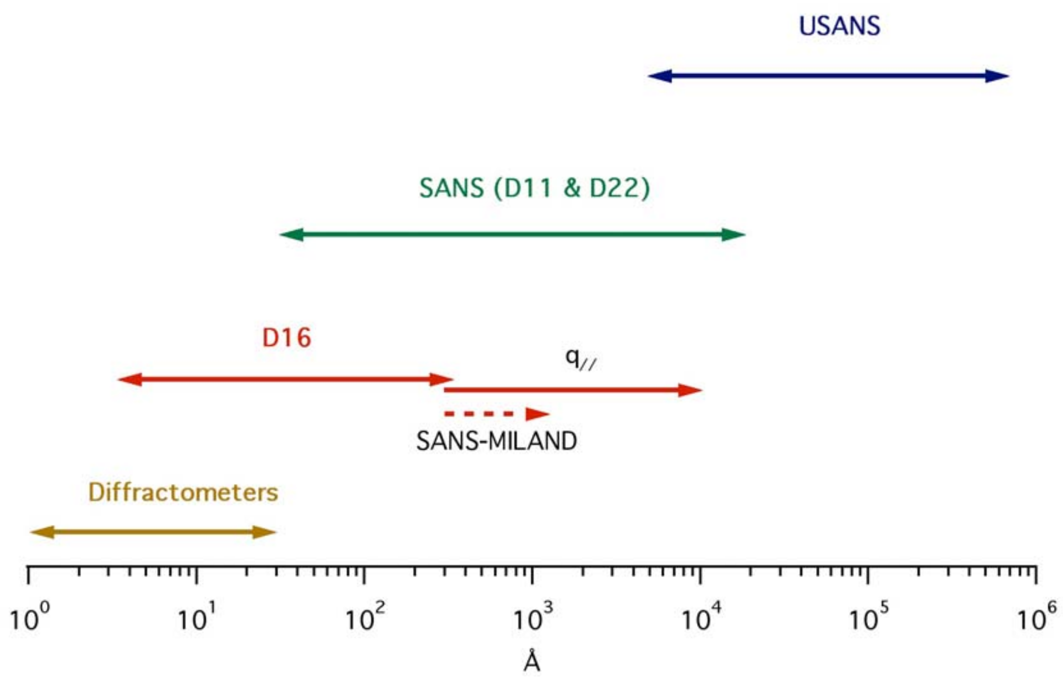
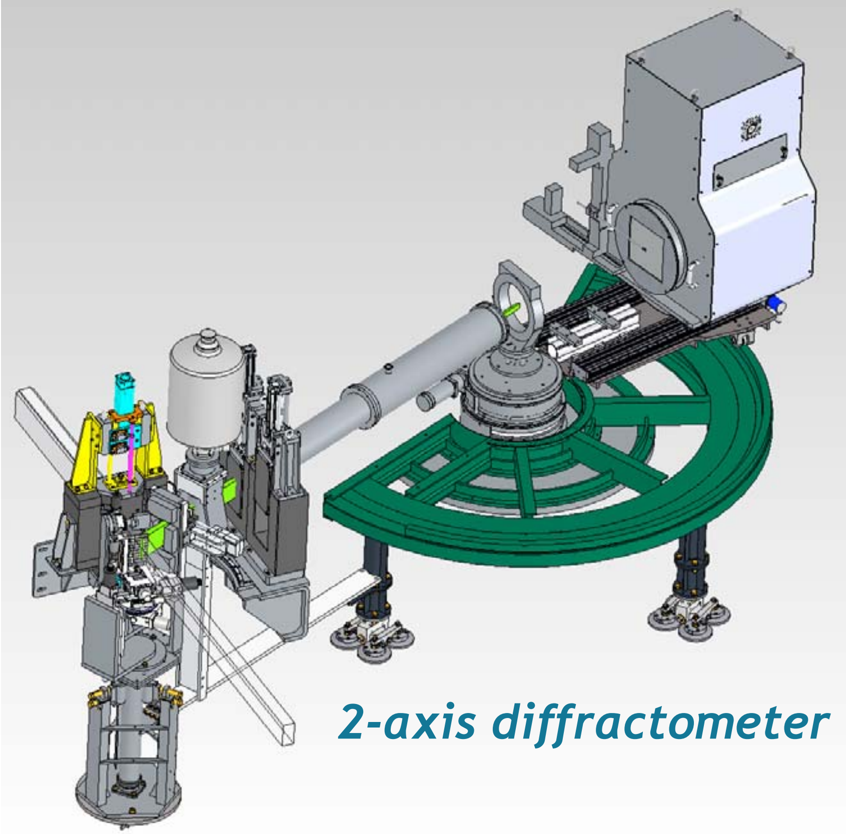
Humidity chambers on D16: past, present, future needs and wishes

Bruno Demé
Institut Laue-Langevin

D16 main characteristics

D16 in real space





2-axis diffractometer

Monochromatic beam: $\lambda = 4.75$ Å and 5.8 Å → *Q-range 0.01–2.5 Å^{-1}*

$\Delta\lambda/\lambda = 0.01$ – *Flux* = 5×10^{6} n.cm^{-2}.s^{-1}

Continuous vertical focussing

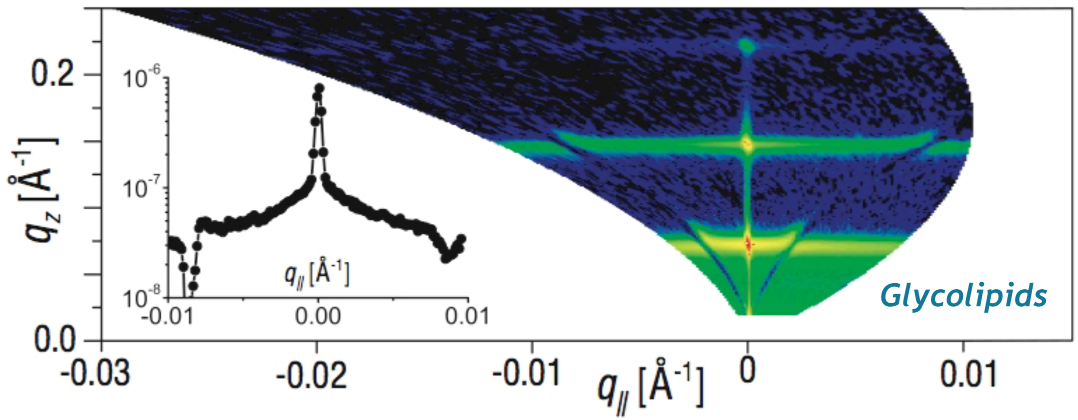
Slit geometry ***(reflectivity on multilayers, powder and liquid diffraction)***

Pinhole geometry ***(SANS, single crystal diffraction)***

Main focus: organic & inorganic multilayers

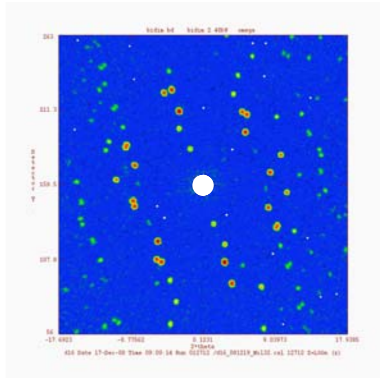
Organic: membrane multilayers

- Membrane models (synthetic lipids and mixtures)
- Lipid extracts (e.g.: thylakoid membrane lipids, skin lipids)
- Biological membranes (purple membrane)

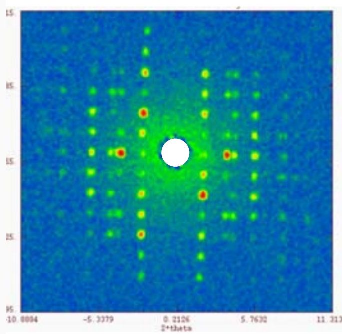


Crystallography

- Single crystals



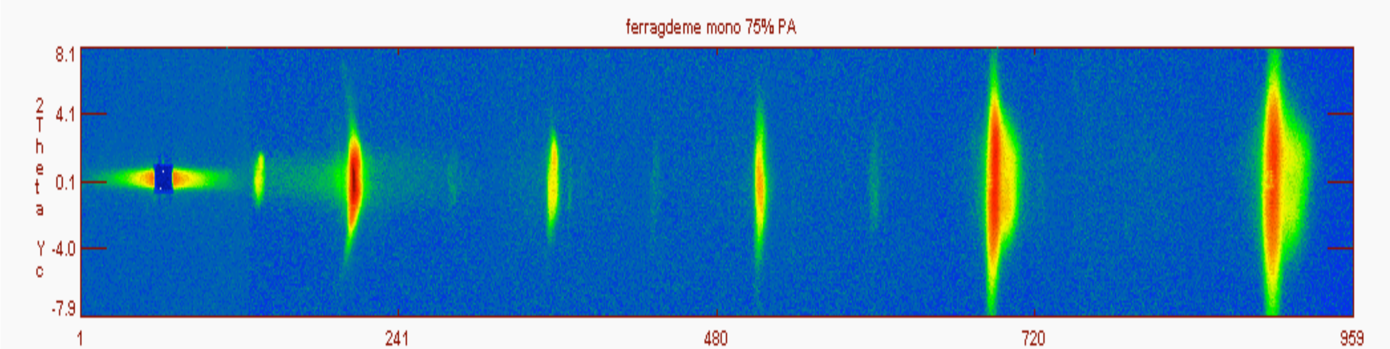
Lysozyme



RC crystal

Inorganic multilayers: clays

Vermiculite



Up to 50% of beamtime with RH control !

Ways of controlling humidity

1. *Saturated salt solutions in equilibrium with air:*

    - *easy and stable*
    - *wide range of salts available -> RH 11% - 98.5%*
    - *weak temperature dependence (no need of high precision T regulation)*
    - *major drawback : needs to open and re-equilibrate from ambient for every new RH*

2. *Two-compartment regulation chamber:* $$RH = \frac{P(Tw)}{P(Ts)}$$

    - *water reservoir generates saturated vapor pressure at $T_w \le T_{sample}$*
    - *easy to change humidity with T controller, no need to open chamber !*
    - *stable only if precise T regulation of both water reservoir (H_2O, D_2O) and sample*

3. External humidity generator that flows humid vapor over the sample

Current situation at the ILL and D16

- Humidity chambers are dedicated D16 sample environments

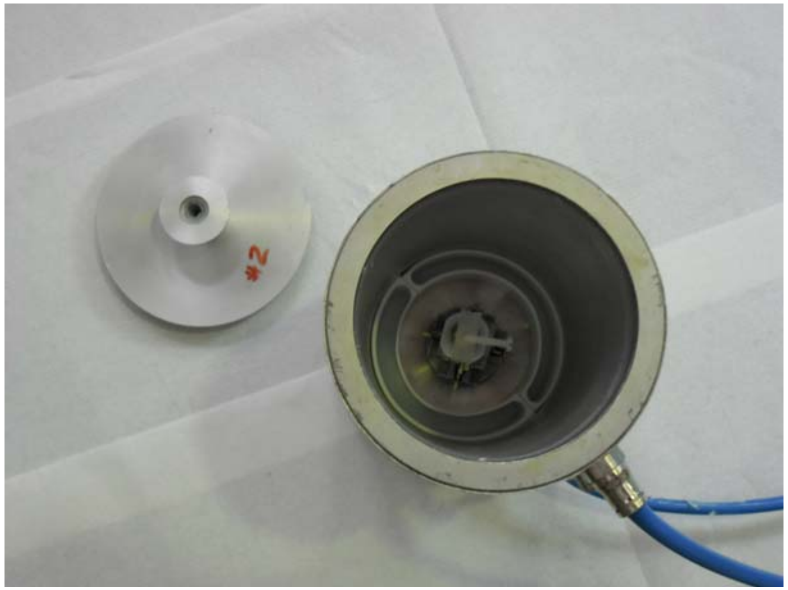
- 2 generations have been intensively used:
- with salt (generation 1): 4 chambers (3 large, 1 small)
- no salt (generation 2): 2 chambers

- Generation 3 (no salt): rather unsuccessful design that has been modified (1 chamber)

- New design (generation 4) started before it became one of the tasks of current FP7 – JRA on sample environment for soft condensed matter

Generation 1: large (3 identical units)

Base can be regulated but not cover (removable)



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Generation 1: small (1 unit)

with cover removable and regulated (but not the base…)

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Relative humidities from saturated salt solutions

Relative Humidity (%RH)			
Temperature °C	Lithium Chloride	Potassium Acetate	Magnesium Chloride
0	11.23 ± 0.54		33.66 ± 0.33
5	11.26 ± 0.47		33.60 ± 0.28
10	11.29 ± 0.41	23.28 ± 0.53	33.47 ± 0.24
15	11.30 ± 0.35	23.40 ± 0.32	33.30 ± 0.21
20	11.31 ± 0.31	23.11 ± 0.25	33.07 ± 0.18
25	11.30 ± 0.27	22.51 ± 0.32	32.78 ± 0.16
30	11.28 ± 0.24	21.61 ± 0.53	32.44 ± 0.14
35	11.25 ± 0.22		32.05 ± 0.13
40	11.21 ± 0.21		31.60 ± 0.13
45	11.16 ± 0.21		31.10 ± 0.13
50	11.10 ± 0.22		30.54 ± 0.13
55	11.03 ± 0.23		29.93 ± 0.16
60	10.95 ± 0.26		29.26 ± 0.18
65	10.86 ± 0.29		28.54 ± 0.21
70	10.75 ± 0.33		27.77 ± 0.25
75	10.64 ± 0.38		26.94 ± 0.29
80	10.51 ± 0.44		26.05 ± 0.34
85	10.38 ± 0.51		25.11 ± 0.39
90	10.23 ± 0.59		24.12 ± 0.46
95	10.07 ± 0.67		23.07 ± 0.52
100	9.90 ± 0.77		21.97 ± 0.60

1 bath to regulate both reservoir and sample

Relative Humidity (%RH)						
Temperature °C	Potassium Carbonate	Magnesium Nitrate	Sodium Chloride	Potassium Chloride	Potassium Nitrate	Potassium Sulfate
0	43.13 ± 0.66	60.35 ± 0.55	75.51 ± 0.34	88.61 ± 0.53	96.33 ± 2.9	98.77 ± 1.1
5	43.13 ± 0.50	58.86 ± 0.43	75.65 ± 0.27	87.67 ± 0.45	96.27 ± 2.1	98.48 ± 0.91
10	43.14 ± 0.39	57.36 ± 0.33	75.67 ± 0.22	86.77 ± 0.39	95.96 ± 1.4	98.18 ± 0.76
15	43.15 ± 0.33	55.87 ± 0.27	75.61 ± 0.18	85.92 ± 0.33	95.41 ± 0.96	97.89 ± 0.63
20	43.16 ± 0.33	54.38 ± 0.23	75.47 ± 0.14	85.11 ± 0.29	94.62 ± 0.66	97.59 ± 0.53
25	43.16 ± 0.39	52.89 ± 0.22	75.29 ± 0.12	84.34 ± 0.26	93.58 ± 0.55	97.30 ± 0.45
30	43.17 ± 0.50	51.40 ± 0.24	75.09 ± 0.11	83.62 ± 0.25	92.31 ± 0.60	97.00 ± 0.40
35		49.91 ± 0.29	74.87 ± 0.12	82.95 ± 0.25	90.79 ± 0.83	96.71 ± 0.38
40		48.42 ± 0.37	74.68 ± 0.13	82.32 ± 0.25	89.03 ± 1.2	96.41 ± 0.38
45		46.93 ± 0.47	74.52 ± 0.16	81.74 ± 0.28	87.03 ± 1.8	96.12 ± 0.40
50		45.44 ± 0.60	74.43 ± 0.19	81.20 ± 0.31	84.78 ± 2.5	95.82 ± 0.45
55			74.41 ± 0.24	80.70 ± 0.35		
60			74.50 ± 0.30	80.25 ± 0.41		
65			74.71 ± 0.37	79.85 ± 0.48		
70			75.06 ± 0.45	79.49 ± 0.57		
75			75.58 ± 0.55	79.17 ± 0.66		
80			76.29 ± 0.65	78.90 ± 0.77		
85				78.68 ± 0.89		
90				78.50 ± 1.0		
95						
100						

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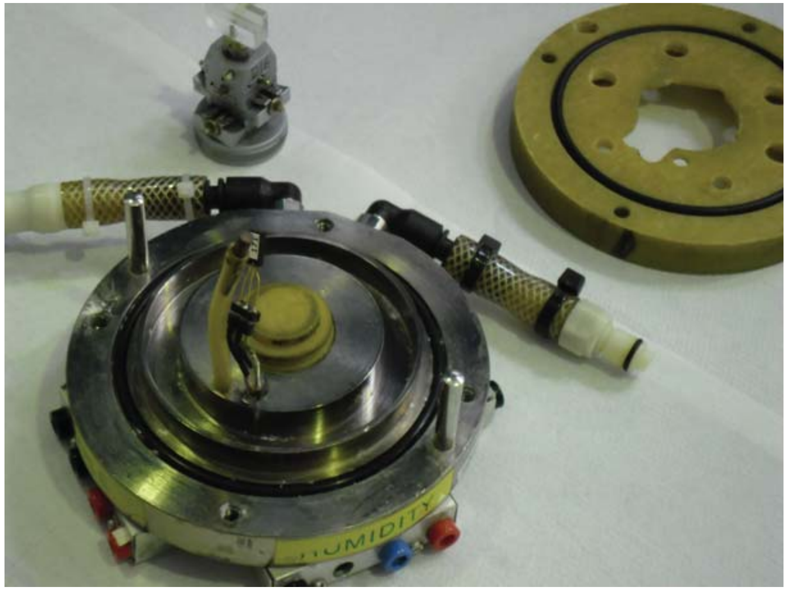
Generation 2: double compartment, no salt solutions

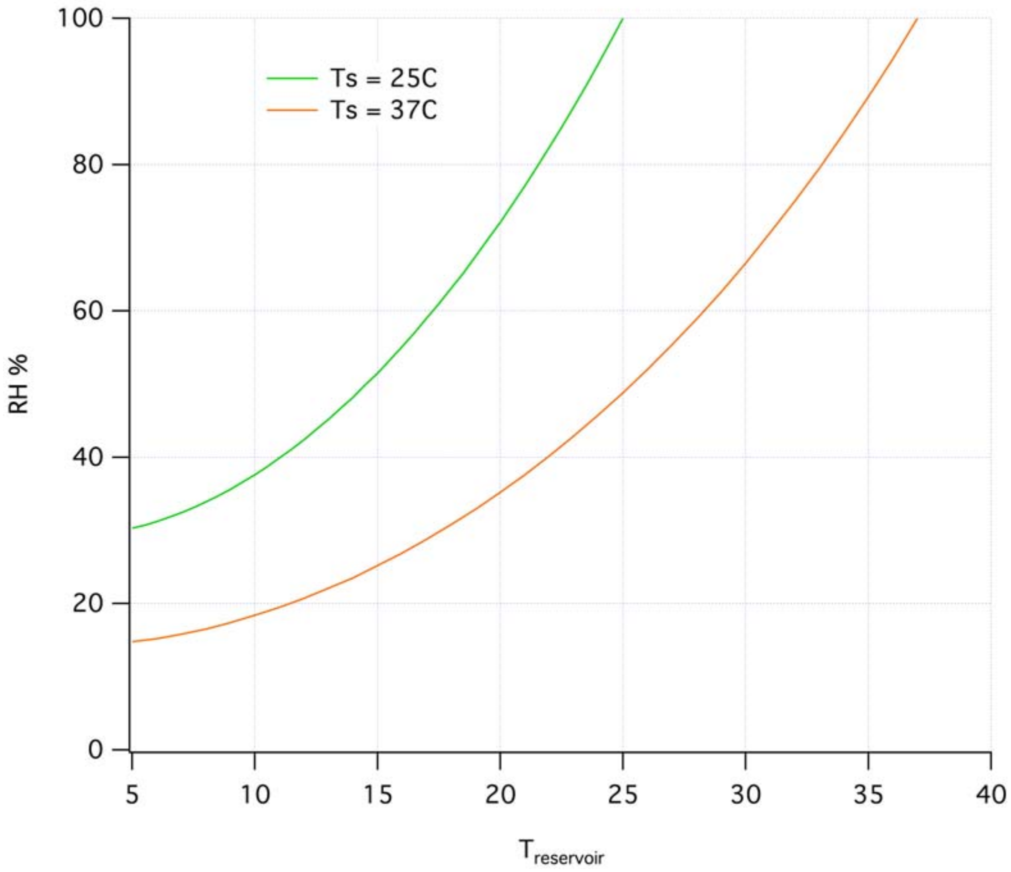


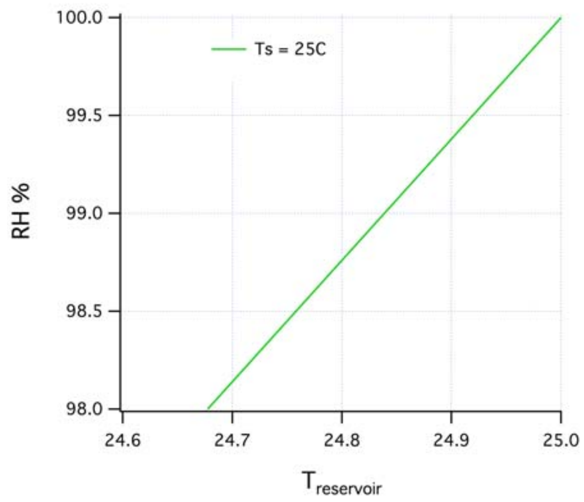
2 independent water baths regulate the bottom (reservoir, T_w) and the top (sample, T_s)

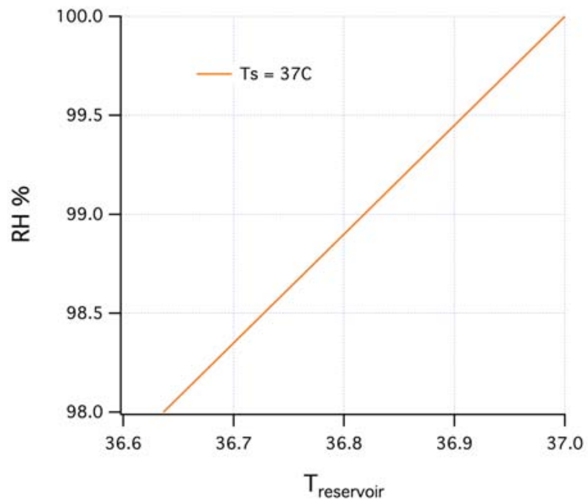
$$RH = \frac{P(Tw)}{P(Ts)}$$

Very important:
bottom well insulated from top

Example of RHs available with double T regulation





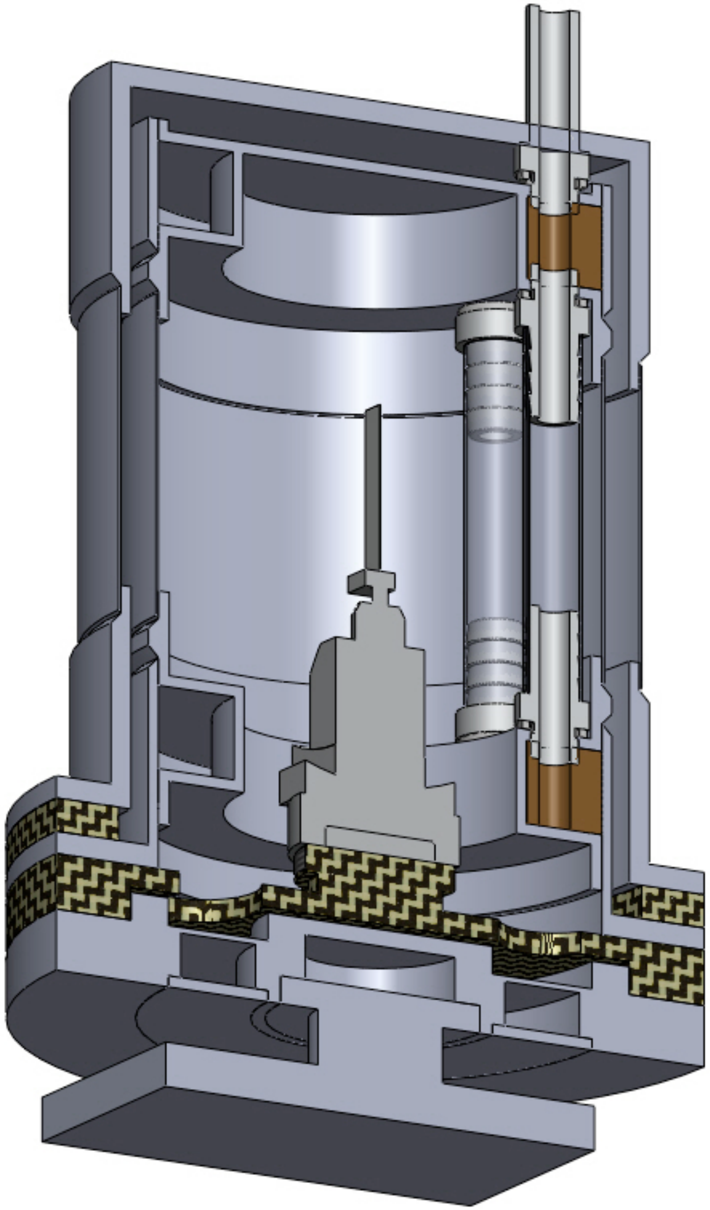


On the D16 rotation table

- 2 Haake bath with +/-0.01C regulation around setpoint, can also regulate on sample

- control and readout by NOMAD software:
  - 2 regulations
  - 2 PT100 sensors (reservoir and sample)
  - 1 humidity sensor

Current developments (4^{th} generation)



Improved insulation and fluid circulation

MOTIVATION for a 4th generation

- More reliable (calibrated) humidity and T sensors
- More temperature sensors
- Save equilibration time (2-3 samples in chamber ?)
- Identical chambers (2-3 for D16, 2 more in the ILL sample environment pool)
- Become standard ILL sample environment
- Agree on design and specifications
- Feedback from other experiences (e.g.: S. Nagle project at CMU)

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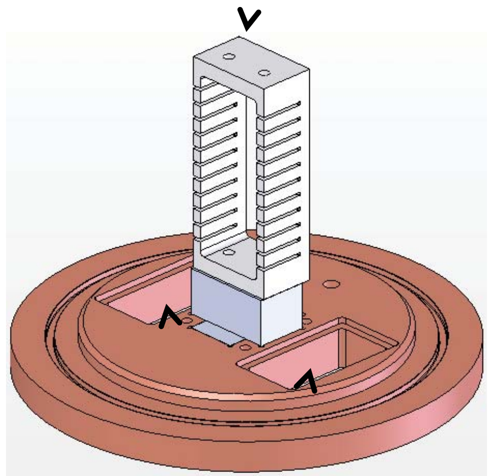
CMU humidity chamber for use at NCNR

Slatted sample holder



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Wells for water or D_2O

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From Conception to Reality

1. **Proposal for humidity chamber was written by Prof. Stephanie Tristram-Nagle and Dr. Frank Heinrich after preliminary data were collected using x-rays at CMU to test the SANS humidity chamber. Proposal was funded at the level of $77K in May, 2008.**

2. **First meeting at NIST in December, 2008. People present: Joe Dura, Frank Heinrich, Andrew Jackson, Juscelino Leao, Dan Dender, Susan Krueger, Stephanie Tristram-Nagle (all Ph.D.'s).**

3. **Goals for the humidity chamber set forth at the first meeting:**

- **Achieve all humidities from 0 to 100 % RH in a reasonable amount of time. Alternate goal, have 2 or 3 chambers for this purpose.**
- **Control humidity by flowing a humidified gas into chamber.**
- **Reduce condensation by using external light bulbs and double walls on the can.**
- **Provide homogenous internal humidity - have an internal circulating fan, circular well.**
- **Exchange humidities quickly – provide liquid exchange without opening chamber.**
- **Hold several samples at once – have a multi-slatted sample holder.**
- **View inside – have a fiberscope.**
- **Accurately measure humidity – have a chilled mirror hygrometer.**

3. **After fabrication and testing, many of these goals were reached, while some concepts were not successful.**

http://www.humidity.frank-heinrich.net/

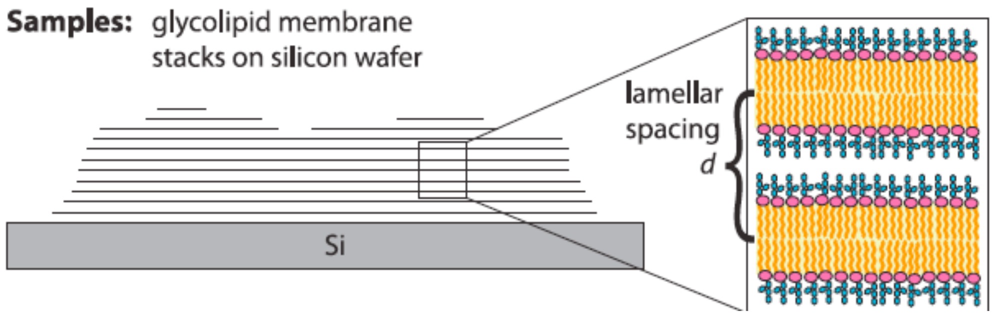
Soft Matter and biology sample environment

Humidity chamber with H_2O/D_2O exchange

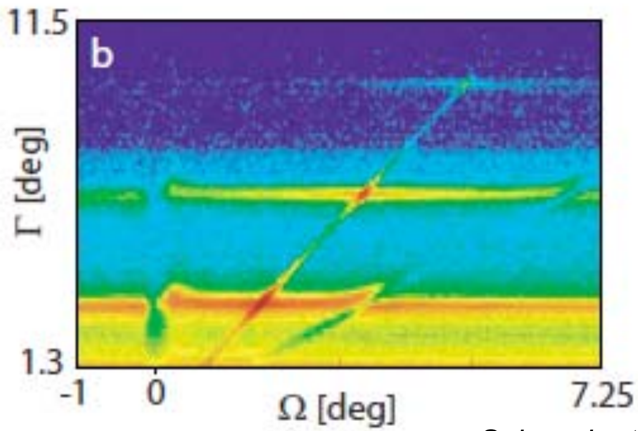
- Temperature range: 10 – 80 C
- Humidity up to 100% with H_2O/D_2O exchange
- Salt solution at fixed humidity

Membrane-bound saccharide chains cell glycocalix on the membrane mechanics

Glycolipid membrane multilayers on planar silicon substrates



→ The specular and off-specular neutron scattering

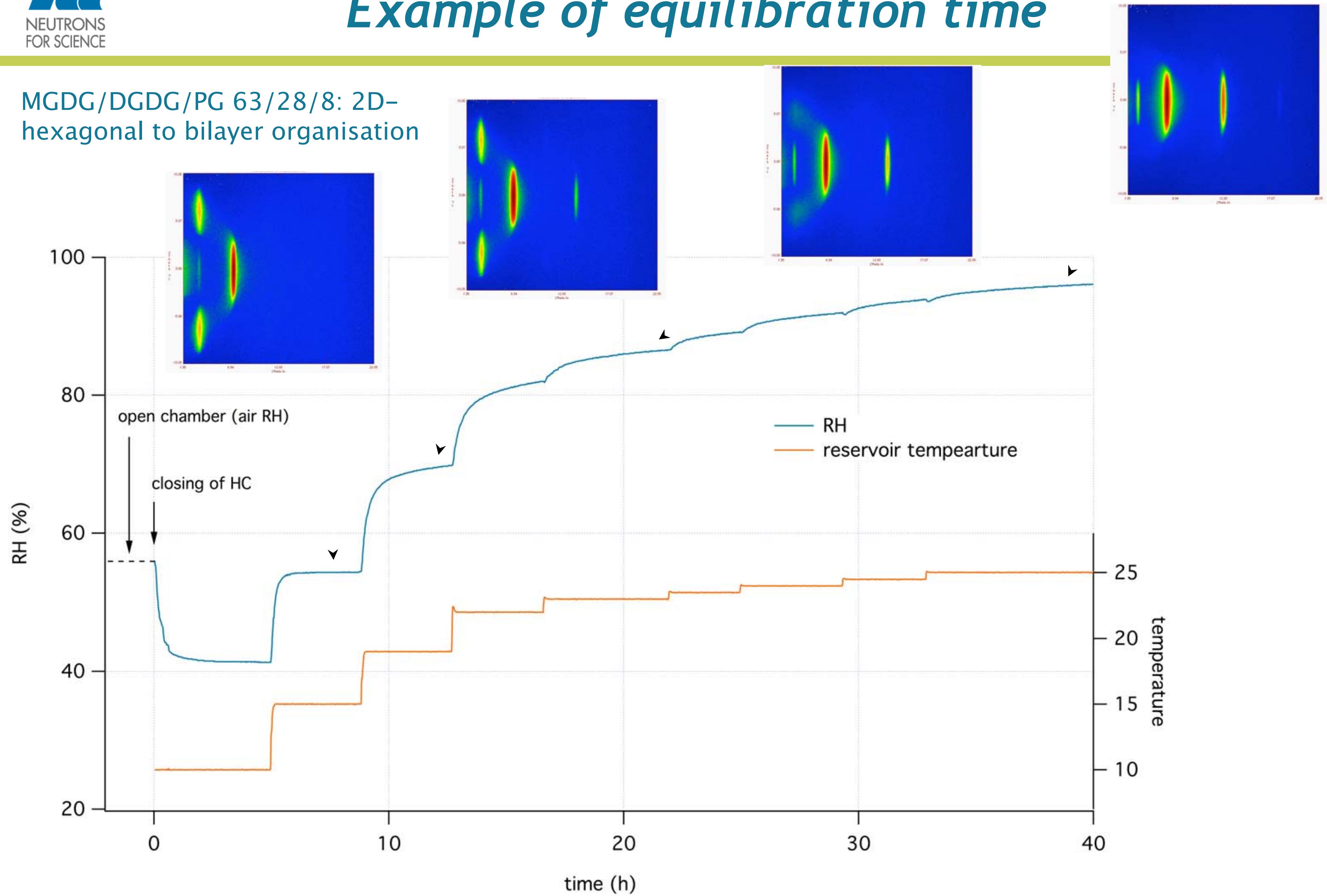


Out-of-plane **and** ***in-plane*** **scattering vector components**

✍ *Schneck et al. Phys. Rev. E, 78 61924 (2008)*

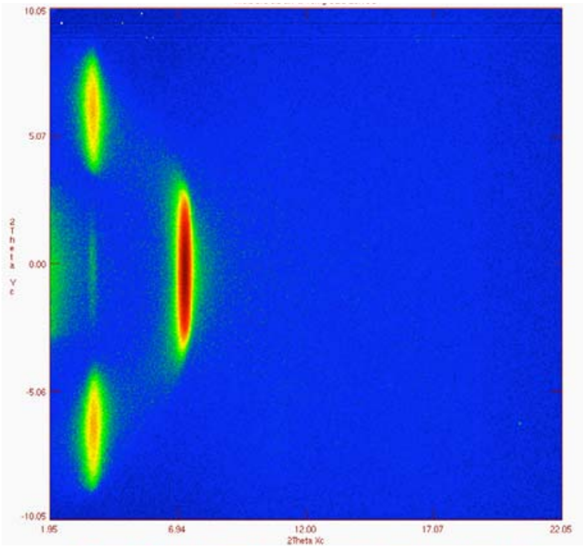
Example of equilibration time

MGDG/DGDG/PG 63/28/8: 2D-hexagonal to bilayer organisation

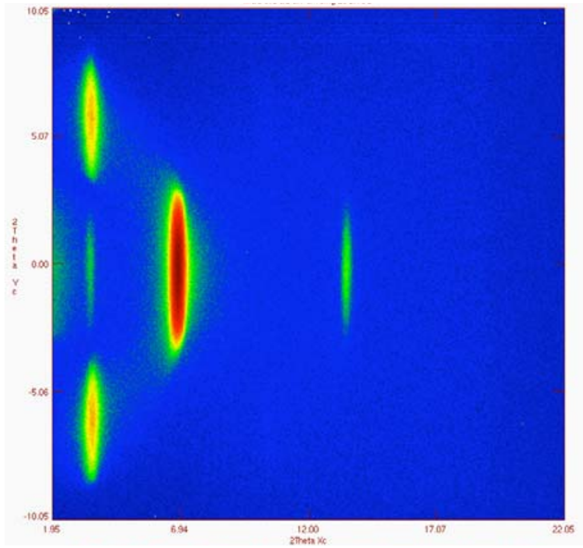


Hexagonal to bilayer phase transition in ternary plant lipid mixture: MGDG/DGDG/PG 63/28/8

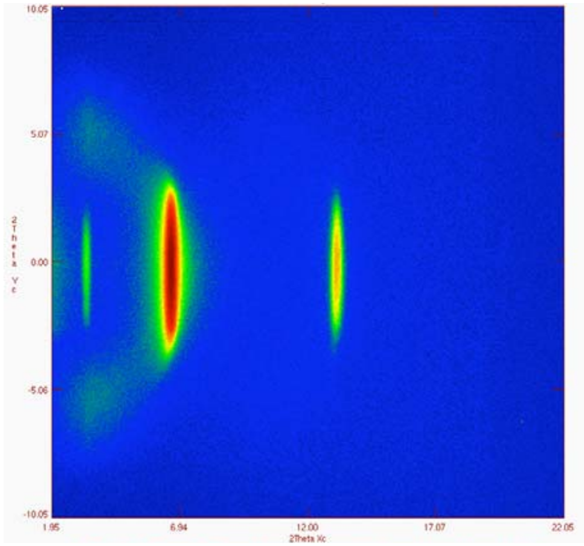
38 % humidity



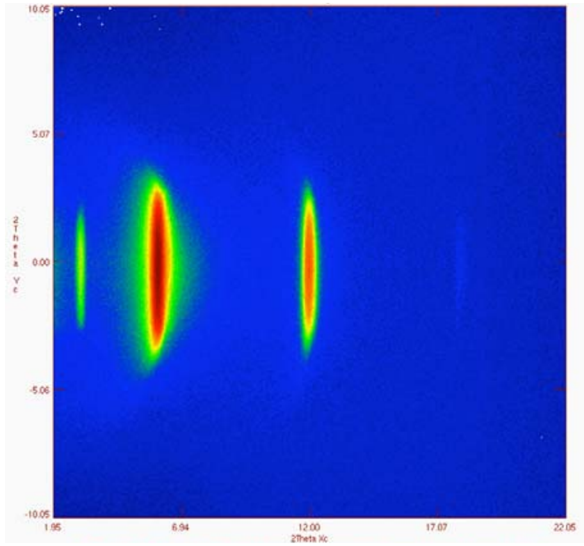
68 % humidity



82 % humidity



99 % humidity



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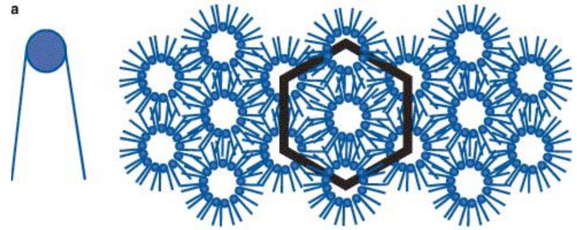
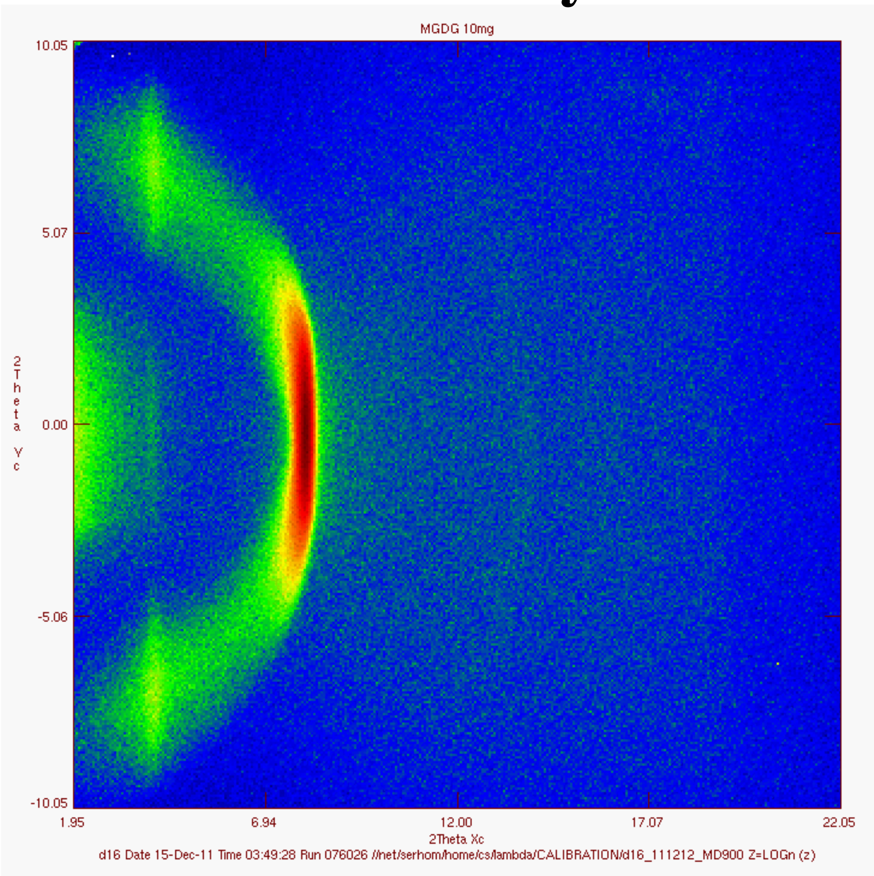
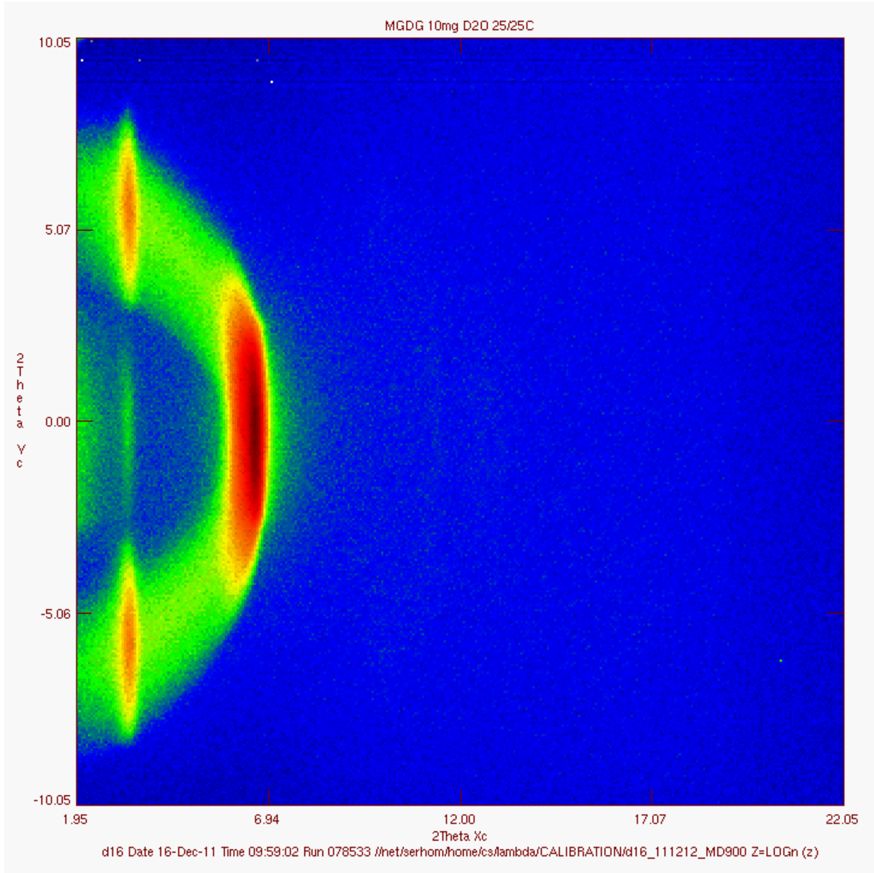
MGDG

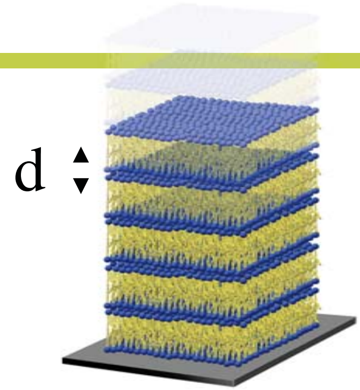


Relative humidity: 38 % **99 %**





DGDG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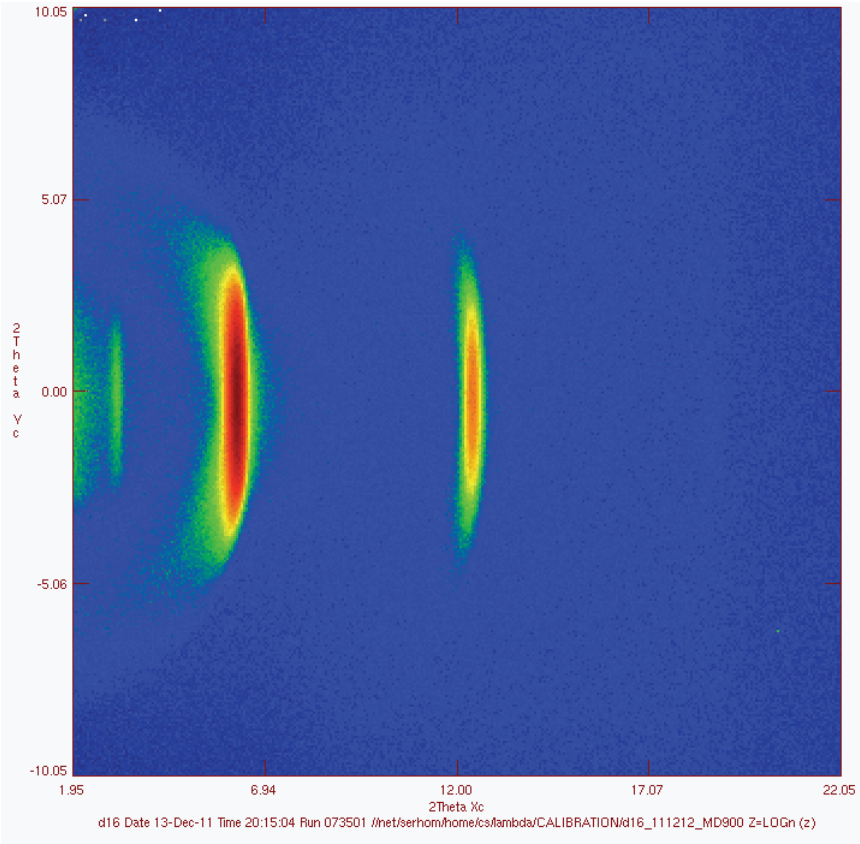
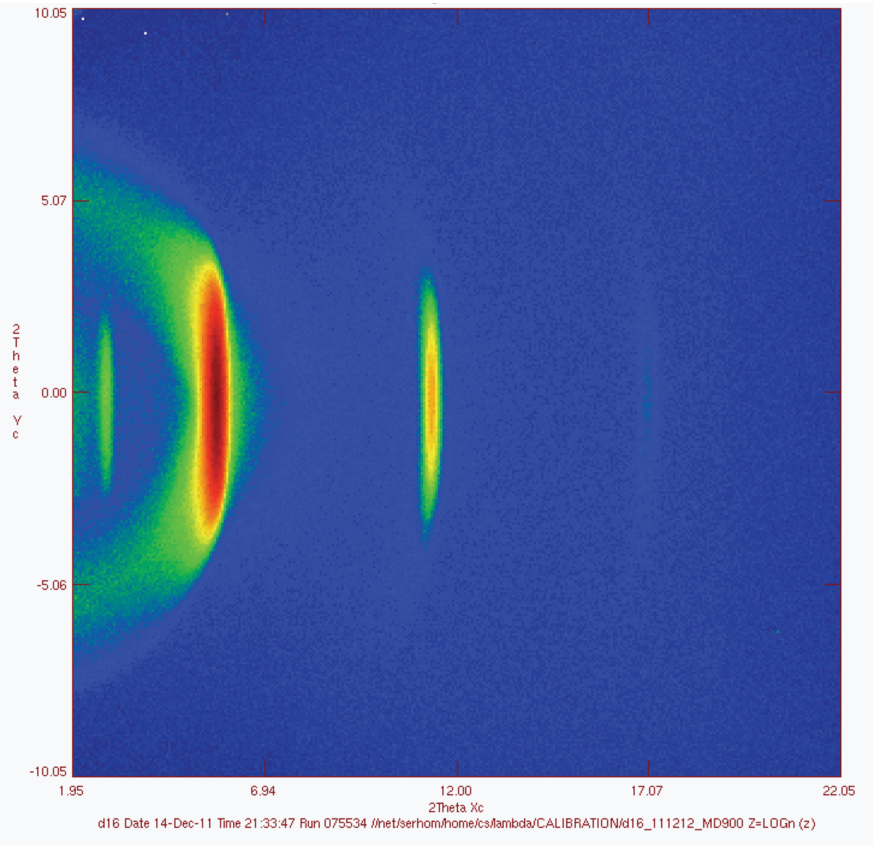
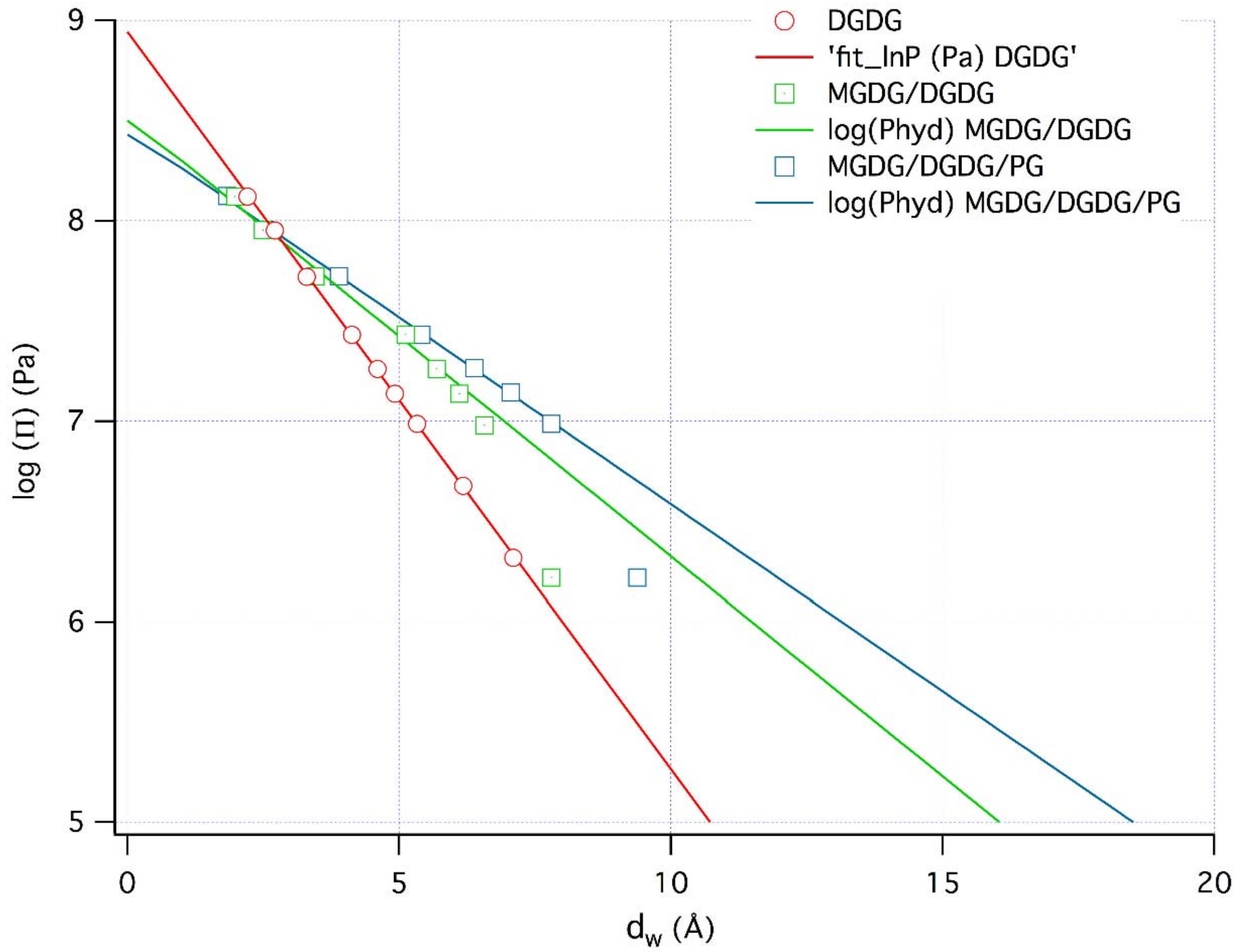
RH 38 %

99 %

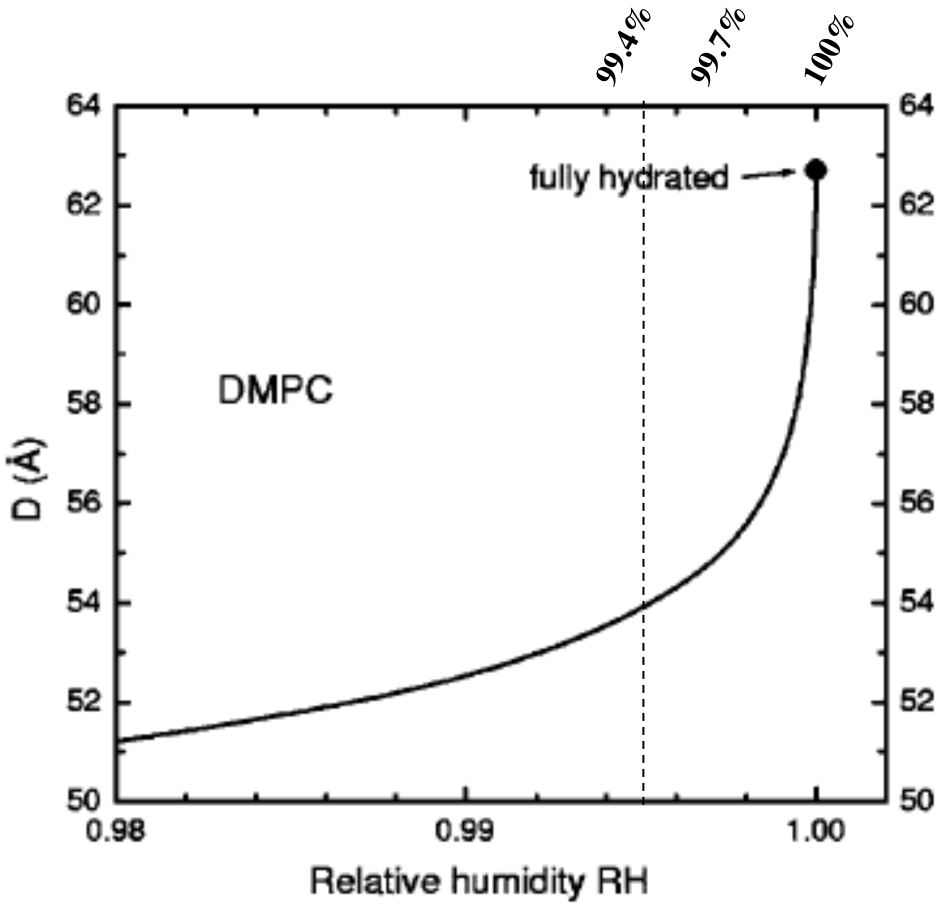

10.05
5.07
0.00
-5.06
-10.05
2Theta Yc
1.95
6.94
12.00
17.07
22.05
2Theta Xc
d16 Date 13-Dec-11 Time 20:15:04 Run 073501 //net/serhom/home/cs/lambda/CALIBRATION/d16_111212_MD900 Z=LOGn (z)


10.05
5.07
0.00
-5.06
-10.05
2Theta Yc
1.95
6.94
12.00
17.07
22.05
2Theta Xc
d16 Date 14-Dec-11 Time 21:33:47 Run 075534 //net/serhom/home/cs/lambda/CALIBRATION/d16_111212_MD900 Z=LOGn (z)

Result: force vs bilayer distance



Why precision is required at high humidity ?



Chu et al. (2005), Phys. Rev. E 71:041904.

Conclusion: needs

- ***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 communication cable for all sensors (T1, T2,..., RH)***
- ***A single can design compatible with the two regulation ways (salts double compartment system) if salt solutions exchangeable***

To be avoided

- ***External humidity generator + air flow (CMU)***
- ***Poorly insulated cans (ILL's generation 3 initial design)***
- ***(Alternative to) Honeywell humidity sensors ?***

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Welcome to the **Humidity Chamber Project Page**. See the menu on the left for project documents.

- *May 10, 2011:* SummaryTalk.pdf, SummaryTalk.ppt, given by Prof. S. Tristram-Nagle at NCNR.
- *December 21, 2010:* Testing of CMU hydration chamber carried out at CMU by S. Tristram-Nagle and K. Akabori.
- *September 2, 2009:* Telephone Conversation between S. Tristram-Nagle and D. Dender discussing some details of the new chamber design.
- *August 2009:* Meeting between S. Tristram-Nagle and F. Heinrich discussing the new chamber design. Drawings were finalized and these will be evaluated by NIST scientists before commencing fabrication of the chamber.
- *June 2009:* Meeting between S. Tristram-Nagle and F. Heinrich discussing the new chamber design
- *May 2009:* Second testing of the SANS chamber with active humidity control on the NG7-reflectometer
- *April 2009:* D. Dender published top-level specifications for the new chamber
- *March 2009:* First testing of the SANS chamber with active humidity control and/or external humidity generator on the NG7-reflectometer
- *December 2008:* Meeting between S. Tristram-Nagle, F. Heinrich, D. Dender, J. Dura, C. Majkrzak, J. Leao, and A. Jackson discussing the new chamber design
- *December 2008:* First drawings for the new humidity chamber based on the SANS humidity chamber

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