Standard Project

Experimental Report template

Proposal title: Uranium speciation in contaminated lake sediments (France)		**Proposal number:** 20140310
Beamline: FAME	**Date(s) of experiment:** from: 27/01/2015 to: 03/02/2015	**Date of report:** 13/02/2015 15/02/2016
Shifts: 18	**Local contact(s):** Olivier Proux	*Date of submission:*

Objective & expected results (less than 10 lines):

The primary objective of this work was to determine uranium speciation in bottom sediment samples from an artificial lake located 20 km downstream of a former U-mining site in the Massif Central, France. These information are expected to serve as basis for modeling early diagenesis of U in these sediments and to evaluate the long term stability of U scavenging processes. A challenging aspect of this work was to probe U speciation in samples containing very low U concentration (< 100 ppm) in complex sediment matrix with high fluorescence background.

Results and the conclusions of the study (main part):

The preliminary analyses performed on the U-LIII-XANES allow us to observe the evolution of U speciation, and to measure the proportion of U(IV) and U(VI) in the sediment samples (Figure 1).

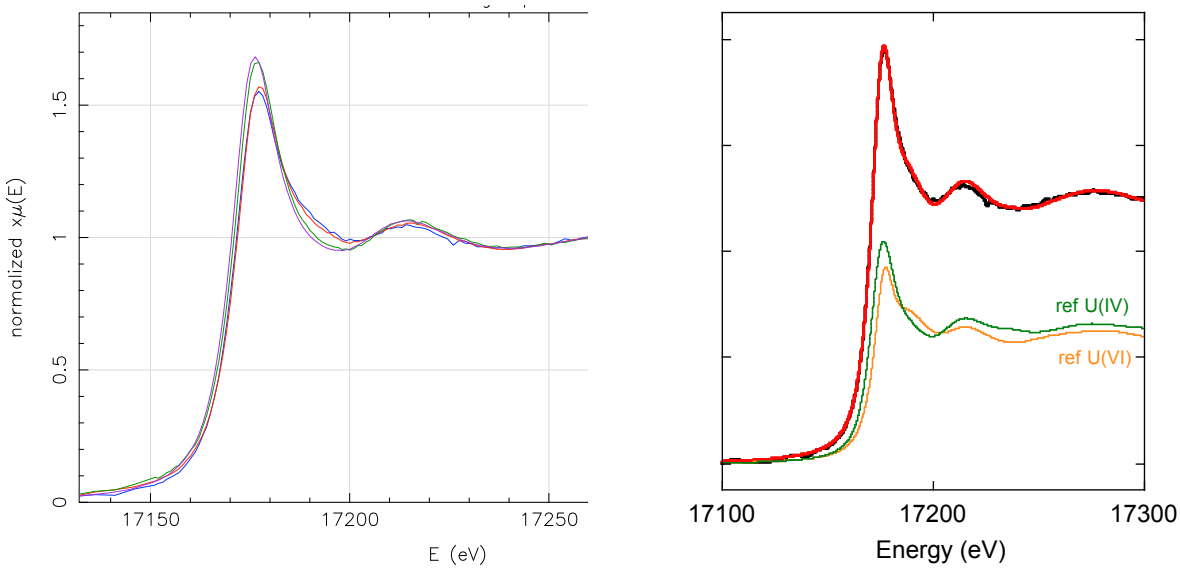

Figure 1: *XANES spectra of sediment samples with U<100ppm (left) and linear combination fit using XANES pectra from U(IV) and U(VI) reference compounds (right). The fit is plotted in red and its components are shown below (in black). Results of the linear combination fit indicate 55 ± 5 % of U(IV) and 45 ± 5 % of U(VI).*

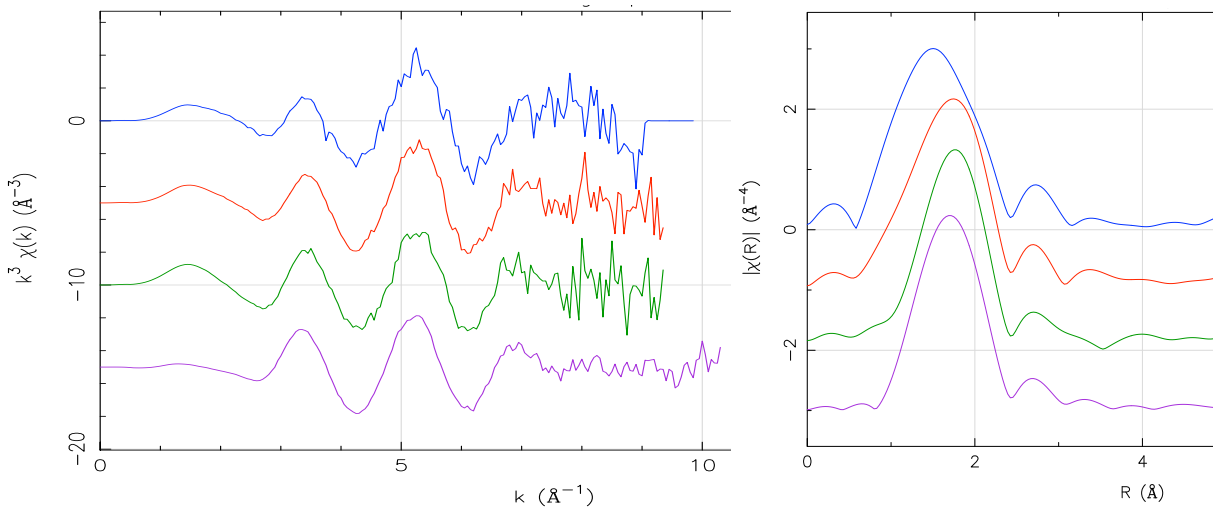

Figure 2: *EXAFS spectra with their FFT of sediment samples with U of 50, 70, 90 and 250 ppm recorded at 20K. A minimum number of 12 EXAFS scans of 40 mn were merged for each sample.*

EXAFS analysis of these sediment samples reveal changes in the U bearing phases. No evidence for uraninite was found, suggesting the presence of other U(IV) bearing phases. Several reference samples were also analysed and will help determining U speciation in theses samples, in combination with microscopy observations. In addition EXAFS data were interpreted using wavelet analysis. In combination with electron microscopy, these data have allowed us to elucidate uranium speciation at trace level (≤ 300 ppm) in these lake sediments.

Justification and comments about the use of beam time (5 lines max.):

The 18 shifts of beamtime were dedicated to XANES and EXAFS measurements at the U LIII-edge. Data were recorded in fluorescence detection mode using the Ge 30 elts on FAME. The high flux of the beamline, allowed to record XAFS data on very diluted samples (down to 50 ppm with high fluorescence background from Rb and Sr) up to k of 9 $Å^{-1}$. XANES data were recorded on 40 samples along 5 sediments cores, requiring 2-4 scans or 20 mn per samples. EXAFS data requiring about 8-10 h per sample were recorded on 6 samples.

Publication :

G. Morin, A. Mangeret, G. Othmane, L. Stetten, M. Seder-Colomina, J. Brest, G. Ona-Nguema, S. Bassot, C. Courbet, J. Guillevic, A. Thouvenot, O. Mathon, O. Proux, J.R. Bargar (2016) Mononuclear U(IV) complexes and ningyoite as major uranium species in lake sediments. ***Geochemical Perspectives Letters*** 2, 95-105.