

**Experiment title:**

The effect of chain-folding and stem length on the lattice parameters and thermal expansion coefficients of long n-alkanes

Experiment**number:**

SC906

Beamline: ID11	Date of experiment: from: 1/3/02 to: 4/3/02	Date of report: 30/8/02
Shifts: 8	Local contact(s): Ann Terry	<i>Received at ESRF:</i>

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Report:

Wide angle X-ray diffraction patterns of ultra-long alkanes whilst subjected to a temperature ramp were collected on ID11 using a Bruker CCD in high resolution mode. 2k x 2k patterns with a pixel size of 70 μ m were collected for 2 seconds, but there was a read-out/dead time of 10 seconds. High resolution diffraction patterns with a minimum of 29 reflections were collected. The experiment was performed at 25keV with a 300 x 300 μ m beam.

The alkane samples were sealed in 0.5mm lindemann tubes and mounted in a specially designed holder on the front face of a Linkam heater. The holder ensured that the sample detector distance was maintained as accurately as possible between sample changes so that individual samples did not have to be internally calibrated. The linkam heater was controlled via a serial line through a Labview interface with SPEC, and could be synchronised with the data collection as the Bruker CCD was controlled via the same SPEC session. The first 5 shifts were used for beamline alignment and to set-up the experiment and to synchronise the data collection with the Linkam.

The diffraction patterns were corrected for spatial distortions prior to integrating to give intensity against 2theta. The actual integration proved troublesome since the detector was actually tilted relative to the beam and so a further correction to account for this had to be incorporated.

Previously, a small but significant change in lattice parameters as the alkanes crystals thicken upon heating had been observed in a lower resolution experiment which only examined the (110) and (200) reflections. These findings were confirmed. Data for 27 samples were collected covering chain lengths from 102 carbons to 390 carbons in the chain. One polymeric sample was also examined. Sufficient data was collected to determine the difference between the effect of chain length, and the effect of number of folds, on the lattice parameters, as this was one of the aims of the proposal. By obtaining hkl reflections it will be possible to see the impact of contraction on the c axis that was not possible from our other studies. The slight misalignment of the detector is slowing down the analysis and has prevented this from having been obtained yet. These data have formed the basis of a joint ESRF/EPSRC studentship due to start September 2002.

During the measurements, some problems were encountered especially with the interface to the Bruker CCD which proved very costly for time wasted during the experiment.

1. Terry A., Hobbs J.K., Phillips T.J., Hanna S., submitted to Science
2. Terry A., Phillips T.J., Hobbs J.K., in preparation

