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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 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: Unknown crystal structures of selected uranyl sulfates: solution from the high resolution powder data combined with EXAFS spectroscopy**	**Experiment number**: CH-3085
Beamline:	**Date of experiment**: from: 14. 5. 2010 to: 18. 5. 2010	**Date of report**: 16. 12. 2010
Shifts: 12	**Local contact(s)**: Dr. Carsten Baehtz	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Jakub Plášil*, National Museum, Václavské nám. 68, Prague 1, 11579, Czech Republic

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Report:

The main objective of the research was to collect high-resolution powder diffraction data on selected uranyl sulfate minerals to provide more structural information on their unknown crystal structures. We have collected data for 3 natural uranyl sulfates – jáchymovite, johannite and rabejacite. They were chosen from the broader selection, but else phases were found to be not quite stable after grinding.

There are considerable difficulties arising from the complexity of the crystal structures of these phases and for dominant U-scattering power. For these reasons the data obtained from the experimental setting of the beamline BM-20 are not sufficient for solving the structures of jáchymovite (found to be a mixture of two phases, probably) and rabejacite, ab-initio. However, the new unit-cell parameters for both phases are proposed and currently are under evaluation. In case of johannite, the Rietveld refinement of its crystal structure was carried out, regarding Mg contents entering the cationic site instead of Cu. Results are acceptable and will be published along with the chemical/structural characterization of johannite samples from different environments (Plášil et al. in prep.).

Besides these three phases, an additional set of samples were measured. The set consists of UO_2^{2+} containing natural phases (J. Plášil), two minerals from the Cu-Se system (J. Plášil) and two samples of pharmaceutical phases (Dr. Hušák). Most promising results carried out an diffraction experiment on the naturally occurring $U(HAsO_4)_2(H_2O)_4$ (Fig. 1).

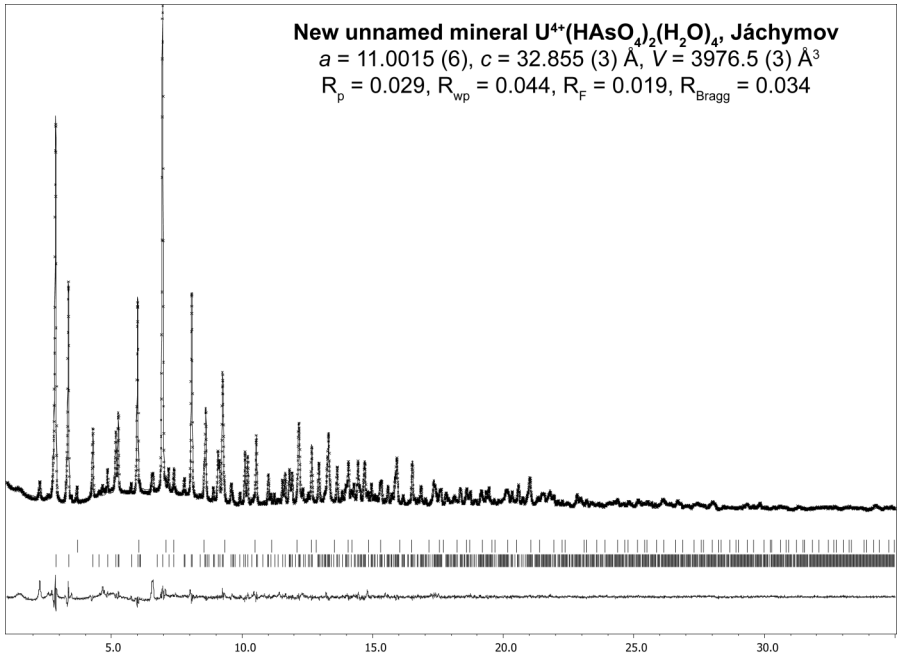

Fig. 1. The final plot from the Rietveld refinement of the new unnamed mineral from Jáchymov, Czech Republic. The data were collected from the mixture, consisting mainly of the new mineral, arsenolite (Bragg reflections bars above those belonging to new mineral) and else, unidentified mineral phase (positive maxima in the difference plot).

The proposal for approval this phase as a new mineral is now prepared to be submitted to the CNMNC of the International Mineralogical Association. Powder diffraction data were further collected for widenmannite, an unique Pb-UO_2^{2+} carbonate. Its crystal structure is now under evaluation (in combination with PED/TEM). The crystal structure of flavine2 (10-Methylisoalloxazine 5-oxide), one of the pharmaceutical phases measured, has been published yet in Acta Crystallographica E (Rohlíček et al. 2010):

The title compound [systematic name: 10-methylbenzo[g]-pteridine-2,4(3H,10H)-dione 5-oxide], $C_{11}H_8N_4O_3$, consists of a large rigid isoalloxazine group which is approximately planar (r.m.s. deviation = 0.037 Å). In the crystal, intermolecular N–H···O hydrogen bonds link the molecules into centrosymmetric dimers. Dimers related by translation along the *c* axis form stacks through π–π interactions [centroid–centroid distances = 3.560 (5) and 3.542 (5) Å]. Weak intermolecular C–H···O interactions further consolidate the crystal packing.

References

Plášil J., Škoda R., Dušek M., Fejfarová K., Hloušek J., Sejkora J., Meisser N., Škácha P. and Čejka J. (in prep.): Johannite from different localities: A chemical and X-ray diffraction study. To be submitted to Journal of Geosciences.

Rohlíček J., Cibulka R., Cibulková J., Maixner J. and Hušák M. (2010): 10-Methylisoalloxazine 5-oxide from synchrotron powder diffraction data. Acta Crystallographica E66, o3350–3351.