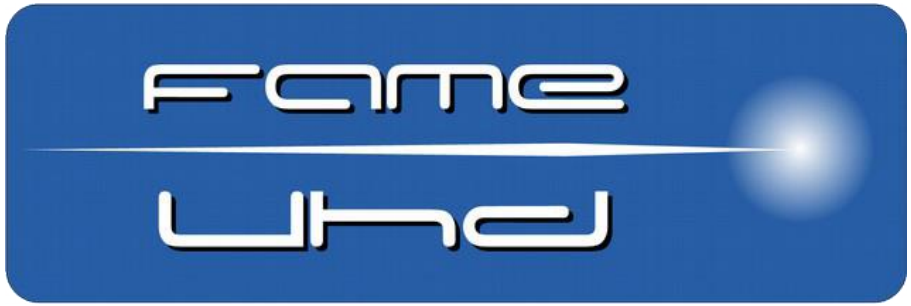
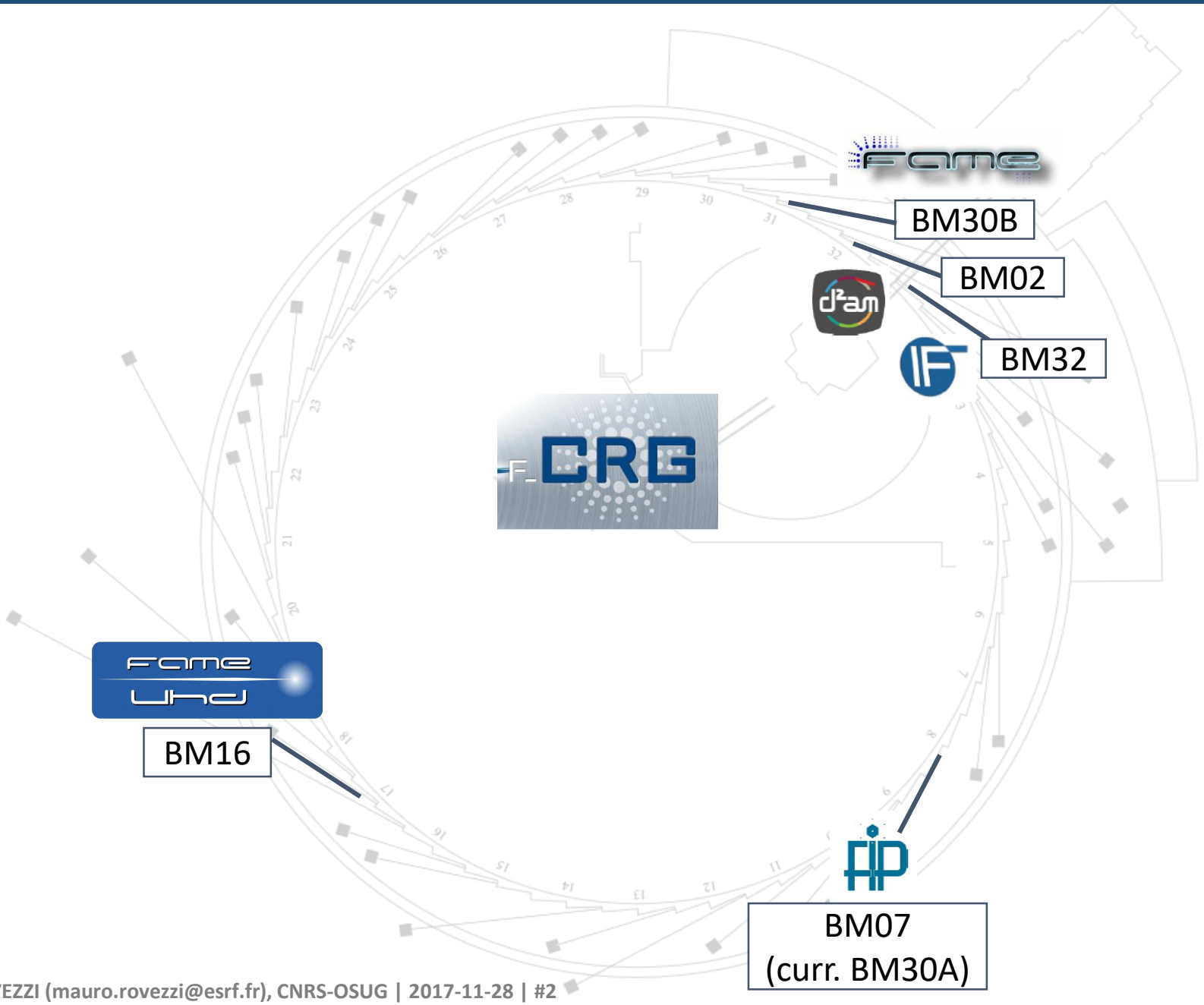
The national X-ray absorption spectrometers FAME and FAME-UHD after the ESRF-EBS phase

French **A**bsorption spectroscopy beamline for **M**aterials and **E**nvironmental sciences



Ultra **H**igh **D**ilution

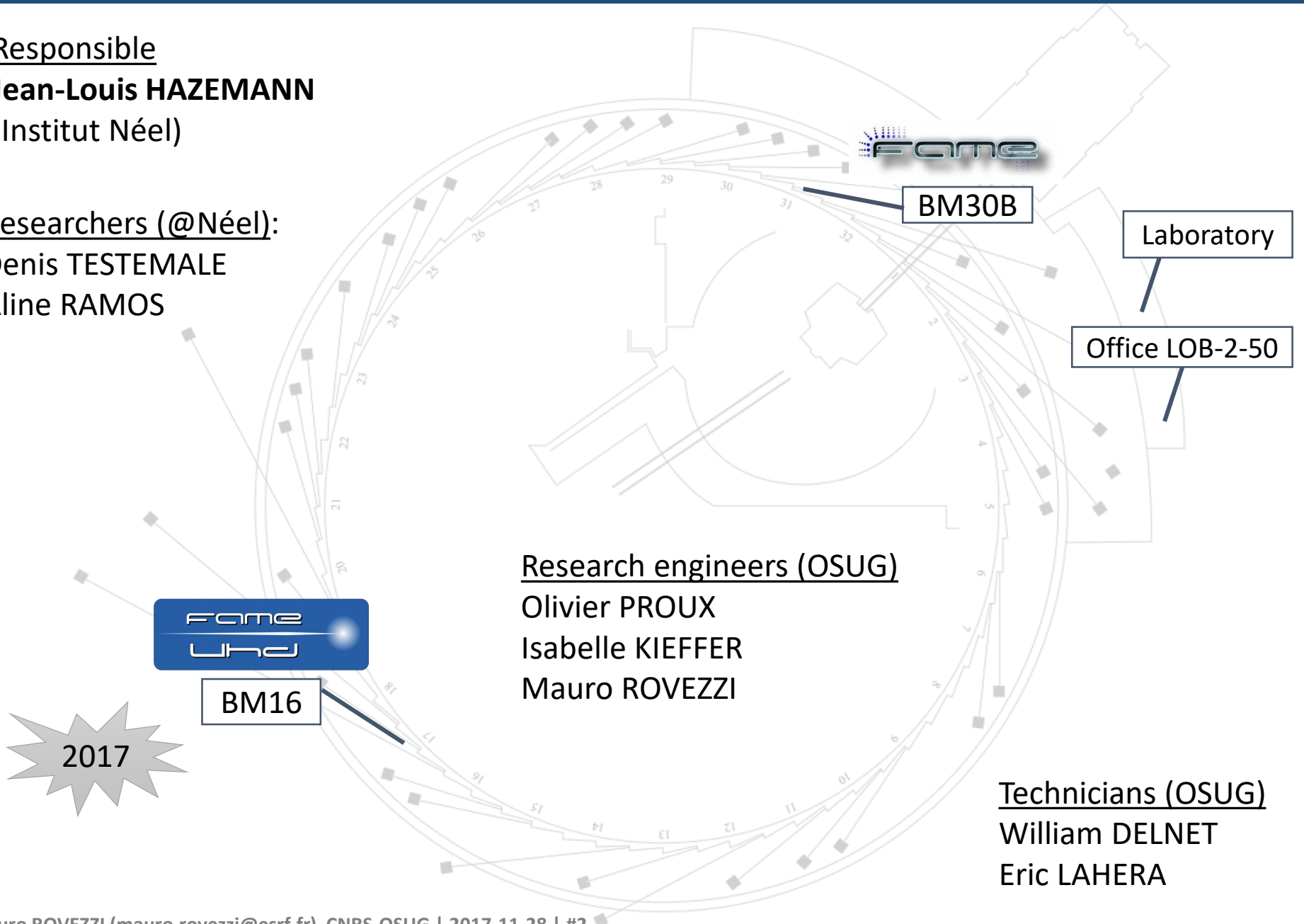
F_CRG pool



FAMEs group

Responsible
Jean-Louis HAZEMANN
(Institut Néel)

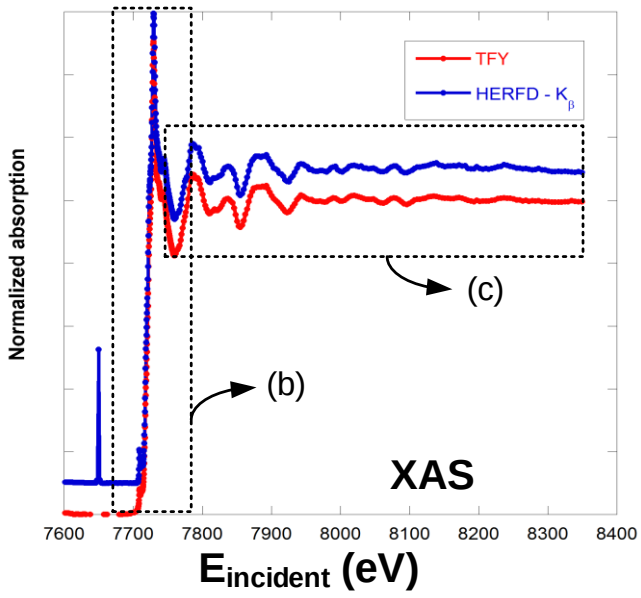
Researchers (@Néel):
Denis TESTEMALE
Aline RAMOS

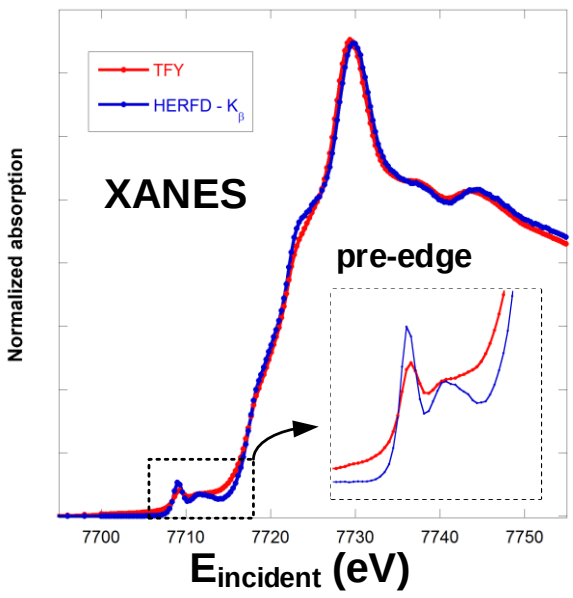


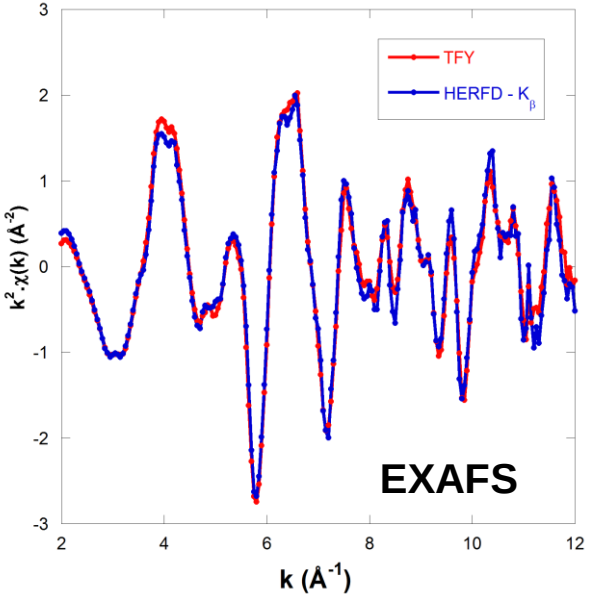
Research engineers (OSUG)
Olivier PROUX
Isabelle KIEFFER
Mauro ROVEZZI

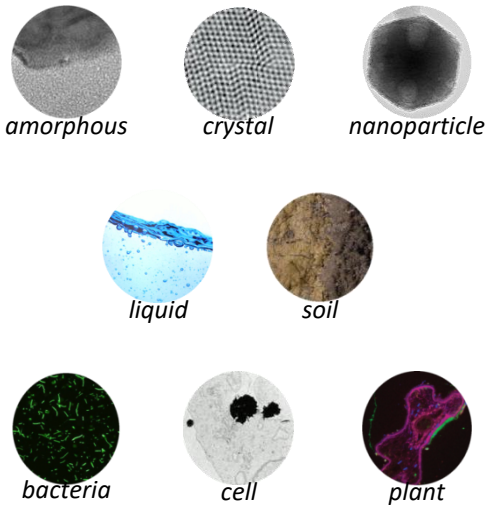
Technicians (OSUG)
William DELNET
Eric LAHERA

Hard X-ray Absorption Spectroscopy





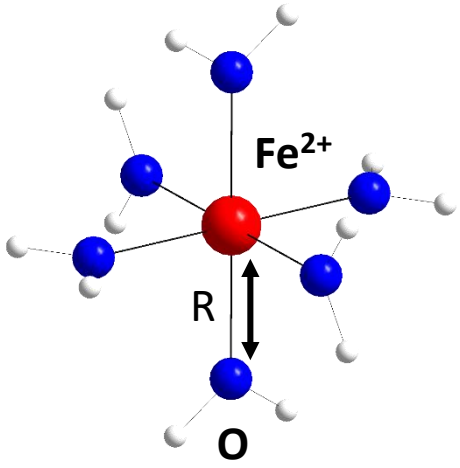




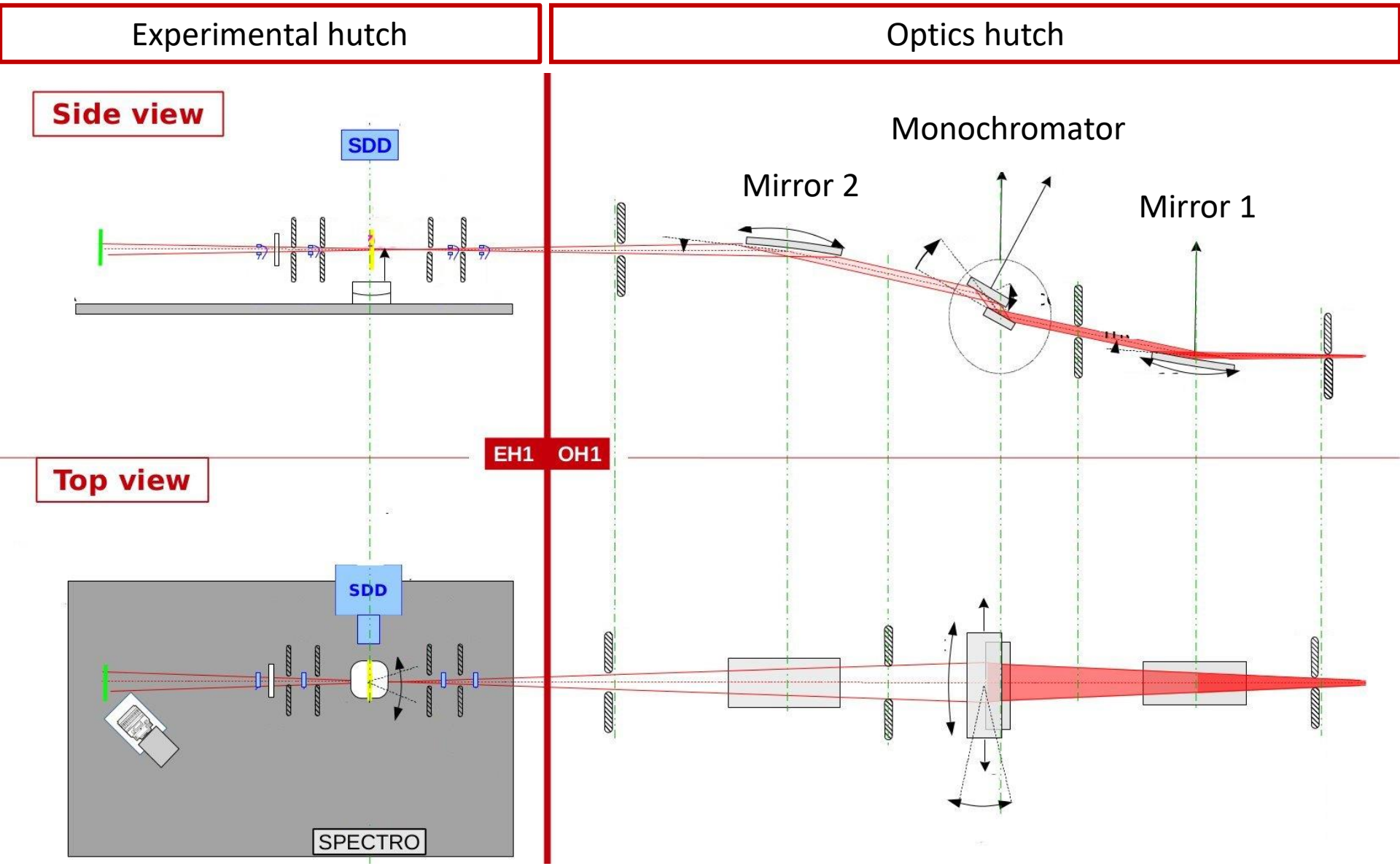
XANES / EXAFS

- Chemical selectivity
  - Valence state
  - Site geometry
  - Local structure

- Energy range: 4.8 → 20 (40) keV
- Elements: Ti→La (K-edges)
  Te→U (L-edges)



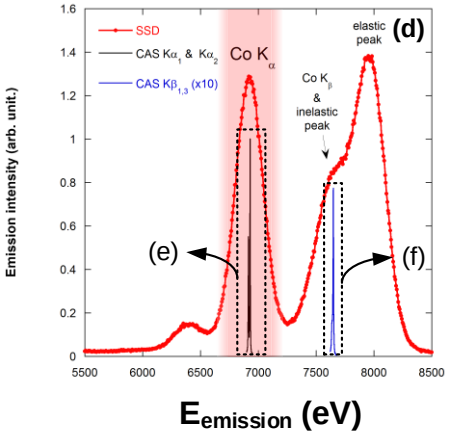
FAME / FAME-UHD: common optical layout



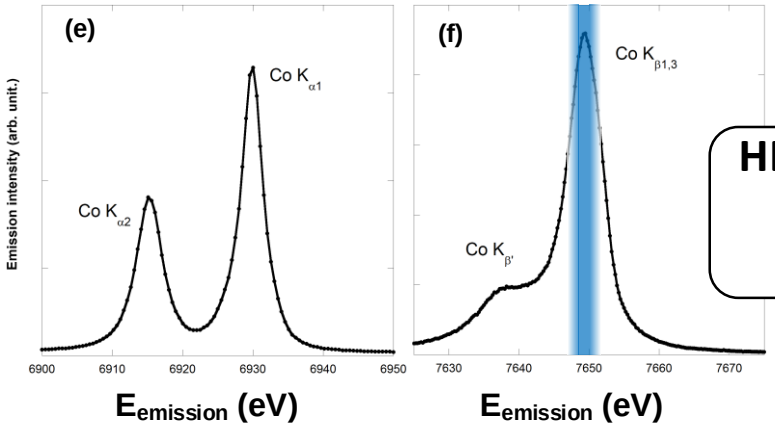
FAME / FAME-UHD: detection schemes

X-ray Fluorescence

EXAFS
XANES



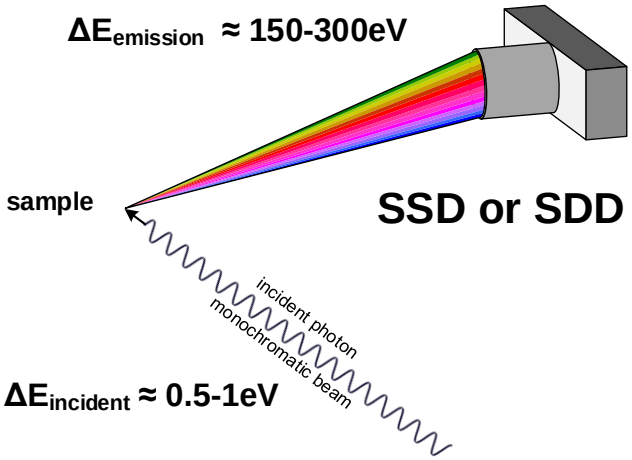
X-ray Emission Spectroscopy



HERFD-XAS
+XES
+RIXS

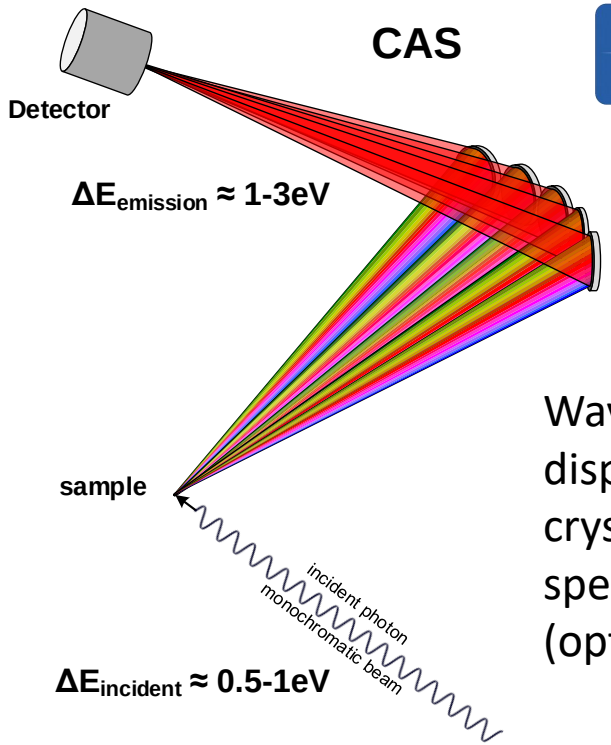
FAME

Energy dispersion with a solid state detector (electronic)



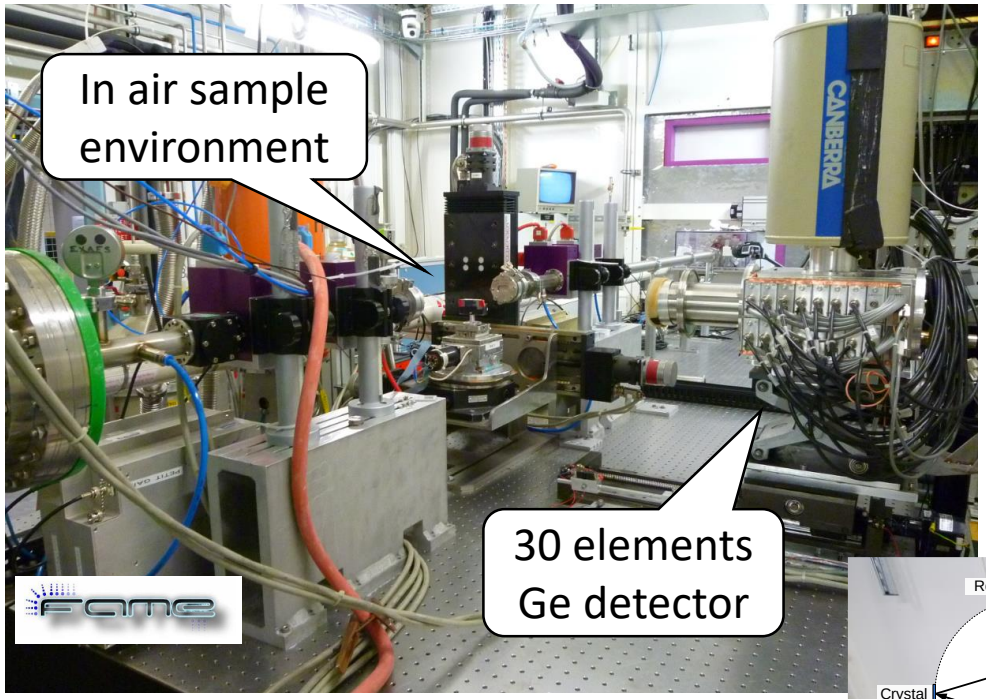
CAS

FAME UHD



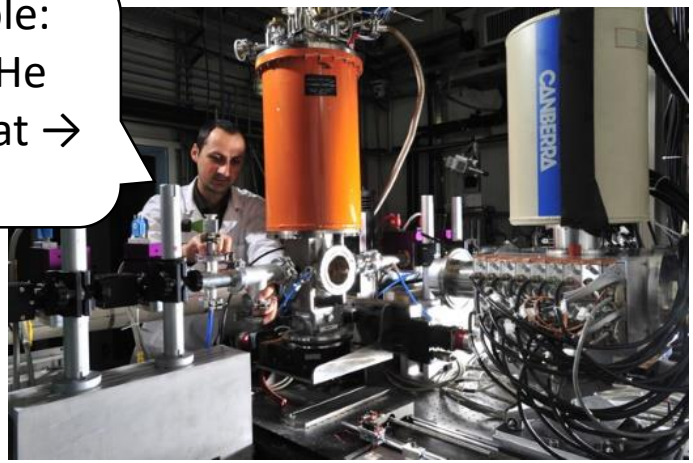
Wavelength dispersion with a crystal analyser spectrometer (optical)

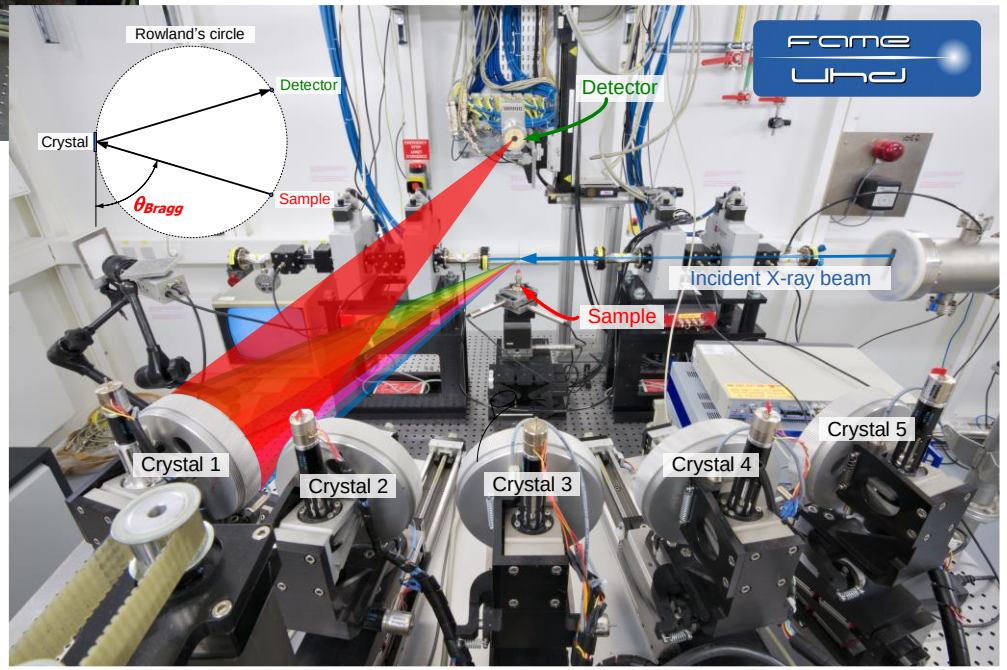
FAME / FAME-UHD: sample environment



Example: high pressure / high temperature vessel → 2000 bar, 1500 °C

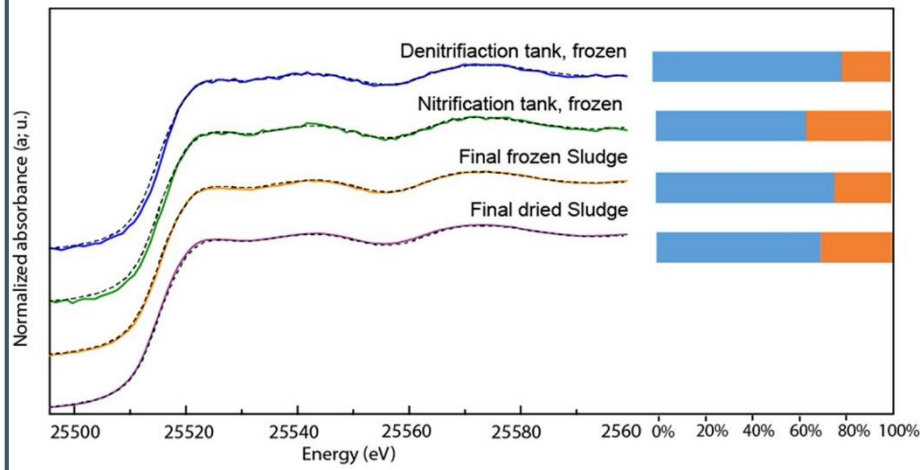
Example: liquid He cryostat → 4 K

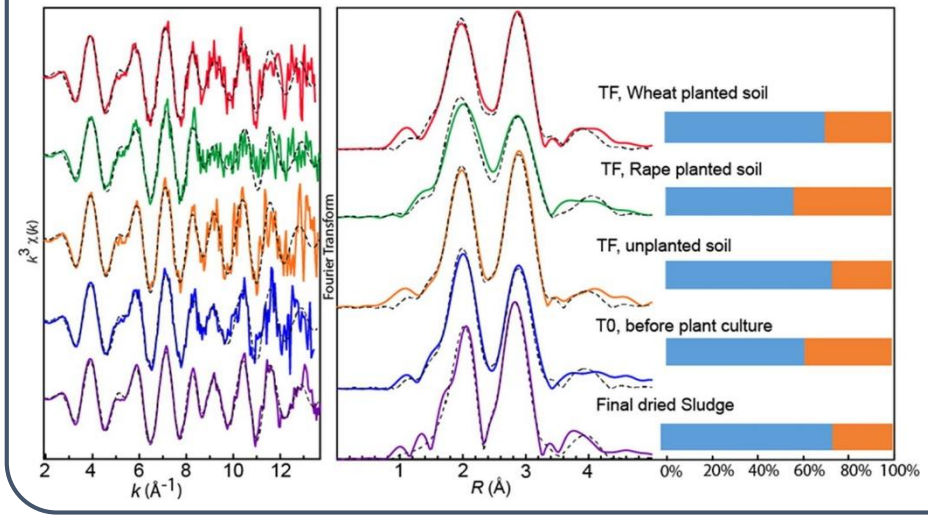




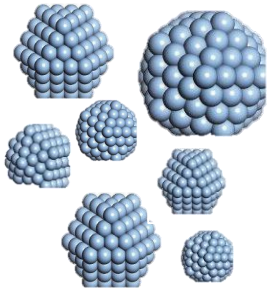
FAME: state-of-the art EXAFS

Environmental Science & Biology: **Ag-NPs in sewage sludge** applied on agricultural soils

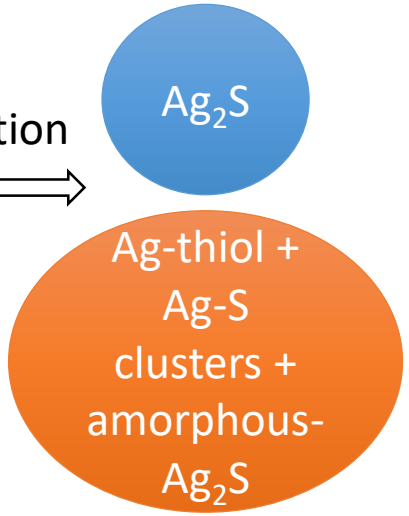




- XANES (PCA + LCF) and EXAFS
- complementary to XRF/TEM microscopy



sulfidation

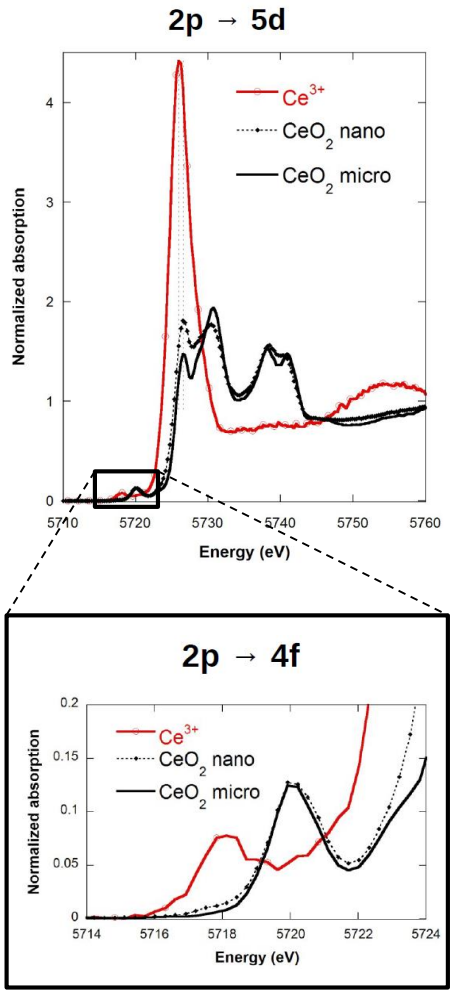


Ana E. Pradas del Real et al., "Fate of Ag-NPs in Sewage Sludge after Application on Agricultural Soils", *Environ. Sci. Technol.* 2016 **50**(4), 1759-1768

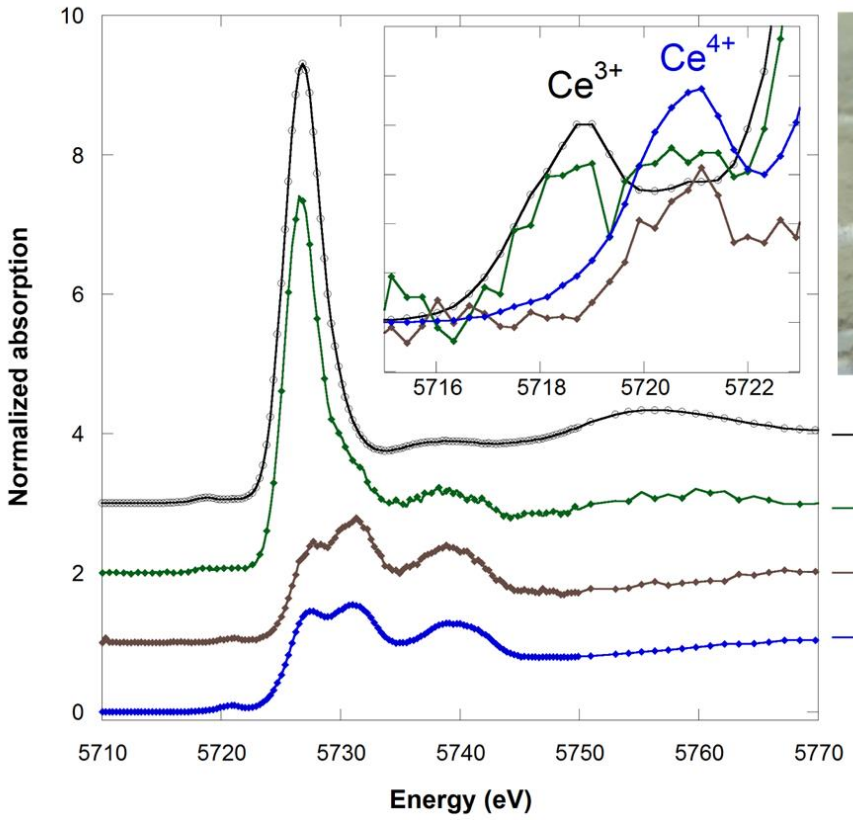
Detection limit: **10-100 ppm**

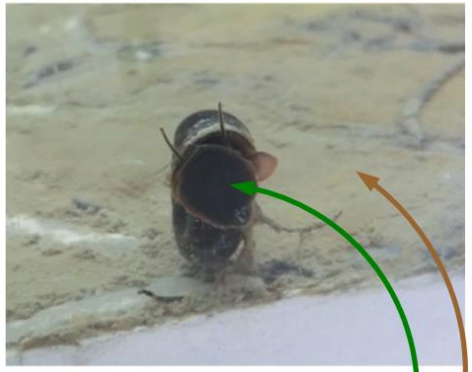
Hazemann et al., "High Resolution Spectroscopy on an X-ray Absorption Beamline", *J. Synchr. Rad.* **16** (2009) 283-292

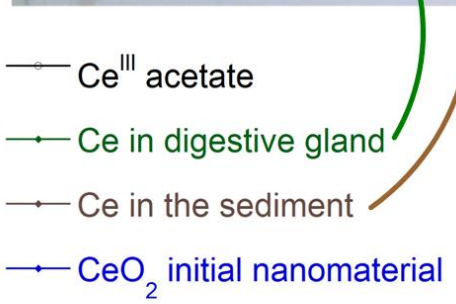
FAME-UHD: lower the detection limit + HERFD-XAS

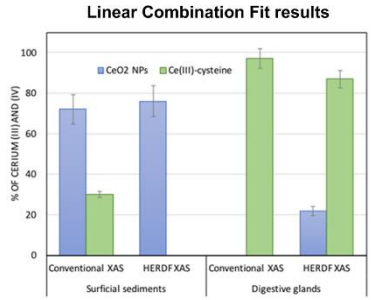


CeO_2-NPs in contaminated pond ecosystems









M. Tella et al, *Environ. Sci.: Nano* **2** (2015) 653-663

Detection limit: ≈**0.5 ppm**

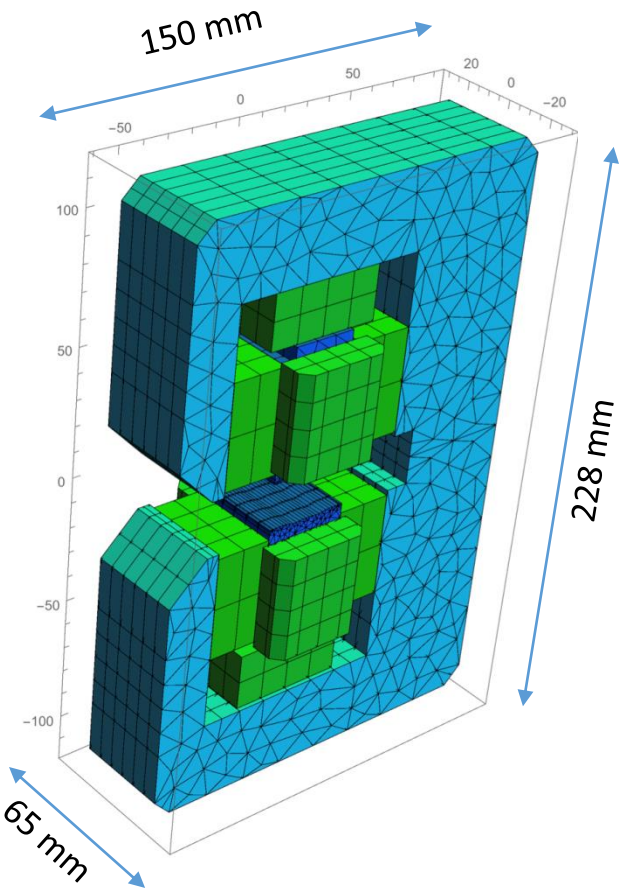
Proux et al., "High-Energy Resolution Fluorescence Detected X-ray Absorption Spectroscopy: A Powerful New Structural Tool in Environmental Biogeochemistry Sciences", *J. Env. Quality* **46** (2017) 1146-1157

EBS for a bending magnet spectroscopy beamline

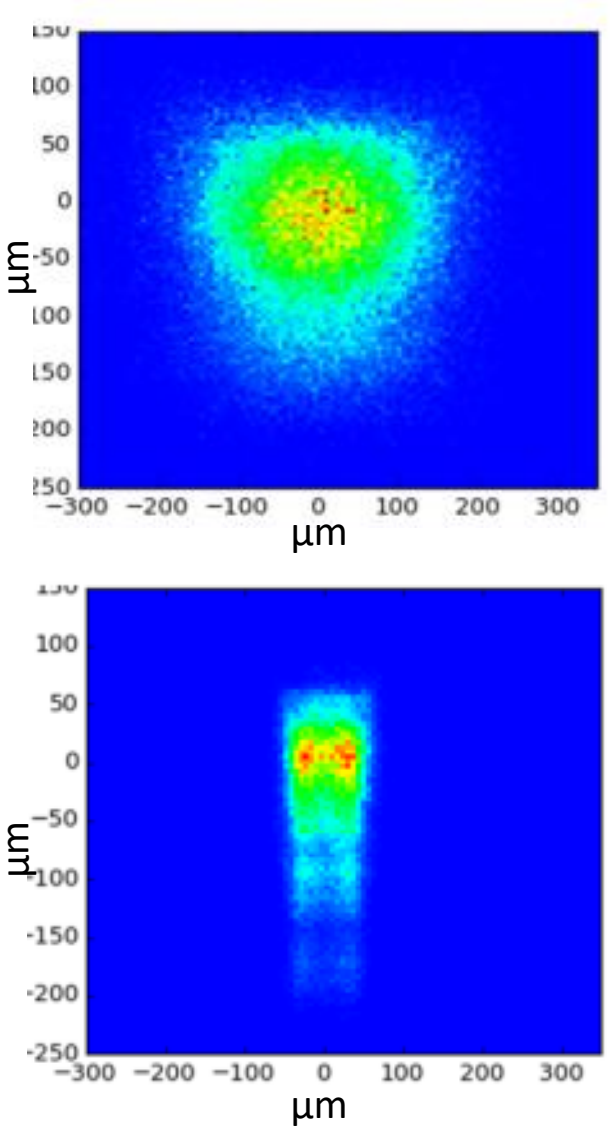
Short bending magnet source

Peak field: 0.856 T

Front end aperture: 2.2 mrad



Ray tracing simulations (Shadow)



2019: heavy works on the beamlines!

FAME

- FIP migrates on BM07 → new spaces
  - Beamline moves up to 146 mm

FAME-UHD

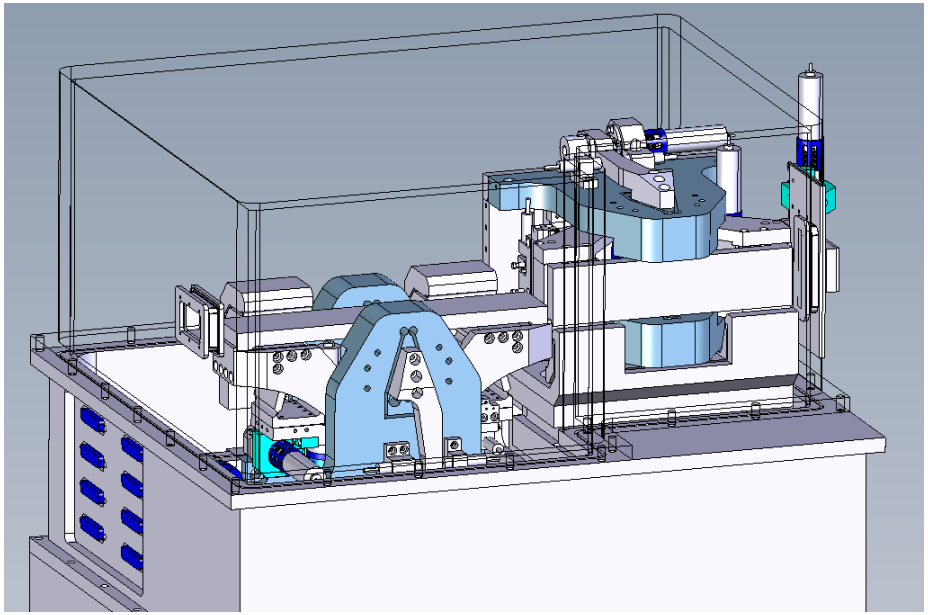
- Beamline moves up to 40 mm

Optics ready to take the power of the new source → restart in 2020

FAME after ESRF-EBS

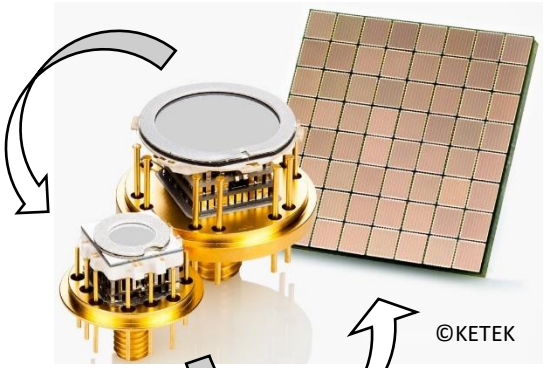
Micro-spectroscopy at 1×1 µm^2

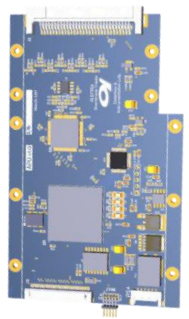
- New optics: mirrors and sagittal bender
  - Full flux in 30×30 µm^2
  - Secondary source + KB mirrors
- <u>Expected gain in flux density up to ≈60</u>



- Compact sample environments (e.g. diamond anvil cell)

New detector and electronics





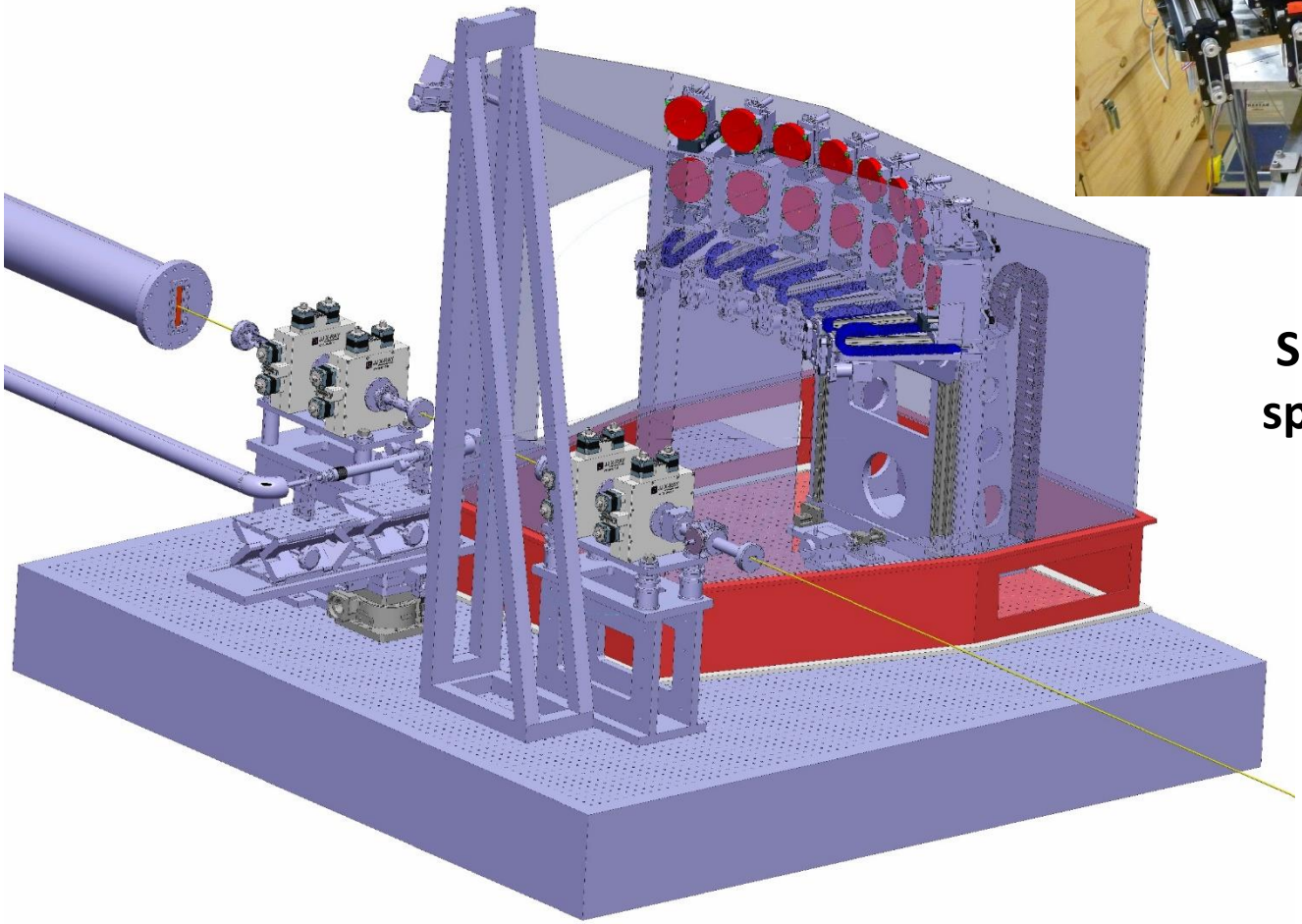
- Improve energy resolution
  - Decrease dead time
- <u>Expected gain in count rate ≈10</u>

Project MAGNIFIX (F_CRG)

FAME-UHD after ESRF-EBS

Gain in flux density

- Full flux in 30×30 µm^2
- Better energy resolution
- Lower background (scattering)



Spectrometer with 14 spherically bent crystal analyzers

- Commissioning in January 2018
- Procurement of new full sets of crystal analyzers

Conclusions

	EXAFS / XANES	HERFD-XAS
High Dilution	10 ppm	0.5 ppm and below
Inhomogeneity	≈30×30 µm^2 (full flux) ≈1×1 µm2 (µbeam)	≈30×30 µm^2 (full flux)

<u>High throughput</u> spectroscopy on natural samples

Conclusions

ESRF-EBS

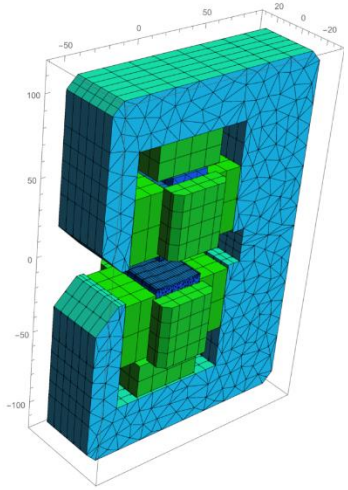
	EXAFS / XANES	HERFD-XAS
High throughput spectroscopy on natural samples		
High Dilution	10 ppm	0.5 ppm and below
Inhomogeneity	≈30×30 µm² (full flux) ≈1×1 µm2 (µbeam)	≈30×30 µm² (full flux)

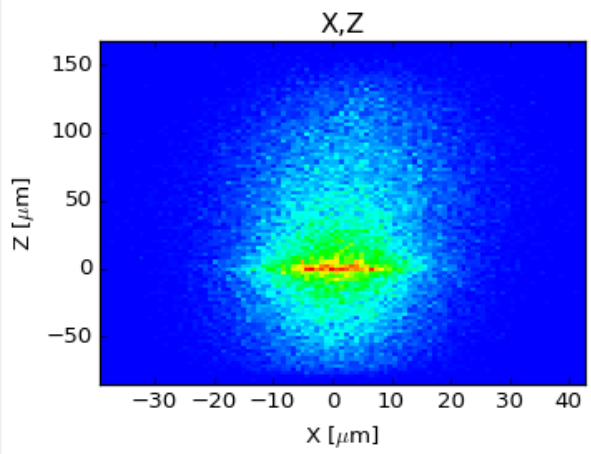
Next call for proposals: March 2018

Thank you for your attention

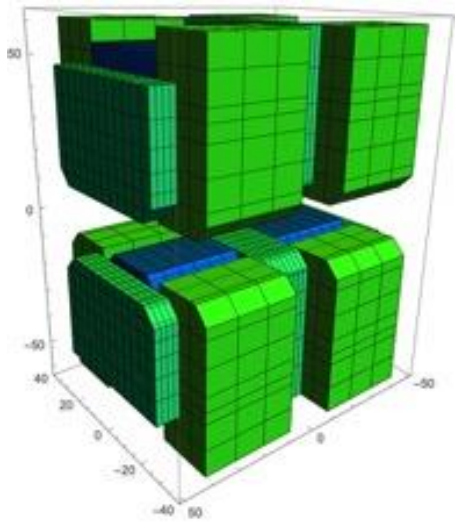
Choice of the new source: SBM, 3PW or 2PW?

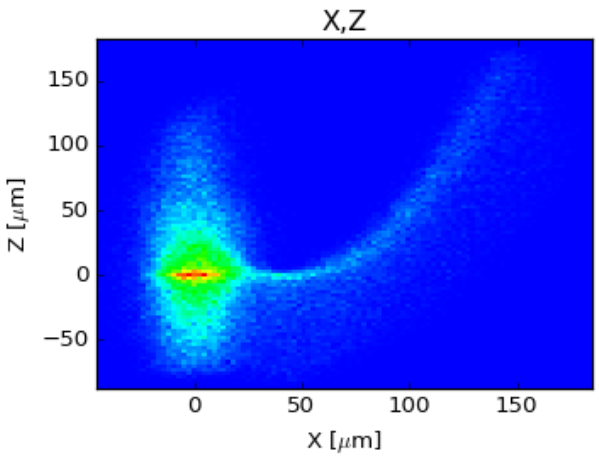
SBM





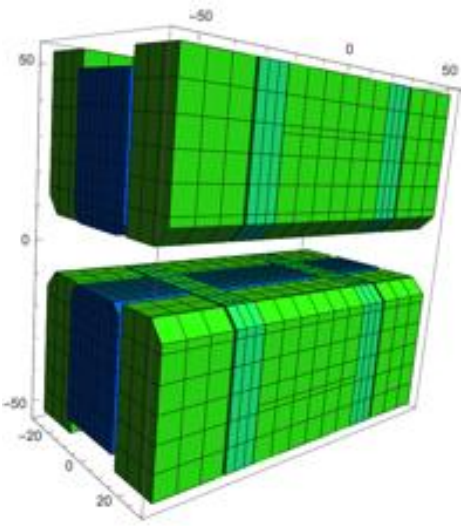
2PW

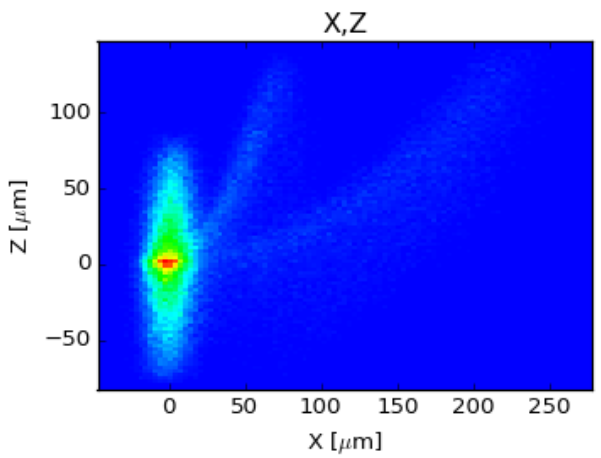




≈50% out of focus

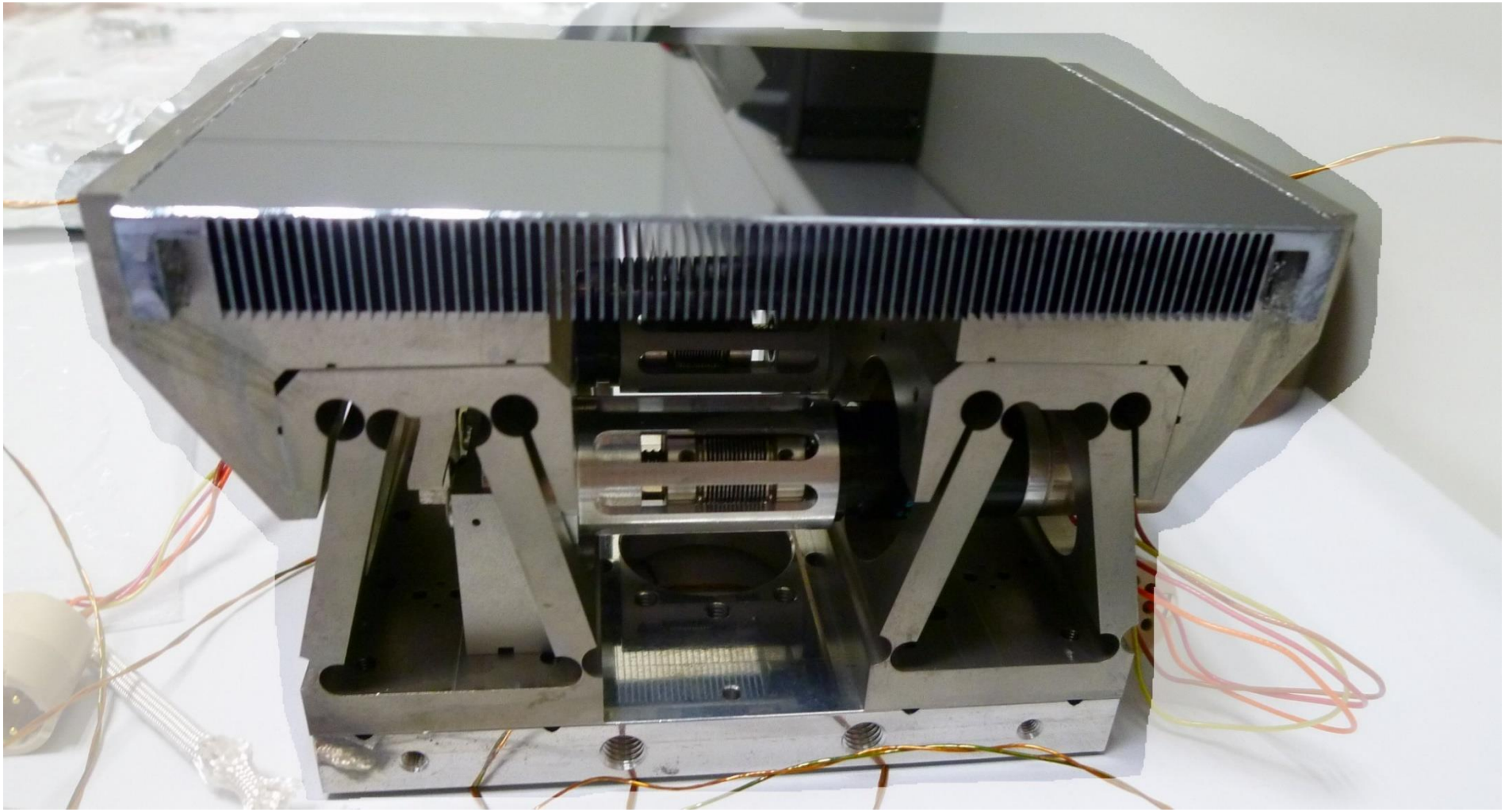
3PW



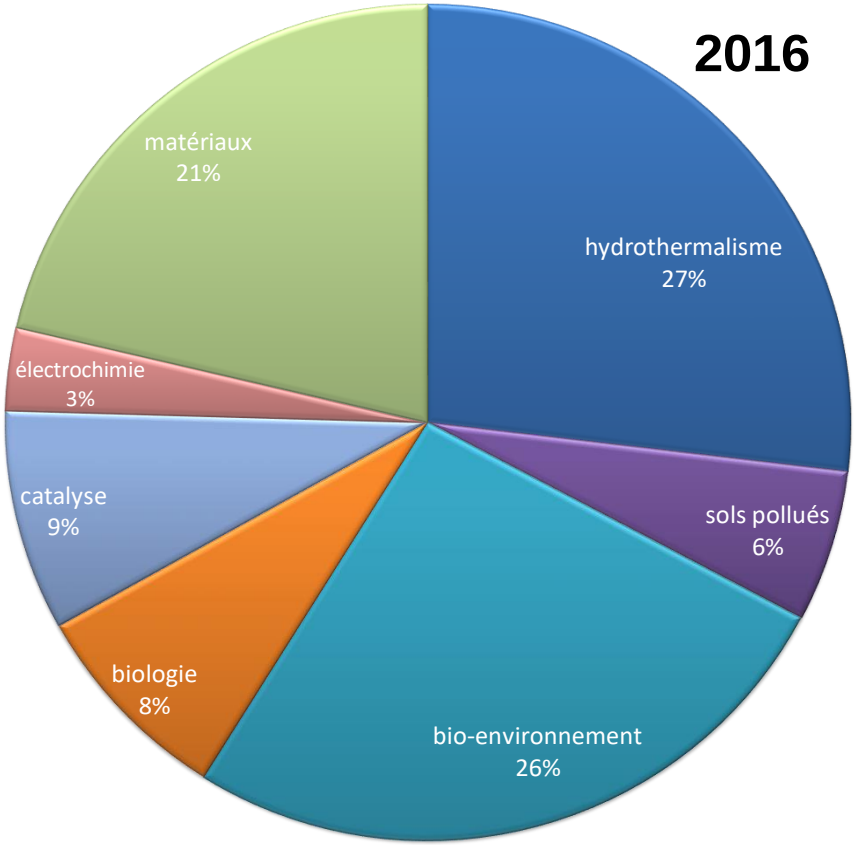


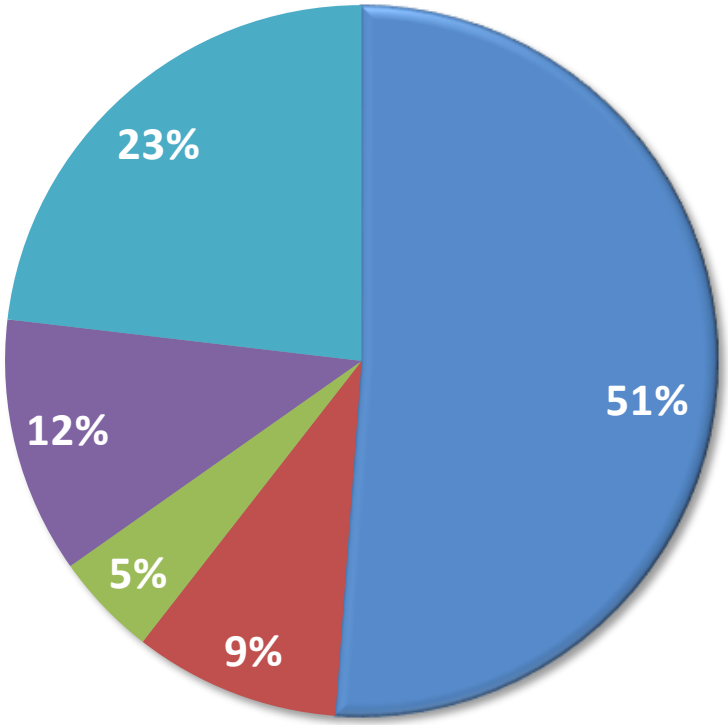
≈40% out of focus

Monochromator sagittal bender and crystal design



Users and scientific fields





Rhône-Alpes
Paris & région parisienne
PACA
Reste de la France
International