

Balder*

External collaborations	SOLEIL, Chalmers and LU
Original budget and funders	72.5 MSEK, KAW and 12 Swedish universities
Official start	September 2011
Expected date of completion	Regular users Mar/April 2017 (with limited performance)

The Balder beamline is dedicated to X-ray absorption spectroscopy (XAS) and X-ray emission spectroscopy (XES) techniques in the medium and hard X-ray energy range. The overall design allows large flexibility in energy range, beam size, data collection time, and sample environments. The focus of the beamline is studies of dilute systems, *in-situ* and *in-operando*, with relevance for natural systems, as well as the micro-scale variability and dynamics of chemical reactions on the sub-second time-scale. The research areas that benefit from Balder include environmental, biological and geochemical sciences, catalysis and energy materials, and cultural heritage.

Technical description

The Balder beamline is a wiggler based high flux beamline operating in energy range 2.4-40 keV. The beamline provides focused beam down to 100x100 μm^2 and a flux of 10^{12} - 10^{13} ph/s with time resolution down to sub-seconds for full EXAFS spectra and ~ 10 times faster for XANES spectra. Data collection can be achieved in transmission mode with ion chambers and in fluorescence mode using 7-element SDD detector that covers energy range 2-20 keV, 7-element Ge detector in energy range 5-40 keV and the X-ray emission spectrometer, SCANIA-2D, for energies 2.3-27 keV.

Technical developments at the beamline are focused on three major development areas: 1) the SCANIA-2D X-ray emission spectrometer, 2) infrastructure and sample environment to allow for *in-operando* materials research and 3) methodology and equipment to allow for XAS and XES measurements on dilute and sensitive samples. A summarised description of the development areas:

- 1) The X-ray emission spectrometer, SCANIA-2D, operates in back scattering mode and is based on the Rowland circle geometry with two independent branches for two individually selectable energies. The working energy range of the spectrometer is covered with three exchangeable sets of crystals. The applications of the X-ray emission spectrometer include:
 - *Non-resonant XES*: Emission lines are sensitive to oxidation state, spin state and chemical identity of ligands, protonation state.
 - *RIXS*: Provides better resolved K pre-edge peaks and possibility to study elements in soft X-ray energy range with hard X-rays.
 - *Range-extended EXAFS*: By integrating $K\alpha$ line of an element, XAS can be extended beyond the K absorption edge of the next element.
- 2) *In-operando* materials research in e.g. Fischer-Tropsch processes, CO_2 conversion, chemical syntheses, energy materials, nano-particle synthesis, and for studies of reaction intermediates:
 - *Gas delivery system* for mixing of inert, oxidising and reducing gases at up to 50 bar with fast switching between gases. The gas delivery system also includes a vapour generator and a mass spectrometer for measuring the composition in residual gas mixtures and *in-situ* reaction products.
 - *Sample environment* for *in-situ* measurements include gas-flow cell for simultaneous XAS/XES and XRD measurements, and a capillary based furnace for temperatures up to 1000°C.
- 3) XAS and XES on dilute and sensitive samples with applications in metallo-proteins, enzyme catalysis, metals in medicine, geological and environmental samples, trace elements, minerals, fossils and cultural heritage:
 - *Methodology for short beam exposure* on samples include scanning in continuous mode with second to sub-second time resolution in combination with fast and sensitive fluorescence detectors. Tuneable beam size, slits and filter system in combination with fast shutter for beam exposure on sample only during data collection.
 - *Closed cycle He cryostat* for temperatures down to 4K and with multiple sample mounting and translation inside the cryostat for high throughput measurements.
 - *Flow cell* for biological and other liquid samples sensitive to radiation damage.

* <https://www.maxiv.lu.se/accelerators-beamlines/beamlines/balder/>

Present status

- Beamline optics and wiggler installed. Preliminary sample environment and detectors installed. Commissioning with X-rays starting Q4 2016.
- Commissioning of the control system started in summer 2016 using laser beam.
- Procurement of the gas delivery system is on-going.
- Experimental table and the X-ray emission spectrometer are in production.
- Design of the closed cycle cryostat ongoing.

Expected status end 2018

Foreseen regular user operation with gas delivery system, X-ray emission spectrometer, cryostat and sample environments fully operational.

Major partners and additional funding

SOLEIL-MAX IV collaboration: Sample environments for catalysis research (59 k€) and wiggler development/construction (4 MSEK).

KAW funded catalysis research project Chalmers University of Technology-Lund University: Funding for 2nd detector for XES (3 MSEK) and salary for a two year scientist position.

Chalmers University of Technology – MAX IV collaboration: Funding for a XRD detector and detector robot (3.8 MSEK) and salary for a two-year postdoc position.

Changes made since the start

The overall optical design for the beamline has been simplified without compromising the performance. Funding was instead used for development of the experimental station, including XES spectrometer, detectors, cryostat and sample environments.

Comparison to beamlines world wide

[CLAESS](#) at ALBA (Spain), [SAMBA](#) and [ROCK](#) at SOLEIL (France), and [I20](#) at Diamond Light Source (UK)

Future development

In addition to continued development of user friendly data analysis tools and general sample environments, the following developments are foreseen:

- Development of an X-ray emission spectrometer for energies above 25 keV.
- Continued design and manufacturing of sample environments, including reactor cells for catalysis and flow through cells for measurements in the tender X-ray energy range, 2.4-6 keV.
- Development and implementation of simultaneous IR/Raman and XAS/XES measurements. Development of an electrochemical sample cell for redox control of biological molecules. The development will be a collaboration between the Balder and CoSAXS beamlines and can be used for both XAS/XES and SAXS measurements.