TOPAZ
Data Reduction & Analysis

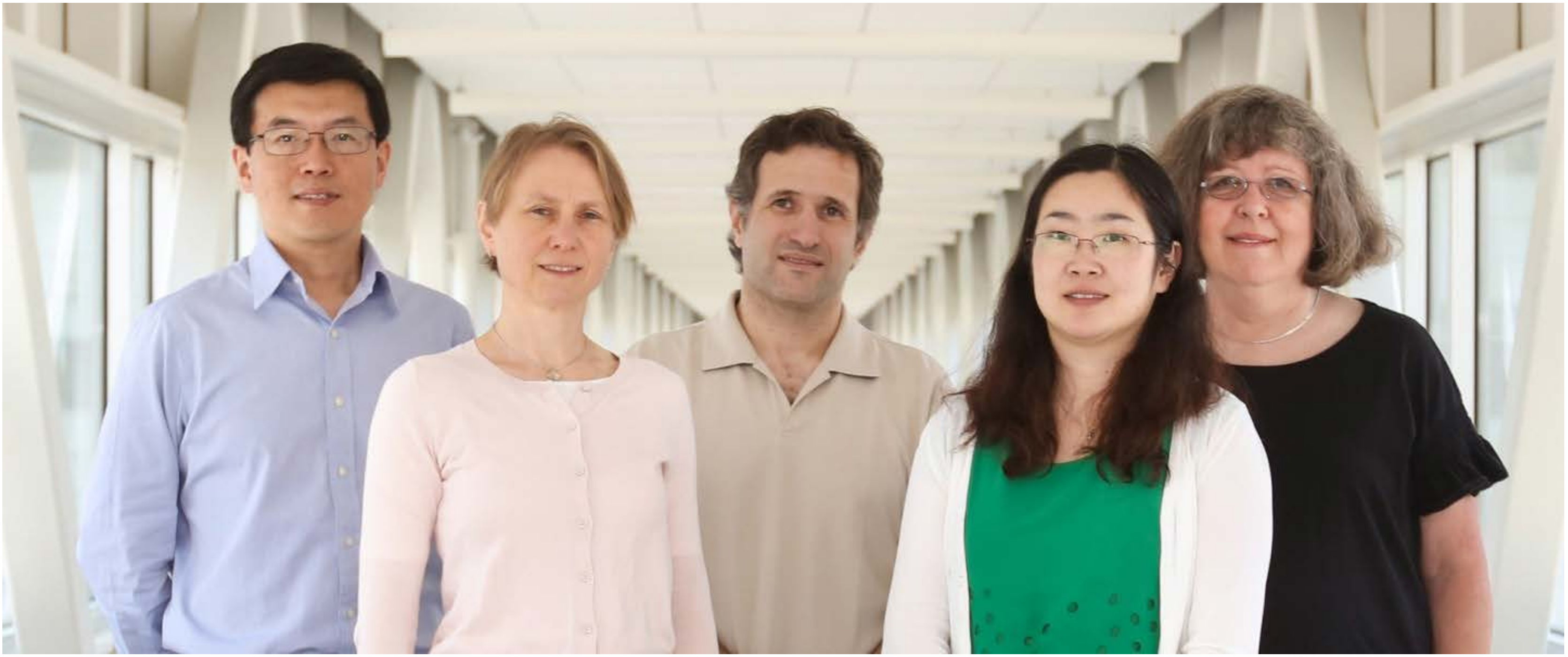
Xiaoping Wang

Neutron Scattering Division
Oak Ridge National Laboratory

Single Crystal Diffraction Workshop
DMSC/ESS, Lund
Sept 12, 2018

ORNL is managed by UT-Battelle
for the US Department of Energy

The SNS TOPAZ Team



Xiaoping Wang, Christina Hoffmann, António M. dos Santos, Helen He, Vickie Lynch

Left to right

neutrons.ornl.gov/topaz

OAK RIDGE National Laboratory | HIGH FLUX ISOTOPE REACTOR | SPALLATION NEUTRON SOURCE

Neutron single crystal instruments at ORNL

http://neutrons.ornl.gov/instruments

HIFR

- **HB-3A** Four-Circle Diffractometer
- **CG-4D Imagine** Laue Diffractometer

SNS

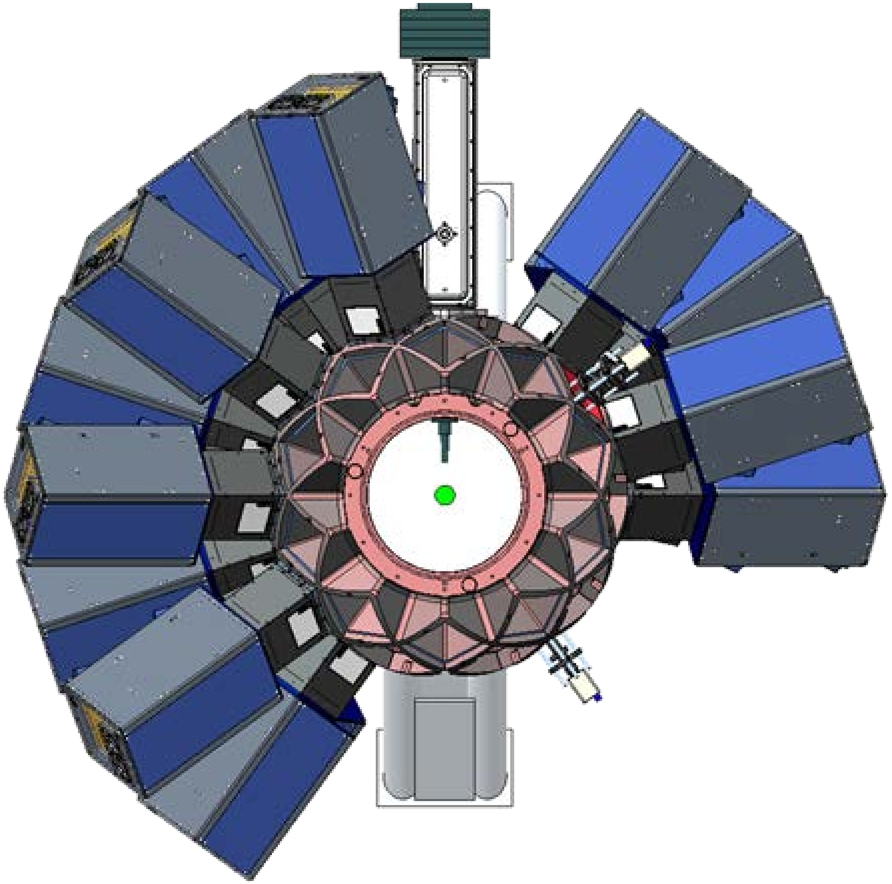
- **BL-3 SNAP** Spallation Neutrons and Pressure Diffractometer
- **BL-9 CORELLI** Elastic Diffuse Scattering Spectrometer
- **BL-11B ManDi** Macromolecular Neutron Diffractometer
- **BL-12 TOPAZ** Single-Crystal Diffractometer

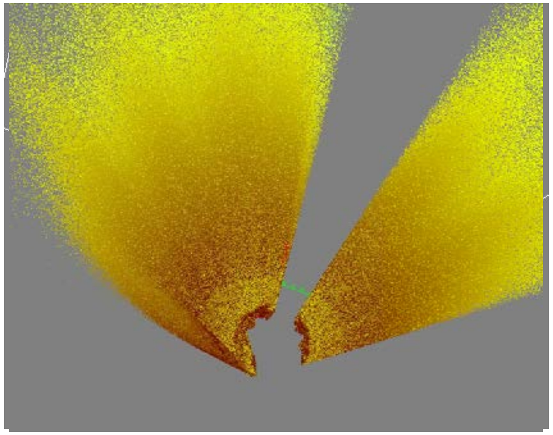
3*D* Reciprocal space mapping

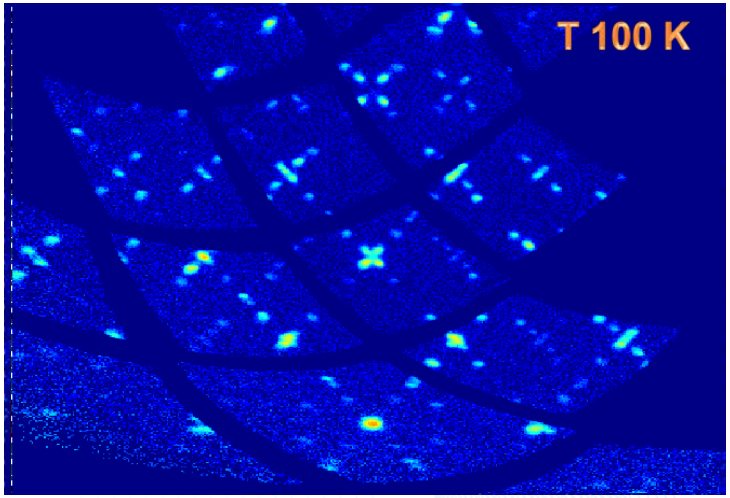
Neutron wavelength-resolved Laue

$\boldsymbol{d_{min}}$ **= 0.25 Å**

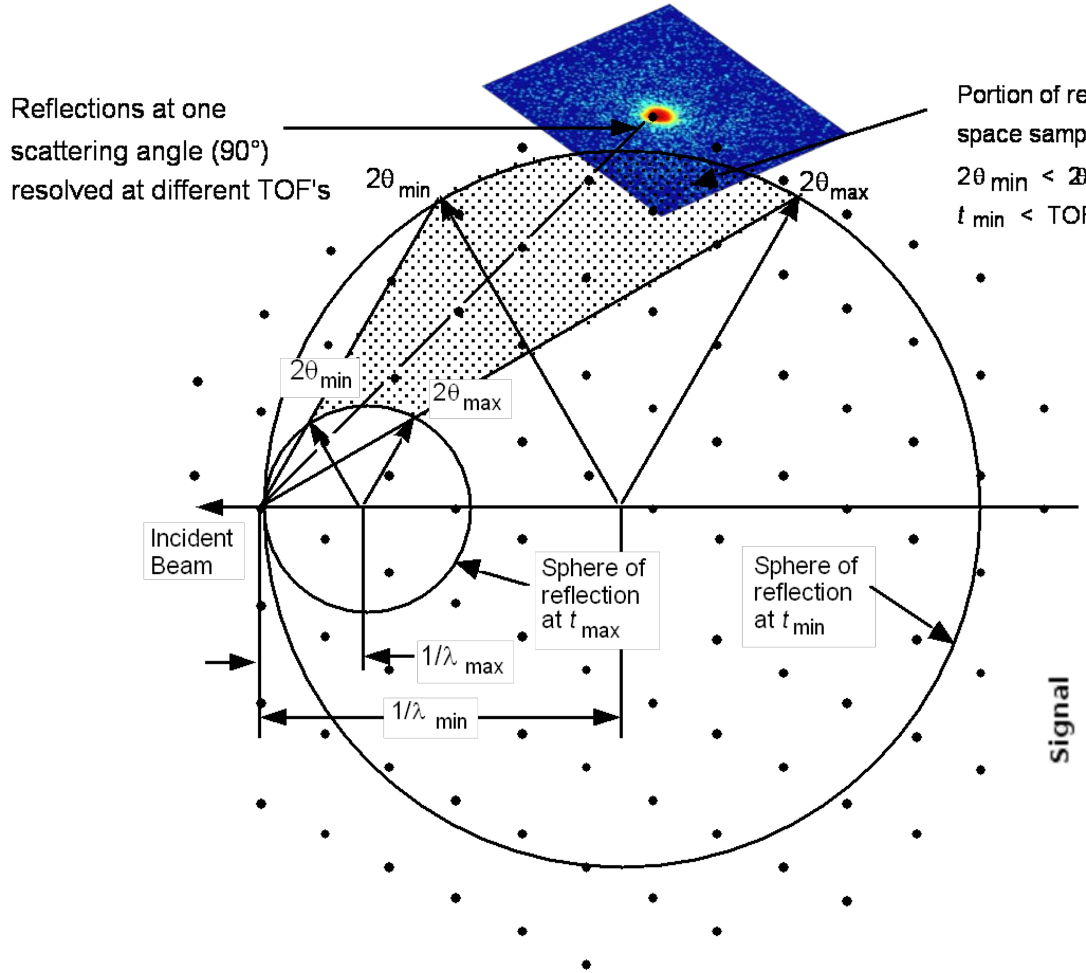
$\boldsymbol{Q_{max}}$ **≈ 25 Å^{-1}**

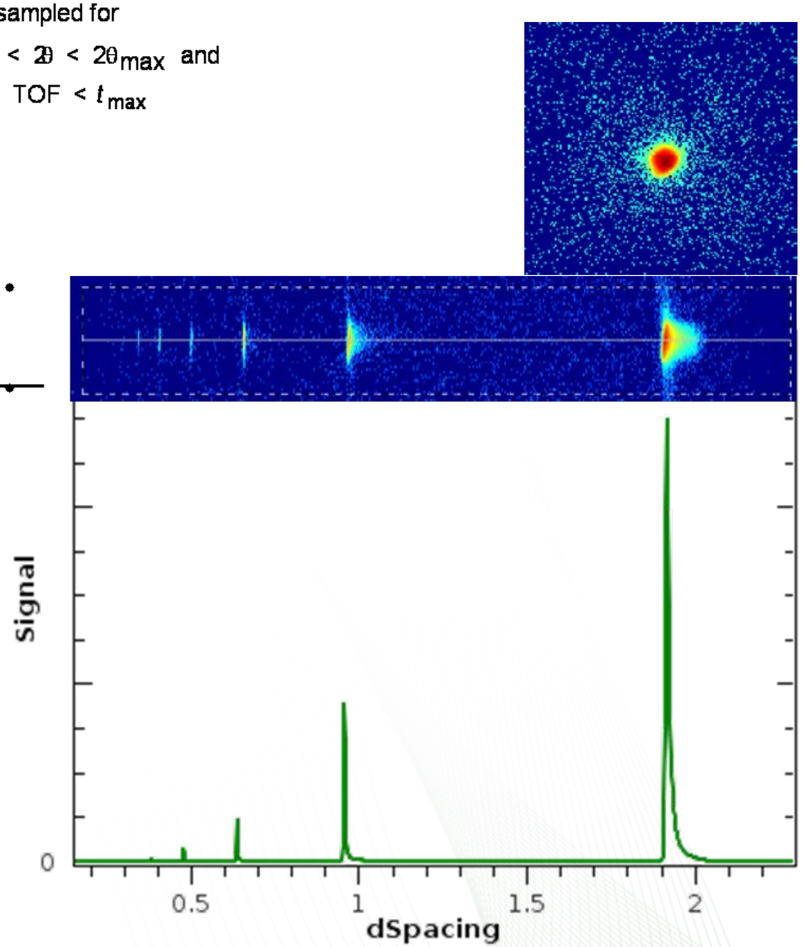




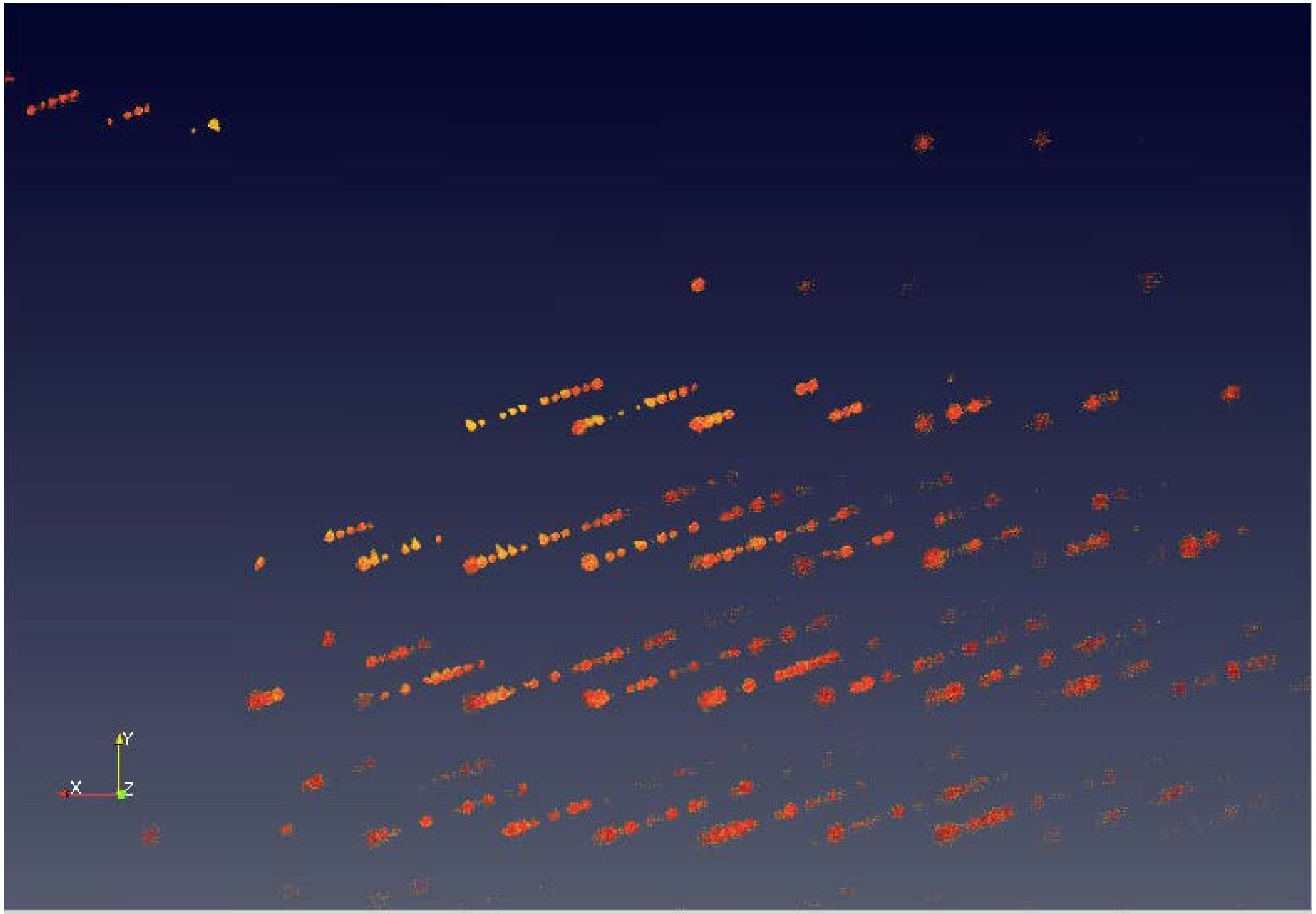


Neutron Wavelength-resolved TOF Laue

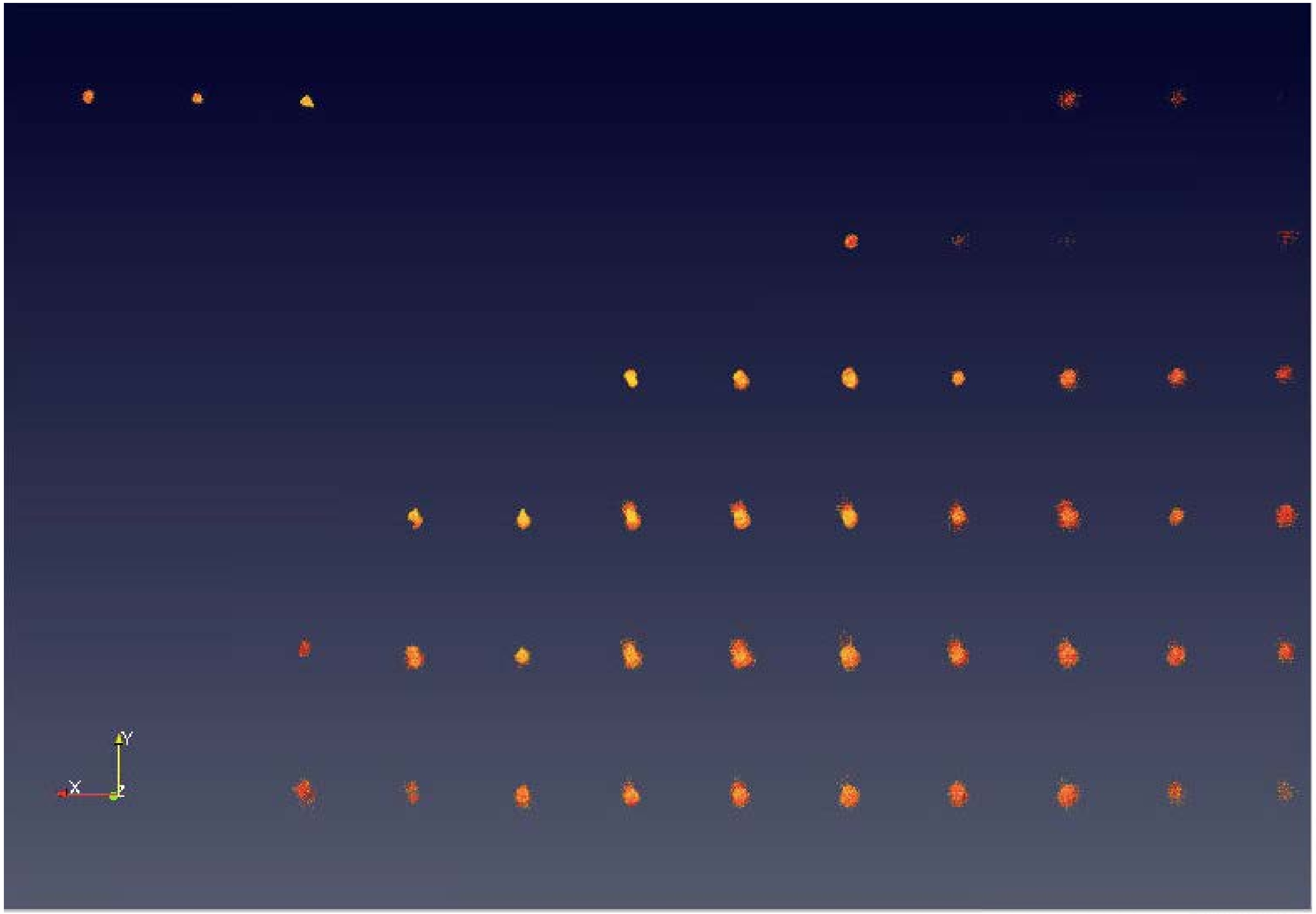




Single crystal peaks in Q space



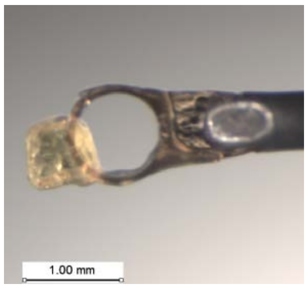
Single crystal peaks in Q space



Sample environment

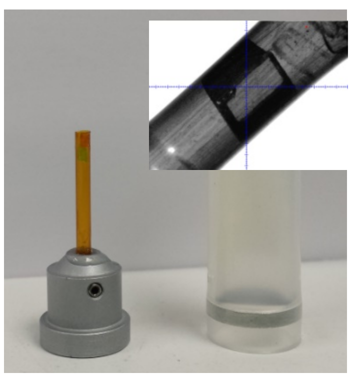
Single Crystal Sample Mount

MiTeGen loop **(1 mm ϕ)**

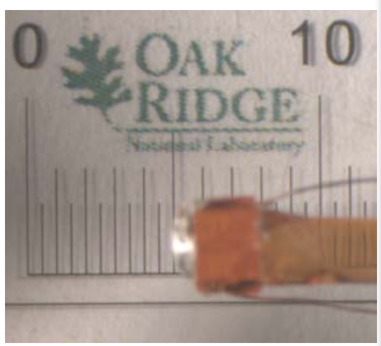


Coated with perfluorinated grease Prytox

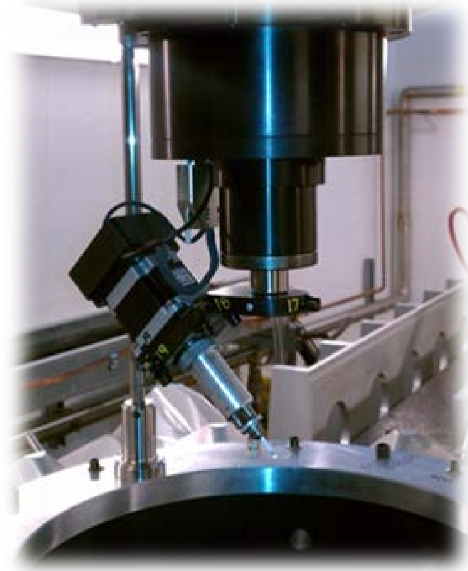
Kapton tube **(1 - 3 mm ϕ)**



Glued onto the tip or inside a Kapton tube



Wired for E-field experiment



Crystal Logic Goniostat

0.005 mm^3
Unit Cell < 500 $Å^3$

Sub-Millimeter Sized Crystals

Diameter: 0.10 – 4.0 mm, Volume: > 0.1 mm^3

Multiple Area Detectors

Solid Angle Coverage: 3 ster.

Detector 2θ Coverage: 13.5° - 160°

Controlled Sample Environment

CryoStream 700 Plus: 90K – 450K

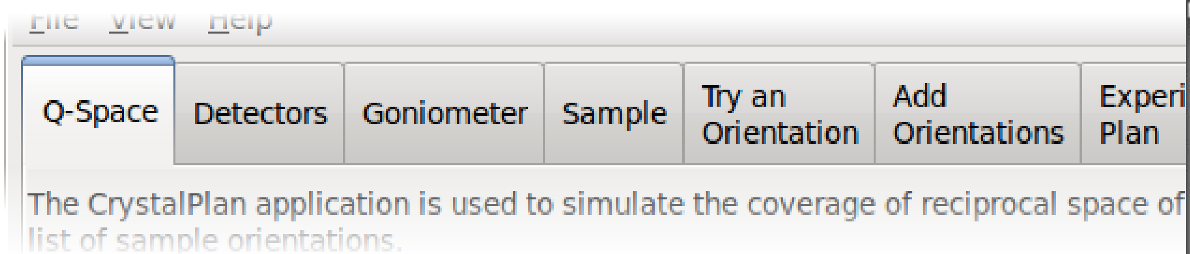
Pulsed Electric Field

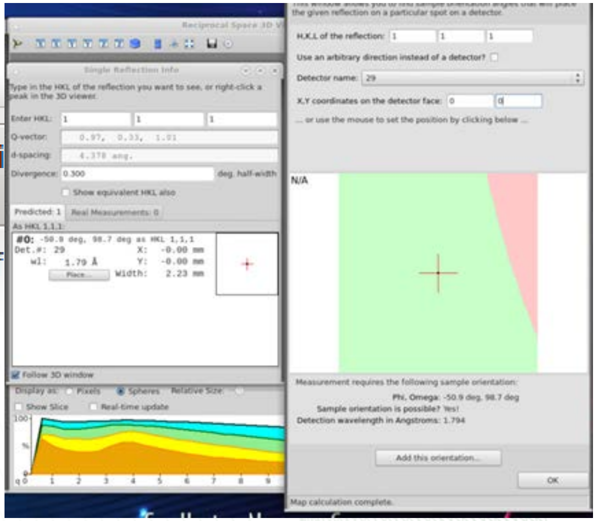
Cryogenic goniometer 5K – 295K (2019)

Experiment planning

CrystalPlan

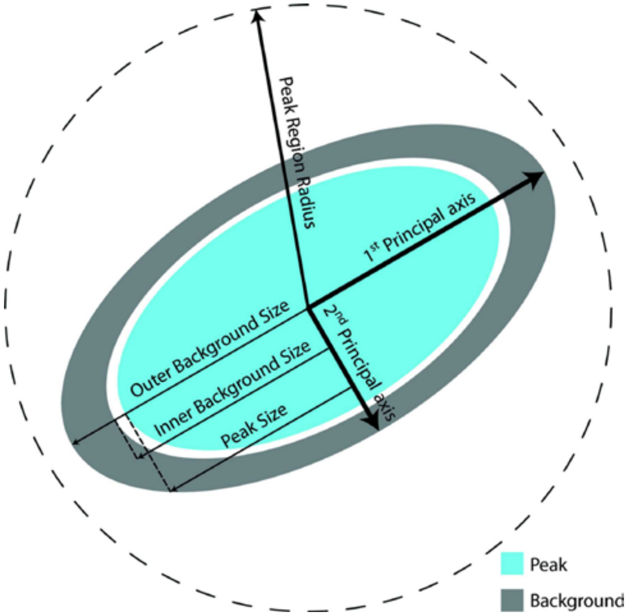
- An experiment planning tool for time-of-flight Laue experiment
- Develop data collection strategy to acquire the most unique data possible, with sufficient coverage but limited redundancy
- User friendly GUI Interfaces
  - Capable of placing an individual peak on selected detector position
  - Maximize the use of available beam time and productivity.

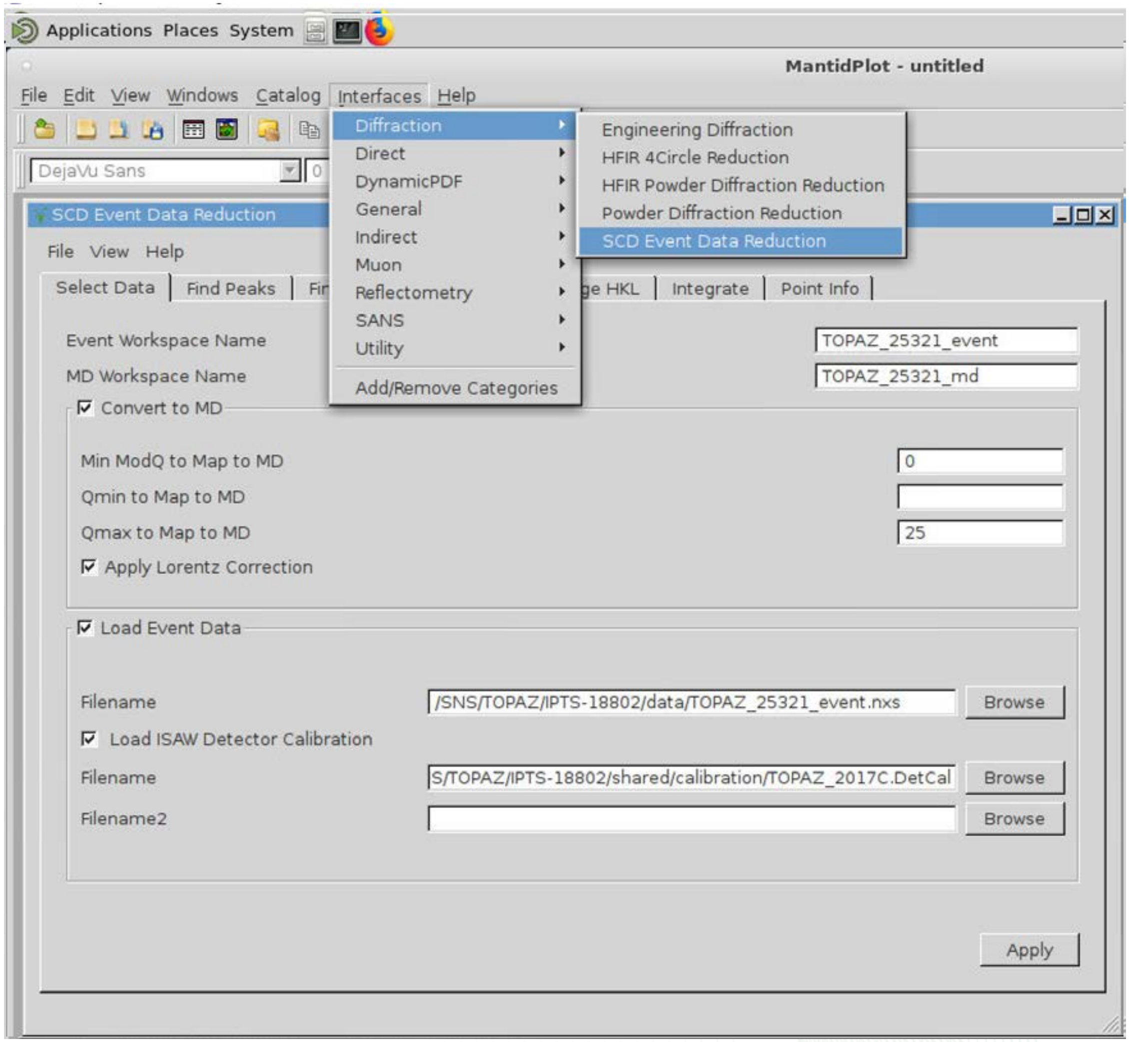




Zikovsky *et. al*, *J. Appl. Cryst.* **44,** 418-423 (2011)

SCD event data reduction





A schematic view of Bragg peak integration using three-dimensional ellipsoids in Mantid.

[IntegrateEllipsoids]

Schultz *et. al*, *J. Appl. Cryst.* **47,** 915-921 (2014)

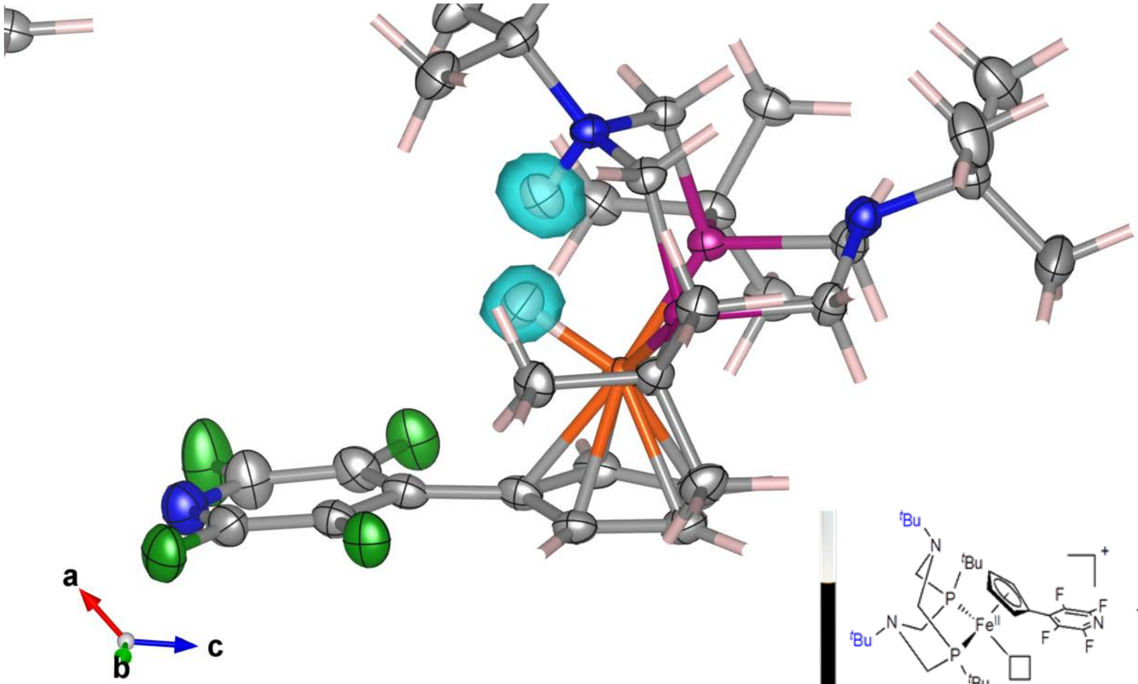
Angewandte Communications

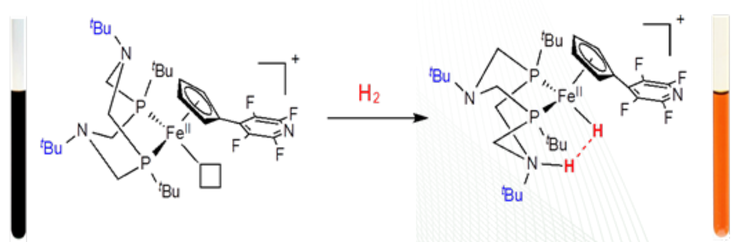
VIP Heterolytic H_2 Cleavage | Very Important Paper

DOI: 10.1002/anie.201402090

Heterolytic Cleavage of Hydrogen by an Iron Hydrogenase Model: An Fe-H···H-N Dihydrogen Bond Characterized by Neutron Diffraction**

Tianbiao Liu, Xiaoping Wang, Christina Hoffmann, Daniel L. DuBois, and R. Morris Bullock**





Bullock *et. Al, Angew. Chem. Int. Ed*. **53**, 5300 –5304 (2014)

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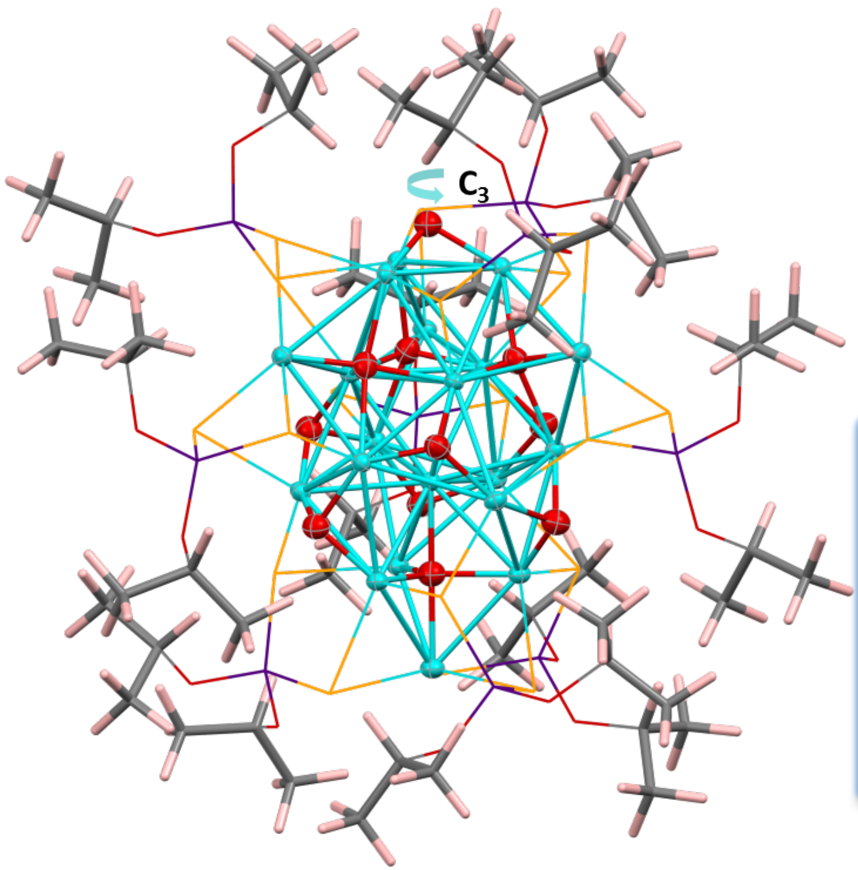
Crystal structure at sub-atomic resolution

Data from a hydrogenated single crystal sample



Cu cyan, Hydride **red**

$C_{81}H_{172}Cu_{20}O_{18}P_9Se_{18}$
a = 18.3282(2) Å
c = 74.752(2) Å
V = 21746.6(6) Å^3
R_1 = 0.0672

Hydrogenated sample

54 Atomic % Hydrogen

Well resolved hydrogen atom positions

10 Hydrides as capping μ_3-H ligands

1 Hydride as a μ_5-H ligand in trigonal-bipyramidal cavity

C.-W. Liu., *et. al*, *Angew. Chem. Int. Ed*. **2015**, *54*, 13604.

Hydrogen bonding in hybrid perovskites

- High power conversion efficiencies (> 22%) for solar cell applications
  - Heavy elements with very high X-ray absorption μ = **526.82** cm^{-1}
  - **Transparent to neutrons** μ = **0.654 + 0.508λ** cm^{-1}
- Effect of H-bonding on structural phase transitions, $CH_3NH_3PbI_3$

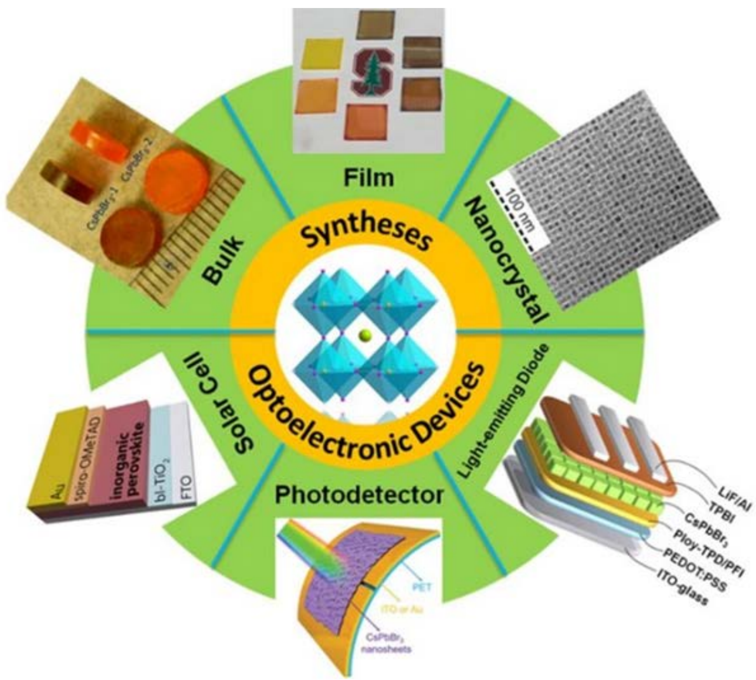
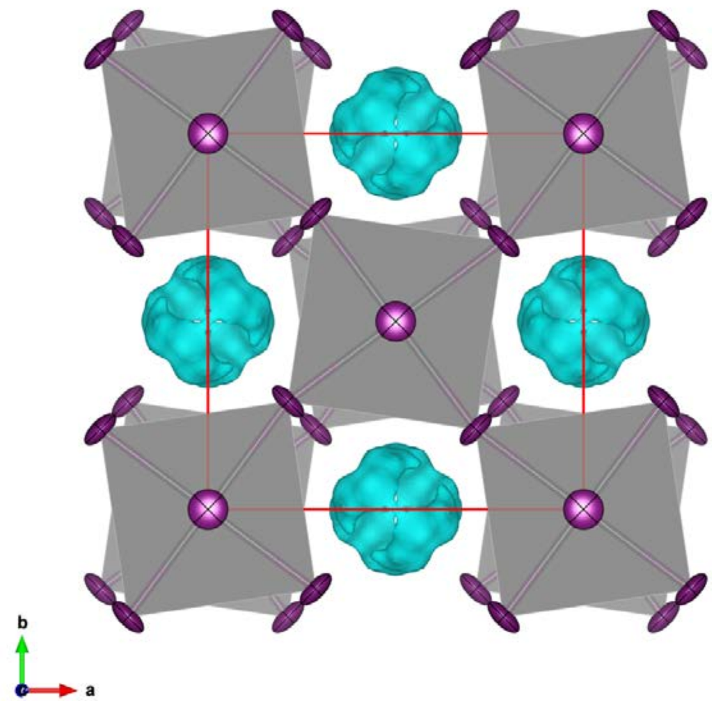


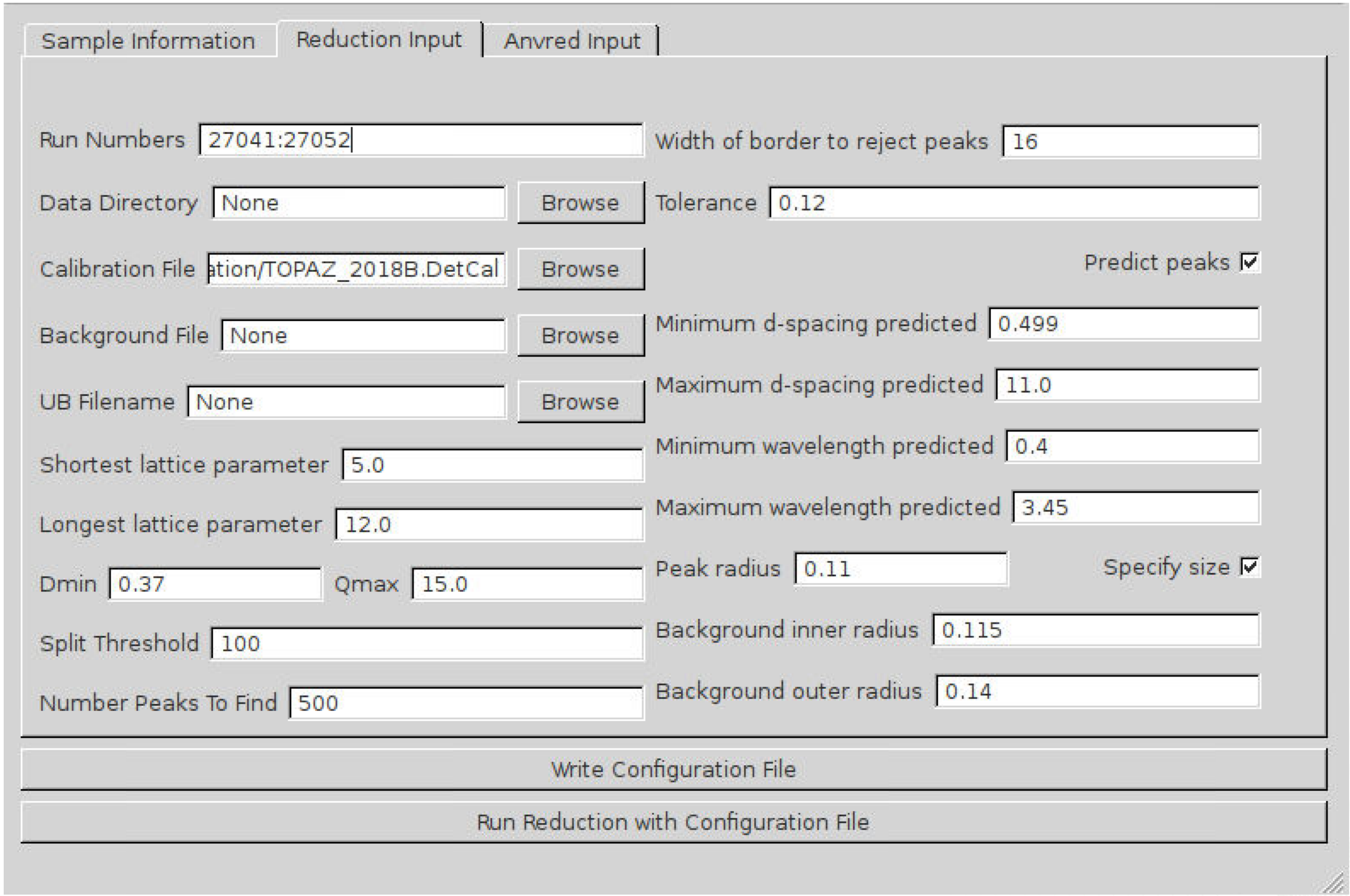
Image from advancedsciencenews.com



J. Chan *et. al*, *Crystal Growth & Design* **2016**, *16*, 2945

Recent development

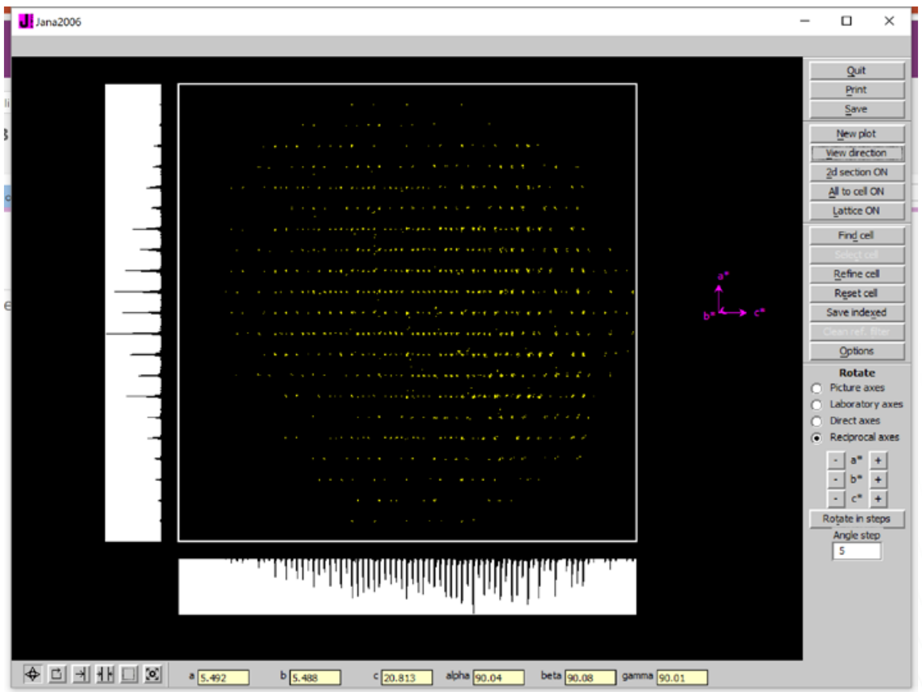
- User friendly GUI based reduction and visualization software



Recent development

- TOF Laue data from modulated single crystal sample
  - Find and index q vectors
  - Integrate main Bragg and satellite peaks
  - Reduce data for structural analysis in JANA2006

Q sample data from Mantid *.topi $\frac{2\pi}{d}$



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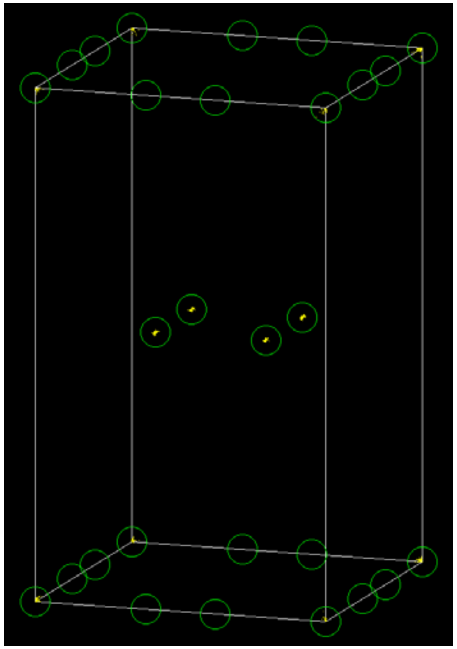
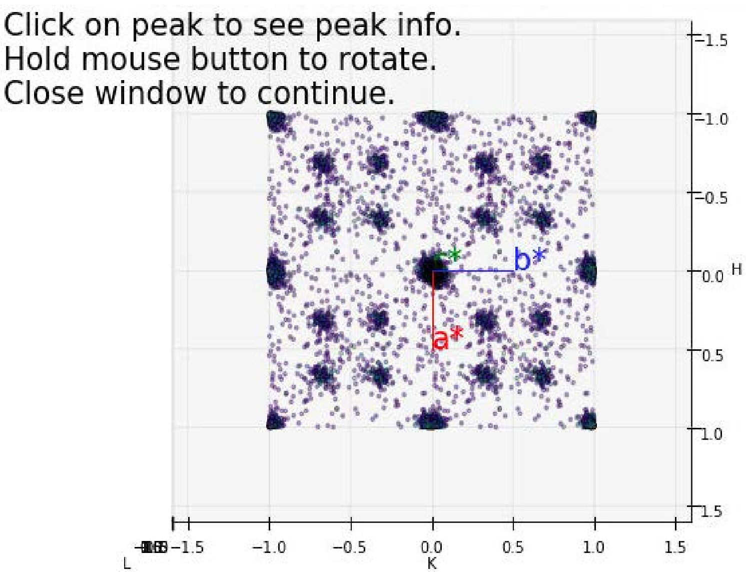
Recent Development

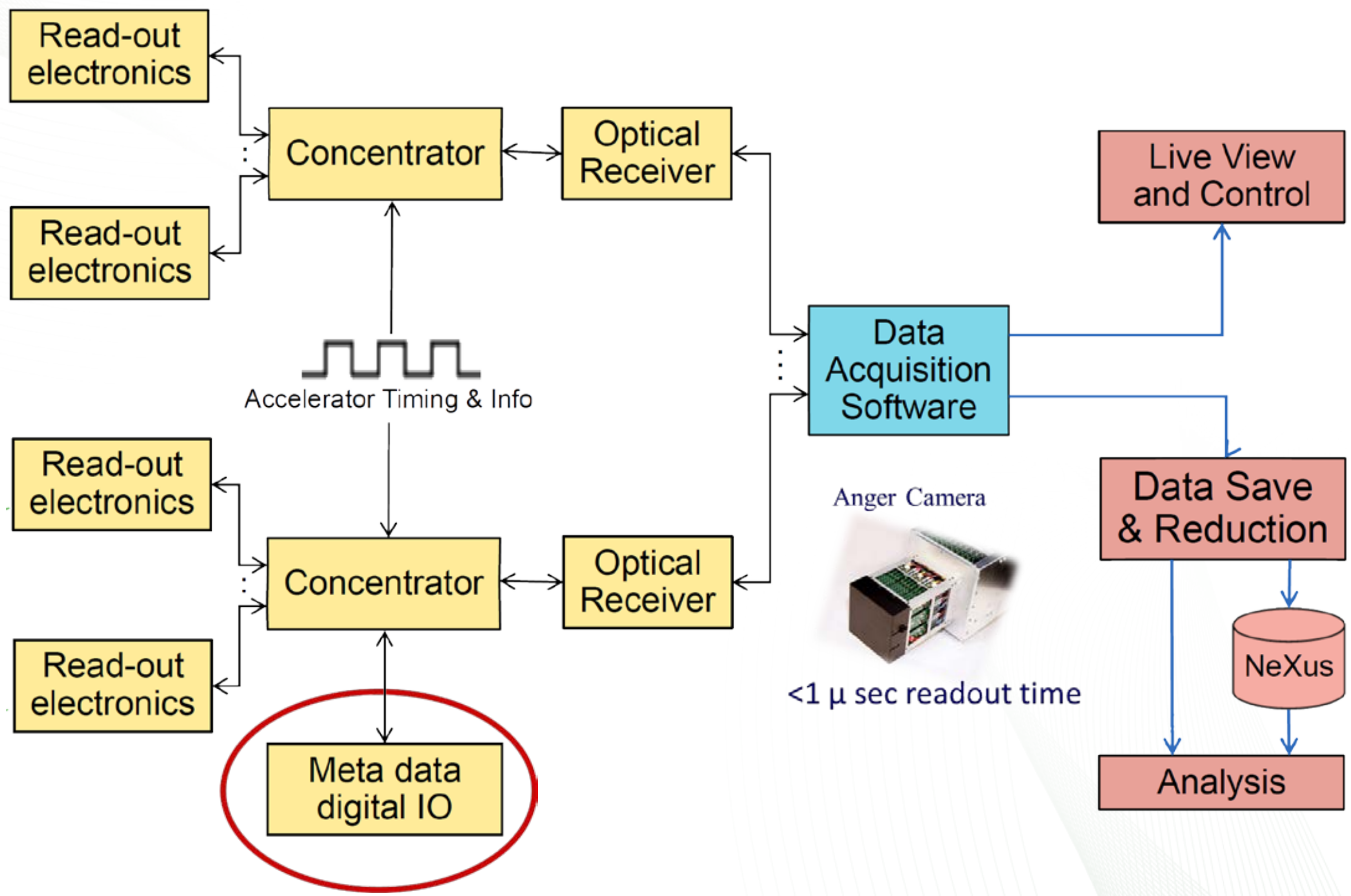
- New Algorithms introduced to Mantid for data reduction from modulated structure

Vickie Lynch
Shiyun Jin ORNL GO! Student





Data Acquisition System at SNS Instruments



SNS Real Time Data Link (RTDL), a 10 MHz, bi-phase mark encoded serial link. Signals have **100 *nanosec.*** resolution.

doi:10.18429/JACoW-ICALEPCS2017-TUBPA05

Event-based data collection for parametric study

Variable temperature study

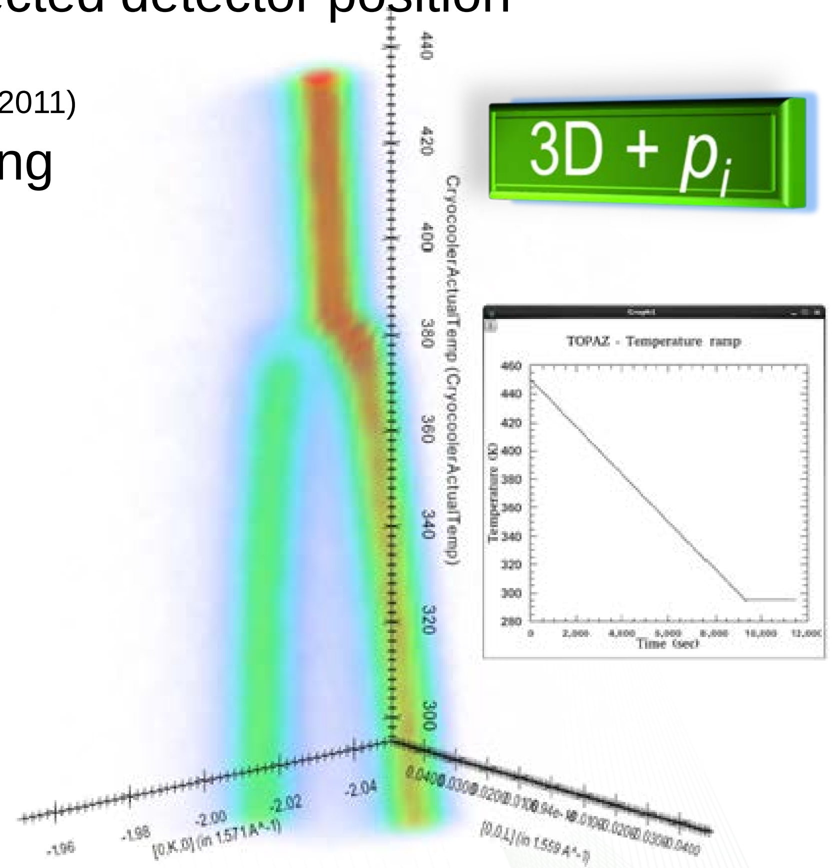
- Place preselected Bragg peak on selected detector position
  - CrystalPlan Zikovsky *et al. J. Appl. Cryst*, **44**, 418 (2011)
- VT measurement while heating / cooling
  - Stationary single crystal
  - Neutron recorded in event mode

    Detector pixel position (x,y)

    Neutron time of flight (λ)
  - Sample temperature (T, K)

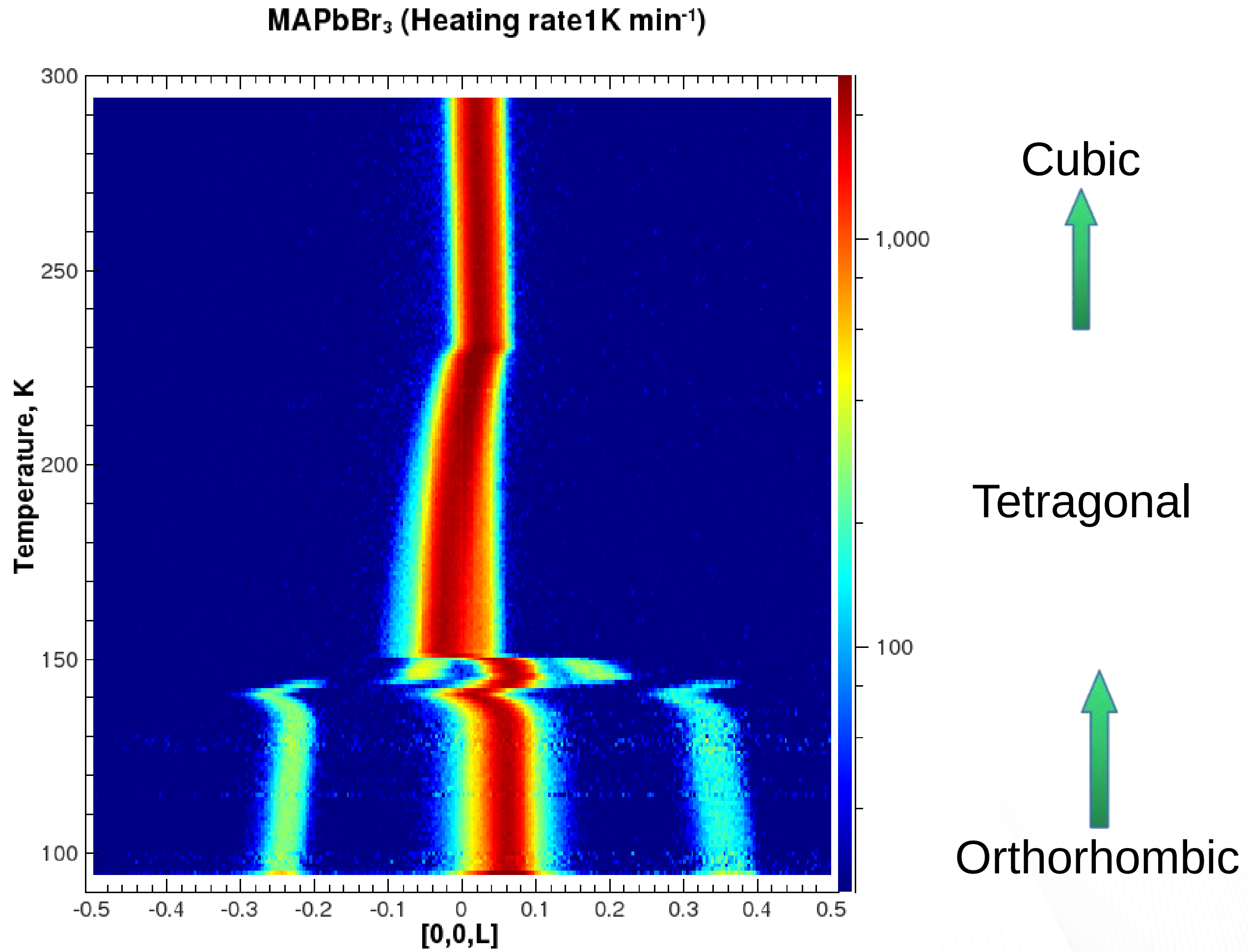
    Link to event data with a time stamp
- Data saved as event nexus file
- 3D diffraction data + external stimuli

  p_i with $i = 1 \dots n$



***Static and Pulsed E*-field**: In user program

Study phase Transitions

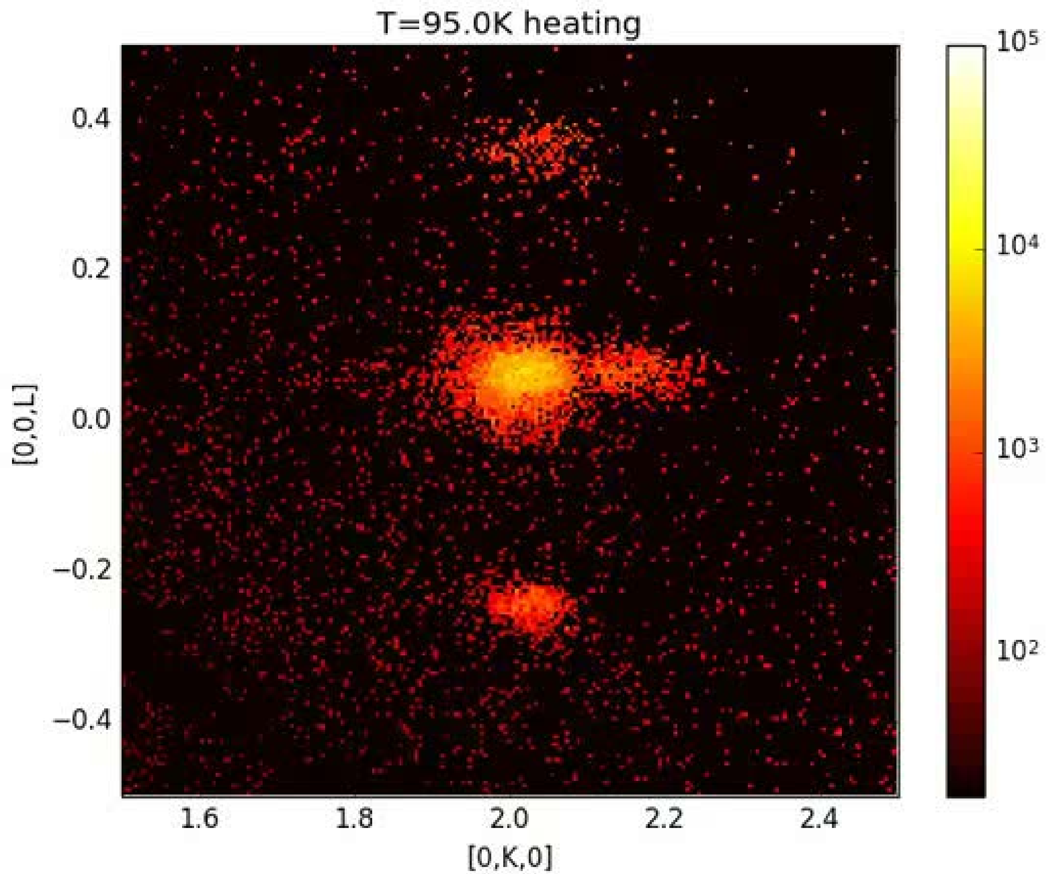


Temperature dependence of the (2 0 0)$_C$ peak

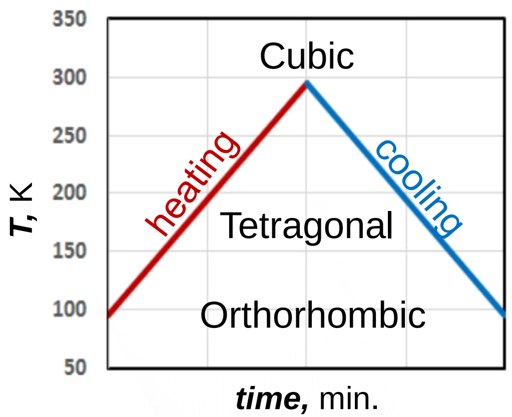
Yang et. al, *Advanced Materials*, **30,** 22, 1705801 (2018).

Visualization Real-Time Data

Evolution of the *o*-$MAPbBr_3$ $(202)_O$ peak upon continuous heating from 95 K to 295 K and cooling from 295 K to 95 K at the rate of 1 K/min.

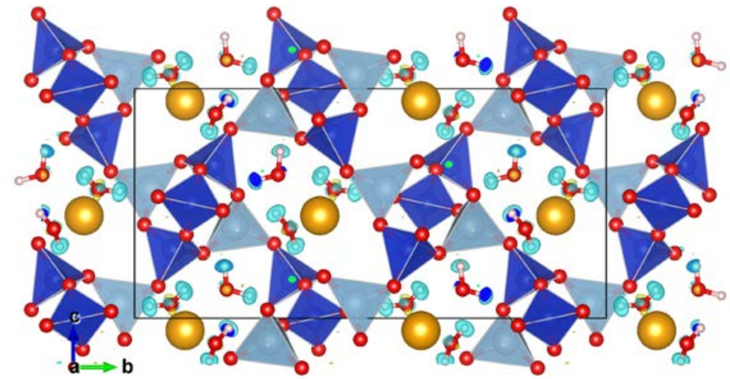


$(202)_O$ / $(\mathbf{220})_T$ / $(200)_C$



Ramping rate 1K per min.

TOPAZ Data Format



- Scolecite
  - ORNL SNS TOPAZ Time of Flight Laue data
  - SHELX HKLF 2 Format [First 7 columns]

scolecite.hkl

h	*k*	*l*	*Fo²*	*σ(Fo²)*	BN	*λ*														
-2	0	-4	481.25	22.08	1	2.50910	0.09279	-0.73919	-0.42826	0.58299	-0.58425	-0.56036	-0.79039	7450	1	0.8545	17	1.78159	1.6134	46.00	75.00
-2	0	-3	0.16	0.67	1	3.01813	0.09328	-0.73919	-0.55975	0.58299	-0.58433	-0.56036	-0.73663	7450	2	0.8375	17	1.84608	1.8924	150.00	142.00
-3	1	-5	65.50	6.91	1	1.96961	0.09433	-0.73919	-0.56637	0.58299	-0.48072	-0.56036	-0.81639	7450	4	0.8702	17	1.94420	1.1922	172.00	46.00
......																					
4	14	-3	19.82	2.98	2	1.59112	0.09256	0.93235	-0.06786	0.35276	0.82220	0.37397	-0.55718	7451	1949	0.8850	17	1.75654	1.0337	107.00	196.00
4	12	-3	124.36	8.03	2	1.78823	0.09338	0.93235	0.04000	0.35276	0.77920	0.37397	-0.57911	7451	1950	0.8774	17	1.83953	1.1240	114.00	99.00
......																					
-4	4	-12	61.52	7.25	3	0.82688	0.08914	-0.24143	-0.64405	0.79045	-0.61564	-0.61164	-0.63746	7452	4143	0.9140	27	1.49461	0.6083	80.00	150.00
-4	6	-11	129.73	8.40	3	0.92910	0.09088	-0.24143	-0.72129	0.79045	-0.49560	-0.61164	-0.69088	7452	4146	0.9090	27	1.62499	0.6399	176.00	118.00
-4	4	-11	256.97	12.37	3	0.88075	0.08918	-0.24143	-0.67051	0.79045	-0.60491	-0.61164	-0.62278	7452	4147	0.9122	27	1.49560	0.6476	99.00	168.00
......																					
......																					
-10	0	7	3.86	6.49	39	0.58899	0.08534	-0.38555	-0.42428	0.88754	-0.88891	0.11391	0.02441	7488	68768	0.9252	28	0.94947	0.6443	200.00	195.00
-10	0	8	-2.95	6.32	39	0.58432	0.08530	-0.38555	-0.39690	0.88754	-0.88897	0.11391	0.08658	7488	68769	0.9254	28	0.95400	0.6364	236.00	222.00
-3	9	3	220.71	10.99	39	2.77421	0.09689	-0.38555	-0.67205	0.88754	0.43063	0.11391	0.35170	7488	68883	0.8398	36	2.48719	1.4648	164.00	181.00
-9	25	8	52.69	6.37	39	0.96310	0.09827	-0.38555	-0.74976	0.88754	0.38316	0.11391	0.26703	7488	68947	0.9007	36	2.44225	0.5126	96.00	160.00
-9	23	8	12.15	4.19	39	0.98299	0.09778	-0.38555	-0.77370	0.88754	0.30488	0.11391	0.27425	7488	68948	0.9005	36	2.35891	0.5317	56.00	197.00
-9	25	9	302.20	15.47	39	0.94543	0.09811	-0.38555	-0.69583	0.88754	0.36055	0.11391	0.36169	7488	68954	0.9015	36	2.41150	0.5061	129.00	216.00
0	0	0	0.00	0.00	0	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0	0	0.0000	0	0.00000	0.0000	0.00	0.00

Structural analysis programs accept TOPAZ data

- ✓ **JANA2006** http://jana.fzu.cz/ [*Index modulated peaks in Q space*]
- ✓ **GSAS** https://www.ncnr.nist.gov/xtal/software/downloads.html
- ✓ **GSAS II** https://subversion.xray.aps.anl.gov/trac/pyGSAS
- o **FullProf** https://www.ill.eu/sites/fullprof/ > need more development

} **nuclear & magnetic**

- ✓ **SHELX-2014** http://shelx.uni-ac.gwdg.de/SHELX/download.php → **nuclear**
  - **GUI**
    - **ShelXle** https://www.shelxle.org/shelx/eingabe.php
    - **Olex² Crystallography Software** http://www.olexsys.org/Software

- **Workshop Talks**
- **George Sheldrick: SHELXL for neutrons (TOPAZ, Oak Ridge 2015)**
  http://shelx.uni-ac.gwdg.de/SHELX/shelxl_for_neutrons.pdf
- **Xiaoping Wang: Refinement of small molecules against neutron data (ACA, 2016)**
  http://shelx.uni-ac.gwdg.de/SHELX/neutrons_SM_ACA2016.pdf