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# SCHOOL ON SYNCHROTRON RADIATION

**6 November - 8 December 2000**

***Miramare - Trieste, Italy***

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# *Basic Experiments at the Austrian SAXS Beamline at ELETTRA*

# *&*

# *Tutorial in Data Treatment*

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# BASIC EXPERIMENTS AT THE AUSTRIAN SAXS BEAM LINE AT elettra

# &

# TUTORIAL IN DATA TREATMENT

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**Trieste, November 2000**

## GENERAL

The 4 hour afternoon (27th and 28th of November 14:00 -18:00) session at the Austrian small angle X-ray scattering beam line is ideal for students who like to get a first impression of how experiments are carried out at a synchrotron facility such as ELETTRA in Trieste, and furthermore, want to learn about the basics of data reduction and data analysis. For those who have already worked at a synchrotron site this course might be too easy, and we therefore recommend this tutorial only for beginners in this field.

## ABSTRACT

After an introduction into the beam line, its optical layout and after a short security lesson, the attendants shall measure themselves unknown samples at the Austrian SAXS beam line and write a little protocol. These experiments will be kept simple, but nevertheless all typical steps of data acquisition shall be done. Thereafter, a more sophisticated time-resolved experiment will be demonstrated by the staff on site.

The second half of the course will take place in the seminar room of ELETTRA. The different data-treatment steps shall be illustrated with help of the experiments done by you at the beam line and shall also be discussed with the presentation of further examples. Learning more about data-reduction and analysis will also be supported by the demonstration of software routines projected onto a screen.

For those who can not wait to know more about us, please have look at our beam line web page:

http://www.oeaw.ac.at/ibr/beamline/

# DEMONSTRATION EXPERIMENT: PRESSURE SCAN INDUCED FORMATION OF A TUBULAR PHOSPHOLIPID PHASE

## Introduction

Phospholipids, the main constituents of the biological membrane-matrix, display a distinct polymorphism depending on thermodynamic parameters (T, p, c). One-dimensional bilayers, two-dimensional tubular structures or three dimensional networks are only a few examples for the structural variety of the supra-molecular associates, wherein the lamellar liquid crystalline phase ($L_\alpha$) is the biologically most relevant phase (Fig. 1).

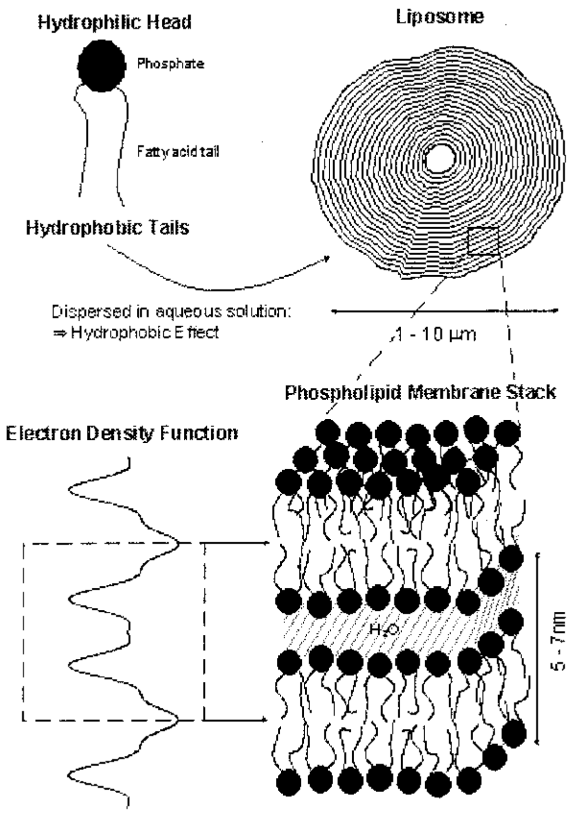


*Fig. 1: The formation of a phospholipid membrane. Phospholipids aggregate spontaneously into ordered supra-molecular structures in the presence of water. This can be explained in simple terms by the fact that phospholipids feature a hydrophilic headgroup (attracting water) and hydrophobic hydrocarbon-chains (sequestered from water). The average 1-dimensional repeat distance d, i.e., bilayer plus water layer of the depicted liquid crystalline phase ($L_\alpha$) is in the range of 5-7 nm. According to Bragg' s law this means that these superstructures show characteristic X-ray diffraction pattern in the small-angle region. The electron density distribution of a bilayer (bottom left corner) has maxima in the headgroup regions and a minimum at the methyl terminus of the hydrocarbon-chains.*

## High-Pressure Cell for Static and Time Resolved SAXS-Experiments

A compact X-ray sample cell allows to measure diffraction patterns at hydrostatic pressures up to 3 kbar. The cone-shaped exit window allows detection of scattered X-rays from the sample within a maximal angular range of 30°. The hydrostatic pressure is generated by using water as the pressure transmitting liquid, directly connected via a high-pressure control network with the cell interior. The temperature in the pressure cell can be regulated in the range from 0°C to 80°C.

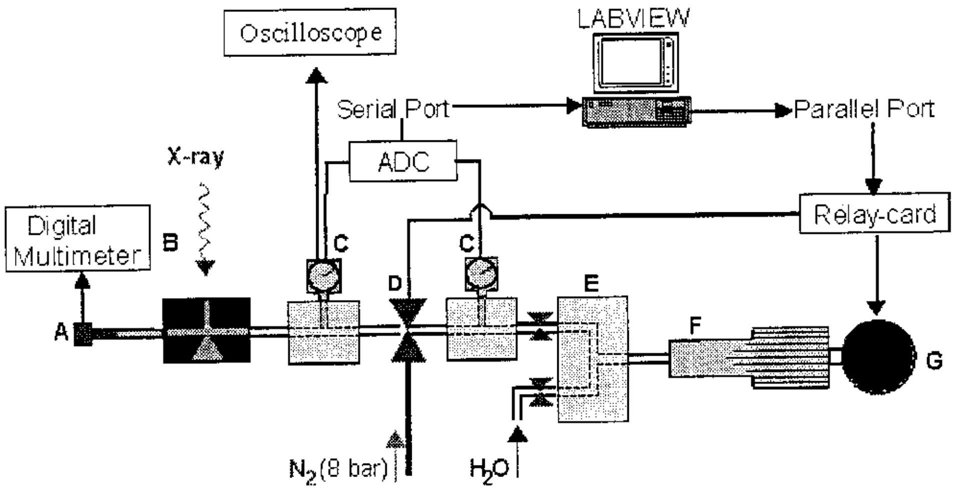


*Fig. 2: Scheme of the high pressure set-up.*

## The Formation of a Tubular Phase Under Release of Pressure

In Fig. 3 the phase sequence in the phospholipid DOPE under the release of pressure at 20 °C is shown. At high pressure (2.5 kbar) a lamellar gel phase forms, thereafter a lamellar liquid crystalline phase appears and finally, an inverted hexagonal phase is induced at ambient pressure.

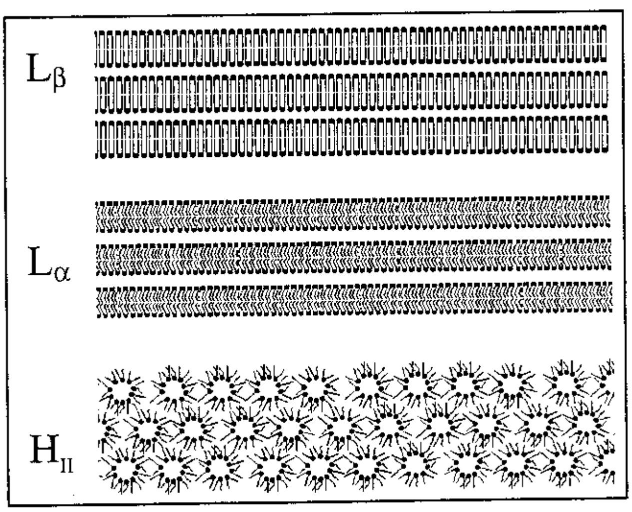


*Fig. 3: Phase sequence in DOPE*

## OUTLINE OF THE AFTERNOON'S PROGRAM (10 - max. 15 people)

- Introduction into the beam line (~ 30 min)

- My first experiment (five unknown different samples have to be characterized; ~ 2 h):

    1. Sample Preparation
    2. Placement of the Sample
    3. Experimental set-up
    4. Measurement of the sample
    5. Writing an experimental protocol

- Demonstration of the time-resolved experiment (~ 15 min)

- Discussion of the experiments & tutorial in data treatment (~ 1 h 15 min)

    1. Corrections and calibrations
    2. Data-reduction
    3. Data-analysis

# PROTOCOL

Date:

**Sample No.:**

Temperature:

Camera length:

Ring current:

Ionization chamber voltage:

Detector voltage (SAXS):

Detector voltage (WAXS):

Attenuation:

Exposure time:

Max. Intensity:

Integral intensity:

**space for the scattering pattern**