Next Generation Humidity Chamber for Neutron Scattering "BERILL"

NMI3-Soft Matter JRA-WP20

Dirk Wallacher, 2015-10-14, Copenhagen

In this talk…

- Motivation
- Project overview and evolution
- Test and results
- Summary and Outlook

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

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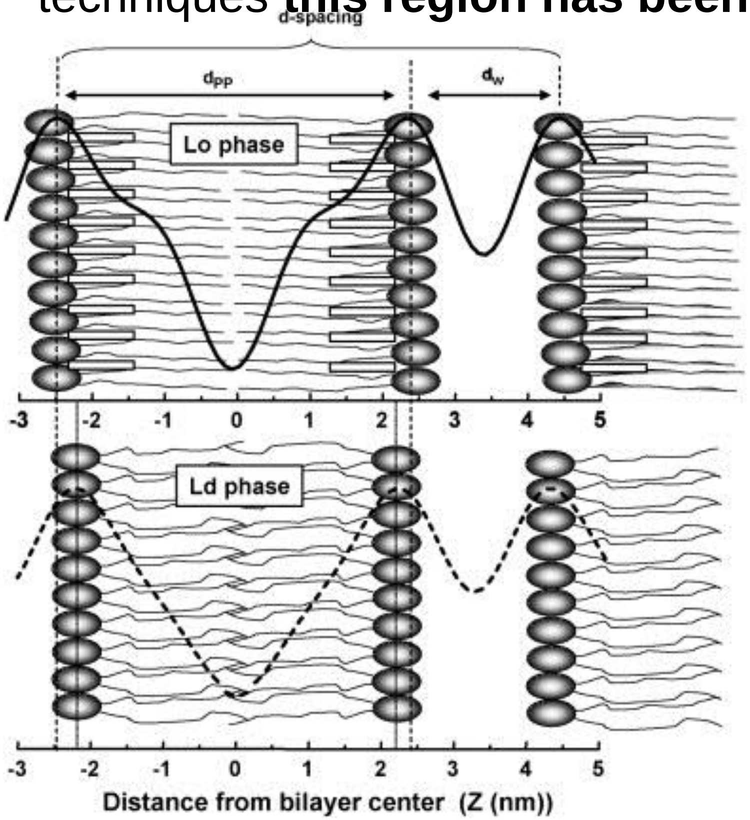
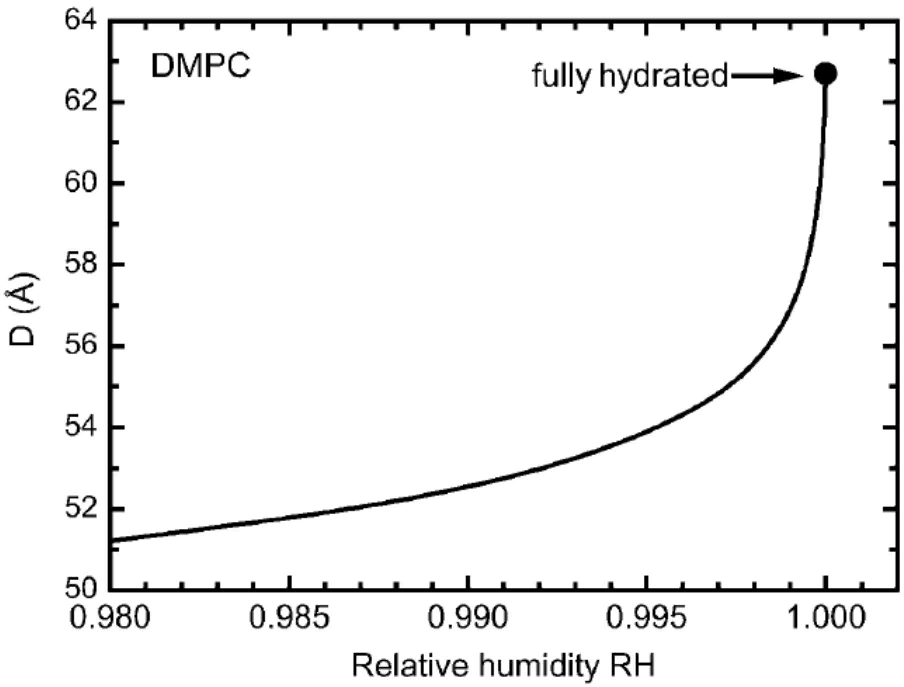
Toward 100% RH

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

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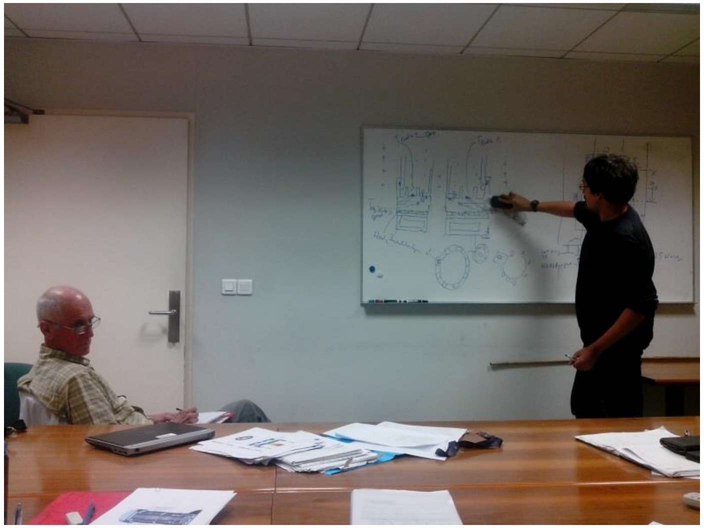
Timeline

Year 1:

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



Year 2:

Produce drawings

Year 3:

Build and commission chamber

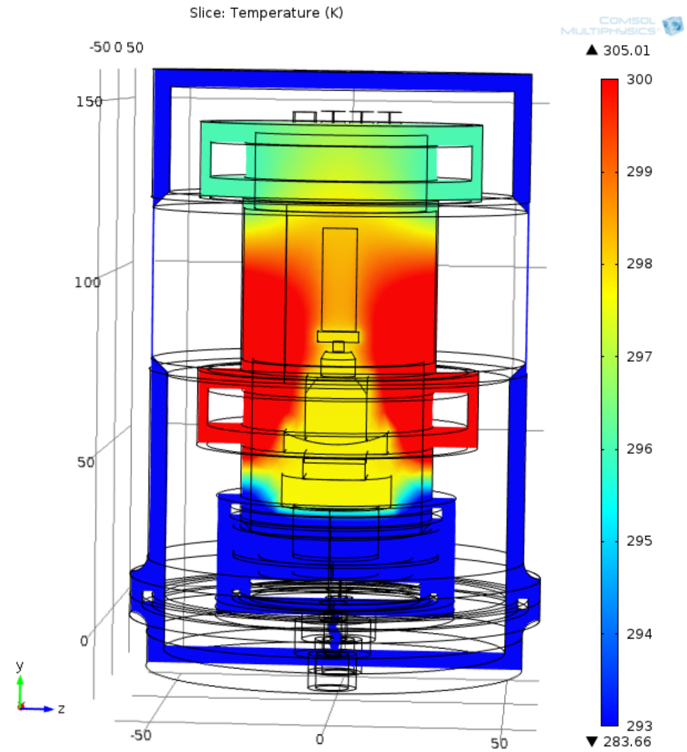
Relative Humidity Control

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

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

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

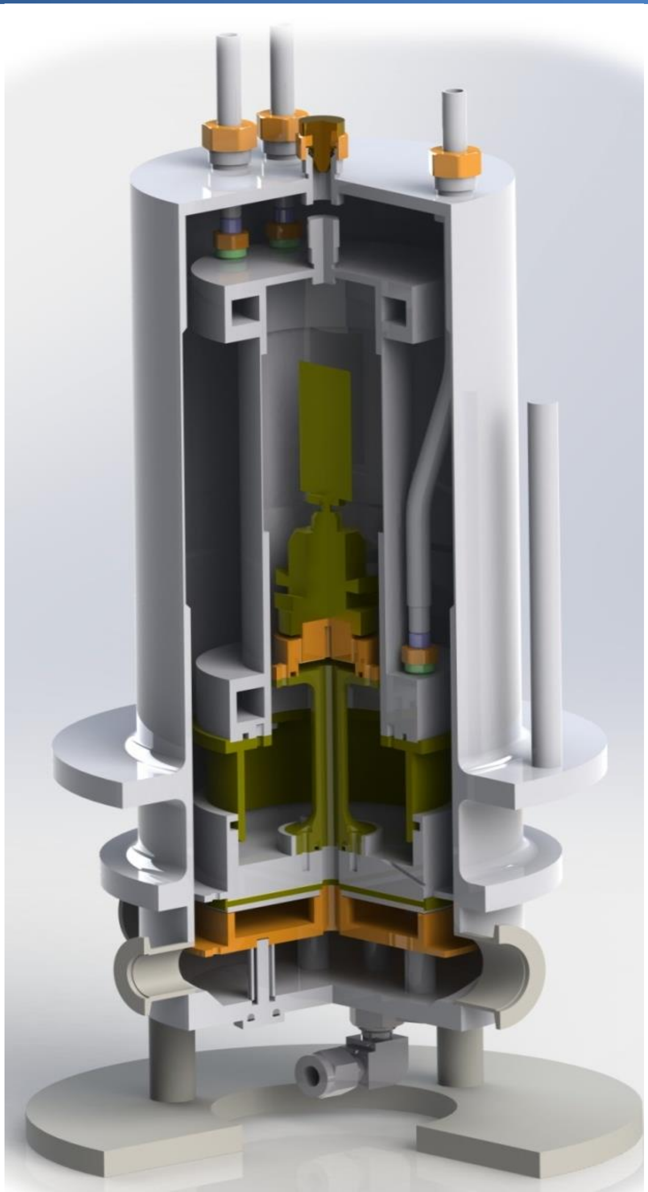
Total height 400 mm, diameter 150 mm

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

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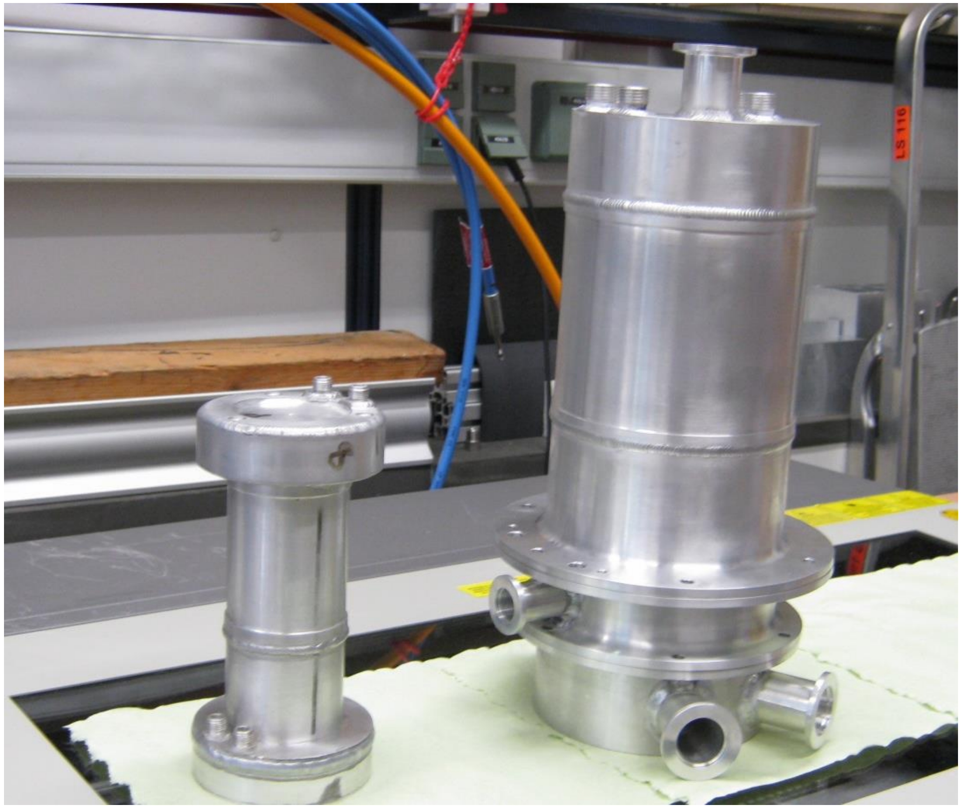
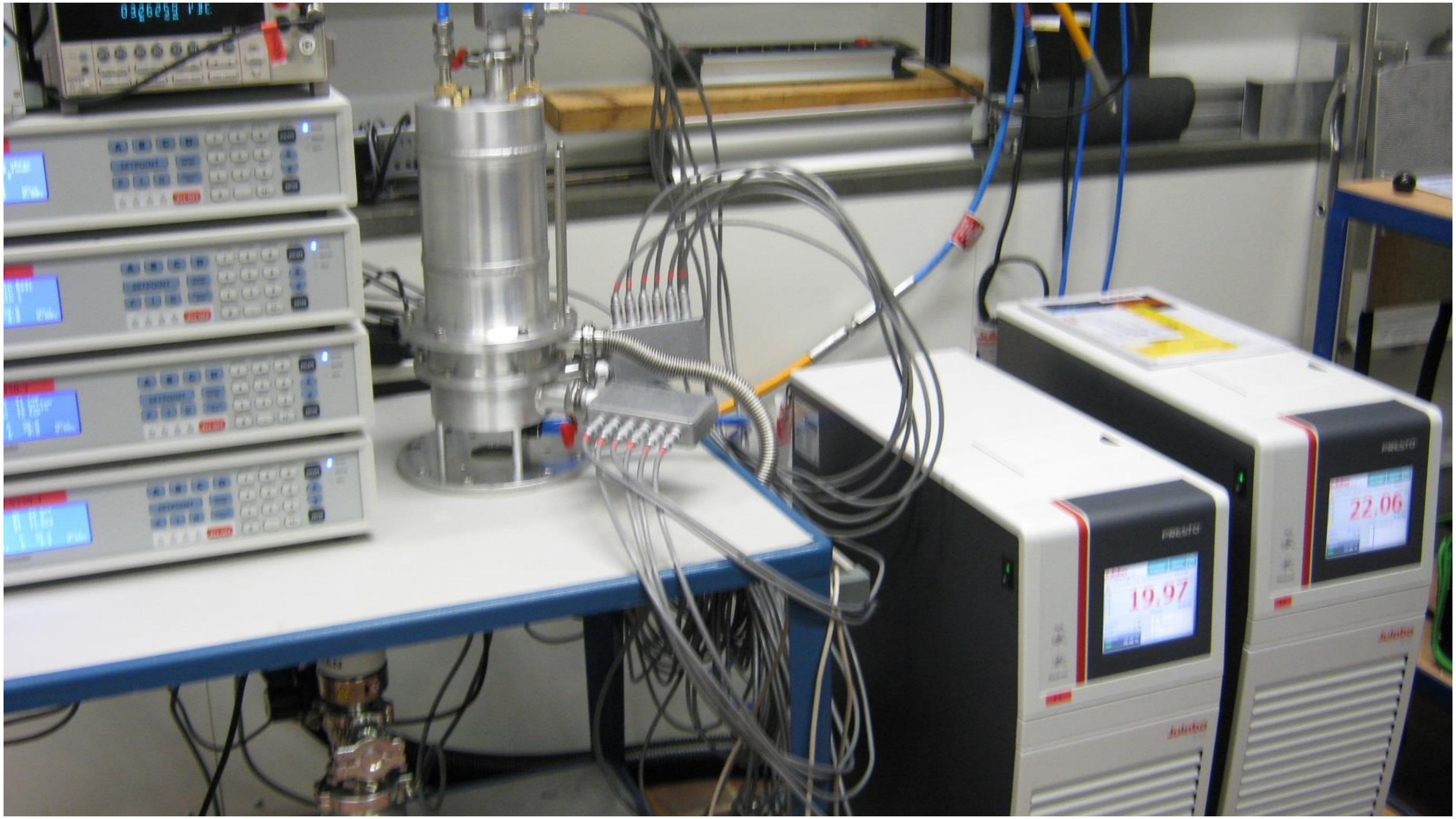
Assembly of 1st prototype

ILL parts + HZB parts

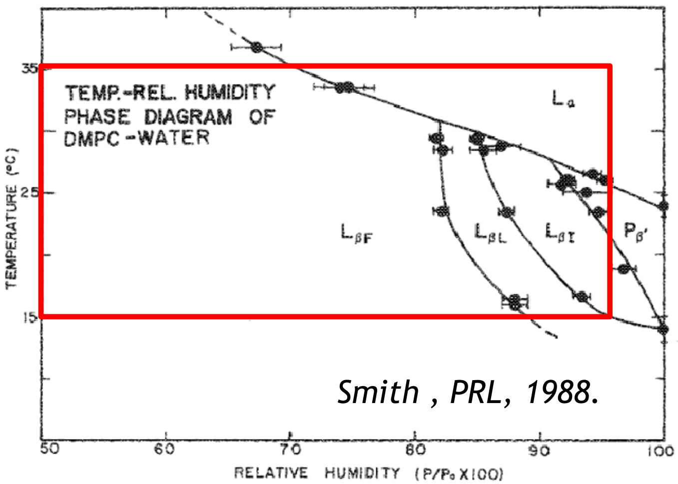


Complete Setup BERILL1.0

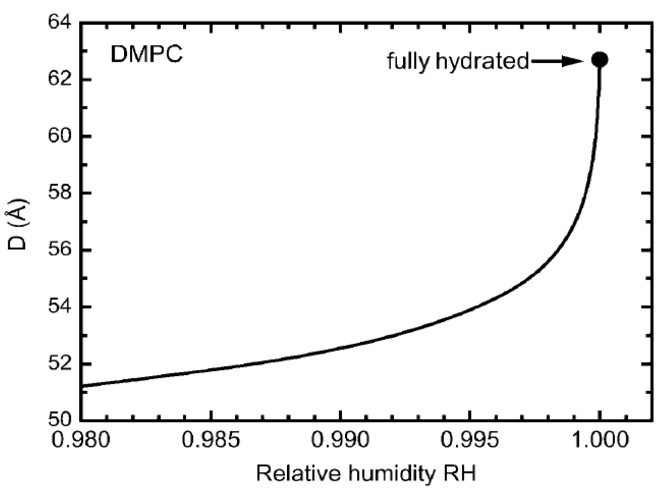


D16 @ ILL: December 2014

D16 results: December 2014

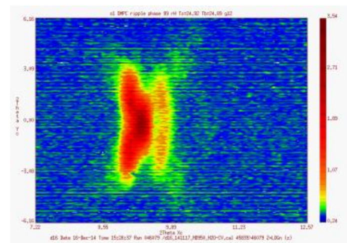


Smith , PRL, 1988.

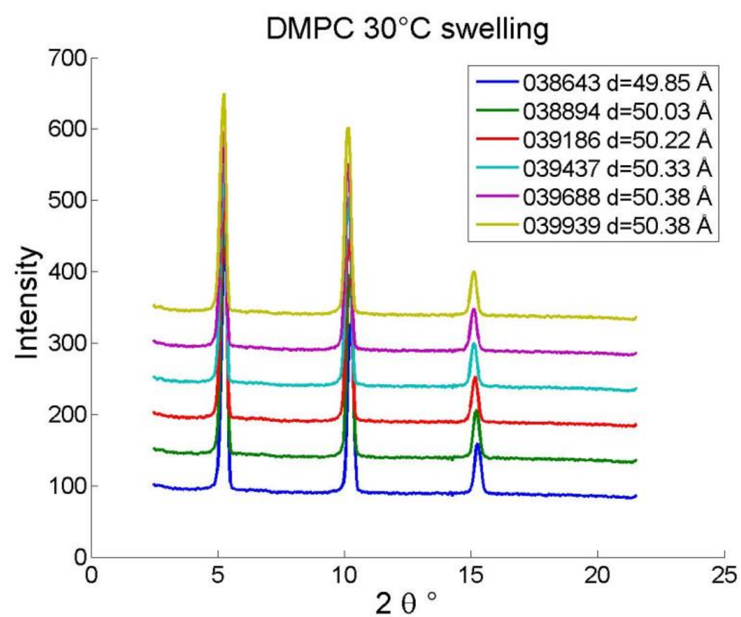


Kučerka, Biophysical Journal, 2005

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



- DMPC fluid phase swelling



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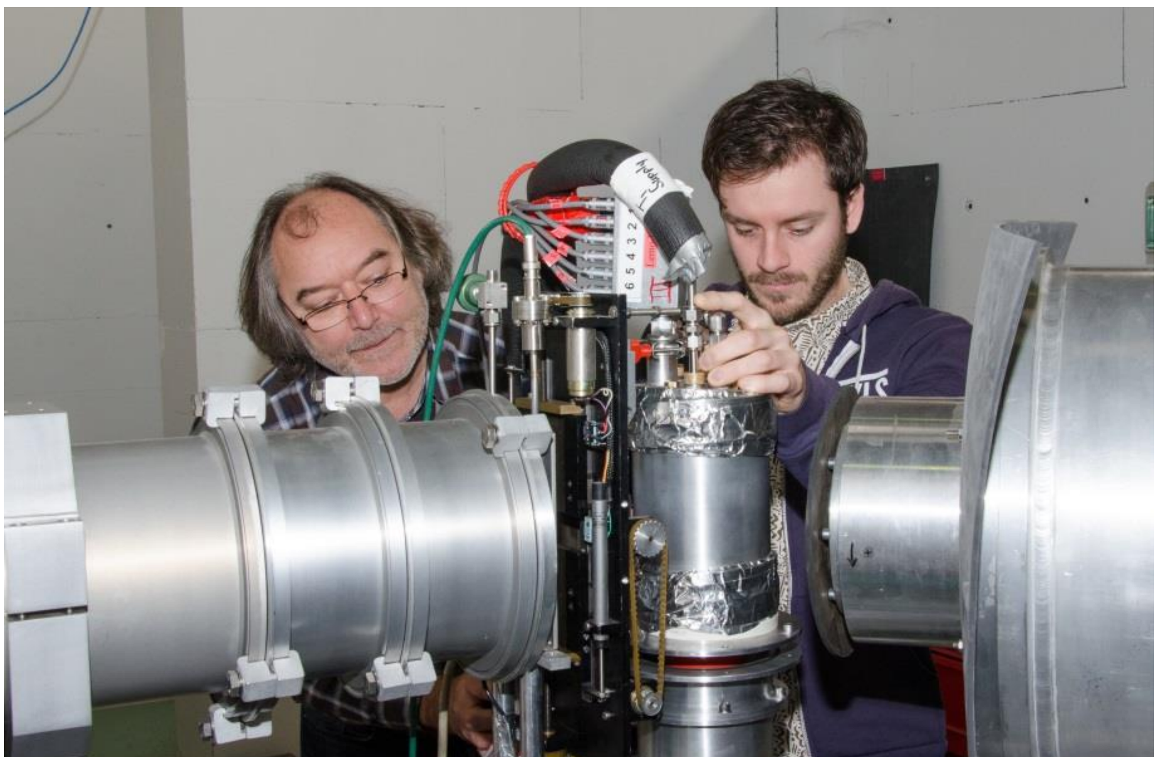
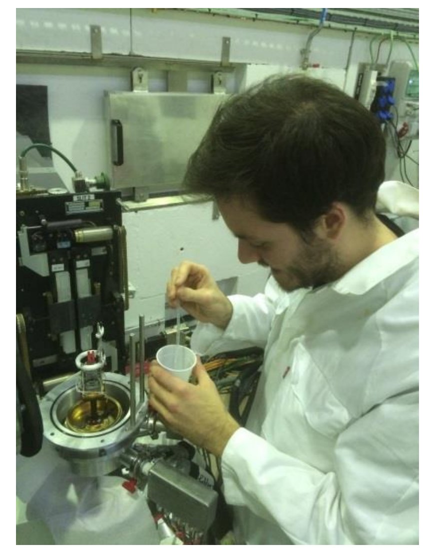
Problems December 2014

- Slow cooling oil leaks inside of chamber
- leaky sample chamber and bad isolation vacuum
- Maximum achieved r.H. 97%

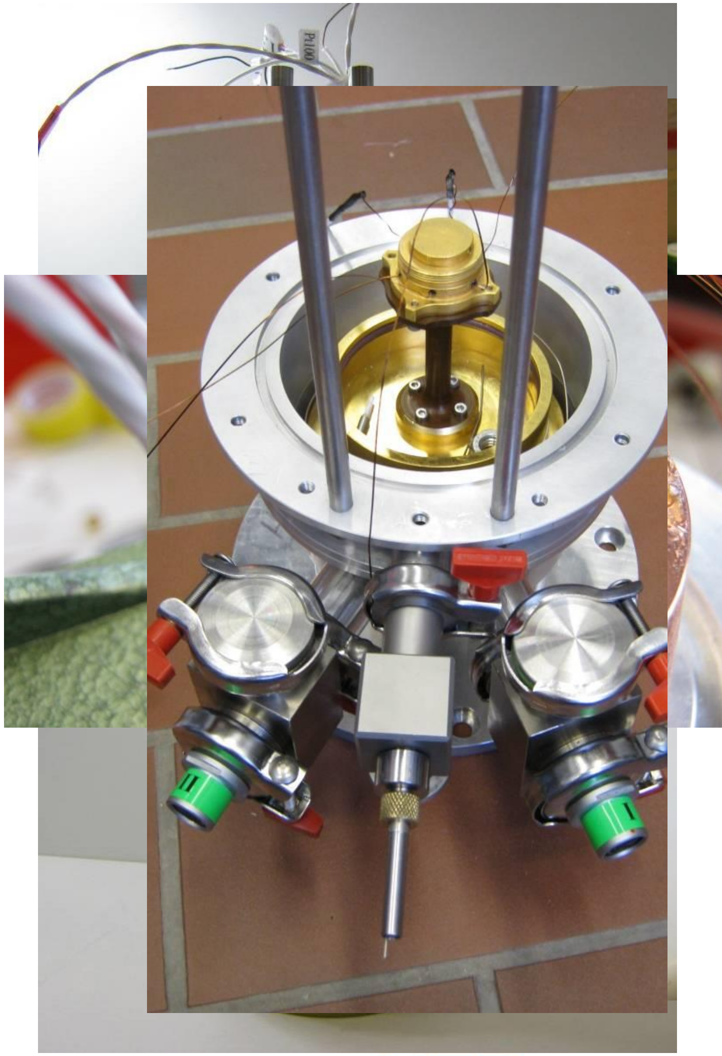




Winter modifications of BERILL1.0

- 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!



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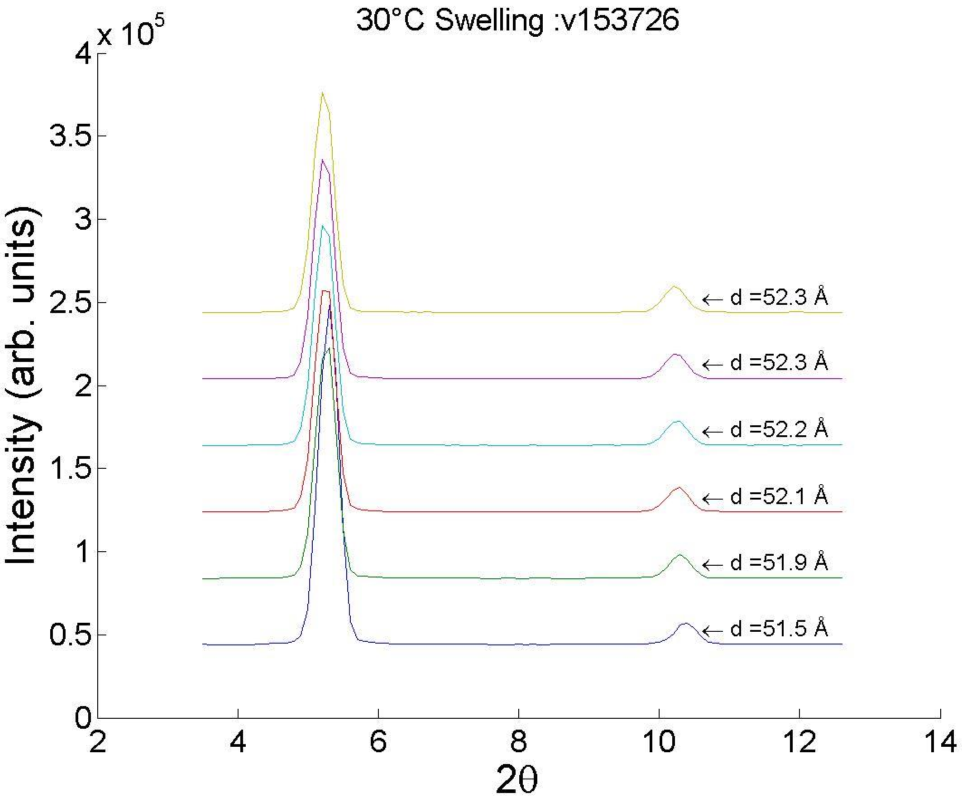
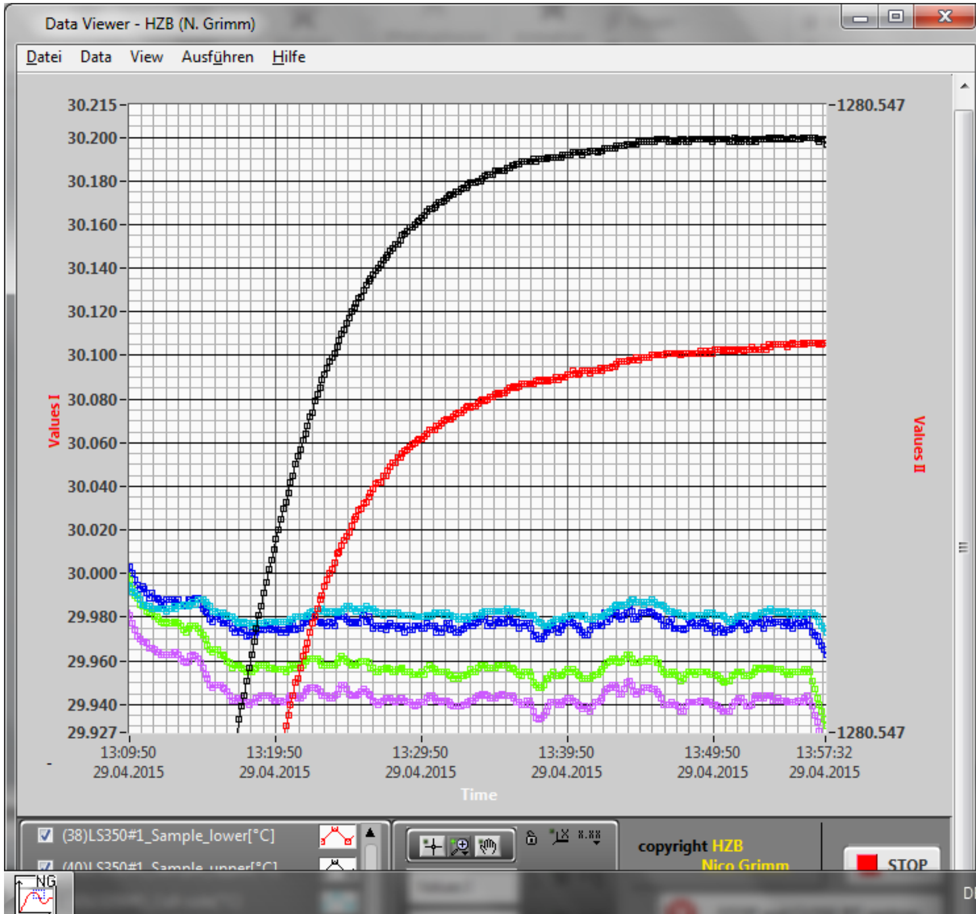
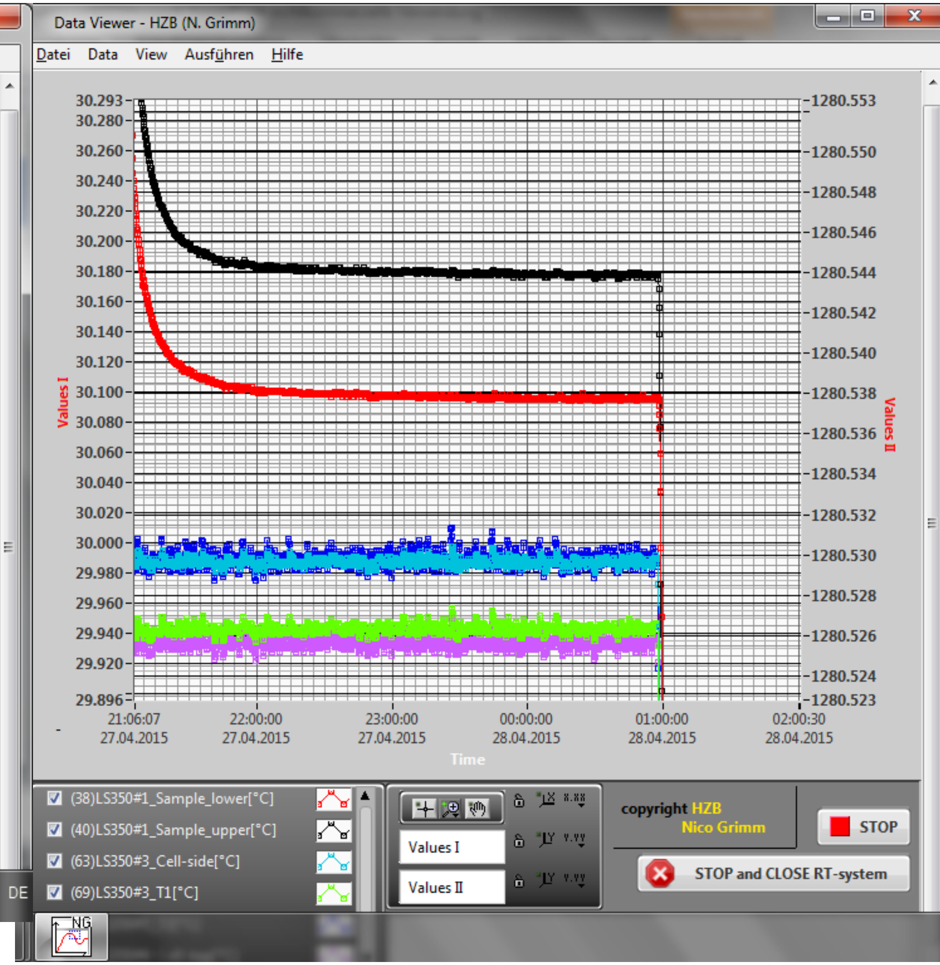
V1 @ HZB: April 2015

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



Modifications: April 2015





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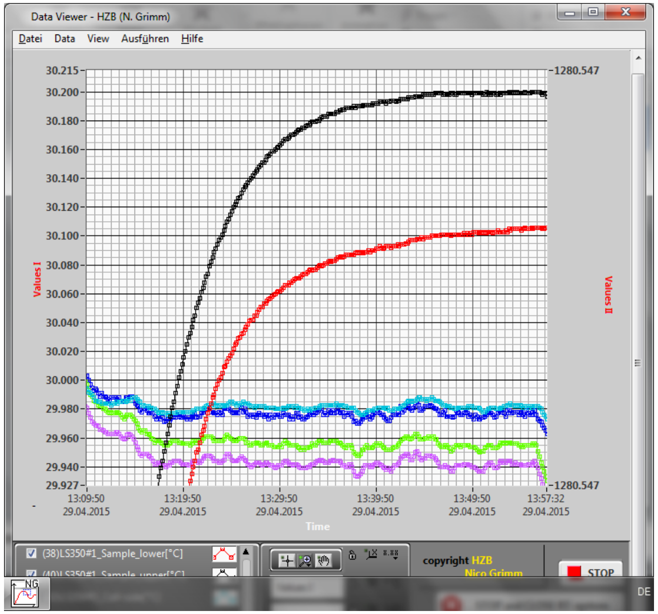
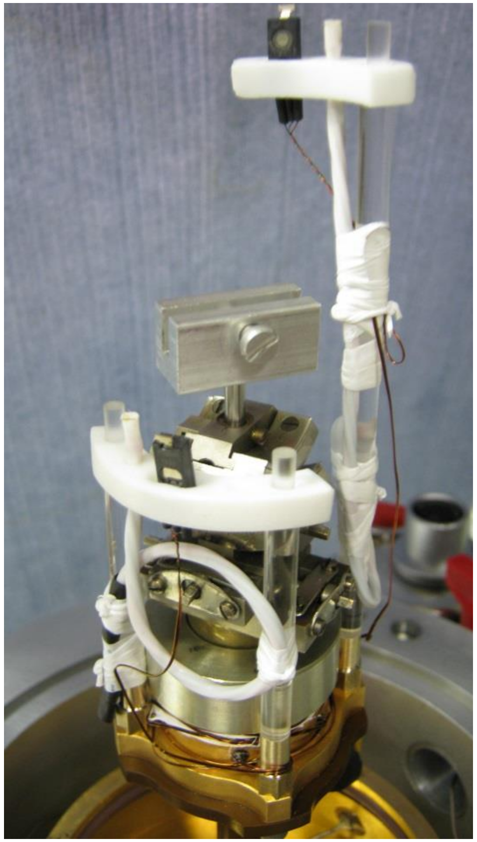
Modifications: April 2015

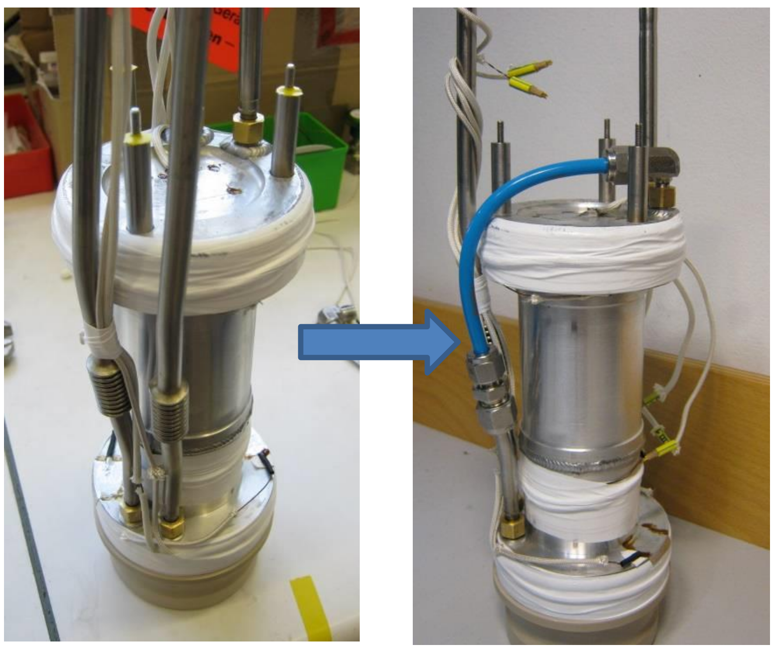
- Sample lower and sample upper consistently 100 and 200 mK warmer than rest of chamber?

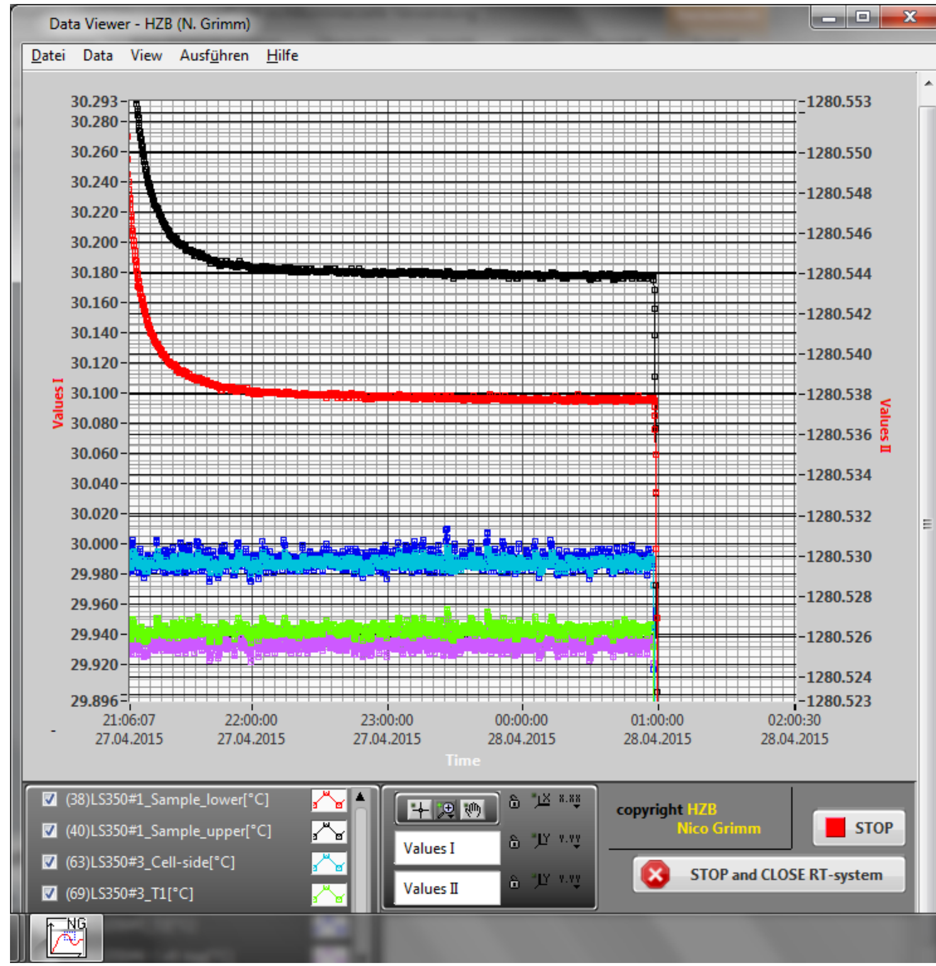




Modifications: April 2015

- Moved T1-T2 connector to inside of vacuum





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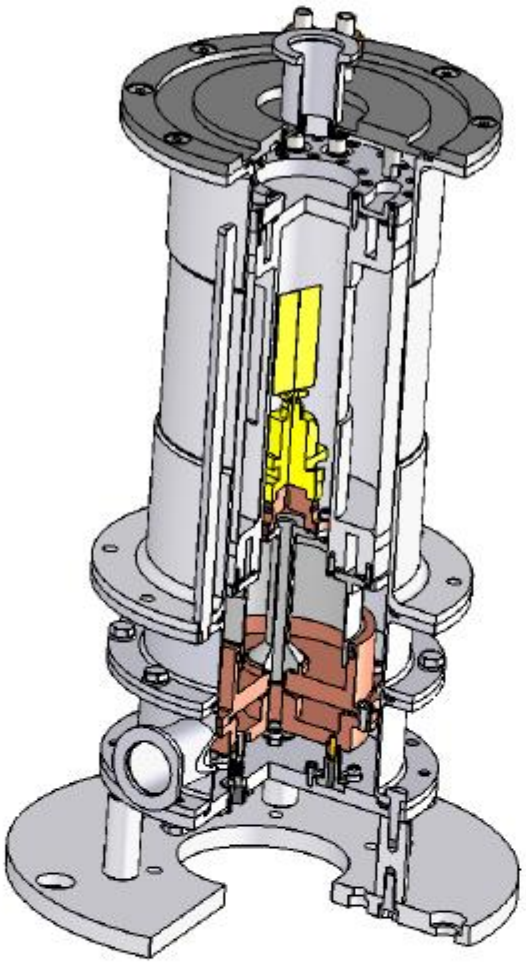
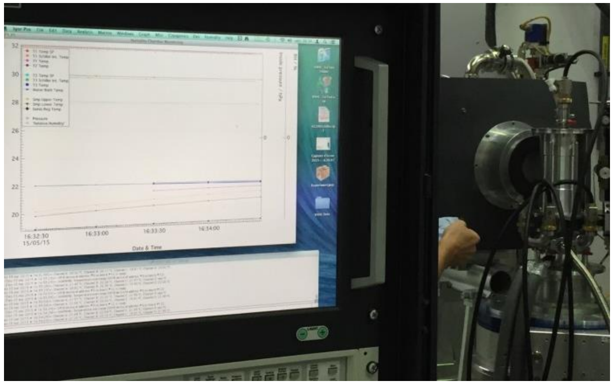
Construction BERILL 2.0

- 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)



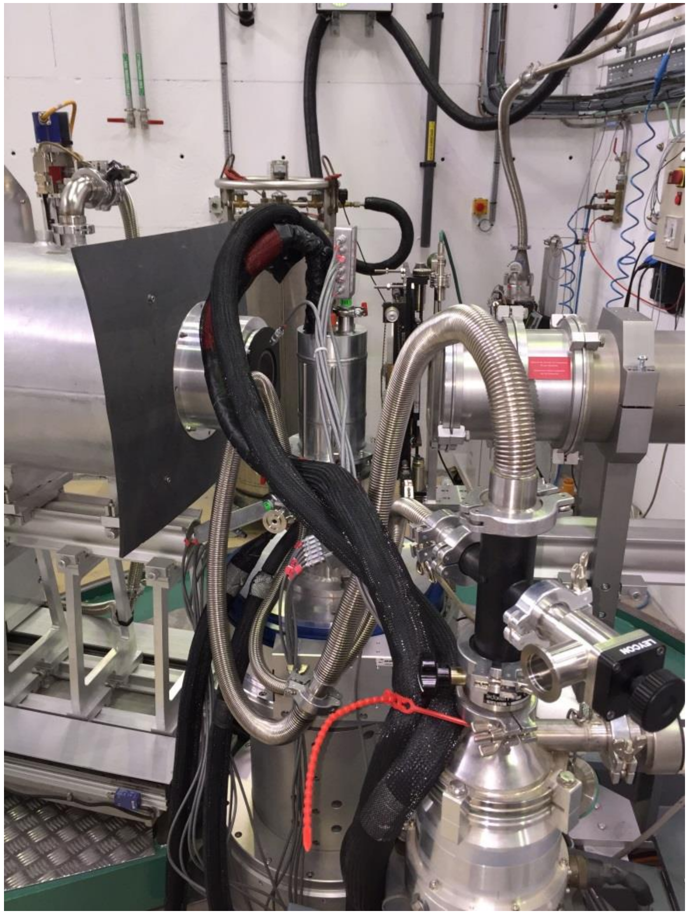
BerILL 2.0

- Inner chamber simplified with single chiller channel
- User friendly Igor interface (set desired sample temperature + humidity)



D16 @ILL: May 2015

BerILL 1.0



BerILL 2.0

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D16 - Results: May 2015

- Used DMPC sample (swelling at 30°C)
- Used DOPC sample (swelling at 20°C)
- Switched to unsaturated salt solution (1 mol% K_2SO_4 relaxes T parameters),

Same result for both old and new chamber:
no dramatic swelling, max. r.H. 98.5%

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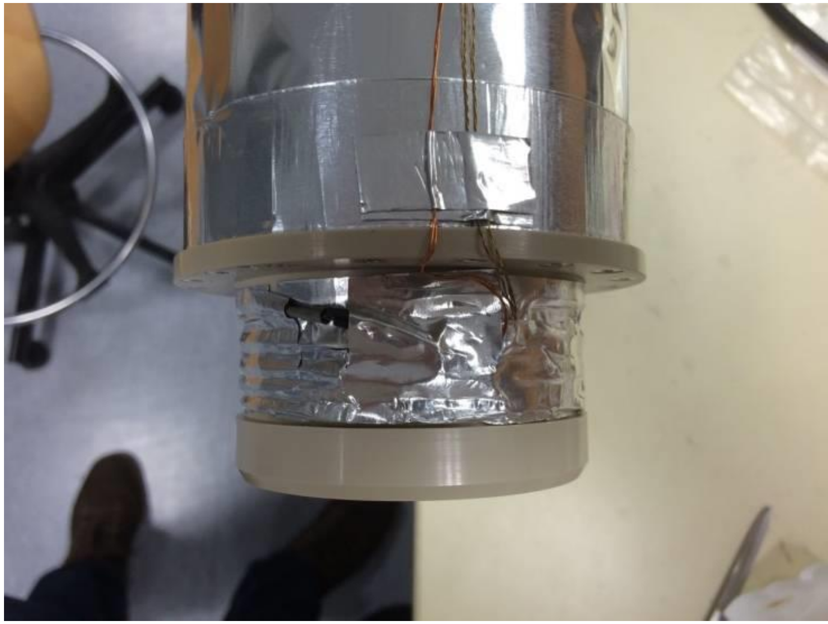
D16 @ ILL: May 2015

- Condensation seen on insulating ring (even with good vacuum of BerILL 2.0)



Conclusions: May 2015

- Why 100 % r.H. can not be acieved?

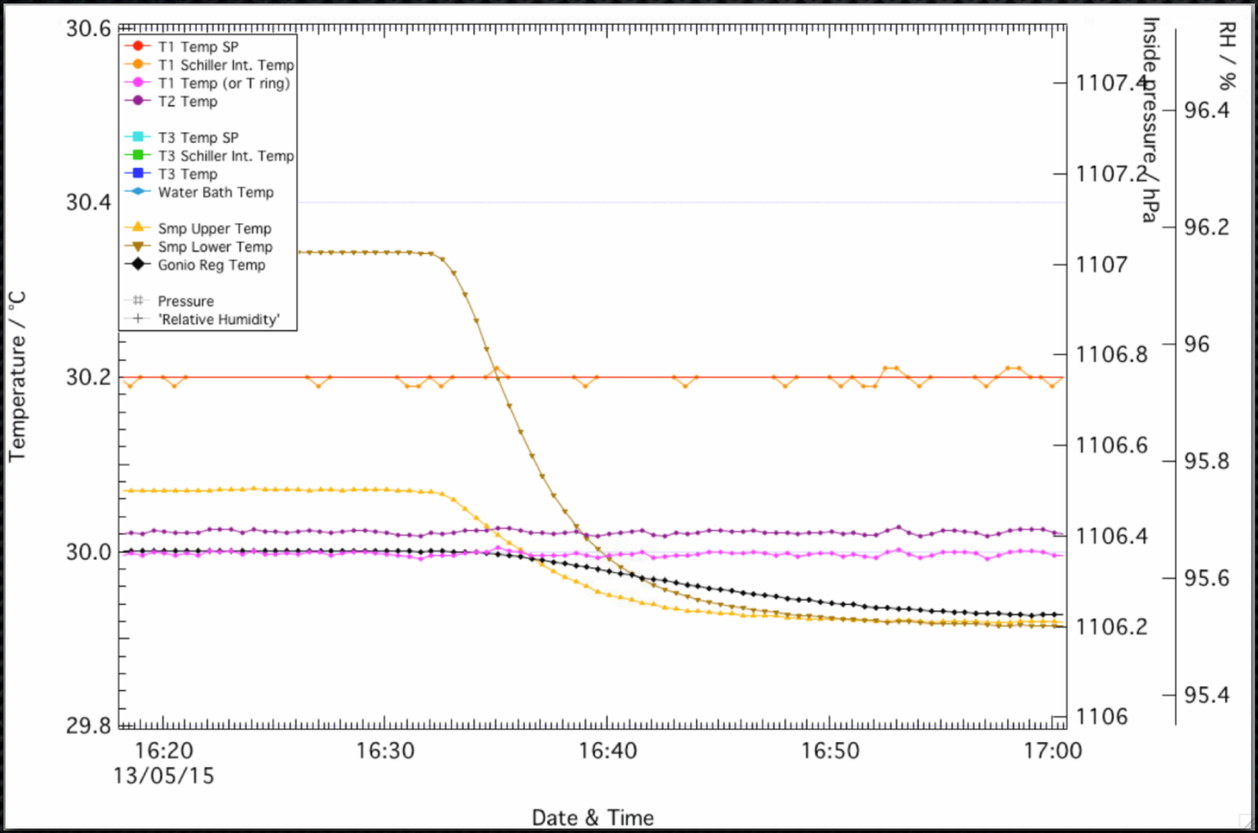
- Hypotheses:
  1. Cold point in chamber causes condensation not on sample, acts as water reservior
  2. Massive goniometer is slow to reach temperature, many moving surfaces create pores which must first fill
  3. No convection causes stagnant vapour density gradient between upper and lower sections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

SBM-JRA Meeting

Humidity Chamber / BerILL 2.0

D. Wallacher, J. Gonthier *et al.*

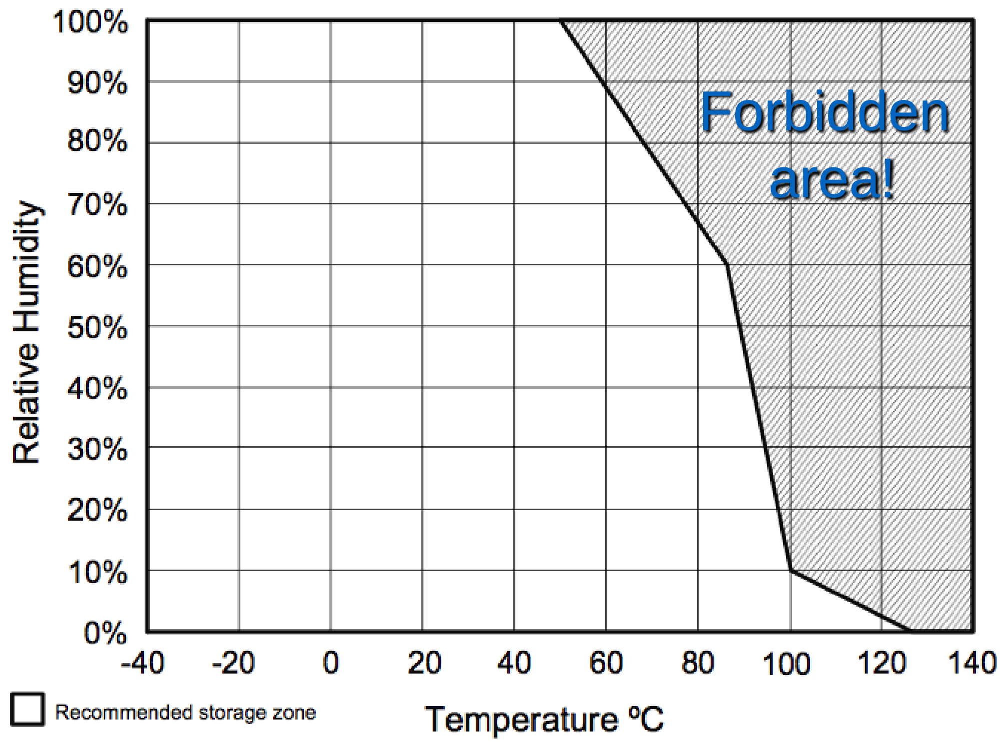
RH sensor influence



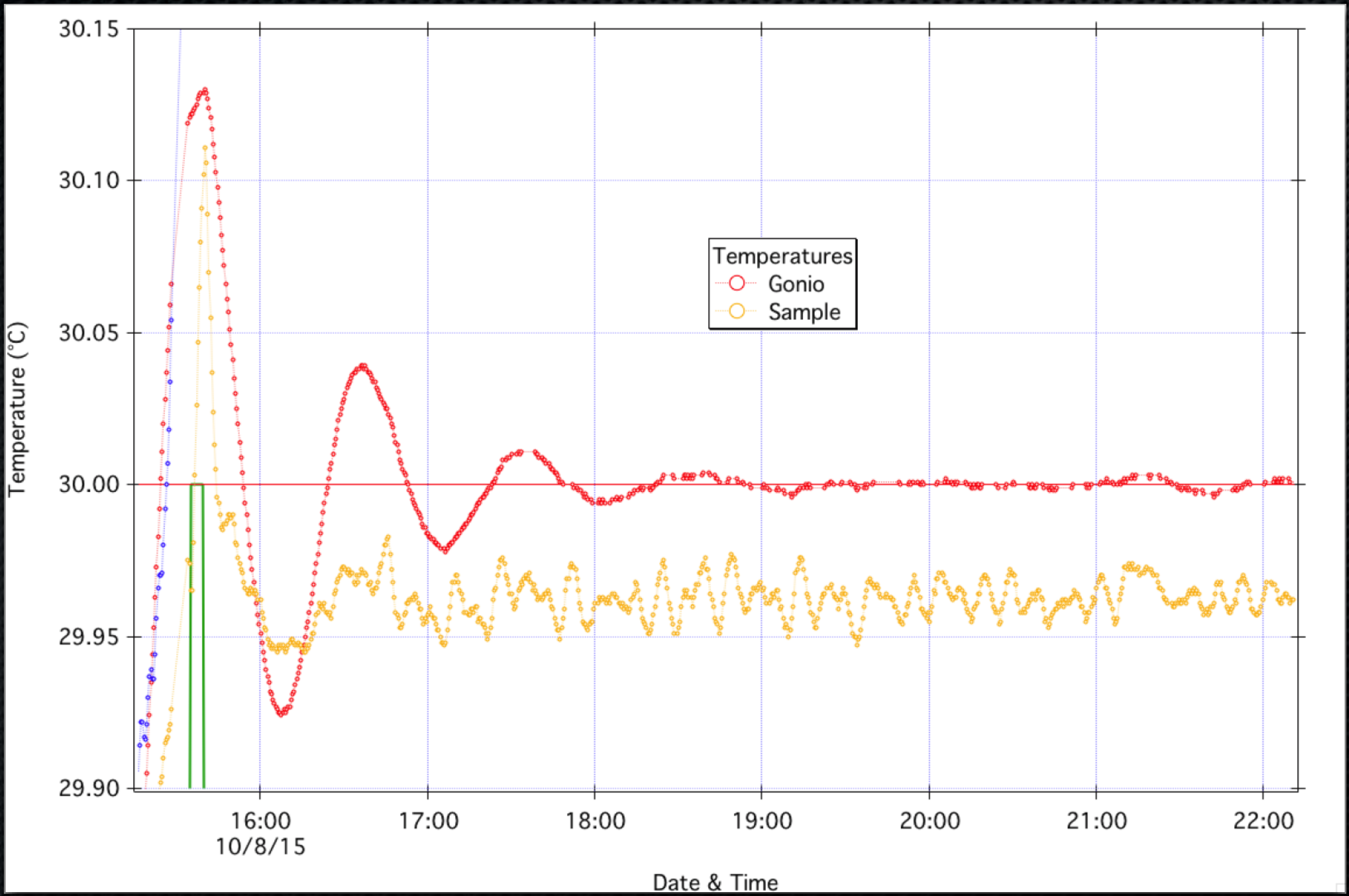
Sample temperature offset due to RH sensor

RH sensor « storage »

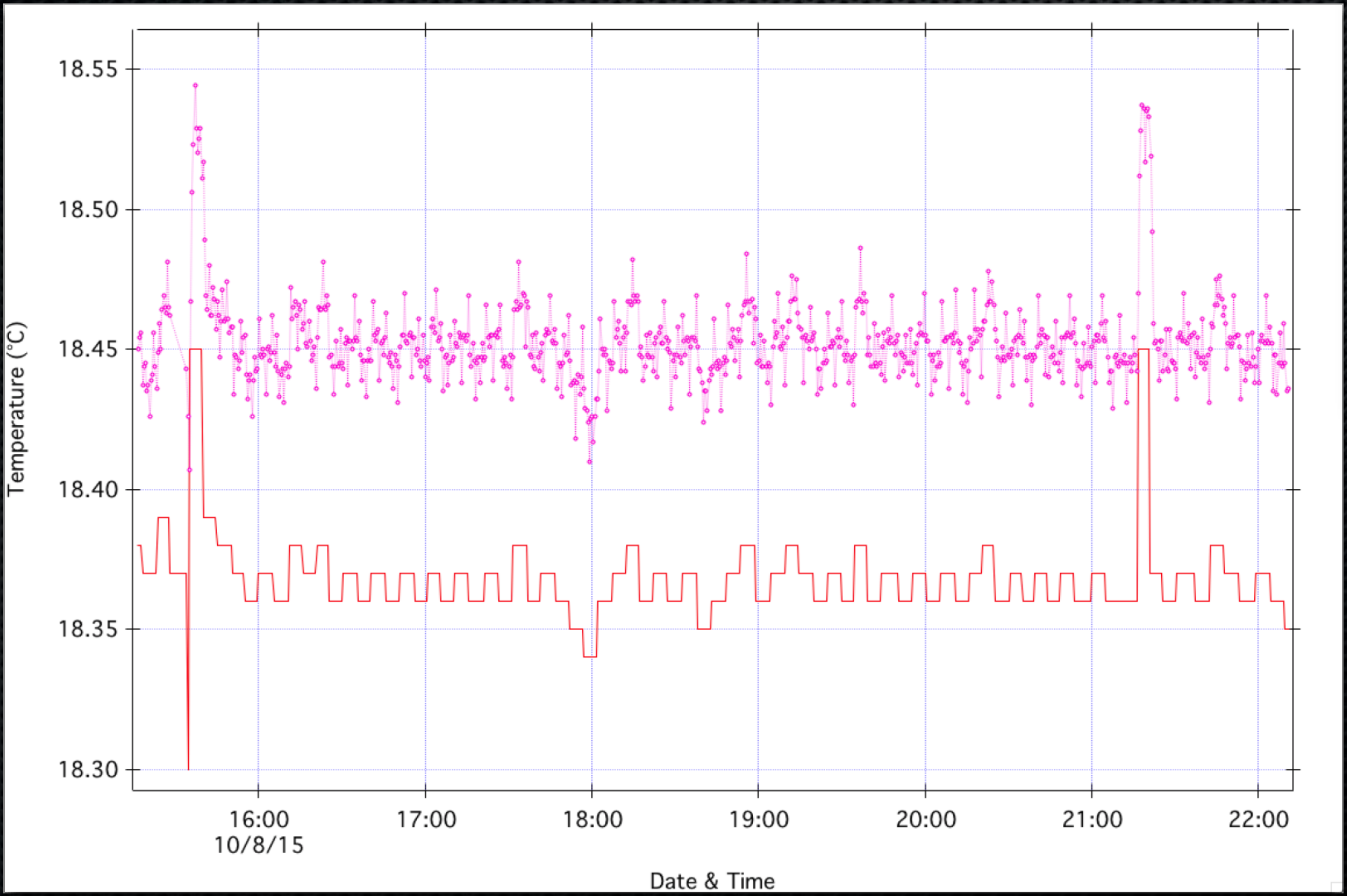
Figure 2. Storage Environment (Non-condensing environment.)



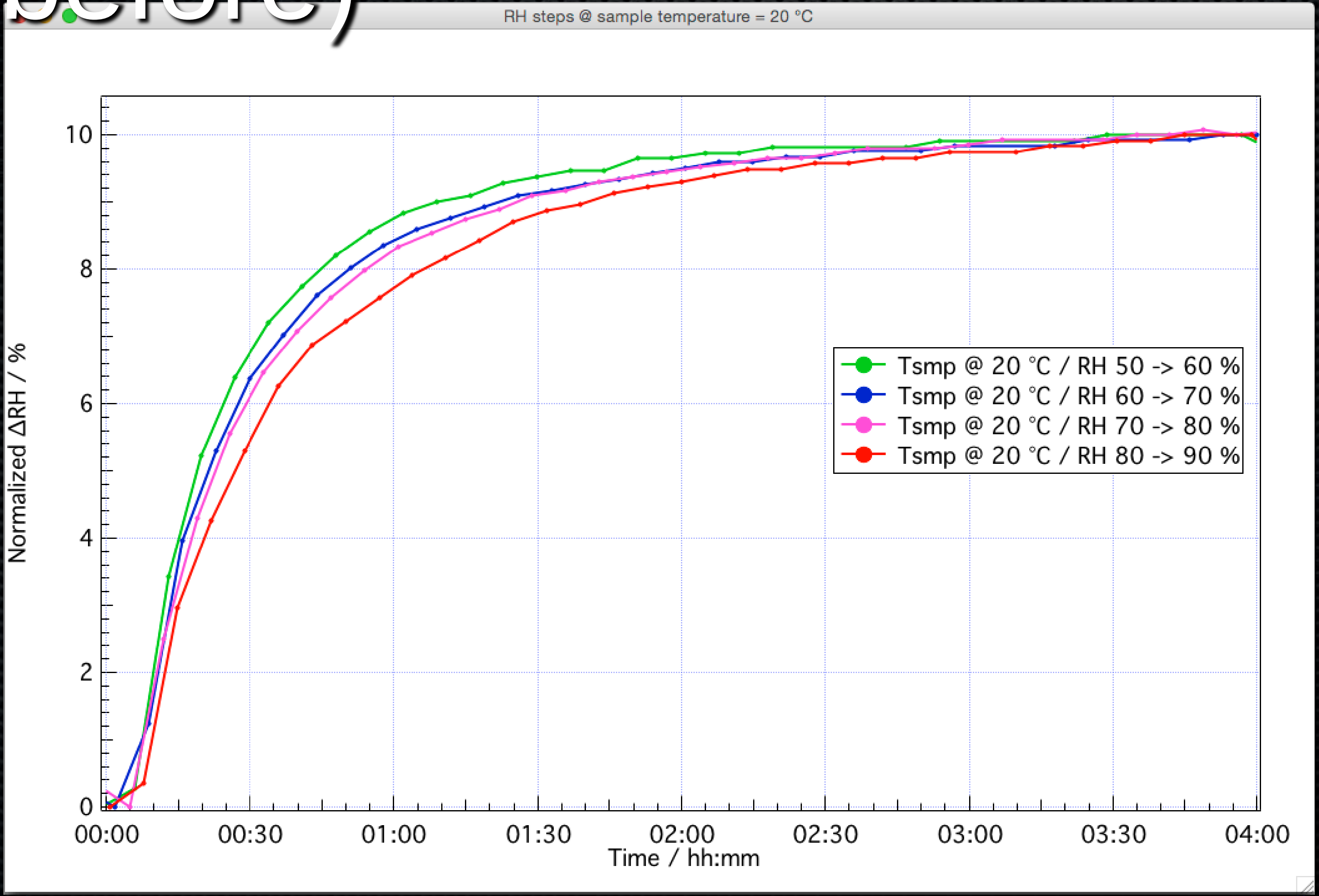
Temp. stability @ 50 % RH



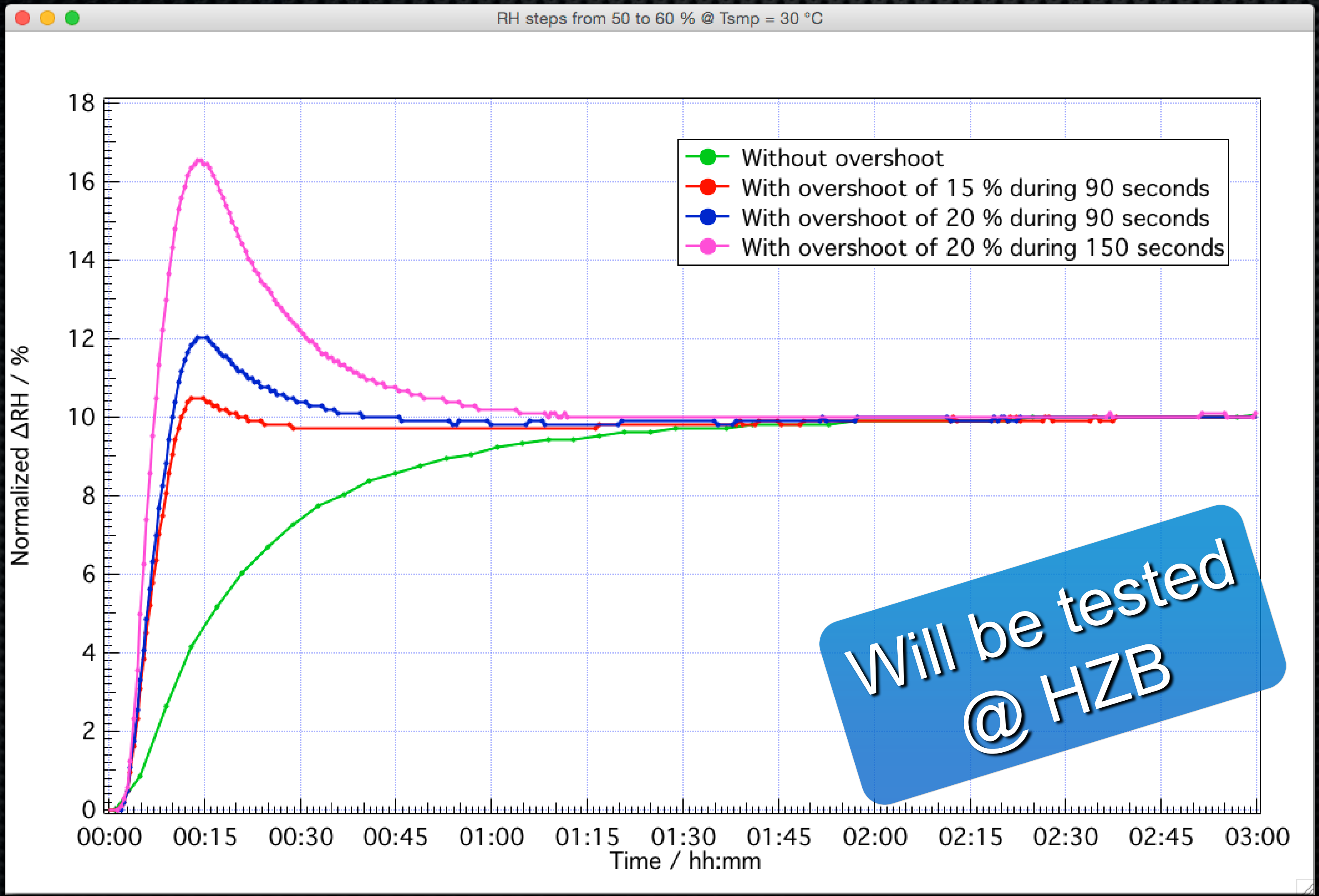
Reservoir Temp. Stability

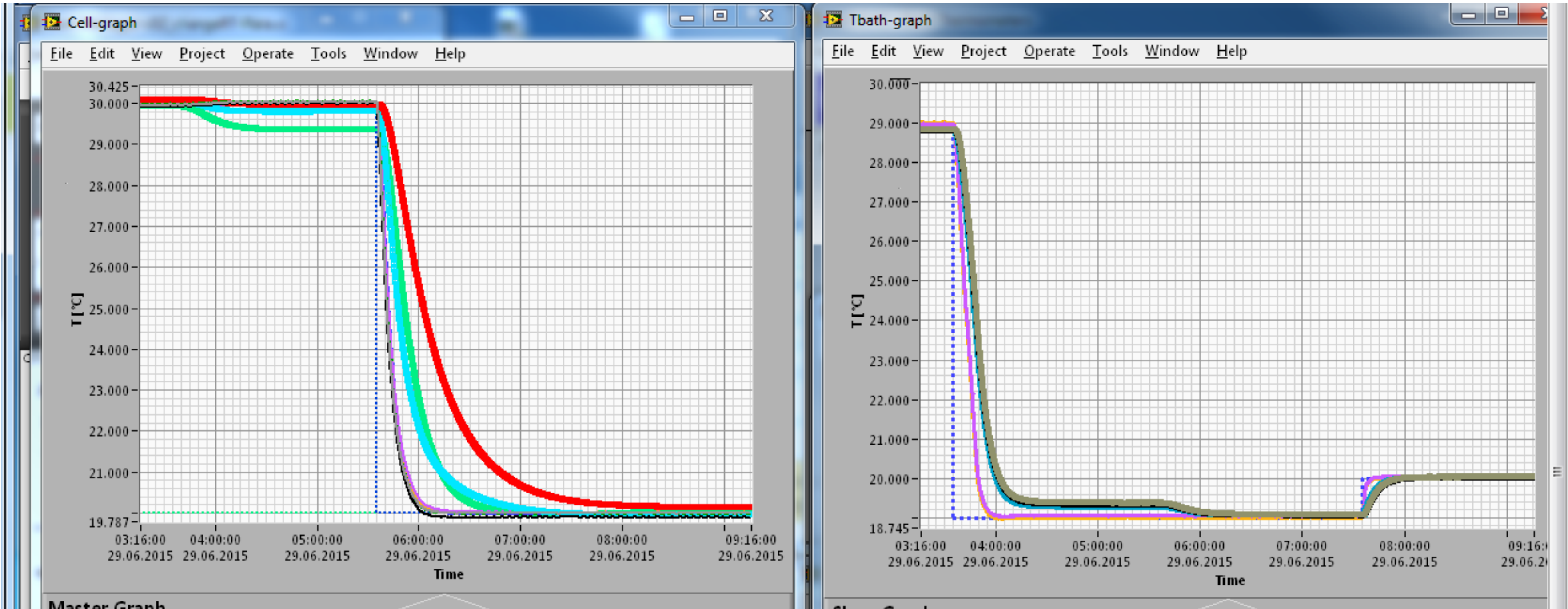


Time for stabilisation (before)



Time for stabilisation (now)





Cell-graph
File Edit View Project Operate Tools Window Help
30.425
30.000
29.000
28.000
27.000
26.000
25.000
24.000
23.000
22.000
21.000
19.787
T [°C]
03:16:00 29.06.2015
04:00:00 29.06.2015
05:00:00 29.06.2015
06:00:00 29.06.2015
07:00:00 29.06.2015
08:00:00 29.06.2015
09:16:00 29.06.2015
Time
Master Graph
Tbath-graph
File Edit View Project Operate Tools Window Help
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21.000
20.000
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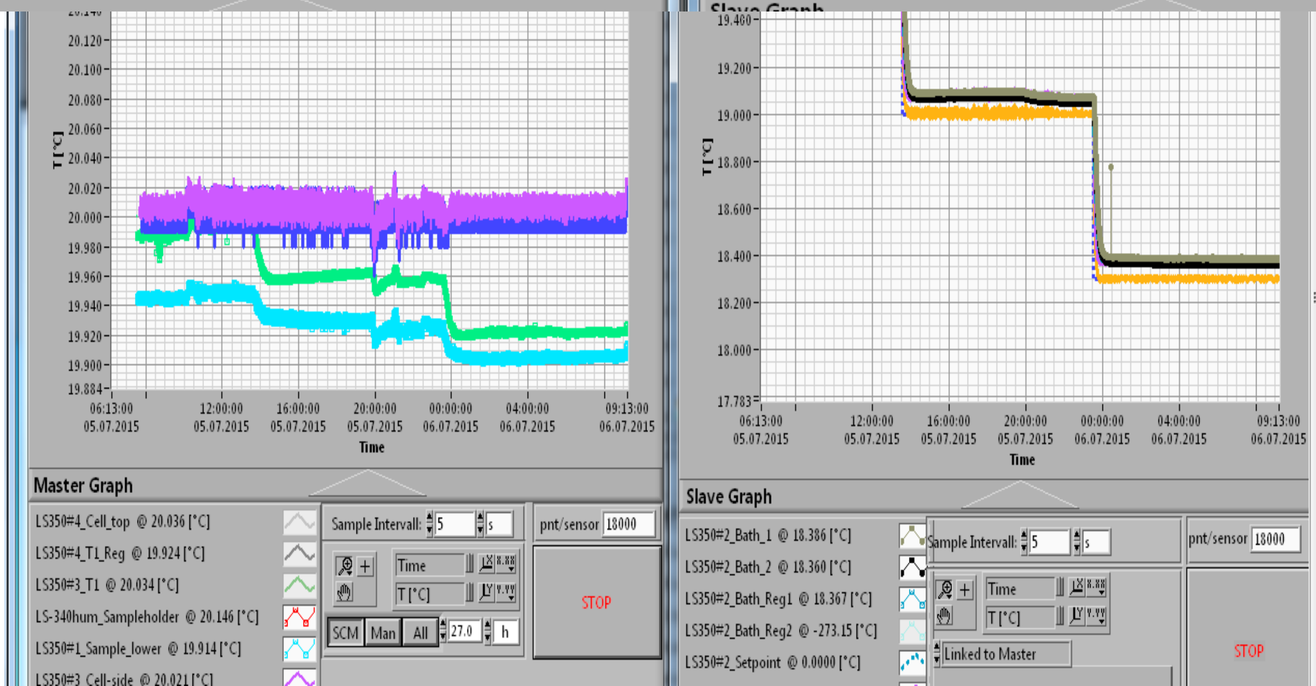
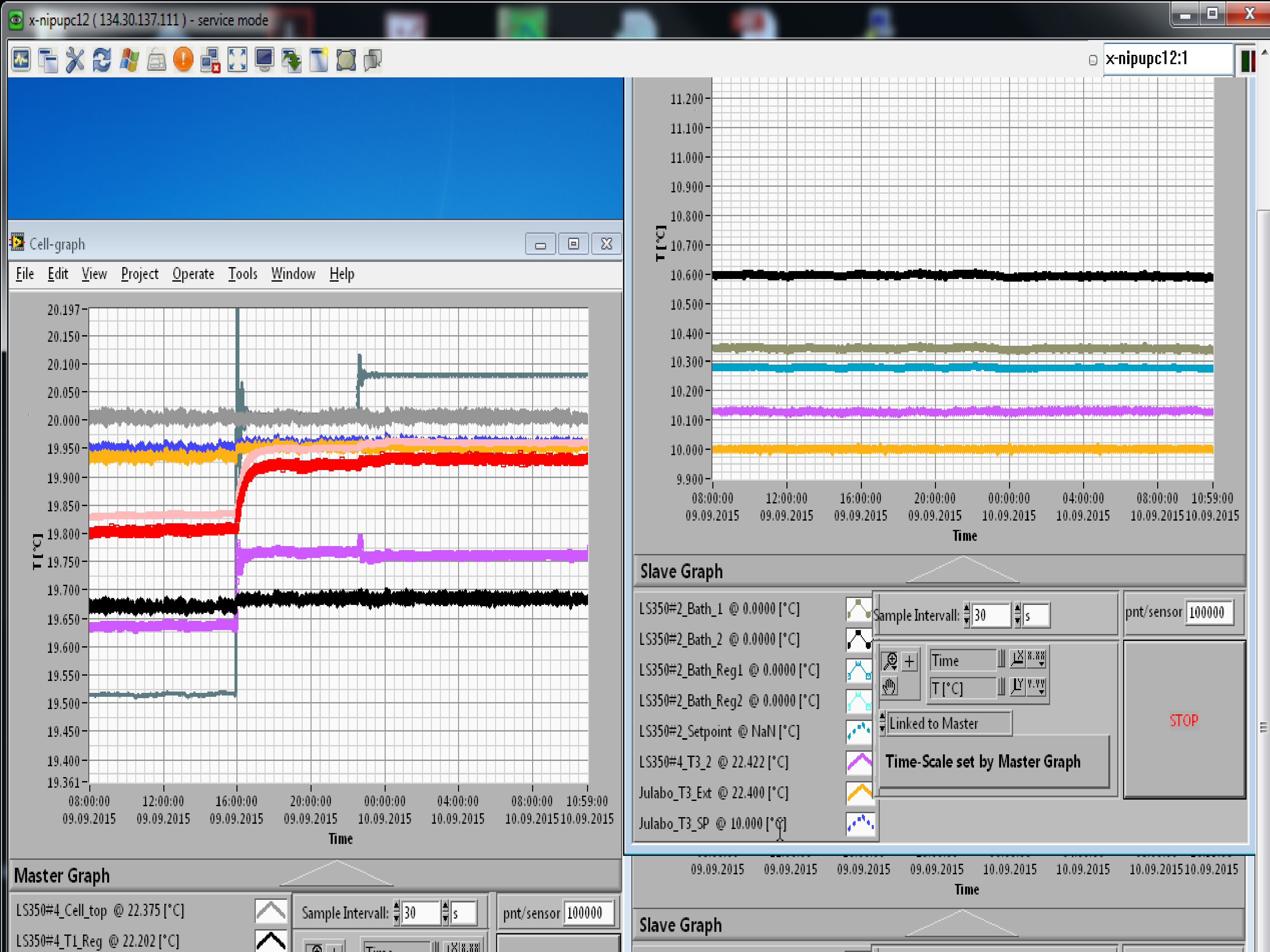
T [°C]
03:16:00 29.06.2015
04:00:00 29.06.2015
05:00:00 29.06.2015
06:00:00 29.06.2015
07:00:00 29.06.2015
08:00:00 29.06.2015
Time



20.120
20.100
20.080
20.060
20.040
20.020
20.000
19.980
19.960
19.940
19.920
19.900
19.884
T [°C]
06:13:00 05.07.2015
12:00:00 05.07.2015
16:00:00 05.07.2015
20:00:00 05.07.2015
00:00:00 06.07.2015
04:00:00 06.07.2015
09:13:00 06.07.2015
Time
Master Graph
LS350#4_Cell_top @ 20.036 [°C]
LS350#4_T1_Reg @ 19.924 [°C]
LS350#3_T1 @ 20.034 [°C]
LS-340hum_Sampleholder @ 20.146 [°C]
LS350#1_Sample_lower @ 19.914 [°C]
LS350#3_Cell-side @ 20.021 [°C]
Sample Intervall: 5 s
pnt/sensor 18000
Time
T [°C]
SCM Man All 27.0 h
STOP
19.400
19.200
19.000
18.800
18.600
18.400
18.200
18.000
17.783
T [°C]
06:13:00 05.07.2015
12:00:00 05.07.2015
16:00:00 05.07.2015
20:00:00 05.07.2015
00:00:00 06.07.2015
04:00:00 06.07.2015
09:13:00 06.07.2015
Time
Slave Graph
LS350#2_Bath_1 @ 18.386 [°C]
LS350#2_Bath_2 @ 18.360 [°C]
LS350#2_Bath_Reg1 @ 18.367 [°C]
LS350#2_Bath_Reg2 @ -273.15 [°C]
LS350#2_Setpoint @ 0.0000 [°C]
Sample Intervall: 5 s
pnt/sensor 18000
Time
T [°C]
Linked to Master
STOP



x-nipupc12 (134.30.137.111) - service mode
x-nipupc12:1
Cell-graph
File
Edit
View
Project
Operate
Tools
Window
Help
T [°C]
Time
08:00:00 09.09.2015
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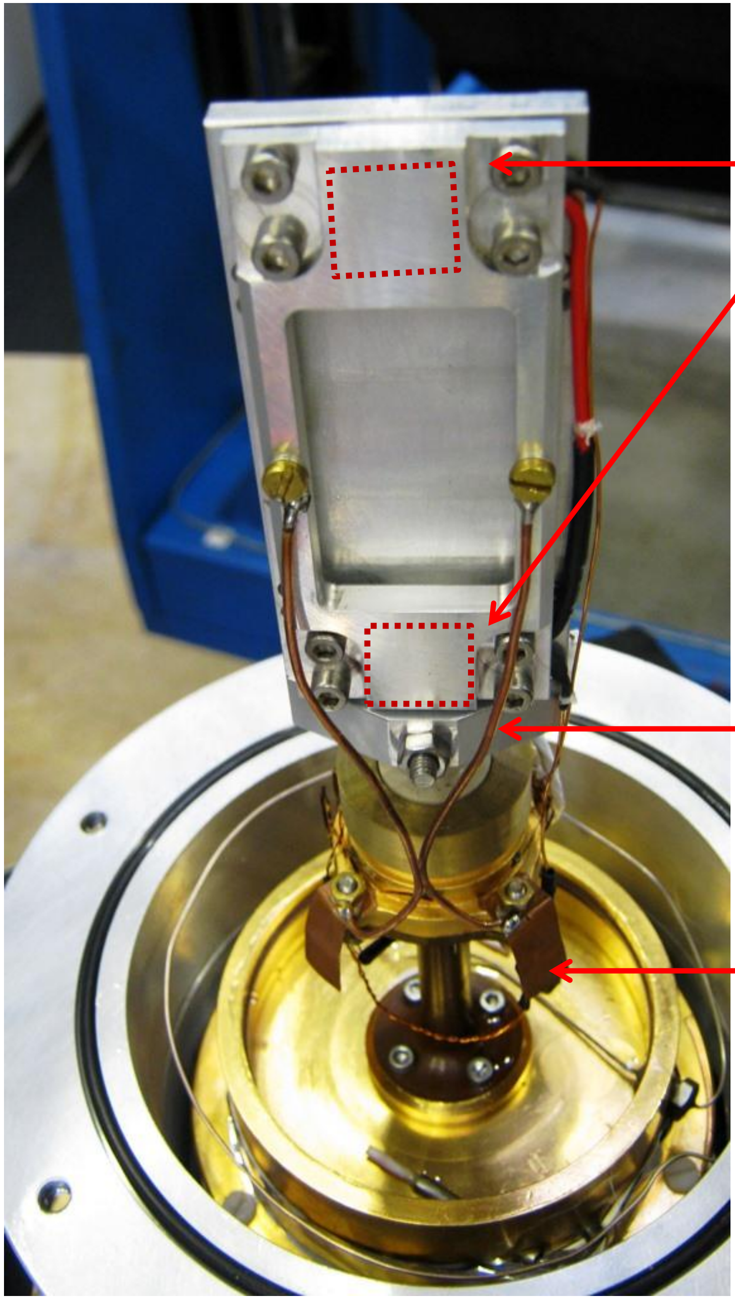
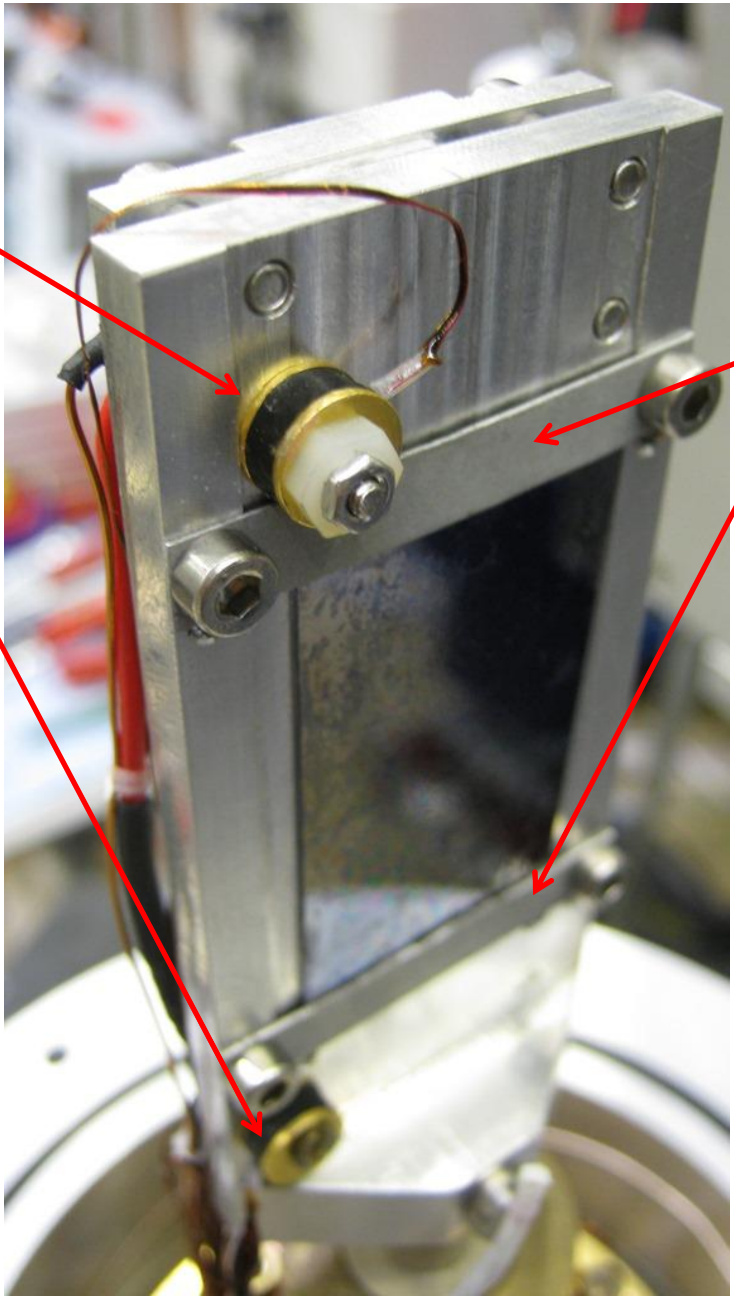
20:00:00 09.09.2015
00:00:00 10.09.2015
04:00:00 10.09.2015
08:00:00 10.09.2015
10:59:00 10.09.2015
Master Graph
LS350#4_Cell_top @ 22.375 [°C]
LS350#4_T1_Reg @ 22.202 [°C]
Sample Intervall: 30 s
pnt/sensor 100000
Slave Graph
LS350#2_Bath_1 @ 0.0000 [°C]
LS350#2_Bath_2 @ 0.0000 [°C]
LS350#2_Bath_Reg1 @ 0.0000 [°C]
LS350#2_Bath_Reg2 @ 0.0000 [°C]
LS350#2_Setpoint @ NaN [°C]
LS350#4_T3_2 @ 22.422 [°C]
Julabo_T3_Ext @ 22.400 [°C]
Julabo_T3_SP @ 10.000 [°C]
Time
T [°C]
Linked to Master
Time-Scale set by Master Graph
STOP

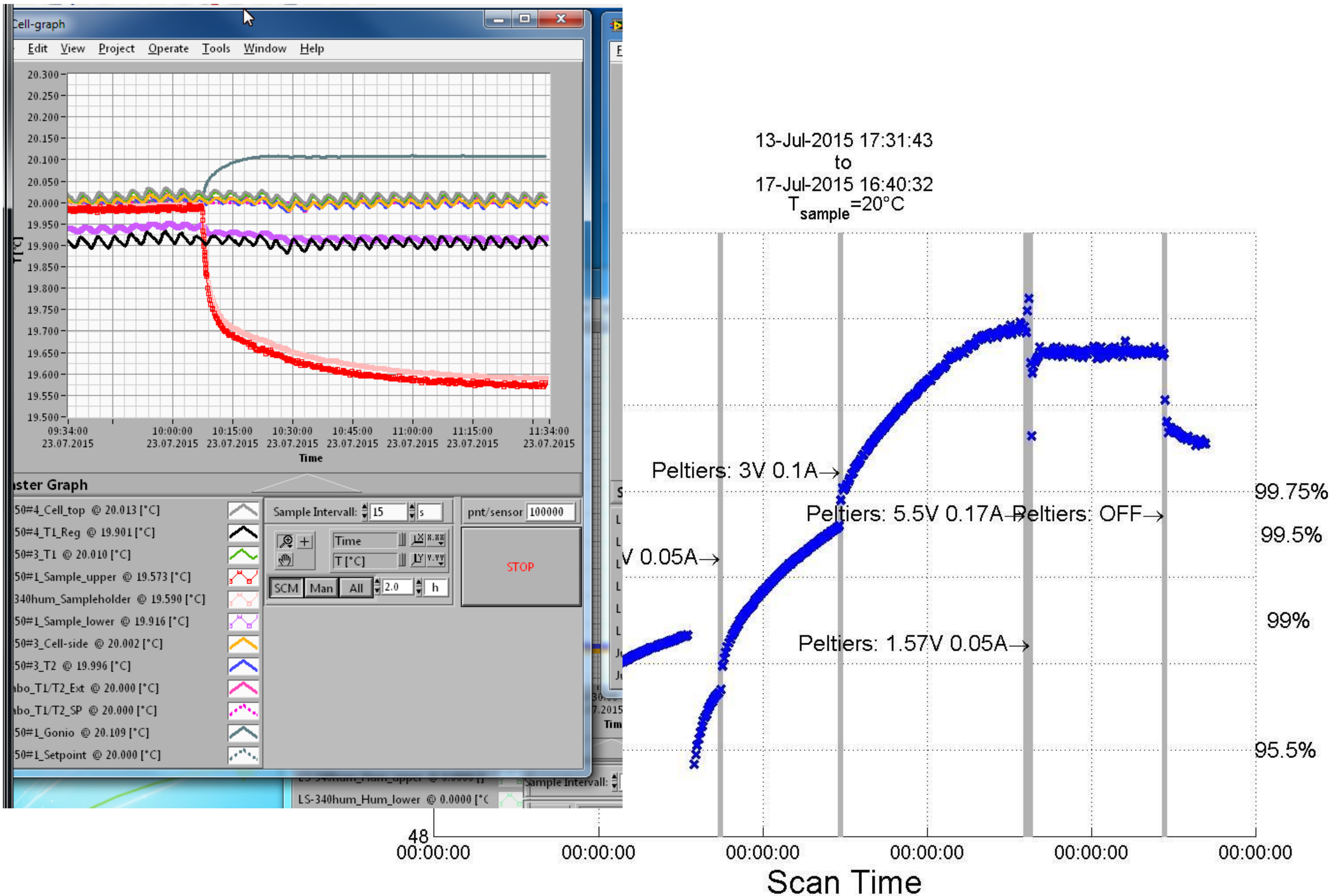

2 x Peltier elements
QC-17-1.0-2.5MS
Quick-Cool-Shop
2 x CU wires for
heat transport to Gonio
head
4 x CU plates for
shorting Gonio and
T1/T2



X64252
calibration sensor

Dt607
Additional sensor

2 x cadmium plates for clamping the Si-wafer



Cell-graph
Edit View Project Operate Tools Window Help
20.300
20.250
20.200
20.150
20.100
20.050
20.000
19.950
19.900
19.850
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19.750
19.700
19.650
19.600
19.550
19.500
T [°C]
09:34:00 23.07.2015
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10:15:00 23.07.2015
10:30:00 23.07.2015
10:45:00 23.07.2015
11:00:00 23.07.2015
11:15:00 23.07.2015
11:34:00 23.07.2015
Time
ster Graph
50#4_Cell_top @ 20.013 [°C]
50#4_T1_Reg @ 19.901 [°C]
50#3_T1 @ 20.010 [°C]
50#1_Sample_upper @ 19.573 [°C]
340hum_Sampleholder @ 19.590 [°C]
50#1_Sample_lower @ 19.916 [°C]
50#3_Cell-side @ 20.002 [°C]
50#3_T2 @ 19.996 [°C]
abo_T1/T2_Ext @ 20.000 [°C]
abo_T1/T2_SP @ 20.000 [°C]
50#1_Gonio @ 20.109 [°C]
50#1_Setpoint @ 20.000 [°C]
Sample Intervall: 15 s
pnt/sensor 100000
Time
T [°C]
SCM Man All 2.0 h
STOP
LS-340hum_Hum_lower @ 0.0000 [°C
Sample Intervall:
13-Jul-2015 17:31:43
to
17-Jul-2015 16:40:32
T_{sample}=20°C
Peltiers: 3V 0.1A→
Peltiers: 5.5V 0.17A→
Peltiers: OFF→
V 0.05A→
Peltiers: 1.57V 0.05A→
99.75%
99.5%
99%
95.5%
48
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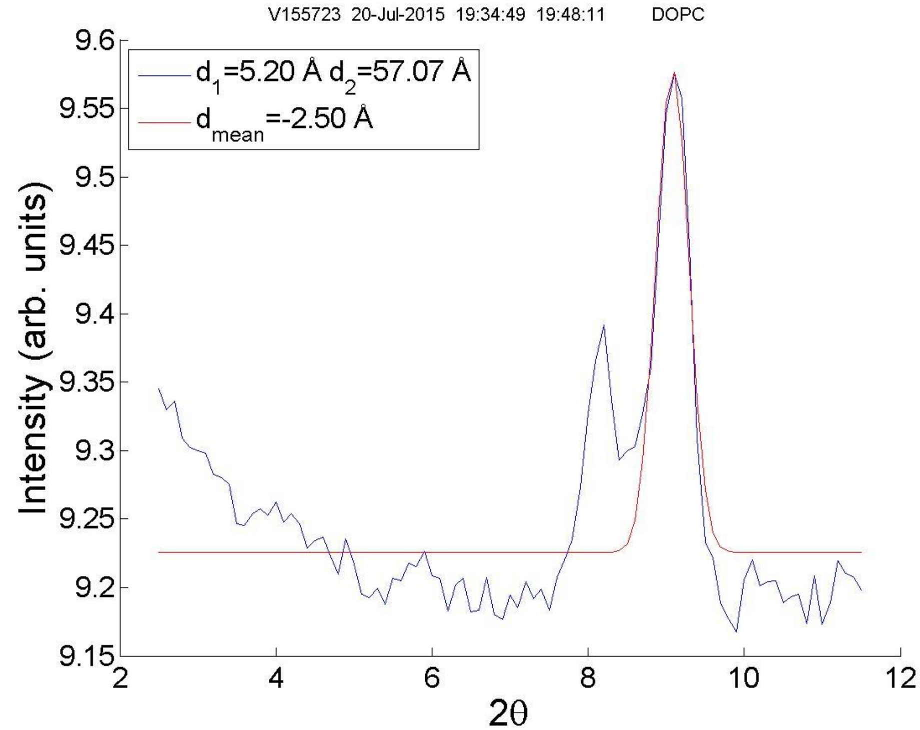
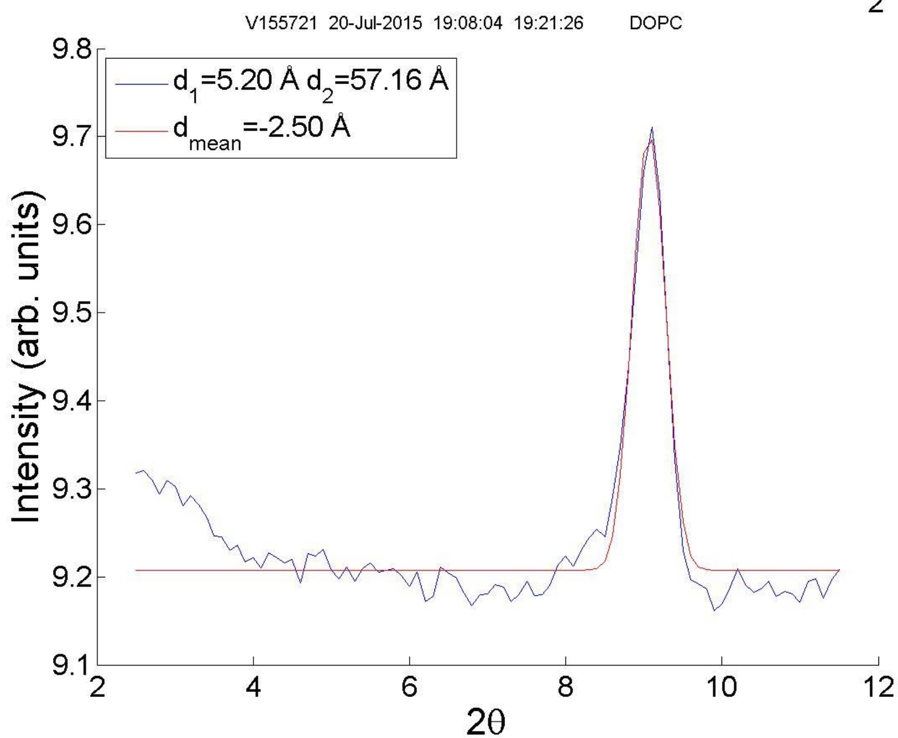
00:00:00
Scan Time


V155723 20-Jul-2015 19:34:49 19:48:11 DOPC
d1=5.20 Å d2=57.07 Å
dmean=-2.50 Å
Intensity (arb. units)
2θ


V155721 20-Jul-2015 19:08:04 19:21:26 DOPC
d1=5.20 Å d2=57.16 Å
dmean=-2.50 Å
Intensity (arb. units)
2θ

DOPC T1=T2=T3= 20 degree Celsius

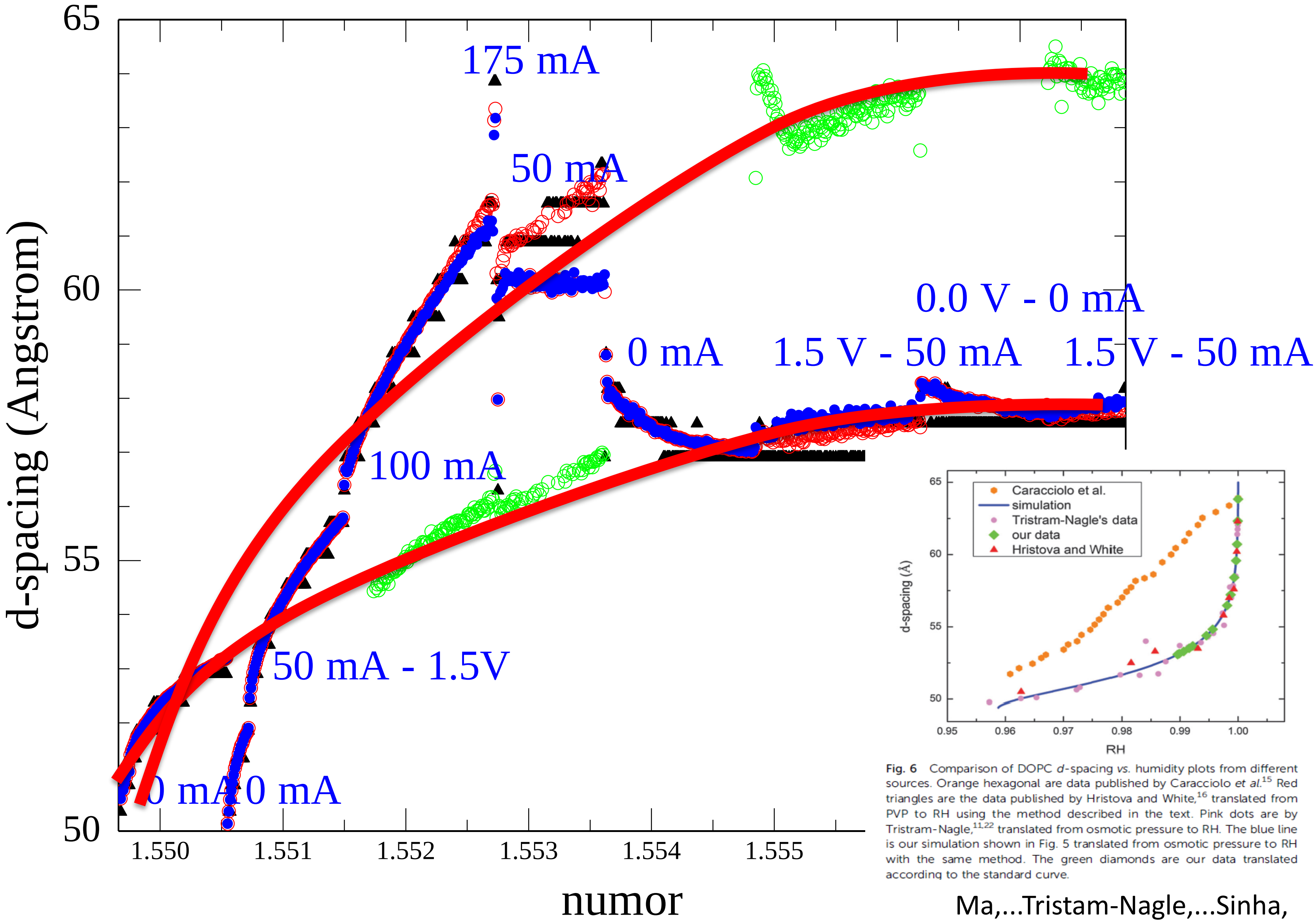
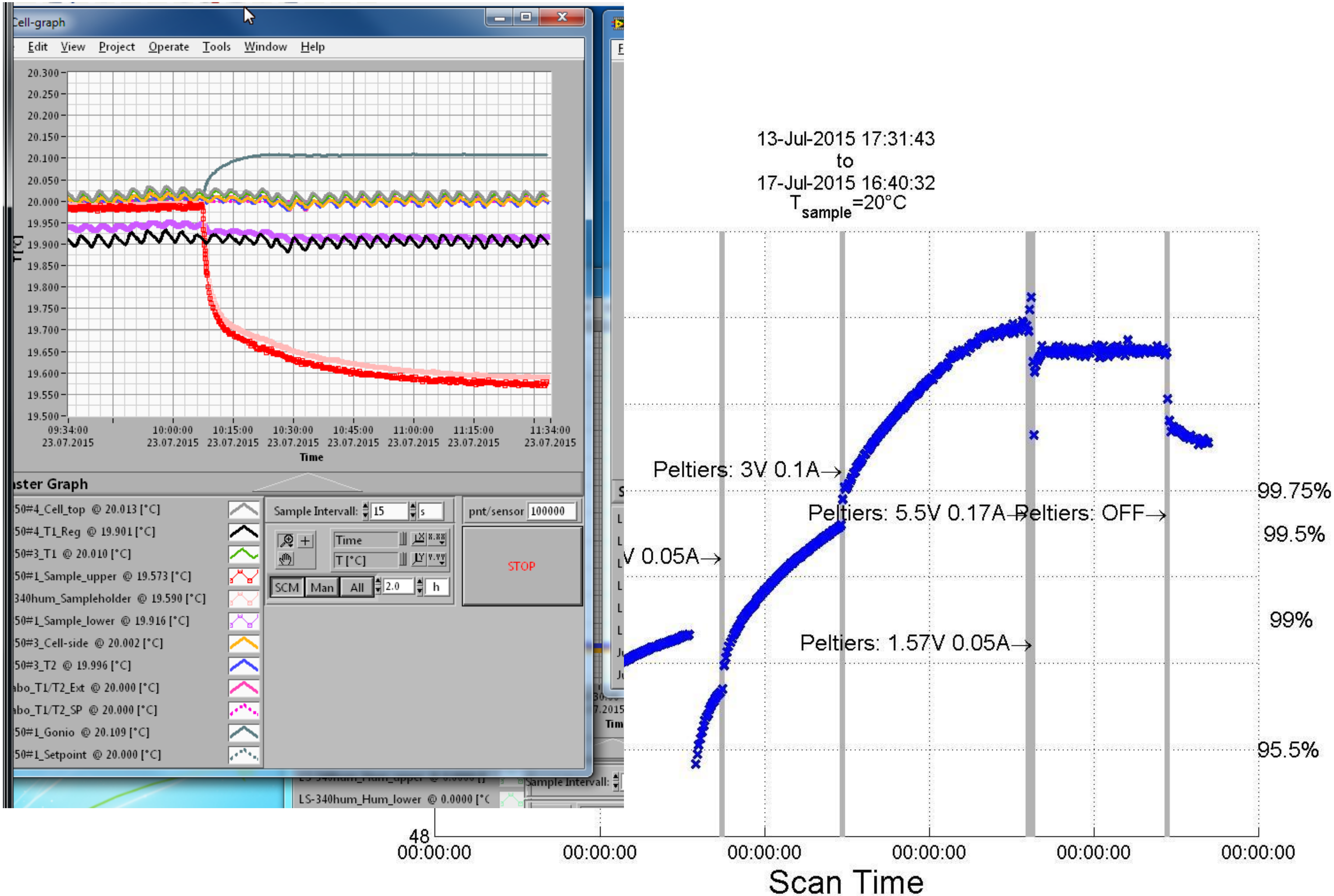


Fig. 6 Comparison of DOPC *d*-spacing *vs.* humidity plots from different sources. Orange hexagonal are data published by Caracciolo *et al.*[15] Red triangles are the data published by Hristova and White,[16] translated from PVP to RH using the method described in the text. Pink dots are by Tristram-Nagle,[11,22] translated from osmotic pressure to RH. The blue line is our simulation shown in Fig. 5 translated from osmotic pressure to RH with the same method. The green diamonds are our data translated according to the standard curve.

Ma,...Tristam-Nagle,...Sinha, PCCP, 2015.

Chamber times:

- Sample sees water bath changes within 10 mins
- Dehydrating sample is fast ~2h
- Hydrating takes from ~4h to ∞, depending on target RH, and step distance (no Peltiers)
- Pelter‘s take ~1h to reach T- goal (depending no target)
- Boost sample hydration speed with humidity


Cell-graph
Edit View Project Operate Tools Window Help
20.300
20.250
20.200
20.150
20.100
20.050
20.000
19.950
19.900
19.850
19.800
19.750
19.700
19.650
19.600
19.550
19.500
09:34:00 23.07.2015
10:00:00 23.07.2015
10:15:00 23.07.2015
10:30:00 23.07.2015
10:45:00 23.07.2015
11:00:00 23.07.2015
11:15:00 23.07.2015
11:34:00 23.07.2015
Time
aster Graph
50#4_Cell_top @ 20.013 [°C]
50#4_T1_Reg @ 19.901 [°C]
50#3_T1 @ 20.010 [°C]
50#1_Sample_upper @ 19.573 [°C]
340hum_Sampleholder @ 19.590 [°C]
50#1_Sample_lower @ 19.916 [°C]
50#3_Cell-side @ 20.002 [°C]
50#3_T2 @ 19.996 [°C]
abo_T1/T2_Ext @ 20.000 [°C]
abo_T1/T2_SP @ 20.000 [°C]
50#1_Gonio @ 20.109 [°C]
50#1_Setpoint @ 20.000 [°C]
Sample Intervall: 15 s
pnt/sensor 100000
Time
T [°C]
SCM Man All 2.0 h
STOP
LS-340hum_Hum_lower @ 0.0000 [°C
Sample Intervall:
13-Jul-2015 17:31:43
to
17-Jul-2015 16:40:32
T sample =20°C
Peltiers: 3V 0.1A→
Peltiers: 5.5V 0.17A→
Peltiers: OFF→
V 0.05A→
Peltiers: 1.57V 0.05A→
99.75%
99.5%
99%
95.5%
48
00:00:00
00:00:00
00:00:00
00:00:00
00:00:00
00:00:00
Scan Time

Current state BERILL1.0

- Full 100% hydration achieved (not over entire sample)
- Humidity Sensor gives just estimate of r.H.
- User friendly operation up to 99% r.H. possible

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

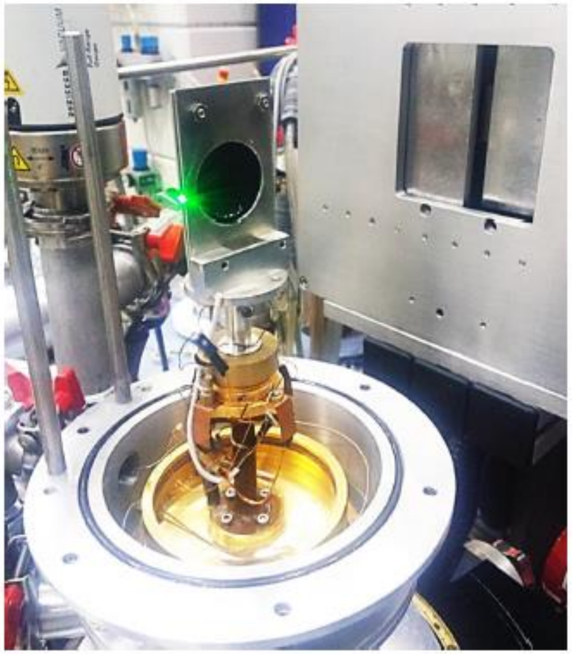
Use of *In-Situ* Small Angle Scattering Techniques to Probe the Dynamic Structure of Graphene-Based Membranes

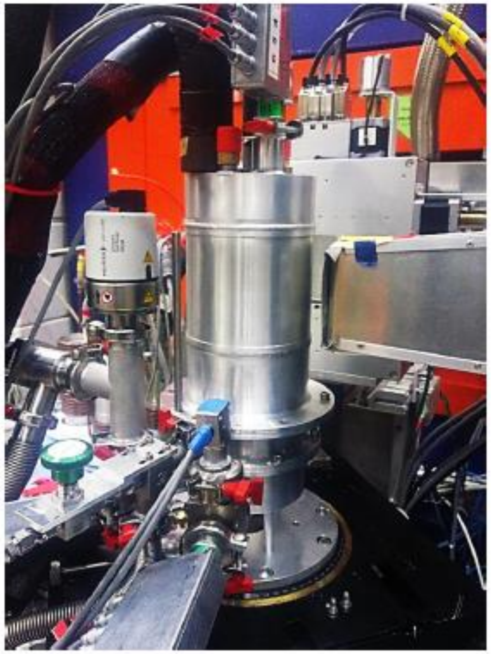
Ashley Roberts

Chris Garvey, Dan Li, George Simon

Neutron Diffraction V1:

Graphene membranes in alumina frame and placed inside humidity chamber





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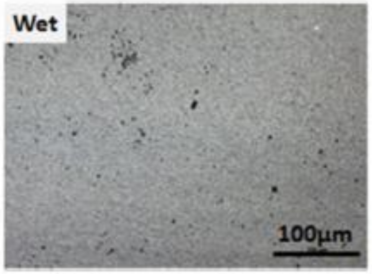
Use of *In-Situ* Small Angle Scattering Techniques to Probe the Dynamic Structure of Graphene-Based Membranes

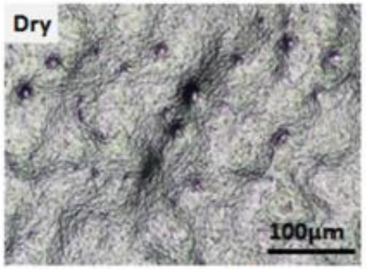
Ashley Roberts

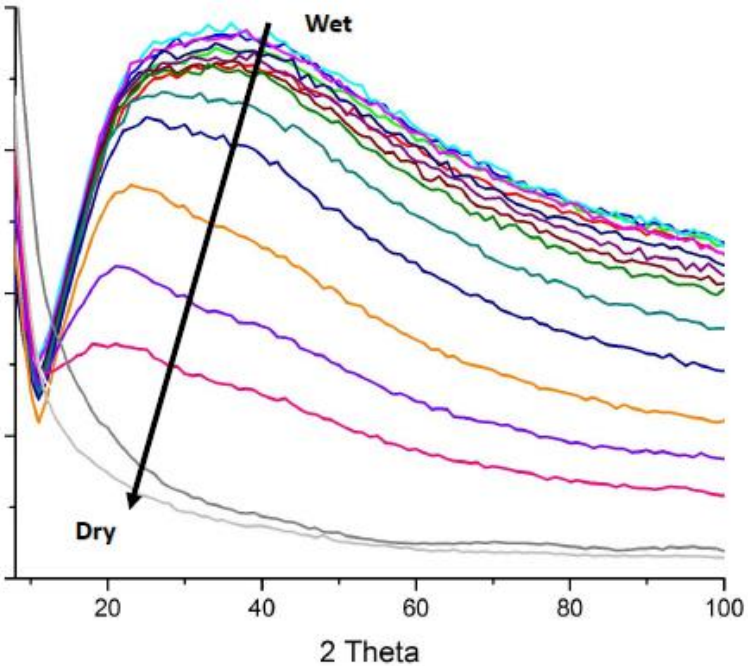
Chris Garvey, Dan Li, George Simon

Neutron Diffraction Results:

Humidity chamber installation on V1







In-situ measurements with the new humidity chamber show the Bragg peak's shape and position are directly related to adsorbed water. These results can be correlated with performance to produce a theory for the role of water in these systems.

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Outlook BERILL2.0

High-perf. chillers

AC200-A28 (borrowed on D33)

Warming power
2000 W

Cooling power
320 W

Int. volume
6 liters

PC200-A40 (new ones)

Warming power
2000 W

Cooling power
900 W

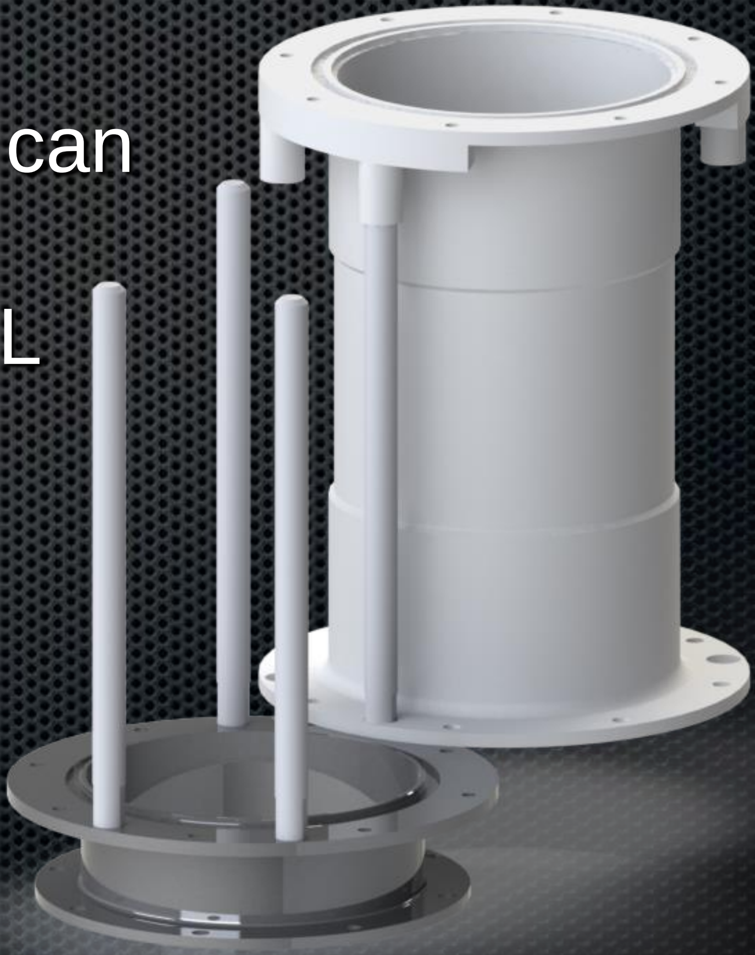
Int. volume
3 liters

Powerful and smaller volume to gain reactivity!

New outside can holder

To avoid breakage of outer can

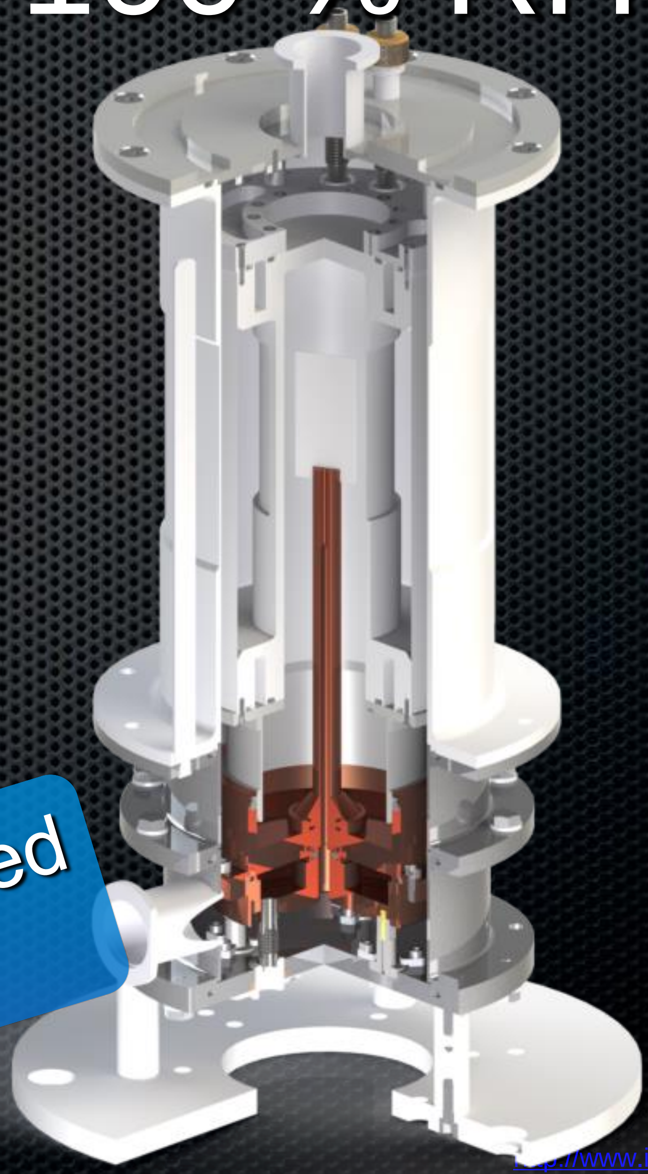
To simplify the use of BerILL



Get the Graal => 100 % RH

Copper sample holder
from the reservoir

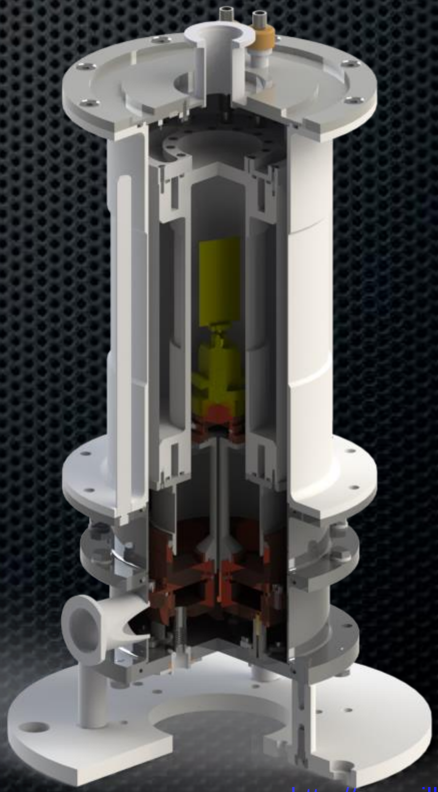
Same temperature
everywhere inside the can



Will be tested
@ HZB

Conclusion & perspectives

- Easy to use / RH controlled automatically
- Very good stability ± 2 mK
- Automatic T. overshoot to speed up
- Further tests on V1 (HZB) in December



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

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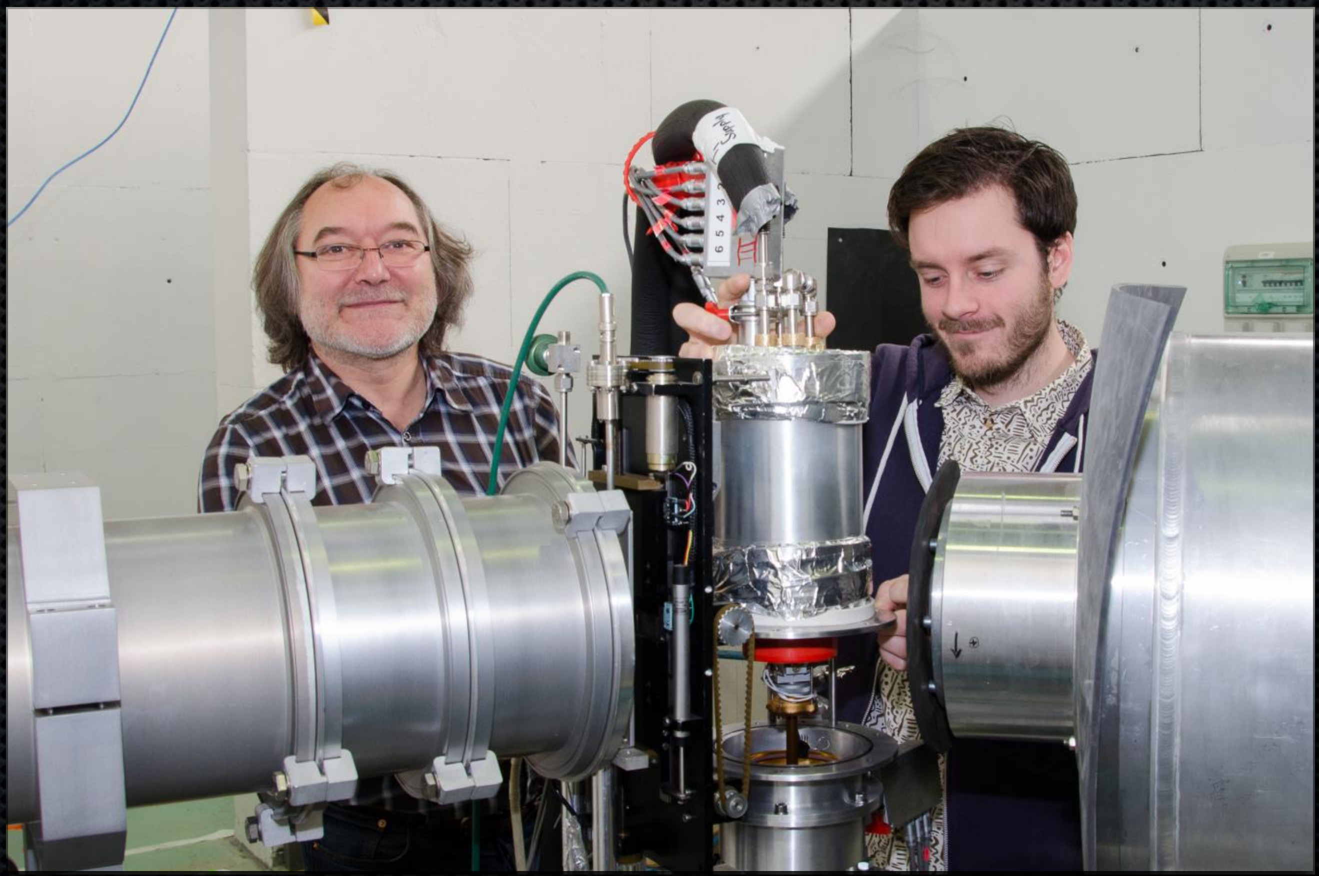
Very special thanks to the HZB team (Dirk, Nico, Matt & Thomas)

Thanks to ILL team too (Adrian, Bruno, Eddy, Eric & Simon)



Thank you for your attention