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:** The effect of the back-bone length on the high-pressure transformations in crystalline amino acids	**Experiment number**: SC-3821
Beamline: BM01A	**Date of experiment**: from: 18/06/2014 to: 21/06/2014	**Date of report**: 05/08/2014
Shifts: 9	**Local contact(s)**: Vladimir Dmitriev, Tel: 0476882851, Email: dmitriev@esrf.fr	*Received at ESRF:*

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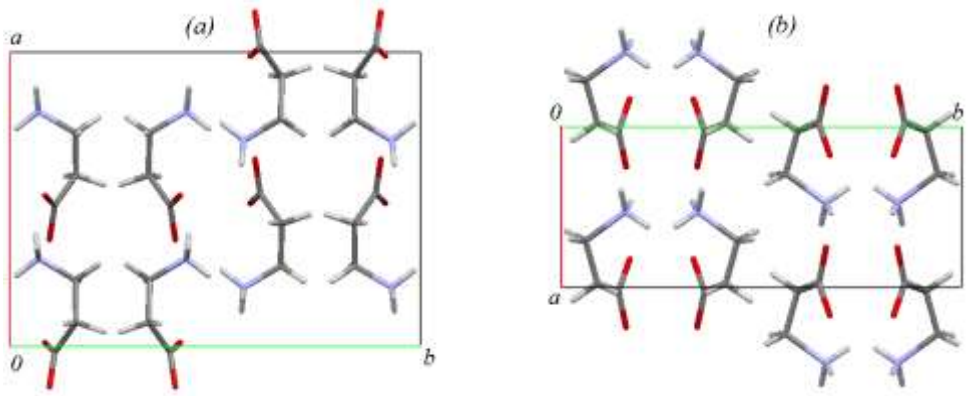
Report:

The main aim of the title experiment was to solve crystal structures of high-pressure polymorphs of β-alanine and to compare structures of β-alanine at high pressure depending on the way how this pressurre was reached. We have performed several measurements of different β-alanine single crystals at high hydrostatic pressures. To achieve maximum possible data completeness we have collected four datasets with different diamond anvil cell (DAC) orientations at each pressure point. This allowed us to solve crystal structure of β-alanine at different pressures. In the first experiment we have solved crystal structures of β-alanine at pressures till 6.2 GPa that took ~10 hours after beginning the experiment. After that we left the sample at 6.2 GPa and after next 10 hours we have visually detected a fragment of a new phase growing at the "parent" crystal. Diffraction data allowed us to confirm the fact that we have several phases in DAC, including remaining part of initial crystal and several single-crystalline blocks of a new phase. Using this data we have calculated cell parameters of new phase which were also measured during preliminary experiments at laboratory X-ray diffractometer. Intensity data were sufficient to solve crystal structure by global optimization in direct space and refine crystal structure in isotropic approximation. Crystal structure fragments of initial and high-pressure phases are shown on the figure.



Initial phase of orthorhombic β-alanine (a) crystallizes in space group *Pcab* while monoclinic high-pressure form (b) is monoclinic with space group $P2_1/a$ that was shown earlier in X-ray diffraction experiment with powder sample. Unfortunately influence of further increasing pressure on this sample was not studied due to continuous transition from initial phase to high-pressure phase at ~6 GPa. After 24 hours after beginning the experiment there were no initial phase in DAC and polycrystalline high-pressure phase was formed. In the next experiment we have increased pressure straight up to 8 GPa. In this case we did'nt see the phase transition, and only deformation of initial phase has been detected. Taking into account previous experiment we left the sample at 8 GPa for several days but no any changes could be visually detected during this time. Thus "prolonged" high-pressure phase transition was detected only for β-alanine at 6 GPa and there were no transitions at higher pressures. One can conclude that this transition is prolonged and strongly depends on the way how we reach high pressure but not only on pressure value. The effect of pressure on the γ-aminobutyric acid was also studied. Since the sample underwent strong fragmentation in the course of a phase transition, we had to switch to powder diffraction technique. The set of diffraction patterns was measured at pressures up to 10 GPa with a step of ~0.5 GPa. The data are being processed.