WP 20 « ADVANCED NEUTRON TOOLS FOR SOFT AND BIO MATERIALS »

4 tasks:

- Platform for model biological membranes	Task 1
- Kinetic & Dynamics experiments	Task 2
- Humidity chamber	Task 3
- Cryogen free cryostat with sample changer	Task 4

Last meeting:

May 28 - 29 2015 at Saclay (LLB)

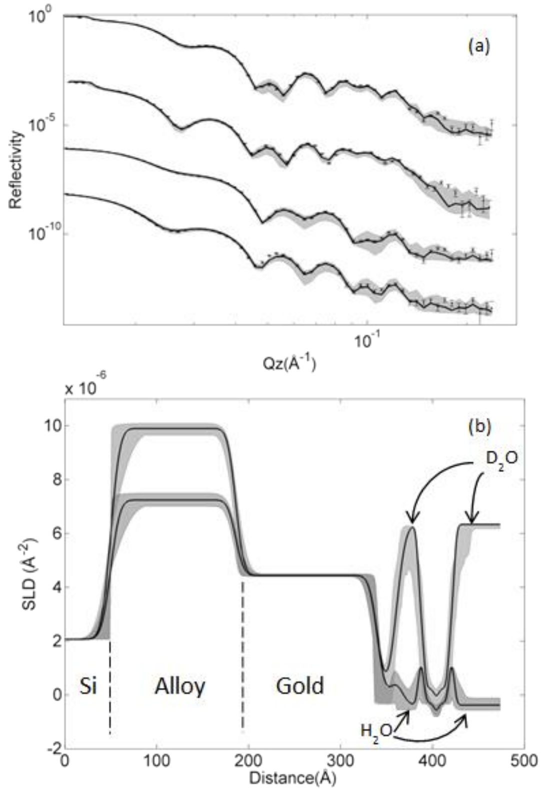
Task 1
A platform for model biological membranes

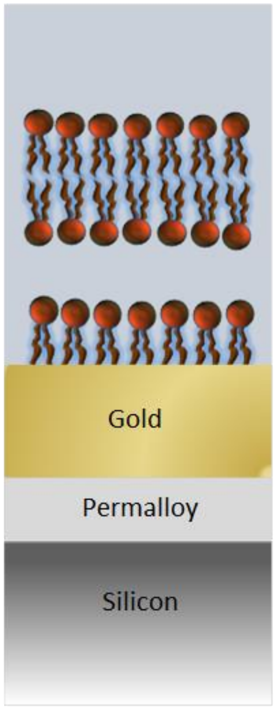
Optimization of model bilayer systems including natural membrane lipids studied by neutron reflectometry

ILL , STFC

New floating membranes : Bilayers supported on thiolipid on gold

ISIS





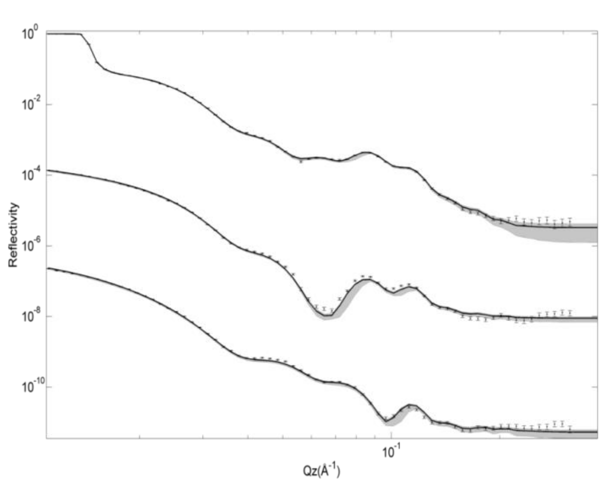
This system is giving 100% coverage bilayers.

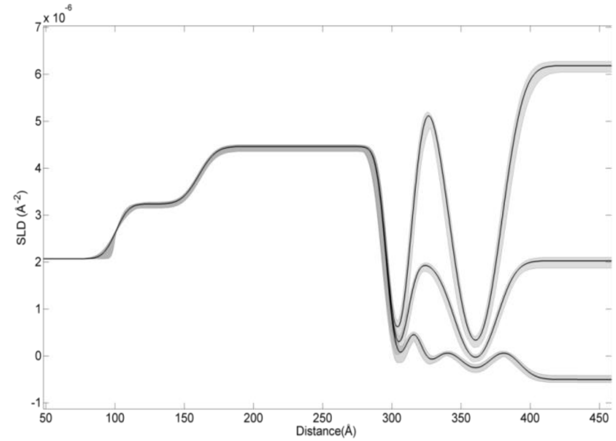
Now use of **magnetic underlayers** and **Polarised Neutrons** to give additional contrasts.

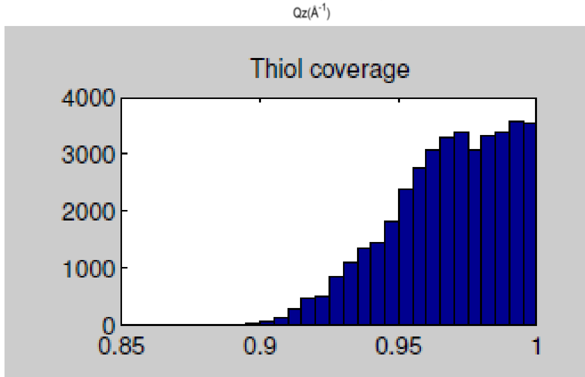
(ANSTO, NIST)

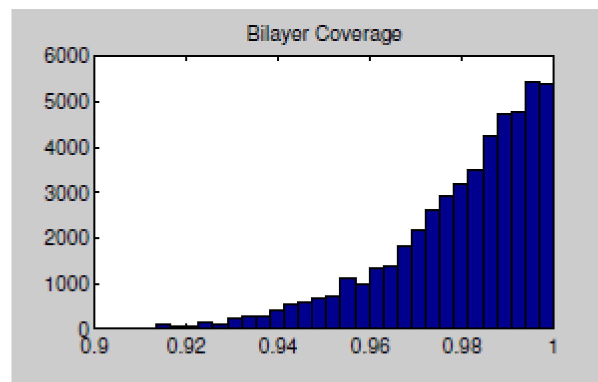
Task 1
A platform for model biological membranes

Data Analysis Development of Bayesian analysis codes for model fitting…





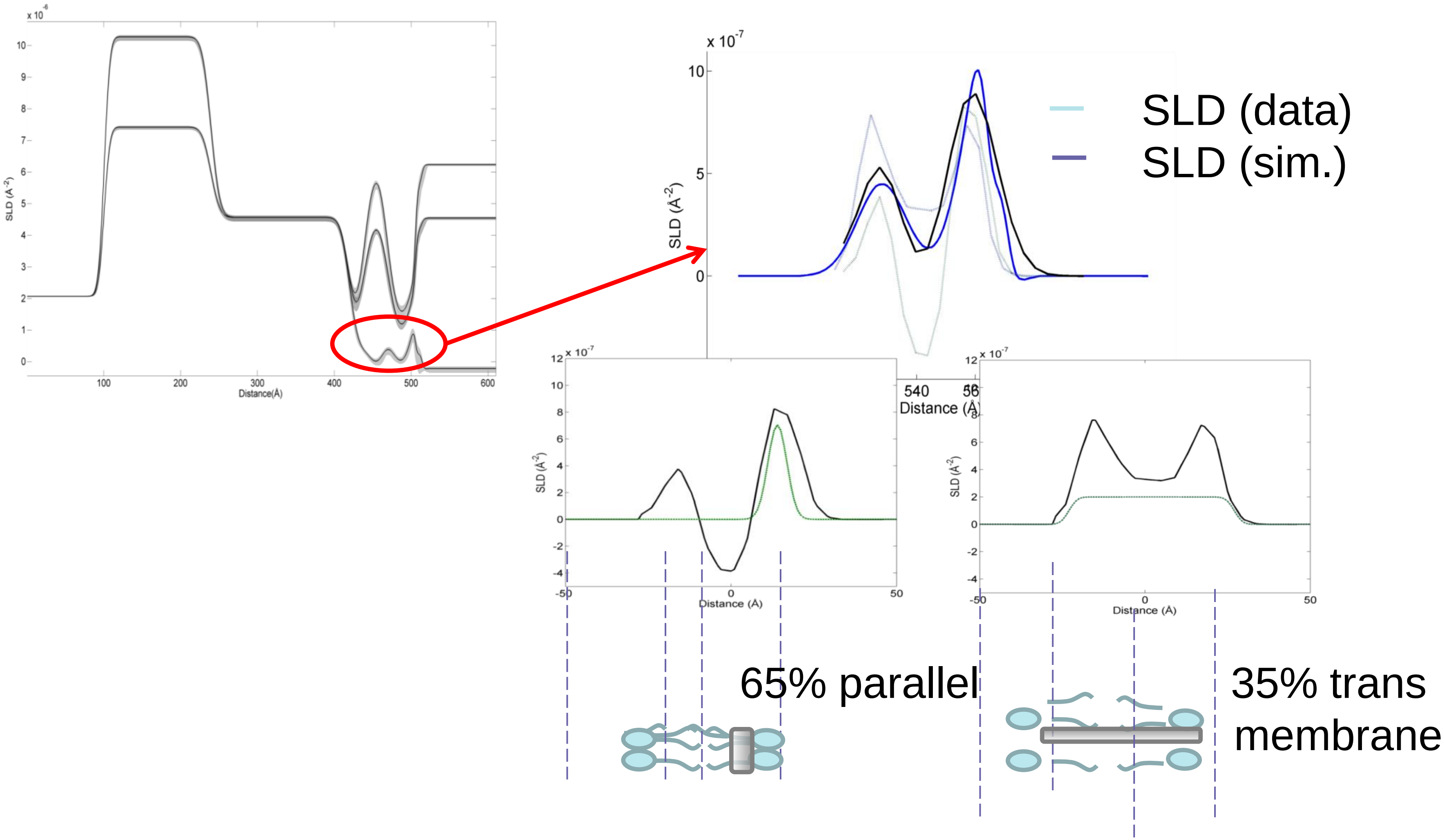




This gives robust methods for parameter (and uncertainty) estimation for 'traditional' scattering models. This is in a beta version soon ready for release…

Task 1
A platform for model biological membranes

Data Analysis … combined with molecular dynamics



Task 1
A platform for model biological membranes

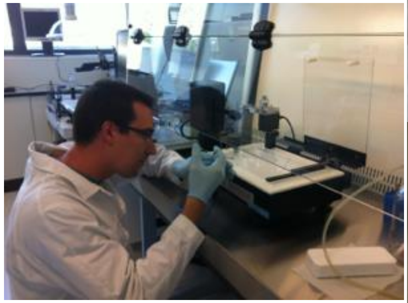
D lipid
(ILL)

- Production from yeast
- Extraction, separation of D lipids
- Membranes reconstruction from these D lipids. Characterization by NR and diffraction

-Study the insertion of biomelecules (sterols, amphotericin) into membranes using D or H lipids.

Several publications

Laboratories and lot of equipments (FTIR, DLS, ellipsometry, ttrough…) at the disposal of users at ILL

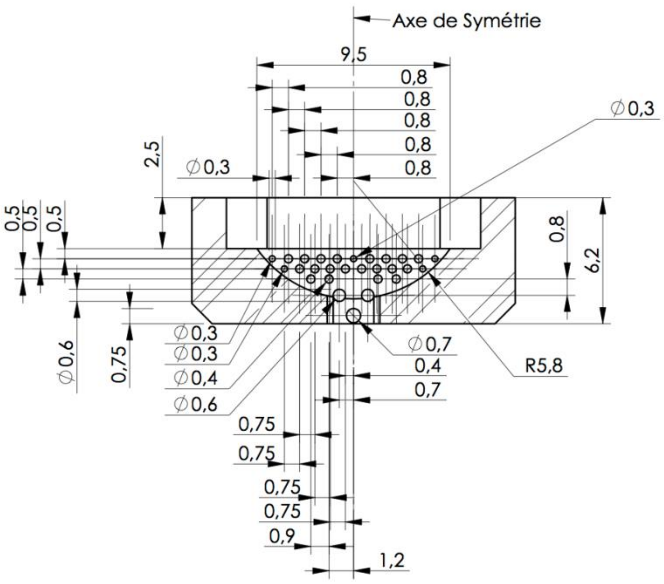


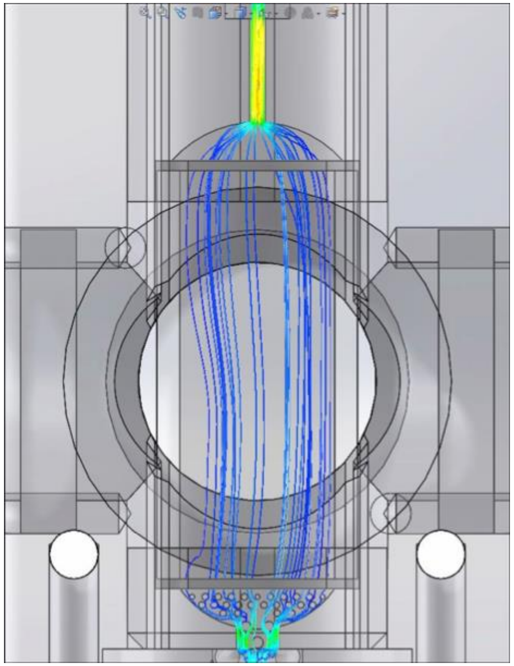
Task 2
Kinetic/dynamic measurements

New observation heads for Stop Flow
ILL

- Reduce wasted sample **with improved mixing process**
- **Improve temperature stability, reuse existing syringes (very costly)**

Design and simulation



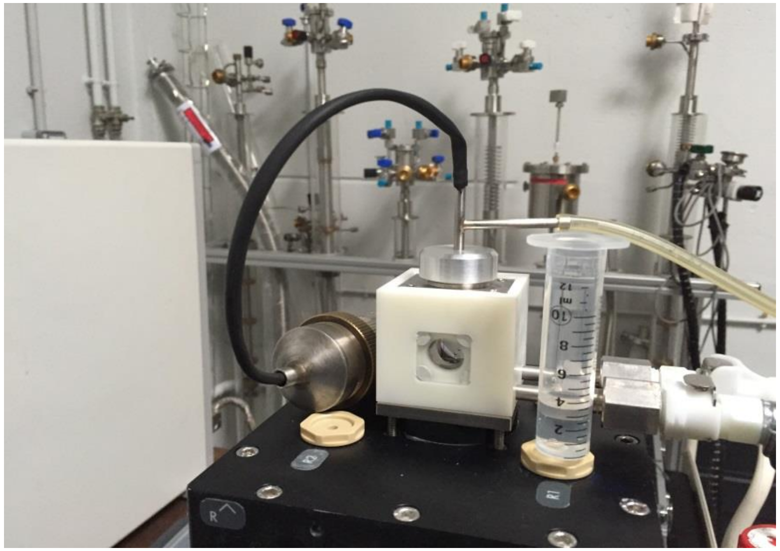


Damping grid designed at ILL, built at ISIS, and successfully tested at ILL

Task 2
Kinetic/dynamic measurements

A new temperature-controlled chamber

- Improve T stability **with fluid circulating inside the head** (0.1 K)



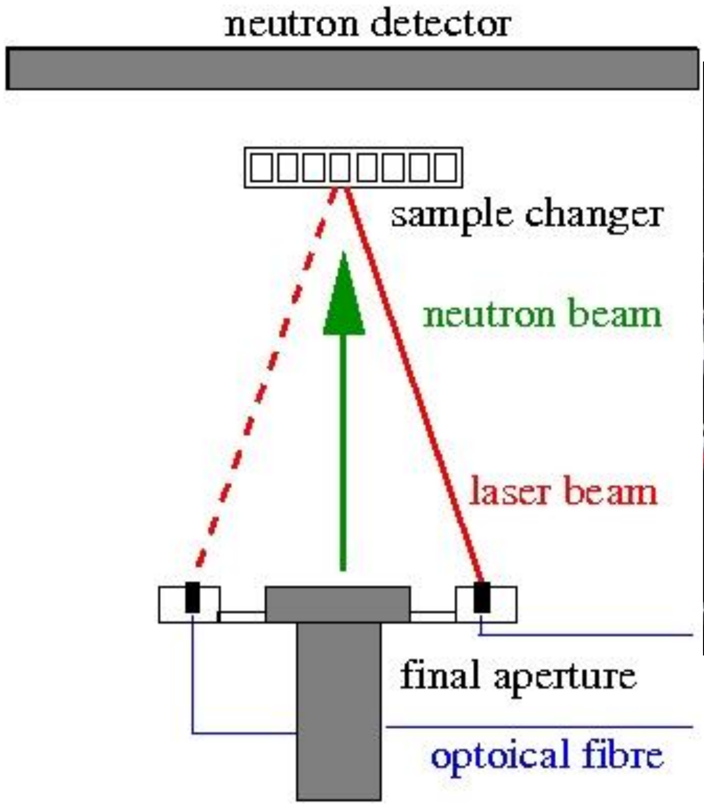
- Insulation
-> Much better T° control than commercia
device Biologic
- 40 % less sample volume
- Warming up at 1.7° C/min with 2000 W
- Cooling down at 0.7 ° C/min with 320 W

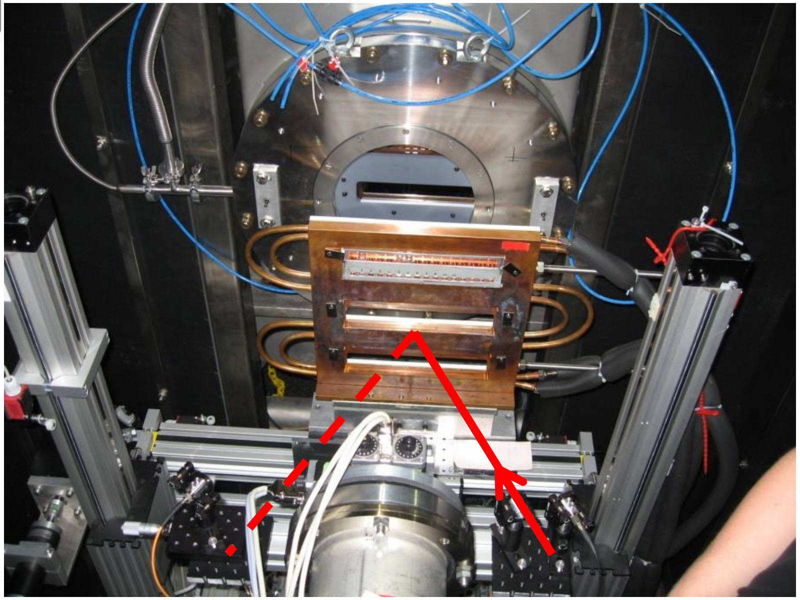
Perspectives
Simultaneous push/pull technics to evacuate the sample

A combined static LS DLS and SANS
JCNS,CEA,ILL

LS in fiber configuration
- Location on the SANS collimator exit   (JCNS)

Advantage: possible to use sample changer

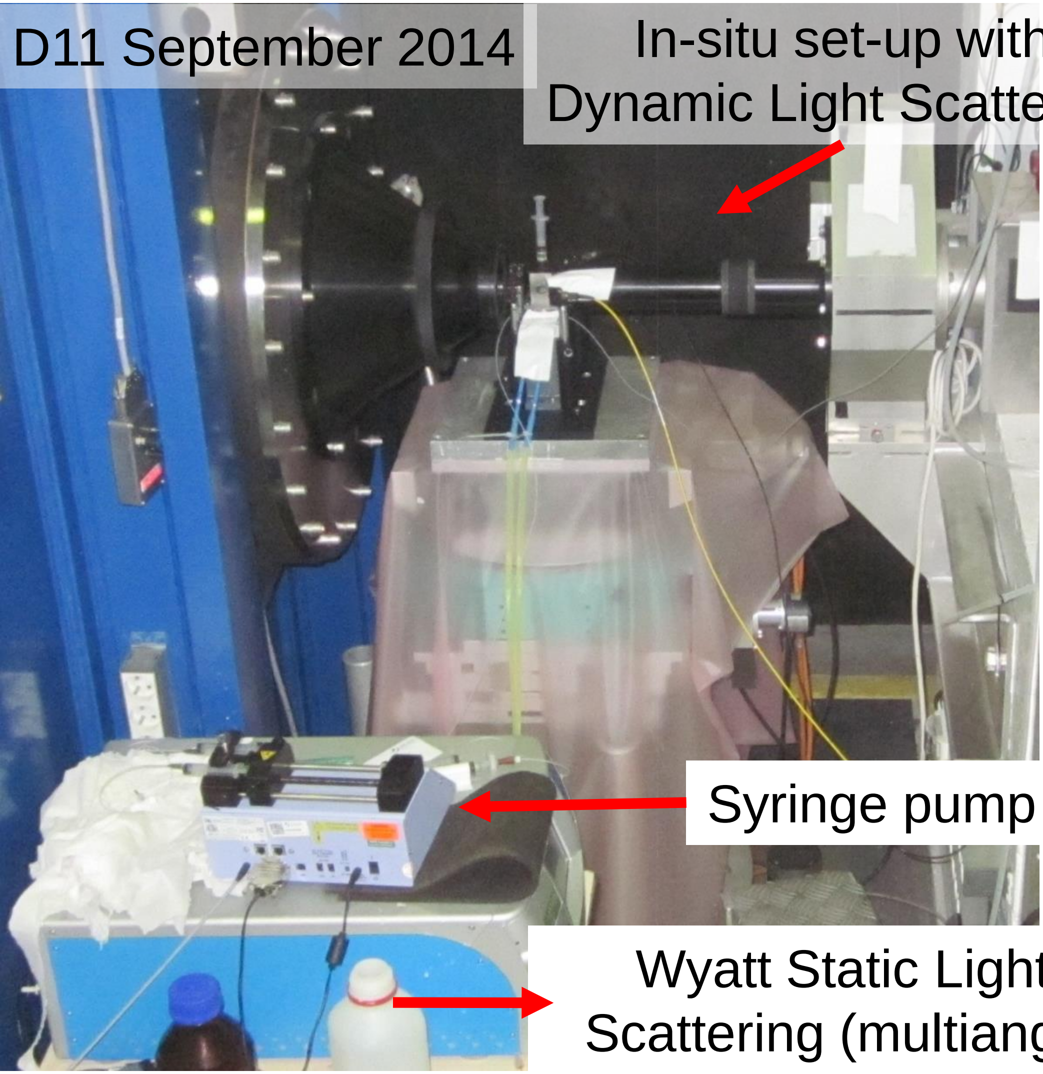




Task 2
Kinetic/dynamic measurements

D11 September 2014

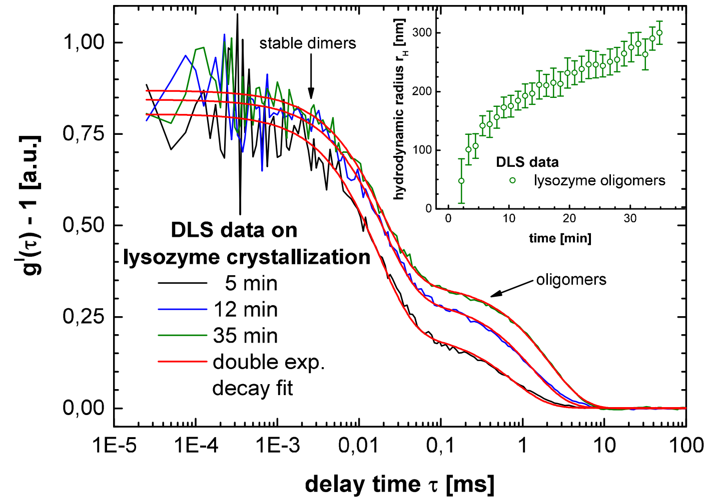
In-situ set-up with Dynamic Light Scattering



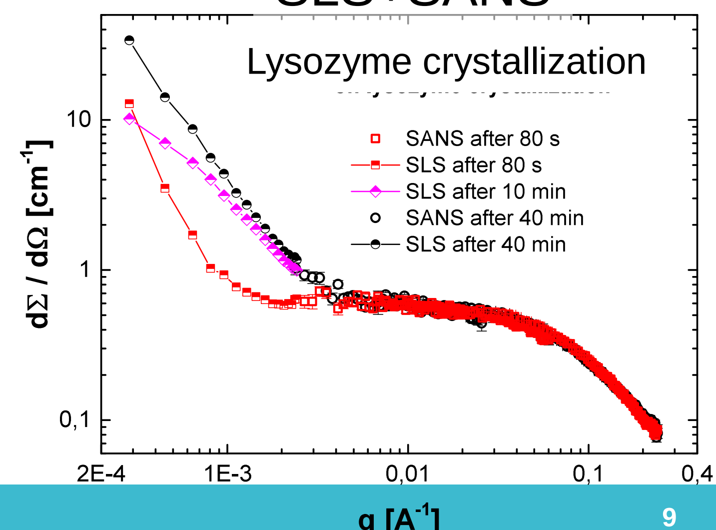
Syringe pump

Wyatt Static Light Scattering (multiangle 18) instrument

Dynamic Light Scattering



SLS+SANS



Electric field cell with electrodes outside the sample

LLB

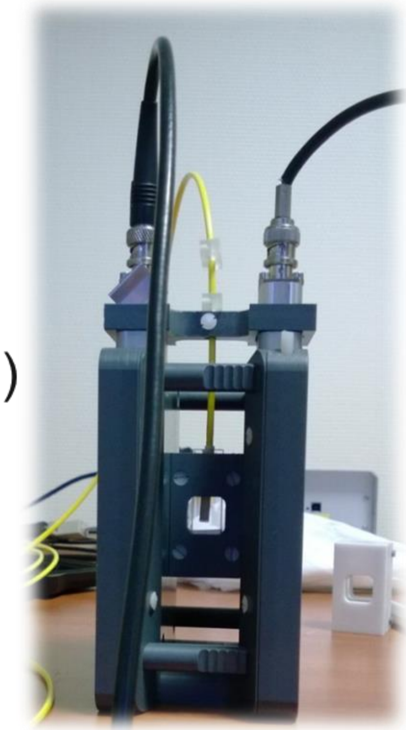
Electric field: from 0.04 to 4 kV/cm

Temperature: from 20 to 60 °C

Prototype #1 at room T°

Measurement of effective EF ✓ (probe at the sample location)

Fluid	Permittivity ε_r	Electric field (kV/cm) at 2kV 10Hz 20C 2.5cm
Air	1.0	3.07E-1
Toluene	2.3	2.45E-1
Ethanol	24.3	2.36E-2
DMSO	46.7	5.32E-3
Distilled water	78.6	2.72E-3

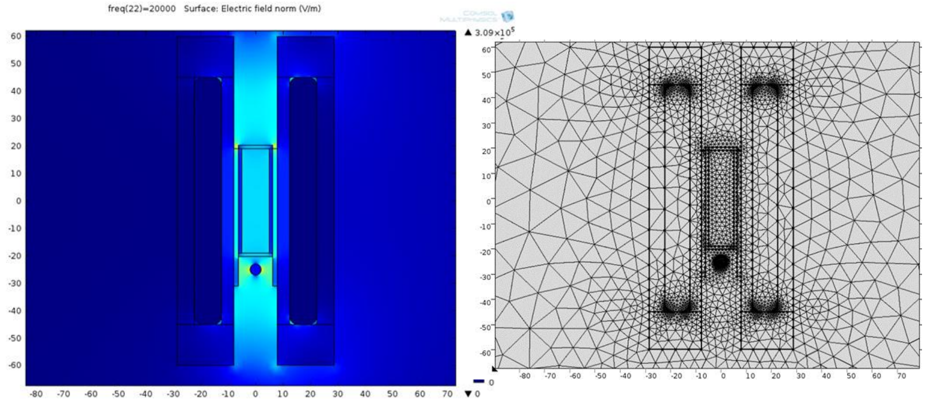


Probe Kaptéos Cie

Low values of EF due to surrounding materials

Task 2
Kinetic/dynamic measurements

Comparison Tests / Simulations ComSol Multiphysics
(LLB / HZB)



-> **Thermalization simulation**

-> **EF calculation**

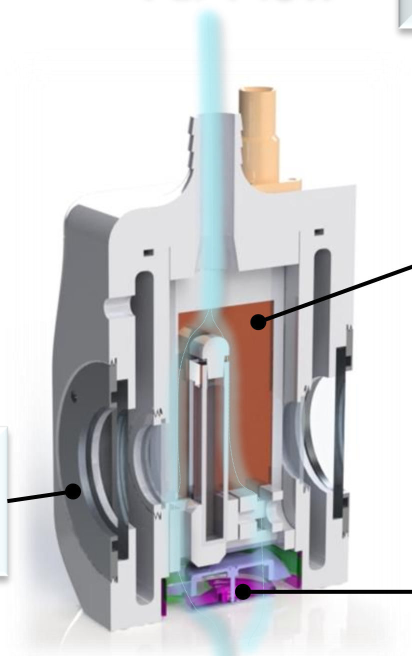
Get rid of materials around the cell

Task 2
Kinetic/dynamic measurements

Prototype #2: Closed and thermalized

High voltage connectors

Air Flow

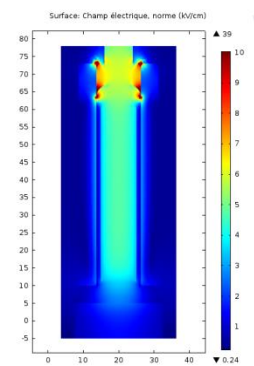


Electrode

Double-walled quartz windows

- **Less dielectric materials** between electrodes
- Sample thermalization from 10 to 60° C

EF ComSol ®



5 kV/cm in toluene (ε_r = 2.3) with 8kV applied

- Thermalization possible✓
- Remains measurements of the effective EF

Pressure cell for Neutron Spin Echo and SANS

JCNS, ILL, LLB

For NSE: Sample area: 3x3 cm^2
Pressure as high as possible … 3kbar?
Non magnetic materials

For SANS: Sample area: 1x1 cm^2
Pressure: 10kbar ?

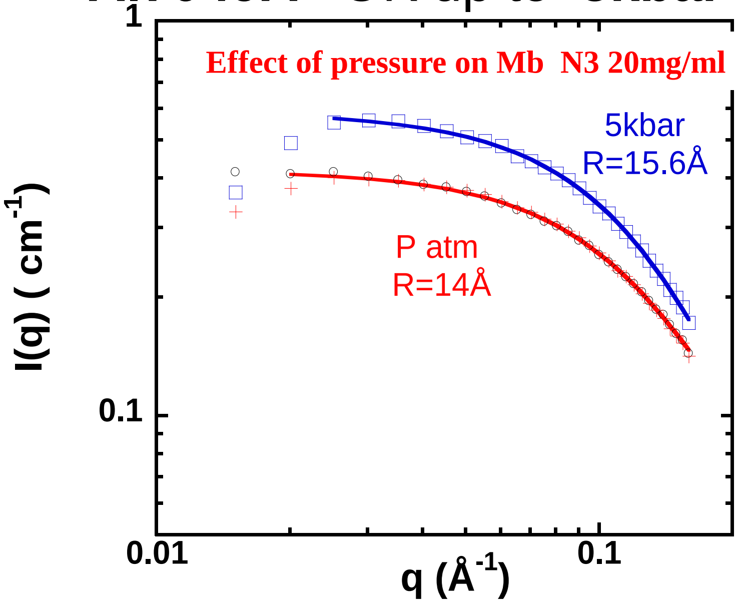
Metallic alloy windows
or sapphire windows

Task 2
Kinetic/dynamic measurements

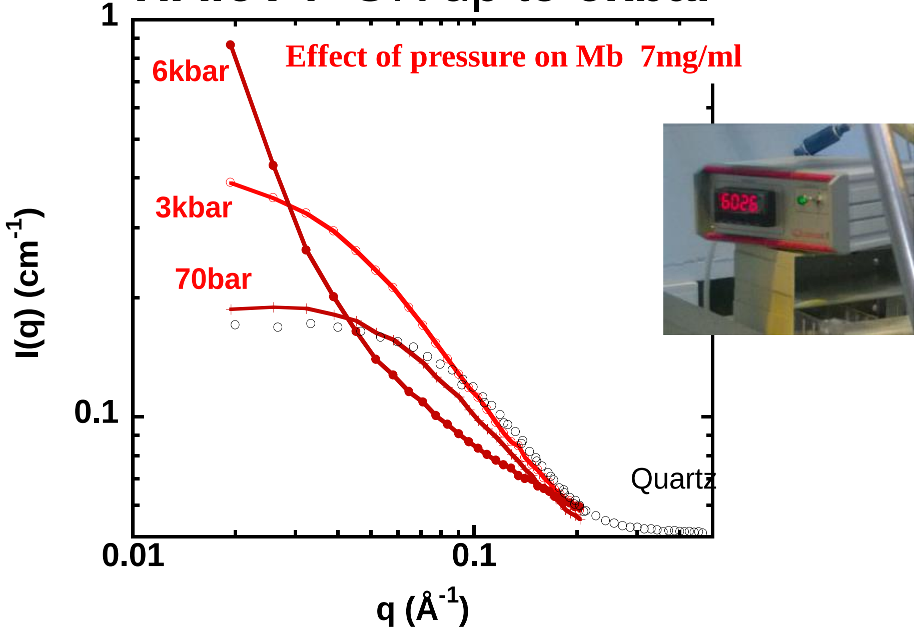
Pressure device (SANS) with metallic alloys windows
LLB

Nb Ok up to **3kbar** but windows have to be **plastified at P_{max} before P experiments**

Al7049A OK up to **5kbar**

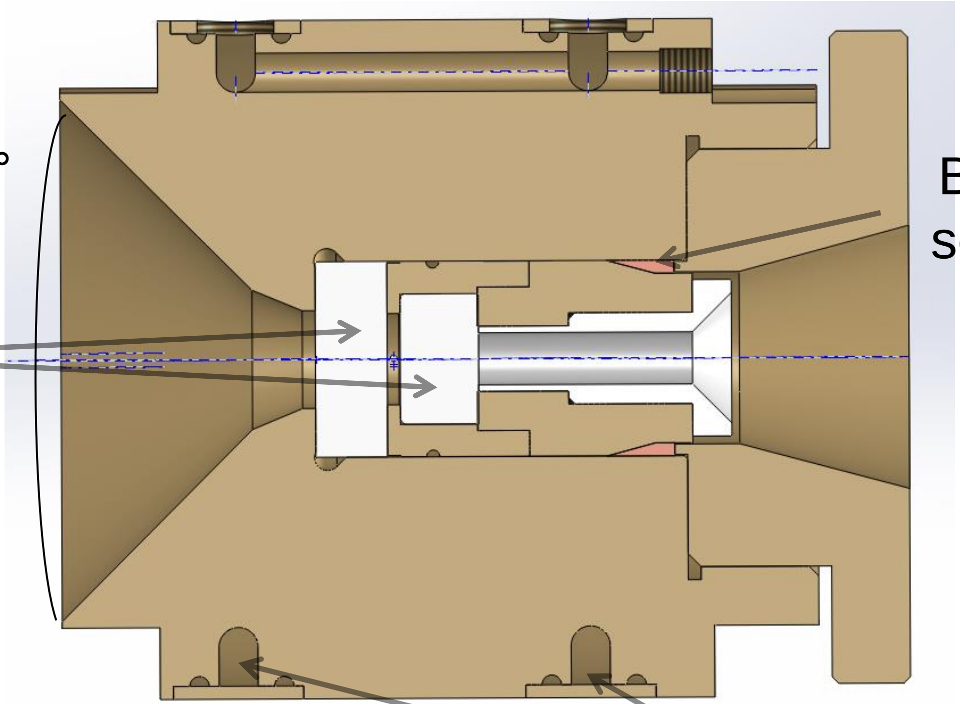


TiAl6V4 OK up to **6kbar**

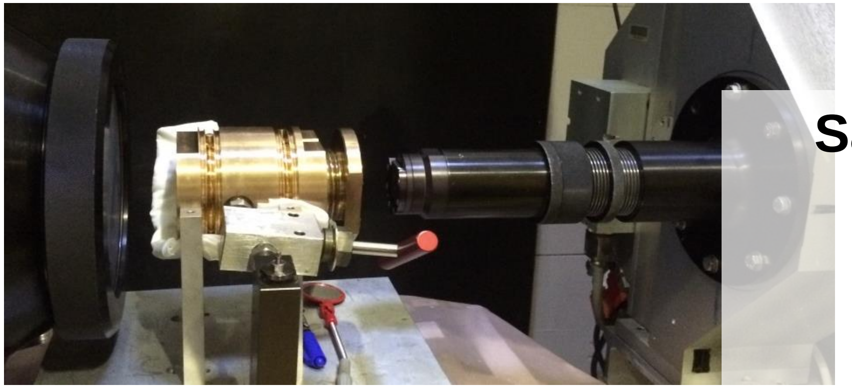


Works rather well **with very low scatterers (**dilute solutions of biological molecules.

A new P cell for SANS up to 5kbar with sapphire windows
ILL, LLB



First experiments on D11 June 2015



Sapphire OK up to 3.5kbar
Solution of apomyoglobine
1.9mg/ml

D11 July 2015
Temperature OK 10-60° C

Tests: Breakage at 4.5Kbar.
Remains to make compromise between opening angle, max pressure and windows thickness.

Task 2
Kinetic/dynamic measurements

First experiments carried out successfully!!!

Pressure up to 3.5 kbar reliable (5 kbar feasible)

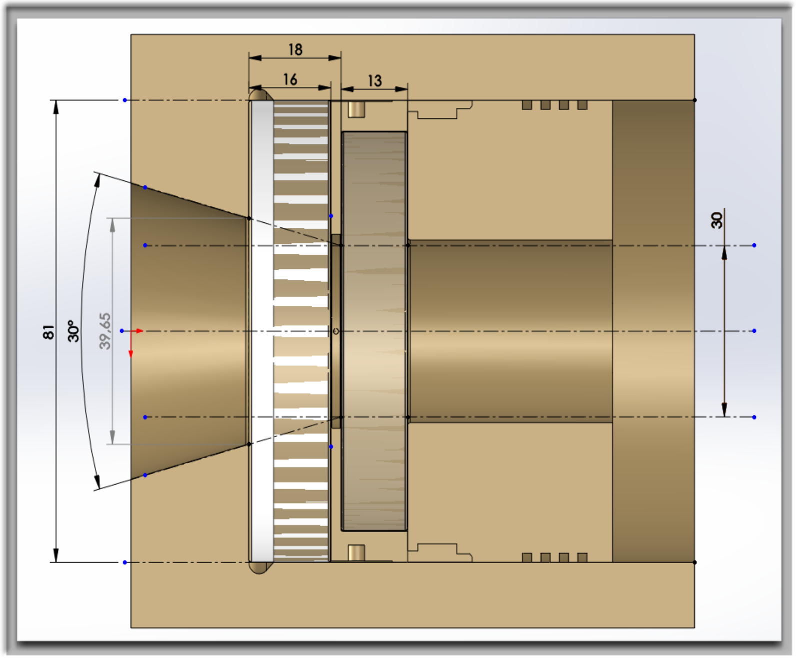
Temperature controlled & stable

Very high transmission (+84 % @ 6 Å)

Incident window displacement => to be fixed

Design a prototype

500 bar with Ø 30 mm bore for NSE/SANS



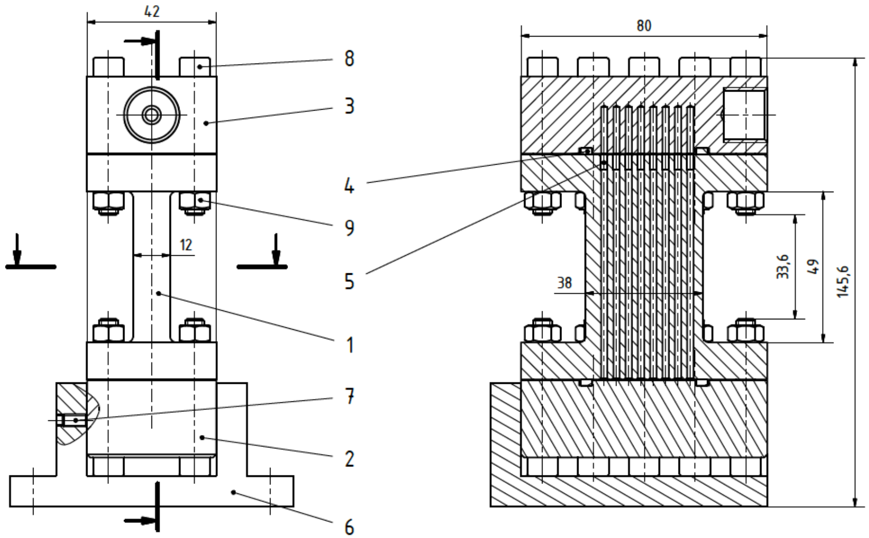
Task 2
Kinetic/dynamic measurements

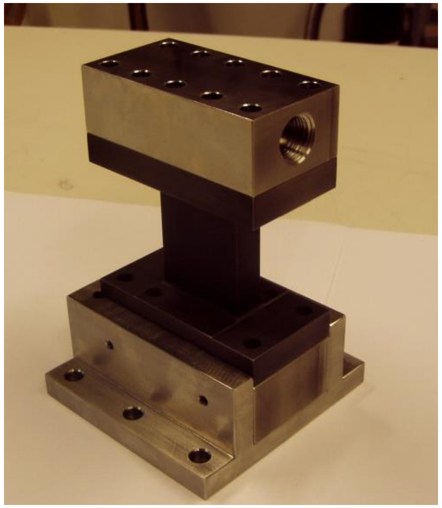
Pressure cell for NSE
JCNS

Prototype Cell # 2

- Several Cylindrical holes Ø=2mm
  Maximize sample area
- TiZr

(copy of ISIS pressure cell)





Seal: Perbunan
$P_{max.}$ 2.5 kbar

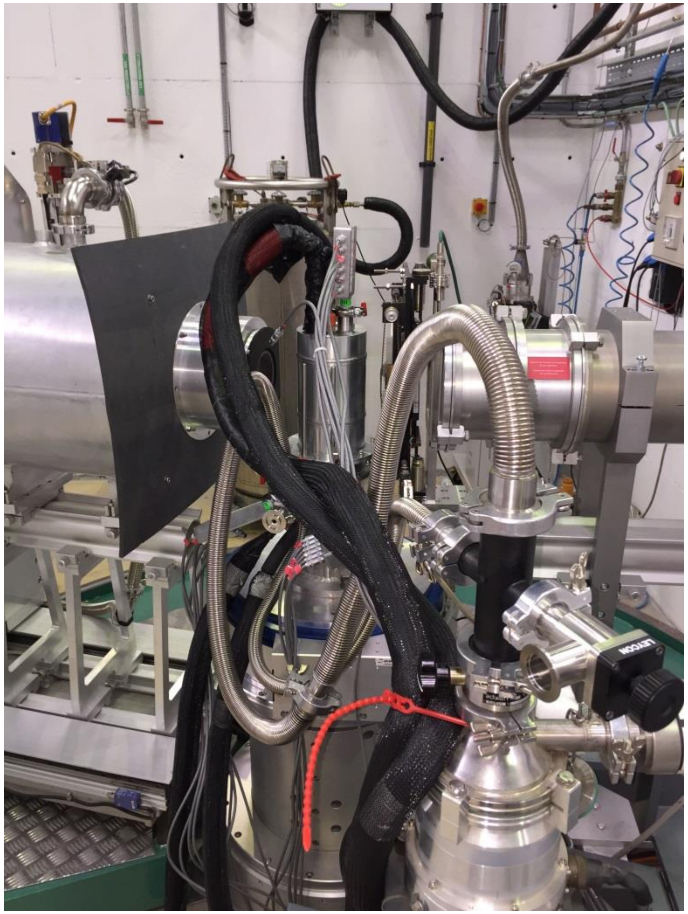
Seal: Copper
P_{max} 7.0 kbar
(operation: 5.6 kb)

Remains to be tested on NSE.

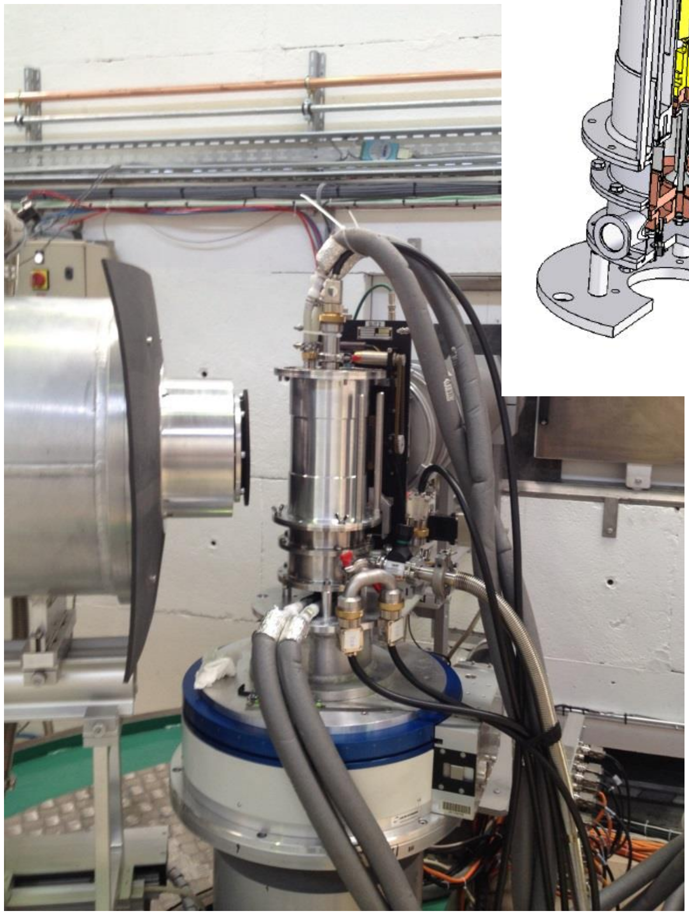
Task 3
Humidity chamber

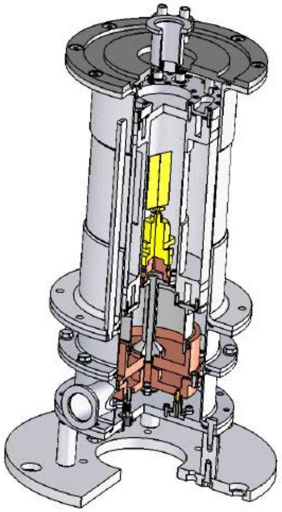
HZB, ILL

BerILL 1.0



BerILL 2.0



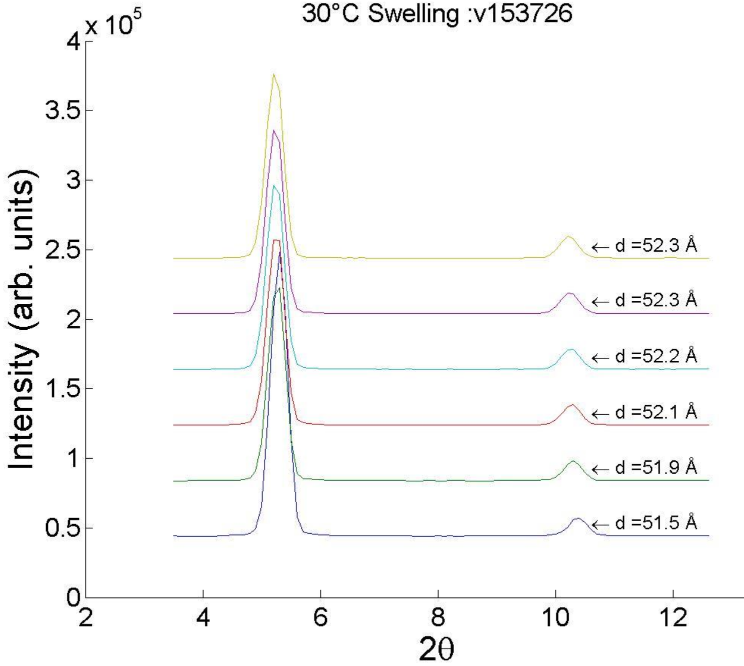


Task 3
Humidity chamber

D16 December 2014
BerILL 1.0

V1 April 2015
BerILL 2.0

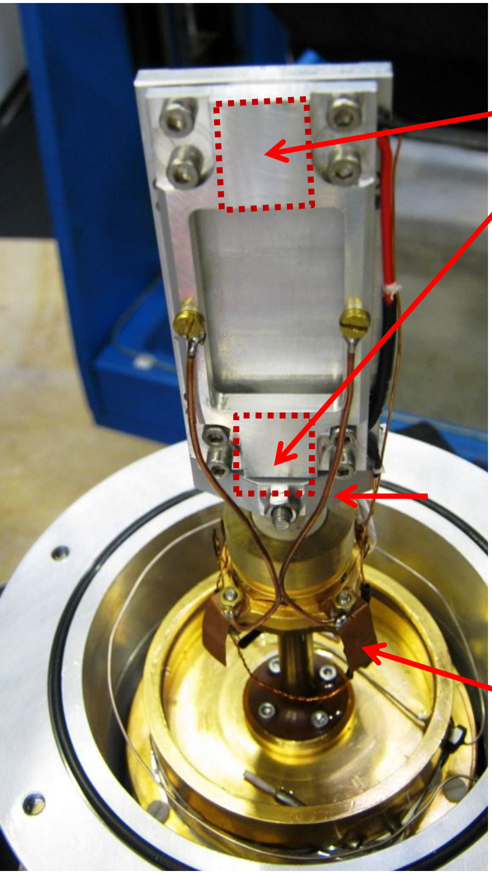
- DMPC fluid phase (30.5° C)



- Swelling 52.3 Å~ **98.5% r.h.**
- Full saturation not possible

D16 May 2015… **99.5% r.h**. check with Dirk

Humidity chamber



2 x Peltier elements *QC-17-1.0-2.5MS* Quick-Cool-Shop

2 x CU wires for heat transport to Gonio head

4 x CU plates for shorting Gonio and T1/T2

Final modifications/ Adjustments

(-Peltier elements top bottom of the sample
- RH sensor reading 1/5mn
- computer control of the Chiller T° setpoin
to speed up the thermalization

- Full 100% hydration achieved (not over entire sample)
- User friendly operation up to 99% r.H. possible

- **In HZB- user service since October 2015**

Task 3
Humidity chamber

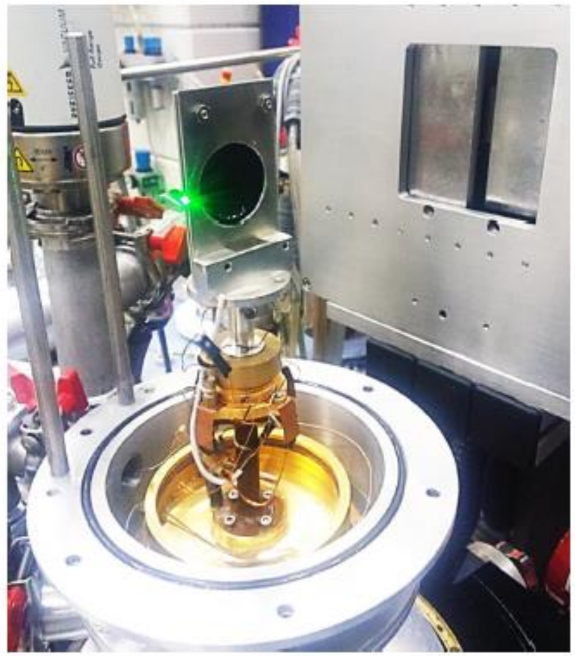
Use of *In-Situ* Small Angle Scattering Techniques to Probe the Dynamic Structure of Graphene-Based Membranes

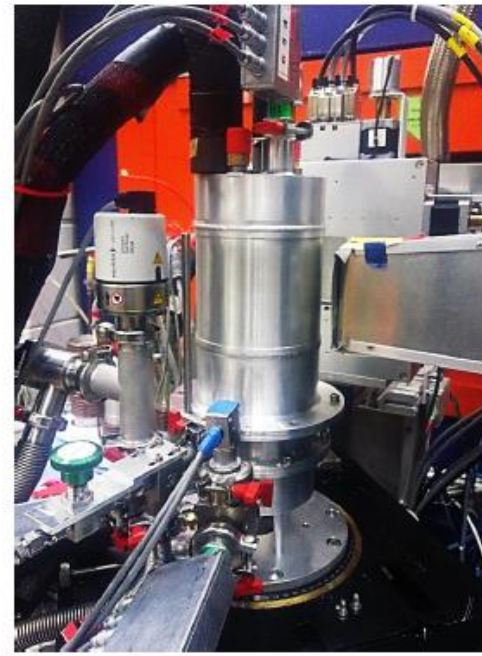
Ashley Roberts

Chris Garvey, Dan Li, George Simon

Neutron Diffraction V1:

Graphene membranes in alumina frame and placed inside humidity chamber





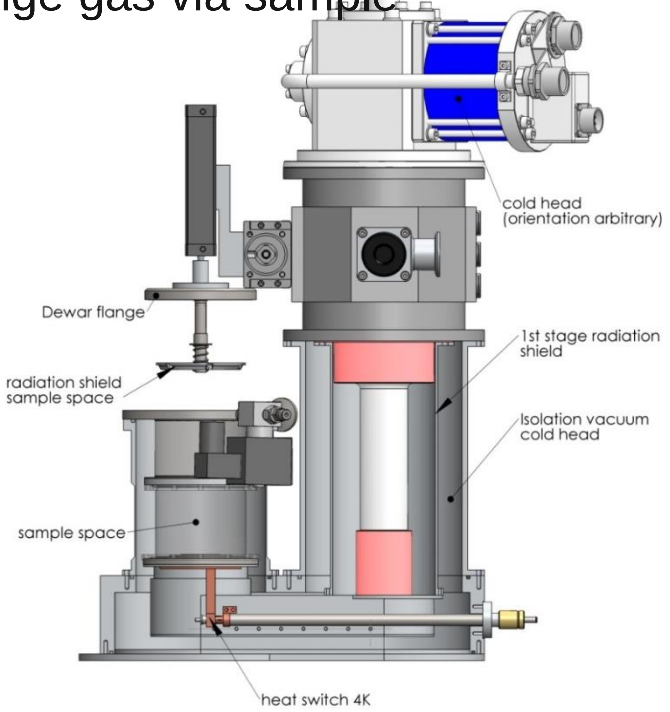
MONASH University

Australian Government | ansto

- **Compact cryostat**

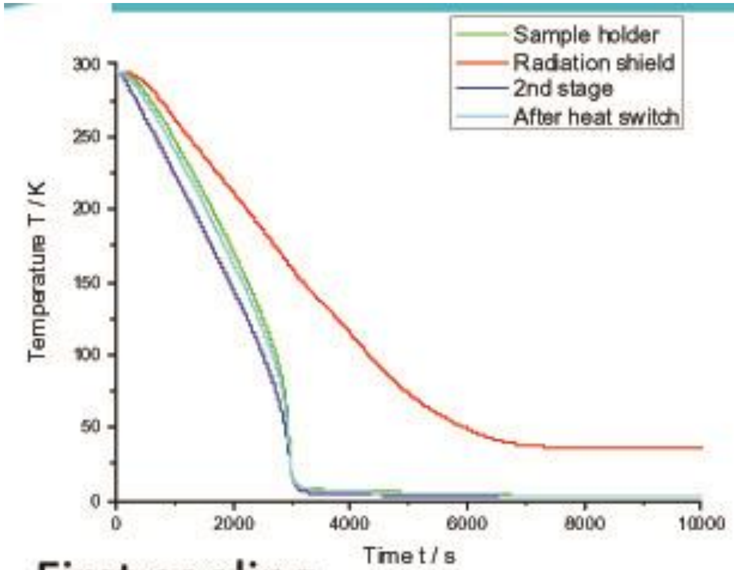
  FRMII

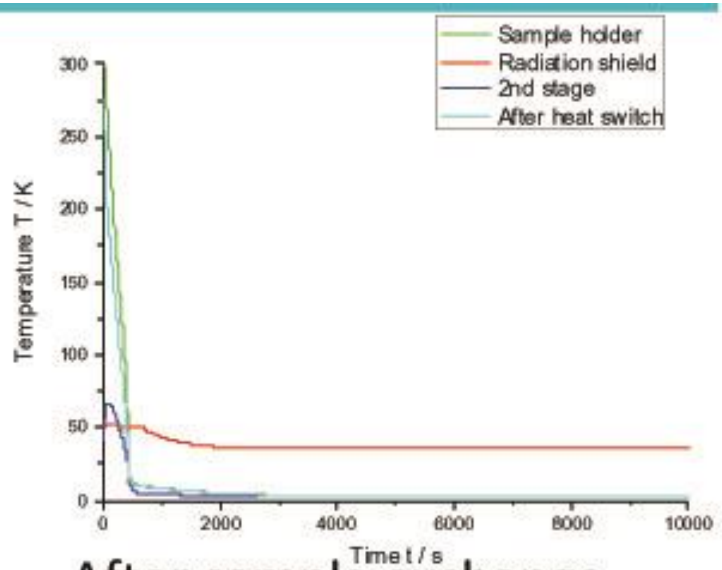
- Separate sample space and cold head isolation vacuum
- Minimized cold mass
- Sample in exchange gas via sample container



Task 4
Cryogen-free cryostat with sample changer

Cooling performances 05 2015





First cooling

1:50 h: 2nd stage at ~2,8 K

2:50 h: 2nd stage at ~2,7 K

2:10 h: Sample at ~3,1 K

$\Delta T \approx 0,4$ K

After sample exchange

0:40 h: 2nd stage at ~2,8 K

0:55 h: 2nd stage at ~2,7 K

0:50 h: Sample at ~3,4 K

$\Delta T \approx 0,6$ K

(0:10 h: sample at ~ 20 K)

- **Robot for sample change under study …**