

Thermogravimetric/Differential Thermal Analysis (TG/DTA).

Thermal analysis involves various techniques such as Thermogravimetry (TG), Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC), Thermo Mechanical Analysis (TMA) etc. Thermal events are usually studied by the change in thermal property. The Thermogravimetric curve which records the loss of mass w.r.t. time and temp. not only give the mode of fragmentation of the sample on heating, but also provide a curve for characterization, comparison and identification of the sample.

The conclusion arrived at by other chemical & instrumental methods, can be refined on the basis of DTG, DTA & DSC analysis.

In the development of DTA technique, Le Chatelier¹⁸⁵³ was the first to record temp. as a function of time in heating curves. The most widely used thermal analysis techniques are :

1. Thermogravimetry (TG) : This is characterized by recording a change in the weight of the substance as a function of temperature.
2. Derivative thermogravimetry (DTG) : This method involves recording of derivative of change in the weight of the substance as a function of temperature.
3. Differential Thermal Analysis (DTA): This is characterized by recording the difference in temperature between a substance and an inert reference material as a function of temperature.
4. Derivative Differential Thermal Analysis (DDTA) : This method records the first derivatives of the difference in the temperature between a substance and an inert reference material as a function of temperature.

These methods are based on the fact that every substance is having a definite free energy, G given by

$$G = H - TS$$

Where H = enthalpy

T = thermodynamic temperature

S = entropy

Every system tends to attain the minimum free energy to become more stable. The formation of more stable solid or another state with lower free energy may take place on gradually heating the sample via intermediate steps.

The origin of thermogravimetry (TG) has been described by Duval, Keatch, Dallimore and Wendlandt which, later on, has emerged as an important technique for the scientific investigations. The most significant contribution was made by Honda in 1915 who used a lever arm balance fitted with an electric furnace to investigate manganese oxy salts. Basically thermogravimetric analysis (TGA) is a type of testing performed on sample that determines change in the weight in relation to change in temperature.

Methodology :

Analysis is carried out by raising the temperature of the sample gradually and plotting weight (percentage) against temperature. The temperature in many testing methods routinely reaches 1000°C or greater. After the data are obtained, curve smoothing and other operations may be done to find the exact points of inflections.

Thermogravimetric analysis uses heat to force reactions and physical changes in materials. TGA provides quantitative measurements of mass change in materials associated with transition and thermal degradation. TGA records change in mass as a result of dehydration, decomposition and oxidation of sample with time and temperature. Characteristic thermogravimetric curves are given for specific materials and chemical compounds due to unique sequences from physico-chemical reactions occurring over specific temperature ranges and heating rates. These unique characteristics are related to the molecular structure of the sample.