ESRF	**Experiment title:** Uranium speciation in mine tailings	**Experiment number**: EC-46
Beamline: ID-21	**Date of experiment**: from: 23.06. to: 27.06.2006	**Date of report**: 29.02.2008
Shifts: 12	**Local contact(s)**: Jean Susini	*Received at ESRF:*
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Report:

Forty years of uranium mining in Eastern German have left a legacy of uranium-contaminated pits, waste piles, mine tailings and surrounding soils. Little is known on the redox processes going on in these sites, which are highly relevant to predict the risk for a contamination of adjacent ground and surface water bodies. In order to shed some light on the redox processes in the former uranium mine tailing at Freital, Saxony, we have investigated two samples with micro-XRF and micro-XRD (Scheinost et al., 2005). To further investigate the uranium redox states and their relation to other redox-sensitiv elements, which may be related to uranium redox, we have measured bulk XANES spectra at the U-L_{III}, Fe-K and As-K edges at BM20. The spectral contributions of oxidized and reduced elemental species were quantified by Iterative Transformation Factor Analysis (Rossberg et al., 2003) (Fig. 3).

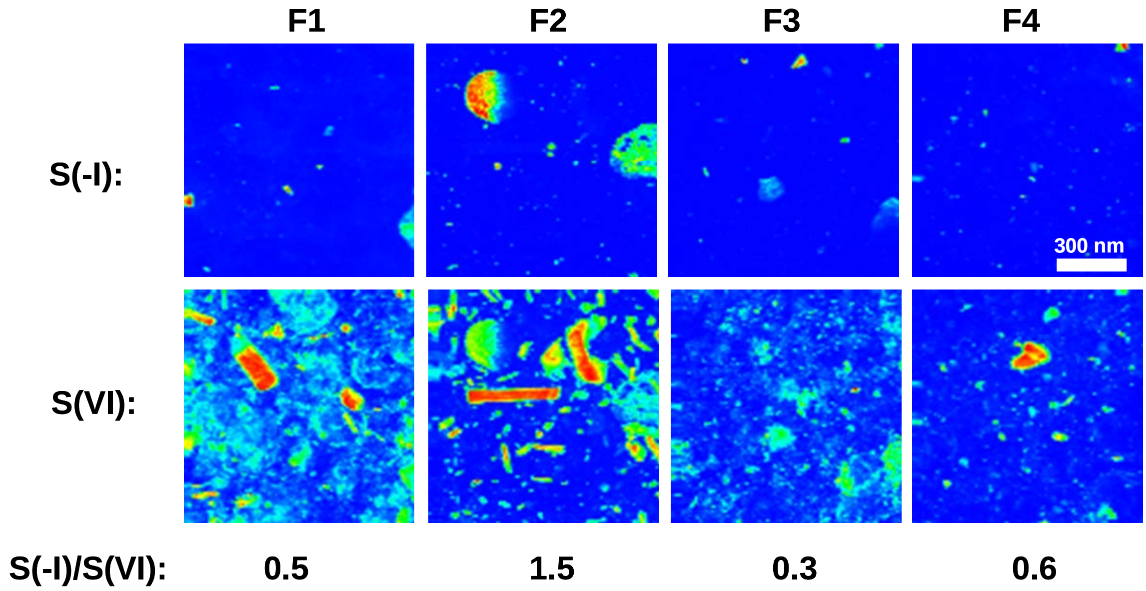

Fig. 1. Sulfur oxidation state mapping of samples F1 to F4 performed by STXM at S- K edge.

Another relevant redox element is sulfur, which we investigated during this project using the STXM facility of ID-21. Figure 1 shows maps collected at two different energies representing S^{-I} and S^{VI} oxidation states. All samples show contributions from both oxidation states, samples F1 and F2 also suggest sulfate-type minerals with lath-shaped crystals. Sulfur-K XANES spectra were measured at selected spots (Fig. 2 a). These spectra show two components, identified as gypsum and pyrite (Fig. 2b). Using physical mixtures of these two minerals, we could established linear relationships between spectral intensities at selected energies and their relative abundance (Fig. 2 c,d). Figure 3 shows the establised S redox in relation to that of Fe, As and U. F1 and F2 show a strong correlation of the redox states of all elements, while correlations for F3 and

F4 are disturbed. Further invstigations are underway to identify the relevant species using micro-EXAFS spectroscopy.

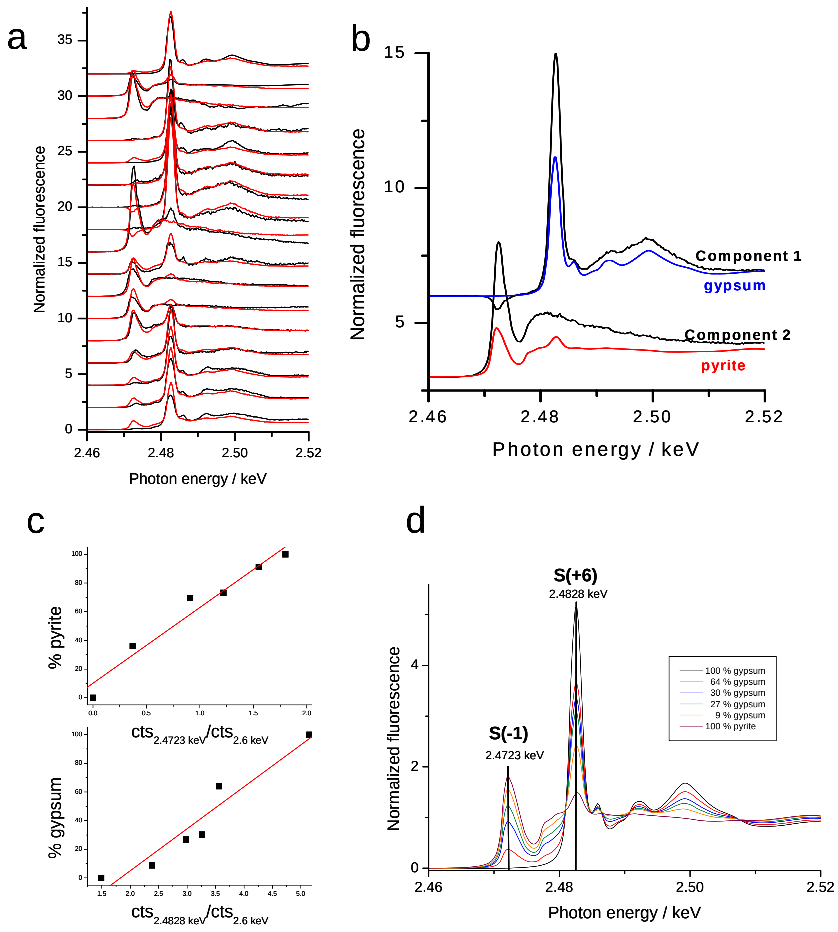

Fig. 2.
Identification and quantification of mineral phases by S-K micro-XANES. a) experimental spectra at selected spots (black lines) and their reconstruction by factor analysis using two components (red lines). b) Extracted statistical components 1 and 2 are similar to gypsum and pyrite, respectively. c) Quantification of pyrite and gypsum based on the intensity ratios at the given energies. d) Spectra of physical pyrite/gypsum mixtures used to establish the relationship in c).

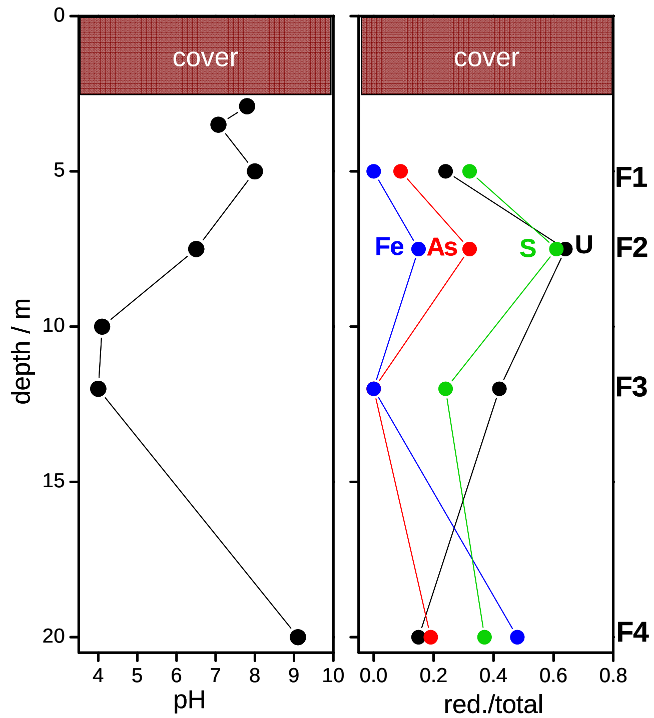

Fig. 3.
Depth profile of the tailings pond at Freital, Saxony, showing pH and ratios of reduced/oxidized elements Fe, As, S and U. Labels F1 to F4 give the position of the samples. Ratios of reduced and oxidized species have been established for Fe (Fe^{II}/Fe^{III}) by Fe-K XANES, for As (As^{III}/As^{V}) by As-K XANES, and U (U^{IV}/U^{VI}) by U-L_{III} XANES.

Rossberg, A., Reich, T., Bernhard, G., 2003. Complexation of uranium(VI) with protocatechuic acid - application of iterative transformation factor analysis to EXAFS spectroscopy. Anal. Bioanal. Chem., 376: 631-638.

Scheinost, A.C., Hennig, C., Somogyi, A., Martinez-Criado, G., Knappik, R., 2005. Uranium speciation in two Freital mine tailing samples: EXAFS, micro-XRD, and micro-XRF results. In: B.J. Merkel, A. Hasche-Berger (Editors), Uranium in the Environment: Mining Impact and Consequences. Springer Verlag, Berlin, pp. 117-126.