ESRF	**Experiment title:** In-situ monitoring of arsenic retention mechanisms in groundwater upon seasonally driven microbial cycles	**Experiment number**: **30-02- 984**
Beamline:	**Date of experiment**: from: 16/04/2010 at 08:00 to 20/04/2010 at 08:00	**Date of report**: Feb 27, 2011
Shifts:	**Local contact(s)**: Marc Newton	*Received at ESRF:*

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Report:

The aim of the present project was to evaluate the fate of arsenic bound to iron oxides in natural anoxic groundwater by monitoring in situ the molecular environment of arsenic and iron upon seasonal redox cycles. XAS data at both Fe and As K-edges were used to determine the nature of the neoformed Fe-minerals controlling As solubility in such complex mineral assemblages resulting from the microbial incubation of finely divided iron-oxides.

Because of the lack of high troughput fluorescence detector on BM29 the beamtime was dedicated to record high quality EXAFS data on iron and arsenic containing laboratory samples obtained after microbial anaerobic incubation, which are used as proxy for understanding the mechanisms of As immobilization in more diluted natural media.

About 10 samples were analyzed at the Fe K-edge in transmission detection mode (e.g. Figure 1) with Fe concentrations varying within the 1-40 wt% range, including specific model compounds synthesized at IMPMC.
About 8 samples were analyzed at the As K-edge in transmission detection mode (e.g. Figure 2) with As concentrations varying within the 0.1-2.0 wt% range.

Special care was taken for preserving samples under anoxic conditions from the sampling to the analysis. Samples were dried and prepared as pellets under anoxic conditions at IMPMC laboratory. Samples were then brought to ESRF under controlled anoxic conditions and mounted in an anoxic glove box on the cryostat sample holder. After mounting on the cryostat rod within the glove box. The sample rod was then put under low pressure He atmosphere for data analysis in the liquid He cryostat at a temperature or 10-15K. The data were of very good quality, with a usable signal to noise ratio after 2 to 5 scans depending on the Fe concentration.

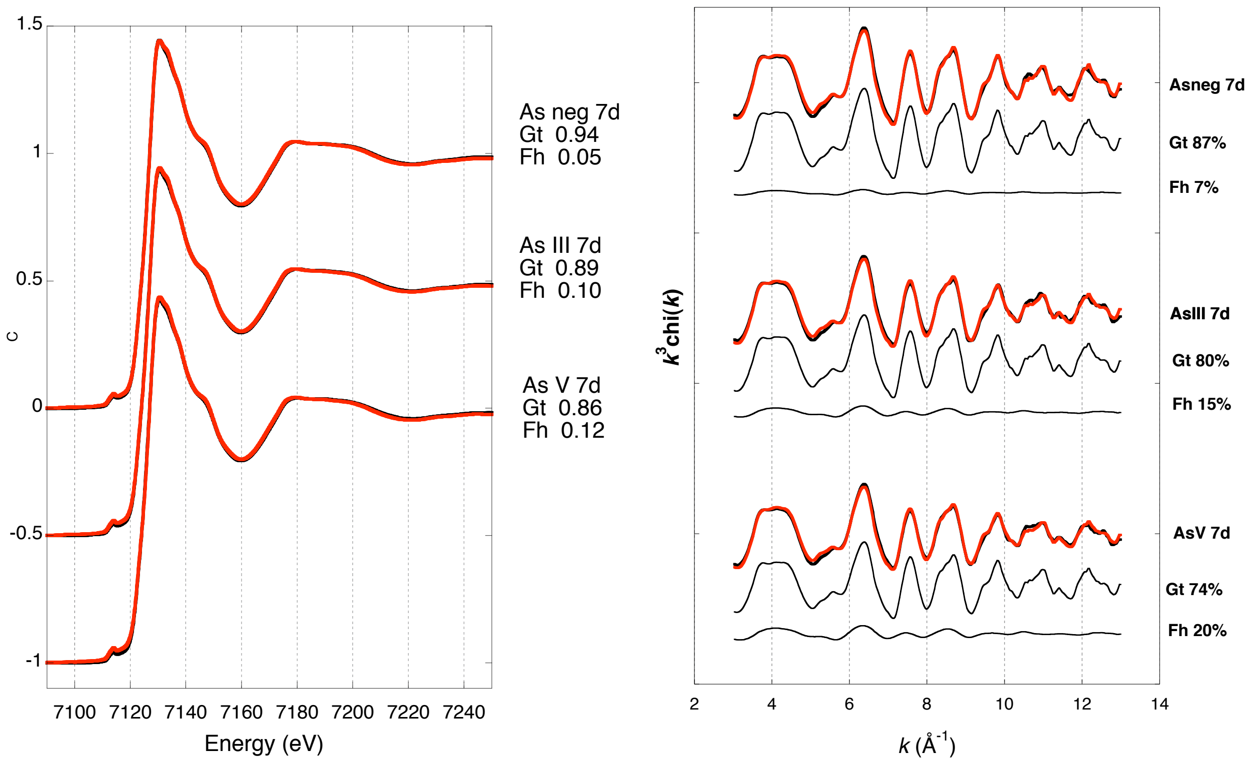

Figure 1. Example of Fe-K edge XANES and EXAFS data recorded in transmission detection mode for some samples studied showing the high signal/noise ratio. Linear combination fitting using appropriate model compounds (fit as red line and components as thin black lines) indicates iron is mainly under the form of goethite (alpha-FeOOH) after incubation of Fe(II) with the anaerobic iron oxidizing bacterial strain studied. The presence of arsenic in the medium tend to favor of the formation of poorly ordered iron oxyhydroxide ferrihydrite.

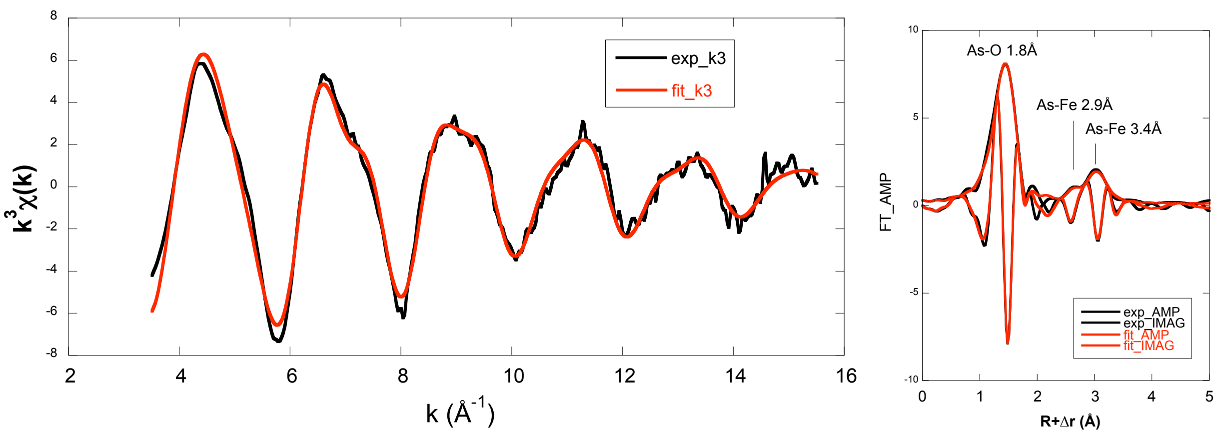

Figure 2. Example of As-K edge EXAFS data recorded in transmission detection mode, here for As(III) incubated in the presence of Fe(II) and an anaerobic oxidizing bacterial strain. Shell-by-shell analysis indicate As(III) inner-sphere surface sorption complexes at the surface of the iron oxyhydroxide, that explain As(III) immobilization in these anaerobic media. As concentration is 2 wt% in the sample studied.

Data analysis is still under progress, a publication is already in preparation.