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.

ESRF	**Experiment title:** Mechanism of Degradation of Frozen Proteins:Investigation of Protein-Ice and Protein-Solute Interactions by Monitoring Ice and Solute Crystallization using High-Resolution XRD	**Experiment number**: CH-5600
Beamline:	**Date of experiment**: from: 08 Nov 2018 to: 11 Nov 2018	**Date of report**: 17 Sep 2021
Shifts: 9	**Local contact(s)**: Gaston Garbarino (email: **gaston.garbarino@esrf.fr**)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Controlling polymorphism of drugs is important for both fundamental science and pharmaceutical applications. High-pressure polymorphism is recognised as a necessary part of modern polymorph screening. Chlorpropamide (CPA) is widely used as a model system to study polymorphism in drugs. It has five conformational polymorphs at ambient conditions, as well as multiple variable-temperature and variable-pressure forms. To date, all documented CPA phases have been periodic. In our previous ESRF experiment (CH-4901) we discovered a unique behaviour of δ-CPA on hydrostatic compression: it forms an incommensurately modulated high-pressure phase, which then transforms on further compression into a periodic superstructure with one of the cell parameters tripled as compared with the original δ-CPA. Unfortunately, the I/σ(I) ratio and data completeness achieved at the BM1A was not sufficient to solve and refine the structures of the high-pressure phases. Therefore we initially applied for a beamtime at the undulator source ID15B. We were allocated the beamtime at ID27 instead. We were able to reproduce experimental results which were previously obtained at BM01A but unfortunately the data quality was still not high enough, to increase data quality and to solve the structure of modulated phases. One of the reasons can be using less efficient CCD detector at ID27 whereas HPC detector at BM01A appeared to provide comparable data quality even with less efficient source. The results obtained at ID27 and its comparison with results previously obtained at BM01A are valuable from methodical point of view and were reported at the series of international conferences:

1) Zakharov B.A. Studying Crystals at High Pressures: Do We Know all the Factors Influencing Data Quality? Abstract book of the 13th High-Pressure Diffraction Workshop In Poznan.2020.– P.22.

2) Zakharov B., Boldyreva E. Studying Molecular Crystals at High Pressures: Experimental Strategy and Hardware Matters, 32nd European Crystallographic Meeting ECM32. Book of Abstract.2019.– P.676.MS42-P13

3) Zakharov B.A., Boldyreva E.V. Selecting Hardware and Experimental Strategy for High-Pressure Single-Crystal X-ray Diffraction Studies, Conference proceeding 2019 IUCr & ECA High pressure workshop. 2019