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:**	**Experiment number**: HC-1763
Beamline: ID11	**Date of experiment**: from: 11/05/2015 to: 12/05/2015	**Date of report**:
Shifts: 3 (24h)	**Local contact(s)**: Jonathan Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Isabelle Gautier-Luneau*, Institut Néel, UPR2940, Grenoble

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Report:

The proposed study concerns mineral iodate material (NaI_3O_8, a promising material for infrared parametric generation),which necessitate accurate analysis of the iodine charge density to be further correlated to NLO properties. Modeling the electron density of inorganic materials containing heavy elements is very far from a routine work: accurate absorption and extinction corrections are the key for a realistic electron density modeling and can only be performed with synchrotron radiation.The main difficulties are

- Extinction: extinction coefficient is around 0.5 for (11-1) (111) (201) and (220) reflections for NaI_3O_8 structure (as refined with MoPro using Ag radiation (0.54 Å) home diffractometer data). So, to minimize extinction, the wavelength should be short (0.15-0.3 Å).
- High core/valence number of electrons ratio for the iodine atom (53/7). Normalized valence scattering factors of iodine decrease very quickly versus ($\sin\theta/\lambda$) (Normalized f_{val} iodine is only 0.002e at 0.25 Å^{-1}) which therefore requires very accurate low resolution data. This is a real challenge due to extinction.
- Iodine presents strong absorption which must be accurately corrected: therefore the choice of the short wavelength limits absorption. Wavelengths around the K absorption edge of iodine (33.17 keV, 0.373 Å) should be avoided.

As the previous experiment was finished earlier, we started our experiment the previous day at 17:00. So we took advantage of 15 hours additional shift.
The experiments were performed on the ID11 beam line, using a two-dimensional CCD detector, at the shortest possible wavelength (0.15815 Å, E= 78.3916 keV) at low temperature (120 K and 30 K) to the highest possible resolution (at $\sin\theta/\lambda \approx 1.25$ Å^{-1}) to ensure a proper deconvolution of the electron density from thermal motion.

Two selected crystals were rounded by controled dissolution to make them almost spherical, to minimize absorption and absorption anisotropy effects: crystal #1 84 < size < 94 µm ; transmission = 90,3 %; crystal #2 67 < size < 99 µm; transmission= 89 to 92%) .
Both were measured at 120 K, and only crystal #1 at 30 K.

The first calculations, realized during experiments, showed that it was necessary to recalibrate the detector (frelon camera) for later optimal data corrections. Fortunately, our local contact managed to perform this sensitive task not only by obtaining a sample that could be used as standard during our shifts but also by devoting more time after our runs to obtain a good calibration of the detector's pixels.

Ultra high resolution data set has been collected at 78.3916 KeV; $\sin\theta / \lambda$ = 0.09-1.25 Å^{-1}; d= 0.23-5.73Å;
The corrections due to the detector have been performed.
Currently, the data analysis is in progress.