



Task 3:

Humidity Chamber

NMI3-Soft Matter JRA-WP20

Overview of existing equipment
and applied methods

Matt Barrett

22.10.2012 - Berlin

Humidity control methods

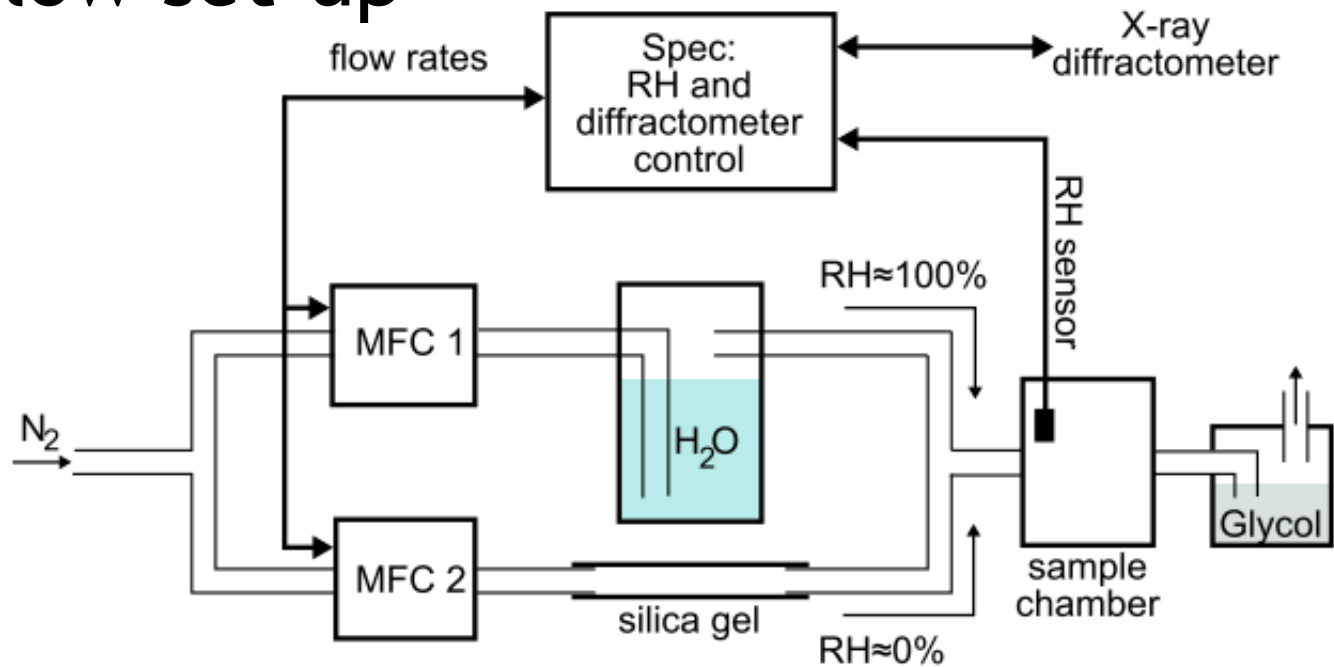
- Gas flow
- Saturated salt solutions
- Temperature control
- Fixed humidity preparation
- Bulk water

Systems in use

- IRP (Göttingen) - X-ray gas flow
- HZB(Berlin) - salt solution/water bath
- ILL(Grenoble) - salt solution/water bath
- CNBC (Chalk River) - bulk water, sponge (refl.)
- McMaster (Hamilton) - salt solution/3 Peltier
- NIST (Gaithersburg) - salt solution/water bath
- CMU (Pittsburgh) - salt solution/2 Peltier/gas flow
- Others?

Gas Flow

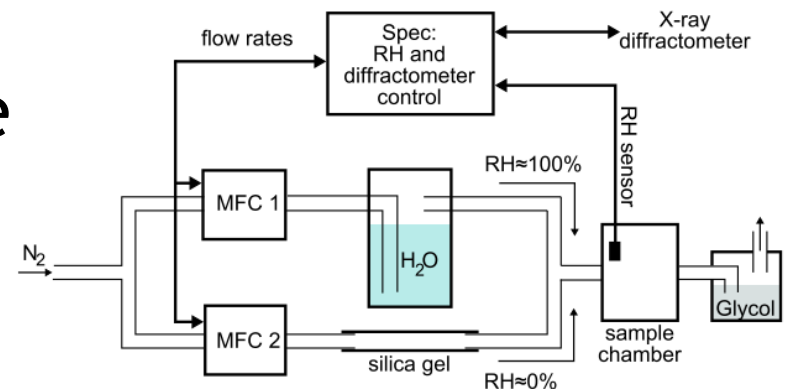
- Salditt group:
X-ray flow set-up



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

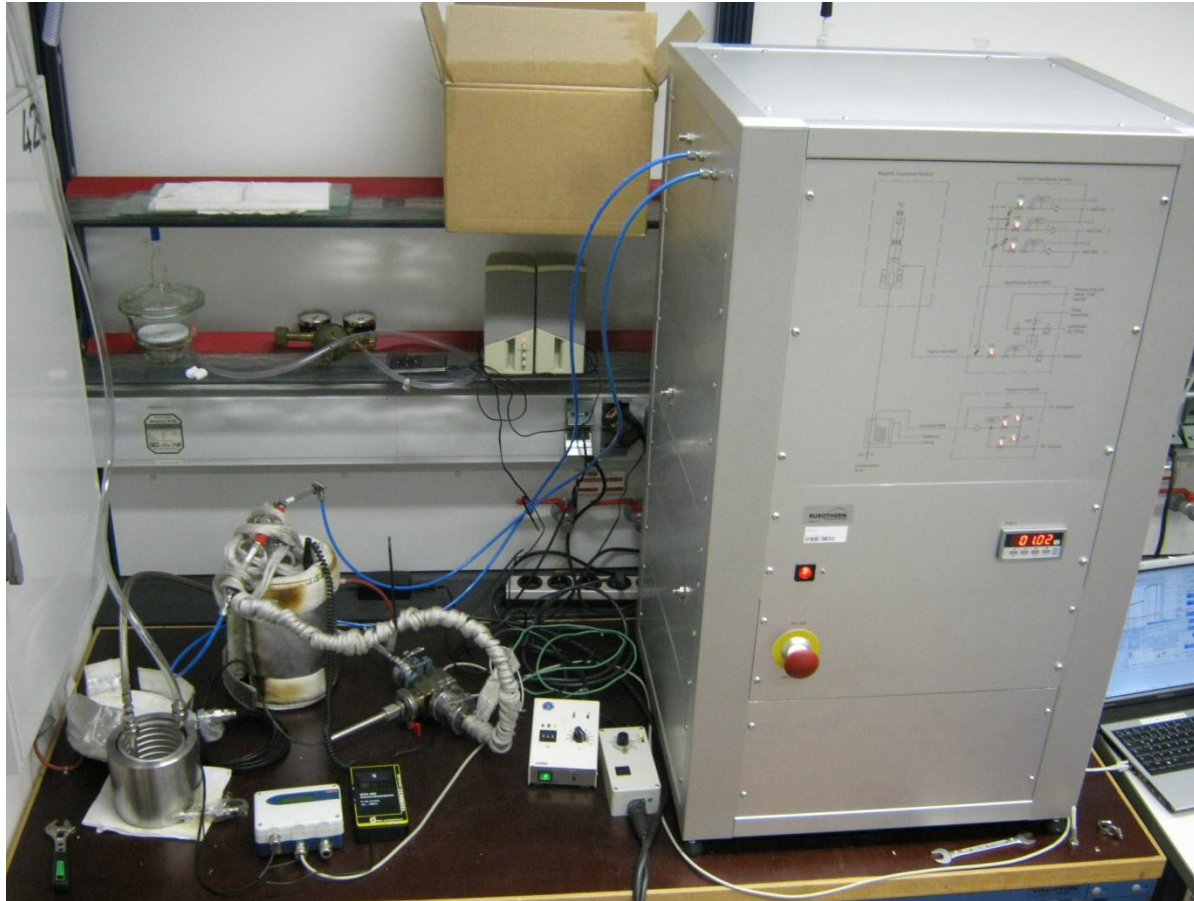
Gas Flow

- ✓ Continuous humidity control from low to ~95%
- ✓ Fast equilibration
- ✓ Easy RH and Temp change
- x Limited by sensor accuracy
- x Temperature gradients = condensation



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

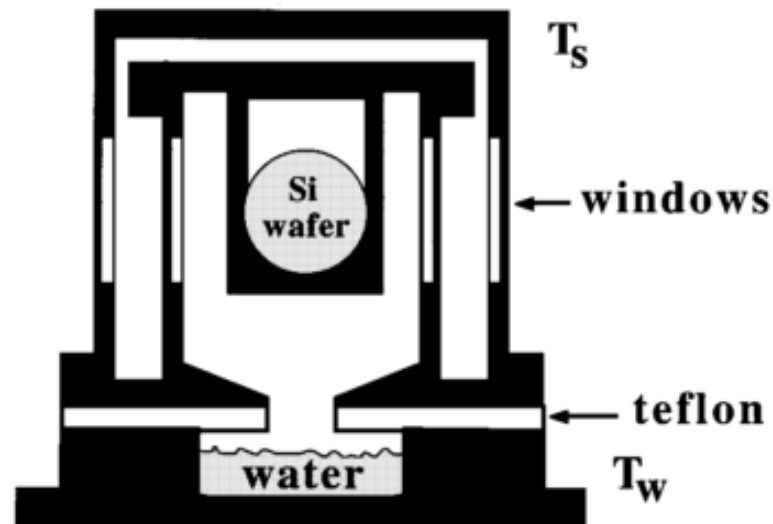
Gas Flow (At HZB)



Saturated Salts

- Wu group

Simple humidity can



Sirota, J. Chem Phys, 1996

Saturated Salts

- Wu group

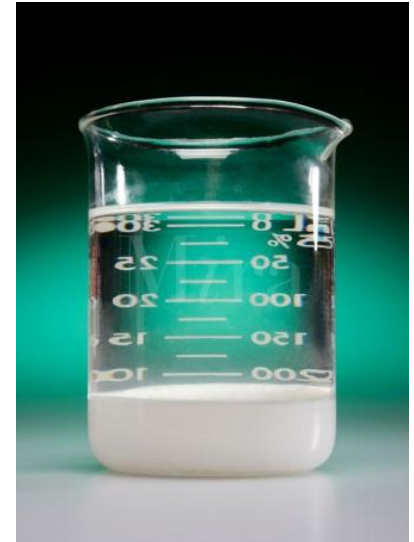
Simple humidity can

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

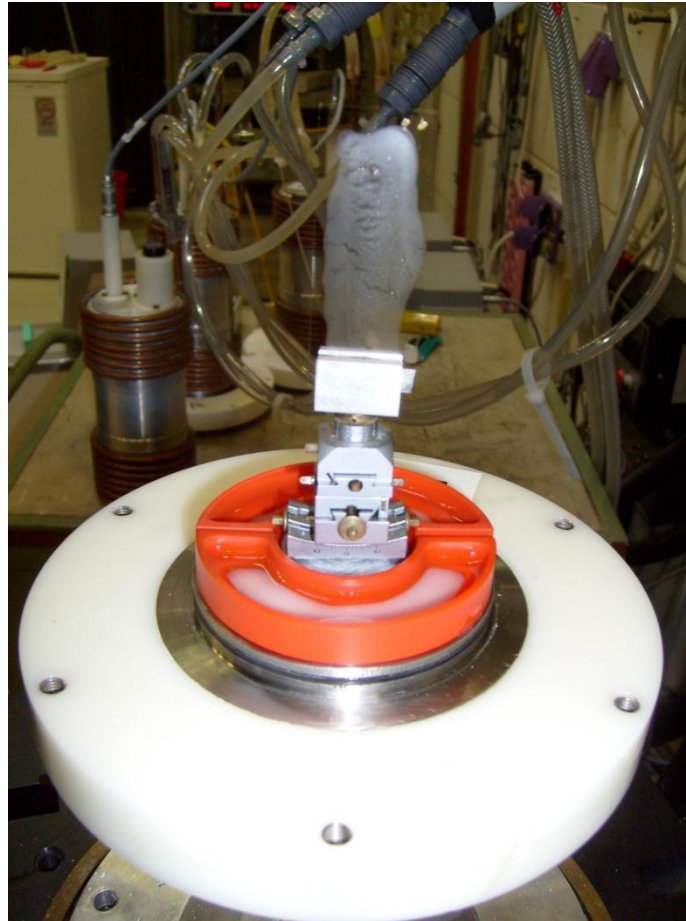
Omega Process Measurement and Control

Saturated Salts

- ✓ Precise and reliable humidity
- ✓ No need for humidity sensor
- x Cleaning salts
- x Re-equilibration
- x Discontinuous (change salt concentration to fill gaps)

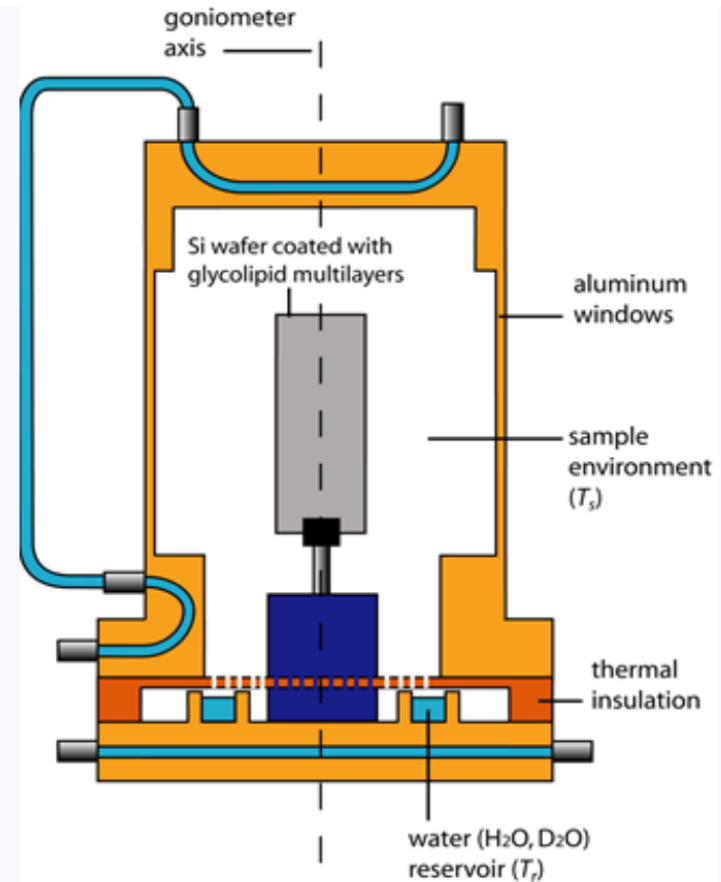


Saturated Salts (at HZB)



Temperature Control

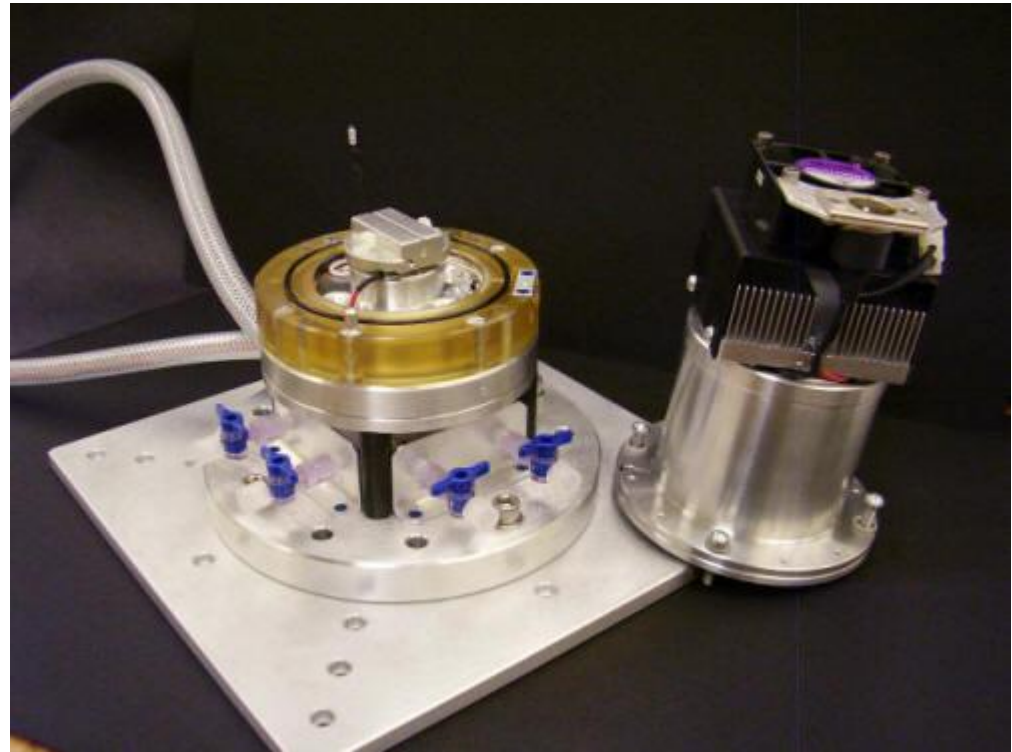
- ILL neutron humidity environment



Schneck, *Phys. Rev. E*, 2008

Temperature Control

- ✓ Individual components adjustable T
- ✓ $\text{H}_2\text{O}/\text{D}_2\text{O}$ exchange
- x Condensation
- x 100% RH?
- x Limited by sensor accuracy



Rheinstädter, 2012

Temperature Control (at HZB)



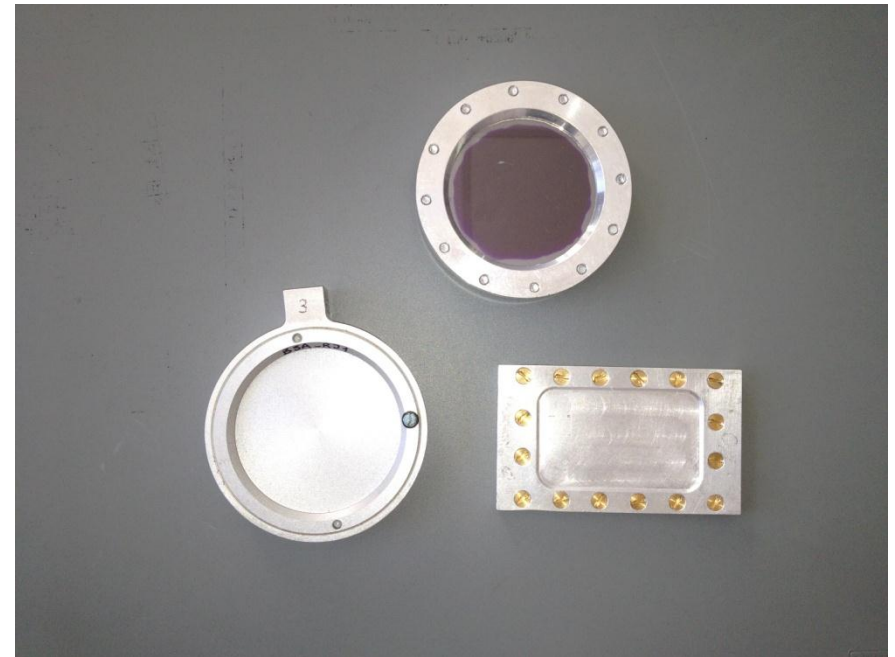
Fixed humidity preparation

- Thomas Hauß -
Purple membrane
samples



Fixed humidity preparation

- ✓ Easy sample changes
- ✓ Large temperature range possible (very low T)
- x Don't know actual humidity
- x Stability of humidity over time?



Bulk water

- Katsaras group

Reflectometry
sample cell

A=temperature controlled
copper

B=water reservoir

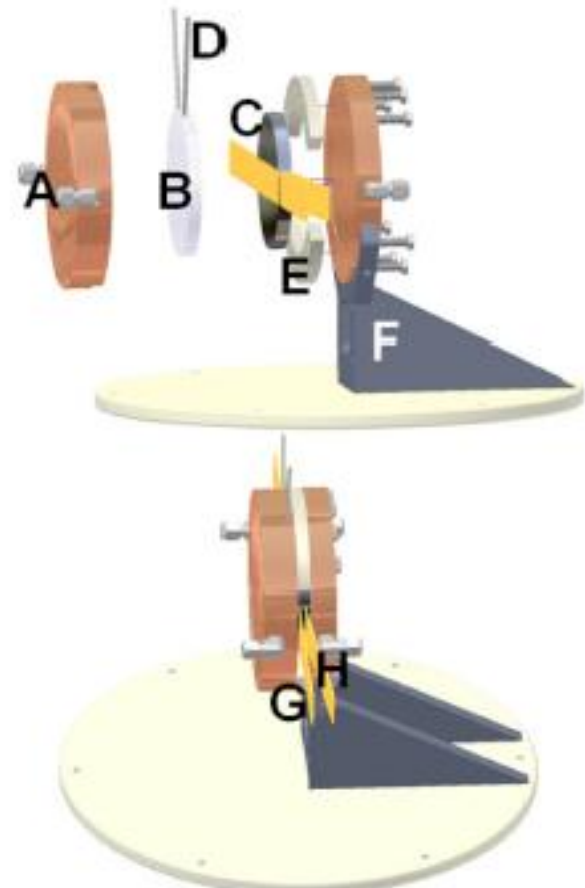
C=silicon sample

D=access ports

E=aluminum sample holder

F=Chamber stand

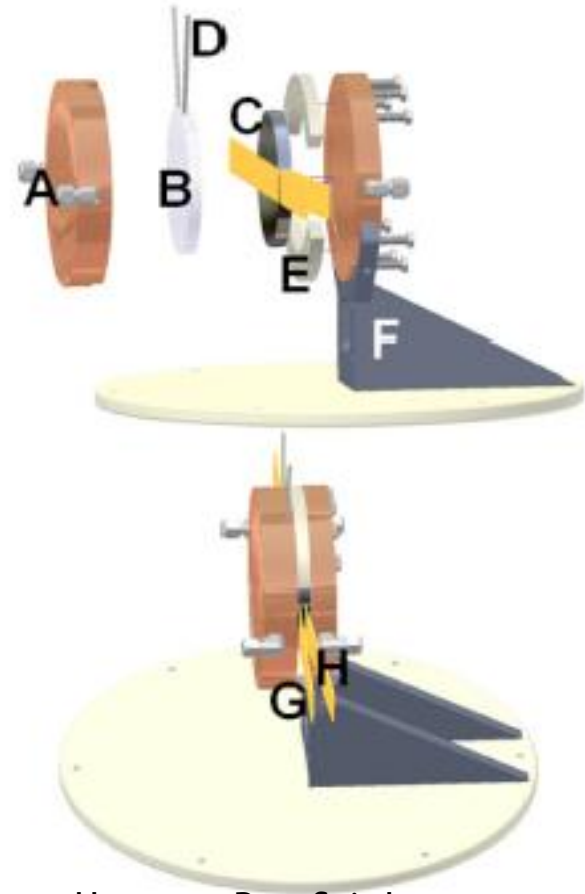
GH=beam path



Harroun, Rev. Sci. Instruments,
2005

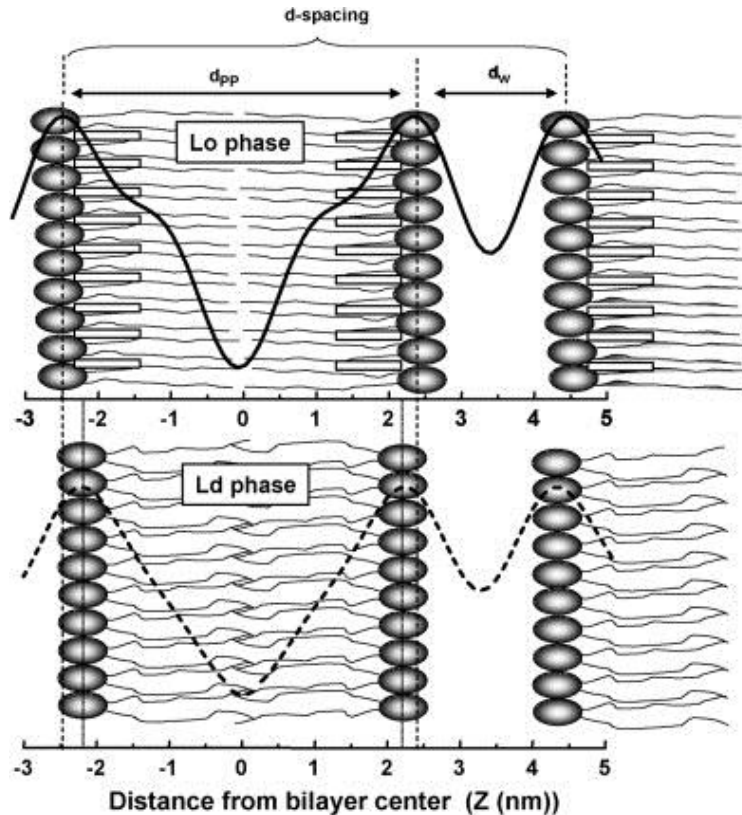
Bulk water

- ✓ 100% hydration
- ✓ Contrast exchange
- ✓ Short equilibration time
- x Sample loss to bulk (esp. charged lipid)
- x Scattering through water

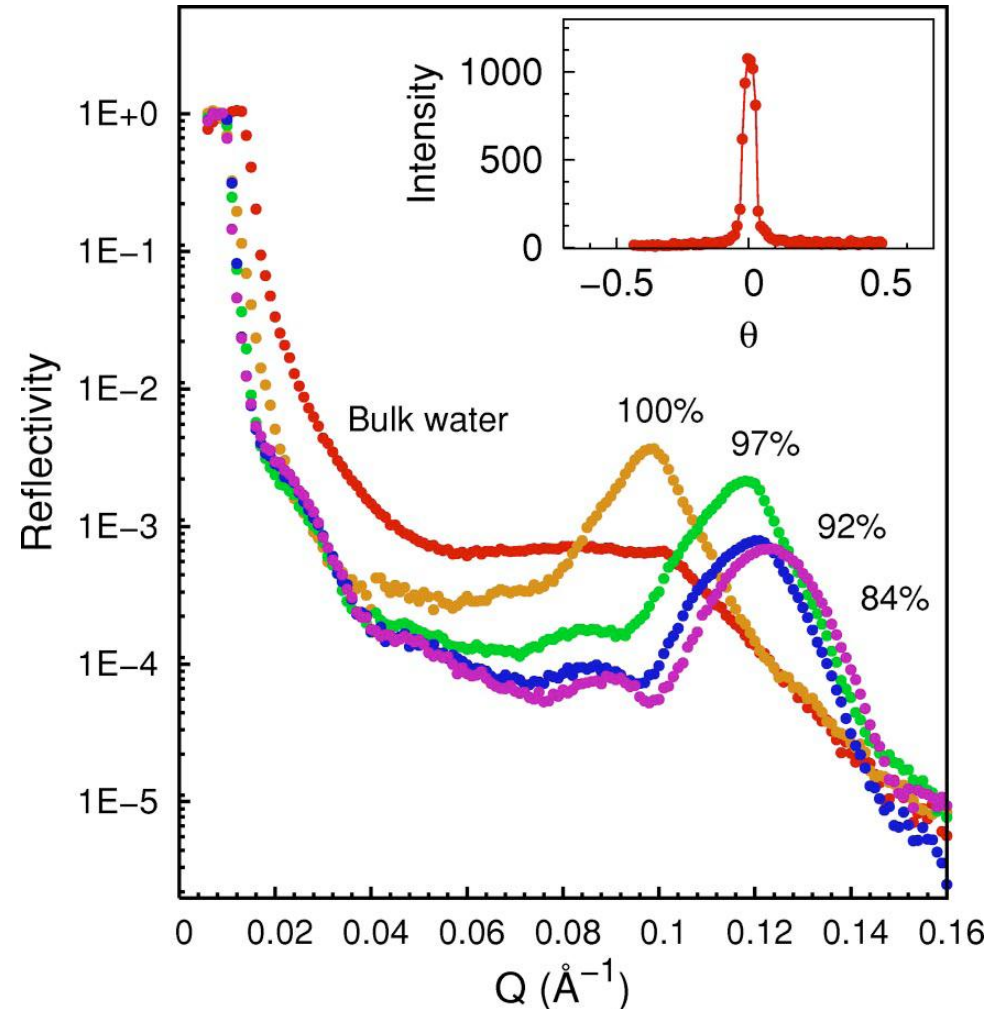


Harroun, Rev. Sci. Instruments, 2005

Toward 100% RH, no condensation

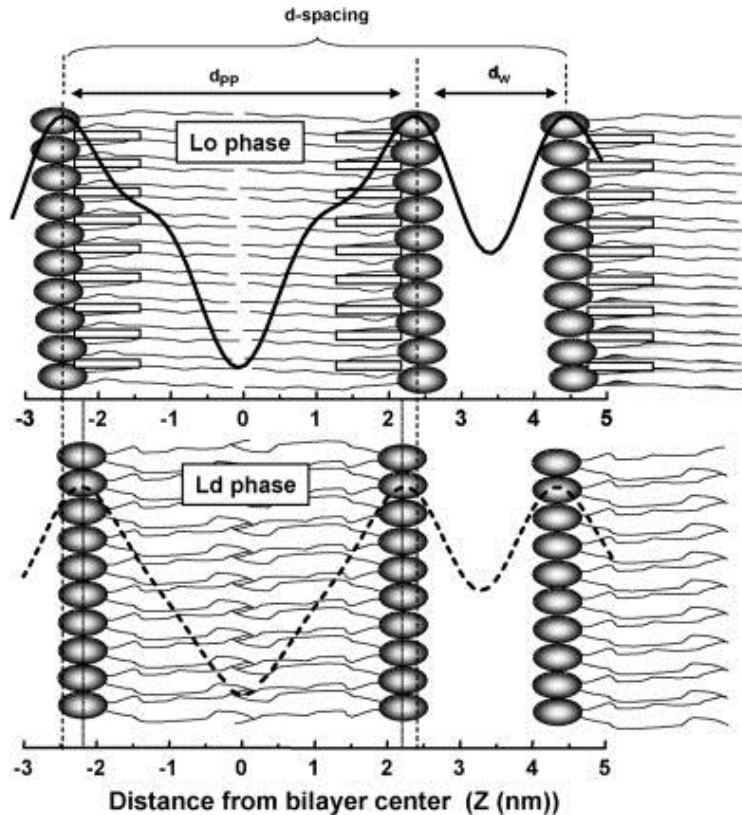


Tessier, J. *Colloid and Interface*, 2008

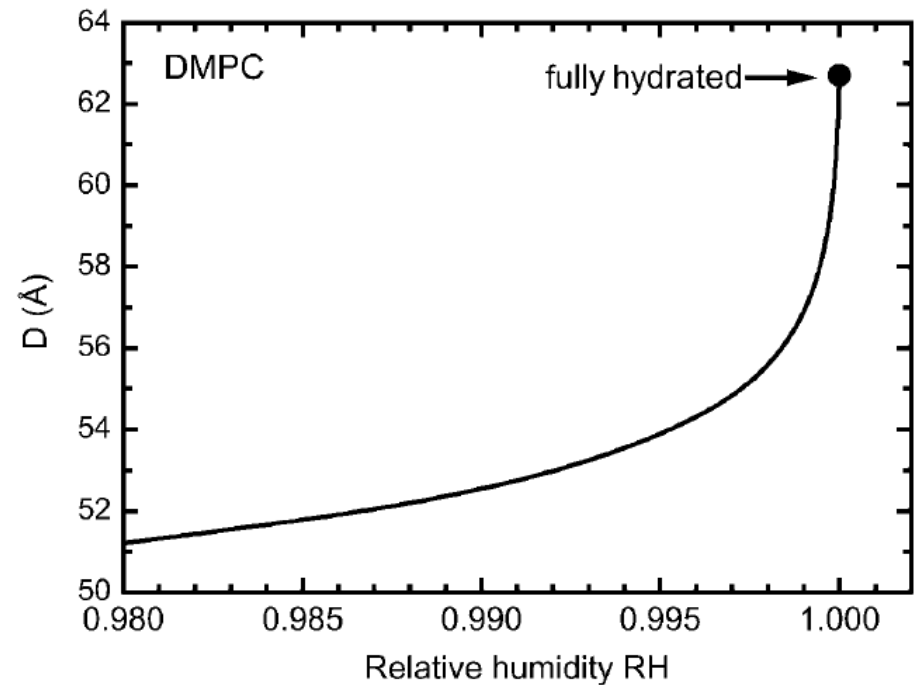


Harroun, *Rev. Sci. Instruments*, 2005

Toward 100% RH, no condensation

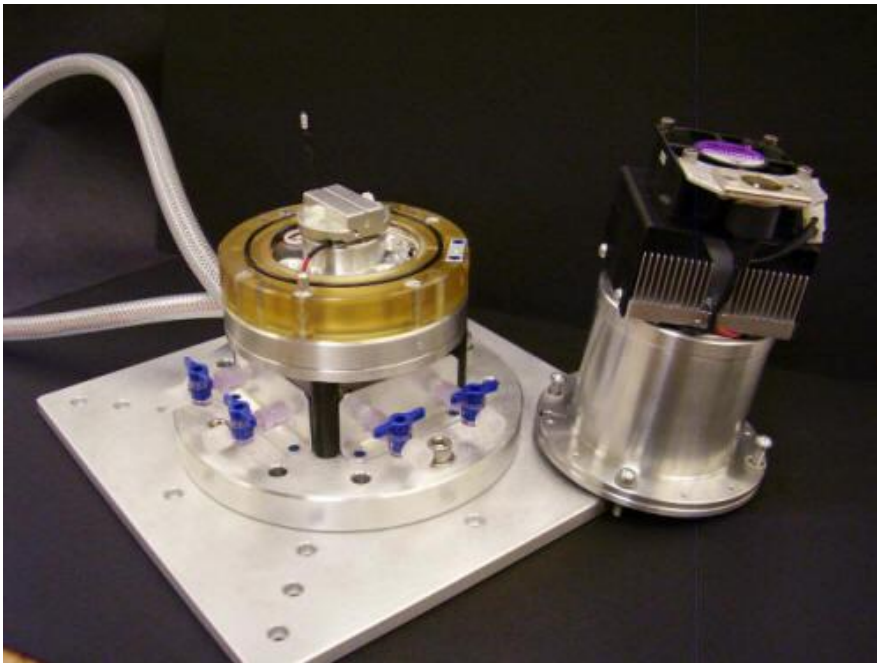


Tessier, J. *Colloid and Interface*, 2008

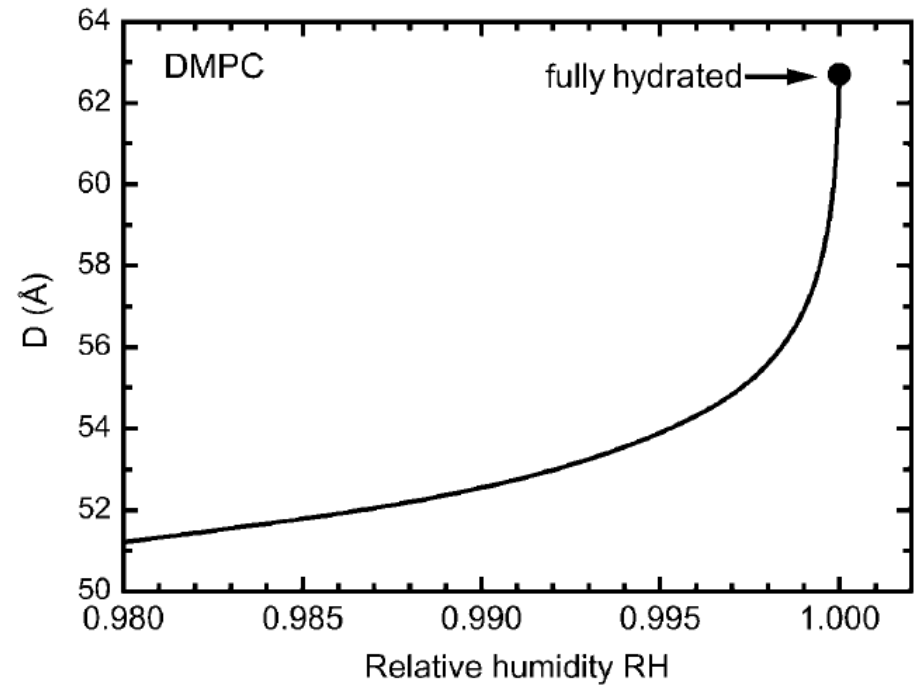


Kučerka, *Biophysical Journal*, 2005

Toward 100% RH, no condensation

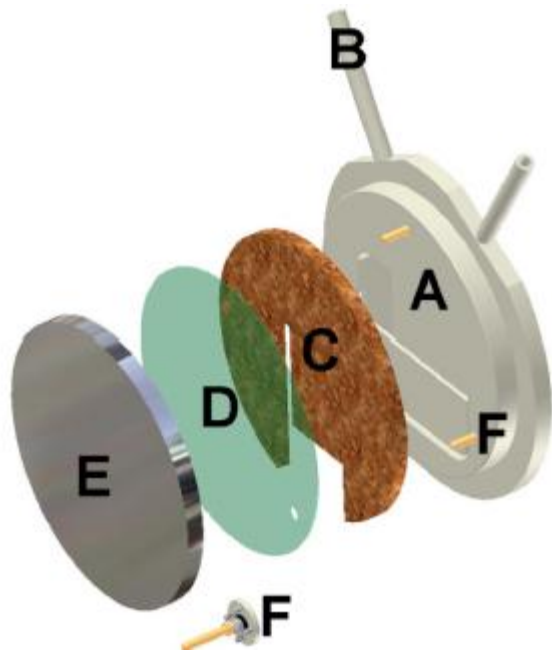


Rheinstädter, 2012



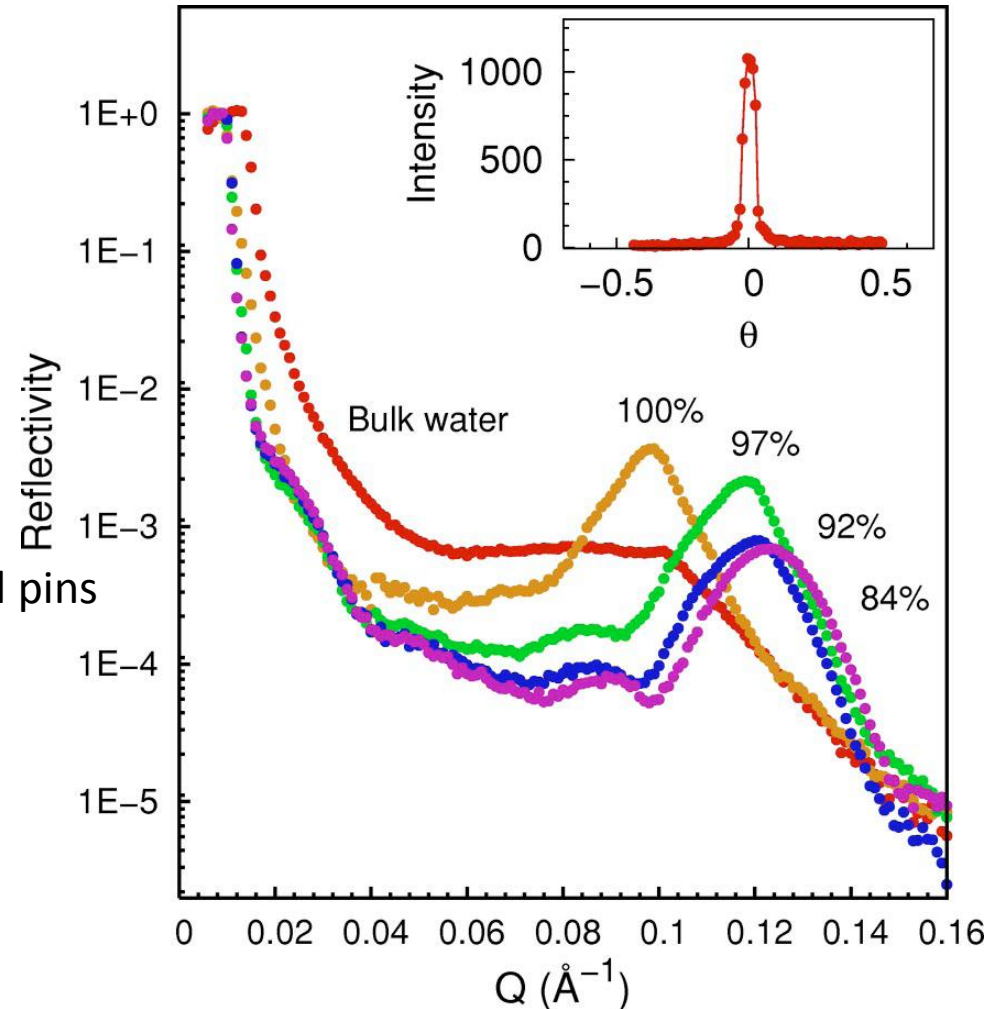
Kučerka, Biophysical Journal, 2005

Toward 100% RH, no condensation



Harroun, Rev. Sci. Instruments, 2005

A=reservoir
B=access ports
C=sponge
D=steel mesh
E=silicon
F=spring loaded pins



Harroun, Rev. Sci. Instruments, 2005

Chamber challenges

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

Summary

	Humidity ceiling (at 25 C)	Humidity Stability	Automation for RH change	Equilibration time (after RH change)	Contrast variation (H ₂ O/D ₂ O)
Gas flow	~95%	~0.1% (or better?)	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%	~0.01% (or better?)	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

Humidity Chamber: Participants

Task Leader



Partners



Observers

