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

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:** High-pressure polymorphism of chlorpropamide in 3 hydrostatic fluids. Solid-state transitions in 5 different starting polymorphs vs recrystallization into different	**Experiment number**: CH-4526
Beamline: BM01A	**Date of experiment**: from: 08/10/2015 to: 11/10/2015	**Date of report**: 05/11/2015
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 study the role of fluid phases used to provide hydrostaticity of compression on structural rearrangements in the crystals of chlorpropamide. Chlorpropamide is known as one of the drug molecules which is very prone to polymorphism in the solid state. The polymorphs differ in molecular conformations and molecular packing, but usually preserve a common hydrogen-bonded motif – bands with robust "urea core". In this series of experiments we aimed to compare the structural rearrangements induced by hydrostatic compression in the polymorphs of chlorpropamide if a) the pressure-transmitting medium can hardly interact with the solid (He, Ne), b) the pressure-transmitting medium does not dissolve the solid, but surface interaction – wetting – is strong, c) the pressure-transmitting medium сат dissolve the solid so that a new high-pressure phase can crystallize from solution. During the experiments we have compared the effect of pressure on three polymorphs of chorpropamide – α-, β-, γ- polymorphs. Each of these polymorphs was studied on compression in different media: a) He / Ne gas, b) paraffine, c) penthane-isopenthane mixture. No significant effect of pressure-transmitting medium on the pressure-induced phase transitions has been observed for α- and γ- polymorphs. The same high-pressure phases were obtained, maybe at slightly different pressures. For the β-polymorph, however, the response of the starting crystal structure to increasing pressure following exactly the same compression – decompression protocol was radically different. Different high-pressure polymorphs were obtained in different media. Their structures were successfully solved and refined. The results are now being analyzed and prepared for publication.

Polymorph	α	β	γ
Space group	$P2_12_12_1$	*Pbcn*	$P2_1$
Z	**4**	8	2
a, Å	**5.230(2)**	14.777(3)	6.126(2)
b, Å	**9.088(2)**	9.316(4)	8.941(6)
c, Å	**26.673(6)**	19.224(5)	12.020(4)
β, °	-	-	99.68(3)
V, Å^3	**1267.6(6)**	2646(1)	648.9(5)
Density (Mg m^{-3})	**1.450**	1.389	1.416
Orientation of alkyl tail	**I**	II	II
Conformation of the propyl tail	*trans*	*trans*	*trans*
H-bonded ribbon type	**z**	π	z