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THE UNIVERSITY OF
BRITISH COLUMBIA

Mineral Matter Content and Gross
Properties of Hat Creek Coal

Second Report - Quarterly
(January-March 31, 1977)

Supported by B.C. Hydro

DEPARTMENT OF METALLURGY

VANCOUVER, BRITISH COLUMBIA • CANADA

"Mineral Matter Content and Gross Properties of
Hat Creek Coal"

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(Financially Supported by The B.C. Hydro and Power Authority)

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Introduction

In the first quarterly report of the research project, efforts were mainly directed to include the results of identification and estimation of the mineralogical constituents of the clay samples (Total 30) supplied by Dr. T. McCollough of B.C. Hydro. This was done with the suggestion of the granting authority. The original objective of this study, which was to determine the mineralogical constituents of the coal samples, was set aside because of the urgency to study the clay samples. The study on the clay samples was completed by the end of December, 1976 and all subsequent studies were on the coal samples supplied by Mr. Max French of B.C. Hydro. The samples are identified as follows:

B.C. Hydro identification Code

76-135-91
76-135-93
76-135-135
76-135-137 (1195' - 1205')
76-135-41 (685' - 695')
76-135-43
76-135-120 (905' - 910')
76-135-121 (910' - 920')
76-135-163
76-135-164

The tests carried out on these samples are:

1. Inorganic mineral constituents (quantitative)
2. Same as above on different size fractions
3. Moisture content

4. Volatile content
5. Fixed carbon content by TGA*
6. Ash
7. Calorific value (Heat content) by a PDSC**cell (also checked by a bomb calorimeter)
8. Same as above on different size fractions
9. Ash fusion temperature by differential thermal analysis (DTA)

It should be noted that most of the samples are from one drill hole (76-135). Two other samples are from another drill hole (76-136). The last two sequential numbers of the identification code e.g. 41 and 43, 91 and 93, 120 and 121, 135 and 137 and 163 and 164 belong to a pair of specimens which are adjacent but different ash fusion temperature, as determined by Dolmage-Campbell Associates of Vancouver. Thus, it was considered that a quantitative analysis of inorganic minerals present in these samples might shed some light on the reason for this variation of ash fusion temperature.

Materials and Procedures

The coal samples as supplied are quite fine (~ 60 mesh minus) and each batch weighs ~ 100 gms. The coal samples as-received are subjected to x-ray analysis for identification and quantitative estimation of inorganic minerals present. The method used for quantitative estimation of minerals has already been reported in the first quarterly report. This essentially involves (i) measurement of the integrated peak area of a particular diffraction peak of a mineral, (ii) normalized this area with an internal standard, and

* Thermogravimetric Analysis

** Pressure Differential Scanning Calorimetry

(iii) compared this peak area with a calibration curve of peak area versus mineral content.

Again it should be pointed out that the estimation of minerals by this method is considered to be accurate to within $\pm 3\%$ for feldspar, quartz, siderite, pyrite, calcite and kaolin but only within $\pm 5\%$ for bentonite.

Inorganic Mineral Content

The results of this part of the investigation i.e. the quantitative estimation of inorganic minerals by x-ray diffraction analysis are shown in Table I. The last column of the table shows the total clay (inorganic) constituents in the coal samples, which varied between 22 and 69%. Calcite was detected in only one sample. Epidote, a mineral belonging to the feldspar family, was also found to be present in several samples. Presence of this particular mineral will definitely lower the ash fusion temperature. The main clay constituent is kaolinite and the other major mineral is quartz.

Subsequent to this quantitative analysis, the coal samples were fractionated into different size fractions by sedimentation. This was done for two purposes: (i) to estimate bentonite, which is very fine $<0.1 \mu\text{m}$, more accurately and (ii) to assess the possibility of removing these inorganic constituents by washing, flotation or sedimentation. This latter aspect required determinations of calorific values (heat content) and total inorganic constituents of the fractionated samples.

The coal samples were fractionated into coarse, medium and fine size fractions by sedimentation. Only deionized water was used as the medium for sedimentation. The solid component settled within 5 minutes constituted the

Table I

QUANTITATIVE ANALYSIS OF HAT CREEK COAL SAMPLES

Sample Number	Clay Constituents of Coal Sample								Total % of Clay Constituents
	Kaolinite	Quartz	Bentonite	Siderite	Epidote	Feldspar	Pyrite	Calcite	
76-135-91	24%	17%	trace	13%	8%	trace	-	-	~ 62
76-135-93	33%	29%	trace	7%	-	trace	trace	-	~ 69
76-135-135	14%	14%	-	7%	3%	-	trace	-	38
76-135-137 1195'→1205'	18%	14%	trace	2%	1%	1%	trace	-	36
76-135-163	17%	17%	trace	6%	trace	trace	-	-	40
76-135-164	22%	20%	trace	trace	trace	trace	-	-	42
76-135-41 685'→695'	38%	17%	1%	-	-	trace	-	-	56
76-135-43	25%	15%	trace	1%	1%	1%	trace	15%	58
76-136-120 905'→910'	12%	10%	trace	trace	-	trace	trace	-	22
76-136-121 910'→920'	11%	17%	-	4%	-	-	-	-	32

coarse fraction. This corresponds to particle sizes greater than 20 μm ($1 \mu\text{m} = 10^{-3} \text{ mm}$). The medium fraction consisted of material settled for 6 hours (after 5 min fraction was removed). The particle size for the medium size fraction is between 20 μm and 1.5 μm . The solid fraction left unsettled was the fine fraction having less than 1.5 μm particle size.

The results of x-ray identification and estimation of minerals present in different size fractions are shown in Table II. The last two columns in the table show the weight of different size fractions in 5 gms of samples and percent of different size fractioned materials, of the total coal samples (and not of clays only). The inorganic mineral constituents varied widely in different sized materials. For example, siderite is mainly present in the coarse and medium fractions and almost none in the fine fraction and conversely, the bentonite is mainly accumulated in the medium and fine fractions and none in the coarse fraction. Similarly, feldspar, calcite and epidote are present mainly in the size fractions larger than 1.5 μm . A more thorough and systematic study is necessary to investigate this aspect, particularly if removal of inorganic components by washing of Hat Creek coal is envisaged.

Proximate Analysis by TGA

The next phase of this investigation was devoted to the proximate analysis of Hat Creek coal by thermogravimetric analysis. It has been recently established⁽¹⁾ that this technique can be easily used to determine (i) moisture content (ii) volatile matter (iii) fixed carbon and (iv) ash. Furthermore, a single experimental run (duration \sim 90 minutes) can give all the information necessary to calculate the four components of a proximate analysis. In terms of experiments, this essentially involves heating about 30-40 milligrams of a sample at a constant

Table II

INORGANICS OF COAL SAMPLES

(5 g. of each was used for fractionation)

Sample Number		Kaolinite	Quartz	Bentonite	Siderite	Epidote	Feldspar	Pyrite	Calcite	+++ Weight Distribution	+++ Percent Distribution
76-135-91	Coarse	36%	27%	-	22%	15%	-	-	-	4.67	93%
	Medium*	51%	29%	9%	-	-	11%	-	-	0.10	2%
	Fine**	65%	25%	10%	-	-	-	-	-	0.23	5%
76-135-93	Coarse	42%	45%	-	13%	-	-	-	-	4.44	89%
	Medium*	48%	39%	-	-	-	5%	3%	-	0.21	4%
	Fine**	69%	28%	3%	-	-	-	-	-	0.35	7%
76-135-135	Coarse	33%	36%	-	22%	9%	-	-	-	3.81	76%
	Medium	41%	45%	-	12%	2%	-	trace	-	0.94	19%
	Fine	62%	37%	-	1%	-	-	-	-	0.26	5%
76-135-137, 1195' → 1205'	Coarse	44%	43%	-	7%	3%	3%	-	-	3.58	72%
	Medium	62%	38%	-	trace	-	-	trace	-	1.05	21%
	Fine	60%	40%	trace	-	-	-	-	-	0.37	7%
76-135-163	Coarse	40%	46%	-	14%	-	-	-	-	3.83	77%
	Medium	49%	32%	trace	19%	trace	trace	-	-	0.96	19%
	Fine	52%	45%	trace	3%	-	trace	-	-	0.21	4%
76-135-164	Coarse	47%	50%	-	1%	1%	1%	-	-	3.42	68%
	Medium	52%	45%	-	trace	-	-	-	-	1.09	22%
	Fine	65%	35%	trace	-	trace	trace	-	-	0.50	10%

(TO BE CONTINUED)

Table II
(Continued)

76-135- 41++	Coarse	67%	33%	trace	-	-	trace	-	-	3.57	71%
	Medium	64%	28%	8%	-	-	-	-	-	1.14	23%
685' → 695	Fine	83%	13%	4%	-	-	-	-	-	0.30	6%
76-135-43	Coarse	44%	27%	-	-	trace	-	-	29%	3.85	77%
	Medium	44%	30%	5%	trace	5%	5%	-	11%	0.99	20%
	Fine	79%	15%	6%	-	-	-	trace	-	0.16	3%
76-136- 120++	Coarse	52%	48%	-	-	-	-	-	-	4.04	81%
	Medium	52%	42%	trace	trace	-	6%	trace	-	0.80	16%
905' → 910	Fine	67%	33%	trace	-	-	-	trace	-	0.16	3%
76-136- 121	Coarse	34%	56%	-	10%	-	-	-	-	4.01	80%
	Medium	34%	46%	-	20%	-	-	-	-	0.82	16%
910 → 920	Fine	47%	47%	-	6%	-	-	-	-	0.18	4%

Coarse fraction: settled for 5 minutes

Medium fraction: settled for 6 hours after coarse fraction was removed

Fine fraction: the remaining portion

Medium fraction*: settled overnight after coarse fraction was removed

Fine fraction** : the remaining portion

+++ Weights were corrected for variation of moisture in the three fractions

++ higher fusion point

heating rate in N_2 up to about $950^\circ C$, then cooling it down to $600^\circ C$ (in N_2) and reheating the same in air (or in O_2) up to $800-900^\circ C$ until all weight loss due to combustion ceases. Some experimental plots are shown in Figures 1-3. During the first heating cycle in N_2 , there are two stages of weight loss - up to $\sim 150^\circ C$, loss of moisture (i.e. moisture content) and then starting from $150^\circ C$ and continuing to $900^\circ C$, the second stage of the weight loss is due to evolution of volatile matter and dehydration of clays (or decarbonation of carbonates if present). During the second reheating cycle in air (after cooling to $600^\circ C$ in N_2), all remaining carbonaceous material will be burnt off. This weight loss corresponds to carbon. The weight of the sample remaining after this stage is the ash content of the sample.

Table III shows the results of the proximate analyses of the coal samples as carried out by the TGA technique. Dolmage-Campbell data are also included in the table for comparison. The first column in the table shows the percentage of moisture present. This corresponds to the weight loss up to $150^\circ C$. The next three columns of the table show the volatile matter, fixed carbon and ash in percent on dry basis i.e. after moisture has been driven off at $150^\circ C$. The data for the reconstituted TGA are the calculated values from the fractionated samples. Some of these calculations are shown in Appendix I. These calculations are carried out from the x-ray estimation of different mineral content in different size fractions of samples. The known theoretical weight loss of the decomposable minerals (e.g. kaolinite, bentonite, calcite, pyrite, siderite etc.) are used to correct the volatile matter present, as the total weight loss in the temperature range 150 to $900^\circ C$ in N_2 is due to both the decomposition of the inorganic minerals and the evolution of organic volatile components. After the correction, the different amount of minerals (%) present in different size fractions are combined to obtain the reconstituted samples. The proximate analyses by TGA,

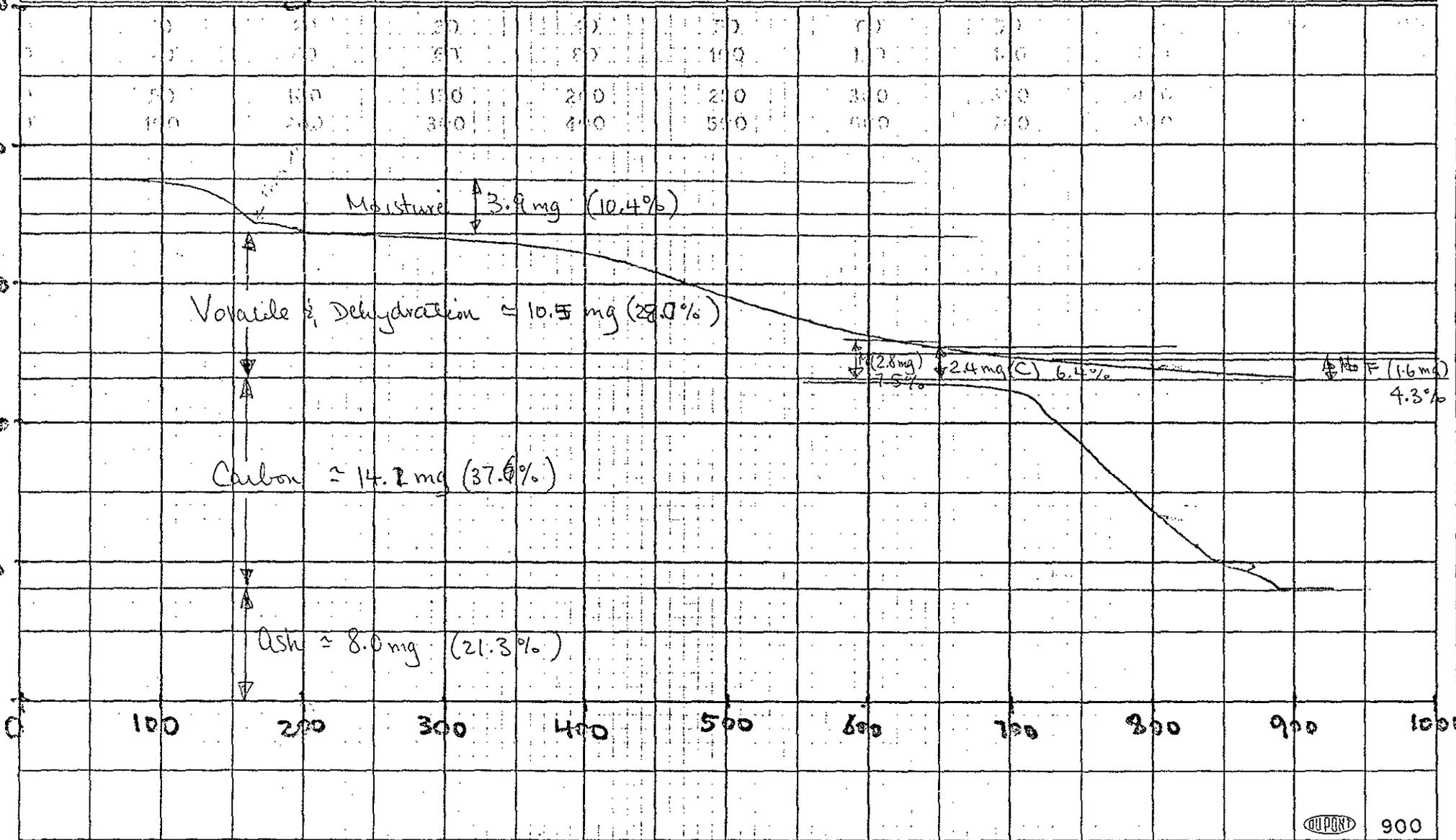
Table III

THERMOGRAVIMETRIC ANALYSIS (TGA) OF HAT CREEK COAL

Sample Number	Moisture Content	TGA of Coal Sample (% of dry coal)			* Reconstituted TGA (% of dry coal)			Dolmage-Campbell Analysis (% of dry coal)		
		Volatile	Carbon	Ash	Volatile	Carbon	Ash	Volatile	Carbon	Ash
76-135-91	3.9%	28.6%	15.7%	55.6%	27.0%	18.6%	54.4%	28.4%	14.9%	56.7%
76-135-93	8.6%	22.4%	15.6%	62.7%	23.6%	13.6%	62.8%	22.3%	15.5%	62.2%
76-135-135	5.7%	35.4%	33.9%	30.6%	34.4%	33.1%	32.5%	36.3%	32.3%	31.4%
76-135-137 1195' → 1205'	6.7%	30.3%	36.3%	30.3%	34.5%	34.5%	31.1%	32.1%	35.3%	32.6%
76-135-163	7.1%	33.6%	35.4%	31.0%	30.9%	38.5%	30.6%	35.2%	32.9%	32.0%
76-135-164	6.1%	27.4%	31.4%	41.4%	27.9%	31.9%	39.8%	28.0%	31.8%	40.2%
76-135-41 685' → 695'	9.2%	22.1%	21.8%	55.9%	28.6%	20.1%	51.3%	23.9%	21.9%	54.2%
76-135-43	10.4%	31.9%	23.2%	44.9%	30.3%	24.6%	44.1%	32.0%	24.1%	43.9%
76-136-120 905' → 910'	10.4%	31.3%	42.0%	23.7%	37.5%	41.8%	20.8%	34.3%	43.6%	22.2%
76-136-121 910' → 920'	8.5%	34.4%	35.6%	30.2%	34.5%	36.4%	28.4%	35.3%	34.8%	29.9%

* See Appendix I

SAMPLE: 120	SIZE -	ATM. 100 min. N ₂ /Air MM	RUN NO. 3
ORIGIN: 37.5 mg	REF. -	SCALE 100 $\frac{^{\circ}\text{C}}{\text{IN.}}$	DATE March 17/77
	PROG. MODE -	SHIFT 0 IN.	OPERATOR LAM
	RATE 15 $\frac{^{\circ}\text{C}}{\text{MIN.}}$, START 0 $^{\circ}\text{C}$	SHIFT 1 IN.	BASE LINE SLOPE 0

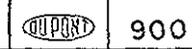


SAMPLE: _____

- 11 -

RUN NO.: _____

Figure 2



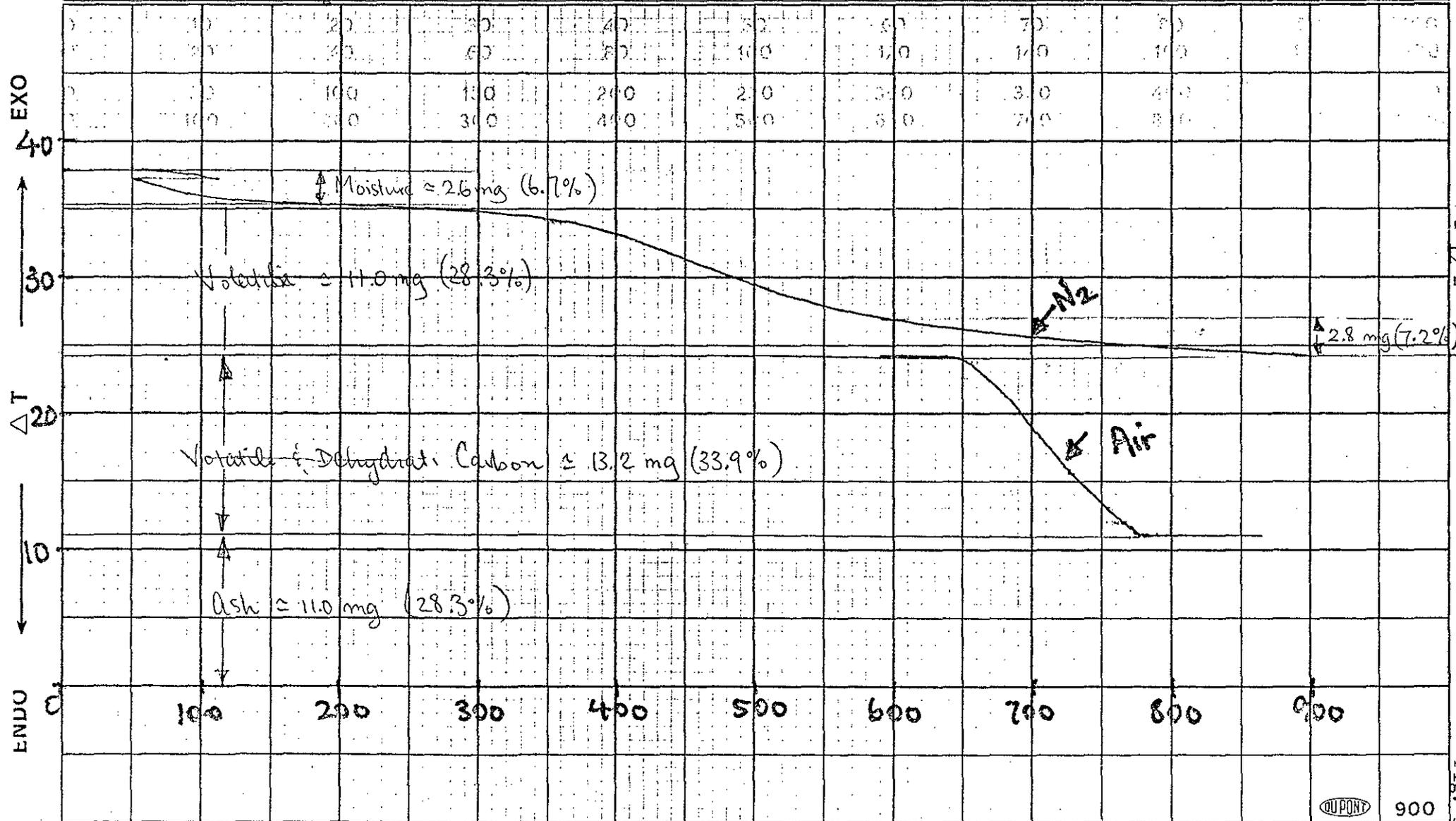
T, $^{\circ}\text{C}$ (CHROMEL: ALUMEL)*

* SEE INSTRUCTION MANUAL FOR SCALE CORRECTION

SAMPLE:	SIZE 38.9 mg	ATM. 100 ml/min N₂/Air	RUN NO. 3
	REF.	T	DATE March 15/77
	PROG. MODE	SCALE 100 $\frac{^{\circ}C}{IN.}$	OPERATOR LAK
ORIGIN:	RATE 20¹⁵ $\frac{^{\circ}C}{MIN}$, START 0⁰ $^{\circ}C$	SHIFT 0 IN.	BASE LINE SLOPE 0
		ΔT	
		10 $\frac{mg}{^{\circ}C IN.}$	
		1 IN.	

137

389.4



T, °C (CHROMEL: ALUMEL)*

* SEE INSTRUCTION MANUAL FOR SCALE CORRECTION

FIG. 3

reconstituted TGA and Dolmage-Campbell data show good agreement (within one to two percentage point).

Heat Content Determination by PDSC

Table IV shows the results of determination of the calorific value (Btu/lb) of the coal samples by a PDSC cell (Pressure differential scanning calorimetry). The method of determination of the heat content by a PDSC cell is discussed in Appendix II. The heat content values of the same coal samples as reported by Dolmage-Campbell are also included in the table for comparison. In addition, a few Par-Bomb tests were also performed on some coal samples to test the accuracy of the calorific value determinations by a PDSC cell. It appears that the determination of heat content by a PDSC cell is within $\pm 5\%$ of the values determined by the ASTM technique. Thus it can be concluded from these tests that the pressure differential scanning calorimetry is a reasonably good technique to determine the heat content value of coal.

Table V shows the proximate analysis data (moisture content, volatile matter, fixed carbon and ash) of different size fractions of coal samples. Again these data are calculated and reported on dry basis. Moisture content of the specimens are shown in the last column. The calorific values of the fractionated samples as determined by a PDSC (Pressure Differential Scanning Calorimetry) are also shown in the table. The heat content values of all coal samples including the fractionated components are plotted as a function grain size in Figure 4. As expected, the heat content of the fine-fraction materials is always lower than the coarse component, indicating that if it is possible to remove the fine fractioned component from the coal sample, the remaining coal (i.e. washed sample) will have higher calorific value than that of the unwashed coal.

Table IV
CALORIFIC VALUE OF COAL SAMPLES

	*Best Value with PDSC Calorific Value BTU/LB	** Percentage Deviation	Dolmage-Campbell Calorific Value BTU/LB	Par-bomb Test Calorific Value BTU/LB
76-135-91	3870	4	4038	-
76-135-93	3640	<-1	3616	3460
76-135-135	8360	-5	7927	-
76-135-137				
1195'→1205'	7990	2	8125	-
76-135-163	7960	-1	7882	7250
76-135-164	6240	7	6676	6580
76-135-41				
685'→695'	4750	2	4840	-
76-135-43	6070	-5	5769	-
76-136-120				
905'→910'	9280	2	9451	-
76-136-121				
910'→920'	8120	2	8285	-
ANTHRACITE	14705	-	-	14330

* These data are calculated using the best experimental calibration coefficient (E = 270).
Formula used is $\Delta H = \frac{E A \Delta T_S T_S}{Ma}$ where A is area on graph, T_S and ΔT_S are x and y axis settings respectively, M is the weight of sample, a is the heating rate and E is the calibration coefficient.

** This is the percent deviation from the Dolmage-Campbell Associates' reported values.

Table V

THERMAL ANALYSIS (TGA) OF FRACTIONS OF COAL SAMPLES

Sample Number	Fraction	*Weight (g.) (5g)	Percent of Sample	Thermogravimetric Analysis (% of dry coal)			Calorific Value by PDSC (BTU/LB)	Moisture Content
				Volatile	Carbon	Ash		
76-135-91	Coarse	4.67	93%	27.6%	20%	52.4%	4570	4.6%
	Medium	0.10	2%	32.8%	0	67.2%	2840	5.3%
	Fine	0.23	5%	13.3%	0	86.7%	750	16.6%
76-135-93	Coarse	4.44	89%	24.3%	14.9%	60.8%	5830	7.6%
	Medium	0.21	4%	19.9%	8.3%	71.7%	1190	5.2%
	Fine	0.35	7%	17.2%	0	82.8%	117	19.0%
76-135-135	Coarse	3.81	76%	34.6%	35.3%	30.0%	8770	9.3%
	Medium	0.94	19%	33.6%	30.5%	35.9%	5950	6.4%
	Fine	0.26	5%	33.7%	9.4%	56.9%	4000	37.2%
76-135-137 (1195'→1205')	Coarse	3.58	72%	34.8%	38.0%	27.2%	7750	6.9%
	Medium	1.05	21%	35.8%	31.1%	33.1%	5800	12.7%
	Fine	0.37	7%	26.3%	8.3%	65.4%	2430	30.3%
76-135-163	Coarse	3.83	77%	32.3%	39.2%	28.5%	7890	10.0%
	Medium	0.96	19%	26.8%	40.2%	33.0%	5530	9.0%
	Fine	0.21	4%	24.8%	15.9%	59.3%	2760	3.7%
76-135-164	Coarse	3.42	68%	29.8%	38.8%	31.4%	6330	7.1%
	Medium	1.09	22%	26.5%	24.7%	48.8%	3090	6.1%
	Fine	0.50	10%	21.9%	0.7%	77.4%	1470	1.7%

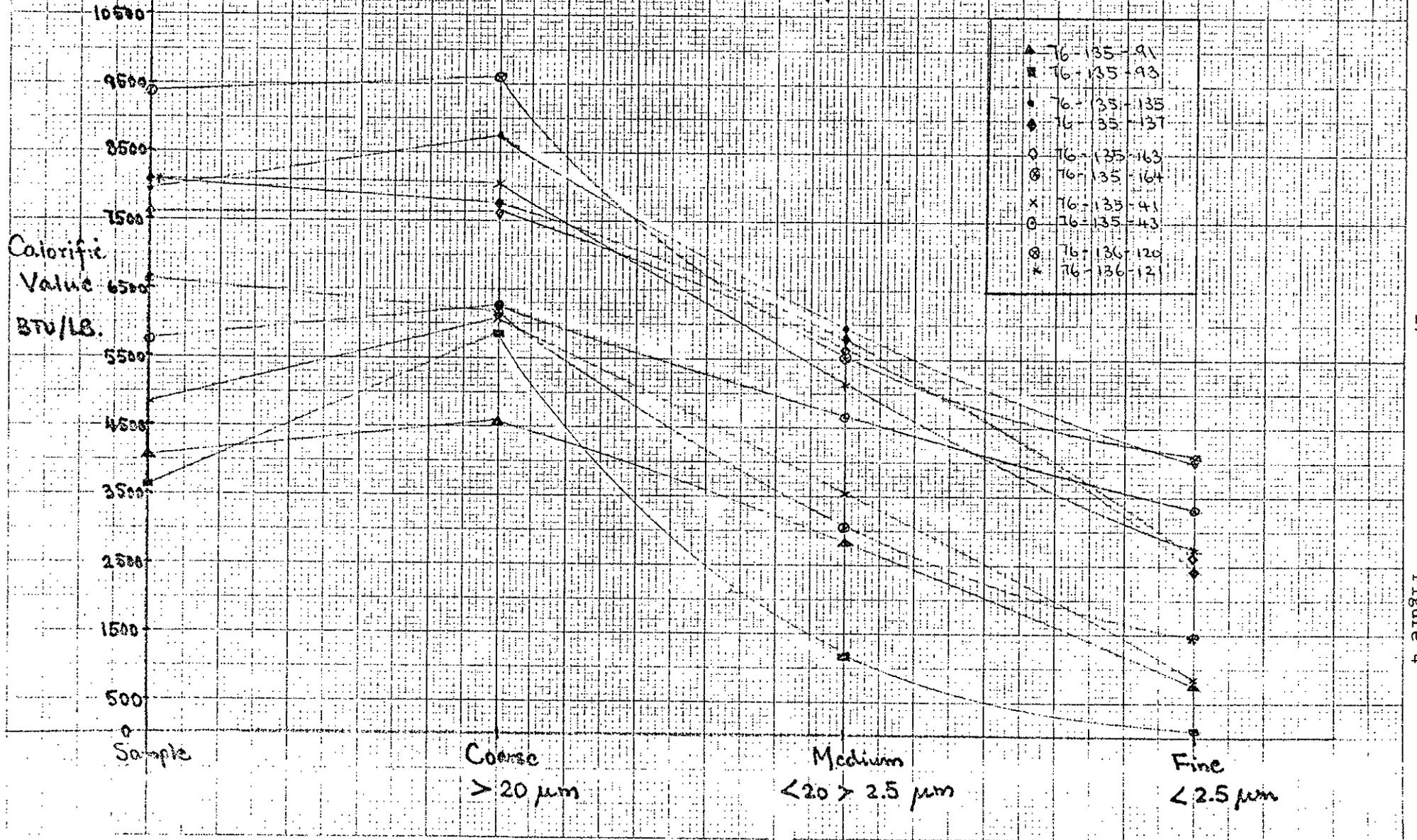
(TO BE CONTINUED)

* Weight corrected due to variation of moisture in each fraction.

Table V
(Continued)

76-135-41 (685'→695')	Coarse	3.57	71%	30.4%	24.3%	45.3%	6090	4.6%
	Medium	1.14	23%	24.8%	12.3%	62.9%	3540	8.9%
	Fine	0.30	6%	22.7%	0	77.3%	860	7.0%
76-135-43	Coarse	3.85	77%	30.9%	27.4%	41.7%	6280	4.8%
	Medium	0.99	20%	25.9%	10.9%	63.2%	4640	10.0%
	Fine	0.16	3%	30.3%	24.6%	44.1%	3330	3.4%
76-136-120 (905'→910')	Coarse	4.04	81%	37.9%	44.0%	18.1%	9630	5.3%
	Medium	0.80	16%	36.7%	35.0%	28.3%	6070	19.8%
	Fine	0.16	3%	29.4%	18.5%	52.2%	4110	4.7%
76-136-121 (910'→920')	Coarse	4.01	80%	35.7%	37.7%	26.6%	8180	6.4%
	Medium	0.82	16%	31.1%	35.0%	29.8%	5290	24.5%
	Fine	0.18	4%	25.3%	16.7%	58.0%	2860	20.4%

Calorific Value As a Function of Particle Size



Ash Fusion Temperature by DTA

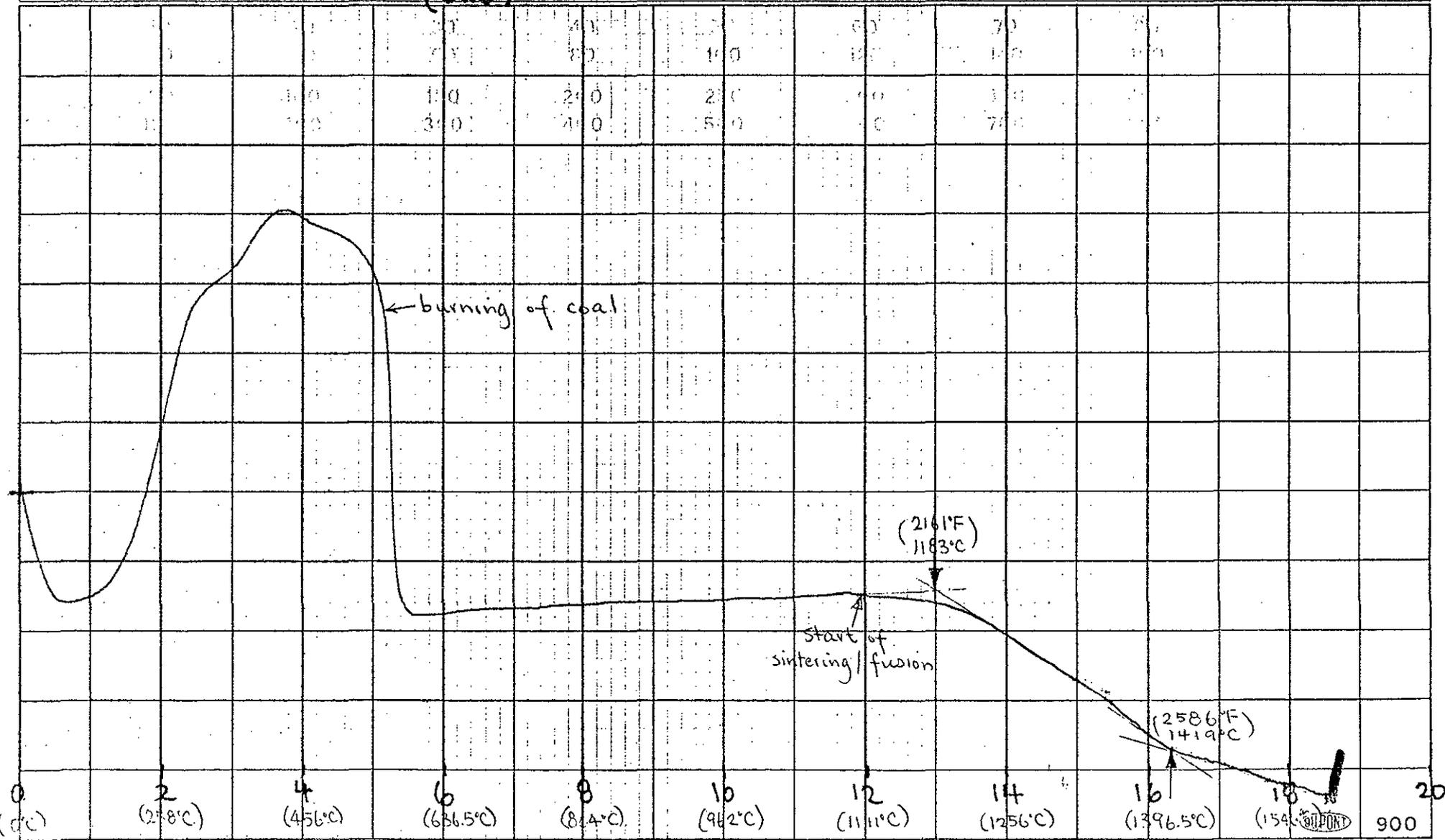
Attempts were made to investigate the possibility of determining the ash fusion temperature of coal by the differential thermal analysis technique (DTA). The differential thermal analysis plots of coal in air show a distinctive exothermic (broad) peak in the temperature range 150 to 650°C corresponding to oxidation of organic components (volatile matter + carbon). This is shown in Figures 5-7. Following this characteristic peak, there is a gradual shift of the line above 1100°C. This shift in the line is shown in the plots by drawing tangent to the line shift. A simple (and single) melting point of ash cannot be expected in view of the presence of complex inorganic minerals in the system as discussed earlier (Tables I and II). The on-set of the line shift may be due to the very initial stage of sintering, which may or may not be associated with the liquid phase formation in the system. The differential thermal analysis technique is extremely sensitive and will easily detect agglomeration of powders even in solid state (i.e. change of free surface energy of the system controlled by diffusion). Thus, it is not possible to state categorically whether the very beginning of the line shift corresponds to the initiation of fusion of ash. To complicate the issue further, the initial solid state sintering temperature is dependent on several factors: particle size (finer the particle size lowers the sintering temperature), impurities in the particles and other phases present. For example, feldspar and epidote will definitely lower the ash fusion temperature of coal. Bentonite has a much lower fusion temperature than kaolinite (at least 300 to 400°C). Bentonite itself has a wide range of fusion temperature depending upon substitution - alkali and iron in the lattice. Its initial fusion temperature can be as low as 900°C. Thus the on-set of the line shift in the DTA plots cannot be considered to be due to ash fusion in all cases. However,

when the line shift becomes progressively larger and reaches a steady state, it may be considered to be due to ash-fusion. With this consideration the "initial ash-fusion temperature" was determined from the DTA plots and shown in Table VI.

The final ash-fusion temperature similarly can be determined from the change of slope in the plots, as shown in Figure 5. In Figure 6, an exothermic reaction $\sim 1325^{\circ}\text{C}$ is indicative of mullitization (recrystallization) of aluminosilicates. If the change of slope is not very apparent (as shown in Figure 7), then it is possible that the melt is very viscous and the melting process is continuing and was not finished in this case. The final ash fusion temperature could not be determined in this particular system.

The change in slope is due to the change of specific heat of the system once melting is completed. The ash fusion temperature obtained by the ASTM cone (Pyramid) fusion technique cannot be directly compared with the values obtainable by DTA. DTA is a much more sensitive and accurate technique to determine a phase change, melting point, sintering etc. The values determined by DTA will be always lower than the ASTM technique, which is done visually from the blanting of the cone tip. On the other hand, the ash fusion temperatures obtained by DTA (Table VI) can be considered quite accurate and reliable. The lower values of ash fusion temperatures reported by Dolmage-Campbell Associates appear to agree quite well with the values determined by DTA (e.g. see the values for sample No. 91, 135, 163 and 43), but in the case of samples having high ash fusion temperatures (as reported Dolmage-Campbell Associates) there is a large discrepancy between the reported values and DTA values. This discrepancy cannot be resolved without further studies.

ORIGIN: 137 DTA	SIZE	ATM. 6 RPM. Air	MM	RUN NO. 2
	REF. ft boat - Al ₂ O ₃	T	Δ T	DATE April 4, 1977
	PROG. MODE	SCALE 2 $\frac{mV}{in}$ $\frac{^{\circ}C}{in}$.08 $\frac{mV}{in}$ $\frac{^{\circ}C}{in}$	OPERATOR LAU
	RATE 30 $\frac{^{\circ}C}{min}$, START 0 $^{\circ}C$ (5x6)	SHIFT 0 IN.	2 $\frac{1}{2}$ IN.	BASE LINE SLOPE 0

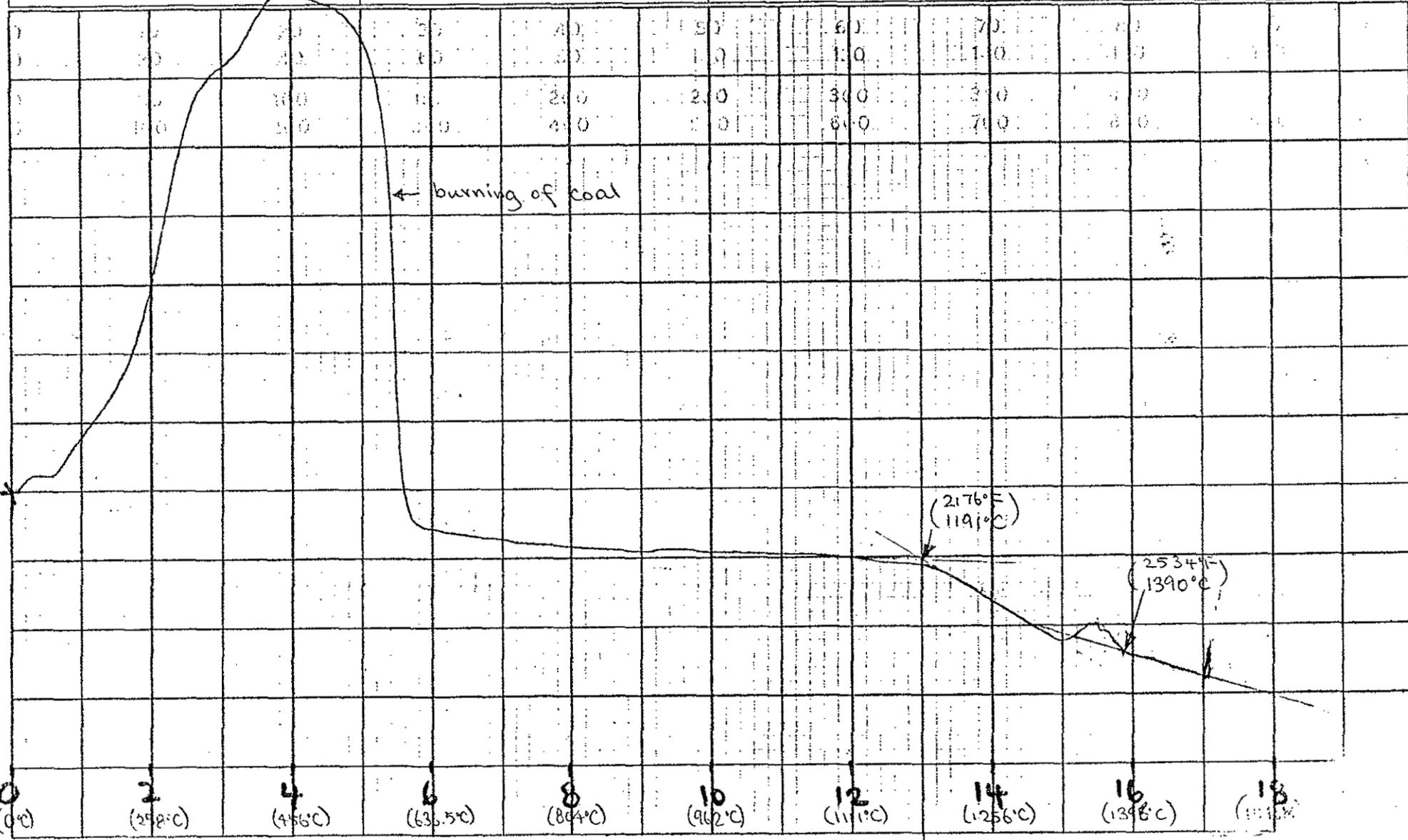


T, °C (CHROMEL: ALUMEL)*

*SEE INSTRUCTION MANUAL FOR SCALE CORRECTION

SAMPLE: 127 RUN NO.: Figure 5

SAMPLE: 121	SIZE /	ATM. .6 LPM Air MM	RUN NO. 261
	REF. Pt boat - Al₂O₃	T	DATE March 27/77
ORIGIN: DTA	PROG. MODE /	SCALE 2 °C/IN.	OPERATOR LAL
	RATE 30 °C/MIN , START 0 °C	SHIFT 0 IN.	BASE LINE SLOPE 0
	ΔT	.04 °C/IN.	
		2 1/2 IN.	

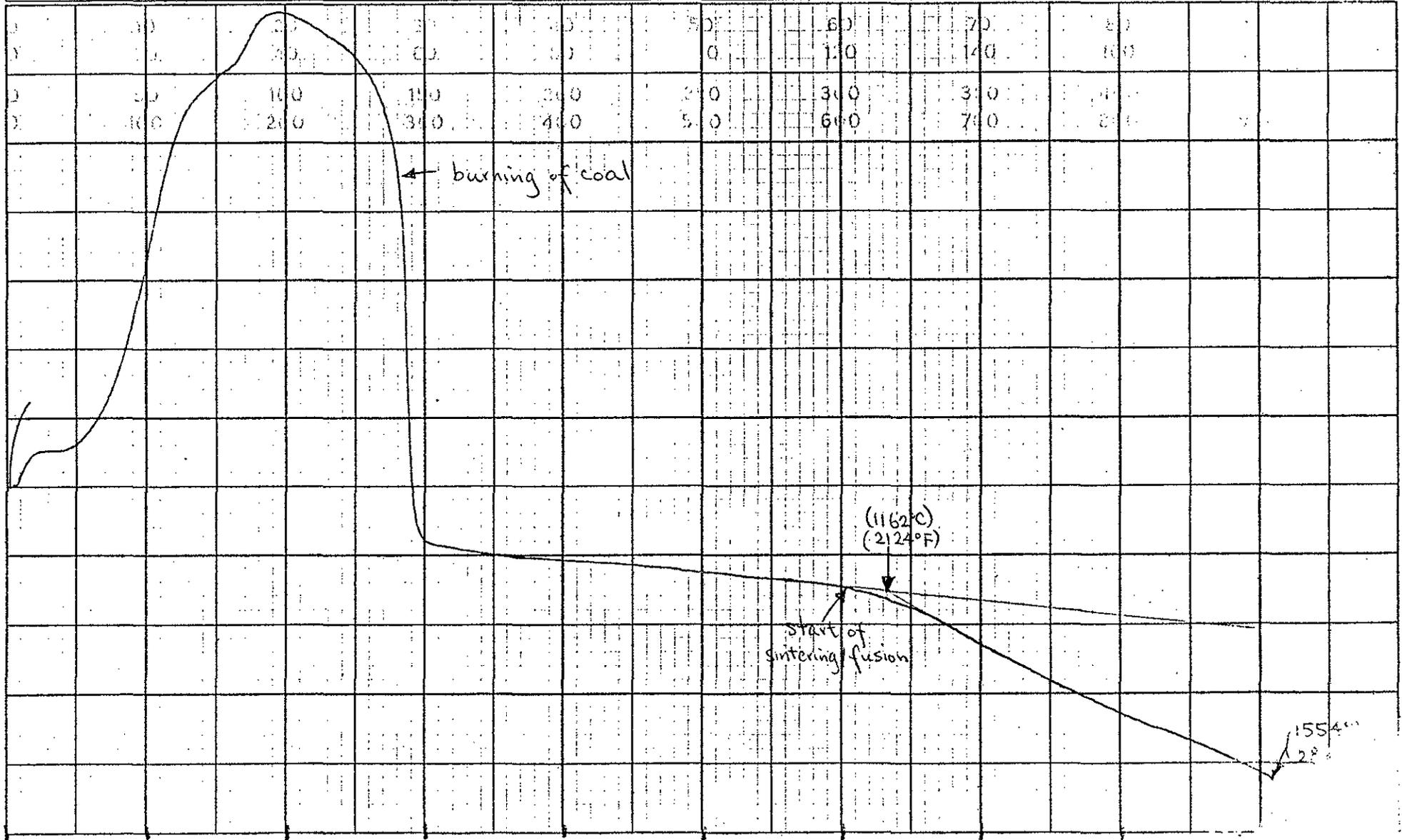


SAMPLE: _____ RUN NO.: _____

Figure 6

SAMPLE:	SIZE <u> </u>	ATM. <u>.6 LPM</u> <u>O₂</u> MM	RUN NO. <u>2</u>
	REF. <u>Al₂O₃</u>	T	DATE <u>March 25/77</u>
ORIGIN:	PROG. MODE <u> </u>	SCALE <u>2 $\frac{mV}{\mu V}$ $\frac{^{\circ}C}{m}$</u>	OPERATOR <u>LAL</u>
	RATE <u>30 $\frac{^{\circ}C}{min}$</u> , START <u>0</u> °C	SHIFT <u>0 IN.</u>	BASE LINE SLOPE <u>0</u>

120
DTA



SAMPLE: 120
RUN NO.: 2
FIGURE: 1

Table VI

Fusion Temperatures of Coal Samples

Sample Number	Fusion Temperature by DTA °F (in air) (Oxidizing Atmosphere)	Fusion Temperature by Dolmage-Campbell Asso.	
		(Oxidizing Atmosphere)°F	(Reducing Atmosphere)°F
76-135-91	2100 → 2550	2200 → 2325	2140 → 2215
76-135-93	2150 → 2600 +	2700 +	2700 +
76-135-135	2190 → 2880	2330 → 2390	2130 → 2200
76-135-137			
1195'→1205	2150 → 2590	2700 +	2700 +
76-135-163	2240 → 2800	2170 → 2230	2020 → 2080
76-135-164	2230 → 2780	2700 +	2700 +
76-135-41			
685'→695')	2160 → 2550	2700 +	2700 +
76-135-43	2300 → 2530	2360 → 2560	2130 → 2440
76-136-120			
905'→910'	2120 → 2030 +	2700 +	2700 +
76-136-121			
910'→920'	2180 → 2600 +	2415 → 2520	2015 → 2140

Conclusions

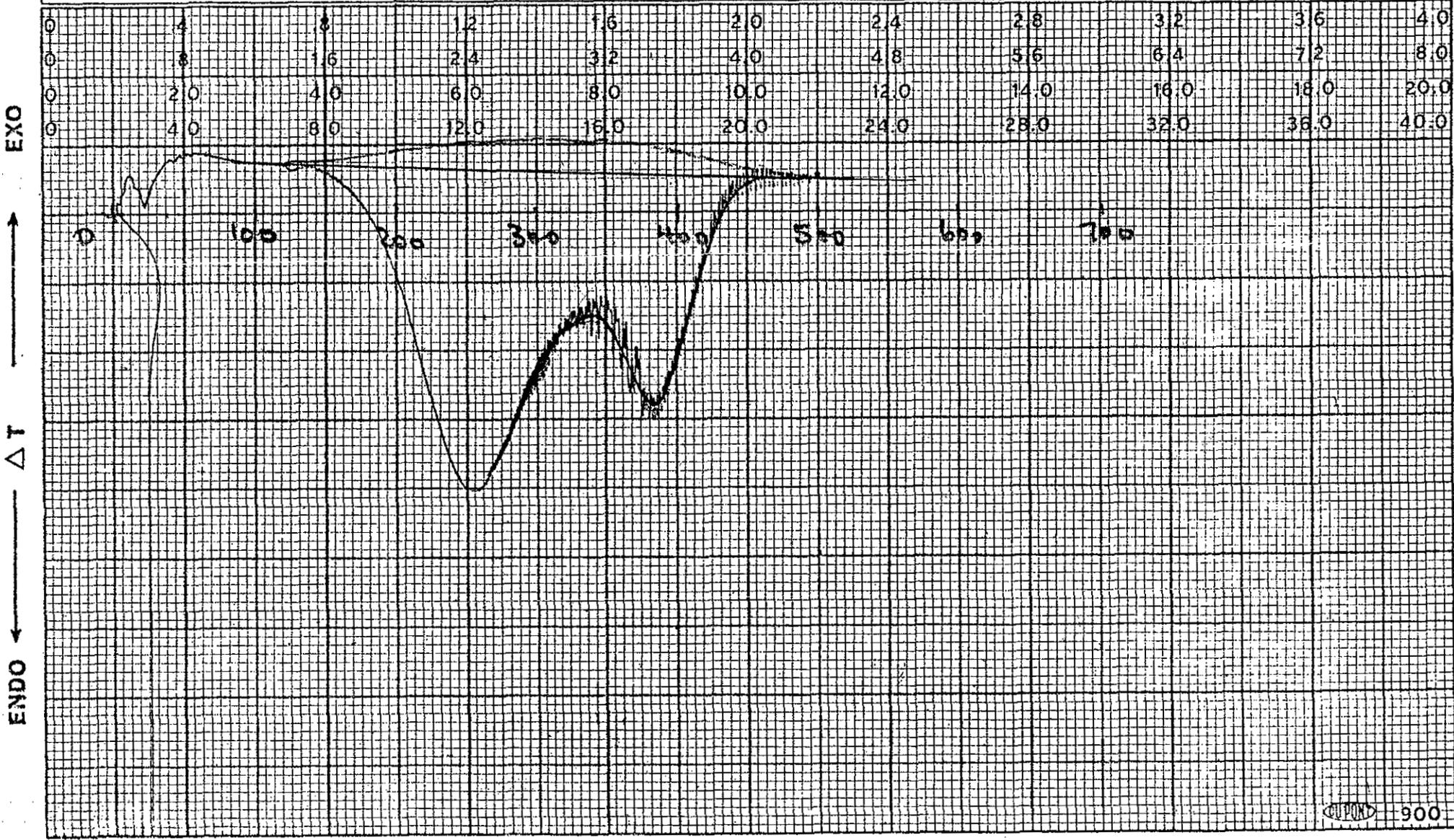
The gross properties of several samples of Hat Creek coal supplied by B.C. Hydro have been evaluated. The inorganic minerals present in the coal samples were identified and estimated by x-ray diffraction. The mineral content varied from 20 to 70%. The major components of minerals are kaolinite and quartz. Thermo-gravimetric analysis was used to perform the proximate analysis of coal samples i.e. determination of moisture, fixed carbon, volatile matter and ash. This technique has been found to be very suitable for this type of analysis. The data obtained by this technique compare very favourably with the data obtained by the conventional ASTM standard techniques. However, the TGA technique is quick and very easy to use. The heat content of the coal samples was determined by a PDSC cell, which gives the calorific value of the coal within $\pm 5\%$ after proper calibration. This technique is not very accurate but is rather easy to use. It utilizes a very small amount of sample (1-3 mg) which may be disadvantageous, as it may not be a representative sample. On the other hand, a PDSC cell produces too broad overlapping peaks corresponding to the heat content of volatile matter and the heat content of fixed carbon, as shown in Figure 8. Thus it is possible to differentiate the heat content values of these two components (VM and fixed carbon) in any sample. However, this has to be done with proper calibration.

The finer size fractions of coal samples indicate much lower calorific values as expected. These are given in Table V and shown in Figure 4. From these results it appears that if the fine fraction of coal can be removed by washing, part of the clay content (i.e. ash) can be eliminated, which should improve the calorific value of coal.

References

1. Robert L. Hassel, Coal Mining and Processing, May 1976, pp. 74-76.

SAMPLE: 120 M A	SIZE <u>16.5 mg</u>	ATM <u>500 PSIG.</u>	MM	RUN NO. _____
	REF. <u>empty Al pan</u>			DATE _____
	PROG. MODE <u>—</u>	SCALE <u>100</u> $\frac{^{\circ}\text{C}}{\text{mV IN.}}$	<u>1</u> $\frac{^{\circ}\text{C}}{\text{mV IN.}}$	OPERATOR _____
	ORIGIN:	RATE <u>20</u> $\frac{\text{min.}}{^{\circ}\text{C}}$ START <u>1</u> $^{\circ}\text{C}$.	SHIFT <u>$\frac{1}{2}$ IN.</u>	<u>$4\frac{1}{2}$ IN.</u>



- 25 -

SAMPLE: 120 M A RUN NO.: 1

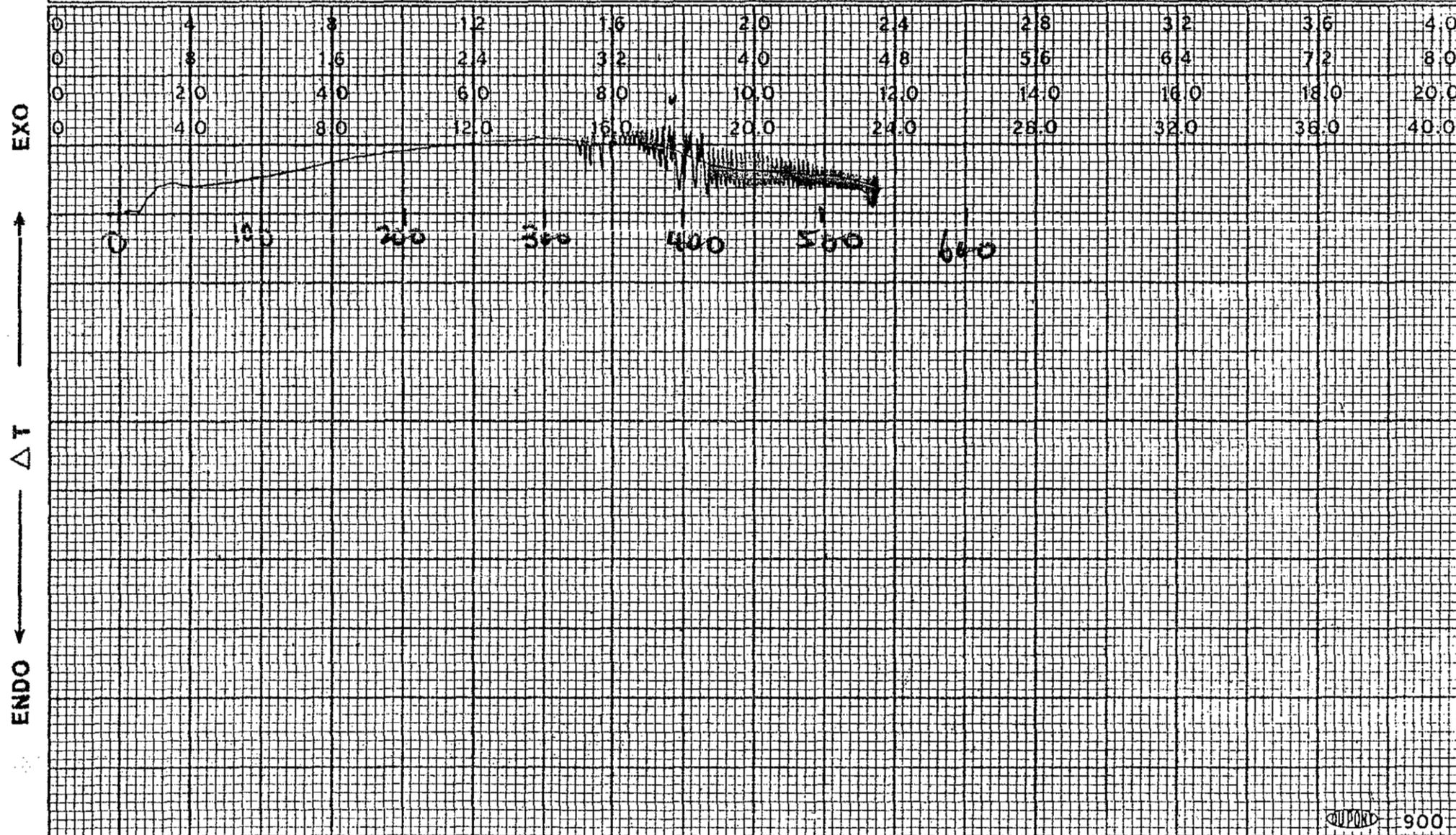
Fig. 8A

T (MILLIVOLTS: _____ THERMOCOUPLE, REF. JUNCT. 0 $^{\circ}\text{C}$)



SAMPLE:	SIZE <u>see A</u>	ATM. <u>500PSIG.</u>	MM	RUN NO. <u>2</u>
	REF. <u>empty Al pan</u>	T	ΔT	DATE <u>March 17/77</u>
ORIGIN:	PROG. MODE <u>-</u>	SCALE <u>100 $\frac{^{\circ}C}{IN.}$</u>	<u>1 $\frac{^{\circ}C}{IN.}$</u>	OPERATOR <u>LAK</u>
	RATE <u>20 $\frac{^{\circ}C}{MIN.}$</u> START <u>/</u> $^{\circ}C$.	SHIFT <u>$\frac{1}{2}$ IN.</u>	<u>4 $\frac{1}{2}$ IN.</u>	BASE LINE SLOPE <u>0</u>

120 M
B



SAMPLE: _____

- 20 -

RUN NO.: _____

FIGURE 00

Appendix I

Reconstituted TGA (% of dry coal): results of TGA of coarse, medium and fine fractions after corrected for the various minerals present.

For example:

	Analysis of Clay Material	Wt. % Distribution	Moisture Content	*Correction Factor for Carbon and Volatile	TGA results (As is)			After Correction			**Correction for Siderite			% dry coal		
					Volatile	Carbon	Ash	Volatile	Carbon	Ash	Volatile	Carbon	Ash	Volatile	Carbon	Ash
Sample	K,Q,S	100	8.5%	-	31.5%	32.6%	27.6%	-	-	-	-	-	-	34.4%	35.6%	30.2%
Coarse	K,Q,10S	80	6.4%	7.7%	27.5%	41.7%	24.5%	35.2%	34.0%	24.5%	32.8%	34.7%	24.5%	35.7%	37.7%	26.6%
Medium	K,Q,20S	16	24.5%	4.9%	23.3%	29.8%	22.5%	28.2%	24.9%	22.5%	23.5%	26.4%	22.5%	31.1%	35.0%	29.8%
Fine	K,Q,6S	4	20.4%	-	24.6%	11.9%	43.1%	24.6%	11.9%	43.1%	18.8%	12.4%	43.1%	25.3%	16.7%	58.0%
														34.5%	36.4%	28.4%

* TGA of the sample were done using N₂ atmosphere from 0°C to about 600°C and in O₂ atmospheres from 600°C to about 900°C. The change of the gas input to the furnace was carried out at about 600°C because volatiles given off after 600°C should be quite low. However, it was found that if the analyses were done this way it gave lower volatile and higher carbon contents than those given by Dolmage-Campbell Associates. It was therefore reasonable to suspect that volatiles were given off after heating over 600°C. Subsequent analyses were carried out using N₂ atmosphere from 0°C to 900°C and O₂ atmosphere from 600°C to 900°C and the results obtained were very close to those given by Dolmage-Campbell Associates. Because the coarse and medium fractions were already analysed by the old procedure, the as-received sample was analysed by the new procedure to get a correction factor for the coarse and medium fractions. The correction factor was added to the volatile and subtracted from the carbon contents.

** TGA of the various clay minerals were used to find their weight losses when heated from 0°C to about 900°C.

Appendix II

Discussion on pressure differential scanning calorimetry (PDSC)

The PDSC was used to find the calorific value of the coal samples and their sedimented fractions. The advantages of the PDSC over the Par bomb method were the extremely small sample size (1.0 → 3.0 mg) required and the ease to study the thermal reactions of the sample on the PDSC graph. The Par bomb was a better method than PDSC with respect to the accuracy of the result and the shorter length of time for each run.

Using the PDSC, two* runs were needed for each sample. Each run took about twenty-five minutes and cooling between runs took about thirty minutes. Therefore, an analysis of a sample required about one hour and twenty minutes. On the other hand, only twenty-five minutes was needed for each sample using the Par bomb, and no cooling time was necessary between runs.

The result obtained from the Par bomb was more accurate because it was representative of a 1 g coal sample. But, result from the PDSC was only representative of about 2.0 mg coal sample. There could also easily be uneven mixing of the coal sample leading to inaccurate results.

* One run for the coal, the other for establishing a baseline.