

Gas Adsorption Control Systems (WP 21.4)

WP 21.4.1 Adsorption Isotherm Sample Preparation System

- OF-Adsorption-Insert (1.5K - 600K, 300bar) with sample cells
- *Cyrogenfree Sorption System (50K – 600K, 50bar) with sample cells*
- Automated Gas-dosing System (500K, 300bar) with dynamic flow option



WP 21.4.2 Gravimetric Measurement System

Nr.	Vorgangsname	Anfang	Ende												
				2009			2010			2011			2012		
				Hälfte 1, 2009	Hälfte 2, 2009		Hälfte 1, 2010	Hälfte 2, 2010		Hälfte 1, 2011	Hälfte 2, 2011		Hälfte 1, 2012	Hälfte 2, 2012	Hälfte 1, 2013
				J	F	M	A	M	J	J	A	S	O	N	D
1	21.4. Development of Gas Adsorption Control Systems for Neutron Scattering Instruments	So 01.02.09	Di 19.03.13												
2	21.4.1 Remote Control Process System	So 01.02.09	Di 31.01.12												
3	21.4.1.1 Build adsorption isotherm sample prep. p<1.5bar; 1.5K<T<600K	So 01.02.09	So 31.10.10												
4	CGA-OF-Insert 1.5K - 600K, 300bar	So 01.02.09	Mi 31.03.10												
5	Sample Stick	So 01.02.09	Di 29.12.09												
6	Sample Cells	Fr 03.07.09	Do 31.12.09												
7	Test and report	Mi 30.12.09	Mi 31.03.10												
8	Pulse tube cooler 50K-600K	So 01.02.09	Sa 31.07.10												
9	Pulse tube refrigerator	So 01.02.09	Mi 30.09.09												
10	Sample cells	Mi 01.07.09	Di 29.12.09												
11	High temperature option	Di 01.12.09	Do 29.04.10												
12	Test and report	Sa 01.05.10	Sa 31.07.10												
13	MS: Review of system parameters	Sa 31.07.10	Sa 31.07.10												
14	Documentation	So 01.08.10	So 31.10.10												
15	Deliverable (21 M)	So 31.10.10	So 31.10.10												
16	21.4.1.2 Build Gas handling System with dynamic flow: 300bar, up to 500K	So 01.11.09	Di 31.01.12												
17	Volumetric Option	So 01.11.09	Fr 01.04.11												
18	Assembling	So 01.11.09	Mo 27.09.10												
19	Calibration	Mo 21.06.10	Fr 31.12.10												
20	Software	Mo 21.06.10	Fr 31.12.10												
21	Test and report	Fr 31.12.10	Fr 01.04.11												
22	Connection to cryostat	Mo 02.11.09	Mi 30.06.10												
23	Tubes up to 600K	Mo 02.11.09	Mi 31.03.10												
24	Capillaries T<300K	Mo 02.11.09	Mi 31.03.10												
25	Test and report	Do 01.04.10	Mi 30.06.10												
26	Continuous Flow Option	Fr 01.10.10	Fr 28.10.11												
27	Flow Controller Software	Fr 01.10.10	Do 24.03.11												
28	Pressure Controller	Fr 01.10.10	Fr 31.12.10												
29	Restgas analysis	Fr 01.10.10	Do 28.07.11												
30	Test and report	Mo 01.08.11	Fr 28.10.11												
31	Documentation	Di 01.11.11	Sa 28.01.12												
32	Deliverable (36 M)	Di 31.01.12	Di 31.01.12												
33	21.4.2 Gravimetric Measurement System	So 01.02.09	Di 19.03.13												
34	21.4.2.1 Build magnetic suspension balance for neutron	So 01.02.09	Sa 30.04.11												
35	Improve Sample Positioning	So 01.02.09	Fr 01.01.10												
36	Ambient Temperature Option, 10bar	Mo 28.12.09	Fr 05.11.10												
37	High Temperature Option 500°C, 10bar	Mo 28.12.09	Fr 05.11.10												
38	Test and report	Mo 01.11.10	Do 27.01.11												
39	Review of system parameters	Mo 31.01.11	Mo 31.01.11												
40	Documentation	Di 01.02.11	Di 26.04.11												
41	Deliverable (27 M)	Sa 30.04.11	Sa 30.04.11												
42	21.4.2.2 Addition of vapour mixing option: p<35bar; T<400K	So 01.08.10	Mo 30.04.12												
43	Build Gas handling	So 01.08.10	Fr 01.07.11												
44	Software	So 01.05.11	Mo 07.11.11												
45	Test and report	Mo 14.11.11	Fr 03.02.12												
46	Documentation	Mi 01.02.12	Fr 27.04.12												
47	Deliverable (39 M)	Mo 30.04.12	Mo 30.04.12												
48	21.4.2.3 Extension of temperature range 20-700K at low pressures	Fr 31.07.09	Di 19.03.13												
49	Build cryostat	So 01.08.10	Fr 10.08.12												
50	Test and report	Mi 01.08.12	Mi 24.10.12												
51	Documentation	Do 01.11.12	Mo 28.01.13												
52	Deliverable (48 M)	Do 31.01.13	Do 31.01.13												

WP 21.4.1 REMOTE CONTROLLED GAS ADSORPTION SYSTEMS

Temperature Environment

Cryo-Furnace Inserts

Closed Cycle Systems (stand alone)

Gas Handling

Volumetric Gas Dosing

Capillaries/Tubes

Flow Option

WP 21.4.2 GRAVIMETRIC MEASUREMENT SYSTEM

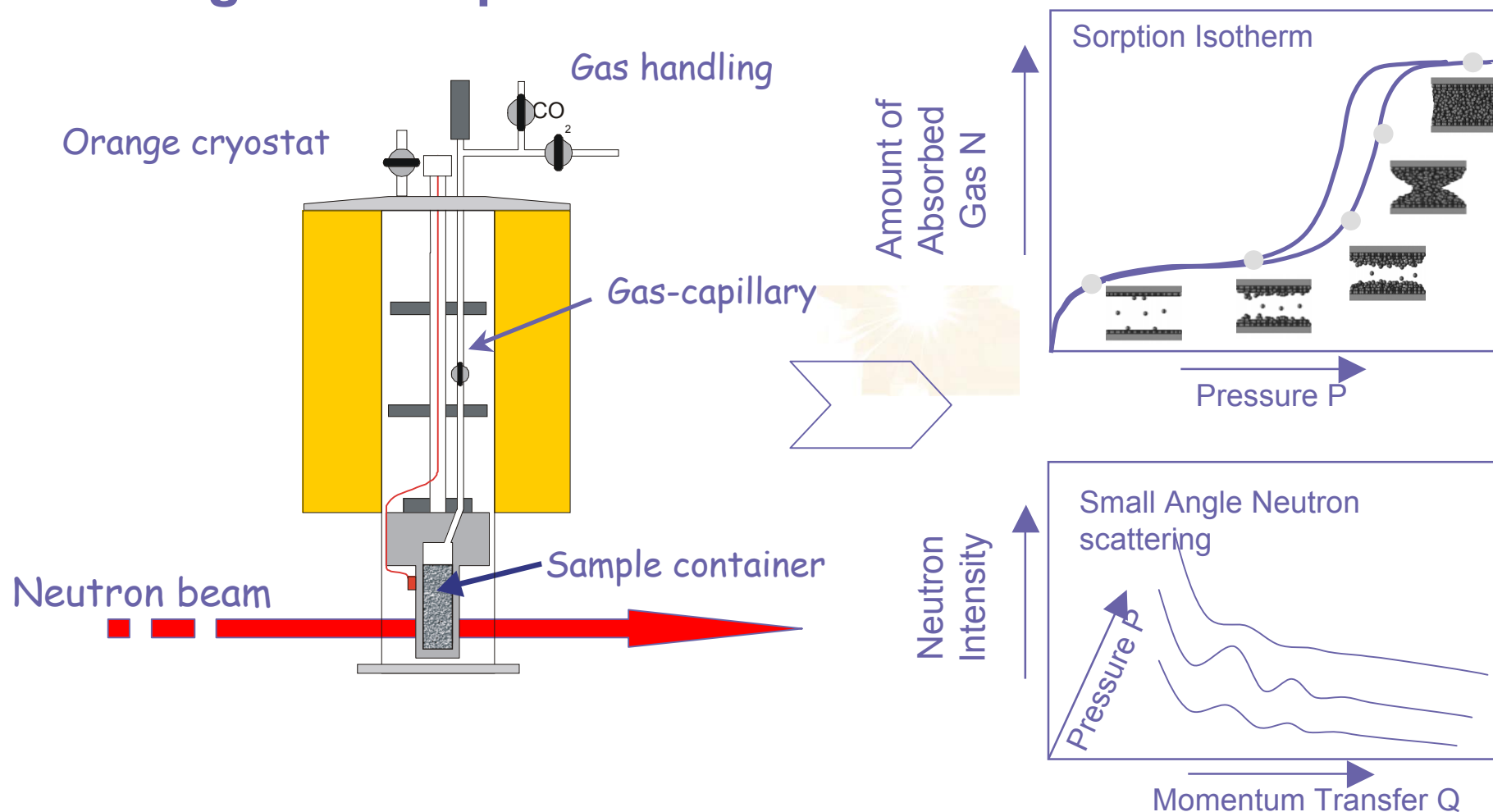
Suspension Balance

Vapour Mixing

Cryostat



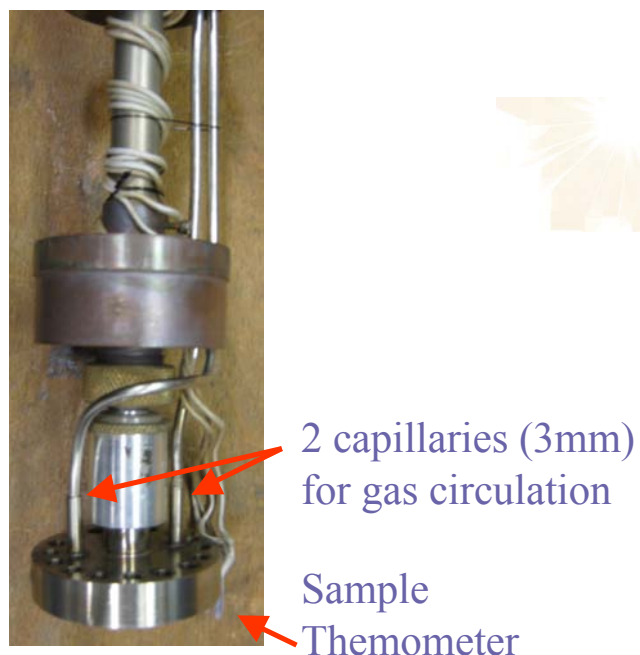
In-situ gas adsorption measurements



WP 21.4.1 REMOTE CONTROLLED GAS ADSORPTION SYSTEMS

21.4.1.1 Temperature Environment 1.5K – 600K, $p < 300$ bar

Cryo-Furnace Inserts (Samplestick)



Gas circulator
(magnetic coupled)



(Gardner Denver GK-M 02)

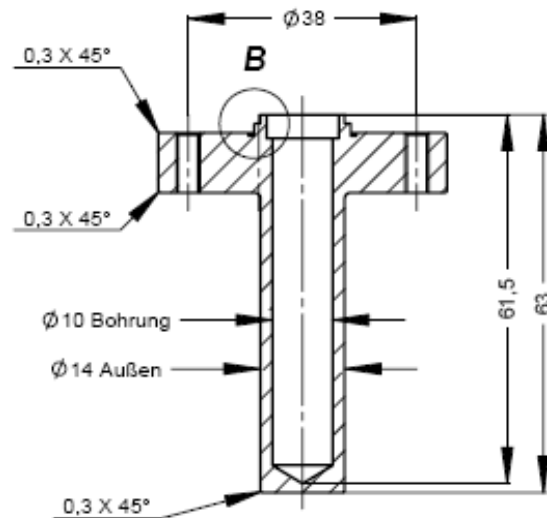
System pressure: 150 bar
Pressure difference: 75 mbar
Gas flow: 2.5 L/min

Capillary heating necessary from 77K to 295K !

Aluminium (AW 7075) Sample Cells

Tensile Strength: 450 MPa at 295K

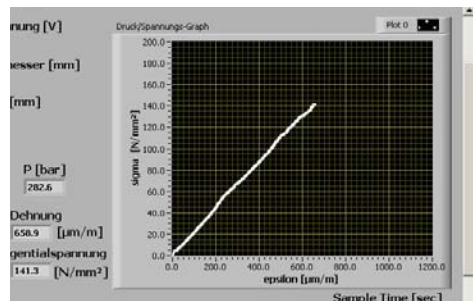
Sealing: 1mm In- or Pb- wire



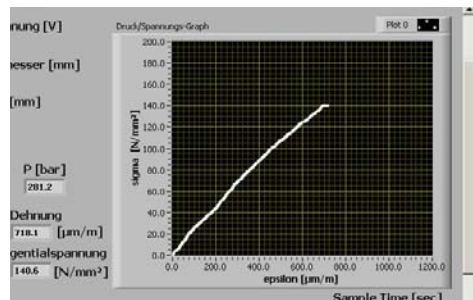
<u>d_a (mm)</u>	<u>300 K</u>	<u>400 K</u>	<u>500 K</u>	<u>600K</u>
12	300 bar	100 bar	10 bar	1 bar
14	300 bar	200 bar	50 bar	20 bar
20	300 bar	300 bar	300 bar	300 bar



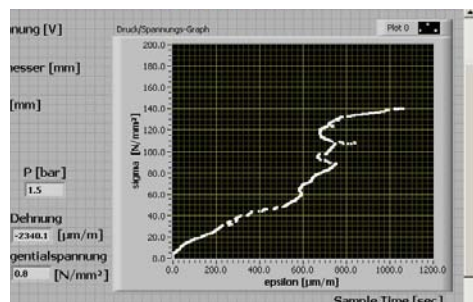
In-situ stress-strain-analysis



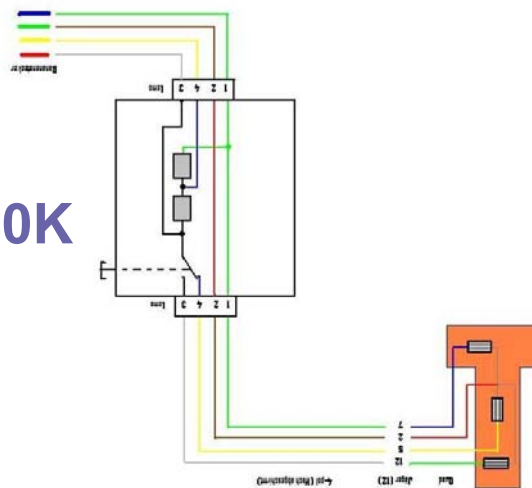
296K



340K



420K



$d_a = 12 \text{ mm}$, $p = 100 \text{ bar}$



WP 21.4.1 REMOTE CONTROLLED GAS ADSORPTION SYSTEMS

Accessories for Air Protected Sample Preparation

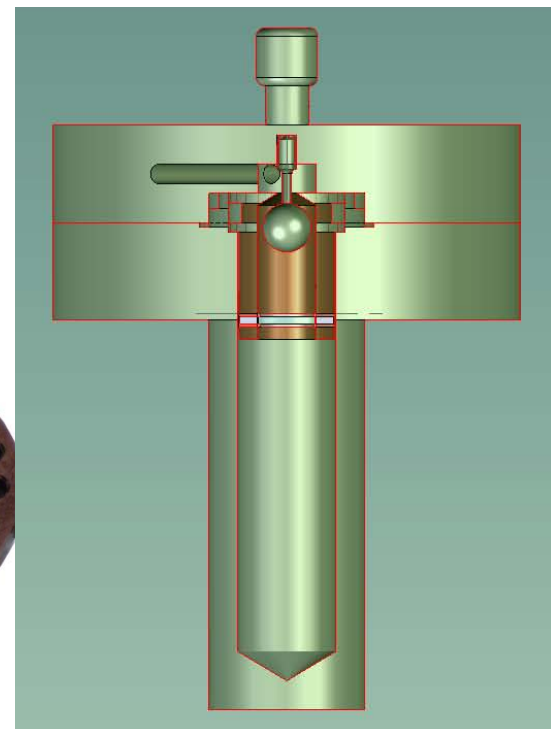
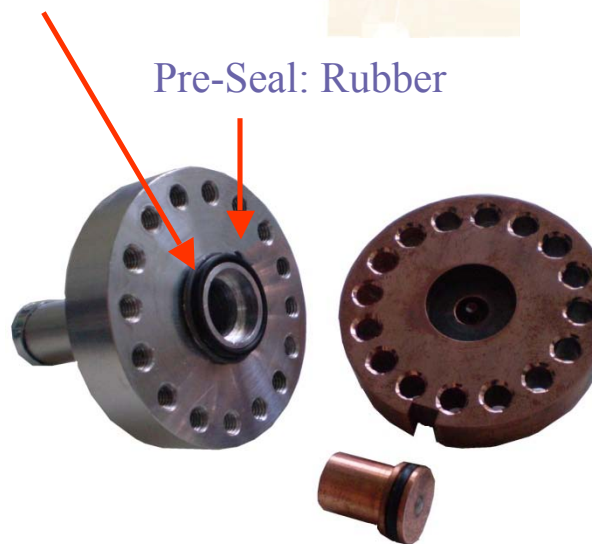
Sample-Stick sluice in Helium Glovebox



Cell Locking Device

Seals: In, Pb, Cu

Pre-Seal: Rubber



21.4.1.1 Temperature Environment 1.5K – 600K, $p < 300$ bar

• Closed Cycle Systems

cryogen free
no cold spot



Pulse Tube
55K .. 350K



Gifford McMahon
6K .. 600K



WP 21.4.1 REMOTE CONTROLLED GAS ADSORPTION SYSTEMS

21.4.1.1 Temperature Environment 1.5K – 600K, $p < 300$ bar

Closed Cycle Systems (Mini Pulse Tube Cooler)



400 mm



2 optimal capillaries



floating shield and copper link



High Temperatures

ARS-Saphire Adapter (600K)
for Gifford McMahon

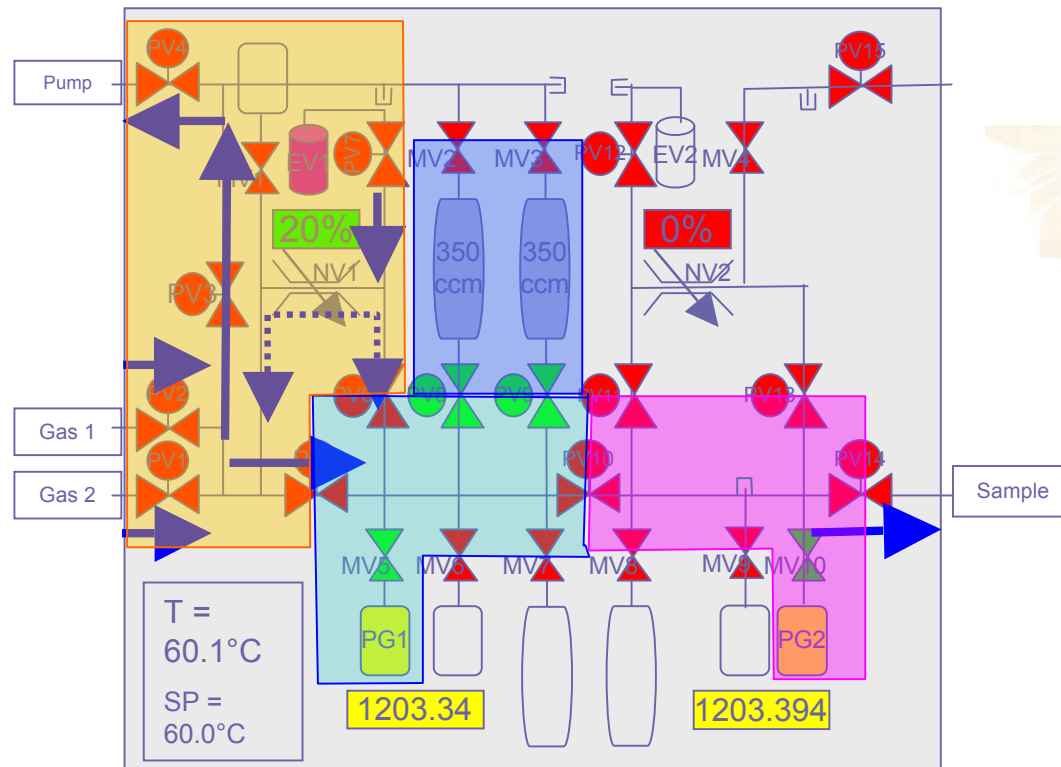


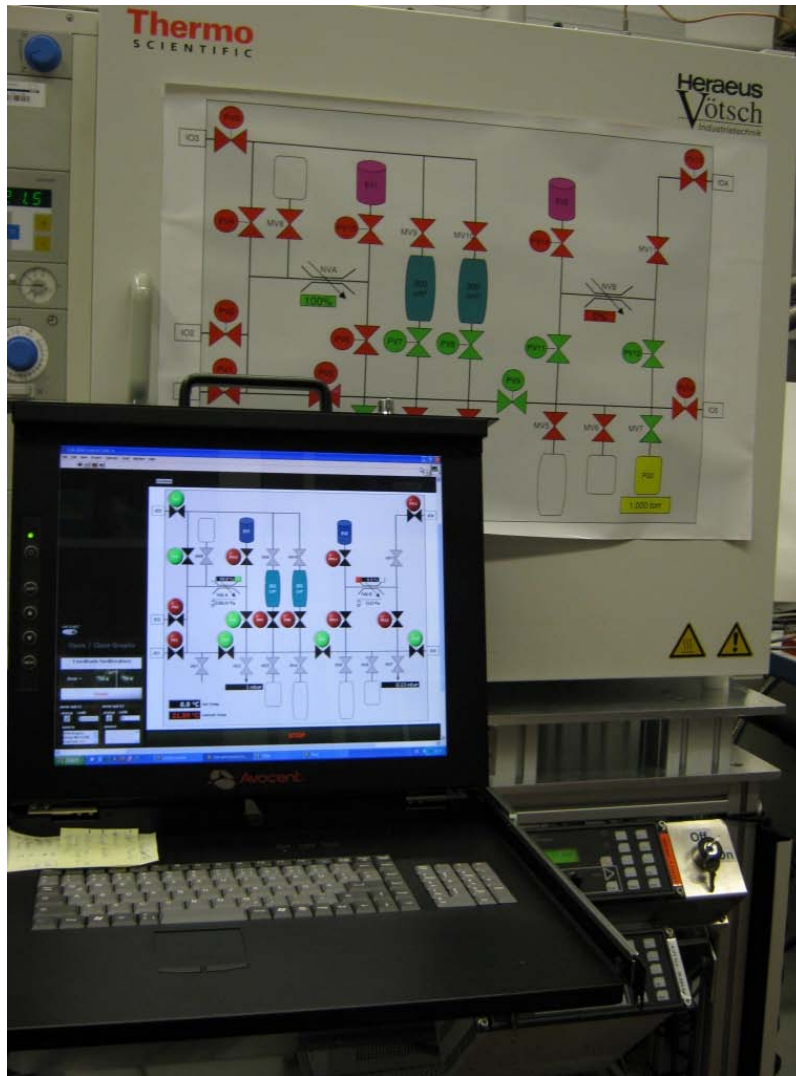
Redesigned 800K-Adapter for Pulse
Tube System



WP 21.4.1 REMOTE CONTROLLED GAS ADSORPTION SYSTEMS

21.4.1.2 Gashandling System 300 bar, 500 K Volumetric Gas Dosing

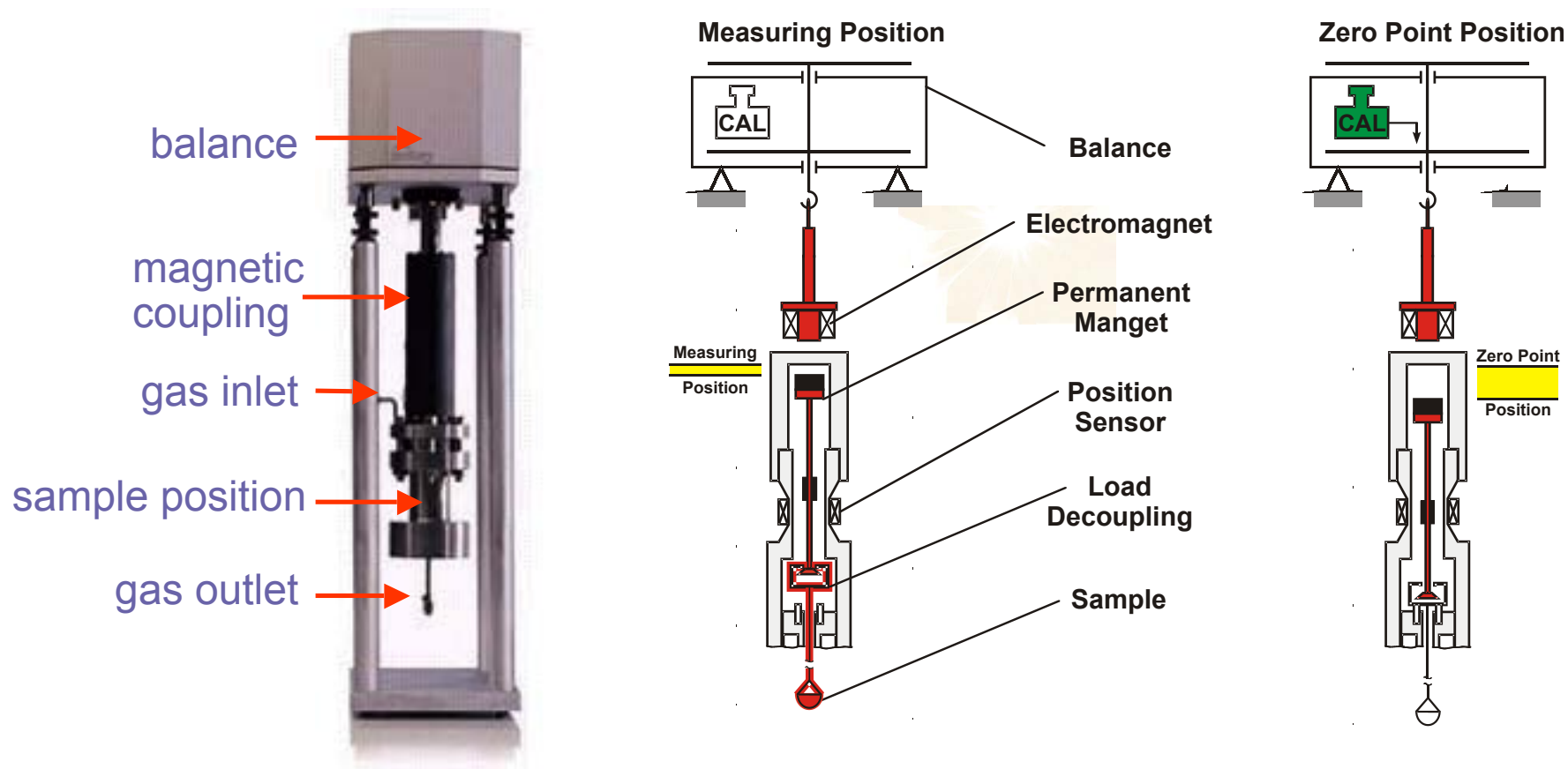




- Temperature dependent calibration of pressure gauges finished
- Software for automated Adsorption measurements is under construction

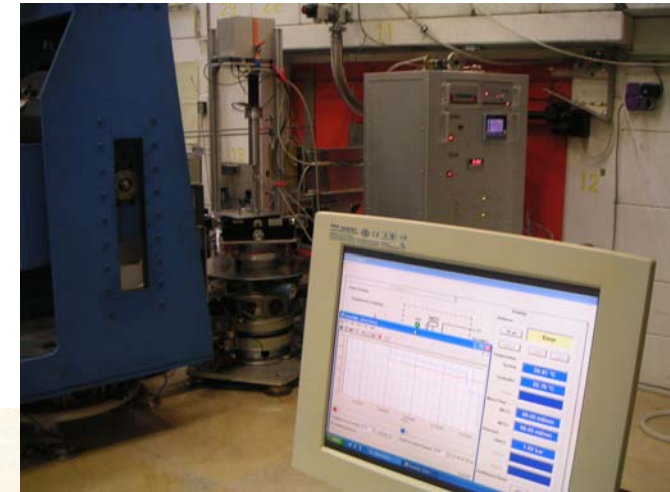
WP 21.4.2 Gravimetric Measurement System

21.4.2.1 Magnetic Suspension Balance for Neutron Scattering



1-10 μg accuracy of weighing under extreme conditions

Sample Repositioning for non powder samples

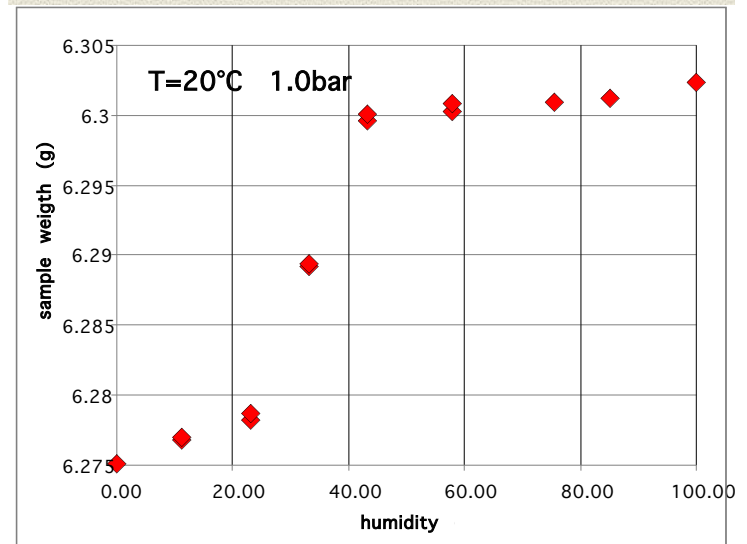


Membrane Diffraktometer V1 at HZB

Sample has to be in fixed Zero Position when the balance is rotating, e.g. in a “rocking scan” !

Sample has to come back in reproduceable beam position after lifting and weighing !

Adapter for Humidity Control by Saturated Salt Solutions





Thanks for your attention

and to my Coworkers



Nico Grimm (Engineer)

Mario Bochnia (Technician)



