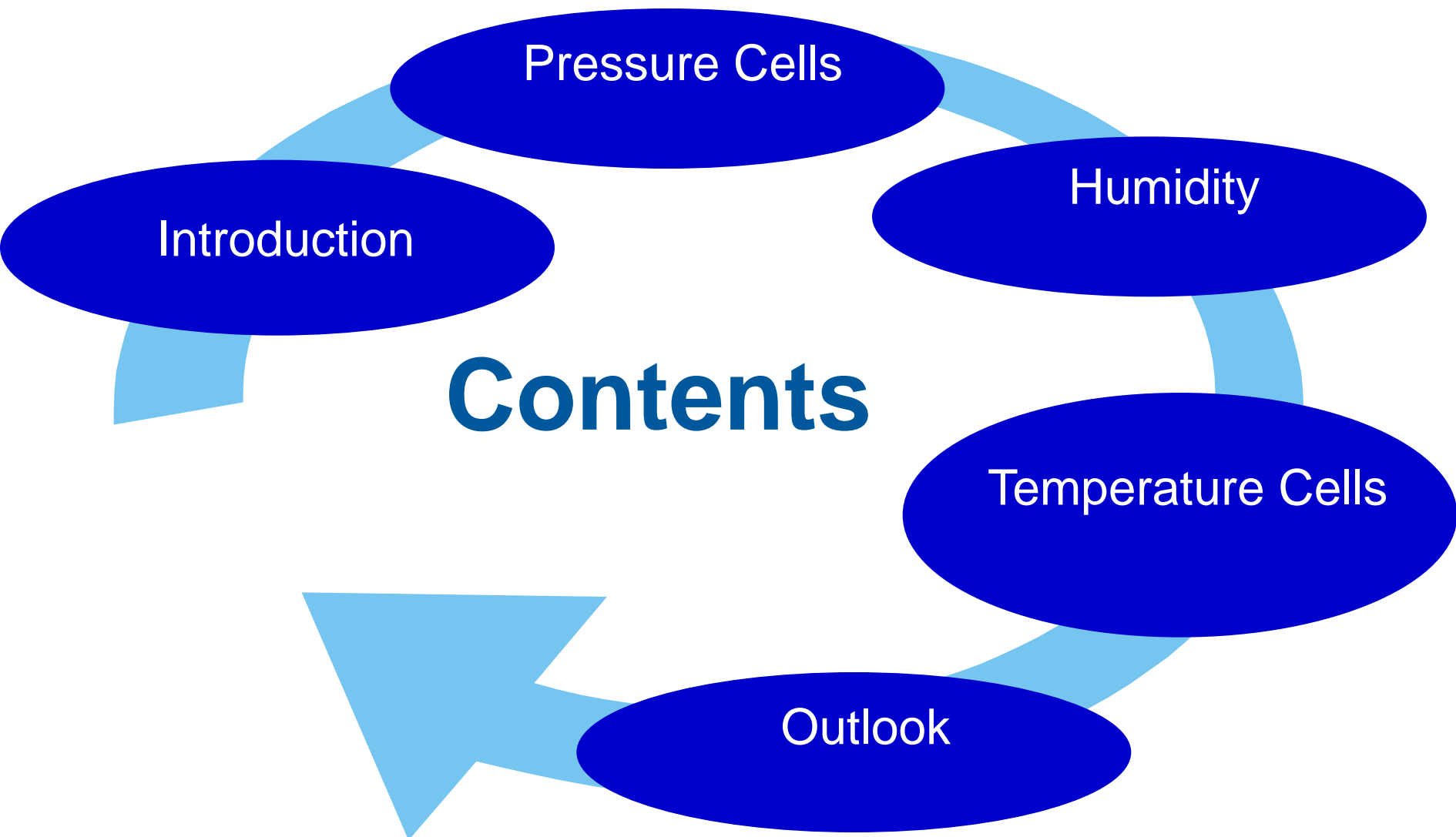




Pressure Cells & Soft Matter Sample Environment at the HZB

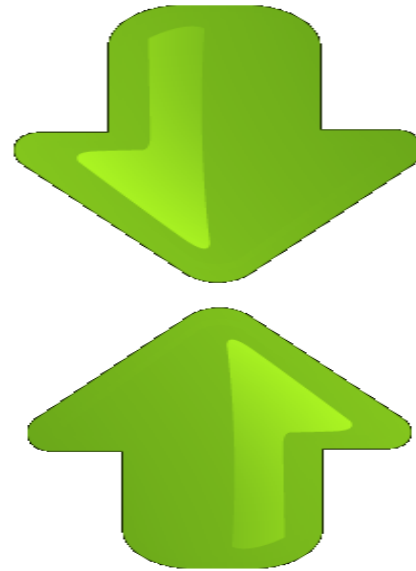
Dirk Wallacher, Helmholtz-Zentrum Berlin





DEGAS Instrumentation at BENSC

- Temperature $T = 2 \text{ K} \dots 1500 \text{ K}$
- Pressure $p = 10^{-6} \text{ mbar} \dots 10 \text{ kbar}$
- Particle number $n = \mu\text{mol} \dots \text{mol}$; Mass $m = \mu\text{g} \dots \text{g}$



Pressures

Investigations at solid liquid interfaces

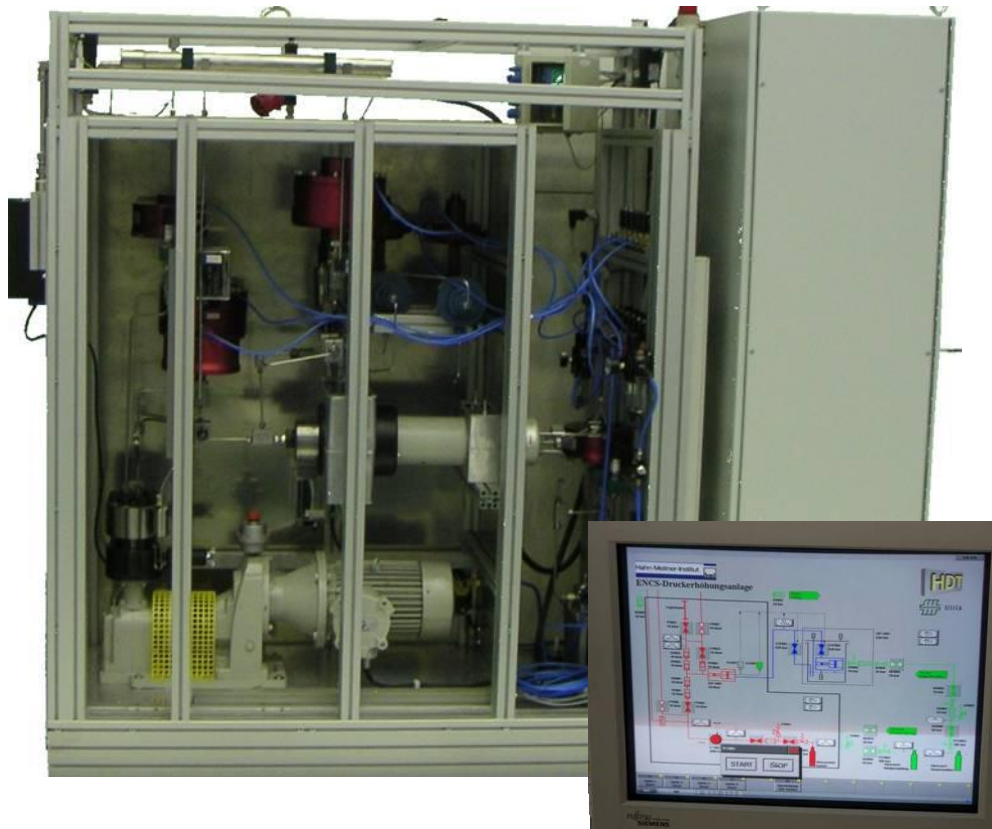
Uni Heidelberg: $P = 1 \text{ kbar}$, $T_{\text{max}} = 60 \text{ C}$

Kreuzer et al, Rev. Scientific Instr. 82, 23902 (2011)

Uni Dortmund: $P = 2.5 \text{ kbar}$, $T_{\text{max}} = 75 \text{ C}$

Jeworrek et al, Rev. Scientific Instr. 82, 25106 (2011)

High Pressure Gas Pressurizer

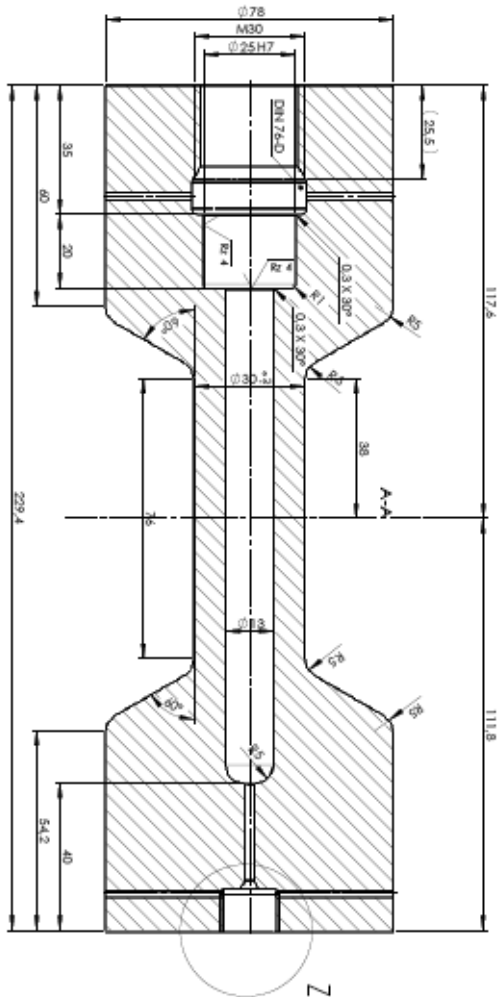


10 kbar pressurizing system for Hydrogen



**3 kbar hydrogen membrane
compressor**

High Pressure Gas Cells



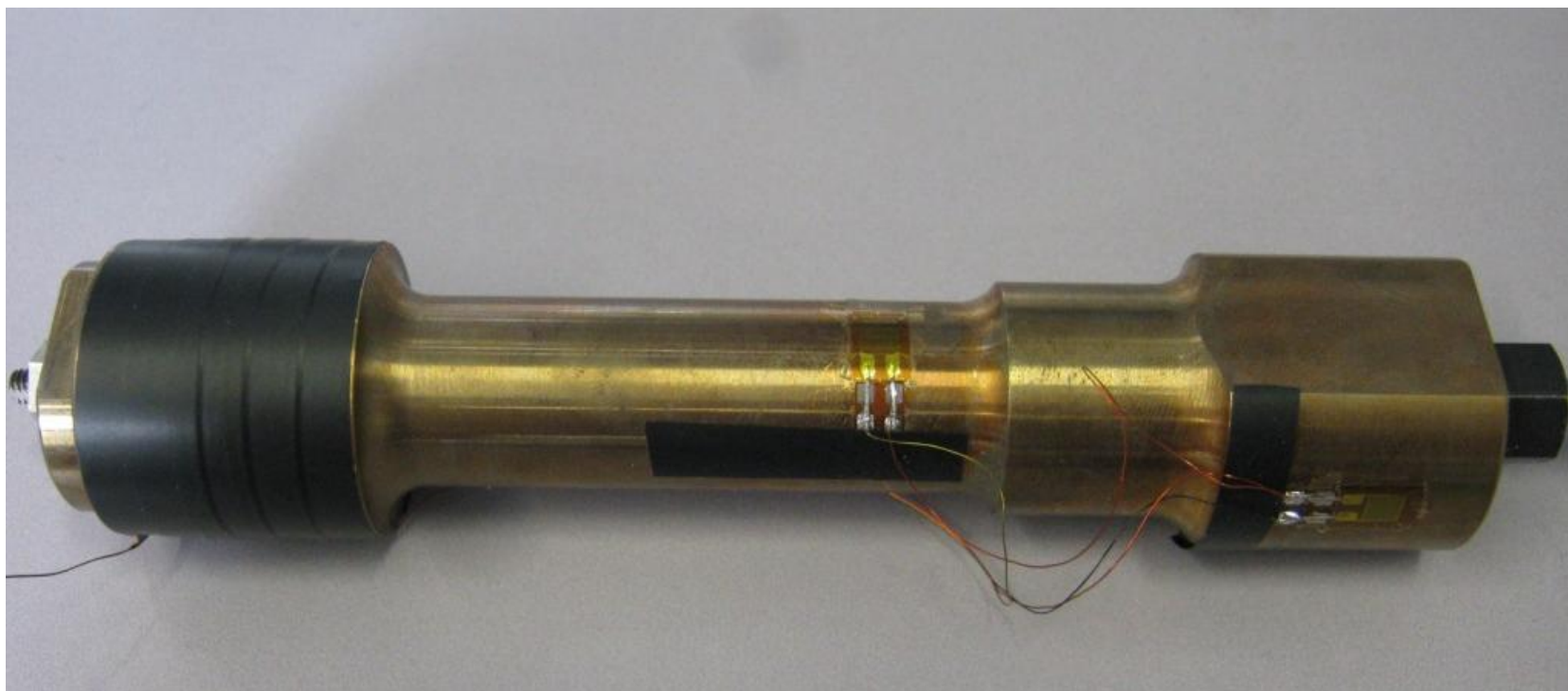
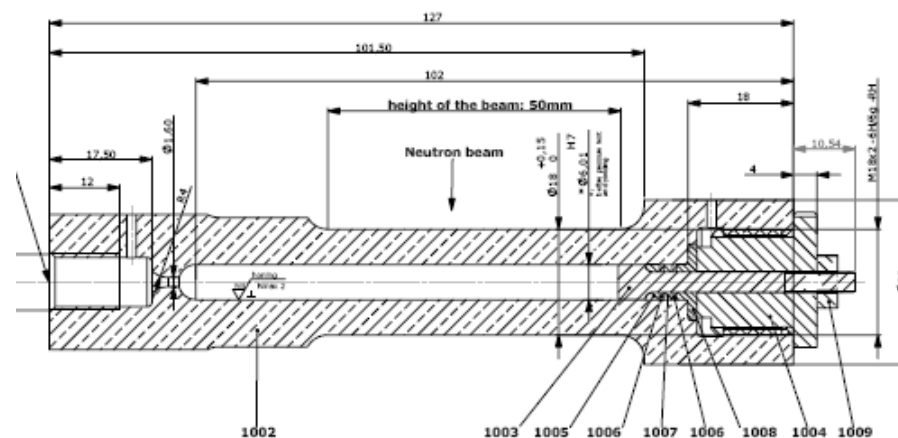
3 kbar gas cell: (Alu 30mm/13mm)



OMF-High Pressure Stick: 10 kbar at 2K

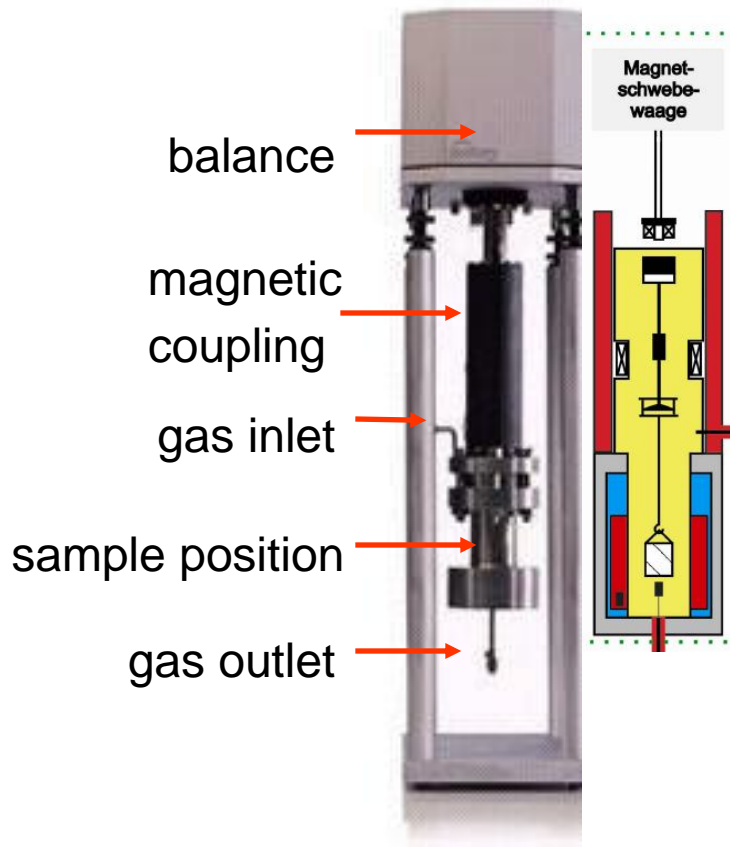


**8 kbar gas cell from LLB
(CuBe 18mm/6mm):**

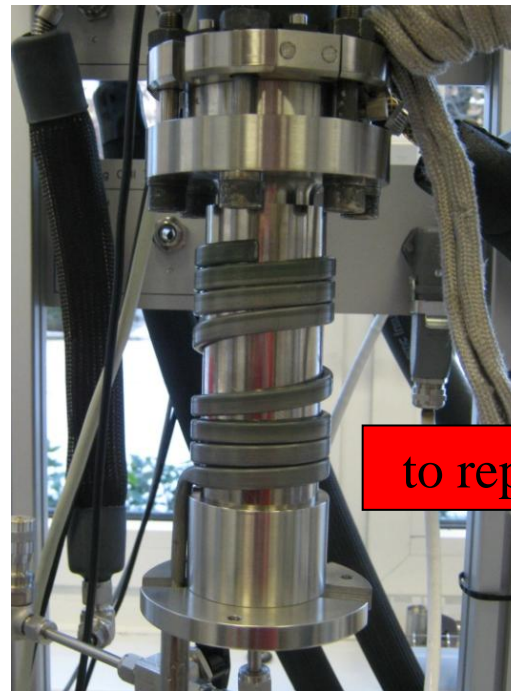


Magnetic Suspension Balance for Neutron Scattering

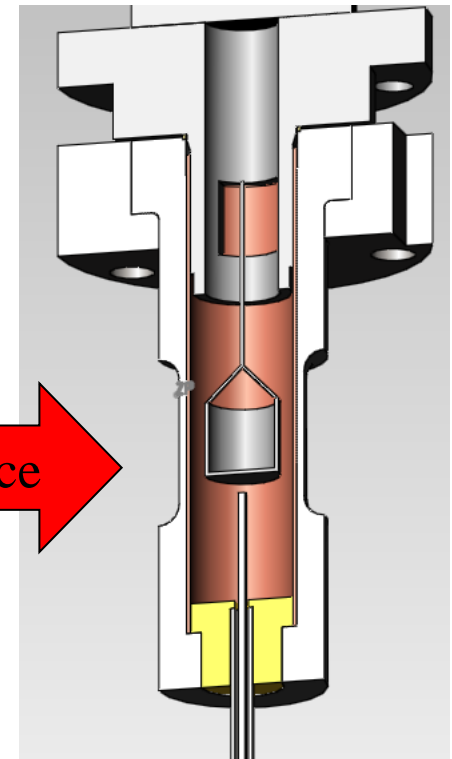
1-10 μg accuracy of weighing QN at extreme conditions (500 °C, 300 bar)



Ex-situ Sample container
from 5mm stainless steel



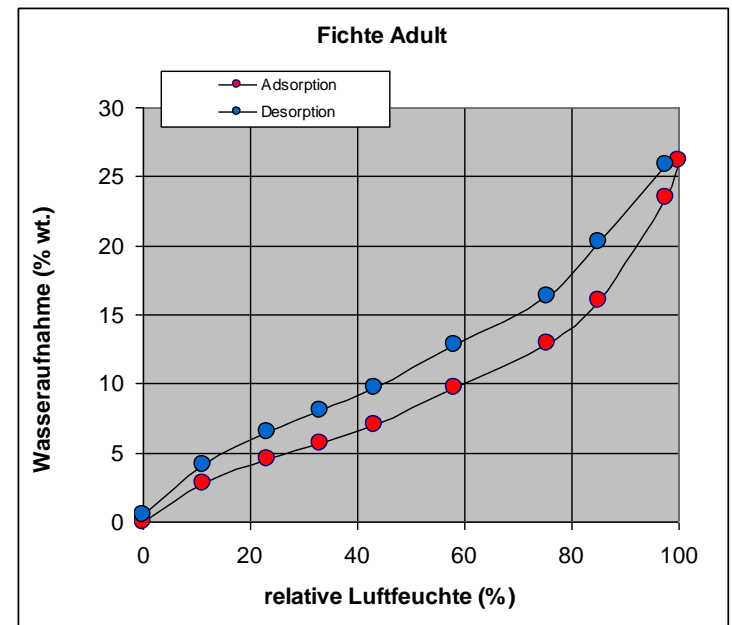
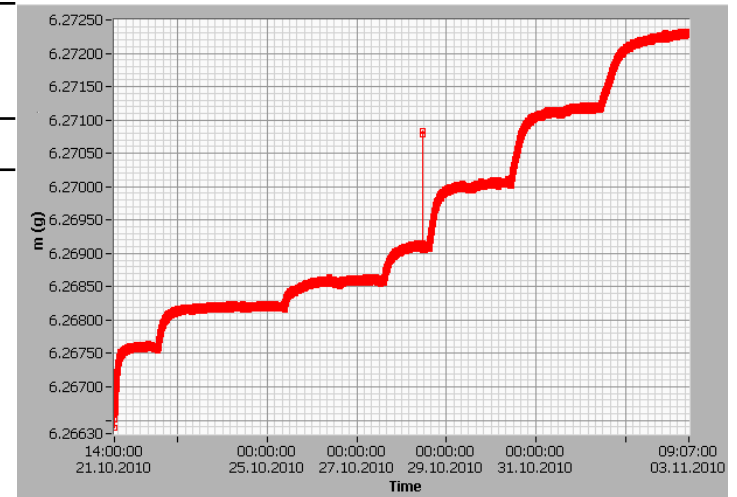
In-situ Sample container
from Ti/Al-alloy



Adapter for Humidity Control by Saturated Salt Solutions



Saturated Salt Solution	Relative Humidity (%) at 20 C
P2O5 (Phosphorpentaoxid)	0.00
LiCl (Lithiumchlorid)	11.31
C2H3KO2 (Kaliumacetat)	23.11
MgCl (Magnesiumchlorid)	33.07
K2CO3 (Kaliumcarbonat)	43.16
NaBr (Natriumbromid)	58.00
NaCl (Natriumchlorid)	75.47
KCl (Kaliumchlorid)	85.11
K2SO4 (Kaliumsulfat)	97.59
H2O (Wasser)	100.00
K2SO4 (Kaliumsulfat)	97.59
KCl (Kaliumchlorid)	85.11
NaCl (Natriumchlorid)	75.47
NaBr (Natriumbromid)	58.00
K2CO3 (Kaliumcarbonat)	43.16
MgCl (Magnesiumchlorid)	33.07
C2H3KO2 (Kaliumacetat)	23.11
LiCl (Lithiumchlorid)	11.31
P2O5 (Phosphorpentaoxid)	0.00





Task 3: Humidity Chamber

NMI3-Soft Matter JRA-WP20

Humidity Chamber: Participants

Task Leader



Partners



Observers



Chamber challenges

REACHING 100% HUMIDITY (NOT BULK WATER)

AVOID TEMPERATURE GRADIENTS = CONDENSATION

PRECISE MEASUREMENT OF HUMIDITY AND TEMPERATURE

CONTRAST VARIATION OPTION WITH D₂O/H₂O MIXTURES

SHORT EQUILIBRATION TIMES

VERSATILITY FOR ALL SETUPS

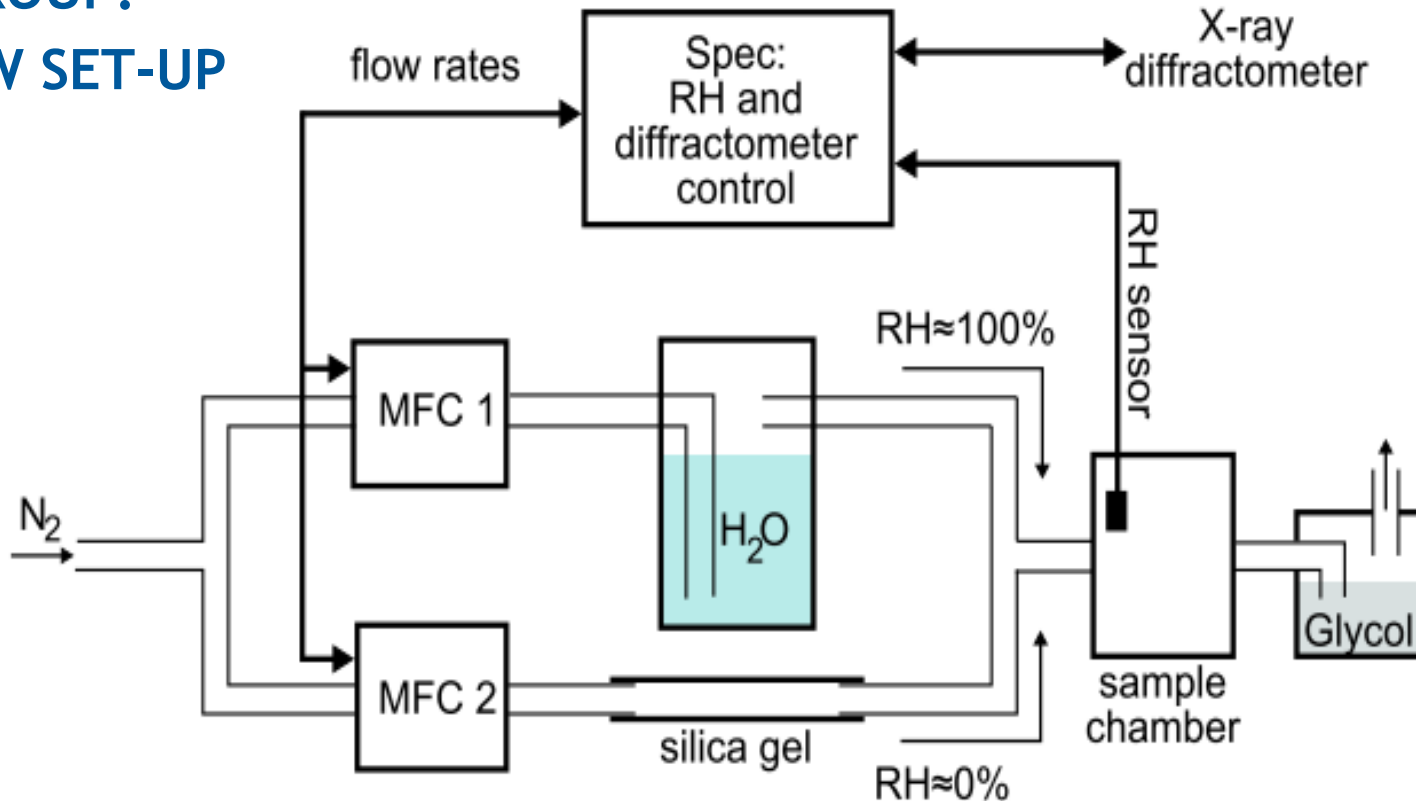
EASE OF USE

Humidity control methods

- Gas flow
- Saturated salt solutions
- Temperature control
- Fixed humidity preparation
- Bulk water

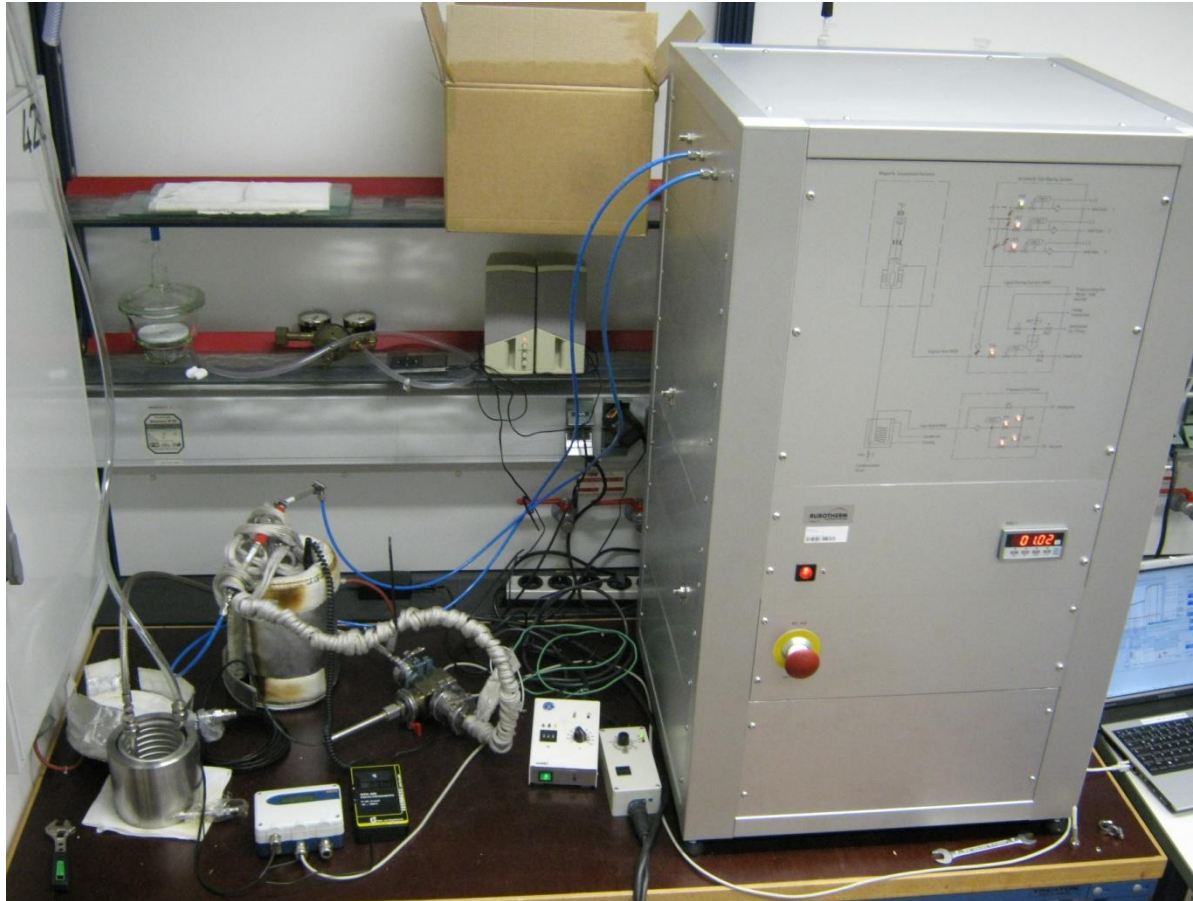
Gas Flow

SALDITT GROUP: X-RAY FLOW SET-UP



Aeffner, S. *The European physical journal. E*, 2009

Gas Flow System



Saturated Salts

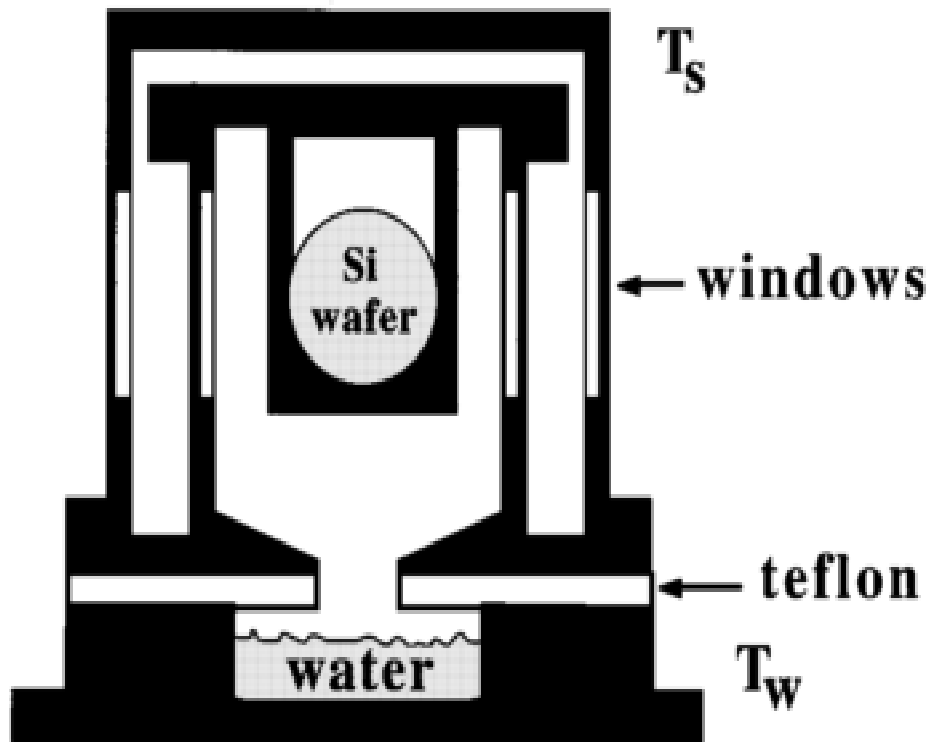
Temperature °C	Relative Humidity (%RH)					
	Potassium Carbonate	Magnesium Nitrate	Sodium Chloride	Potassium Chloride	Potassium Nitrate	Potassium Sulfate
0	43.13 ± 0.66	60.35 ± 0.55	75.51 ± 0.34	88.61 ± 0.53	96.33 ± 2.9	98.77 ± 1.1
5	43.13 ± 0.50	58.86 ± 0.43	75.65 ± 0.27	87.67 ± 0.45	96.27 ± 2.1	98.48 ± 0.91
10	43.14 ± 0.39	57.36 ± 0.33	75.67 ± 0.22	86.77 ± 0.39	95.96 ± 1.4	98.18 ± 0.76
15	43.15 ± 0.33	55.87 ± 0.27	75.61 ± 0.18	85.92 ± 0.33	95.41 ± 0.96	97.89 ± 0.63
20	43.16 ± 0.33	54.38 ± 0.23	75.47 ± 0.14	85.11 ± 0.29	94.62 ± 0.66	97.59 ± 0.53
25	43.16 ± 0.39	52.89 ± 0.22	75.29 ± 0.12	84.34 ± 0.26	93.58 ± 0.55	97.30 ± 0.45
30	43.17 ± 0.50	51.40 ± 0.24	75.09 ± 0.11	83.62 ± 0.25	92.31 ± 0.60	97.00 ± 0.40
35		49.91 ± 0.29	74.87 ± 0.12	82.95 ± 0.25	90.79 ± 0.83	96.71 ± 0.38
40		48.42 ± 0.37	74.68 ± 0.13	82.32 ± 0.25	89.03 ± 1.2	96.41 ± 0.38
45		46.93 ± 0.47	74.52 ± 0.16	81.74 ± 0.28	87.03 ± 1.8	96.12 ± 0.40
50		45.44 ± 0.60	74.43 ± 0.19	81.20 ± 0.31	84.78 ± 2.5	95.82 ± 0.45

Omega Process Measurement and Control

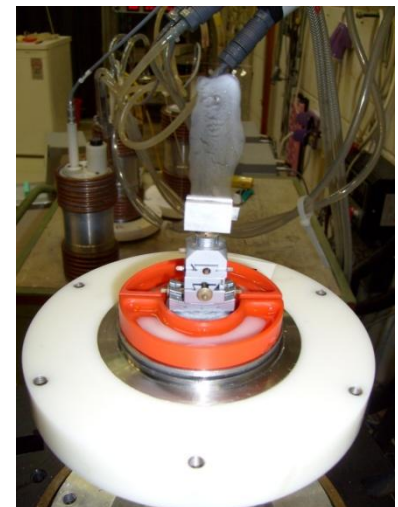


- PRECISE AND RELIABLE HUMIDITY
- NO NEED FOR HUMIDITY SENSOR
- CLEANING SALTS
- RE-EQUILIBRATION
- DISCONTINUOUS (CHANGE SALT COCENTRATION TO FILL GAPS)

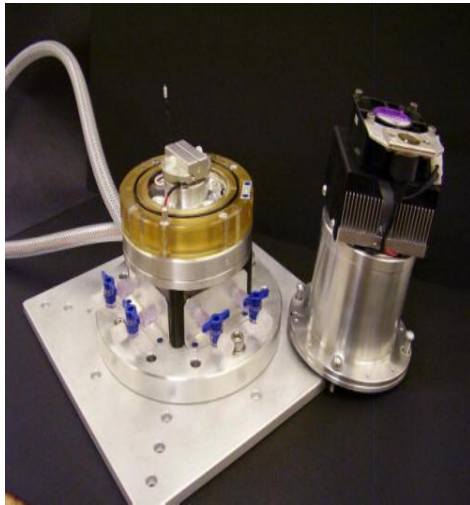
Saturated Salts



Sirota, J. Chem Phys, 1996

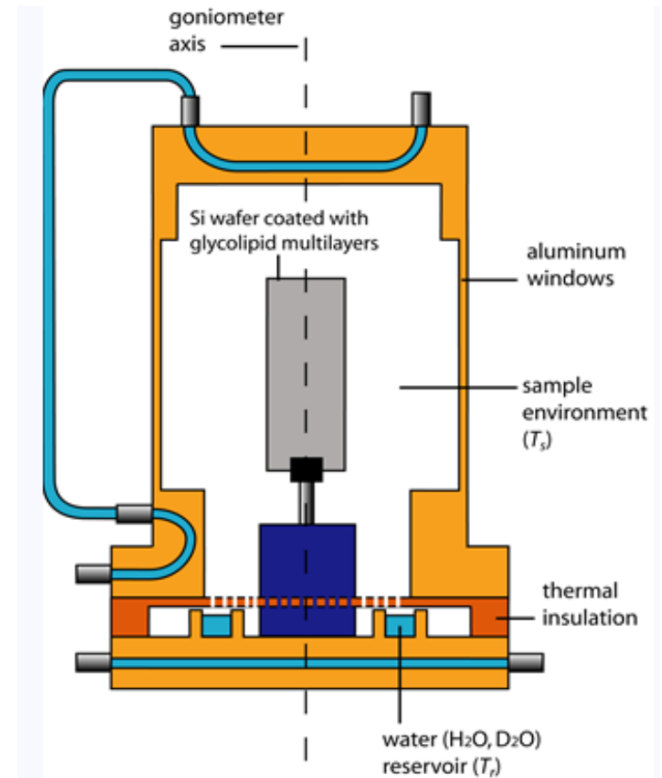


Temperature Controlled Water bath



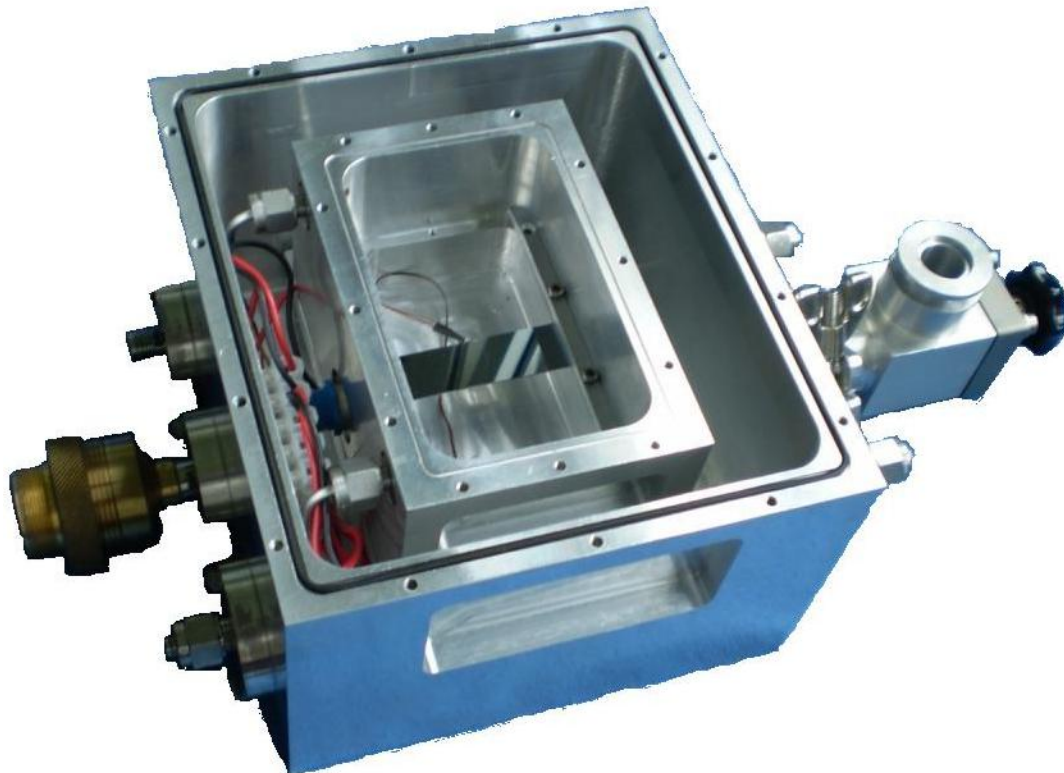
Rheinstädter, 2012

INDIVIDUAL COMPONENTS ADJUSTABLE T
 H₂O/D₂O EXCHANGE
 CONDENSATION
 100% RH?
 LIMITED BY SENSOR ACCURACY



Schneck, *Phys. Rev. E*, 2008

Reflectometer cell for gas adsorption in thin films and multi-layers structures.



Peltier controlled temperatures from 250 K to 350 K at pressures up to 10 bars.

COMPLEMENTARY DEGAS LABORATORY (GASLAB)

- **Sample characterization** by volumetric and gravimetric gasadsorption
 - > surface area, porosity, pore volume, etc.
- **Sample preparation**
 - > cleaning (degassing), precharging, etc.
- **Experiment prearrangement**
 - > encapsulating under inert gas conditions
 - > „dry-runs“ under neutron scattering conditions



Automated Volumetric Gas adsorption station combined with pulse tube cooler



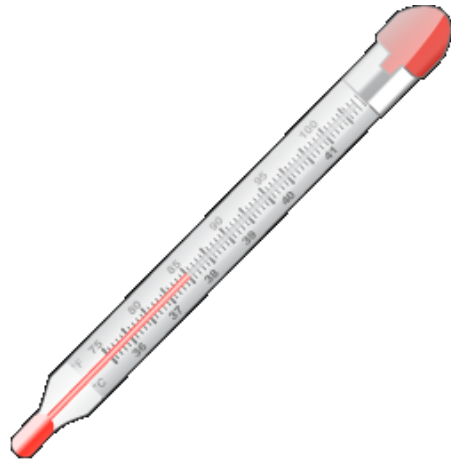
RUBOTHERM
Magnetic suspension
balance



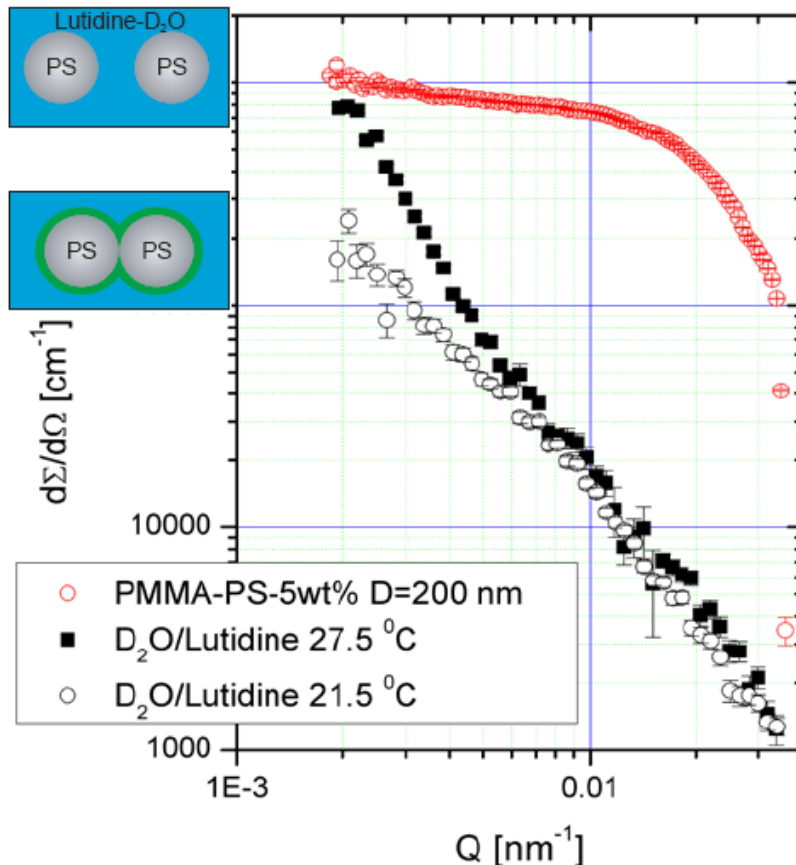
Helium Glove Box



Cryocooler for
Gasadsorption at low
Temperatures



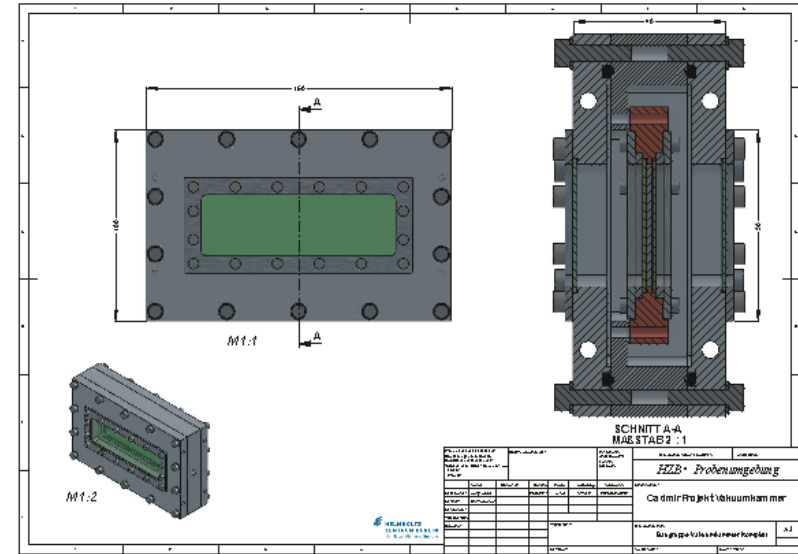
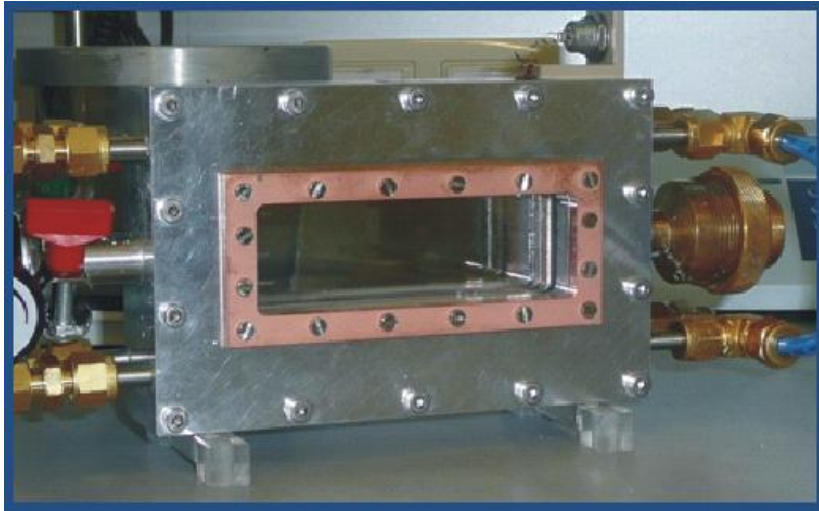
Temperature Cells



- a soft-matter analogue of the quantum-electrodynamic Casimir effect
- critical phenomenon near transition point to continuous phase
- Q^{-2} vs. Q^{-4} -behaviour for different regimes
- concentration fluctuations near critical point become visible at low Q -values
- to trace critical behaviour, accurate adjustment of temperature is required

V-SANS curves of D₂O-Lutidine mixtures at different temperatures as well as of of PMMA-PS-particles

USANS Sample Cell for Liquid Samples "CASIMIR-CELL"



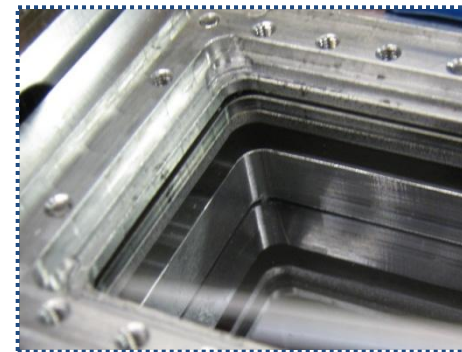
250 K < T < 350 K

Stability and Homogeneity +/- 5mK

Window Size 130 mm x 30 mm

Volume 1ml - 6 ml

completely dismountable



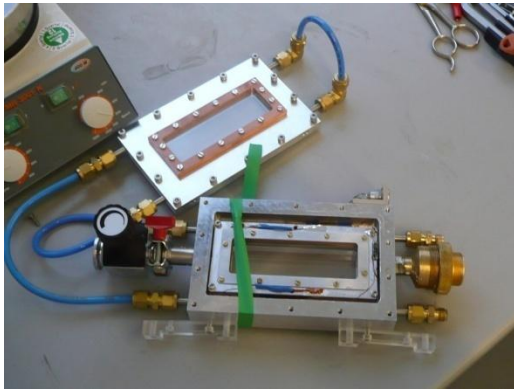
sapphire windows:

(chemo-mechanic polished)

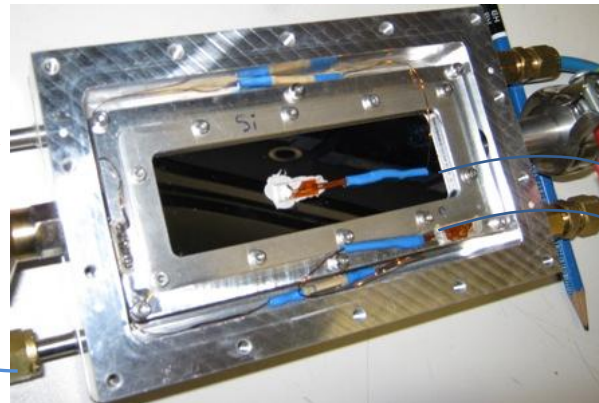
Surface roughness 0.25 nm, Pollerscharge 3 nm

Two independent systems:

- outer circuit for „rough“ adjustment
- inner electric device for fine tuning



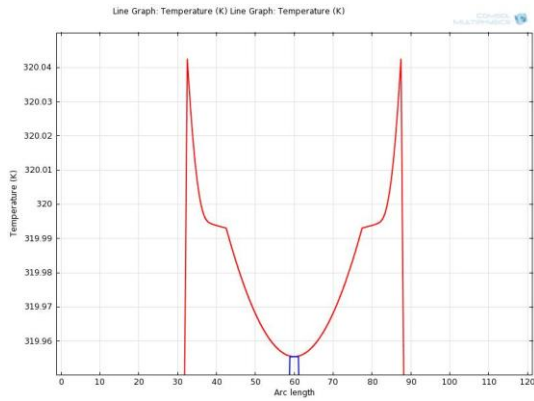
H₂O/oil



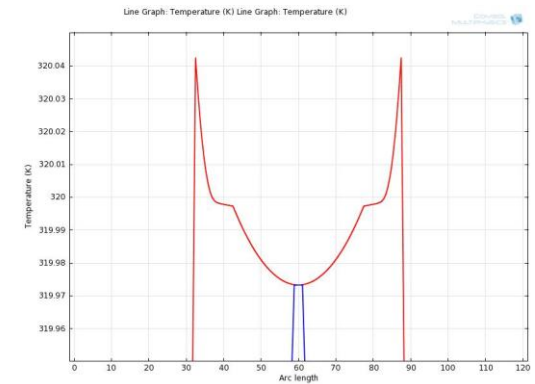
$$250 \text{ K} < T_i < 350 \text{ K}$$

+/- 0.005 K (relative scale)

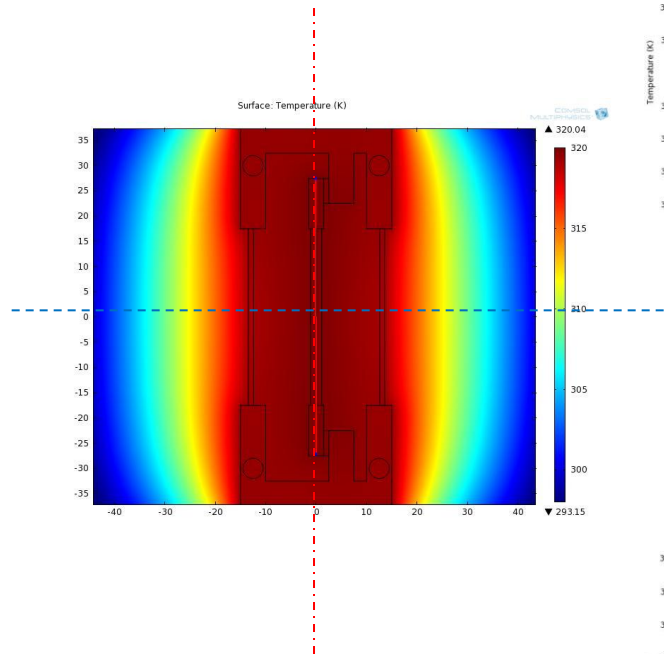
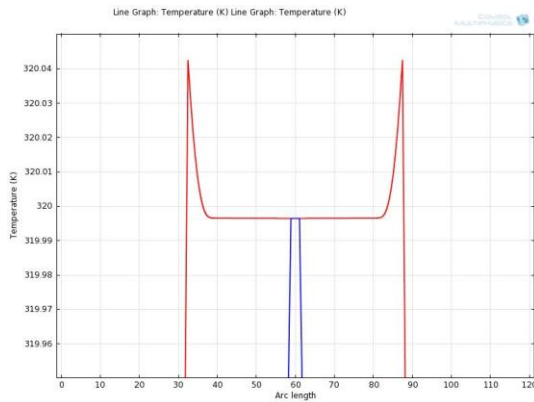
Gas (o) & Gas (i)



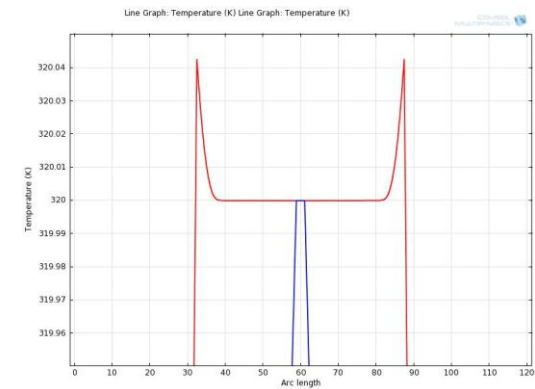
Vak (o) Gas (i)



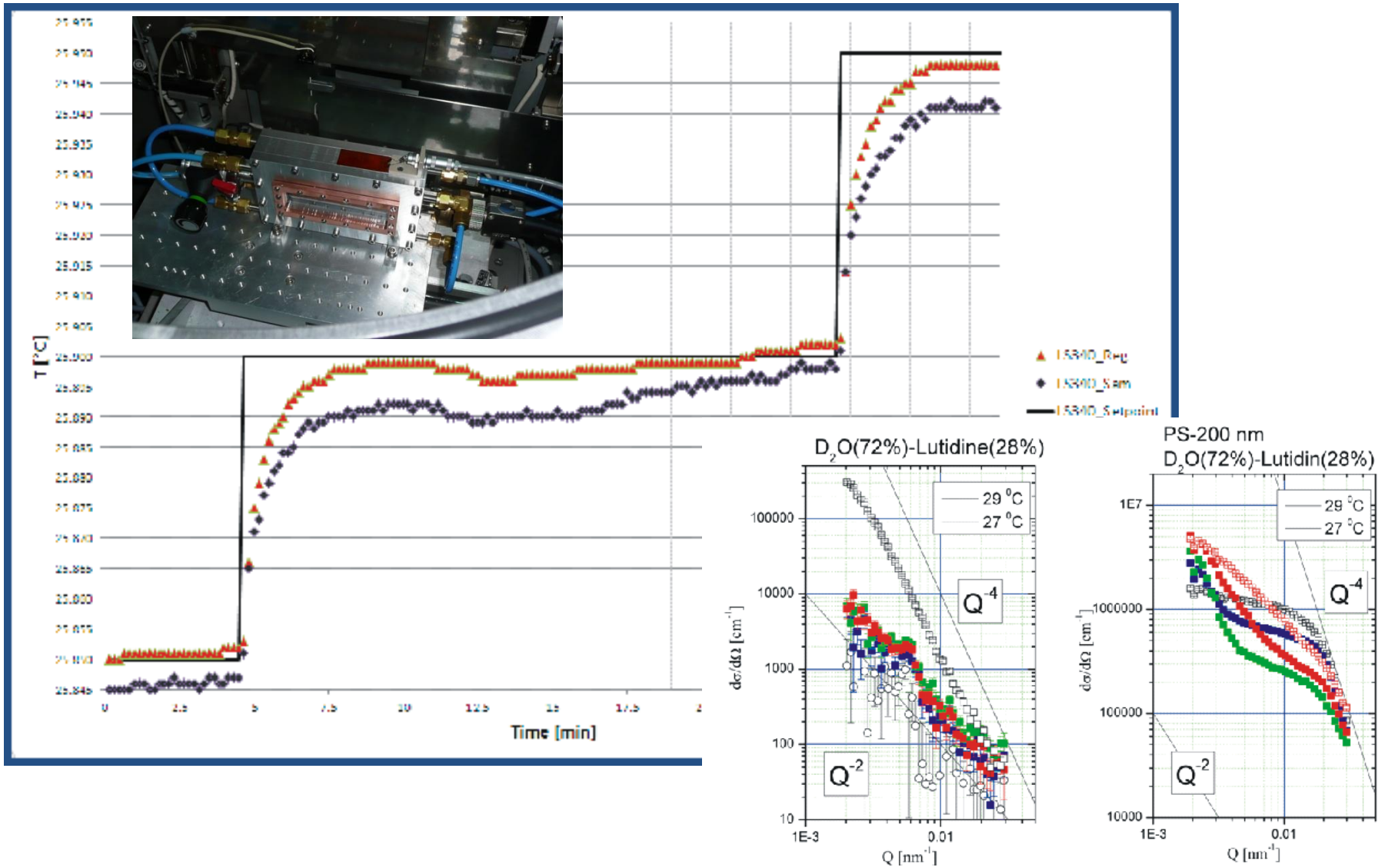
Gas (o) & Vak (i)



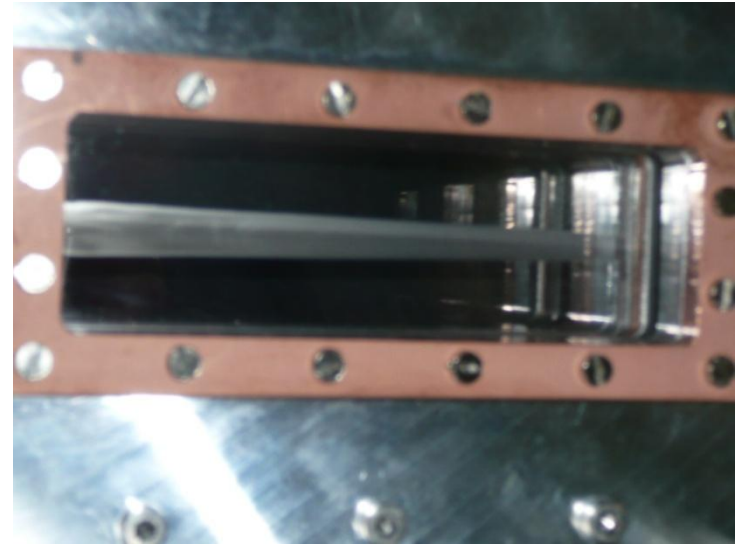
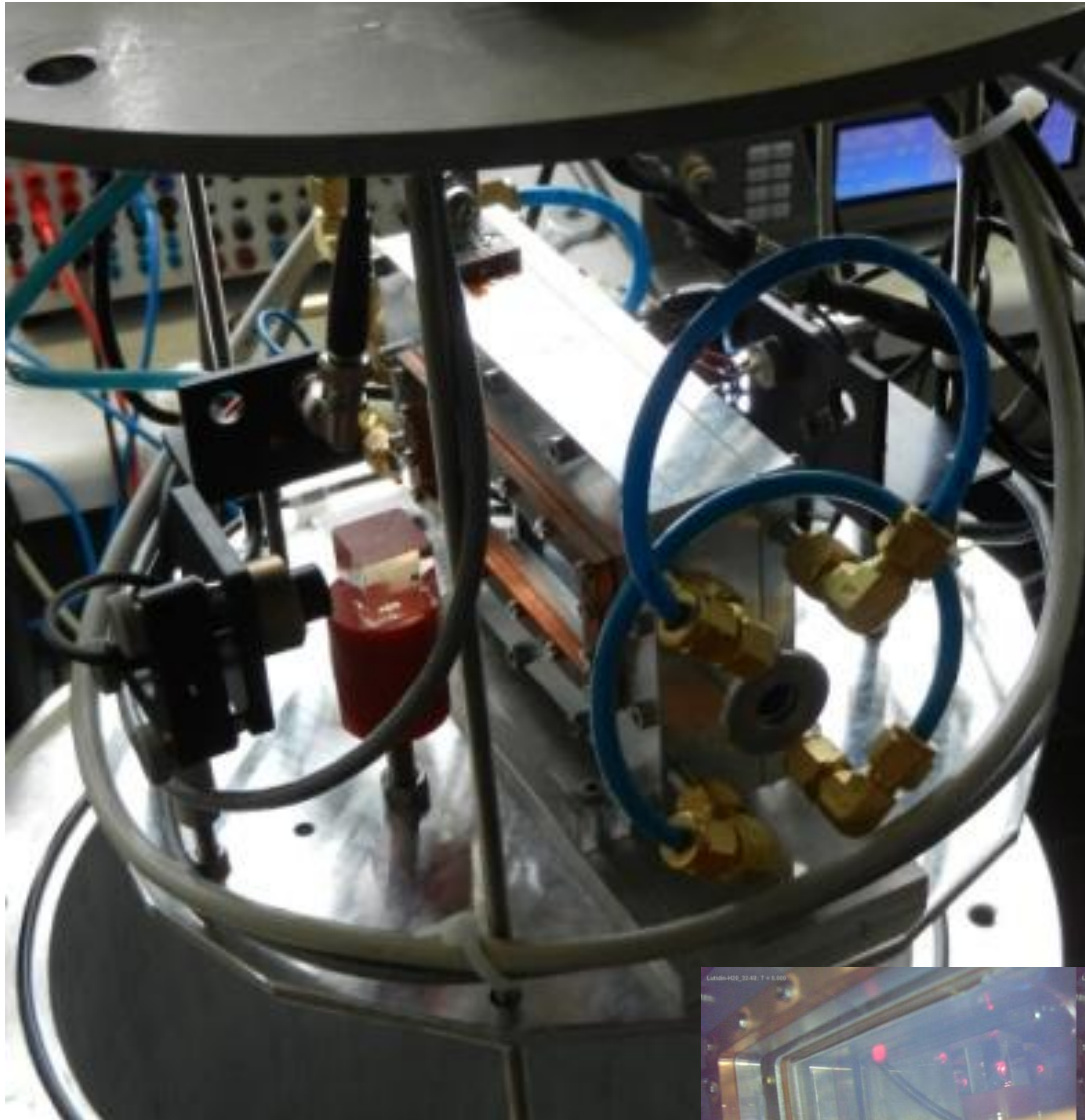
Vak (o) & Vak (i)



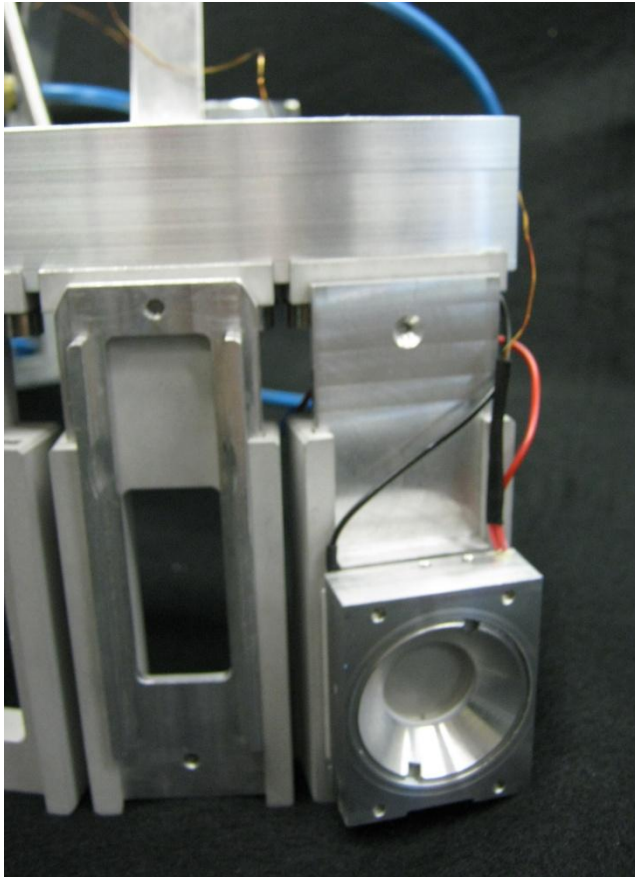
USANS Sample Cell for Liquid Samples



V-SANS curves of D₂O-Lutidine mixtures in absence and presence of polystyrene particles at temperatures between 27 and 29 °C.

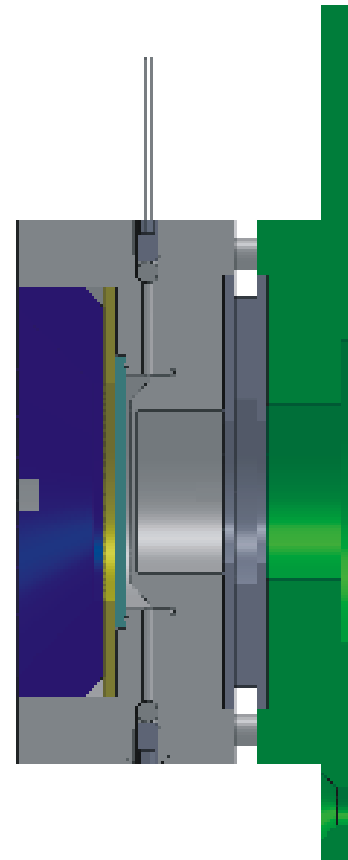


SANS Cell for Liquid Samples

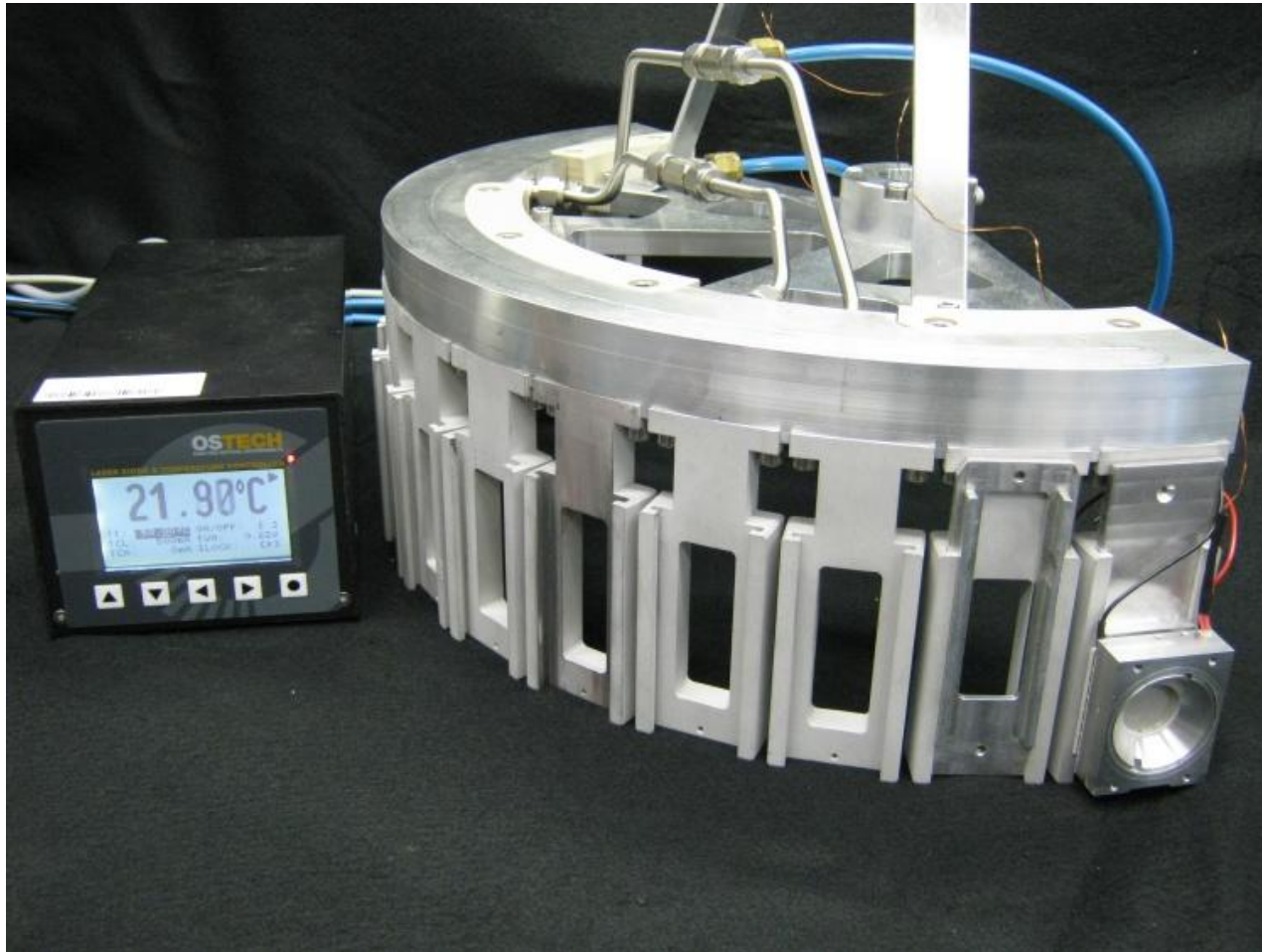


$250\text{ K} < T < 350\text{ K}$
Stability $\pm 5\text{ mK}$

Diameter 13 mm



SANS Sample Cell for Liquid Samples



Outlook

- **(stopped) flow cells**
- **high pressure (6 kbar) SANS cells**
- **improved humidity chamber with sample changer**
- **in-situ light scattering cell**