Thermogravimetric analysis (TGA)

Thermogravimetric analysis, also known as TGA is a technique frequently used in thermal analysis in which a change in the weight of a substance is recorded as a function of temperature or time. Composition, purity, decomposition processes, decomposition temperatures, and absorbed moisture content are among the characteristics and behaviours that can be measured by TGA.

- History of TGA: The concept of measuring weight changes in materials as they are heated dates back to the 18th and 19th centuries. Early experiments involved simple balances and furnaces to observe weight loss due to thermal decomposition.
- Development of Instruments: Significant advancements occurred in the early 20th century with the development of more precise instruments. The introduction of thermobalances allowed for more accurate measurements of weight changes under controlled temperature conditions.
- Modern TGA: By the mid-20th century, TGA had become a well-established technique in materials science. Modern thermogravimetric analyzers are highly sophisticated and capable of measuring minute weight changes with high precision under various atmospheric conditions. TGA is widely used today for studying the thermal stability and composition of materials, including polymers, composites, and pharmaceuticals. It provides critical data for understanding material properties and behaviours under thermal stress.
- **Definition:** TGA is a branch of thermal analysis which examines the mass change of a sample as a function of temperature or time as the sample is subjected to a controlled temperature program in a controlled atmosphere.
- **Principle:** The sample is heated in a given environment (air, N_2, CO_2, He, Ar, etc.) at a controlled rate. The change in the weight of the substance is recorded as a function of temperature or time. The temperature is increased at a constant rate for a known initial weight of the substance, and the weight changes are recorded as a function of time at different temperature intervals.
- The plot of weight change against temperature is called a thermogravimetric curve or thermogram.

Terms in Thermogravimetric Analysis (TGA)

In Thermogravimetric Analysis (TGA), several terms are commonly used to describe the process and results. Here are some of the most popular terms:

- **Thermo:** Thermal
- **Gravimetric:** Measurement of weight
- **Thermogravimetric Analysis (TGA):** It is a thermo-analytical technique that measures the weight change of a sample at a given time and temperature.
- **Thermogram:** A plot of weight change against temperature or time.
- **Decomposition Temperature:** The temperature at which a material starts to decompose.

- **Weight Loss:** The reduction in the mass of the sample as it is heated.
- **Residual Mass:** The remaining mass of the sample after heating to a specific temperature.
- **Isothermal or Static TGA:** TGA is conducted at a constant temperature for a period of time, during which any changes in weight are carefully observed.
- **Dynamic TGA:** TGA analysis with a continuous increase in temperature at a predetermined rate, which is typically linear with time.
- **Quasistatic TGA:** TGA conducted with stepwise increases in temperature with constant weight at each of a series of increasing temperatures.
- **Derivative Thermogravimetry (DTG):** The rate of weight change with respect to temperature or time. It helps identify the exact temperature at which weight loss events occur. The DTG curve is the first derivative of the TGA curve.
- **The glass transition temperature (Tg):** is the temperature at which an amorphous material, such as a polymer, transitions from a hard, glassy state to a soft, rubbery state. This transition is characterized by a significant change in mechanical properties, such as stiffness and brittleness.
- **Onset Temperature:** The temperature at which mass loss or gain begins.
- **End Temperature:** The temperature at which mass loss or gain ends.
- **Baseline:** The line representing the mass of the empty crucible or the initial mass of the sample.
- **Decomposition Temperature:** The temperature at which a material decomposes or breaks down.
- **Moisture Content:** The amount of water present in a sample.
- **Ash Content:** The inorganic residue remaining after organic matter is burned off.
- **Thermal Stability:** The ability of a material to withstand high temperatures without significant mass loss or degradation.
- **Oxidation Kinetics:** The rate at which a material oxidizes.

In Thermogravimetric Analysis (TGA), several calculation terms are essential for interpreting the data and understanding the thermal behaviour of materials

- **Weight Loss (%):** The percentage of the initial mass that is lost during the heating process. It is calculated as:

$$\text{Weight Loss (\%)} = \frac{\text{Initial Mass} - \text{Final Mass}}{\text{Initial Mass}} \times 100$$

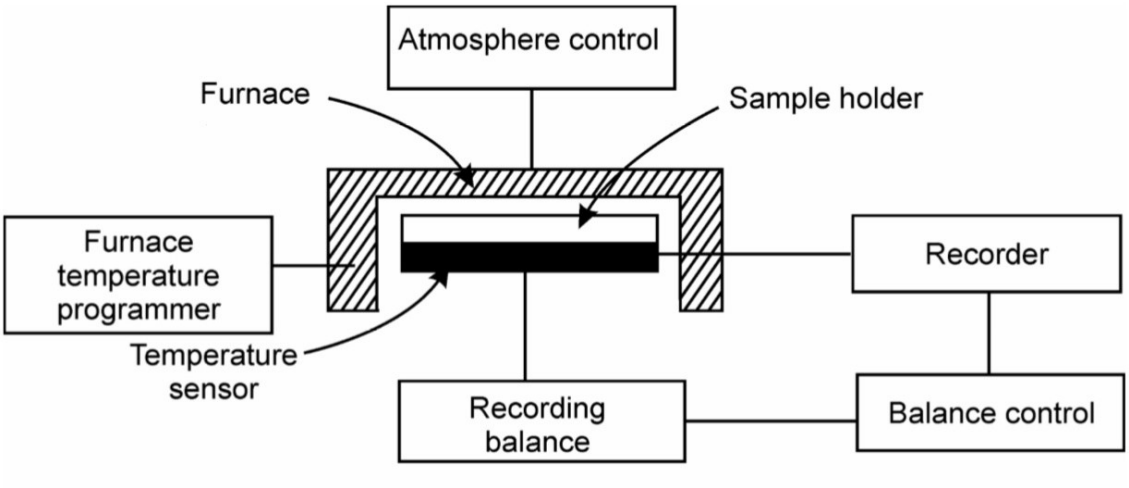
- **Residual Mass (%):** The percentage of the initial mass that remains after the heating process. It is calculated as:

$$\text{Residual Mass (\%)} = \frac{\text{Final Mass}}{\text{Initial Mass}} \times 100$$

- **Decomposition Temperature:** The temperature at which a significant weight loss occurs, indicating the decomposition of the material.

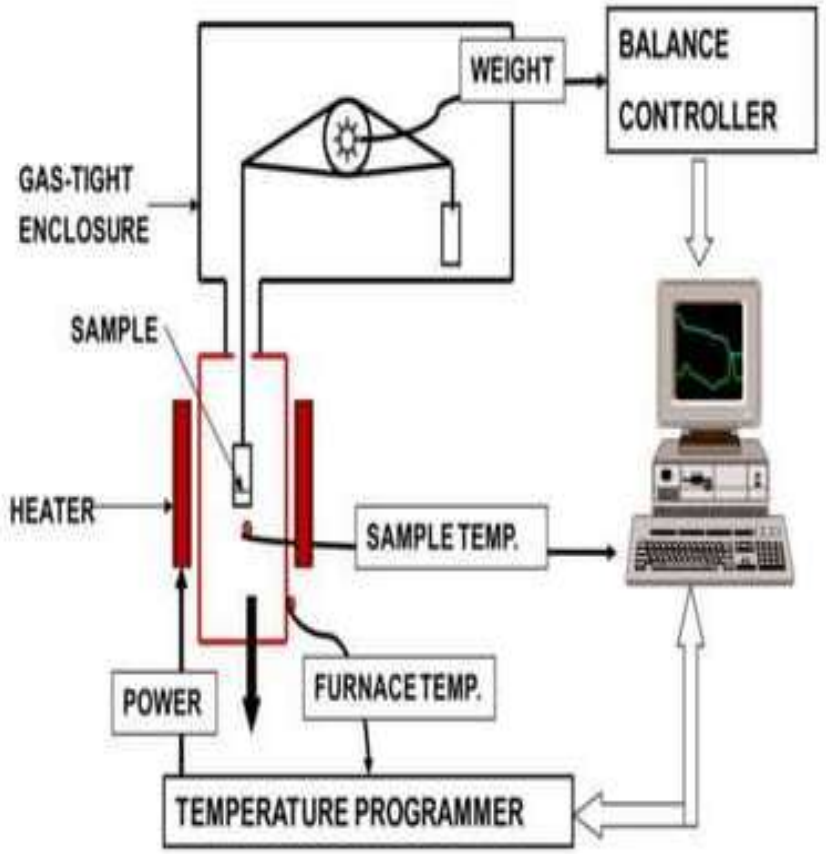
- **Activation Energy (Ea):** The energy required to initiate a thermal decomposition reaction. It can be calculated using methods like the Kissinger method or the Flynn-Wall-Ozawa method.
- **Reaction Order (n):** The order of the reaction, which can be determined from the TGA data is essential for understanding the kinetics of the decomposition process.
- **Peak Temperature:** The temperature at which the maximum rate of weight loss occurs, often seen as a peak in the DTG curve.
- **TG Curve:** The result from the above process is recorded as a plot of Weight Change vs. Temperature or Time. This is referred to as the TG Curve. In Static TGA, the graph will be of Weight vs. Time. In Dynamic TGA, the graph will be of Weight vs. Temperature.

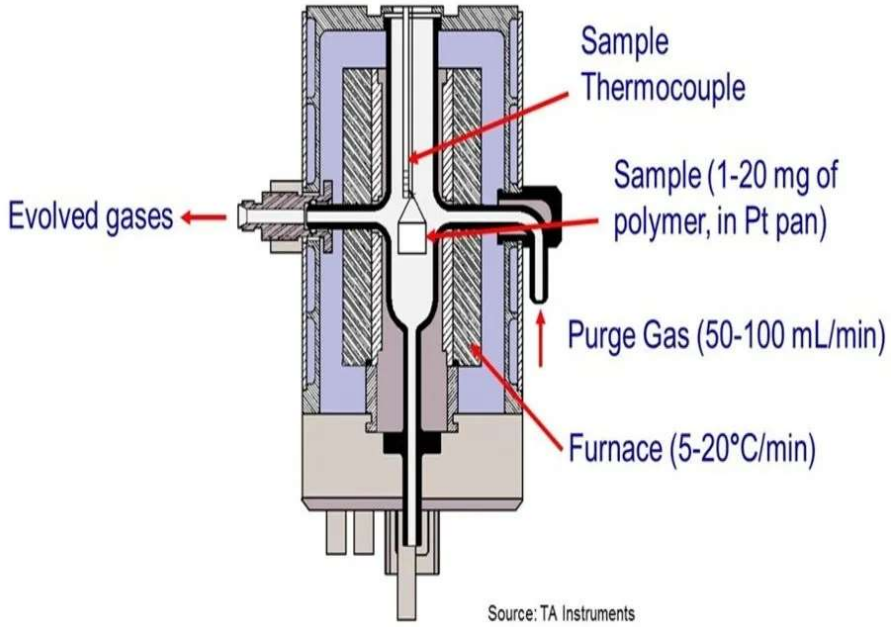
Instrumentation of Thermogravimetric Analysis (TGA)



Main Components of Thermogravimetric analysis (TGA) instruments

- **Balance**
- **Sample Holder**
- **Furnace**
- **Temperature Measurement (Thermocouples)**
- **Data Recorder**


WEIGHT
BALANCE
CONTROLLER
GAS-TIGHT
ENCLOSURE
SAMPLE
HEATER
SAMPLE TEMP.
POWER
FURNACE TEMP.
TEMPERATURE PROGRAMMER
To balance mechanism


Sample
Thermocouple
Sample (1-20 mg of
polymer, in Pt pan)
Evolved gases
Purge Gas (50-100 mL/min)
Furnace (5-20°C/min)
Source: TA Instruments