# PET Analysis by Rapid Heat-Cool 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 analysis of polyethylene terephthalate (PET), by rapid heat-cool (RHC) DSC.

## RESULTS and DISCUSSION

PET is a common thermoplastic polymer used in a variety of applications including plastic beverage bottles. It is easily quenched into a 100% amorphous state by rapid cooling from above its melt. Traditional (20 °C/min) DSC analysis of an amorphous sample of PET will include a glass transition around 80 °C, a cold crystallization exotherm near 120 °C and a melt with onset around 245 °C. This confirms the metastable nature of the amorphous PET sample on heating, which is common for many thermoplastic materials. In order to understand the structure of the as-received sample, high-scanning rates are required in order to suppress any kinetic activity.

Figure 1 contains an overlay of seven PET scans collected on a Q2000 DSC at successively higher rates, beginning at 1 °C/min up to 50 °C/min. The data are plotted in heat capacity units for comparison. Before each analysis, the sample was quench-cooled from the melt to ensure a 100% amorphous structure. As the heating rate is increased, the cold crystallization exotherm shifts to higher temperatures and its relative size (along with the corresponding melt peak), noticeably decreases. But even at 100 °C/min, there is still a detectable melt endotherm indicating some crystal structure has been formed on heating.

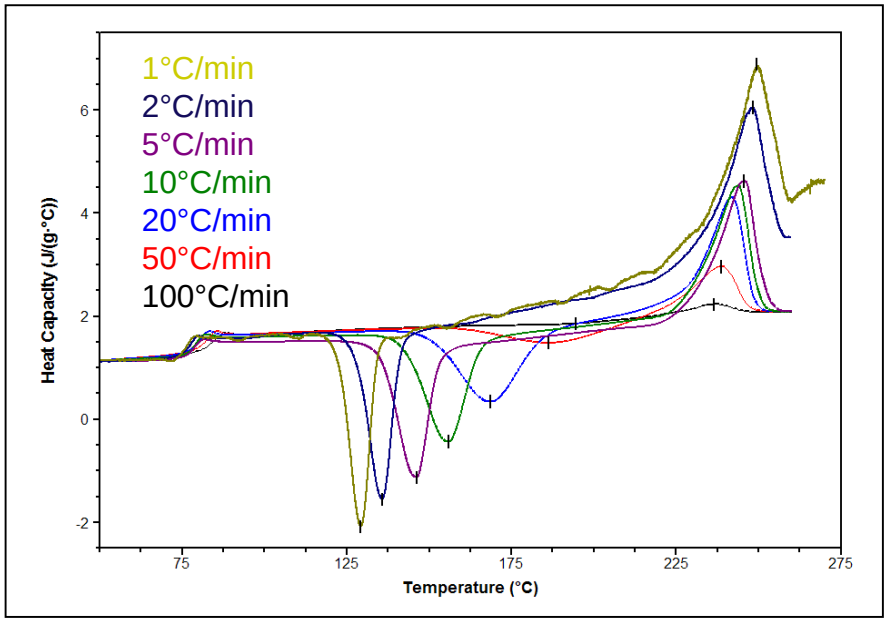


Figure 1. DSC data (Q2000DSC) of amorphous PET at a variety of heating rates

Figure 2 contains an overlay of two scans of PET collected at a rate of 500 °C/min on the RHC-DSC. As indicated, one sample was quench-cooled as in the above set of data. At this rate, the cold crystallization is completely suppressed and only the glass transition is detected. In order to demonstrate sensitivity for melting at these rates, a second sample was cooled from the melt at a rate of 10 °C/min. A reasonable crystal structure should have formed at these under these conditions. As expected, upon heating at the higher rate, a melt endotherm is detected. This data demonstrates the utility of the RHC in analysis of metastable structure in polymers. Only at elevated heating rates are the kinetic processes sufficiently suppressed, so that the original amorphous structure can be accurately measured.

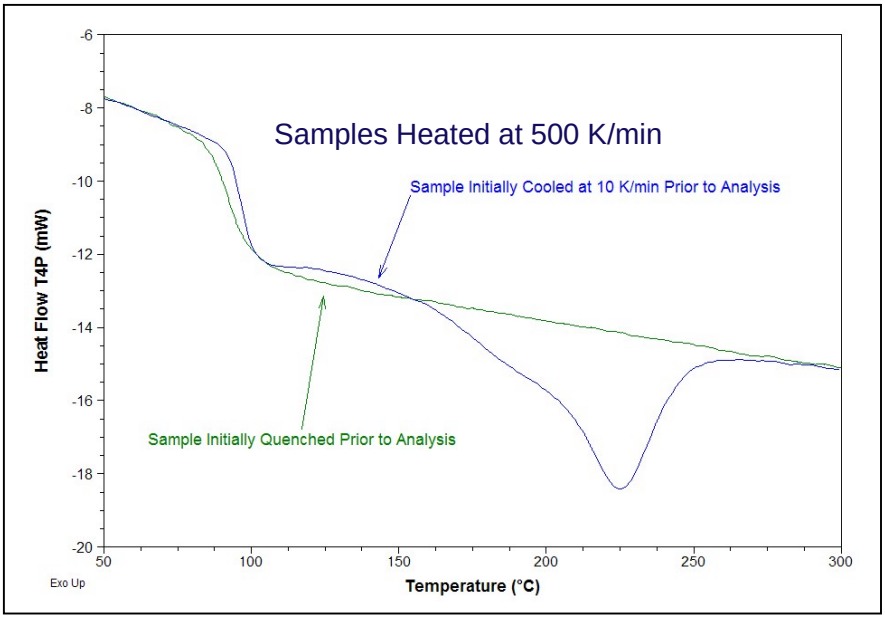


Figure 2. Scan of quenched and slow-cooled PET at 500°C/min.

## REFERENCES

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

## TA INSTRUMENTS

**United States**
109 Lukens Drive, New Castle, DE 19720 • Phone: 1-302-427-4000 • E-mail: info@tainstruments.com

**Canada**
Phone: 1-905-309-5387 • E-mail: shunt@tainstruments.com.

**Mexico**
Phone: 52-55-5200-1860 • E-mail: mdominguez@tainstruments.com

**Spain**
Phone: 34-93-600-9300 • E-mail: spain@tainstruments.com

**United Kingdom**
Phone: 44-1-293-658-900 • E-mail: uk@tainstruments.com

**Belgium/Luxembourg**
Phone: 32-2-706-0080 • E-mail: belgium@tainstruments.com

**Netherlands**
Phone: 31-76-508-7270 • E-mail: netherlands@tainstruments.com

**Germany**
Phone: 49-6196-400-7060 • E-mail: germany@tainstruments.com

**France**
Phone: 33-1-304-89460 • E-mail: france@tainstruments.com

**Italy**
Phone: 39-02-2742-11 • E-mail: italia@tainstruments.com

**Sweden/Norway**
Phone: 46-8-555-11-521 • E-mail: sweden@tainstruments.com

**Japan**
Phone: 813-5479-8418 • E-mail: j-marketing@tainstruments.com

**Australia**
Phone: 613-9553-0813 • E-mail: sshamis@tainstruments.com

**India**
Phone: 91-80-2839-8963 • E-mail: india@tainstrument.com

**China**
Phone: 8610-8586-8899 • E-mail: info@tainstruments.com.cn

**Taiwan**
Phone: 886-2-2563-8880 • E-mail: skuo@tainstruments.com

**Korea**
Phone: 82.2.3415.1500 • E-mail: dhrhee@tainstruments.com

**To contact your local TA Instruments representative visit our website at www.tainstruments.com**