

<p>SUL-X</p> <p>X-ray beamline of the Synchrotron Radiation Laboratory for Environmental Studies (SUL)</p> <p>Technology: Bulk and microfocus (down to about 40 µm x 30 µm beamsize at sample position):</p> <ul style="list-style-type: none"> ▪ X-ray fluorescence spectroscopy (tunable excitation energy 2.2 - 20 keV) ▪ X-ray absorption spectroscopy (2.14 - 20 keV; P K-edge - Rh K-edge, U L-edge) ▪ X-ray diffraction <p>Equipment: 4 circle diffractometer, Vacuum chamber (1x10⁻⁵ mbar), Detectors: 3 Ionization chambers, 7 element Si(Li) solid state detector, 1 element SDD detector, CCD detector, Photodiodes</p>	<p>Category: C. Particle Characterization in- and ex-situ</p> <p>Institute: Karlsruhe Institute of Technology (KIT)</p> <p>Location: Karlsruhe Institute of Technology Institute for Synchrotron Radiation (ISS) Building 348 Hermann-v.-Helmholtz-Platz 1 D-76344 Eggenstein-Leopoldshafen Germany</p> <p>Contact Details of Technology Experts:</p> <p>Names: Dr. Ralph Steininger, Dr. Jörg Göttlicher</p> <p>Phone: +49 721 608-26173 +49 721 608-26070</p> <p>Fax: +49 721 608-26789</p> <p>E-mail: ralph.steininger@kit.edu; joerg.goettlicher@kit.edu</p>
<p>Short technology description/Overview (<i>approx 300 words</i>):</p> <p>The SUL-X beamline enables to investigate samples - without remounting them - sequentially with X-ray fluorescence spectroscopy (XRF) to image elemental distributions, X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD) to determine chemical states and mineral phases at µm-scale resolution.</p> <p>The wide energy range will allow XAS for elements between P and U. XAS provides essential information about the local atomic geometry (EXAFS) and of the valence/chemical state of the absorbing atom (XANES). It can be applied equally to investigate both ordered (crystalline) and disordered (amorphous, liquid) materials. Dilute species (down to the ppm range) can be measured in the fluorescence mode of XAS.</p> <p>X-ray diffraction measurements (e.g., of powder, of aggregates of crystals, nanoparticles or laterally resolved on sample sections) enable the identification of mineral phases and allow to find correlations between them and pollutants.</p> <p>Adjustable beam dimensions in combination with automated sample-positioning and a precise diffractometer will allow investigation of elemental distribution, chemical states of elements and their associations to mineral phases down to the µm scale, essential key parameters for environmental and health risk assessment.</p>	
<p>Main Features (Equipment Capabilities):</p>	
<ul style="list-style-type: none"> ▪ Source: Wiggler (27 pole each 74 mm) ▪ Double crystal monochromator Si(111) and Si(311) crystal pairs; energy resolution ($\Delta E/E$) about 2x10⁻⁴ Si(111) 1x10⁻⁴ (Si311) ▪ two step focusing principle; beam size at sample position from collimated about 0.8 mm x 0.8 mm to focused 40 µm(hor.) x 30 µm(vert.) 	<ul style="list-style-type: none"> ▪ Detectors: 3 ionizations chambers (for XAS and 1st as I₀ monitor); 7 element Si(Li) solid state and 1 element SDD detector (XRF, XAS in fluorescence mode); energy resolution about 150 eV at optimum peaking times; CCD detector with 80 mm(hor) and 120 mm (vert) active area (XRD); optical microscope;

- Sample stage:
Diffractometer with two horizontal goniometers (omega, phi), a cradle (chi) and x,y,z stages
- Vacuum chamber (for low energies and oxidation sensitive samples): about 1×10^{-5} mbar

all vacuum compatible

- Sample mounting:
Various holders for six pellets, sections (up to about 70 mm (hor) x 30 mm (vert)).
Other sizes and mountings of irregular shaped samples can be realized on request

Limitations / constrains

- depending on the chemical composition and the degree of focusing, samples might be sensitive to X-ray radiation.
- no single nano-particles can be analyzed, but the micrometer beam size at sample position enables e.g. the characterization of aggregates of nano-particles and the detection and distribution of nano-particles e. g. in tissue.

Typical structures & designs

(1) Chemical speciation of Pb in tidemarks of human articular cartilage (μ -XRF, μ -XAS)

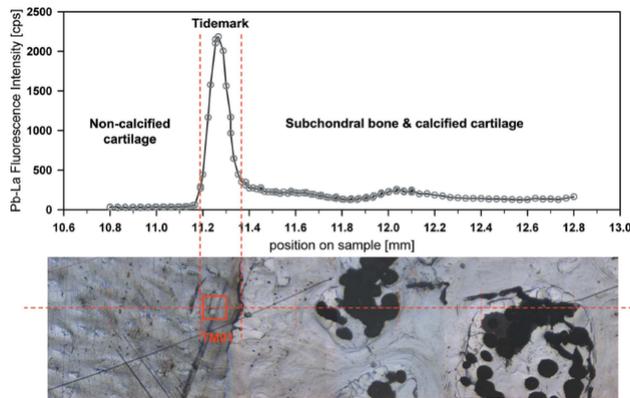


Fig. 1a: Pb L_3 fluorescence intensities of a line scan across the tidemark and reflected light image.
Red box: position for XANES scans, spot size, about $150 \times 100 \mu\text{m}^2$.

(2) Speciation of copper in dendrites of enriched agricultural lime (μ -XRF, μ -XAS)

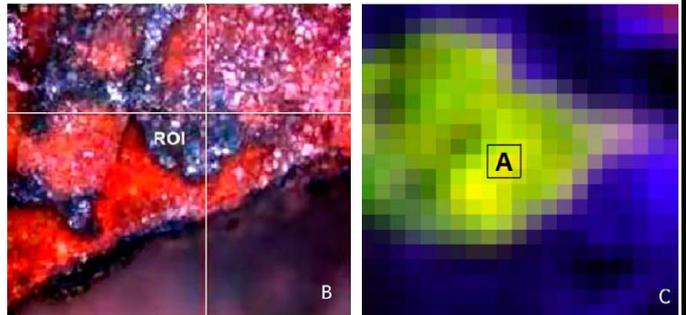
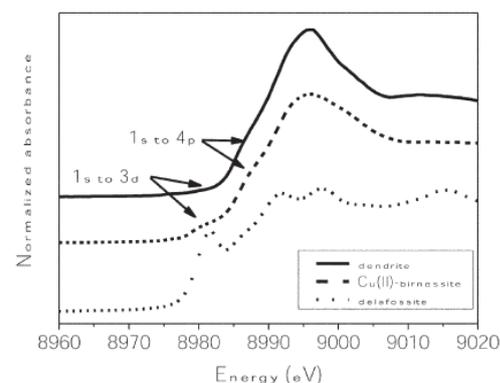
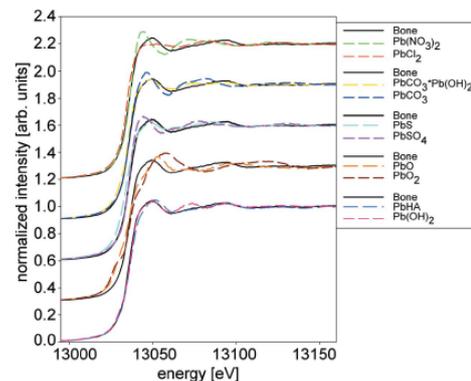


Fig. 2a, b:
(a) Optical image with red Fe oxide, black Mn oxide dendrites; and
(b) multicolor μ -XRF element distribution map (Cu, Mn, Fe) of a dendrite area $30 \mu\text{m}^2$ pixel size (map area $600 \times 600 \mu\text{m}^2$). Combination red (Cu) and green (Mn) is yellow, red (Cu) and Fe (blue) is green. Hotspot A $70 \times 70 \mu\text{m}^2$ for μ -XAS



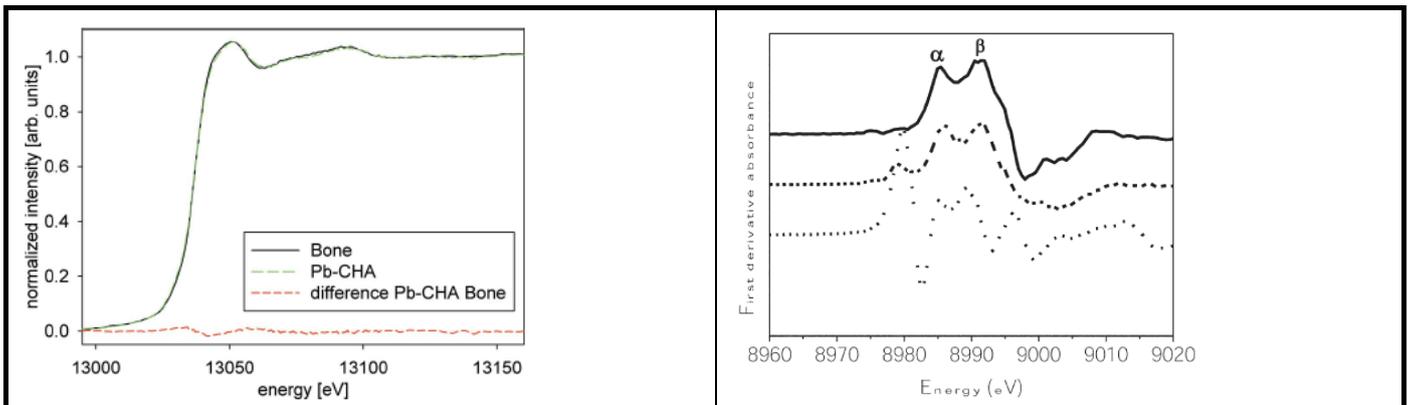


Fig. 1b, c: Merged Pb L_3 -edge XANES spectra of tidemark and bone (Bone) compared to (b) all used reference substances and to (c) the best matching spectra of synthetic carbonated hydroxyapatite.

(Meirer F., et al. (2011) *J. Synchrotron Rad.*, 18, 238-244)

Fig. 2c, d: Cu K-edge μ -XANES spectra (c), with (d) their first derivatives, from the sample and the Cu(I)- or Cu(II)-references. The α/β splitting of the main-edge peak is characteristic of the anisotropic symmetry of Cu(II) compounds due to the Jahn–Teller distortion effect.

(Kersten M., et al. (2011) *Soil Sci. Soc. Am. J.*, 75, 509-520)

Any further Information; concerning the possibilities of characterizing nanoparticles at the SUL-X beamline:

When nanoparticles are exposed to the biosphere it is important to know where and in what chemical form they are deposited in our environment, especially in life tissues. Both questions can be addressed by combining microfocused X-ray techniques as they are offered by the X-ray beamline for environmental studies (SUL-X*) of the Institute for Synchrotron Radiation (ISS) at the synchrotron radiation source ANKA. As a core technique for determining the chemical form (e.g., valence, coordination, bond distances between adjacent atoms) X-ray absorption spectroscopy will be applied. The following scenario of characterizing nanoparticles with a combination of X-ray techniques is suggested:

- (1) The location of indicator elements of nanoparticles (e.g. Ti for TiO_2 , Zn for ZnO, Ag, Fe for silver and iron nanoparticles) by measuring their distribution, for example, in tissues that were exposed to nanoparticles using X-ray fluorescence spectroscopy.
- (2) In a second step the chemical form of the indicator elements will be investigated at sample positions where nanoparticles have been localized using X-ray absorptions spectroscopy.
- (3) If crystalline phases are involved additionally the relationship between nanoparticles and mineral phases hosted in the sample material can be determined with X-ray diffraction.

Hence, SUL-X may contribute significantly in characterizing nanoparticles in-situ and ex-situ for biological and environmental impacts assessment (Category C in "Current list of NA and JRA Work Packages", see attachment).

* SUL = Synchrotron Umwelt-Labor