EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** Short range and critical diffuse scattering in quasi-liquid guests confined crystals, leading to aperiodic modulated composite.	**Experiment number**:
Beamline: BM01A-SNBL	**Date of experiment**: from: 4 may 2016 to: 10 may 2016	**Date of report**: 09/09/2016
Shifts: 18	**Local contact(s)**: Dmitry Chernyshov	*Received at ESRF:*

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Report:

Alkane (C_nH_{2n+2})/urea compounds are prototype examples of confined liquid phases in aperiodic crystals. We performed an experimental study of the phase transition from the quasi-liquid state to the long range order in these composite crystals. To achieve this, we followed the evolution of the diffuse scattering of five compounds with the shortest guest molecules: n from 8 (octane) to 12 (dodecane). Measurements consisted of full data acquisition for different temperature in the limit range from 260K down to 100K depending on the compound. These evolutions give us key information on the critical phenomena and the origin of the phase transition to the aperiodic solid.

Experimental conditions:

Full data collection with single axis rotation and 0.4° rotation step were performed at 17.07keV energy (λ = 0.68 Å). The Dectris Pilatus 2M detector was placed at distances of 150, 200 and 400 mm from the sample according to the required resolution. The synchrotron was operating in 16 bunch filling mode (90mA). Temperature was controled using a nitrogen cryo-cooler.

Results:

The experimental study was very successful allowing the study of five compounds: octane (n=8), nonane (n=9), decane (n=10), undecane (n=11) and dodecane (n=12). The quality of the data are excellent allowing a very precise study of the sequence of phases in these composite organic crystals. These data are shown for example in the case of decane/urea (see figure).

Data analysis are now under progress. The preliminary results give us very important information on the ordering phenomena in these materials. This is illustrated in the fig. d) where a very strong modification of the intensity is observed on the semi-log scale.

A first consequence of these reliable data with the very high resolution and high quality of the crystals is that our rough knowledge of the phase diagram is seriously modified. In particular, the transition temperatures from quasi-liquid phase to solid state, defined by the appearance of sharp Bragg peaks, is actually much higher than what we announced up to now. In addition, new phases have been revealed for instance a monoclinic four dimensional phase in decane/urea (see fig. c, low intensity Bragg peaks)

Of course since our set of temperature was determined according to our knowledge before the experiment it appears that we lack information for several compounds at either high or at low temperature, which should require a complementary similar experiment.

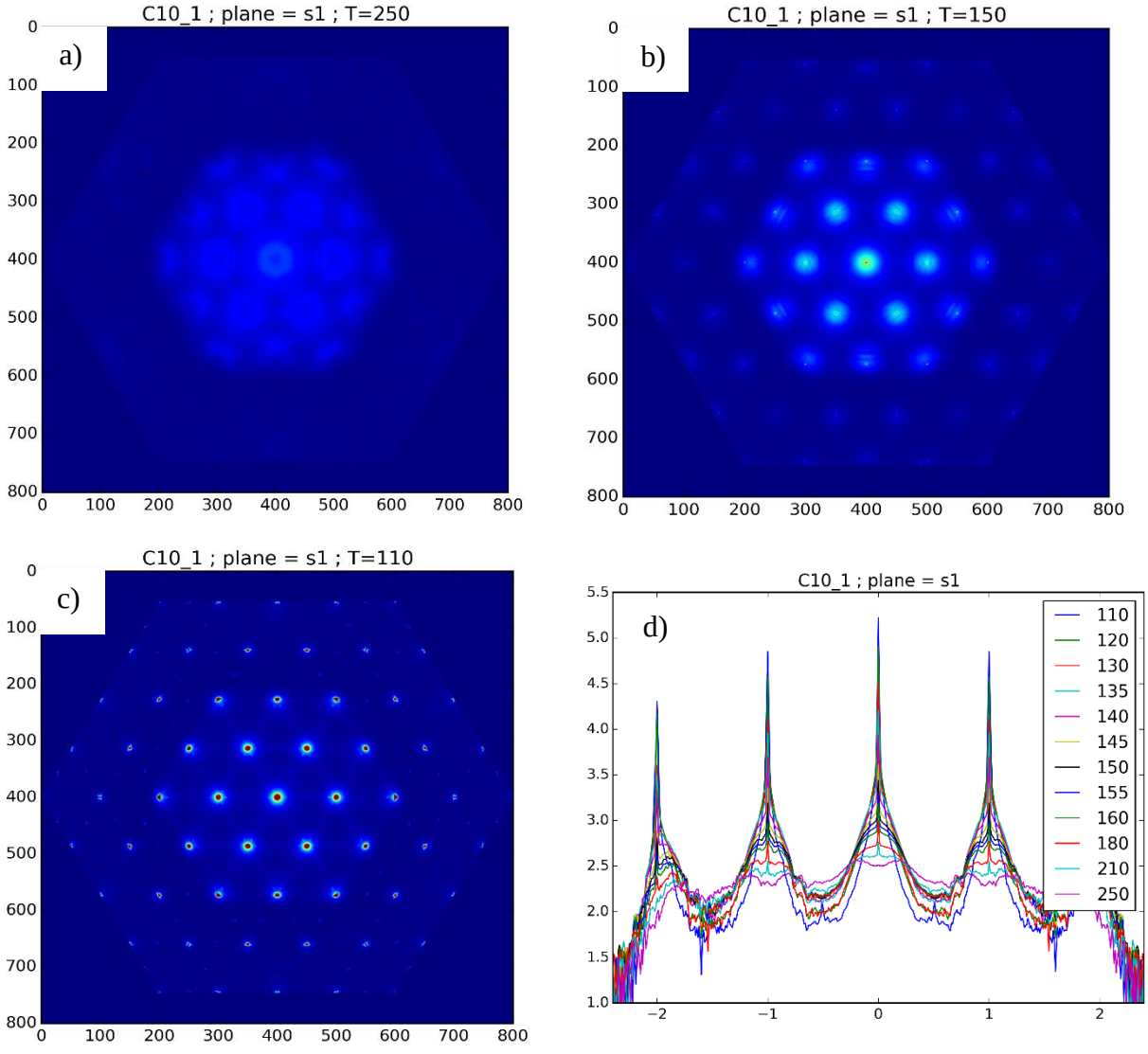

FIG.: (a) to (c) reconstructed diffuse scattering planes in decane/urea (a) in the liquid-like phase,(b) in the intermediate four-dimensional hexagonal phase, and (c) in the low symetry four-dimensional monoclinic phase. (d) Temperature evolution of the logarithm intensity profiles along the $\mathbf{a}^*$ direction showing the appearance of Bragg peaks between 250K and 210K and the monoclinic structure at 110K

References

Alfredo Salazar, Master thesis Erasmus Mundus Mamaself, Univerté de Rennes 1, July 2016.