ESRF	**Experiment title:** Monolayer structure of the homologous serie n-CB adsorbed on MoS_2: Commensurability as a function of chain length	**Experiment number:** SC-443
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Report:

We have worked in a standard grazing incidence X-ray diffraction (GIXD) at an energy of 8.2 KeV (MoS_2, critical angle θ_c= 3.08 mrad) with a vertically mounted sample to gain access to the whole reciprocical space of interest. The incoming beam, monochromatised by the two diamond crystals, was prepared by a set of two Pt-coated mirrors to reject high-order harmonics. The full beam spot of 0.3x0.7 mm^2 was delimited close to the sample by a pair of slits to a typical size of 0.05x.0.5 mm^2 (horizontal x vertical gaps). The diffracted beam was detected by a NaI detector and controlled by two set of slits placed at 220 mm and 700 mm of the sample. Typical size of the detector slits of 1x10 mm^2 led to an angular resolution of $0.08° \times 0.8°$, i.e. a somewhat relaxed Qz resolution to integrate partly the truncation rods. The whole set up performed extremely well, except of some "jumps" of the second monochromator which had to be realigned every two days.

We have aligned three different substrates of MoS_2 coming from natural single crystals that were freshly cleaved and on which we have deposited 8CB LC molecules. The substrates revealed to be quite tedious to align precisely due to their natural mosaicity and the very small divergence of the beam both vertically and horizontally. Despite their natural origin, the substrates displayed the nominal hexagonal structure with the tabulated lattice parameter values and relatively good moncristalline properties: The c* mosaicity was quite good for this kind of lamellar compounds but of the order the critical angle . i.e, of the order of 0.2°

and so was the miscut when it exists. The in plane mosaicity is very good (.01-.03°) but still the sample roughness is quite important, due to the loose Van der Waals couplings between MoS_2 planes. One can conclude that this experiment is on the borderline of the GIXD techniques with a highly collimated beam when the divergence is not large enough to accommodate the substrate structure as this was the case on the BM32 french CRG beam line. Therefore we faced pronounced difficulties to image the 8CB layers and we were only able to measure broad peaks at low Q values (fig.l) in this first experience.

We performed GIXD measurements on the last Troika II sample at LURE-DC1 that confirmed that the 8CB molecules were ordered and found an unusually tilted commensurate order of the LC monolayer. We do not know if this small tilt was present at the time of the Troika II experiment or if it came later after a slow structural relaxation during the month between the two experiments. Nevertheless we can conclude that the best set up to measure this kind of order is to keep the incidence angle a under the critical angle value to optimize the signal to noise ratio. Indeed, the substrate signal can rapidly become too high (fig 2) in the standard symmetric mode ($\alpha=\beta$) we have used.

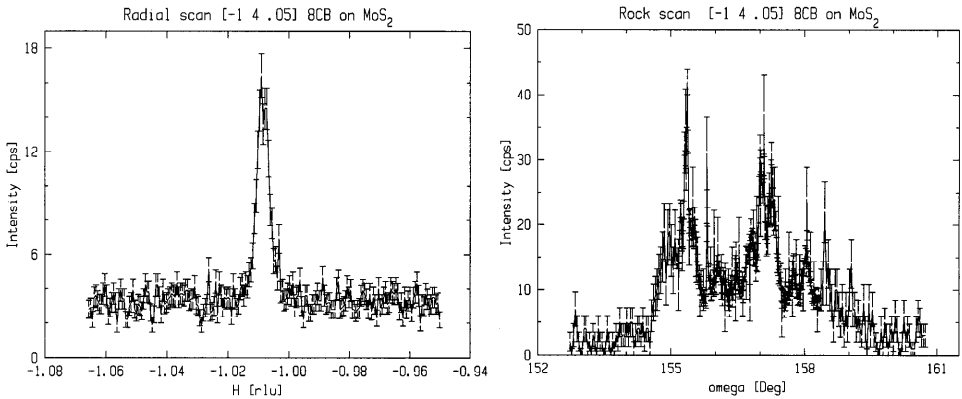

Figure 1: Radial and rock scans of the [-1 4 ε] peak of 8CB on MoS_2. The full width at half maximum of the radial scan, equal to 0.075°, is determined by the Q resolution but the rocking curve is very large.

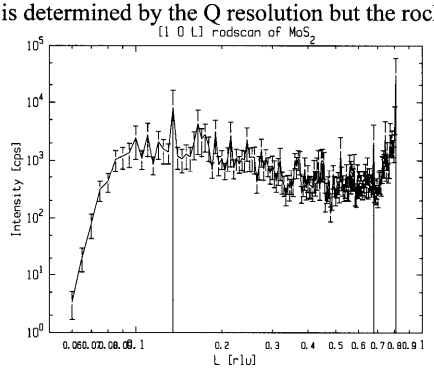

Figure 2: Example of [1 0 L] rodscan of MoS_2. The Vineyard peak is smeared out by the sample roughness but the intensity is quite high all along the rod upto the [1 0 l] Bragg peak. Scan done with a 0.4 mm Al attenuator (attenuation factor of 120 at 8.2 KeV)