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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 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:** Structure determination and structure dynamics of novel zeolites and open-framework materials	**Experiment number**: CH-3891
Beamline: ID31	**Date of experiment**: from: Nov. 25th, 2013 to: Nov. 28th, 2013	**Date of report**: March 3, 2014
Shifts: 9	**Local contact(s)**: Christina Drathen	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Xiaodong Zou, Jie Su*, Louise Samain*, Yifeng Yun*, Ilich Ibarra*

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Report:

We measured 12 samples in ID31 for structure refinement and in-situ PXRD for structure dynamics study. Our preliminary results are as follows.

1. PKU-14, a pure germanate zeolite, was solved by in-house PXRD data combined with rotation electron diffraction (RED), IR and NMR techniques. The symmetry was first determined as tetragonal with the space group *I*4/*mcm*. We collected the high resolution powder diffraction data in ID31 for performing Rietveld refinement on the sample to confirm the structure model. As the better resolution for synchrotron data, we notice that the first peak observed in in-house data is split to two peaks in synchrotron data, which indicates the lower symmetry should be applied for PKU-14. Finally, the structure is refined by the monoclinic space group *I*2/*m*, and the location of structure directing agents (SDA) in the pores is also confirmed by HR-PXRD. It is helpful for us to understanding the interaction between the framework and the template. The structure is built by the [$4^6 6^{12}$] cages interconnected with D4R units, showing 3D 12-ring channel system. Now the paper is in manuscript, and we hope that it can be submitted in May.

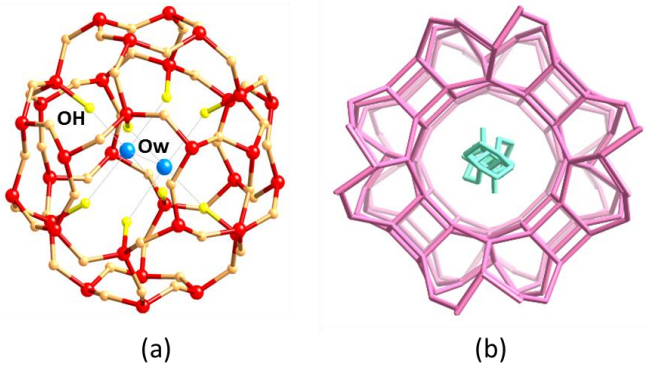

Fig. 1 The structure of PKU-14. (a) Water molecules act as a template in the [$4^6 6^{12}$] cage, and (b) 12-ring channel along the [010] direction with the organic SDA inside.

2. The refinement of the silicogermanate YP-1 by the HR-PXRD data collected in ID31 confirms our structure model from a poor quality single crystal X-ray diffraction data, and the SDAs can be located.

3. The data for EMM-9 collected in ID31 is used for the structure refinement. The missing atoms from our structure model from RED can be found during the refinement and R_{wp} value is about 10%. The final Rietveld refinement result matches the other complementary information very well. Now the paper is in manuscript and will be submitted soon.

4. The data for a novel zeolite EMM-26 is used for structure refinement as well. The resolution can reach about 1.1Å. The missing parts from our structure model can be found. But the peak profile fitting is not so good. It might be related with the morphology of this sample. It is still good enough for us to interpret this structure and also can guide us to improve our synthesis.

5. The structure model of a novel zeolite EMM-25 was initially solved using electron crystallography. Data with much higher peak resolution was collected in ID31 for Rietveld refinement. However, due to the unexpected peak broadening, we are working on improving the Rietveld refinement. Now the paper is in manuscript, and at the mean time we try to improve the synthesis to obtain better samples.

6. The initial model of a germanatesilico zeolite ITQ-54 with extra-large intersecting 20×14×12-ring channels was solved by electron crystallography. The data collected at ID31 was used for Rietveld refinement. Even though that the sample contains impurity, the refinement considering the impurity phase converged with R_p = 0.0510, R_{wp} = 0.0683 and R_{exp} = 0.0258. We would like to locate the organic structure directing agency (OSDA) in the pores so that a better HR-PXRD data from a purer sample is needed. We have now managed to synthesize a purer ITQ-54 sample. We are preparing a manuscript for publication.

7. The i*n-situ* HR-PXRD for CO_2 adsorption of MOFs was applied on NOTT-400 and NOTT-401. Unfortunately, NOTT-401 showed a serious beam damage problem. For NOTT-400, we are still working on the data analysis. With the combination of energy calculation and Rietveld refinement, we hope that we can locate the preferential positions of CO_2 under different pressures.

Publications from the earlier proposal CH3746 at ID31:

8. A. Ken Inge, Henrik Fahlquist, Tom Willhammar, Yining Huang, Lynne B. McCusker and Xiaodong Zou, "Solving Complex Open-Framework Structures from X-ray Powder Diffraction by Direct-Space Methods using Composite Building Units" *J. App. Crystallogr*. **46** (2013) 1094-1104.

9. We also solved a new open-framework germanate SU-77 with 12×12×10-ring channels using the RED data. The structure is built from a novel building unit Ge_8 and was refined using the PXRD data collected in March 2013 at ID31, ESRF. A manuscript is ready to be submitted to *Cryst. Growth Des.*.