Did you get all Information from your Sample?

Gisbert Naundorf [a], Tim Mann [b], Paul Gabbott [b]

[a] PerkinElmer (Schweiz) AG, Bahnstrasse 8, 8603 Schwerzenbach

[b] PETA Solutions, Beaconsfield, UK

Increasing the sensitivity and throughput of Differential Scanning Calorimetry (DSC) analysis has always been a challenge for thermal analysts in research and development. A relatively fast temperature scanning rate (20°C/min or 40°C/min) using traditional DSC has been sufficient for most applications. However, DSC analysis becomes difficult when a sample size is small due to the weak signal. Slow heating rates allow many materials to change, some materials may experience re-crystallization or annealing during the slow heating process, and they may dry out or decompose. This causes difficulty in obtaining a true analysis of the original sample.

HyperDSC ™ is a new DSC technique recently introduced by PerkinElmer. With fast scanning rates up to 500°C/min this produces vastly increased sensitivity and permits analysis of materials without change due to annealing or re-crystallization phenomena. With very fast temperature scanning rates in cooling and as well as in heating, sensitivity have dramatically increased by a factor of ten over most conventional DSC analysis. Due to the fast scanning rate, the re-crystallization during melting, decomposition after melting, and unknown thermal behavior are either completely eliminated or significantly reduced by this new method.

To achieve the fast scanning rates used in HyperDSC, ultra low-mass furnaces (1 g) and small dimensions are required to ensure the system is under control when making measurements at rates of 200-50°C/min. A power compensation DSC is the only system able to provide all the features and to demonstrate this technique in practice.

To obtain the most data from HyperDSC, the analyzer should have the ability to collect and measure data very quickly at the beginning of the scan by having small thermal transients, preferably of 12 sec or less. This means that it will quickly collect meaningful data without having to start at very low temperatures.

Because of the large heat flows of transitions that are associated with fast scan rates, it is vital that a large dynamic range is available during the measurements. It is possible that unless the instrument chosen allows a wide dynamic range, the analyzer signal may be swamped by the size of the transitions, which may be over 150 mW in size.

In this contribution, we will show the HyperDSC technique in detail and the technical aspects, and demonstrate the benefits of this method for several applications in pharmaceutical and relevant application areas, like analysis of Polymorphism and small amorphous content of pharmaceutical materials.

References

1. Thijs F.J.Pijpers, Vincent B.F.Mathot, Bart Goderis, Rolf L.Scherrenberg, Eric W. van der Vegte; Macromoelecules 202, 35, 361-3613.
2. James L Ford, Tim Mann; HyperDSC of Polymorphic Transition in Nifedipine. J Pharm Pharmacol 202; 54: S1