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*

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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:** Structural origins of baracaloric properties in hybrid plastic crystals	**Experiment number**: A01-2-1262
Beamline: BM01	**Date of experiment**: from: 03/05/2022 to: 09/05/2022	**Date of report**: 19.09.2023
Shifts: 18	**Local contact(s)**: Charles Mcmonagle	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Julian Walker – Norwegian University of Science and Technology, Norway
Mari-Ann Einarsrud – Norwegian University of Science and Technology
Charles Mcmonagle – ESRF
Jorge Salgado Beceiro – University a Coruna, Spain
Manuel Sanchez – University a Coruna, Spain

Report:

The experiment was to look at powder diffraction of a variety of plastic crystal mateirals – ionic supramolecular materials – in situ with both variable temperature and variable pressure.
The experimental equipment enabled both isothermal-variable pressure and isobaric variable temperature measurements.

The variable temperature utilized the cryostream to control temperautre between 200K and 500K – this set up had been proven to work exceptionally well for these mateirals in the past and was a continuing success.

The variable pressure was applied utilizing two difference pressure cells. First a gas based pressure cell, which was very effective but had manual control of gas inside the hutch so the experiemnt was labour intensive.

The second pressure cell was a custom built sell – made by Charles Mcmonagle – which utilized hydralic fluid to apply the pressure to a quartz capillary.
This was also highly successful, however, there were a few key challenges.

1. the gratest challenge encountered was that the sample would gradually move along the capillary and out of the beam during sweeps of increasing and decreasing pressure. This meant that several sampels had to be prepared.
2. The preparation time for getting the materials in and out of the specialized quartz capillary was relatively long – 1 hour.
3. The temperature sweeps were limited due to the thermal mass of the capillary holder and high thermal conductivity of the capillary, which also meant that significant time was needed to stabilize the temperature between each thermal step.

In all this experiemtn was highly successful and the new presure cell enabled us to map out the pressure and temperature phase diagrams fro a number of new promising barocaloric plastic crystal materials.