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Co-Firing High Sulfur Coal with Refuse Derived Fuels

Technical Progress Report #3

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1. Summary

The TG-FTIR-MS system was used to identify molecular chlorine, along with HCl, CO, CO₂, H₂O, and various hydrocarbons in the gaseous products of the combustion of PVC resin in air. This is a significant finding that will lead us to examine this combustion step further to look for the formation of chlorinated organic compounds. The combination of TG-FTIR and TG-MS offers complementary techniques for the detection and identification of combustion products from coals, PVC, cellulose, shredded newspaper, and various blends of these materials.

The pilot atmospheric fluidized bed combustor (AFBC) at Western Kentucky University has been tested. The main purpose of these preliminary AFBC runs were to determine the compatibility of coal and pelletized wood in blends and to explore the effects of flue/air ratio. Our objective is to conduct AFBC burns with 90 percent sulfur capture and more than 96% combustion efficiency.

2. Studies with TGA / FTIR / MS

The raw materials used in this experiment include two bituminous coals, a medium molecular weight PVC resin from the OxyChem Corporation, cellulose from the Whatman Co., and shredded newspaper from Western Kentucky University. In the previous progress report¹ we described the characteristics and behavior of these five materials. We now report on the behavior of various blends of these materials.

The blends were prepared and labelled as follows:

03PVC50 is a mixture of coal 90003 and PVC (50% each)

03NEW50 is a mixture of coal 90003 and newspaper (50% each)

03CEL50 is a mixture of coal 90003 and cellulose (50% each)

03P2N25 is a mixture of coal 90003(50%), PVC (25%) and newspaper (25%)

035122 is a mixture of coal90003 (50%), PVC (10%), newspaper (20%) and cellulose (20%)

Samples 73PVC50, 73NEW50, 73CEL50, 73P2N25 and 735122 are the corresponding blends using coal 92073 in place of coal 90003.

Figure 1 is a schematic of the TGA/FTIR/MS system for this study. A Du Pont 951 Thermogravimetric Analyzer (TG) interfaced to a Perkin Elmer 1650 Fourier Transform Infrared Spectrophotometer (FTIR) was used in this study. The horizontal quartz furnace of the TG was connected to the 10 cm gas cell of the FTIR using an insulated teflon tube heated to a temperature of 150°C with resistance tape controlled by a Powerstat variable auto transformer. The TG was also interfaced to a Fisons Instruments Thermolab Mass

Spectrometer (MS) using a fused silica capillary sampling inlet which is heated to approximately 170°C, Figure 2. A teflon splitter divides the flue gas from TGA into two parts, one to the FTIR (~95%), the other to the MS (~5%).

In the experiments all samples (~100 mg each) were heated in air (50 ml/min) at a rate of 10°C/min to 700°C. The spectra and profiles of gaseous species evolving from the TG system were recorded and analyzed by the TGA-FTIR-MS analytical system.

2.1 Results with TG/DTG

Table 1 is a summary of TG/DTG results for the individual raw materials and for mixtures of the individual components. Compare sample 03PVC50 (50%PVC and 50% coal) with coal 90003 and PVC. It is obvious that the first weight loss is mainly due to the evolution of HCl in PVC. The second weight loss comes from the co-decomposition of coal and PVC.

In sample 03NEW50 (50% newspaper and 50% coal), the first weight loss corresponds to moisture in newspaper, the second weight loss is due to the decomposition of newspaper alone, and the third decomposition stage is due to a second weight loss in coal 90003. It is notable that the weight loss at T_{max} 690°C in newspaper was not observed in the newspaper-coal blends, 03NEW50 and 73NEW50. This may be due to some reactions between coal and the residue of newspaper to form volatile products at a lower temperature. This pattern also occurs with blends using other newsprint stock.

Table 1

Summary of TG/DTG Results

[at 10°C/min to 700°C in air, 50 mL/min flow]

sample	$\Delta W_1(\%)$	$T_{\max}$	$R_{\max}$	$\Delta W_2(\%)$	$T_{\max}$	$R_{\max}$	$\Delta W_3(\%)$	$T_{\max}$	$R_{\max}$
PVC	63.64	317	1.5	10.48	477	0.2	25.88	561	0.4
90003	1.9	80	0.1	89.35	454	1.2			
92073	6.1	80	0.1	69.12	458	0.5			
NEWS ^A	4.57	70	0.2	86.82	346	2.2	2.035	690	0.1
CELL ^A	3.43	60	0.1	96.00	345	5.2			
03PVC50				30.1	307	0.6	64.3	457	0.6
03NEW50	3.23	70	0.1	29.9	340	1.9	59.17	463	0.77
03CEL50	3.8	60	0.1	43.15	341	1.7	48.3	442	1.8
03P2N25	3.2	60	0.1	26.67	302	0.7	64.06	419	1.3
035122	41.21	304	1.0	22.21	420	2.4	19.6	463	1.0
73PVC50				37.7	306	0.7	51.03	437	0.5
73NEW50	4.29	70	0.1	51.02	337	3.3	23.81	444	1.4
73CEL50	4.29	70	0.1	53.41	346	2.3	29.63	423	1.9
73P2N25	30.48	297	1.1	25.81	336	3.1	28.3	432	1.2
735122				66.7	327	5.2	23.56	418	2.1

notes: ΔW is weight loss, in % of original weight

$R_{\max}$ is a maximum rate of weight loss, in weight %/°C

$T_{\max}$ is the temperature at $R_{\max}$ in °C

^A NEWS is newspaper fiber; CEL is cellulose fiber

2.2 Results with FTIR

2.2.1 Gases Released from Individual Raw Materials

Figures 3-7 show the three dimensional FTIR spectra of the compounds obtained on the combustion of the individual raw materials. The relative axes are absorbance (vertical), wave number (horizontal) and time-temperature (perspective), respectively. The time scale corresponds to the decomposition temperature. The spectra obtained correspond to the TGA thermograms starting at 100°C and with a scan frequency of one per minute.

In coal 90003 and 92073 spectra, a similar trend for decomposition is observed for the CO₂, CO and CH₄ peaks. The peak at 712 cm⁻¹ can be attributed in part to the absorbance of HCN from the decomposition of pyridine structures in coal at 400-670°C. This conclusion can be supported by the double peaks at 3330 and 3280 cm⁻¹ which appear in the same temperature range. However, there are considerable differences between the two spectra. There are more water peaks in coal 90003 than in coal 92073 in the region from 1300 to 1700 cm⁻¹ and 3500 to 4000 cm⁻¹. This can be attributed to more volatile matter and higher hydrogen content in 90003. The COS (2073 cm⁻¹) and SO₂ (1374 cm⁻¹) peaks are stronger for coal 92073 than 90003, reflecting the higher sulfur content. The SO₂ peaks of 92073 provide some evidence about the sulfur forms present. The aliphatic sulfur decomposes first at 300-400°C; then pyrite and thiopene rings decompose at 400-600°C; and finally sulfate salts are predominant at 700-800°C. In coal 92073 spectra, the peaks at 1771 and 1171 cm⁻¹ can be attributed to acetic acid by

comparing their shapes and wave number with standard spectra. The HCl peaks at about 2800 cm^{-1} cannot be identified clearly, even though there is some chlorine content (289 ppm) in coal 92073, owing to masking by the stronger absorption peaks from 3300 to 2500 cm^{-1} .

Another way to present results from the FTIR spectra is to construct evolved gas profiles as shown in Figures 8-10 for coal 90003, PVC, and newspaper, respectively. CO_2 is much more abundant than the other gases released during combustion, and as a consequence must be plotted on a scale different from the other gases. In the PVC evolved gas profile, HCl is released first at $230\text{--}400^\circ\text{C}$, with some CO_2 from the low molecular weight PVC groups. This is in accordance with the TG/DTG results. In the three dimensional graph of PVC (Figure 5), a notable phenomenon is observed at 674 cm^{-1} . The profile at this wave number has two maxima. It is often attributed to absorbance by carbon dioxide. The first maximum, however, is not due to absorbance by CO_2 (no corresponding absorbances at 2356 and $3600\text{--}3700\text{ cm}^{-1}$). From the peaks at 1500 and 3050 cm^{-1} , the first maximum would appear to be due to absorbance by benzene. CH_4 peaks are obvious, while they are not observed in the spectra of newspaper and cellulose. In the spectra for newspaper and cellulose, a number of peaks between $250\text{--}450^\circ\text{C}$ are observed that correspond to the release of water, CO_2 and CO. This reflects the larger oxygen content and OH functionalities in these materials. Also, compared to PVC, many more organic acids (mainly formic and acetic acids) are produced during the combustion of newspaper and cellulose. These can be identified in the three dimensional spectra by groups of peaks at $2500\text{--}3400$ (OH), $1700\text{--}1800$ (C=O), 1033 for methanol, 1106 for formic acid and

1175 cm^{-1} for acetic acid. The appearance of these materials can be attributed to the polyhydroxy structures in newspaper and cellulose.

The evolved gas profiles for PVC are quite characteristic. HCl gas is released first and the absorbance reaches its maximum at approximately 320°C. However, combustion of PVC occurs at 570°C, which is shown by the profiles of carbon dioxide and carbon monoxide. This corresponds to the third weight loss in its TG-DTG curve. These results imply that the PVC is not readily flammable due to its chlorine content. In the profiles of newspaper and cellulose, the absorbance curves of carbonyl (C=O) and C-O have the same shape and reach their maxima at about 370°C. This suggests that these two peaks belong to the same compound, for example, formic acid.

Figure 11 is a comparison of methane profiles for the two coals and PVC. It shows that methane forms at about 500°C. As previously mentioned, because of the high oxygen content in newspaper and cellulose, no methane is formed during the combustion of these materials. It is notable (Figure 11) that there is a little methane released from coals between 230°C to 380°C, but not from PVC. The early methane is thought to be that already present in the micropores of coals. The methane peak at around 500°C is produced by the pyrolytic decomposition of the coal matrix and, to a lesser extent, the PVC carbon chain.

The identified characteristic peaks (not including water peaks which appear in every sample) of FTIR spectra for five raw materials are listed in Table 2.

Table 2

Tentative Identification of FTIR peaks from unblended material^A

[TGA offgas from runs at 10°/min to 700°C in air at 50 mL/min]

<u>Compound</u>	<u>Coal</u> <u>90003</u>	<u>Coal</u> <u>92073</u>	<u>PVC</u>	<u>Newspaper</u>	<u>cellulose</u>
CH ₄	3016	3016	3016		
HCl	2798	2798	2798		
CO ₂	2356	2356	2356	2356	2356
CO			1798	1790	1790
			1788	1773	1777
			1776	1747	1746
			1734	1734	1734
			1717	1717	1724
SO ₂	1374	1374			
Acetic acid			1175	1175	1175
Formic acid			1106	1107	1107
Methanol			1036	1033	
Ethylene	950	950	950	950	950
1,3-Butadiene	910		910		
Furan				745	744
Chlorobenzene(?)	740	741	741		
HCN	712	712			
Benzene			674		

^A numbers in this and following tables are absorbance maxima in wavenumbers (cm⁻¹)

2.2.2 Gases Released from Coal 90003 and 92073 Mixtures

Figures 12 and 13 are 3-D graphs for mixtures of coal 90003(50%) with newspaper (50%) and cellulose(50%). They are similar to one another. A substantial amount of water ($3300-4000$ and $1300-1600\text{ cm}^{-1}$) and organic acids ($2800-3000$, $1700-1900$ and $1000-1200\text{ cm}^{-1}$) are released with small amounts of carbon dioxide and carbon monoxide at approximately 320°C ; these reach their maxima at around 340°C . From TGA data, we know that this decomposition stage can be attributed to the decomposition of newspaper and cellulose. Methane ($2900-3200\text{ cm}^{-1}$) and sulfur dioxide (1374 and 1350 cm^{-1}) begin to appear at about 400°C and reach their maxima at 460°C . Emissions at this stage are mostly due to decomposition of coal. Compared to the spectra of newspaper and cellulose individually, the tails of carbon dioxide peaks in their mixtures are obviously lower. This can be related to lower residues in TGA curves of mixtures after 500°C .

Figure 14 is a 3-D graph of 03PVC50. It is almost a combination of those of coal 90003 and PVC. Similar combination graphs are obtained for 03P25N25, 035122 and all coal 92073 mixtures.

The identified characteristic peaks (not including water peaks which appear in every sample) of FTIR spectra for coal 90003 and 92073 mixtures are listed in Tables 3 and 4. Figures 15 - 22 are the spectra of the identified peaks of 735122 and 035122 mixtures.

Table 3

Tentative Identification of FTIR Peaks from Blends of Coal 92073

<u>Compound</u>	<u>73PVC50</u>	<u>73CEL50</u>	<u>73NEW50</u>	<u>73P2N2</u>	<u>735122</u>
CH ₄	3018	3018	3018	3018	3018
HCl	2798	2798	2798	2798	2798
CO ₂	2360	2360	2360	2360	2360
CO	2175	2175	2175	2175	2175
COS	2074	2074	2074	2074	2074
C=O	1700-	1700-	1700-	1700-	1700-
	1800	1800	1800	1800	1800
SO ₂	1374	1374	1374	1374	1374
Ethyl acetate (?)					1245
Acetic acid	1176	1176	1176	1176	1176
Formic acid	1107	1107	1107	1107	1107
Methanol			1036		
Ethylene	950	950			
1,3-Butadiene	910	906		906	906
p-Xylene	793				
CH ₂ Cl ₂ (?)	756	756	756	756	756
Furan		745	745	745	745
Chlorobenzene (?)	741	741	741	741	741
HCN	712	712	712	712	712

Table 4

Tentative Identification of FTIR Peaks from Blends of Coal 90003

<u>Compound</u>	<u>03PVC50</u>	<u>03CEL50</u>	<u>03NEW50</u>	<u>03PO2N2</u>	<u>035122</u>
CH ₄	3018	3018	3018	3018	3018
HCl	2798	2798	2798	2798	2798
CO ₂	2360	2360	2360	2360	2360
CO	2175	2175	2175	2175	2175
COS	2074	2074	2074	2074	2074
C=O	1700-	1700-	1700-	1700-	1700-
	1800	1800	1800	1800	1800
SO ₂	1374	1374	1374	1374	1374
Acetic acid	1176	1176	1176	1176	1176
Formic acid		1107	1107	1107	1107
Methanol		1033	1036	1033	1033
Ethylene	950			950	950
1,3-Buradiene	910				
CH ₂ Cl ₂ (?)	756	756	756	756	756
Furan		745	745	745	745
Chlorobenzene (?)	741	741	741	741	741
HCN	712	712	712	712	712

2.3 Results with TGA/MS

The Thermolab Gas Analyzer is a low-range, low-sensitivity quadrupole mass spectrometer. As it analyzes mixtures of gaseous molecules which are not subjected to separation techniques, the data obtained requires great care in interpretation. Nevertheless, some useful information can be obtained, especially in conjunction with other sources of structural information, such as FTIR spectra. The MS spectra were collected as the sample in the TGA furnace was being heated.

Figure 23 is a mass spectrum at scan 26 of the offgas of coal 92073, and Figure 24 is the profile of some of the peaks of coal 92073. From Figure 23, the profiles of peaks M/Z 18, 32, 44, 60 and 64 can be attributed to the H_2O , O_2 , CO , COS and SO_2 , respectively. The water profile shows three peaks. The first peak, at around 100°C , is the free moisture in the coal. The second and third peaks are produced due to the combustion and decomposition of coal. The oxygen profile remains stable up to about 350°C , after which it decreases due to its consumption by combustion of the coal matrix. The inverse peak indicates a maximum consumption of oxygen. In addition, sulfur dioxide shows three peaks. This result is the same as that obtained in the FTIR spectra. In 92073, carbonyl sulfide is very obvious and SO_2 shows three decomposition zones.

Figures 25 and 26 show some mass peak profiles for PVC. The M/Z 36 and 38 are formed at the same time over a range of 200 to 450°C and indicate the isotopes H^{35}Cl and H^{37}Cl . Also, the M/Z 70, 72 and 74 peaks suggest the isotopes $^{35}\text{Cl}_2$, $^{35}\text{Cl}^{37}\text{Cl}$ and $^{37}\text{Cl}_2$. These assignments are supported by the ratios of their intensities. The ratio of M/Z 38 to M/Z 36 is 0.333, which is close to the chlorine isotope fraction 0.325. Furthermore, M/Z

M/Z 70 to 72 to 74 appear at exactly the same point, with mass ratio 1.00 : 0.663 : 0.26, as compared with the predicted mass ratio of 1.00 : 0.65 : 0.11. This confirms earlier indications of the formation of Cl_2 via the Deacon reaction.¹ The major aromatic volatile components, benzene (M/Z 78), toluene (92), styrene (104) and indene (116) and their derivatives evidently form at the second decomposition step at 420-460°C.²

Figures 27-36 show mass spectra of the mixtures at specific scans. It is difficult to clearly decipher these complicated spectra. However, we can use FTIR data as a second set of clues and concentrate our attention on the sulfur and chlorine species in the evolved gases.

Figures 37-42 are profiles for the peaks, 36, 38, 70, 72 and 74, for six different PVC-containing blends. All of these show similar trends as that observed in PVC, that is, mass spectrometric evidence that Cl_2 is produced concurrently with the strong HCl release.

Figures 43 and 44 are profiles of peak 112 in spectra 735122 and 035122. The temperature at which it is evolved ($\sim 320^\circ\text{C}$) is the same as that of the peak at M/E 36. This is suggestive of a relationship with the chlorine species, for example, chlorobenzene. In the FTIR spectra, the observed peak at 741 cm^{-1} , matching that of chlorobenzene, appears in the spectra of all of the blends except those of newspaper and cellulose.

Figures 45 and 46 are profiles of the M/Z 60 signal (attributed to both COS and acetic acid), and 64(SO_2) in spectra 035122 and 735122. The peaks are much stronger in 735122 than in 035122. This is due to the higher sulfur content in coal 92073. Also there are three peaks in SO_2 profiles. This is consistent with the profile of coal 92073 and

the FTIR results. For M/Z 60, however, it shows only two peaks in the profile. This may be because after the carbon species is burnt completely, there is enough oxygen to convert the sulfur species into SO_2 and hence the partially oxygenated product COS is not observed.

3. Fluidized Bed Combustion

The pilot atmospheric fluidized bed combustor (AFBC) at Western Kentucky University was built by the University with support from the U.S. Department of Energy. The active bed area is 125 in². The freeboard zone of the combustor is 10 feet high, providing adequate residence time for the combustion of fine fuel particles which may be entrained in the gases leaving the bed. This also allows time for the entrained fine particles of bed material and ash to lose their upward velocity and fall back into the bed, thus minimizing dust loading in the cyclone and providing stable bed height. The fuel is injected into the fluidized bed by using pneumatic injectors. The injectors used for these tests are located about 9" above the air distributor of the combustor. The bed temperature is controlled by fuel feed rate adjustment.

The main purposes of these preliminary AFBC runs are to determine the compatibility of coal and pelletized wood in blends, and to explore the effects of fuel/air ratio. Our objective is to conduct AFBC burns with 90 percent sulfur capture and more than 96% combustion efficiency.

3.1 Burn of April 25, 1995

The first of two tests during this reporting period was made on April 25, 1995, using WKU coal NO.95010, and wood pellets offered by the Jim C. Hamer Co. (Bowling Green, KY).

Characteristics of the coal used are given in Table 5, and those of the wood used are given in Table 6. The combustor operating conditions are listed in Table 7. The fuel

Table 5

Analysis of Coal #95010^A

Moisture (wt %)	2.32%
Ash	7.05
Carbon	77.54
Hydrogen	5.45
Nitrogen	1.59
Sulfur	0.65
Oxygen	7.63
HHV (Btu/lb)	13,750

^A as determined

Table 6

Analysis of Wood Used

Moisture (wt %)	4.71%
Ash	1.09
Carbon	47.56
Hydrogen	6.37
Nitrogen	0.07
Sulfur	0.11
Oxygen	44.73
HHV (Btu/lb)	7,950

Table 7

Combustion Test Matrix

Bed area	125 in ²
Coal/wood feed rate	21-34 lbs/hr
Coal/wood feed size	0.2 x 0 in
Bed temperature	1,500°F [815°C]
Fluidized velocity	3.69 fps
Bed material/sorbent	0.16 x 0 in. limestone

Table 8

Feed Schedule and CO₂ Concentration

<u>Time</u>	<u>Feed Ratio Coal/Wood</u>	<u>CO₂ Concentration (by FTIR)</u>
0900-1325	100% coal	9.9%
1325-1400	79%/21%	10.1%
1400-1440	56%/44%	9.9%
1440-1522	34%/66%	11.1%
1522-1531	14%/86%	11.7%

composition schedule and offgas CO₂ concentration are given in Table 8. Evolved gas was analyzed by gas chromatography (GC) and Fourier Transform infrared spectrometry (FTIR). In addition, gas samples for ion chromatography (IC) and fly ash samples were collected.

During stable combustion, CO and CH₄ disappear, as has also observed from GC analysis. During combustion, we changed the ratio of coal/wood, the results for which are shown in Table 8.

IC was used to measure chloride and sulfate. Gas samples (100 mL) were collected every 15 minutes. IC results are shown in Table 9. Sulfur ranges from 0.73 to 0.95 lbs/MMBtu in coal and wood samples, and the IC data shows that sulfate was not detected, in agreement with FTIR data. Thus limestone appears to be an effective sorbent, absorbing almost all of the sulfur dioxide evolved from the coal and coal/wood combustion. Chlorine is in the range of 1,000 ppm [0.74 lbs/MMBtu] in coal and wood samples, and it is found in the range 0.1-0.5 ppm in IC samples.

The SO₂ lbs/Btu value is calculated as follows.

1. The sulfur content of the coal is 0.65%, and of the wood is 0.11%. If the ratio of coal/wood is 79%/21%, then sulfur in 1 lb of blended fuel is $0.65\% \times 79\% + 0.11\% \times 21\%$, or 0.54%. So 1 lb of blended fuel contains 0.0054 lb. S which can produce 2×0.0054 lbs of SO₂.
2. Similarly, the HHV in 1 lb of 79/21 blended fuel is $13,750 \times 79\% + 7,950 \times 21\%$, or 12,532 Btu/lb.
3. Therefore the SO₂ lb/Btu value $= 0.0054 \text{ lb} \times 2 / 12,532 = 0.86 \text{ lb} / 10^6 \text{ Btu}$.

Table 9

IC Results

<u>Time</u>	<u>Chlorine (ppm)</u> <u>Coal & Wood</u>	<u>IC</u>	<u>SO₂ (lbs/MBtu)</u> <u>Coal & Wood</u>	<u>IC</u>	<u>Experimental Condition</u> <u>ratio of coal/wood</u>
9:15-10:15	1015	0.0	0.95	0.0	100% coal
10:15-10:30	1015	0.155	0.95	0.0	100% coal
10:30-10:45	1015	0.153	0.95	0.0	100% coal
10:45-11:00	1015	0.0	0.95	0.0	100% coal
11:15-11:30	1015	0.248	0.95	0.0	100% coal
11:45-12:00	1015	0.456	0.95	0.0	100% coal
12:30-12:45	1015	0.199	0.95	0.0	100% coal
3:00-13:15	1015	0.172	0.95	0.0	100% coal
13:15-13:30	1015	0.195	0.95	0.0	100% coal
13:30-13:45	951.6	0.195	0.86	0.0	79%/21%
13:45-14:00	951.6	0.147	0.86	0.0	79%/21%
14:00-14:15	882.1	0.151	0.73	0.0	56%/44%
14:15-14:45	882.1	0.0	0.73	0.0	56%/44%
14:45-15:15	815.7	0.0	0.59	0.0	34%/44%
15:15-15:30	755.3	0.0	0.42	0.0	14%/86%

Combustion efficiencies were determined from ash analyses by calculating the amount of carbon burnt, using the following formula:

$$\text{Combustion Efficiency} = \frac{\text{Total Carbon} - \text{Unburned Carbon}}{\text{Total Carbon}}$$

By collecting the fly ash samples. We can analyse the carbon content in the fly ash. The results are listed in Table 10. Combustion efficiencies greater than 97% and frequently around 98%~99% were achieved without employing char recycle. This value of combustion efficiency compares well with values reported elsewhere⁴. The data for coal and wood is also shown in Table 10.

Thermal efficiency is calculated by the thermal balance using the following formula:

$$\text{Thermal Efficiency} = \frac{\text{Exchanged Heat Value}}{\text{Coal and Wood Heat Value}}$$

The thermal efficiency values in Table 11 range from 69.6% to 83.5%. This range of thermal efficiency values for coal and wood compare well with values reported by Dugum and associates³.

3.2 Burn of May 10, 1995

The burn protocols are in general similar to those described above for the burn of April 25, 1995. In this burn, however, only coal was used as fuel, a new gas transfer line (at 1,350°F) was tested, and the air-to-fuel ratio was altered deliberately during the run. Analysis of the coal used (WKU #95011) is shown in Table 12. The experimentally varied conditions are given in Table 13, and the combustor operating conditions in Table 14.

Table 10

Carbon Content in the Fly Ash and Combustion Efficiency

<u>Sample No.</u>	<u>Carbon (%)</u>	<u>Ash (%)</u>	<u>Combustion Efficiency (%)</u>	<u>Ratio of coal/wood</u>
4250930	21.37	76.98	97.5	100% coal
4251000	23.87	74.42	97.1	100% coal
4251030	18.38	80.42	97.9	100% coal
4251100	21.70	76.88	97.4	100% coal
4251130	13.41	84.97	98.6	100% coal
4251200	17.49	81.32	98.0	100% coal
4251230	21.16	77.40	97.5	100% coal
4251300	18.90	79.57	97.8	100% coal
4251330	16.60	82.12	98.2	100% coal
4251400	20.33	78.41	97.9	79%/21%
4251430	15.78	83.10	98.8	56%/44%
4251500	7.84	90.79	99.3	34%/66%
4251540	5.29	93.13	99.8	14%/86%

Table 11

Table 11

Combustion Efficiency

<u>Coal/Wood Wt. (lbs/lbs)</u>	<u>Coal/Wood Wt. Rate (%/%)</u>	<u>Combustion time (min)</u>	<u>Fuel output (Btu/min)</u>	<u>Thermal Efficiency (%)</u>
98.2	100% coal	280	4822.32	69.6
12.2/3.3	79/21	39	4973.97	81.6
8.8/3.3	56/44	36	4862.78	83.5
6.3/12.4	34/66	38	4873.82	83.3
3.4/20.6	14/86	43	4895.81	82.9

Table 12

Analysis of Coal #95011^A

Moisture (wt %)	7.32%
Ash	8.68
Carbon	68.66
Hydrogen	5.53
Nitrogen	1.43
Sulfur	2.97
Oxygen	12.74
HHV (Btu/lb)	12,240

^A as determined

Table 13

Varying the Air to Fuel Ratio

<u>time</u>	<u>ratio of air to fuel (by weight)</u>
8:30-9:00	38.2 : 1
9:00-9:30	25.4 : 1
9:30-10:00	20.7 : 1
10:00-10:30	27.5 : 1
10:30-11:00	31.2 : 1
11:00-11:30	35.5 : 1
11:30-12:00	30.8 : 1
12:00-12:30	27.9 : 1
12:30-13:00	30.7 : 1
13:00-13:30	27.6 : 1
13:30-14:00	20.5 : 1
14:00-14:30	68.9 : 1
14:30-15:00	25.3 : 1

GC results. Again, gas chromatography was used to monitor the principal fixed gases, and it was found that the nitrogen concentration was nearly constant at 70-76%. Moisture did not fluctuate greatly, remaining in the range 8.3-10.8%. CO again appears in the start-up period of unstable combustion, after which it is no longer evident. The reciprocal fluctuations of CO₂ and O₂ as the air/fuel ratio is altered are seen in Figure 50.

Table 14

Combustion Test Matrix

Bed area	125 in ²
Coal feed rate	11.5 to 22 lbs/hr
Coal feed size	0.2 x 0 in
Bed temperature	~1,500°F
Fluidized velocity	2.69 fps
Bed material/sorbent	0.16 x 0 in Limestone

Table 15

Some CO₂ Concentrations (by FTIR)

<u>Time</u>	Port 1		Port 2	
	<u>I</u>	<u>Concn</u>	<u>I</u>	<u>Concn</u>
0928-1004			1365°	10.1%
1008-1027	1015°	7.4%		
1030-1049			1410°	9.4%
1052-1116	1038°	6.2%		
1120-1139			1390°	8.9%

3.3 Discussion

GC was used to measure CO_2 , CO , N_2 , O_2 , H_2O and other gases, and it was found that N_2 concentration was almost constant during the combustion, at 70%~76%, the moisture concentration was also almost constant, ranging from 8.3% to 10.8%. CO occurs in the early period of unstable combustion and briefly when changing the ratio of air/fuel, but once stability is achieved the CO peak disappears, indicating efficient combustion of carbon.

During the combustion, the air / fuel ratio has been deliberately varied (Table 13). To measure the CO_2 concentration, gas samples were collected from two different bed heights [port 1 and port 2 in Table 15].. The concentration of CO_2 at port 2 is higher than that at port 1. But the bed height of port 1 is greater than that of port 2. Theoretically, if there is CO in port 2, there is less CO and more CO_2 in port 1 than in port 2, because CO and O_2 can produce CO_2 as the gases flow upwards. If there is no CO in port 2, the CO_2 concentration in port 1 should be as almost the same as in port 2. The unexpected results (Table 15) suggest a possible air leak between the ports. Corrective modifications are being made.

IC has again been used to measure chloride and sulfate. Every 15 minutes, we collected an IC sample of 100 mL. IC results are shown in Table 16.

The IC data shows that SO_2 was not detected. However, water drops were found on the inside surface of transfer line from the GC outlet to the pump. Based on experience in using TGA-FTIR-MS for analysis in the TA lab., we believe that when moisture in a gas stream condenses into water drops, most of the acid gases HCl and SO_2 dissolve in the

Table 16

IC Results

<u>Time</u>	<u>Chlorine (ppm)</u>		<u>SO₂ (lbs/MBtu)</u>		<u>Ratio of Air/Fuel</u>
	<u>Coal</u>	<u>IC</u>	<u>Coal</u>	<u>IC</u>	
8:45-9:00	24	0.116	4.85	0.0	38.2:1
9:00-9:15	24	0.129	4.85	0.0	25.4:1
9:15-9:30	24	0.0	4.85	0.0	25.4:1
9:30-9:45	24	0.027	4.85	0.0	20.7:1
9:45-10:00	24	0.053	4.85	0.0	20.7:1
10:00-10:15	24	0.101	4.85	0.0	27.5:1
10:15-10:30	24	0.045	4.85	0.0	27.5:1
10:30-10:45	24	0.036	4.85	0.0	31.2:1
10:45-11:00	24	0.037	4.85	0.0	31.2:1
11:00-11:15	24	0.034	4.85	0.0	35.5:1
11:15-11:30	24	0.034	4.85	0.0	35.5:1
11:30-11:45	24	0.078	4.85	0.0	30.8:1
11:45-12:00	24	0.069	4.85	0.0	30.8:1
12:00-12:15	24	0.049	4.85	0.0	27.9:1
12:15-12:30	24	0.043	4.85	0.0	27.9:1
12:30-12:45	24	0.070	4.85	0.0	30.7:1
12:45-13:00	24	0.112	4.85	0.0	30.7:1
13:00-13:15	24	0.108	4.85	0.0	27.6:1
13:15-13:30	24	0.088	4.85	0.0	27.6:1
13:30-13:45	24	0.055	4.85	0.0	20.5:1
14:00-14:15	24	0.046	4.85	0.0	20.5:1
14:15-14:30	24	0.0	4.85	0.0	68.9:1
14:30-14:45	24	0.0	4.85	0.0	68.9:1
14:45-15:00	24	0.067	4.85	0.0	25.3:1
15:00-15:15	24	0.124	4.85	0.0	25.3:1

droplets. Hence these IC results are not a true estimate of chloride and SO_2 . In order to get the correct IC result, we will collect future IC samples at the GC outlet.

During the May 10th burn, the thermal efficiencies were in the range from ~32% to ~59%. The detailed calculation results are listed in Table 17. Compared to the result of April 25th combustion, the thermal efficiencies are lower, because the emphasis this time was variation in the air-to-fuel ratio.

Ratio of Air/Fuel and Bed Temperature. Figure 51 shows that a sharp increase in the air/coal ratio results in a sharp drop in temperature. When the high air/coal ratio is reduced, bed temperature rises sharply.

Combustion Efficiency. Combustion efficiencies were determined, as before, from the ash analyses by calculating the amount of carbon that was burnt.

By collecting the fly ash samples to analyze the carbon content in the fly ash. The results are listed in Table 18. Combustion efficiencies greater than 95% and frequently around 96%~99% were achieved without employing char recycle. This range of combustion efficiency value compares well with values reported elsewhere³. Other data for coal combustion efficiency are presented in Table 11.

3.4 Discussion

Gas chromatography was used to provide semicontinuous measures of CO_2 , CO , N_2 , O_2 , H_2O and other gases in the combustion offgas. N_2 concentration was found to be almost constant during the combustion, at 70%~76%. Moisture content was also almost constant, ranging from 9.5% to 10.3%. CO only occurs in the early unstable combustion

Table 17

Thermal Efficiency

<u>Time</u>	<u>Thermal Efficiency(%)</u>	<u>Ratio of Air/Fuel</u>
08:30-09:00	58.84	38.2:1
09:00-09:30	39.11	25.4:1
09:30-10:00	31.87	20.7:1
10:00-10:30	42.40	27.5:1
10:30-11:00	48.11	31.2:1
11:00-11:30	54.76	35.5:1
11:30-12:00	47.46	30.8:1
12:00-12:30	42.98	27.9:1
12:30-13:00	47.35	30.7:1
13:00-13:30	42.56	27.6:1
13:30-14:00	51.80	20.5:1
14:00-14:30	46.80	68.9:1
14:30-15:00	39.10	25.3:1

Table 18

Carbon Content in the Fly Ash and Combustion Efficiency

<u>Sample No.</u>	<u>Carbon(%)</u>	<u>Ash(%)</u>	<u>Combustion Efficiency(%)</u>	<u>Ratio of Air/Fuel</u>
0510-0900	46.39	53.61	92.49	38.2:1
0510-0930	31.58	68.42	95.99	25.4:1
0510-1000	34.16	65.84	95.50	20.7:1
0510-1030	29.22	70.78	96.42	27.5:1
0510-1100	31.84	68.16	95.95	31.2:1
0510-1130	29.88	70.12	96.30	35.5:1
0510-1200	30.52	69.48	96.19	30.8:1
0510-1230	20.40	79.60	97.78	27.9:1
0510-1300	25.02	74.98	97.10	30.7:1
0510-1330	36.24	63.76	95.07	27.6:1
0510-1400	9.03	90.97	99.14	20.5:1
0510-1430	41.12	58.88	93.94	68.9:1
0510-1500	16.43	83.57	98.29	25.3:1

period. Once stability was achieved, the CO peak disappeared, showing the carbon combustion to be efficient. Figures 47 and 48 show the concentrations of CO₂ and O₂ in two periods of this run. When CO₂ concentration decreases, O₂ concentration increases, and vice versa. Indeed, the sum of CO₂ and O₂ concentrations remains constant. Bed and flue gas temperatures remain throughout in fairly narrow ranges (Figure 49). Thus with excess O₂, at about 1500 °F bed temperature, the main reaction of carbon⁵ is

Owing to the stoichiometry of the above reaction, the molar sum of O₂ and CO₂ is constant.

FTIR was employed to measure CO₂, CO, CH₄, SO₂ and other gases. From FTIR's wavenumber charts, CO and CH₄ can be seen only in the early period of instability.

The bench-scale fluidized bed combustion tests were conducted on a low sulfur (~2.97% sulfur) WKU coal No. 95011 in the WKU fluidized bed. The bed was operated at ~1500 °F at a fluidizing velocity of 2.69 fps. SO₂, CO₂ and other gas emissions were monitored by FTIR.

Summary. The bench-scale fluidized bed combustion tests were conducted on a low sulfur (~0.65% sulfur) WKU coal No. 95010 in the WKU fluidized bed on April 25, 1995. The six-hour burn started with coal, gradually switching to sawdust pellets. The first gas analysis line detected CO₂, SO₂, CO, H₂O, O₂, and N₂ quantitatively. No difficulty was encountered in using a mixture of 95% sawdust and 5% coal to maintain the bed

temperature around 1500 °F. [A plot of bed temperature vs time is shown in Figure 49.]

The fluidizing velocity is 3.69 fps. The results of tests show that:

1. Combustion efficiencies greater than 97 percent were achieved without employing char recycle. Generally, the combustion efficiencies of mixtures of coal and wood were greater than those of pure coal.
2. Thermal efficiencies from 69.6% to 83.5% were realized in each of the tests conducted with all of the fuel blends tested.
3. By using limestone as bed material, sulfur capture of more than 90% was achieved.
4. By employing techniques of IC, GC and FTIR, CO₂, CH₄, CO, SO₂, O₂, N₂, Cl and other gases could be effectively analyzed and quantified.
5. Port 2 works better than Port 1, because we obtain quantitative results for CO₂ and other gases.

4. GC/Mass Spectrometry

In preparation for the identification and proximate quantitation of individual organic compounds formed in the course of coal combustion (task C-3), the Shimadzu QP-5000 GC/Mass Spectrometer has been fully evaluated, using the group of 27 organic compounds most frequently reported as coproducts of coal combustion.¹ An analytical program has been developed (Table 19) which separates each of these compounds, and in addition can distinguish 32 additional suspected product compounds as well as several spiking standards [reference compounds which themselves will not be formed in coal combustion]. The spiking standards are fully deuterated compounds: phenol-d6 [b. 180°C], naphthalene-d8 [b. 218°], phenanthrene-d10 [b. 340°], p-terphenyl-d14 [b. 390°] and chrysene-d12 [b. 448°].

Two compounds, anthracene and phenanthrene, are commonly found in combustion products and are often reported together, since their GC retention times are sometimes indistinguishable and their mass spectra are virtually identical. We have resolved this pair, using a 60-m x 0.32-mm capillary column coated with the polydimethylsiloxane Rtx-1 (Table 19). Each member of this pair has been identified and verified using pure analytes.

When compounds have been identified and their mass spectra determined, the most sensitive way to use the mass spectrometer is in its selected ion monitoring (SIM) mode. SIM runs with this instrument provide both higher signals -- since the instrument is spending most of its time measuring the signals from the principal mass fragments of the targeted compounds -- and also lower background noise. For a group of test

hydrocarbons, the useful threshold sensitivity for integrating peak areas in the scanning mode is ~100 pg. [1 pg = 10^{-12} g.] In the SIM mode using four selected m/e fragments this threshold sensitivity drops to ~ 1 pg.

Table 19

GC/MS Conditions for the Simultaneous Analysis of 65 Compounds

GC	injector	300°	MS	electron multiplier	1.50 kV
	transfer line	290°		commence scan	5.0 min
	column	60m x 0.32mm		mass range	40-400 amu
		Rtx-1, 1.0 μ m		scan rate	4 Hz
	col temp	100° + 5°/min to		integrator width	1 sec
		330°, then isothermal		threshold	100 μ v
	carrier flow	1.50 mL/min throughout			
	lin velocity	31.1 cm sec ⁻¹			

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7. Figures-

Figure 1. TG-FTIR-MS Analytical System

Figure 2. TGA-MS Capillary Interface

Figure 3. FTIR Spectra of Cellulose

Hide3D Y-Zoom CURSOR

12/21/94 11:39 AM Res=4 cm⁻¹

Absorbance / Wavenumber (cm⁻¹)

File # 1 = CELLULOSE#20 @ 100124

Scanned in air EXAM/min, 100/min to 7000

Figure 4. FTIR Spectra of Newspaper

Absorbance / Wavenumber (cm-1)

File # 1 = NEWSPEAPE#16 @ 793 329

Scanned in air 1000/min, 100/min to 7000

Hide3D Y-Zoom CURSOR

12/21/94 1:37 PM Res=4 cm-1

Figure 5. FTIR Spectra of PVC

Hide3D Y-Zoom CURSOR

11/27/94 10:00 AM Res=4 cm-1

Absorbance / Wavenumber (cm-1)

File # 1 = PVC3XMG#14 @ 600 6533

pvc3x.mg, air 60ml/min, 100/min to 700C

Figure 6. FTIR Spectra of Coal 90003

Hide3D Y-Zoom CURSOR

11/27/94 12:59 PM Res=4 cm⁻¹

Absorbance / Wavenumber (cm⁻¹)

File # 1 = C003000#15 @ 766.4512

0.00001, 0.00001, 100mg, air CO/Min, 10C/min to 700C

Figure 7. FTIR Spectra of Coal 92073

Hide3D Y-Zoom CURSOR

11/4/94 9:26 AM Res=4 cm⁻¹

File # 1 = C02072#14 @ 000 4735

100/Min/Kilohertz 00.3XXmg

Figure 8. Evolved Gas Profiles for Coal 90003

Figure 9. Evolved Gas Profiles for PVC

Figure 10. Evolved Gas Profiles for Newspaper

Figure 11. CH₄ Gas Release Profiles

Absorbance / Wavenumber (cm-1)

File # 1 = 03NEWS50#10 @ 481.4844

50mg90003 50mgnewspaper in air 50ml/min, 10C/min to 700C

Hide3D Y-Zoom CURSOR

12/25/94 12:55 PM Res=4 cm-1

Figure 12

Absorbance / Wavenumber (cm-1)

File # 1 = 03CELL50#16 @ 802.3779

50mg/50003.50mgcellulose in air 50ml/min, 10C/min to 700C

Hide3D Y-Zoom CURSOR

12/24/94 11:27 AM Res=4 cm-1

Figure 13

90003-50mg,pvc50mg in air 50ml/min,10c/min to 700c

File # 1 = 03PVC50#14 @ 696.144

Absorbance / Wavenumber (cm⁻¹)

12/23/94 2:56 PM Res=4 cm⁻¹

Hide3D Y-Zoom CURSOR

Figure 14

Absorbance / Wavenumber (cm-1)

File # 1 = 035122#40 @ 2067.511

50mg90003, 10mgpvc, 20mgcellulose, 20mgnewsaper in air 50ml/min, 10C/min to 700C

Paged Y-Zoom CURSOR

1/7/95 2:34 PM Res=4 cm-1

Figure 15

Absorbance / Wavenumber (cm-1)

File # 1 = 035122#22 @ 1113.275

50mg90003, 10mgpvc, 20mgcellulose, 20mgnewspaper in air 50ml/min, 10C/min to 700C

Paged Y-Zoom CURSOR

1/7/95 2:34 PM Res=4 cm-1

Figure 16

Absorbance / Wavenumber (cm-1)

File # 1 = 035122#40 @ 2067.511

50mg90003, 10mgpvc, 20mgcellulose, 20mgnews paper in air 50ml/min, 10C/min to 700C

Paged Y-Zoom CURSOR

1/7/95 2:34 PM Res=4 cm-1

Figure 17

Absorbance / Wavenumber (cm-1)

File # 1 = 035122#20 @ 1007.249

50mg90003, 10mgpvc, 20mgcellulose, 20mgnews paper in air 50ml/min, 10C/min to 700C

Paged Y-Zoom CURSOR

1/7/95 2:34 PM Res=4 cm-1

Figure 18

Absorbance / Wavenumber (cm-1)

File # 1 = 735122#43 @ 2247.889

92073-50mg,pvc10mg,news20mg,cellulose10mg in air 50ml/min,10c/min to 700c

Paged Y-Zoom CURSOR

12/23/94 1:29 PM Res=4 cm-1

Figure 19

Absorbance / Wavenumber (cm-1)

File # 1 = 735122#20 @ 1016.902

92073-50mg,pvc10mg,news20mg,cellulose10mg in air 50ml/min,10c/min to 700c

Paged Y-Zoom CURSOR

12/23/94 1:29 PM Res=4 cm-1

Figure 20

Absorbance / Wavenumber (cm-1)

File # 1 = 735122#43 @ 2247.889

92073-50mg,pvc10mg,news20mg,cellulose10mg in air 50ml/min,10c/min to 700c

Paged Y-Zoom CURSOR

12/23/94 1:29 PM Res=4 cm-1

Figure 21

Absorbance / Wavenumber (cm-1)

File # 1 = 735122#28 @ 1445.071

92073-50mg,pvc10mg,news20mg,cellulose10mg in air 50ml/min,10c/min to 700c

Paged Y-Zoom CURSOR

12/23/94 1:29 PM Res=4 cm-1

Figure 22

Figure 23 Mass-to-Charge Ratios for Scan 26 in TG-MS Analysis of Coal 92073

Time: 00:07:10

Scan: 26

File: C82073

05:06 14-Apr-85

A1: 0.07 A2:

Cursor = 1

Created: 19:36:21

29-Dec-84

Figure 24 Evolved Gas Profiles for Combustion Gases for Coal 92073

Figure 25 Evolved Gas Profiles for Combustion Gases for PVC

Figure 26

Profiles for HCl and Cl₂ Evolved During Combustion of PVC

Figure 27

Time: 00:29:28

File: 73NEW50

Scan: 75

22:18 26-May-95

A1: -0.02 A2:

Cursor = 10

Created: 14:08:05

12-Jan-95

Time: 00:31:07

File: 73CEL50

Scan: 80

22:13 26-May-95

A1: -0.02 A2:

Cursor = 10

Created: 08:52:19

12-Jan-95

22:24 26-May-95

File: 73P25N25

Scan: 77

Time: 00:30:54

A1: -0.02 A2:

Cursor = 10

Created: 18:28:44

12-Jan-95

22: 08 26-May-95

File: 735122

Scan: 76

Time: 00: 30: 17

3.94M-9

Figure 31

A1: -0.10 A2:

Cursor = 48

Created: 09: 19: 03

17-Jan-95

22: 01 26-May-95

File: 03PVC50

Scan: 73

Time: 00:29:18

A1: 0.12 A2:

Cursor - 0

Created: 11:12:05

30-Dec-94

Time: 00:30:50 Scan: 78 File: 03CEL50 21:50 26-May-95

Time: 00:30:46

Scan: 78

File: 03NEW50

21:39 26-May-95

A1: 0.00 A2:

Cursor = 1

Created: 19:23:06

30-Dec-94

21: 55 26-May-95

File: 03P25N25

Scan: 70

Time: 00: 27: 57

31-Dec-94

Created: 10: 38: 05

Cursor = 0

A1: -0.02 A2:

09: 12 28-May-95

File: 73PVC50M

LC: 00: 00: 14

Profile: BLANK

No: 1

74*0.3	0.00M-20
72*0.3	1.51M-13
70*0.3	4.04M-13
38*0.01	6.12M-13
36*0.01	1.65M-12

Figure 37

08:52 28-May-95

File: 73P25N25

Scan: 1

LC: 00:00:14

Profile: BLANK

74*0.2

72*0.2

70*0.2

38*0.002

36*0.002

Figure 38

Time (h m s)

Scan No

Created: 19:48:16

12-Jan-95

LC: 00:00:12 Scan: 1 File: 735122

OB: 41 28-May-95

Profile: BLANK

74

72

70

38*0.05

36*0.05

Figure 39

Time (h m s)

Scan No

1 21 41 61 81 101 121 141 161 181 201 221

Created: 10:19:48

17-Jan-95

LC: 00:00:09 Scan: 1 File: 03PVC50 11:37 27-May-95

Profile: BLANK

Time (h m s)	Temp (Deg C)
00 10 00	713
00 20 00	
00 30 00	
00 40 00	
00 50 00	
01 00 00	

Created: 11:51:51 30-Dec-94

LC: 00:00:08 Scan: 1 File: 03P25N25 08:32 28-May-95

Profile: blank

Time (h m s)

Scan No

Created: 11:08:54

31-Dec-94

LC: 00:00:08 Scan: 1 File: 035122 08:22 28-May-95

Profile: blank

Time (h m s) Scan No
00 10 00 21
00 20 00 41
00 30 00 61
00 40 00 81
00 50 00 101
00 55 00 121
01 00 00 141
Created: 10:44:17 04-Jan-95

LC: 00:00:08 Scan: 1 File: 035122 16:48 29-May-95

Profile: BLANKLI2

Figure 43

112*1500

Time (h m s)

Temp (Deg C)

637

Created: 10:44:17 04-Jan-95

LC: 00: 00: 12 Scan: 1 File: 735122 16: 43 29-May-95

Profile: BLANKLI2

112*150

Figure 44

112*150

LC: 00: 00: 08 Scan: 1 File: 035122 09: 24 28-May-95

Profile: BLANK1

Figure 45

60*5

64*2

60*5

64*2

Time (h m s) 00 10 00 00 20 00 00 30 00 00 40 00 00 50 00 01 00 00
Temp (Deg C) 637
Created: 10: 44: 17 04-Jan-95

CO2 O2 Concentration (GC)

April 25, 1995 Combustion

Figure 47

CO2 O2 Concentration (GC)

April 25, 1995 Combustion

Figure 48

Bed Temperature(°F) with time

April 25,1995 Combustion

Figure 49

CO₂, O₂ Concentration (GC)

May 10, 1995 Combustion

Figure 50

Bed Temperature(oF) with time

May 10 '95 Combustion

Figure 51