Humidity Chamber Update

16.05.2014 – JRA Meeting

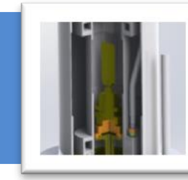
Matt Barrett

Project goal

Develop a humidity chamber which has:

- faster and better controlled temperature and humidity response
- the ability to access large a T and RH range
- adaptability to different neutron instrument geometry
- option for multi-sample holder

Timeline



Year 1:

Review the existing systems determine the specifications of the next-generation chambers (proposal suggested goal of 10 mK stability in T and 0.1% stability in RH)

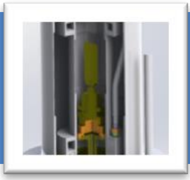
Year 2:

Produce drawings

Year 3:

Build and commission chamber

Participants



Task Leader

HZB Helmholtz Zentrum Berlin

Partners

ILL Neutrons for Science® Institut Laue-Langevin

Jülich Forschungszentrum

Observers

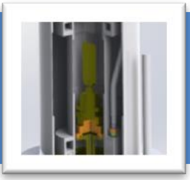
Science & Technology Facilities Council

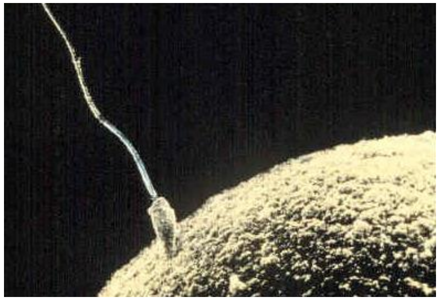
TUM Technische Universität München

cea energie atomique • energies alternatives

McMaster University

Biological studies with Humidity

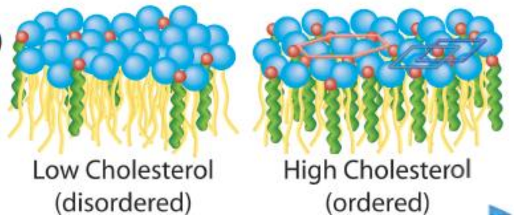




Variable Humidity

– Stalk formation and membrane fusion

– Dehydration protection (sugars)

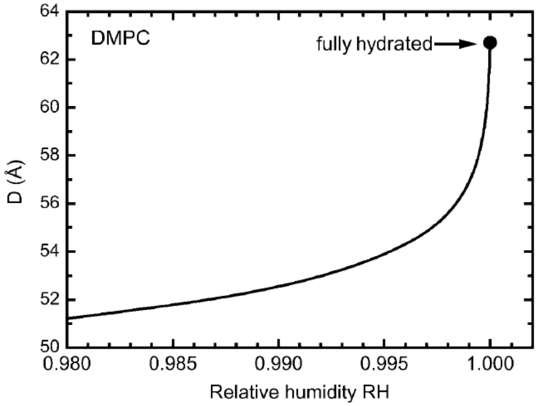


High Humidity (Bio-conditions)

– Lipid rafts

– Response to disease

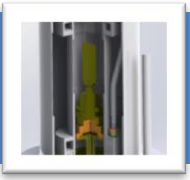
– Function of medications

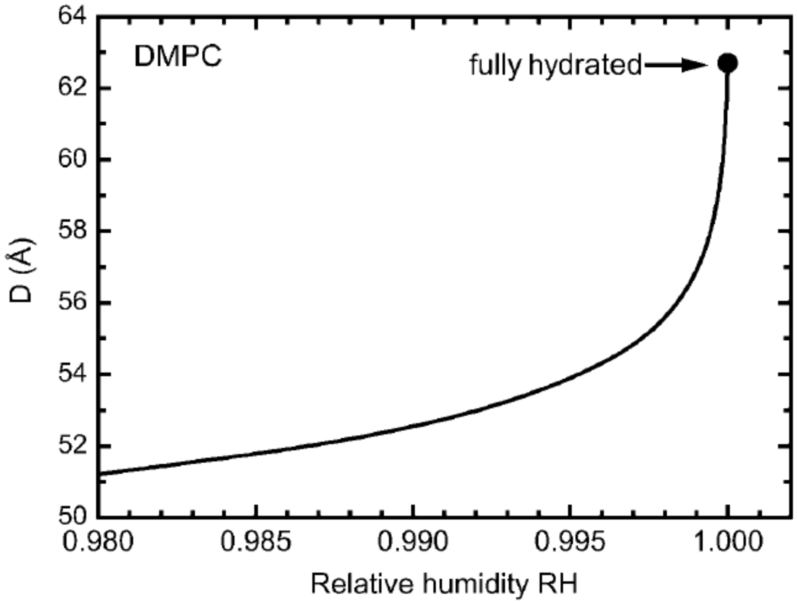


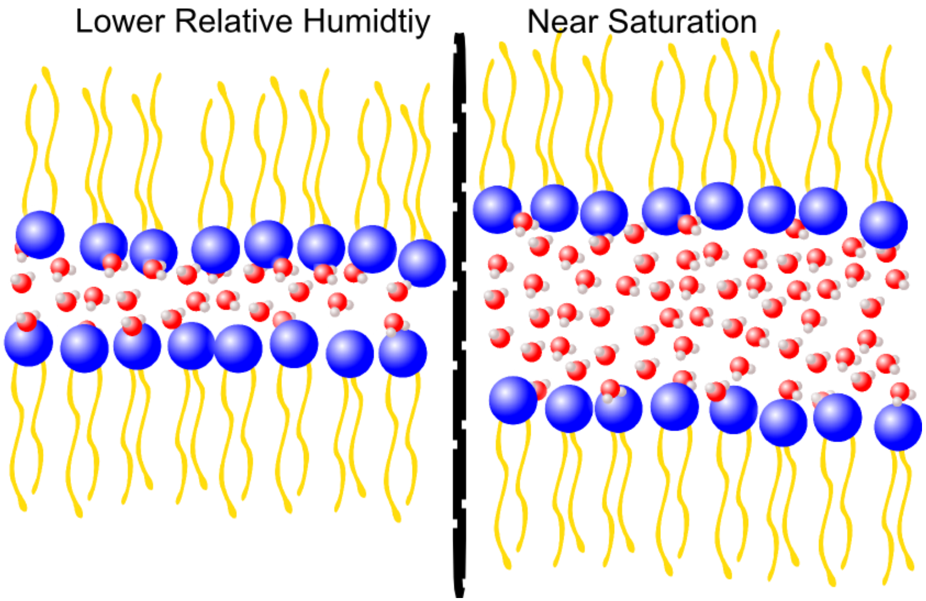
Kučerka, Biophysical Journal, 2005

HZB Helmholtz Zentrum Berlin

Biological studies with Humidity

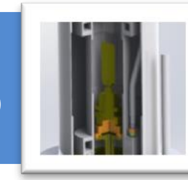






Kučerka, Biophysical Journal, 2005

Humidity control techniques



Saturated salt solution

Figure 4. Saturated salt humidity chamber. Hauß, V1, HZB.

- ✓ precise and reliable (tables available)
- ✓ no calibration necessary
- ✗ discrete humidity steps
- ✗ slow equilibration times

Gas vapour flow

- ✓ continuous humidity range possible
- ✓ automated humidity change (with mass flow controllers)

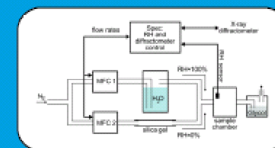


Figure 5. Humidity control setup Salditt, IRP.[a]

- ✓ fast equilibration time
- ✗ upper limit of humidity ~95%
- ✗ temperature gradients in cell or tubing could cause condensation

Bulk water

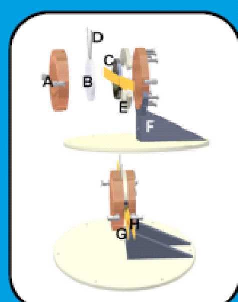


Figure 6. Reflectometry bulk water cell. Harroun, CINS.[c]

- ✓ 100% relative humidity achievable
- ✓ quick deuterium contrast change in-situ
- ✗ sample loss to bulk solution (charged lipids)
- ✗ limited to reflectometry

Temperature controlled

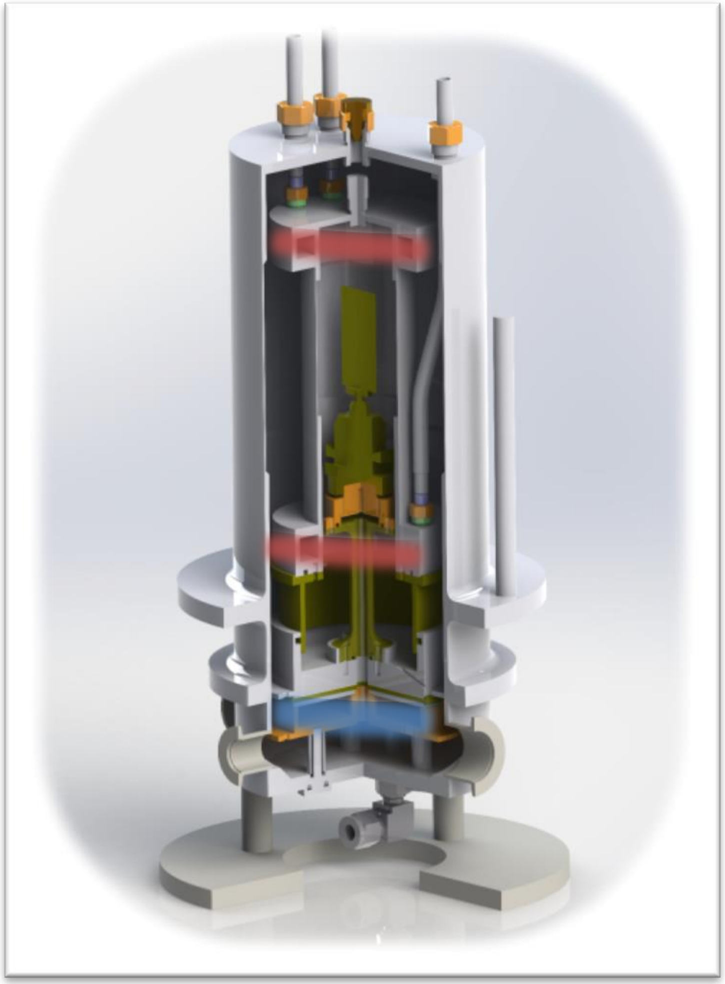
Figure 7. Temp. controlled cell Rheinstädter, McMaster.

Figure 8. Temp. controlled cell. Heinrich, NIST.

- ✓ high (>95% r.h.) possible
- ✓ quick deuterium contrast in-situ
- ✗ temperature gradients (from Peltier or external) lead to condensation
- ✗ difficult to calibrate heaters for desired r.h.

HZB Helmholtz Zentrum Berlin

Working principle

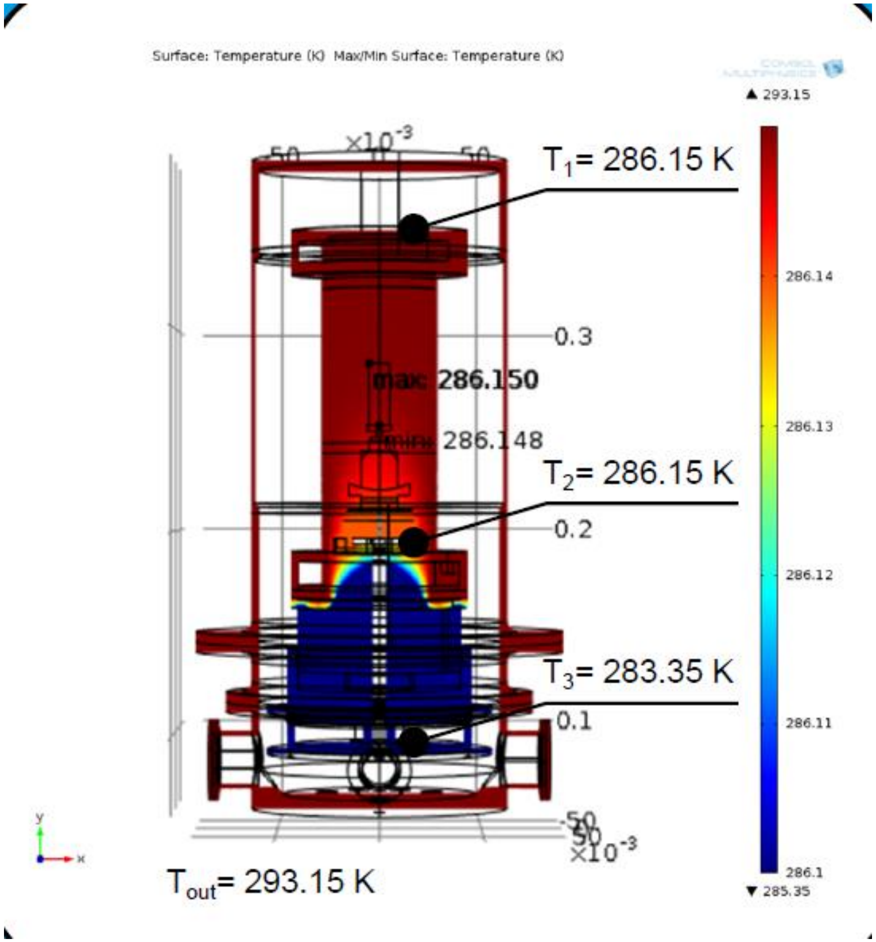


$$r.h. = \frac{partial\ vapour\ pressure}{saturation\ vapour\ pressure}$$

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

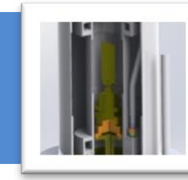
Bridgeman and Aldrich, 1964

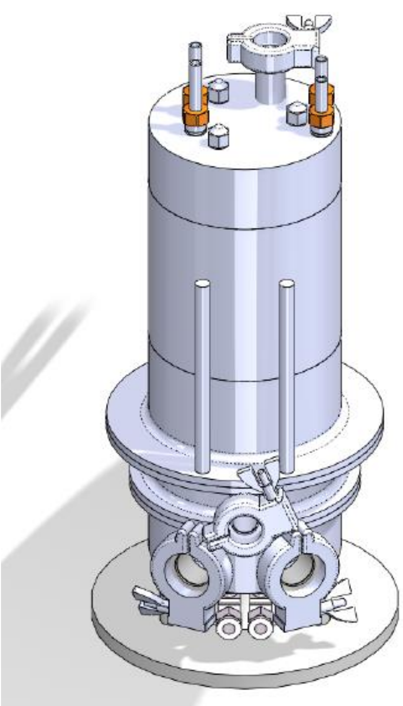
Simulation verification

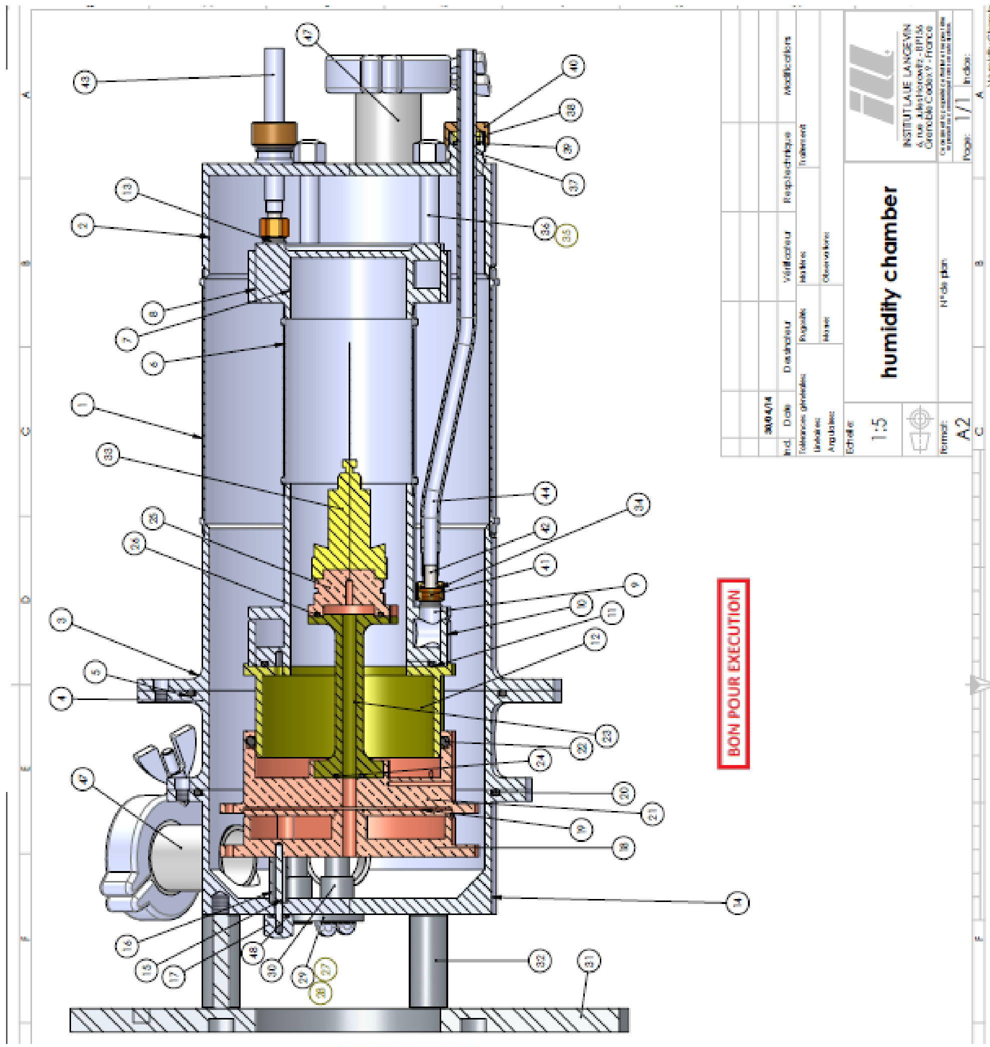


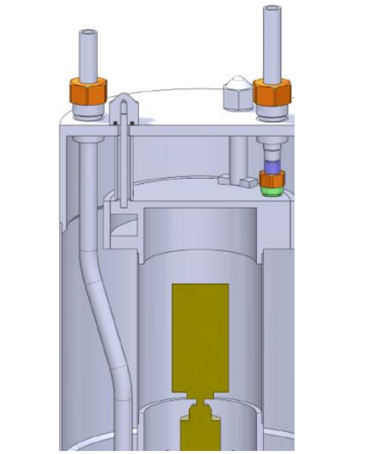
- Extreme temperature control, 0.002°C across our sample limited only by heater accuracy
- ~0.01% gradient in r.h.

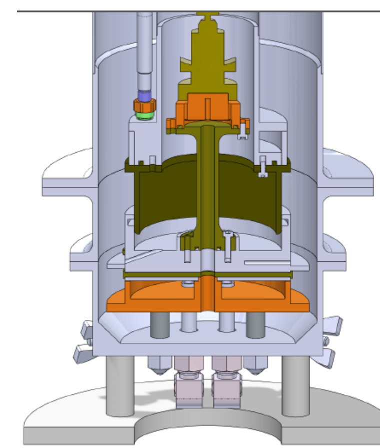
Drawings complete – April 2014





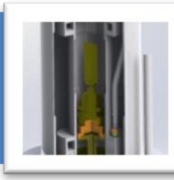






HZB Helmholtz Zentrum Berlin

Prototype construction



1. Outer Can
   a. Can lid
   b. Big tube
   c. Body
   d. Spacer
   e. Can base

Corrosion proof Aluminum

2. Inner can
   a. Top ring
      i. T1
      ii. T1/T2 ring
   b. Bottom ring
      i. T2
      ii. T2 ring
   c. Small tube
   d. **Bath**

Plastic – Delmat Epoxy 68 660

3. Inner components
   a. insulating ring
   b. gonio leg

Copper

4. Inner components
   a. T3
      i. t3
      ii. Plate t3
   b. Gonio copper base

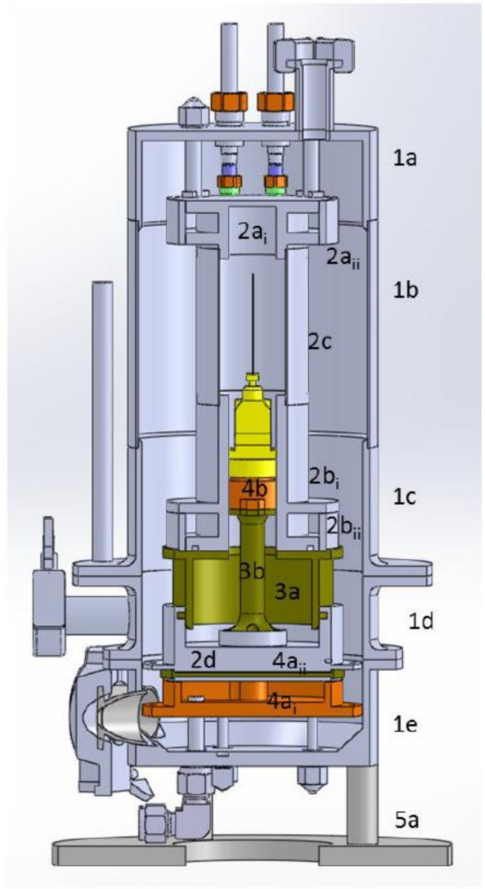
Steel

5. Stand
   a. Base plate

ILL

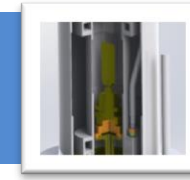
HZB

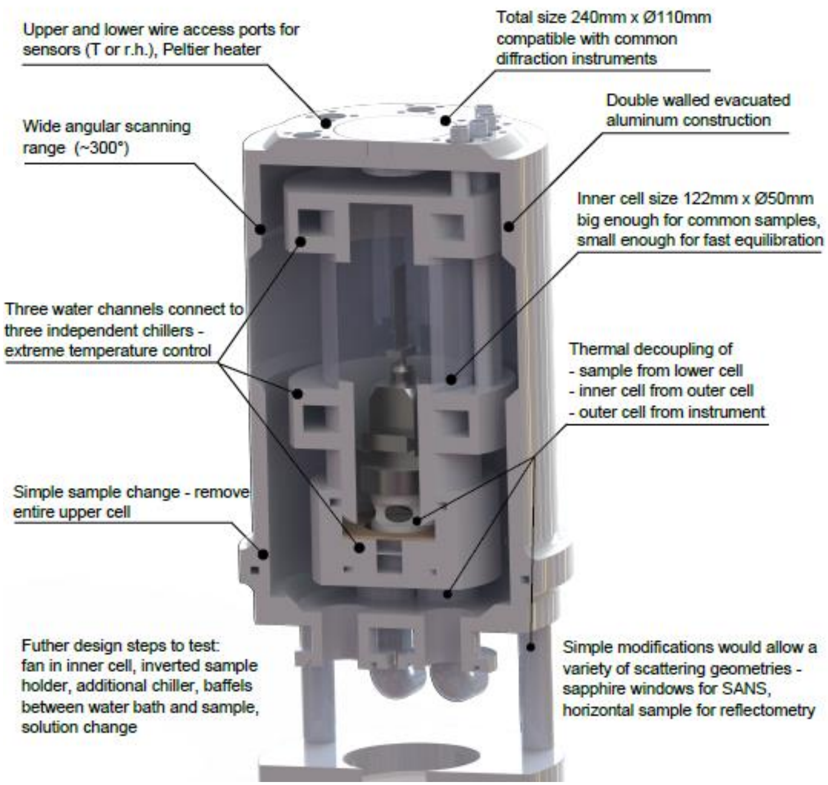
Accessories: Pipes, stilts, guiding posts, connectors (Wilson seals, KF Flanges)

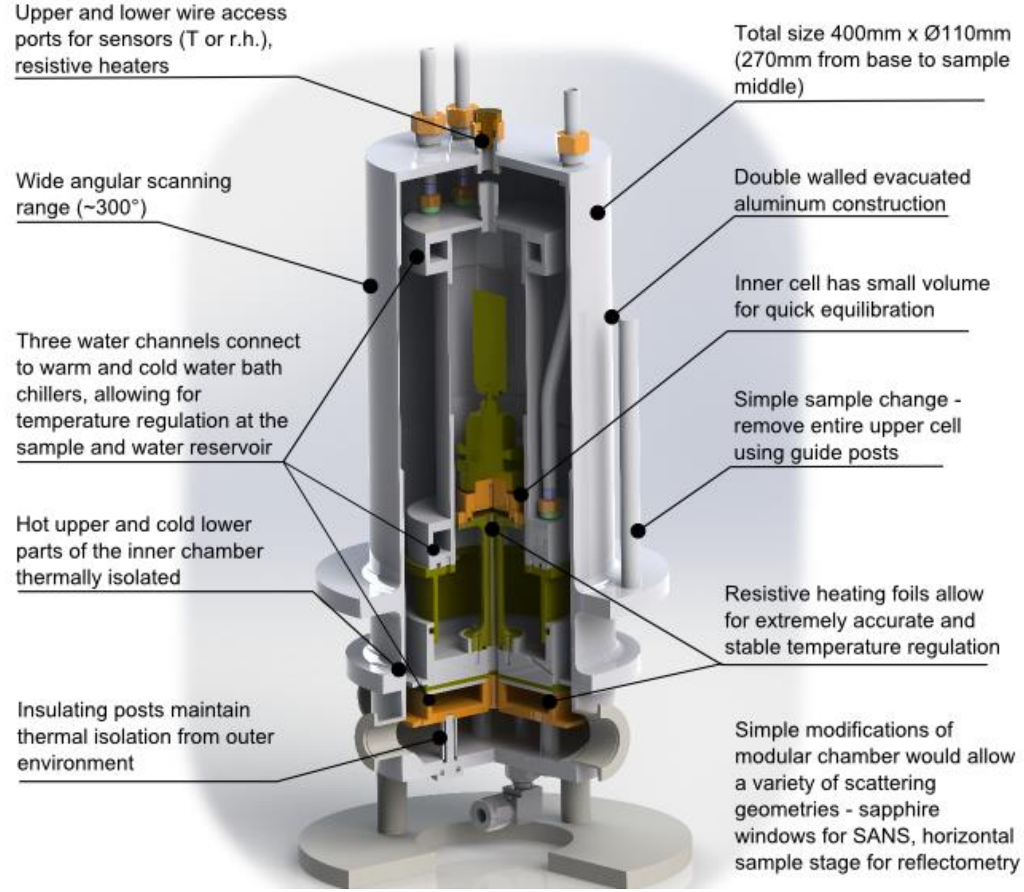


- ILL and HZB making parts, completed by end of May?
- First offline tests in Sept, in beam test, summer

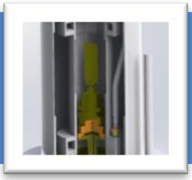
Steps to new geometery

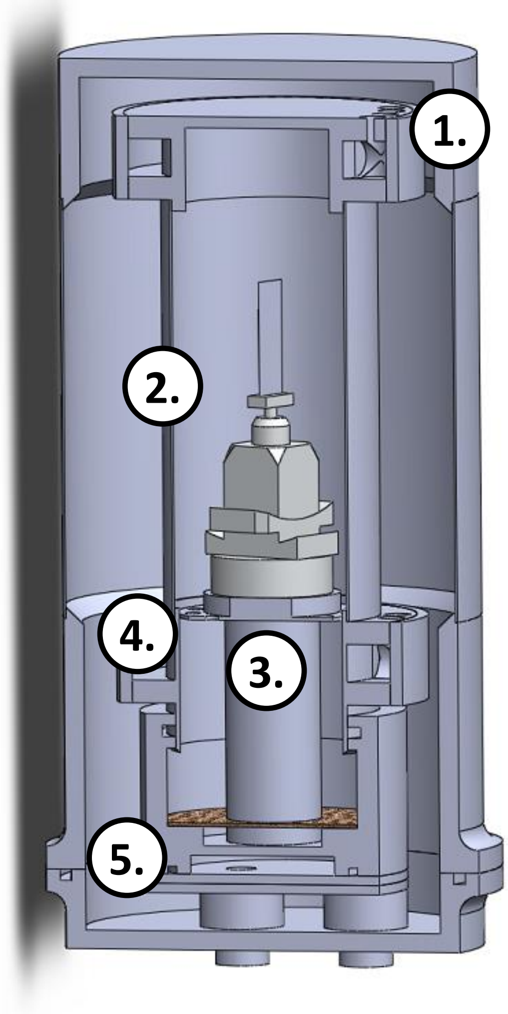






Suggestions for design modifications





1. Forced T gradient in upper cell *not* necessary ($T_1=T_2$ is ok)

2. Stretched upper cell (maximize distance between T_1,T_2)

3. Heater on top of post, under goniometer ($T_1=T_2=T_4$)

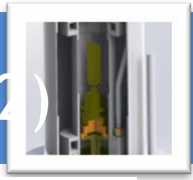
4.´´Thermalizer´´ plate and mesh of high conductivity (placed above T_2)

5. Redesign of bottom cell: completely decouple T_2 from T_3 (stainless steel?)

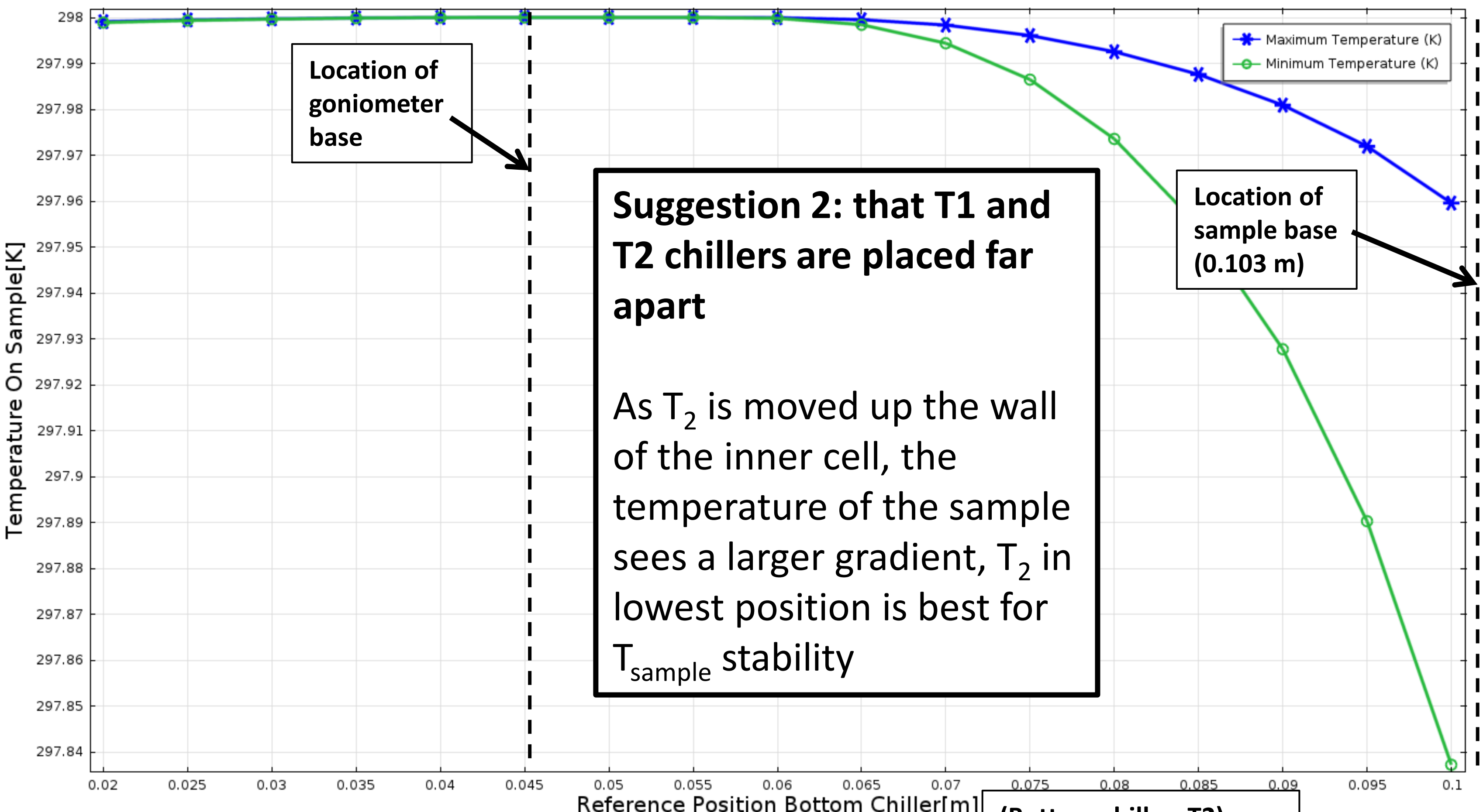
All proceeding simulations with plastic goniometer and quartz sample unless otherwise stated

HZB Helmholtz Zentrum Berlin

2. Stretched upper cell (max. distance between T1,T2)



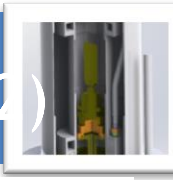
40% rh parametric study of T2 chiller height, plastic goniometer base fixed at 0.048 m



Suggestion 2: that T1 and T2 chillers are placed far apart

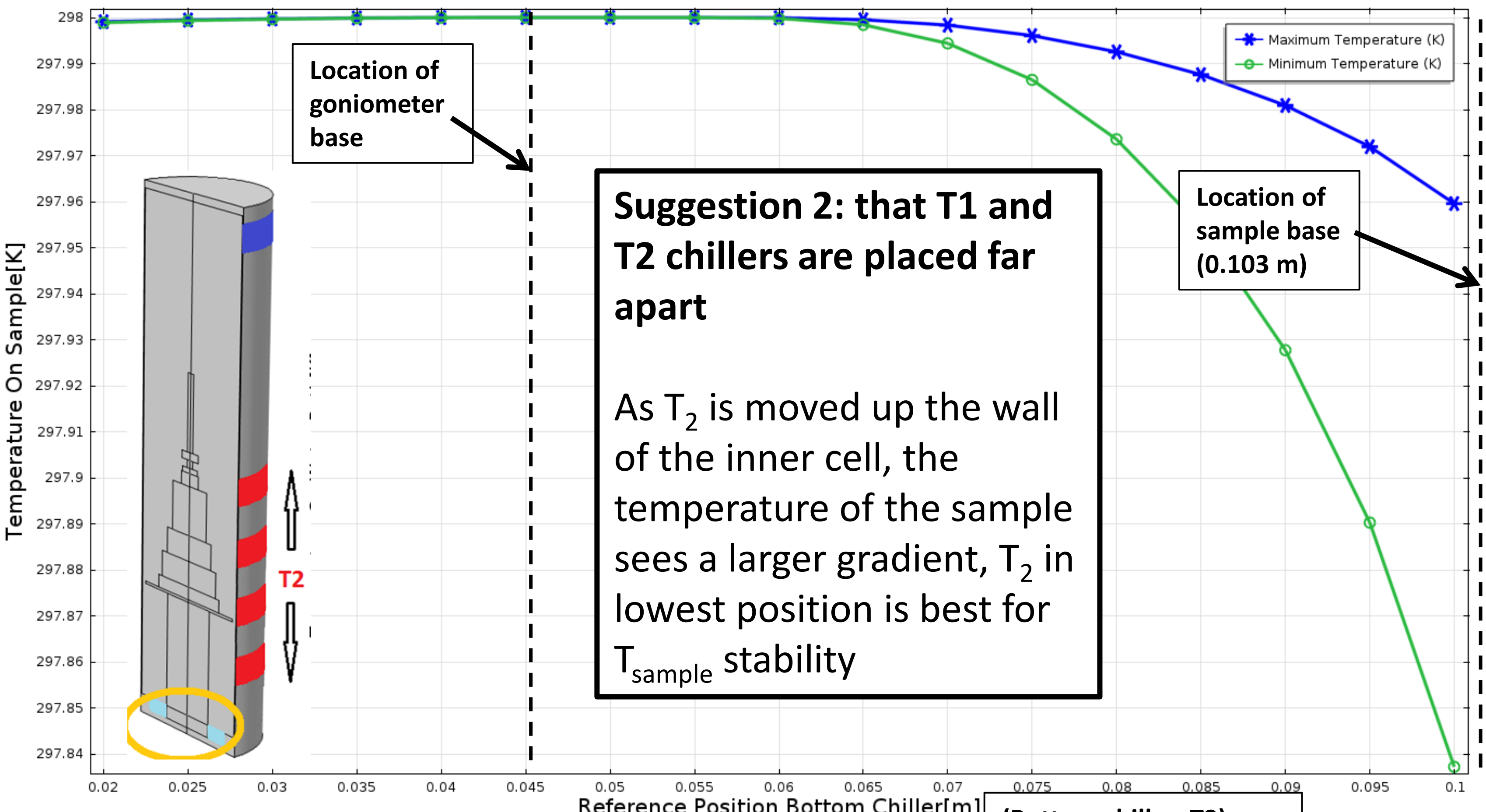
As T_2 is moved up the wall of the inner cell, the temperature of the sample sees a larger gradient, T_2 in lowest position is best for T_{sample} stability

(Waterbath at 0.00 m)



2. Stretched upper cell (max. distance between T1,T2)

40% rh parametric study of T2 chiller height, plastic goniometer base fixed at 0.048 m



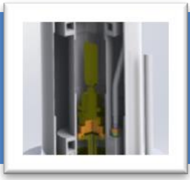
Suggestion 2: that T1 and T2 chillers are placed far apart

As T_2 is moved up the wall of the inner cell, the temperature of the sample sees a larger gradient, T_2 in lowest position is best for T_{sample} stability

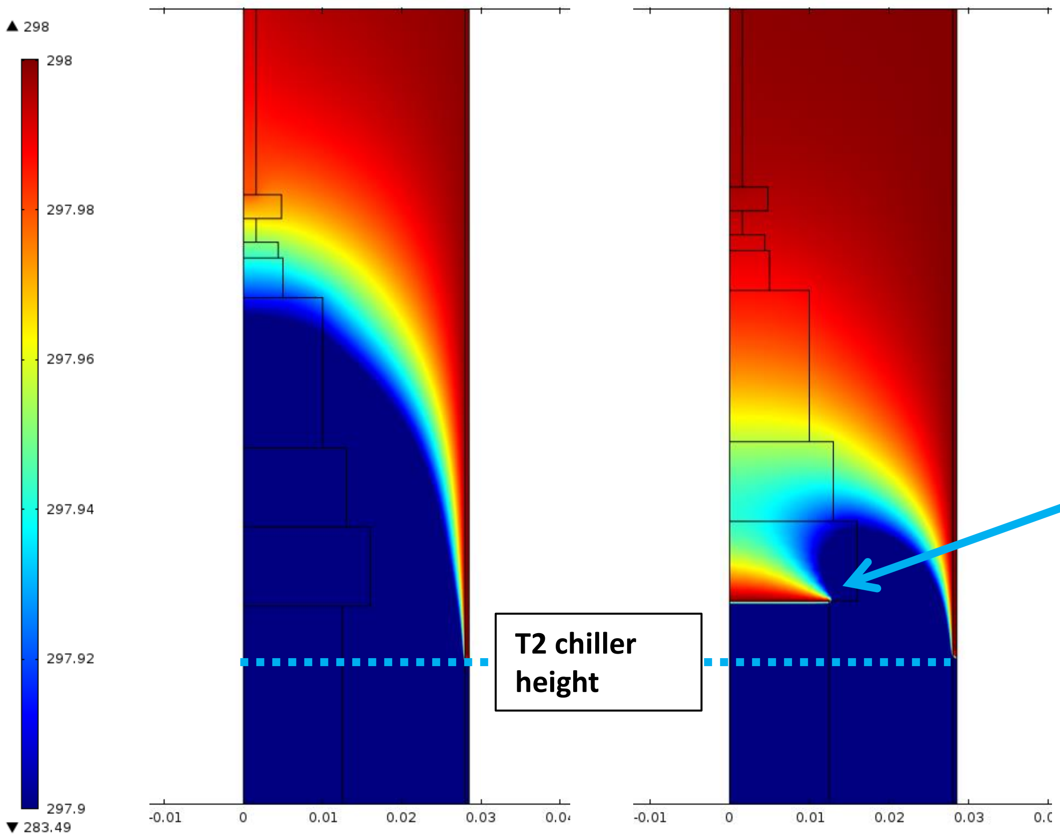
(Bottom chiller=T2)

(Waterbath at 0.00 m)

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



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

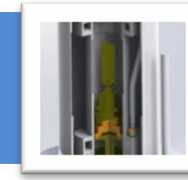


No heater **Heater at 298 K**

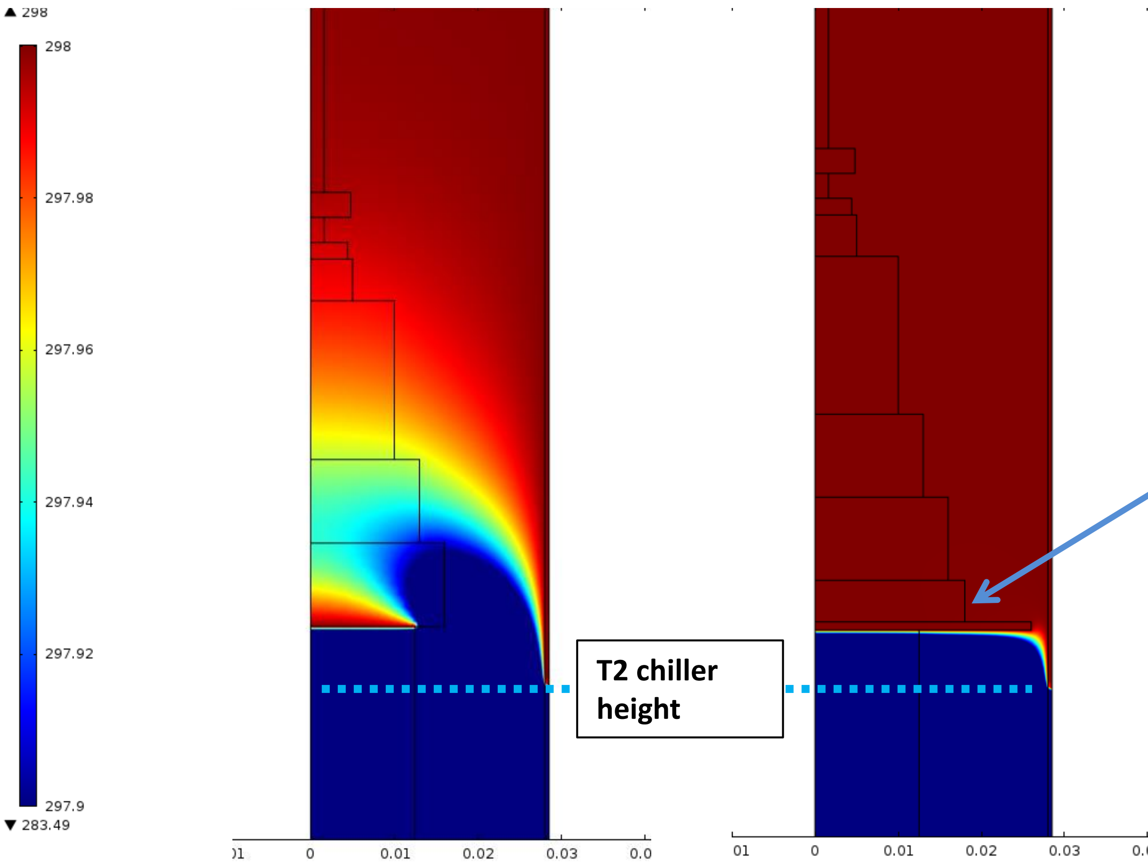
Suggestion 3: a heater is added between post and gonio

Heater shielding the cold bottom chamber temperature from the goniometer

4. Thermalizer plate and mesh of high conductivity (placed above T2)

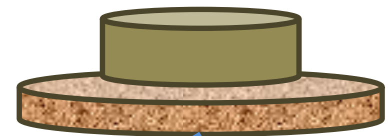


40% (r.h.) study of temperature gradients with and without plate and mesh



No Thermalizer

Solid cylinder and mesh ring

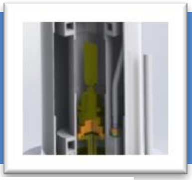


Suggestion 4: Add high conductivity material as thermalizer

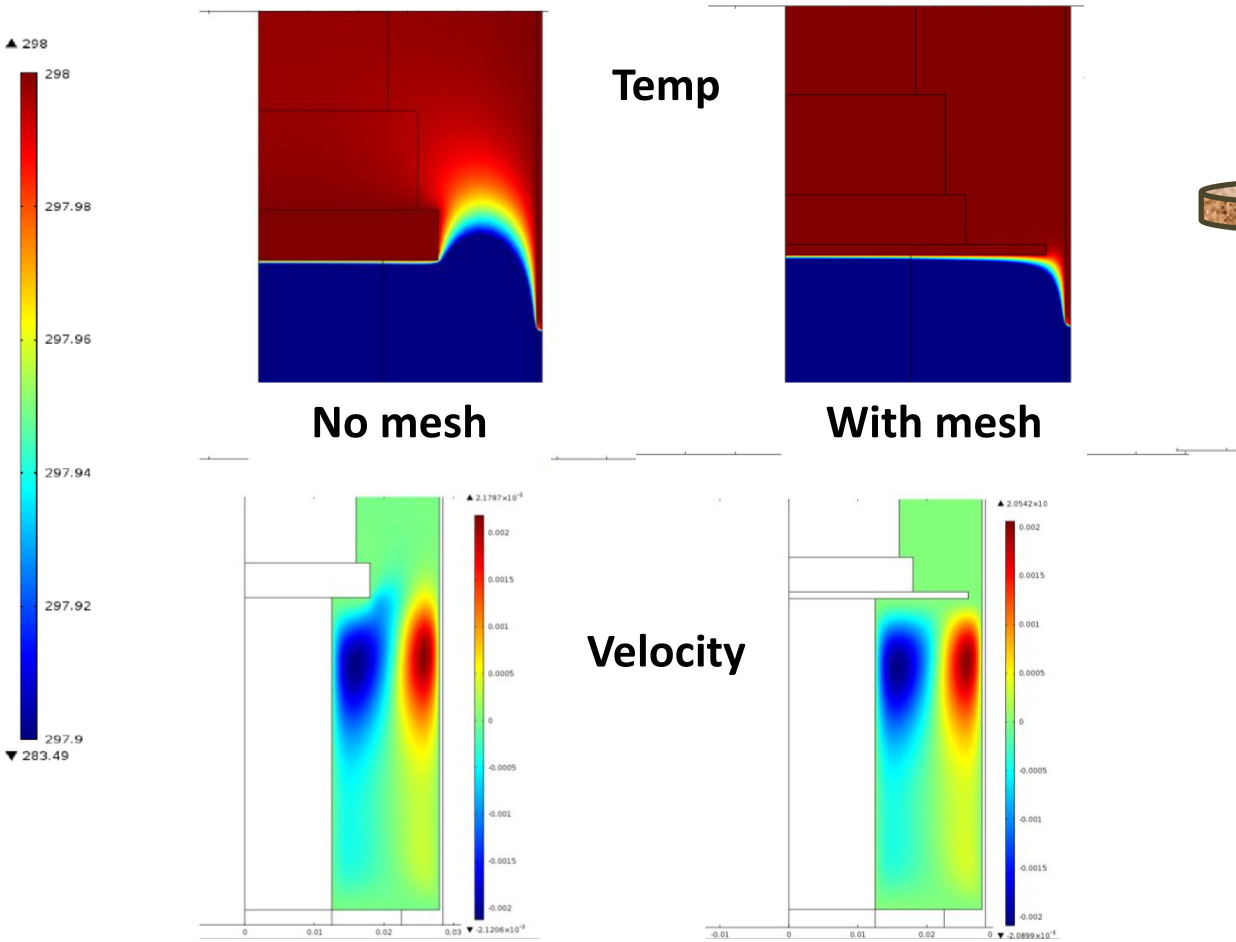
Inhibit cold influence by spreading heat from heater using cylindrical plate and wide mesh ring

HZB Helmholtz Zentrum Berlin

4. Thermalizer plate and mesh of high conductivity (placed above T2)



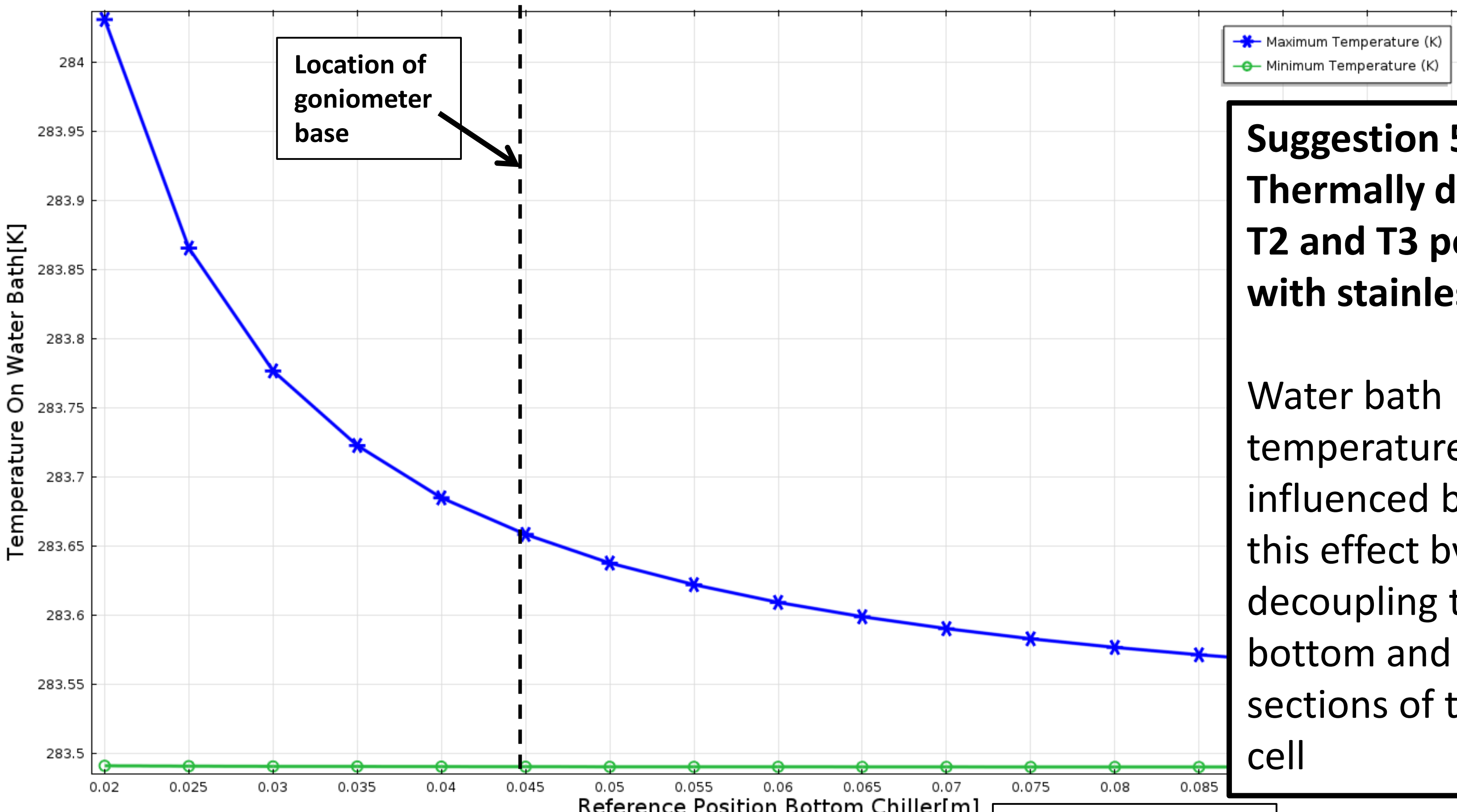
40% (r.h.) study of temperature gradients and air velocity with and without mesh ring



Mesh ring inhibits cold gradient moving up the side of the cell

5. Redesign of bottom cell: completely decouple T_2 from T_3 (stainless steel?)

40% rh parametric study of T2 chiller height, goniometer base fixed at 0.048 m

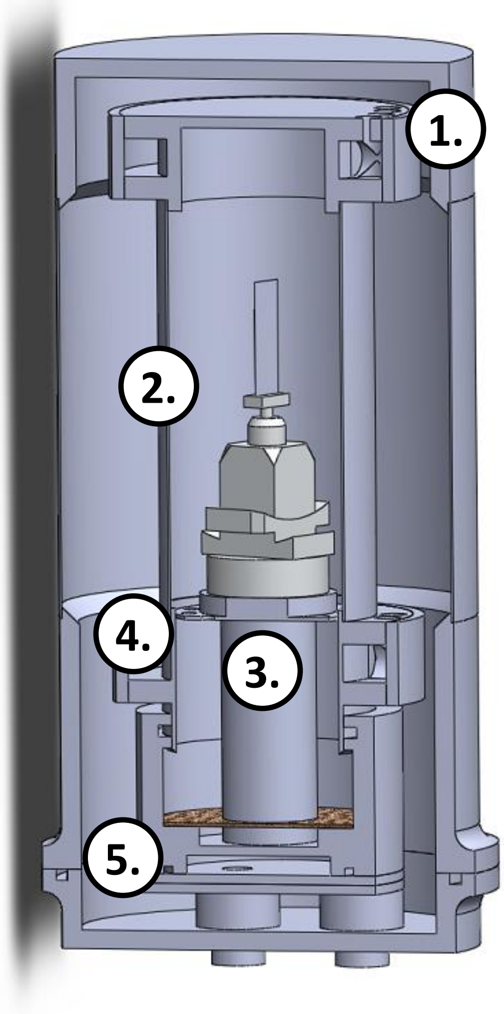


(Bottom chiller=T2)

Suggestion 5: Thermally decouple T2 and T3 possibly with stainless steel

Water bath temperature influenced by T_2, stop this effect by decoupling the bottom and top sections of the inner cell

Suggestions for design modifications



1. Forced T gradient in upper cell *not* necessary (T_1=T_2 is ok)

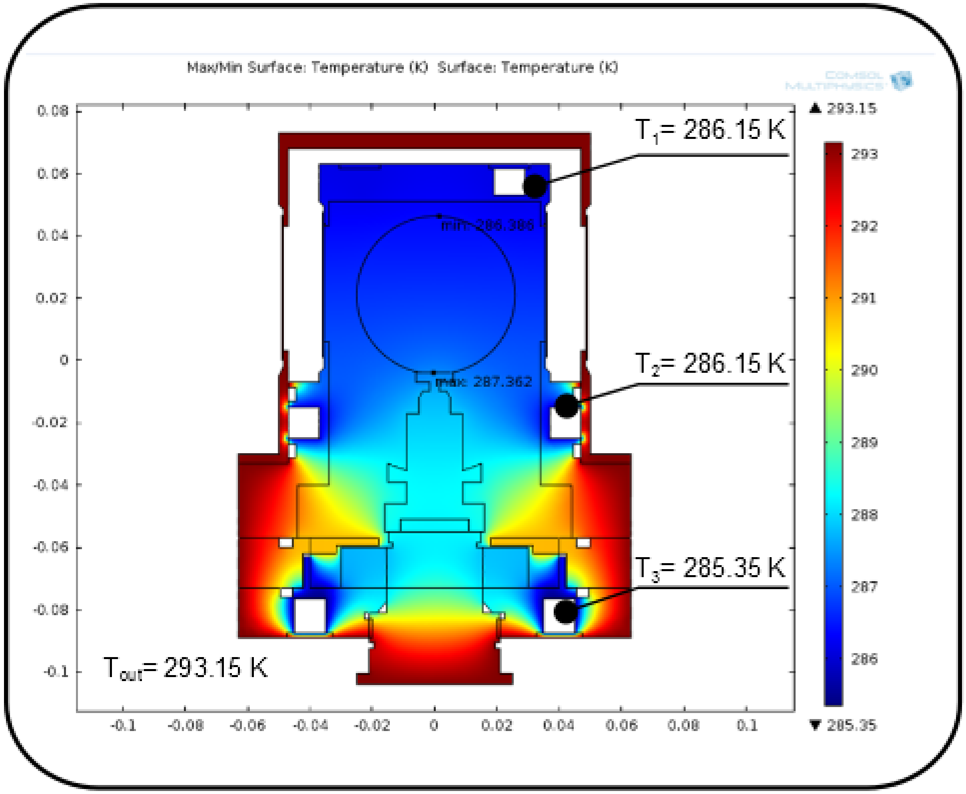
2. Stretched upper cell (maximize distance between T_1,T_2)

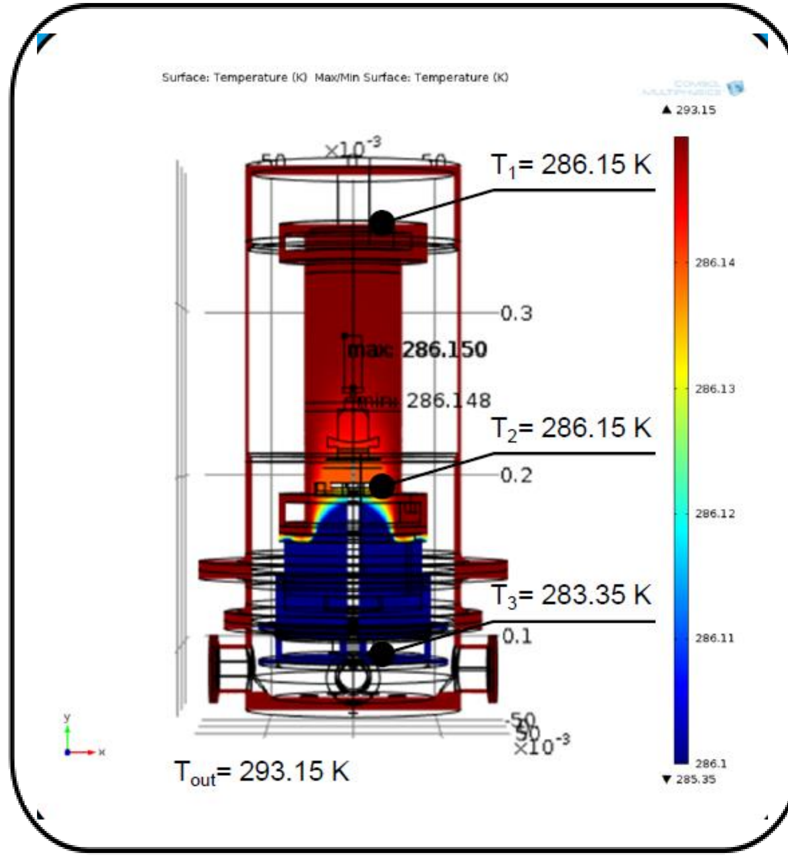
3. Heater on top of post, under goniometer (T_1=T_2=T_4)

4.´´Thermalizer´´ plate and mesh of high conductivity (placed above T_2)

5. Redesign of bottom cell: completely decouple T_2 from T_3 (stainless steel?)

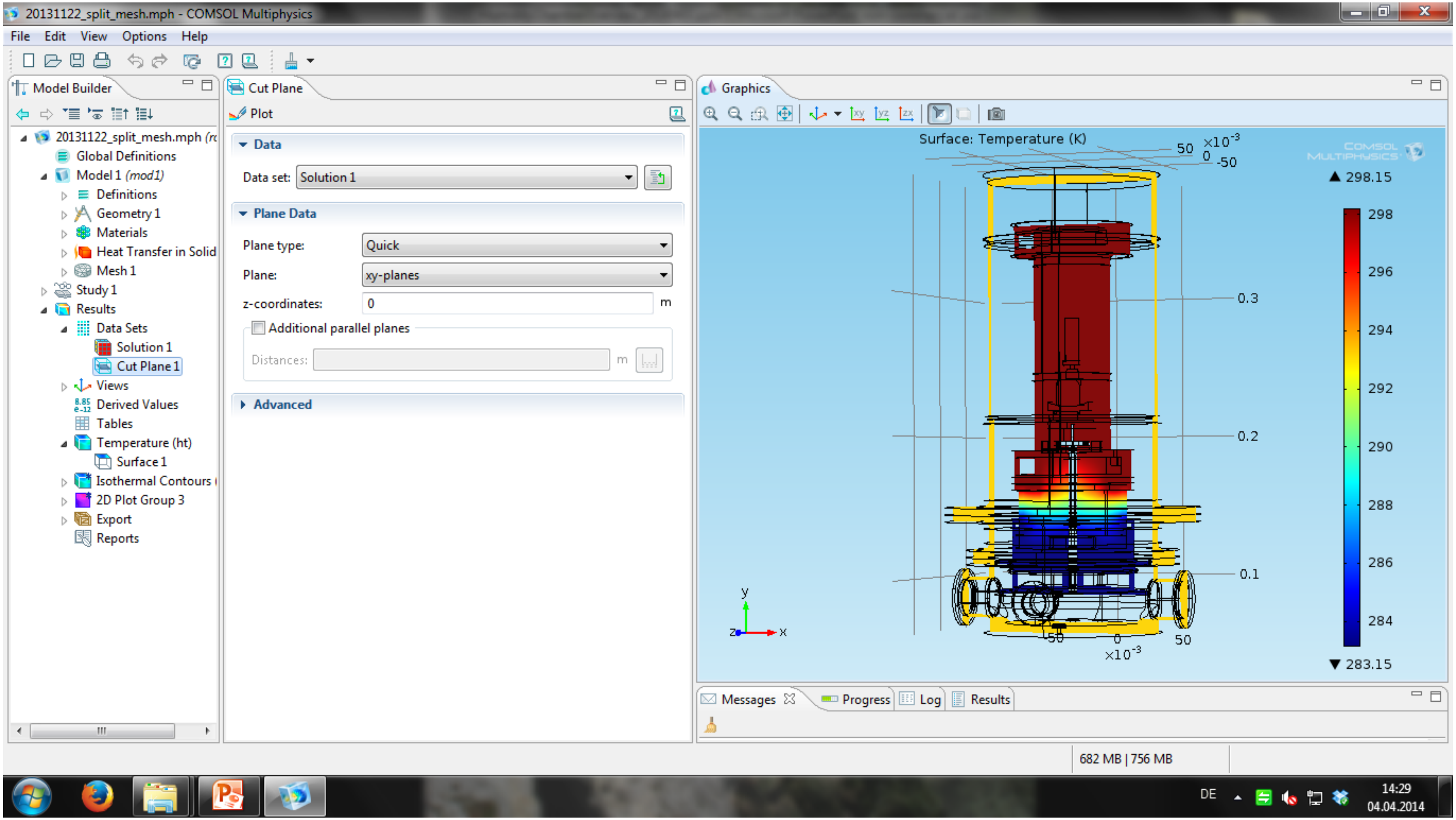
Old and new (and tube problem)



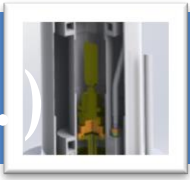


HZB Helmholtz Zentrum Berlin

Old and new

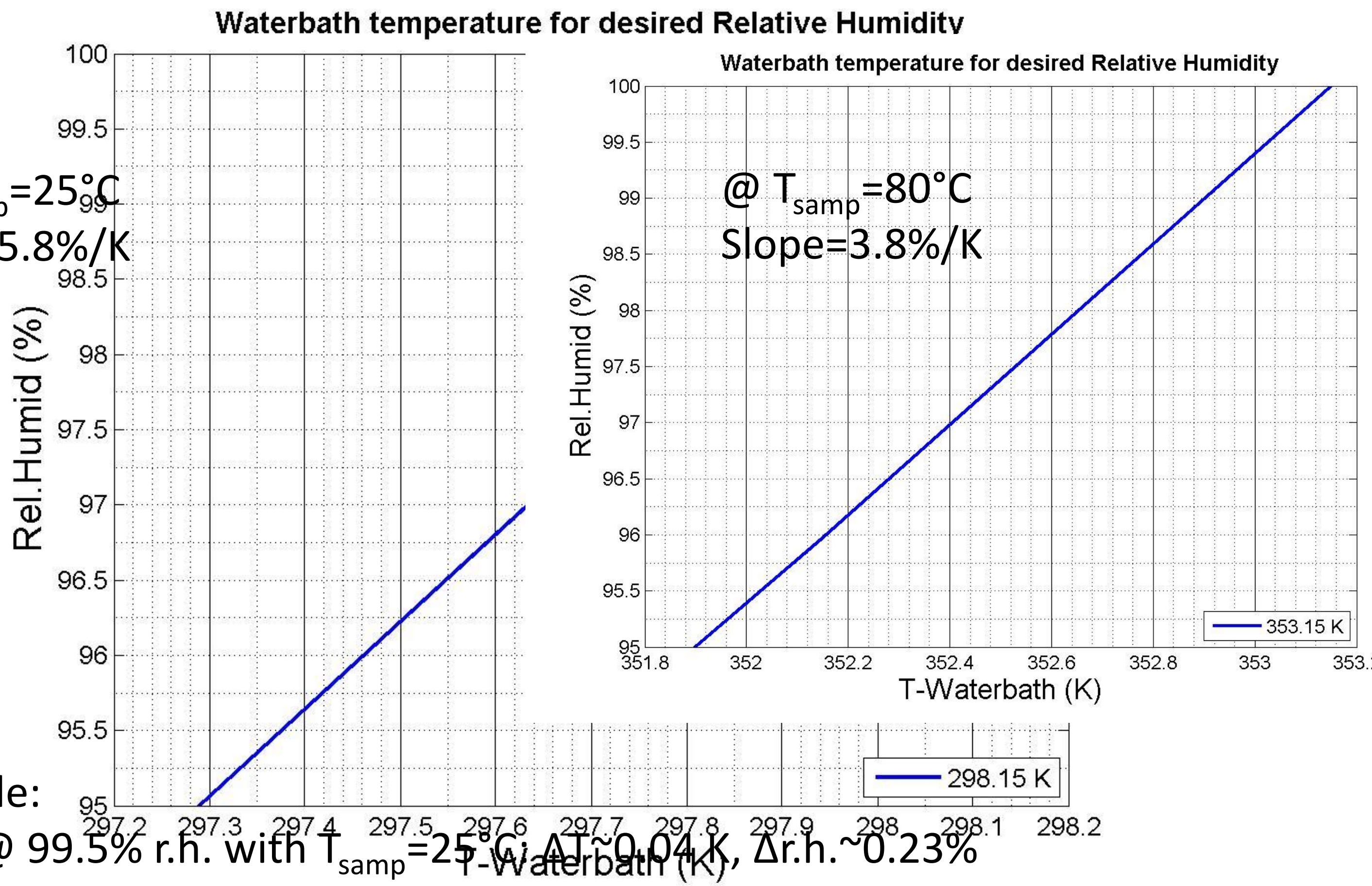

20131122_split_mesh.mph - COMSOL Multiphysics
File Edit View Options Help
Model Builder
20131122_split_mesh.mph
Global Definitions
Model 1 (mod1)
Definitions
Geometry 1
Materials
Heat Transfer in Solid
Mesh 1
Study 1
Results
Data Sets
Solution 1
Cut Plane 1
Views
Derived Values
Tables
Temperature (ht)
Surface 1
Isothermal Contours
2D Plot Group 3
Export
Reports
Cut Plane
Plot
Data
Data set: Solution 1
Plane Data
Plane type: Quick
Plane: xy-planes
z-coordinates: 0 m
Additional parallel planes
Distances: m
Advanced
Graphics
Surface: Temperature (K)
298.15
298
296
294
292
290
288
286
284
283.15
0.3
0.2
0.1
-50
0
50
×10⁻³
Messages
Progress
Log
Results
682 MB | 756 MB
DE
14:29
04.04.2014

Tolerance of T_{bath} uncertainty (above 90%r.h.)



@ T_{samp}=25°C
Slope=5.8%/K

@ T_{samp}=80°C
Slope=3.8%/K



Example:
Error @ 99.5% r.h. with T_{samp}=25°C: ΔT~0.04 K, Δr.h.~0.23%

HZB Helmholtz Zentrum Berlin