Task 3: Humidity Chamber

NMI3-Soft Matter JRA-WP20

Matt Barrett

27.11.2013 - Garching

In this talk…

- Project overview
- Proposed changes since INCS in July
- New (and almost final) design and accompanying simluations

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)

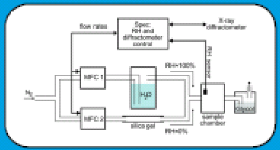


Figure 5. Humidity control setup Salditt, IRP.[a]

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

Bulk water

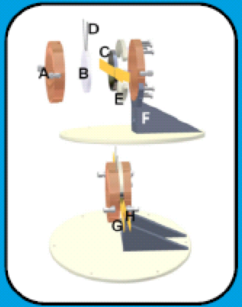


Figure 6. Reflectometry bulk water cell. Harroun, CINS.[c]

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

Temperature controlled

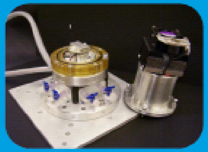


Figure 7. Temp. controlled cell Rheinstädter, McMaster.

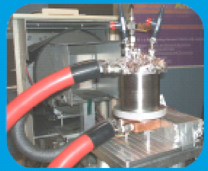


Figure 8. Temp. controlled cell. Heinrich, NIST.

- ✓ 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.

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	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

Design phase – working principle

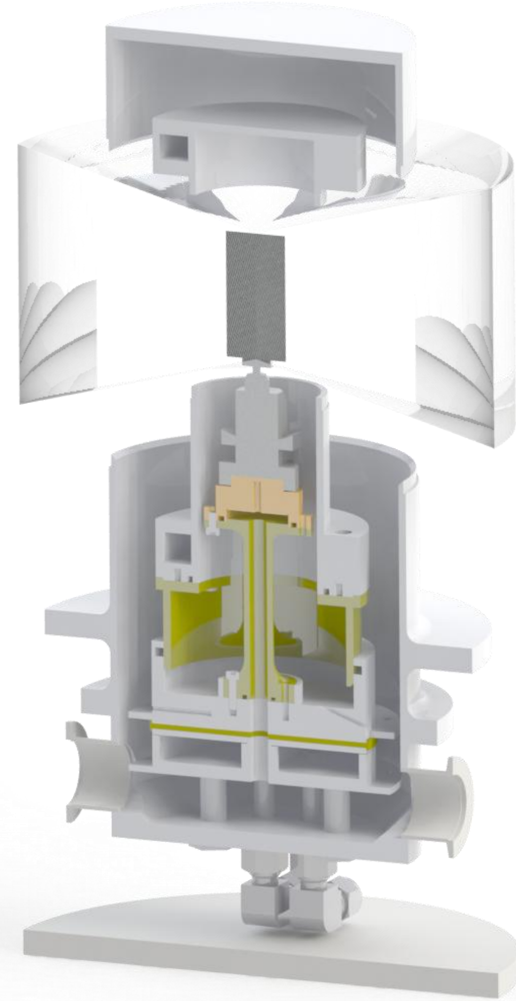
( )

Bridgeman and Aldrich, 1964

Since July…

11.07.2013 - ICNS

Today

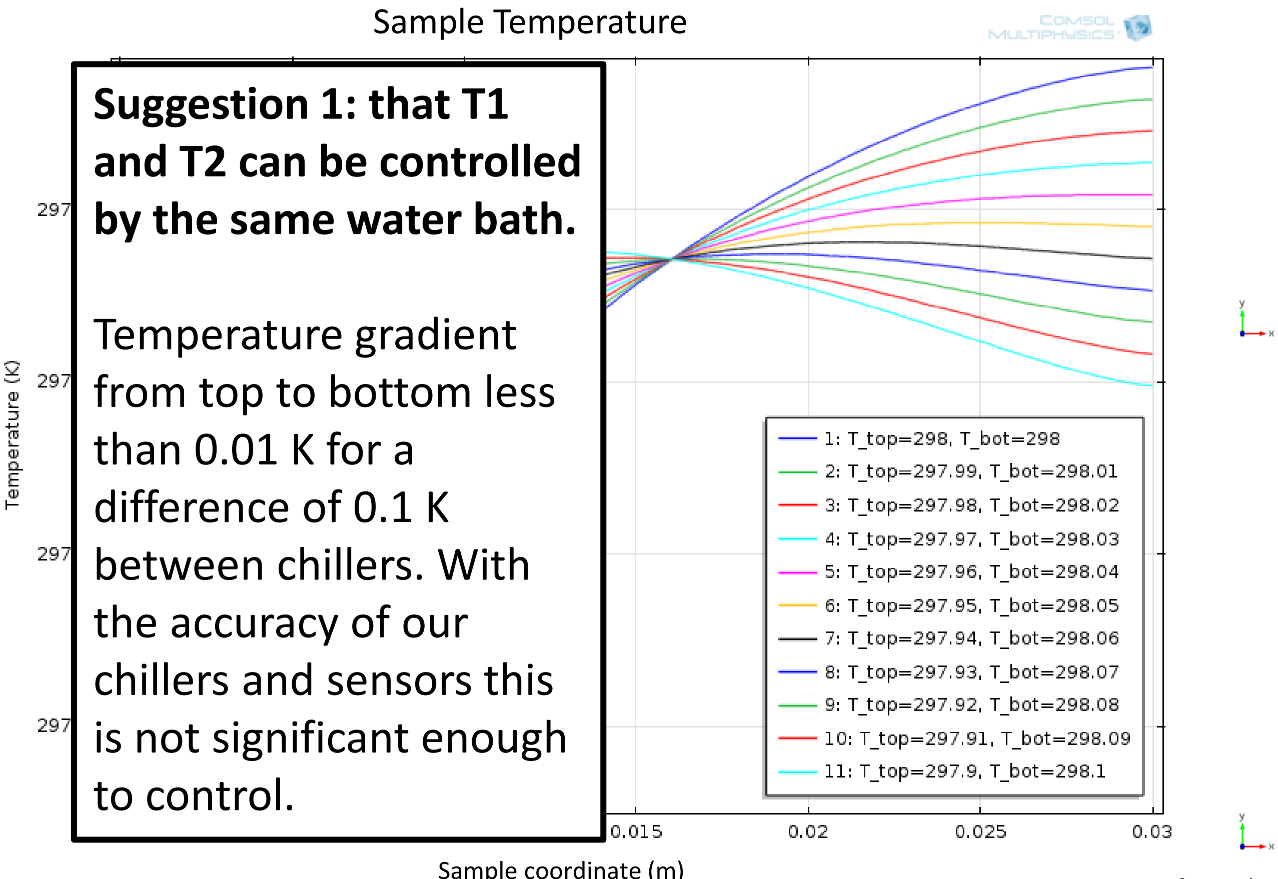


1. No T gradient in upper cell (T1=T2)

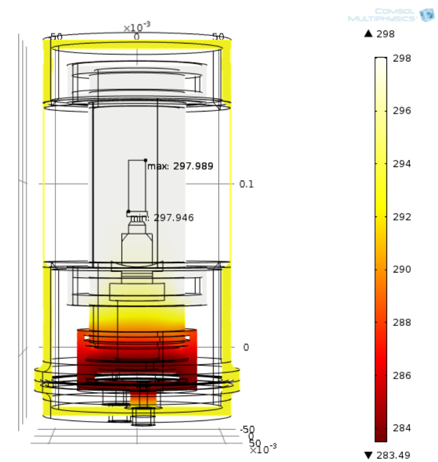
40% (r.h.) parametric study of bottom and top chiller temperature divergence

Suggestion 1: that T1 and T2 can be controlled by the same water bath.

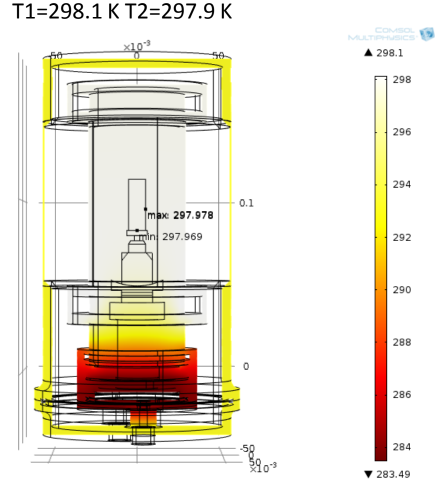
Temperature gradient from top to bottom less than 0.01 K for a difference of 0.1 K between chillers. With the accuracy of our chillers and sensors this is not significant enough to control.



T1=T2=298 K

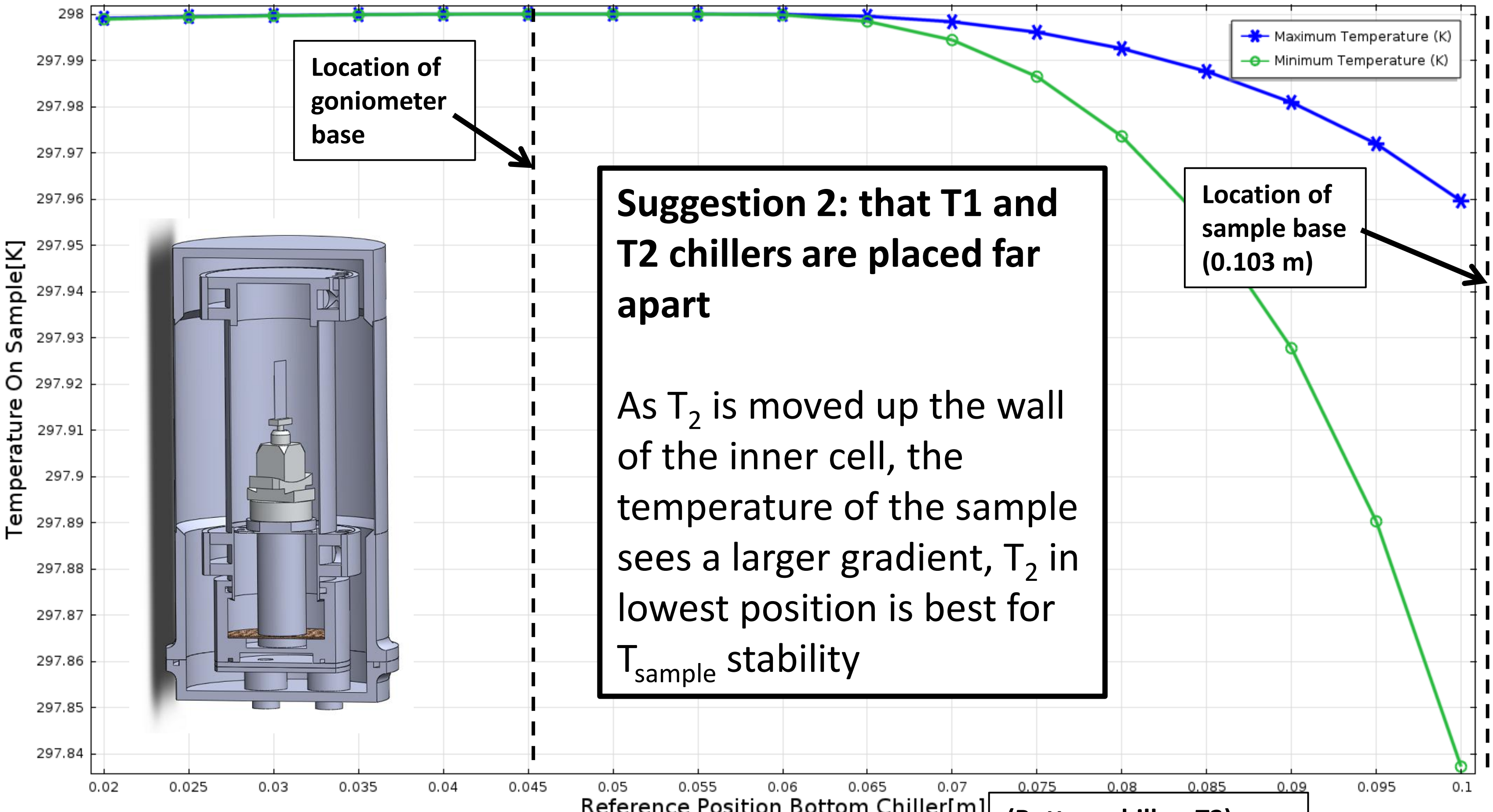


T1=298.1 K T2=297.9 K



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

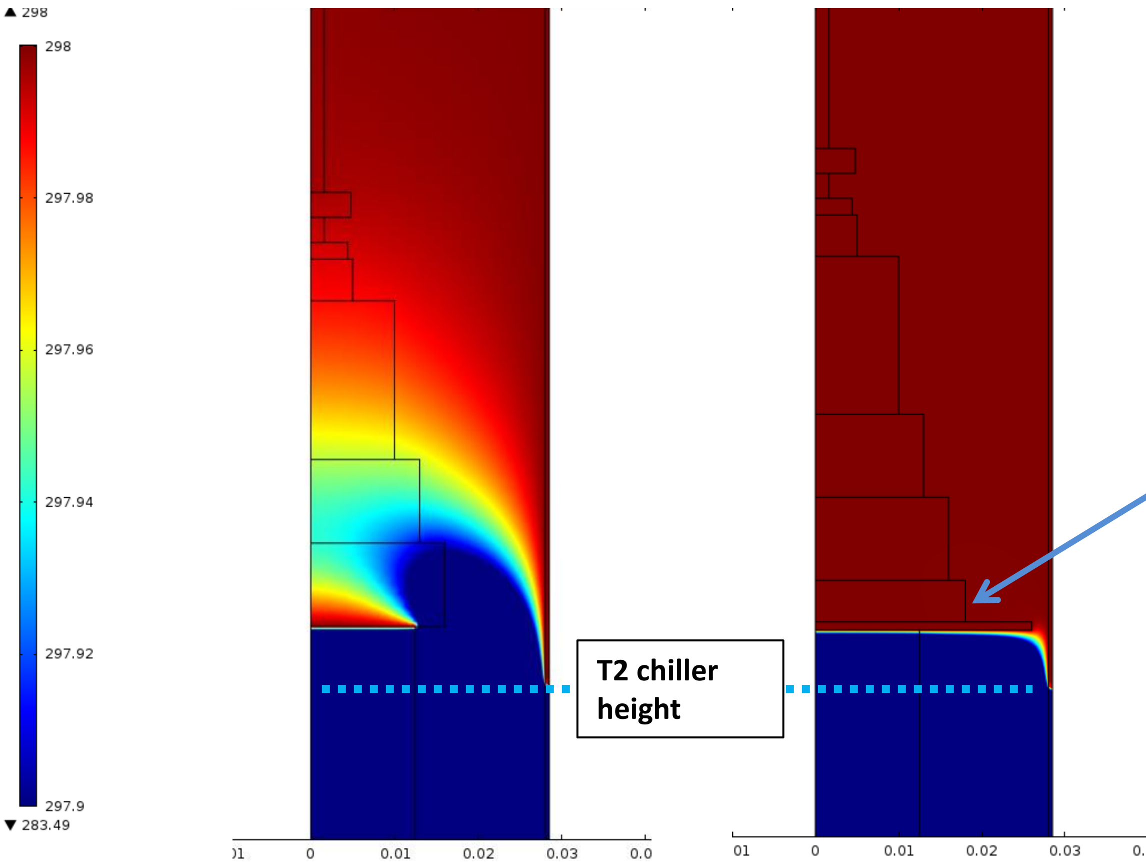
As T_2 is moved up the wall of the inner cell, the temperature of the sample sees a larger gradient, T_2 in lowest position is best for T_{sample} stability

(Waterbath at 0.00 m)

(Bottom chiller=T2)

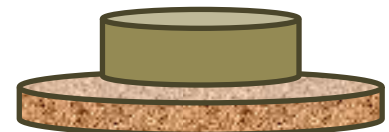
4. ´´Thermalizer´´ plate and mesh of high conductivity (placed above T2)

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



No Thermalizer

Solid cylinder and mesh ring



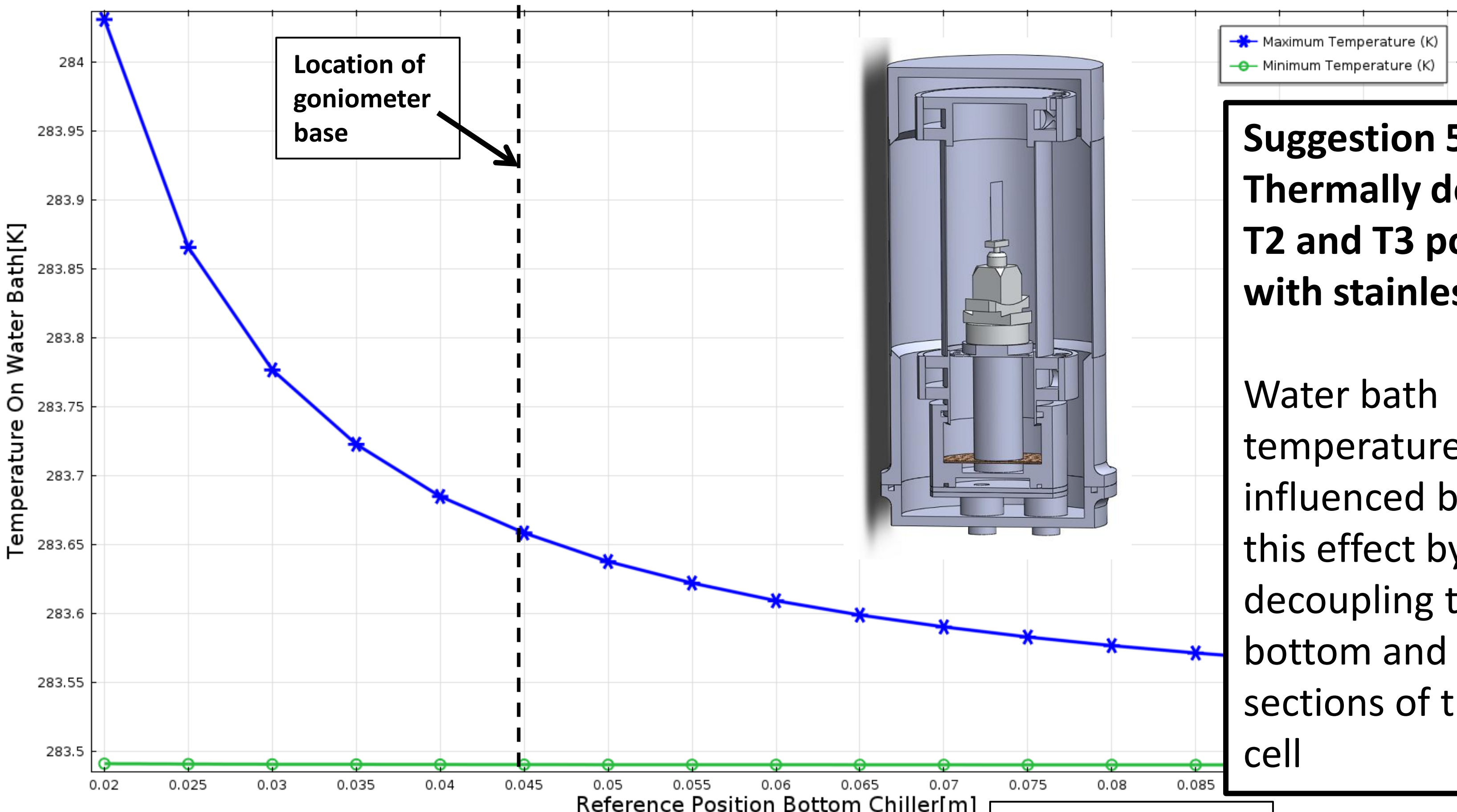
Suggestion 4: Add high conductivity material as thermalizer

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

HZB Helmholtz Zentrum Berlin

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



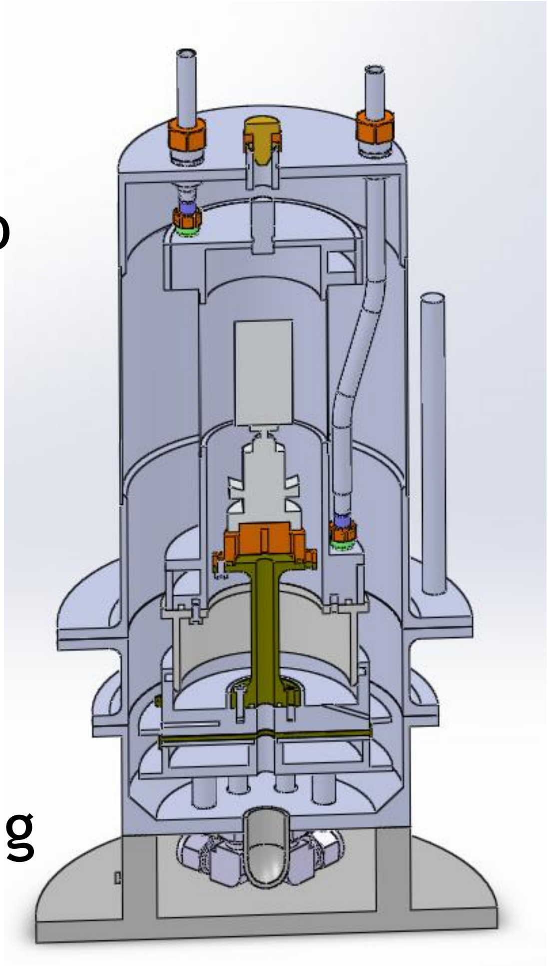
(Bottom chiller=T2)

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

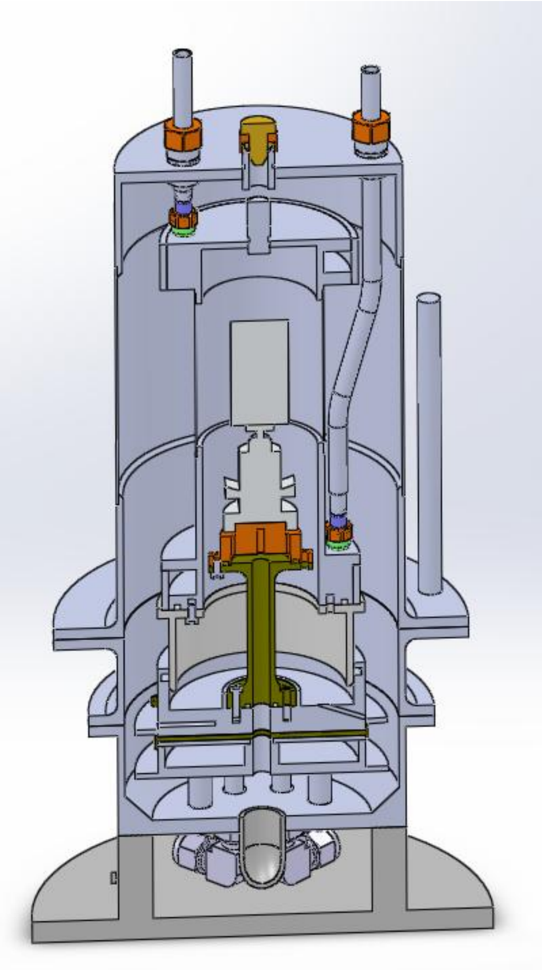
Inner can view

- Double walled (evacuated) Al
- Total size=400xØ110 (270 mm from base to sample) with
- Inner cell=220xØ50 (170 mm above cold chamber region)
- Wide angular scanning range possible (~300°), neutron windows with 15° opening

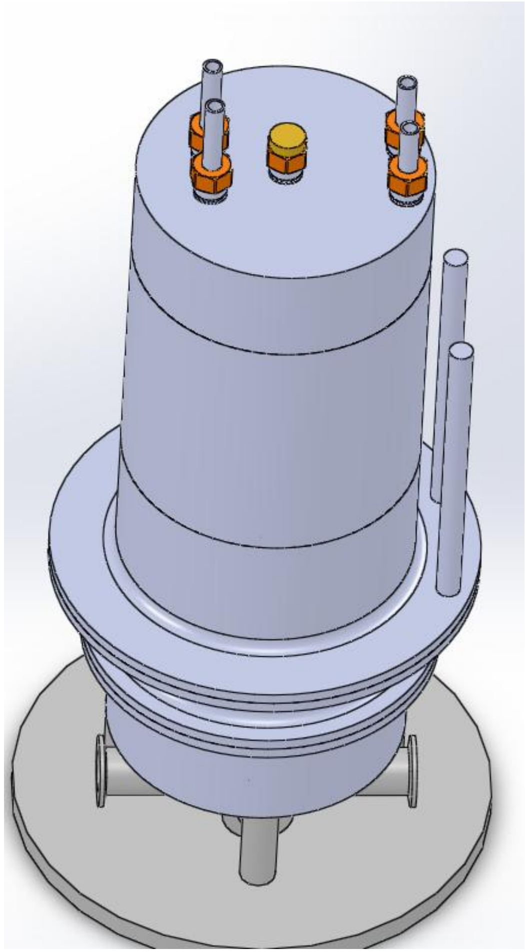


Inner can view

- 3 water chiller channels for precise temp.
- 2 resistive heating foils possible (below gonio and below reservoir)
- Sample cell thermally isolated (plastic post steel supports), T2 and T3 well thermally isolated
- Access for wiring above and below sample (T or RH sensors, Peltier element)

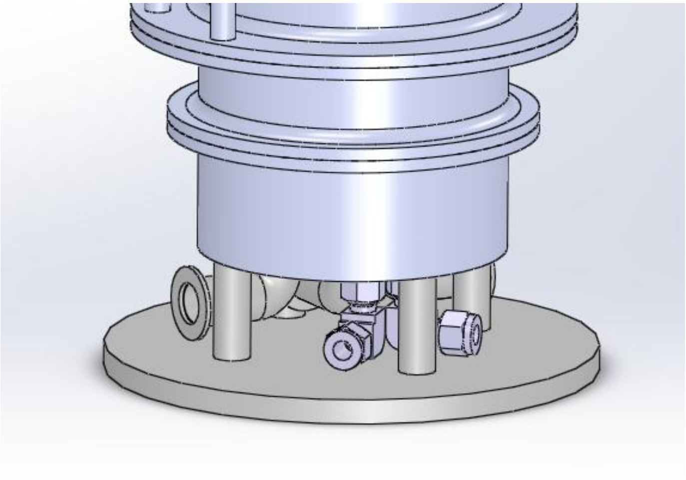


Outer can view



- Above: 4 Wilson seals for T1, T2 water chillers, option for sensors from above

- Below: 3 KF16 flanges and 2 Swagelok fittings for T3 water chiller, heating plates, and sensors



Jäger Connectors

Thanks

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Soft Matter JRA Observers