Name of Unit:	Thermal Methods of Analysis and X-Ray Diffraction Methods
Course/Subject Name:	Advanced Instrumentation Techniques
Course/Subject Code:	BP811ET
Class: B. Pharm. Semester	VIII
Faculty:	Ms. Manpreet Kaur
Email id:	manipreet663@gmail.com
Mobile No.	9592378971

Learning Outcome of Module-II

LO	**Learning Outcome (LO)**	**Course Outcome Code**
LO1	To understand the basic concept of Thermal Methods of Analysis	BP811.1
LO2	To know about the components of a TGA, DSC, DTA instrument	BP811.3
LO3	To understand about the DSC, DTA and TGA curves and the factors affecting the curves.	BP811.3
LO4	To understand the basic concept and principle of X-Ray Diffraction and types of X-Ray Diffraction methods.	BP811.1, BP811.2

TABLE OF CONTENT

Topic

THERMAL METHODS OF ANALYSIS

Introduction

Thermoanalytical methods essentially encompass such techniques that are based entirely on the concept of heating a sample followed by well-defined modified procedures, such as : gravimetric analysis, differential analysis and titrimetric analysis. In usual practice, data are generated as a result of continuously recorded curves that may be considered as 'thermal spectra'. These thermal spectra also termed as 'thermograms, often characterize a single or multicomponent system in terms of

(a) Temperature dependencies of its thermodynamic properties, and

(b) Physicochemical reaction kinetics.

Broadly speaking the thermoanalytical methods are normally classified into the following three categories, namely :

(i) Thermogravimetric Analysis (TGA),

(ii) Differential Thermal Analysis (DTA), and

(iii) Thermometric Titrations.

All the above mentioned techniques shall be discussed briefly with specific reference to their theory, instrumentation, methodology and applications wherever necessary.

DIFFERENTIAL THERMAL ANALYSIS (DTA)

The difference of temperature between the sample under estimation and a thermally-inert reference material is continuously recorded as a function of furnace temperature in differential thermal analysis (DTA).

Differential Thermal Analysis (DTA)

1. Theory

The difference of temperature between the sample under estimation and a thermally-inert reference material is continuously recorded as a function of furnace temperature in differential thermal analysis (DTA). In actual practice both TGA and DTA are regarded as complementary techniques whereby information gathered by the usage of one approach is invariably supplemented and enhanced by the application of the other method. The range of phenomena measurable during a DTA-run is found to be much larger than in a TGA-run.

It is pertinent to mention here that in the course of TGA many vital processes, for instance : crystalli-zation, crystalline transitions, pure fusion reactions, glass transitions, and solid-state reactions devoid of vola-tile components might not be indicated as they happen to cause little change in weight of the sample. TGA invariably describes with ample precision the stoichiometry related to chemical changes that are indi-cated during DTA by an endothermal or exothermal duration from the base-line.

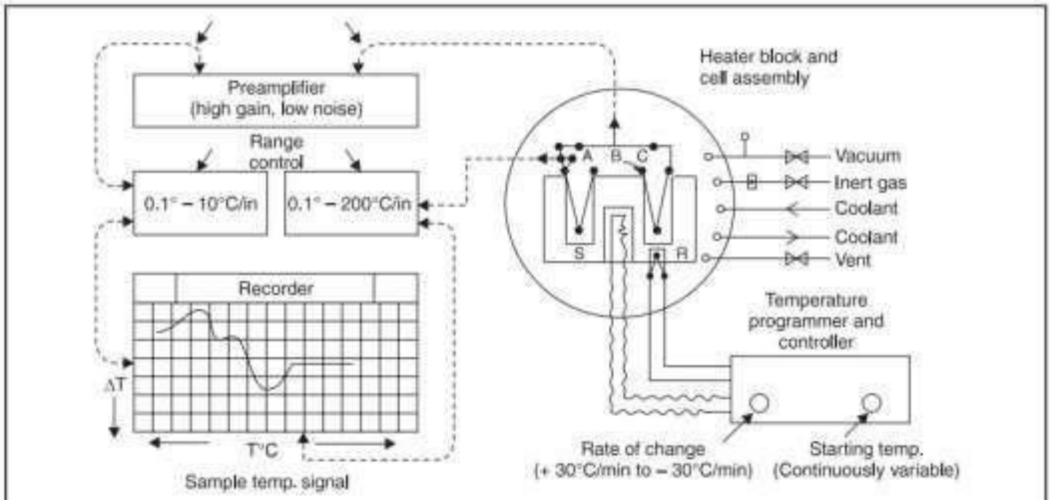


Figure 11.3 : Schematic Diagram of a Differential Thermal Analyzer.
(*Source :* E.I. Du Pont De Nemours, Inc, USA)

2. Instrumentation

A differential thermal analyzer is composed of five basic components, namely :

(i) Sample holder with built-in thermocouple assembly,
(ii) Flow-control system,
(iii) Furnace assembly,
(iv) Preamplifier and Recorder, and
(v) Furnace Power Programmer and Controller.

A typical commercial differential thermal analyzer is schematically illustrated in Figure 11.3.

(a) Thermocouples employed are normally unsheathed Platinum Vs Platinum and Sodium Vs 10% Rhodium. The said two thermocouples help in measuring the difference in temperature between a sample S and an absolutely inert reference substance R, as both are subjected to heating in a ceramic or metal block inside a furnace being operated by a temperature programmer and controller.

(b) The output of the differential thermocouple is amplified adequately through a high gain, low-noise preamplifier and subsequently hooked to the recorder, one axis of which is driven by the block temperature signal and is measured by a third thermocouple.

(c) Heating/Cooling Device : A sufficient versatility is achieved by the aid of a pressure-vacuum, high-temperature electric furnace. An almost constant heating rate is usually achieved by using a motor-driven variable auto transformer.

Both heating rates and cooling rates may be conveniently adjusted continuously :

(i) From 0°-30°C/minute by some instruments, and

(ii) From a choice of several commonly employed heating rates viz., 2°, 4°, 8° and 16°C/minute.

Usual workable sample temperatures are upto : 500°C. Exceptional maximum temperatures are upto : 1000°C.

(d) Relatively small sample volumes help in two ways : first, they make evacuation easy ; and secondly, they minimize thermal gradients.

Methodology

1. Insert a very thin thermocouple into a disposable sample tube 2 mm in diameter and containing 0.1-10 mg of sample,
2. Another identical tube is either kept empty or filled with a reference substance, such as quartz, sand, alumina or alundum powder,
3. The two tubes are simultaneously inserted into the sample block and subsequently heated (or cooled) at a uniform predetermined programmed rate, and
4. DTA—being a dynamic process, it is extremely important that all aspects of the technique must be thoroughly standardised so as to obtain reproducible results. A few of these aspects vital aspects are :

- Pretreatment of the specimen,
- Particle size and packing of the specimen,
- Dilution of the specimen,
- Nature of the inert diluent,
- Crystalline substances must be powdered, and sieved through 100-mesh sieve,
- For colloidal particles (e.g., clays), micelle-size is very critical, and Either to supress an unwanted reaction (e.g., oxidation), or to explore the study of a reaction (e.g., gaseous reaction product)—the atmosphere should be controlled adequately.

Figure 11.4, depicts the differential thermal analysis investigation of calcium acetate monohydrate at a uniform programmed heating rate of 12°C/minute..

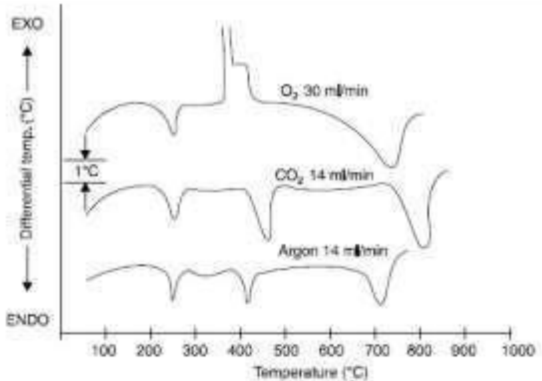


Figure 11.4 : DTA-Investigation of Calcium Acetate Monohydrate, 12°C. min^{-1} (After-American Inst. Co. Inc., USA, Bulletin).

The chemical reactions involved in the differentiated thermal analysis of calcium acetate monohydrate may be expressed as follows :

$$Ca(CH_3COO)_2.H_2O \xrightleftharpoons[\text{(Stage-I)}]{200\text{-}250°C} Ca(CH_3COO)_2 \xrightleftharpoons[\text{(Stage-II)}]{350\text{-}400°C} CaCO_3 \xrightleftharpoons[\text{(Stage-III)}]{800\text{-}950°C} CaO$$

Stage I : The endothermal dehydration of calcium acetate monohydrate occurs giving rise to the anhydrous salt. It is easily noticed by an endothermal band on DTA curve between 200°C and 250°C.

Stage II : The anhydrous salt undergoes endothermal decomposition reaction at 350-400°C resulting into the formation of $CaCO_3$. It has been observed that this decomposition reaction seems to be almost alike in the presence of either CO_2 or Ar.

Stage III : The decomposition of calcium carbonate to calcium oxide, which is a function of the partial pressure of the CO2 in contact with the sample. The endothermal band for the carbonate decomposition is sharply peaked spread over a relatively narrower temperature range in an atmosphere of CO2.

3. Applications

The various important applications of DTA are :

(i) Rapid identification of the compositions of mixed clays,

(ii) Studying the thermal stabilities of inorganic compounds,

(iii) Critically examining in a specific reaction whether a new compound is actually formed orthe product is nothing but an unreacted original substance, and

(iv) DTA offers a wide spectrum of useful investigations related to reaction kinetics, polymerization, solvent retention, phase-transformations, solid-phase reactions and curing or drying properties of a product.

THERMOGRAVIMETRIC ANALYSIS (TGA)

A large number of chemical substances invariably decompose upon heating, and this idea of heating a sample to observe weight changes is the underlying principle of thermogravimetric analysis (TGA).

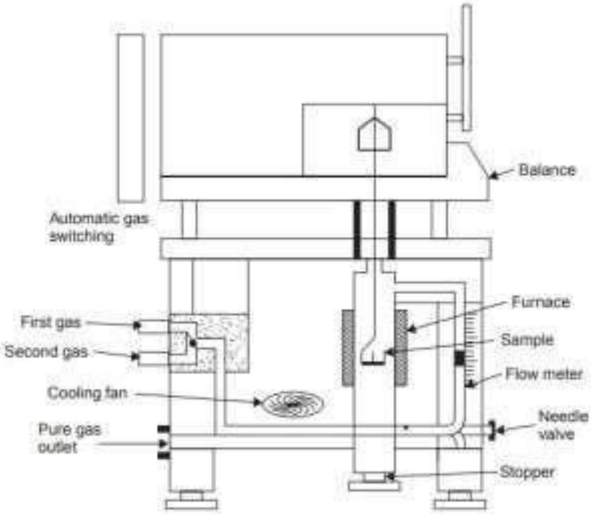


Figure 11.1 : Thermobalance for Thermogravimetric Analysis in a Controlled Atmosphere upto 1000°C.

Thermogravimetric Analysis (TGA)

1. Theory

A large number of chemical substances invariably decompose upon heating, and this idea of heating a sample to observe weight changes is the underlying principle of thermogravimetric analysis (TGA). However, TGA may be sub-divided into *two* heads, namely :

(a) Static (or Isothermal) Thermogravimetric Analysis, and

(b) Dynamic Thermogravimetric

Analysis. Static Thermogravimetric

Analysis

In this particular instance the sample under analysis is maintained at a constant temperature for a period of time during which any changes in weight are observed carefully.

Dynamic Thermogravimetric Analysis

In dynamic thermogravimetric analysis a sample is subjected to conditions of predetermined, carefully controlled continuous increase in temperature that is invariably found to be linear with time.

2. Instrumentation

The essential requirements for an instrument (Figure 11.1) meant for thermogravimetric analysis are, namely :

(a) A high-precision balance,

(b) A furnace adequately programmed for a linear rise of temperature with time, and

(a) A sensitive recorder.

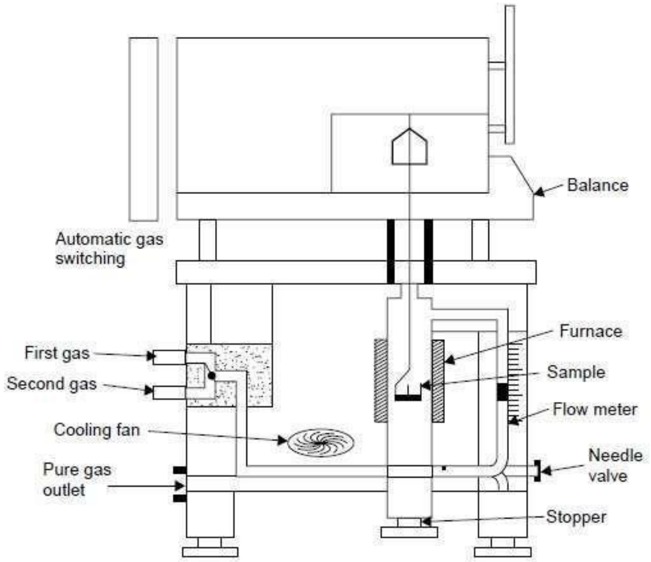


Figure 11.1 : Thermobalance for Thermogravimetric Analysis in a Controlled Atmosphere upto 1000°C.

Balances

They are usually of two types :

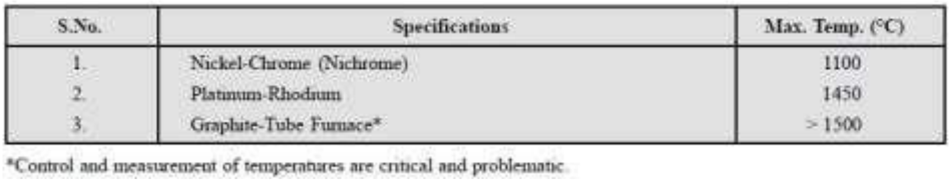
(a) Null-point Type : It makes use of an appropriate sensing-element which aptly detects any slightest deviation of the balance beam and provides the application of a restoring force, directly propor-tional to the change in weight, thereby returning the beam to its original null-point. The restoring-force is subsequently recorded either directly or with the aid of a transducer.

(b) Deflection Type : It is essentially based on either a conventional analytical balance consistingof helical spring, cantilever beam and strain gauze or a torsion analytical balance involving the conversion of deviations directly into a record of the weight change.

Furnace

The furnace must be designed in such a fashion so as to incorporate an appropriate smooth input thereby maintaining either a fixed temperature or a predetermined linear-heating programme (*e.g.*, 10°C-600°C per hour).

The temperature control of the furnace is satisfactorily achieved via a thermocouple mounted very close to the furnace-winding. The maximum operational temperature may be obtained by using different thermocouples as indicated below :



S.No.	Specifications	Max. Temp. (°C)
1.	Nickel-Chrome (Nichrome)	1100
2.	Platinum-Rhodium	1450
3.	Graphite-Tube Furnace*	> 1500

*Control and measurement of temperatures are critical and problematic.

Recorder

The recording device must be such so as to :

(i) record both temperature and weight continuously, and

(ii) make a definite periodic record of the time.

3. Methodology

The 'thermogram' for calcium oxalate monohydrate ($CaC_2O_4.H_2O$) is presented in Figure 11.2. The successive plateaus correspond to the anhydrous oxalate (100-250°C), calcium carbonate (400-500°C), and finally calcium oxide (700-850°C). In other words, these plateaus on the decomposition curve designate two vital aspects, namely :

(a) Clear indication of constant weight, and

(b) Stable phases within a specified temperature interval. The chemical reactions involved may be summarized as follows :

Interpretation of Thermogram

In the thermogram (Figure 11.2), which vividly illustrates the thermogravimetric evaluation of CaC2O4.H2O, it is ensured that the weight of this product decreases in several stages, namely :

Stage 1 : The water of hydration (or crystallization) from calcium oxalate monohydrate is lost which corresponds to 2.46 mg (12.3%) equivalent to 1 mole of H_2O in the temperature range 100-250°C.

Actually, the 12.3% weight loss that took place between 100-250°C should correspond to 12.3%

of the original formula weight for $CaCO_3$ H_2O (FW = 146). Hence, the product being lost has a formula weight of 0.123 × 146 = 17.958 (~ 18.0), and it corresponds to H_2O.

Stage 2 : One mole of carbon monoxide is evolved subsequently from calcium oxalate, corresponding to 3.84 mg (19.2%) in the temperature range 400-500°C.

The 19.2% weight loss that occurred between 400-500°C should correspond to 19.2% of the original formula weight of 146. Therefore, the product being given out has a formula weight of 1.92 × 146 = 28.0, that corresponds to CO.

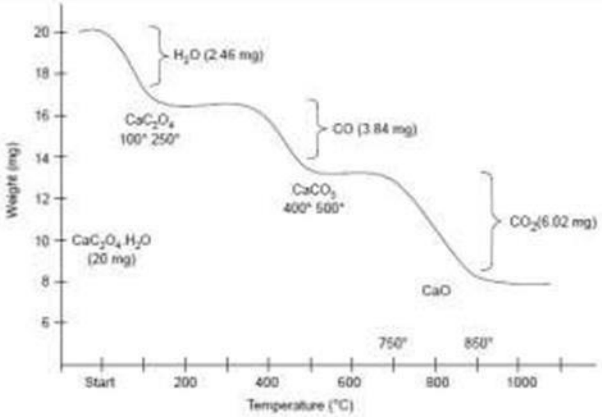


Figure 11.2 : Thermogravimetric Evaluation of Calcium Oxalate Monohydrate heated at the rate of 6°C min^{-1}.

Stage 3 : Finally, a mole of CO_2 is evolved from calcium carbonate that corresponds to 6.02 mg (3.01%) in the temperature range 700-850°C.

The weight loss amounting to 3.01% which took place in the range 700-850°C must, in fact, corresponds to 3.01% of the original formula weight of 146. Therefore, the product being evolved has a formula weight of 0.301 × 146 = 43.946 (˜ 44), and it corresponds to CO_2.

It is quite evident that in a multicomponent system wherein more than one component exhibits weight variations and that too at different temperature regions, the composition of the original compound may be estimated as depicted in Figure 11.2.

In a situation whereby an inert material is present along with a pure substance, from the generated thermogram the composition of the mixture may be derived from the percentage weight variation which takes place relative to the percentage weight variation observed with the pure compound (A), by employing the following expression :

$$\text{Component A (wt \%)} = \frac{\text{\% wt. change for mixture}}{\text{\% wt. change for pure compound A}} \times 100\ (\%)$$

4. Applications

The most broadly based application of the thermogravimetric analysis (TGA) has been visualized and exploited in the investigation of analytical methods, such as :

(i) Determining appropriate forms for many elements,

(ii) Screening and testing of substances which may be used as potential analytical standards (primary standard), and

(iii) Direct application of the technique in analytical assays.A few typical applications of TGA are, namely :

(iv) Plateaus for hydrates are sometimes based on the initial water content (*i.e.*, water of crystalliza-tion). It has been observed that in humidified air at low heating rates, hydrates usuallygive rise to good plateaus.

Example : Dehydration of sodium tungstate 28-hydrate [$Na_2WO_4.28\ H_2O$ (5 : 12)]

Experimental parameters* :

A. Humidified air, 300°C/hour,

B. Humidified air, 150°C/hour,

C. Humidified air 10°C/hour,

D. Room air, 10°C/hour,

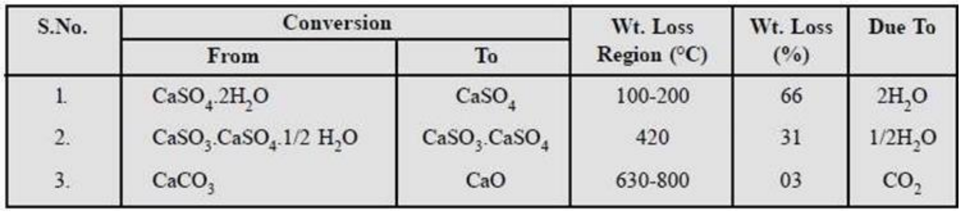
Sample weight : 0.5000 g

n = Moles water per $5Na_2O$, 12 WO_3

(a) Analysis of flue-gas scrubber system in environmental analysis.

The flue-gas emitted from a coal-fired-power-plant is subjected to scrubbing by the aid of wet limestone to get rid of sulphur dioxide (SO_2) as completely as possible. TGA helps in monitoring the system by carrying out the analysis of the products resulting from the scrubbing process, that mainly consist of (*i*) $CaCO_3$; (*ii*) $CaSO_3$. $CaSO_3$. 1/4 H_2O, and (*iii*) $CaSO_4$. $2H_2O$.

The thermogram obtained from TGA provides the following valuable informations which suggests the decomposition occurring at three distinct stages thereby causing the loss due to two moles of water, half-a-mole of water and one mole of CO_2.



S.No.	Conversion		Wt. Loss Region (°C)	Wt. Loss (%)	Due To
	From	To			
1.	$CaSO_4.2H_2O$	$CaSO_4$	100-200	66	$2H_2O$
2.	$CaSO_3.CaSO_4.1/2\ H_2O$	$CaSO_3.CaSO_4$	420	31	$1/2H_2O$
3.	$CaCO_3$	CaO	630-800	03	CO_2

(b) The stepwise degradation of organic polymers has received adequate attention which has broadened the in-depth knowledge of polymer chemistry. In this specific instance the sample is either heated under vacuum or in an inert atmosphere (of N_2).

(c) The thermogravimetric data may be employed to evaluate the kinetic parameters of weight varia-tions in reactions.

DIFFERENTIAL SCANNING CALORIMETRY (DSC)

Definition: DSC is a thermal method in which the difference in heat flow into the sample and the reference material is measured as a function of linear increase or decrease of the sample temperature.

Principle: DSC measures the difference in heating power (heat flow) required to keep the temperature of a sample and a reference material the same**.** The sample and the reference are heated by separate electrical heaters exactly at the same rate. Whenever the sample undergoes an endothermic or exothermic reaction, there is a change in sample temperature with respect to the reference temperature. The power to the sample heater is modified so that the difference in temperature between the sample and the reference, $\Delta T = 0$. The difference in power supplied to the sample and the reference represents the energy change or enthalpy change (ΔH) in the sample.

Instrumentation: DSC instrumentation is the same as DTA. DSC is a calorimetric method in which the difference in heat energy (ΔH) is recorded as a function of sample temperature. The block diagram of DSC instrument is shown in **Fig 3.13.**

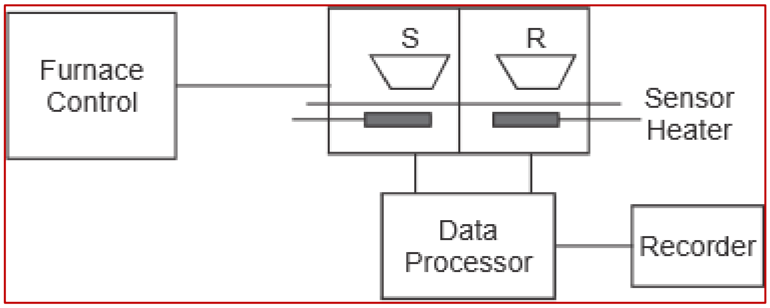


Fig 2.1 Block diagram of DSC instrument

The main components are a microprocessor controlled furnace provided with individual heater and thermocouples for measuring the temperature of sample and reference, data processor and recorder, and a facility for atmosphere control.

1. **Sample holder assembly:** The solid sample and the reference material (usually an inert substance) like alumina of 10 mg is placed in a separate platinum crucible (sample container) and connected to a sensitive microbalance. The sample and the reference are heated separately and the temperature of the sample and reference is measured by individual thermocouple. The thermocouples measure the difference in heat flow.
2. **Microprocessor controlled furnace**: The whole sample holder assembly is placed inside the furnace. The sample and the reference are heated at the same heating rate from ambient to 700°C. A temperature programmer or furnace control maintains a constant heating rate of 1°C/min -100°C/min.
3. **Facility to control atmosphere**: Sample and reference chamber are designed to permit the circulation of inert gases such as nitrogen or reactive gases such as oxygen or air.
4. **Data processor and recorder**: The difference in heat flow (ΔH) between the sample and the reference (S and R) thermocouples is continuously measured. After amplification, the difference in signal is recorded on the y-axis. The temperature of the furnace is measured by an independent thermocouple and recorded on the x axis. The balance and furnace data collected is sent to the PC for manipulation and the DSC thermogram which is a plot of ΔH vs. T is obtained. A typical DSC thermogram for the decomposition of calcium oxalate monohydrate is shown in **Fig 3.14.**

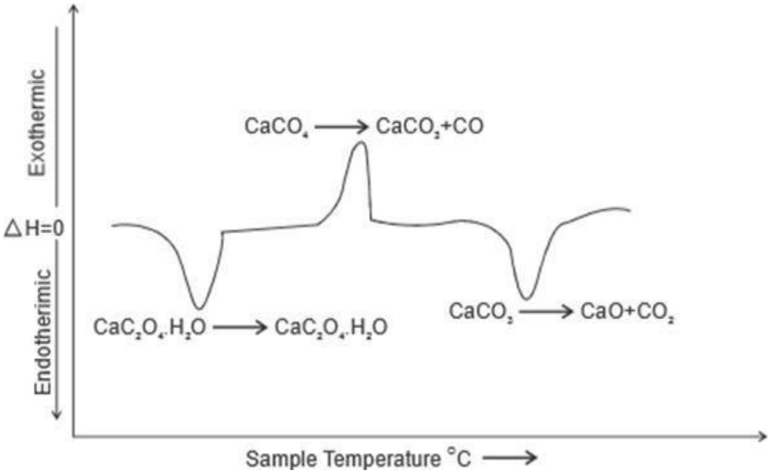


Fig 3.14 DSC thermogram for the decomposition of calcium oxalate monohydrateApplications of DSC

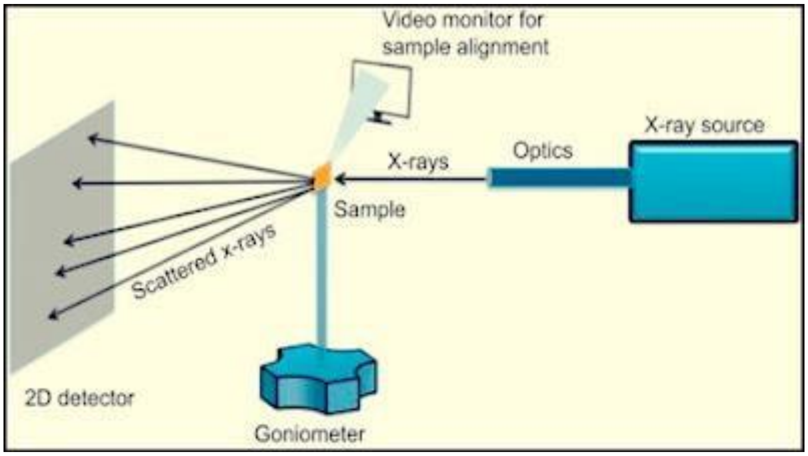
1. DSC is highly useful in the study of phase transitions and temperature induced changes that occur at very low temperatures in the case of polymers, biological samples, pharmaceutical samples, etc.
2. It finds widespread application in calculating the enthalpy of transitions such as enthalpy of melting, enthalpy of crystallization, enthalpy of fusion, etc. of polymeric materials.
3. DSC also provides accurate method of determining melting, boiling and decompositionpoints for organic compounds.
4. It is used for the determination of purity of drug samples.

X-RAY DIFFRACTION

X-Ray Diffraction is a rapid analytical technique primarily used for phase identification of crystalline materials. It can provide information on unit cell dimensions.

Principle

Based on the constructive interference of monochromatic x-rays and a crystalline sample in which the crystalline structure causes a beam of incident x-rays to diffract into many specific directions.



Concept

The concept of XRD can be explained as follows:

X-Rays

X-rays are the form of high energy electromagnetic radiation. These have a wavelength ranging from 10 picometers to 10 nanometers. X-rays have much higher energy and much shorter wavelengths than UV light. These are useful to take the images of the human body, i.e., they can see through a person's skin and reveal images of the bones beneath it.

Diffraction

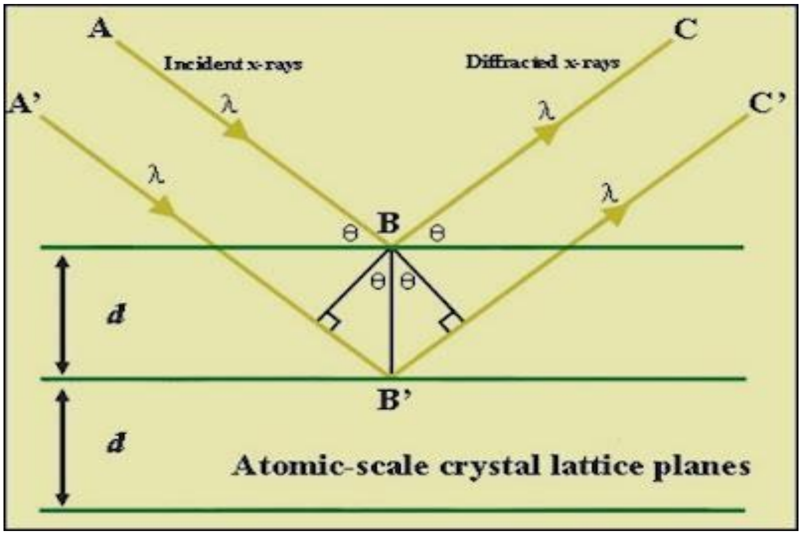
It is the slight bending of the light as it passed around the edge of an object. It occurs when a wave encounters an obstacle or a slit.

X-Ray Production

The x-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and directed toward the sample. Accelerating electrons with high voltages are allowed to collide with a metal target. The X-rays are produced when the electrons are suddenly decelerated upon collision with the metal target, and these x-rays are commonly called " Breaking Radiation". If the bombarding electrons have sufficient energy, they can knock an electron out of an inner shell of the target metal atoms. Then the electrons from the higher states drop down to fill the vacancy, emitting x-ray photons with precise energies determined by the electron energy levels. These rays are called characteristic x-rays.

Bragg's Law

It explains the relationship between an x-ray light shooting into, and it is reflected off from the crystal surface. The law states that when an x-ray is an incident onto a crystal surface, with an angle of incidence θ, it will reflect with the same angle of scattering θ. When the path difference (d) is a whole number (n), of wavelength (λ), constructive interference will occur.



Bragg's Law is given by:

$$n\lambda = 2d \sin\theta$$

Where,

λ = wavelength of the x-ray.

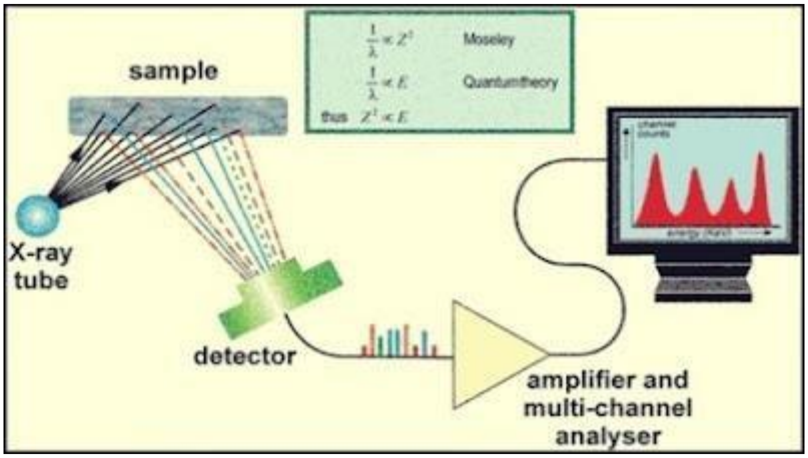
d = spacing of the crystal layers(path difference).

θ = incident angle.

n = integer.

Instrumentation

The XRD have following parts



1. Collimator

X-rays are generated by the target material when allowed to pass through a collimator. It consists of two sets of closely packed metal plates separated by a small gap.

Collimator absorbs all the x-rays, but the narrow beam that passes between the gaps is not absorbed.

2. Monochromators

Monochromators used are the following types:

Filters

The filter is a window of material that absorbs undesirable radiation but allows the radiation of the required wavelength to pass.

Crystal Monochromators

These are made up of a suitable crystalline material positioned in the x-ray beam so that the angle of reflecting planes satisfied the Bragg's equation for the required wavelength, and the beam is split up into component wavelengths.

The crystals used in monochromators are made up of materials like Sodium Chloride, Lithium Fluoride, and Quartz.

3. Detectors

Detectors use different types of methods to detect X-rays. They are:

a. Photographic Methods

Plane or cylindrical film is used, which is developed after exposure to x-rays.

Blackening of the developed film is always expressed in terms of density units D.

$$D = \log I0 / I$$

Where,

I0 = Incident intensities.

I = Transmitted intensities.

D = Total energy that causes the blackening of the film.

b. Counter Methods

It is further divided as follows:

i) Geiger-Muller Tube Counter

Filled with an inert gas like Argon. Anode (central wire) is maintained at a positive potential of 800-2500 V. The electrons are accelerated by the potential gradient and cause the ionisation of a large number of argon atoms resulting in the production of an avalanche of electrons that are travelling towards the central anode.

ii) Proportional Counter

Construction is similar to the Gieger tube counter. It is filled with heavier gases like Xenon and Krypton. Heavier gases are preferred because they are easily ionised.

It is operated at a voltage below the Gieger plateau. The dead time is very short (~0.2 microseconds); it can be used to count high rates without significant error.

iii) Scintillation Detectors

In a scintillation detector, there is a large sodium iodide crystal activated with a small amount of thallium. When an x-ray is an incident upon the crystal, the pulses of visible light are emitted which can be detected by a photomultiplier tube. It is useful for measuring x-rays of short wavelengths.

Crystals used in these detectors include Sodium Iodide, Anthracene, Naphthalene and *p*-terpineol.

iv) Solid State Semiconductor Detectors

In these types of detectors, the electrons produced by the x-ray beam are promoted into conduction bands, and the current which flows is directly proportional to incident x-ray energy.

Disadvantage: Semiconductor devices should be maintained at low temperatures to minimise noise and prevent deterioration.

v) Semiconductor Detectors

When x-rays fall on silicon lithium drifted detectors, an electron (-e) and a hole(+e) are produced. Pure silicon made up with a thin film of lithium metal is placed onto one end. Under the influence of voltage, the electrons will move towards positive charge and holes towards negative charge. The voltage generated is the measure of x-ray intensity falling on the crystal.

Upon arriving at lithium, the pulse is generated,

Voltage of pulse, $V = q/c$

Where,

q = total charge collected on electrode

c = detector capacity

X-Ray Diffraction Methods

XRD methods are of following types:

1. Laue's Photographic Method

It is again divided into two types:

a. Transmission Laue Method

A film is placed behind the crystal. It records the beams which are transmitted through the crystal. One side of the cone of Laue reflections is defined by the transmitted beam. The film always intersects the cone, with the diffraction spots generally lying on an ellipse.

This method is used in the determination of symmetry of single crystals.

b. Back Reflection Method

In the back reflection method, a film is placed between the crystal and the x-ray source. The beams which are diffracted backwards are recorded. One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on a hyperbola.

This is similar to the Transmission method; however, back reflection is the only method for the study of large and thick specimens.

Crystal orientation is always determined from the position of the spots. The spots can be indexed, i.e., attributed to a particular plane, using special charts.

The Laue technique can also be used to assess crystal perfection from the size and shape.

A Greninger chart is used for back reflection patterns, and the Leonhardt chart is for transmission patterns.

2. Bragg's X-Ray Spectrometer Method

Single plane generates several diffraction lines. Sum total of diffraction lines gives diffraction patterns. From the pattern, we can deduce different distances between planes.

By plotting a graph between ionisation current and the glancing angle θ, we can obtain the X-ray spectrum.

Ratios will be different for different crystals. Experimentally observed ratios are compared with the calculated ratios; the particular structure may be identified.

3. Rotating Crystal Method

It is two types:

a. Complete Rotation Method

In this method, a series of complete revolutions occur. Each set of planes in a crystal diffracts four times during a single rotation. These beams are distributed into a rectangular pattern in the central point of the photograph.

b. Oscillation Method

The crystals oscillated at an angle of 15° or 20°. The photographic plate is moved back and forth with the crystal. The orientations of the crystals at which the spot was formed are indicated by the position of the spot on the plate.

4. Powder Crystal Method

The analysed material is finely grounded, homogenised, and average bulk composition is determined. The fine powder is struck on a hair with a piece of gum; it is suspended vertically in the axis of a cylindrical camera.

When the monochromatic beam is allowed to pass different possibilities may happen:

In a fine powder, there will be some particles out of random orientation of small crystals.

Reflections are possible in different orders for each set.

Another fraction of grains will have another set of planes in the correct positions for the reflections to occur.

Applications of XRD

- Structure of crystals is determined.
- Polymer characterisation
- Particle size determination
- Spot counting method.
- Broadening of diffraction lines
- Low angle scattering
- Applications of diffraction methods to complexes
- Determination of cis-trans isomerism.
- Determination of linkage isomerism.

IMPORTANT QUESTIONS

Long answer type Questions (10 Marks)

1. Write application and instrumentation of TGA
2. Give application and instrumentation of DSC.
3. Draw a well labeled diagram for the instrumentation of DTA. Write its applications.
4. Explain in detail the instrumentation and applications of XRD.

Short answer type questions (5Marks)

1. Write briefly about various types of balances used in TGA.
2. Explain the components of DSC curve.
3. Draw a DTA curve and explain.
4. What is a TGA curve?
5. What is powder diffraction method?
6. Name the factors affecting DTA curve.
7. Explain the factors affecting DSC curve.
8. Compare DTA and DSC.
9. Compare DTA and TGA.
10. Write about the various factors affecting TGA.

Very Short answer type questions (2 Marks)

1. Define the principle of DTA.
2. State the principle of DSC.
3. Give two applications of TG.
4. Mention two applications of DTA.
5. State the principle of TGA.
6. Give two applications of DSC.
7. Name the reference material used in DSC
8. Define thermal methods of analysis.
9. Write Bragg’s equation.
10. Enlist the various sources of X-rays.