



# Use of the New Pyris 1 DSC for the Study of Low Energy Phase Changes

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## Introduction

Liquid crystalline materials can exhibit a wide range of liquid crystalline phases with varying degrees of molecular order. These changes can be seen by DSC studies but the energies involved in these transitions can be very small, making their identification difficult. In the past most of these small energy transitions have been identified by optical microscopy alone. In this example we show that it is possible to see these small energy transitions plainly with the new Pyris 1 DSC.

## Purpose

To determine the phase sequence and transition temperatures of a liquid crystalline material

## Experimental

The sample studied was a liquid crystalline material that was kindly supplied by the Liquid Crystals and Advanced Organic Materials Group at the University of Hull.

The phase transitions that were found by optical microscopy for this material were as follows:

$$K - S_C^*_{\text{anti}} - S_C^*_{\text{ferri}} - S_C^*_{\text{ferro}} - S_A - I$$

I = Isotropic liquid

$S_A$  = Smectic A phase, a layered type structure

$S_C^*_{\text{ferro}}$  = Ferroelectric Smectic C phase, a tilted layered type structure (see later)

$S_C^*_{\text{ferri}}$  = Ferrielectric Smectic C phase (see later)

$S_C^*_{\text{anti}}$  = Antiferroelectric Smectic C phase (see later)

K = Crystalline

A DSC study of this material was made to confirm this phase sequence and allow the quotation of accurate phase transition temperatures and energies for this material.

The sample was placed into an aluminum standard sample pan and crimped shut. The program used for the study was a temperature scan between 40° and 120°C at a heating rate of 10°C/min. An analysis of the subsequent DSC trace was then made.

## Results

The following DSC trace for the material was obtained Figure 1. As seen below, there are three distinct thermal events that correspond to phase changes, within the structure of the liquid crystal material.

The first transition upon heating is a crystal to crystal transition followed by

## DSC Method

Instrument	PerkinElmer Pyris 1 DSC
Method	DSC Temperature Scan
Heating rate	10° C/min from 40° to 120°
Pan	Crimped Standard Aluminum
Purge gases	Nitrogen 20 ml/min
Cooling Device	Water
Sample Description	Liquid Crystalline Material, Powdered
Sample Weight	4.091 mg

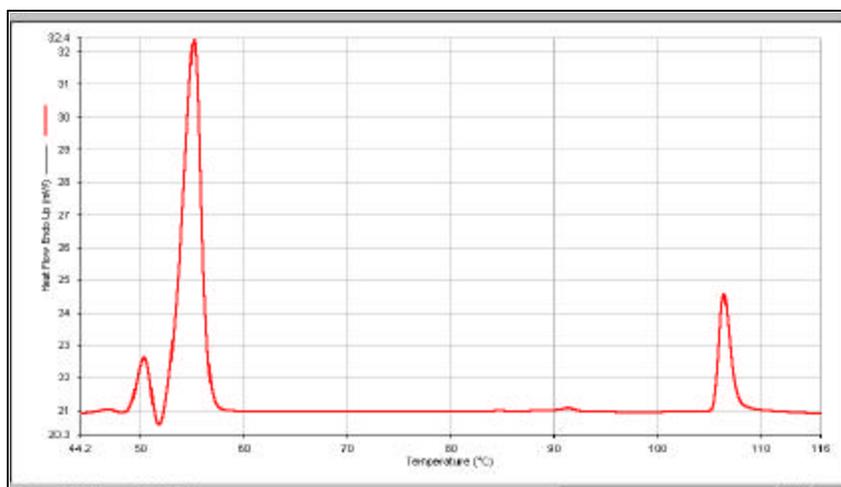


Figure 1: DSC of Liquid Crystalline Sample, Heat Flow Data

a recrystallisation due to the change in crystalline structure. There then follows a major endothermic event at 52.9°C corresponding to the melting of the crystalline material into a liquid crystalline smectic C\* phase. From this initial DSC trace it appears that the next liquid crystalline transition occurs at 89.73°C which is the smectic C\* to smectic A transition. The final endothermic event seen in Figure 1 at 105.5°C is where the material undergoes a smectic A to isotropic liquid transition.

If the DSC trace is zoomed into the area of 80-95°C as in Figure 2 then it is possible to identify some small endothermic events that occur in this

The energies involved in these transitions are very small and therefore push the instrumental sensitivity to new limits.

For the transition from the ferroelectric smectic C structure to the ferrielectric structure was calculated to be 0.021 J/g. The change in structure associated with this transition is shown schematically in Figure 3. There is a change in the tilt orientation of the layers in the macrostructure. The transition is therefore the change in tilt of some of the layers throughout the bulk structure and hence the energies involved in the tilting of the layers are small. Similarly, the transition from the ferrielectric to the anti-ferroelectric

smectic C phase was found to have a low transitional energy, calculated to be 0.015 J/g. Again the transition is associated with a change in the tilt of the layers in the structure of the material, this change is shown schematically in Figure 3.

### Conclusion

With the new Pyris 1 DSC it is possible to easily identify very small energy transitions for these types of materials. These small energy phase changes were clearly seen above the baseline and sample noise and show the sensitivity and resolution of the new instrument. The increased sensitivity afforded by the instrument has allowed the definite identification of the liquid crystalline phases discussed previously.

### References

- Collings, P.J., Hird, M. In *Introduction to Liquid Crystals Chemistry and Physics*, Taylor and Francis: London, 1997.

### Acknowledgments

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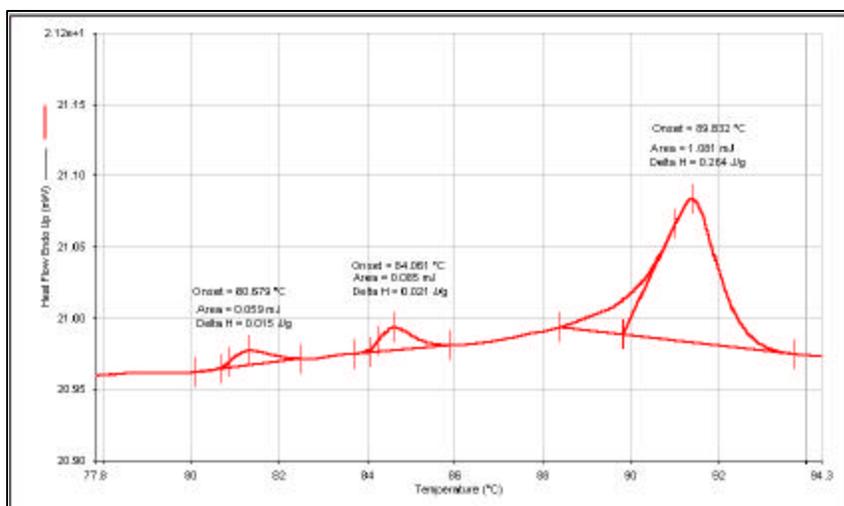


Figure 2: DSC of Liquid Crystalline Sample, Expanded DSC Trace

range. Phase transitions were expected for this material in this temperature range because changes in the optical textures using polarising optical microscopy had been noted and attributed to a series of frustrated liquid crystalline phases. The phase transitions identified by both optical microscopy and by DSC in the temperature range 80-95°C were identified as the following.

$$S_{C^*_{anti}} 80.7^\circ S_{C^*_{ferri}} 84.1^\circ S_{C^*_{ferro}} 89.8^\circ S_A$$

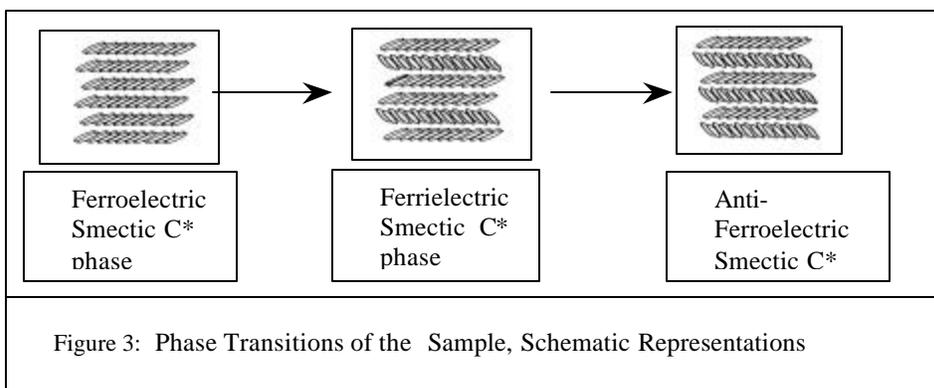


Figure 3: Phase Transitions of the Sample, Schematic Representations