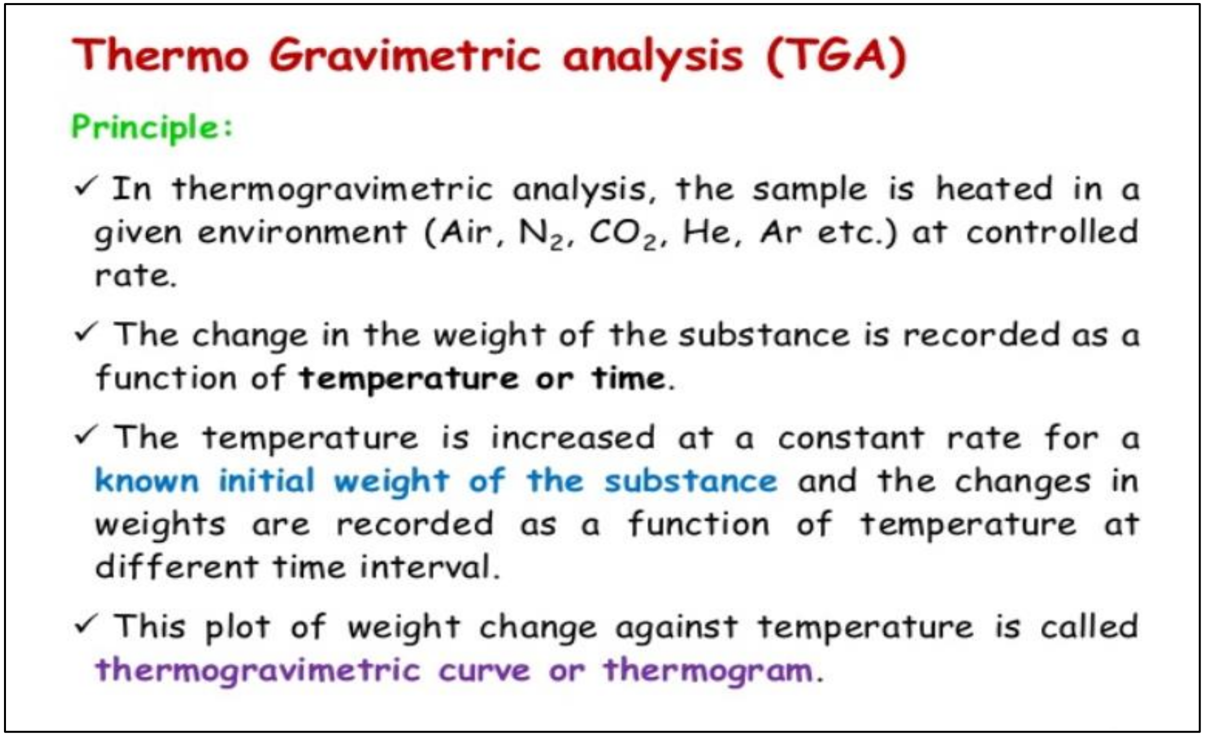
Thermal Methods:

Thermal methods are based upon the measurement of the dynamic relationship between temperature and some property of the system such as mass and heat absorbed or evolved by/from it.

- Differential Scanning Calorimetry,
  DSC
- Differential Thermal Analysis,
  DTA
- Thermogravimetry,
  TG

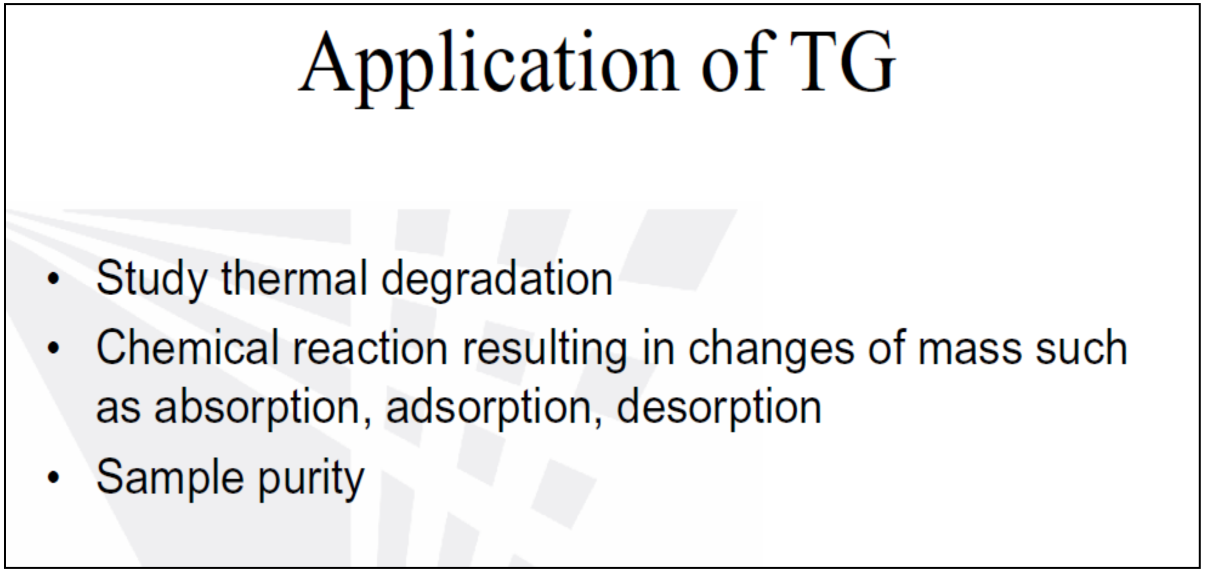
are the most important thermal methods



Thermo Gravimetric analysis (TGA)

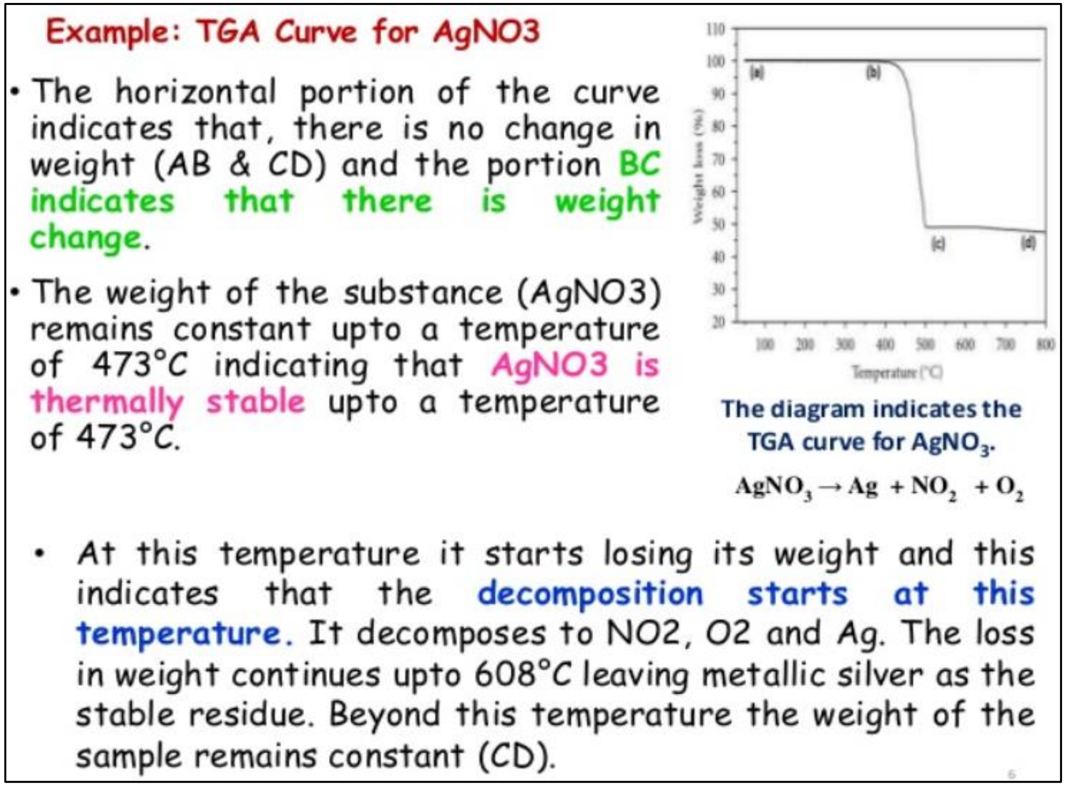
Principle:

- ✓ In thermogravimetric analysis, the sample is heated in a given environment (Air, N_2, CO_2, He, Ar etc.) at controlled rate.
- ✓ The change in the weight of the substance is recorded as a function of **temperature or time**.
- ✓ The temperature is increased at a constant rate for a **known initial weight of the substance** and the changes in weights are recorded as a function of temperature at different time interval.
- ✓ This plot of weight change against temperature is called **thermogravimetric curve or thermogram**.



Application of TG

- Study thermal degradation
- Chemical reaction resulting in changes of mass such as absorption, adsorption, desorption
- Sample purity



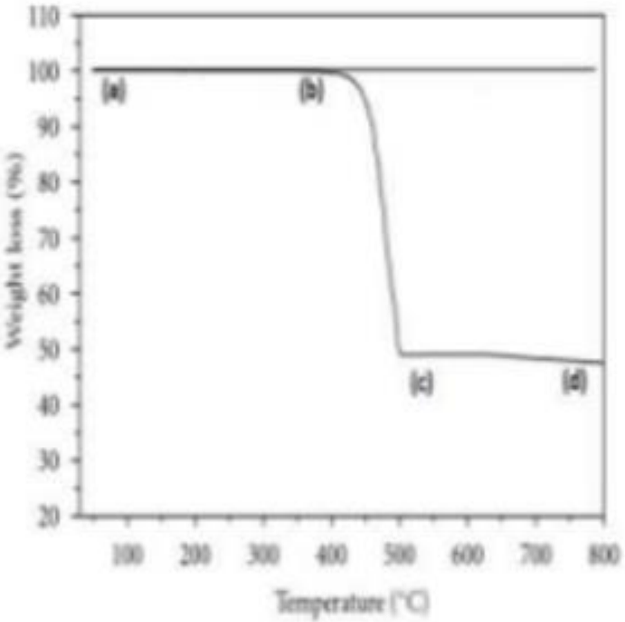
Example: TGA Curve for AgNO3

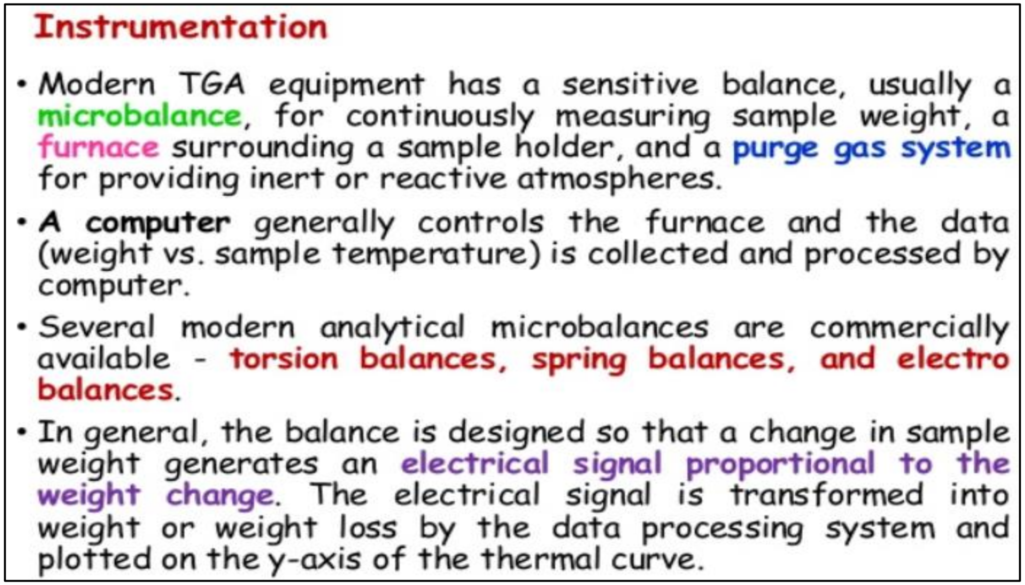
- The horizontal portion of the curve indicates that, there is no change in weight (AB & CD) and the portion BC indicates that there is weight change.
- The weight of the substance (AgNO3) remains constant upto a temperature of 473°C indicating that AgNO3 is thermally stable upto a temperature of 473°C.

The diagram indicates the TGA curve for $AgNO_3$.

$$AgNO_3 \rightarrow Ag + NO_2 + O_2$$

- At this temperature it starts losing its weight and this indicates that the decomposition starts at this temperature. It decomposes to NO2, O2 and Ag. The loss in weight continues upto 608°C leaving metallic silver as the stable residue. Beyond this temperature the weight of the sample remains constant (CD).

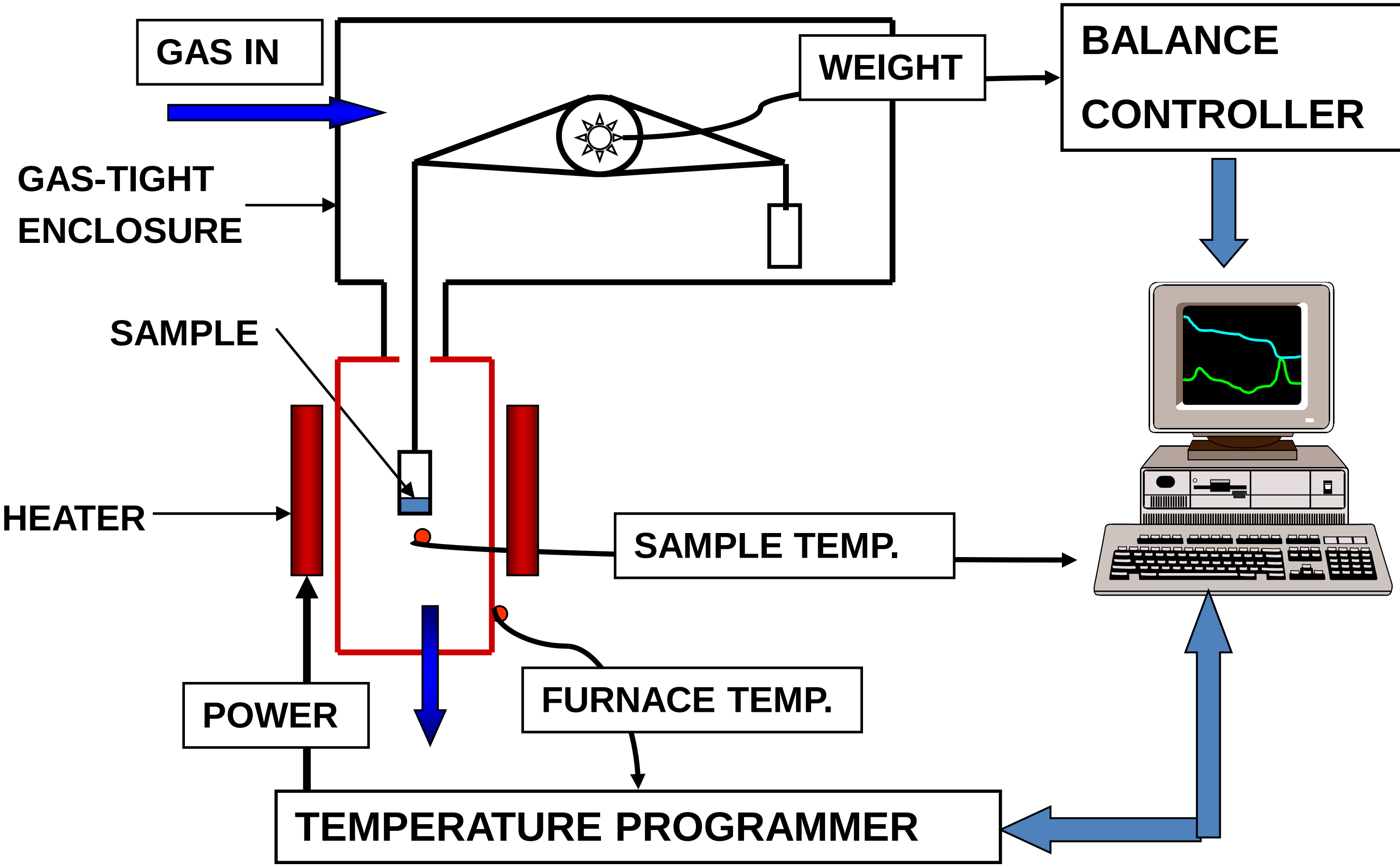




Instrumentation

- Modern TGA equipment has a sensitive balance, usually a microbalance, for continuously measuring sample weight, a furnace surrounding a sample holder, and a purge gas system for providing inert or reactive atmospheres.
- **A computer** generally controls the furnace and the data (weight vs. sample temperature) is collected and processed by computer.
- Several modern analytical microbalances are commercially available - **torsion balances, spring balances, and electro balances**.
- In general, the balance is designed so that a change in sample weight generates an **electrical signal proportional to the weight change**. The electrical signal is transformed into weight or weight loss by the data processing system and plotted on the y-axis of the thermal curve.

Instrumentation



GAS IN

GAS-TIGHT ENCLOSURE

WEIGHT

BALANCE CONTROLLER

SAMPLE

HEATER

SAMPLE TEMP.

FURNACE TEMP.

POWER

TEMPERATURE PROGRAMMER

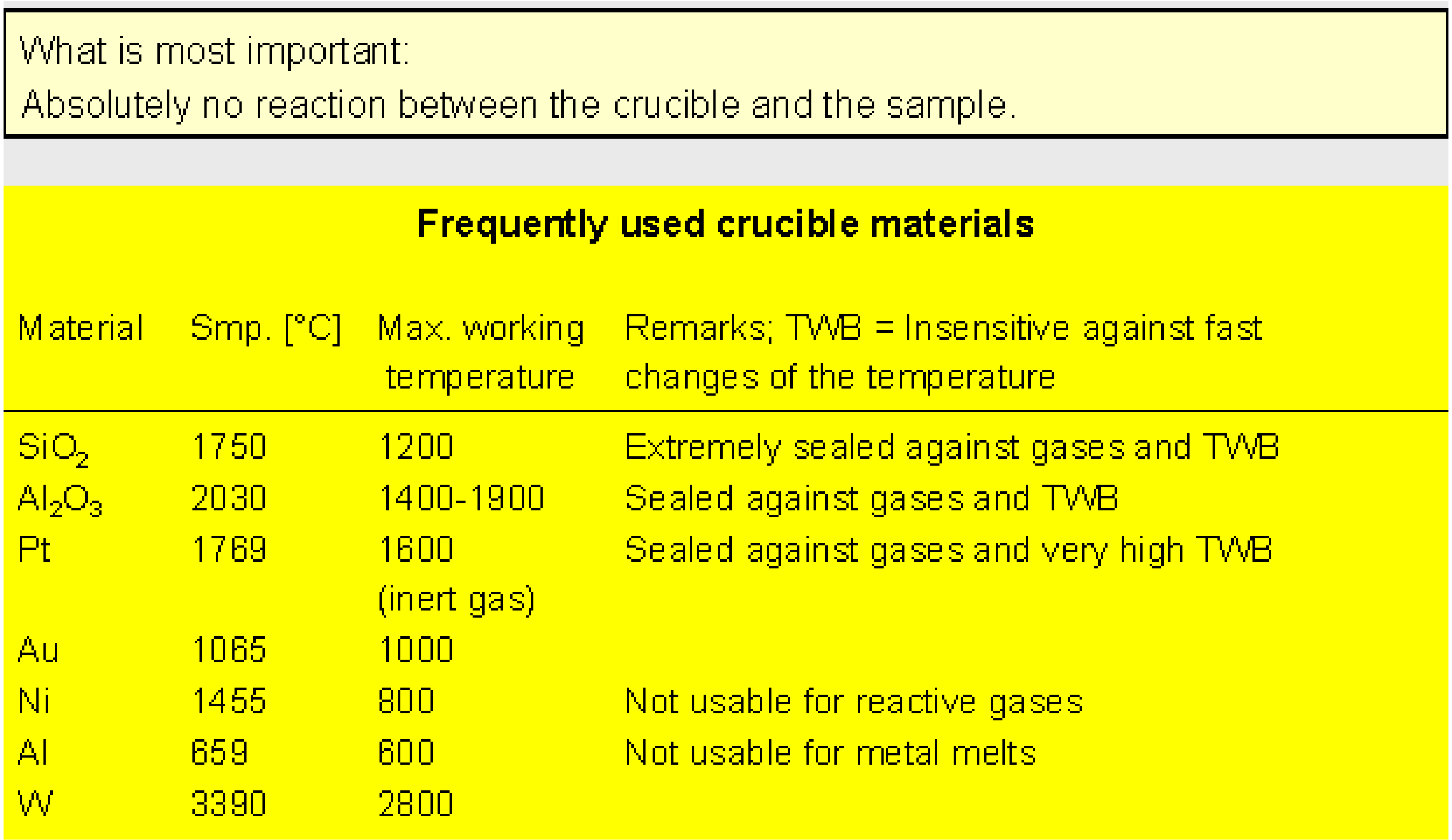
Thermogravimetric instrumentation should include several basic components:

balance,; A heating device,; A unit for temperature measurement and control,; Automatically recording the mass and temperature changes,; A system to control the atmosphere around the sample.

- **The furnace** surrounds the sample and sample holder. It must be capable of being programmed for a linear heating rate.
- **Modern instruments** can be heated and cooled rapidly. These instruments that heat at rates of up to 1000^{0}C/min are available.
- **Commercial instruments** can heat at rates up to 200^{0}C/min from room temperature to about 1200^{0}C; cooling by forced air can be done at 50^{0}C/min.
- There are furnaces available with upper temperatures of 1500^{0}C, 1700^{0}C, or 2400^{0}C; these higher temperature instruments are useful for studying refractory materials and engineering materials.

- The furnace **must be** able to be **purged with a desired gas, to provide the correct atmosphere** for the experiment and to remove gaseous products from the sample compartment.
- **Argon or nitrogen** is used when an inert atmosphere is desired.
- **Air** is often used for oxidation and combustion studies.
- **Hydrogen gas** may be used to provide a reducing atmosphere, with the appropriate precautions to prevent explosions.
- Modern instruments permit the purge gas to be switched automatically, so that the sample can start heating in an inert atmosphere and be switched to air or other reactive gas at high temperatures,

Crucibles

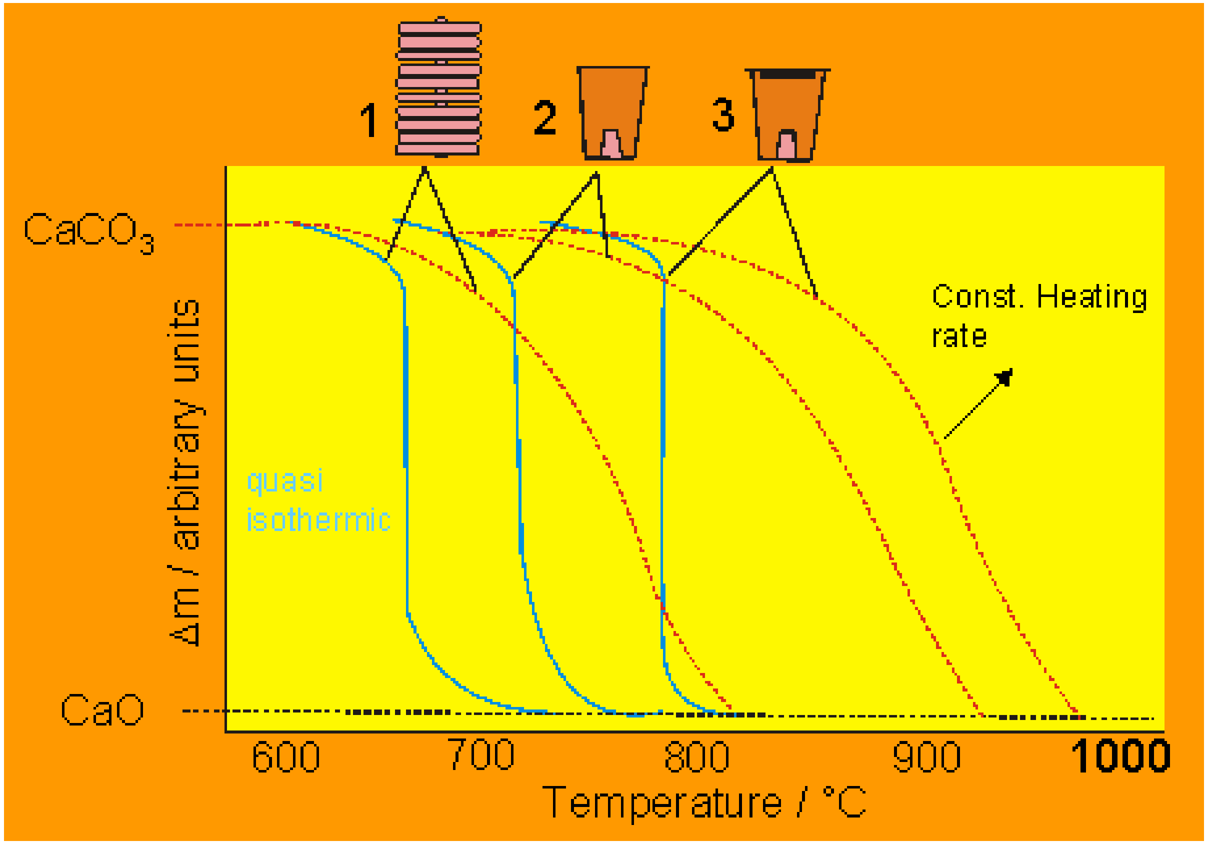


What is most important:
Absolutely no reaction between the crucible and the sample.

Frequently used crucible materials

Material	Smp. [°C]	Max. working temperature	Remarks; TWB = Insensitive against fast changes of the temperature
SiO_2	1750	1200	Extremely sealed against gases and TWB
Al_2O_3	2030	1400-1900	Sealed against gases and TWB
Pt	1769	1600 (inert gas)	Sealed against gases and very high TWB
Au	1065	1000	
Ni	1455	800	Not usable for reactive gases
Al	659	600	Not usable for metal melts
W	3390	2800	

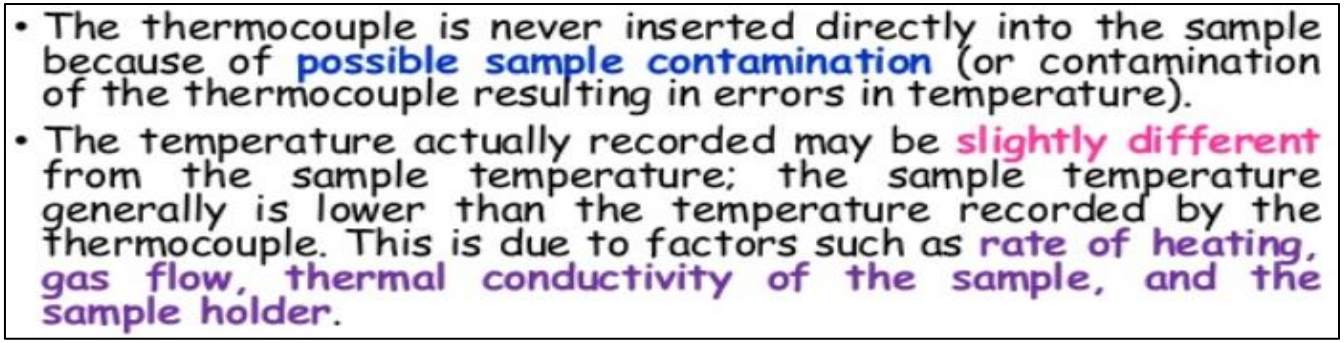
Decomposition temperatures of $CaCO_3$ as function of crucibles



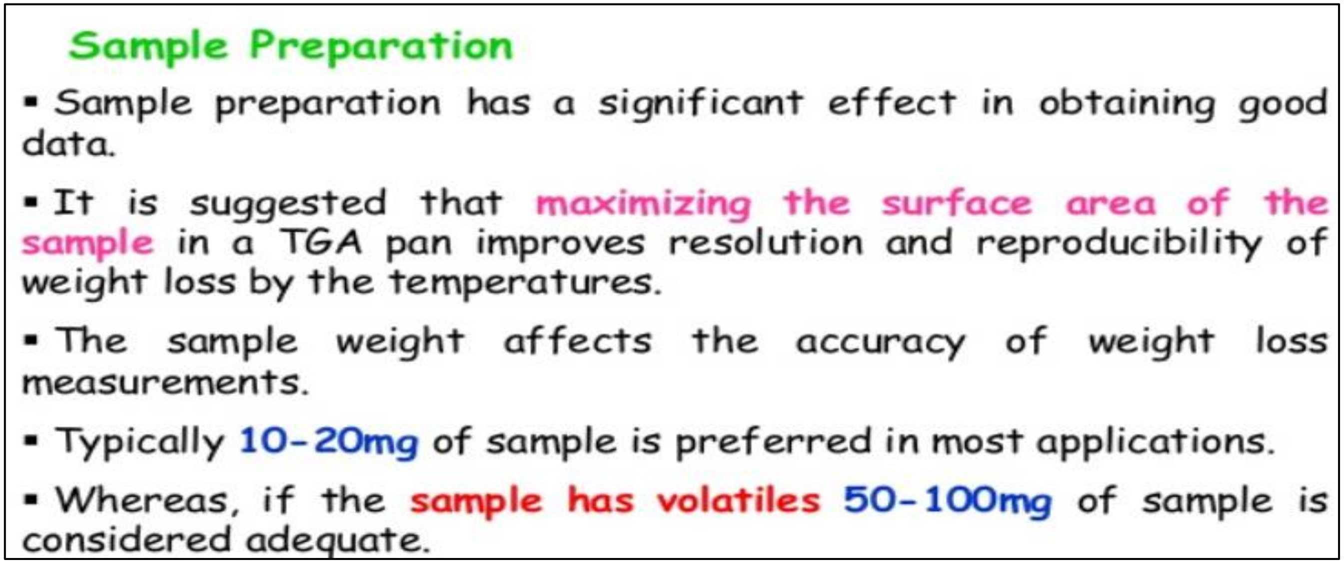
1: Open crucible with large surface

2: Open crucible

3: Closed crucible



- The thermocouple is never inserted directly into the sample because of **possible sample contamination** (or contamination of the thermocouple resulting in errors in temperature).
- The temperature actually recorded may be **slightly different** from the sample temperature; the sample temperature generally is lower than the temperature recorded by the thermocouple. This is due to factors such as **rate of heating, gas flow, thermal conductivity of the sample, and the sample holder**.



Sample Preparation

- Sample preparation has a significant effect in obtaining good data.
- It is suggested that **maximizing the surface area of the sample** in a TGA pan improves resolution and reproducibility of weight loss by the temperatures.
- The sample weight affects the accuracy of weight loss measurements.
- Typically **10-20mg** of sample is preferred in most applications.
- Whereas, if the **sample has volatiles** **50-100mg** of sample is considered adequate.

TGA of calcium oxalate monohydrate (CaC2O4.H2O)

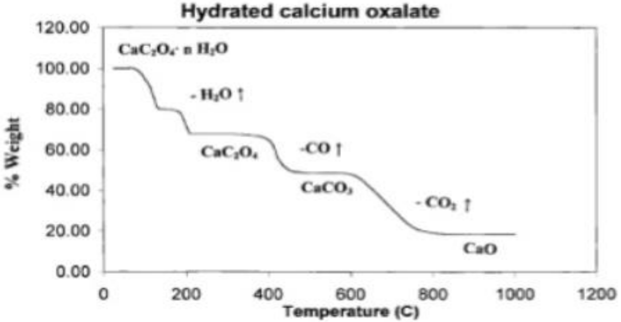
- The successive plateau corresponds to the formation of anhydrous salt, calcium carbonate and calcium oxide.

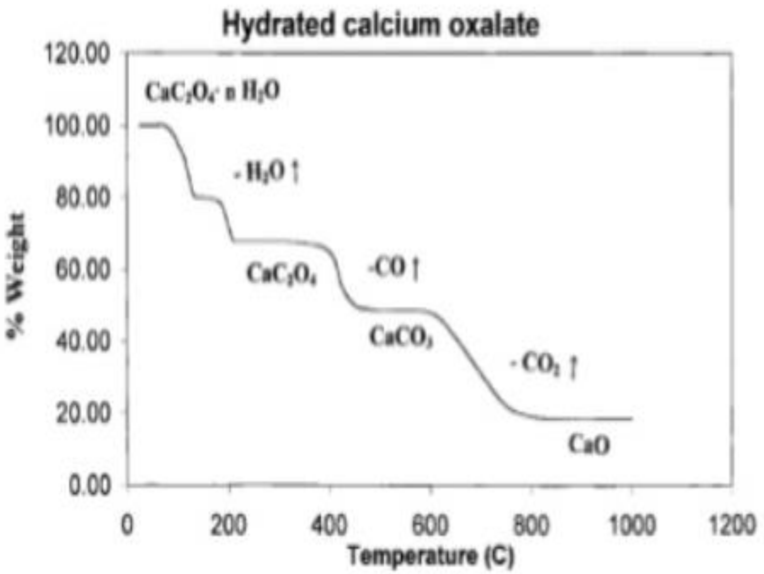
$$\text{(a) } CaC_2O_4.H_2O \rightarrow CaC_2O_4 + H_2O$$

$$\text{(b) } CaC_2O_4 \rightarrow CaCO_3 + CO$$

$$\text{(c) } CaCO_3 \rightarrow CaO + CO_2$$

- The thermogram indicates that the loss of water begins at 100°C and loss of CO at 450°C and CO2 at 600°C





Factors affecting the TG curve

The factors which may affect the TG curves are classified into two main groups.

(1) Instrumental factors

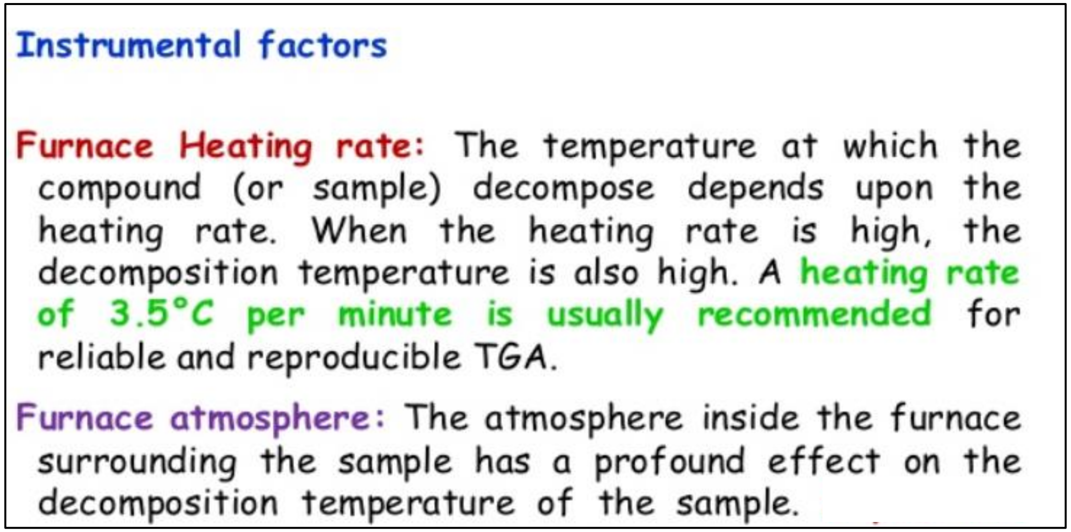
(a) Furnace heating rate

(b) Furnace atmosphere

(2) Sample characteristics includes

(a) Weight of the sample

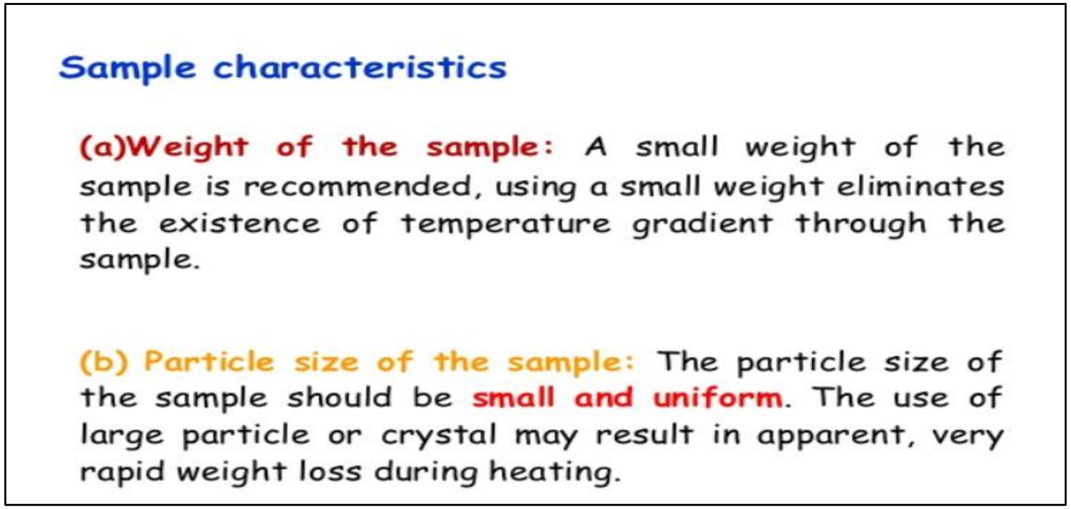
(b) Sample particle size



Instrumental factors

Furnace Heating rate: The temperature at which the compound (or sample) decompose depends upon the heating rate. When the heating rate is high, the decomposition temperature is also high. A **heating rate of 3.5°C per minute is usually recommended** for reliable and reproducible TGA.

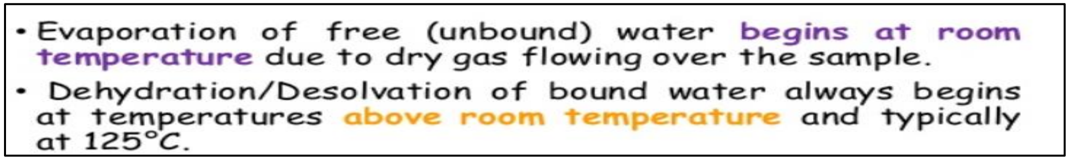
Furnace atmosphere: The atmosphere inside the furnace surrounding the sample has a profound effect on the decomposition temperature of the sample.



Sample characteristics

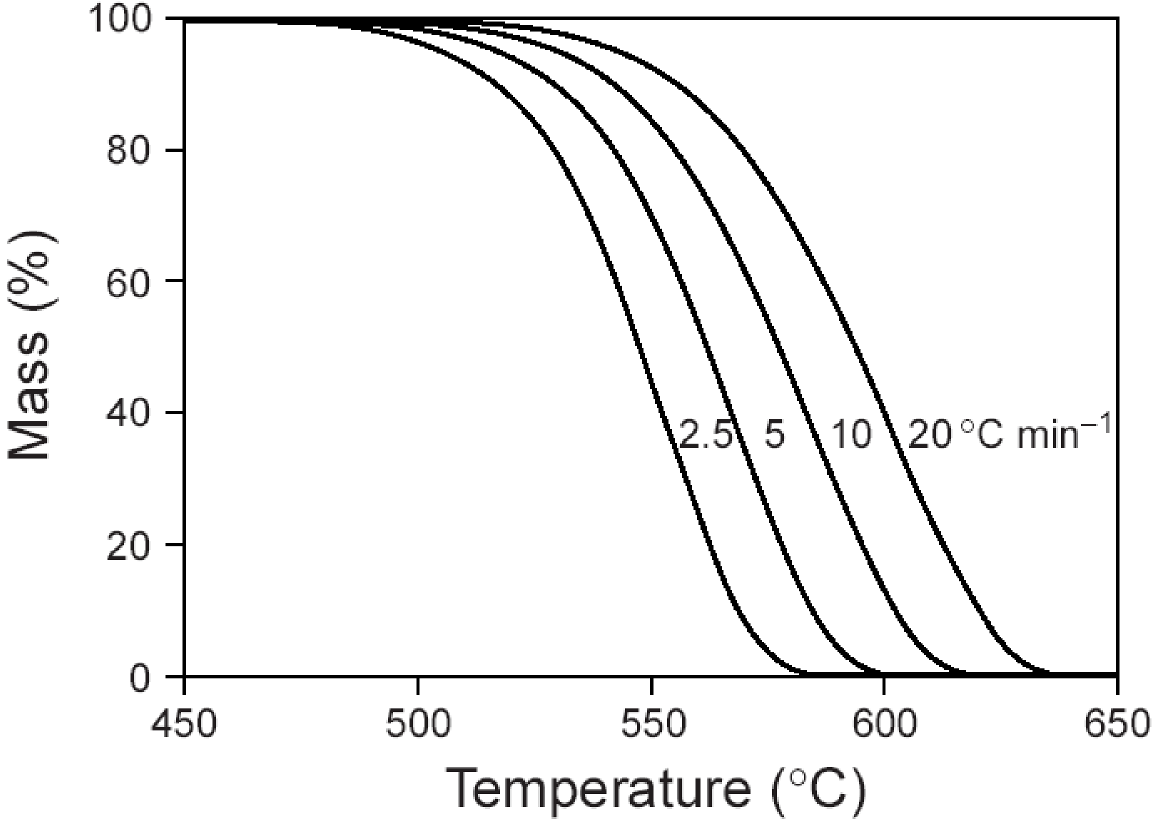
(a)Weight of the sample: A small weight of the sample is recommended, using a small weight eliminates the existence of temperature gradient through the sample.

(b) Particle size of the sample: The particle size of the sample should be **small and uniform**. The use of large particle or crystal may result in apparent, very rapid weight loss during heating.



- Evaporation of free (unbound) water **begins at room temperature** due to dry gas flowing over the sample.
- Dehydration/Desolvation of bound water always begins at temperatures **above room temperature** and typically at 125°C.

effect of heating rate



10 mg samples of PTFE, heated at 2.5, 5, 10 and 20 °C/min in nitrogen

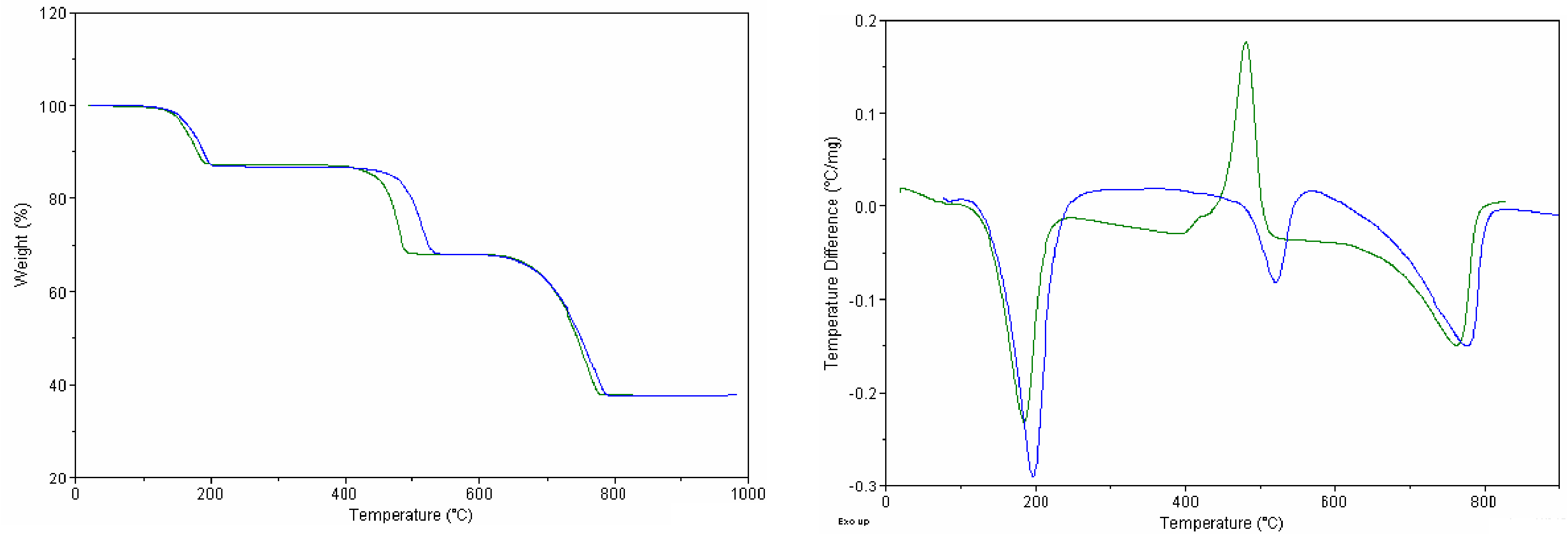
General considerations

Suitable samples for TG are solids that undergo one of the two general types of reaction:

Reactant(s) → Product(s)+Gas	(a mass loss)
Gas + Reactant(s) → Product(s)	(a mass gain)

Processes occuring without change in mass (e.g., the melting of a sample) obviously cannot be studied by TG.

effect of atmosphere



$CaC_2CO_4.H_2O$ in air and nitrogen

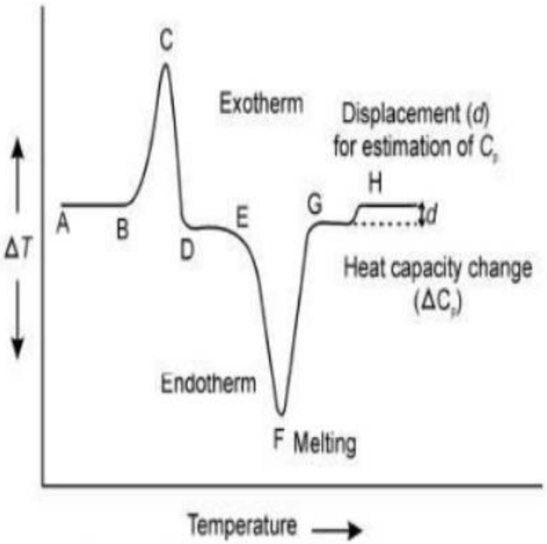
- In an overview of thermal analysis testing it is always preferable to do a TGA experiment on unknown samples before doing a DSC experiment (especially for pharmaceuticals) because Decomposition of pharmaceuticals renders products which are insoluble and generally sticky on the inside of a DSC cell. These products will lower the life use of a DSC cell.
- Therefore, knowing of the decomposition temperatures of all drugs, heat in a DSC evaluation should maintain 50°C below to those decomposition temperatures.

Differential Thermal Analysis (DTA)

Definition: DTA is a technique in which the temperature between sample & thermally inert reference substance is continuously recorded as a function of temperature /time.

DTA Principle

- Differential thermal analysis is a technique in which the temperature of the substance under investigation is compared with the temperature of a thermally inert material.



This differential temperature is then plotted against time, or against temperature (**DTA curve, or thermogram**).

The area under a DTA peak is the **enthalpy change** and is not affected by the heat capacity of the sample.

- Both sample and reference material must be heated under **carefully controlled conditions.**

- If **zero temperature** difference b/w sample & reference material - sample does not undergo any chemical or physical change.

- If any reaction (physical or chemical change) takes place **temperature difference (ΔT)** will occur b/w sample & reference material .

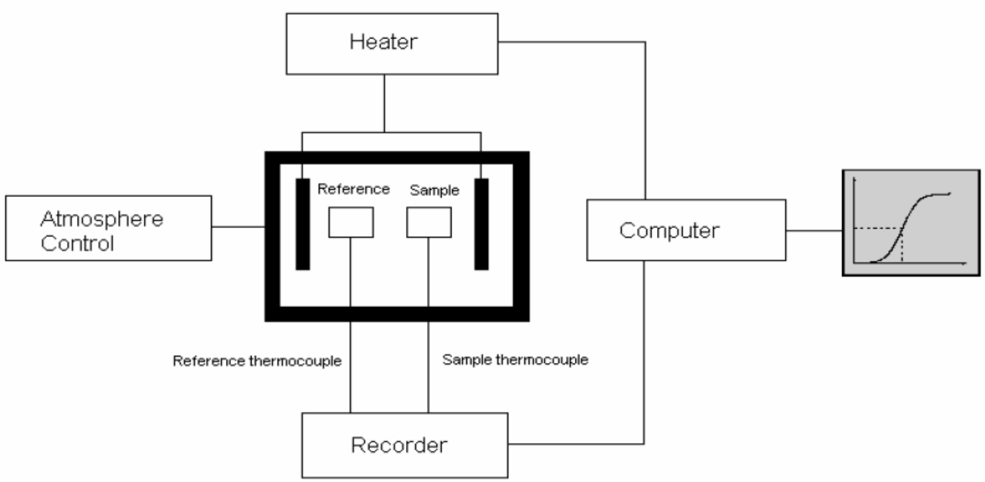
- Some changes result in heat being absorbed by the sample. These types of changes are called **endothermic**.
- Examples of endothermic changes include **phase changes such as** melting (fusion), vaporization, sublimation, and some transitions between two different crystal structures for a material. **Chemical reactions** can be endothermic, including dehydration, decomposition, oxidation-reduction, and solid-state reactions.
- Other changes result in heat being given off by the sample. Such changes are termed **exothermic.**

Instrumentation

➢Sample holder

- ✓ Sample & reference crucibles are generally metallic (Al, Pt) or ceramic (silica) and may or may not have a lid.
- ✓ Best results are obtained when the area of contact between the sample and the pan or crucible is maximized.
- ✓ Samples are generally in the 1-10 mg range for analytical applications.

- The temperature difference between reference and sample is monitored as a function of temperature



➢Furnace

- ✓ The sample and reference should be matched thermally and arranged symmetrically with the furnace so that they are both heated or cooled in an identical manner.
- ✓ The metal block surrounding the wells acts as a heat sink.
- ✓ The temperature of the heat sink is slowly increased using an internal heater.
- ✓ The sink in turn simultaneously heats the sample and reference material.

➢Sensors & Recording system

- ✓ A pair of matched thermocouples is used.
- ✓ One pair is in contact with the sample or the sample container, the other pair is in contact with the reference.

- Operating temperatures for DTA instruments are generally room temperature to about 1600^{0}C,

- When a physical change takes place in the sample, heat is absorbed or generated.
- For example, when a metal carbonate decomposes, CO2 is evolved. This is an endothermic reaction; heat is absorbed and the sample temperature decreases. The sample is now at a lower temperature than the reference. The temperature difference between the sample and reference generates a net signal, which is recorded.

Applications of DTA

DTA is based on changes of heat flow into the sample

- Using DTA, we can detect the **decomposition or volatilization of the sample**, just as we can with TGA.
- In addition, however, **physical changes that do not involve weight changes can be detected** by DTA. Such changes include crystallization, melting, changes in solid crystal phases, and homogeneous reactions in the solid state. In each of these changes there is a **flow of heat** between the sample and its surroundings caused by **endothermic or exothermic transitions** or by changes in the heat capacity.
- The main use of DTA is to detect thermal processes and characterize them as **exothermic or endothermic, reversible or irreversible,** but only qualitatively.

summary

PROCESS	WEIGHT GAIN	WEIGHT LOSS
Ad- or absorption	✓	
Desorption, drying		✓
Dehydration, desolvation		✓
Sublimation		✓
Vaporisation		✓
Decomposition		✓
Solid-solid reactions (some)		✓
Solid-gas reactions	✓	✓
Magnetic transitions	✓	✓

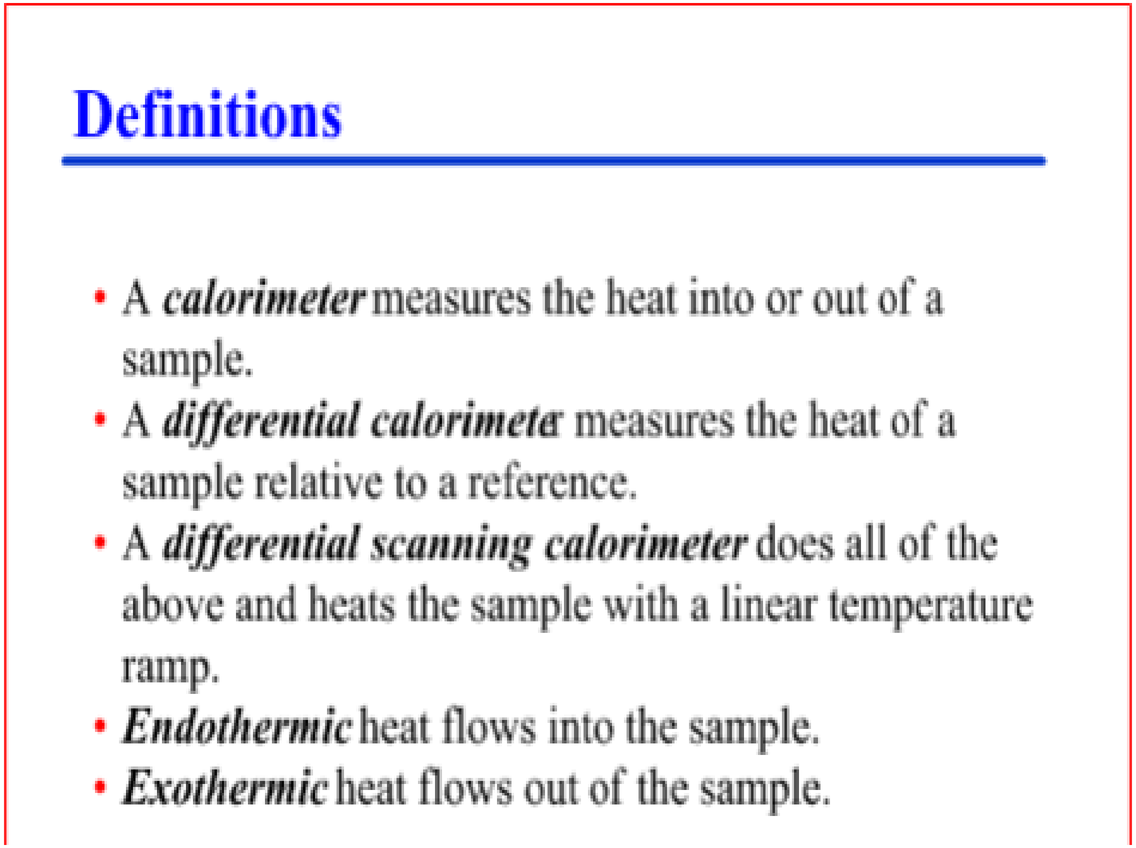
The sample

- Sample form, defect content, porosity and surface properties has influence to the behaviour on heating, e.g. single crystal sample give different response from powdered sample.

- Large sample size cause problems like heat transfer, and gas exchange with the surrounding is reduced; in general, the use of small (~ 20 mg) specimen is preferable if sensitivity of balance permits.

- Sample should be powdered and spread thinly and uniformly in the container.

Differential Scanning Calorimetry (DSC)



Definitions

- A ***calorimeter*** measures the heat into or out of a sample.
- A ***differential calorimeter*** measures the heat of a sample relative to a reference.
- A ***differential scanning calorimeter*** does all of the above and heats the sample with a linear temperature ramp.
- ***Endothermic*** heat flows into the sample.
- ***Exothermic*** heat flows out of the sample.

Basic Difference between DSC and DTA

1. One is based on the temperature difference while the other is based on the heat flow difference.

2. DSC is a technique in which the difference is calculated between the amount of heat needed (heat flow) to increase the temperature of the sample and the heat required to increase the temperature of the reference while DTA is a technique in which the difference is calculated between the temperatures required by the reference and the sample when the heat flow is kept the same for both.

3. DSC is an instrument based on the technique used to measure heat released or absorbed during the transition phase.

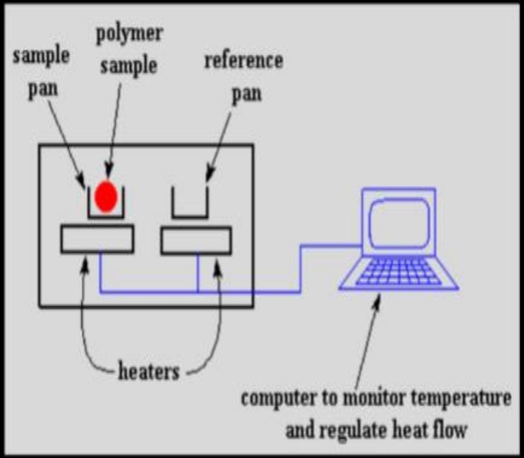
4. The experiment is designed in such a way that the temperature increases in a linear manner with time. During the phase transition, the heat required is either more or less than the reference depending upon the process being endothermic or exothermic.

5. The difference in heat flow between a reference and a sample helps the DSC to precisely measure the heat released or absorbed during the transition phase.

Principle

- The sample and reference are maintained at the same temperature, even during a thermal event in the sample
- The energy required to maintain zero temperature difference between the sample and the reference is measured
- During a thermal event in the sample, the system will transfer heat to or fro from the sample pan to maintain the same temperature in reference and sample pans

WHAT WE DO IN DSC?



- **There are two pans, In sample pan, polymer is added, while the other, reference pan is left empty**
- **Each pan sits on top of heaters which are controlled by a computer**
- **The computer turns on heaters, and let them heat the two pans at a specific rate, usually 10°C/min.**

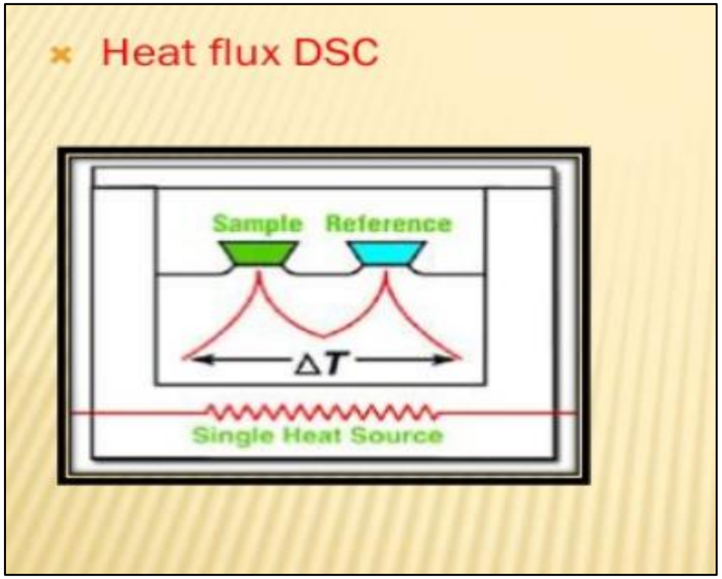
- **The computer makes absolutely sure that the heating rate stays exactly the same throughout the experiment**

Why heaters don't heat at the same rate?

- The simple reason is that the two pans are different. One has polymer in it, and one doesn't. The polymer sample means there is extra material in the sample pan. Having extra material means that it will take more heat to keep the temperature of the sample pan increasing at the same rate as the reference pan

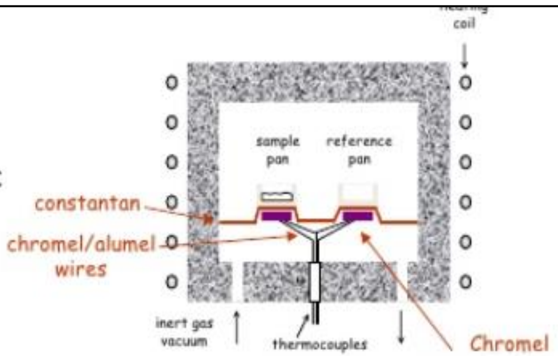
- So the heater underneath the sample pan has to work harder than the heater underneath the reference pan. It has to put out more heat. How much more heat it has to put out is what measured in DSC experiment?

- Specifically, a plot is drawn as the temperature increases. The temperature is taken on x-axis whist the difference in heat output of the two heaters at a given temperature on *y*-axis



- Heat flux DSC

Heat Flux DSC



Sample and reference holders

- Al or Pt pans placed on constantan disc
- Sample and reference holders are connected by a low-resistance heat flow path

Sensors

- **Chromel®** (an alloy made of 90% nickel and 10% chromium)**-constantan area thermocouples (differential heat flow)**
- **Chromel®-alumel** (an alloy consisting of approximately 95% nickel, 2% manganese, 2% aluminium and 1% silicon) **thermocouples (sample temperature)**

Thermocouple is a junction between two different metals that produces a voltage due to a temperature difference

Furnace

- One block for both sample and reference cells

Temperature controller

- The temperature difference between the sample and reference is converted to differential thermal power, which is supplied to the heaters to maintain the temperature of the sample and reference at the program value

---

DSC Calibration

Baseline

- Evaluation of the thermal resistance of the sample and reference sensors
- Measurements over the temperature range of interest

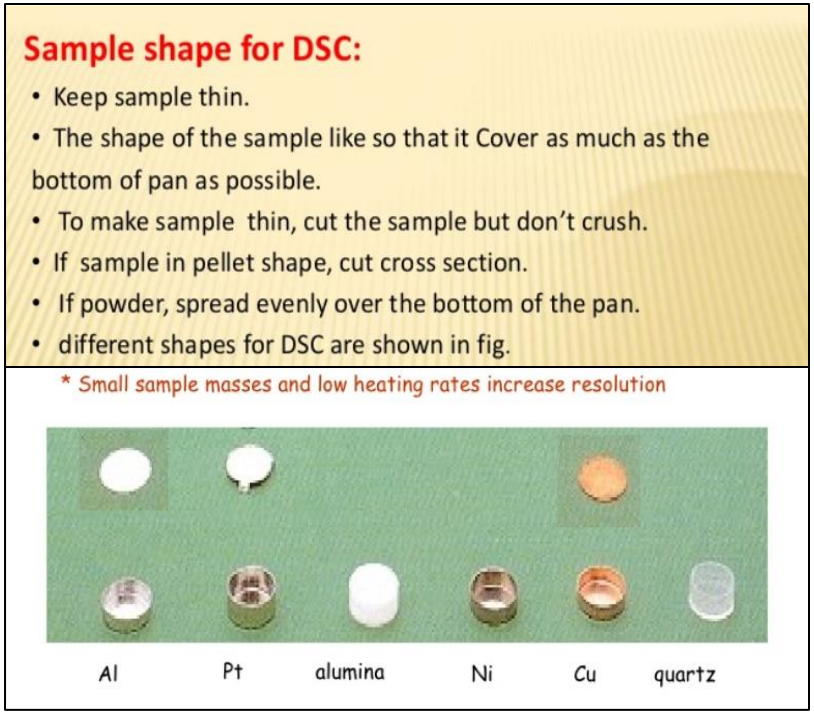
2-Step process

  - The temperature difference of two empty crucibles is measured
  - The thermal response is then acquired for a standard material, usually sapphire, on both the sample and reference platforms

- Amplified DSC signal is automatically varied with the temperature to maintain a constant calorimetric sensitivity with temperature

Sample preparation:

- Accurately-weigh samples (~3-20 mg)
- Small sample pans (0.1 mL) of inert or treated metals (Al, Pt, Ni, etc.) are used for sample preparation.
- Several pan configurations, e.g., open, pinhole, or hermetically-sealed (airtight) pans
- The same material and configuration should be used for the sample and the reference
- Material should completely cover the bottom of the pan to ensure good thermal contact,
- Avoid overfilling the pan to minimize thermal lag from the bulk of the material to the sensor



Sample shape for DSC:

- Keep sample thin.
- The shape of the sample like so that it Cover as much as the bottom of pan as possible.
- To make sample thin, cut the sample but don't crush.
- If sample in pellet shape, cut cross section.
- If powder, spread evenly over the bottom of the pan.
- different shapes for DSC are shown in fig.

* Small sample masses and low heating rates increase resolution

DSC Curve

- The result of a DSC experiment is a curve of heat flux versus temperature or versus time. There are two different conventions: exothermic reactions in the sample shown with a positive or negative peak, depending on the kind of technology used in the experiment.
- This curve can be used to calculate enthalpies of transitions, which is done by integrating the peak corresponding to a given transition. The enthalpy of transition can be expressed using equation: $\Delta H = KA$

- Where ΔH is the enthalpy of transition,
- K is the calorimetric constant,
- A is the area under the peak.
- The calorimetric constant varies from instrument to instrument, and can be determined by analyzing a well-characterized material of known enthalpies of transition.
- Area under the peak is directly proportional to heat absorbed or evolved by the reaction,
- height of the peak is directly proportional to rate of the reaction

Factors affecting DSC curve

Two types of factors effect the DSC curve

1-Instrumental factors

a- Furnace heating rate
b- Recording or chart speed
c- Furnace atmosphere
d- Geometry of sample holder/location of sensors
e- Sensitivity of the recoding system
f-Composition of sample containers

2-Sample characteristics

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

Determination of Heat Capacity

- DSC plot can be used to determine Heat Capacity. Suppose a polymer is being heated.

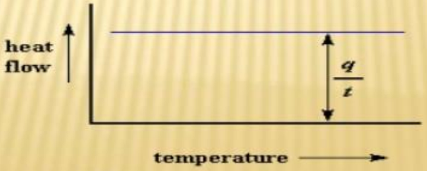
Difference in Heat output of two heaters

Heating two pans

against

temperature

- When we start heating two pans, the computer will plot the difference in heat output of the two heaters against temperature that is plot of heat absorbed by the polymer against temperature. The plot will look like this at first.



- **The heat flow is heat (*q*) supplied per unit time (*t*), whereas,**
- **The heating rate is temperature increase (Δ*T*) per unit time (*t*)**

$$\text{heat flow} = \frac{\text{heat}}{\text{time}} = \frac{q}{t}$$

$$\text{heating rate} = \frac{\text{temperature increase}}{\text{time}} = \frac{\Delta T}{t}$$

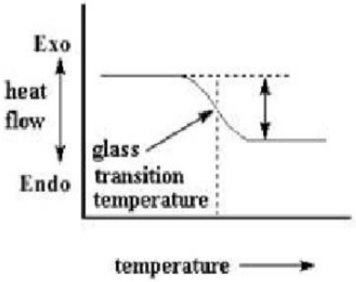
- By dividing heat flow *(q/t)* by the heating rate *(ΔT/t)*. It ends up with heat supplied divided by the temperature increase, which is called heat capacity.

$$\frac{\frac{q}{t}}{\frac{\Delta T}{t}} = \frac{q}{\Delta T} = C_p = \text{heat capacity}$$

- When a certain amount of heat is transferred to the sample, its temperature increases by a certain amount, and the amount of heat it takes to get a certain temperature increase is called the heat capacity, or C_p, it can be figured up from the DSC plot

The Glass Transition Temperature

- On further heating the polymer to a certain temperature, plot will shift downward suddenly, like this:

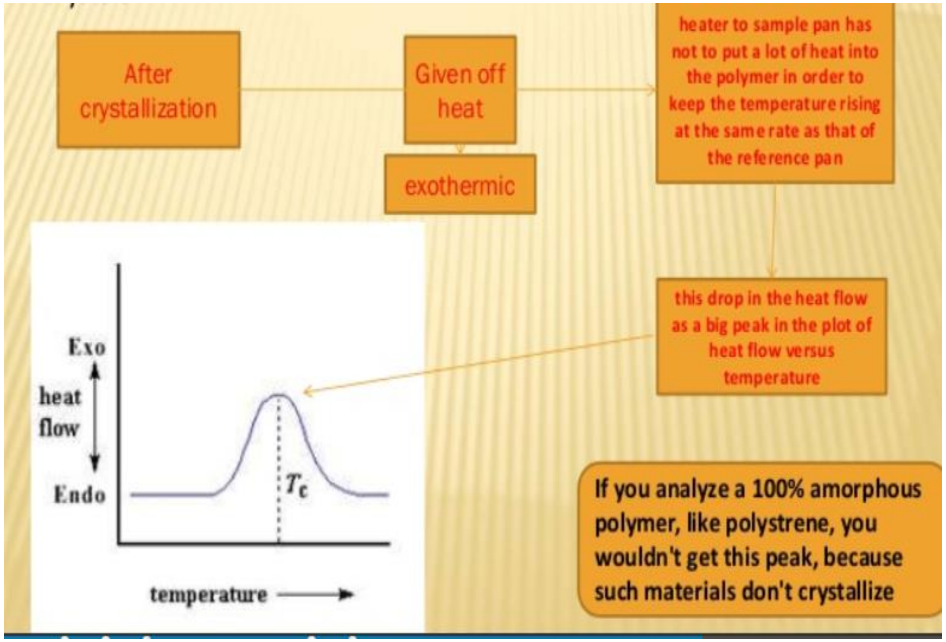


- This means there is more heat flow. There is an increase in the heat capacity of the polymer. This happens because the polymer has just gone through the glass transition. Because of this change in heat capacity that occurs at the glass transition, we can use DSC to measure a polymer's glass transition temperature.
- Take note that this change doesn't occur suddenly, but takes place over a temperature range. This makes picking one discreet *Tg* a kind of tricky matter, but we usually just take the middle of the incline to be the *Tg*.

Crystallization

- After glass transition, the polymers have a lot of mobility. They wiggle and squirm, and never stay in one position for very long time. But when they reach the right temperature, they will give off enough energy to move into very ordered arrangements, which is called crystals.

- When polymers fall into these crystalline arrangements, they give off heat. When this heat is dumped out, it makes the little computer-controlled heater under the sample pan really happy. It's happy because it doesn't have to put out much heat to keep the temperature of the sample pan rising. You can see this drop in the heat flow as a big peak in the plot of heat flow versus temperature



After crystallization

Given off heat

exothermic

heater to sample pan has not to put a lot of heat into the polymer in order to keep the temperature rising at the same rate as that of the reference pan

this drop in the heat flow as a big peak in the plot of heat flow versus temperature

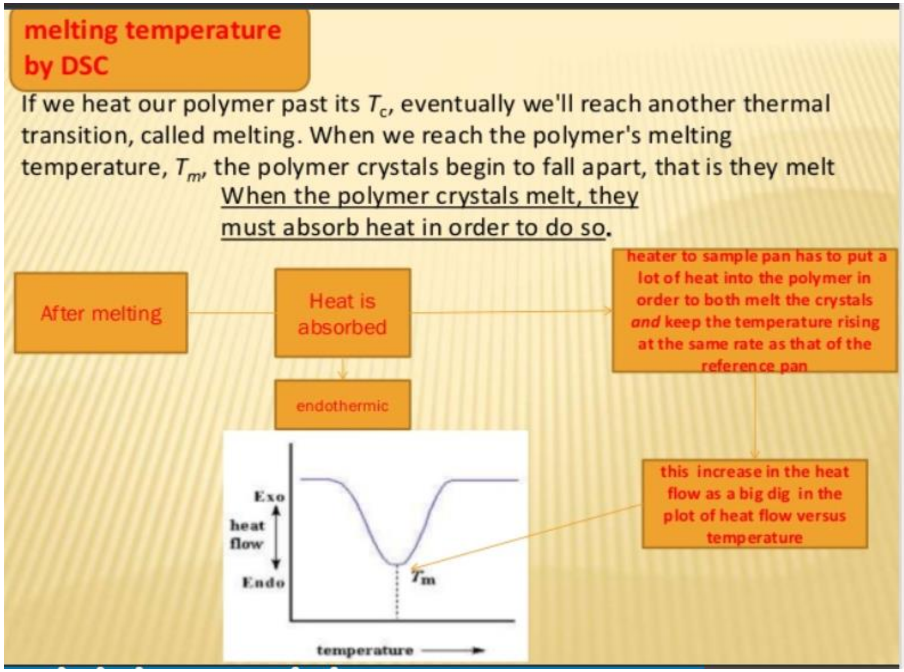
If you analyze a 100% amorphous polymer, like polystrene, you wouldn't get this peak, because such materials don't crystallize

- Also, we can measure the area of the peak, which tells us the latent energy of crystallization of the polymer
- But most importantly, this peak tells us that the polymer can in fact crystallize
- If you analyze a 100% amorphous polymer, like polystrene, you wouldn't get this peak, because such materials don't crystallize
- Also, because the polymer gives off heat when it crystallizes, we call crystallization is an *exothermic* transition

Melting

- If we heat our polymer past its T_c, eventually we'll reach another thermal transition, called melting. When we reach the polymer's melting temperature, T_m, the polymer crystals begin to fall apart, that is they melt. It comes out of their ordered arrangements, and begin to move around freely that can be spotted on a DSC plot

- Remember, the heat which polymer give off when crystallized is absorbed when reached at T_m. That is a latent heat of melting like latent heat of crystallization. When the polymer crystals melt, they must absorb heat in order to do so. Remember melting is a first order transition. This means that at the melting temperature, the polymer's temperature won't rise until all the crystals have melted.



melting temperature by DSC

If we heat our polymer past its T_c, eventually we'll reach another thermal transition, called melting. When we reach the polymer's melting temperature, T_m, the polymer crystals begin to fall apart, that is they melt

When the polymer crystals melt, they must absorb heat in order to do so.

After melting

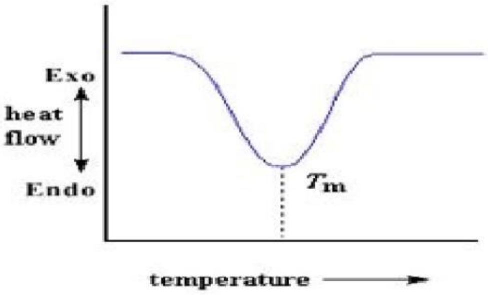
Heat is absorbed

endothermic

heater to sample pan has to put a lot of heat into the polymer in order to both melt the crystals *and* keep the temperature rising at the same rate as that of the reference pan

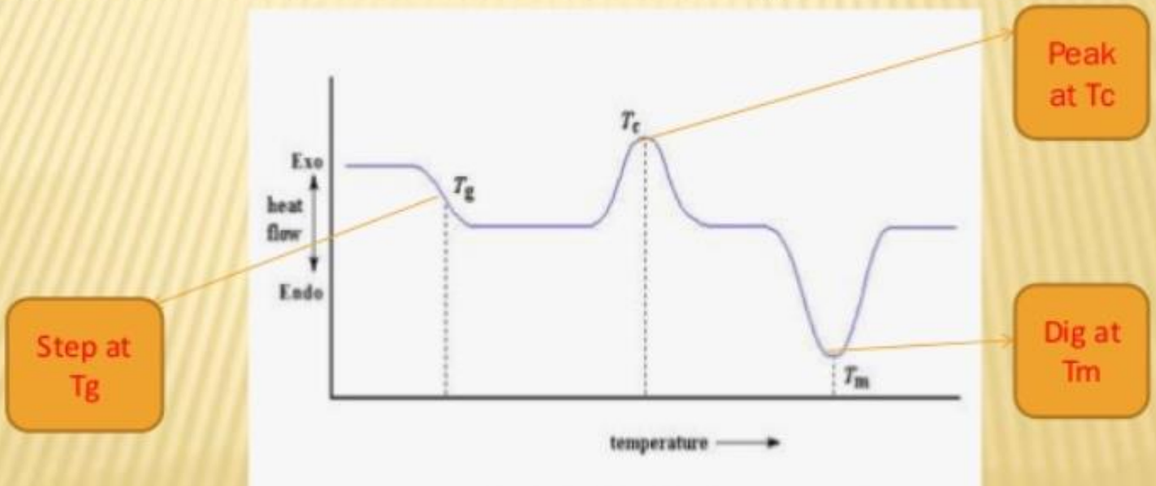
this increase in the heat flow as a big dig in the plot of heat flow versus temperature

This means that the little heater under the sample pan has to put a lot of heat into the polymer in order to both melt the crystals *and* keep the temperature rising at the same rate as that of the reference pan. This extra heat flow during melting shows up as a big dip on DSC plot, like this:



Putting It All Together

we saw a step in the plot when the polymer was heated past its glass transition temperature. Then we saw a big peak when the polymer reached its crystallization temperature. Then finally we saw a big dip when the polymer reached its melting temperature. To put them all together, a whole plot will often look something like this:

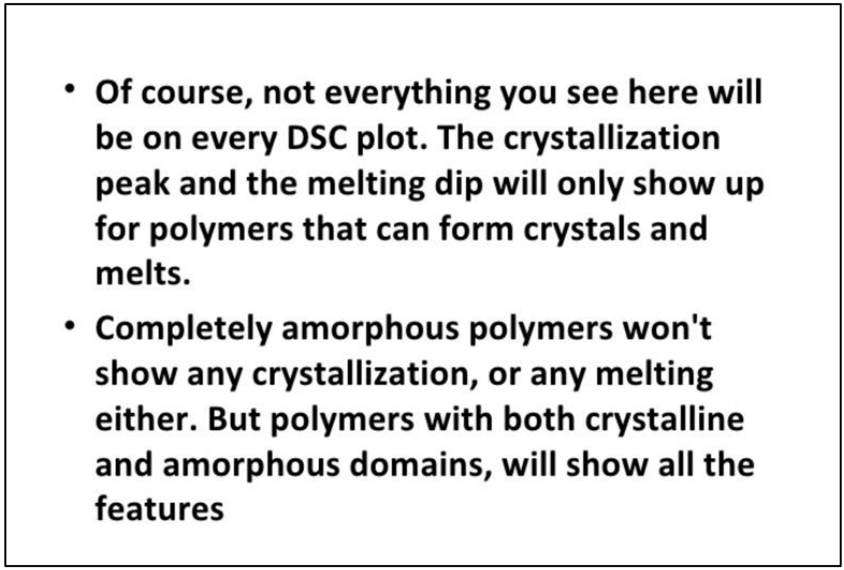


Peak at Tc

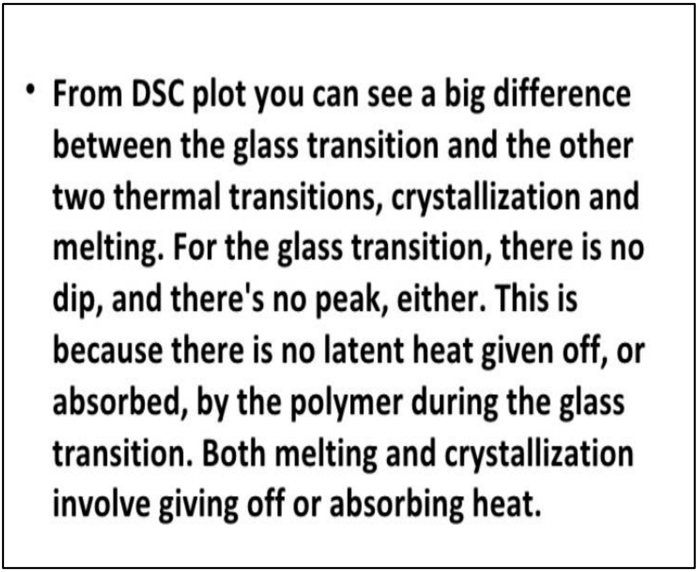
Step at Tg

Dig at Tm

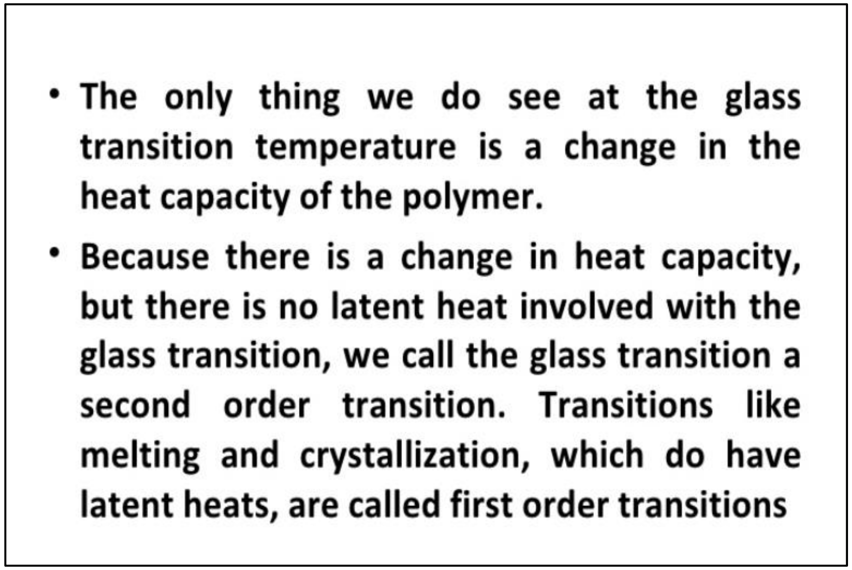
The entire DSC plot, right before your very eyes!



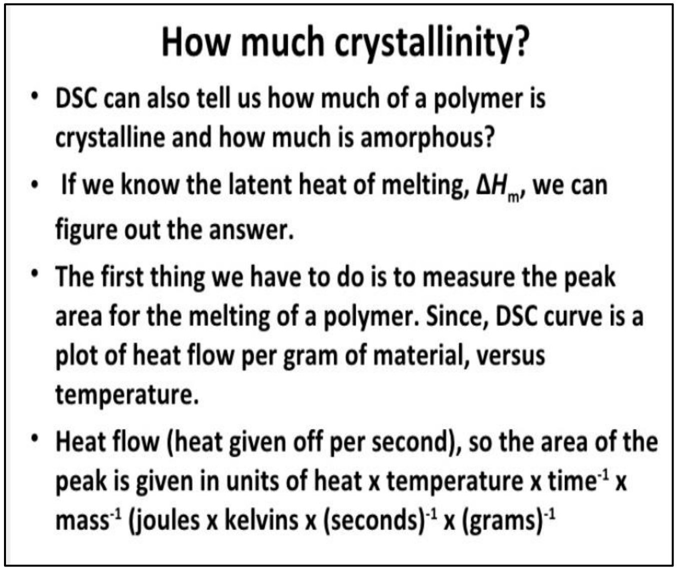
- Of course, not everything you see here will be on every DSC plot. The crystallization peak and the melting dip will only show up for polymers that can form crystals and melts.
- Completely amorphous polymers won't show any crystallization, or any melting either. But polymers with both crystalline and amorphous domains, will show all the features



- From DSC plot you can see a big difference between the glass transition and the other two thermal transitions, crystallization and melting. For the glass transition, there is no dip, and there's no peak, either. This is because there is no latent heat given off, or absorbed, by the polymer during the glass transition. Both melting and crystallization involve giving off or absorbing heat.

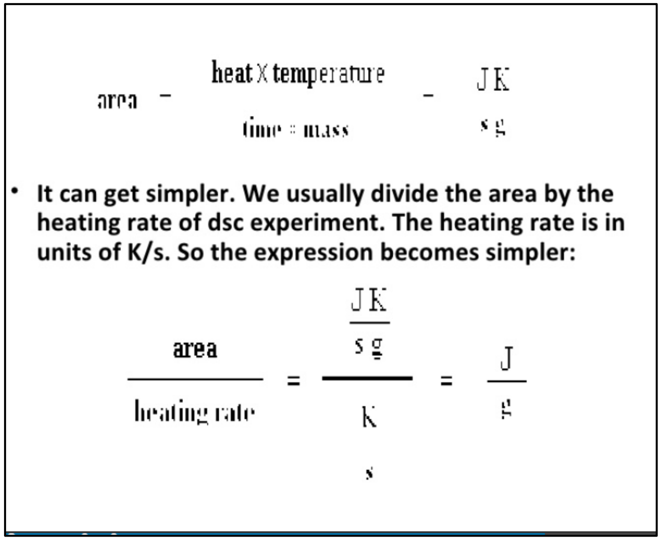


- The only thing we do see at the glass transition temperature is a change in the heat capacity of the polymer.
- Because there is a change in heat capacity, but there is no latent heat involved with the glass transition, we call the glass transition a second order transition. Transitions like melting and crystallization, which do have latent heats, are called first order transitions



How much crystallinity?

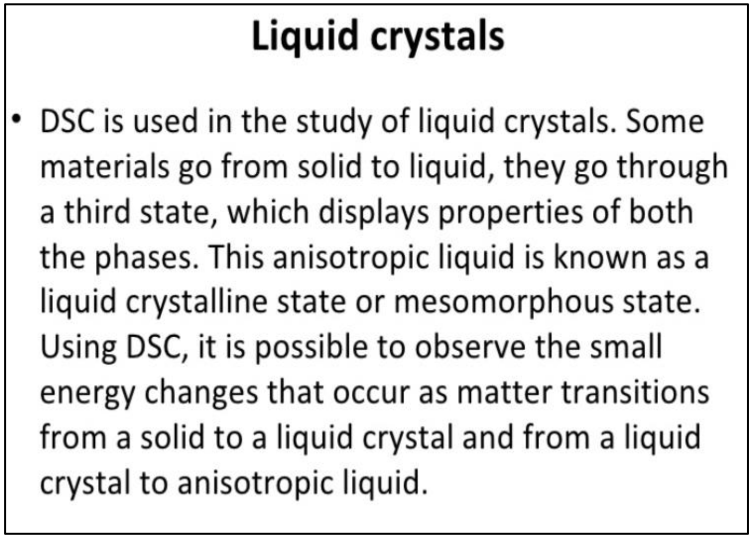
- DSC can also tell us how much of a polymer is crystalline and how much is amorphous?
- If we know the latent heat of melting, ΔH_m, we can figure out the answer.
- The first thing we have to do is to measure the peak area for the melting of a polymer. Since, DSC curve is a plot of heat flow per gram of material, versus temperature.
- Heat flow (heat given off per second), so the area of the peak is given in units of heat x temperature x time^{-1} x mass^{-1} (joules x kelvins x (seconds)$^{-1}$ x (grams)$^{-1}$



$$\text{area} = \frac{\text{heat} \times \text{temperature}}{\text{time} \times \text{mass}} = \frac{\text{J K}}{\text{s g}}$$

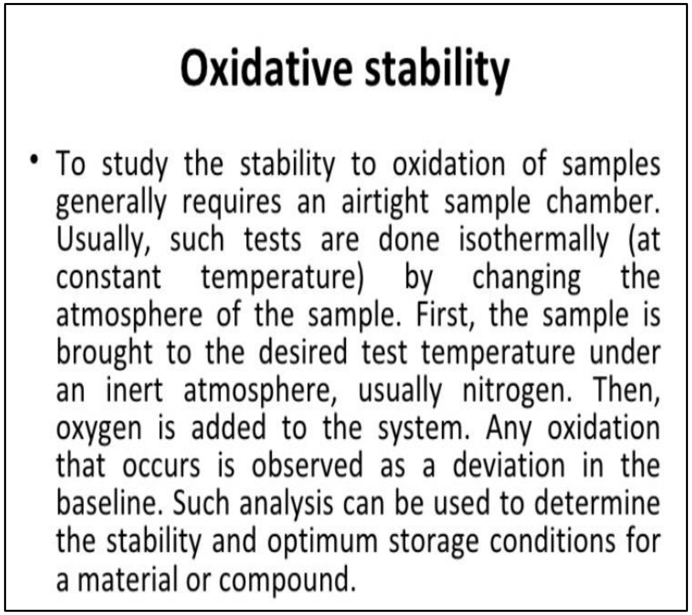
- **It can get simpler. We usually divide the area by the heating rate of dsc experiment. The heating rate is in units of K/s. So the expression becomes simpler:**

$$\frac{\textbf{area}}{\text{heating rate}} = \frac{\frac{\text{J K}}{\text{s g}}}{\frac{\text{K}}{\text{s}}} = \frac{\text{J}}{\text{g}}$$



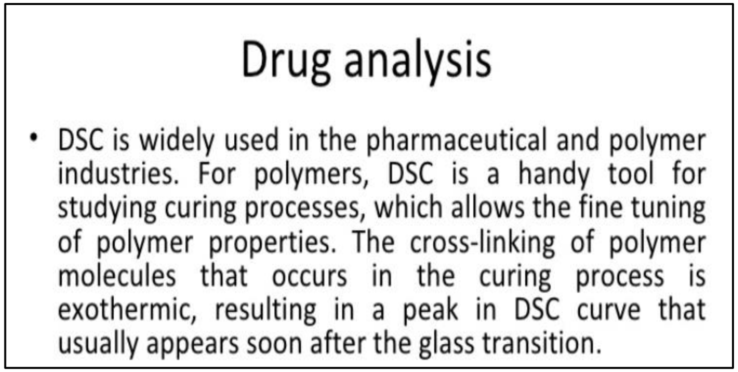
Liquid crystals

- DSC is used in the study of liquid crystals. Some materials go from solid to liquid, they go through a third state, which displays properties of both the phases. This anisotropic liquid is known as a liquid crystalline state or mesomorphous state. Using DSC, it is possible to observe the small energy changes that occur as matter transitions from a solid to a liquid crystal and from a liquid crystal to anisotropic liquid.



Oxidative stability

- To study the stability to oxidation of samples generally requires an airtight sample chamber. Usually, such tests are done isothermally (at constant temperature) by changing the atmosphere of the sample. First, the sample is brought to the desired test temperature under an inert atmosphere, usually nitrogen. Then, oxygen is added to the system. Any oxidation that occurs is observed as a deviation in the baseline. Such analysis can be used to determine the stability and optimum storage conditions for a material or compound.



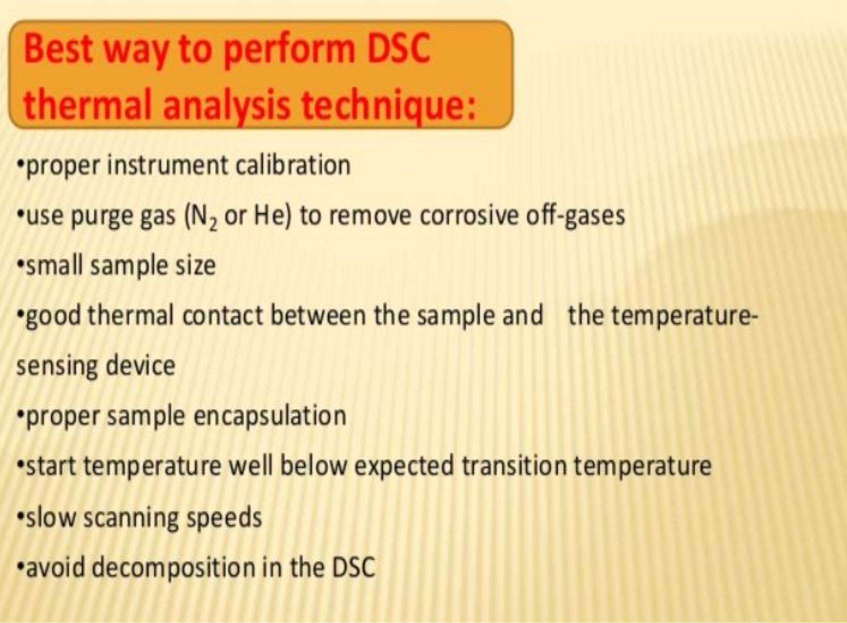
Drug analysis

- DSC is widely used in the pharmaceutical and polymer industries. For polymers, DSC is a handy tool for studying curing processes, which allows the fine tuning of polymer properties. The cross-linking of polymer molecules that occurs in the curing process is exothermic, resulting in a peak in DSC curve that usually appears soon after the glass transition.

Curing is a chemical process employed in polymer chemistry and process engineering that produces the toughening or hardening of a polymer material by cross-linking of polymer chains. ... Curing can be effected by heat, radiation, electron beams, or chemical additives.

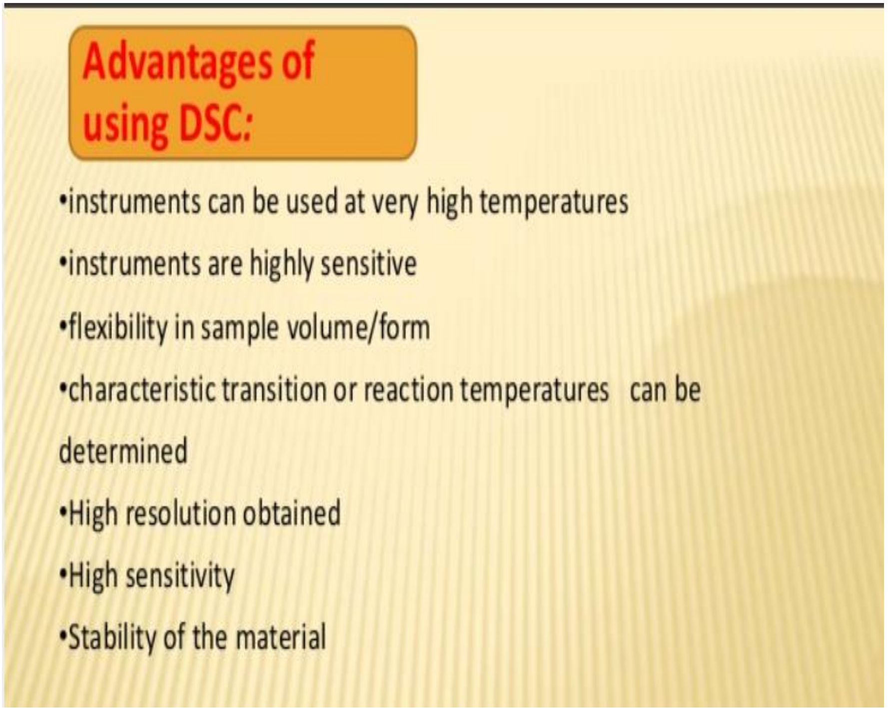
General chemical analysis

- Melting-point depression can be used as a **purity analysis** tool when analysed by Differential scanning calorimetry. This is possible because the temperature range over which a mixture of compounds melts is dependent on their relative amounts. Consequently, less pure compounds will exhibit a broadened melting dip that begins at lower temperature than a pure compound.



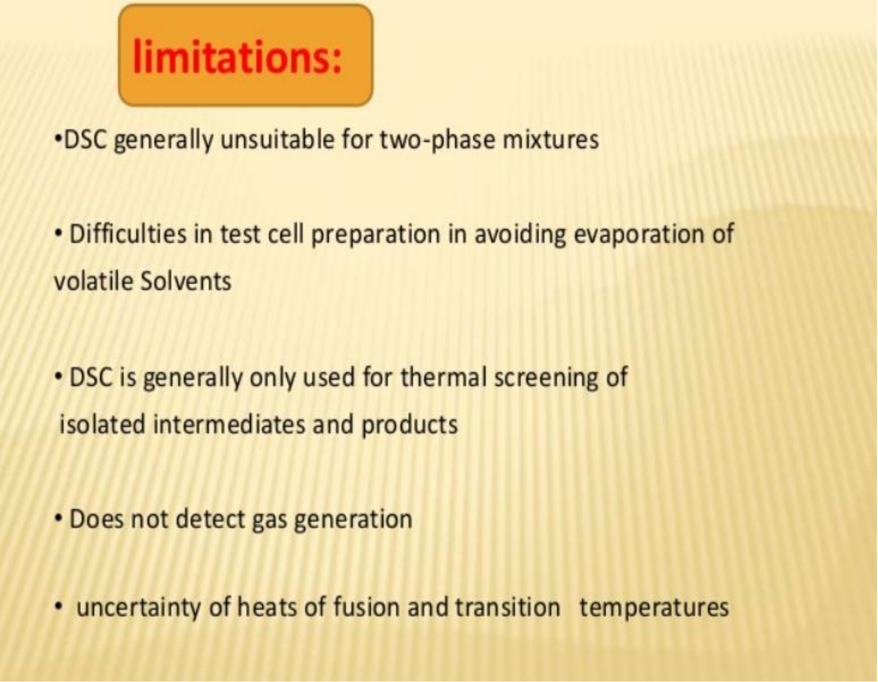
Best way to perform DSC thermal analysis technique:

- proper instrument calibration
- use purge gas (N_2 or He) to remove corrosive off-gases
- small sample size
- good thermal contact between the sample and the temperature-sensing device
- proper sample encapsulation
- start temperature well below expected transition temperature
- slow scanning speeds
- avoid decomposition in the DSC



Advantages of using DSC:

- instruments can be used at very high temperatures
- instruments are highly sensitive
- flexibility in sample volume/form
- characteristic transition or reaction temperatures can be determined
- High resolution obtained
- High sensitivity
- Stability of the material



limitations:

- DSC generally unsuitable for two-phase mixtures
- Difficulties in test cell preparation in avoiding evaporation of volatile Solvents
- DSC is generally only used for thermal screening of isolated intermediates and products
- Does not detect gas generation
- uncertainty of heats of fusion and transition temperatures