



	Experiment title: <i>Ordering in a low-mass organogelator: a high resolution powder diffraction investigation of related gels and xerogels.</i>	Experiment number: <i>CH 437</i>
Beamline: <i>BM 16</i>	Date of Experiment: from: <i>18.01.98</i> to: <i>25.01.98</i>	Date of Report:
Shifts: <i>18</i>	Local contact(s): <i>DOORYHEE Eric</i> <i>FITCH A,</i>	<i>Received at ESRF :</i>

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Report:

The complete schedule has been performed and high quality data have been obtained with the BM16 spectrometer. First of all, we would like to emphasize the very high quality of the data that we have collected (especially when comparing with preliminary experiments obtained with a rotatory anode and which are shown in the corresponding proposal).

We studied 2 derivatives of an androstanol organogelator: diamagnetic one (STNH) and a paramagnetic one (STNO, a nitroxide compound). The crystalline powders have been studied and will constitute the "reference states" from which structural reorganizations will be searched when the fibrillar networks of related organogels and xerogels are considered. The xerogels (solids formed by slow evaporation of the organic liquid) have given nice crystalline-like patterns superimposed to a broad component at $2\theta \approx 9$ deg. The corresponding gels have been studied and clearly show that diffraction peaks are emerging from the intense broad components of the structure factor of the related organic liquid (this could lead to the important conclusion, after complete data treatment, that the local structures within the fibers is not modified from gels to xerogels).

Figure 1 illustrates the data obtained for the crystal, the xerogel (from cyclohexane gel) and a gel at $C \approx 10$ %wt in cyclohexane.

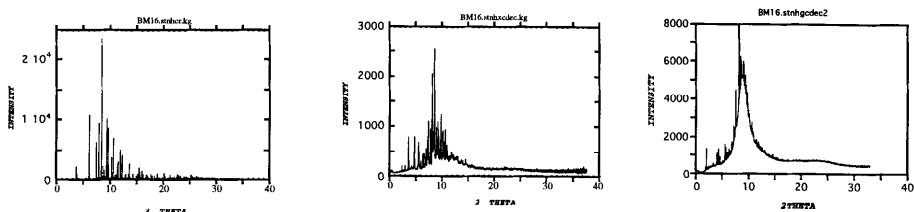
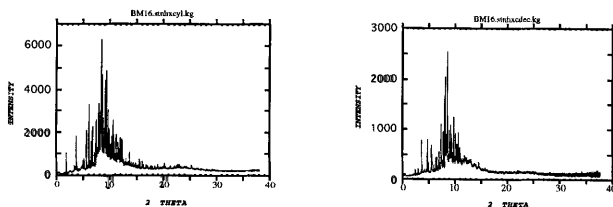


Figure 1: STNH: crystal xerogel (cis-decaline) gel

In such a sequence (crystal/xerogel/gel), the molecular re-ordering from crystal to gel fibers will be analyzed also as a function of the solvent type. Indeed, it has been shown that major differences exist (structural, thermal, rheological) as a function of the solvent type for this class of materials. In this context, we have recorded the diffraction patterns of STNH and STNO systems (gels, xerogels, pure liquids) from cyclohexane, trans-decaline, cis-decaline liquids.

Figure 2 illustrates the differences that exist between STNH xerogels from cyclohexane (left) and cis-decaline (right).



The driving force of the aggregation mechanism for the androstanol gels is known to be the hydrogen bonding.

STNH has 2 potential sites (OH and NH) while STNO has mainly one potential center (OH) and an unsaturated paramagnetic site (NO). Different behaviors have been observed for STNH and STNO organogels which suggest that the molecular structures are also different.

The knowledge of the molecular structures and their evolution between the crystal, the gel, the xerogel states should provide important clues for the understanding of the gelation mechanism of this class of crystalline organogels. This work should explain the mechanism of the evolution from a 3-d to a 1-d crystallization process. The data treatment is under progress.