### EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



## **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.

<b>ESRF</b>	<b>Experiment title:</b> Liquid-mercury-supported Langmuir films of ionic liquids	<b>Experiment</b> <b>number</b> : SC-3765
Beamline:	Date of experiment:	Date of report:
ID10-EH1	from: 27/11/13 to: 04/12/13	12/1/2016
Shifts:	Local contact(s):	Received at ESRF:
21	Oleg Konovalov	
Names and affiliations of applicants (* indicates experimentalists):		
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#### **Report:**

The structure of mercury-supported Langmuir films of imidazolium-based ionic liquids was investigated by X-ray reflectivity (XRR) measurements. We used a Langmuir trough designed for simultaneous surface tension and X-ray measurements. Densely packed monolayers of the ionic liquids  $[C_4 mim]^+$   $[NTf_2]^-$ ,  $[C_{12}mim]^+$   $[NTf_2]^-$ ,  $[C_{18}mim]^+$   $[NTf_2]^-$  and  $[C_{18}mim]^+$   $[FAP]^-$  were deposited on the surface of liquid mercury and XRR measurements were subsequently performed. The reflectivity curves exhibited a time evolution from a smooth low-surface-roughness curve upon deposition to a modulated curve as time goes on. A dip developed and moved towards lower qz values with time, indicating the formation of a layer of increasing thickness on the mercury surface. As an example, the measured X-ray reflectivities at specified times after film deposition is shown in Fig. 1 for the ionic liquid  $[C_{12}mim]^+$   $[NTf_2]^-$ . The dip's positions as a function of time were phenomenologicaly fitted with simple exponentials and time



constants of hours were found with a systematic trend for all the ionic liquids, suggesting slow reorganization of the ions on the mercury surface, probably into polar and non-polar domains. A modified distorded crystal



model (DCM) was used for a full-curve fit of the reflectivity data and to get the laterally averaged surfacenormal electron density profile. The Fresnel-normalized reflectivities (data points) with their fits (dashed lines) are shown in Fig. 2A for  $[C_{12}mim]^+$  [NTf<sub>2</sub>]<sup>-</sup> and the corresponding density profiles of the ionic liquid layer (with the mercury density profile substracted) are given in Fig. 2B (with electron density of mercury as unity). The charge-delocalized ionic liquids studied here exhibited no 2D lateral order but showed diffuse surface-normal electron density profiles exhibiting gradual mercury penetration into the ionic liquid film. For the last measured scans, mercury concentration gradients with lower electron densities were observed for the ionic liquids with longer non-polar alkyl chains, suggesting that the affinity for mercury is reduced with increasing non-polar alkyl chain.

A full analysis of the measured results for all ionic liquids studied has been completed and is now being prepared for publication as a paper.