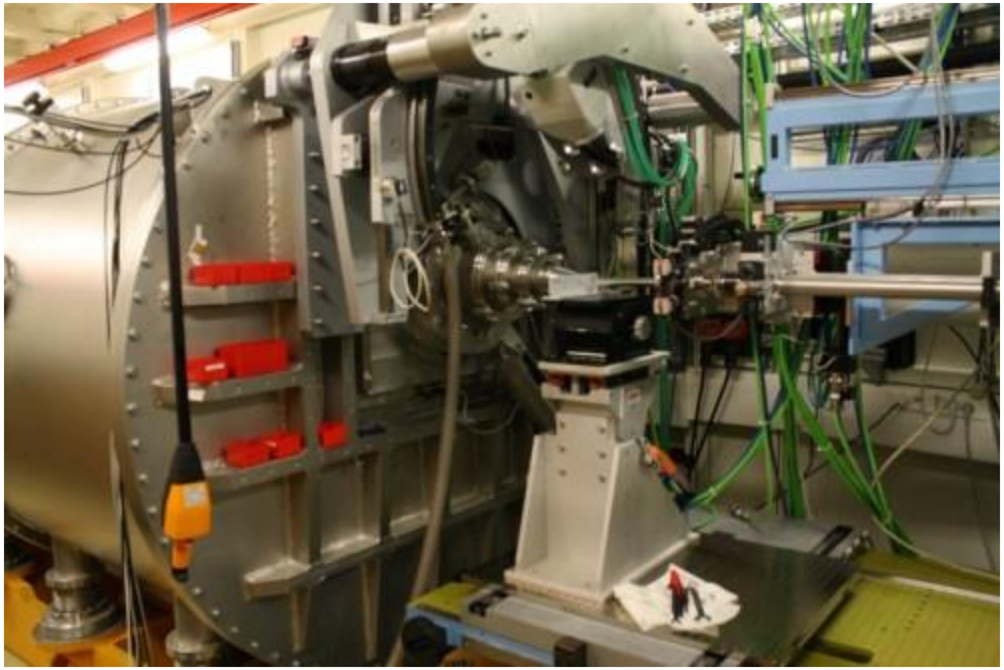
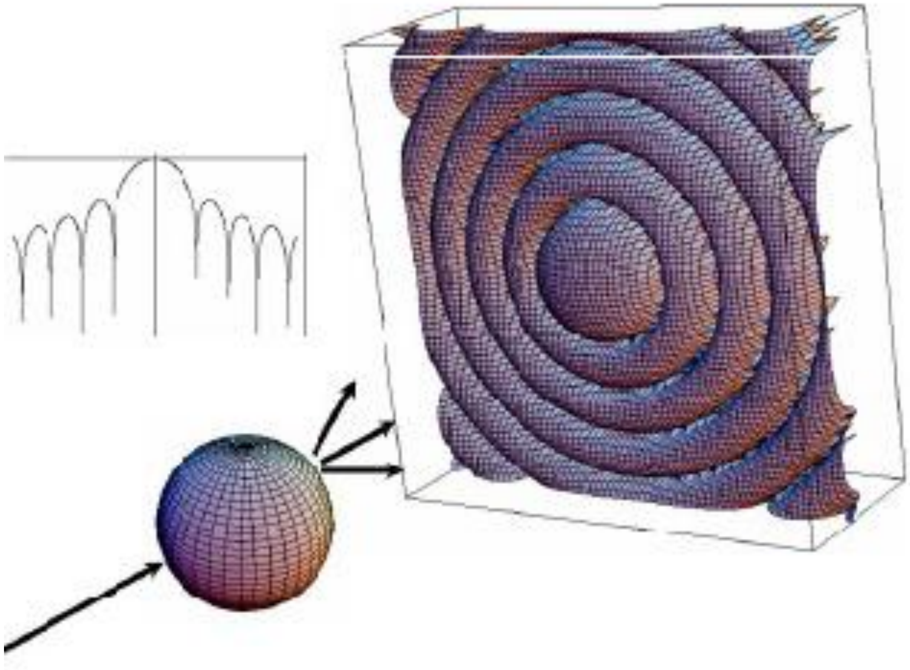
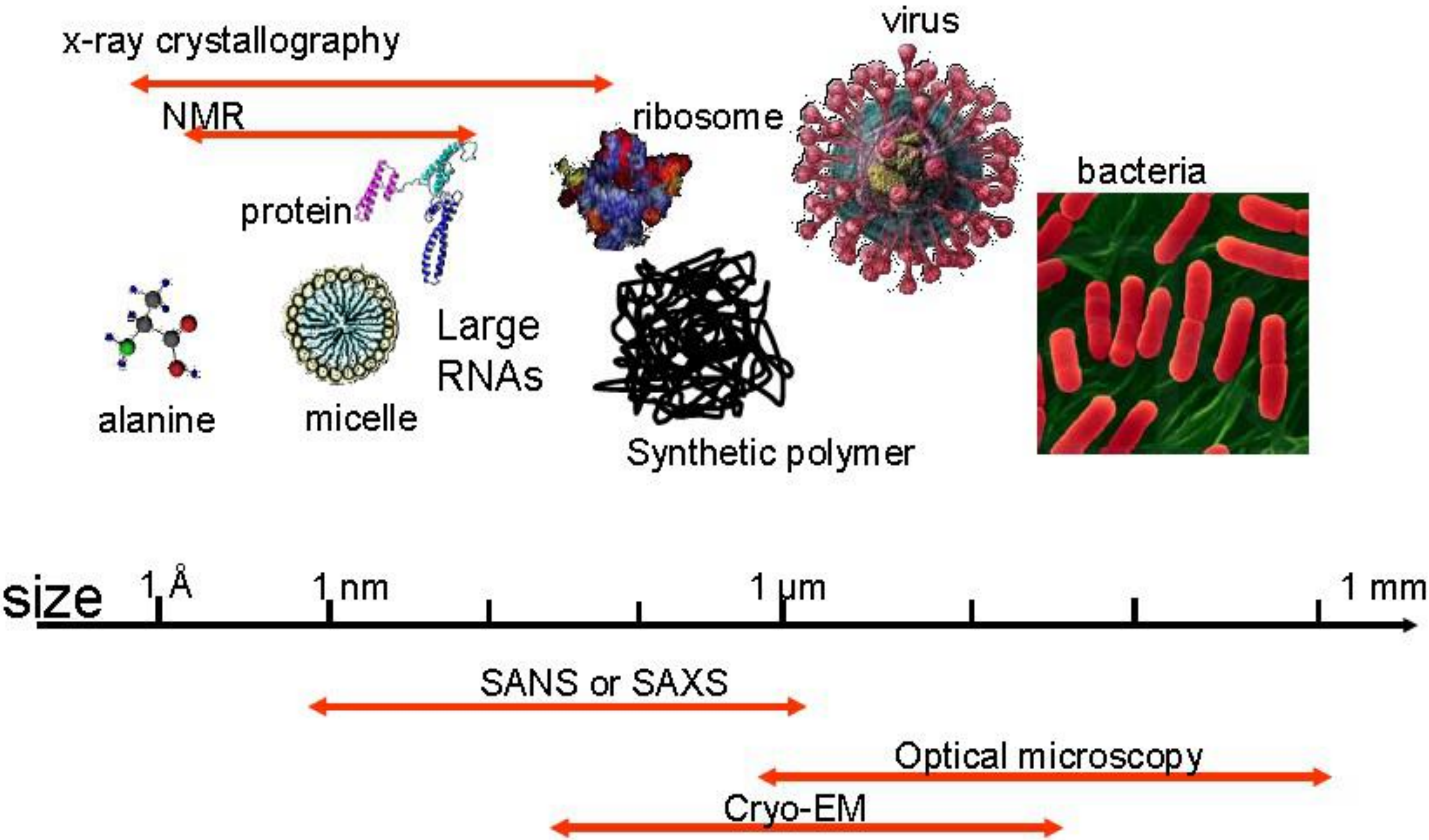
Sample environments in Small Angle (X-Ray) Scattering





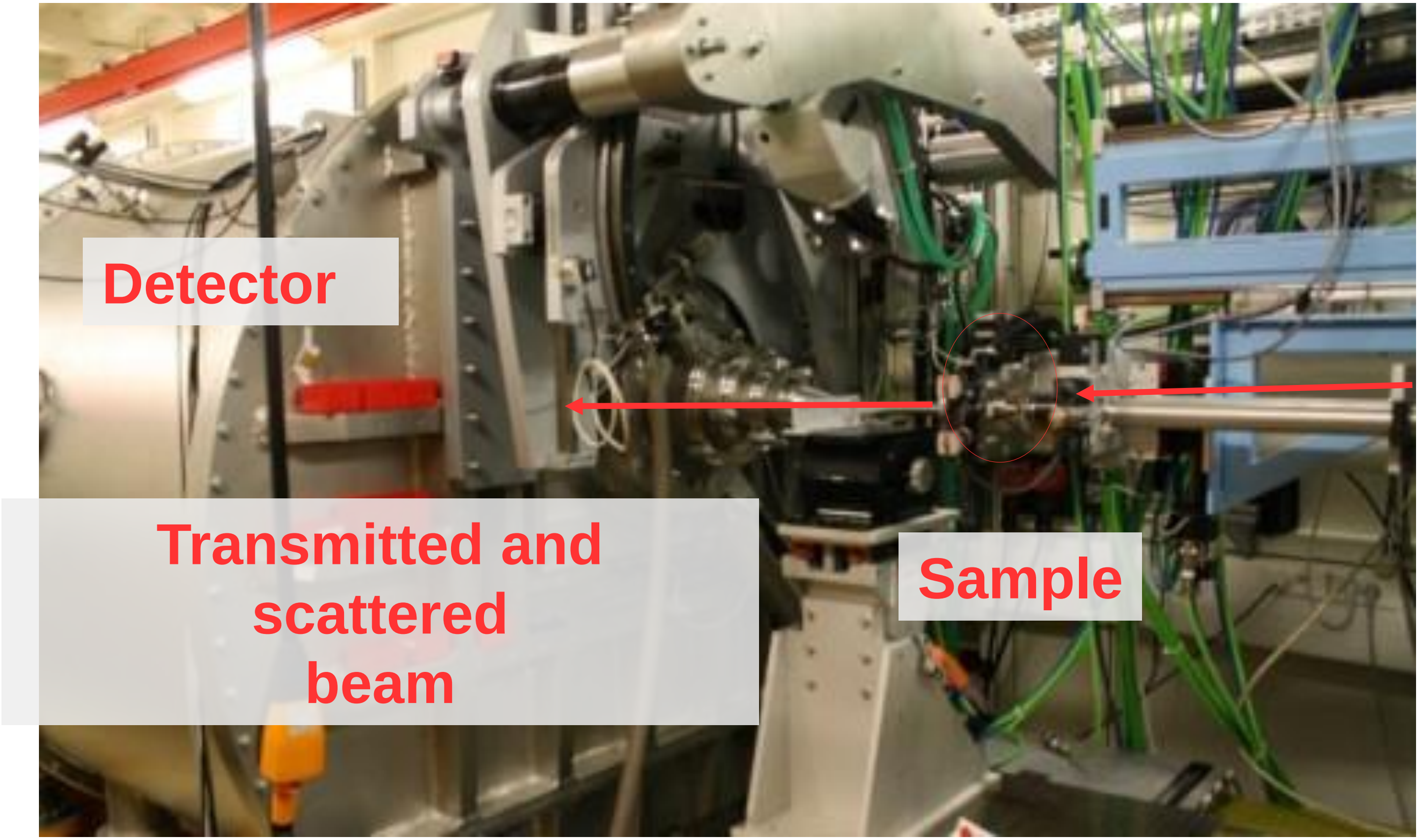
Scales of various methods



The scope of small angle X-ray scattering in terms of spatial dimension covers ~1nm to ~1µm ranges, perfectly suitable for biomolecular structrual study.

SWING Beamline, SOLEIL

SWING Beamline, SOLEIL

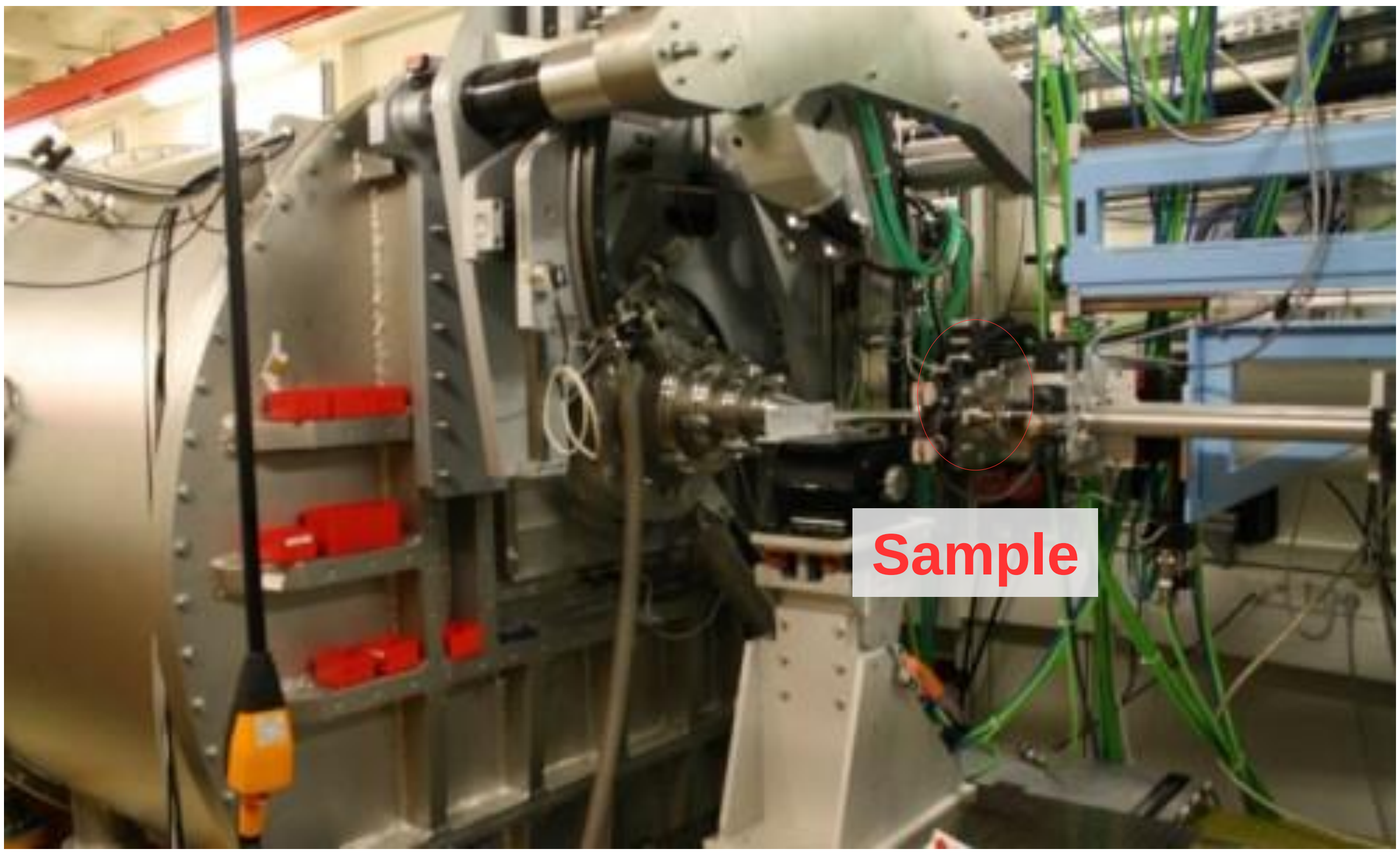


Incident beam

SWING Beamline, SOLEIL

Inside the vacuum chamber

SWING Beamline, SOLEIL



Sample environment can virtually be *anything*, proposed by the beamline or home-made

Sample environments proposed by ID02 (ESRF) and SWING (SOLEIL)

SOLEIL SYNCHROTRON

Sample Environments

- HPLC: SEC-SAXS
- AutoSampler for BioSaxs
- BioSaxs: Vacuum cell
- High Pressure for Liquids
- Static Capillaries Holder
- Circulation Capillaries Holder
- Gels / Solids (polymer slice, pellet) Holder
- Liquids / Gels holder
- Mixing and pipetting cell
- Linkam Temperature Controle Stage
- Traction Cell
- Rheometer
- Stop-Flow
- Microfluidics
- ChemSaxs Liquid AutoSampler
- StopFlow-MALS-SAXS

ID02 - Time-Resolved Ultra Small-Angle X-Ray Scattering

ID02 SAMPLE ENVIRONMENTS

- Liquid sample changer coupled to a flow-through capillary cell
- Stopped-flow rapid mixing device
- Stress controlled rheometer
- Mettler heating stage
- Linkam heating stage
- High pressure cell
- Magnetic field setup
- Four position sample changer
- Industrial sample changer
- Capillary sample changer

Users bring their own

UV-Vis Cell

M. Goldmann
Sorbonne Université

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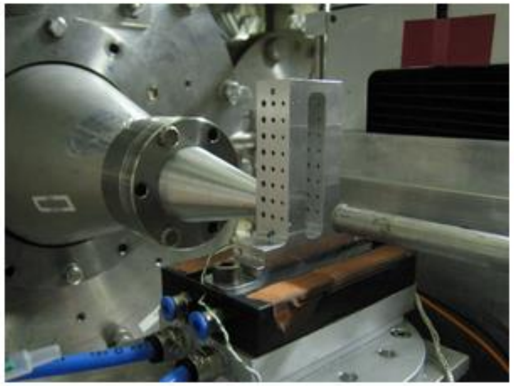
Important:

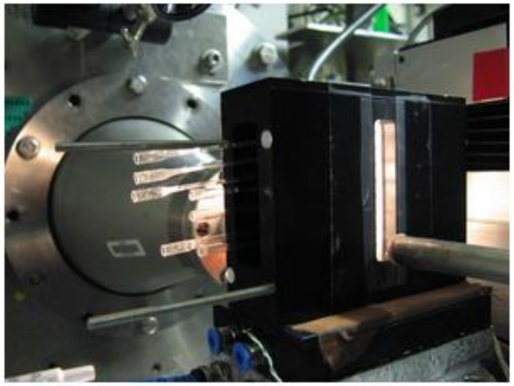
sample environments also work on **Lab-scale SAXS**!!

- Provided by the producer
- Your own

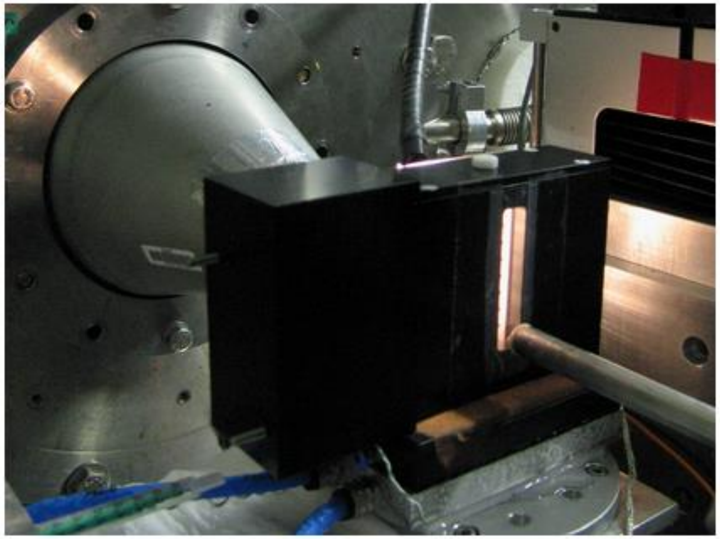
Capillary sample changer

ID02





SWING



Advantages

Samples can be pre-prepared

Fast changing

Gravity

Problems

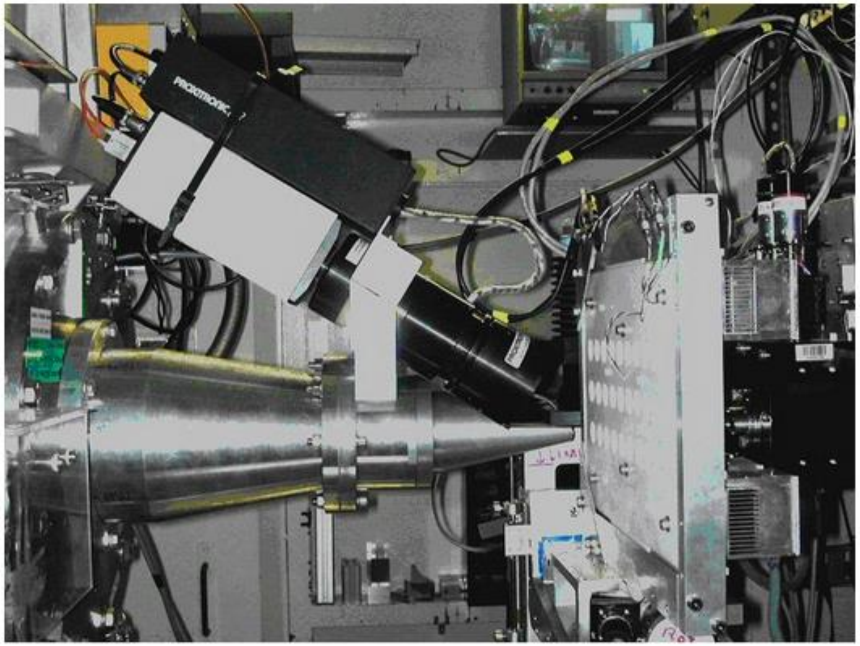
Background subtraction

No in situ possible, single scan only

Gravity

Other sample changers

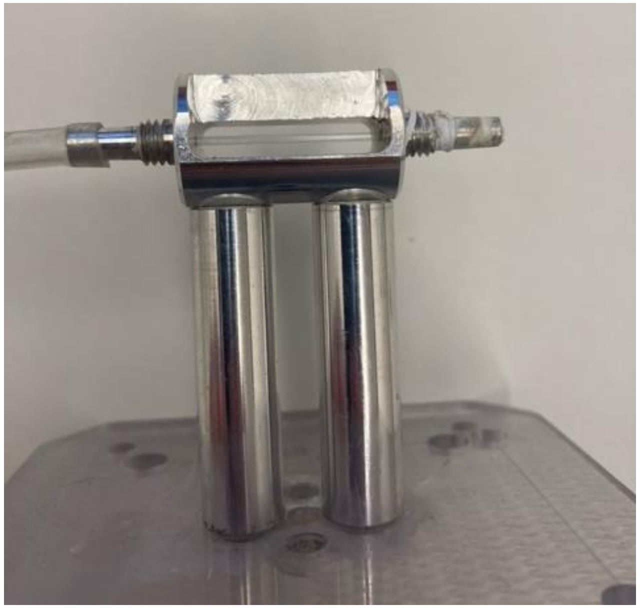
Industrial sample changer
ID02

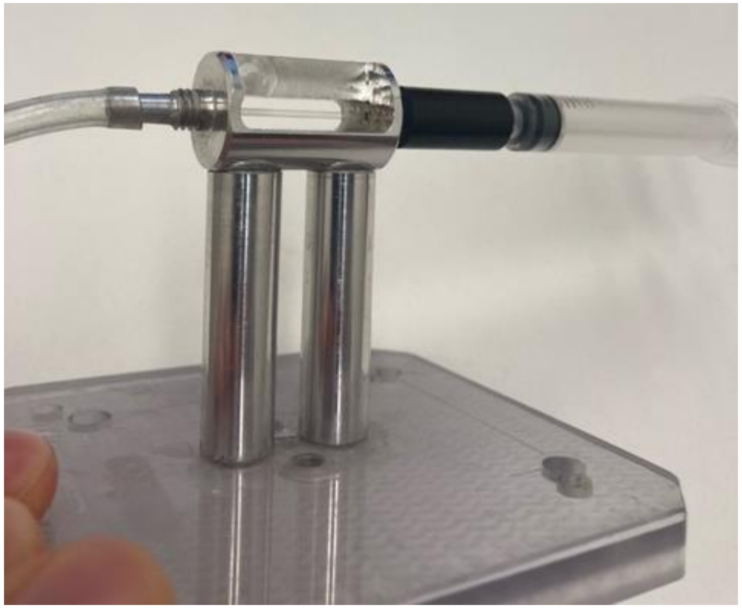


Sample changer for Gels
SWING

Flow-through capillary

My own





Advantages

Reliable background subtraction

Both single scan and *in situ* experiments

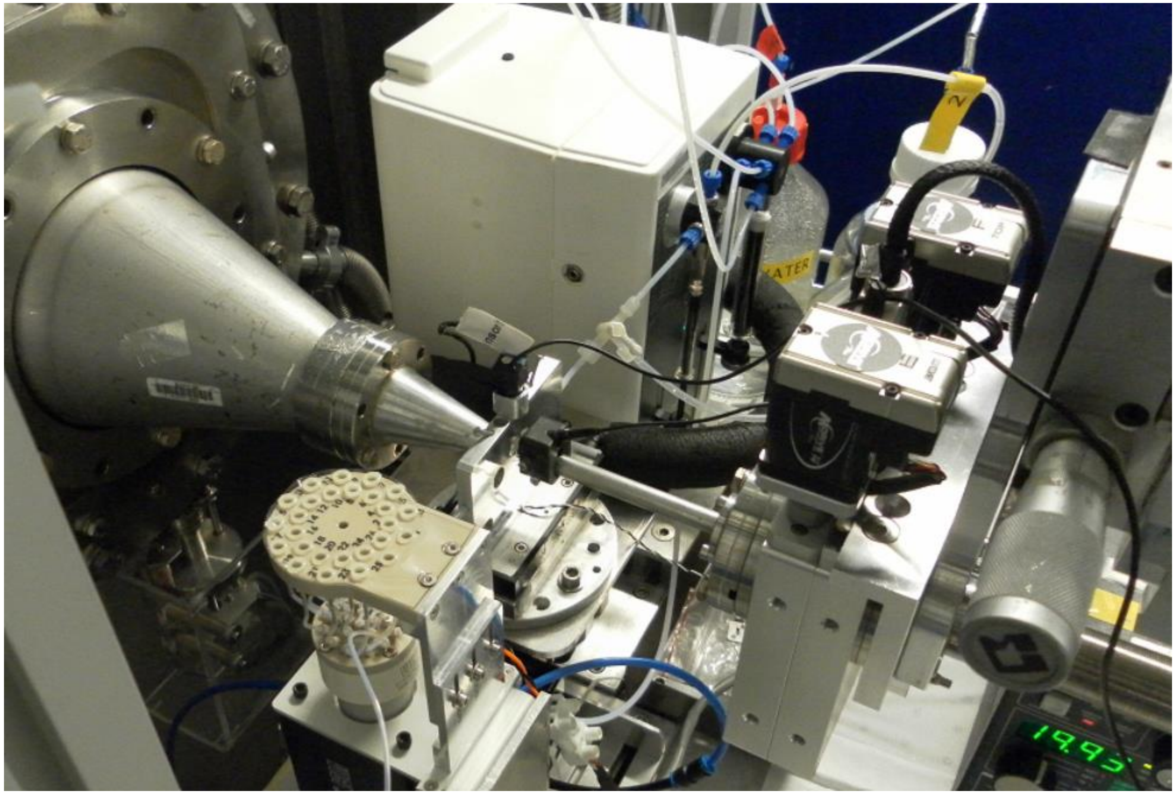
Problems

Slow if many samples

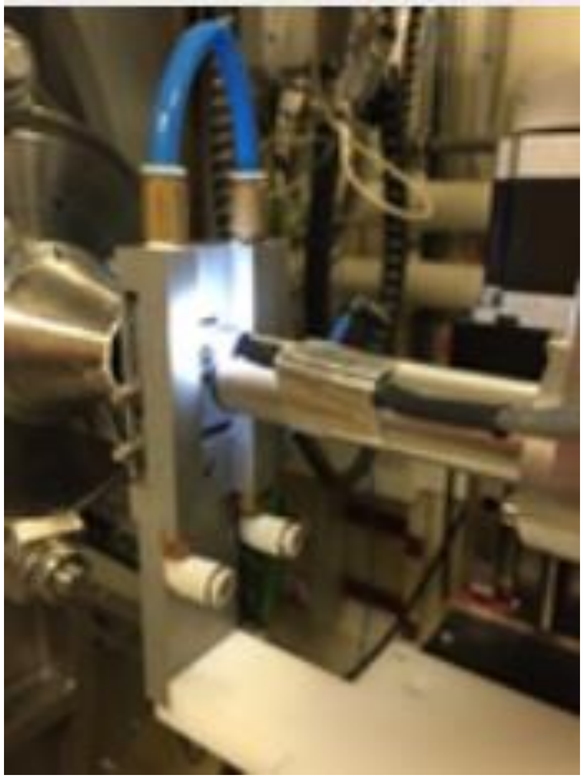
Time-consuming if it breaks

Flow-through capillary

ID02

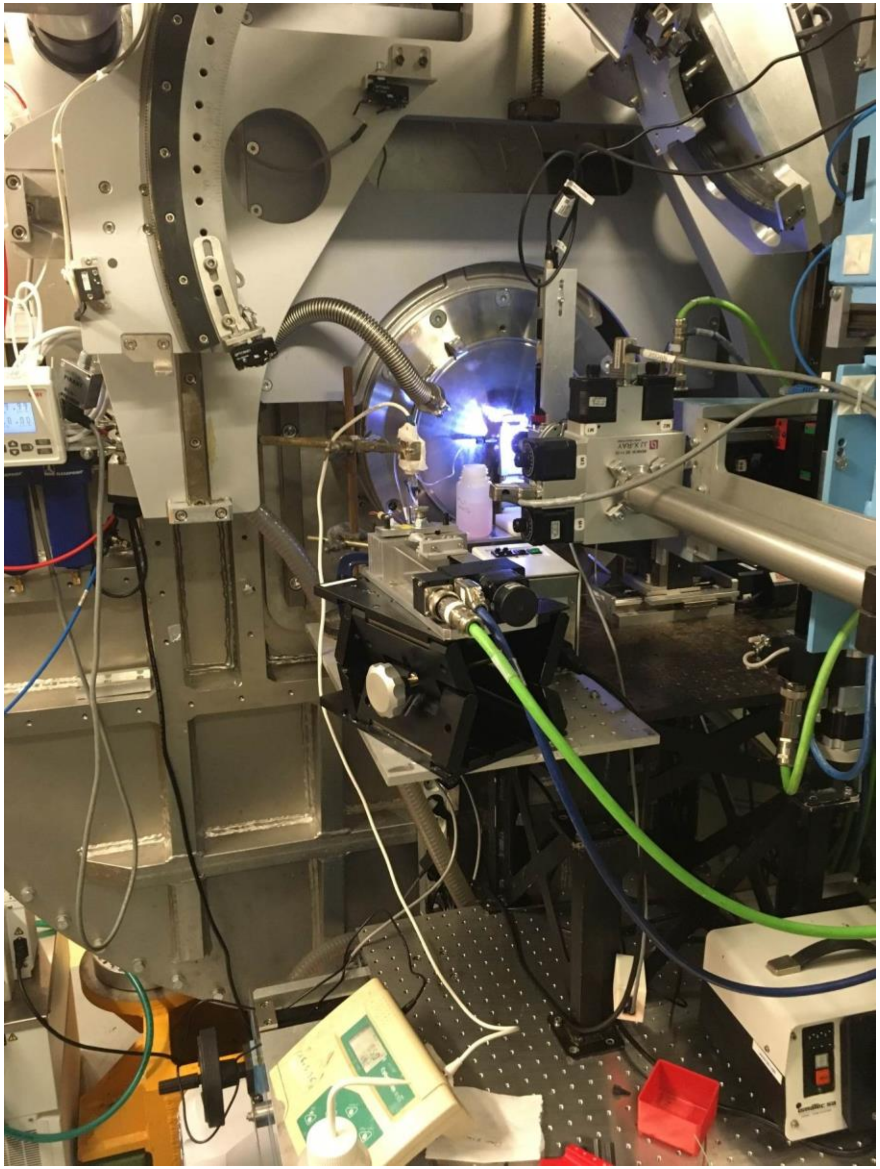


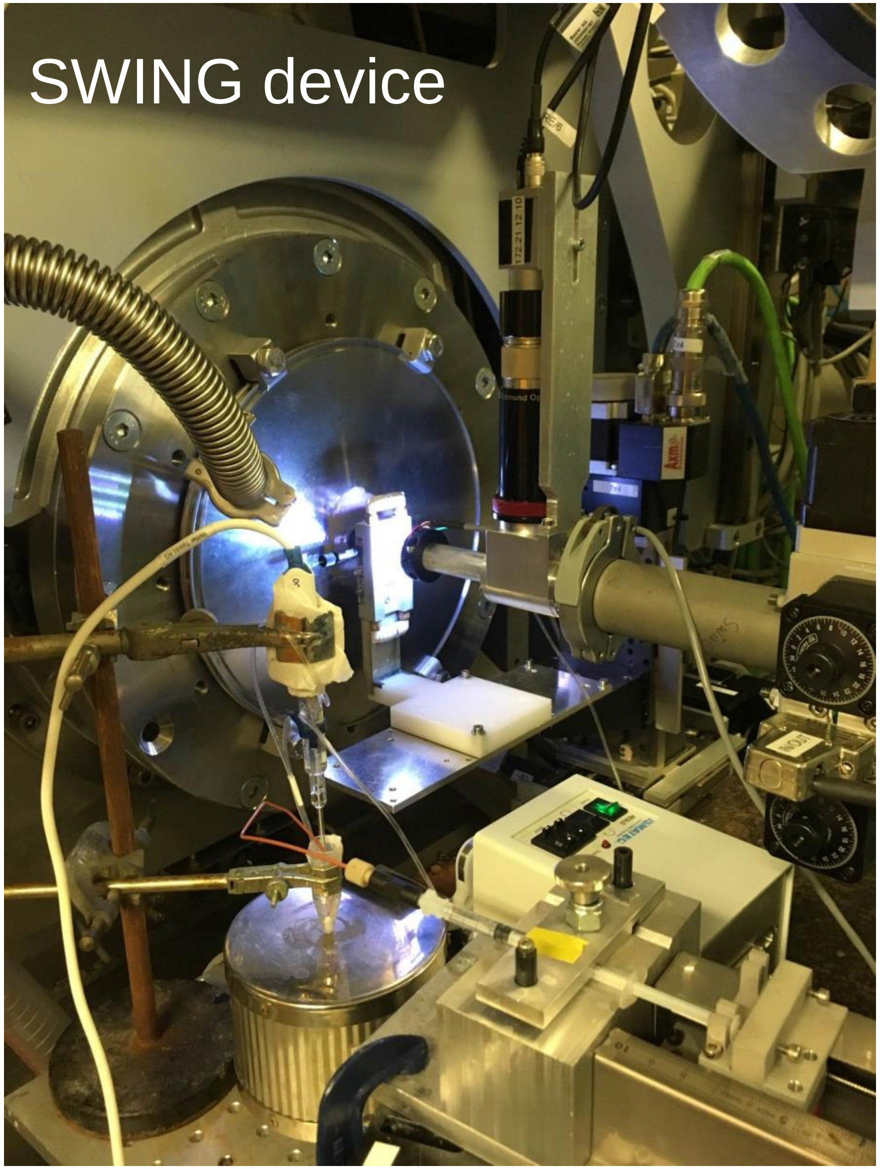
SWING

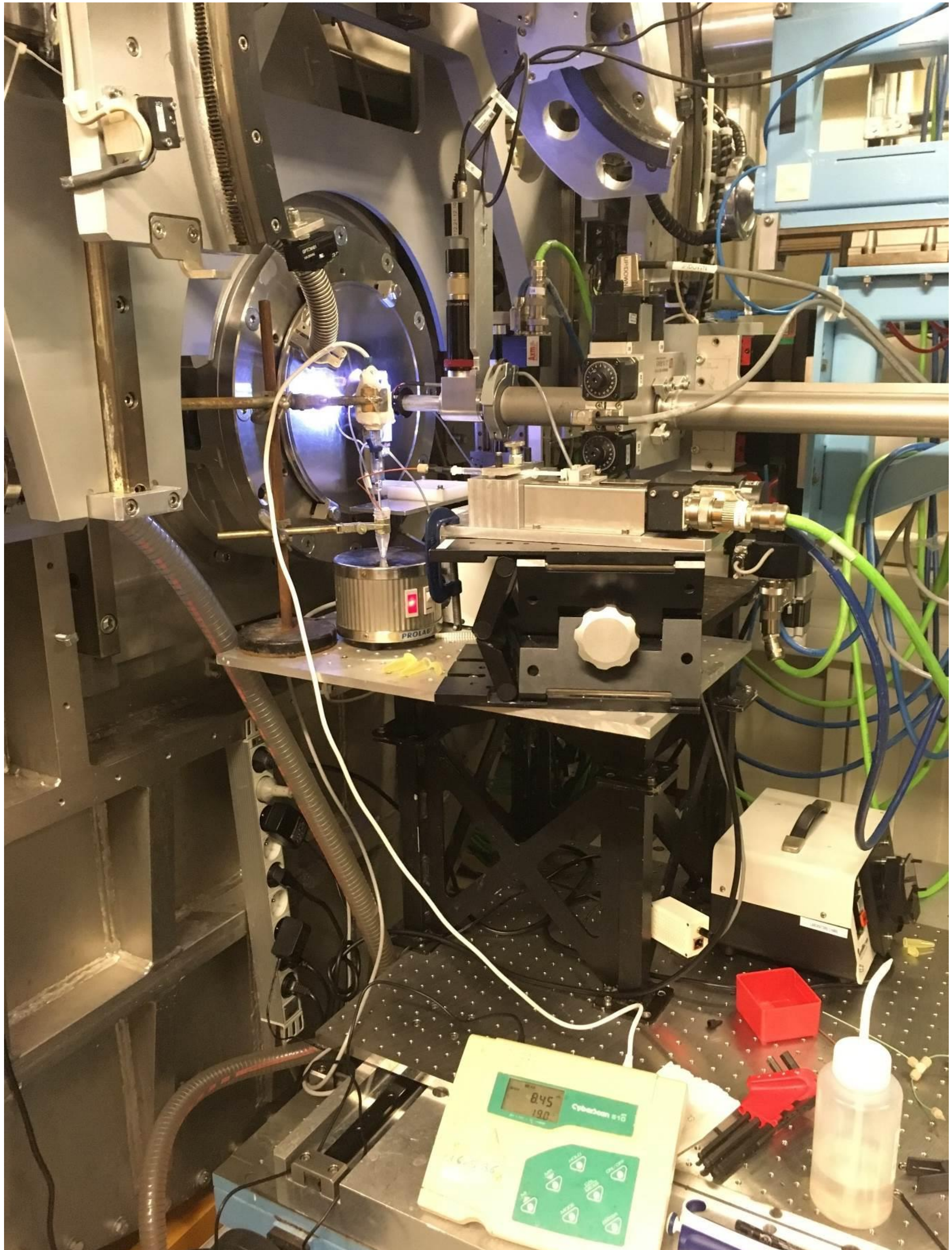


Flow-through capillary for *in situ* experiments

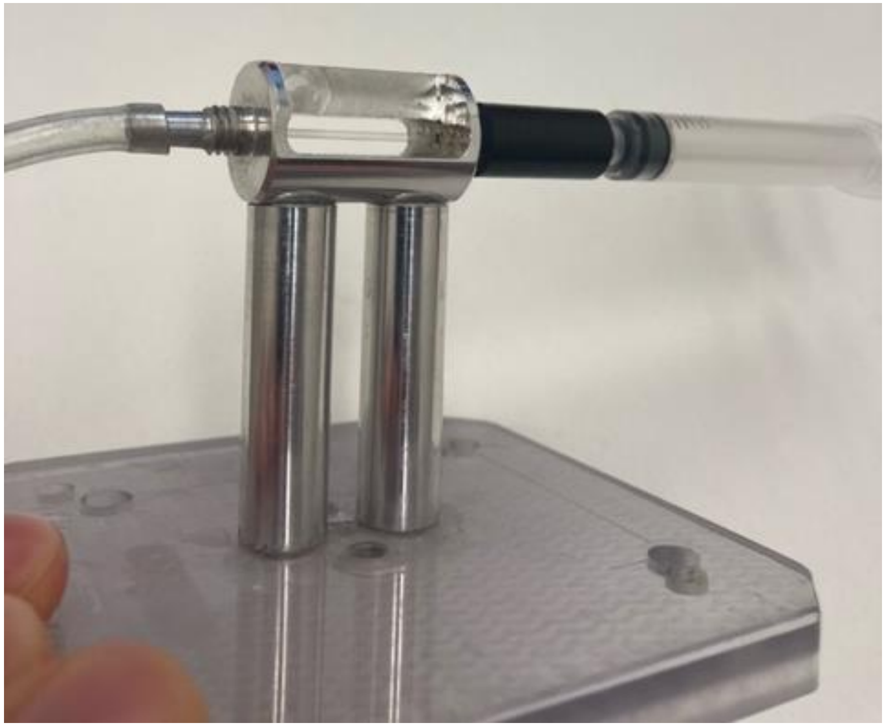
Time-resolved (Dead time: 3 min, then ms-scale)
pH, ionic strenght, co-solvent, chemical reactions…






PROLAB

My own device



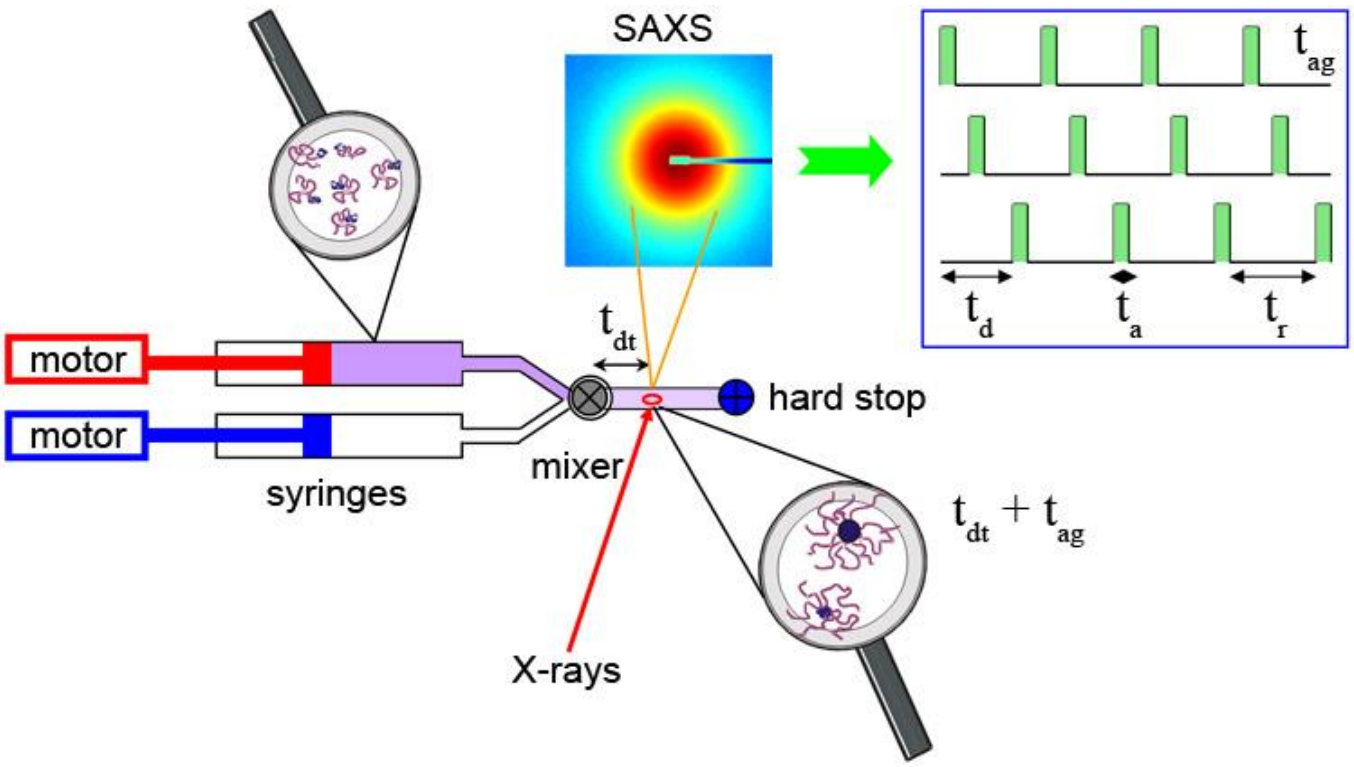
Flow-through capillary for *in situ* experiments

Time-resolved (Dead time: 3 min, then ms-scale)
pH, ionic strenght, co-solvent, chemical reactions…

Advantages
Time-resolved data
Bulk experiment
Statistiscs

Problems
Leaks
Capillary breaks
« short » time scales
Poor homogeneity

Stopped-flow device for *in situ* experiments



Advantages
Real time resolved
Solid setup

Problems
Low intensity depending on contrast

Initial stage only

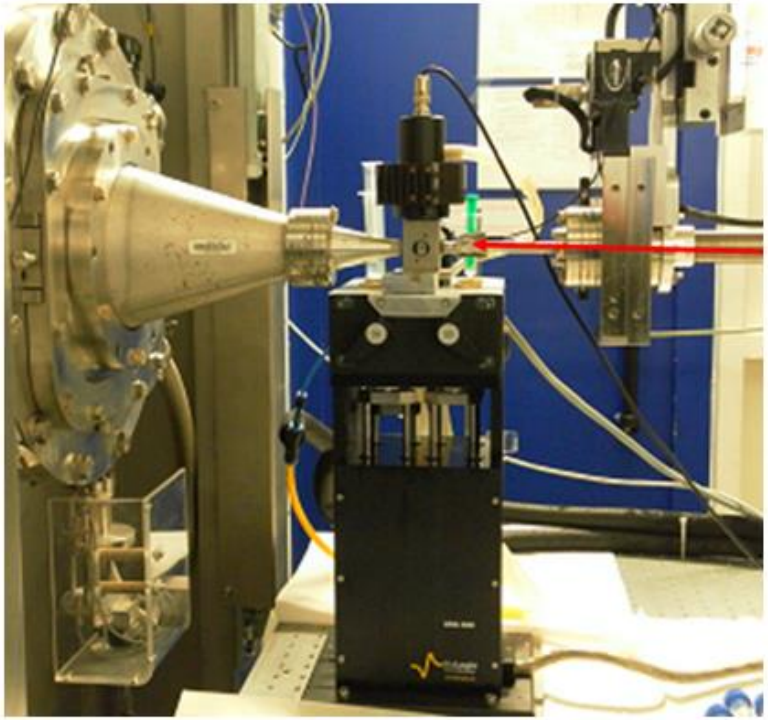
P. Panine, S. Finet, T. Weiss, and T. Narayanan, *Adv. Colloid Interf. Sci.* 127, 9 (2006)

Stopped-flow device for *in situ* experiments

L. Matthews and T. Narayanan, Colloid Polym. Sci., **301**, 721 (2023).

T. Narayanan, J. Gummel and M. Gradzielski, in *Advances in Planar Lipid Bilayers and Liposomes,* **20**, p 171-196, (Elsevier, 2014). [Reprint is available upon request].

Photos of the stopped-flow devices (SFM-400 and SFM-4000) installed at the beamline:

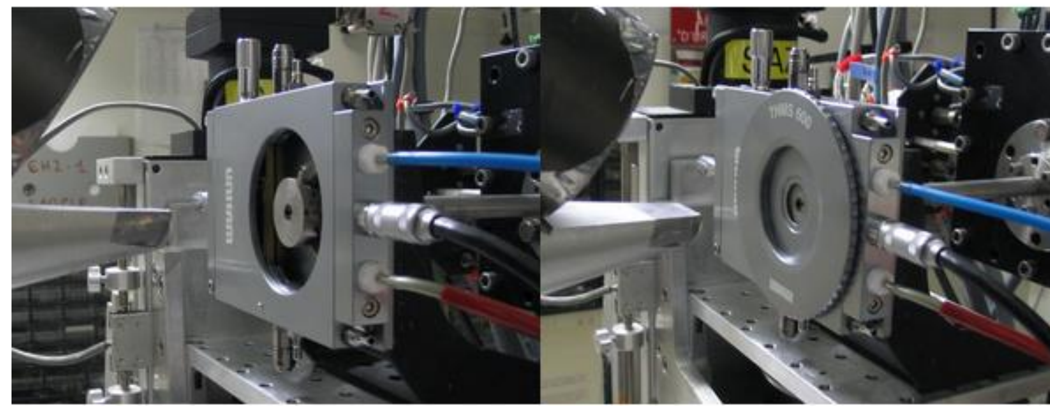


Temperature: single scan and flow-through

Single scan (ID02 version)

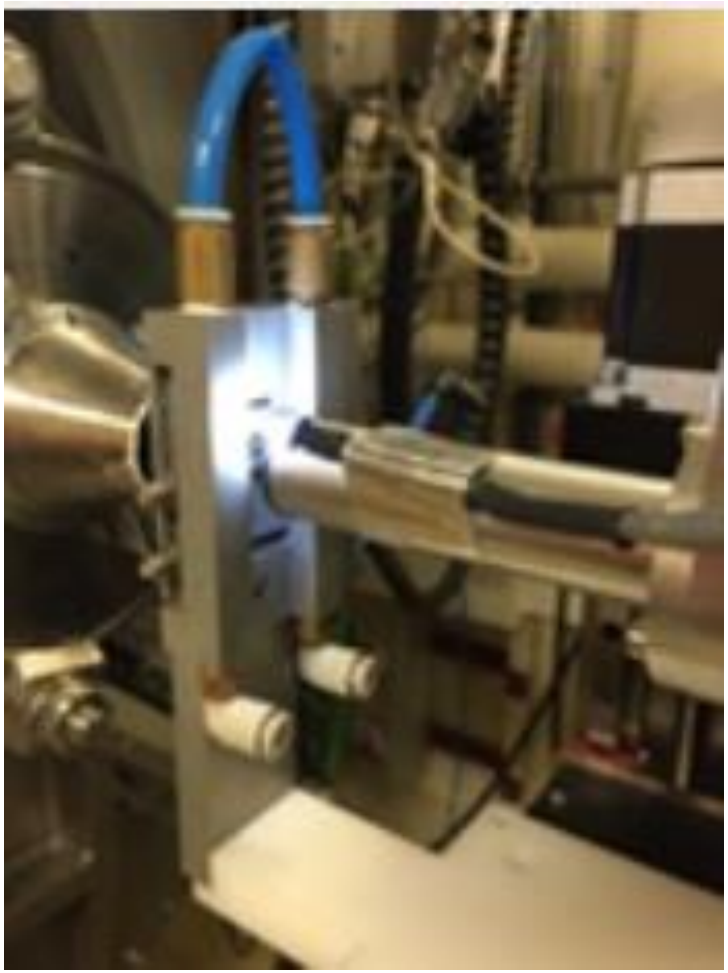
Mettler

Linkam



Temperature: single scan and flow-through

Flow-through (SWING version)

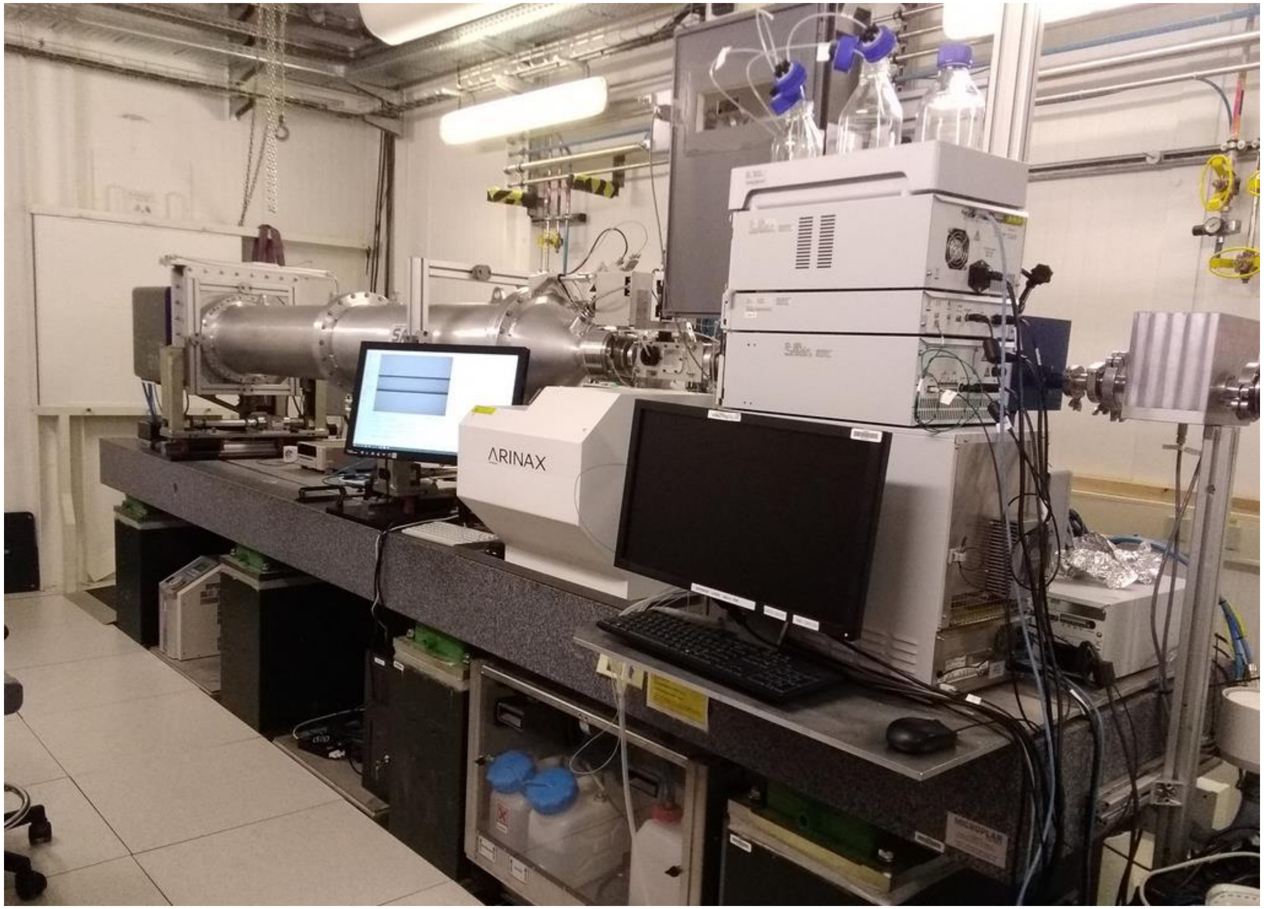


High throughput flow-through

Fully automated robotic sample changer

Advantages
Full automation
Many samples (hundreds)

Problems
Liquid to slightly viscous samples only
One distance (dedicated beamlines)
Setup (multiuse beamlines)

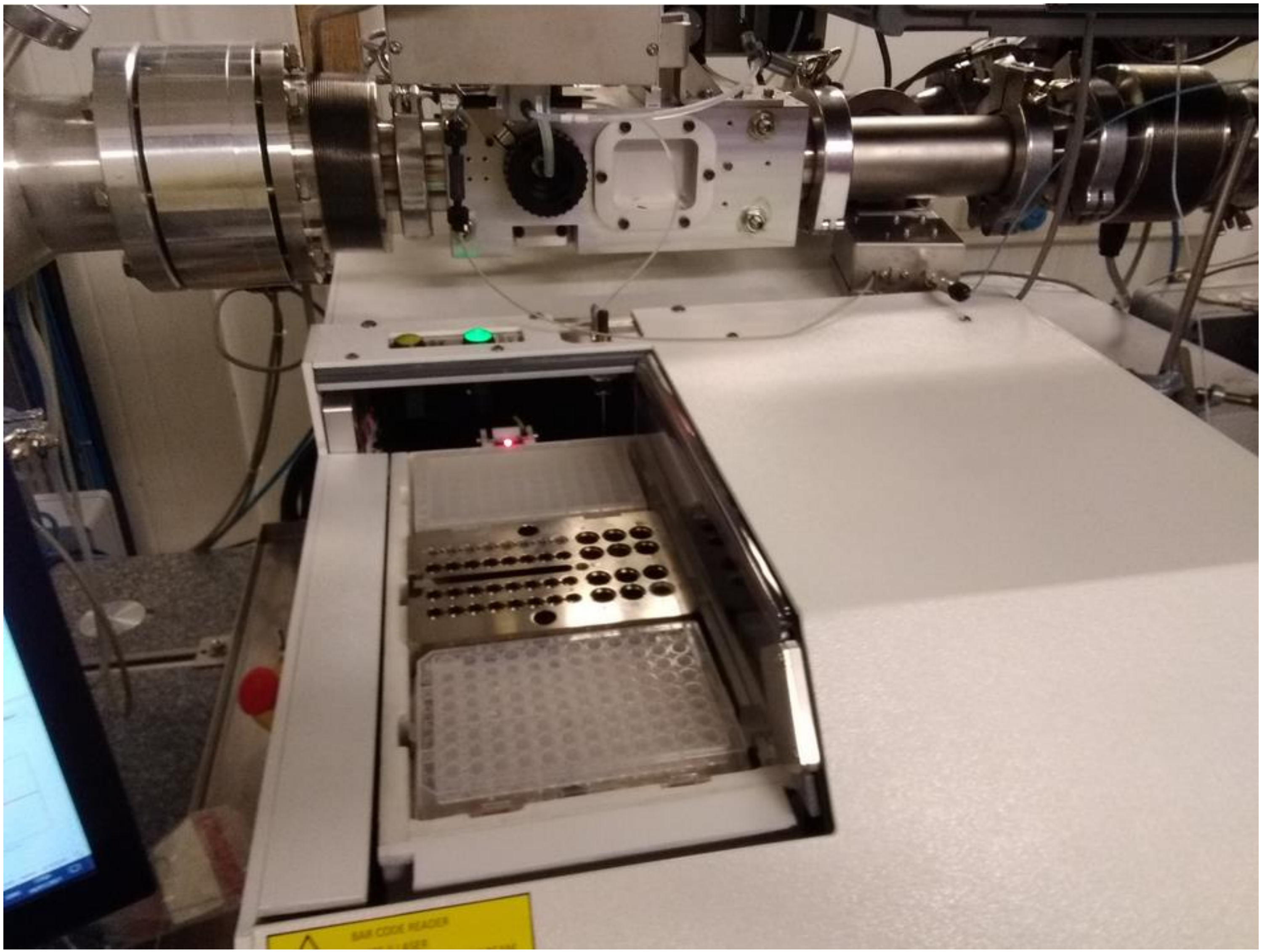


BM29 beamline ESRF

Existing as fixed device: Diamond (UK), BM29 (ESRF): Ready to use
Existing as mountable device: SWING (SOLEIL), ID02 (ESRF): installation/testing time

High throughput flow-through

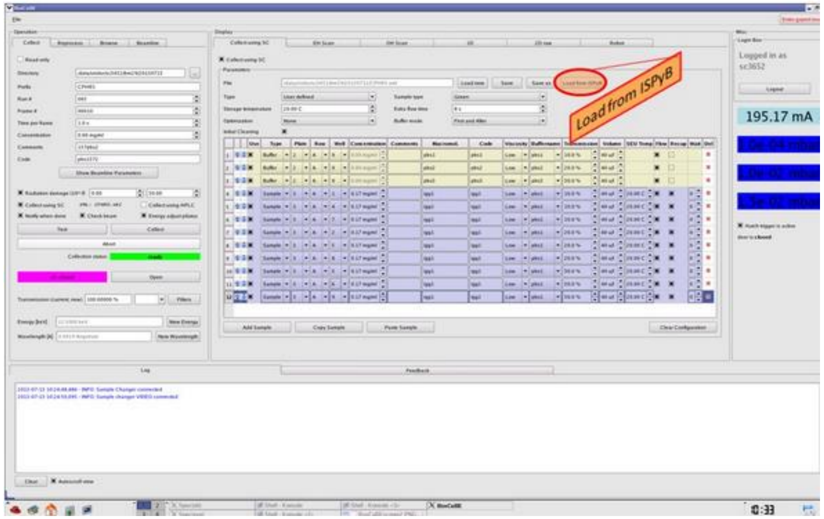
Fully automated robotic sample changer



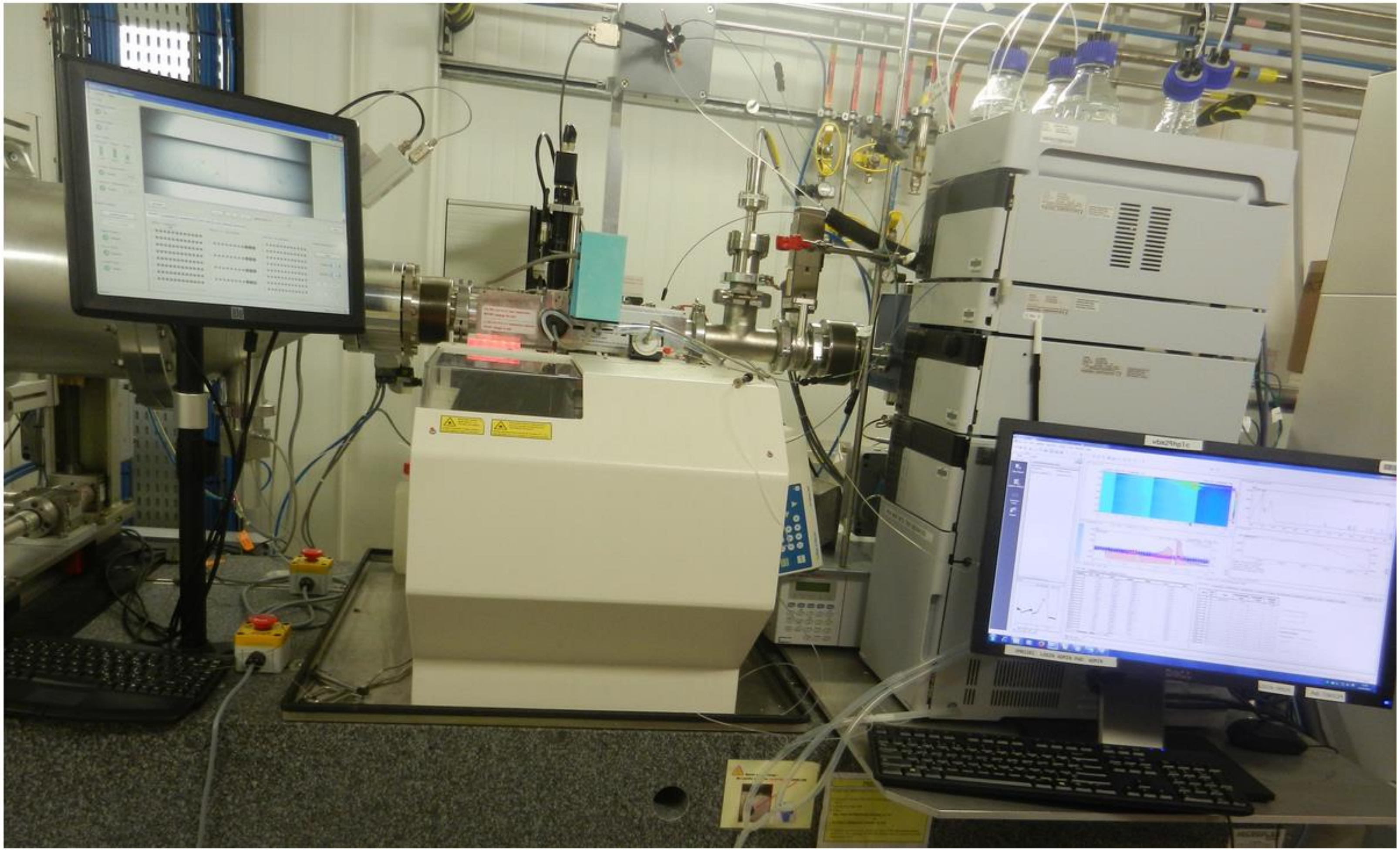
BM29 beamline ESRF

High throughput flow-through

Fully automated robotic sample changer



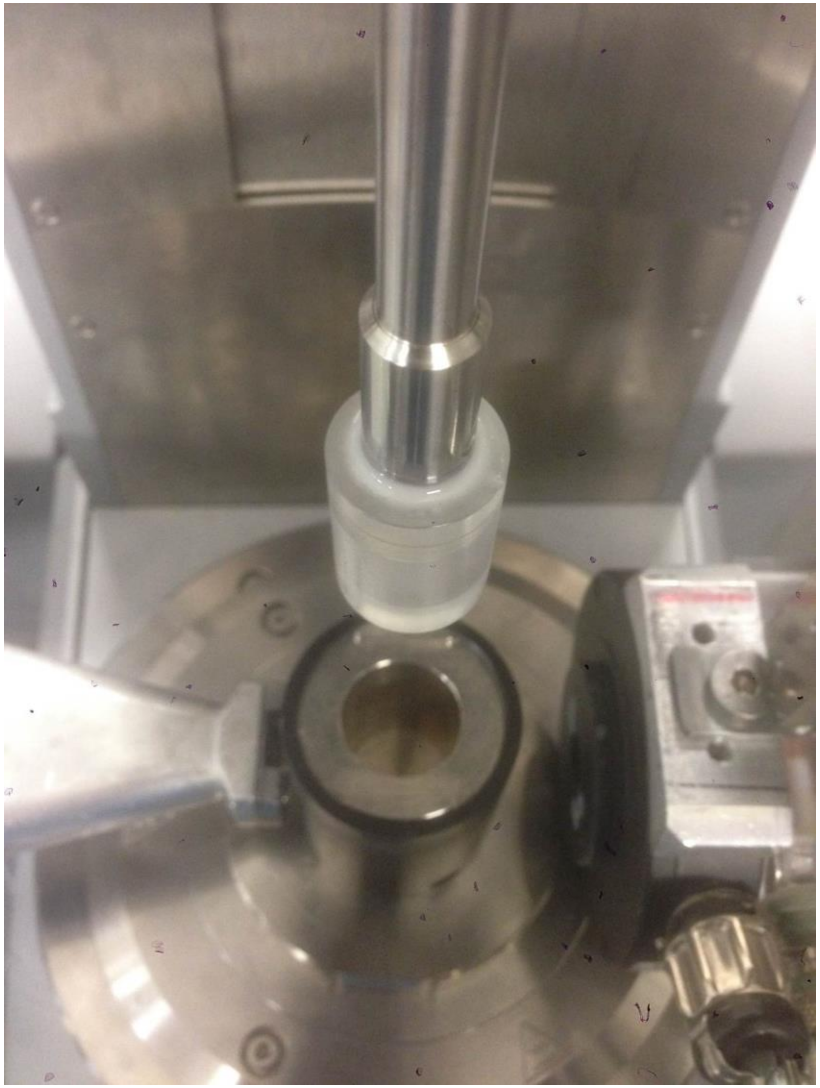
Size Exclusion Chromatography flow-through



Existing as fixed device: Diamond (UK), BM29 (ESRF): Ready to use
Existing as mountable device: SWING (SOLEIL): installation/testing time

Less common sample environments

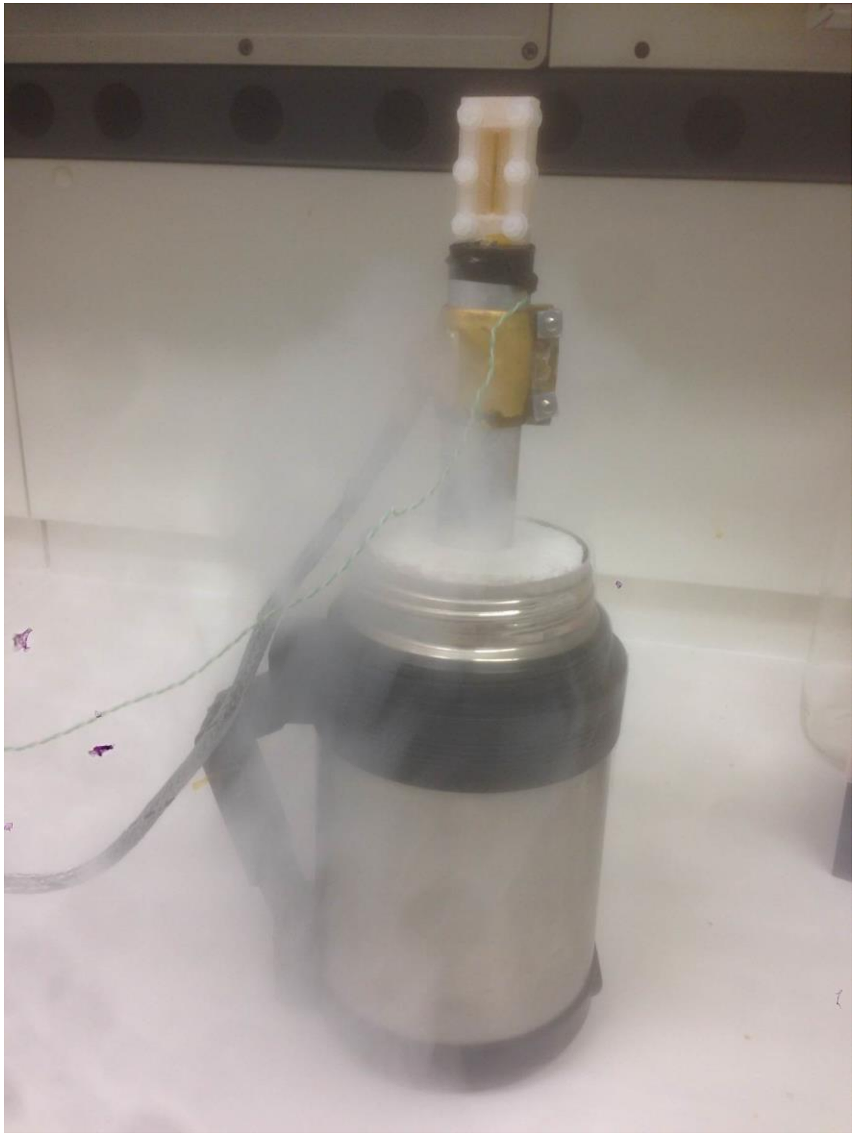
Rheo-SAXS



Existing as mountable device: SWING (SOLEIL), ID02 (ESRF)

A ajouter: Exemple donnée SAXS + Rhéo

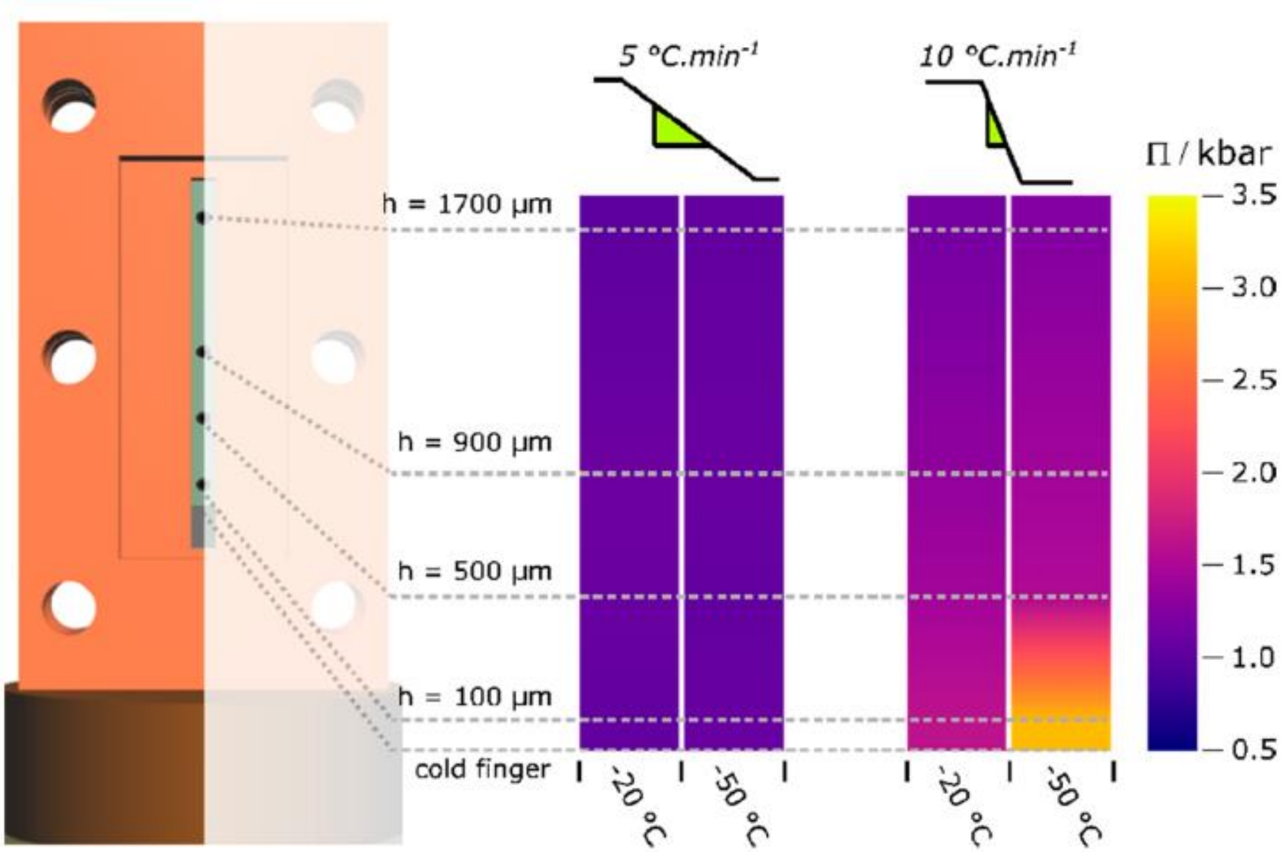
Freezing-SAXS



10.1039/c9mh00371a

Home made

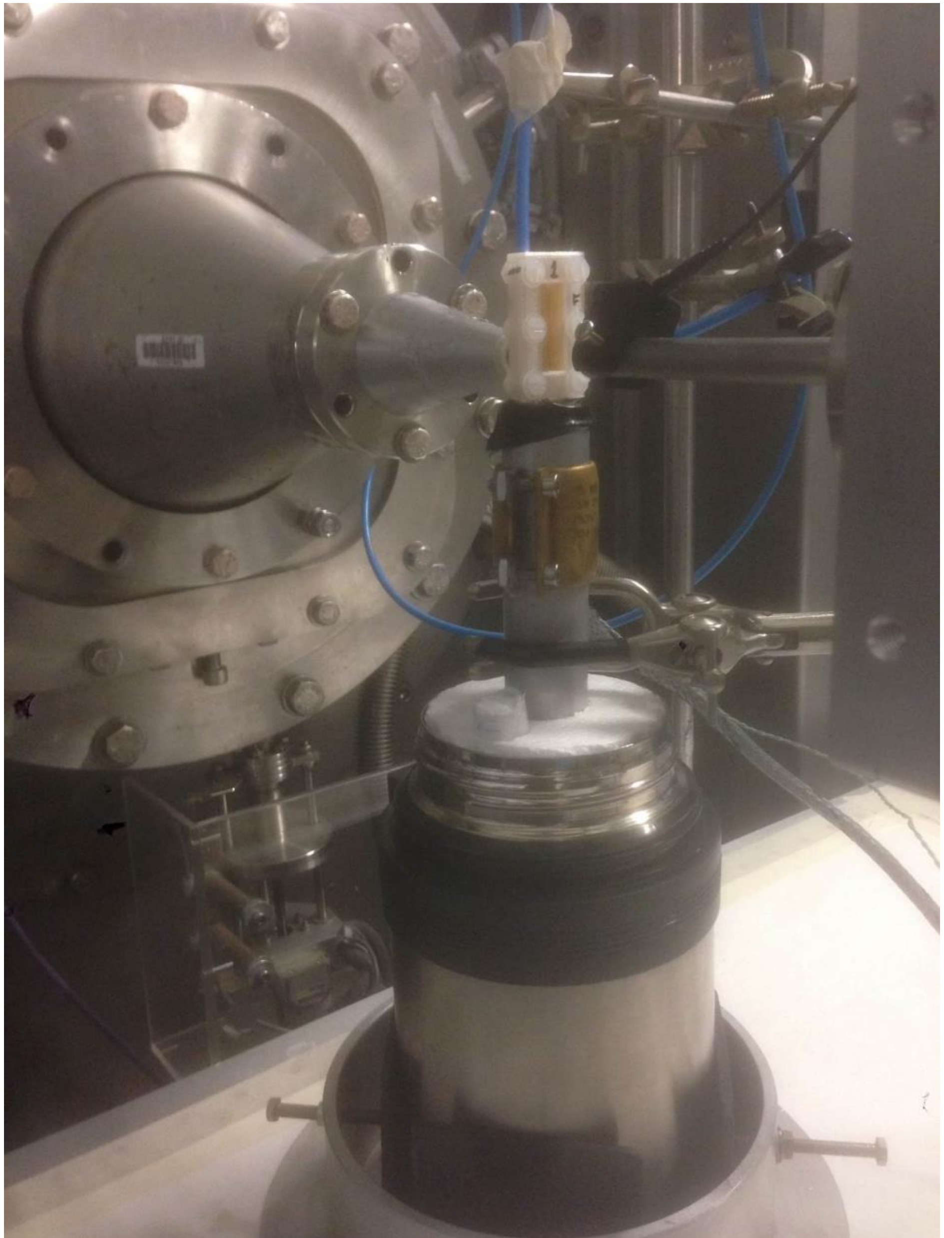
Freezing-SAXS



10.1039/c9mh00371a

Home made

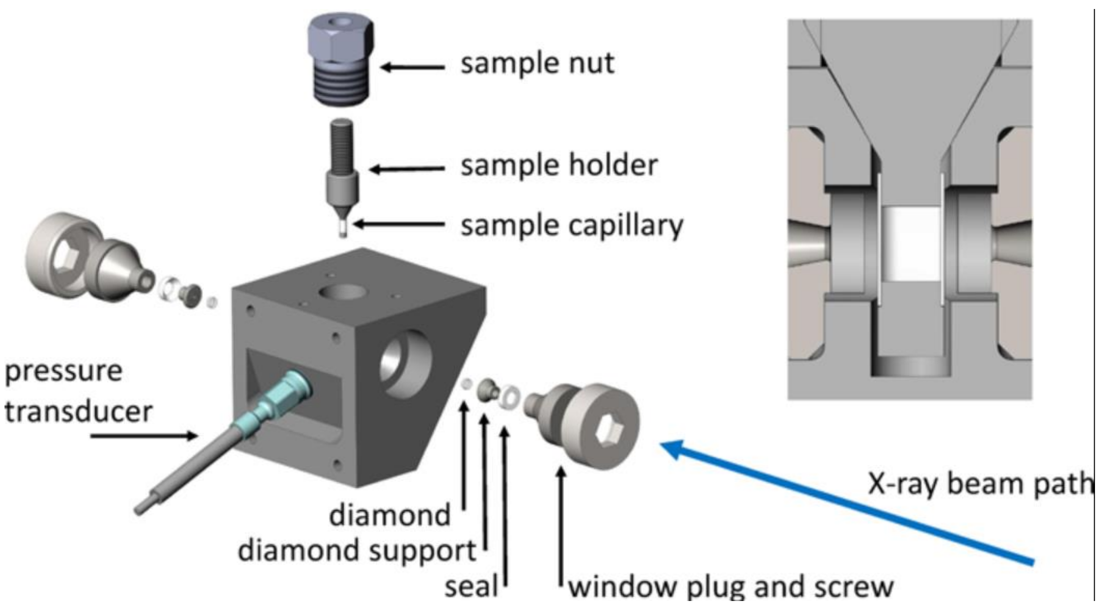
Freezing-SAXS

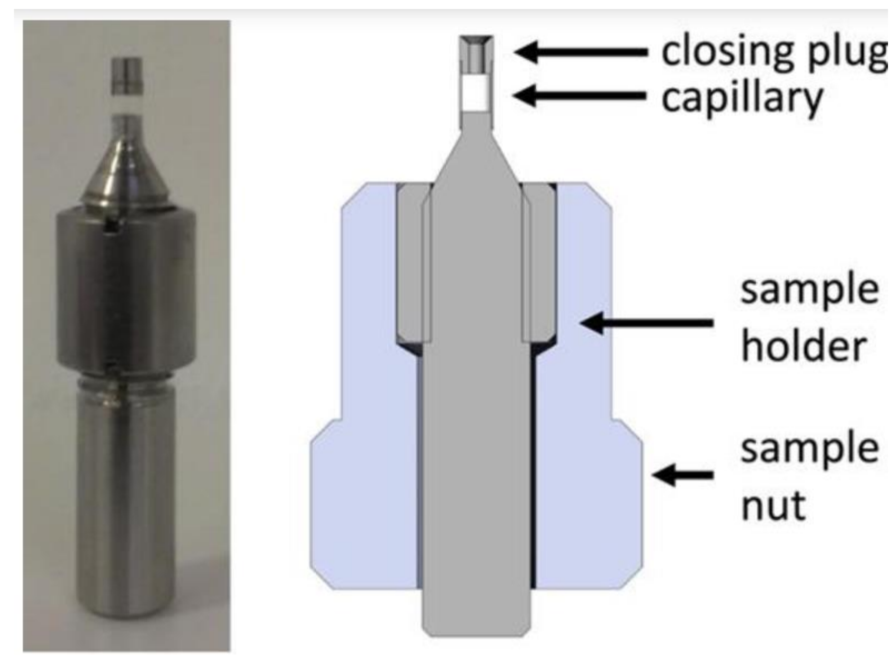


Home made

10.1039/c9mh00371a

Homeostatic pressure-SAXS

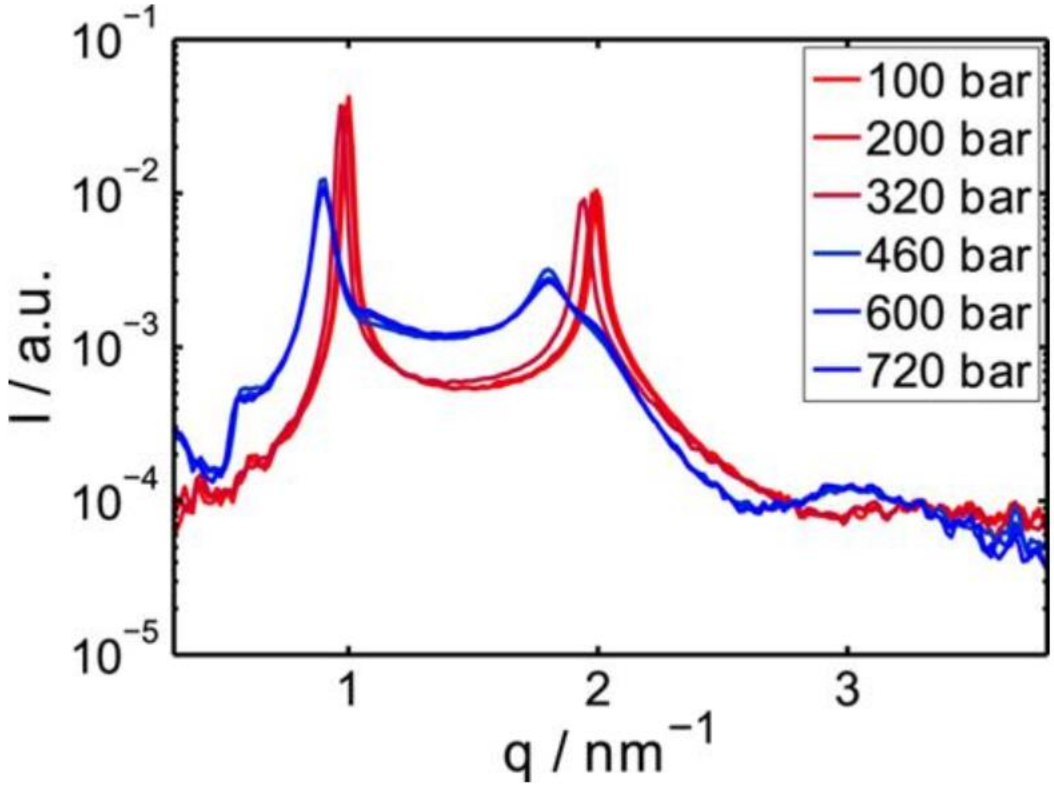




ID02 (ESRF)

Rev. Sci. Instrum. 87, 125116 (2016)

Homeostatic pressure-SAXS



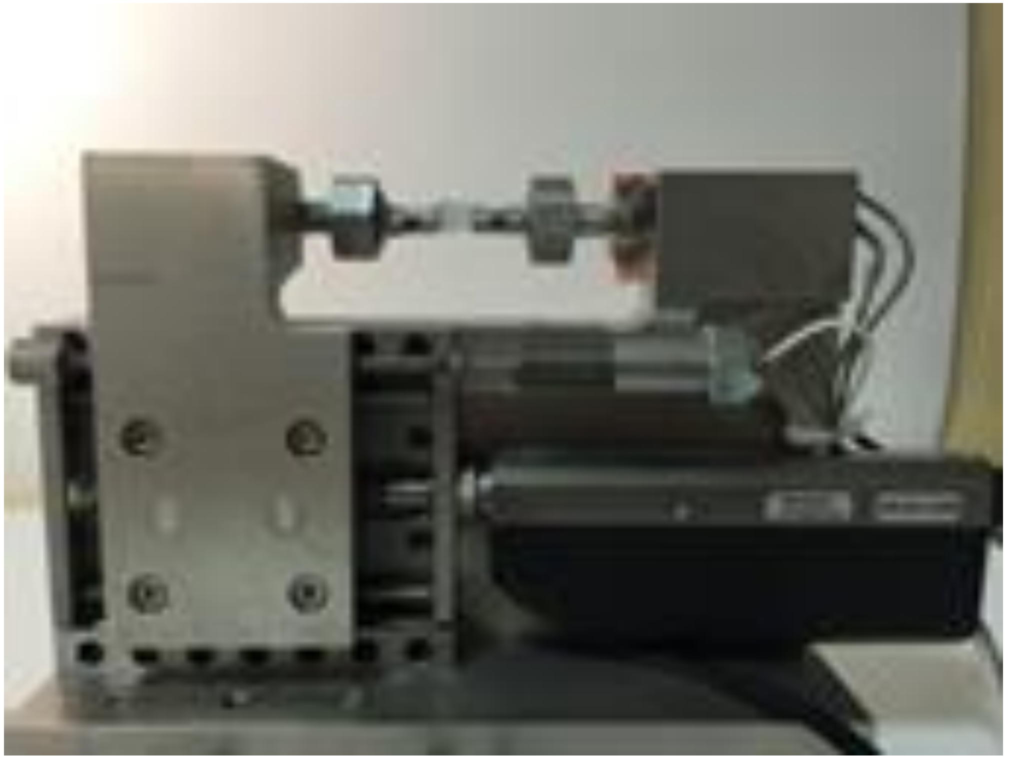
VIEW LARGE DOWNLOAD SLIDE

Static SAXS measurements of 20 wt. % DMPC at 30 °C as a function of pressure. The chain melting transition occurs between 320 and 460 bars.

ID02 (ESRF)

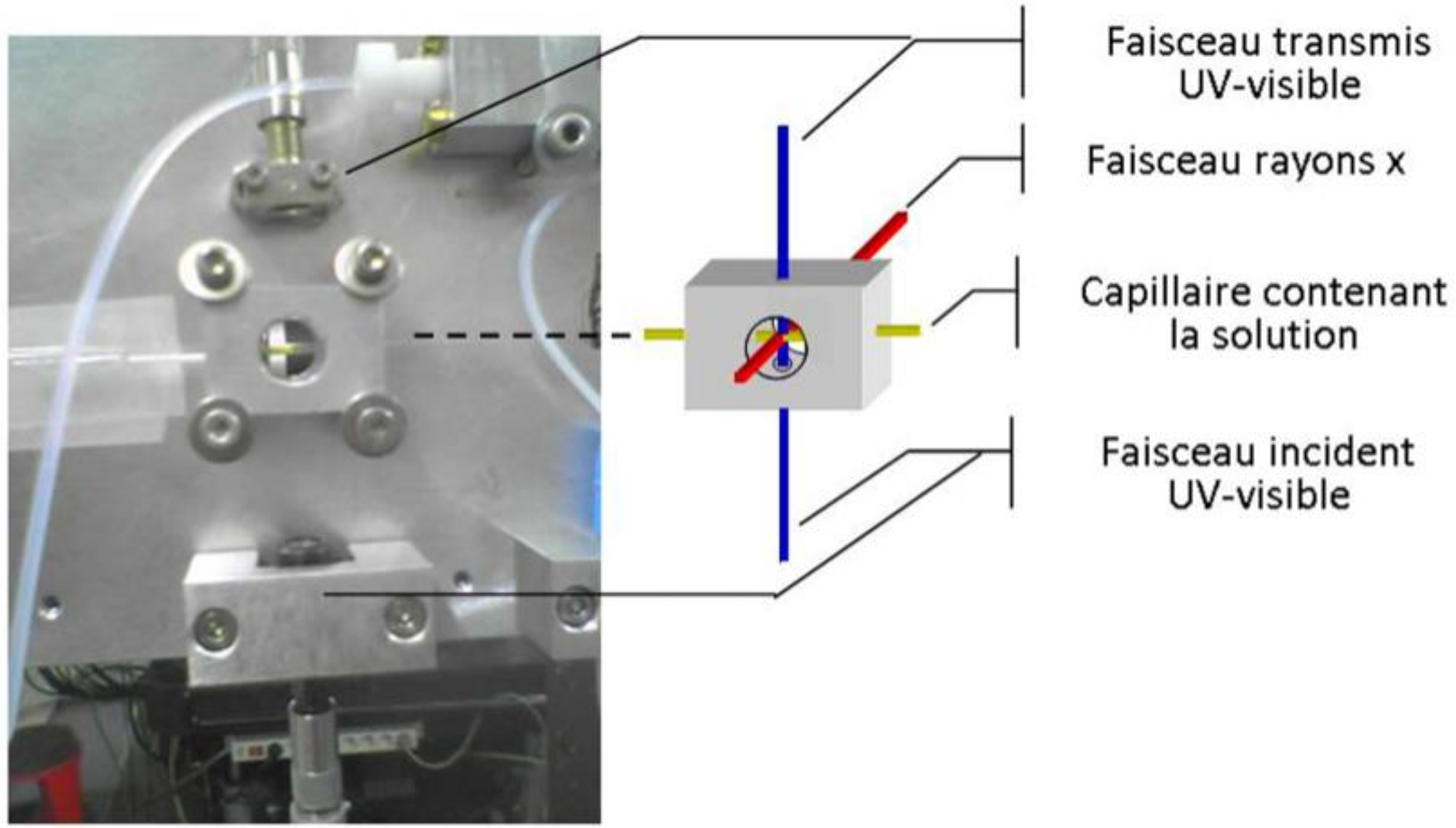
Rev. Sci. Instrum. 87, 125116 (2016)

Traction cell-SAXS



SWING (SOLEIL)

UV-Vis-SAXS



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UV-Vis-SAXS

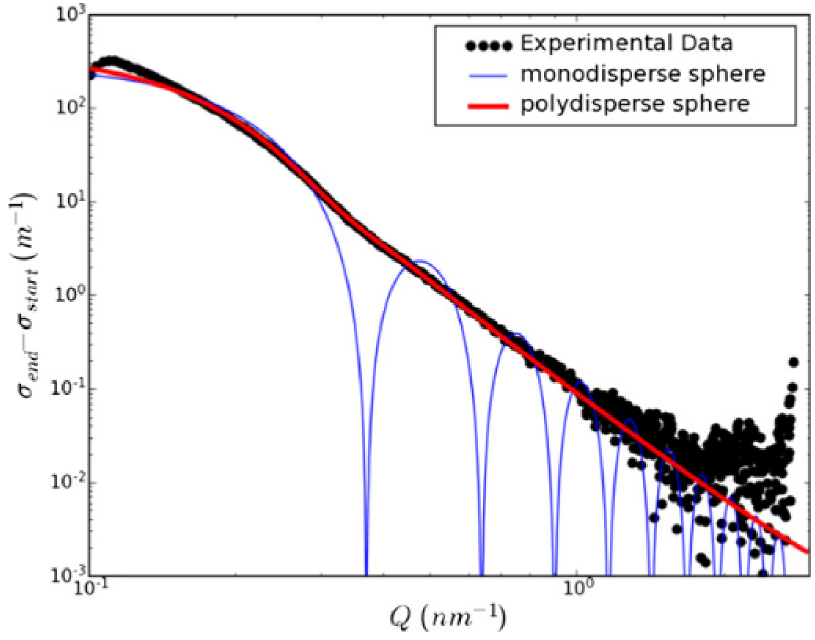

Fig. 2. Scattering cross section of silver nano-particles formed by X-ray radiolysis of a silver sulfate and ethanol solution at the end of the irradiation. The points are the experimental SAXS data. The thin line is a fit using a monodisperse sphere model with the radius $R = 14.5$ nm. The thick line is the scattering cross section calculated for polydisperse spheres with a mean radius of $R_0 = 14.5$ nm and a polydispersity ratio of $\eta = w/R_0 = 0.295$ where w is the width of the gaussian size distribution.

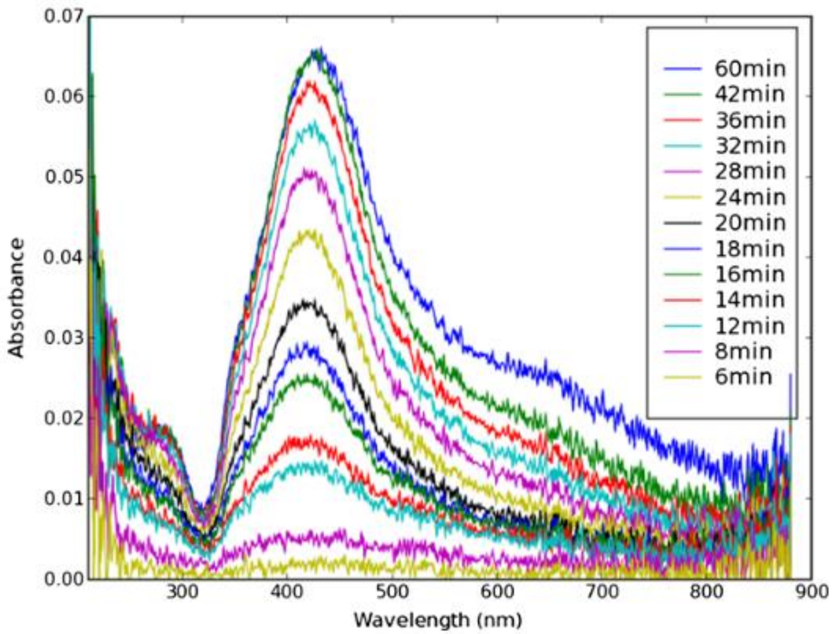

Fig. 3(a). Time evolution of the UV–visible absorption spectrum during the X-ray irradiation of an aqueous solution of silver sulfate. The optical length is 1.5 mm.

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Conclusion

Read papers and discuss with colleagues

Collaborate with scattering scientists

Be creative in analyzing your system

Be curious in discovering the beamlines around you and afar

Be motivated to set up your own environment for your own needs

Use old and new technologies (3D printing…)