EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

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:** Anisotropy of strain in pharmaceutically relevant materials for description of brittleness	**Experiment number**: CH6147
Beamline: ID09b	**Date of experiment**: from: 09/10/2021 to: 12/10/2021	**Date of report**: 04/09/2021
Shifts: 9	**Local contact(s)**: Davide Comboni	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Iain Oswald, University of Strathclyde

Julia Gasol-Cardona, University of Strathclyde

Martin Ward, University of Strathclyde

Report:

This beamtime investigated the compression of a number of different antibiotics under hydrostatic and non-hydrostatic environments. During this time, we were somewhat limited by the detector (see below) hence did not collect data on as many samples as was initiatlly proposed. From the data we collected, we were able to compare the compression of ofloxacin, anhydrous levofloxacin and levofloxacin hemi-hydrate. The compression data for ofloxacin is shown in Figure 1 as an example. Pawley refinement of all the data has been performed on these systems and we have been able to determine the Bulk modulus for each of these systems (Figure 2).

We are able to observe some differences in the behaviour between the non-hydrostatic and hydrostatic compression. The non-hydrostatic environment is indicating a lower rate of compression but feedback from conferences has provided a few different experimental avenues to explore that might take into account these differences. We have supplemented the powder diffraction data with in-house single crystal diffraction in the ofloxacin system over the past few months. These data fit well with the powder diffraction data and enables us to explore the system even further. The single-crystal analysis of the levofloxacin and levofloxacin hydrate system are still to be examined.

This data has been presented at two conferences orally (European Crystallographic Association meeting in Versailles; Cambridge Crsytallographic Data Centre Science Day (Youtube), and as a poster (ECM, EPSRC CMAC Open day).

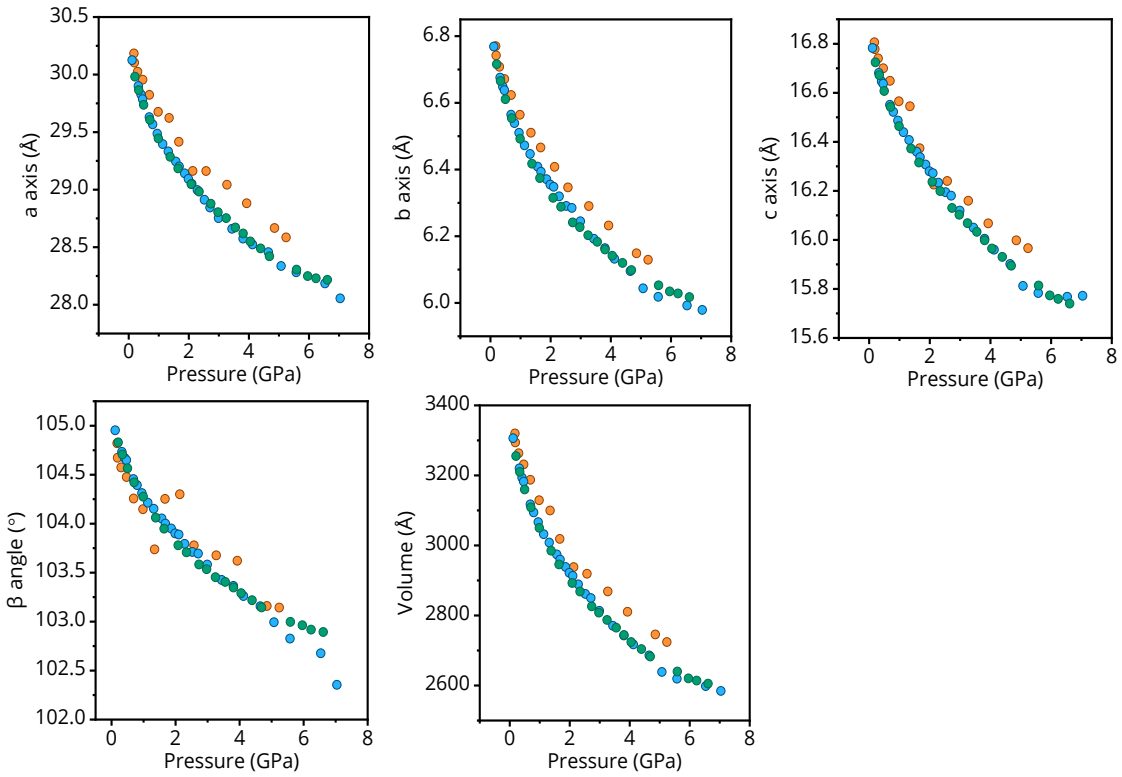

Figure 1: Unit cell parameters and volume of ofloxacin as a function of pressure from Pawley fitting of the data. The samples were compressed in (blue) 4-1 methanol:ethanol, (green) petroleum ether, and (orange) no pressure transmitting medium.

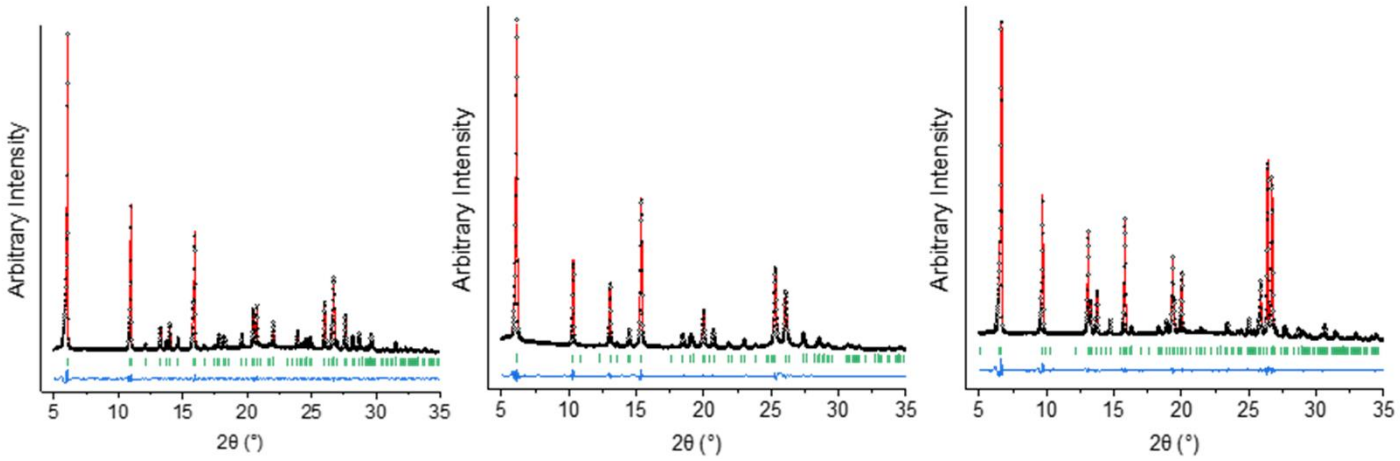

Figure 2: Pawley-fitted X-ray diffraction data of (left to right) ofloxacin, levofloxacin anhydrate and levofloxacin hemihydrate

Key issues: The detector was causing issues througout the beamtime with panels missing from the data. This led to multiple patterns being collected to ensure we had quality data for the compression. The detector was investigated during lunchtime of our final day. This led to the beamtime being cancelled (it was fixed later in the day after packing our samples). Davide collected a set data for of anhydrous pipemidic acid to account for our lost day of beamtime.