

Short description of the laboratory SAXS-facility at the Graz University of Technology

Source and Cameras

The lab-SAXS facility consists of a sealed tube X-ray generator (DebyeFlex3000, with three opening ports and shutters where three SAXS cameras (A. Paar, Graz, Austria) are attached. Two of them are attached to the line-focus port, one to the point-focus port. All cameras can be operated at the same time and independently from each other. The sealed X-ray tube has a Cu-target emitting Cu-K_α-radiation ($\lambda = 0.154$ nm), and can be operated at a maximum power of 2000 Watt.

At each outlet the X-rays are monochromatized and focused in vertical direction (Multilayer-mirror optics) and then confined by a vertical asymmetric Kratky slit collimation-system by which the line-shaped X-ray beam can be collimated down to 50 μm , depending on the desired resolution towards small scattering angles (max. d-value ~ 100 nm). The adjustable horizontal slit can be opened up to 2 cm, or - by decreasing the slit-width - can be used to generate a point-shaped beam for GISAXS measurements. Source-sample distance and sample-detector are fixed and are 40 and 30 cm, respectively. The beam-path between mirror-exit and the detector with the sample stage in between is usually evacuated to avoid air scattering.

Resolution (accessible range of scattering angles)

SAXS-mode: (1D or 2D-detector) with an angular range from 0-8° (corresponding to d-values from 100 to 1 nm)

SWAXS-mode: (SAXS: 1D or 2D, WAXS: 1D) with an additional WAXS range from 17.5° to 35° (corresponding to d-values from 0.5 to 0.25 nm)

Continuous SWAXS-mode: with an image plate the entire angular range from 0°-35° is covered.

The WAXS-tubus attachment can be mounted on each camera

X-ray detectors

2D-Detector: CCD-detector (PI-SCX from Roper Scientific, Germany): 2084x2084 array with 24x24 μm pixels. Active area: 5 x 5 cm

1D-Diode array detector: Mythen 1K (Dectris, Switzerland), 1280 pixels a 50 μm . Active length: 6.4 cm

Image Plate Reader System: Cyclone (Perkin Elmer) with Software Optiquant. Image plates with dimensions SAXS: 65 x 60 mm and SWAXS: 65 x 200 mm, respectively (pixel resolution 50 x 50 μm) can be used.

Sample holders

Thermostatted sample holder stages (transmission mode): For individual measurements most sample holders (as well as detectors) are interchangeable on the 3 cameras.

Peltier heating/cooling controlled sample holder block in the range from -30 to 120°C (0.1° precision).

Resistance heater controlled sample holder block in the range from room temperature to 300°C.

Liquid samples are usually filled into a quartz cuvette holder, which contains a re-useable quartz capillary (1 mm diameter, 10 µm wall thickness) with sealable screw-caps on both ends.

Solid samples, powders or very viscous samples can be filled in a special paste-and powder holder where the sample is sandwiched between two foils and sealed vacuum-tight.

Both sample holders can be inserted into the two respective sample holder blocks for temperature control.

GI-SAXS sample stage (GISAXS-mode): GISAXS samples can be rotated in the X-axis (ω , normal to the X-ray beam) and rotated in the axis normal to the surface (φ). The maximum rotation angles are $-8^\circ < \omega < 5^\circ$ and $0^\circ < \varphi < 360^\circ$. The minimum step sizes are 0.0005° in ω and in φ . The maximum sample dimensions are (length x width x thickness) 21 x 21 x 4 mm.

Software

SAXSquant (A. Paar, Graz, Austria) is used as the comprehensive control/data-acquisition/data-evaluation software for automated control of the camera system components, for automated measurements and for producing scattering patterns (2D) and curves (1D) from detector data. It is designed to perform raw-data processing (data normalization, background subtraction, q-scale calibration, desmearing).