
DSC AS PROBLEM-SOLVING TOOL: ENHANCING MELTING PEAK RESOLUTION

Problem

Closely occurring DSC melting transitions can overlap each other and hinder good enthalpic characterization.

Solution

Resolution is the ability of any analytical instrument to separate two closely spaced events. In the case of DSC, good resolution is required when attempting to separate two, or more, closely occurring melting transitions.

When performing DSC experiments, better resolution on two closely spaced melting events can be obtained by:

- decreasing the heating rate
- decreasing the sample mass.

A 50% reduction in the heating rate will generally produce a greater degree of improvement in resolution as compared to a 50% reduction in mass.

To demonstrate these points, a sample of azoxyanisole (as obtained from the Aldrich Chemical Co.) was characterized using the DSC220C at various samples masses and heating rates. The material undergoes two melting events at 118 and 135°C.

Displayed in Figure 1 is a overlay of the DSC results obtained at a constant heating rate of 20^oC/min using varying masses (1.40, 2.57, 4.40 and 10.6 mg). As the data shows, better resolution between the two melting peaks is

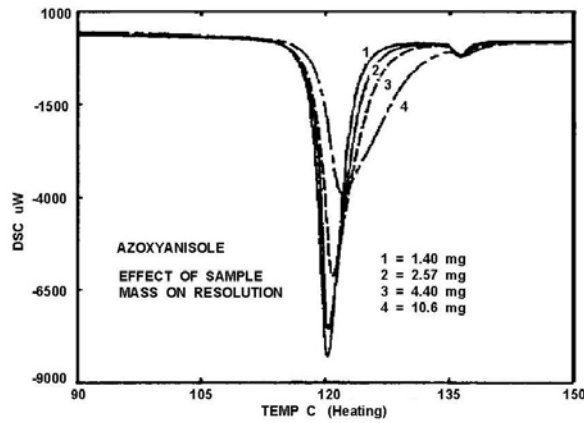


Figure 1

obtained at lower masses. An enlargement of the melting data for azoxyanisole is shown in Figure 2. At a mass of 1.40 mg, there is a good DSC baseline between the two melting peaks. As the sample mass increases, the definition of the baseline occurring between the two melting peaks decreases.

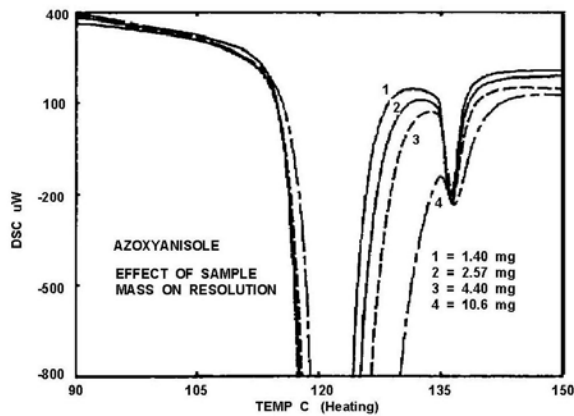


Figure 2

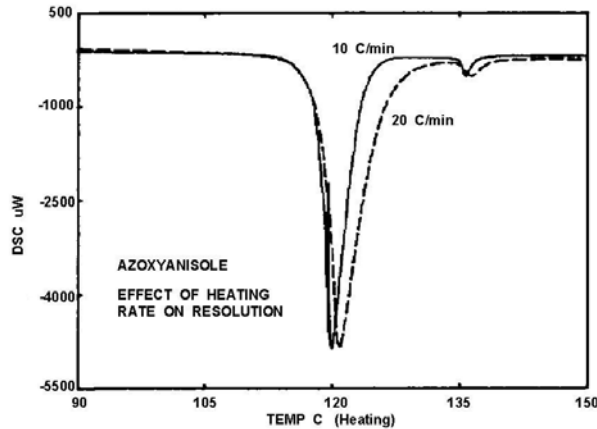


Figure 3

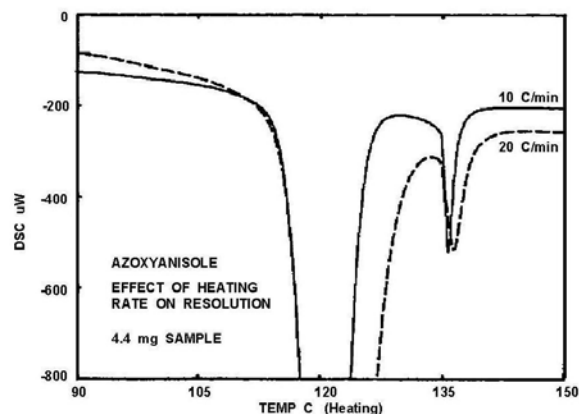


Figure 4

Using a lower heating rate significantly improves resolution. Displayed in Figures 3 and 4 are DSC results obtained on a 4.4 mg azoxyanisole sample at heating rates of 10 and 20°C/min. The data demonstrates that a more well defined baseline, between the two melting peaks, is obtained at the lower heating rate.

Summary

Improved resolution, on two or more closely occurring melting transitions, can be obtained by:

- reducing the heating rate
- reducing the sample mass.

