Thermal Methods of Analysis

Thermogravimetric Analysis (TGA)

Principle:

- TGA measures the mass of a sample as it is heated, cooled, or held at a constant temperature in a controlled atmosphere.
- The change in mass helps identify decomposition, oxidation, or volatilization processes.
    - Weight loss or gain corresponds to loss of volatile components, decomposition, or oxidation/reduction.
    - The rate of weight change can provide information on reaction kinetics.

Typical Thermal Events Detected by TGA

1. Moisture loss (evaporation of water)
2. Decomposition (breakdown of organics, carbonates, etc.)
3. Oxidation or combustion (gain or loss of mass)
4. Reduction (mass changes under reducing atmosphere)
5. Volatilization of components

Instrumentation of TGA

A standard TGA system consists of the following components:

1. **Balance (Microbalance)**
    - Very sensitive (±1 microgram)
    - Measures mass changes as small as micrograms.
2. **Furnace**
    - Heats the sample from room temperature to ~1000–1600 °C.
    - Heating rates can be programmed (e.g., 10 °C/min).
    - Provides isothermal or dynamic heating.
3. **Sample Pan**
    - Made from inert, thermally stable materials (e.g., platinum, alumina).
    - Holds the sample (1–50 mg typically).
4. **Reference Pan (Optional)**
    - Used in some systems for comparison or baseline correction.
5. **Temperature Measurement System**
    - Thermocouple placed near or in contact with the sample.

- Provides accurate sample temperature reading.

6. Gas Control System

- Supplies a controlled atmosphere:
  - Inert gases (e.g., nitrogen, argon) for pyrolysis or decomposition.
  - Oxidizing gases (e.g., air, oxygen) for combustion studies.

7. Data Acquisition System

- Computer records the TGA curve: Mass (%) vs. Temperature (or Time).
- Allows derivative TGA (DTG) plotting: rate of mass loss vs. temperature.

TGA Curve Interpretation:

- A typical TGA thermogram shows:
  - Flat region: No mass loss (stable region).
  - Stepwise drops: Indicate distinct mass loss events (e.g., moisture loss, decomposition).
  - Final plateau: Residue (e.g., ash or metal oxide).
- The onset temperature, rate of mass loss, and residual mass help identify and quantify materials.

Applications:

- Determination of moisture content and volatile matter in a sample.
- Thermal stability and decomposition temperature of materials (e.g., polymers, drugs).
- Oxidation behavior of metals or organics.
- Compositional analysis (e.g., filler content in rubber).
- Kinetic studies of thermal degradation.

Differential Thermal Analysis (DTA)

Principle:

- DTA measures the temperature difference between a sample and an inert reference material while both are subjected to the same heating/cooling program.
  - When the sample undergoes a physical or chemical change (e.g., melting, crystallization), it absorbs or releases heat, causing a temperature difference from the reference.
  - This difference is plotted as a function of temperature/time.

Types of Thermal Events Detected:

1. Endothermic Reactions:

- Absorption of heat (e.g., melting, dehydration).
- DTA curve shows a dip or downward peak.

2. Exothermic Reactions:

- Release of heat (e.g., crystallization, combustion).
- Appears as an upward peak.

Instrumentation of DTA

A typical DTA system includes:

1. Furnace

- Heats both sample and reference simultaneously.
- Temperature range usually up to 1500 °C.
- Controlled heating rates (commonly 5–20 °C/min).

2. Sample and Reference Holders

- Two crucibles: one for the sample, the other for an inert reference (usually alumina).
- Made from thermally stable, non-reactive materials.

3. Thermocouples

- Embedded beneath or inside both sample and reference crucibles.
- Measure temperature of both materials to calculate $\Delta T = T_{sample} - T_{reference}$.

4. Data Acquisition System

- Records and plots the temperature difference (ΔT) vs. time or temperature.
- Output is the DTA thermogram.

5. Gas Supply System

- Supplies air, oxygen, or inert gases (e.g., nitrogen, argon).
- Controls the reaction environment.

DTA Curve Features:

- Endothermic peaks appear as downward deflections.
- Exothermic peaks appear as upward deflections.
- Onset, peak, and completion temperatures can be derived from each thermal event.

Applications:

- Identification of phase transitions (melting point, boiling point, glass transition).
- Characterization of reactions (exothermic/endothermic).
- Purity analysis – sharp endothermic peak = pure compound.

First Hope

FREE PREVIEW

Click here to visit our website

PHARMACY PREMIUM NOTES

Complete subject and semesters are available on FirstHope app only.

Scan or click here

Click here to get the app >

Available on Google Play

JOIN THE FIRSTHOPE COMMUNITY

Join our Telegram channel

Click here to join Telegram >

Scan or click here

@FirstHopeNotes

Instagram

@firsthope.official

Click here to follow >

YouTube channel

FirstHope-Pharmacy / Live streams

Click here to watch >

WhatsApp channel

Follow FirstHope on WhatsApp

Click here to join >

FirstHope website

firsthope.co.in

Click here to visit >

B.Pharm / D.Pharm / M.Pharm / Pharm.D

Preview notes. Complete subjects and semesters in the FirstHope app.