

INSTRUMENTAL METHODS OF ANALYSIS

- Principle and application of Thermal methods of Analysis. (TGA, DTA, DSC),
- Basic concepts of spectroscopy, Lambert and Beers law, Absorption and Emission spectroscopy
Different spectroscopic Techniques (UV- Visible and IR spectroscopy)
- Flame photometry.

Analytical chemistry deals with the qualitative and quantitative methods of analysis of compound or molecule in precise and accurate way. Some analytical methods are

PNMR

ESR

Electron diffraction

X-ray diffraction

Mass spectroscopy

UV-Visible and IR spectroscopy, etc

THERMAL METHODS OF ANALYSIS

An instrumental technique is regarded as a thermal analysis method ,if the physical parameters are taken as a function of temperature or time.

Some thermal methods are

TGA

DTA

DSC

TGA

Introduction-It is a technique where weight of the substance ,in an environment ,heated or cooled at a controlled rate is recorded as a function of time or temperature.

- a) Isothermal or static -Sample weight is recorded as a function of time at constant temperature.
- b) Dynamic TGA-Sample is heated in an environment ,whose temperature is changing in a predetermined manner, generally at linear rate.

RECORDING OF RESULT

It is a precision balance for linear rise of temperature and known as **thermobalance**.

Result of thermobalance-Result of thermobalance are represented by plot of weight change v/s temp. or time, which is known as TG curve.

Weight should plotted on Y-axis, and temp. or time on X-axis, increasing from left to right.

DIFFERENTIAL THERMAL ANALYSIS

Principle: This technique involve the recording of difference in temperature between substance and reference material against time or temperature ,as the two specimens are subjected to identical temperature change in an enviroment heated or cooled at the controlled rate.

Recording of result:DTA thermogram consist of record of difference in sample and reference temperature plotted as a function of Time,or sample temperature or reference temperature or furnace temperature.

Information from DTA curve:

- 1) There are exothermic and endothermic peaks and shape and size of these peaks may furnish good information about the nature of test sample.
- 2) In most of the cases physical changes give endothermic while chemical changes gives exothermic peaks.
- 3) Generally sharp endothermic peaks gives good idea about crystallinity and fusion process, while broad endothermic peaks signify dehydration reaction.
- 4) The DTA curve would be parallel to the temperature axis till the sample undergoes any physical or chemical change of state.

INSTRUMENTATION

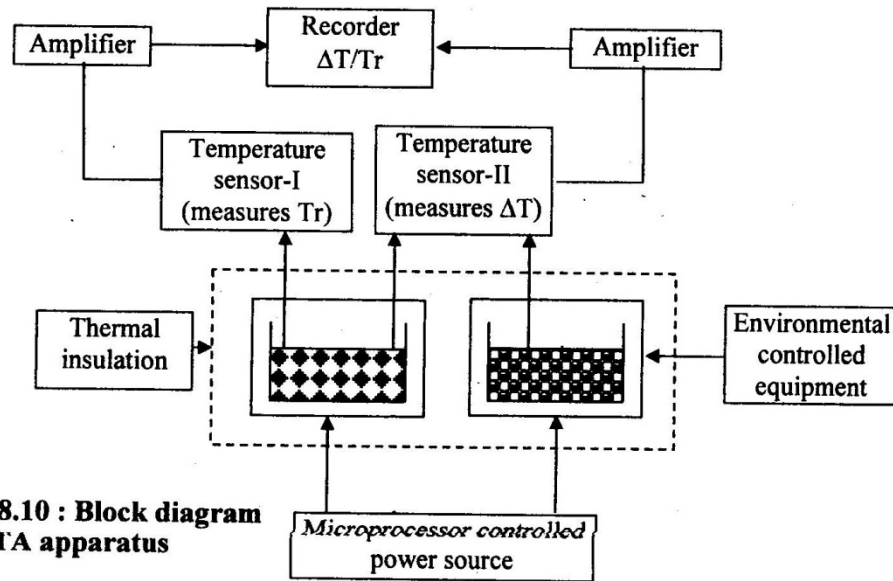


Fig. 8.10 : Block diagram of DTA apparatus

APPLICATION

Applications in

1)Physical Chemistry

Generation of phase diagram and study of phase transitions

In determination of heat of reaction.

In specific heat determination.

In determination of thermal diffusivity.

2)Analytical Chemistry

Identification of substance

Identification of products

Melting point and boiling determination and study of effect of pressure on it.

Quantitative analysis

Quality control

3)Inorganic Chemistry:To study thermal stability of inorganic compounds and complexes

4)Organic Chemistry

Qualitative analysis

For identification and purity determination for oils fats and pharmaceutical substances.

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Principle: In this technique the heat energy is supplied at the varying rate to the sample or reference so as to keep their temperature equal and this heat energy is recorded as a function of temperature or time when both substance and reference material are heated or cooled at a predetermined rate. (approx. 10 degree celcius per minute) alternatively

It is a thermal method where energy necessary to establish zero temperature difference between substance and reference material is recorded as a function of time or temp., when both are heated or cooled at a predetermined rate.

DSC THERMOGRAM

- 1) The heat supplied to sample is given a “positive sign” while reference material is given a “negative sign”
- 2) The DSC curve is a record of transition temperature on x-axis and dH/dt on y-axis.
- 3) The DSC thermogram is similar to DTA curve only difference that dH/dt instead of ΔT (of DTA)
-) The area under DSE peak is directly be related to enthalpy change occurring.

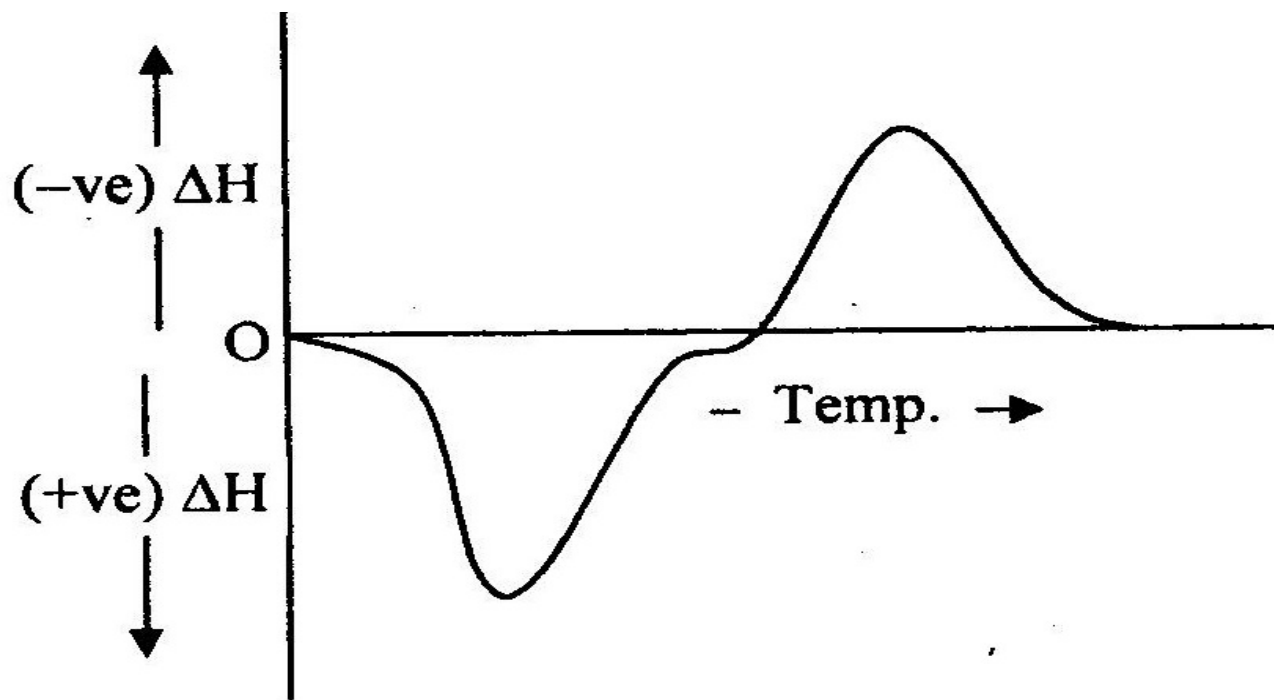


Fig. 8.11. DSC Curve

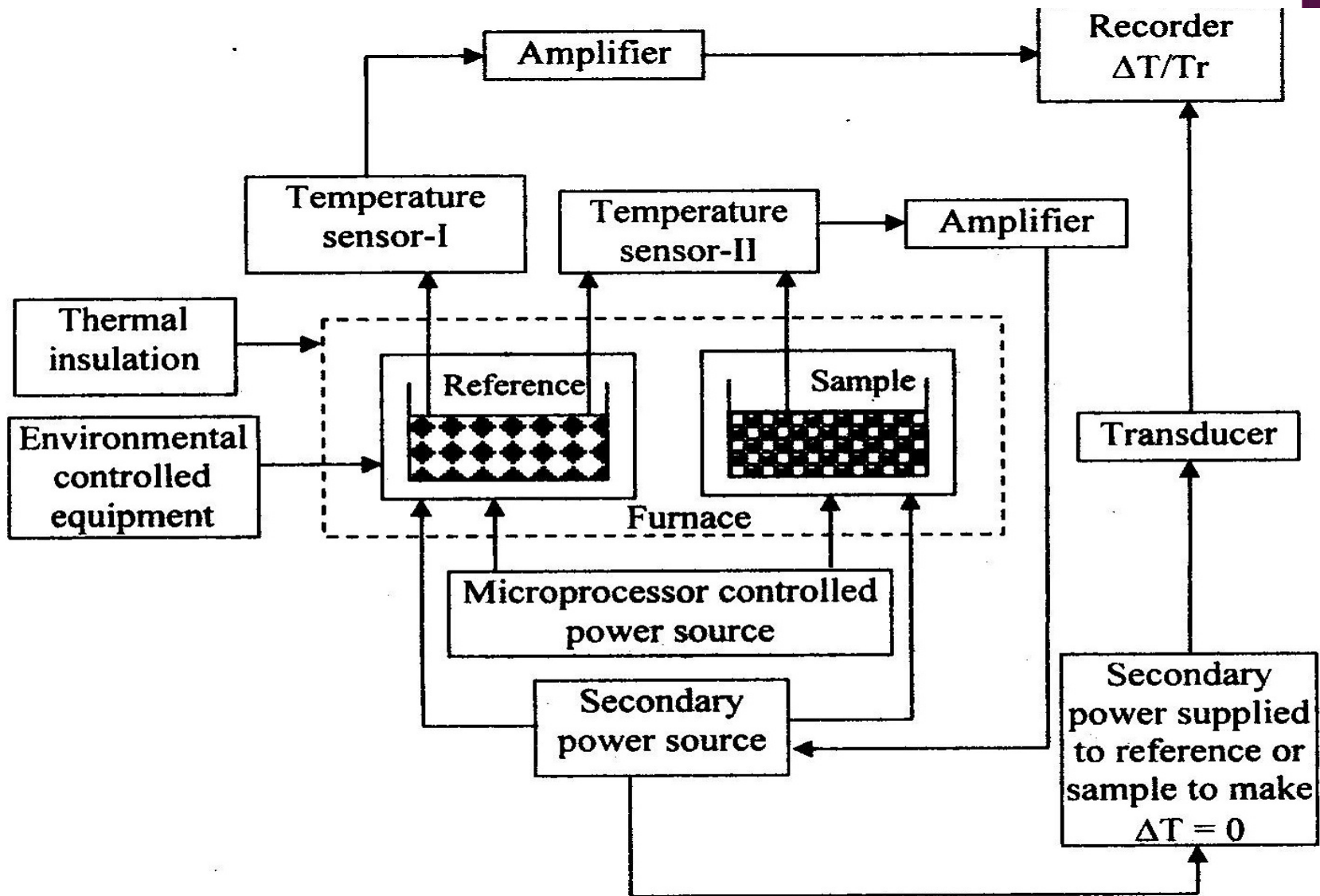


Fig. 8.12 : Block diagram of the DSC apparatus

APPLICATIONS OF DSC

- 1) Determination of enthalpy of transition.
- 2) Determination of percentage crystallinity of polymeric material.
- 3) Determination of purity of drug samples of drug phenacetin.