Sample Environment JRA-Meeting

Progress Report October 2012

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JRA-Sample Environment Meeting

Berlin (HZB)

22nd October 2012

Sample Environment JRA-Meeting

Task 21.4: Development of Gas Adsorption Control Systems for Neutron Scattering Instruments

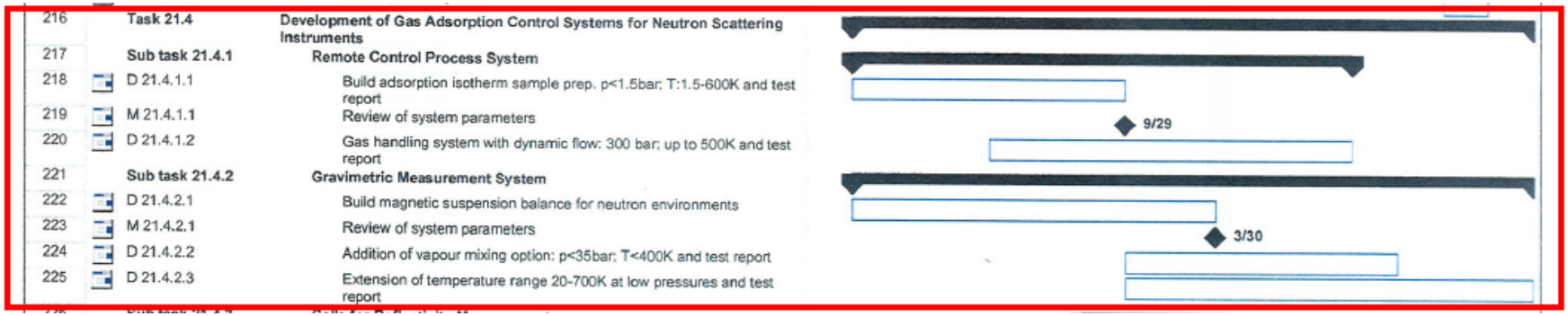
The measurement of hydrogen storage materials, as well as the characterisation of chemical and catalyst reactions in porous materials, is of significant interest. All facilities have some capability to work in this area but the aim of this task is to significantly extend this to allow real-time in-situ measurements of many diverse chemical and physical phenomena.

We plan to start by developing a volumetric low pressure (<1.5 bar) gas adsorption measurement system for experiments in an Orange cryofurnace (1.5-600 K) and, alternatively, in a cryogen-free miniature pulse tube refrigerator (50-600 K). These systems will then be enhanced by further developments to extend the temperature and pressure ranges up to 300 bar at 200°C. The gas control systems will also be increased to provide mass spectroscopy and constant pressure and flow conditions. Finally, a system with a magnetic gravimetric system with a pressure range up to 100 bar and a temperature range up to 500°C will also be delivered.

Deliverables

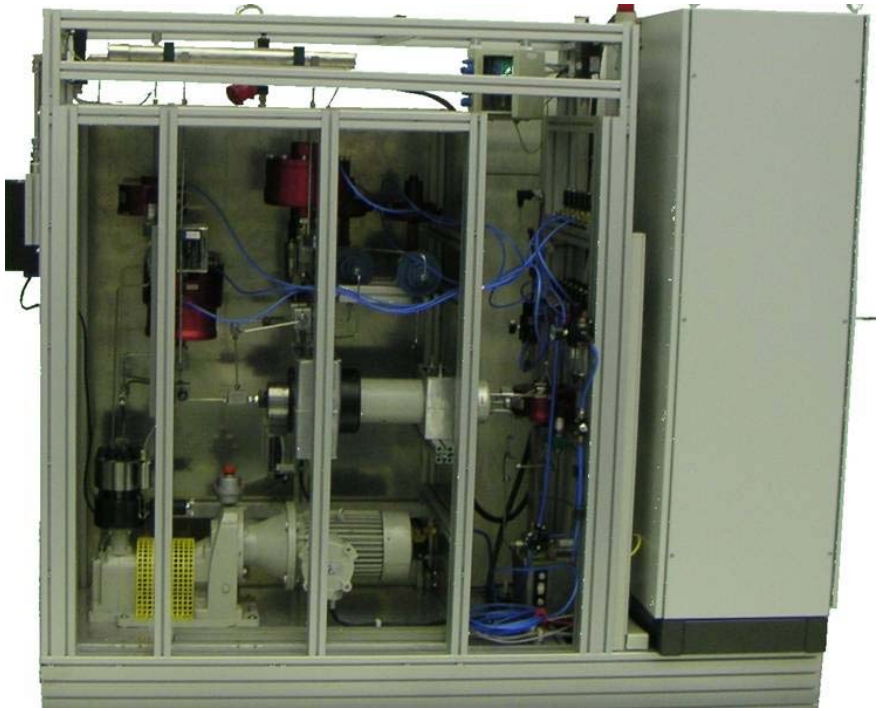
Task 21.4: Development of Gas Adsorption Control Systems for Neutron Scattering Instruments

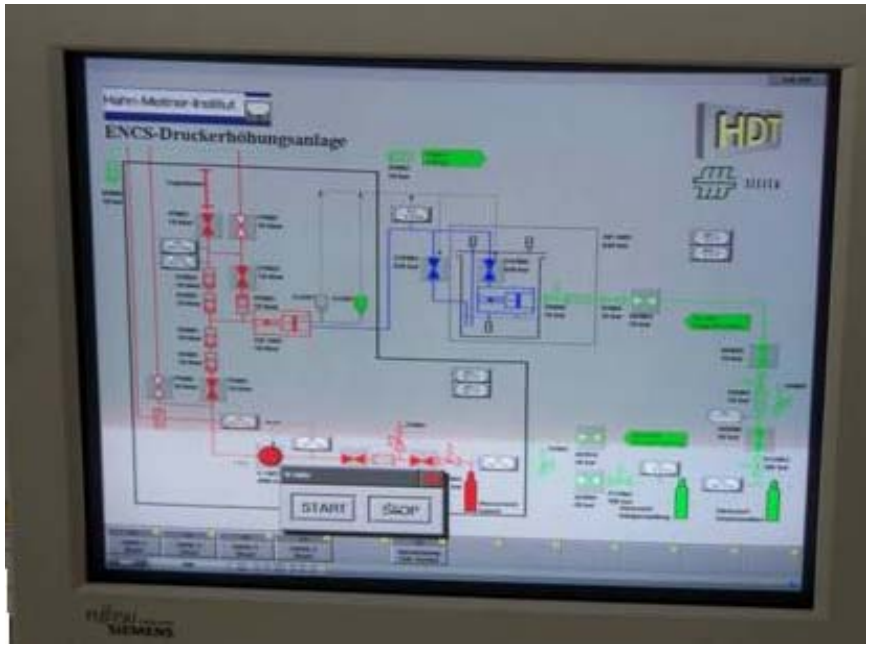
D 21.2.2.2 Commissioning of HMI 10 kbar H2 handling system (12)
D 21.4.1.1 Build adsorption isotherm sample prep. p<1.5bar; T:1.5-600K (21)
D 21.4.1.2 Gas handling system with dynamic flow: 300 bar; up to 500K (36)
D 21.4.2.1 Build magnetic suspension balance for neutron environments (27)
D 21.4.2.2 Addition of vapour mixing option: p<35bar; T<400K (39)
D 21.4.2.3 Extension of temperature range 20-700K at low pressures (48)



216	Task 21.4	Development of Gas Adsorption Control Systems for Neutron Scattering Instruments	
217	Sub task 21.4.1	Remote Control Process System	
218	D 21.4.1.1	Build adsorption isotherm sample prep. p<1.5bar; T:1.5-600K and test report	
219	M 21.4.1.1	Review of system parameters	9/29
220	D 21.4.1.2	Gas handling system with dynamic flow: 300 bar; up to 500K and test report	
221	Sub task 21.4.2	Gravimetric Measurement System	
222	D 21.4.2.1	Build magnetic suspension balance for neutron environments	
223	M 21.4.2.1	Review of system parameters	3/30
224	D 21.4.2.2	Addition of vapour mixing option: p<35bar; T<400K and test report	
225	D 21.4.2.3	Extension of temperature range 20-700K at low pressures and test report	

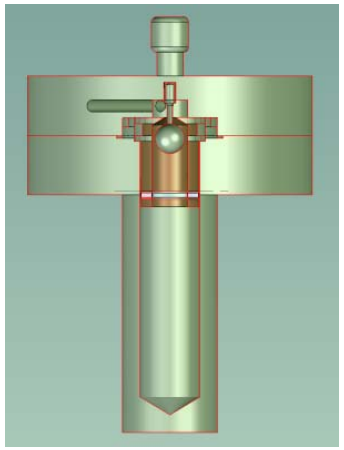
D 21.2.2.2 Commissioning of HMI 10 kbar H_2 handling system





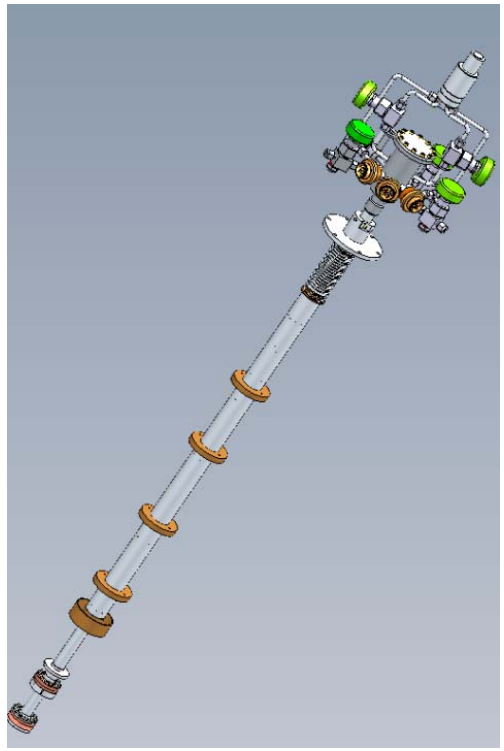
D 21.4.1.1 Build adsorption isotherm sample prep. p<1.5bar; T:1.5-600K

OF-Adsorption-Stick (1.5K - 600K, 300bar) with Al- sample cells



d_a (mm) 300 K		400 K	500 K	600K
12	300 bar	100 bar	10 bar	1 bar
14	300 bar	200 bar	50 bar	20 bar
20	300 bar	300 bar	300 bar	300 bar

Improved design by ILL



Cyogenfree Sorption Systems (10K – 600K)

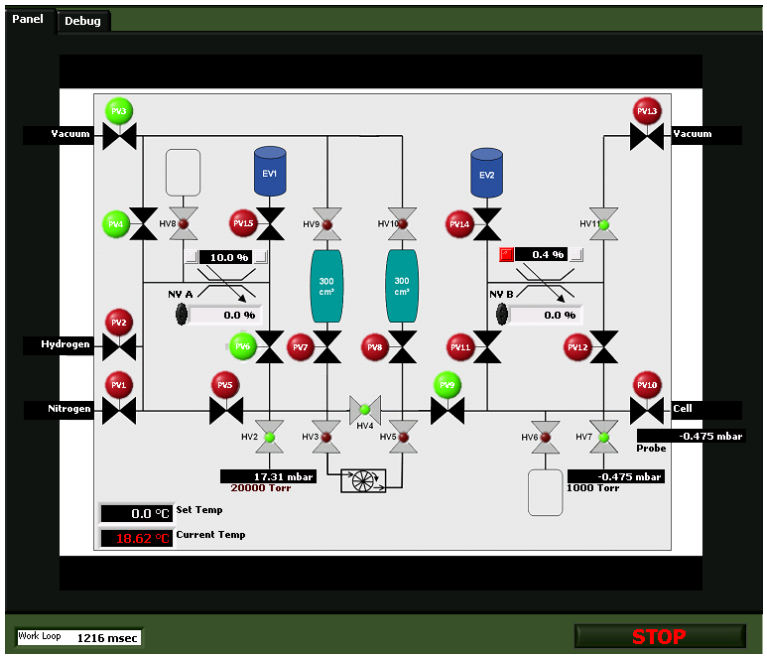
Mini-Pulsetube

Gifford-McMahon

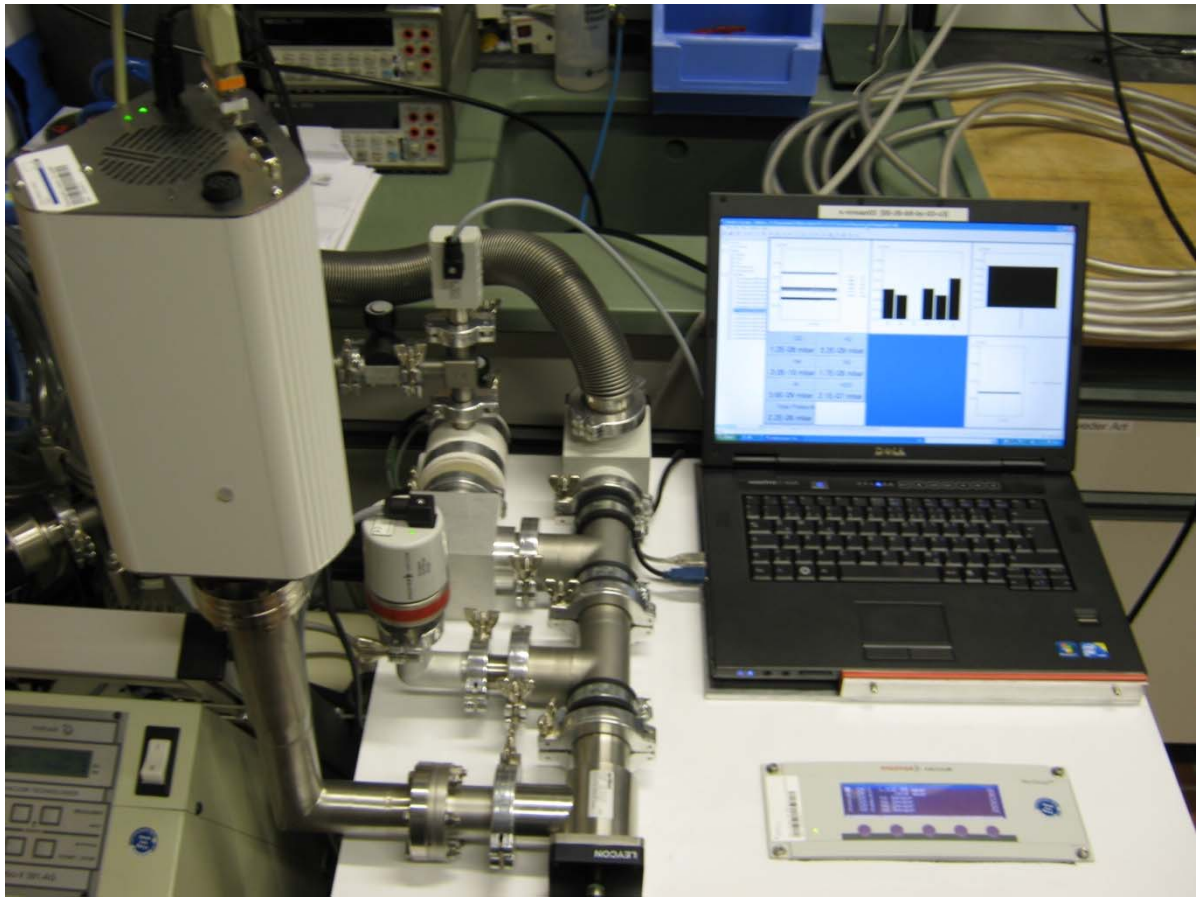
High temperature stage 800K

D 21.4.1.2 Gas handling system with dynamic flow: 300 bar; up to 500K

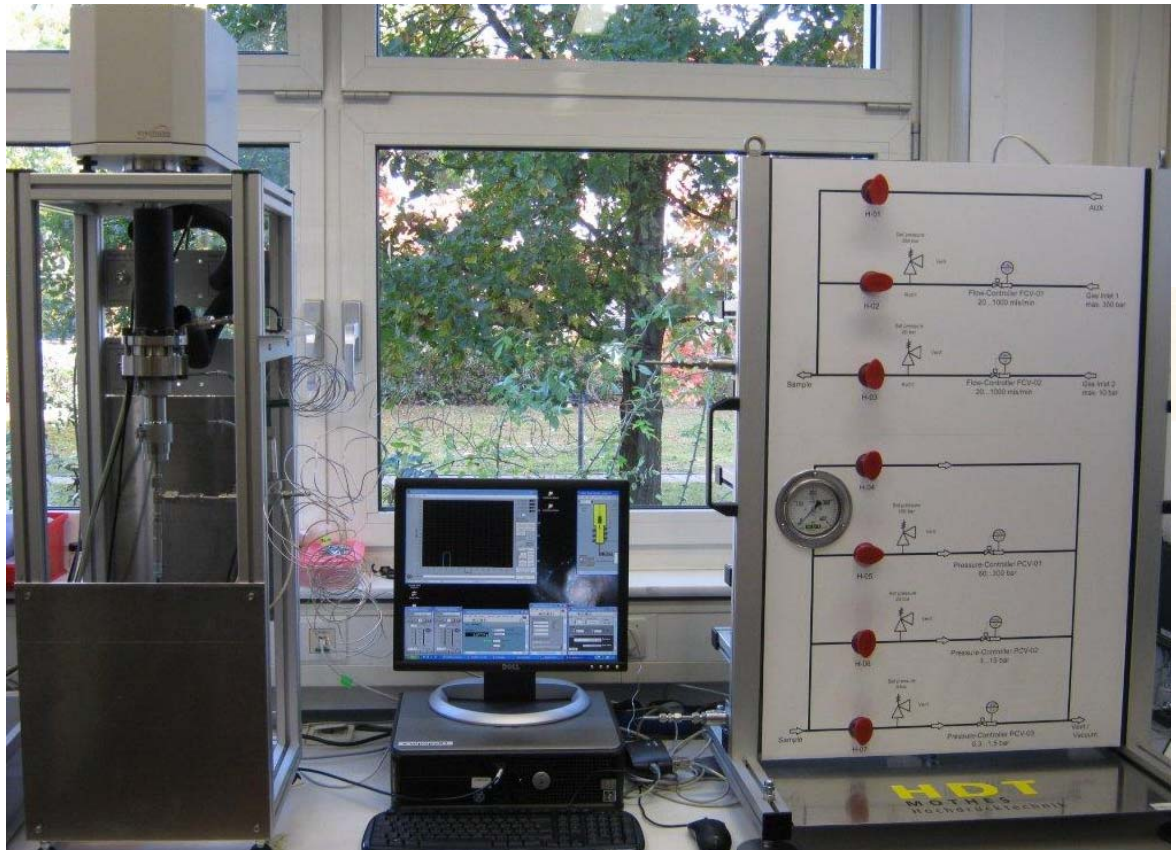
(in user service since August'12)



Residual gas analysis station

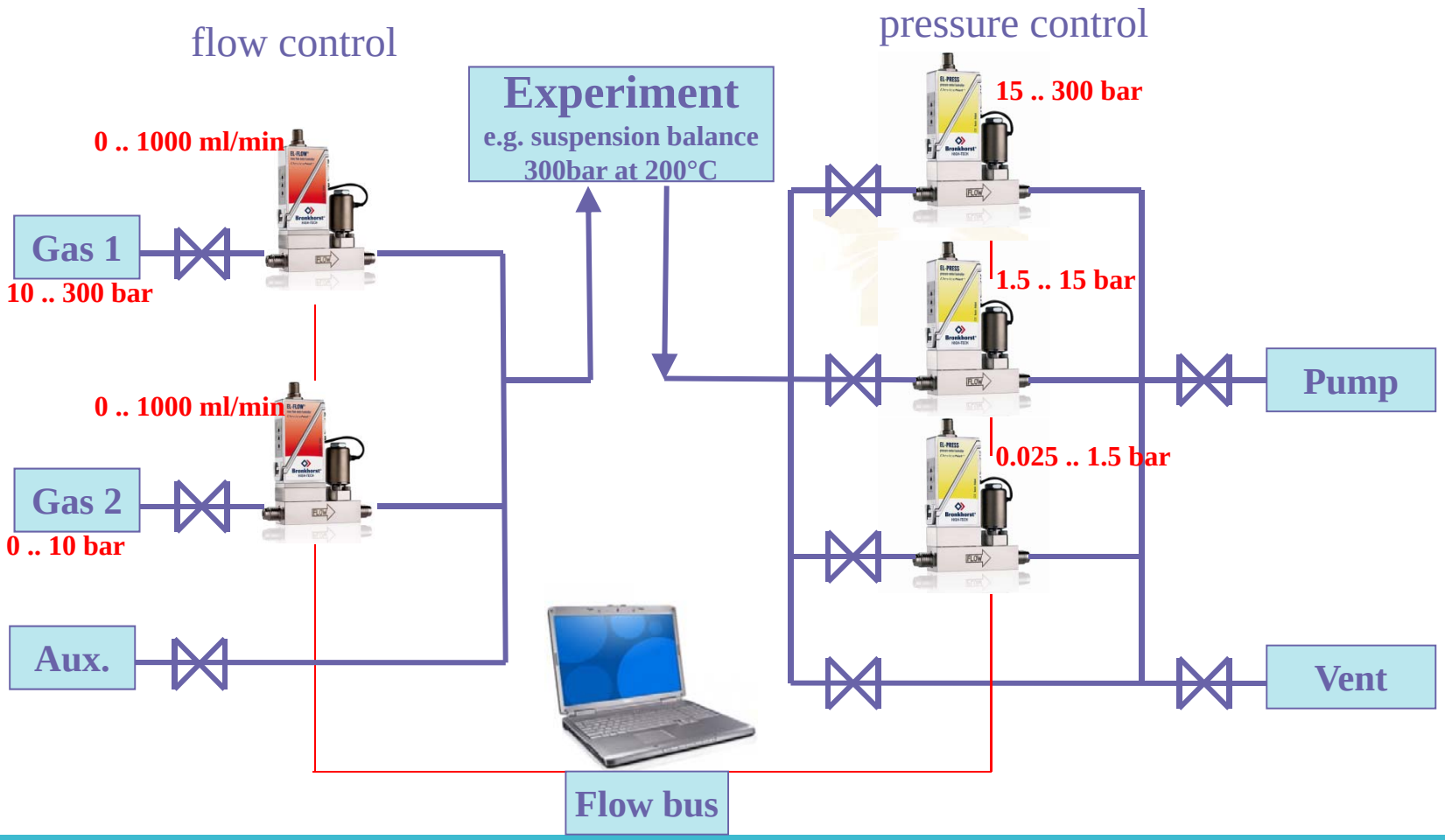


D 21.4.2.2 Addition of vapour mixing option: p<35bar; T<400K

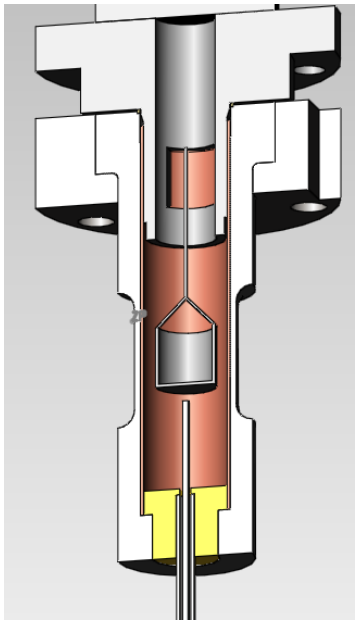


Gas handling system 300 bar – continuous flow

improved pressure and flow control at ambient temperatures



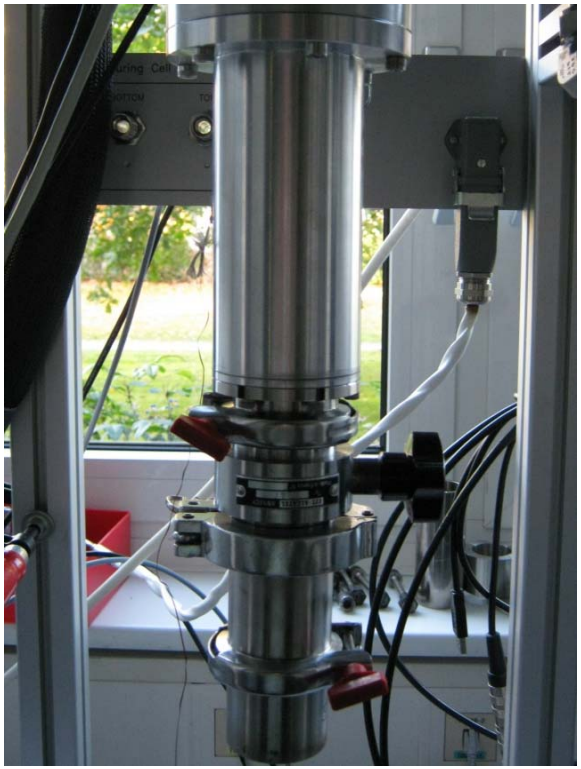
D 21.4.1.1 Build adsorption isotherm sample prep. p<1.5bar; T:1.5-600K



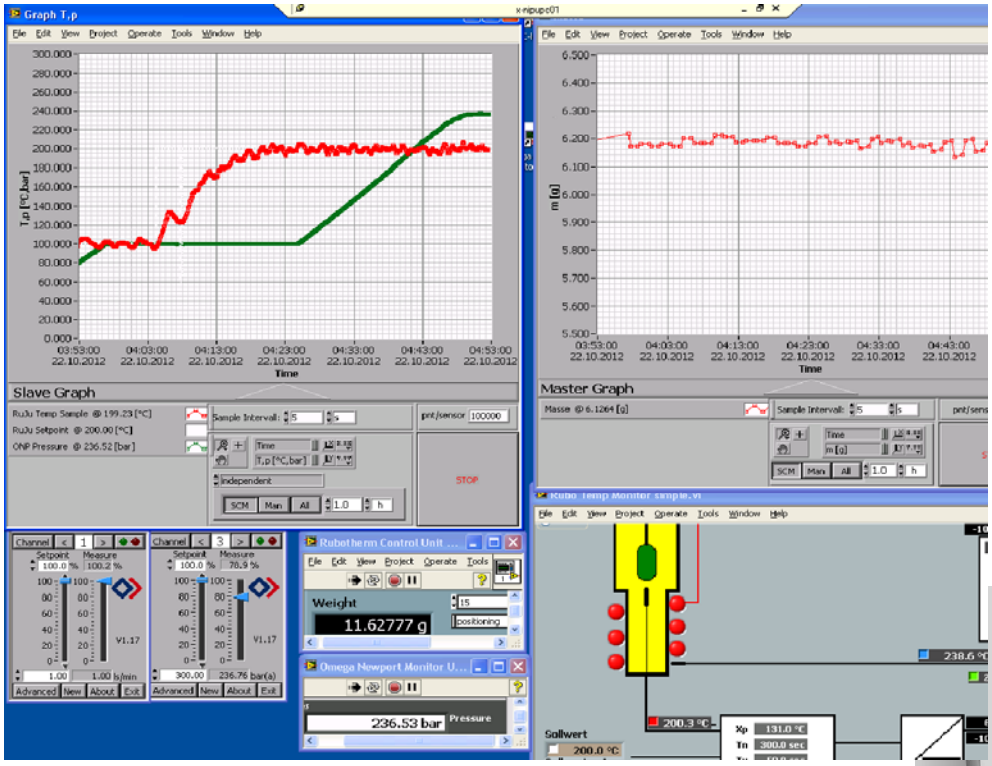
- T = 500 C / P = 100 bars
- T = 200 C / P= 300 bars
- hydrogen resistant

High Pressure/temperature Cell

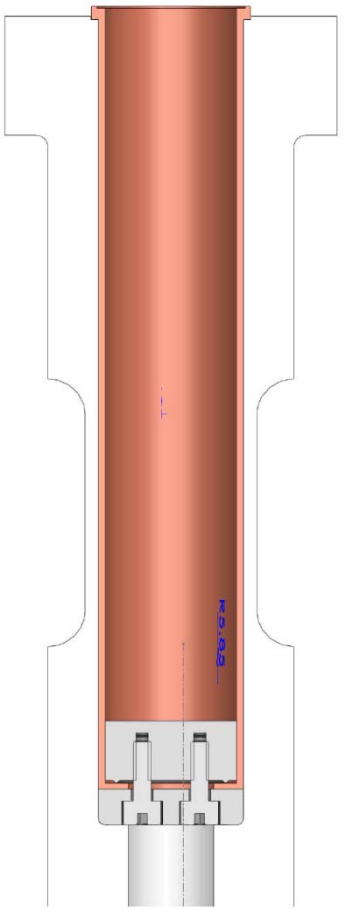
Humidity Option



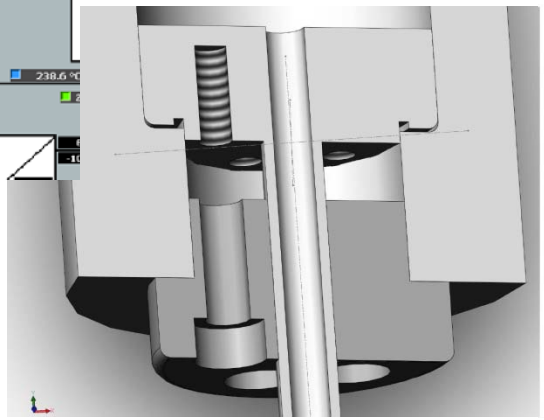
First Tests: Leakage at 240 bar / 200°C



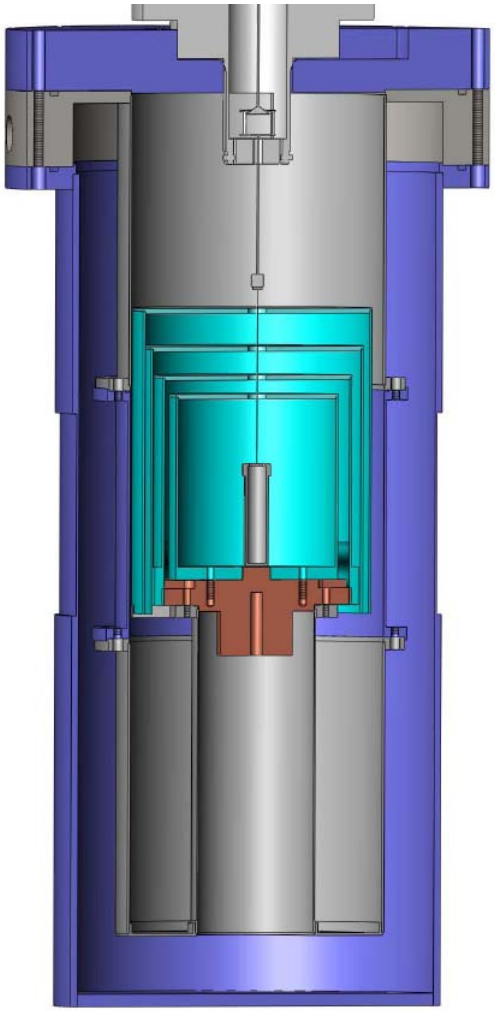
H2-Protection-Liner



Inert gas Seal



D 21.4.2.3 Extension of temperature range 20-700K at low pressures



- Design finished
- Parts under ordered
- Assembly under construction

- Scheduled for end of November‘12

Deliverables

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Outstanding parts:

- Report gashandling
- Sealing and testing of HTP-option for balance
- Low temperature expansion balance

Thanks for your attention

and

Nico Grimm