



Experiment title: High Resolution Small Angle X-ray Scattering of Sliding Phases in DNA/Cationic Lipid Complexes. Study of the 2D-3D transition.

Experiment number:
SC801

Beamline: ID2A	Date of experiment: from: 03.03.2001 to: 06.03.2001	Date of report: 07.2001
Shifts: 6	Local contact(s): 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 200x200 μm^2 on sample and on detector. Attenuation was only due to the beam definition slits. Two kind of detections were used, the CCD and the Image Plate. In the case of the CCD detector, the sample/detector distances were 0.9m, 1.5m,6.5m. In this condition, the accessible scale was $q=0.005-0.7\text{\AA}^{-1}$, and the resolution is due to the detector pixel resolution (300/400 μm). A few image plates of the best samples were measured at 4m with a very large $q_{\text{max}}/q_{\text{min}} = 100$ and a resolution due to the beam size $\Delta q=5.10^{-4}\text{\AA}^{-1}$ (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 :

- beamline alignment, setup installation and tests.
- sample stability tests. Under these conditions, samples are stable a few ten seconds, and a degradation is observed after 1 minutes.
- temperature scans studies with 20 samples measured at each temperature. As expected in these conditions, it was possible to collect a large number of scan, 40 temperatures for 20 samples in a few hours.
- selected samples were studied with the Image Plate, yielding very high resolutions.
- New project were tested : actin/lipid complexes and polymorphism studies of Solid Lipid Nanoparticles. Results are promising

DNA COMPLEXES RESULTS : Different systems were investigated :

DNA-polyelectrolytes form spontaneously complexes with or without liquid-crystal organization. Powder and fiber diffraction should be performed on these samples.

DMPE/DMTAP/DNA is a system whose PE lipid should be protonated or not. 120 samples with varying composition pH, and ionic strength have investigated. In the better condition it possible to follow the internal pH from the DNA peak position and consequently, to investigate the pH equilibrium with the buffer by X-ray diffraction. Results are in contradiction with theory available for pure membranes.

DPPC/Calcium/DNA phase diagram was investigated over 60 samples by changing Calcium, lipid, DNA concentrations. DNA Complexation should occurs without cationic lipids.

DMPC/DMTAP/DNA system was studied with the CCD detector in order to selected the best powder samples close to the sliding coupling transition. A few samples were selected for measurement with image-plate. The main results are shown bellow. By adding neutral lipids, the DNA-DNA interdistance increase (DNA peak is moving to smaller q), and the coupling is increasing (DNA peak is smaller). A suspected, a first order transition is observed between lorentzian peaks and power-law like peaks. Theoretically [Lubensky et al, PRE, 2000], the crystallization should produce a true columnar phase and true bragg peaks.

This unexpected phase is a crystallization intermediate and is not yet well understood

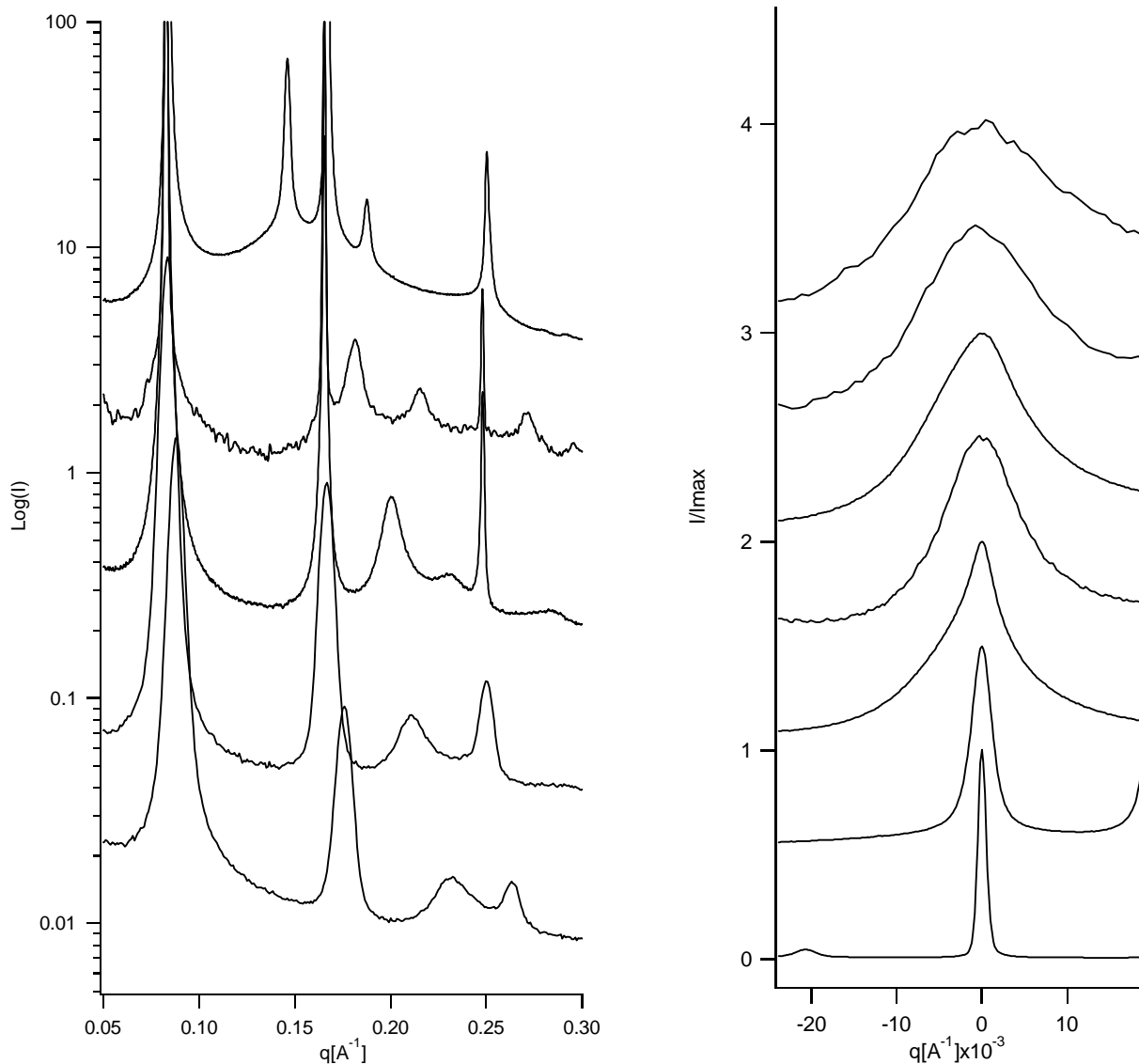


Figure 1(left): Evolution of the DNA super-lattice diffuse scattering from a low coupling regime (bottom) with a asymmetric broad peak, to a strong coupling regime with a power-law like peak.

Figure 1 (right) : selected high resolution data of the DNA diffuse scattering. Bottom is experimental bragg peaks. Note the evolution from a sharp peak to a broad diffuse peak.