# HRPT

# High Resolution Powder Diffractometer for Thermal Neutrons

http://sinq.web.psi.ch/hrpt


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*Laboratory for Neutron Scattering, ETHZ and PSI*



AIC Information Day on "Large Facilities for Crystallography Studies: Synchrotron and Neutron sources"
October 19th, 2009 , Paul Scherrer Institut, Villigen, Switzerland

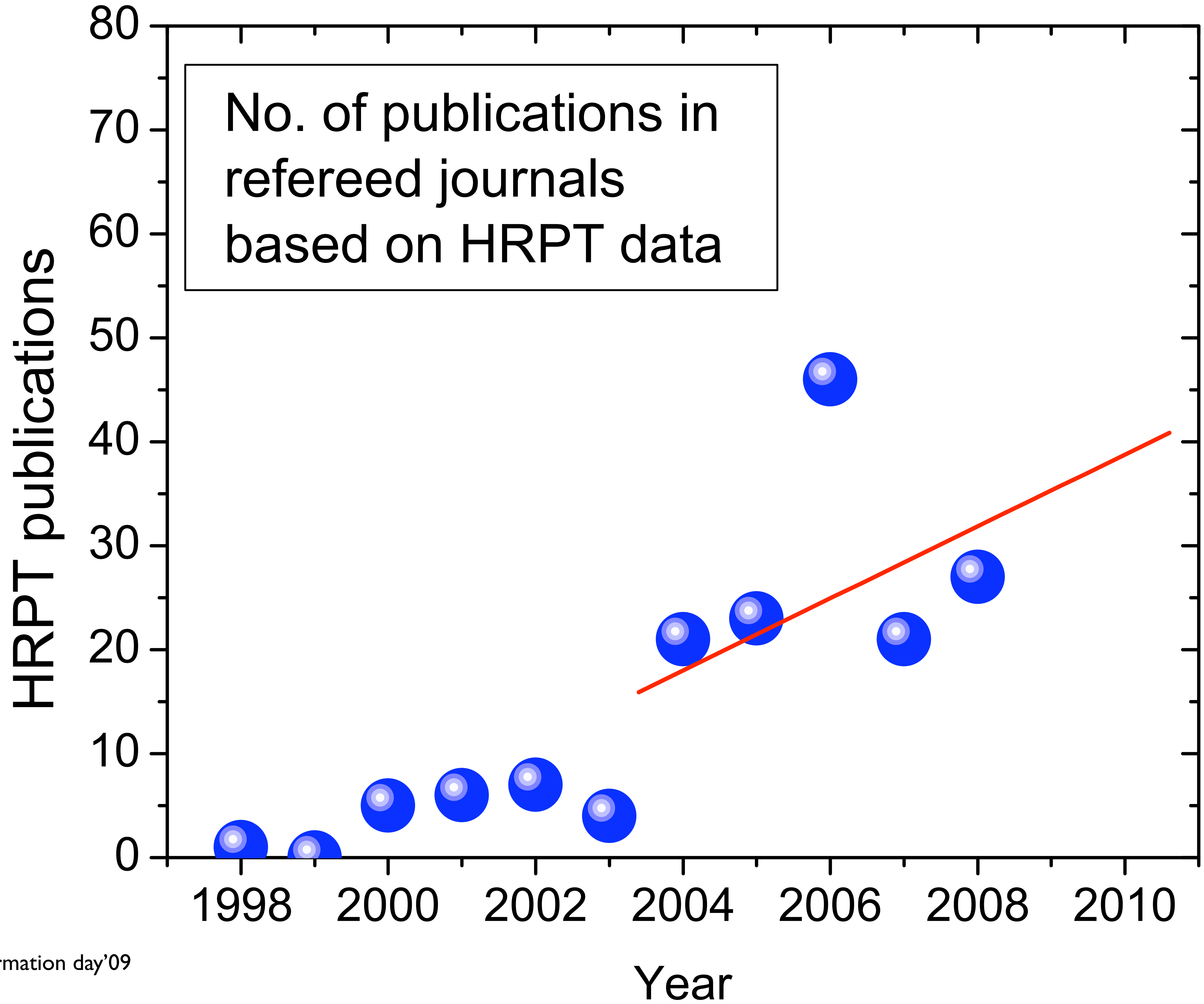
# Instead of introduction (1): HRPT history

- Design, construction (CERCA) started ~1991
- Commissioned in 1999. HRPT father is Peter Fisher

# Instead of introduction (2): Applications of HRPT diffractometer

1) Precise structure refinement complementary to x-rays
2) Magnetic ordering phenomena
3) Direct structure solution. Phase analysis of (new) materials

# Instead of introduction (2): Applications of HRPT diffractometer



# More information about HRPT

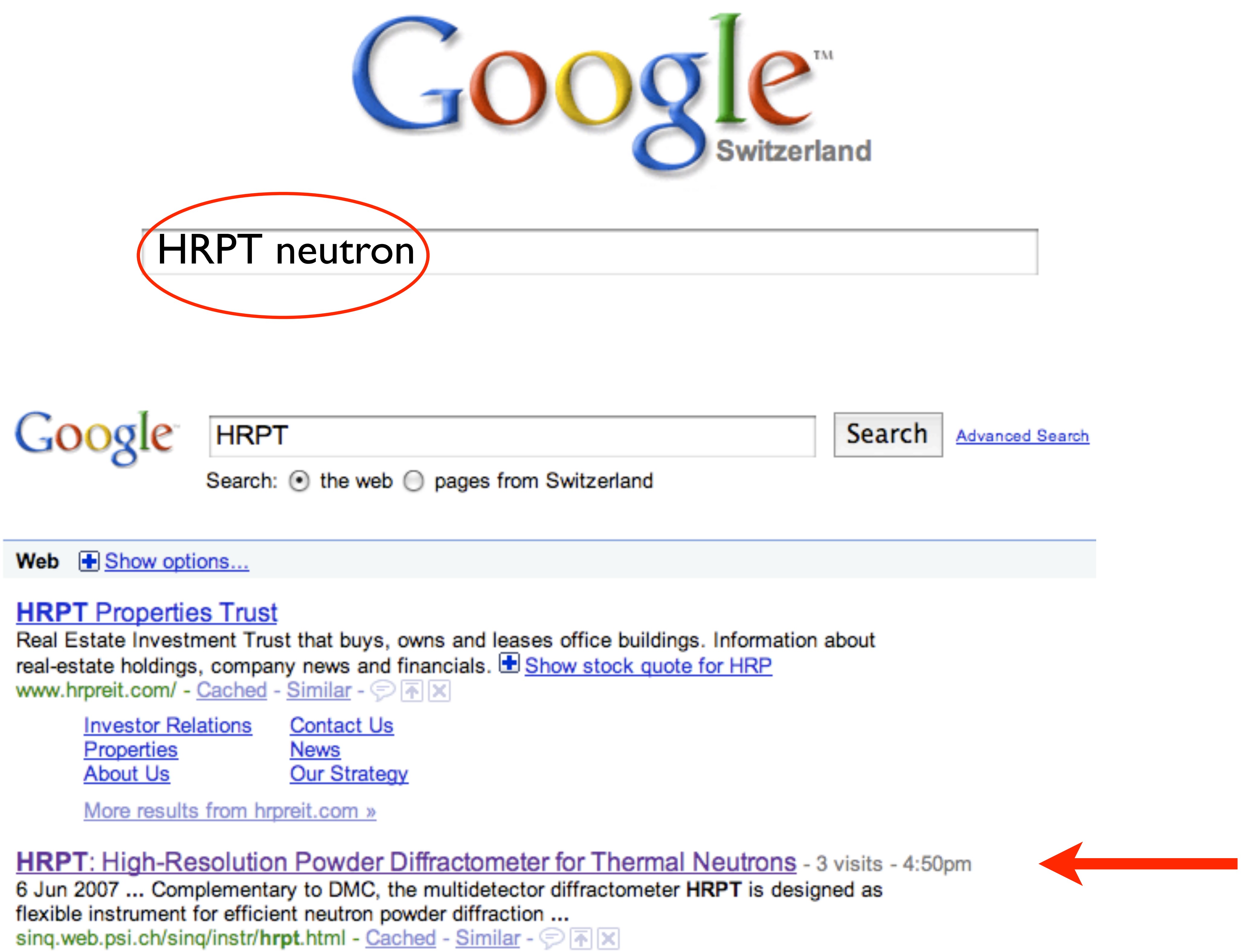
Google
Switzerland

# More information about HRPT

Google Switzerland

HRPT neutron

# More information about HRPT



# Powder neutron diffractometers

*European Portal for Neutron Scattering*

*http://pathfinder.neutron-eu.net*

# Powder neutron diffractometers

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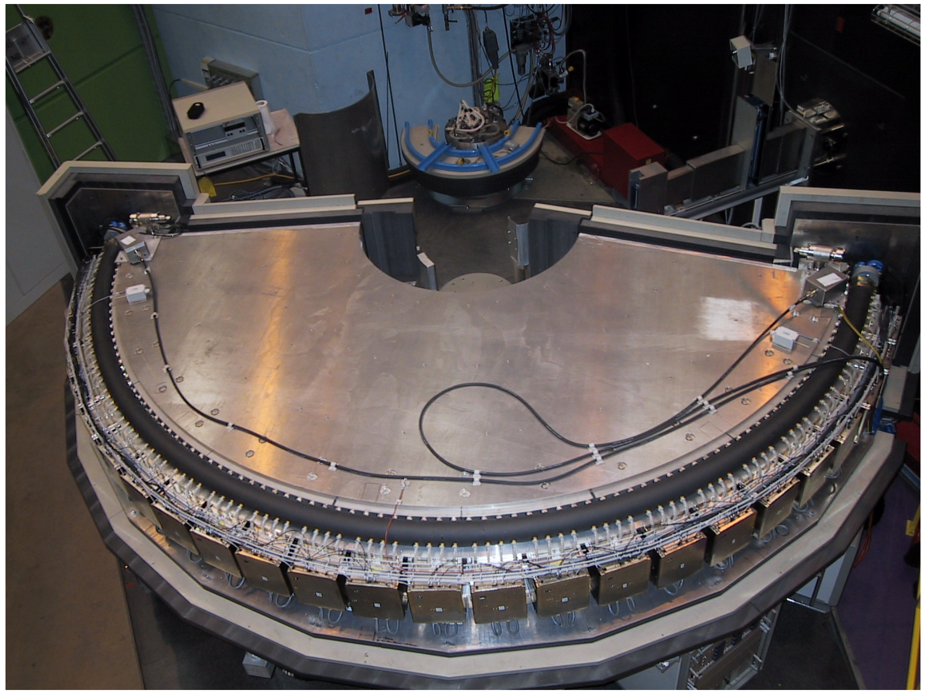
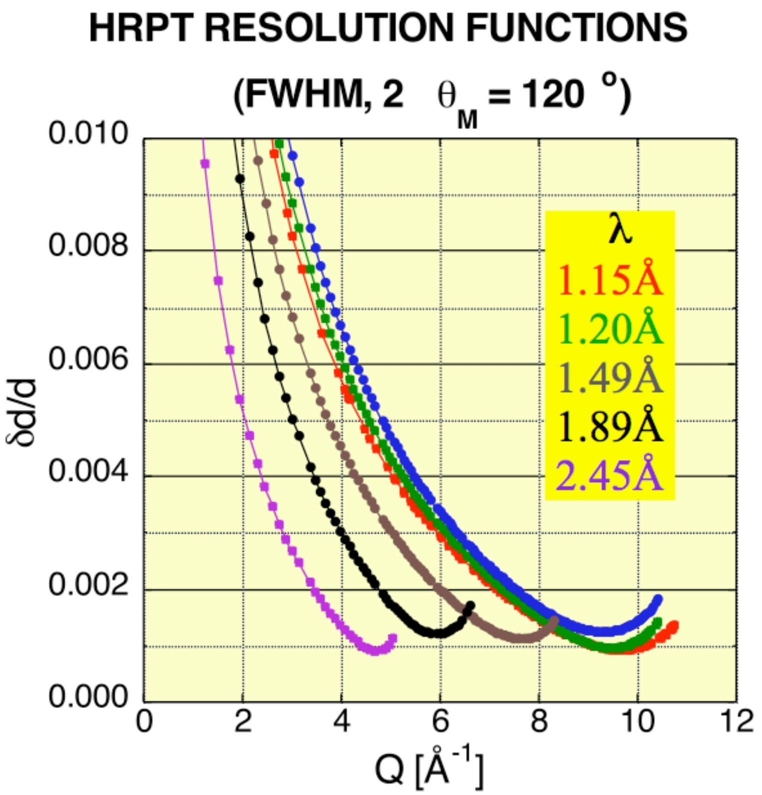
| | |
|---|---|
| SINQ/PSI, CH | DMC, HRPT, POLDI |
| LLB, FR | G41, G42 |
| ISIS, UK | GEM, HRPD, PEARL |
| FRM-II, DE | SPODI |
| FLNP/Dubna, RU | HRFD, DN2, DN12 |
| ILL, FR | D20, D2B |

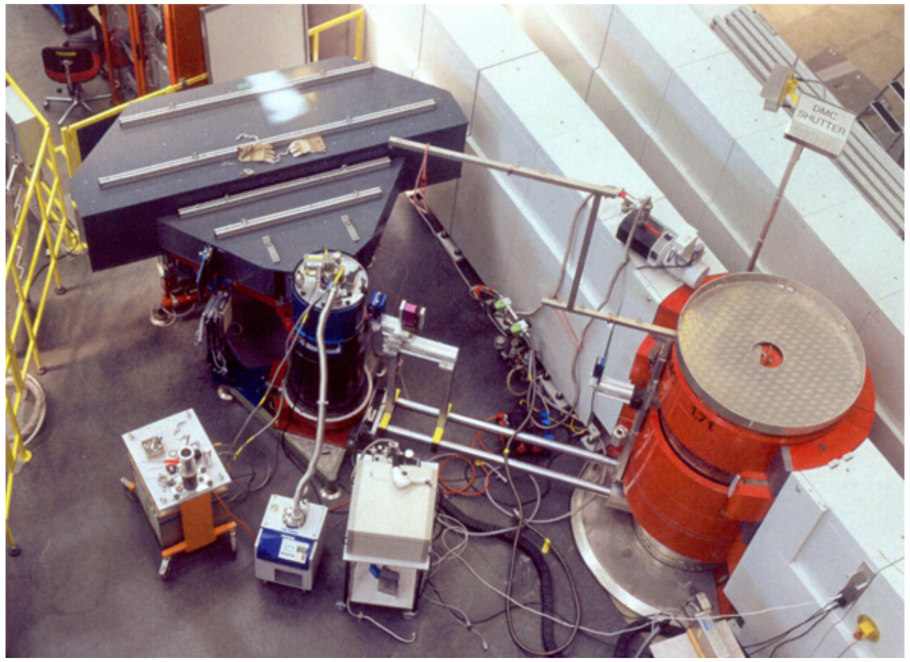
# Powder ND at SINQ/PSI

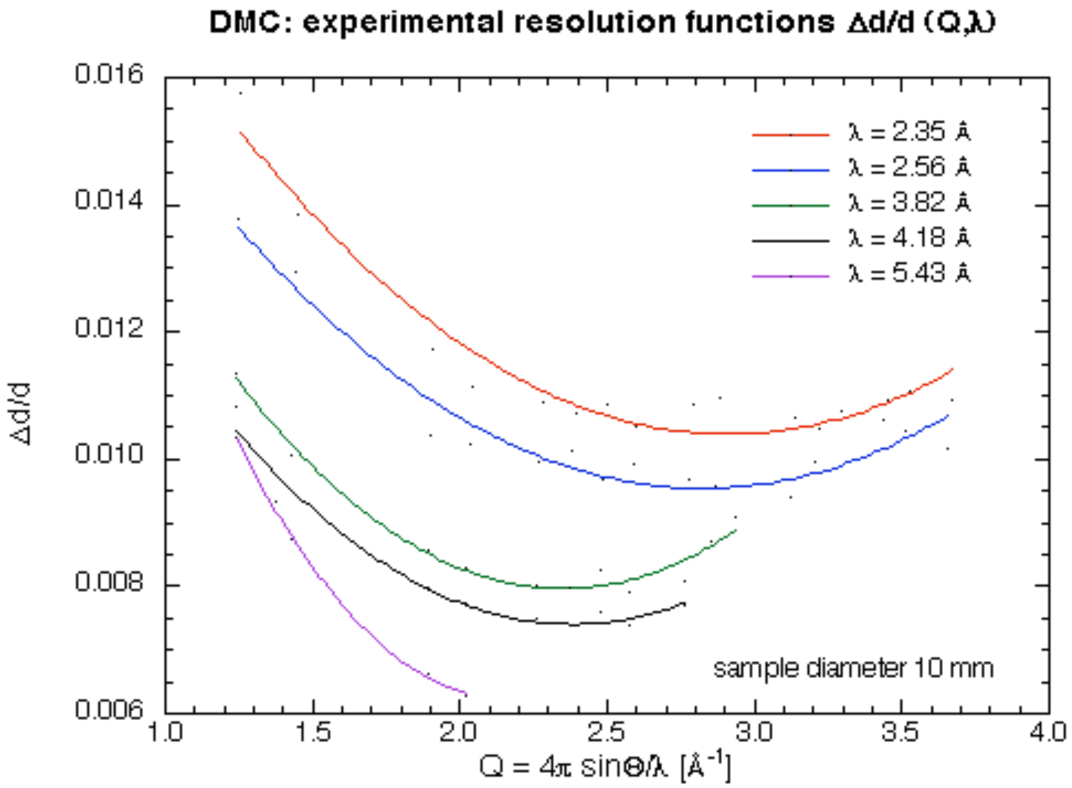
## HRPT - High Resolution Powder Diffractometer for Thermal Neutrons at SINQ





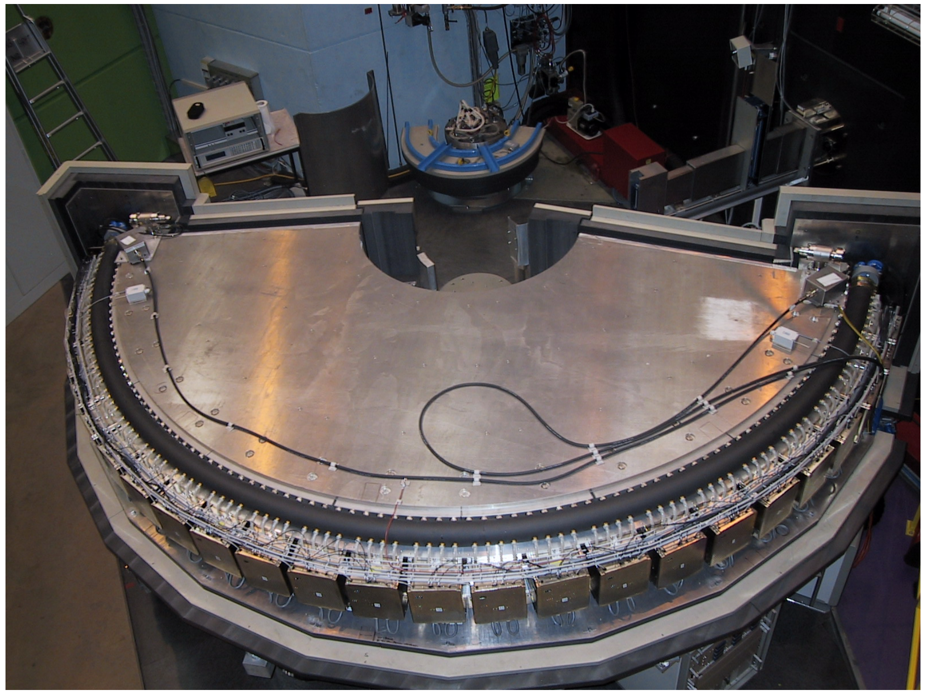

## DMC - cold neutron powder diffractometer





# Powder ND at SINQ/PSI

## HRPT - High Resolution Powder Diffractometer for Thermal Neutrons at SINQ



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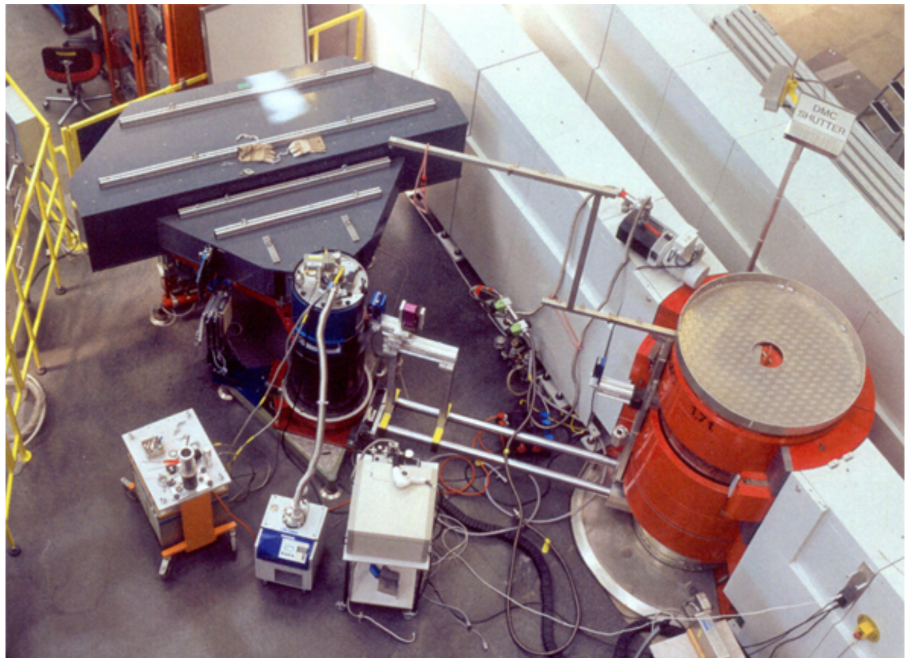
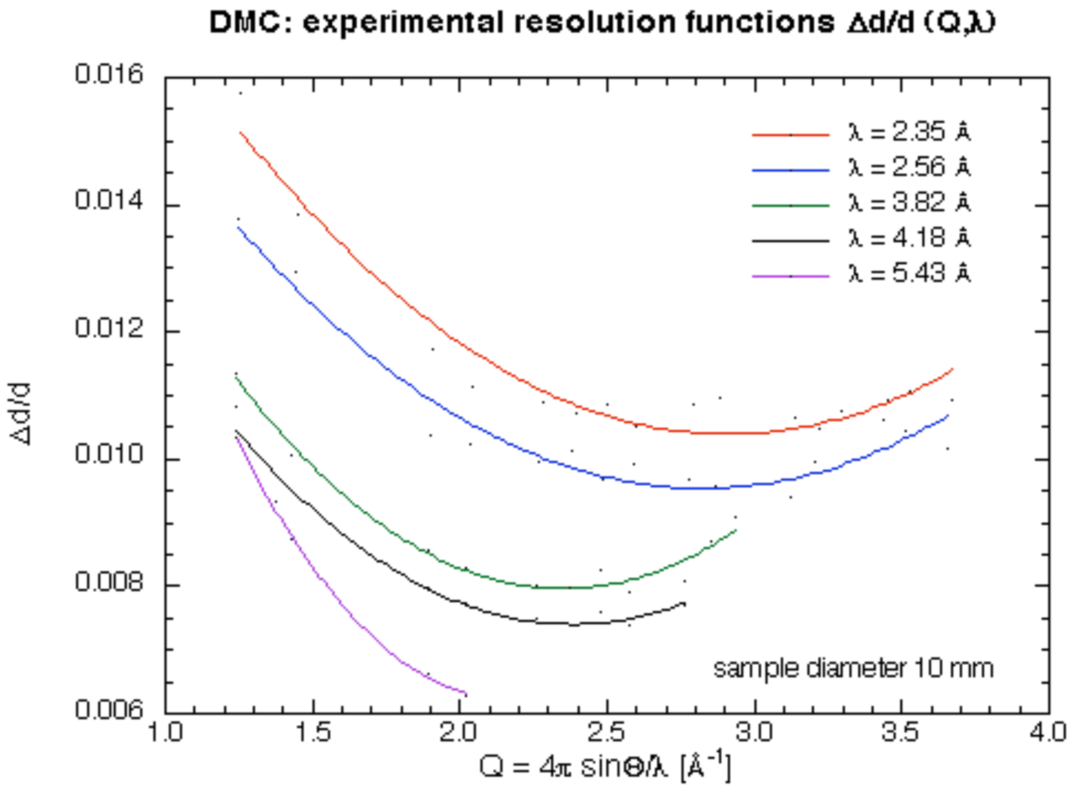
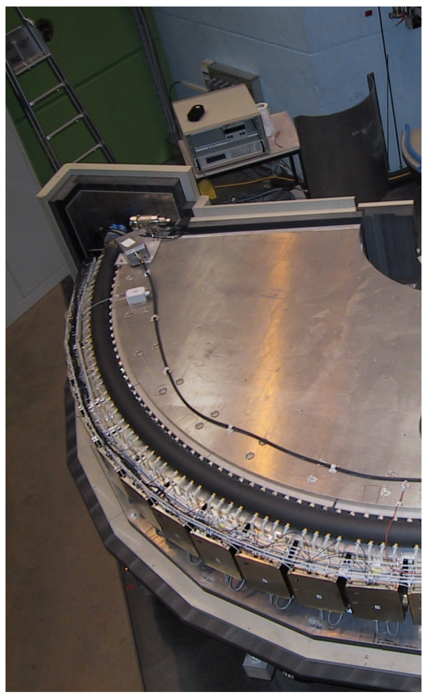
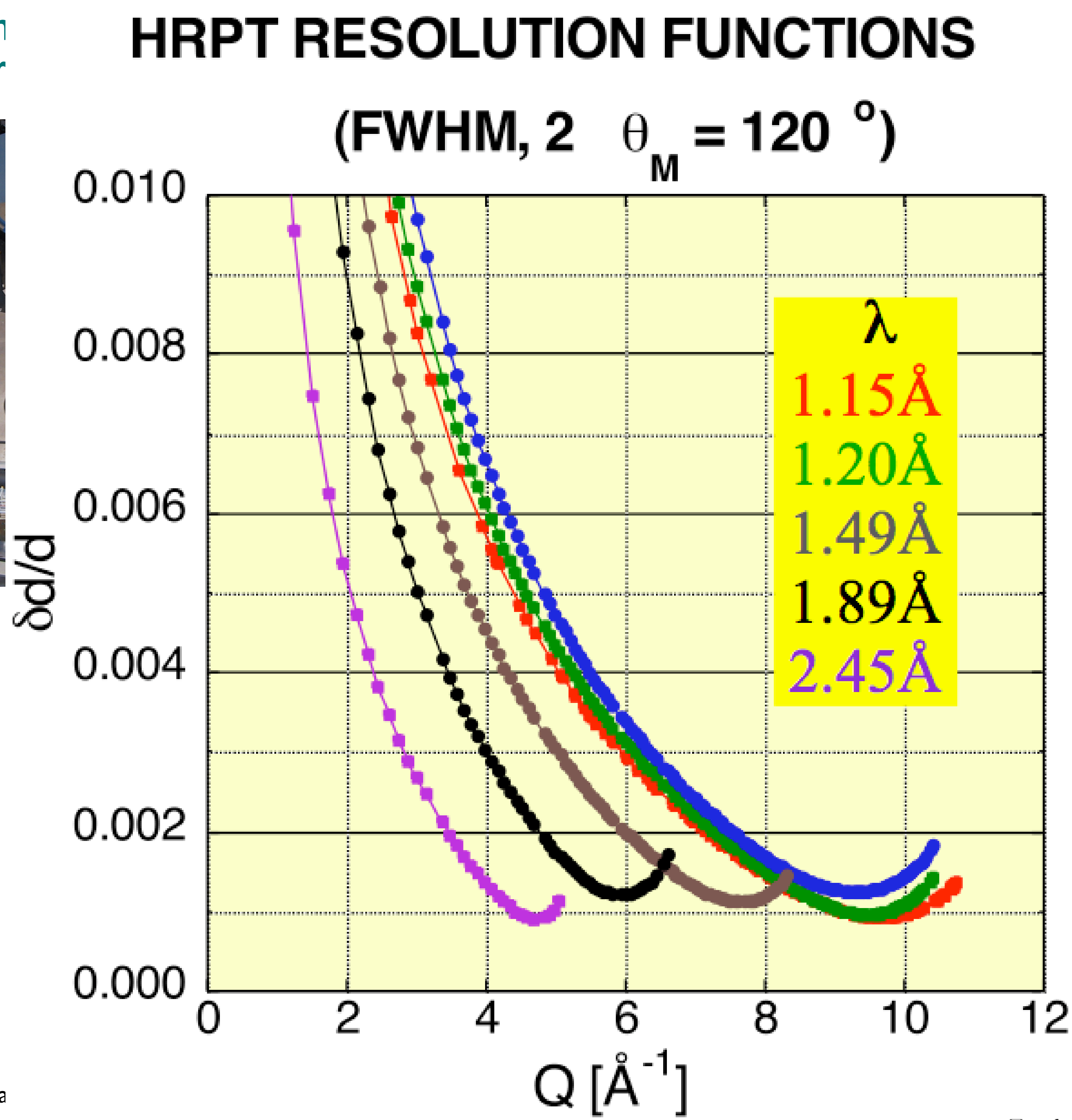




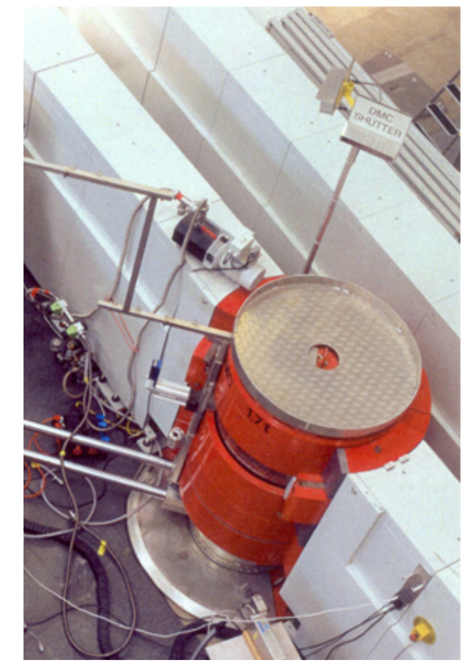
# Powder ND at SINQ/PSI

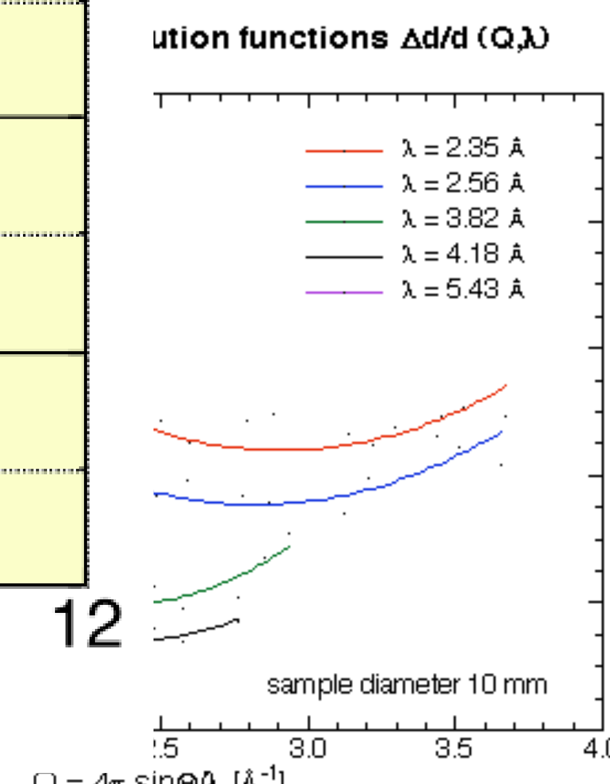
## HRPT - High
**Diffractometer for**





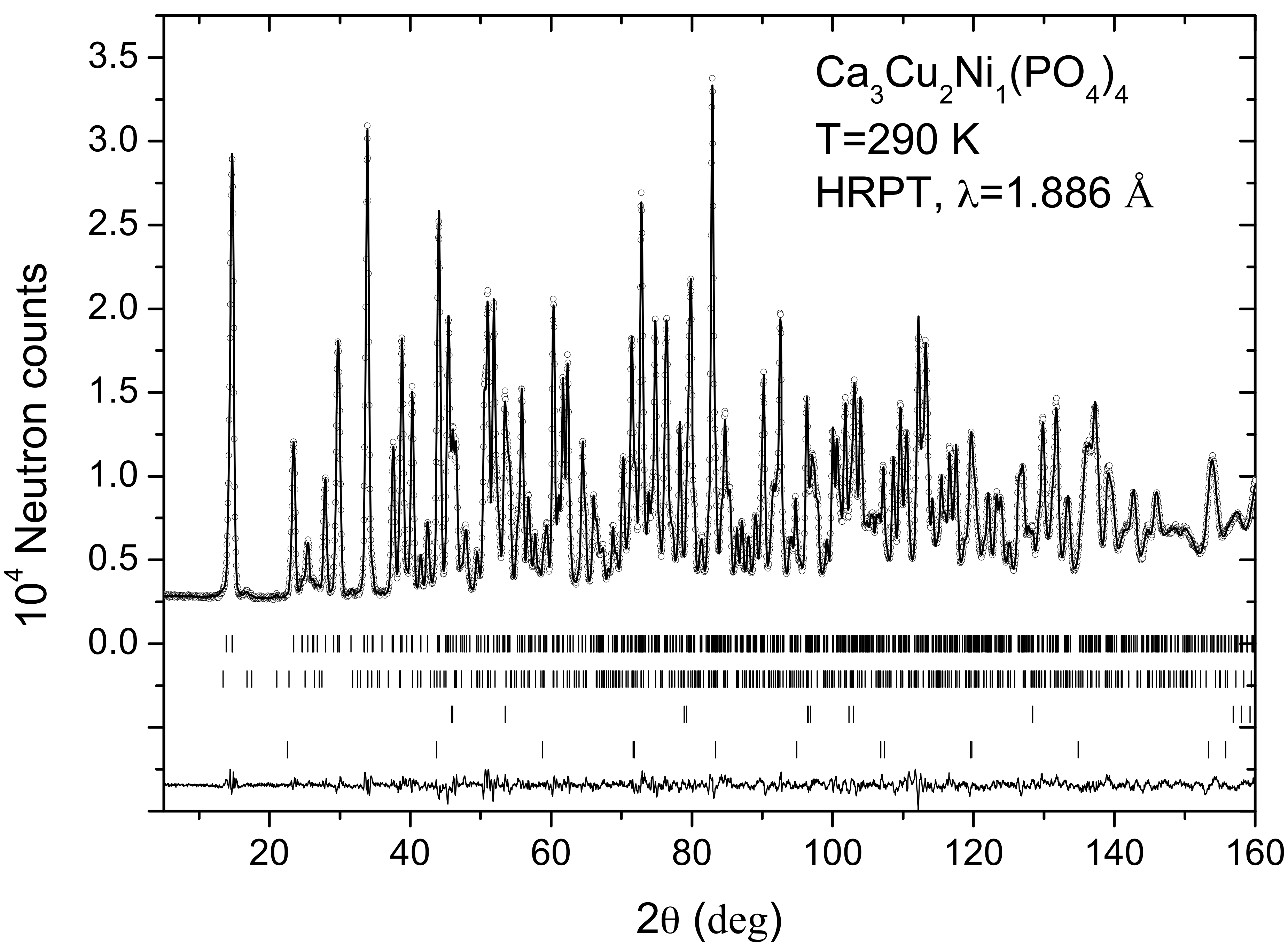

**powder diffractometer**





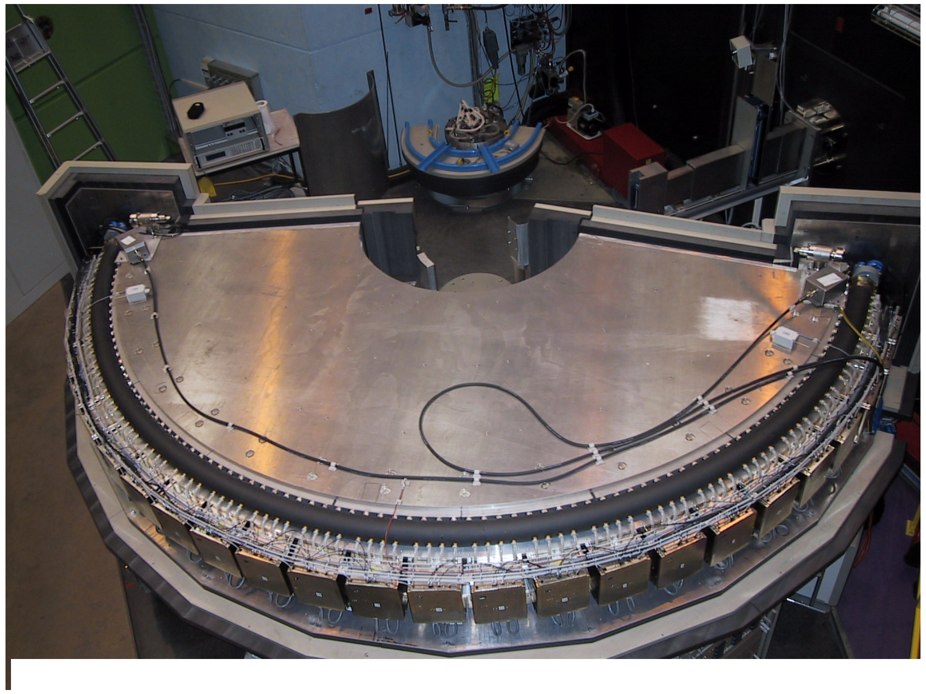

PSI information da

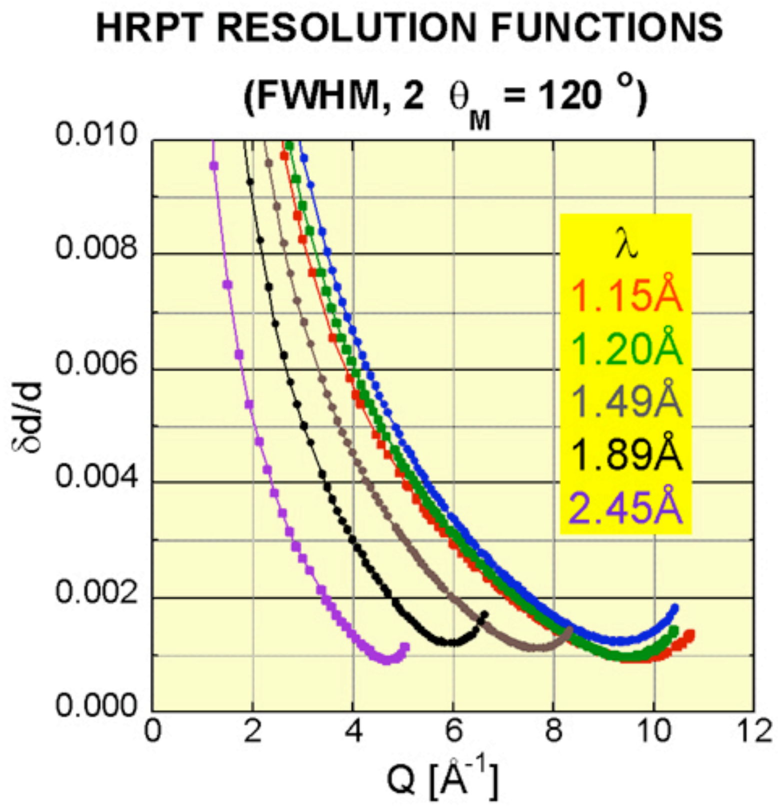
# Example of HRPT diffraction pattern



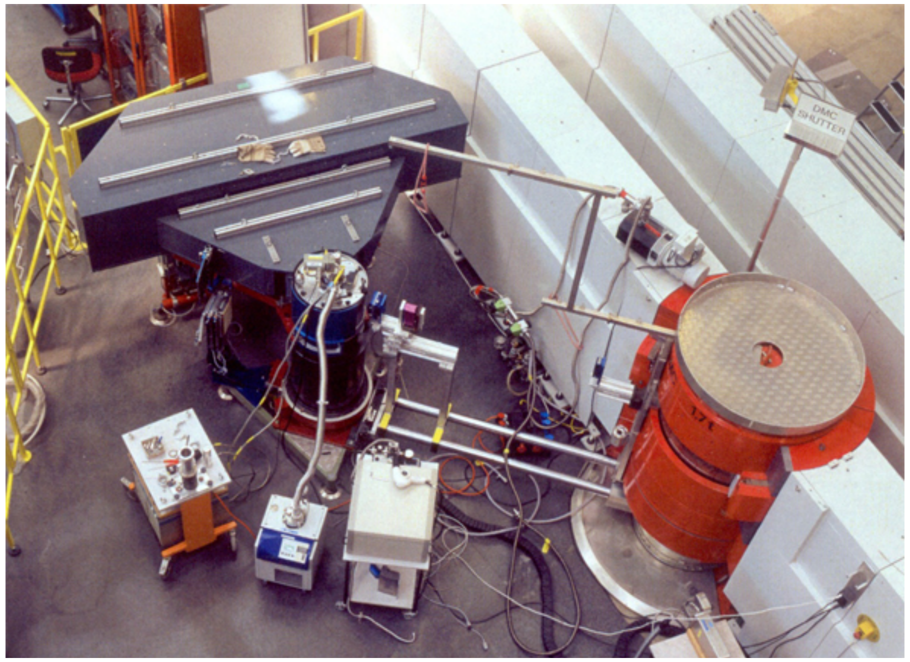
# Powder ND at SINQ/PSI

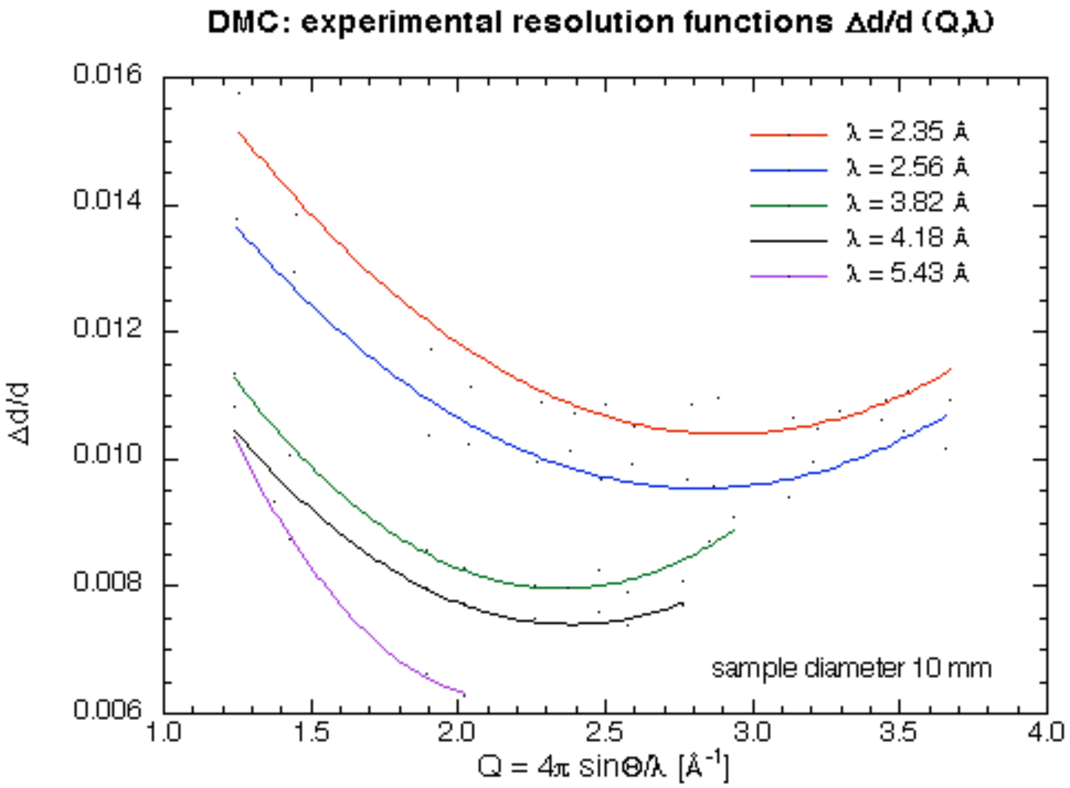
**HRPT** - High Resolution Powder Diffractometer for Thermal Neutrons at SINQ



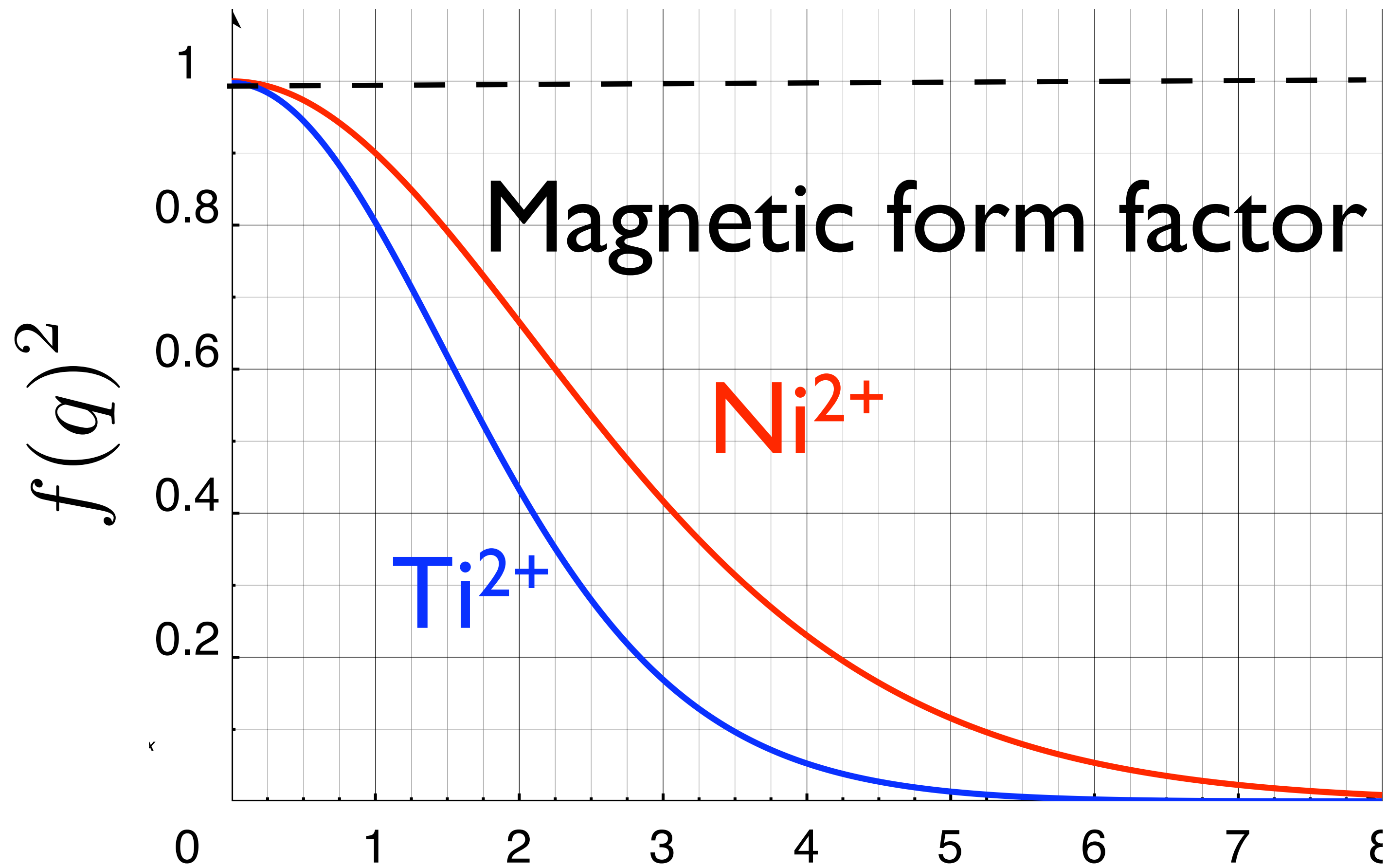


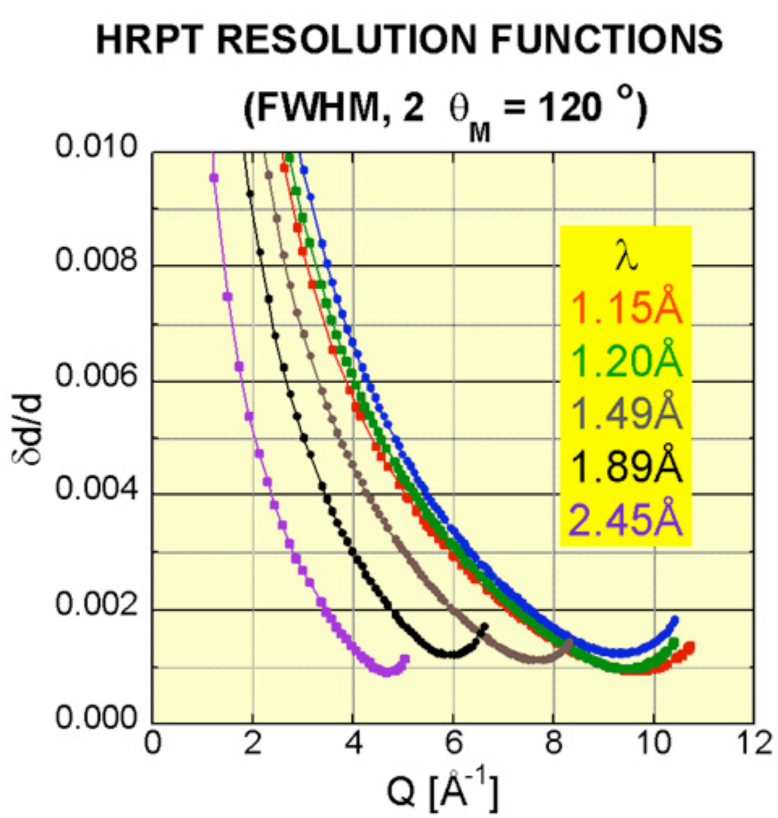

**DMC** - cold neutron powder diffractometer

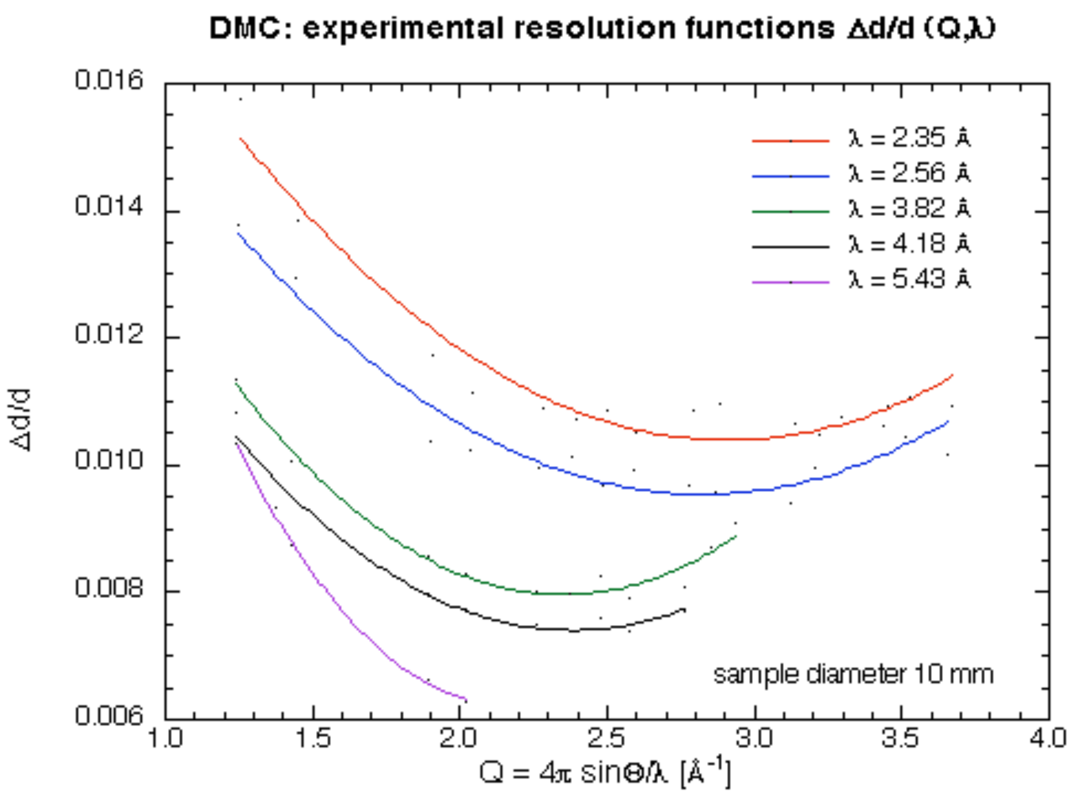




# Powder ND at SINQ/PSI

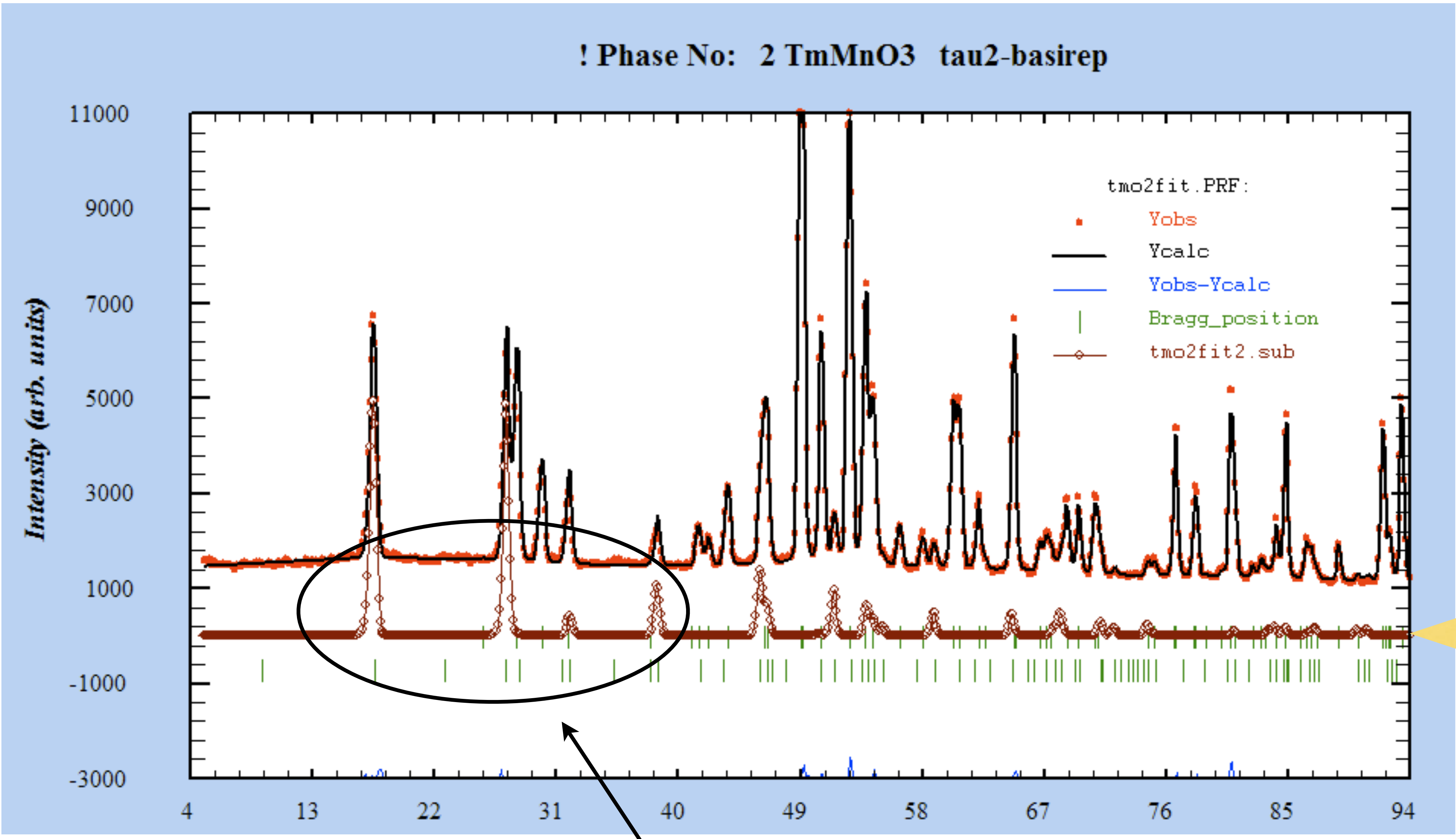


# cf. resolution/q-range

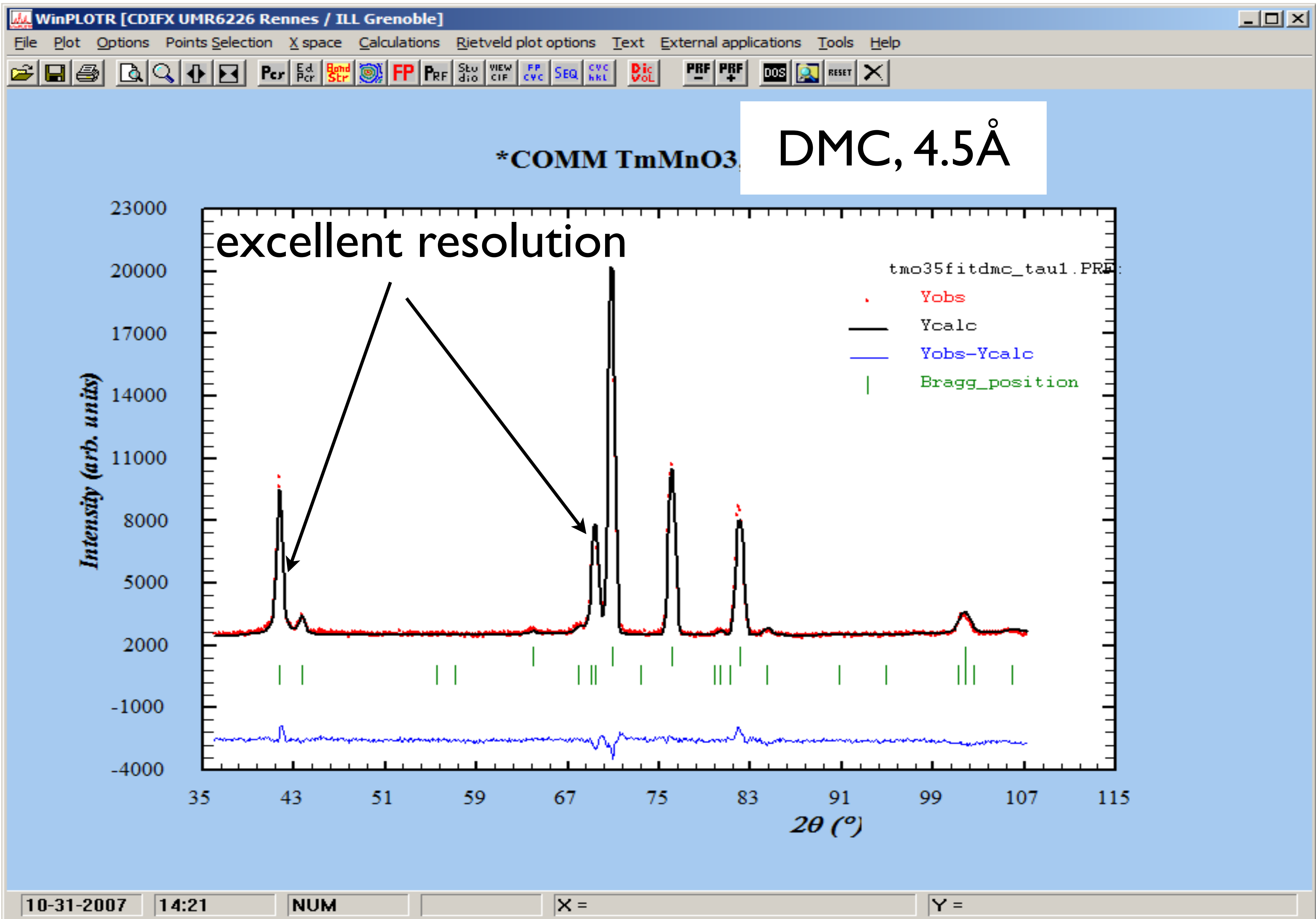
HRPT 1.9Å



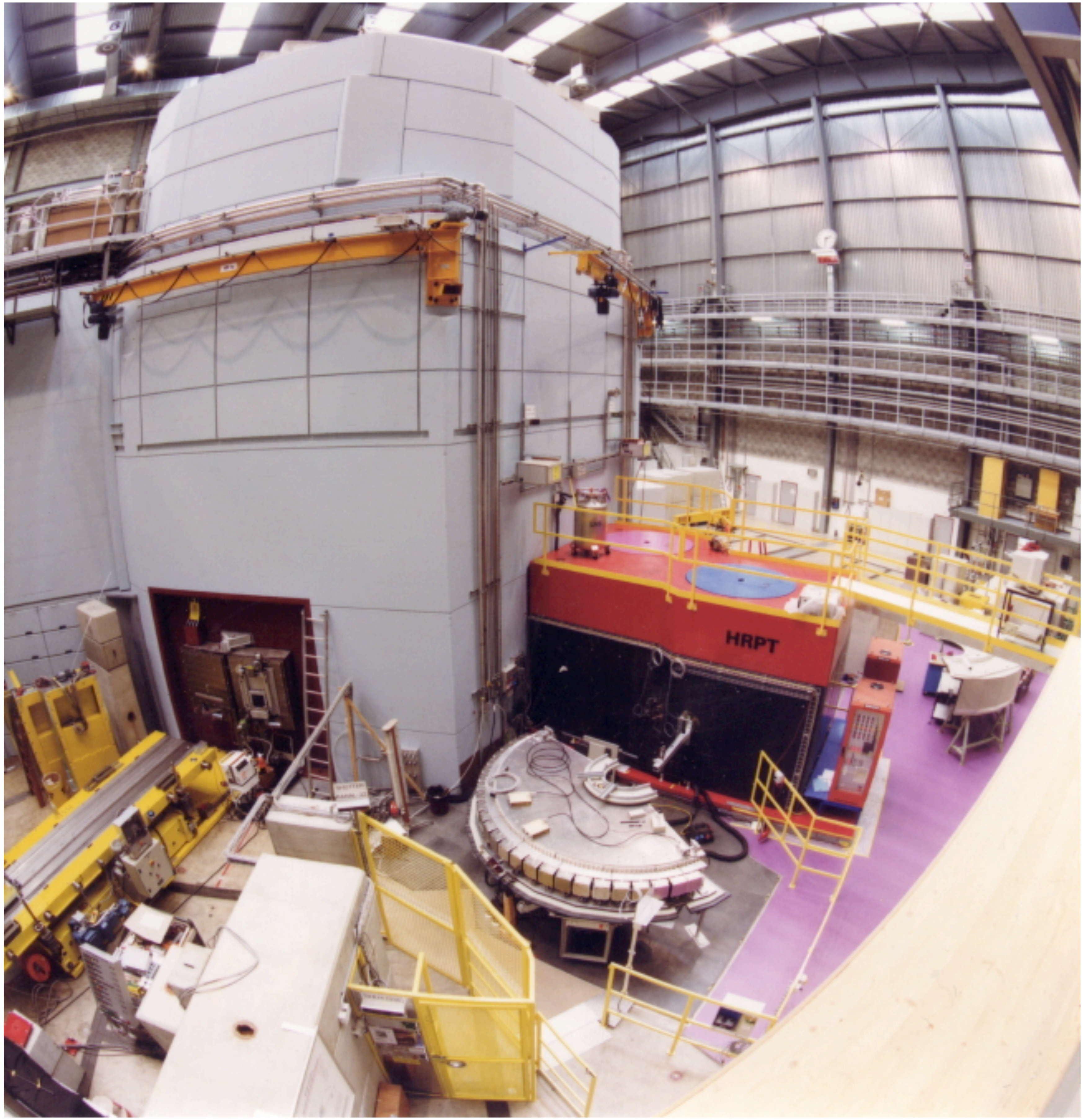

magnetic contribution

DMC range at 4.5Å

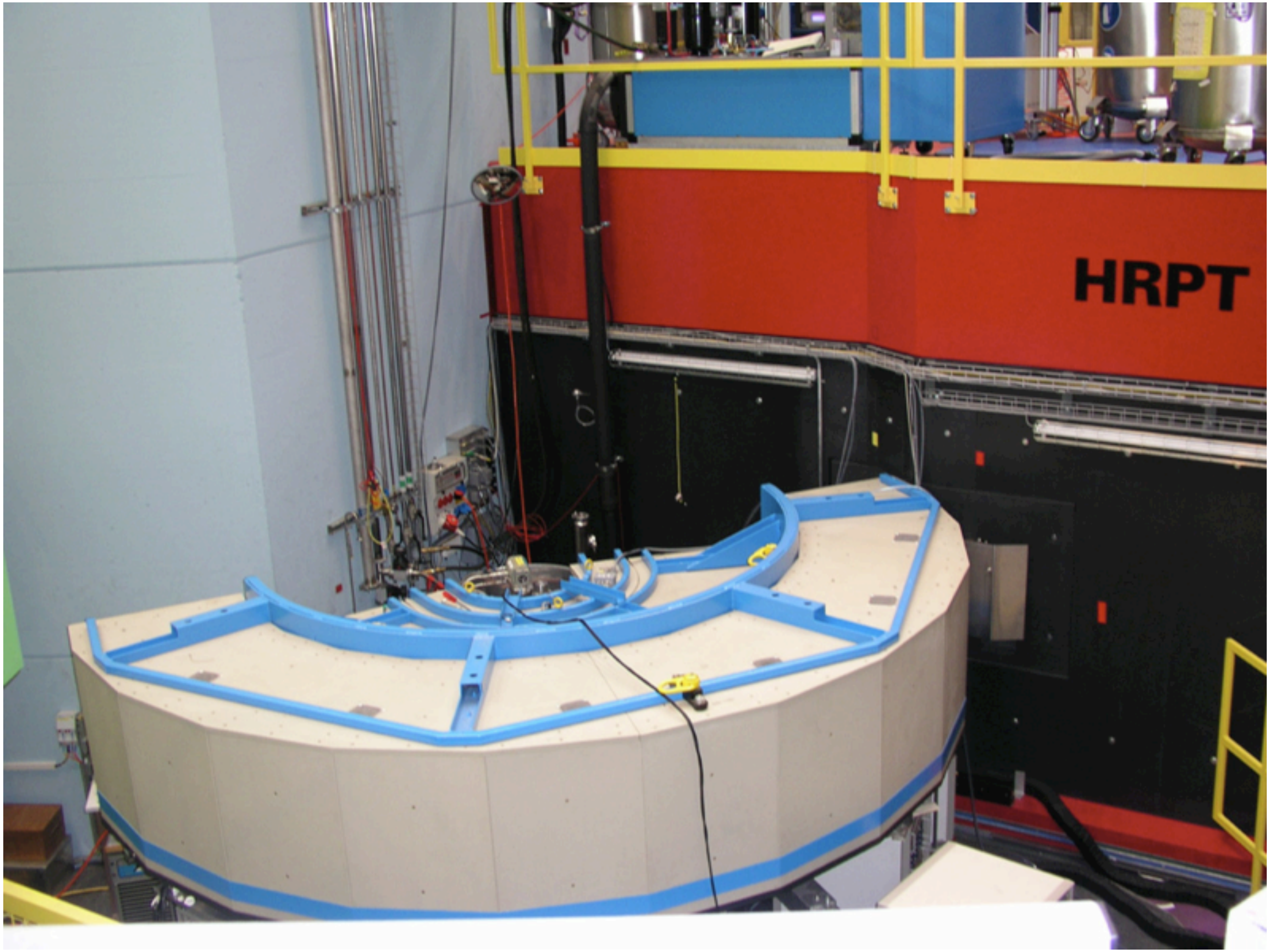
# Cf. resolution/q-range



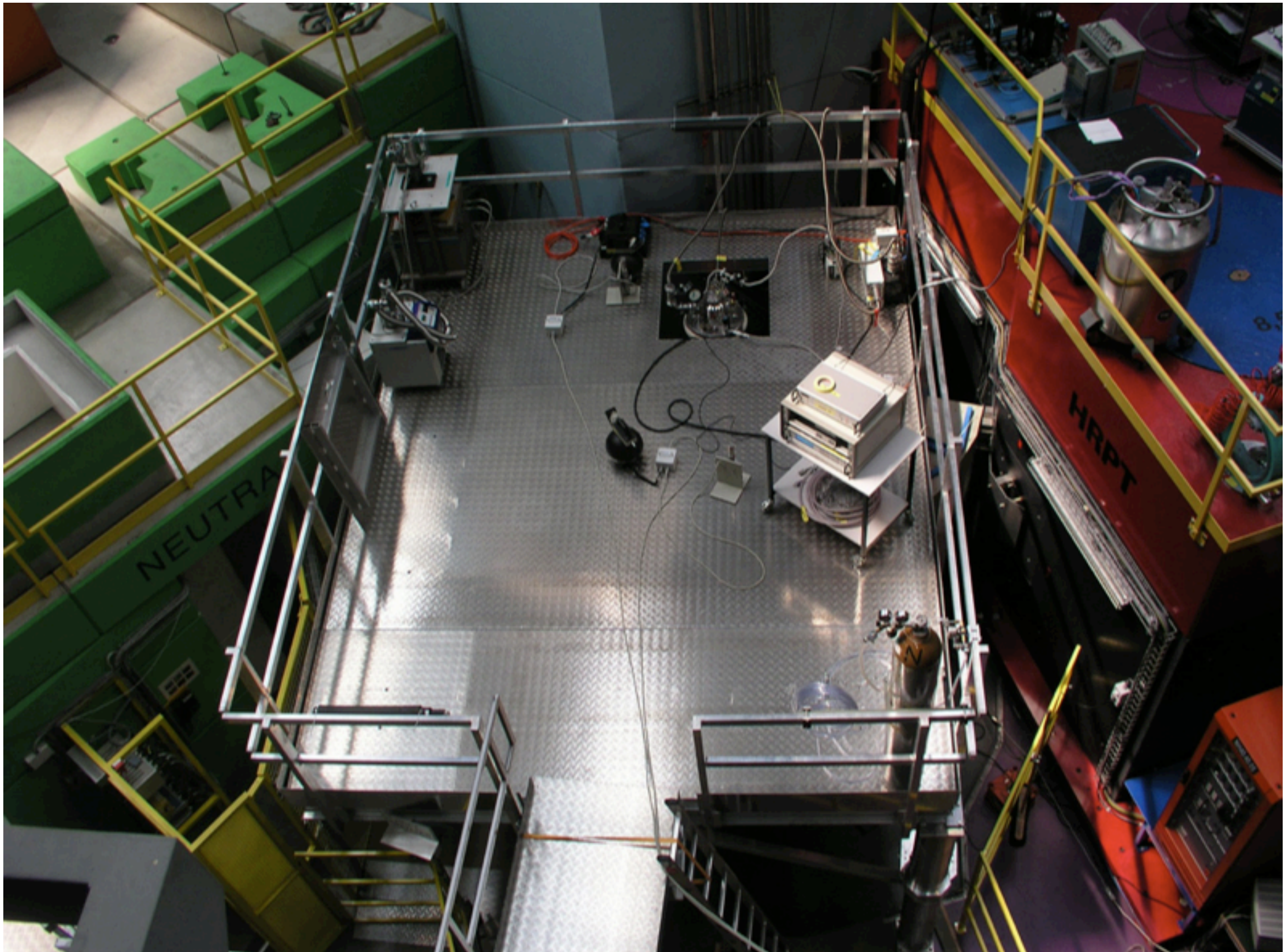
# SINQ hall



# Instrument view at SINQ taget station



# Instrument view at SINQ taget station



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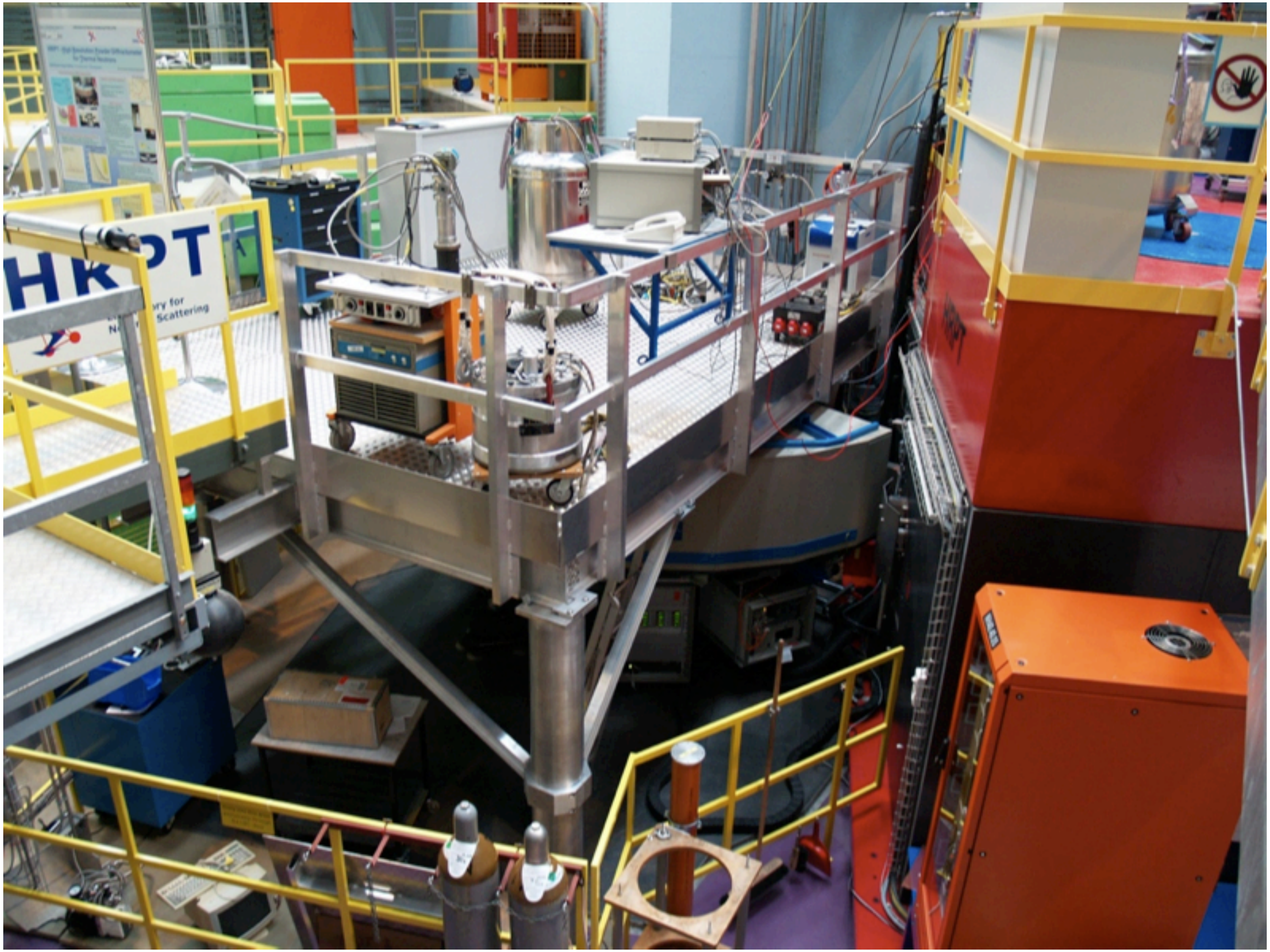
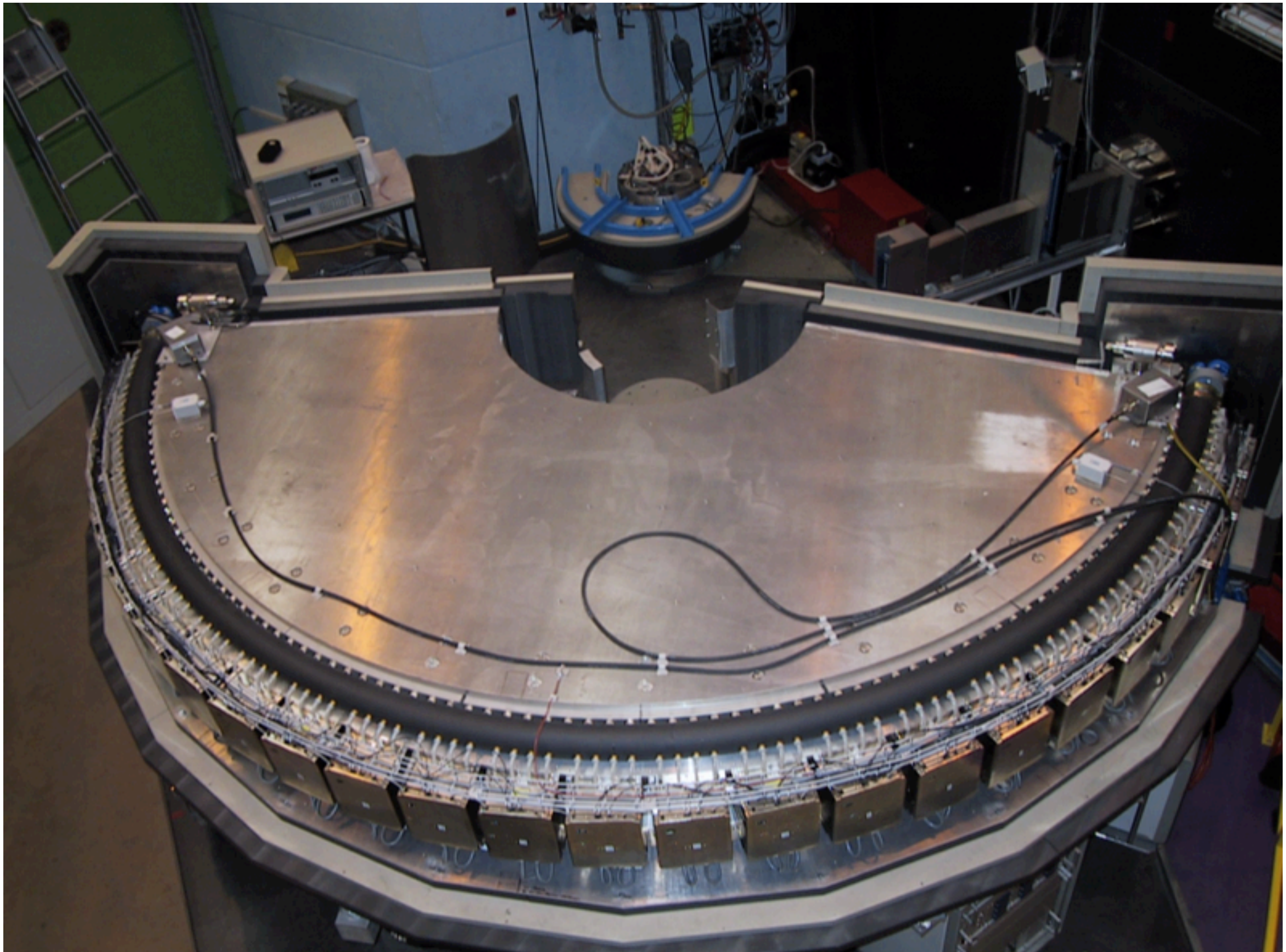
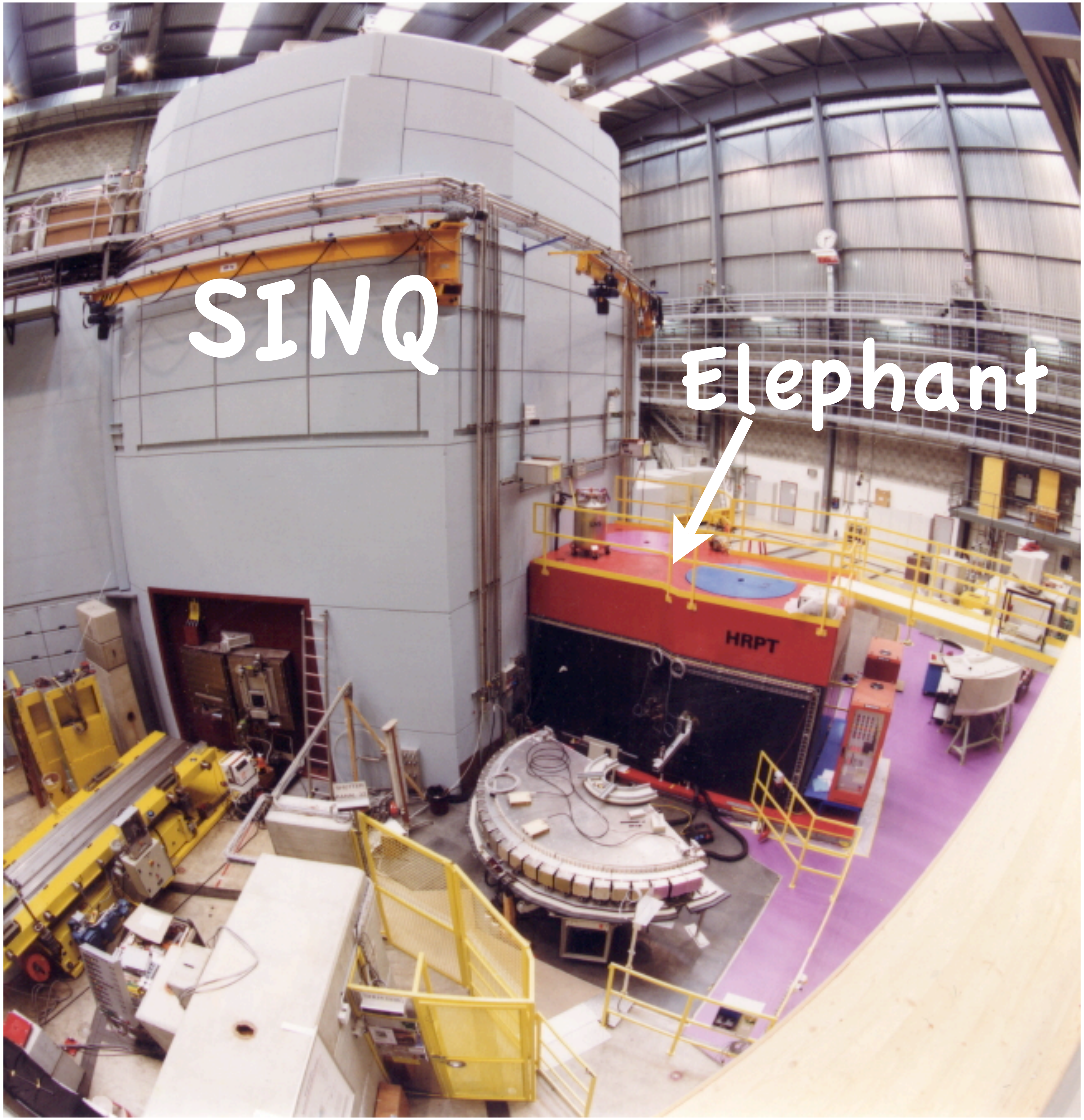
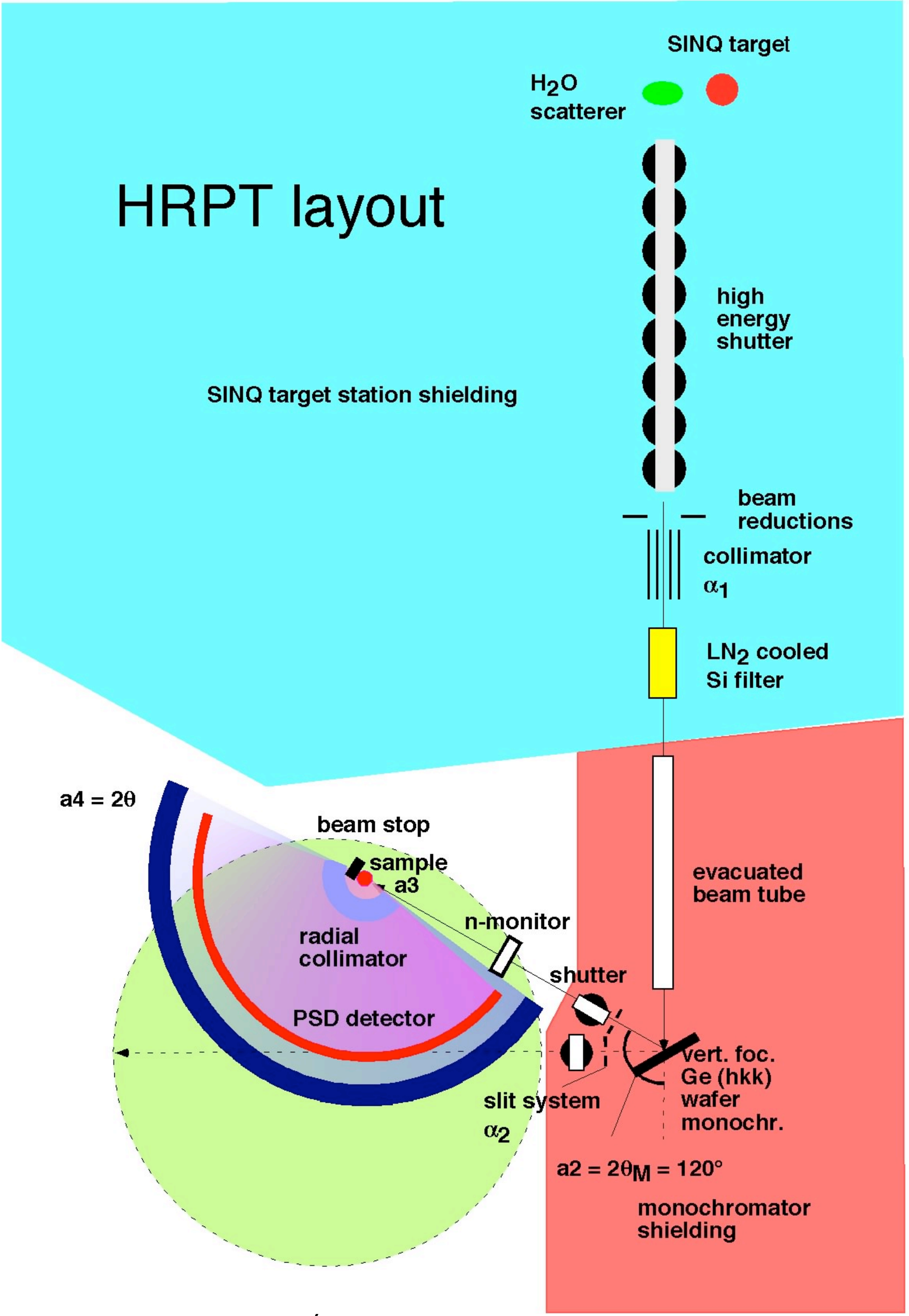


# Instrument view at SINQ taget station

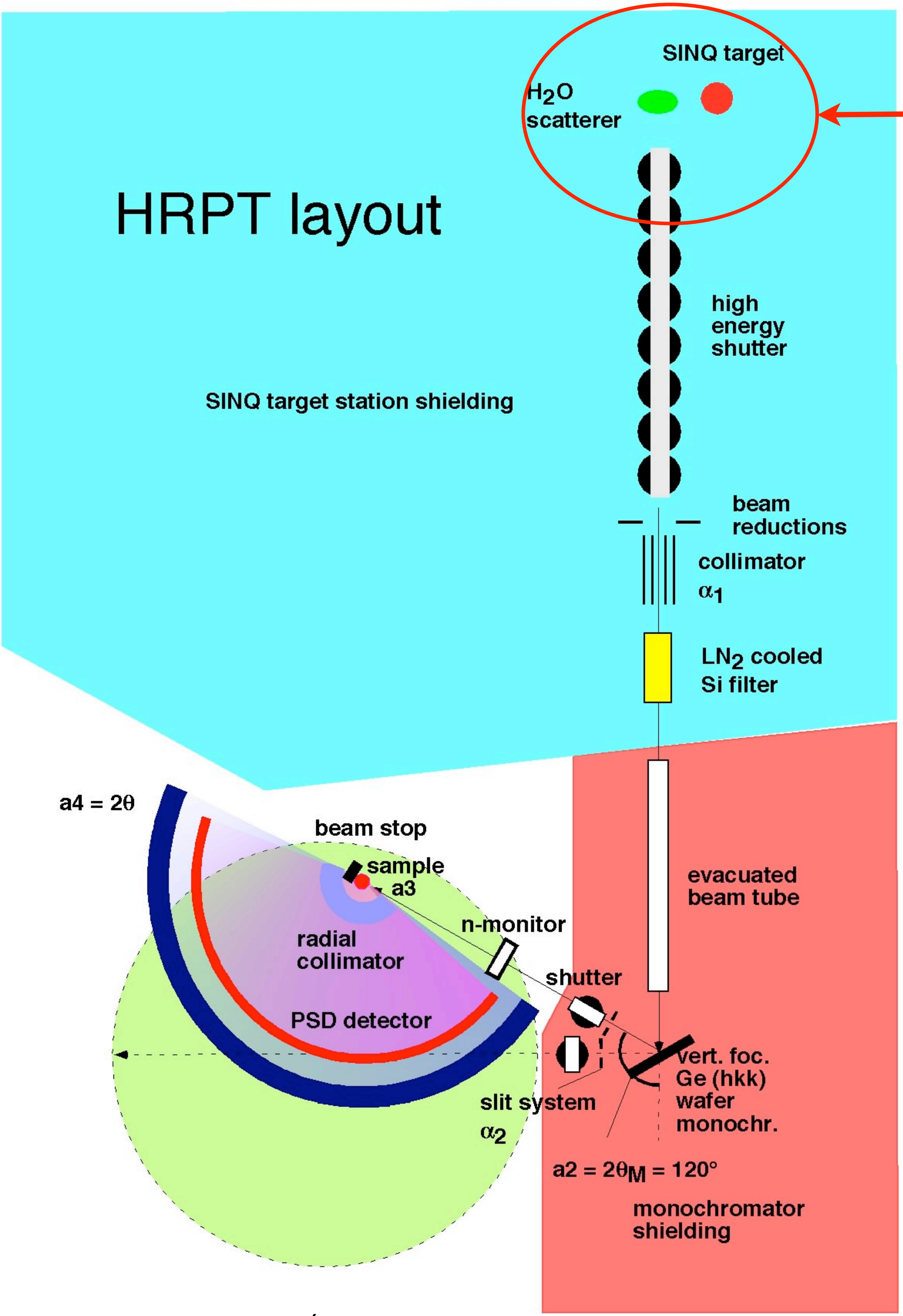


# SINQ hall



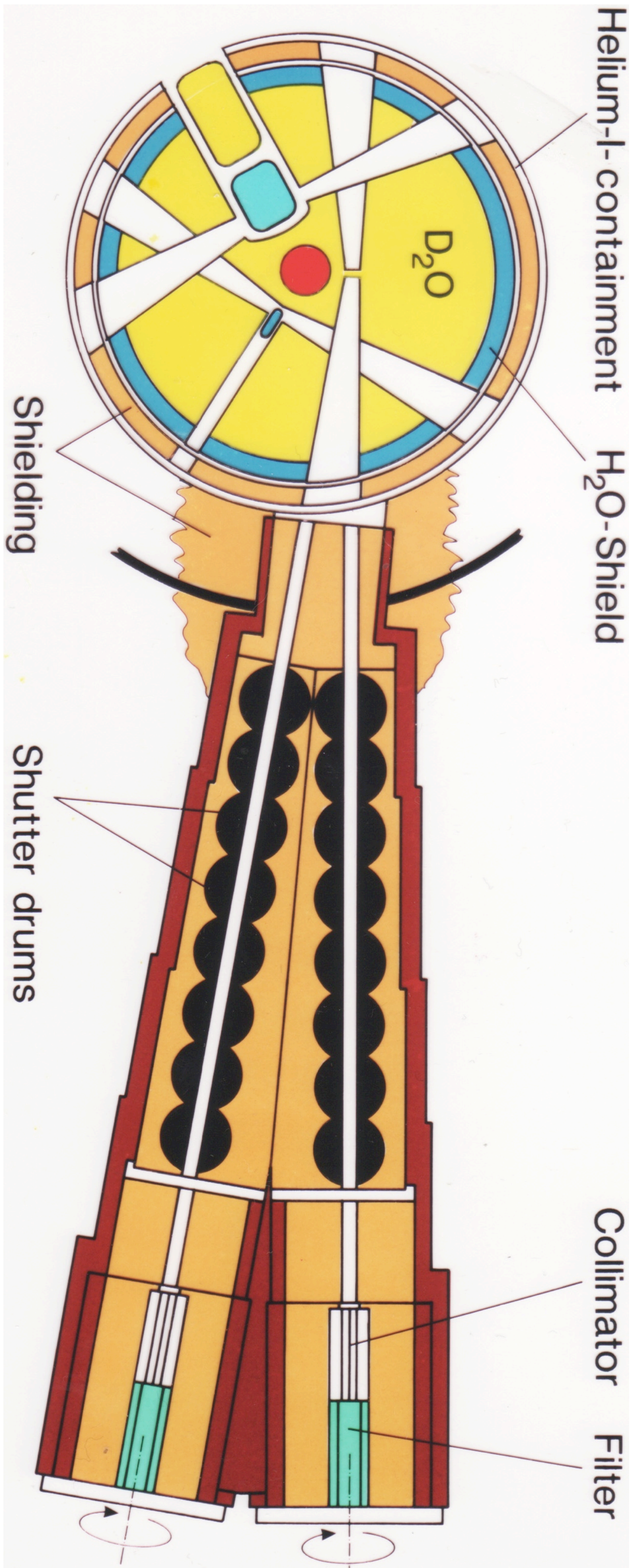


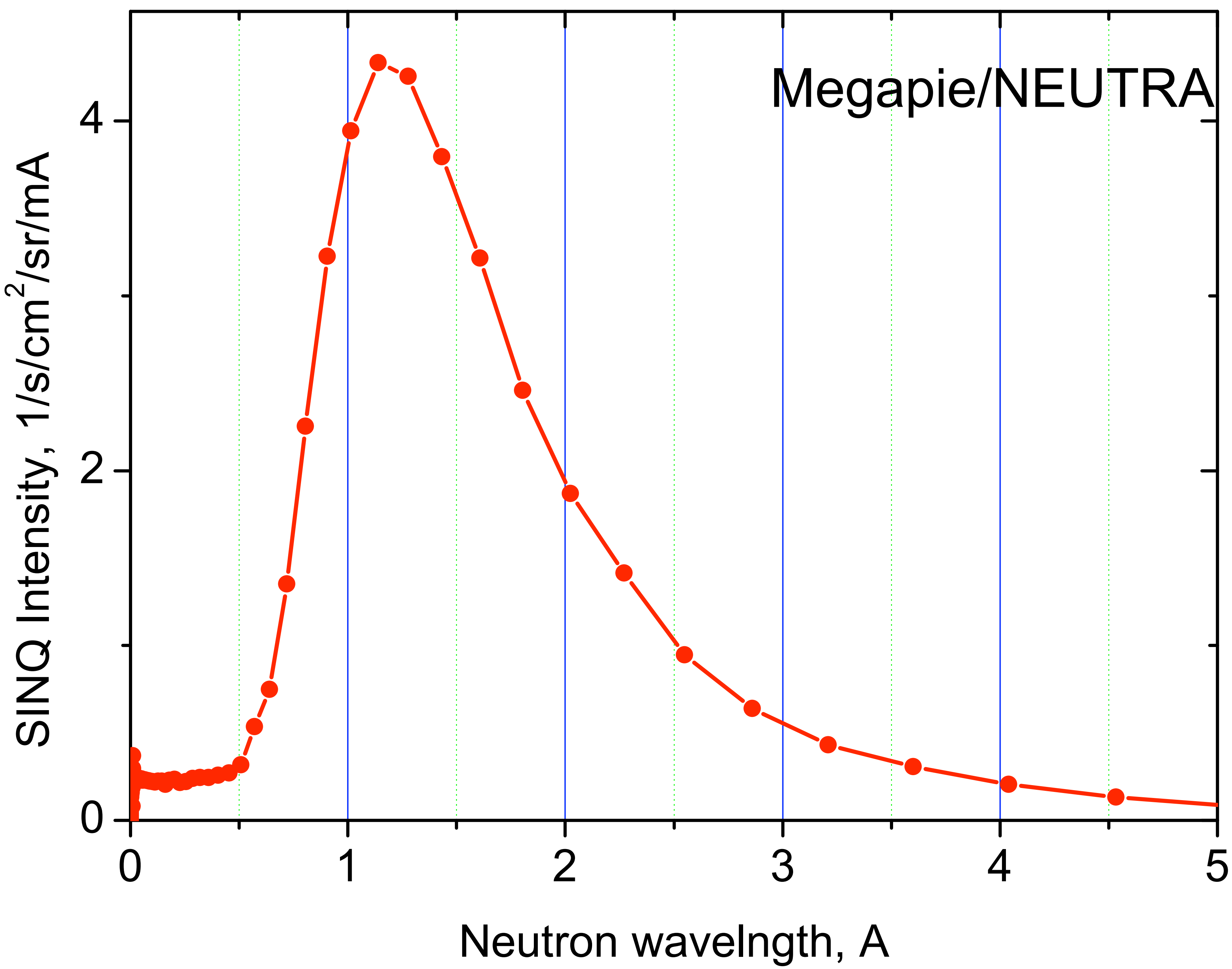
SINQ target
$H_2O$ scatterer
HRPT layout
high energy shutter
SINQ target station shielding
beam reductions
collimator $\alpha_1$
$LN_2$ cooled Si filter
$a4 = 2\theta$
beam stop
sample
a3
evacuated beam tube
n-monitor
radial collimator
shutter
PSD detector
vert. foc. Ge (hkk) wafer monochr.
slit system $\alpha_2$
$a2 = 2\theta_M = 120°$
monochromator shielding

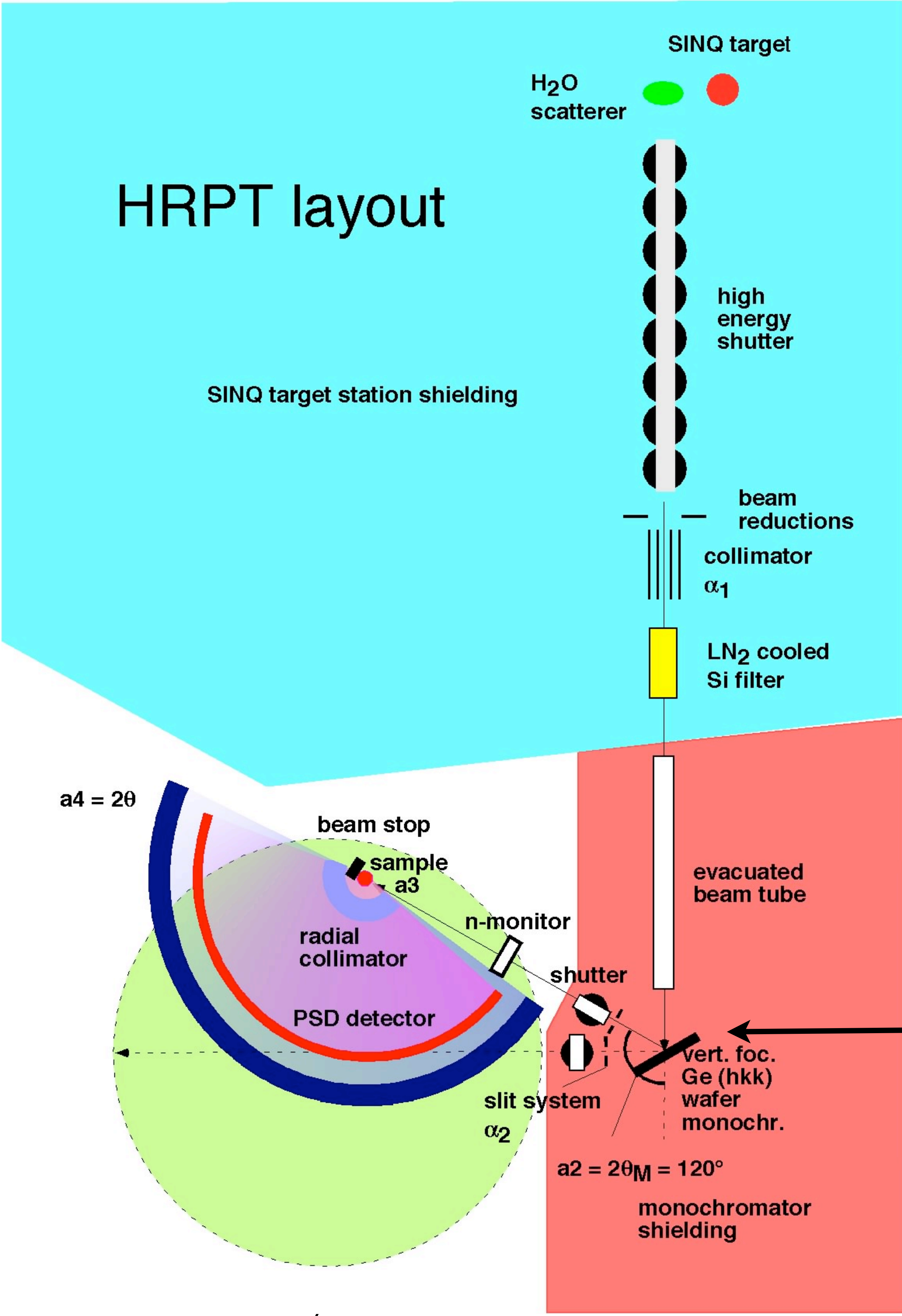



neutron flux from the moderator

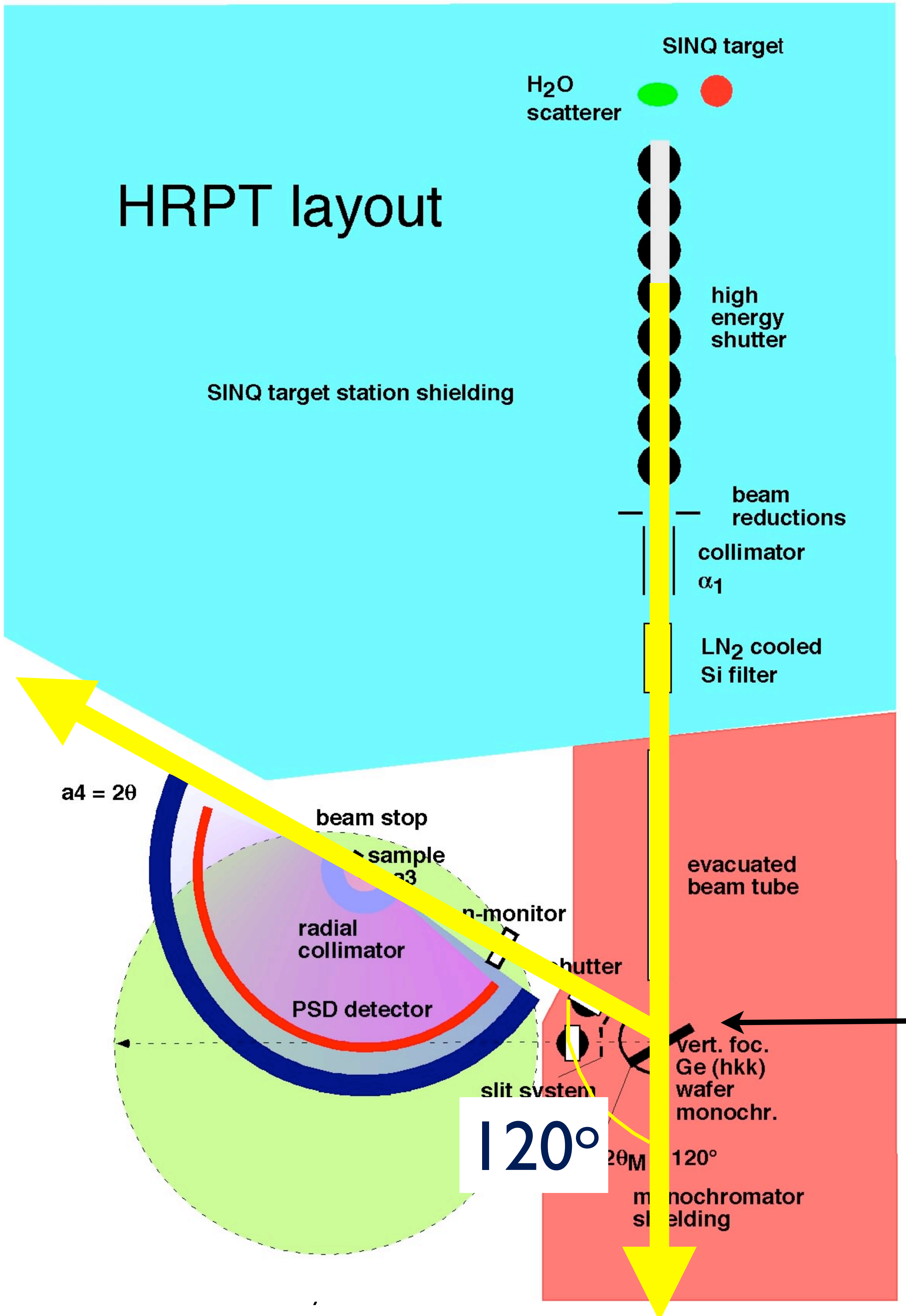
# Neutron flux from the $D_2O$ moderator at NEUTRA (white beam)



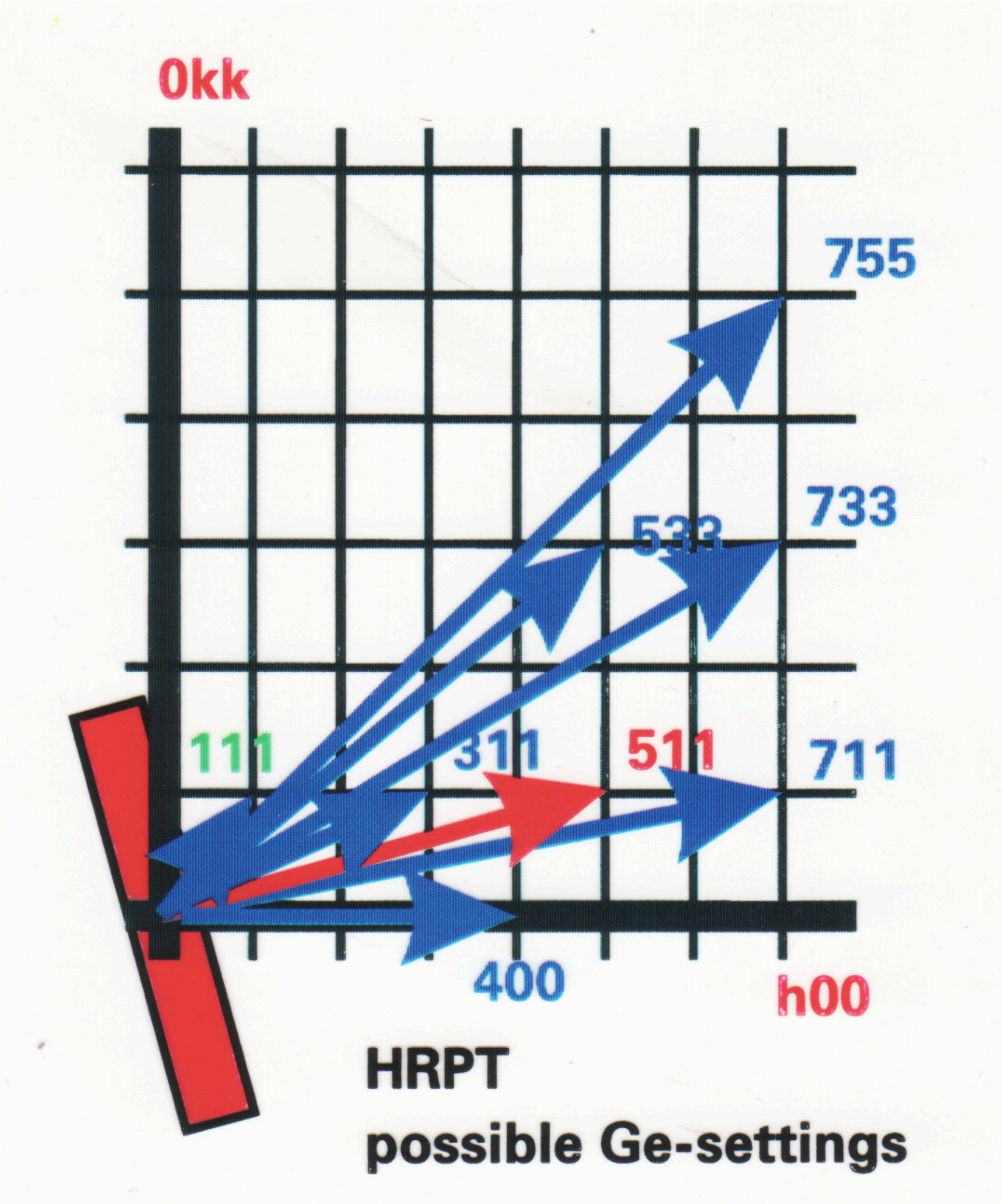



neutron monochromator
fixed 120 take off angle



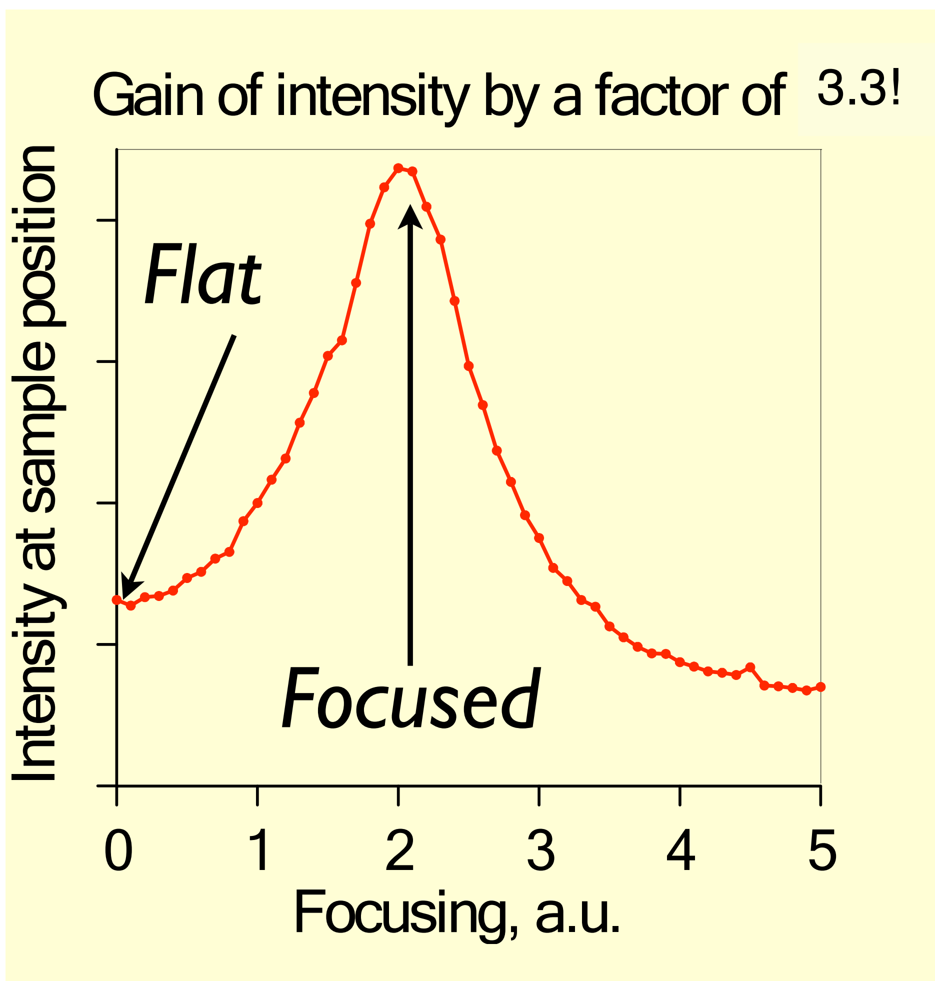

neutron monochromator
fixed 120 take off angle

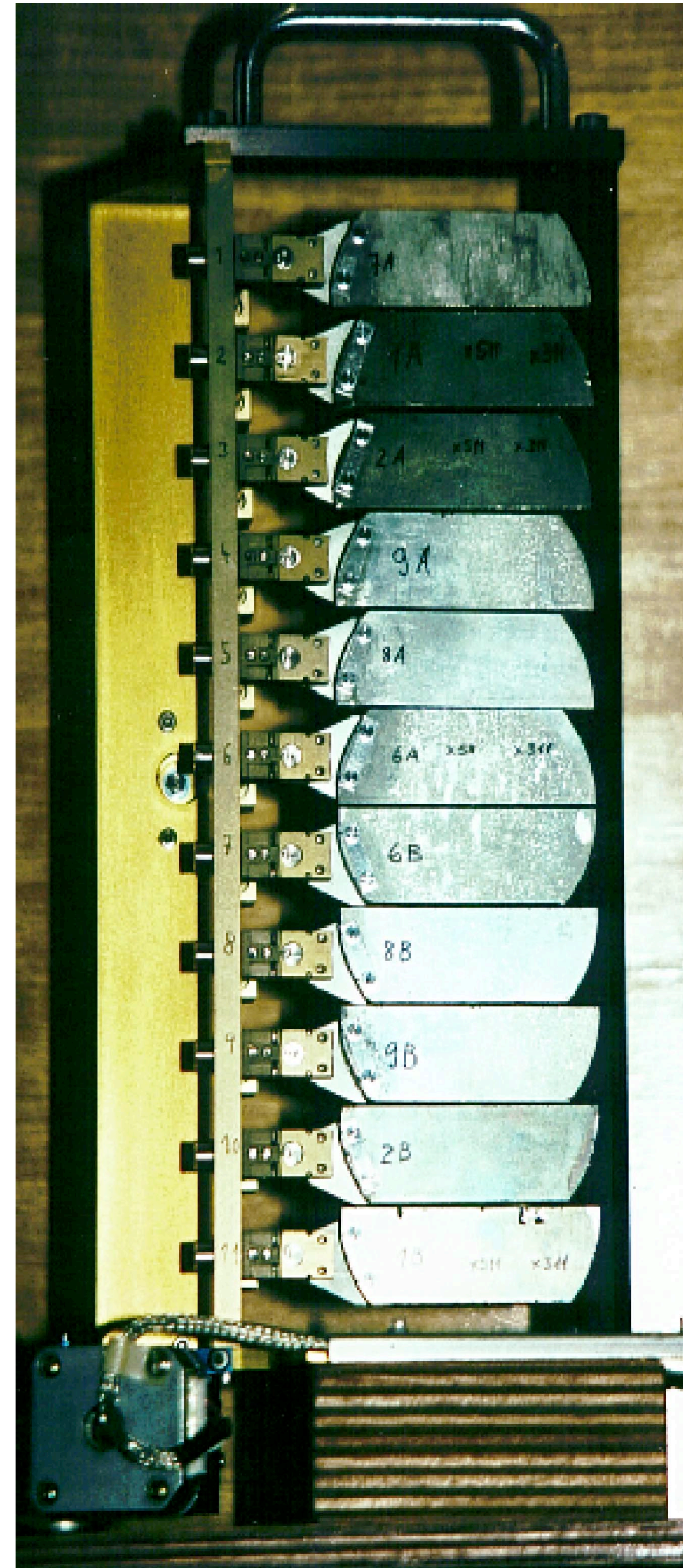
# Ge hkk focusing monochromator



# Ge hkk focusing monochromator

<u>Monochromator hight</u>: 11slabs*25=255mm
<u>Beam spot hight</u> at sample position
is smaller due to vertical focusing: 60mm
<u>Wavelength</u> is chosen by rotation along (hkk)
<u>Mosaic</u> 15'

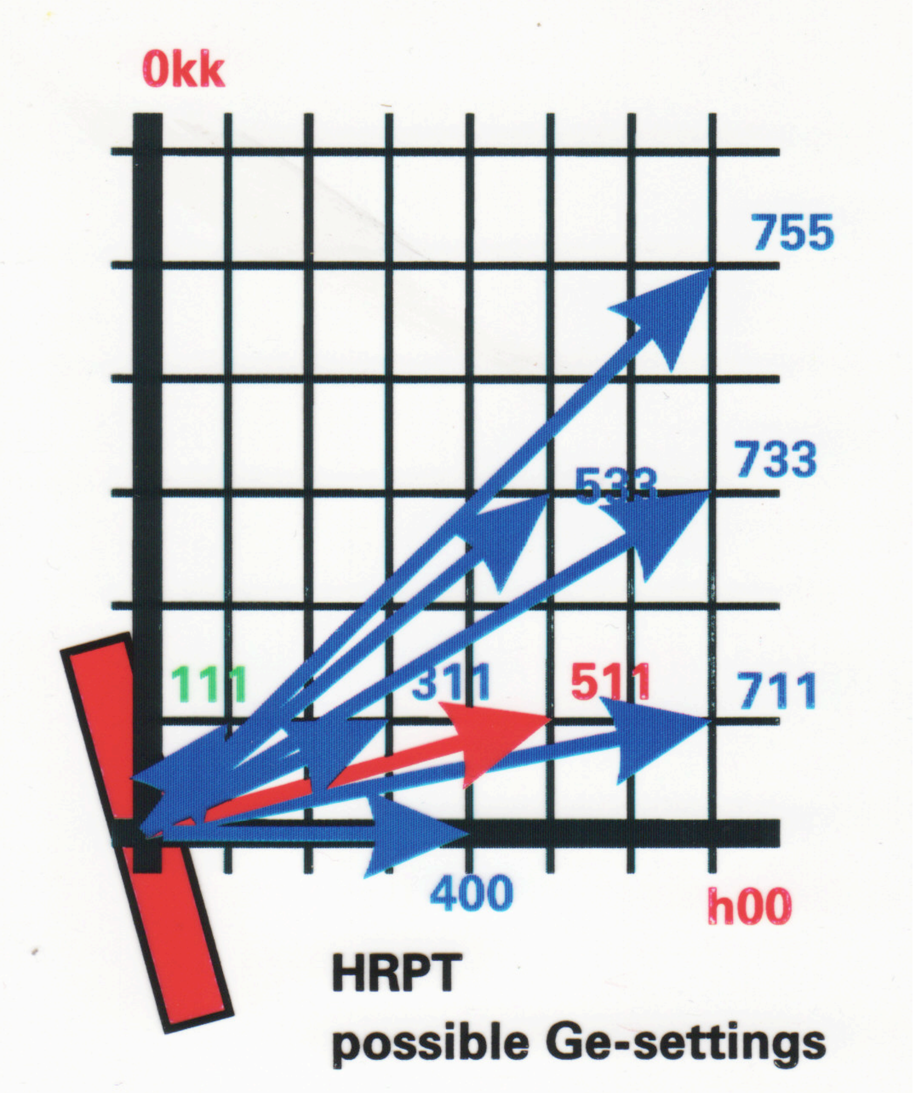


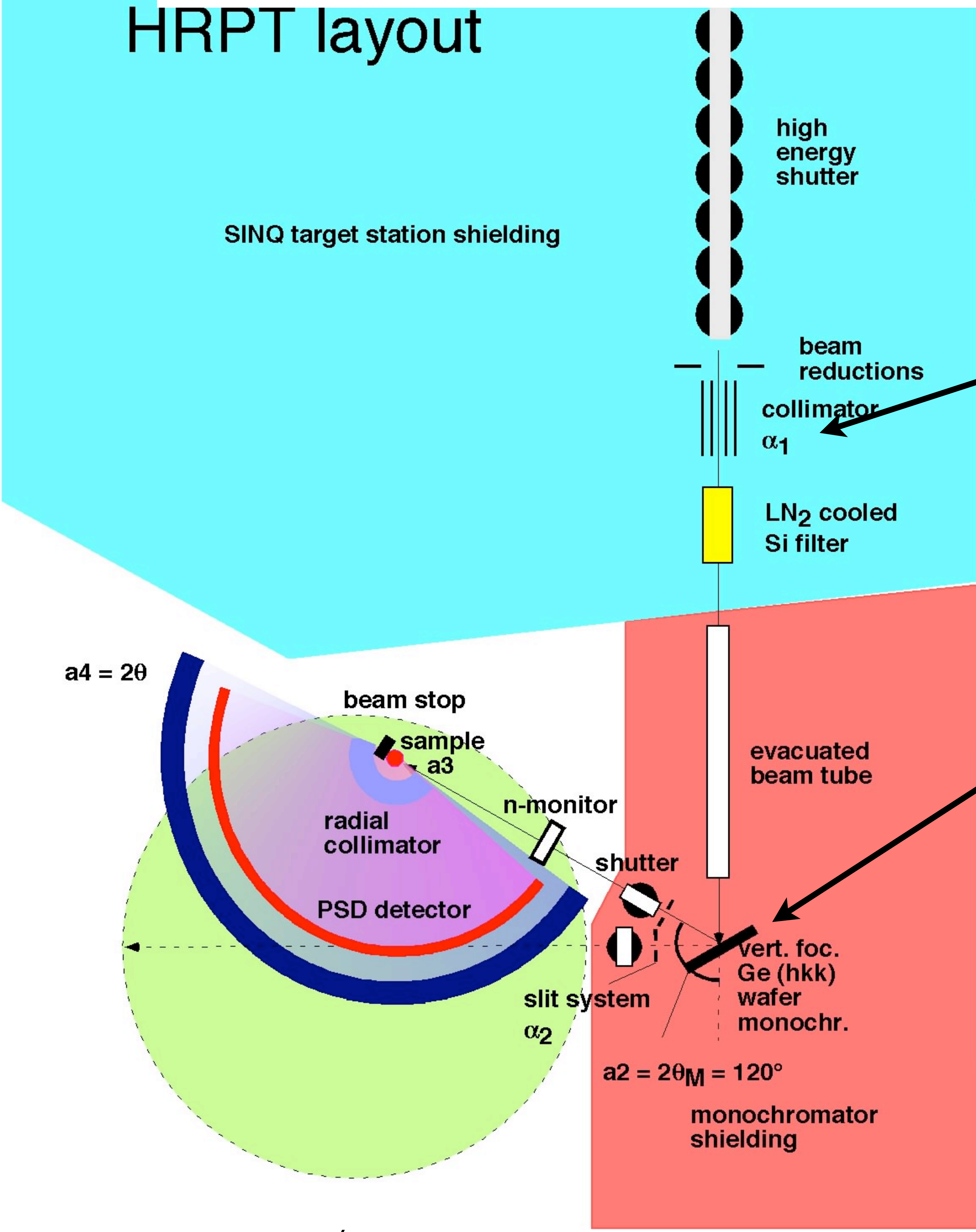
# Flexible choice of wavelength, resolution/intensity

- Wavelength is selected by (hkk) plane of Ge-monochromator
- Resolution and intensity are controlled by appropriate primary/secondary collimations and take-off-angle of the monochromator (120° or 90°)

| | $2\theta_M$=90° | | $2\theta_M$ = 120° | |
|---|---|---|---|---|
| (hkk) Ge | λ, Å | Effective intensity | λ, Å | Effective intensity |
| 311 | 2.40971 | 0.64 | 2.9536 | ~0.16 |
| 400 | 1.9984[4,5] | | 2.449[1,3] | 0.19 |
| 133 | 1.8324 | 1.00 | 2.246[1,2] | |
| 511 | 1.5384 | 1.55 | 1.886 | 1.0 |
| 533 | 1.2183 | 0.83 | 1.494 | 0.90 |
| 711 | 1.1194 | 0.60 | 1.372 | 0.71 |
| 733 | 0.9763 | 0.34 | 1.197 | 0.63 |
| 822 | 0.9419 | 0.48 | 1.154 | 0.79 |
| 466 | | | 1.044 | 0.27 |



# HRPT resolution
# horizontal angular divergence control




$\alpha_1$

primary beam collimator(s):
6’, 12’, 24’, 30’

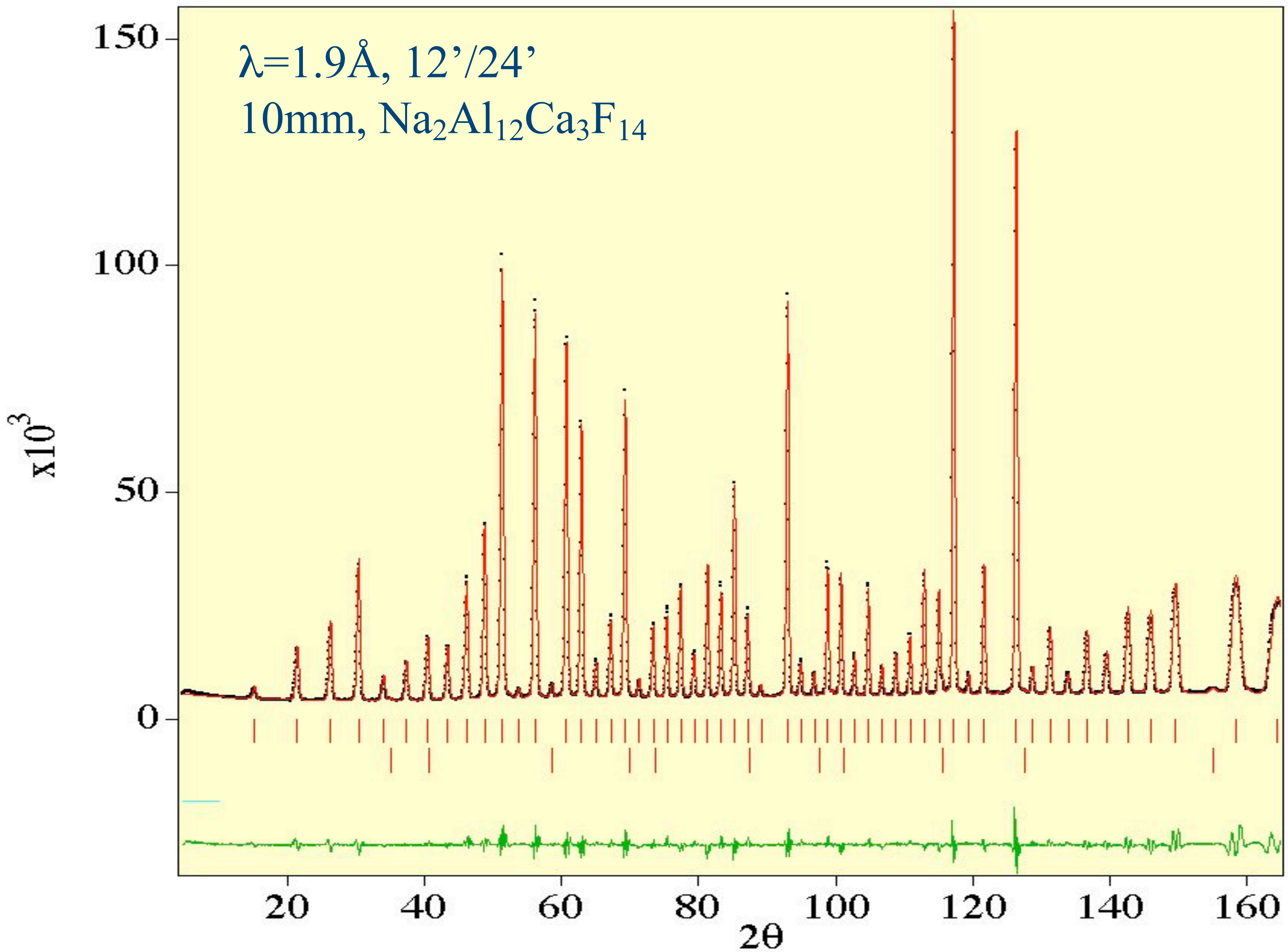
$\alpha_2$

mosaic spread of the
monochromator 15’

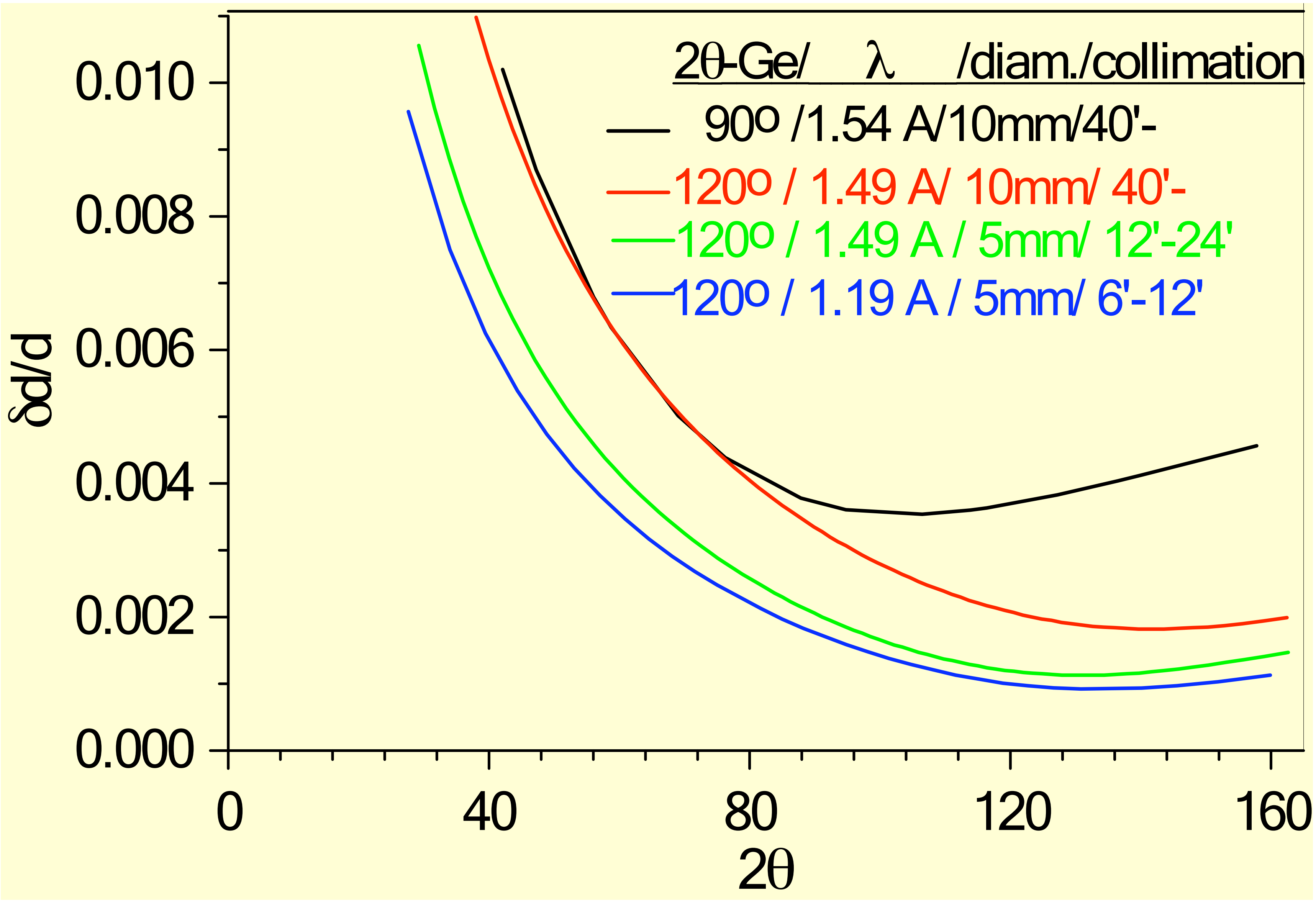
$\alpha_3$

slit system for
monochromatic beam
and
sample diameter

# Resolution calibration



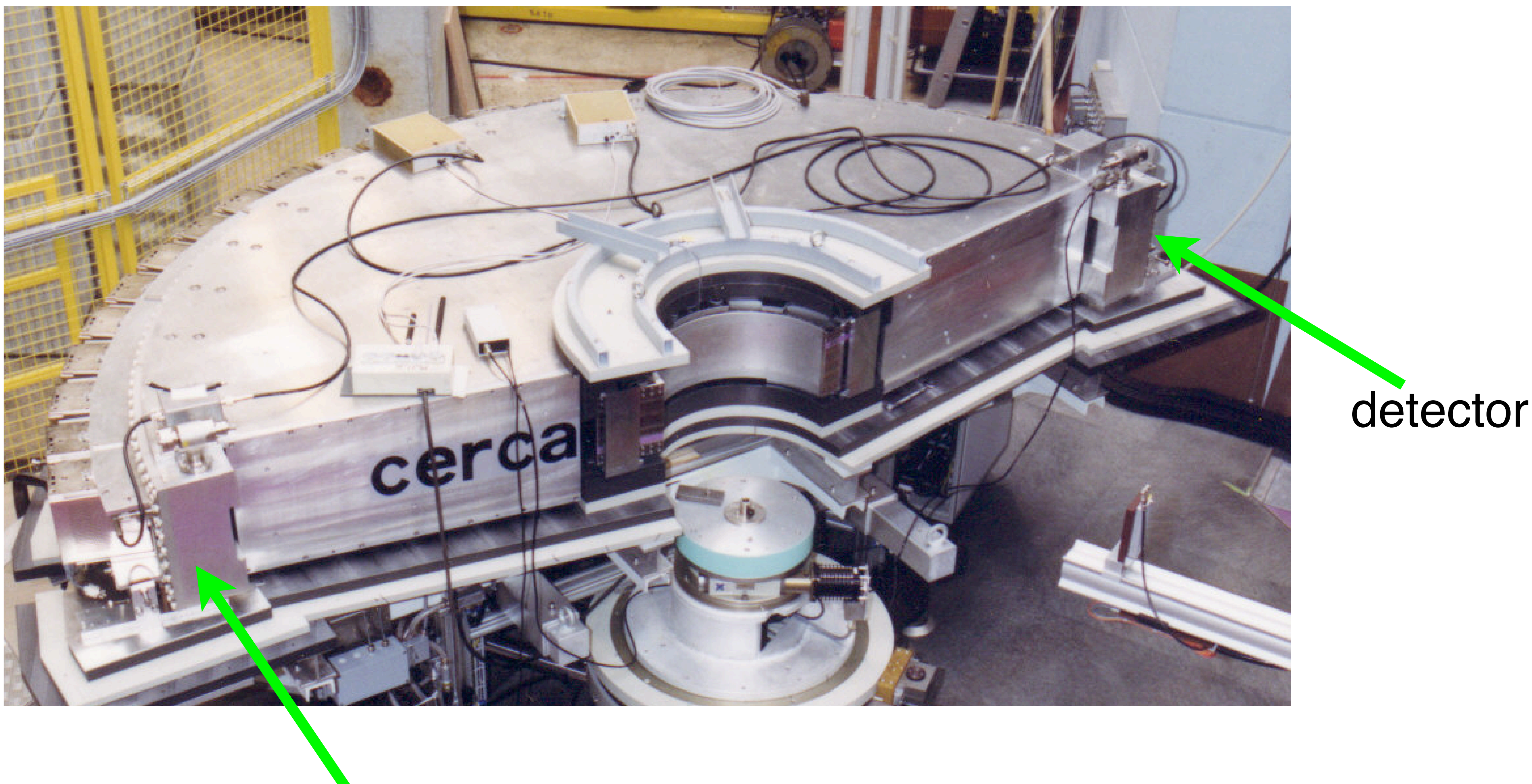
# Resolution and intensity (2)




Comparison of resolution functions for different primary-secondary collimations. Typical modes are HI:40'-, MR:12'-24', HR:6'-12'. Counting rates are decreased by a factor of ~3 and ~(8-10) for MR and HR, respectively.

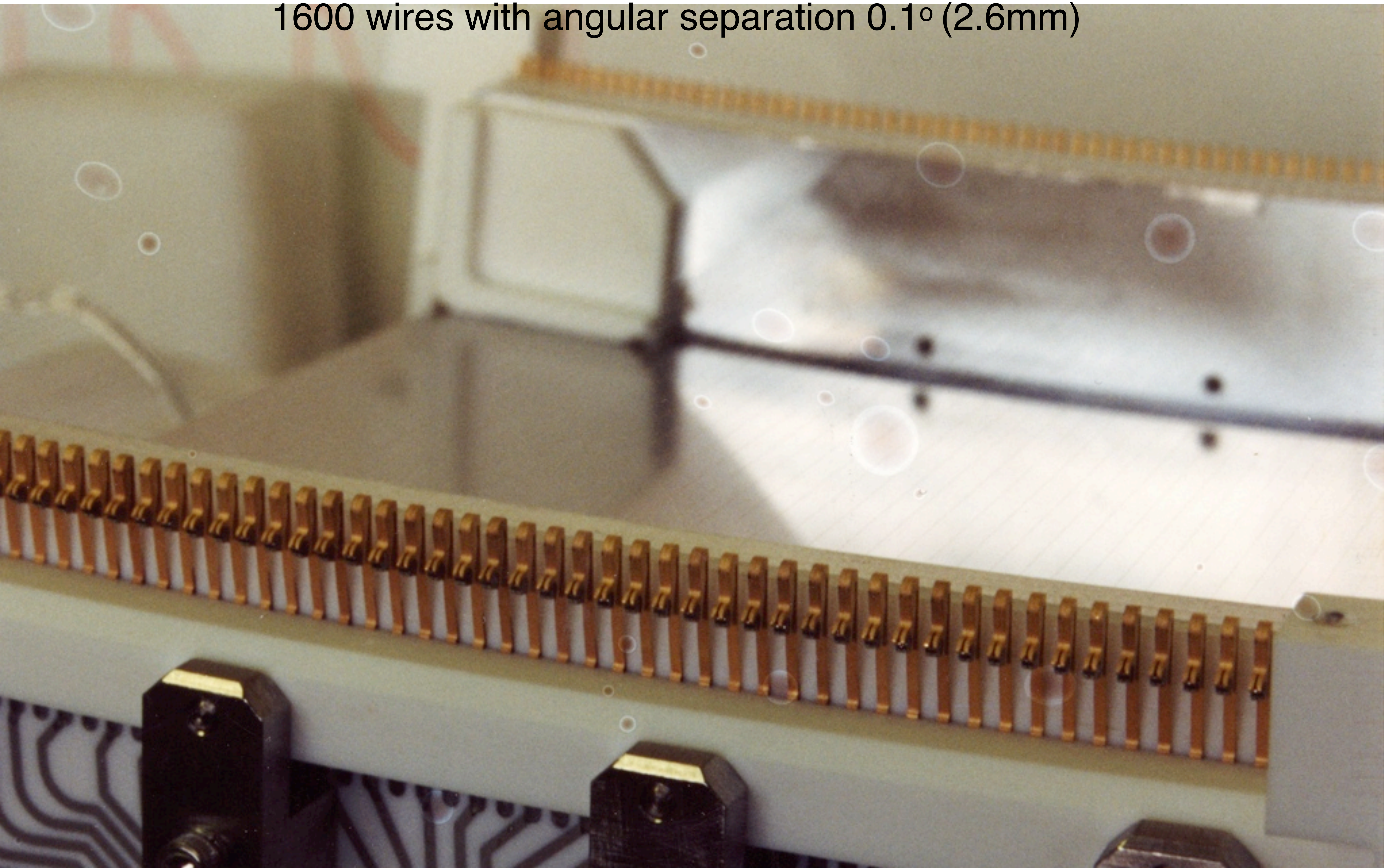
# Detector

- $^3He$ (3.6 bar) + $CF_4$ (1.1 bar), effective detection length 3.5 cm, 15 cm hight
- Volume 100L, Voltage -6.7kV
- Efficiency 80% @ 1.5 Å
- 1600 wires with angular separation 0.1° (2.6 mm), 1500 mm to sample

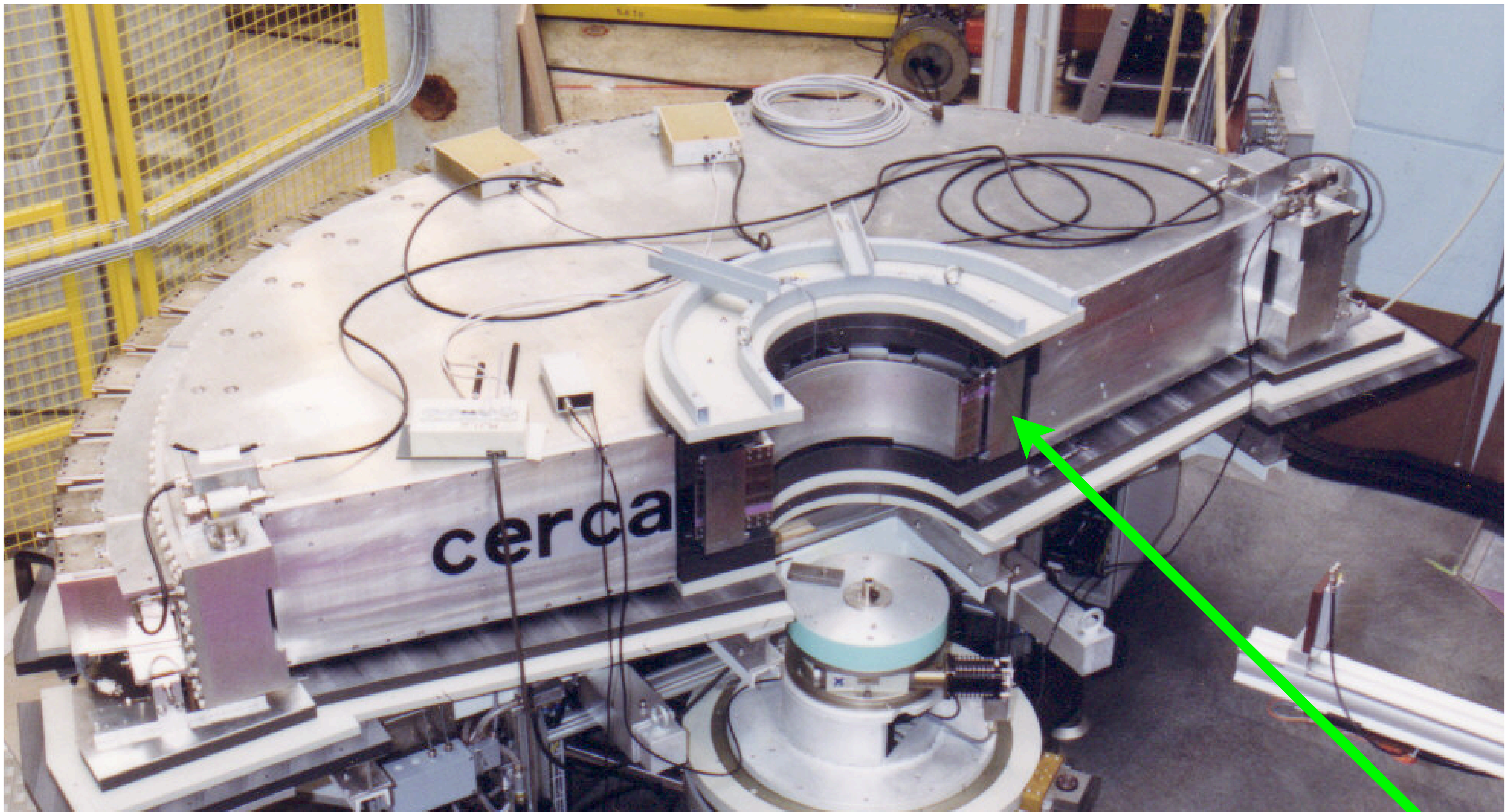


# Detector chamber. 1600 wires

1600 wires with angular separation 0.1° (2.6mm)

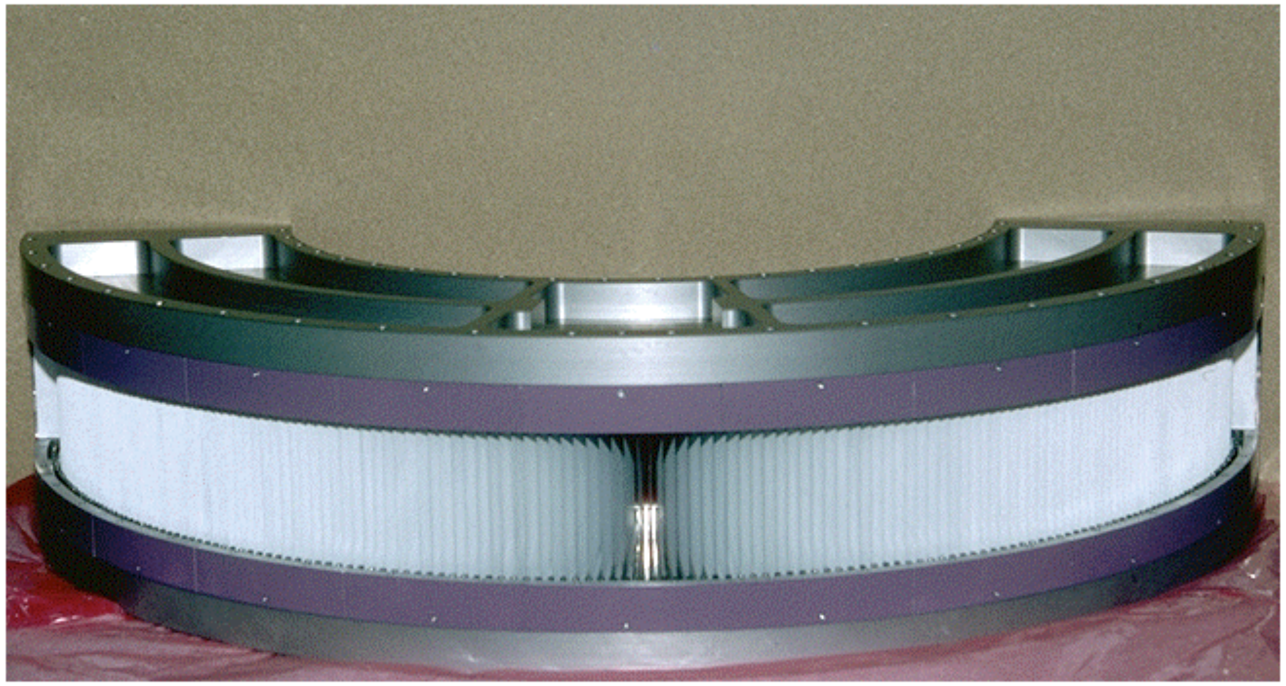


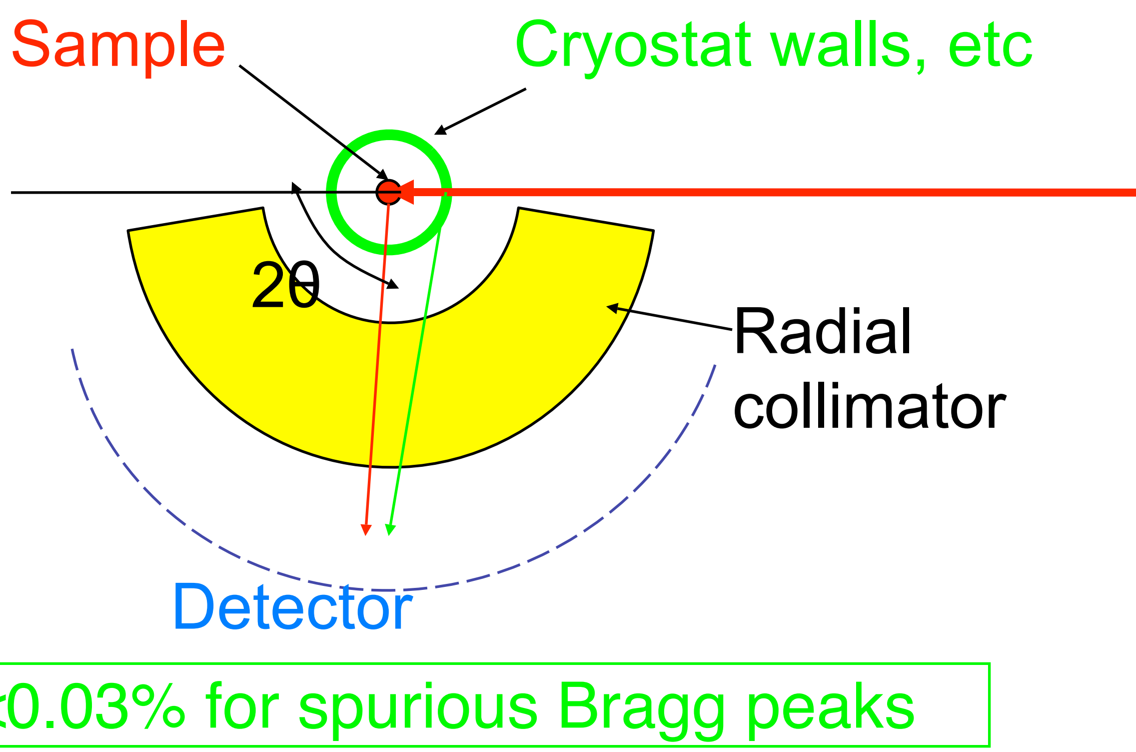
# Oscillating radial collimator to avoid scattering from sample environment.




radial
collimator

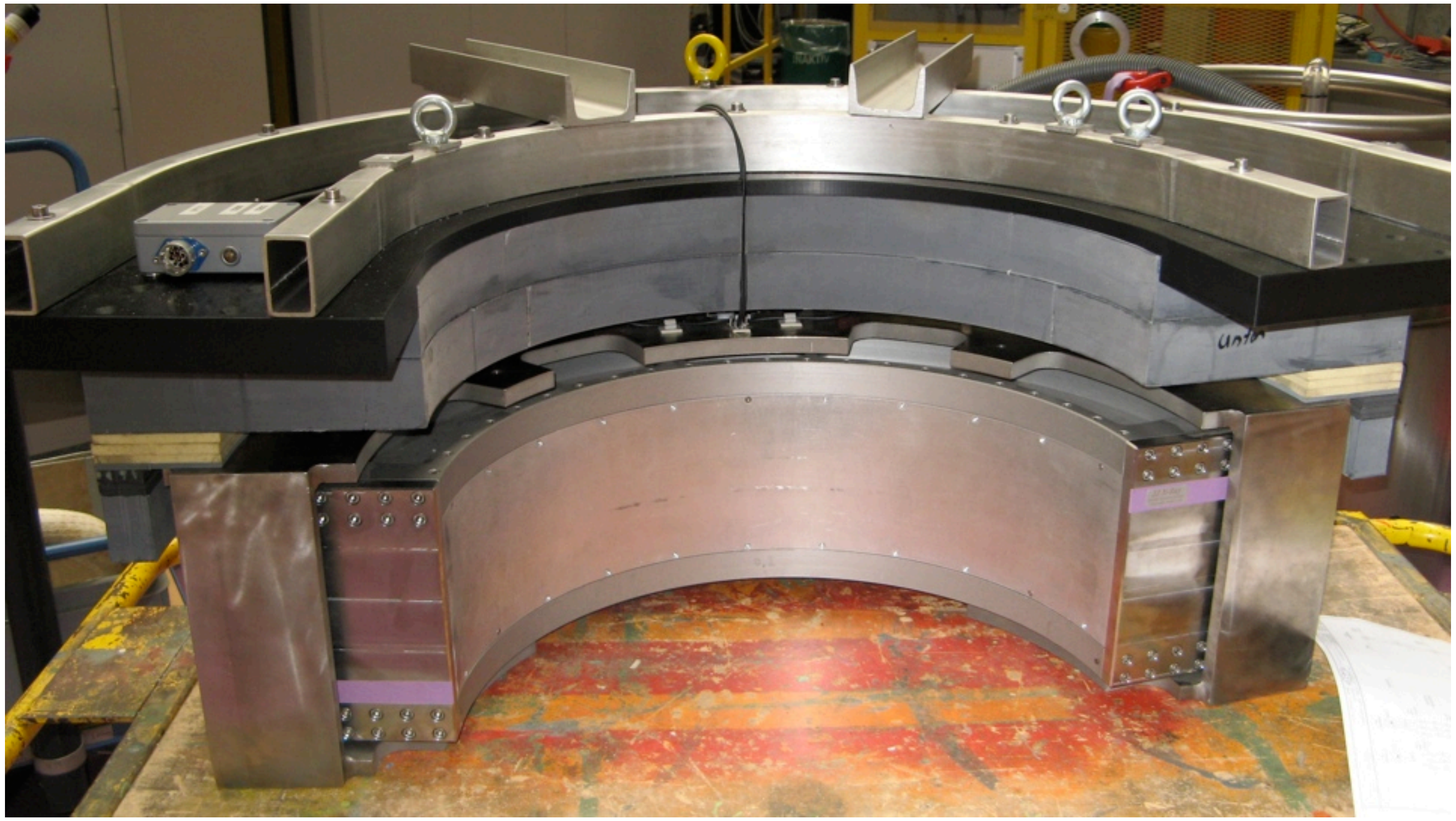
# HRPT radial collimators






Radial collimator with the shielding.

There are two radial collimators with 14mm and 28mm full width full maximum triangular transmission function.



# Samples, T, P, H and other equipment

- **standard sample container: 6-10 mm dia x 50 mm (<4cm$^3$)**
- due to low background small samples can be measured (30 mm$^3$)
- zero matrix high pressure cells:
  - clamp cells for 9 and 15 kbar
  - Paris Edinburgh cell 100 kbar
- standard LNS sample environment:
  - Temperature = 50 mK—1800K,
  - Magnetic field H = 4 T (vertical)
- Sample changers 4-8 samples, T=1.5-300 K

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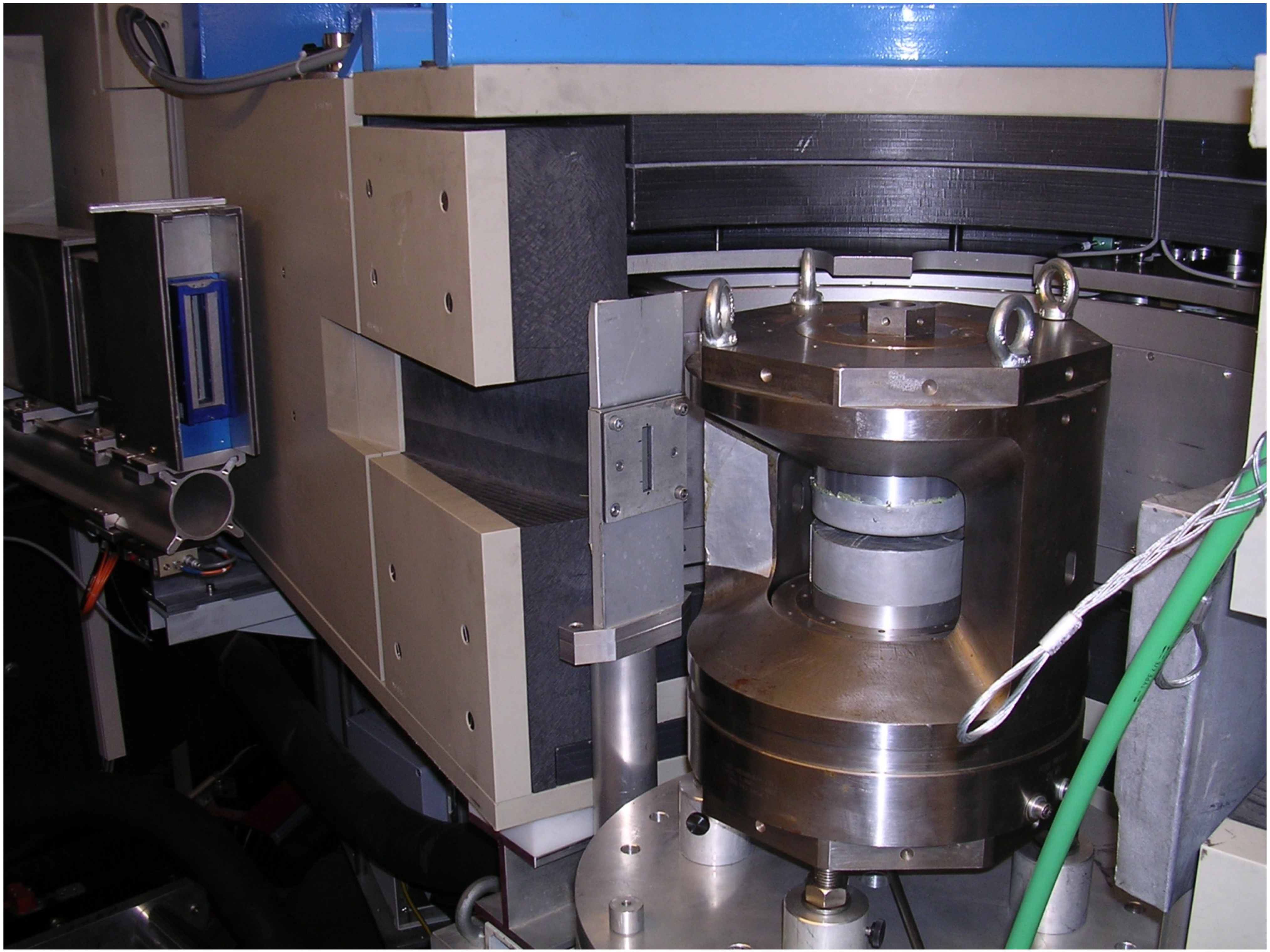
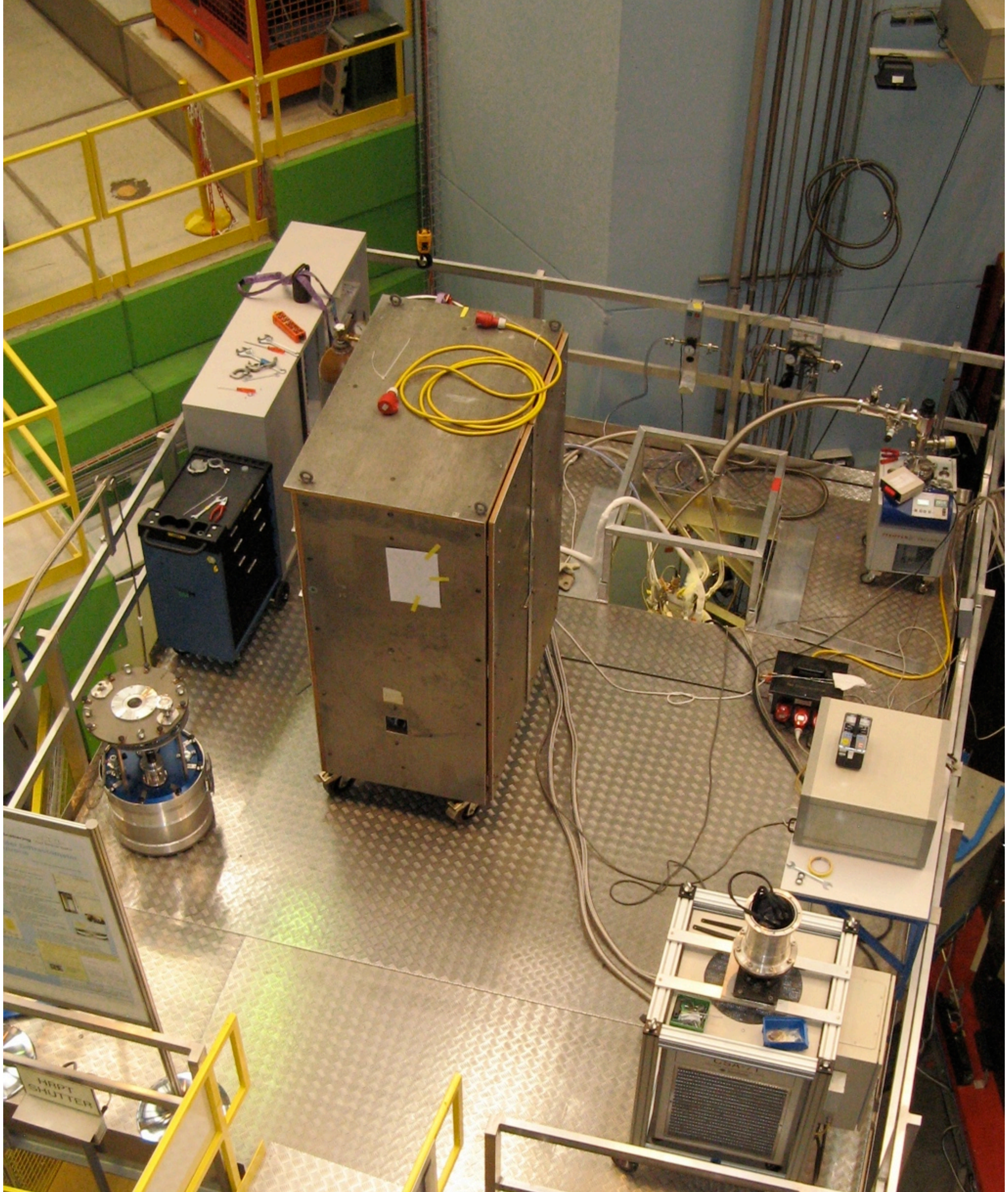
# clamp cells for 9 and 15 kbar

# Paris Edinburgh cell 100 kbar





PSI information

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  - Paris Edinburgh cell 100 kbar
- **standard LNS sample environment:**
  - **Temperature = 50 mK—1800K,**
  - **Magnetic field H = 4 T (vertical)**
  - **Automatic He, $N_2$ refilling systems**
- Sample changers 4-8 samples, T=1.5-300 K

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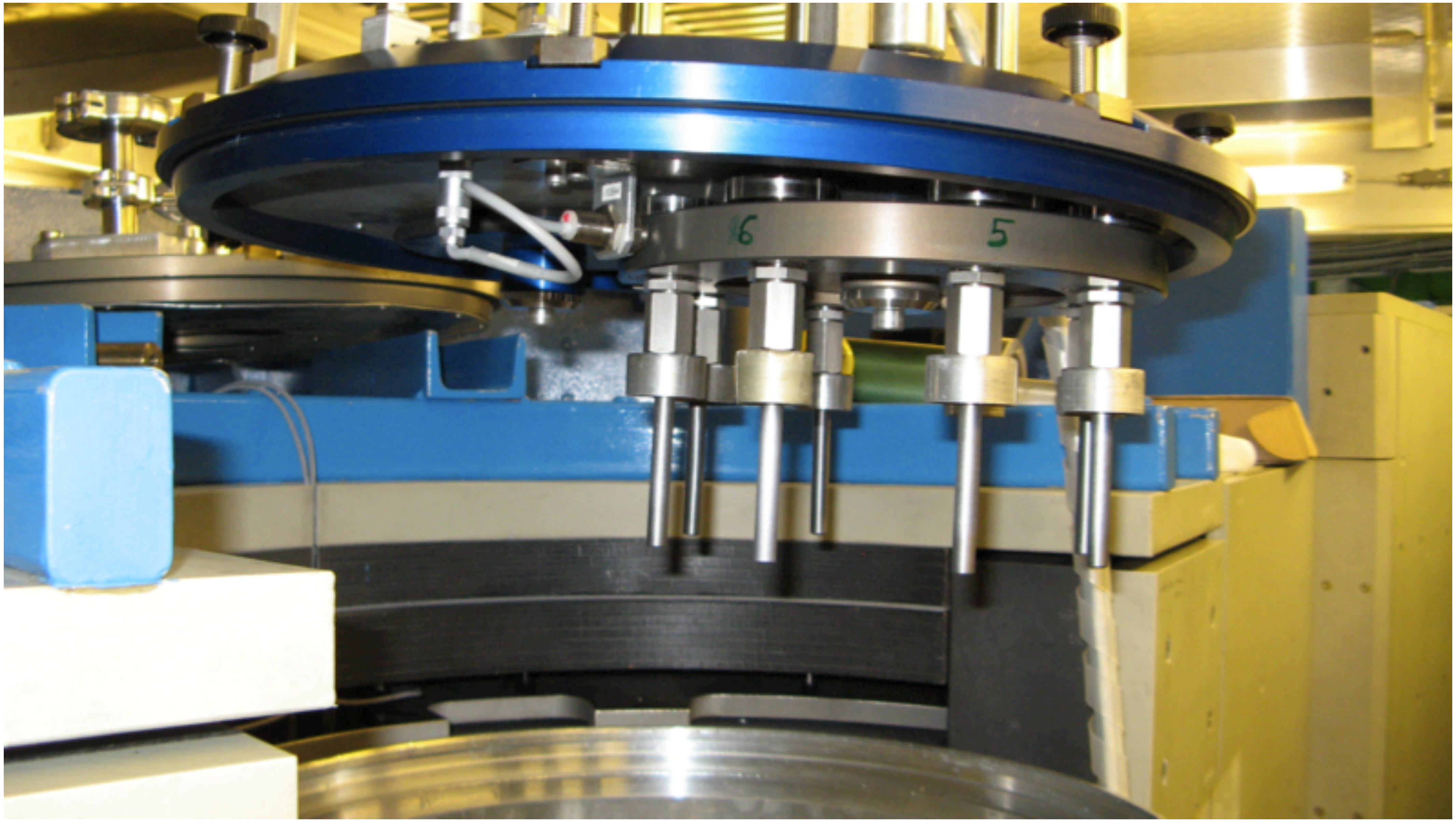
  - Automatic He, $N_2$ refilling systems
- **Sample changers 4-8 samples, T=1.5-300 K**

# HRPT room temperature 8-sample changer

- Eight samples mounted on a caroussel-type changer, few seconds to bring the next one into the measurement position;

- Independent sample rotation mechanism – for reducing the preferred orientation aberrations.




*Fully loaded with 8 samples, the sample changer is ready to be installed in-place on the HRPT sample table.*

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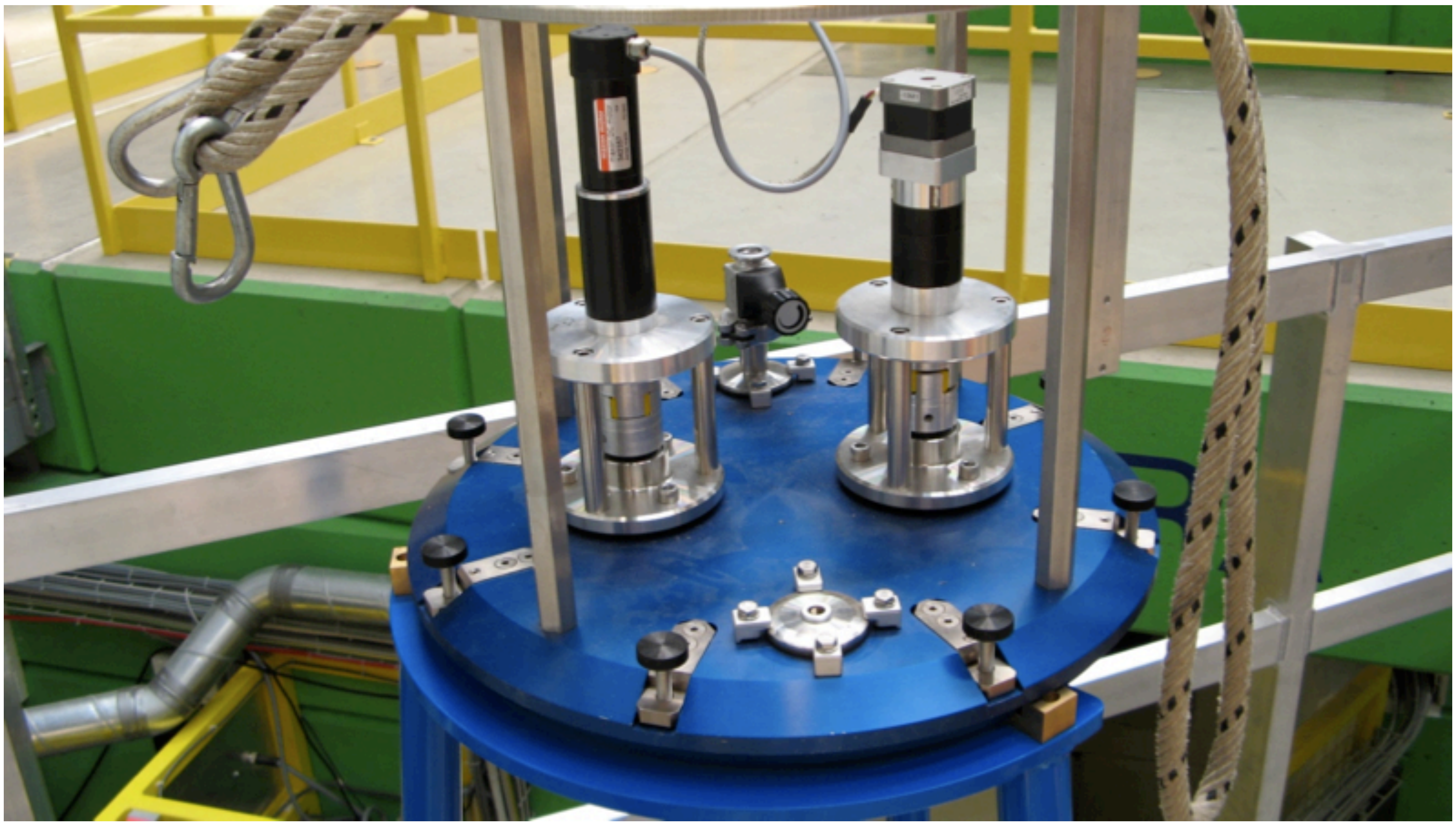
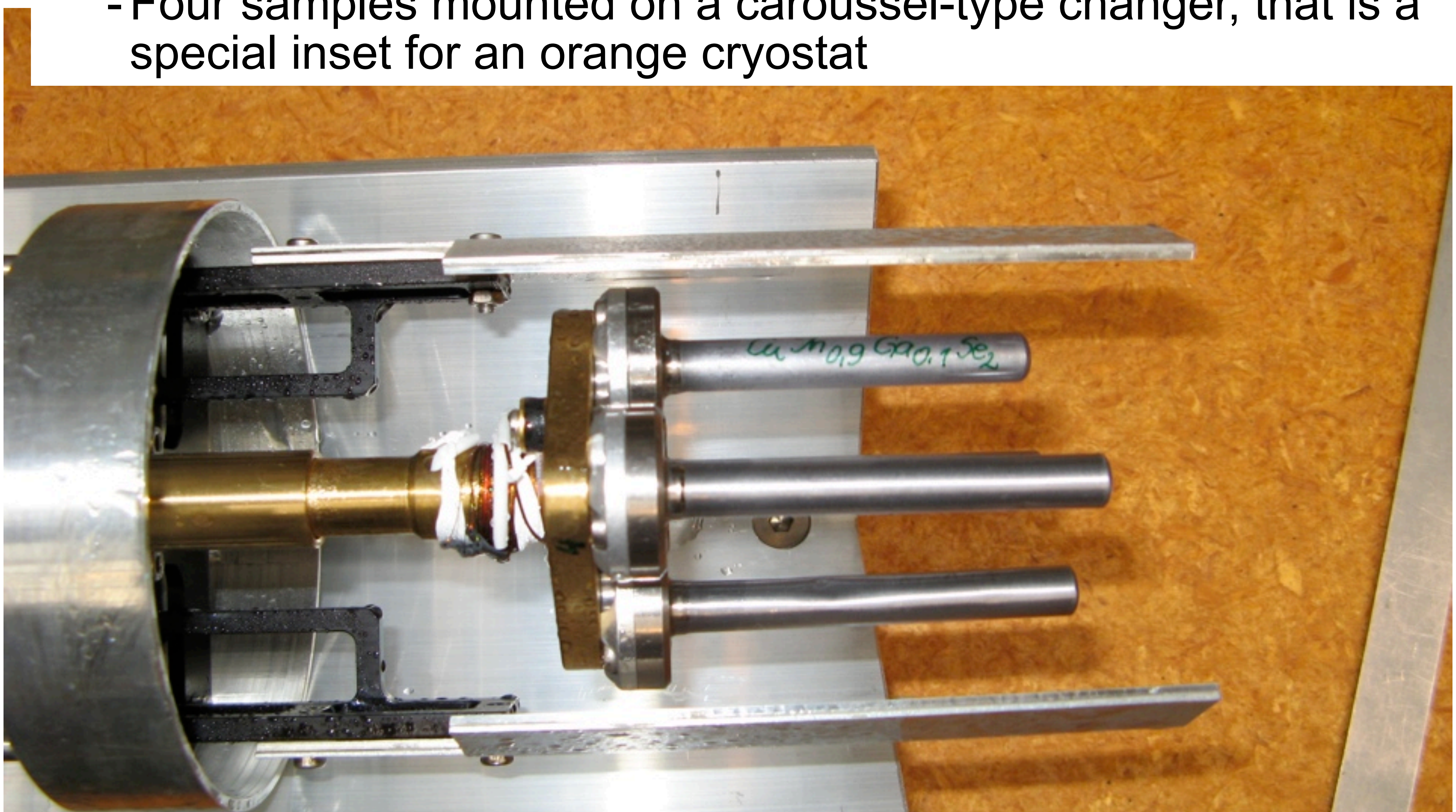


*Fully loaded with 8 samples, the sample changer is ready to be installed in-place on the HRPT sample table.*

# HRPT low temperature 4-sample changer

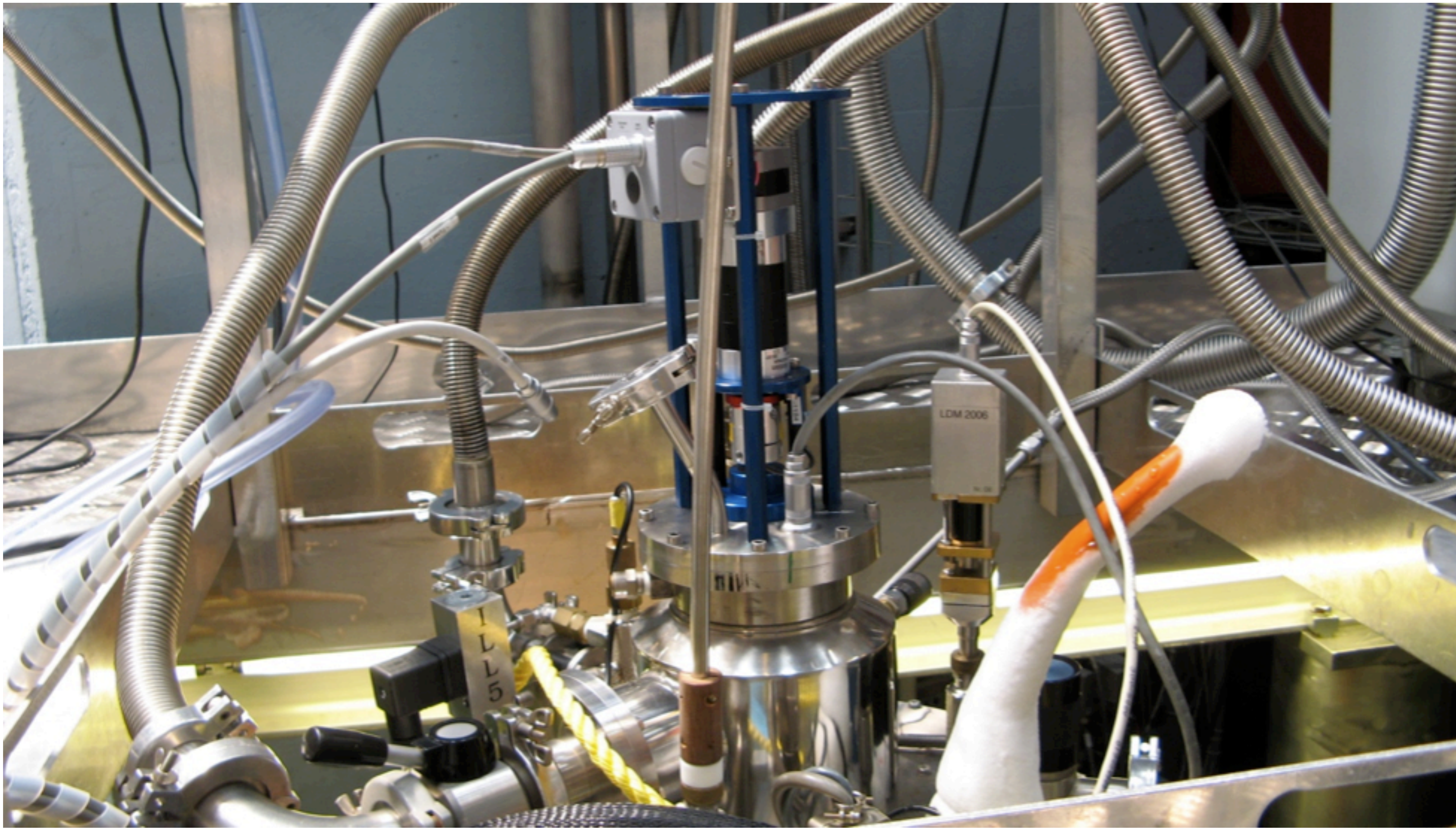
A device for routine powder diffraction measurements at temperatures between 1.5K -300K.

- All samples have the same temperature, i.e. time for temperature change is saved;
- Four samples mounted on a caroussel-type changer, that is a special inset for an orange cryostat



# HRPT low temperature 4-sample changer

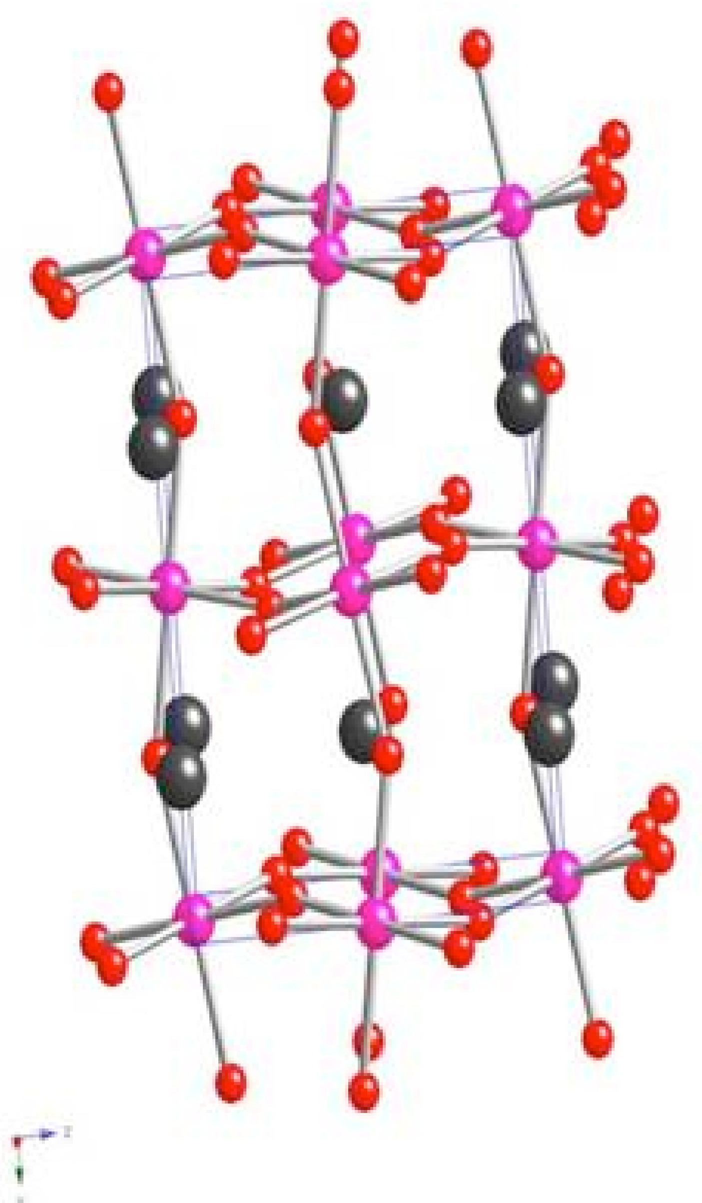
# HRPT low temperature 4-sample changer



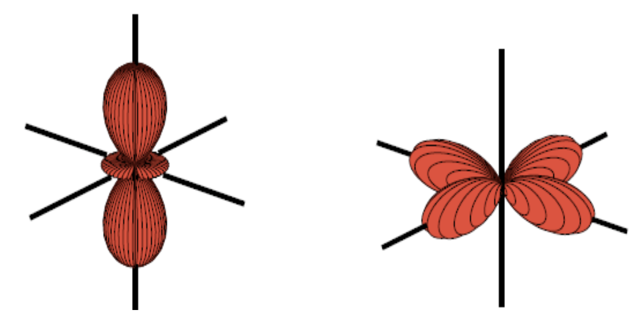
# Examples of HRPT applications

# Mn-O bond lengths

$(La_{1-y}Pr_y)_{0.7}Ca_{0.3}(Mn^{3+})_{0.7}\,(Mn^{4+})_{0.3}O_3$



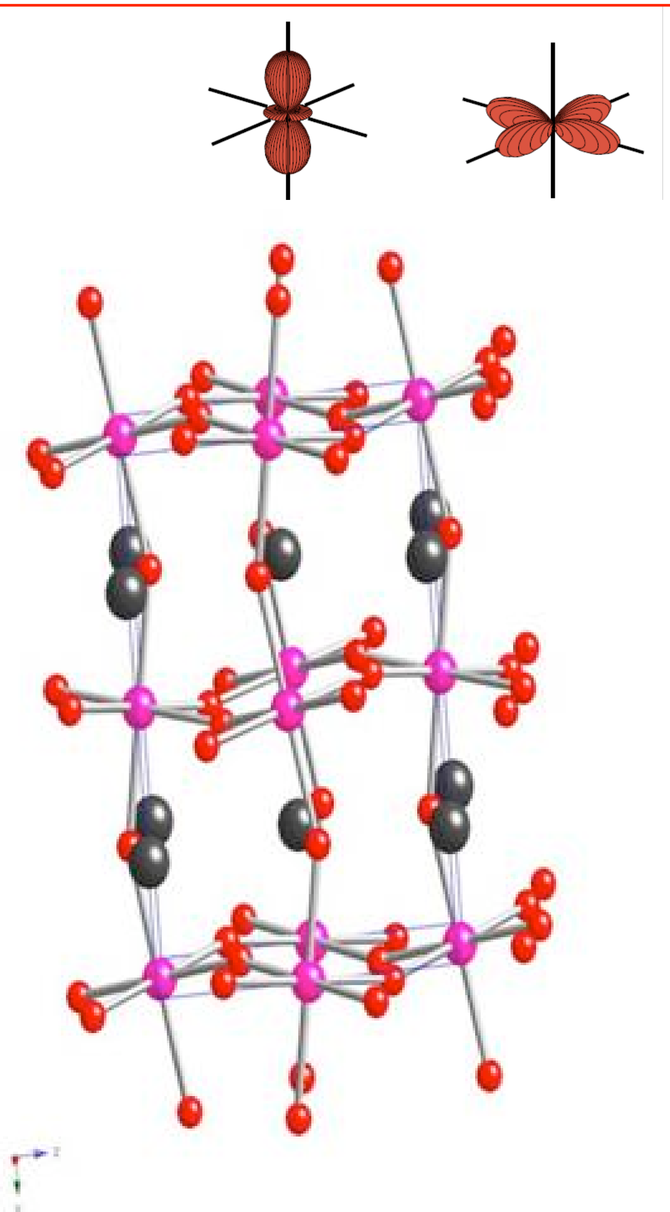
$$|\theta\rangle = \cos\frac{\theta}{2}|3z^2 - r^2\rangle + \sin\frac{\theta}{2}|x^2 - y^2\rangle$$

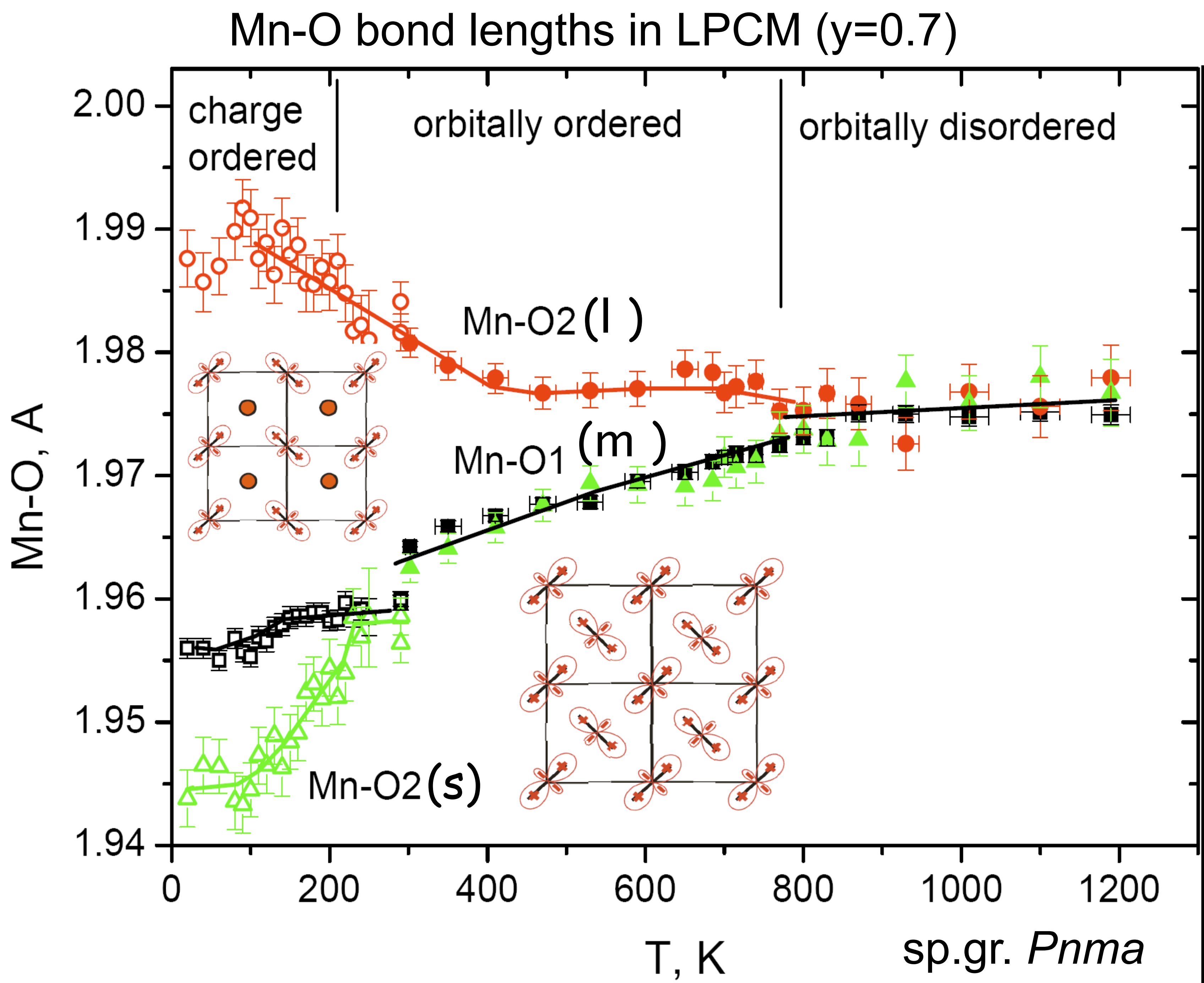


# Orbital and charge ordering OO/CO

$(La_{1-y}Pr_y)_{0.7}Ca_{0.3}(Mn^{3+})_{0.7}(Mn^{4+})_{0.3}O_3$

$$|\theta\rangle = \cos\frac{\theta}{2}|3z^2 - r^2\rangle + \sin\frac{\theta}{2}|x^2 - y^2\rangle$$

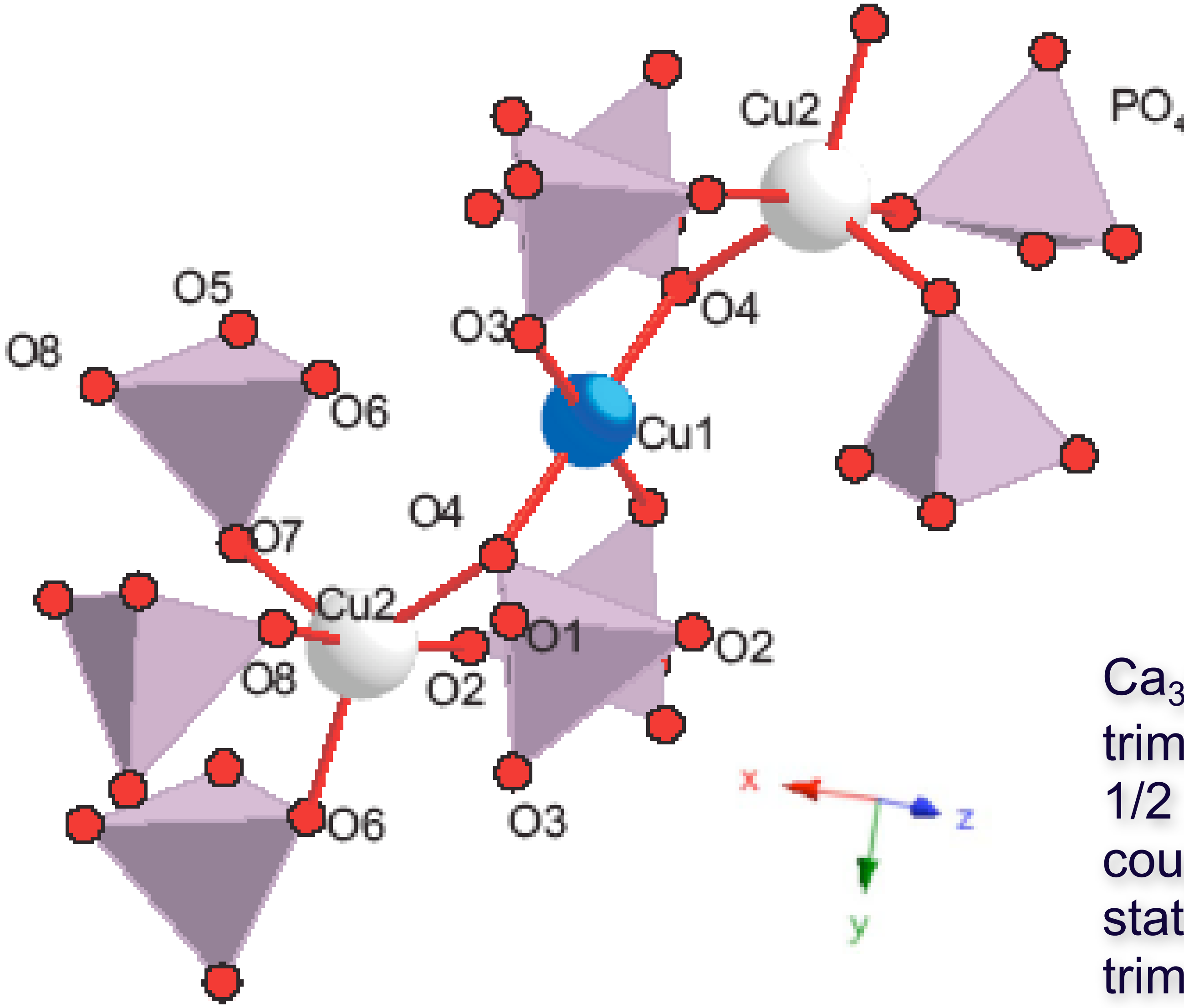





PSI information day'09

# Where are Ni ions in the trimer?

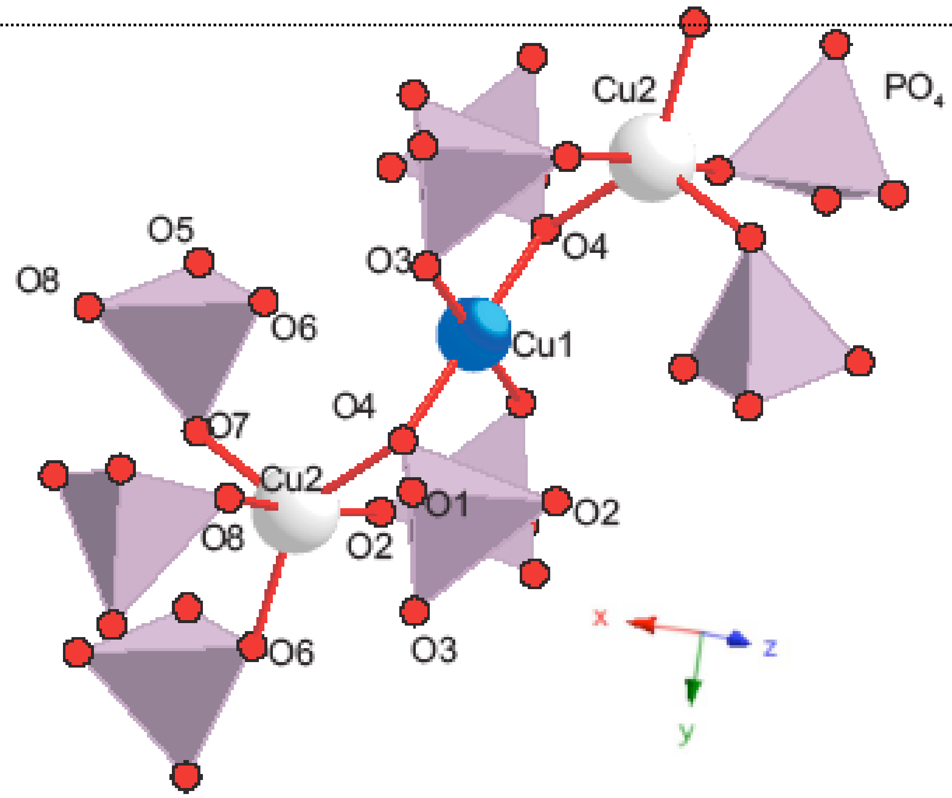
## $Ca_3Cu_{3-x}Ni_x(PO_4)_4$ (x=0,1,2)

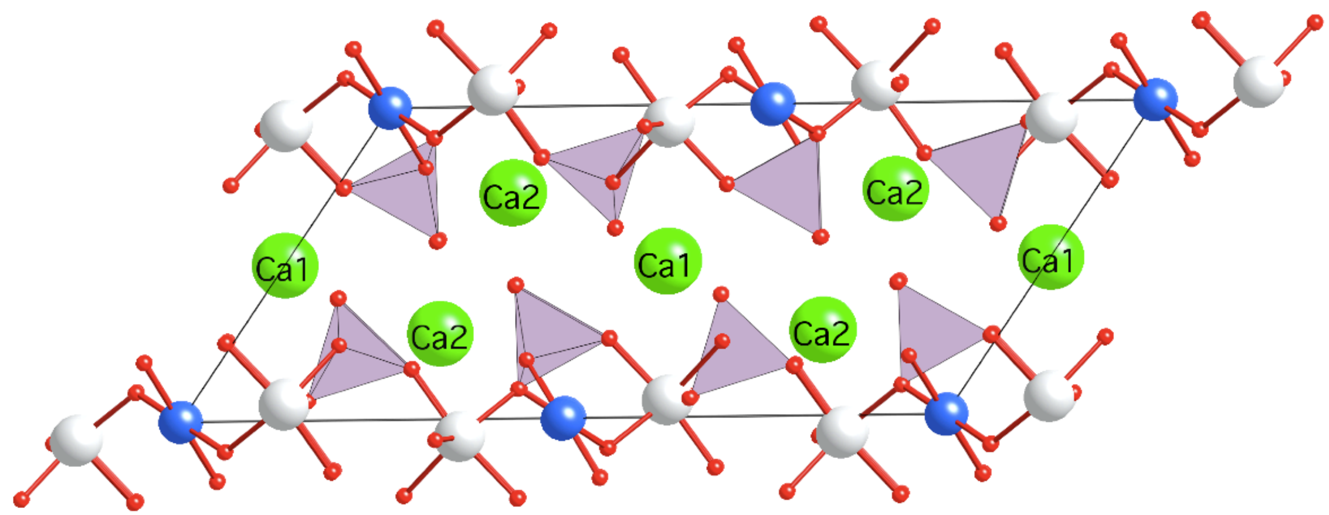


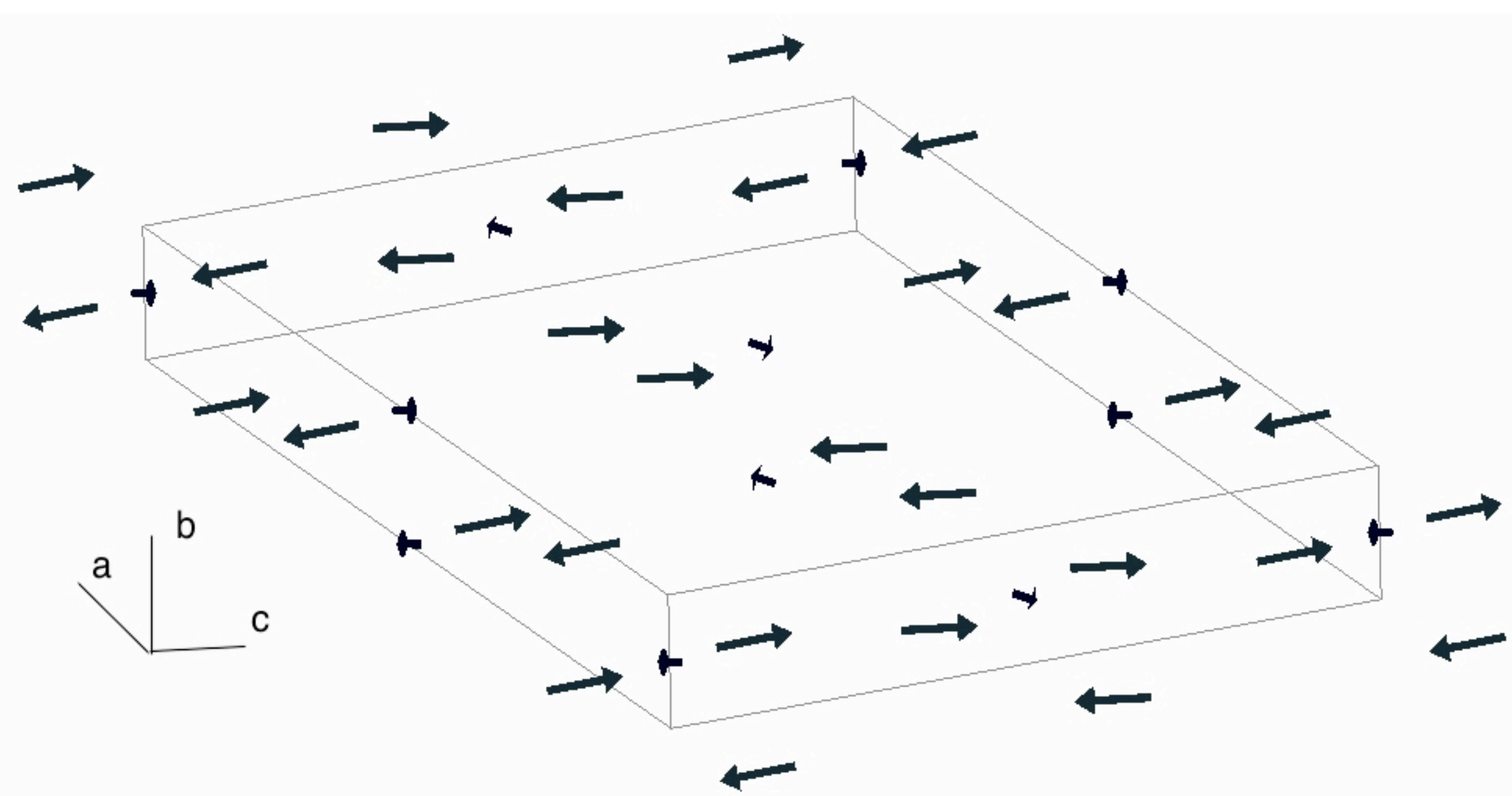

$Ca_3Cu_3(PO_4)_4$ is a novel quantum spin trimer system in which the three $Cu^{2+}$(S = 1/2 ) spins are antiferromagnetically coupled giving rise to a doublet ground state. By substituting a $Cu^{2+}$ spin in the trimer by $Ni^{2+}$ (S = 1) a singlet ground state could be in principle realized offering the observation of the Bose-Einstein condensation in a quantum spin trimer system.

# Crystal and magnetic structures and magnetic excitations spin-trimer system $Ca_3Cu_{3-x}Ni_x(PO_4)_4$ (x=0,1,2)

Where are Ni ions in the trimer?

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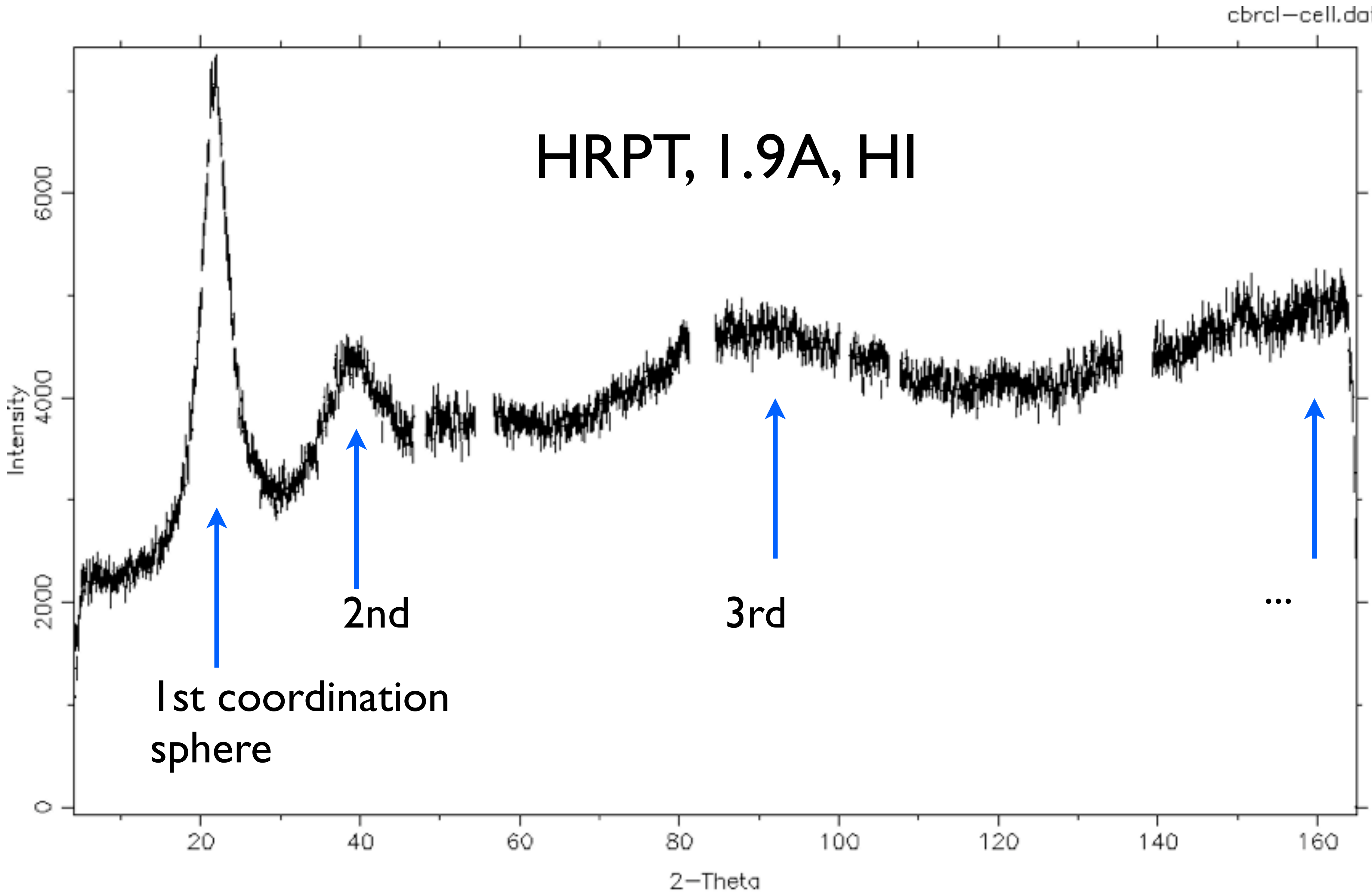
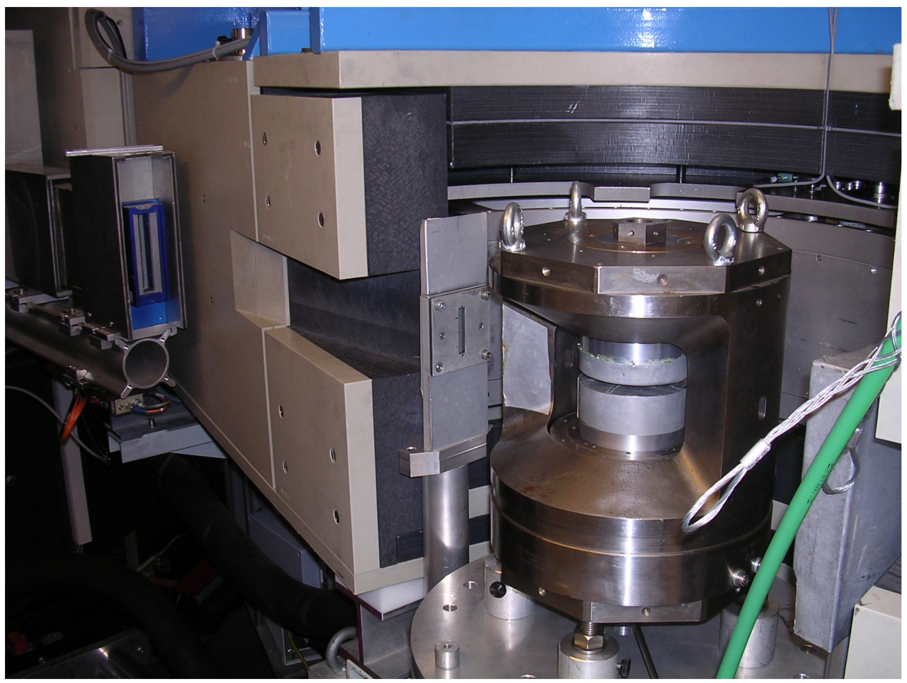
# $C(CrBr)_4$-liquid in gas pressure cell. T-P phase diagram




M.Barrio, et al (2009)

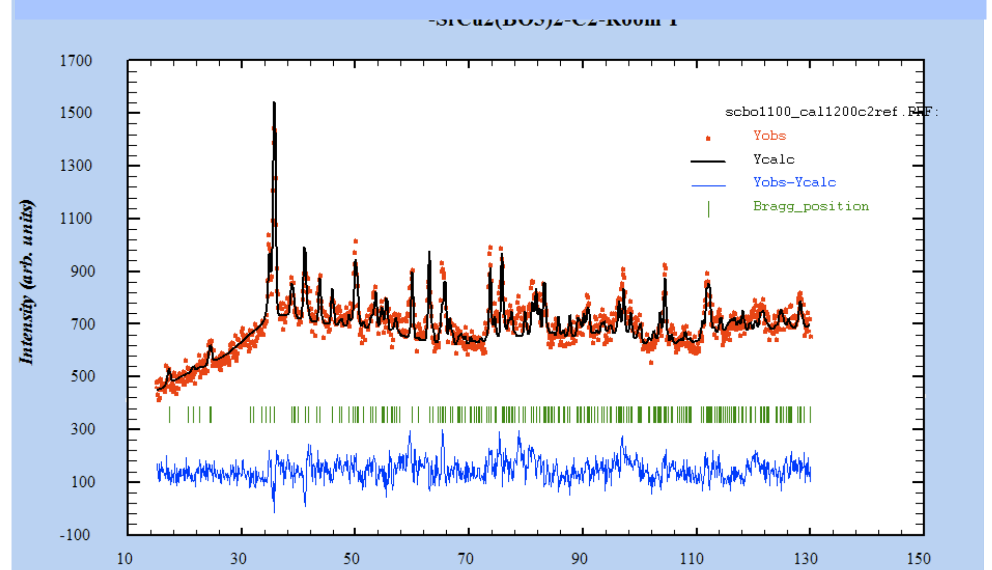
# High pressure structure transition in quantum dimer system $SrCu_2(BO_3)_2$

Anvil pressure cell installed at HRPT diffractometer

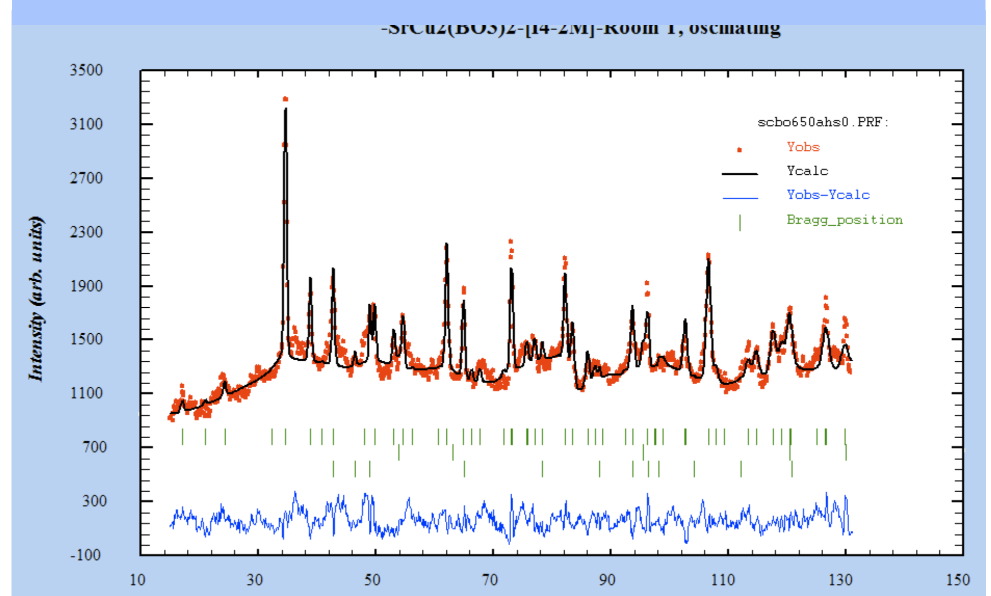


detector shielding + radial collimator + BN anvils + low noise ellectronics = excellent peak to background ratio

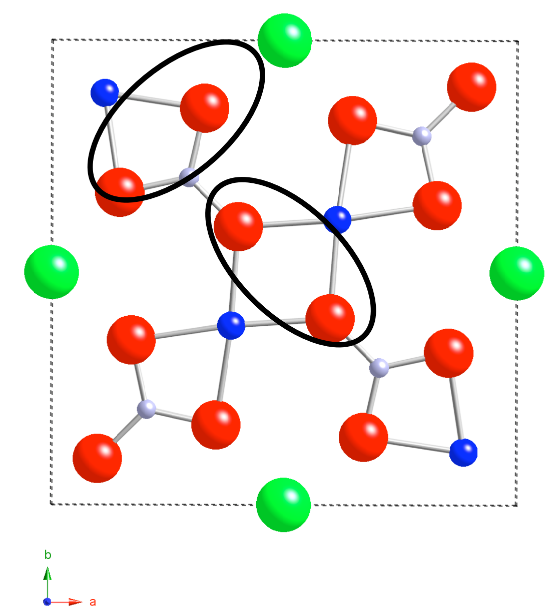
p=8GPa: monoclinic *C2*: the new structure solved from the HRPT data!




p=3.7GPa, known tetragonal *I-42m* structure




The S=1/2 moments of the $Cu^{2+}$ ions are arranged in a 2D lattice of strongly coupled dimers (J=85 K).

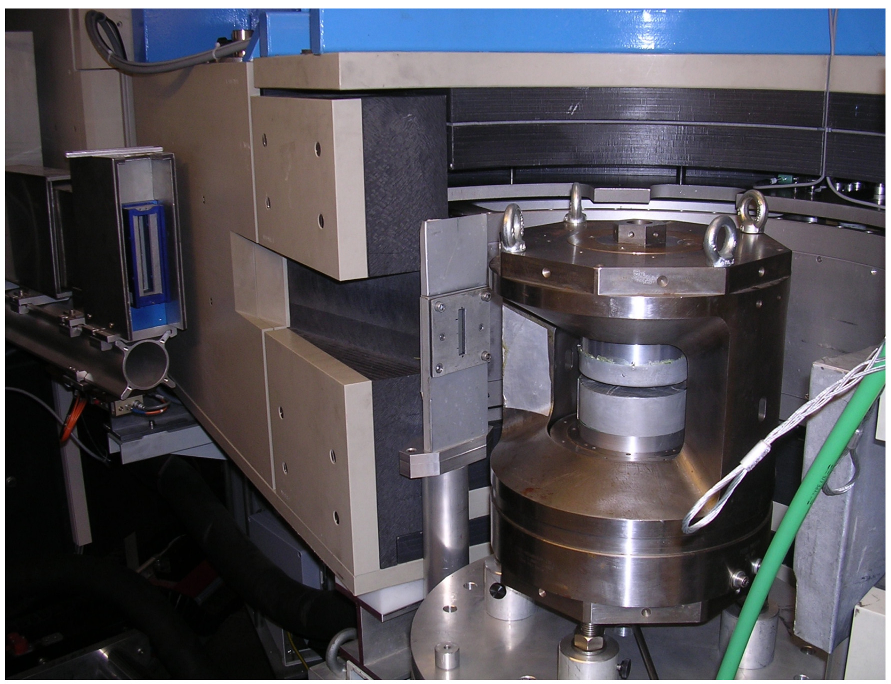


- The material is predicted to undergo a quantum phase transition by application of hydrostatic pressure.
- To fully understand the magnetic properties of the material the knowledge of the exchange paths as a function of pressure is mandatory.

*LNS, PSI:* V. Pomjakushin, Th. Strassle, K. Conder, E. Pomjakushina
*EPFL:* M. Zayed, H. Ronnow

# High pressure structure transition in quantum dimer system $SrCu_2(BO_3)_2$

Anvil pressure cell installed
HRPT diffractometer



detector shielding + radial collimator + BN anvils + low noise ellectronics = excellent peak to background ratio

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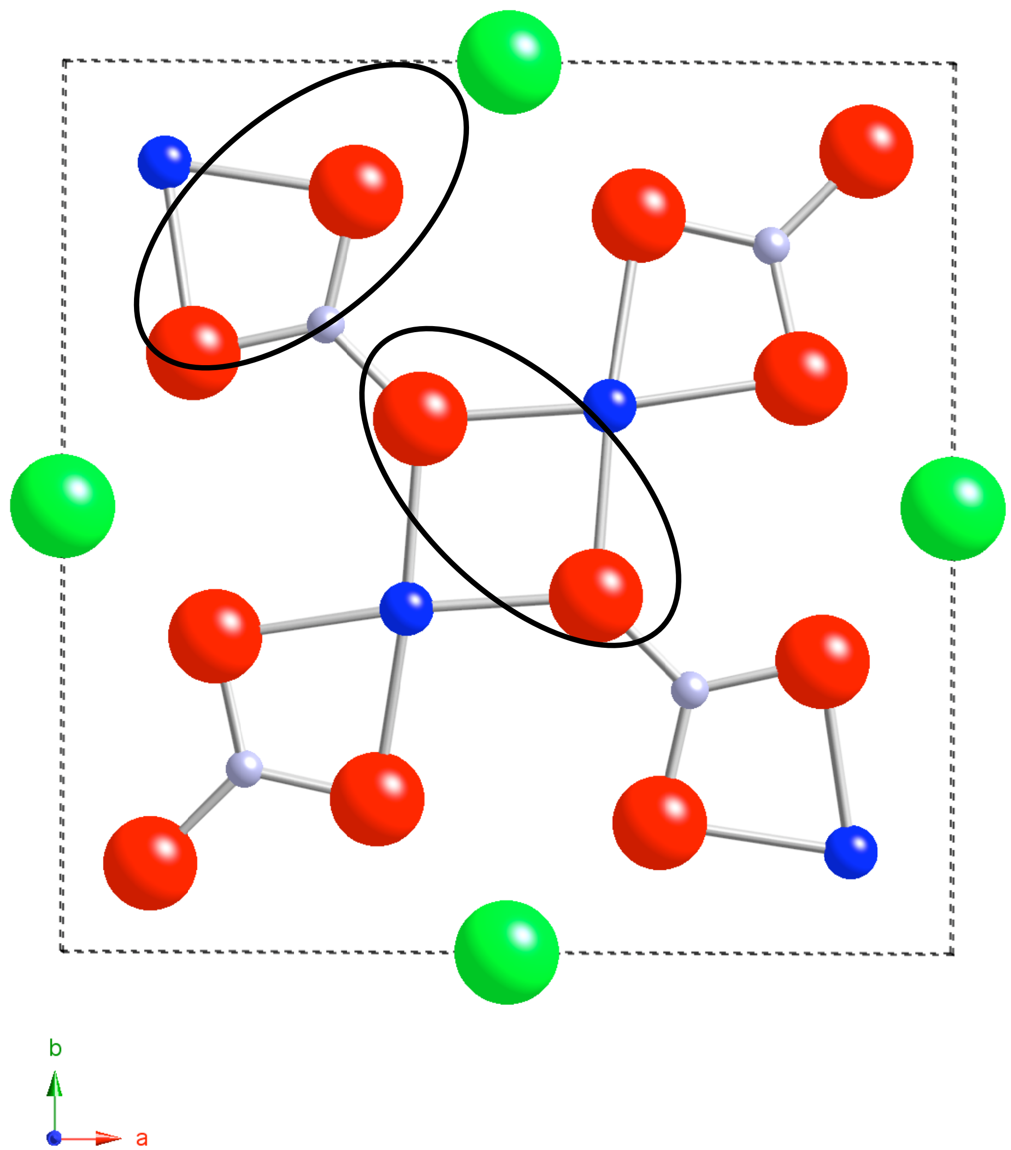


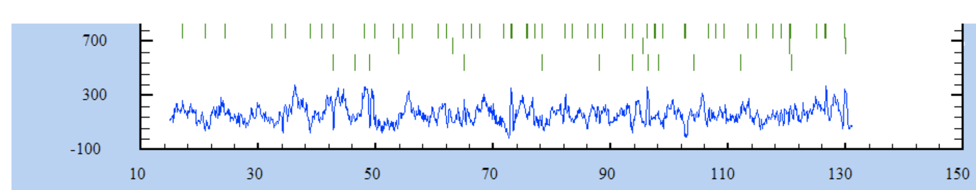


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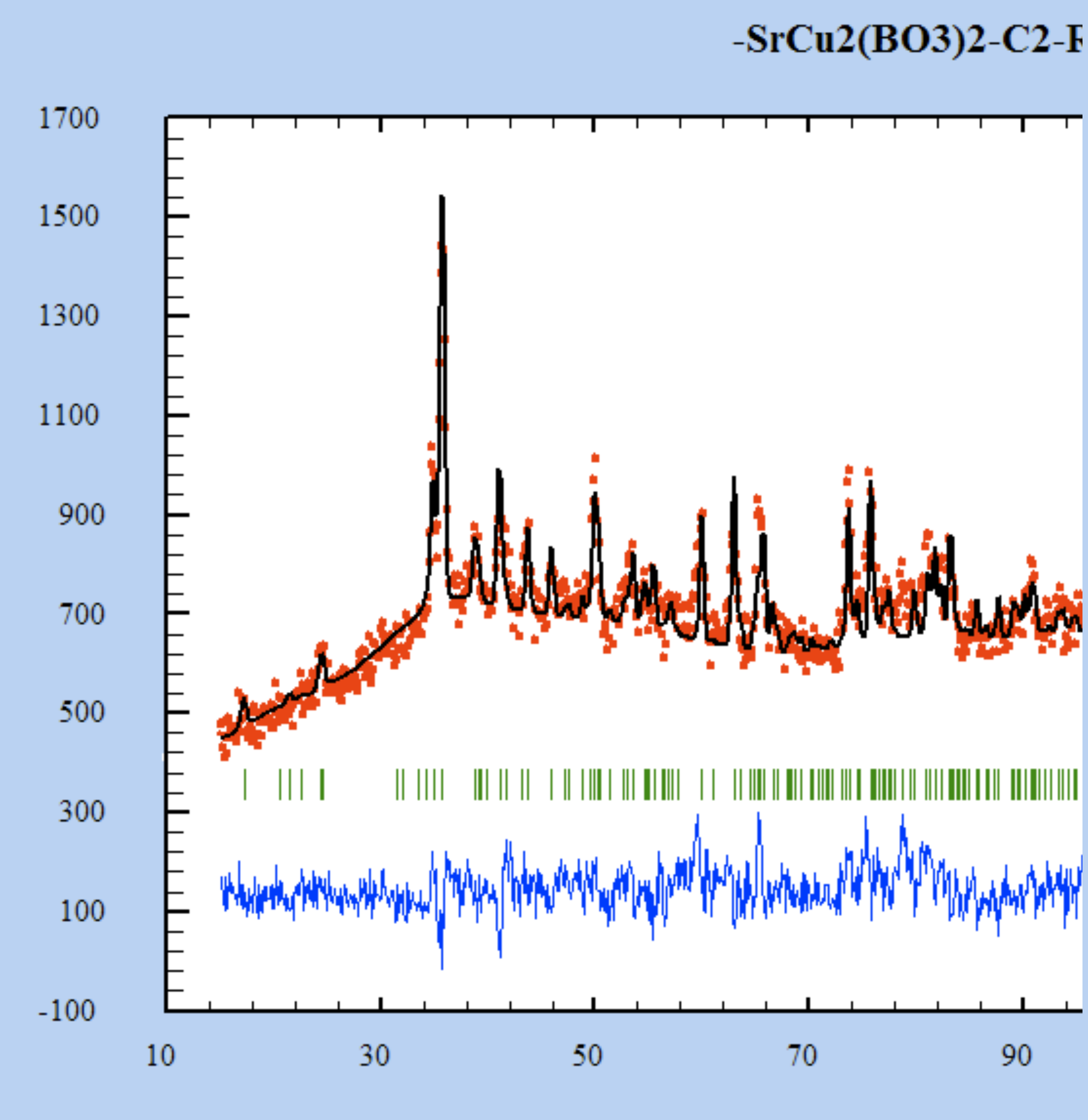
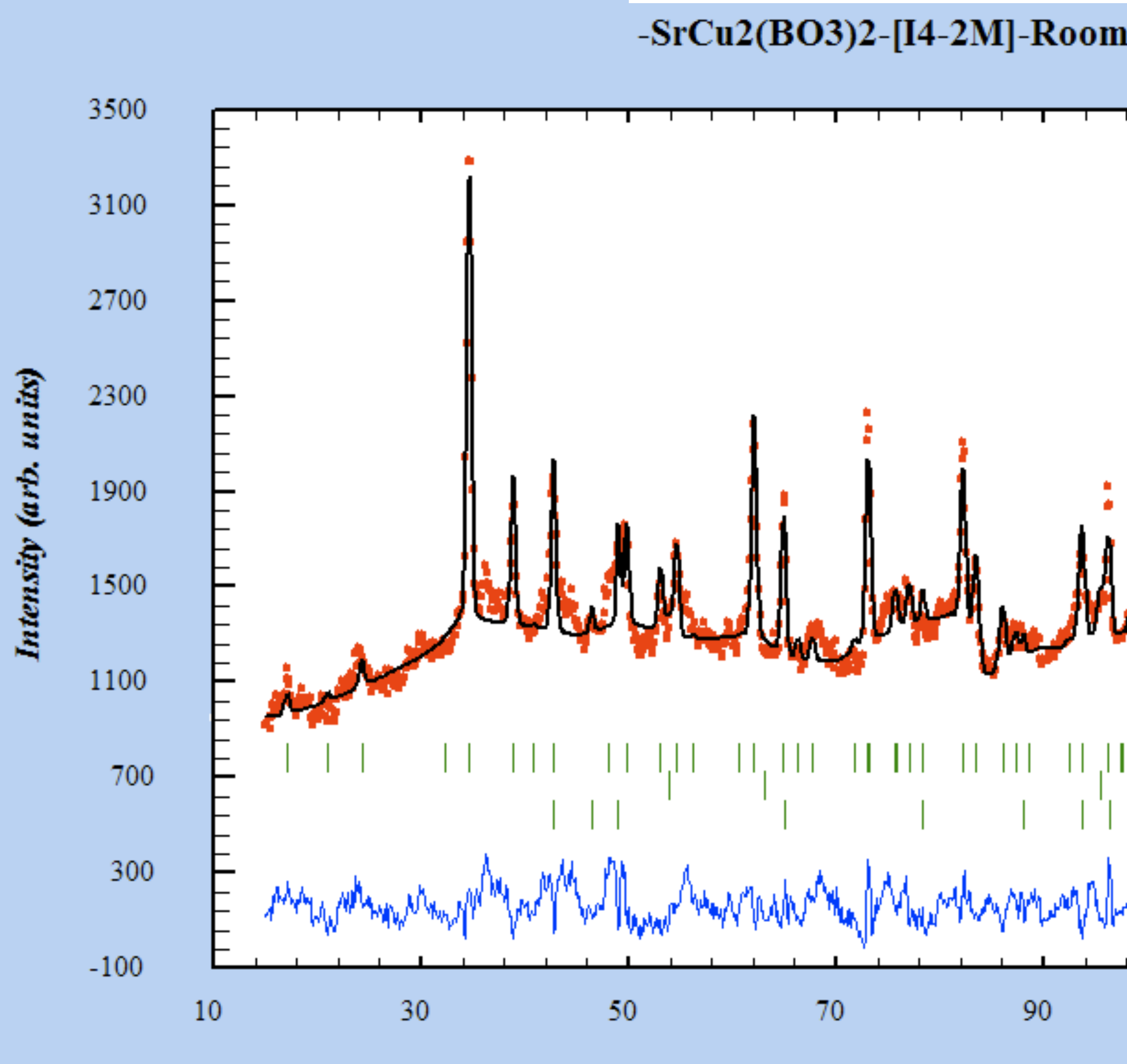
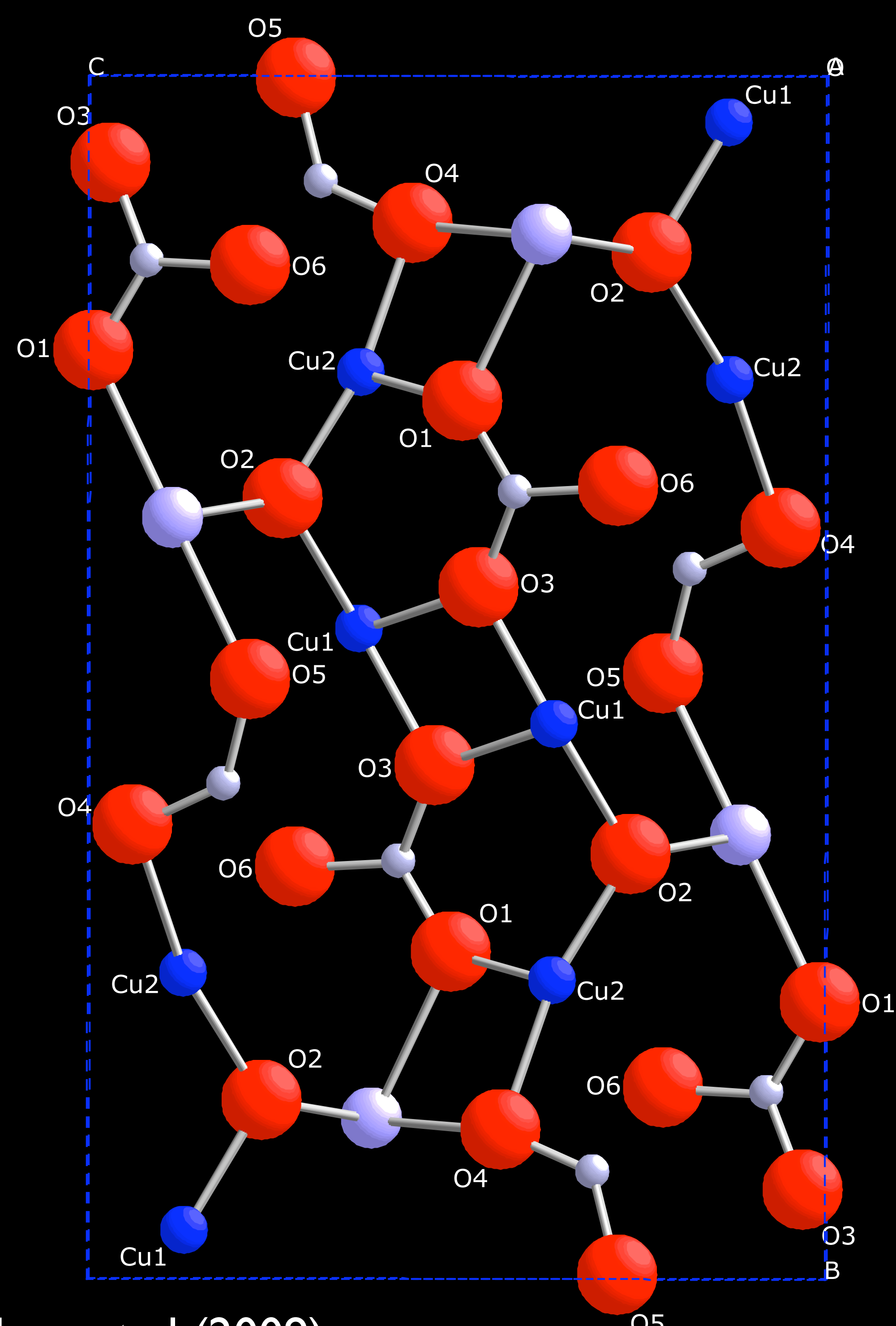
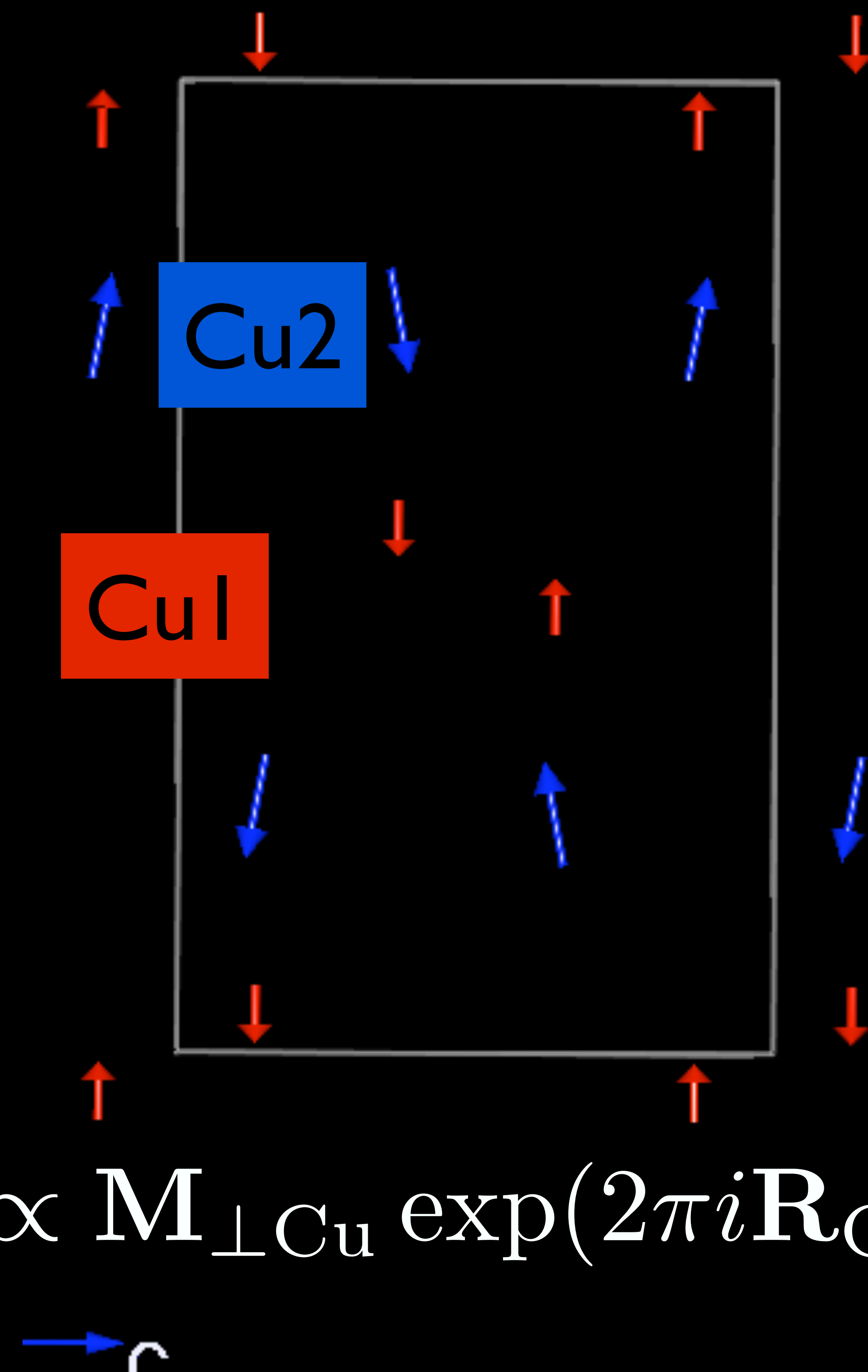


# Magnetic structure of $Cu_2CdB_2O_6$ exhibiting a quantum-mechanical magnetization plateau and classical antiferromagnetic long-range order



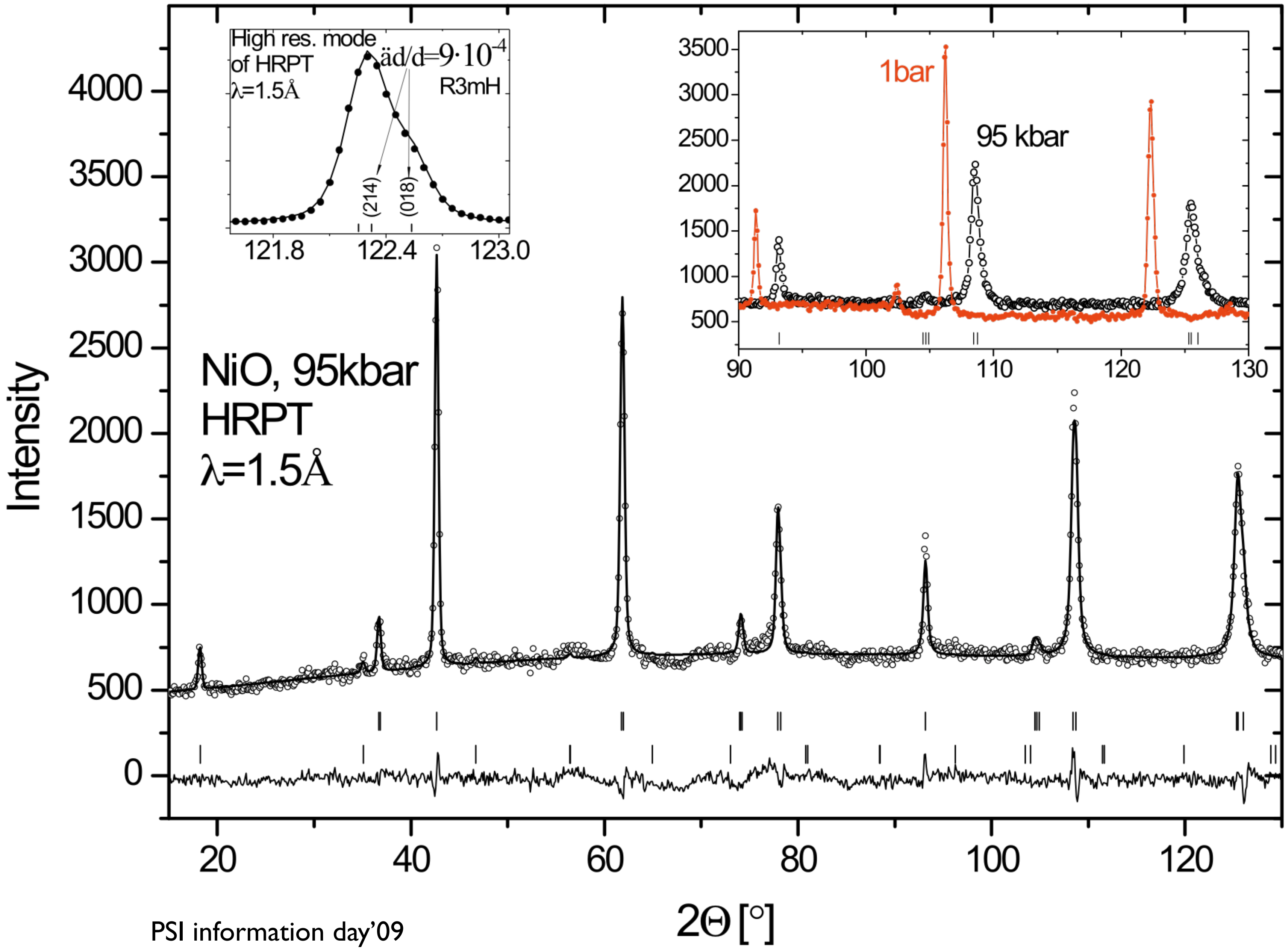

M.Hase, et al (2009)

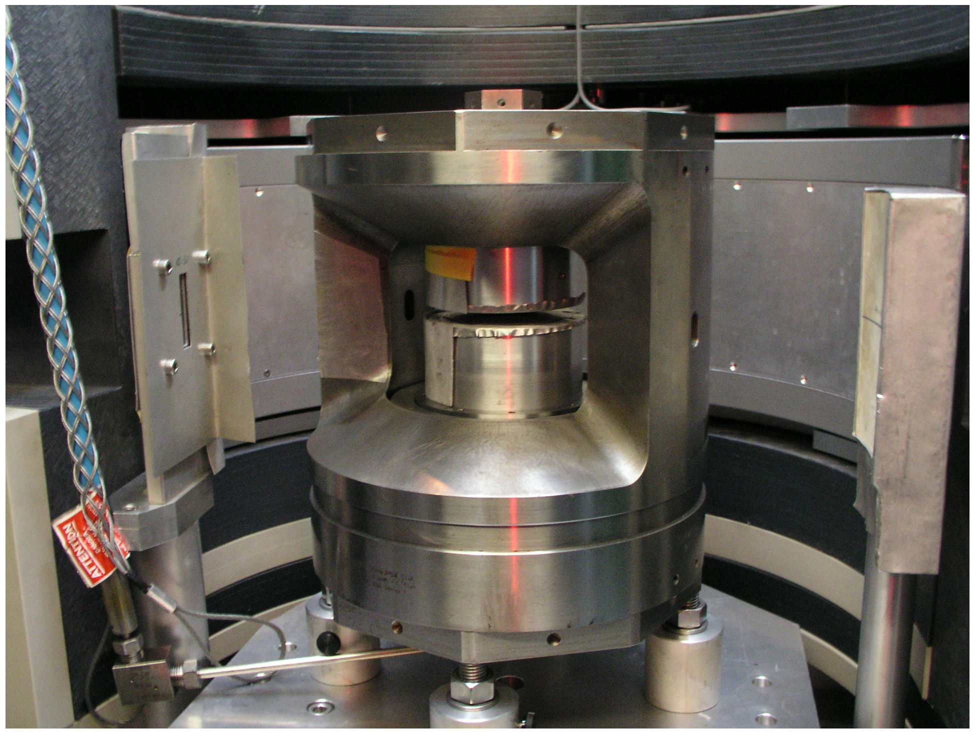



$$\mathbf{F}(\mathbf{H}) \propto \mathbf{M}_{\perp \mathrm{Cu}} \exp(2\pi i \mathbf{R}_{\mathrm{Cu}} \mathbf{H})$$

# Lattice distortion (0.1%) and magnetic structure in NiO under high pressures (up to 130kbar) at HRPT

@ p=1bar: $\mu_{Ni}$=1.73(9) $\mu_B$, k =[½ ½ ½] in *Fm3m*
*R3-m*: a=2.9534(2)Å, α=60.061(2)°

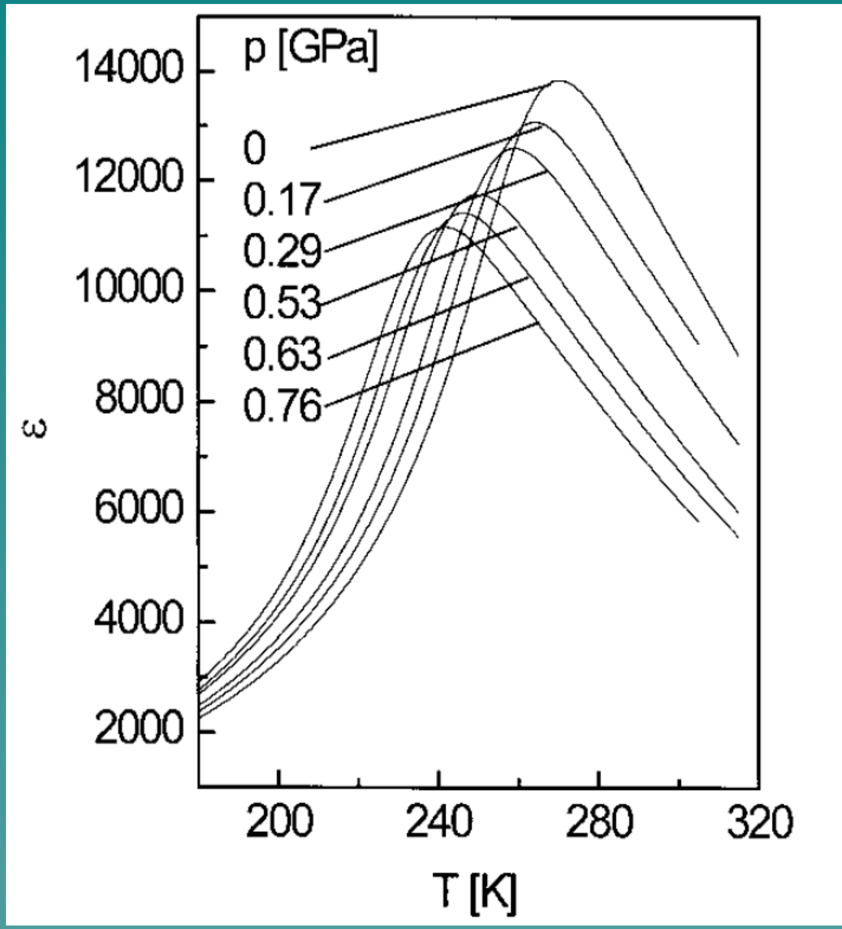


S. Klotz, Th. Strässle, G. Rousse, G. Hamel, V. Pomjakushin, *APL 2005.*

# High-pressure studies of $PbMg_{1/3}Ta_{2/3}O_3$ relaxor ferroelectric

S. Gvasaliya, V. Pomjakushin, B. Roessli, Th. Strässle, S. Klotz, S. Lushnikov




Relaxor ferroelectrics are peculiar crystals where the giant dielectric permittivity appears without structural phase transition. There is no theory which describe their properties. Among other anomalies, there is a suppression of the peak in the dielectric permittivity and of the intensity of diffuse scattering under hydrostatic pressure. In order to understand underlying physics the structure of a model relaxor was studies up to hydrostatic pressure P~7 GPa

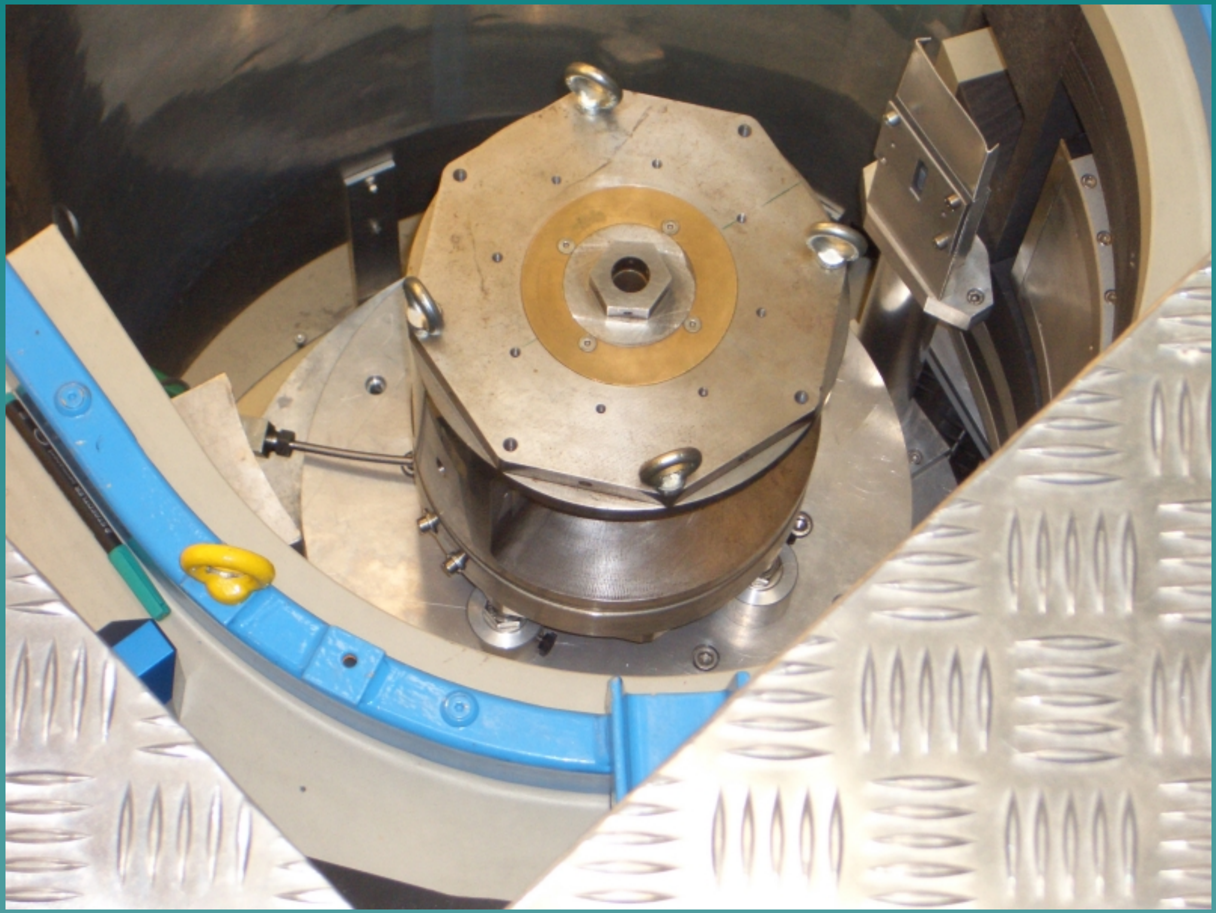
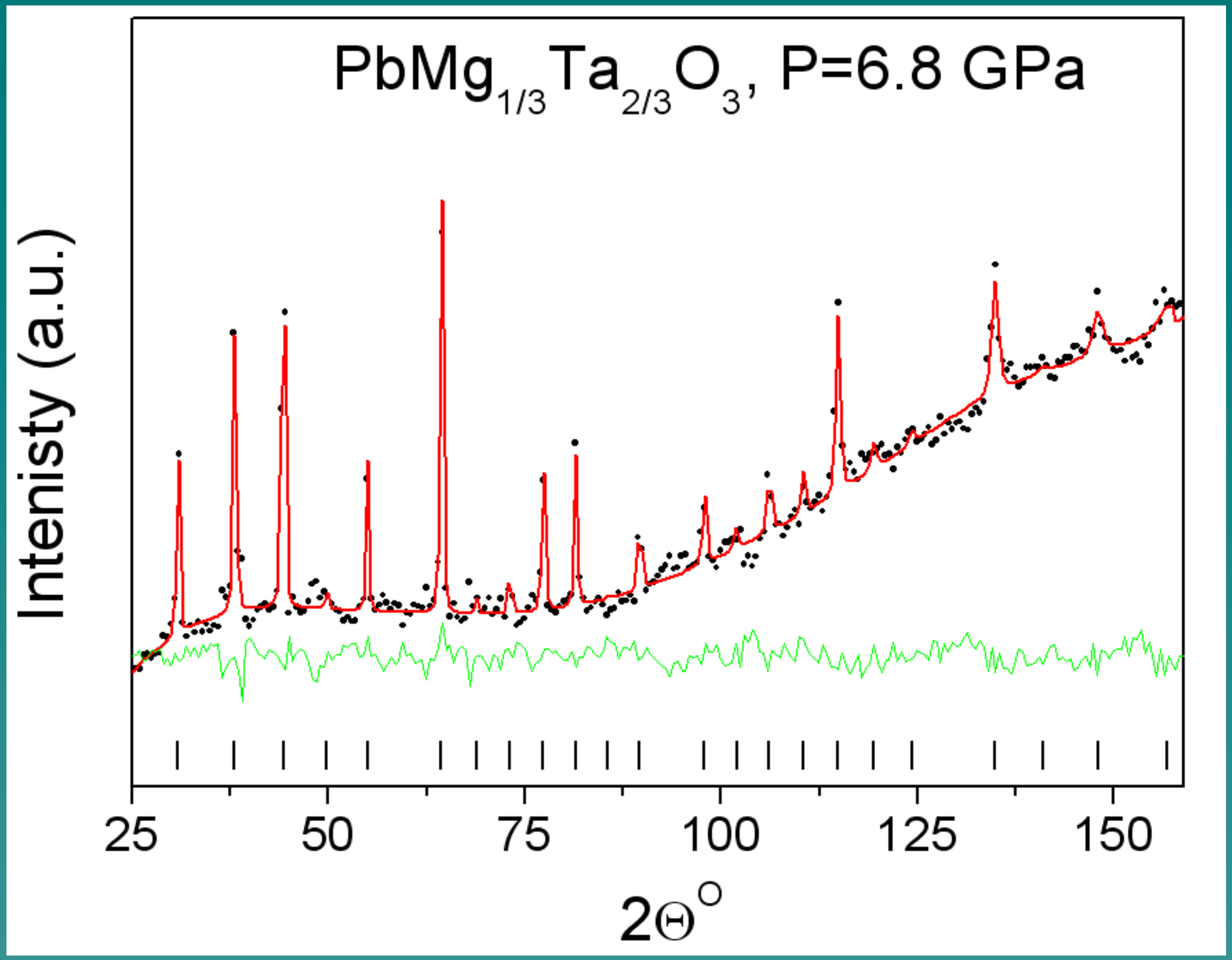
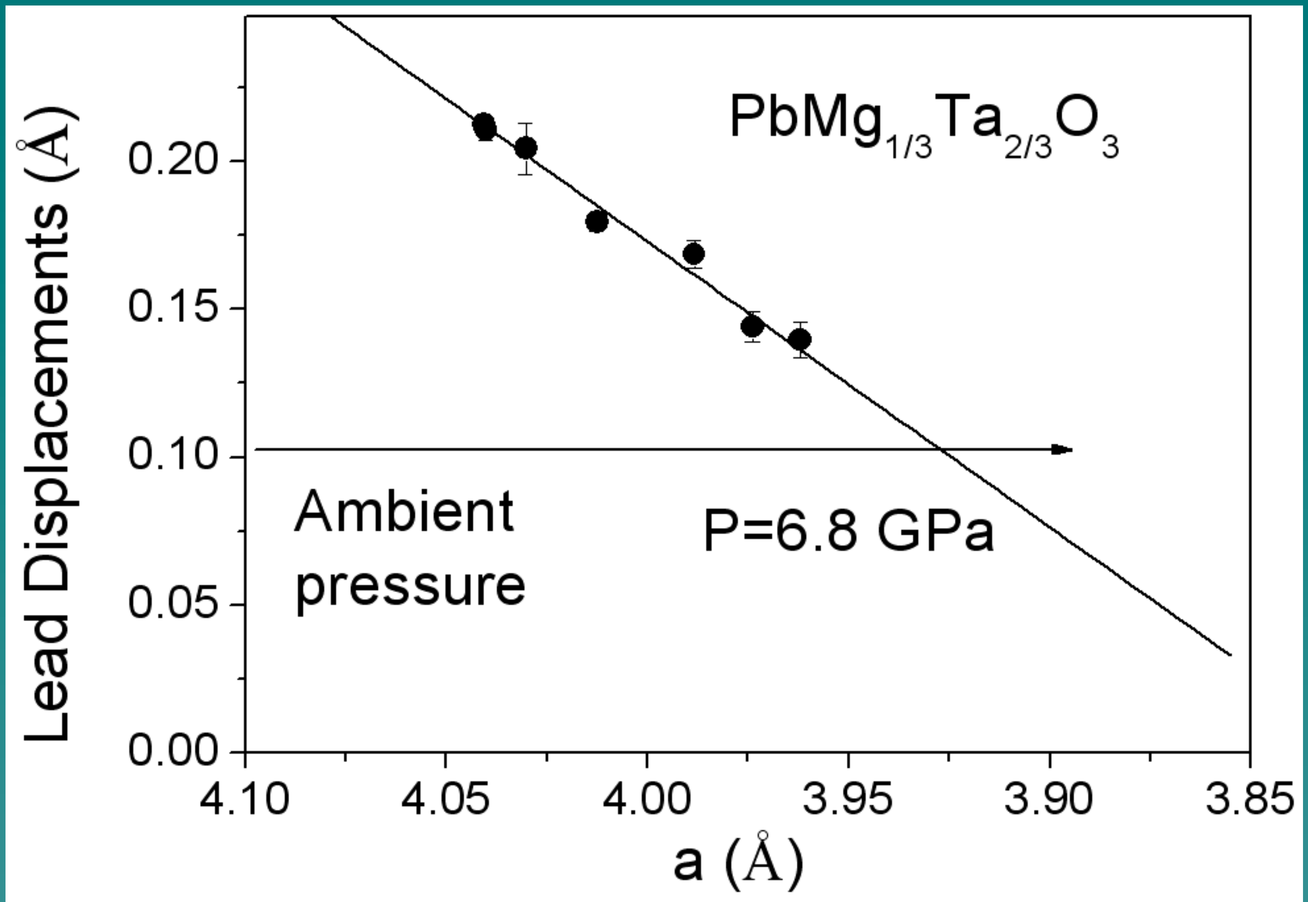
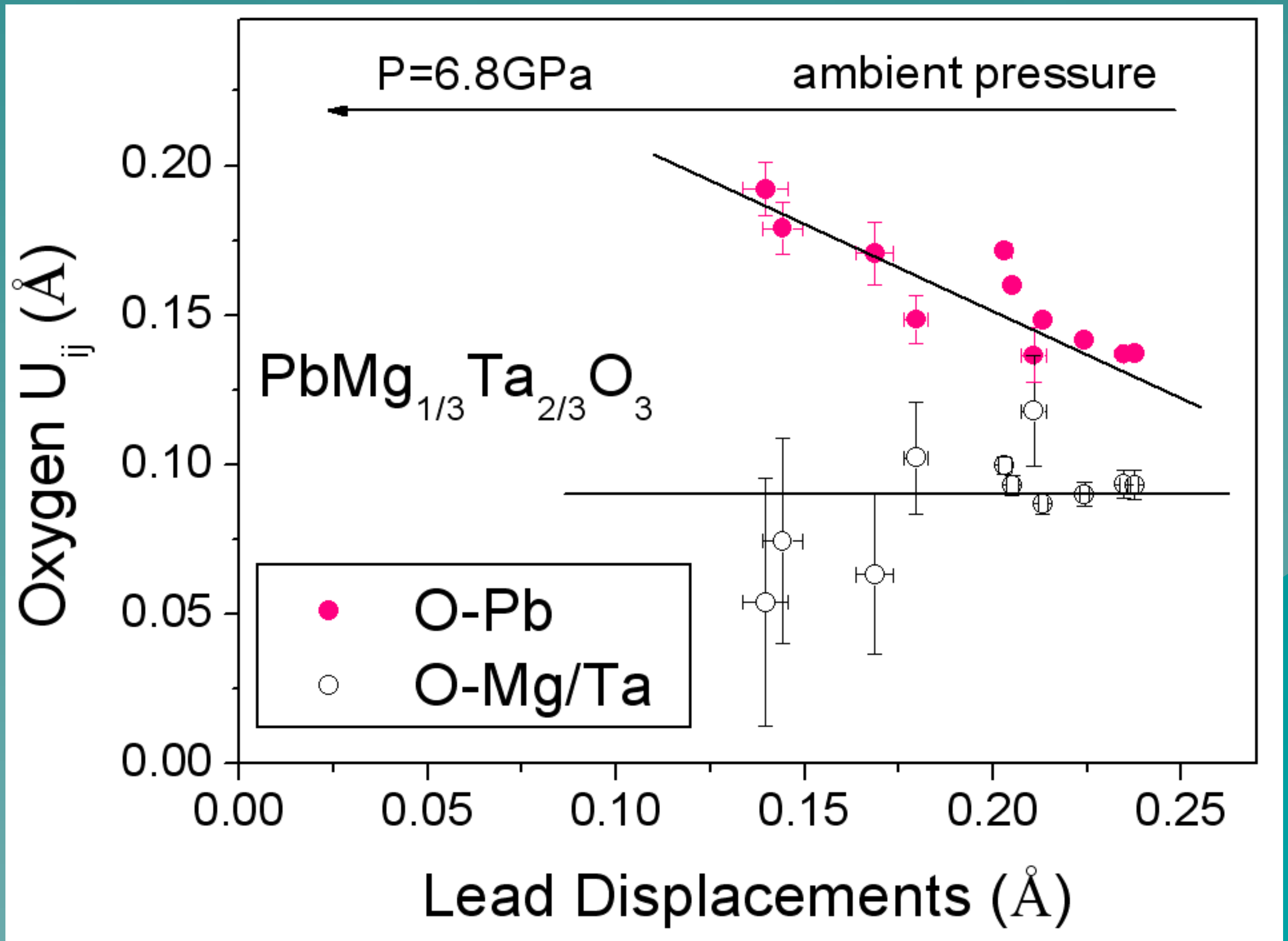


Photo of a high pressure setup using Paris-Edinburgh pressure cell at HRPT diffractometer. The sample volume is less than 100 $mm^3$, approximately two orders of magnitude smaller than in a standard setup.



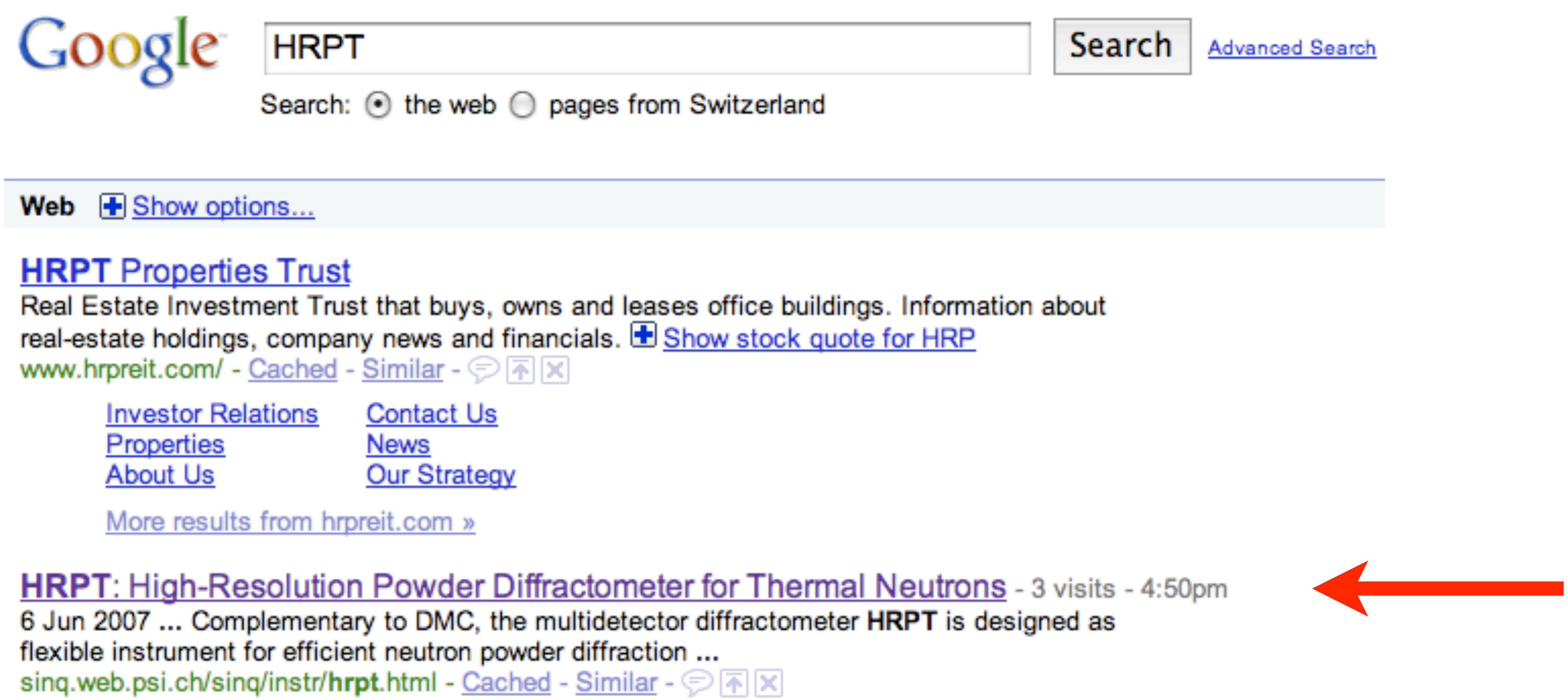

Observed and calculated diffraction spectrum from $PbMg_{1/3}Ta_{2/3}O_3$. Increased background is probably due to the unmasked part of the steel leg of the pressure cell. The crystal structure remains cubic at all pressures. The important changes are: (i) Reduction of the Lead displacements at increased pressures (ii) Appearance of the anisotropy in the Oxygen thermal motion – its ellipsoid becomes significantly elongated toward the Lead ions. Thus these change are responsible for the suppression of the peak in dielectric permittivity and of the diffuse scattering. Similar behaviors were never reported earlier.



# More information about HRPT

http://sinq.web.psi.ch/hrpt

OR



# The End