BLiX
Berlin Laboratory for innovative X-ray Technologies

Concepts and Instrumentation for Scanning free X-ray Emission Spectroscopy

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Outline

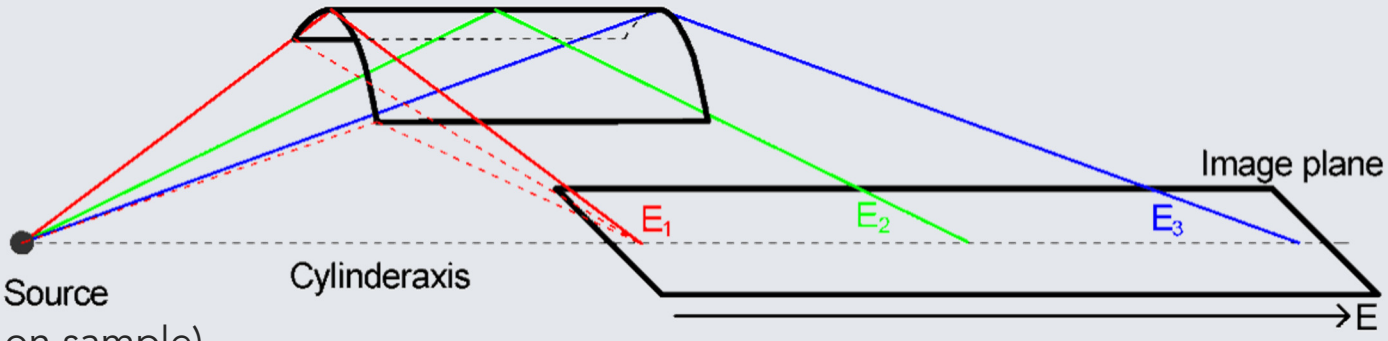
- Theory
  - Principle of Von Hamos geometry
- Spectrometer at PINK Beamline (Bessy II)
- Spectrometer in the lab
  - Motivation
  - HAPG Optic
  - Full cylinder
  - Components and mechanics
  - Alignment
  - Stability/ Performance/ Resolution
- Conclusion

Theory – Principle of Von Hamos Geometry

- due to Bragg's law, wavelength information is converted into spatial information
- offers the possibility of high resolution X-ray spectroscopy
- scanning free method

$$n\lambda = 2d \sin \theta_B$$

sagittal bent reflective optic

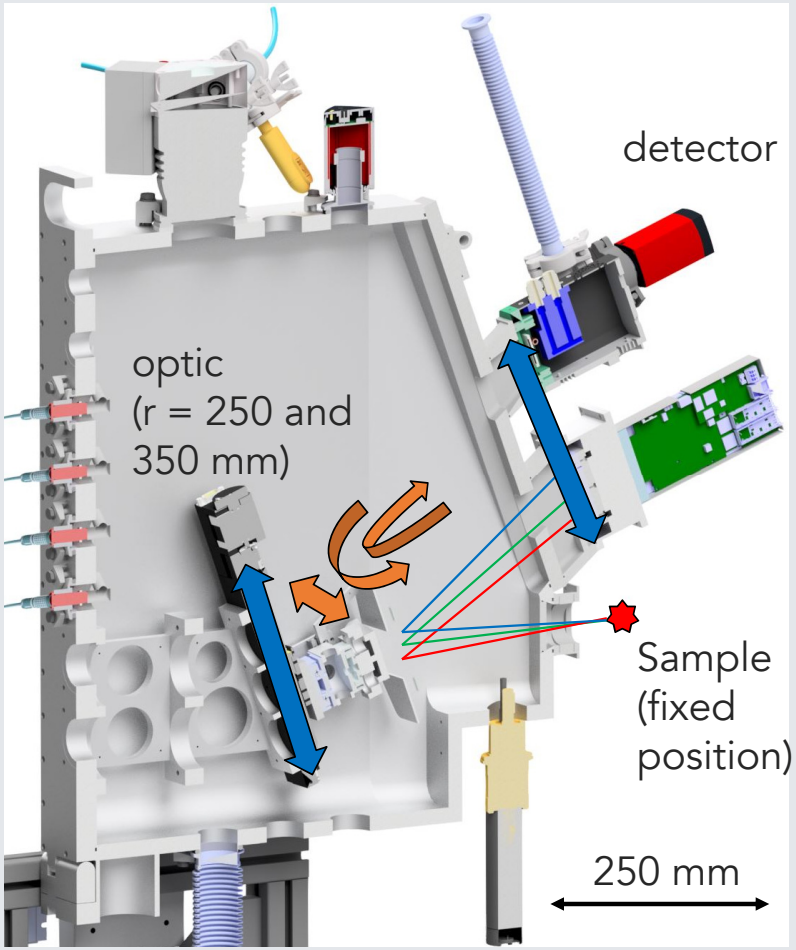


(spot on sample)

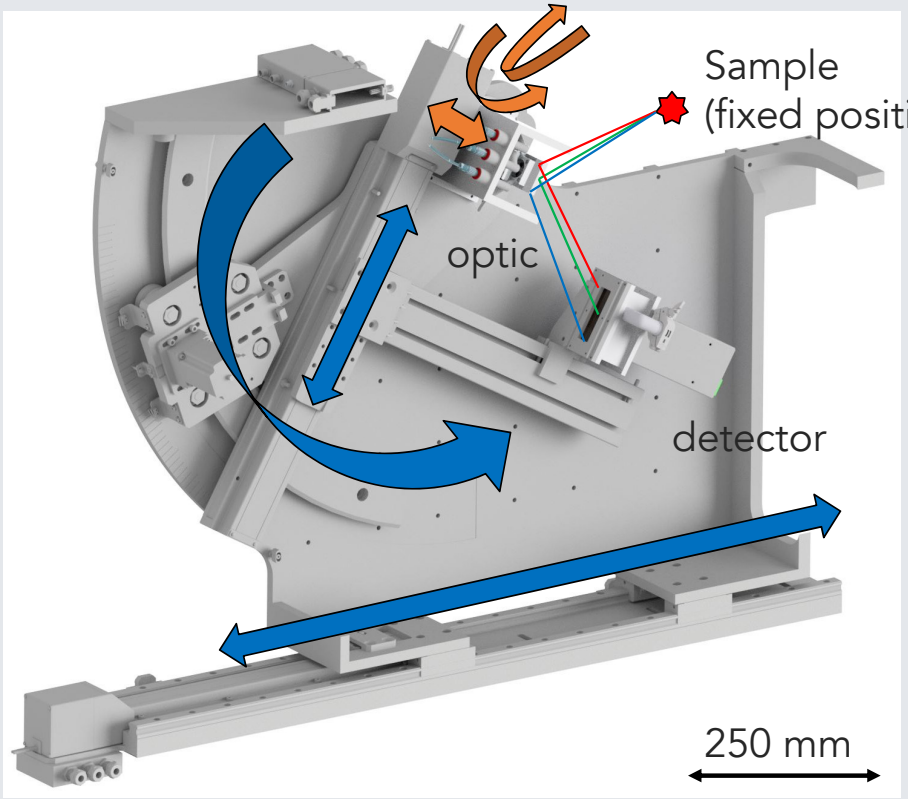
Von Hamos Spectrometers at PINK Beamline (BESSY II)

- different single crystals for different elements (energies, 2 …10 keV)

in vacuum

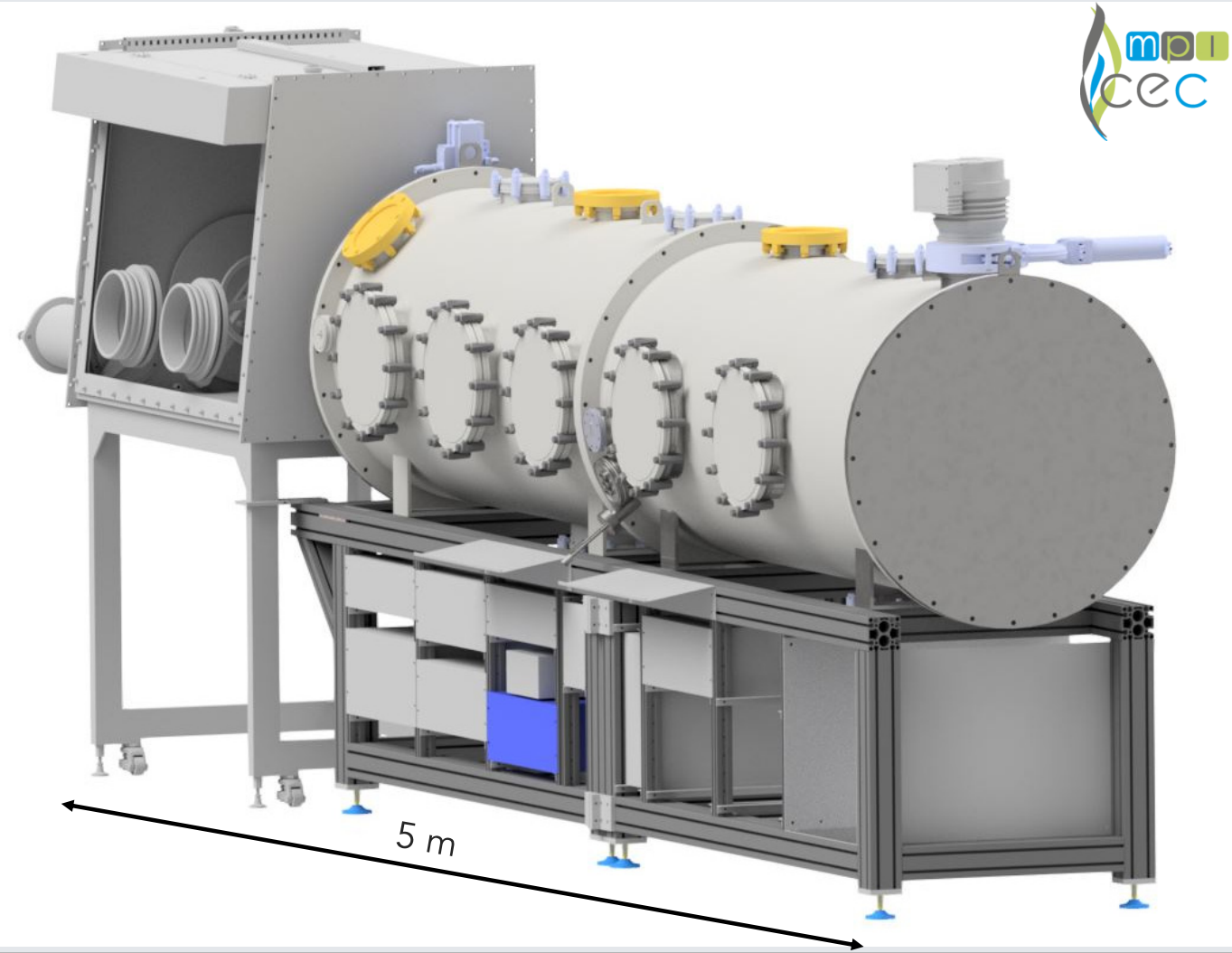


atmospheric



Von Hamos Spectrometers in the Lab at MPI CEC

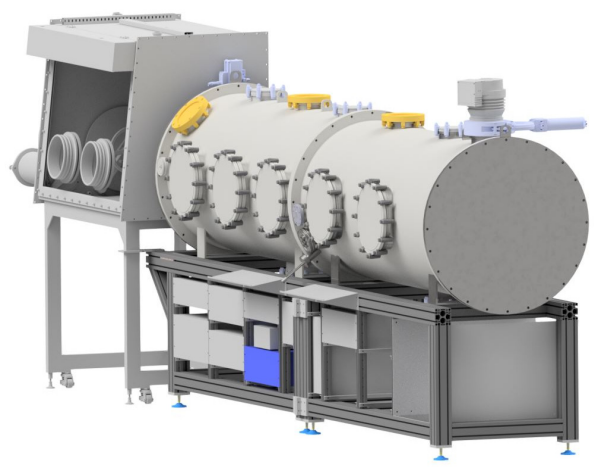
- Von Hamos spectrometer with full cylinder HAPG optic



Motivation for Von Hamos full cylinder Lab Spectrometer

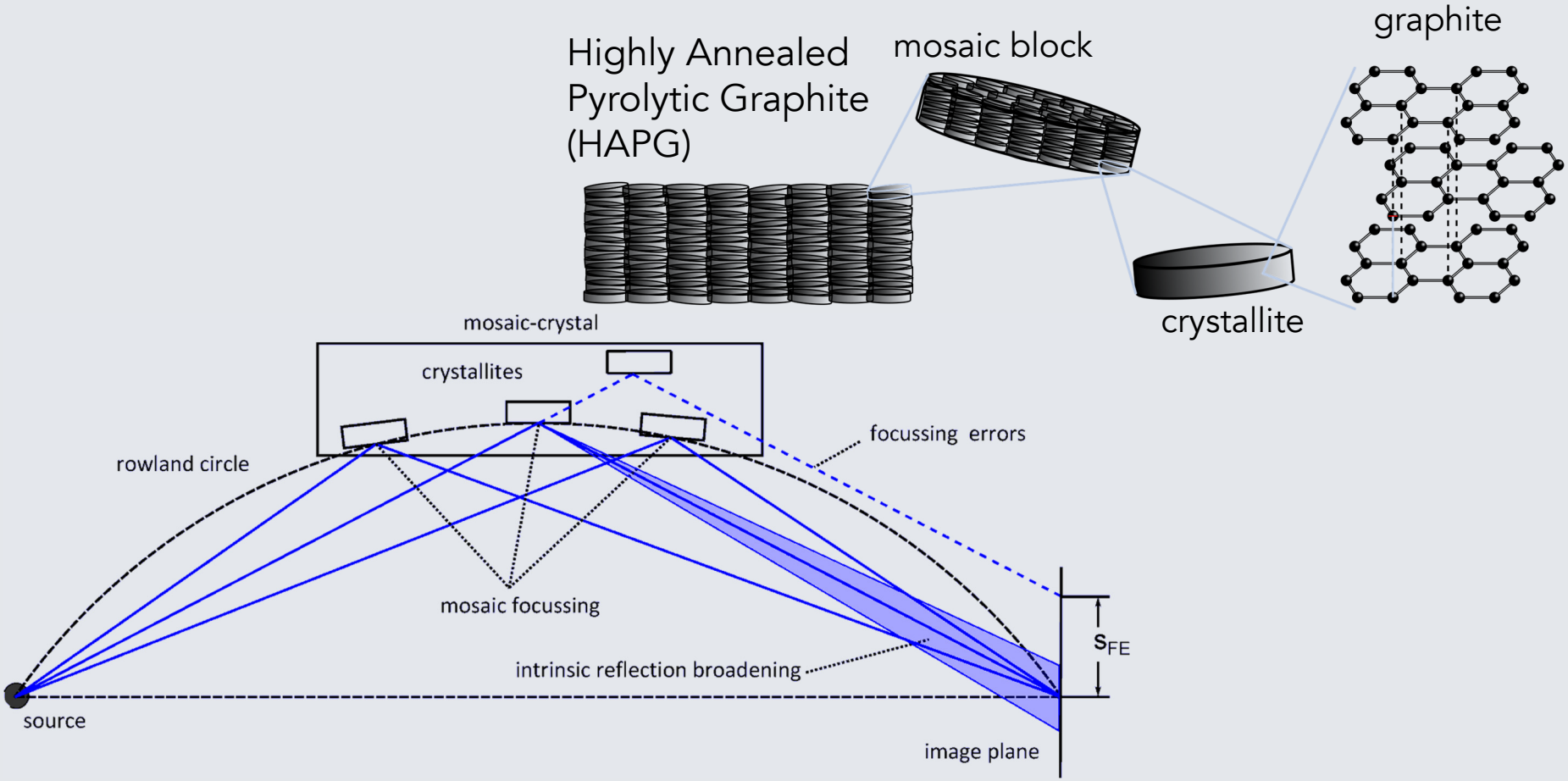
- tool for standard measurements
- spectroscopy parallel to chemistry
- 24/7 beam time next to your research lab

- stable and easy to align spectrometer
  - scanning free measurement
  - easy mechanics
  - less degrees of freedom
- reference samples are available for energy calibration
- from tests and simulations expected resolving power of $E/\Delta E \approx 4000$ is useful for XES
- O_2-free sample preparation and environment (cryo cooling)
- measurement time in the range of hours – compensate low beam intensity of lab source



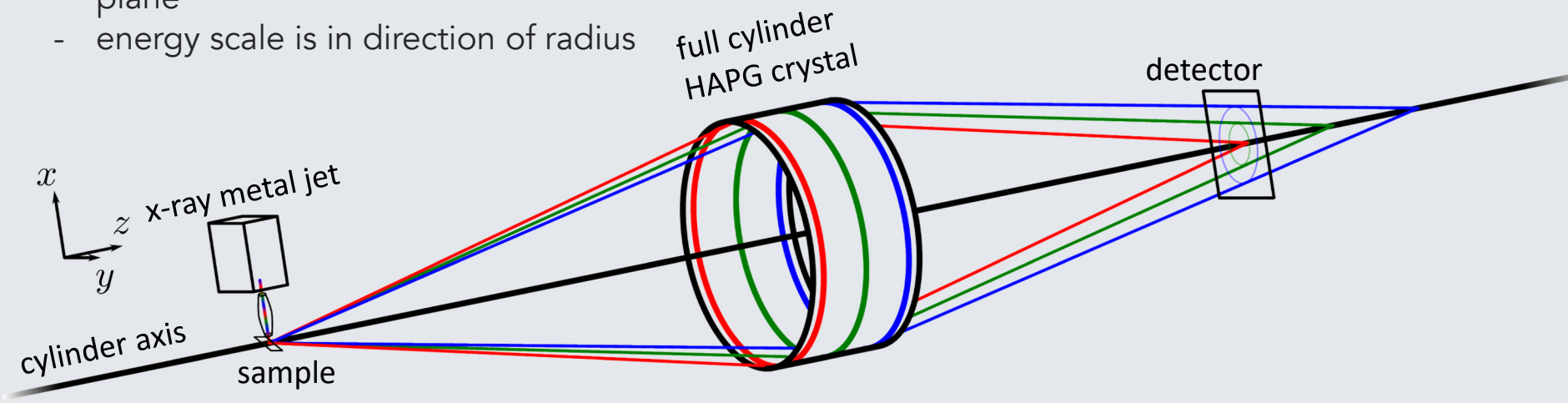
Mosaic Crystals in X-ray spectrometers

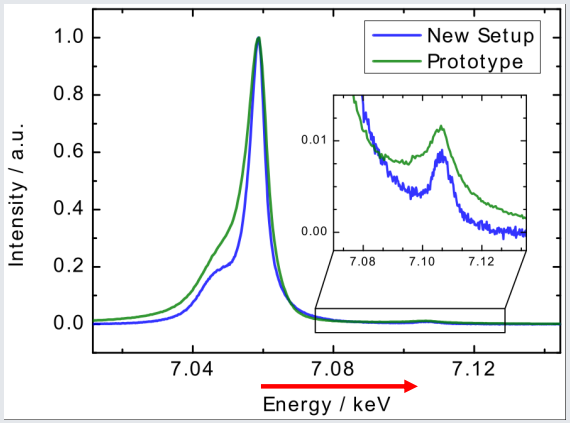
- mosaic spread is about 0,05° -> makes the alignment less sensitive, but also energy resolution less accurate
- 20 to 30 times more intensity of reflected beam compared to single crystal material -> reduces measurement time rapidly

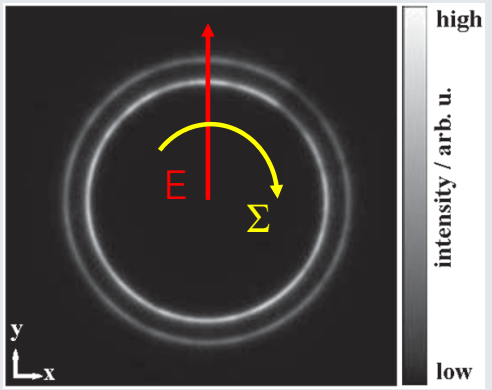


Full Cylinder HAPG Optic

- enlarge the optic to a full cylinder -> increase solid angle of detection -> reduce measurement time
- necessary: changing orientation of detector –> gives rings on the image plane
- energy scale is in direction of radius

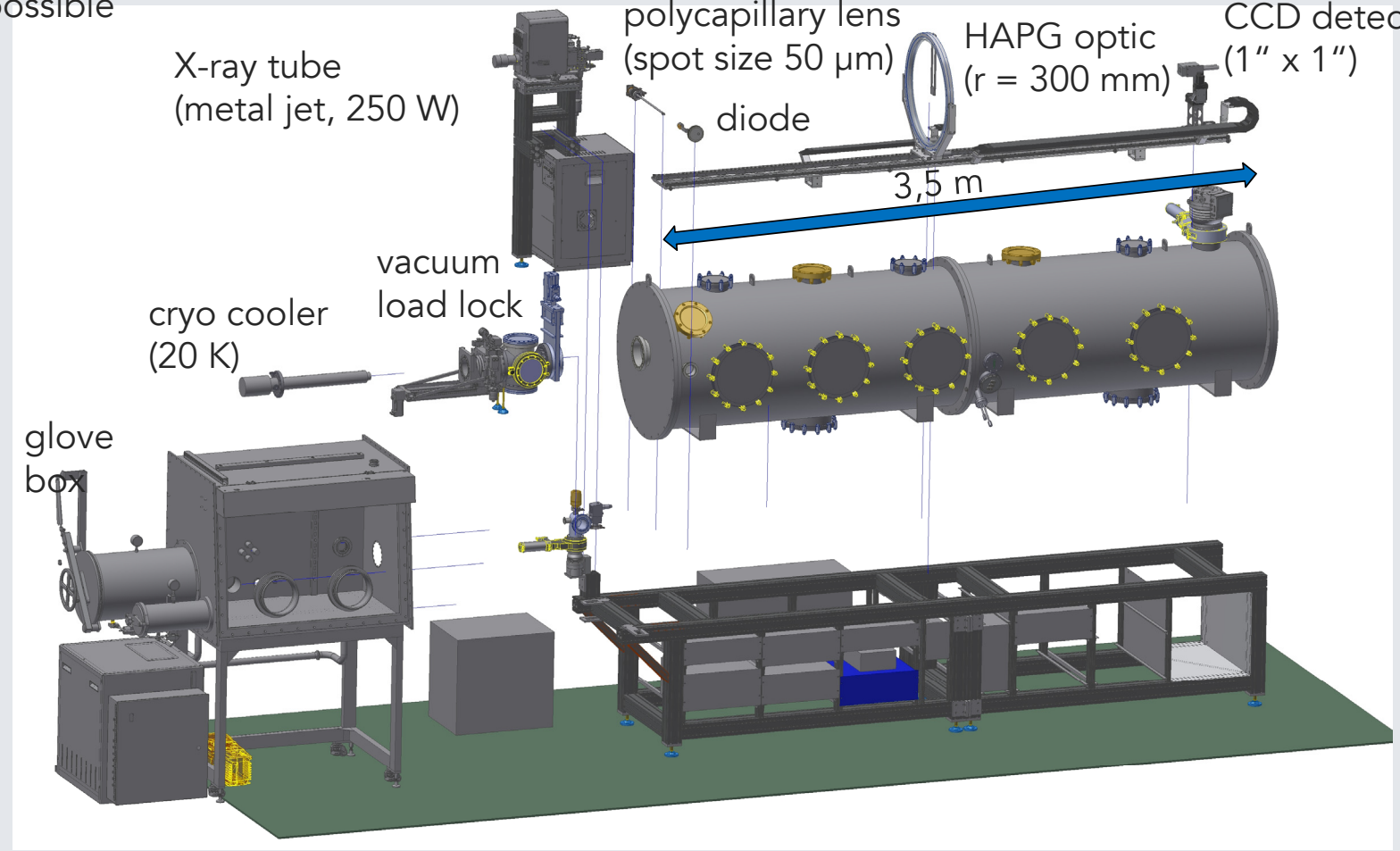




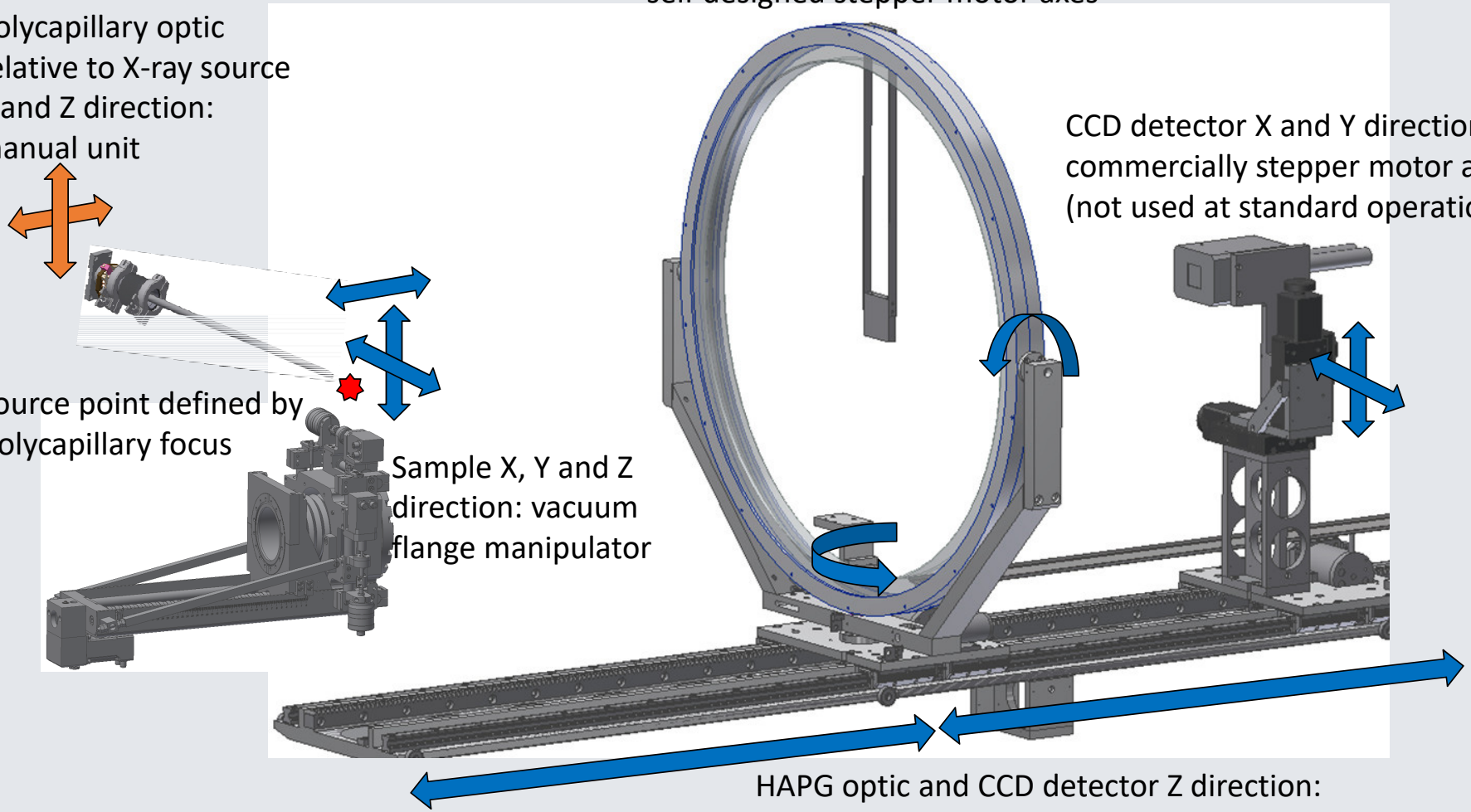


Full Cylinder HAPG Spectrometer

- developed at BLiX Berlin Laboratory for innovative X-ray Technologies situated at mpi cec
- used in laboratory with a metal jet X-ray source as excitation, but synchrotron excitation is possible

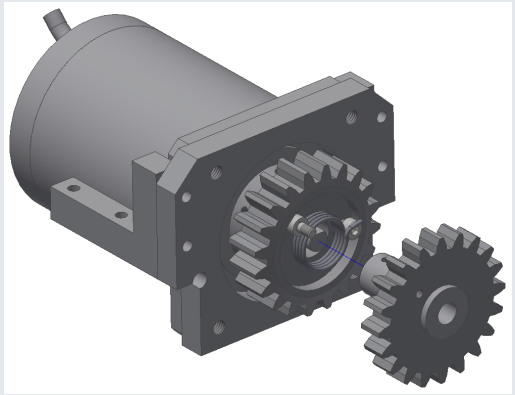


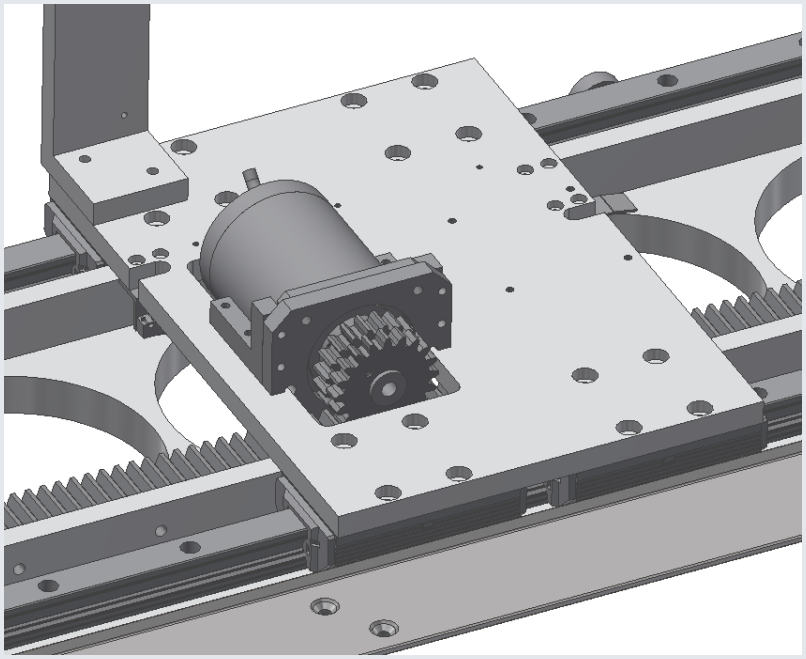
Mechanics of Full Cylinder HAPG Spectrometer



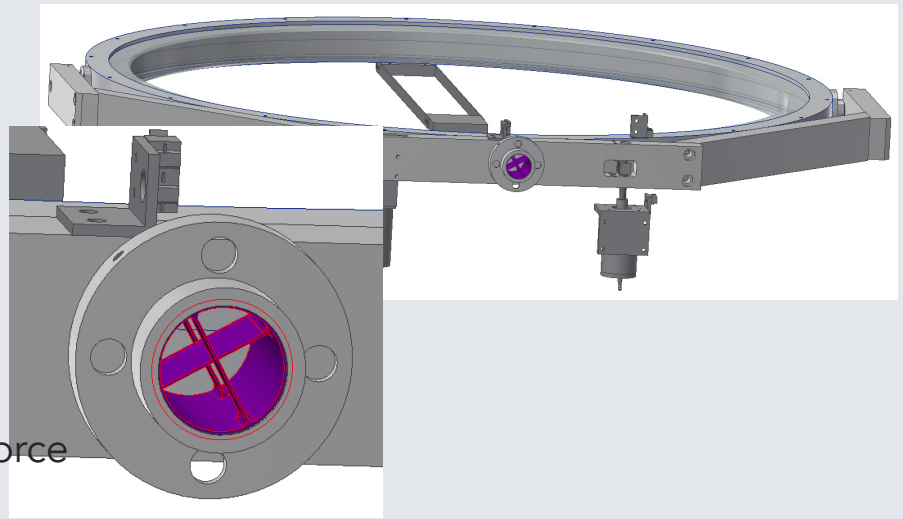
Mechanics of HAPG Full Cylinder Optic

- z direction (used also for CCD detector)
  - simple teeth bar and –wheel
  - two parted teeth wheel to reduce gap





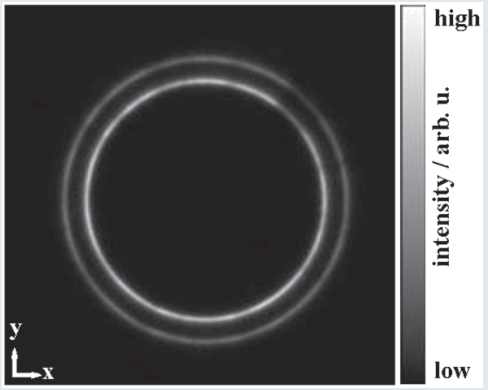
- α and β rotation motorized with M6 thread
- rotation bearings are cross spring joints
  - gap free
  - always preloaded by mounting with offset to eliminate gap of M6 thread

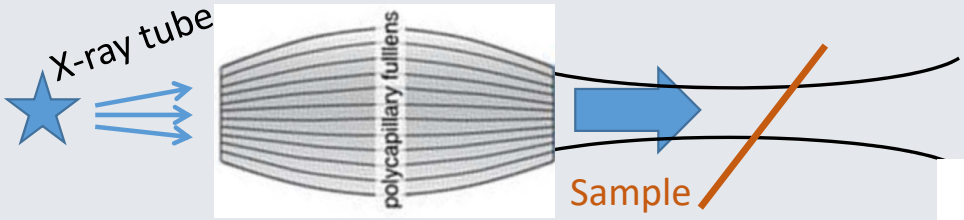


- sample manipulator is preloaded by pressure force

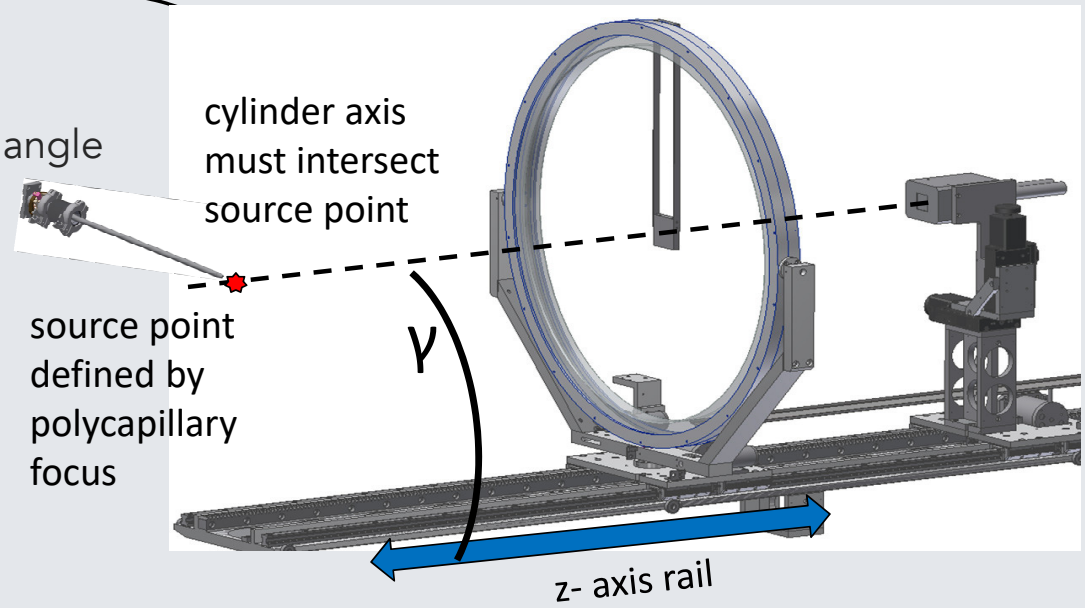
Alignment of Full Cylinder HAPG Spectrometer

- polycapillary to X-ray tube focus – signal from diode
  - stable manual unit
  - realignment just in case of X-ray tube maintenance (2…3 month)
- all alignment of spectrometer with signal from CCD
- sample to the focal spot of the polycapillary



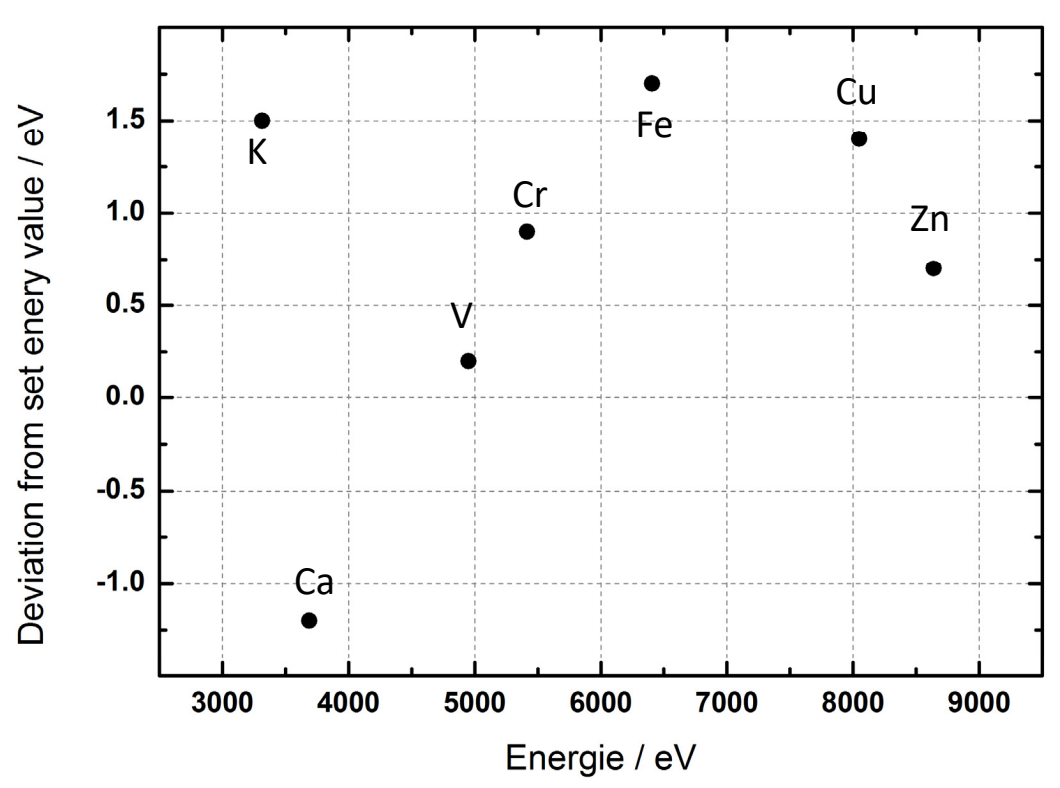


- HAPG optic in z direction and a, b angle
- CCD detector to correct position
- correct tilt of z-rail with rotation of optic for each energy



Stability of Full Cylinder HAPG Spectrometer

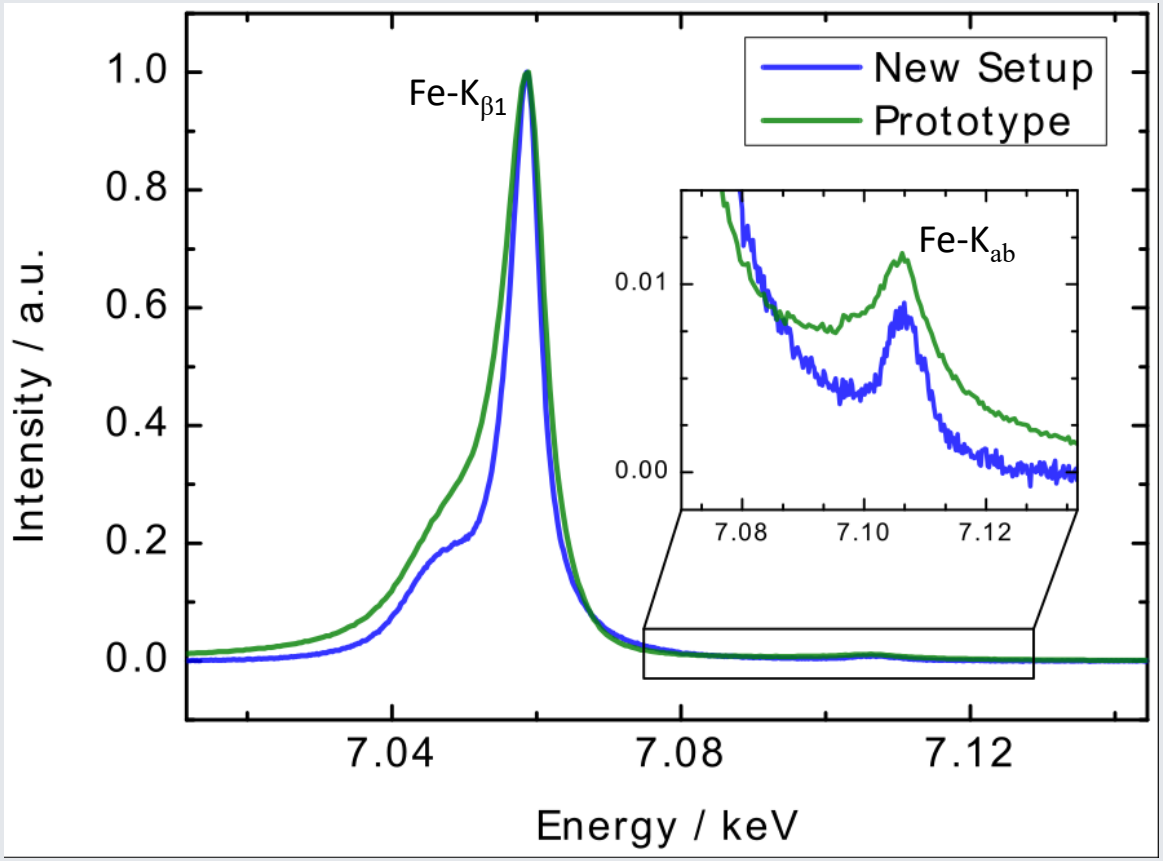
- place all components just by calculated positions
- possible range 2 to 10 keV, first order of reflection



Stability of Full Cylinder HAPG Spectrometer

- measure reference sample to define exact energy scale
- change from reference to sample
- HAPG and CCD are fixed
- sample align with respect to the center of the ring on CCD
- standard deviation for some elements:

Element	Std. Dev. / eV
Cu Solid	0,013
Cu powder	0,022
Cr	0,050
K (Ka1)	0,023



Conclusion

- high resolution, scanning free X-ray emission spectroscopy is possible in the lab with moderate measuring times (several hours)
- the alignment is easy and fast with stepper motor axes, no encoders needed
- measurement of reference sample is in principle „push button“ (no alignment)
- fast and reproducible sample change (e.g. reference to real sample or a set of samples)
- energy resolution of $E/\Delta E \approx 4000$ is as expected
- it’s stable over hours and days
  - concept of pre forced axes is working well
- powerful lab tool for daily use since 2 years of operation

Thank you for your attention

BLiX Berlin Laboratory for innovative X-ray Technologies — Sven-Uwe Urban

mpi cec — Fabian Kowalewski

Ina Holfelder WEOPMA06:
A compact and calibratable von Hamos X-Ray Spectrometer based on two full-cylinder HAPG mosaic crystals for high-resolution XES