The next generation humidity chamber for neutron scattering

NMI3-Soft Matter JRA-WP20

Dirk Wallacher, 15.10.2014, Eynsham Hall

NMI3 - Soft Matter JRA – Task 3

Task Leader

HZB Helmholtz Zentrum Berlin

Partners

Institut Laue-Langevin — Neutrons for Science

Jülich Forschungszentrum

Observers

Science & Technology Facilities Council

Technische Universität München

CEA — energie atomique • energies alternatives

McMaster University

In this talk...

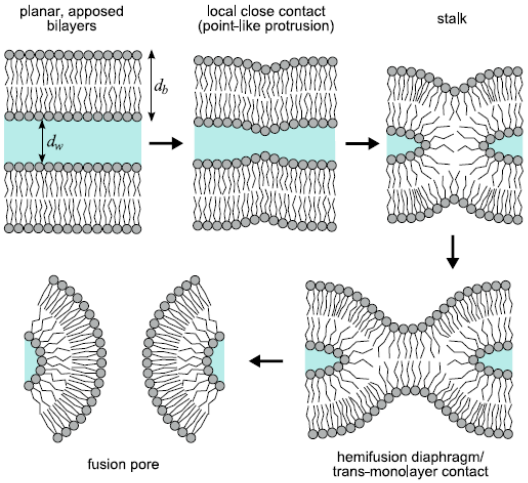
- Motivation
- Project overview and evolution
- First test and results

Motivation

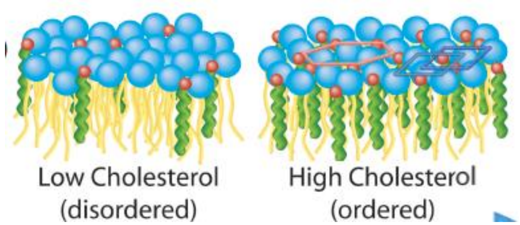
Biological investigations combined with neutron scattering in warm and humid environments

- 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).



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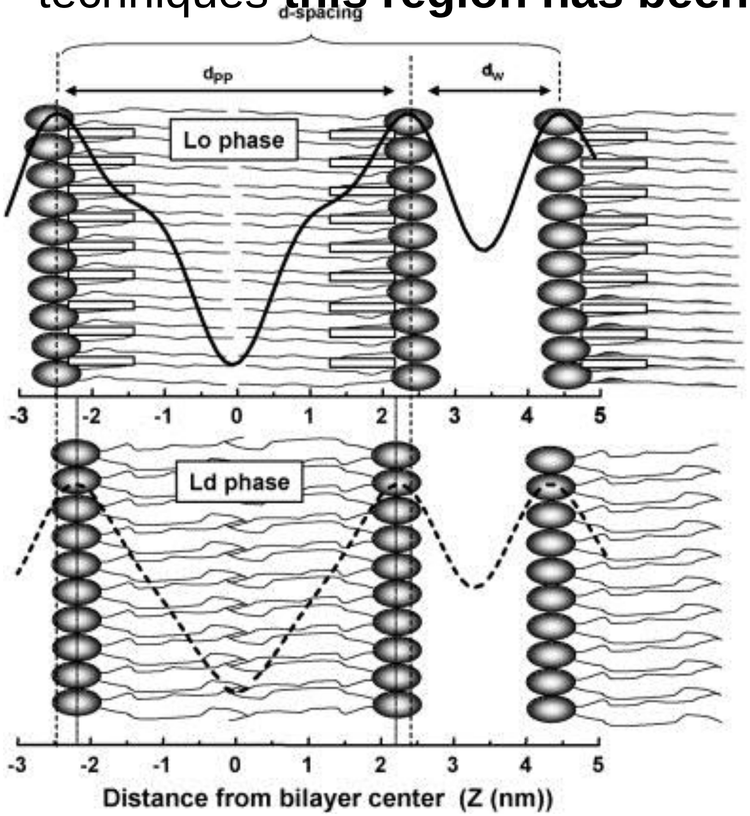
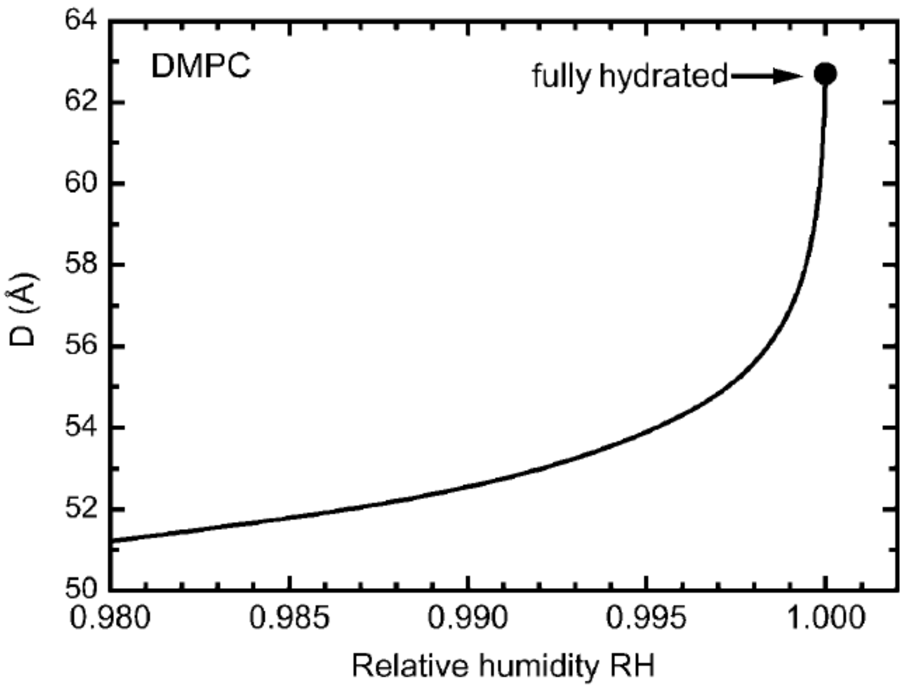
Toward 100% RH

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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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:

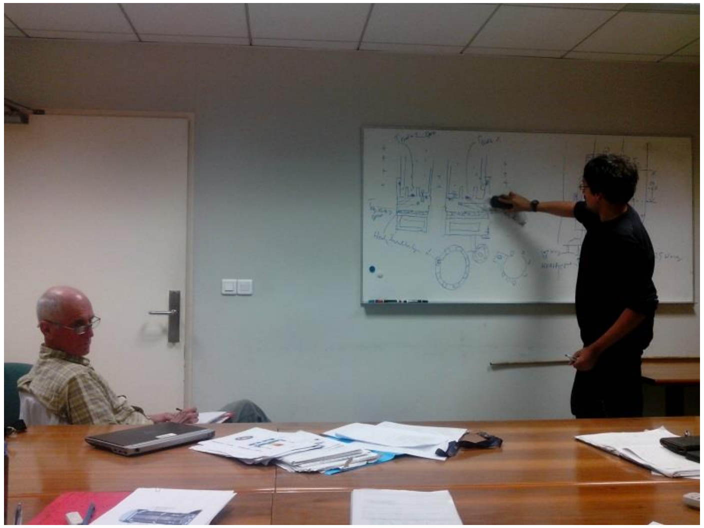
Review the existing systems determine the specifications of the next-generation chambers

Year 2:

Produce drawings

Year 3:

Build and commission chamber



Humidity control techniques

	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%	~0.1% (or better?)	MFCs	minutes	Bubble through mix
Saturated salt solution	98% (discrete steps)	Weak temperature dependence	Syringe pumps (Sat. and distilled)	hours	Change of sample/ flow into reservoir
Temperature controlled water bath	≤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

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Temperature precision

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

For the temperature region from 10°C to 80°C: $\Delta T/T = 1/6 \; \Delta P/P$

0.1% r.H. -> 50mK

Need for high accuracy T sensors!

see poster Nico Grimm

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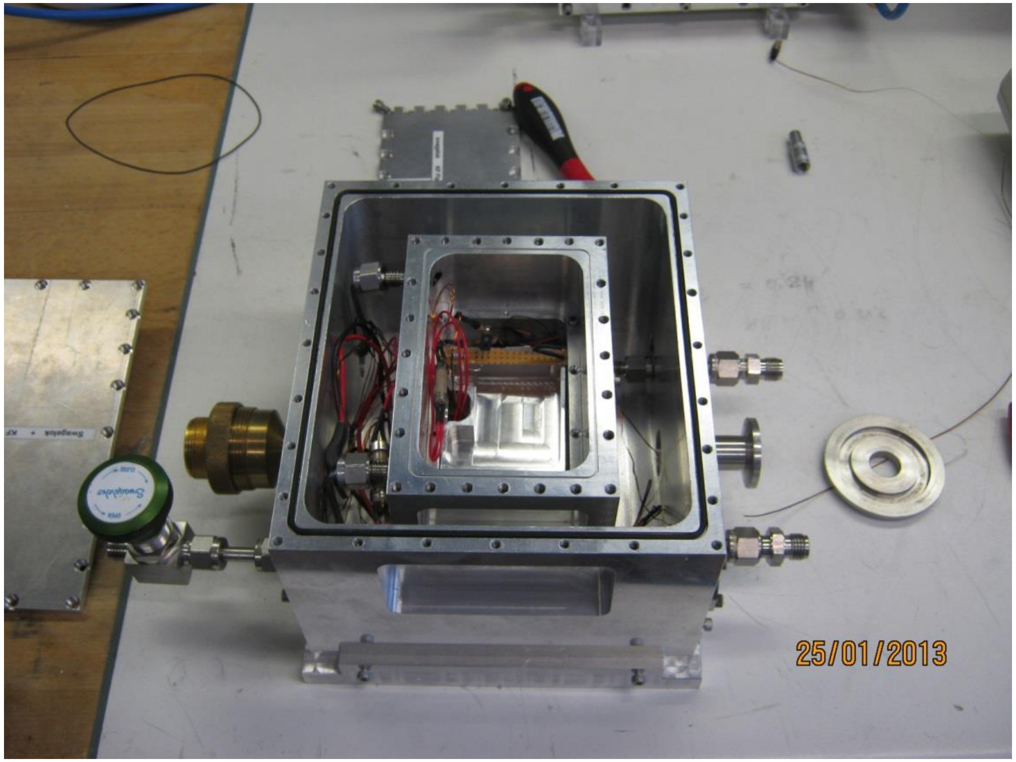
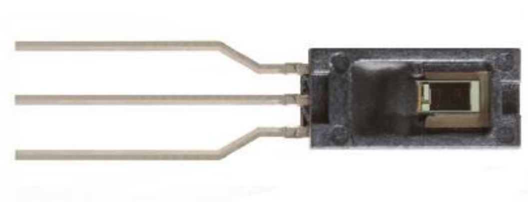
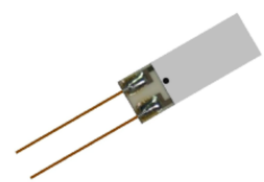
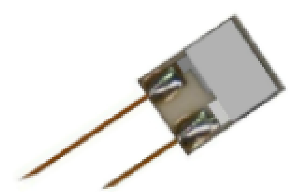
Sensor Testing



5 to test

- HIH 4000 (Honeywell)
- HIH 5030 (Honeywell)
- ChipCap2 (GE)
- MP-33 (Inovative Sensor Technology)
- P-14 (Innovative Sensor Technology)



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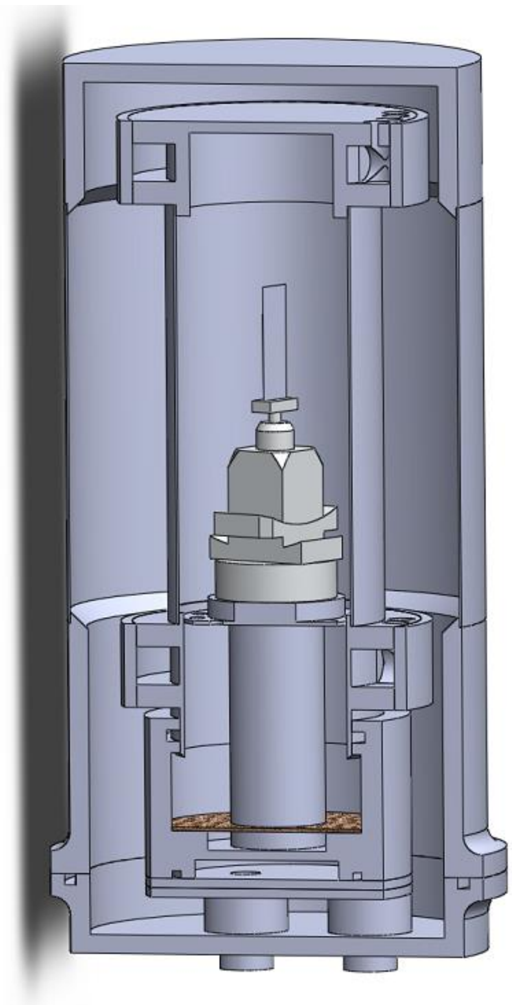
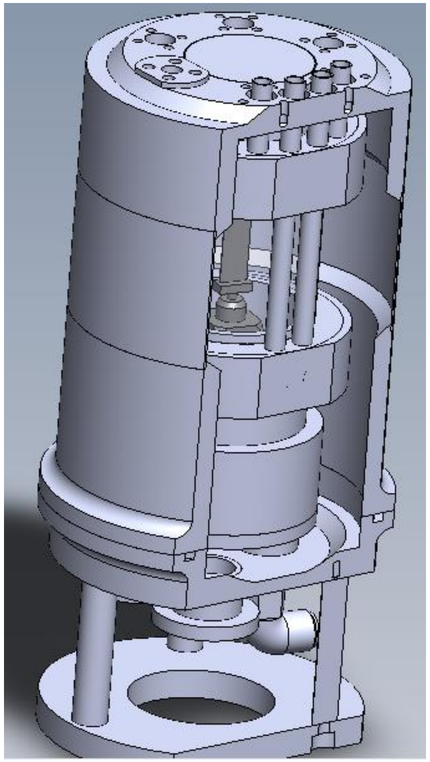
1st suggestions for design

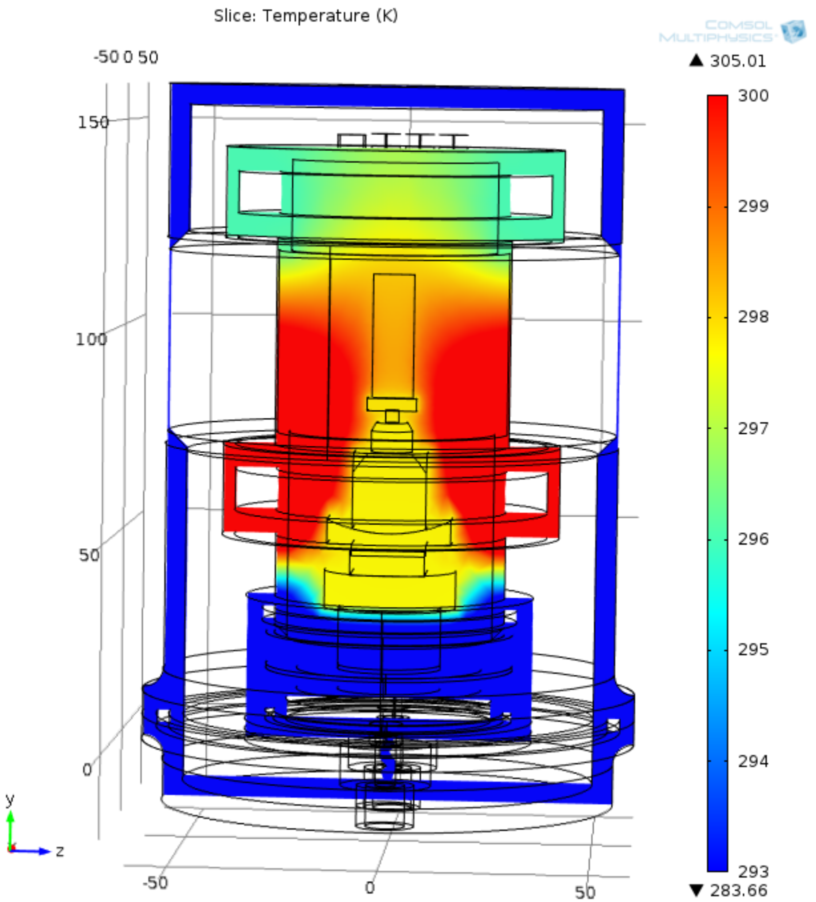


- Double walled (evacuated) Al-cell
- T-control by 3 Chillers
- Total size=240xØ110
- Inner cell=122xØ50
- Wide angular scanning range possible (~300°)



FE Simulations of cell geometry

Variation of materials, geometry and temperature scenarios to minimize temperature inhomogeneity across sample area



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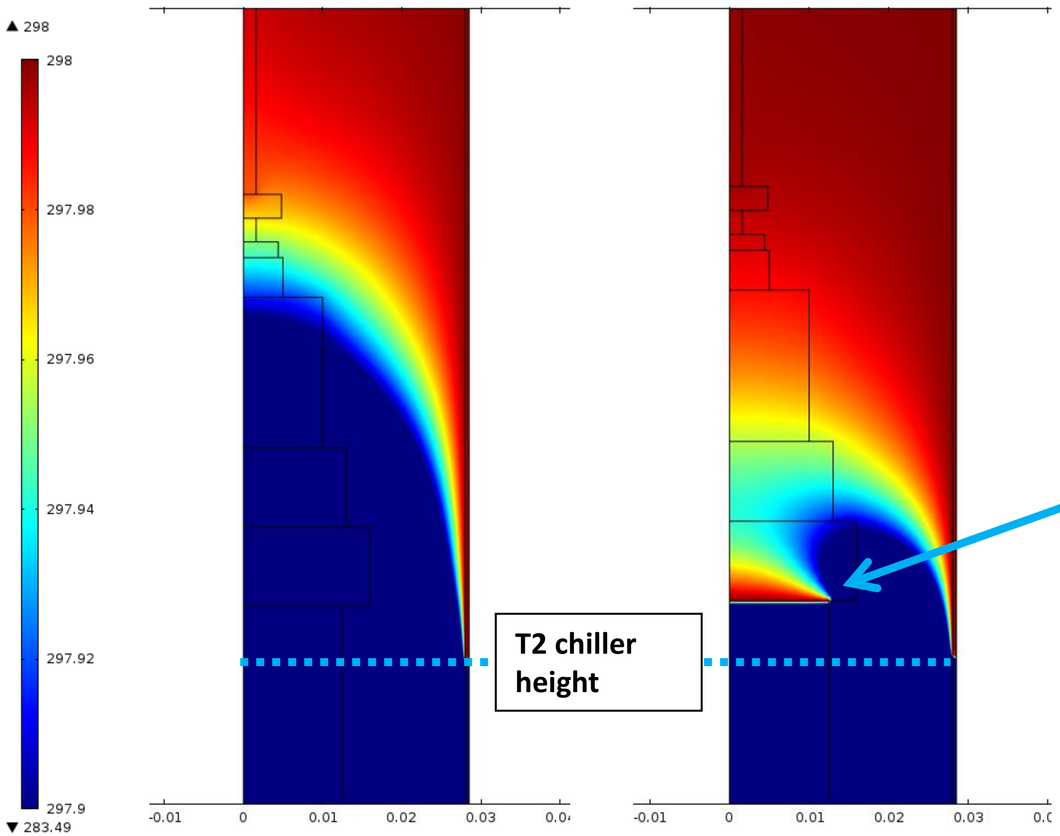
Heater on top of post (T1=T2=T4)

40% (r.h.) study of temperature gradients with and without heater on top of post



No heater **Heater at 298 K**

Suggestion 3: a heater is added between post and gonio

Heater shielding the cold bottom chamber temperature from the goniometer

3D render of the new chamber

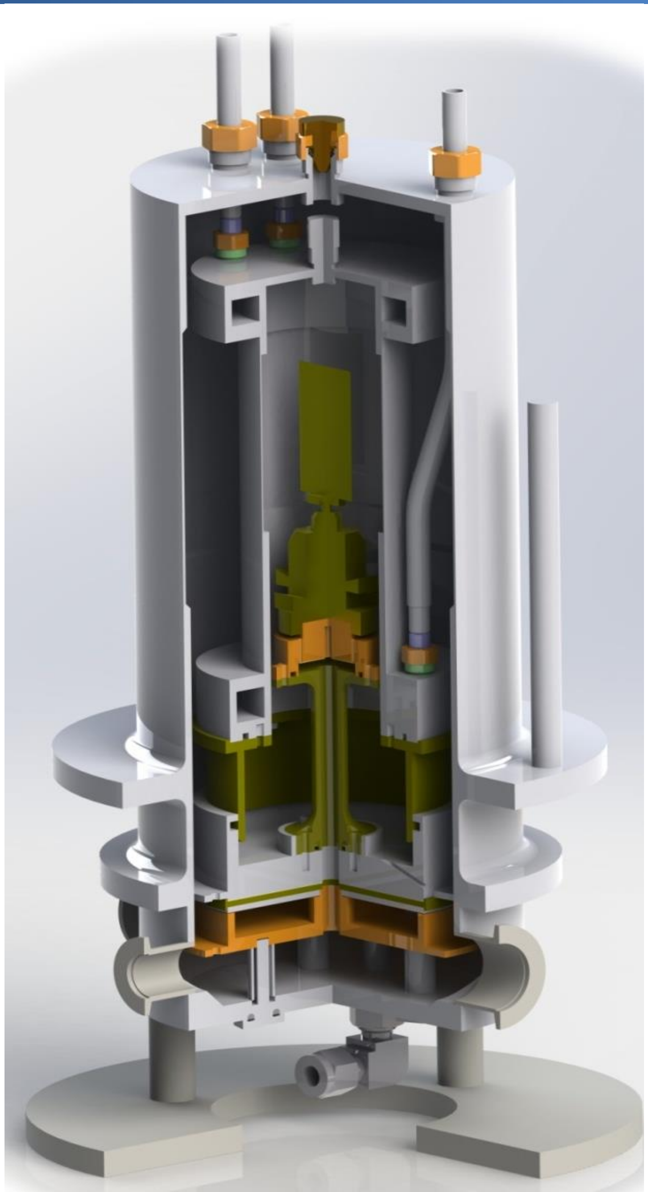
Total height 400 mm, diameter 150 mm

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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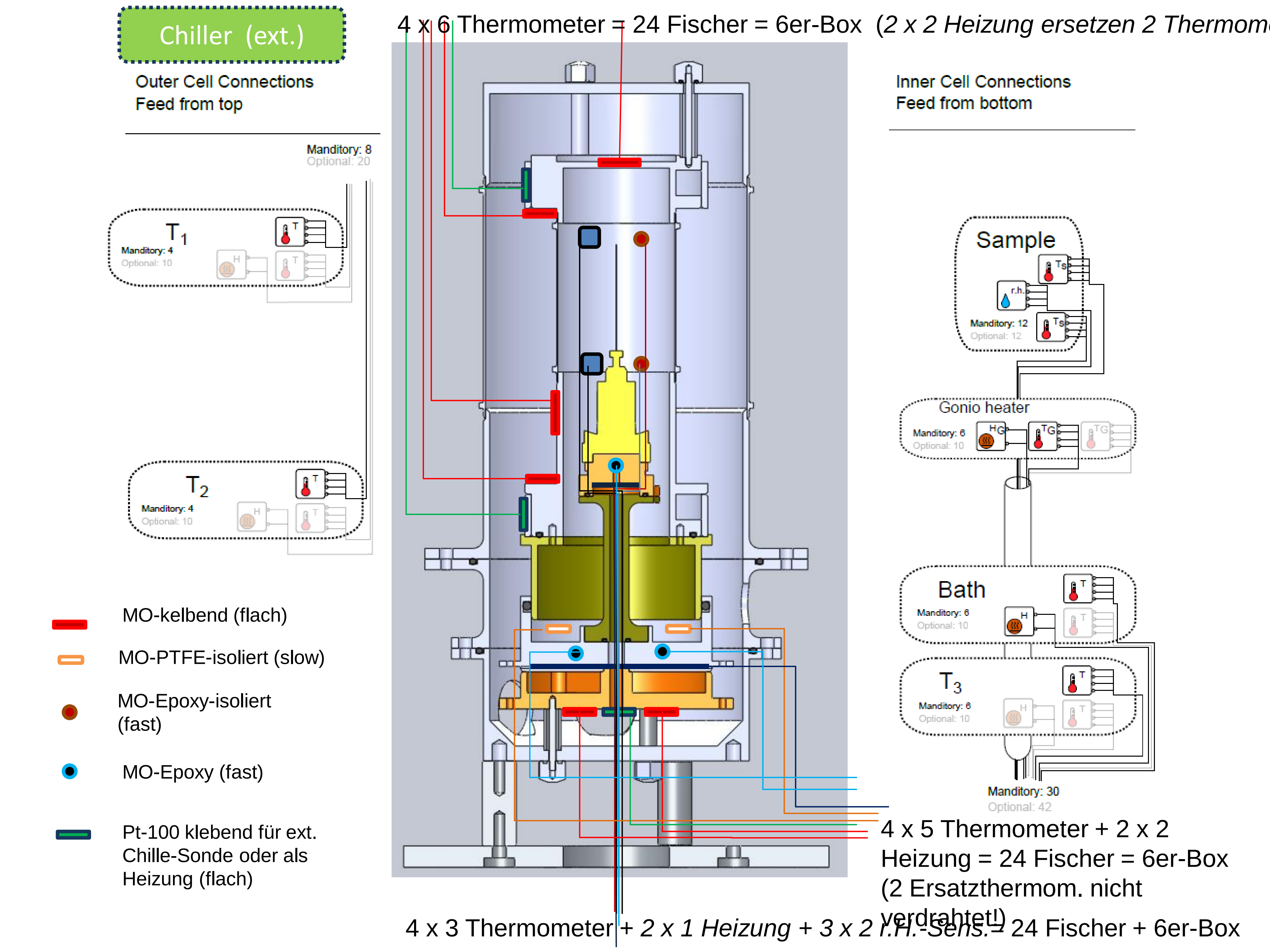
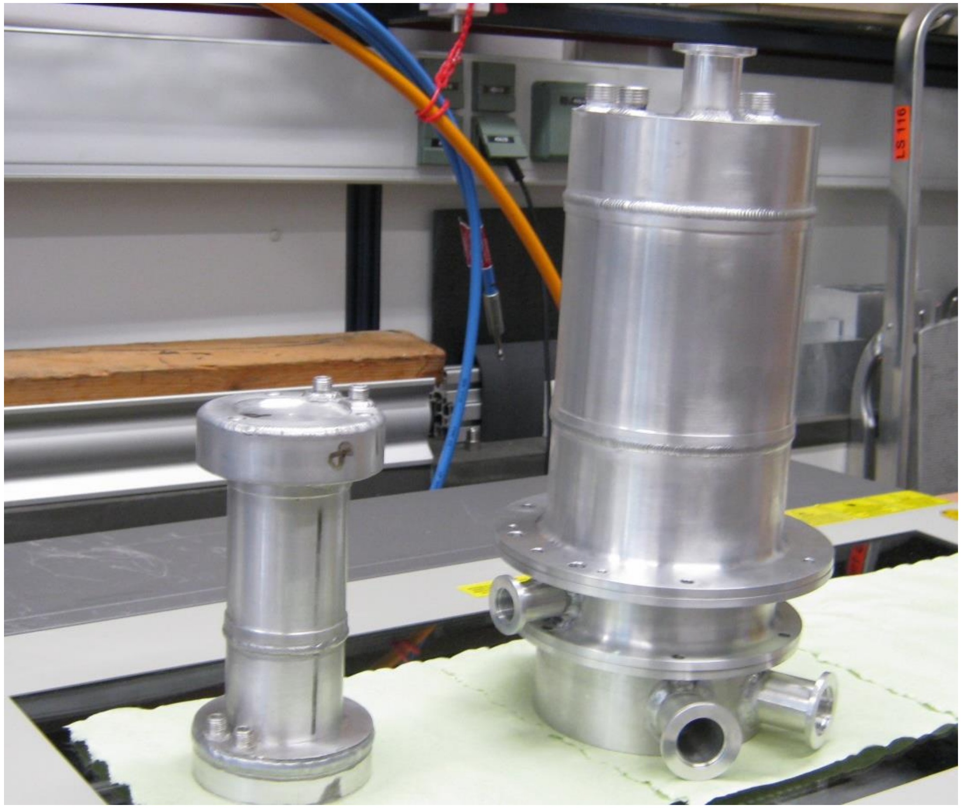


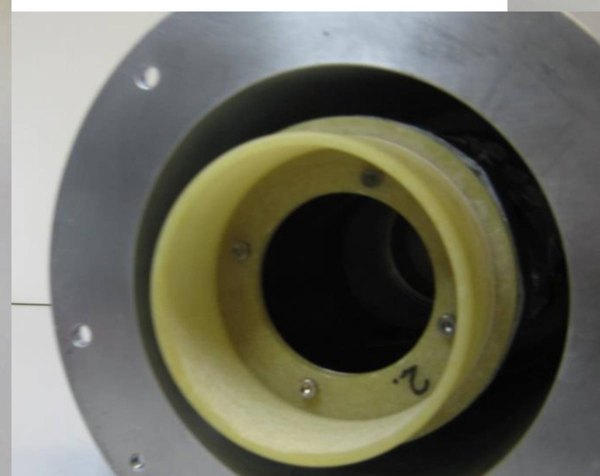
Chiller (ext.)
4 x 6 Thermometer = 24 Fischer = 6er-Box (2 x 2 Heizung ersetzen 2 Thermom
Outer Cell Connections
Feed from top
Inner Cell Connections
Feed from bottom
Manditory: 8
Optional: 20
T1
Manditory: 4
Optional: 10
T2
Manditory: 4
Optional: 10
Sample
Manditory: 12
Optional: 12
r.h.
Gonio heater
Manditory: 6
Optional: 10
Bath
Manditory: 6
Optional: 10
T3
Manditory: 6
Optional: 10
Manditory: 30
Optional: 42
MO-kelbend (flach)
MO-PTFE-isoliert (slow)
MO-Epoxy-isoliert (fast)
MO-Epoxy (fast)
Pt-100 klebend für ext. Chille-Sonde oder als Heizung (flach)
4 x 5 Thermometer + 2 x 2 Heizung = 24 Fischer = 6er-Box (2 Ersatzthermom. nicht verdrahtet!)
4 x 3 Thermometer + 2 x 1 Heizung + 3 x 2 r.H.-Sens. = 24 Fischer + 6er-Box

Assembly of prototype I

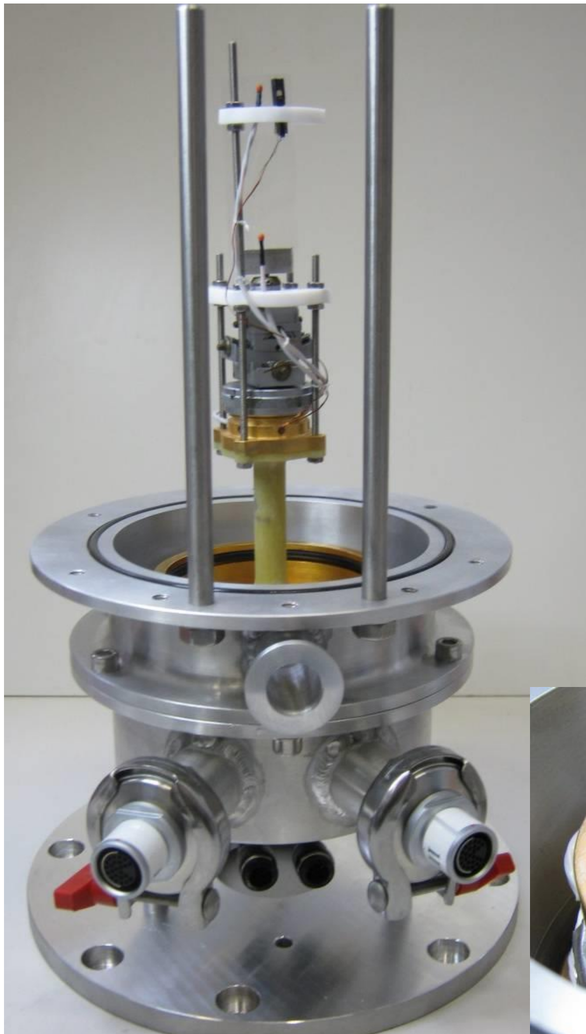
ILL parts + HZB parts

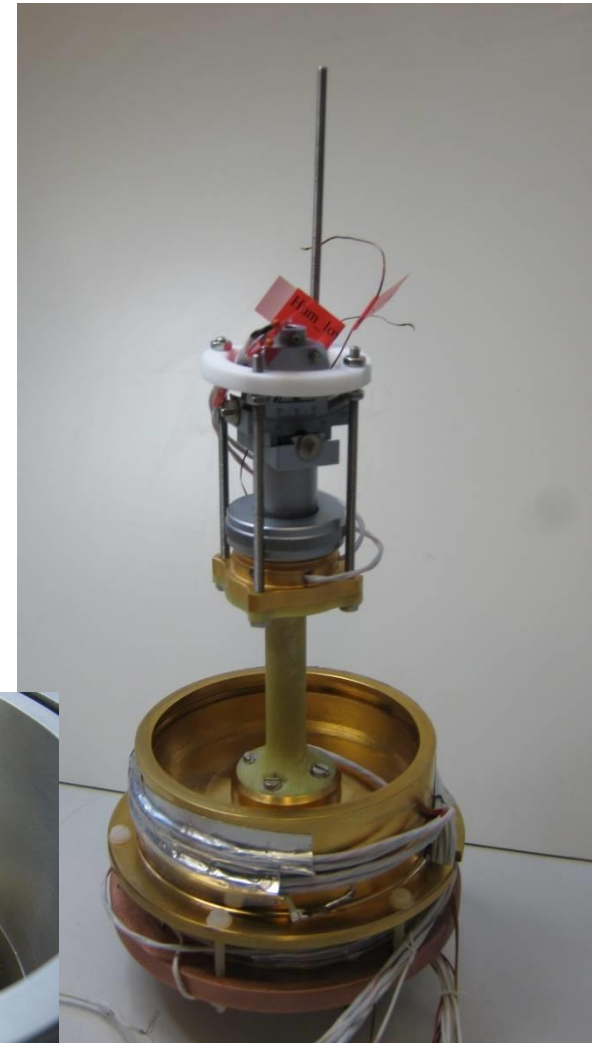


Assembly: top parts

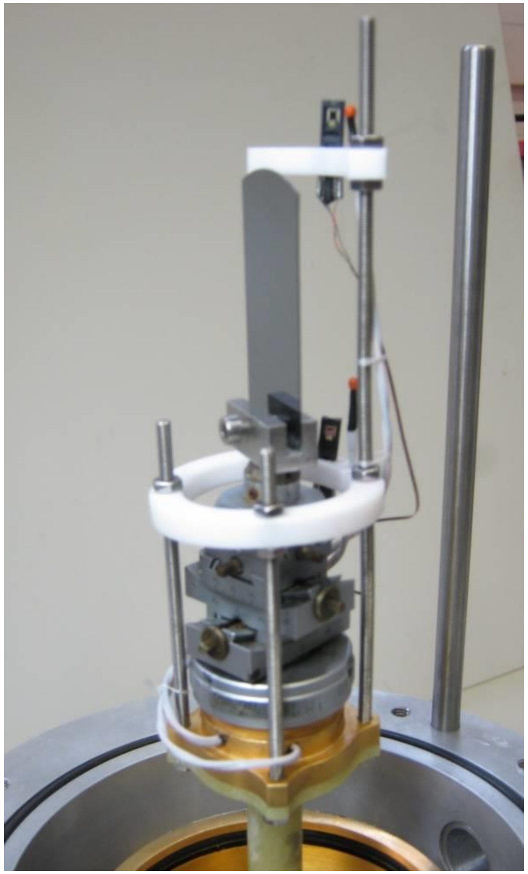


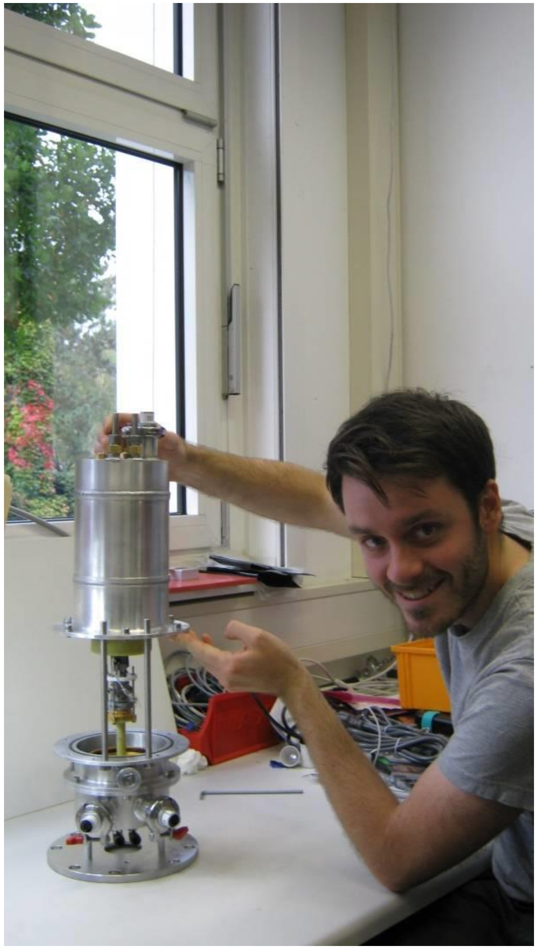
Assembly: bottom parts



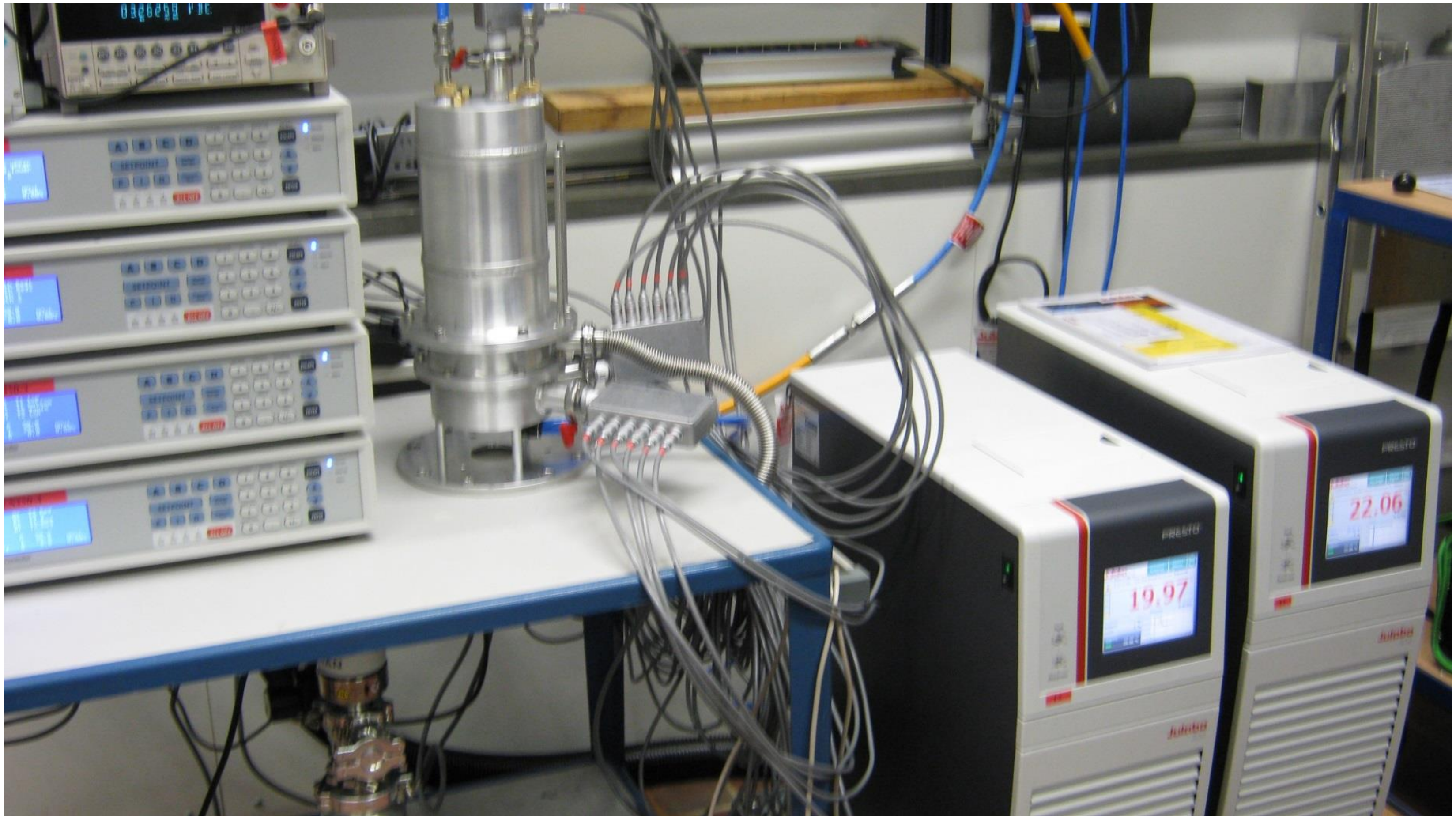


Final Assembly





Complete Setup



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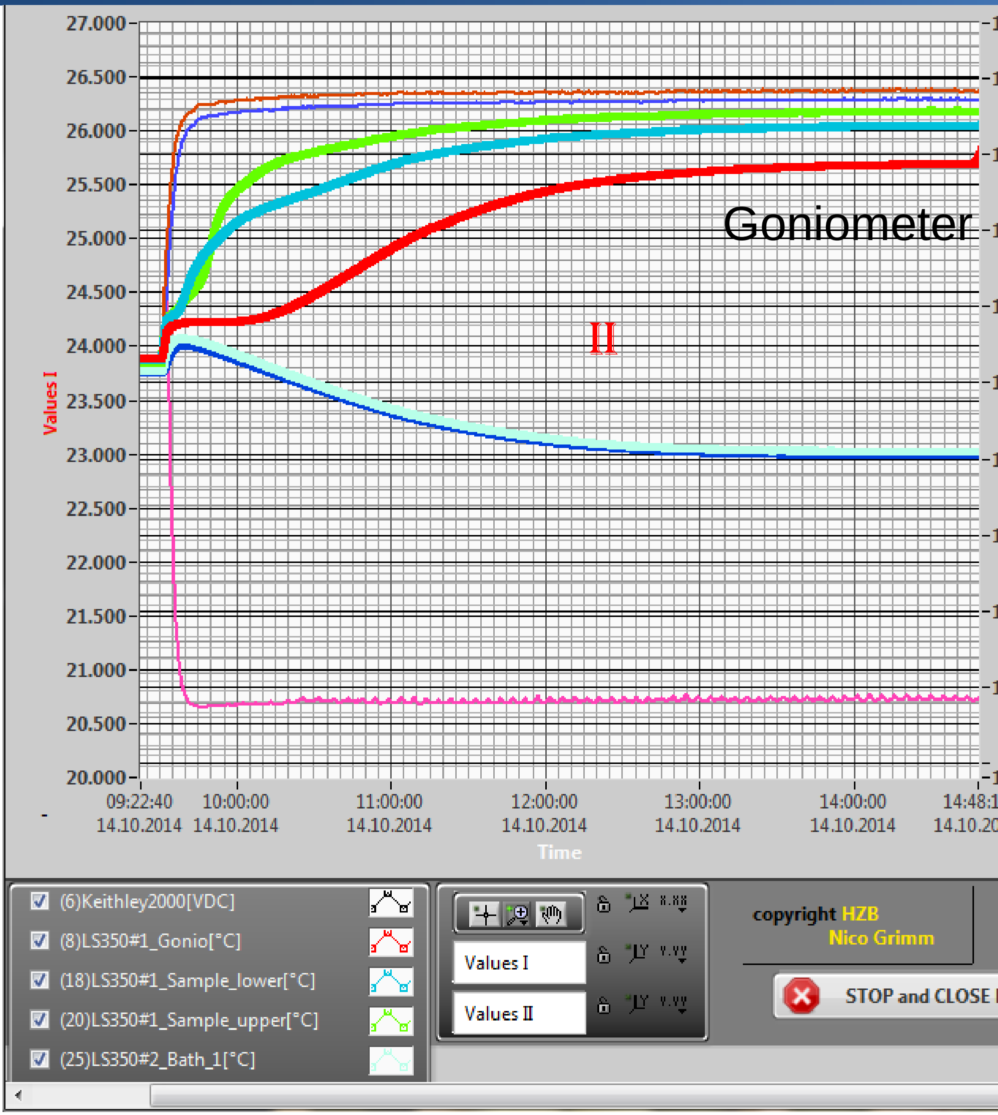
First tests and results



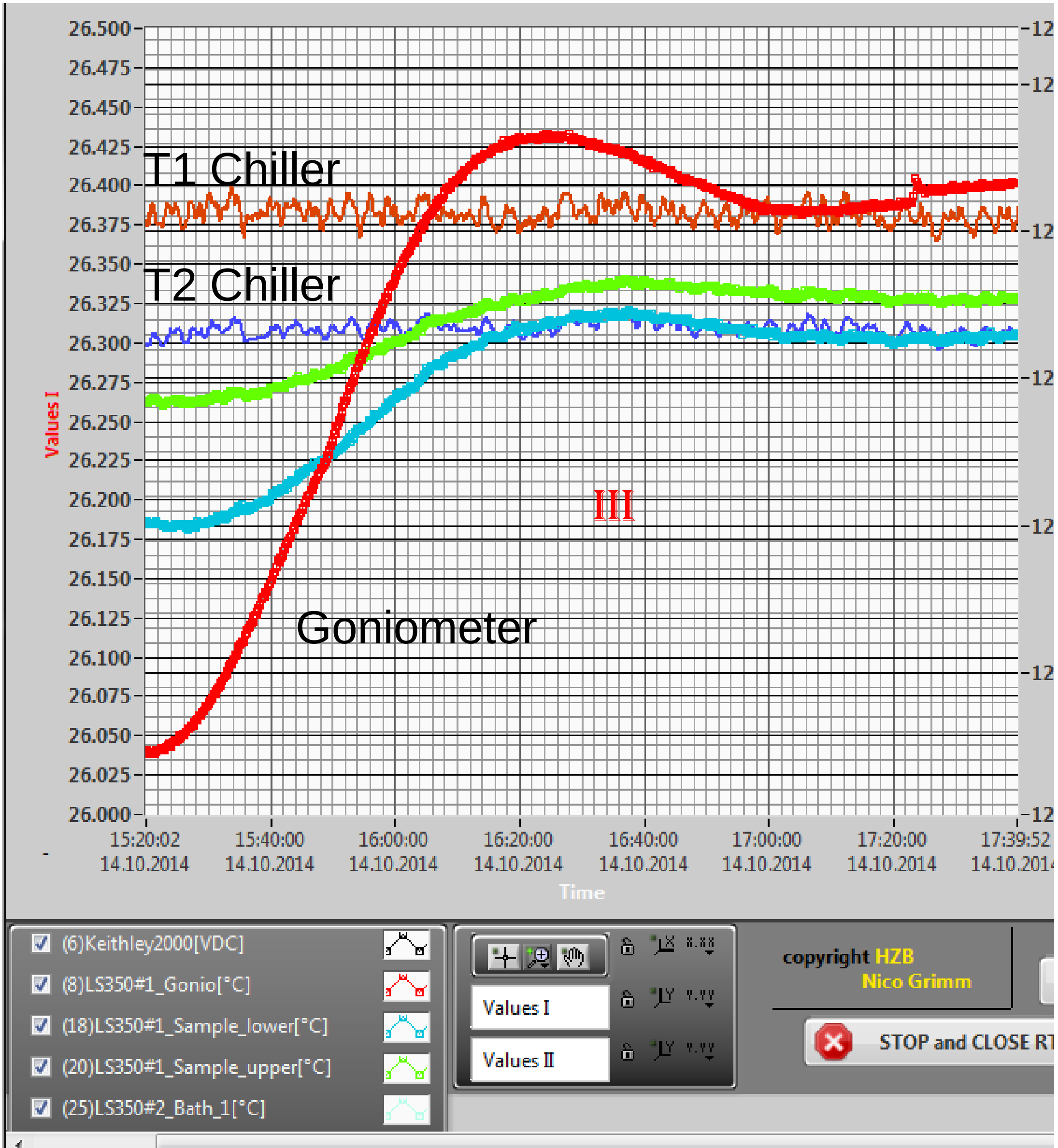
T1 + T 2 Chiller

Sample upper and lower

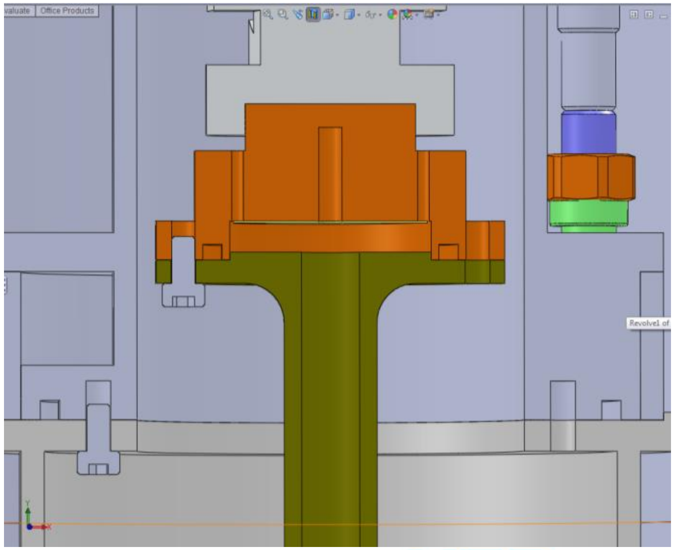
Bath

T3 Chiller

First tests and results



Sample upper and lower



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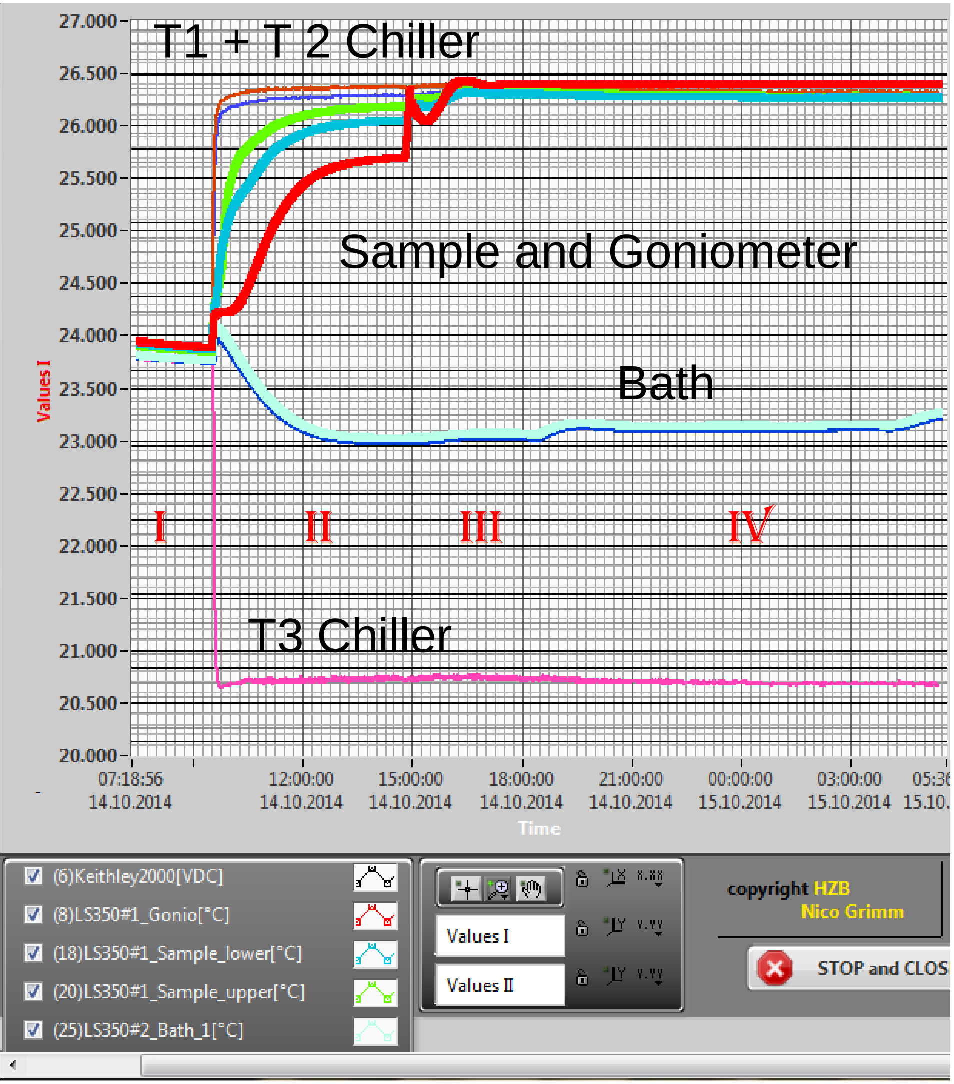
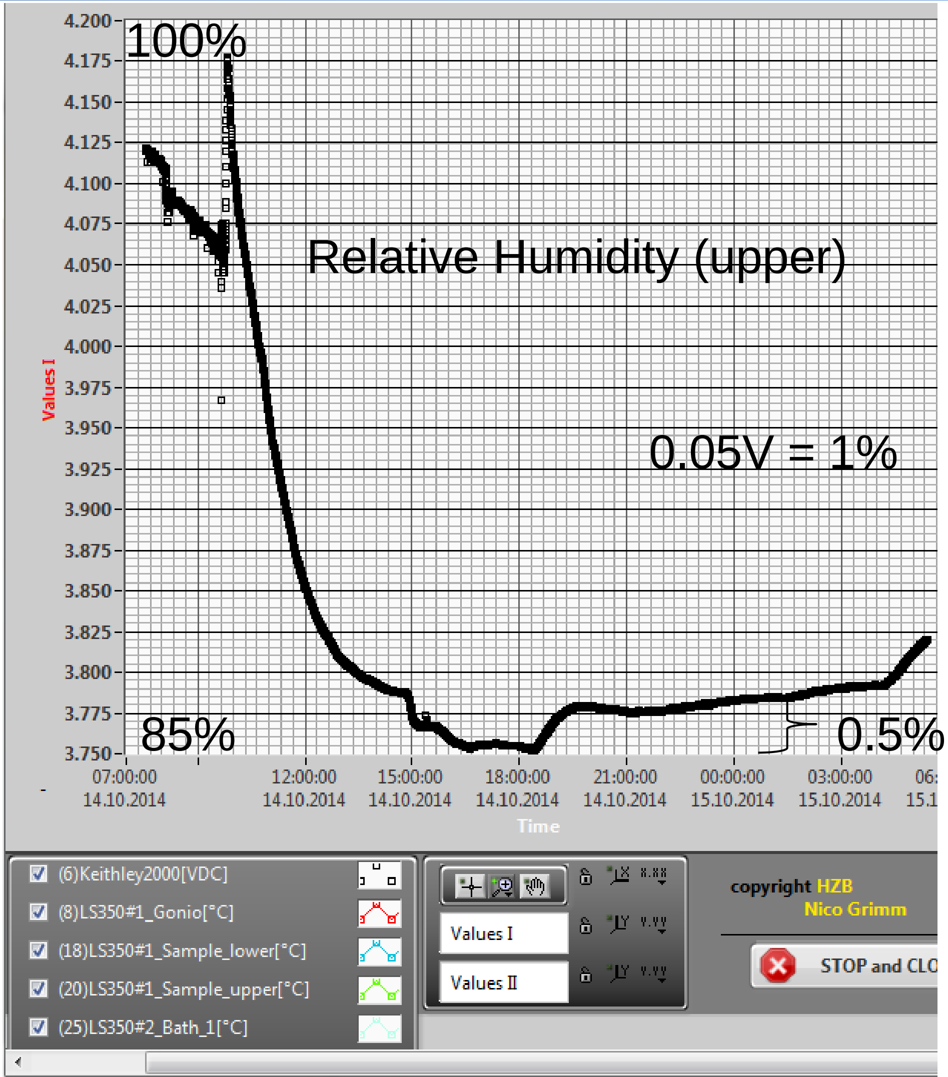
First tests and results





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Summary and next steps

- design, drawings and 1st prototype finished
- commissioning started with promising result

- further tests and improvments of temperature control
- calibration of r.h. sensors
- 1st in-situ test in December 2014 on D16
- assembly of 2nd prototype
- documentation and final report

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Acknowledgments

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

A. Perkins[2], J. Gonthier[2], S. Baudoin[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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