Powder neutron diffraction at continuous spallation source SINQ

Vladimir Pomjakushin

Laboratory for Neutron Scattering and Imaging, LNS, Paul Scherrer Institute

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Applications of (high resolution) neutron powder diffraction

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4) materials science with big non-standard shape “real life” samples, e.g. electrical batteries or residual stresses in industrial materials.

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5) Automatic data reduction system. E.g., let's consider 5 samples/day each measured at 20 temperatures (~15'/point)

Overview of the talk

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- Swiss neutron spallation course SINQ
- ND @ PSI
- Q-range, resolution, maximal cell volume, peak overlap.
- complementarity of DMC and HRPT diffractometers

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- Examples of results demonstrating the possibilities of SINQ powder diffractometers.
  - Accuracy on crystal metric in multiferroic $TmMnO_3$
  - Topologically nontrivial skyrmionic incommensurate superspace magnetic structure in Well semimetal CeAlGe.
  - A quantum liquid of magnetic octupoles in pyrochlore Ce2Sn2O7

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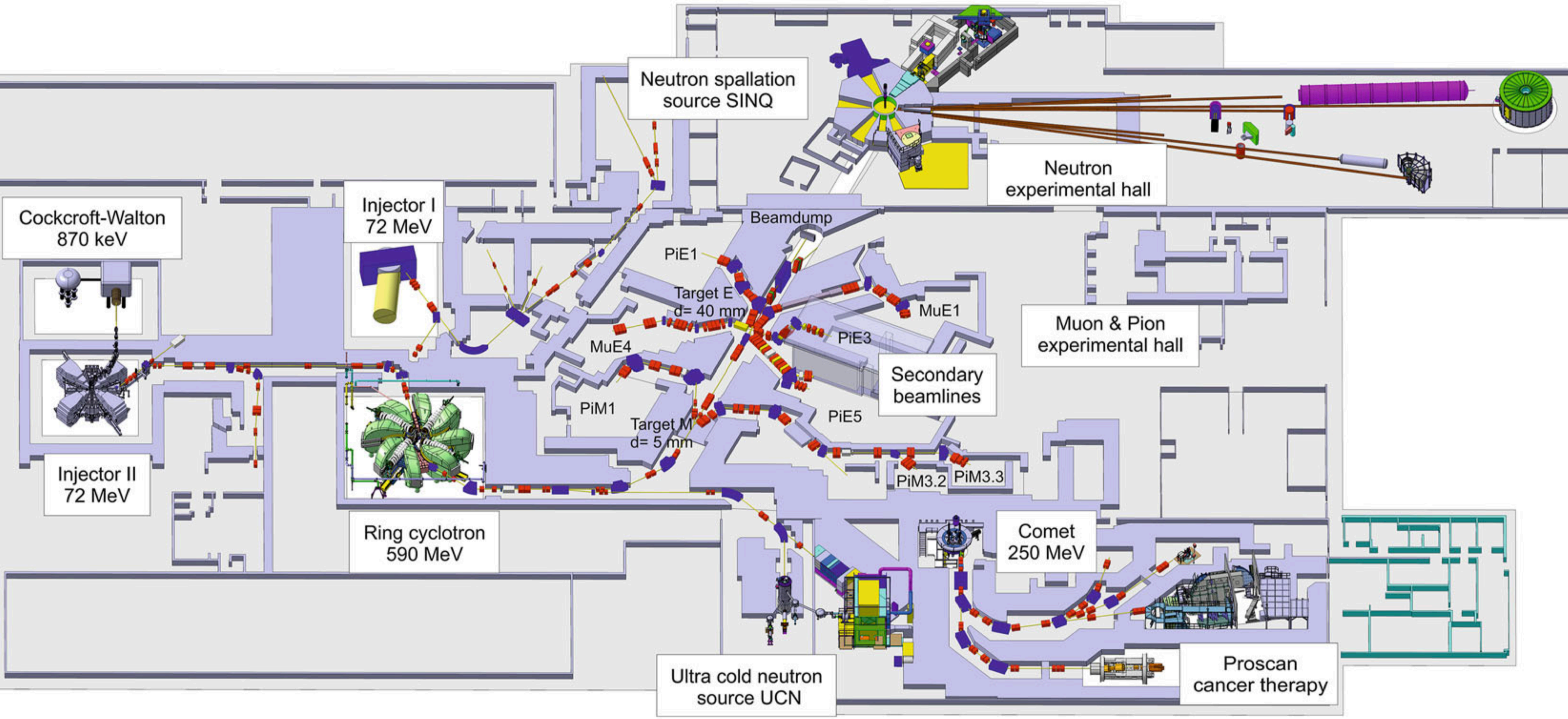
- HRPT specific features
  - radial collimators: pluses and one minus
  - Sample changers
  - sample positioning and atomic displacement parameters
- Sample environment. Other non-dedicated equipment

- Wish list for the future

Overview of the accelerator facility HIPA/PSI



D. Kiselev, et al J Radioanal Nucl Chem (2015) 305:769

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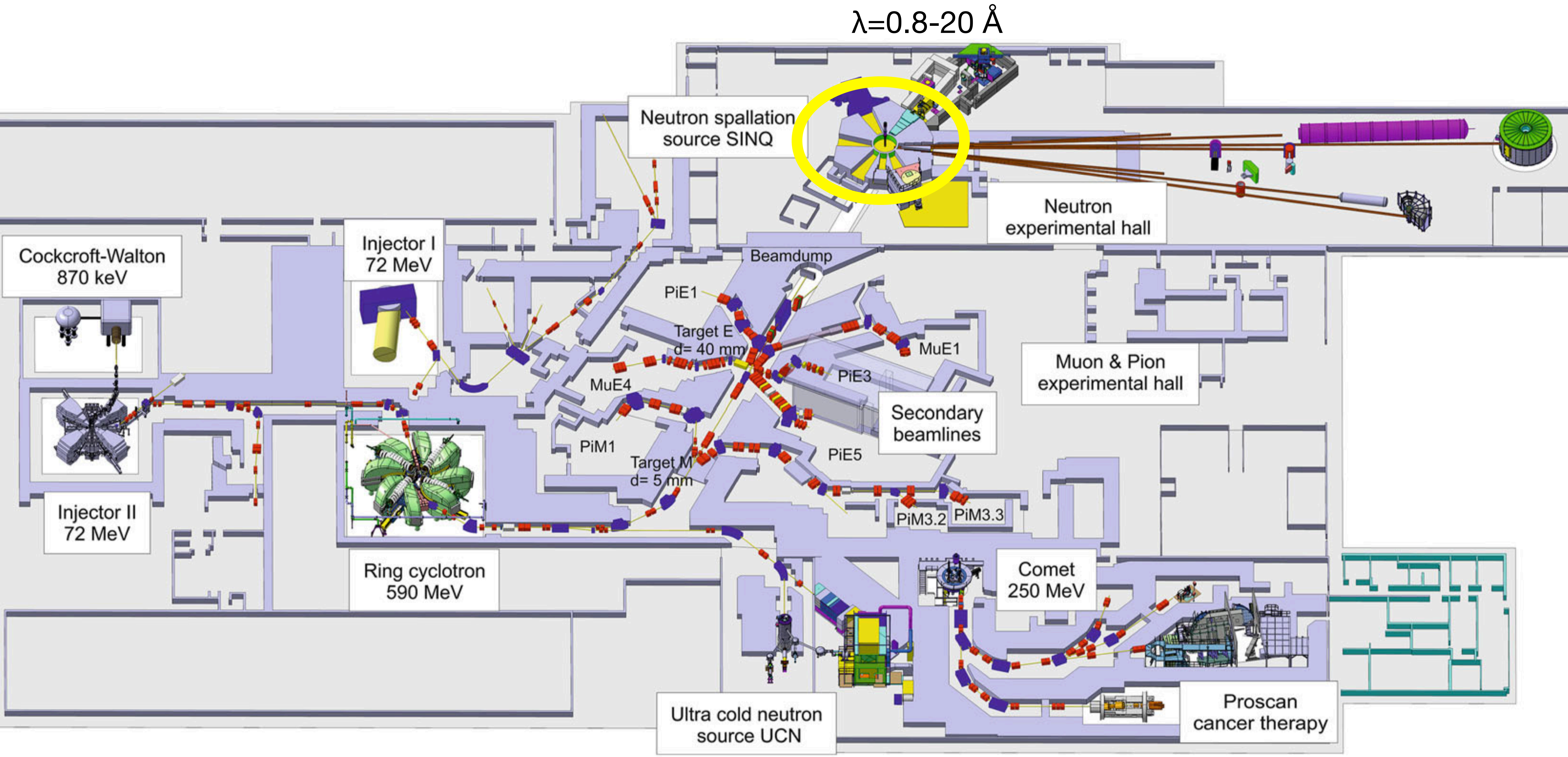

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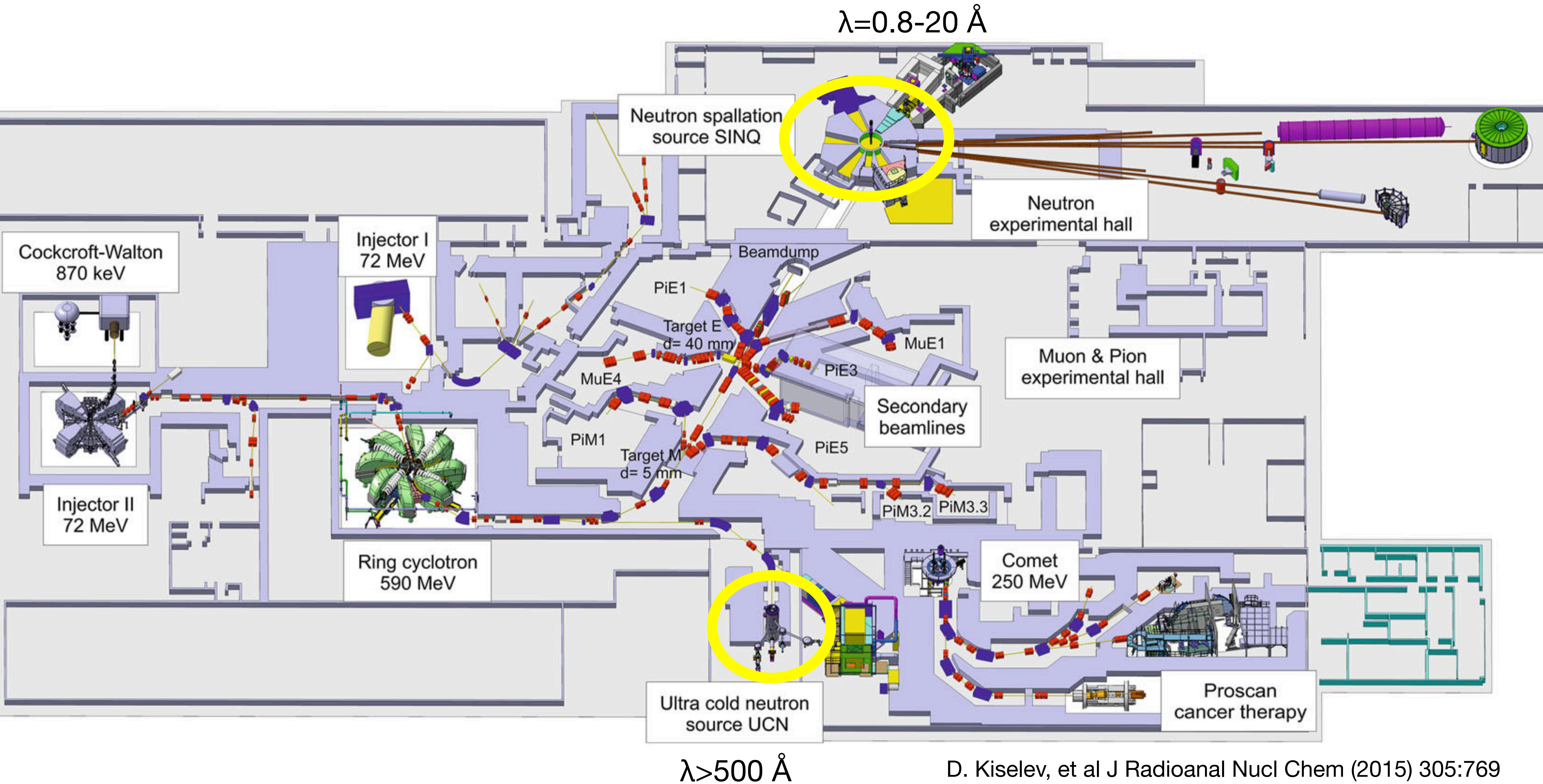

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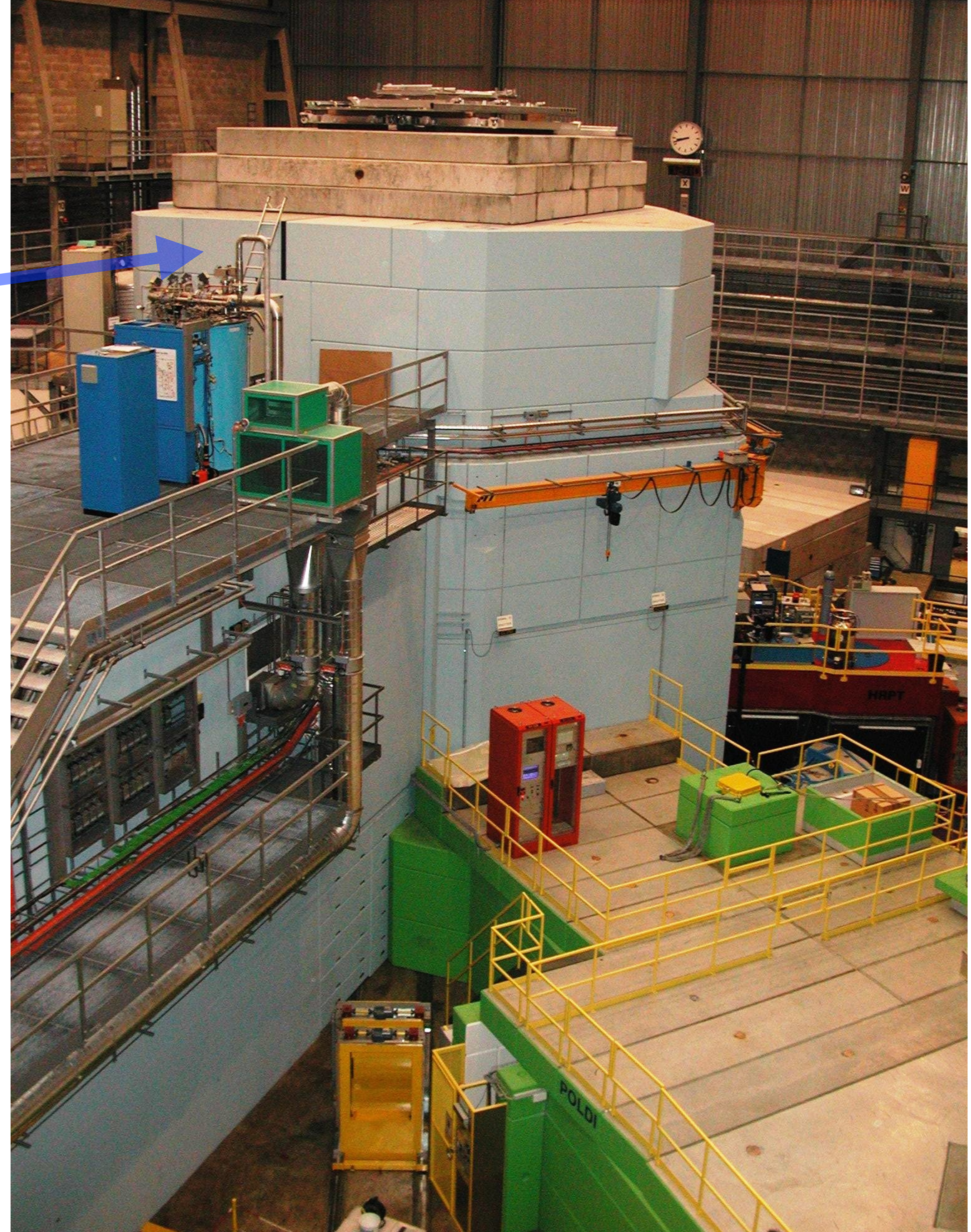
The *spallation neutron source* SINQ is a *continuous* source - the first and *the only of its kind in the world* - with a **flux of about 4 10^{14} n/cm^2/s**. Beside thermal neutrons, a cold moderator of liquid deuterium (cold source) slows neutrons down and shifts their spectrum to lower energies.

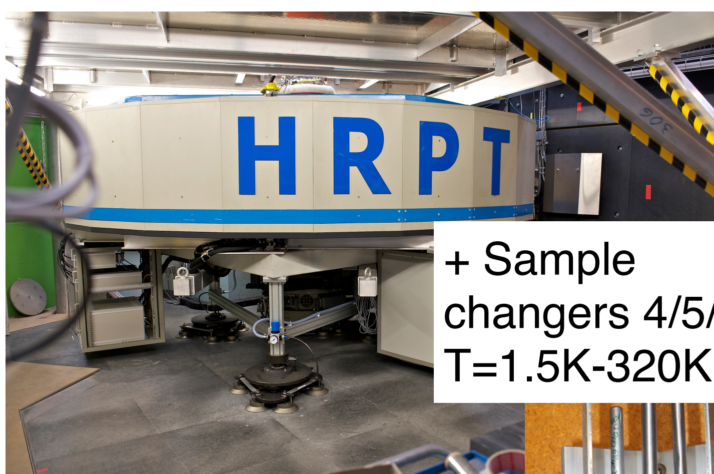
Flux of the monochromatic beam at diffraction instruments is about 10^5 - 10^6 n/cm^2/s



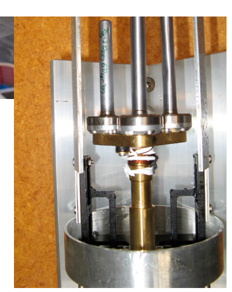
SINQ diffraction instruments overview

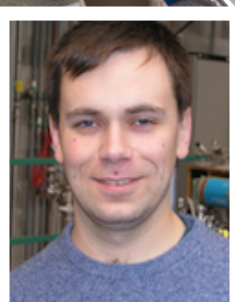
Instruments HRPT&DMC (Powder), TriCS (Single crystal), POLDI (strain)
and TASP/MuPAD (polarised, 3D spherical neutron polarimetry)

New materials in condensed matter physics, chemistry and materials science with a focus on magnetism
Examples are: energy research, frustrates systems, crystallography, ferroelectrics



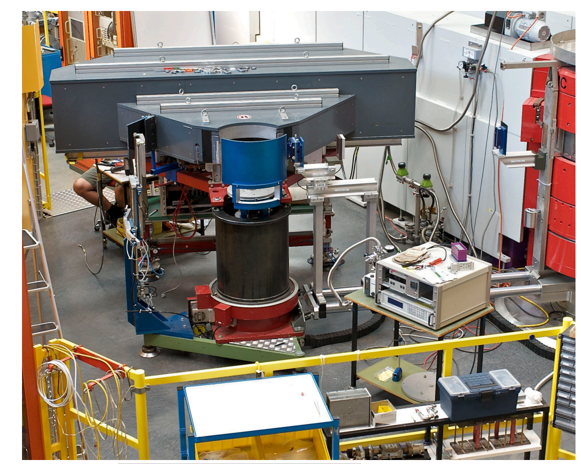
+ Sample
changers 4/5/8
T=1.5K-320K





λ=0.84 - 2.96 Å

HRPT: V. Pomjakushin, D. Sheptyakov



λ=2.35-5.4 Å

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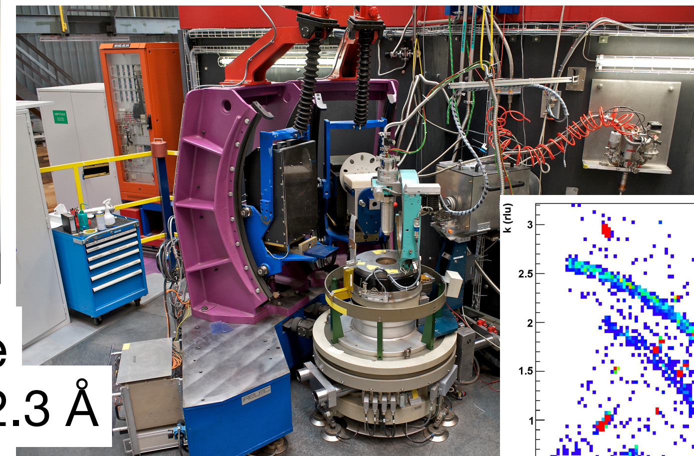
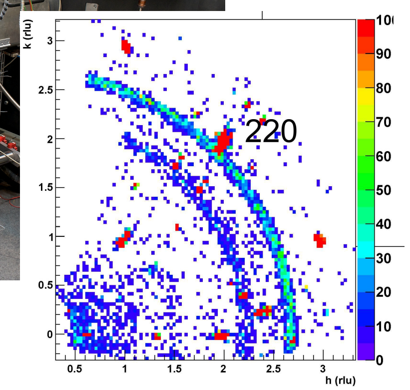
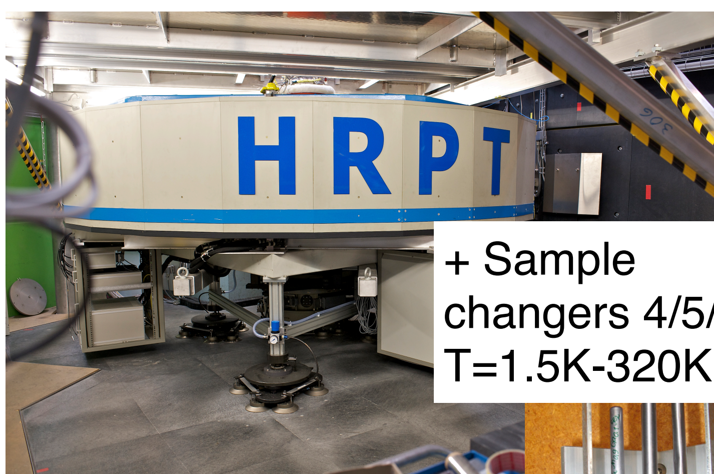




ZEBRA, O.Zaharko, R.Sibille
λ=1.18, 2.3 Å



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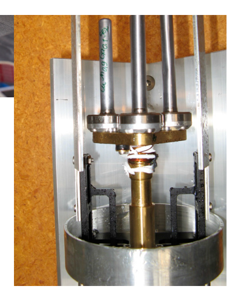

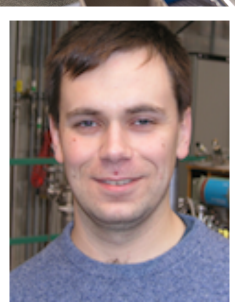

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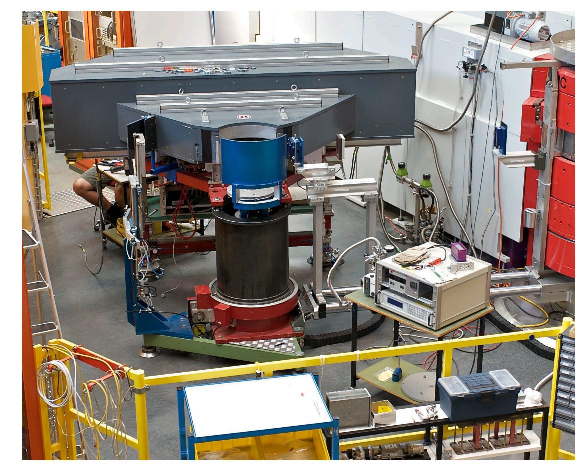

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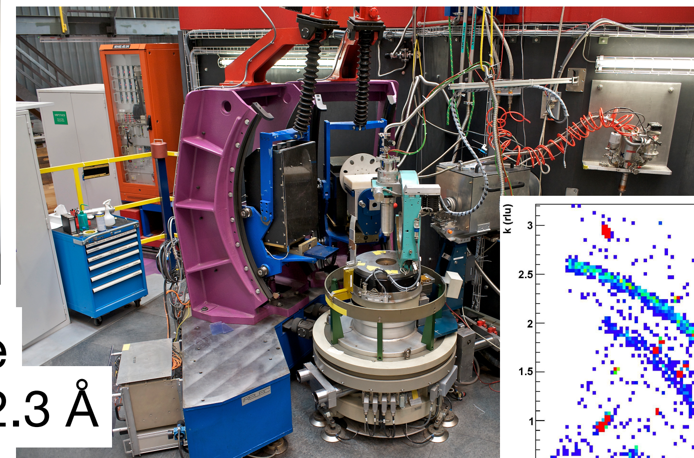

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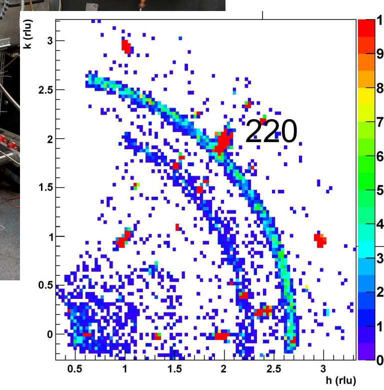
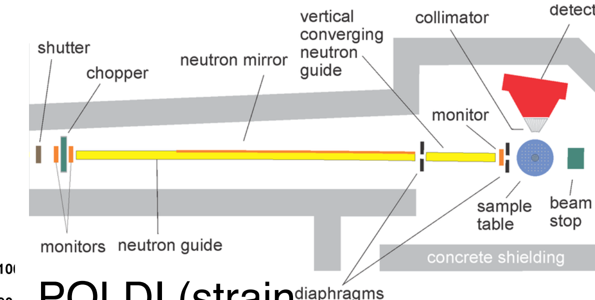
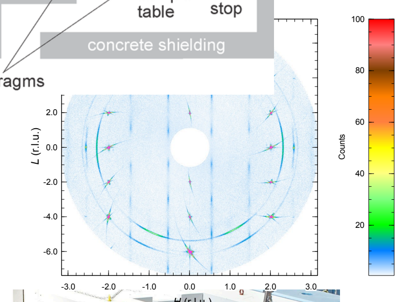
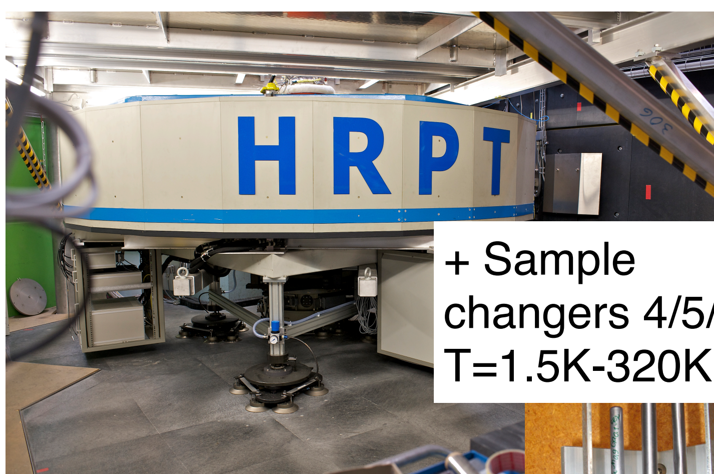


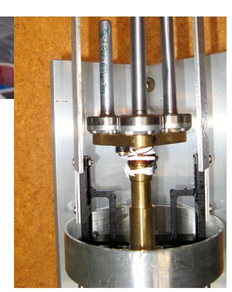


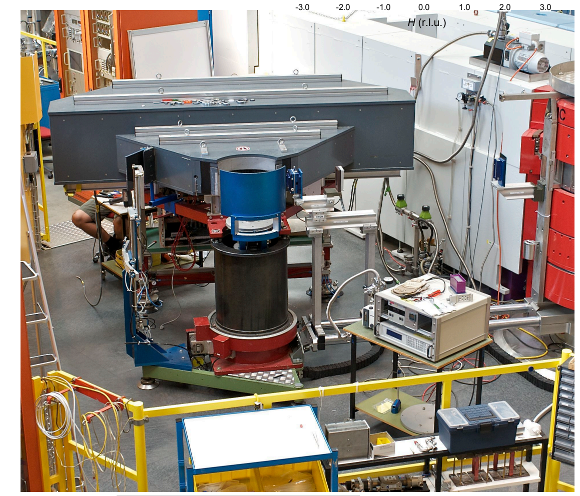
POLDI (strain scanner) TOF
Laue option
λ=white beam

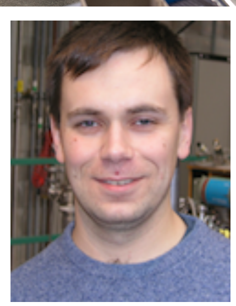




+ Sample changers 4/5/8
T=1.5K-320K





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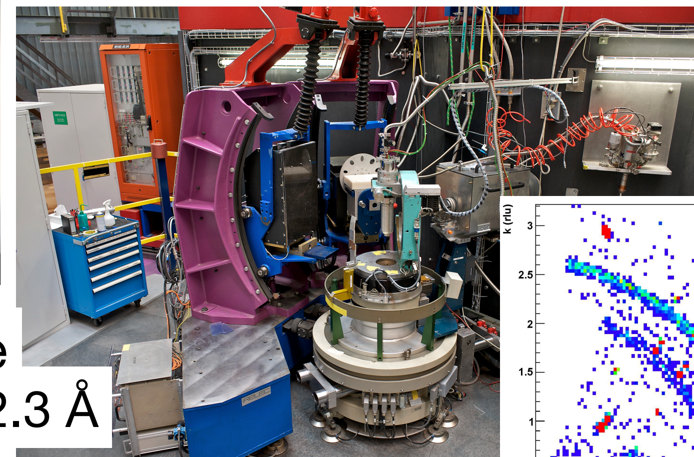

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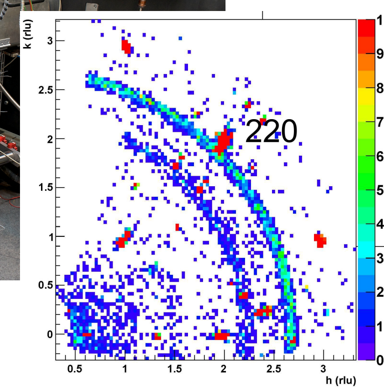

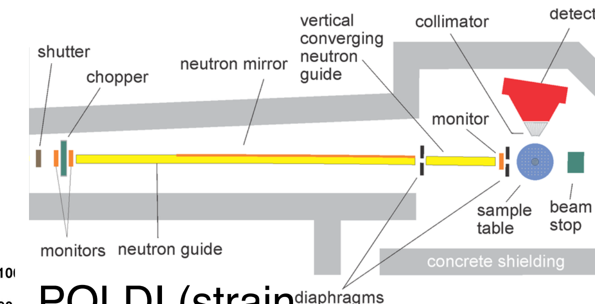

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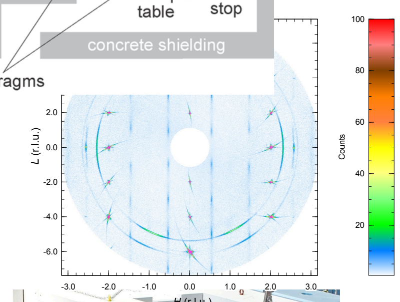

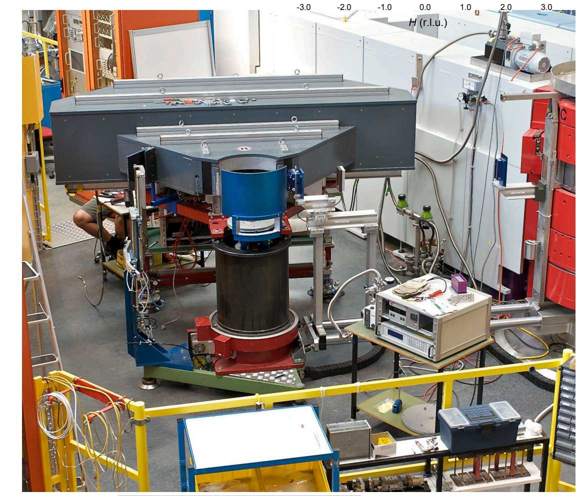

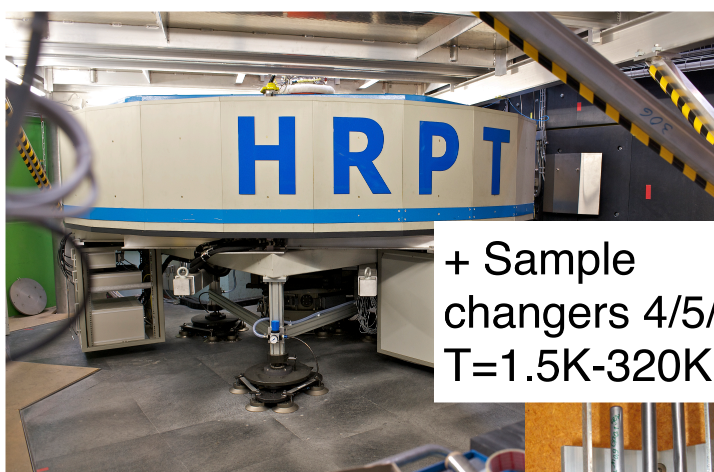

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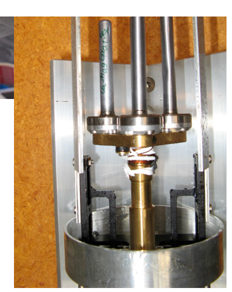
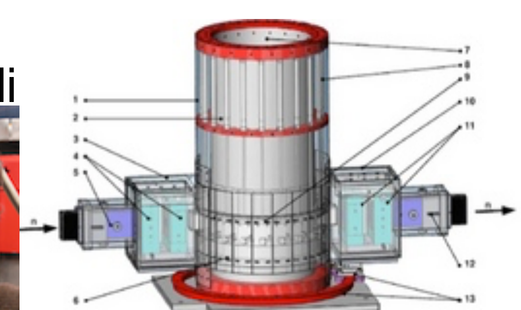
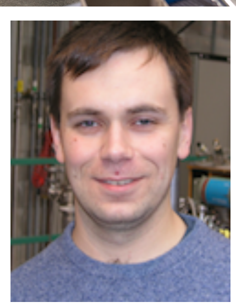


TASP/
MuPAD:
B. Roessli


λ=2.35-5.4 Å

λ=0.84 - 2.96 Å

HRPT: V. Pomjakushin, D. Sheptyakov

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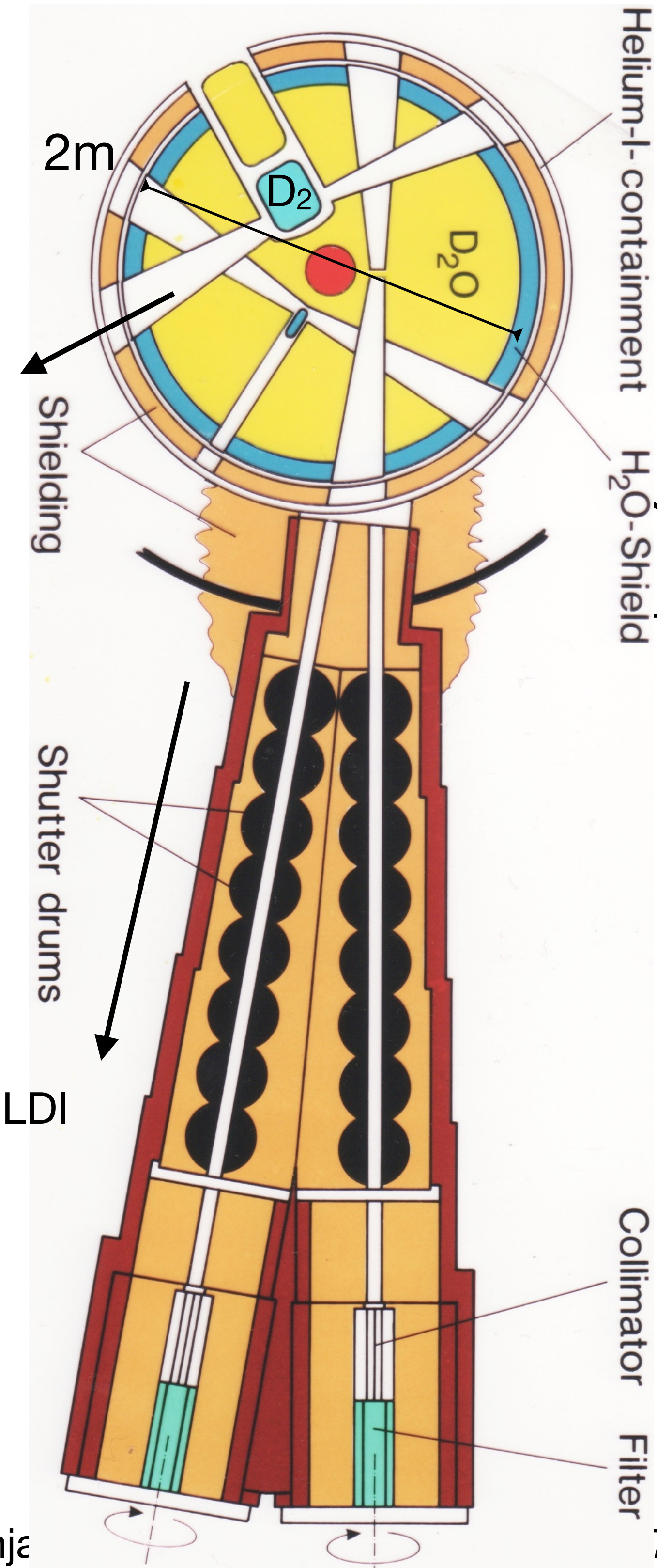
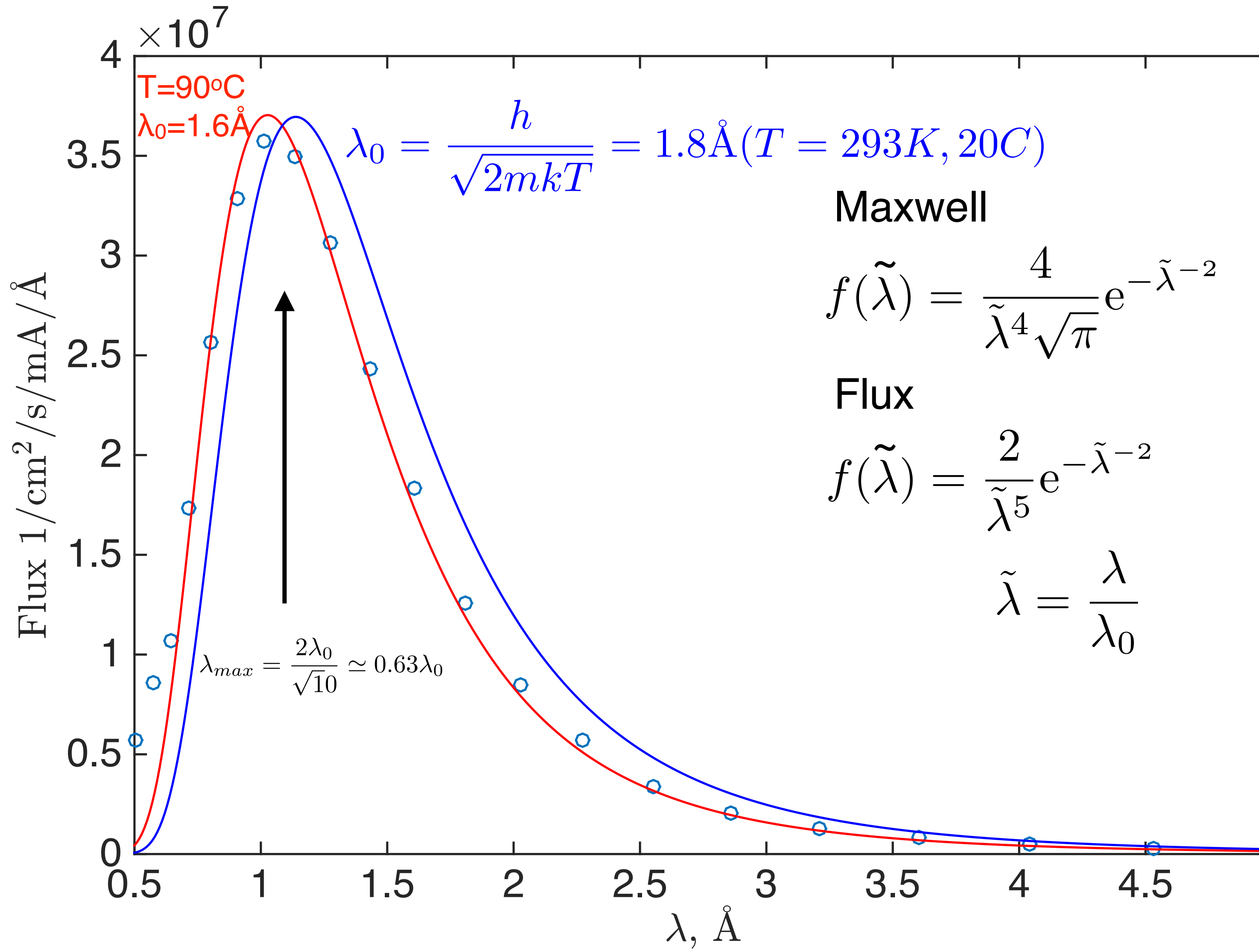
Neutron (thermal) flux from the D_2O moderator, Maxwellian at 90°C (HRPT, ZEBRA, POLDI)





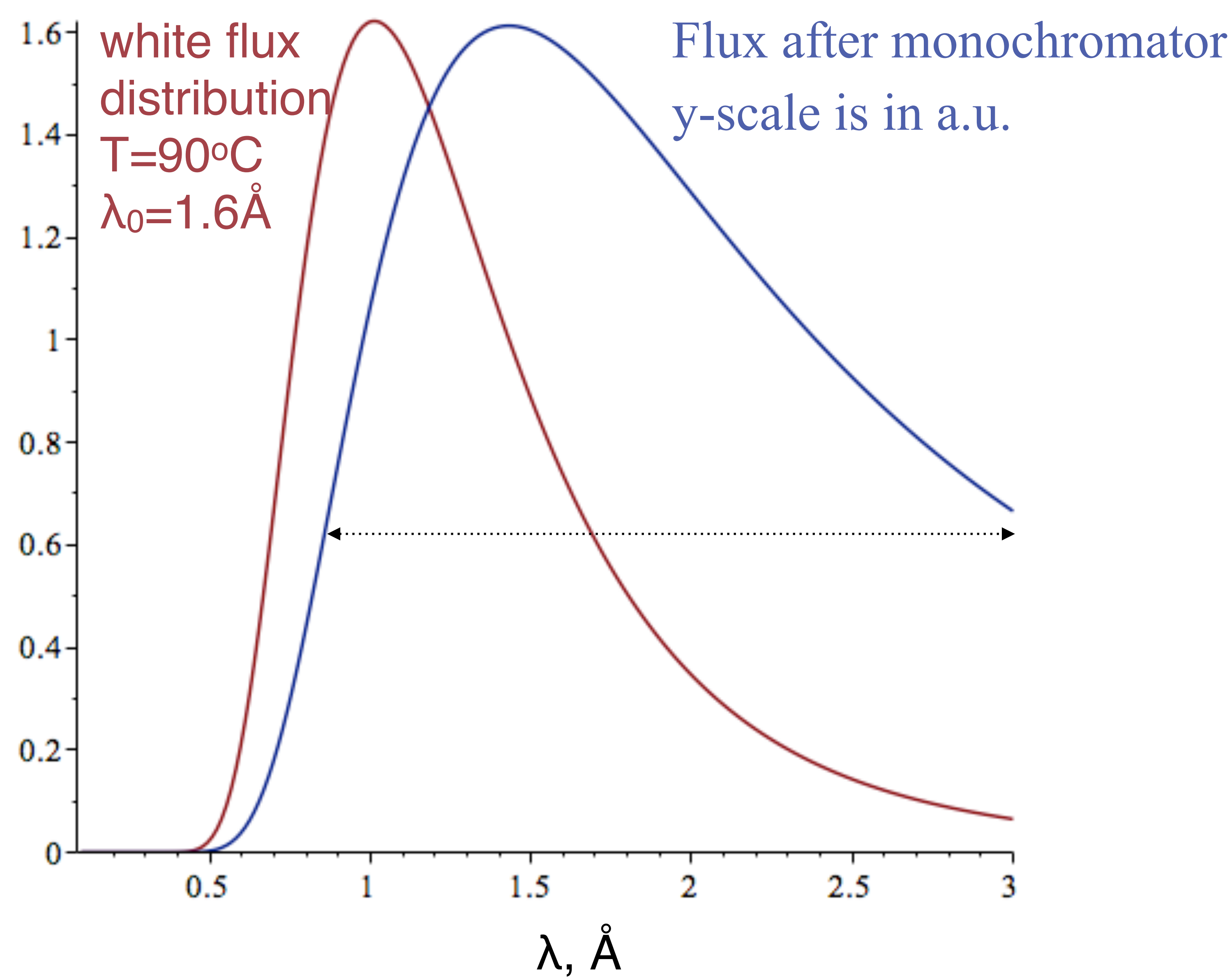
Total: $5 \cdot 10^7$ $1/\text{cm}^2/\text{s}/\text{mA}$
at SINQ current 2mA: 10^8

wavelength range from the "white" flux. at HRPT λ=0.84 - 2.96 Å

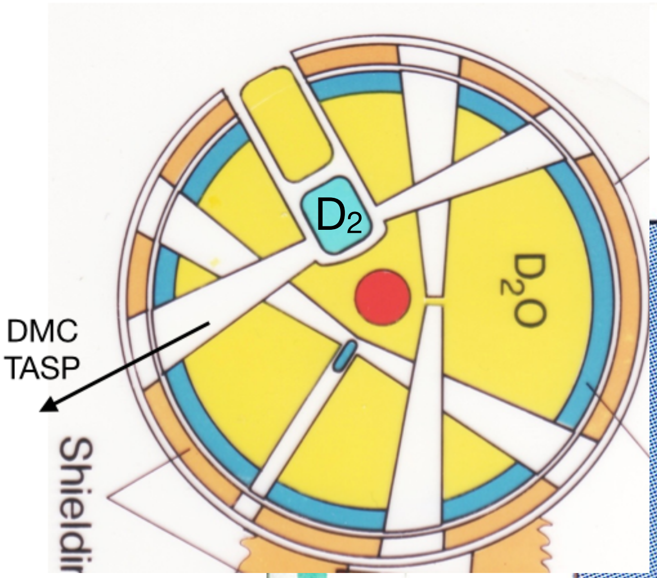
Intensity of Bragg scattering from big single crystal: Lorentz factor, extinction, geometry, …

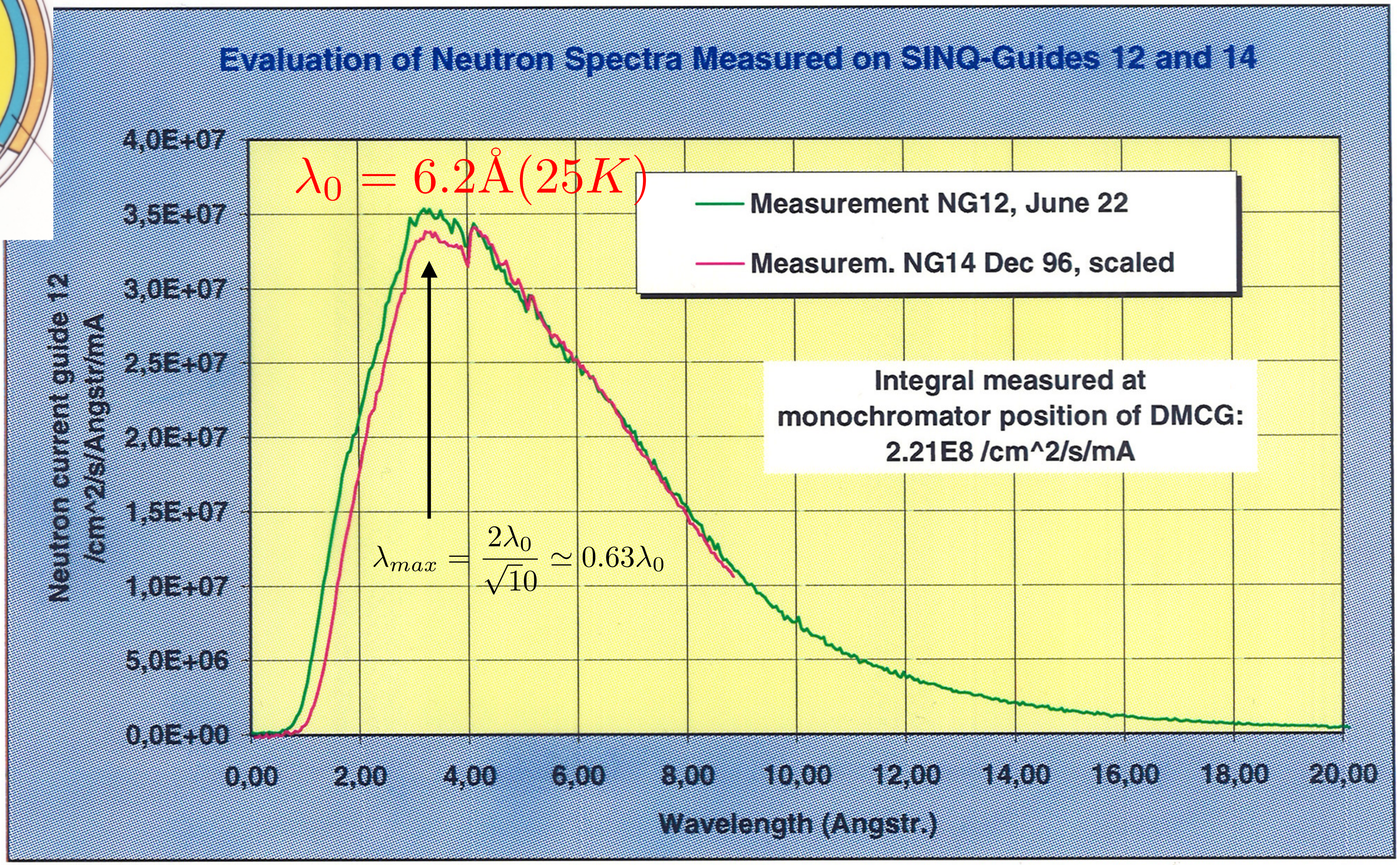
$$I \sim f(\lambda)\, \Delta\lambda C(\lambda,\theta) \sim f(\lambda)\, \lambda^{2.5} C'(\theta)$$

for fixed monochromator take-off 2θ for HRPT



Neutron flux from cold moderator (DMC,SANS,TASP), liquid D_2, T=25K or -248C





BQ36 / 23.07.1997 SINQ-NL12_tof.xls Diagramm4

Diffraction instruments for solid state physics problems at swiss spallation source SINQ

- **HRPT** - High Resolution Powder Diffractometer for Thermal Neutrons, λ=0.84 - 2.96 Å (max intensity at 1.15-1.89 Å), High resolution 10^{-3} and high Q-range ≤14.3 $Å^{-1}$

$$Q = \frac{4\pi \sin\theta}{\lambda} = 2\pi/d$$

- **DMC** – High Intensity Powder Diffractometer for Cold Neutrons,
λ=2.35 - 5.4 Å (max intensity at 4-5 Å), high Bragg scattered intensity (up to x10 HRPT) and good resolution at low and moderate Q ≤4$Å^{-1}$. min Q~ 0.1$Å^{-1}$

magnetic structure oriented

General purpose

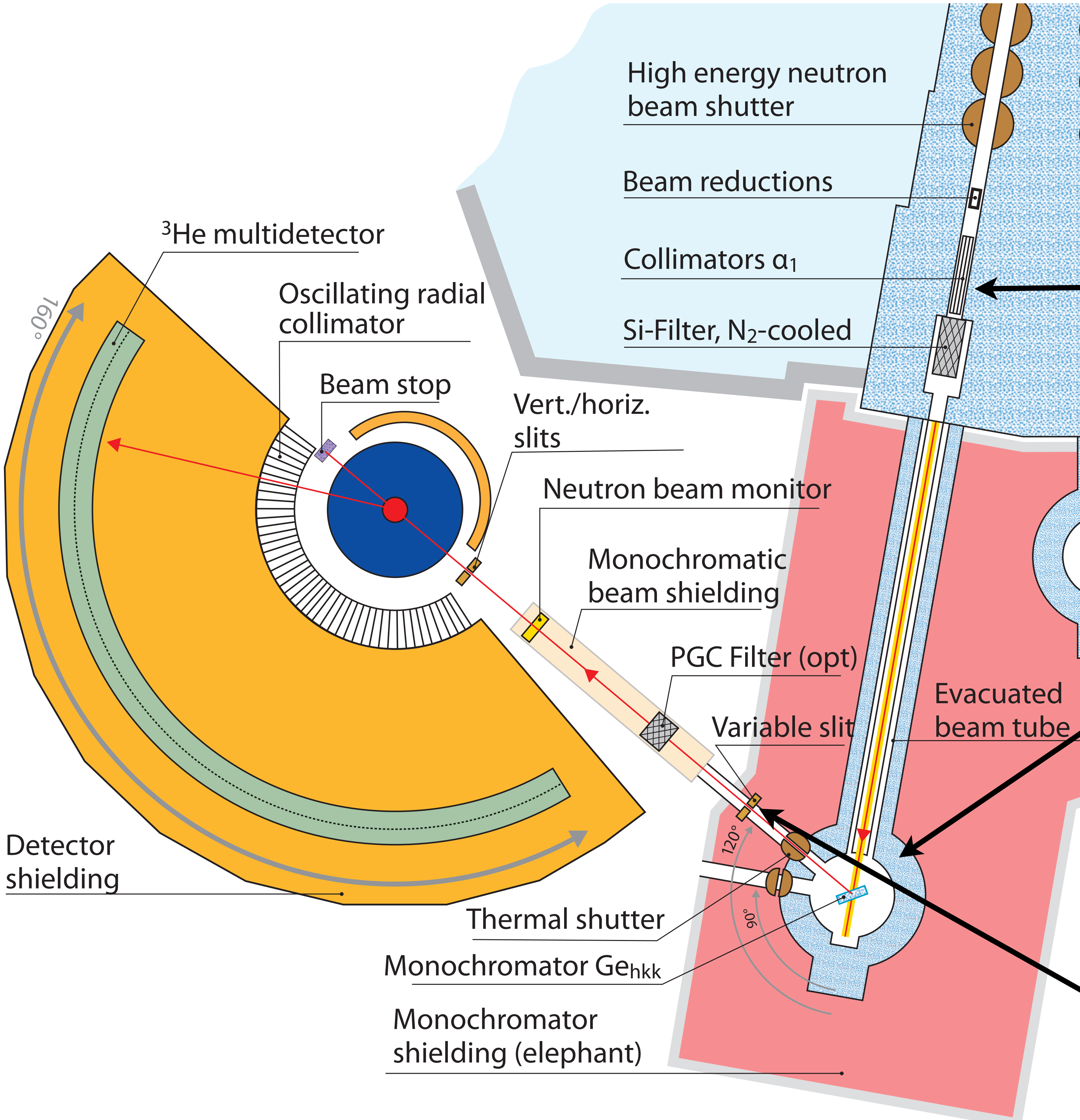
- **ZEBRA** - Single crystal diffractometer, λ=1.18, 2.3 Å, Thermal Neutrons

- **TASP** (triple axes) with **MuPAD** for polarised ND, Cold Neutrons

- small angle neutron instrument **SANS-I**, Q-range: $6{\cdot}10^{-3}$ nm^{-1} (0.0006 $Å^{-1}$) to 5.4 nm^{-1} (0.54 $Å^{-1}$) - up to 1$Å^{-1}$ with lateral shift by 50 cm

HRPT layout

High Resolution Powder Diffractometer
for Thermal Neutrons



horizontal angular divergence control

α_1

primary beam collimators:
6', 12', 24', ~40' (open)

α_2

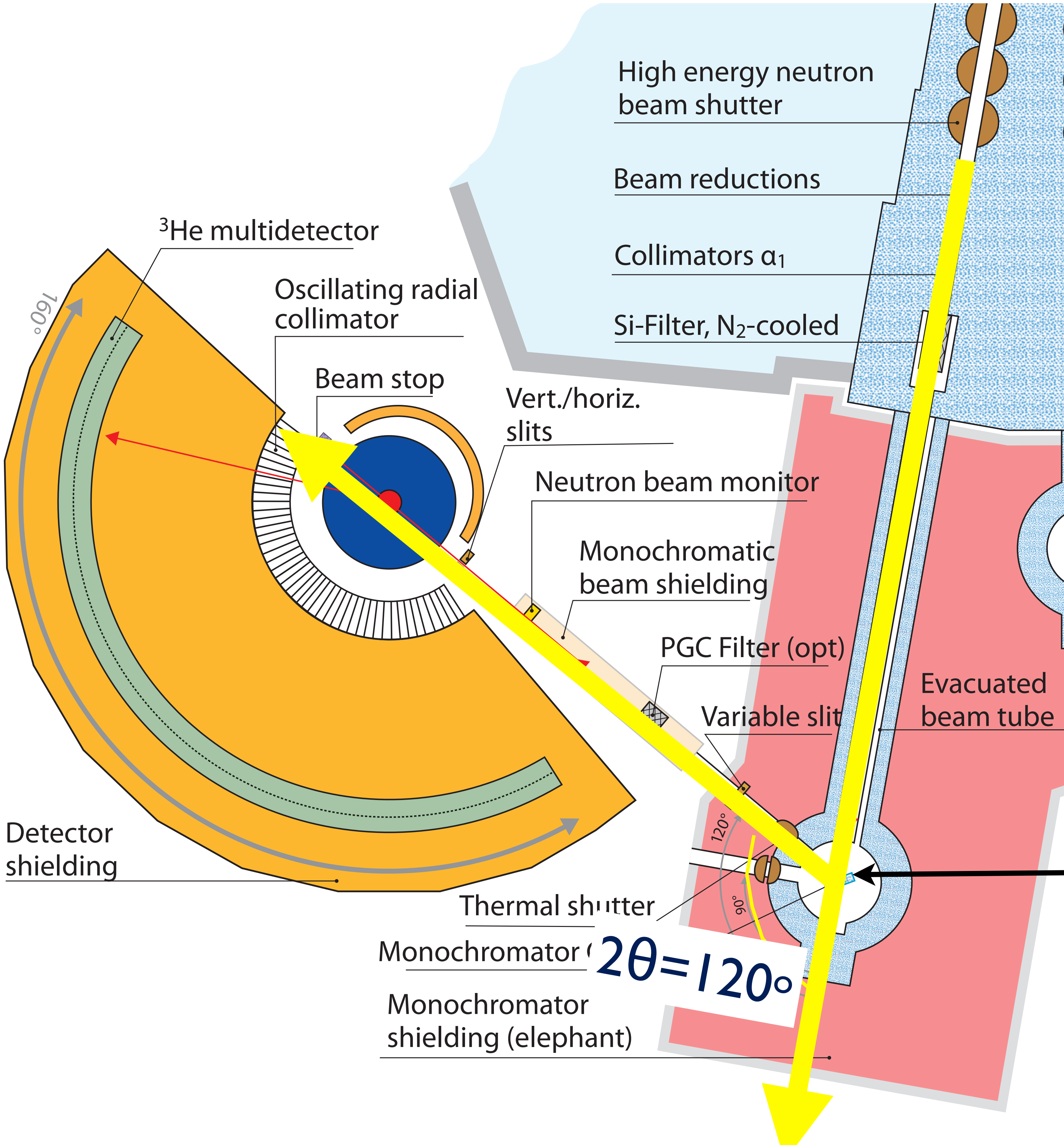
mosaic spread of the monochromator 15'

α_3

slit system for monochromatic beam and sample diameter

HRPT layout

High Resolution Powder Diffractometer for Thermal Neutrons

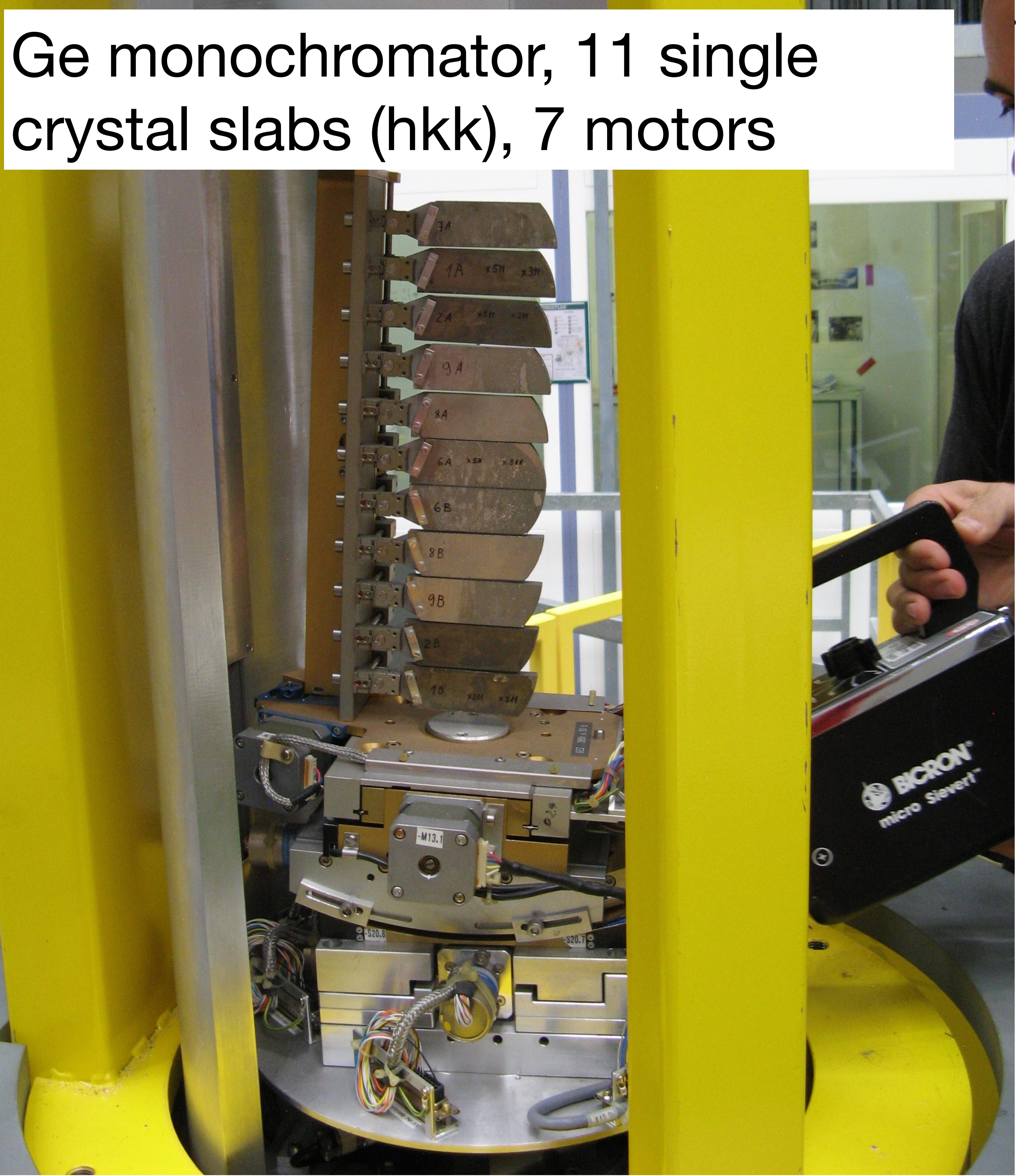


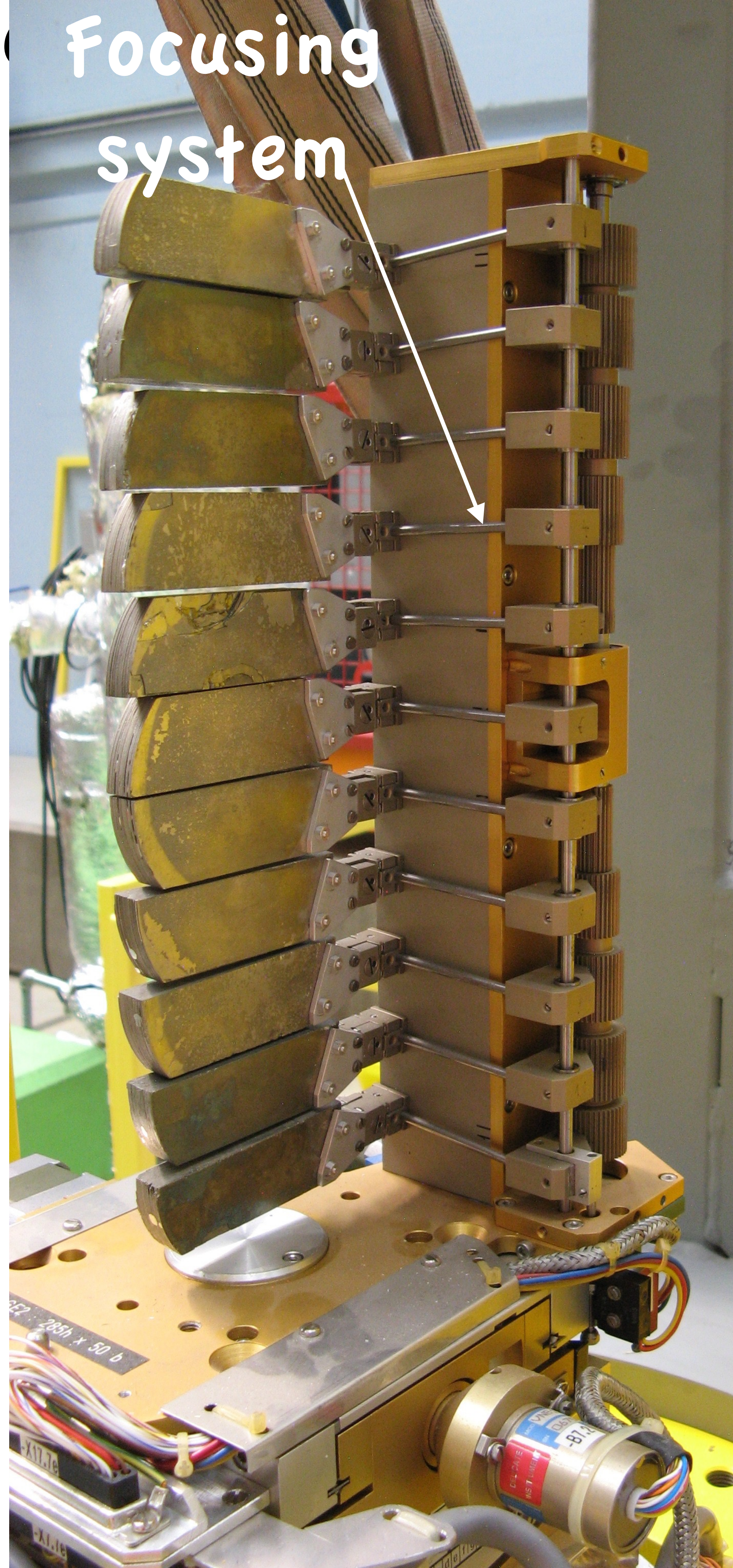
Ge neutron monochromator
fixed 120 take off angle

$$\lambda = 2\,d\sin\left(\theta\right)$$

$$\lambda = 2\,d\sin\left(60^{\circ}\right)$$

Ge monochromator, 11 single crystal slabs (hkk), 7 motors



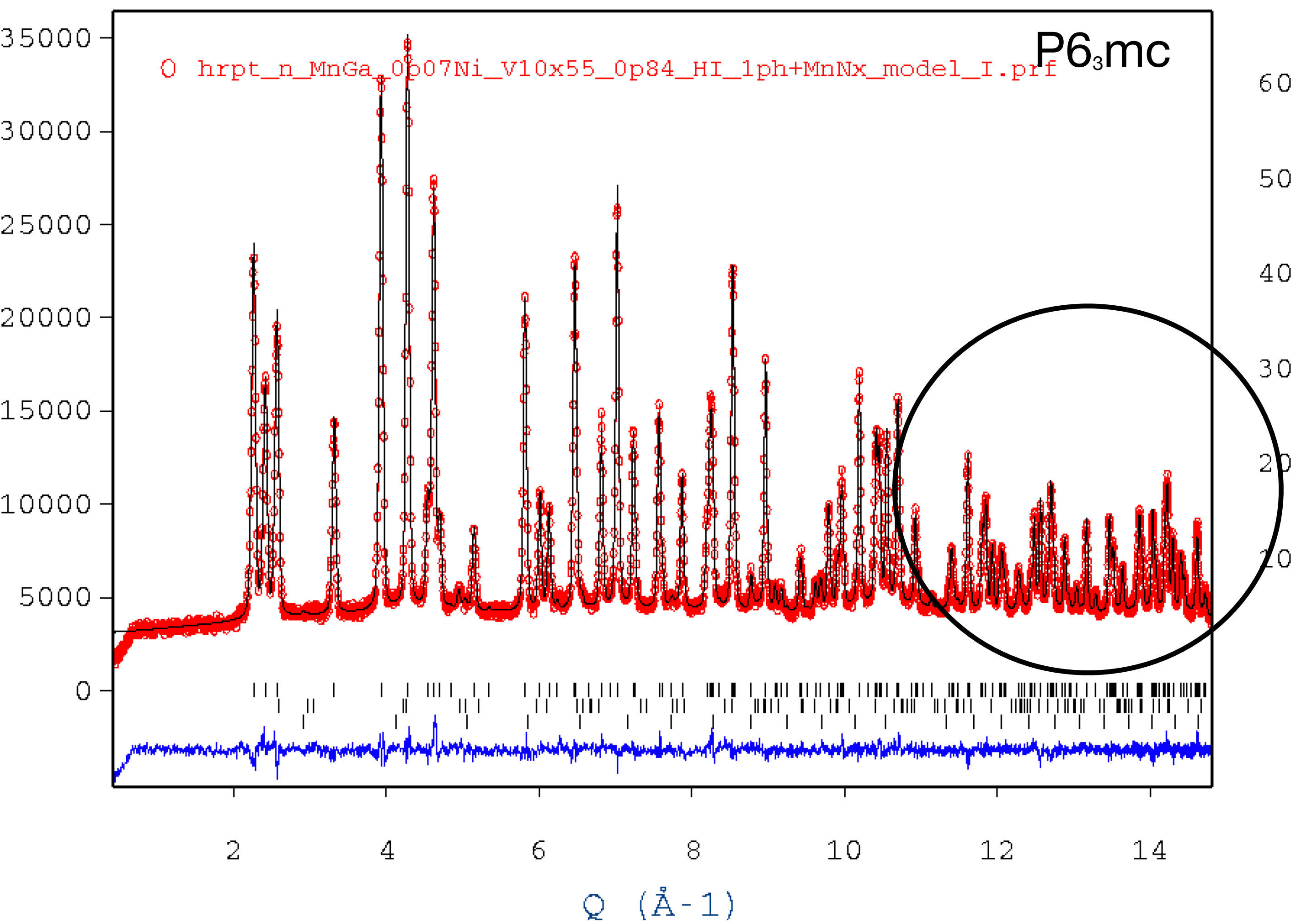


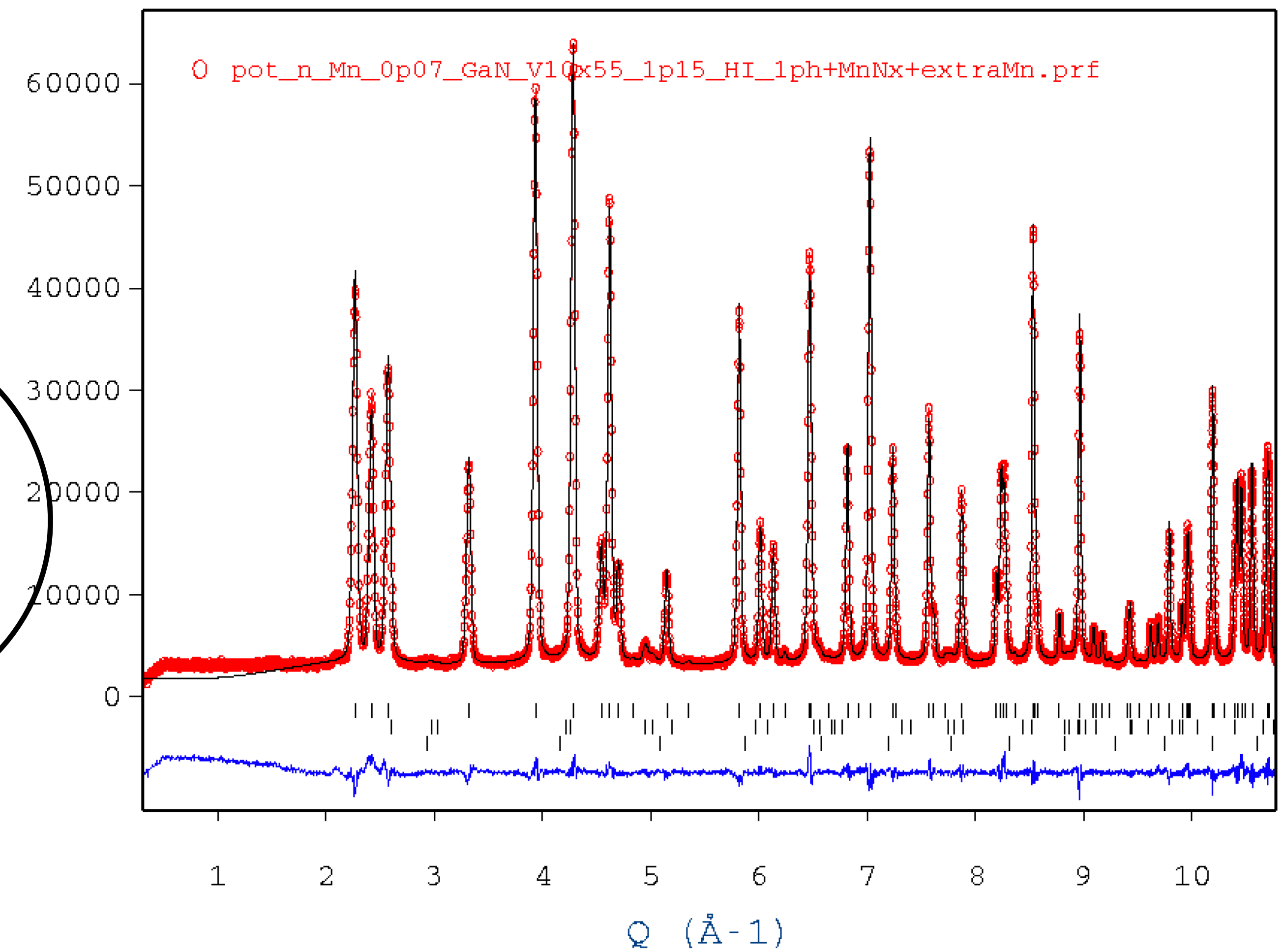
choice of wavelength at HRPT

(hkk) Ge	$2\theta_M=90°$ λ, Å	$2\theta_M=90°$ Effective intensity	$2\theta_M = 120°$ λ, Å	$2\theta_M = 120°$ Effective intensity
311	2.40971	0.64	2.9536	~0.16
400	1.9984[4,5]		2.449[1,3]	0.53
133	1.8324	1	2.246[1,2]	
511	1.5384	1.55	1.886	1
533	1.2183	0.83	1.494	0.88
711	1.1194	0.6	1.372	0.71
733	0.9763	0.34	1.197	0.63
822	0.9419	0.48	1.154	0.70
466			1.044	0.24
866			0.840	0.08

[1]PG(C) filter
[2]1/3 λ contamination
[3](2/3)λ contamination due to double Bragg scattering is avoided by rotating the monochromator along the Q.
[4] 1/2 λ contamination

max Q: from 1.15 to 0.84A





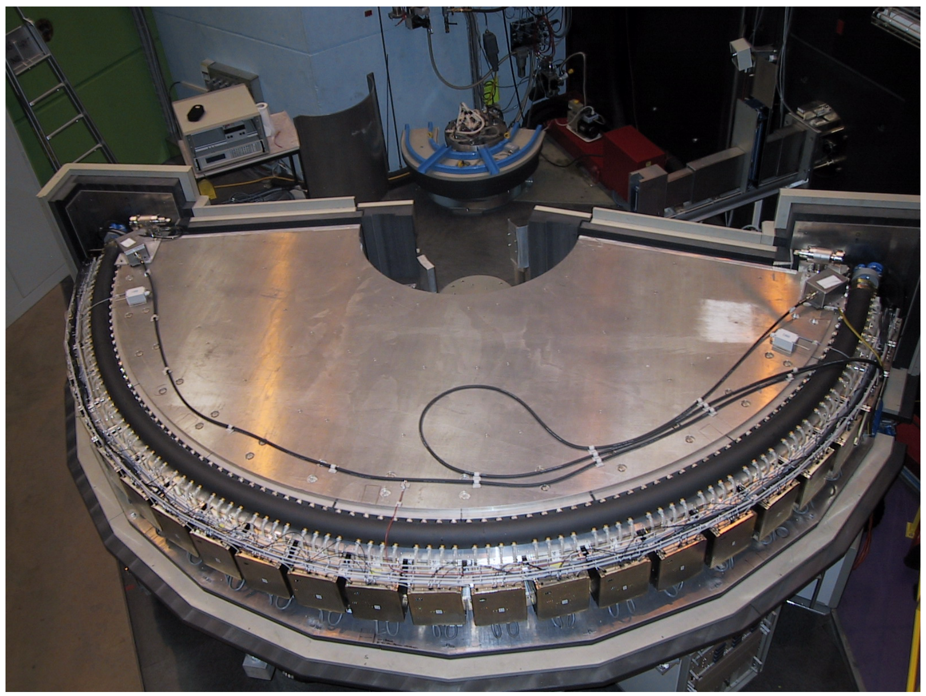
$Mn_x Ga_{1-x} N$, 97, PRB 140102(R) (2018)

Powder ND at SINQ/PSI

HRPT - High Resolution Powder Diffractometer for Thermal Neutrons.

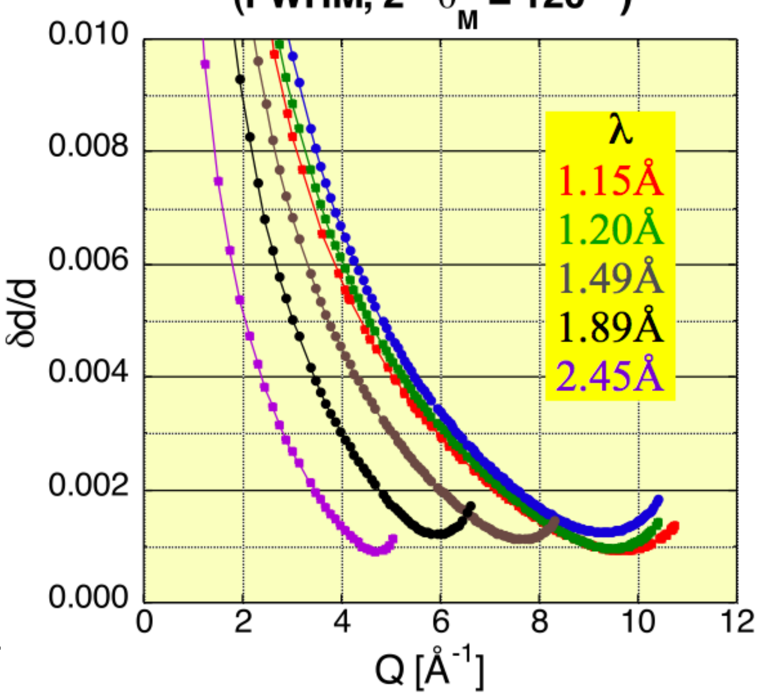
linear detector with 1600 channels, 0.1°

Responsible: Vladimir Pomjakushin, Denis Sheptyakov



HRPT RESOLUTION FUNCTIONS

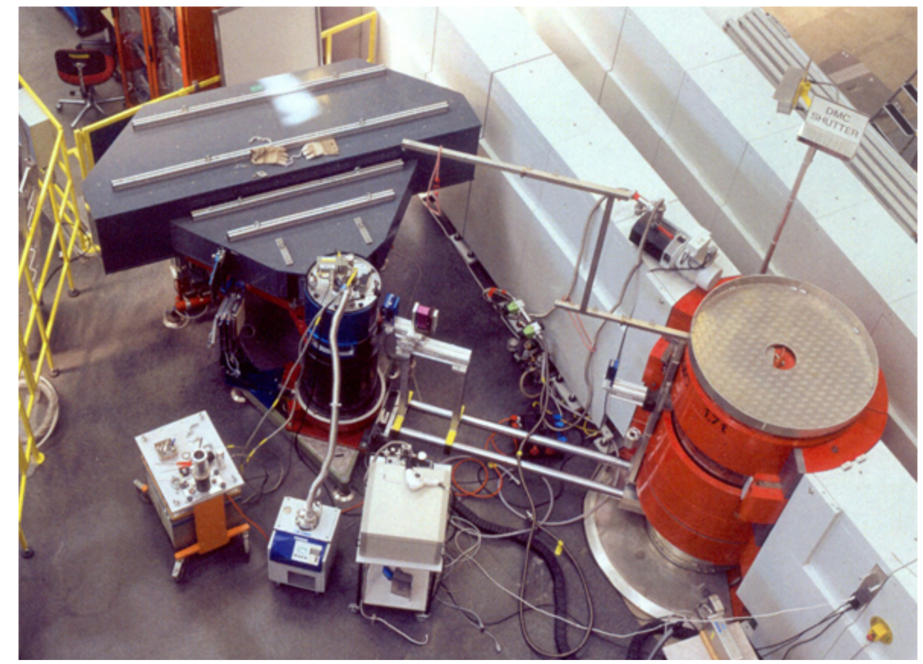
(FWHM, 2 θ_M = 120 °)



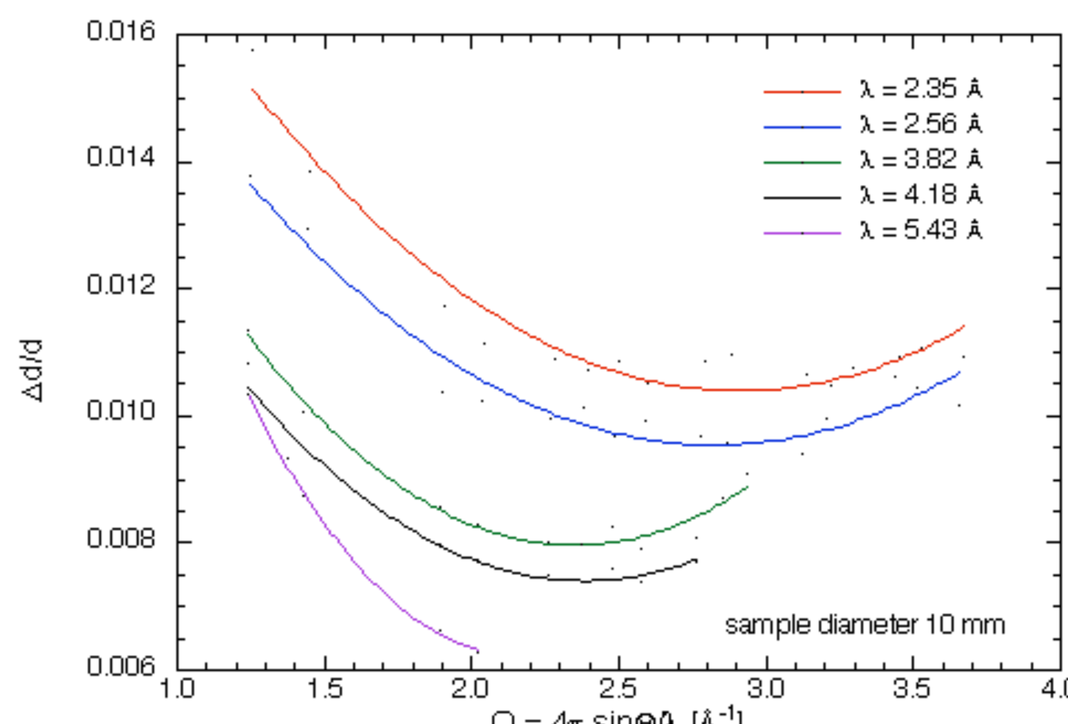
DMC - cold neutron powder diffractometer

linear detector with 400 channels, 0.2°

Responsible: Lukas Keller, Matthias Frontzek



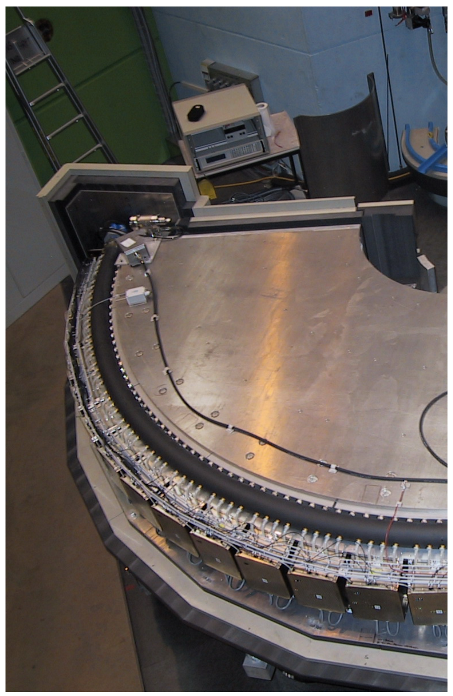
DMC: experimental resolution functions Δd/d (Q,λ)

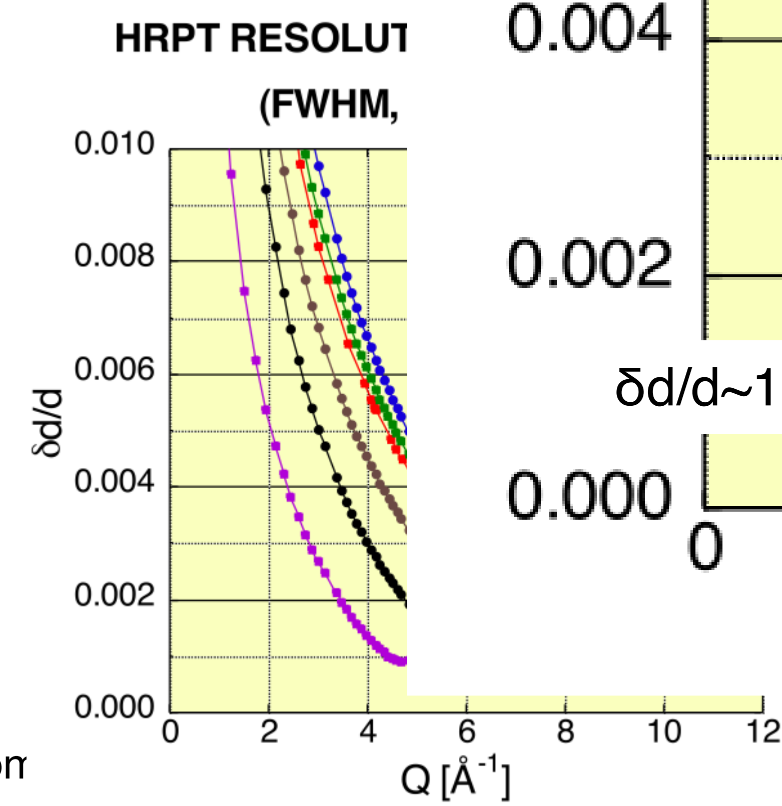


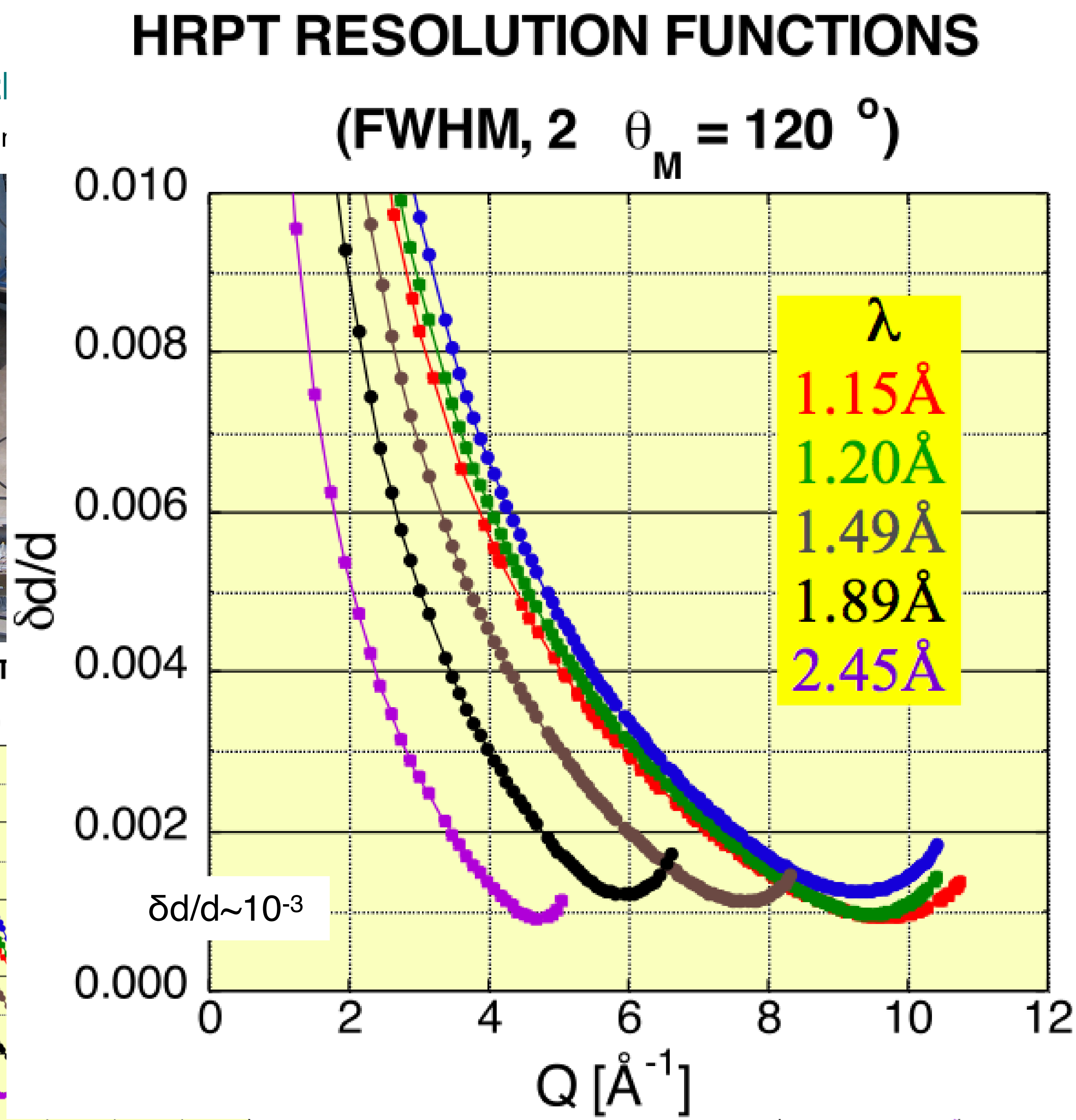
Powder ND at SINQ/PSI

HRPT - High
Diffractometer for
linear detector wit

Responsible: Vladimir Po

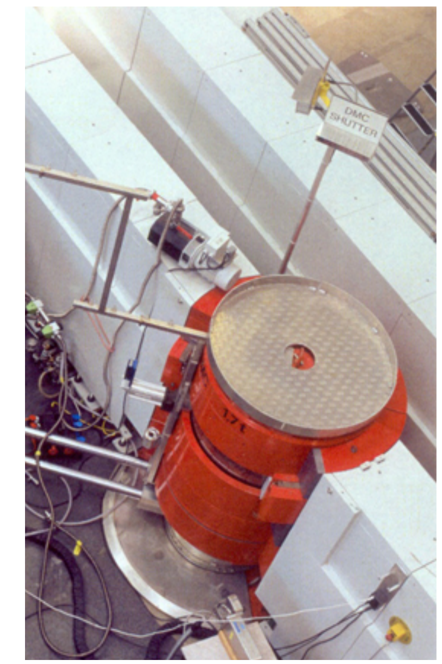


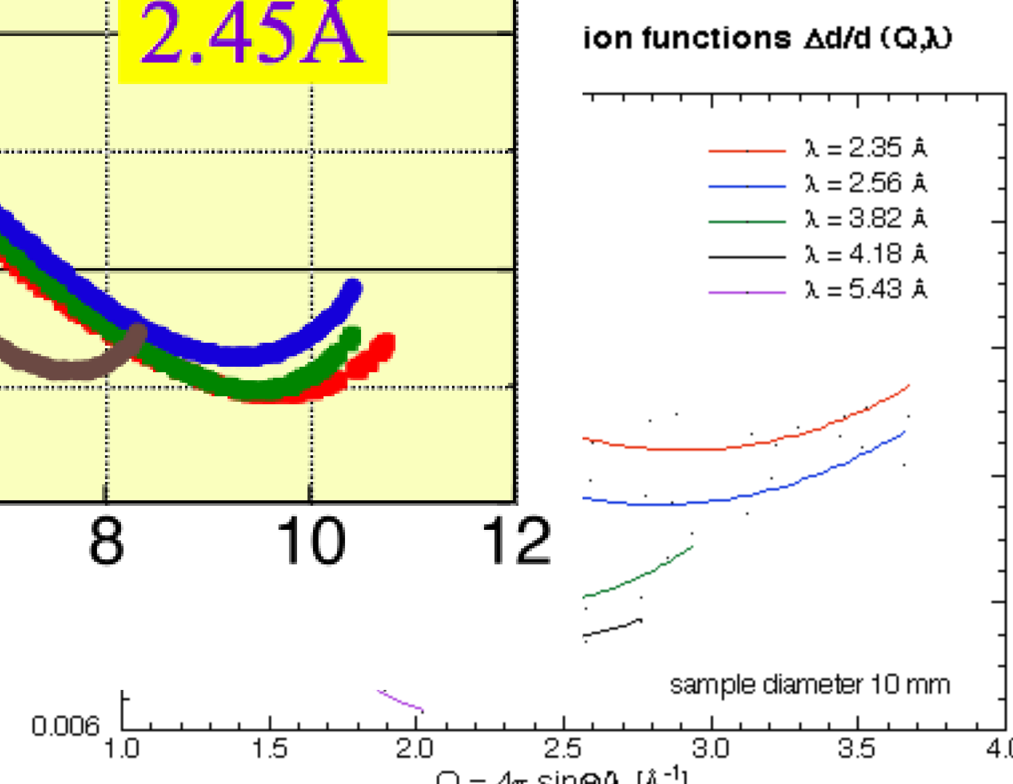




owder diffractometer
annels, 0.2°

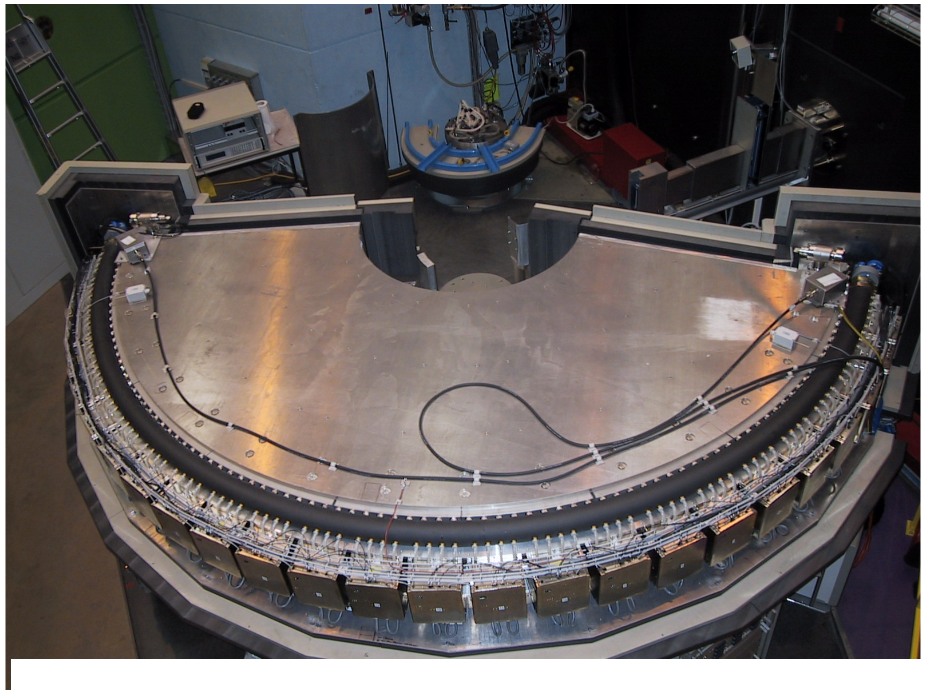
as Frontzek

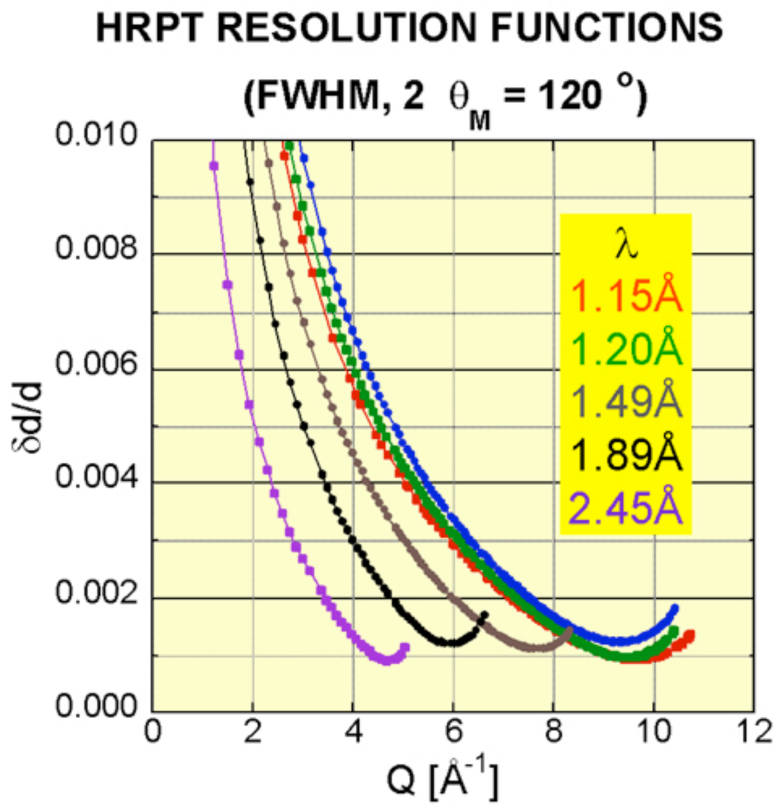




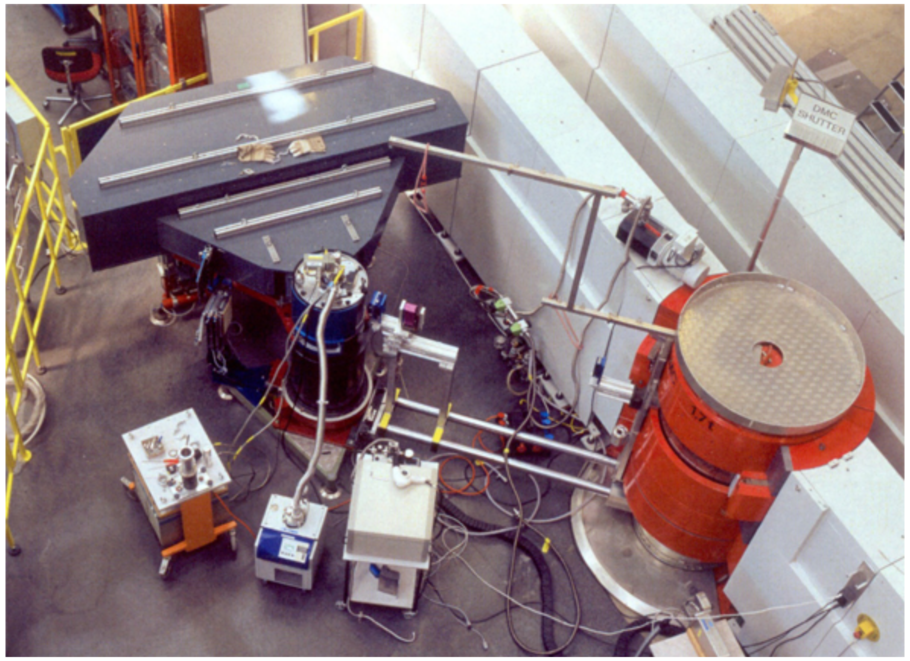
Powder ND at SINQ/PSI

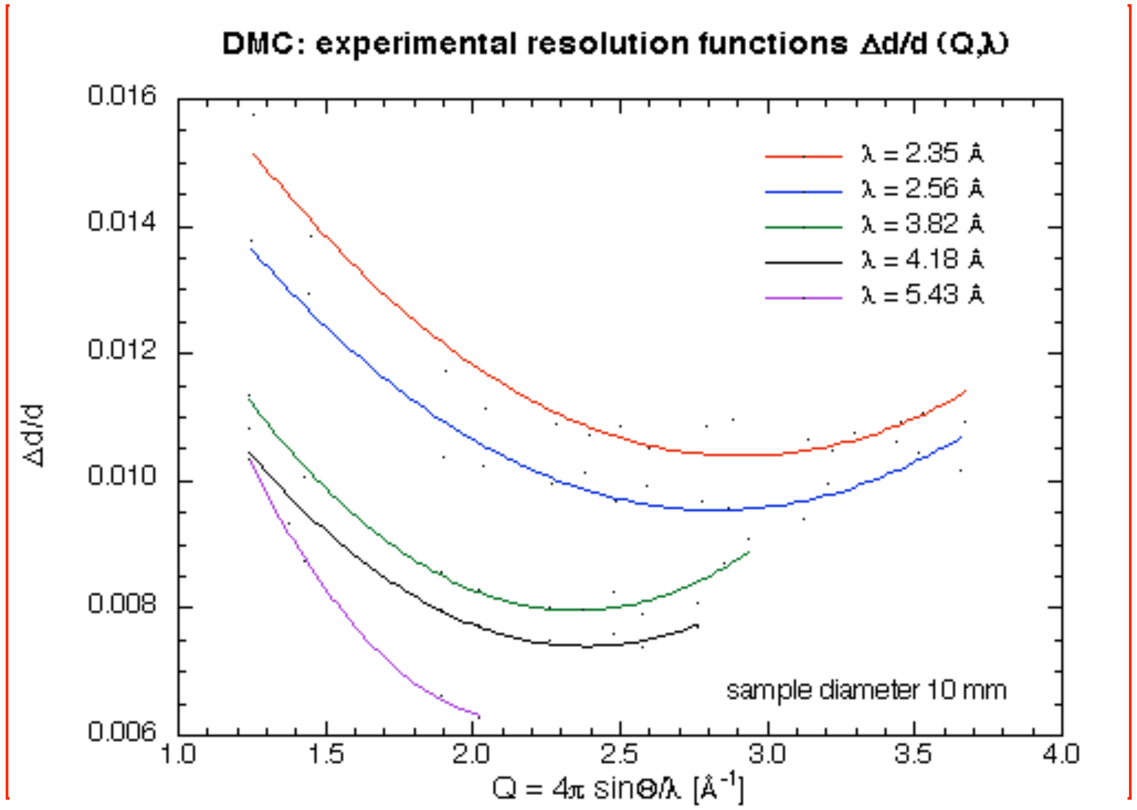
HRPT - High Resolution Powder Diffractometer for Thermal Neutrons at SINQ





DMC - cold neutron powder diffractometer

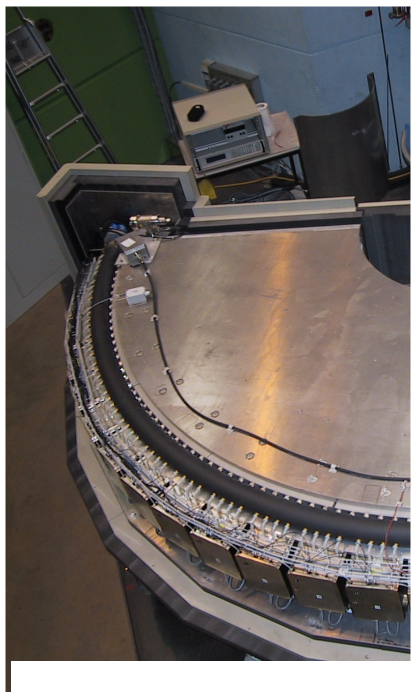


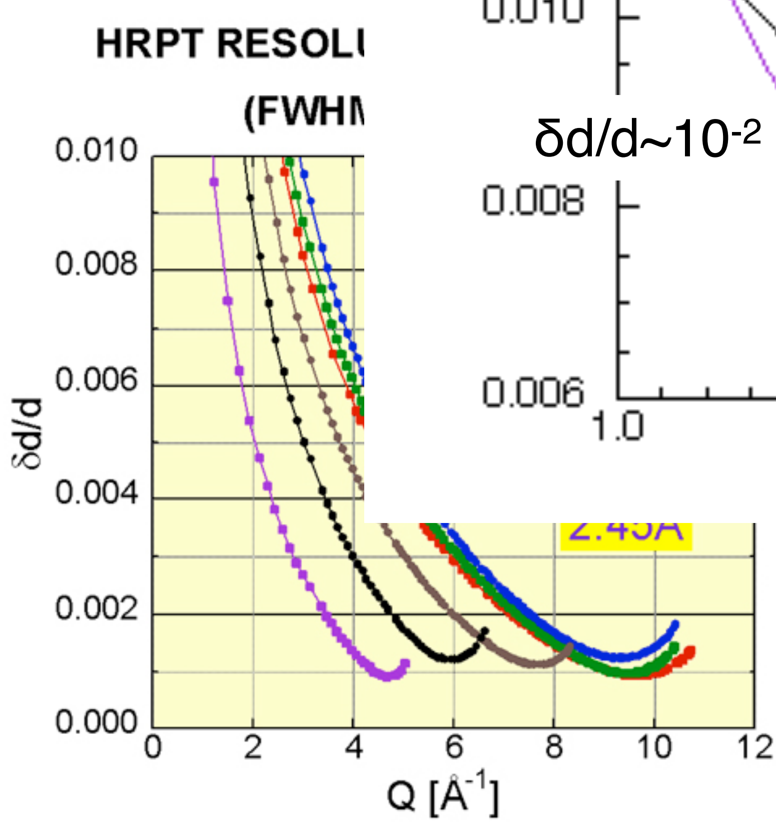


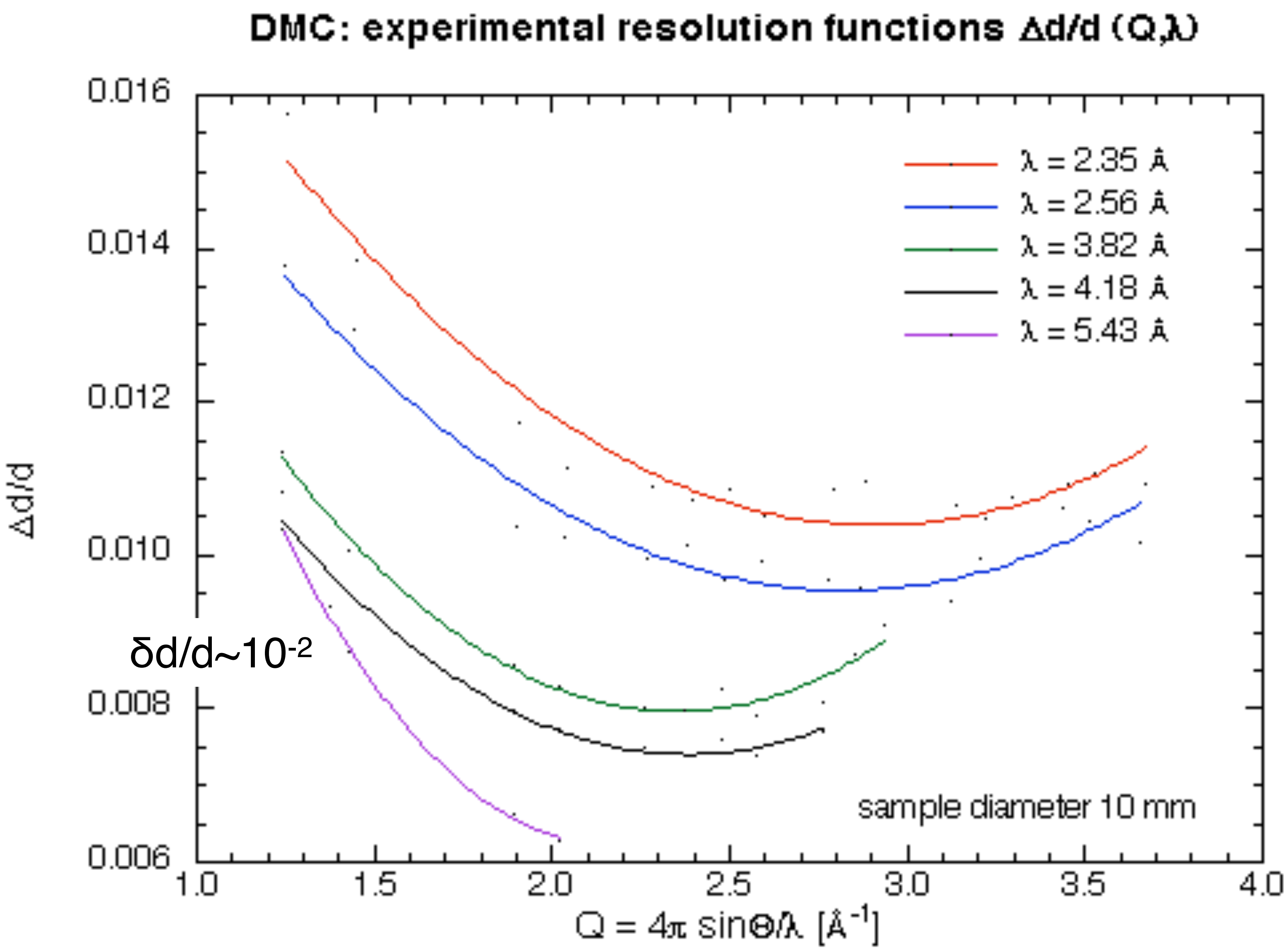
Powder ND at SINQ/PSI

HRPT - High Resolution Powder Diffractometer for Thermal Neutrons at SINQ

DMC - cold neutron powder diffractometer





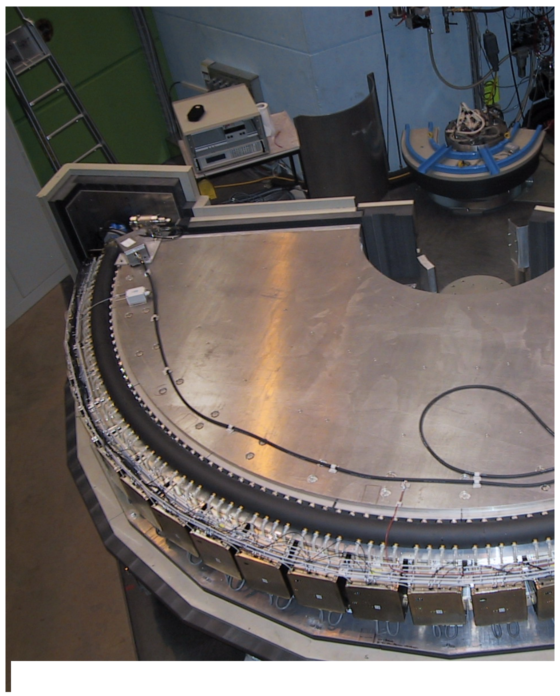


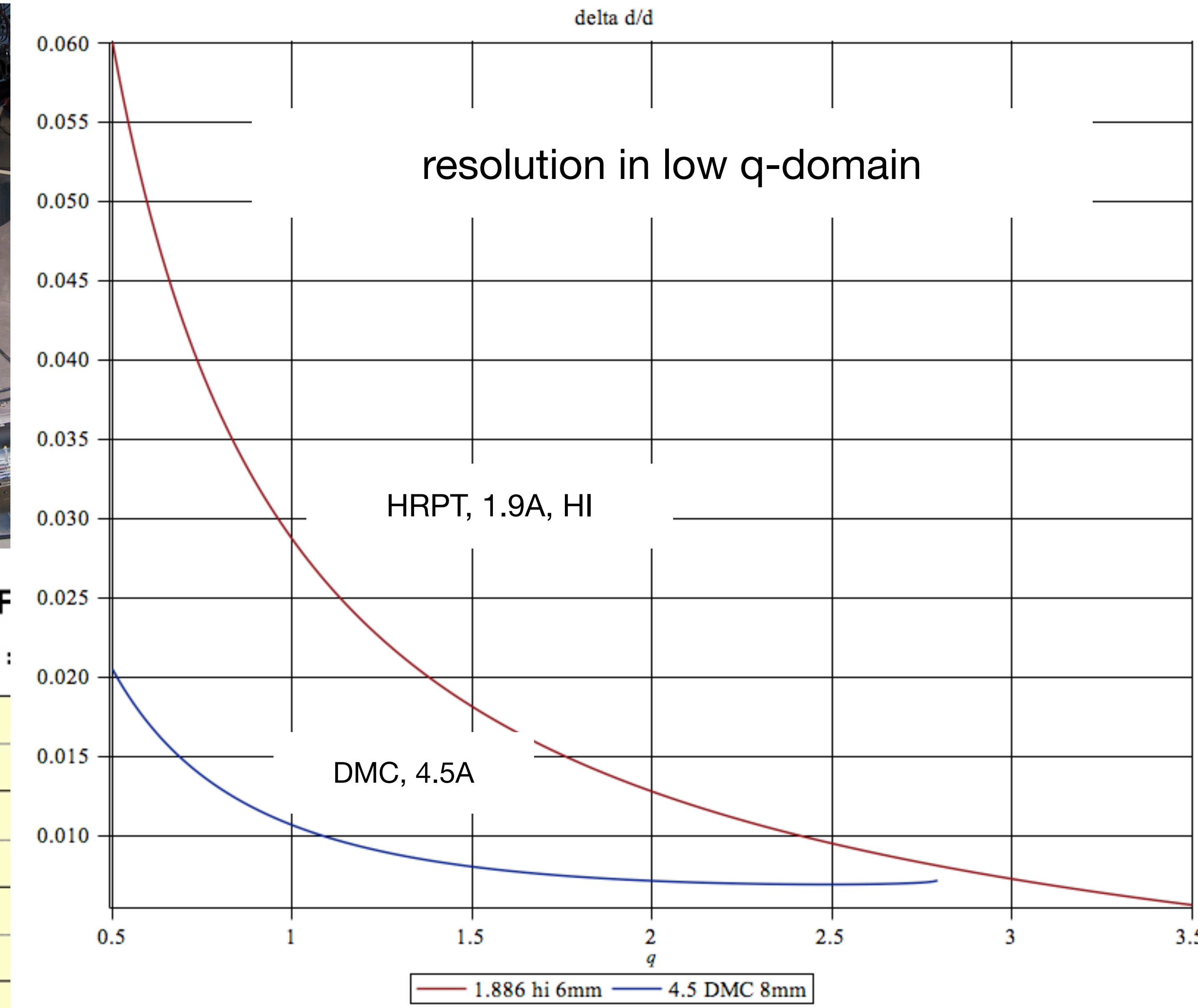
$\delta d/d \sim 10^{-2}$

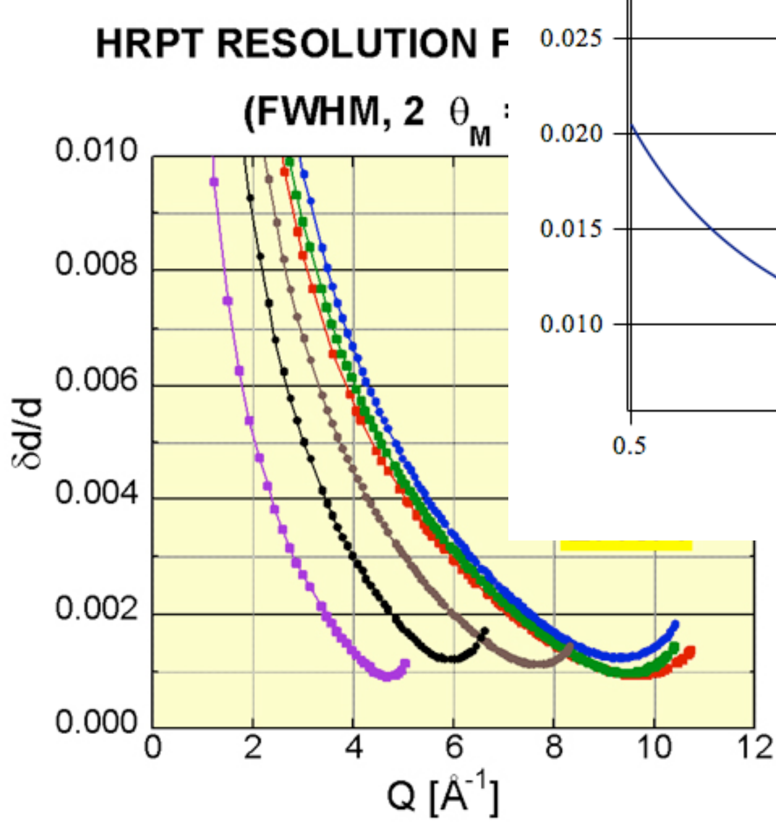
Powder ND at SINQ/PSI

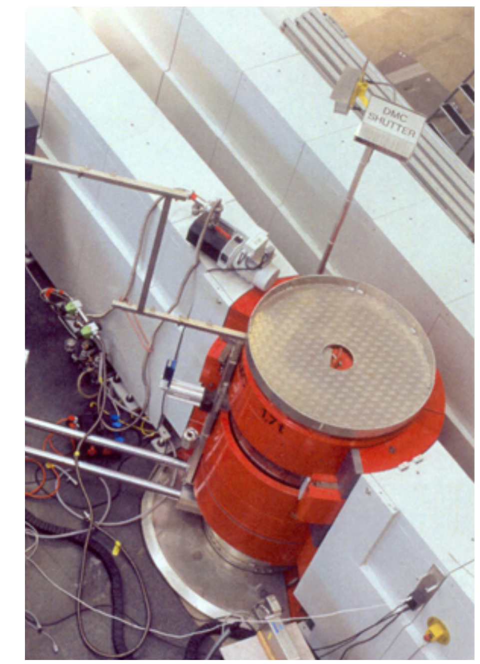
HRPT - High Resolution Powder Diffractometer for Thermal Neutrons at SINQ

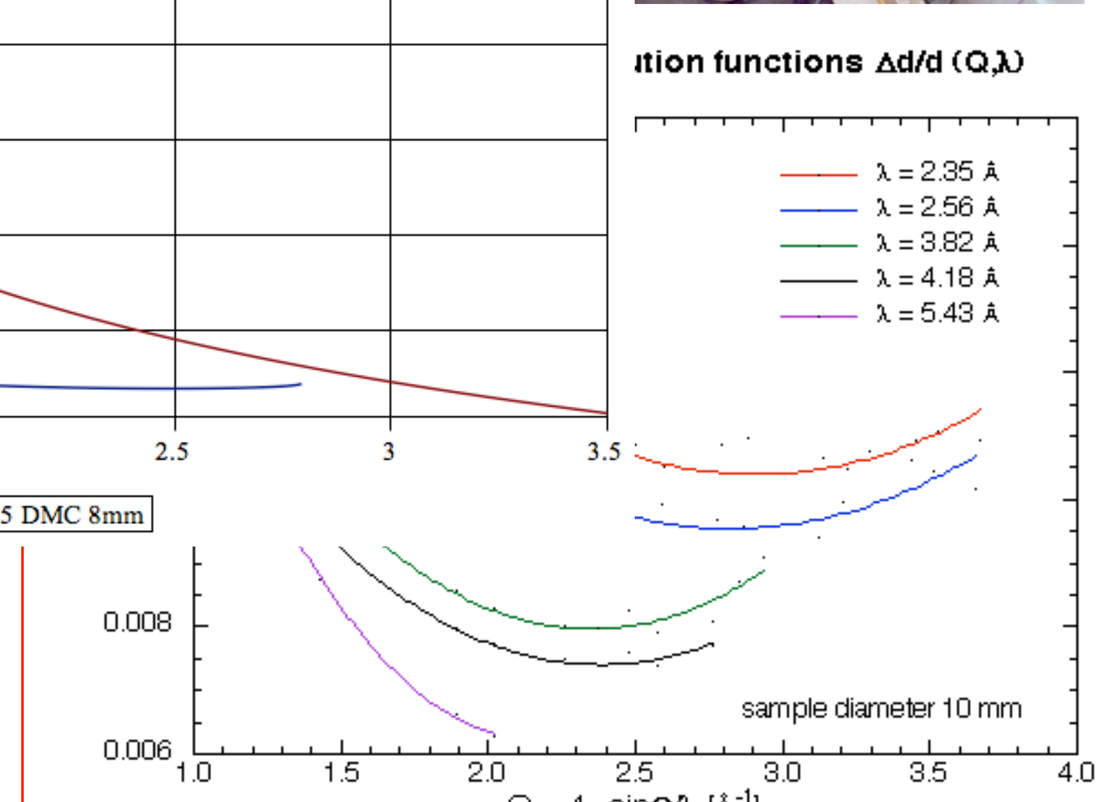
DMC - cold neutron powder diffractometer





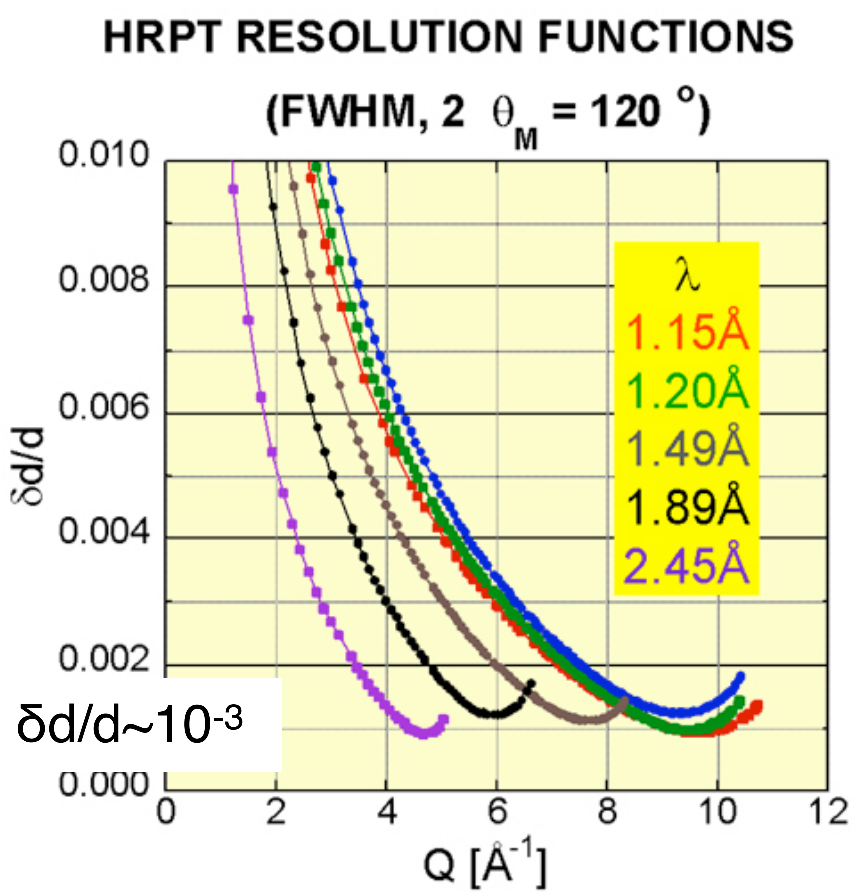


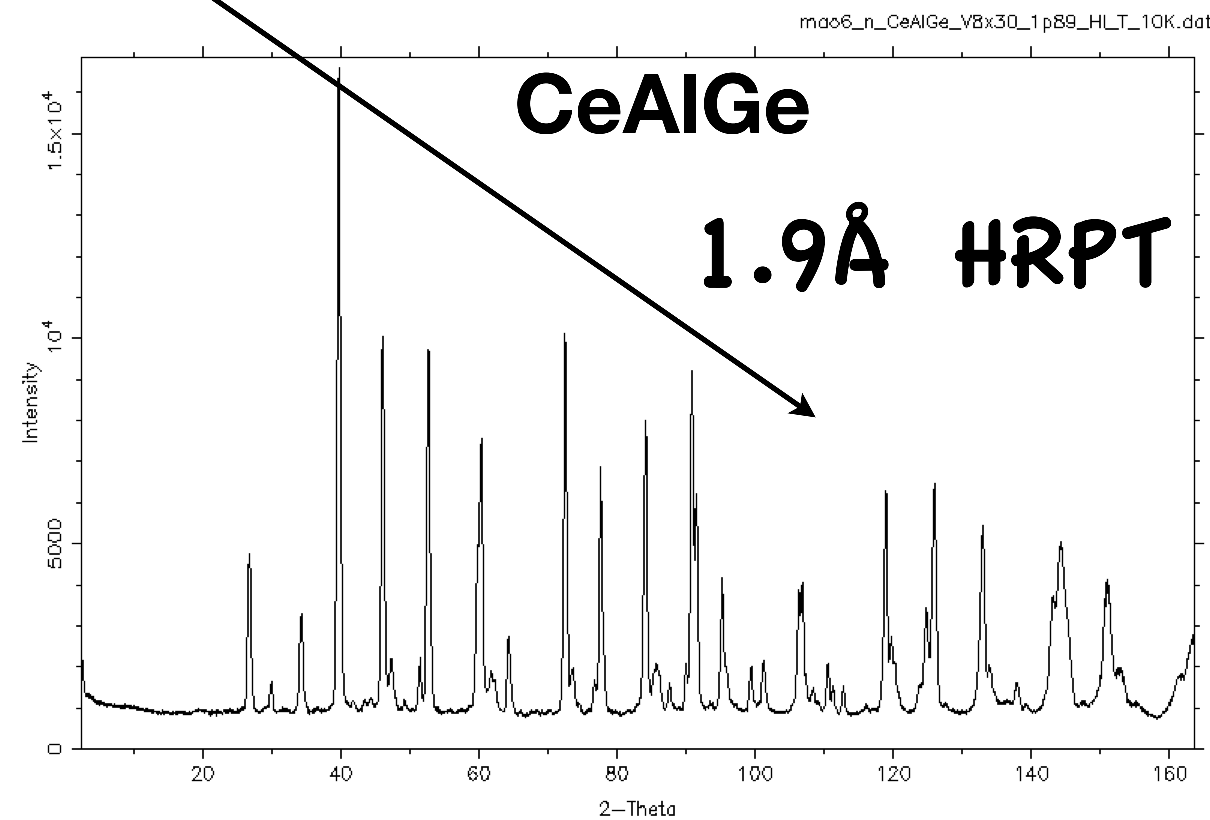


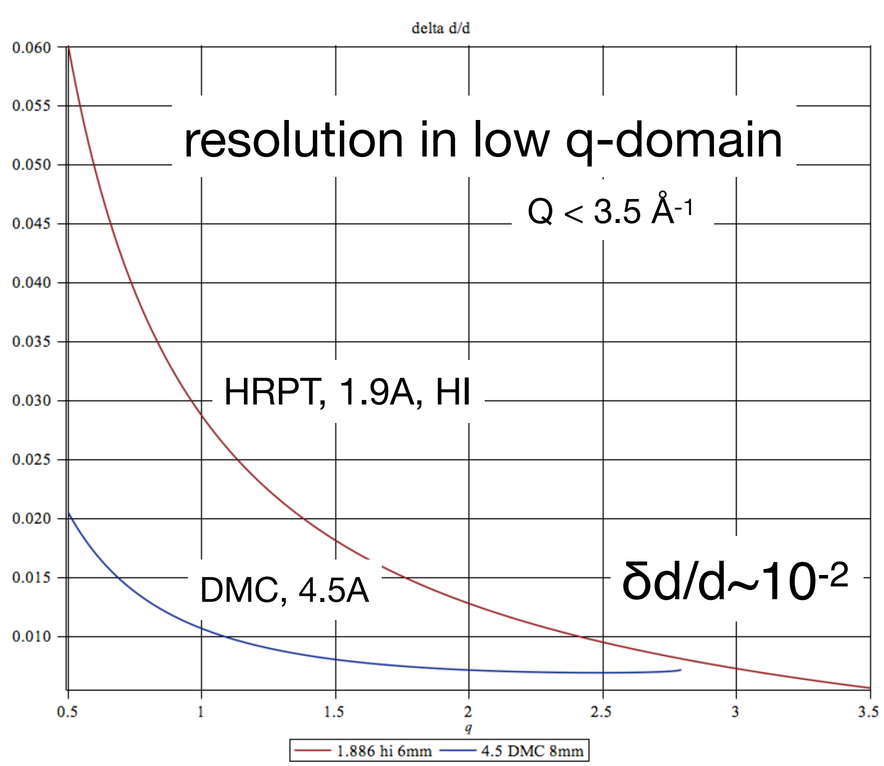


Complementarity 1.9Å HRPT and 4.5Å DMC

excellent resolution and high Q-range



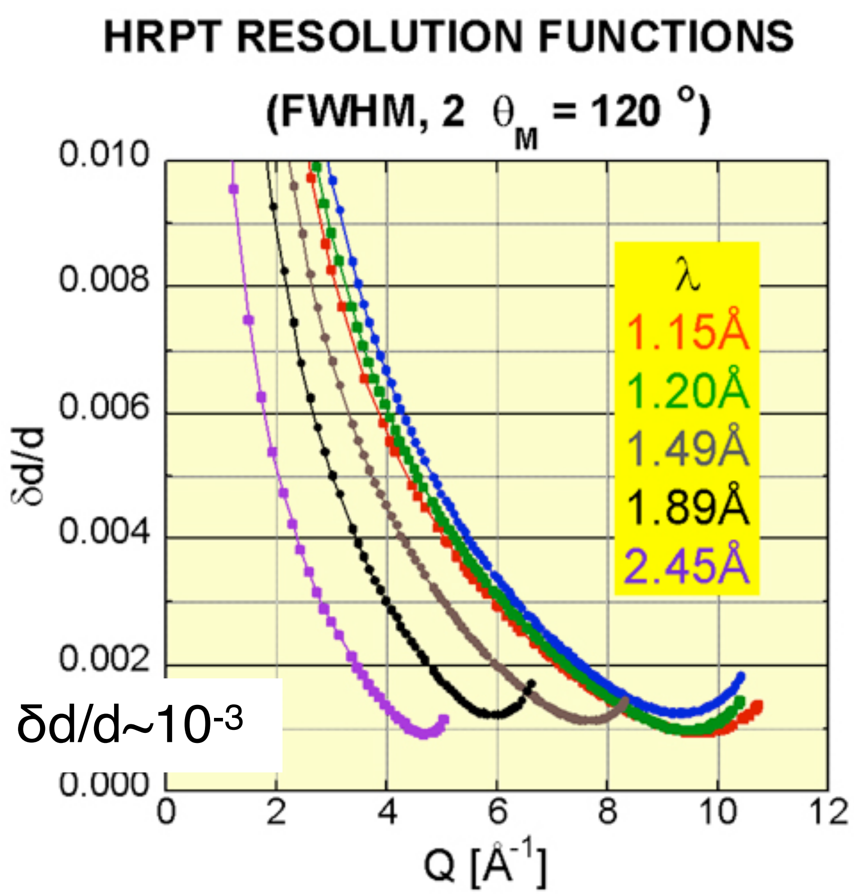


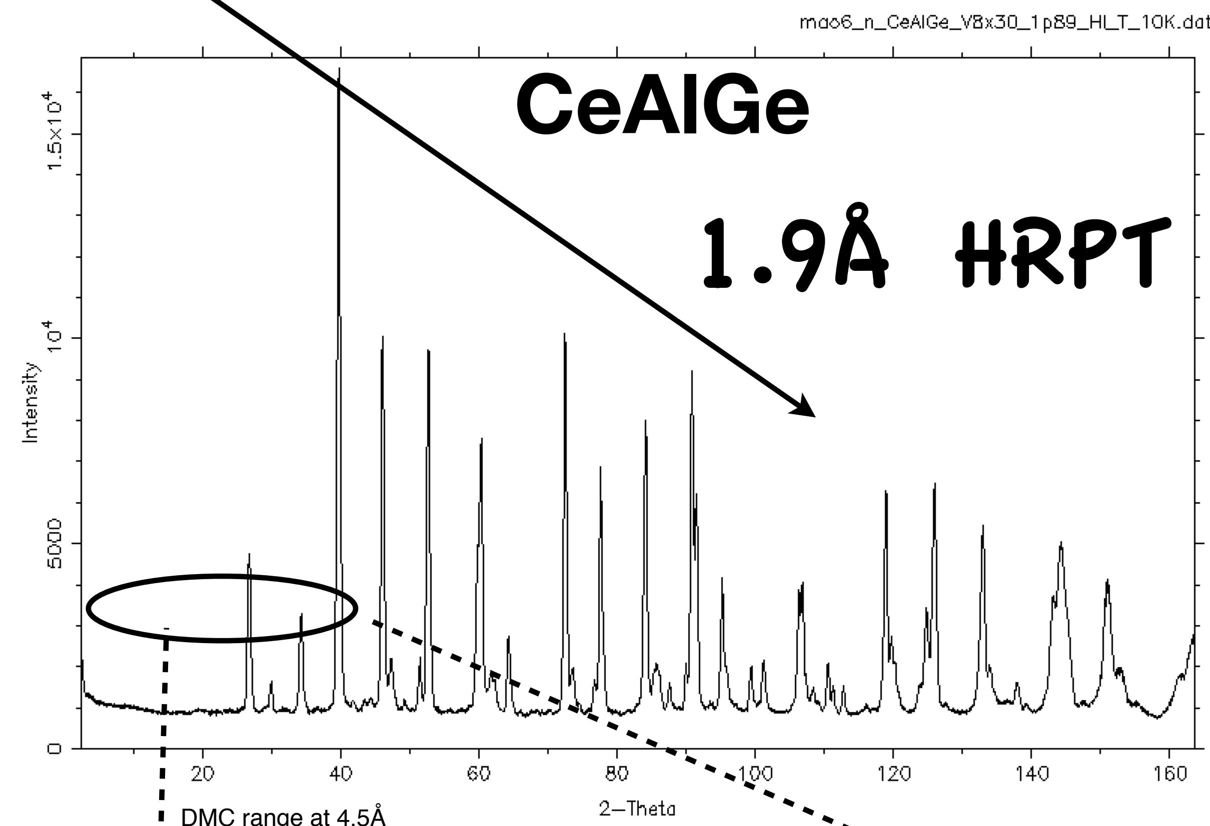


Example of HRPT & DMC complementarity: resolution&intensity issue

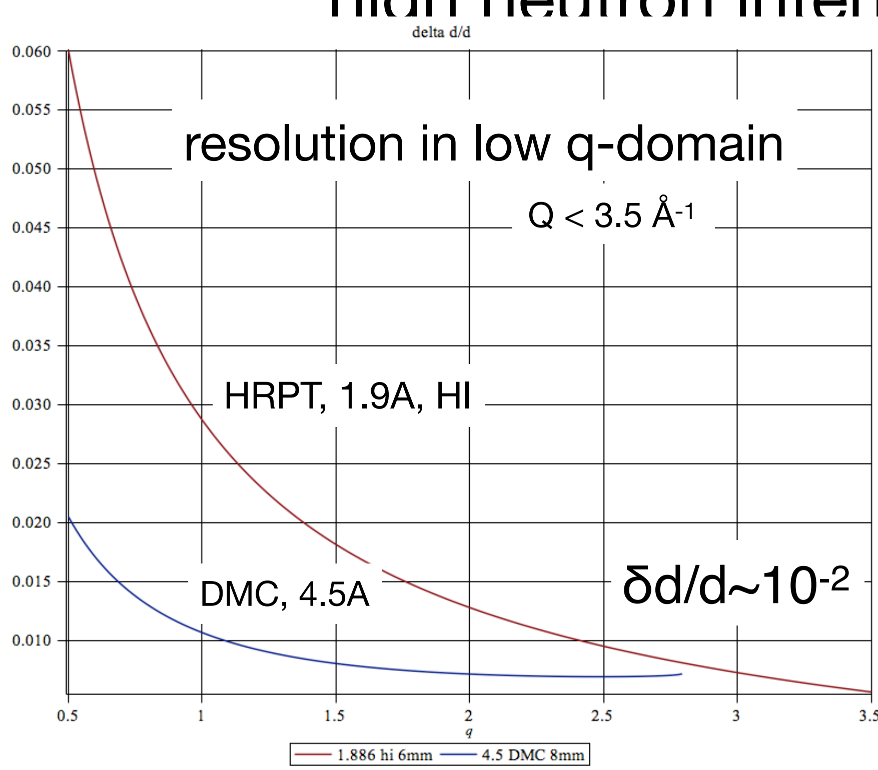
Complementarity 1.9Å HRPT and 4.5Å DMC

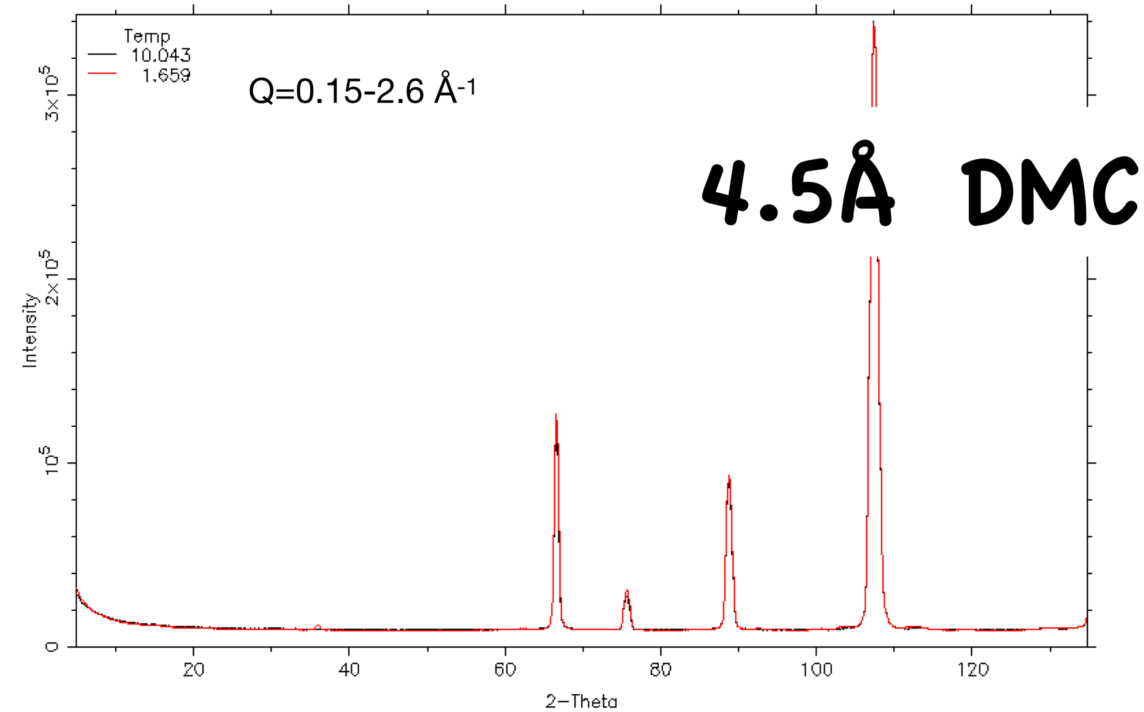
excellent resolution and high Q-range





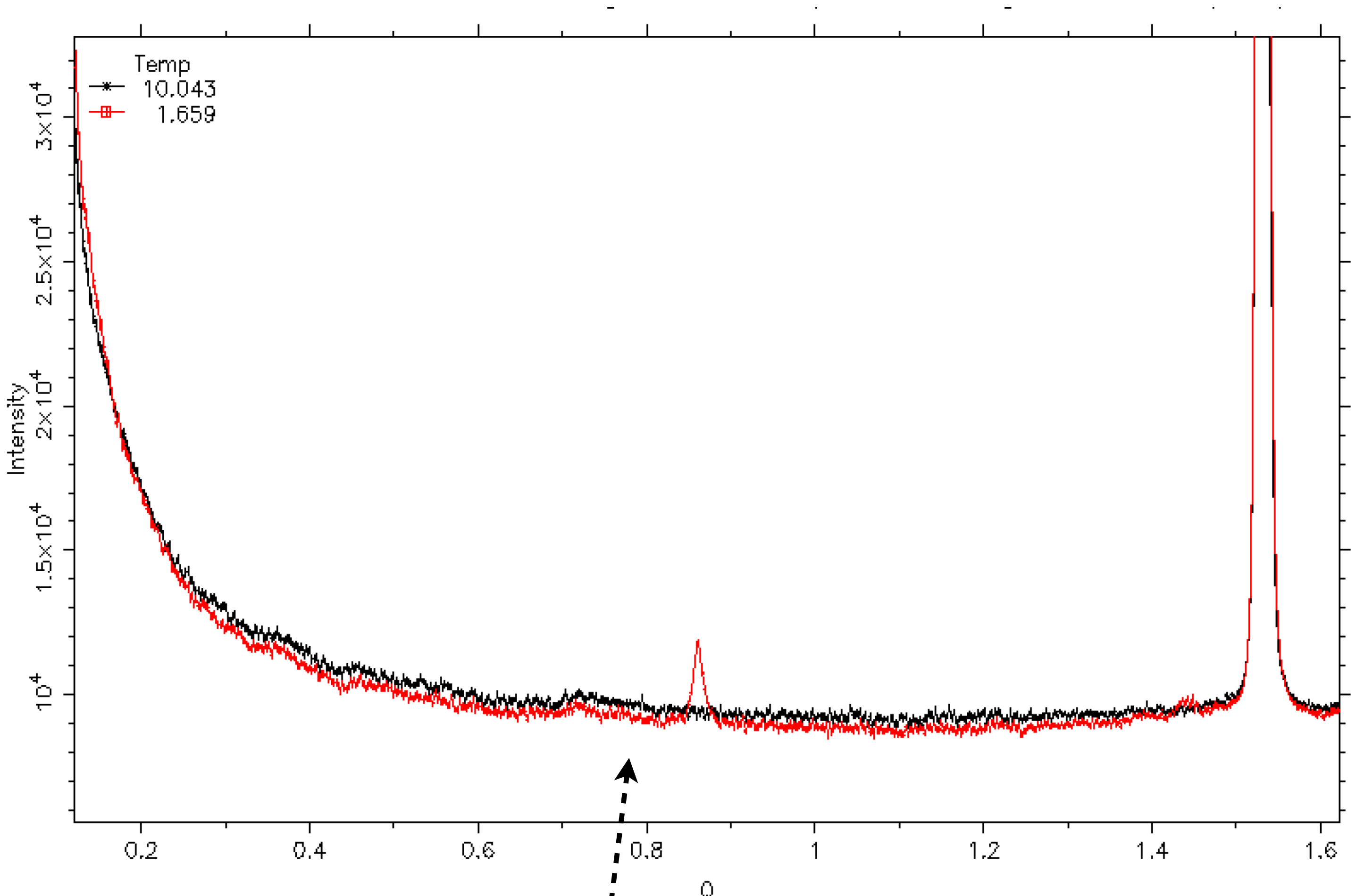
high neutron intensity and excellent resolution at low Q

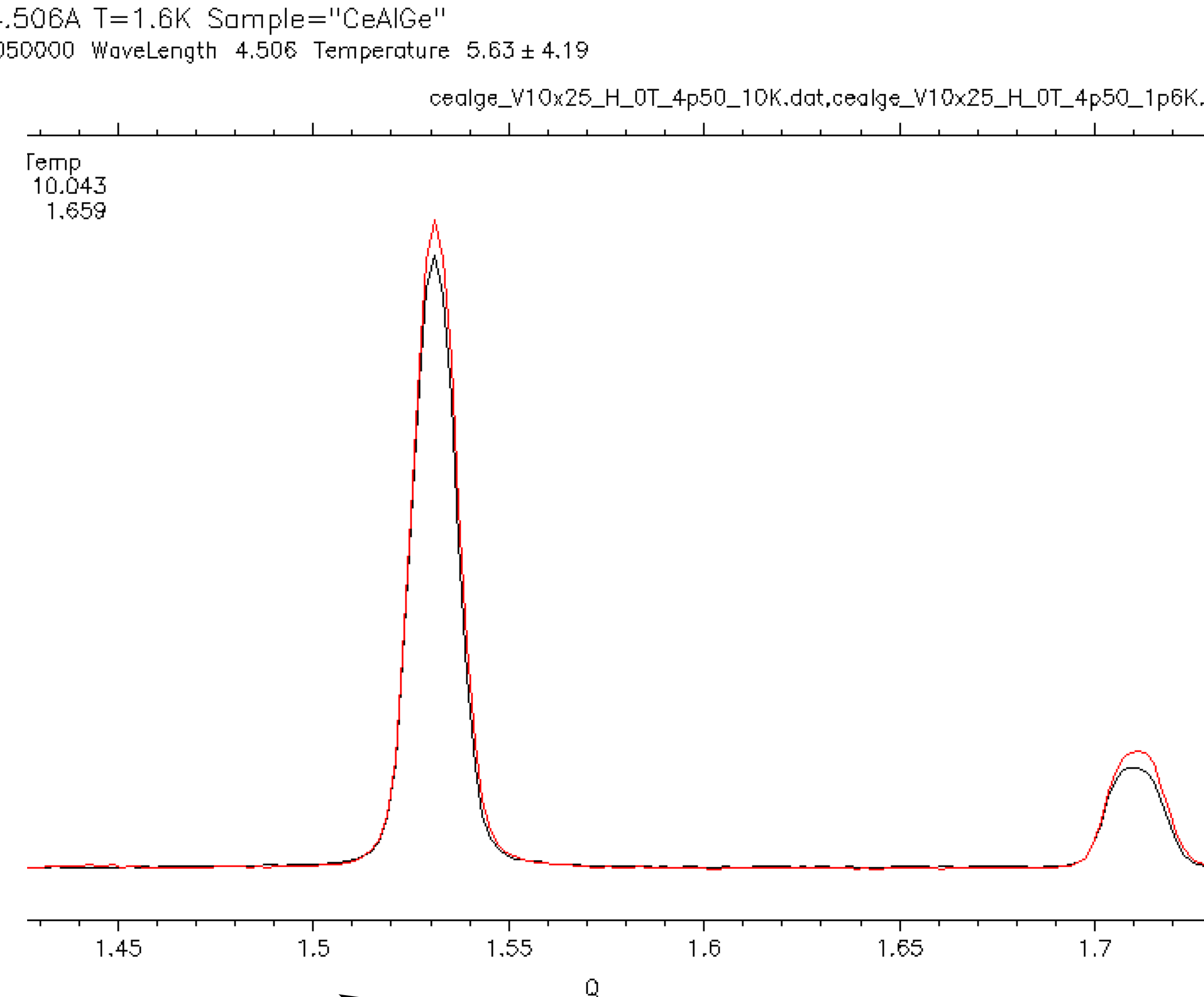


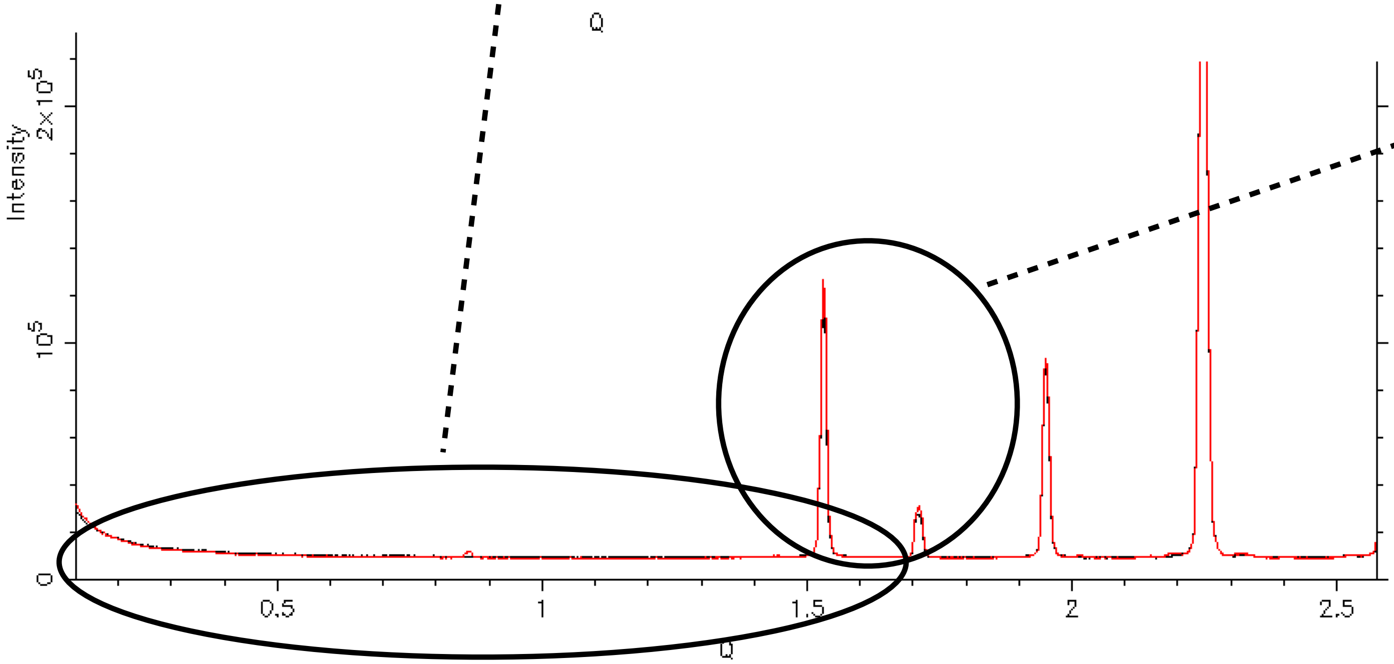


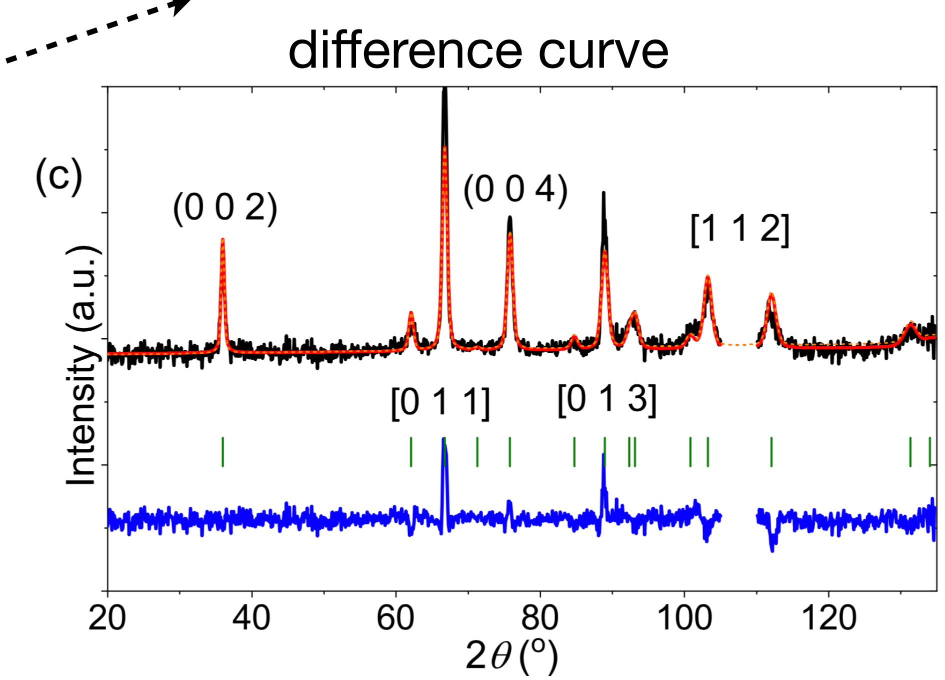
powder diffraction patterns in CeAlGe skyrmion structure

4.5Å DMC



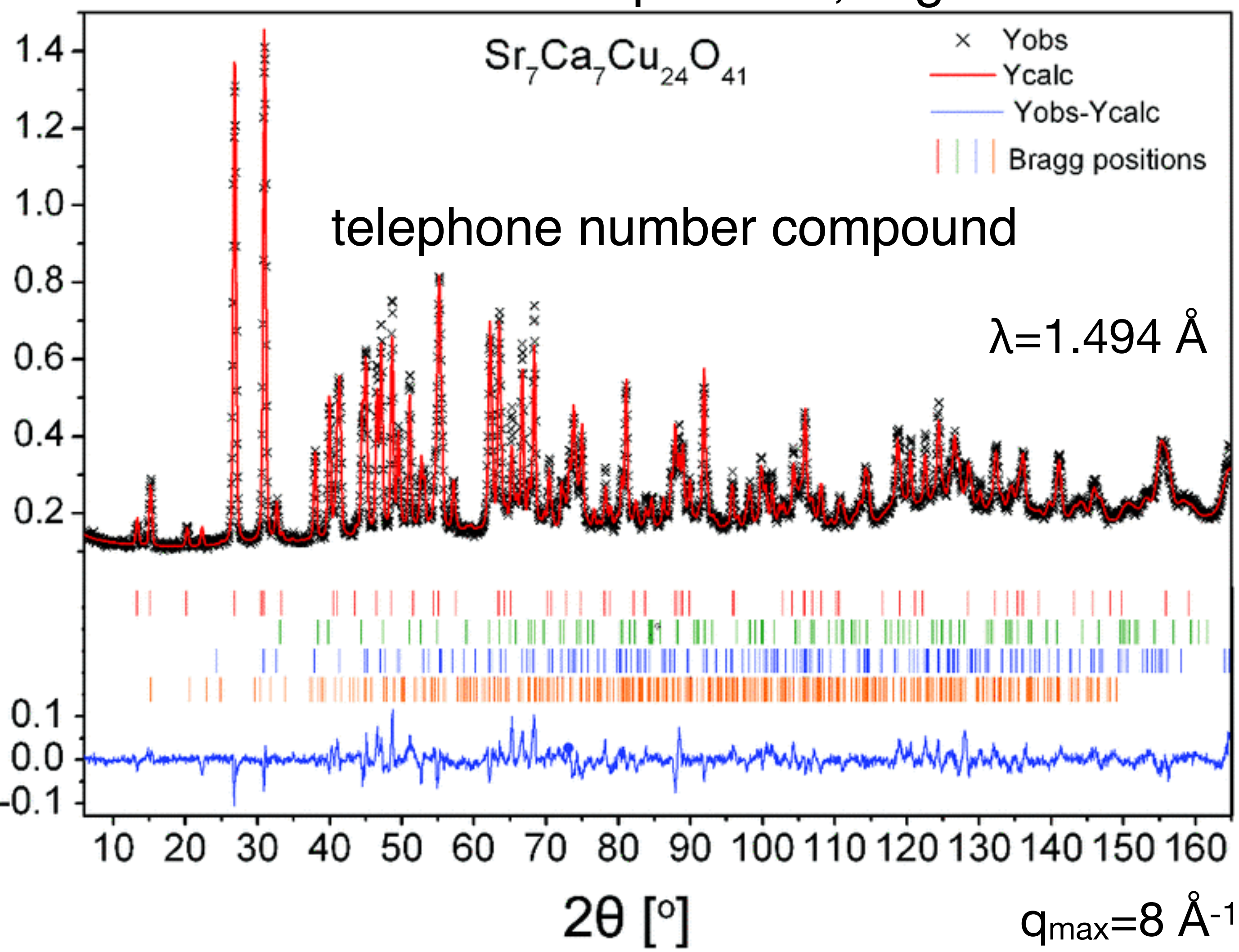


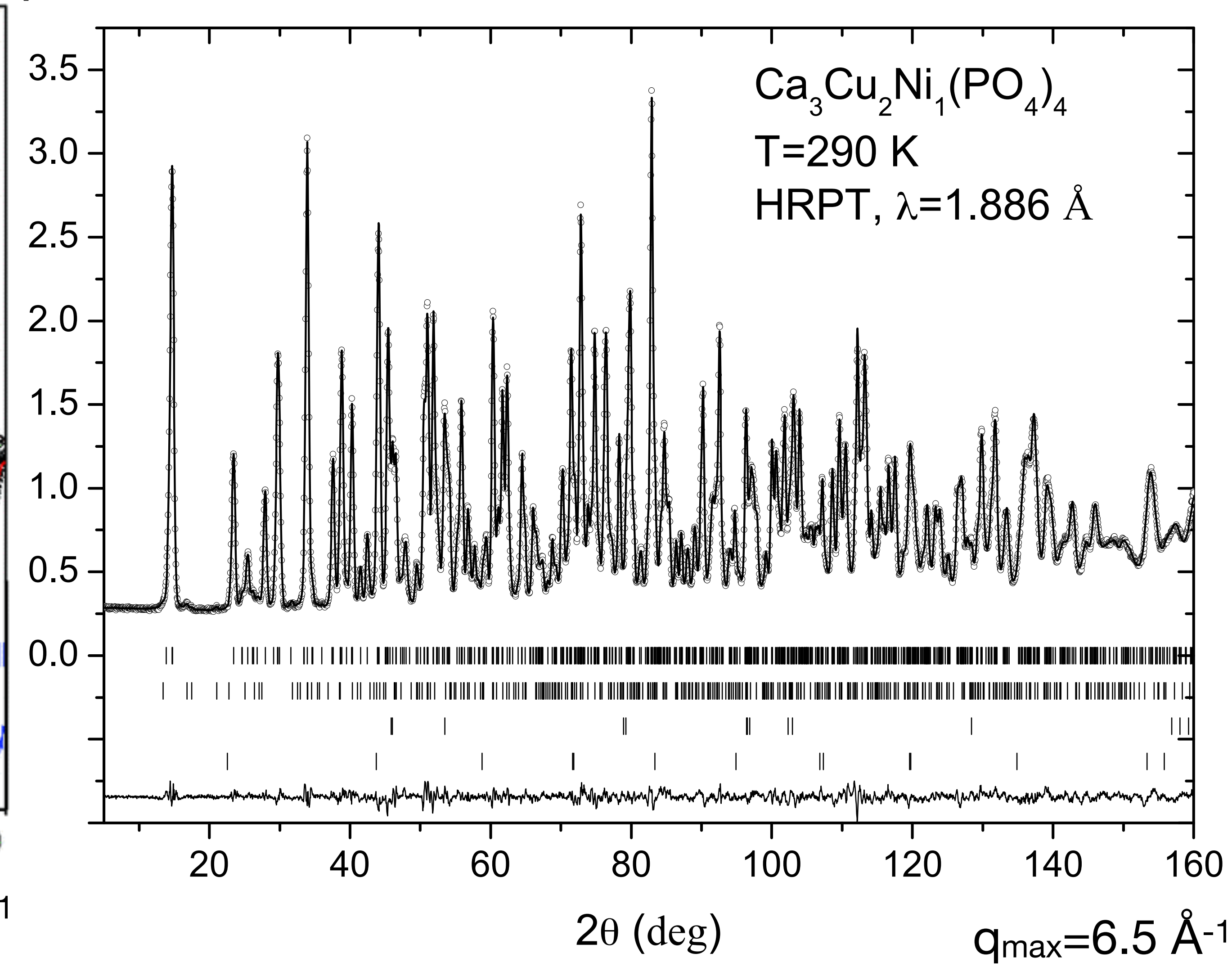




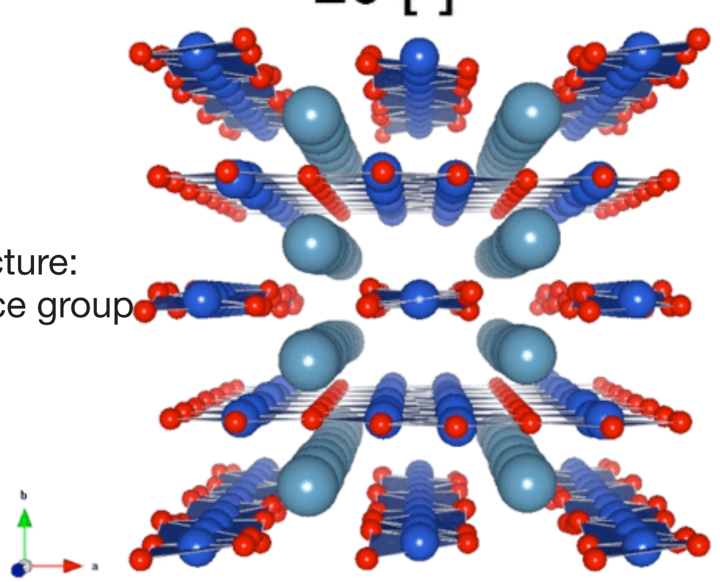
Q-range/resolution in powder diffraction. Peak overlap.

Diffraction patterns, High resolution powder diffractometer HRPT @ SINQ

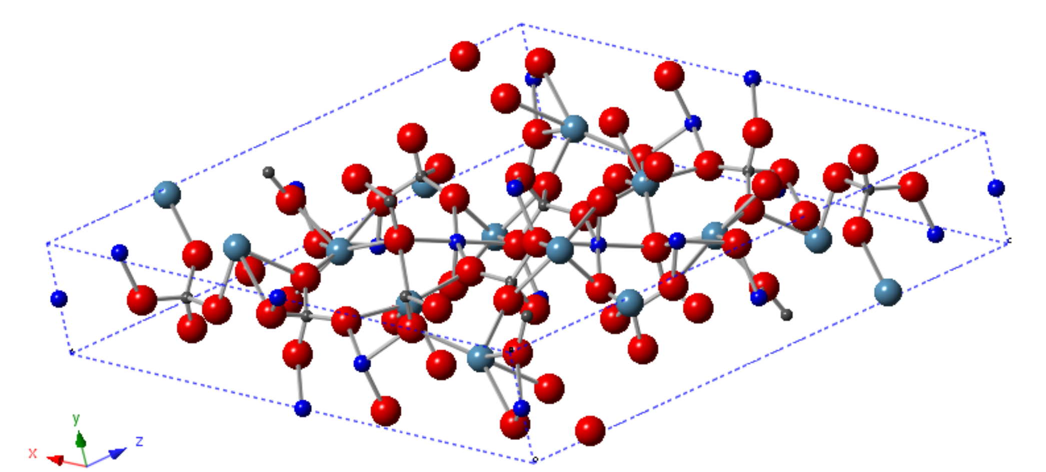




Modulated structure:
3D+1 superspace group
Xmmm(00γ)ss0



V. Pomjakushir

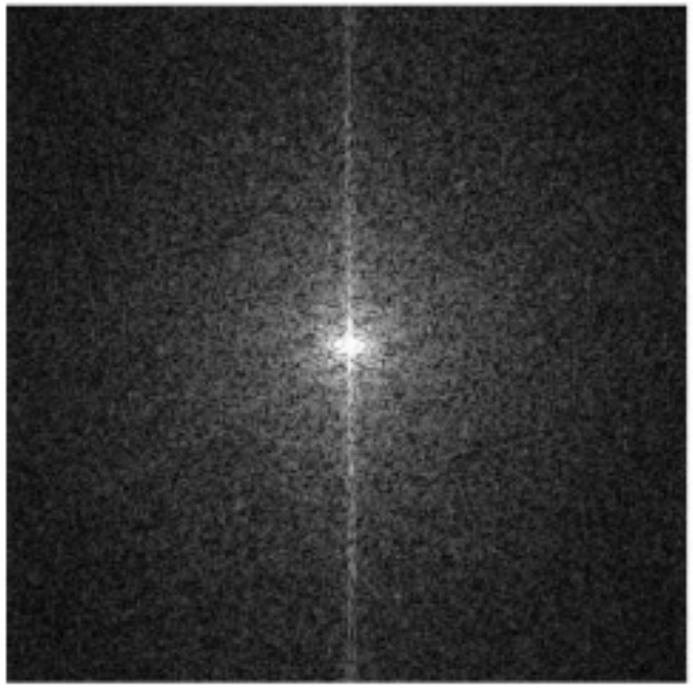


Q-range limitation – image quality in Fourier transform

min $\delta r \sim \pi / Q_{max}$

Object

$$\mathbf{b}(r) \sim \int_0^\infty e^{-iqr}\, \mathbf{f(q)dq}$$



Fourier image

$$\mathbf{f(q)} \sim \int e^{iqr}\, \mathbf{b}(r)\mathbf{d}r$$

Structure factor

$$F(\mathbf{q}) = \sum_j b(\mathbf{r}_j) \exp(i\mathbf{q}\mathbf{r}_j)$$

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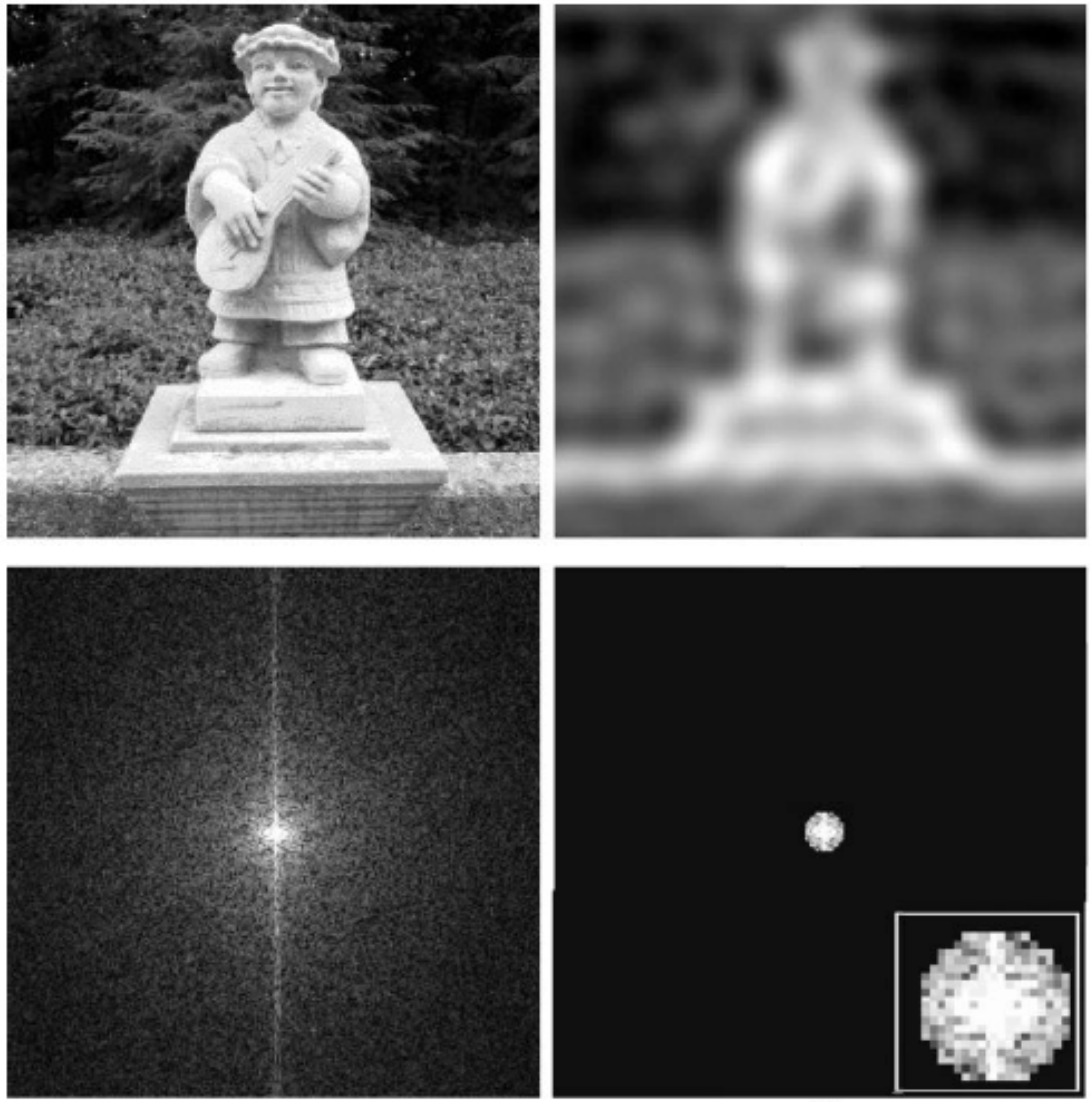


$$b(r) \sim \int_0^{q_{max}} e^{-iqr} f(q)dq$$

Fourier image

$$f(q) \sim \int e^{iqr} b(r)dr$$

Fourier image without high q

Structure factor

$$F(\mathbf{q}) = \sum_j b(\mathbf{r}_j)\exp(i\mathbf{q}\mathbf{r}_j)$$

Limitations on maximal unit cell volume (number of atoms) in powder neutron diffraction

Volumes up to 1000-2000Å, about 100-200 atoms, concentration 0.08-0.1 at/A^3

bond lengths accuracy ~0.001Å

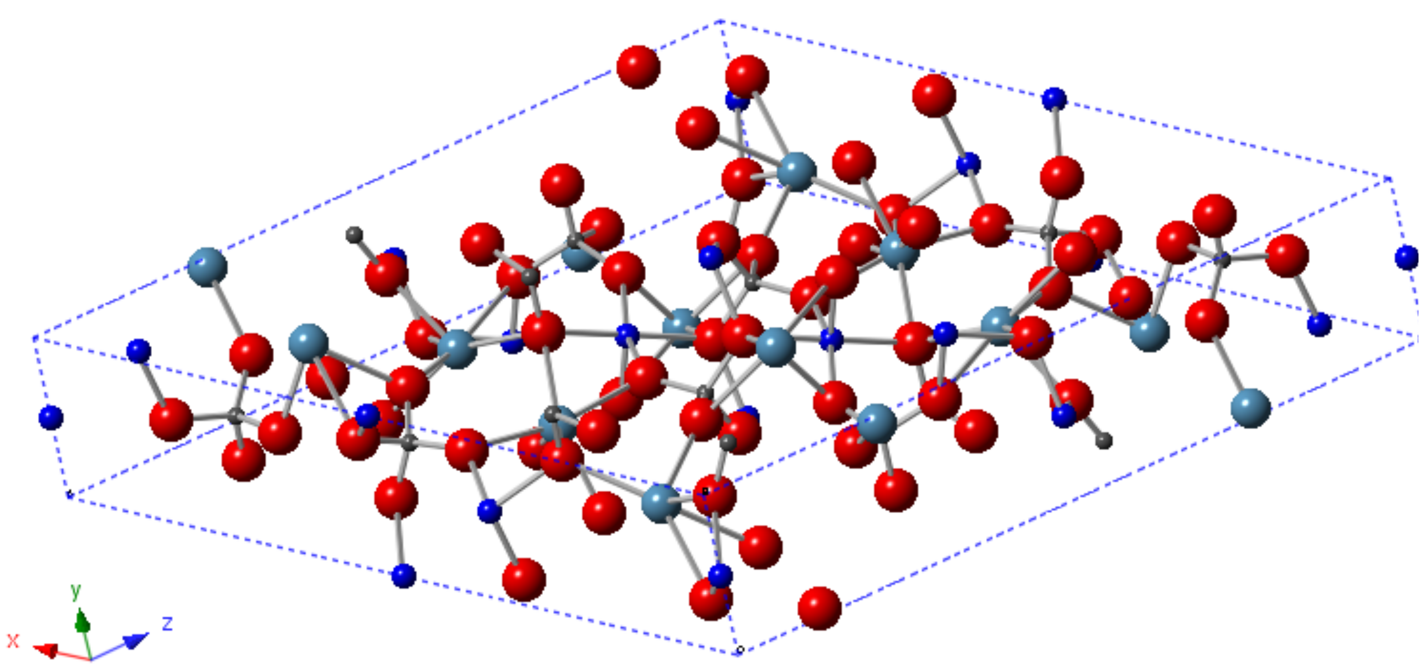
$Ca_3Cu_xNi_{2-x}(PO_4)_4$
Quantum spin trimer

18x5x18Å, V=1300Å^3

C2/c sp.gr.

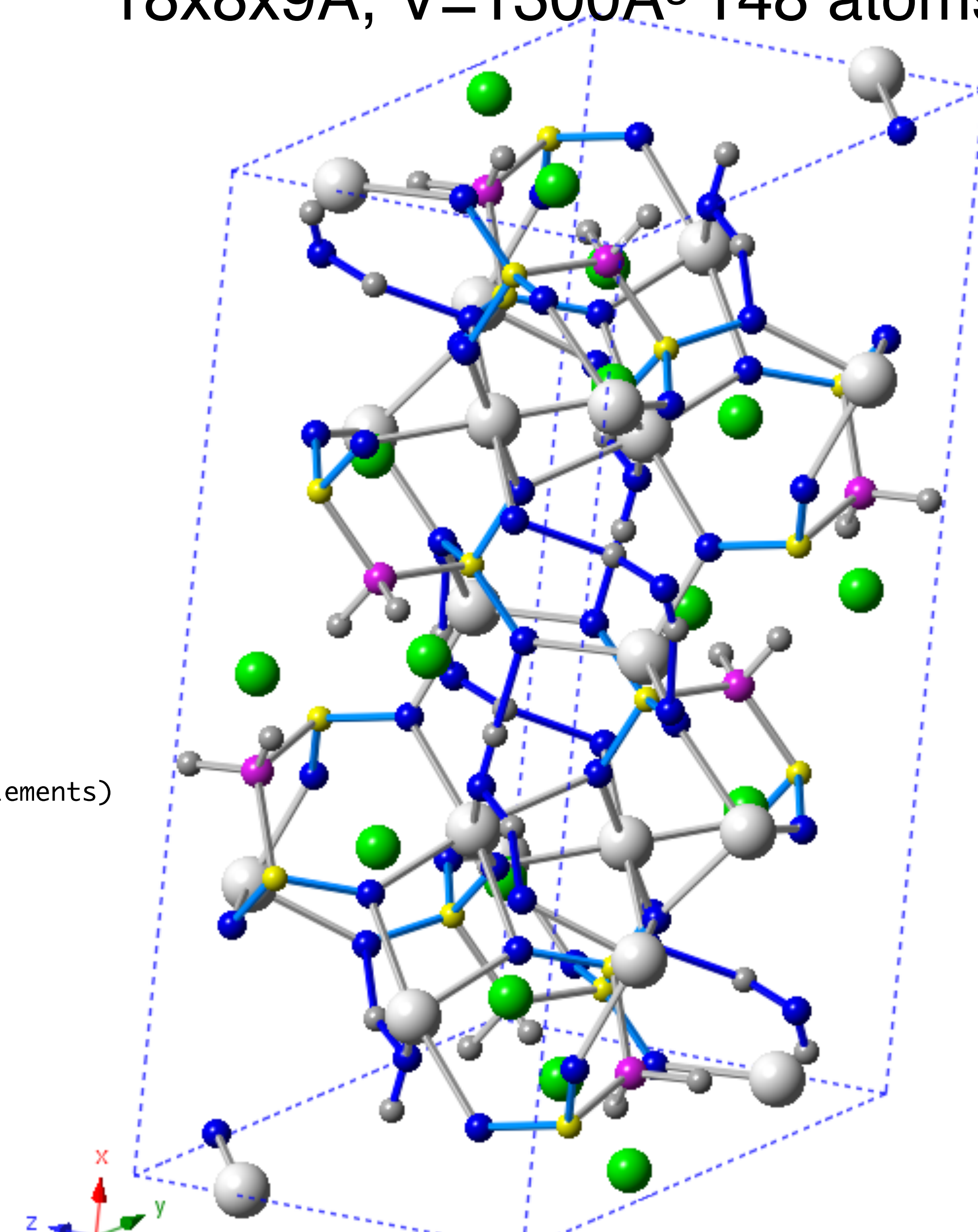
$(Li_{1.4}Fe_{6.8}[CH_2(PO_3)_2]_3[CH_2(PO_3)(PO_3H)]\cdot 4H_2O)$
Lithium Iron Methylenediphosphonate

18x8x9Å, V=1300Å^3 148 atoms



(114 atoms, 14 sites, 4 elements)

(148 atoms, 19 sites, 6 elements)



Structures: solved/refined from HRPT NPD data

Chemistry of Materials 2015, 27, 7889–7895.

Hardly can be done at SINQ, due to intensity/resolution limitations…

Macromolecular crystallography:
Crystal structure of the eukaryotic 60S ribosomal subunit…

Method: X-RAY DIFFRACTION X06SA of the Swiss Light Source, PSI
Exp. Data:
Structure Factors

Figure: Model of the eukaryotic ribosome (taken from Klinge *et al.*)

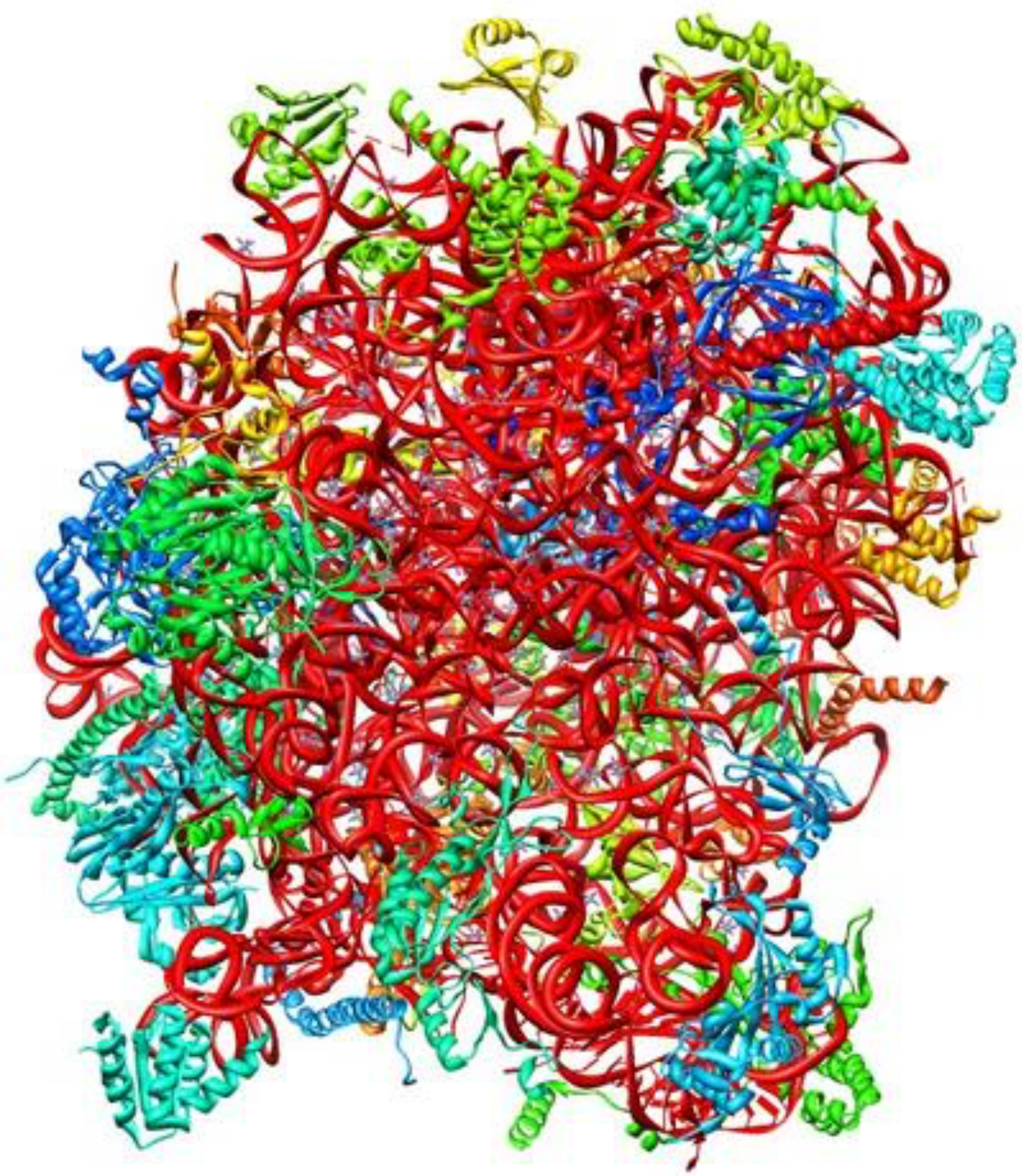
Science **334:** 941

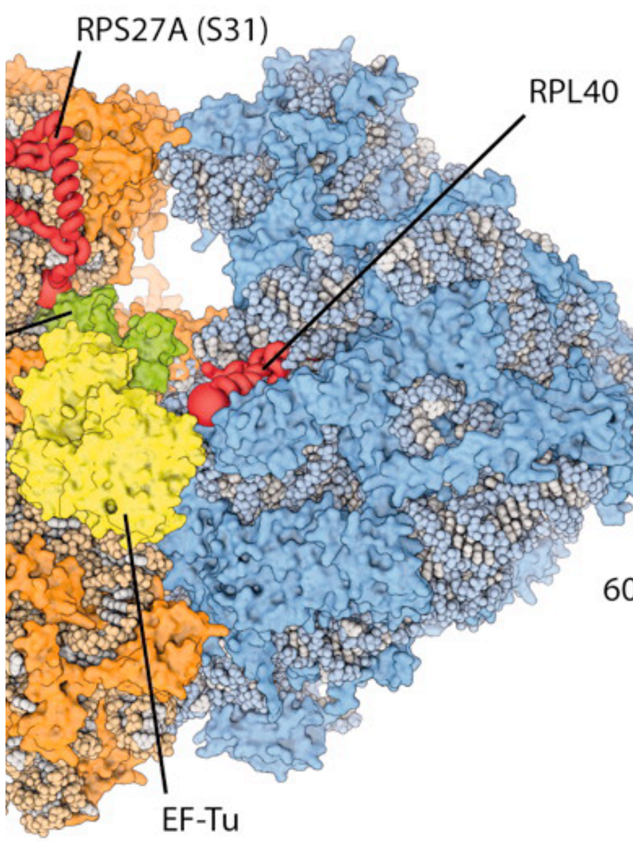
Unit Cell:

Length [Å]	Angles [°]
a = 320.19	α = 90.00
b = 289.25	β = 91.22
c = 535.04	γ = 90.00

V= 66’596'800 Å^3

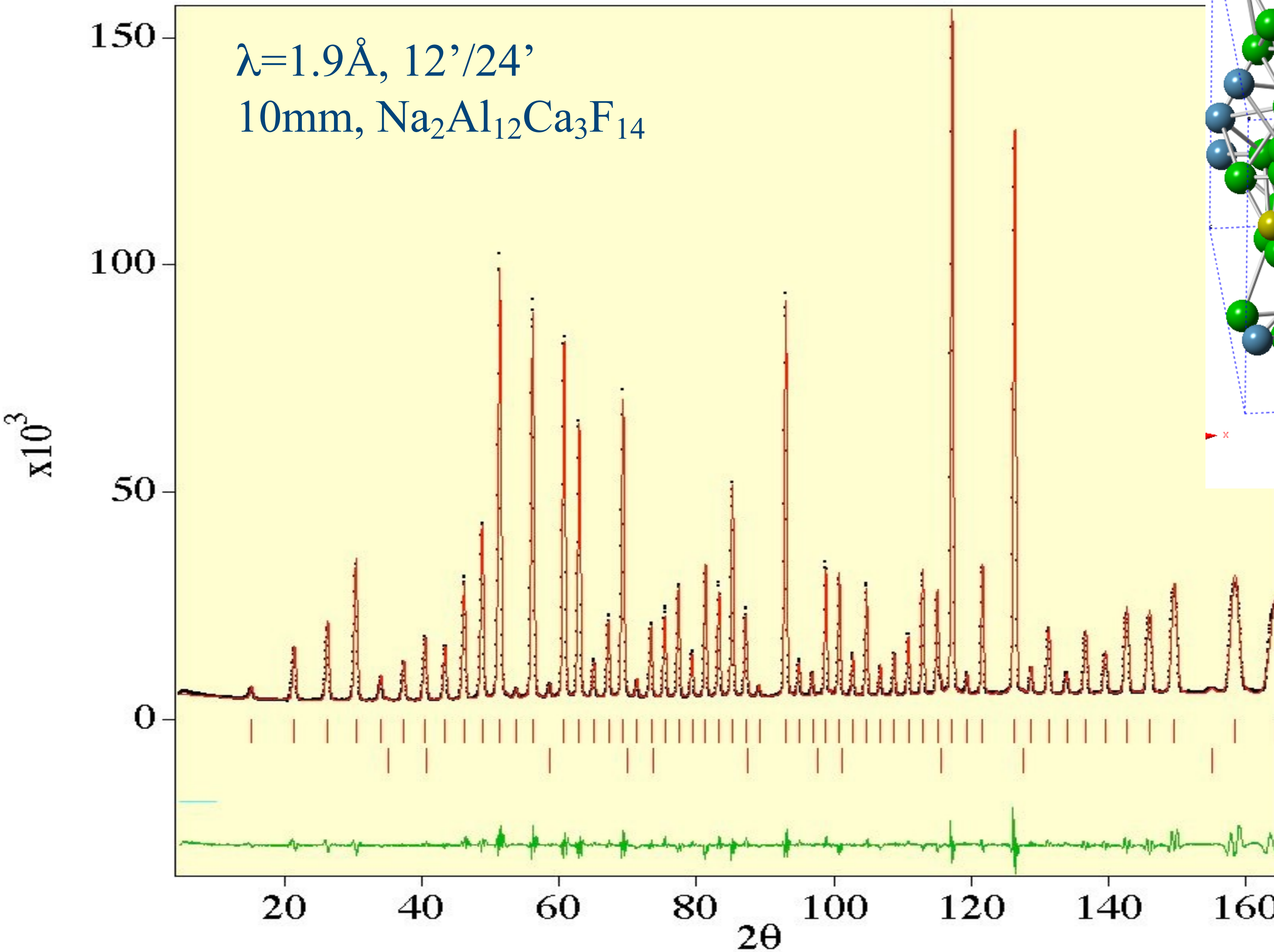
Resolution[Å]:	
R-Value:	0.216 (obs.)
R-Free:	
Space Group:	P 1 2_1 1

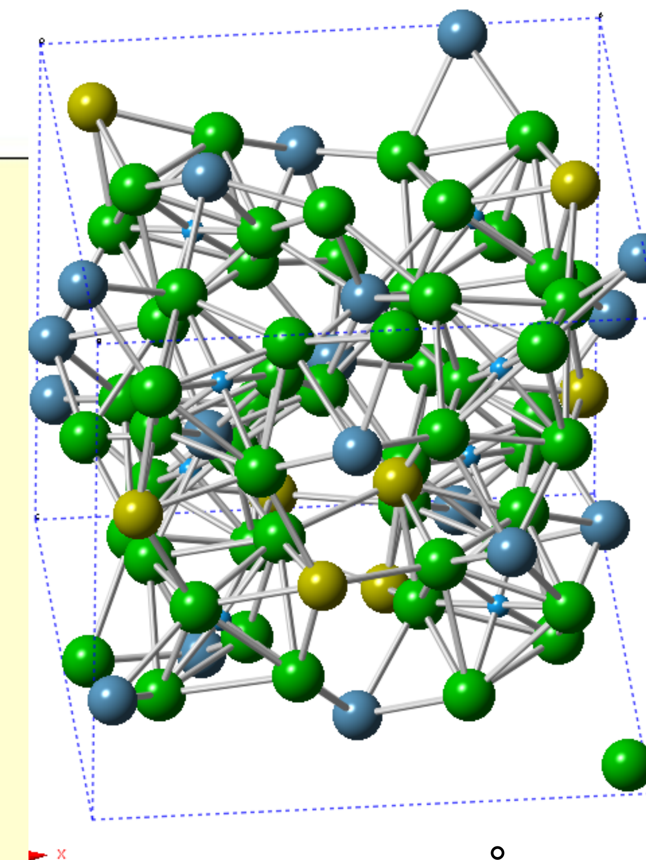




rms bonds ~0.01Å

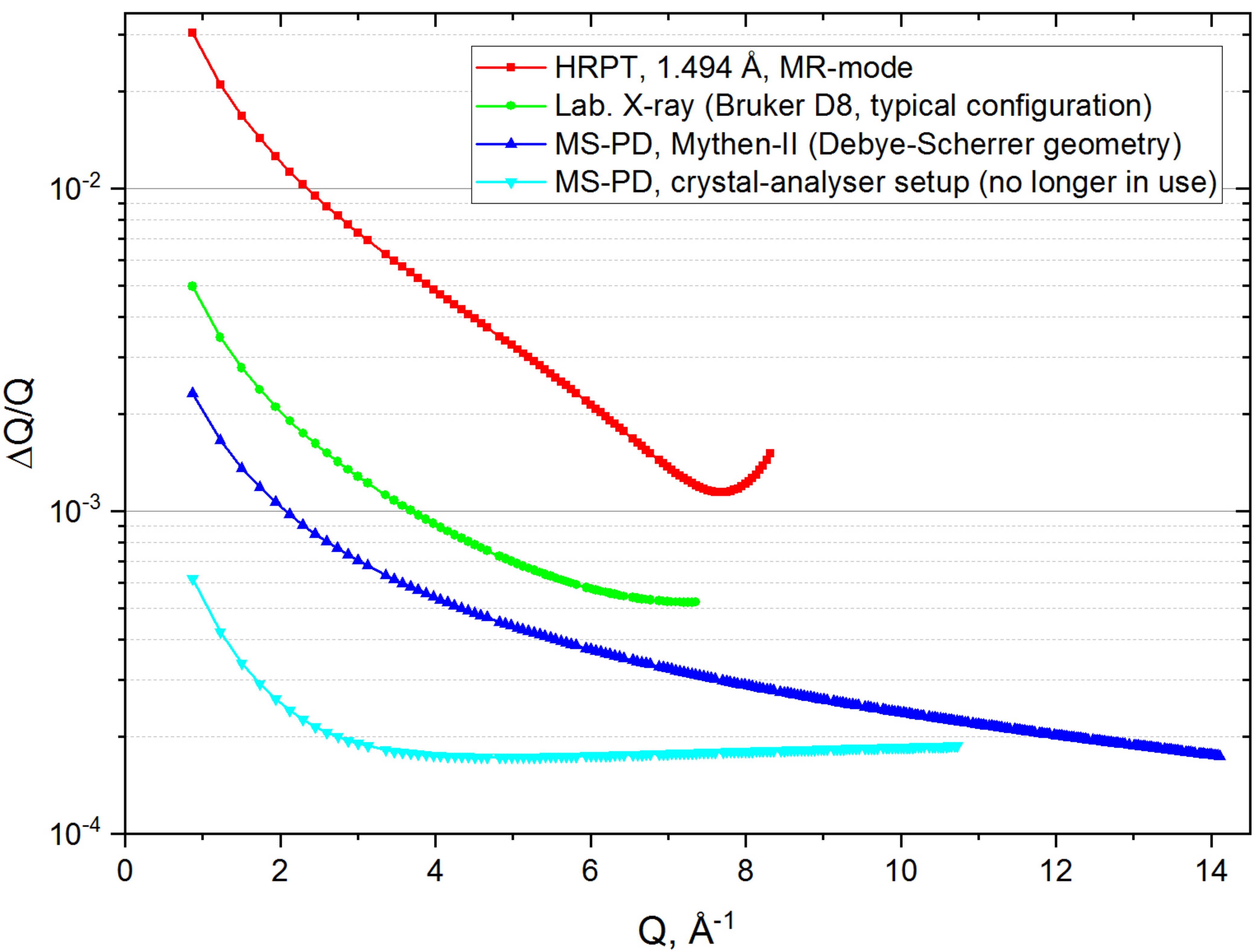
HRPT resolution calibration



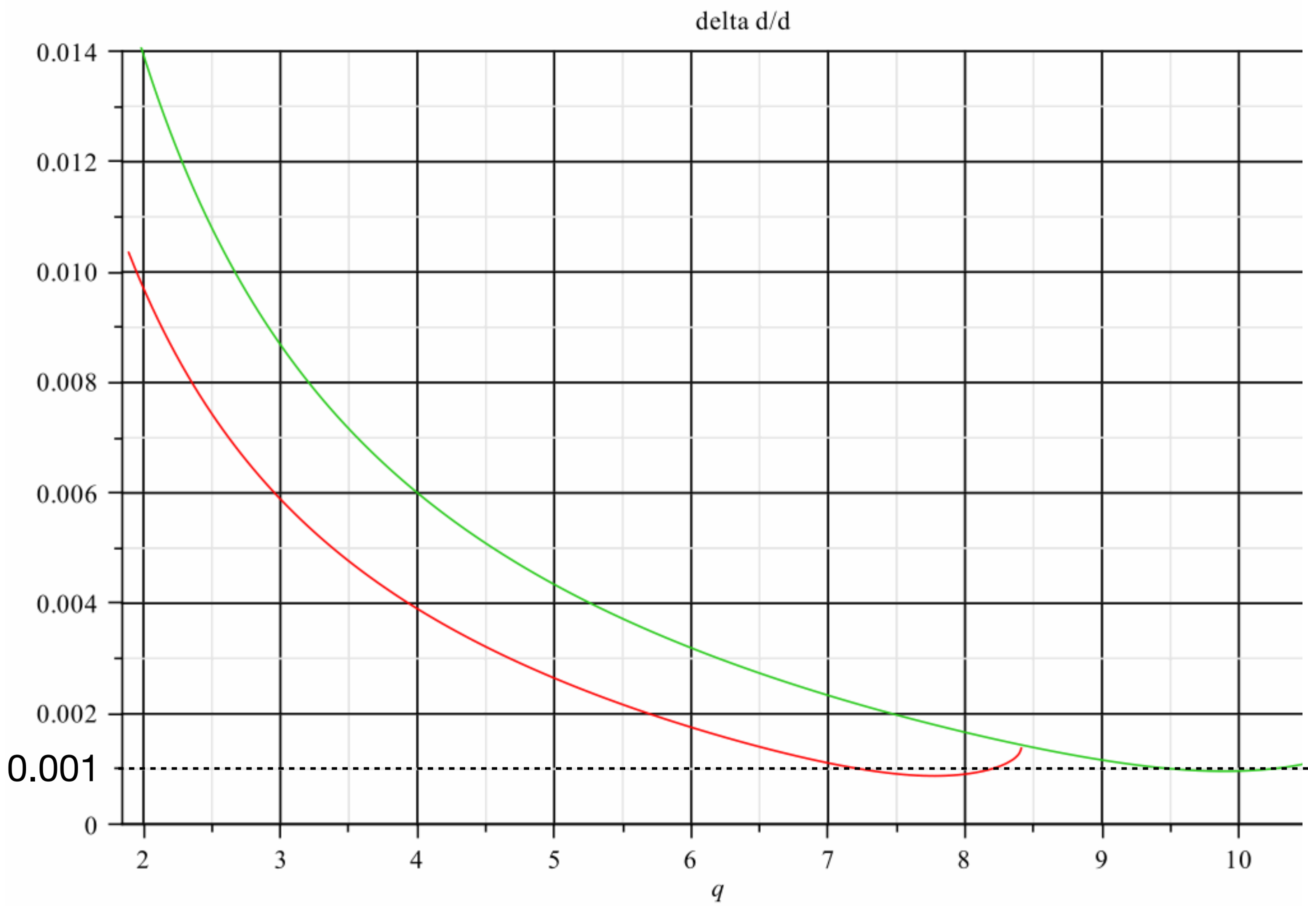


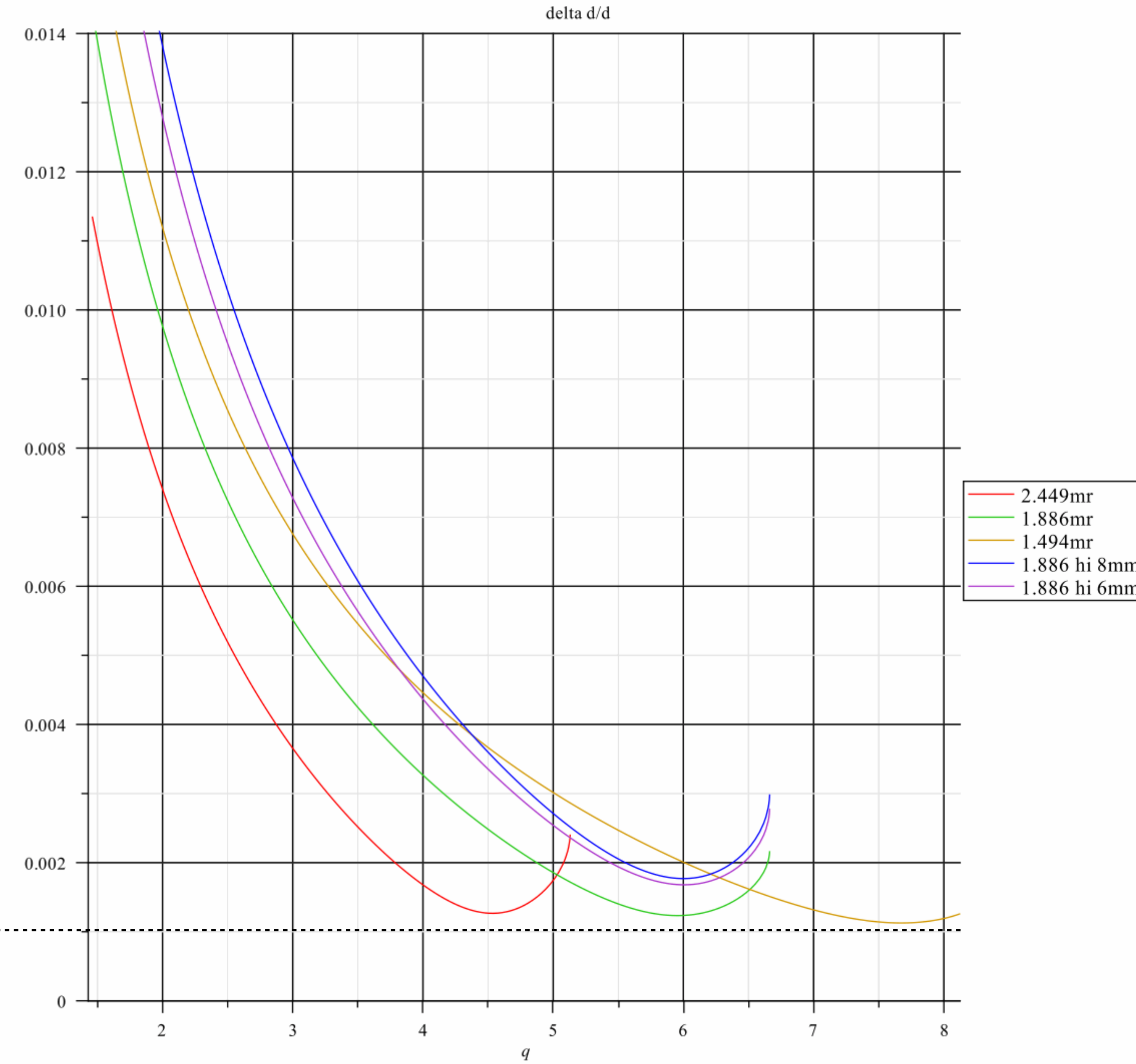
10x10x10Å

comparison of neutrons HRPT and lab. & SLS synchrotron x-ray resolutions



comparison of HRPT resolution curves for HR and HI





High Resolution HR
1.15 Å and 1.5 Å

Medium Resolution MR
High Intensity HI

How often we use high resolution HR at HRPT?

Statistics of the use of different resolutions at HRPT (2010-2017):

High Intensity	HI	289157	92.2%
Medium Resolution	MR	22774	7.3%
High Resolution	HR	1580	0.5%

How often we use high resolution HR at HRPT?

When do we need HR,MR? It costs x3, x10 increase in data collection time with respect to high intensity

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Beam-time is finite…,
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- Indexing of peaks
  - structure solution
  - small deviations from high symmetry metrics (space group)

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Must use/have high resolution in the following cases

- Indexing of peaks
    - structure solution
    - small deviations from high symmetry metrics (space group)

- Peak/background, for small (magnetic) peaks

Statistics of the use of different resolutions at HRPT (2010-2017):

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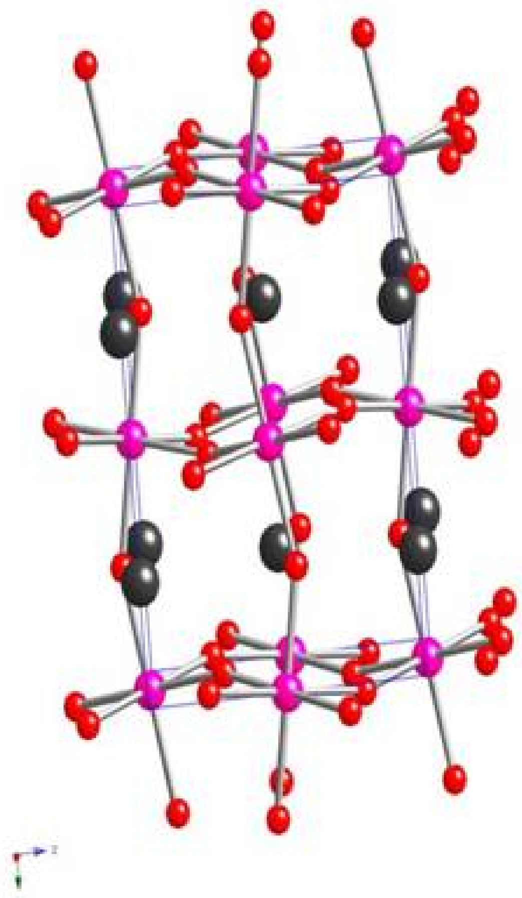
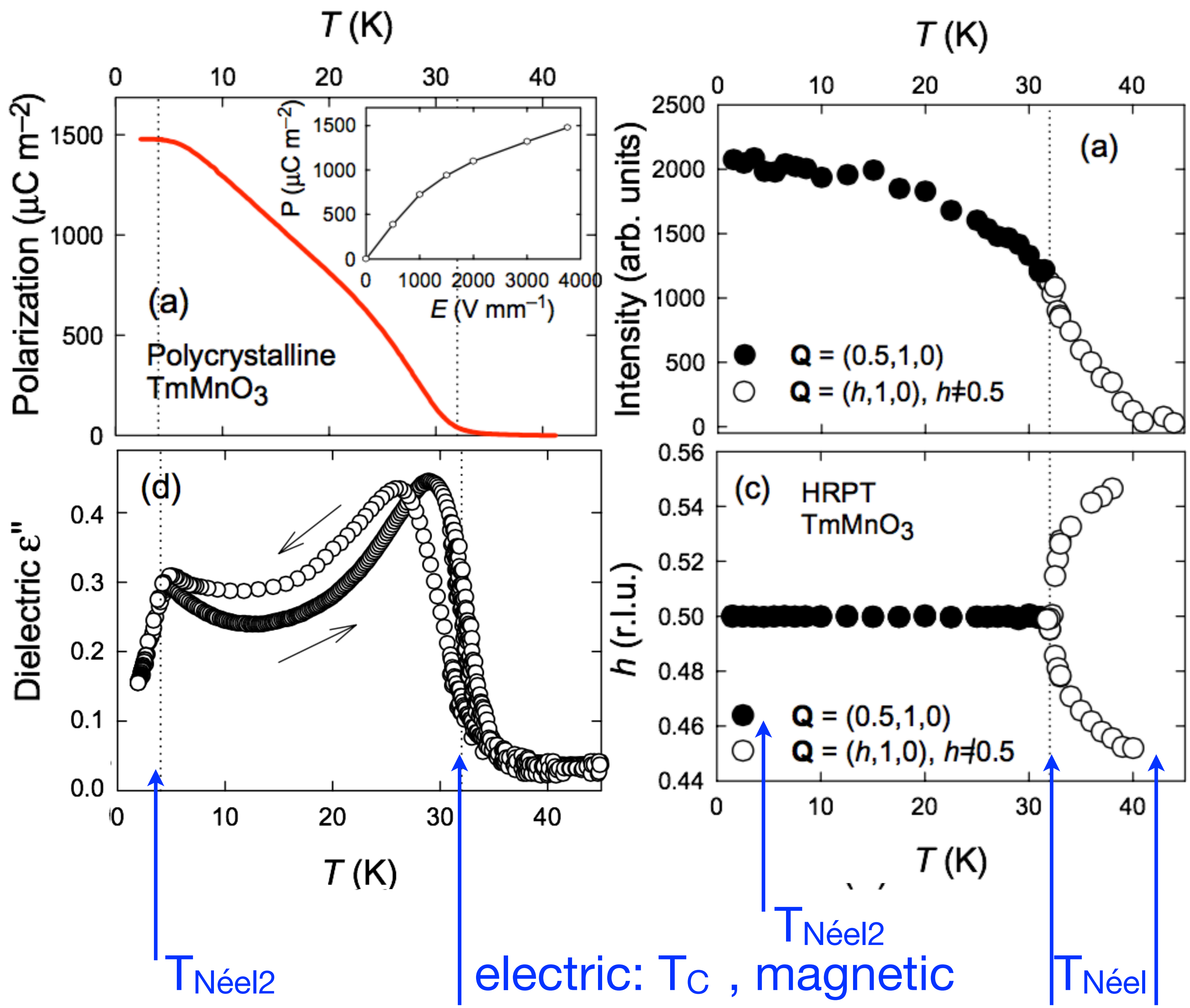
Spin-lattice coupling and antiferromagnetic order in orthorhombic multiferroic* $TmMnO_3$





* materials that have coupled electric, magnetic and structural order parameters

V. Y. Pomjakushin, M. Kenzelmann, A. Donni, A. B. Harris, T. Nakajima, S. Mitsuda, M. Tachibana, L. Keller, J. Mesot, H. Kitazawa, and E. Takayama-Muromachi, New J. Phys. 11, 043019 (2009).

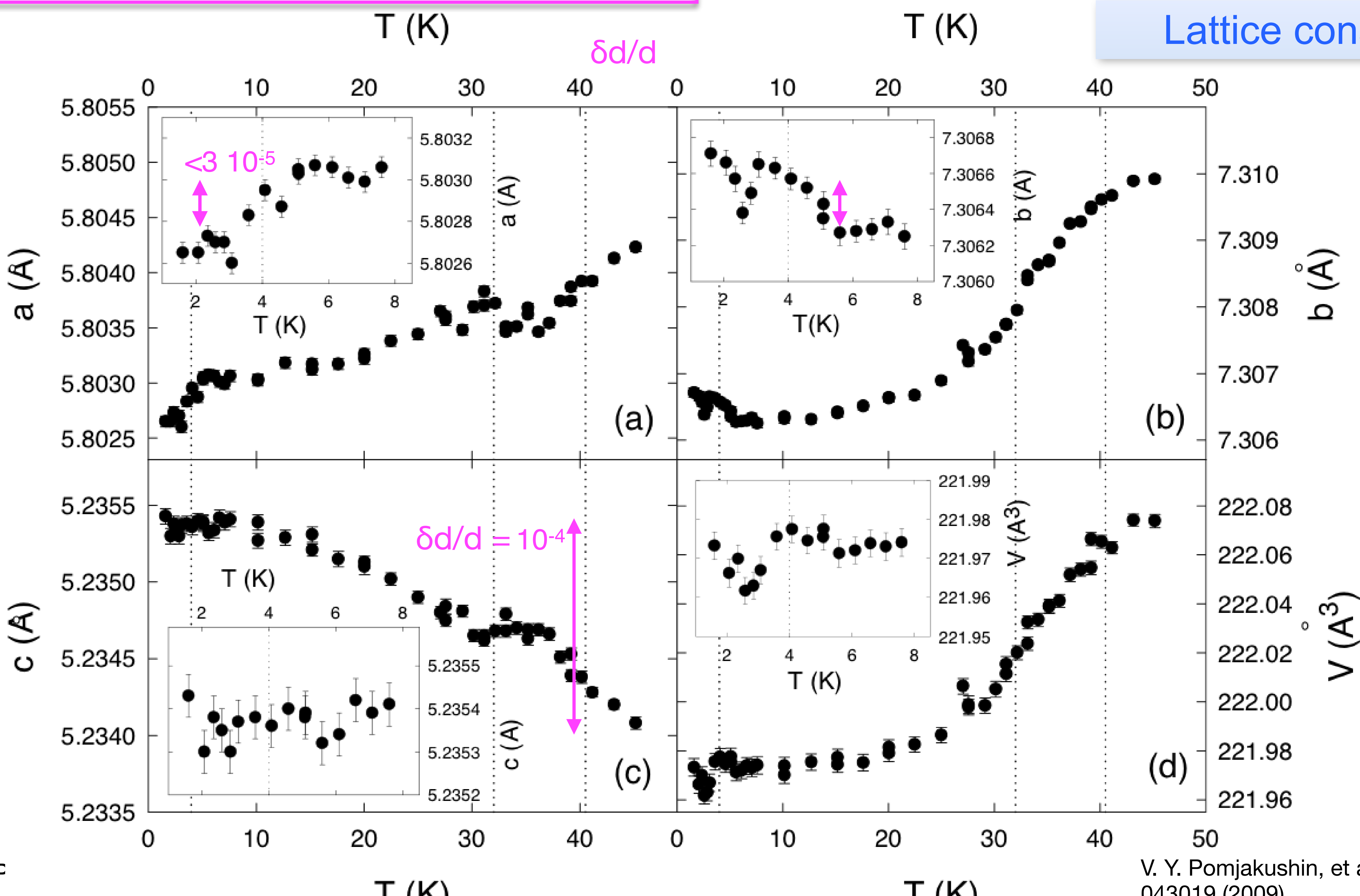
HRPT resolution in HI mode
$\delta d/d > 2\ 10^{-3}$

Example of accuracy on metric : orthorhombic multiferroic $TmMnO_3$

material that have coupled electric, magnetic and structural order parameters

~0.0001Å=10 fm (proton radius 2fm)

Lattice constants



V. F

V. Y. Pomjakushin, et al New J. Phys. 11, 043019 (2009).

Examples of PND@HRPT applications to magnetic structures

Examples of PND@HRPT applications to magnetic structures

1. Limitation from the medium resolution at low Q. Impossibility to resolve two very different magnetic models in $La_{1/3}Sr_{2/3}FeO_3$ (to find out if it has charge ordering CO)

Examples of PND@HRPT applications to magnetic structures

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2. Good enough resolution at low-Q domain: modulated with long period *magnetic structure and topological charges (skyrmions) in Weyl semimetal CeAlGe*

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2. Good enough resolution at low-Q domain: modulated with long period *magnetic structure and topological charges (skyrmions) in Weyl semimetal CeAlGe*

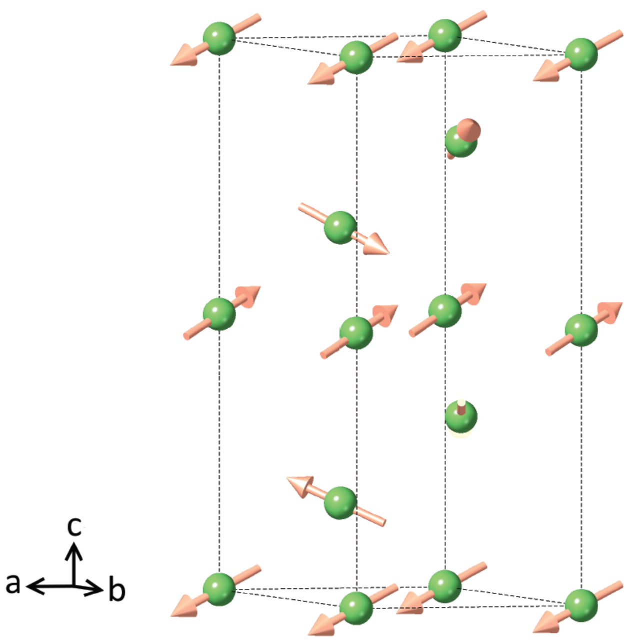
3. high-Q range and resolution is not important: *Magnetic octupole-octupole correlations on the pyrochlore lattice in* $Ce_2Sn_2O_7$

Limitation from the resolution. Impossibility to resolve two very different magnetic models.

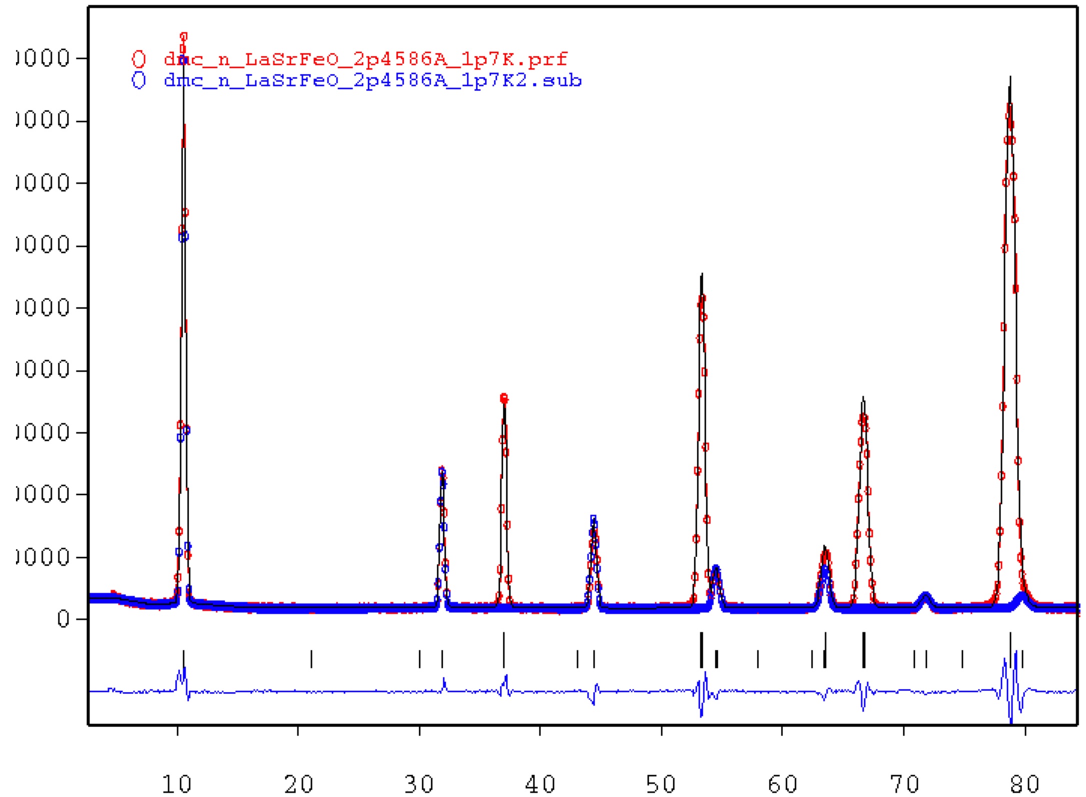
Crystal and magnetic structure of $R_{1/3}Sr_{2/3}FeO_3$ (R = La, Pr, Nd), F. Li *et al*, Phys. Rev. B 97, 174417(2018)

Fm3m -> R-3c at above RT, rhombohedral distortion 5 10^{-4}
In R-3c AFM below 200K in $La_{1/3}Sr_{2/3}FeO_3$

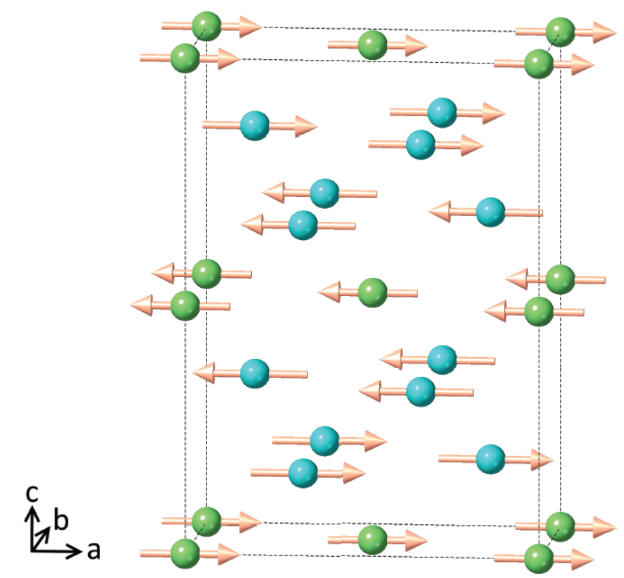
canted helical model ($P3_221$)



vs.



collinear model (C2/c)

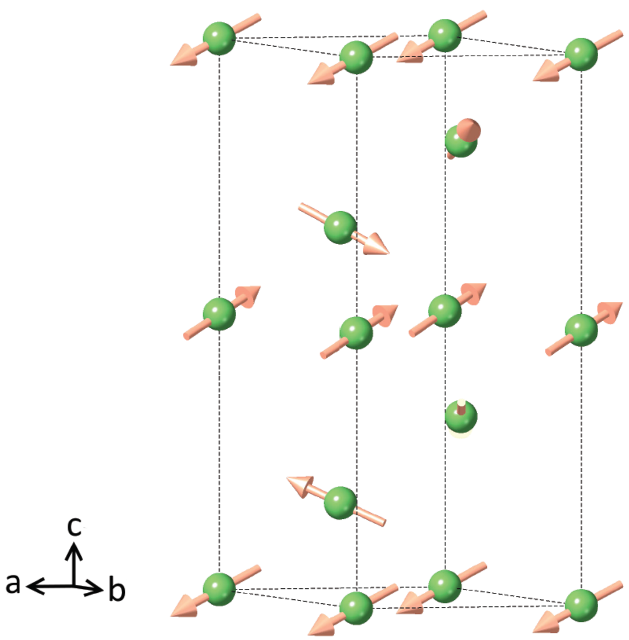


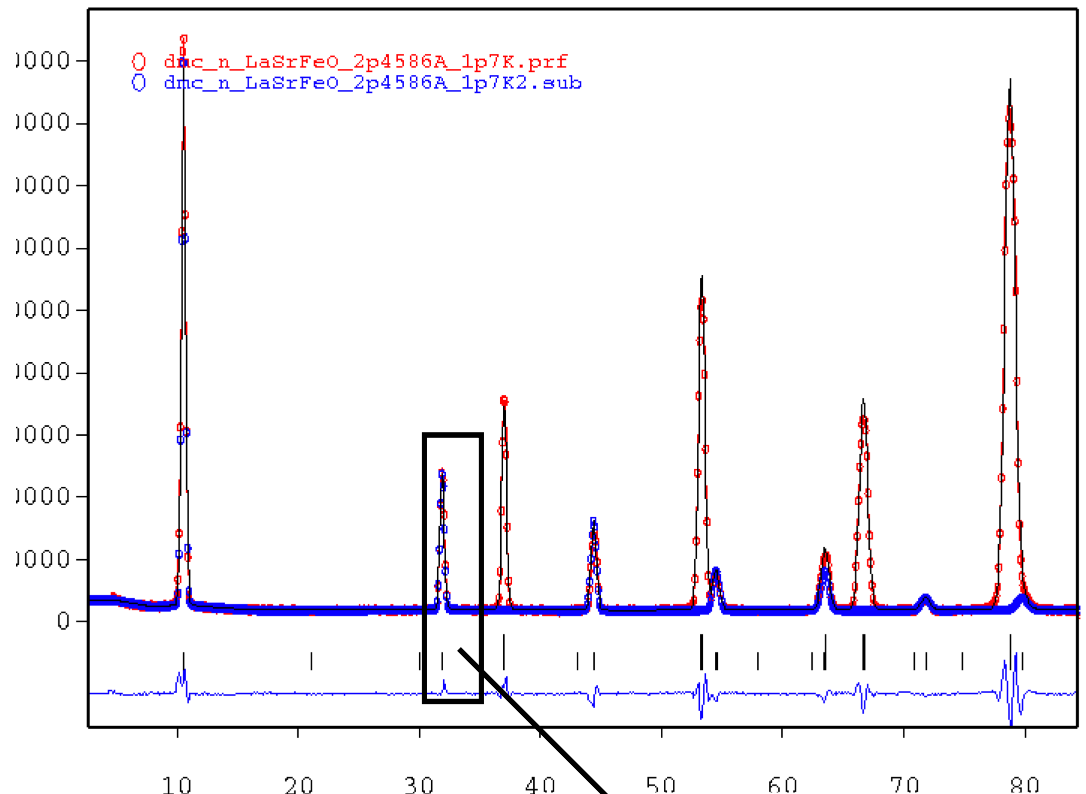
Limitation from the resolution. Impossibility to resolve two very different magnetic models.

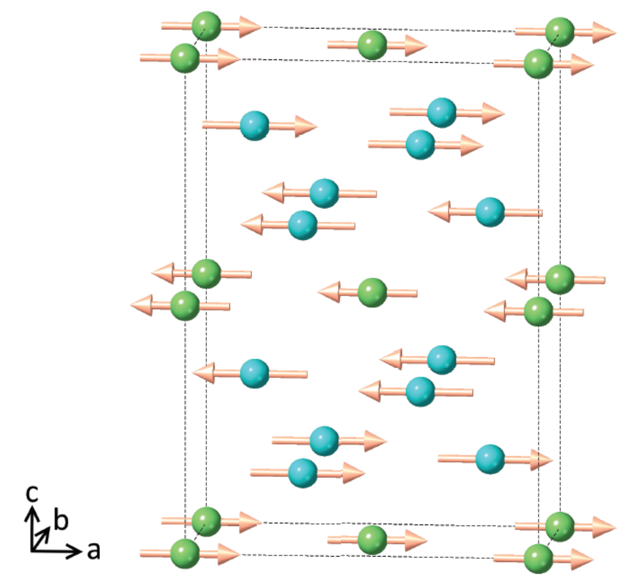
Crystal and magnetic structure of $R_{1/3}Sr_{2/3}FeO_3$ (R = La, Pr, Nd), F. Li *et al*, Phys. Rev. B 97, 174417(2018)

Fm3m -> R-3c at above RT, rhombohedral distortion 5 10^{-4}
In R-3c AFM below 200K in $La_{1/3}Sr_{2/3}FeO_3$

canted helical model ($P3_221$) vs. collinear model (C2/c)



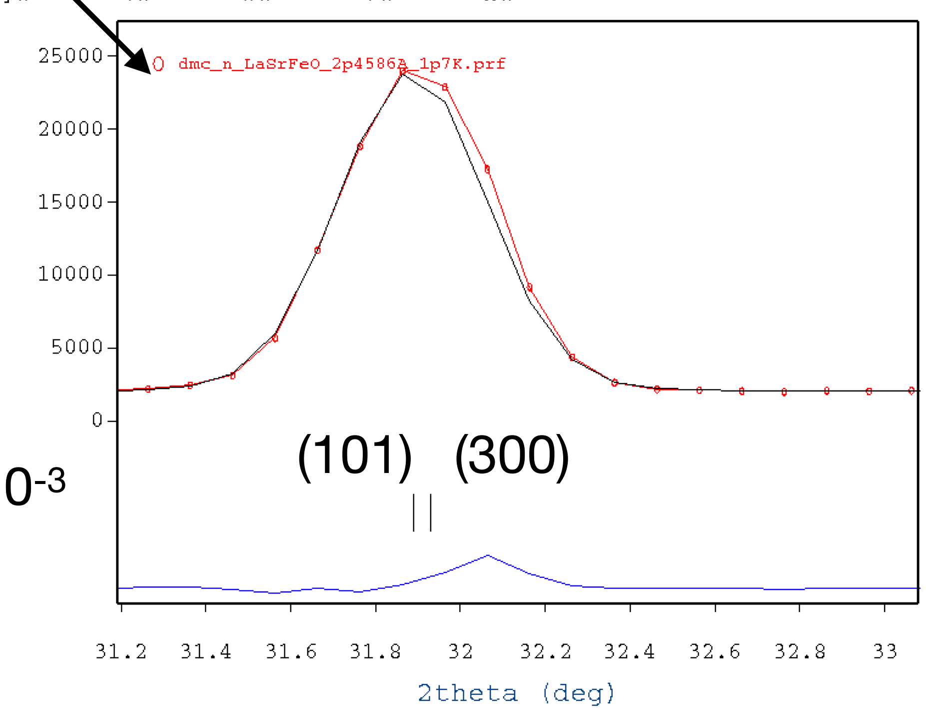




I(300)="very large"

I(300)==0

we have δd/d ~10^{-2}
but
we need δd/d better than10^{-3}
to distinguish the models

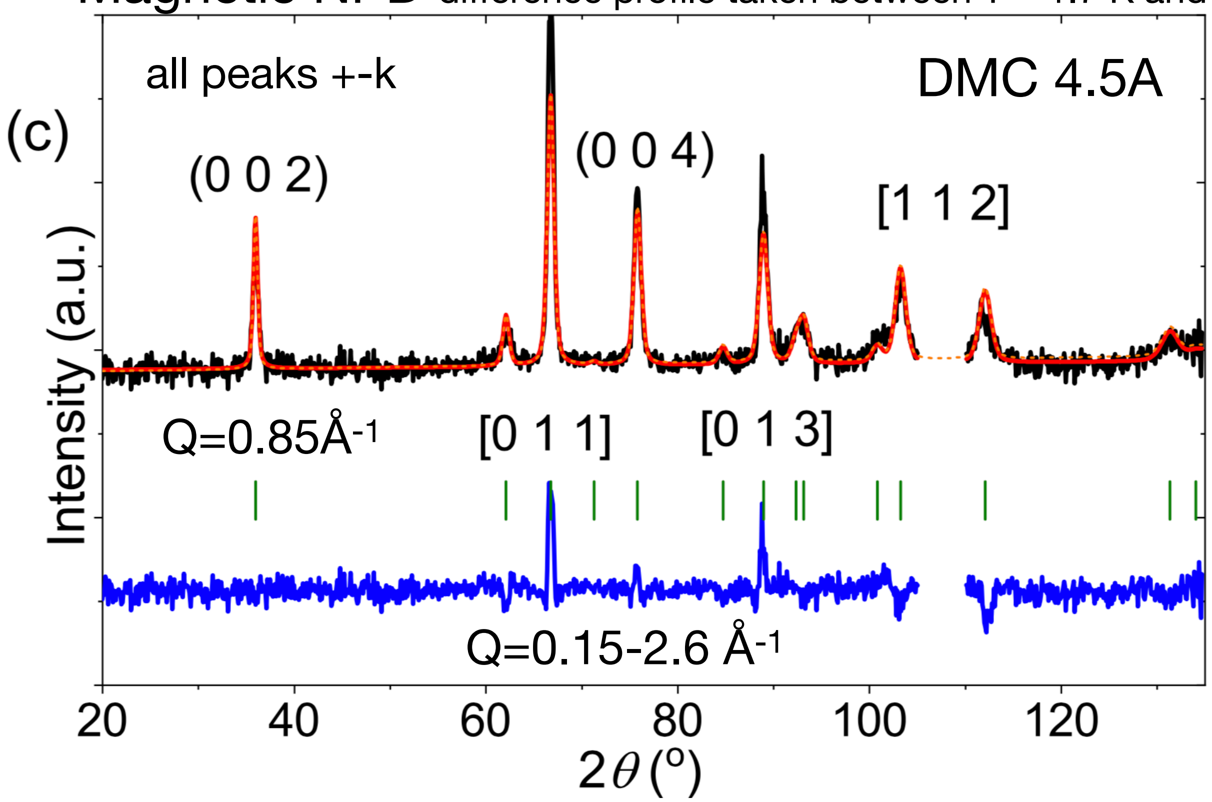


Superspace magnetic structure and topological charges in Weyl semimetal CeAlGe

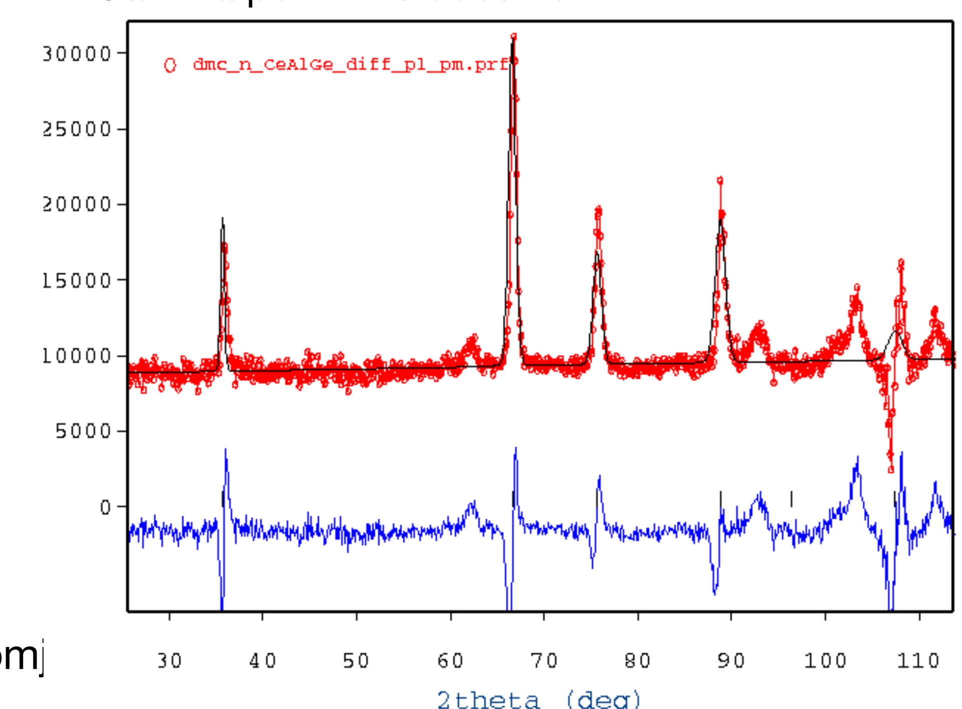
P. Puphal, et al, Physical Review Letters, 124, 017202 (2020)

k1=[g,0,0], SM point of BZ, g=0.06503(22) ~65Å

Magnetic NPD difference profile taken between T = 1.7 K and 10 K



Gamma point k=0 does not fit NPD



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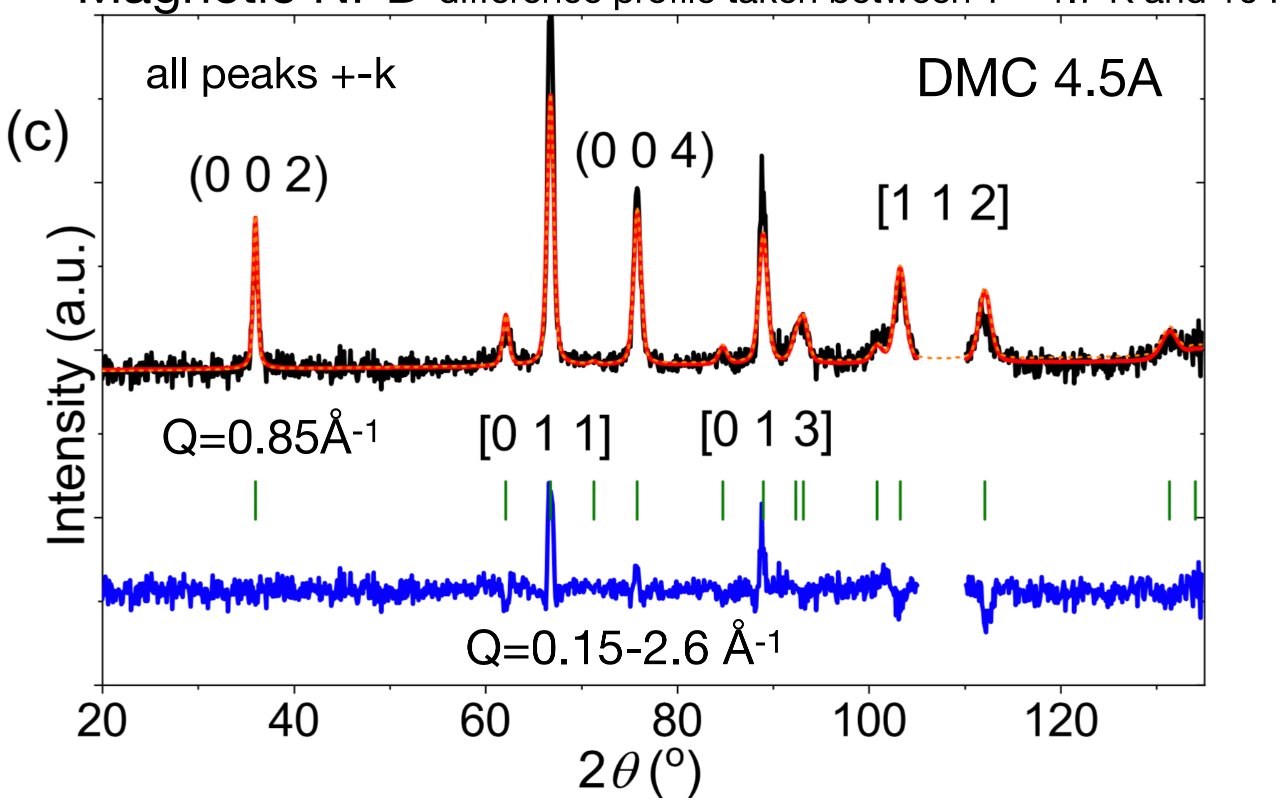
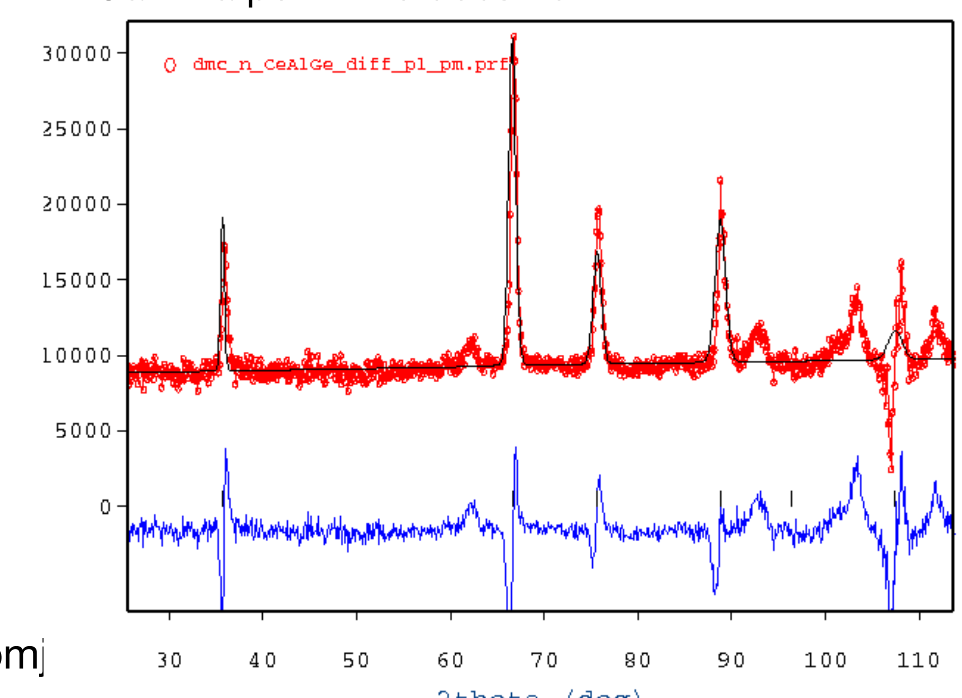
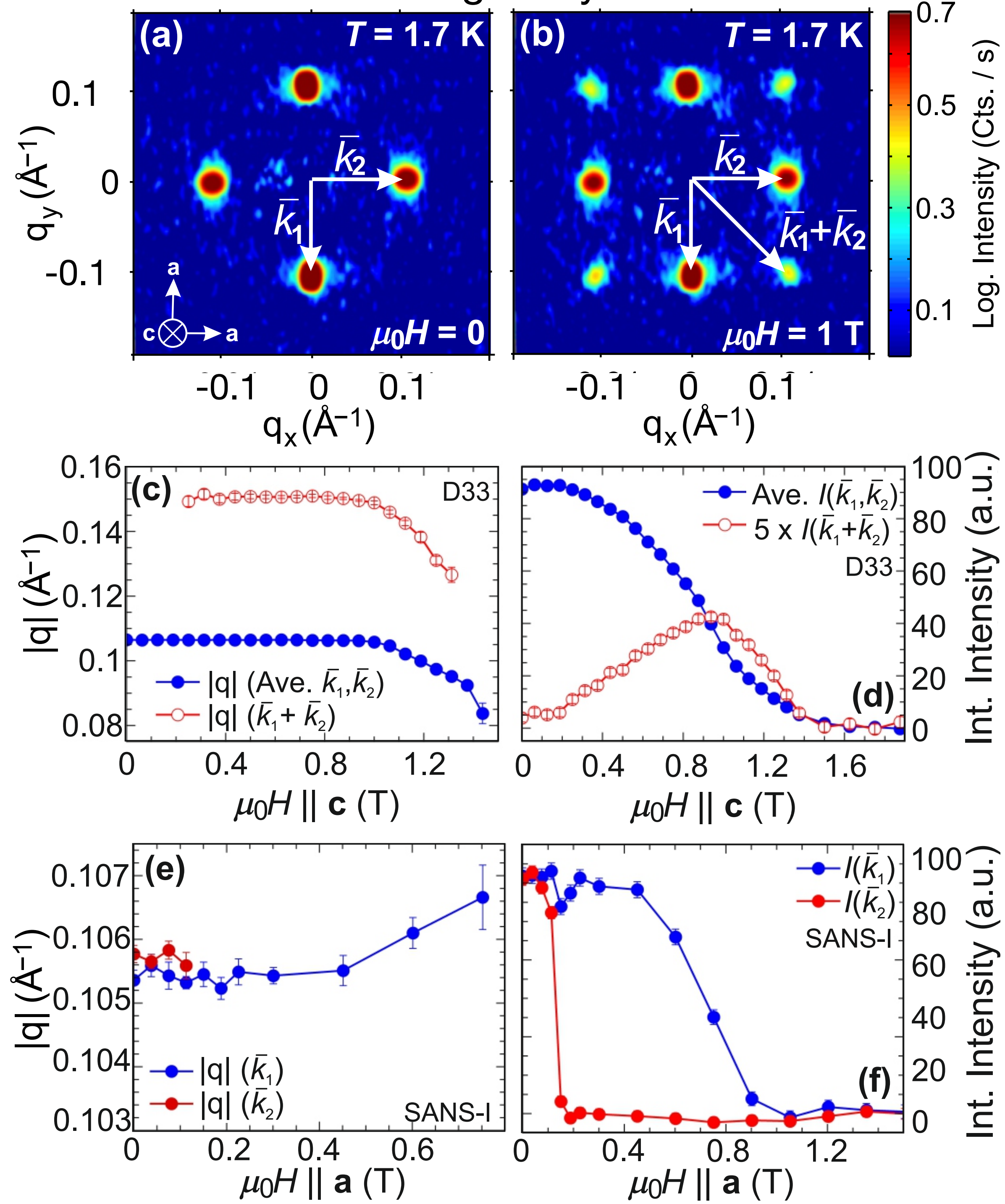


Gamma point k=0 does not fit NPD



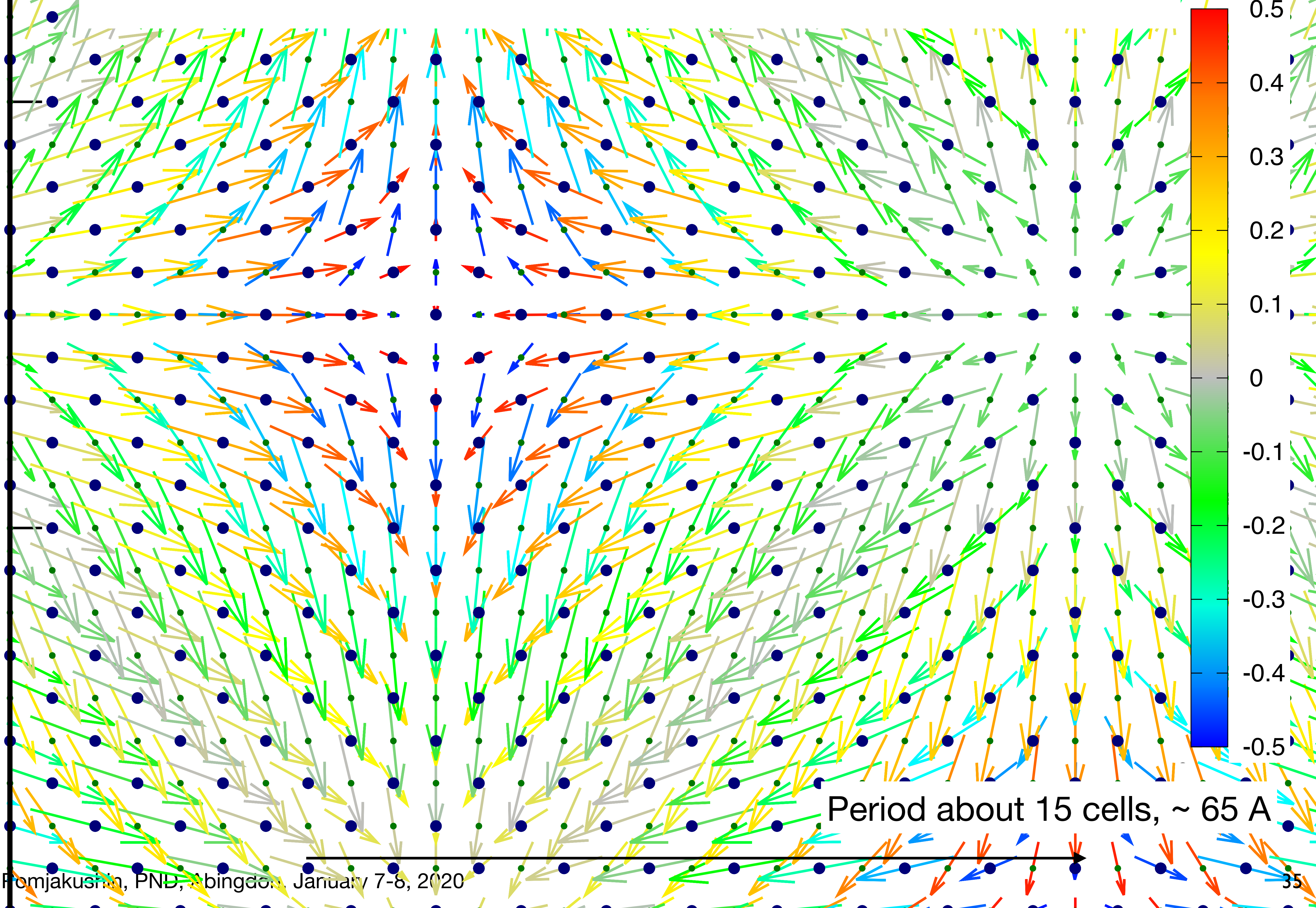
k1=[g,0,0], k2=[0,g,0]

Single crystal



V. Pom

CeAlGe: Maximal symmetry full star superspace 3D+2 magnetic group I4_1md1'(a00)000s(0a0)0s0s



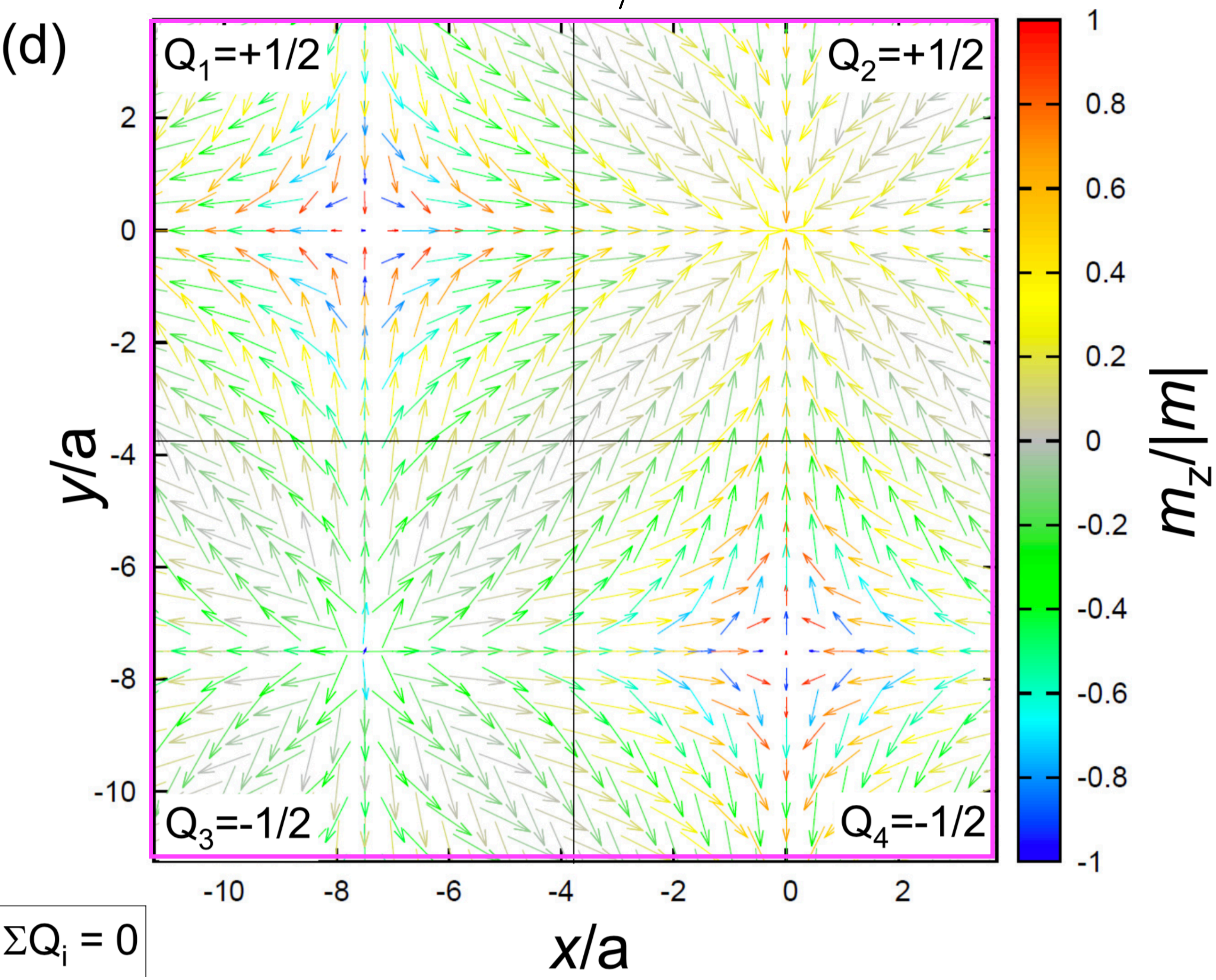
P. Puphal, et al, Physical Review Letters, 124, 017202 (2020)

Topological density and charge

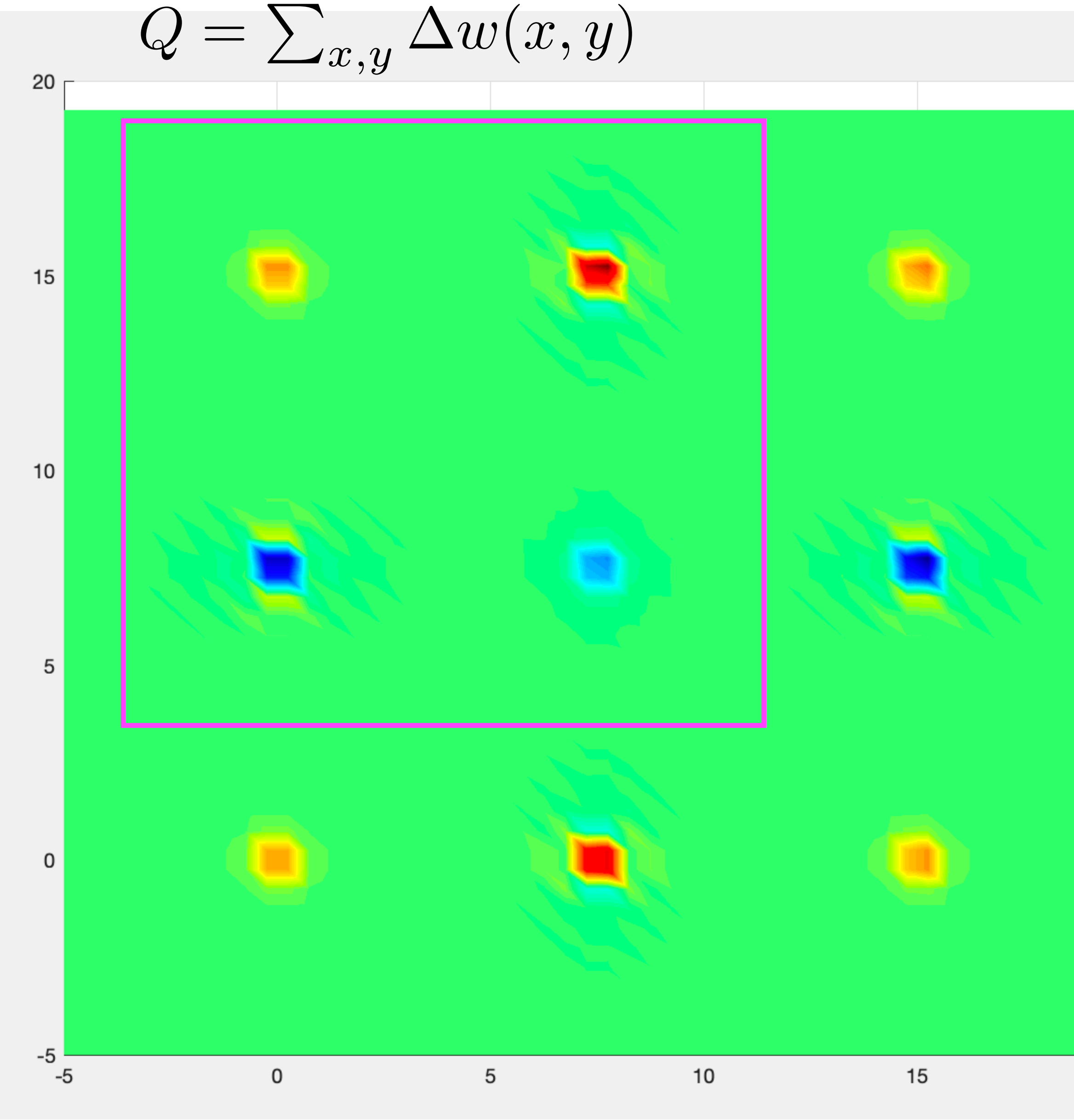
experiment: (m1,m2,m3,m4) = (0.44(1), 1.02(1), −0.21(5), 0.29(7)) μB.

$$\Delta w(x, y) = \frac{\text{solid angle per square placket}}{4\pi}$$

$$\mathbf{n} = \mathbf{M}/M$$



$$Q = \sum_{x,y} \Delta w(x, y)$$



$\Sigma Q_i = 0$

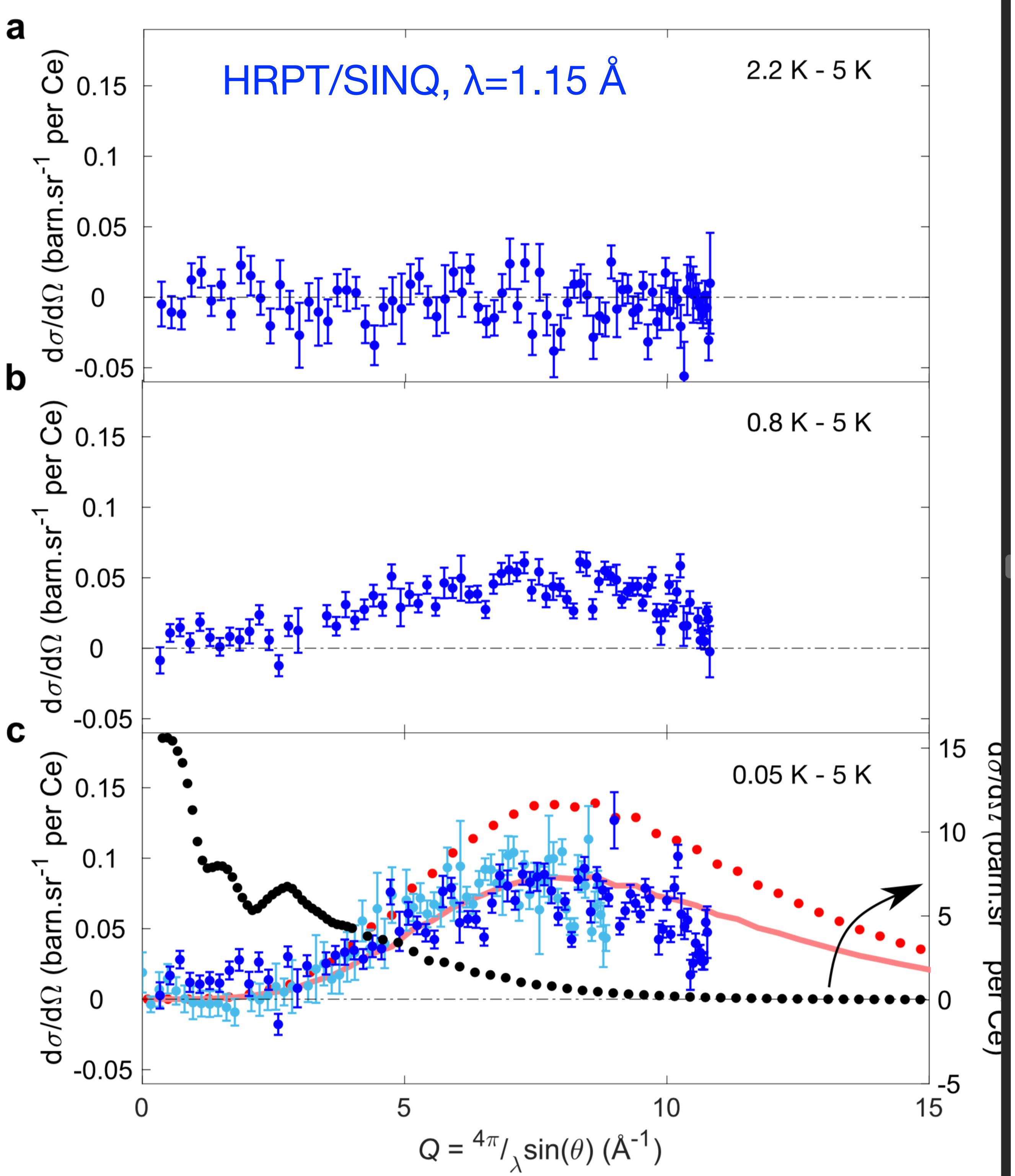
$$\mathbf{M}_{\mathrm{Ce1}} = m_1 \sin(\tilde{k}x)\mathbf{e}_{\boldsymbol{x}} + m_2 \sin(\tilde{k}y)\mathbf{e}_{\boldsymbol{y}} + \left(m_3 \cos(\tilde{k}x) + m_4 \cos(\tilde{k}y)\right)\mathbf{e}_{\boldsymbol{z}}$$

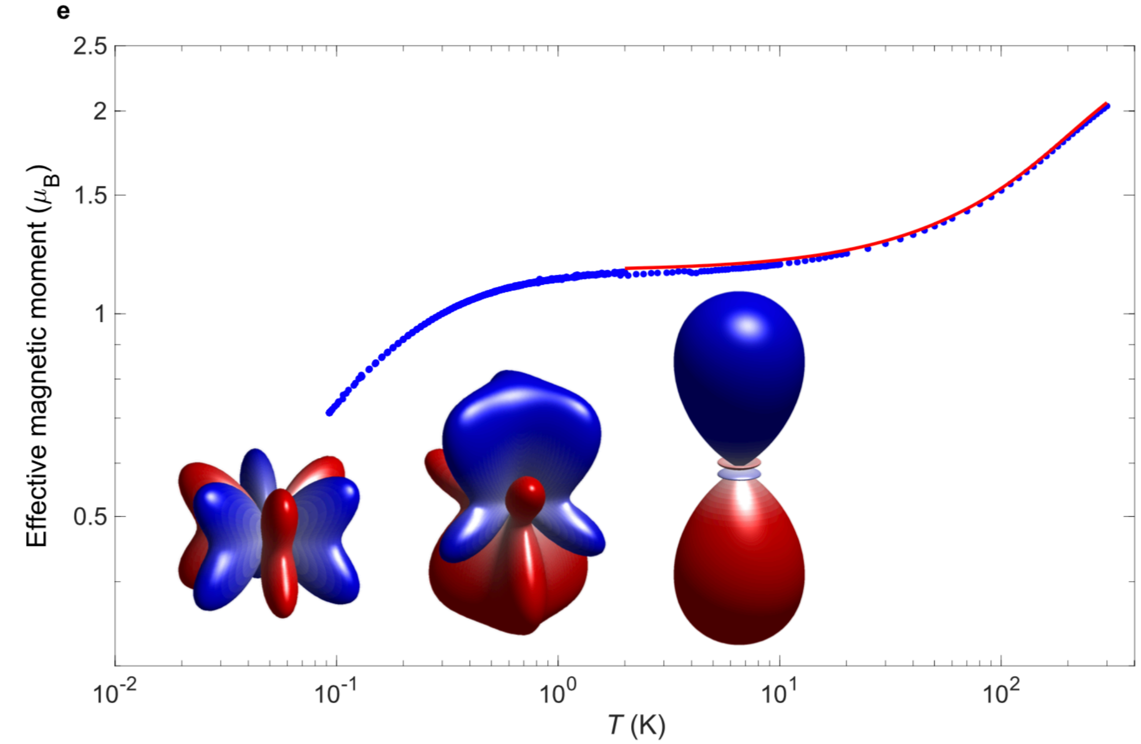
$$\mathbf{M}_{\mathrm{Ce2}} = m_2 \sin(\tilde{k}x)\mathbf{e}_{\boldsymbol{x}} + m_1 \sin(\tilde{k}y)\mathbf{e}_{\boldsymbol{y}} + \left(m_4 \cos(\tilde{k}x) + m_3 \cos(\tilde{k}y)\right)\mathbf{e}_{\boldsymbol{z}}$$

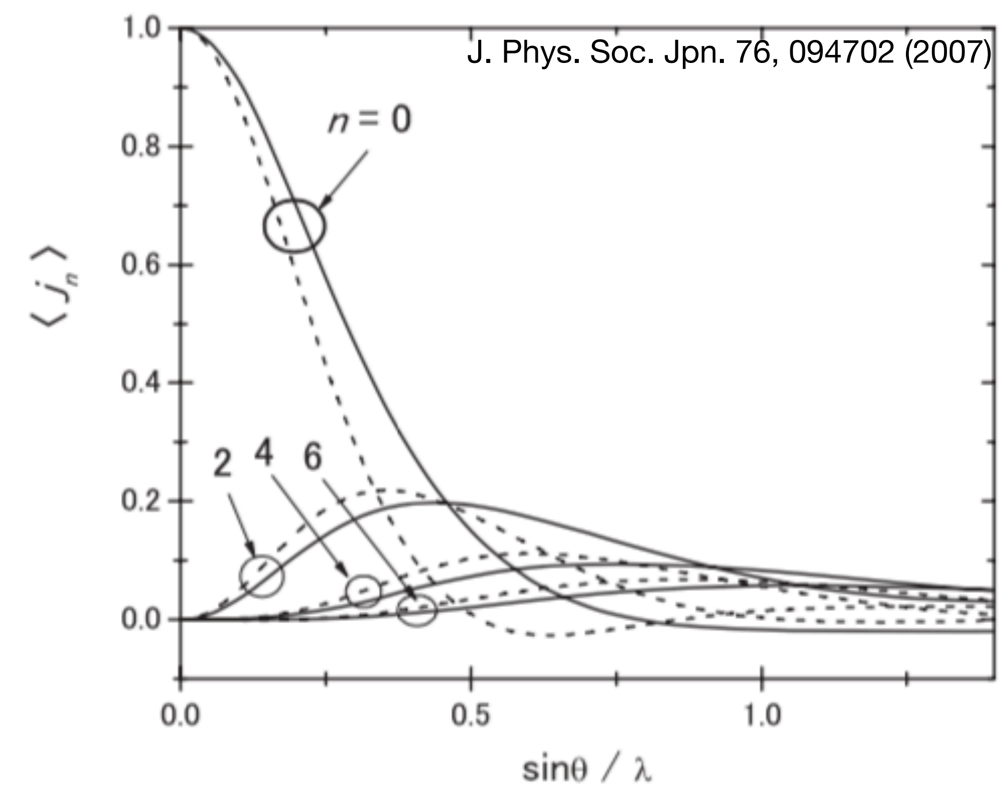
$\tilde{k}=2\pi|\boldsymbol{k}_1|=2\pi|\boldsymbol{k}_2|=2\pi g$

Magnetic octupole-octupole correlations on the pyrochlore lattice in Ce2Sn2O7

Romain Sibille, et al arXiv:1912.00928 [cond-mat.str-el]



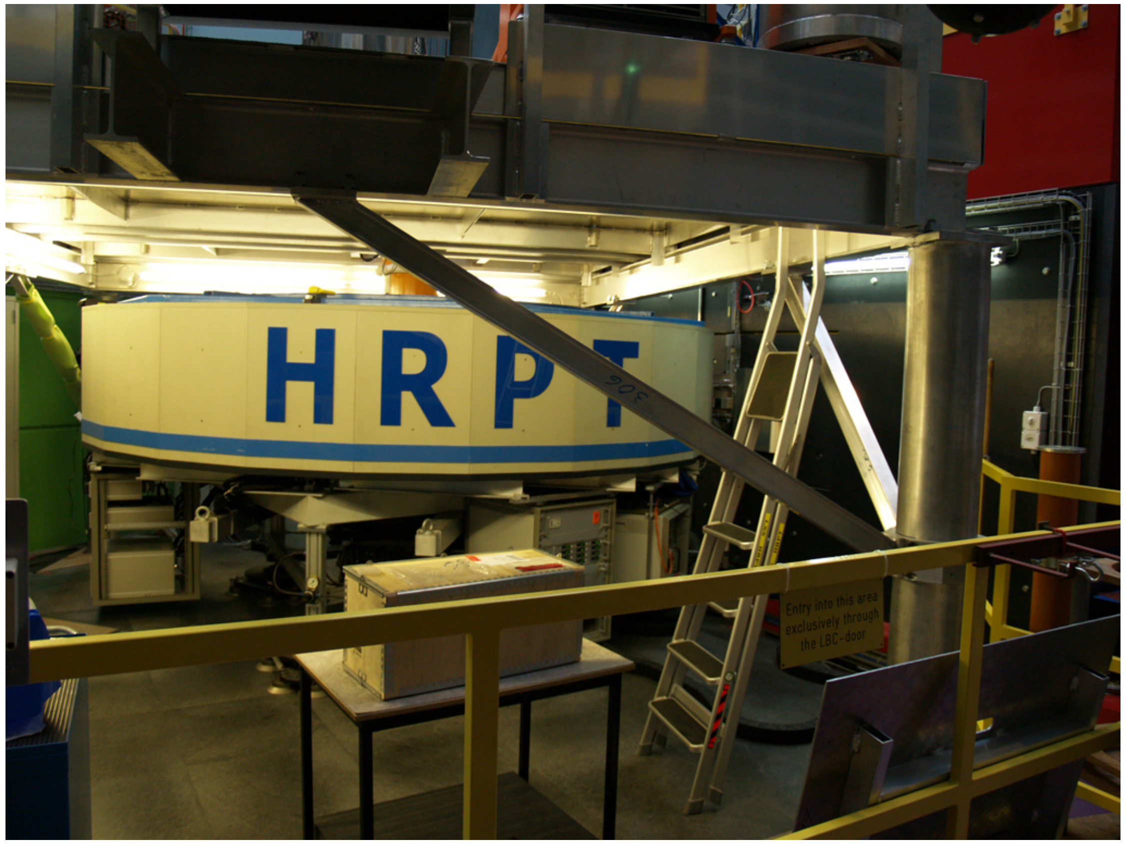




Radial integrations of spherical Bessel function $\langle j_n \rangle$ as fun
e neutron momentum transfer $\sin\theta/\lambda$, reproduced from ref. 4
and broken lines represent the results of Ce and Np

Samples, T, P, H and other equipment at HRPT/SINQ

- standard sample container: 6-10 mm dia x 50 mm (<4cm^3)
- due to low background small samples can be measured (30 mm^3)
- Radial collimators
- Sample changers 4-8 samples, T=1.5-300 K
- standard LNS sample environment:
  - Temperature = 50 mK—1800K,
  - Magnetic field H = 6 T (vertical)
  - Automatic He, N_2 refilling systems
- zero matrix high pressure cells:
  - clamp cells for 9 and 15 kbar
  - Paris Edinburgh cell 100 kbar



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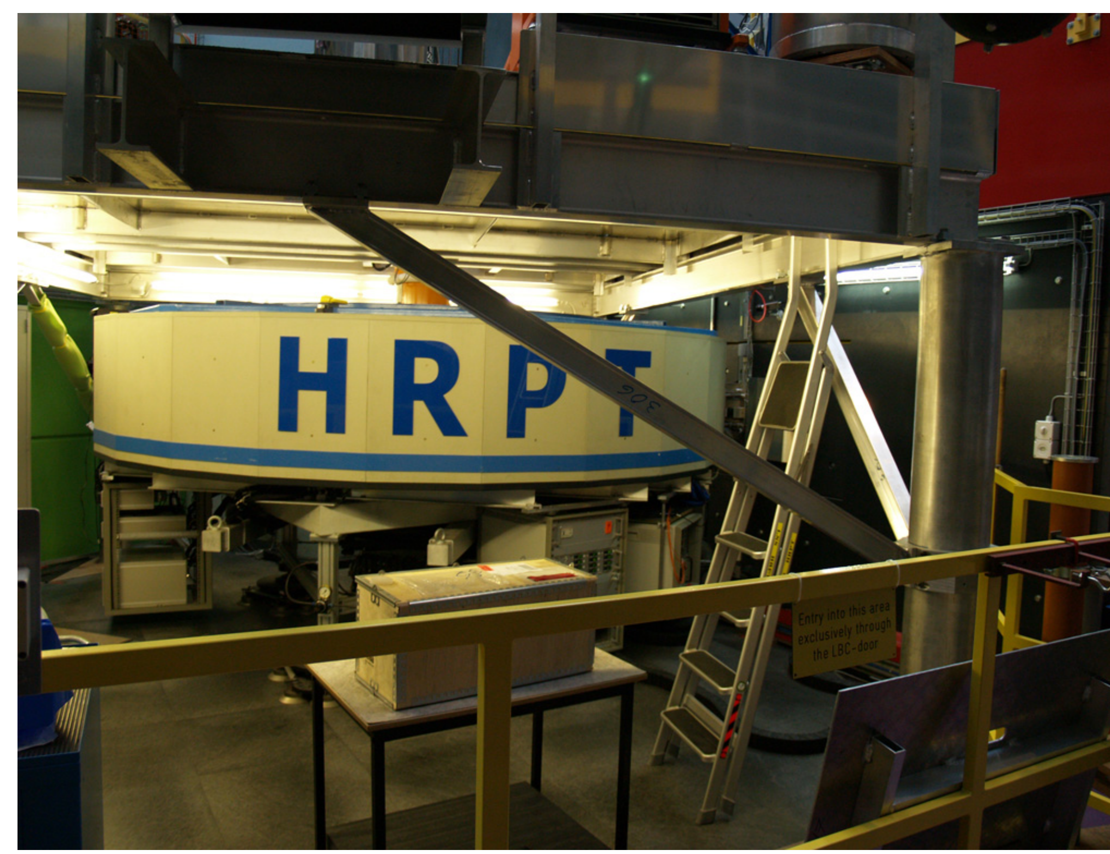
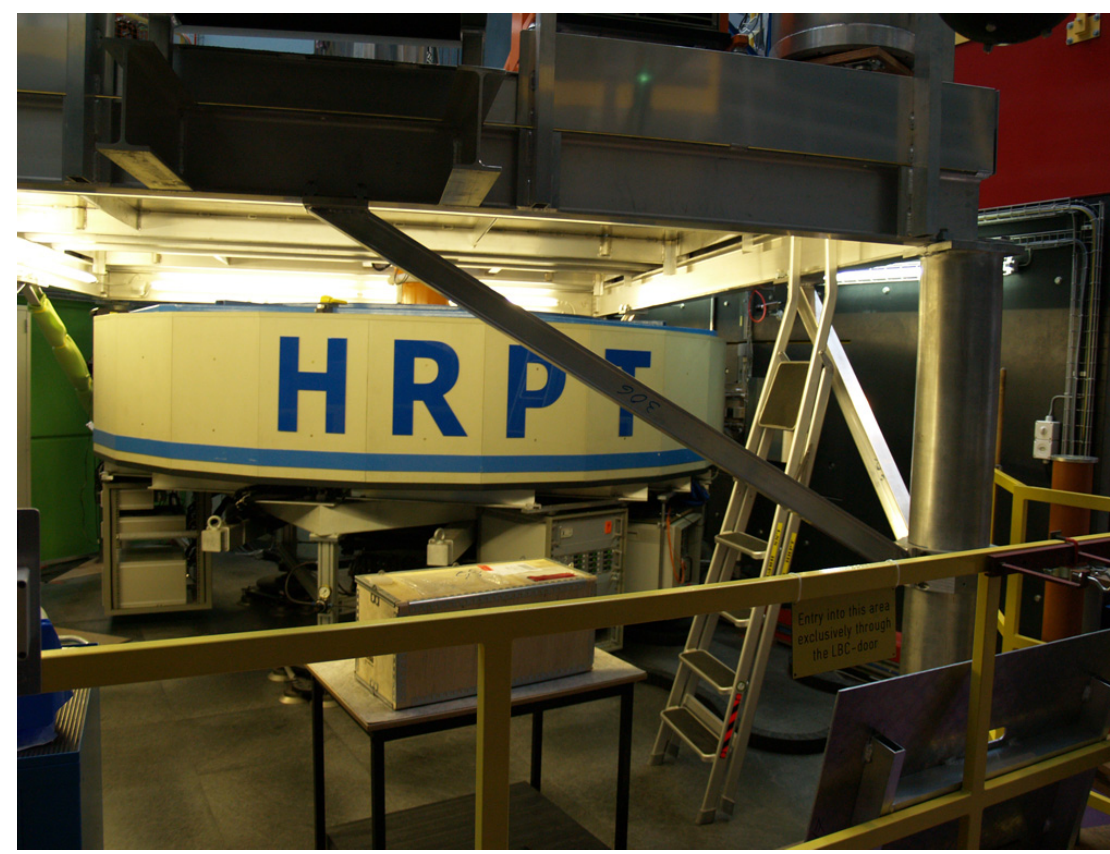
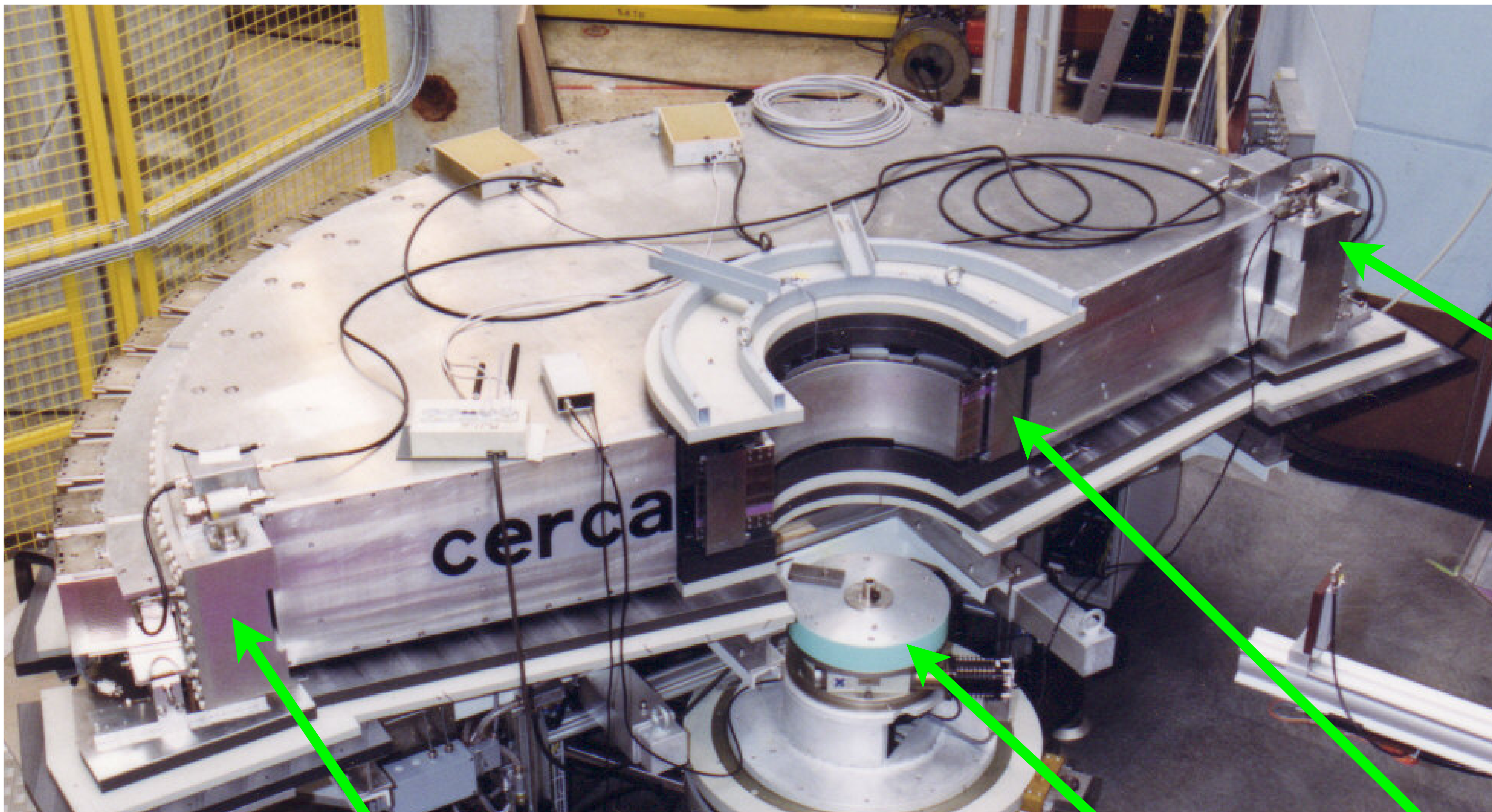


Samples, T, P, H and other equipment at HRPT/SINQ

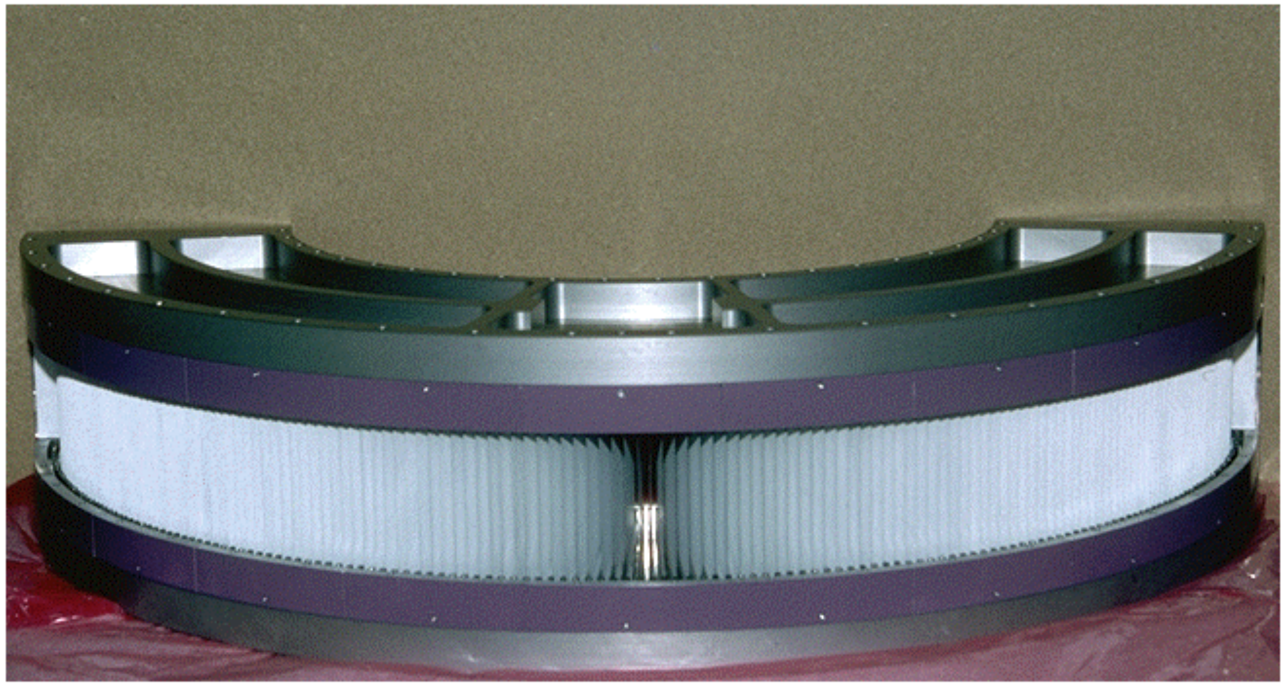
- standard sample container: 6-10 mm dia x 50 mm (<4cm^3)
- due to low background small samples can be measured (30 mm^3)
- Radial collimators: many pluses & one minus
- Sample changers 4-8 samples, T=1.5-300 K
- standard LNS sample environment:
  - Temperature = 50 mK—1800K,
  - Magnetic field H = 6 T (vertical)
  - Automatic He, N_2 refilling systems
- zero matrix high pressure cells:
  - clamp cells for 9 and 15 kbar
  - Paris Edinburgh cell 100 kbar

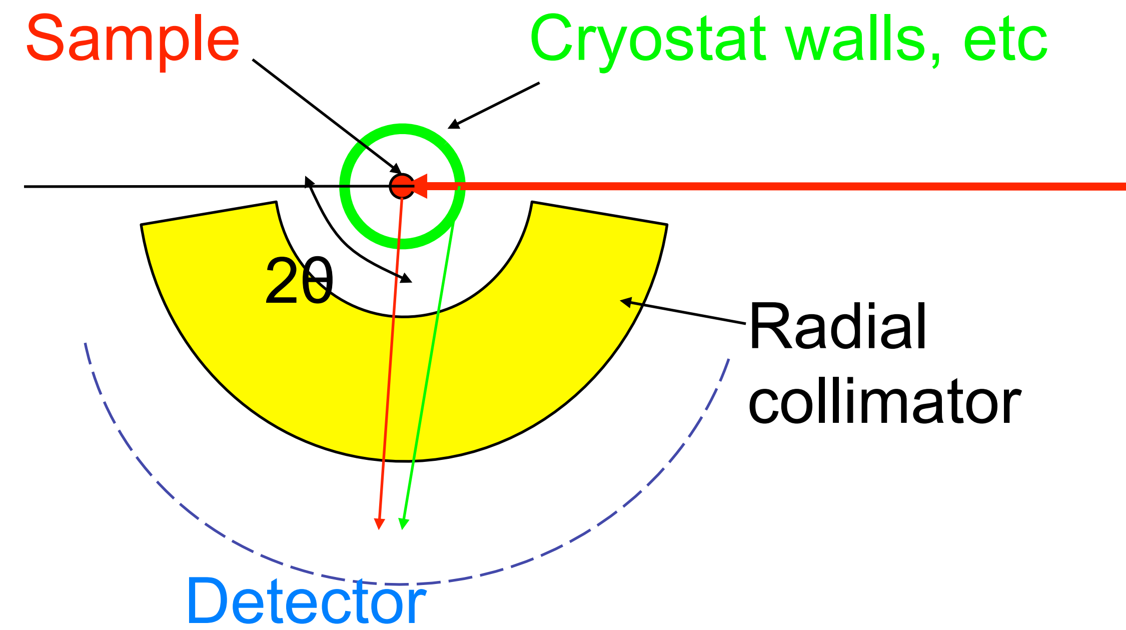


Oscillating radial collimator to avoid scattering from sample environment.



HRPT radial collimators

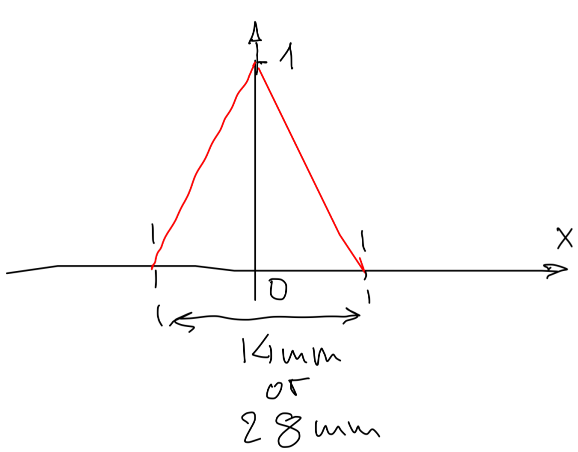


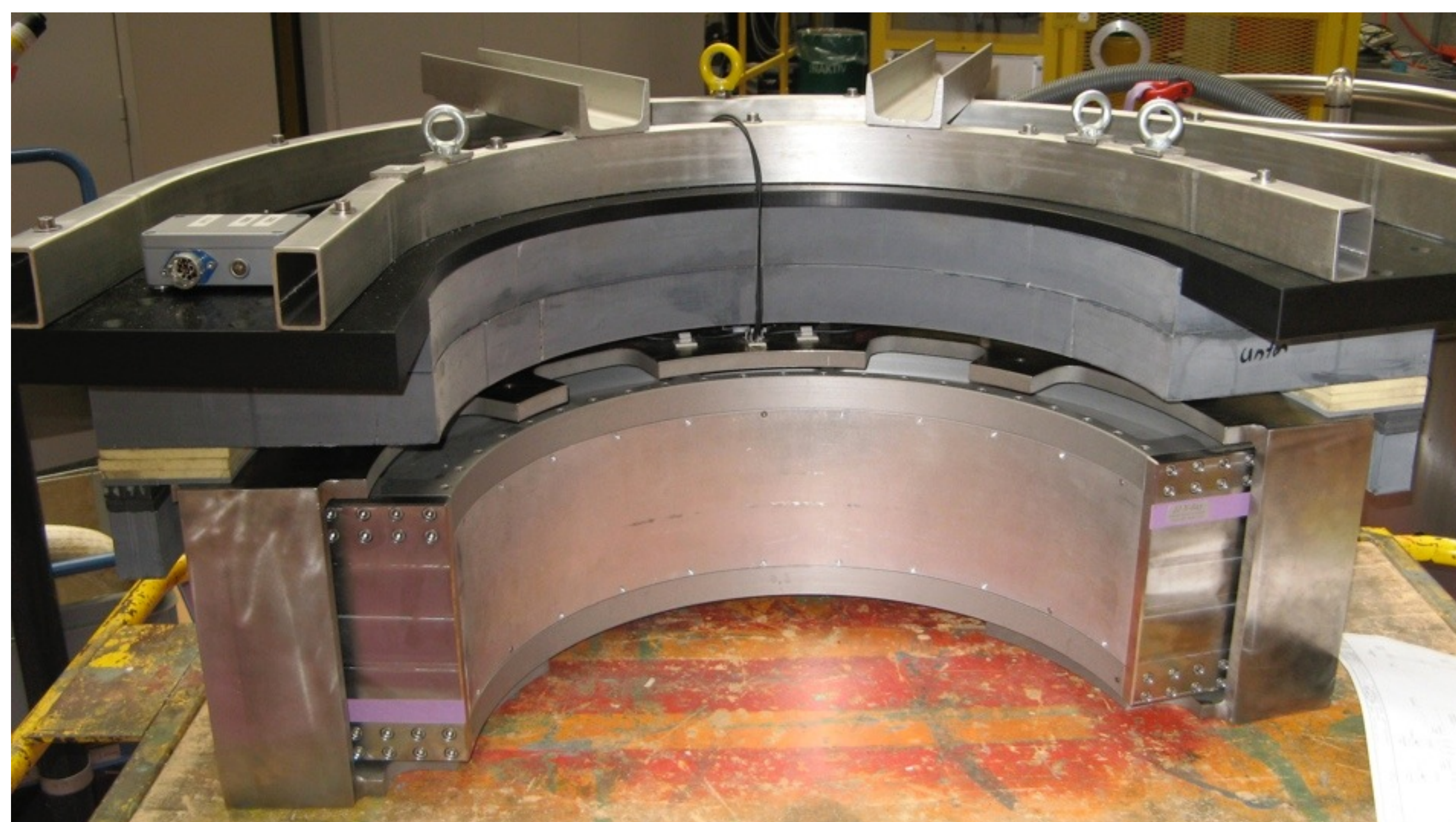


<0.03% for spurious Bragg peaks

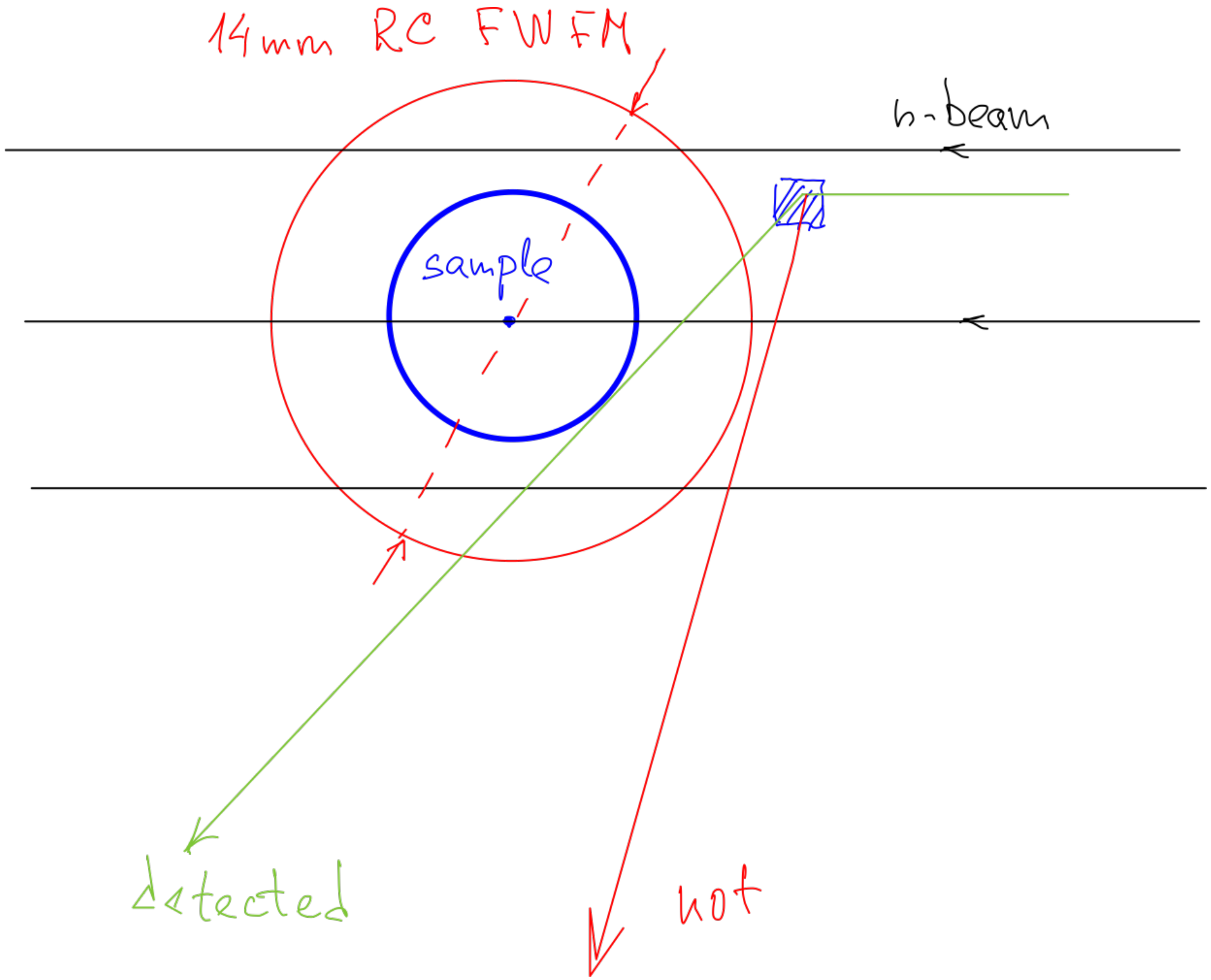
Radial collimator with the shielding.

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

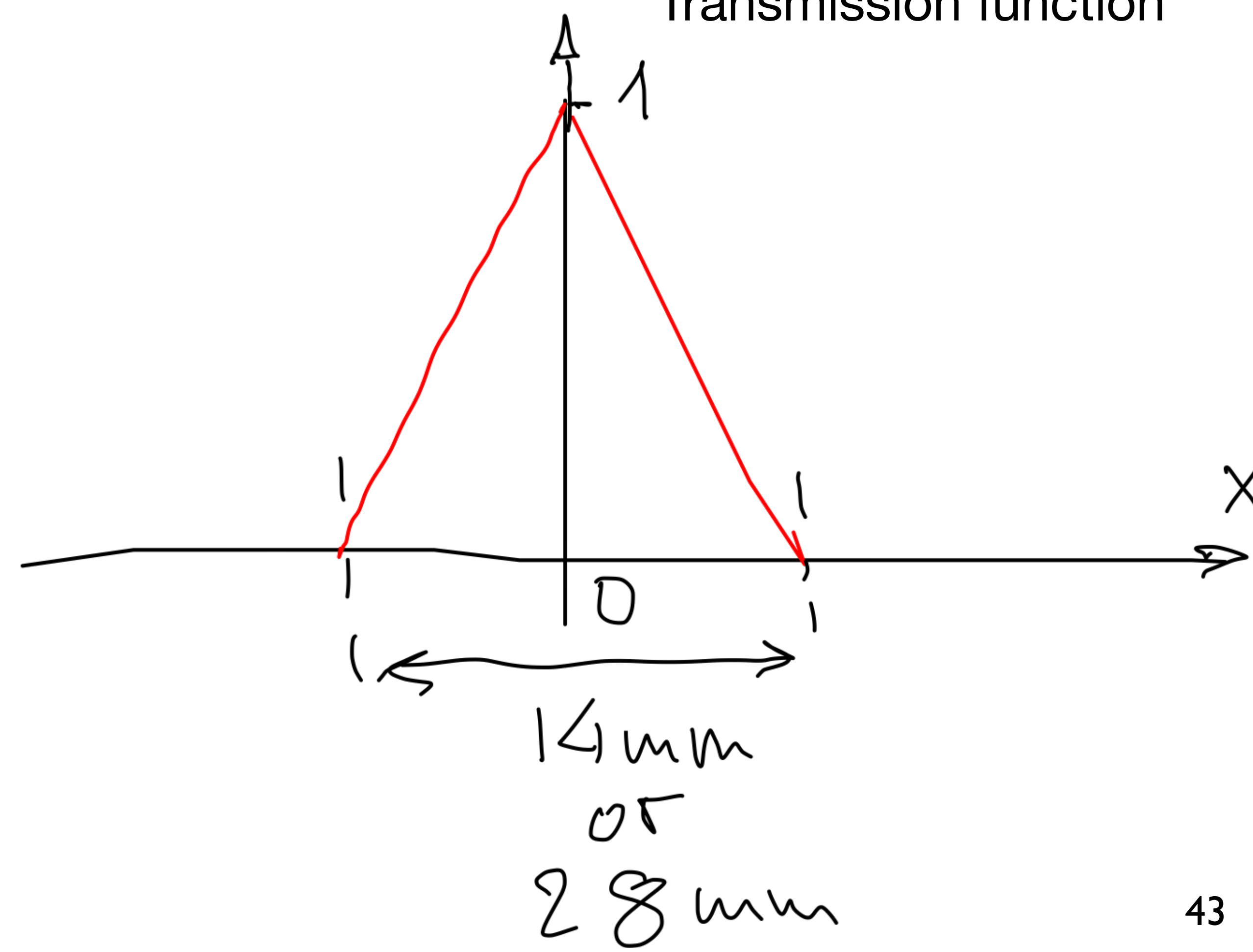




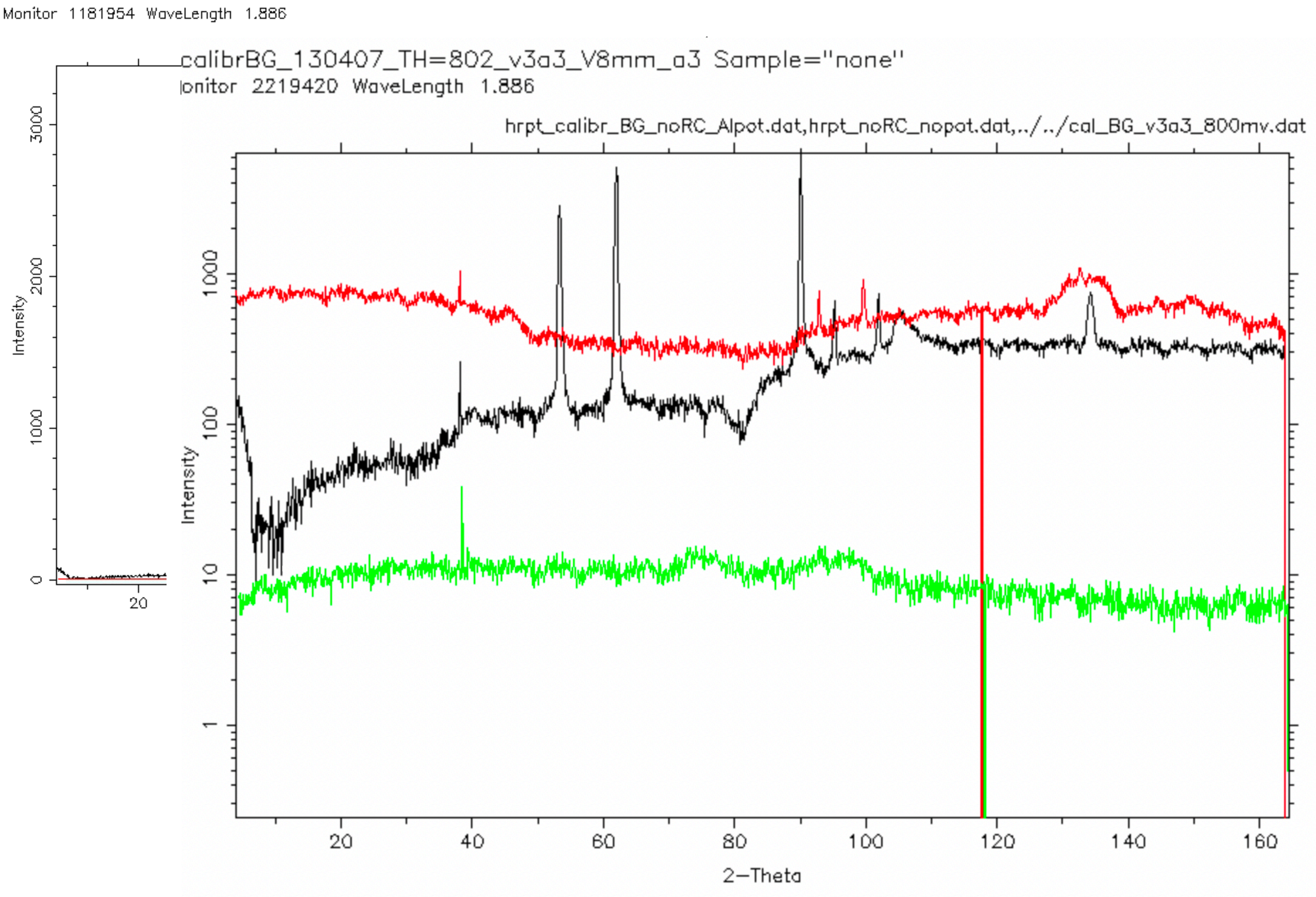
Scheme of radial collimator



Transmission function

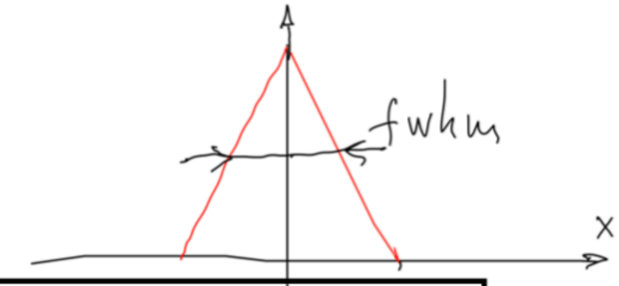


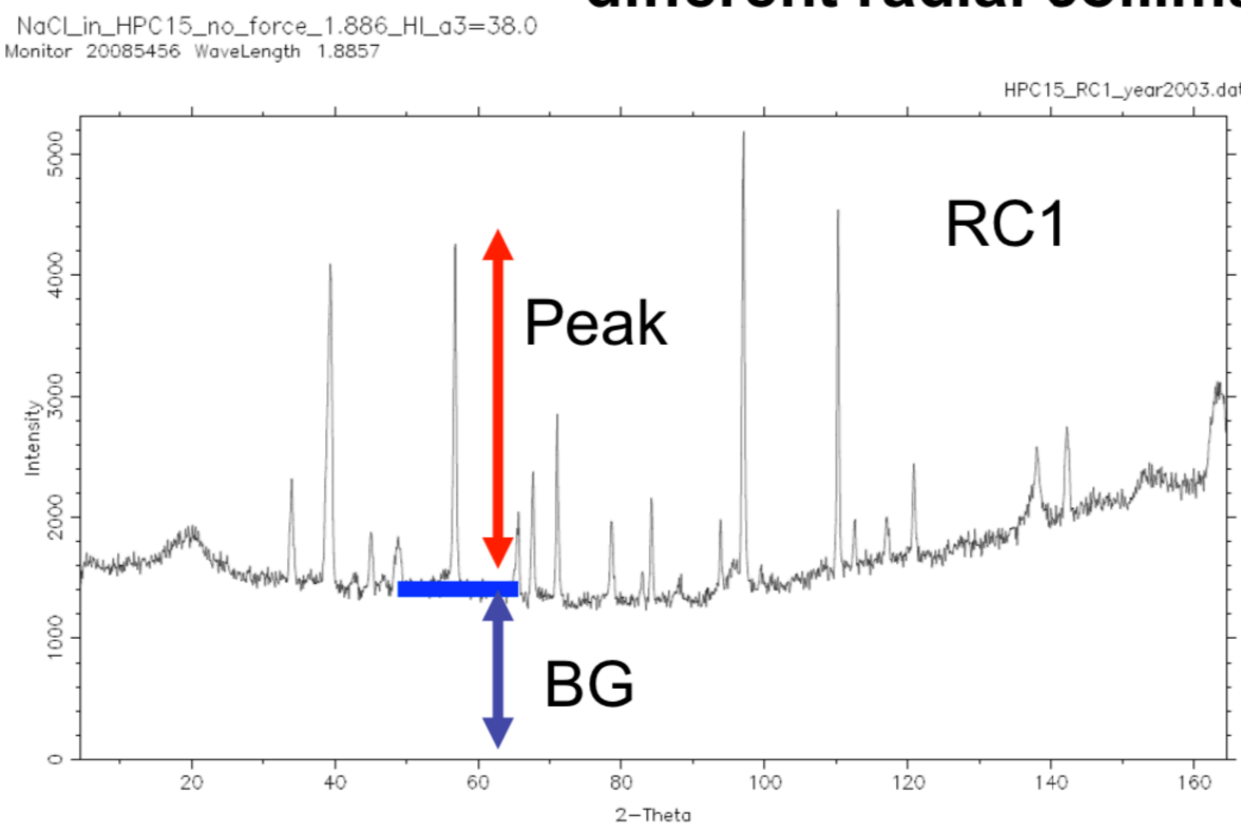
Radial Collimator HRPT (green)



clamp cells for 9 and 14 kbar

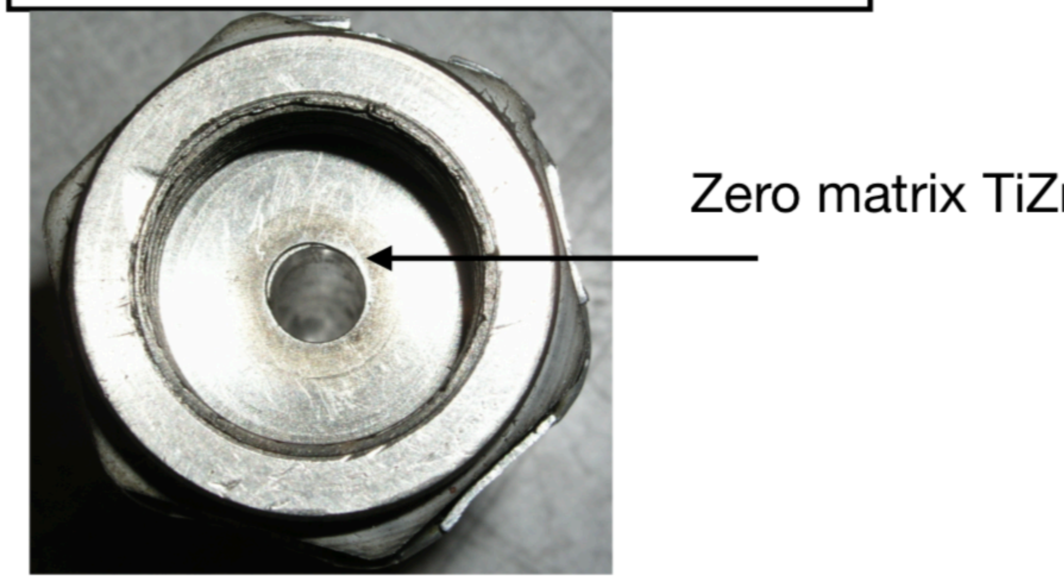
NaCl in High Pressure Cell (HPC15) at HRPT for different radial collimators (RC)

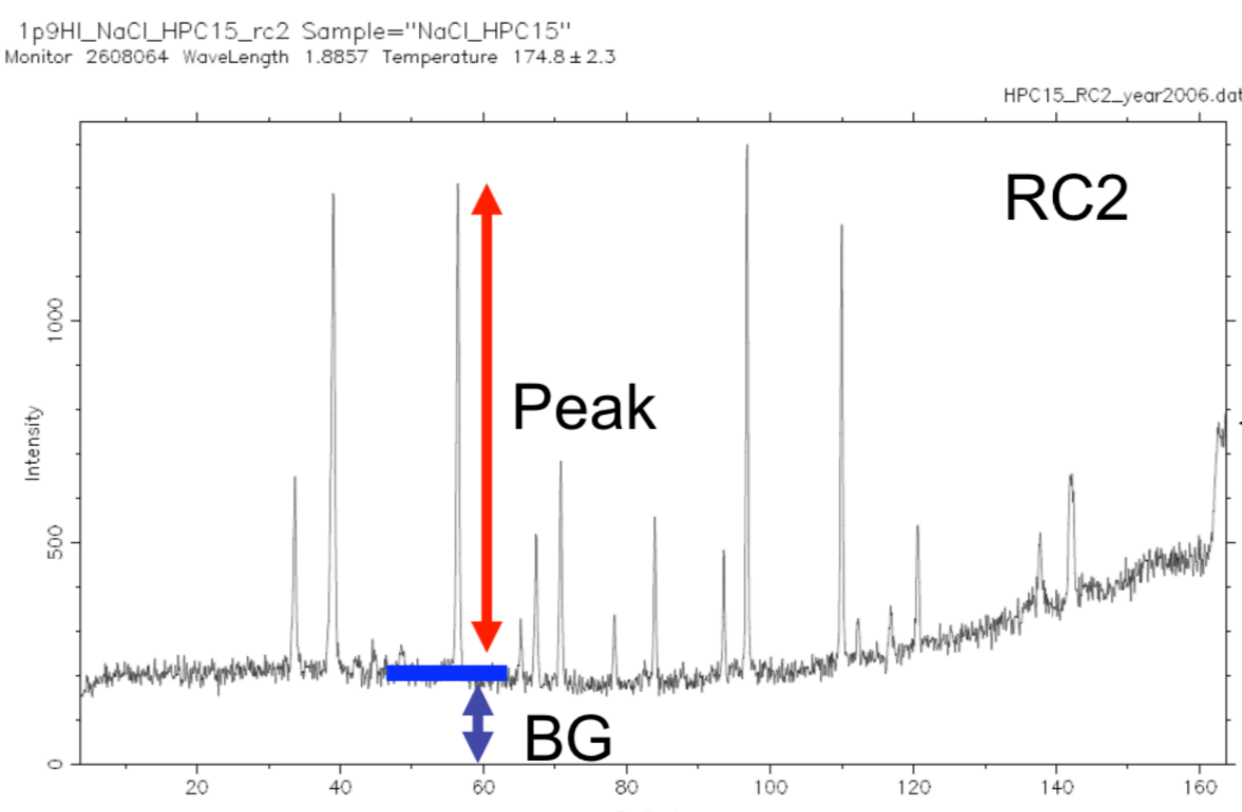




Old RC1 (fwhm=14mm)

Peak/BG=1.85





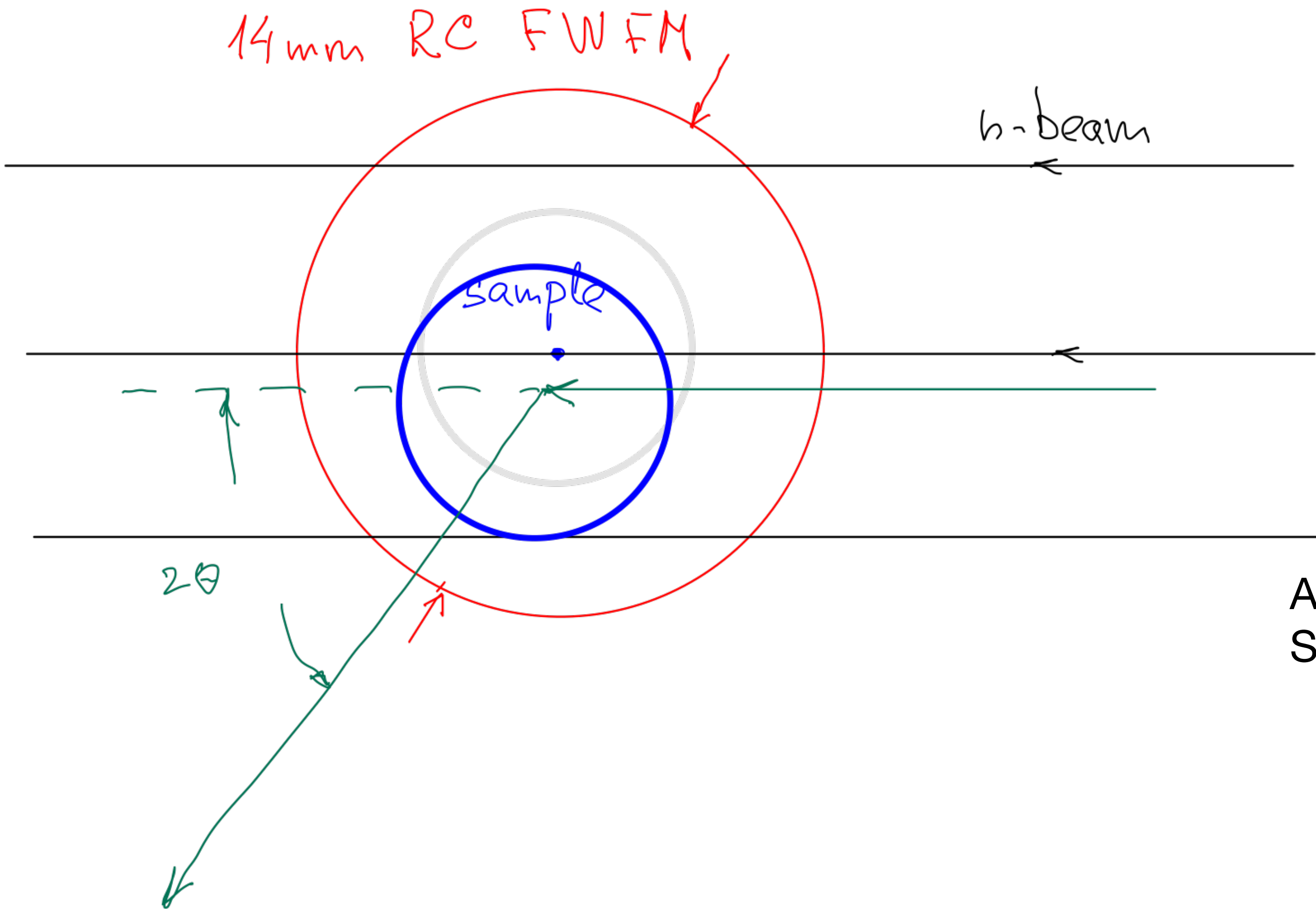
New RC2 (fwhm=7mm)

Peak/BG=5.5 (gain factor 2.9 in comparison with RC1)

Now the Peak/BG ration is similar to one in the Paris-Edinburgh pressure cell (~5 for NiO)

Some drawbacks of radial collimators (RC)

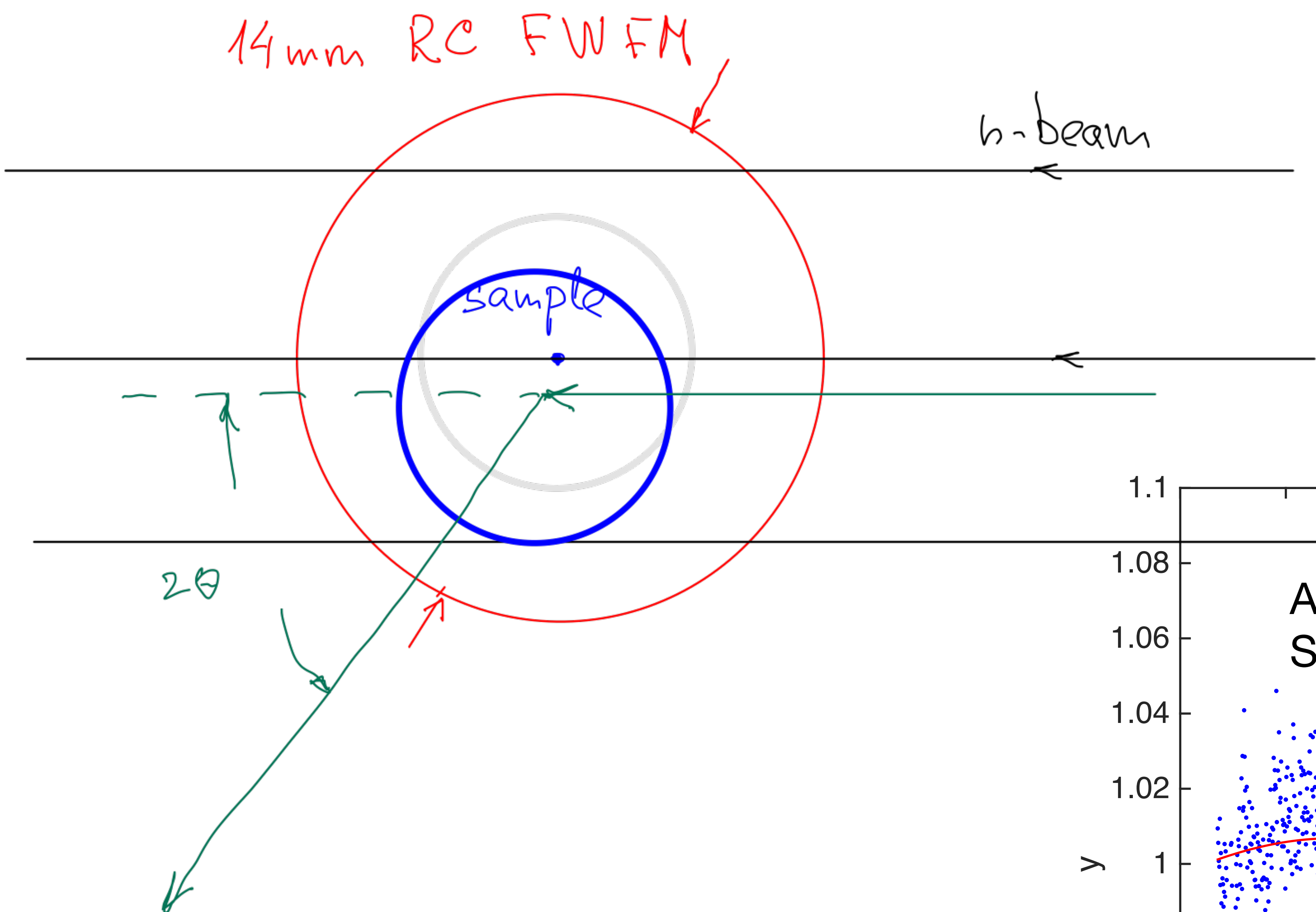
Related to RC and positioning business

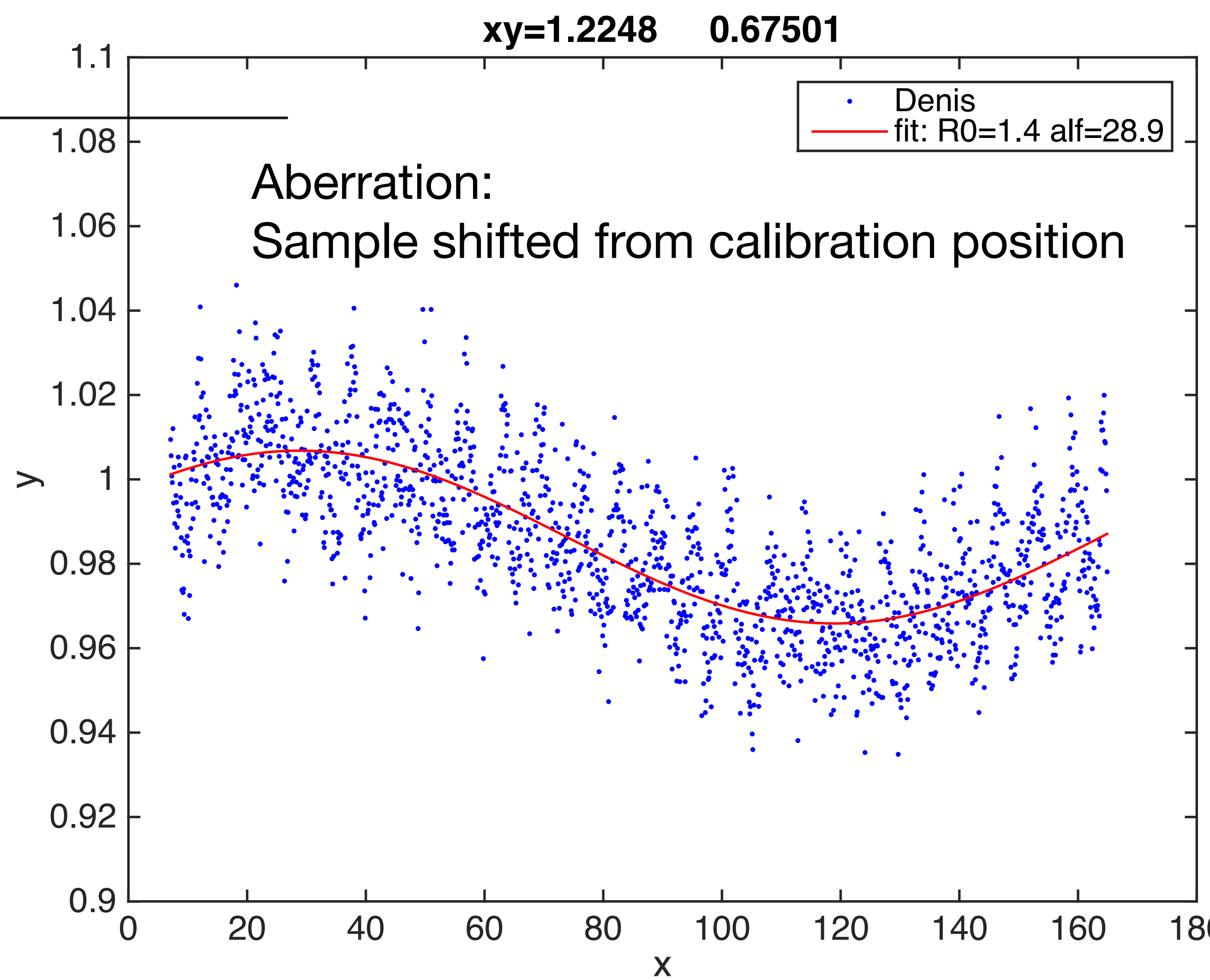


Aberration:
Sample shifted from calibration position

Some drawbacks of radial collimators (RC)

Related to RC and positioning business





Some drawbacks of radial collimators (RC)

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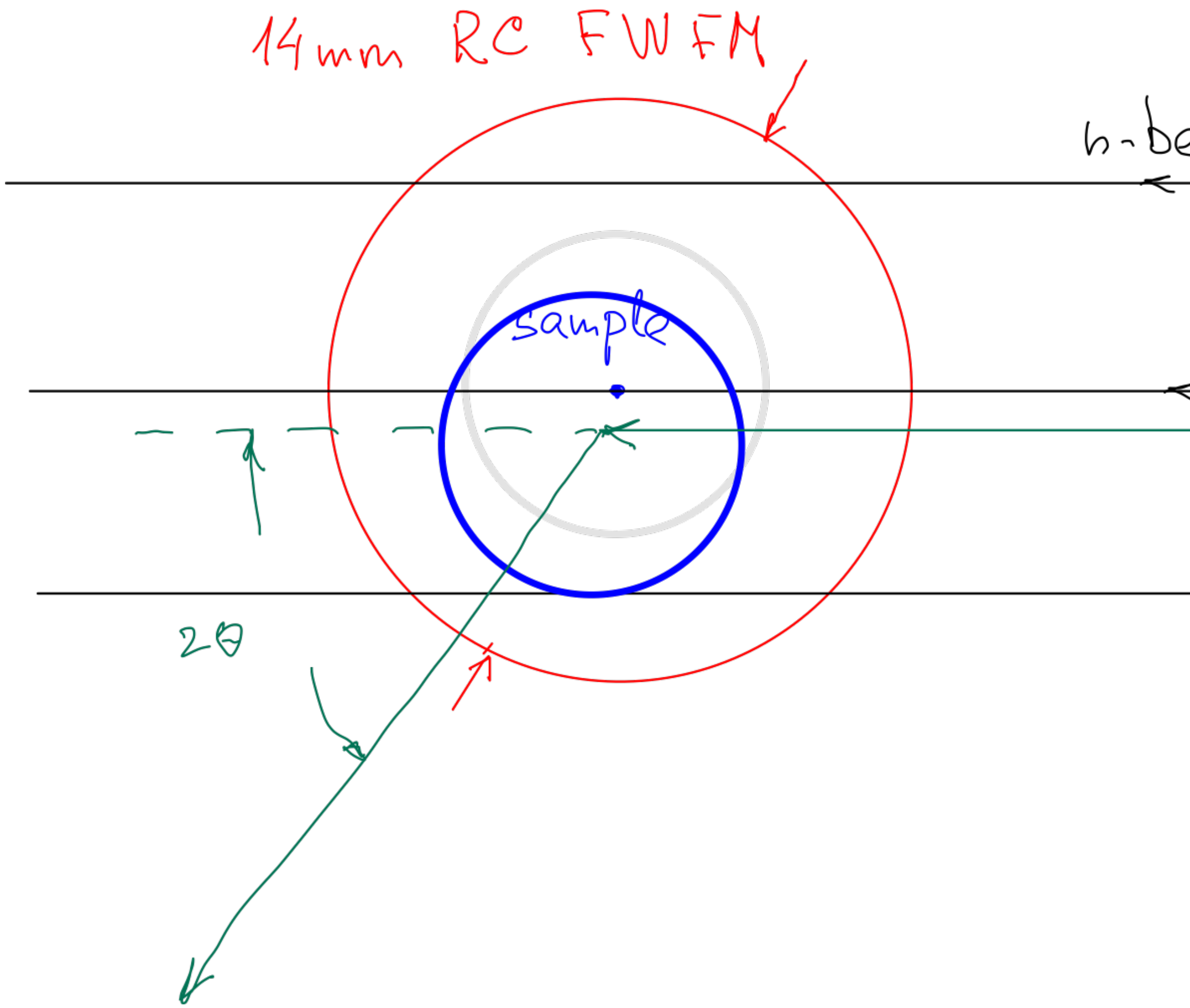
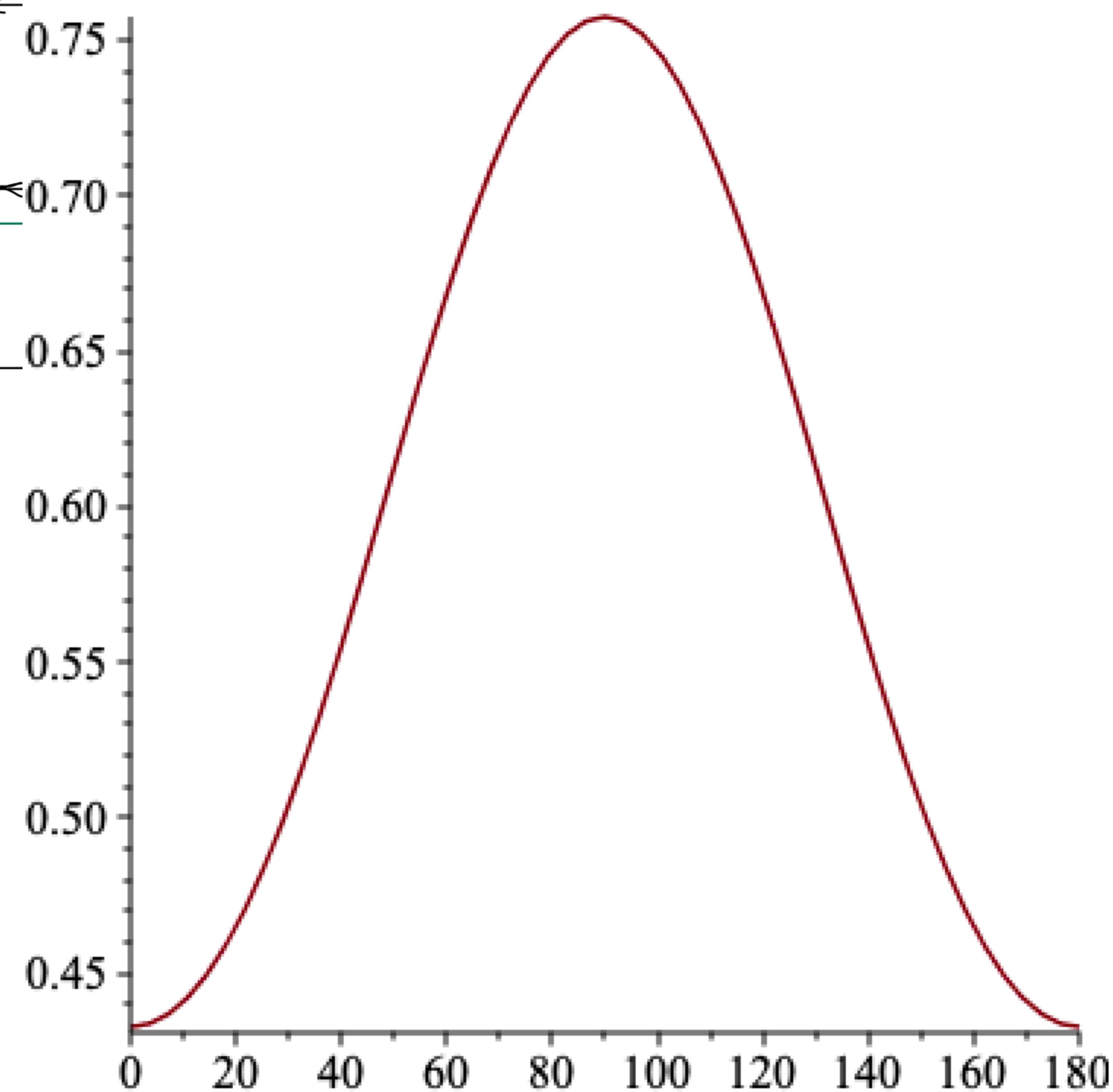
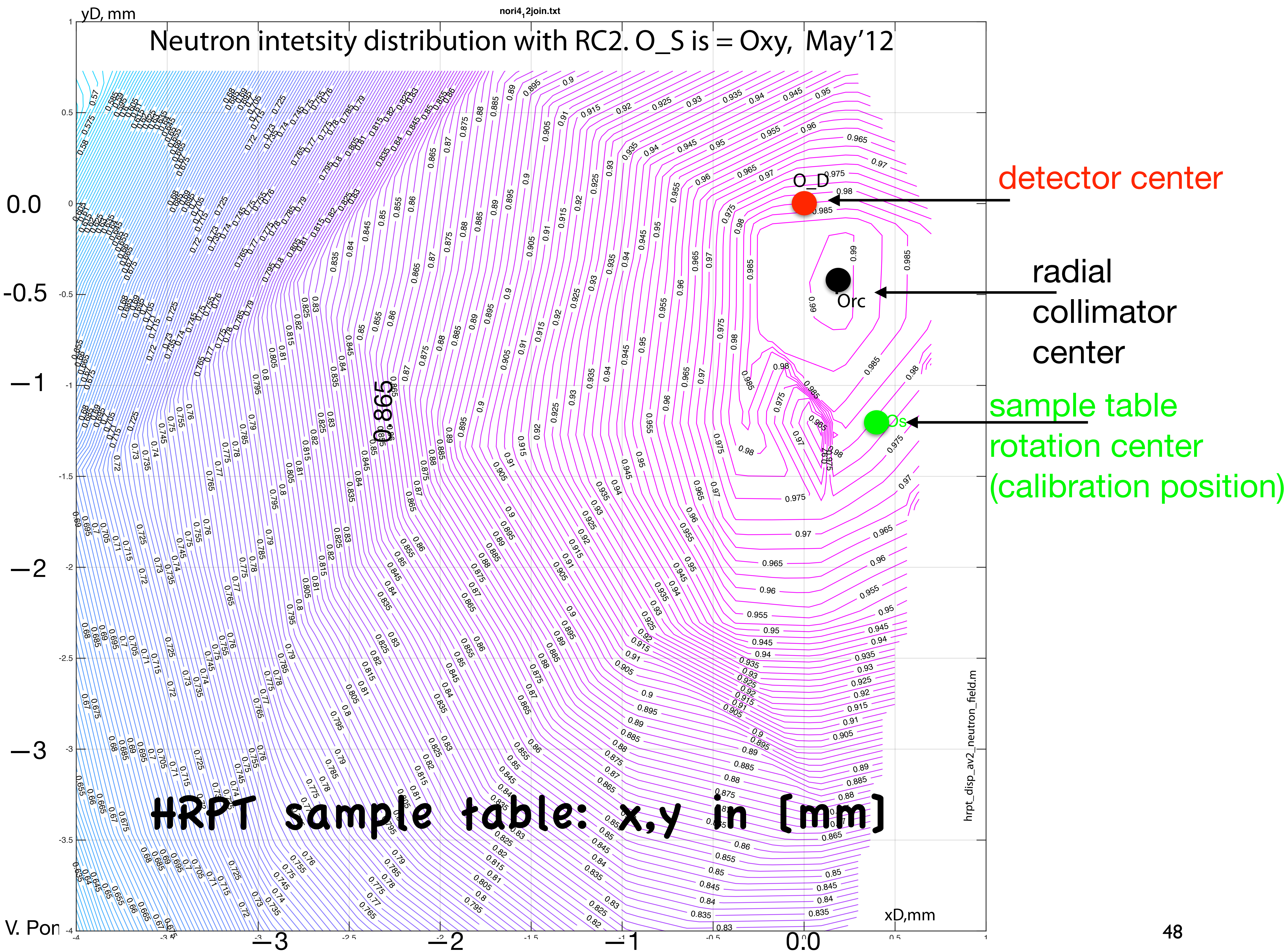
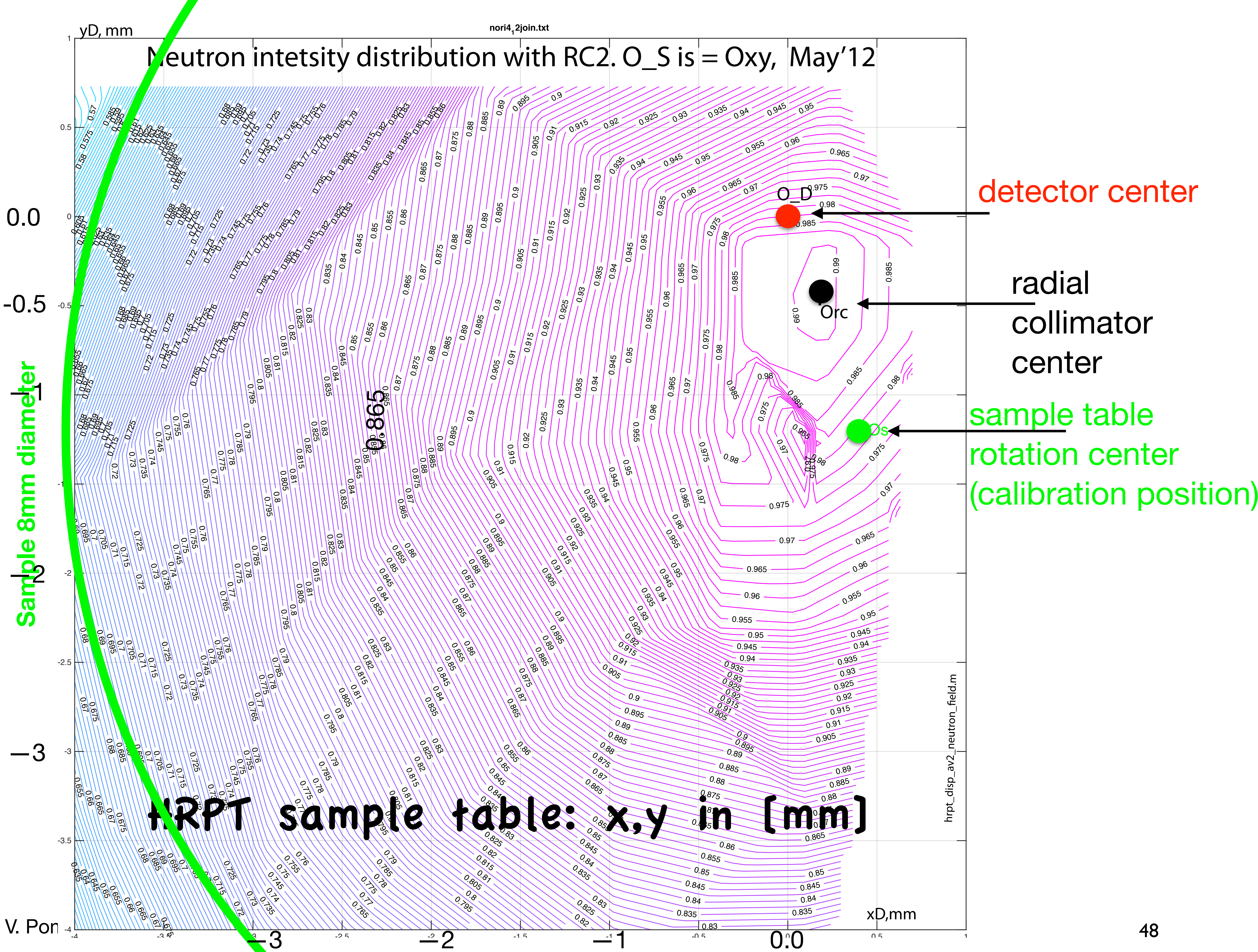


8mm dia sample shifted by 4mm in direction of 2θ=90deg




nori4_2join.txt
Neutron intetsity distribution with RC2. O_S is = Oxy, May'12
yD, mm
xD,mm
O_D
Orc
Os
detector center
radial collimator center
sample table rotation center (calibration position)
HRPT sample table: x,y in [mm]
hrpt_disp_av2_neutron_field.m
0.0
-0.5
−1
−2
−3
−3
−2
−1
0.0

V. Pon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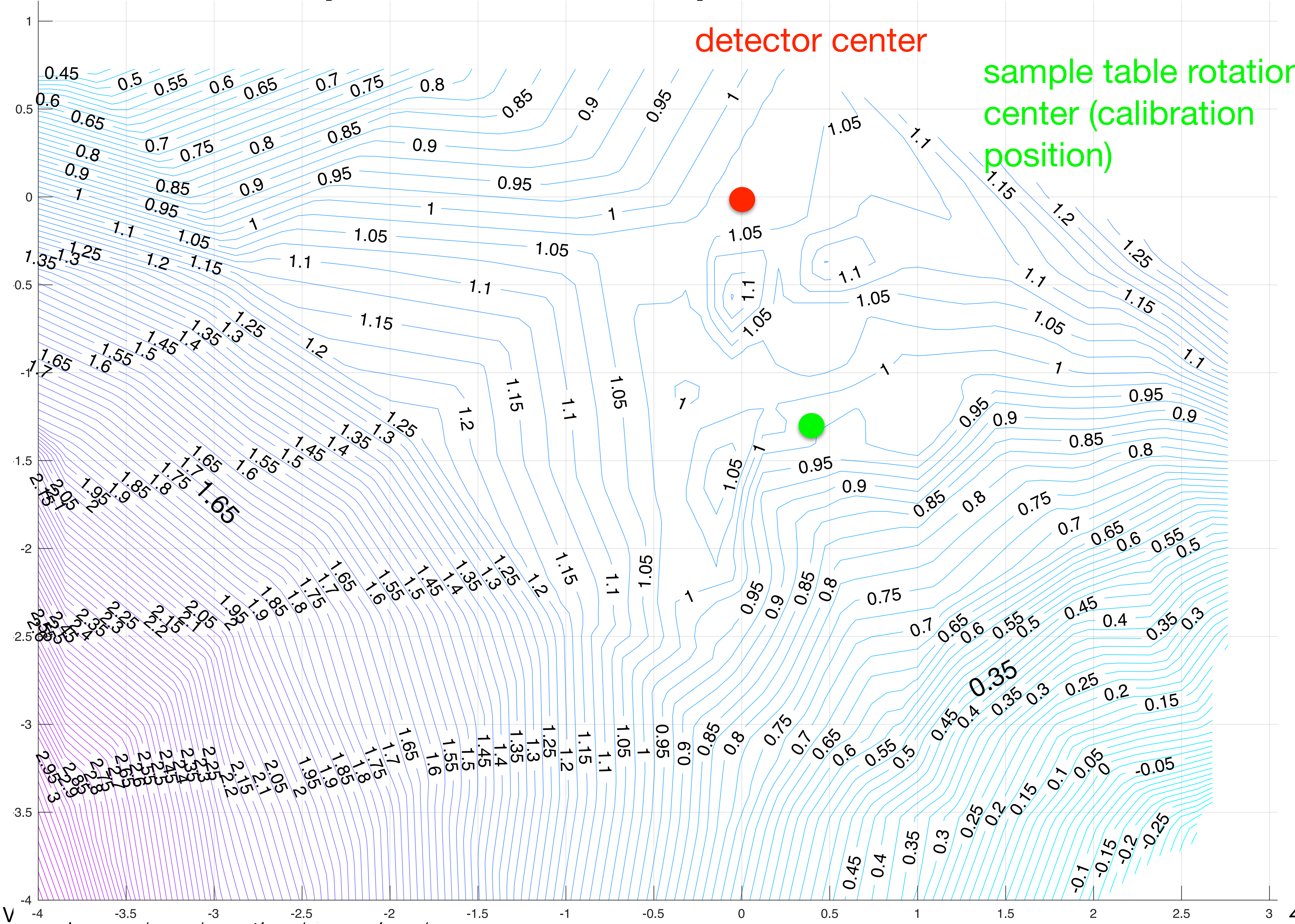
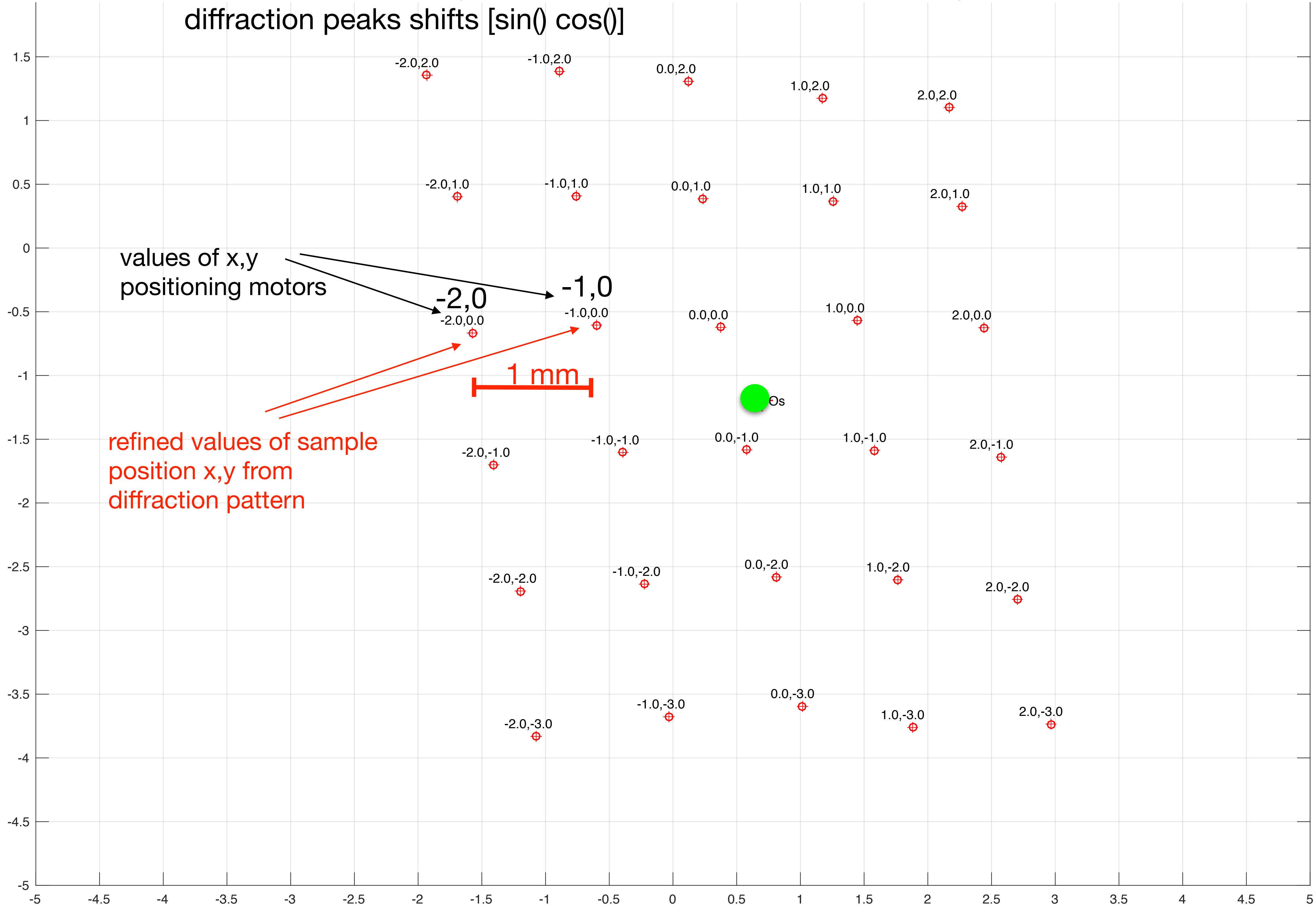
nori4_2join.txt
yD, mm
xD,mm
0.0
-0.5
−1
−2
−3
−3
−2
−1
0.0
O_D
detector center
Orc
radial collimator center
Os
sample table rotation center (calibration position)
Sample 8mm diameter
0.865
HRPT sample table: x,y in [mm]
hrpt_disp_av2_neutron_field.m
V. Pon

average Debay-Waller ADP(x,y) of $Na_2Ca_3Al_2F_{14}$ at 1.9A



precise sample positioning with respect to calibration

We can determine by diffraction the (x,y) position of sample with the accuracy better than 0.1mm! by the detector (radius 1500mm) from systematic diffraction peaks shifts [sin() cos()]



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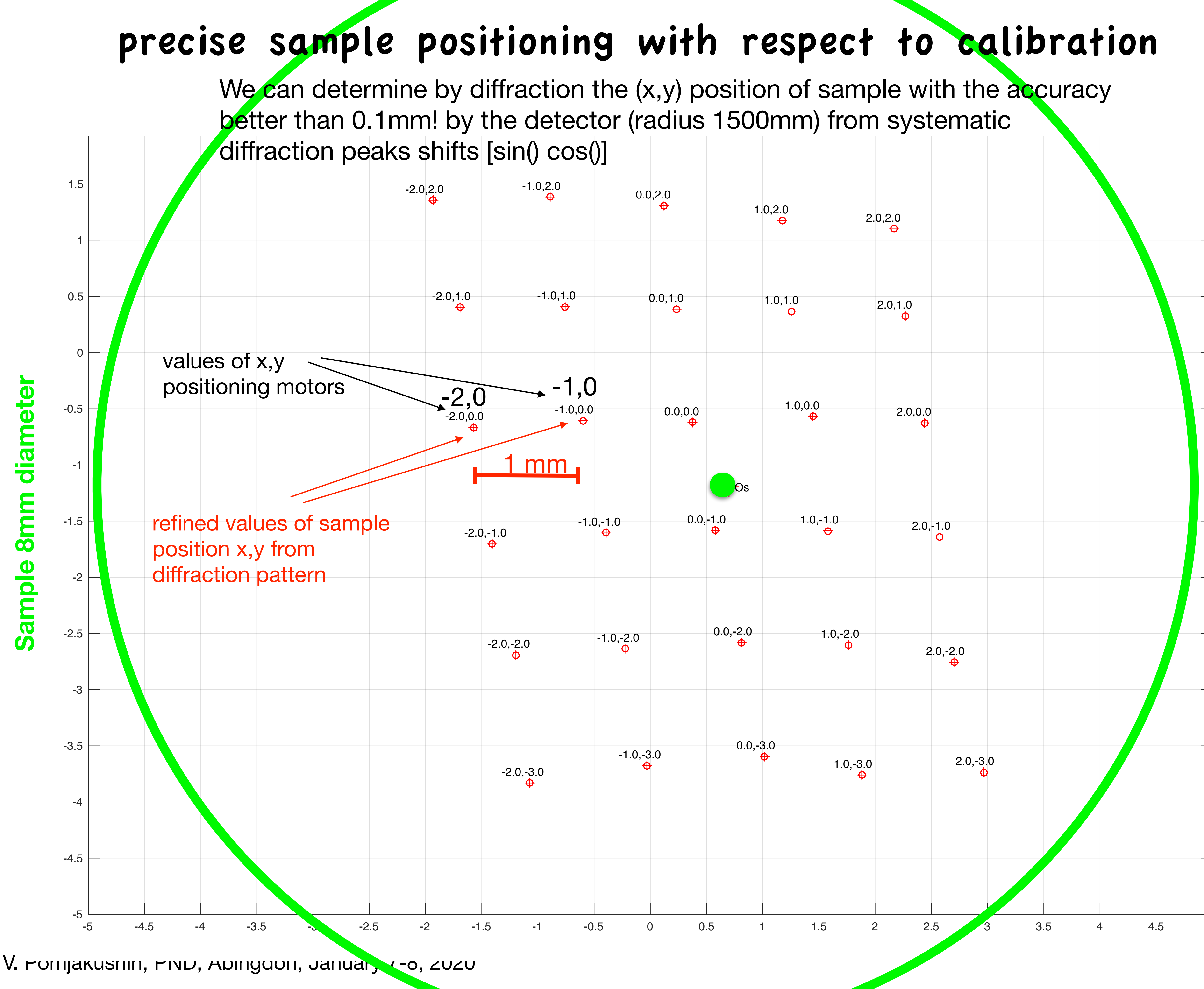

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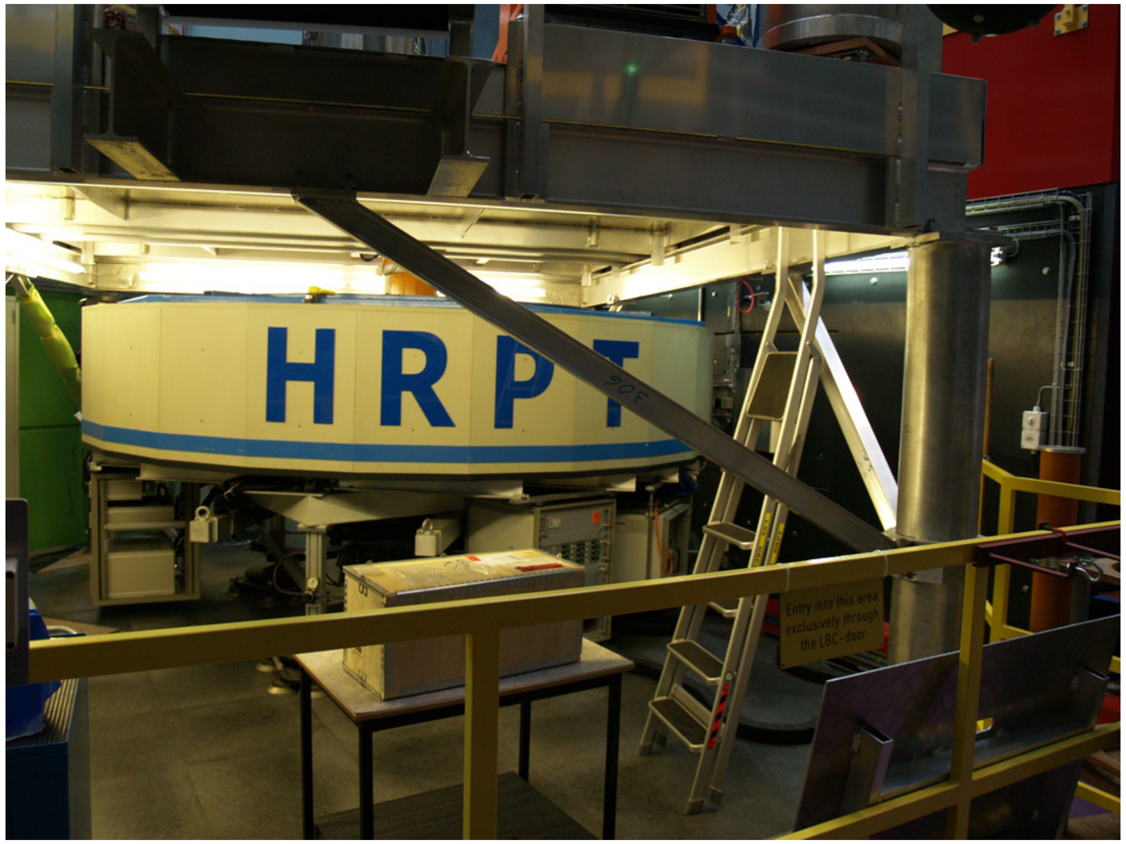
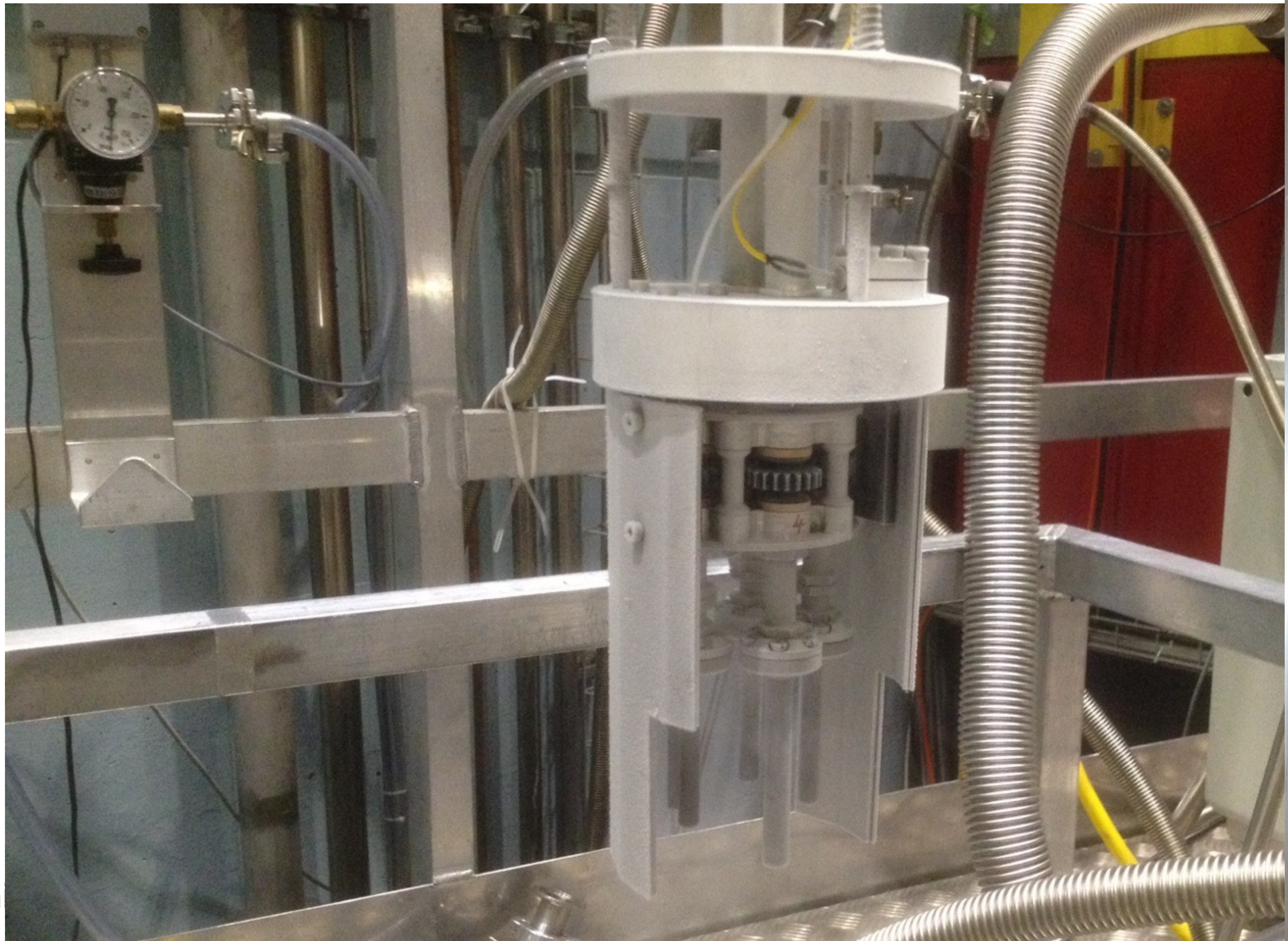


HRPT low temperature 5-sample changer

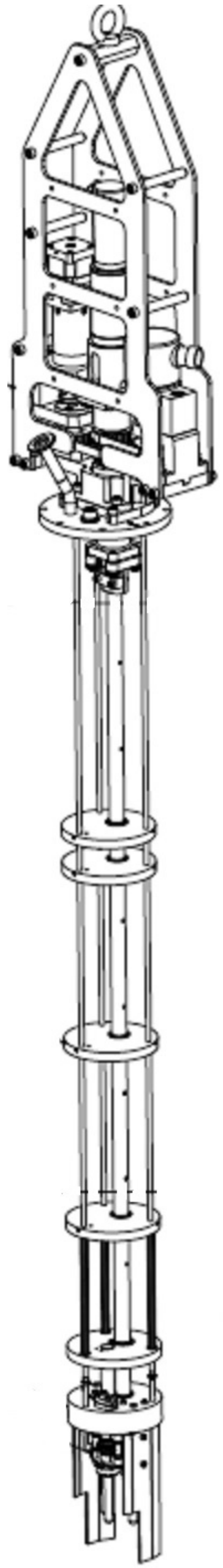
A device for routine powder diffraction measurements at temperatures between 1.5K -300K.

- All samples have the same temperature, i.e. time for temperature change is saved;
- Five samples mounted on a caroussel-type changer, that is a special inset for an orange cryostat
- The sample is rotated to avoid preferred orientation and achieve “ideal” centering

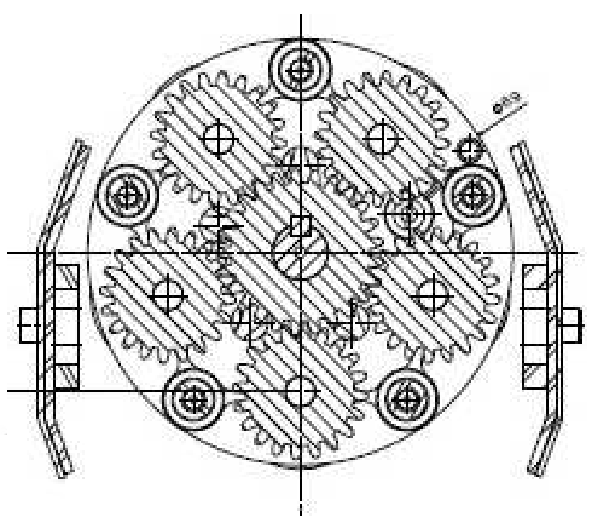


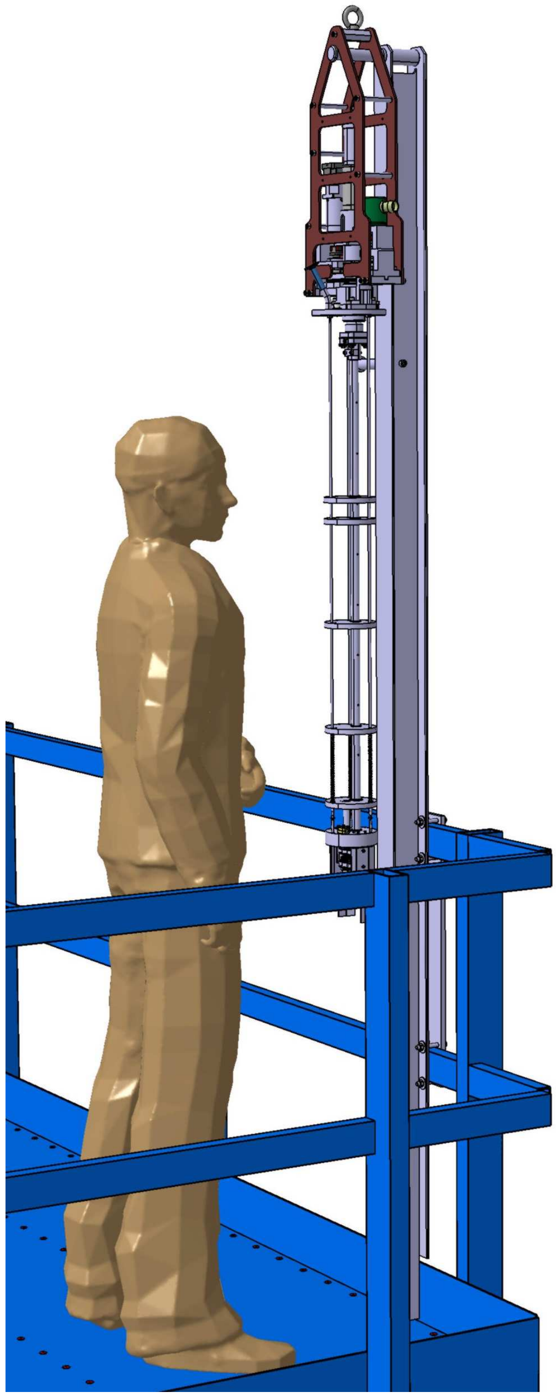
V. Pomjakush

HRPT-Probenwechsler

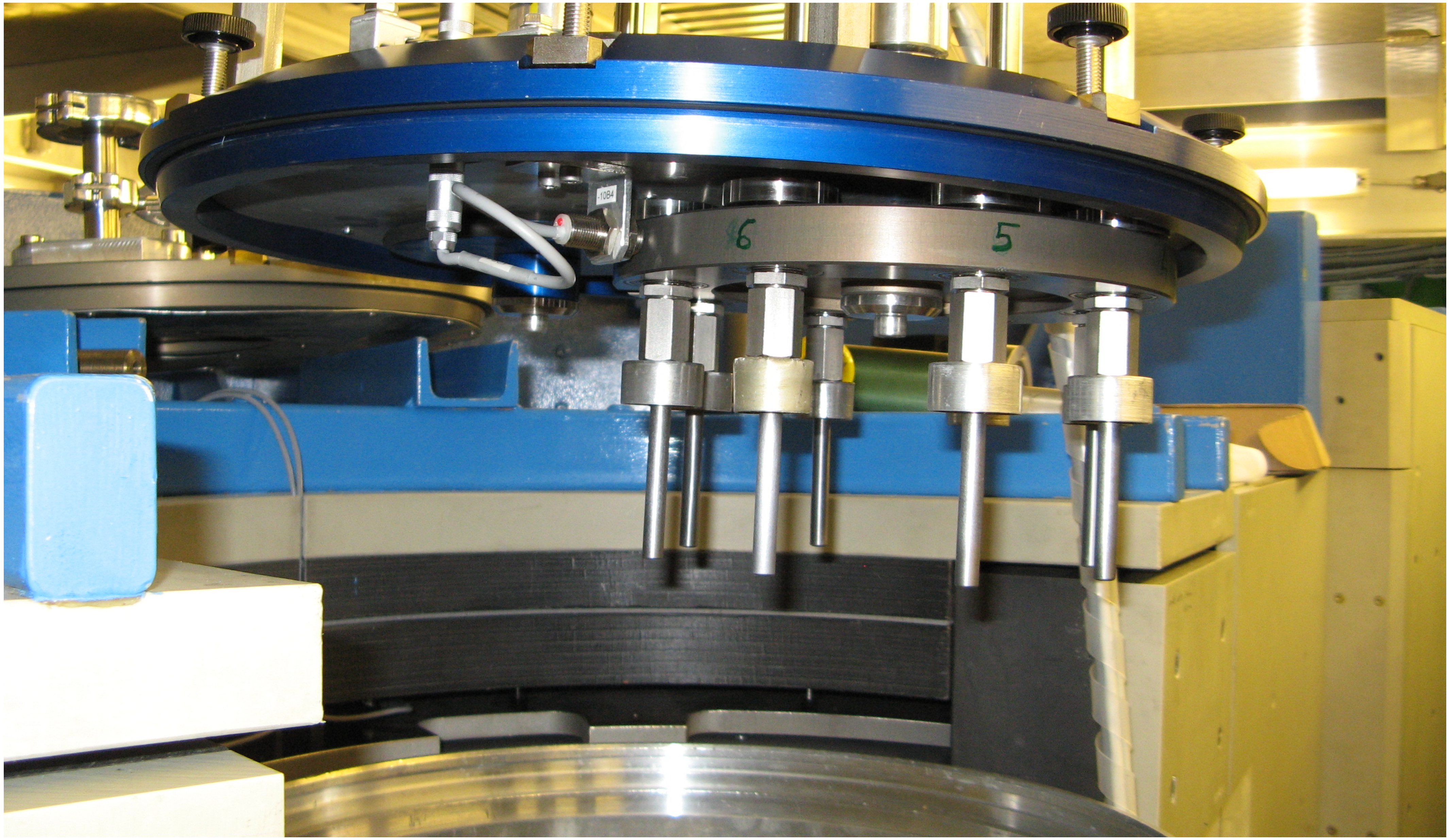


- 1.5 K
- 5 wechselbare Proben
- Zusätzlich rotierende Probe
- Geringes Getriebespiel bei Einsatz unterschiedlicher Materialien



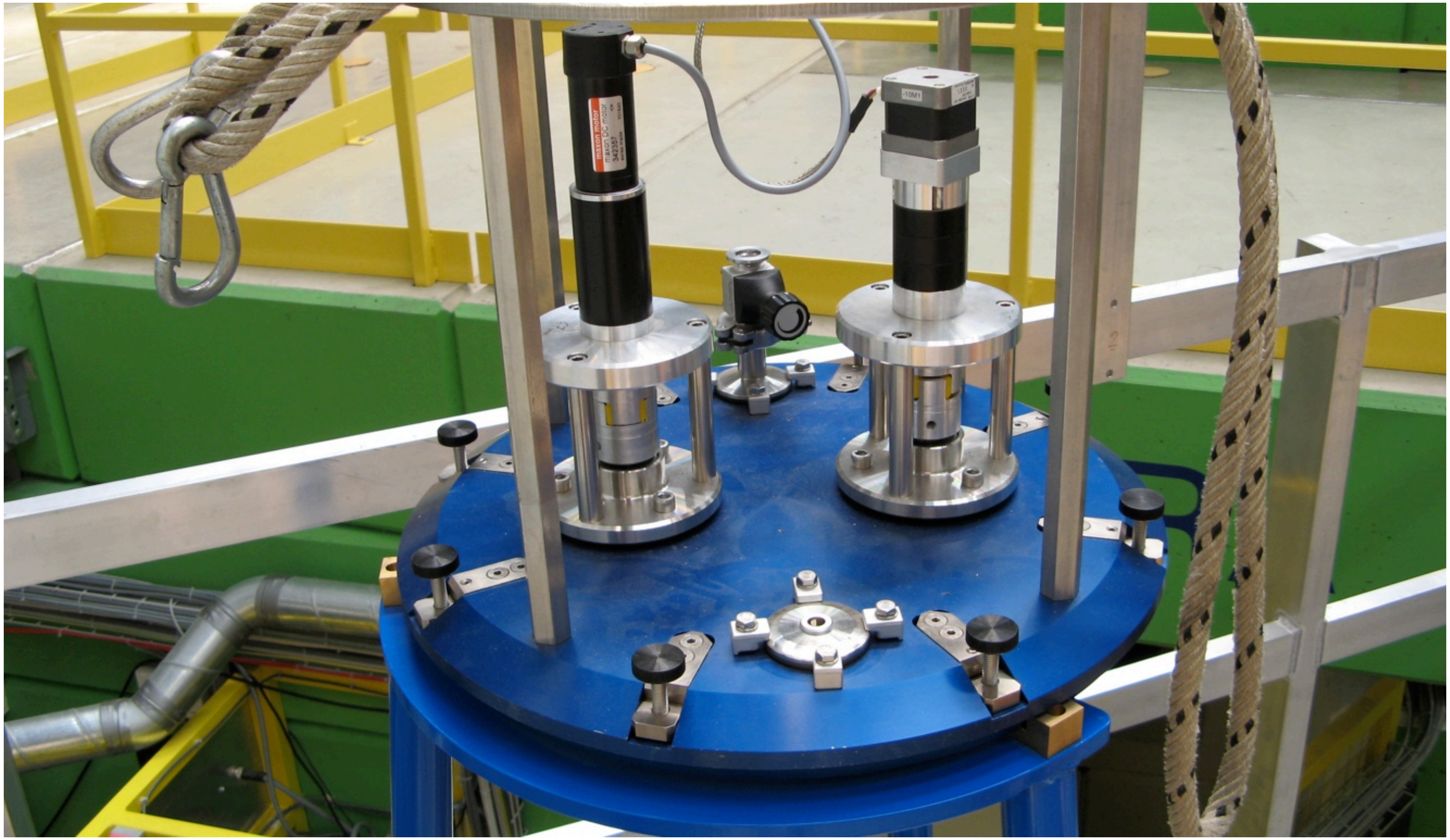


HRPT room temperature 8-sample changer



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

HRPT room temperature 8-sample changer



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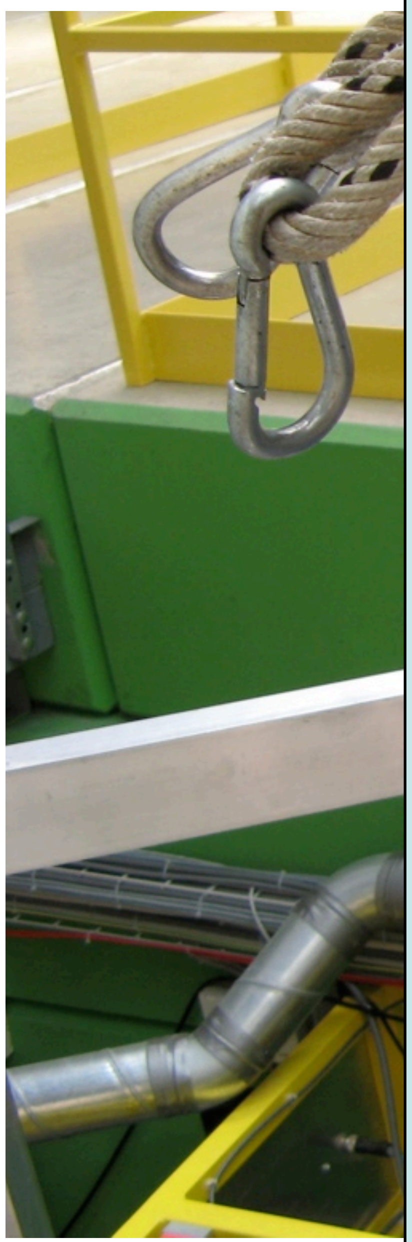
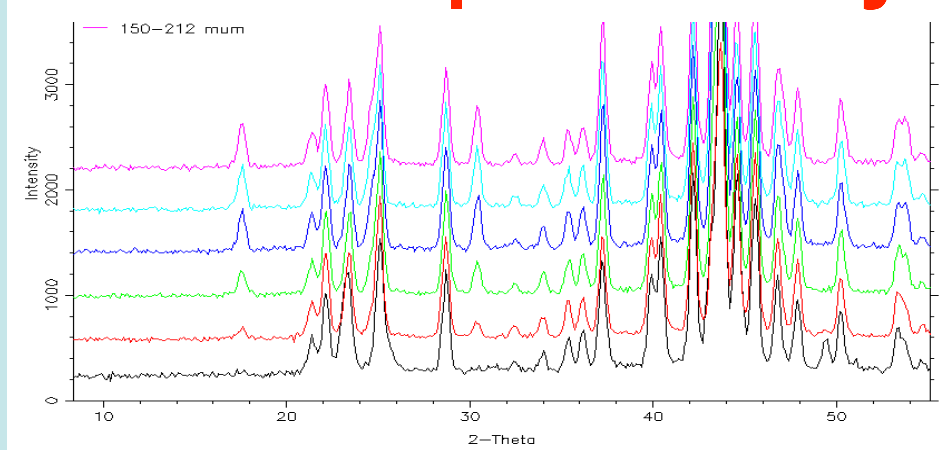
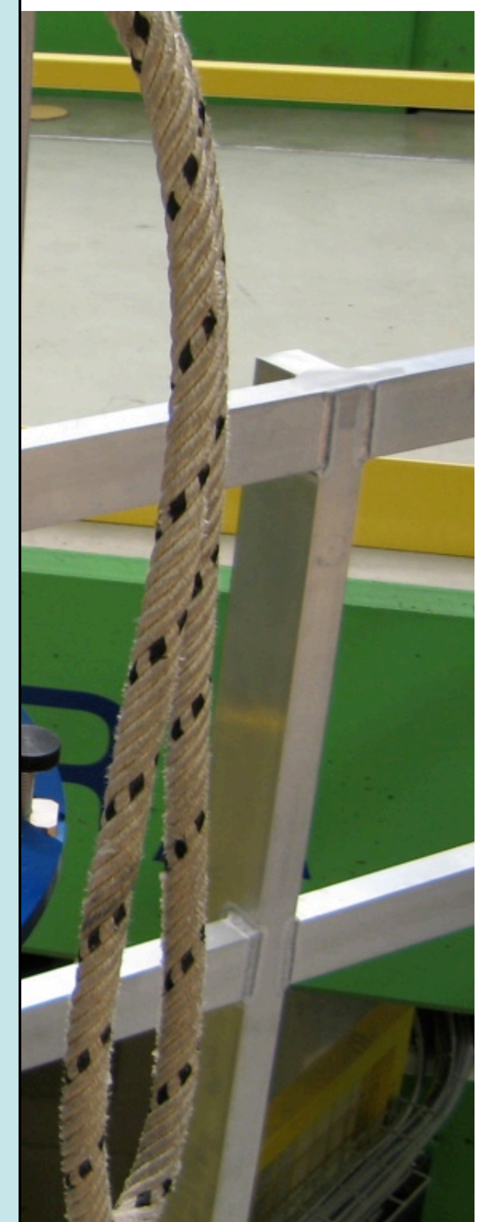
HRPT room temperature 8-sample changer



User Experiment 20061119
“Structure of leached Raney Ni alloys” (Nov. 2007):
~80 samples measured in 4 beam days:

20 samples/day!





Fully loaded with 8 samples

HRPT sample table.

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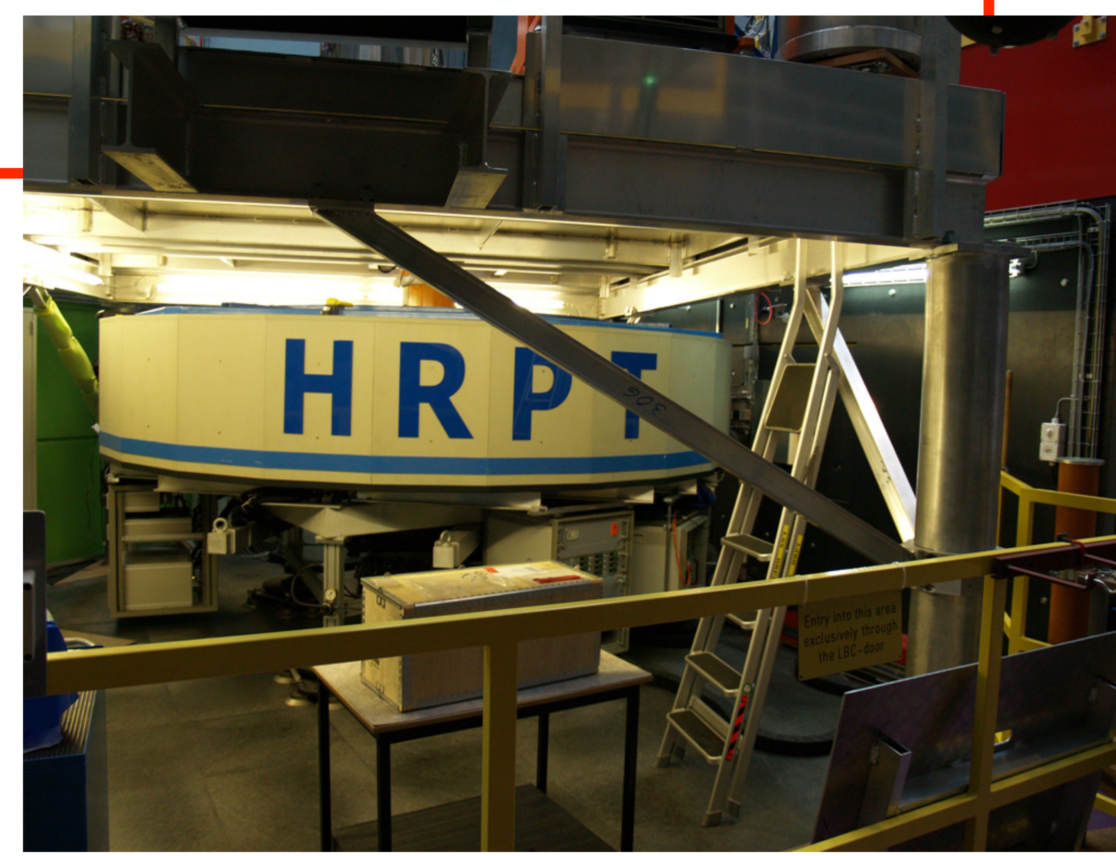
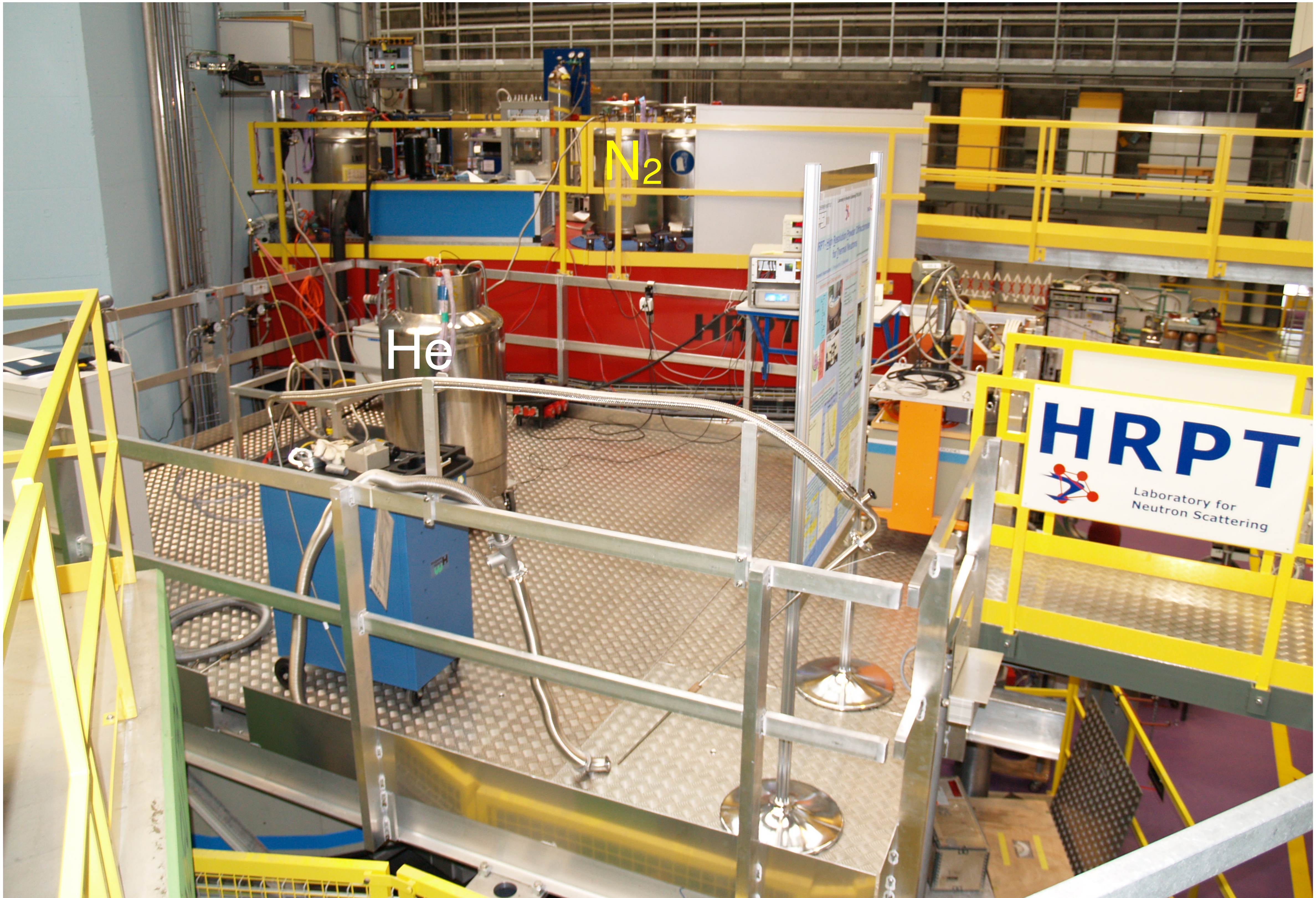
- due to low background small samples can be measured (30 mm^3)
- Radial collimators
- Sample changers 4-8 samples, T=1.5-300 K
- standard LNS sample environment:
  - Temperature = 50 mK—1800K,
  - Magnetic field H = 6 T (vertical)
  - Automatic He, N_2 refilling systems
- zero matrix high pressure cells:
  - clamp cells for 9 and 15 kbar
  - Paris Edinburgh cell 100 kbar



Automatic He, N_2 refilling systems using temperature sensors in cryostat. Computer controlled with remote access



Samples, T, P, H and other equipment at HRPT/SINQ

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- Completely automatic experimental control system and the data reduction by Perl scripts. End user get datafiles and logs with all necessary experimental conditions. No run-numbers anymore. Remote control from anywhere.

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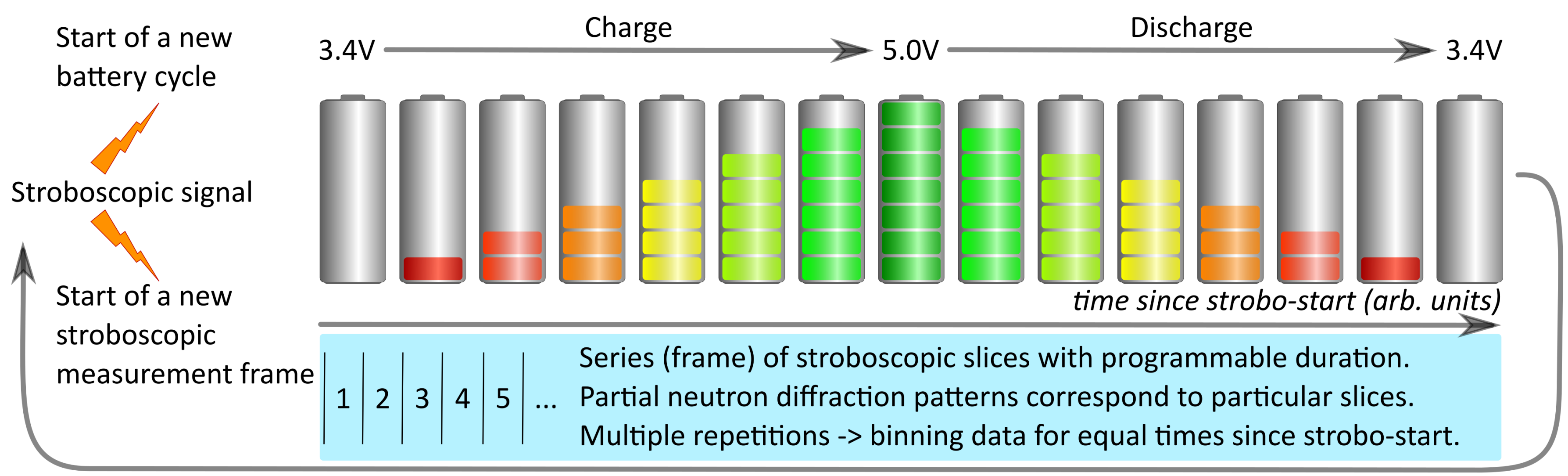
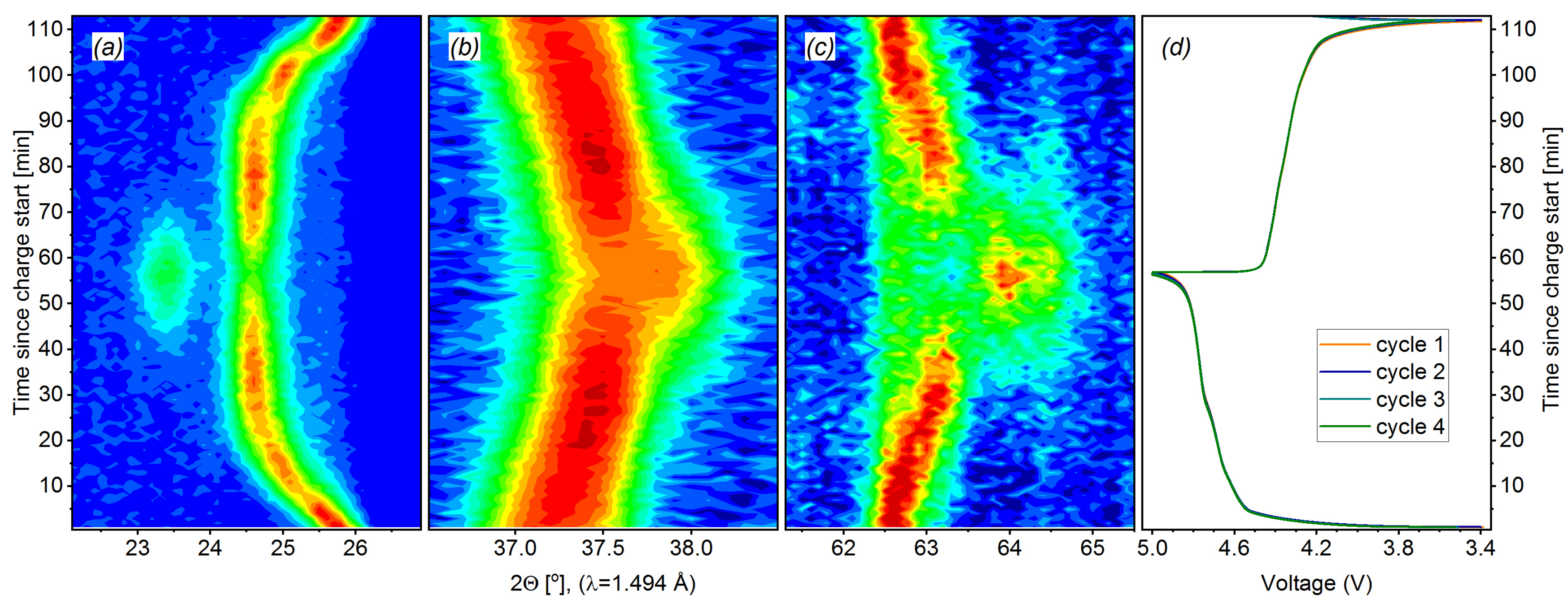
- Stroboscopic mode of operation

HRPT: stroboscopic mode of operation, time slices down to 20 ms





Application to study the ageing mechanisms in the industrial-sized real batteries.
The 112 patterns above (each just 1 minute) are a merge of 4 consecutive charge-discharge cycles, and are having the quality sufficient for Rietveld refinement.

D. Sheptyakov, L. Boulet-Roblin, V. Pomjakushin, C. Villevieille et. al., in press.

My wish/thoughts list

- ND Resolution to <=10^{-4} (possible for both CW and TOF), to be able to study what?
  - the magnetic structure forces lower symmetry space group, which change in metric 10^{-3} - 10^{-4} and smaller. Many examples…
    - Transitions in multiferroics: small distortions in subgroup, often <<10^{-3}
    - crystallographic twins/magnetic domains in single crystal ND: if unresolved mimic the powder averaging
  - intrinsic phase separation, HTSC, AFeSe, …

- Q-range for both crystal structure and magnetic multipoles, beyond dipole approximation.

- analyser in the scattered beam to get only elastic scattering.

- Sample changers, completely computer controlled experiments and data analysis in case of “predictable” results.

Thank you!