Mitglied der Helmholtz-Gemeinschaft

POWHOW

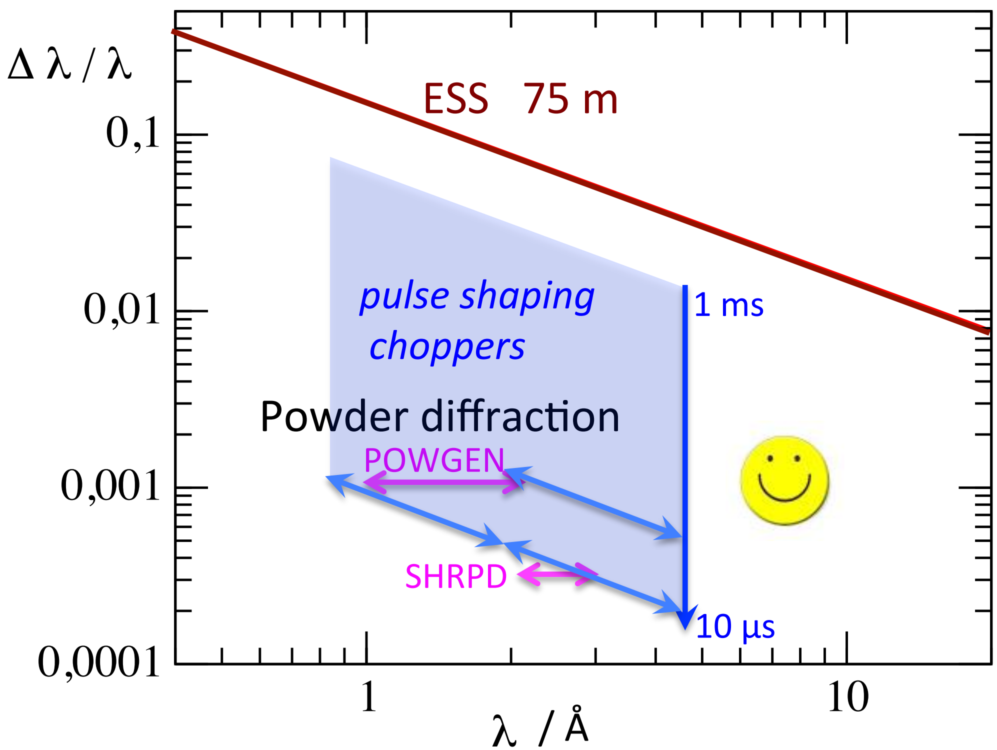
Bi-spectral Powder Diffraction at the ESS

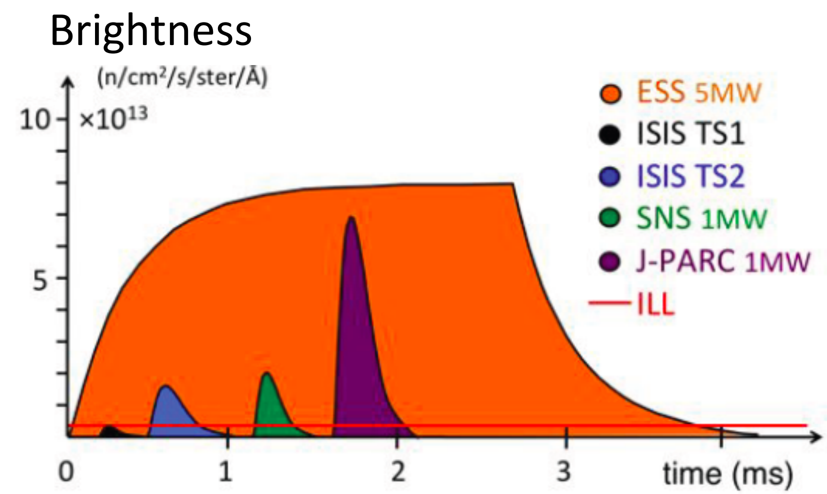
W. Schweika, N. Violini
K. Lieutenant, D. Nekrassov, C. Zendler
A. Houben
P. Henry

IKON5 Lund September 24, 2013

Powder Diffraction at the ESS

time – wavelength resolution





high flexibility in trading resolution *versus* intensity
ideal peak shape

Science

Chemistry **Physics** **Materials Science**

light elements *O-N-F, H*
combined with heavy elements

magnetism
low T physics
multiferroics

close to application
in-situ parametric studies of complex samples
batteries
multiple phases
large unit cells
multiple length scales

=> **thermal and cold neutrons**

bispectral powder diffractometer

Instrument scheme and components

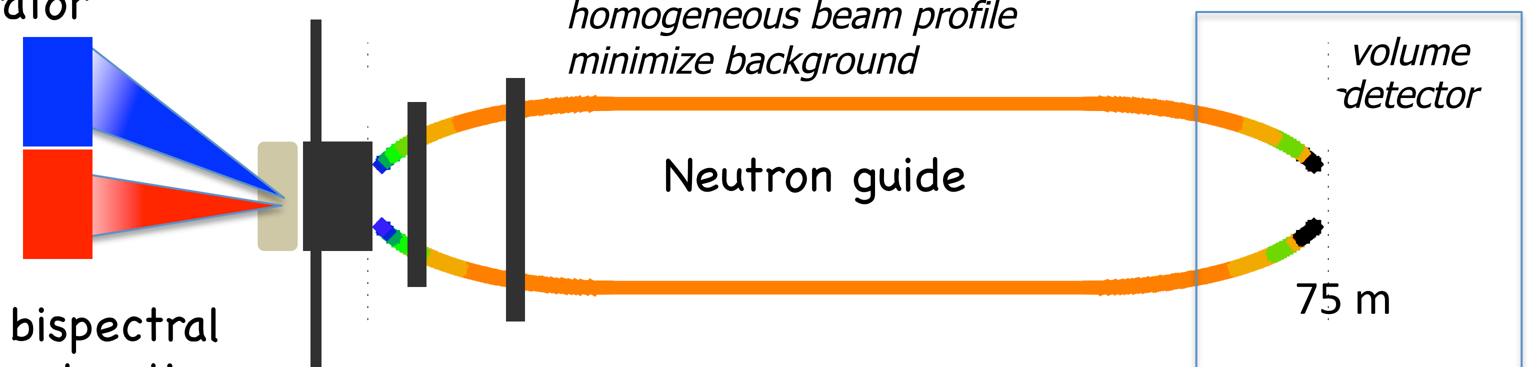
Primary instrument

Needle's eye

Moderator

cold

thermal



high brilliance transfer
homogeneous beam profile
minimize background

Secondary instrument

bispectral extraction

thermal beam in transmission
cold beam in reflection

Chopper system

flexible time resolution
WFM
ideal pulse shape
Prompt pulse suppression

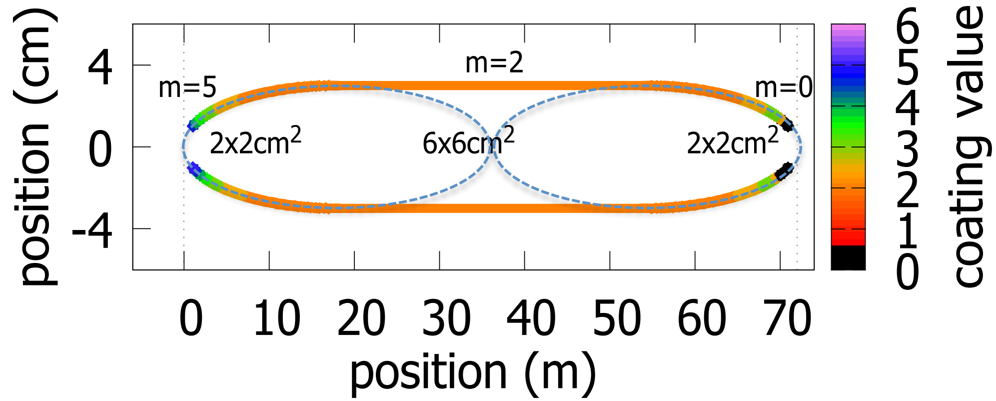
Ternary instrument

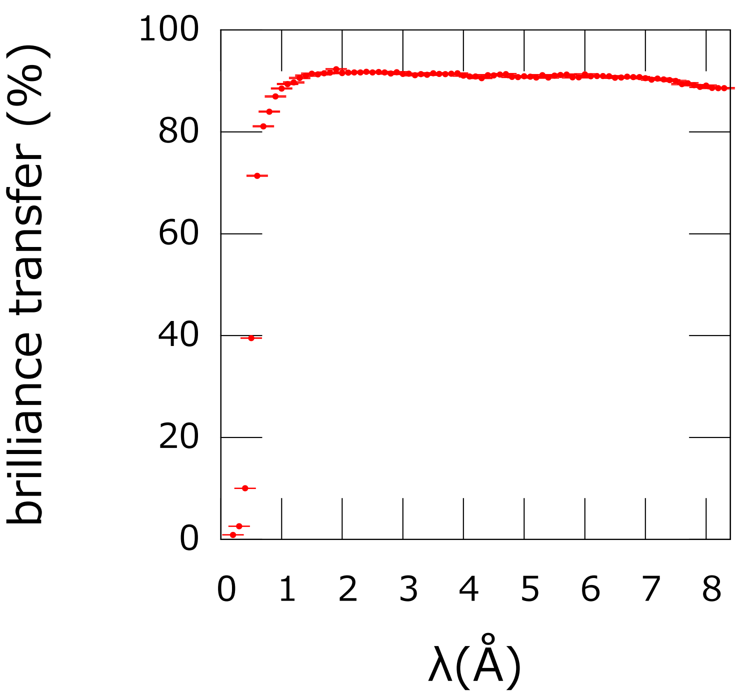
new routes for data analysis

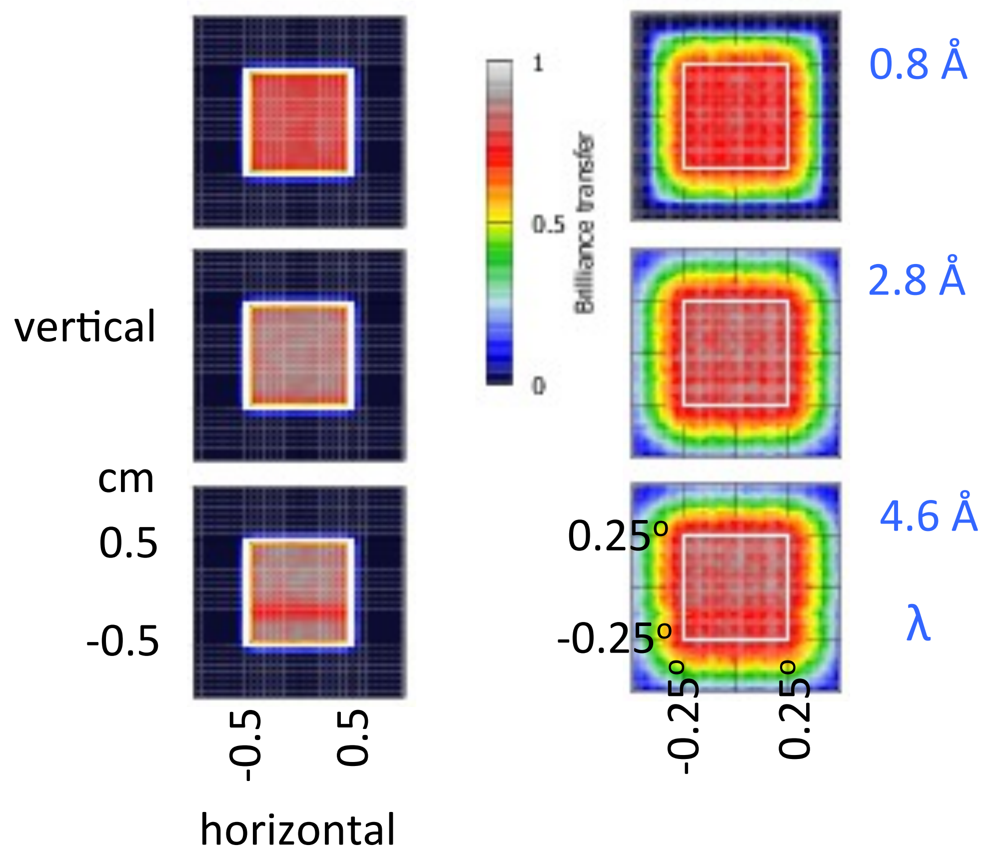
NIMA, **2012**, 680, 124.

Neutron guide simulations

Nicolo Violini

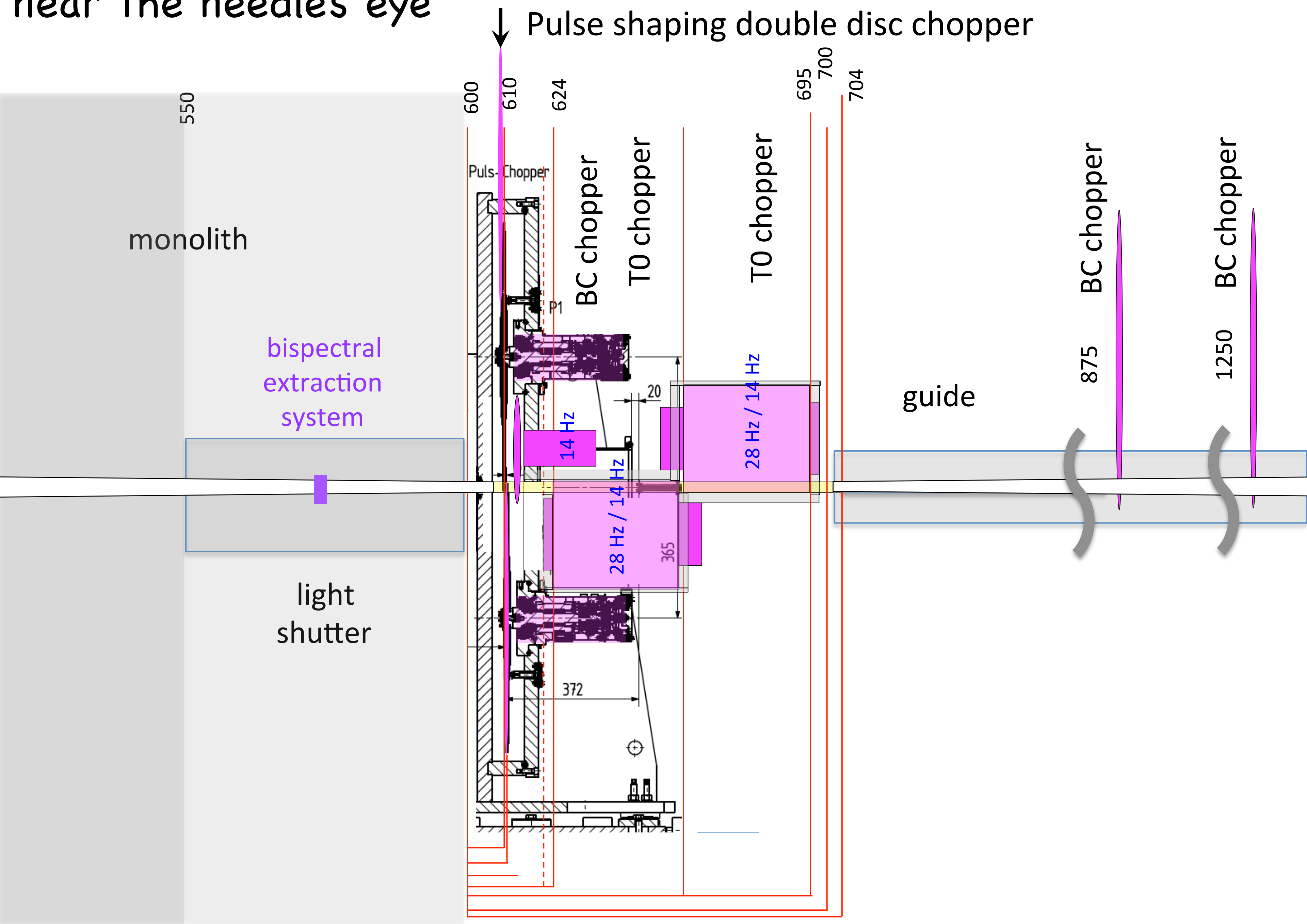






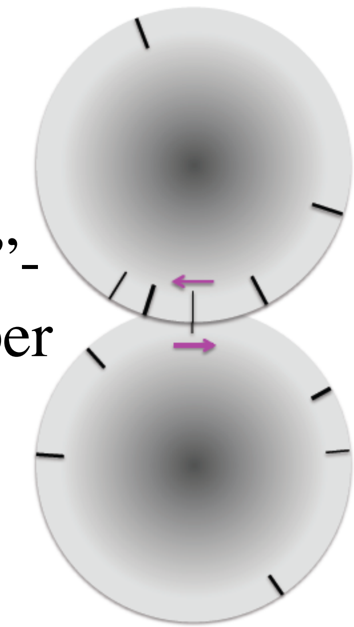
near the needle's eye

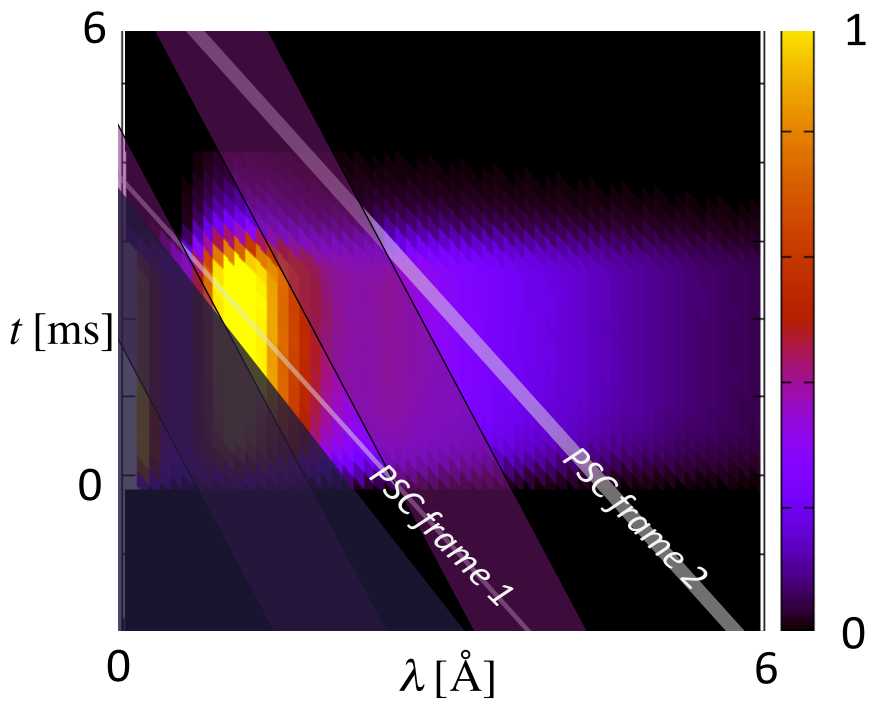
Choppers

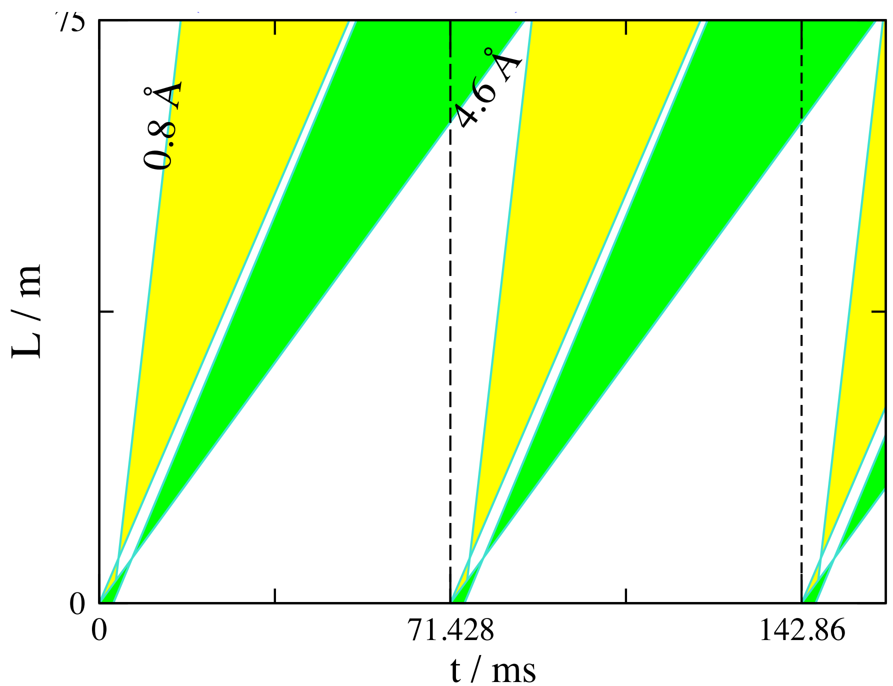


frame 1 $\delta t(\lambda_1)$	frame 2 $\delta t(\lambda_1 + 1.9\text{Å})$	$\omega_1 : \omega_2$ (Hz) : (Hz)
10.5 μs	31.3 μs	210 : 196
27.5 μs	82.5 μs	84 : 70
60.6 μs	181.8 μs	42 : 28
151.5 μs	353.5 μs	28 : 14
227.4 μs	606.3 μs	14 : 14
378.8 μs	681.8 μs	0 : 14

“Beat”-Chopper







Time resolution options

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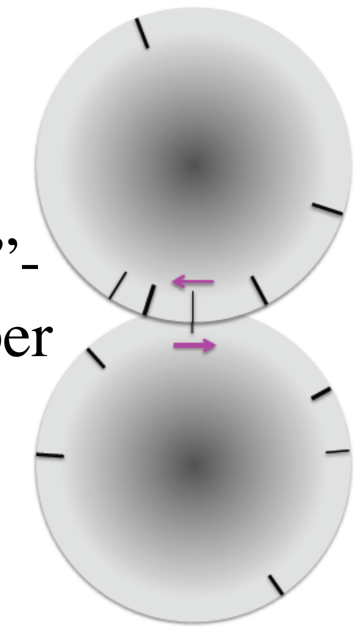
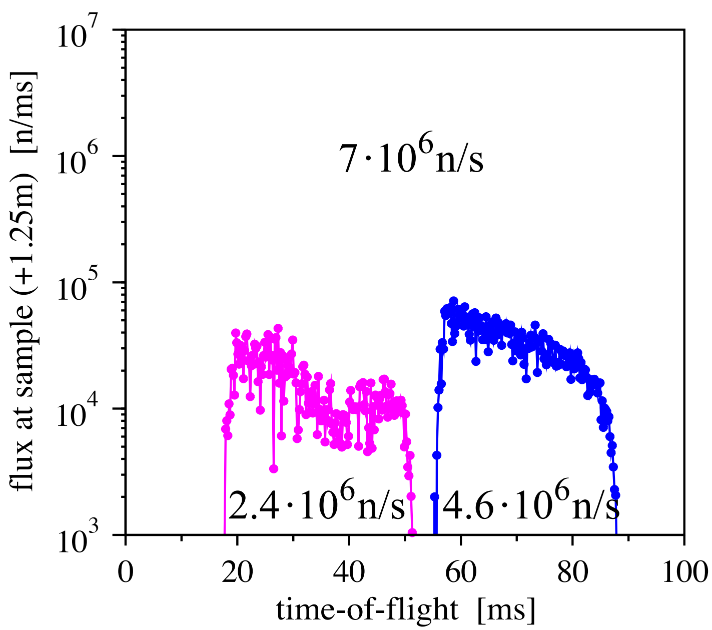
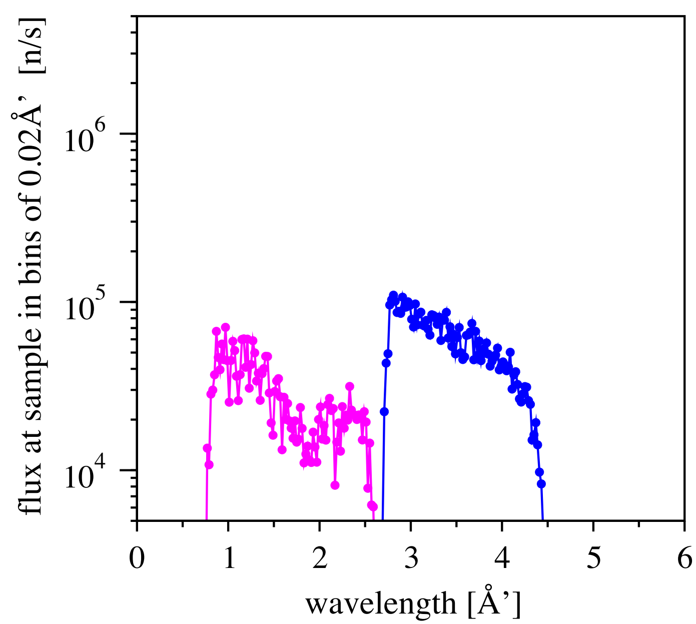


VITESS simulations *flux at sample*

K. Lieutenant





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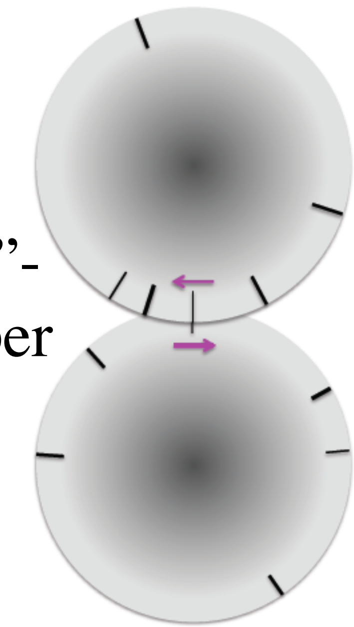

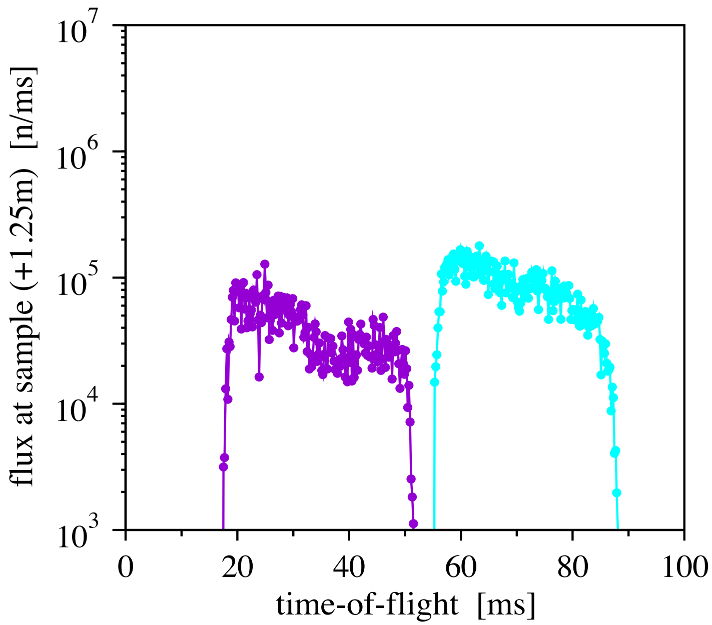

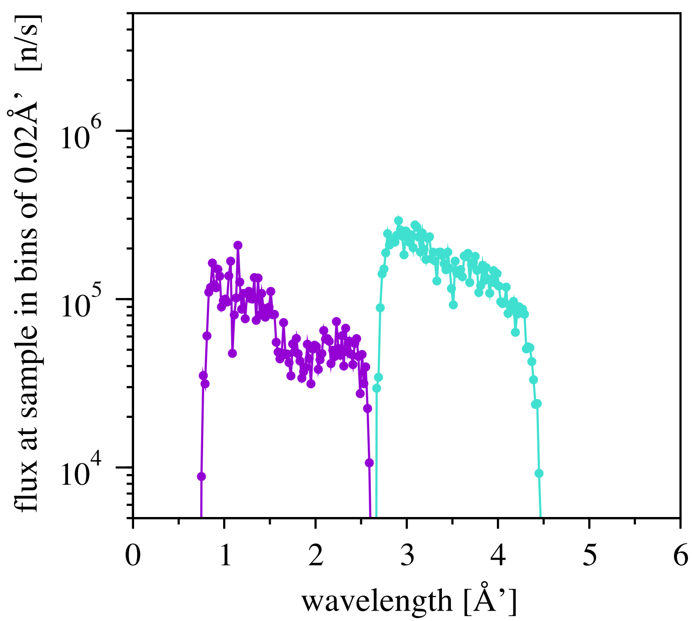

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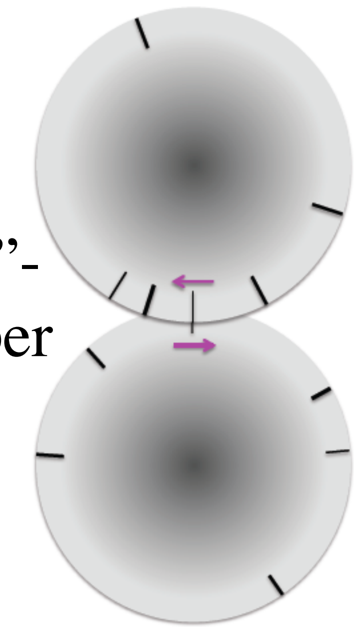

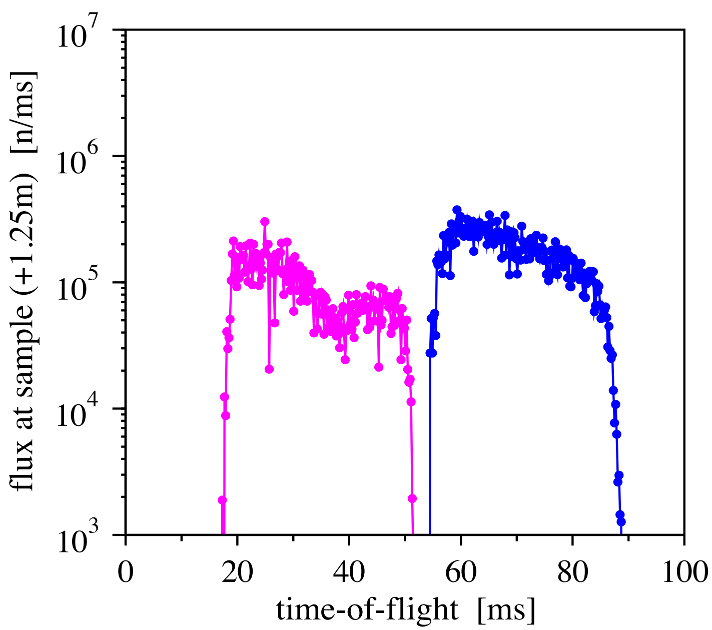

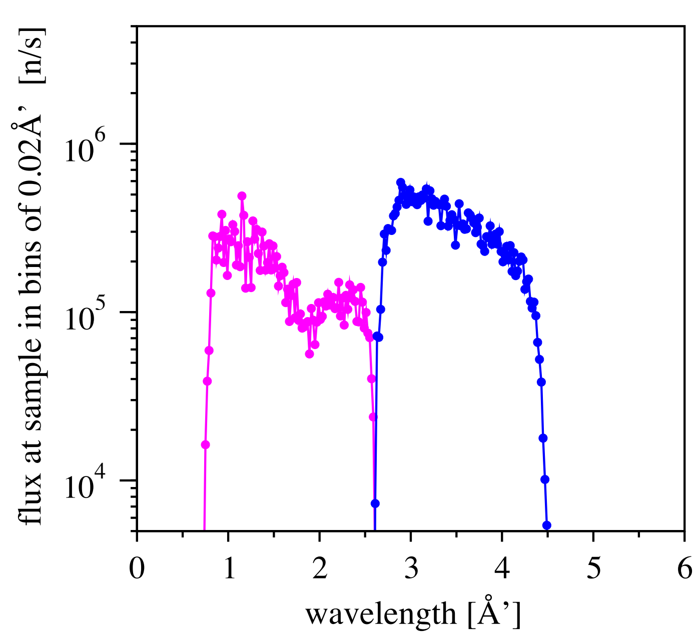

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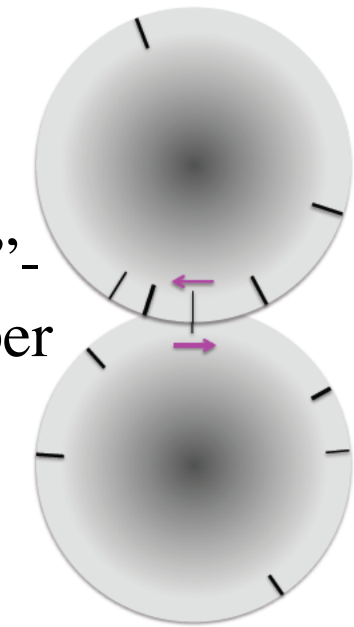

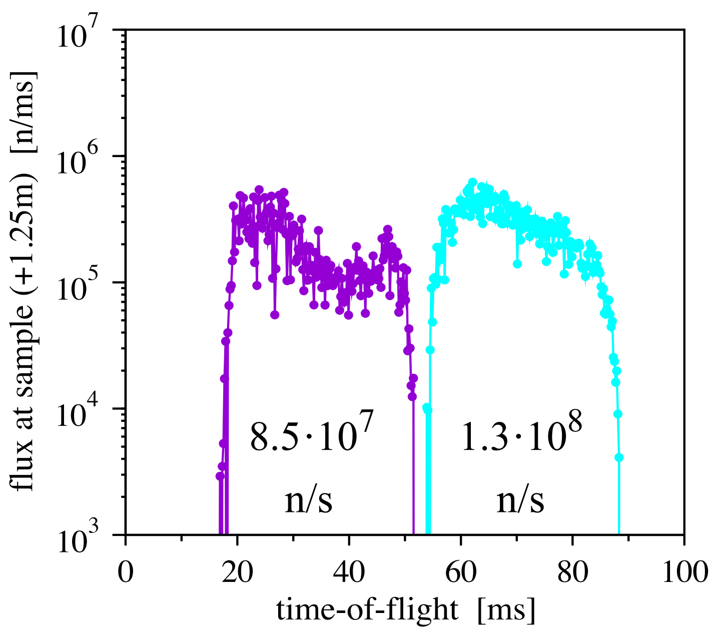

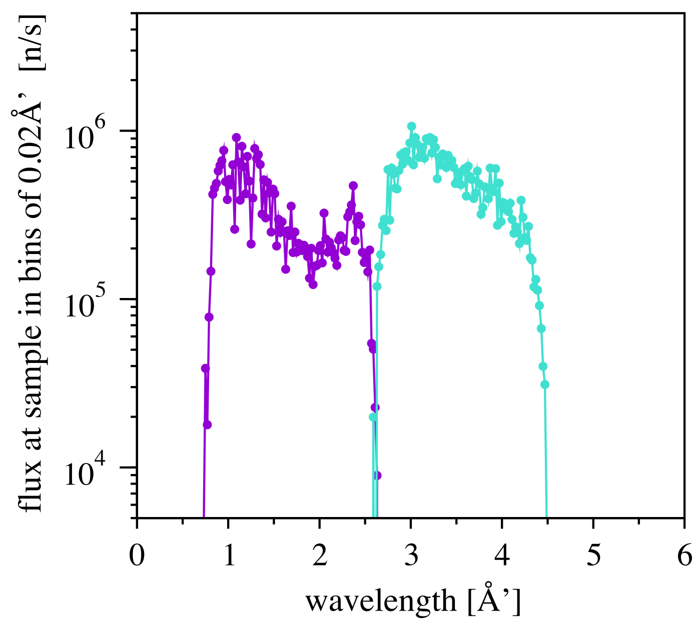

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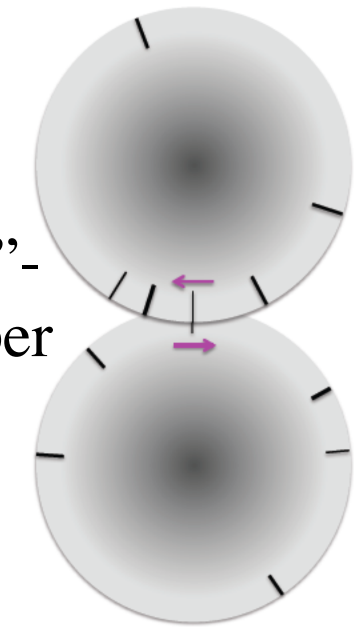

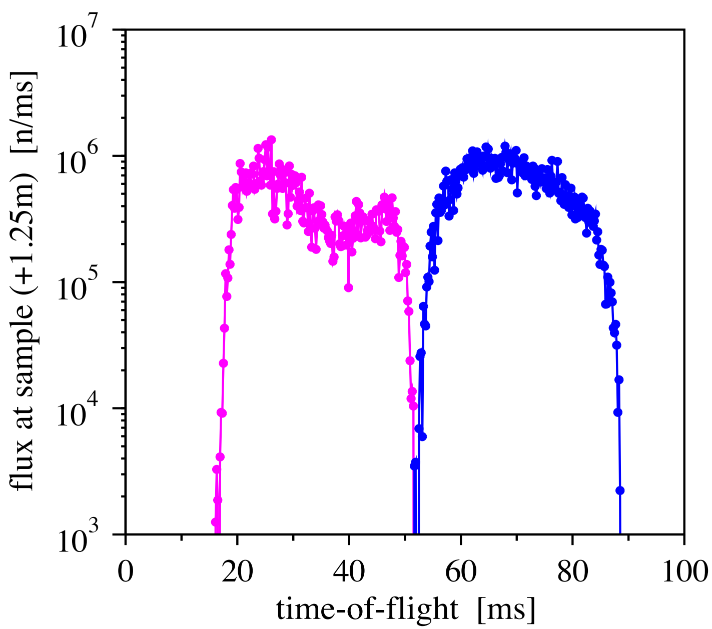

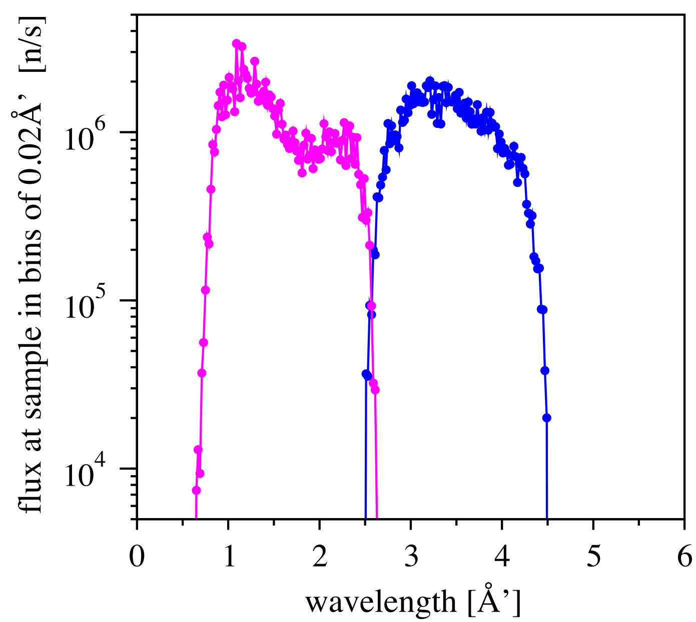

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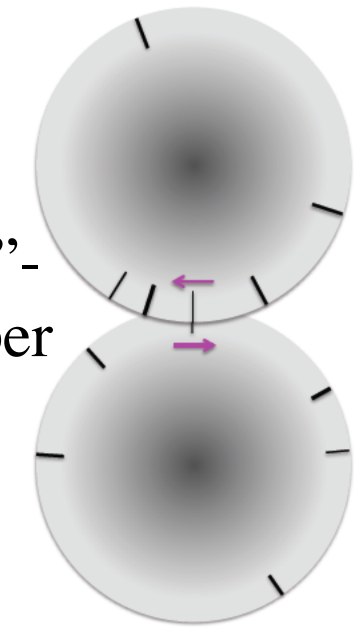
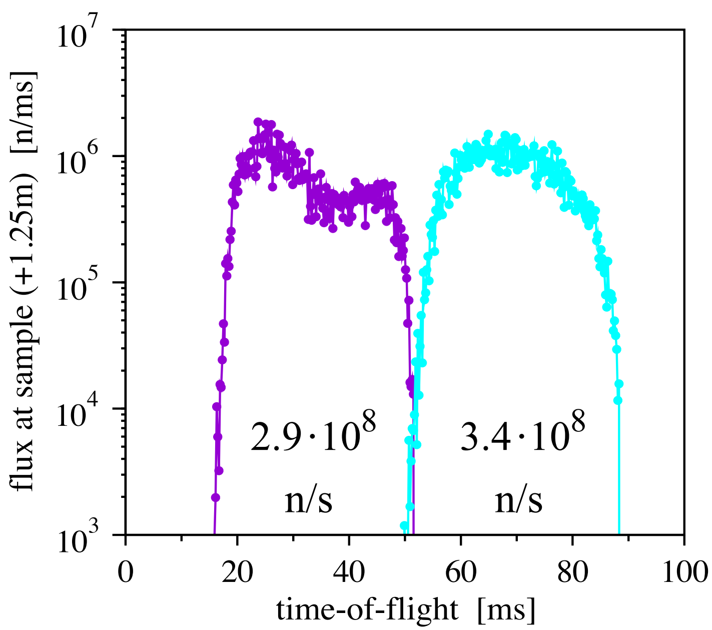
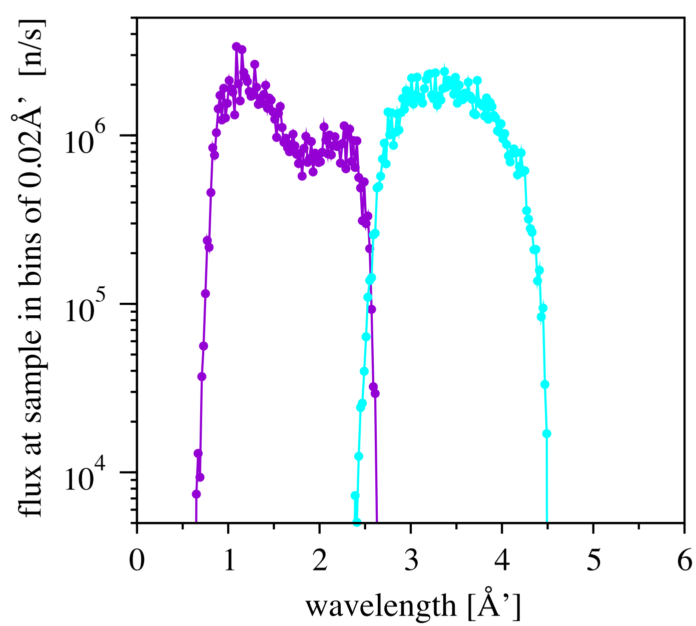
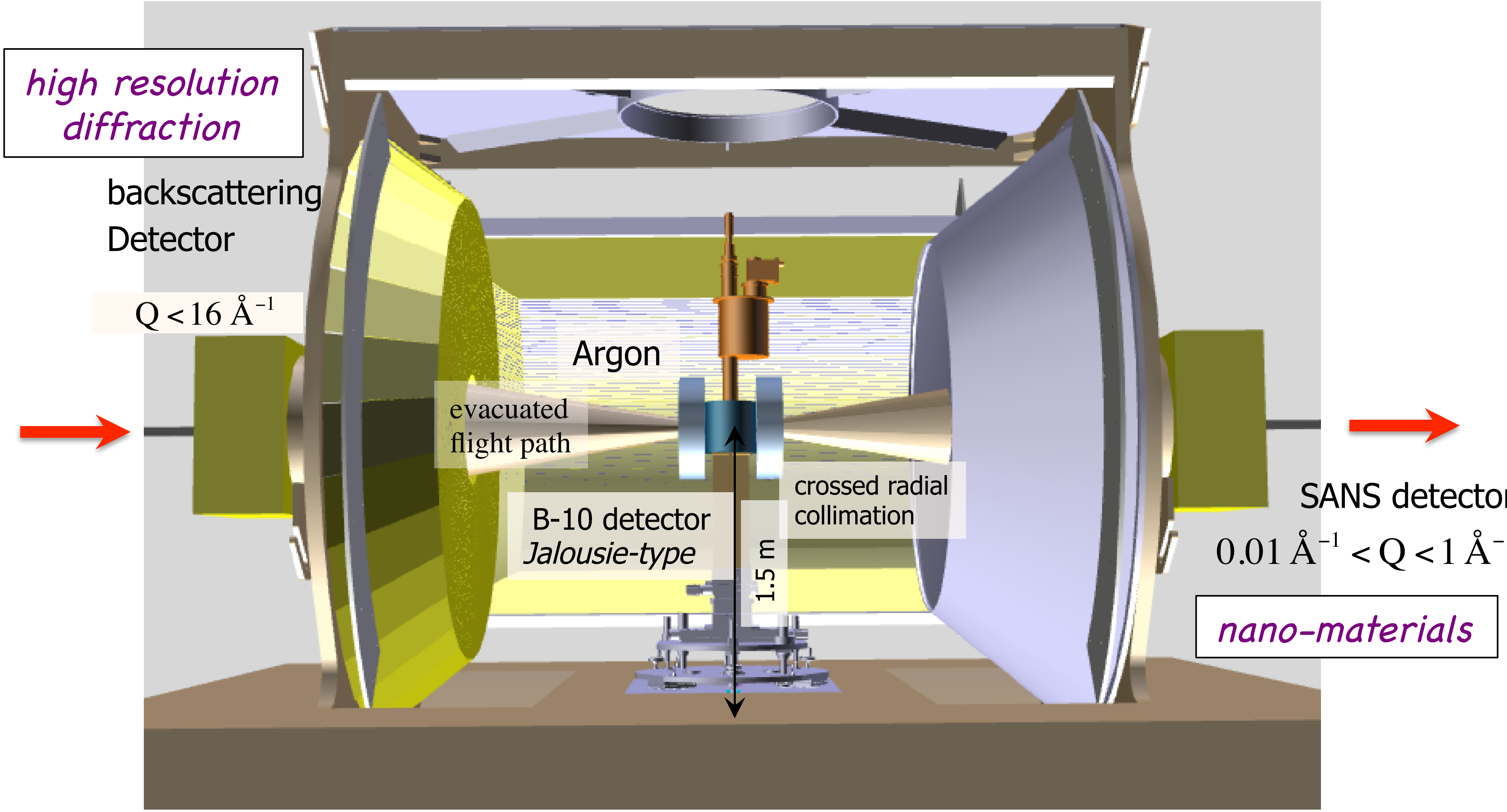








Volume detector 6 steradian 3D multi-wire chamber 3 mm spatial resolution

(i) solid B-10 film converter "Jalousie" efficiency >50% @ 1 Å *POWTEX-tested*
(ii) Gas converter $^{10}BF_3$ at 1 bar (!) efficiency >90% @ 0.75 Å !

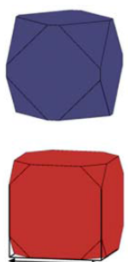
intrinsic collimation and energy sensitivity

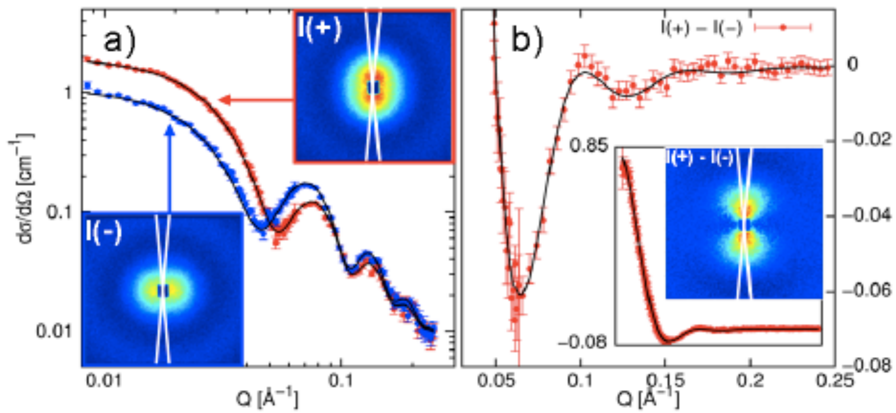
Magnetic nano-particles

powder diffraction atomic and magnetic structure

What can be done at small Q ? > 0.01 Å^{-1}

Polarized SANS Spatial magnetization distribution in iron oxide nanocubes and nanospheres



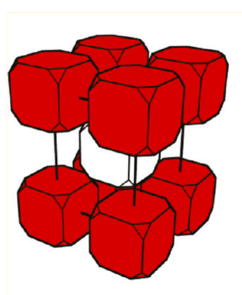


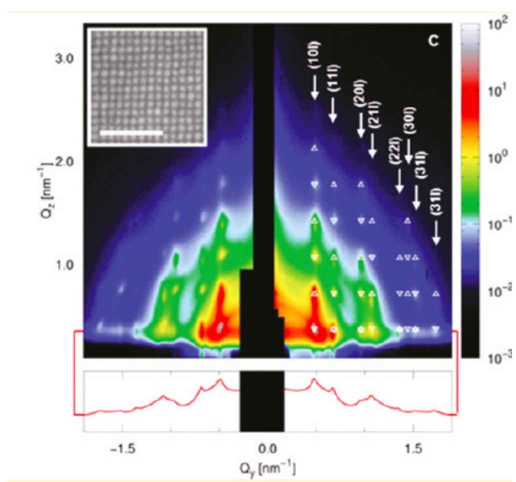
S. Disch *et al.*
New Journal of Physics **14**
013025 (2012)

GiSANS

2D resolution

Self-Assembly of Mesocrystals of iron oxide



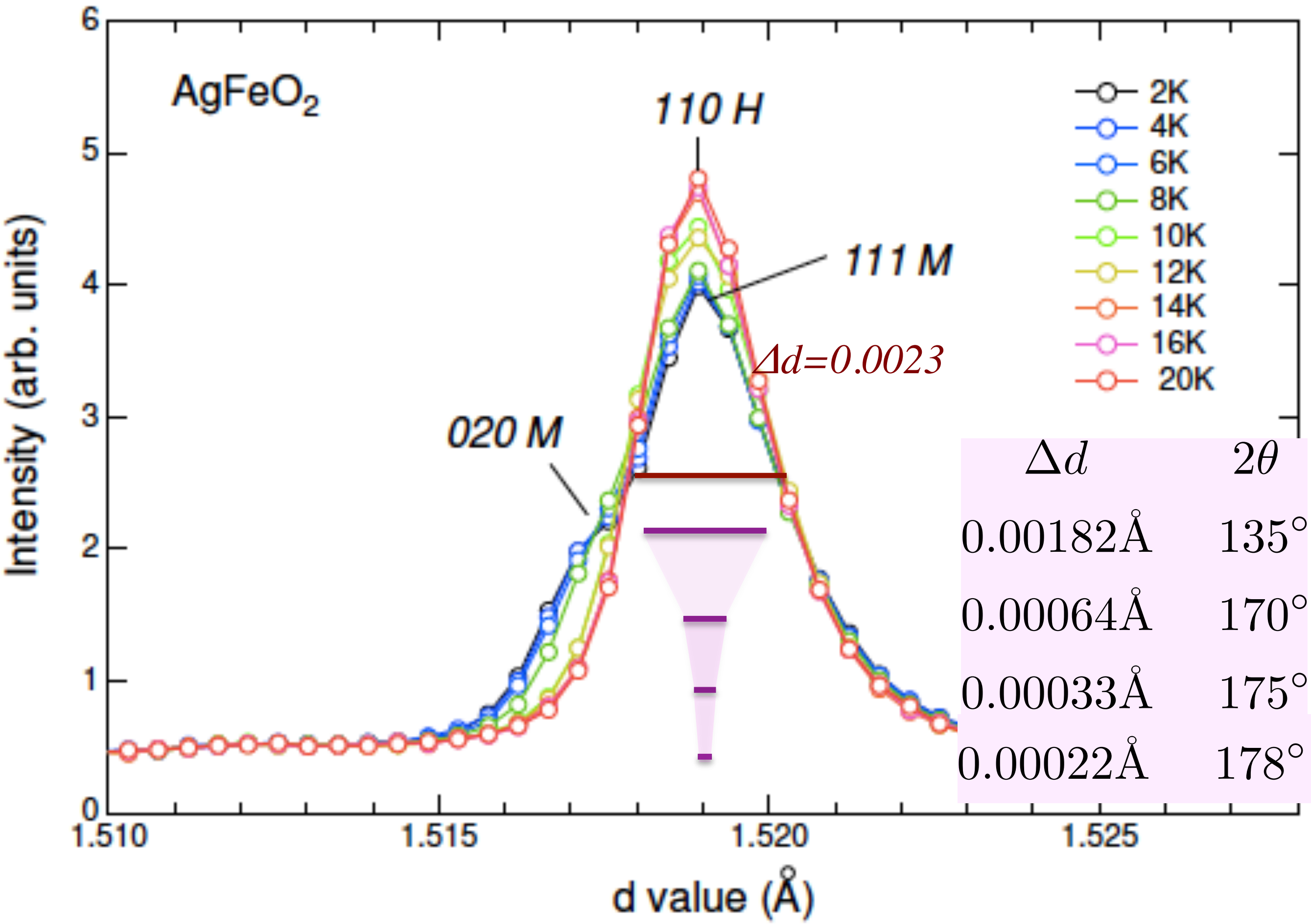


GiSAXS

S. Disch *et al.*
Nano Letters **11**
1651-1656 (2011)

high resolution – an example from HRPD

Delafossite, multiferroic $AgFeO_2$
hexagonal to monoclinic peak splitting



Δd	2θ
0.00182Å	135°
0.00064Å	170°
0.00033Å	175°
0.00022Å	178°

backscattering at POWHOW

Noriki Terada et al., EPJ Web of Conferences 40 , 15008 (2013)

New routes to data analysis

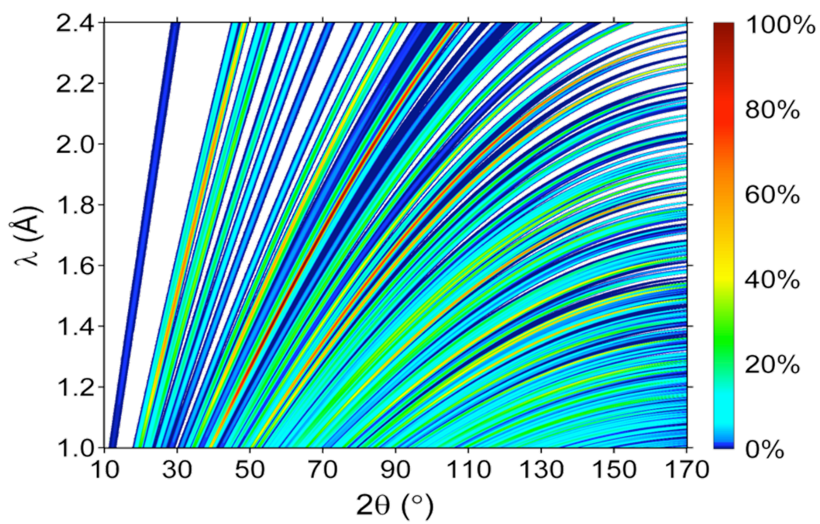
2D Rietveld refinement

***Philipp Jacobs**, Andreas Houben, WS*

POWTEX MLZ - Munich

"experimental" simulated data

CuNCN



- 416 peaks
- 1.12 million data points
- 23 minutes
- R-Profile = 3.49%
- $\chi 2$ = 6.1
- *Cmcm*

New routes to data analysis

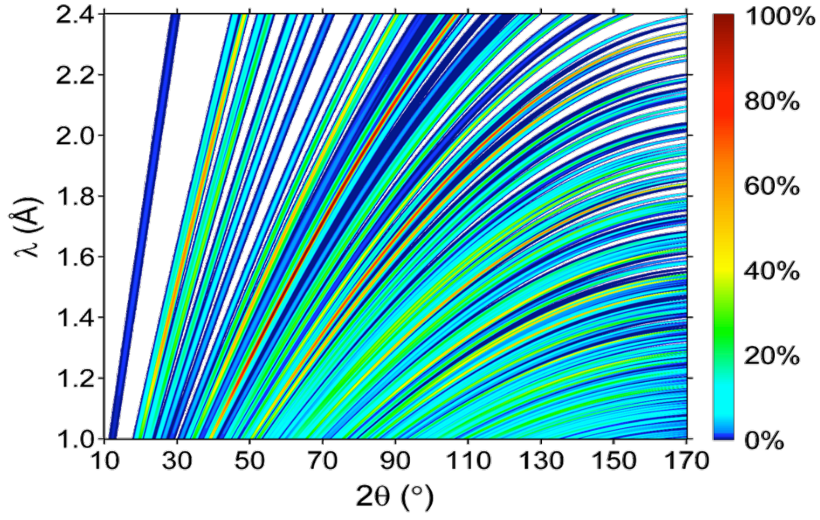
2D Rietveld refinement

***Philipp Jacobs**, Andreas Houben, WS*

POWTEX MLZ - Munich

Intensity modeled based on

the structure of CuNCN



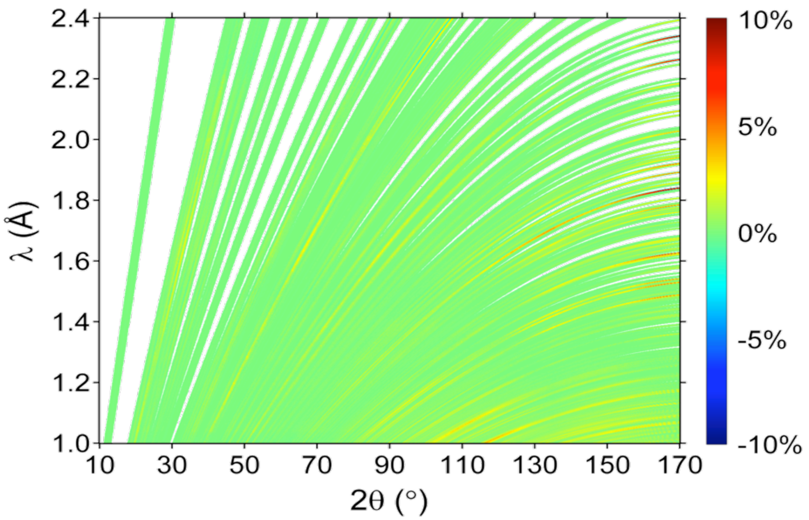
New routes to data analysis

2D Rietveld refinement

Philipp Jacobs*, Andreas Houben, WS*

POWTEX MLZ - Munich

difference CuNCN



New routes to data analysis

2D Rietveld refinement

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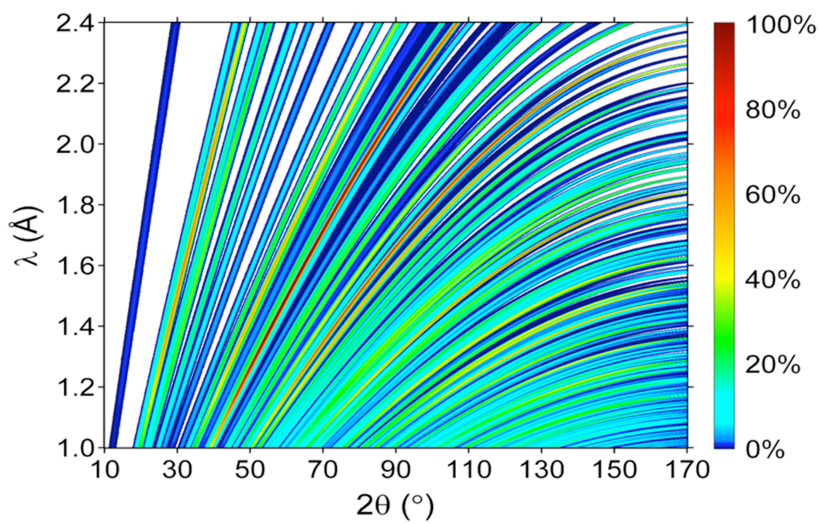
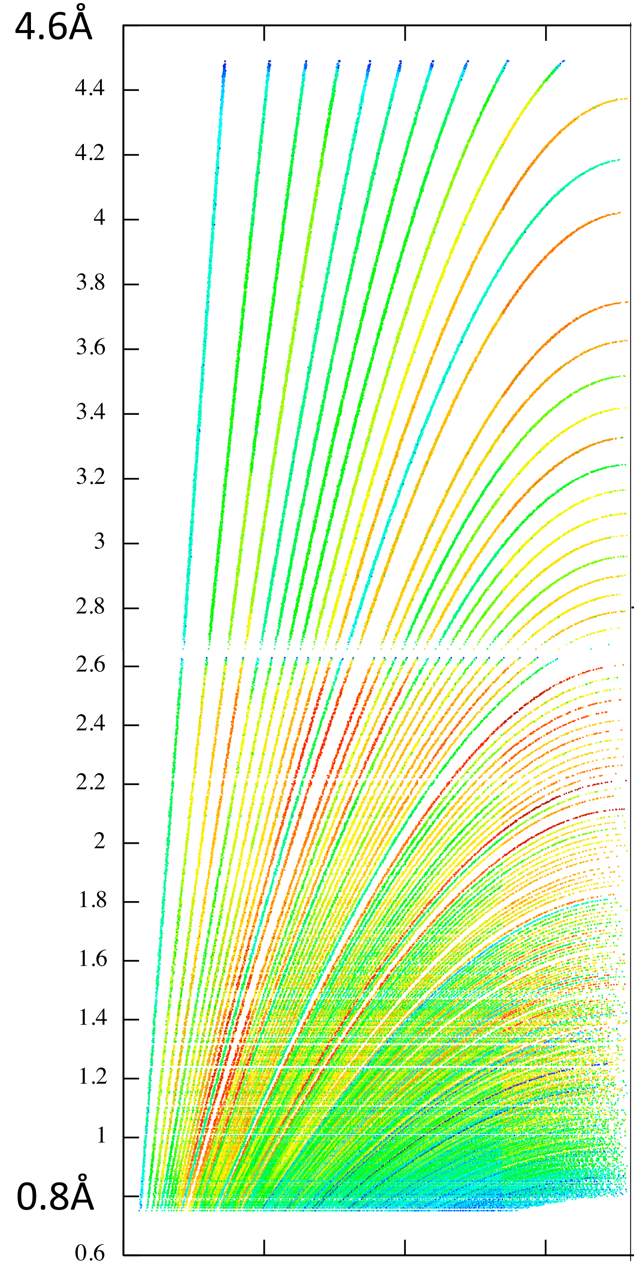
POWTEX MLZ - Munich

CuNCN



POWHOW



$Na_2Ca_3Al_2F_{14}$

VITESS *Klaus Lieutenant*

POWHOW

Sample 0.4 cm³
Efficency 0.53 (1Å)
Solid angle 6.2 sr

VITESS *Caroline Zendler*

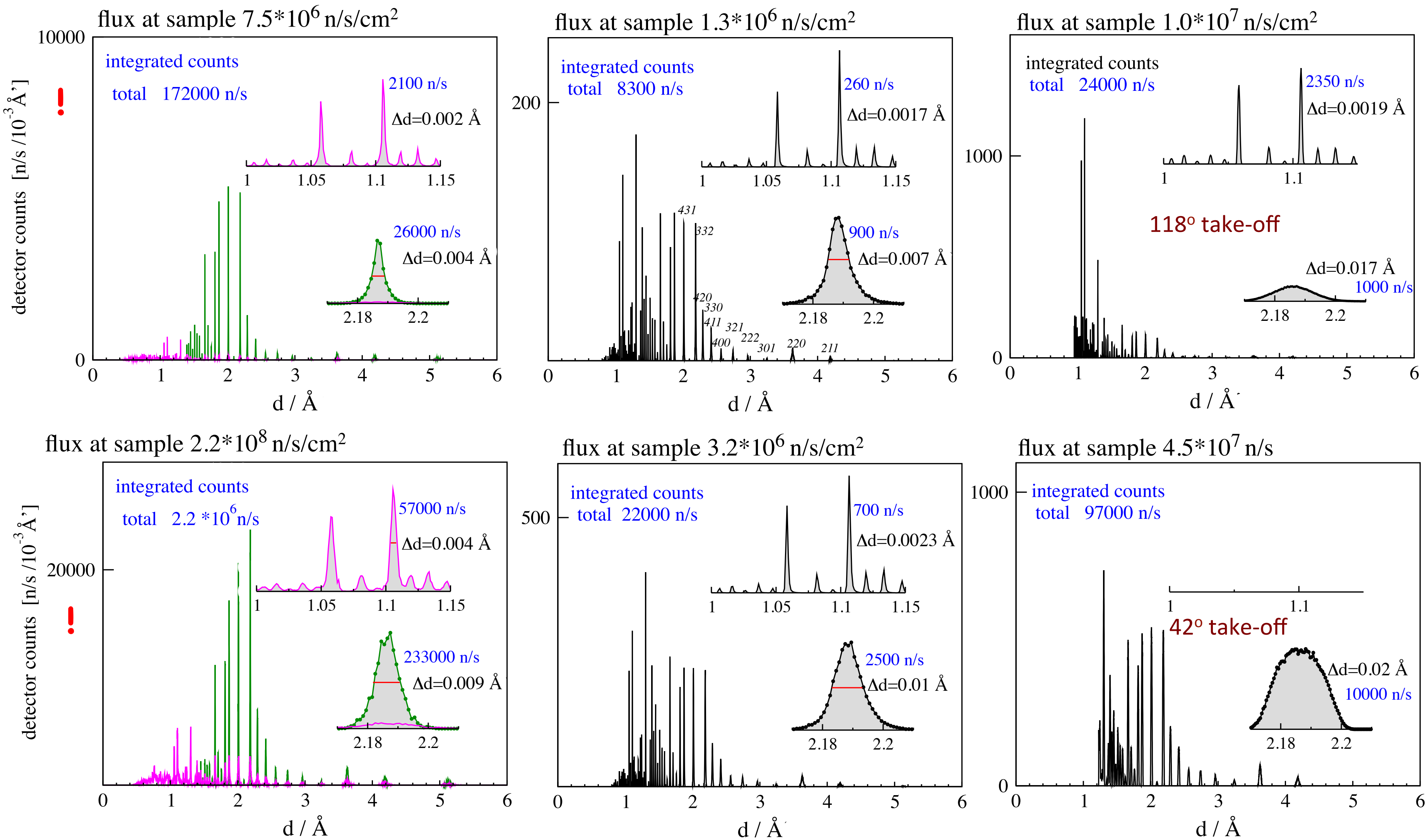
PowGen
SNS 1MW

Sample 0.8 cm³
Efficency 0.36 (1Å)
Solid angle 1.41 sr

VITESS *Daniil Nekrassov*

D20
ILL

Sample 0.8 cm³
Efficency 0.64 (1Å)
Solid angle 0.28

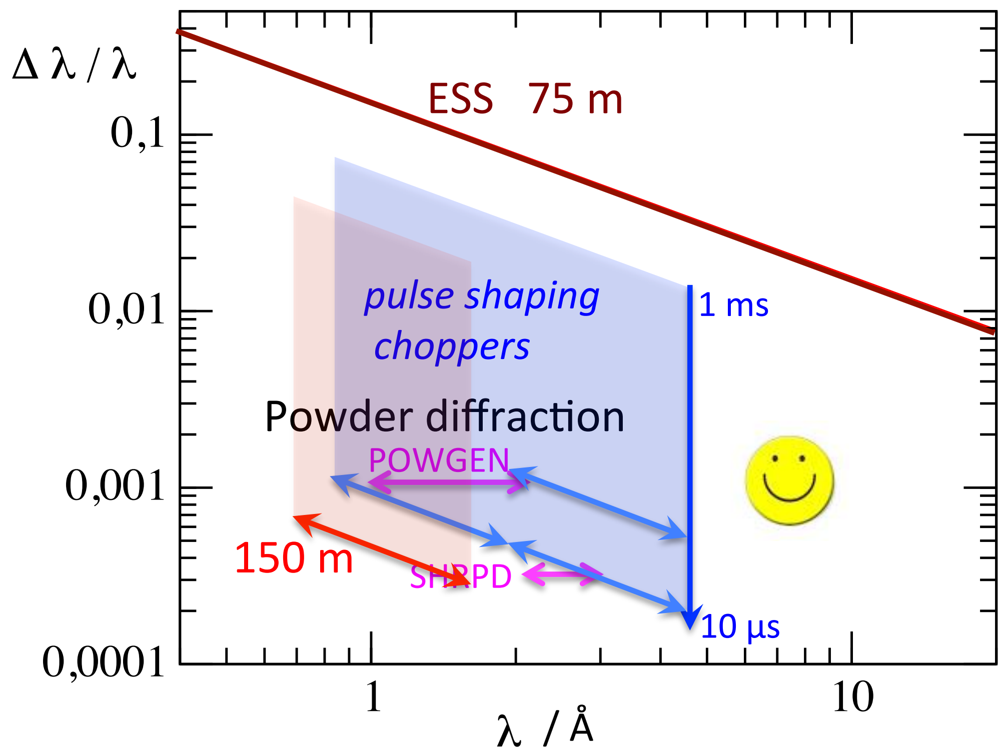


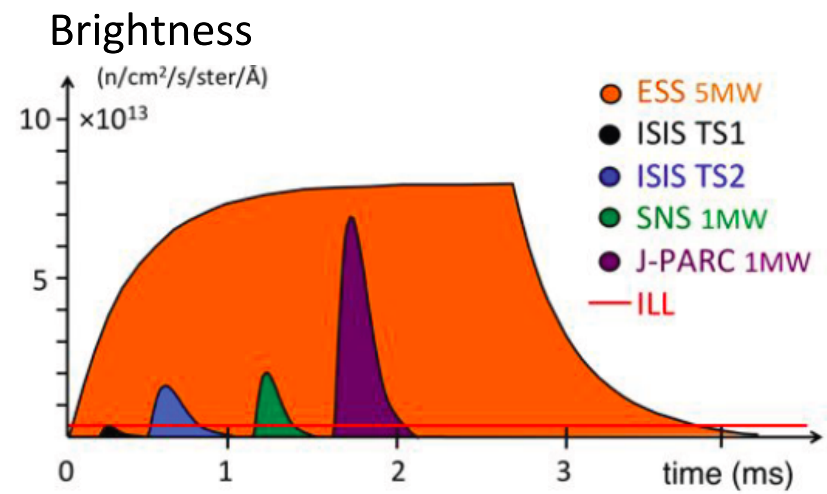
$Na_2Ca_3Al_2F_{14}$ cubic ($I2_13$) a = 10.257(1) Å J. Solid State Chem. 76 (1988) 426.

Thank you

Powder Diffraction at the ESS

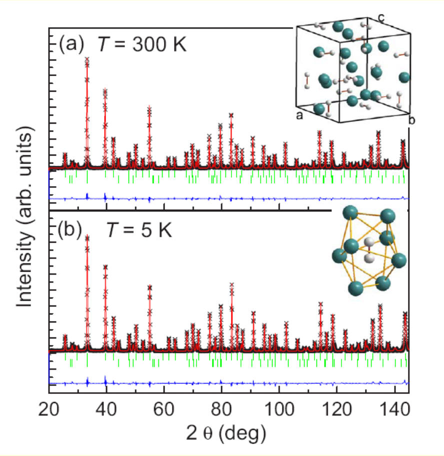
time – wavelength resolution





high flexibility in trading resolution *versus* intensity
ideal peak shape

What have we been using NPD for?
Structural and magnetic phase transitions below room-temperature!



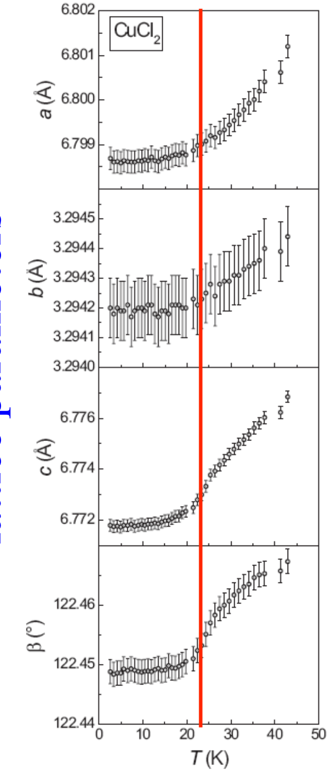
La_2C_3

non-centrosymmetric superconductor T_c=13.4 K

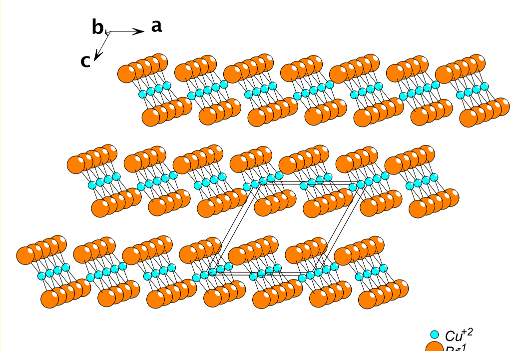
C-C dumbbell, C-C distance

impurity phase LaC_2

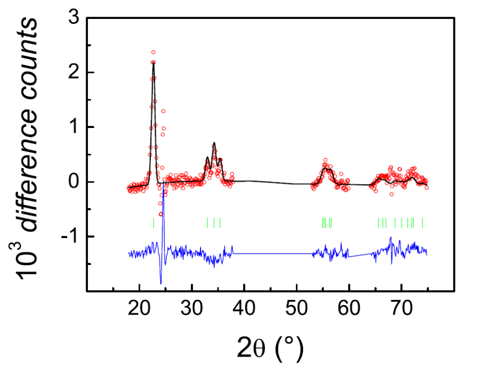
lattice parameters



The Helicoidal Chain Multiferroic Quantum AFM $CuCl_2$



CuBr2 after Oeckler and Simon, Z. Krist. 215 (2000) 13

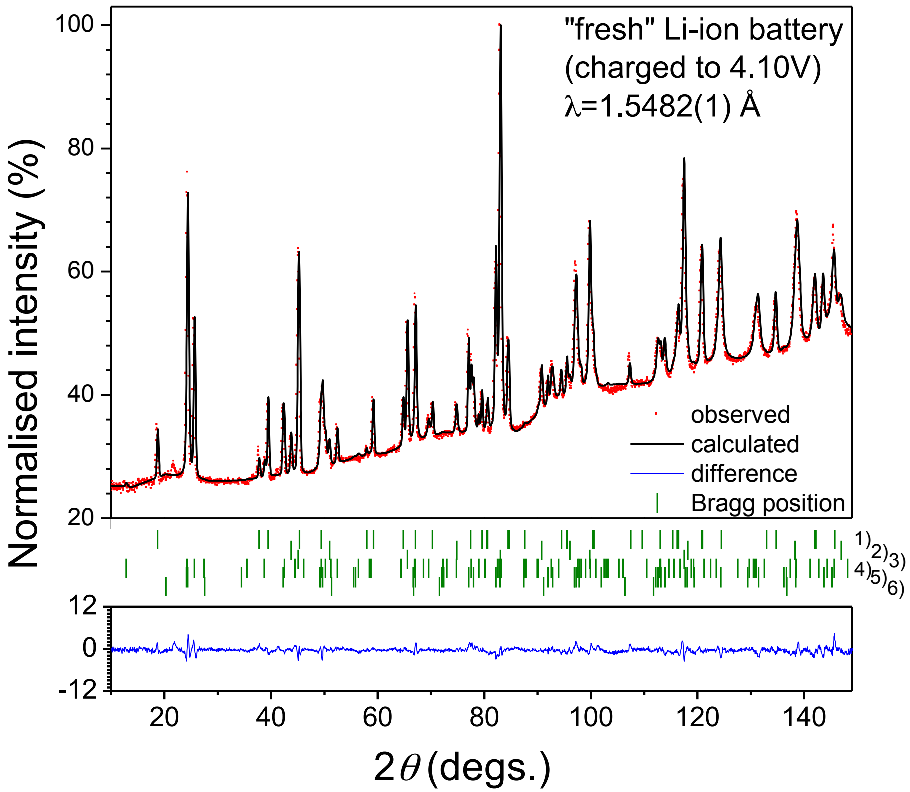


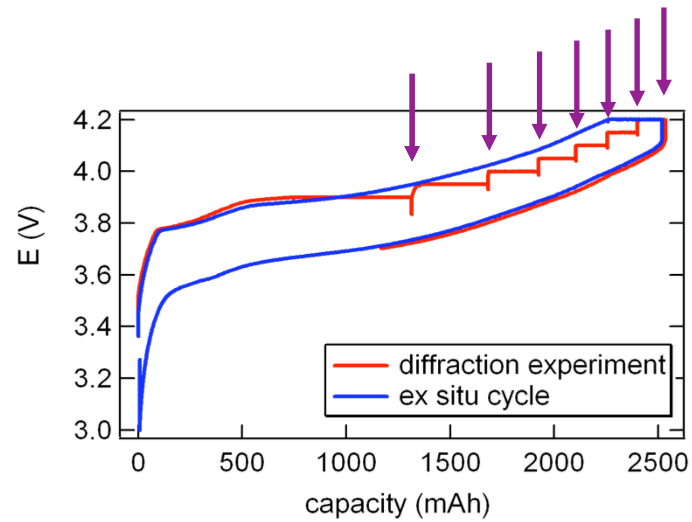
$$\vec{\tau}(2\ \ \mathrm{K}) = (1, 0.2257(6), 0.5)$$

$$\mu(2\ \ \mathrm{K}) = 0.50(1)\mu_B.$$

Neutron powder diffraction on commercial Li-ion battery

($LiCoO_2$ based, 18650 type)





multi-phase

A. Senyshyn, W. Schmahl LMU Munich