



	<b>Experiment title:</b> Characterization of chemical reactions between nitrogen compounds and recycling phosphorus-fertilizers in soils from agricultural pot experiments	<b>Experiment number:</b> EV-284
<b>Beamline:</b> ID26	<b>Date of experiment:</b> from: 20.09.2017 to: 26.09.2017	<b>Date of report:</b> 20.02.2018
<b>Shifts:</b> 18	<b>Local contact(s):</b> Ana Elena Pradas del Real	<i>Received at ESRF:</i>

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**Report:**

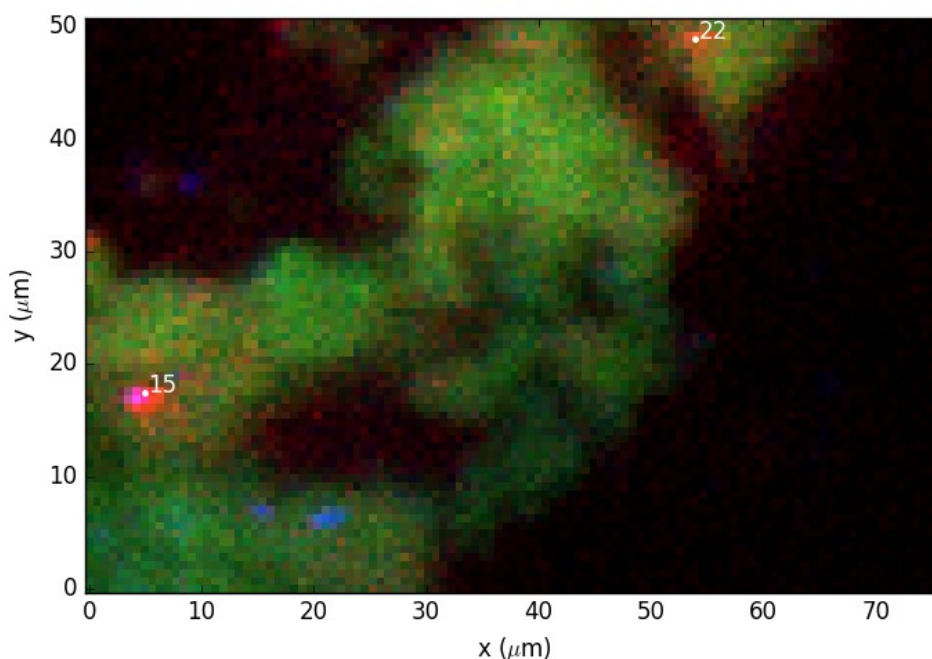
We analyzed the speciation of phosphorus (P) in fertilized soils from a pot experiment with a combination of micro-FTIR (Fourier transform infrared) and micro-XANES (X-ray absorption near edge structure) spectroscopy.

Agricultural pot experiments are often performed to analyze the plant-availability of nutrients. However, these experiments do not determine the compounds which are responsible for the yield performance. The aim of this project was to gain a thorough understanding of the chemistry controlling P availability by combining two in situ spectroscopic techniques ( $\mu$ -FT-IR and P K-edge  $\mu$ -XANES) to determine inorganic and organic P and N phases in soils from pot experiments.

Therefore, the soil samples from pot experiments were compressed in a diamond compression cell to get a thin layer (approx. 10  $\mu$ m) of the soil which can be analyzed by  $\mu$ -FTIR,  $\mu$ -XRF and P K-edge  $\mu$ -XANES spectroscopy. First,  $\mu$ -FT-IR spectroscopy maps of the soils were collected to avoid X-ray radiation damage of organic phosphates and ammonium compounds in the soils. Afterwards, the thin soil layers were electrostatically transferred on to an ultralene film to preserve its structure, and mounted on a sample holder for  $\mu$ -XRF and  $\mu$ -XANES measurements. Subsequently, 2D-fluorescence maps were recorded above P emission line to locate P-bearing phases and determine their association with other elements (Al, Si, Mg, Na), before  $\mu$ -XANES spectra at P K-edge were recorded on selected points.

The micro-FTIR spectra gave information on different major soil compounds (e.g. silicates, carbonates), but phosphorus and nitrogen compounds were not detectable with this technique in our soils. Probably the larger lateral resolution of  $\mu$ -FTIR spectroscopy ( $5\text{-}10\ \mu\text{m}^2$ ), instead of  $\mu$ -XRF/-XANES spectroscopy ( $<1\ \mu\text{m}^2$ ), is the reason that no P and N compounds could be detected by  $\mu$ -FTIR spectroscopy because the major silicate IR bands overlap with the weak absorption bands of the phosphates. Maybe an  $\mu$ -IR instrument with a comparable lateral resolution to the  $\mu$ -XRF/-XANES system, like the new mIRage AFM-IR system, could be a good comparable technique to the beamline. Because IR spectroscopy has the advantage in opposite to XANES spectroscopy that it is very sensitive for organic phosphorus and nitrogen compounds.

However, the collected  $\mu$ -XRF/-XANES data gave us information on the phosphorus distribution and speciation in the soils. Fig. 1 shows a micro-XRF map of P in a soil sample. P hotspots could be detected and  $\mu$ -XANES spectra collect at the hotspots.



*Fig. 1: P K-edge micro-XANES of soil from pot experiment*