



	<b>Experiment title:</b> Determination by SAXS of the form factor of cylindrical channels in nuclear membranes	<b>Experiment number:</b> ME160
<b>Beamline:</b> ID01	<b>Date of experiment:</b> from: July 11, 2001 to: July 17, 2001	<b>Date of report:</b> August 29, 2001
<b>Shifts:</b>	<b>Local contact(s):</b> Dr. Myles Hamilton	<i>Received at ESRF:</i>
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## Report:

### 1 – Introduction

The goal of the experiment was to measure the Small Angle Scattering intensity from a set of “nuclear membranes”. These membranes are 10 µm thick polymer foils irradiated by heavy ions and subsequently etched by a chemical agent.

In this case the membranes were

- PET (polyethyleneterephtalate)
  - made in Russia (brand name Lavsan) and irradiated by Kr heavy ions in Dubna, or by Xe ions in GANIL
  - made in Germany (brand name Hostaphan) and irradiated by Xe heavy ions in GANIL,
- PC (polycarbonate) irradiated by Xe heavy ions in GANIL. These membranes had been etched in the Laboratoire Léon Brillouin, with a 0.25N NaOH solution, at 80 C, for times vaying from 5 to 90 mn. Some samples had been prepared either together or several times in the same way in order to check the reproducibility.

### 2 - experiment

Up to 8 samples were pressed between two aluminium plates with 2mm holes to let the X-ray beam pass. This sample holder was then put on a goniometer. Unfortunately the vertical movement used as a sample changer allowed access to 5 samples only. The detector was a gaz detector about 200 mm x 200 mm placed at 4.5 m from the sample. Each measurement consisted in a scan around the vertical axis. Before each measurement each sample had been tilted vertically in order to make sure that the polymer foil plane included this vertical axis.

The execution of the experiment has been exceptionally difficult as the detector was destroyed 3 times, thanks to an inadequate control of the beam. Meanwhile our local contact (M. Hamilton) did tremendous efforts and spent long hours to keep control of the incoming X-ray beam while this one was animated by erratic drifts. We want also to thank the detector responsible, M. Kocsis, who repaired twice the detector, including on Sunday 15<sup>th</sup> July ! Finally about a single shift was really useful, over the 15 shifts scheduled.

### 3 - results

Nevertheless we had the opportunity to measure a few samples :

- old PET samples irradiated in Dubna with Kr ions; these samples had been also studied by SANS in the ILL, it was important to observe them in order to compare the advantages and drawbacks of SANS and SAXS for the field of nanochannel study
- new samples, PET and PC, irradiated in GANIL with more energetic Xe ions, etched during the same time as the first ones; for both PET and PC we observed a correct scattering, which was by itself an interesting result as previous samples irradiated in GANIL with Ca ions scattered about 100 times less then the comparable Dubna ones.

As by SANS, on some samples, we have been able to observe the oscillations of the Bessel function, Fourier transform of a coaxial cylindrical channel as is displayed on the figure below.

```

PXN written by Papy/LIB/SAXRAY release August 2001      30/ 8/2001/ 15h 17mn 15s
PATH
FILE ku1005          WAVELENGTH 1.6083
LABEL ME180         DISTANCE 4500.02
GENERAL TITLE Papy  X ORIGIN 61.00
SAMPLE Dubna_45     Y ORIGIN 69.00
                   Beam catcher 7.00
DATE 15-07-2001    Detector cell 0.8100
TIME 15:12:53     DISPERSION 0.0000
ACQUISITION TIME 120. INC. DIVERGENCE 0.0000
MONITOR 3688.     MAXIMUM 16823
                   AVERAGE 209
                   MINIMUM 1
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Header /tmp/ku1005-140016
Dim 1 variable rho
FIT : Cycles 1 NbPar 5 Fin 2
Well finished
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IO 1.00 1.000 27.3
function 1 type Bessel
I 29918.12 29919.121 815679.4
width 5.10 5.103 0.00359 278.88
power 0.45 0.454
distSZ 0.03 0.031
TOTAL 5.0861 0.0018
+ B03 2.9043 0.0011
- 586 2.1817 0.0007
1 smoothing(s)

```

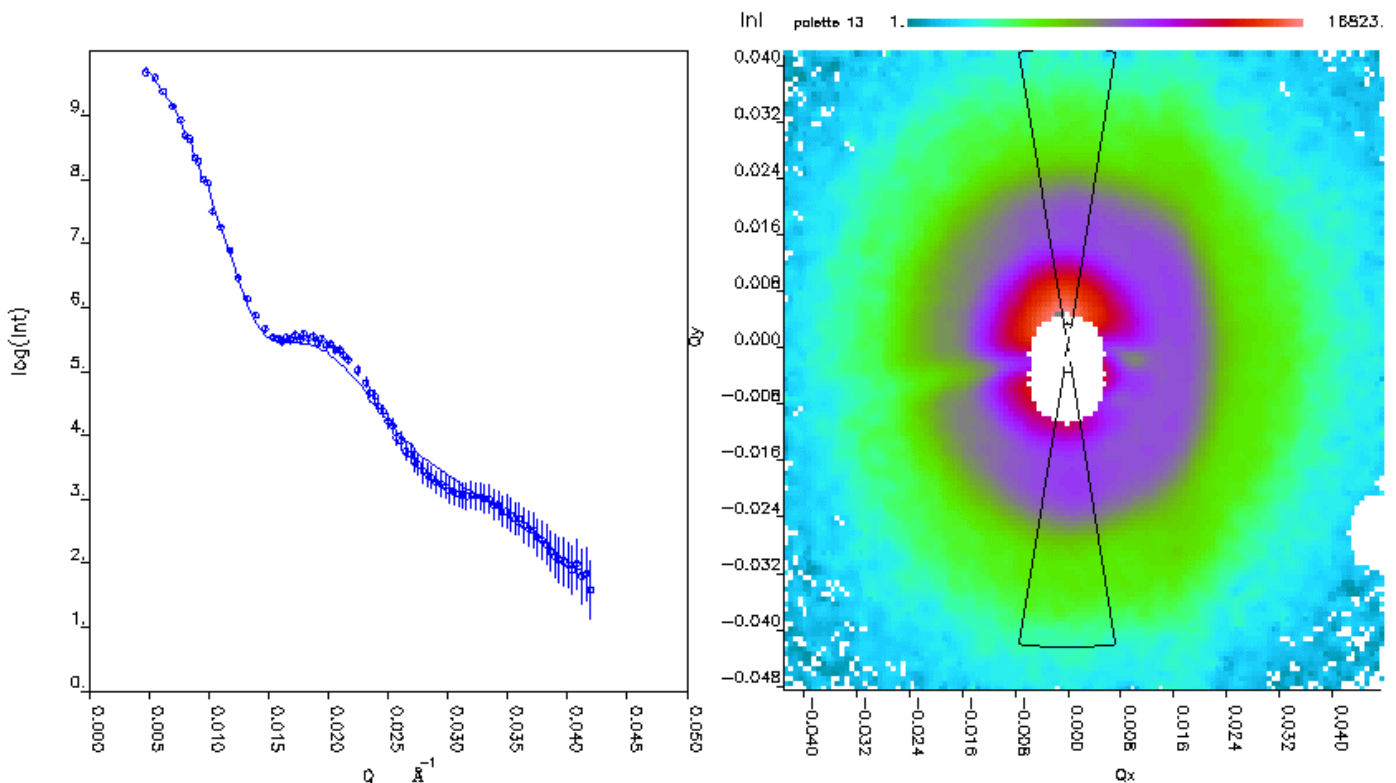


Fig 1 Fit of the spectra for a sample with channel axis coaxial to the incoming beam. The fitting function is the square of a Bessel function attenuated by a gaussian factor. The fit includes a Schutz-Zimm dispersion for the channel radius. The points displayed on the left are the pixels inside the symmetric sector on the right.

However we had the time to observe only very few samples, let alone to study full series versus the etching temperature as was foreseen. We concentrated our work on a few well known samples, in order to ascertain the method, and on new samples irradiated in GANIL.

## **Contributions**

During the experiment I installed the PXY program at ID01. The PXY program allows to visualize 2D data according to various formats. It also allows to make fits with various functions, as shown on Fig 1.

After the experiment I also proposed a method in order to stabilize the beam.

## **Conclusion**

SAXS is very powerful for studying nanochannels. The two main advantages versus SANS are

- the big scattering vector range
- the excellent wave-length resolution

## **Acknowledgements**

D. Fernandez was very helpful to dig out the relevant information in order to install PXY.

Notwithstanding a persistent bad luck, the ID01 personal, H. Metzger, M. Hamilton and F. Lesimple did all their best to make the experiment work.

M. Kocsis was utterly devoted to get detectors working.