



Determination of Fructose Equilibrium Melting Temperature by Rapid Heat-Cool (RHC) DSC

INTRODUCTION

As a technique, interest has been growing in performing differential scanning calorimetry (DSC) at higher than typical (10 °C/min) temperature-scanning rates. This is because a variety of material characterization challenges exist that can benefit dramatically from rapid heating or cooling rate experiments. For example, the investigation of metastable states and time-dependent transitions would profit greatly from fast scanning rates. In general, higher scanning rates will also increase the heat flow sensitivity for subtle transitions although this benefit is usually tempered by the small mass requirement of the rapid scanning rates.

A DSC has been designed specifically for operation at high scanning rates – up to 2000 °C/min in heating with similarly high cooling rates.¹ Key technologies introduced by TA Instruments are essential to, and have been incorporated into the instrument known as Project RHC. For example, Tzero® technology improves the resolution and the sensitivity of the measured sample heat flow rates, especially for very weak effects, and improves the instrument baseline. Also, infrared heating, introduced in the Q5000IR TGA, provides a “massless” infrared heat source. Readers interested in further details on the instrument design should refer to reference 1.

This applications note reports on the use of RHC-DSC in the investigation of the equilibrium melting temperature of fructose.

RESULTS and DISCUSSION

Upon heating, some materials will begin to decompose prior to melting. The decomposition endotherms can easily be misinterpreted as melting transitions if the baseline after the transition is well-behaved. However, since decomposition is a kinetic transition, the temperature at which it occurs is condition-dependent and will shift to higher temperatures at higher rates. As melting is an equilibrium thermodynamic process, the melting transition onset temperature should be rate-independent (assuming compensation for thermal lag within the instrument). Thus, experiments conducted at multiple heating rates are advisable to determine whether an endothermic transition is characteristic of melting or decomposition. Also, because of the large shifts in decomposition temperature induced by scanning at higher rates, the potential exists for elevating the decomposition to a temperature beyond the equilibrium melt temperature of the material, thereby allowing for the detection of the thermodynamic melt point. Figure 1 illustrates this concept.

Fructose is a naturally occurring carbohydrate, which decomposes prior to melting for typical DSC scan rates up to 20 °C/min. Figure 2 shows the results of scanning a fructose sample at elevated rates from 50 °C/min to 2000 °C/min. For rates up to 500 °C/min, the onset of the endotherm shifts significantly indicating that decomposition is responsible for the endotherm. For rates of 1000 °C/min and above, the negligible onset temperature shift indicates that the true melting of the sample is being

observed. From this data it is concluded that the equilibrium melting temperature of fructose is $\sim 136^\circ\text{C}$.

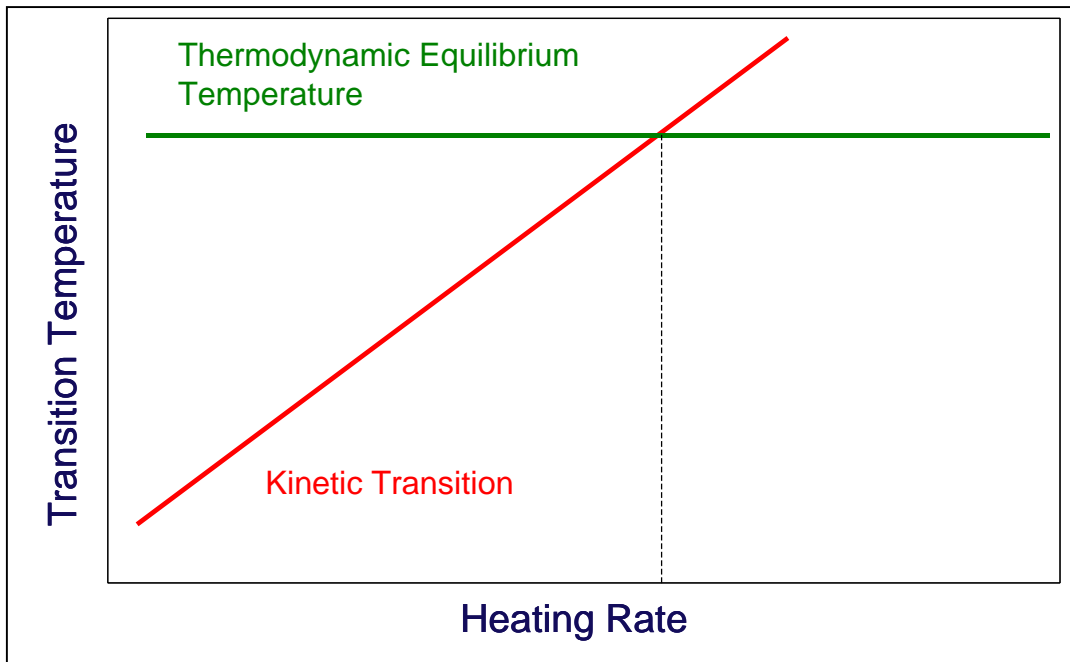


Figure 1 – Graph showing transition temperature dependence on heating rate for equilibrium processes versus kinetic processes.

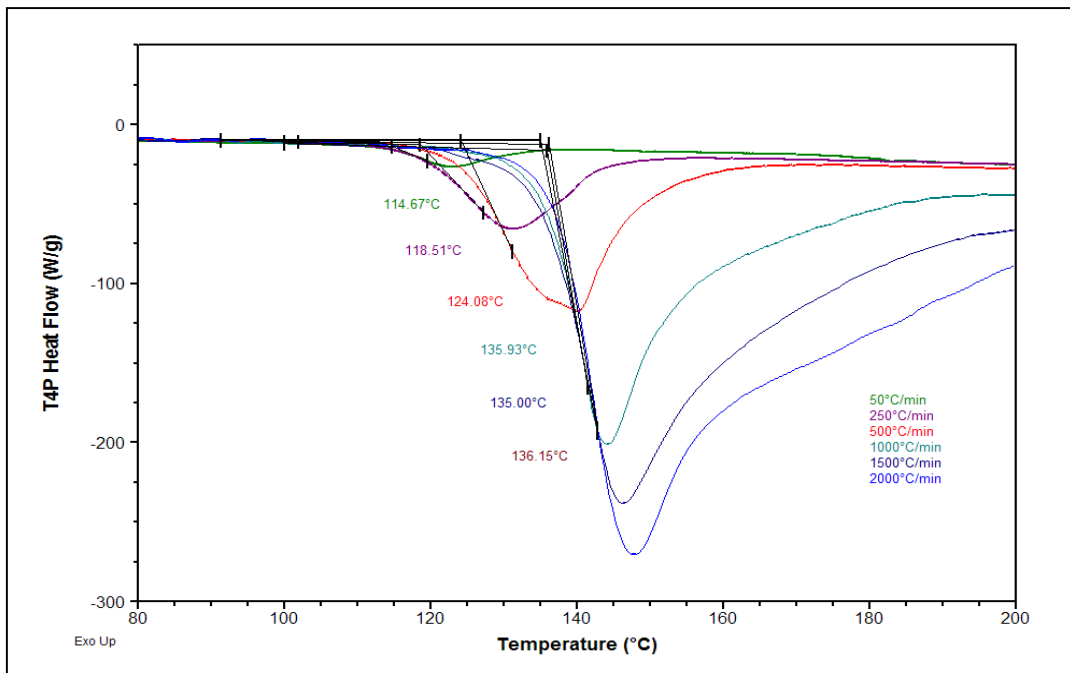


Figure 2 – DSC plots of fructose at various scan rates.

REFERENCES

1) Robert L. Danley, Peter A. Caulfield and Steven R. Aubuchon, *American Laboratory*, January 2008, pp. 9-11.

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