ESRF	**Experiment title:** Liquid crystalline behaviour of Polyhedral Oligomeric Silsesquioxanes (POSS)	**Experiment number**: SC 2207
Beamline: Bm26b	**Date of experiment**: from: 06/07/07 to: 09/07/07	**Date of report**: 16/02/09
Shifts: 9	**Local contact(s)**: Kristina Kvashnina	*Received at ESRF:*

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Experimental: Several Polyhedral Oligomeric Silsesquioxanes (POSS) compounds [1-3] were synthesised and the LC behaviour of the mesogens (vinyl terminated phenoxy-esters) with and without tethering to the POSS T_8 cage (Fig. 1) was investigated during several heat/cool regimes. The heat-cool regimes where performed at a rate of 5 ^{o}C/min.

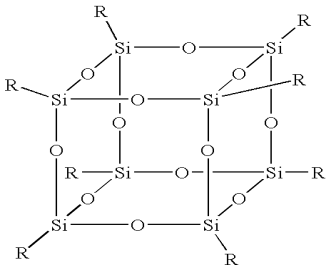
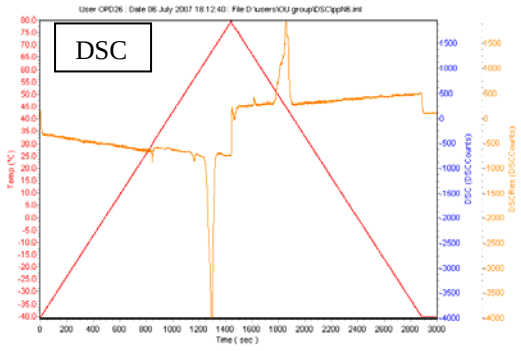
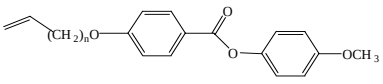


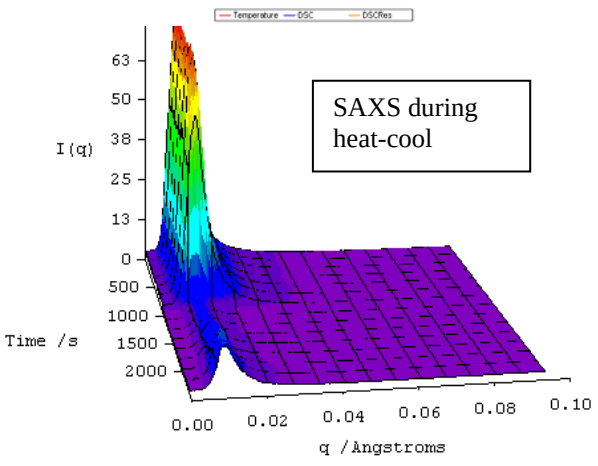
Fig 1. POSS T_8 cage with eight R group substitutents.

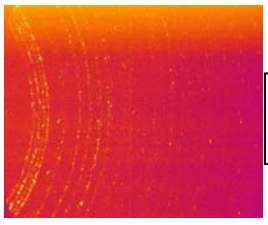
Results: The SAXS/WAXS and DSC data below in Fig 2, shows the heat-cool of the just the mesogen (pictured).



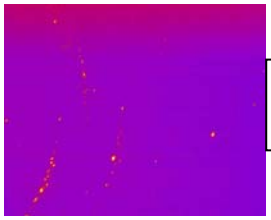


LC Mesogen





WAXS Frame during heat



WAXS Frame during cool

Fig 2. SAXS/WAXS/DSC data for heat-cooling of an LC mesongen.

From the DSC data it can be seen that the mesogen undergoes several phase transitions during the heating regime: 1: 30 ^{o}C, 2: 56 ^{o}C and melting at 65 ^{o}C [4]. During cooling similar phase transitions are seen. The SAXS data shows that some structure (near the maximum at the backstop) is present before melting and disappears on melting . However, once cooled the structure does not appear to return. The WAXS data show crystalline Bragg reflections, which are strong during the heat, but again as the sample is cooled the Bragg peaks return but are less intense. In the data below in Figure 3, represents this mesogen now being attached to each corner of a POSS cage.

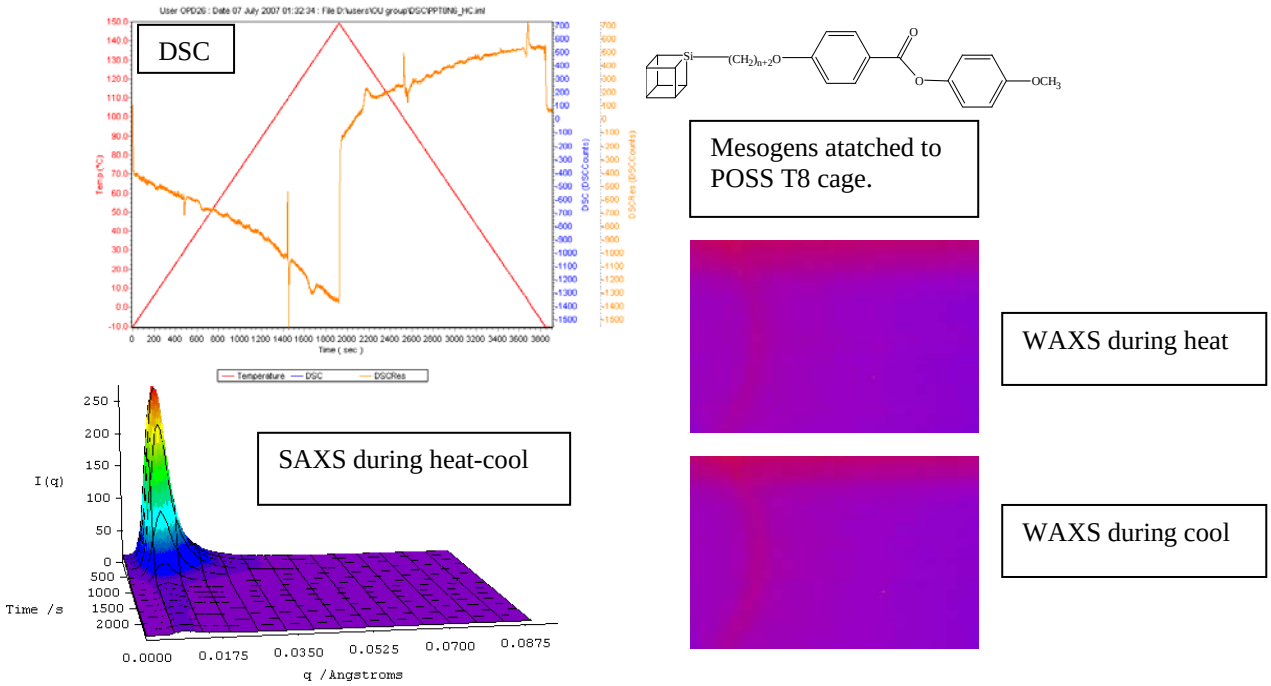


Fig 3. SAXS/WAXS/DSC data for heat-cooling of an POSS-LC T8 cage .

Here, similar trends can be seen in the data. However the WAXS data shows now crystalline Bragg peaks of silicon, which appear to become more crystalline on the cool. The SAXS shows that the order in the cool regime is not restored.

Generally, it has been observed by microscopy in such samples, that POSS-LC systems, do not resume their crystalline lattice form once cooled but, remain in the LC state at room temperature, as a result of supercooling. Both sets of results here seem to support this theory, where the crystalline structure is destroyed when the sample is heated and the crystalline structure in the SAXS and WAXS does not return on cooling. However, some regular structure is seen in the WAXS of the POSS cage sample, this may indicate a regular ordering of the cage structure now in the LC state.

1. A.R. Bassindale, Z. Liu, I.A. MacKinnon, P.G. Taylor, Y.Yang, M.E. Light, P.N. Horton, M.B. Hursthouse, J. Chem. Soc. Dalton Trans. (2003) 2945.
2. A.R. Bassindale, Z. Liu, D.J. Parker, P.G. Taylor, P.N. Horton, M.B. Hursthouse, M.E. Light, J. Organomet. Chem (2003), 687, 1.
3. A. R. Bassindale, H. Chen, Z. Liu, I. A. MacKinnon, D. J. Parker, P. G. Taylor, Y.Yang, M. E. Light, P. N. Horton and M.l B. Hursthouse, J. Organomet. Chem (2004) 689, 3287.
4. J. Hardy, PhD thesis, OU, 2001.