A highly automated beamline for analytical XAFS at the future PETRA IV storage ring

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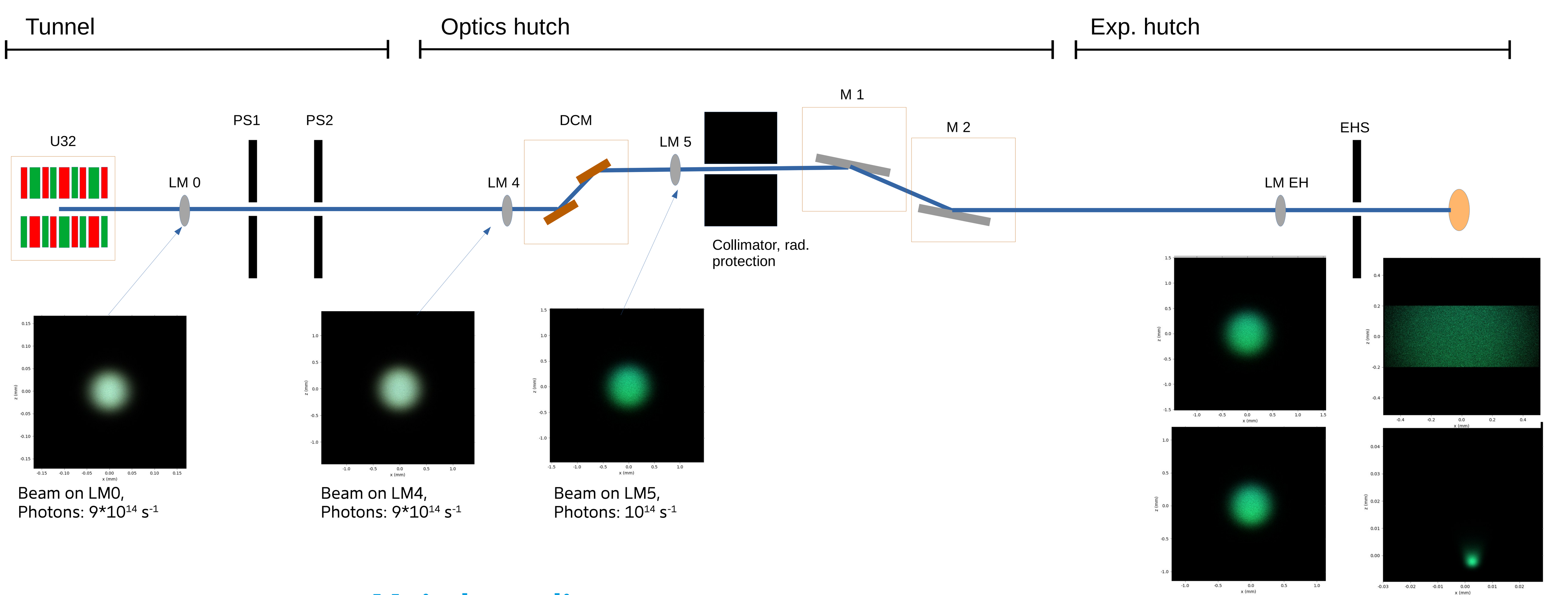
Analytical XAFS at PETRA IV

XAFS spectroscopy is applied in a broad variety of different fields of science like environmental and geoscience or solid state physics and material sciences but by far the lions share of XAFS applications is found in the whole spectrum of catalysis and battery research where it is an indispensable tool for the elucidation of fundamental relations between structure and function of a catalyst. In this context XAFS spectroscopy is used as one, often decisive, analytical method among other analytical methods needed to get a full understanding of the material or process under investigation.

Key beamline parameter

- Working range: 4 keV – 45 keV
- Beamsize: ~ I mm^2 to 100*100 µm^2
- Monochromatic flux: > 10^{13} s^{-1}
- Infrastructure for in-situ experiments (gases etc.)
- Chemistry lab near to the beamline
- Fast scans with time resolution of 1 – 10 s per scan
- High degree of automation for ex- and in-situ experiments

Beamline schematic



Beam on LM0, Photons: 9*10^{14} s^{-1}

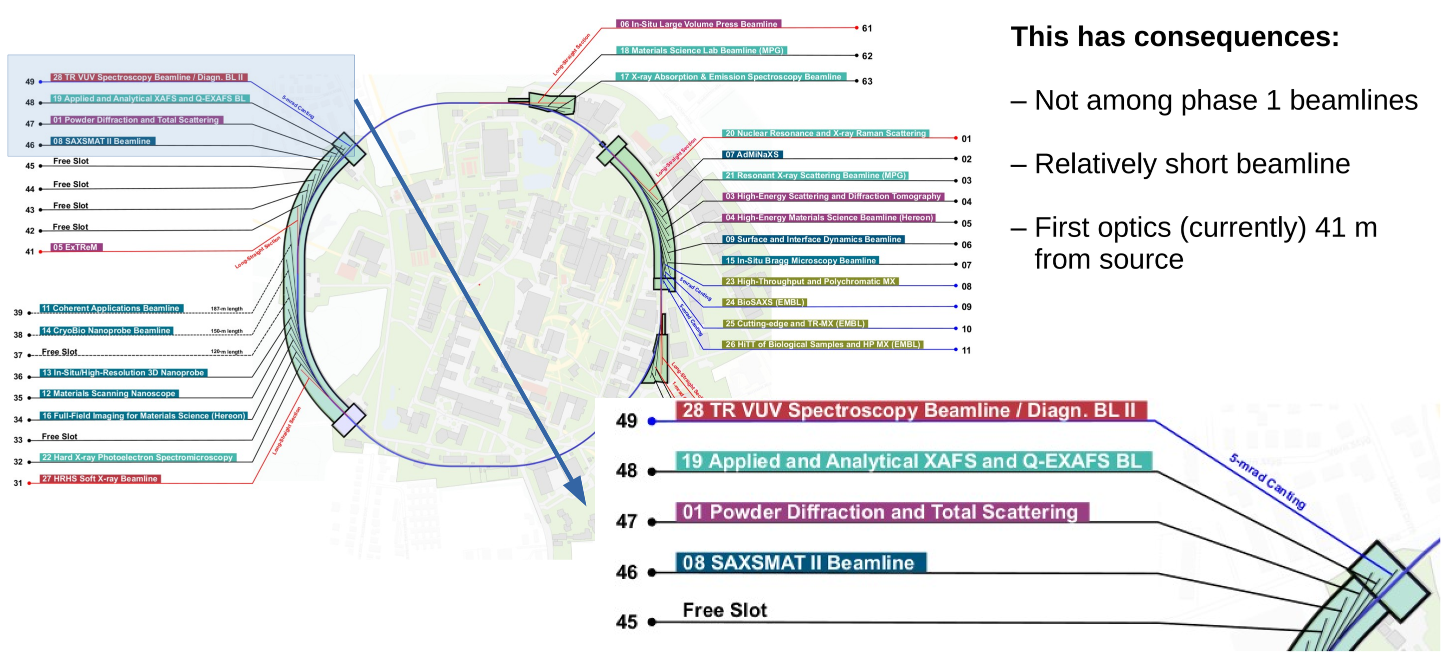
Beam on LM4, Photons: 9*10^{14} s^{-1}

Beam on LM5, Photons: 10^{14} s^{-1}

Beam on LM7, just before the final slit in the experimental hutch. Top unfocused, bottom focused beam. Photons: 10^{14} s^{-1}

Final beam spot on the sample Top unfocused, bottom focused beam. Photons: 10^{14} s^{-1}

Analytical XAFS – BL48 in PETRA IV hall West



This has consequences:

– Not among phase 1 beamlines

– Relatively short beamline

– First optics (currently) 41 m from source

Main beamline components

- 4 m long U33 undulator
- Standard PETRA IV frontend
- N_2 cooled double crystal monochromator (Si 111 and Si 311)
- Radiation protection collimator
- 2 mirrors, first mirror plane or toroid, second mirror plane, optical surfaces Si, Rh, Pt
- Final slit in exp. hutch to define beam size and potion of the unfocused beam
- Infrastructure for work with all kinds of gases, gas cabinets, tubes, sensors etc.
- Sample environments:
  - Liq. He cryostat for experiments down to 4 K
  - Ovens for work at temperatures up to ~ 1000°C
  - Equipment for automated and remote controlled in-situ and ex-situ experiments (see below)

The ROCK-IT project

(**R**emote, **O**perando **C**ontrolled, **K**nowledge-driven, **IT**-based)

Cooperation between DESY, HZDR, KIT and HZB

Concepts for highly automated and/or remote controlled in-situ experiments are currently developed and tested within the *ROCK-IT* project. At DESY the XAFS beamline P65 is chosen as the place were a demonstrator experiment is set up until the end of 2025. While ex-situ experiments are relatively easy to automate, the automation experimental techniques like protein crystallography

Objectives of ROCK-It:

- Automation/remote control of in-situ experiments involving gases
- AI-based experiment control and data evaluation
- Sample tracking and handling
- Standardized and interchangeable data formats
- Standardized metadata collection and interfaces to electronic lab books
- Setting up a demonstrator at P65 (operando XAFS – catalysis research)

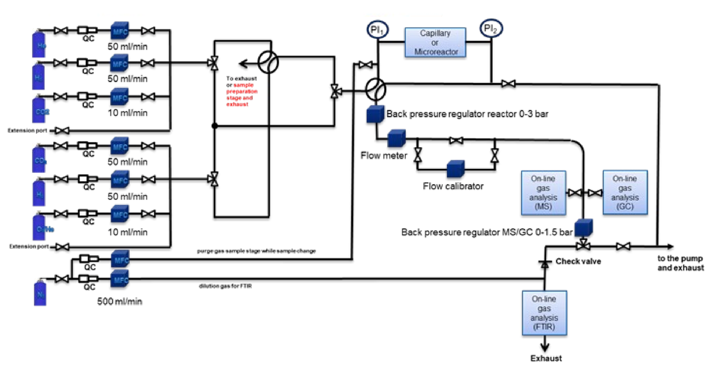
Automation of in-situ experiments at PETRA III XAFS beamline P65

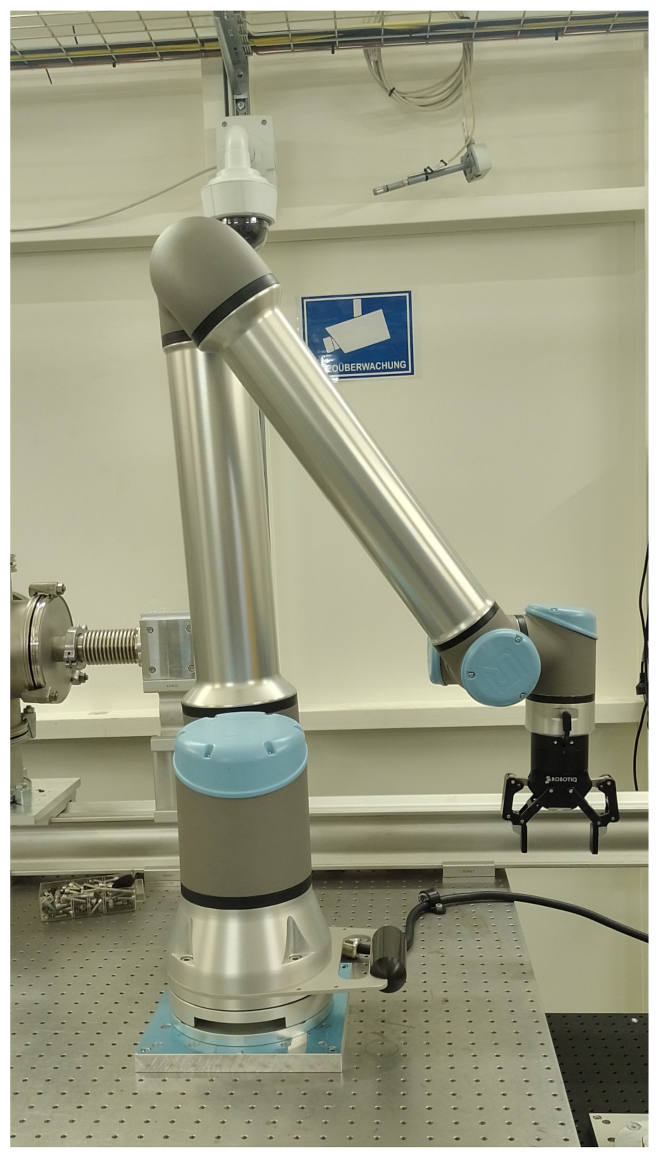
Tailored sample environments for in-situ and operando experiments

Exemplary reactor cells with robotic sample mounting

Standardized gas supply and product analysis infrastructure

Full automation including allocation of complete data and metadata





Universal robots UR10e at beamline P65, will be used as sample changer for automated and remote controlled in-situ experiments.

Use of the automated beamline

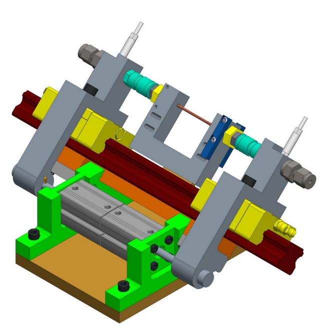
- User send samples in by mail and define the experiment to be performed
- No need to learn about how the beamline works or travel to the facility
- Real time analysis of experimental data and adaption of experimental plans depending on the outcome of the real time analysis
- Ability to live-monitor the experiment => decide whether or not to continue the experiment with this sample
- Ability to access the data as it is being produced to run own analysis scripts and change the experimental plan if needed
- Remote control to allow more experienced users to interact directly with the components of the experiment.

ROCK-IT at PETRA III beamline P65

Gas supply for toxic, flammable and inert gases at beamline P65

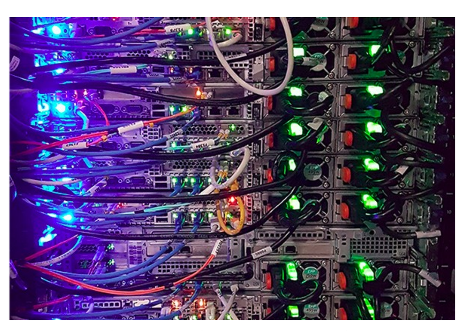
Mass flow controllers as part of the gas mixing and dosing system.

Robotic arm, used as sample changer



In situ cell for experiments with gases. The central U-shaped part is mounted by the robot. It is filled and prepared by the user and send to the facility.

Product analysis in the exhaust stream of the experiemnt. Here, the mass spectrometer at P65. Alternativly an FTIR gas analyser will be available.



Data and meta-data storage following FAIR principles. All data and meta-data of the experiment is stored in HDF5 format and made accessible via (Meta) data catalogues.

Workflow: Samples are prepared in standardised capillary reactors by the users. The robot loads the samples into the experimental station where a prepared experimental plan is executed. Data from the experiment is evaluated in real time and results form this evaluation can result in modifications of the running experimental plan if necessary. All data and meta data is stored in standardised formats. Common experimental control software, based on the *Bluesky — Experiment Specification & Orchestration* suite, including a graphical user interface will be used at all participating facilities to provide a consistent "look & feel" at the synchrotron sources PETRA III, BESSY II, KARA.

HELMHOLTZ

Bundesministerium für Bildung und Forschung