APPLICATION NOTE

Thermal Analysis

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HyperDSC for Detecting Pure Protein Tg's and Other Weak Transitions

Introduction

HyperDSC™ or High Ramp Rate DSC leads to a significant increase in apparent sensitivity due to the unique design of the power compensated DSC. One advantage of this technique is that very small sample sizes can be handled, such as pieces of bio-medical devices collected from inside a patient or very rare samples. However, it also increases the ability to detect very weak thermal transitions from samples.

It has been suggested that certain materials, like pure proteins, have transitions too weak to be detected by Differential Scanning Calorimetry, because they are too low energy and/or too broad. HyperDSC provides a viable way of measuring these weak transitions. Similarly, TMA can be used to measure very weak transitions and to confirm the data collected by DSC.

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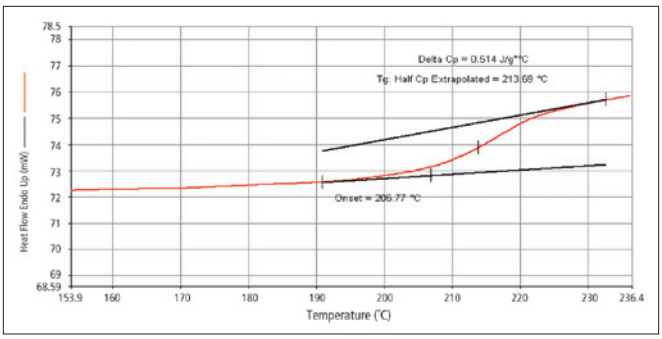

Figure 1. HyperDSC run on ova albumin at 100 °C/minute.

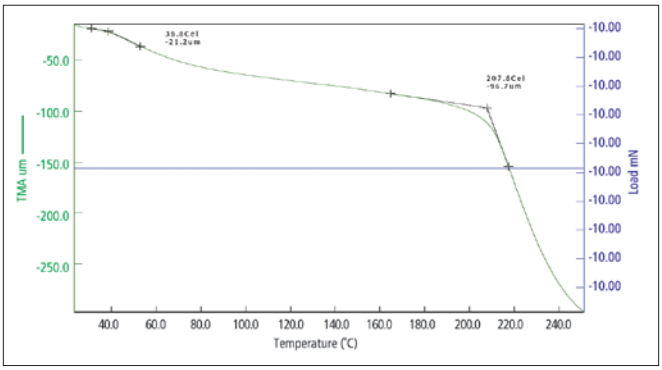

Figure 2. Ova albumin protein's Tg measured by the Diamond TMA.

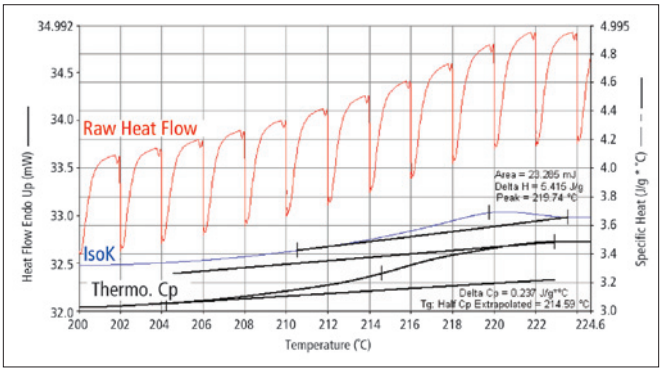

Figure 3. StepScan DSC of ova albumin lyophilized from DI water.

During our experiment, approximately 5 milligrams of pure protein, lyophilized from deionized water, were run at 100 °C/min in the Diamond™ DSC, using hermetically sealed aluminum pans. An Intercooler 2 was used for cooling, and a nitrogen purge was maintained. Figure 1, shows the resultant scan on ova albumin. When this run was repeated at 2 °C/minute, the transition was undetectable even with larger sample sizes. Similar results were obtained for dried samples of pure bovine serum albumin (197.88 °C), bovine gamma globulin (173.53 °C) and Ribonuclease (211.38 °C). We also applied this methodology to hydroxyethylstarch (HES: 271.97 °C). In order to confirm these results, the same samples were analyzed using the Diamond TMA, heating about 10 milligrams in a quartz dilatometer cup at 5 °C/min under Nitrogen purge. The resulting scans are shown in Figure 2 and correspond well with the data from the DSC. In addition, we also attempted StepScan™, a temperature modulated DSC technique, on these materials, heating for the 2 °C jump at 20 °C/minute and holding for 30 seconds. The resultant scan for ova albumin is shown in Figure 3. Again the protein Tg is visible.

Power compensation DSC combined with HyperDSC and StepScan provide a unique way to identify weak transitions for compounds traditionally thought to be undetectable by traditional, heat flux furnace, DSC. Transitions that are hard to detect in classical DSC are accessible by using more novel techniques like StepScan and HyperDSC.

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