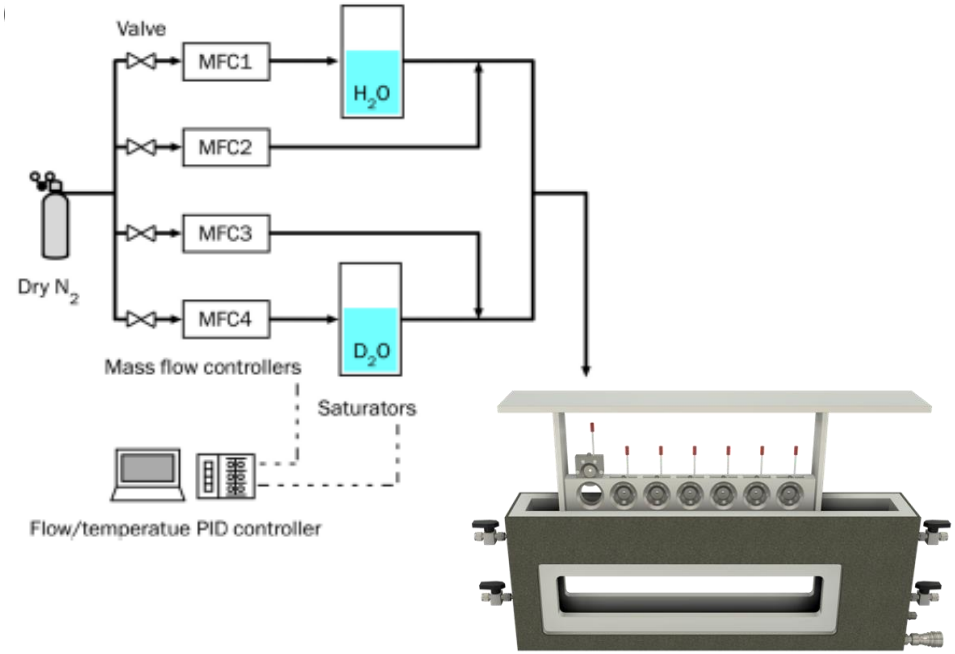
Current developments and future wishes for soft matter sample environment

Henrich Frielinghaus

(27.11.2023)

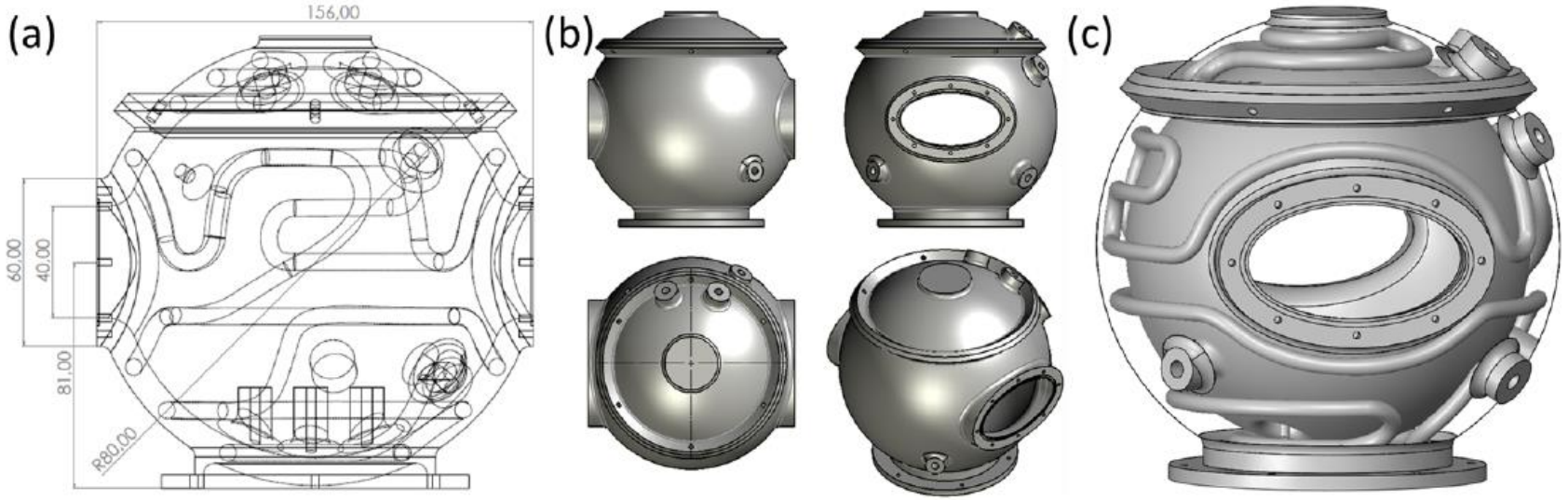
on behalf of the soft matter group

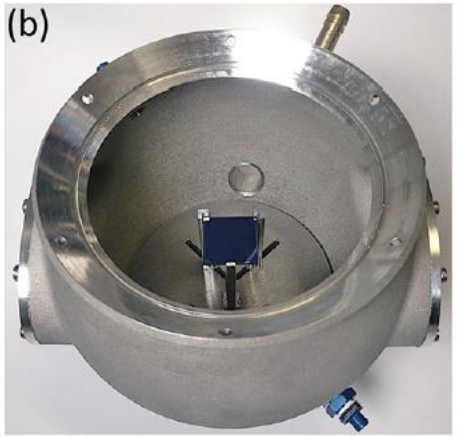
Humidity generator at JCNS

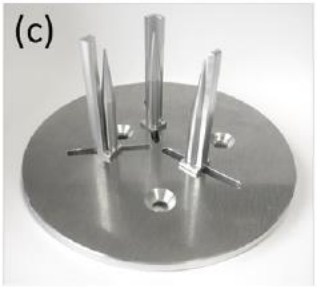


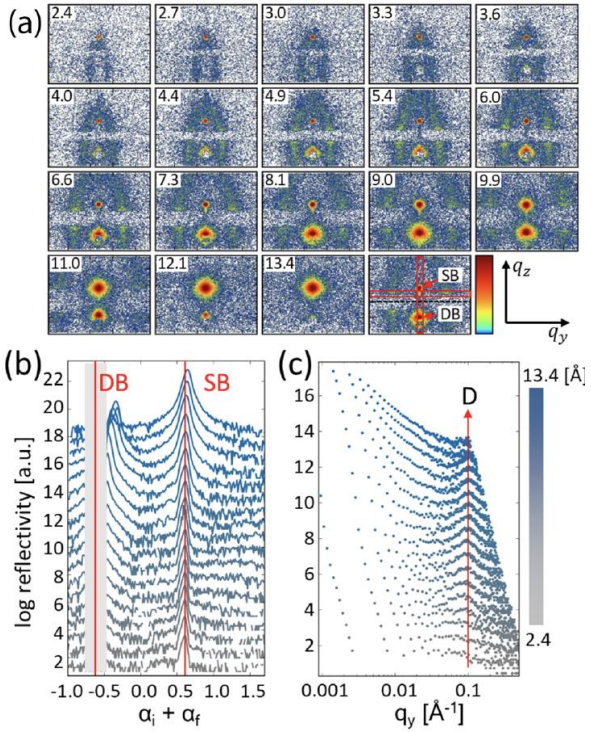
- for the investigation of hydration of polymer and biological membranes: ordered from TEKHNE Corporation & MicroEquipment Inc, Tokyo, Japan, expected delivery March-April
- fast achievement and stable RH (10-85 %) and T (10-85 oC)
- versatile H2O/D2O vapor supply, two tanks, suitable for in-situ change of neutron contrast
- works according to the two-temperature principle (T reservoir and T sample)
- controlled by Nicos
- multiposition sample chamber (SANS) with conductivity measurement option, in planning and production (PGI/JCNS-TA)
- cold commissioning - maybe April 2024; full commissioning - early June with professional conductivity cell, during MLZ Energy Conference (with guests from Japan)

Humidity for GISANS (Flexiprob project)

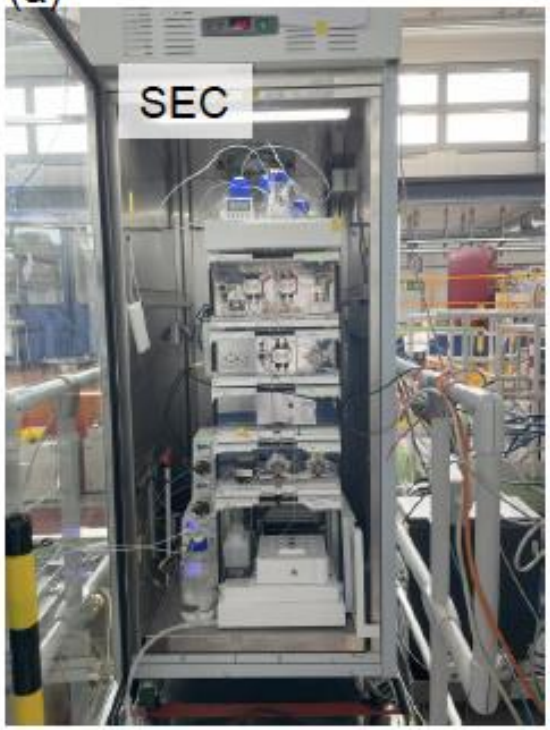


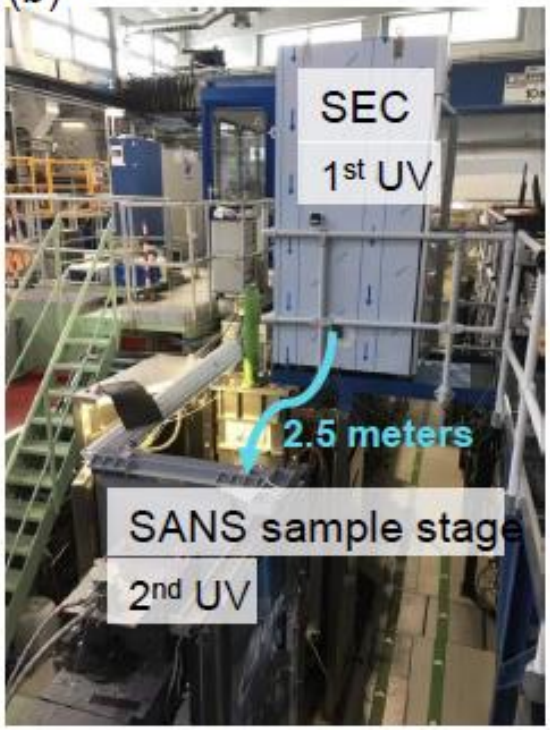


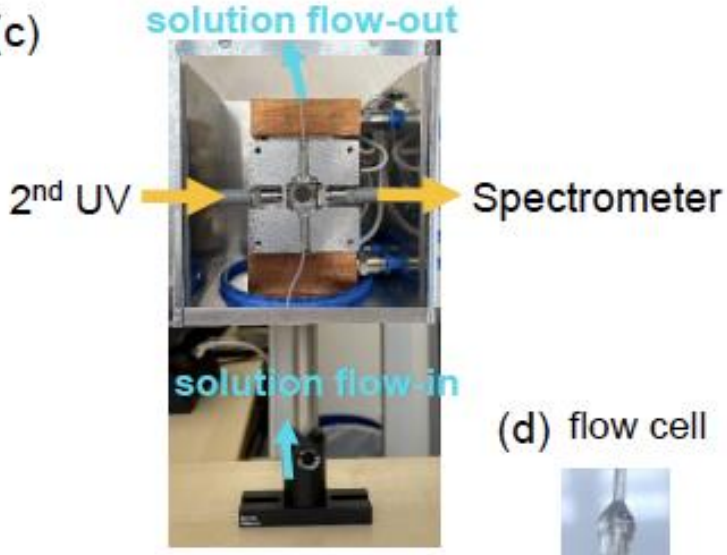




SEC – SANS on KWS2



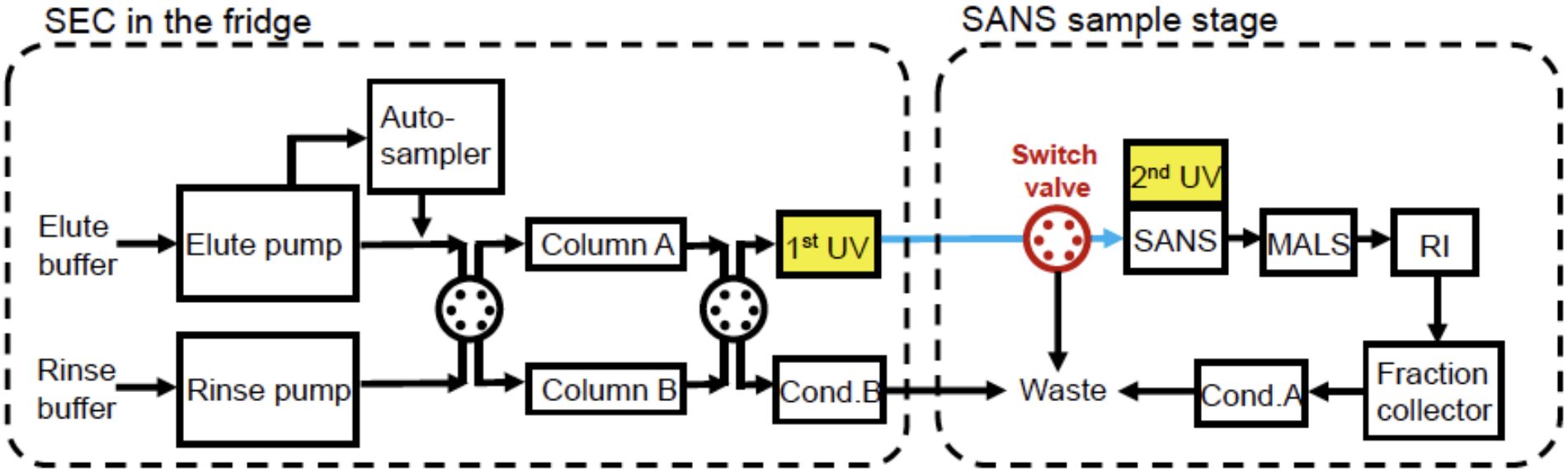




(a) SEC instrument in a fridge.
(b) The spatial arrangement: SEC is located at an elevated platform above KWS-2 sample stage.
(c) Thermo-controlled SANS cell holder, installed with the 2nd UV detection setup.
(d) Customized SANS flow cell. The blue square marked on the window shows the exposure position by neutron beam (5*5 mm²).

- 4 clear quartz windows
- neutron light path 1 mm
- UV light path 9 mm
- inner volume 135 µl

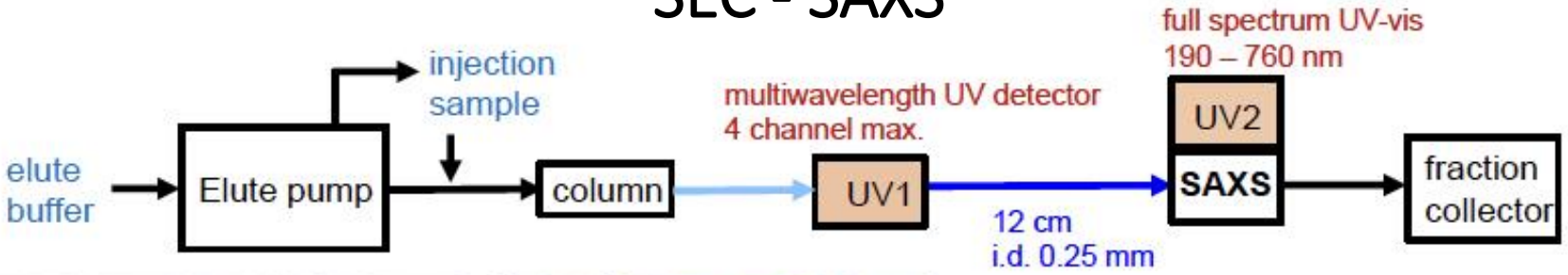
SEC – SANS on KWS2

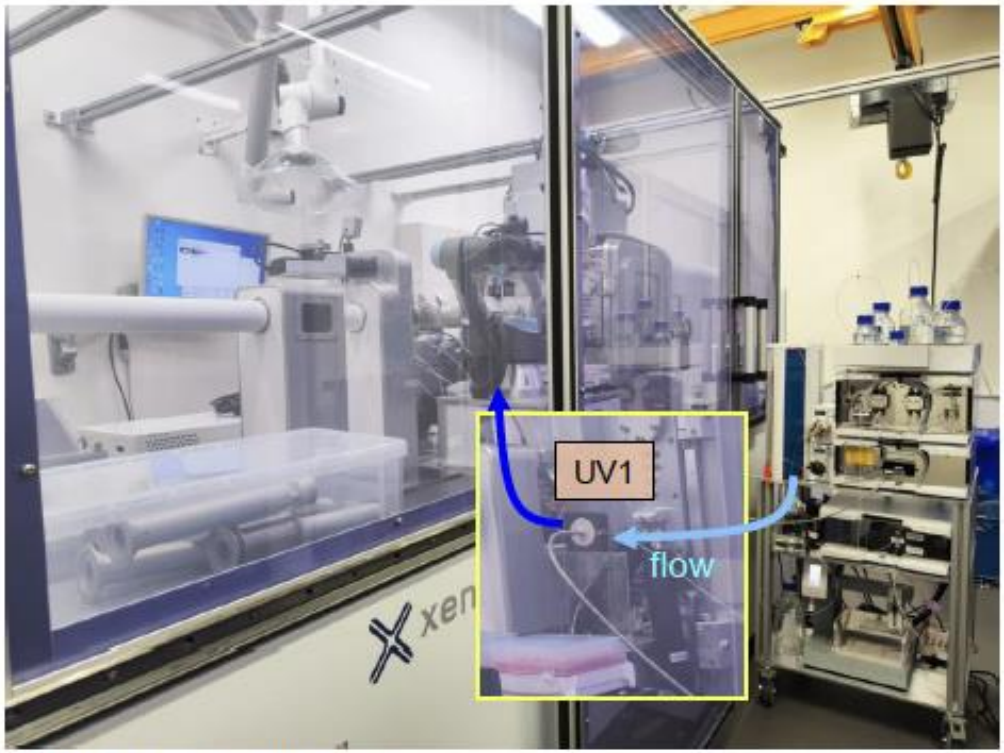


Design features:

- Dual pumps: simultaneous elute and rinse of two columns
- Auto-sampler: programmed injection of samples in desired series
- Both UV and RI detectors
- Switching valve, installed upstream of the SANS cell
- 2nd UV-vis setup installed to monitor the purified samples flowing into the SANS cell
- SEC-SANS-MALS, to determine molecular weight independently from SANS data

SEC - SAXS

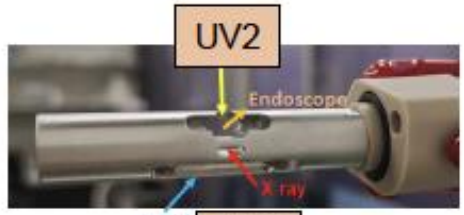


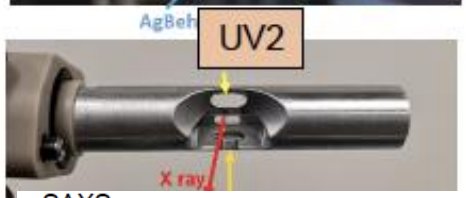


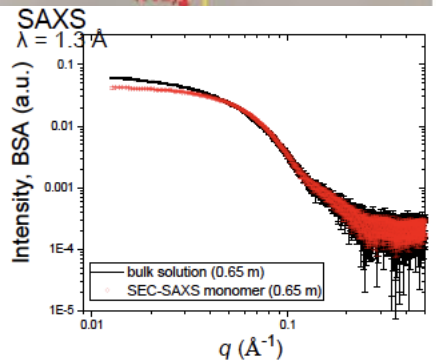
XENOCS SAXS machine, Xeuss

- Refractive Index Detector
- Conductivity Monitor CM 2.1S

Biocube Temperature controlled (XENOCS)
flow cell + UV-Vis + Video camera
inner diameter 1.9 mm





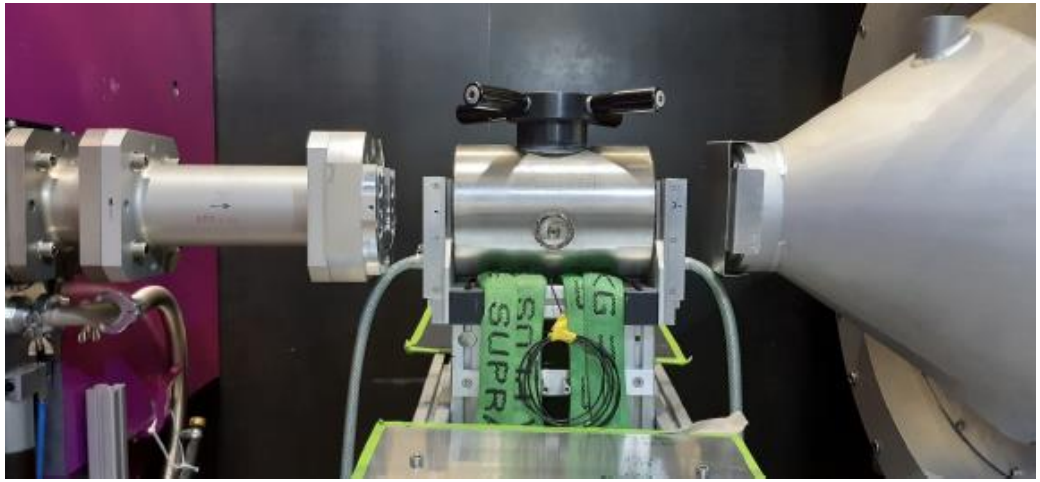


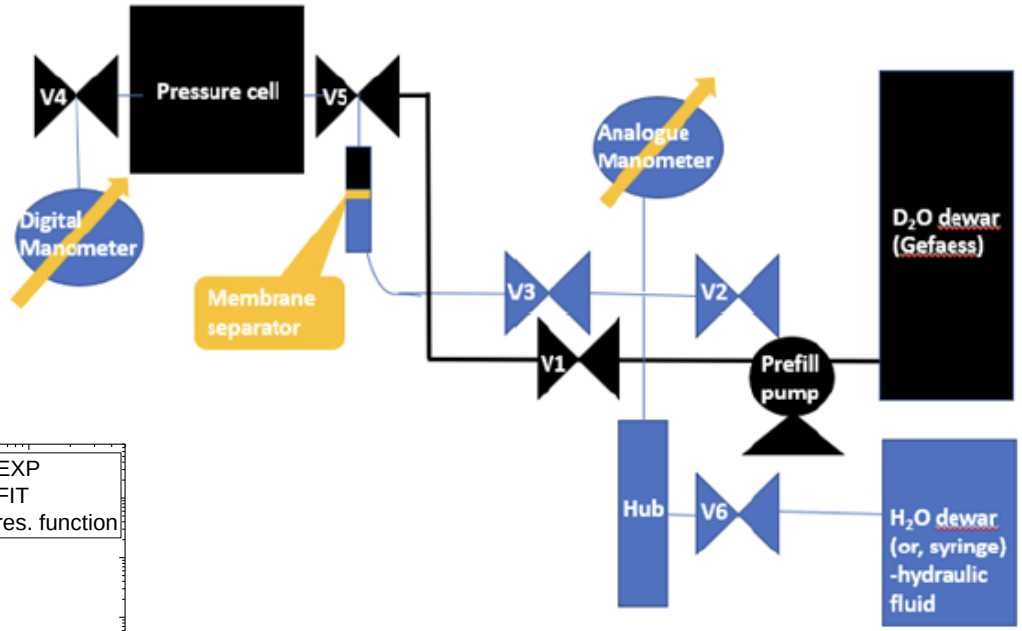
First TEST have been conducted !!
Proteins for Proteincrystallography, Food Science,
Protein structure as such

High Pressure Cell for GISANS

Pressure: approx. 1 kbar

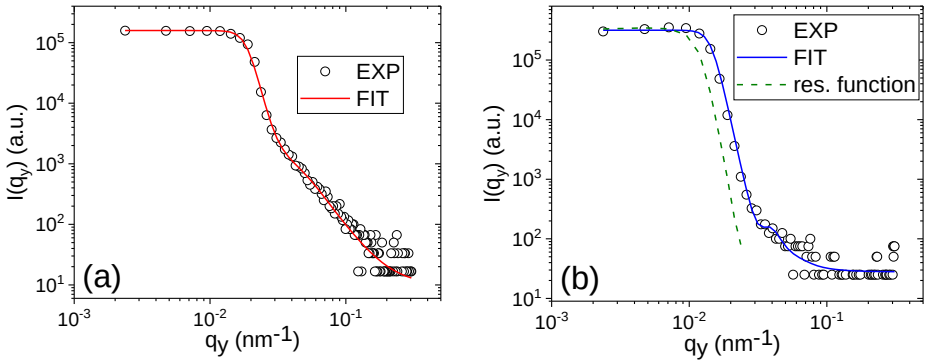
Advantages: easy sample exchange
Disadvantage: limited Q-range
for diffuse scattering





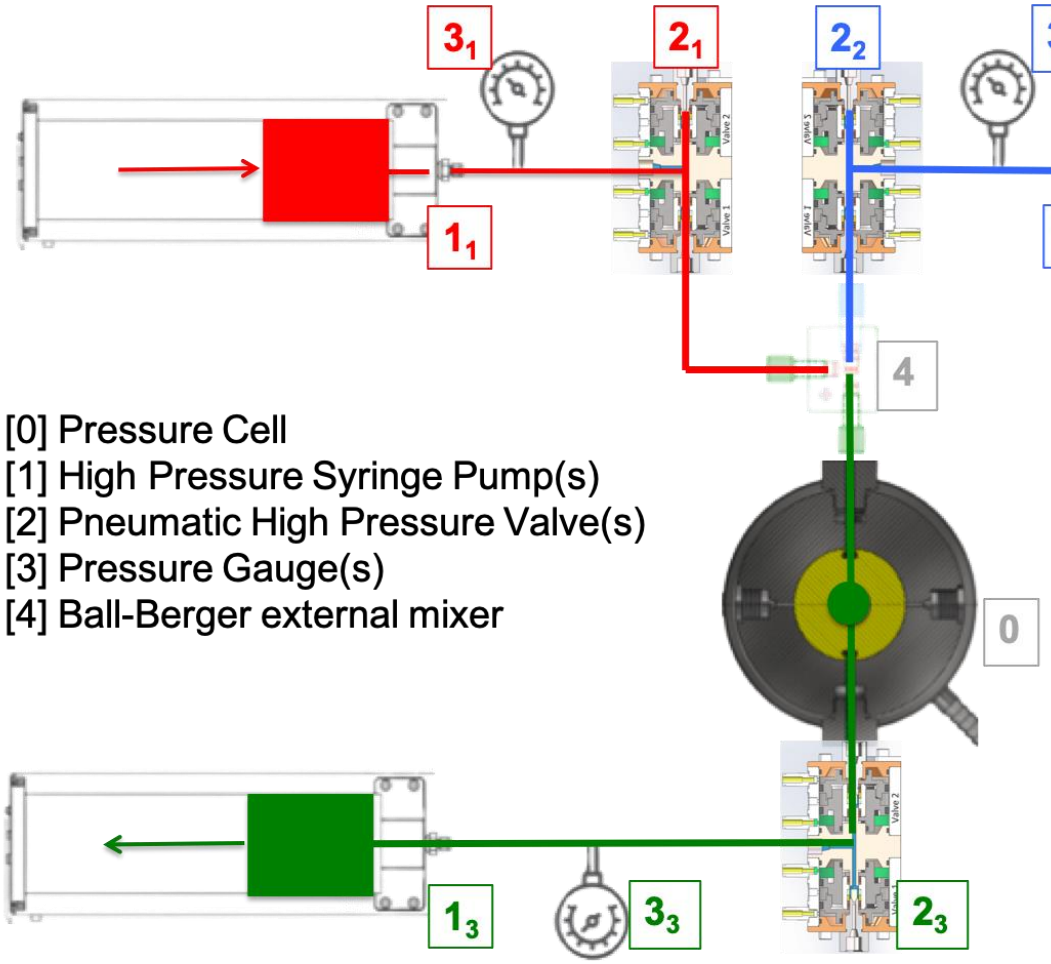
Fesibility tests at D22
Polymer brush

1 bar vs. 1k bar

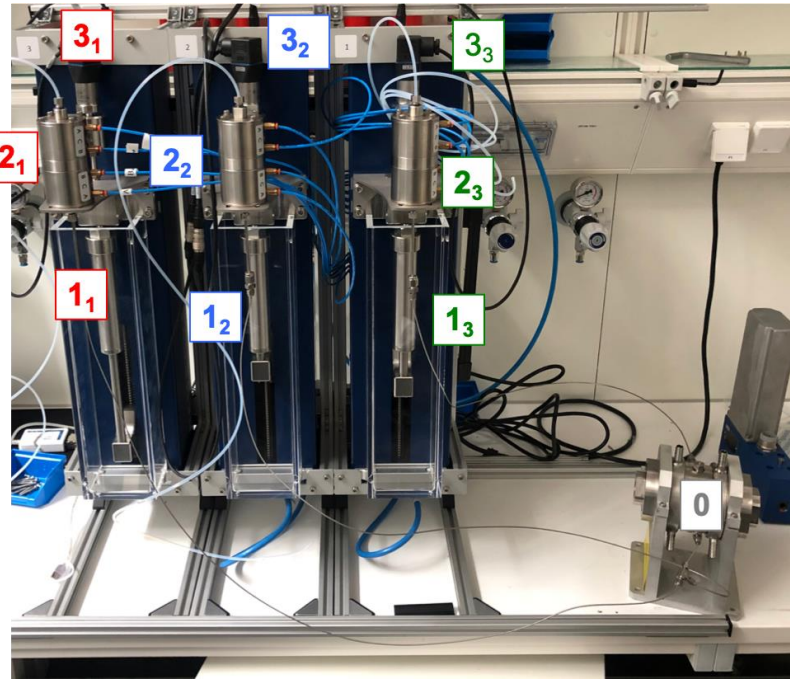


Wishlist: 2-3 kbar !!!!!!!

Stopped Flow Pressure Cell for SANS



[0] Pressure Cell
[1] High Pressure Syringe Pump(s)
[2] Pneumatic High Pressure Valve(s)
[3] Pressure Gauge(s)
[4] Ball-Berger external mixer

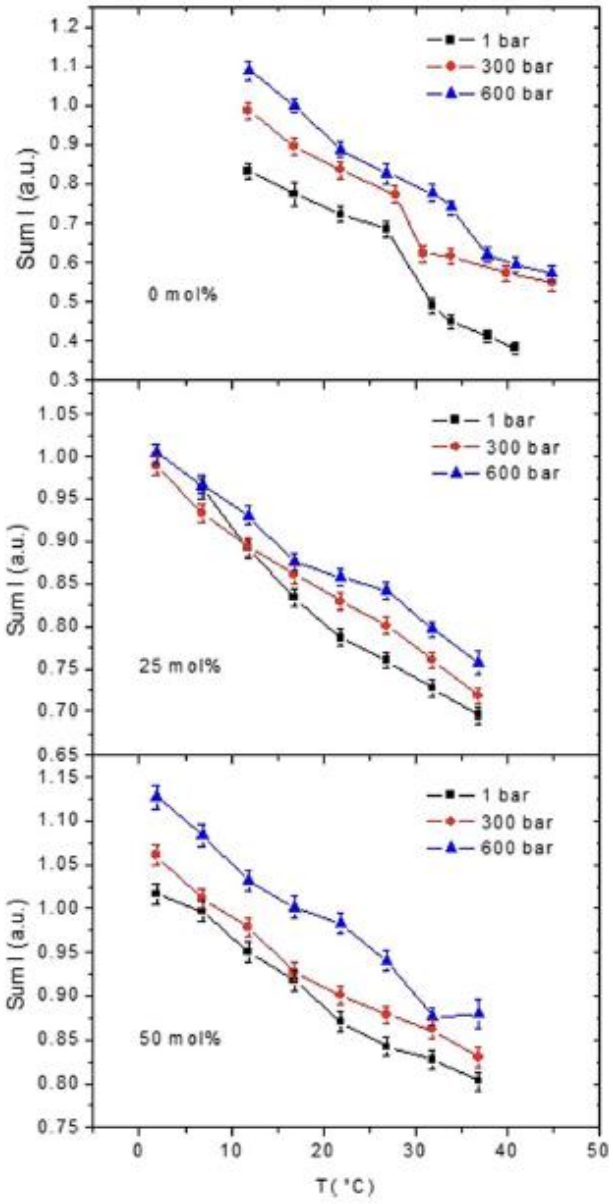


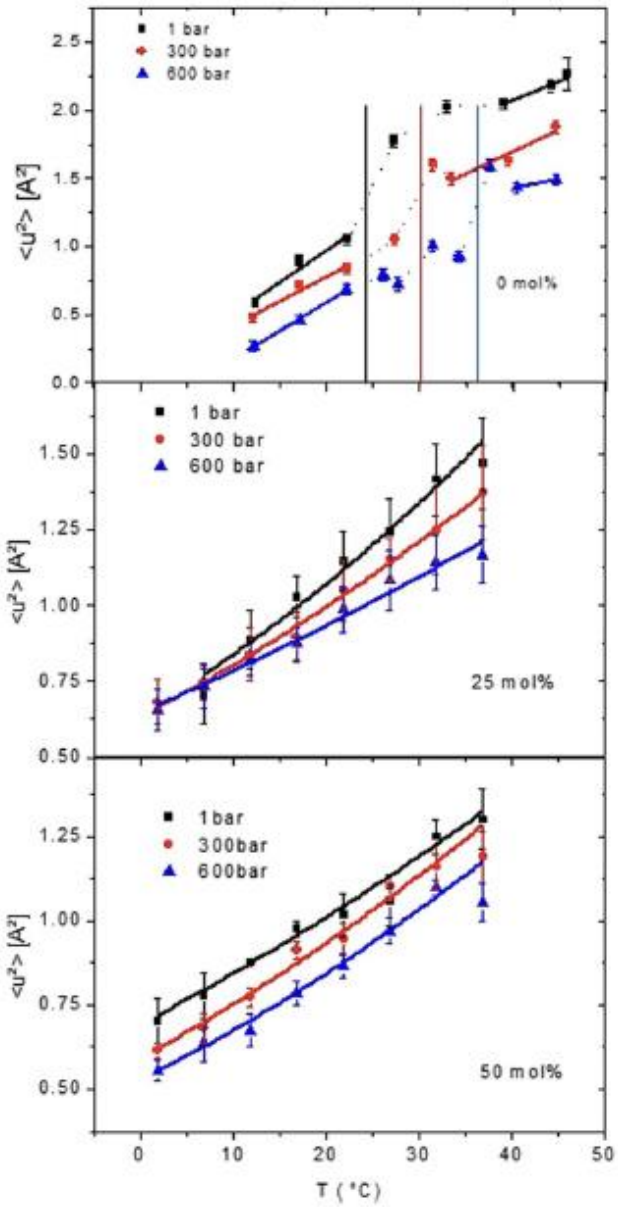
Features:

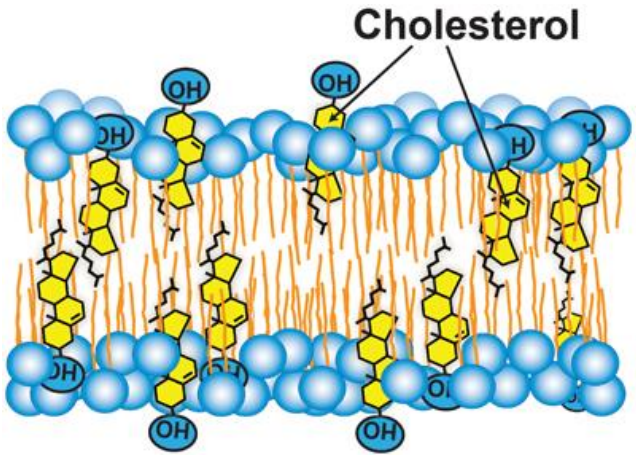
Max. Pressure: ~ 600 bar

Large sample cross section (MORIS update program for KWS1 – higher fluxes/count rates)

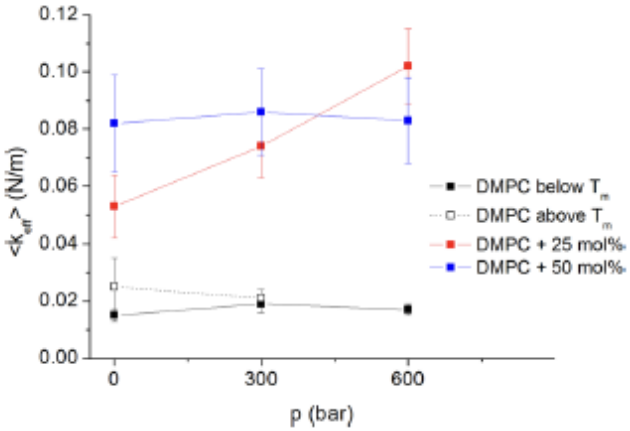
ROADMAP for future:
Pressure Cell for SPHERES







Thermodynamic Parameters
Force Constants

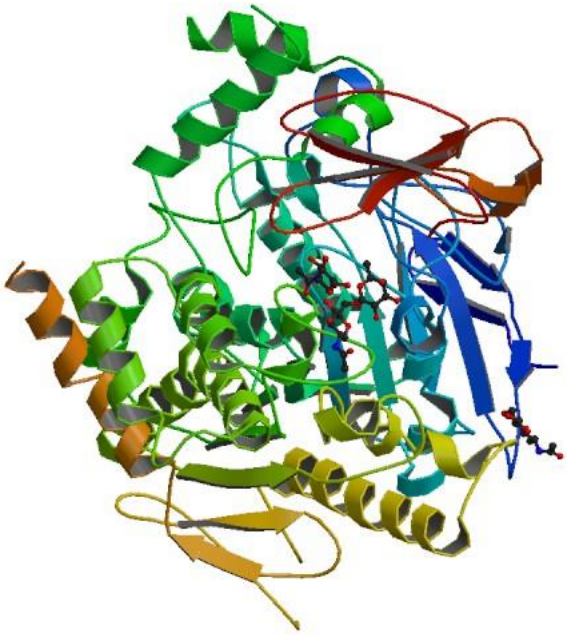


Peters, J., Marion, J., Becher, F. J., Trapp, M., Gutberlet, T., Bicout, D. J., & Heimburg, T. (2017). Thermodynamics of lipid multi-lamellar vesicles in presence of sterols at high hydrostatic pressure. *Scientific reports*, *7*(1), 15339.

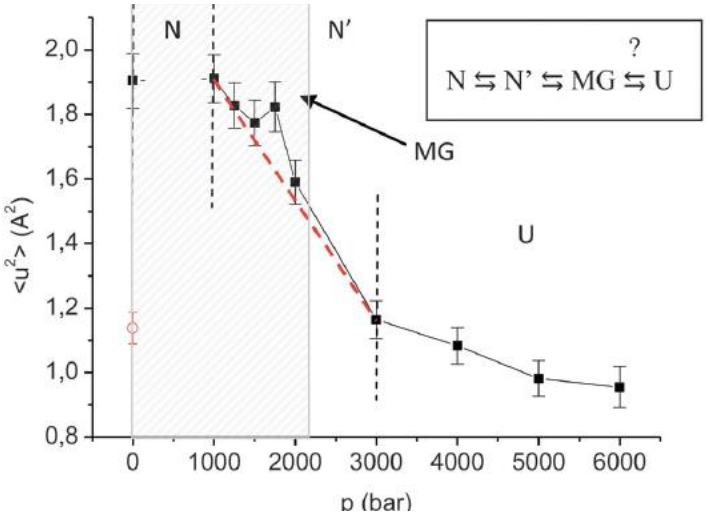
ROADMAP for future:
Pressure Cell for SPHERES

Denaturation:
Acetylcholinesterase

Different stages identified !!!!



IN13



Marion, J., Trovaslet, M., Martinez, N., Masson, P., Schweins, R., Nachon, F., ... & Peters, J. (2015). Pressure-induced molten globule state of human acetylcholinesterase: structural and dynamical changes monitored by neutron scattering. *Physical Chemistry Chemical Physics*, *17*(5), 3157-3163.

Food preservation

Food homogenization

Deep Sea Life

ROADMAP for future:
Pressure Cell for SPHERES

Water in concrete !
Pressure at larger depths
(deep sea)

Concrete with Super Plasticizer

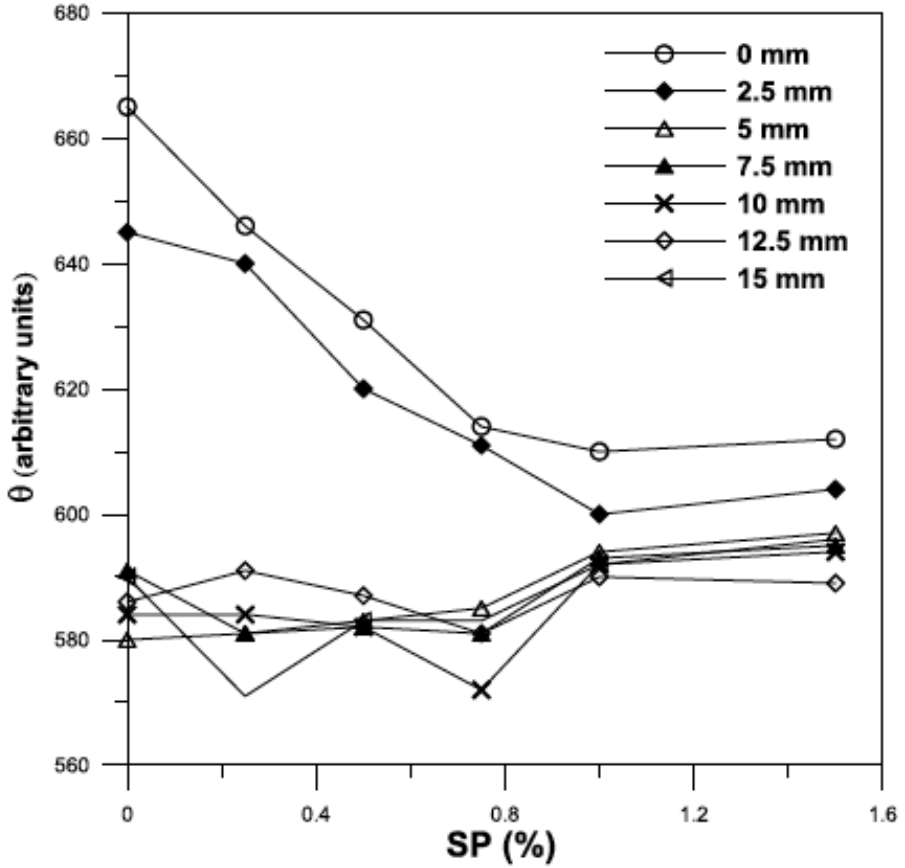


Fig. 8. Neutron backscattering at different depths from ordinary concrete mixed with SP submerged in water for 7 days.

Osman, A. M., & Abdelmonem, A. M. (2023). Study of water intrusion in admixture concrete using neutron backscattering technique. *Radiation Physics and Chemistry*, *203*, 110585.

ROADMAP for future:
Pressure Cell for SPHERES

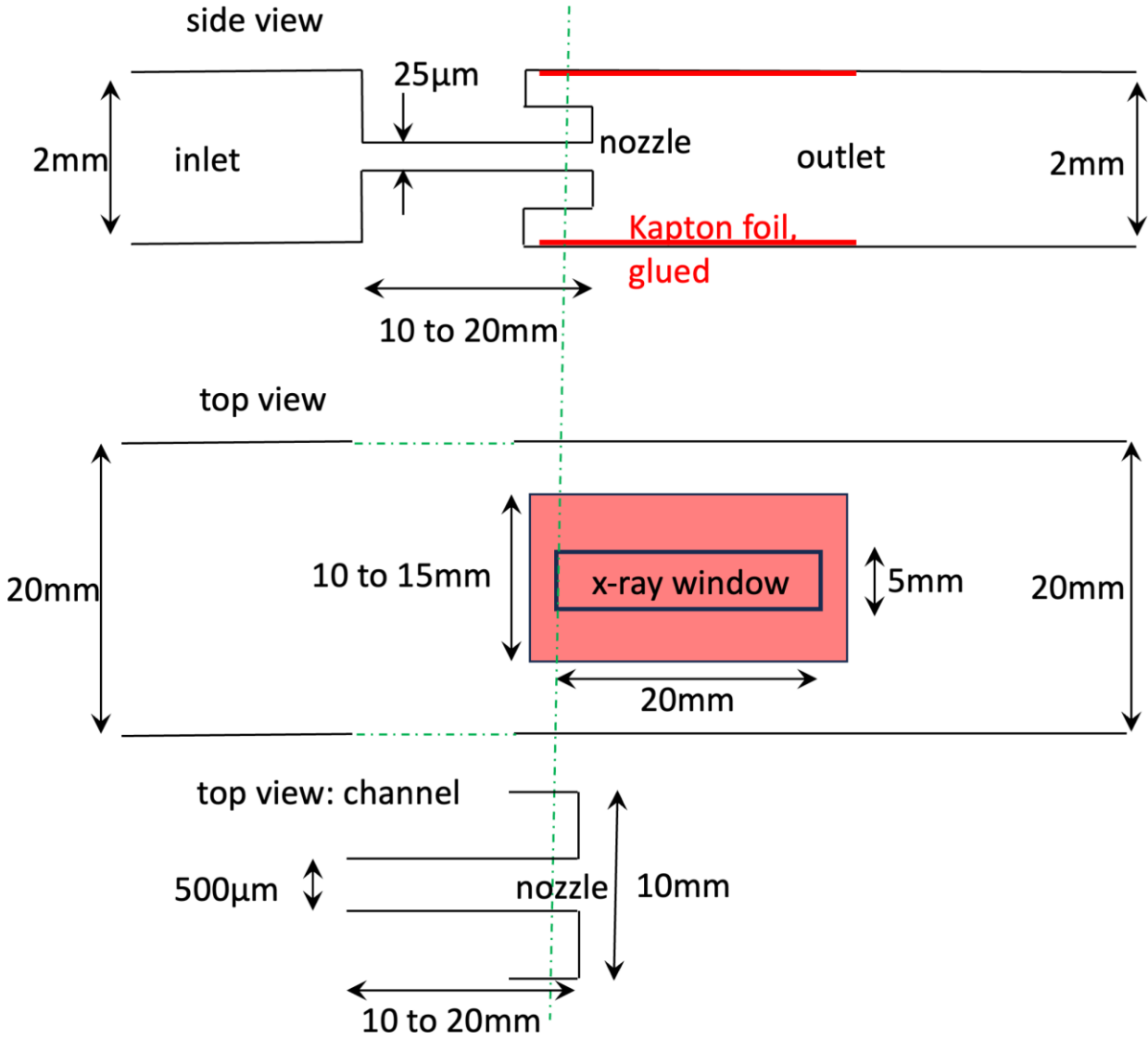
Wishlist:

P_{max} = 6 kbar ??
Large sample cross section
Large incident angles (45°???)
Slab geometry, ~45° inclination
Shielding

Who will be responsible?
Who actually needs it?

ROADMAP for future:
Homogenizer Cells for Small Angle Scattering

Aluminium



ROADMAP for future:
Homogenizer Cells for Small Angle Scattering

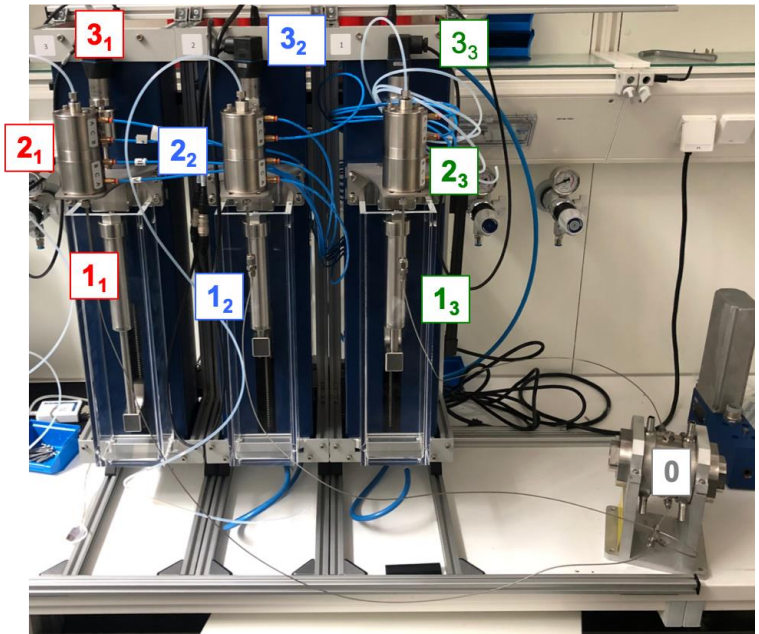
SAXS:

Either µm channel

Or: capillary

Pressure ~80 bar

One go sample supply



SANS/GISANS:

µm channel in sapphire

Pressure ~80 bar

Recycling of sample (??)

ROADMAP for future:
Homogenizer Cells for Small Angle Scattering

RÅC application
Project will be done anyhow

Also GEA will produce a homogenizer from titanium.

The Homogenizer cells will be produced step by step.

Whole apparatus needs to be cleaned by conc. NaOH.