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:** Characterisation of iron precipitates and minerals by Fe K-edge EXAFS spectroscopy and total X-ray scattering	**Experiment number**: 01-01-1009
Beamline: BM31	**Date of experiment**: from: 29.11.2016 to: 5.12.2016	**Date of report**: 7.9.2017
Shifts: 18 (16-bunch)	**Local contact(s)**: Michela Brunelli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Andreas Voegelin*; Ralf Kaegi; Stephan Hug; Thomas Hofstetter; Michael Sander

Ashley Brown*; Jagannath Biswakarma*; Silvan Wick*

Report

For proposal 01-01-1009, we received 6 days of beamtime in 16-bunch mode.

Total X-ray scattering

The first part of the beamtime was used to collect total X-ray scattering data on amorphous Fe(III)-precipitates, namely subnanometric Fe(III)-hyroxyphosphate and Fe(III)-hydroxyarsenates, formed under conditions relevant with respect to Fe, P, and As dynamics in natural groundwater resources and surface waters as well as with respect to water treatment for P or As removal. The analysis of the respective samples was complemented by analyses on reference materials (amorphous and crystalline Fe(III)-phosphate and Fe(III)-arsenate and lepidocrocite).
The scattering data were collected with the MAR detector at 55 keV incident photon energy. Using this setup, we were able to collect interpretable data over a Q-range up to ~22 $Å^{-1}$ for the amorphous materials. Due to the 16-bunch mode and the selected incident photon energy, 2-3 h were required for the collection of the scattering patterns of amorphous materials. Considering the data recorded on the amorphous materials, a lower incident photon energy (with higher flux) could have been used to increase the photon flux without substantial loss in resolution.
The total X-ray scattering data collected on our samples were in line with expectations based on the previous characterization of the same materials by Fe K-edge EXAFS spectroscopy. A manuscript on the structure of amorphous Fe(III)-hydroxyphosphate and Fe(III)-hydroxyarsenate based on the Fe K-edge EXAFS and PDF results is currently in preparation.

Fe K-edge EXAFS spectroscopy

The second part of the beamtime was used for the collection of Fe K-edge EXAFS spectra at room temperature in transmission mode, making use of the sample changer wheel for effective data collection. In total, about 60 EXAFS spectra were recorded on samples from different experiments addressing the redox properties of Fe(III)-oxides and Fe-bearing clay minerals.
A first set of spectra was collected on ferrhydrites, goethites and hematites with varying levels of Al-for-Fe(III)-substitution that will be used in electrochemical reduction experiments. A second set of spectra was collected on samples collected over the course of abiotic (electrochemical) or biotic (microbal) reductive transformation of ferrihydrite into mainly magnetite. A third set of spectra was collected on different Fe-bearing clay minerals collected after microbial Fe(III) reduction. Finally, a fourth set of spectra was collected on lepidocrocites used in experiments on Fe(II)-catalyzed ligand-promoted Fe(III)-oxide dissolution. These EXAFS results contribute to two ongoing PhD studies and one postdoctoral project.