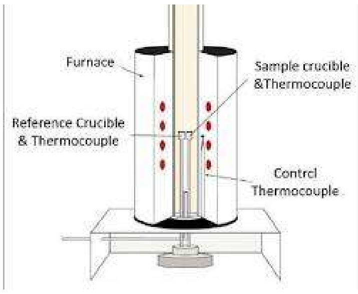
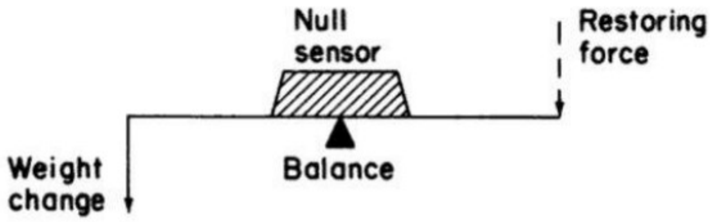
Working on different Parts in TGA

Balance: In TGA Microbalances are used which has been a critical component that ensures precise and accurate measurement of mass changes in the sample. Range: 1 μg to 1 g, Sensitivity: 0.1 μg to 1 μg, mainly it has two types.





- **Null Point Balance:** The balance beam moves away from its null position when there is a change in weight. A sensor detects this deviation and applies a force to restore the balance to the null position. It will offer High precision and stability, essential for detecting small changes in mass during thermal analysis.

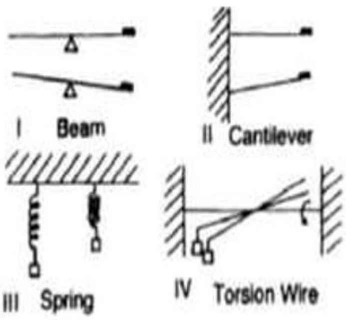


Fig – Types of deflection balance

Deflection type balances: are a type of weighing instrument used in thermogravimetric analysis (TGA). These balances are based on the principle of measuring the deflection of a mechanical element caused by the weight of the sample. It is also further explained by the following examples:

- **Beam balance:** A traditional type of balance with a beam that pivots on a fulcrum. Weights are added to one side of the beam to balance the weight of the sample on the other side.
- **Cantilever balance:** A balance with a long, flexible arm that bends under the weight of the sample. The deflection of the arm is measured to determine the weight.
- **Spring balance:** A balance that uses a spring to measure weight. The extension of the spring is proportional to the weight of the sample.
- **Torsion wire balance:** A balance that uses a torsion wire to measure weight. The twisting of the wire is proportional to the weight of the sample.

Sample Holder: A sample holder, also known as a crucible or pan, is a vital component of a thermogravimetric analysis (TGA) instrument. It holds the sample during the analysis and ensures accurate weight measurements.

Common types of sample holders:

- **Crucibles**: These are typically made of materials like platinum, alumina, or quartz. They are suitable for a wide range of samples and can be reused after cleaning.
- **Pans**: Pans are often made of materials like aluminium, ceramic or stainless steel. They are disposable and are commonly used for volatile or corrosive samples.
- ***Platinum Pans, Stainless steel, Ceramic Pans, Aluminum foil, Quartz or Alloy. Generally, Pan volumes of 20, 50, 100, and 250 µL are common.***

Furnace

A furnace is a crucial component of a thermogravimetric analysis (TGA) instrument. It provides the controlled heating environment necessary for the thermal decomposition or other processes being studied. The furnace surrounds the sample and sample holder. In TGA instruments commonly three types of furnaces are available which are described in the below figure.

 **Resistance furnace:** This type of furnace uses electrical resistance to generate heat. It is a common choice due to its versatility and ease of control.	 **Induction furnace:** Induction furnaces use electromagnetic induction to generate heat. They are often used for high-temperature applications or for materials that are difficult to heat using other methods.	 **Infrared furnace:** Infrared furnaces use infrared radiation to heat the sample. They are often used for delicate or temperature-sensitive materials.

It was capable of producing a wide range of temperature programs accurately. **Nichrome (1100°C), Platinum, Alloy of platinum and rhodium (1500°C), and Tungsten molybdenum (1750°C)** are used as materials for the furnace. Temperature control of the furnace is achieved via a thermocouple mounted very close to the furnace winding. A high-size furnace yields a larger hot zone whereas a small size may not. Temperature controlling with a small-size furnace may not be possible.

Temperature measurement (Thermocouple)

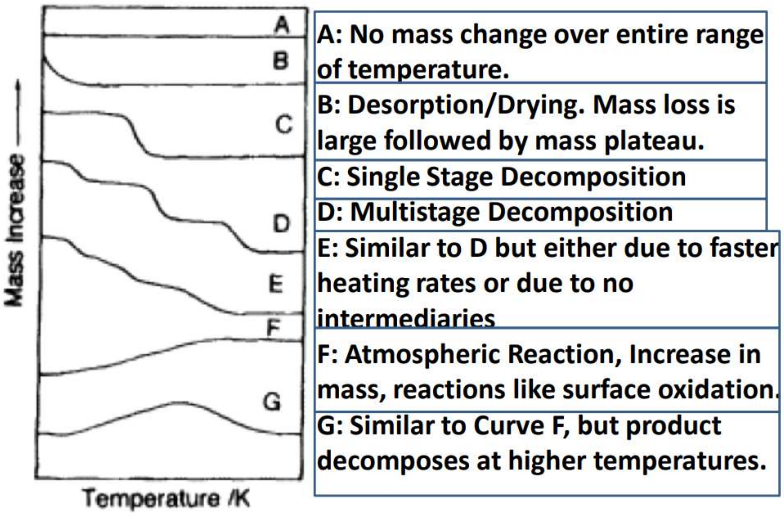
Thermocouples are essential components of TGA instruments that work as sensors. They are used to accurately measure the temperature of the sample and the furnace, providing essential data for the analysis. The position of the thermocouple plays an important role in temperature measurement. Thermocouples in contact with the sample or sample holder are preferred. Generally, it is constituted of **Chromel, Tungsten, and Rhenium** thermocouples.

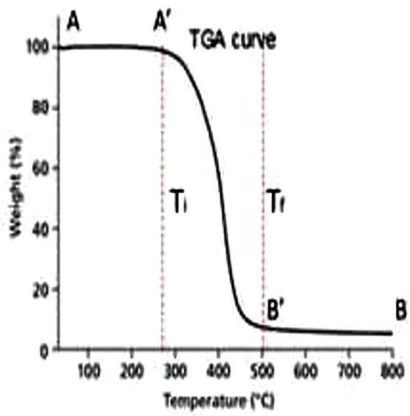
It is associated with ***Temperature monitoring, Data correlation, Calibration, and Temperature gradient control.***

Data Recorder:

The data recorder in a TGA instrument works as a computer workstation which is used to control, collect, and process data received from the sensors which is accurately converted and processed to produce a thermogram, which is a graphical representation of the weight loss or gain of the sample during the analysis.

Understanding of TGA Graphs





Procedural Decomposition Temperature (PDT): It is the lowest temperature at which cumulative mass change reaches a magnitude that the thermocouple can detect. Indicated by $\mathbf{T_i}$.

Final Temperature: It is the temperature at which cumulative weight change reaches its maximum value, corresponding to the complete reaction indicated by $\mathbf{T_f}$.

Reaction Interval: The difference between $\mathbf{T_f}$ and $\mathbf{T_i}$ is defined as the reaction interval. Horizontal Position indicates that there is no weight change. From this, we can know about the thermal stability of the compound (no change in properties). Curve A to A' is perfectly horizontal, so it will be stable up to T°C. Curved portion (A' to B') indicates weight loss.

Factors Affecting TGA Analysis

Thermogravimetric analysis (TGA) is a sensitive technique that measures the mass of a sample as it's heated or cooled. Several factors can influence the accuracy and reliability of TGA results.

Instrument Sensitivity: Balance precision: The balance used in the TGA instrument should have sufficient precision to accurately measure mass changes. Noise: Electronic noise can affect the accuracy of the weight measurements.

Calibration: Regular calibration: The TGA instrument should be calibrated regularly to ensure accurate weight measurements. **Reference materials:** Standard reference materials can be used to verify the accuracy of the instrument.

Heating Rate: Rapid heating: Can cause rapid weight loss and make it difficult to accurately identify thermal events. Slow heating: This can extend the analysis time and may not capture transient events.

Furnace Atmosphere: The atmosphere in the furnace causes irregularities in the TG curve. Inert atmosphere: Prevents oxidation and other reactions with the sample. **Oxidizing atmosphere:** This can cause oxidation of the sample, leading to weight gain. **Reducing atmosphere:** This can reduce oxides in the sample, leading to weight loss. **Gas flow rate:** The flow rate of the gas can affect heat transfer and the rate of chemical reactions.

Sample Holder: The shape and size of the holder affect the heating rate of the sample, which may affect the TG curve. Crucible Material, Thermal conductivity, Chemical reactivity and Weight.

Sample Preparation: Particle size: Smaller particle sizes can lead to faster heating and cooling rates.

Sample homogeneity: Inhomogeneous samples can result in uneven heating and inaccurate measurements.

Sample packing: Loose packing can cause air gaps and affect heat transfer.

Moisture content: Moisture can evaporate during the analysis, affecting the weight loss and making it difficult to interpret results.

Application of TGA

Thermogravimetric analysis (TGA) is a versatile technique with a wide range of applications in various fields. Here are some common applications:

To study the kinetics of reaction rate.
To study thermal stability.
To study the sublimation behaviour of various substances.
To study catalysts - change in the chemical state of catalysts is determined.
Used for purity determination of primary and secondary standards.

Materials Characterization: Thermal stability: Determining the temperature at which a material decomposes or undergoes other thermal transformations. **Moisture content**: Measuring the amount of water present in a sample. **Ash content**: Determining the inorganic residue remaining after the organic matter is burned off. **Volatile content**: Quantifying the number of volatile substances in a sample. **Oxidation kinetics:** Studying the rate at which a material oxidizes.

Polymer Analysis: Polymer degradation: Investigating the thermal stability and degradation mechanisms of polymers. **Polymer blending:** Analyzing the compatibility and phase separation of polymer blends. **Polymer additives:** Studying the effects of additives on polymer properties.

Pharmaceutical Industry: Drug purity: Assessing the purity of pharmaceutical compounds. Drug release: Investigating the release kinetics of drugs from formulations. **Thermal stability:** Evaluating the thermal stability of drugs and excipients.

Environmental Science: Waste characterization: Analyzing the composition and thermal behaviour of waste materials. **Soil analysis:** Determining the organic matter content and thermal properties of soils. **Air pollution:** Monitoring particulate matter and other pollutants in the air.

Food Industry: Moisture content: Measuring the moisture content of food products. **Ash content:** Determining the nutritional value of food. **Thermal stability:** Evaluating the thermal stability of food ingredients and products.

Other Applications: Catalyst characterization: Studying the thermal stability and activity of catalysts. **Forensic analysis:** Investigating the composition of unknown materials. **Geological analysis:** Analyzing the mineral composition of rocks and soils.

TGA is a valuable tool for researchers and scientists in various fields, providing insights into the thermal behaviour of materials and aiding in the development of new products and processes.

Advantages of TGA

Accurate method
Easy to use
Minimal sample preparation
Convenient and time-saving technique
The instrument can be used at very high temperatures

Versatility: TGA can be used to study a wide range of materials, including solids, liquids, and gases. It applies to various fields such as materials science, chemistry, pharmaceuticals, and environmental science.