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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: Single crystal diffraction experiments on birnessite**	**Experiment number**: CH1032
Beamline: ID13	**Date of experiment**: from: 05 April 2001 to: 08 April 2001	**Date of report**: 31/08/2001
Shifts: 6	**Local contact(s)**: Dr David Flot	*Received at ESRF:*

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Report:

This experiment is a continuation of the CH923 experiment. In order to complete the promising results obtained during this latter experiment, we focused our attention on sample K151, which has an hexagonal unit cell, probably without twinning. The main aim of the present experiment was to improve the resolution, which was previously too low (0.82 Å) to perform a structure refinement with a satisfactory ratio number of reflections over number of parameters.

As outlined in the previous report, the crystal quality is difficult to assess during the mounting stage because of the very small size of the crystals (~5 µm in their largest dimension). As this sample preparation stage is tedious and time-consuming it was decided for this experiment to test crystal quality with a new technique, using the specific features of the ID13 micro-diffractometer (enlarged sample visualization, easy translation + alignment procedures).

The sample is first dispersed on a kapton foil and diffraction images from individual crystals selected on the kapton foil are recorded using a limited oscillation range. When a crystal suitable for structure refinement is encountered, it is then mounted as usual on the tip of a glass capillary to perform a classical data collection with the oscillation method. This crystal selection method allowed us to obtain two good crystals. It should be noted that because of their extremely small size (< 3 µm in their largest dimension) these two crystals would likely not be selected by using the usual mount-and-test procedure.

With the first « good » crystal, we recorded data sets at different wavelengths. Beamsize was 4 µm diameter. It seemed that the best compromise between short wavelength, low background and exposure time was obtained using λ=0.729 Å, as for shorter wavelengths the flux is significantly decreased and the background significantly increased because of W fluorescence. However, the resolution obtained using this new

wavelength (~0.73Å) was still too low to satisfactorily carry out a structure refinement of this sample, the number of independent reflections being increased from 60 to 95.

To further increase the number of recorded reflections, it was decided to use the new ID13 detector arm. After several tests to determine the optimal compromise between the tilt angle and the detector distance, which had to be increased because of clutter problems, it was possible to decrease the resolution limit down to 0.55 Å and, as a consequence, to about double the number of recorded reflections (185 as compared to 95 with no tilt angle). We finally recorded on this sample 4 complete data sets with (40° - 75 mm) and without (0° - 45 mm) detector tilt at both room temperature and 100K.

Although data analysis is not straightforward because available data reduction softwares (DENZO, XDS) are usually optimized for protein crystallography images containing much more reflections per image but we can reasonably hope to solve the structure using the available data. Data analysis is still in progress.

Image below : diffraction pattern of a K151 crystal (wavelength: 0.729Å; oscillation range: 6°, detector tilt: 40° - detector distance ~75 mm)