



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 using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now 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.

**Experiment title:**

Fe spectromicroscopy on oxic and anoxic soil and lake sediment samples

Experiment**number:**

ME-1093

Beamline: ID 21	Date of experiment: From: 6.7.2005 to: 11.7.2005	Date of report: 31.8.2005
Shifts: 16	Local contact(s): Diane Eichert	<i>Received at ESRF:</i>

Names and affiliations of applicants (* indicates experimentalists):***Jörg Prietzel, Lehrstuhl für Bodenkunde, Technische Universitaet Muenchen****Jürgen Thieme, Institut für Röntgenphysik, Universitaet Goettingen*****Karin Eusterhues, Lehrstuhl für Bodenkunde, Technische Universitaet Muenchen****Report:**

This experiment was based on the results of an earlier experiment (ME-944) which had documented the principal capability of Fe-K edge XANES to quantify the Fe(II)/Fe(III) ratio and the contribution of different Fe-bearing phases to mineral mixtures and soil samples. Now, we applied the prepeak analysis to (i) a soil toposequence from a forested watershed in the Fichtelgebirge/Germany constituting a hydrological gradient, to (ii) a dissected aggregate from a Eutric Gleysol near Freising/Germany, and (iii) to annually-laminated lake sediments.

For organic and mineral horizons of the investigated soil toposequence, an increasing groundwater influence was clearly reflected in an increased contribution of Fe(II) species to the total iron content of the respective soil samples (Figure 1). Similarly, the concentration of Fe(II) increased from the most oxic soil (Dystric Cambisol) to the most anoxic soil (Rheic Histosol) and the contribution of the Fe-oxyhydroxides, as calculated by LCF, decreased. Iron oxides are an important soil component in terms of stabilizing soil aggregates and reducing erosion losses. In different soils with different water regimes and/or different physico-chemical conditions, dissolved Fe species can be transported both between and inside aggregates and root channels. Solution or precipitation processes according to changing redox conditions may take place at the rims or in the centre of soil aggregates. Several focussed XANES spectra on a profile across the dissected aggregate uncover the differences in the contribution of ferrous and ferric iron compounds, depending on the specific location and the total Fe content of the investigated spot (Figure 2). Moreover, X-ray microscopy revealed that in the studied aggregate the distribution of Mn minerals is uncoupled from the distribution of Fe oxides. These results underline how much can still be learned using the unique ability of XANES to analyse chemistry as well as mineralogy on a micrometer scale. Another advantage of this technique is its applicability to poorly crystalline minerals, which often dominate Fe phases in soils. We expect that by

examining the microscale complexity and inhomogeneity of Fe minerals in different soils XANES can greatly contribute to a better understanding of the cycling of Fe and related nutrient elements (e.g., S, P).

The capability of Fe-XANES to distinguish between sulfidic and oxidic Fe minerals was also applied to samples of an annually-laminated lake sediment. The undisturbed sediments were cut perpendicular to the lamination, so that the sedimentation of certain years was exposed at the surface. Spatially resolved Fe-XANES show pyrites in close vicinity with Fe-oxides, i.e. within a distance of a few microns. Non-sulfidic phases are sometimes enriched in Mn, while in other sediment sections P-rich Fe minerals (Fe-phosphates?) occur. Since the pyrites show no signs of oxidation, we must assume a simultaneous deposition of sulfides and oxides, i.e. these minerals must have formed at different redox conditions at different waterdepths.

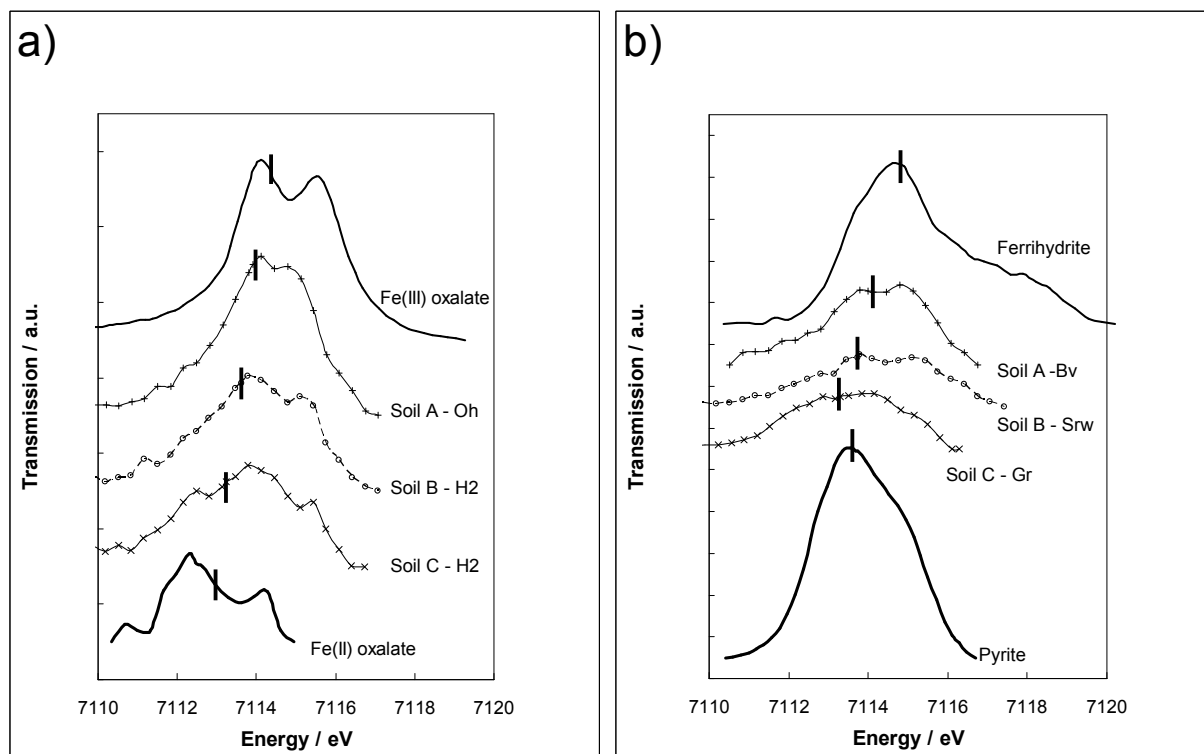


Figure 1: Pre-edge peak spectra for (a) organic topsoil horizons and, (b) mineral subsoil horizons of three soils in the Fichtelgebirge. The groundwater influence increases from soil A to soil C. Pre-edge peak centroid energies are indicated with a solid bar.

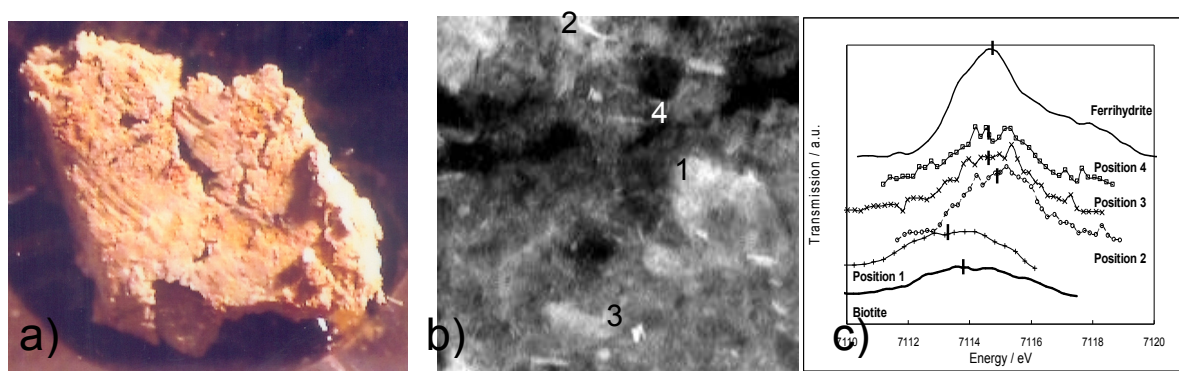


Figure 2: (a) Aggregate taken from a Gleysol, (b) close-up view taken with an optical microscope, (c) Fe-XANES pre-edge peak spectra taken at positions 1 to 4, representing mottles (positions 1,2), the “average” aggregate interior (position 3), and the edge of the root channel (position 4). Pre-edge peak centroid energies are indicated with a solid bar.