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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Ionic liquid crystals in Langmuir trough	**Experiment number**: SC-4222
Beamline: ID10	**Date of experiment**: from: 10/12/2015 to: 14/12/2015	**Date of report**: 02/03/15
Shifts: 15	**Local contact(s)**:Oleg Konovalov	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Louis Bondaz [1, 2], Nathalie Bonatout [2, 3], Ignacio Gascon [4,5], Michel Goldmann [1,3], François Muller [2]

[1] Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin BP48, 91192 Gif-Sur-Yvette, France

[2] LICORNE, ECE-Paris, 37 Quai de Grenelle, 75015 Paris, France

[3] Institut des NanoSciences de Paris, INSP - 4 place Jussieu - 75252 Paris, France

[4] Departamento de Quimica Fisica, Universidad de Zaragoza, C/ Pedro Cerbuna,12, 50009 Zaragoza, Spain

5 Instituto de Nanociencia de Aragon, C/ Mariano Esquillor s/n, 50018 Zaragoza, Spain

Report:

Ionic liquid crystals (ILC) are a class of liquid-crystalline compounds with some relevant properties, like ionic conductivity, different from those of conventional liquid crystals. Moreover, the ionic interactions in ILC tend to stabilize uncommon mesophases, such as the nematic columnar phase. ILC are promising materials for the development of ion-conductive materials ; in the design of biosensors and also as building blocks to obtain functional molecular materials by ionic self-assembly or Langmuir-Blodgett deposition. We focus on one ionic pyrazole derivatives Ipz and Ipz-2 (*Figure 1*).

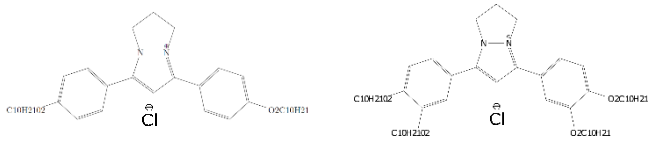

Figure 1: Structural Formulas of Ipz (lef) and Ipz-2 (right)

We have studied the behaviour at the air-liquid interface of both Ipz and Ipz-2. We have proved that the slight structural modification (four chains instead of two) significantly affects the supramolecular organisation of the compounds. We observed a remarkable difference between the supramolecular organization of Ipz and Ipz-2 in Langmuir films. Our goal was to determine the arrangement of these compounds at the air-liquid interface in the monolayer regime and after the collapse which seems to be the formation of multi-layers. These results have been considered with respect to to the liquid crystalline phases in order to understand the parameters that govern the organization of this kind of compounds in supramolecular architectures.

We performed X-Ray Reflectivity (XRR) and Grazing Incidence X-Ray Diffraction (GIXD) on both compounds at various surface pressures.

1. Ipz-2

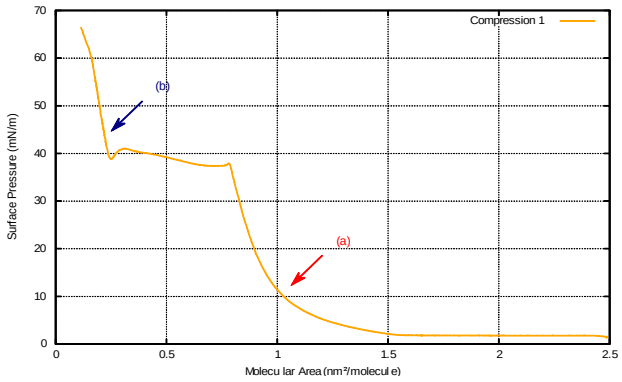

Figure 3: Isotherm of Ipz-2 at 20°C
(a) – (b) Pressures of the different diffraction and reflectivity scans

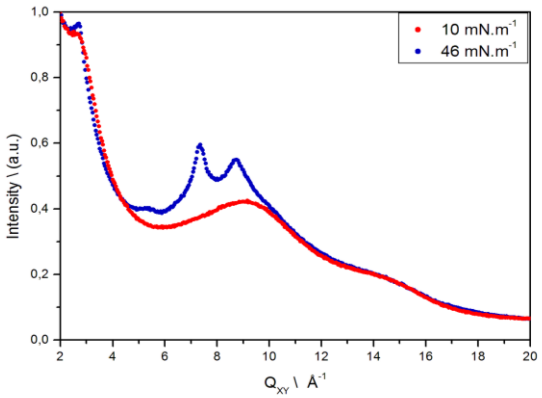

Figure 2: Diffraction Patterns of Ipz-2 before and after the collapse

At low surface pressure (before the collapse of the monolayer):

- No diffraction signal was observed (*Figure 3*) indicating a disordered monolayer.
- XRR can be adjusted by a single layer of thickness of 0.95 nm and a density of 1.805 g.cm^{-3}, which is in agreement with a single monolayer phase (*Figure 4*).

At high surface pressure (at the end of the plateau):

- GIXD evidences 3 diffraction peaks (*Figure 3*), wich can be indexed as (10), (21) and (30) of an hexagonal lattice with a parameter a = 1.13 nm. This is in agreement with the columnal structure observed for the Liquid Crystal Phase of this compound. The analysis of the Qz shape of the diffraction peaks to determine the thickness of these columns indicates a thickness of about 1 nm.
- XRR scans are fitted by a triple layer model (*Figure 5*), which can be divided as follow:
  - First layer of thickness 0.62 nm and density 0.74 g.cm^{-3}, which corresponds to the head groups of the molecules,
  - A second layer, of thickness 1.2 nm and density 0.94 g.cm^{-3}, which corresponds to tails of the molecules,
  - A third layer of thickness 1.5 nm and density 0.96 g.cm^{-3} which corresponds to a double layer of head groups,
  - The fourth layer, of thickness 0.94 nm and density 0.41 g.cm^{-3}, which corresponds to the last tail layer.

As a result, the tri-layer can be described by a first layer of oriented molecules with the chains upright at the water interface, superposed by two layers of molecules which are randomly oriented up or down. The chains are interdigited between two layers. Only the first interdigited chains and denser layer appear as organized. This confirms that the Ipz2 amphiphile character is strong enough to orient the molecules which interact with the-water subphase as the ones at the Ipz2-Ipz2 and Ipz2-air interfaces do not present any preferential orientation.

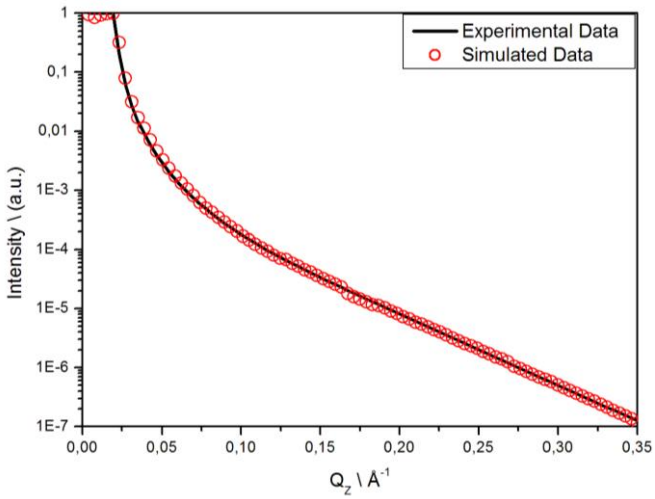

Figure 4: XRR scan of Ipz-2 at Π=10mN/m

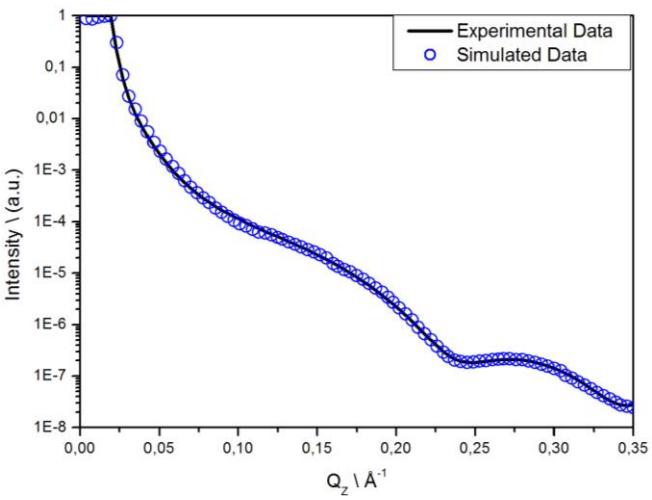

Figure 5: XRR scan of Ipz-2 at Π=46mN/m

2. Ipz

No diffraction signal was observed before or after the collapse (*Figure 7*) indicating a disordered layer.

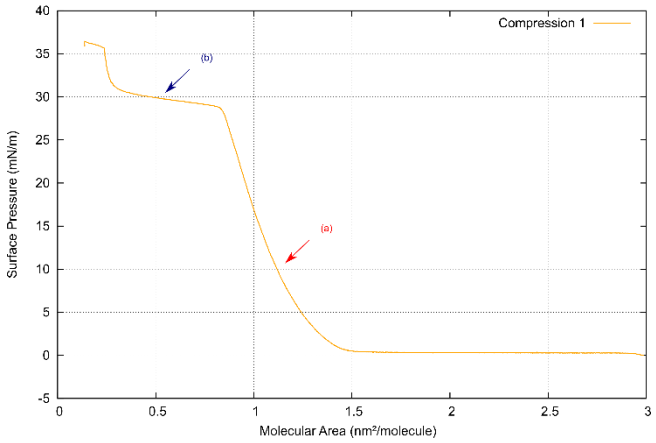

Figure 7: Isotherm of Ipz at 20°C
(a) – (b) Pressures of the different diffraction scans

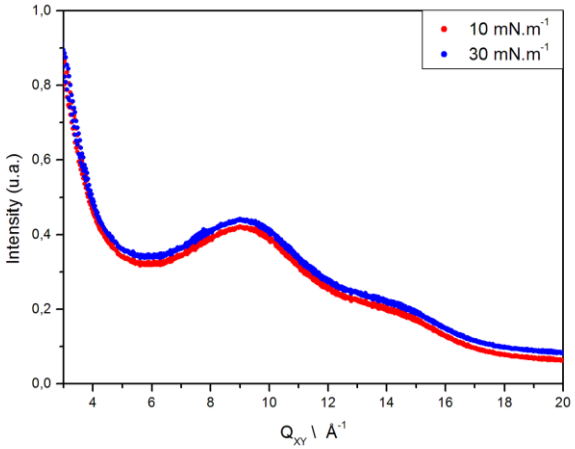

Figure 8: Diffraction Patterns of Ipz before and after the collapse

At low surface pressure (before the collapse of the monolayer):

- XRR can be adjusted by a single layer of thickness of 1 nm and a density of 1.1 g.cm^{-3}, which is in agreement with a single monolayer phase (*Figure 8*).

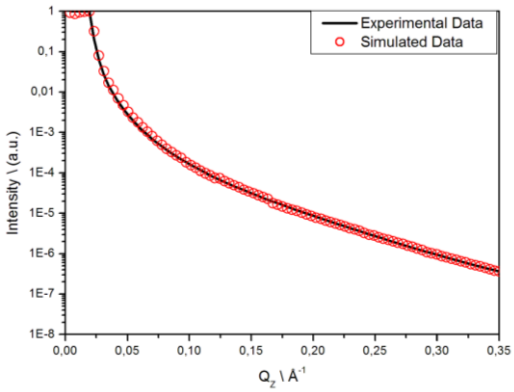

Figure 6: XRR scan of Ipz at Π=10mN/m

Spectras performed at other surface pressure and physico-chemical conditions are under treatment.