

Topic

**DIFFERENTIAL THERMAL
ANALYSIS**

Definition:

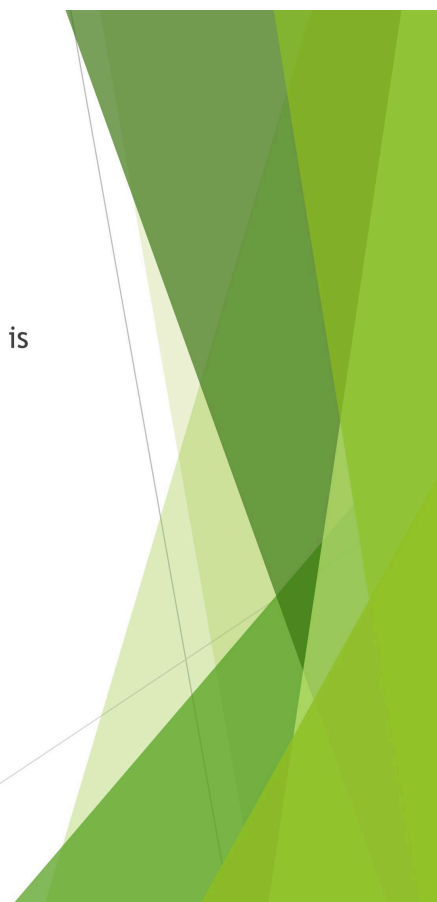
Differential thermal analysis (DTA):

Differential thermal analysis is a thermal analysis technique in which the temperature of the substance under investigation is compared with the temperature of a thermally inert material and is recorded with furnace as the substance is heated or cooled at a determined uniform rate.



Principle:

The Basic principle involved in DTA is the temperature difference (T) between the test sample and an inert reference sample under controlled and identical conditions of heating or cooling is recorded continuously as a function of temperature or time, thus the heat absorbed or emitted by a chemical system is determined



Working of DTA:

- ▶ The sample and reference standard are placed in the furnace on flat, highly thermally conductive pans and the thermocouples are physically attached to the pans directly under the sample
- ▶ This procedure avoids or reduces any thermal lag resulting from the time required for the heat to transfer to the sample and reference material then to the thermocouples.
- ▶ The thermocouples are connected in opposition.
- ▶ In a similar manner any change in state that involves a latent heat of transition will cause the temperature of the sample to lag or lead that of the reference standard and identify the change of state and the temperature at which it occurred.

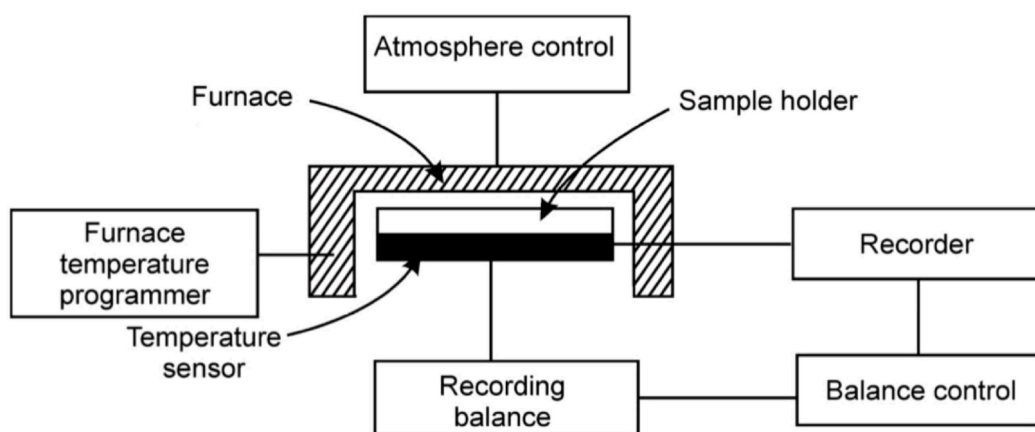


Instrumentation for DAT/Block Diagram

The DAT apparatus consists of the following components:

- ▶ Furnace sample and reference holder with thermocouple assembly.
- ▶ Sample holder furnace (To heat the sample).
- ▶ Furnace temperature controller(to increase the furnace temperature steadily)
- ▶ Furnace atmospheric control system (To maintain a suitable atmosphere in the furnace and sample holder)
- ▶ Low level DC amplifier
- ▶ Recording device(Recorder)
- ▶ Differential temperature sensor (to measure the temperature difference between the sample and reference material) the sample and reference holder are kept inside the furnace and the temperature of the furnace and sample holder is controlled by using furnace controller.

Block Diagram:



Factors affecting the DTA Curve:

- ▶ Instrumental Factors.
- ▶ Size and shape of the sample and furnace holder.
- ▶ Material from which sample holder is made and its corrosive attack.
- ▶ Heating rate(furnace heating rate).
- ▶ Sample characteristics:
- ▶ Amount of the sample(sample weight)
- ▶ Particle size of the sample

Factors that influence DTA Curve:

Factor	Effect	Suggestions
1. Heating rate	Change in peak size and position	Use low heating rate
2. Location of thermocouple	Irreproducible curve	Standardise thermocouple
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

Application of DTA:

- ▶ DTA curves for two substances are not identical. Hence they serve as finger prints for various substance.
- ▶ Used to study the characteristic of polymeric material.
- ▶ This technique is used for testing the purity of the drug sample and also to test the quality control of number of substances like cement, soil, glass, etc.
- ▶ Used for the determination of heat of reaction, specific heat and energy change occurring during melting etc.
- ▶ Trend in ligand stability (thermal stability of the ligands) gives the information about the ligands in the coordination sphere.

Advantage:

- ▶ Instruments can be used at high temperatures
- ▶ Instruments are highly sensitive
- ▶ Characteristic transition or reaction temperature can be accurately determined
- ▶ Uncertainty of heats of fusion, transition, or reaction estimation is 20-50%

Disadvantage:

- ▶ Uncertainty of heats of fusion, transition, or reaction estimation is 20-50%
- ▶ Reaction or transition estimations is only 20% to 50% DTA

THANK YOU

