ESRF	**Experiment title:** Local environment of lead and zinc in smelter-impacted soils	**Experiment number**: CH-531
Beamline: ID 26	**Date of experiment**: from: 20 / 11 / 98 to: 24 / 11 / 98	**Date of report**: 27 / 02 / 99
Shifts: 12	**Local contact(s)**: Christophe GAUTHIER	*Received at ESRF:*

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Report:

EXAFS and XANES data at the As K-edge (11.866 keV) and Zn K-edge (9.659 keV) were recorded at RT and 20K in fluorescence mode using Si-photodiodes and appropriate Ge and Cu filters. Pb L edge was not investigated because of the Pt coating of the harmonic rejection mirrors. The very good quality of the As K-edge EXAFS spectra recorded in 2 to 6 step-scans attests for the excellent capabilities of ID 26 for studying complex and highly diluted natural samples.

1. Natural Zn- and As-bearing amorphous hydrous ferric oxides.

For the first time, Zn and As EXAFS data were recorded on natural 2-lines ferrihydrites (Fig. 1), which are dominant scavengers for these elements in mine-drainage systems.

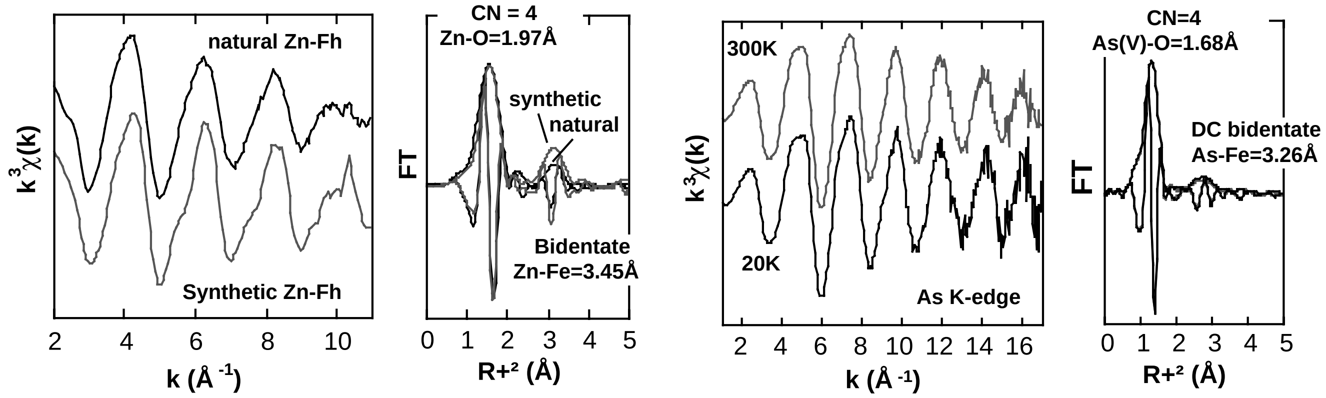

Figure 1. RT Zn-K and As-K edges EXAFS data from natural 2-lines ferrihydrites containing1wt. % Zn or As and originating from mine-drainage environments. Data from the natural Zn-ferrihydrite are compared with those from a synthetic analogue recorded at SSRL. Zn and As are both mainly sorbed as double corner bidentate inner sphere complexes.

2. Zn-contaminated soil.

Zinc speciation was investigated in a sewage sludge contaminated agricultural soil (500 - 200 mg/kg Zn in the bulk topsoil (A) and 30-60 cm horizon(B), resp.). In the B-horizon, Zn(II) is associated to kaolinite, likely as an Al rich Zn-Al layered precipitate (Zn-O = 2.03 Å; CN=6; Zn-Al = 3.06 Å, CN=3) which nature is not yet understood. In the topsoil, the Zn-kaolinite component is still present (Fig. 2b) but Zn(II) is mainly in 4-fold coordination (Zn-O = 1.96; CN=4) in relation with the possible presence of organic Zn-phosphate complexes. Additionnal EXAFS data on chemically treated samples would help to elucidate Zn speciation in the topsoil.

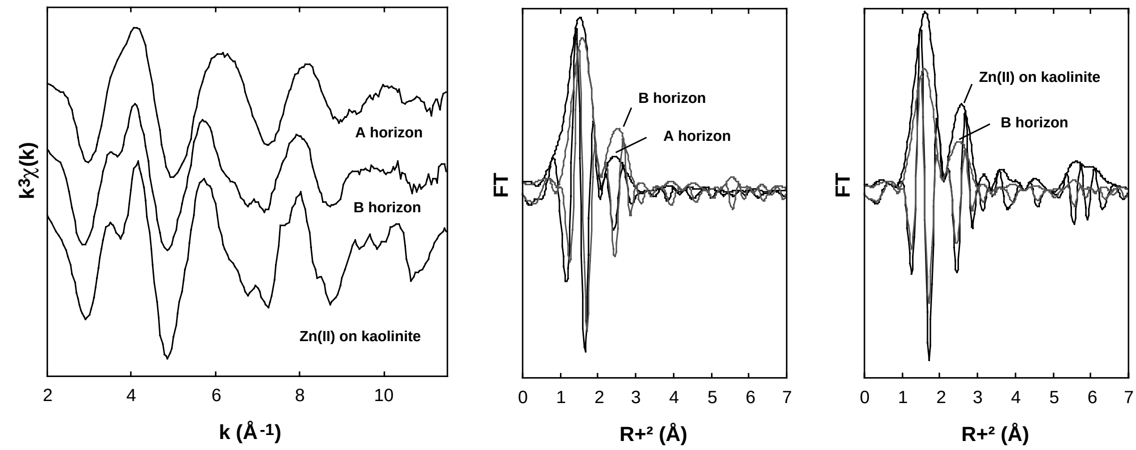

Figure 2. RT Zn K-edge EXAFS data from the <2 µm fractions of the A- and B- soil horizons (1500 and 500 mg/kg Zn, respectively) compared with data from Zn(II) sorbed on kaolinite (1000 mg/kg Zn). This latter spectrum was recorded at SSRL.

3. Soil developed on an As geochemical anomaly.

The study of arsenic speciation along a soil profile developed upon an arsenopyrite mineralized schist (1000 - 5000 mg/kg As) allowed to identify a weathering phases able to immobilize As at geological time-scale. Arsenic was found to accumulate, in As(V) form, at depth 60-90 cm, mainly as pharmacosiderite, K Fe (AsO) (OH) .7H O and not as the common iron-arsenate, scorodite, FeAsO .2H O. The remaining As(V) is likely sorbed onto ferrihydrite as inner sphere complexes. In the topsoil, this latter component dominates As speciation..

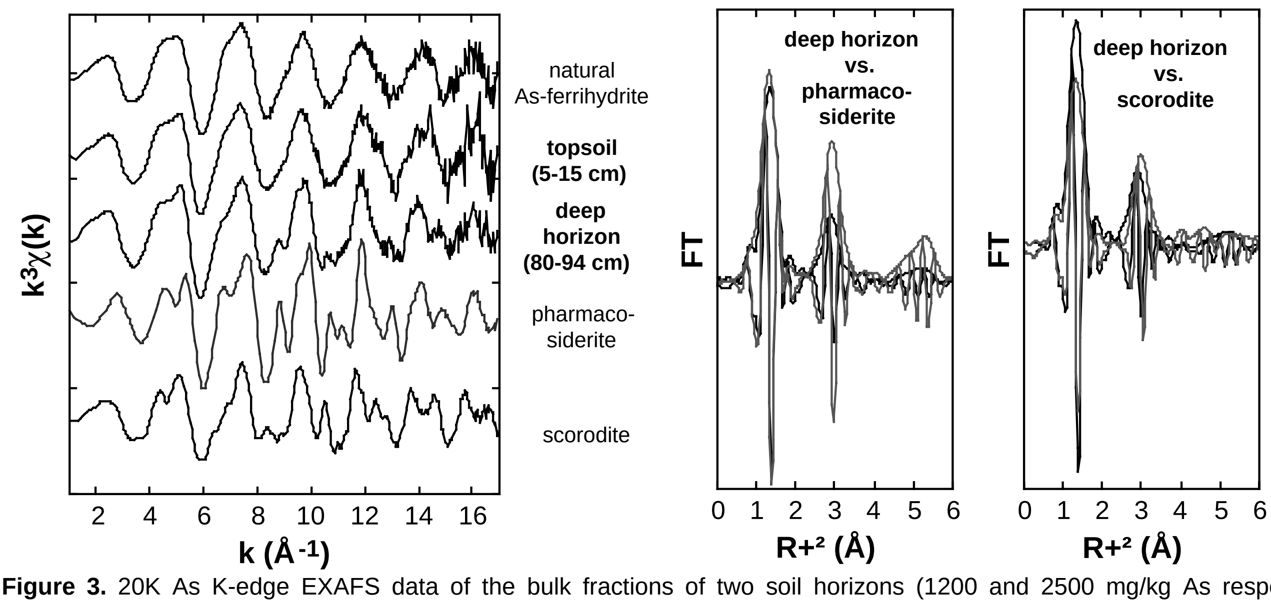

Figure 3. 20K As K-edge EXAFS data of the bulk fractions of two soil horizons (1200 and 2500 mg/kg As respectively) compared with model compounds.

Communication : LECOQ D., MORIN G. JUILLOT F., ILDEFONSE Ph., CALAS G., GAUTHIER Ch. (1999) Arsenic speciation in buried wastes at an industrial site and in a soil developed on a mineralized schist. 10th European Union of Geosciences (EUG10), 28th march - 1st April, Strasbourg.

Important remark:

Ring orbit instabilities (1/3 filling instead of 2/3) and frequent shutdowns shortened the usable beamtime (see the following mail from the Machine Division Director). As a consequence much time was spent in beam alignment. Misalignements limited many EXAFS spectra to a short k-range (<12) (see Zn data).

tX-Authentication-Warning: leo.esrf.fr: pcgauthier.esrf.fr [160.103.206.16] didn't use HELO protocol
X-Sender: gauthier@leo.esrf.fr
Date: Tue, 01 Dec 1998 10:07:12
To: Guillaume Morin <Guillaume.Morin@lmcp.jussieu.fr>
From: Jean-Marc Filhol <filhol@esrf.fr> (by way of Gauthier christophe <gauthier@esrf.fr>)
Subject: Change in closed orbit after the las MDT
Mime-Version: 1.0
X-MIME-Autoconverted: from quoted-printable to 8bit by ardoise.lmcp.jussieu.fr id KAA35866
Status:

Dear Users,

One week ago, we experienced several problems with the electron Beam Position Monitors (BPMs). Some of you have been affected by this situation. Since then, all of them were put back in good working conditions.

During the Machine Dedicated Time (MDT) of Tuesday 24th November and following the BPMs repair, a new orbit was defined and left for the USM which started Wednesday 25th November. The RMS orbit parameters were indeed much better than before the MDT.

However and following a first complain of ID22 this Wednesday evening , we found that for some of you, this has resulted in non negligible change in the angle of the beam (in one case, the difference of angle before and after MDT is even reaching about 75 µrad in vertical plane, i.e. about 2 mm at the location of the
sample !). You will find hereafter the table of change in angle for each straight section.

Our proposal is to stay like it is now until next tuesday MDT (Dec 1st), and to go back (or at least much closer) to what it was before the 24 November after the 1st of December.

If some of you are being affected by this situation still now, please, let us now as soon as possible (send an e-mail to 'position@esrf.fr'). Please give us some feedback even if you didn't suffer from that unexpected event.

Difference	V angle(micro-rad)	H angle(micro-rad)
ID 1	-11.3	13.8
... etc...		
ID26	-64.8	-33.3
... etc...		
ID32	-23.8	-4.7

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