Micro Reaction Calorimeter - µRC™
Technical Application Note 6
Melting Temperature and Enthalpy of Fusion

Introduction

Melting point standards can be used to check the temperature calibration of an instrument. Some compounds also have a known enthalpy of fusion which can be compared to a literature value as a further test of heat measurement accuracy.

The Micro Reaction Calorimeter (µRC™) from THT has a scanning function which allows the user to ramp the temperature from –10 to 160°C at a specified rate. This method can be used to determine the melting point of an unknown compound (see Figure 3 overleaf).

Scanning can also be used to measure the enthalpy of fusion based on the absorbed heat, and gain qualitative information on purity based on the shape of the peak.

Experimental

For a typical scan test, 19.4 mg indium was weighed into a sample vial and secured with a heat-resistant green lid. An empty vial, also equipped with a heat-resistant lid, was placed in the reference cell. A scan test was initiated starting at 100°C and ending at 160°C, with a ramp rate of 1°C/min.

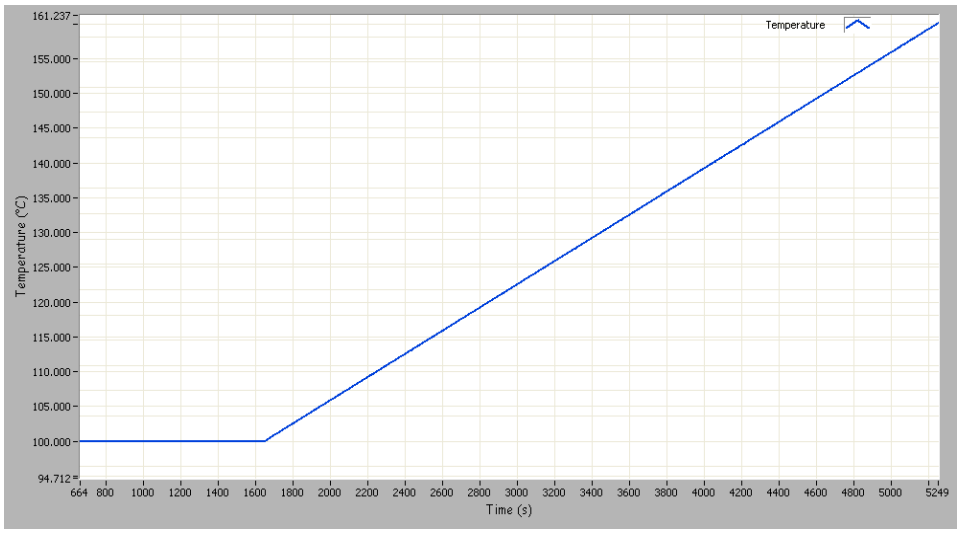

Figure 1: temperature signal from a scan test

Results

Figure 1 (left) shows the temperature data from the experiment. The temperature is held constant at 100°C then increases steadily up to 160°C.

Figure 2 (below) shows the power data from the experiment. The baseline shows a linear decline as the temperature decreases. The endothermic peak corresponds to heat absorption by the indium as it melts.

The integrated area of the peak in Figure 2 is 0.544 J. This can be divided by the mass of indium in the sample to give the enthalpy of fusion.

0.544 J / 0.0194 g = 28.0 J/g

This compares well to the literature value of 28.7 J/g [1].

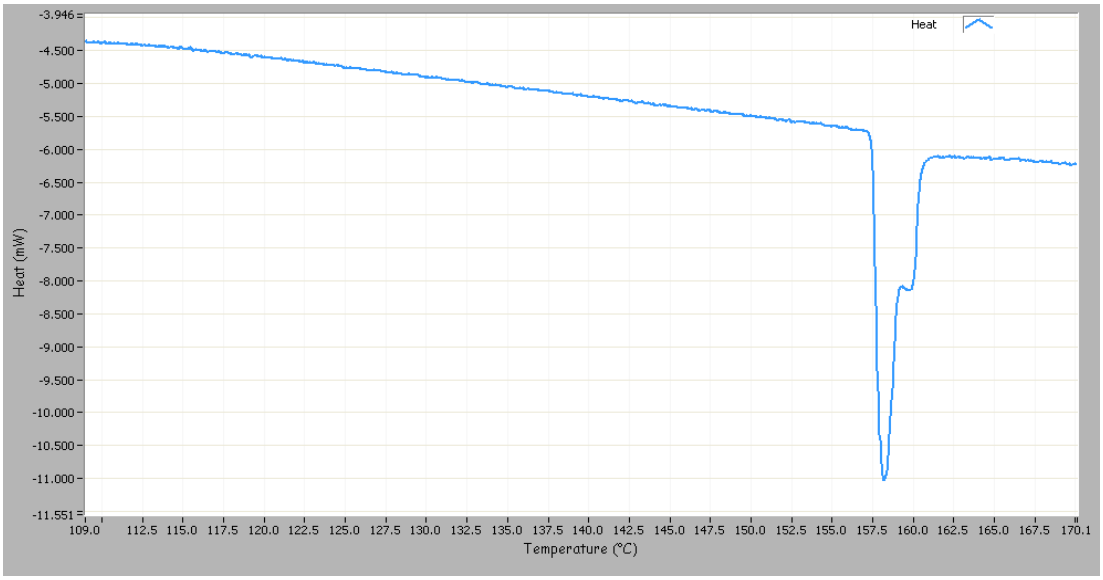

Figure 2: heat signal from a melting point scan

thermal hazard technology

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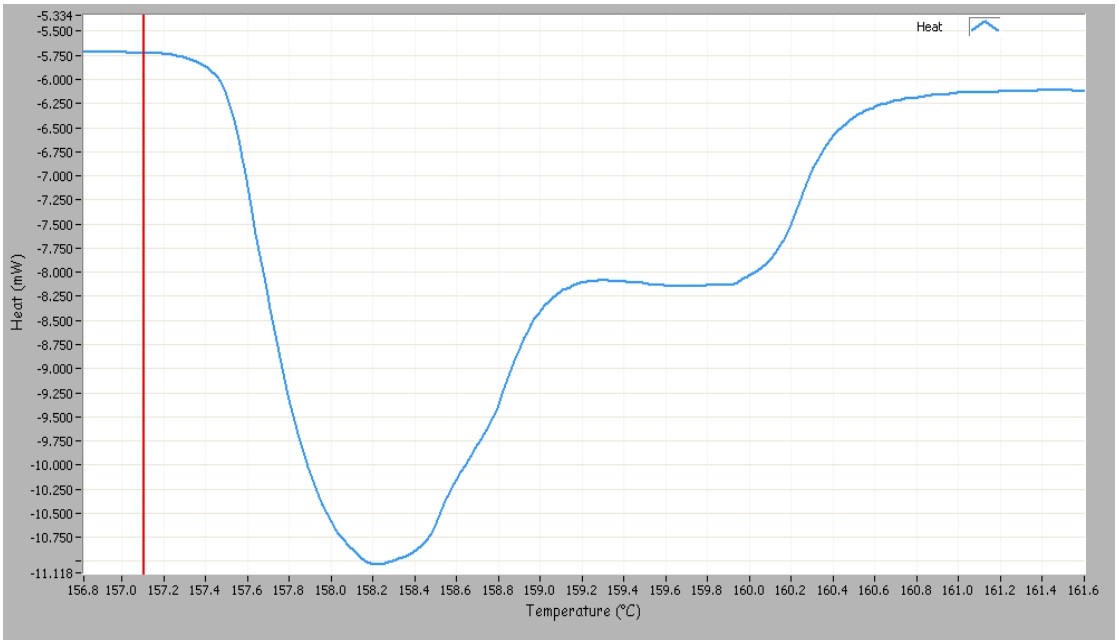

Figure 3: determination of the melting point of indium using scan data

The µRC Analysis software allows the heat signal to be viewed as a function of temperature or time. Figure 3 shows the heat/ temperature graph being used to determine the melting point of indium.

The melting point is defined as the temperature at which melting begins, which in terms of heat is the very start of the peak. This corresponds to a temperature of 157.1°C. The literature value is 156.6°C [2].

Discussion and Conclusions

It has been demonstrated that a simple, 60-minute scan test on the µRC™ can be used to obtain quantitative melting data from a small sample. The method is not limited to pure substances, and should be accurate with samples as small as 10 mg.

The sharp drop in the heat signal in Figure 3 at the beginning of the melting process suggests a compound of high purity. A more gradual decline, or a decrease in the melting point, would indicate impurities in the sample.

It should be noted that not all melting point standards are suitable for measurement of the enthalpy of fusion. Some solids may partially sublime, and the nature of the sealed vials in the µRC™ make the instrument unsuitable for determining enthalpies of sublimation.

Other melting point standards which have been used for temperature calibration testing include diphenyl ketone, benzoic acid, naphthalene and phenyl salicylate.

References

[1] D. G. Archer, S. Rudtsch; *J. Chem. Eng. Data*, **2003**, *48* (5), pp. 1157–1163

[2] **http://www.chemicalelements.com/elements/in.html**

Figure 4: the µRC™ available from THT