



<b>Experiment title: High Resolution Small Angle X-ray Scattering of a Sliding Phase in DNA/Cationic Lipid Complexes. Study of the thermotropic transitions</b>		<b>Experiment number: SC593</b>
<b>Beamline:</b> ID2A	<b>Date of experiment:</b> from: 10.11.1999 to: 13.11.1999	<b>Date of report:</b> 03.2000
<b>Shifts:</b> 9	<b>Local contact(s):</b> T. NARAYANAN	<i>Received at ESRF:</i>
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The wavelength energy was 12.0keV, the beam was not focussed with a size of  $200 \times 200 \mu\text{m}^2$  on sample and on detector. Attenuation was 200 due to use of 2D gas filled detector saturation. The sample/detector distance was 1.4m. In this condition, the accessible scale was  $q=0-0.5 \text{ \AA}^{-1}$ , and the resolution  $\Delta q=4.10^{-3} \text{ \AA}^{-1}$  (HWHM). A few image plates of the best samples were measured, yielding a better resolution  $\Delta q=5.10^{-4} \text{ \AA}^{-1}$  (HWHM).

The 9 shifts, 72 hours, were used in order to obtain a maximum of results and testing some configurations that could be used in the future for this subject. The beamtime was separated as described in the following :

- 12 hours were dedicated to the beamline alignment and solving the detector problem: The expected CCD Camera being out of order and we decided to use the 2D gas filled detector. This has two main disadvantage in our case. i) due to the high intensity of the first Bragg peak, the beam was attenuated by a factor 200 to avoid detector saturation. The needed acquisition time for observing DNA diffuse scattering was then 1 minute, instead of less than 1s with the CCD detector. ii) The spatial resolution of the gas filled detector is  $700 \mu\text{m}$  which is 3.5 time the beam size, yielding a resolution 3.5 worse than expected.

- 4 hours were dedicated to the sample stability tests. Under these conditions, samples are stable a few minutes, and a degradation is observed after a few 10 minutes. This confirm their huge stability previously observed at DESY and LURE, and let "long" exposure time available (a few minutes for the image plate).

20 hours were dedicated to the DMPC/DMTAP/DNA system, that is studied for 2 years at LURE and DESY. As expected in these conditions, it was possible to collect a large number of scan, 12 temperatures for 40 samples. With the CCD detector, 40 temperatures should be available. A few samples were selected for measurement with image-plate. Briefly, the previous results were confirmed, and it is now possible to draw a first phase diagram of the DNA-interlayer correlations.

20 hours were dedicated to a new system DMPE/DMTAP/DNA. Scans of 35 samples at 12 temperatures were collected. The results are close to the DMPC/DMTAP/DNA results,

but the DNA peaks exhibit some new behaviour. For example, the coexistence of the DNA peak at the  $L_\alpha$ - $L_\beta$  transition has been observed (figure 1).

4 hours were dedicated to test new projects. Results were successful.

4 hours were dedicated to aligned samples of complexes (figure 2). The bilayer membranes are easily aligned, but the DNA 2D-smectic is a powder in 2D, and the DNA peak are detectable only if they are in Bragg position, that was not the case on the tested samples.

The last 8 hours of experiment were dedicated to measurement with the image plate as detector, in order to obtain a high resolution. Only a few samples were studied due to the long time for developing the image-plate. But the results exhibit a high resolution and a high statistic (figure 3,4). Two new phases were then observed: the correlation in the fluid phase (fig. 3) were definitely proved and the power law peak (figure 4) has to be confirmed on a non-demixing samples.

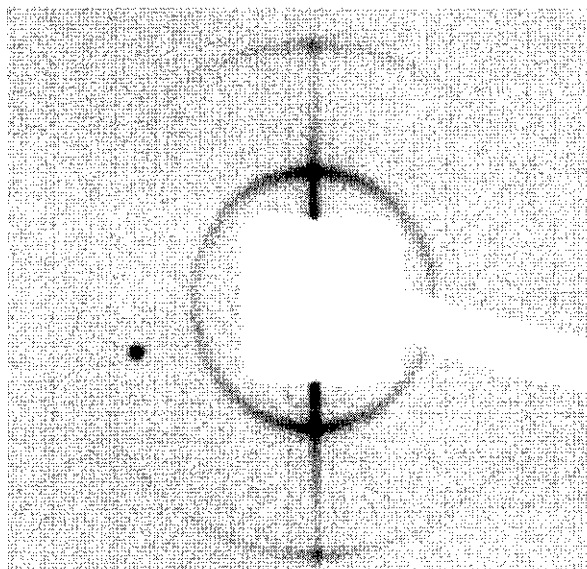
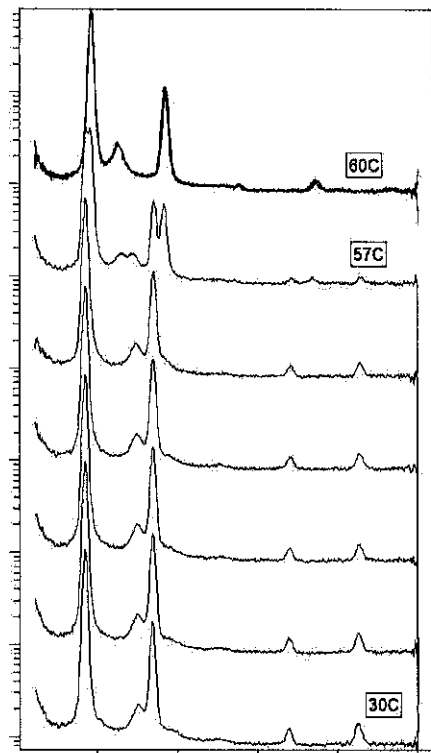


Figure 1: Example of temperature scan on a DMPE/DMTAP/DNA complexes. Note the DNA peak coexistence observed at 57. Figure 2: Aligned samples of complexes and the two first bragg peaks

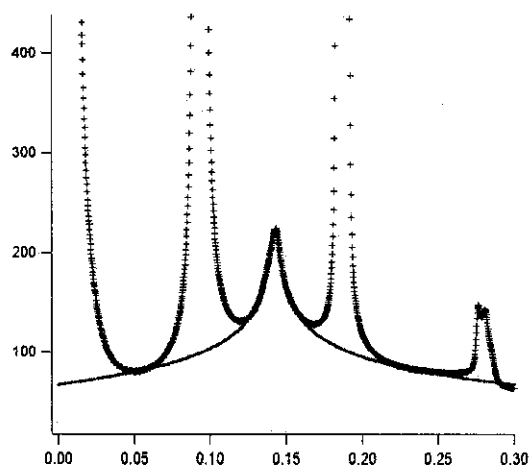
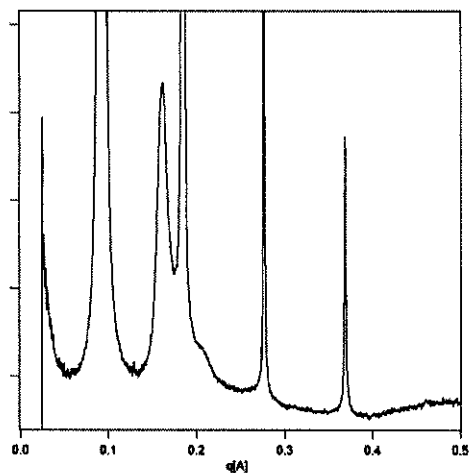


Figure 3: Diffuse scattering of the DNA super lattice at 50C (fluid phase). Note the presence of a higher order indicating a correlation between DNA layer as observed in the gel phase.

Figure 4. Diffuse scattering of the DNA superlattice, and a powerlaw fit superimposed.