Thermal Analysis

Thermal Analysis: Introduction

What is Thermal Analysis?

- Thermal analysis refers to a group of techniques in which the physical or chemical properties of a material are measured as a function of temperature or time while the material is subjected to a controlled temperature program.
- This means that as a material is heated, cooled, or held isothermally, we track how its properties change with temperature and/or time.
- These changes can include:
    - Weight loss or gain (e.g., due to decomposition or oxidation)
    - Heat absorption or release (e.g., due to melting, crystallization, or chemical reaction)
    - Changes in physical dimensions (e.g., expansion)
    - Evolution of gases, and more.

Why is Thermal Analysis Important?

- Materials behave differently under various thermal conditions. Understanding these behaviors is crucial for:
    - Designing materials with specific thermal properties
    - Predicting product performance under real-world conditions
    - Quality control in manufacturing
    - Research and development of new materials
- Thermal analysis helps in determining key thermal transitions such as:
    - Melting point
    - Glass transition temperature (Tg)
    - Decomposition temperature
    - Oxidation stability
    - Phase transitions

Main Techniques under Thermal Analysis:

- **TGA (Thermogravimetric Analysis)** – Measures weight change with temperature.
- **DSC (Differential Scanning Calorimetry)** – Measures heat flow associated with transitions.
- **DTA (Differential Thermal Analysis)** – Measures temperature difference between sample and reference.
- **TMA (Thermomechanical Analysis)** – Measures dimensional changes.

- **DMA (Dynamic Mechanical Analysis)** – Measures mechanical properties under periodic stress.
- **EGA (Evolved Gas Analysis)** – Identifies gases released upon heating.

Thermal Analysis: Instrumentation

Each technique under thermal analysis requires specialized instrumentation. Despite variations, most systems follow a common architecture.

Core Components of Thermal Analysis Instruments:

1. Sample Holder and Furnace

- The sample is placed in a small crucible (metallic, ceramic, or platinum).
- The furnace heats or cools the sample at a **controlled rate** (typically 1–20 °C/min, but can vary).
- An inert atmosphere (like nitrogen or argon) or oxidative atmosphere (like air or oxygen) is maintained, depending on the test.

2. Temperature Measurement and Control

- Thermocouples (e.g., type K or type R) are placed close to or in contact with the sample to accurately measure its temperature.
- The system is designed to perform **precise ramping**, **isothermal holds**, or **cyclic temperature programs**.

3. Detection System

- Different techniques use different sensors:
  - **TGA** uses a microbalance to detect minute changes in mass.
  - **DSC** uses heat flux sensors or power compensation to detect heat flow.
  - **DTA** uses thermocouples to detect temperature differences.
  - **TMA/DMA** use displacement sensors to measure changes in size or mechanical behavior.

4. Data Acquisition and Analysis Software

- Thermal analysis instruments are connected to computers via specialized software.
- The software records:
  - Temperature,
  - Time,
  - Sample mass,
  - Heat flow,

- Dimensional changes, etc.

➢ It also processes data to provide plots (e.g., weight vs temperature, heat flow vs time), and detects peaks, transitions, and onset points.

5. Gas Flow Control

➢ To maintain an inert or reactive atmosphere, gases like **nitrogen**, **oxygen**, or **air** are purged through the system.

➢ Gas flow controllers manage the flow rate (typically in mL/min).

Thermal Analysis: Applications

Thermal analysis finds widespread application across a variety of fields:

1. Polymer Industry

➢ Determine **glass transition temperature (Tg)**, **melting point**, **crystallinity**, and **thermal stability**.

➢ Assess curing behavior of thermosetting polymers (via DSC).

➢ Detect degradation onset (via TGA).

2. Pharmaceuticals

➢ Determine **polymorphism**, **purity**, and **compatibility** of drugs with excipients.

➢ Study **melting behavior**, **crystallization**, and **hydration/dehydration** transitions.

➢ Analyze thermal decomposition pathways.

3. Metals and Alloys

➢ Study **phase transitions** (e.g., solid-solid transformations, melting, eutectic points).

➢ Detect impurities or composition differences based on heat flow or mass changes.

4. Ceramics and Inorganic Compounds

➢ Evaluate sintering behavior, phase stability, and thermal expansion.

➢ Determine decomposition of carbonates, nitrates, etc.

5. Food and Agriculture

➢ Analyze thermal stability of fats, oils, and proteins.

➢ Study glass transition in freeze-dried foods.

➢ Determine moisture content (via TGA).

6. Environmental Science

➢ Analyze degradation products or volatile organic compounds (VOC) from materials.

- Study the thermal stability of waste or recycled materials.

7. Forensic and Quality Control

- Characterize unknown substances.
- Verify material authenticity or detect contamination.

Differential Scanning Calorimetry (DSC)

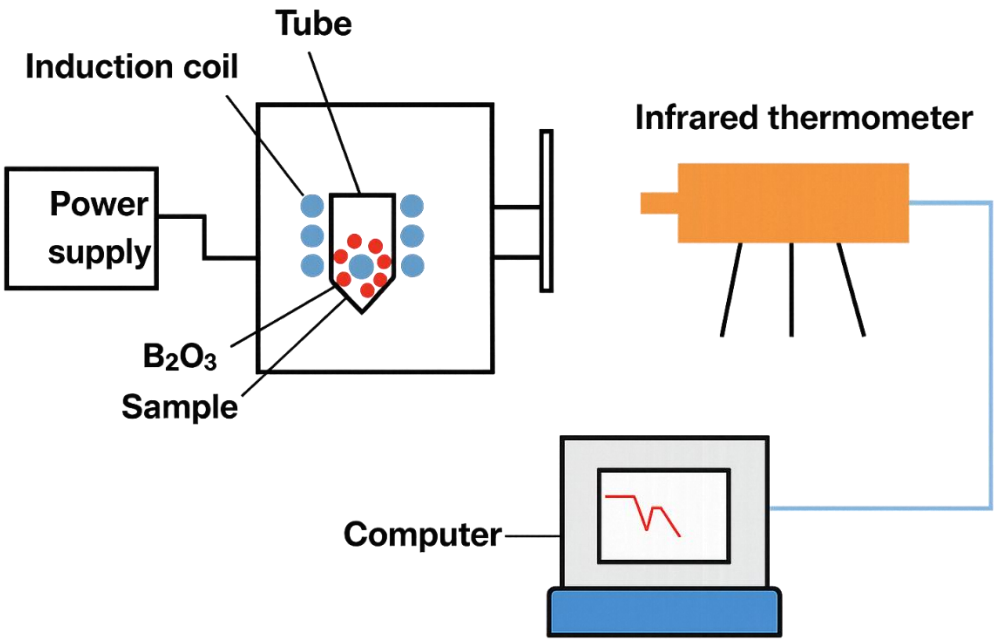
Principle:

- DSC measures the **heat flow** into or out of a sample **compared to a reference**, as a function of temperature or time, under controlled heating or cooling.
- Endothermic processes (e.g., melting, glass transition) require heat input → positive peaks.
- Exothermic processes (e.g., crystallization, oxidation) release heat → negative peaks.

Types of DSC

1. **Heat-Flux DSC**: Sample and reference are heated together; measures temperature difference across a sensor.
2. **Power-Compensated DSC**: Sample and reference are kept at the same temperature using independent heaters.

Instrumentation:



A typical DSC setup includes:

1. Sample and Reference Pans

- Small crucibles (~10 mg sample).
- Made of inert materials (e.g., aluminum, platinum).

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