

Multimodal X-ray Tomography and auxiliary equipments at SAXSMAT beamline

Dr. Andre L. C. Conceição

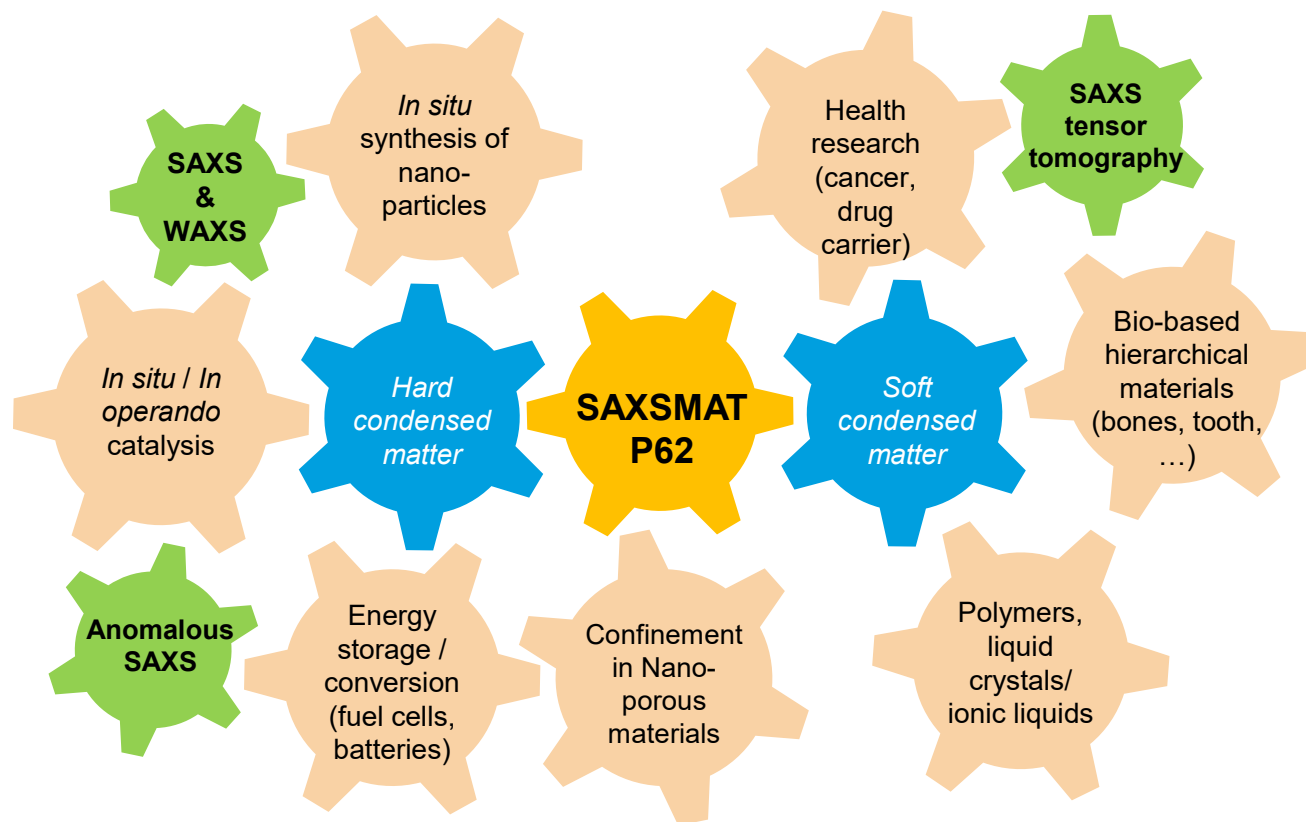
SAXSMAT beamline P62

Paul P. Ewald Hall (Hall North)



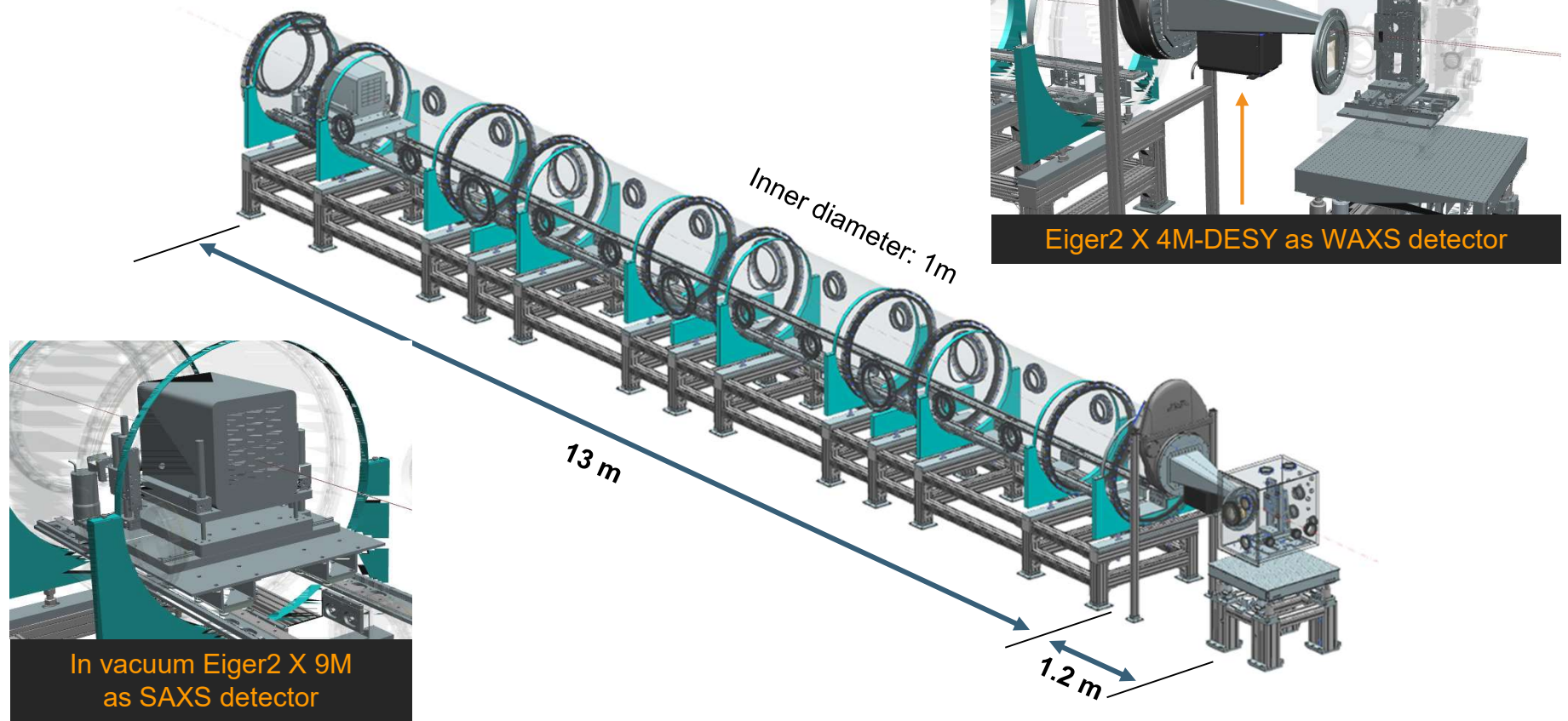
Science @SAXSMAT beamline

Key research activities and methods



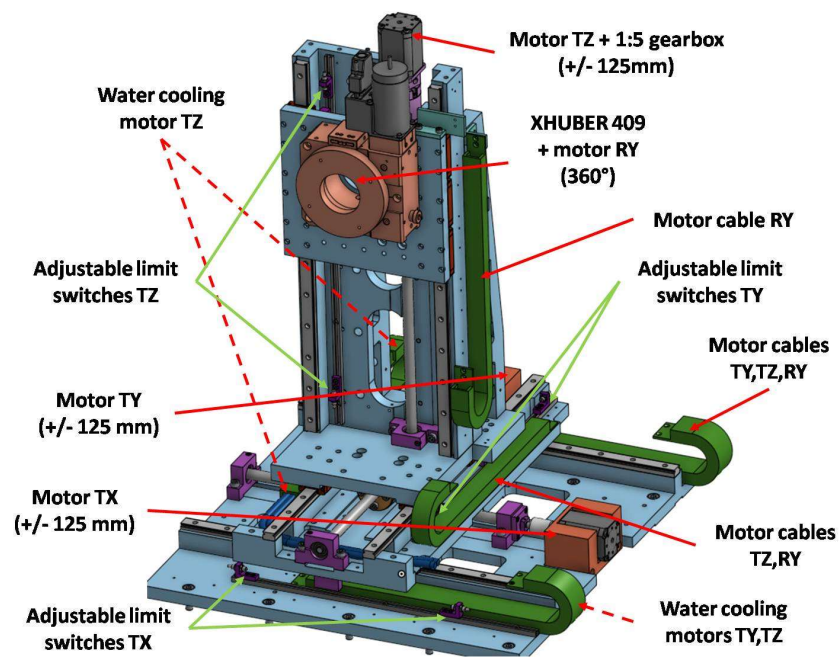
Experimental hutch

SAXS Instrument

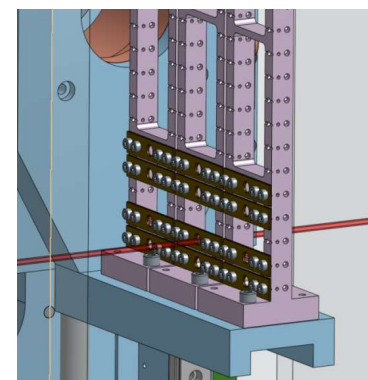
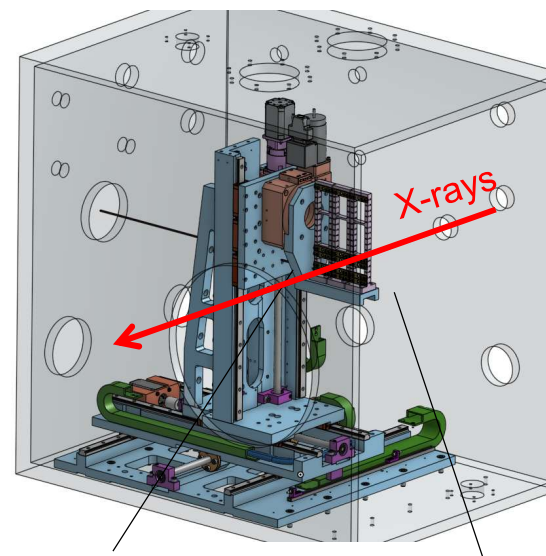


Sample environment

Standard sample environment in air or sample chamber in vacuum

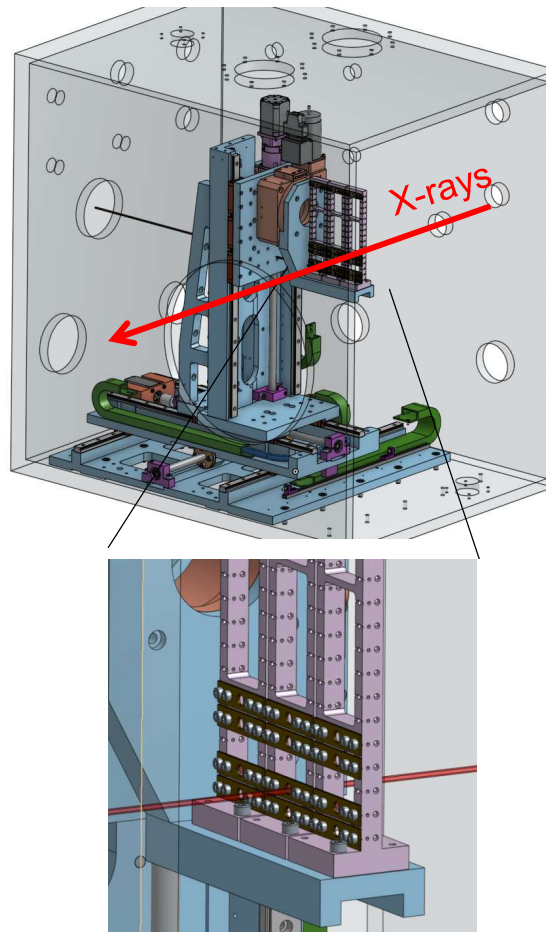


Standard coarse motion sample positioning tower



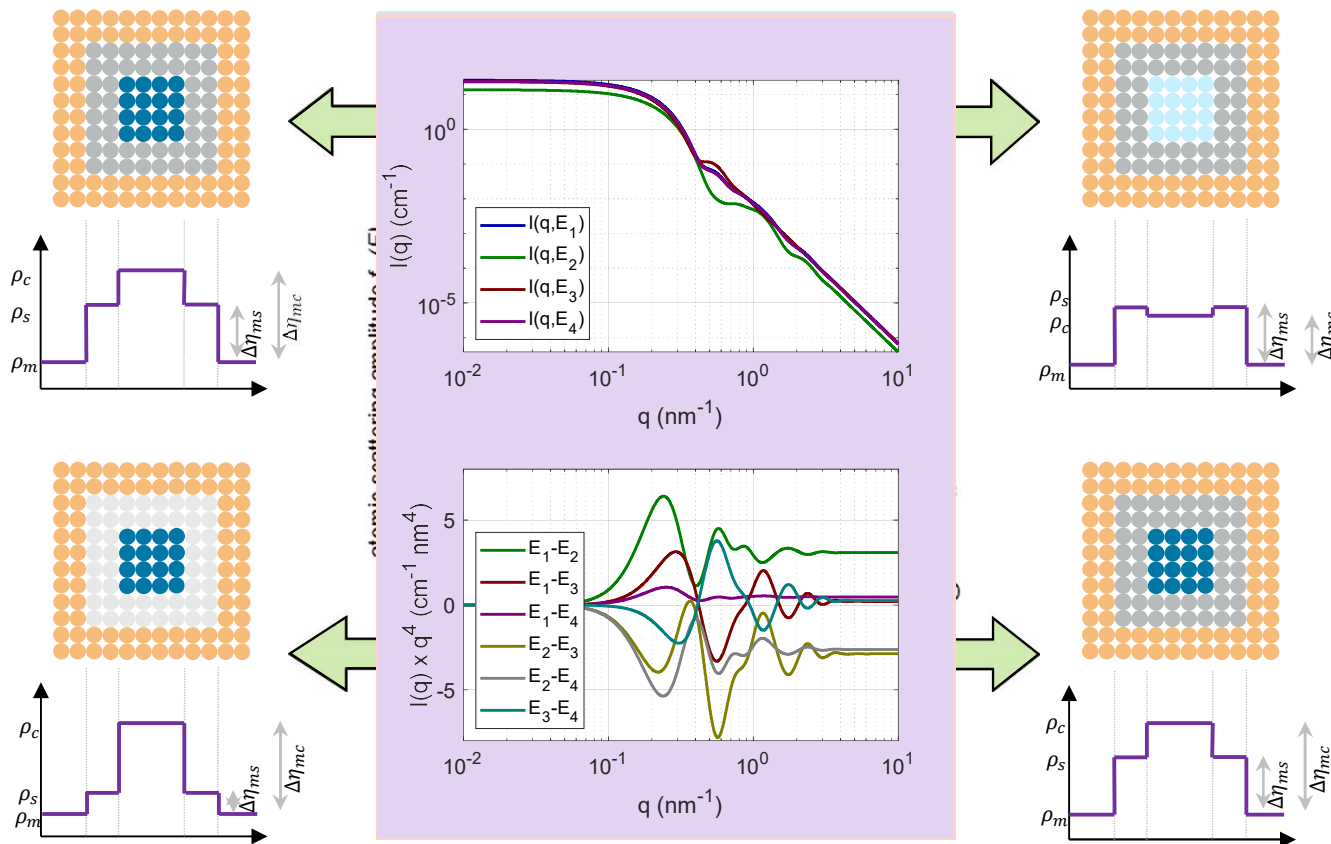
Techniques @SAXSMAT

High-throughput SAXS/WAXS



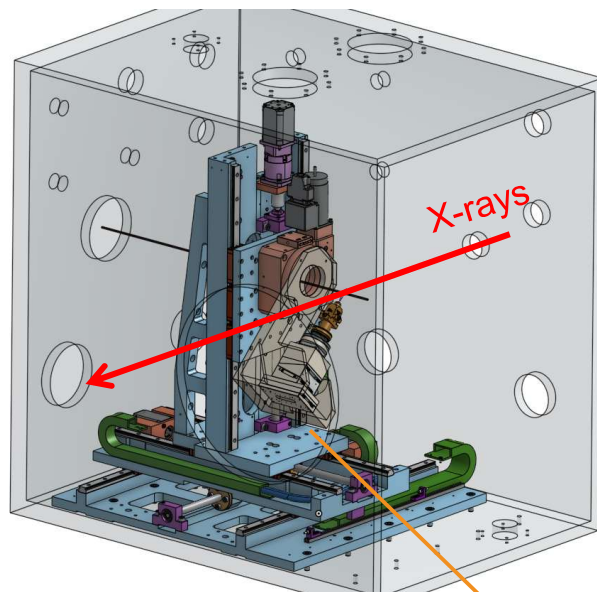
Techniques at SAXSMAT

Anomalous SAXS

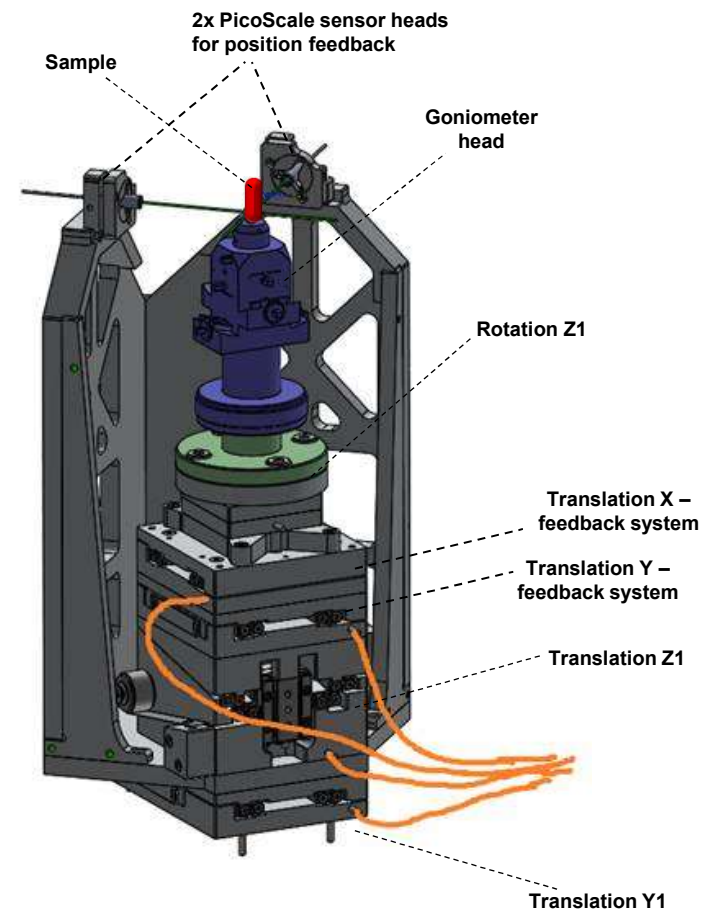


Techniques @SAXSMAT

SAXS/WAXS Tensor tomography

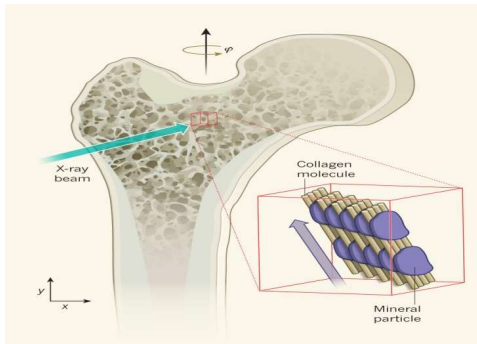
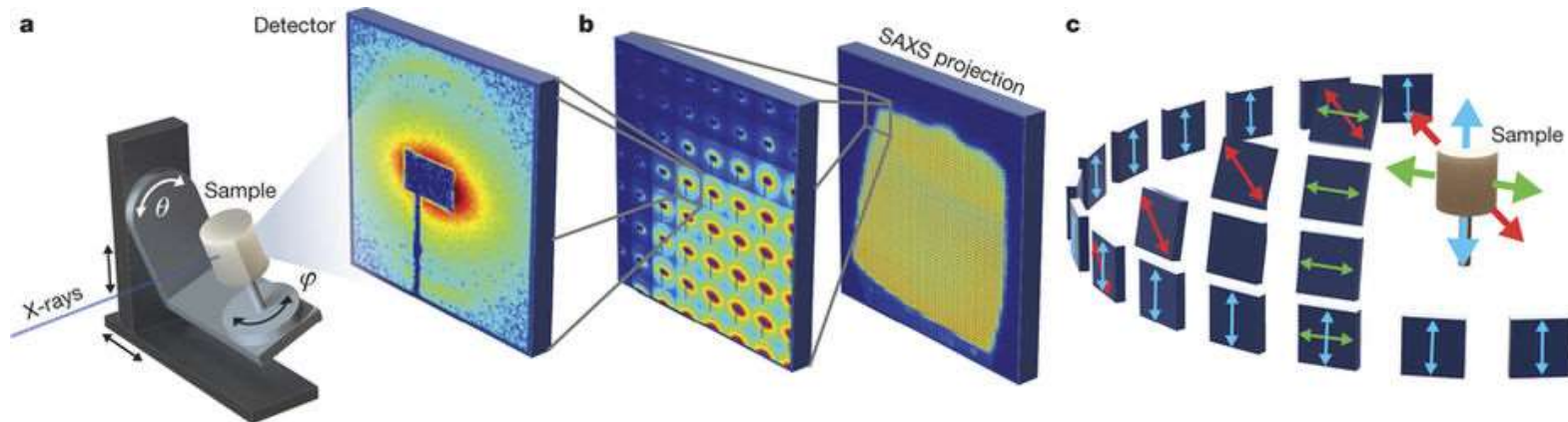


System is build by SmarAct.
Delivery is planned for end of May

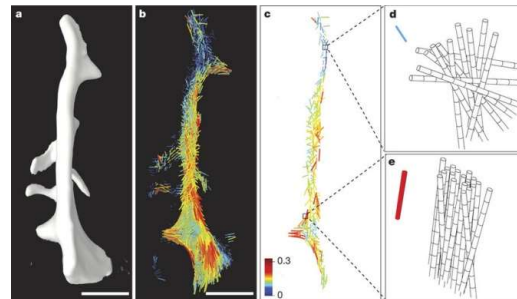


Techniques @SAXSMAT

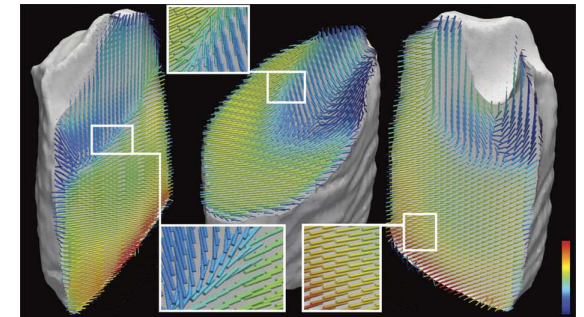
SAXS tensor tomography / SAXS computed tomography



P. Fratzl *et al.* Nature, 527: 308-309, 2015



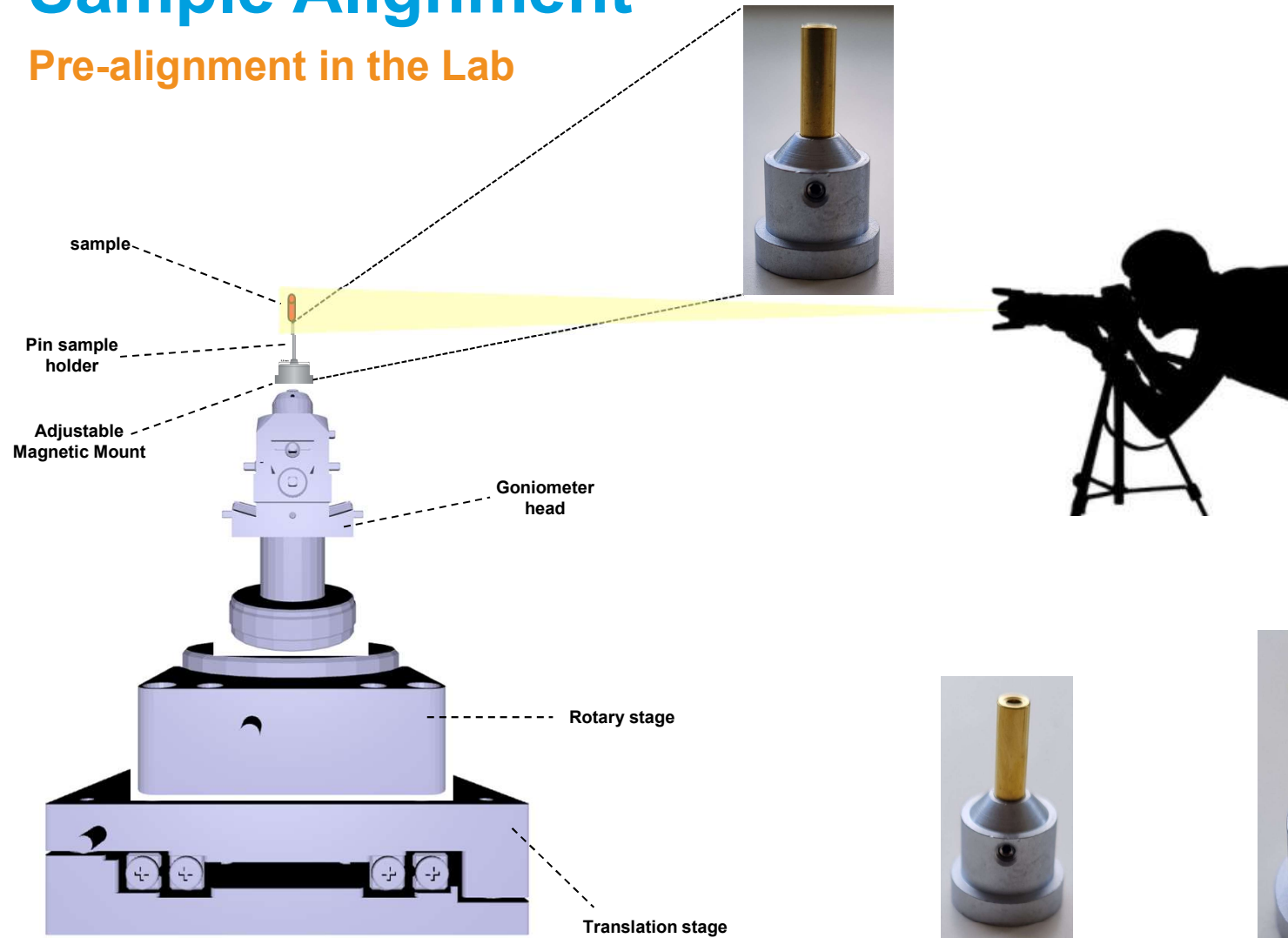
M. Liebi *et al.* Nature, 527: 349-353, 2015



F. Schaff *et al.* Nature, 527: 353-356, 2015

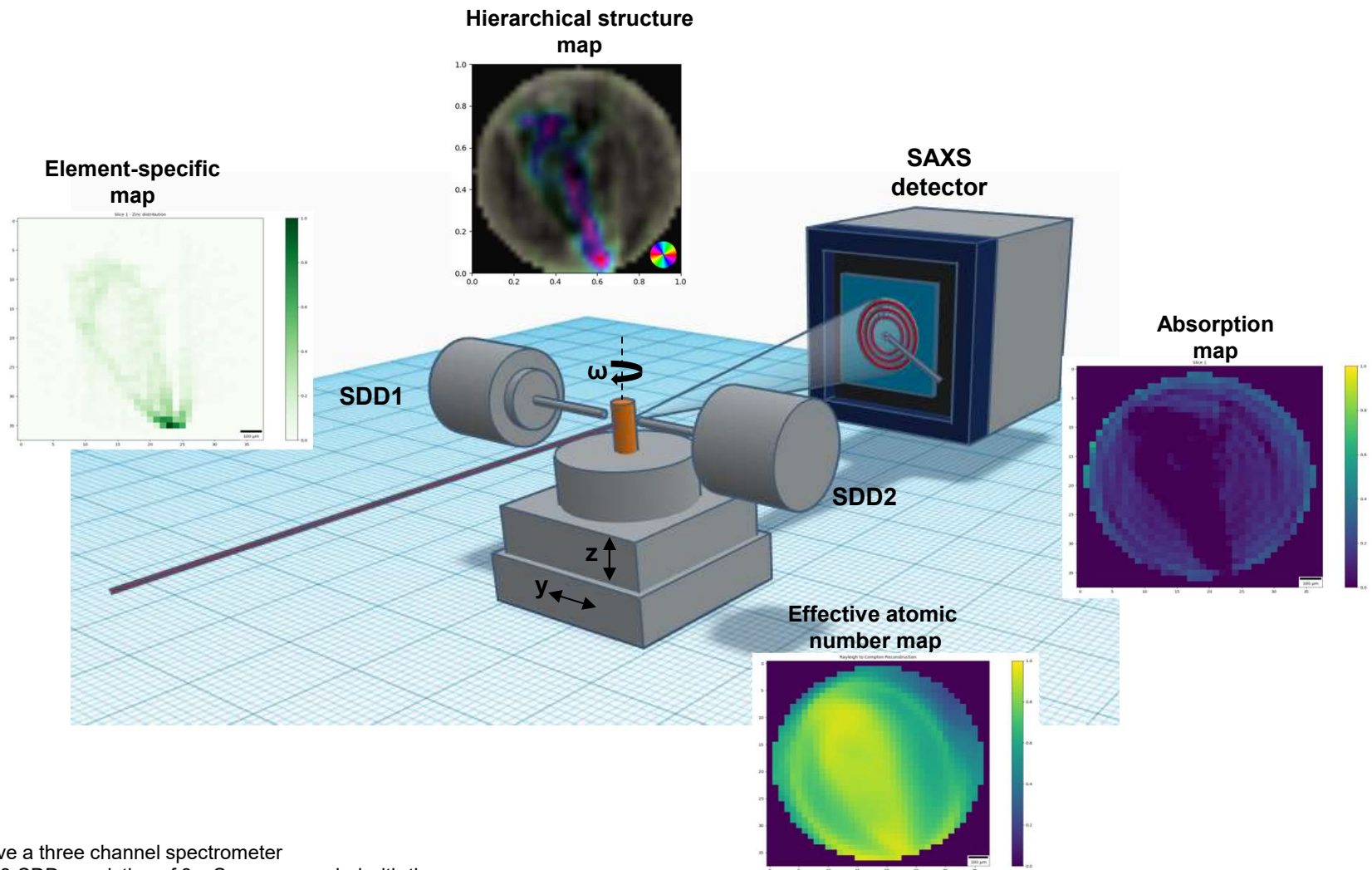
Sample Alignment

Pre-alignment in the Lab



X-ray Scattering and Emission Tomography

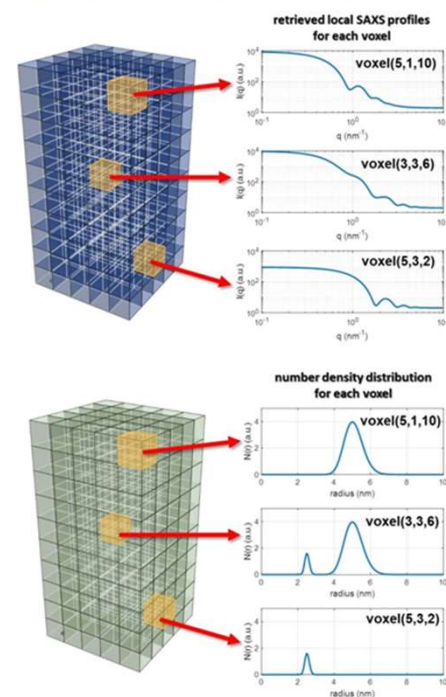
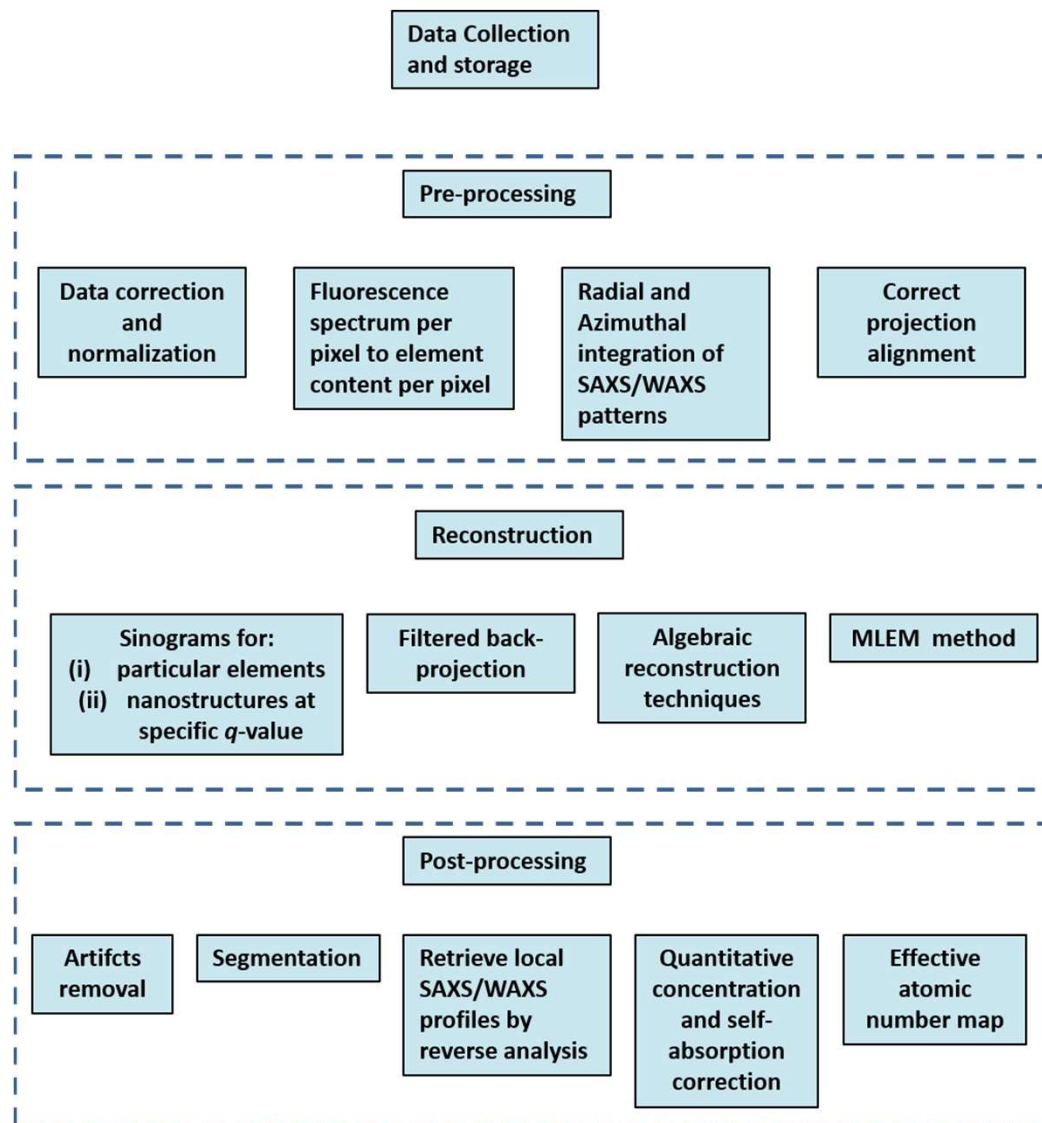
3D elemental-specific, electron density and hierarchical structure map



* Plan to have a three channel spectrometer
Vortex@ME3 SDD consisting of 3 x Sensors coupled with the
Readout system Xspress 3-channel

Ongoing pipeline for Multimode Tomography

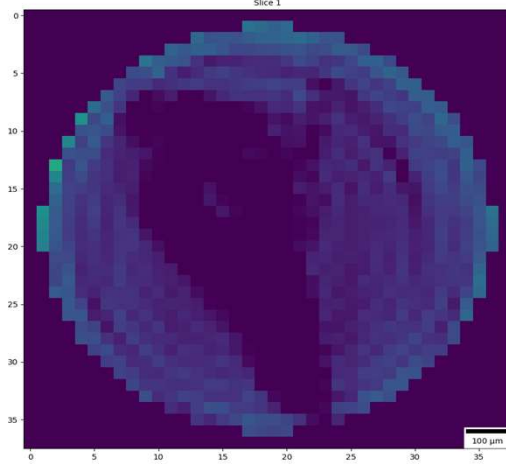
SAXS/WAXS/XRF/Compton-CT pipeline for on-line reconstruction



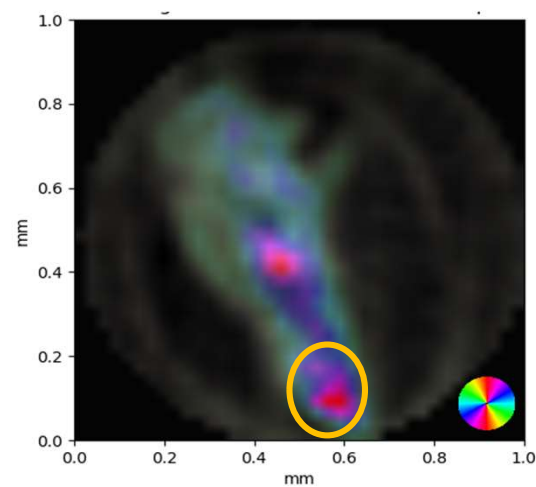
3D Multimodal X-ray Imaging

Correlation between collagen rearrangement and Fe, Cu and Zn accumulation

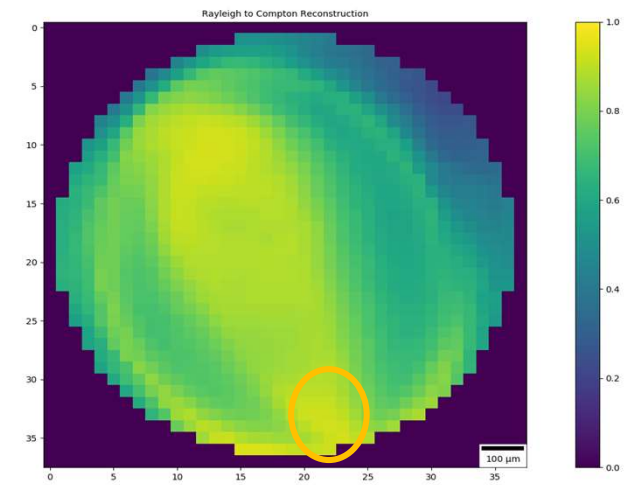
Absorption
contrast



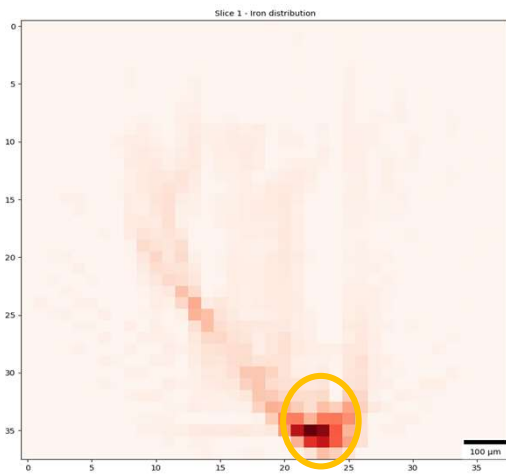
Collagen content and orientation



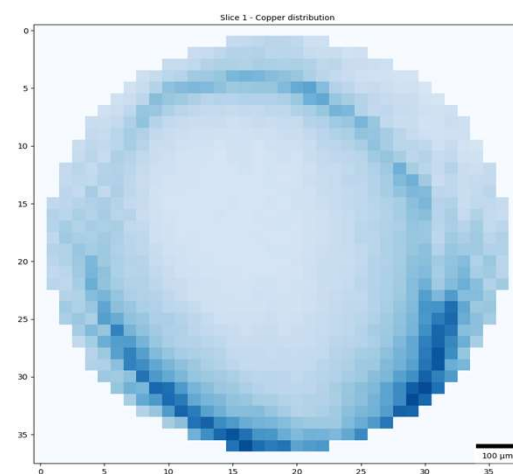
R/C tomogram



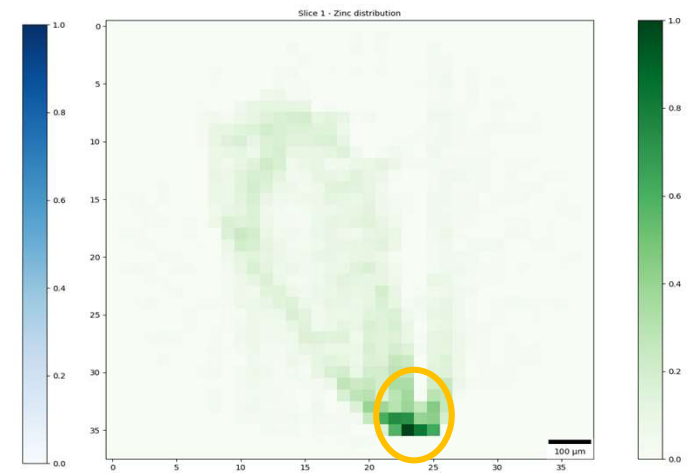
Fe distribution



Cu distribution

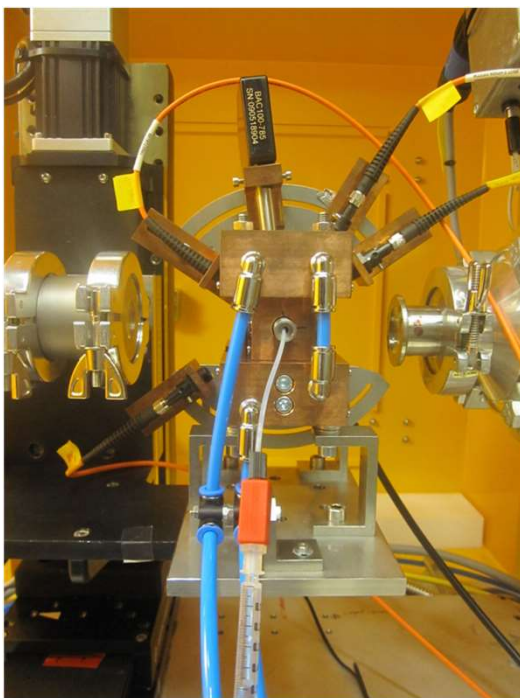


Zn distribution

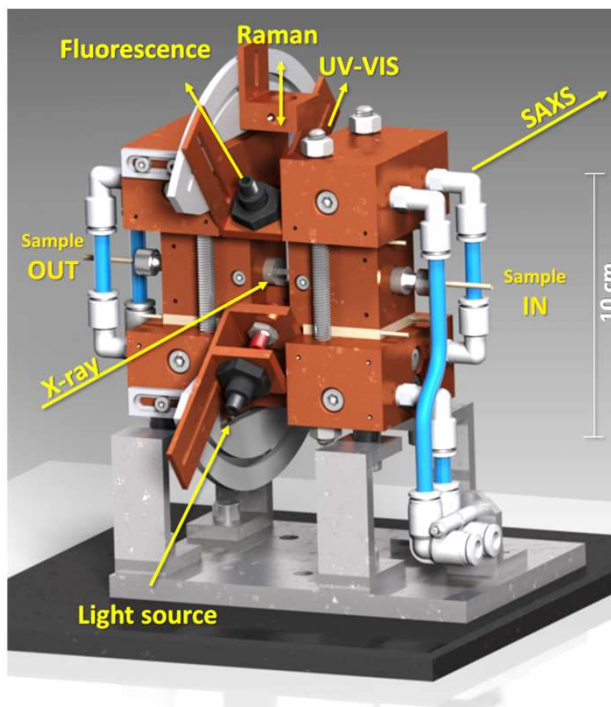


Multiprobe @ SAXSMAT

in situ / operando SAXS & WAXS



S. Haas et al., *Phys. Chem. B.* 118(8), (2014), 2264-2273



UV-VIS spectroscopy	
Source 1:	100 Watt Xe Arc lamp
Source 2:	78 Watt Deuterium / 5 Watt Halogen lamp
Detector:	Oceans Optics – HR4000 200 nm – 1100 nm
Integration time:	3 ms - several minutes
Raman spectroscopy	
Source:	785 nm laser / 50 – 350 mW
Detector:	B&W Tek Inc. – i-Raman 175 – 3200 cm^{-1} (3 cm^{-1})
Fluorescence spectroscopy	
Source:	100 Watt Xe Arc lamp
Monochromator:	Newport Cronerstone 130 1/8 m
Gratings:	1.) 185 nm – 650 nm , 250 blaze 2.) 200 nm – 1600 nm, 350 blaze
Detector:	Oceans Optics – QE 65 Pro 200 nm – 1100 nm
Integration time:	3 ms - several minutes
Temperature	
Active Heat/Cooling:	4 TEM modules (33 W each)
Heat sink:	Thermostat controlled water bath
Temperature range:	-50 °C – +150°C
Heat/Cooling rate:	max ~ 0.2 K/s

*RAC proposal submitted: low frequency Raman, humidity controller

Freeze-dryer

Sample preparation and “*in situ*” dehydration experiments



VACO 2

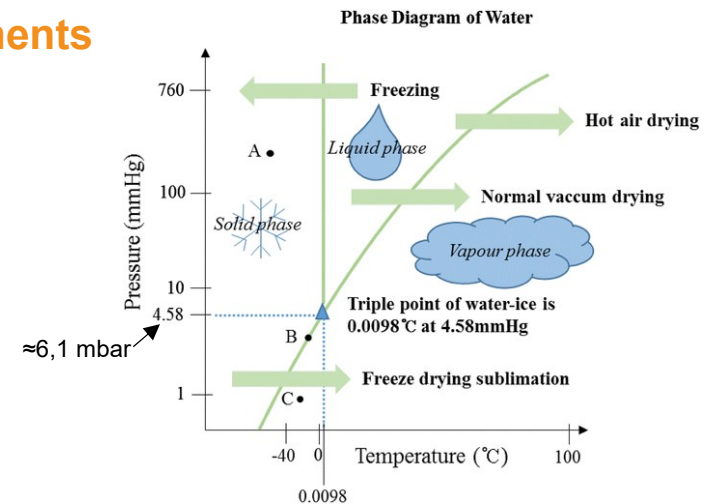
Two-stage cooling system

Capacitor volumes	5,7 Liters
Condensor temperature	< -80°C
Condensor capacity	2kg / 24h
Max. ice capacity	3kg
Max. vacuum	2×10^{-3} mbar

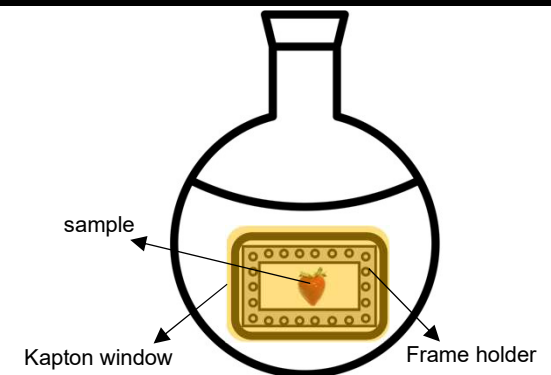
Paraffin-Embedded



Freeze-dried



Sample environment for
in-situ freeze-drying
experiments



Control:

- cooling rate
- heating rate
- vacuum

VIA @SAXSMAT

Summary

- VIA is an interesting initiative which will be very helpful to all beamlines, in particular to the SAXSMAT beamline where multimode experiments are planned.
- So far, we have only the freeze-dryer to share.
- We hope in the near future to be contribute with other devices such as the SAXS/UV–vis/Raman multiprobe system.

Acknowledgements

SAXSMAT P62 team

