SANS under high pressure

overview and recent developments of pressure cells used at the ILL

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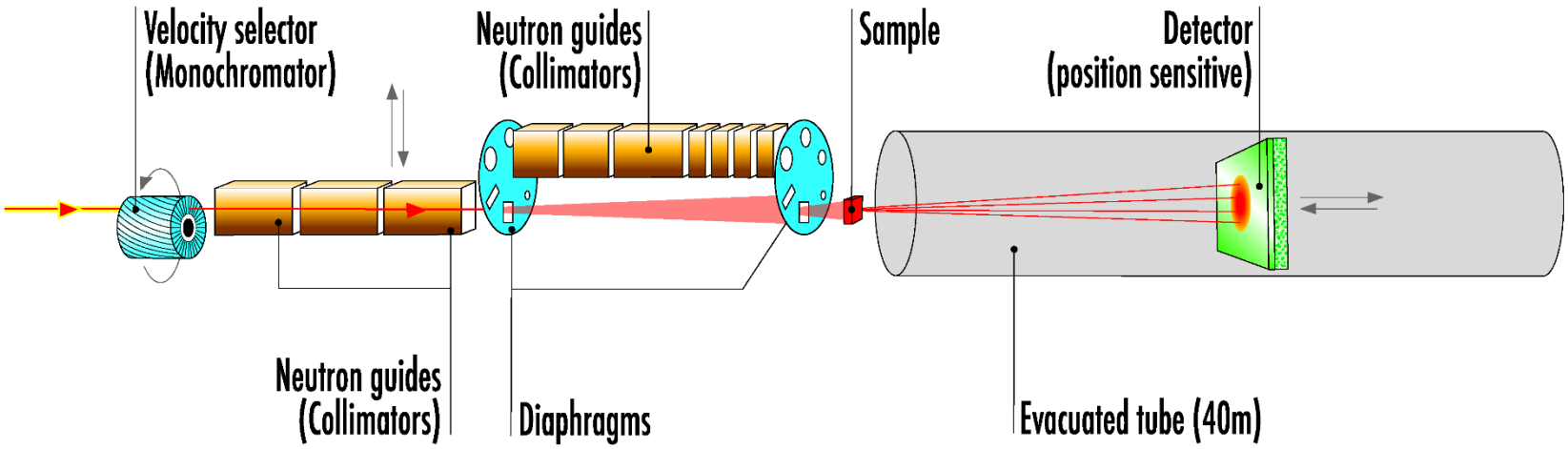
Small Angle Neutron Scattering

The ILL operates 3 SANS instruments: D11, D22, D33

Layout of D11



$2 \cdot 10^{-4}$ Å^{-1} < q < 0.8 Å^{-1}

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E. Lelièvre-Berna (ILL)

D. Bowyer, P. Lindner (ILL)

B. Annighöfer (LLB)

T. Sottmann and the members of the Strey group (U Cologne)

J. Peters et al. (hAChE project) (U Grenoble & ILL)

A. Gelissen, A. Steinschulte et al. (W. Richtering, F. Plamper and groups, RWTH Aachen)

The NMI3 WP20 program for (partial) funding and the discussion meetings

M. Lechner, K. Ibel (U Osnabrück, ILL)

Since mid 80´s

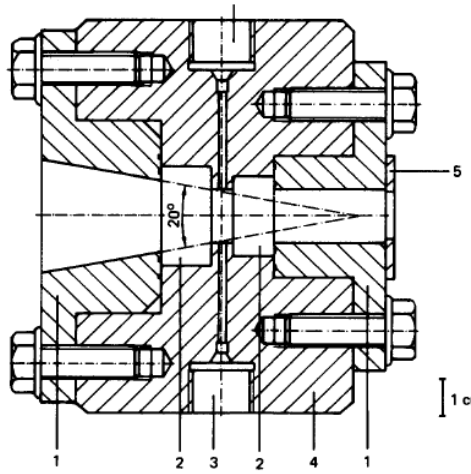
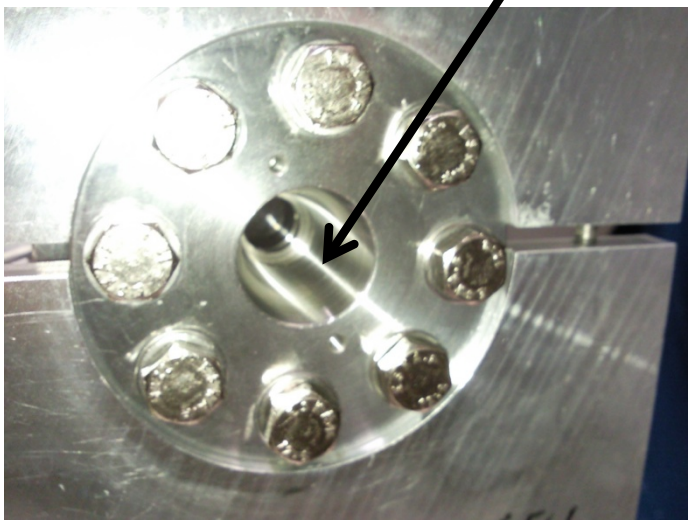
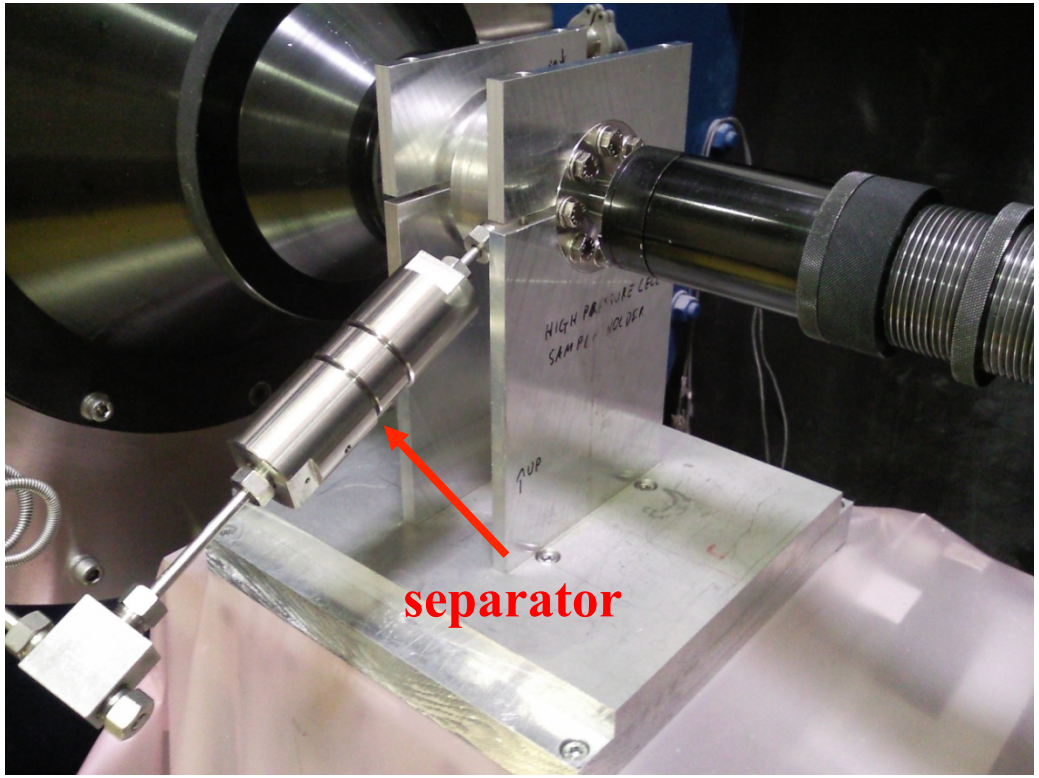


Figure 8 Cross section of the high-pressure cell: 1, steel cap; 2, sapphire windows; 3, pressure connection; 4, steel body; 5, cadmium diaphragm

(poor) T control above ≈ 30°C

restricted exit angle (20°)





Purchased from Lechner´s workshop

for liquid samples, hydrostatic pressure up to p = 2.5 kbar

electrical heating jacket -> T≈ 250 °C

sapphire windows (2 x 12mm) + 4 gold sealings

sample thickness 4.5mm, optional 2.5mm

M. Lechner, K. Ibel (U Osnabrück, ILL)

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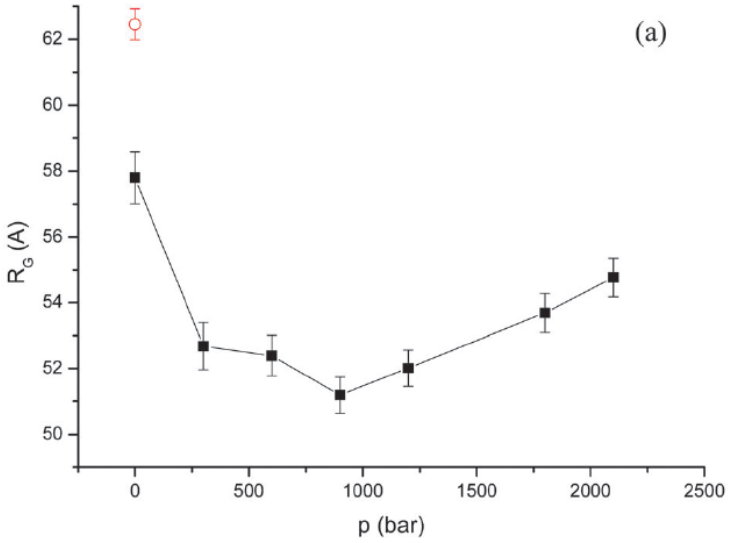
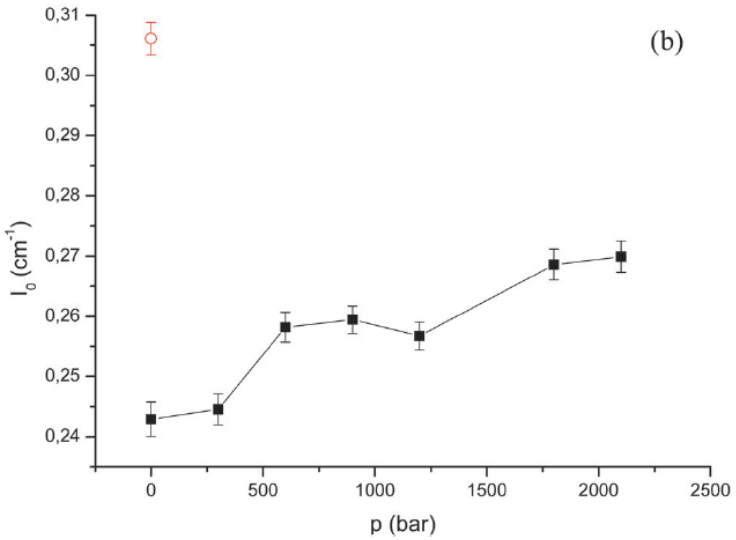
Pressure-induced molten globule state of human acetylcholinesterase: structural and dynamical changes monitored by neutron scattering†

J. Marion,‡[ab] M. Trovaslet,‡[ac] N. Martinez,[ab] P. Masson,[ad] R. Schweins,[b] F. Nachon,[ab] M. Trapp[ef] and J. Peters*[ab]

Radius of gyration vs. p



Izero vs. p



hAChE is a mixture of monomers, dimers, tetramers
compression until 900 bar, then expansion due to (partial) unfolding

M. Lechner, K. Ibel (U Osnabrück, ILL)

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Molten globule

intermediate state characterised by ...

absence of tertiary structure,

but secondary structure still present to a great extent

Rg increase between 10 and 30 %

EINS data from IN13

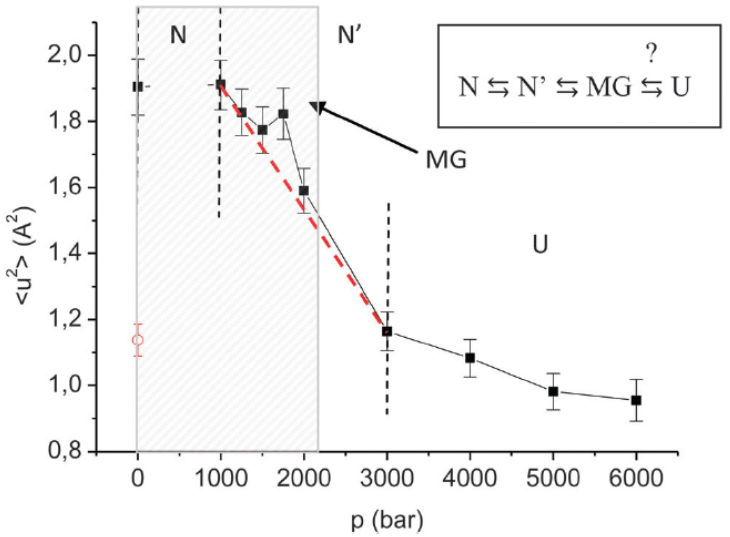
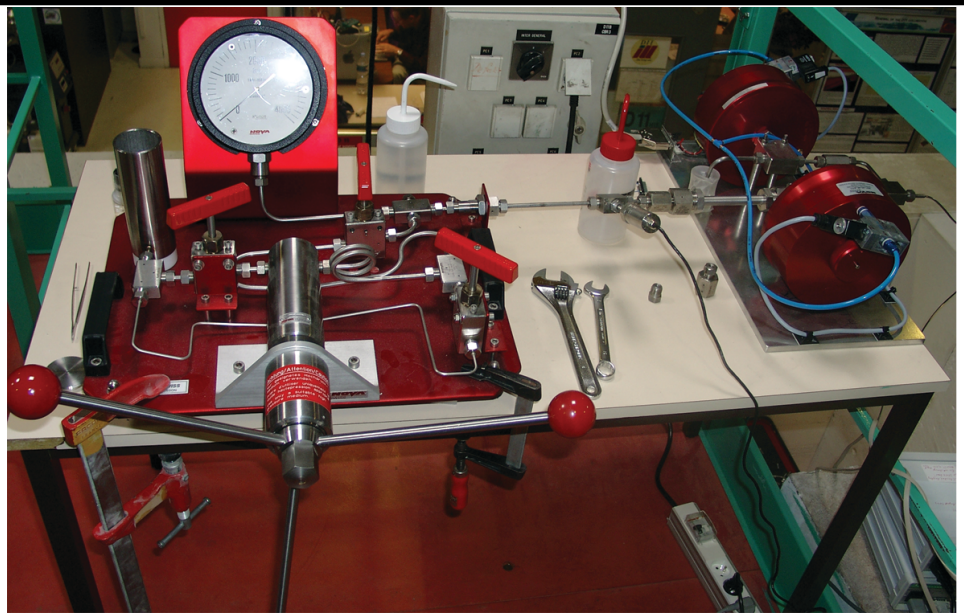
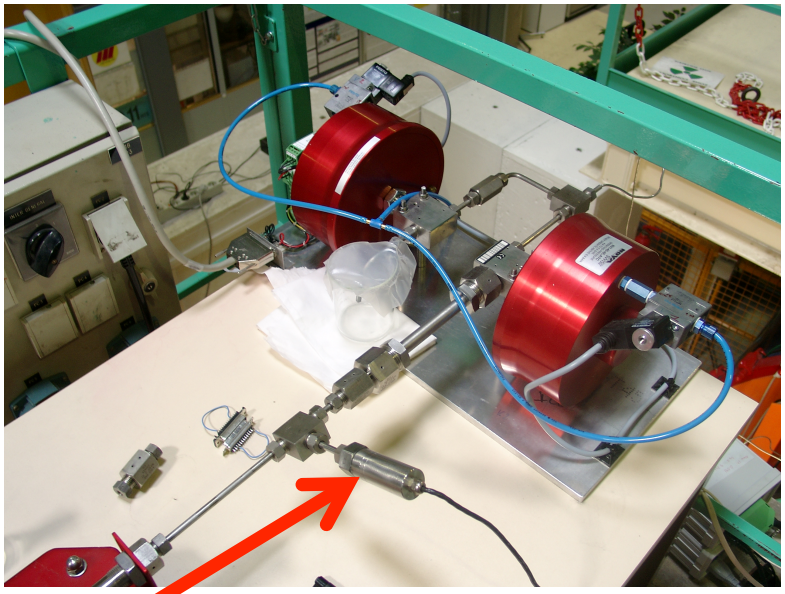


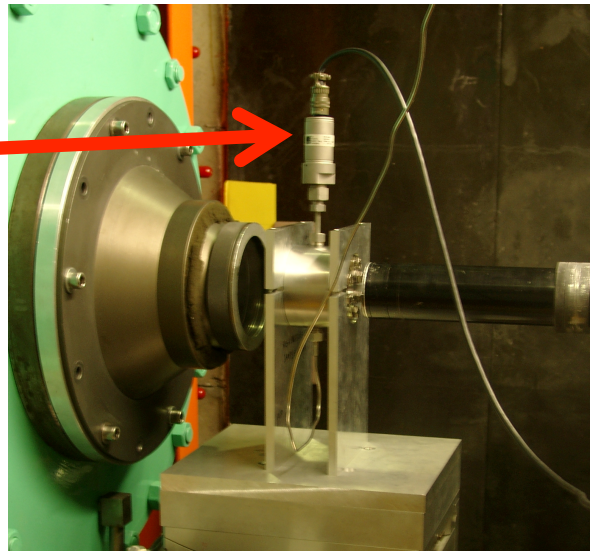
Fig. 4 Mean square displacement of hAChE, measured on IN13, at different pressures up to 6 kbar. The red point (open circle) corresponds to the MSD after releasing pressure. The red dashed line is guidance to the eye to illustrate the straight slope of this part of the curve. The dashed region corresponds to the pressure domain covered by the SANS measurement. The sketch on the right side illustrates the 4-step-model, N being the native, N′ the transitional intermediates, MG the molten globule and U the unfolded state.

Pressure jumps @ D11 (2003/04)



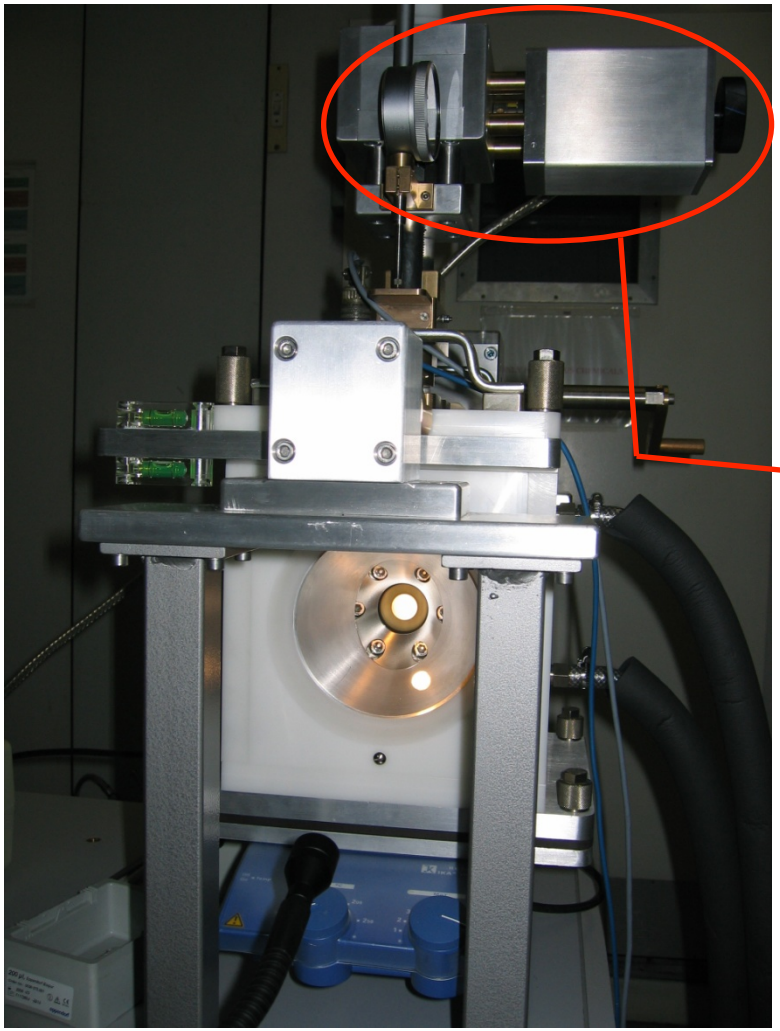


pressure sensors



hand pump to build up pressure
PC controlled automatic valves
PC readable pressure sensors
start of data acquisition via TTL of pressure sensor
(initial) dead time 100 msec
used with Lechner / Ibel cell ($p < 2500$ bar)

Cologne pressure cell



Strey, Sottmann (U Cologne) & D11

Design discussion in 2006, construction done in the university workshop at the university Cologne

first experiment on D11 in spring 2007

Sample can be homogenized with magnetic stirrer

Characteristics:

Pressure applied via a plunger / stepper motor

!!! no pressure pump & separator needed !!!

p < 300 bar

2 sapphire windows (2 x 12mm)

Sample volume ≈ 18 mL

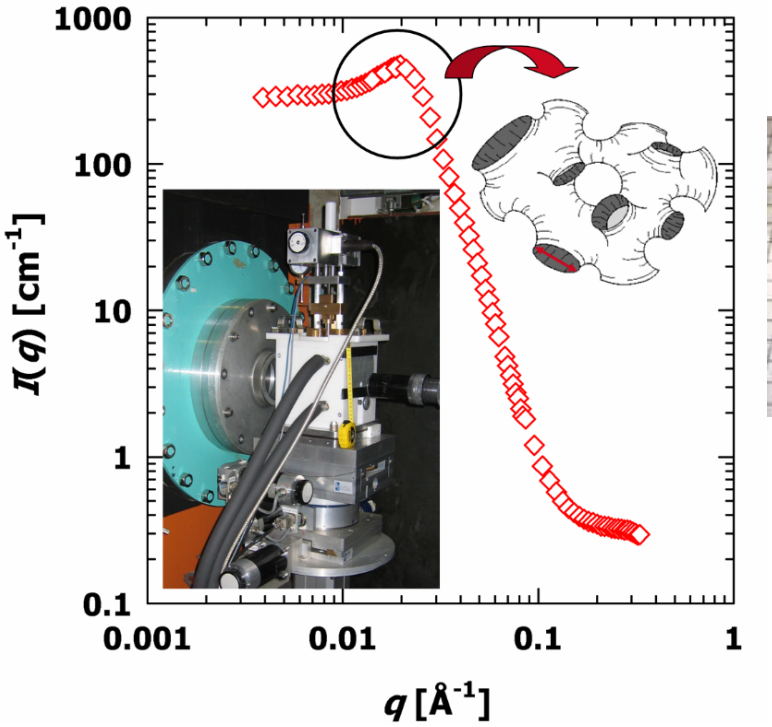
sample thickness 2mm

highly precise (± 0.05 °C) T-control

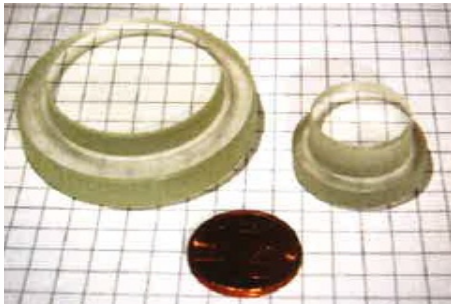
10°C < T < 80 °C

weight: 30 kg (!)

Cologne pressure cell



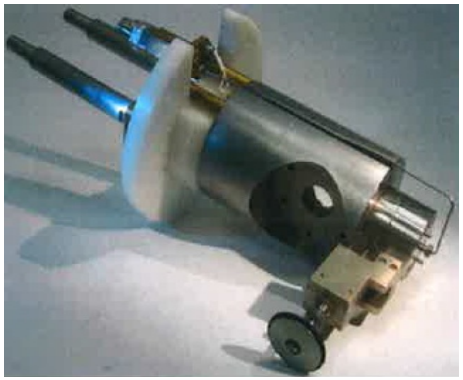
Filling done from the bottom by turning the cell upside down

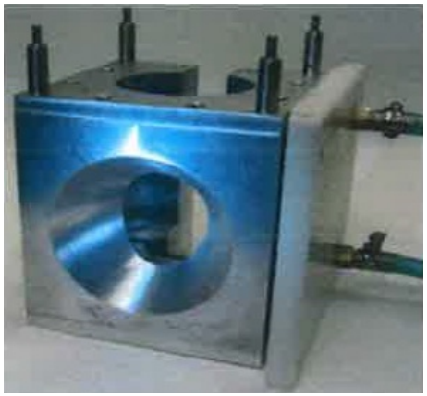


sapphire windows (2 x 12mm)

stainless steel inner core

external thermal insulation jacket



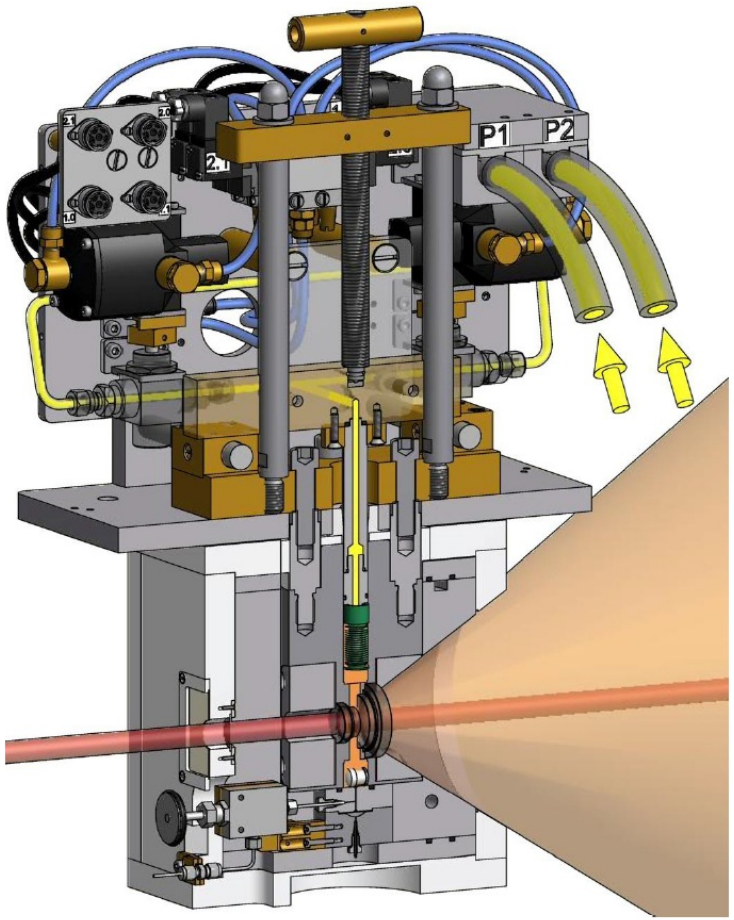


study of structure & phase transitions of supercritical CO_2-water microemulsions

Photos taken from PhD thesis of L. Kramer (U Cologne)

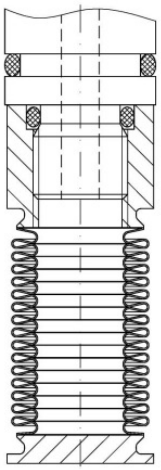
Cologne stroboscopic pressure cell

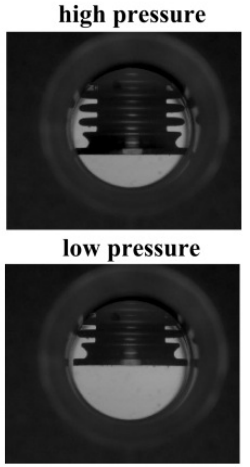
Developed for the TISANE option on D22



Characteristics:

metal bellow reaching into the sample volume
valve circuit to switch between pressures
sample volume ≈ 18 mL
10 °C < T < 50 °C 0.1 °C T precision
p_{max} 350 bar
highest possible p cycle frequency 10 Hz





taken from PhD thesis of Y. Pütz (U Cologne)

Cologne stroboscopic pressure cell

Developed for the TISANE option on D22

CrossMark click for updates

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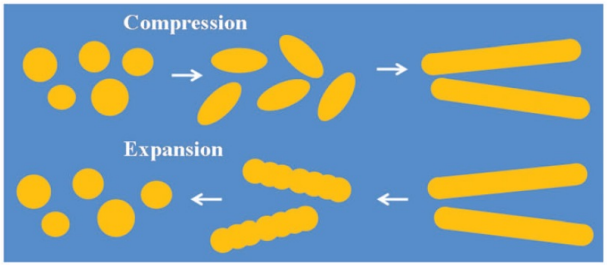
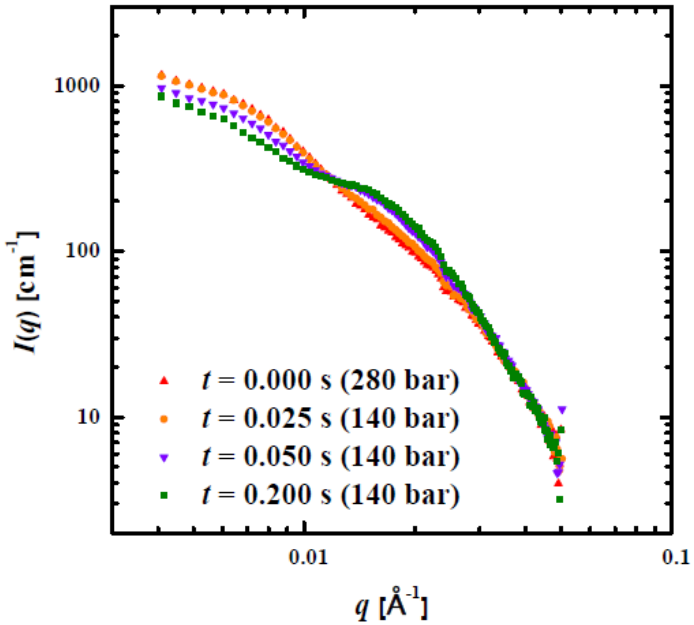
Kinetics of pressure induced structural changes in super- or near-critical CO_2-microemulsions†

Alexander Müller,[a] Yvonne Pütz,[a] Roland Oberhoffer,[a] Nils Becker,[a] Reinhard Strey,[a] Albrecht Wiedenmann[b] and Thomas Sottmann*[c]



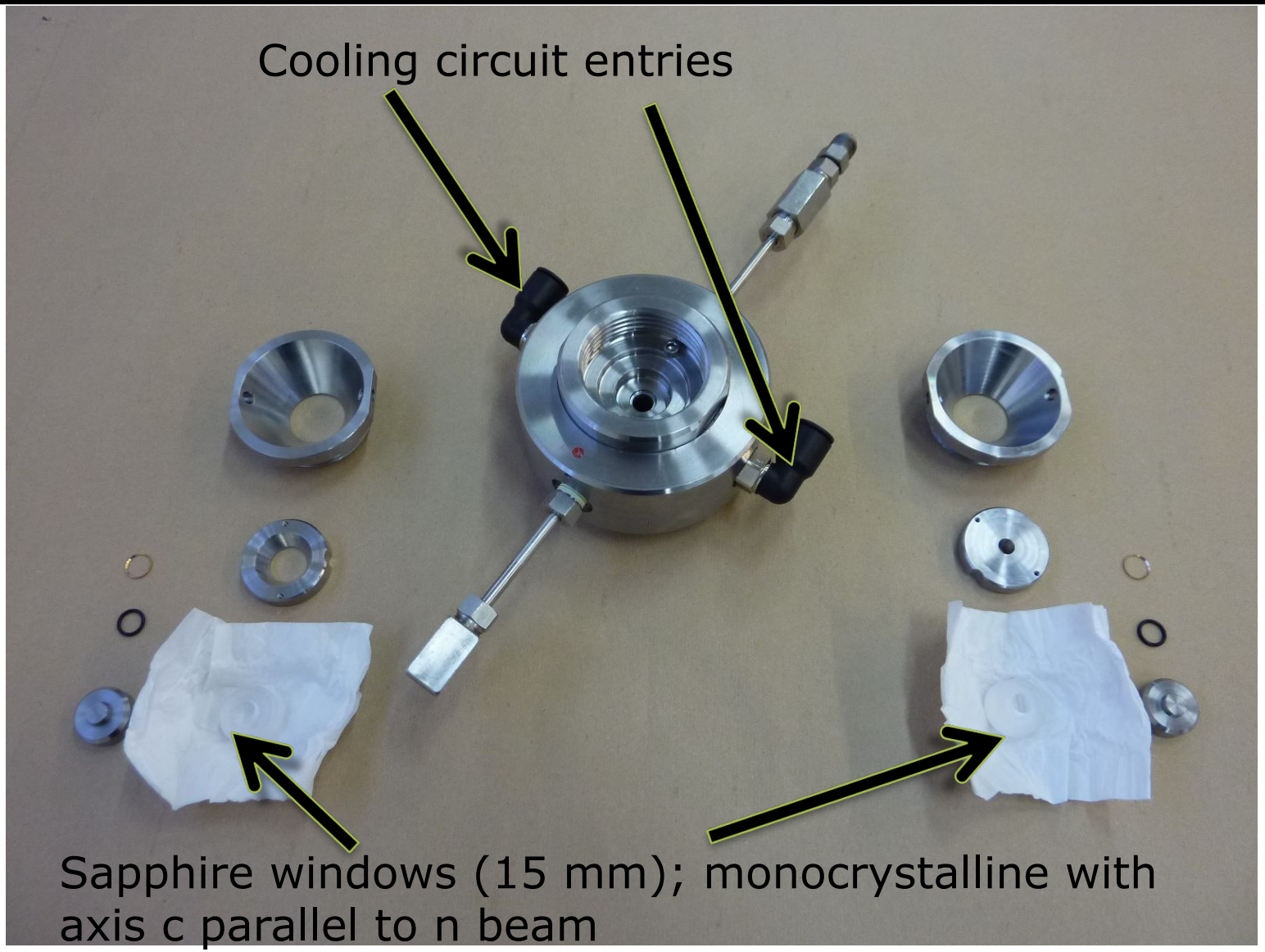
Scheme 2 Schematical illustration of the two different mechanisms of structural reorganization for compression and expansion.

CO_2-microemulsions show strong pressure dependent properties. Using time-resolved SANS to investigate the kinetics of structural changes upon periodic pressure jumps of adjustable amplitude, we found that the compression-induced formation of cylinders occurs on a timescale of one second, whereas the expansion-induced disintegration into CO_2 swollen spherical micelles is much faster.



taken from PhD thesis of Y. Pütz (U Cologne)

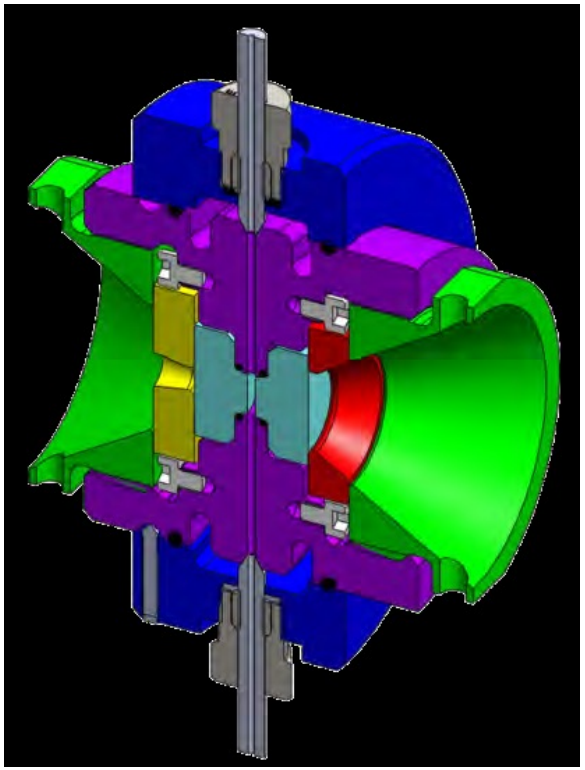
Nova Swiss 5 kbar hp cell



Nova Swiss 5 kbar hp cell

(Nominal) specifications:

p_{max}: 5 kbar continuous
T-control with ≈ 1.5 °C precision
T-range: 10 °C < T < 100 °C
sample thickness: 2mm
variable exit angle of 35°, 45°, 63°

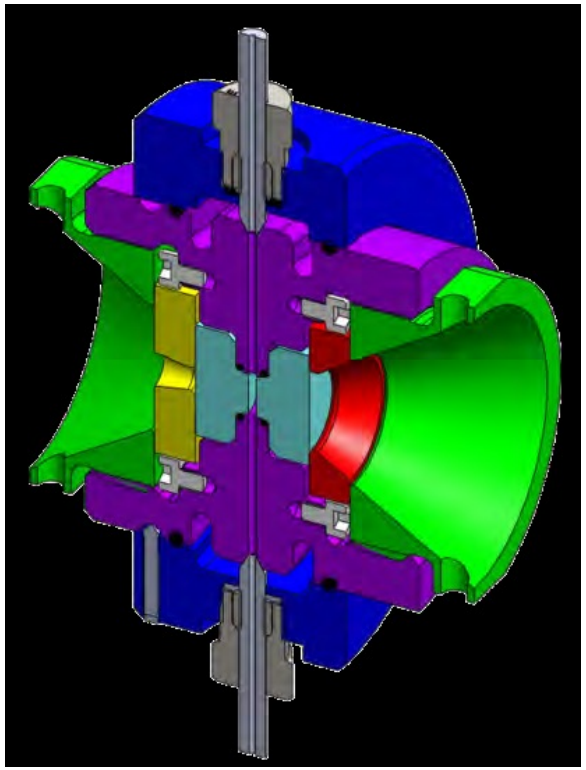


Nova Swiss 5 (?) kbar hp cell

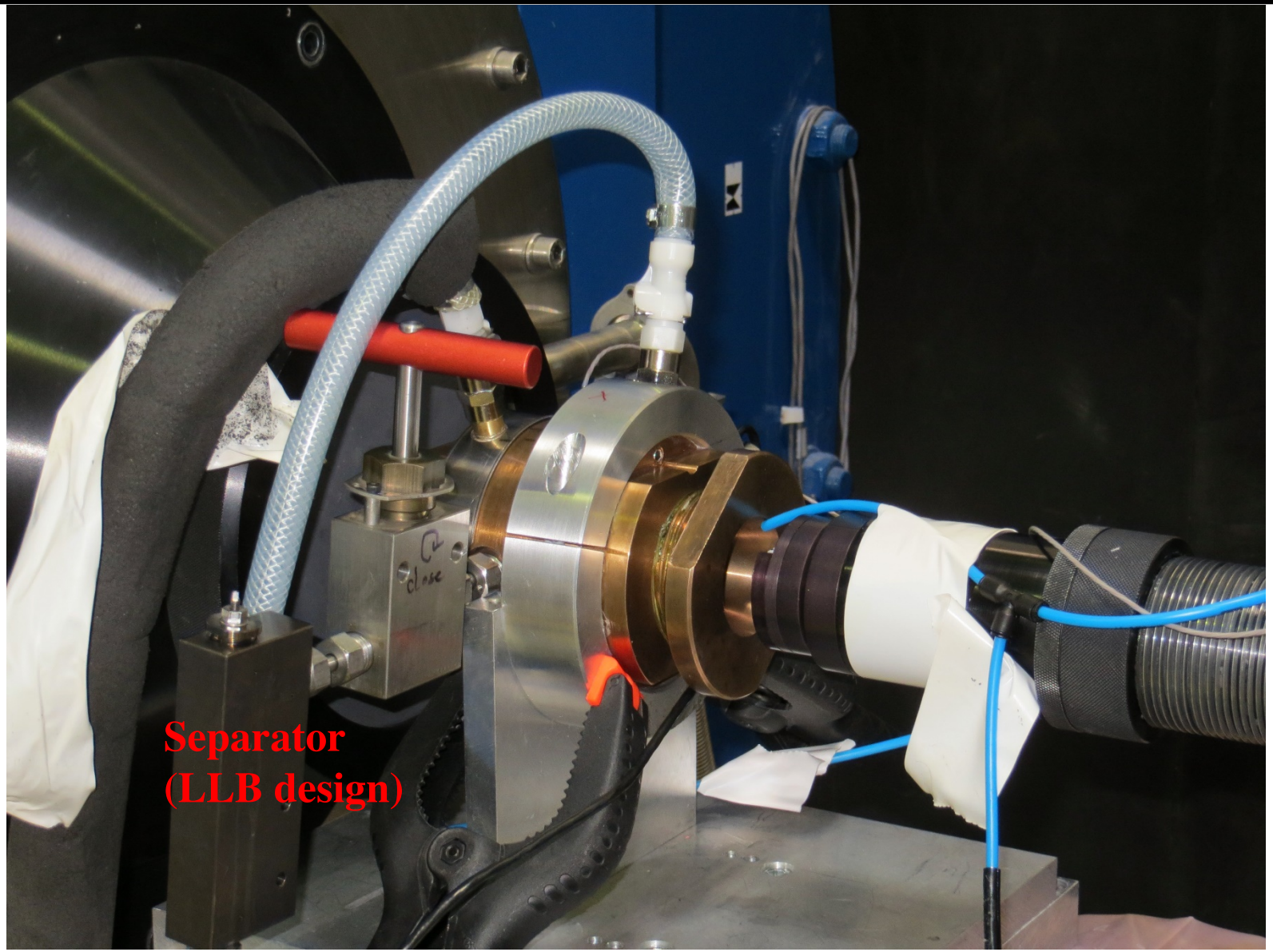
The bitter truth:

∅ 12 mm incident, exit cone 35°: incident window broken at 3.2 kbar
∅ 9.5 mm incident, exit cone 35°: incident window broken at 2.3 kbar
∅ 6 mm incident, exit cone 35°: exit window broken at 2.5 kbar

Variable exit angle of 35°, 45°, 65°



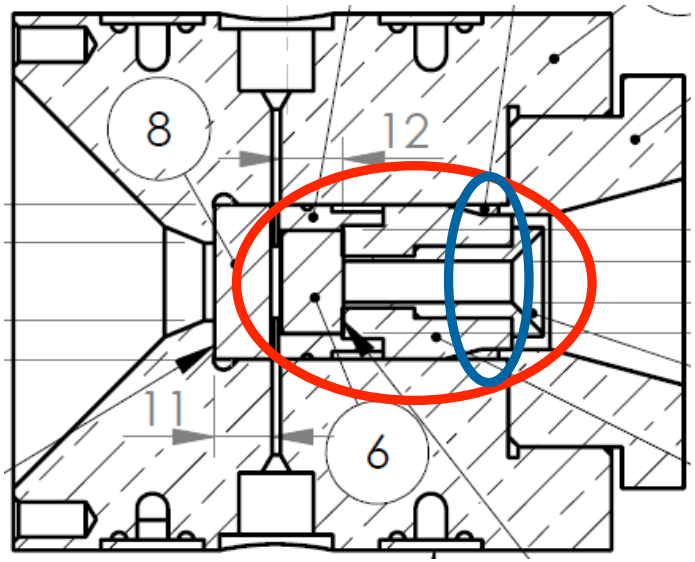
New generation 5 (!) kbar hp SANS cell



New generation 5 (!) kbar hp SANS cell

Cell body made out of CuBe (common cell for NSE & SANS):

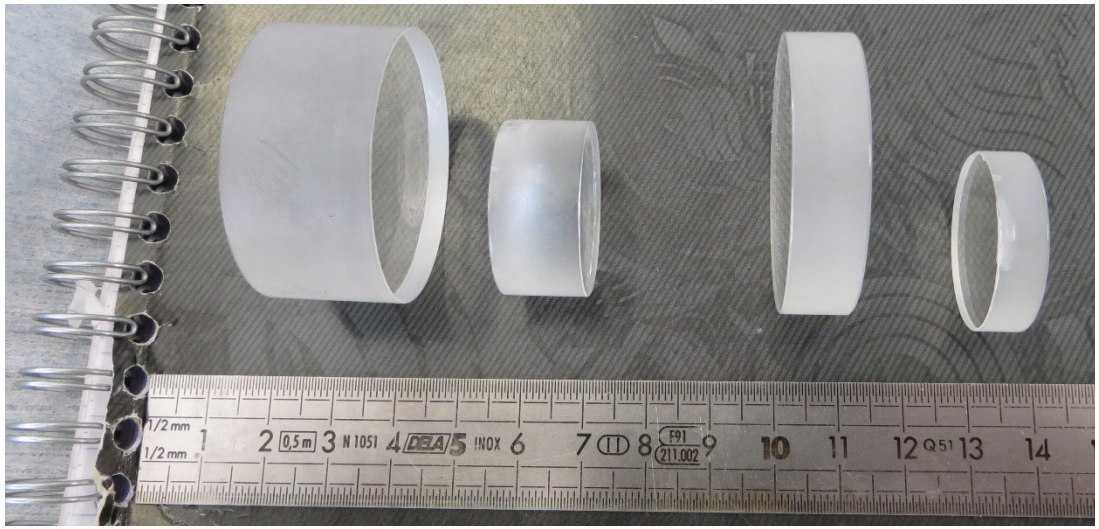
p_{max}: 5 kbar
T-control with ≈ 1.5 °C precision
T-range: 5 °C < T < 100 °C
(initial) sample thickness: 2mm
exit angle for 2 kbar: 50°
5 kbar: 35°



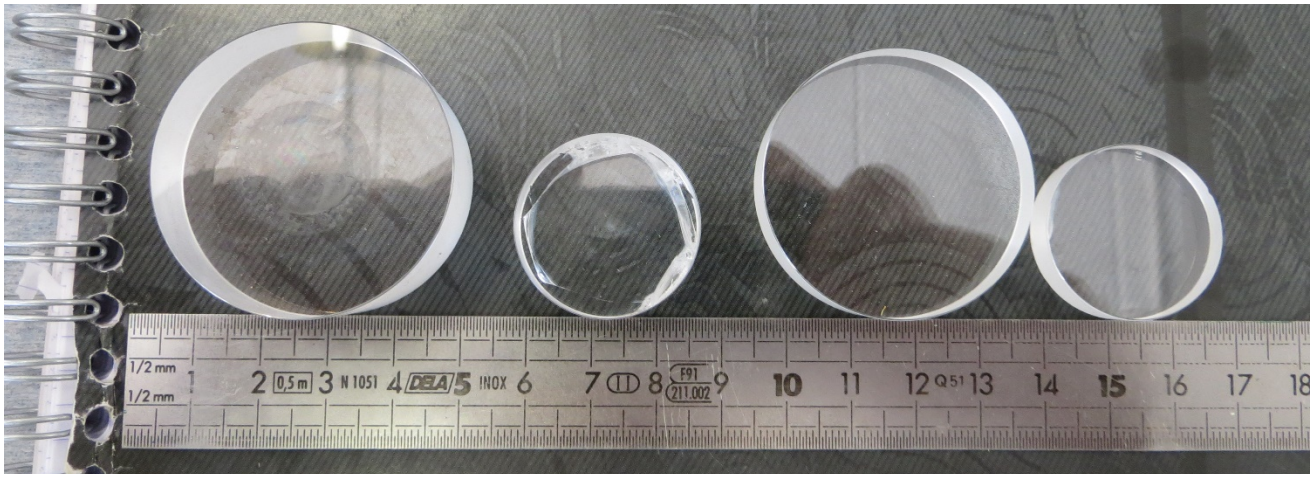
New generation 5 (!) kbar hp SANS cell

5 kbar **Sets of sapphire windows** **2 kbar**

5 kbar
entrance d 13 mm
exit d 20 mm
Tr@ 6Å 84 %



2 kbar
entrance d 7 mm
exit d 10 mm
Tr@ 6Å 89 %

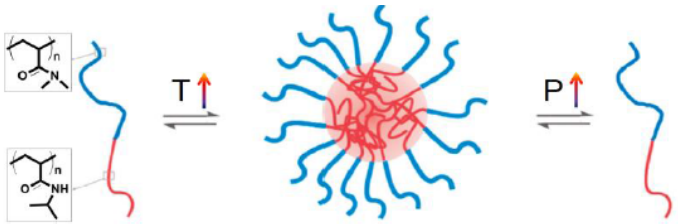


New generation 5 (!) kbar hp SANS cell

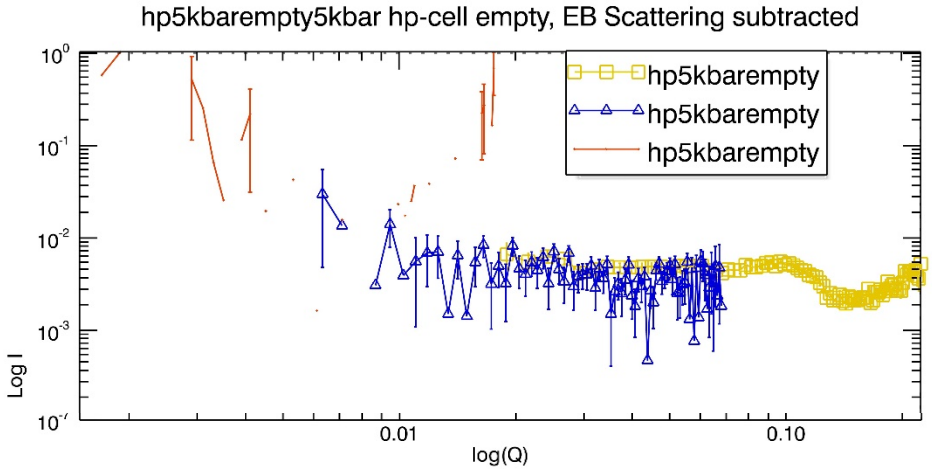
NEUTRONS FOR SCIENCE

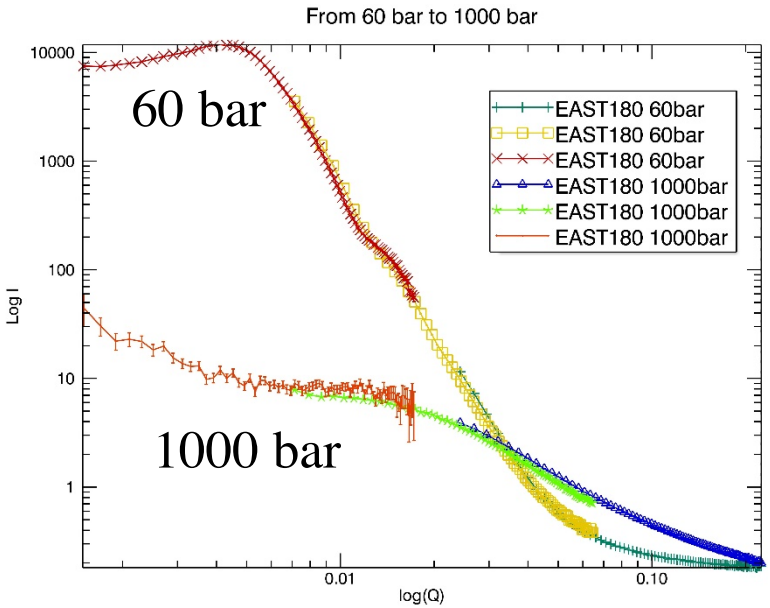
Pressure-induced reverse micellisation of PNIPAM based micelles

groups of Plamper and Richtering (RWTH Aachen)



Scheme 1: Diblock copolymers are present unimolecularly at ambient pressure and below LCST; at temperatures above the LCST micellization occurs and it is supposed that the micelle dissolves again upon pressure application (pressure acts antagonistically to temperature)





Thank you for your attention !