Chapter 5
TGA Analysis & Thermal Conductivity

5.1 Introduction

The thermal methods usually employed are differential scanning calorimetry (DSC), differential thermal analysis (DTA), Thermogravimetric analysis (TGA) and derivatographic analysis. For comparative study of thermal behavior of related polymer or simple molecules, each molecule is analyzed by any one or more of these methods of analysis under identical experimental condition. For example TGA is carried out in air and in oxygen free nitrogen[1-3]. It is carried out at different heating rates. It may be method that the result of thermal analysis of a given sample by a given method depends on various aspects. The amount and particle size of the material being examined, influence the nature of the thermogram[4]. The speed of the recorder noting the change in weight and the shape of the sample container also influence the thermogravimetric results. The rate of heating the sample and the ambient atmosphere during analysis are very important factors to be controlled during thermal analysis. The information furnished by TGA and DTA by DGA are to some extent complimentary. From the results of DTA and TGA, it is possible it note the temperature up to which the material does not loss weight. It is also possible to know the temperature at which material starts decomposing[5-7]. It is possible to know whether the decomposition occurs in one or more stages.
Besides the quantitative information derived on mere inspection of the thermogram, other information about the order of the degradation reaction can be obtained by the analysis of the thermal data furnished by either DTA or TGA.

Thermogravimetry deals with the change in the mass of a substance, continuously monitored as a function of temperature or time, when it is heated or cooled at a predetermined rate. It provides information on the thermal stability of the sample at different temperatures and pressures of the environmental gases.

The apparatus used for obtaining TG (thermograms) curves is referred to as a thermobalance. It consists of a continuously recording balance, furnace, temperature, programmer and a recorder. The components of the thermobalance are discussed below in brief[8-11].

I) There are two types of balances:

(a) deflection / extension type and (b) null balance

II) Sample holders: The type of the sample holder decides the shape and nature of the TG curves. The requirements of the sample holders are:

1. The capacity should be large enough to avoid loss due to spurting or creeping.
2. Heat transfer between the furnace and the sample holder should be efficient. This is better with metal holders. There should also be a virtual absence of temperature gradients within the sample.
3. Since most of the reactions of interest are of gas-solid type, the sample holder should permit easy outflow of the gases from the sample and also facilitates free diffusion of the gases into the sample when the reaction involves uptake of the gas.
Here a cylindrical, platinum crucible which has sufficient height is used and three different types of sample holders have been recommended, viz. gauze crucible, polyplate sample holder and labyrinth crucible.

Thermal Gravimetric Analyzer measures the change in the mass of a sample as the sample is heated, cooled or held at a constant (isothermal) temperature[12-14]. The measurement will also depend on the type of atmosphere whether it is inert or oxidative. Chemical changes occur in an oxidative atmosphere providing very useful information regarding characterization of the sample.

The thermal analysis of a specimen involves studying the evolution of several physical properties as a function of the temperature. When the material is subjected to heating or cooling, its chemical composition and crystal structure undergo such changes as reaction, oxidation, decomposition, fusion, expansion, contraction, crystallization, or phase transition. All these changes can be detected using differential thermal analysis.

Differential thermal analysis (DTA) measures the temperature, the direction and the magnitude of thermal transitions induced by heating or cooling a material in a controlled way. DTA measures these properties by comparing the temperature of the sample and that of a reference material, which is inert under similar conditions. This temperature difference is measured as a function of time or temperature under a controlled atmosphere and it provides useful information about the transition temperature but also about its thermodynamics and kinetics. Thermogravimetric analysis (TGA) determines the weight gain or loss of a phase due to gas absorption or release as a function of temperature under a controlled atmosphere. This technique provides information
about the purity of the sample, as well as its water, carbonate and organic content. It is also useful for studying decomposition reactions.

5.2 Thermo gravimetric analysis

The thermal behaviour of the ligand samples described in chapter 2 has been studied by TGA. The loss in weight due to pyrolysis of the material normally the shape of the TG curve depends upon the nature of the in situ degradation reaction of the sample. The analysis of these data by Broido method is often carried out with a view to estimate kinetic parameters like energy of activation of the degradation reaction.

The advantages of TGA are enumerated here[16].

(I) A relatively small set of data is to be treated.
(II) Continuous recording of weight loss as a function of temperature ensures Equal weightage to examination over the whole range of study.
(III) As a single sample is analysed over the whole range of temperature, the variation in the value of the kinetic parameters, if any, will be indicated.

For the estimation of kinetic parameters, several methods are employed. All involve two assumptions. It is assumed that the difference in the thermal and diffusion processes is negligible. It is also assumed that Arrhenius relation is valid over the whole range of temperature. Since small materials are employed in TG analysis the barriers between the thermal and diffusion processes are quite negligible. Hence it is reasonable to ignore it. Besides it is reasonable to assume validity of Arrhenius relation.
Various methods are proposed to analyse the TG data depending upon the nature of experimentation. As the present study deals with azo polymers. Hence attempt was not made for Application at these methods.

5.3 Experimental (TGA)

The thermogravimetric analyses (TGA) of sample have been carried out by using “PERKIN ELMER PYRIS 1 TGA” in a slow stream of air. The boat prepared from platinum foil would hold the sample for analysis. It is properly washed and dried. It was suspended on the quartz rod in the TG balance. The powdered sample (about 5 mg) was placed in the boat. The sample in the boat is covered by a quartz tube in which the flow of air was maintained. The weight of sample was noted on TGA balance. The whole assembly was brought down in the furnace. It was ascertained that the boat was hanging on quartz rod. The experiment was started by the heating the system at a constant rate of 10°C/min. Simultaneously change in the weight was recorded automatically with time (temperature). This will reveal percentage weight loss of material as a function of the time and also of temperature. The experiment was stopped at about 650 °C, when there was no further decrease in weight[17].

The thermograms were analyzed to obtain information about percentage weight loss at different temperatures[18]. The results of these analyses are presented in Table 3.8 and 3.9. Selected thermograms are shown in Figures 3.14 to 3.16.

Thermal Gravimetric Analyzer measures the change in the mass of a sample as the sample is heated, cooled or held at a constant (isothermal) temperature. The measurement will also depend on the
type of atmosphere whether it is inert or oxidative. Chemical changes occur in an oxidative atmosphere providing very useful information regarding characterization of the sample. The TGA Make: Perkin Elmer, Model: TGA-7 is based on a rugged microbalance which is highly sensitive measurement of weight changes as small as few micrograms. This has superb atmosphere control including computer controlled gas switching. The measurements, high temperature studies, thermal stability and compositional analysis. The equipment used in the present study is shown in Fig.5.1 The sensitivity of this equipment is 0.1\(\mu\)gm (0.0001mg) with temperature range from ambient to 1000\(^\circ\)C.

![Fig. 5.1 Photograph of thermal gravimetric analysis](image)

**Make Model**

Perkin Elmer Pyris-1 DSC, Pyris-1 TGA, DTA-7
Small Description:
**Thermal Analyzer (DSC, TGA, DTA),** PerkinElmer Pyris1 DSC, Pyris1 TGA, DTA 7, Temp.
Range: TGA: Room Temperature to 1000ºC; DSC: -100ºC to 600ºC and DTA: Room Temperature to 1200ºC. All types of thermal analysis studies /testing.

Specifications:
(TGA)
- Temperature Range: Ambient to 1000º C
- Sensitivity: 0.1mg (0.0001mg)
- Atmosphere: N₂, or Air

(DSC)
- Temperature range: -100 ºC to 600 ºC
- Temperature Accuracy: ± 0.2 ºC
- Heating Rate: 0.1 to 100 ºC / min

(DTA)
- Temperature range: -Ambient to 1200 ºC
- Heating Rate: 0.1 to 100 ºC / min
- Atmosphere: Air or Nitrogen

Applications:
Thermal Analysis (DSC,TGA,DTA)
Thermal Gravimetric Analyzer measures the change in mass of a sample as a function of time or temperature in inert or oxidative atmosphere.
- Chemical changes occurring in an oxidative atmosphere provide useful information regarding characterization of the sample.
- TGA-1 is based on a rugged microbalance which is sensitive to measure even a few micrograms of weight loss.
• It employs computer controlled gas switching to regulate furnace atmosphere.
• There are preset programs to allow compete separations, Decomposition studies, proximate analysis of coal, auto stepwise analysis of filled polymers, curing characteristics High temperature studies, Thermal stability and compositional analysis are possible with this instrument.
• Used for Melting, Crystallization, Glass Transitions Temperature, Polymorphism, Kinetic Studies, Curing Reaction.
• Endothermic and Exothermic effects
• Isothermal Cure kinetics studies.
• Characterization of polymorphism of pharmaceuticals.
• Characterization of pharmaceuticals formulations.
• Characterization of multicomponent materials by TGA.
• Thermal decomposition study
• Widely used in polymer, pharmaceuticals, cosmetics industry etc.

5.4 Results and discussion

The brief account of the thermal behavior of samples in air is given below:

(I) Each crystals degradation in two steps.

(II) The degradation of all the crystals start in the temperature Range of 200 to 350°C depending upon the natures of crystals.

(III) The wt. loss amount in this first stage is in between 4.5 to 7 % this may be due to water molecules associated in to the crystals.
(IV) The second stage of decomposition of all crystals is rapid with the loss of mass about 50%. This is due to “in situ” formation of metal oxide during degradation. Which accelerate the rapid degradation of crystals.

(V) The last stage of digression cause a mass loss of about 80%. This is due to loss molecular fragments of polymers.
Figure – 5.2 TGA Thermogram of Schiffbase
Figure 5.3: TGA Thermogram of Ni(II) Crystal

Perkin Elmer Pyris 1 TGA

\[ \text{Diel} \gamma = 86.126 \% \]
Figure – 5.4 TGA Thermogram of Cu(II) Crystal
Table 5.1

Thermogravimetric analysis of Crystals and Schiffbase

<table>
<thead>
<tr>
<th>Crystal</th>
<th>% wt loss at temperature</th>
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</thead>
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<tr>
<td></td>
<td>250 °C</td>
</tr>
<tr>
<td>(Cu^{2+}) Crystal</td>
<td>6.2</td>
</tr>
<tr>
<td>(Ni^{2+}) Crystal</td>
<td>2.8</td>
</tr>
<tr>
<td>Schiffbase</td>
<td>6.6</td>
</tr>
</tbody>
</table>
5.5 Thermal Conductivity

In physics, thermal conductivity, \( k \), is the property of a material reflecting its ability to conduct heat. It appears primarily in Fourier's Law for heat conduction. Thermal conductivity is measured in watts per kelvin-metre \((W\cdot K^{-1}\cdot m^{-1})\), i.e. \( W/(K\cdot m) \). Multiplied by a temperature difference (in kelvins, K) and an area (in square meters, \( m^2 \)), and divided by a thickness (in meters, m), the thermal conductivity predicts the rate of energy loss (in watts, W) through a piece of material. The reciprocal of thermal conductivity is thermal resistivity[18]. There are a number of ways to measure thermal conductivity. Each of these is suitable for a limited range of materials, depending on the thermal properties and the medium temperature[19,20]. There is a distinction between steady-state and transient techniques. In general, steady-state techniques are useful when the temperature of the material does not change with time. This makes the signal analysis straightforward (steady state implies constant signals). The disadvantage is that a well-engineered experimental setup is usually needed. The Divided Bar (various types) is the most common device used for consolidated rock samples. The transient techniques perform a measurement during the process of heating up. Their advantage is quicker measurements. Transient methods are usually carried out by needle probes. The reciprocal of thermal conductivity is thermal resistivity, usually measured in kelvin-metres per watt \((K\cdot m\cdot W^{-1})\). When dealing with a known amount of material, its thermal conductance and the reciprocal property, thermal resistance, can be described. Unfortunately, there are differing definitions for these terms. For general scientific use, thermal conductance is the quantity of heat that passes in unit time through a plate of particular area and thickness when its opposite faces differ in temperature by one kelvin. For a plate of thermal conductivity \( k \), area \( A \) and thickness \( L \) this is \( kA/L \), measured in \( W\cdot K^{-1} \) (equivalent to: \( W/^\circ C \)).
Thermal conductivity and conductance are analogous to electrical conductivity (A·m$^{-1}$·V$^{-1}$) and electrical conductance (A·V$^{-1}$). There is also a measure known as heat transfer coefficient: the quantity of heat that passes in unit time through unit area of a plate of particular thickness when its opposite faces differ in temperature by one kelvin. The reciprocal is thermal insolence. In summary:

Thermal conductance = $kA/L$, measured in W·K$^{-1}$

Thermal resistance = $L/(kA)$, measured in K·W$^{-1}$ (equivalent to: °C/W)

Heat transfer coefficient = $k/L$, measured in W·K$^{-1}$·m$^{-2}$

Thermal insolence = $L/k$, measured in K·m²·W$^{-1}$.

The heat transfer coefficient is also known as thermal admittance. When thermal resistances occur in series, they are additive. So when heat flows through two components each with a resistance of 1 °C/W, the total resistance is 2 °C/W. A common engineering design problem involves the selection of an appropriate sized heat sink for a given heat source. Working in units of thermal resistance greatly simplifies the design calculation. For example, if a component produces 100 W of heat, and has a thermal resistance of 0.5 °C/W, what is the maximum thermal resistance of the heat sink? Suppose the maximum temperature is 125 °C, and the ambient temperature is 25 °C; then the ΔT is 100 °C. The heat sink's thermal resistance to ambient must then be 0.5 °C/W or less.

A third term, thermal transmittance, incorporates the thermal conductance of a structure along with heat transfer due to convection and radiation. It is measured in the same units as thermal conductance and is sometimes known as the composite thermal conductance. The
term U-value is another synonym. In summary, for a plate of thermal conductivity \( k \) (the k value \([21]\)), area \( A \) and thickness \( t \):

\[
\text{Thermal conductance} = \frac{k}{t}, \text{measured in W·K}^{-1}·m^{-2};
\]
\[
\text{Thermal resistance (R-value)} = \frac{t}{k}, \text{measured in K·m}^2·W^{-1};
\]
\[
\text{Thermal transmittance (U-value)} = \frac{1}{\sum (\frac{t}{k})} + \text{convection} + \text{radiation}, \text{measured in W·K}^{-1}·m^{-2}.
\]

K-value refers to the total insulation value of a building. K-value is obtained by multiplying the form factor of the building (= the total inward surface of the outward walls of the building divided by the total volume of the building) with the average U-value of the outward walls of the building. K value is therefore expressed as \((m^2·m^{-3})·(W·K^{-1}·m^{-2}) = W·K^{-1}·m^{-3}\). A house with a volume of 400 m\(^3\) and a K-value of 0.45 (the new European norm. It is commonly referred to as K45) will therefore theoretically require 180 W to maintain its interior temperature 1 K above exterior temperature. So, to maintain the house at 20 °C when it is freezing outside (0 °C), 3600 W of continuous heating is required.

In metals, thermal conductivity approximately tracks electrical conductivity according to the Wiedemann-Franz law, as freely moving valence electrons transfer not only electric current but also heat energy. However, the general correlation between electrical and thermal conductance does not hold for other materials, due to the increased importance of phonon carriers for heat in non-metals. Highly electrically conductive silver is less thermally conductive than diamond, which is an electrical insulator. Thermal conductivity depends on many properties of a material, notably its structure and temperature. For instance, pure crystalline substances exhibit very different thermal conductivities along different crystal axes, due to differences in phonon coupling along a
given crystal axis. Sapphire is a notable example of variable thermal conductivity based on orientation and temperature, with 35 W/(m·K) along the c-axis and 32 W/(m·K) along the a-axis.[22] Air and other gases are generally good insulators, in the absence of convection. Therefore, many insulating materials function simply by having a large number of gas-filled pockets which prevent large-scale convection. Examples of these include expanded and extruded polystyrene (popularly referred to as "styrofoam") and silica aerogel. Natural, biological insulators such as fur and feathers achieve similar effects by dramatically inhibiting convection of air or water near an animal's skin. Light gases, such as hydrogen and helium typically have high thermal conductivity. Dense gases such as xenon and dichlorodifluoromethane have low thermal conductivity. An exception, sulfur hexafluoride, a dense gas, has a relatively high thermal conductivity due to its high heat capacity. Argon, a gas denser than air, is often used in insulated glazing (double paned windows) to improve their insulation characteristics[23].

Thermal conductivity is important in building insulation and related fields. However, materials used in such trades are rarely subjected to chemical purity standards. Several construction materials' k values are listed below. These should be considered approximate due to the uncertainties related to material definitions. Heat flux is exceedingly difficult to control and isolate in a laboratory setting. Thus at the atomic level, there are no simple, correct expressions for thermal conductivity. Atomically, the thermal conductivity of a system is determined by how atoms composing the system interact. There are two different approaches for calculating the thermal conductivity of a system. The first approach employs the Green-Kubo relations. Although this employs analytic expressions which in principle can be solved, in order to calculate the thermal conductivity of a dense fluid or solid using this relation requires the use of molecular dynamics computer simulation.
The second approach is based upon the relaxation time approach. Due to the anharmonicity within the crystal potential, the phonons in the system are known to scatter. There are three main mechanisms for scattering:

1. Boundary scattering, a phonon hitting the boundary of a system;
2. Mass defect scattering, a phonon hitting an impurity within the system and scattering;
3. Phonon-phonon scattering, a phonon breaking into two lower energy phonons or a phonon colliding with another phonon and merging into one higher energy phonon.

A kinetic theory of solids follows naturally from the standpoint of the normal modes of vibration in an elastic crystalline solid (see Einstein solid and Debye model) – from the longest wavelength (or fundamental frequency of the body) to the highest Debye frequency (that of a single particle). There are simple equations derived to describe the relationship of these normal modes to the mechanisms of thermal phonon wave propagation as represented by the superposition of elastic waves—both longitudinal (acoustic) and transverse (optical) waves of atomic displacement[24-27]. Longitudinal compression (or acoustic or pressure) wave oscillating within the dimensions of a 2-dimensional lattice. Transverse (shear) wave in a 2-dimensional lattice, oscillating in the 3rd spatial dimension. The velocities of longitudinal acoustic phonons in condensed matter are directly responsible for the thermal conductivity which levels out temperature differentials between compressed and expanded volume elements. For example, the thermal properties of glass are interpreted in terms of an approximately constant mean free path for lattice phonons. Furthermore, the value of the mean free path is of the order of magnitude of the scale of structural (dis)order at the atomic or molecular level[28-30]. Thus, heat transport in both
glassy and crystalline dielectric solids occurs through elastic vibrations of the lattice. This transport is limited by elastic scattering of acoustic phonons by lattice defects. These predictions were confirmed by the experiments of Chang and Jones on commercial glasses and glass ceramics, where mean free paths were limited by "internal boundary scattering" to length scales of $10^{-2}$ cm to $10^{-3}$ cm. [31,32]. The phonon mean free path has been associated directly with the effective relaxation length for processes without directional correlation. The relaxation length or mean free path of longitudinal phonons will be much greater. Thus, thermal conductivity will be largely determined by the speed of longitudinal phonons. [31,33]. Regarding the dependence of wave velocity on wavelength or frequency (aka "dispersion"), low-frequency phonons of long wavelength will be limited in relaxation length by elastic Rayleigh scattering. This type of light scattering form small particles is proportional to the fourth power of the frequency. For higher frequencies, the power of the frequency will decrease until at highest frequencies scattering is almost frequency independent. Similar arguments were subsequently generalized to many glass forming substances using Brillouin scattering. [34-37]. Often in heat transfer the concept of controlling resistance is used to determine how to either increase or decrease heat transfer. Heat transfer coefficients represent how much heat is able to transfer through a defined region of a heat transfer area. The inverse of these coefficients are the resistances of those areas. If a wall can be considered, it would have a heat transfer coefficient representing convection on each side of the wall, and one representing conduction through the wall. To obtain an overall heat transfer coefficient, the resistances need to be summed up. Due to the nature of the above reciprocal relation, the smallest heat transfer coefficient (h) or the largest resistance is generally the controlling
resistance as it dominates the other terms to the point that varying the other resistances will have little impact on the overall resistance:

5.6 Experimental

Thermal Conductivity of All the materials were measured at Laljibhai Chaturbhai Institute of Technology, Gujarat. Thermal conductivity measured in powder form with hot-wire method with precision measurement. The measurement was done at equal interval of 5°K.

![Quick Thermal Conductivity Meter](image)

**Figure 5.5 - Quick Thermal Conductivity Meter**

Model No. : QTM-500

Brand Name: KEM

Country: Japan

**Specification:**

Thermal conductivity of all types of sample materials can be measured quickly and easily. Just place the probe on sample surface of
temperature equilibrium and wait for 60 seconds. You will obtain the results. For thin sample materials like film (30 - 100µm), sheet or board (0.1 - 8mm), use the optional software for measurement of thin films

### Measurement method

**Hot Wire Method**

- **Range**: 0.023 - 12W/m • K (standard probe)
- **Precision**: ±5%/reading value per reference plate
- **Reproducibility**: ±3%/reading value per reference plate

### 5.7 Result and Discussion

The thermal conductivity of all crystals was measured using Hot Wire Method and measured on Quick Thermal Conductivity Meter. Thermal conductivity of all crystals measured between 283°K to 373°K with equal interval of 5°K. Thermal conductivity increased as temperature increased in all the crystals which indicate that all the crystals are good conductor of heat but thermal conductivity almost remain constant in schiffbase. In Ni(II) Crystal a thermal conductivity increased continuously up to 363°K and then become constant. The rate of increased in thermal conductivity of Ni(II) Crystal is higher and it is near about metal. This is observed because crystal synthesized by metal of Ni(II). The maximum value of thermal conductivity observed in this work was 0.828 W/cmK. The tabulation and graphical result shown in Table – 4.3 and Figure 4.9 respectively. In Cu(II) Crystal a thermal conductivity increased continuously but the rate of change observed very lower than the Ni(II) crystal. The maximum value of thermal conductivity of Cu(II) Crystal was observed in this work is 0.8161 W/cmK. The tabulation and graphical result shown in Table – 4.3 and Figure 4.10
respectively. In schiffbase a thermal conductivity almost remain constant and observed very lower than the crystals. The maximum value of thermal conductivity of schiffbasel was observed in this work is 3.0314W/cmK. The tabulation and graphical result shown in Table – 5.2 and Figure 4.8 respectively. The rate of change in thermal conductivity is higher in Ni(II) crystal and lower in Cu(II) crystal and schiffbase of in Ni(II) crystal a composition of metal is higher than the Cu(II) crystal
<table>
<thead>
<tr>
<th>Temperature in °C</th>
<th>Temperature in K</th>
<th>Thermal Conductivity in W/cmK</th>
</tr>
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<tr>
<td></td>
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<td>Schiffbase Crystal</td>
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<tr>
<td>10</td>
<td>283</td>
<td>0.072</td>
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<tr>
<td>15</td>
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</table>
Figure 5.6 Thermal conductivity of Schiff base
Figure 5.7 Thermal conductivity of Ni(II) crystal
Figure 5.8 Thermal conductivity of Cu(II) crystal
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Phonon thermal transport in noncrystalline materials

Boundary scattering of phonons in noncrystalline materials