

**Experiment title:**

X-ray Grazing incidence diffraction on two-dimensional protein crystals at the surface of water

**Experiment number:LS-598****Beamline:**

ID10

**Date of experiment:**

from: 27-Jan-97 to: 01-feb-97

**Date of report:**

25-feb-97

**Shifts:**

12

**Local contact(s):**G. Gruebel, J. Lal.*Received at ESRF:***3 MAR. 1997****Names and affiliations of applicants** (all are experimentalists):

B. Berge, Univ. Grenoble

P.F. Lenne, Anne Renault idem.

A. Brisson, Univ. Groningen, N.L.

C. Venien, I.B.S. Grenoble.

J.F. Legrand, SI3M/DRFMC/CEA Grenoble

N. Boudet, idem

F. Balavoine, C.E.A Saclay.

**Report:** We started the experiment by aligning and checking the set-up with a monolayer of dodecanol on the surface of water at room temperature. The wavelength selected by the Si monochromator was  $1.407\text{\AA}$  the incidence angle  $2.2\text{mrad}$  and the 28 instrumental resolution was  $0.007''$ . We could use four kinds of 2D protein crystals. Figure 1 shows a 28 scan for a streptavidin monolayer, exhibiting sharp peaks. For each peak we could record the Bragg rod, shown on the right. For AnnexinV, not shown here, only two peaks were clearly detectable. For the Protein HupR bound to lipid Nickel headgroups, the results are shown on the Figure 2. We could in addition estimate a lower bound for the coherence length of the Hup crystals  $L > 4\mu\text{m}$ : The insert shows the peaks width, limited by the resolution. On the right, the Bragg rods associated to the peaks. We are in the process of indexing and analysing these recent results but a few thing happened surprising: 1) the lifetime of the monolayer is extremely short under the beam, of the order of 60s. The trough we use has a translation stage which was used in order to scan the water surface. 2) one of the best candidates for 2D crystals, the cholera Toxin subunit B didn't show any diffraction peak.

In conclusion, this experiment was extremely successful to our point of view. It is too early to know if new structural information will be brought. Our measurements

don't go beyond the resolution of electron diffraction for determination of the horizontal projection of the 2D structure. Nevertheless one should note that the rod intensity distributions cannot be measured by electron diffraction, and are interesting to bring information along the vertical.

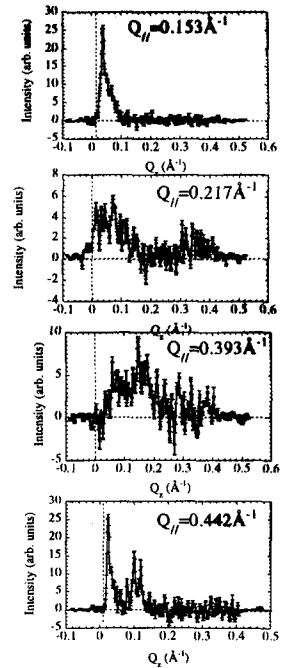
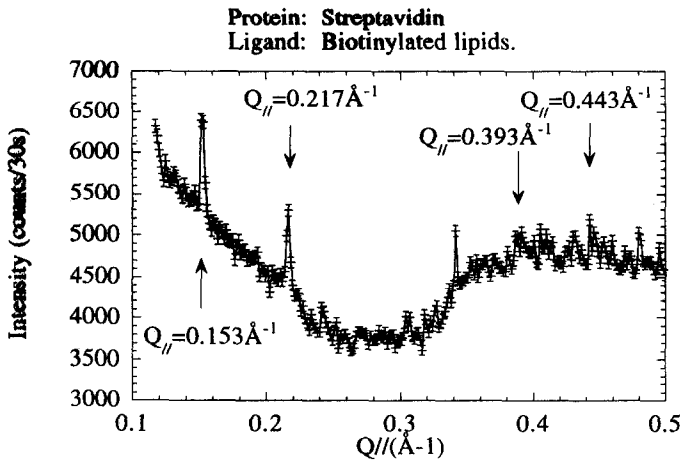


Figure 1

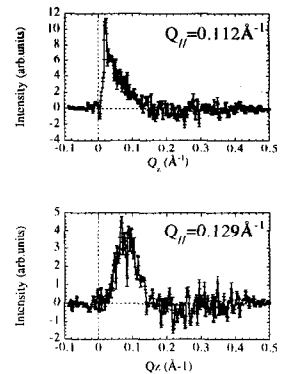
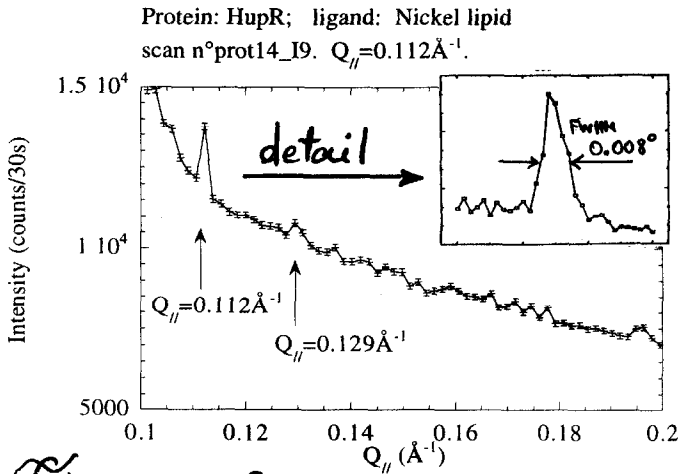


Figure 2