P65 – beamline set-up and first results

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Applications:

- Catalytical chemistry
- Energy storage / batteries
- Material science
- Environmental and geoscience
- ...

Key parameter:

- Beamsize: 1 x 1 mm²
- Photon flux: 10^{12} s^{-1}
- Energy range: 4 – 44 keV
- Easy to handle standard operating procedures for inexperienced users
- Complementary to P64

Infrastructure:

- Sardana/Tango based beamline control system
- 2.5 x 1.2 m² large experimental table with standard EXAFS set-up
- Exp. Hutch temperature stabilised to +/- 1° C
- Ample space for in-situ set-ups
- Infrastructure for problematic gases
- Detectors: Ionisation chambers, PIPS, 7 pixel HPGe detector (Canberra)
- Sample preparation lab shared with beamline P64
- Fume hut, glove box, lab benches and equipment for sample preparation like an analytical balance and pellet press

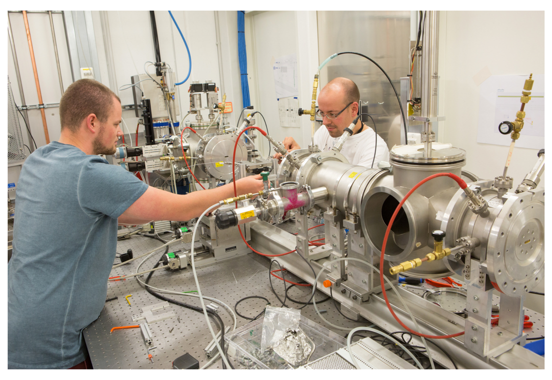
Beamline design:

- Source: Short undulator
  λ = 32.8mm
  K_{max} = 2.70
  N = 11
  E_1= 2.3 keV
- 2 water cooled plane mirrors for higher harmonics rejection and power load reduction, Si/Rh/Pt stripes
- Water cooled double crystal monochromator (DCM)
- Short distance between DCM and sample for improved stability

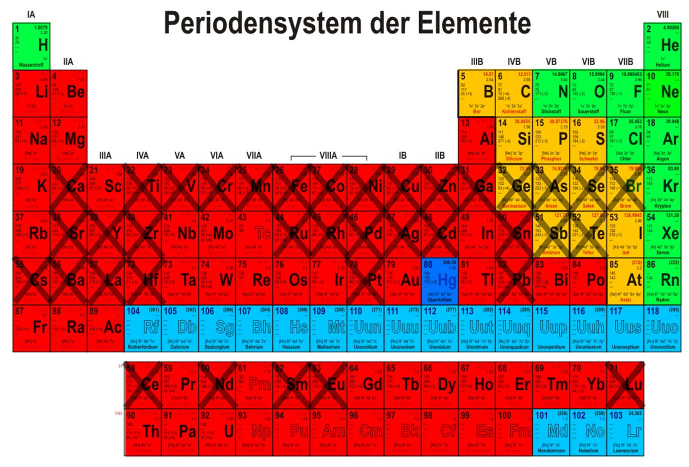
Start of user operation at P65:

	Shifts requested	Shifts available	Overbooking
I-2016	285	156	1.83
II-2016	417	213	1.96
I-2017	317	168	1.89

- More than 50 % of all applications were in-situ experiments, catalysis and energy storage (batteries/fuel cells)
- 2/3 of the applications required fluorescence detection
- About 40 % required the liquid He cryostat

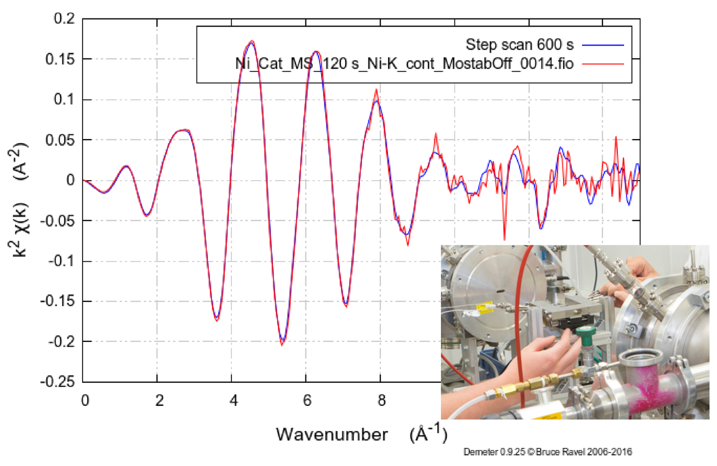


Users from the TU-Munich set up their sample-cell of for an in-situ experiment

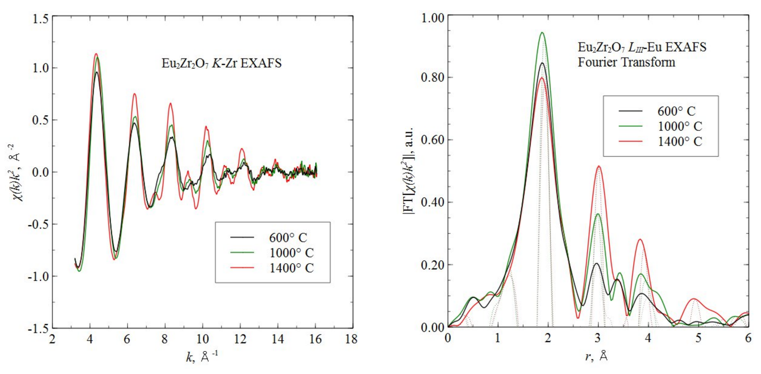


Elements measured so far at P65. All elements heavier than K can be measured at P65 using either their K or L_3 edges.

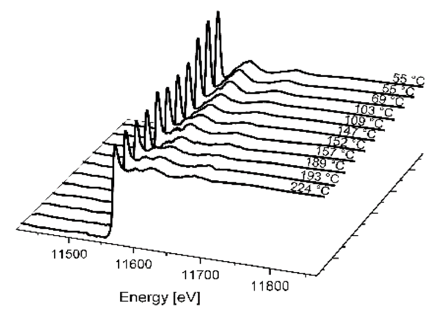
Results from user experiments:



Total fluorescence yield Ni K-edge EXAFS, powder sample (Ni catalyst on Al_2O_3 in a capillary 1 mm diameter), room temperature, Si 111, Si/Si mirrors, 2 mrad, step scan ~600 s, cont. scan 120 s (M. Steib, Ak Jentys, TU München)

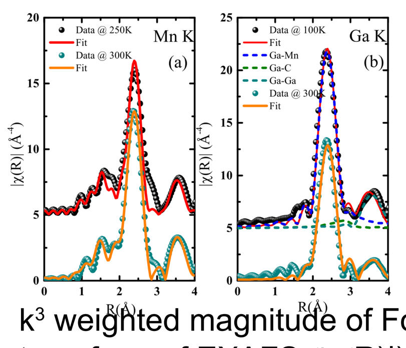


Zr K EXAFS spectra measured in $Eu_2Zr_2O_7$ nano particles, illustrating the evolution of crystallinity versus annealing temperature. Step scans, 10 min per scan, powder samples (R. Chernikov, DESY), substances like these are used as thermal barrier coatings for gas turbine transition ducts (see right picture) etc.
A. P. Menushenkov et al, Moscow Engineering Physics Institute, Moscow, Russia

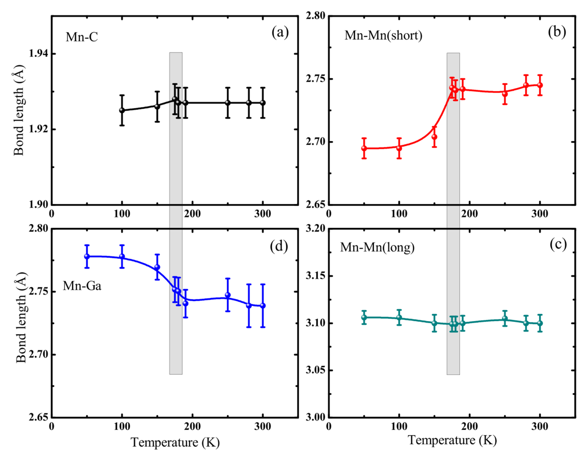


Very first in-situ experiment at P65: XANES data during heating of a Pt-catalyst in 10% O_2 5000 ppm CH_4, 3% H_2O in He
A. Gaenzler, Ak Grunwaldt, KIT

Mn_3GaC undergoes a ferromagnetic to antiferromagnetic, volume discontinuous cubic-cubic phase transition as a function of temperature, pressure, and magnetic field. Through a series of temperature dependent XAFS experiments at the Mn K and Ga K edge, it was shown that the first order magnetic transformation in Mn_3GaC is entirely due to distortions in the Mn sub-lattice.



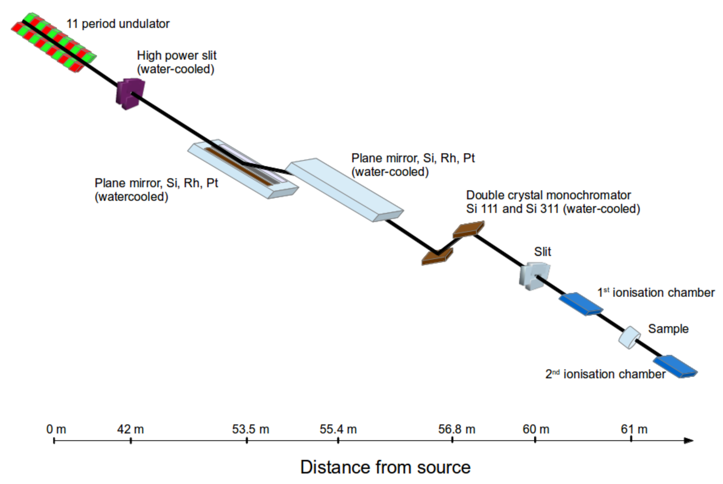
k^3 weighted magnitude of Fourier transform of EXAFS (|χ(R)|) along with best fitted lines at (a) Mn K edge and (b) Ga K edge at two different temperatures.

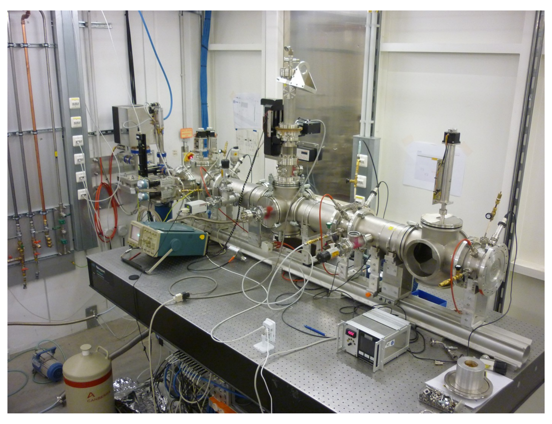


Plot of variation of different bond distances obtained from analysis of Mn K and Ga K XAFS. (a) Mn-C bond distance, (b) Mn-Mn short bond distance, (c) Mn-Mn long bond distance, and (d) Mn-Ga bond distance. While Mn-C and Mn-Mn bond distances have been obtained from Mn XAFS analysis, Mn-Ga bond distances have been obtained from Ga K XAFS analysis.

From: K. R. Priolkar et al., APPL. PHYS. **122**, 103906 (2017)

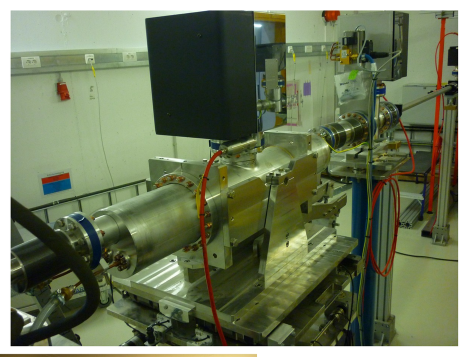
Beamline components:

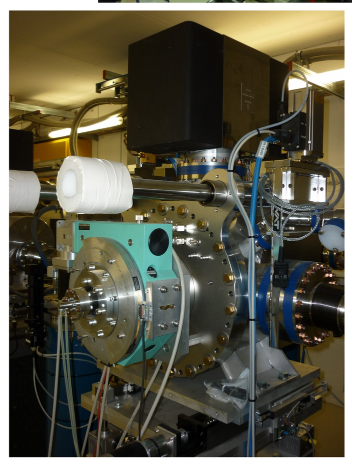




Standard set-up for ex-situ experiments

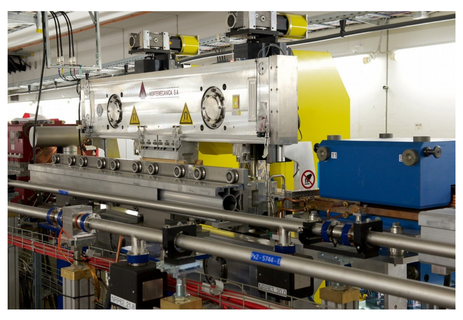
The first mirror chamber





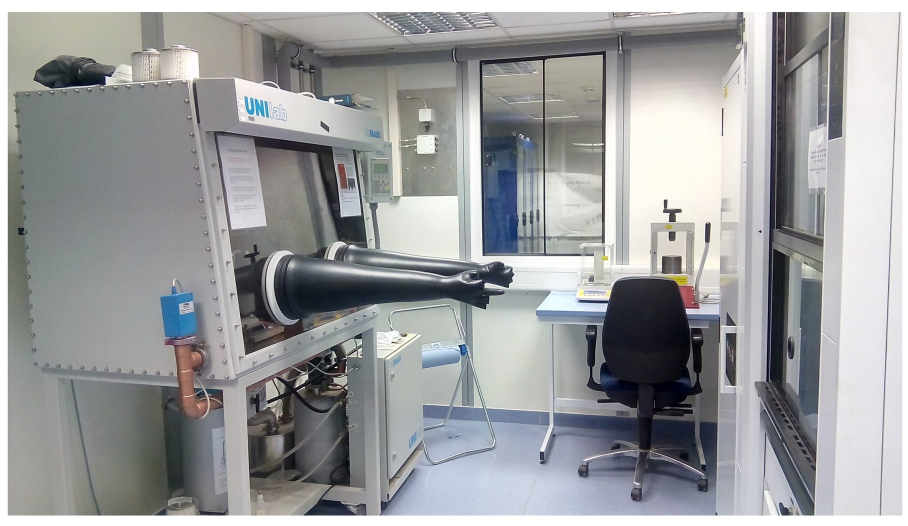
The double crystal monochromator for P65 installed at the beamline.

View into the optics hutch



The short 11 period undulatr of P65

Infrastructure for in-situ experiments:



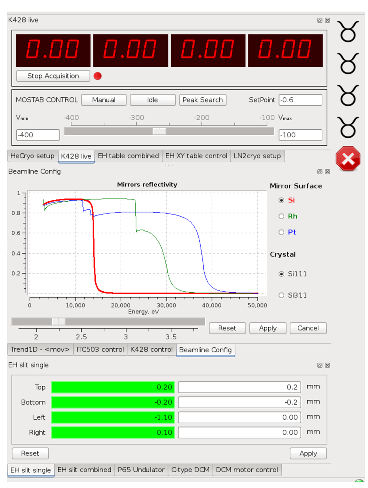
P64/65 sample preparation lab with equipped with a fume hutch, glove box, infrastructure for use of gases, sample press, analytical balance...

Gas connectors, pressurised air and cooling water at P65

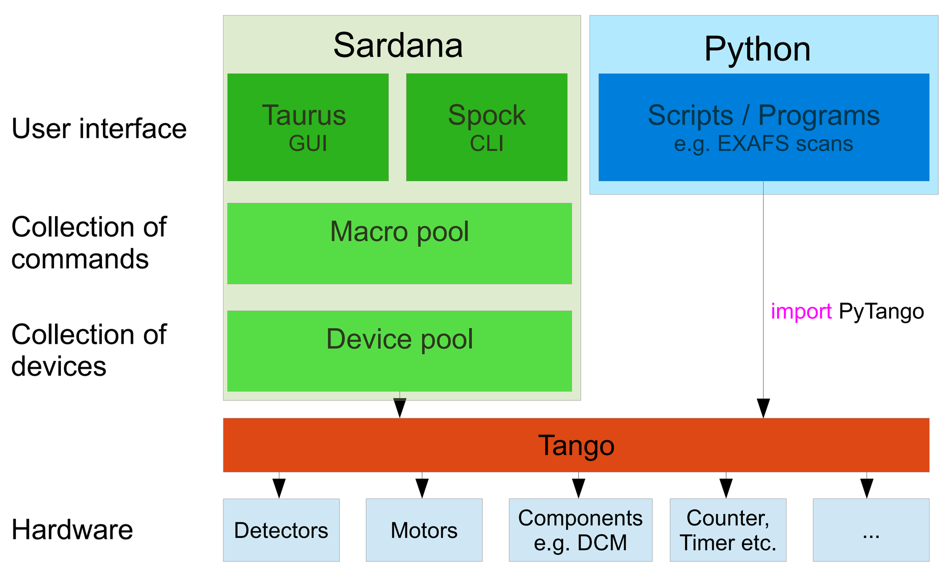
Gas cabinets for toxic, inflammable and oxidising gases

Safety system for sensors and exhaust control

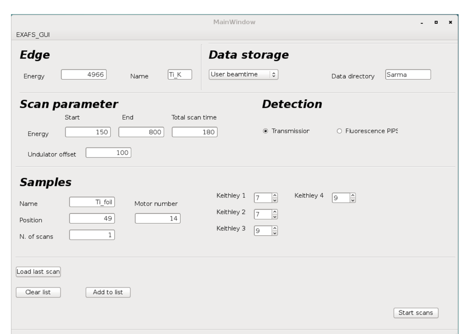
Beamline control software:

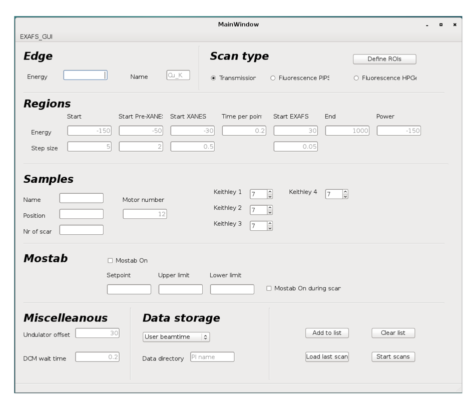


Taurus GUI for Keithleys, Mostab and beamline configuration



Scheme of the beamline control software, currently EXAFS scans are not intergrated into Sardana but done using pure Python scripts





GUIs for continuous (left) and step mode scans (above).