

## Topic 5-Thermal Analysis

**Syllabus** – Thermal analysis and its types, Thermal events, Thermal analysis methods Thermo gravimetric Analysis (TGA), Differential Thermal Analysis (DTA)and Differential Scanning Calorimetry (DSC) w. r. t. Principle, instrumentation, and applications, Interpretation of Thermogram.

### 5.1 Introduction:

Thermal analysis is group of techniques to study properties of materials as a function of temperature.

By using thermal analysis properties such as enthalpy, thermal stability, mass change, coefficient of thermal expansion etc can be studied. Solid state chemistry uses thermal analysis for studying solid state reactions, thermal degradation, phase transitions, preparing phase diagrams etc.

Thermal analysis includes several methods depending on the property of material to be studied/ measured. The important of them are

Sr.No	Name of Technique	Abbr.	Parameter Measured
i	Thermo gravimetric analysis	TGA	Change in Mass as a function of Temperature
ii	Differential Thermal Analysis	DTA	Temperature difference between Sample and Reference as function of temperature/Time
iii	Differential Scanning Calorimetry	DSC	Heat difference between Sample and Reference as function of temperature/Time.
iv	Thermo Mechanical Analysis	TMA	Deformation of material as function of temperature
v	Evolved Gas Analysis	EGA	Study of gases evolved due to decomposition
vi	Dilatometry	DL	Change in volume of material as function of temperature

**5.1.1. Thermal Events** - The main characteristic feature of the solid state is the relatively ordered arrangement of the Constituent atoms, molecules or ions. Just as the concept of an “ideal gas” is useful in describing the behavior of real gases, the concept of a “perfect solid” or “perfect crystal” is useful as the reference point for real solids.

A perfect (crystalline) solid has a completely ordered arrangement of its constituents, while real solids have imperfections of many kinds. When the order present is marginally greater than for liquids, but considerably less than in a perfect crystal, the substance is sometimes referred to as a “non-crystalline solid”.

When liquids composed of complex molecules or ions (e.g. sucrose or silicates and a vast number of organic polymers) are cooled rapidly a glass may be formed. A glass resembles a solid in many of its physical properties, e.g. rigidity, but differs in that the constituents do not show the regular (lattice) arrangement of a crystalline solid. Glasses are thus examples of non-crystalline solids. They do not melt at a sharply-defined temperature, but soften over a temperature interval. This transition from the rigid glassy state to a more flexible form is known as the **glass transition** and the temperature interval over which this change occurs,

known as the glass transition temperature, is of tremendous importance in the practical use of polymers.

The materials subjected to thermal analysis are initially in solid state. When solids are heated they undergo structural changes as shown in following tables which are referred as **thermal events**.

Thermal events on heating a single solid, A, in an inert atmosphere

			$\Delta H$	sign	$\Delta \text{Mass}$
A (s,structure 1)	$\rightarrow$ A (s,structure 2)	Phase transition	yes	+ or -	no
A (s)	$\rightarrow$ A (l)	Melting	yes	+	no
A (glass)	$\rightarrow$ A (rubber)	Glass transition	no	no	no
A (s)	$\rightarrow$ A (g)	Sublimation	yes	+	yes
A (s)	$\rightarrow$ B (s) + gas or gases	Thermal decomposition	yes	+ or -	yes

Other possibilities are

### Decomposition of Solids

Decomposition of a single pure solid substance A may be represented as:

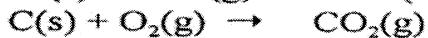


Numerous examples of such decompositions exist, e.g.



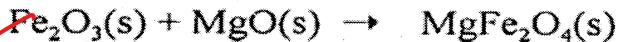
### Reaction with the Surrounding Atmosphere

The sample may react with the surrounding atmosphere e.g.



### Solid-Solid Interactions

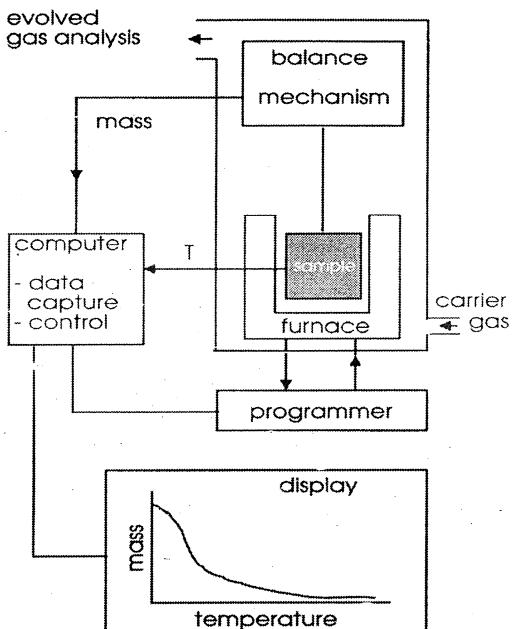
When more than one solid substance is present initially, there are correspondingly more possibilities for interaction on heating. New phases, such as solid solutions or eutectic mixtures, may form, as well as new compounds formed by addition or double decomposition reactions.



The above changes are nearly always accompanied by enthalpy changes, and sometimes also by changes in mass.

## 5.2. Thermo Gravimetric Analysis –TGA

**Principle-** It records change in mass of material under characterization as a function of temperature. The schematic diagram of TGA equipment is as shown in figure.



**TGA setup**

The equipment consists of

1. **Furnace assembly** – It is one of important component. It is precise, compact and programmable. The initial temperature, final temperature, heating rate are programmed between 1°C to 100°C per minute. The temperature range is wide -100 to 2000°C. The lower temperatures are attained by passing cryogenic gases like liquid nitrogen. The furnace assembly is provided with gas inlet, which allows us to change furnace environment. Also a gas outlet is provided to collect and study gaseous decomposition products during sample scan. Temperature of sample is continuously monitored by thermocouple.
2. **Thermo balance** – This is second important component. It records change in mass of sample as and when it undergoes the change. It is null type balancing unit and shows +ve or -ve deviation corresponding to rise or fall in mass of sample. On recording change it again set to null position. The sample holder is platinum crucible which rests on thermo balance.

**Working** – i) The furnace assembly is heated to 1000 °C by passing pure dry N<sub>2</sub> gas for 5-7 minutes in order to remove remaining of previous characterization and cooled to ambient temperature. ii) A small quantity of sample (< 100 mg) is taken in platinum crucible and is properly placed on thermo balance. iii) Furnace assembly is closed and desired furnace environment is chosen. iv) The initial, final temperatures and heating rates are programmed using control unit. v) A sample is then scanned and output is obtained as thermogram.

**Interpretation of TG and DTG Curves** - Possible interpretations of the curves shown in Figure are as follows:

**Type (i) curves:** The sample undergoes no decomposition with loss of volatile products over the temperature range shown. No information is obtained on whether solid phase transitions, melting, polymerization or other reactions involving no volatile products have occurred. The sample is stable over the temperature range considered.

**Type (ii) curves:** The rapid initial mass-loss observed is characteristic of desorption or drying. It could also arise, when working at reduced pressures, from effects such as thermo molecular flow or convection. To check that the mass-loss is real, it is advisable to rerun the sample, which should then produce a type (i) curve, unless the carrier gas contained moisture or was very readily re adsorbed on the sample at the lower temperature.

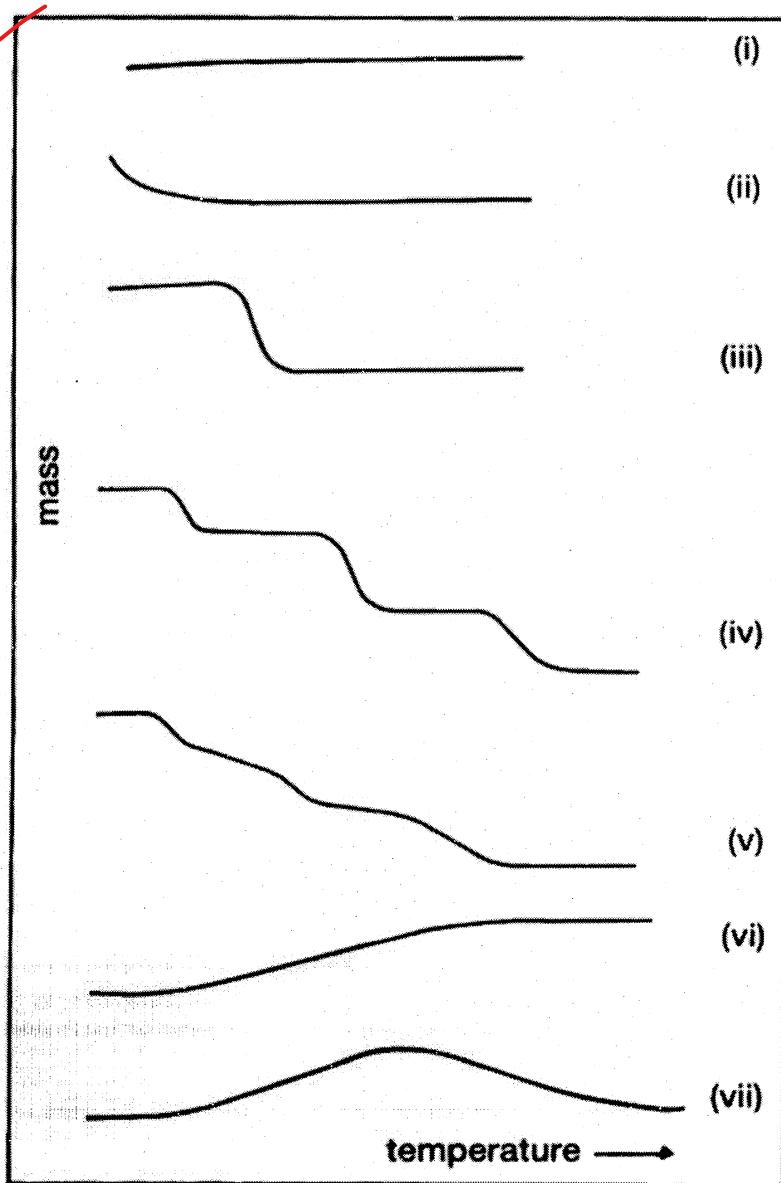
Type (iii) curves: represent decomposition of the sample in a single stage. The curve may be used to define the limits of stability of the reactant, to determine the stoichiometry of the reaction, and to investigate the kinetics of reaction.

Type (iv) curves: indicate multi-stage decomposition with relatively stable intermediates. Again, the temperature limits of stability of the reactant and of the intermediates can be determined from the curve, together with the more complicated stoichiometry of reaction.

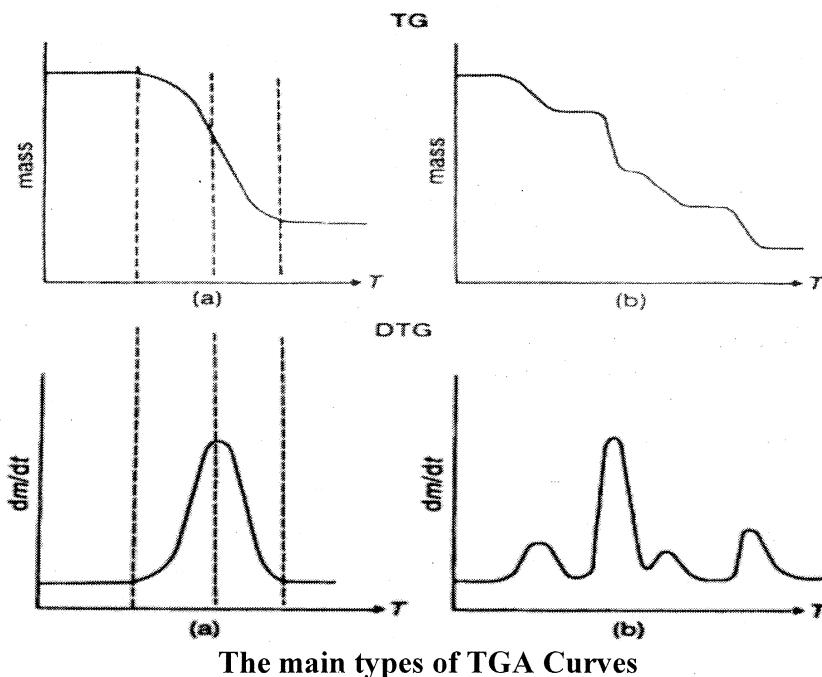
Type (v) curves: also represent multi-stage decomposition, but in this example stable intermediates are not formed and little information on all but the stoichiometry of the overall reaction can be obtained.

Type (vi) curves: show a gain in mass as a result of reaction of the sample with the surrounding atmosphere. A typical example would be the oxidation of a metal sample.

Type (vii) curves: are not often encountered. The product of an oxidation reaction decomposes again at higher temperatures (e.g., Resolution of the individual stages of more complex TG curves can be improved by examining the derivative DTG curves).



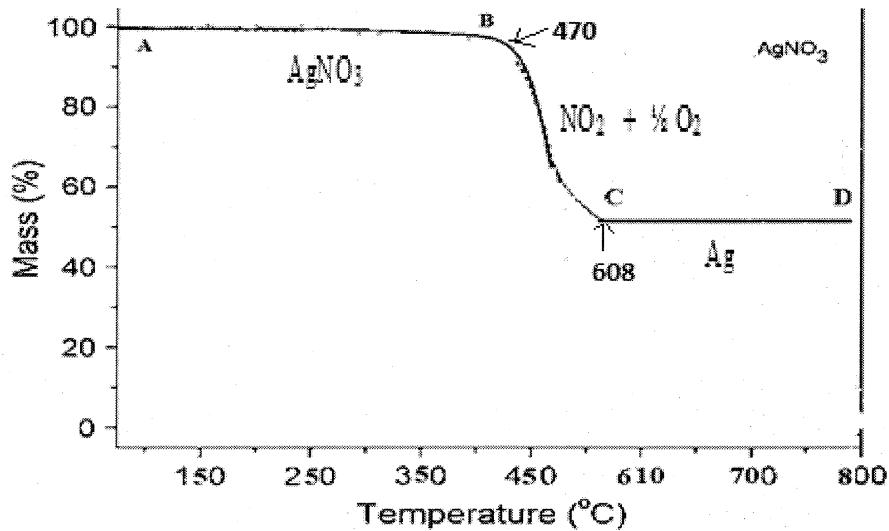
## TGA and DTG curves



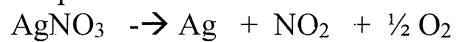
The main types of TGA Curves

### Examples- 1. TG Curve for AgNO<sub>3</sub>

The TGA curve for AgNO<sub>3</sub> is as shown in figure.

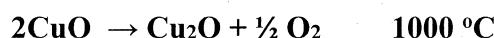
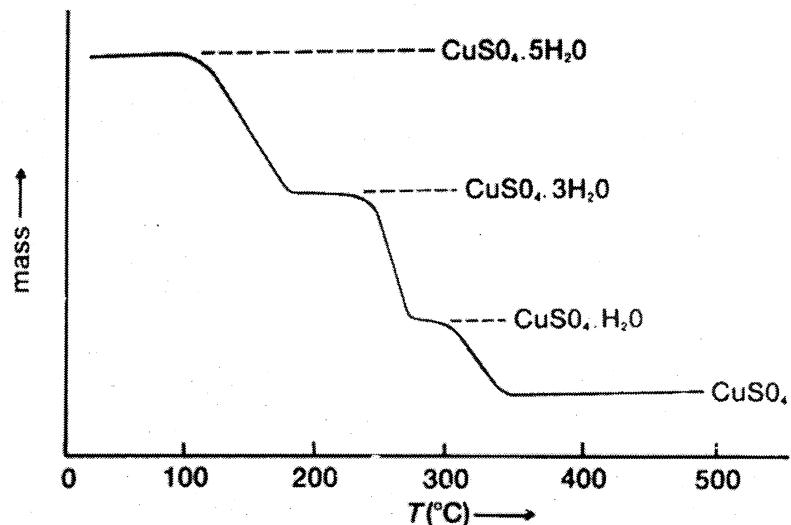


The curve indicates that there is no change in mass up to point B. The weight remains constant up to 470 °C. And AgNO<sub>3</sub> starts to decompose after 470 °C. The mass decreases from point B to point C i.e. up to 608 °C and then it remain constant from C to D.

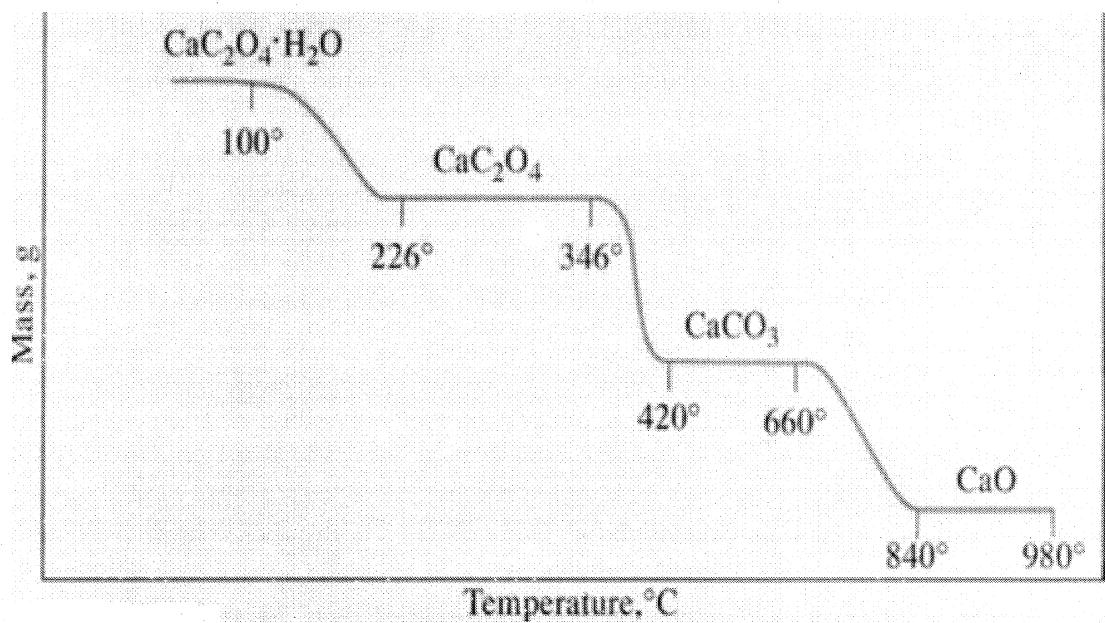


Above 608 °C metallic silver is left as residue.

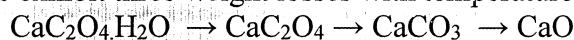
### Example 2: TG Curve for CuSO<sub>4</sub>.5H<sub>2</sub>O



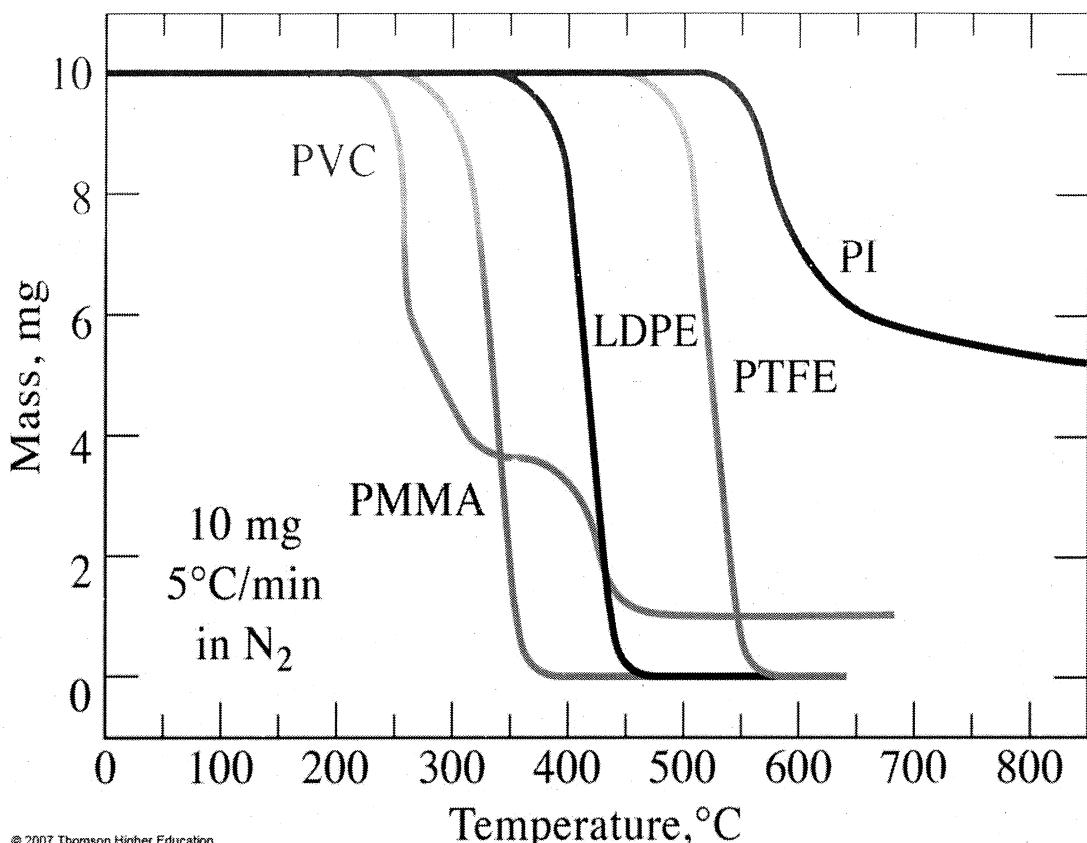
### Example 3: TG Curve for decomposition of CaC<sub>2</sub>O<sub>4</sub>·H<sub>2</sub>O in an inert atmosphere



Calcium oxalate monohydrate is a standard material used to check the performance of TGA. The curve exhibit three weight losses with temperature in inert environment (N<sub>2</sub>)



TGA Thermograms for some common polymeric materials.  
 PVC = polyvinyl chloride; PMMA = polymethyl-methacrylate;  
 LDPE= low-density polyethylene; PTFE = polytetrafluoroethylene;  
 PI = aromatic polypyromelitimide.



© 2007 Thomson Higher Education

### Factors affecting TGA curve –

The lowest temperature,  $T_1$  at which the onset of a mass change can be detected by the thermo balance operating under particular conditions and  $T_f$  is the final temperature at which the decomposition completed. We may like to call this as decomposition temperature, which is not correct. Actually in TGA experiments, both  $T_1$  and  $T_f$  do not have fundamental significance, but they can still be a useful characteristic of a TG curve and termed procedural decomposition temperature. It is often used for the temperature at which mass change appears to commence. This indicates us that procedural decomposition temperature does not have a fixed value, but depends on the experimental procedure employed to get it. Similar to this there are many factors which influence a TGA curve. These factors may be due to instrumentation or nature of sample. We have listed the main factors which affects the shape, precision and accuracy of the experimental results in thermogravimetry.

#### 1. Instrumental factors:

- 1) Furnace heating rate. ii) Recording or chart speed iii) Furnace atmosphere iv) Geometry of Sample holder/ location of sensors v) Sensitivity of recording mechanism. vi) Composition of sample container.

#### 2. Sample Characteristics:

- a) Amount of sample b) Solubility of evolved gases in sample. c) Particle size d) Heat of reaction e) Sample packing f) Nature of sample g) Thermal conductivity.

### a) Furnace heating rate

At a given temperature, the degree of decomposition is greater the slower the heating rate, and thus it follows that the shape of the TG curve can be influenced by the heating rate. For a single stage endothermic reaction it has been found that:

- i)  $(T_i)F > (T_i)S$
- ii)  $(T_f)F > (T_f)S$
- iii)  $(T_f - T_i)F > (T_f - T_i)S$

Where subscripts F and S indicate fast and slow heating rate respectively. For example, calcium carbonate would not show any mass loss below 600 °C, when heated in a thermo balance at heating rate of 3 °C per min., and yet it is known that CO<sub>2</sub> is evolved at 250 °C. Similarly, polystyrene decomposes 10% by mass when heating rate is 1 °C per min by 357 °C and 10% by mass when heating rate is 5 °C per min by 394 °C. More specifically, it is observed that the procedural decomposition temperature  $T_i$ , and also  $T_f$  (the procedural final temperature) will decrease with decrease in heating rate and the TG curve will be shifted to the left. This effect is illustrated in following Fig. The appearance of an inflection in a TG curve at a fast heating rate may well be resolved into a plateau at a slower heating rate. Therefore, in TGA there is neither optimum no standard heating rate, but a heating rate of 3 °C per min. gives a TG curve with maximum meaningful resolution.

### b) Recording or Chart Speed

The chart speed on the recording of the TG curve of rapid or slow reaction has pronounced effect on the shape of the TG curves. For a slow decomposition reaction low chart speed is recommended for recording the TG curve because at high chart speed the curve will be flattened and it will not show the sharp decomposition temperature. For a slow reaction followed by a rapid one at the lower chart speed the curve will show less separation in the two steps than the higher chart speed curve. For fast-fast reaction followed by slower one similar observation was observed in shorter curve plateaus.

### c) Furnace Atmosphere

The effect of atmosphere on the TG curve depends on (i) the types of the reaction (ii) the nature of the decomposition products and (iii) type of the atmosphere employed. The effect of the atmosphere on TG curve may be illustrated by taking the example of thermo decomposition of a sample of monohydrates of calcium oxalate in dry O<sub>2</sub> and dry N<sub>2</sub> as shown Fig.

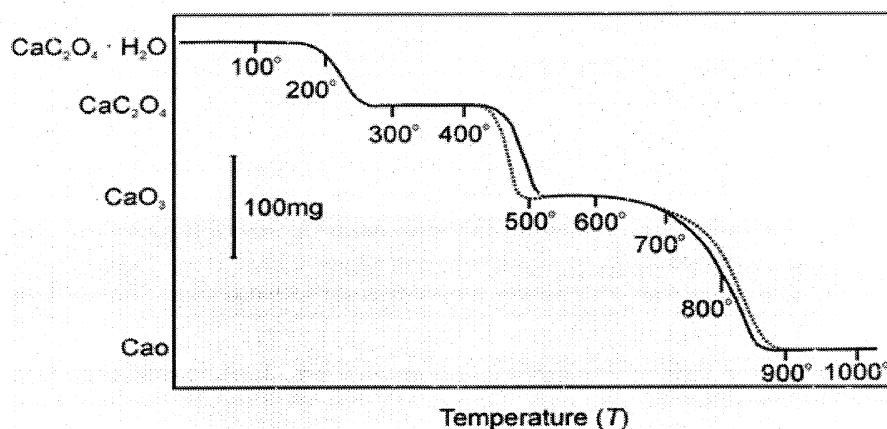
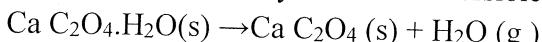


Fig. TG Curve of Calcium Oxalate in O<sub>2</sub> and N<sub>2</sub> atmosphere: [—] N<sub>2</sub>, [----] O<sub>2</sub>

The first step, which is dehydration is reversible reaction.



This is unaffected because both gases are equally effective in sweeping the evolved water vapours away from the sample surface.

For the second step,



The curve diverges in O<sub>2</sub> atmosphere because the oxygen reacts with evolved CO, giving a second oxidation reaction which is highly exothermic and so raises the temperature of the un-reacted sample. The temperature accelerates the decomposition of the compound more rapidly and completely at a lower temperature as shown in the above diagram in dry O<sub>2</sub> than in N<sub>2</sub> atmosphere.

The third step in decomposition reaction is also reversible reaction.



This step should not be influenced by O<sub>2</sub> or N<sub>2</sub>. However there is a slight difference in curves for the two gases as shown in diagram. The small difference was due to the difference in the nature/composition of CaCO<sub>3</sub> formed in the two atmospheres. This is due to the particle size, surface area, lattice defects or due to the other physical characteristics of CaCO<sub>3</sub> formed.

#### d) Sample Holder

The sample holders range from flat plates to deep crucible of various capacities. The shape of the TG curve will vary as the sample will not be heated in identical condition. Generally, it is preconditioning that the thermocouple is placed on near the sample as possible and is not dipped into the sample because it might be spoiled due to sticking of the sample to the thermocouple on heating. So actual sample temperature is not recorded, it is the temperature at some point in the furnace near the sample. Thus it leads to source of error due to the thermal lag and partly due to the finite time taken to cause detectable mass change. If the sensitivity of recording mechanism is not enough to record the mass change of the sample then this will also cause error in recording the weight change of the sample. If the composition of the sample contains is such that it reacts either with the sample, or product formed or the evolved gases then this will cause error in recording the mass change of the sample.

#### e) Effect of Sample Mass

The sample mass affects the TG curve in following.

- i) The endothermic and exothermic reactions of the sample will cause sample temperature to deviate from a linear temperature change.
- ii) The degree of diffusion of evolved gases through the void space around the solid particles.
- iii) The existence of large thermal gradients throughout the sample particularly, if it has a low thermal conductivity. Thus, it is preferable to use as small a sample as possible depending on the sensitivity of the balance.

#### f) Effect of Sample Particle Size

The particle size will cause a change in the diffusion of the evolved gases which will alter the reaction rate and hence the curve shape. The smaller the particle size, greater the extent of decomposition at any given temperature. The use of large crystal may result in apparent very rapid mass loss during heating. This may be due to the mechanical loss of part of the

sample by forcible ejection from the sample container, when the accumulated evolved gases within the coarse grains are suddenly released.

### g) Effect of Heat of Reaction

The heat of reaction will affect the extent to which sample temperature proceeds or succeeds the furnace temperature. This depends on whether the reaction is exothermic or endothermic and consequently the extent of decomposition will also be affected. The other sample characteristics such as sample packing, nature of the sample and its thermal conductivity will also affect the shape of TG curves. If the sample is packed loosely then the evolved gases may diffuse more easily than if the sample packed tightly. If the sample reacts with the sample container on heating then it will not give the mass of the product formed so the sample will change. We can avoid this effect by a sensible choice of sample container.

## Applications of TGA

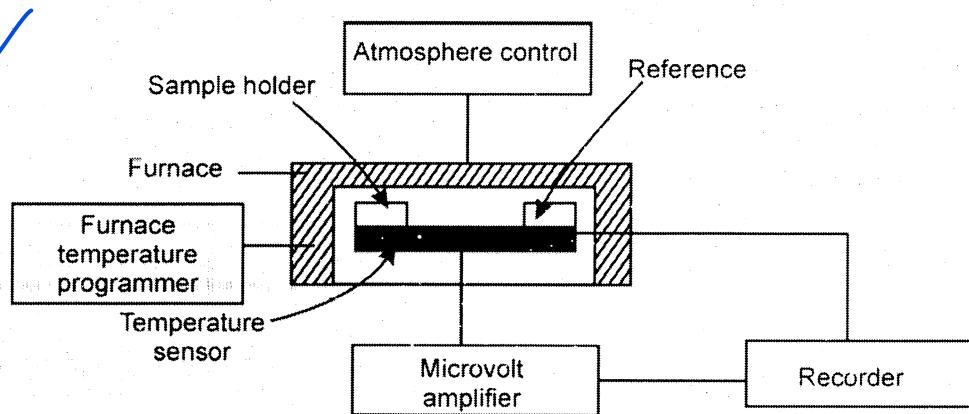
- i) Purity and thermal stability.
- ii) Solid state reactions.
- iii) Decomposition of inorganic and organic compounds.
- iv) Determining composition of the mixture.
- v) Corrosion of metals in various atmosphere.
- vi) Pyrolysis of coal, petroleum and wood.
- vii) Roasting and calcinations of minerals.
- viii) Reaction kinetics studies.
- ix) Evaluation of gravimetric precipitates.
- x) Oxidative and reductive stability.
- xi) Determining moisture, volatile and ash contents.
- xii) De solvation, sublimation, vaporizations, sorption, desorption, chemisorption's.

## 5.3 Differential Thermal Analyses - DTA

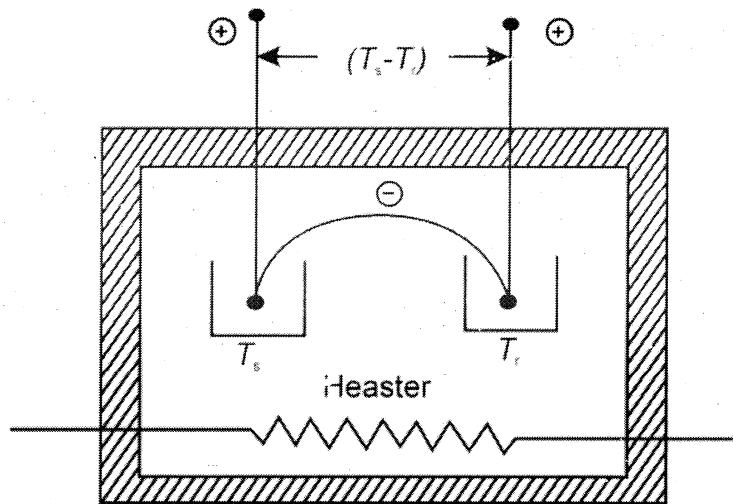
DTA involves measurement of temperature difference between sample under study and reference material kept under identical conditions as a function of temperature or time.

When sample does not undergo any physical/ chemical change there is no temperature difference between sample and reference, but when such changes occur then there exist temperature difference between sample and reference, ( $\Delta T$ ) which may be positive or negative depending on exothermic or endothermic type of reaction.

The schematic diagram of DTA equipment is as shown in following figure.



DTA Set up



### **DTA Furnace**

The equipment consist of furnace assembly. The furnace unit is precise and programmable. Heating rate with time can be programmed according to sample type under test. The furnace unit consist sample and reference holders. Reference is a thermally inert material. For sample Silica, Fire clay, Pyrex, Ni or Pt crucibles can be used depending on type of sample to be tested. Temperature of sample and reference is monitored by using separate thermocouples. ( $T_s$  and  $T_r$ ). Furnace unit is protected from electrical interference arise of furnace wiring by ceramic coated platinum sheets. The data acquisition system continuously receives temperatures from thermocouples over selected temperature range and sent it to data processor.

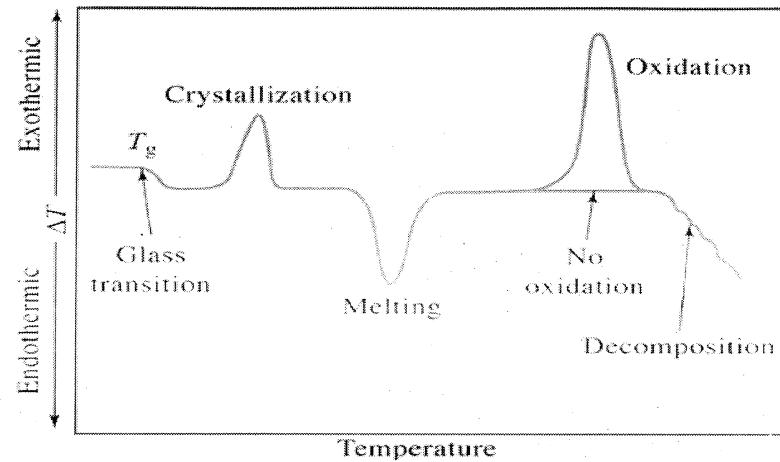
### **Working –**

- i) A known mass of sample under test (up to 100 mg) is kept in sample holder.
- ii) Furnace unit is closed
- iii) The unit is programmed with initial, final temperature along with heating/ cooling rates
- iv) A suitable furnace environment is created by passing inert gas/  $N_2$  or  $O_2$  gas.
- v) The sample is then scanned to get Thermogram.

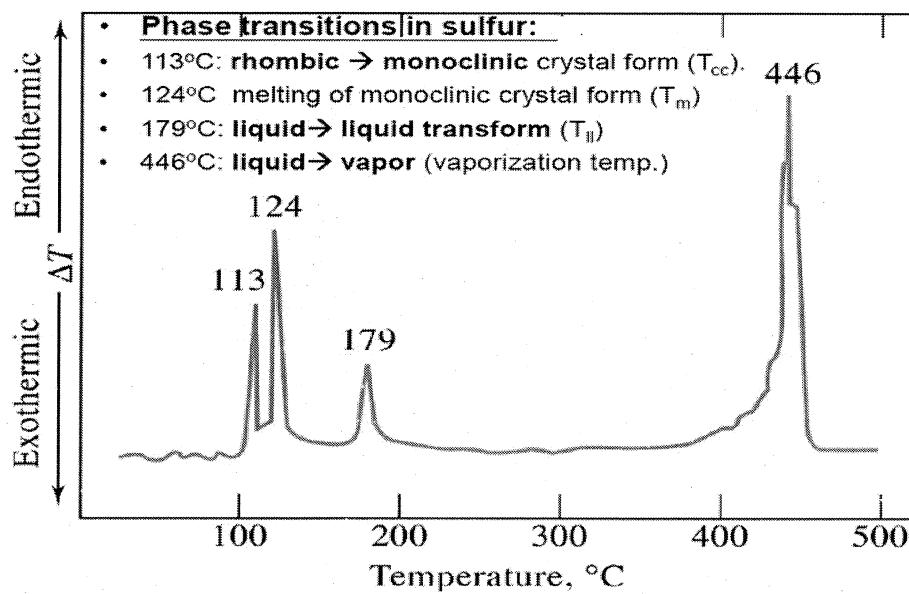
### **DTA Thermograms:**

A typical DTA thermogram is shown in figure. It explains nature of peaks for various physical (Glass Transition, Crystallization, melting) transitions and chemical changes (Decomposition, oxidation etc.)

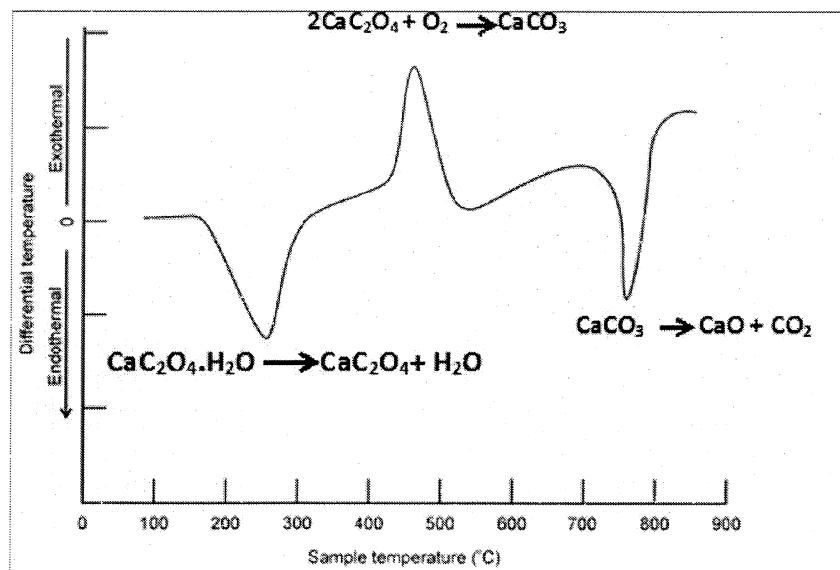
Also it depicts whether change is exothermic or endothermic.



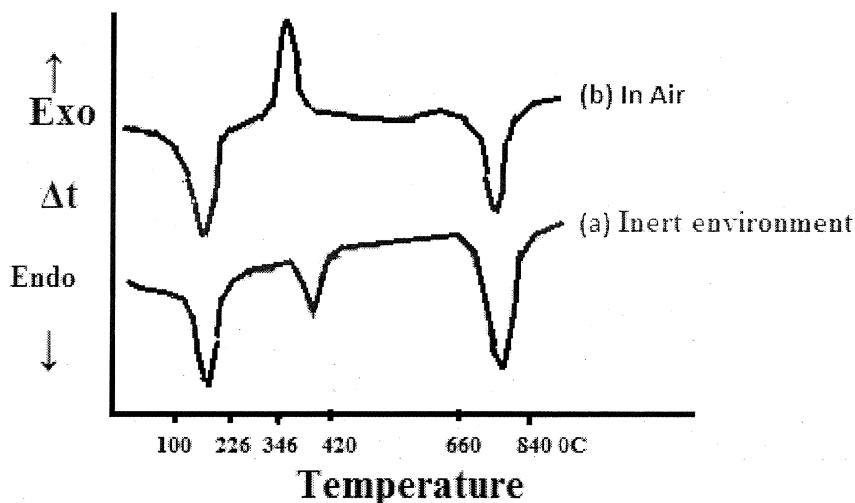
### 1.Typical DTA thermogram



### 2. DTA thermogram showing Phase transitions for sulphur



### 3.DTA thermogram for decomposition of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in air



#### 4. DTA thermogram for decomposition of $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in air and Inert environment

In above decomposition of calcium oxalate in air there are two minima and one maxima (exothermic peak).

When same decomposition is carried in inert environment maxima changes to minima as it will not provide oxygen for reaction  $\text{C} + \text{O}_2 \rightarrow \text{CO}_2$  which is highly exothermic.

Applications – DTA is used for i) Studying phase transformations ii) Study structure inversions iii) Study dehydration, decomposition of material/ substance iv) Study corrosion and oxidation behavior of material at different temperatures v) Study crystallization and crystal structure transitions vi) Study release of structural/residual stress or strain vii) Study reactions with other ingredients.

#### Factors that influence DTA Curve

Factor	Effect	Suggestions
1. Heating rate	Change in peak size and position	Use a low heating rate
2. Location of thermocouple	Irreproducible curve	Standardise thermocouple location
3. Atmosphere around sample	Change in the curve	Inert gas should be allowed to flow
4. Amount of sample	Change in peak size and position	Standardise sample mass
5. Particle size of sample	Irreproducible curves	Use small, uniform particle size
6. Packing density	Irreproducible curves	Standardise packing technique
7. Sample container	Change in peak	Standardise container

#### 5.4. Differential Scanning calorimetry (DSC)

**Principle** -In DSC the heat provided to sample or reference in order to keep zero temperature difference between them is measured and plotted against temperature of furnace or time to get a thermogram. This is the basis of Differential Scanning Calorimetry (DSC). The curve obtained in DSC is between  $dH/dt$  in  $\text{mJ s}^{-1}$  or  $\text{mcal s}^{-1}$  as a function of time or temperature.

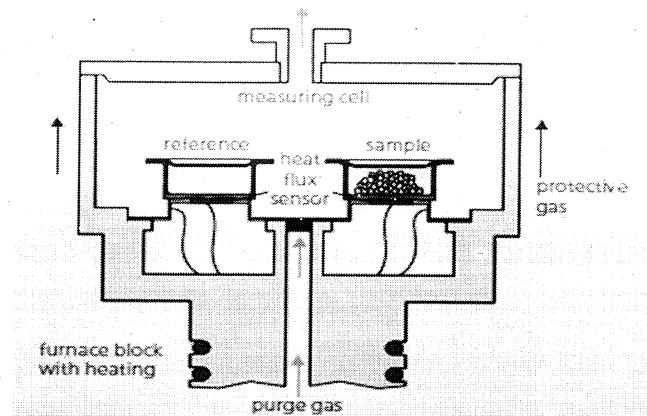


Fig a

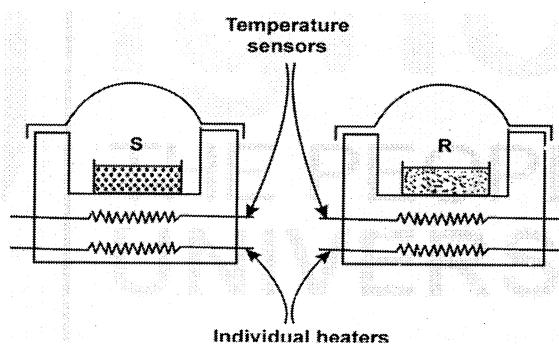


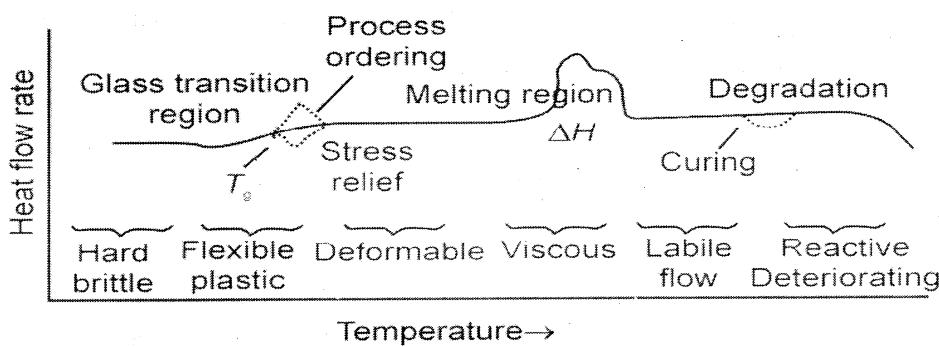
Fig b

#### a) DSC Cell schematic b) Secondary heating sources for sample and reference

**Instrumentation** – The schematic diagram of DSC set up is as shown in figure a and b. It consist of furnace assembly called DSC Cell. It is compact, thermally insulated, precise and programmable to preset heating and cooling rates along with temperature range of study. The DSC cell consists of sample and reference holders with additional separate heating arrangements as shown in fig b. The cell is protected from electric interferences arising out of furnace wiring. The sample and reference material is taken in aluminium, gold, copper, nickel, platinum etc. pans closed with lids. The cell environment can be changed by passing pure oxygen, nitrogen, inert gas or air through gas inlet. The temperatures of sample and reference are monitored by separate thermocouples. The temperature range is from -100 to 750°C. Some equipment's are designed to work up to 1500°C.

**Working**- The DSC cell is heated to 500°C by passing air to eliminate remaining of previous characterization and cooled to ambient temperature. The sample (up to 100mg) is taken in suitable pan (Al, Au, Cu, Pt, Ni etc.) by considering reactivity. Sample and reference pan is kept properly on sample and reference holder positions. The DSC cell is then closed. The initial, final temperature range with rates of heating, cooling are programmed. A suitable cell environment is chosen by passing required gas. Then

sample is scanned to get thermogram. A typical DSC thermogram for polymeric material is shown below.



**Schematic DSC Curve for change in behavior of Polymeric materials**

### Applications of DSC-

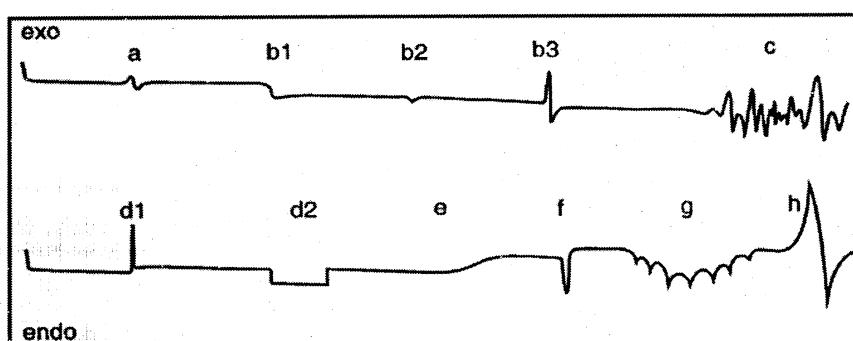
DSC is used to measure melting temperature, heat of fusion, latent heat of melting, reaction energy, glass transition temperature, crystalline phase transitions, specific heat, oxidation induction time etc. The major applications are i) Metal alloy melting temperature and heat of fusion, ii) Metal structure transition temperature and heat of transformation, iii) Inter metallic phase transition temperature and exothermal energies, iv) Oxidation temperature and energy, v) Melting behavior of complex organic material – temperature and enthalpies of melting determine purity, vi) Glass transition temperature of polymer, vii) Crystalline to amorphous transition temperature of polymers, plastics, viii) To study reaction kinetics of materials, ix) To study thermal properties related to plastics, adhesives, sealants, pharmaceuticals, waxes, food, lubricants, oils, fertilizers, catalysts etc.

### Interpreting DSC curves

The art of interpreting curves has yet to be integrated into commercially available computer programs. The interpretation of a DSC measurement curve is therefore still something you have to do yourself. It requires a considerable amount of experience in thermal analysis as well as a knowledge of the possible reactions that your particular sample can undergo.

### Recognizing artifacts

The first thing to do is to examine the curve for any obvious artifacts that could lead to a possible misinterpretation of the results. Artifacts are effects that are not caused by the sample under investigation. Figure 1 shows examples of a number of such artifacts.



**Fig. 1. DSC artifacts (details are given in the text):** An artifact can very often be identified by repeating the measurement with a new sample of the same substance and observing whether the effect occurs again either at the same place or at a different place on the curve. Exceptions to this are f and h, which can be very reproducible.

They include:

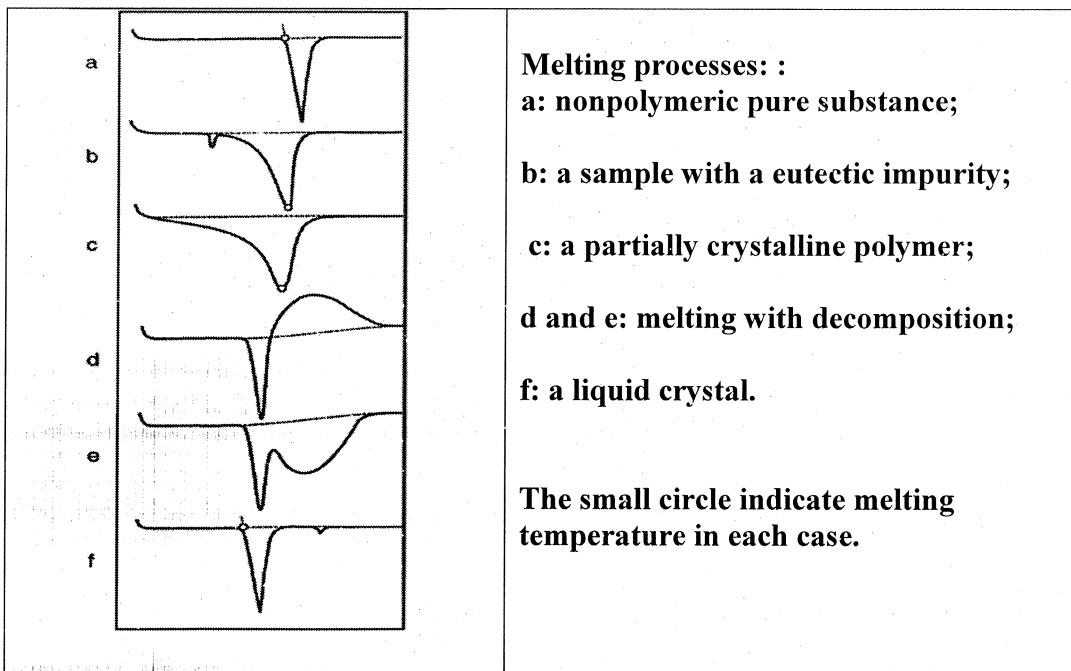
- a) An abrupt change of the heat transfer between the sample and the pan:
- b) An abrupt change of the heat transfer between the pan and the DSC sensor:
  - 1) Distortion of a hermetically sealed Al pan due to the vapor pressure of the sample.
  - 2) Slight shift of the Al pan during a dynamic temperature program due to different coefficients of expansion
  - 3) The measuring cell suffers a mechanical shock: The pans jump around on the sensor and can move sideways.
- c) The entry of cool air into the measuring cell due to a poorly adjusted measuring cell lid leads to temperature fluctuations which cause a very noisy signal.
- d) Electrical effects:
  - 1) Discharge of static electricity in a metallic part of the system, or power supply disturbances (spikes)
  - 2) Radio emitters, mobile (cellular) phones and other sources of high frequency interference.
- e) A sudden change of room temperature, e.g. through sunshine.
- f) The lid of the pan bursts as a result of increasing vapor pressure of the sample.
- g) Intermittent (often periodic) closing of the hole in the lid of the pan due to droplets that condense.
- h) Contamination of the sensors caused by residues of a sample from previous experiments.

### The thermal effects

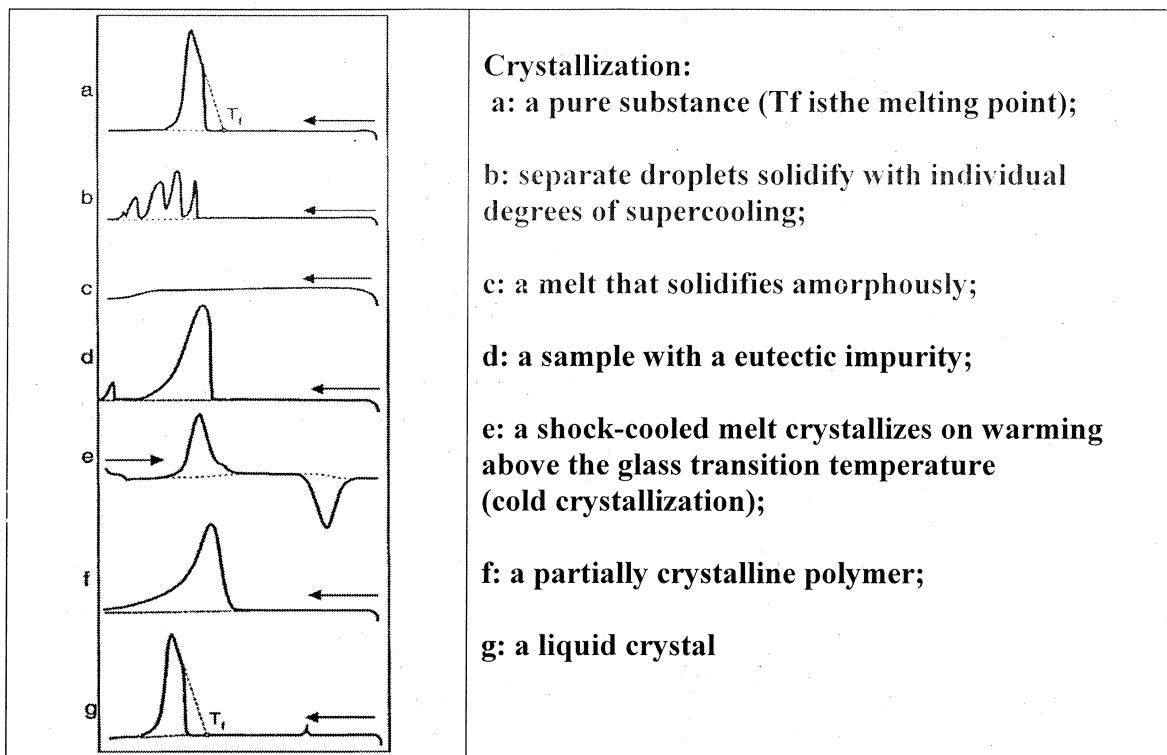
This is characteristic for this substance always occur at the same temperature. This problem can often be overcome by heating the system in air or oxygen.

Consider examples of some physical transitions like Crystallization, Melting, Glass transition, Monotropic (Non reversible) and Enantiotropic (Reversible Transitions) with respect to nature of curve and its interpretation as shown in diagram (**Reference- Metler Toledo Usercom 11 manual**) The nature of curve may slightly vary depending on manufacturer of equipment.

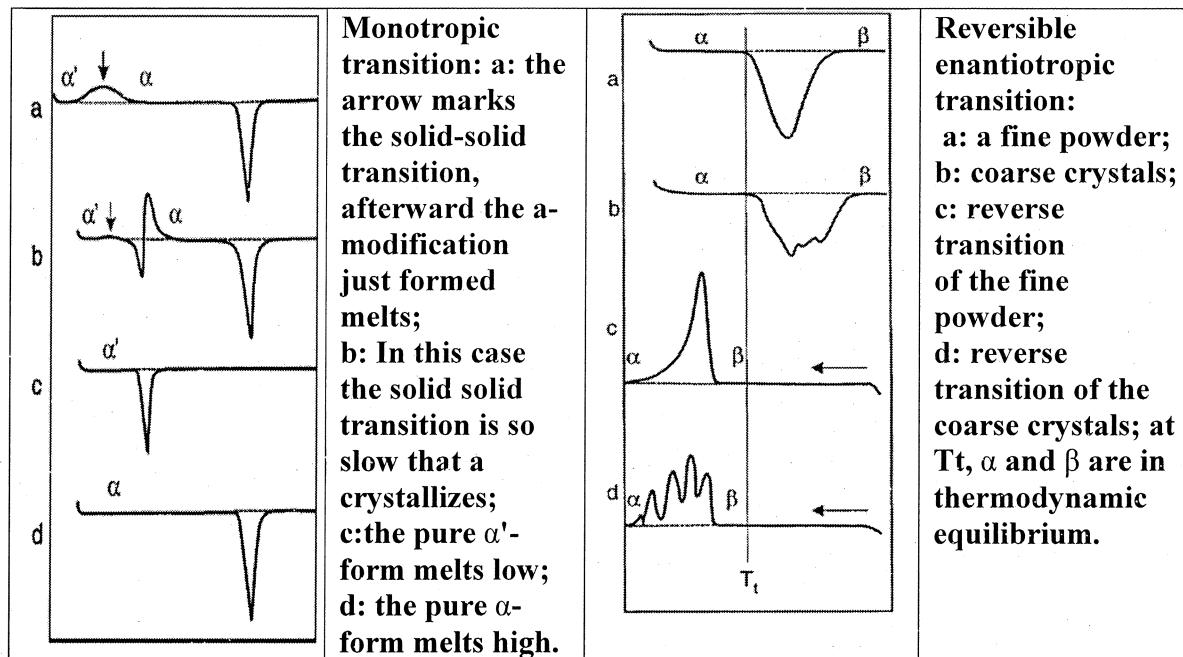
#### A) Melting –



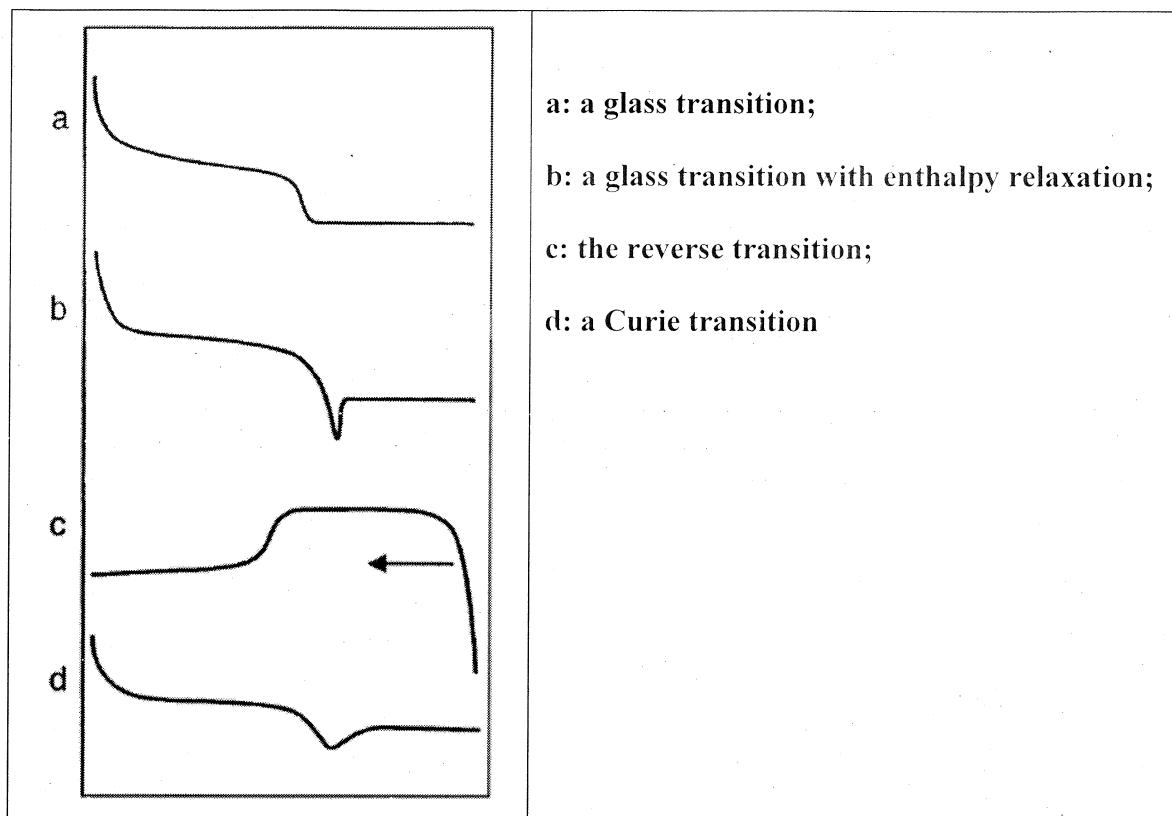
## B) Crystallization



## C) Monotropic and Enantiotropic Transitions –



#### D) Glass Transition –



- a: a glass transition;
- b: a glass transition with enthalpy relaxation;
- c: the reverse transition;
- d: a Curie transition

#### Comparison - TGA, DTA and DSC

Point to Compare	TGA	DTA	DSC
1. Parameter measured	Change in mass versus temperature ( $\Delta$ mass)	Temperature Difference between sample and inert reference with respect to temperature/Time	Heat Difference between sample and inert reference with respect to temperature
2. Thermogram obtained	$\Delta$ mass versus temperature	$\Delta T$ versus Temperature or Time	$\Delta$ Heat versus Temperature or
3. Sample Holder	Platinum Crucible	Platinum, Silica, Fireclay, Pyrex, Nickel crucible depending on type of sample	Gold, Aluminum, Ni, Cu, Alumina, Silica pans with lid depending on sample type
4. Temperature range of study	-100 to 1800 °C	-100 to 1500 °C	-100 to 750 °C some equipments up to 1500 °C
5. Secondary Heating source	No	No	Yes
6. Amplification of temperature signal	No	Yes	Yes
7. Protection from interference due to furnace wiring	No	Yes	Yes
8. Characterization type	Thermal Stability, Thermal Decomposition, Phase transitions	Physical transitions and exothermic or endothermic nature	Physical transitions and Heat of such transitions
9. Limitation	No	No	Not suitable for Halogenated samples as they damage DSC cell

## **Questions-**

### **Answer the following**

1. What is thermal analysis? What are different ways / methods of thermal analysis?
2. Discuss Thermal events occurring when a solid material/ substance is subjected to thermal analysis.
3. With neat diagram explain construction, working and interpretation of thermograms for i) TGA ii) DTA iii) DSC
4. Discuss / List / Summarize applications of TGA / DSC / DTA.
5. Compare and contrast / Distinguish between TGA-DTA-DSC, TGA-DTA, TGA-DSC, DSC-DTA.
6. Discuss factors that influence TGA, DTA
7. Draw neat diagram of TGA thermogram and explain i)  $\text{AgNO}_3$  ii)  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$  iii)  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

### **Give reason**

1. TGA uses platinum crucible.
2. The crucible used for TGA must be flat bottomed.
3. In thermal analysis sample size must be as small as possible.
4. DSC require secondary heating source.
5. Temperature signals are amplified in case of DTA & DSC
6. Halogenated samples are undesirable for DSC.
7. The flow rate of purge gas must be properly controlled in thermal analysis.