SII NanoTechnology Inc.

# TG/DTA AS PROBLEM-SOLVING TOOL: SIMULTANEOUS MEASUREMENT OF TGA AND DSC SIGNALS

## Problem

A customer in a thermal analysis laboratory wishes to conduct simultaneous measurements of both the TGA (thermogravimetric analysis) weight loss signal and DSC (differential scanning calorimetry) signals. The customer needs to do this for two reasons: economic, since only one instrument can be purchased; and, to better help in the interpretation of the data. For the latter reason, the use of simultaneous TGA/DSC ensures that the sample is exposed to the identical thermal treatment and environment and thus directly comparable data is obtained simplifying data interpretation.

## Solution

The Seiko EXSTAR TG/DTA6000 system provides the following key features:

- simultaneous measurement of TGA and DTA signals
- ability to convert, via imbedded software, the DTA signal to user-friendly DSC units
- high sensitivity TGA measurements
- stable baseline performance
- true horizontal purge for optimizing coupled techniques (TGA-MS or TGA-FTIR)
- auto stepwise isothermal analysis for the highest possible resolution of overlapping decomposition events
- automated 20-point temperature calibration for the highest possible accuracy
- automated 10-point DTA-DSC calibration
- direct temperature calibration from simultaneous DTA(DSC) signal

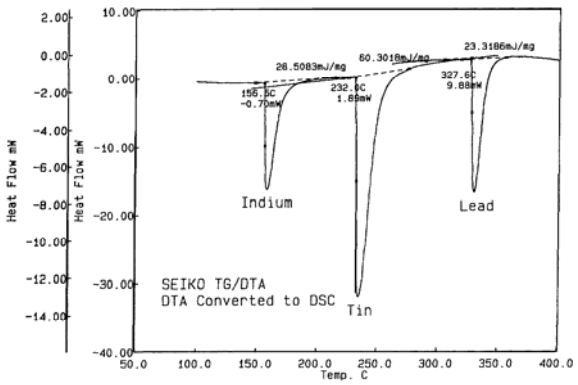


**Figure 1**



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Displayed in Figure 1 are the DTA signal results obtained from the Seiko TG/DTA for three high purity metal standards (indium, tin and lead). The DTA signal has been automatically converted to DSC output (mW) based on the heats of fusion of the metal standards. (The heats of melting of indium, tin and lead are 28.59, 60.62 and 23.22 mJ/mg, respectively.)

The simultaneous DTA or DSC signal permits the accurate and precise temperature calibration of the TGA signal, based on the melting onset temperatures. The use of the DTA or DSC onset temperature provides a greater degree of accuracy and precision than is possible with traditional Curie standards. Figure 1 shows that excellent accuracy is obtained from the Seiko TG/DTA for the three metal standards. The onset temperatures are all within 0.1°C of the literature values (156.6°C for indium, 232.0°C for tin and 327.5°C for lead). This high degree of accuracy is beneficial for certification purposes and gives the user additional confidence that the TGA results are indeed correct and accurate.

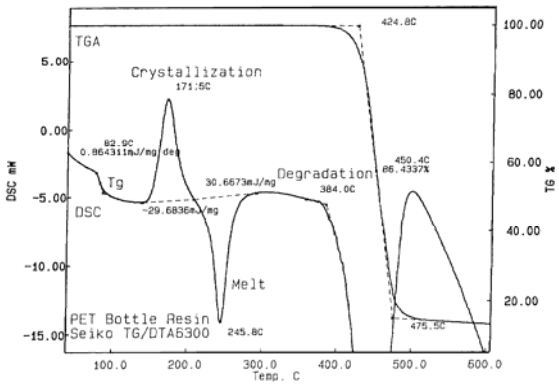


**Figure 2**

The benefits of the simultaneous TGA and DSC signals are shown in Figure 2. This plot displays the TGA weight loss and simultaneous DSC signal for PET (polyethylene terephthalate) resin. The PET polymer exhibits a single thermal decomposition mass loss beginning at 430.1°C and with a total weight loss of 86.26%.

With the Seiko TG/DTA, not only is the TGA weight loss obtained, but also the simultaneous DSC data as well as is shown in the lower

portion of Figure 2. The glass transition of the PET bottle resin is observed at 80.7°C, followed by an immediate cold crystallization event at 117.6°C with a heat of crystallization of 14.0 mJ/mg. The melting of the PET resin is observed at 247.7°C with a



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heat of melting of 37.3 mJ/mg. As the resin is heated well above the melting transition, an exothermic response is obtained in the DSC signal at 387.2°C. This represents the start of significant decomposition in the sample, even though the TGA signal is still relatively flat at this temperature. The DSC signal, obtained from the TG/DTA instrument, is a more sensitive indicator of the thermal stability of the resin.

## Summary

The Seiko TG/DTA6000 provides simultaneous measurement of the TGA and DTA signals providing directly comparable data thus aiding and simplifying data interpretation. The DTA signal can be automatically converted to user-friendly DSC units (mW) through the use of high purity metal standards.



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