EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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:** Magnetite Precursor Structures in Magnetotactic Bacteria and Aqueous Chemistry	**Experiment number**: **SC-3610**
Beamline: ID26	**Date of experiment**: from: 17/04/2013 to: 22/04/2013	**Date of report**: 27/08/2013
Shifts: 17	**Local contact(s)**: Jean-Daniel CAFUN	*Received at ESRF:*

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Report:

Here we report on our X-ray spectroscopy (XAS) experiments at ID26 on magnetite precursor structures in magnetotactic bacteria and a biomimetic synthetic system (1). These experiments are the continuation of our studies on biogenic magnetite formation in the bacteria (2). In this study we measured quasi-time-resolved XAS of cryo-quenched magnetotactic bacteria and stages of synthetically formed magnetite in solution. We investigated different bacterial mutants and strains known for abnormal mineralization behavior for comparison with wild-type cells investigated in SC-3451 (2, 3).

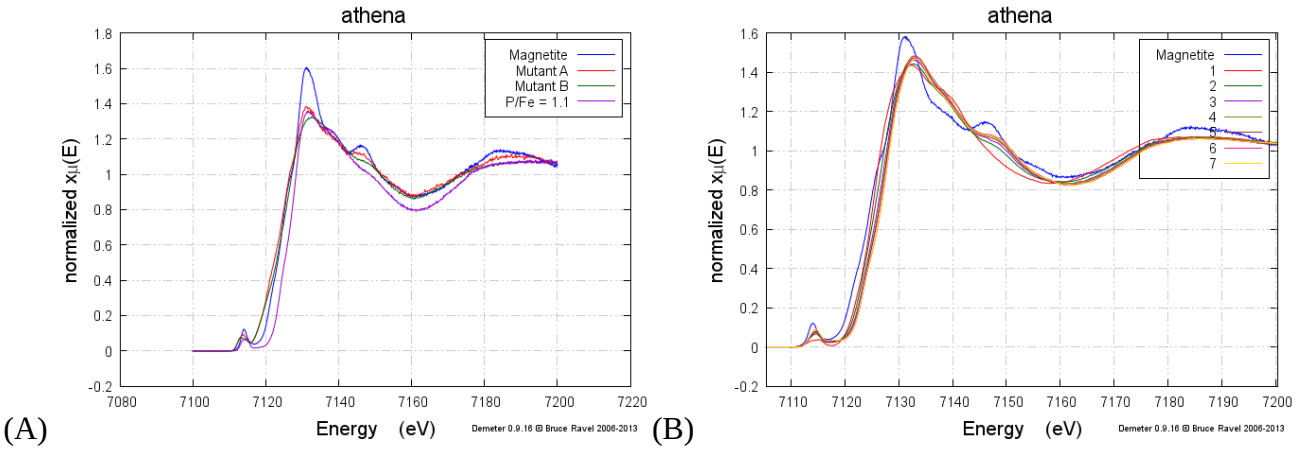
Magnetotactic bacteria were sampled at consecutive time points and centrifuged after induction of magnetite biomineralization. The pellets were re-suspended in 100 µL TBS plus 25 µL glycerol and frozen in liquid nitrogen on sample holders with Kapton film support. Samples were stored at -80°C, sent to ESRF on dry ice where they were stored at -80°C until measurement. In solution, magnetite was formed by alkaline co-precipitation of ferrous and ferric chloride. Fe K-edge X-ray absorption near edge structure (XANES) and extended X-ray absorption fine structure (EXAFS) were recorded in fluorescence mode at beamline ID26. During measurements biological samples were cooled to 10 K using a liquid He cryostat. In solution, samples were measured in a custom-made reaction vessel with Kapton window. XANES spectra and pre-edge spectra were recorded with 0.1 eV resolution from 7100 to 7200 eV and from 7103 to 7122 eV, respectively. EXAFS spectra were recorded up to maximum $k \approx 12$ Å^{-1}. Recorded spectra were averaged using PyMca 4.6.0 after evaluation for photo-reduction and other

artefacts. Averaged XAS spectra were then normalized using Athena 0.8.059. Linear combination fitting of XANES data as well as EXAFS data extraction were performed with the respective functions of Athena.

Figure 1 shows a selection of Fe K-edge XANES spectra of biogenic and synthetic time series samples of data recorded by our team at ID26 in April 2013. We could also obtain good quality EXAFS up to $k \approx 12$ Å^{-1} for bacterial samples and throughout all in situ stages of synthetic samples. XANES of the Fe-induction in mutant cells shows that the Fe concentrations even in maximally iron-depleted cells are sufficiently high to obtain good quality spectra. The investigated bacterial mutants show distinct spectral features as the wild-type bacteria studied earlier (2, 3). In particular, XANES of the mutants evidences characteristic differences to the biogenic system in terms of structure and iron oxidation state (Fig 1A), which will allow us to infer the biochemical role of the affected mutations. Detailed analysis is currently under progress.

The performed in situ experiments for synthetic magnetite (Fig 1B) show the feasibility of our approach to investigate the iron oxyhydroxide cluster structure during formation in solution at ID26. However, the material is prone to oxidation in the employed reaction vessel and therefore the setup needs improvement to exclude such artefacts.

Figure 1. (A) Fe K-edge XANES of magnetotactic bacterial mutants, magnetite and a ferric phosphate. (B) Series of spectra of magnetite formation experiments in solution.



(A) (B)

1. J. Baumgartner, G.Morin, T. Perez Gonzalez, M.Widdrat, D. Faivre, Proposal SC-3610.
2. J. Baumgartner, G. Morin, N. Menguy, T. Perez Gonzalez, M. Widdrat, J. Cosmidis, D. Faivre, PNAS 2013 (early edition), doi: 10.1073/pnas.1307119110.
3. J. Baumgartner, G.Morin, T. Perez Gonzalez, M.Widdrat, D. Faivre, Report SC-3451.