BerILL Humidity Chamber for Neutron Scattering

NMI3-Soft Matter JRA-WP20

Matt Barrett, 2015-05-28/29, LLB

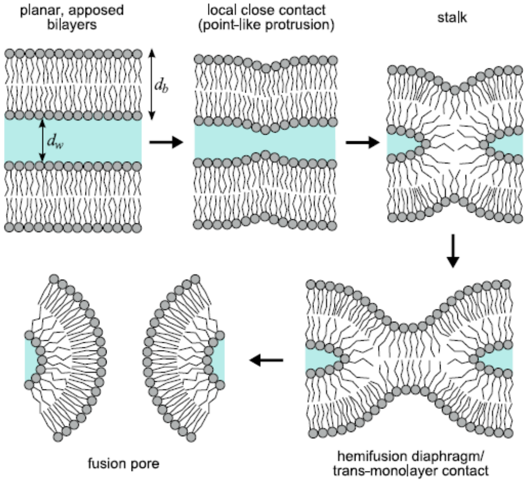
In this talk…

- Motivation
- Project overview and evolution
- First test and results

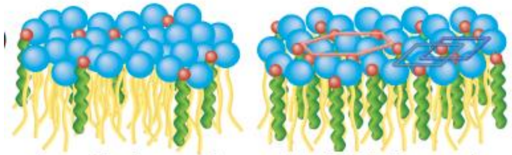
Motivation

Biological investigations combined with neutron scattering in warm and humid environments

- Stalk formation in membranes
  - Tuneable humidity facilitates phase transition from bilayer to stalk, normally protein facilitated
- Cholesterol solubility in DMPC membranes
  - Determine cholesterol solubility limit when approaching physiological conditions at high humidity



Pathway of liquid layer formation (Aeffner, 2009).

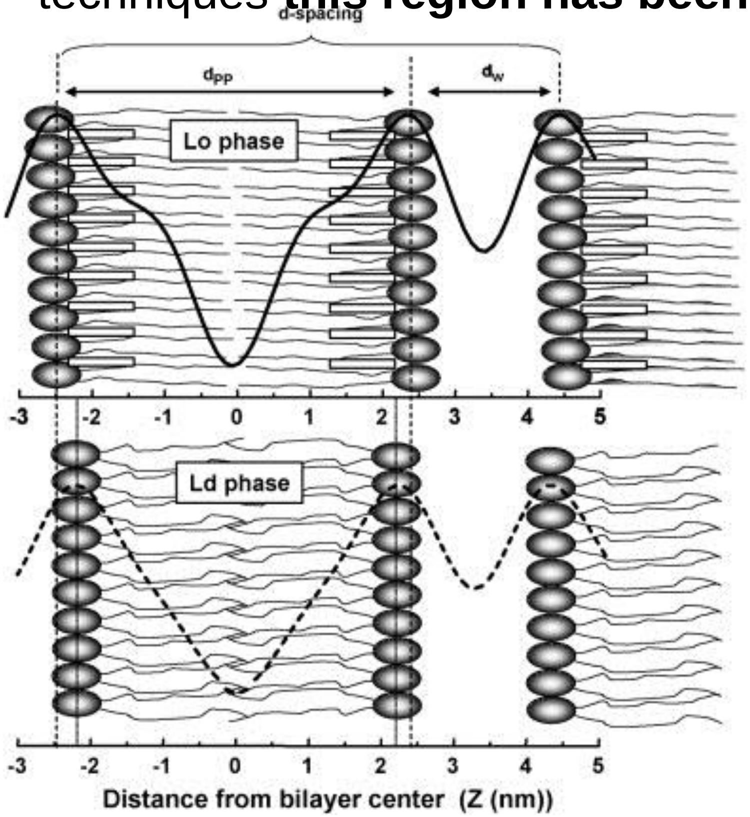


Low Cholesterol (disordered)

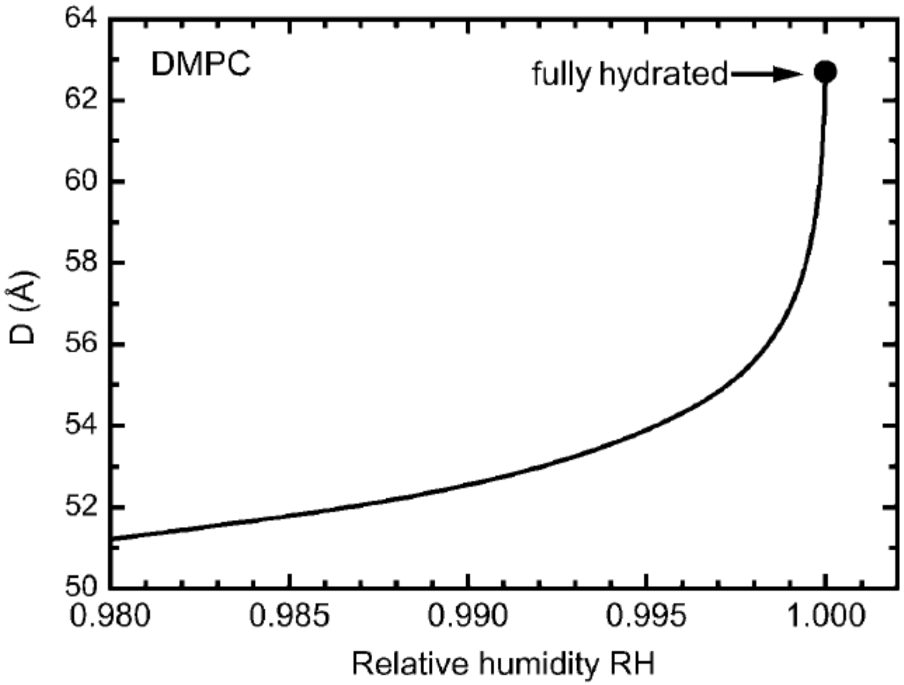
High Cholesterol (ordered)

Toward 100% RH

The dramatic dependence of d-spacing of lipid bilayer on humidity close to saturation makes high r.h. region extremely interesting, but with todays humidity control techniques **this region has been largely inaccessible!**



Tessier, J. Colloid and Interface, 2008



Kučerka, Biophysical Journal, 2005

Project goal

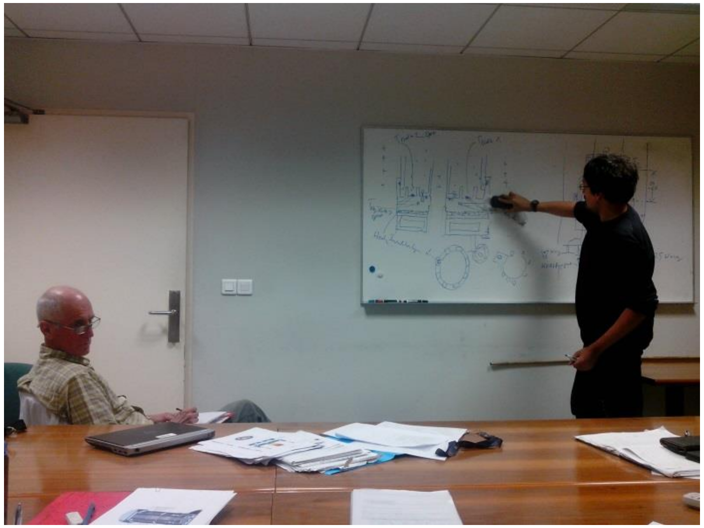
Develop a new humidity chamber which has:

- the ability to access large T and RH range especially above 95% r.H.
- faster and better controlled temperature and humidity response than existing cells (proposal suggested goal of 10 mK stability in T and 0.1% in r.H.)
- adaptability to different neutron instrument geometries
- large sample space with option for multi-sample holder

Timeline

Year 1:

Review the existing systems determine the specifications of the next-generation chambers



Year 2:

Produce drawings

Year 3:

Build and commission chamber

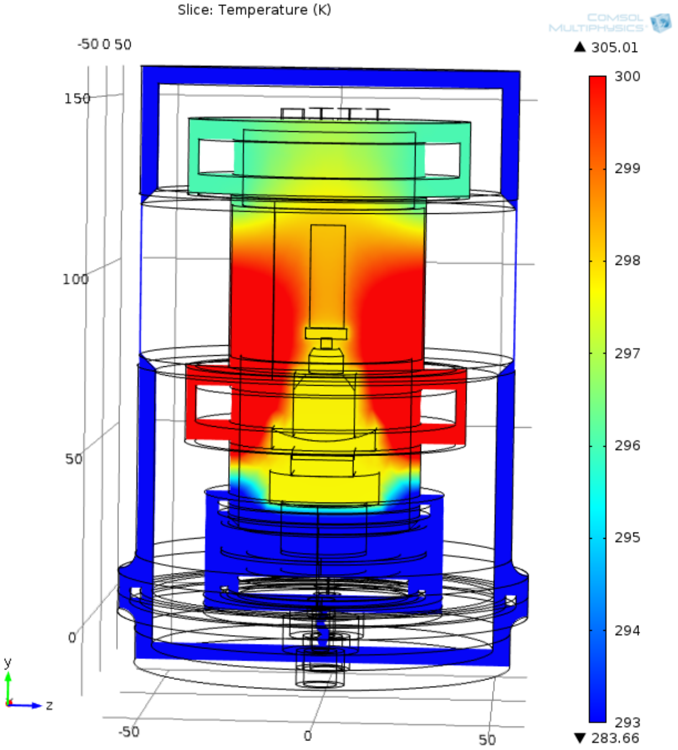
Relative Humidity Control

$$\mathrm{r.}h. = \frac{partial\ vapour\ pressure}{saturation\ vapour\ pressure}$$

$$\log_{10} P = 5.402 - \frac{1838.7}{T(K) - 31.7}$$

Bridgeman and Aldrich, 1964

r.h. (%) sample at 298 K	P_{needed} (mbar)	$T_{water\ bath}$ (K)
100	31.42	298
98	30.8	297.7
90	28.3	296.3
75	23.6	293.2
40	12.6	283.5



3D render of the new chamber

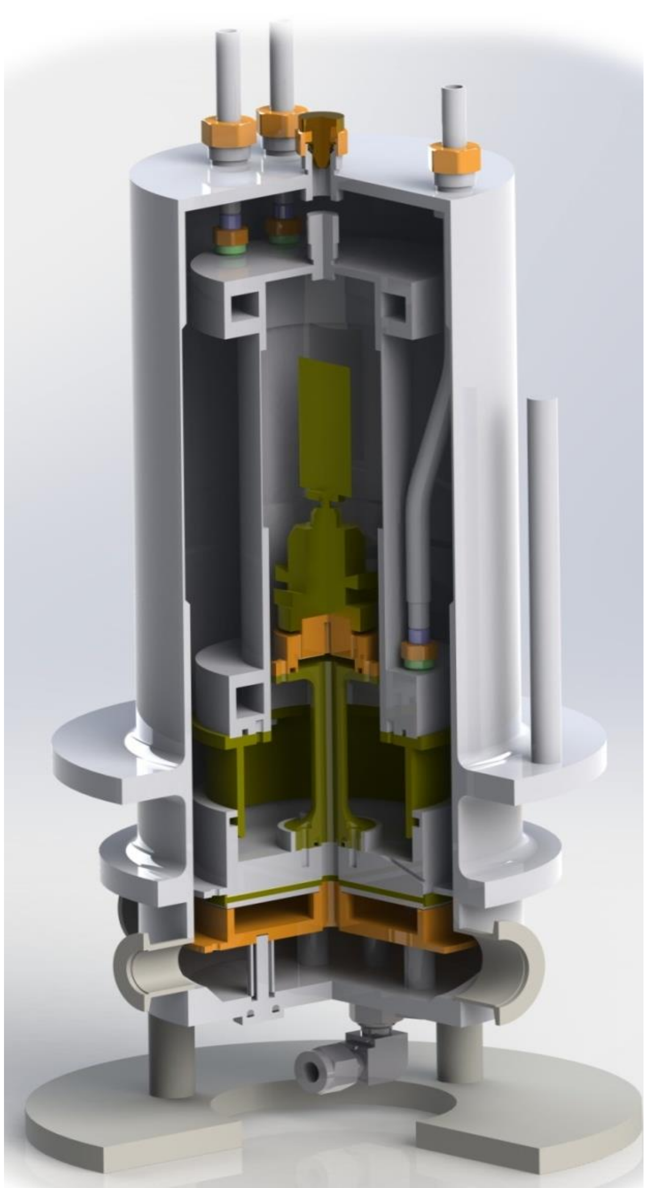
Total height 400 mm, diameter 150 mm

Wide angular scanning range about 300°

Three water channels connect to warm and cold water bath chillers allowing for temperature regulation at the sample and water reservoir

Hot upper and cold lower parts of the inner chamber thermally isolated

Insulating posts connect inner and outer chambers while maintaining thermal isolation from outer environment



Double walled evacuated Aluminum construction

Inner cell has small volume for quick equilibration

Simple sample change remove entire upper cell using guide posts

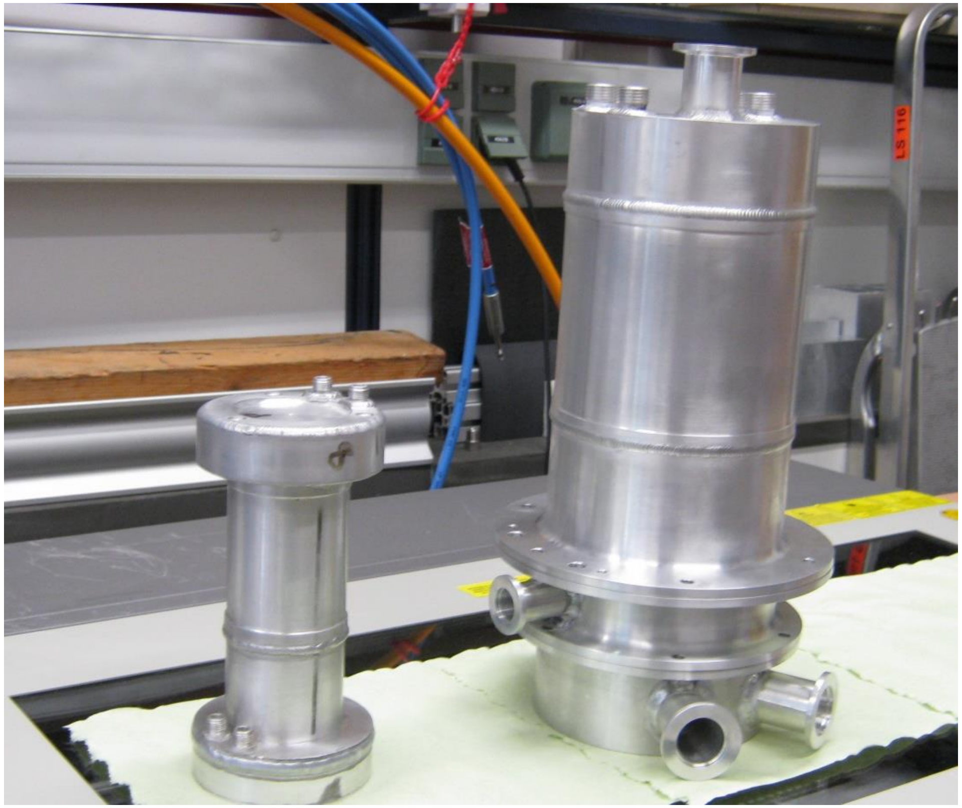
Resistive heating foils which heat against the constant water chillers allow for extremely accurate and stable temperature regulation

Simple modifications of modular chamber would allow a variety of scattering geometries by sapphire windows for SANS horizontaly sample stage for reflectometry

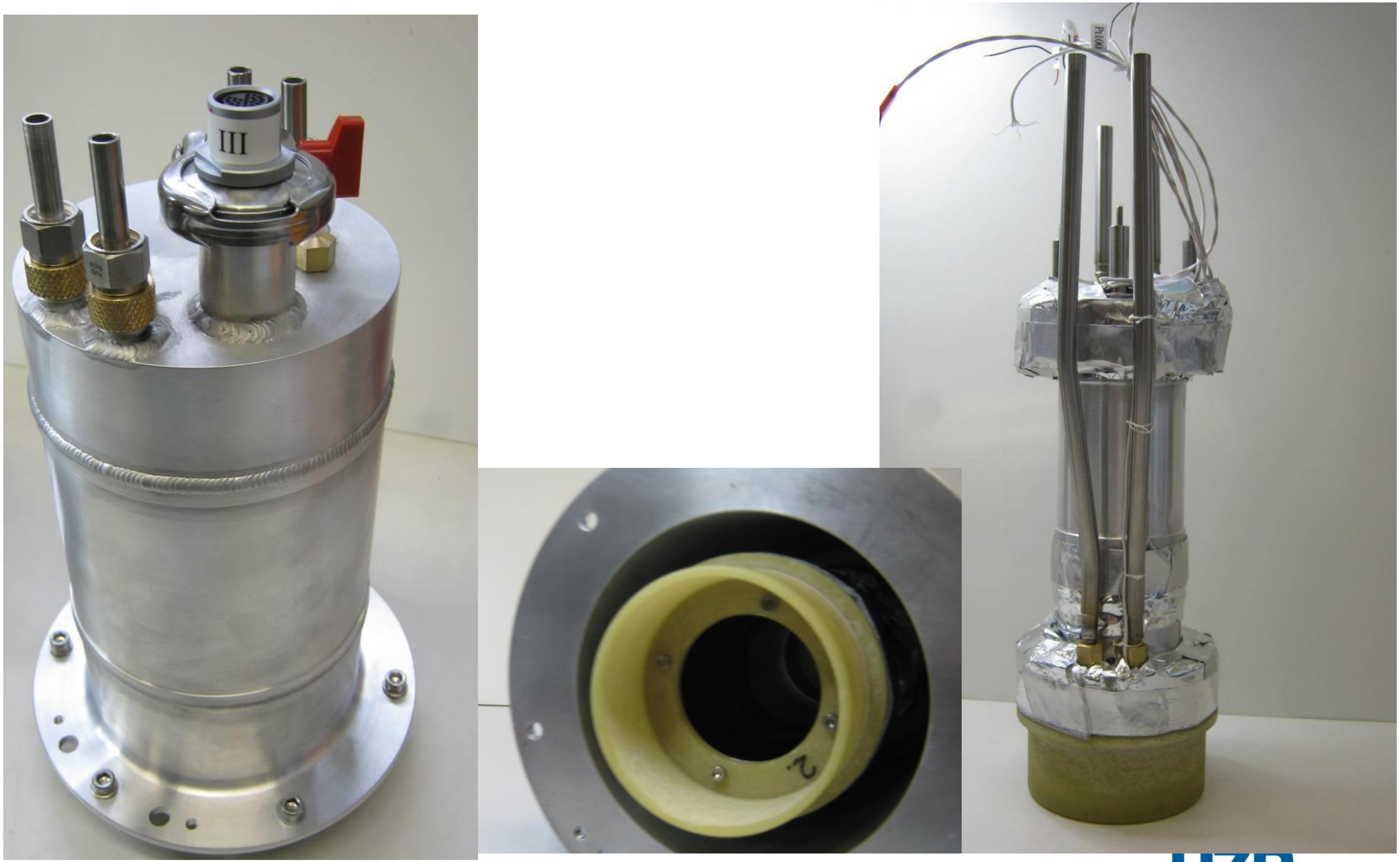
HZB Helmholtz Zentrum Berlin

Assembly of prototype I

ILL parts + HZB parts



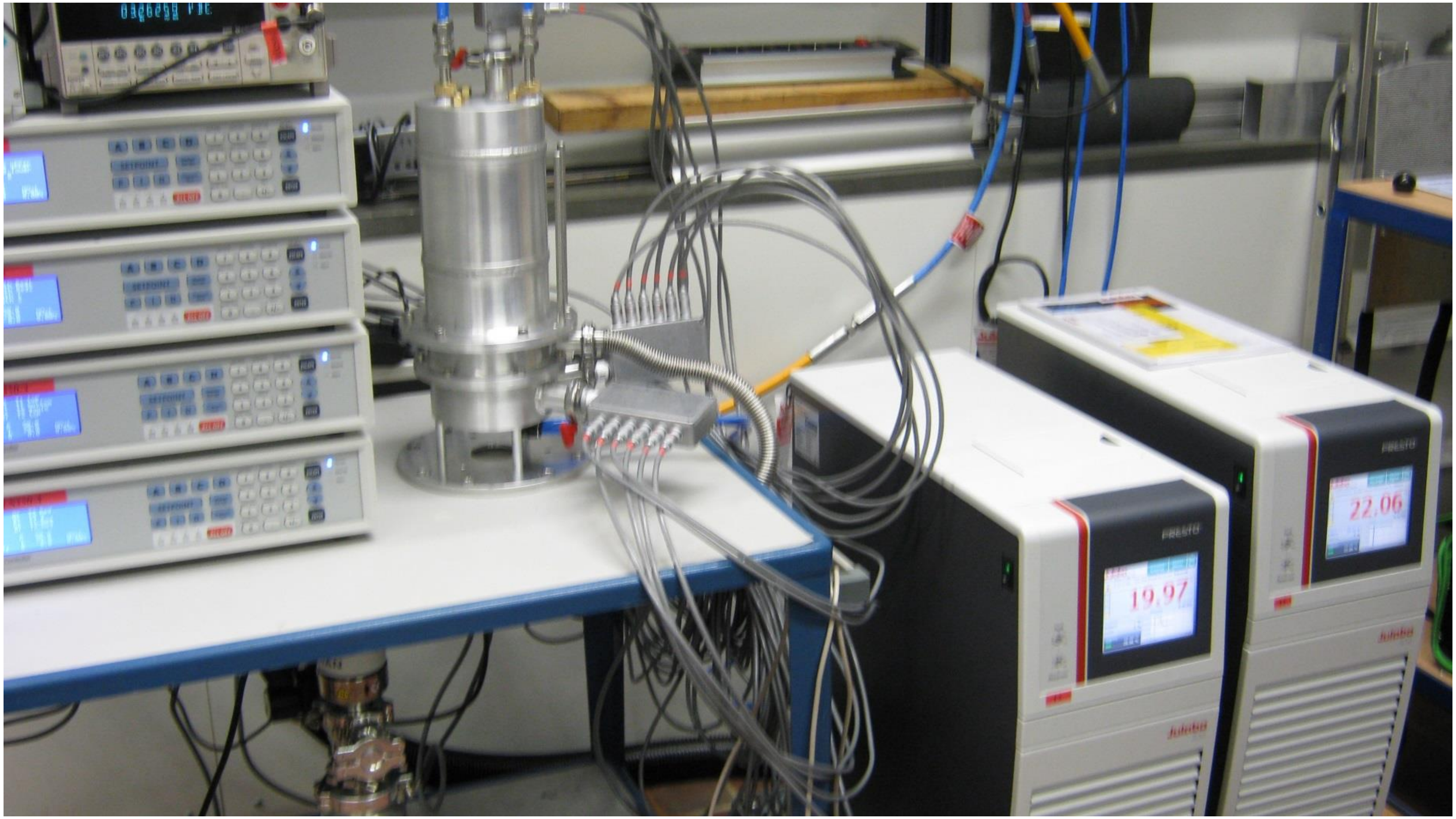
Assembly: top parts



Assembly: bottom parts

Final Assembly

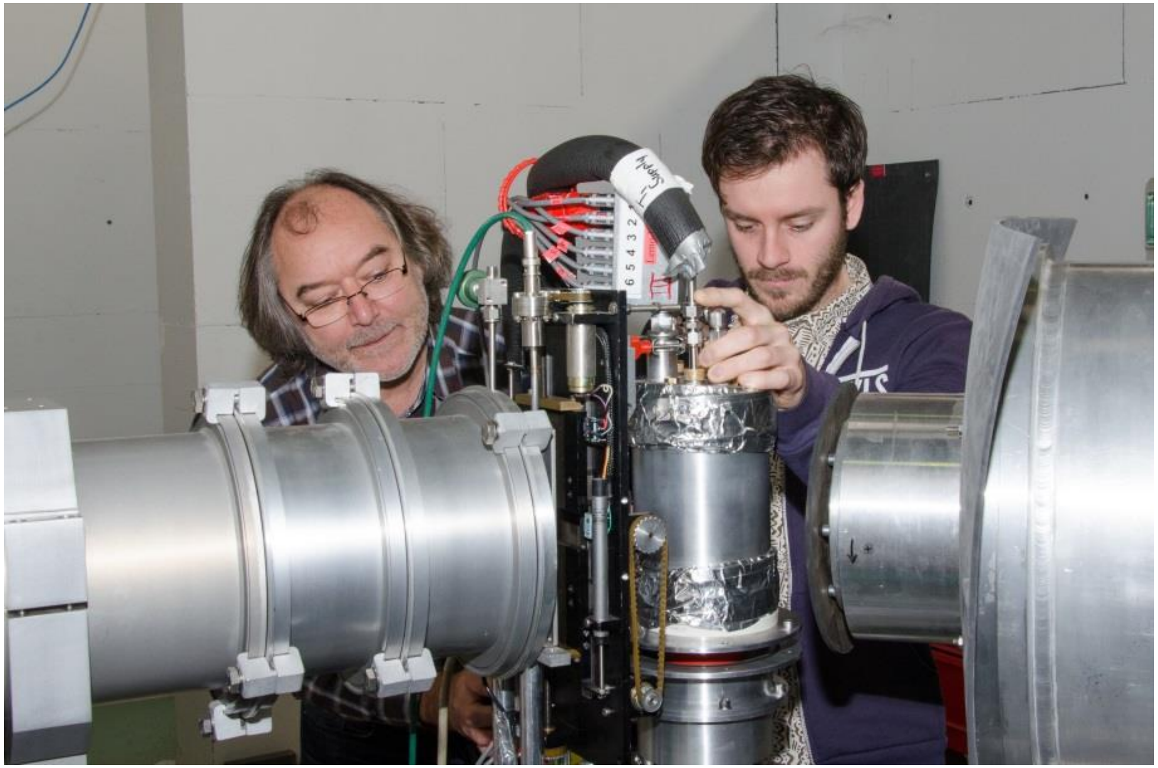
Complete Setup

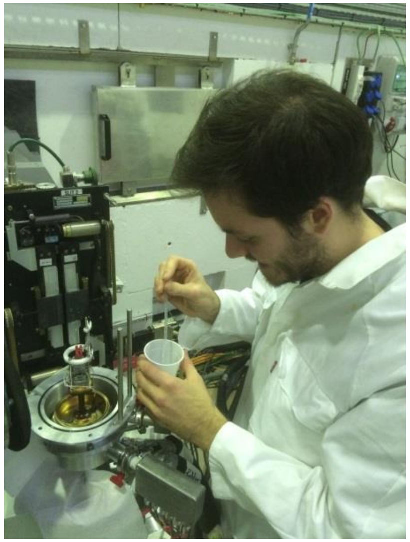


D16: December 2014

D16: December 2014

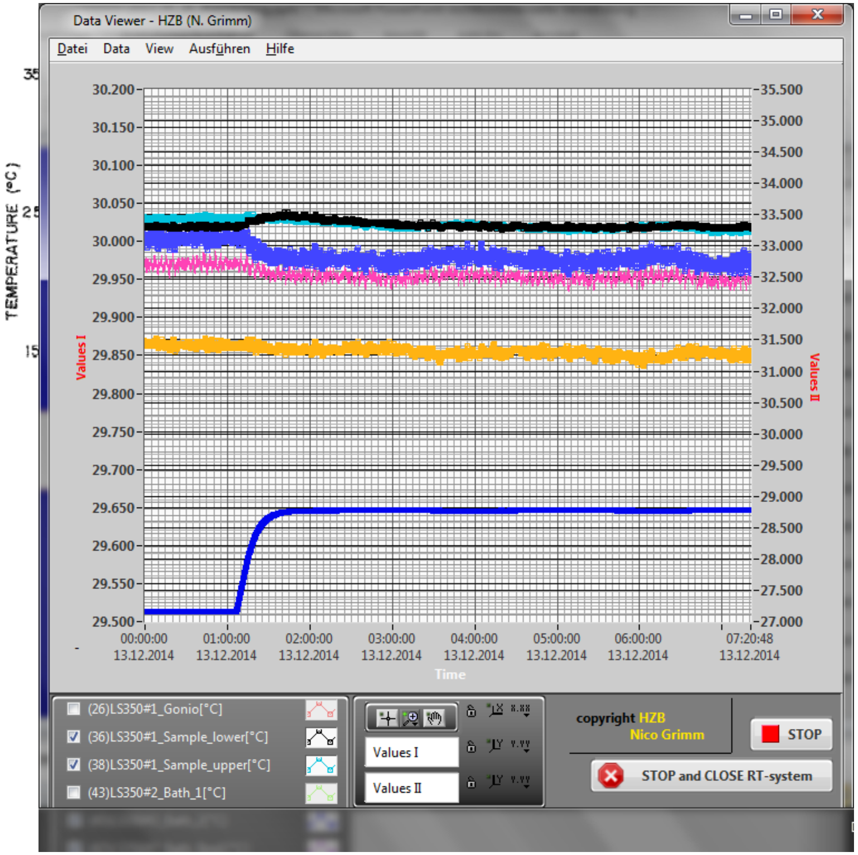
- Slow cooling oil leaks inside of chamber
- Good vacuum not achieved (No turbo pump)
- Heating bands around outer chamber added (garage door)





D16: December 2014

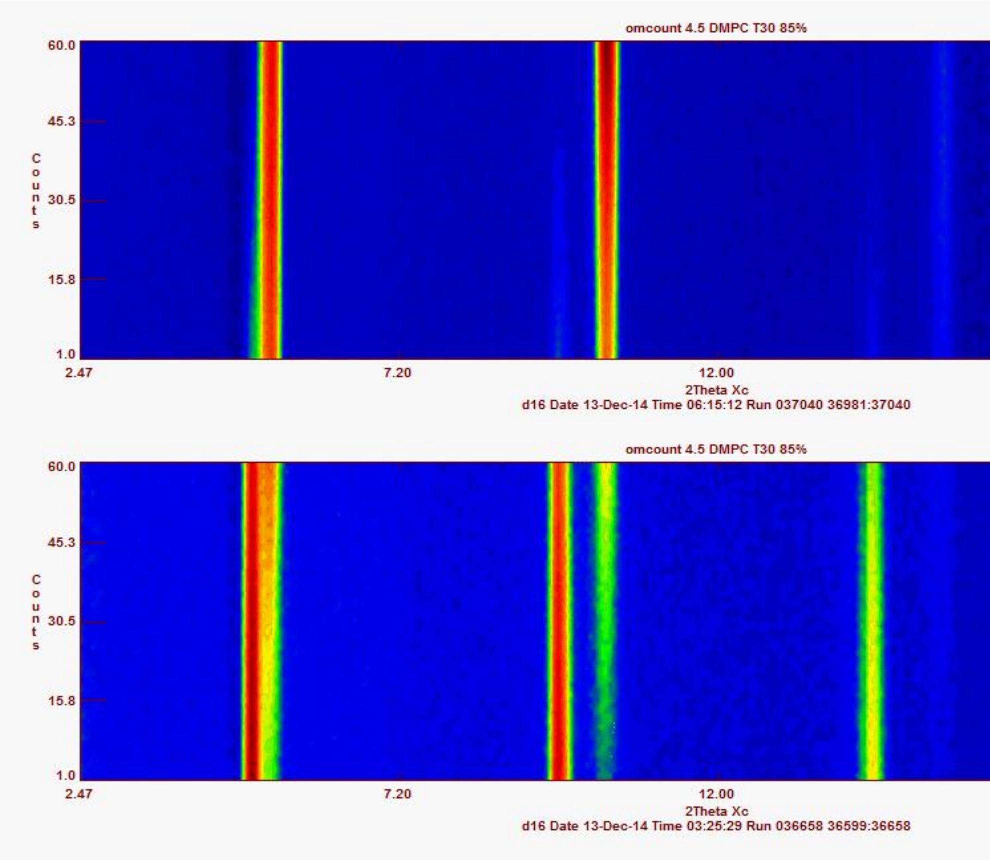
- DMPC main phase transition (gel to fluid) at 30°C ~85% r.h.



Data Viewer - HZB (N. Grimm)

☐ (26)LS350#1_Gonio[°C]
☑ (36)LS350#1_Sample_lower[°C]
☑ (38)LS350#1_Sample_upper[°C]
☐ (43)LS350#2_Bath_1[°C]

copyright HZB Nico Grimm



omcount 4.5 DMPC T30 85%

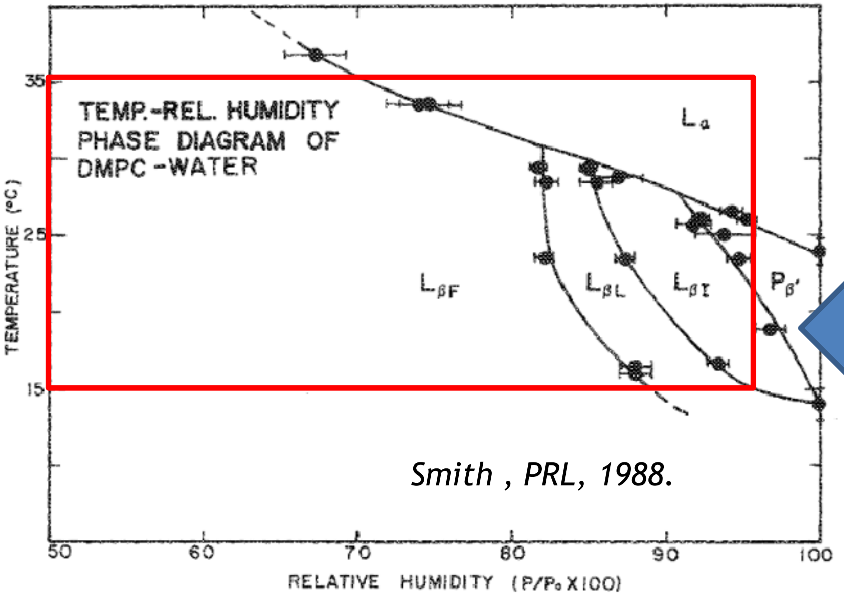
d16 Date 13-Dec-14 Time 06:15:12 Run 037040 36981:37040

omcount 4.5 DMPC T30 85%

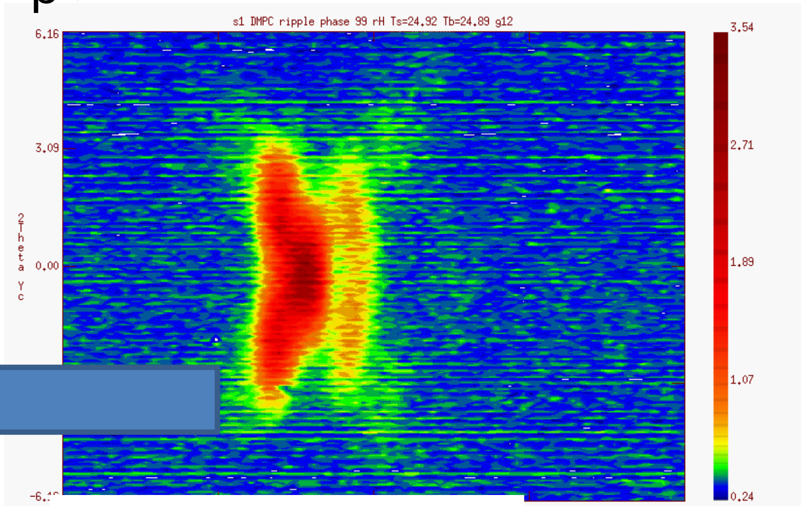
d16 Date 13-Dec-14 Time 03:25:29 Run 036658 36599:36658

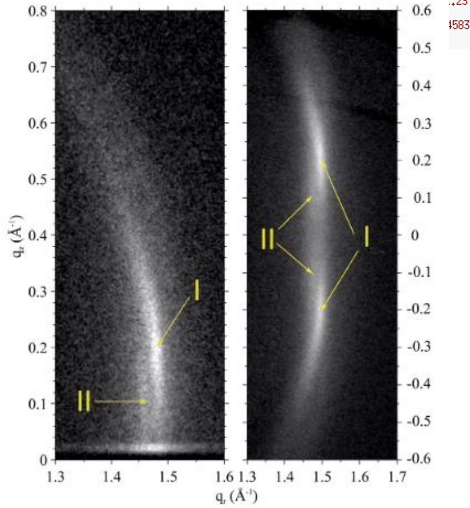
D16: December 2014

- DMPC ripple phase ($P_{\beta'}$)



Smith , PRL, 1988.

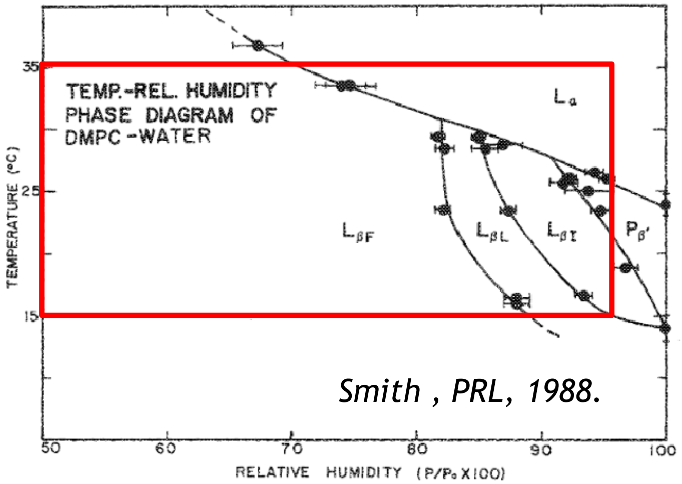




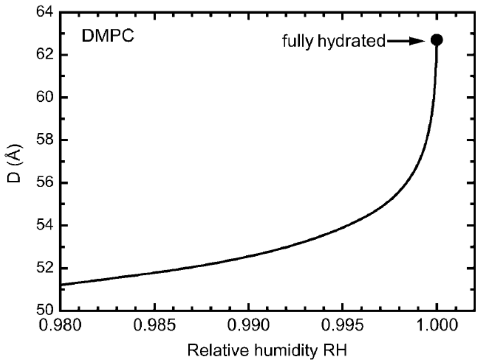
Akabori, Soft Matter, 2015.

D16: December 2014

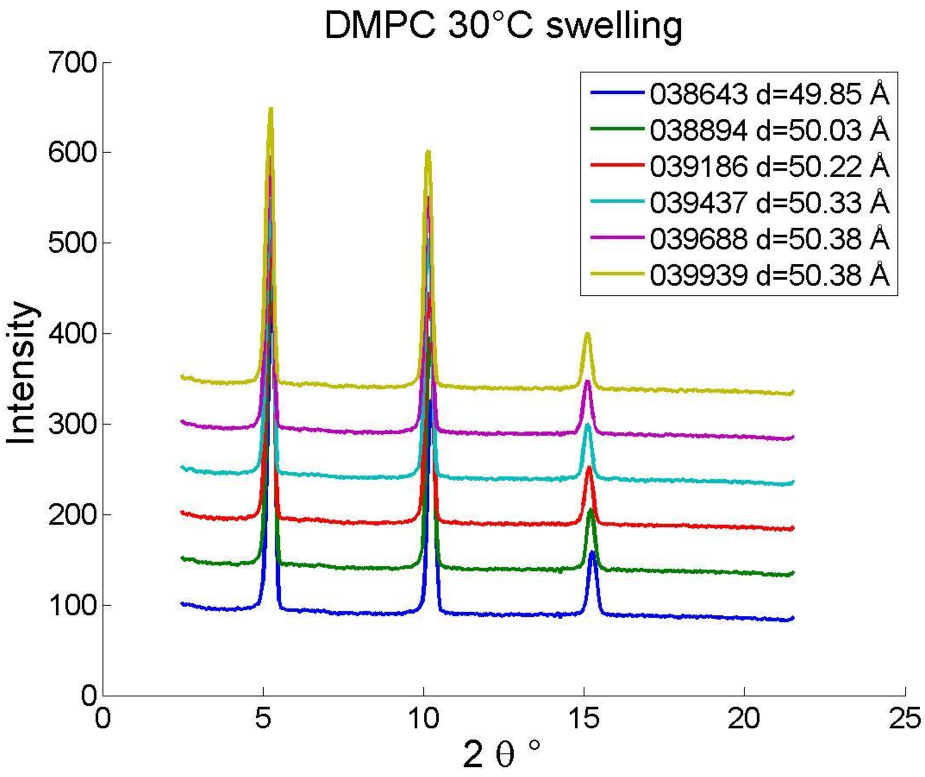
- DMPC fluid phase swelling (50.38 Å ~ 97% r.h.)



Smith , PRL, 1988.



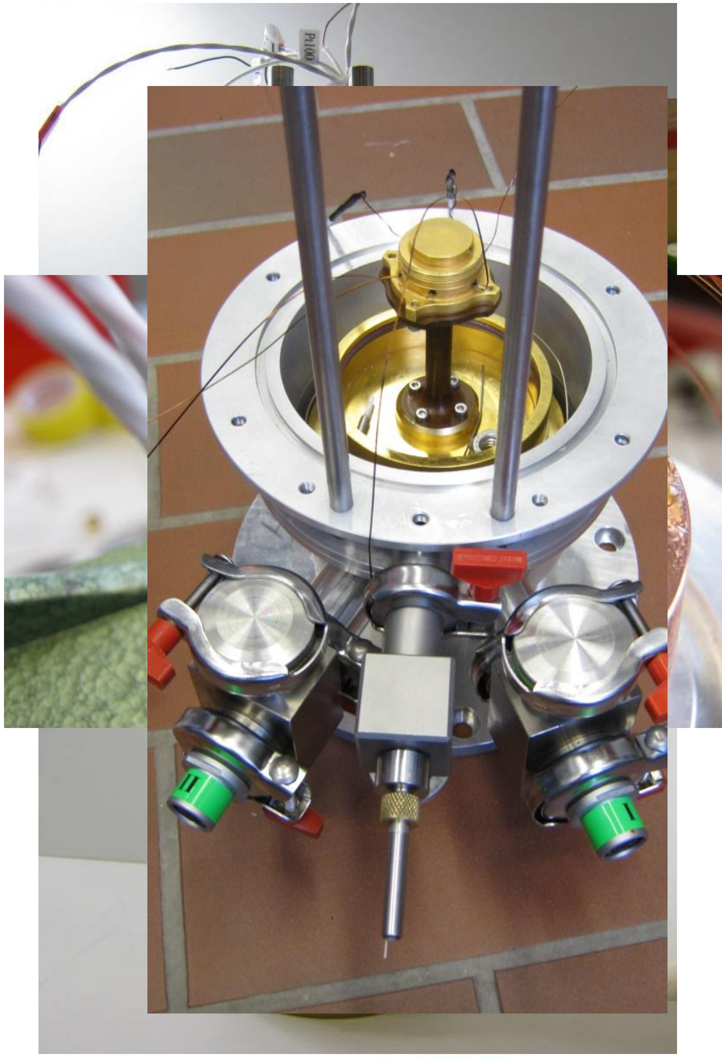
Kučerka, Biophysical Journal, 2005



Winter modifications

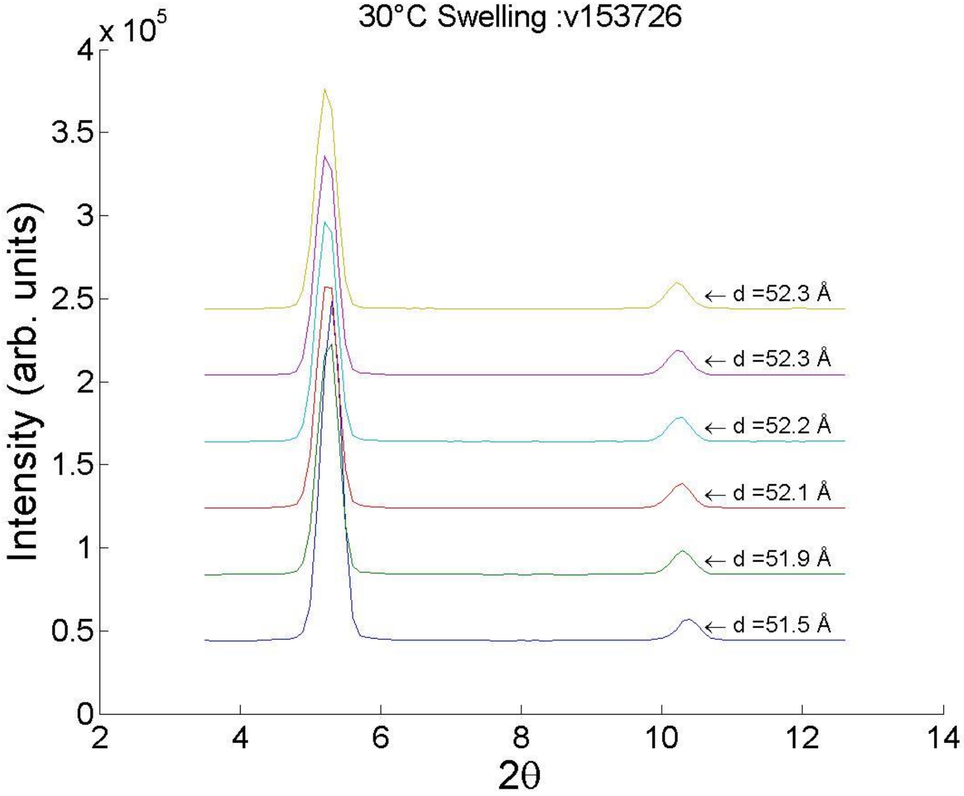
- Flexible pipes for chiller connectors to inner chamber
- New o-rings, stopped oil leaks
- Redesign of insulating ring/o-ring connection
- Thermometer recalibration
- Better access to wiring, easier to reassemble

Major problems from December solved!

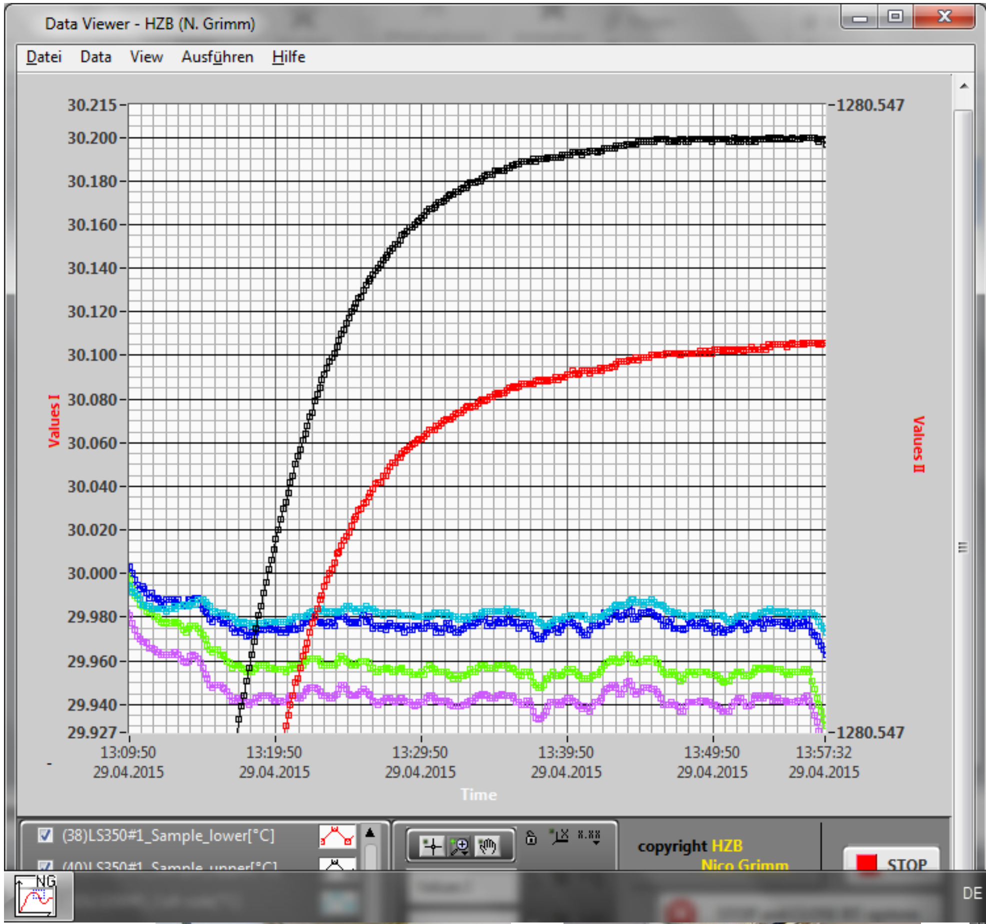


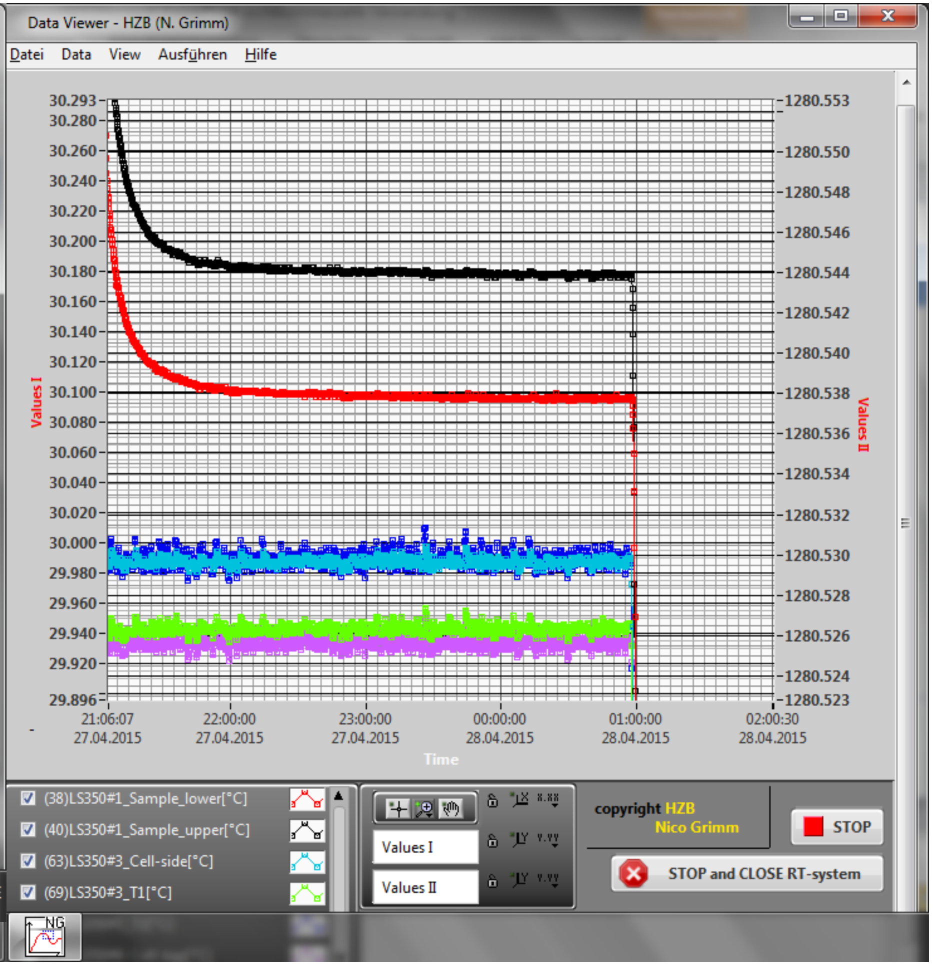
V1: April 2015

- DMPC fluid phase (30.5 °C) swelling (52.3 Å ~ 98.5% r.h.)
- Full saturation not possible



V1: April 2015

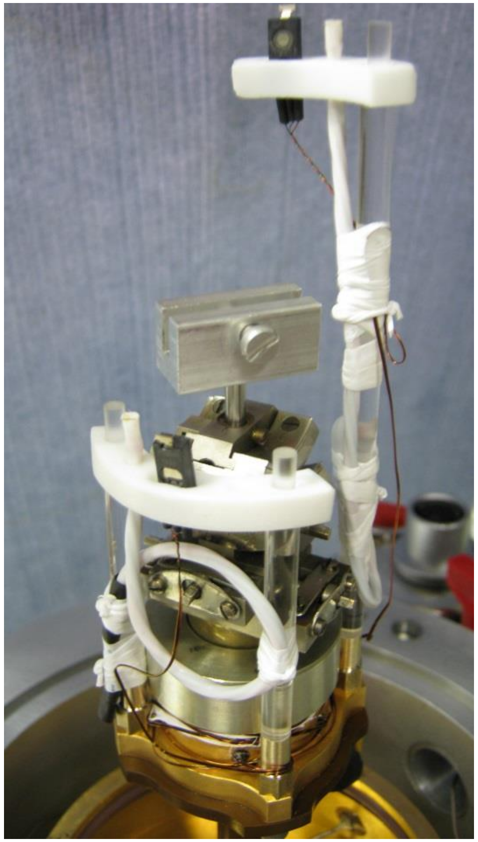




V1: April 2015

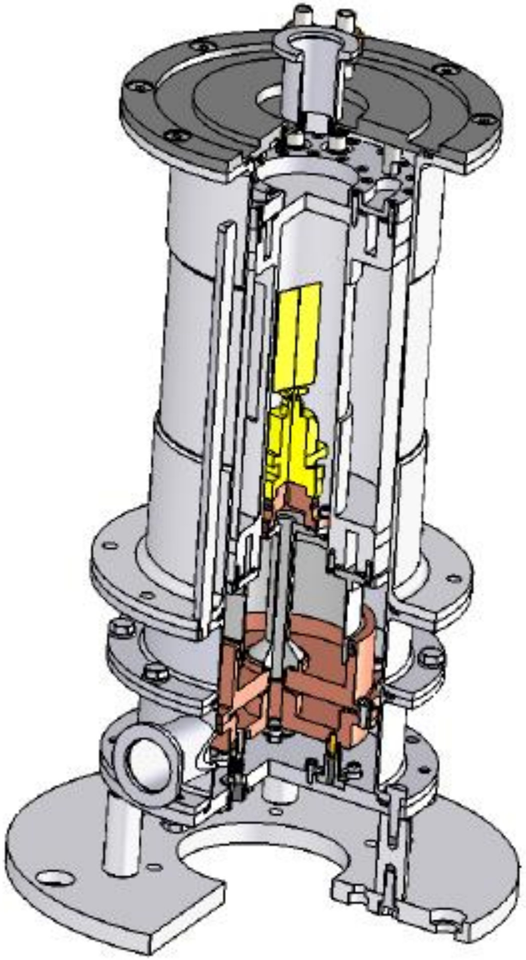
- Sample lower and sample upper consistently 100 and 200 mK warmer than rest of chamber?

- Unplugged voltage source, immediate drop in sample temperature (~100 mK), (although still not perfect, more on this later)



BerILL 2.0

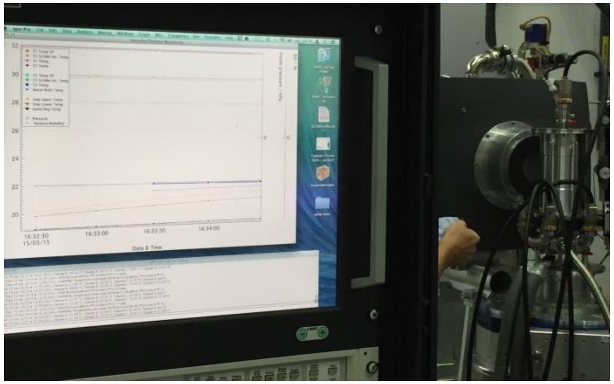
- Design/drawings by Julien Gonthier and Eric Bourgeat-Lami (ILL)
- Modular design for easier assembly
- Improved vacuum/o-ring sealing (order of magniude better than 1.0 version)



BerILL 2.0

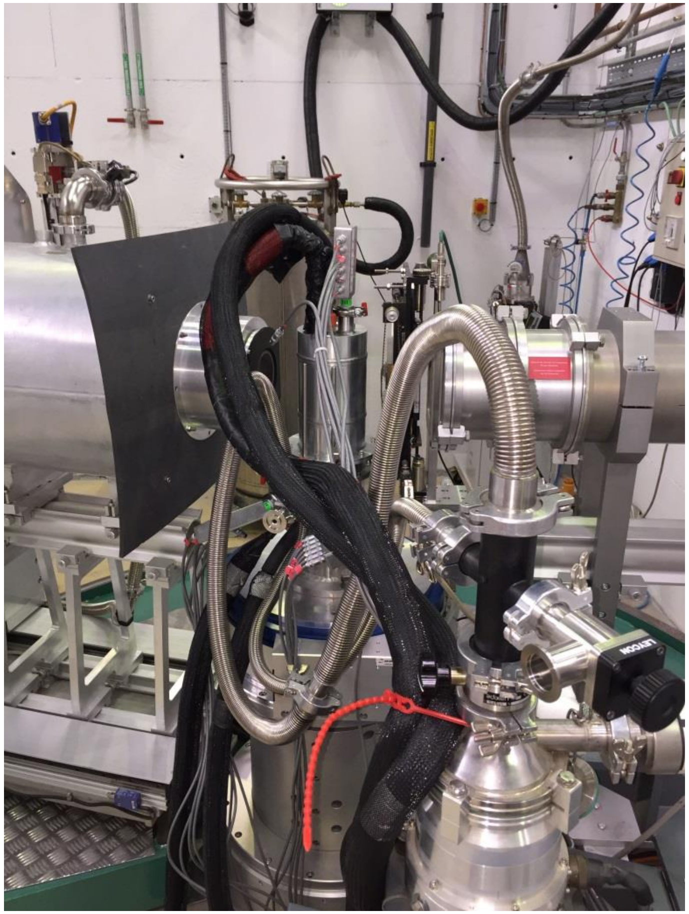
- Inner chamber simplified with single chiller channel

- User friendly Igor interface (set desired sample temperature + humidity)



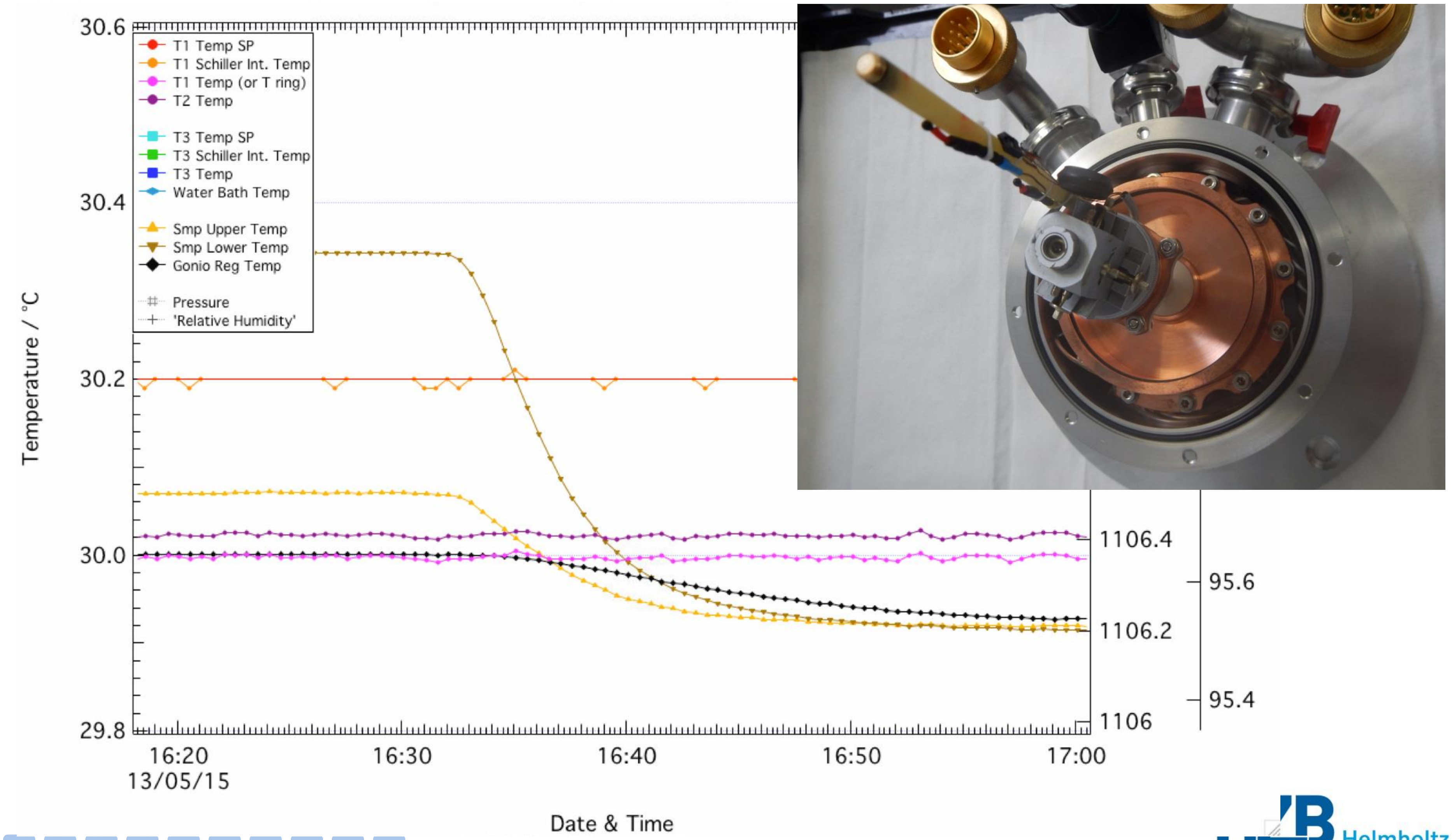
D16: May 2015

BerILL 1.0



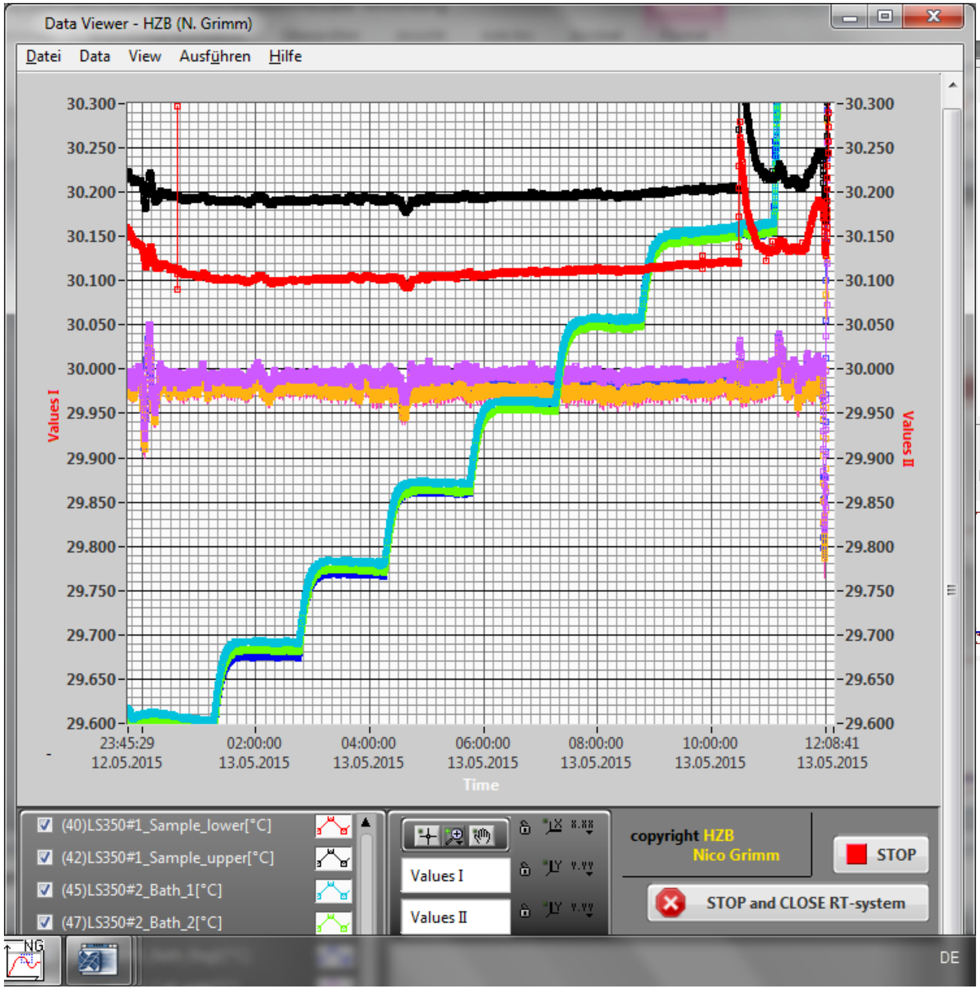
BerILL 2.0

D16: May 2015

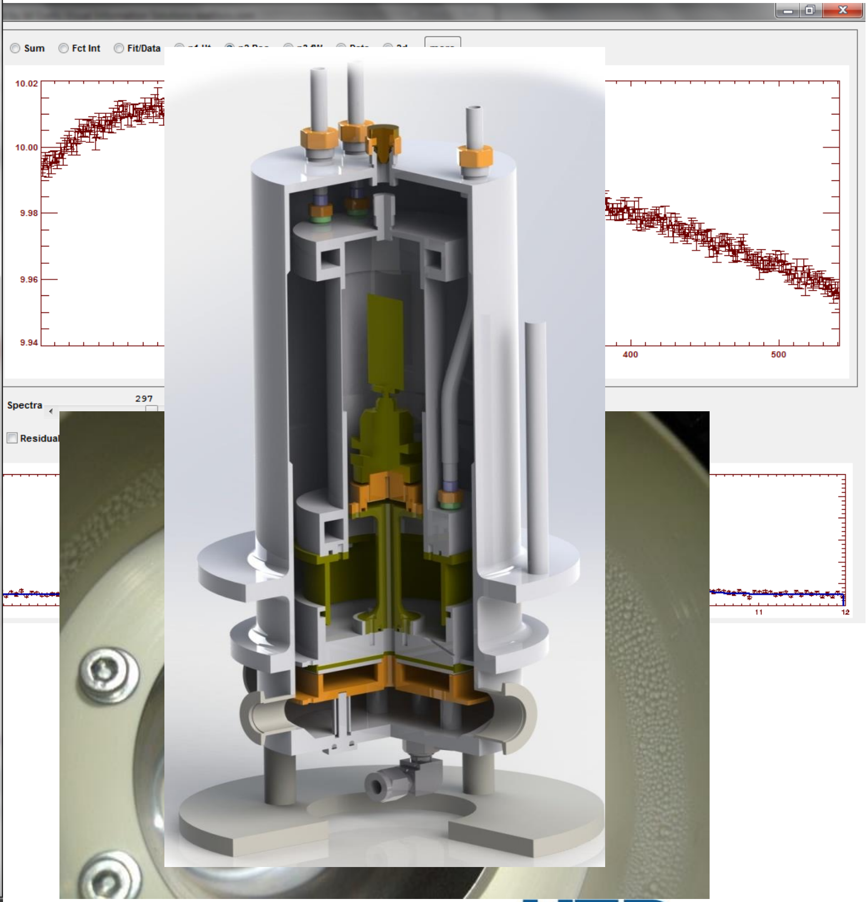

T1 Temp SP
T1 Schiller Int. Temp
T1 Temp (or T ring)
T2 Temp
T3 Temp SP
T3 Schiller Int. Temp
T3 Temp
Water Bath Temp
Smp Upper Temp
Smp Lower Temp
Gonio Reg Temp
Pressure
'Relative Humidity'
Temperature / °C
30.6
30.4
30.2
30.0
29.8
1106.4
1106.2
1106
95.6
95.4
16:20
13/05/15
16:30
16:40
16:50
17:00
Date & Time

D16: May 2015

- DMPC Humidity run at 30°C: condensation, 51 Å max. (~98% r.h.)



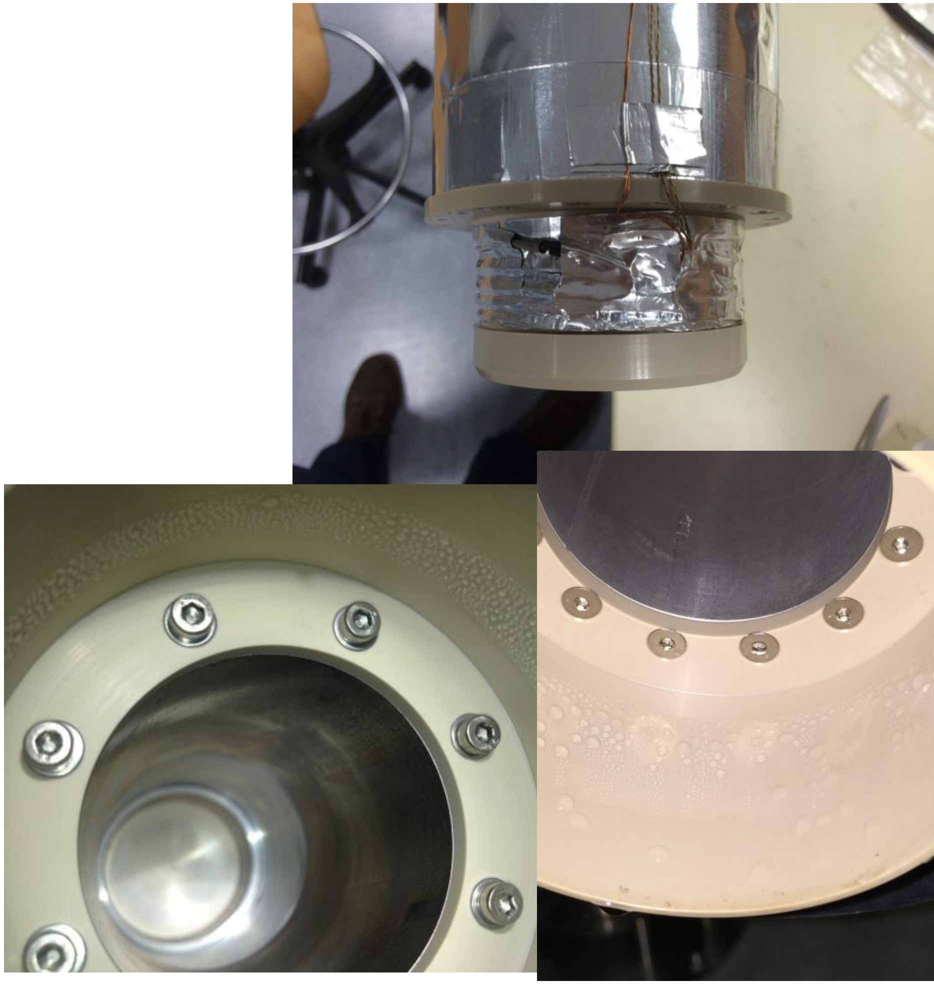
☑ (40)LS350#1_Sample_lower[°C]
☑ (42)LS350#1_Sample_upper[°C]
☑ (45)LS350#2_Bath_1[°C]
☑ (47)LS350#2_Bath_2[°C]



D16: May 2015

- Goal: dramatic (and stable) DMPC swelling at 30°C
  - Not seen with BerILL 1.0 or 2.0?

– No swelling when slowly approaching 100% r.h. from below with pure water
  - Condensation seen on insulating ring (even with good vacuum of BerILL 2.0)

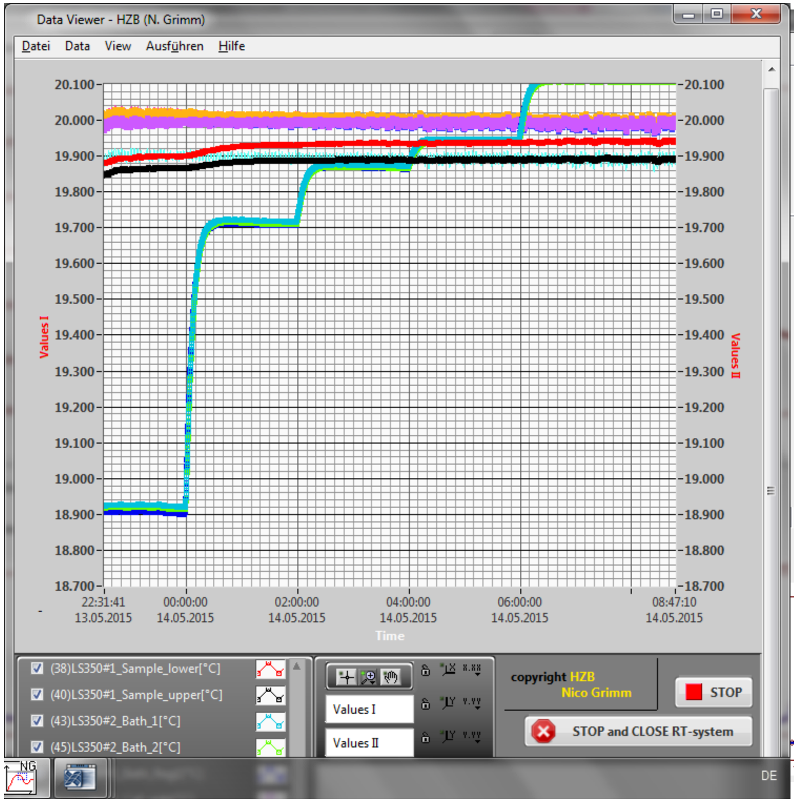


D16: May 2015

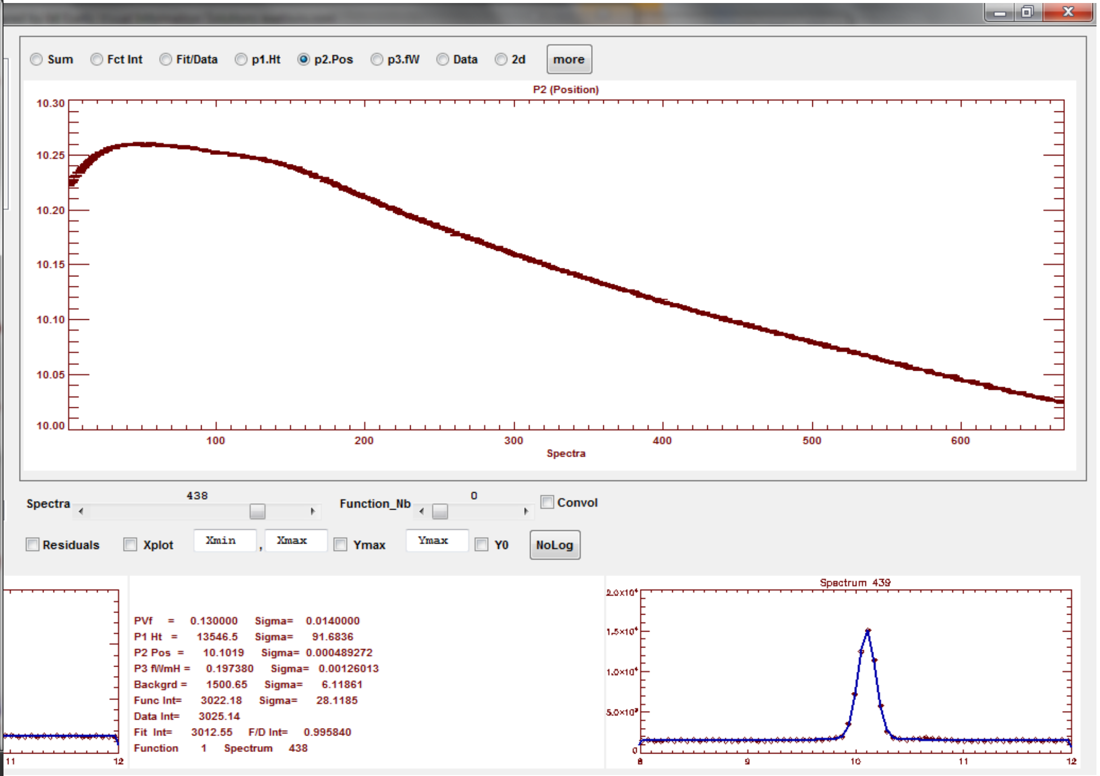
- Used DOPC sample (transition temperature below room temp)
- Switched to unsaturated salt solution (1 mol% K_2SO_4 relaxes T parameters), still no dramatic swelling
- Same result for both old and new chamber

D16: May 2015

- DOPC Humidity run at 20°C: no condensation, r.h. ~98.5%



☑ (38)LS350#1_Sample_lower[°C]
☑ (40)LS350#1_Sample_upper[°C]
☑ (43)LS350#2_Bath_1[°C]
☑ (45)LS350#2_Bath_2[°C]



Spectra 438

Function_Nb 0

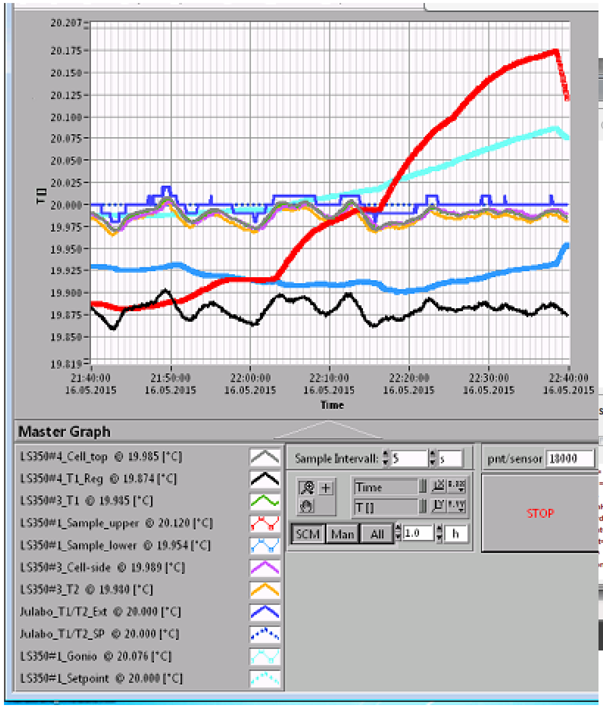
```
PVf     =   0.130000    Sigma=   0.0140000
P1 Ht   =    13546.5    Sigma=     91.6836
P2 Pos  =    10.1019    Sigma= 0.000489272
P3 fWmH =   0.197380    Sigma=  0.00126013
Backgrd =    1500.65    Sigma=     6.11861
Func Int=    3022.18    Sigma=     28.1185
Data Int=    3025.14
Fit  Int=    3012.55   F/D Int=   0.995840
Function      1   Spectrum    438
```

D16: May 2015

- Hypotheses:
  1. Cold point in chamber causes condensation not on sample, acts as water reservior
  2. Massive goniometer is slow to reach temperature, many moving surfaces create pores which must first fill
  3. No convection causes stagnant vapour density gradient between upper and lower sections of chamber

D16: May 2015

1. Cold point in chamber causes condensation not on sample, acts as water reservior
   - Force cold onto sample with Peltier
   - Rethink insulating ring and post



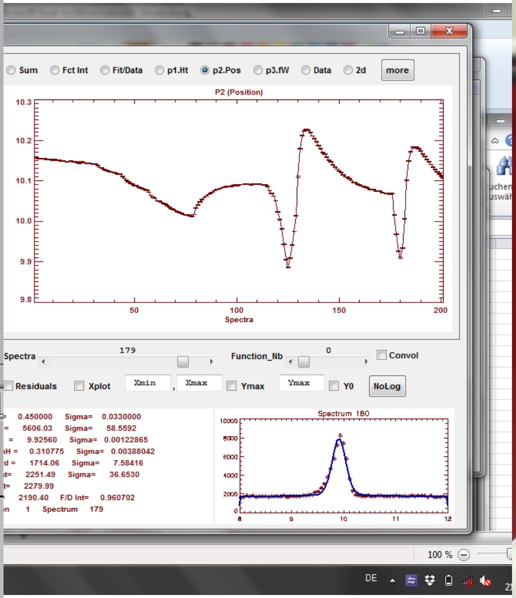
Master Graph

LS350#4_Cell_top @ 19.985 [°C]
LS350#4_T1_Reg @ 19.874 [°C]
LS350#3_T1 @ 19.985 [°C]
LS350#1_Sample_upper @ 20.120 [°C]
LS350#1_Sample_lower @ 19.954 [°C]
LS350#3_Cell-side @ 19.989 [°C]
LS350#3_T2 @ 19.980 [°C]
Julabo_T1/T2_Ext @ 20.000 [°C]
Julabo_T1/T2_SP @ 20.000 [°C]
LS350#1_Gonio @ 20.076 [°C]
LS350#1_Setpoint @ 20.000 [°C]

Sample Intervall: 5 s

pnt/sensor 18000

1.0 h



Spectra 179

Function_Nb 0

☐ Convol

☐ Residuals ☐ Xplot Xmin , Xmax ☐ Ymax Ymax ☐ Y0 NoLog

```
=    0.450000    Sigma=   0.0330000
=     5606.03    Sigma=     58.5592
 =    9.92560    Sigma=  0.00122865
nH =   0.310775    Sigma=  0.00388042
d =    1714.06    Sigma=     7.58416
t=    2251.49    Sigma=     36.6530
t=    2279.99
     2190.40    F/D Int=    0.960702
n      1    Spectrum    179
```

D16: May 2015

2. Massive goniometer is slow to reach temperature, many moving surfaces create pores which must first fill
    - Replace gonio with thin aluminum sample holder, connect with walls to encourage

- Reactor down, experiment finished…

D16: May 2015

3. No convection causes stagnant vapour density gradient between upper and lower sections of chamber

  - Install fan or stirrer to encourage convection

Summary

- Accurate and simple temperature and humidity control up to 98% r.h. (soon to be in friendly user service at ILL and HZB)

- Strategies to reach highest hydration levels are necessary (Aluminum sample mount with built in Peltier?, fan or stirrer?)

Acknowledgments

ILL[2] and HZB[1] sample environment groups

A. Perkins[2], J. Gonthier[2], S. Baudoin[2], E. Bourgeat-Lami[2], E. Lelièvre-Berna[2], M.A. Barrett[1], C. Teixeira[1], K. Kiefer[1], N. Grimm[1], J. Dathe[1]

Scientific input from

- T. Hauß[1], B. Demé[2] and M. Rheinstädter

Thank you for your attention