

Sample Environment at the Swiss Spallation Neutron Source SINQ:

the Strength of Combination

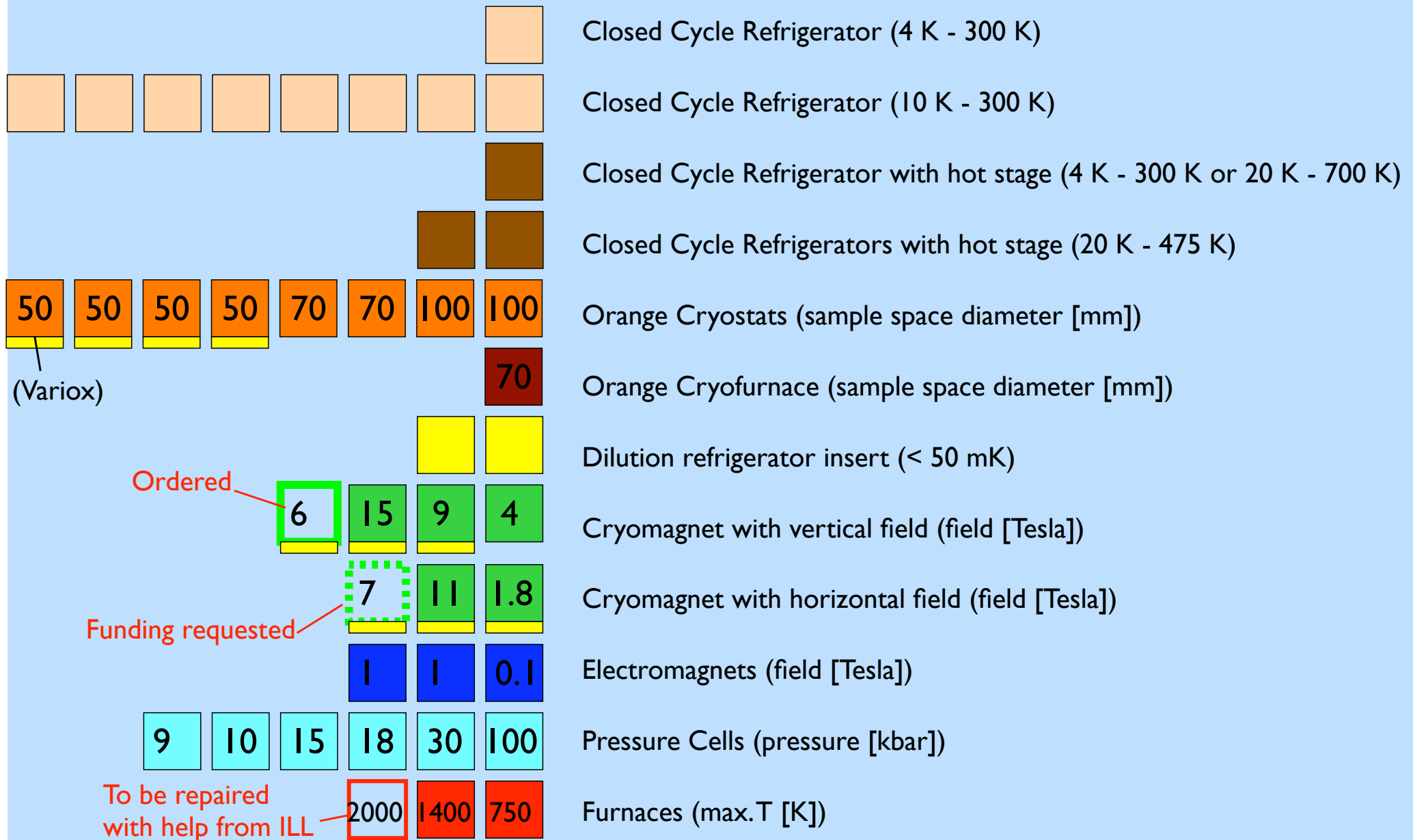
Markus Zolliker

Paul Scherrer Institute, Villigen, Switzerland

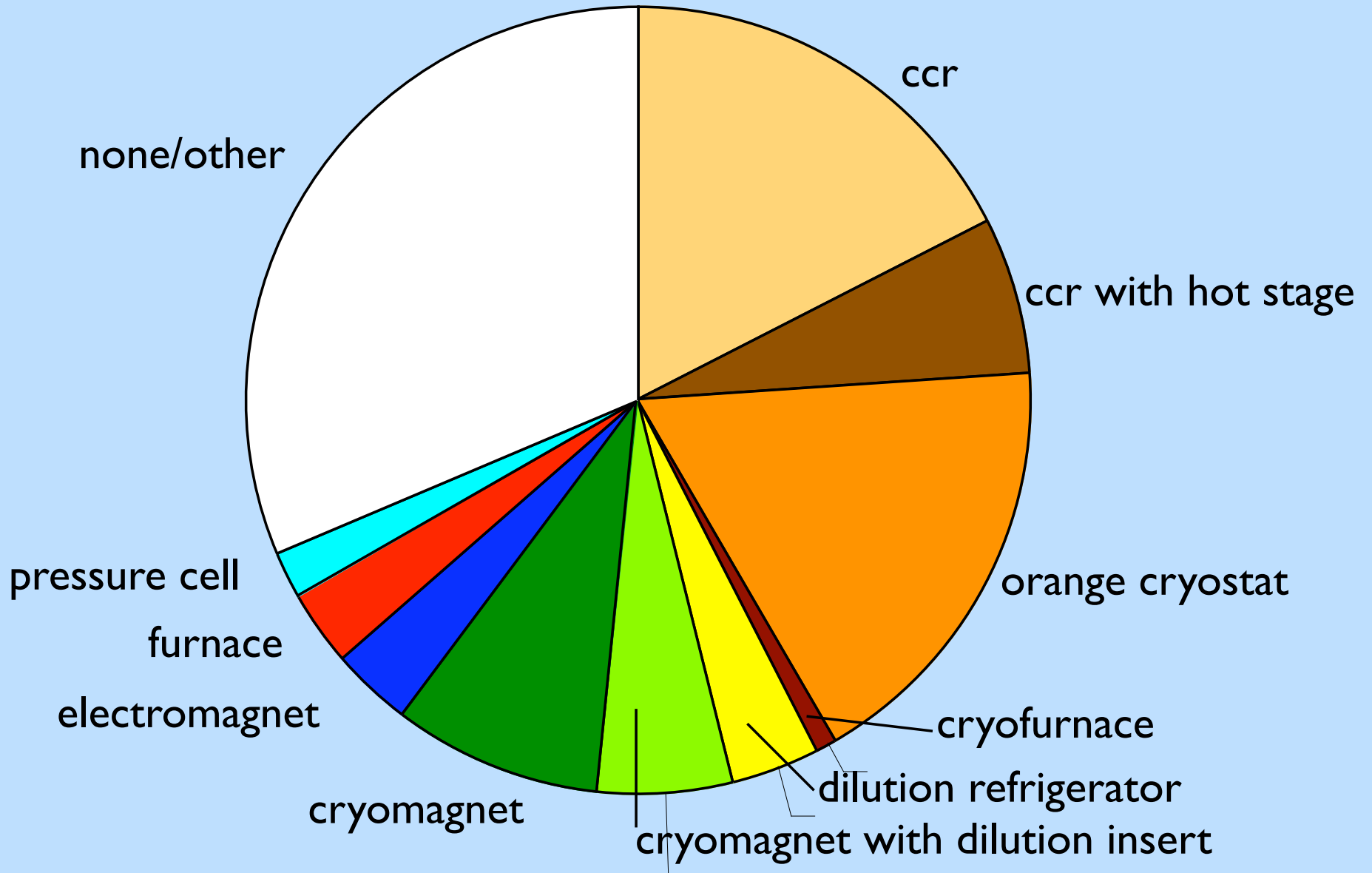
SINQ, the Swiss Spallation Neutron Source

- SINQ is a spallation neutron source at the Paul Scherrer Institute in Switzerland
- Built in 1990 as a replacement of the beam dump of an already existing proton accelerator
- SINQ has a continuous source instrumentation as on a reactor
- The flux is about 10^{14} n/cm²/s, this is not a lot, compared to larger sources (1/15 of ILL)
- But:
 - A sophisticated guide system and instrumentation increases the flux at the sample substantially
 - Reliable sample environment and especially the combination of different devices makes SINQ competitive

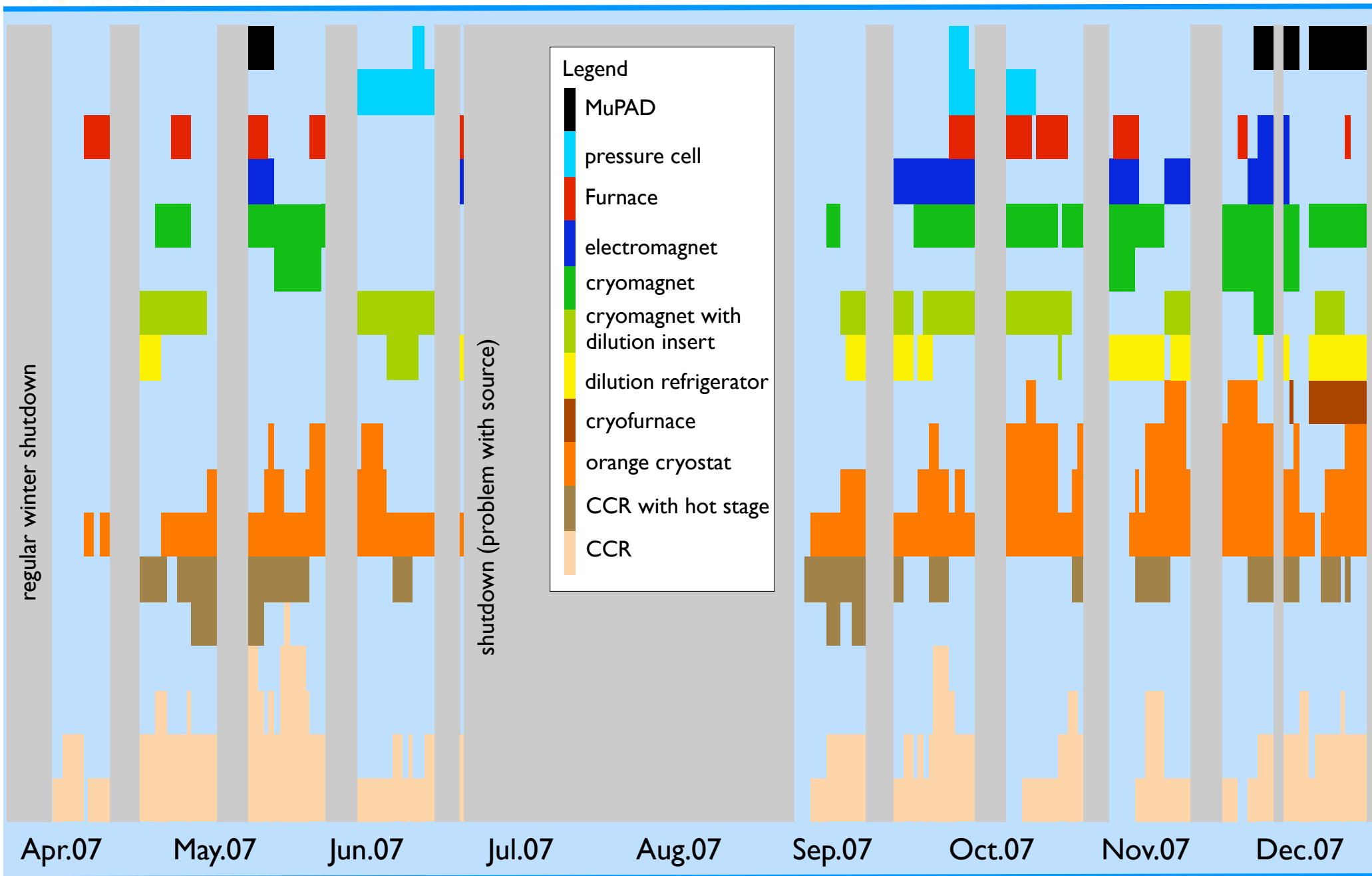
Sample Environment Equipment at SINQ



Sample Environment Usage in 2007



Sample Environment Usage in 2007



- Very low T (<50 mK) and high field
 - 14.9 Tesla vertical (as at HMI, ILL, FRM ...)
 - 11 Tesla horizontal → see talk of Michel Kenzelmann tomorrow
- **High electric field and low/high T (20 K ... 475 K)**
- **Furnace with controlled atmosphere**
- **Dilution refrigerator in MuPAD**
- **High pressure (>50 kbar) at low T (20 K)**

Hi Electric Field at Low and High T

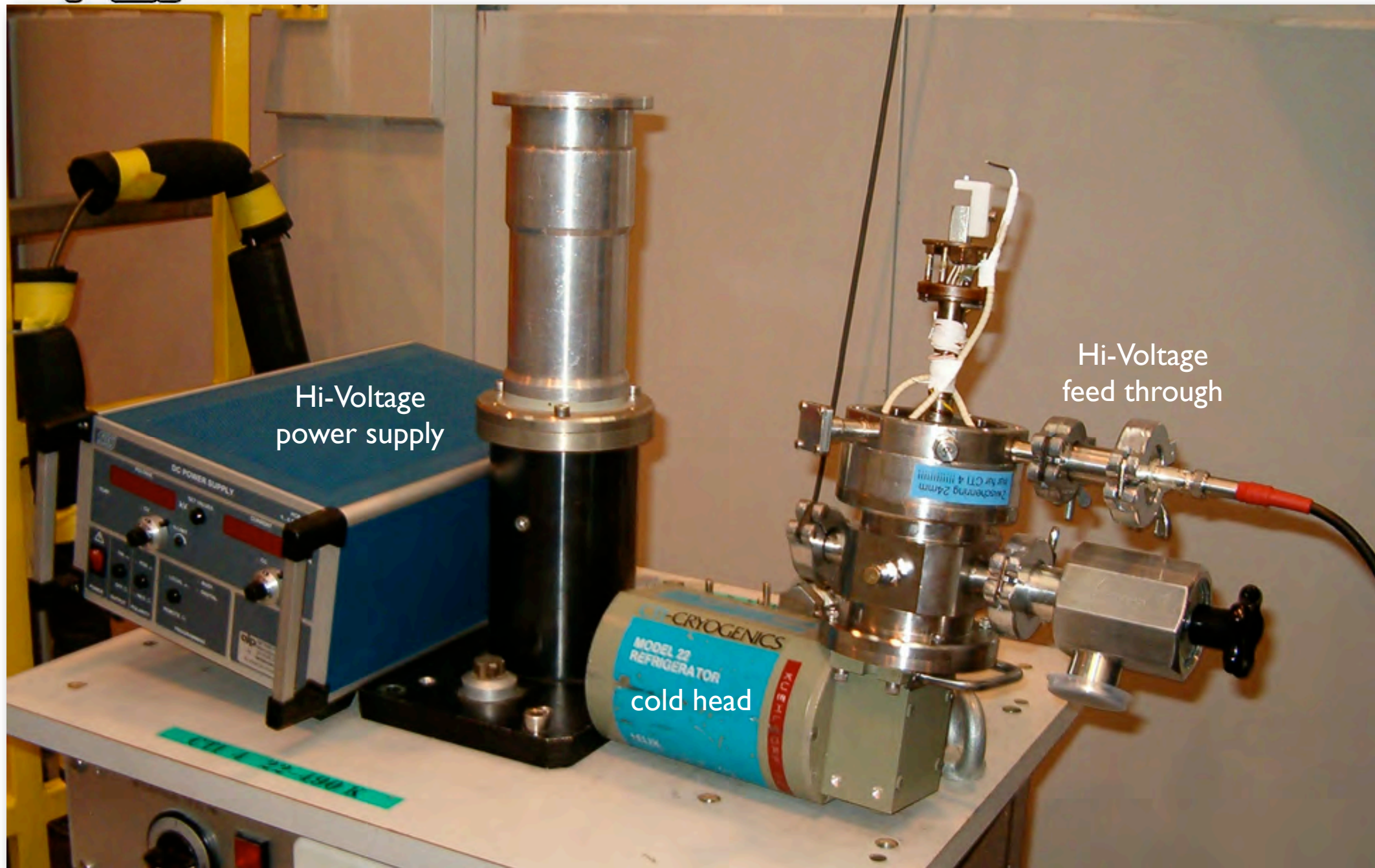
Jochen Stahn, LNS, PSI Villigen

High Electric Field at Low/High Temperature

- High voltage feed through into CCR with hot stage
- 6 kV, $< 2 \mu\text{A}$
- An other setting with much lower electrical field was even combined with a magnetic field (1 Tesla, electromagnet)



High Electric Field at Low/High Temperature



Controlled Atmosphere at High T

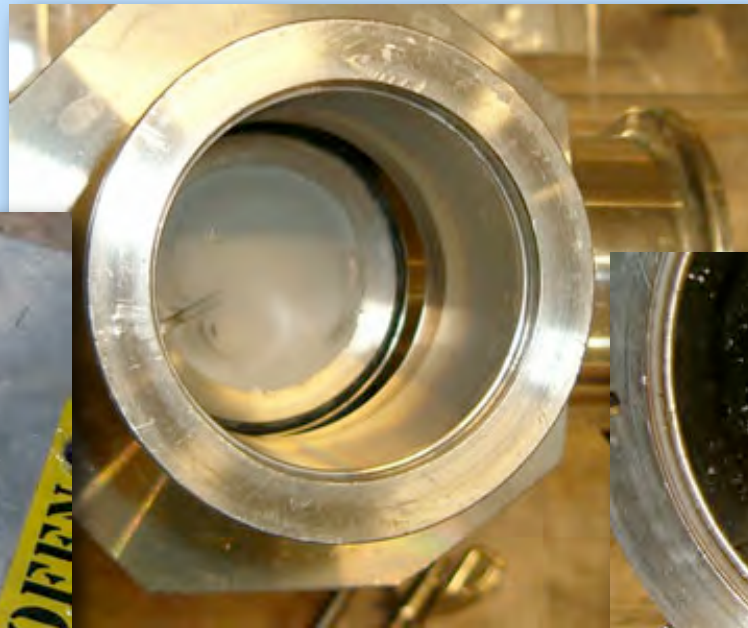
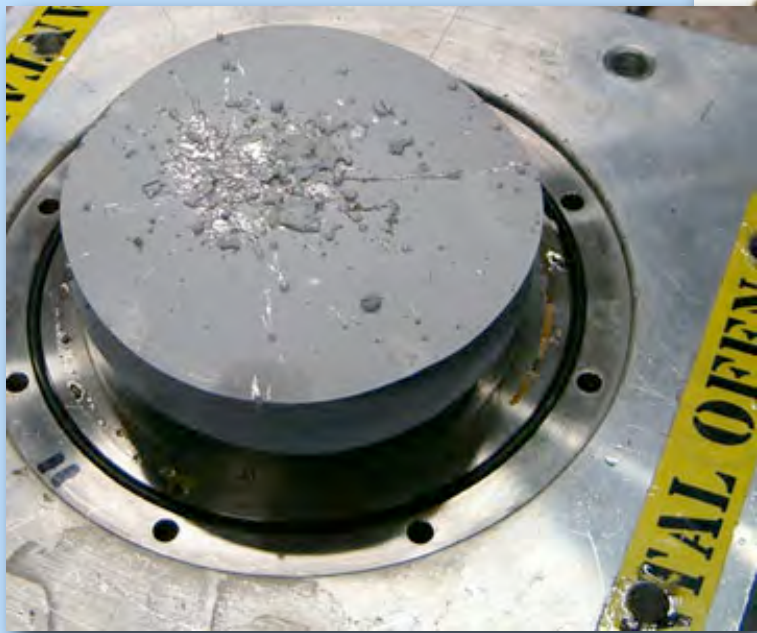
Furnace with Controlled Atmosphere

- Furnaces break very often, due to improper usage:
 - sample reacts with sample container

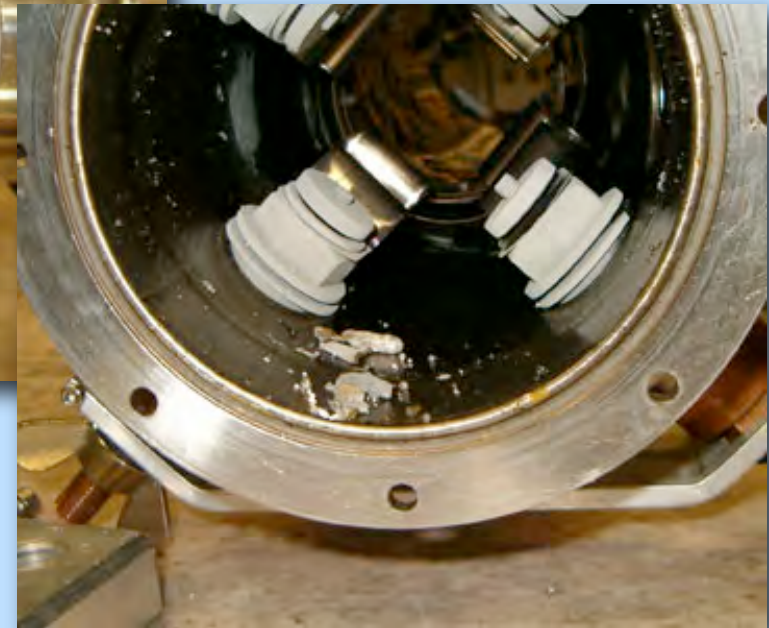


Furnace with Controlled Atmosphere

- Furnaces break very often, due to improper usage:
 - sample reacts with sample container
 - pieces on the sample container melt



Cadmium
molten and frozen



Furnace with Controlled Atmosphere

- Furnaces break very often, due to improper usage:
 - sample reacts with sample container
 - pieces on the sample container melt
 - sample in a hermetically sealed container explodes due to outgasing powder sample
 - → why not put the sample in an open container in vacuum?
 - some samples change its composition due to outgasing
 - **Solution: hermetically sealed container, but with a connection to the outside of the furnace**

Furnace with Controlled Atmosphere



Spherical Spin Polarisation Analysis at Very Low T

Bertrand Roessli, Oksana Zaharko

Laboratory for Neutron Scattering, PSI Villigen

Marc Janoschek

Laboratory for Neutron Scattering, PSI Villigen & TU München

Serguei Klimko

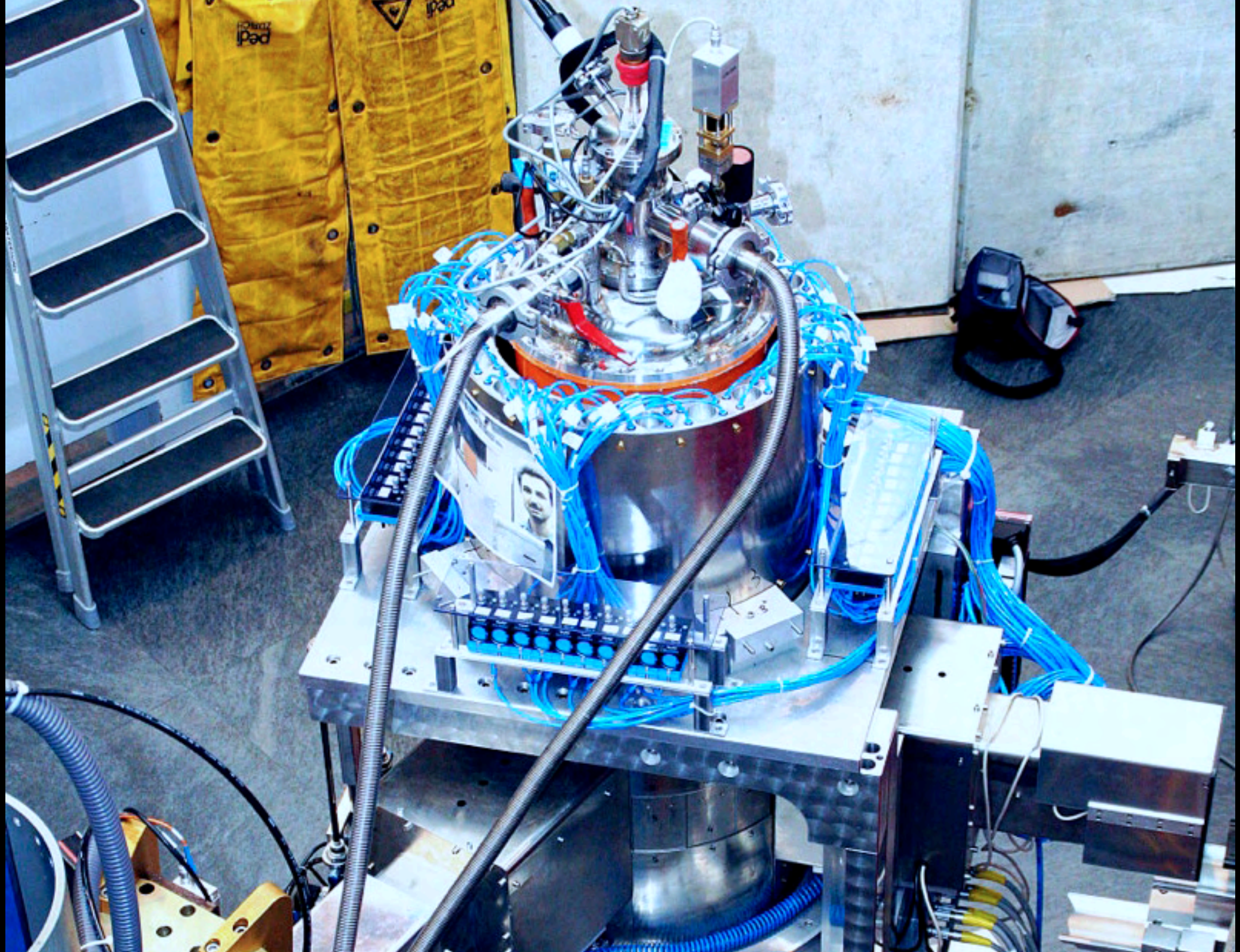
Laboratory for Neutron Scattering, PSI Villigen & TU München & ILL Grenoble

- Spherical neutron spin polarimetry exists since 1989 at ILL (CRYOPAD)

F. Tasset, *Physica B*, **156-157**:627, 1989.

- Another device (MuPAD) was developed by TU München & PSI and is installed at TASP (SINQ)

M. Janoschek & al., *Physica B*, **397**:125, 2007.



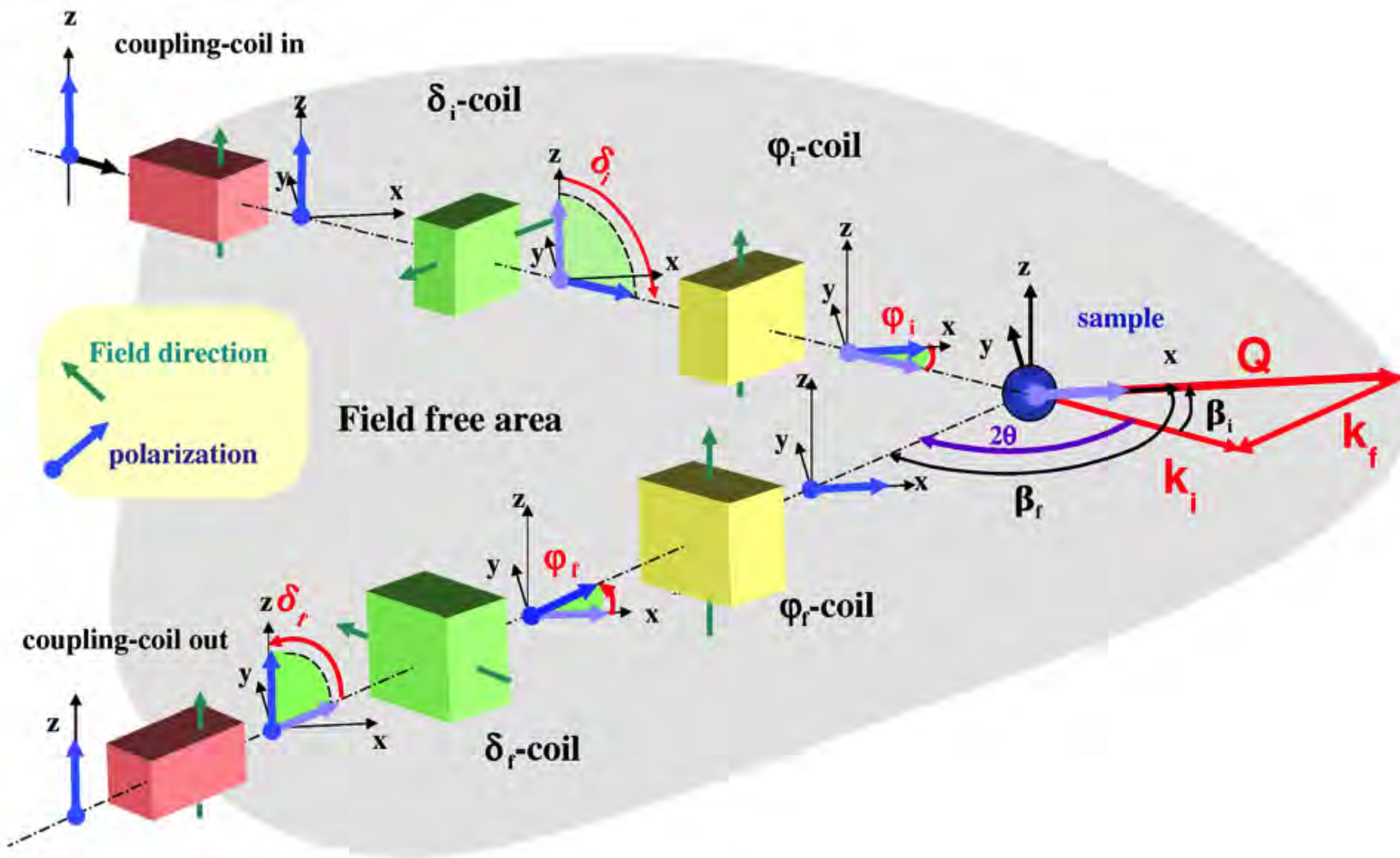
Spherical Spin Polarimetry

- polarisation e.g. $P_{xx} = (I_{xx} - I_{x-x}) / (I_{xx} + I_{x-x})$
- longitudinal polarimetry
 - guide field along the whole beam path
 - → we can only measure P_{xx}, P_{yy}, P_{zz}
- spherical spin polarimetry:
 - we can also measure the off diagonal elements

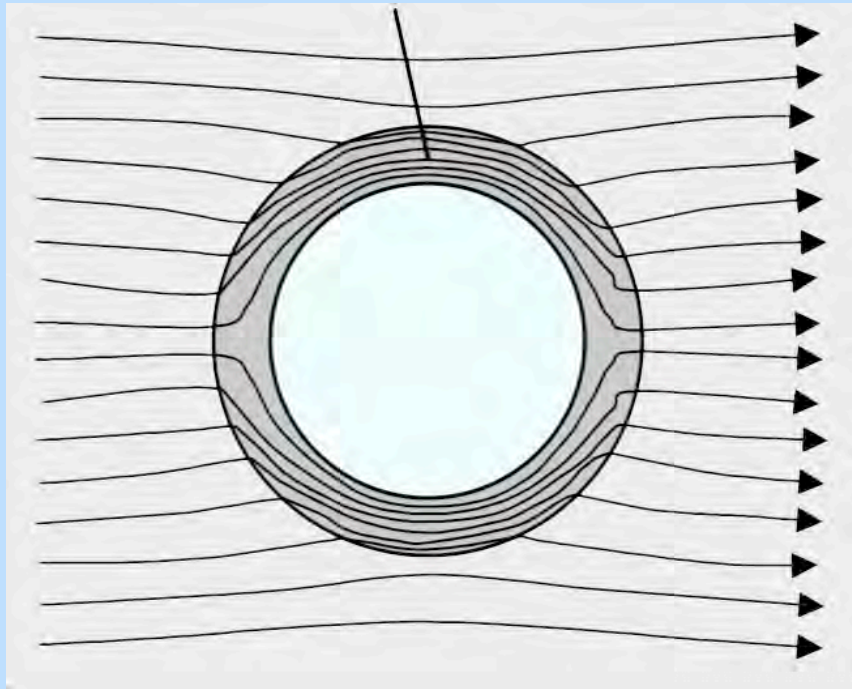
$$P_{xx}, P_{xy}, P_{xz}$$

$$P_{yx}, P_{yy}, P_{yz},$$

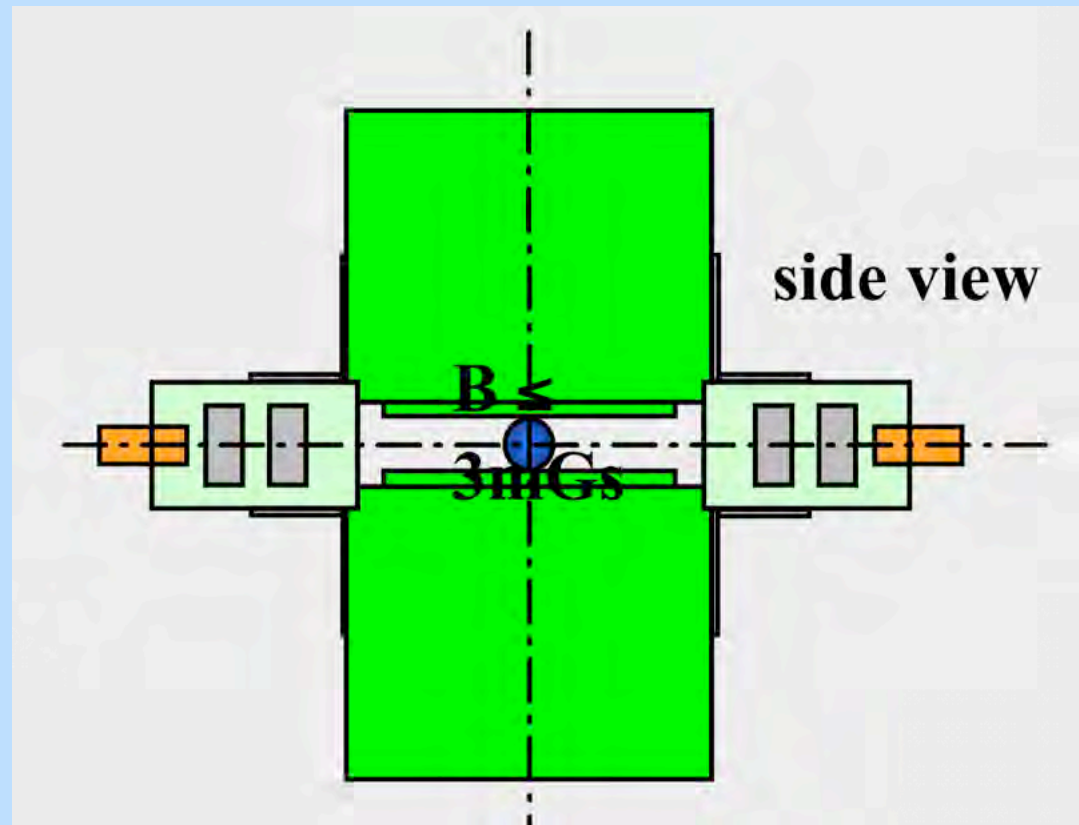
$$P_{zx}, P_{zy}, P_{zz}$$
 - only with a field free area around the sample

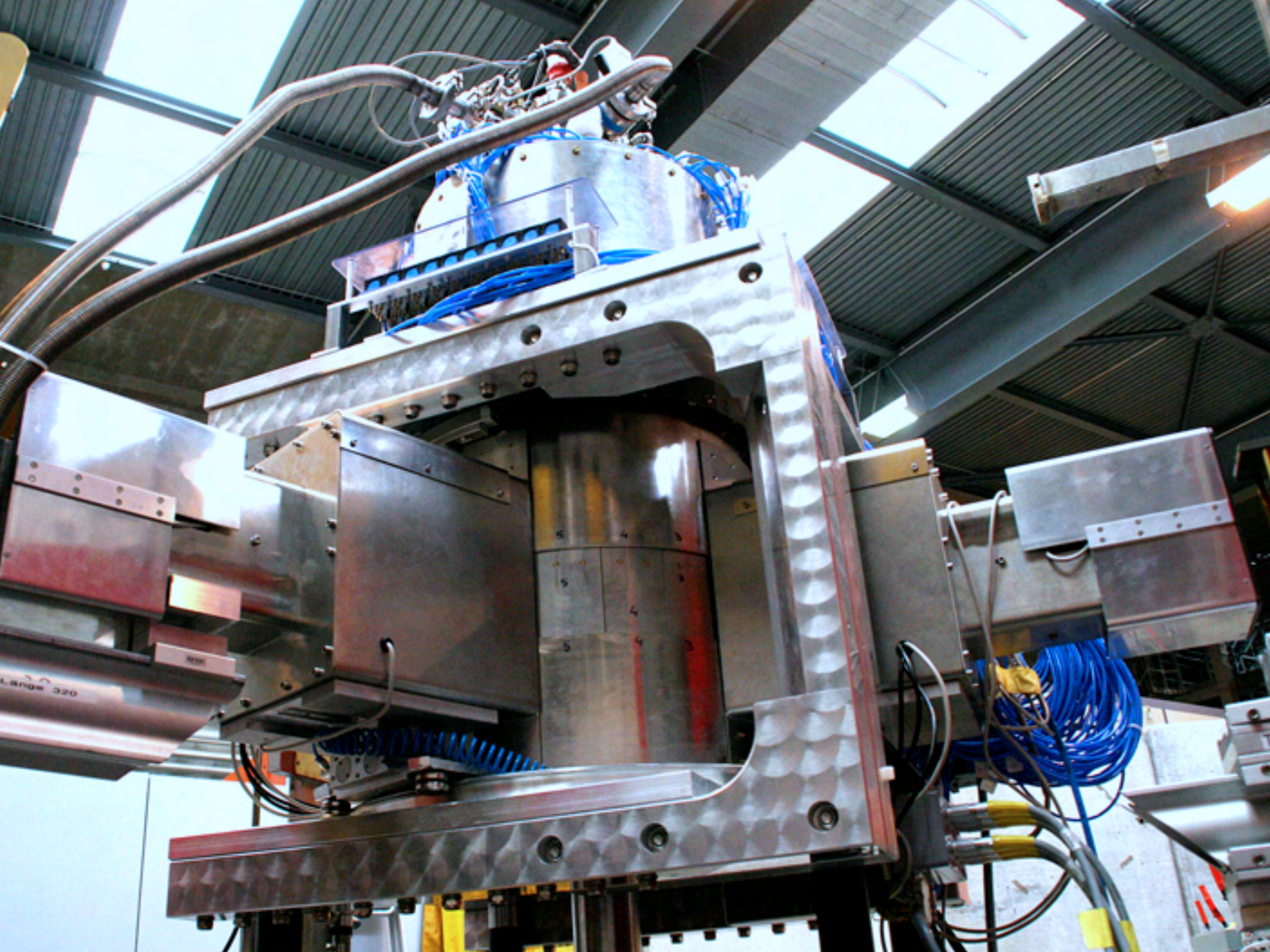


mu-metal pushes the field out

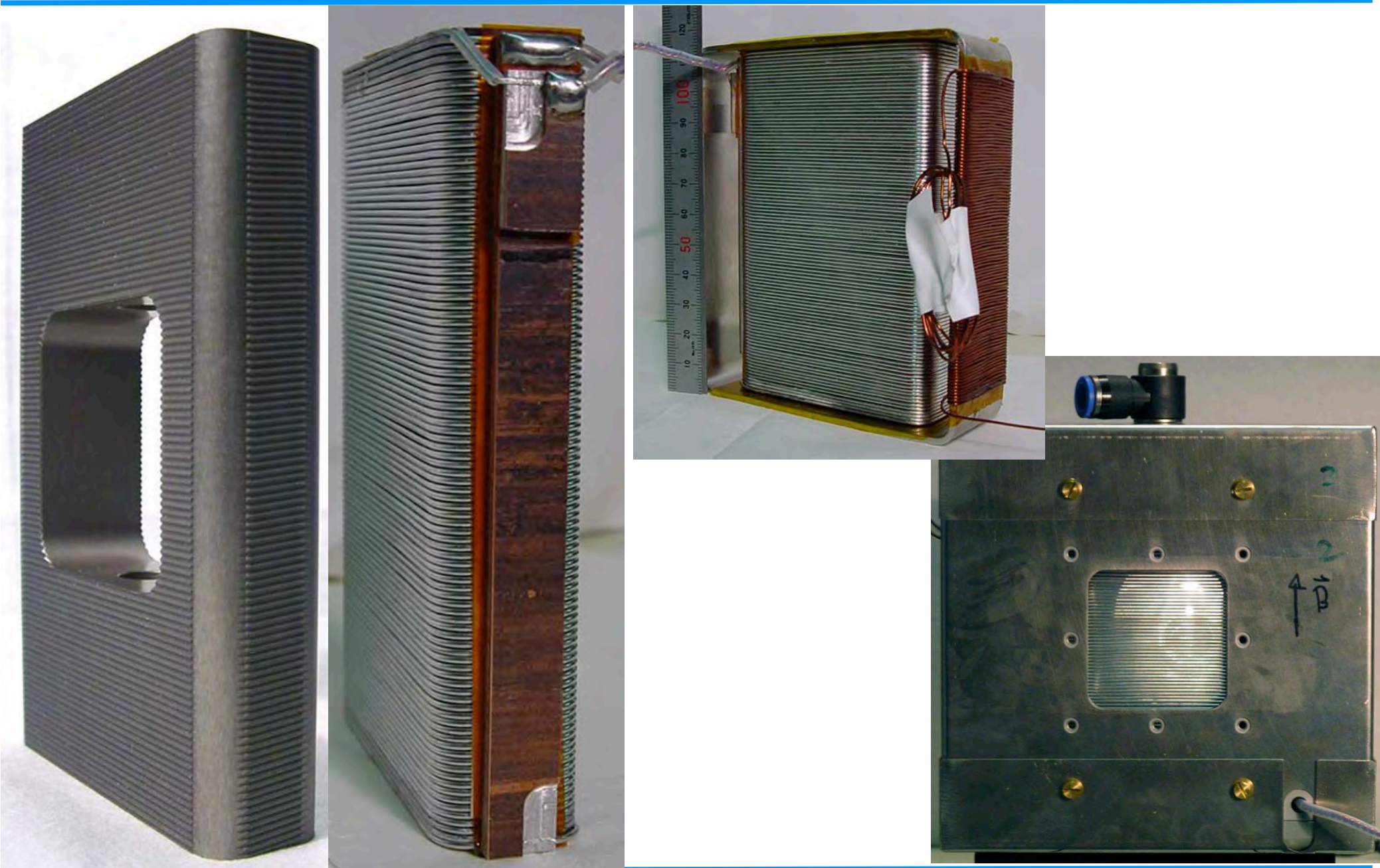


double wall mu-metal shield:
field 60 times smaller than
earth magnetic field



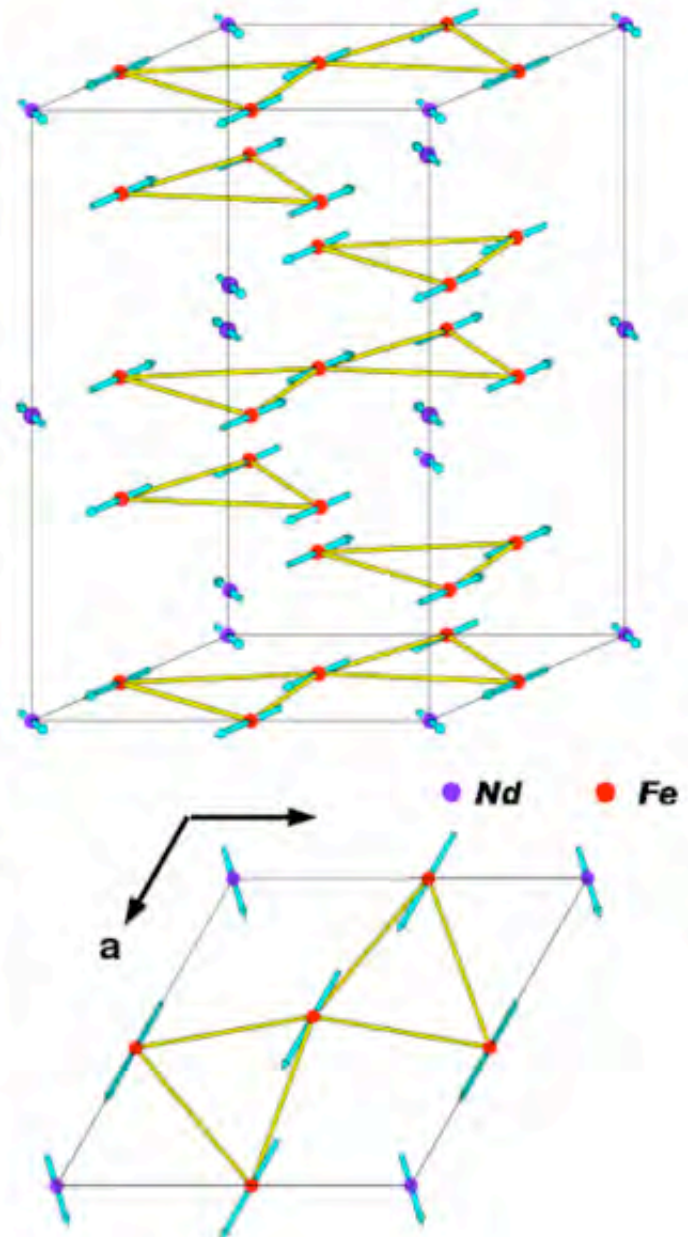


MuPAD precession coil



Example: $\text{NdFe}_3(\text{BO}_3)_4$ (May 2007)

H	K	L	P_i	$P_{\text{meas.}}$			$P_{\text{calc.}}$		
0.000	2.000	-0.500	+1	-0.88	+0.03	+0.03	-0.85	+0.00	-0.00
			+1	-0.03	-0.66	-0.06	+0.00	-0.66	+0.00
			+1	+0.08	+0.04	+0.66	+0.00	+0.00	+0.66
			-1	+0.86	-0.07	-0.04	+0.85	-0.00	+0.00
			-1	+0.06	+0.66	+0.05	+0.00	+0.66	-0.00
			-1	-0.04	+0.05	-0.65	+0.00	-0.00	-0.66
0.000	0.000	-1.500	+1	-0.88	+0.05	+0.12	-0.85	+0.00	+0.00
			+1	+0.04	+0.01	+0.01	+0.00	-0.03	+0.03
			+1	+0.02	+0.03	-0.02	-0.00	+0.03	+0.03
			-1	+0.87	-0.06	-0.11	+0.85	+0.00	+0.00
			-1	+0.01	-0.01	-0.02	+0.00	+0.03	-0.03
			-1	+0.03	-0.02	-0.00	-0.00	-0.03	-0.03
0.000	1.000	0.500	+1	-0.88	+0.00	+0.04	-0.85	-0.00	+0.00
			+1	-0.01	-0.49	-0.08	-0.00	-0.40	-0.04
			+1	+0.10	-0.07	+0.48	-0.00	-0.04	+0.40
			-1	+0.86	-0.04	-0.08	+0.85	+0.00	-0.00
			-1	+0.08	+0.49	+0.08	-0.00	+0.40	+0.04
			-1	-0.07	+0.06	-0.48	-0.00	+0.04	-0.40
0.000	4.000	0.500	+1	-0.86	-0.02	-0.03	-0.85	-0.00	-0.00
			+1	+0.01	-0.82	-0.08	-0.00	-0.79	+0.00
			+1	+0.08	-0.08	+0.86	-0.00	+0.00	+0.79
			-1	+0.86	-0.06	-0.01	+0.85	+0.00	+0.00
			-1	+0.05	+0.85	+0.05	-0.00	+0.79	-0.00
			-1	-0.03	-0.02	-0.83	-0.00	-0.00	-0.79
0.000	1.000	-2.500	+1	-0.86	+0.07	+0.10	-0.85	+0.00	+0.00
			+1	+0.02	-0.10	-0.02	+0.00	-0.06	+0.03
			+1	+0.03	-0.02	+0.05	+0.00	+0.03	+0.06
			-1	+0.87	-0.07	-0.10	+0.85	+0.00	+0.00
			-1	+0.04	+0.08	-0.01	+0.00	+0.06	-0.03
			-1	+0.04	+0.01	-0.11	+0.00	-0.03	-0.06



Spherical Spin Polarimetry below 100 mK

- Worked well at 20 K, why not at 100 mK?
- Inner diameter of MuPAD designed for a standard orange cryostat
- Oxford Instruments dilution insert runs best within VariOX
 - BUT: The VariOX cryostat has a slightly larger diameter (10 mm more) → **does not fit**
- Test dilution refrigerator in orange cryostat: works, with a higher LHe consumption
 - → First experiment done in December 2007 (O. Zaharko & al.)

High pressure at low T

Thierry Strässle, Balasubramanian Padmanabhan

Laboratory for Neutron Scattering, PSI Villigen

S. Klotz, G. Hamel

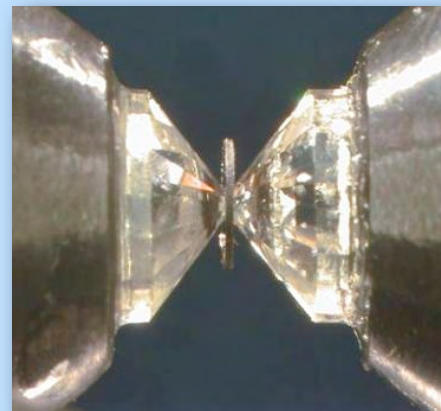
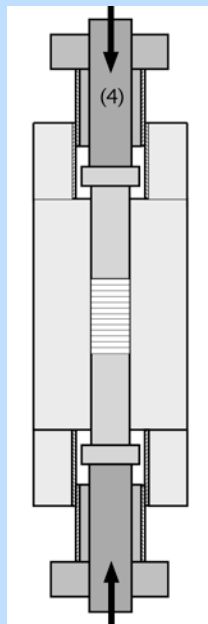
Université Pierre et Marie Curie, Paris

High pressure (>50 kbar) at low T (4 K)

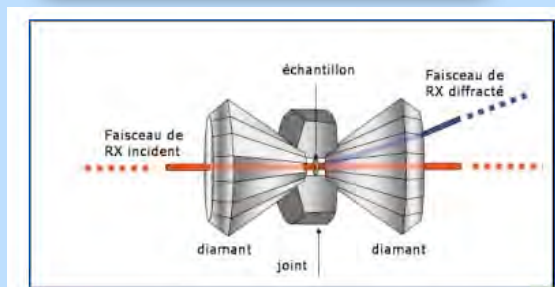
cylindrical geometry

VS

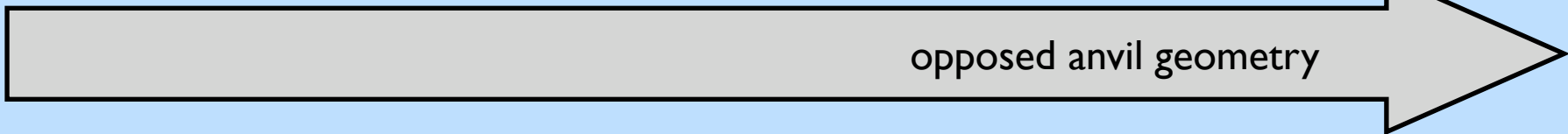
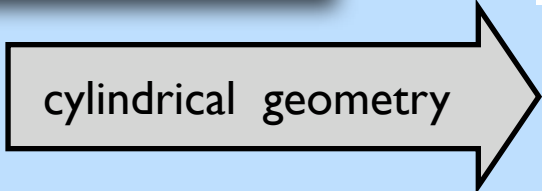
opposed-anvils geometry



Ph.Gillet



available since many years at SINQ
 $1.5 \text{ K} < T < 300 \text{ K}$



few μm^3

few cm^3

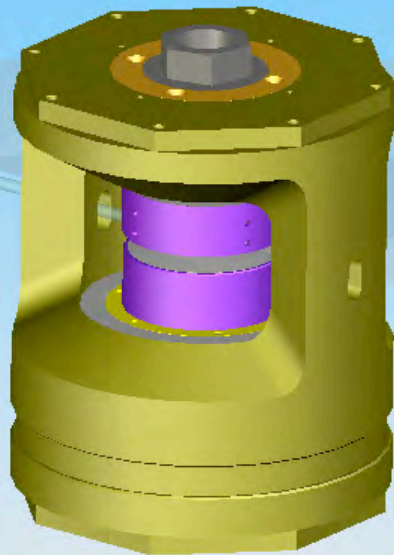
1 GPa/10 kbar

10 GPa/100 kbar

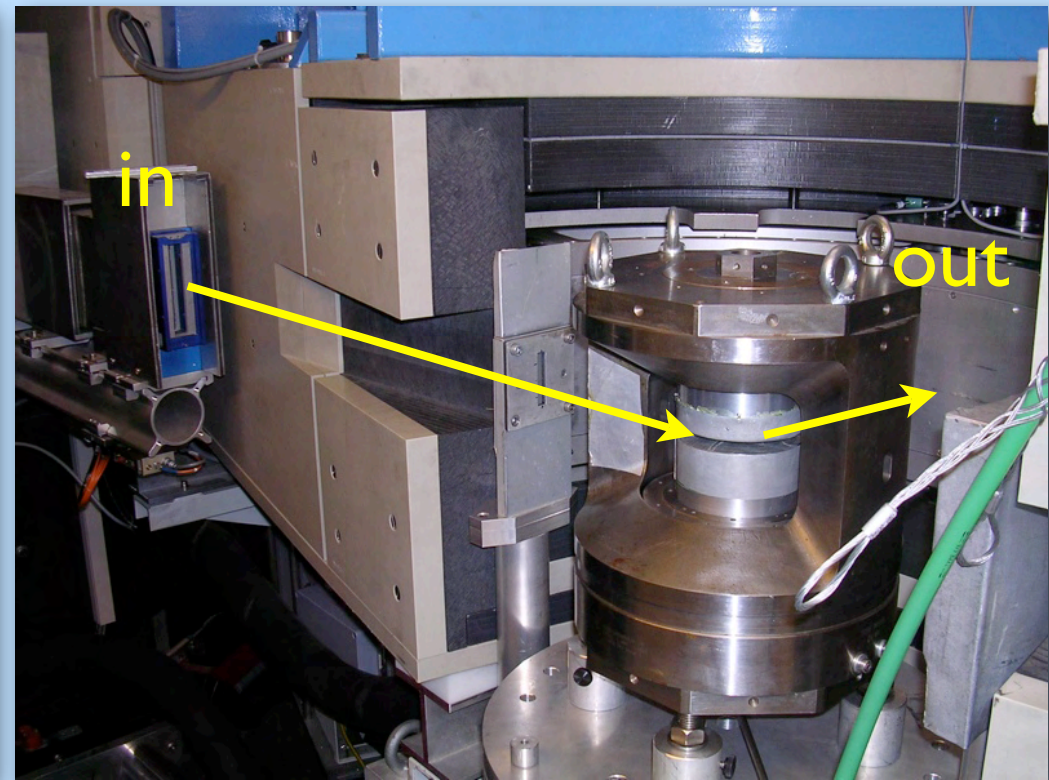
few 100 GPa/Mbar

High pressure (>50 kbar) at low T (4 K)

- “Junior version” of conventional Paris-Edinburgh cell for cooling with a CCR, ~ 100 kbar
- several successful experiments at RT in 2007



from G.Hamel, IMPMC, Paris



HRPT/SINQ, PSI

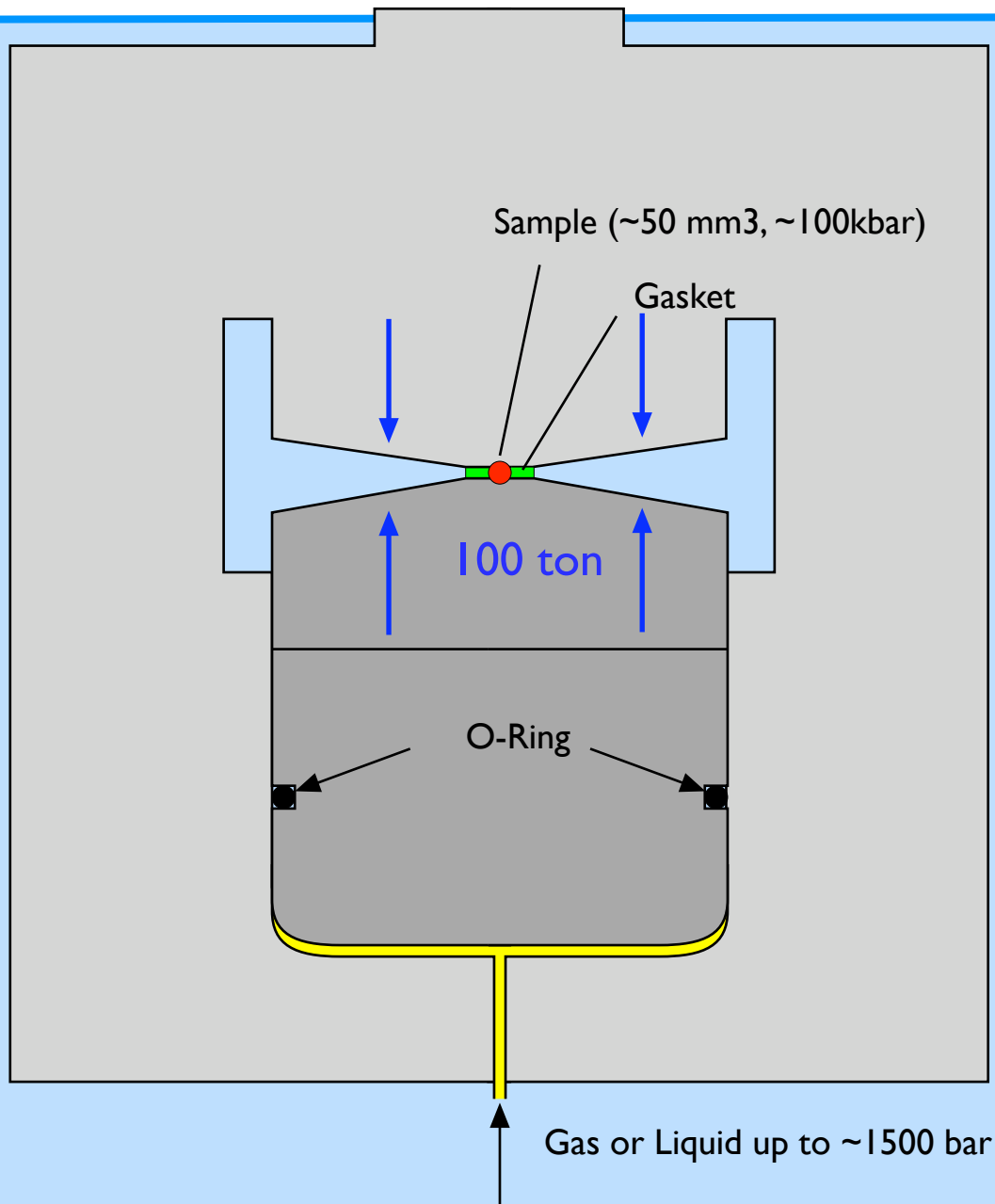
High pressure (>50 kbar) at low T (4 K)

- ILL developed a special cryostat for this cell with 4 K CCR (Sumitomo)
- PSI bought one, our technician participated the assembling at ILL.



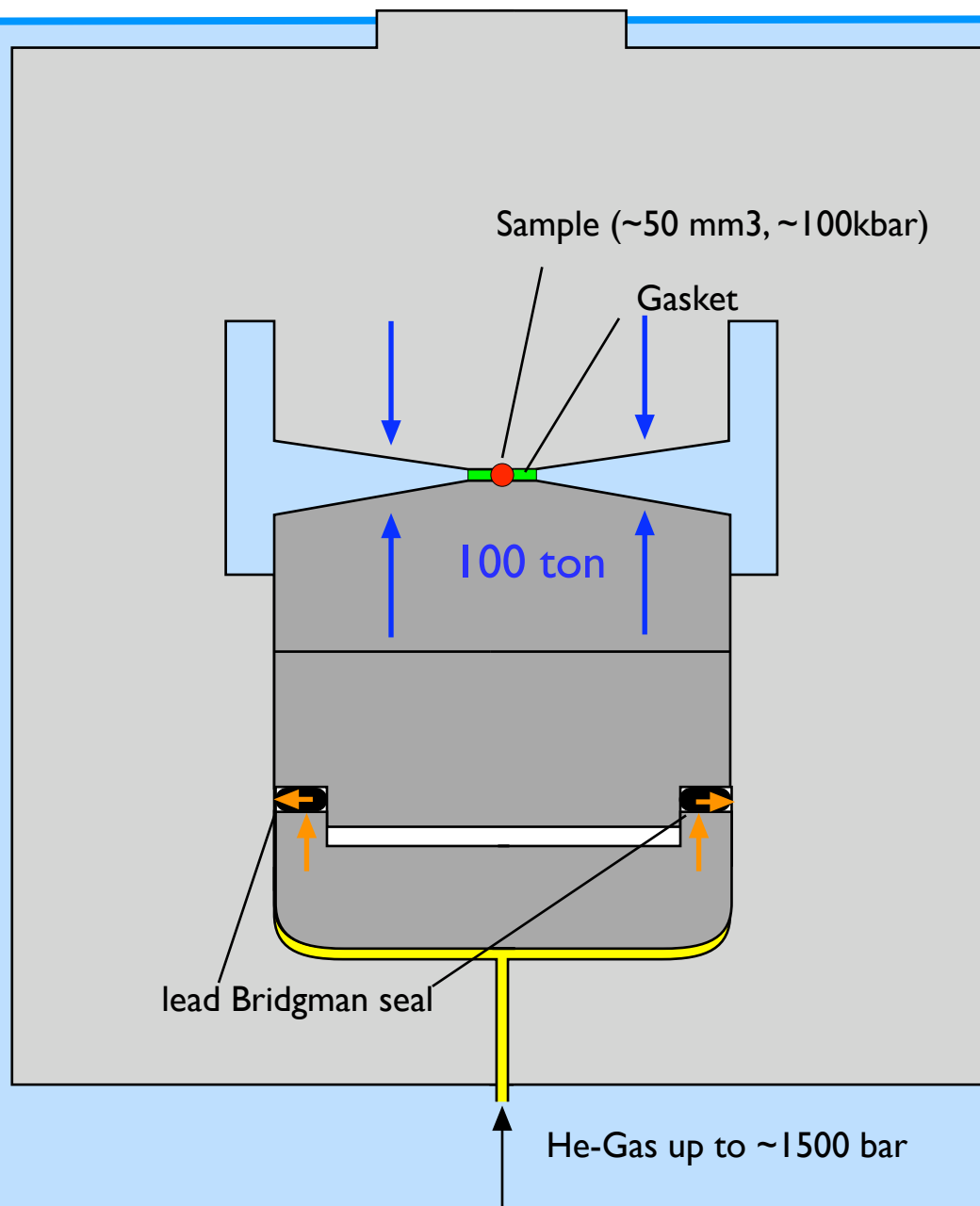
Paris-Edinburgh Anvil Pressure Cell

at RT

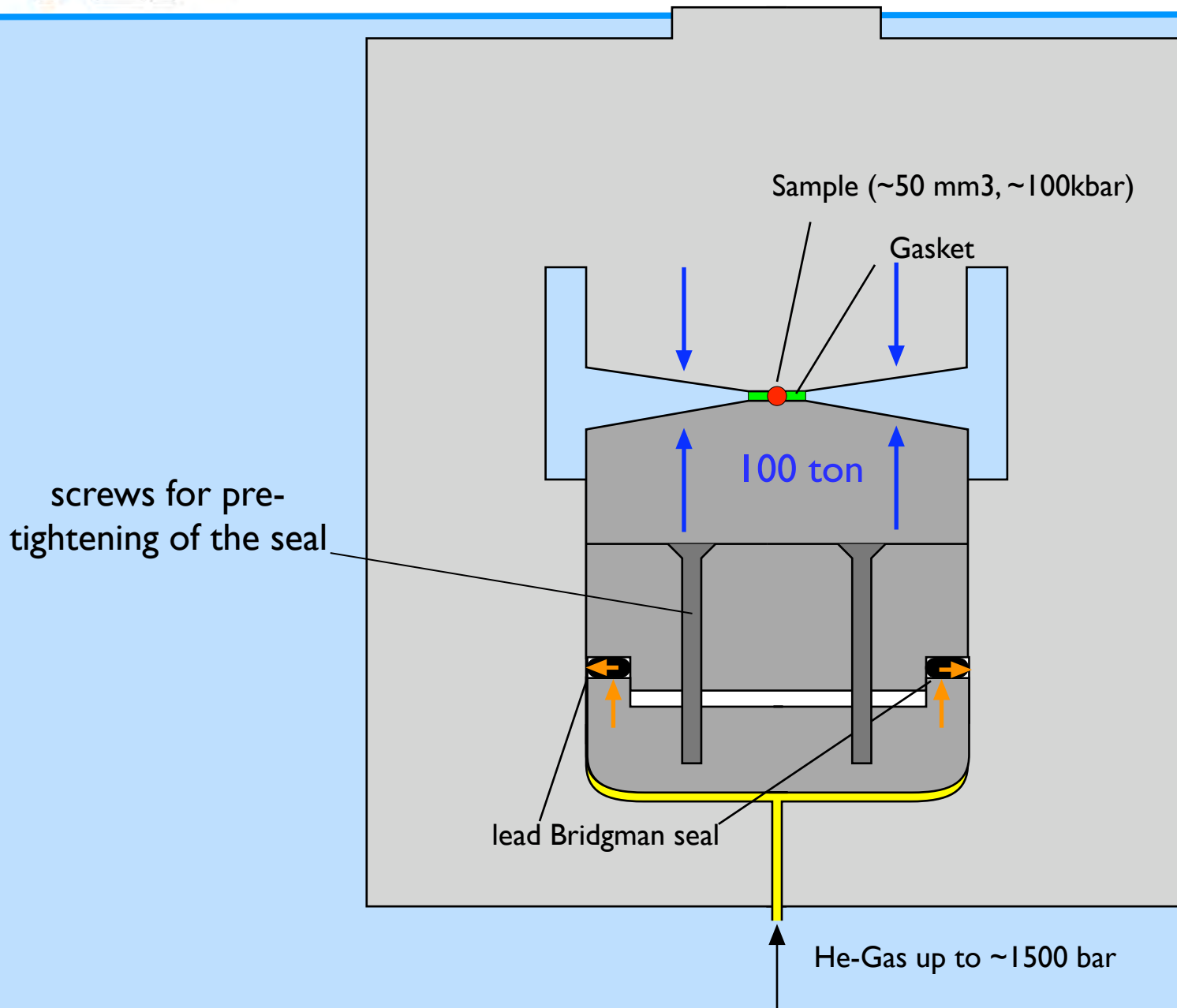


Paris-Edinburgh Anvil Pressure Cell

at low T
(20 K)

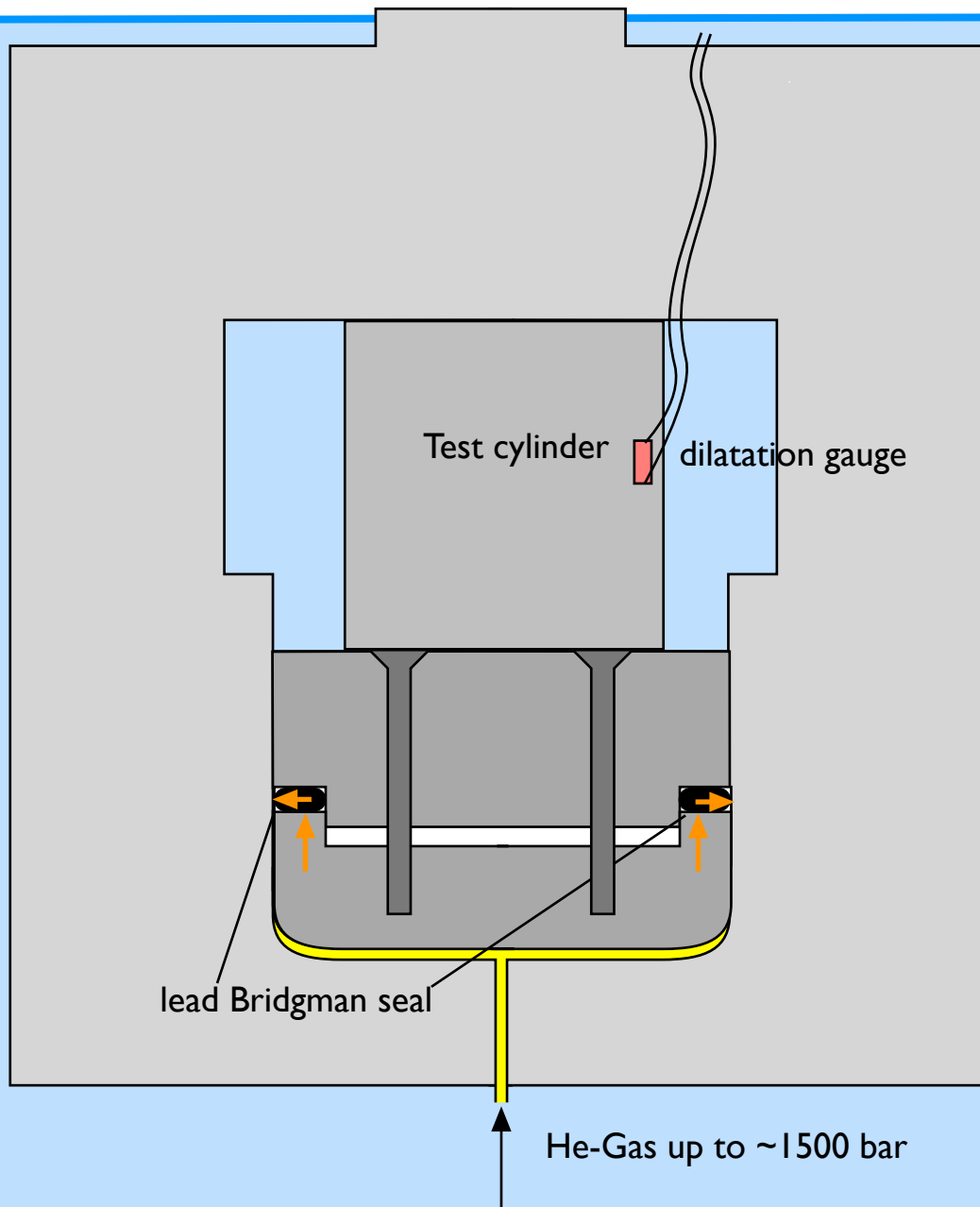


Paris-Edinburgh Anvil Pressure Cell



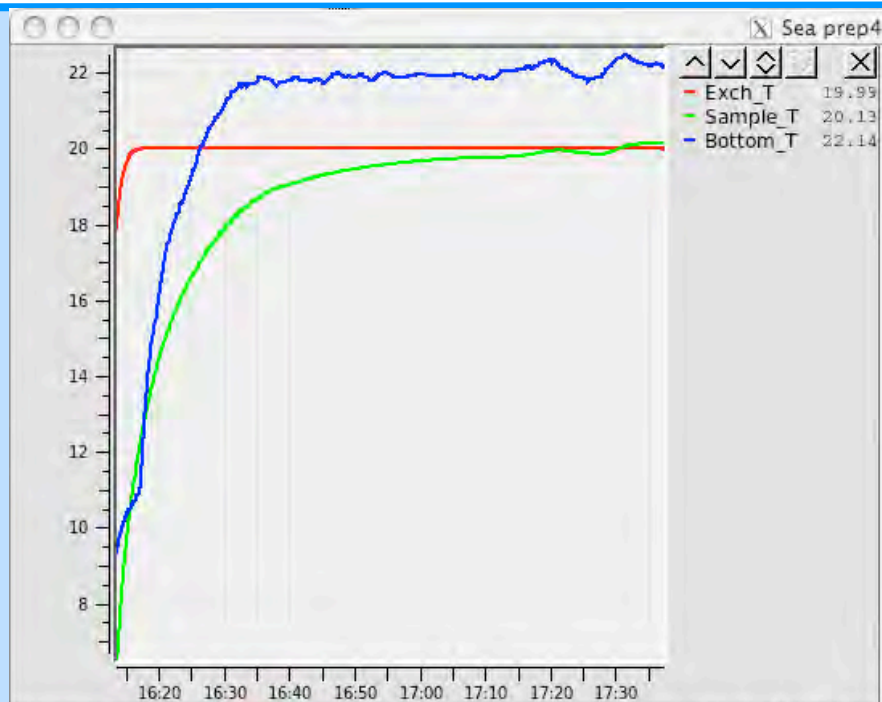
Paris-Edinburgh Anvil Pressure Cell

Testing
the
Bridgman
seal

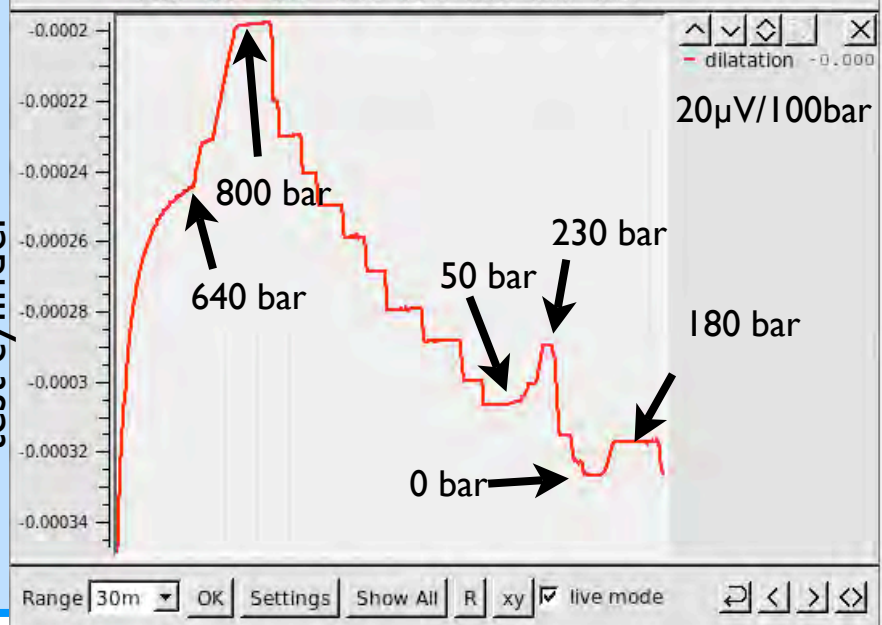


PE low-T test with Pb Bridgman piston

temperature [K] bottom cell



dilatation measured on test cylinder



cooled from RT at 600 bar
(resulted in 520 bar at 4K)

then at 20K

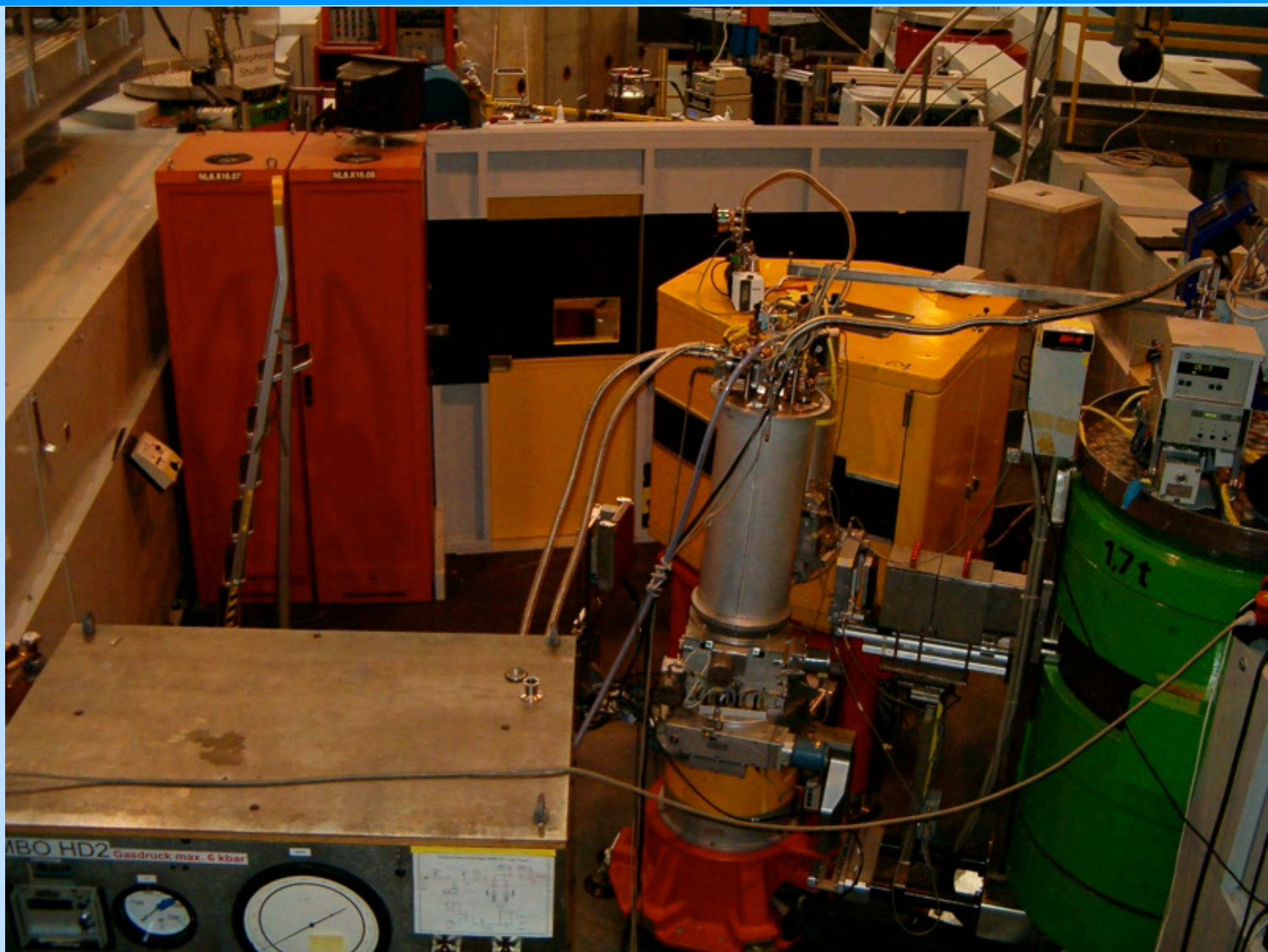
- increase to 800 bar
- decrease in steps of 50 bar
- increase from 50 bar to 230 bar
- complete release to 0 bar
- increase to 180 possible again

dilatation of test cylinder
could be measured for all changes
of the gas pressure
→ piston completely movable at 20K

BP,TS 7/02/08

High pressure at low T (4 K)

first
experiment
last week
at SINQ,
RITA-2:
30 kbar



**An innovative and
reliable sample
environment helps to
make a neutron
scattering facilities
competitive**