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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:**	**Experiment number**: SC2235
Beamline: ID11	**Date of experiment**: from: 18th May 2007 to: 22nd May 2007	**Date of report**:
Shifts: 9	**Local contact(s)**: Caroline CURFS (e-mail: curfs@esrf.fr)	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Sanjay Rastogi*

Jules Harings*

Sainath Vaidya*

Report: A symmetrical, hydrogen bonded low molecular weight molecule N,N'-1,2-ethanediyl-bis(6-hydroxy-hexanamide), either crystallized from the melt or from superheated water, is examined from a structural point of view. Electron exchange between hydrogen bonded motifs on one hand and conformational changes in the aliphatic sequences on the other, govern the thermally triggered crystalline transitions in correspondence to nylons. However, limited conformations of the short central diamine methylene moieties induce a rather peculiar triclinic crystalline structure with potential existence of intersheet hydrogen bonding. Crystallization from superheated water entails remarkable differences in the physical behavior. A novel crystalline structure, where the influence of the aliphatic diamine segment is suppressed, transforms via sequential temperature cycles gradually into another crystal, in which the hydroxylic protons seem to participate in the unit cell. Nevertheless, the role of particular water molecules, either strengthening or weakening hydrogen bonding, remains unambiguous.

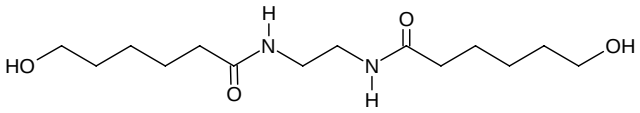


Figure 1: Chemical structure of N,N'-1,2-ethanediyl-bis(6-hydroxy-hexanamide) with two centrally positioned amide moieties and two hydroxylic end groups.

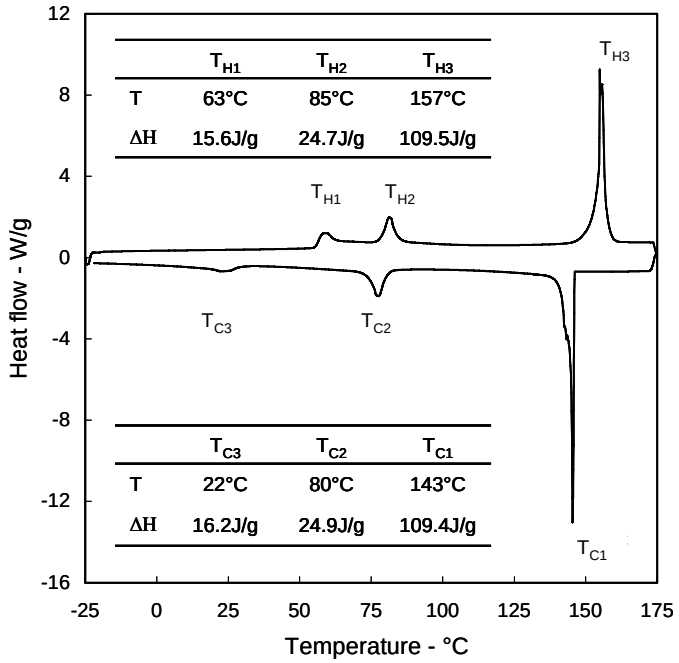


Figure 2: DSC thermogram of melt crystallized EDHA. The tables present the transition temperatures and the corresponding enthalpies

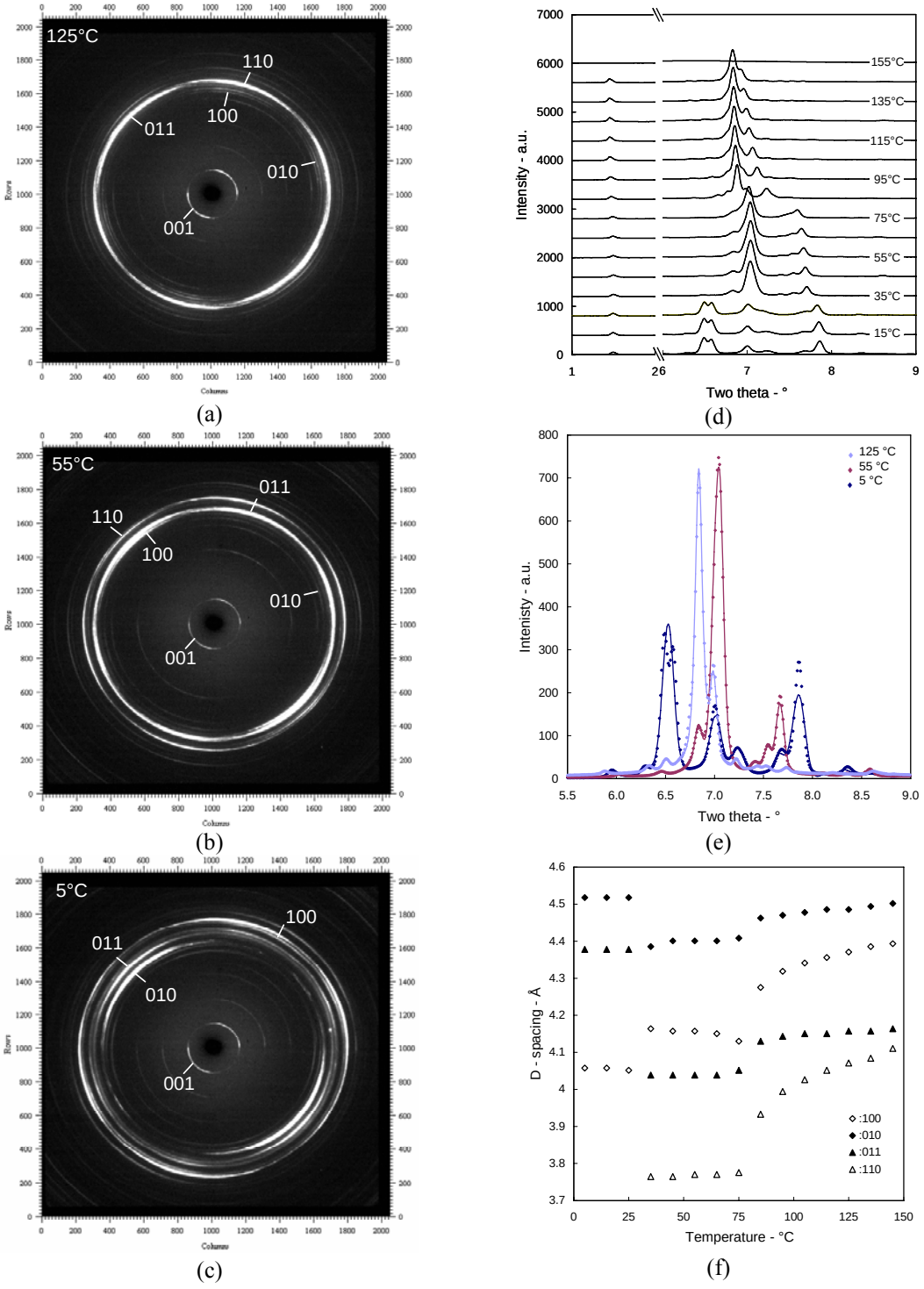
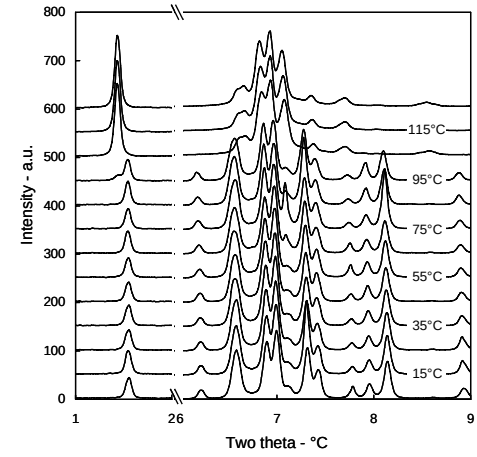
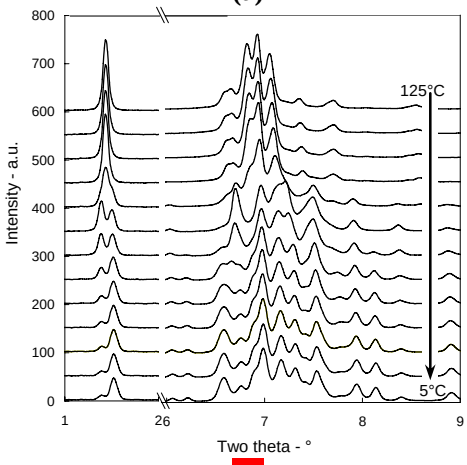


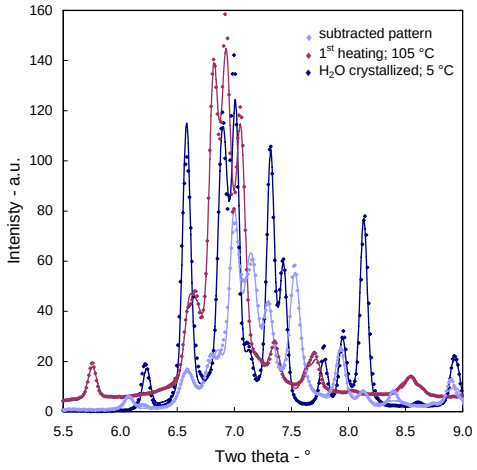
Figure 3: Structural changes by time resolved WAXD as EDHA was cooled from the melt at 10 °C/min. Diffraction images at representative temperatures, (a), (b) and (c), in which the projected length (00l) and the interchain (010/011) and intersheet (100/110) reflections are labeled. Careful analysis of the reflections as function of temperature (d), resulted in the selection of representative diffraction patterns that were used for powder indexing, where the diamonds and lines represent the experimental and simulated patterns respectively (e). As such, the variations of the interchain and intersheet spacings could be followed as function of temperature (f).



(a)



(b)



(c)

Figure 4: Structural changes in water crystallized EDHA at 95 °C on heating (a) and two different crystalline rearrangements (b) of which the first transition results in a novel crystal with a diffraction pattern closely matching the subtracted pattern in figure (c). Here, correlations between the experimental (diamonds) and simulated (lines) diffraction patterns, after powder indexing and refinement, are presented.

Table 1: Reflections and unit cell parameters of melt crystallized EDHA in its three crystalline forms

temperature	system	(010)	(100)	(011)	(110)	*a*	*b*	*c*	*α*	*β*	*γ*	*V*
°C		nm	nm	nm	nm	nm	nm	nm	°	°	°	nm^3
125	triclinic	0.449	0.437	0.416	0.407	0.475	0.483	1.96	93.2	98.3	68.2	0.414
55	triclinic	0.440	0.416	0.404	0.377	0.456	0.482	2.02	102	77.7	72.7	0.396
5	triclinic	0.452	0.406	0.438	-	0.432	0.478	1.91	90.4	92.4	110	0.372

Table 2: Reflections and unit cell parameters of water crystallized EDHA prior[1] and after two sequential temperature cycles[3]

temperature	system	(010)	(100)	(011)	(110)	*a*	*b*	*c*	*α*	*β*	*γ*	*V*
°C		nm	nm	nm	nm	nm	nm	nm	°	°	°	nm^3
5[1]	triclinic	0.432	0.412	0.400	0.383	0.469	0.497	1.96	109	107	63.2	0.379
125[1]	triclinic	0.427	0.417	0.410	0.387	0.481	0.478	2.09	102	108	63.9	0.407
5[3]	triclinic	0.432	0.418	0.406	0.378	0.470	0.482	1.99	106	107	65.2	0.385