only three of the Kerr-McGee materials demonstrated any fluidity as determined by the Gieseler method.⁽¹⁾ Of these, only the KM-236, with a maximum fluidity of 665 DDPM, could actually be considered a moderately fluid material. This same sample, KM-236, which was produced during a run with Illinois No. 6 coal, is also the only one to demonstrate significant swelling properties as defined by the ASTM Free Swelling Index (FSI).⁽²⁾ The FSI value of 5 1/2 measured for this ash concentrate is actually quite a bit higher than that of the coal used in this run. This fact, together with the high fluidity of this material suggests that solvent removal may not have been as efficient as with the other samples.

The elemental composition of the ash derived from each of the Kerr-McGee ash concentrate samples is given in Table 2-2. These compositions are typical of the ashes from most Eastern and Midwestern U.S. bituminous coals. The alkali and alkaline earth contents, with the exception of KM-218 and KM-236 which appear to be contaminated with sodium or a sodium salt, are fairly low and the main constituents of these ashes are Fe_2O_3 , SiO_2 and Al_2O_3 .

The high sodium concentration in the ash from KM-218 was almost surely the result of addition of Na_2CO_3 at Wilsonville

to control chloride corrosion in the SRC-I process. The concentration of sodium in the ash concentrate can have a significant deleterious effect on gasifier performance as noted in Section 1, and described in more detail in Section 4.

The ASTM ash fusion temperatures are low under both oxidizing and reducing conditions, Table 2-2. These low ash fusion temperatures are desirable for the gasification of these materials in slagging type gasifiers such as Koppers-Totzek or Texaco.

The chemical analyses of the four coals included in this program are given in Table 2-3. For coals from four different seams, the chemical compositions of these materials are remarkably similar to each other. With the exception of the high moisture value of 9.7% for the Burning Star coal (a value typical for Illinois basin coals), the composition of the four coals are the same to within $\sim \pm 3\%$ absolute. This uniformity of composition is a rare occurrence even for coal samples within the same seam.

Three of the coals, Kentucky 9 and 14 and Illinois 6, are essentially nonfluid with maximum Gieseler fluidities of 2-7 DDPM, and demonstrate only a slight caking and swelling tendency ($\text{FSI} = 2 \frac{1}{2} - 3 \frac{1}{2}$). The Kentucky 11 coal is about an order of magnitude more fluid than the other coals with a significantly larger Free Swelling Index (FSI) of 5.

The elemental composition of the ashes from the four coals are given in Table 2-4. Here, in contrast to the chemical composition of the coals, we find significant differences. In particular, the iron and silica contents are quite different in each ash with Fe_2O_3 , ranging from 14.5% in the Burning Star ash to nearly 37% in the Kentucky 14 coal ash.

The composition of the ash from the Burning Star, Illinois No. 6 coal, is nearly identical to that of the Kerr-McGee ash concentrate from Run 236, KM-236. This was expected since the KM-236 material was obtained during a Wilsonville run which used the Burning Star, Illinois No. 6, as the feed coal.

The ASTM ash fusion temperatures, also given in Table 2-4, are quite low, as was the case for the ash from the Kerr-McGee ash concentrates. Based on ash melting characteristics, these too would be expected to perform well in slagging type coal gasifiers.

The petrographic analyses of the four coals used in this work are given in Table 2-5. Except for the Burning Star, Illinois 6 coal, all the coals studied were received already ground to -200 mesh (Tyler). Since this is considerably finer than the 20 mesh prescribed by the ASTM for petrographic analysis,^(3, 4) the petrographic data for the three Kentucky coals in Table 2-5 may differ from ASTM values.

Each coal is evaluated using the entity classification⁽³⁻⁵⁾ system in which the composition is expressed in terms of the volume percent of each petrographic entity present, and the reflectance of the vitrinoids is used as an index of the coal rank.

All types of vitrinoids, all resinoids and exinoids together with a third of the actual volume of semifusinoids are shown as reactive macerals on the basis of their thermal behavior. They soften and re-solidify during carbonization, whereas the inerts (fusinoids, micronoids, the mineral matter and the remaining semifusinoids) remain relatively unchanged.

Once the coal rank and physical parameters have been established, the behavior of the coal for certain technological processes can be predicted. The most common use of this data is to predict the coke stability.

Coke stability is largely controlled by two inherent qualities of the feed coal. These are the number of bond forming reactive entities and the bonding ability of these reactive entities. The bonding ability of each reactive group (vitrinoid type) is different and 18" coke oven tests were used to empirically predict the optimum amount of inerts that each vitrinoid type can bond. The composition balance index of a coal is determined by dividing the inerts present by the optimum amount of inerts required for the highest coke strength based

on the specific vitrinoids present. The strength index was defined by determining the relative strengths of cokes made from coals of various rank (as defined by vitrinoid reflectance type) and entity composition. Using the composition balance index and strength index, it is possible to predict the coke stability for a given coal or blend using microscopic data only. (5)

Empirical correlations are used to calculate the strength index based on the mean reflectance distribution of the reactive entities. The composition balance index gives the ratio of the actual and the empirical optimum content of the inert entities. A correlation of both indices as above gives the calculated ASTM stability of coke produced from the tested coal in a standard coke oven.

The calculated stabilities of cokes produced from the four coals in this study are so low as to preclude the use of any of them as the sole feedstock to a conventional slot oven. It is possible, however, that any or all of them could be used as part of a blended feedstock for the production of metallurgical coke.

TABLE 2-1
CHEMICAL ANALYSES OF KERR-McGEE ASH CONCENTRATES

Sample Identification	KM-143	KM-156	KM-162	KM-211	KM-218	KM-236
Coal of Origin	Indiana No. 5	Kentucky No. 6 + 11	Kentucky No. 9	Kentucky No. 9	Kentucky No. 9	Illinois No. 6
<u>Moisture, As Det. (wt %)</u>	0.91	2.38	2.80	2.08	2.55	1.29
<u>Proximate, Dry (wt %)</u>						
Ash	47.97	42.31	41.95	22.55	35.55	25.05
Volatile Matter	20.34	15.92	20.26	22.85	21.24	25.04
Fixed Carbon	31.69	41.77	37.79	54.60	43.21	49.91
<u>Proximate, MAF (wt %)</u>						
Volatile Matter	39.08	27.60	34.90	29.50	32.96	33.41
Fixed Carbon	60.92	72.40	65.10	70.50	67.04	66.59
<u>Ultimate, Dry (wt %)</u>						
Hydrogen	2.57	2.67	2.75	3.38	2.91	3.63
Carbon	43.15	49.84	47.27	63.81	53.75	63.29
Nitrogen	1.02	1.19	1.09	1.56	1.31	1.38
Oxygen (by diff.)	0.12	0.65	3.75	5.79	2.74	4.33
Sulfur	5.13	4.14	3.19	2.92	3.74	2.32
Ash	47.97	42.31	41.95	22.54	35.55	25.05
<u>Ultimate, MAF (wt %)</u>						
Hydrogen	4.94	4.62	4.74	4.36	4.51	4.85
Carbon	83.01	85.01	81.43	82.37	83.39	84.44
Nitrogen	1.95	2.06	1.08	2.02	2.04	1.84
Oxygen (by diff.)	1.83	3.13	6.90	7.98	4.69	5.99
Sulfur	8.26	5.18	5.05	3.27	5.37	2.88
<u>Sulfur Forms, Dry (wt %)</u>						
Pyritic	0.37	0.12	0.19	0.27	0.11	0.12
Sulfate	0.45	1.02	0.87	0.12	0.16	0.04
Organic (by diff.)	4.31	3.00	2.93	2.53	3.47	2.16
<u>HHV, Dry (Btu/lb)</u>	7,759	8,737	8,725	11,111	9,450	11,151
<u>Gieseler Fluidity (DDPM)</u>	0	0	3.6	0	0.8	665
<u>Free Swelling Index</u>	1	1	1	1½	1½	5½

TABLE 2-2
PHYSICAL AND CHEMICAL PROPERTIES OF ASHES DERIVED FROM
KERR-McGEE ASH CONCENTRATES

Sample Identification	KM-143	KM-156	KM-162	KM-211	KM-218	KM-236
Coal of Origin	Indiana	Kentucky	Kentucky	Kentucky	Kentucky	Illinois
	No. 5	No. 6 + 11	No. 9	No. 9	No. 9	No. 6
<u>Elemental Composition (wt %)</u>						
Na ₂ O	0.48	0.50	0.58	0.67	4.48	1.35
K ₂ O	2.08	2.59	2.95	2.13	2.09	1.84
CaO	4.80	1.53	1.73	2.74	2.68	6.97
MgO	0.84	0.76	1.09	0.72	0.73	0.93
Fe ₂ O ₃	22.56	22.10	15.96	21.43	20.88	15.33
TiO ₂	0.72	0.80	0.90	1.05	0.96	0.94
P ₂ O ₅	0.18	0.23	0.20	0.07	0.07	0.08
SiO ₂	47.50	48.79	53.86	45.26	45.81	45.19
Al ₂ O ₃	16.45	18.24	17.88	16.56	21.56	20.77
SO ₃	4.39	0.90	1.15	2.37	2.41	4.20
Cl ⁻	0.03	0.38	0.42	0.17	0.27	0.15
<u>Fusion Temperatures (°F)</u>						
<u>Reducing Atmosphere</u>						
Initial Deformation	1940	1980	2060	1940	2030	2030
Softening	2160	2260	2280	2060	2130	2120
Hemispheric	2220	2360	2340	2160	2240	2160
Fluid	2240	2380	2406	2320	2290	2280
<u>Oxidizing Atmosphere</u>						
Initial Deformation	2240	2480	2300	2180	2250	2250
Softening	2390	2500	2500	2450	2340	2340
Hemispheric	2440	2530	2560	2510	2400	2360
Fluid	2500	2560	2620	2560	2470	2420

TABLE 2-3
CHEMICAL ANALYSES OF ICRC COALS

Sample Identification	FSK-9 APRC-17	FSM-23 APRC-19	FSM-15 APRC-20	Burning Star
Coal Seam	Kentucky No. 9	Kentucky No. 11	Kentucky No. 14	Illinois No. 6
<u>Moisture, As Det. (wt %)</u>	2.53	2.08	2.15	9.69
<u>Proximate, Dry (wt %)</u>				
Ash	10.20	8.17	8.05	10.96
Volatile Matter	39.98	40.08	41.11	43.49
Fixed Carbon	49.82	51.75	50.84	45.65
<u>Proximate, MAF (wt %)</u>				
Volatile Matter	44.52	43.65	44.71	48.74
Fixed Carbon	55.48	56.35	55.29	51.26
<u>Ultimate, Dry (wt %)</u>				
Hydrogen	4.80	5.06	5.04	4.63
Carbon	71.15	74.13	72.75	69.97
Nitrogen	1.58	1.59	1.65	1.47
Oxygen (by diff.)	9.37	8.05	8.83	9.89
Sulfur	2.90	3.00	3.68	3.08
Ash	10.20	8.17	8.05	10.96
<u>Ultimate, MAF (wt %)</u>				
Hydrogen	5.35	5.51	5.48	5.21
Carbon	79.23	80.73	79.12	78.58
Nitrogen	1.76	1.73	1.79	1.65
Oxygen (by diff.)	11.81	10.18	11.90	12.27
Sulfur	1.85	1.85	1.71	2.29
<u>Sulfur Forms, Dry (wt %)</u>				
Pyritic	1.17	1.09	2.00	0.96
Sulfate	0.09	0.21	0.10	0.08
Organic (by diff.)	1.64	1.70	1.58	2.04
<u>HHV, Dry (Btu/lb)</u>	12,762	13,347	13,144	12,464
<u>Gieseler Fluidity (DDPM)</u>	1.9	61	6.5	5.8
<u>Free Swelling Index</u>	2½	5	3½	2½

TABLE 2-4
PHYSICAL AND CHEMICAL PROPERTIES OF
ASHES DERIVED FROM ICRC COALS

Sample Identification	FSK-9 APRC-17	FSM-23 APRC-19	FSM-15 APRC-20	Burning Star
Coal Seam	Kentucky No. 9	Kentucky No. 11	Kentucky No. 14	Illinois No. 6
<u>Elemental Composition (wt %)</u>				
Na ₂ O	0.55	0.54	0.36	0.90
K ₂ O	2.10	2.50	1.59	1.93
CaO	6.23	2.13	1.62	7.47
MgO	0.77	0.75	0.46	0.93
Fe ₂ O ₃	17.89	22.93	36.97	14.63
TiO ₂	1.06	1.25	0.95	1.01
P ₂ O ₅	0.07	0.12	0.28	0.06
SiO ₂	44.97	46.24	35.29	46.51
Al ₂ O ₃	16.42	19.62	18.55	20.71
SO ₃	6.56	1.61	0.92	3.47
Cl ⁻	0.03	0.28	0.07	0.07
<u>Fusion Temperatures (°F)</u>				
<u>Reducing Atmosphere</u>				
Initial Deformation	2040	1960	1940	2080
Softening	2120	2060	2000	2100
Hemispheric	2180	2100	2040	2150
Fluid	2420	2360	2220	2230
<u>Oxidizing Atmosphere</u>				
Initial Deformation	2160	2300	2300	2270
Softening	2280	2460	2480	2310
Hemispheric	2340	2480	2500	2350
Fluid	2480	2520	2530	2370

TABLE 2-5
PETROGRAPHIC ANALYSES OF ICRC COALS

Sample Identification	FSK-9 APRC-17	FSM-23 APRC-19	FSM-15 APRC-20	Burning Star
Coal Seam	Kentucky No. 9	Kentucky No. 11	Kentucky No. 14	Illinois No. 6
<u>Reactive Entities (vol %)</u>				
<u>Vitrinoid Types</u>				
4	3.3	--	--	3.9
5	34.3	2.5	40.4	49.0
6	32.8	43.1	37.1	24.9
7	11.5	38.8	4.9	--
Vitrinoids (total)	81.9	84.4	82.4	77.8
1/3 Semifusinoids	1.3	0.5	1.0	1.6
Resinoids	--	--	--	--
Exinoids	3.6	4.0	5.6	3.1
Total Reactives	86.7	88.9	89.0	82.5
<u>Inert Entities (vol %)</u>				
Fusinoids	2.7	1.7	1.2	2.9
2/3 Semifusinoids	2.5	1.1	2.0	3.2
Micrinoids	1.6	3.0	2.3	4.4
Mineral Matter	6.5	5.3	5.5	7.0
Total Inerts	13.3	11.1	11.0	17.5
<u>Mean Reflectance</u>	0.612	0.689	0.602	0.569
<u>Strength Index</u>	2.53	2.57	2.46	2.54
<u>Comp. Balance Index</u>	0.53	0.40	0.43	0.76
<u>Calculated Stability</u>	16	19	16	8

Section 3

CO₂-CARBON CHAR REACTIVITIES

Samples of each of the materials studied in this program were pyrolyzed in the Conoco pressurized coker at 365 psia (roughly 25 atm) in a gas consisting of 31.5 mole % hydrogen and 68.5 mole % nitrogen. All the Kerr-McGee samples and three of the four coals were received as -200 m powders. These were pressed into ~1" dia x 1/2" long briquettes which were charged into the coker in 100 gram batches. Four hundred grams of each material was coked in this manner. The Burning Star, Illinois No. 6, coal which was received in lump form was prepared from a uniformly mixed quarter-drum sample which had been ground to -1/4". Approximately one gallon of the -1/4" coal was riffled, ground to -65 mesh and briquetted as described above.

The yields from the pressurized coking of the ten samples studied in this work are shown in Table 3-1. The yields of coke, gas and liquids are shown both on a dry basis and on a moisture-ash free (MAF) basis. The spread of coke yield values of from ~60 - 70% on an MAF basis for these quite dissimilar samples is relatively small.

After coking, the char was ground to maximize the quantity of 8 x 20 mesh material. All oversize material was reground to assure that 100% of the sample was -8 mesh. Approximately 30 ml of the 8 x 20 mesh char was pretreated in hydrogen at 1750°F (955°C) for 10 minutes to assure complete devolatilization and to remove any remaining organically bound oxygen from the char. Twenty-five ml of the pre-treated material was then loaded into the 0.512" dia. reactor of the Conoco pressurized CO₂-carbon reactivity apparatus which is shown schematically in Figure 3-1.

The reactor containing the samples under a prepurified nitrogen atmosphere was lowered into the furnace which was preheated to 800°C. After the sample temperature, as measured by a thermocouple in the center of the bed, equilibrated at 800°C, the reactor was evacuated and then pressurized to 300 psig with pure CO₂ gas. A flow of CO₂ through the sample bed was maintained at 300 ml/min for two hours with the CO

content of the off gas being constantly monitored with a Perkin-Elmer MGA-1200 gas analyzer and the product gas composition being recorded every two minutes. The flow is shown schematically in Figure 3.2.

After two hours the reactor was again evacuated and heated to 950°C under prepurified nitrogen. When the sample temperature stabilized at 950°C, pure CO₂ was passed through the sample bed at a flow rate of 180 ml/min as prescribed by Gesellschaft-Kohle Technology, GKT, in their test procedure for the evaluation of coals for use in the unpressurized Koppers-Totzek gasifier (6-8). As described for the pressurized case above, the CO content of the exit gas was constantly monitored and recorded at two minute intervals. The 950°C/1 atm run was continued for 50 min. At the end of the run, the reactor was purged with nitrogen and cooled to room temperature. The sample was removed, weighed and quantitatively ashed at 800°C in air in a muffle furnace.

For the 800°C, 300 psig runs the reactivity was reported each two minutes as grams of carbon gasified per gram carbon in the bed per minute. The 950°C atmospheric pressure data are reported as the volume of CO produced per 100 volumes of CO₂ fed and are calculated by the formula:

$$\text{Vol \% CO} = \frac{\text{Vol \% CO} * 100}{\text{Vol \% CO}_2 + 1/2 \text{ Vol \% CO}} \quad (1)$$

From the equation:

it is clear that for total conversion of the CO₂, the vol % CO would be 200. Hence the reactivities reported in this manner range between 0 and 200 vol % CO. This vol % CO reactivity is the unit used by GKT in their evaluation of chars for K-T gasification (6).

According to GKT⁽⁶⁾ materials with reactivities below 50 vol % CO are non reactive, those between 50 and 100 vol % CO are moderately reactive and those greater than 100 vol % CO are highly reactive in K-T gasifiers. A total of five vol % CO reactivities are given for

each material. These, as prescribed by the GKT method (6) are the average vol % CO for five successive 10 minute intervals.

The results of the pressurized 800°C CO₂-carbon reactivity determinations are given in Table 3-2. Both the R values (reactivity in terms of ml CO produced per gram sample per second) and the R' values (the specific carbon gasification rate in units of carbon gasified per g carbon in bed per minute) are the average of the values obtained during each two minute interval between T = 40 minutes and T = 120 minutes. The first 40 minutes of the run are allotted for equilibration and stabilization and are not considered in the reactivity calculation.

Based on these pressurized CO₂-carbon reactivities, the char produced from every material examined in this program is relatively unreactive. These reactivities of from $\sim 7\text{--}12 \times 10^{-4}$ g carbon gasified/g carbon *min are just slightly above those of average Eastern bituminous coal chars and are considerably lower than the values of $20\text{--}50 \times 10^{-4}$ which are typical of more reactive western U.S. coals and lignites. These low char reactivities suggest that none of these materials would be preferred feedstocks to non-slugging type gasifiers. When coupled with the low ash fusion temperatures of these materials (Tables 2-2 and 2-4) these low reactivities virtually preclude the economic gasification of these materials in a standard Lurgi gasifier.

The atmospheric pressure CO₂-carbon, GKT, reactivities are given in Tables 3-3 and 3-4. Three of the four coals (FSK-9, FSM-15 and Burning Star), Table 3-3, would be characterized by GKT (6-8) as highly reactive coals while the fourth coal, FSM-23, would be considered moderately reactive. In general, the reactivity of each coal declines gradually over the fifty minute time period. This decrease in reactivity, when reported on the volume basis used by GKT, is expected because of the decrease with time of the total quantity of carbon due to the ongoing reaction with CO₂. The actual specific reactivity, in terms of grams carbon gasified per gram of available carbon per unit time shows a slight increase in time. This may be due to a slight degree of catalysis from the ash as the ash-to-carbon ratio increases during carbon burn-off.

All six Kerr-McGee ash concentrate samples, Table 3-4, form chars which would be classified by GKT as highly reactive. Here again, as was the case for the coal chars, a general decrease in the reactivity is found as the reaction time is increased. The only exception is the first 10 minute point for the KM-218 which shows considerably less reactivity than the remainder of the points. This suggests the temperature may not have been equilibrated at the start of the run, although the temperatures recorded during this interval do not reflect such a discrepancy. The specific reactivity for these materials, as was found to be the case for the coal chars above, show a slight increase at the higher carbon burn-off levels.

In summary, the reactivities of all materials tested are within the range specified as moderately to highly reactive by GKT (6-8). Thus on the basis of CO_2 -carbon reactivity, these would be expected to have a suitable gasification rate in a Koppers-Totzek gasifier.

TABLE 3-1

YIELDS OF THE PRODUCTS OF PYROLYSIS OF THE ICRC MATERIALS

Identification	Coal	Yields, Dry Basis, Wt %					Yields, MAF, Wt %				
		Liquor*	Gas†	Coke	Tar	Water	Liquor**	Gas†	Coke	Tar	Water
FSK-9 APRC-17	Kentucky No. 9	11.3	22.9	65.8	6.7	4.6	12.6	25.5	61.9	7.5	5.1
FSM-23 APRC-19	Kentucky No. 11	9.0	25.5	65.5	6.2	2.8	9.8	27.7	62.5	6.7	3.1
FSM-15 APRC-20	Kentucky No. 14	14.2	20.8	65.0	7.6	6.6	15.4	22.7	61.9	8.3	7.1
Burning Star	Illinois No. 6	11.3	24.7	64.0	6.5	4.8	12.6	27.8	59.6	7.3	5.3
KM-143	Indiana No. 5	5.1	13.7	81.2	2.9	2.2	9.9	26.3	63.8	5.6	4.3
KM-156	Kentucky Nos. 6 & 11	6.2	11.1	82.7	3.5	2.7	11.0	19.0	70.0	6.1	4.9
KM-162	Kentucky No. 9	3.4	16.2	80.4	2.9	0.5	5.9	27.8	66.3	5.0	0.9
KM-211	Kentucky No. 9	5.6	17.6	76.8	2.6	3.0	7.3	22.6	70.1	3.4	3.9
KM-218	Kentucky No. 9	7.0	14.8	78.2	3.6	3.4	10.9	22.9	66.2	5.6	5.3
KM-236	Illinois No. 6	7.7	18.0	74.3	4.5	3.2	10.3	24.0	65.7	6.1	4.2

Coking Conditions: 365 psia Total Pressure
31.5% Hydrogen, 68.5% Nitrogen

* Tar and Water included in Liquor

† Gas yield obtained by difference

/Ys

TABLE 3-2

REACTIVITY OF ICRC MATERIALS TO CO₂ AT 300 PSIG AND 800°C

<u>Coals</u>	<u>R (ml/CO/g Sample *min)</u>	<u>R' (gC gasified/gC *min) (x 10⁴)</u>
FSK-9	0.043	8.7
FSM-23	0.029	5.7
FSM-15	0.050	9.9
Burning Star	0.037	7.8
<u>Kerr-McGee Residues</u>		
KM-143	0.029	11.9
KM-156	0.020	6.8
KM-162	0.026	9.0
KM-211	0.042	10.6
KM-218	0.037	11.6
KM-236	0.037	9.4

/ls

TABLE 3-3

GKT REACTIVITIES OF ICRC COALS

<u>Coal</u>	<u>Volume % CO for the Time Interval</u>				
	<u>1-10 Min</u>	<u>10-20 Min</u>	<u>20-30 Min</u>	<u>30-40 Min</u>	<u>40-50 Min</u>
FSK-9	114.6	108.9	105.3	104.5	102.2
FSM-23	80.4	73.1	73.1	74.1	73.6
FSM-15	117.0	111.0	108.6	107.6	105.1
Burning Star	123.1	116.5	112.7	105.6	105.2

/ls

TABLE 3-4

GKT REACTIVITIES OF ICRC KERR-McGEE RESIDUES

<u>Material</u>	<u>Volume % CO for the Time Interval</u>				
	<u>1-10 Min</u>	<u>10-20 Min</u>	<u>20-30 Min</u>	<u>30-40 Min</u>	<u>40-50 Min</u>
KM-143	181.5	170.8	147.8	135.1	124.0
KM-156	129.7	114.9	107.8	104.0	100.5
KM-162	171.2	144.6	133.1	128.4	123.0
KM-211	137.9	125.1	117.2	111.5	107.6
KM-218	108.6	181.4	172.4	160.6	162.9
KM-236	150.2	135.9	117.0	107.8	99.8

/ls

Figure 3-1. Furnace and CO₂-Carbon Reactivity Reactor.

Figure 3-2. CO₂-Carbon Reactivity Testing Assembly.

Section 4 SLAG VISCOSITIES

Viscosities were measured on the molten ashes from each of the four coals and six ash concentrates at temperatures between 1200°C and 1550°C. All of the ashes were partially reduced to convert iron oxides to FeO prior to the viscosity measurements. Ferrous iron is the expected valence state in the Texaco or GKT gasifier. Figure 4-1 is a schematic diagram of the Conoco slag viscometer. A complete description of the viscometer and the details of operation are given in an experimental discussion at the end of this section.

The results presented below indicate that none of the ashes from the materials tested would present problems if used in slagging, entrained flow gasifiers. The slags of these ashes exhibit Newtonian behaviour at temperatures above 1300°C, which is well below the expected operating temperatures of most slagging gasifiers. Attempts to correlate slag viscosities with elemental ash compositions were not successful.

A significant finding is that one of the ashes which contain large amounts of sodium loses most of that sodium upon melting, presumably through volatilization. High sodium content in this ash is probably the result of Na_2CO_3 addition to the feed coal at Wilsonville, to control chloride corrosion. The concentration of sodium in the ash could cause significant operability problems in gasification if the volatile sodium is deposited on heat exchange surfaces downstream of the gasifier. Such a consideration might preclude the use of Na_2CO_3 to control corrosion in a grass-roots liquefaction plant.

The results of the slag viscosity measurements are given in Tables 4-1 through 4-15. Tables 4-1 through 4-5 are the slag viscosity run data for the ashes which were derived from the four coals, FSK-9 (Table 4-1), FSM-15 (Table 4-2), FSM-23 (Table 4-3) and Burning Star (Tables 4-4 and 4-5).

The duplicate runs on Burning Star Ash, as well as the duplicate runs on several of the Kerr-McGee ash concentrates (KM-143, 156, and 211) materials were made specifically to determine the reproducibility of the data. The agreement between the Burning Star slag runs is shown on Figure 4-2 which is a plot of $1/T$ (K) vs log viscosity for

the four coal ash slags. The open and filled circles represent the data from the two separate runs on Burning Star ash. The agreement between most points is quite good. Only at lower temperatures ($\leq 1300^{\circ}\text{C}$) do the curves begin to diverge significantly. This is probably due to the non-Newtonian nature of the slag at these high viscosities (> 1000 poise). Here the calculated viscosity depends strongly on the rotational speed of the bob. In general the higher the shear rate (rotational speed) the lower the indicated viscosity. Also at lower temperatures, various components begin to "freeze" out of the melt. Since this solidification would not necessarily be a reproducible phenomenon - due mainly to different cooling rates used - it is reasonable to expect some differences between runs of the same ash. Generally speaking, however, the agreement between duplicate runs as seen in Figures 4-2 and 4-3 is good. Individual viscosities at a given temperature agree in all cases to $\pm 10\%$ and to better than $\pm 5\%$ in most cases.

Of the four coal ashes examined, only that from the FSM-15 tends toward "shortness". "Shortness" is the phenomenon of an abrupt change in the slope of the viscosity vs temperature curve. Typically "shortness" occurs very near the freezing point of the slag and is seen as a much more rapid increase in viscosity with lowered temperature that is found prior to the onset of "shortness". The use of any material with an ash which displays this "shortness" in a process which requires liquid slag removal or slag tapping requires caution. The slag pool must be maintained at a temperature significantly above the temperature of the onset of "shortness" to avoid a sudden catastrophic freezing of the slag pool. This is less critical for ashes which are more "glassy" in nature and display a smooth, single slope viscosity-temperature relationship. Ashes from the remaining three coals, FSK-9, FSM-23 and Burning Star all produce "glassy" type slags and would be expected to be good performers in slagging applications.

None of the Kerr-McGee ash concentrate samples were derived from Wilsonville runs on the FSM-15 (Kentucky 14) coal; hence, it is not possible to determine whether the above-described "shortness" carries over to ash derived from the Kerr-McGee deashing process. There is,

however, no reason to expect the Kerr-McGee process to change the viscosity properties of the coal ash. None of the Kerr-McGee ash concentrate samples, tables 4-6 through 4-15, show a clear tendency towards "shortness" of the slag. A plot of the Kerr-McGee materials slag viscosity data, Figure 4-3, shows only the slag from KM-156 has any sign of a double slope (i.e., onset of "shortness"). Here, however, the differences between the two slopes is not nearly as pronounced as was the case for the FSM-15 coal ash slag found earlier. It should be noted that the slag from every ash studied in this program is well-behaved (i.e., Newtonian) above 1300°C and that here again, as was the case for the coal ash slags, duplicate runs on the same material agree fairly well with one another. Since most slagging gasification processes are designed to run well above this temperature, the slags from all studied materials should, from a viscosity point of view, cause no unexpected operating problems.

The slags from every material studied were examined by scanning electron microscope (SEM) and energy dispersive X-ray analysis (EDX) to determine if segregation of various components occurs while the slag is molten or during freezing. Particular attention was paid to iron because this element is known to segregate in slags which are fully reduced (iron as metallic iron). No such segregation of iron or any other element was found for any of the slags. Figures 4-4 and 4-5 are typical of the SEM micrographs obtained from these materials. Figure 4-4 is from the FSK-9 Kentucky 9 coal and Figure 4-5 is from the KM-236 slag. Both of these materials, as well as all the others, are quite homogeneous in chemical composition. Although there appear to be differences in the surfaces as seen in Figures 4-4 and 4-5, no chemical differences were detected when different areas of the sample were examined by EDX. These optical differences are probably due to fracture planes, reflectance angles or other phenomena associated with breaking the slag sample. Gross examinations of the samples do not reveal any textural inhomogeneities. Thus, it is anticipated that these materials should yield homogeneous slags with little or no iron segregation under partially reducing conditions in the Texaco or K-T gasifier.

The overall elemental compositions of the slags, as determined after each viscosity determination, are given in Tables 4-16 (coal ash slags) and Table 4-17 (Kerr-McGee ash concentrates). These analyses can be compared to those of the raw, unmelted ashes shown in Tables 2-2 and 2-4 to get an idea of the effect of high temperature, and/or melting on the elemental compositions of these materials.

An important chemical difference between the ashes and slags is apparent in the sodium (Na_2O) content of the KM-218 ash. The KM-218 ash contained $\sim 4.5\%$ Na_2O , which decreased to $\sim 0.6\%$ in the respective slag. For the other ashes and slags, sodium contents are the same to within an average deviation of 0.1%. This reduction in sodium content reflects the volatilization of sodium at high temperature. The use of such high sodium materials in high temperature gasification processes would probably result in rather high sodium concentrations in the product gas stream.

This implies that adding sodium carbonate during the liquefaction step to control chloride, as is done in Wilsonville, may lead to problems in the gasification of these residues to produce process hydrogen. Almost certainly the volatilized sodium would be deposited downstream of the gasifier, probably in heat exchangers or shift converters.

Perhaps the most obvious difference between the ashes and the slags is the reduction in sulfate content. The original ashes contain roughly 1 - 6.5% SO_3 . The slags each contain less than 1% SO_3 and most have less than 0.5% SO_3 . This is due to the decomposition of calcium sulfate (CaSO_4) which occurs at the very high temperatures used in the slag viscometer. The majority of the SO_3 in the ashes from the material tested is in the form of CaSO_4 .

Another fairly uniform difference between ash and slag composition is in the alumina (Al_2O_3) content. The Al_2O_3 contents of the slags are uniformly higher than those of the corresponding ash. This is undoubtedly due to the incorporation of some Al_2O_3 from the crucible into

the sample. Part of the increase in Al_2O_3 may be due to dissolution of the .998 alumina crucibles used in the viscometer. This would produce different levels of Al_2O_3 enrichment depending upon both the nature of the slag and the time of exposure at elevated temperatures. Additional Al_2O_3 can be incorporated into the slags by pieces of the crucible physically sticking to the frozen slag when the crucible is broken away from the sample at the end of a run. After completion of a run, the sample and crucible are cooled and then broken with a hammer into pieces small enough to fit into a small grinder. The pieces of broken crucible are separated by hand from the slag and discarded, but occasionally pieces of crucible stick to the slag and end up being crushed with the slag prior to analysis. Although care is taken in this step, some contamination by the crucible is unavoidable.

Several attempts were made to correlate the slag viscosity-temperature data with chemical compositions of the ash. Various functions of the acid-base ratio (9) and silica ratio (10, 11), as well as other elemental ratios, were used in an attempt to obtain a correlation. No correlation was developed using the data obtained in this project. This does not preclude the possibility that a correlation can be derived from a more extensive data set.

Experimental

Approximately 500 g of ash was prepared from each of the six Kerr-McGee ash concentrate samples and four coal samples. After the initial ashing at 1600°F (871°C) in air in a muffle furnace, any final trace of combustible material was removed by oxidation in pure oxygen in a fluidized bed at 1800°F (982°C). The fully oxidized ash was ground to pass a 200 mesh (Tyler) screen and the entire sample was mixed in a rotating jar mixer. The well-mixed ash was fluidized in a 1 1/2" diameter quartz tube reactor by a gas containing 60 mol % CO and 40 mol % CO_2 at 1500°F (816°C). Exposure of the ash to these conditions resulted in the partial reduction of the ash, as determined by the reduction of free iron from ferric (Fe_2O_3) to ferrous (FeO). This is the form of iron which is expected to exist inside a K-T or Texaco gasifier. After reduction, the ashes were stored in sealed glass jars until the

viscosity determinations were made. Just prior to loading the viscometer, the ash was pressed into 1" diameter pellets to facilitate loading into the viscometer crucible.

The slag viscometer apparatus is shown in Figure 4-1. The furnace is a Lindberg, vertical, high temperature tube furnace. Heat is supplied by eight resistance type glo-bar heating elements connected in series. A 2 1/4" ID 998 alumina tube is in the center of the furnace. Inside the center tube is a vertically adjustable 2" OD 998 alumina pedestal which supports two alumina crucibles. The outer crucible (1 7/8" ID x 2 1/8" OD x 8" long) contains a thin layer of aluminum oxide beads in the bottom for levelling the inner sample crucible (1 1/2" ID x 1 3/4" OD x 5" long). The outer crucible also collects any spillage when the sample is charged to the inner crucible. Inside the pedestal is a thermocouple (Platinum vs Platinum - 13% Rhodium) for determining the sample temperature.

Viscosity is a fluid property relating the rate of fluid flow to the fluid stress. It is defined as the ratio of shear stress to shear rate. A Newtonian fluid is defined as a fluid in which this ratio is constant. A non-Newtonian fluid is one in which the viscosity varies with changing shear rates.

The actual viscosity determination is made by spinning a 1/2" diameter bob at a constant shear rate (rpm) in the slag sample and measuring the shear stress (torque) which is directly proportional to the viscosity. This is done on the Conoco instrument through the use of a Haake Rotovisco II measuring head and control unit. this instrument, which has a dual range measuring head has an effective viscosity range (with a 1/2" diameter bob) of $\sim 1-10^5$ poise. This range can, of course, be extended by the use of different bob sizes and geometries.

The bob used in this work is a 1/2" diameter by 1/2" long platinum cylinder which is suspended from the measuring head by a 1/8" diameter by 18" long platinum shaft.

The bob is always located 1/2" from the bottom of the sample crucible during a run. This position is determined by measurements made in a cool furnace. The prepositioning measurement is done to facilitate inserting the assembly at the time of a run.

To ensure that the partially reduced sample remains in the desired chemical state, a nitrogen purge is maintained at the bottom of the furnace. During the entire time the samples are in the furnace, a 60-40 carbon monoxide-carbon dioxide purge is fed to the top of the sample through an alumina tube placed part way down the center tube of the furnace (see Figure 4-1). The bottom of the center tube is sealed tightly with a graphite gasket material (1/4" diameter) through which the pedestal slides up and down.

Prior to the start of a run, the furnace is heated to about 1550°C.

With the CO-CO₂ purge gas flowing, the pelletized sample (150 g) is then charged through a quartz tube into the inner crucible. It is charged in 20 g portions with a short wait between chargings so that any evolved gases can escape without getting trapped. Foaming creates gas pockets which result in inaccurate viscosity readings. After the sample is completely charged, the sample is stirred with a platinum rod to ensure removal of trapped gases and to obtain good sample mixing. After stirring, the bob and shaft assembly is inserted so that the bottom of the bob is 1/2" up from the bottom of the sample as described above. Rotation of the bob and shaft assembly is begun immediately.

After approximately 15 minutes, the first actual viscosity reading is taken. The viscosity is determined at several shear rates (rpm) and recorded. The temperature is then lowered, initially in 25°C increments, and when stabilized after a minimum 15 minute wait, the viscosity is determined. The temperature equilibrates in less than 10 minutes, hence a 15 minute wait guarantees a stable sample temperature. Again, at least four torque readings are taken at various rotational speeds. As the sample nears either its critical viscosity point or solidification temperature, the temperature drop is decreased to 5° or 10°C increments to improve the accuracy of defining these points. The critical viscosity is defined as the temperature where the character of a fluid changes from Newtonian to non-Newtonian. Both the critical and solidification temperatures are sometimes difficult to detect and occasionally the viscosity determination must be repeated, with a fresh sample in order to define them.

Calibration is required whenever the pedestal thermocouple must be replaced, after service on the measuring head, or when a check of the accuracy of the viscometer is desired. To calibrate the thermocouple in the pedestal, an NBS glass standard and two pure salts (potassium sulfate with a melting point of 1069°C and calcium fluoride with a melting point of 1418°C) are used. An empty crucible is half-filled with one of the salts, placed into the furnace, and melted. A thermocouple in a closed alumina sheath is placed directly into the molten salt. The furnace is turned off and the temperature is recorded every ten seconds and plotted to determine the solidification points. The above procedure is repeated with the second salt. The experimental solidification temperatures are plotted against the actual solidification temperatures of the salts. This procedure provides a calibration of the thermocouple which is inserted in the sample. Next the glass standard is melted in the furnace with a thermocouple directly in the sample.

As the temperature is lowered in 25°C increments, similar to a viscosity run determination, the temperatures of both the thermocouple in the sample and the one in the pedestal are recorded. A plot is then made of the actual sample temperature, known by the previous calibration of the thermocouple, versus the observed pedestal thermocouple reading. An equation is derived from this plot to correlate the actual sample temperature with the observed pedestal thermocouple reading. Hence, accurate knowledge of the sample temperature is possible during a run without having a thermocouple in the sample where it would interfere with the viscosity measurement.

To calibrate the bob and shaft assembly, Brookfield standards of known viscosity are used. The equation for determining viscosity is:

$$\eta = \frac{G \cdot S}{N \cdot V} \left(\frac{\text{Shear Stress}}{\text{Shear Rate}} \right) \quad (3)$$

where η is the viscosity in poise, G is an instrument factor which must be derived for each bob and shaft assembly, S is the torque meter reading, N is the rotational speed in rpm, and V is the amplification of the torque reading.

The bob and shaft assembly is suspended in a viscometer crucible filled with the known Brookfield viscosity standard. The torque is recorded at several speeds and the G factor is then calculated. The G factor is determined on two or three different Brookfield viscosity standards and this G factor is then used in each calculation of the viscosity during a slag viscosity determination. The G factor for the 1/2" diameter 1/2" long bob currently in use is 364.0. A change in bob size or geometry, of course, would necessitate recalculation of the G factor.

The bob and shaft assembly must be absolutely straight, centered, and 1/2" from the bottom of the sample crucible to obtain accurate results. Platinum shafts must be straightened after each use due to heat warpage and the natural malleability of platinum. To keep the bob centered, the entire viscometer apparatus must be level. To assure that the bob will be 1/2" from the bottom of the sample, it must be premeasured and the depth settings noted when the furnace is cool.

Control of the atmosphere in the furnace is very critical. A partially reduced sample will be oxidized if air is allowed to enter the system. The bottom of the furnace must be sealed tight. A graphite gasket material permits the pedestal to slide up and down; also a nitrogen purge on the bottom of the furnace aids in eliminating air from the system. An alumina gas tube extends part way down the center tube. During runs under reducing conditions the 60-40 CO-CO₂ mixture is passed through the tube over the top of the sample to ensure maintenance of the desired oxidation state of the sample. The top of the furnace is closed with transite doors which have small holes for the shaft and dip tubes.

At the completion of a run, the bob assembly was removed and the slag sample was allowed to cool. the inner crucible containing the frozen slag was broken away from the sample and discarded. The sample was ground to -100 mesh (Tyler) mixed in a glass jar and an aliquot was analyzed for chemical composition.

TABLE 4-1
SLAG VISCOSITY OF FSK-9 ASH
Kentucky No. 9 Coal

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1498	2728	5.647	20	1.294
1473	2683	5.727	41	1.616
1448	2638	5.811	57	1.757
1423	2593	5.896	75	1.877
1398	2548	5.984	95	1.978
1388	2530	6.020	104	2.016
1378	2512	6.057	118	2.073
1368	2494	6.094	131	2.117
1359	2478	6.127	144	2.159
1349	2460	6.165	167	2.221
1339	2442	6.203	182	2.260
1329	2424	6.242	199	2.300
1319	2406	6.281	232	2.366
1309	2388	6.321	258	2.412
1299	2370	6.361	286	2.456
1289	2352	6.402	331	2.520
1279	2334	6.443	382	2.581
1269	2316	6.495	412	2.615
1259	2398	6.527	490	2.690
1249	2280	6.570	549	2.739
1239	2262	6.614	626	2.797
1229	2244	6.658	724	2.860
1218	2224	6.707	838	2.923
1208	2206	6.752	961	2.983

/ls

TABLE 4-2
SLAG VISCOSITY OF FSM-15 ASH
Kentucky No. 14 Coal

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1438	2620	5.845	14	1.137
1412	2374	5.935	16	1.207
1387	2529	6.024	20	1.297
1362	2484	6.116	24	1.373
1338	2440	6.207	28	1.442
1313	2395	6.305	40	1.603
1288	2360	6.406	66	1.816
1263	2305	6.510	140	2.146
1238	2260	6.618	433	2.636
1214	2217	6.725	1118	3.048

/ls

TABLE 4-3

SLAG VISCOSITY OF FSM-23 ASH
Kentucky No. 11 Coal

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1512	2754	5.602	27	1.435
1486	2707	5.685	34	1.528
1461	2662	5.767	44	1.646
1435	2615	5.855	55	1.744
1410	2570	5.942	70	1.849
1385	2525	6.031	92	1.964
1376	2509	6.064	101	2.008
1366	2491	6.101	114	2.058
1356	2473	6.139	124	2.092
1345	2453	6.180	137	2.135
1336	2437	6.213	157	2.197
1326	2419	6.254	170	2.231
1317	2403	6.289	194	2.289
1306	2383	6.333	214	2.331
1297	2367	6.369	244	2.387
1287	2349	6.410	276	2.440
1277	2331	6.452	316	2.500
1268	2314	6.489	363	2.560

/ls

TABLE 4-4

SLAG VISCOSITY OF BURNING STAR ASH - Run 1

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1530	2786	5.546	41	1.616
1505	2741	5.624	61	1.786
1450	2696	5.705	110	2.041
1455	2651	5.787	149	2.171
1429	2604	5.875	194	2.287
1403	2561	5.959	258	2.412
1380	2516	6.050	352	2.547
1355	2471	6.143	465	2.668
1330	2426	6.238	623	2.794
1305	2381	6.337	861	2.935
1280	2336	6.439	1187	3.074
1255	2291	6.545	1763	3.246
1230	2246	6.653	2558	3.408
1205	2201	6.766	3670	3.565

/ls

TABLE 4-5

SLAG VISCOSITY OF BURNING STAR ASH - Run 2

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1533	2791	5.537	33	1.522
1507	3745	5.618	60	1.791
1483	2701	5.695	91	1.960
1458	2656	5.777	107	2.030
1434	2613	5.858	151	2.178
1409	2568	5.945	232	2.366
1384	2523	6.035	338	2.516
1359	2478	6.127	489	2.689
1334	2133	6.223	692	2.840
1309	2388	6.321	974	2.989
1284	2343	6.423	1514	3.180
1259	2298	6.527	2092	3.321
1235	2255	6.631	3272	3.515
1210	2210	6.743	4639	3.666

/15

TABLE 4-6

SLAG VISCOSITY OF KM-143 ASH - Run 1

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1548	2818	5.491	20	1.297
1520	2768	5.577	23	1.360
1495	2723	5.656	29	1.459
1469	2676	5.741	37	1.569
1444	2631	5.824	46	1.658
1418	2584	5.914	56	1.748
1392	2538	6.006	72	1.858
1366	2491	6.101	94	1.975
1358	2476	6.131	104	2.016
1348	2458	6.169	114	2.057
1338	2440	6.207	128	2.106
1328	2422	6.246	142	2.153
1318	2404	6.285	158	2.199
1308	2386	6.325	171	2.232
1298	2368	6.365	192	2.284
1288	2350	6.406	221	2.345
1277	2331	6.452	249	2.396
1267	2313	6.494	276	2.441
1257	2295	6.536	333	2.522
1247	2277	6.579	363	2.560
1238	2260	6.618	409	2.611

/ls

TABLE 4-7

SLAG VISCOSITY OF KM-143 ASH - Run 2

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1525	2777	5.562	16	1.196
1499	2730	5.643	22	1.346
1474	2685	5.724	37	1.563
1449	2640	5.807	46	1.663
1425	2597	5.889	57	1.754
1399	2550	5.981	77	1.885
1390	2534	6.013	84	1.923
1380	2516	6.050	90	1.952
1370	2498	6.086	98	1.989
1360	2480	6.124	110	2.040
1350	2462	6.161	121	2.081
1341	2446	6.196	135	2.130
1331	2428	6.234	151	2.180
1321	2410	6.274	172	2.237
1311	2392	6.313	199	2.298
1301	2374	6.353	220	2.343
1292	2358	6.390	238	2.377
1281	2338	6.435	266	2.425
1272	2322	6.472	293	2.467

/ls

TABLE 4-8

SLAG VISCOSITY OF KM-156 ASH - Run 1

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1539	2802	5.519	39	1.595
1513	2755	5.599	49	1.688
1487	2709	5.682	58	1.766
1460	2660	5.770	72	1.854
1436	2617	5.851	96	1.980
1427	2601	5.882	104	2.017
1417	2583	5.917	115	2.060
1407	2565	5.952	125	2.095
1397	2547	5.988	139	2.143
1387	2529	6.024	156	2.193
1378	2512	6.057	171	2.233
1367	2493	6.098	189	2.277
1357	2475	6.135	216	2.334
1347	2457	6.173	234	2.369
1337	2439	6.211	269	2.430
1327	2421	6.250	304	2.483

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TABLE 4-9

SLAG VISCOSITY OF KM-156 ASH - Run 2

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁻⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1542	2808	5.510	60	1.777
1519	2766	5.580	63	1.798
1493	2719	5.663	73	1.862
1468	2674	5.744	85	1.931
1442	2628	5.831	104	2.015
1421	2590	5.903	117	2.067
1396	2545	5.992	138	2.139
1369	2496	6.090	170	2.231
1346	2455	6.177	222	2.347
1336	2437	6.215	239	2.378
1325	2417	6.258	268	2.429
1317	2403	6.289	316	2.500
1307	2385	6.329	366	2.564

/ls

TABLE 4-10

SLAG VISCOSITY OF KM-156 ASH - Run 3

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1453	2647	5.794	125	2.095
1428	2602	5.879	139	2.142
1403	2557	5.967	161	2.208
1381	2518	6.046	190	2.280
1355	2471	6.143	230	2.362
1329	2424	6.242	332	2.521
1319	2406	6.281	372	2.570
1310	2390	6.317	407	2.610
1300	2372	6.357	442	2.645
1291	2356	6.394	484	2.685
1281	2338	6.435	585	2.767
1270	2318	6.481	709	2.851
1260	2300	6.523	938	2.972
1251	2284	6.562	1274	3.105

/1s

TABLE 4-11

SLAG VISCOSITY OF KM-162 ASH

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1535	2795	5.531	121	2.082
1524	2775	5.565	133	2.124
1514	2757	5.596	142	2.154
1503	2737	5.631	155	2.191
1494	2721	5.659	167	2.221
1484	2703	5.692	179	2.252
1474	2685	5.724	198	2.298
1463	2665	5.760	220	2.343
1453	2647	5.794	246	2.390
1443	2629	5.828	277	2.443
1433	2611	5.862	315	2.498
1423	2593	5.896	350	2.544
1412	2574	5.935	385	2.585

/ls

TABLE 4-12

SLAG VISCOSITY OF KM-211 ASH - Run 1

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1530	2786	5.546	39	1.585
1504	2739	5.627	43	1.629
1479	2694	5.708	53	1.723
1454	2649	5.790	63	1.796
1429	2604	5.875	80	1.903
1404	2559	5.963	101	2.005
1378	2512	6.057	126	2.100
1353	2467	6.150	164	2.215
1328	2422	6.246	213	2.328
1303	2377	6.345	284	2.454
1278	2332	6.447	392	2.593
1254	2289	6.549	546	2.738
1229	2244	6.658	744	2.872
1204	2199	6.770	1065	3.027

/ls

TABLE 4-13

SLAG VISCOSITY OF KM-211 ASH - Run 2

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1499	2730	5.643	39	1.587
1470	2678	5.737	49	1.687
1444	2631	5.824	60	1.780
1418	2584	5.914	75	1.877
1404	2559	5.963	87	1.937
1402	2556	5.970	86	1.934
1391	2536	6.010	94	1.973
1380	2516	6.050	106	2.025
1369	2496	6.090	118	2.070
1359	2478	6.127	132	2.119
1349	2460	6.165	148	2.171
1339	2442	6.203	163	2.213
1329	2424	6.242	180	2.255
1319	2406	6.281	203	2.308
1308	2386	6.325	226	2.354
1298	2368	6.365	256	2.409
1288	2350	6.406	291	2.464
1278	2332	6.447	335	2.525
1267	2313	6.494	371	2.569
1257	2295	6.536	417	2.620
1246	2275	6.583	480	2.682
1236	2257	6.627	541	2.733
1226	2239	6.671	635	2.803
1216	2221	6.716	723	2.859
1206	2203	6.761	874	2.942

/ls

TABLE 4-14
SLAG VISCOSITY OF KM-218 ASH

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1524	2775	5.565	167	2.224
1498	2728	5.647	206	2.314
1472	2682	5.731	262	2.418
1448	2638	5.811	344	2.537
1423	2593	5.896	460	2.662
1398	2548	5.984	600	2.778
1373	2503	6.075	808	2.907
1348	2458	6.169	1083	3.034
1323	2413	6.266	1577	3.198
1299	2370	6.361	2222	3.347
1274	2325	6.464	3187	3.500
1249	2280	6.570	4377	3.641
1224	2235	6.680	6279	3.798

/1s

TABLE 4-15
SLAG VISCOSITY OF KM-236 ASH

<u>Temp</u> <u>(°C)</u>	<u>Temp</u> <u>(°F)</u>	<u>1/T (K)</u> <u>x 10⁴</u>	<u>Viscosity</u> <u>(Poise)</u>	<u>Log</u> <u>Viscosity</u>
1535	2795	5.531	74	1.872
1510	2750	5.609	82	1.912
1485	2705	5.688	97	1.987
1461	2662	5.767	123	2.089
1436	2617	5.851	153	2.186
1411	2572	5.938	199	2.298
1386	2527	6.028	261	2.417
1361	2482	6.120	356	2.551
1337	2439	6.211	468	2.670
1312	2394	6.309	621	2.793
1287	2349	6.410	909	2.958
1262	2304	6.515	1245	3.095

/ls

TABLE 4-16

CHEMICAL COMPOSITION AND ASH FUSION TEMPERATURES
OF COAL ASH SLAGS

<u>Elemental Composition</u>	<u>Coal</u>			
	<u>FSK-9</u>	<u>FSM-23</u>	<u>FSM-15</u>	<u>Burning Star</u>
Na ₂ O	0.74	0.67	0.56	0.87
K ₂ O	2.34	2.35	1.78	1.97
CaO	6.50	2.68	1.58	7.67
MgO	0.96	0.81	0.62	1.03
Fe ₂ O ₃	17.96	19.83	32.42	14.50
TiO ₂	1.14	1.23	0.94	0.95
P ₂ O ₅	0.07	0.14	0.30	0.10
SiO ₂	44.65	44.37	34.13	45.85
Al ₂ O ₃	23.95	25.38	23.75	22.65
SO ₃	0.63	0.09	0.18	0.52

Ash Fusion Temperatures (°F)

Reducing Atmosphere

Initial Deformation	2100	2090	1920	2060
Softening	2230	2230	2100	2140
Hemispheric	2280	2290	2260	2180
Fluid	2320	2370	2360	2300

Oxidizing Atmosphere

Initial Deformation	2290	2370	2400	2240
Softening	2340	2430	2460	2300
Hemispheric	2370	2460	2480	2340
Fluid	2430	2520	2530	2440

/ls

TABLE 4-17
CHEMICAL COMPOSITION AND FUSION TEMPERATURES
OF KERR-McGEE ASH CONCENTRATE SLAGS

Elemental Composition	Kerr-McGee Ash Concentrate					
	KM-143	KM-156	KM-162	KM-211	KM-218	KM-236
Na ₂ O	0.52	0.53	0.57	0.75	0.59	1.10
K ₂ O	2.06	2.66	2.92	2.08	4.03	1.95
CaO	4.32	1.36	1.95	2.60	1.75	6.98
MgO	0.90	0.75	1.14	0.71	1.18	0.97
Fe ₂ O ₃	21.32	22.54	16.67	24.05	16.99	16.13
TiO ₂	0.80	0.99	1.01	0.91	0.89	1.01
P ₂ O ₅	0.19	0.22	0.20	0.10	0.22	0.08
SiO ₂	46.15	49.12	48.49	43.12	43.49	44.16
Al ₂ O ₃	24.05	21.08	22.41	21.50	21.12	22.33
SO ₃	0.33	0.03	0.03	0.35	0.13	0.63
Ash Fusion Temperatures (°F)						
Reducing Atmosphere						
Initial Deformation	1970	1940	2000	1880	2000	2000
Softening	2060	2170	2040	2100	2100	2060
Hemispheric	2090	2260	2130	2220	2260	2120
Fluid	2220	2280	2310	2280	2440	2220
Oxidizing Atmosphere						
Initial Deformation	2380	2380	2400	2360	2320	2280
Softening	2420	2460	2440	2430	2380	2340
Hemispheric	2440	2500	2470	2460	2440	2420
Fluid	2500	2620	2540	2500	2540	2500

/ls

Figure 4-1. Slag Viscometer Apparatus.

Figure 4-2. Slag Viscosity-Temperature Relationship of Coal Ashes.

Figure 4-3. Slag Viscosity-Temperature Relationships of Kerr-McGee Ash Concentrate.

200X

1000X

Figure 4-4
Typical Coal Ash Slag
FSK-9 - Kentucky No. 9 Coal

200X

1000X

Figure 4-5
Typical Kerr-McGee Ash Slag
KM-236

Section 5

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