

# **Assessment of Spontaneous Heating of Coals by Thermal Analysis Technique**

**Aditya Acharya**

Roll No. 112MN0497



**Department of Mining Engineering  
National Institute of Technology**

**Rourkela - 769008**

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# **Assessment of Spontaneous Heating of Coals by Thermal Analysis Technique**

Thesis submitted  
in partial fulfillment of the requirements for the degree  
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**Bachelor of Technology  
in  
Mining Engineering**

By

**Aditya Acharya**

Roll No. 112MN0497

Under the guidance of

**Dr. Himanshu Bhushan Sahu**

Associate Professor



**Department of Mining Engineering  
National Institute of Technology  
Rourkela - 769008  
2015 - 16**



Department of Mining Engineering  
National Institute of Technology  
Rourkela-769008

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## CERTIFICATE

This is to certify that the work in the thesis entitled “*Assessment of Spontaneous Heating of Coals by Thermal Analysis Technique*” submitted by **Aditya Acharya** (Roll No. 112MN0497) in partial fulfillment of the requirements for the degree of Bachelor of Technology in Mining Engineering at the National Institute of Technology, Rourkela is an authentic record of research work done by him in this department under my guidance and supervision.

To the best of my knowledge, the matter embodied in this thesis has not been submitted to any other Institute/University for the award of any Degree or Diploma.

Date:

**Dr. Himanshu Bhushan Sahu**

Associate Professor  
Department of Mining Engineering  
NIT Rourkela

# **DECLARATION OF ORIGINALITY**

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I, **Aditya Acharya**, Roll No. 112MN0497 hereby declare that this thesis entitled "***Assessment of Spontaneous Heating of Coals by Thermal Analysis Technique***" represents my original work carried out as an undergraduate student of NIT Rourkela and to the best of my knowledge, it contains no material previously published or written by someone else, nor any material presented for the award of any other degree of NIT Rourkela or any other institution. Any contribution made to this research by others, with whom I have worked at NIT Rourkela or elsewhere, is explicitly acknowledged in the thesis. Works of other authors cited in this thesis have been duly acknowledged under the section "Reference." I have also submitted my original research records to the scrutiny committee for evaluation of my thesis.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present thesis.

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**Aditya Acharya**

**Date:**

112MN0497

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**Place:** Rourkela

**Aditya Acharya**

**Date:**

# ABSTRACT

## INTRODUCTION

Coal mine fire is a major issue not only in India but also all over the world. But around 80% coal fires occurring in Indian coal mines are due to spontaneous heating of coal. Due to coal fire many precious lives are lost, and economical loss also occur to the organization and to the nation. With rapid growth of India, the requirements for energy are increasing rapidly for industrial sectors and domestic use. To cater the increased demand for energy, we are relying on thermal power plants as the international prices of coal keep decreasing. So more stress is now given on high production of coal. With increase in coal production the spontaneous heating incidents are also bound to increase. That's why special attention needs to be given for more study on spontaneous heating susceptibility of coal. So that early precautionary measures could be taken to prevent the coal to catch fire.

## EXPERIMENTATION

In this study, 26 coal samples were collected from different coal fields of India viz. MCL, SECL, SCCL, ECL, CCL, BCCL, NECL, NCL. All the samples of coal were studied using Differential thermal analysis and proximate analysis was also done in order to get the intrinsic properties of the coal. Using the obtained thermogram from DTA study, the coal samples were classified into three categories based on their potential to spontaneous heating. As the intrinsic properties like moisture content and volatile matter are known to influence the spontaneous heating properties of the coal, correlation study was done in order to find the correlation in between intrinsic properties of coal and the key indicators of spontaneous heating from thermogram viz. onset temperature, slope IIA, slope IIB, overall slope II.

## RESULTS

Sample No.	M.C.	V.M.	Ash	F.C.	Onset Temp.	Slope IIA	Slope IIB	Slope II
Sample 1	2.35	29.57	28.71	39.37	154.6	1.089474	0.797551	0.870364
Sample 2	2.27	24.55	11.08	62.1	133.1	1.271919	0.841426	0.965846
Sample 3	1.475	26.59	18.79	53.145	142.2	0.882934	0.63574	0.696237
Sample 4	1.6	22.57	40.42	35.41	114.38	0.713402	0.474752	0.552174
Sample 5	6.33	28.38	23.6	41.69	114.64	1.526227	0.669922	0.86169
Sample 6	6.41	29.61	34.68	29.3	128.62	1.903421	0.842697	1.004607
Sample 7	2.2	31.74	36.76	29.3	146.81	0.808057	0.657838	0.698125
Sample 8	4.62	28.57	22.16	44.65	194.68	0.091296	0.310805	0.21814
Sample 9	6.45	28.49	20.11	44.95	159.65	0.78806	0.634343	0.667091
Sample 10	8.73	34.23	16.5	40.54	112.43	2.532847	0.783463	1.169553
Sample 11	1.55	41.05	5.86	51.54	167.61	0.256034	0.640731	0.529803

Sample 12	0.91	20.64	27.41	51.04	223.81	0.034967	1.082625	0.228507
Sample 13	0.88	25.07	21.02	53.03	192.09	0.132378	0.387288	0.309609
Sample 14	0.91	21.47	23.89	53.73	199.61	0.050439	0.8677	0.225716
Sample 15	0.88	25.91	20.06	53.15	203.06	0.159783	0.311594	0.276289
Sample 16	11.51	33.49	28.2	26.8	125.31	1.921933	0.844214	1.151644
Sample 17	0.69	22.81	15.52	60.98	206.05	0.078107	0.374714	0.24257
Sample 18	6.975	26.22	15.57	51.235	137.08	1.435746	0.757396	0.927172
Sample 19	3.56	34.96	6.17	55.31	123.79	0.819672	0.670822	0.719359
Sample 20	2.71	29.92	29.01	38.36	144.53	0.527533	0.531835	0.530552
Sample 21	3.05	28.47	25.24	43.24	133.99	0.637731	0.503735	0.546019
Sample 22	6.13	36.11	7.97	49.79	133.43	0.952088	0.538237	0.671547
Sample 23	4.95	31.035	7.29	56.725	122.2	1.139726	0.687831	0.81374
Sample 24	2.9	26.43	46.36	24.31	134.32	0.748512	0.355194	0.444973
Sample 25	9.68	24.45	36.36	29.51	111.63	1.462926	0.447269	0.640488
Sample 26	7.36	23.34	32.19	37.11	120.51	1.096825	0.518536	0.653122

- M.C. = Moisture Content, V.M. = Volatile Matter, F.C. = Fixed Carbon.
- Sample Nos. 1, 2, 3, 7, 9 are from SECL; 5, 6, 8, 10, 16, 18, 25, 26 are from MCL; 4, 19, 20, 21 are from SCCL; 11, 13 are from ECL; 12, 14 are from BCCL; 15, 23, 24 are from CCL; 11 and 22 are from NECL and NCL respectively.

## CONCLUSION

From the study of the thermogram, 14 samples were found to be highly prone to spontaneous heating while rest 6 each were moderately susceptible and poorly susceptible to spontaneous heating. The slope value of Stage II A is a better indicator of determining spontaneous heating. The slope of stage IIA was found to be higher for highly susceptible coals. The correlation between onset temperature and slope IIA & slope II was found to be encouraging. The correlation study confirms that the moisture content of coal is a key factor affecting the spontaneous heating of coal. While the volatile matter content is seemed to have some correlation but the ash content and fixed content of coal are found to have little or no correlation with thermogram key parameters which are already defined above.

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# CHAPTER 1

## 1. Introduction

### 1.1 Overview

Spontaneous Heating is a process of self-heating of coal or other carbonaceous material as a result of auto-oxidation process, which can be the cause of fire in the end. For fire to occur, generally three things are needed, viz. fuel, oxygen, and source of ignition. In spontaneous heating external source of ignition is not required, that makes the process a bit unique. The coal, exposed to the air reacts with oxygen, the process is an exothermic reaction. The products of the reaction are carbon dioxide, water vapor and most importantly heat, as it's an exothermic reaction. If the heat generated from the reaction does not go away in a rate faster than its production, the resultant is the accumulation of heat. The accumulated heat accelerates the process of oxidation of coal, which can be well understood from principles of chemical kinetics. The cumulative heat generated from reaction when remain static at the place of formation, that ends up in igniting a fire.

Fire in the coal fields is a major obstacle in mining operation. The problem is not just confined to any specific region rather it is a global phenomenon. In India the problem is a big headache to every mining industry. Coal fire occurring due to spontaneous combustion accounts for closed to 80%. The main reason behind the large contribution of spontaneous heating in coal fire is that Indian coal seams are largely thick seam. Due to coal fire many lives are lost, not to mention heavy financial losses occurring because of stoppage of production and providing compensation. In underground mines, Hugh sum of coal are left behind due to coal fire viz. Jharia coalfields. In opencast mines, the fire, contributes towards global warming.

Being the third largest coal producer of world, India is one of the biggest consumer of this fossil fuel. Interestingly, India is also the third largest importer of coal. Import of coal is basically for the metallurgical requirements, coking coal. Many industries are also importin thermal coals for the power plant for many reasons, falling international price is being one of them. The dream to become a developed country and to provide employment to its people, rapid industrialization is

taking place. To cater the energy requirements of both industrial and domestic needs, the govt. is focusing on setting up many thermal power plants. Though the reason is obvious as the coal being the cheapest fossil fuel, but the move seems like a step backwards, as the global warming is a major challenge to mankind and our decision to stick to coal, how much justifiable is a moot point.

Spontaneous heating is a problem associated to coal mining since the beginning. So naturally many researchers have been engaged in analyzing the concept behind this complex phenomenon. In order to avoid coal-fire, early measures like prevention of spontaneous heating is a wise thing to do. In order to prevent spontaneous heating, the susceptibility of coal toward it is analyzed. There are many standardized experimental methods to analyze the susceptibility of coal. They are illustrated in Fig 4.1. Many popular methods are for example Crossing Point Temperature (CPT), Differential Thermal Analysis (DTA), Wet oxidation potential method (WOPD), etc.

## 1.2 Objective

The objective of this work was to analyze the spontaneous heating susceptible of collected coal samples by Differential Thermal Analysis (DTA), and its correlation study with the intrinsic properties obtained from proximate analysis.

## 1.3 Methodology

Following steps were followed, in order to complete the project, in a sequential way.

- ❖ **Literature review** – Study and collection of the relevant previous works of different authors, and also collecting informations on this topic.
- ❖ **Collection of sample and its preparation** – Coal samples studied in this work were collected from different coalfields viz. BCCL, CCL, ECL, MCL, NECL, and SECL; for experimentation samples were prepared according to the IS (Indian Standards).
- ❖ **Experimentation** – The experimentation is divided into two stages:
  - Study of spontaneous heating susceptibility of coal by differential thermal analysis (DTA),
  - Study of intrinsic properties of coal by proximate analysis.
- ❖ **Analysis** – The obtained results from DTA were used assess the susceptibility of coal, and the proximate analysis results were used to study the correlation with key DTA Thermograms parameters.

## CHAPTER 2

### 2. Literature Review

There are many works on spontaneous heating of coal. Many research papers have been studied for the requirements of the thesis. Followings are the summaries of some important research works which have been referred in this thesis.

**Banerjee and Chakravarty (1967)** studied the spontaneous heating susceptibility of coal using differential thermal analysis method. A standardized experimental method devised by them is still being used as a guideline. Thermograms obtained from DTA study was used to analyze the susceptibility of coal and coals were classified depending upon their liability to spontaneous heating.

**Mahajan et al. (1976)** analyzed twelve coal samples from different coalfields in the USA using differential scanning calorimetry method. The experimentation was conducted in specific condition like, inert atmosphere using helium; pressure of 5.6 Mpa; temperature up to 580°C. For higher rank coal viz. anthracite, bituminous, the thermal effect was found to be endothermic. But for lower rank coals viz. lignite, sub-bituminous, thermal effect was exothermic. In order to get a comparative analysis, the obtained results from differential scanning calorimetry were compared with DTA results. Some critical conclusions were made on the way DTA Thermograms were being analyzed.

**Marinov (1977)** studied the effect oxygen (O<sub>2</sub>) on the black coal and lignite using DTA-TG method. The medium used in the experiment was air. The layer thickness of coal sample was varied in the experiment. DTA-TG plot obtained from different coal samples showed that the curve was depended upon the layer thickness and rate of heating. Ignition was only found when the layer thickness was high. On thin-layers of coal endothermic zones were created. In thick-layers oxidative de-hydrogenation continued at lower temperature for a small band of temperature. This was a result of parallel reaction, controlled by generation of gas enclosed in the thickness of the layer. An equivalent loss in mass was found varying layer thickness in the absence of self-ignition.

Endothermicity in some reaction phases was believed to be occurring due to hydrogen peroxide development and elimination reaction.

**Clemens et al. (1990)** analyzed the way lower rank coal behave with oxygen at different temperatures from 30°C to 180°C at an interval of 30°C. The thermogram obtained from the DTA showed a sharp exothermic reaction in between oxygen and coal. The exotherm was found to be rising with increase in temperature and that is an indicator about the coal being highly susceptible. DTA was used presuming that it would be helpful while studying the low temperature isothermal oxidation of coal.

**Garcia et al. (1999)** studied the spontaneous heating liability of coal using differential scanning calorimetry (DSC). Non isothermal oxidation of three Columbian coal sample was done and the enthalpies were measured at certain condition for a long 105 days' duration. It was noted that the overall oxidation enthalpies of coal decreased with rise in oxidation, however the fall was not planned. The loss in mass during the long period of experimentation was believed to be the reason for this random behavior. The onset temperature of oxidation increased with increase in oxidation in a highly disciplined way also with increasing rank of coal. It was concluded that the onset temperature is a better indicator in classifying coal according to their liability to spontaneous combustion.

**Panigrahi and Sahu (2004)** analyzed the spontaneous heating liability of coal using ANN (Artificial Neural Network). In the study 31 samples of coal, from different subsidiaries Coal India, were tested. The chemical constituents of coal were determined by ultimate analysis, proximate analysis, and petrographic study. Using five methods viz. DTA, DSC, CPT, WOPD (wet oxidation potential method), and critical air blast method, the susceptibility indices of coal samples under study were determined. A correlation study was done in between susceptibility indices and the results from chemical constituent's analysis. The correlations of proximate analysis components with indices were found to be higher. Classification of coals into four different groups were made using the indices obtained from mentioned five experimentation methods along with the proximate analysis components viz. ash content, volatile matter and moisture content.

**Sahu et al. (2004)** concluded that use of characteristics temperature obtained from differential scanning calorimetry is a better alternative to crossing point temperature for determining the spontaneous heating susceptibility. In the study DSC method was used to determine the onset temperature. In order to compare the result with CPT, CPT was also carried on the coal samples. The results were also predicted with higher precision.



**Kök (2005)** studied temperature controlled combustion of seventeen coal sample, obtained from various collieries of Turkey, using thermogravimetry and differential scanning calorimetry (DSC-TG). In DSC, the heating rate was maintained at 10<sup>0</sup>C per minute till 600<sup>0</sup>C. DSC plots showed different transition stages of coal. It was also analyzed to determine the curve peaks and intervals of the reaction. Using Coats-Redfern and Arrhenius equations, the kinetics was studied and analysis was done on obtained results.

**Okoh (2005)** Okoh focused their study on spontaneous heating susceptibility of coals having low rank. The research was confined to coals of the USA. Spontaneous heating caused fire only constitutes 11% of the total fires in coal mines in USA. The lower rank coal utilization is increasing for power generation purpose, so fire risk is also increasing with it. Coal fires are occurring during handling and transportation of lower rank coals. The moisture percentage in lower rank is generally higher, that is believed to be the reason behind higher susceptibility of lower rank coals. In order to understand the spontaneous combustion process of lower rank coals, the study was focused on to depict the preliminary and final product during oxidation amidst temperature range of 10<sup>0</sup>C to 60<sup>0</sup>C at varying fractional pressure of O<sub>2</sub>. The rate of reaction and tendency towards spontaneous heating was analyzed by initial rate method, for increased mass due to oxygen adsorption. The equipments for the experiment were a FTIR, differential scanning calorimeter and a porosimeter. From the experimentation it was conformed about the relation of oxygen adsorption and heat of the reaction. Using the gas chromatograph, volatiles removed from the coal during oxidation were identified. Lignite and sub-bituminous coals were experimented. The oxygen absorption rate for lignite coals were found to be lower 0.202 mg O<sub>2</sub>/mg, while that of sub-bituminous coals were found to be 6.05 mg O<sub>2</sub>/mg.

**Mohalik et al. (2009)** applied differential thermal calorimetry method to assess the spontaneous heating susceptibility of coal. Various researcher had used different techniques to analyze spontaneous heating potential of coal, some of them were time taking. Under similar experimental condition, the DSC apparatus was used to study the chemical kinetics and types of reaction viz. exothermic or endothermic. A comparative study was done on the way DSC experiments were carried out by various researchers to analyze the spontaneous heating of coal.

**Slovák and Taraba (2010)** studied the oxygen chemisorption process of coal by changing the temperature in the range of 150<sup>0</sup>C to 300<sup>0</sup>C with different experimenting conditions by using DSC-TG. The experimenting conditions like inert material, sample weight, oxygen flow etc. were studied in accordance to reliability and repeatability of results from thermogravimetry curves. The

parameter measuring the auto-oxidation viz. change in mass, sample temperature (at both minimum and maximum), heat evolved at the time of oxidation process, chemical kinetics parameters (E and A) were evaluated. The observed values of E, A and  $T_{\max}$  were found to be within 95% confidence interval. That made sure that these parameters as reliable in characterizing the oxygen chemisorption state of coal. Heat generated during chemisorption was found to be depending upon the initial mass of sample. The values of  $T_{\min}$ ,  $W_H$ ,  $W_o$  were found to be extremely poor. This results complicated the evaluation experiment conditions. The effect of stream of oxygen on the parameters like  $T_{\max}$ , A, E was found to negligible.

**Xu et al. (2013)** examined the way moisture content affects the spontaneous combustion of coal. The spontaneous combustion of coal is affected by moisture percentage. Exothermicity of 4 different types coal with different moisture percentage was analyzed by DSC-TG. The moisture content of could have entirely different for different ranks of coal and different moisture percentage in same rank coals had varying exothermicity altogether. The critical moisture content, which can be defined as the optimum moisture content at which coal is most likely to undergo spontaneous heating, was calculated. The critical moisture was found to be dependable upon the rank of the coal like higher rank coals of anthracite and bituminous had higher value (20 – 25%) and lower rank coals had lower values. Lower critical moisture content increases the susceptibility of coal towards spontaneous heating.

**Li et al. (2014)** devised an experimentation method to measure the slow oxidation kinetics by using DSC-TG method. The analysis of DSC-TG on non-isothermal oxidation of 3 samples was done at a particular rate of heating. The obtained heat release rates and kinetic parameters of low-temperature oxidation were compared with the results of CPT and heat discharge method. The results showed the similar trend of DSC-TG method and CPT techniques while calculating the heat release rate and oxidation kinetics of coal in the temperature ranging from 100°C to 150°C. Moreover, DSC method could be used to get oxidation kinetics of coal for the temperature range of 150°C to the ignition point of the coal. These were different in comparison to those below 150°C. Thus the DSC-TG method referred here was found to be economic while studying the low temperature oxidation kinetics of coal to represent spontaneous heating before its ignition.

**Avila et al. (2014)** estimated the spontaneous combustion liability of coal using thermogravimetry analysis. The reactive properties of coals associated with oxidation at lower temperature were studied with Thermogravimetry. Coal samples of size less than 106 microns were prepared and analyzed using 2 non-isothermal thermogravimetry methods. The coal samples were exposed to

slow rates of heating in air in oxygen adsorption test simultaneously the mass change were recorded. There were two heating rates which were maintained  $3^{\circ}\text{C}$  and  $5^{\circ}\text{C} / \text{min}$ , the total mass was increased during the temperature range of  $20^{\circ}\text{C}$  to  $250^{\circ}\text{C}$ . The mass showed gain up to 4.4%. The lower rate of heating provided higher level of adsorption which eventually led to higher mass gain. The other thermogravimetry test for spontaneous ignition potential ( $\text{TG}_{\text{spi}}$ ) was done which focused on the linear part of mass change plot in between  $150\text{-}350^{\circ}\text{C}$  with varying heating rate ( $3, 5, 7, 10, \text{ and } 20^{\circ}$  per min). A correlation between change in mass and temperature was found with highly reactive coals having higher values. The chemisorption of oxygen was followed by spontaneous heating. The results also had some resemblances with the behaviors of the known samples.

**Zhang et al. (2015)** analyzed the oxidation potential of bituminous coal by FTIR and DTA-TG methods. DTA and FTIR analysis were conducted while changing the heating rate on three different types of bituminous coals. The mass loss, enthalpies and gas obtained in the process of experimentation were analyzed. The observed results from the experiment showed that above three were in mutual correspondence at stages 1 to 4. The important gaseous products from the processes were carbon monoxide, carbon dioxide, methane, and water vapor. The released gas depended upon the reaction characteristics of active structures. The concentration of released gases had the following decreasing order  $\text{CO}_2, \text{H}_2\text{O}, \text{CO}, \text{CH}_4$ . Carbon dioxide constitutes around 90% of the total gas. The correlation coefficient was more than 0.95 for absorbance and temperature of released gas. The obtained results from the experimentation showed that, the increase in volatile matter decreases the characteristics temperature and increases spontaneous heating tendency while the released gas concentration increases and the rate of heat release also higher.

## CHAPTER 3

### 3. Theories of Spontaneous Heating of Coal

“It may be defined as a complex physico-chemical process by which the freshly exposed coal at ordinary atmospheric temperature when comes in contact with oxygen catches fire due to auto-oxidation and without the application of heat from any external source.”

#### 3.1 Mechanism of Spontaneous Heating

The spontaneous heating of coal generally occurs due to tendency of self-heating caused by auto-oxidation process. The oxidation process, is an exothermic reaction, occurs when coal is in contact with air. The products of the reactions are carbon dioxide, vapors and heat. If the heat is dissipated, the process towards self-sustained combustion can be stopped. But it is important to point out that this will not stop oxidation and it will not accelerate the process either. That is called low-temperature oxidation. But if the heat generated if remains static surrounding the coal, then the rate of oxidation reaction increases following the principle of chemical kinetics. In due course of time, accumulated heat become the cause of coal fire in mine.

Coal fire which is caused by spontaneous heating depends, firstly on the coal quality, and secondly the surrounding conditions to which coal is exposed. If the following three conditions are satisfied, then spontaneous heating is sort of inevitable.

- The form in which coal is present, if that can help in oxidation is at ambient temperature.
- If oxygen ( $O_2$ ) is available for oxidation.
- If heat accumulation is possible.

#### 3.2 Major Theories of Spontaneous Heating of Coal

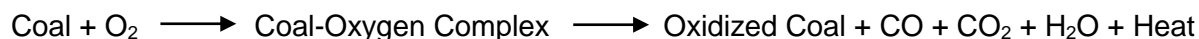
There are several theories for explaining the mechanism involving the spontaneous heating of coal. Among them generally five theories are of quite important in nature. They are discussed briefly in the following page.

- i. Coal-Oxygen Complex Theory
- ii. Pyrite Theory
- iii. Bacterium Theory
- iv. Phenol Theory
- v. Electrochemical Theory

### 3.2.1 The Coal-Oxygen Complex Theory

The formation of peroxy-radical and hydro-peroxide is believed to be the process by which moisture and oxygen initially come to organic matrix. These kinds radical can re-arrange, react or may be decomposed to generate various range of oxygen functionality in the product coming out of the reaction process. The important factors favoring oxidation process are coal type, moisture, temperature. Sequential Stages of spontaneous heating is given in the Fig. 3.1.

When freshly exposed coal is exposed to the atmosphere, it absorbs the oxygen in the atmosphere onto its surface. When the coal is absorbed, a series of chain reaction occurs which oxidizes the coal. As all oxidation reactions produces heat, the reaction between coal and oxygen is no different. The reaction described can simply be expressed as below.



The generated heat from the oxidation process is around 2 – 4 cal/ml of absorbed oxygen at N.T.P.

As a result of this, temperature of coal rises which further increases the rate of absorption of oxygen ( $\text{O}_2$ ) and generation of heat. If the heat produced from the reaction does not dissipate by conduction or radiation or by both in rate quicker than the production of heat, a rapid increase in the temperature of coal is observed which again accelerates the rate of oxygen absorption and generation of heat till the ignition point of coal. The ignition point temperature is around  $160^\circ - 170^\circ\text{C}$  for bituminous coal and for anthracite, it is around  $185^\circ\text{C}$ .

The reaction mechanism between coal and oxygen is very complex. The reaction generally occurs in the following four steps.

- In the first stage, coal oxygen complex is formed and heat is generated.
- In second stage, the complexes formed in the first stage are decomposed, which gives carbon dioxide ( $\text{CO}_2$ ) and water molecules. In addition to this some more groups of molecules are formed specially carbonyl ( $-\text{C}=\text{O}$ ), carboxyl ( $-\text{C}(=\text{O})-\text{OH}$ ), and phenolic ( $-\text{OH}$ ) and also heat.

- In the third stage, the decomposition of groups formed in the second stage takes place at temperature higher than  $100^{\circ}\text{C}$  which results in formation of  $\text{H}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{CO}$ ,  $\text{CO}_2$  and other higher degree of hydrocarbons viz. ethane, ethylene, propylene and heat.
- In the final stage aliphatic structure consisting unsaturated and saturated hydrocarbons are decomposed and  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{H}_2\text{O}$  are produced.

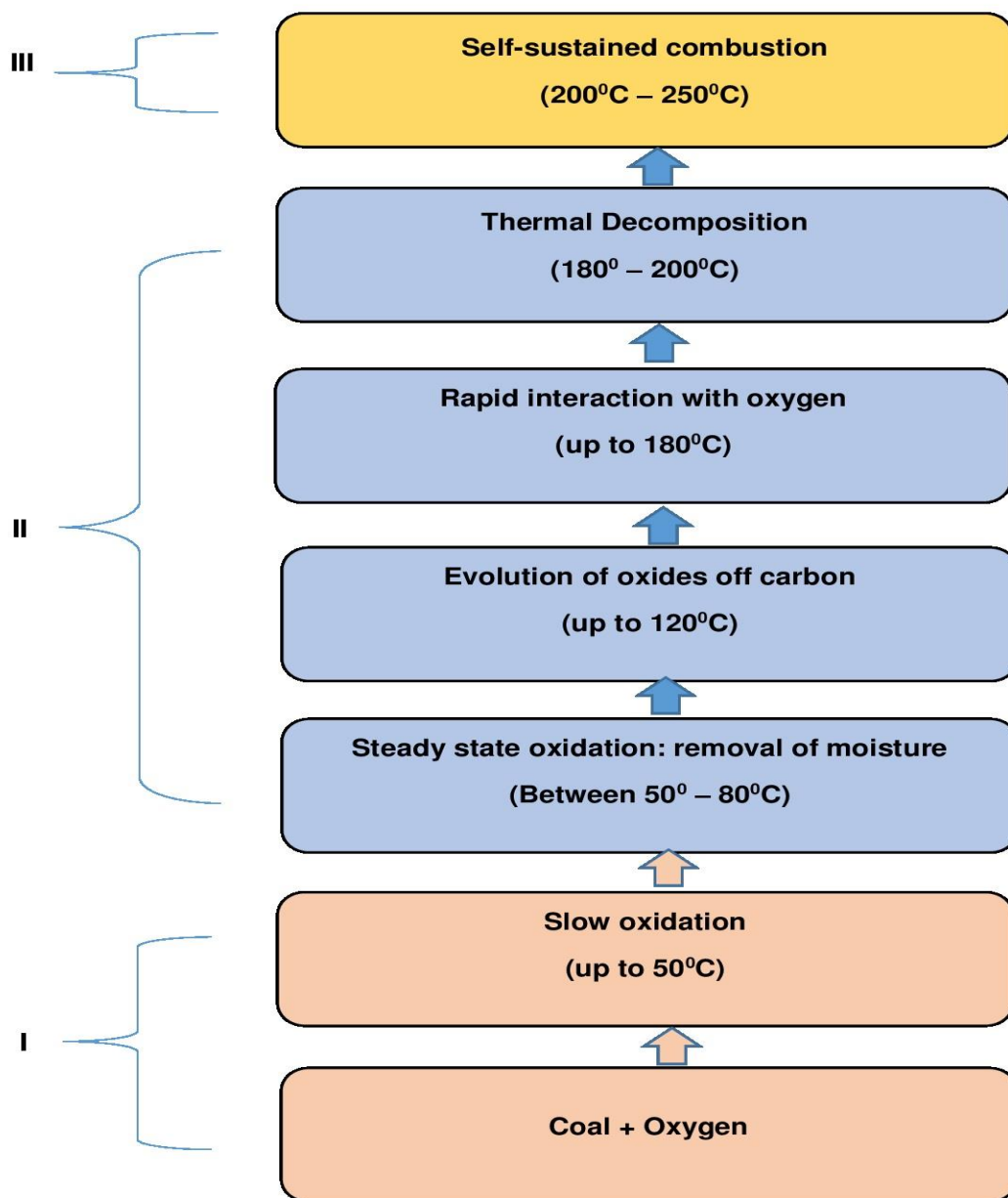


Fig 3.1 Sequential stages in spontaneous heating of coal (Benerjee et al., 1985)

### 3.2.1 Pyrite Theory

Pure pyrite (Iron-polysulfide) consists of iron (47.67%) and sulfide (53.33%). It reacts with the oxygen present in the atmosphere at N.T.P. which generates heat and ultimately responsible for spontaneous heating. Pyrite can be expressed by Fe-(S<sub>3</sub>)-Fe-(S<sub>3</sub>)-Fe or simply FeS<sub>2</sub>.

The presence of iron pyrite in fine powder form, dispersed state can trigger the heating in the presence of moisture. The pyrite present in the coal can break down the coal into smaller pieces and thus the surface area of the coal is increased which is exposed to the atmosphere. It also increases the temperature of coal, as its own oxidation process generates heat. When the iron-pyrite reacts with oxygen and moisture, the products generated are of higher volume than the initial volume of pyrite, which results in cracking open the coal and exposing it to more oxygen. The reaction is also an exothermic reaction as it's an oxidation process.



Sulphonated coal have greater tendency to react with oxygen, with the presence of iron oxide reactivity is also increased. From the previous research it has been found that if the pyrite is below 5%, the effect is miniscule, but it has significant implication if the amount exceeds 5% to 10%.

### 3.2.3 The Bacterium Theory

The bacteria present in the coal encourage the self-heating of coal due to bacterial-action. For an example spontaneous heating seen in in the wood and haystack are caused due to the action of bacteria. Well, there is no definitive material to prove or discard this theory. So it is accepted that may cause a little bit of heating in coal but that is not in a big way.

### 3.2.4 The Phenol Theory

The phenol theory considers the phenolic hydroxyls and poly-phenols found in the coal have higher oxidation rates in comparison to other groups present in the coal. Thus the method helps in assessing the spontaneous heating liability of coal.

### 3.2.5 The Electrochemical Theory

This theory suggests that the auto-oxidation process of coal is due to the oxidation-reduction reaction occurring in the micro-galvanic cells constituted by coal components.

## 3.3 Factors Affecting the Spontaneous Heating of Coal

The process of spontaneous heating is a result of complex set of events contributing this phenomenon. The phenomenon is arduous to apprehend thanks to many factors viz.

- Intrinsic factors (Nature of Coal, characteristics of coal, & seam factors)
- Extrinsic factors (Atmospheric, Geological & Mining Conditions)

### 3.3.1 Intrinsic Factors

The intrinsic factors causing the spontaneous heating in coal are the followings

- i. Composition, rank of the coal, & petrographic constituents of coal.
- ii. Moisture Content
- iii. Iron pyrites
- iv. Bacteria
- v. Friability, size of coal particles, and surface area exposed to atmosphere
- vi. Other minerals

### 3.3.2 Extrinsic Factors

#### Mining factors

- i. Mining methods
- ii. Rate of advance
- iii. Pillar conditions
- iv. Roof condition
- v. Crushing
- vi. Packing
- vii. Presence of timber or other organic waste material in abandoned areas or dumps
- viii. Leakage
- ix. Multi-seam working
- x. Coal losses
- xi. Worked out areas
- xii. Heat from machines
- xiii. Stowing
- xiv. Ventilation system and airflow rate
- xv. Ventilation pressure
- xvi. Method of stockpiling and stockpile compaction

#### Geological factors

- i. Coal seam and surrounding strata condition
- ii. Seam thickness
- iii. Seam gradient
- iv. Caving characteristics
- v. Faulting and other geological disturbances
- vi. Coal outbursts
- vii. Friability
- viii. Depth of cover

#### Climatic conditions

- i. Temperature
- ii. Moisture content/relative humidity
- iii. Barometric pressure
- iv. O<sub>2</sub> concentration



### 3.3.3 Coal Characteristics

#### ❖ Rank of Coal

The capability to absorb the oxygen depends upon the rank of the coal. The oxidation rate decreases with increase in rank of the coal. Lower rank coals have higher susceptibility to spontaneous heating. Because the lower rank coals have higher moisture content, volatile matter, and oxygen (O<sub>2</sub>).

#### ❖ Pyrite in the coal

The iron-pyrite (FeS<sub>2</sub>) present in the coal substantially increases the chances of coal to undergo auto-oxidation process. The situation become serious when the pyrite concentration is above 2% and present in a finely divided state. Iron pyrites accelerates the rate of spontaneous heating by swelling and fracturing the coal to smaller pieces. This increases the total surface area exposed to the atmosphere. More surface area leads to more rapid oxidation of the coal. The oxidation of pyrite further accelerates the spontaneous heating / auto-oxidation of coal. The reaction between pyrite and oxygen & moisture is given by the following equation.



#### ❖ Exposed Area of Coal Surface

The self-heating of coal is greatly influenced by the exposed surface area of coal and also the internal surface area of coal. The total pore surface area of coal can be more than 80m<sup>2</sup>/g. The smaller size coal has more surface area exposed to atmosphere, so the rate of oxidation is also higher in this state of coal. It can be concluded that the rate of oxidation increases with rise in fineness of coal. From previous research (Schmidt, 1945) it has been believed that the rate of oxidation is proportional to cube root of the internal coal surface area. For an average bituminous coal, the spontaneous heating is less likely to occur if the size is larger than 38mm.

#### ❖ Freshness of Exposed Surface of Coal

The rate of coal oxidation decreases with time depending upon the freshness of the exposed surface area of coal. If the coal is weathered and the exposed area reaches the saturation state, then no further interaction occurs with oxygen at ambient temperature.

#### ❖ Petrographic Constituents of Coal

Petrographic constituents of coal like clarain, fusain, durain, and vitrain are known to affect the spontaneous heating liability of coal. The susceptibility of coal toward spontaneous heating follows the following decreasing order vitrain > clarain > durain > fusain.

#### ❖ **Volatile Matter of Coal**

The more is the volatile matter content of coal, the more is the tendency towards auto-oxidation. From a finding, it is known that the coal having 38% VM oxidizes three time faster than the coal having volatile matter 18%. From various research it has been found that coals having VM content more than 28% are more prone to spontaneous heating.

#### ❖ **Moisture Content**

The moisture in the coal is of generally two type viz. inherent moisture and extraneous moisture. Inherent moisture is dependent upon the way coal was formed millions year ago. Moisture in the coal acts as catalytic agent in the oxidation reaction. The higher the moisture content of coal the higher is the coal likely to undergo self-heating. From a study by Nandy et. al. (1967) it is found that the optimal moisture content of coal is around 5%, at which the tendency of coal towards spontaneous heating is highest.

#### ❖ **Oxygen Content**

The tendency of coal towards spontaneous heating is directly dependent upon the oxygen content of coal. If the oxygen content is less than 2%, then it does not help in increasing the oxidation rate of coal.

#### ❖ **Ash Content**

The ash present in the coal decreases the rate of oxidation of coal. The composition of ash also affects the spontaneous heating of coal.

#### ❖ **Thermal Conductivity of Coal**

The rate of dissipation of heat is determined by thermal conductivity of coal. Lower the thermal conductivity of coal, higher is the tendency of coal towards spontaneous heating.

### **3.3.4 Geological Factors**

#### ❖ **Thickness of seam**

Thickness of coal seam is responsible for spontaneous heating tendency of coal. The thicker seams are highly inclined towards spontaneous heating. In thick seams, complete extraction is not possible, so huge amount of coal is left behind. These coals in contact with oxygen present in mine atmosphere cause oxidation. The unmined part which is left in the goaf also prone to heating due to sluggish ventilation system. When the floor heaves due to high stress, floor coal is broken and more surface area is exposed to air. In the coal seam some bands are more prone to spontaneous heating than other bands.

**❖ Dip of Seam**

As the dip of the coal seam increases the spontaneous heating problem also increases. The temperature difference caused due to oxidation process triggers the convection of air in goaf. Due to difference in densities of the constituents of mine air viz. CH<sub>4</sub>, N<sub>2</sub>, CO<sub>2</sub>, the flow of air occurs by buoyancy. The air flow in the goaf area increases the chances of spontaneous heating of coal.

**❖ Depth of Mine**

At shallow depth the coal seam is connected to the surface by fractures, joints. The air and moisture can get easy access to the seam and increases the liability towards spontaneous heating. At medium depth also due to geo-thermal gradient the coal is liable spontaneous heating. At more depth the in-situ stress is very high, which crushes the pillar and eventually exposes more surface area of coal.

**❖ Multiple Seams**

In the presence of multiple seams affects the spontaneous heating of coal. The joints, fractures etc. developed in the partings leads to spontaneous heating in another unaffected seam especially unmined seams.

**❖ Caving Characteristics**

During the depillaring operation the strata is caved in which causes the fracture in the coal and in the presence of shale etc. as roof material the oxidation process catches the peak rate.

**❖ Faulting**

The fault present in the coal seams gives easy access to the air and moisture to get in the coal seams and helps in oxidation process. And the grinding of coal in the fault plane also leads to self-heating.

**❖ Coal Friability**

If the coal is easily friable the fine coal is produced, owing to larger surface area exposed to the atmosphere the coal is likely to undergo spontaneous heating.

**3.3.4 Mining Factors****❖ Mining Methods**

In long-wall advancing method, there is a chance of air getting into excavated portion of mine, which leads to oxidation of coal in the goaf. The differential ventilating pressure encourages flow of air across these portion which triggers the problem.

#### ❖ **Multi Seam Workings**

In multi-seam workings the unmined seams are generally affected by the working in the upper seams from where the cracks are opened and connected to the lower seams. It results in self-heating of coal.

#### ❖ **Rate of Advance**

The rate of advancement in the mining operation highly affects the spontaneous heating of coal. If the rate of advance is slow then the coal is exposed to the air for longer duration which causes the coal to oxidize for a longer period which worsens the spontaneous heating of coal.

#### ❖ **Size of Pillar**

The proper design of pillar should be formed or else the pillar will not sustain heavy stress coming on it and will gradually be crushed and which will lead the air through the cracks developed in the pillar. The loose coal formed by crushing of pillar causes serious problem in the case of sluggish ventilation.

#### ❖ **Regulators, Stoppings, Doors, and Air Crossings**

The leakage points should be checked regularly and must be properly sealed. The higher air pressure can cause the air to leak through the cracks in the coal. Air leakage is dependent upon the pressure across the stoppings.

#### ❖ **Obstruction in the Roads**

The obstruction in the road-ways viz. mine cars, stockpiles etc. can force the air to take a route through the cracks and cause spontaneous heating problem.

#### ❖ **Air Flow Rates**

The rate of production of heat should be more than the dissipation of heat, in order to trigger the spontaneous heating. In a good ventilating air current sweeps the generated heat from the coal surface which results in lower rate of oxidation. But the sluggish ventilating current cannot clear the generated heat from the oxidation which results in higher rates of self-oxidation. "A critical flow rate is one that provides sufficient oxygen for widespread oxidation but does not dissipate the heat generated."

### **3.4 Stages of Spontaneous Heating**

There are generally three stages of spontaneous heating of coal in the coal mine.

- Incubation Period
- Indication Period
- Open fire

### 3.4.1 Incubation Period

“The incubation period is the time period in between the onset of first oxidation and the time-point when one can detect it by senses.” Otherwise it can be said that the time period in between the starting of coal excavation in a panel / district and the appearance of first sign of heating. One simply cannot know about incubation period just by passing through some mine workings. The incubation period depends upon wide range of factors.

- Coal characteristics
- Thickness of coal seam
- Type of roof
- Mining method
- Continuity in working
- Leakage of air and accumulation of heat in the environment

The incubation period normally varies from 3 to 6 months for lower rank coals, for higher rank coals it's in between 9 to 18 months.

### 3.4.2 Indication Period

The indication period follows the end of incubation period. It can be observed that strata is sweating and haze can be seen during this period which is formed by warmed air coming in contact with the cooler coal seams, metallic surfaces depositing moisture. The duration of indication period is not as long as incubation period. The period can sometimes last only for few hours and often ends with the appearance of 'fire stink.'

### 3.4.3 The Open Fire

The open fire immediately follows the indication period. During open fires the seldom burn with a bright-blue flame rather it glows with a bluish-white smoke cloud.

## CHAPTER 4

### 4. Experimental Investigation

In order to study the spontaneous heating susceptibility, various types of standardized experiments are there. The experimental techniques can be broadly divided into three categories which are illustrated in the following Fig 4.1

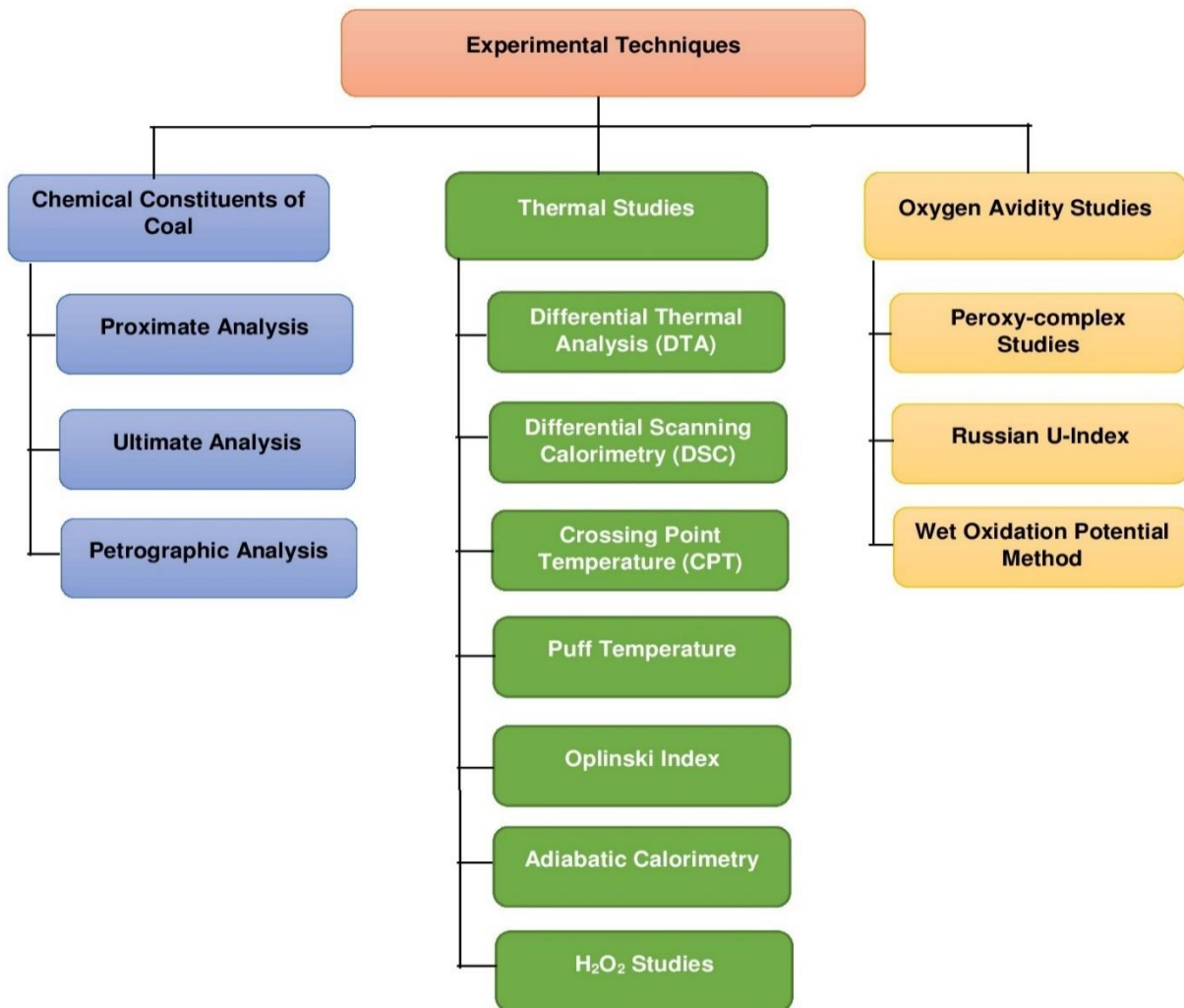


Fig 4.1 Experimental Techniques for Studying Spontaneous Heating (Mohalik et al., 2009)

## 4.1 Collection of Sample and its Preparation

Sampling is the process through which chemical and physical properties of the collected mineral/ore could be determined with required precision. Sample collection should be done in a such a way that the collected sample could represent the entire material from where it is collected. In this project work, total 26 samples of coals were collected from different coalfields viz. MCL, SECL, SCCL, ECL, CCL, BCCL, NECL, and NCL are studied. There are different types of sampling methods viz. Channel Sampling, Grab Sampling, Chip Sampling, Drill Hole Sampling, Bulk Sampling. The Channel Sampling process is briefly discussed below.

### 4.1.1 Channel Sampling

The channel sampling method is done as per the IS 436 Part I / Section I – 1964. The coal face exposed to the air is subjected to the process of oxidation. So in the channel sampling a cross section of  $30 \times 10 \text{ cm}^2$  is marked i.e. width, 30 cm and depth, 10 cm. Two lines separated by 30 cm is marked which are perpendicular to the bed of the coal seam. Excluding the dirt bands, the channel in between the marked zone is chiseled to get fresh non-oxidized coal sample. The collected coal samples are collected on clean cloth sort of things and immediately sealed in the air tight container. The process can be easily understood from the following figure Fig. 4.2.

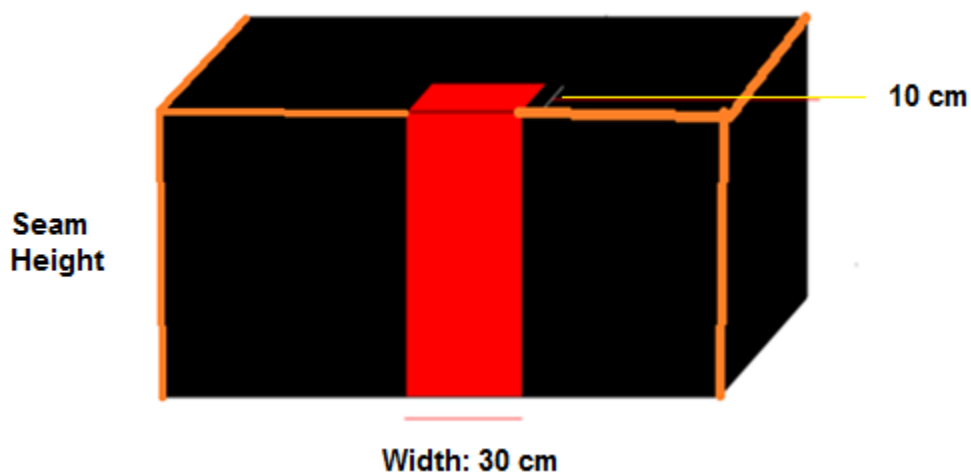


Fig 4.2 Channel Sampling

### 4.1.2 Preparation of Sample

The sample was crushed and sieved as per required size for experimentation. The sieve of  $-212\mu$  (BSS 72) was used to sieve the prepare the sample. The prepared samples were kept in the air tight polythene pouch.

## 4.2 Proximate Analysis

Proximate analysis is done generally to find out the constituents of the coal, while ultimate analysis is conducted to determine the elemental constituents of the coal. There are four parameters which are determined by proximate analysis. They are discussed below briefly. For the determination of components of proximate analysis IS 1350 – Part 1 (1969) is followed.

### 4.2.1 Moisture Content

The moisture associated with the coal is of two types viz. inherent moisture and external moisture. The external moisture is added to the coal during its handling time. So it can be simply removed by air-drying the coal. But the inherent moisture was added to the coal during its formation, million years ago, which is chemically bound to the coal, so it cannot be simply removed by just air-drying the coal like for external moisture. The inherent moisture can be removed from the coal sample by heating the coal above 100°C.

#### ❖ Procedure

For the determination of moisture content of coal, around 1 gm of coal sample of size -212 $\mu$  was taken in a watch glass. Its initial weight was taken and then it was placed in an electric oven for a duration of 90 minutes at temperature of 110°C. After the completion of 90 minutes the watch glass was taken out from the oven using protective gloves and kept in a desiccator for a period of 15 minutes. After that the watch glass was taken out and measured with the help of high precision digital balance. The moisture was calculated as per the following formula:

$$\text{Percentage of Moisture (M.C.)} = \frac{B-C}{B-A} \times 100$$

Where,

A = empty watch glass weight, in gram;

B = weight of the watch glass with around 1 gm of coal sample, in gram;

C = final weight of watch glass with coal after completion of the heating period, in gram;

B – C = weight of the moisture, in gram;

B – A = weight of the coal sample, in gram.

### 4.2.2 Volatile Matter

Volatile Matter content can be defined as the components of coal, excluding moisture, which are liberated at high-temperature in the absence of air. These components are generally consisting



of smaller and longer chain hydro-carbons, aromatic-hydrocarbons, some sulfur, hydrogen, oxygen containing compounds, carbon monoxide, etc.

#### ❖ Procedure

To determine the volatile matter of coal a different kind of silica crucible is used (height: 38 mm, diameter external: 25 mm, diameter internal: 22 mm). At first the empty crucible with lid was weighed. Then 1 gm coal of -212 $\mu$  was weighed on a paper and then the sample was put in the crucible and the lid was put on the crucible. After that the crucible was put in the muffle furnace for a period of 7 minutes at temperature of 925 $^{\circ}$ C. After the completion of 7 minutes the crucible was carefully taken out of the muffle furnace using steel tongue and gloves, and allowed to cool for some period. Then the final weight of the crucible was taken. The calculation of volatile matter was done using the following formula.

$$\text{Percentage of Volatile Matter} = \frac{B-C}{B-A} \times 100 - M. C.$$

Where,

A = Weight of the crucible with lid, in gram;

B = Weight of the crucible (+ lid) with the coal in it, in gram;

C = Weight of the crucible (+lid) with coal in it after heating, in gram;

B – A = Weight of coal sample, in gram;

B – C = Weight of the volatile matter content and moisture, in gram.

#### 4.2.3 Ash Content

The ash in the coal can be defined by the residue left behind after the complete combustion of coal. It is to be noted that the ash and mineral matter are different, not the same. There are two types of mineral matter viz. inherent and extraneous mineral matter. The inherent mineral matter is associated with coal during its formation process. The inorganic matter combined with the organic matter of the coal during its formation. The inherent mineral matter cannot be separated by washing of the coal. Indian coals have generally high amount of inherent mineral matter owing to its drift origin of formation.

The extraneous mineral matter of coal consisting of materials viz. pyrite, shale, gypsum, calcium, magnesium, ferrous carbonate etc. The rocks, dirt getting mixed with the coal while handling the coal is of extraneous type of mineral matter. It can be separated with higher efficiency by coal-

washing process. Overall Indian coals have higher ash content making it unsuitable for metallurgical process.

#### ❖ Procedure

The empty silica crucible was heated in muffle furnace in order to ensure that no other mineral matter is present in the coal. The crucible was properly cleaned and then its initial weight was taken. 1 gm of coal sample of -212 $\mu$  was put in the crucible. Then the crucible was put in the muffle furnace for a duration of 30 minutes at temperature 450°C and then the temperature of the furnace was raised to 850°C for a 60 minutes' period. The crucible was taken out of the furnace carefully using steel tongue with gloves on hand. The final weight of the crucible was taken. The calculation was done as per the following formula.

$$\text{Percentage of Ash} = \frac{C-A}{B-A} \times 100$$

Where,

A = Weight of the empty silica crucible, in gram;

B = Weight of the crucible with the coal, in gram;

C = Weight of the crucible after heating, in grams;

C – A = Weight of the ash, in gram;

B – A = weight of the coal sample, in gram

#### 4.2.4 Fixed Carbon

The fixed carbon content of coal is different from the total carbon content of coal. The fixed carbon content is always less than the total carbon content of coal. Fixed carbon gives a rough estimation about of amount of coke will be obtained from that coal. The fixed carbon content was calculated using the following formula:

$$\text{F.C.} = 100 - (\text{M.C.} + \text{V.M.} + \text{A})$$

#### 4.2.5 Results of Proximate Analysis

The proximate analysis of 26 coal samples was done in order to determine the intrinsic properties of the coal samples under study. The results of the proximate analysis are presented in the Table 4.1

**Table 4.1 - Proximate Data of Coal Samples**

Sample No.	M.C.	V.M.	Ash	F.C.
Sample 1	2.35	29.57	28.71	39.37
Sample 2	2.27	24.55	11.08	62.1
Sample 3	1.475	26.59	18.79	53.145
Sample 4	1.6	22.57	40.42	35.41
Sample 5	6.33	28.38	23.6	41.69
Sample 6	6.41	29.61	34.68	29.3
Sample 7	2.2	31.74	36.76	29.3
Sample 8	4.62	28.57	22.16	44.65
Sample 9	6.45	28.49	20.11	44.95
Sample 10	8.73	34.23	16.5	40.54
Sample 11	1.55	41.05	5.86	51.54
Sample 12	0.91	20.64	27.41	51.04
Sample 13	0.88	25.07	21.02	53.03
Sample 14	0.91	21.47	23.89	53.73
Sample 15	0.88	25.91	20.06	53.15
Sample 16	11.51	33.49	28.2	26.8
Sample 17	0.69	22.81	15.52	60.98
Sample 18	6.975	26.22	15.57	51.235
Sample 19	3.56	34.96	6.17	55.31
Sample 20	2.71	29.92	29.01	38.36
Sample 21	3.05	28.47	25.24	43.24
Sample 22	6.13	36.11	7.97	49.79
Sample 23	4.95	31.035	7.29	56.725
Sample 24	2.9	26.43	46.36	24.31
Sample 25	9.68	24.45	36.36	29.51
Sample 26	7.36	23.34	32.19	37.11

- ❖ Sample Nos. 1, 2, 3, 7, 9 are from SECL; Sample Nos. 5, 6, 8, 10, 16, 18, 25, 26 are from MCL; Sample Nos. 4, 19, 20, 21 are from SCCL; Sample Nos. 11, 13 are from ECL; Sample Nos. 12, 14 are from BCCL; Sample Nos. 15, 23, 24 are from CCL; Sample No. 11 and 22 are from NECL and NCL respectively.

### 4.3 Differential Thermal Analysis

Differential Thermal Analysis (DTA) is one among the different thermo-analytic methods. In DTA the studied-material and the inert reference material (generally alpha- alumina) are made to under-go same thermal cycles, at the same time the temperature difference between the sample and the reference is monitored. The differential temperature is then plotted against the time, or temperature.

#### 4.3.1 DTA Apparatus

A DTA apparatus (Schematic Diagram Fig 4.3) consists of sample holder, sample container, thermocouple, a furnace, metallic post, temperature programmer, recording system. The important feature is that the two thermos-couples are connected to a single voltmeter. On one thermos-couple reference material (alpha alumina:  $\text{Al}_2\text{O}_3$ ) is kept in sample container and on another sample container sample under study (coal sample) is kept. When the temperature is increased there will be some deflection in the voltmeter if the sample is undergoing a phase change process.

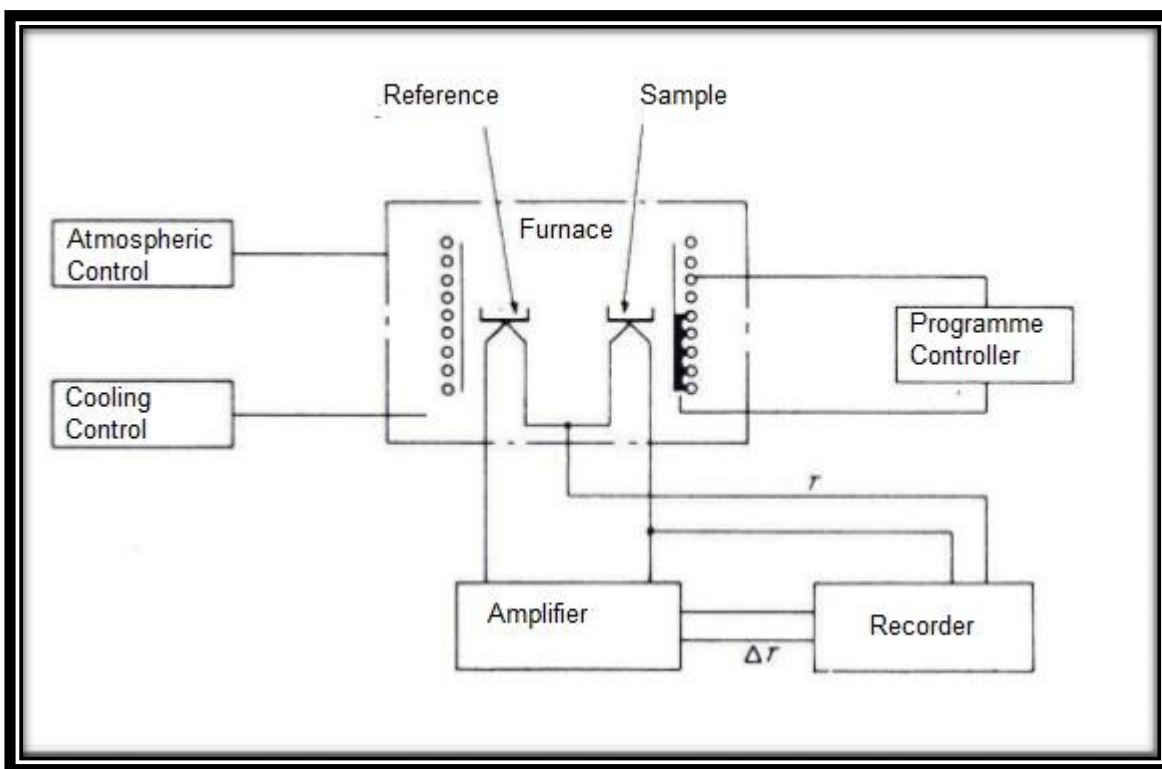


Fig 4.3 Schematic Diagram of Differential Thermal Analysis Apparatus. ([http://bit.ly/DTA\\_](http://bit.ly/DTA_))

### 4.3.2 Experimental Procedure

The DTA study is conducted by the standardized procedure laid down by Banerjee and Chakraborty (1967). At first in the tiny sample holder some coal was put, such that the combined weight of the sample and the sample holder was about 66 mg. Then the sample holder was put back in the DTA ceramic / metallic post and the weight was zeroed. After that the sample holder was taken out and more 10 mg of coal was put in the sample holder. The sample holder was then put back on the thermocouple and the furnace was lowered. The Programme was opened and the heating rate was specified at 5°C/min up to 450°C. The data were automatically recorded and after approximately 90 minutes the Programme was stopped automatically. The generated thermogram was then analyzed for determining the onset / characteristics temperature ( $T_c$ ) and various stages of slope values. The set up for the experimentation is given below in Fig. 4.4 and a sample thermogram obtained from DTA is given in Fig. 4.5



**Fig 4.4 Photograph of the experimental setup of DTA**

1) Differential Thermal Analyzer (TG-DTA 60/60H, Shimadzu, Japan), 2) TA –60 WS Data Logger, 3) Furnace 4) Blower for cooling 5) Computer for Analyzing the Data.

#### ❖ Onset Temperature Determination

The characteristics temperature or the onset temperature is determined by the below given way.

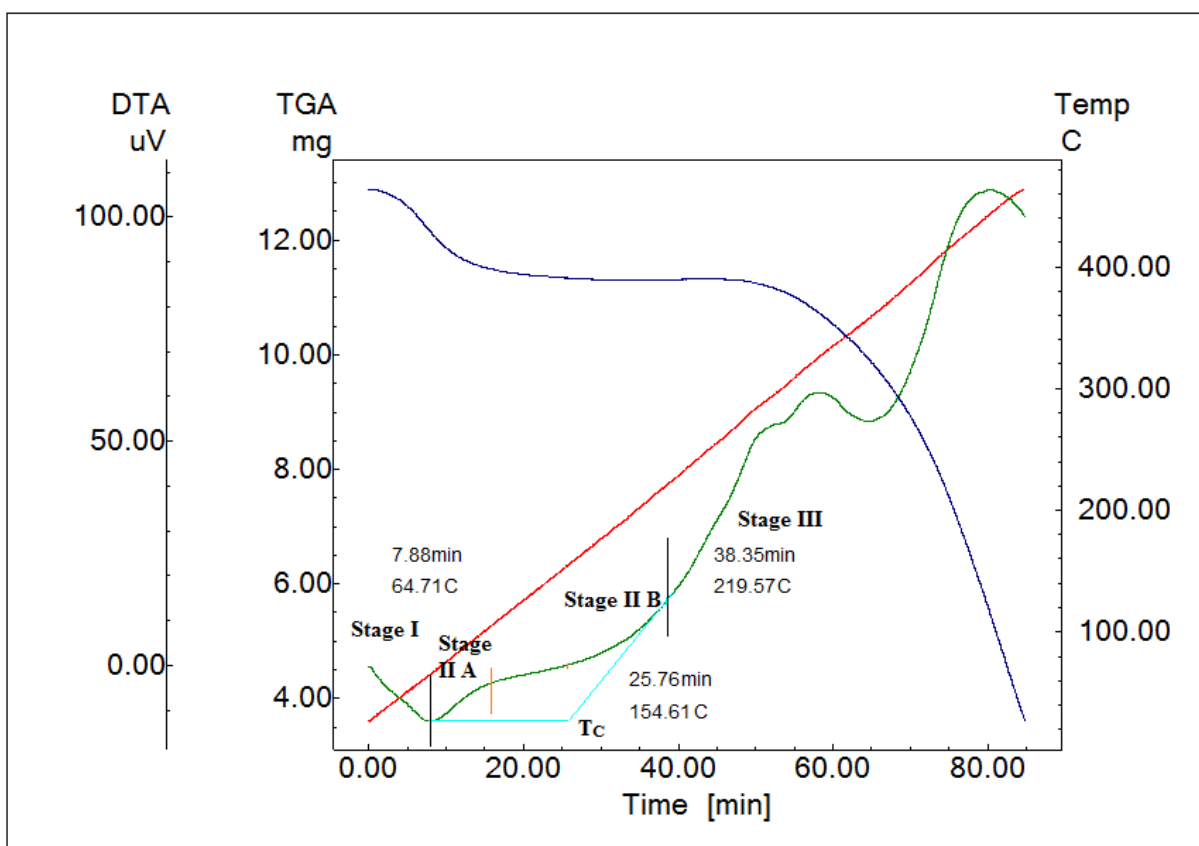
- The peak point of the endothermic region on thermogram is marked.
- For drawing the tangent, the peak point which is also known as inflection point is first selected and then another point is selected at the rising portion of the stage III. Then

clicking on the specific tab (exit) the tangent is generated. The tangent is little bit adjusted to make the tangent to inflexion point horizontal.

- The intersection of the two tangents provides the onset temperature / characteristics temperature.

#### ❖ Determining the Slope Angle

The generated thermogram from the DTA is further analyzed for determining the slope angle. The thermogram is basically divided into three stages. The first stage is the endothermic region; it is due to the releasing of moisture present in the sample (coal). The stage III is not so reliable for studying the spontaneous heating nature of coal. So the stage II is given utmost importance. The stage II is further divided into two region stage - IIA and stage - IIB. The slopes of the stage - II, IIA, IIB are obtained for further analysis of spontaneous heating potential of the coal.



**Fig 4.5 Different stages of Thermogram**

#### 4.3.3 Results from DTA Analysis

The Differential Thermal Analysis was done on 26 coal samples in order to study their spontaneous heating susceptibility. A sample thermogram is given in Fig 4.5. The results from

the experimentation are provided in the Table 4.2. All the DTA Thermograms obtained from the analysis are given in the Appendix – 1.

**Table 4.2 - DTA results of Coal Samples**

Sample Name	Onset Temp. (in °C)	Slope Values		
		II A	II B	II
Sample 1	154.6	1.089474	0.797551	0.870364
Sample 2	133.1	1.271919	0.841426	0.965846
Sample 3	142.2	0.882934	0.63574	0.696237
Sample 4	114.38	0.713402	0.474752	0.552174
Sample 5	114.64	1.526227	0.669922	0.86169
Sample 6	128.62	1.903421	0.842697	1.004607
Sample 7	146.81	0.808057	0.657838	0.698125
Sample 8	194.68	0.091296	0.310805	0.21814
Sample 9	159.65	0.78806	0.634343	0.667091
Sample 10	112.43	2.532847	0.783463	1.169553
Sample 11	167.61	0.256034	0.640731	0.529803
Sample 12	223.81	0.034967	1.082625	0.228507
Sample 13	192.09	0.132378	0.387288	0.309609
Sample 14	199.61	0.050439	0.8677	0.225716
Sample 15	203.06	0.159783	0.311594	0.276289
Sample 16	125.31	1.921933	0.844214	1.151644
Sample 17	206.05	0.078107	0.374714	0.24257
Sample 18	137.08	1.435746	0.757396	0.927172
Sample 19	123.79	0.819672	0.670822	0.719359
Sample 20	144.53	0.527533	0.531835	0.530552
Sample 21	133.99	0.637731	0.503735	0.546019
Sample 22	133.43	0.952088	0.538237	0.671547
Sample 23	122.2	1.139726	0.687831	0.81374
Sample 24	134.32	0.748512	0.355194	0.444973
Sample 25	111.63	1.462926	0.447269	0.640488
Sample 26	120.51	1.096825	0.518536	0.653122

## CHAPTER 5

### 5. Discussion & Conclusion

#### 5.1 Discussion

From previous studies it has been found that coal can be categorized into different groups using onset temperature as a key indicator. Panigrahi and Sahu (2004) concluded that coal can be divided into three types depending upon their tendency towards spontaneous heating using characteristics temperature obtained from DTA studies.

**Table 5.1 - Classification of Coal using Onset Temperature**

Temperature Range	Remark
120°C – 140°C	Highly Susceptible
140°C – 170°C	Moderately Susceptible
➤ 170°C	Poorly Susceptible

Using the above classification, we can easily conclude that the sample no. 2, 4, 5, 6, 10, 16, 18, 19, 21, 22, 23, 24, 25, 26 are highly liable to spontaneous heating, sample no. 1, 3, 7, 9, 11, 20 are moderately susceptible towards spontaneous heating while sample no. 8, 12, 13, 14, 15, 17 are less prone to spontaneous heating.

#### 5.1.1 Correlation Study

The intrinsic properties of coal like moisture content and volatile matter content are known to influencing the spontaneous heating potential of coal. In order to analyze the effect of intrinsic properties, proximate analysis of all coal samples were conducted in this study. After obtaining the proximate data of coal samples correlation plots were made using MS-Excel. The correlation plots were obtained in between individual the proximate parameters vs key thermogram parameters from DTA study. The obtained correlation coefficients are presented in the Table 5.2. All the correlation plots are given in the Appendix - 2.



**Table 5.2 – Correlation Analysis Results (Correlation Coefficient R)**

Variables	Moisture Content	Volatile Matter	Ash Content	Fixed Carbon
Onset Temperature ( $T_c$ )	0.8459	0.4428	0.3776	0.4407
Slope IIA	0.8132	0.5409	0.1403	0.4403
Slope IIB	0.3983	0.2273	0.2356	0.0574
Slope II	0.7291	0.5749	0.0374	0.3039

From the correlation analysis the moisture content of coal was found to have impressive correlation coefficient with onset temperature, slope IIA, and slope II.

The correlation of volatile matter content of coal was found to be somewhat correlated, but the correlation was not quite as expected (to be higher). The correlations with ash content and fixed carbon were found to be little to none. The ash content and fixed carbon content do not have any direct effect on spontaneous heating of coal.

## 5.2 Conclusion

In this thesis work, using the differential thermal analysis 26 coal samples were studied for determining their susceptibility towards spontaneous heating. The proximate analysis of the coal samples was conducted for determining the intrinsic properties of the coal. The correlation study was also done to relate the proximate parameters with the key properties of the thermogram.

Some important results obtained from this study are described below:

- First of all, the objective of the study to classify the tested coal samples according to their susceptibility towards spontaneous heating was done i.e. the sample no. 2, 4, 5, 6, 10, 16, 18, 19, 21, 22, 23, 24, 25, 26 were highly liable to spontaneous heating, sample no. 1, 3, 7, 9, 11, 20 were moderately susceptible towards spontaneous heating while sample no. 8, 12, 13, 14, 15, 17 were less prone to spontaneous heating.
- As per expectation the moisture content of the coal had good correlation with the onset temperature, slope IIA, and overall slope II of stage II of the thermogram.
- The ash content and fixed carbon content did not seem to have much correlation with the key parameters of the thermogram.
- The onset / characteristics temperature was found to be have significant correlation with the slope value of stage IIA and good correlation with overall slope value of stage II.

- The coal samples whose moisture content was around 4 – 7%, found to be highly susceptible toward spontaneous heating.
- The moisture content and volatile matter contents are contributing in a major way combining towards spontaneous heating potential of coals.

**Scope for Future Studies:**

- The spontaneous heating susceptibility can also be studied by other thermal analysis methods, especially using Differential Scanning Calorimetry (DSC).
- The thermogram obtained from the DTA also has thermogravimetry curve, which can be used to obtain the mass loss during the experimentation. From that, moisture can be obtained which can be correlated with the moisture content obtained from the proximate analysis.

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# **APPENDIX - 1**

## **(DTA Thermograms)**

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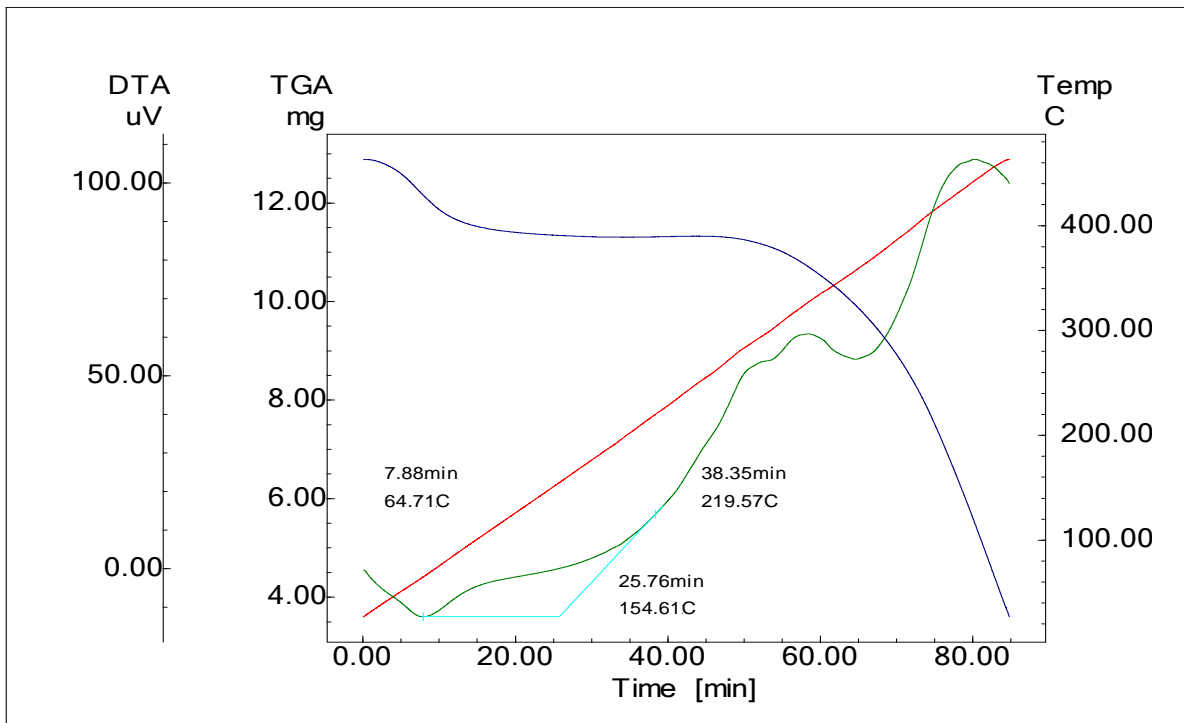


Fig. A1.1 DTA thermogram for sample no. 1

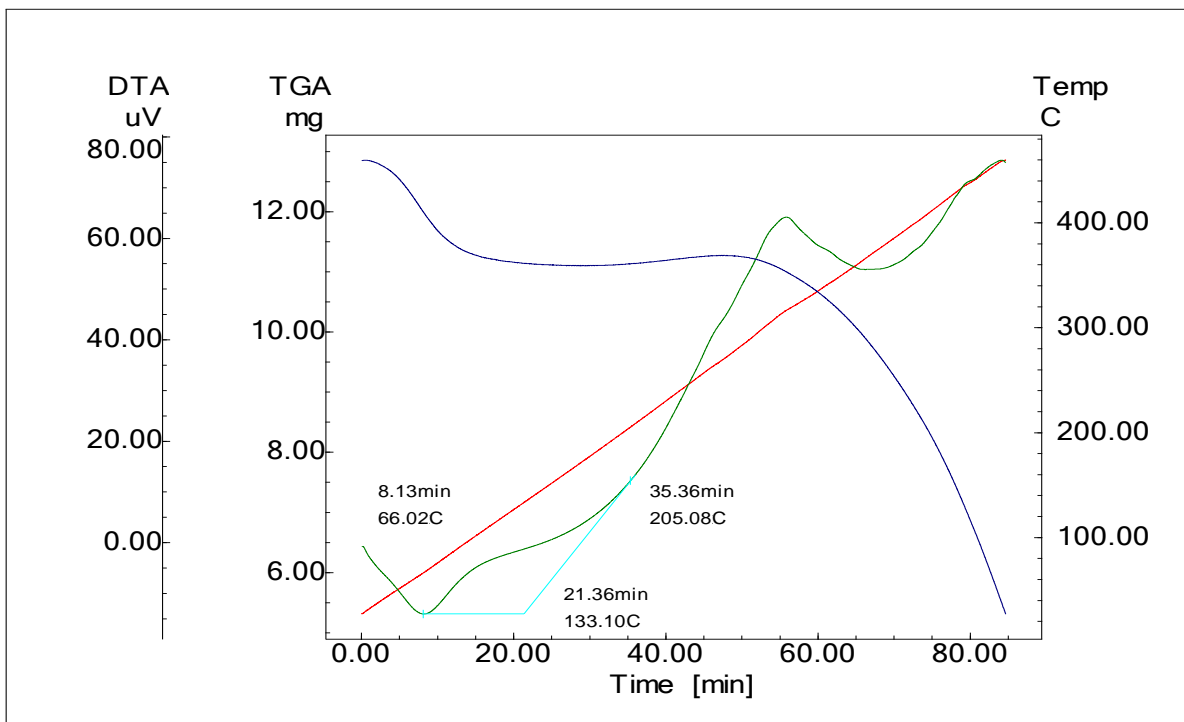


Fig. A1.2 DTA thermogram for sample no. 2

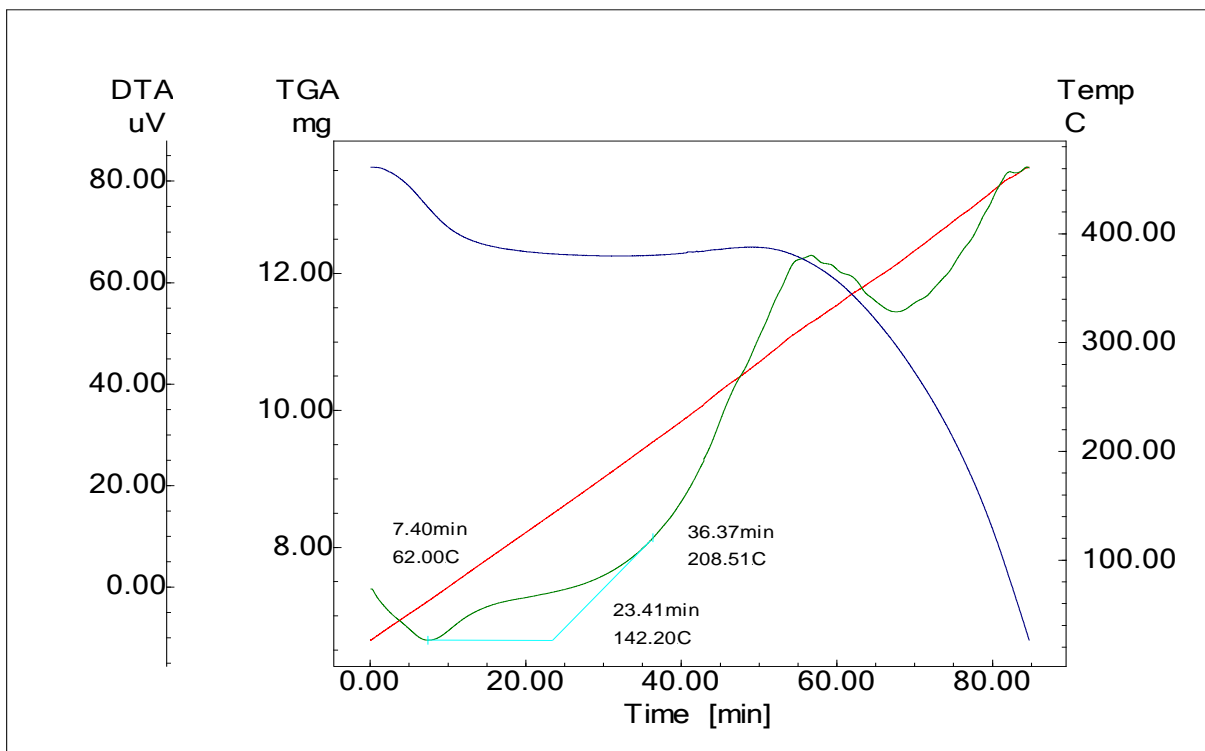


Fig. A1.3 DTA thermogram for sample no. 3

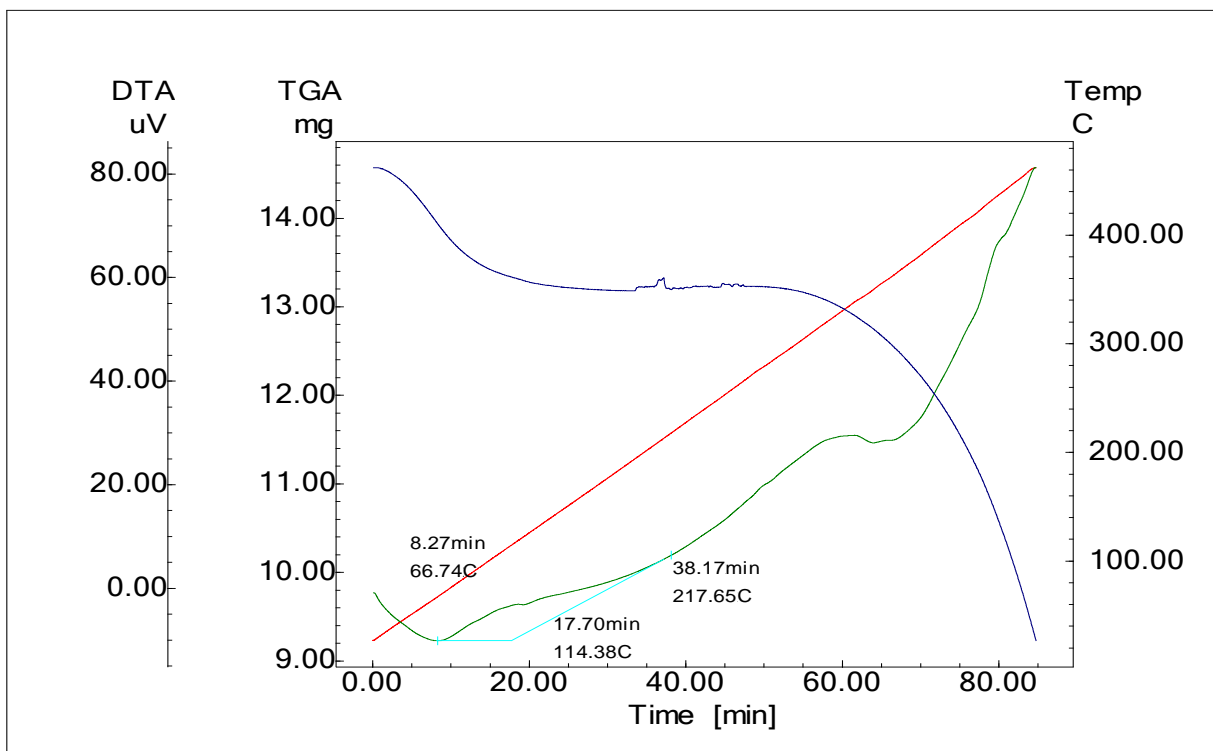


Fig. A1.4 DTA thermogram for sample no. 4

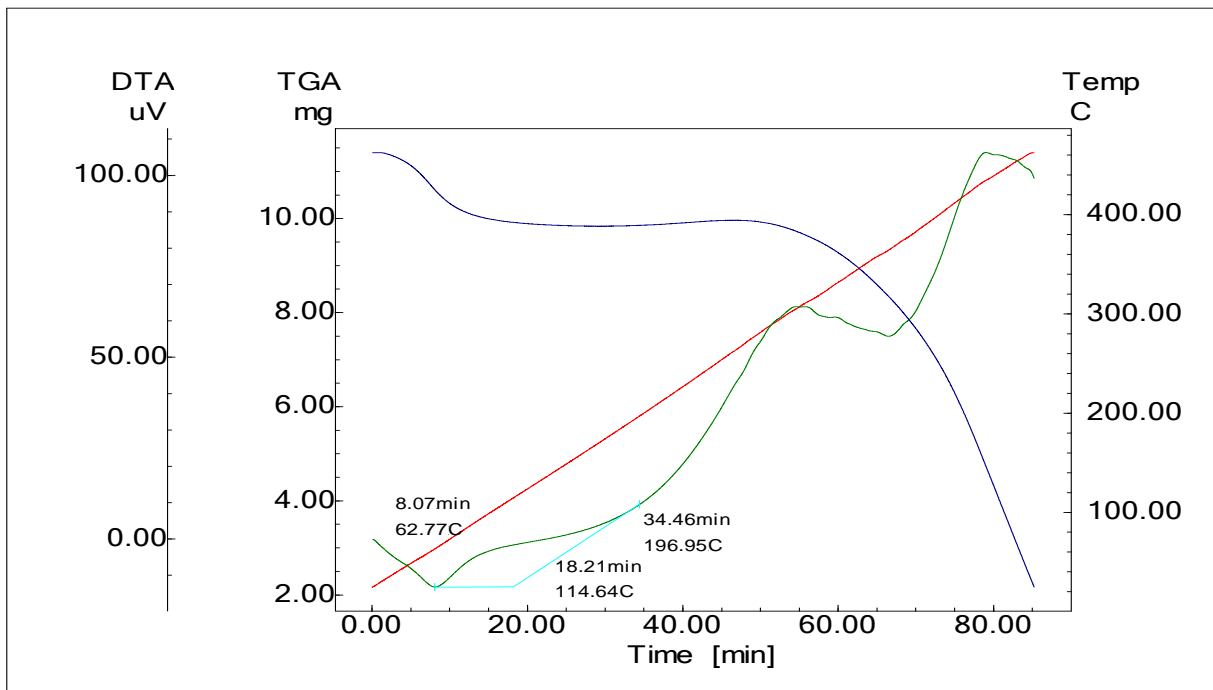


Fig. A1.5 DTA thermogram for sample no. 5

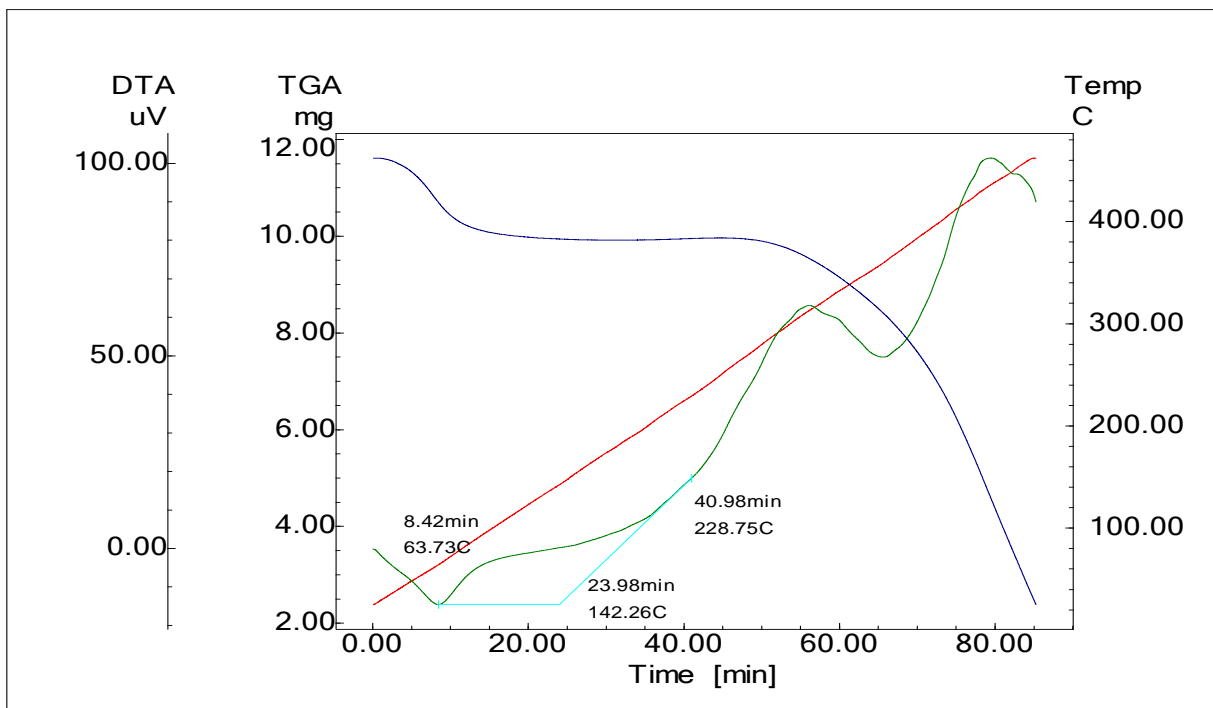


Fig. A1.6 DTA thermogram for sample no. 6



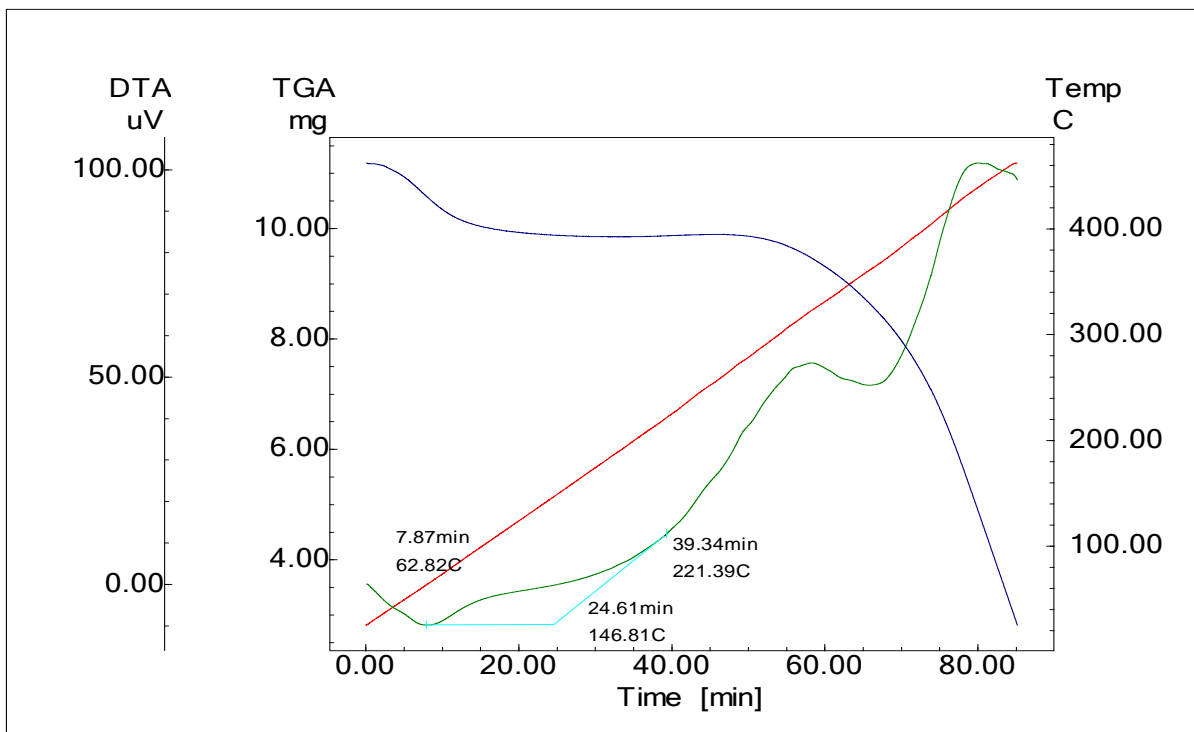


Fig. A1.7 DTA thermogram for sample no. 7

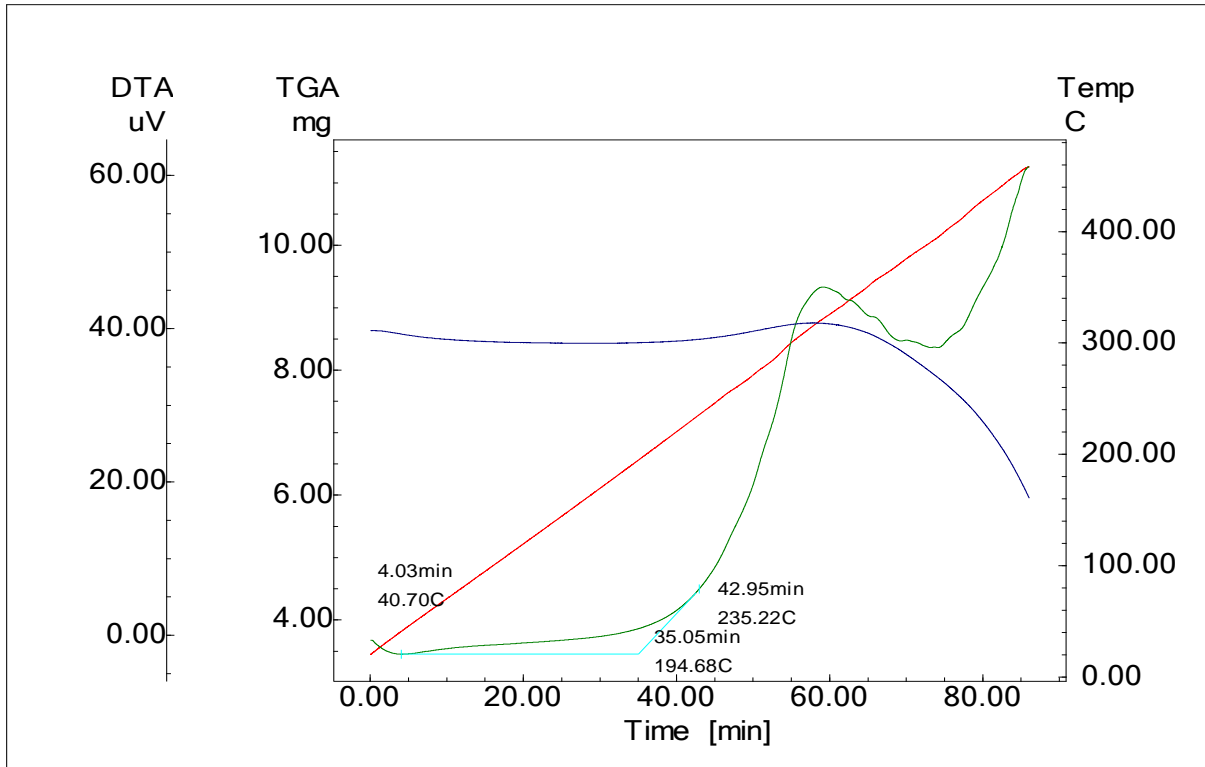


Fig. A1.8 DTA thermogram for sample no. 8

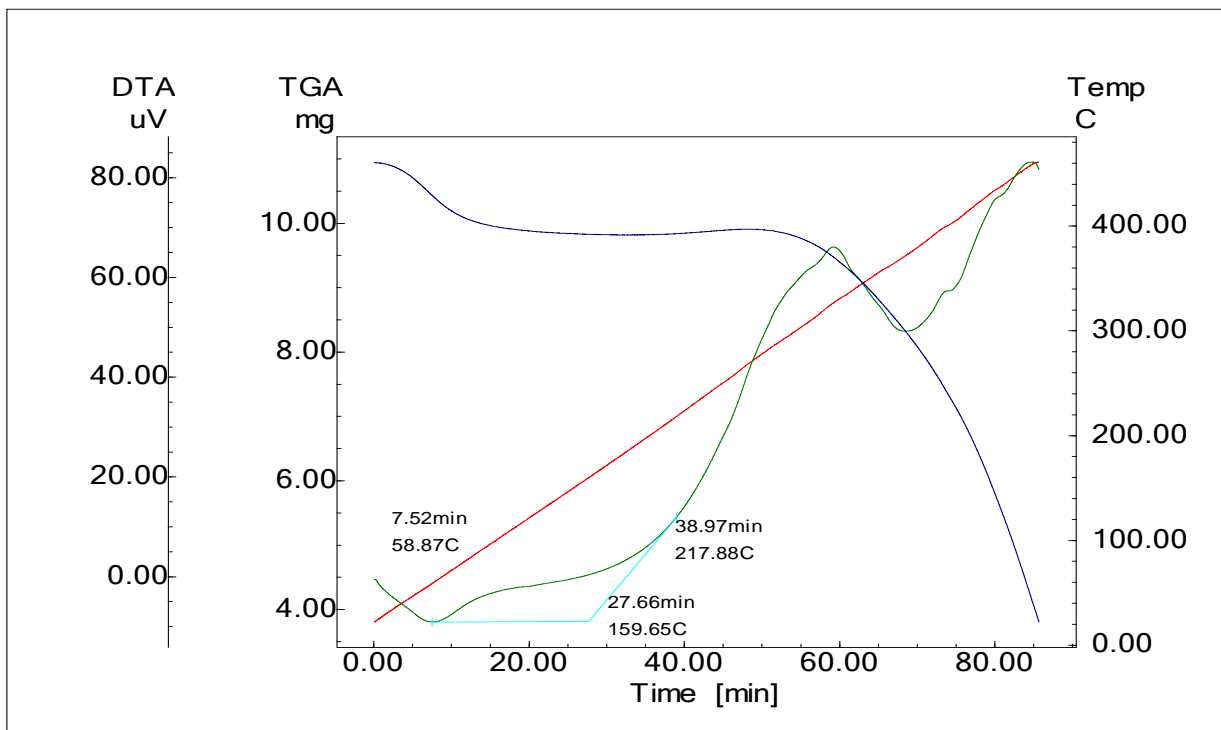


Fig. A1.9 DTA thermogram for sample no. 9

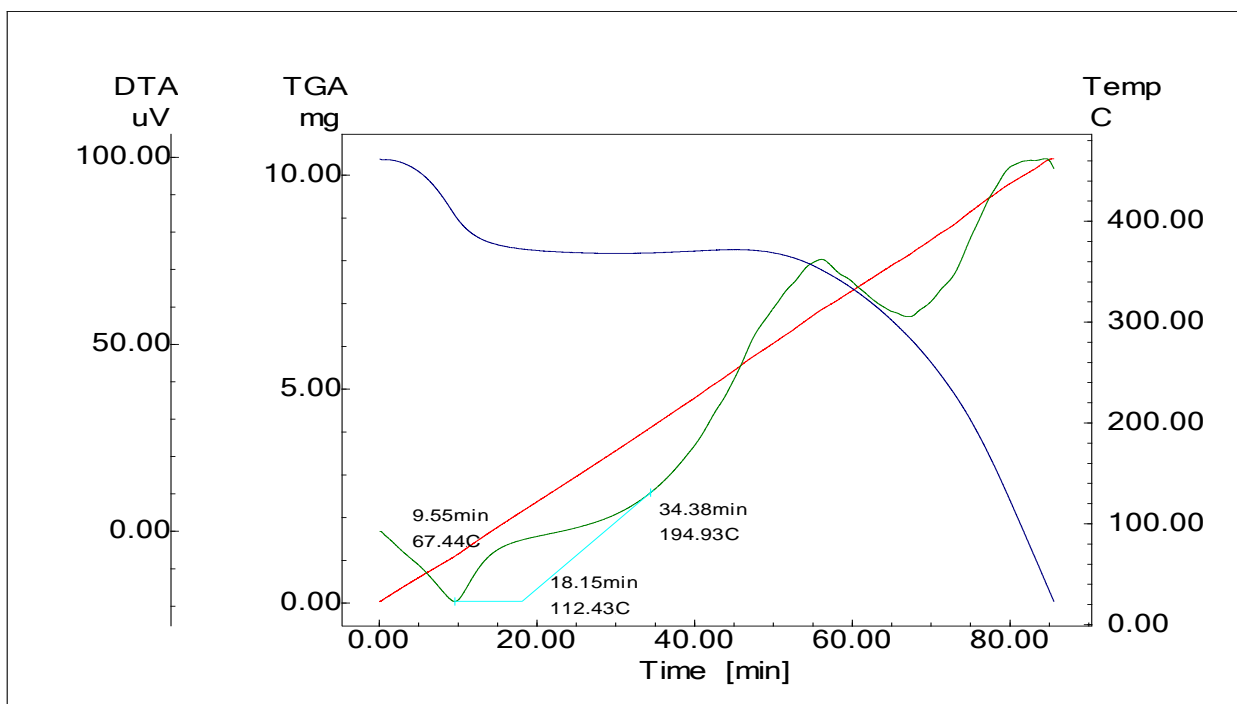


Fig. A1.10 DTA thermogram for sample no. 10

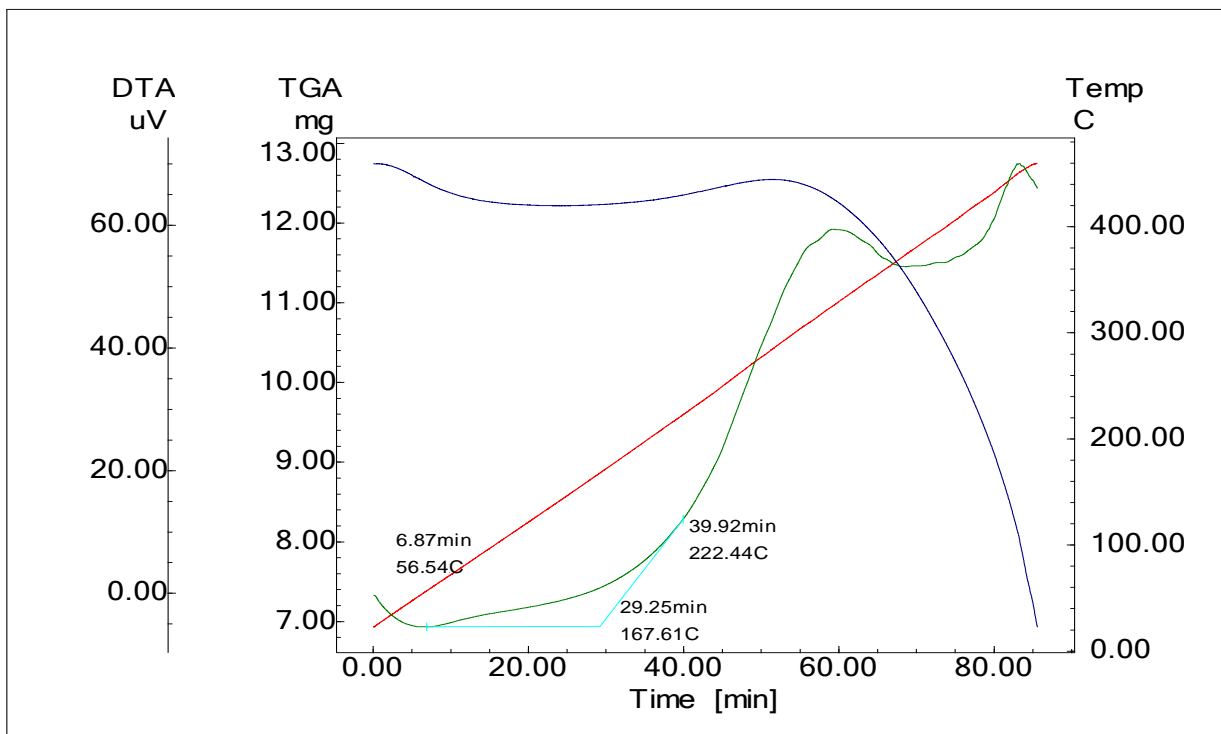


Fig. A1.11 DTA thermogram for sample no. 11

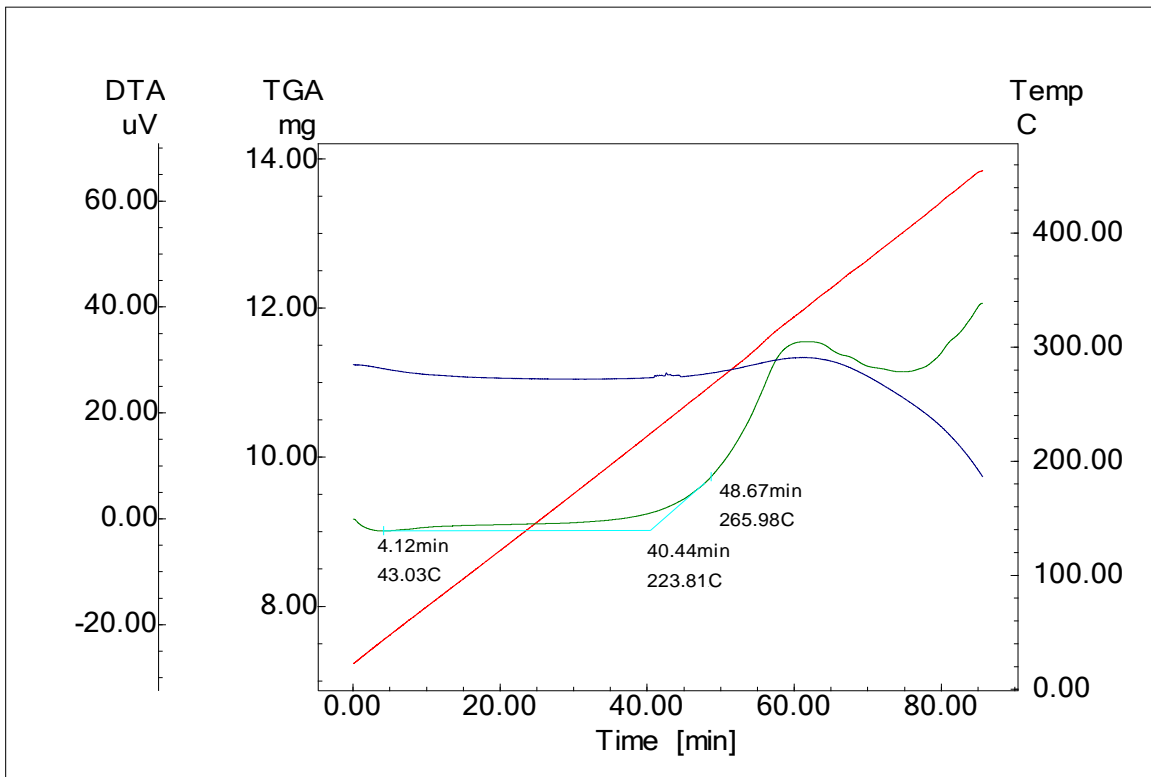


Fig. A1.12 DTA thermogram for sample no. 12

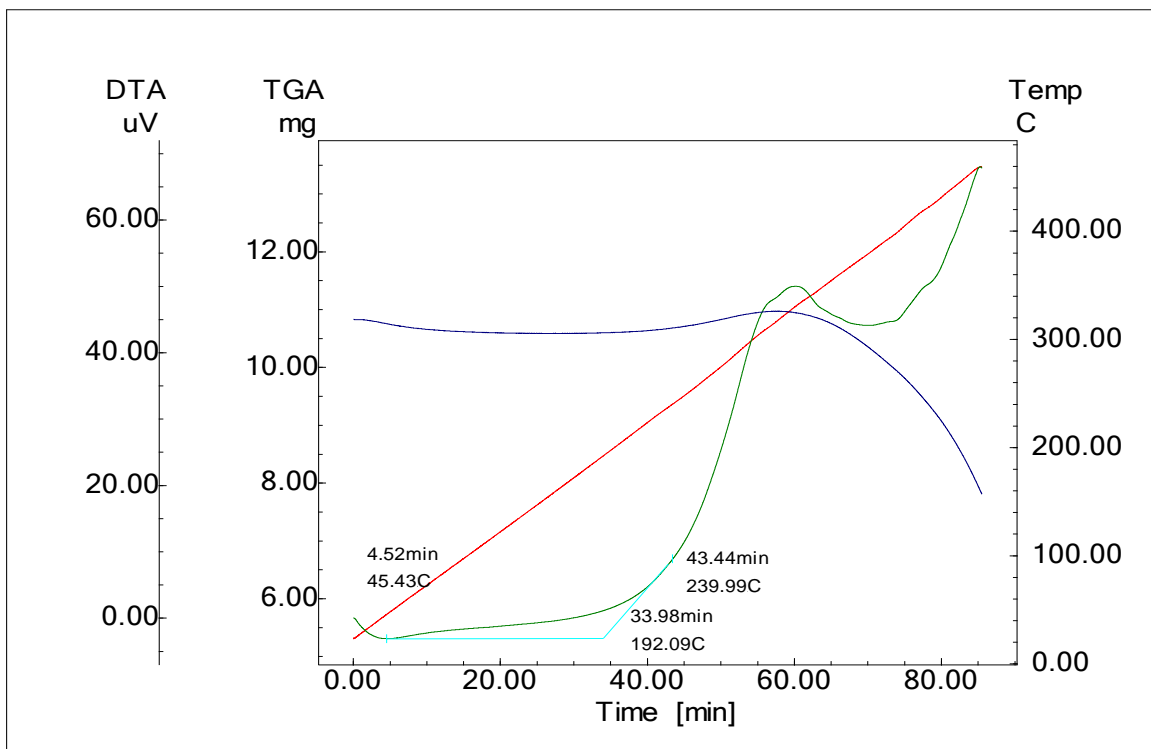


Fig. A1.13 DTA thermogram for sample no. 13

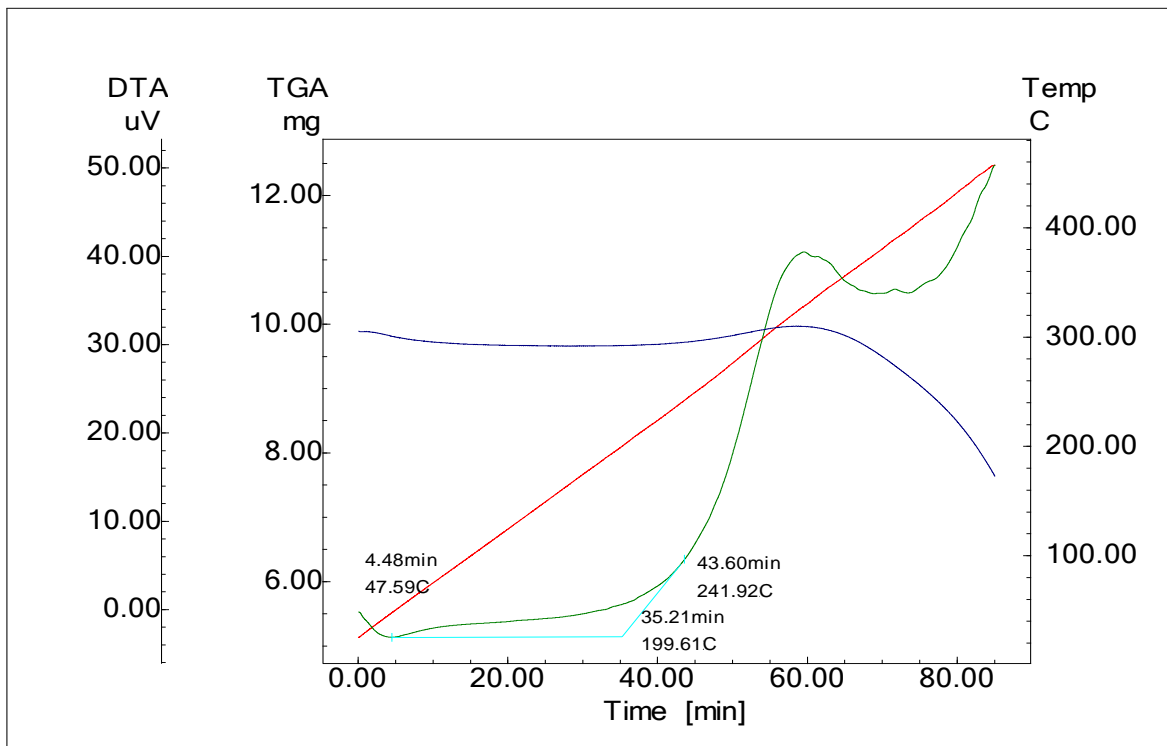


Fig. A1.14 DTA thermogram for sample no. 14

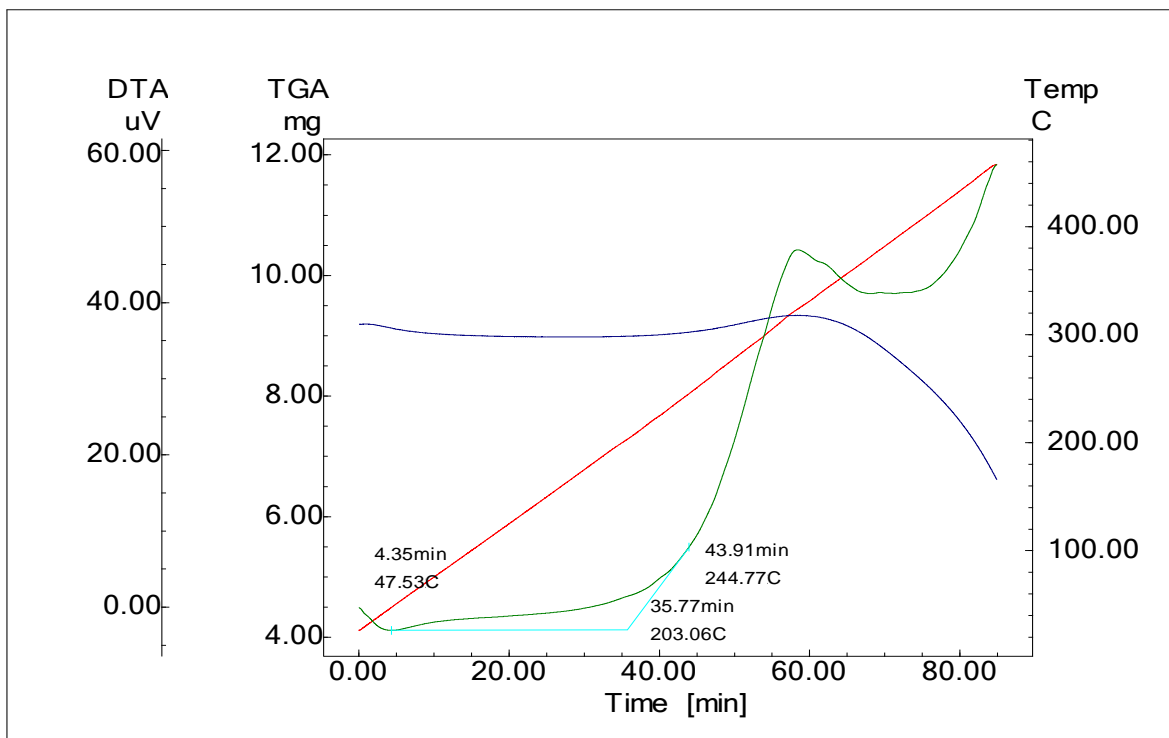


Fig. A1.15 DTA thermogram for sample no. 15

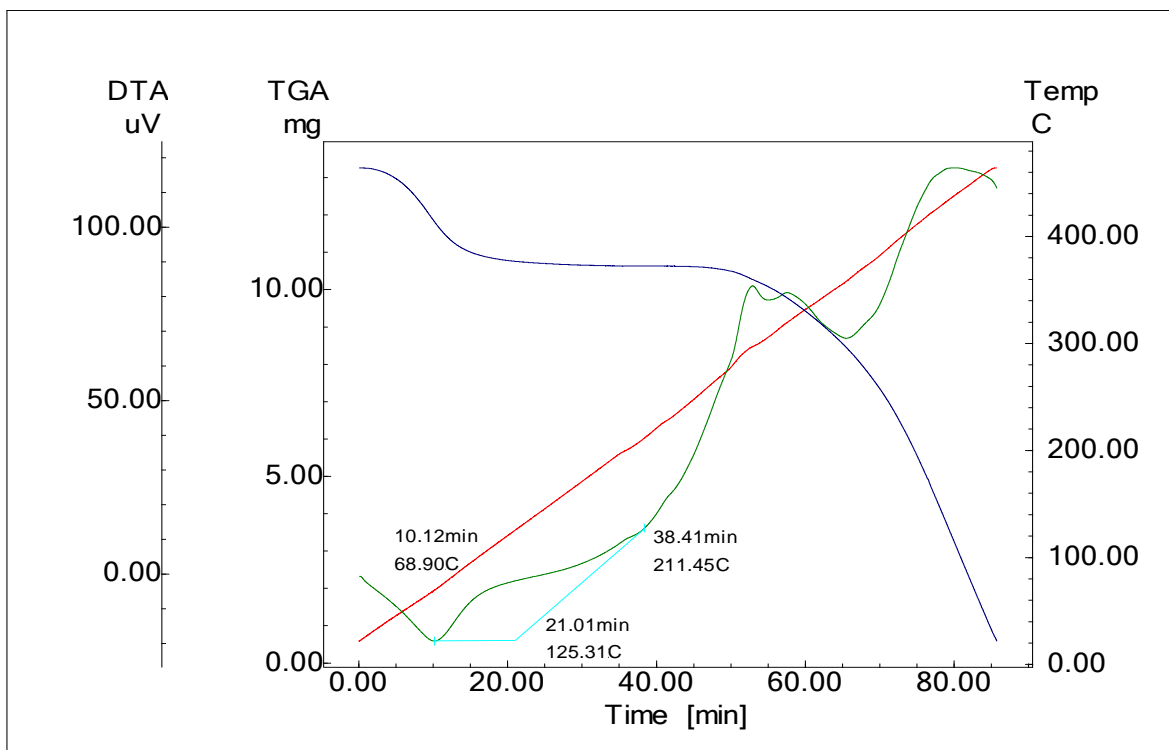


Fig. A1.16 DTA thermogram for sample no. 16

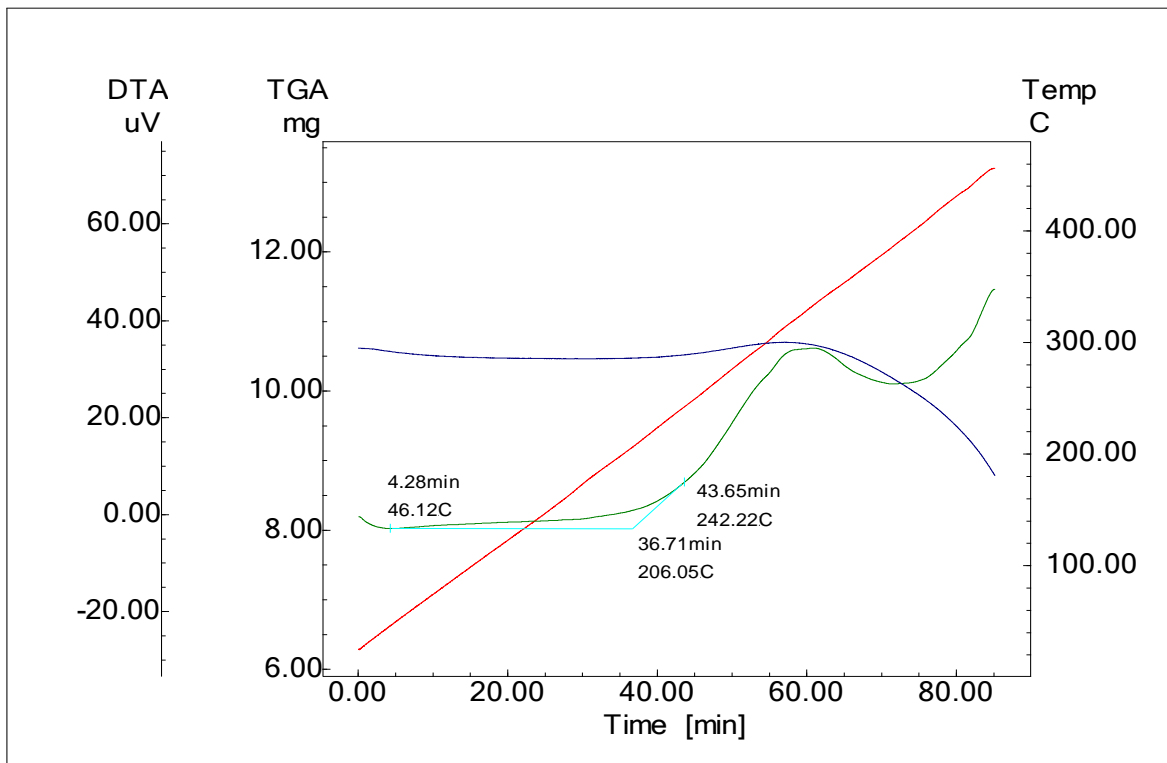


Fig. A1.17 DTA thermogram for sample no. 17

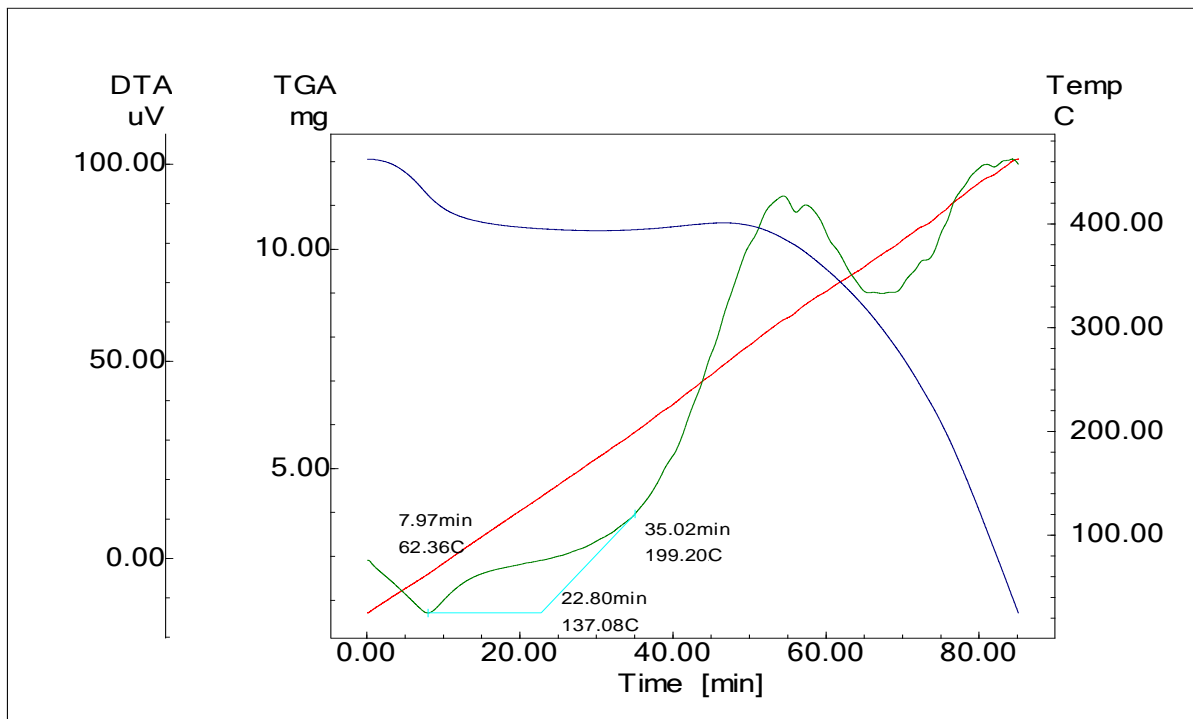


Fig. A1.18 DTA thermogram for sample no. 18

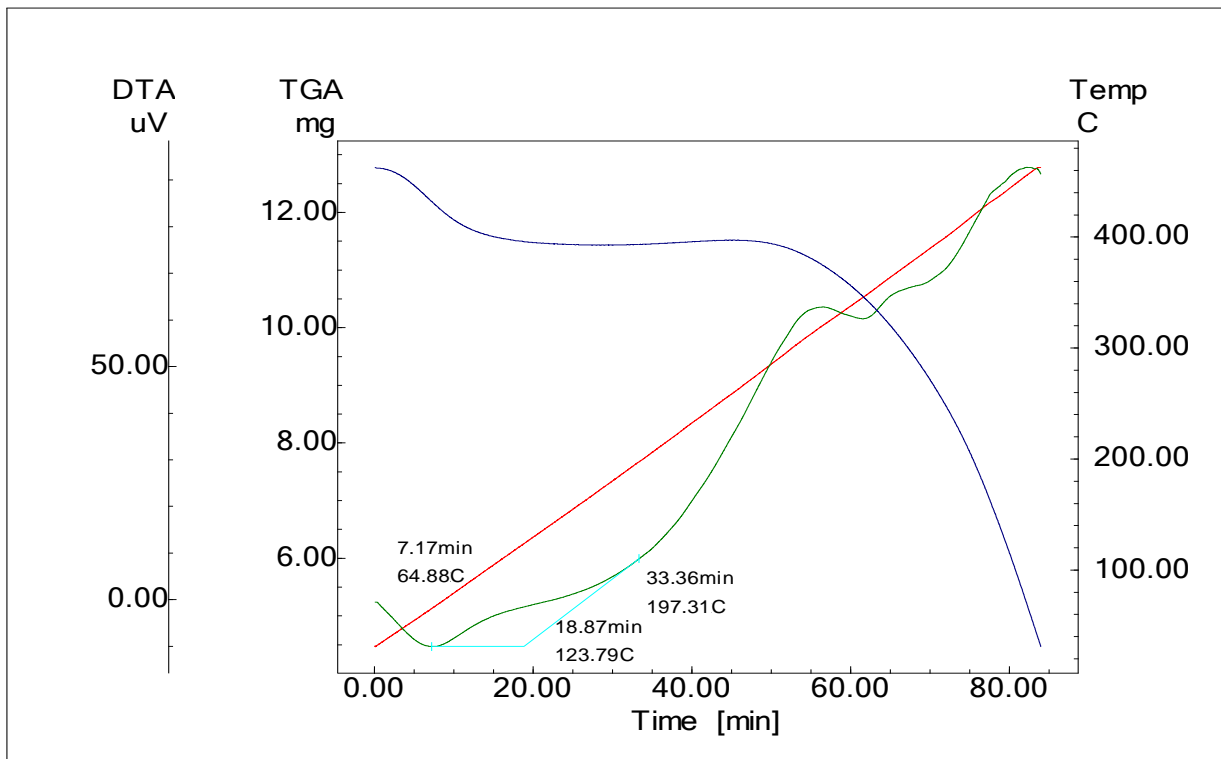


Fig. A1.19 DTA thermogram for sample no. 19

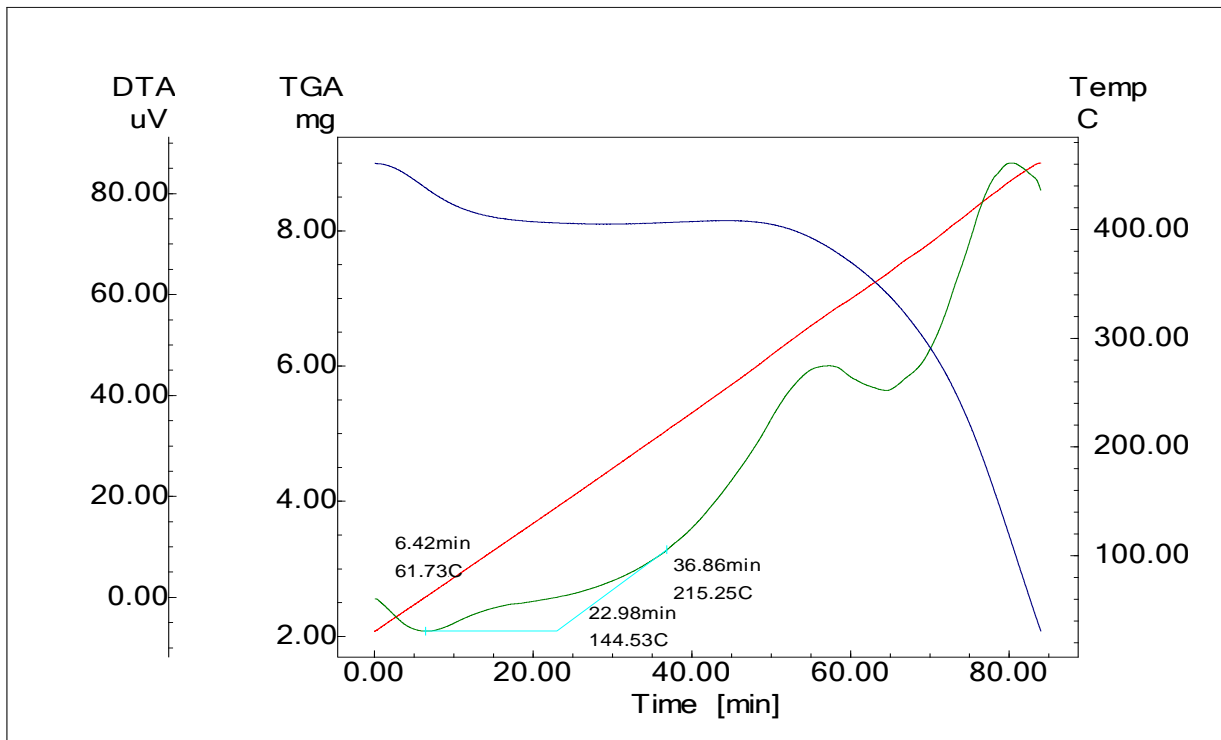


Fig. A1.20 DTA thermogram for sample no. 20

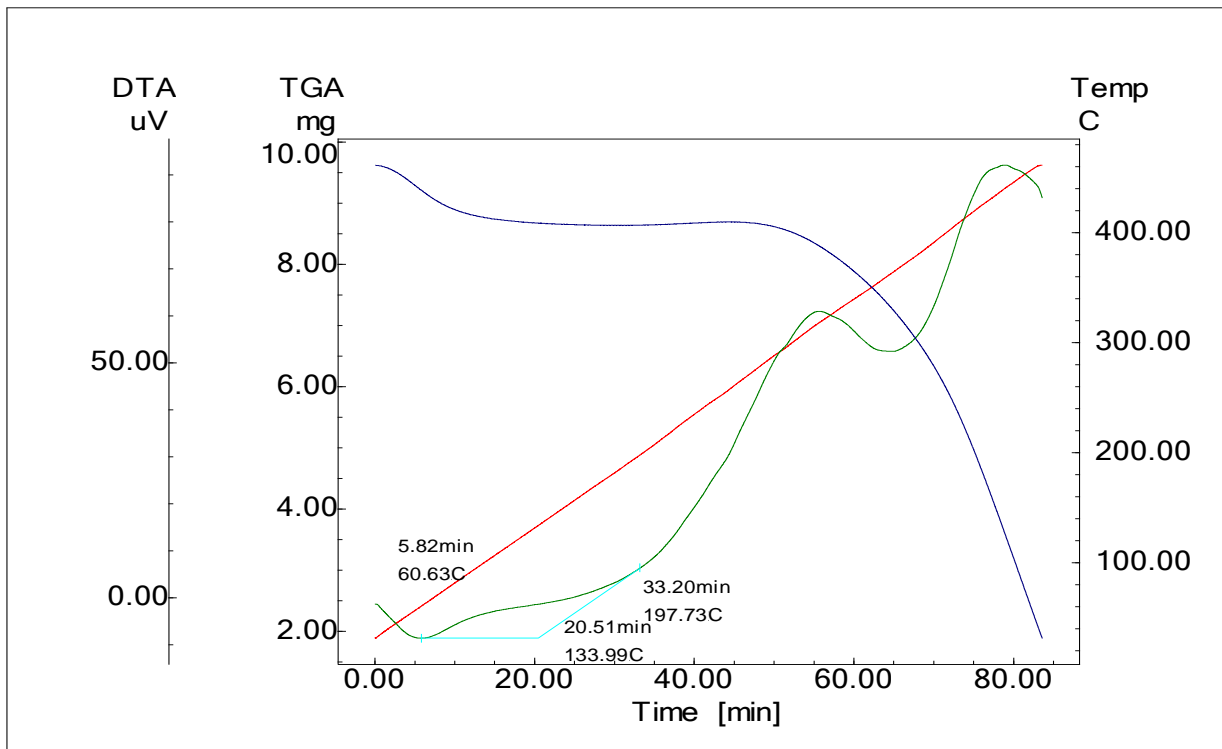


Fig. A1.21 DTA thermogram for sample no. 21

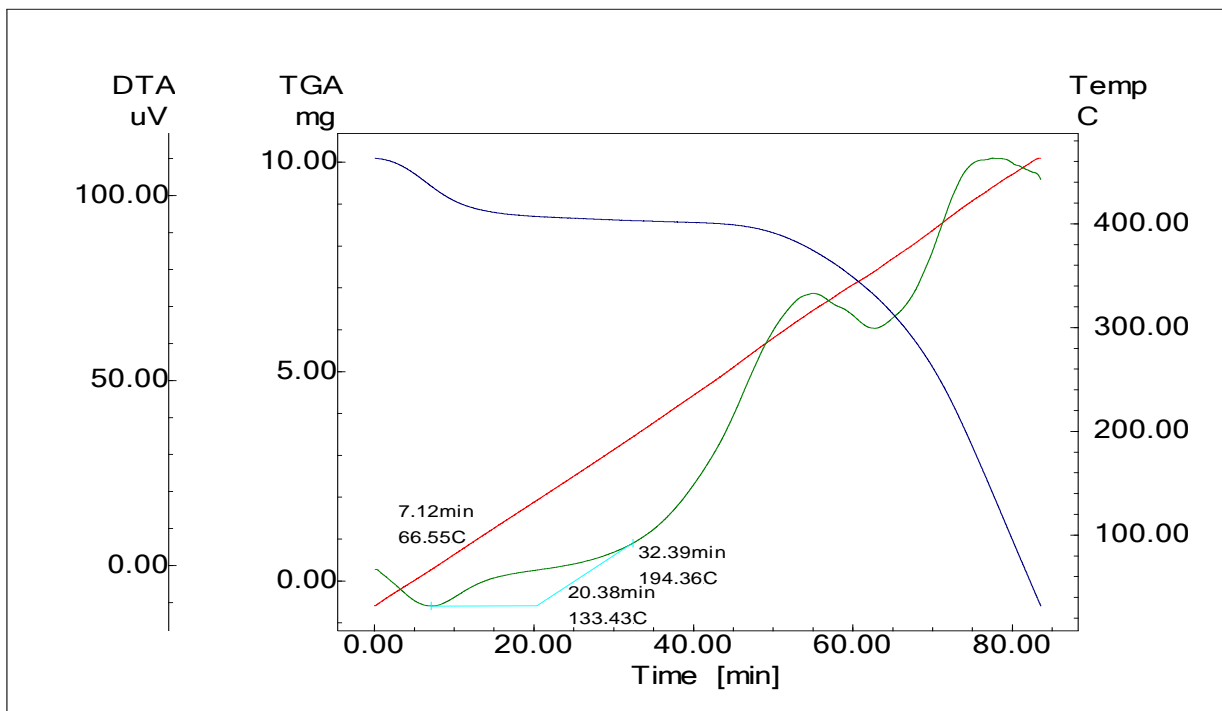


Fig. A1.22 DTA thermogram for sample no. 22



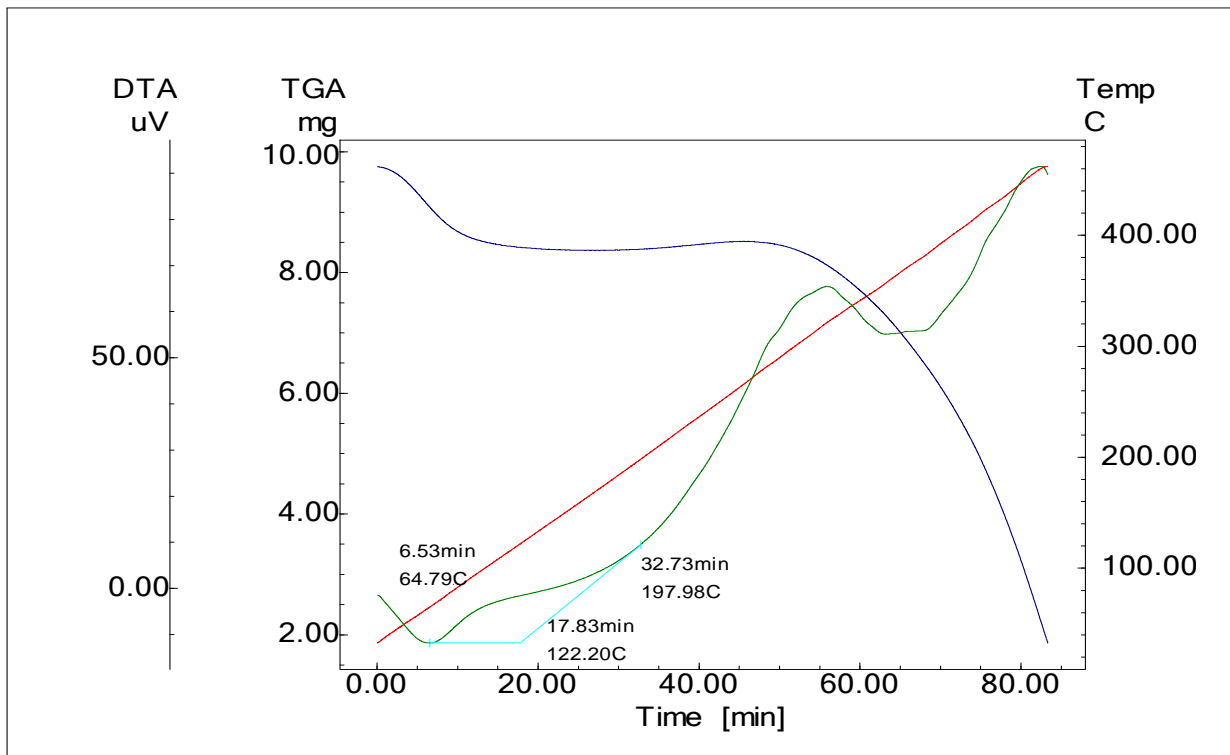


Fig. A1.23 DTA thermogram for sample no. 23

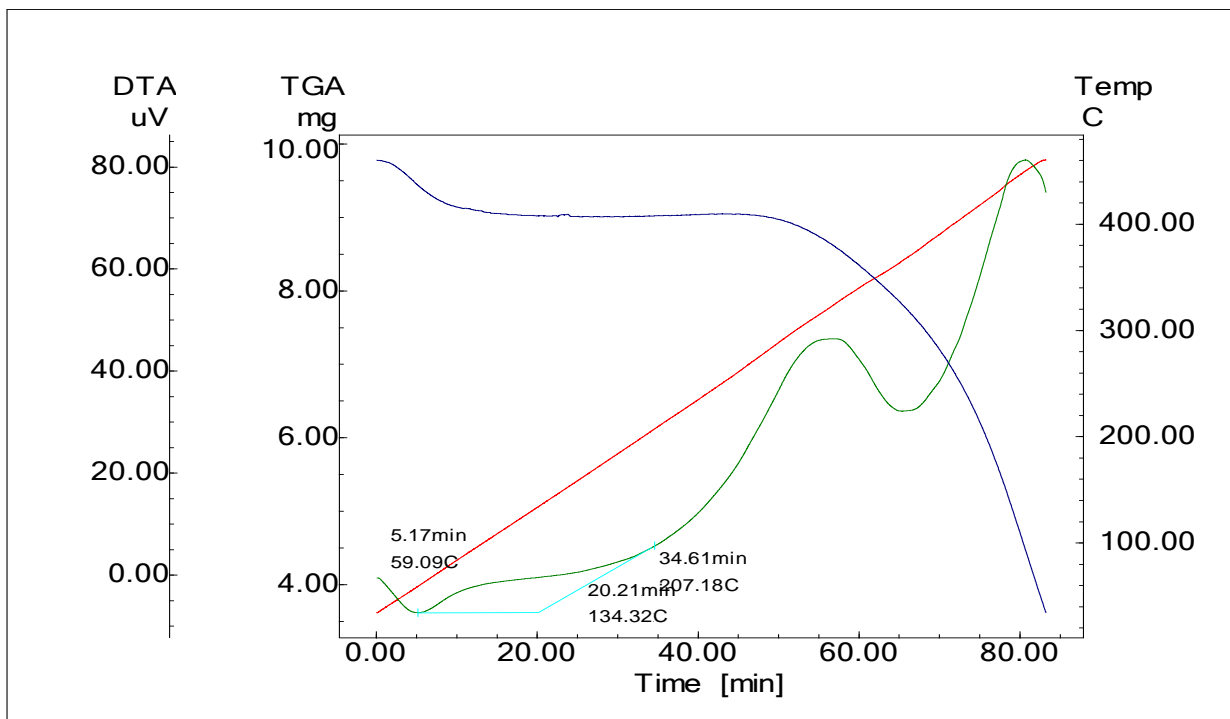


Fig. A1.24 DTA thermogram for sample no. 24

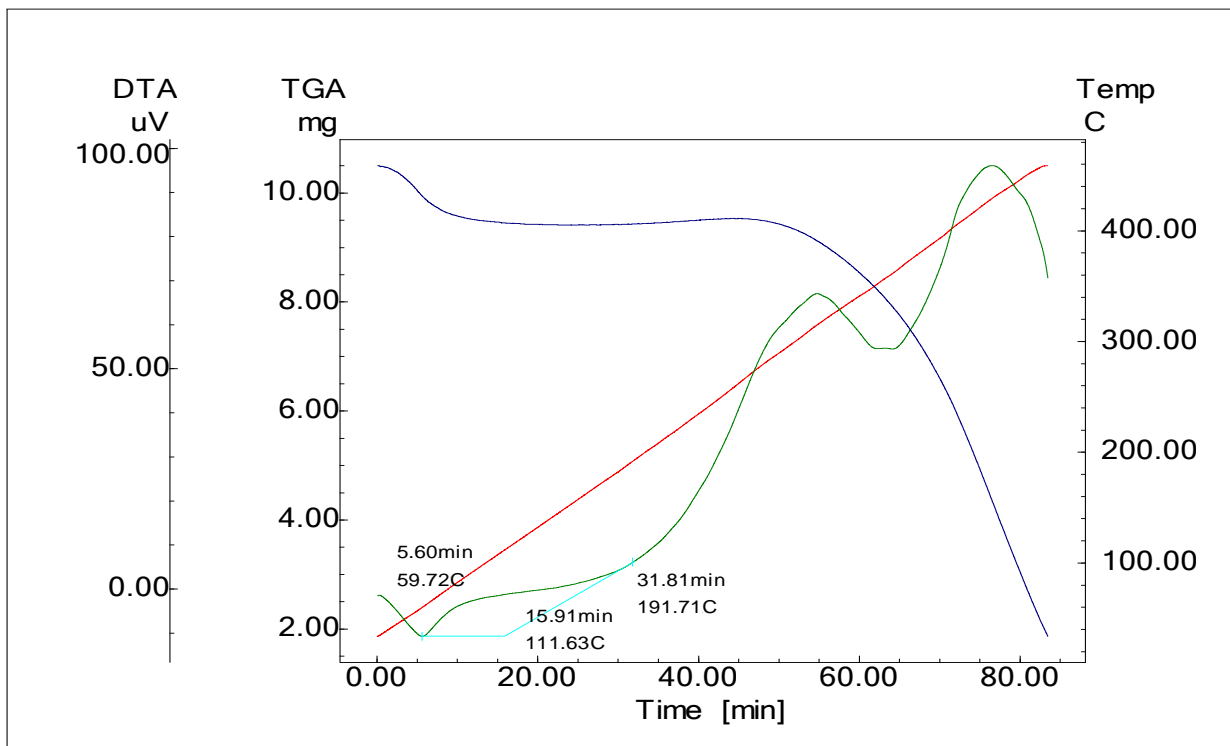


Fig. A1.25 DTA thermogram for sample no. 25

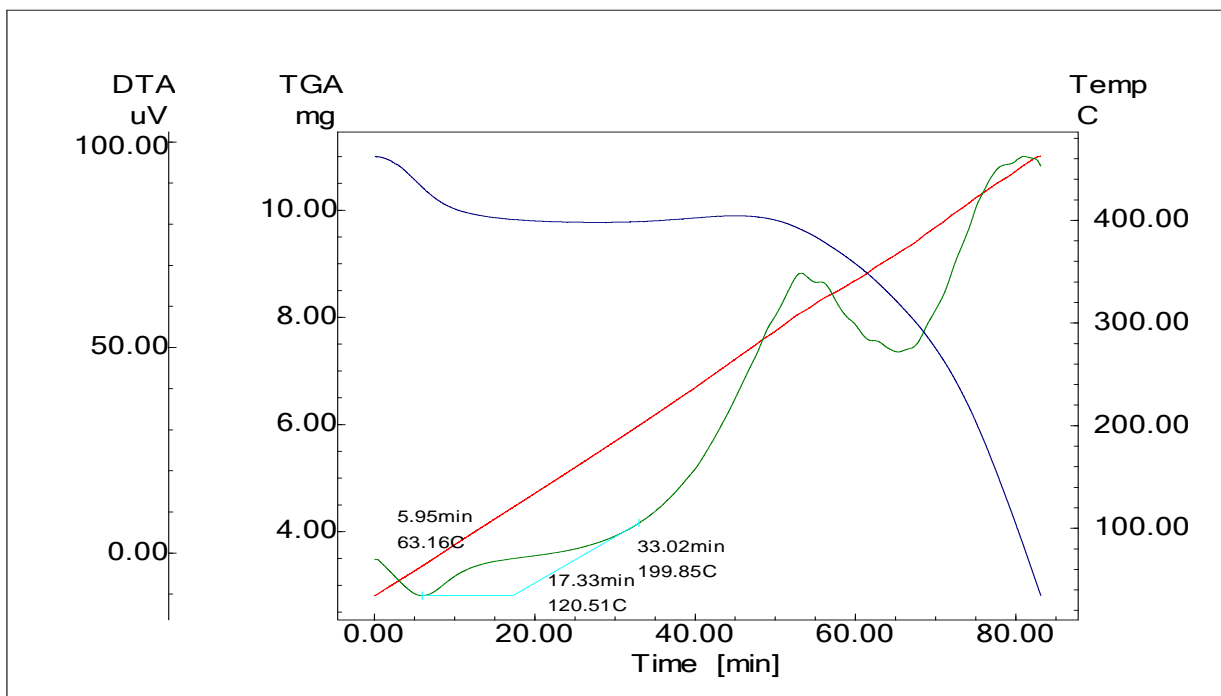


Fig. A1.26 DTA thermogram for sample no. 26

# **APPENDIX – 2**

## **Correlation Plots**

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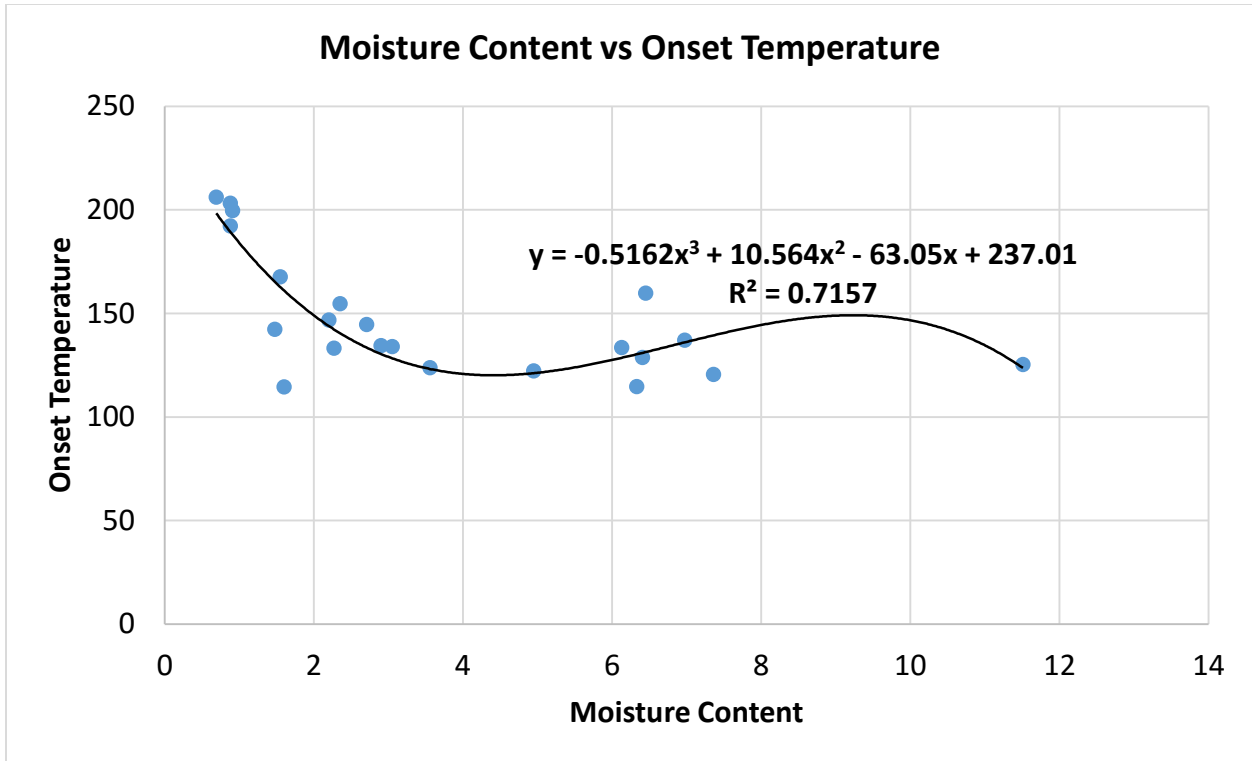


Fig. A2.1 Moisture Content vs Onset Temperature Plot

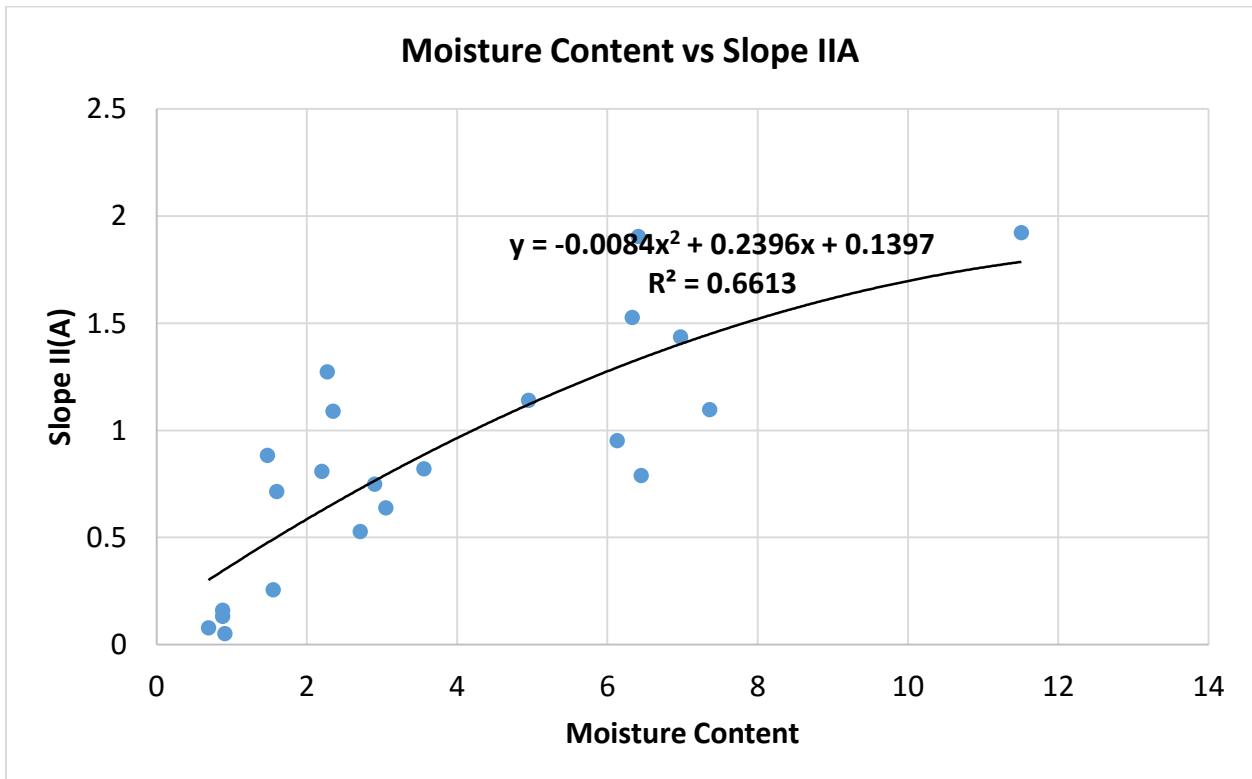


Fig. A2.2 Moisture Content vs Slope IIA Plot

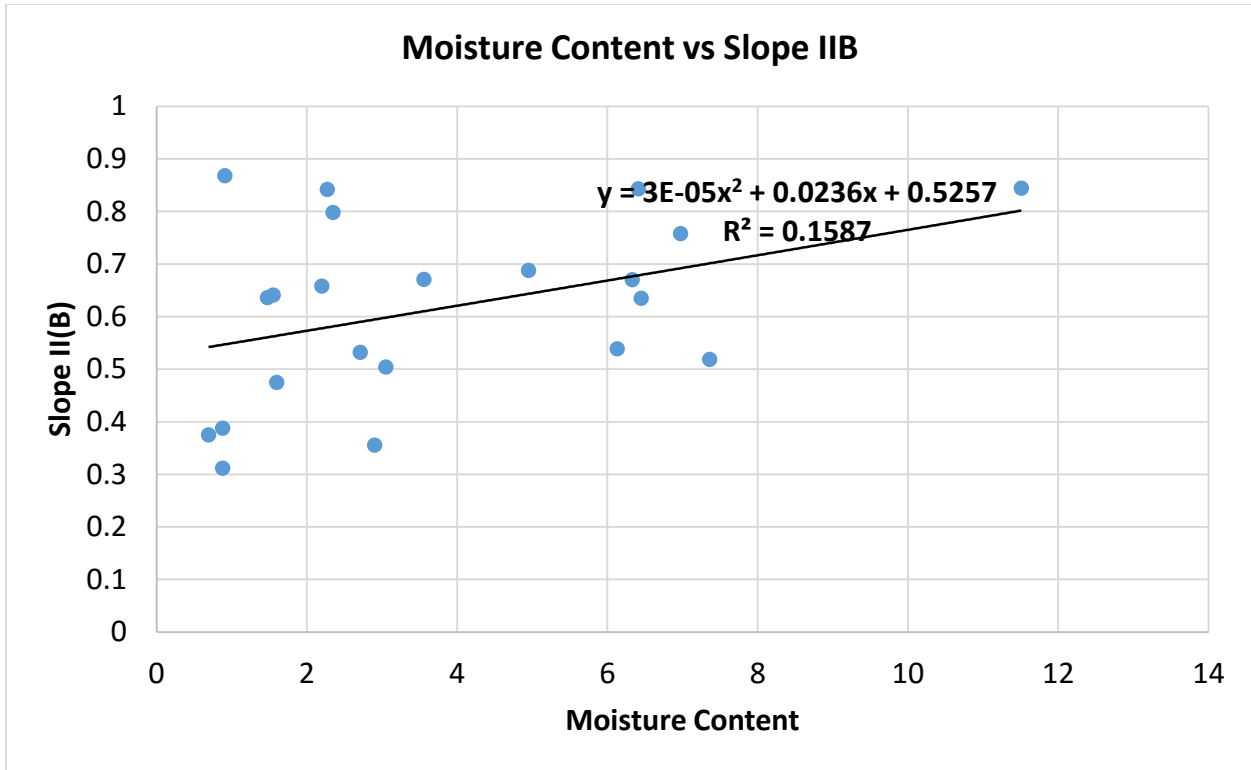


Fig. A2.3 Moisture Content vs Slope IIB Plot

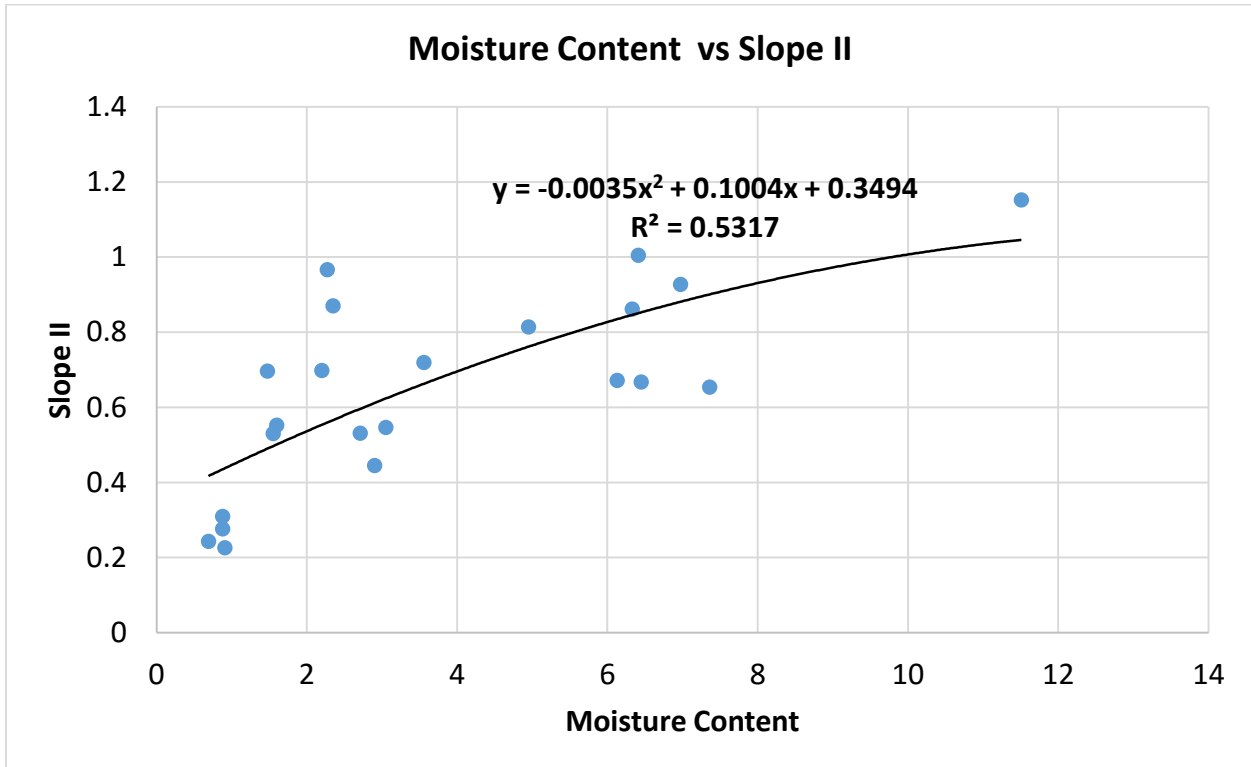


Fig. A2.4 Moisture Content vs Slope II Plot

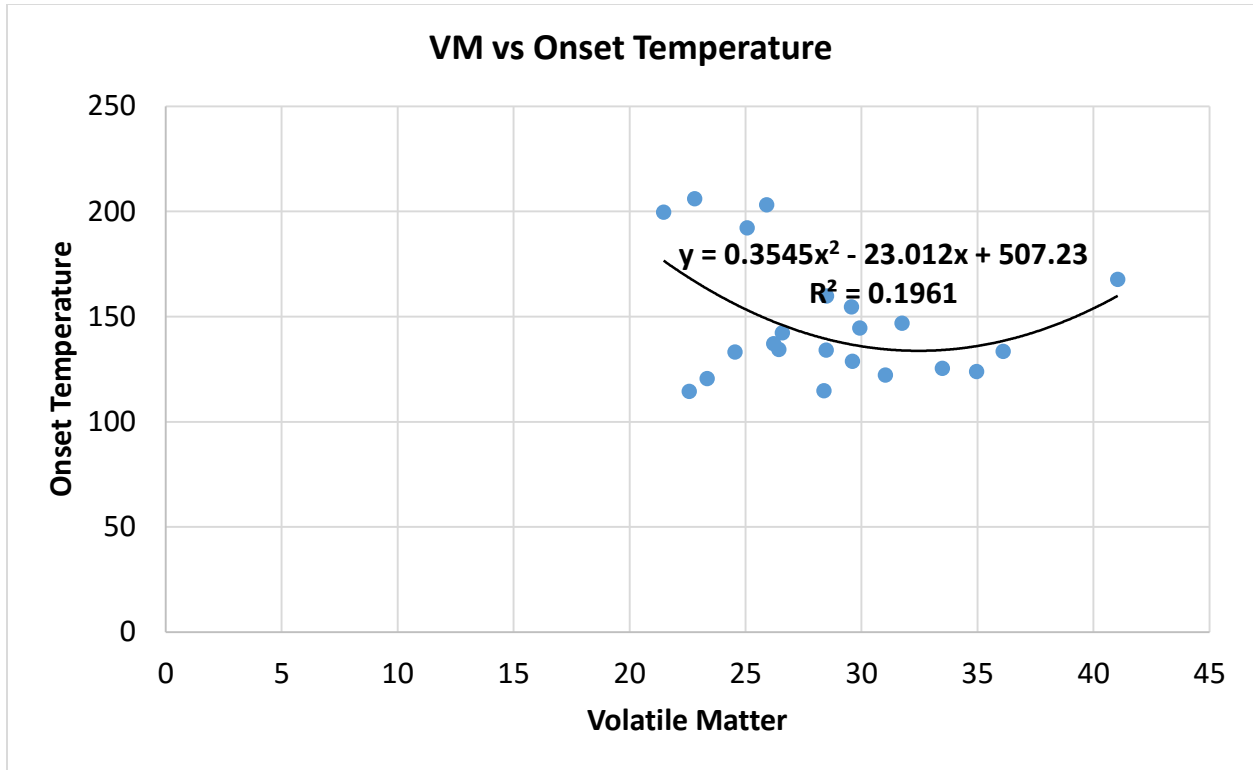


Fig. A2.5 VM vs Onset Temperature Plot

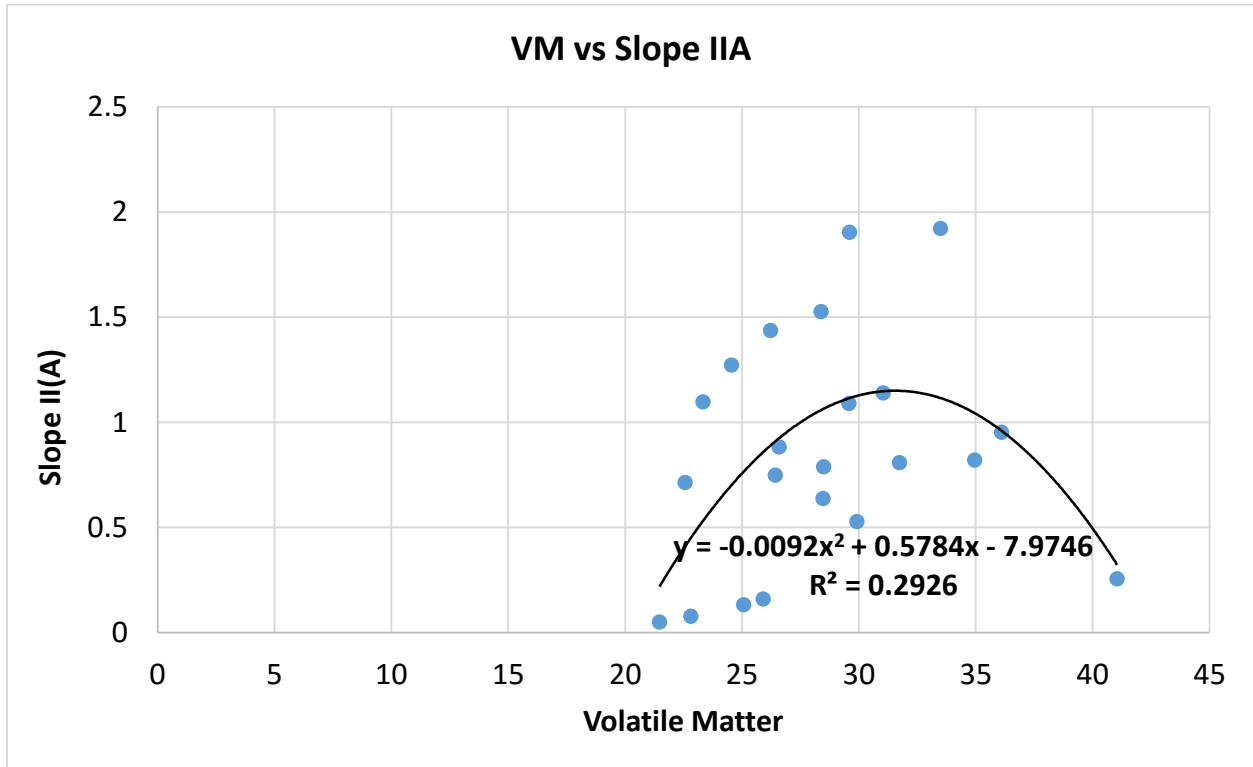


Fig. A2.6 VM vs Slope IIA Plot

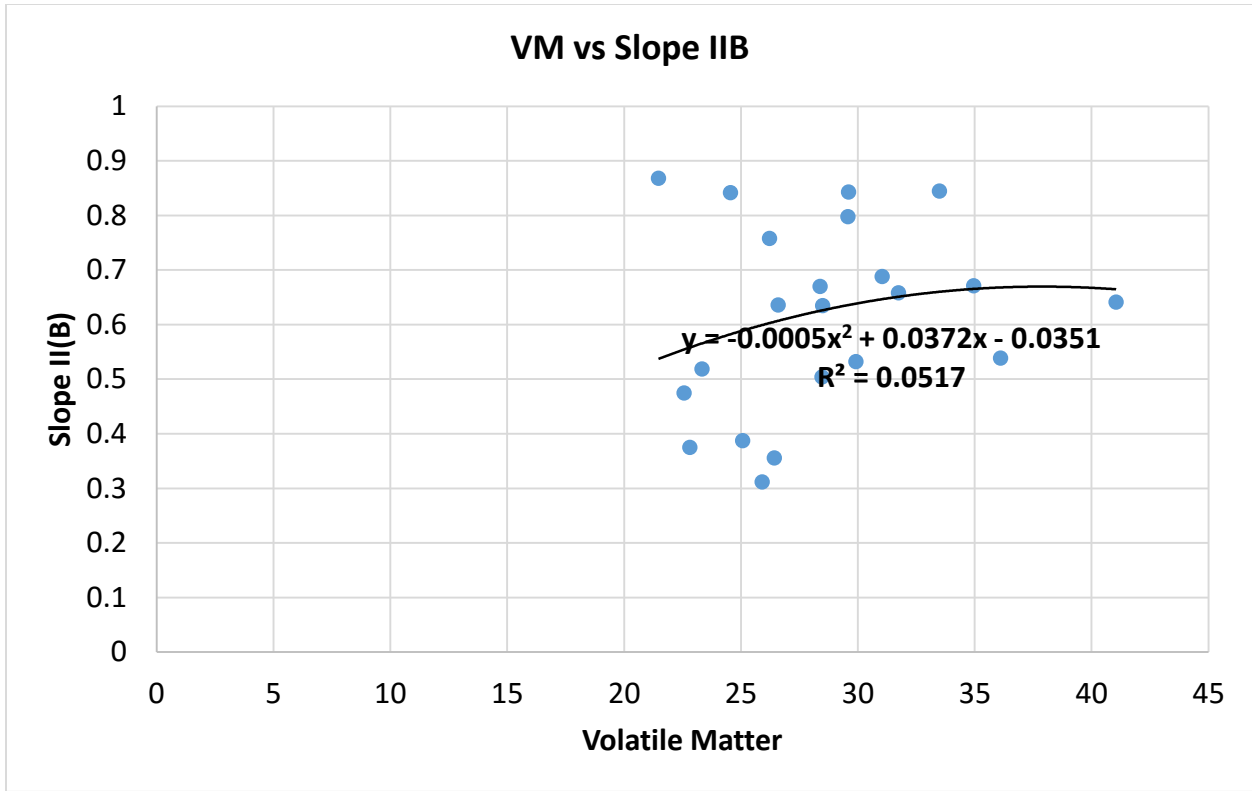


Fig. A2.7 VM vs Slope IIB Plot

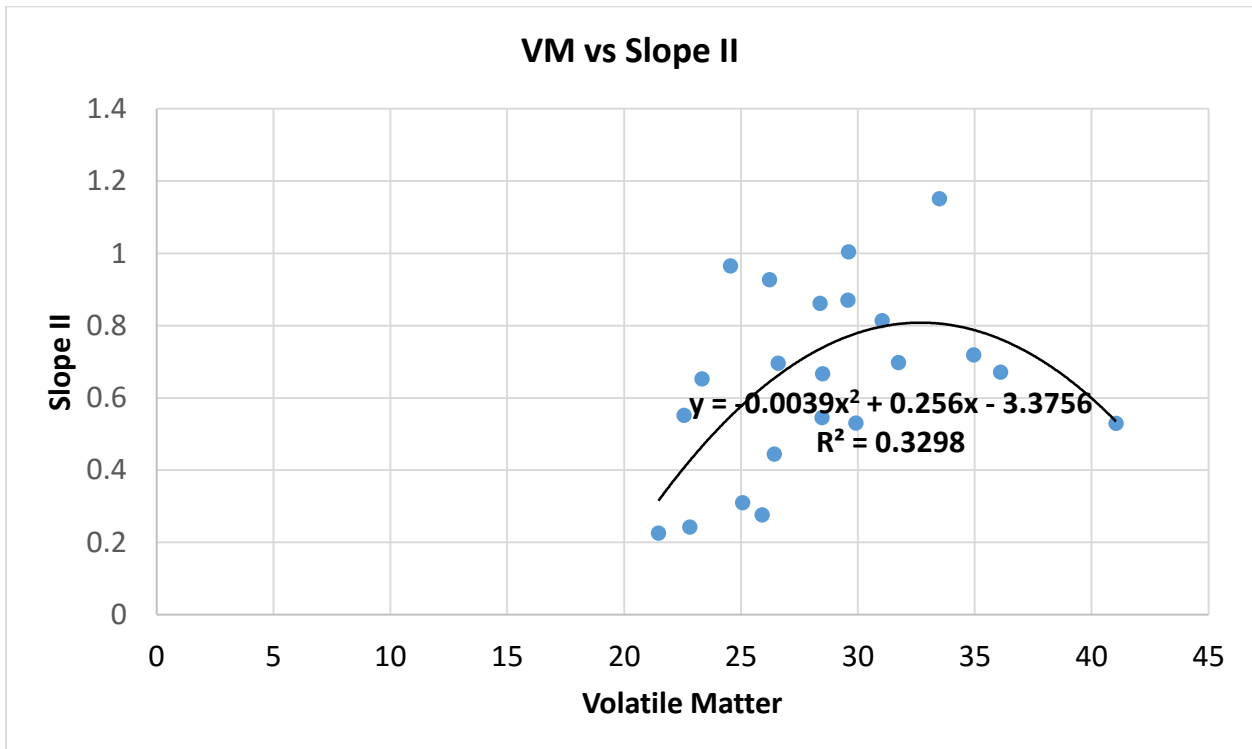


Fig. A2.8 VM vs Slope II Plot

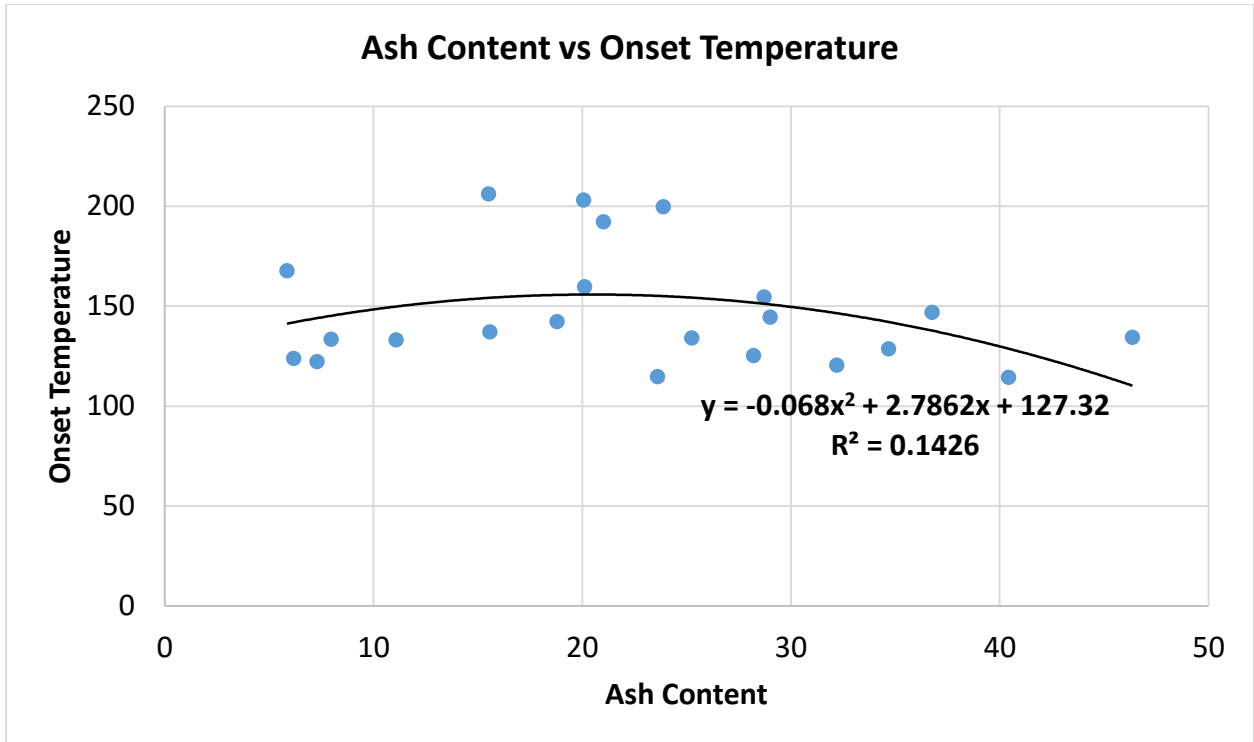


Fig. A2.9 Ash Content vs Onset Temperature Plot

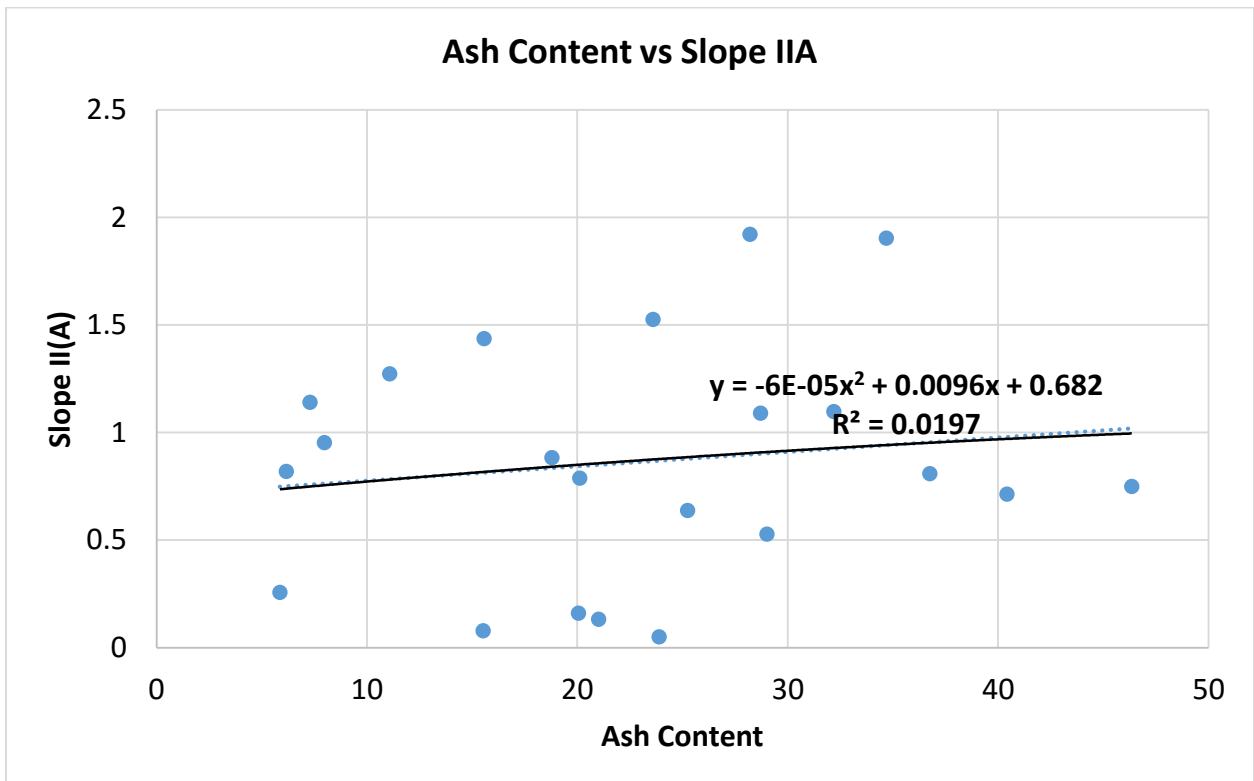


Fig. A2.10 Ash Content vs Slope IIA Plot



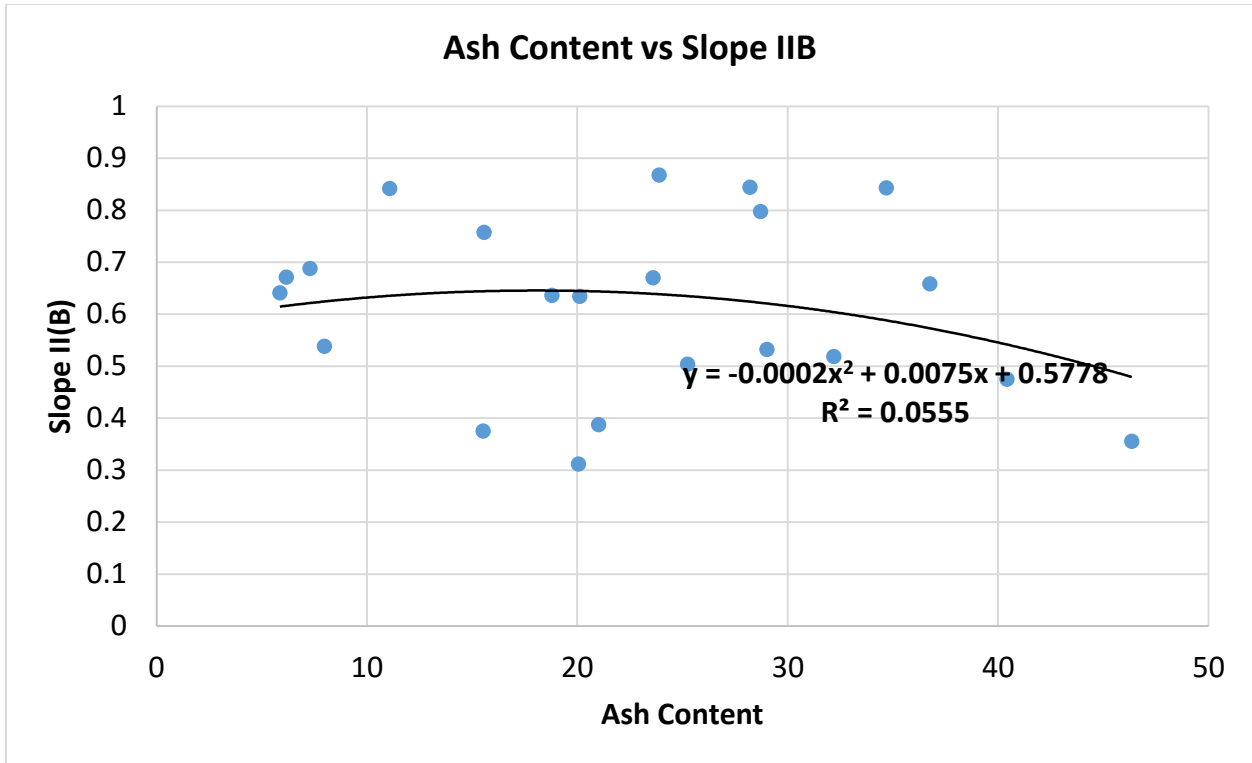


Fig. A2.11 Ash Content vs Slope IIB Plot

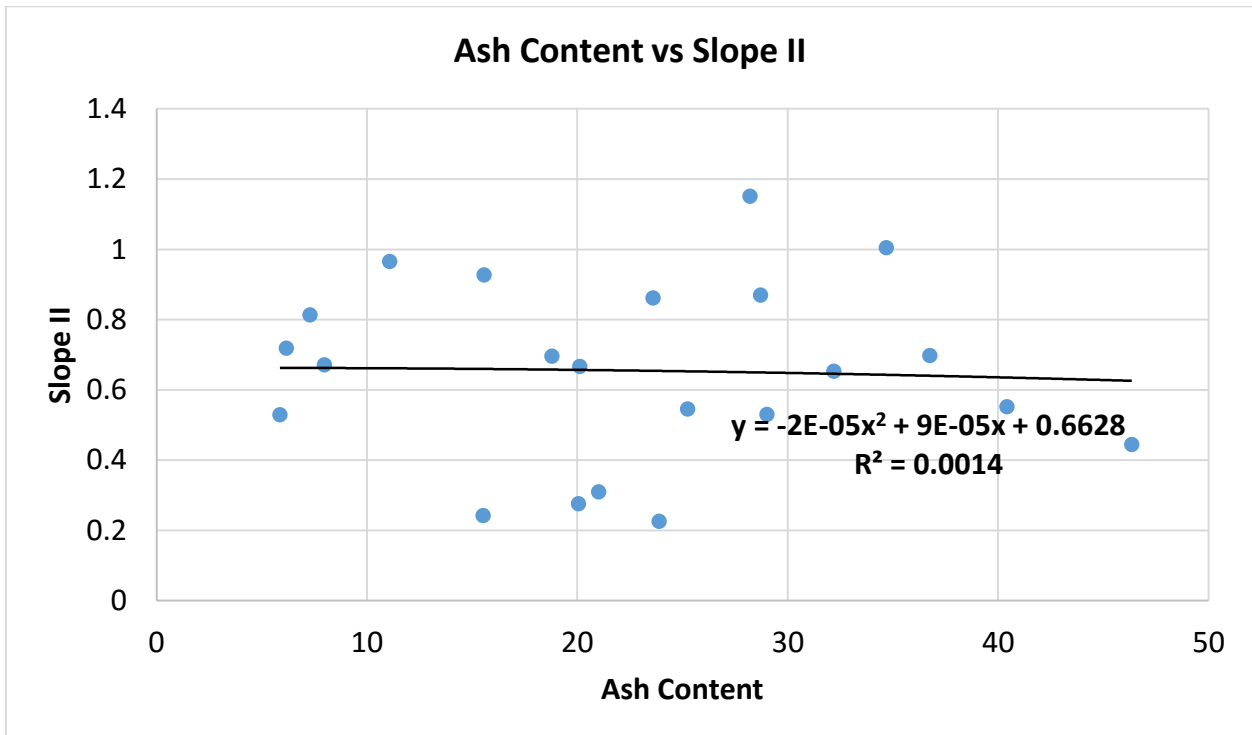


Fig. A2.12 Ash Content vs Slope II Plot

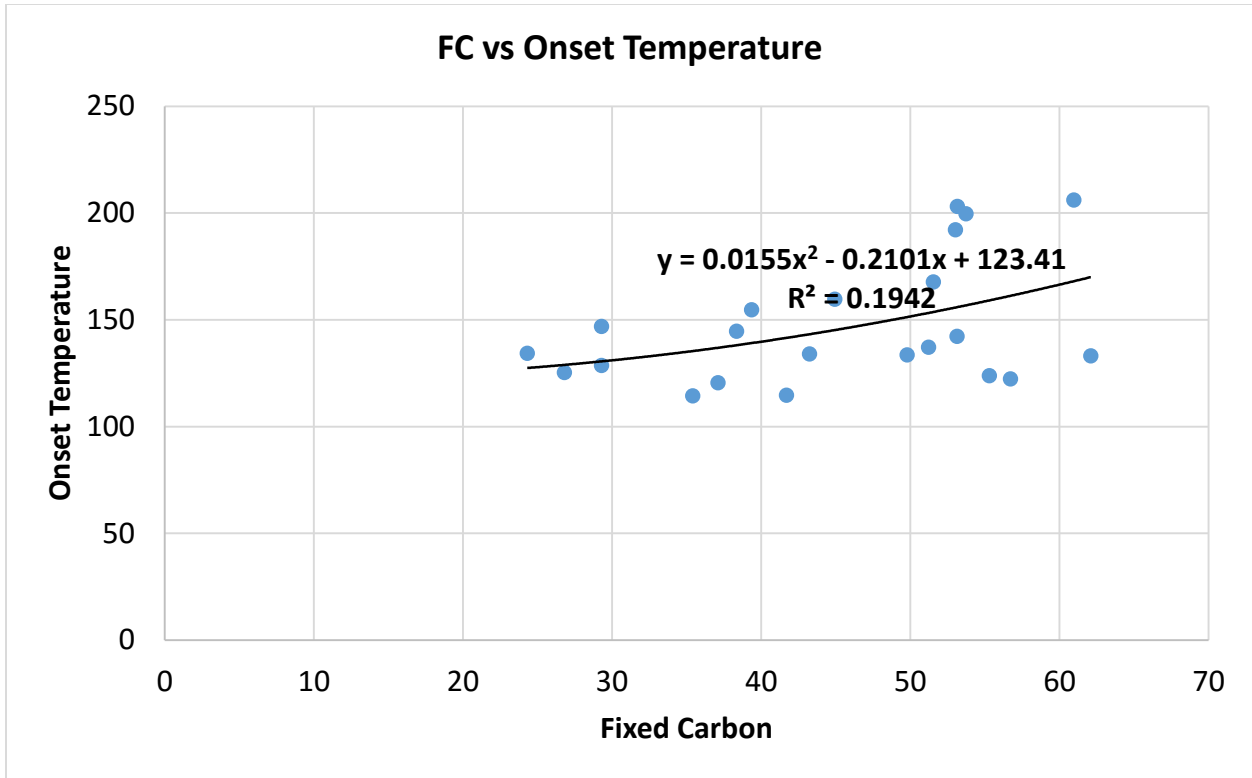


Fig. A2.13 FC vs Onset Temperature Plot

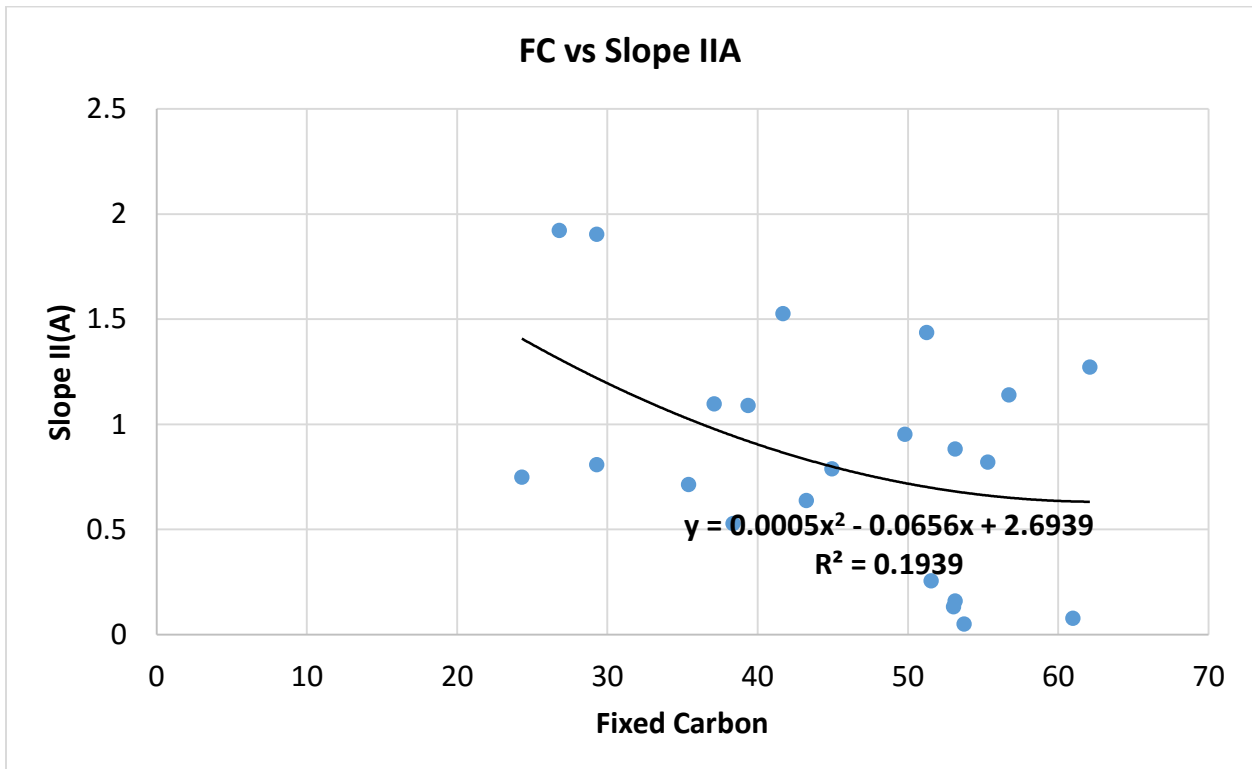


Fig. A2.14 FC vs Slope IIA Plot

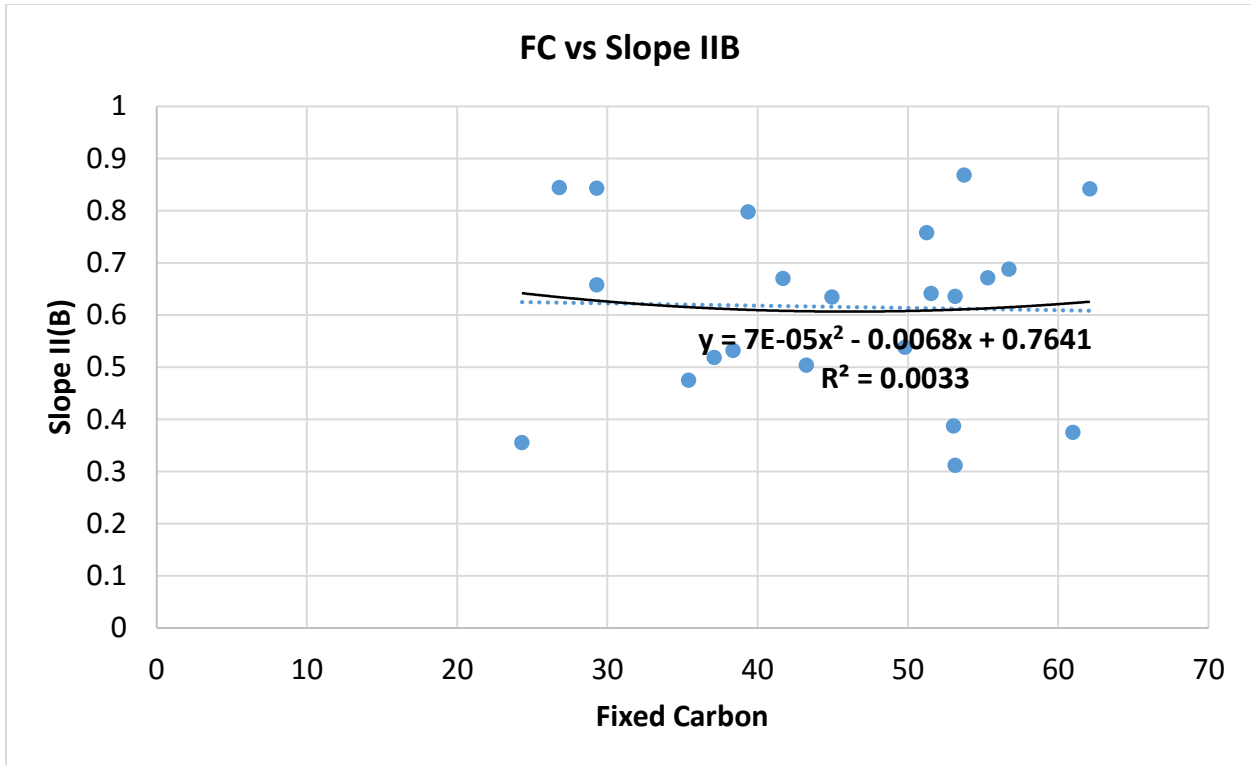


Fig. A2.15 FC vs Slope IIB Plot

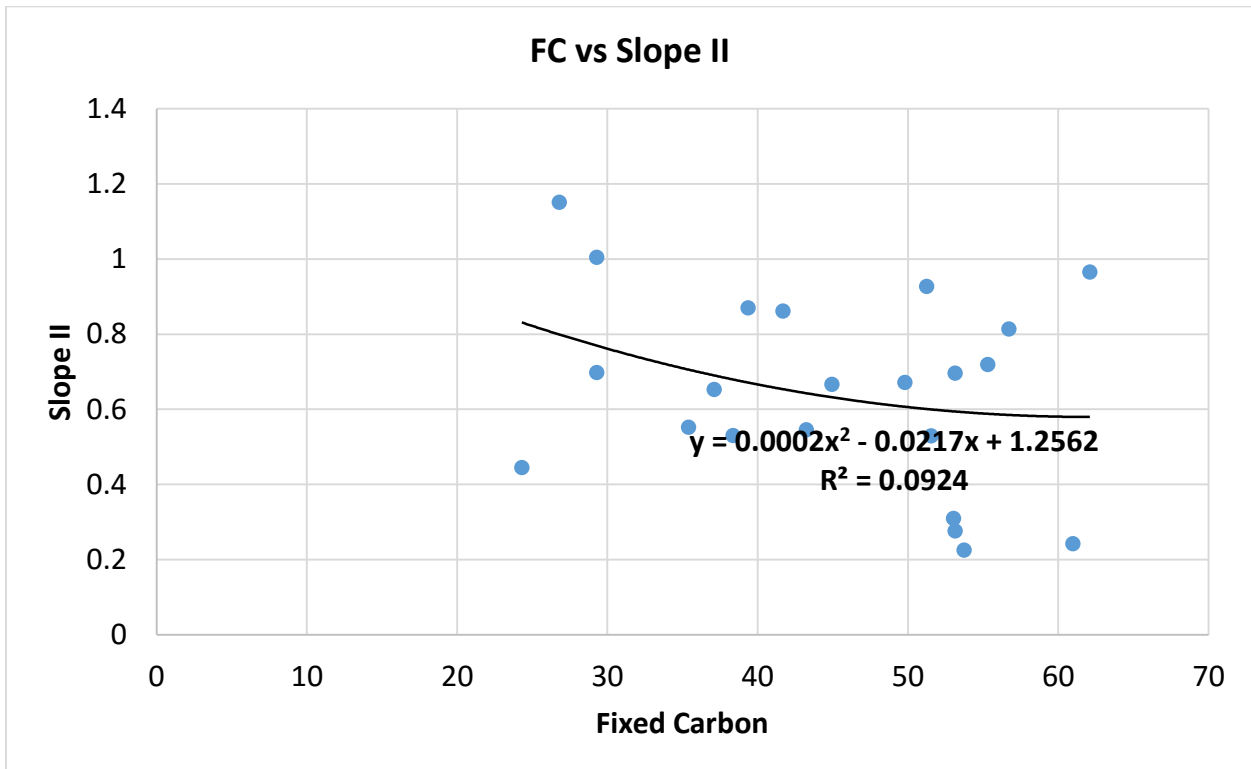


Fig. A2.16 FC vs Slope II Plot