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THERMAL ANALYSIS

application note

DSC and Resolution

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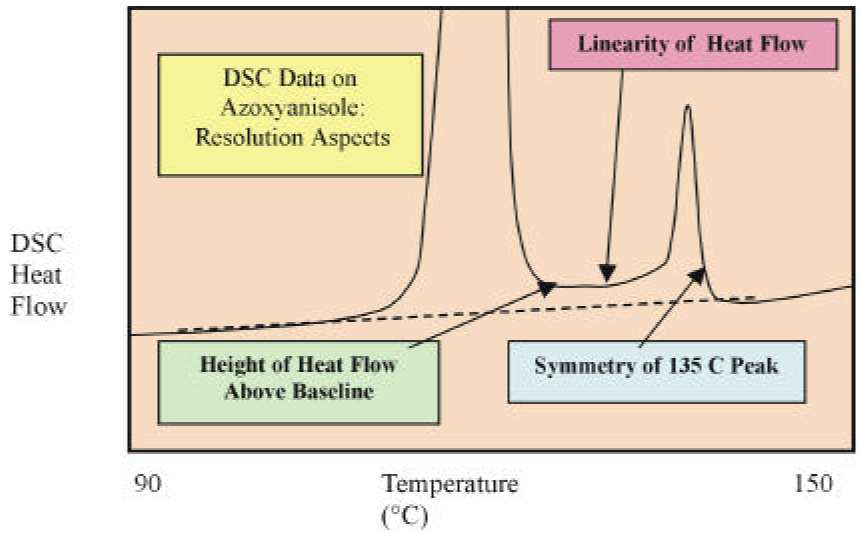
One of the most important performance characteristics of a DSC instrument is its *resolution*. This refers to the ability of the DSC to separate or resolve closely occurring thermal transitions. The resolution of a DSC cell is dependent upon its particular design properties including the mass of the furnace, the temperature measuring system employed and the response time of the cell.

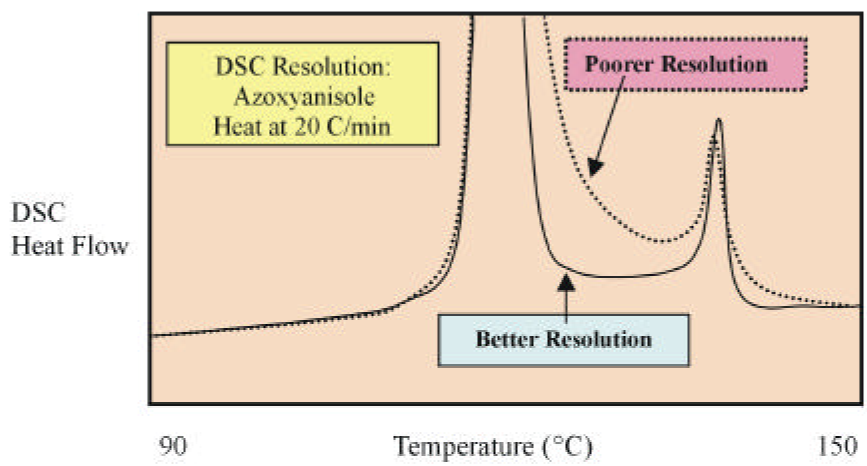
The power compensation design of the PerkinElmer Pyris 1 DSC provides a low mass furnace, especially when compared to the much more massive furnaces used with heat flux DSC instruments. The low mass of the Pyris 1 DSC along with the use of a platinum resistance thermometer (PRT) temperature measuring system provides outstanding inherent resolution.

The resolution of a DSC is also a function of the given experimental conditions, including:

- Purge gas
- Sample mass
- Heating rate

Enhanced resolution can be obtained using a helium rather than a nitrogen, air or oxygen purge, due to the significantly higher thermal conductivity of helium. Lower sample masses can provide improved resolution over larger masses. Slower heating rates will yield significantly better resolution than faster rates.





The issue of resolution becomes important for certain applications, especially when overlapping melting transitions are to be measured. Applications requiring a DSC with high resolution include:

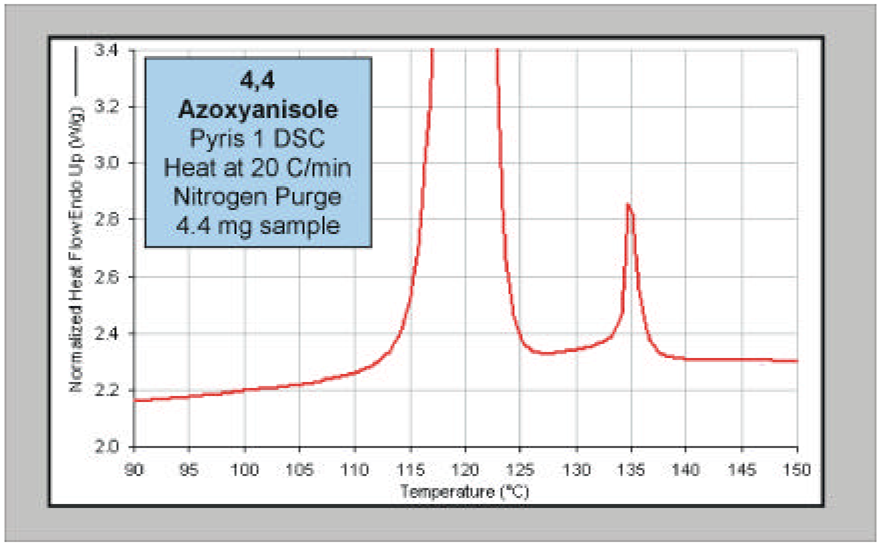
PerkinElmer™ instruments.

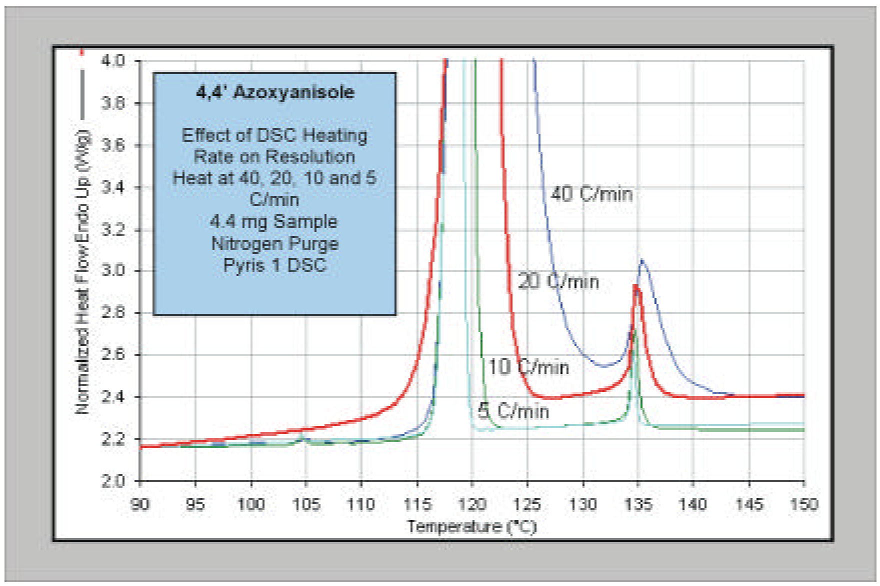
- Pharmaceutical materials where polymorphism (multiple melting transitions) frequently occur
- Foods containing fats which exhibit polymorphism due to storage conditions
- Polymers where the material has been heat treated or exposed to
- elevated temperatures (textured fibers, for example).

One means of quantitatively defining or measuring DSC resolution is through the analysis of 4,4' azoxyanisole. This substance has two closely occurring endothermic transitions at 120 and 135 C. The material melts at 120 C and has a smaller, liquid crystalline transformation at 135 C. The resolution of the DSC instrument can be defined as how well the heat flow response returns to a linear baseline in the region between the two transitions when the sample (4 to 5 mg) is heated at a rate of 20 C/min using a nitrogen purge gas. The better the resolution of the instrument, the better the heat flow response returns to the baseline. An instrument with a high inherent resolution will yield a linear heat flow response between the two transitions and a better the return to the baseline as is represented in the following figure.

The differences in resolution between two different DSC instruments are represented below for the analyses of azoxyanisole at a rate of 20 C/min.

To test a DSC instrument for resolution, the following experimental conditions are recommended:





Sample mass:	4.5 mg
Sample pan:	Crimped aluminum pan
Heating rate:	20 C/min
Temperature range:	80 to 160 C
Purge gas:	Nitrogen
Display of heat flow:	Normalized (W/g)

The resolution response of the DSC can be then be defined using the criteria displayed in the first figure.

Displayed in the following figure are the DSC results obtained from the PerkinElmer Pyris 1 DSC using a sample of azoxyanisole

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(Aldrich Co.) analyzed with the experimental procedure described previously.

These results demonstrate that the Pyris 1 DSC yields outstanding resolution performance based on the following performance criteria:

- Linear heat flow response between 125 and 133 C
- Excellent return to baseline in this temperature region
- Obtainment of symmetrical peak at 135 C

The azoxyanisole sample was analyzed at four different heating rates (40, 20, 10 and 5 C/min) on the Pyris 1 DSC to demonstrate the changes in resolution that can be obtained by varying the heating rate:

These results demonstrate that increasingly better resolution can be achieved by going to slower heating rates. However, with the high performance of the Pyris 1 DSC, excellent resolution is obtainable even at 20 C/min. The data generated at 40 C/min with the Pyris 1 DSC is equivalent to that of most heat flux DSC instruments at 20 C/min.

Summary

One of the most important performance aspects of a DSC instrument is its resolution. This refers to the ability of the DSC to separate or resolve two closely occurring thermal transitions. An excellent means of testing a DSC with regards to resolution is analyzing a 4.5 mg sample of 4,4' azoxyanisole, which has two closely spaced endothermic transitions at 120 and 135 C. The PerkinElmer Pyris 1 DSC, with its power compensation design, yields outstanding resolution.

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