Task 3: Humidity Chamber

NMI3-Soft Matter JRA-WP20

Matt Barrett *07.12.2012 – Garching*

matthew.barrett@hemlholtz-berlin.de

In this talk…

- Initial outlook
  - Project goal
  - Timeline
  - Participants
- Summary of 22 October
- Moving forward
  - Sensors
  - Equilibration time
- Scientific Interests

Project goal

Develop a humidity chamber which has:

- faster and better controlled temperature and humidity response
- the ability to access large T and RH range
- adaptability to different geometry (SANS, reflectrometry, inelastic)

Timeline

Year 1:

Review the existing systems determine the specifications of the next-generation chambers (goal of 10 mK stability in T and 0.1% stability in RH suggested in proposal)

Year 2:

Produce drawings, obtain components, begin testing

Year 3:

Build and commission chamber

Participants

Task Leader

HZB Helmholtz Zentrum Berlin

Partners

Institut Laue-Langevin (ILL) – Neutrons for Science®

Jülich Forschungszentrum

Observers

Science & Technology Facilities Council

Technische Universität München (TUM)

cea – energie atomique • energies alternatives

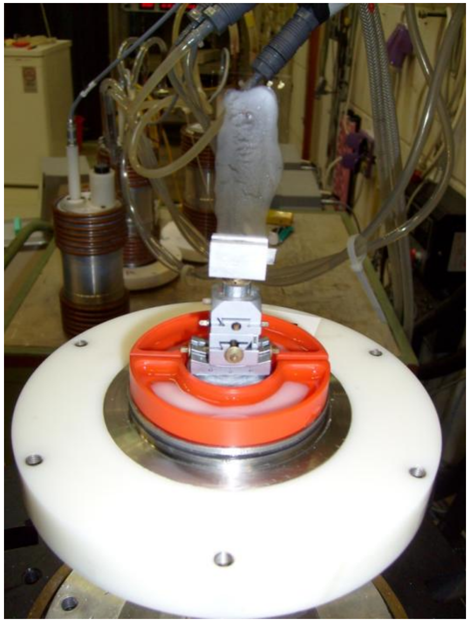
McMaster University

Participants

	HZB	ILL	JCNS
Sub Total Months-Task 3	20 (6+6+7)	7 (2+3+2)	4 (1+1+2)

Summary of 22 October Meeting

- Dirk Wallacher: Introduction, Goals, Timeline
- Thomas Hauß: HZB investigations with fixed humidity
  - Diffraction of membranes containing Aß, cholesterol raft formation
  - Quasi and inelastic scattering of purple membrane

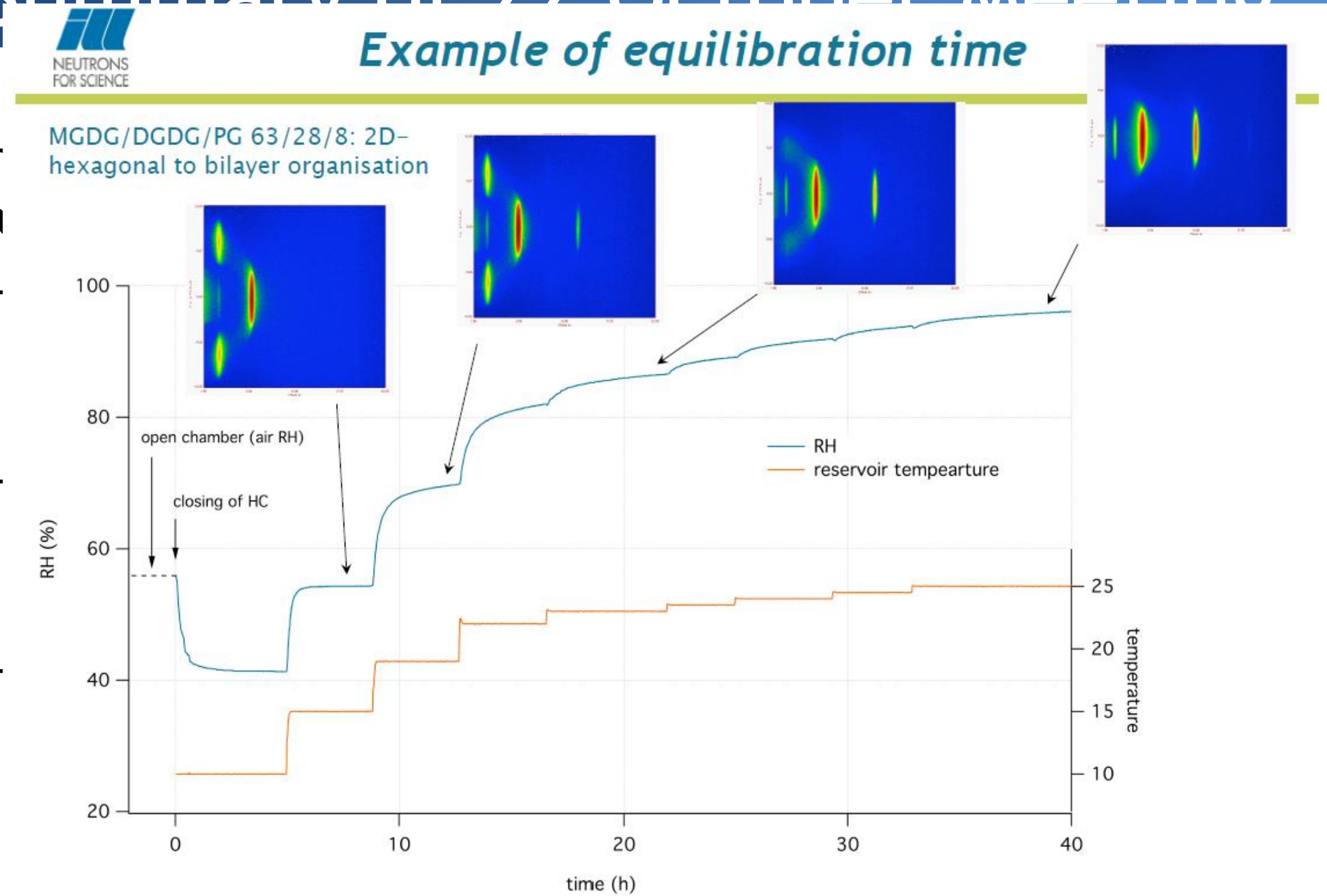


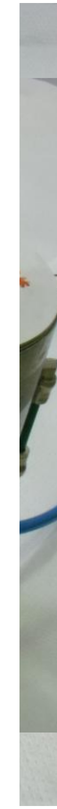
Summary of 22 October Meeting

- Bruno Demé: ILL humidity control capabilites
  - 3 generations of humidity chambers (salt and 2 chamber temperature control)
  - Showed how long equilibration time can change organization from hexagonal to bilayer
  - Presented CMU humidity chamber details

Summary of 22 October Meeting

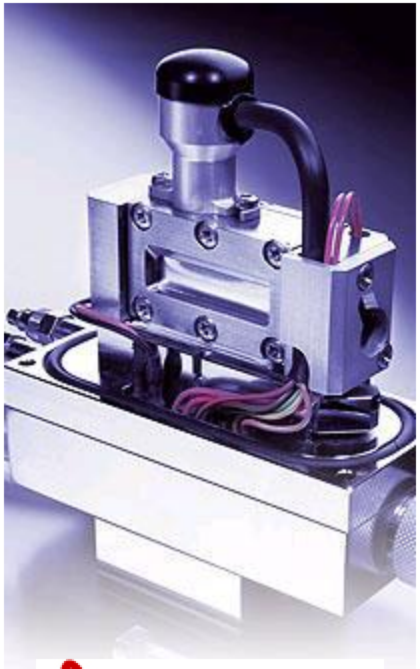
- Br
  ca
  –
  –
  –





Summary of 22 October Meeting

- Noemi Szekely: JNCS-SAXS cell modification for neutrons (Anton Paar)
  - 5-95% RH, 10-60°C, gas mixing
  - Very small cell volume and pre-heated gas flow allows quick environment changes
  - Presented humidity effect on hydrogels and polyelectrolyte membranes



Anton Paar

Summary of 22 October Meeting

- Noemi Szekely: JNCS-SAXS cell modification for neutrons (Anton Paar)
    - 5-95% RH, 10-60°C, gas mixing

Technical specifications	
Operating temperature	
In air, inert gas, vacuum	5 °C to 120 °C
At relative humidity (5% to 95%)	15 °C to 60 °C
Temperature measurement	Pt 100 acc. to DIN 43760
Temperature control	TCU 50 temperature control unit
Control accuracy	± 0.1 °C
Heating	Peltier heating

Dimensions/weight	
Sample holder material	Stainless steel
Sample types	Powders, pastes, foils, films, fiber
Sample size	
Powders, pastes	Approx. 28 mm³
Foils, films	21 x 7 mm

hydrogels and polyelectrolyte membranes

Anton Paar 01/07 C23IP03-A

HZB Helmholtz Zentrum Berlin

Summary of 22 October Meeting

- Matt Barrett – review of existing humidity chambers
  - Salt solution, temperature and water bath control, gas flow and bulk water techniques discussed
  - Presented chart comparing these techniques

Summary of 22 October Meeting

	Humidity ceiling (at 25 C)	Humidity Stability	Automation for RH change	Equilibration time (after RH change)	Contrast variation (H_20/D_20)
Gas flow	~95%	~1% (to be tested)	MFCs	minutes	Bubble through mix
Saturated salt	98% (discrete steps)	Weak temperature dependence	Syringe pumps (Sat. and distilled)	hours	Change of sample/ flow into reservoir
Temperature controlled	≤100%	~1% (to be tested)	Peltier/water bath temp	hours	Flow liquid into reservoir
Fixed humid	Same as salt	No regulation	X	X	Change of sample
Bulk water	Saturated	Stable	X	X	Flow into bulk volume

Summary of 22 October Meeting

DW: New chamber, enhanced and time efficient humidity control over wide parameter range, sample change options, scattering geometries

TH: Long term stability in humidity and temperature, Measurements up to 100% r.H. (or as close as possible), Computer controlled set points of T and r.H., Ease of use

Summary of 22 October Meeting

BD: • Increase in-beam sample vertical section (30 to 45mm) with large Al windows (currently 45mm to 60mm)

- Several samples in 1 can vs several cans on precision translation
- "Perfectly" insulated can (materials + double wall design, no thermal bridge): inside-outside & top-bottom
- Higher precision T & RH sensors (individually calibrated)
- On-chip temperature sensor ?
- A single can design compatible with the two regulation ways (salts double compartment system) if salt solutions exchangeable

Summary of 22 October Meeting

MB:

- Reaching 100% humidity (not using bulk water)
- Avoid temperature gradients = condensation
- Precise measurement of RH and T in-situ
- Contrast variation
- Time for equilibration
- Versatility for all setups
- Ease of use

Summary of 22 October Meeting

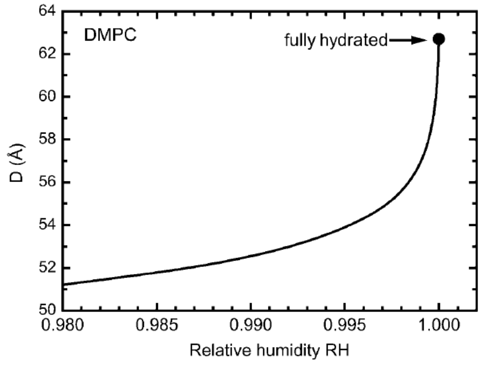
Important questions raised –

- Why is 95% the limit for humidity in gas flow?
- How to speed the equilibration?
- What are the best sensors, how can we avoid sensor constraints?
- What well characterized sample can be used for testing? (DMPC?)

Sensors

- Humidity sensor accuracy
  - Above 95% RH best stated accuracy 1% RH
  - Must test accuracy and reproducibility
- High accuracy T sensors
  - Are RH sensors necessary? $RH=P(T_w)/P(T_s)$
- Determine perfect reference sample
  - Wide humidity dependence, repeatability and accuracy at high RH

Ohmic Industries, 2005

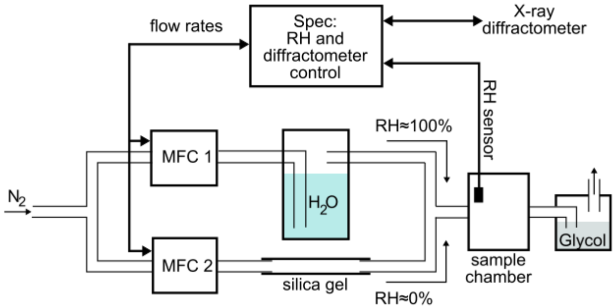


Kučerka, Biophysical Journal, 2005

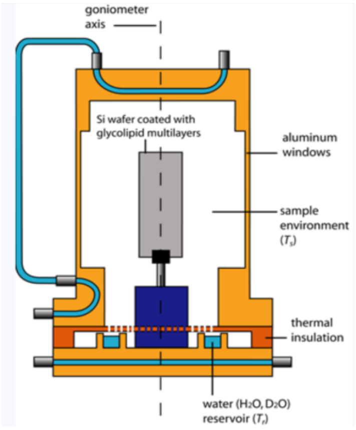
Equilibration time

- Water bath or gas flow
  - See what is the limiting step in equilibration (sensor, sample or container)

- Fan circulation of air
  - Prevent droplet formation and speed equilibration time



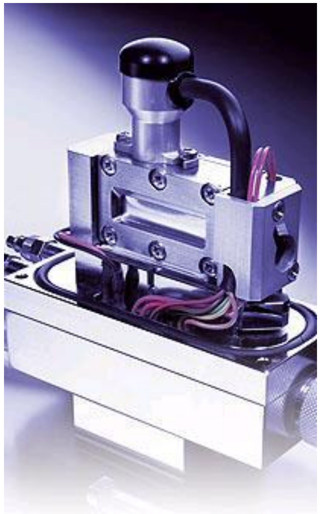
Aeffner, S. *The European physical journal. E*, 2009



Schneck, Phys. Rev. E, 2008

Scientific Visits

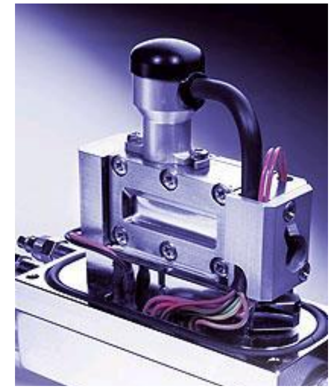
- Garching, JNCS – 31 October
  - Short discussion with Noemi Szekely and Harald Schneider and saw their SANS chamber



- Grenoble, ILL – 29 November
  - Met with Bruno Demé and Giovanna Fragneto to see the multiple generations of humidity chambers
  - Discussed possibility of future tests at ILL

Scientific Visits

- Garching, JNCS – 31 October
  - Short discussion with Noemi Szekely and Harald Schneider and saw their SANS chamber



Report

<u>Humidity chamber full characterization</u>

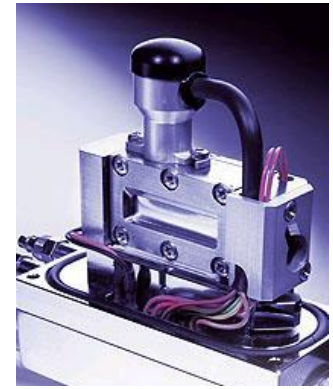
Leide Cavalcanti
Institut Laue Langevin (ILL), Grenoble, FRANCE

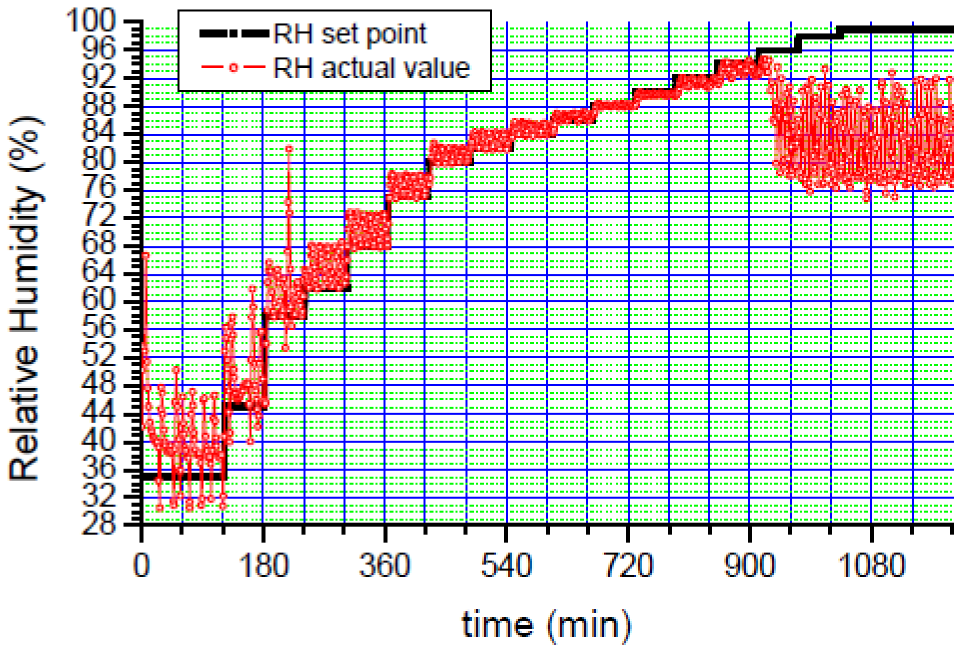
Version: 12 September 2006

This is a partial report on the work of full characterization and improvement of the Humidity Chamber belonging to D17/IN11 and used at elastic and inelastic instruments at the ILL, as D16, D17, IN12, IN8, IN3, IN11 and IN15. This is a contribution to the original project of Maikel Rheinstadter and Giovanna Fragneto and it was performed during the period of July 14th to September 13th, 2006. The work was done with the collaboration of Nadir Belkhier on the electronics and John Allibon on the computing.

Scientific Visits

- Garching, JNCS – 31 October
  - Short discussion with Noemi Szekely and Harald Schneider and saw their SANS chamber

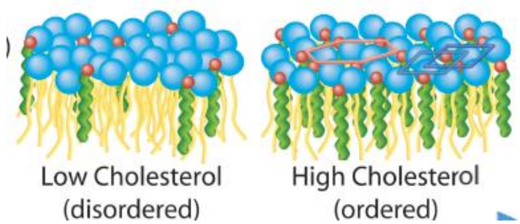


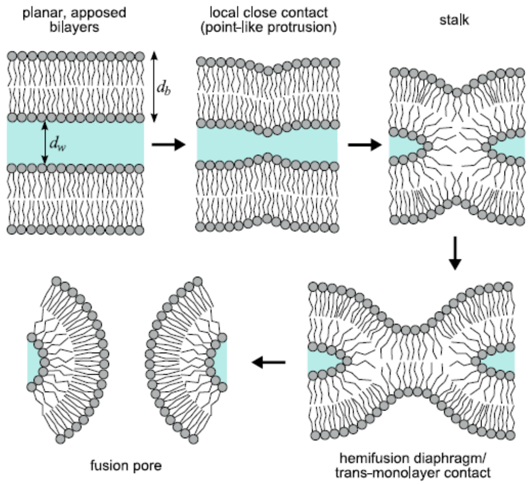


This is a pa… e Humidity Chamber belonging to… D16, D17, IN12, IN8, IN3, IN11 a… tadter and Giovanna Fragneto a… 006. The work was done with the col… e computing.

Scientific Interests

- Cholesterol solubility in DMPC membranes
  - Determine cholesterol solubility limit - where raft like formations occur when approaching physiological conditions (high humidity)
- Stalk formation in membranes
  - Tuneable humidity facilitates phase transition from bilayer to stalk, normally protein facilitated





Aeffner, 2012.

New Humidity Cell

Consensus

- Double walled aluminum cylinder
- Isolation of base from walls (Teflon or ceramic)
- High humidity necessary
- Fast equilibration and long stability necessary

To test

- Gas flow or water troughs
- Addition of circulation fan
- Sensor accuracy and reproducibility
- Finding best ‘characterization sample’
- Several samples in one can/several identical cans

matthew.barrett@helmholtz-berlin.de