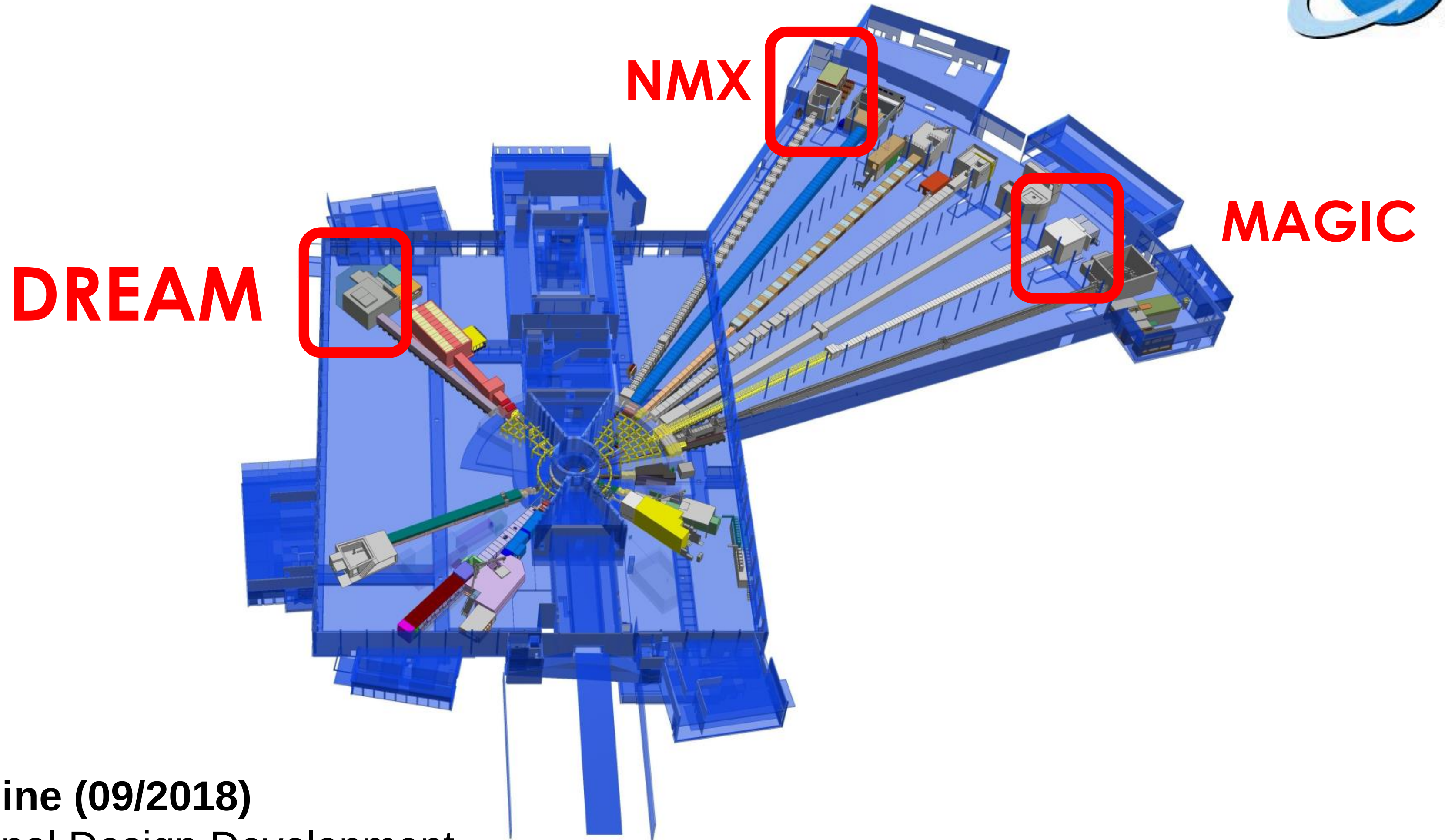
workshop on single crystal diffraction IKON15

DATA FOR DREAM

Wednesday, September 12th, Lund

F. Porcher

DREAM OVERVIEW

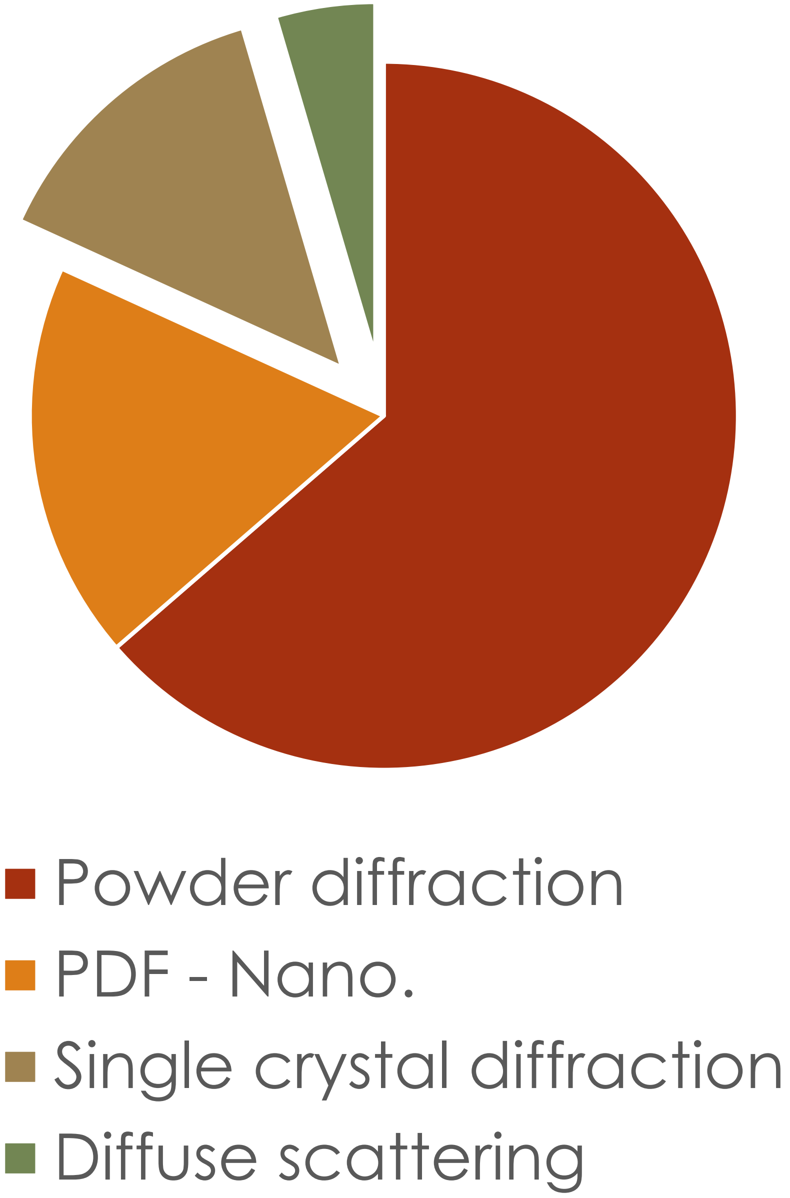


DREAM Timeline (09/2018)

- 2017-2018: Final Design Development
- 2018-2023: Construction & Installation & Cold Commissioning
- 2023-2023: Hot Commissioning (Friendly Users)
- 2023 (end): General User Operations

1 year of Hot commisionning before SOUP
Be ready ! (CC ?)

DREAM's SCIENCE CASE



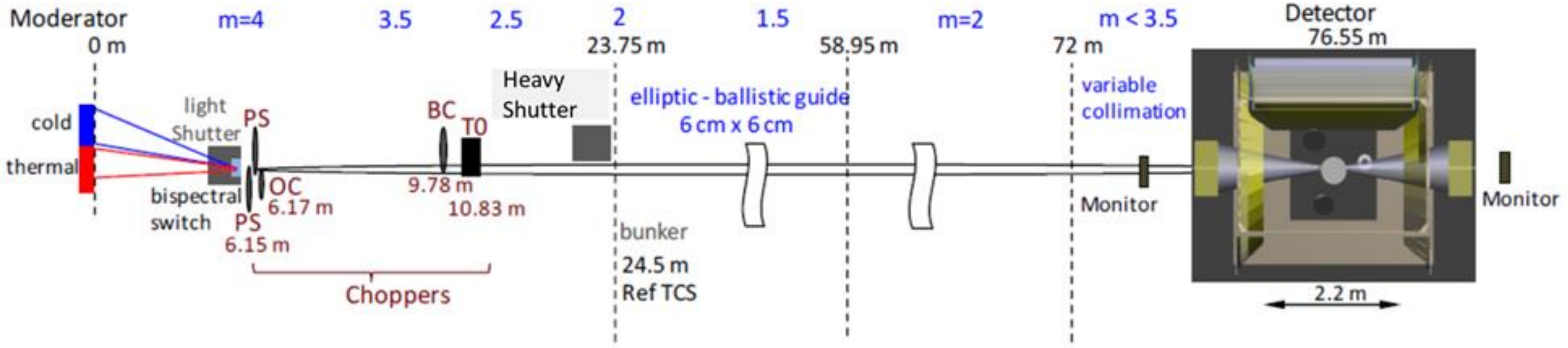
Polarized SXD ➔ Magic
Very large cells ➔ NMX

SXD on DREAM :

- **Weakest effects (magnetic/nuclear)**
  orbital ordering
  charge ordering
  distortion
  magnetic exchange
- **Anisotropic excitations ($\vec{P}$, $\vec{H}$, $\vec{E}$,…)**
- **Epitaxial systems ?**
- **???**

DREAM PHYSICAL SETUP IN 3 SLIDES

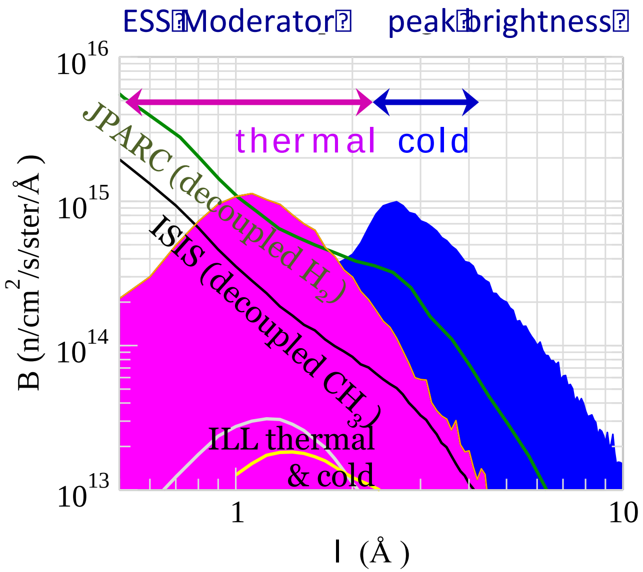
Diffraction Resolved by Energy and Angle Measurement

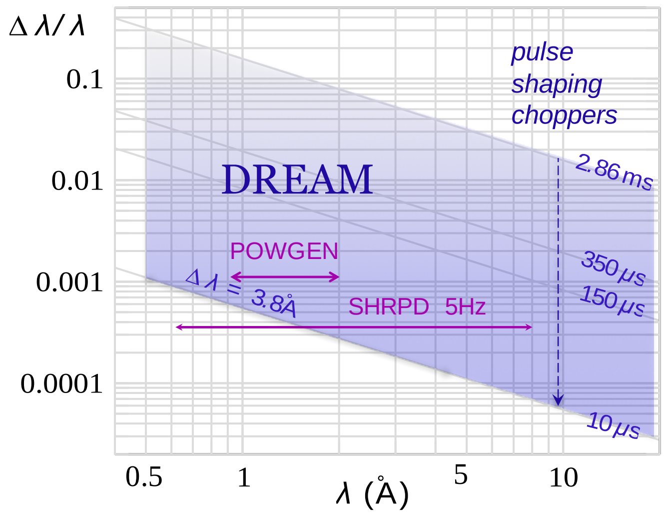


Bispectral instrument with

- **Large bandwidth (Δλ ~3.6 Å)**
- **High flux & High resolution flexibilty (PSC)**

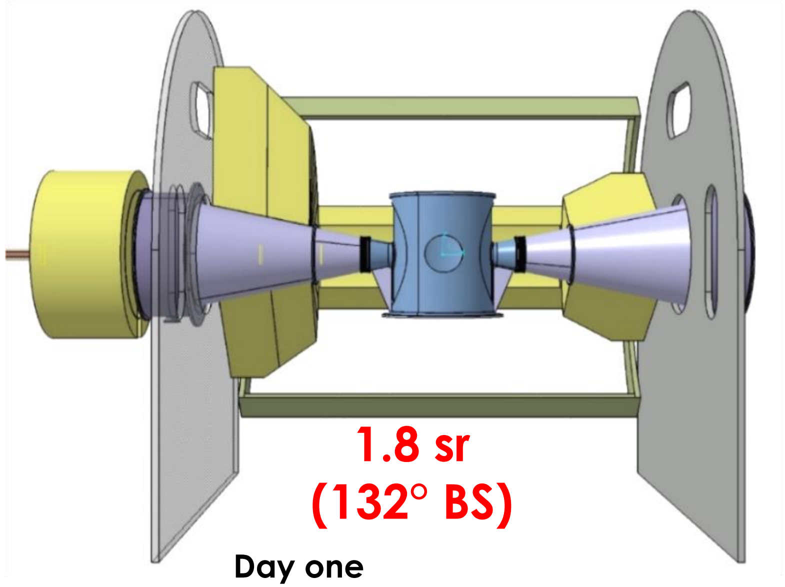
Choppers, Collimators
Monitors



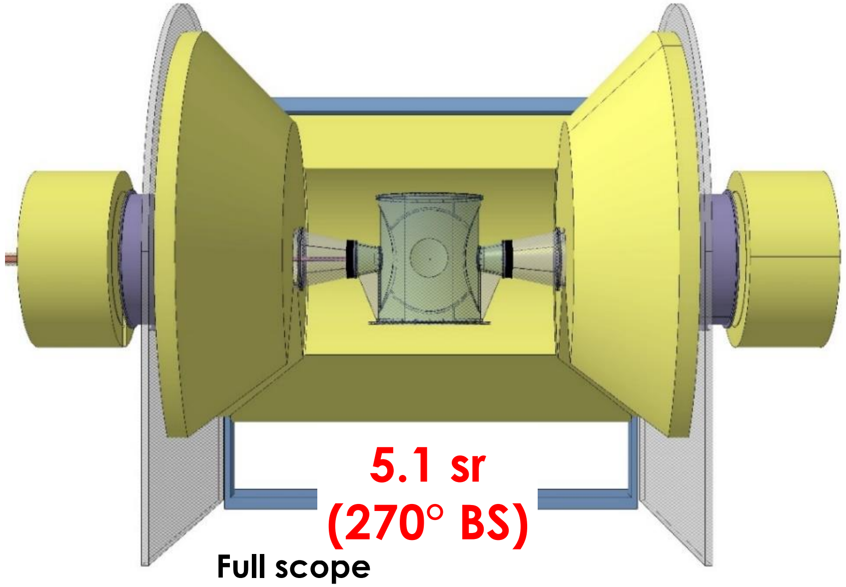


DREAM PHYSICAL SETUP IN 3 SLIDES

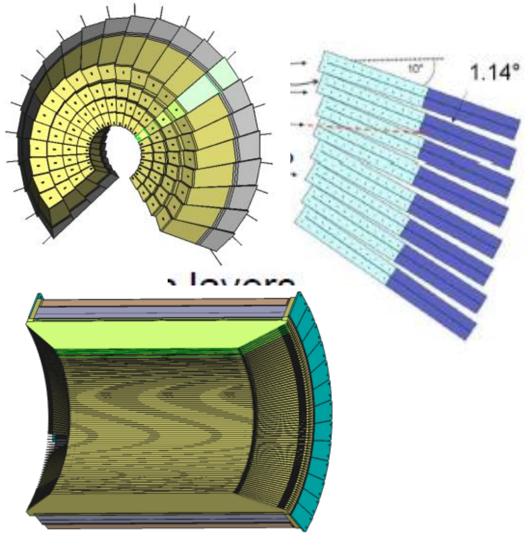
Diffraction Resolved by Energy and Angle Measurement



Day one



Full scope



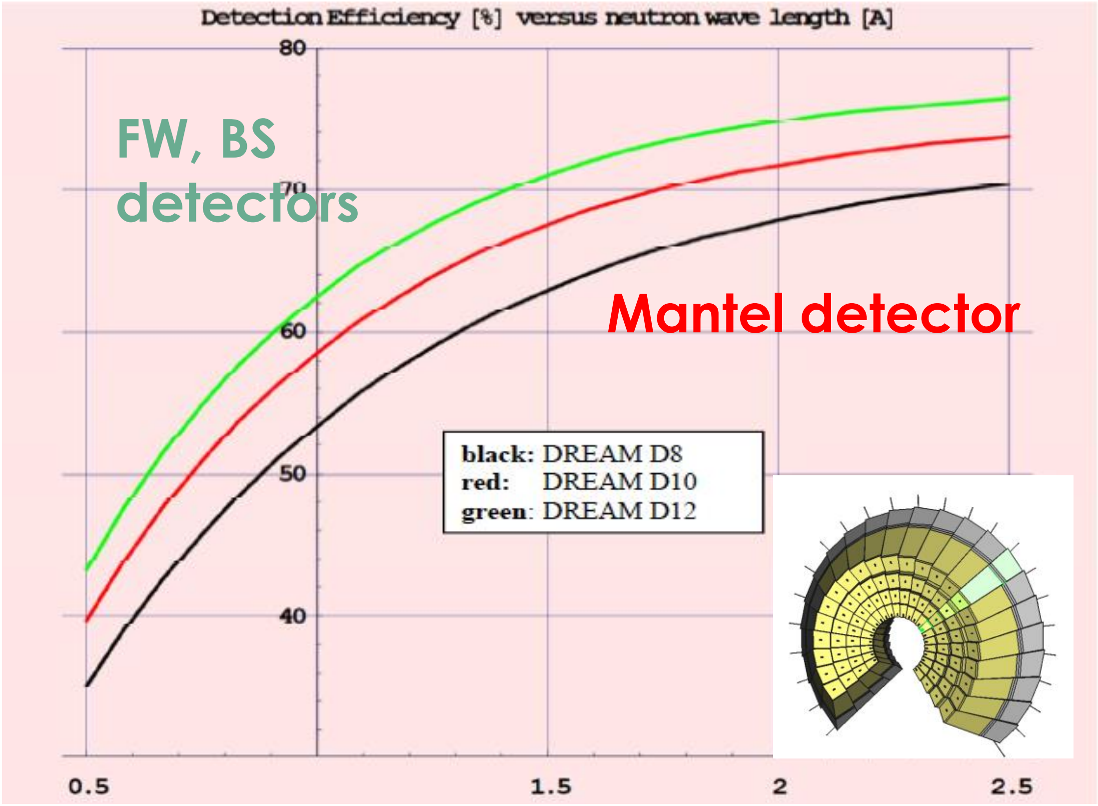
Detector config.				Day one	Full scope
Detector banks	Δ2Θ	Δϕ	ΔL	dΩ (sr)	dΩ (sr)
HR Forward 0.6°-12°	< 0.1°	~0.1°	20mm	0	0.14
Forward 12° - 45°	0.32°	~0.5°	16.4-19.4mm	0.29 (60°)	1.30 (270°)
Mantle 45° - 135°	0.29°	0.53°	5.4mm	0.77 (30°)	2.23 (91°)
Backward 135° -168°	**0.32°**	**~0.5°**	**16.4-19.4mm**	**0.61 (132°)**	**1.30 (270°)**
HR Backward > 168°	**< 0.1°**	**~0.1°**	**7-10mm**	**0.14**	**0.14**
Total coverage				1.81 (35%)	5.11 (100%)

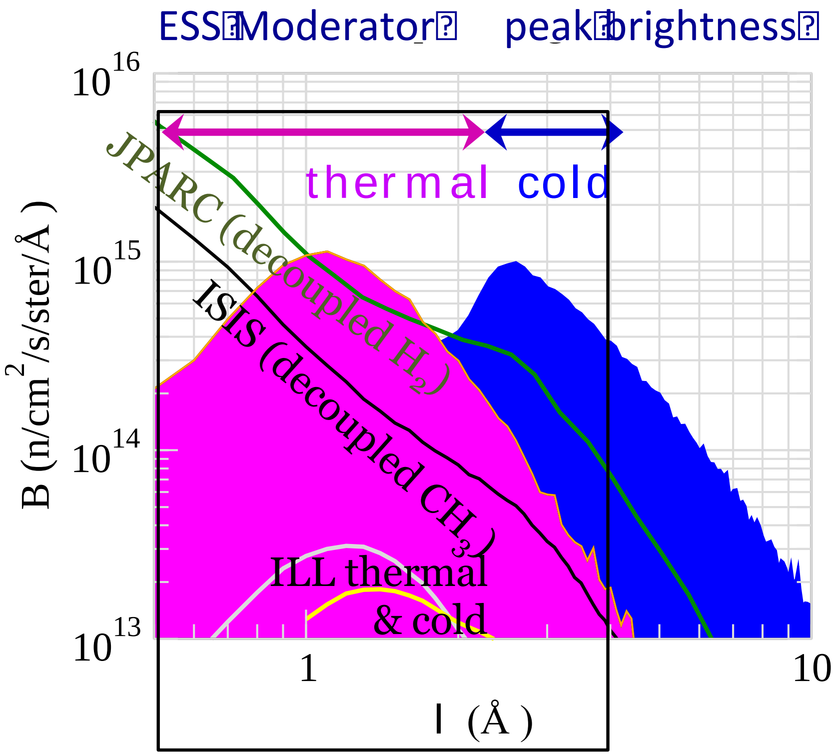
Geometry

Voxels with variable size
Distorsion
Binning

DREAM PHYSICAL SETUP IN 3 SLIDES

Diffraction Resolved by Energy and Angle Measurement





Detection

Efficiency (« sector »)
Efficiency (lambda)

Time stamping

Monitors

RAMP-UP OF DATA ON DREAM

HC - SOUP :	coverage 1.81 sr	-	accelerator power 0.5 MW
Full Detector :	coverage 5.11 sr	-	accelerator power 2 MW
Complete Scope:	*coverage 5.11 sr*	*-*	*accelerator power 5 MW*

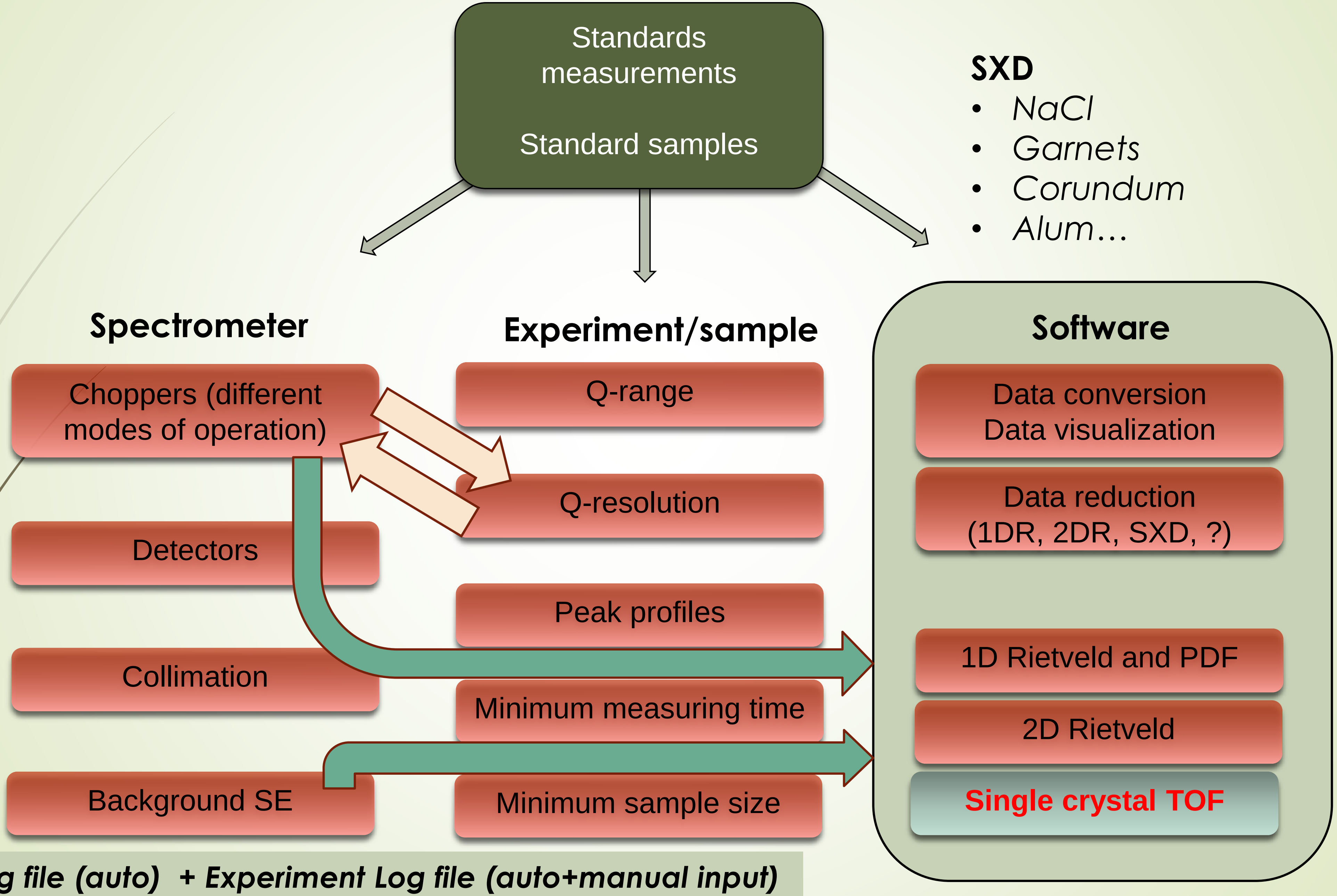
Voxels:
HC - SOUP (Reduced config.): ~ 762.000
Full detector - Complete Scope : ~ 2.167.000

Counting Rates:
HC - SOUP : ~ 2 660 000 n/s
Full Detector : ~ 30 000 000 n/s
Complete Scope :~ 75 000 000 n/s

Estimated typical event rates (for single-crystal peaks) :
HC - SOUP : $\lesssim$ 0,18 Gbit/s
Full Detector : $\lesssim$ 2 Gbit/s
Complete Scope : $\lesssim$ 5 Gbit/s

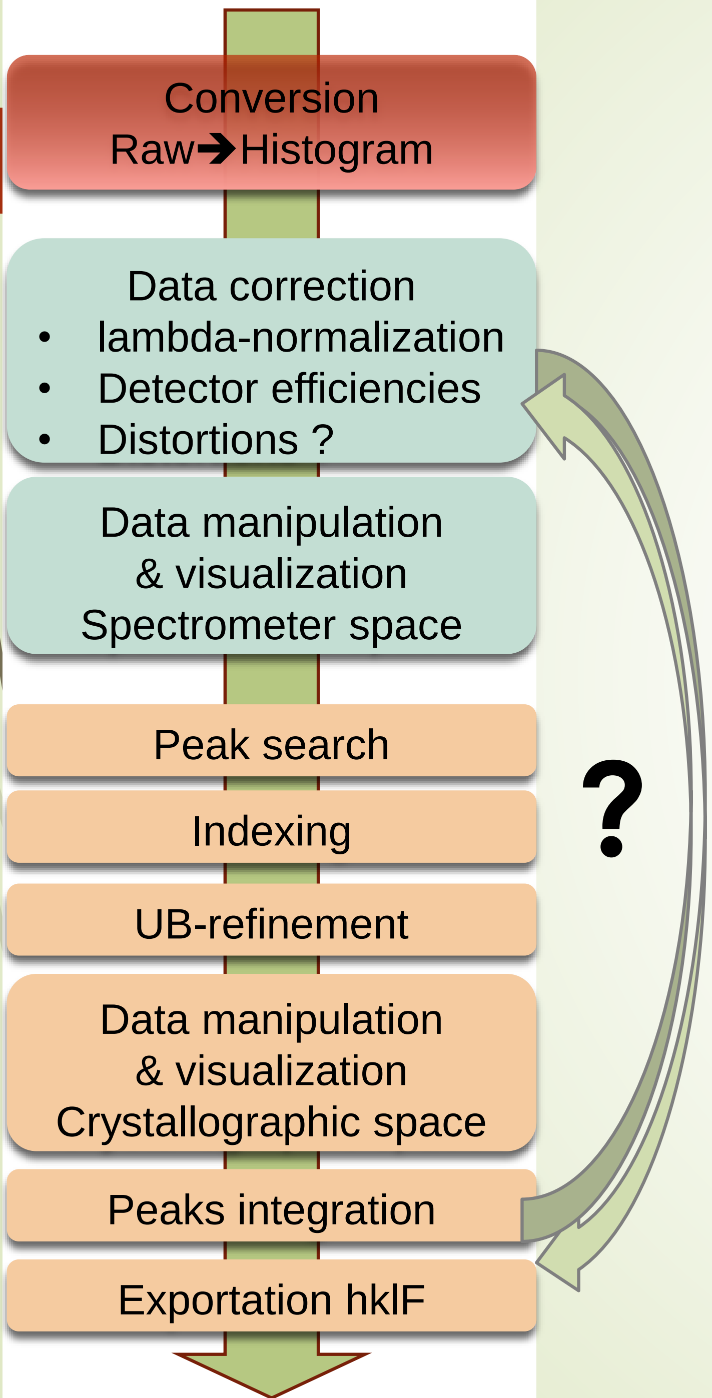
PSC settings ➔ Various configurations from high-intensity to high-resolution mode
Wavelength Frame Multiplication (WFM)

Hot Commissioning : data corrections, instrument functions, tools



Instrument Log file (auto) + Experiment Log file (auto+manual input)

Data processing flow



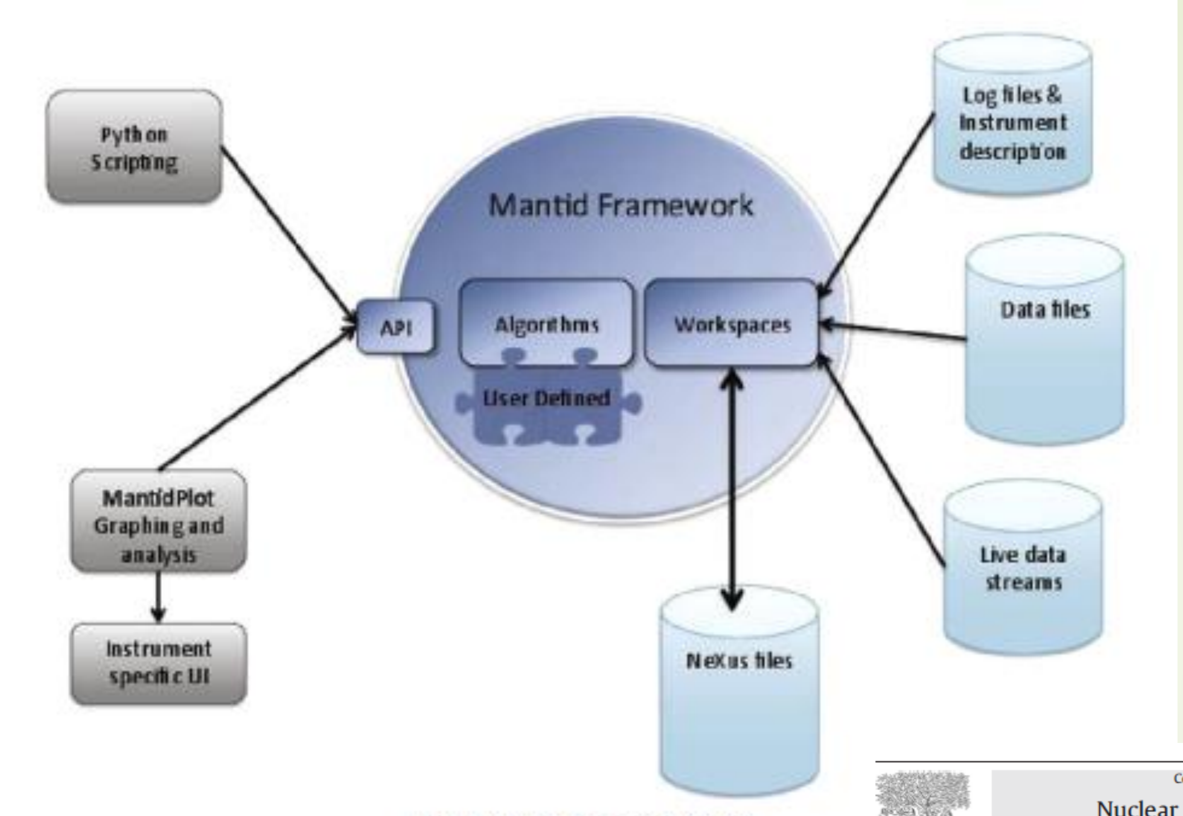


Fig. 1. Mantid framework design.

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

ELSEVIER

Mantid—Data analysis and visualization package for neutron scattering and μ SR experiments

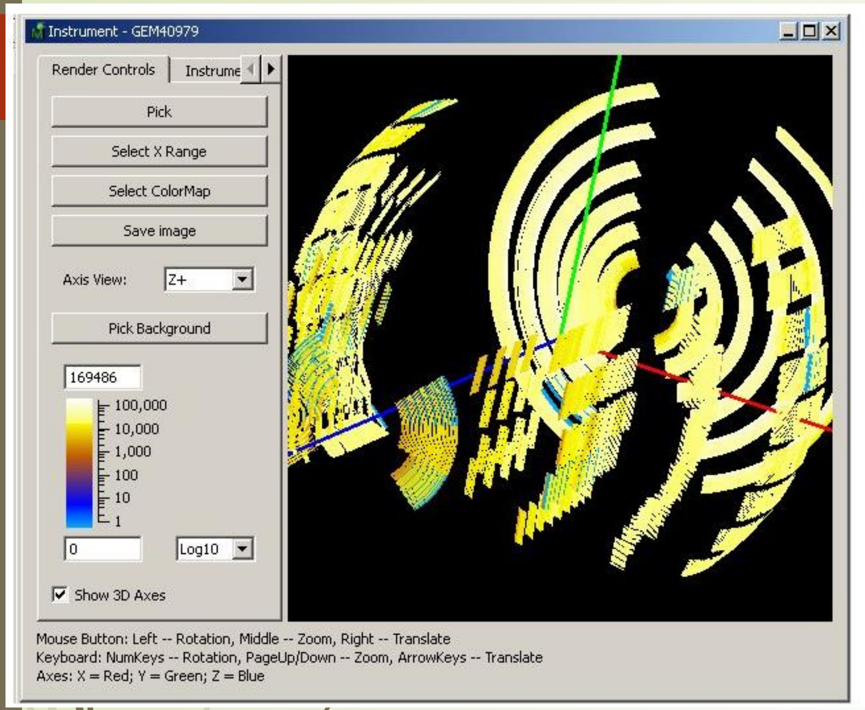
O. Arnold [a,b], J.C. Bilheux [c], J.M. Borreguero [c], A. Buts [a], S.I. Campbell [c], L. Chapon [a,d], M. Doucet [c], N. Draper [a,b], R. Ferraz Leal [d], M.A. Gigg [a,b], V.E. Lynch [c], A. Markvardsen [a], D.J. Mikkelson [e,c], R.L. Mikkelson [e,c], R. Miller [f], K. Palmen [a], P. Parker [a], G. Passos [a], T.G. Perring [a], P.F. Peterson [c], S. Ren [c], M.A. Reuter [c], A.T. Savici [c,*], J.W. Taylor [a], R.J. Taylor [c,g], R. Tolchenov [a,b], W. Zhou [c], J. Zikovsky [c]

Common to all instruments

Common to SXD, NPD experiments on DREAM

~Common to Magic, NMX, DREAM for SXD

Data manipulation in spectrometer space



Detector representation + selection of sectors
Space projections (2θ, ω, ϕ, Tof, time(*in situ*), T, H…)

OK

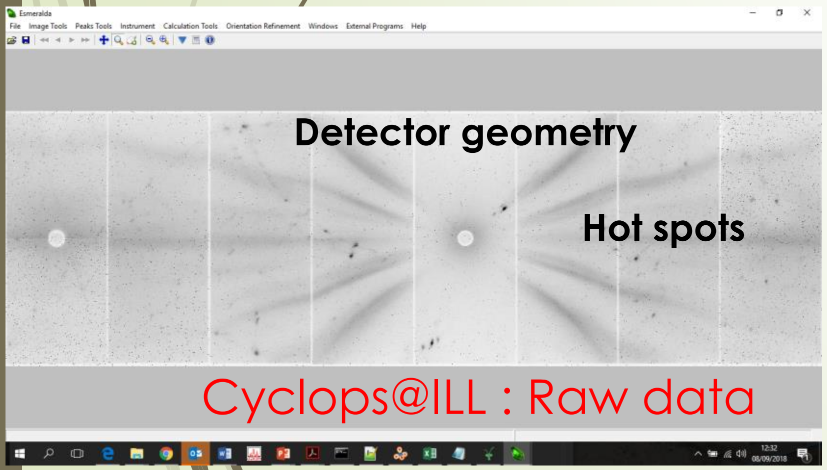
Already in Mantid
➔ Adaptation for CC
(test + ergonomy)

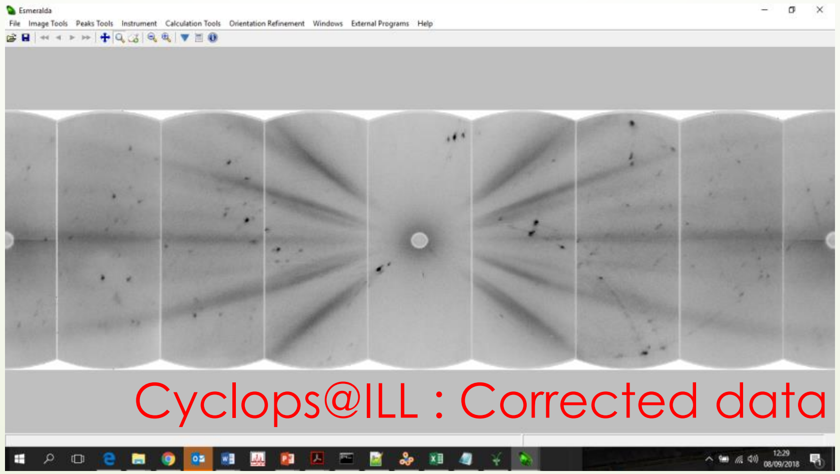
Correction of detectors electronic efficiency + distortion
Correction of beam intensity variation /monitor
WFM mode handling/stitching

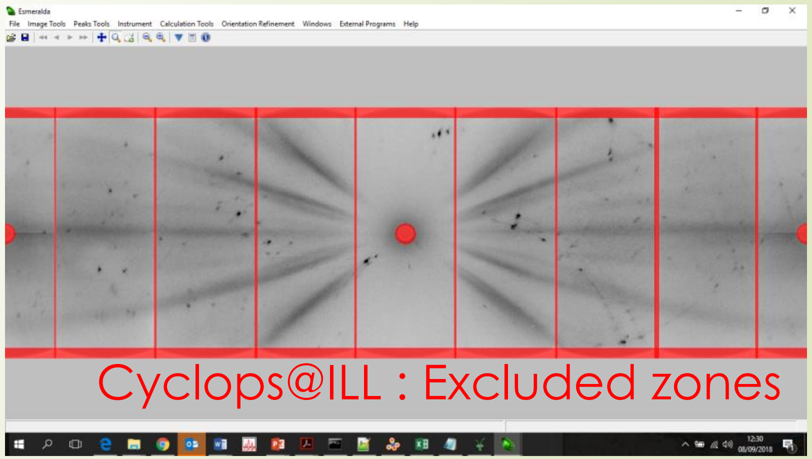
Some in Mantid
➔ Adaptation for HC

Easy configuration of excluded zones
Excluded zones » // Sample Environment

In Mantid ?
➔ Adaptation for UP







CC : Cold commissioning HC : Hot Commissionning UP : User Program

Peak search – Indexing –Reciprocal space

Peak search
- I/noise threshold
- LFM (Fourier)
- Manual option ???

OK

Indexing
- Cell limits + FFT (reciprocal space)
- Cell limits + Duisenberg approach (direct space)
- Known cell + few peaks
- Import UB (Definitions !)

OK

Reciprocal space
- Layers, Rows, Difference,…

OK

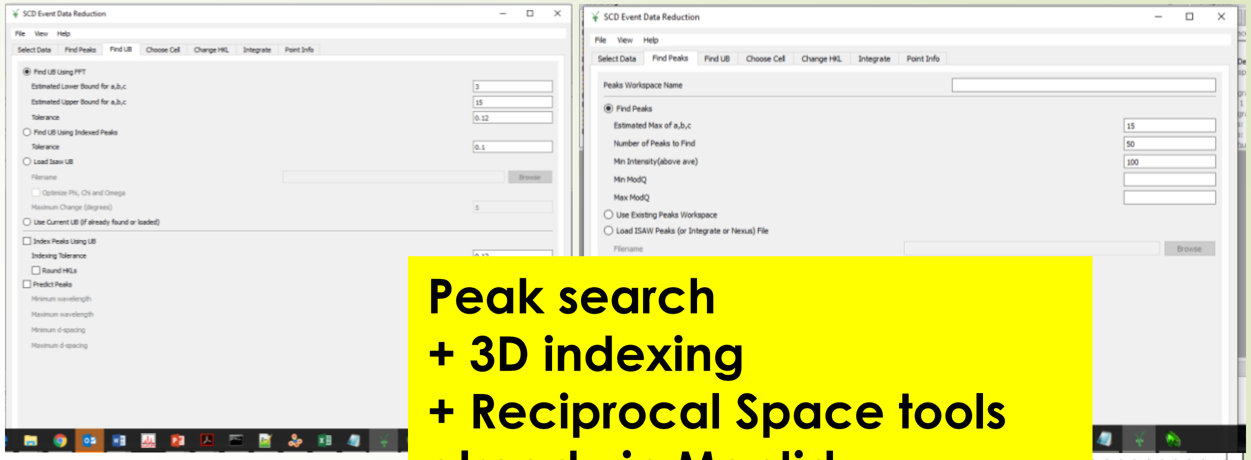
Handling of multiple crystals ?
- Polycrystal (1-3 components)
- Twinned crystal
- Epitaxial growth

Incommensurate case (mag./nuc.)

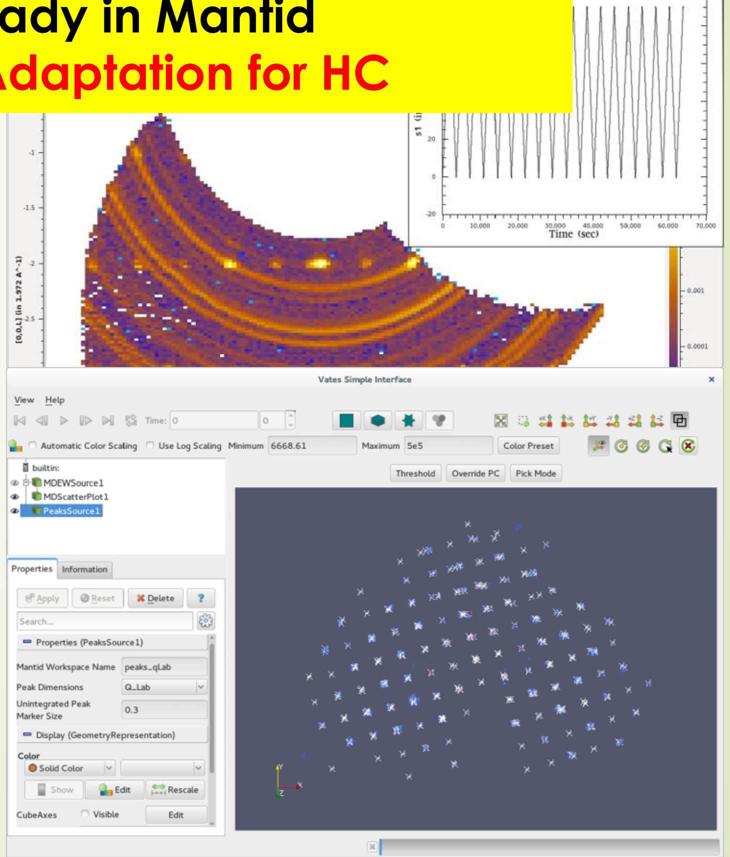
Known cell from XRD
+ manual peak assignment ?

Reciprocal space mapping

Hand assignment of peaks

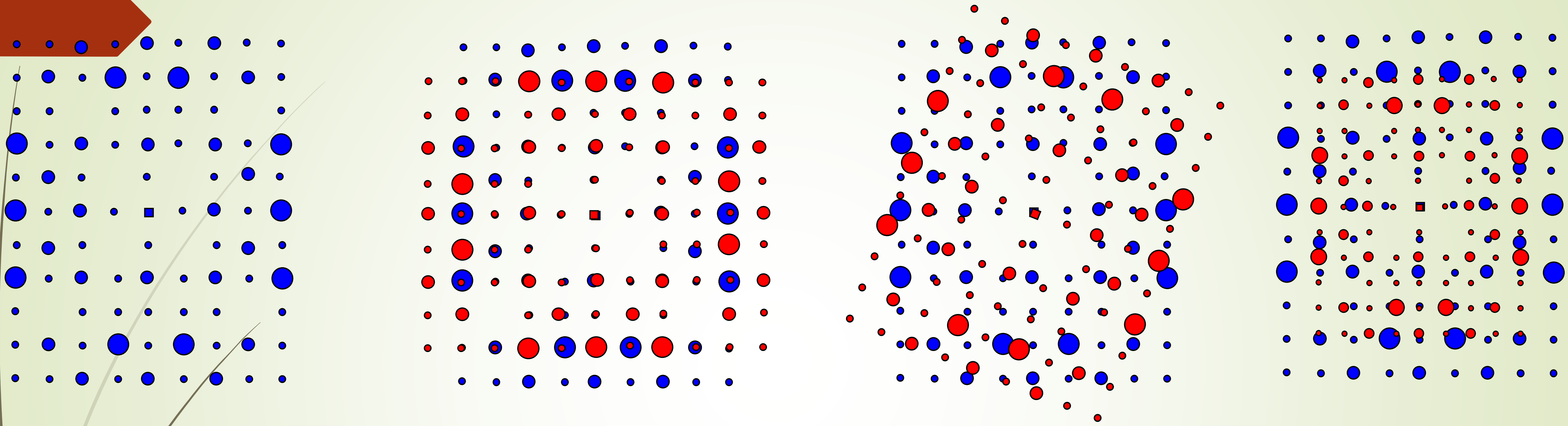


Peak search
+ 3D indexing
+ Reciprocal Space tools
already in Mantid
➔ Adaptation for HC



Steller *et al*. J. Appl. Cryst. (1997). 30, 1036-1040.
Duisenberg, J. Appl. Cryst. (1992). 25, 92-96.

Handling of multiple cells



Definition of multiple UBs

- **Hand assignment of peaks ➔ domain 1, domain 2,…**
- **Exclusion list + iterative indexing** → ***K-search***
- **Exclusion list + twin law**

Maximum number of UBs ?
Combination of twinning + incommensurate

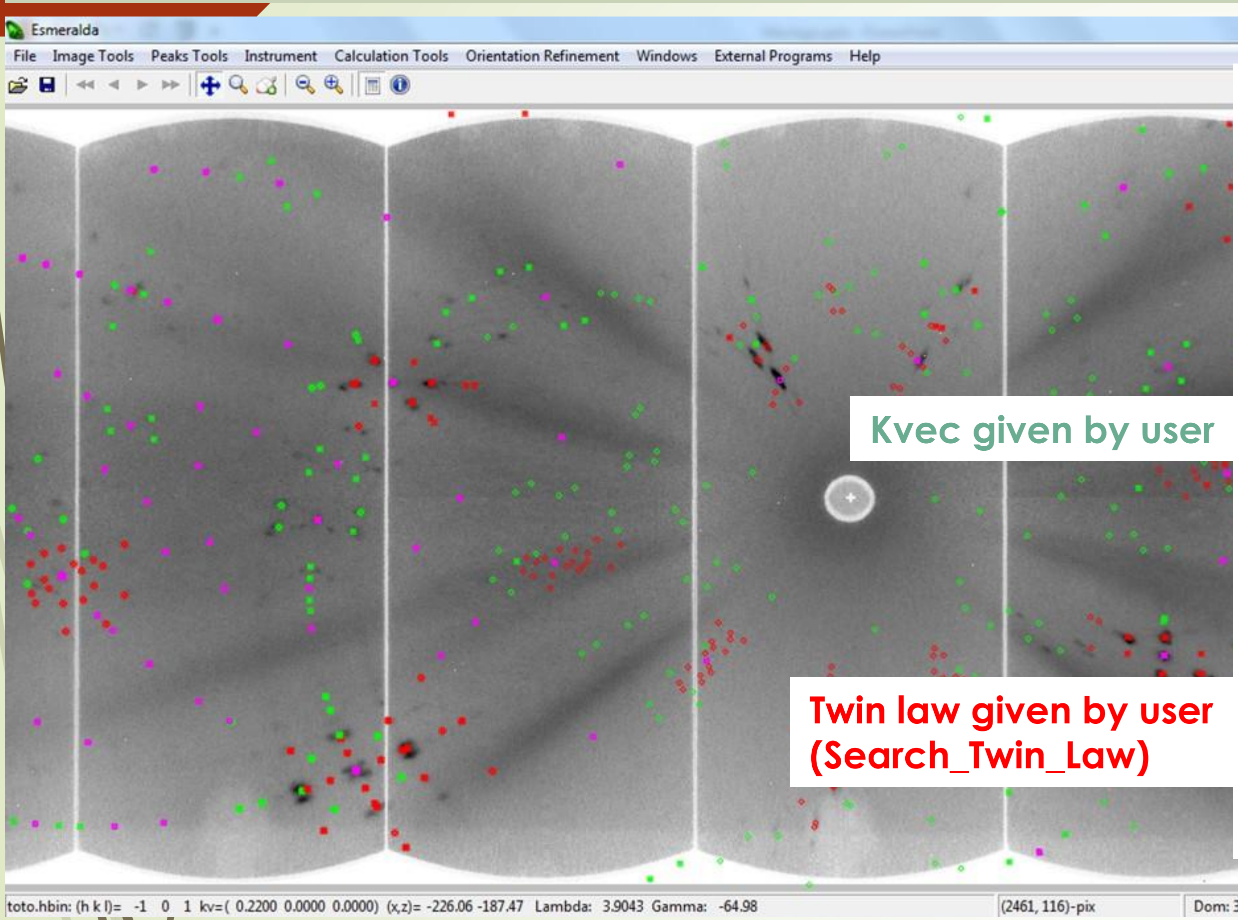
Mag. Incommensurate on Magic
➔ SOUP for DREAM / HC for Magic ?

Historical references :
DIRAX : Duisenberg, A. J. M. (1992). J. Appl. Cryst. 25, 92±96
BRUKER : Sparks, R. A. (1999). GEMINI. Bruker AXS, Madison, Wisconsin, USA.

An example from last week (CYCLOPS@ILL + ESMERALDA LAUE SUITE)

Twinning + Incommensurate phase at low Temp.

Coll. H. Dufour, L. Porcar, D. Bourgault, S. Pailhès Institut Néel & ILM Lyon




```
TITLE  Hypothèse Minimaliste : 3 variants quadratiques pour la modulation

!       Maille type D10 I4/mmm
!          a          b          c        alpha      beta       gamma
Cell     4.21400    4.21400    5.95367    90.0000    90.0000    90.0000
!       Space Group symbol
SpGR    I 4/m m m

! Modulation (Introduire +k et -k imperativement)
KVEC  0.2250 0.0000 0.0000 harmonic 2 multik    ! +k1vec
KVEC  0.7750 0.0000 0.0000 harmonic 2 multik    ! -k1vec

!         Phi_x      Phi_y     Phi_z   of  Busing-Levy U-matrix
ORIENT      17.6627  301.5359  161.1652

TWIN_nam  2 axes 4 de maclage [110] et [1-10] obligatoires
TWIN_typ  4
!TWIN_rot  1.0000 -1.0000  0.0000       0.0000  ! Cubi==>Tetra Individu 0 (Reference)
TWIN_rot   1.0000 -1.0000  0.0000      90.0000  ! Cubi==>Tetra Individu 1
TWIN_rot   1.0000  1.0000  0.0000      90.0000  ! Cubi==>Tetra Individu 2
```

Twin law from the 2 UBs (Oxford Crysalis)

```
UM SARRAY 0  -0.00240   0.01006   0.03419   -0.00319  -0.03378   0.01005    0.05291  -0.00158   0.00275
13.37086 20.10458 19.84878  89.9989 90.9448 90.0070  5334.93
TL is the matrix for the TWIN card in SHELXL provided it describes the comp 1 to comp N transf.
TL=UB1-1*UB2:  -1.0000  -0.0001  -0.0004     0.0002  -1.0002  -0.0004     0.0446   0.0001   0.9999   det 1.000087
Rot= 179.9919 deg around -0.0021 -0.0006 1.0000 (rec)  0.0164 -0.0001 0.9999 (dir)
```

Peak integration (Standard 3D single crystal)

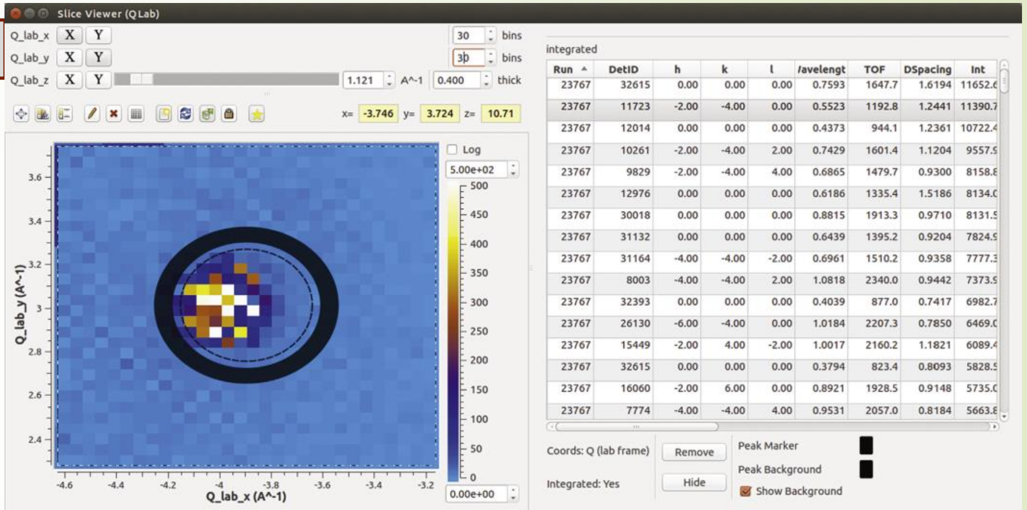
Integration at HKL calculated position :

- « Shoebox » (spherical, cylindrical, elliptic) **OK**
- Intensity-based (2D fitting : Seed-skewness, Lehman-Larsen)
- Learnt profile (area-averaged, intensity-averaged, profile function)

Background substraction **OK**

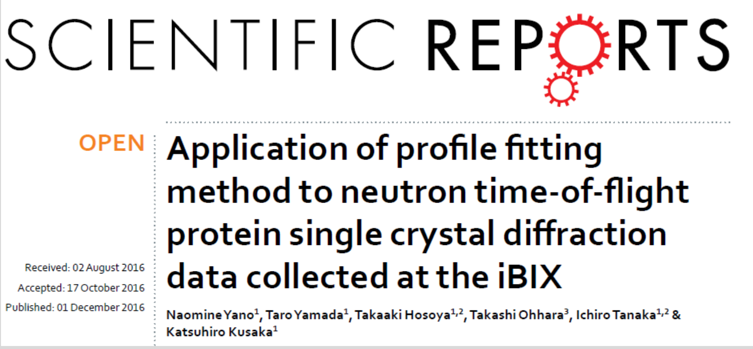
Standard deviations on Bragg intensities (« sigmas »)

(Shoebox) + 2D fitting integration already in Mantid
➔ **Adaptation for HC**



Stargazer (TOF adaptation of Denzo, MosFLM…)

Weak peaks /Strong peaks (supercell, mag.)
Weak peak / High Background (H)
Irregular background (SE, dif. streaks)
Overlapping

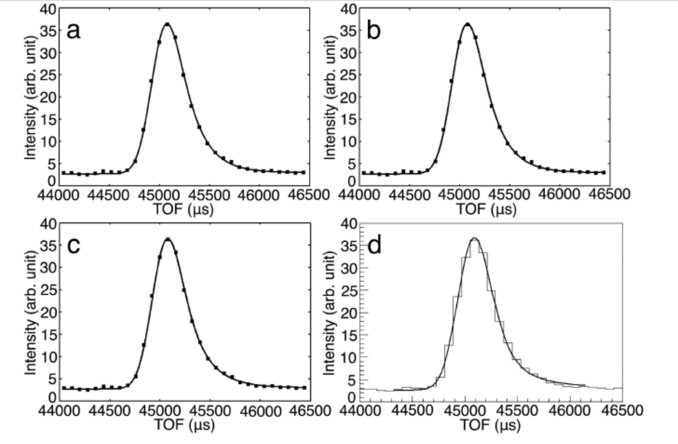

SCIENTIFIC REPORTS

OPEN

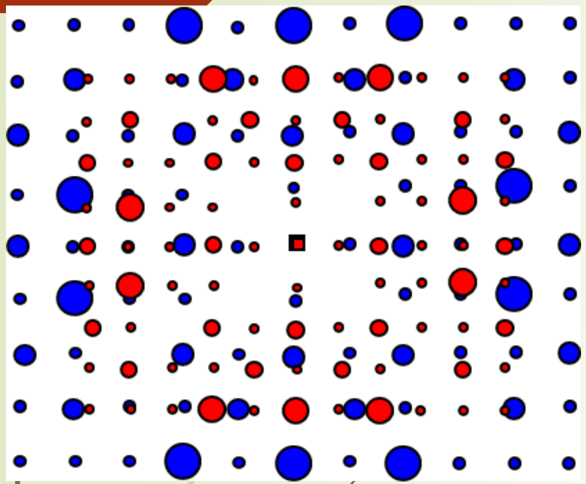
Application of profile fitting method to neutron time-of-flight protein single crystal diffraction data collected at the iBIX

Received: 02 August 2016
Accepted: 17 October 2016
Published: 01 December 2016

Naomine Yano[1], Taro Yamada[1], Takaaki Hosoya[1,2], Takashi Ohhara[3], Ichiro Tanaka[1,2] & Katsuhiro Kusaka[1]



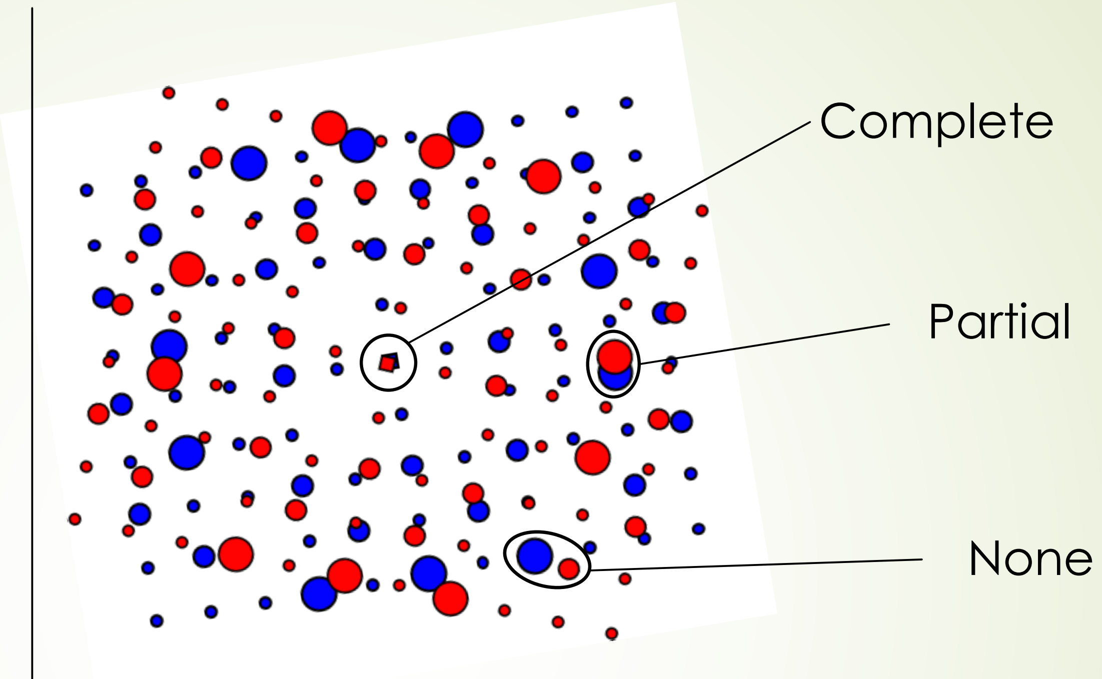
Peak integration : Incommensurate – Twins – Multiple crystals



Indexing
h k l m + **qvec** (n_1**a*** + n_2**b*** + n_3**c***)

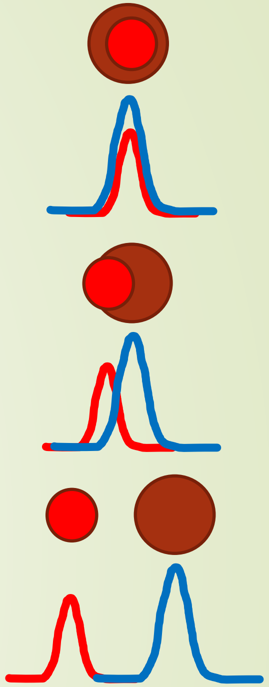
Limited overlap (neglected)
- 0 < m < 4 e.g.
- Extinction rules (superspace group)

Not in Mantid
➔ Adaptation for HC-UP…



Indexing
h1 k1 l1 / h2 k2 l2 /… domains

Variable overlap
- Complete
- Partial
- None



Profile fitting

Not in Mantid
➔ Adaptation for UP…

Normalizations – Corrections – Bragg intensities…

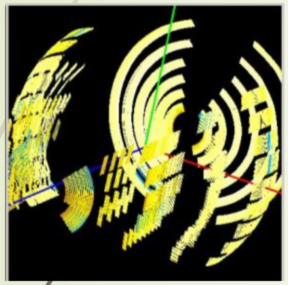
Least-squares optimization / **Known dependencies**

$$I(\mathbf{h})_{xz} = s\,Lp(\theta,\varphi)\Phi(\lambda,\mathbf{p}_{\varphi})\lambda^3 E_{ff}(\lambda,x,z)A(\lambda,x,z,\mathbf{p}_{abs})G(\mathbf{h},\mathbf{p}_g)E(\lambda,|F(\mathbf{h})|,\mathbf{p}_{ext})F^2(\mathbf{h},\mathbf{p}_s)$$

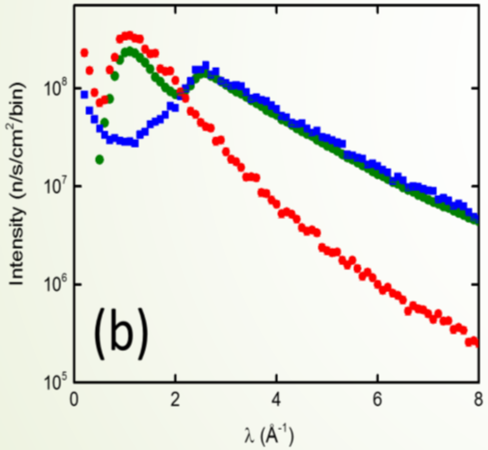
~OK ?

Beam Monitor

Geometry

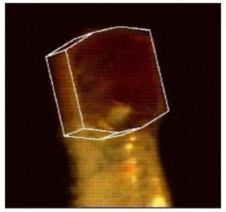


PSC settings



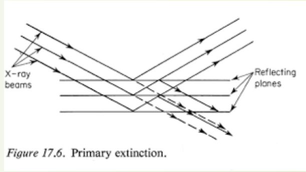
Detector Calibration

Absorption



Other !!!

Empirical extinction



Adaptation in Mantid
→ HC

Least square optimization : Laue averaging ?

From Esmeralda Laue Suite – Laue Intensity (http://lauesuite.com/)

OUTPUT FILES

Diffraction data

3D
- SHELX hklF4 Format

  h k l F2(HKL) scale t1 t2 t3 t4 t5 t6

Superspace 4D, 5D..
- SHELX hklF4 Format

  h k l m (n…) F2(HKL) scale t1 t2 t3 t4 t5 t6

Twins, Overlapped
- SHELX hklF5 Format

h k l F2(HKL) domain/overlap flag

w/o scale factor, direction cosines

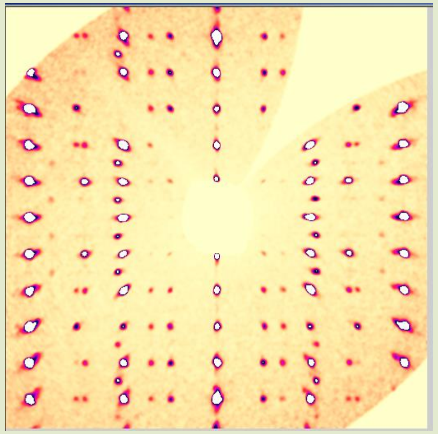
JANA2006, SHELX, FULLPROF,…

Cell parameters / UB-Matrix

Homogene XYZ referentiel at ESS
Transformation between definitions
Export tools

Other instrument

Reciprocal volume/layers for diffuse scattering



Tiff format ?

DISCUS, FAULTS,…

DREAM TIMELINE

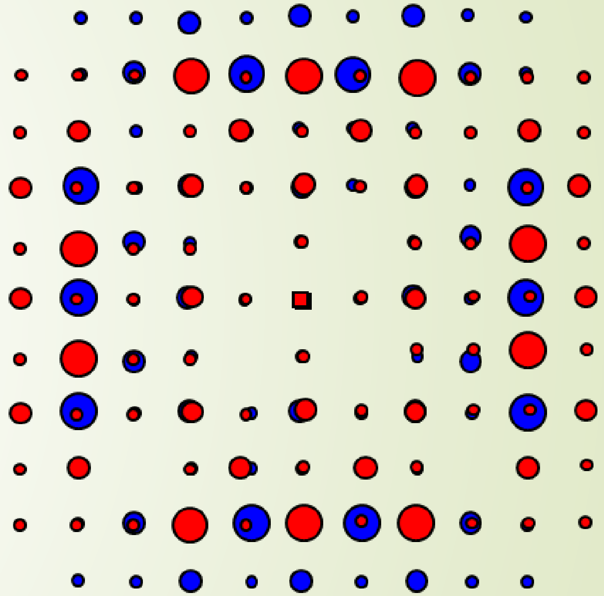
- 2017-2018: Final Design Development
- 2018-2023: Construction & Installation & Cold Commissioning
- 2023-2023: Hot Commissioning (Friendly Users)
- 2023 (end): General User Operations

HKLF5 file format for twins

Warning : Indices refers to twin operation (not only twin law)

(assuming point group mmm)

h	*k*	*l*	component	
1	-2	3	1	equivalent singles
-1	-2	-3	1	
-1	-2	-3	2	not equivalent to the above singles
-1	-2	-3	-2	equivalent groups
2	0	-4	1	
1	2	-3	-2	
-2	0	-4	1	
4	1	1	-2	not equivalent to the other groups shown here
1	-2	-3	-3	
-1	1	2	1	



1. **DREAM shall enable data collection up to a Q_{max} of 25 $Å^{-1}$.**
2. **DREAM shall enable data collection down to a Q_{min} of 0.2 $Å^{-1}$ (the upgrade design will allow for Q_{min} = 0.01 $Å^{-1}$).**
3. DREAM shall provide a flexible choice between the high resolution and high intensity by pulse shaping within two orders of magnitude from ~10^{-2} ms to ~ms.
4. DREAM should deliver data for samples as small as 1 mm^3, in high intensity mode within 30 minutes.
5. DREAM shall deliver a best resolution of Δd = 0.00035(10) Å near backscattering.
6. DREAM shall enable data collection for samples as small as 10 mm^3 with best resolution.
7. DREAM shall be able to detect structural changes with a time resolution of 10 ms for samples as large as 0.5 cm^3.
8. **DREAM shall enable TOF Laue single crystal measurements.**
9. DREAM shall provide the infrastructure to support fast in-situ measurements and quick user turnover.
10. DREAM should provide user community with the wide range of generic and instrument-specific sample environment within its upgrade path.
11. DREAM should reduce the sample environment turnaround time as much as reasonable achievable.
12. DREAM shall be compatible with high magnetic field sample environments.