Time resolved SANS combined with a stopped flow equipment

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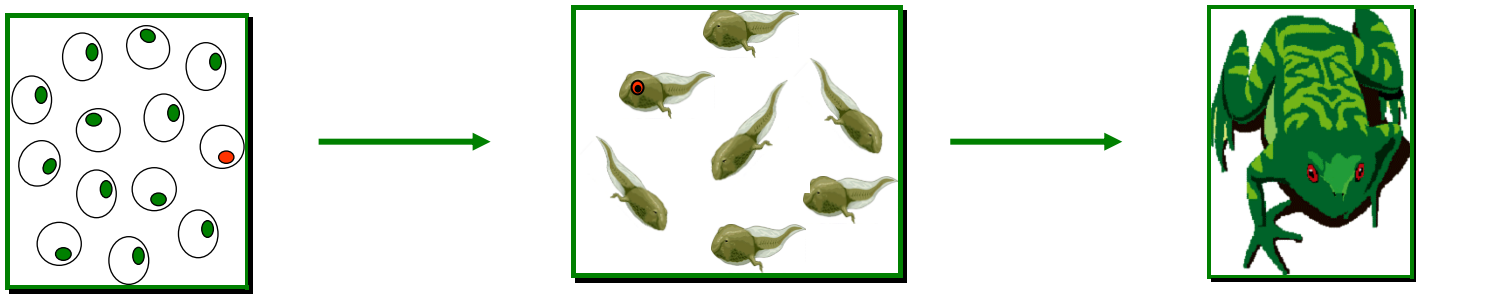
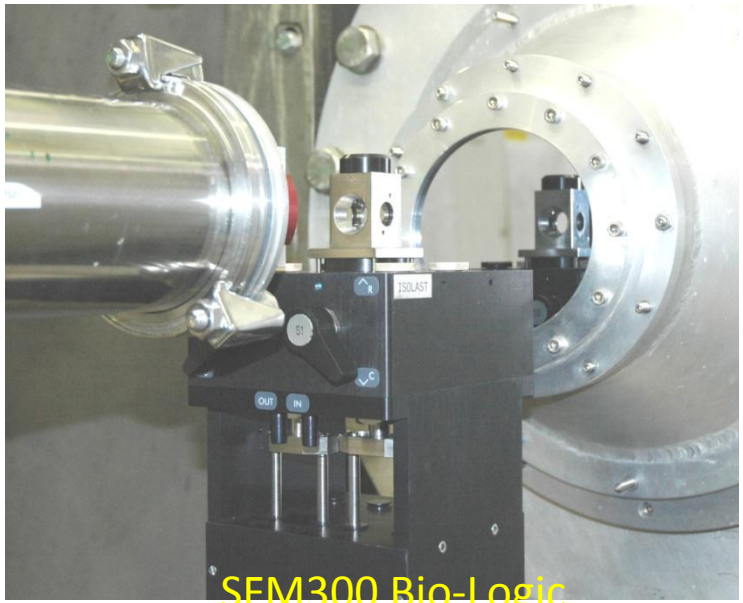


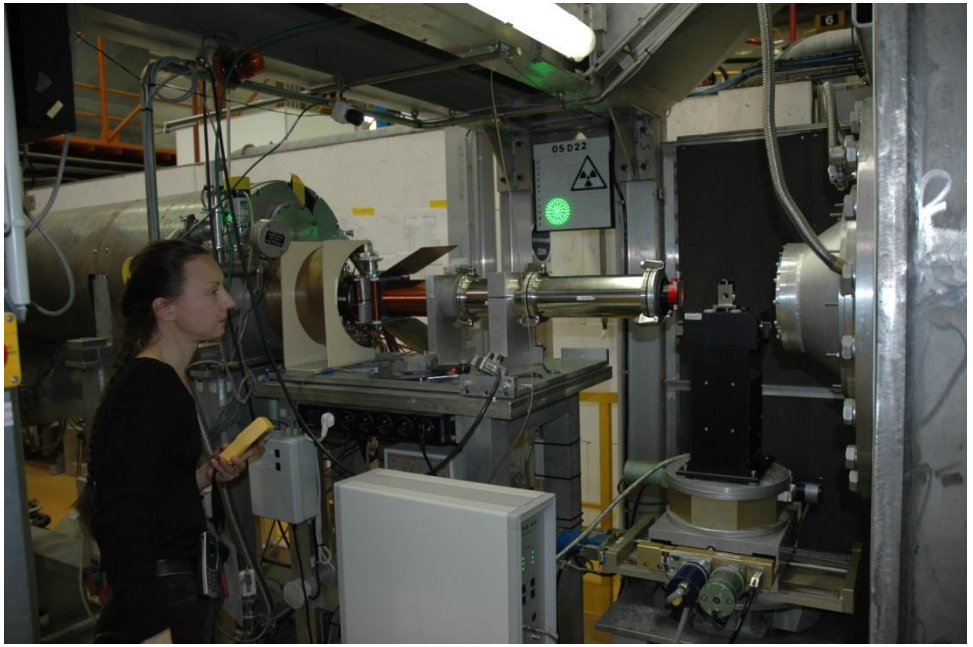
t_0 t_1, intermediate state t_∞, equilibrium state

Stopped-Flow equipment

SFM 300 from Bio-logic with 3 syringes controlled by step motors



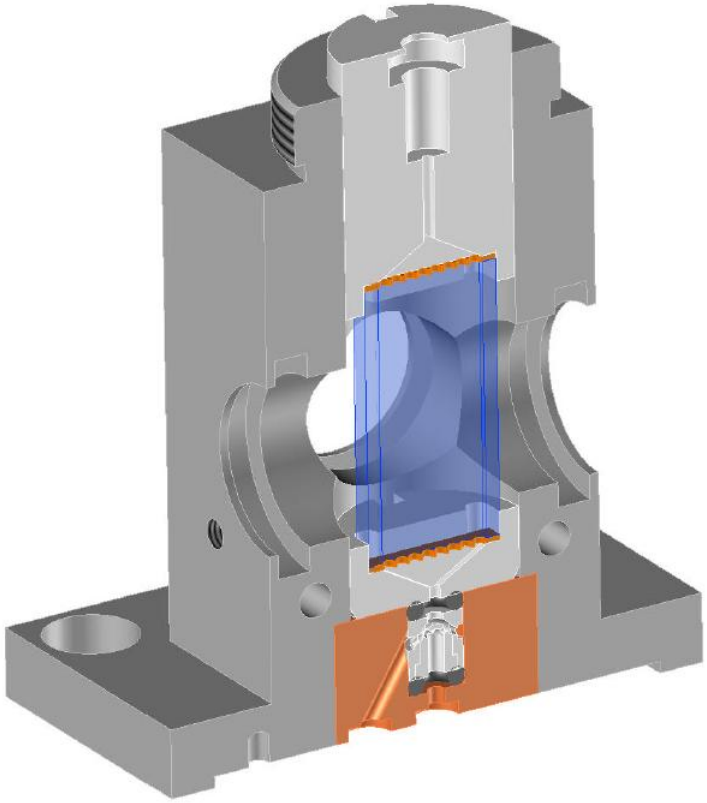
SFM300 Bio-Logic

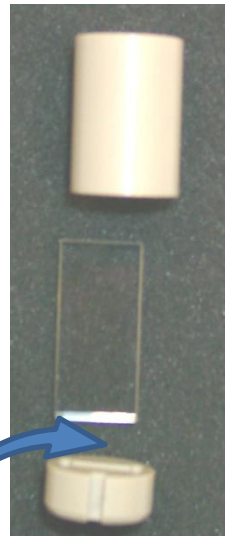


Interest of a SF device

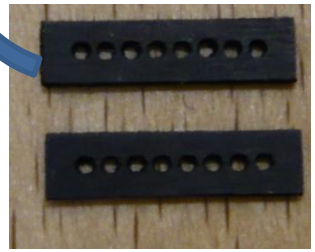
- precise control of volumes, flow rates and times of mixing
- synchronization of mixing with the beginning of the acquisition
- **reproducibility**

Observation Head specially designed for SANS





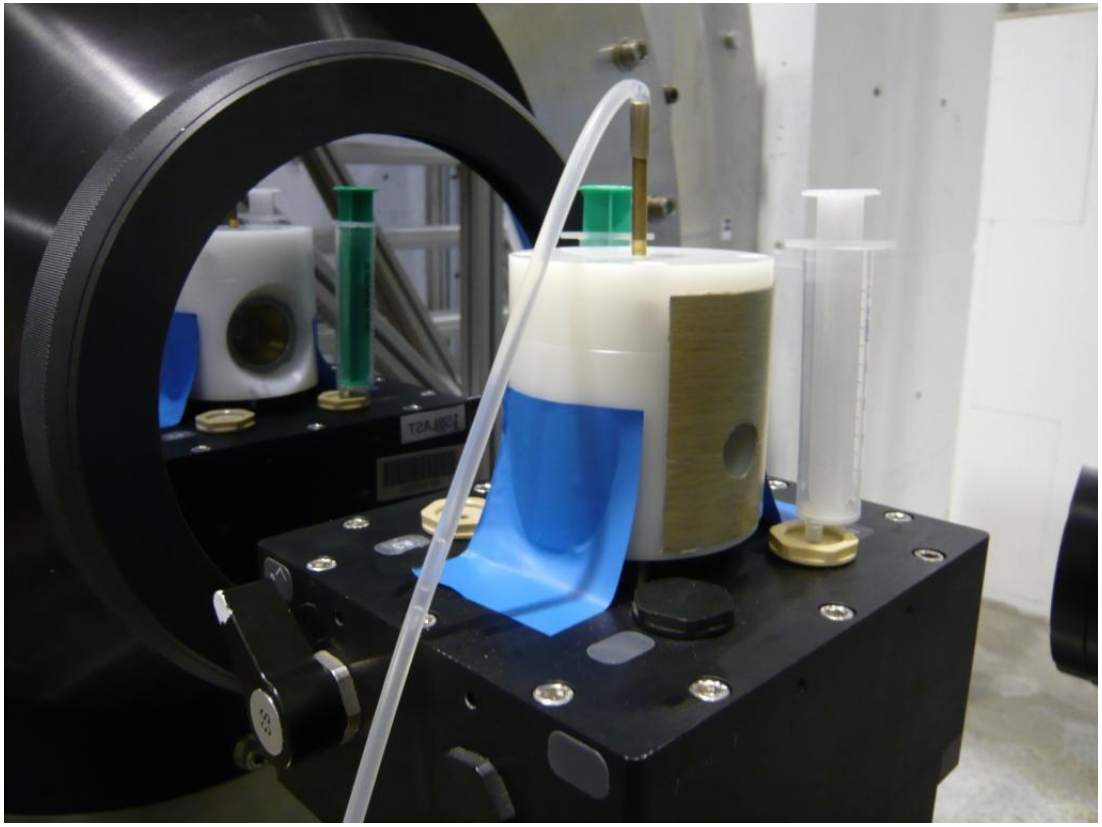
Hellma cell open at the 2 edges
10 x 25 x 1 mm^3
Ref cat. Hellma 690.225-QS

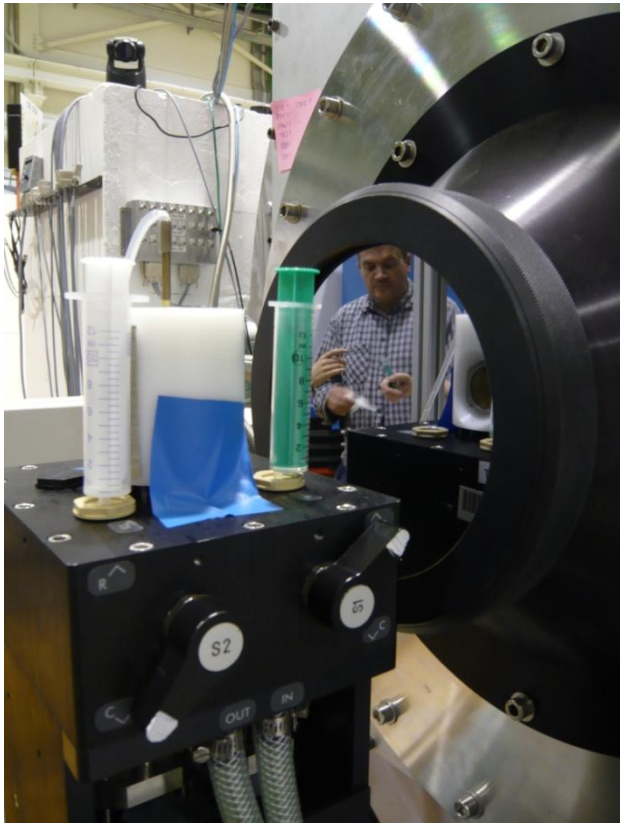


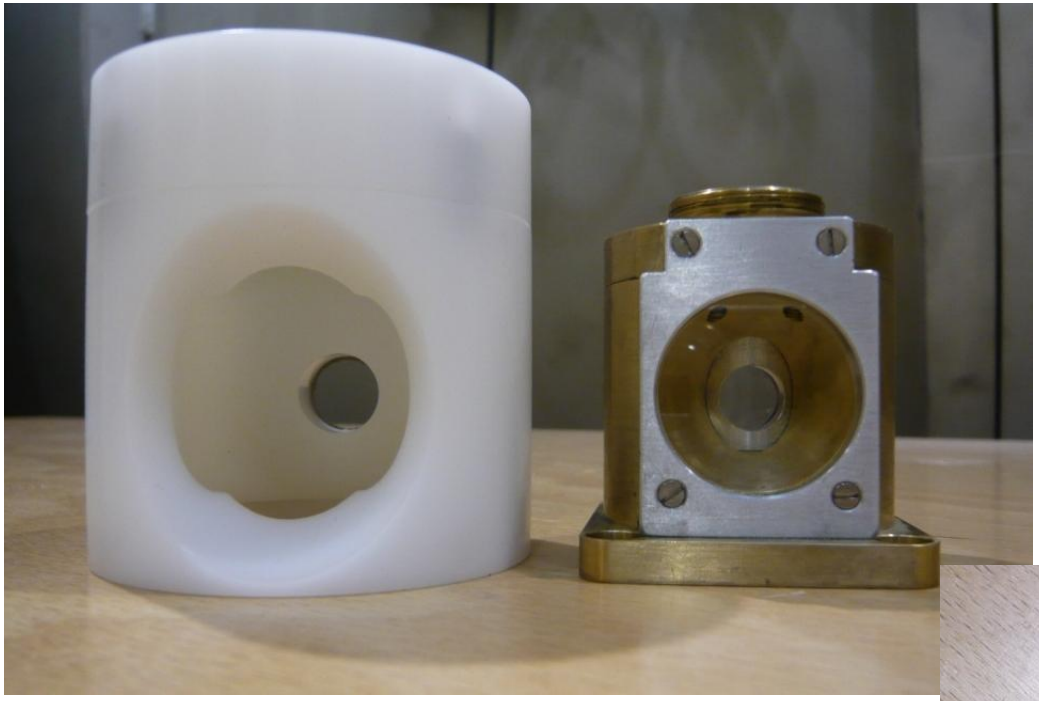
Sealing (Isolast®)

New observation head with a precise temperature control

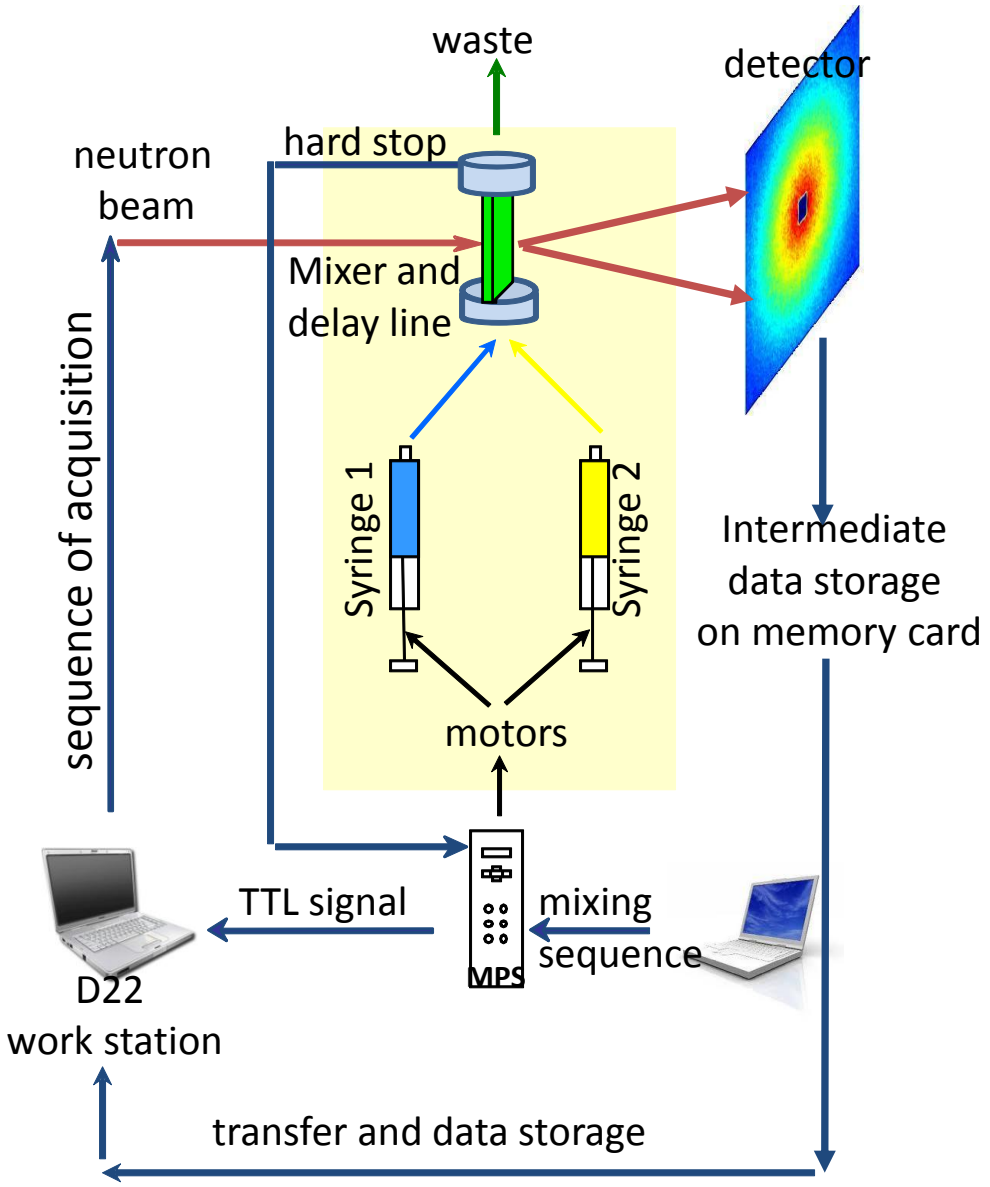
(Thomas Sottmann, Institut für Physikalische Chemie, Köln)







Principle of a real time SANS experiment



Acquisition time

-t_{min} 10 – 100 ms per frame

Dead time (time to fill the cell)

-50 – 150 ms

Acquisition sequences

- constant time t1 =t2= .. =tn
- geometric series $t_n=t_1\ r^{n-1}$
-any other personal choice

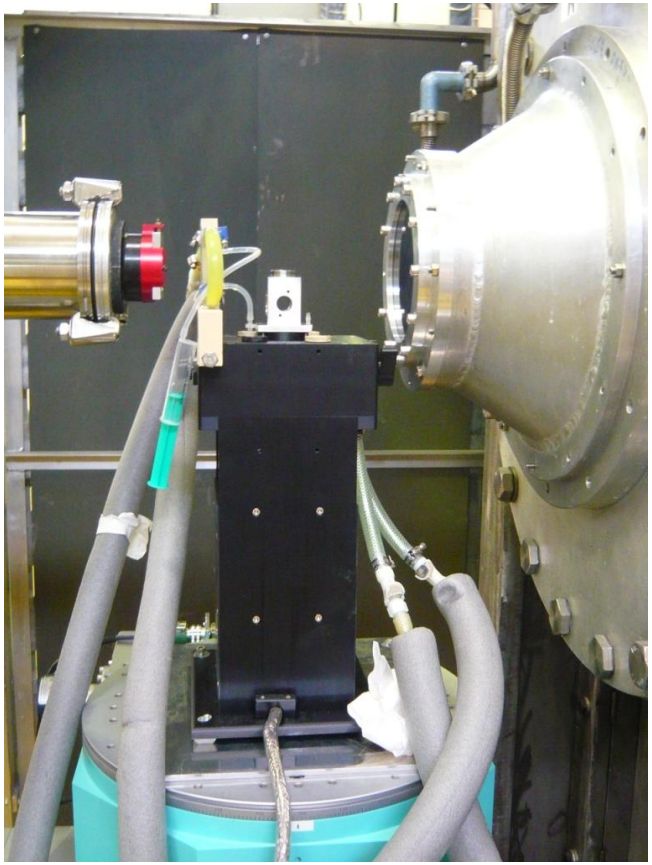
Electronics

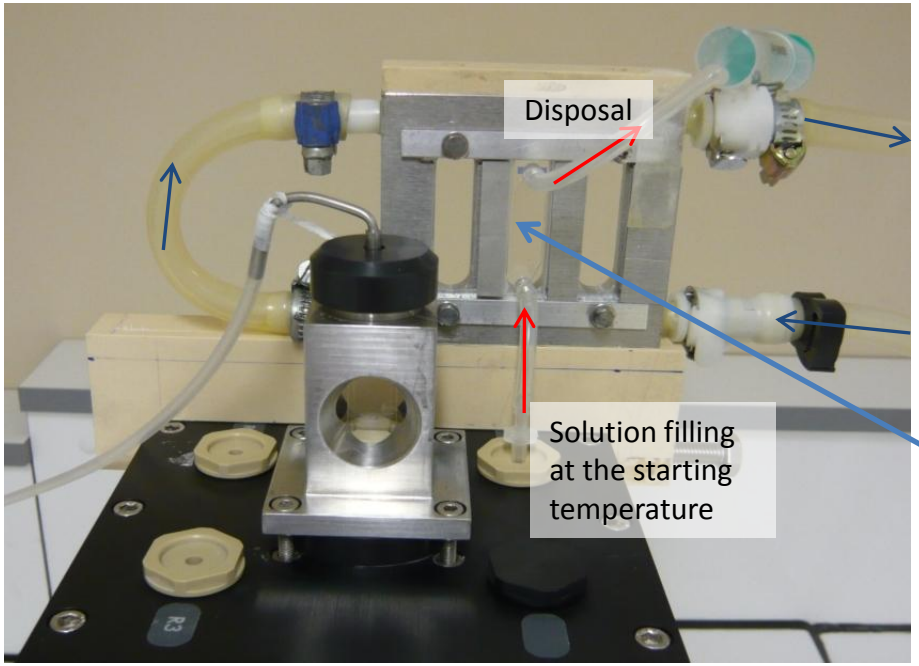
- up to 450 frames for the intermediate storage
- no dead time between two frames

Cycling

- increase of statistics

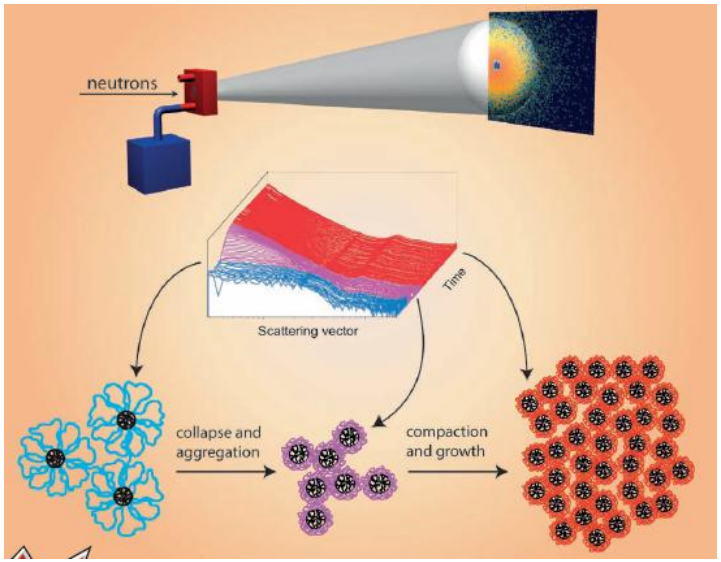
Side use of the stopped-flow equipment for T-jump





Water thermalization to the target temperature

Neutron beam



Kinetics of Collapse Transition and Cluster Formation in a Thermoresponsive Micellar Solution of P(S- *b* -*NIPAM*- *b* -*S) Induced by a* Temperature Jump
J. Adelsberger, C. Papadakis et Al, Macromol. Rapid Commun. 2012, 33, 254–259

Papers published in the last decade (D11 , D22)

Mesoporous materials

How does ZrO_2/surfactant mesophase nucleate? Formation and mechanism
Né et al, Langmuir, 2003, **19**, 8510
Growth of mesoporous silica nanoparticles monitored by time-resolved small-angle neutron scattering
Hollamby et al Langmuir 2012, **28**, 4425

Surfactant systems

Formation and growth of anionic vesicles
Grillo et al, Langmuir 2003, 19, 4573
Monomer-aggregate exchange rates in mixed di-alkyl chain cationic-nonionic surfactant microstructures
Tucker et al, Langmuir, 2009, **25**, 2661
Time-resolved small-angle neutron scattering as a lamellar phase evolves into a microemulsion
Tabor et al, Soft Matter, 2009, **5**, 2125
Mesodynamics: watching vesicle formation in situ by small-angle neutron scattering
Bressel et al, Colloid Polym Sci, 2010, **288**, 827

Polymers

Equilibrium Chain Exchange Kinetics of Cylindrical and Spherical Diblock Copolymer Micelles
Lund et al, Macromolecules, 2011, **44**, 6145
Rupture of pluronic micelles by di-methylated β-cyclodextrin is not due to polypseudorotaxane formation
Valera et al, J Physical Chemistry B, 2012, **116**, 1273

Applications of stopped-flow in SAXS and SANS
Grillo, COCIS, 2009, **14**, 402 **(review paper)**

What do we need?

Improve the cell filling.

Cell volume 250 μL but 600 μL really needed to be sure to remove completely the old solution and replace it by the fresh solution.

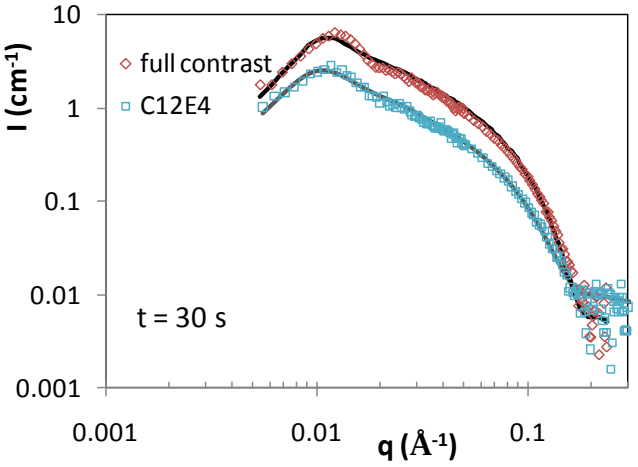
New cell geometry (not rectangular?)

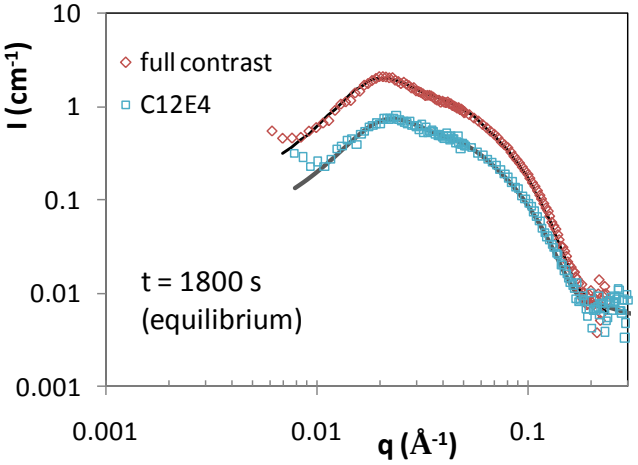
Different cell thicknesses

New furnace for T-jump.

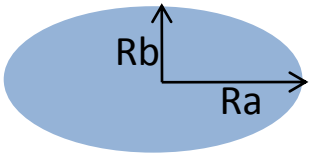
Any suggestions /need?

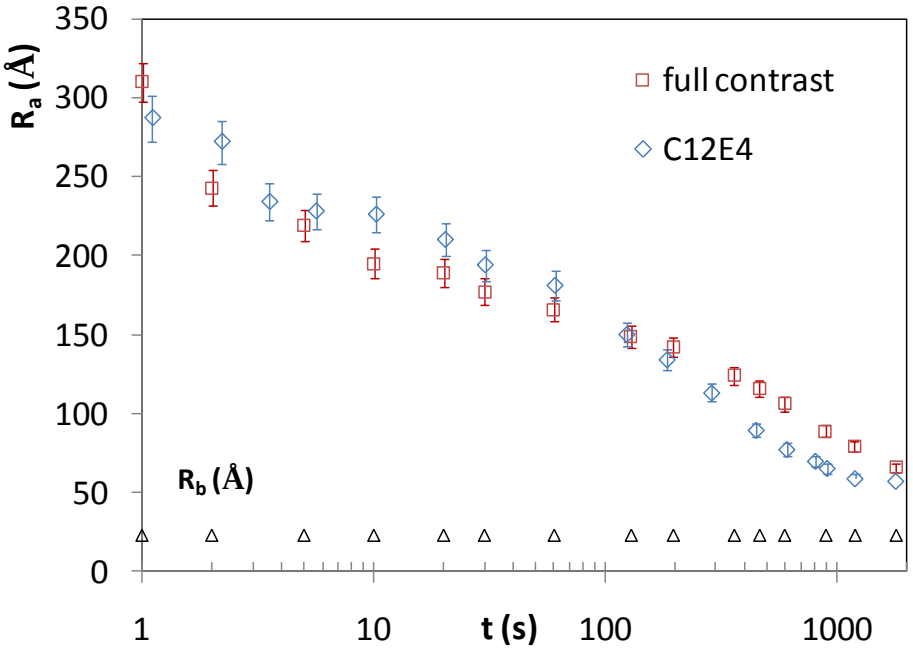
Size evolution with time





Oblate ellipsoid





- Continuous decrease of the rotation axis Ra down to 60 Å
- Rb remains constant at 23 Å, as for pure $C_{12}E_4$ worm-like micelles
- Equilibrium reached in 30 min