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FACTORS AFFECTING
THE SPONTANEOUS COMBUSTION INDEX

Hüseyin Nehar Eroğlu

A thesis submitted to the Faculty of Engineering, University of the Witwatersrand, Johannesburg, in fulfilment of the requirements for the degree of Doctor of Philosopy.

Johannesburg, 1992

DECLARATION

I declare that this thesis is my own, unaided work. It is being submitted for the degree of Doctor of Philosopy in the University of the Witwatersrand, Johannesburg. It has not been submitted before for any other degree or examination in any other University.

Signed _____

Dated this _____ day of _____ 1992

ABSTRACT

The self-heating of coal is a problem w ich concerns the mining industry not only in South Africa, but also in many other countries.

This thesis deals with the contouring of spontaneous combustion liabilities (WITS-EHAC index values) of different seams at the Durban Navigation Colliery and the relationship between the WITS-EHAC index values and the properties of the coal samples. In the determination of the spontaneous combustion liabilities 58 different coal samples were tested with an ignition-temperature test apparatus. Crossing-point temperature tests and differential thermal analysis were conducted simultaneously. The WITS-EHAC index values were contoured on the mine plan to isolate the areas of high risk for top and bottom seams. A higher index value represents an increased liability of a coal to spontaneously combust.

Statistical analyses were carried out to determine the correlation between the WITS-EHAC index values and the coal properties. No significant relationship were found because all the coal samples were of similar rank.

In the second phase of the thesis, the adiabatic calorimeter was used to simulate the ignition temperature

test apparatus. A good relationship between the results of the ignition temperature tests and the adiabatic calorimeter indicated that the frequency factor (from the adiabatic calorimeter) is a strong indicator of a coal to spontaneously combust. The adiabatic calorimeter results confirmed the ignition temperature test results.

DEDICATION

With my sincere loving to my wife : Berrin EROĞLU

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1. INTRODUCTION

1.1. Spontaneous Combustion and Its History

Spontaneous combustion has been a major hazard in the coal mining industry over the centuries, both from safety and economic aspects.

One of the first recorded underground heating was mentioned in the records of Griff Colliery for the year 1604 (MORRIS; 1986).

In 1686 Dr Plott published one of the earliest authoritative discussions on the spontaneous of coal and considered that pyrites probably helped the coal to ignite (quoted by WHITAKER; 1927 and COWARD; 1957).

The term "spontaneous combustion" is defined in THE NEW MERRIAM-WEBSTER POCKET DICTIONARY as " a bursting into flame of combustible material through heat produced within itself by chemical action (as oxidation) ".

During the coalification process there is no oxygen in the environment. When mining activities (underground or open-cast) are applied to a coal body, the freshly broken coal is exposed to the air and begins to react with the oxygen. All coal has a greater or lesser susceptibility

to being oxidised. The oxidation reaction increases with increasing temperature When the heat released by oxidation cannot be dissipated, there is an accumulation of heat which quickens the reaction, possibly causing an ignition. Ventilation rate plays a dominant role in this particular case. A low ventilation rate causes a heat accumulation because of there not being enough ventilation air to dissipate the heat produced. However, a high ventilation rate brings more oxygen into the incident area and allows the reaction to continue.

Different terms have been used by researchers to refer to phenomenon of spontaneous combustion. These include: spontaneous combustion, self-heating, incipent heating, spontaneous heating, spontaneous ignition and autogenous heating (WADE; 1988). In this thesis the terms spontaneous combustion and self-heating are used.

1.2. Hazards of Spontaneous Combustion

The possible consequences of an ignition in an underground mine are (WADE and GOUWS; undated):

a) Death or injury due to fire,
b) Death or injury due to gassing by carbon monoxide if the coal is smouldering,
c) Death or injury due to roof collapse if coal pillars

are consumed by fire,

d) Loss of reserves in the burnt coal,

e) Loss of reserves in the virgin coal left behind due to the need to seal off the site of the incident.

Asphyxiating, toxic and flammable gases are the most serious consequences of an incident of spontaneous combustion. Firstly, there is a danger of suffocation or of poisoning workmen involved in fire-fighting activities because of the asphyxiating or toxic gases. Secondly, there is a danger of an explosion due to the flammable gases (CUDMORE and SANDERS; 1984).

The maximum legal limits of the dangerous gases in South African underground mines are shown in Table 1.1.

Table 1.1 Maximum legal limits of the dangerous gases in coal mines

Gas	Maximum Limit
CH_4	14000 ppm
CO_2	5000 ppm
CO	100 ppm
H_2S	20 ppm
N_xO_y	5 ppm

Carbon monoxide is one of the most hazardous gases in coal mines. It is a colourless, tasteless, odourless and poisonous gas. The oxygen carrying component, haemoglobin, has an affinity for CO which is 300 times greater than the affinity for oxygen. Once saturated by CO, the haemoglobin forms carboxyhaemoglobin and prevents the carrying of oxygen through the body. The symptoms of various blood saturation levels are as in Table 1.2 (GILL AND BROWNING; 1971).

Table 1.2 Effects of blood saturation with CO

Blood Saturation (%)	Symptoms
0-20	No symtoms
20-30	Throobing headache
30-50	Dizziness, nausea, muscular weakness, danger of collapse
Above 50	Unconciousness and dead

Carbon dioxide is the major gaseous product after an actual ignition. It is a colourless and odourless gas. It has a slight acid taste. It is not a poisonous gas but causes death by suffocation. A concentration of 25% carbon dioxide causes death after a short period.

One of the other dangerous gases in underground coal mines is methane. It is colourless, tasteless and odourless. It has a relative density of 0.559 which means that is much lighter than air and may form roof layers. It explodes in the range of 5% to 15% and at 10% it reaches its greatest potential explosive intensity. The amount of flammable gas is not permitted to exceed 1.4% in South African coal mines.

Indications and preventative techniques regarding spontaneous combustion are discussed in Chapter 2.

1.3. Importance to the Mining Industry

The spontaneous combustion of coal in underground collieries has the potential to cause serious loss of life, machinery and coal reserves.

Spontaneous combustion is the major cause of the underground fires in South African collieries. Many other parts of the world, including Turkey, have the same problem. In an accident in the Armutcuk Colliery, Turkey, on the 7th March 1983 an explosion occurred and 102 miners were killed and 86 were injured. The explosion occurred as a result of a fire started by spontaneous combustion (BILGIN; 1985). Also, an explosion caused by

spontaneous combustion claimed 65 lives in the Yeni Celtek Colliery in Turkey in 1990.

Incidents of spontaneous combustion in underground coal mines are likely to increase. Underground coal mining will be more prevalent than open-cast mining in the future because only 4-20 percent of South African coals can be mined using open-cast mining methods (PETRICK et al; 1975). Secondly, the underground methods using total extraction techniques such as pillar extraction and longwalling are becoming more prevalent in areas known to be liable to spontaneous combustion (GOUWS; 1987). It was stated by DE JAGER, in 1983, that more than 40 percent of South Africa's coal reserves are found in seams between 3,5 and 6 metres thick. In order to disperse the larger quantities of methane, dust and heat produced with increased production rates, more ventilation is required. The higher ventilating pressure leads to more ventilation leakage. The increased ventilation leakage enables coal to be oxidised and heat to accumulate, increasing the probability of an incident of spontaneous combustion occurring. With the potential for an increase in the number of incidents, it will benefit mining engineers to know more about this phenomenon in order to determine whether their working areas are liable to spontaneous combustion or not.

1.3.1. Occurrences in Mines

Information relating to the occurrence of underground fires in South African collieries has been obtained from the work of MORRIS and BADENHORST (1987) and HOLDING (1992).

The list of underground fires and ignitions in South African collieries according to the types of the incidents which occurred during the years 1970-1990 is shown in Table 1.3 and Figure 1.1. However, it should be noted that these figures include only the incidents which have progressed to a serious stage and have been reported to the Inspector of Mines. Minor incidents which were treated successfully in their early stages are not reported.

Table 1.4 and Figure 1.2 describes the number of underground fires and incidents per 10 megatons of underground coal production.

During the early 1980's the incidence of spontaneous combustion was low and steady. It showed an increasing trend from 1986 to 1990. In 1990 no incidents of spontaneous combustion were reported. However, as can be seen in Table 1.4 and Figure 1.2, while 3 spontaneous combustion incidents have occurred in 1986 and 1987, 13

Table 1.3 Underground incidents in South African collieries 1970-1990; after HOLDING (1992)

Year	Spon.Com.	Electric.	Flam.gas.	Other	Total
1970	7	3	-	-	10
1971	2	-	1	-	3
1972	3	1	2	2	8
1973	6	1	2	2	11
1974	6	1	-	1	8
1975	6	1	-	1	8
1976	3	2	-	-	5
1977	7	2	1	3	13
1978	3	4	1	-	8
1979	9	5	-	-	14
1980	2	2	2	-	6
1981	4	3	5	1	13
1982	2	10	-	1	13
1983	1	4	2	1	8
1984	5	6	5	1	17
1985	4	10	5	5	24
1986	1	6	10	4	21
1987	2	5	5	5	17
1988	6	7	-	4	17
1989	7	5	4	3	19
1990	-	3	9	4	16

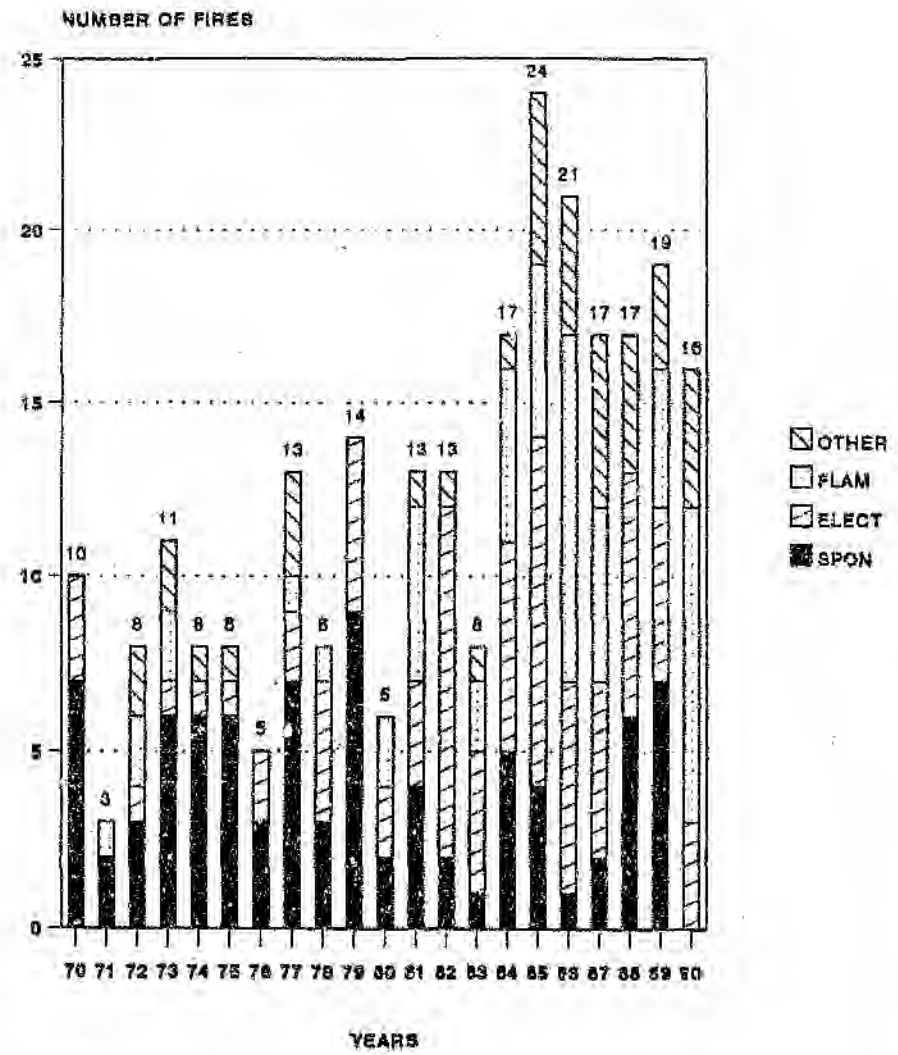

Figure 1.1 Underground incidents in South African collieries

incidents were reported during the next two years.

As can be seen from these figures, spontaneous combustion is still an important problem in South African collieries and should be investigated in detail.

Table 1.4 Underground incidents per 10 megatons; after HOLDING (1992)

Year	Spon.Com.	Electric.	Flam.gas.	Other	Total
1970	1.32	0.57	-	-	1.89
1971	0.38	-	0.19	-	0.57
1972	0.56	0.19	0.38	0.38	1.51
1973	1.09	0.18	0.36	0.36	1.99
1974	1.04	0.17	-	0.17	1.38
1975	1.00	0.17	-	0.17	1.34
1976	0.47	0.31	-	-	0.78
1977	0.99	0.28	0.14	0.42	1.83
1978	0.40	0.54	0.13	-	1.08
1979	1.09	0.60	-	-	1.69
1980	0.24	0.24	0.24	-	0.72
1981	0.45	0.34	0.57	0.11	1.47
1982	0.21	1.07	-	0.11	1.39
1983	0.10	0.40	0.20	0.10	0.80
1984	0.47	0.56	0.47	0.09	1.59
1985	0.34	0.86	0.43	0.43	2.06
1986	0.09	0.52	0.87	0.35	1.83
1987	0.18	0.45	0.45	0.45	1.53
1988	0.55	0.63	-	0.36	1.54
1989	0.64	0.46	0.37	0.27	1.74
1990	-	0.26	0.79	0.35	1.40

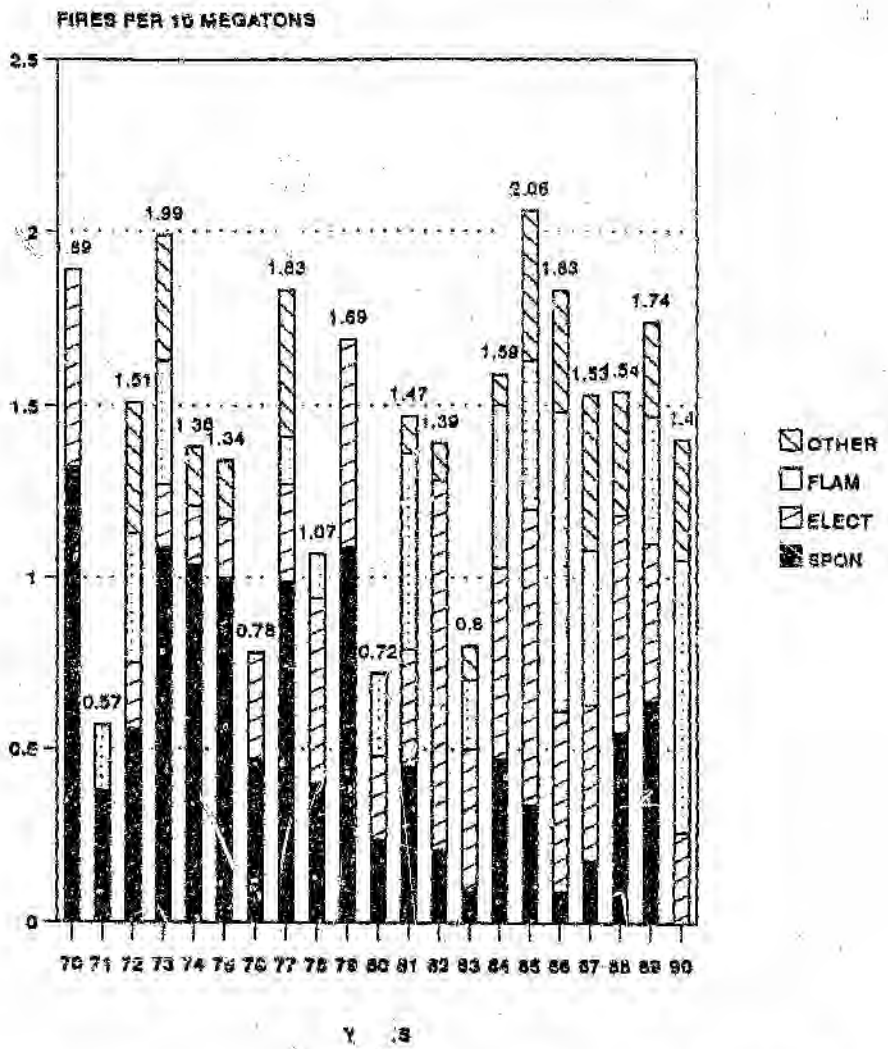

Figure 1.2 Underground incidents per 10 megatons

1.3.2. Storage of Coal

The widespread use of coal and increasing consumption worldwide necessitate large stockpiles of coal close to the big power stations, ports etc.

Oxygen provokes a slow progressive oxidation starting with the external layers of the piles, causing, besides a not easily calculated economic loss, a potential fire hazard resulting from spontaneous combustion. For these reasons, particular attention in the stockpiling of coal is required in order to reduce the risk of deterioration or overall loss to a minimum (DE FAVERI et al.; 1984).

An important factor in the storage of coal, mentioned by Pechin, speaking before the American Institute of Mining Engineers in 1872 is (quoted by PORTER and OVITZ; 1912):

> "Fuel suffers materially by storage; especially with bituminous and semi-bituminous coals is the loss heavy, an exposure of only two weeks causing a loss of carbon to the extend of 10 to 25%."

The most serious problem resulting from self-heating in a coal stock is that it would lead to an outbreak of fire in the stockpile, or worse in a ship's hold.

1.3.2.1. Storage of Coal in Stockpiles

The importance of the storage of coal in stockpiles was realised in the early of 1900's because of the uncertainties of supply due to strike and transportation difficulties (PORTER AND OVITZ; 1912).

Some stockpile tests were carried out by the Bureau of Mines to determine the coal storage problems. Their main conclusions were:

- Do not pile over 12 feet deep,
- If possible, store only lump, and
- Pile so that lump and fine are distributed as evenly as possible.

In 1879, FAYOL found that spontaneous combustion had never occurred in piles less than 2 metres high, while it often occurred in piles higher than 4 metres (quoted by DAVIS and REYNOLDS; 1928 and COWARD; 1957).

In 1911, PARR and KRESSMANN noted that piles of coal 91*2,8*1,8 metres high and 113*12,5*1,8 metres did not fire after standing for a long time, while a pile of 101*24,4*3 metres high took fire in several places.

SONDREAL and ELLMAN published their results about the storage of North Dakota Lignite in 1974. They concluded that care should be taken to avoid segregation of coarse and fine particles in the stockpile because the fines had greater liability for heating and it was easier to achieve required bulk densities when sizes were uniformly distributed.

DIPPENAAR conducted a research program about the design of the stockpiles of Grootegeluk Coal Mine in 1984. Plant discard (waste) posed a problem of spontaneous combustion at the Grootegeluk Coal Mine. Dippenaar proposed that:

- a layer of compacted fly ash be placed on the dump surface,
- the angle of repose of the dump sides be reduced to 18 to 20 degrees,
- the radial stacking method be changed to a rectangular stacking method in order to prevent self-heating of the central pivot section of the dump.

Spontaneous combustion tests (ignition temperature tests and differential thermal analysis) were carried out in the Department of Mining Engineering of the University of the Witwatersrand with the waste and weathered waste (104 days)of Grootegeluk mine. Both samples, taken by the staff of the mine, showed no liability to spontaneous combustion. Spontaneous combustion could have been experienced in the past due to the mistakes in the stockpile dimensions.

COETZEE drew up some do's and don'ts in 1985 about the stockpiling of South African coals. Do's are:

- sweep ground floor of bins and piles before loading or stacking,
- test stacks, piles etc. for temperature,

- test stacks, piles etc. for CO gas,
- test stacks, piles etc. for methane gas,
- transport or load wet coal if possible.

Don'ts are as follows:

- allow segregation of coal sizes,
- blow or allow air to seep through the coal,
- mix coal from different seams,
  coal from different mines,
  new coal with old coal,
  dry coal with wet coal,
  hot coal with cool coal,
- let wind blow onto the sides of stacks or piles.

1.3.2.2. Storage of Coal in Shipping

A fire in a ship's hold is certainly a more hazardous situation than a fire in a stockpile.

SONDREAL and ELLMAN, in 1974, made some general recommendations about the storage of coal in shipping:

- Lignite leaving a drier should be cooled to as low a temperature as practical before shipping.
- Time in transit should be reduced to a minimum.
- The exposure of coal to oxygen should be limited as much as possible.

Washing or screening of coal has been suggested to decrease the risk of self-heating in ships. Coals which have been washed or screened have a smaller number of fine particles and hence a smaller surface area available for the reaction. Coals which have been washed also have the benefit of having contaminants removed and moisture equilibrated before being shipped (RIGSBY; 1983).

Transportation time has an important role, especially for bituminous coals. In general terms, it is said that bituminous coal can normally only be stored for eight to ten weeks without risk of spontaneous combustion(HARRIS; 1985). If there is a danger of exceeding this period, precautionary measures should be taken.

KELLEY (1985) made the following recommendations about the safe shipping of coal:

- coal pile heights of 10 to 20 feet have been recommended, but lesser heights might be advisable for highly susceptible coal,
- the most frequently observed practice that promotes coal self-heating is size segregation, but coal is often segregated by type as well,
- a mixed product for export should be temperature monitored for several hours before leaving port in order to ensure its temperature stability.

If spontaneous combustion was detected in a ship's hold then the ship should sail to the nearest port. Thermocouples should then be inserted into the cargo and nitrogen pumped into the hold. Once the temperature of cargo is at a safe level then, after testing for gas, the hatch may be opened and any damaged coal removed from the ship (HARRIS; 1985).

During the stockpiling and shipping of coal, if sufficient detection and preventative methods are taken, it is possible to avoid or minimise the risk of spontaneous combustion of coal, even in the case of coals with a higher propensity to self-heating.

1.4. Factors Contributing to Spontaneous Combustion

The basic conditions necessary for spontaneous combustion to occur in a coal mine are (FENG, CHAKRAVORTY and COCHRANE; 1973):

a) Coal must be present in a form which can readily oxidize at ambient temperature;
b) Oxygen must be available to support the oxidation process; and
c) The mine environment must favour the accumulation of heat.

Factors contributing to the spontaneous combustion of coal were classified by BHATTACHARYYA in 1982. Spontaneous combustion is said to occur as a result of the aggregate effect of the factors, which are listed below:

a) Coal factor

b) Geological factor

c) Mining factor

Each factor is, in turn, the aggregate effect of a number of sub-factors.

Contributory factors may be classified as being either intrinsic or extrinsic. The mining factor is extrinsic while the other two factors are intrinsic. This means that the mining factor can be controlled and altered to decrease the overall risk of an incident occurring (EROGLU, GOUWS and PHILLIPS; 1991).

The intrinsic and extrinsic factors were summarized as follows (WADE, PHILLIPS & GOUWS; 1986 and WADE;1988):

1- Intrinsic factors

   a) Coal related factors

      - Low rank
      - Rich in reactive macerals
      - High sulphur content
      - High alkaline content

- High porosity
- Easily friable

b) Geologically related factors
- Thick seams
- Highly faulted
- Poor caving characteristics
- High virgin coal temperature
- Outburst prone
- Carbonaceous strata or roof

2- Extrinsic factors

a) Mining related factors
- Accumulation of broken coal
- Stress distribution causing fracture
- Low production rates
- Limited ventilation through worked out areas
- Heat from machines
- Relative humidity differential between air and coal

The total risk of an occurrence of spontaneous combustion is defined by BHATTACHARYYA (1982) as being the product of these factors;

TOTAL RISK=Coal Factor*Geological Factor*Mining Factor.....(1)

It is important to note that no single factor is responsible for spontaneous combustion. Laboratory experiments could identify high risk coals, allowing the total risk factor to be decreased by altering the mining factor. Knowledge of this risk index will enable mining engineers to design with greater emphasis on safety.

1.5. Objectives of the Thesis

The objectives of the research programme for this thesis can be summarized as follows:

1- A literature survey based on;
   a) the history of the spontaneous combustion and its hazards,
   b) the importance to the industry
   c) the influencing factors,
   d) the detection and preventative methods,
   e) the evaluation of the theories of spontaneous combustion
   f) the determination and the selection of the spontaneous combustion testing technique.
2- A laboratory testing programme for the coal samples of Durban Navigation Colliery.
3- Determination of the simple and composite indices of the coal samples.
4- Contouring the top and bottom seam coal samples of

Durban Navigation Colliery to identify the areas having high risk to spontaneously combust.

5- Statistical correlation of the simpl. and composite indices against the ultimate, proximate and petrographic analyses results.

6- Simulation of ignition temperature test apparatus.

2. DETECTION AND PREVENTION OF SPONTANEOUS COMBUSTION

2.1. Indications and Monitoring the Spontaneous Combustion

The early detection of spontaneous combustion in mines is of considerable importance to the mining industry in order to prevent a possible incident. The development of a heating is accompanied by the progressive appearance of haze, sweating of the strata, gob stink, fire stink, smoke and fire if the heating is not stopped at some stage.

GRAHAM suggested that the chemical analysis of return air could give valuable information of incipent heatings in mines and developed a ratio for this purpose. Graham's ratio, CO/CO_2, has been used over many years in mines where heatings were suspected (quoted by COWARD; 1957).

In the 1960's, unsaturated hydrocarbons were used for the early detection of spontaneous combustion. It was suggested that the appearance of ethylene is one of the earliest signs of a heating. The concentration of ethylene increased with the CO/CO_2 deficiency ratio generally. When the ratio increased to three or four times the normal for the district, propylene was detected, and when it increased to five to seven times

the normal, acetylene was detected (PURSALL and GHOSH; 1965).

CHAMBERLAIN and HALL, in 1973, used the tube bundle system for the detection of underground fires. They mentioned that more than twenty installations were operating and their detection record had been outstandingly successful.

The rate of carbon monoxide production is believed to be the best parameter to use for the early detection of underground heating (CRIDDLE; 1961, CHAMBERLAIN; 1974, HERTZBERG, LITTON & GARLOFF; 1977 and BARNES, JOUBERT & BADENHORST; 1977).

Mining Research Laboratories (CANMET), in Canada, developed a system suitable for the early detection of heating in the hydraulic mines. Field and laboratory tests indicted that infra-red technology would prove to be a powerful system in the early detection of heating (CHAKRAVORTY and FENG; 1978).

CHAKRAVORTY and WOOLF (undated) believed that two systems were likely to have potential for the early detection of heating in coal mines. These were:

i) Continuous monitoring of carbon monoxide in the mine air; and

ii) Measurement of temperature changes using infra-red techniques.

They recommended that a combination of CO monitoring and an infra-red detection system would prove useful for the early detection of spontaneous combustion.

In general, methods of detection of fires and underground heatings were classified as follows (FAUCONNIER; 1981):

1- Non-instrumental detection
   - a- Smoke
   - b- Haze
   - c- Sweating or condensation on cool surfaces
   - d- Heat radiated from hot surfaces
   - e- Characteristic smell
2- Instrumental detection
   - a- Chemical detectors (detector tubes or meters)
   - b- Physicochemical detectors
   - c- Physical detectors

The types of early detection systems being used in South African collieries are (FAUCONNIER; 1981):

1- Fire patrols
2- Systematic gas sampling
3- Continuous instrumental monitoring
   - a- Tube bundle system
   - b- Telemetric systems, and
   - c- Upcast-shaft monitors.

2.2. Precautionary Measures and Prevention of Spontaneous Combustion

The application of precautionary measures can make it possible to decrease the possibility of a coal body undergoing self-heating, especially in mines known to be liable to spontaneous combustion. Even if an underground incident occurred, the hazard can be limited with different prevention methods.

COWARD (1957) mentioned some recommendations about the precautionary measures to prevent a possible incident of spontaneous combustion. He concluded that;

- as regards the access of air to coaly matter, roadside packs should be tight,
- roof and floor breaks that can admit air to the goaf or to ribs or buried pillars of coal should be reduced to a minimum,
- solid stowing with incombustible materials may often be very advantageous and even indispensable with the most oxidizable coals;
- timber should not be left in the goaf, for it helps to maintain small passages for air;
- the water gauge between intake and return round a goaf should be kept low by the construction of wide roads and by the avoidance of obstructions such as falls;
- in special circumstances such as occur particularly in

steep seams firedamp may be allowed to accumulate in the goaf so as to exclude air.

BERKOWITZ and SPEIGHT (1973) investigated the prevention of spontaneous heating in stockpiles by low temperature immersion carbonization of coal. Their laboratory studies indicated that autogenous heating of low rank coal could be prevented by a prior treatment which involved immersing the coal for short periods (5-15 min.) in an inert liquid at temperatures between ≈350 and 400°C.

In the event of a combustion, a reduction in the ventilation feeding would normally have the immediate effect of reducing the activity of the heating. In the case of local spontaneous combustion at a roadside, it is important, first of all, to seal and fireproof the sides of the roadway before injecting a grout into the affected area (THORP; 1974).

In 1980, nitrogen was injected into the waste of a working face in Daw Mill Colliery, in England, to control a spontaneous combustion heating. The process was successful and the injection of nitrogen prevented the district from being sealed off. But it should not be forgotten that nitrogen does not extinguish a heating, it only makes it dormat and buys time to do other additional measures (HARRIS; 1981).

3. THEORIES OF SPONTANEOUS COMBUSTION

Various spontaneous combustion theories have been advanced and discussed by researchers over the centuries. The theories are namely:

- The pyrite theory,
- The bacterial theory,
- The oxidation theory, and
- The humidity theory

This section will deal with each of these spontaneous combustion theories in turn.

3.1. The Pyrite Theory

The pyrite theory had begun to be questioned in the middle of the nineteenth century. In 1848, DE LA BECHE and PLAYFAIR (quoted by COWARD; 1957) deduced that when iron pyrites was present in considerable quantity in a coal still changing under the action of the atmosphere, a second powerful heating was introduced, and both acting together could produce what is termed spontaneous combustion.

In 1870, RICHTER (quoted by COWARD; 1957) argued that the high pyrites content in an Upper Silesian coal would heat too slowly and too little to bring about spontaneous

combustion of coal. He suggested that in spontaneous combustion of coal there was no correlation with the pyrite content.

In 1875, PERCY (quoted by COWARD; 1957) mentioned that the Ten - Yard coal of South Staffordshire, which was very liable to spontaneous combustion, contained only a small proportion of pyrites. He would not assert that pyrites might not, when present in considerable quantity, develop sufficienc heat to set coal on fire.

On the other hand, in 1866, LIEBIG (quoted by COWARD; 1957) gave the weight of his great authority as a chemist to the pyrite theory, but apparently made no experimental investigation on his own on the subject.

In 1879-80, KIMBALL (quoted by COWARD; 1957) pointed out that if the pyrites were locally more concentrated, the temperature produced would be higher. He seems also to have been the first to call attention to the difference between marcasite and cubical pyrites, the former being the less stable form of FeS_2.

In 1880-81, HAEDICKE (quoted by COWARD; 1957) succeeded in igniting small quantities of precipitated ferrous sulphate in oxygen at 200°C, and suggested that the oxygen absorbed by the coaly matter itself would aid the

ignition of pyrites in the coal.

The pyrite theory can be shown with this well known chemical reaction (Equation 2):

$$2FeS_2 + 7O_2 + 2H_2O = 2H_2SO_4 + 2FeSO_4 \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

In 1915-16, WINMILL mentioned that this reaction generated heat at a rate of 4,3 calories per cubic centimetre of oxygen absorbed. Since the heat production was so large, it was important to know the rate of the oxidation of pyrites, as if this rate approaches that of coal, the former would evidently be a possible cause of heating.

In 1916, BRAKELEY (quoted by COWARD; 1957) found that iron pyrites had a minor effect on the oxidation of coal.

In 1922, ERDMANN (quoted by COWARD; 1957) pointed out that even 10% of marcasite added to coal did not affect its ignition temperature.

GRAHAM and JONES concluded in 1924-25 (quoted by COWARD; 1957) that spontaneous combustion in South Wales was initiated by finely divided pyrites in normal seams, but in thick seams by broken coal assisted by pyrites.

In 1912, PORTER and OVITZ indicated that sulphur content contributed very little to spontaneous combustion.

LI and PARR, in 1926, concluded that under suitable conditions the pyrites in coal would oxidise rapidly and might be a dominating factor in certain cases for the self-heating of coal. Marcasite and pyrite oxidise at about the same rate, but the former breaks down more easily producing fine particles, thus facilitating its oxidation.

In 1951, SCHEIN (quoted by COWARD; 1957) tested certain Commonwealth coals that were almost free of sulphur but were liable to spontaneous combustion. He also tested coal containing 10% of colloidally dispersed inorganic sulphur that was found to be safe.

In conclusion, pyrite cannot be the major causative factor of spontaneous combustion unless:
- it is present in large quantities
- it is in a finely divided state

Something else must be the cause, with pyrite possibly being a contributory factor.

3.2. The Bacterial Theory

One of the first studies concerning the effect of bacteria on the spontaneous combustion of coal was conducted by POTTER in 1908 (quoted by COWARD; 1957). He isolated a pure culture of diplococcus from a soil extract cultivated on sterilised charcoal. He inoculated coal and charcoal with it and found that the inoculated charcoal maintained a temperature 0.19°C above that of a sterilised sample for a period of one week. Potter mentioned that the inoculated coal developed a similar small heating when compared with a sterilised coal sample.

In 1910, GALLE (quoted by COWARD; 1957) grew seven different bacteria from five coal samples. He identified four species of bacteria, namely; Bacillus subtilis, Bacillus nacreceus, Bacillus mesentericus and Bacillus pseudosubtilis. Heat developed by the effect of these bacteria on coal produced increases of temperature of 0.28 to 1.9°C. He suggested that the bacteria might play an important role in the initiation of the spontaneous combustion of coal but that it could not be the major effect.

In 1914-15, GRAHAM (quoted by COWARD; 1957) worked with two coals. One of them was sterilised while the other was

unsterilised. He oxidised two of them and showed that the bacteria did not affect the oxidation rate of the unsterilised coal and the absorption of oxygen was independent of bacterial activity. In the same year, WINMILL (quoted by COWARD; 1957) compared the decreasing rate of oxygen absorption by coal with the increasing rate to be expected if the action was bacterial. He suggested that it was impossible to be a rapid oxidation of some substance produced by bacteria which are no longer active at high temperatures.

In 1911, MIEHE (quoted by HALDANE and MAKGILL; 1923) investigated the spontaneous combustion of hay. He showed that the heating was mainly due to the action of two species of bacteria, namely; Bacillus coli and Bacillus colefactor.

The former bacterium grew at ordinary temperatures but died at about 42°C. The second one did not grow at all at less than 30°C, and flourished at 40 to 60°C and could still live up to 70°C. Then the hay was sterilised or treated with an antiseptic, no heating occurred in his apparatus. Above the temperatures at which the bacteria die, the temperature continued to rise as a result of the hay becoming subject to a purely chemical oxidation.

In 1927, FUCHS (quoted by COWARD; 1957) concluded that the bacteria were able to live on different kinds of coal causing a slight evolution of gas and a slight rise of temperature.

The similar findings of the researchers is that the bacterial activity is generally increased with temperatu e until a temperature is reached at which the bacteria die.

Since the acceptability of the oxidation theory, as with the pyrite theory, the bacterial theory has lost its importance in the research of the spontaneous combustion of coal.

3.3. The Oxidation Theory

The importance of oxidation in the spontaneous combustion of coal was mentioned in the late 19th century.

In 1868, RICHTERS (quoted by GUNEY; 1968) discovered that a dry coal sample exposed to air at 180 to 200°C, increased its weight for some hours, but after 20 hours it began to lose weight. In the beginning of his research, he suggested that the pyrites was the major cause of self-heating. From later experiments he

concluded that surface condensation of oxygen was the first stage in the oxidation of coal, and that oxidation itself must be the cause.

In 1879, FAYOL (quoted by GUNEY; 1968) pointed out that the ignition of coal was due to a reaction between the oxygen of the air and the organic constituents of coal, which produced heat. The magnitude of this heat was concluded to dependent on the physical conditions present.

Several researchers mentioned the heat liberated at low temperature oxidation. In 1912-13 LAMPLOUGH and HILL (quoted by GUNEY; 1968) obtained a value of 3.3 cal/ml of oxygen consumed for a -0.5 mm coal. WINMILL, in 1914-15 (quoted by GUNEY; 1968), gave a lower value of 2.1 cal/ml of oxygen consumed and concluded that Lamplough and Hill had underestimated several factors thus obtaining results that were too high. In 1944, SCOTT (quoted by GUNEY;1968) found 3.1 to 4.4 cal/ml of oxygen consumed from the changes in weight and calorific value.

In 1988, WADE pointed out the intrinsic and extrinsic factors effecting the oxygen adsorption of coal (Table 3.1).

Table 3.1 Parameters effecting the oxygen adsorption of coal; after WADE (1988)

Parameter	Effect on oxidation rate as parameter increases
Particle size	Decreases
Temperature	Increases
Wetness	Increases
Previous heating	Increases
Oxygen partial pressure	Increases
Volatile content	Increases
Rank	Decreases
Carbon content	Decreases
Oxygen content	Increases
Inherent moisture	Increases

The mechanism of coal oxidation includes four stages:

1- Coal + oxygen = physical adsorption of oxygen on coal surface

2- Saturated coal + oxygen = chemisorption to coal-oxygen + heat

3- Coal-oxygen complex + oxygen = CO_2 + CO + H_2O + aromatics + heat

4- Aromatics + coal + heat = ignition

The oxidation theory is supported by many researchers.

3.4. The Humidity Theory

The effect of moisture on the oxidation of coal has been investigated by various researchers.

In 1915-1916 WINMILL (quoted by GUNEY; 1971) found that a sample of dry coal absorbed oxygen more slowly than a moist coal.

In 1926, DAVIS and BYRNE, however, indicated that it was better to store the coal moist than dry since pores in the coal which were filled with moisture had no space for adsorption of oxygen.

ROSIN, in 1928 (quoted by GUNEY; 1968), noted that spontaneous combustion took place in coal dumps particularly frequently after warm rainy weather.

In 1950-51, BERKOWITZ and SCHEIN postulated that whilst the normal heat of oxidation would cause a limited temperature rise in small and medium piles, ignition

could only occur in these piles if they were firstly partially dried by exposure to dry weather and subsequently rapidly wetted. They proposed that the heat of wetting (or the heat of condensation) would act as an important "trigger". They also mentioned that while the heat of wetting would decrease as the initial moisture content of coal increase, it remained large enough to influence oxidation velocity of any coal whose moisture content had been depressed below its normal equilibrium value by a succession of warm dry days.

In 1954, PETSCHUK and MAJEWSKAYA, and in 1956, BUDRYK (quoted by GUNEY; 1971) reported that the number of underground fires directly caused by spontaneous heating increased after the drainage of pits or levels which had been flooded or waterlogged.

In 1956, STOTT (quoted by CUDMORE and SANDERS; 1984) designed a micro-calorimeter and showed that the quantity of heat liberated by the atmospheric oxidation of coal was much less than the quantity of heat required to remove water from the coal. He concluded that if the evaporation of water can be induced at the site of a heating, then the temperature of heating would decrease.

In 1969, BHATTACHARYYA, HODGES and HINSLEY concluded that an increase in the air humidity could cause heating in

coal. They pointed out that since the sorption of water raised the coal temperature, and hence the oxidation reaction, it would be a serious danger. If water vapour could be desorbed from the coal, the risk of self-heating would be reduced.

In 1971, GUNEY suggested that in addition to a temperature increase in coal due to self oxidation, the spontaneous combustion of coal was also accelerated by wetting with water.

In 1979, BERKOWITZ pointed out that ignition occurred generally at the interface between dry and wet coal. He proposed that heat associated with wetting was proportional to the extent of the wetted surface and was a functio.l of the capacity moisture content of the coal.

4. SPONTANEOUS COMBUSTION TESTING TECHNIQUES

Various testing techniques have been used by the researchers to determine the liability of a coal to spontaneous combustion. In the literature, the spontaneous combustion testing techniques are categorized mainly into six groups:

- Direct observations
- Chemical tests
- Oxygen sorption rates
- Isothermal calorimetry
- Adiabatic calorimetry, and
- Ignition-temperature determinations

The former two techniques, direct observations and chemical tests, have lost their importance and application in research fields because of the difficulties of experimental control and providing the incorrect results. The advantages and disadvantages of the remaining four techniques are shown in Table 4.1.

4.1. Direct Observations

In 1879, FAYOL (quoted by DAVIS and REYNOLDS; 1928, and COWARD; 1957) investigated 25 kinds of coal, ranging

Table 4.1 Comparison of spontaneous combustion tests

Method	Advantages	Disadvantages
Ignition temperatures	-Rapid tests -Simple, cheap apparatus	-Crossing-point artificial -Difficult to recognise ignition -Insulation characteristics not similar to practical situation
Adiabatic calorimetry	-Duplicates practical insulation characteristic	-Lengthy tests -Apparatus costly, difficult to make and calibrate
Isothermal calorimetry	-Negates variability of rate of reaction with temperature	-Doesn't consider self-magnifying effect of increased temperature -Difficult to maintain true isothermal conditions
Oxygen sorption	-Simple, cheap apparatus -Multiple simultaneous tests are possible	-Rate of oxygen consumption *assumed* indicative of heat generation

from anthracite to lignite. His important conclusions were:

- Whilst coal over 20 mm diameter did not heat, under 20 mm heated and fired spontaneously.

- Raw coal dust was more inflammable than washed coal dust.
- Spontaneous combustion had never occurred in piles less than 2 meters high while it often occurred in piles higher than 4 meters.
- Atmospheric influences had little or no influence on the progress of oxidation.
- Coals were inflammable in the following order:
    - Lignite (most flammable)
    - Gas coal
    - Coking coal
    - Anthracite
- The most favorable conditions for spontaneous combustion to occur were a mixture of coal fragments and dust of large mass and a limited quantity of air.

Unfavorable conditions were encountered with lump coals, a low temperature, small mass and either exclusion of air or very good through ventilation.

In 1909, THREFALL (quoted by COWARD; 1957) filled two cubical bins with slack. The samples in one bin were wetted during the loading. The wet bin had a lower initial temperature than the dry bin due to the cooling by the spray used to wet the coal. It is mentioned that in about 1 month the wet bin had become as hot as the dry bin. The temperature in dry bin rose gradually at first

and then more rapidly. At the end of six weeks the dry bin reached 135°C, while the wet coal had not yet risen to 50°C.

In 1910, PARR and KRESSMANN (quoted by COWARD; 1957) noted that piles of coal 91*2,8*1,8 metres high and 113*12,5*1,8 metres did not fire after standing for a long time, while a pile of 101*24,4*3 metres high took fire in several places.

In 1926, the SAFETY IN MINE RESEARCH BOARD (quoted by COWARD; 1957) sealed a pile of coal during tests and found that a fire might occur again after having been apparently suffocated if air leakage allowed the oxygen content of the atmosphere in the chamber to rise to as little as 3 per cent.

In 1931-32, GRAHAM and REYNOLDS (quoted by COWARD; 1957) found that a pile of coal that contains less dust is more likely to self-heat, and concluded that the better ventilation of coal, due to the spaces not being choked was mainly responsible for the heat.

In 1933-34, MASON and TIDESWELL (quoted by COWARD; 1957) carried out their experiments directly in a gob-fire chamber. They fed air at 35°C into the chamber for 7 months. They measured no heating but a little carbon

monoxide. The temperature of the air was raised to about 68°C. No smell was detected in the return air for 12 days. There was, however, a definite rise in the amount of carbon monoxide produced. They detected a sweetish odour in the return air and the start of more rapid heating on the thirteenth day. The next day smoke was evolved and one of the thermometers recorded a temperature 650°C. The chamber was sealed off and 5 hours later an explosion occurred.

In 1985, CHAUVIN, LODEL and PHILIPPE conducted spontaneous combustion experiments in a test installation. They used two criteria to classify the coals according to their spontaneous combustion liability:

- the minimum temperature that gives rise to self-heating under the heat,
- the time necessary for the increase in temperature to surpass 40°C.

They mentioned that the lower the experimental result, the more liable the coal to spontaneously combust.

Direct observations are the most absolute technique of determining the physical and chemical changes in a coal liable to spontaneous combustion. In this method, experiments are carried out with bulky samples. There must be no oxidation before the experiment. While placing

the coal in the experimental area it is not possible to prevent the oxidation of coal and, hence, to obtain real results. Other disadvantages of this method are:

- it is very difficult to control the behaviour of the sample, which can end up with a fire or uncontrolled combustion, and
- it takes a long time, up to 60 days.

4.2. Chemical Tests

Chemical test studies were being used to investigate spontaneous combustion by the end of the 1800's.

In 1899, FISCHER (quoted by DAVIS and REYNOLDS; 1928 and COWARD; 1957) proposed to compare the reactivities of coal. He found that coals which had been exposed to air for a few hours at about 130°C absorbed much less bromine than the unexposed coals. He suggested the self-heating of coal was due largely to unsaturated compounds which were tested for by a bromine-absorption method. He concluded that a coal contained two classes of compounds. The first one was the compound that was oxidized by oxygen with the evolution of water and carbon dioxide and a decrease in weight. The second one was the unsaturated compound which absorbed oxygen with an increase in weight. He believed that the reaction in the

unsaturated compound was faster than the other compound and much more important in spontaneous combustion.

In 1908, DENNSTEDT and BÜNZ (quoted by COWARD; 1957) used iodine instead of bromine in experiments and concluded that the iodine-absorption value was a measure of liability to spontaneous combustion. They pointed out that the unsaturated residue left after the extraction of coal with pyridine was responsible for the spontaneous combustion of coal.

In 1915, NUBLING and WANNER (quoted by DAVIS and REYNOLDS; 1928 and COWARD; 1957) extracted three coals with pyridine. They determined that the ignition temperature of pyridine-extract was lower, and that of the residues higher than the ignition temperature of the raw coal. The residues absorbed more bromine than the raw coal; while none of the extracts absorbed bromine.

In 1917-18, GRAHAM and HILL (quoted by COWARD; 1957) supported Dennstedt and Bünz's conclusion. They found that a pyridine-extract absorbed almost no oxygen at 30 and 90°C, whereas the residue was as absorbent as the original coal.

In 1920, GODCHOT (quoted by DAVIS and REYNOLDS; 1928) found that both the residue and the extract absorbed

oxygen at 100°C and showed an increase in weight, much the same as the original coal.

In 1926, SINKINSON and TURNER used dry and saturated carbon dioxide to determine the influence of moisture on the temperature rise. They concluded that the moisture, in the presence of carbon dioxide, played a part in influencing the rise in temperature. They found that:

- The natural charcoal content of high-grade coals has a greater capacity for adsorption of carbon dioxide than the charcoal of bituminous coal.
- Adsorption of carbon dioxide produce greater elevations of temperature than those obtained with pure oxygen under similar conditions.
- Carbon dioxide plays an important role in spontaneous combustion, by producing an immediate rise in temperature through the adsorptive energy between the coal and the gas.

As mentioned by the researchers, if carbon dioxide was a major factor in self-heating, anthracite must be more liable to spontaneous combustion than the bituminous coal because of having a greater capacity for adsorption of carbon dioxide. This phenomeon can not be accepted because of it is known that bituminous coals are more prone to self-heating than antracites.

In 1926, DAVIS and REYNOLDS (quoted by DAVIS and REYNOLDS; 1928) conducted iodine-absoption tests of the solid and oily bitumens, and insoluble portions of Utah coal. The results obtained did not agree with the rates of oxidation as measured directly.

In 1928, FRANCIS (quoted by DAVIS and REYNOLDS; 1928) summarized the results of his, as yet, uncompleted work for Davis and Reynolds in a letter. He concluded that of the fractions of coal which may be obtained by the solvent-extraction of pyridine and chloroform, the most easy to oxidize were the gamma compounds (soluble in pyridine and chloroform).

4.3. Oxygen Sorption Rates

RICHTER, in 1868 (quoted by FRANCIS and WHEELER; 1926), was one of the first researchers to mention the importance of oxidation of coal. He found that:

- Coal absorbs oxygen, part of which enters into combination with the coal substance, increasing its weight, whilst the other part forms carbon dioxide and water;
- The absorption of oxygen is continuous. There is a rapid initial rate of absorption, but this quickly gives place to a long-continued slow absorption; and

- The oxidation is accompanied by the production of heat and is accelerated by increased temperature.

In 1917, KATZ and PORTER (quoted by DAVIS and REYNOLDS; 1928, COWARD; 1957 and WADE;1988) compared the oxidation rates of Pittsburgh and Illinois coals at 26°C. They conducted the tests simultaneously with wet and dry samples of the same coal. They found that the Pittsburgh coal absorbed oxygen more rapidly when moist than when dry, whilst the test produced exactly the opposite result of the Illinois coal.

TIDESWELL and WHEELER, in 1920, concluded the following:

- Bright ingredients of coal, (vitrain and clarain) show greater ability to oxidize and to ignite than the dull durain; and
- The influence of fusain is uncertain. Fusain has an influence in determining the actual ignition of the coal after self-heating has begun; but it is conceivable that the rapid absorption of oxygen by fusain at low temperatures may be attended by a sufficient evolution of heat to raise appreciably the temperature of the main mass of the coal, thereby causing the most inflammable ingredient (vitrain) to react more readily with oxygen.

In 1922, SCHRADER moistened each sample with 5N sodium hydroxide solution. He exposed the samples to oxygen and noted the absorption. He found for the bituminous coal that their oxygen absorption increased as their geological age decreased. In other words, the oxygen absorption is inversely proportional to the rank of the coal.

In 1923, GRAHAM investigated the absorption of oxygen by fusain. He pointed out that fusain samples absorbed oxygen at a considerable slower rate than a raw coal liable to spontaneous combustion.

GRAHAM and JONES, in 1924, carried out tests to determine the absorption of oxygen by coal. They measured the rate of absorption of oxygen periodically by noting the rate of air flow and by analyzing samples of the air current. They concluded that spontaneous combustion depends more upon physical factors. These factors include the thickness of the seam, the mining method, and the friable nature of the coal.

In 1925, TIDESWELL and WHEELER circulated oxygen in a closed system through coal or fusain which was packed into a tube and maintained at a constant temperature. They calculated the absorption of oxygen from the reduction of pressure. They found that the oxidisability

of fusain increased less rapidly with temperature than that of the coal. In the same year PARR and MILLER (quoted by COWARD; 1957) exposed small samples of Illinois coals to oxygen at 25 to 140°C for about 40 days and mentioned that their results confirm the great influence or temperature upon the rate of oxidation.

In 1952, SEVENSTER carried out tests to determine the oxygen absorption characteristics of South African coals. He pointed out that a relationship existed between the moisture content of the coal and the volume of oxygen consumed. He also mentioned that within certain limits, the absorptive capacity of the coals tested were dependent upon their rank. He established no relationship between the oxygen consumption and the time. He concluded that any pyrites present in the coal played no appreciable part in the absorption of oxygen.

In 1964, CARPENTER and GIDDINGS investigated the effect of time, temperature and coal rank on the rate of oxygen consumption. They summarized their findings as follows:

- The oxidation proceeds in stages in which the rate of oxygen consumption remains constant, the initial stage having the highest oxidation rate with each succeeding stage showing progressively lower rates;
- The rate of oxidation increases with an increase in temperature, and

- The rate of oxidation decreases with an increase in rank of coal.

In 1967, VARGA pointed out that the rate of oxygen adsorption was much higher than the rate of carbon dioxide desorption and there could be no connection between the carbon dioxide volume of the atmosphere and the amount of heat produced.

In 1978, SCHMELING, KING and SCHMIDT-COLLERUS developed the S-index which reflects the liability of the coal to spontaneous combust. Heat was supplied to an oil bath at a rate of 25°C per hour. A coal sample of 100 grams was heated to 200°C or higher over a period 8 hours. Temperatures of the oil bath and coal were recorded every 30 minutes. Gas chromatography was used to analyse the effluent gases. They mentioned that it was rapid and sensitive to low concentration of the constituent gases. They determined the spontaneous combustion liability S-index for the coal samples using the following equation (Equation 3):

$$S = \frac{h_1 + h_2}{2}(x_1) + \frac{h_2 + h_3}{2}(x_2) \quad \ldots\ldots\ldots\ldots\ldots (3)$$

where: S = spontaneous combustion liability index

h_1 = oxygen deficiency at 125°C

h_2 = oxygen deficiency at 150°C

h_3 = oxygen deficiency at 175°C

x_1 = increase of CO_2 between 125 and 150°C

x_2 = increase of CO_2 between 150 and 175°C

They mentioned that the S-index values ranged from 13 to 47 and values greater than 30 were considered to be in dangerous limits.

In 1985, FENG researched the spontaneous combustion liabilities of Canadian coals. He pointed out that coal samples having high CO/O_2 ratios were found to have self-heating problems. He concluded that the CO/O_2 ratio obtained was the best indicator of susceptibility to spontaneous combustion. The relative susceptibility of spontaneous combustion could also be predicted by the oxygen/carbon ratio in the coal, obtained from ultimate analysis. Coal with a higher methane content would have a lower CO/O_2 ratio, which in turn indicated that a lower susceptibility to spontaneous combustion.

Oxygen sorption tests have been used and are still being used in tests to determine the spontaneous combustion propensity of coal. In this method it is possible to maintain a simple cheap setup and to conduct multiple simultaneous tests although the tests are somewhat lenghty.

4.4. Isothermal Calorimetry

In this testing technique, a coal sample is placed in a bath which allows isothermal conditions to be maintained. The temperature changes exhibited by the coal sample as a result of oxidation, wetting or drying are measured by thermocouples. The measured rate of heat generation is taken to be indicative of the combustibility of the coal sample.

A typical isothermal calorimeter is shown in Figure 4.1.

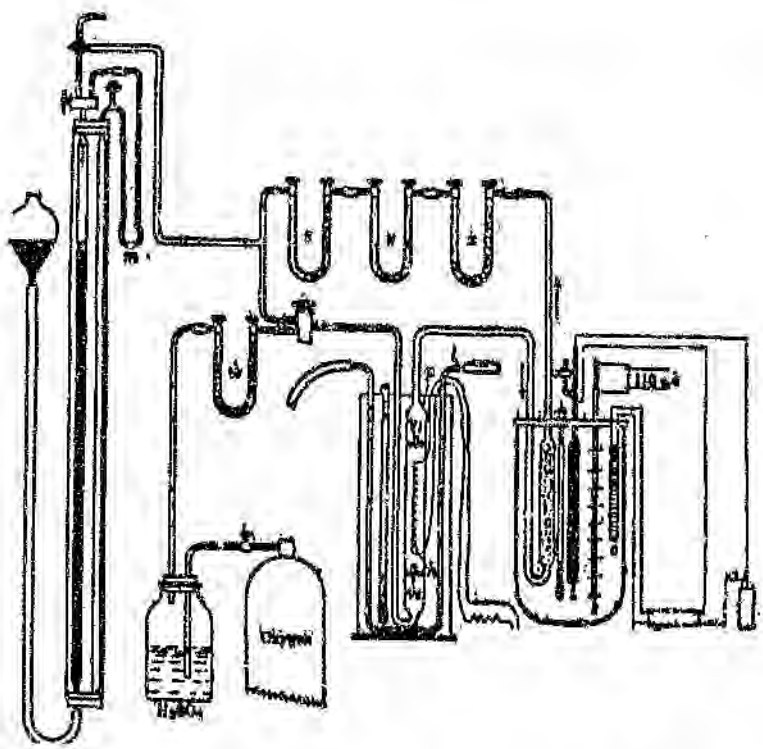

Figure 4.1 Diagram of a typical isothermal calorimeter; after DAVIS and REYNOLDS (1928)

In 1954, VAN DOORNUM conducted experiments with a calorimeter under very nearly isothermal conditions. The calorimeter was a cylindrical aluminium block containing 19 holes. For the tests he charged these holes with 1 kg of coal. He concluded that the intensity of the spontaneous heating process increases with temperature and decreases with time. He found that Equation 4 represented the rate of heat generation for the purpose of computing the temperature rise:

$$q = \Sigma_i A_i \exp (a_i v - a_i t) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

where; q = rate of heat generation ($cal/cm^3/hr$)

A_i = rate of heat generation ($cal/cm^3/hr$)

a_i = temperature coefficient ($°C^{-1}$)

v = temperature rise above any convenient reference temperature (°C)

t = time (hr)

In 1956-60, STOTT developed an isothermal micro-calorimeter which could be used over a wide range of temperatures to measure the amount of heat generated by oxidizing coal. His apparatus was sensitive enough to measure the rate of heat evolution caused by the oxidation of coal and was also able to measure the amount of heat absorbed as moisture was driven from the coal. In his apparatus, a reasonable flow of gas could be passed

through the calorimeter without sacrificing sensitivity. The sensitivity was independent of the specific heat of the reacting materials. During his experiments, sub-bituminous coals always showed a greater rate of heat loss by evaporation of moisture in a dry air stream than their rate of heat generation by oxidation.

In 1964, HODGES and HINSLEY showed that when coals were oxidized with moisture-saturated oxygen the rise in temperature of a coal mass was higher than when it was oxidized with dry oxygen.

HODGES and ACHERJEE (1964) developed a micro-calorimeter to measure the small amounts of heat, (as small as 10^{-1}cal/hr) generated during the low temperature coal oxidation process, directly. They described the advantages of the calorimeter as follows:

(i) It was capable of measuring the rate of as well as the total heat output.

(ii) It could, with a suitable recording instrument, record thermal outputs of less than 10^{-1}cal/hr.

(iii) It was suitable for studying slow temperature process, and for sorption studies.

(iv) It was easy to operate

(v) A reasonable flow of gas could be passed through the calorimeter without sacrificing sensitivity.

(vi) The sensitivity was independent of the specific

heat of the reacting materials.

They carried out the tests on 4 gram samples ground to pass through 72 mesh (-212 microns). The temperature of oxidation was maintained at about 30°C and four types of experiments were conducted:

(1) Oxidation of dry coal with dry air

(2) Oxidation of dry coal with moisture saturated air

(3) Oxidation of moist coal with moisture saturated air

(4) Oxidation of moist coal with dry air

They pointed out that the heat output and the rate of heat released was largest for dry coal and saturated air.

BHATTACHARYYA, HODGES and HINSLEY, in 1969, measured the rate of heat change in coal by means of a calorimeter. They concluded that the danger of spontaneous heating becomes greater with the increase in the water vapour content of the air. In mines the ventilation takes up moisture from several sources. By reducing the artificial sources of moisture the air would tend to desorb water vapour from the coal seam and surrounding strata producing a cooling effect, and thus resulting in conditions less favourable to the development of heatings. They established that the role of humidity in the problem of spontaneous heating of coal was significant in the initial stages of the process and an

increase in the humidity could cause heating in coal. The sorption of water raises the coal temperature to an appreciable degree, and the oxidation reaction may then be a serious danger. On the other hand, by maintaining a condition when water vapour can be desorbed from the coal, the risk of heatings occurring is reduced.

In 1970, BHATTACHARYYA carried out the oxidation tests under isothermal conditions. The results he got showed that the presence of moisture may not necessarily promote the generation of heat for all types of coal. He published three papers about the effect of oxidation on spontaneous combustion between 1970 and 1972. His experiments showed that at low temperatures the desorption of water from a moist coal to its surroundings has an inhibiting effect on its self-ignition. As long as the partial pressure of water vapour in the air is less than that on the coal surface, the chance of self-heating of the coal is lower. In practice, by keeping the ventilation air in a mine dry, the danger of spontaneous heating of the coal can be reduced.

4.5. Adiabatic Calorimetry

In this testing technique, the sample is placed in an sealed container or bath and is heated to a certain

temperature. When the air or oxygen is added, the temperature of coal rises. Heat losses to the surroundings are kept to a minimum by ensuring that the temperature of the bath is kept equal to that of the coal. The change in the temperature of the coal in a given time, the time needed to reach a preselected temperature, or the amount of heat generated per unit of time is used to evaluate the spontaneous combustion liability of the coal. A typical adiabatic calorimeter design is shown in Figure 4.2.

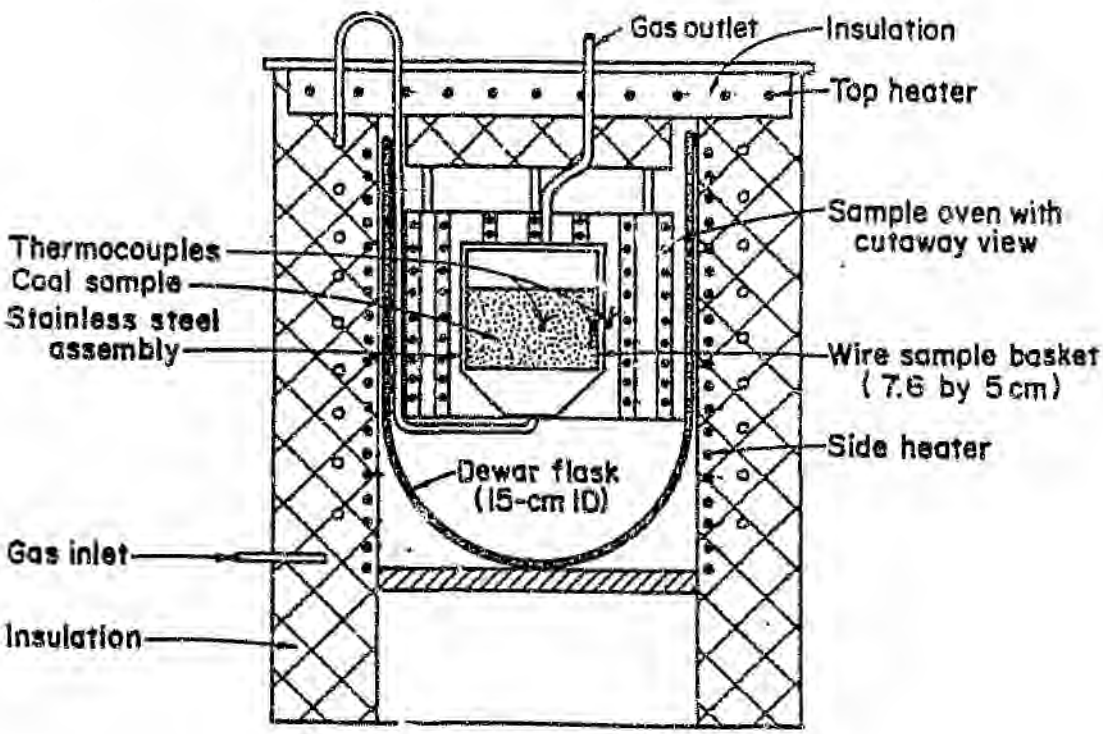

Figure 4.2 Adiabatic calorimeter design; after SMITH, MIRON and LAZZARA (1988)

WINMILL (1914) (quoted by DAVIS and REYNOLDS; 1928 and COWARD; 1957) was one of the first known researchers to use adiabatic calorimetry to investigate the self-heating of coal. His apparatus consisted of an oxidation tube inclosed in a vacuum flask which was immersed in a liquid bath. A temperature control system maintained the temperature of the bath to within 0.2°C of the temperature inside the flask. A temperature rise due to oxidation of the coal caused the temperature of the bath to be increased automatically. He showed that certain coals would self-heat in oxygen at temperatures ranging from as low as 19°C and 30°C to ignition point.

In 1925, DAVIS and BYRNE determined that the heating tendency of coals high in oxygen is greater, in general, than those of low oxygen content. The tendency was, however, not proportional in all cases. They mention that the rate of heat generation is directly proportional to the oxygen content of the coal.

In 1964, CUDMORE developed an adiabatic calorimeter to investigate the spontaneous combustion liability of the Greta Seam in New South Wales. The apparatus was used to determine the self-heating rates of coal and the effect of possible inhibitors. His findings showed that the evaporation of water played a vital role in the process of spontaneous combustion. The results indicated that a

rise in the CO/O_2 percentage of the air occurred long before any physical perception of "firestink", but it was pointed out that the reverse might well be the case with large underground heatings.

In 1969, GUNEY and HODGES designed an adiabatic apparatus to investigate the influence of moisture at the initial stages of self-oxidation of coal. In the same year, 1969, GUNEY, HODGES and HINSLEY concentrated their research on the measurements of adiabatic temperature rise in coal during the initial stages of the phenomenon. They conducted their tests under different adiabatic conditions and concluded that moisture was chiefly responsible for the spontaneous heating of coal at relatively low temperatures, at least in the initial stages. The heat generated by the wetting of coal was more than that produced by oxidation. With the accumulation of heat from both sources a serious incident may develop.

GUNEY, in 1971, carried out an investigation to determine the tendency of coals to heat spontaneously in terms of time-temperature curves. He mentioned that moisture played a pronounced role in the oxidation of coal and therefore in the development of self-heating in mines.

In 1972, SHEA and HSU conducted the self-heating tests with dry raw lignite. They carried out different tests with oxygen saturated with water vapour, nitrogen saturated with water vapour and dry oxygen. With saturated oxygen the sample ignited spontaneously in less than 5 hours.

In 1975 , GUNEY investigated the spontaneous combustion liabilities of some Turkish hard coals. The samples, consisting of coals known from experience to be liable to spontaneous combustion, were collected from collieries of the Zonguldak and Armutcuk Coalfields. The results of the study showed that there was a close correlation between the adiabatic rise in the coal temperature and production of carbon monoxide and the differences between the equilibrium humidities of coal and air, resulting in greater temperature increases and gas evolution with time of exposure of coal to air.

From his experimental study and data from previous investigations, it was concluded that no single parameter could be used to account for the spontaneous heating process. Among, other contributory factors, the humidity of coal and of the mine environment, the behaviour of coals in the presence of water vapour and the moisture retention characteristics of coal have decided effects on spontaneous heating.

In 1980, KUCHTA, ROWE and BURGESS studied the gas desorption characteristics and self-heating tendencies of 29 U.S. coals using an adiabatic calorimeter. Their experiments showed that the CO, CO/O_2 index, and O_2 absorption rate increase with decreasing rank and increasing oxygen content of the coal. An important finding was that the presence of CO alone in a mine is not necessarily an indication of an incident of self-heating.

HUMPHREYS, ROWLANDS and CUDMORE, in 1981, constructed an adiabatic calorimeter to study the self-heating liabilities of Queensland coals. The results they obtained showed a strong relationship between rank (reflectance of vitrinite) and self-heating characteristics. It was suggested that the rate of self-heating of a coal is a function of the vitrinite content and the position of the coal in the coalification series; i.e. rank.

In 1986, SINGH used a micro-calorimeter for his adiabatic tests. He indicated that oxidation alone does not sufficiently increase the temperature of coal to create a heating problem. It was also indicated that the oxidation of coal and adsorption of water vapour occurred simultaneously and that the heat generated could increase the coal temperature considerably, giving rise to serious

heating.

In 1987, SMITH and LAZZARA tried to investigate the relative self-heating tendencies of 24 coal samples in an adiabatic oven. It was indicated that the self-heating tendency of a coal increased when the coal was dried and exposed to humidified air, and was dependent on the particle size and oxygen concentration of the air. The main conclusions from the experiments are summarized by the researchers as follow:

- The relative self-heating tendencies of coals increased with decreasing rank.
- The minimum self-heating temperature (SHT) of a bituminous coal was strongly dependent on the dry ash-free oxygen concentration of the coal.
- The heating curves can be divided into three sections. Initially, the rate of temperature rise depends on the heat of wetting. The curves pass through an inflection point, in which no mechanism dominates. In the final phase, the oxidation mechanism dominates.
- The activation energy, in general, decreased with coal rank.
- Heat of wetting decreased with increasing rank.
- The lowest SHT's and the corresponding highest self-heating risks were found using humidified air instead of dry air, as the test gas.
- Decreasing the particle size of the coal sample

to a critical size of 74 to 150 microns resulted in a decrease in the minimum SHT. Below 74 microns, the SHT was not effected, probably because of particle agglomeration.

- The tendency of a coal to self-heat decreases with reduced O_2 concentration and is slightly dependent on rank.

In 1988, SMITH, MIRON and LAZZARA tried to evaluate the effectiveness of 10 additives to inhibit the self-heating of coal in an adiabatic oven. Aqueous solutions containing the additives were applied to a bituminous coal with a high spontaneous combustion potential, and the minimum SHT of the coal-additive mixture was measured. $NaNO_3$, $CaCO_3$ and NaCl were found to be the most effective additives tested. The relative order of the effectiveness was:

$$NaNO_3 > NaCl = CaCO_3 > CaCl_2$$
$$> NH_4H_2PO_4 > CaCO_3 \text{ (dry)}$$
$$> NH_4Cl > NaC_2H_3O_2 > KCl$$

$NaNO_3$, however is not a practical inhibitor because of its reactivity with coal at higher temperatures (>300°C). Two additives tested, NaOOCH and Na_3PO_4, acted as promoters, and their minimum SHT's were not determined.

The advantages and disadvantages of adiabatic calorimetry are summarized by WADE (1988):

"In brief, adiabatic calorimetry is an technique which has been applied to determination of spontaneous combustion liability of coals with some success. Its major advantage is the close approximation of the thermal insulation characteristics to be found in coal heaps. This approximation is, however, obtained in controlled laboratory circumstances, rather than in the bunkers or open heaps of direct observation techniques. Samples are thus smaller and time frames shorter.

Disadvantages of adiabatic calorimetry are threefold. Firstly, the apparatus is rather costly. Secondly, the technique presents practical difficulties in ensuring the true adiabatic conditions and preventing the formation of hot spots. Thirdly, in comparison to ignition temperature techniques, adiabatic tests are quite lengthy, particularly with the need for repeated recalibration of the apparatus. The use of ramping temperature methods offers the possibility of accelerated experimental procedures."

4.6. Ignition - Temperature Determinations

Ignition temperature tests include both crossing-point temperature tests and differential thermal analysis (D.T.A.).

In crossing-point temperature tests, the coal sample is placed in a bath and heated at a constant rate. The temperature at which the coal temperature equals the bath temperature is defined as the crossing-point temperature of the coal.

In differential thermal analysis (D.T.A.), the coal sample is heated in a bath at a constant rate as in the crossing-point temperature tests. The temperature difference between the bath and the coal is measured. At the first stage the bath temperature is higher than the coal temperature because of the cooling effect of the evaporation of moisture in the coal (endothermic reaction). When the moisture is evaporated, the coal begins to heat at a faster rate than the bath because of its tendency to self-heat (exothermic reaction) and its attempt to attain the bath temperature.

The term "ignition temperature" is defined by COWARD (1957) as:

a) The minimum temperature necessary for the immediate

imflammation of a combustible substance in a suitable medium such as air or oxygen, or

b) The lowest temperature at which an exothermic reaction can just be detected.

In 1915, NUBLING and WANNER (quoted by COWARD; 1957) heated powered samples in an oxygenated tube in an oil bath and observed the temperature at which the coal began to heat above the temperature of the bath. For a Ruhr coal the observed temperature was 152°C, for a Yorkshire coal 138°C and for a weathered Saar coal 165 to 182°C.

In 1920, SINNATT and MOORE mentioned that, in all cases, except at the higher temperatures, glowing in the mass of coal commenced before ignition took place. The time interval before the former occurred was said to decrease with a rise in the temperature. They defined the ignition temperature as the temperature at which no glowing occurred within a time limit of four minutes.

WHEELER, in 1924, conducted his tests in an electrically heated sand-bath with constant air supply. He obtained two time-temperature curves, as shown in Figure 4.3. One curve shows the rate of rise of the temperature of the sand-bath while the other shows the rate of rise of the temperature of the coal. The temperatures at which the two curves intersect was taken as the ignition

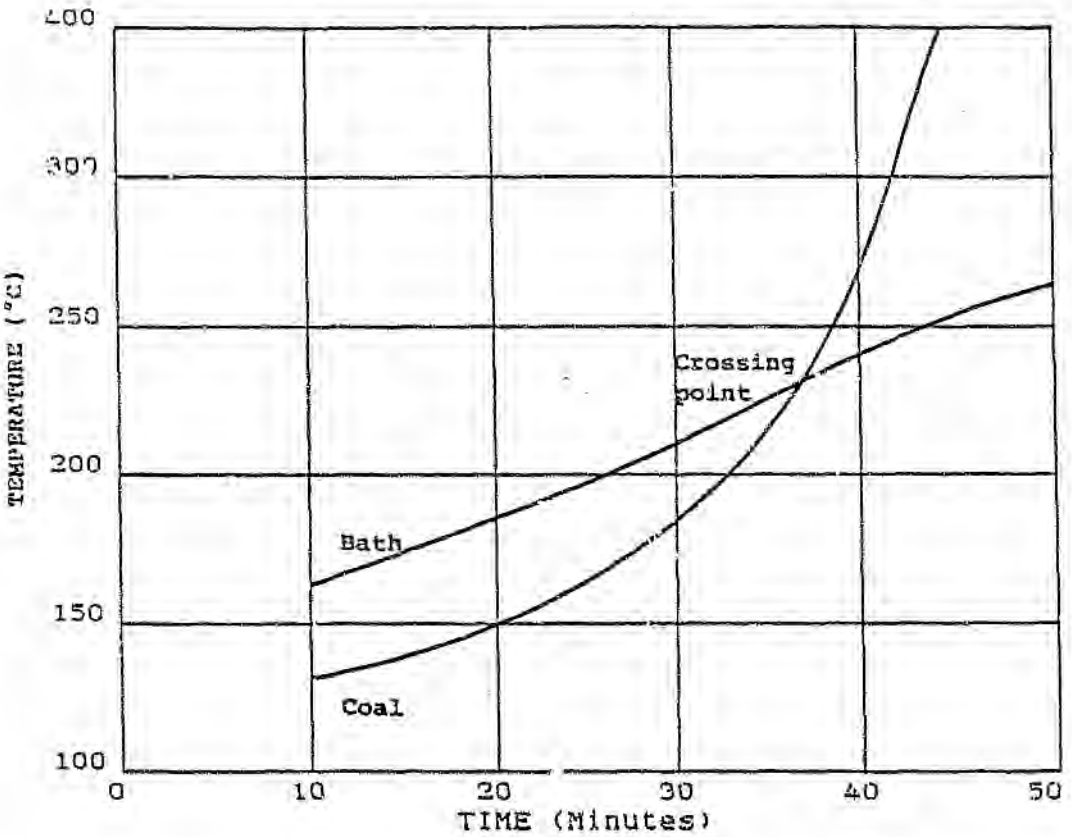

Figure 4.3 Temperature measurements during heating of coal; after WHEELER (1924)

temperature of the coal. He tested 40 different coal samples and pointed out that the coals that most readily self-heated were those containing the highest proportions of oxygen.

In 1926, PARR and COONS determined the ignition-temperatures of Illinois and Pocahontas coals. The critical oxidation temperatures for the Illinois coal

samples were within a range of 138 to 143°C, and the corresponding temperature of the Pocahontas coal was 202°C.

KREULEN, in 1934, tested 76 different coal samples. He regarded the ignition temperature as the point where the temperature curves for the coal and for the apparatus intersected. It was determined that ignition was very rapidly attained after the coal had risen 6°C above the temperature of the apparatus. In 1967, BANERJEE and CHAKRAVORTY used the differential thermal analysis technique (D.T.A.) to classify coals with respect to their susceptibility to self-heating. The D.T.A. technique involves heating a sample at a constant rate and continuously recording the temperature difference (ΔT) between it and an identically heated inert reference material. A typical D.T.A. thermogram is shown in Figure 4.4.

They mentioned it was necessary that the sample and the inert material should have similar packing density. It was recommended that calcined alumina be used as the inert material. They examined different particle sizes of the same sample under identical conditions and compared the thermograms obtained. The three different particle sizes investigated were:

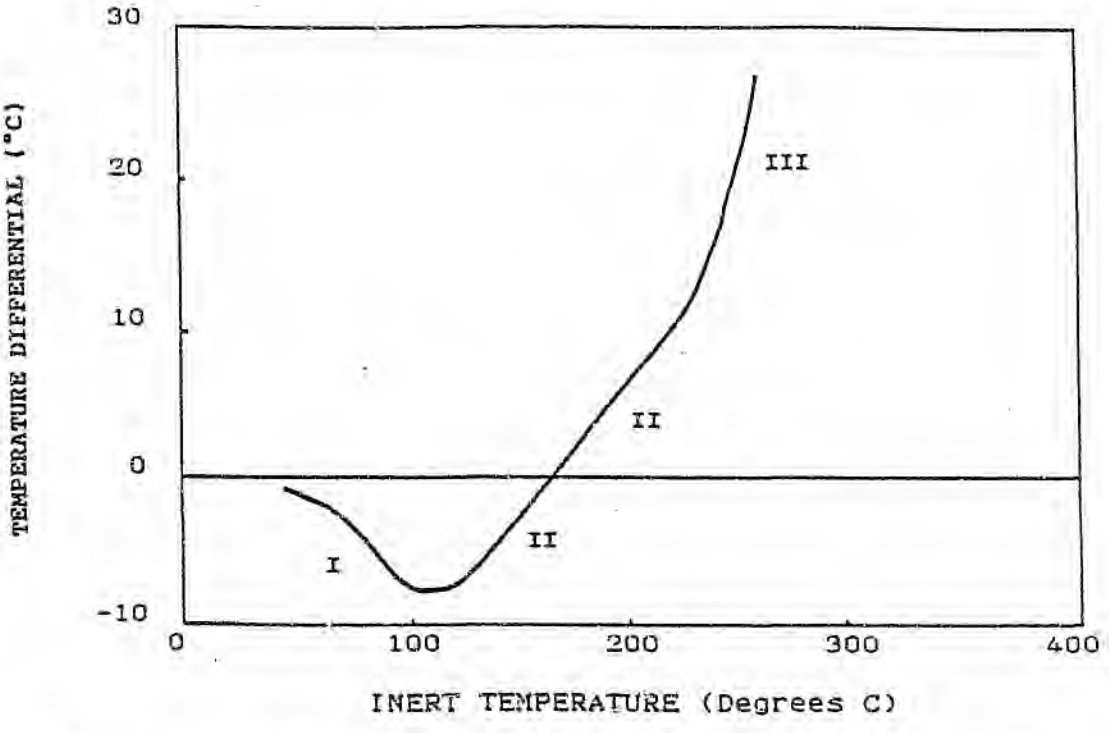

Figure 4.4 D.T.A. thermogram; after BANERJEE and CHAKRAVORTY (1967)

(i) -10 +60 mesh

(ii) -72 mesh

(iii) -200 mesh

The best results were obtained using -72 mesh and -200 mesh particle sizes. Since there was no considerable change between the thermograms obtained with particle sizes of -72 and -200 mesh, it was decided to carry out experiments with -72 mesh (approximately -212 microns). A heating rate of 5°C/min was found to be suitable for D.T.A. tests.

In 1985, BANERJEE, described the stages shown in Figure 4.4. Stage I is the endothermic reaction that predominates mainly from the release of moisture from coal. This followed by an exothermic reaction, in stage II of the process, which ultimately leads to a reaction of very high exothermicity in stage III. The rate of rise in heat evolution in stage II is observed to be much lower for coals with lower susceptibility to spontaneous combustion. In cases of poorly susceptible coals, stage II is seen to persist over a considerable range of temperature, delaying the beginning of stage III. The exothermicity in stage III is not a definite indicator since even poorly susceptible coals may exhibit a heating rate as high as that of highly susceptible coals.

The criteria was summarized for categorizing coals from D.T.A. studies as follows:

a) The sharpness of the slope of thermograms in stage II

b) The temperature of initiation of stage III

BANERJEE, NANDY, BANERJEE and CHAKRAVORTY, in 1972, conducted ignition temperature tests to categorize Indian coals according to their liabilities to spontaneously combust. It was suggested that coals having crossing-point temperatures from 120 to 140°C could be considered to be highly susceptible to self-heating. Samples having a crossing-point temperature of between

140-155°C were said to be moderately susceptible while poorly susceptible coals showed rather high values (160 to 190°C or more). In addition to crossing-point temperature values, they made the classification from the thermograms obtained from D.T.A. studies.

In the same year, in 1972, it was stated by NANDY, BANERJEE and CHAKRAVORTY that coals which showed a sudden and sharp rise in the slope of the temperature-time curve just before the crossing-point temperature curve could be considered to be dangerous.

They concluded that the crossing-point temperature decreased with the increase in moisture, volatile matter and the oxygen content of the coal. It was confirmed that the crossing-point temperature is a function of the coal rank. Higher rank coals usually being less susceptible to spontaneous combustion than the lower rank coals. The spontaneous combustion liability of a coal was considered to increase with:

a) a decrease in crossing-point temperature, and

b) an increase in the slope of the temperature-time curve

In 1972, BAGCHI investigated the crossing-point and ignition temperatures of various Indian coal sample (Figure 4.5). His ignition temperature is described as being the temperature at which the coal burst into flame.

It was pointed out that a coal which reaches the ignition point at a faster rate was likely to be more liable to spontaneous combustion than a coal which follows at a slower rate, even when the crossing-point temperatures were similar.

FENG, CHAKRAVORTY and COCHRANE, in 1973, mentioned that the ignition temperature and the heating rate indicated the spontaneous combustion liability. They proposed an index based on the ratio of heating rate to the relative

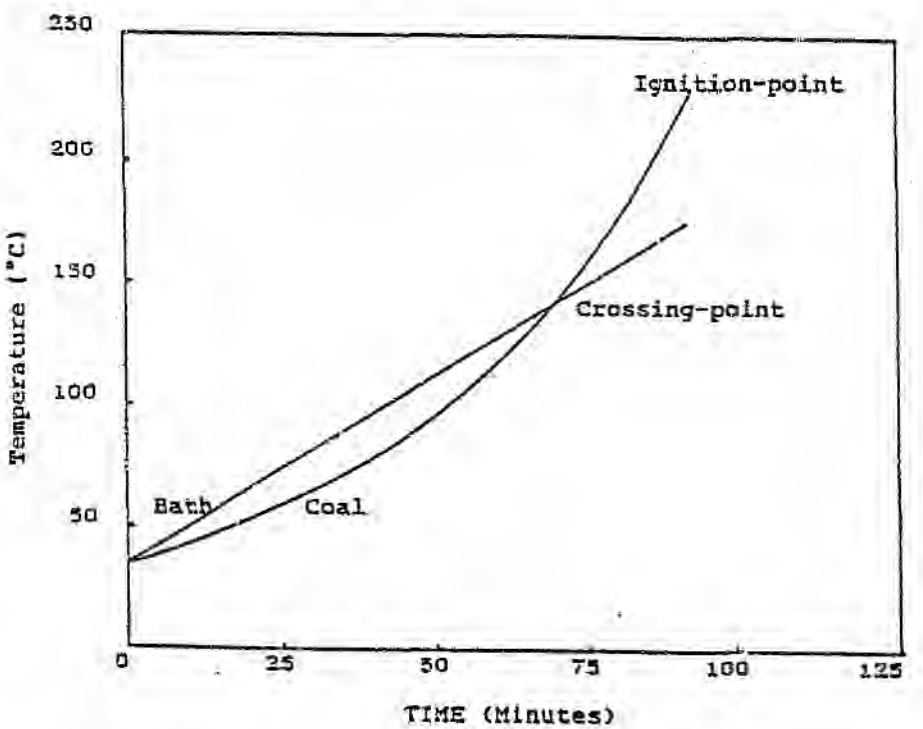

Figure 4.5 Temperature-time graph to explain the crossing-point and ignition point; after BAGCHI (1972)

ignition temperature:

$$\text{Index} = \frac{\text{ave. heat. rate between 110 to 220°C}}{\text{relative ignition temperature}} * 1000 \quad \ldots\ldots (5)$$

In this index they determined the average heating rate between 110 to 120°C to avoid the effect of evaporation of moisture and the exit of volatile matter from the coal. The classification shown in Table 4.2 was proposed to indicate the self-heating tendencies of different coals.

Table 4.2 Spontaneous combustion liability index; after FENG, CHAKRAVORTY and COCHRANE (1973)

Index	Spontaneous Combustion Liability Index
0-5	Low
5-10	Medium
>10	High

In 1983, MOHAN, MAHADEVAN and RAMLU tested coal samples from various sections of six seams of the same colliery. They found that the crossing-point decreases as:

- the moisture increases,
- the volatile matter increases, and
- the ash content decreases

MAHADEVAN and RAMLU, in 1985 , devised a liability index based on the entire heating curve and utilizing the "inflection or perceptible self-heating point" (Fig. 4.6-point b). The heating curve had been divided into three zones as shown in Figure 4.6(a-b, b-c, c-d).

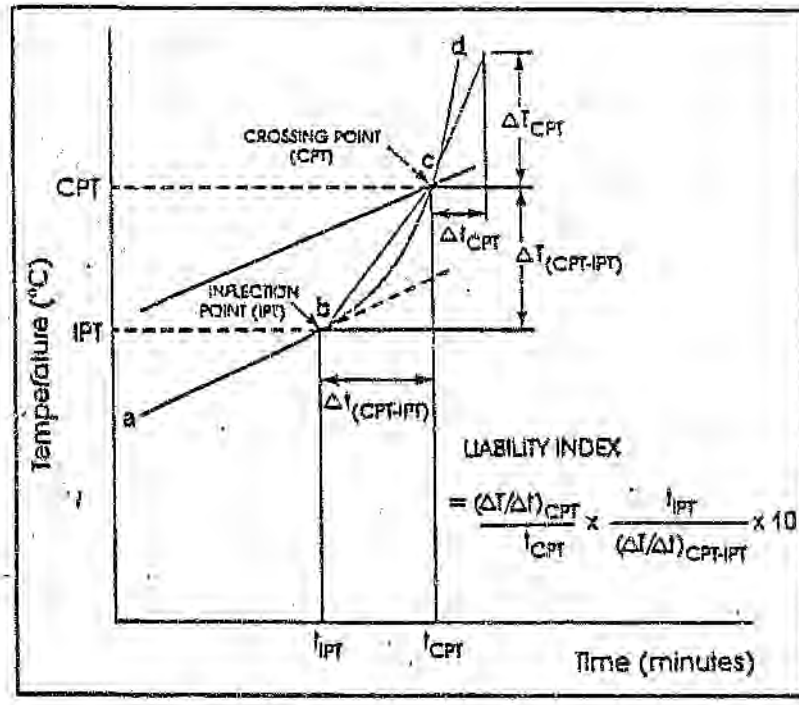

Figure 4.6 Heating curve analysis of coal; after MAHADEVAN and RAMLU (1985)

Point "b" is called the perceptible self-heating generation point or simply the inflection point, point "c" is the crossing-point and point "d" is the active

combustion point. Point "a" is not defined by the researchers.

The new index was defined as follows:

$$\text{Index} = \frac{\text{heating rate at the crossing-point}}{\text{time to reach the crossing-point}} * \frac{\text{time to reach the inflection point}}{\text{average heating rate between the inflection and crossing-point}} * 10$$

$$= \frac{(\Delta T/\Delta t)_{CPT}}{t_{CPT}} * \frac{t_{IPT}}{(\Delta T/\Delta t)_{CPT\text{-}IPT}} * 10 \quad \ldots\ldots (6)$$

It was stated that the index decreases with the increasing liability to spontaneously combust.

In 1986, WADE, PHILLIPS and GOUWS reviewed spontaneous combustion experimental techniques and decided to carry out the crossing-point and D.T.A. tests to determine the liabilities of South African coals to self-heat. The apparatus was designed in 1987.

In 1989, GOUWS and WADE conducted ignition temperature tests for 58 South African coals. They heated an oil bath at a constant rate rate of 1°C/min and kept the air flow rate constant at 400 ml/min. WADE, GOUWS and PHILLIPS

described the advantages of this method and their design in 1987;

- Elimination of the effects of temperature gradients in the bath medium
- Removal of the thermal conductivity consideration from observations of the crossing-point temperature
- Improved accuracy of temperature measurements
- Simultaneous crossing-point temperature determinations and differential thermal analysi on the same sample
- A simple process which can be performed speedily and cheaply.

They pointed out that a coal with a lower crossing-point temperature was generally considered to be more prone to spontaneous combustion than a coal with a higher crossing-point temperature. It was suggested that crossing-point temperatures alone are not a suitable index. The stage II exothermicity slope 'Figure 4.4) was also accepted as an indicator of self heating propensity.

By using identical sample holders for the coal sample and the reference inert material they reduced thermal gradients in the system. This caused their crossing-po' t temperatures to be lower than those recorded by previous researchers. Five coal samples had lower crossing-point temperatures than Feng's lower limit of heating rate (i.e. below 110°C). The index was mo ified by replacing

the heating range with the stage II slope as follows:

$$\text{FCC Index}_{\text{mod}} = \frac{\text{stage II slope}}{\text{crossing-point temp.}} \quad \ldots\ldots\ldots\ldots\ldots (7)$$

They also investigated the index which was described by Mahadevan and Ramlu, and modified it as follows:

$$\text{MR Index}_{\text{mod}} = \frac{\text{stage II slope}}{\text{crossing-point temperature}} * \frac{\text{inert temp. at max.neg.temp.diff.}}{\text{stage IIA slope}} * \ldots (8)$$

The MR index was not accepted because of contradictory results. While Mahadevan and Ramlu stated that the index decreases with increasing liability, they found that the index decreases with decreasing liability.

The crossing-point temperature and stage II slope was used to derive a new index (Figure 4.7). The inverse of the crossing-point temperature was plotted against the stage II slope, which indicates self-heating liability increases with distance from the origin of the graph. The values corresponding to the stage II slope and the crossing-point temperature were joined with a straight line to enclose a triangle. The area of the triangle calculated in Equation 9 was found to increase with the spontaneous combustion liability.

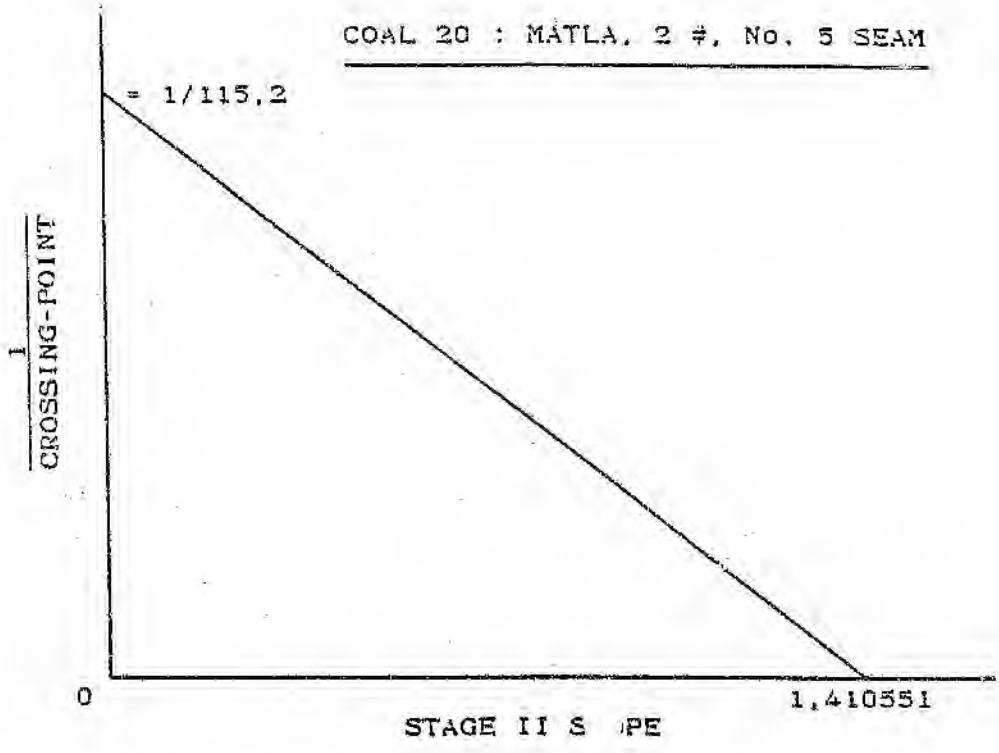

Figure 4.7 Triangle formed by plotting stage II slope against 1/crossing-point temperature; after GOUWS (1987)

$$\text{Area} = 0.5 * \text{stage II slope} * \frac{1}{\text{XPT}} * 1000 \quad \ldots\ldots\ldots (9)$$

The new index, named as WITS-EHAC index, is shown in Equation 10.

$$\text{WITS-EHAC Index} = \frac{\text{stage II slope}}{\text{crossing-point temp.}} * 500 \quad \ldots\ldots (10)$$

In 1990, WADE and GOUWS referred to a coal sample which had a high risk value but had never been the subject of a heating, while another was identified as having a medium risk, but was known to have fired spontaneously. The situation was explained as follows:

" Only from knowledge of the relative contributions to spontaneous combustion of both the coal type and the environment will it be possible to quantify the risk of a heating occurring in any given mining situation."

5. EXPERIMENTAL EQUIPMENT

5.1. Ignition Temperature Test Equipment

D.T.A. and crossing-point temperature tests are carried out with an experimental setup, the main constitutions of which are shown in Figure 5.1.

D.T.A. and crossing-point temperature tests are both related to the thermal characteristics of coal and, due to the design of the apparatus, can be carried out simultaneously. In these tests the thermal behaviour of coal is changed due to exothermic oxidation which occurs in the coal mass. This exothermic oxidation is a function of the temperature of the medium. It is therefore necessary to maintain the temperature increase of the medium at a uniform rate and eliminate thermal gradients in the medium.

5.1.1. Oil Bath

During the experiments a 40 ℓ stainless steel oil bath (Figure 5.2), with dimensions 710*300*200 mm is used.

Different researchers used solid, liquid and gas as the medium. As a solid medium, WHEELER (1924) used an

electrically heated sandbath and SINNAT and MOORE (1920) used cast-iron.

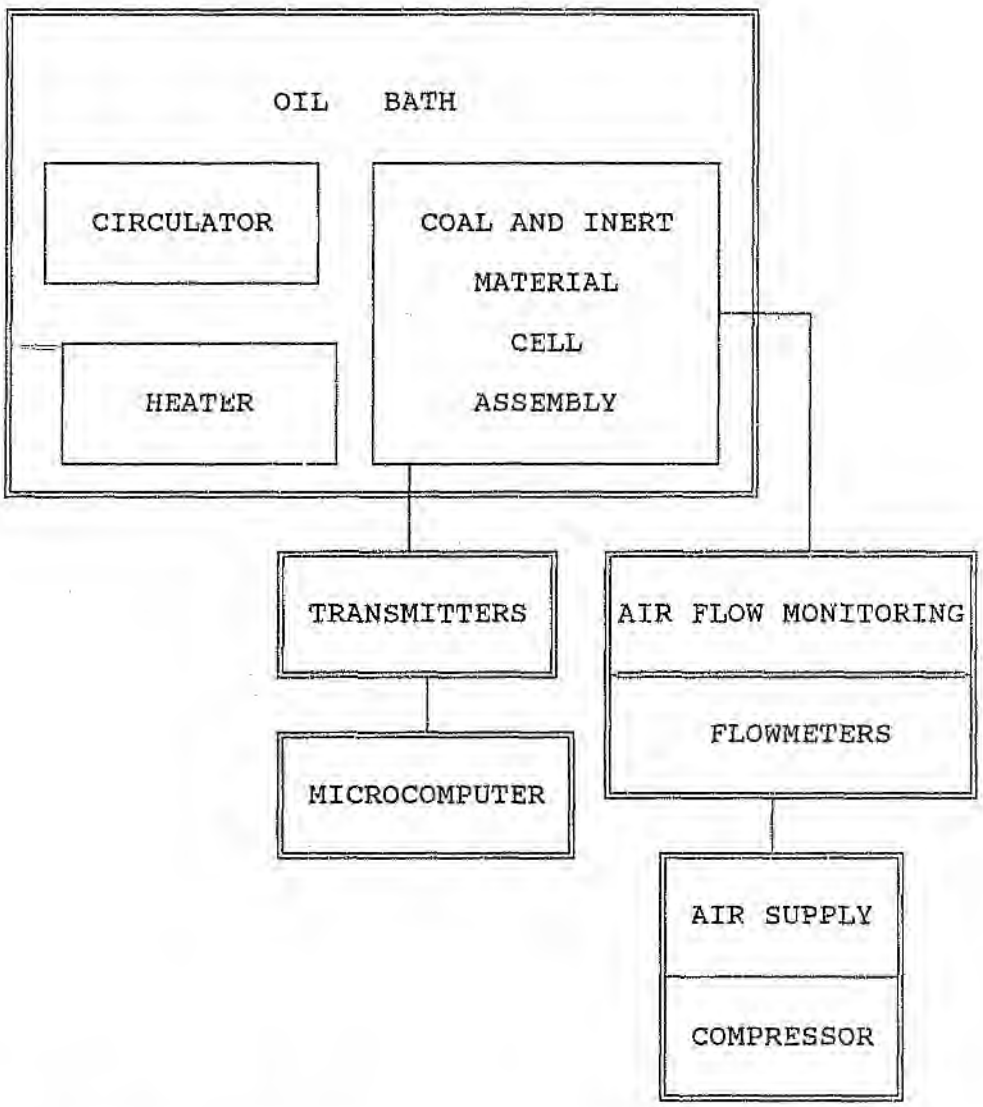

Figure 5.1 The main constitutions of the experimental set up

As a gas medium, PARR and COONS (reported by WADE; 1988) used air in a furnace.

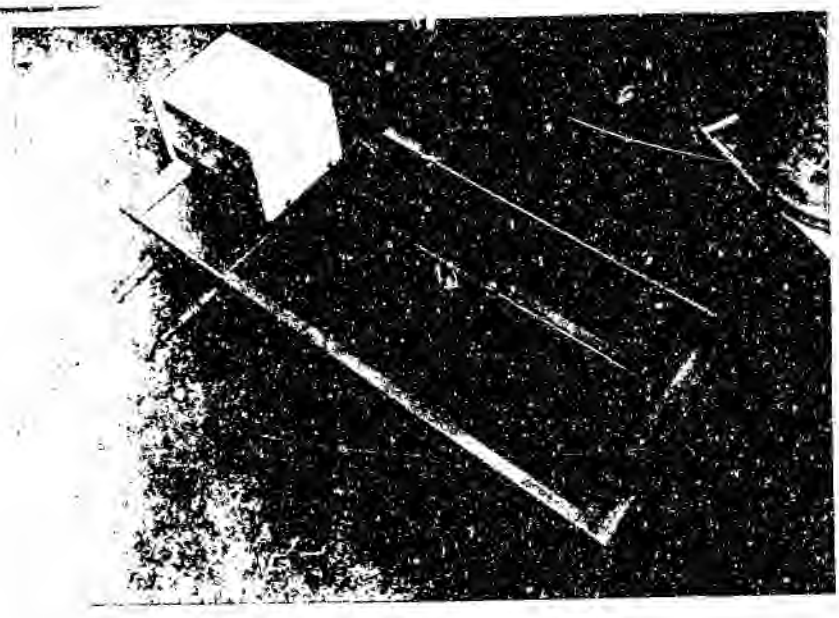

Figure 5.2 The oil bath

In both of these media, hot spots occurred and difficulties were experienced in heating the medium at a uniform rate and eliminating thermal gradients.

As a liquid medium, BANERJEE et.al (1972) used glycerine and BAGCHI (1972) used liquid paraffin. These liquids are not acceptable because of having high viscosities in the lower temperature range, which allow hot spots to occur, and being combustible in nature.

It was decided to investigate using oil as a medium. The oil had to have a low viscosity and high flash-point and breakdown temperatures. SHELL THERMIA-B satisfied these criteria. It is a heat transfer oil and has a 27

centistokes viscosity at 40°C, a flash-point of 210°C and maximum usable temperature of 320°C.

To prevent thermal gradients in the oil, an external pump (Figure 5.3) circulates heated oil to all parts of the bath.

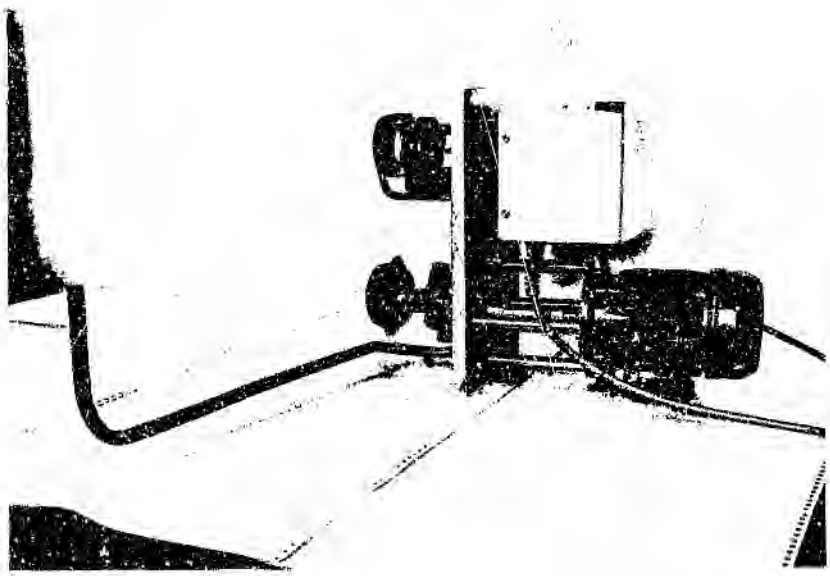

Figure 5.3 External oil pump

The characteristics of this external pump are shown in Table 5.1.

5.1.2. Cell Assembly

During the D.T.A. and the crossing-point temperature tests a cell assembly which consists of 6 cells is used.

Table 5.1 The characteristics of the external pump

Frequency	50 Hz
Power (at sea level)	0.25 kW
Potential difference	220 V
Electric current	2.7 A
Motor speed	1400 rpm

A single cell is shown in Figure 5.4.

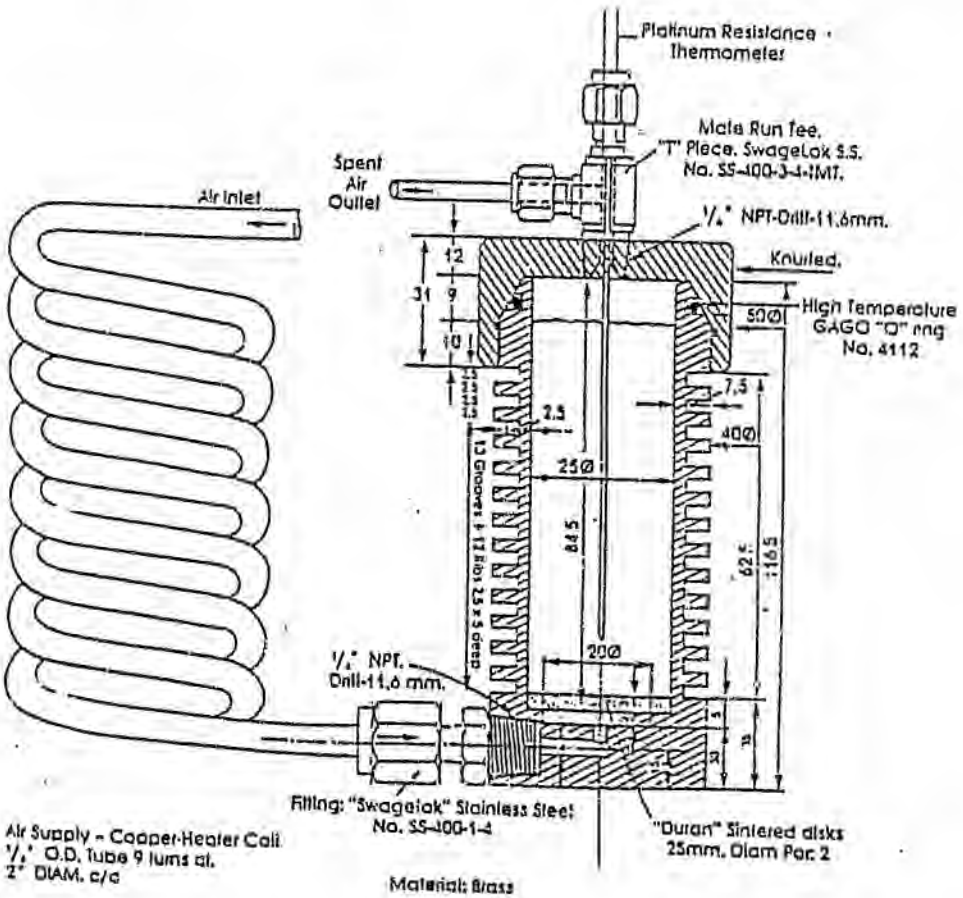

Figure 5.4 Cross - section of a cell

Being immersed in oil, the cell assembly must be oil tight, to prevent any seepage of oil into the cell. A high temperature GAGO "O" ring is used between the cell and its cap to seal the cell. The T-piece on the cap which allows the exit of the platinum resistance thermometer (P.R.T.) is packed with asbestos fibre. The asbestos fibre also prevents the movement of the P.R.T., shown in Figure 5.4.

The cell assembly contains three units for coal and three units for a thermally inert reference material. These six units are connected to a framework equipped with a handle, to place it in and remove it from the oil bath as a single entity (Fig. 5.5).

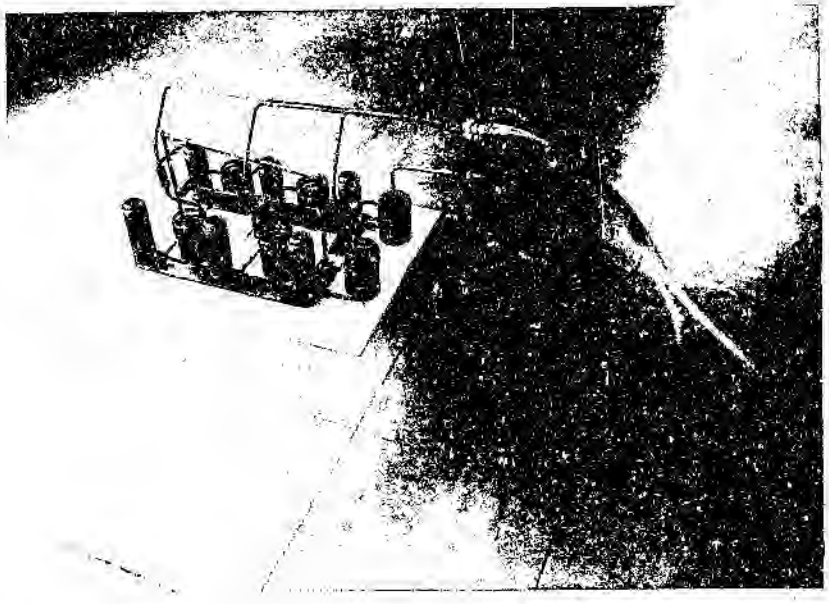

Figure 5.5 Cell assembly

Air is supplied to the bottom of the cell through a copper spiral which is submerged in the oil. Before entering the bottom of the cell, the air is heated by the oil as it passes through the copper spiral. When the air reaches to the bottom of the cell, it passes through a sintered glass disc and percolates through the cell. The spent air leaves the cell via a chimney. During the experiments a fibreglass filter is used inside the cap to prevent any blockages of coal in the T-piece.

Six P.R.T.'s are inserted through the caps of the cells (Figure 5.6), with one more P.R.T. being placed in a holein the cell assembly (Figure 5.7) to monitor the temperature of the oil. The P.R.T.'s used in the spontaneous combustion tests have an accuracy of 0.1°C at

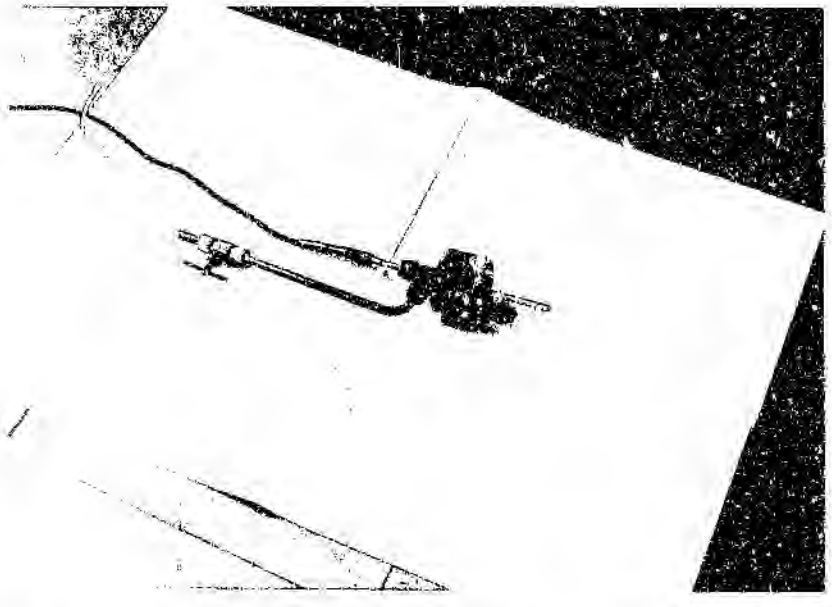

Figure 5.6 P.R.T. packed to the cap of the cell

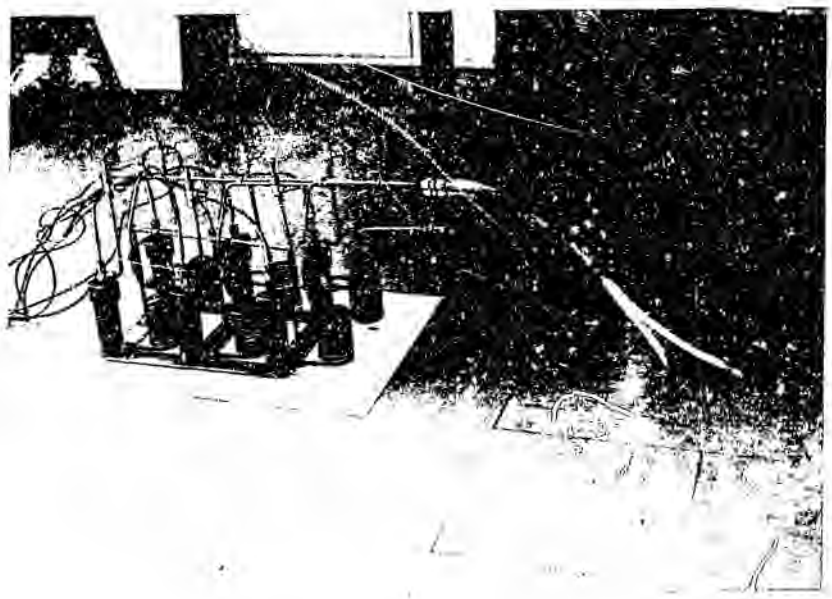

Figure 5.7 Cell assembly with P.R.T.'s

all temperatures and their electrical resistance changes are shown in Table 5.2.

In practice, the temperature of the sample is never the same as the temperature of the oil. An inert material is used and its temperature is plotted in place of the oil temperature to be compared with the coal temperature for crossing-point temperature tests. For D.T.A. tests the coal temperature minus the temperature of the inert material is plotted against the temperature of the inert material.

The inert material must have a thermal conductivity and specific heat characteristics similar to that of coal. It

Table 5.2 Resistance changes of P.R.T.'s

Resistance of thermometer (ohm)	Equivalent temperature (°C)
100.00	0
109.73	25
119.39	50
128.98	75
138.50	100
157.31	150
175.84	200
194.08	250
212.03	300

was found by PAMPUCH and ROGA (quoted by BANERJEE and CHAKRAVORTY; 1967) that the material which has the closest thermal characteristics to coal is coke. Coke, however, suffers from the disadvantage of having volatile materials. During the heating process, these volatile materials vaporise. This endothermic reaction causes a decrease in the heating rate of the inert material.

BANERJEE and CHAKRAVORTY (1967) suggested using calcined alumina as the inert material for crossing-point temperature and D.T.A. tests. Calcined alumina is thermally inert, showing no change in its structure during the heating process. It was decided to use calcined alumina in this research programme. The calcined alumina used is a product of BDH Chemicals. The maximum limits of the impurities of this aluminium oxide is shown in Table 5.3.

Table 5.3 Maximum limits of the impurities of the calcined alumina

Impurities	per cent (%)
Loss on ignition	1.0
Water - soluble matter	0.5
Sulphate	0.05
Iron	0.02

5.1.3. Air Supply

An INGERSOLL RAND ENERGAIR electric compressor is used for the air supply. The characteristics of the motor and the pressure tank are given in Table 5.4.

Table 5.4 Specifications of the motor and the pressure tank

MOTOR	
Electric current	4.0 A
Frequency	50 Hz
Power	0.56 kW
Motor speed	1400 rpm
Operating pressure	
(continuous rating)	750 kPa
(1 hour rating)	800 kPa
PRESSURE TANK	
Safeworking pressure	18 kPa
Test pressure	1766 kPa
Capacity	0.025 m^3

The air from pressure tank is fed to a PLATON model flowmeter which has a flow rate capacity from 10 to 250 cm^3/min via a FMN/AL type flostat. The details of the flowmeter and the flostat are shown in Figure 5.8.

The air flow rate is adjusted to 200 cm^3/min with a valve on the flostat. Air flow rates must be equal in all cells. The exhausts of the inert cells are kept closed in

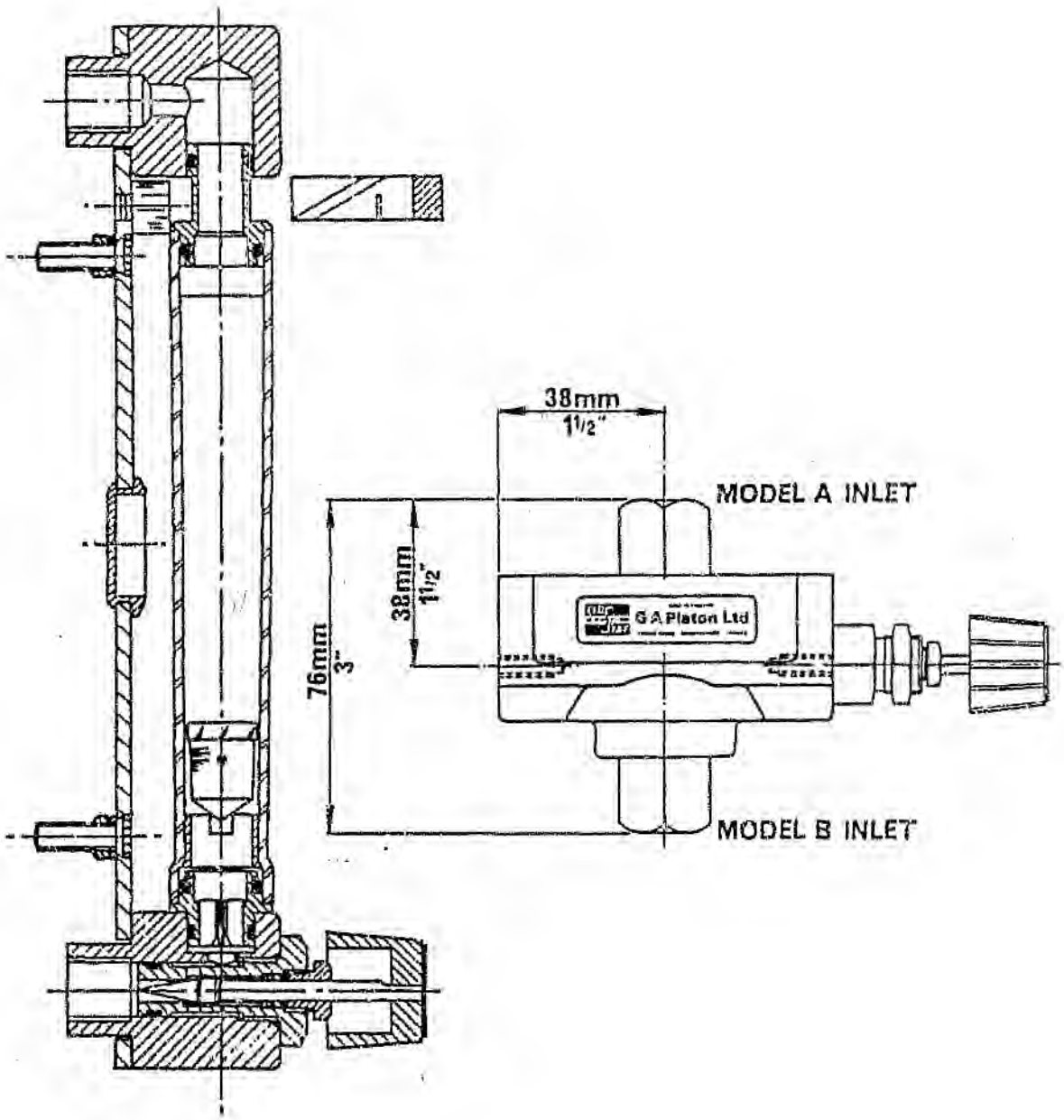

Figure 5.8 Details of the flowmeter and the flostat

order to allow the easier regulation of the air flow in the coal cells.

Air flow rates are monitored continuously by flowmeters (Figure 5.9).

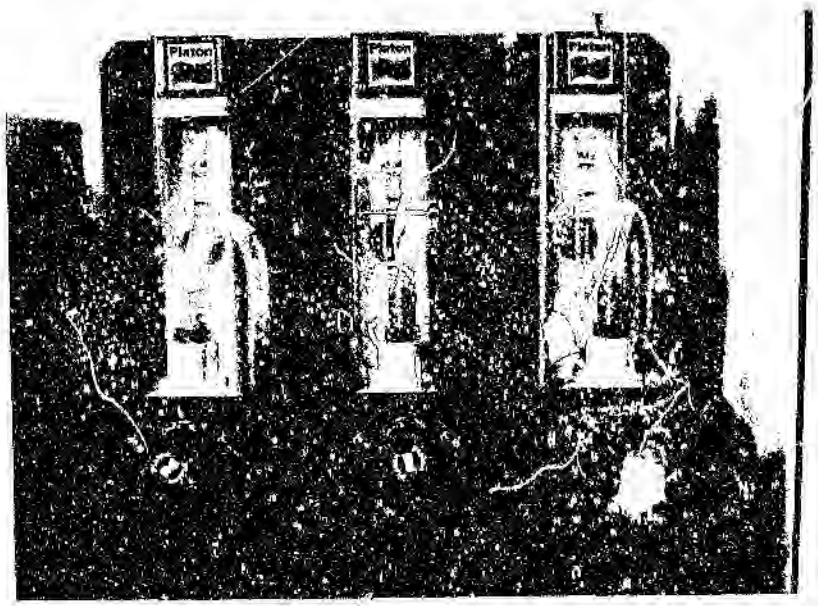

Figure 5.9 The flowmeters

5.1.4. Data Storage

The seven P.R.T.'s are connected to seven SINEAX V 920 transmitters which are shown in Figure 5.10.

Temperatures are converted from analog to digital (A/D) by an A/D converter having sixteen single-ended input channels with a range of 0-10 volts. The temperature of

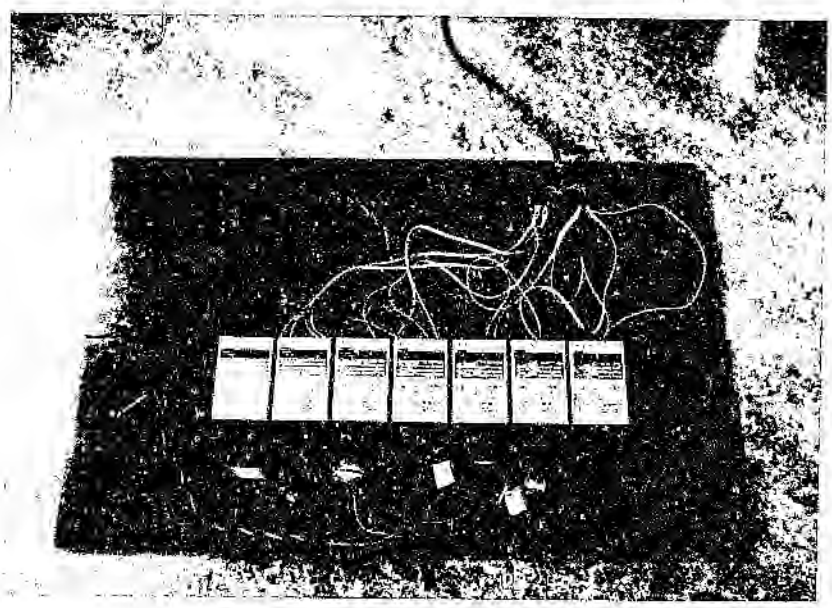

Figure 5.10 SINEAX V 920 transmitters

a sample is converted from analog to digital in 35 μsec with a 12 bit resolution. The transmitters are calibrated to zero and the output voltage readings are checked against the resistance of the thermometers (Table 5.5).

A MINTA PCC-822 B IBM PC compatible microcomputer is used for display and storage. The A/D circuitry is controlled by software which is specially written in Borland International's Turbo-Pascal™.

Temperatures are monitored at 15 second intervals and saved in an ASCII file on a floppy diskette. The graphical display (temperature versus time) is updated every 2 minutes (Figure 5.11). The graphical display of

Table 5.5 Resistance of the thermometers against the output voltage of the SINEAX V 920 transmitters

Resistance of thermometers (ohms)	Output voltage (voltage)
100.00	0.0000
109.73	0.8333
119.39	1.6667
128.98	2.5000
138.50	3.3333
157.31	5.0000
175.84	6.6667
194.08	8.3333
212.03	10.0000

the temperatures of the P.R.T.'s is plotted against time at the top of the screen. The top section is also divided into left and right hand side areas.

The left hand side displays the whole temperature range (0-300°C) while the right hand side displays the range in which the temperature of the coal sample is likely to exceed that of the inert material (110-150°C).

This graphical display can be reviewed as an expanded plot after the experiment has been completed (Figure

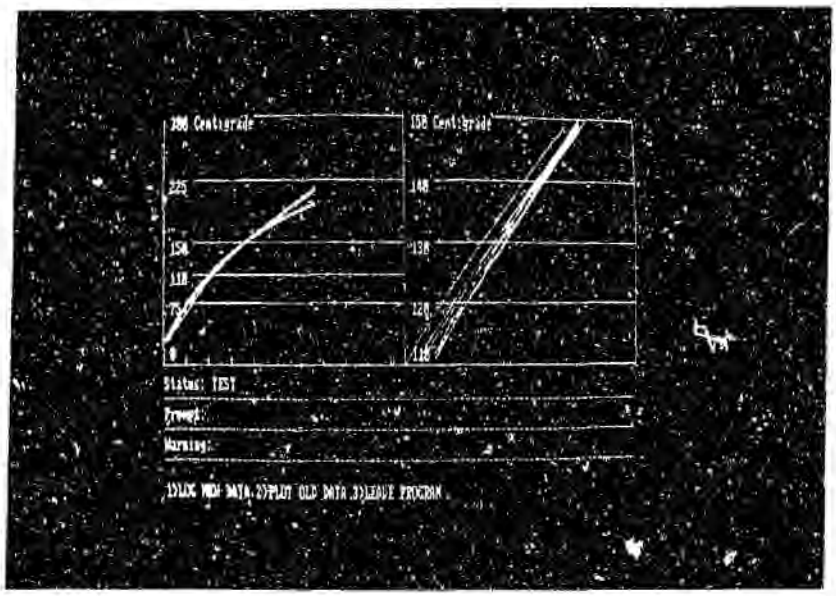

Figure 5.11 Graphical display updated every 2 minutes

5.12).

Status, prompt and warning lines are placed at the bottom section of the screen (Figure 5.13).

In the status line, the name of the file in which the temperature readings are being stored during the experiment is displayed.

The temperatures of the seven P.R.T.'s are monitored every 15 seconds and displayed on the prompt line. A safety circuit is included to switch off the oil bath heater if any of the P.R.T.'s exceed a temperature of 250°C. This prevents the oil bath overheating and the

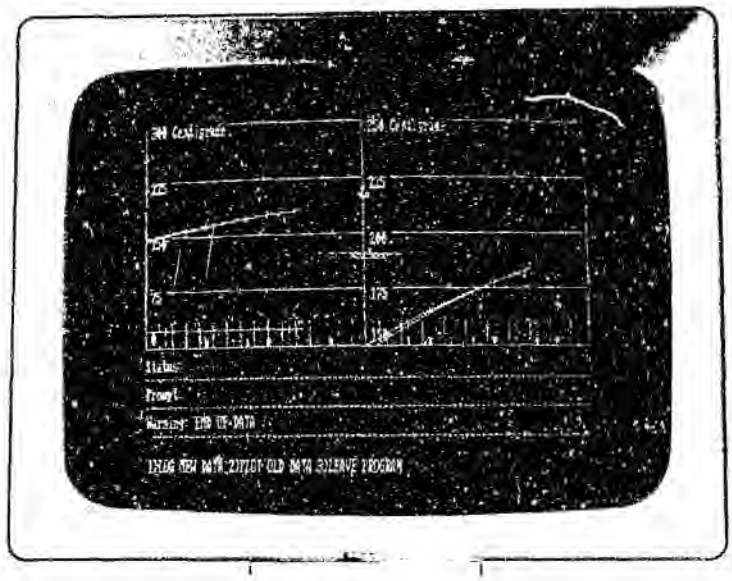

Figure 5.12 Expanded view of an experiment plotted every 15 seconds

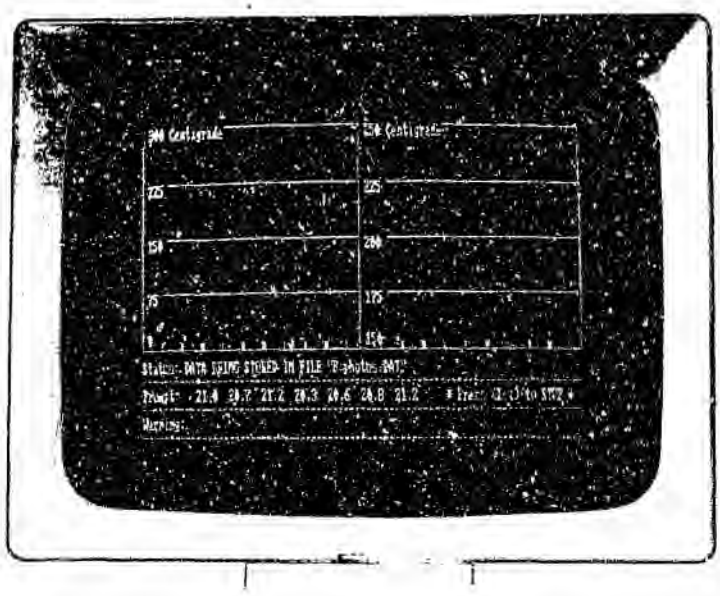

Figure 5.13 PRT temperatures displayed on the screen

possible combustion of the oil. The safety circuit triggers an audible signal if any of the P.R.T.'s exceed a temperature of 250°C.

5.2. Adiabatic Calorimetry Test Equipment

A technique, using an adiabatic calorimeter, was developed by GOUWS (1992) in order to simulate the ignition-temperature tests. The apparatus, which is fully automatic, was designed and constructed in the Department of Mining Engineering of the Witwatersrand University. The apparatus is described by GOUWS, GIBBON, WADE and PHILLIPS (1991), GIBBON (1991) and GOUWS (1992). The major components of the apparatus are:

- calorimeter oven,
- sample holder,
- gas supply and
- data collector.

The new apparatus enabled this research to confirm the ignition-temperature test results.

5.2.1. Calorimeter Oven

The calorimeter oven, which consists of an insulated aluminium heat sink, contains the sample holder and a copper spiral (Figure 5.14).



Figure 5.14 The calorimeter oven

One edge of the copper spiral is connected to a pre-heater, controlled by computer, which heats the inlet gas to the temperature of the sample. The other side of the spiral is connected to the bottom of the sample holder and that ensures the temperature of the gas is almost equal to the temperature of the sample.

A 1000 watt band heater has been manufactured to the size of the heat sink. A similar heater is used to heat the gas before it enters the calorimeter. A step-down transformer with output voltages of 75V, 25V and 10V enables the different voltage ranges to be selected by the computer by means of solid state relays. The four voltage levels with cyclic time control at each level are shown in Table 5.6.

5.2.2. Sample Holder

The brass sample holder, which holds 100 grams of ground coal is shown in Figure 5.15. The coal is sieved to -212 micrometers under the nitrogen environment.

Table 5.6 Heater power range for different voltages (after GOUWS, GIBBON, WADE AND PHILLIPS; 1991)

Voltage (V)	Power Range (W)
220	100-1000
75	11.6-116
25	1.3-13
10	0.2-2

Gas is supplied to the bottom of the sample holder through the copper spiral. The gas passes through a sintered glass disc filter and percolates through the

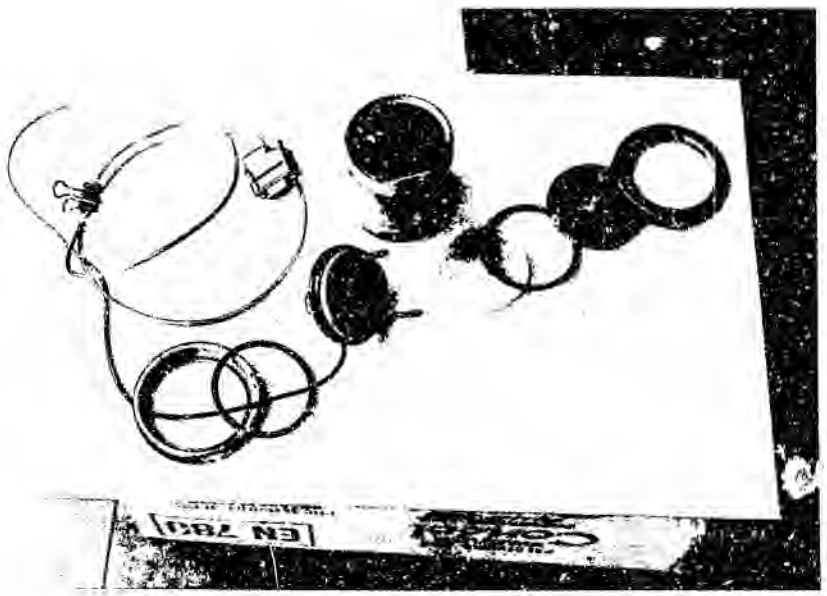

Figure 5.15 The sample holder

sample holder. Before leaving the sample holder, the gas passes through a second sintered glass disc filter. The second sintered glass disc filter, which is located on the top of the sample holder, prevents coal from existing the sample holder.

The temperature of the sample is measured with three platinium resistance thermometers (PRT) which have a 2% accuracy. The three PRT's are connected in series. At each temperature measurement the average of the temperatures of the PRT's is obtained, negating the effect of hot spots in the sample.

5.2.3. Gas Supply

The coal sample and the calorimeter are stabilized at a pre-determined temperature before the experiment starts. Nitrogen is fed through the sample during the stabilization period in order to prevent any oxidation of coal sample before the experiment. After the stabilization of the environment, air is fed to the apparatus instead of the nitrogen. The type of the gas is controlled by a personal computer via selonoid valves.

The gas is controlled by a FMN/AL type flostat, which has a control accuracy of about 3% and a reproducibility of about 1% with a range of 2 to 5000 ml/min. The flow rate is adjusted with a valve on the flostat and displayed on a type GI gapmeter, which has a range of 10 to 250 cm^3/min.

The gases are fed from the gas bottles via pressure regulators. The pressure is manually adjusted to 200 kPa and the actual setting for the gapmeter is calculated by the software and displayed for the operator.

The temperature and the humidity of the inlet gas is measured with a temperature/humidity meter and is recorded during the experiment. A pre-heater is used to heat the gas to the same temperature of the coal sample

in the calorimeter oven. The temperature of the gas is measured with platinum resistance thermometers (PRT's) before entering the oven. The gas is passed through a copper spiral, to ensure the temperature of the gas is equal to the coal sample, before entering the sample holder. The gas then enters the bottom of the sample holder and is percolated through the coal. The gas then leaves the sample holder via a chimney. The temperature and the humidity of the output gas is again measured and recorded.

5.2.4. Data Storage

A personal computer is used to control the equipment. Data collection and data storage is also managed by the computer. The measurements, which are recorded in an ASCII file on a floppy diskette, are:

- sample temperature,
- calorimeter oven temperature,
- ambient temperature,
- inlet gas temperature and humidity, and
- outlet gas temperature and humidity.

The measurements, mentioned above, are monitored continuously. The measurements are stored on a floppy diskette when the temperature of the coal sample differs

from the previous reading by a predetermined (user defined) amount. The measurements are stored in an ASCII file for later analysis.

A plot of temperature against time is displayed on the monitor during the experiment. All current measurements are also displayed.

6. EXPERIMENTAL PROCEDURES

The crossing-point temperature apparatus and the adiabatic calorimeter have been described in Chapter 5 and more detailed descriptions can be found in GOUWS (1987), WADE (1988) and GOUWS (1993). This chapter will, however, deal with:

- sample collection
- crushing
- coal analyses
- glove box and sample preparation
- calibration of the apparatus
- procedures for conducting the tests
- data processing

6.1 Sample Collection

The D.T.A. and the crossing-point temperature tests were conducted on coal samples from one colliery. Durban Navigation Colliery (D.N.C.) was chosen for this research project because of its history of self-heating problems.

Fifty-eight coal samples from different areas and different seams were taken with the aim of contouring the spontaneous combustion liabilities of different seams in this colliery.

The coal samples, which were freshly broken, were obtained from working areas (faces) where they were sealed in five litre plastic buckets before being transported to the laboratory. The air inside the bucket was replaced with nitrogen in the glove box to prevent any further oxidation of the coal.

6.1.1. The Durban Navigation Colliery

The Durban Navigation Colliery, which is one of the few collieries in the Klip River Coalfield of Northern Natal, is situated 35 kilometers south/south-east of Newcastle (Figure 6.1).

It was founded in 1902 and mining was started at the No.1 Mine in 1903 for the supply of the bunker coal to the shipping industry. The No.2 Mine was established in 1918. The No.1 Mine flourished until 1945 and the No.2 Mine continued until 1958. The No.3 Mine was opened in 1936 and is still in operation.

Iscor took over the colliery from a shipping firm in 1954, and opened No.4 Shaft in 1957, No.5 and No.6 Shafts in 1960, No.7, No.8 and No.9 Shafts in 1971, and No.10 and No.11 Ventilation shafts in 1982 (BEUKES; 1989 and DURNACOAL; 1982).

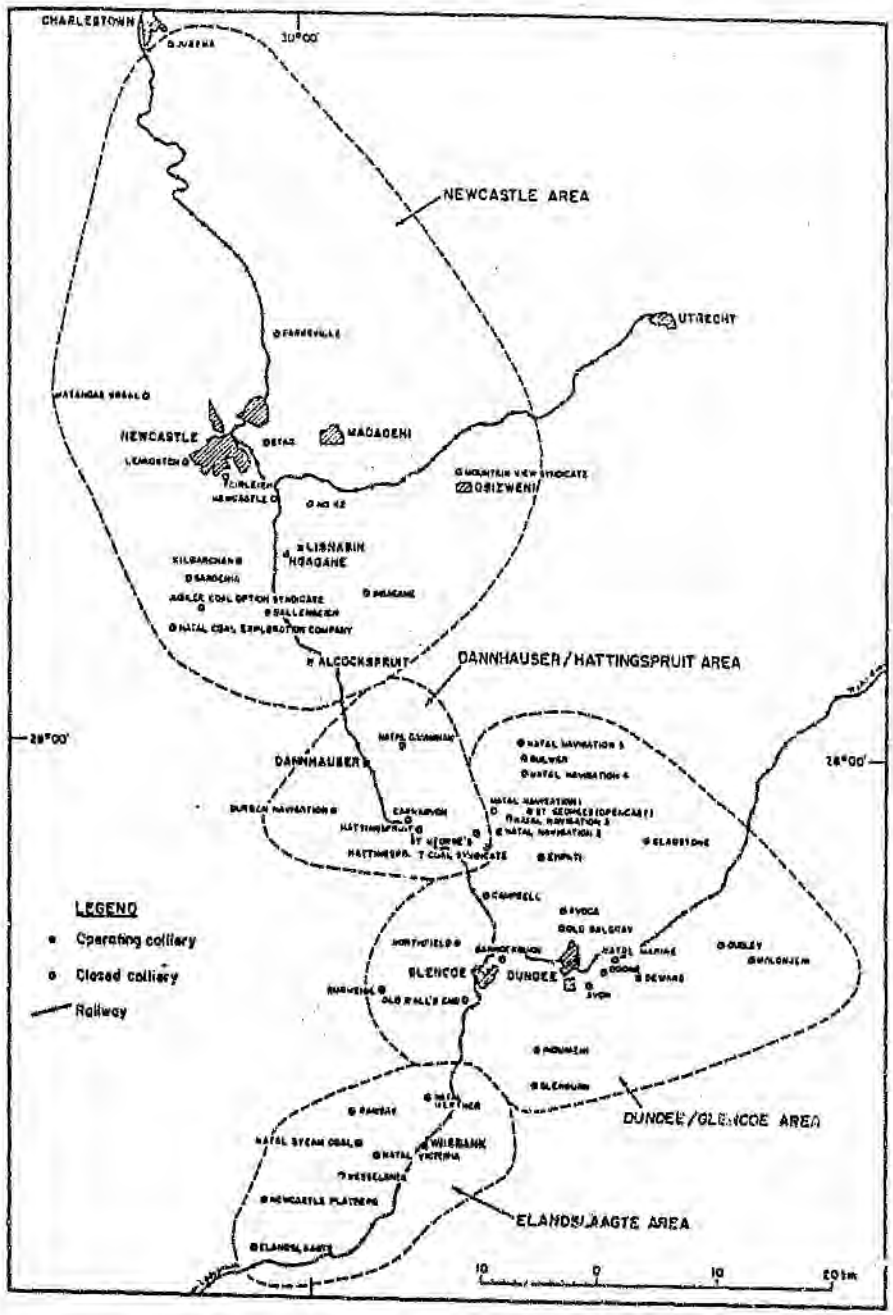

Figure 6.1 Durban Navigation Colliery; after BELL and SPURR (1986)

6.1.1. Geology and Structure

The geology in the Durban Navigation Collieries area consists of sediments of the Volksrust and Vryheid Formations of the Ecca group. The Volksrust Formation consists predominantly of argillaceous material while the Vryheid Formation consists of arenaceous material with shaley sandstones, occasional interbedded shales and coal seams (Figure 6.1 and 6.2).

A thick dolerite sill, the Ingogo, is present over virtually the entire mine area. The sill is on average 80 to 90 metres thick and lies some 80 to 90 meters above the coal zone. In the northern area a second dolerite sill, the Talana, is present as a transgressive feature cutting through the Ingago sill in some places and through the coal zone in others.

Occurring irregularly in the mine area are thick sandstone bodies lying either with the bottom seam or as part of the midstone separating the top and bottom seams.

A thin (0,02-0,03 m) coal seam occurs some 18-20 m above the main coal zone and provides an important stratigraphic marker. The main coal zone consists of two coal seams, the top and the bottom, separated by 0,90-1,10 m of sandstones and carbonaceous mudstones. The

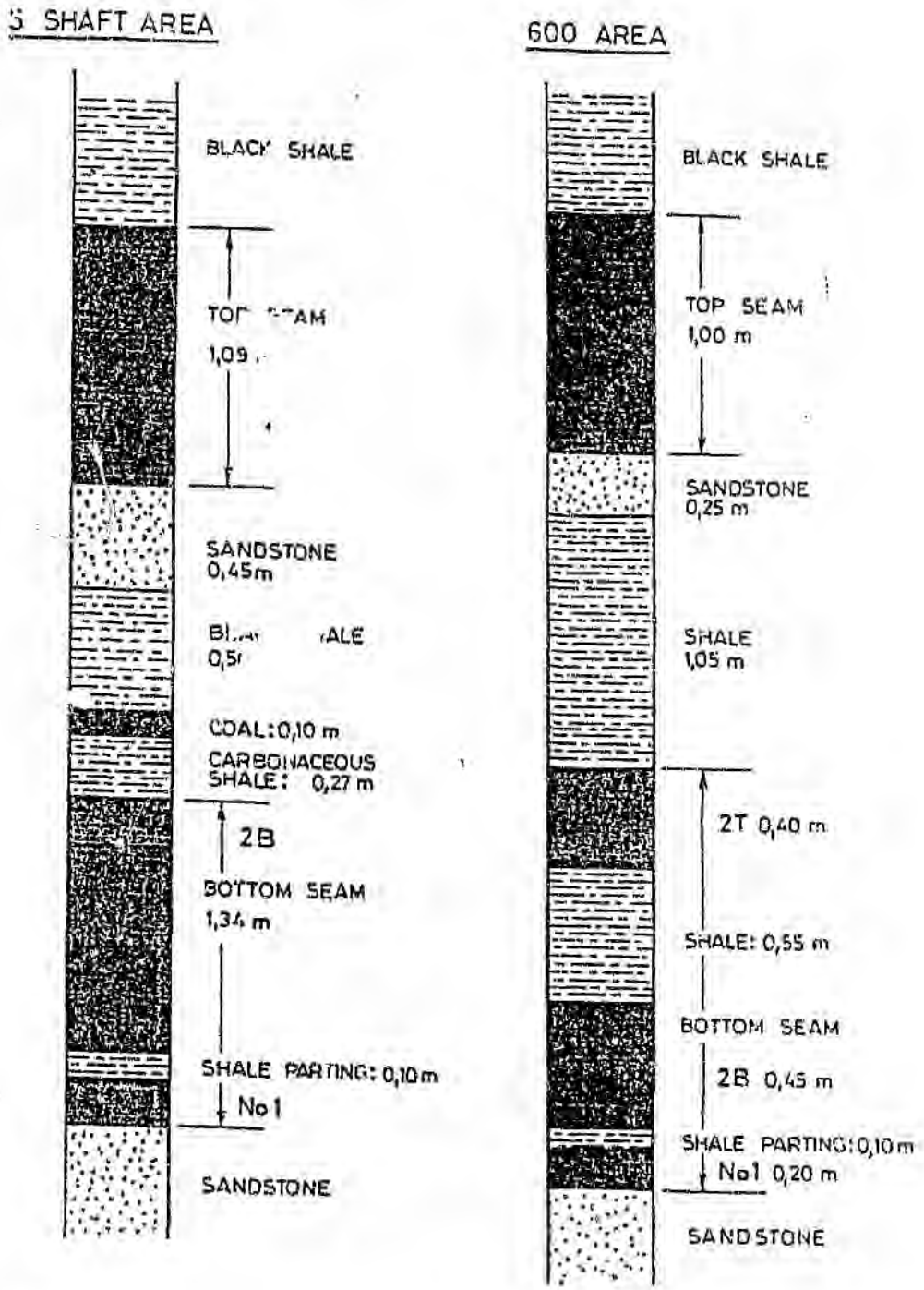

Figure 6.2 Structure of D.N.C. seams; after BEUKES (1989)

top seam averages 1,10 m in thickness and frequently contains minor shale partings. The bottom seam is actually a composite seam consisting of two or three individual coal seams depending on its location in the mine area. These seams are separated by sandstone, shale and carbonaceous shales (Figure 6.2) (BEUKES; 1989).

6.2. Crushing

The crushing of the coal samples is conducted using a jaw crusher.

The jaw crusher is cleaned with a brush before the crushing of each sample. The throat opening of the crusher is set to -5 millimeters. The crushing is performed under the atmospheric conditions. It is carried out as quickly as possible to minimize any uncontrolled oxidation of new surfaces of the samples.

The crushed coal samples are then returned to the laboratory where they are stored in a nitrogen environment. The samples are stored in two 2 litre preserving jars for use in the experiments and two half litre preserving jars for proximate, ultimate and petrographic analyses.

6.3. Coal Analyses

The crushed and stored coal samples are sent to coal analysis laboratories in order to obtain knowledge about the characteristics (quantity and quality) of these samples. These analyses also made it possible to make regressions of WITS-EHAC index values, crossing-point temperatures and stage II slopes against the results of the coal analyses. The results of the regressions are considered in Chapter 7.

The coal analyses include proximate, ultimate and petrographic analyses. The results of these analyses are given in Appendix I,II,III,IV and V. The contents of the proximate, ultimate and petrographic analyses are listed as follows:

a) Proximate analysis
   - Moisture content
   - Ash content
   - Volatile matter content
   - Fixed carbon content

b) Ultimate analysis
   - Phosphorus content
   - Elemental carbon content
   - Hydrogen content
   - Nitrogen content
   - Oxygen content

- Total sulphur content
- Pyritic sulphur content
- Sulphate sulphur content
- Organic sulphur content
- Gross calorific value
- Roga index
- Swelling index

c) Petrographic analysis
- Maceral analyses (8 types)
- Microlithotype analyses (8 types)
- Reflectance of vitrinite analyses (rank)

6.4. Glove Box and Sample Preparation

The samples are prepared in a glove box (Figure 6.3) under a nitrogen environment to prevent oxidation of the coal samples before the D.T.A. and the crossing-point temperature tests.

High purity nitrogen is supplied to the glove box by a 7,4 kg cylinder via a flexible pipe. Before closing the glove box three cells are charged with 25 grams of calcined alumina.

Their caps and the valves affixed to the chimneys are closed and screwed on. Fibreglass filters are installed

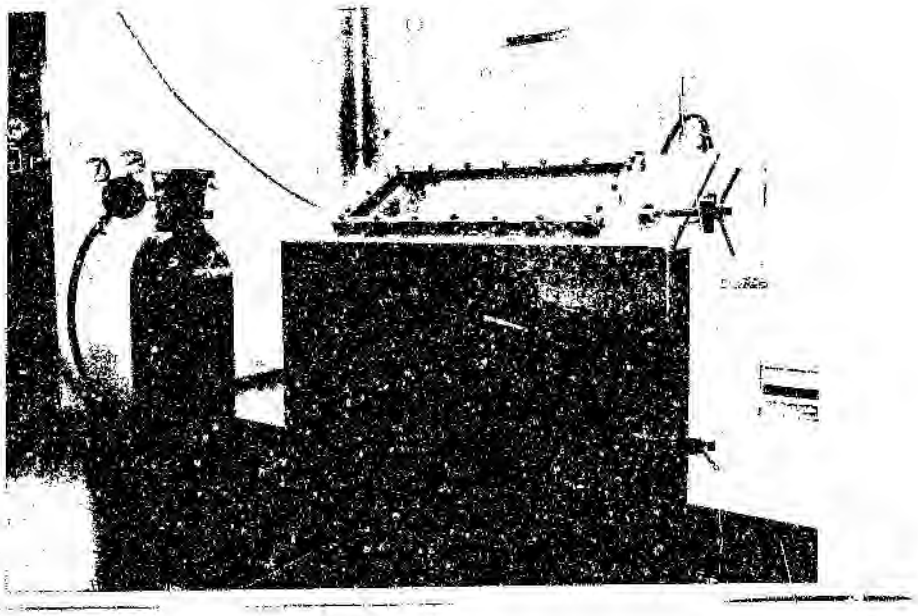

Figure 6.3 The nitrogen glove box

in the remaining three caps to prevent any blockage at the chimneys by the coal sample during the experiment.

The glove box (Figure 6.4) is sealed after ensuring that the following equipment has been placed in the glove box:

- the cell assembly
- the two litre preserving jar including the coal sample
- the sieve assembly
- the chimney caps
- the grinder
- the teaspoon
- the brush
- the balance

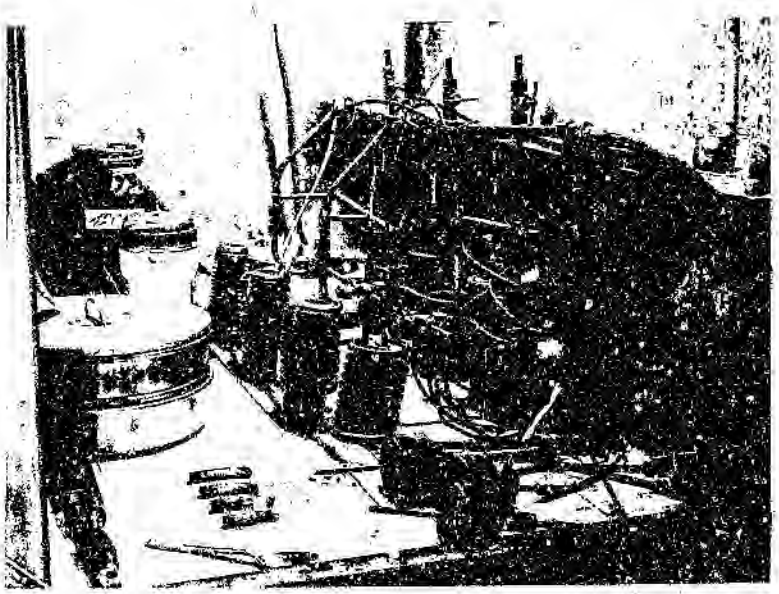

Figure 6.4 The glove box before sealing

- the beaker
- the funnel

Once the glove box has been sealed, nitrogen is fed into the box to replace the oxygen. This process takes approximately 1,5 hours.

Two teaspoons of crushed coal are placed in the grinder and ground for about 10-15 seconds. The grinding must be conducted after ensuring that the level of the coal sample does not cover the blades of the grinder in order to not to overload the motor. The ground coal is tipped onto the sieve and the grinder and its lid are cleaned

out using the brush. The ground coal is sieved to minus 212 microns. The sieving procedure is completed when no more coal passes through the sieve.

These steps are repeated until 60 grams of coal are collected in the sieve pan. It is important to grind the oversized coal because of the affect of the coal maceral groups on the spontaneous combustion liability.

In spontaneous combustion tests, vitrinite has an important role. It is the most brittle and most reactive maceral group to the oxidation reaction (FALCON; 1985). Since spontaneous combustion is an oxidation reaction, the vitrinite component of the sample must represent its source. The oversized coal is ground again to grind all of the vitrinite. Once 60 grams of ground coal are obtained, the empty beaker is placed on the balance and it is switched on. 20 grams of coal are measured out into the beaker. The coal is poured into a cell from the beaker using the large funnel. The remaining two cells are filled with ground coal in the same way. The caps of the cells containing ground coal are then screwed on and the chimney caps are fitted onto the chimneys and the air inlet manifold of the cell assembly. Before opening the cabinet, the preserving jar containing the crushed coal is sealed again and the valve on the nitrogen cylinder is closed.

6.5. Ignition Temperature Test

6.5.1. PRT Calibration

Since each of the PRT's have different circuit resistances, it was necessary to calibrate each of the PRT's to obtain the real temperature readings.

The P.R.T.'s are calibrated against a high precision mercury thermometer. The P.R.T.'s are bundled together and immersed in the oil bath. The heater is adjusted to 30°C and the temperature of the P.R.T.'s are recorded with the computer. When the temperatures of the P.R.T.'s remain static, the system is considered to have stabilised. The temperatures of the P.R.T.'s are written on a calibration sheet, as is the temperature reading of the mercury thermometer. This process is repeated from 30 to 200°C at ten degree intervals (Table 6.1).

The temperature readings of P.R.T.'s are corrected to real temperatures with a basic program called SPLIT which is shown in Appendix VI.

6.5.2. Carrying Out the Test

The cell assembly is placed in the oil bath and the heater is set to 30°C to stabilise for half an hour. The

Table 6.1 The results of the calibration of PRT's

CIR. HEAT. °C	Hg THERM °C	1 °C	2 °C	3 °C	4 °C	5 °C	6 °C	7 °C
30	29.8	30.6	30.4	30.8	30.0	32.0	30.6	31.2
40	39.8	40.4	40.2	40.6	39.9	42.1	40.4	41.0
50	49.6	50.3	50.0	50.5	49.6	51.9	50.2	50.8
60	59.8	60.6	60.2	60.7	59.9	62.1	60.5	61.0
70	69.5	70.3	70.0	70.5	69.6	71.8	70.3	70.8
80	79.8	80.7	80.4	81.0	80.0	82.2	80.6	81.2
90	89.7	90.6	90.1	90.7	89.9	92.1	90.3	90.9
100	99.6	100.7	100.1	100.8	99.9	102.0	100.4	101.1
110	109.3	110.5	110.0	110.6	109.6	112.1	110.3	110.8
120	118.9	120.1	119.6	120.2	119.4	121.5	119.8	120.5
130	128.4	129.7	129.3	129.8	129.0	131.1	129.5	130.2
140	138.0	139.8	139.3	139.6	139.0	141.0	139.4	140.1
150	147.6	149.5	149.0	149.4	148.7	150.6	149.2	149.9
160	157.6	159.6	159.2	159.3	159.7	160.7	159.6	160.1
170	167.2	170.1	169.5	169.7	169.2	171.1	169.7	170.5
180	177.4	180.7	180.1	180.4	179.9	181.6	180.5	181.2
190	187.4	191.1	190.4	190.6	190.2	192.2	190.9	191.5
200	197.1	200.8	200.2	200.3	199.8	201.9	200.6	201.1

temperatures are monitored with the computer. Once the stabilisation period is completed, the caps on the

chimneys are removed and air is fed to the cells via the flowmeters. The flowmeters are adjusted to 200 cm^3/min and the air flow rate is kept stable during the test. The cell assembly is again allowed to stabilise for approximately 15 minutes. The computer program is then restarted after the heater is set to 30°C. The remainder of the experiment is controlled by the computer.
The experiment is not completed until one of two conditions is satisfied. Firstly, the experiment should not run for less than two and a half hours. Secondly, the temperature should at no time exceed 300°C.

When the experiment is completed, the computer program is stopped. The circulator, heater and air flow is then shut off. The valves on the chimneys are closed in order to starve the sample of oxygen since self-heating is an oxidation reaction. The fume cabinet is then switched on to disperse oil fumes and to allow the bath to cool as quickly as possible. The heater is then set to 30°C and switched off again. The bath is left to cool overnight. After the compressor is switched off, the air in the tank is discharged.

The following day the cell assembly is cleaned using benzine and cotton waste and the cells are checked for any traces of oil seepage.

6.5.3. Data Processing

The data storage was described in section 5.4 where it was stated that the temperatures of all the P.R.T.'s are measured at 15 seconds and saved in an ASCII file (Appendix VII). Since these temperatures are not the true temperatures, as mentioned in section 6.4, they have to be corrected by applying calibration factors. The temperatures are corrected with a SPLITFIX program (Appendix VI). With this program, every fourth reading (i.e. one reading per minute) is written to another file called .OOO (Appendix VIII).

The corrected data is then used to create a .DTA file (Appendix IX) which contains the temperature differential between the coal and the inert material.

The .DTA data is then transferred to a software package which enables one to create and plot the curves (Figure 6.5). The curves are averaged with a program called AVERAGE (Appendix X).

The average values of the inert temperature and the temperature differential are written to a data file called .ZZZ (Appendix XI). This file is then used in a basic program called ANALYSIS to analyse the composite curve. The output of this program is shown in Appendix

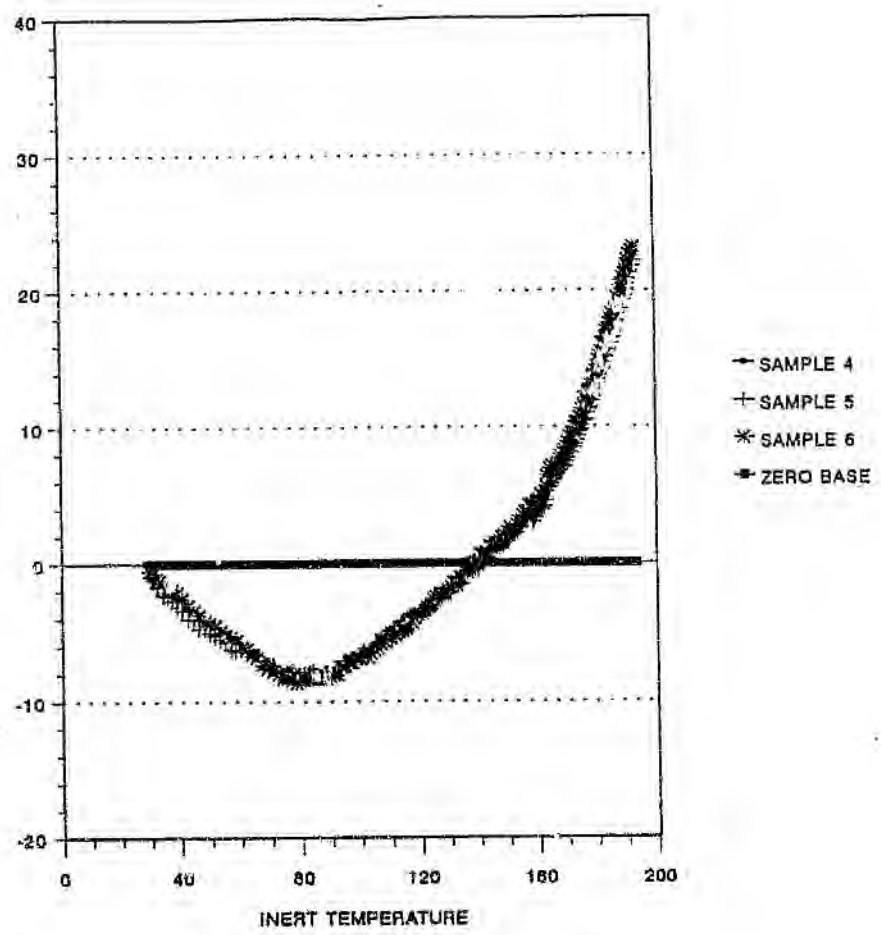

Figure 6.5 The curves of a .DTA file

XII. The output of the ANALYSIS program includes the following:

— Endothermic reaction
  - the point of maximum negative temperature differential
  - the slope of the negative side of the thermogram

— Exothermic reaction
  - characteristics of the crossing point

- positive slope of stage II
- characteristics of the positive slope change

— High exothermicity
- maximum temperature differential
- positive slope of stage III

6.6. Adiabatic Calorimeter Test

The calorimeter, which is used to simulate the results of the ignition temperature tests, has three test options:
- incubation test,
- minimum self-heating temperature test, and
- crossing-point temperature test.

In this phase of the research, it was decided to use the calorimeter in the incubation mode to simulate the tests conducted with the ignition temperature test assembly described in section 6.5.

6.6.1. Carrying Out Calorimeter Test

During the crossing-point temperature test option, the sample holder and the sample are heated at the same heating rate as that used during the ignition temperature

tests. The heating rate was chosen 1°C/min, but it could not be achieved by the sample holder. The experiment was stopped by a controller when the calorimeter oven reached 200°C in order to prevent any damage to PRT's. The temperature of the coal was far behind the oven temperature and the temperature of the coal obtained in the ignition-temperature tests could not be reached in this experiment.

In order to eliminate this discrepancy, a band-heater controlled by a variac was used to heat the sample holder and the sample while the calorimeter was used in the incubation mode. The band-heater was wrapped around the sample holder and connected to a 0-280 volt variac set 53 volts. This arrangement heated an inert material in the sample holder at the required rate. Calcined alumina was used as the inert material, as was used in the ignition temperature tests.

The variac was switched on at the beginning of the experiment. Thirty degrees centigrade was chosen as the starting temperature for the experiment. The experiment stopped when the temperature of the oven, hence the sample, reached 190°C. The same experiment was then repeated using a coal sample. The data obtained from the inert material was used as a datum, to examine the heat characteristics of the coal sample.

6.6.2. Calorimeter Data Processing

The rising temperature mode was used to achieve experiment results. It is similar to the differential thermal analysis which was used to obtain results from the ignition temperature tests.The heating rate of coal was compared to the heating rate of inert material.

The log of temperature rise ($\ln {}^{dT}/_{dt}$) was plotted against the inverse of the absolute temperature of the coal ($^{1}/_{T}$). This plot is called Arrhenius plot and is shown Figure 6.6.

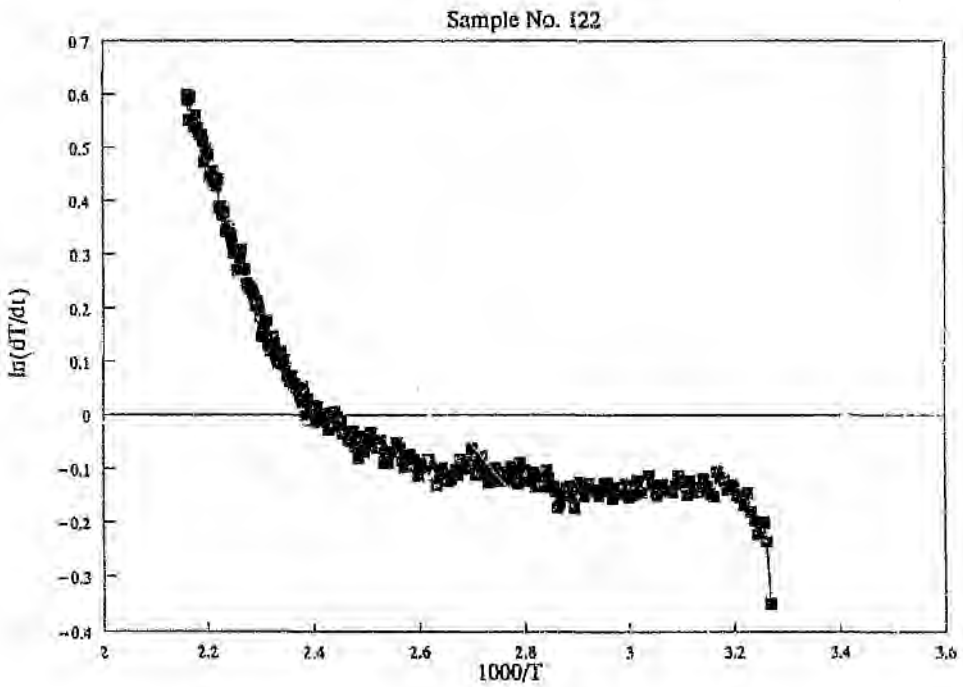

Figure 6.6 Typical Arrhenius plot

The slope the straight line is equal to $-E/R$. The activation energy can be calculated from this slope. The vertical intercept, which is called rate coefficient or frequency factor, is greater for coals more likely to spontaneous combustion (BANERJEE; 1985).

The temperature of the coal increases at a reasonably constant rate in the first part of the experiment (the horizontal part of the curve), and increases at an increasing rate because of the tendency of coal to oxidation reaction. The point where the horizontal curve finished is called "Threshold temperature" (BANERJEE; 1985).

7. RESULTS AND DISCUSSIONS

7.1 Ignition Temperature Test Results

The results and the discussions in this chapter are based on experiments conducted on 58 coal samples collected from Durban Navigation Colliery. Some of the samples were taken from different seams at the same working area, eg. sample 104 was taken from the top seam at the location marked 260 on the plan, while sample 105 was taken from the bottom seam at the same horizontal reference location. The list of the samples is shown in Table 7.1. As can be seen from Table 7.1, the samples were obtained from 28 different locations.

For each sample, crossing-point temperature and differential thermal analysis tests were conducted simultaneously and the data was processed as described in Chapter 6.6.

7.1.1. Simple Indices

Simple liability indices are the characteristics obtained from the crossing-point temperature and differential thermal analysis experiments. The characteristics suggested to be indicative of the tendency of a coal to

Table 7.1 List of sample sources

SAMPLE	LOCATION	SAMPLE	LOCATION	SAMPLE	LOCATION
101	833/2T	121	601/T	140	721/B
102	833/T	122	601/B	141	823/B
103	839/T	123	410/B	142	823/T
104	260/T	12[illegible]	811/T	143	846/B
105	260/B	125	811/B	144	846/T
106	6RD/B	126	512/T	145	401/T
107	358/B	127	512/B	146	401/B
108	358/T	128	846/2B+1	147	502/T
109	354/B	129	611/T	148	502/B
110	354/T	130	611/B	149	530/T
111	725/B	131	611/2T	150	530/B
112	417/T	132	410/T	151	650/T
113	457/2T	133	413/T	152	650/B
114	457/T	134	413/B	153	700/T
115	457/2B+1	135	833/2B+1	154	700/B
116	404/2B+1	136	304/T	155	703/T
117	404/T	137	304/B	156	703/B
118	404/2T	138	310/T	157	900/T
119	751/B	139	310/B	158	900/B
120	751/T				

T: Top seam B: Bottom seam

spontaneously combust are listed by BANERJEE (1985) as follows:

(i) the crossing-point temperature

(ii) the slope of stage II, and

(iii) the kick-point (transition) between stages II and III

7.1.1.1. Crossing-Point Temperatures

The crossing-point temperature can be defined as the temperature at which the coal temperature curve and the bath temperature curve intersect one another. A typical crossing-point temperature curve is shown in Figure 7.1.

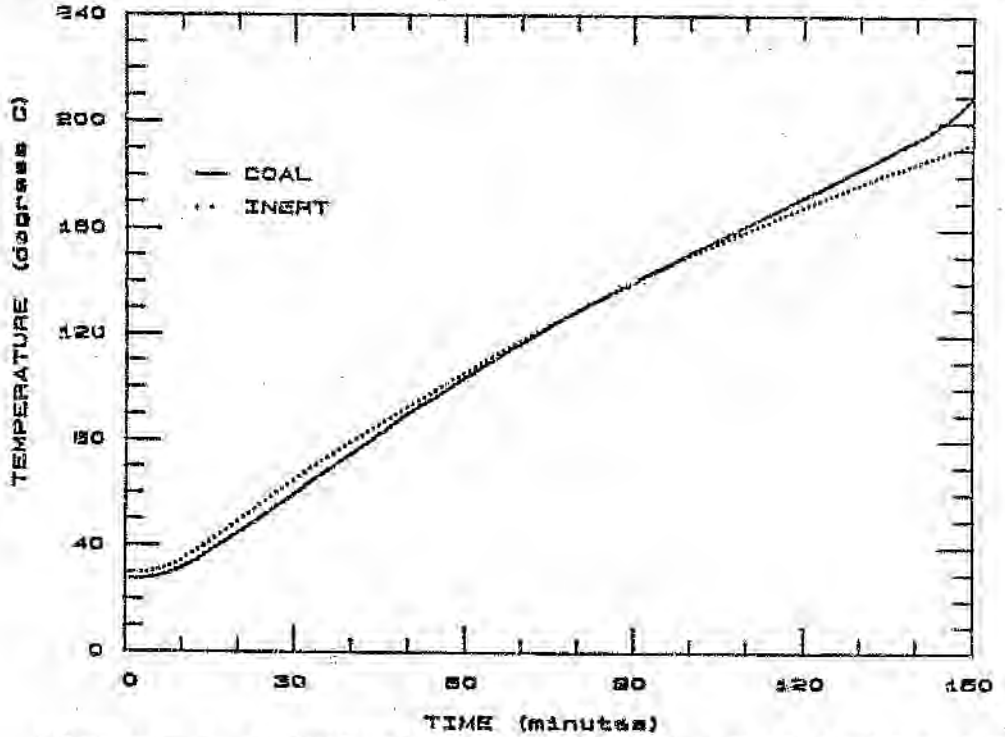

Figure 7.1 A typical crossing-point temperature curve

The crossing-point temperature curve is obtained by plotting the temperatures of the coal and the inert material against time.

It has been maintained by many researchers, as mentioned in Chapter 4.6, that a lower crossing-point temperature represents an increased liability to spontaneously combust. In Table 7.2 the crossing-point temperatures of the coal samples are listed in order of decreasing liability to spontaneous combustion. The crossing-point temperatures ranged from 102.2 to 154.1.

It is not always easy to read the crossing-point temperature from the crossing-point temperature curves due to the tangential approach of the coal and the inert temperature curve. Coals having low moisture content are particularly prone to this problem (Figure 7.2).

In the determination of the characteristics of the curves, D.T.A. curves are examined using a program called ANALYSIS (described in Chapter 6.6) using data from a .ZZZ file.

Table 7.2 Crossing - point temperatures of D.N.C. coal samples in descending order of risk

SAMPLE	XPT	SAMPLE	XPT	SAMPLE	XPT
134	102.2	117	135.7	124	142.6
140	109.1	146	136.9	121	142.7
130	121.3	152	138.2	138	142.9
135	122.0	112	138.3	126	144.8
128	123.7	111	138.8	105	145.1
155	125.2	144	138.8	115	145.4
141	125.6	145	138.8	119	145.4
147	126.0	150	139.1	122	145.4
153	126.9	154	139.5	120	145.5
116	127.0	149	139.6	139	147.3
143	12 .1	125	140.3	137	147.5
133	128.0	127	141.0	123	147.8
151	128.1	157	141.1	103	148.1
131	128.7	114	141.7	106	149.8
156	130.0	129	141.8	109	153.1
132	131.5	102	142.3	110	153.5
158	131.7	113	142.3	108	153.6
101	133.6	118	142.4	107	154.0
142	134.9	148	142.4	104	154.1
136	135.5				

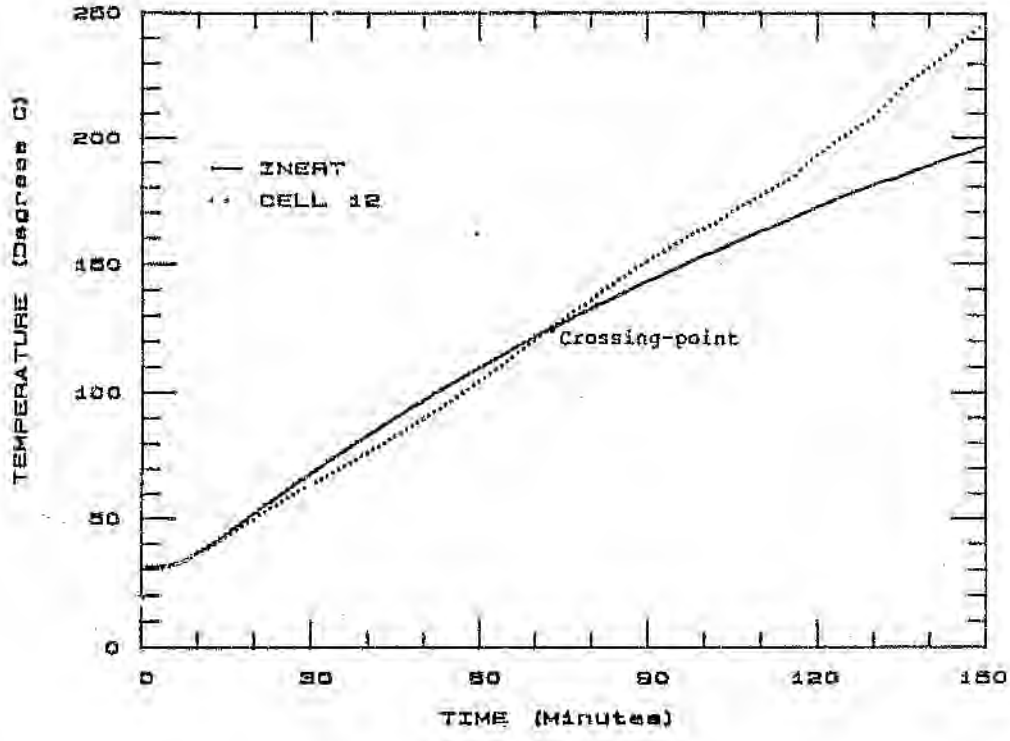

Figure 7.2 Tangentially approaching crossing-point curve

7.1.1.2. Differential Thermal Analysis

The stage II slope is one of the indicators of spontaneous combustion as was mentioned in Chapter 4.6. As it is alleged by BANERJEE (1985), that, the steeper the stage II slope, the more liable the coal to spontaneously combust. The stage II slope is the segment of the differential curve between the maximum negative differential point and the transition-point between stage II and stage III.

Table 7.3 Stage II slopes of D.N.C coal samples in descending order of risk

SAMPLE	STAGE II SLOPE	SAMPLE	STAGE II SLOPE	SAMPLE	STAGE II SLOPE
152	1.249874	157	1.136878	141	1.117763
124	1.198697	109	1.135802	102	1.116167
117	1.184650	123	1.135338	127	1.115522
125	1.171240	142	1.135191	112	1.114987
118	1.169405	122	1.134120	156	1.114832
126	1.163060	158	1.133498	108	1.113951
114	1.157576	129	1.133460	147	1.107488
145	1.156640	144	1.131129	153	1.100679
106	1.155074	154	1.129821	128	1.098456
107	1.150059	119	1.128514	133	1.097938
149	1.146608	151	1.127294	113	1.096819
148	1.143498	155	1.126685	134	1.088600
110	1.141278	143	1.126157	137	1.087571
121	1.140947	120	1.125767	139	1.076246
146	1.139821	101	1.125176	111	1.074839
150	1.138298	140	1.123340	130	1.068927
138	1.138202	132	1.123246	136	1.065030
116	1.137920	105	1.122826	131	1.031250
04	1.137405	135	1.122270		
103	1.137063	115	1.119580		

The stage II slopes from the thermograms of coals tested are listed in Table 7.3 in the order of decreasing liability of spontaneous combustion. The stage II slopes had a range from 1.031250 to 1.249874.

The kick-point represents the transition temperature from stage II to stage III. A lower kick-point is believed to indicate a greater liability to spontaneous combustion. Some of the samples have no kick-point for two reasons. Firstly, they showed no high exothermicity during the experiments. Secondly, they did not exhibit this transition point due to the inability of the test apparatus to reach a sufficiently high temperature (a maximum temperature of only 200°C for the heater/circulator). Kick-point temperatures as measured by GOUWS and WADE (1989) showed that this is an unreliable index and consequently they will not be examined further.

7.1.1.3. Comparison of Simple Indices

In the determination of the tendency of a coal sample to spontaneously combust, the crossing-point temperatures and stage II slopes of 58 samples can be compared.

The spontaneous combustion tendency of a coal increases with a lower crossing-point temperature and a steeper stage II slope. These factors individually, however, are not a reliable indicator of self-heating. While sample 134 has the lowest crossing-point temperature, it has the 7th lowest stage II slope of 58 coal samples. Contradictory results are also possible. Whilst sample 131 has the lowest stage II slope, it has the 14th lowest crossing-point temperature in 58 coal samples in Table 7.2 and Table 7.3.

After examining these results, therefore, the self-heating liability of a coal can be concluded to be some function of these characteristics. Composite liability indices which were developed by previous researchers will be investigated to determine the liability of a coal to spontaneously combust.

7.1.2. Composite Indices

Composite liability indices are used to develop an index in the determination of the tendency of a coal to undergo self-heating. Composite liability indices consist of two or more characteristics of a D.T.A. coal curve.

The major indices are, the FCC index developed by FENG, CHAKRAVORTY and COCHRANE (1973), the MR index developed by MAHADEVAN and RAMLU (1985) and the WITS-EHAC index developed by GOUWS (1987). The MR index will not be examined because of contradictory results due to fundamental mathematical problems. While Mahadevan and Ramlu stated that the index increases with decreasing liability, WADE and GOUWS (1990) found that the index increases with increasing liability. The FCC and WITS-EHAC indices will be examined in turn for D.N.C. coal samples.

7.1.2.1. The FCC Index

The FCC Index was proposed by FENG, CHAKRAVORTY and COCHRANE in 1973 in Canada, and is based on crossing-point temperature curves. The FCC Index is given as follows:

$$\text{Index} = \frac{\text{av. heat. rate between 110 to 220°C}}{\text{relative ignition temperature}} * 1000 \quad \ldots (11)$$

The term "relative ignition temperature" has the same meaning as "crossing-point temperature". The minimum temperature limit was chosen as 110°C to ensure that all moisture had evaporated. However, WADE and GOUWS (1990) found 5 South African coals in their research which had

crossing-points lower than 110°C. Two coal samples, in this research, also had crossing-point temperatures lower than 110°C (102.2 and 109.1°C). This was attributed to the use of identical sample holders which resulted in the elimination of the thermal gradients in the system. The index was modified as followings:

$$\text{FCC Index}_{mod} = \frac{\text{Stage II slope}}{\text{crossing-point temperature}} * 1000 \quad \ldots(12)$$

The decreasing liability of the D.N.C. coal samples to spontaneously combust, using the modified FCC index, is listed in Table 7.4. Modified FCC indices of the coal samples ranged from 10.62 to 7.26.

7.1.2.2. The WITS-EHAC Index

The WITS-EHAC index was developed by GOUWS in 1987. As many of the previous researchers, GOUWS concentrated on the D.T.A. curves and the effects of the crossing-point temperature and stage II slope. It is accepted that a lower crossing-point temperature and a steeper stage II slope indicates a higher risk for a coal to undergo self-heating.

Table 7.4 Modified FCC indices of D.N.C. coal samples in descending order of risk

SAMPLE	FCC INDEX	SAMPLE	FCC INDEX	SAMPLE	FCC INDEX
134	10.62	125	8.34	136	7.86
140	10.30	145	8.34	102	7.84
135	9.20	146	8.34	122	7.80
155	9.00	152	8.32	119	7.76
116	8.96	118	8.22	105	7.74
141	8.90	149	8.22	106	7.74
128	8.88	150	8.18	111	7.74
143	8.86	114	8.16	120	7.74
130	8.82	144	8.14	113	7.70
147	8.80	154	8.10	115	7.70
151	8.80	112	8.06	103	7.68
117	8.74	157	8.06	123	7.68
153	8.68	126	8.04	107	7.46
158	8.60	131	8.02	110	7.44
133	8.58	121	8.00	109	7.42
156	8.56	129	8.00	104	7.38
132	8.54	148	7.98	137	7.38
101	8.42	138	7.96	139	7.30
142	8.42	127	7.90	108	7.26
124	8.40				

These two characteristics were accepted as x and y axes. While the inverse of the crossing-point temperature values were marked on the y axis, the stage II slope values were marked on the x axis.

Crossing-point temperature and stage II slope values were joined with a straight line which forms a triangle (Figure 7.3). The area of the triangle was calculated from the well known trigonometrical formula:

$$\text{AREA} = 0.5 * \text{base} * \text{height} \quad \ldots\ldots(13)$$

$$= 0.5 * \text{stage II slope} * \frac{1}{\text{crossing-point}} \quad \ldots\ldots(14)$$

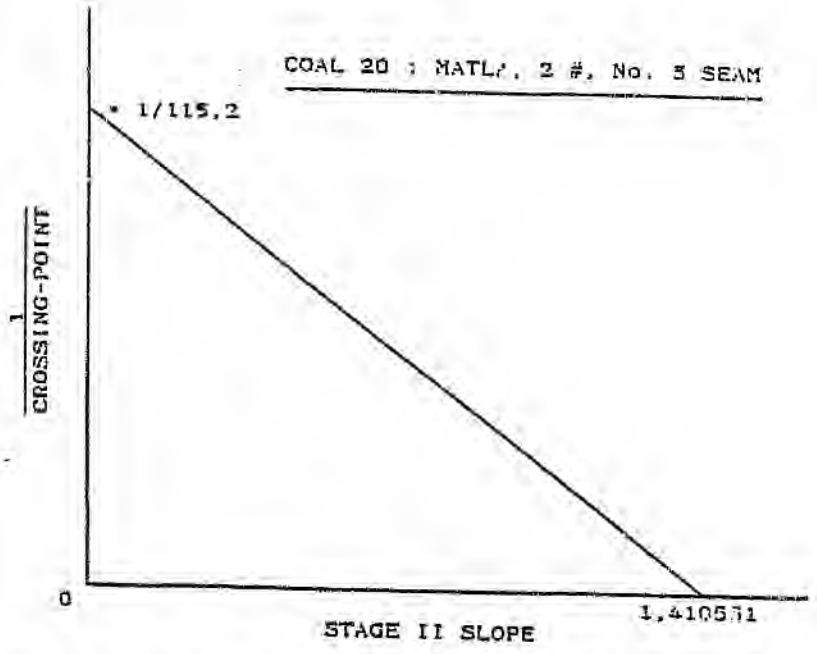

Figure 7.3 Triangle formed by joining the two characteristic values of a coal; after GOUWS (1987)

Higher index values represent the high risk of a coal to spontaneous combustion. The index value increases with increasing stage II slope and decreasing crossing-point temperature values.

The spontaneous combustion liabilities of D.N.C. coal samples, according to the WITS-EHAC index, are shown in Table 7.5. WITS-EHAC index values of the D.N.C. coal samples showed a range from 5.31 to 3.65.

7.1.3. Liability Contours

In the determination of the spontaneous combustion liabilities of the D.N.C. coal samples, two basic seams, top and bottom, were taken into consideration.

On completion of the experiments on 58 coal samples, 24 data points (WITS-EHAC values) for the bottom seam and 26 data points for the top seam were obtained. Two mine plans were contoured separately, using the WITS-EHAC indices of bottom and top seams respectively (Figure 7.4 and 7.5).

The x and y coordinates of the sample points and the mine boundaries, and the WITS-EHAC index values were entered into a program called LOTUS 1-2-3. These data values were

Table 7.5 WITS - EHAC indices of D.N.C. coal samples in descending order of risk

SAMPLE	WITS-EHAC INDEX	SAMPLE	WITS-EHAC INDEX	SAMPLE	WITS-EHAC INDEX
134	5.33	124	4.20	136	3.93
140	5.15	125	4.17	102	3.92
133	4.60	145	4.17	122	3.90
152	4.52	146	4.16	119	3.88
155	4.50	118	4.11	105	3.87
116	4.48	149	4.11	111	3.87
141	4.45	150	4.09	120	3.87
128	4.44	114	4.08	106	3.86
143	4.43	144	4.07	113	3.85
130	4.41	154	4.05	115	3.85
147	4.40	112	4.03	103	3.84
151	4.40	157	4.03	123	3.84
117	4.37	126	4.02	107	3.73
152	4.34	148	4.02	110	3.72
158	4.30	131	4.01	109	3.71
133	4.29	121	4.00	104	3.69
156	4.29	129	4.00	137	3.69
132	4.27	138	3.98	139	3.65
101	4.21	127	3.95	108	3.63
142	4.21				

then imported by a program, called SURFER.

The spontaneous combustion incidents that occurred in the last 50 years are shown in Figure 7.4 and 7.6. The liability contours of the bottom and top seam samples were obtained separately, using SURFER, to identify the areas having higher liabilities to self-heating (Figure 7.5 and 7.7). Figure 7.4 and 7.6 were plotted on transparency in order to compare the incident sites with the identified high risk areas.

In addition to the 2-dimensional contours of the bottom and top seams, 3-dimensional contour plans were obtained to identify the high risk areas in more detail (Figure 7.8 and 7.9).

While the WITS-EHAC index values of the bottom seam ranged from 3.65 to 5.31, the top seam values were in the range of 3.63 to 4.53. For the bottom and the top seam several areas prone to spontaneous combustion were identified (Fig 7.8 and 7.9).

The bottom seam samples showed greater liability to spontaneous combustion than the top seam samples. Only two top seam samples took place (4th and 10th order) in the descending order of risk of first ten samples (Table 7.5).

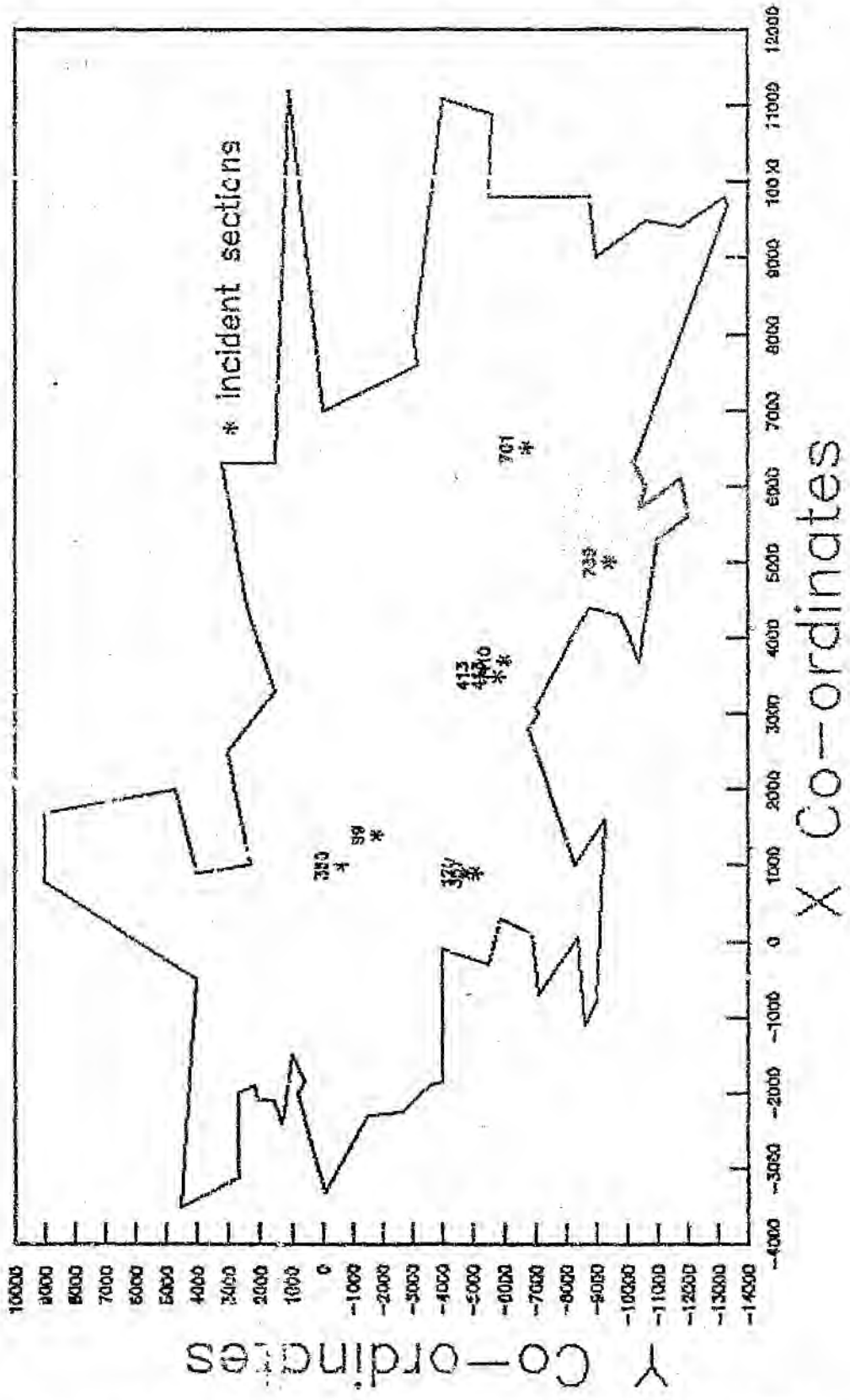

Figure 7.4 Sites of spontaneous combustion incidents

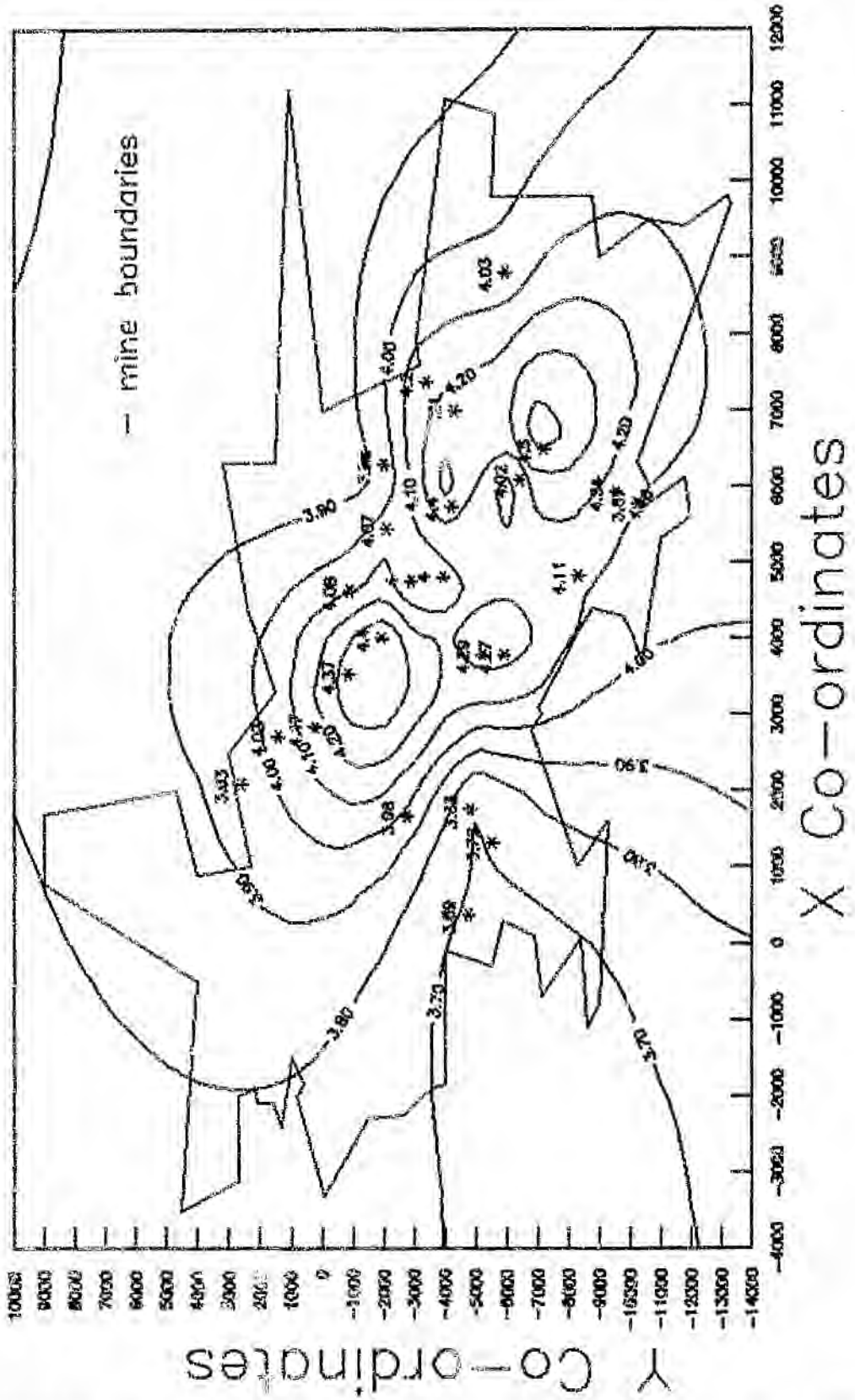

Figure 7.5 WITS-EHAC contours showing the D.N.C. top seam

Figure 7.6 Sites of spontaneous combustion incidents

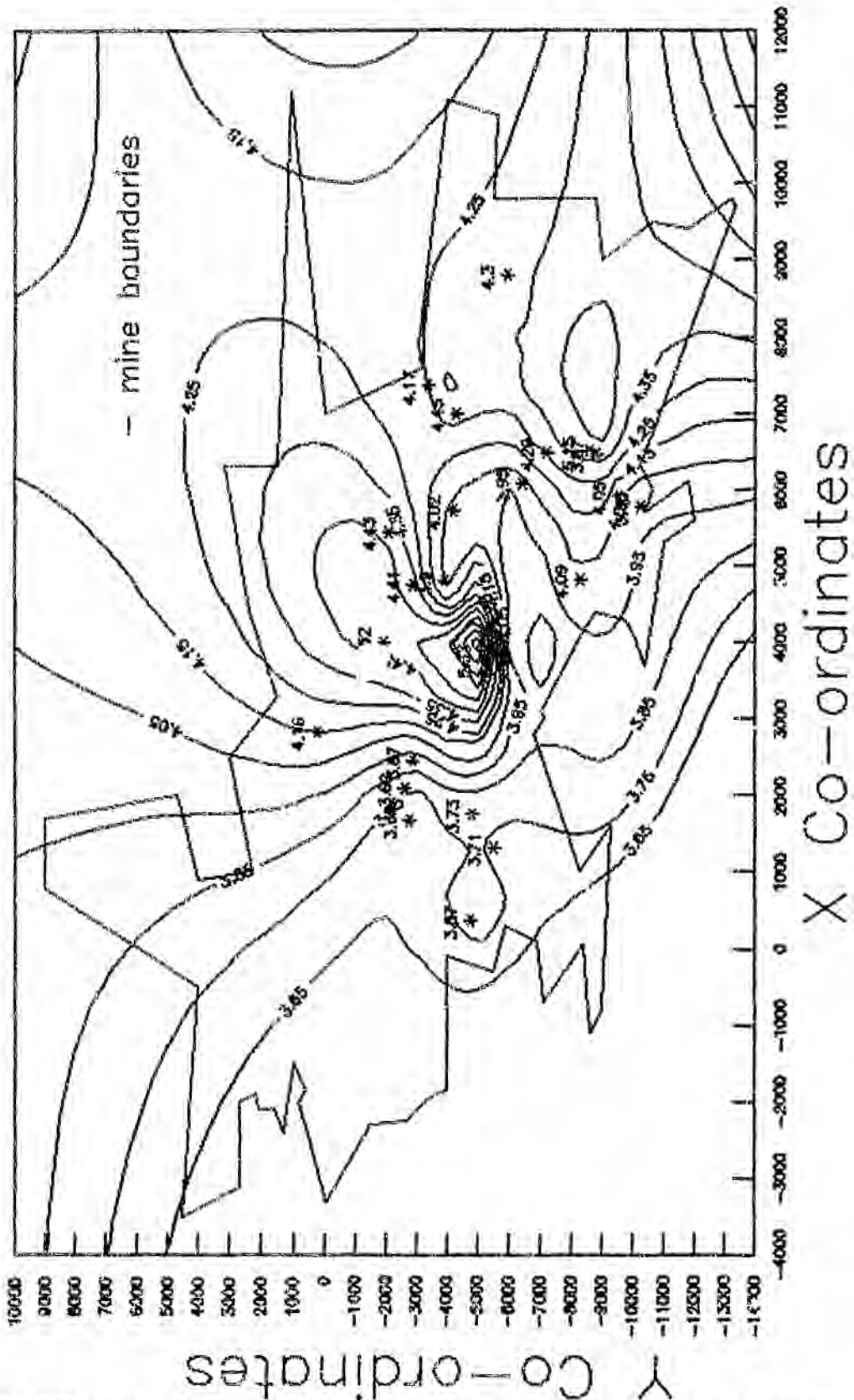

Figure 7.7 WITS-EHAC contours showing the D.N.C. bottom seam

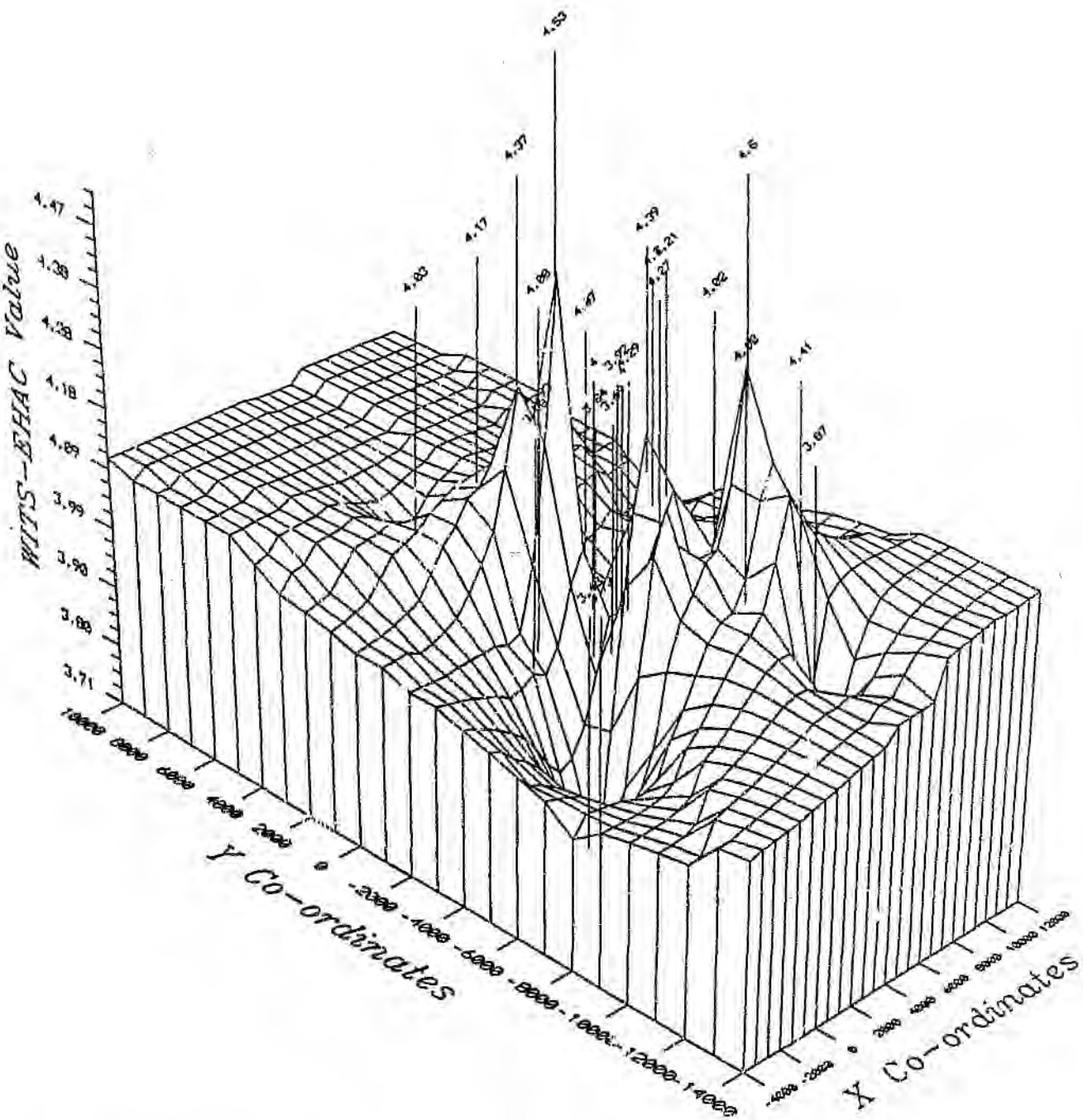

Figure 7.8 3-dimensional view of the WITS-EHAC index values of D.N.C. top seam samples

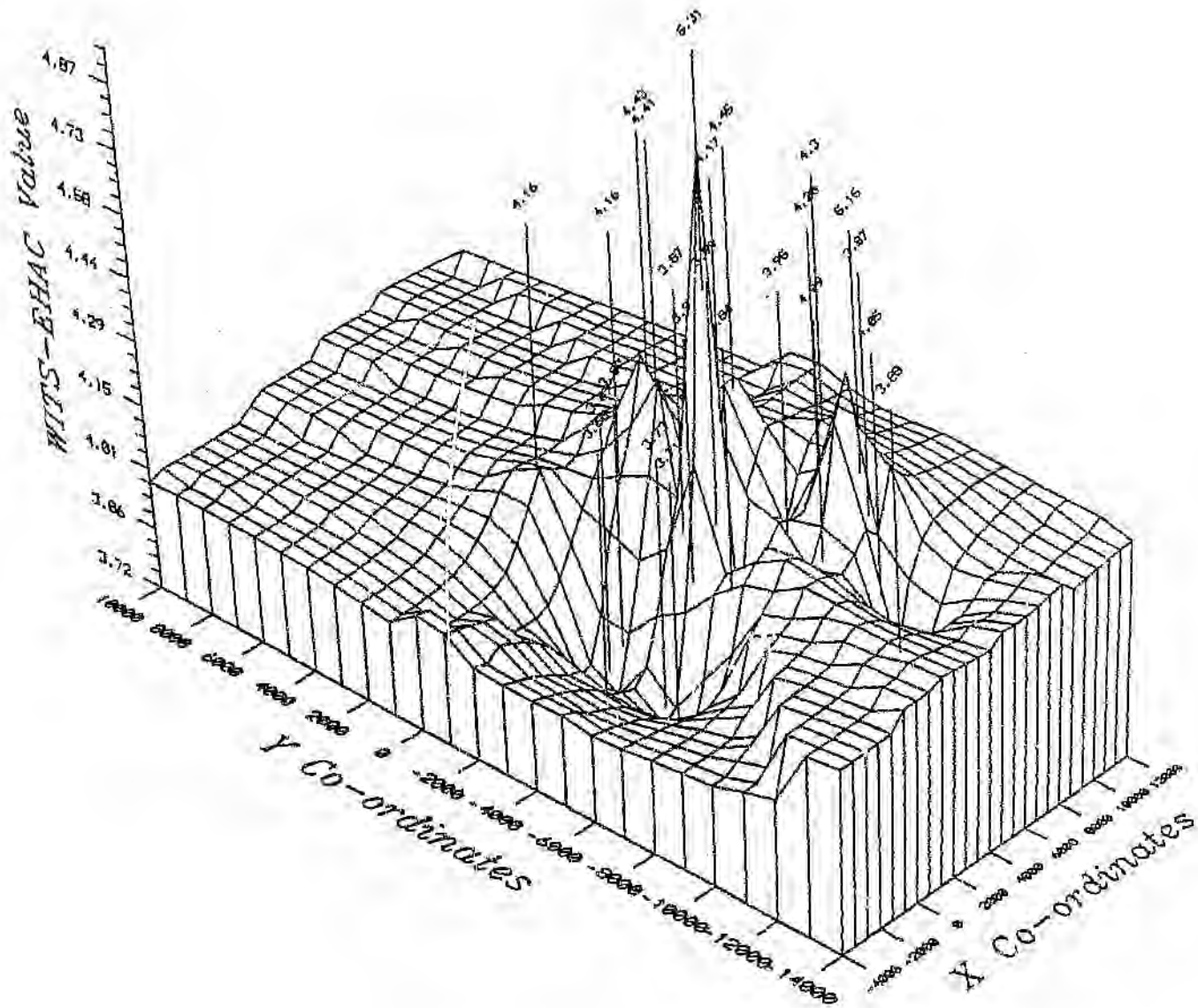

Figure 7.9 3-dimensional view of the WITS-EHAC index values of D.N.C. bottom seam samples

As can be seen from Table 7.5, sample 134 (which was taken from the bottom seam in section 413) showed the highest risk value. The records of the colliery revealed that an incident of spontaneous combustion occurred in this section in 1987 and 1989.

Sample 132 (top seam), which is the 17th in order of magnitude in Table 7.5 and the 7th in order of magnitude for top seam samples, was taken from section 410. The high risk index value assigned to this section has been confirmed by the records of the colliery in that an underground fire, initiated by spontaneous combustion, occurred in this section 1989.

All sites of past heatings could not be investigated for reasons of inaccessibility. However, the majority of the fires initiated by spontaneous combustion occurring over the 50 years have occurred in the areas that have been identified as having high WITS-EHAC index values (Figure 7.4, 7.5, 7.6 and 7.7).

7.1.4. Statistical Analysis

It is expected that the correlations of proximate, ultimate and petrographic analyses against the liability index will be low because of having the coal samples with

similar rank values. This, in fact, is shown to be true in the subsequent statistical analyses.

The results of the proximate, ultimate and petrographic analyses were compared with the crossing-point temperatures, stage II slopes and the WITS-EHAC index values in order to determine the contributory factors of self-heating. A programme, called STATGRAPHICS, was used for the statistical regressions.

The results of the statistical regressions are given in Table 7.7, 7.8 and 7.9. Some abbreviations were used in these tables. The key to these abbreviations is given in Table 7.6. These abbreviations are also used for the results of the proximate, ultimate and petrographic analyses in Appendix I, II, III, IV and V.

Four different types of simple regression models were conducted for each regression. The best model was then accepted for each parameter. These simple regression models are shown with symbols, L, M, E and R, in Table 7.7, 7.8 and 7.9. These symbols represent the following simple regression models:

L	Linear	$Y = a + bx$
M	Multiplicative	$Y = ax^b$
E	Exponential	$Y = \exp(a + bx)$
R	Reciprocal	$1/Y = a + bx$

Table 7.6 Key to abbreviations

Symbol	Parameter	Symbol	Parameter
Moist	Moisture content	RSF	Reactive semi-fusinite
Ash	Ash content	ISF	Inert semi-fusinite
Vol	Volatile matter	SF	Total semi-fusinite
Fix.C	Fixed carbon	F	Fusinite
CV	Calorific value	Mic	Micronite
P	Phosphorus	RNT	Reactive inertodetrinite
C	Elemental carbon	INT	Inert inertodetrinite
H	Hydrogen content	NT	Inertodetrinite
S	Sulphur content	Inert	Inertinite
O	Oxygen content	RoV	Reflectance of Vitn
O/C	Oxygen/Carbon	Vit/C	Vitrite/clarite
Pyr.S	Pyritic sulphur	IM	Intermediates
Sulp.S	Sulphate sulphur	S/Fus	S/Fusite
Org.S	Organic sulphur	Iint	Inertodentrite
Roga	Roga index	Carbo	Carbominerite
Swell	Swelling index		
Vitn	Vitrinite		
Exin	Exinite		

7.1.4.1. Crossing-Point Temperature Statistics

The results of the correlations of the crossing-point temperature against the independent variables are shown

Table 7.7 Crossing-point temperature statistics

VARIABLE	MODEL	R	VARIABLE	MODEL	R
Moist	M	0.06	Exin	L	-0.11
Ash	R	-0.05	RSF	L	0.32
Vol	R	0.17	ISF	L	0.05
Fix.C	M	0.16	SF	L	0.16
CV	R	0.04	F	L	-0.25
P	M	-0.12	Mic	L	0.03
C	L	0.23	RNT	R	-0.18
H	R	0.19	INT	R	-0.08
N	M	-0.16	NT	R	-0.18
S	R	-0.12	Inert	E	0.22
O	L	-0.48	RoV	M	0.05
Pyr.S	R	-0.08	Vit/C	L	-0.20
Sulp.S	R	-0.05	IP	L	0.24
Org.S	L	0.12	S/Fus	L	0.04
Roga	R	0.22	Iint	R	-0.14
Swell	M	-0.17	Carbo	R	0.06
Vitn	M	-0.22			

in Table 7.7.

Although no satisfactory correlations were determined, oxygen, reactive semi-fusinite, fusinite and elemental carbon showed a slight relationship with crossing-point temperatures. Crossing-point temperatures are lower for coals having high oxygen and fusinite contents and low reactive semi-fusinite and carbon contents. However, the correlations were unsatisfactory. The best fitted correlation, which was oxygen content, is shown in Figure 7.10.

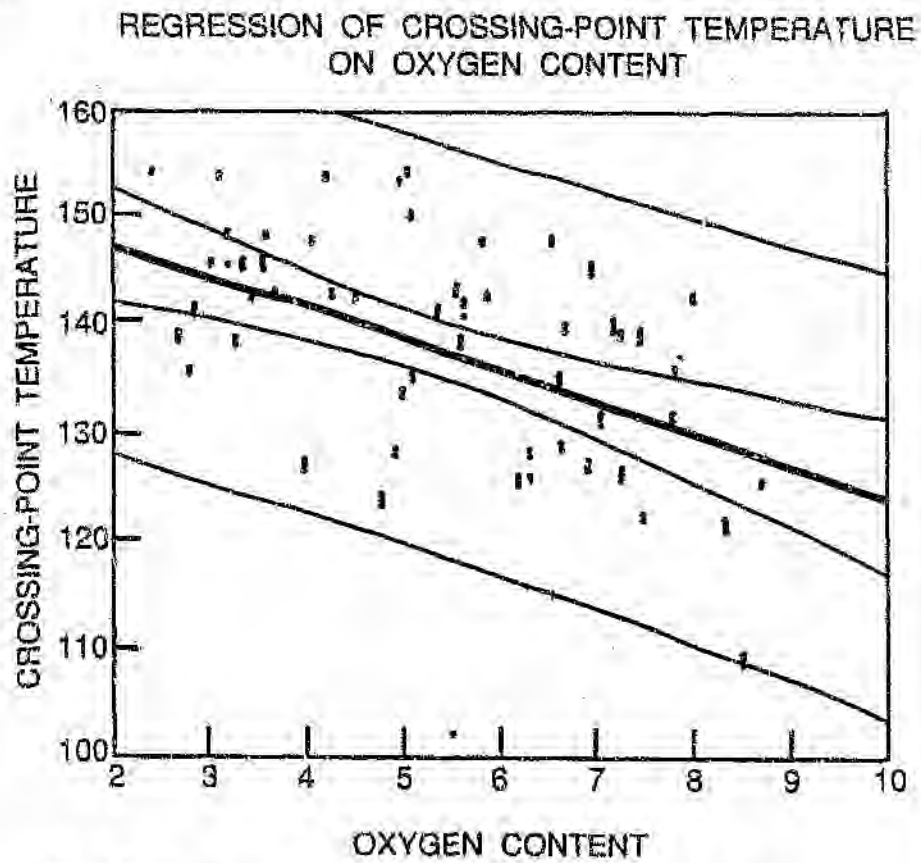

Figure 7.10 Regression of crossing-point temperature on oxygen content

The equation derived from the regression was:

Crossing-point temperature = 153.2 + (-2.9 * Oxygen)

7.1.4.2. Stage II Slope Statistics

Regression results of the stage II slope on independent variables are shown in Table 7.8.

Stage II slope statistics gave better results than the crossing-point temperature statistics. According the regression results, sulphate sulphur, moisture, inertodetrinite, roga index, inert inertodetrinite, sulphur and fusinite showed a slight relationship with stage II slopes. Stage II slopes are lower for coal samples having a high roga index and a low sulphate sulphur, moisture, inertodetrinite, inert inertodetrinite, sulphur and fusinite contents. The best fitted correlation, which was sulphate sulphur, is shown on Figure 7.11.

The equation derived from the linear regression was:

Stage II Slope = 1.11 + (0.13 * Sulphate Sulphur)

Table 7.8 Stage II slope statistics

VARIABLE	MODEL	R	VARIABLE	MODEL	R
Moist	L	0.41	Exin	L	0.13
Ash	M	0.08	RSF	M	0.07
Vol	R	0.23	ISF	R	-0.11
Fix.C	M	0.17	SF	R	-0.12
CV	L	-0.16	F	R	0.37
P	R	-0.04	MIc	L	-0.01
C	L	-0.05	RNT	R	-0.27
H	R	0.20	INT	M	0.39
N	L	0.14	NT	M	0.41
S	M	0.38	INERT	R	-0.29
O	L	0.16	RoV		
Pyr.S	R	0.23	VIT/C	M	-0.35
Sulp.S	L	0.51	IM	M	0.27
Org.S	L	0.06	S/FUS	M	0.07
Roga	M	-0.40	Iint	R	-0.31
Swell	M	-0.35	CARBO	L	0.16
Vitn	M	-0.34			

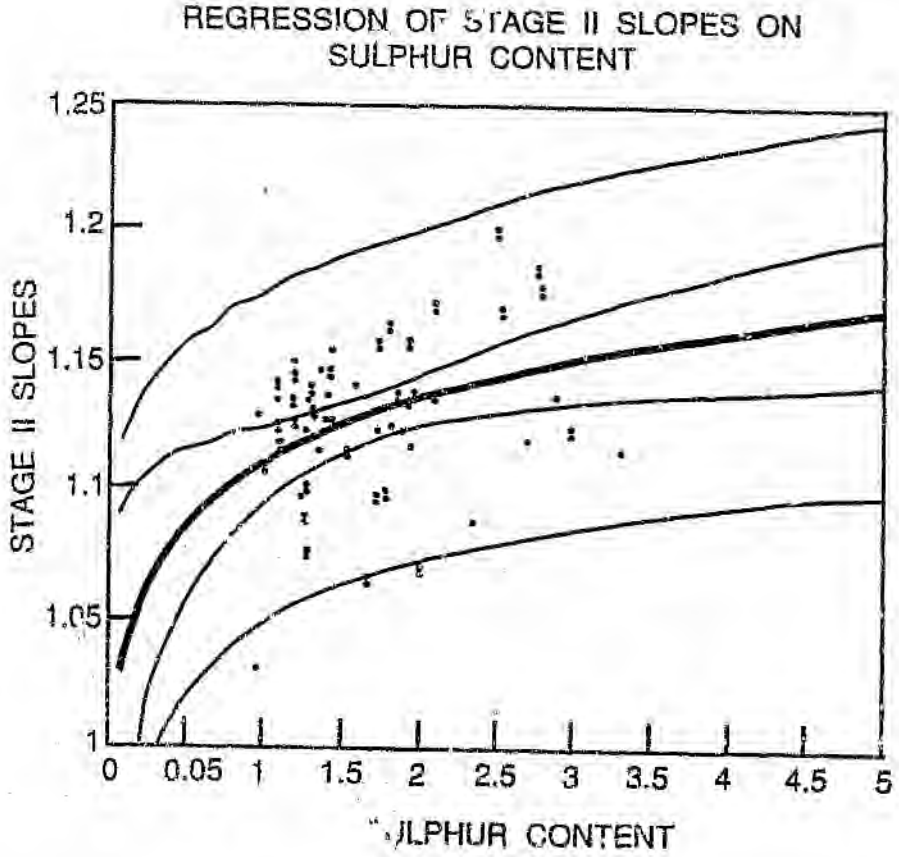

Figure 7.11 Regression of stage II slope on sulphate sulphur content

7.1.4.3. WITS-EHAC Index Statistics

The correlation coefficients of the regressions of the WITS-EHAC indices against the independent variables are given in Table 7.9.

No significant correlations were determined, as was the case with the crossing-point temperature and the stage II

Table 7.9 WITS-EHAC index statistics

VARIABLE	MODEL	R	VARIABLE	MODEL	R
Moist	L	0.04	Exin	R	-0.13
Ash	L	-0.02	RSF	R	0.31
Vol	L	0.08	ISF	R	-0.02
Fix.C	L	-0.07	SF	R	0.11
CV	L	-0.02	F	M	0.15
P	M	0.12	MIC	R	0.02
C	R	0.26	RNT	L	-0.08
H	L	0.12	INT	M	0.11
N	M	0.11	NT	L	-0.06
S	L	-0.02	INERT	L	-0.10
O	R	-0.44	RoV	M	-0.01
Pyr.S	M	0.05	VIT/C	L	0.06
Sulp.S	M	0.17	IM	R	0.17
Org.S	M	-0.11	S/FUS	M	0.01
Roga	L	0.08	Iint	M	0.04
Swell	L	0.04	CARBO	M	-0.04
Vitn	L	0.09			

slope. Oxygen, reactive semi-fusinite and elemental carbon showed a slight relationship. Although having a low correlation coefficient of the oxygen content, the nighest regression value, was -0.44. Its graphical

display is shown in Figure 7.12. The formula derived from the regression was:

1 / WITS-EHAC Index = 0.26 * (-4.05 E-3 * Oxygen)

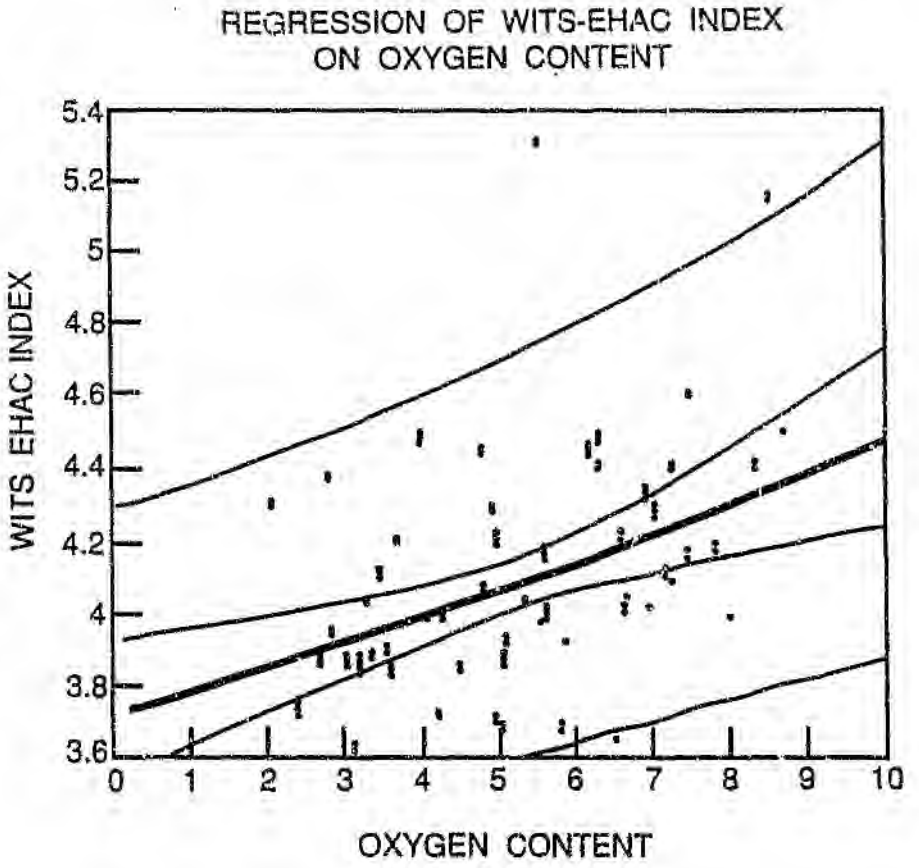

Figure 7.12 Regression of WITS-EHAC index on oxygen

7.1.4.4. Comparison of Statistical Analysis

Poor results from the statistical analysis of the crossing-point temperature, the stage II slope and the WIT5-EHAC index oriented the research towards multiple regression. It has already been stated that spontaneous combustion occurs as a result of the aggregate effect of different factors.

No effective multiple regression results were achieved and it was decided to investigate the relationship between the rank and the vitrinite content of the samples (Figure 7.13).

Vitrinite is the most brittle and most reactive maceral group to the oxidation reaction. The reactivity of a coal sample depends on the rank and the vitrinite content of the sample. Two coal samples having similar rank values could show different liabilities to spontaneous combustion according to their vitrinite contents (FALCON; 1985 and FALCON; 1986). As can be seen from Figure 7.11, while the rank values of the coal samples, apart from sample 157 and 158, had a very narrow range, the vitrinite content of the samples varied in a wide range from 15.2 to 73.3. Working with coal samples having a similar rank values made it impossible to get reasonable regression results as was expected before the regression.

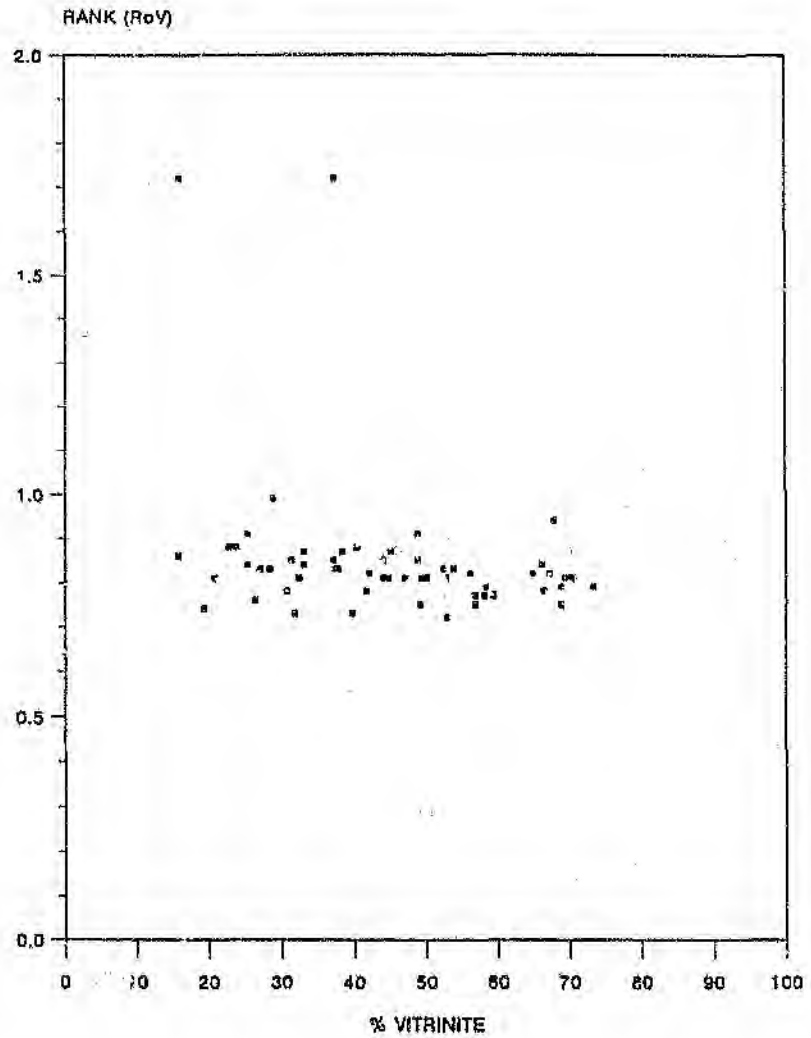

Figure 7.13 Rank distribution against vitrinite content

Satisfactory regression results may have been obtained if the samples had widely varying rank values.

7.2. Adiabatic Calorimeter Results

The use of the adiabatic calorimeter to obtain WITS-EHAC index is discussed in this section of the thesis. The ignition temperature test results are, then, compared to adiabatic calorimeter test results. A comparison of the results obtained from the two different testing techniques shows a good relationship between them.

7.2.1. Kinetics and The WITS-EHAC Index

The adiabatic calorimeter test results were obtained as explained in Section 6.6.2. The Arrhenius equation was used to examine the results:

$$w\ C_p\ {}^{dT}/_{dt} = w\ Q\ Z\ e^{-E/RT} + \text{conduction term} \quad \ldots\ldots\ldots\ldots (15)$$

where:

w = density

C_p = specific heat of coal at constant temperature

T = temperature (Kelvin)

t = time

Q = heat of reaction

e = exponential (natural log)

E = activation energy

R = molar gas constant

Z = a specific rate constant

Under adiabatic conditions the conduction term can be neglected, and the formula can be simplified (GOUWS; 1992):

$$C_p \, {}^{dT}/_{dt} = Q \, Z \, e^{E/RT} \quad \ldots\ldots(16)$$

If $A = QZ/C_p$, it can be written as follows:

$${}^{dT}/_{dt} = A \, e^{-E/RT} \quad \ldots\ldots(17)$$

$$\ln {}^{dT}/_{dt} = {}^{-E}/_{RT} + \ln A \quad \ldots\ldots(18)$$

The activation energy can be found from the slope of the Arrhenius plot (Figure 6.6). The slope is equal to $-{}^{E}/_{R}$, where R is the molar gas constant which is equal to 8,32 J/g mole. The point where the extrapolated curve intersects the vertical axis ($\ln {}^{dT}/dt$) is ln A and is called the frequency factor. A higher frequency factor (ln A) represents a higher liability of a coal to spontaneously combust (BANERJEE; 1985).

After these findings, the WITS-EHAC index, could be rewritten as:

$$\text{WITS-EHAC} = {}^{E}/_{RPT} * \text{constant} \quad \ldots\ldots(19)$$

During early spontaneous combustion tests, the crossing-point temperature of the coal samples were determined on temperature versus time graphs because it was an easy characteristic to identify. Researchers concentrated on the crossing-point temperature tests because they were not able to identify the temperature at which the exothermic reaction commences. The crossing-point temperature of a coal sample, however, is not the exclusive means of predicting the liability of a coal to spontaneously combust.

It would, of course, be far better to use the starting temperature of the exothermic reaction instead of the crossing-point temperature. The Arrhenius plot, which was explained in Section 6.6.2. (Figure 6.6), was used to identify this point. It is a simple matter to identify the point where the horizontal curve changes to an increasing vertical trend. It is believed that this is the point referred to by BANERJEE (1985), who called this point the "Threshold point". This point can be defined as the minimum temperature where the exothermic reaction begins in the coal body.

After these findings, the crossing-point temperature was replaced with the threshold temperature (Ttemp) in the WITS-EHAC index and was written as:

WITS-EHAC Index = $^{E}/_{T_{temp}}$ * constant.....(20)

E, however, corresponds to $^{\log(A)}/_{(1/T_{temp})}$, and the WITS-EHAC Index can be rewritten as in the following:

WITS-EHAC Index $\sim \log(A)$.....(21)

7.2. Comparison of Ignition-Temperature Tests and Adiabatic Calorimeter Results

Thirty-one coal samples were tested with the adiabatic calorimeter in order to simulate the ignition-temperature test apparatus. During the selection of the thirty-one coal samples, the WITS-EHAC index values of all the coal samples, obtained from the ignition-temperature tests, were taken into consideration. As can be seen from the Table 7.5, some coals had the same WITS-EHAC index values according to the crossing-point temperature tests. Coal samples, having different WITS-EHAC index values, were chosen for the adiabatic calorimeter tests. The WITS-EHAC index values of these samples were calculated according to the new index formula explained in Section 7.2.1. (formula 21).

After testing thirty-one coal samples, it was found that sample 101 and 102 had very low index values according to

the trend showed with the other coal samples. The experiments of coal 101 and 102 were examined again and it was found that while the experiments of the other coal samples were stopped at 190°C, the experiments with coal 101 and 102 were stopped at 180°C. It was decided to re-test and heat these two samples until 190°C. Expected results were achieved after the two experiments were stopped at the same temperature with the other coal samples.

The results of the WITS-EHAC index from the adiabatic calorimeter and the WITS-EHAC index from the ignition-temperature tests are shown in Table 7.10.

The results obtained from the adiabatic calorimeter, generally, were lower than the results obtained from the ignition-temperature tests. It was, then, decided to compare the results obtained from these two different techniques. A linear regression analysis showed a good relationship between the results of these two techniques (Figure 7.14).

A linear correlation of the New WITS-EHAC index (the frequency factor) from the adiabatic calorimeter against the WITS-EHAC index from the ignition-temperature apparatus yields a correlation (R) of 88%.

Table 7.10 Results from the adiabatic calorimeter and the ignition-temperature test

Sample No	WITS EHAC	NEW WITS EHAC	Sample No	WITS EHAC	NEW WITS EHAC
101	4.21	4.11	127	3.95	3.78
102	3.92	3.90	132	4.27	4.08
103	3.84	3.68	134	5.33	4.59
104	3.69	3.55	135	4.60	4.33
105	3.87	3.63	138	3.98	3.75
106	3.86	3.74	139	3.65	3.48
108	3.63	3.24	140	5.15	4.39
110	3.72	3.90	141	4.45	4.14
112	4.03	3.71	146	4.16	4.03
114	4.08	4.17	147	4.40	4.25
117	4.37	4.31	152	4.52	4.17
118	4.11	3.86	153	4.34	4.16
121	4.00	3.78	154	4.05	4.05
122	3.90	3.88	155	4.50	4.22
124	4.20	4.05	158	4.30	4.11
125	4.17	4.05			

After the linear regression of the WITS-EHAC index values, the WITS-EHAC index formula can be rewritten as follows:

WITS-EHAC Index = -0.459575 + 1,16631 (ln A).....(22)

or,

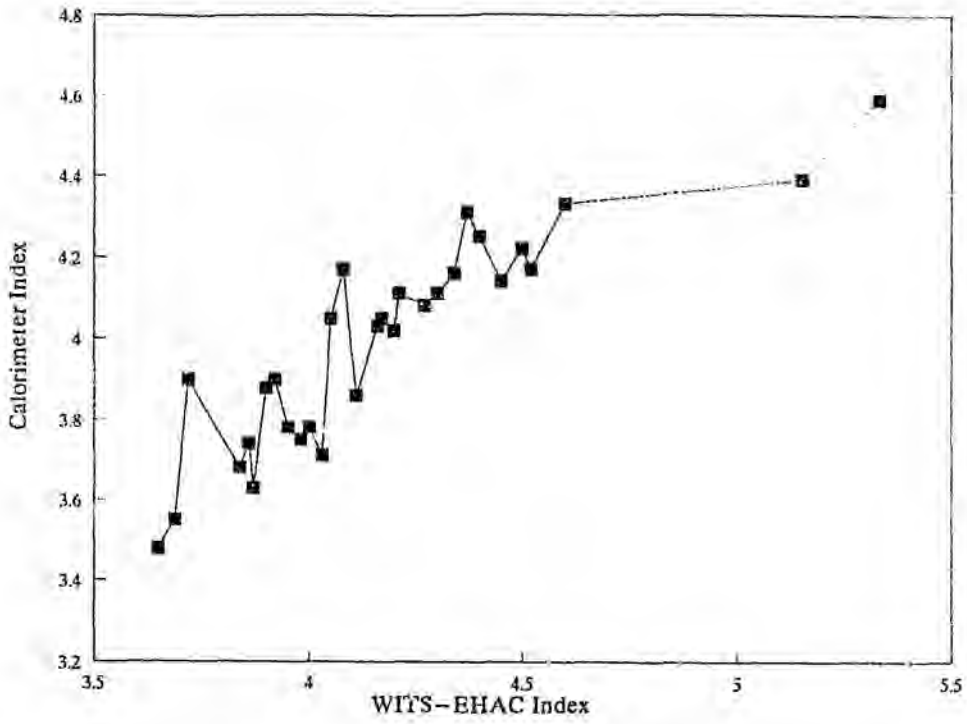

Figure 7.14 Comparison of WITS-EHAC index values obtained using the ignition-temperature apparatus and the adiabatic calorimeter

$$\ln(A) = 1.20282 + 0.663379\ (\text{WITS-EHAC Index}) \quad \ldots\ldots\ldots (23)$$

The relationship between the WITS-EHAC index and the frequency factor (ln A) supported the idea, mentioned previously, that the frequency factor is a very strong indicative of a coal to self-heat.

The good correlation between the index values of the different testing apparatus supported the idea that the adiabatic calorimeter can be used to determine the liability of a coal to spontaneously combust as well as

the ignition-temperature apparatus. It, also, succeeded in simulating the ignition-temperature testing technique.

The good relationship made it possible to use the formula to estimate the calorimeter index for the coal samples which were tested in the ignition-temperature test apparatus. The formula used for the calculation is:

Calorimeter Index = ln (A).....(24)

Calorimeter Index = 1.20282 + 0.663379 (WITS-EHAC Index).....(25)

The calculated calorimeter index values of the coal samples tested in the ignition-temperature test apparatus are listed in Table 7.11.

Table 7.11 The calorimeter index and the WITS-EHAC index values of the D.N.C. samples

Samp. No	CAL. INDEX	WITS EHAC	Samp. No	CAL. INDEX	WITS EHAC	Samp. No	CAL. INDEX	WITS EHAC
101	4.00	4.21	121	3.86	4.00	140	4.62	5.15
102	3.80	3.92	122	3.79	3.90	141	4.15	4.45
103	3.75	3.84	123	3.77	3.84	142	4.00	4.21
104	3.65	3.69	124	3.99	4.20	143	4.14	4.43
105	3.77	3.87	125	3.97	4.17	144	3.90	4.07
106	3.76	3.86	126	3.87	4.02	145	3.97	4.17
107	3.68	3.73	127	3.82	3.95	146	3.96	4.16
108	3.61	3.63	128	4.15	4.44	147	4.12	4.40
109	3.66	3.71	129	3.85	4.00	148	3.87	4.02
110	3.67	3.72	130	4.13	4.41	149	3.93	4.11
111	3.77	3.87	131	3.86	4.01	150	3.92	4.09
112	3.88	4.03	132	4.04	4.27	151	4.12	4.40
113	3.76	3.85	133	4.05	4.29	152	4.20	4.52
114	3.91	4.08	134	4.74	5.33	153	4.08	4.34
115	3.76	3.85	135	4.25	4.60	154	3.89	4.05
116	4.17	4.48	136	3.81	3.93	155	4.19	4.50
117	4.08	4.37	137	3.65	3.69	156	4.05	4.29
118	3.93	4.11	138	3.84	3.98	157	3.88	4.03
119	3.78	3.88	139	3.62	3.65	158	4.06	4.30
120	3.77	3.87						

8. CONCLUSION

The reseach conducted in this thesis was used to contour the liabilities of Durban Navigation Colliery seams to spontaneously combust in order to identify high risk areas and to determine the relationship between the WITS-EHAC index and the coal properties.

For a better understanding of the problem, a detailed review of the literature was conducted. The history and hazards to the mining industry were examined. The importance of the spontaneous combustion of coal was revealed by the number of incidents occurring in South African collieries in past 20 years. The detection and prevention of spontaneous combustion was also investigated.

The factors contributing to spontaneous combustion of coal are:

a) Coal factor,

b) Geological factor, and

c) Mining factor.

Coal and geological factors are called intrinsic factors because they cannot be altered. The mining factor, however, is extrinsic. This means that by altering the mining factor the overall risk of an incident could be

decreased. The coal factor should be known in order to alter the mining factor.

For a better selection of testing technique, theories of spontaneous combustion of coal were examined. Various spontaneous combustion testing techniques were compared with each other and their advantages and disadvantages were noted. Ignition temperature tests and the adiabatic calorimetry were found to be the more appropriate testing techniques in the determination of the spontaneous combustion liabilities of coal samples. It was decided to use the ignition temperature determinations in the first phase of the research. The adiabatic calorimeter was used in the second phase of the thesis. The ignition temperature determinations included:

(i) Crossing-point temperature tests, and

(ii) Differential thermal analysis.

These tests were carried out simultaneously with an apparatus designed in the Mining Engineering Department of the University of the Witwatersrand. Some of the components of the appr atus were changed to improve accuracy and ease of use.

Fifty-eight coal samples were collected from Durban Navigation Colliery to determine the WITS-EHAC indices of these samples and to identify the areas having a high

risk to spontaneously combust.

In the evaluation of the results, modified FCC and WITS-EHAC index values were calculated. The derivation of the modified FCC index was described in Chapter 7. The evaluation of the results was based on the WITS-EHAC index values. The index values were plotted on the mine plan at the coordinates of the relevant sample sources, and contours were generated.

The behaviour of the samples taken from the top and bottom seam showed different index values. While the top seam sample index values ranged from 4.50 to 3.63, bottom seam samples had index values between 5.33 to 3.65. This large difference enabled values to be contoured and enabled the identification of areas more liable to self-heating. The bottom seam samples, generally, showed a greater liability to spontaneously combust than samples obtained from the top seam. The records of the colliery revealed that some of the spontaneous combustion incidents occurred in the areas identified more liable to self-heating. These areas can be seen in Figure 7.5, 7.7, 7.8 and 7.9.

WITS-EHAC index values were correlated against the results of the proximate, ultimate and petrographic analysis in order to find any relationship between the

test results and the coal properties. No satisfactory regression results were obtained because of working with coal samples having similar rank values.

It was, then, decided to simulate the ignition temperature test apparatus. The adiabatic calorimeter, which was designed and constructed in the Department of Mining Engineering of the Witwatersrand University, was used for this purpose. The adiabatic calorimeter has three test options:

- incubation test,
- minimum sel-heating temperature test, and
- crossing-point temperature test.

The adiabatic calorimeter was commissioned as a replacement for the ignition temperature test apparatus. The advantages of the calorimeter, when compared with the ignition temperature test apparatus, are:

- test duration is similar to ignition temperature test,
- requires very little time for cleaning and sample preparation,
- it is fully automatic, and
- requires no supervision at all.

It was decided to use the incubation mode of the calorimeter to simulate the tests conducted with the ignition temperature test apparatus. In the ignition

temperature tests, the crossing-point temperature could be identified. It is possible to identify the temperature at which the exothermic reaction commences with the adiabatic calorimeter. This point was called "Threshold temperature" by BANERJEE (1985) and can be defined the minimum temperature where the exothermic reaction begins.

Thirty-one coal samples from Durban Navigation Colliery, having different WITS-EHAC values obtained from the ignition temperature tests, were chosen for the adiabatic calorimeter tests. The ignition temperature test results were compared to the results obtained from the adiabatic calorimeter.

The adiabatic calorimeter confirmed the ignition temperature test results.

A linear regression analysis showed a good relationship between these two techniques. A linear correlation of the frequency factor (from the adiabatic calorimeter), against the WITS-EHAC index (from the ignition temperature test apparatus), yields a correlation coefficient of 88%. The relationship between the WITS-EHAC index and the frequency factor supports the idea that the frequency factor is a very strong indicator of a coal to self-heat. It is, also, shown that the adiabatic calorimeter can be used to determine the

liability of a coal to spontaneously combust as well as the ignition temperature apparatus.

In future work, experiments should focus on the working areas having a greater liability to self-heat. Conditions were held constant during the experiments for this thesis. The conditions affecting the spontaneous combustion of coal, like air flow rate, humidity, coal particle size etc., should be investigated.

APPENDICES

APPENDIX I: PROXIMATE ANALYSIS RESULTS

SAMPLE	MOIST.	ASH	VOL	FIX C
101	1.3	18.7	26.4	53.6
102	1.1	19.3	29.4	50.2
103	1.1	17.2	24.5	57.2
104	0.9	10.9	31.3	56.9
105	1.0	12.7	32.0	54.3
106	1.5	12.0	33.9	52.6
107	0.9	12.0	32.3	54.8
108	1.0	14.8	30.3	53.9
109	1.4	16.2	25.9	56.5
110	1.1	15.2	25.0	58.7
111	1.0	14.5	28.3	56.2
112	1.4	17.8	30.8	50.0
113	1.3	20.5	29.5	48.7
114	1.5	15.6	26.9	56.0
115	1.2	13.6	34.5	50.7
116	1.1	17.5	34.4	47.0
117	1.1	19.7	25.4	53.8
118	1.6	18.0	26.8	53.6
119	1.1	14.4	25.0	59.5
120	0.9	12.5	27.8	58.8
121	0.9	18.2	28.3	52.6
122	1.3	13.1	34.2	51.4
123	1.1	16.3	27.4	55.2
124	1.1	15.2	25.0	58.7
125	1.2	17.8	27.7	53.3
126	1.1	10.8	28.9	59.2
127	1.0	14.3	33.2	51.5
128	1.2	16.2	31.4	51.2
129	1.0	10.1	34.7	54.2
130	0.9	16.2	34.7	48.2
131	1.1	22.1	33.0	43.8
132	1.1	10.9	31.2	56.8
133	0.9	9.4	33.3	56.4
134	0.9	10.8	32.8	55.5
135	1.5	16.4	35.2	46.9
136	1.1	9.9	32.8	56.2
137	0.9	8.5	37.5	53.1
138	1.1	18.5	24.7	55.7
139	1.1	12.6	28.5	57.8
140	0.8	12.1	31.4	55.7
141	1.2	14.6	33.5	50.7
142	1.3	16.8	27.4	54.5

SAMPLE	MOIST.	ASH	VOL.	FIX C
143	1.4	13.0	30.9	54.7
144	1.8	21.9	27.5	48.8
145	1.7	15.7	27.2	55.4
146	1.3	13.8	33.7	51.2
147	1.5	12.3	26.6	59.6
148	1.6	14.4	30.9	53.1
149	1.2	11.3	28.4	59.1
150	1.1	6.4	31.6	60.9
151	1.6	17.0	32.0	49.4
152	1.8	12.7	34.9	50.6
153	1.1	14.6	26.7	57.6
154	1.1	11.8	30.1	57.0
155	1.5	13.3	28.4	56.8
156	1.2	16.4	28.8	53.6
157	2.0	18.5	12.1	67.4
158	1.6	17.1	11.4	69.9

APPENDIX II: ULTIMATE ANALYSIS RESULTS

SAMPLE	C.V.	P	C	H	N
101	27.38	0.295	67.71	3.83	1.68
102	26.48	0.464	66.99	3.95	1.68
103	28.30	0.010	70.13	3.85	1.63
104	31.48	0.025	75.48	4.49	1.79
105	30.98	0.015	74.75	4.65	1.83
106	29.85	0.006	73.39	4.86	1.77
107	30.95	0.016	75.99	4.62	1.85
108	29.73	0.069	73.35	4.32	1.88
109	28.50	0.014	70.12	4.01	1.91
110	29.14	0.029	72.42	4.08	1.91
111	29.88	0.052	74.26	4.36	1.92
112	28.01	0.091	67.93	4.41	1.87
113	26.56	0.283	66.23	3.96	1.81
114	28.65	0.008	70.34	3.92	1.92
115	29.71	0.021	72.09	4.76	2.07
116	28.67	0.010	68.69	4.76	2.01
117	27.54	0.038	68.08	3.83	1.72
118	27.94	0.207	68.69	3.96	1.77
119	29.58	0.013	74.12	4.19	1.88
120	30.72	0.037	76.18	4.19	2.01
121	28.18	0.011	69.02	4.14	1.90
122	30.17	0.022	73.15	4.86	2.14
123	28.94	0.012	71.76	4.23	1.93
124	28.81	0.043	71.71	3.92	1.89
125	28.21	0.016	67.41	4.01	1.87
126	30.78	0.005	73.21	4.23	1.91
127	29.88	0.007	73.64	4.82	2.04
128	28.85	0.011	69.79	4.40	1.86
129	31.15	0.095	75.36	4.56	2.08
130	28.97	0.140	66.03	4.62	1.92
131	26.98	0.012	63.04	4.33	1.82
132	30.78	0.017	72.58	4.45	1.92
133	31.87	0.010	76.45	4.92	2.18
134	31.27	0.029	74.55	4.87	2.10
135	28.45	0.015	65.2	4.50	1.94
136	31.41	0.035	75.32	4.84	2.10
137	32.35	0.007	76.18	4.18	2.09
138	28.33	0.124	64.83	3.60	1.64
139	30.12	0.009	72.12	4.42	1.94
140	30.73	0.060	70.47	4.58	1.82
141	28.82	0.011	69.6	4.53	1.95
142	28.21	0.324	67.41	4.06	1.92

SAMPLE	C.V.	P	C	H	N
143	29.84	0.016	71.46	4.56	2.07
144	25.77	0.599	63.82	3.92	1.70
145	28.02	0.016	67.65	4.02	1.73
146	29.37	0.013	68.35	4.69	1.94
147	29.60	0.152	71.88	4.14	1.91
148	29.03	0.022	68.23	4.55	2.02
149	30.46	0.007	72.57	4.49	1.90
150	32.38	0.038	77.04	4.84	2.07
151	27.35	0.206	67.6	4.15	1.91
152	29.44	0.015	70.7	4.50	2.03
153	29.62	0.104	70.03	4.20	1.88
154	33.07	0.042	72.4	4.72	1.99
155	29.33	0.026	68.96	4.30	1.86
156	28.74	0.101	67.99	4.41	1.87
157	27.15	0.078	68.52	2.19	1.59
158	28.15	0.077	72.85	2.20	2.20

SAMPLE	S	C	PYR. S	SULP. S	ORG. S
101	1.81	4.97	1.11	0.103	0.60
102	1.11	5.87	0.55	0.011	0.55
103	2.89	3.2	2.11	0.007	0.77
104	1.41	5.03	0.46	0.040	0.91
105	1.88	3.19	1.01	0.021	0.85
106	1.43	5.05	0.28	0.402	0.75
107	2.24	2.4	1.19	0.010	1.04
108	1.53	3.12	0.58	0.024	0.93
109	1.42	4.94	0.54	0.086	0.79
110	1.09	4.2	0.24	0.048	0.80
111	1.27	2.69	0.20	0.007	1.06
112	3.31	3.28	2.30	0.234	0.78
113	1.72	4.48	1.08	0.100	0.54
114	1.93	1.79	1.19	0.222	0.52
115	2.70	3.58	1.62	0.141	0.94
116	1.96	3.98	1.17	0.010	0.78
117	2.77	2.8	2.06	0.069	0.64
118	2.54	3.44	1.62	0.261	0.66
119	0.97	3.34	0.10	0.010	0.86
120	1.20	3.02	0.23	0.003	0.97
121	1.58	4.26	0.62	0.052	0.91
122	1.92	3.53	0.79	0.199	0.93
123	1.09	3.59	0.37	0.014	0.71
124	2.51	3.67	1.45	0.189	0.87
125	2.10	5.61	1.22	0.010	0.87
126	1.80	6.95	0.81	0.086	0.90
127	1.35	2.85	0.46	0.014	0.88
128	1.78	4.77	0.81	0.076	0.89
129	1.29	5.61	0.29	0.024	0.98
130	2.00	8.33	1.08	0.017	0.90
131	0.97	6.64	0.29	0.007	0.67
132	1.27	7.78	0.30	0.103	0.87
133	1.24	4.91	0.28	0.007	0.95
134	1.26	5.52	0.50	0.010	0.75
135	2.98	7.48	1.74	0.213	1.03
136	1.66	5.08	0.60	0.014	1.05
137	2.35	5.8	1.17	0.024	1.16
138	4.79	5.54	3.94	0.096	0.75
139	1.29	6.53	0.55	0.065	0.68
140	1.72	8.51	0.53	0.065	1.13
141	1.94	6.18	1.15	0.120	0.67
142	1.90	6.61	1.10	0.110	0.69

SAMPLE	S	O	PYR. S	SULP. S	ORG. S
143	1.21	6.3	0.45	0.127	0.63
144	2.80	4.06	0.37	0.481	1.95
145	1.74	7.46	1.08	0.326	0.33
146	2.10	7.82	1.06	0.210	0.83
147	1.01	7.26	0.37	0.192	0.45
148	1.20	8	0.03	0.333	0.84
149	1.36	7.18	0.33	0.185	0.85
150	1.30	7.25	0.04	0.069	1.19
151	1.43	6.31	0.61	0.299	0.52
152	2.68	5.59	1.25	0.491	0.94
153	1.27	6.92	0.21	0.079	0.98
154	1.32	6.67	0.10	0.072	1.15
155	1.39	8.69	0.21	0.292	0.89
156	1.09	7.04	0.23	0.134	0.73
157	1.86	5.34	0.73	0.495	0.64
158	1.19	2.08	0.27	0.168	0.75

SAMPLE	ROGA	SWELL.
101	20	2.5
102	20	2
103	24	1
104	68	4
105	75	5
106	71	6.5
107	88	6.5
108	72	5
109	17	2
110	23	2.5
111	63	6
112	46	6.5
113	29	3.5
114	22	1
115	77	8
116	69	6.5
117	22	1
118	23	1
119	33	3.5
120	51	5.5
121	27	2
122	78	6
123	41	4
124	8	1
125	23	2
126	37	3
127	78	5
128	75	4.5
129	81	6
130	73	5.5
131	78	5.5
132	65	5
133	77	4.5
134	86	5
135	72	7.5
136	80	6.5
137	86	7.5
138	22	1
139	34	2.5
140	71	6.5
141	67	7
142	22	2

SAMPLE	ROGA	SWELL
143	36	3
144	17	1.5
145	18	1
146	74	6.5
147	19	1
148	56	6.5
149	28	2.5
150	76	7.5
151	38	3
152	69	7
153	25	2.5
154	69	7.5
155	28	3.5
156	45	4.5
157	Nill	Nill
158	Nill	Nill

APPENDIX III: PETROGRAPHIC (MACERAL) ANALYSIS

SAMPLE	VITRINITE	EXINITE	RSF	ISF	F
101	48.7	4	10.3	16	4.7
102	48.7	3.7	7.3	6.7	1.3
103	23.7	3	12.3	10.7	1
104	41.7	2.3	15.3	17	4.3
105	44.7	7	13.3	14	5.3
106	67.7	6.7	6	6	3.3
107	66	4	8.3	9.3	4
108	50	3	13.7	16	4.7
109	40.3	4.7	20	12.3	0.7
110	25.3	3	9.7	16.7	1.3
111	44	3.7	9	14.3	4
112	42	6.7	5	11	3
113	31.7	12.3	7	16	6.7
114	33	4.3	8.7	6.7	2
115	59.3	4	6	8	5
116	67	7.7	4.3	3.3	6.7
117	28.3	1.3	12	11	4
118	25.3	2.3	14.3	12.7	1
119	28.7	0.3	15.3	19.7	2.3
120	45	2	17	20.7	3
121	30.7	3.7	1.7	5.3	1.7
122	66.3	5	6.7	7	2.3
123	31.3	2	20.7	27	1.7
124	15.7	4.7	9.7	22	2.7
125	32.3	6.7	8.7	24.7	3
126	37.3	2.7	9.7	7.7	4
127	56.7	9	3.7	10.3	4.3
128	58.2	6.7	5.7	4.7	3.7
129	49	7	7	12.7	3.3
130	44	2	7.3	19.3	5.7
131	73.3	2.3	3.3	9.3	10
132	52.7	8	11.3	10	3.7
133	49.3	3	16.7	17.7	3.3
134	52.7	4	9	18.7	4
135	69.3	9.3	4	6.7	2.7
136	64.7	3.7	10	6.3	7.7
137	68.7	0.3	19	6.3	3.7
138	26.3	2	12	25.7	2.7
139	33	2.7	13	25	4.7
140	52.2	4.7	5.4	17.1	4.3
141	70.2	2.7	8.4	6.7	2.3
142	38.3	3.7	11	10	7.7

SAMPLE	VITRINITE	EXINITE	RSF	ISF	F
143	47	4.7	15	14.7	4.3
144	20.7	4.7	15	37.3	4
145	37	4.7	10.3	18	4
146	68.7	2.3	7	6.7	7
147	22.7	2.3	10.3	13.7	1
148	56	4.7	7.3	12.3	7
149	19.3	0.7	16.7	28	1.3
150	53.7	2.7	10.7	21	3.7
151	39.7	6.3	4.7	16.7	2.3
152	58.7	8.7	6.7	10	5
153	37.7	2.3	11.7	17.3	8.7
154	58	3.7	7.3	12.3	6.3
155	27	3	11.3	22.3	3.7
156	47	7	12	15	6
157	16	0	3.3	56.7	3.3
158	37.3	0	0.7	45.7	4.7

SAMPLE	MIC	RNT	INERT	INERTOD	INERTINI
101	1.7	6.3	8.3	14.6	47.3
102	4.3	12.3	15.7	28	47.6
103	1.7	39.3	8.3	47.6	73.3
104	3.3	5.3	10.7	16	55.9
105	2.3	4.3	9	13.3	48.2
106	2.7	1.3	6.3	7.6	25.6
107	1.7	1	5.7	6.7	30
108	1.7	4.7	6.3	11	47.1
109	6.7	5.7	9.7	15.4	55.1
110	1.7	36.3	6	42.3	71.7
111	1.7	12.7	10.7	23.4	52.4
112	3.3	8	21	29	51.3
113	5	7.7	13.7	21.4	56.1
114	2	28	15.3	43.3	62.7
115	3.3	4	10.3	14.3	36.6
116	2.7	2	6.3	8.3	25.3
117	3	29.7	10.7	40.4	70.4
118	2.3	31	11	42	72.3
119	1	18.7	14	32.7	71
120	1	4.3	7	11.3	53
121	2	32.7	22.3	55	65.7
122	3	2	7.7	9.7	28.7
123	2	9.3	6	15.3	66.7
124	0.7	25.7	19	44.7	79.8
125	3	8.3	13.3	21.6	61
126	1.7	28.7	8.3	37	60.1
127	4.3	3.7	8	11.7	34.3
128	1.3	14.4	5.4	19.8	35.2
129	2.3	5.7	13	18.7	44
130	2.7	5.7	13.3	19	54
131	0.7	0	1	1	24.3
132	5.7	1.3	7.3	8.6	39.3
133	0.7	4.7	4.7	9.4	47.8
134	3	2.7	6	8.7	43.4
135	2	0.3	5.7	6	21.4
136	1	3.3	3.3	6.6	31.6
137	0.3	1.3	0.3	1.6	30.9
138	0.7	25.3	5.3	30.6	71.7
139	0.7	14	7	21	64.4
140	3.3	1.3	11.7	13	43.1
141	4	1.7	4	5.7	27.1
142	3.3	18.3	7.7	26	58

SAMPLE	MIC	RNT	INERT	INERTOD	INERTINIT
143	2.7	7.3	4.3	11.6	48.3
144	0	12.7	5.7	18.4	74.7
145	1.3	14	10.7	24.7	58.3
146	1.3	2	5	7	29
147	1	35.7	13.3	49	75
148	4	1.3	7.3	8.6	39.2
149	0	26	8	34	80
150	0	2.3	6	8.3	43.7
151	0.7	16.3	13.3	29.6	54
152	0.7	2	10.3	12.3	34.7
153	0.3	11.7	10.3	22	60
154	1	4	7.3	11.3	38.2
155	0.3	23.7	8.7	32.4	70
156	0.7	4.7	7.7	12.4	46.1
157	0	4.3	16.3	20.6	83.9
158	0	0.3	11.3	11.6	62.7

APPENDIX IV: PETROGRAPHIC (MICROLITHOTYPE) ANALYSIS

SAMPLE	VIT/CLAR	IMEDIATE	S/FUS	INERTO	CARBO	Ce	Te	De
101	41.8	32.1	17.7	3.3	5	0	0	0
102	30.8	33.8	15.1	15.1	5.4	0	0	0
103	18.3	27.7	14.3	35.7	4	0	0	0
104	32.3	52	13.7	1	1	0	0	0
105	35.8	29.8	25.8	5.4	3.3	0	0	0
106	62.3	25.7	8	1.3	2.7	0	0	0
107	44	35.3	11.3	1.7	7.7	0	0	0
108	37	35.3	20.7	2.7	4.3	0	0	0
109	25.7	34.7	23	12.3	4.3	0	0	0
110	15.7	28.3	21	29.3	5.7	0	0	0
111	26	32	24	12	6	0	0	0
112	37.7	25.7	11.7	20	3.3	0.7	0.7	0.3
113	23.7	31.3	22.3	13.7	8.3	0	0	0.7
114	23.3	32.3	12.3	25.3	6.3	0	0	0.3
115	51	28	5.3	2.7	10.3	0	1	1.7
116	50.7	27.3	7.3	0.7	13	0.7	0	0.3
117	12.3	28	20.3	33.3	5.7	0	0	0.3
118	10	32.7	21	33	3.3	0	0	0
119	16	31	41.3	6.7	5	0	0	0
120	21.7	38	29.3	7	4	0	0	0
121	13	41	14	23.3	8.7	0	0	0
122	37.3	44.3	10	3.3	2	1	1	1
123	19.7	45.7	27.7	3.3	3.7	0	0	0
124	7.3	23	26.3	37.7	5.3	0	0	0.3
125	28.7	27.	26.7	13.7	2.3	0	0.3	0.7
126	15.9	41.7	15.5	26.9	0	0	0	0
127	60.5	27.4	7	1.3	3	0	0.7	0
128	50.7	24.7	11.3	10	1.7	0.3	0.7	0.7
129	47.3	23.7	14.3	5.7	8.7	0	0	0.3

SAMPLE	VIT/CLAR	IMEDIATE	S/FUS	INERTO	CARBO	Ce	Te	De
130	44.3	31.3	18.3	4	2	0	0	0
131	76.3	9.3	8.7	0	5.7	0	0	0
132	50.7	27	10.7	3	4.7	1	3	0
133	41.3	30.3	21	4	1.7	0	1	0.7
134	34.7	36.3	20.3	5	1	0	2	0.7
135	66.7	18.3	5.3	2	5.7	0.7	0.7	0.7
136	49.3	30.3	14	2.7	3.7	0	0	0
137	70.7	19	7	0.7	2.7	0	0	0
138	19.7	16.7	22	31	10.3	0	0.3	0
139	22	42.3	23.3	10.7	0.7	0	1	0
140	35.3	39	17.3	4	4	0	0.3	0
141	56	29.3	10	1	3.7	0	0	0
142	18	41.3	19.3	16.7	4	0.7	0	0
143	37	32.7	23.3	5.7	1	0	0.3	0
144	16.7	38	29.3	10	6	0	0	0
145	21.7	35.3	20.3	17.7	3.7	0.3	1	0
146	60.3	24.7	7.7	2.3	4	0.7	0.3	0
147	8	21.3	24.3	44.3	2	0	0	0
148	50.3	29.3	14.3	3.3	2.7	0	0	0
149	10.3	43.7	12	32.7	0.7	0	0.7	0
150	57.7	28.3	12.3	0	1.7	0	0	0
151	33	25.7	17	13	9.3	0	0.3	1.7
152	48	29.7	10.7	1.3	8.7	1	0.3	0.3
153	19	26.7	34.3	11.3	8.7	0	0	0
154	46.3	32	14.3	2.7	4.7	0	0	0
155	19.3	29.7	28.3	15.7	7	0	0	0
156	46.3	27	11.3	6.7	7.3	1	0.3	0
157	16	46.7	23.7	9	4.7	0	0	0
158	21	34.7	34.3	7.7	2.3	0	0	0

APPENDIX V: REFL[illegible]NCE DISTRIBUTION (V CLASSES)

SAMPLE	RoV
101	0.91
102	0.85
103	0.88
104	0.78
105	0.81
106	0.94
107	0.84
108	0.81
109	0.88
110	0.91
111	0.85
112	0.82
113	0.73
114	0.84
115	0.77
116	0.82
117	0.83
118	0.84
119	0.99
120	0.87
121	0.78
122	0.78
123	0.85
124	0.86
125	0.81
126	0.83
127	0.77
128	0.79
129	0.75
130	0.81
131	0.79
132	0.72
133	0.81
134	0.81
135	0.81
136	0.82
137	0.75
138	0.76
139	0.87
140	0.83
141	0.81
142	0.87

SAMPLE	RoV
143	0.81
144	0.81
145	0.85
146	0.79
147	0.88
148	0.82
149	0.74
150	0.83
151	0.73
152	0.75
153	0.83
154	0.77
155	0.83
156	0.81
157	1.72
158	1.72

APPENDIX VI: SPLITFIX PROGRAM LISTING

```
10 REM ***
20 REM ***** SPLIT1
30 REM ***** Splits captured data for thermometer set 1
40 REM *****  Uses  several  tangents  to  represent
   calibration curve
50 REM ***
60 KEY OFF
70 CLS
80 INPUT "WHAT DATA FILE IS TO BE SPLIT"; DF$
90 LONGDF$ = DF$ + ".DAT"
100 OPEN LONGDF$ FOR INPUT AS #1
110 OPDF$ = DF$ + ".ooo"
120 LPRINT "FILE :"; OPDF$
130 PRINT
140 OPEN OPDF$ FOR OUTPUT AS #2
150 LINE INPUT #1, X$
160 PRINT X$
170 DIM X(20)
180 FOR I = 1 TO 7
190 INPUT #1, X(I)
200 PRINT X(I);
210 NEXT I
220 PRINT : PRINT
230 PRINT "  PRT1  PRT2  PRT3  PRT4  PRT5  PRT6  PRT13
    TIME"
```

```
240 PRINT "  ====  ====  ====  ====  ====  ====  =====
    ====="
250 LPRINT "  PRT1  PRT2  PRT3  PRT4  PRT5  PRT6  PRT3
    TIME"
260 LPRINT "  ====  ====  ====  ====  ====  ====  =====
    ====="
270 FOR J = 1 TO 2000
280 '
290 INPUT #1, AA, BB, CC, DD, EE, FF, GG
300 INPUT #1, AA, BB, CC, DD, EE, FF, GG
310 INPUT #1, AA, BB, CC, DD, EE, FF, GG
320 INPUT #1, A, B, C, D, E, F, G
330 IF A < 35 THEN NEWA = A * 28.8 / 30
340 IF A > 35 THEN NEWA = A * 39.4 / 40.4
350 IF A > 45 THEN NEWA = A * 48.8 / 50
360 IF A > 55 THEN NEWA = A * 59.5 / 60.7
370 IF A > 65 THEN NEWA = A * 68.9 / 70.2
380 IF A > 75 THEN NEWA = A * 79 / 80.4
390 IF A > 85 THEN NEWA = A * 88.5 / 90
400 IF A > 95 THEN NEWA = A * 99 / 100.7
410 IF A > 105 THEN NEWA = A * 108.7 / 110.6
420 IF A > 115 THEN NEWA = A * 118.3 / 120.3
430 IF A > 125 THEN NEWA = A * 127.8 / 130.1
440 IF A > 135 THEN NEWA = A * 137.6 / 140.1
450 IF A > 145 THEN NEWA = A * 147.4 / 150.3
460 IF A > 155 THEN NEWA = A * 157 / 160.1
470 IF A > 165 THEN NEWA = A * 167.1 / 170.5
```

```
480 IF A > 175 THEN NEWA = A * 178.6 / 182.4
490 IF A > 185 THEN NEWA = A * 186.4 / 190.6
500 IF A > 195 THEN NEWA = A * 194.4 / 199.1
510 IF B < 35 THEN NEWB = B * 28.8 / 30
520 IF B > 35 THEN NEWB = B * 39.4 / 40.9
530 IF B > 45 THEN NEWB = B * 48.8 / 50.1
540 IF B > 55 THEN NEWB = B * 59.5 / 61
550 IF B > 65 THEN NEWB = B * 68.9 / 70.5
560 IF B > 75 THEN NEWB = B * 79 / 80.6
570 IF B > 85 THEN NEWB = B * 88.5 / 90.3
580 IF B > 95 THEN NEWB = B * 99 / 100.8
590 IF B > 105 THEN NEWB = B * 108.7 / 110.8
600 IF B > 115 THEN NEWB = B * 118.3 / 120.4
610 IF B > 125 THEN NEWB = B * 127.8 / 130.3
620 IF B > 135 THEN NEWB = B * 137.6 / 140.3
630 IF B > 145 THEN NEWB = B * 147.4 / 150.5
640 IF B > 155 THEN NEWB = B * 157 / 160.3
650 IF B > 165 THEN NEWB = B * 167.1 / 170.7
660 IF B > 175 THEN NEWB = B * 178.6 / 182.5
670 IF B > 185 THEN NEWB = B * 186.4 / 190.8
680 IF B > 195 THEN NEWB = B * 194.4 / 199.4
690 IF C < 35 THEN NEWC = C * 28.8 / 29.6
700 IF C > 35 THEN NEWC = C * 39.4 / 40.1
710 IF C > 45 THEN NEWC = C * 48.8 / 49.5
720 IF C > 55 THEN NEWC = C * 59.5 / 60.4
730 IF C > 65 THEN NEWC = C * 68.9 / 69.8
740 IF C > 75 THEN NEWC = C * 79 / 79.7
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750 IF C > 85 THEN NEWC = C * 88.5 / 89.5
760 IF C > 95 THEN NEWC = C * 99 / 100.1
770 IF C > 105 THEN NEWC = C * 108.7 / 110
780 IF C > 115 THEN NEWC = C * 118.3 / 119.6
790 IF C > 125 THEN NEWC = C * 127.8 / 129.4
800 IF C > 135 THEN NEWC = C * 137.6 / 139.3
810 IF C > 145 THEN NEWC = C * 147.4 / 149.7
820 IF C > 155 THEN NEWC = C * 157 / 159.6
830 IF C > 165 THEN NEWC = C * 167.1 / 169.7
840 IF C > 175 THEN NEWC = C * 178.6 / 181.7
850 IF   > 185 THEN NEWC = C * 186.4 / 189.9
860 IF     195 THEN NEWC = C * 194.4 / 198.3
880 IF D < 35 THEN NEWD = D * 28.8 / 28.9
890 IF D > 35 THEN NEWD = D * 39.4 / 39.6
900 IF D > 45 THEN NEWD = D * 48.8 / 48.9
910 IF D > 55 THEN NEWD = D * 59.5 / 59.7
920 IF D > 65 THEN NEWD = D * 68.9 / 69.2
930 IF D > 75 THEN NEWD = D * 79 / 79.3
940 IF D > 85 THEN NEWD = D * 88.5 / 88.9
950 IF D > 95 THEN NEWD = D * 99 / 99.5
960 IF D > 105 THEN NEWD = D * 108.7 / 109.5
970 IF D > 115 THEN NEWD = D * 118.3 / 119.2
980 IF D > 125 THEN NEWD = D * 127.8 / 128.9
990 IF D > 135 THEN NEWD = D * 137.6 / 139
1000 IF D > 145 THEN NEWD = D * 147.4 / 149.1
1010 IF D > 155 THEN NEWD = D * 157 / 159
1020 IF D > 165 THEN NEWD = D * 167.1 / 169.4
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1030 IF D > 175 THEN NEWD = D * 178.6 / 181.2
1040 IF D > 185 THEN NEWD = D * 186.4 / 189.5
1050 IF D > 195 THEN NEWD = D * 194.4 / 198.1
1060 IF E < 35 THEN NEWE = E * 28.8 / 30.3
1070 IF E > 35 THEN NEWE = E * 39.4 / 41.1
1080 IF E > 45 THEN NEWE = E * 48.8 / 50.3
1090 IF E > 55 THEN NEWE = E * 59.5 / 61.2
1100 IF E > 65 THEN NEWE = E * 68.9 / 70.5
1110 IF E > 75 THEN NEWE = E * 79 / 80.8
1120 IF E > 85 THEN NEWE = E * 88.5 / 90.3
1130 IF E > 95 THEN NEWE = E * 99 / 101
1140 IF E > 105 THEN NEWE = E * 108.7 / 111
1150 IF E > 115 THEN NEWE = E * 118.3 / 120.7
1160 IF E > 125 THEN NEWE = E * 127.8 / 130.4
1170 IF E > 135 THEN NEWE = E * 137.6 / 140.4
1180 IF E > 145 THEN NEWE = E * 147.4 / 150.4
1190 IF E > 155 THEN NEWE = E * 157 / 160.1
1200 IF E > 165 THEN NEWE = E * 167.1 / 170.7
1210 IF E > 175 THEN NEWE = E * 178.6 / 182.4
1220 IF E > 185 THEN NEWE = E * 186.4 / 190.6
1230 IF E > 195 THEN NEWE = E * 194.4 / 199.3
1240 IF F < 35 THEN NEWF = F * 28.8 / 30.5
1250 IF F > 35 THEN NEWF = F * 39.4 / 40.9
1260 IF F > 45 THEN NEWF = F * 48.8 / 50.2
1270 IF F > 55 THEN NEWF = F * 59.5 / 61.1
1280 IF F > 65 THEN NEWF = F * 68.9 / 70.5
1290 IF F > 75 THEN NEWF = F * 79 / 80.5
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1300 IF F > 85 THEN NEWF = F * 88.5 / 90.3
1310 IF F > 95 THEN NEWF = F * 99 / 100.8
1320 IF F > 105 THEN NEWF = F * 108.7 / 110.8
1330 IF F > 115 THEN NEWF = F * 118.3 / 120.6
1340 IF F > 125 THEN NEWF = F * 127.8 / 130.3
1350 IF F > 135 THEN NEWF = F * 137.6 / 140.3
1360 IF F > 145 THEN NEWF = F * 147.4 / 150.5
1370 IF F > 155 THEN NEWF = F * 157 / 160.4
1380 IF F > 165 THEN NEWF = F * 167.1 / 170.7
1390 IF F > 175 THEN NEWF = F * 178.6 / 182.5
1400 IF F > 185 THEN NEWF = F * 186.4 / 190.8
1410 IF F > 195 THEN NEWF = F * 194.4 / 199.4
1420 IF G < 35 THEN NEWG = G * 28.8 / 30.3
1430 IF G > 35 THEN NEWG = G * 39.4 / 41
1440 IF G > 45 THEN NEWG = G * 48.8 / 50.4
1450 IF G > 55 THEN NEWG = G * 59.5 / 61.2
1460 IF G > 65 THEN NEWG = G * 69.9 / 70.8
1470 IF G > 75 THEN NEWG = G * 79 / 80.8
1480 IF G > 85 THEN NEWG = G * 88.5 / 90.5
1490 IF G > 95 THEN NEWG = G * 99 / 101.1
1500 IF G > 105 THEN NEWG = G * 108.7 / 111.2
1510 IF G > 115 THEN NEWG = G * 118.3 / 120.9
1520 IF G > 125 THEN NEWG = G * 127.8 / 130.8
1530 IF G > 135 THEN NEWG = G * 137.6 / 140.8
1540 IF G > 145 THEN NEWG = G * 147.4 / 151.1
1550 IF G > 155 THEN NEWG = G * 157 / 161
1560 IF G > 165 THEN NEWG = G * 167.1 / 171.4
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1570 IF G > 175 THEN NEWG = G * 178.6 / 183.2
1580 IF G > 185 THEN NEWG = G * 186.4 / 191.5
1590 IF G > 195 THEN NEWG = G * 194.4 / 200
1600 TIM = J * 1
1610 PRINT USING "####.#"; NEWA; NEWB; NEWC; NEWD; NEWE;
     NEWF; NEWG; TIM
1620 LPRINT USING "####.#"; NEWA; NEWB; NEWC; NEWD;
     NEWE; NEWF; NEWG; TIM
1630 IF B < 1 THEN 1680
1640 PRINT #2, USING "####.#"; NEWA; NEWB; NEWC; NEWD;
     NEWE; NEWF; NEWG; TIM
1650 NEXT J
1660 GOTO 1680
1670 NEXT J
1680 CLOSE
```

APPENDIX VII: ASCII DATA FILE

30.8	30.1	30.7	29.5	31.1	29.9	31.3
30.8	30.2	30.7	29.5	31.3	30.0	31.4
30.8	30.2	30.8	29.5	31.1	30.0	31.6
30.8	30.1	30.8	29.5	31.1	30.1	32.0
30.8	30.2	30.9	29.5	31.2	29.9	32.3
31.0	30.3	30.9	29.5	31.3	30.0	32.7
31.1	30.2	31.1	29.6	31.3	30.0	32.9
31.1	30.3	31.0	29.6	31.4	30.0	33.2
31.2	30.5	31.2	29.6	31.4	30.1	33.5
31.4	30.5	31.3	29.5	31.3	30.3	33.8
31.5	30.8	31.4	29.6	31.4	30.4	34.1
31.7	30.8	31.6	29.6	31.4	30.5	34.7
31.9	31.0	31.7	29.7	31.6	30.5	35.1
32.1	31.1	31.9	29.7	31.6	30.7	35.5
32.4	31.3	32.0	30.0	31.7	30.8	36.0
32.6	31.5	32.3	30.1	31.9	31.0	36.6
33.0	31.7	32.5	30.2	31.9	31.1	36.8
33.3	31.9	32.6	30.4	32.1	31.3	37.3
33.6	32.3	32.8	30.6	32.2	31.4	37.7
33.9	32.5	33.1	30.8	32.4	31.6	38.2
34.2	32.7	33.3	31.0	32.6	31.9	38.5
34.7	32.9	33.6	31.3	32.8	32.0	38.9
35.0	33.2	33.8	31.5	33.1	32.2	39.3
35.5	33.6	34.1	31.7	33.5	32.6	39.8
35.8	33.8	34.4	31.9	33.4	32.8	40.3

36.3	34.2	34.7	32.2	33.7	33.0	40.8
36.6	34.5	34.9	32.6	34.0	33.3	41.2
37.1	34.9	35.2	32.8	34.3	33.5	41.7
37.4	35.2	35.7	33.0	34.4	33.8	42.3
37.9	35.5	35.9	33.4	34.9	34.1	42.6
38.3	35.9	36.3	33.7	35.0	34.4	43.1
38.8	36.3	36.6	34.0	35.4	34.8	43.6
39.3	36.6	37.1	34.2	35.5	35.1	44.0
39.7	37.0	37.4	34.7	35.9	35.2	44.5
40.2	37.4	37.8	34.9	36.3	35.7	45.0
40.7	37.7	38.4	35.5	36.6	35.9	45.5
41.1	38.2	38.7	35.7	36.9	36.3	45.9
41.7	38.5	39.2	36.0	37.1	36.6	46.5
42.1	39.0	39.6	36.4	37.6	37.0	47.0
42.5	39.3	39.9	36.6	37.9	37.4	47.5
43.1	39.8	40.4	37.0	38.2	37.6	47.9
43.4	40.2	40.7	37.4	38.5	37.9	48.3
44.0	40.7	41.2	37.8	39.0	38.3	48.7
44.4	41.0	41.7	38.1	39.3	38.7	49.2
44.8	41.5	42.1	38.4	39.6	39.0	49.7
45.3	41.9	42.6	38.8	39.9	39.4	50.1
45.8	42.4	43.1	39.0	40.3	39.9	50.6
46.2	42.9	43.4	39.6	40.7	40.1	50.9
46.7	43.4	43.9	39.9	41.1	40.5	51.5
47.1	43.7	44.4	40.2	41.5	40.8	51.9
47.5	44.2	44.8	40.7	41.9	41.1	52.2
48.1	44.7	45.2	41.0	42.5	41.5	52.6

48.4	45.1	45.7	41.3	42.7	41.9	53.0
48.9	45.6	46.2	41.7	43.1	42.3	53.5
49.3	46.1	46.6	42.1	43.4	42.6	54.0
49.7	46.5	47.0	42.5	43.8	43.0	54.3
50.2	47.0	47.5	42.7	44.2	43.5	54.7
50.6	47.4	47.9	43.1	44.5	43.9	55.2
51.0	47.8	48.4	43.4	44.9	44.2	55.6
51.4	48.4	48.9	43.8	45.3	44.5	56.1
51.9	48.8	49.2	44.4	45.6	44.8	56.5
52.3	49.2	49.6	44.7	46.2	45.2	56.8
52.7	49.6	50.0	45.0	46.4	45.7	57.2
53.2	50.1	50.4	45.5	46.8	45.9	57.7
53.6	50.5	50.9	45.7	47.3	46.4	57.9
54.1	51.0	51.3	46.2	47.6	46.6	58.5
54.5	51.4	51.7	46.5	48.1	47.2	58.7
54.9	51.8	52.2	46.9	48.3	47.5	59.2
55.3	52.2	52.5	47.4	48.7	47.7	59.6
55.8	52.7	53.0	47.8	49.2	48.2	59.9
56.1	53.2	53.6	48.0	49.5	48.6	60.4
56.6	53.6	54.0	48.5	49.9	48.9	60.7
56.9	54.0	54.4	48.7	50.4	49.4	61.1
57.4	54.4	54.9	49.2	50.7	49.7	61.6
57.8	54.9	55.2	49.5	51.1	50.2	61.9
58.2	55.4	55.8	50.0	51.6	50.4	62.3
58.6	55.8	56.1	50.3	51.9	50.8	62.8
59.0	56.2	56.6	50.6	52.2	51.3	63.1
59.4	56.6	57.0	51.1	52.7	51.5	63.5

59.9	57.1	57.5	51.4	52.8	52.0	64.0
60.2	57.4	57.8	51.7	53.4	52.2	64.2
60.6	57.9	58.4	52.1	53.6	52.7	64.8
61.0	58.3	58.7	52.4	54.1	53.0	65.1
61.4	58.7	59.1	52.9	54.4	53.3	65.6
61.8	59.2	59.5	53.2	54.9	53.8	66.0
62.3	59.6	59.9	53.6	55.2	54.0	66.4
62.6	60.0	60.4	54.0	55.5	54.4	66.9
63.0	60.5	60.8	54.3	56.0	54.8	67.2
63.4	60.9	61.3	54.6	56.3	55.3	67.6
63.9	61.4	61.7	55.1	56.8	55.7	67.9
64.2	61.8	62.1	55.5	57.2	56.0	68.2
64.7	62.2	62.6	56.0	57.5	56.3	68.6
65.1	62.6	63.2	56.2	57.8	56.6	69.1
65.5	63.1	63.6	56.5	58.3	57.1	69.5
65.9	63.4	64.0	57.0	58.7	57.3	69.9
66.2	63.9	64.5	57.3	59.0	57.6	70.3
66.7	64.5	65.1	57.6	59.3	58.1	70.7
67.0	64.8	65.4	58.0	59.8	58.2	71.0
67.5	65.3	65.9	58.3	60.1	58.8	71.4
67.8	65.6	66.3	58.9	60.5	59.0	71.7
68.3	66.2	66.8	59.1	61.0	59.5	72.2
68.6	66.6	67.2	59.4	61.3	59.9	72.5
68.9	67.0	67.6	59.9	61.6	60.1	73.0
69.5	67.5	68.1	60.3	61.9	60.4	73.3
69.8	67.9	68.4	60.7	62.4	60.9	73.6
70.3	68.3	68.9	60.9	62.7	61.2	74.1

70.5	68.7	69.4	61.5	63.1	61.5	74.5
70.9	69.2	69.7	61.8	63.5	61.8	74.8
71.4	69.6	70.0	62.1	63.9	62.2	75.2
71.7	70.1	70.4	62.5	64.2	62.6	75.5
72.2	70.5	70.9	62.9	64.6	62.9	75.9
72.5	70.9	71.2	63.2	65.1	63.3	76.2
72.9	71.3	71.7	63.5	65.4	63.7	76.6
73.3	71.7	72.0	63.9	65.8	64.0	77.0
73.6	72.2	72.4	64.2	66.2	64.5	77.3
73.9	72.6	72.8	64.6	66.4	64.7	77.7
74.4	73.0	73.2	64.9	66.8	65.1	78.1
74.7	73.5	73.6	65.3	67.0	65.4	78.5
75.0	73.8	73.9	65.7	67.5	65.8	78.8
75.4	74.2	74.3	66.1	67.8	66.1	79.3
75.8	74.6	74.7	66.5	68.3	66.4	79.6
76.0	74.9	75.0	66.9	68.6	66.8	79.9
76.5	75.4	75.4	67.3	68.9	67.1	80.2
76.8	75.8	75.8	67.6	69.5	67.6	80.6
77.1	76.1	76.2	67.9	69.8	68.0	81.0
77.5	76.5	76.5	68.4	70.0	68.3	81.5
77.9	76.8	76.9	68.7	70.4	68.6	81.7
78.2	77.2	77.2	69.2	70.8	68.9	82.1
78.6	77.6	77.6	69.5	71.1	69.3	82.3
79.0	77.9	77.9	69.7	71.5	69.7	82.8
79.3	78.4	78.3	70.3	71.8	70.0	83.1
79.6	78.8	78.6	70.5	72.4	70.3	83.4
79.9	79.1	79.0	70.9	72.7	70.8	83.7

80.3	79.5	79.3	71.1	73.1	71.1	84.1
80.7	79.8	79.8	71.5	73.4	71.5	84.5
81.0	80.1	80.1	71.9	73.7	71.9	84.8
81.3	80.5	80.4	72.4	74.1	72.2	85.2
81.8	80.9	80.7	72.7	74.6	72.6	85.6
82.1	81.3	81.2	72.9	74.7	73.0	85.9
82.4	81.6	81.5	73.4	75.2	73.3	86.2
82.8	82.0	81.8	73.8	75.7	73.6	86.4
83.2	82.3	82.3	74.1	75.8	74.1	87.0
83.5	82.6	82.6	74.5	76.3	74.4	87.3
83.9	83.0	82.9	74.8	76.7	74.9	87.5
84.2	83.4	83.3	75.2	76.9	75.1	88.0
84.5	83.7	83.6	75.5	77.4	75.5	88.2
84.8	84.1	84.0	75.9	77.7	75.8	88.6
85.3	84.4	84.2	76.3	78.2	76.2	88.8
85.6	84.7	84.7	76.6	78.4	76.6	89.2
85.9	85.1	85.1	77.0	78.8	76.8	89.7
86.2	85.4	85.3	77.4	79.3	77.2	89.9
86.6	85.7	85.7	77.7	79.6	77.7	90.3
86.9	86.1	86.0	78.1	79.9	77.9	90.6
87.3	86.4	86.4	78.5	80.3	78.5	90.9
87.6	86.8	86.7	78.8	80.6	78.7	91.3
87.9	87.2	87.1	79.2	81.0	79.2	91.5
88.3	87.5	87.5	79.6	81.3	79.3	91.9
88.6	87.8	87.8	79.9	81.7	79.9	92.3
88.9	88.1	88.1	80.4	82.2	80.1	92.6
89.3	88.5	88.6	80.7	82.4	80.5	92.9

89.7	88.8	88.7	81.0	82.9	81.0	93.2
89.9	89.2	89.1	81.4	83.2	81.2	93.6
90.3	89.5	89.5	81.7	83.4	81.6	93.9
90.6	89.8	89.8	82.1	84.0	82.0	94.2
90.9	90.1	90.3	82.4	84.1	82.3	94.7
91.3	90.4	90.5	82.9	84.5	82.7	94.9
91.6	90.8	90.8	83.1	84.9	83.2	95.2
91.9	91.1	91.2	83.6	85.3	83.4	95.5
92.2	91.4	91.5	83.9	85.6	83.8	95.9
92.6	91.8	91.9	84.3	85.9	84.2	96.3
93.0	92.1	92.2	84.6	86.3	84.5	96.5
93.3	92.5	92.5	85.1	86.7	84.8	96.8
93.6	92.7	92.8	85.3	87.2	85.2	97.1
93.9	93.0	93.2	85.6	87.4	85.6	97.5
94.2	93.4	93.5	85.9	87.8	85.9	97.8
94.5	93.7	93.8	86.3	88.1	86.4	98.1
94.9	94.0	94.1	86.9	88.6	86.5	98.4
95.2	94.4	94.6	87.1	88.7	86.9	98.8
95.5	94.7	94.8	87.3	89.2	87.5	99.0
95.8	94.9	95.2	87.9	89.6	87.7	99.3
96.2	95.3	95.6	88.1	89.9	88.1	99.8
96.6	95.6	95.8	88.5	90.3	88.5	100.0
96.8	95.9	96.2	88.9	90.6	88.7	100.3
97.1	96.3	96.5	89.2	90.9	89.2	100.7
97.4	96.6	96.8	89.5	91.4	89.6	101.0
97.7	96.8	97.1	90.0	91.7	89.8	101.2
98.1	97.3	97.5	90.3	91.9	90.3	101.6

98.4	97.5	97.8	90.7	92.5	90.4	101.8
98.8	97.9	98.1	91.0	92.7	90.8	102.3
99.0	98.2	98.4	91.3	93.0	91.2	102.6
99.3	98.5	98.8	91.7	93.4	91.4	102.9
99.6	98.8	99.0	91.9	93.7	92.0	103.2
100.0	99.1	99.4	92.5	94.1	92.2	103.4
100.4	99.4	99.7	92.7	94.6	92.5	103.7
100.7	99.8	100.0	93.0	94.8	93.0	104.0
100.9	100.1	100.4	93.3	95.1	93.3	104.4
101.2	100.4	100.7	93.7	95.5	93.8	104.5
101.6	100.7	101.0	94.3	95.9	93.9	105.0
101.9	101.0	101.4	94.6	96.0	94.3	105.3
102.2	101.2	101.6	94.7	96.6	94.8	105.6
102.6	101.5	102.0	95.2	96.8	95.0	105.9
102.8	101.8	102.3	95.6	97.2	95.3	106.2
103.1	102.3	102.6	95.8	97.4	95.8	106.6
103.4	102.6	102.9	96.2	97.8	96.1	106.8
103.8	102.9	103.2	96.5	98.2	96.4	107.1
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104.3	103.4	103.8	97.4	99.0	97.1	107.6
104.6	103.7	104.2	97.6	99.2	97.5	108.0
105.0	104.0	104.5	98.0	99.6	97.7	108.2
105.2	104.3	104.8	98.3	100.1	98.2	108.5
105.6	104.7	105.1	98.5	100.3	98.7	108.9
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106.2	105.2	105.6	99.3	101.0	99.1	109.5
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194.7 193.8 194.2 214.8 216.4 216.7 196.6

194.9 194.0 194.4 214.9 216.5 217.0 196.7

194.9	194.1	194.4	215.1	216.7	217.3	196.8
195.0	194.2	194.6	215.3	217.1	217.4	196.8
195.2	194.3	194.7	215.5	217.2	217.5	196.9
195.3	194.4	194.8	215.7	217.4	217.8	197.1
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195.5	194.7	195.0	216.3	217.8	218.1	197.3
195.6	194.8	195.1	216.5	218.0	218.2	197.4
195.8	194.9	195.2	216.7	218.3	218.3	197.5
195.8	195.0	195.3	216.8	218.5	218.6	197.5
196.0	195.2	195.5	217.0	218.8	218.8	197.7
196.0	195.2	195.6	217.1	219.0	219.0	197.8
196.2	195.3	195.7	217.4	219.0	219.0	197.9
196.3	195.5	195.8	217.7	219.2	219.2	198.1
196.3	195.5	195.8	217.9	219.6	219.3	198.0
196.5	195.6	196.0	217.9	219.6	219.6	198.2
196.6	195.8	196.0	218.3	219.8	219.6	198.4
196.8	195.8	196.2	218.5	220.2	219.8	198.3
196.8	196.0	196.3	218.6	220.2	220.0	198.7
196.9	196.1	196.4	218.8	220.5	220.2	198.7
197.1	196.2	196.6	219.0	220.6	220.2	198.8
197.1	196.3	196.6	219.0	220.9	220.6	198.9
197.2	196.5	196.7	219.3	221.0	220.5	199.0

APPENDIX VIII: CORRECTED .000 DATA FILE

30.0	29.5	29.8	29.3	29.0	29.3	30.6	1.0
30.3	29.7	30.0	29.4	29.2	29.5	31.7	2.0
30.9	30.2	30.6	29.4	29.2	29.7	33.1	3.0
31.7	30.9	31.3	29.9	29.7	30.2	35.5	4.0
33.0	31.9	32.0	30.6	30.2	30.8	37.1	5.0
35.0	32.9	33.0	31.5	31.2	31.7	38.6	6.0
36.5	34.2	34.5	32.6	31.9	32.6	40.5	7.0
38.2	35.9	35.9	33.8	33.5	33.9	42.3	8.0
40.1	37.3	37.6	35.4	34.6	35.4	44.4	9.0
41.9	38.9	39.1	36.5	35.8	36.8	46.4	10.0
43.7	40.6	40.9	38.0	37.2	38.1	48.0	11.0
45.6	42.5	42.5	39.5	38.5	39.5	49.7	12.0
47.4	44.3	44.4	40.9	40.2	40.9	51.4	13.0
49.0	46.1	46.2	42.4	41.4	42.4	53.0	14.0
50.7	48.0	48.0	43.7	43.3	43.8	55.0	15.0
52.5	49.7	49.5	45.5	44.7	45.4	56.6	16.0
54.1	51.4	51.3	46.9	46.2	46.9	58.0	17.0
55.9	53.2	53.0	48.5	47.7	48.3	59.5	18.0
57.4	55.0	55.0	50.0	49.3	49.8	61.1	19.0
59.1	56.7	56.6	51.4	50.5	51.4	62.7	20.0
60.6	58.3	58.2	52.9	52.0	52.7	64.4	21.0
62.2	60.1	59.9	54.3	53.9	54.1	66.0	22.0
63.8	61.8	61.7	55.9	55.4	55.6	67.3	23.0
65.4	63.5	63.5	57.2	56.8	56.9	69.0	24.0
67.0	65.1	65.4	58.8	58.3	58.3	70.4	25.0

68.7	67.0	67.1	60.2	59.6	59.7	72.0	26.0
70.1	68.7	68.7	61.7	61.1	61.1	73.4	27.0
71.7	70.4	70.2	63.1	63.0	62.6	74.9	28.0
73.1	72.1	71.8	64.5	64.3	64.0	76.4	29.0
74.6	73.7	73.2	66.0	65.6	65.3	77.9	30.0
75.9	75.2	74.7	67.5	67.3	66.8	79.2	31.0
77.3	76.6	76.1	69.1	68.5	68.1	80.7	32.0
78.7	78.2	77.4	70.4	70.1	69.5	82.0	33.0
80.1	79.5	78.9	71.8	71.3	71.1	83.3	34.0
81.5	81.0	80.3	73.3	73.0	72.5	85.1	35.0
83.0	82.4	81.7	74.7	74.5	74.0	86.3	36.0
84.5	83.8	83.0	76.1	75.9	75.4	87.6	37.0
85.7	85.3	84.8	77.5	77.3	76.9	89.1	38.0
87.0	86.8	86.1	79.0	78.6	78.4	90.3	39.0
88.4	88.1	87.6	80.5	80.0	79.7	91.7	40.0
89.7	89.4	88.8	81.9	81.5	81.2	93.0	41.0
91.0	90.7	90.2	83.4	83.1	82.6	94.1	42.0
92.4	92.1	91.5	84.9	84.4	84.0	95.4	43.0
93.6	93.3	92.8	86.1	85.8	85.8	96.6	44.0
94.8	94.5	94.1	87.7	87.3	87.1	97.8	45.0
96.0	95.8	95.4	89.0	88.5	88.6	99.2	46.0
97.3	97.0	96.6	90.5	90.1	89.8	100.3	47.0
98.5	98.3	97.8	91.7	91.3	91.4	101.7	48.0
99.8	99.6	99.2	93.1	92.9	92.7	102.9	49.0
101.1	100.7	100.4	94.5	94.3	94.2	104.2	50.0
102.3	102.1	101.7	95.9	95.5	95.3	105.4	51.0
103.5	103.2	103.0	97.3	96.9	96.7	106.5	52.0

104.7	104.4	104.1	98.6	98.2	98.1	107.7	53.0
105.9	105.4	105.2	100.1	99.6	99.3	108.8	54.0
107.1	106.6	106.5	101.4	101.0	100.8	109.9	55.0
108.3	107.8	107.7	102.8	102.1	102.1	111.2	56.0
109.4	108.9	108.9	104.1	103.4	103.6	112.3	57.0
110.6	110.1	110.1	105.4	104.6	104.7	113.3	58.0
111.8	111.2	111.3	106.8	105.9	106.0	114.5	59.0
112.9	112.4	112.4	108.1	107.3	107.4	115.5	60.0
114.0	113.6	113.5	109.6	108.6	108.7	116.6	61.0
115.1	114.6	114.5	110.8	109.9	110.0	117.8	62.0
116.2	115.7	115.7	112.0	111.0	111.2	119.1	63.0
117.4	116.8	116.8	113.3	112.1	112.6	120.2	64.0
118.5	117.9	117.8	114.5	114.0	113.9	121.2	65.0
119.6	119.0	119.0	115.6	115.1	115.3	122.3	66.0
120.7	120.1	120.1	116.9	116.4	116.5	123.3	67.0
121.7	121.1	121.0	118.1	117.5	117.7	124.4	68.0
122.8	122.2	122.1	119.4	118.7	119.0	125.4	69.0
123.8	123.3	123.1	120.6	120.0	120.3	126.2	70.0
124.8	124.2	124.1	121.7	121.1	121.6	127.4	71.0
125.9	125.2	125.1	123.2	122.5	122.7	128.3	72.0
126.8	126.2	126.1	124.2	123.7	123.9	129.3	73.0
127.8	127.2	127.1	125.3	124.8	125.0	130.3	74.0
128.8	128.2	128.0	126.4	126.0	126.0	131.2	75.0
129.8	129.2	129.0	127.5	127.0	127.3	132.1	76.0
130.7	130.2	130.0	128.5	128.1	128.5	133.0	77.0
131.7	131.0	130.8	129.7	129.2	129.6	133.8	78.0
132.6	131.9	131.9	130.8	130.2	130.6	134.7	79.0

133.3	132.9	132.7	131.7	131.3	131.9	135.3	80.0
134.0	133.8	133.4	133.0	132.3	132.8	136.2	81.0
134.8	134.3	134.2	133.8	133.3	133.9	137.1	82.0
135.7	135.1	135.1	134.5	134.3	134.6	138.0	83.0
136.5	136.0	136.0	135.6	135.3	135.7	138.9	84.0
137.5	136.9	136.9	136.5	136.3	136.8	139.7	85.0
138.3	137.8	137.7	137.5	137.2	137.8	140.7	86.0
139.2	138.6	138.6	138.6	138.2	138.7	141.4	87.0
140.0	139.6	139.4	139.5	139.2	139.8	142.2	88.0
140.8	140.3	140.2	140.5	140.3	140.9	143.1	89.0
141.6	141.2	141.0	141.4	141.1	141.8	143.7	90.0
142.3	142.0	141.8	142.4	142.3	142.8	144.4	91.0
143.3	142.8	142.6	143.4	143.2	143.5	145.4	92.0
144.0	143.4	143.4	144.1	144.2	144.6	146.1	93.0
144.8	144.3	144.1	145.1	145.0	145.5	146.9	94.0
145.6	145.1	144.9	146.2	145.9	146.5	147.7	95.0
146.4	145.9	145.8	147.0	146.8	147.4	148.5	96.0
147.2	146.8	146.6	147.8	147.8	148.4	149.3	97.0
148.0	147.5	147.4	148.7	148.6	149.4	150.2	98.0
148.8	148.3	148.2	149.7	149.6	150.3	150.8	99.0
149.6	149.1	149.0	150.5	150.6	151.4	151.4	100.0
150.3	149.9	149.7	151.6	151.5	152.1	152.2	101.0
151.1	150.6	150.4	152.4	152.5	153.2	153.1	102.0
151.7	151.3	151.2	153.5	153.4	153.7	153.8	103.0
152.5	152.2	151.8	154.4	154.3	154.6	154.4	104.0
153.3	152.9	152.7	155.1	155.1	155.7	155.2	105.0
154.0	153.5	153.6	156.0	156.0	156.5	155.8	106.0

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161.0 160.4 160.5 163.9 164.3 165.0 161.9 116.0

161.6 161.1 161.2 164.7 165.0 166.1 162.6 117.0

162.2 161.8 161.9 165.8 165.7 166.7 163.2 118.0

162.8 162.4 162.5 166.6 166.5 167.6 164.0 119.0

162.8 162.9 163.2 167.5 167.4 168.5 164.6 120.0

163.5 163.1 163.2 168.1 168.3 169.5 165.1 121.0

164.1 163.6 163.8 169.2 169.1 170.2 165.8 122.0

164.6 164.3 164.4 169.9 169.8 171.2 166.5 123.0

165.2 165.0 165.0 170.7 170.8 172.1 167.0 124.0

166.0 165.6 165.6 171.5 171.6 172.5 167.7 125.0

166.6 166.2 166.3 172.5 172.4 173.3 168.3 126.0

167.1 166.8 166.9 173.0 173.3 174.2 168.9 127.0

167.8 167.4 167.5 173.7 174.1 175.0 169.5 128.0

168.4 168.0 168.2 174.6 174.8 175.8 170.1 129.0

169.0 168.7 168.8 175.4 175.7 176.8 170.6 130.0

169.6 169.3 169.4 176.2 176.4 177.6 171.3 131.0

170.1 169.9 170.0 177.1 177.3 178.4 171.5 132.0

170.6 170.4 170.5 177.9 178.1 179.2 172.1 133.0

171.2 170.9 171.0 178.7 179.0 180.2 172.6 134.0

171.8 171.5 171.6 179.5 179.6 181.0 173.1 135.0

172.2 172.1 172.2 180.4 180.6 181.7 173.7 136.0

172.8 172.5 172.5 181.0 181.0 182.5 174.3 137.0

173.3 173.0 173.1 181.9 181.7 183.3 174.9 138.0

173.9 173.7 173.7 182.8 182.7 184.1 175.2 139.0

174.5 174.1 174.2 183.6 183.4 184.8 175.8 140.0

174.9 174.6 174.6 184.4 184.2 185.6 176.3 141.0

175.4 175.2 175.2 184.9 185.0 186.5 176.9 142.0

176.0 175.7 175.8 185.9 185.8 187.3 177.4 143.0

176.5 176.2 176.2 186.5 186.6 188.3 177.9 144.0

177.1 176.8 176.9 187.5 187.3 189.0 178.5 145.0

177.5 177.2 177.4 188.4 188.2 189.8 179.0 146.0

178.1 177.7 177.8 189.1 189.0 190.5 179.4 147.0

178.5 178.3 178.3 189.9 189.7 191.4 179.8 148.0

179.1 178.8 178.8 190.6 190.7 192.5 180.4 149.0

179.6 179.3 179.4 191.4 191.5 193.2 181.0 150.0

180.1 179.9 179.9 192.6 192.4 194.0 181.3 151.0

180.6 180.4 180.3 193.3 193.0 194.9 181.8 152.0

181.1 180.8 180.8 194.1 194.0 195.7 182.2 153.0

181.6 181.2 181.2 195.0 194.7 196.4 182.8 154.0

181.8 181.7 181.8 195.8 195.5 197.2 183.3 155.0

182.3 182.2 182.2 196.4 196.3 198.3 183.8 156.0

182.8 182.7 182.8 197.3 197.0 198.9 184.3 157.0

183.3 183.1 183.3 198.2 197.8 199.7 184.8 158.0

183.8 183.6 183.7 198.8 198.7 200.6 185.1 159.0

184.2 184.1 184.2 199.7 199.3 201.2 185.6 160.0

184.8	184.5	184.6	200.7	200.1	202.1	186.1	161.0
185.1	185.0	185.1	201.3	201.1	202.9	186.4	162.0
185.6	185.4	185.5	202.0	201.5	203.7	187.0	163.0
186.0	185.9	186.0	202.8	202.4	204.6	187.5	164.0
186.5	186.3	186.5	203.7	203.3	205.3	187.9	165.0
187.0	186.8	187.1	204.5	203.9	206.0	188.6	166.0
187.5	187.3	187.4	205.3	205.0	206.8	189.9	167.0
187.9	187.8	188.0	206.1	205.6	207.7	189.4	168.0
188.4	188.2	188.4	207.0	206.5	208.6	189.7	169.0
188.8	188.8	188.9	207.9	207.4	209.3	190.1	170.0
189.3	189.2	189.3	208.7	208.2	210.2	190.5	171.0
189.7	189.7	189.8	209.4	209.0	211.0	191.4	172.0
190.1	190.1	190.2	210.4	209.7	211.6	191.9	173.0
190.6	190.5	190.6	211.2	210.6	212.4	192.3	174.0
191.1	190.9	191.1	212.0	211.4	213.2	192.8	175.0
191.7	191.3	191.5	212.8	212.2	214.0	193.2	176.0
192.2	191.8	192.1	213.8	213.1	214.5	193.6	177.0
192.6	192.3	192.6	214.5	213.8	215.2	194.0	178.0
193.0	192.8	192.9	215.4	214.6	215.8	194.5	179.0
193.5	193.2	193.5	216.0	215.4	216.4	194.8	180.0

APPENDIX IX: THE .DTA FILE

29.5	-0.2	-0.5	-0.2	1.0
29.7	-0.3	-0.5	-0.5	2.0
30.2	-0.8	-1.0	-0.5	3.0
30.9	-1.0	-1.4	-0.7	4.0
31.9	-1.3	-1.7	-1.1	5.0
32.9	-1.4	-1.7	-1.2	6.0
34.2	-1.6	-2.3	-1.6	7.0
35.9	-2.1	-2.4	-2.0	8.0
37.3	-1.9	-2.7	-1.9	9.0
38.9	-2.4	-3.1	-2.1	10.0
40.6	-2.6	-3.4	-2.5	11.0
42.5	-3.0	-4.0	-3.0	12.0
44.3	-3.4	-4.1	-3.4	13.0
46.1	-3.7	-4.7	-3.7	14.0
48.0	-4.3	-4.7	-4.2	15.0
49.7	-4.2	-5.0	-4.3	16.0
51.4	-4.5	-5.2	-4.5	17.0
53.2	-4.7	-5.5	-4.9	18.0
55.0	-5.0	-5.7	-5.2	19.0
56.7	-5.3	-6.2	-5.3	20.0
58.3	-5.4	-6.3	-5.6	21.0
60.1	-5.8	-6.2	-6.0	22.0
61.8	-5.9	-6.4	-6.2	23.0
63.5	-6.3	-6.7	-6.6	24.0
65.1	-6.3	-6.8	-6.8	25.0

67.0	-6.8	-7.4	-7.3	26.0
68.7	-7.0	-7.6	-7.6	27.0
70.4	-7.3	-7.4	-7.8	28.0
72.1	-7.6	-7.8	-8.1	29.0
73.7	-7.7	-8.1	-8.4	30.0
75.2	-7.7	-7.9	-8.4	31.0
76.6	-7.5	-8.1	-8.5	32.0
78.2	-7.8	-8.1	-8.7	33.0
79.5	-7.7	-8.2	-8.4	34.0
81.0	-7.7	-8.0	-8.5	35.0
82.4	-7.7	-7.9	-8.4	36.0
83.8	-7.7	-7.9	-8.4	37.0
85.3	-7.8	-8.0	-8.4	38.0
86.8	-7.8	-8.2	-8.4	39.0
88.1	-7.6	-8.1	-8.4	40.0
89.4	-7.5	-7.9	-8.2	41.0
90.7	-7.3	-7.6	-8.1	42.0
92.1	-7.2	-7.7	-8.1	43.0
93.3	-7.2	-7.5	-7.5	44.0
94.5	-6.8	-7.2	-7.4	45.0
95.8	-6.8	-7.3	-7.2	46.0
97.0	-6.5	-6.9	-7.2	47.0
98.3	-6.6	-7.0	-6.9	48.0
99.6	-6.5	-6.7	-6.9	49.0
100.7	-6.2	-6.4	-6.5	50.0
102.1	-6.2	-6.6	-6.8	51.0
103.2	-5.9	-6.3	-6.5	52.0

104.4	-5.8	-6.2	-6.3	53.0
105.4	-5.3	-5.8	-6.1	54.0
106.6	-5.2	-5.6	-5.8	55.0
107.8	-5.0	-5.7	-5.7	56.0
108.9	-4.8	-5.5	-5.3	57.0
110.1	-4.7	-5.5	-5.4	58.0
111.2	-4.4	-5.3	-5.2	59.0
112.4	-4.3	-5.1	-5.0	60.0
113.6	-4.0	-5.0	-4.9	61.0
114.6	-3.8	-4.7	-4.6	62.0
115.7	-3.7	-4.7	-4.5	63.0
116.8	-3.5	-4.7	-4.2	64.0
117.9	-3.4	-3.9	-4.0	65.0
119.0	-3.4	-3.9	-3.7	66.0
120.1	-3.2	-3.7	-3.6	67.0
121.1	-3.0	-3.6	-3.4	68.0
122.2	-2.8	-3.5	-3.2	69.0
123.3	-2.7	-3.3	-3.0	70.0
124.2	-2.5	-3.1	-2.6	71.0
125.2	-2.0	-2.7	-2.5	72.0
126.2	-2.0	-2.5	-2.3	73.0
127.2	-1.9	-2.4	-2.2	74.0
128.2	-1.8	-2.2	-2.2	75.0
129.2	-1.7	-2.2	-1.9	76.0
130.2	-1.7	-2.1	-1.7	77.0
131.0	-1.3	-1.8	-1.4	78.0
131.9	-1.1	-1.7	-1.3	79.0

132.9	-1.2	-1.6	-1.0	80.0
133.8	-0.8	-1.5	-1.0	81.0
134.3	-0.5	-1.0	-0.4	82.0
135.1	-0.6	-0.8	-0.5	83.0
136.0	-0.4	-0.7	-0.3	84.0
136.9	-0.4	-0.6	-0.1	85.0
137.8	-0.3	-0.6	0.0	86.0
138.6	0.0	-0.4	0.1	87.0
139.6	-0.1	-0.4	0.2	88.0
140.3	0.2	0.0	0.6	89.0
141.2	0.2	-0.1	0.6	90.0
142.0	0.4	0.3	0.8	91.0
142.8	0.6	0.4	0.7	92.0
143.4	0.7	0.8	1.2	93.0
144.3	0.8	0.7	1.2	94.0
145.1	1.1	0.8	1.4	95.0
145.9	1.1	0.9	1.5	96.0
146.8	1.0	1.0	1.6	97.0
147.5	1.2	1.1	1.9	98.0
148.3	1.4	1.3	2.0	99.0
149.1	1.4	1.5	2.3	100.0
149.9	1.7	1.6	2.2	101.0
150.6	1.8	1.9	2.6	102.0
151.3	2.2	2.1	2.4	103.0
152.2	2.2	2.1	2.4	104.0
152.9	2.2	2.2	2.8	105.0
153.5	2.5	2.5	3.0	106.0

154.2	2.8	2.6	3.1	107.0
154.9	3.0	3.0	3.5	108.0
155.5	3.3	3.1	3.7	109.0
156.3	3.5	3.3	3.8	110.0
156.9	3.7	3.3	4.2	111.0
157.7	3.7	3.6	4.2	112.0
158.4	3.9	3.0	4.3	113.0
159.1	3.9	3.3	4.4	114.0
159.7	3.4	3.6	4.5	115.0
160.4	3.5	3.9	4.6	116.0
161.1	3.6	3.9	5.0	117.0
161.8	4.0	3.9	4.9	118.0
162.4	4.2	4.1	5.2	119.0
162.9	4.6	4.5	5.6	120.0
163.1	5 0	5.2	6.4	121.0
163.6	5.6	5.5	6.6	122.0
164.3	5.6	5.5	6.9	123.0
165.0	5.7	5.8	7.1	124.0
165.6	5.9	6.0	6.9	125.0
166.2	6.3	6.2	7.1	126.0
166.8	6.2	6.5	7.4	127.0
167.4	6.3	6.7	7.6	128.0
168.0	6.6	6.8	7.8	129.0
168.7	6.7	7.0	8.1	130.0
169.3	6.9	7.1	8.3	131.0
169.9	7.2	7.4	8.5	132.0
170.4	7.5	7.7	8.8	133.0

170.9	7.8	8.1	9.3	134.0
171.5	8.0	8.1	9.5	135.0
172.1	8.3	8.5	9.6	136.0
172.5	8.5	8.5	10.0	137.0
173.0	8.9	8.7	10.3	138.0
173.7	9.1	9.0	10.4	139.0
174.1	9.5	9.3	10.7	140.0
174.6	9.8	9.6	11.0	141.0
175.2	9.7	9.8	11.3	142.0
175.7	10.2	10.1	11.6	143.0
176.2	10.3	10.4	12.1	144.0
176.8	10.7	10.5	12.2	145.0
177.2	11.2	11.0	12.6	146.0
177.7	11.4	11.3	12.8	147.0
178.3	11.6	11.4	13.1	148.0
178.8	11.8	11.9	13.7	149.0
179.3	12.1	12.2	13.9	150.0
179.9	12.7	12.5	14.1	151.0
180.4	12.9	12.6	14.5	152.0
180.8	13.3	13.2	14.9	153.0
181.2	13.8	13.5	15.2	154.0
181.7	14.1	13.8	15.5	155.0
182.2	14.2	14.1	16.1	156.0
182.7	14.6	14.3	16.2	157.0
183.1	15.1	14.7	16.6	158.0
183.6	15.2	15.1	17.0	159.0
184.1	15.6	15.2	17.1	160.0

184.5	16.2	15.6	17.6	161.0
185.0	16.3	16.1	17.9	162.0
185.4	16.6	16.1	18.3	163.0
185.9	16.9	16.5	18.7	164.0
186.3	17.4	17.0	19.0	165.0
186.8	17.7	17.1	19.2	166.0
187.3	18.0	17.7	19.5	167.0
187.8	18.3	17.8	19.9	168.0
188.2	18.8	18.3	20.4	169.0
188.8	19.1	18.6	20.5	170.0
189.2	19.5	19.0	21.0	171.0
189.7	19.7	19.3	21.3	172.0
190.1	20.3	19.6	21.5	173.0
190.5	20.7	20.1	21.9	174.0
190.9	21.1	20.5	22.3	175.0
191.3	21.5	20.9	22.7	176.0
191.8	22.0	21.3	22.7	177.0
192.3	22.2	21.5	22.9	178.0
192.8	22.6	21.8	23.0	179.0
193.2	22.8	22.2	23.2	180.0

APPENDIX X: AVERAGE PROGRAM LISTING

```
10   DIM COAL(200,15)
20   DIM DTA(200,3)
25   FOR A=1 TO 200
27   FOR B=1 TO 15
29   COAL(A,B)=0
31   NEXT B
33   FOR C=1 TO
35   DTA(A,C)=0
37   NEXT C
39   NEXT A
40   PRINT "COALx, where x=No."
41   INPUT COLE$
50   PRINT "number of rows = ? (<200)"
60   INPUT N
70   COUNT=0
80   INPUT "which dta file to read ?";DF$
81   IF DF$="n" THEN 230
82   IF DF$="N" THEN 230
90   LONGDF$=DF$+".dta"
92   IF COUNT=0 THEN 100
94   IF COUNT=1 THEN 122
96   IF COUNT=2 THEN 125
100  OPEN LONGDF$ FOR INPUT AS #1
110  OPDF$=COLE$+".zzz"
120  OPEN OPDF$ FOR OUTPUT AS #2
```

```
121  GOTO 130
122  OPEN LONGDF$ FOR INPUT AS #3
123  GOTO 130
125  OPEN LONGDF$ FOR INPUT AS #4
130  FOR I=1 TO N
150  IF COUNT=0 THEN 180
160  IF COUNT=1 THEN 190
170  IF COUNT=2 THEN 200
180  INPUT#1,COAL(I,1),COAL(I,7),COAL(I,8),COAL(I,9),
     COAL(I,4)
181  GOTO 210
190  INPUT #3,COAL(I,2),COAL(I,10),COAL(I,11),
     COAL(I,12),COAL(I,5)
191  GOTO 210
200  INPUT #4,COAL(I,3),COAL(I,13),COAL(I,14),
     COAL(I,15),COAL(I,6)
201  GOTO 210
210  NEXT I
215  COUNT=COUNT+1
220  GOTO 80
230  PRINT "which column not required ? (7-15)"
240  INPUT L
250  FOR M=1 TO N
260  COAL(M,L)=0
270  NEXT M
280  PRINT "another column not required ? (y/n)"
290  INPUT Z$
```

```
300  IF Z$="y" THEN 230
310  IF Z$="n" THEN 320
320  PRINT "how many prt columns being consider ? (1-9)"
330  INPUT O
340  FOR P=1 TO N
350  SUM=0
360  FOR Q=1 TO 9
370  R=Q+6
380  SUM=SUM+COAL(P,R)
390  NEXT Q
400  DTA(P,2)=SUM/O
410  NEXT P
420  PRINT "how many inert columns ? (1-3)"
430  INPUT T
440  FOR S=1 TO N
450  SINT=O
460  FOR U=1 TO 3
470  SINT=SINT+COAL(S,U)
480  NEXT U
490  DTA(S,1)=SINT/T
500  DTA(S,3)=COAL(S,4)
510  NEXT S
520  FOR I=1 TO N
522  PRINT USING "####.#";DTA(I,1),DTA(I,2),DTA(I,3)
524  PRINT #2, USING "####.#";DTA(I,1),DTA(I,2),DTA(I,3)
```

```
527  NEXT I
530  END
```

APPENDIX XI: THE .ZZZ FILE

29.5	-0.3	1.0
29.7	-0.4	2.0
30.2	-0.8	3.0
30.9	-1.0	4.0
31.9	-1.4	5.0
32.9	-1.4	6.0
34.2	-1.8	7.0
35.9	-2.2	8.0
37.3	-2.2	9.0
38.9	-2.5	10.0
40.6	-2.8	11.0
42.5	-3.3	12.0
44.3	-3.6	13.0
46.1	-4.0	14.0
48.0	-4.4	15.0
49.7	-4.5	16.0
51.4	-4.7	17.0
53.2	-5.0	18.0
55.0	-5.3	19.0
56.7	-5.6	20.0
58.3	-5.8	21.0
60.1	-6.0	22.0
61.8	-6.2	23.0
63.5	-6.5	24.0
65.1	-6.6	25.0

67.0	-7.2	26.0
68.7	-7.4	27.0
70.4	-7.5	28.0
72.1	-7.8	29.0
73.7	-8.1	30.0
75.2	-8.0	31.0
76.6	-8.0	32.0
78.2	-8.2	33.0
79.5	-8.1	34.0
81.0	-8.1	35.0
82.4	-8.0	36.0
83.8	-8.0	37.0
85.3	-8.1	38.0
86.8	-8.1	39.0
88.1	-8.0	40.0
89.4	-7.9	41.0
90.7	-7.7	42.0
92.1	-7.7	43.0
93.3	-7.4	44.0
94.5	-7.1	45.0
95.8	-7.1	46.0
97.0	-6.9	47.0
98.3	-6.8	48.0
99.6	-6.7	49.0
100.7	-6.4	50.0
102.1	-6.5	51.0
103.2	-6.2	52.0

104.4	-6.1	53.0
105.4	-5.7	54.0
106.6	-5.5	55.0
107.8	-5.5	56.0
108.9	-5.2	57.0
110.1	-5.2	58.0
111.2	-5.0	59.0
112.4	-4.8	60.0
113.6	-4.6	61.0
114.6	-4.4	62.0
115.7	-4.3	63.0
116.8	-4.1	64.0
117.9	-3.8	65.0
119.0	-3.7	66.0
120.1	-3.5	67.0
121.1	-3.3	68.0
122.2	-3.2	69.0
123.3	-3.0	70.0
124.2	-2.7	71.0
125.2	-2.4	72.0
126.2	-2.3	73.0
127.2	-2.2	74.0
128.2	-2.1	75.0
129.2	-1.9	76.0
130.2	-1.8	77.0
131.0	-1.5	78.0
131.9	-1.4	79.0

132.9	-1.3	80.0
133.8	-1.1	81.0
134.3	-0.6	82.0
135.1	-0.6	83.0
136.0	-0.5	84.0
136.9	-0.4	85.0
137.8	-0.3	86.0
138.6	-0.1	87.0
139.6	-0.1	88.0
140.3	0.3	89.0
141.2	0.2	90.0
142.0	0.5	91.0
142.8	0.6	92.0
143.4	0.9	93.0
144.3	0.9	94.0
145.1	1.1	95.0
145.9	1.2	96.0
146.8	1.2	97.0
147.5	1.4	98.0
148.3	1.6	99.0
149.1	1.7	100.0
149.9	1.8	101.0
150.6	2.1	102.0
151.3	2.2	103.0
152.2	2.2	104.0
152.9	2.4	105.0
153.5	2.7	106.0

154.2	2.8	107.0
154.9	3.2	108.0
155.5	3.4	109.0
156.3	3.5	110.0
156.9	3.7	111.0
157.7	3.8	112.0
158.4	3.7	113.0
159.1	3.9	114.0
159.7	3.8	115.0
160.4	4.0	116.0
161.1	4.2	117.0
161.8	4.3	118.0
162.4	4.5	119.0
162.9	4.9	120.0
163.1	5.5	121.0
163.6	5.9	122.0
164.3	6.0	123.0
165.0	6.2	124.0
165.6	6.3	125.0
166.2	6.5	126.0
166.8	6.7	127.0
167.4	6.9	128.0
168.0	7.1	129.0
168.7	7.3	130.0
169.3	7.4	131.0
169.9	7.7	132.0
170.4	8.0	133.0

170.9	8.4	134.0
171.5	8.5	135.0
172.1	8.8	136.0
172.5	9.0	137.0
173.0	9.3	138.0
173.7	9.5	139.0
174.1	9.8	140.0
174.6	10.1	141.0
175.2	10.3	142.0
175.7	10.6	143.0
176.2	10.9	144.0
176.8	11.1	145.0
177.2	11.6	146.0
177.7	11.8	147.0
178.3	12.0	148.0
178.8	12.5	149.0
179.3	12.7	150.0
179.9	13.1	151.0
180.4	13.3	152.0
180.8	13.8	153.0
181.2	14.2	154.0
181.7	14.5	155.0
182.2	14.8	156.0
182.7	15.0	157.0
183.1	15.5	158.0
183.6	15.8	159.0
184.1	16.0	160.0

184.5	16.5	161.0
185.0	16.8	162.0
185.4	17.0	163.0
185.9	17.4	164.0
186.3	17.8	165.0
186.8	18.0	166.0
187.3	18.4	167.0
187.8	18.7	168.0
188.2	19.2	169.0
188.8	19.4	170.0
189.2	19.8	171.0
189.7	20.1	172.0
190.1	20.5	173.0
190.5	20.9	174.0
190.9	21.3	175.0
191.3	21.7	176.0
191.8	22.0	177.0
192.3	22.2	178.0
192.8	22.5	179.0
193.2	22.7	180.0

APPENDIX XII: OUTPUT OF ANALYSIS FILE

```
ANALYSING  july16.zzz
**********************************************************************
****                                                              ****
****                STAGE 1:ENDOTHERMIC REACTION                  ****
****                ----------------------------                  ****
**********************************************************************

  THE POINT OF MINIMUM NEGATIVE TEMPERATURE VARIANCE
  --------------------------------------------------
   INERT TEMPERATURE          : 78.2 degrees celsius

   TEMPERATURE DIFFERENTIAL :-8.2

   DATA READING and TIME      : 33 minutes

  THE SLOPE OF THE NEGATIVE SIDE OF THE THERMOGRAM
  ------------------------------------------------

   NEGATIVE SLOPE         =  : .8377825
                          =  : 39.95569 degrees

**********************************************************************
****                                                              ****
****             STAGE 2 : EXOTHERMIC REACTION                    ****
****             ----------------------------                     ****
**********************************************************************

  CHARACTERISTICS OF THE CROSSING POINT
  -------------------------------------
   TEMPERATURE DIFFERENTIAL    : .3
   INERT TEMPERATURE           : 140.3
   DATA READING                : 89

  POSITIVE SLOPE OF STAGE 2 (AVERAGE OF 3 SLOPES)
  -----------------------------------------------
 1 : SLOPE IIa                                  : 1.136876
                                                  48.66504 degrees
 2 : SLOPE II (COMPOSITE)                       : 1.17124
                                                  49.50945
 3 : SLOPE IIb                                  : 1.244827
                                                  51.22425
```

CHARACTERISTICS OF THE POSITIVE SLOPE CHANGE

2 METHODS WERE USED TO CALC. THE SLOPE CHANGE
METHOD 2:SUCCESIVE CHANGES IN THE +IVE SLOPE
METHOD 1:DISTANCES FROM A STRAIGHT LINE GRAPH

	METHOD 1	METHOD 2
TEMP. DIFFERENTIAL =	7.4	4.9
INERT TEMPERATURE =	169.3	162.9
DATA READING =	131	120

****
****
****
****
****

STAGE 3:HIGH EXOTHERMITY

MAXIMUM TEMPERATURE DIFFERENTIAL

TEMPERATURE DIFFERENTIAL : 22.7
INERT TEMPERATURE : 193.2
DATA READING : 180

POSITIVE SLOPE OF STAGE 3

SLOPE = : 1.640168
58.62962

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