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Appraisal of thermal methods used in the analysis of API and Pharmaceutical Raw Materials

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Abstract

This review provides an overview of the application of thermal analysis techniques in the characterization of Active Pharmaceutical Ingredients (APIs) and pharmaceutical raw materials. Thermal analysis, encompassing methods such as Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), Thermogravimetric Analysis (TGA), Thermomechanical Analysis (TMA), Dynamic Mechanical Analysis (DMA), Thermo-microscopy or Hot Stage Microscopy (HSM), Melting Point (MP), plays a crucial role in understanding the physicochemical properties, stability, and compatibility of these materials. These techniques offer valuable insights into various thermal events, including melting points, glass transition temperatures, crystallization, decomposition, and polymorphism. By precisely measuring changes in heat flow, mass, and other thermal properties as a function of temperature or time, researchers can assess the purity, identify different solid forms, determine moisture content, and evaluate the thermal stability of APIs and excipients. Furthermore, thermal analysis is instrumental in pre-formulation studies for predicting drug-excipient interactions, optimizing manufacturing processes, and ensuring the overall quality and shelf-life of pharmaceutical products. The data obtained from thermal analysis is essential for regulatory submissions and for establishing robust quality control parameters throughout the drug development lifecycle.

Keywords: Thermal Analysis, Differential Scanning Calorimetry, Thermogravimetry, Melting Point

1. Introduction

Analysis of Active Pharmaceutical Ingredients (APIs) and Pharmaceutical Raw Materials (PRMs) can be done employing either thermal processes/methods or non-thermal methods.

Thermal analytical methods are defined as a group of physical-chemical methods that measure the properties of studied materials as a function of temperature or time while the sample is placed under a controlled temperature program [1]. International Confederation of Thermal Analysis and Calorimetry (ICTAC) defined thermal analysis as a group of techniques in which a physical property of a substance is measured as a function of temperature whilst

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the substance is subjected to a controlled temperature program. This controlled temperature program may be dynamic (heating and cooling), isothermal (fixed temperature) or a combination of both [1,2,3]. Some of the thermal methods used in the analysis of APIs and PRMs include Differential Scanning Calorimetry (DSC), Differential Thermal Analysis (DTA), Thermogravimetric Analysis (TGA), Thermomechanical Analysis (TMA), Dynamic Mechanical Analysis (DMA), Thermo-microscopy or Hot Stage Microscopy (HSM), Melting Point (MP).
Thermal analytical methods have found numerous applications in Pharmaceuticals as shown in Table 1. below;

Table 1. Applications of Thermal Analytical Methods in Pharmaceuticals

Pharmaceutical Field	Applications
Active Pharmaceutical Ingredients and Pharmaceutical Raw Materials	Polymorphism, Melting Point, Glass Transition of Amorphous Fractions, Content Determination, Effect of Moisture, Existence of Solvates and Purity Analysis
Formulations	Compatibility of Excipients, Shelf Life, Thermal Degradation, Moisture Determination
Packaging Materials	Testing of Primary and Secondary Packaging Materials, Thermal Stability, Moisture Determination, Coatings, Blister Package Interactions, Effect of Repackaging

These thermal analytical methods have a general advantage of utilizing only a small amount of the substance for the analysis.

1.1 Differential Scanning Calorimetry

Differential Scanning Calorimetry, (DSC), is a thermal analysis technique that looks at how a material's heat capacity (Cp) is changed by temperature. It is a technique used to study what happens to any material e.g. Active Pharmaceutical Ingredients and Pharmaceutical raw materials when they are heated under a controlled temperature. It was developed in 1962 by E.S. Watson and M.J. O'Neill. DSC is used to study the thermal transitions of a material i.e. the changes that happen in the material when it is heated.

A sample of known mass is heated or cooled and the changes in its heat capacity are tracked as changes in the heat flow. This allows the detection of transitions such as melts, glass transitions, phase changes, and curing. Because of this flexibility, since most materials exhibit some sort of transitions, DSC is used in many industries, including pharmaceuticals, polymers, food, paper, printing, manufacturing, agriculture, semiconductors, and electronics. DSC is a popular thermo-analytical technique ranging from the pharmaceutical science to applied research [1].

DSC monitors the difference in the amount of required heat to increase the temperature of a sample and reference (which should have an acceptable heat capacity in the range of scanned temperatures) as a function of temperature [4,5].

The figure 1 below shows a typical DSC setup comprising of two pans; one containing the sample while the other is the reference pan, the heat source and the computer to monitor and regulate the temperature and heat flow respectively.

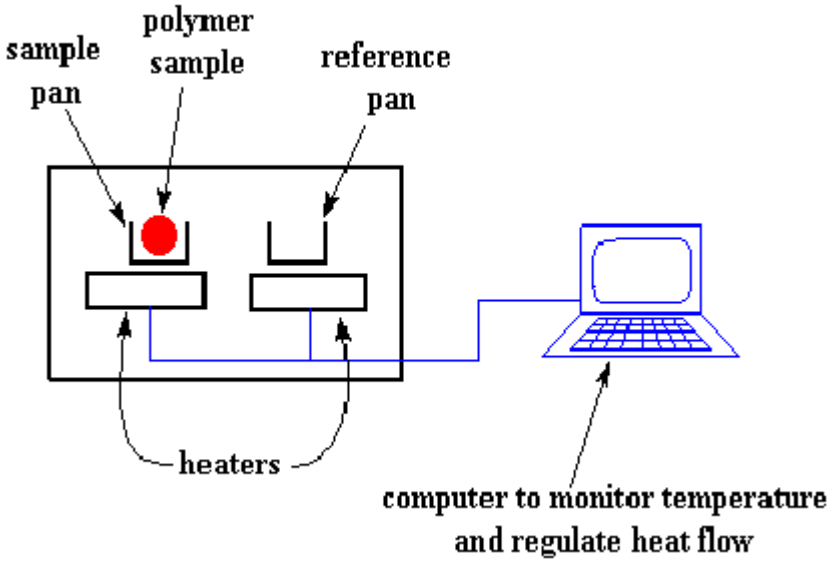

Fig.1 Typical Differential Scanning Calorimetry Setup

1.1.1 DSC Measurements

Heat Capacity: When the two pans (reference pan and sample pan) are heated, the computer will plot the difference in heat output of the two heaters against temperature i.e. the heat absorbed by the polymer against temperature. The plot will look like fig. 2 below.

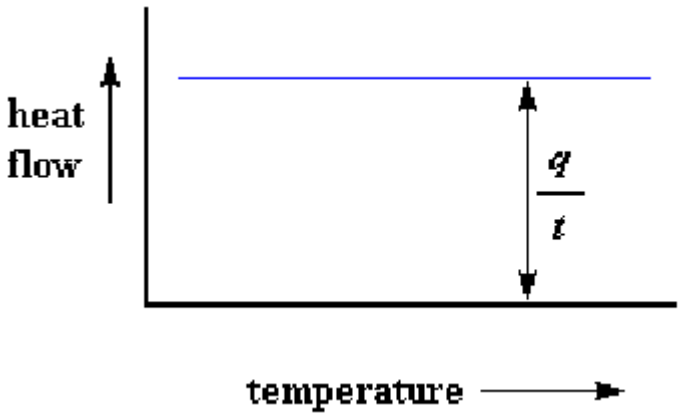

Fig.2 Thermograph of heat-capacity

When a certain amount of heat is put into something, its temperature will go up by a certain amount, and the amount of heat it takes to get a certain temperature increase is called the heat capacity represented as Cp. Heat capacity is gotten by dividing the heat supplied by the resulting temperature increase.

$$Cp = \frac{q}{\Delta T}$$

Where Cp is the heat capacity, q represents heat supplied while ΔT is the change in temperature.

The Glass Transition Temperature: As more heat is being applied to the polymer, after a certain temperature there will be an upward shift in the plot as shown in fig.3 below

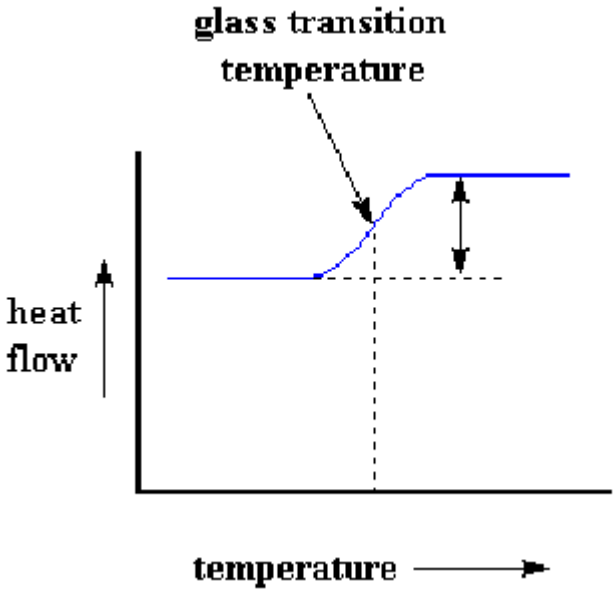

Fig.3 Thermograph of glass transition

This signifies that there is now an increased heat flow as well as an increase in the heat capacity of the polymer. This happens because the polymer has undergone a glass transition. Polymers have a higher heat capacity above the glass transition temperature than they do below the glass transition temperature. Because of this change in heat capacity that happens at glass transition, DSC can be used to measure the glass transition temperature of a polymer.

The Crystallization Temperature: Above the glass transition, the polymers have a lot of mobility, after a certain temperature, they will have gained enough energy to move into very ordered arrangements called crystals. When polymers fall into these crystalline arrangements, they give off heat. When this heat is dumped out, heater under the sample pan doesn't have to put out much heat to keep the temperature of the sample pan rising. This drop in the heat flow is observed as a big dip in the plot of heat flow versus temperature as shown below

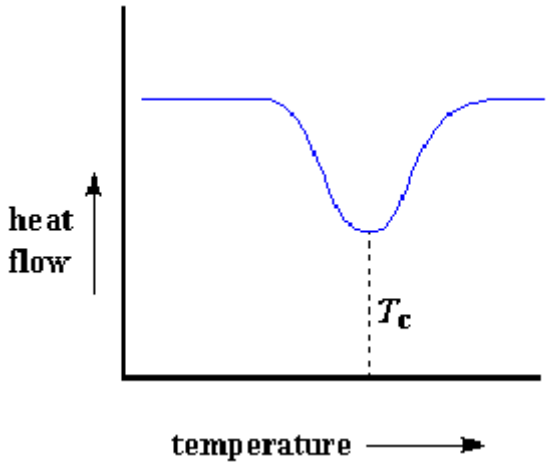

Fig.4 Thermograph of Crystallization temperature

The temperature at the lowest point of the dip is usually considered to be the polymer's crystallization temperature represented as Tc.

The melting temperature: As more heat is applied on the sample (polymer) beyond the crystallization temperature (Tc), another thermal transition process known as Melting occurs. The temperature at which this occurs is called the melting temperature represented as Tm. When the polymer crystals melt, they must absorb heat in order to do so. This means that when the melting temperature is reached, the polymer's temperature will not rise until all the crystals have melted. This means that the heater under the sample pan is going to have to put a lot of heat into the polymer in order to both melt the crystals and keep the temperature rising at the same rate as that of the reference pan. This extra heat flow during melting shows up as a big peak on our DSC plot as shown below;

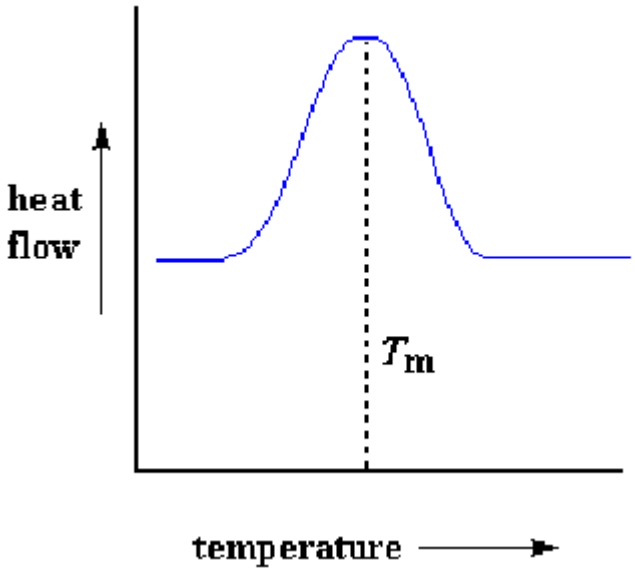

Fig.5 Thermograph of Melting temperature

1.1.2 Applications of DSC

Differential Scanning Calorimetry has found widespread applications in the following areas:

1. Purity determination of samples directly – DSC provides an easy and quick way of determining the purity of crystalline substances. A very small amount of the sample is required for the measurement. The purity evaluation runs automatically while the calculation is based Van't Hoff equation which takes the melting point depression due to the presence of impurities into account. Application of this method requires that the impurities will dissolve in the melt and are insoluble in the solid phase. [6]
2. Detection and quantification of polymorphism - Many pharmaceutical materials exhibit polymorphic behavior in which the substance may exist in several crystalline forms depending upon processing conditions. Differential Scanning Calorimetry (DSC) is a useful tool for detecting and quantifying these different forms.
3. Detection of meta stable polymorph - Both the stability of a chemical compound and its solubility in a given medium depend on the compound's structure. For example, in the development of a pharmaceutical substance it is important to identify possible polymorphs and assess their stability. Differential scanning calorimetry, or DSC, is often used for rapid polymorph detection.

4. Detection of isomerism – The linkage isomerization of chemical complexes in solid-state can be investigated using DSC.[7]
5. Stability/compatibility studies – DSC analysis is the most common thermal analytical method use in evaluating the drug-excipient compatibility studies.[8]
6. Percentage crystallinity determination - The assessment of a polymer's percent crystallinity can be most easily performed using differential scanning calorimetry (DSC) which measures the heat flow into or from a sample as it is either heated, cooled or under isothermally.[9]
7. Lyophilization studies – DSC has been said to be useful in determining the glass transition (Tg) of lyophilized products, to evaluate storage conditions.
8. Finger printing – DSC step annealing has been used to study the fine molecular structure of polyvinylidene fluoride (PVDF) homopolymers and copolymers. [10]
9. Solvent selection – DSC has been used as a useful thermal technique in solvent and non-solvent selection procedures.[11]

1.1.3 Advantages of Differential Scanning Calorimetry

The core advantages of DSC as a thermal analytical method include;

1. The ease and speed with which transition properties of pharmaceutical materials can be studied using DSC is its biggest advantage
2. The degree of purity in materials and phase changes or polymorphs of pharmaceuticals, liquid crystals, metals and pure organics can be studied with DSC

1.1.4 Disadvantages of Differential Scanning Calorimetry

1. Interpretation of result is often difficult
2. Quantitative analysis of individual processes is impossible
3. The technique is very sensitive is any changes

1.2 Differential Thermal Analysis (DTA)

Differential Thermal Analysis is a thermal analytical technique for measuring the heat release or absorption-induced temperature change of a sample during the programmed temperature-increasing process under a certain atmosphere, which is often used to determine the special temperature of a sample during endothermic or exothermic phase transition or reaction [12]. International Confederation of Thermal Analysis and Calorimetry (ICTAC) has defined Differential Thermal Analysis (DTA) as a thermal analytical technique in which the difference in temperature between the sample and a reference material is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed.

The disadvantage of DTA lies in that it is difficult to quantitatively obtain mass change during the test, such as chemical composition change and content (e.g., moisture) of the sample [12].

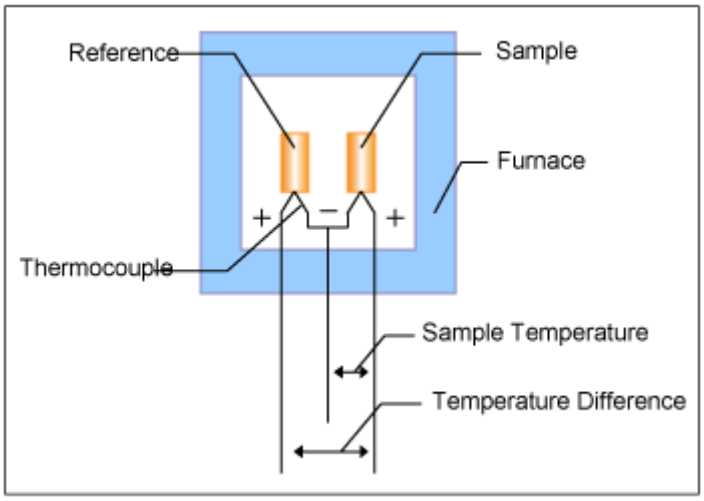

Fig. 6 Block diagram of DTA

The sample and the reference are placed symmetrically in the furnace. The furnace is controlled under a temperature program and the temperature of the sample and the reference are changed. During this process, a differential thermocouple is set up to detect the temperature difference between the sample and the reference. Also, the sample temperature is detected from the thermocouple on the sample side.

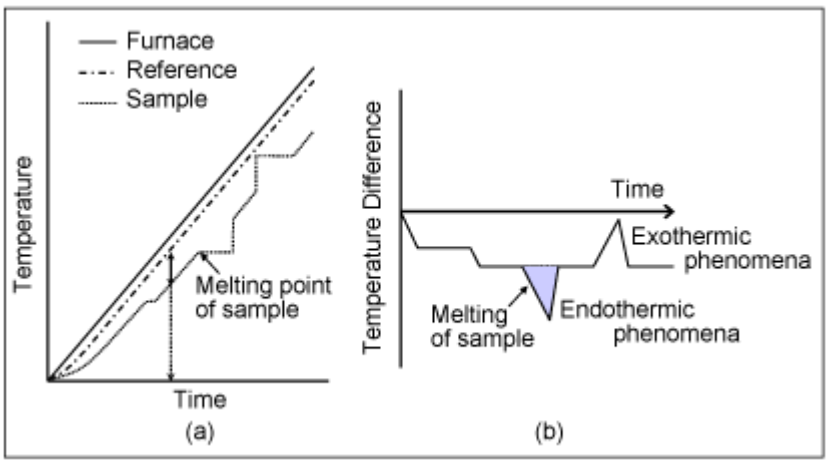

Figure 7 Measurement principles of DTA

Graph (a) shows the temperature change of the furnace, the reference and the sample against time.
Graph (b) shows the change in temperature difference (ΔT) against time detected with the differential thermocouple.
ΔT signal is referred to as the DTA signal.
Matters that do not change in the measurement temperature range (usually α-alumina) are used as reference.
When the furnace heating begins, the reference and the sample begin heating with a slight delay depending on their respective heat capacity, and eventually heat up in according to the furnace temperature.
ΔT changes until a static state is reached after the heating begins, and after achieving stability, reaches a set amount compliant with the difference in heat capacity between the sample and the reference. The signal at the static state is known as the baseline.

When the temperature rises and melting occurs in the sample, for example, the temperature rise stops as shown in graph (a) and the ΔT increases. When the melting ends, the temperature curve rapidly reverts to the baseline. At this point, the ΔT signal reaches the peak, as shown in graph (b). From this, we can detect the sample's transition temperature and the reaction temperature from the ΔT signal (DTA signal).
In graph (b), the temperature difference due to the sample's endothermic change is shown as a negative direction and the temperature difference due to the sample's exothermic change is shown as a positive direction.

1.3 Thermo-Gravimetric Analysis (TGA)

Thermo-gravimetric Analysis is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. It can also be defined as a technique in which, upon heating a material, its weight increases or decreases. This simply means that TGA is used to measure a sample's weight as it is heated or cooled in a furnace. TGA measures weight changes in a material as a function of temperature (or time) under a controlled atmosphere. Its principle uses include measurement of a material's thermal stability, filler content in polymers, moisture and solvent content, and the percent composition of components in a compound.

1.3.1 Principle of operation of TGA

A typical TGA instrumentation consists of a sample pan that is supported by a precision balance. That pan resides in a furnace and is heated or cooled during the experiment. The mass of the sample is monitored during the experiment. A sample purge gas controls the sample environment. This gas may be inert or a reactive gas that flows over the sample and exits through an exhaust.

A TGA analysis is performed by gradually raising the temperature of a sample in a furnace as its weight is measured on an analytical balance that remains outside of the furnace. In TGA, mass loss is observed if a thermal event involves loss of a volatile component. Chemical reactions, such as combustion, involve mass losses, whereas physical changes, such as melting, do not. The weight of the sample is plotted against temperature or time to illustrate thermal transitions in the material – such as loss of solvent and plasticizers in polymers, water of hydration in inorganic materials, and, finally, decomposition of the material.

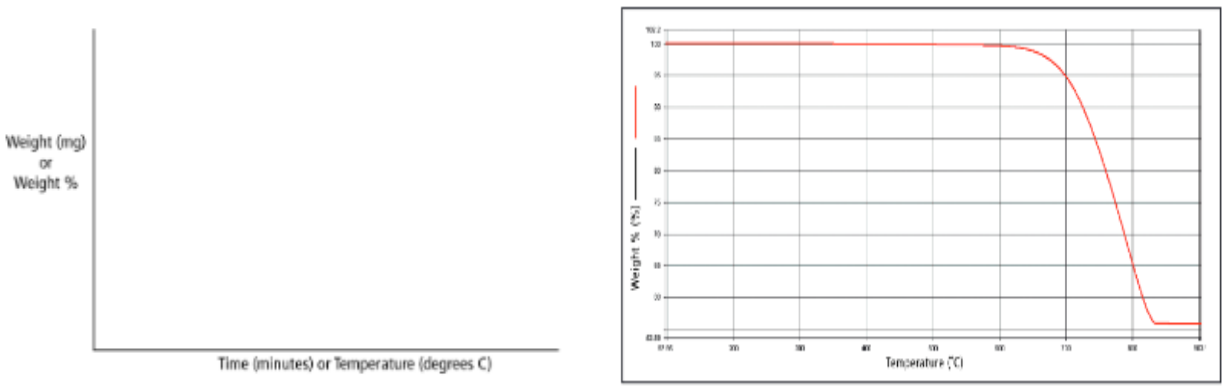

Fig.8 Typical plot of a TGA curve

1.3.2 Applications of TGA

Principle uses of TGA include measurement of a material's thermal stability and its composition. Typical applications include:

- Filler content of polymer resins

- Residual solvent content
- Carbon black content
- Decomposition temperature
- Moisture content of organic and inorganic materials
- Plasticizer content of polymers
- Oxidative stability
- Performance of stabilizers
- Low molecular weight monomers in polymers

1.4 Thermo-Mechanical Analysis (TMA)

TMA is a thermal analytical technique in which a deformation of the sample under non-oscillating stress is monitored against time or temperature while the temperature of the sample, in a specified atmosphere, is programmed. The stress may be compression, tension, flexure or torsion. TMA provides valuable characterization information on the dimensional properties of a wide range of materials. The technique provides a large amount of valuable information on polymers and other materials that is difficult or even impossible to obtain by other analytical techniques. TMA offers a higher degree of sensitivity compared to DSC for the detection of the glass transition temperature of highly filled or highly cross-linked materials, such as composites.

1.4.1 Principles of TMA

The TMA allows you to measure quickly and easily the change of dimension of the sample (expansion, shrinkage, movement, etc.) as a function of temperature, time and applied force. The probes and the media sample are generally quartz. The geometry of these parts requires a mode of measurement. The possible modes are the following:

- Expansion (probe used no force on the sample) for the determination of coefficient of expansion in dilatométrie.
- Penetration; a strong constraint, created by a strong force applied by a probe of small diameter, increases the contribution of the penetration relative to the expansion.
- Traction (sample attached by two small clamps) to the study of films or fibers under traction.
- Three-point bending (assembly consisting of support in the form of a knife).
- Volume expansion (mounting consisting of a crucible and a tube with a flat bottom) for the study of the dilation of powders, for example.

For the modes in both penetration and bending, a stress that is more important is imposed to the sample.

1.4.2 TMA Instrumentation:

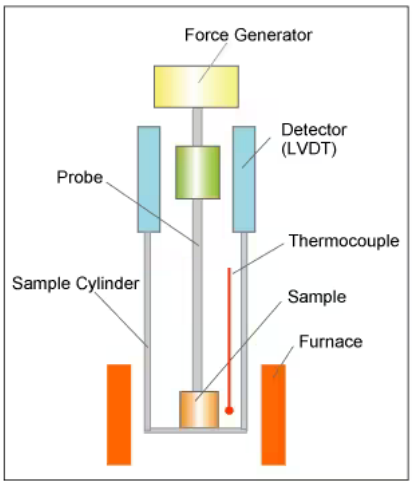

Fig. 9 Block Diagram of TMA

The sample is inserted into the furnace and is touched by the probe which is connected with the Length Detector and the Force Generator. The thermocouple for temperature measurement is located near the sample.
The sample temperature is changed in the furnace by applying the force onto the sample from the Force Generator via probe.
The sample deformation such as Thermal Expansion and Softening with changing temperature is measured as the probe displacement by the Length Detector. Linear Variable Differential Transformer (LVDT) is used for Length Detection sensor.
There are several types of the probe for TMA. The choice is dependent on the measurement purpose.

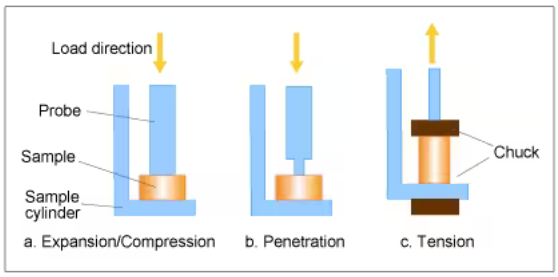

Fig.10 TMA Probe types

(a) Expansion/Compression Probe: It is used for the measurement of the deformation by the thermal expansion and the transition of the sample under the compressed force is applied.

(b) Penetration Probe: It is used for the measurement of the softening temperature.

(c) Tension Probe: It is used for the measurement of the thermal expansion and the thermal shrinkage of the sample such as the film and the fiber.

The materials of probes are quartz glass, alumina, and metals. The choice is dependent on the temperature range and/or the measurement purpose.

1.5 Dynamic Mechanical Analysis

Dynamic Mechanical Analysis (DMA) is a thermal analytical technique in which the sample's kinetic properties are analyzed by measuring the strain or stress that is generated as a result of strain or stress, varies (oscillate) with time, applied to the sample. It is also a technique where a small deformation is applied to a sample in a cyclic manner which allows the materials response to stress, temperature, frequency and other values to be studied.

DMA is also referred to as Dynamic Mechanical Thermal Analysis (DMTA). It is an important technique used to measure the mechanical and viscoelastic properties of materials such as thermoplastics, thermosets, elastomers, ceramics and metals.

In DMA, the sample is subjected to a periodic stress in one of several different modes of deformation (bending, tension, shear and compression).

Modulus as a function of time or temperature is measured and provides information on phase transitions.

DMA technology is the perfect solution when maximum accuracy is required and the material has to be characterized over a wide range of stiffness and/or frequency. In addition, DMA technology is extremely versatile and therefore, DMA can characterize materials even in liquids or at specific relative humidity levels.

1.5.1 DMA Instrumentation:

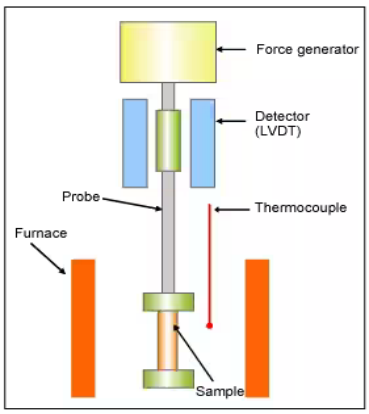

Fig.11 Block Diagram of DMA (Tension mode)

The sample is clamped in the measurement head of the DMA instrument. During measurement, sinusoidal force is applied to the sample via the probe. Deformation caused by the sinusoidal force is detected and the relation between the deformation and the applied force is measured. Properties such as elasticity and viscosity are calculated from the applied stress and strain plotted as a function of temperature or time.

DMA is used for measurement of various types of polymer materials using different deformation modes. There are tension, compression, dual cantilever bending, 3-point bending and shear modes, and the most suitable type should be selected depending on the sample shape, modulus and measurement purpose.

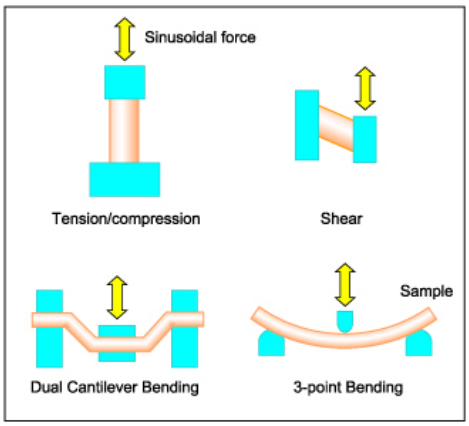

Fig.12 DMA Deformation modes

Analysis of the glass transition temperature and temperature dependence of the modulus can be measured by the temperature dispersion measurement. By performing simultaneous measurement of temperature dispersion and frequency dispersion, relaxation phenomena such as glass transition, side-chain relaxation and local mode relaxation can be observed. This approach allows us to obtain information about molecular structure and molecular motion of polymers.

1.6 Hot Stage Microscopy (HSM)

Fig.13 Image of a modern Hot-stage microscope

Hot stage microscopy (HSM) also known as thermo-microscopy is a thermal analytical technique that combines the best properties of thermal analysis and microscopy for the solid-state characterization of materials as a function of temperature and time. It is rapidly gaining interest in pharmaceuticals as well as in other fields as a regular characterization technique.

Hot-stage microscopy is a powerful method which is widely used to visually examine all kinds of thermal transitions when the sample is heated or cooled. In the heart of all hot-stage systems is the furnace with a heating element

beneath and above the sample, which guarantees outstanding temperature uniformity in the sample. HSM can be used with a variety of methods, such as Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, polarized light microscopy, and even X-ray and SAX/WAX.

The introduction of computer-controlled programmable HSM offers further advantages when coupled with data analysis software. Such software can allow researchers to run multiple heating and cooling cycles during the same thermal experiment, enabling clearer results that offer a more complete picture during in-situ experiments. [13]

HSM has many uses in pharmaceutical and biopharmaceutical research, such as obtaining data on the morphology of pharmaceutically relevant compounds such as active pharmaceutical ingredients (APIs) and interactions with the excipients. HSM allows researchers to observe the crystallization process, melting/boiling points, polymorphisms, desolvation, and glass transitions, amongst other transitions that may take place within a sample. [13]

1.7 Melting Point Analysis (MPA)

Melting point (Mp) is a quick and easy analysis that may be used to qualitatively identify relatively pure samples (approximately <10% impurities). It is also possible to use this analysis to quantitatively determine purity. Melting point analysis, as the name suggests, characterizes the melting point, a stable physical property, of a sample in a straightforward manner, which can then be used to identify the sample.

MPA is fairly specific and accurate given its simplicity. Because melting point is a unique physical characteristic of a substance, melting point analysis does have high specificity. Although, many substances have similar melting points, so having an idea of possible chemicals in mind can greatly narrow down the choices.

1.7.1 Advantages of MPA

Melting point analysis is a quick, relatively easy, and inexpensive preliminary analysis if the sample is already mostly pure and has a suspected identity. Additionally, analysis requires small samples only.

1.7.2 Limitations of MPA

As with any analysis, there are certain drawbacks to melting point analysis. If the sample is not solid, melting point analysis cannot be done. Also, analysis is destructive of the sample. For qualitative identification analysis, there are now more specific and accurate analyses that exist, although they are typically much more expensive. Also, samples with more than one solute cannot be analyzed quantitatively for purity.

2. Conclusion

In conclusion, thermal analysis methods are indispensable tools in pharmaceutical research and development, offering comprehensive characterization of APIs and raw materials. Their ability to provide critical information on thermal behavior, stability, and intermolecular interactions makes them vital for ensuring the quality, safety, and efficacy of pharmaceutical products from early development stages through to manufacturing and beyond.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest is to be disclosed

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