

**Experiment title:**

Origin of the medium angle reflections in biologically active polymers containing crown ether groups

Experiment number:
SC-1914

Beamline: BM26	Date of experiment: from: 4-April-2006 to: 8-April-2006	Date of report: 22-January-2007
Shifts: 12	Local contact(s): Nicolas VILAYPHIOU	<i>Received at ESRF:</i>

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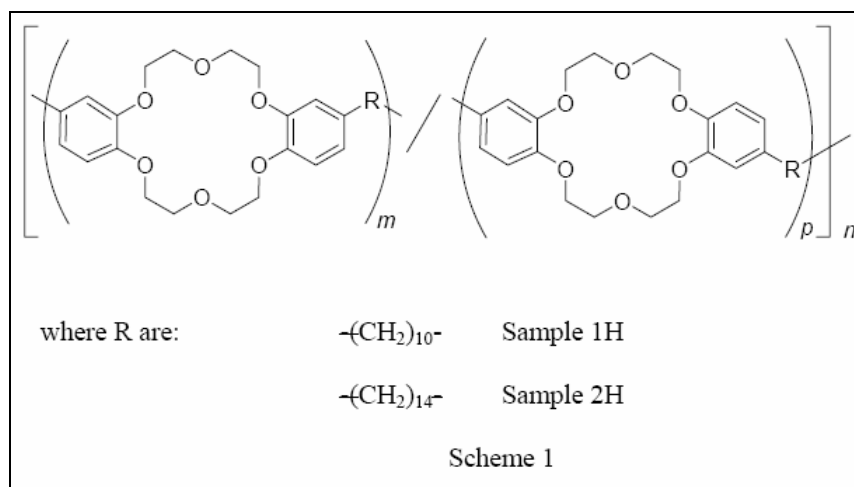
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Report:

The structure and thermal properties of polymers containing in the main chain dibenzo-18-crown-6 ether unit linked to an aliphatic spacer of different length (C10 or C14) (see scheme 1) have been studied by simultaneous calorimetry and small, medium (SAXS-MAXS) and wide (WAXS) x-ray measurements during cooling and subsequent heating of the samples.



The X-ray diffraction patterns of all the studied samples exhibit a peak in the medium angle region, revealing the existence of a lamellar structure. This indicates that a layer phase is formed upon cooling.

In the case of the homopolymers, this phase is almost simultaneously accompanied by the appearance of some reflections in the wide angle region as an indication of lateral crystallization. However, by copolymerization, the formation of the layer phase is decoupled from lateral crystallization, being stable in a wide temperature region.

To perform these simultaneous measurements, pieces of the solution cast film were encapsulated in aluminium pans and melted for 5 min at 180°C. DSC and X-ray scattering patterns were recorded during cooling at 10°C/min. Subsequent heating from 30°C to 180°C were also recorded.

The wide and medium angle X ray patterns obtained, simultaneously with the DSC traces, during cooling at 10°C/min for sample 1H are presented in figure 1 and the ones obtained during subsequent heating are shown in figure 2.

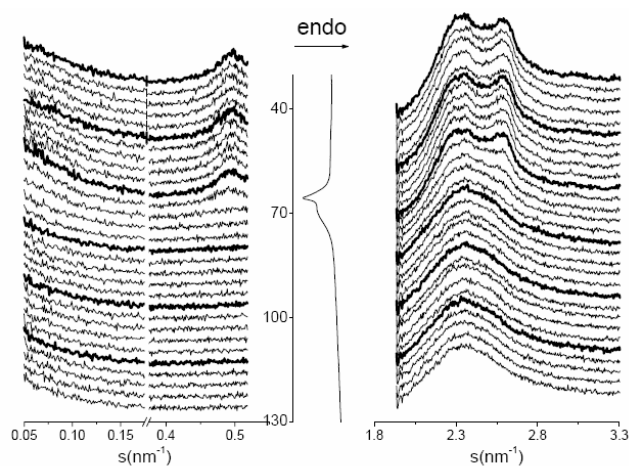


Fig. 1: X ray diffraction patterns in the SAXS-MAXS region (left) and WAXS region (right) of sample 1H during cooling from the isotropic melt (180°C) at 10°C/min down to 30°C. Only frames from 130°C to room temperature are presented. In the central panel, the cooling DSC is presented for comparison. The vertical axis indicates temperature

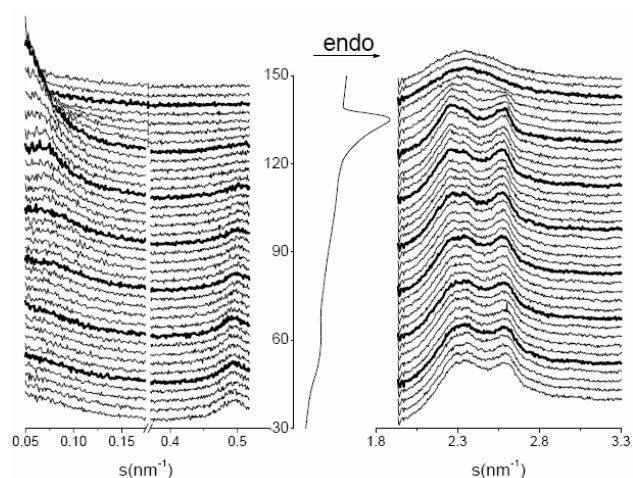


Fig. 2: X ray diffraction patterns in the SAXS-MAXS region (left) and WAXS region (right) of sample 1H during heating at 10°C/min. In the central panel, the heating DSC is presented for comparison. The vertical axis indicates temperature.

Further analysis of the results is currently being performed.

References:

- Zolotukhin, M. G. et al. *Macromolecules* 2006, 39, (14), 4696-4703.
- A. Nogales et al. Paper in preparation.