

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://www.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.

**Experiment title:**Single crystal studies to unravel the NH₃-SCR deNO_x mechanism in Cu-SSZ-13**Experiment number:**

01-02-1045

Beamline:

BM01A

Date of experiment:

from: 26 Jun 2014 to: 30 Jun 2014

Date of report:

01.03.2016

Shifts:

15

Local contact(s):

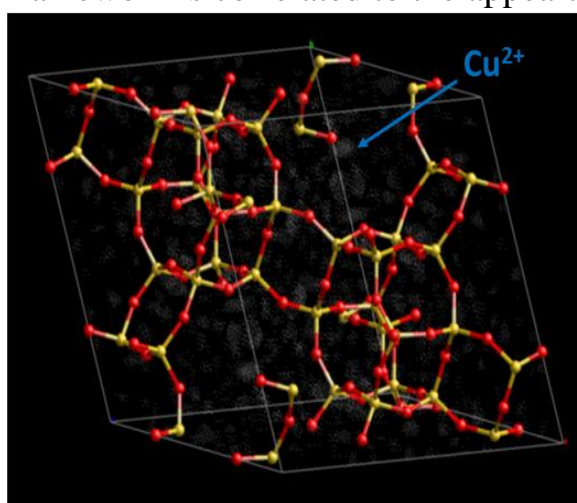
Dmitry Chernyshov

*Received at ESRF:***Names and affiliations of applicants** (* indicates experimentalists):

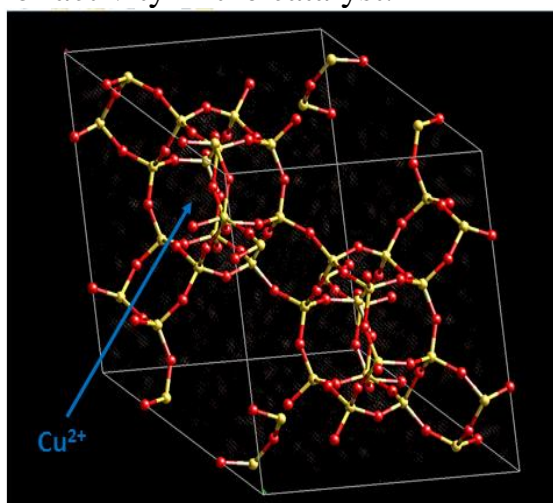
David Wragg, Wojtek Slawinski (University of Oslo, Norway)

Andrew Beale, Ines Lezcano-Gonzalez (Research Complex at Harwell, UK)

Report: The planned single crystal study was not possible, however we performed a highly successful in situ powder diffraction experiment which revealed totally new information on the position of the Cu atoms in the Cu-SSZ-13 catalyst when activated and under working conditions. Migration of the Cu from the 8-ring to the double 6-ring sites in the CHA framework is correlated to the appearance of activity in the catalyst.



(a)



(b)

Figure 1. Difference Fourier map of Cu-SSZ-13-2 sample recorded at 100 °C (a) and 500 °C (b) respectively during activation. Atom colour key (framework): Si⁴⁺/Al³⁺ - Yellow, O²⁻ - Red. White clouds represent regions of electron density. In (a) the strongest electron density cloud is clearly located in the vicinity of the 8r/cha cage which was assigned again to Cu²⁺. In (b) the map is less clear but again there is no obvious contribution in the 8r/cha cage that can be assigned to a

significant portion of Cu^{2+} whereas a significant sphere of electron density does appear in the $6r$ which we again assign to Cu^{2+} .

Two papers have resulted from the experiment:

[1] Lezcano-Gonzalez, I., Wragg, D. S., Slawinski, W. A., Hemelsoet, K., Van Yperen-De Deyne, A., Waroquier, M., Van Speybroeck, V. & Beale, A. M. J. Phys Chem. C 119, 24393-24403 (2015)

[2] Lezcano-Gonzalez, I., Wragg, D. S., Slawinski, W. A., & Beale, A.M. Chem. Commun. (submitted)

