APPLICATION NOTE

Thermal Analysis

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StepScan DSC for Obscured Transitions

StepScan DSC is a temperature modulated DSC technique that operates in conjunction with the Power Compensation Diamond DSC from PerkinElmer. The approach applies a series of short interval heating and isothermal hold steps to cover the temperature range of interest. With the StepScan™ DSC approach, two signals are obtained: the *Thermodynamic Cp* signal represents the thermodynamic aspects of the material, while the Iso K signal reflects the kinetic nature of the sample during heating. The following basic equation mathematically describes the StepScan DSC approach:

$$\text{Heat Flow} = Cp(dT/dt) + f(T,t)$$

In this equation, Cp is the sample's heat capacity, dT/dt is the applied heating rate and f(T,t) is the kinetic response. The first Cp term represents the thermodynamic aspects of the sample and, while the Power Compensation DSC applies a purely linear heating ramp for the best results rather than a sine wave where the heating rate is continuously varying. When the sample is held under isothermal conditions, as does take place with the Power Compensation DSC and the StepScan DSC approach, the heating rate becomes 0 and the sample's heat flow is purely described by the kinetic term. Because the sample is either *linearly* heated or held *isothermally* (true isothermal), the StepScan DSC approach is straightforward and provides the purest approach to TMDSC measurements.

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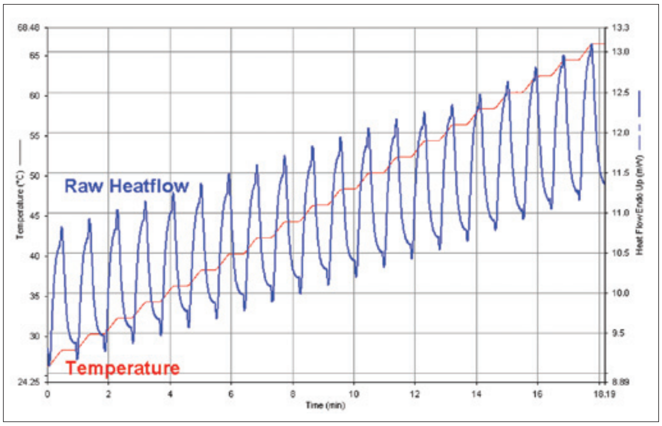

Figure 1. A StepScan run showing the temperature cycle and the resultant heat flow.

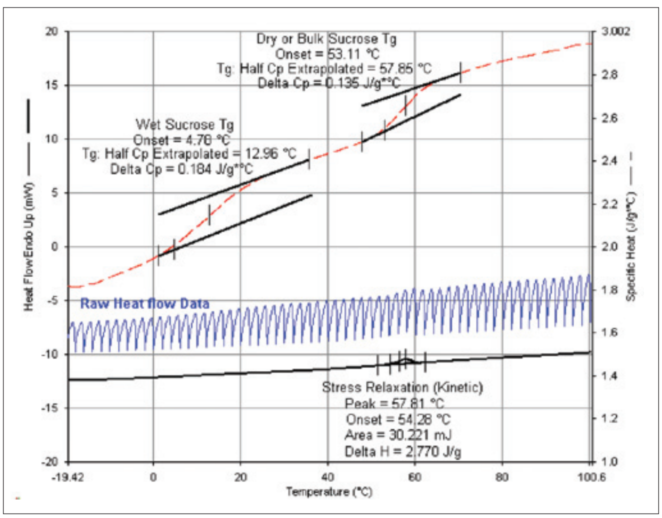

Figure 2. StepScan run on a cough drop (5.8 mg) with 2 degree temperature jumps increases, ramped at 5 °C/min and followed by 30 sec isothermal holds. Nitrogen was used for the purge gas with automated LN2 cooling.

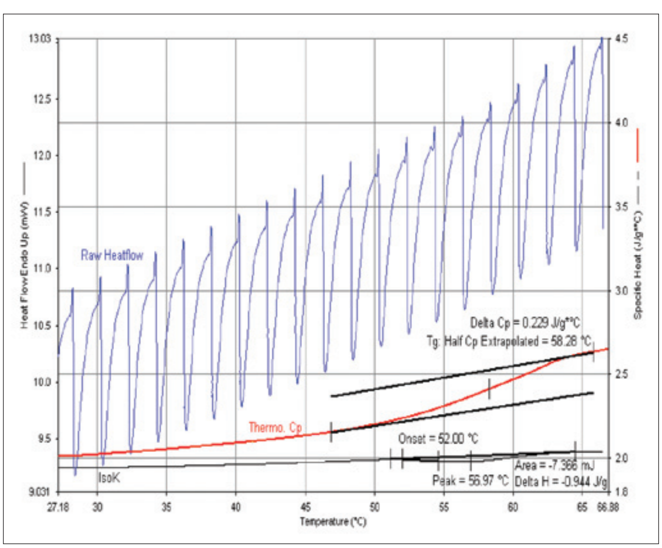

Figure 3. A 6.1 mg sample of a sucrose-BSA lyophilized formulation was run under the same conditions as in Figure 2. The glass transition is easily seen in the thermodynamic Cp curve.

The Tg of a sample can frequently be obscured or masked with another simultaneously occurring event, such as enthalpic relaxation, evolution of moisture, crystallization, or stress relief. StepScan DSC separates out the Tg from these kinetic effects making the identification of the Tg much easier and more readily identifiable. The DSC results shown in Figure 2, are those of a cough drop. While conventional DSC shows a Tg that is masked by an endothermic enthalpic relaxation peak, the Step Scan DSC run separates this out from the Tg. In the Thermodynamic Cp curve, the Tg near room temperature reflects the water-plasticized outer surface of the cough drop, while the higher temperature Tg corresponds to the dry interior sucrose. The Iso K baseline shows the peak caused by the enthalpic overshoot in the sample.

For a 10% sucrose-bovine serum albumin (BSA) formulation, the Thermodynamic Cp data set reveals that a Tg's occur at approximately 65 °C. In addition, the Iso K baseline shows that an enthalpic overshoot is associated with this transition with a DH of 3.85 J/g.

This data can be obtained fairly quickly in one run. The same technique can be used at sub-ambient temperatures to measure the Tg'.

Summary/conclusions

StepScan DSC ability to clean up and see weak or obscured transitions makes it a powerful addition for pharmaceutical research, development, and quality control.

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