Recent Advances of Single Crystal Neutron Diffraction Data Reduction and Analysis

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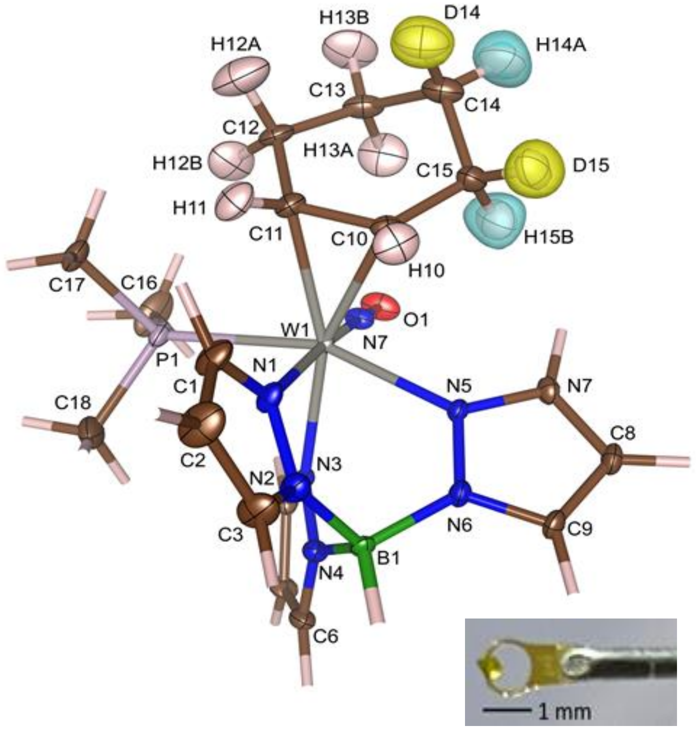
Neutron Scattering Division
Oak Ridge National Laboratory

Single Crystal Neutron Diffraction Data Reduction and Analysis Workshop

ORNL June 22, 2024

Single crystal neutron diffraction



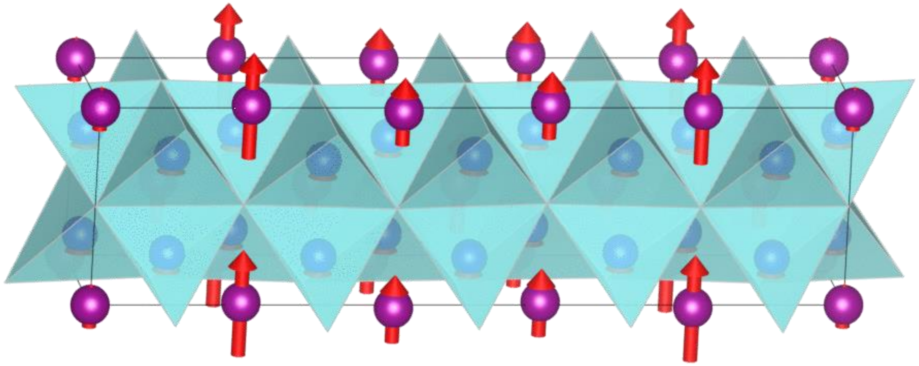
0.05 mm^3

- **Why neutrons?**

  Weak neutron ~ nuclear interaction E = 81.8 meV at 1.0 Å

  - *Scattering length b*:
    - Fourier transform of the nuclear density of an atom
    - *b is* isotope specific
    - independent of scattering angle and wavelengths
  - *Magnetic scattering*

    Neutron (*spin* ± ½) ~ (out-shell) electron interaction, strong Q dependence



J. A. Smith, *et al. Nature*, **581**, 288-293 (2020)

A. Pramanick, *et al. Phys. Rev. B*, **85**, 1444412 (2012)

1944 - The birth of single crystal neutron diffraction

LINTON LABORATORIES

Date: May 25, 1944

To: R. L. Doan

From: E. O. Wollan

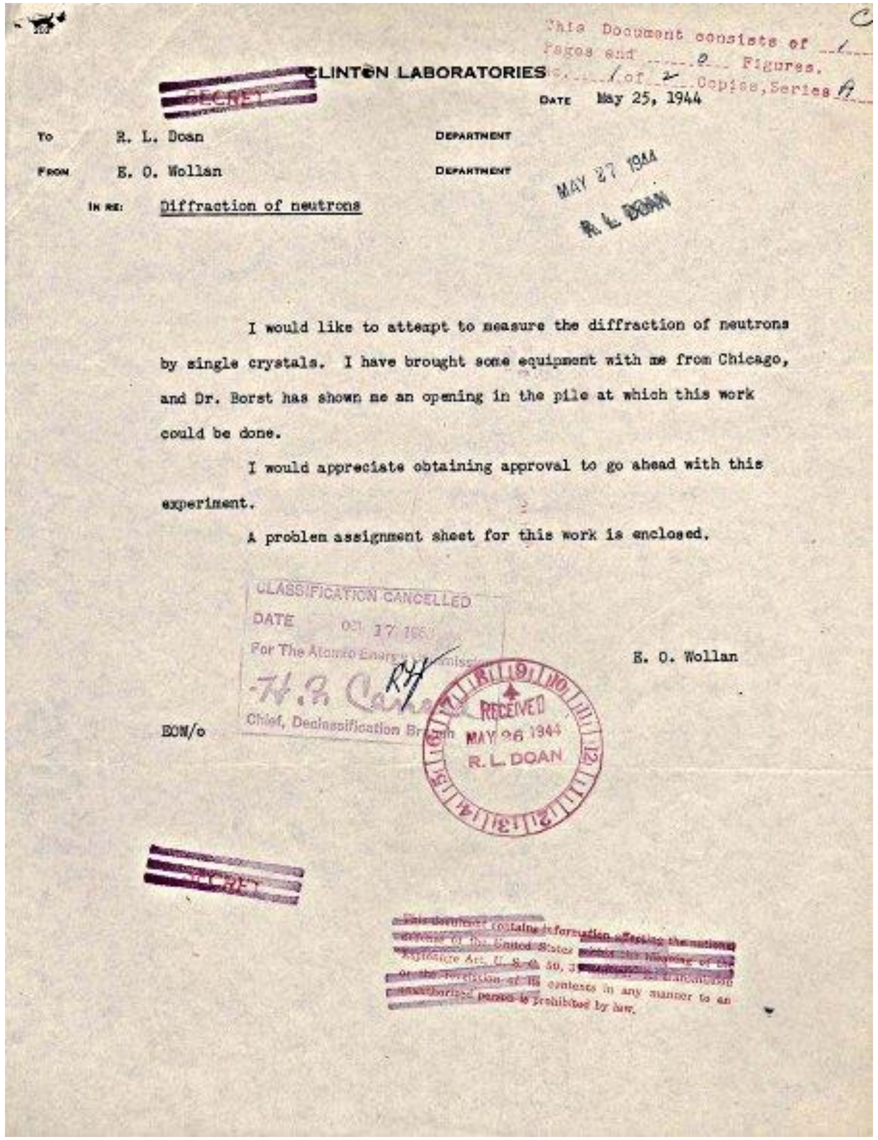
In re: Diffraction of Neutrons

I would like to attempt to measure the diffraction of neutrons by single crystals. I have brought some equipment with me from Chicago, and Dr. Borst has shown me an opening in the pile at which this work could be done.

I would appreciate obtaining approval to go ahead with this experiment.

A problem assignment sheet for this work is enclosed.

E. O. Wollan



CLINTON LABORATORIES

SECRET

DATE May 25, 1944

TO R. L. Doan DEPARTMENT

FROM E. O. Wollan DEPARTMENT

IN RE: Diffraction of neutrons

MAY 27 1944
R. L. DOAN

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E. O. Wollan

EOW/c

CLASSIFICATION CANCELLED
DATE
For The Atomic Energy Commission
Chief, Declassification Branch

RECEIVED
MAY 26 1944
R. L. DOAN

SECRET

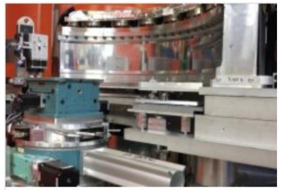
The original letter Ernest Wollan wrote in 1944 to Richard Doan, director of research at Clinton Laboratories, requesting funding for neutron experiments at the X-10 pile.

https://neutrons.ornl.gov/content/history-neutron-scattering-ornl

The suite of single crystal diffraction instruments at ORNL

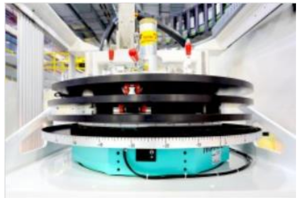
- Monochromatic source
  - Point detector – record photon/neutron counts using a point counter
    - 1D peak integration using peak profile from step scans
  - Area detector – record photon/neutron counts at 2D (x, *y*) pixel positions
    - 2D and 3D peak integration possible by combining frame images from step scans

DEMAND
Small unit-cell nuclear & magnetic structural studies



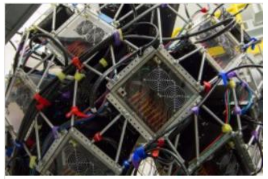
WAND²
Diffuse-scattering studies of single crystals and time-resolved phase transitions

- White beam
  - Laue
    - [Cylindrical] Image plate – 2D (x, *y*) pixels
    - Spatial and harmonic overlap of higher order reflections
    - Quasi–Laue, limit $\Delta\lambda/\lambda$, for example, to 15%
  - Wavelength-resolved Laue
    - Area detectors with large Q coverage at spallation neutron sources
    - Neutron Time of Flight (TOF) provides wavelength resolution in 3rd dimension

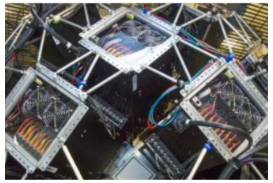


IMAGINE
Atomic resolution structures in biology, chemistry and complex materials

CORELLI
Detailed studies of disorder in crystalline materials



TOPAZ
Atomic-level structures in chemistry, biology, earth science, materials science, condensed matter physics



MANDI
Atomic level structures of proteins, macromolecules and DNA

Single Crystal Diffraction Group – Instrument Scientists


Dean Myles


Flora Meilleur


Andrey Kovalevskyi

IMAGINE / MANDI


Feng Ye


Arianna Minelli

CORELLI


Zachary Morgan

CIS-SNS


Huibo Cao

DEMAND


Yan Wu

DEMAND / WAND^2


Yiqing Hao

CIS-HFIR


Christina Hoffmann


Xiaoping Wang

TOPAZ

Postdocs in Single Crystal Diffraction Group

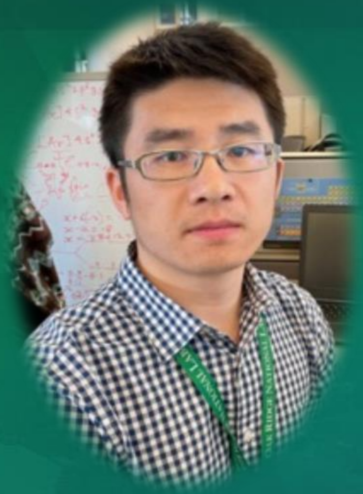
Tori Drago

Gaurav Vishwakarma

Megan Smart

Sylwia Pawledzio

James Beare

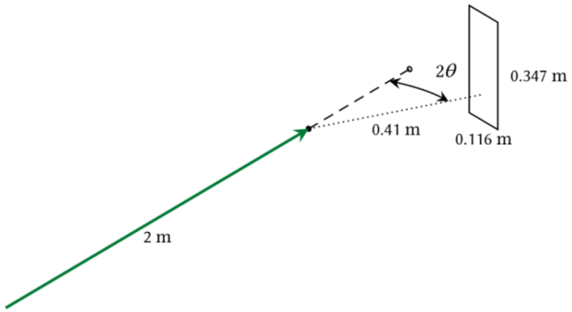


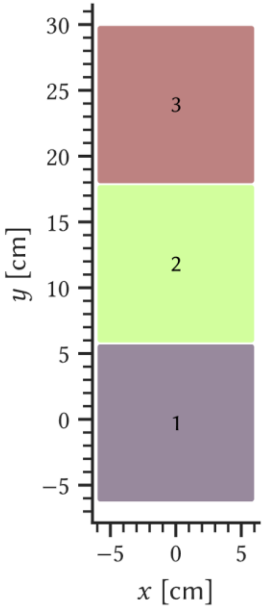
Jiasen Guo

Data acquisition at HFIR

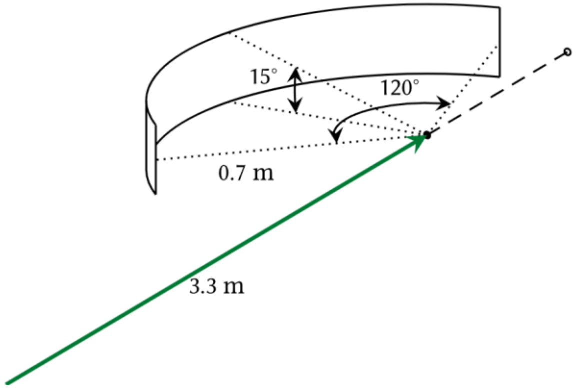
- Monochromatic source
  - Point detector – record photon/neutron counts using a point counter
    - 1D peak integration using peak profile from step scans
  - Area detector – record photon/neutron counts at 2D (*x*, *y*) pixel positions
    - 2D and 3D peak integration possible by combining frame images from step scans

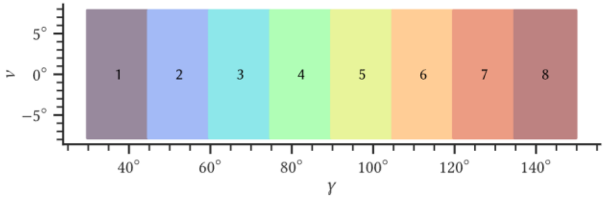
DEMAND





WAND^2





Cao H.B., Chakoumakos B.C., Andrews K.M., Wu Y., Riedel R.A., Hodges J.P., Zhou W., Gregory R., Haberl B., Molaison J.J., Lynn G.W., "DEMAND, a Dimensional Extreme Magnetic Neutron Diffractometer at the High Flux Isotope Reactor", *Crystals*, **9,** 1, 5 (2019)

Frontzek M., Andrews K.M., Jones A.B., Chakoumakos B.C., Fernandez-Baca J.A., "The Wide Angle Neutron Diffractometer squared (WAND2) - Possibilities and future", *Physica B: Condensed Matter*, **551,** 464-467 (2018)

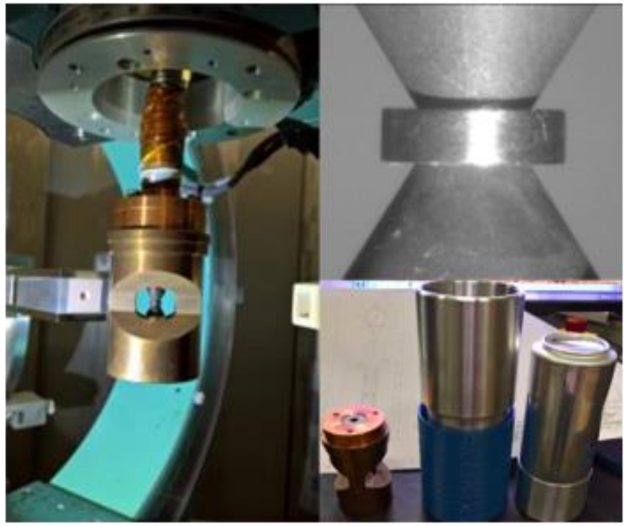
Courtesy of Zach Morgan

DEMAND HB-3A HFIR

Dimensional Extreme Magnetic Neutron Diffractometer

Four-circle mode

Two-axis mode



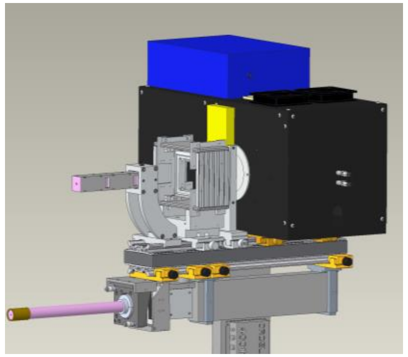
Extreme sample environment

Temperature: **0.03-1300 K**
Magnetic Field: **0-6 T**
Pressure: **0-10 GPa**
Electric field: **0-10000 v/cm**

Chakoumakos B.C., Cao H.B., et al., "Four-circle single-crystal neutron diffractometer at the High Flux Isotope Reactor", *Journal of Applied Crystallography, 44, 655-658 (2011).*

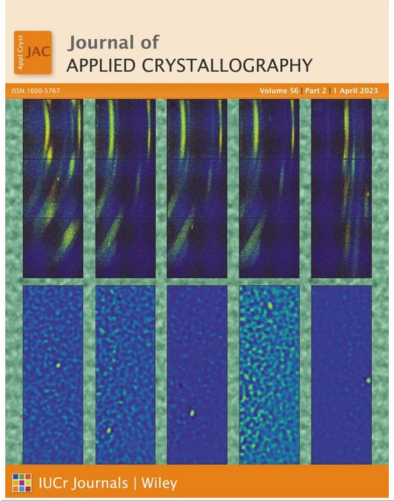
Cao H.B. et al., "DEMAND, a Dimensional Extreme Magnetic Neutron Diffractometer at the High Flux Isotope Reactor", *Crystals*, **9,** 5 (2019).

Hao Y., et al., Cao H.B., "Machine-learning-assisted automation of single-crystal neutron diffraction", Journal of Applied Crystallography, 56, 519-525 (2023).



Polarized Neutron diffraction
Unpolarized Neutron diffraction

Auto-data-reduction
Background filtering

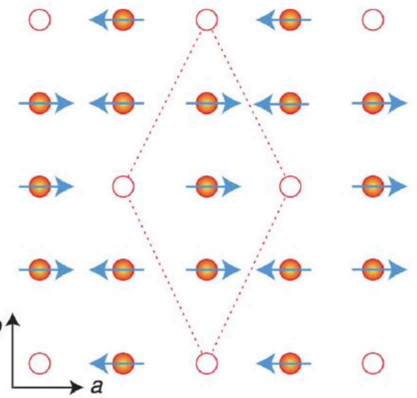


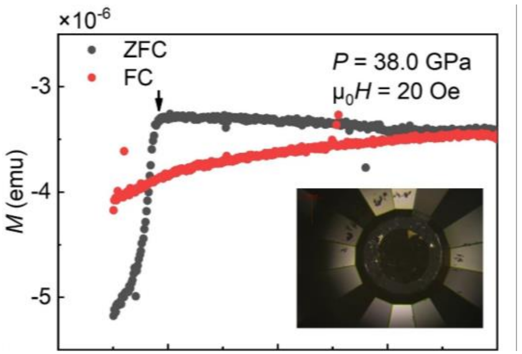
Courtesy of Huibo Cao

Science @ DEMAND

Courtesy of Huibo Cao

Superconductivity





Magentism

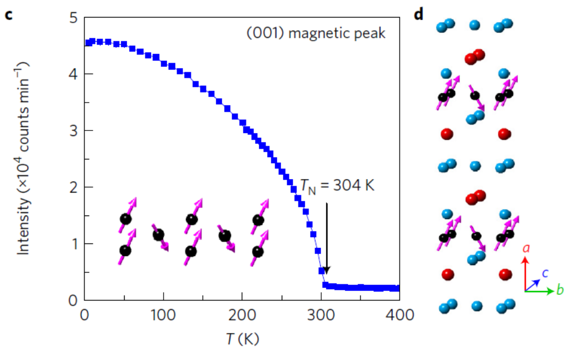
Zhao et al. PRL, 2012

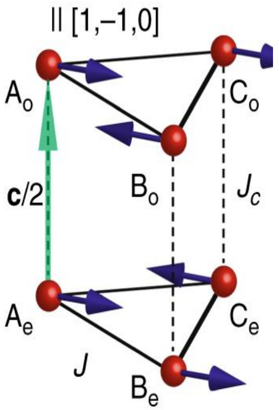
Structural symmetry

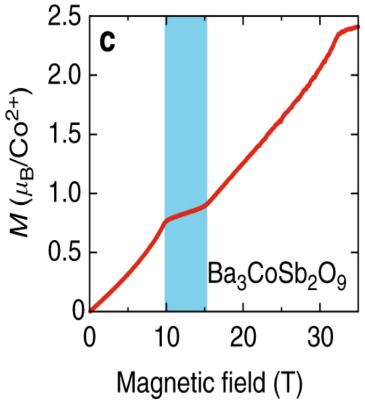
Zhu et al., Nature (in press 2024)

Superconductivity in $La_4Ni_3O_{10}$

Topological & Quantum magnets







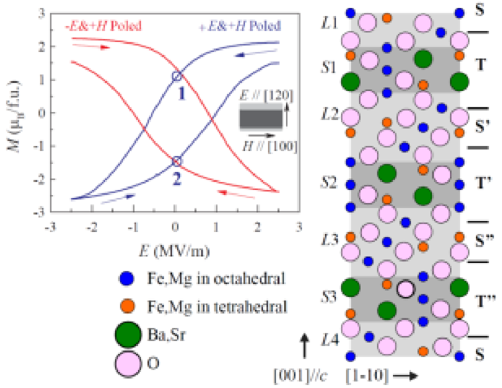
Nature Materials 16, 905 (2017)

Magnetic Weyl semi-metal

Nature Communications 9, 2666 (2018)

Magnetization plateau

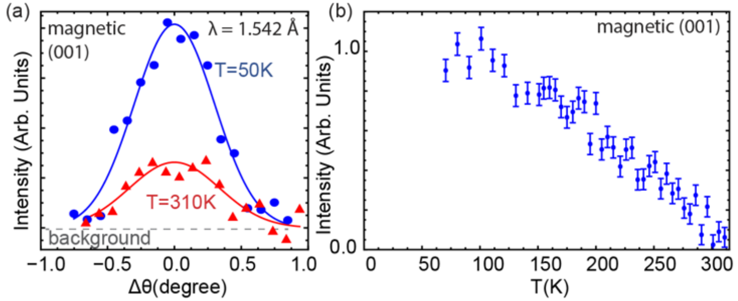
Multifunctional materials



Nature Communications 8, 519 (2017)

Giant magnetoelectric effects

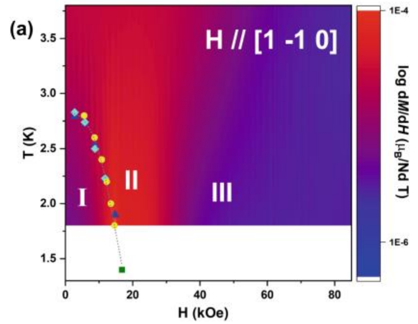
Novel magnetism in thin film

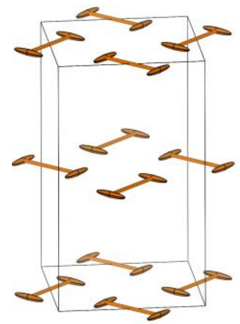


M. Chilcote et al., Adv. Funct. Mater, (in press 2024)

Altermagnetic candidate MnTe for spintronics

Magnetic anisotropy with PND



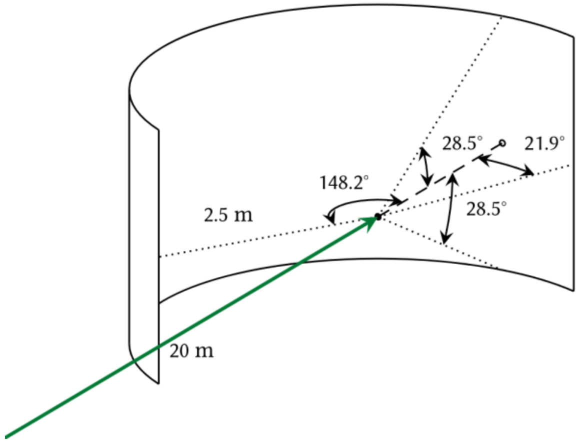


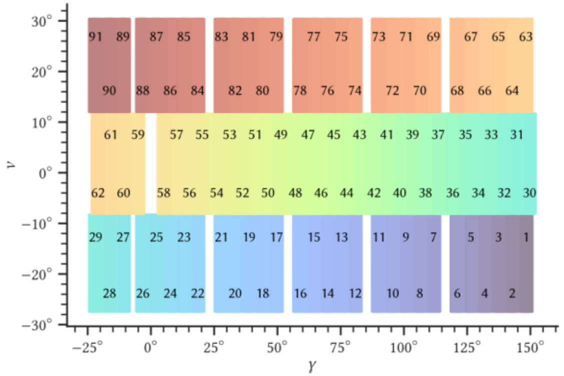
M.Marshall, et al. Nature Communications 14 (1), 3641 (2023)

Data acquisition at the SNS

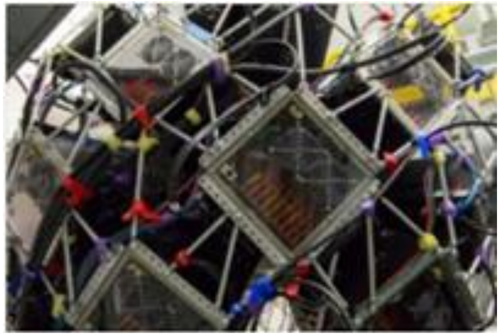
- Neutron Time-of-Flight, aka Wavelength-resolved, Laue

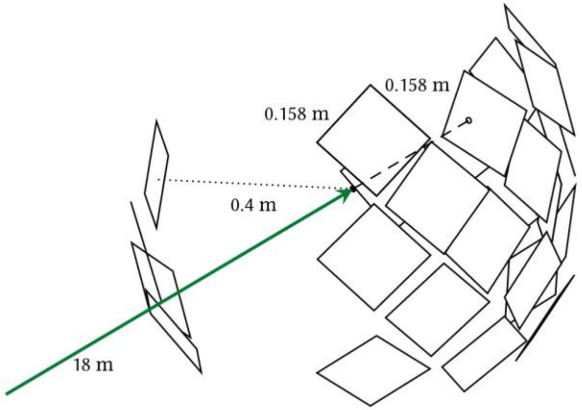
CORELLI

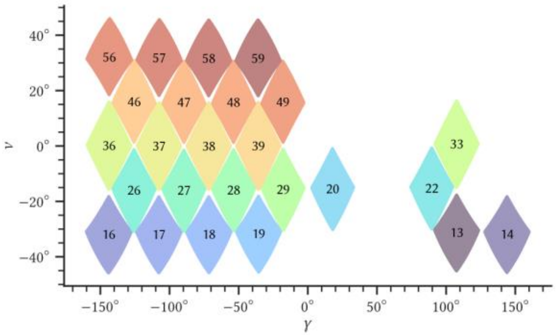




TOPAZ







- Neutron Time of Flight (TOF) provides wavelength resolution in 3rd dimension

Ye F., Liu Y., Whitfield R.E., Osborn R., Rosenkranz S., "Implementation of cross correlation for energy discrimination on the time-of-flight spectrometer CORELLI", *Journal of Applied Crystallography*, **51,** 2, 315-322 (2018)

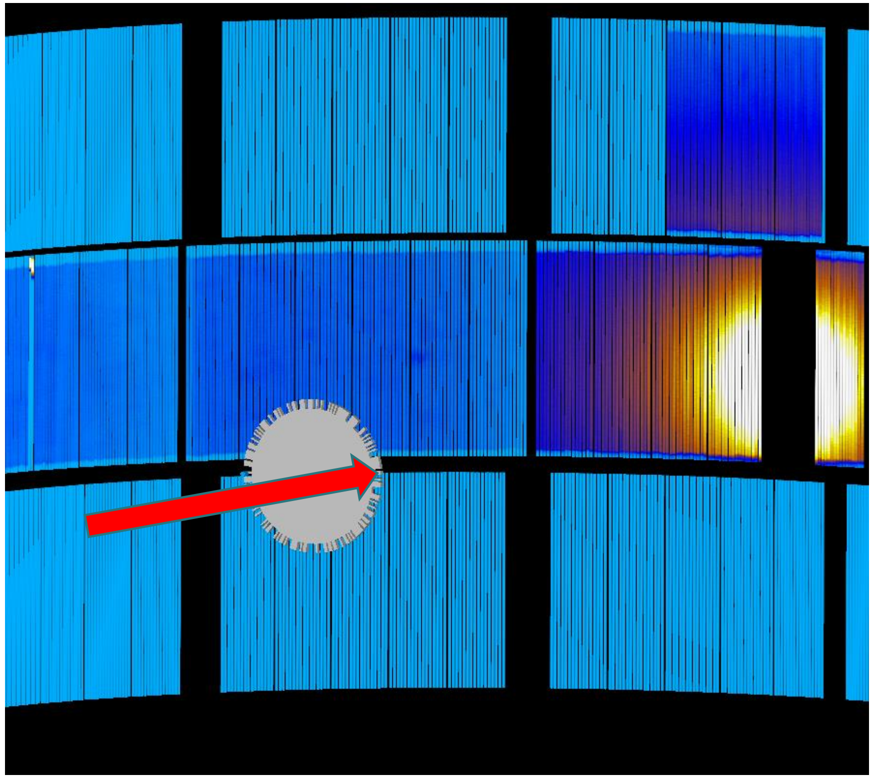
Schultz A.J., Joergensen M.R., Wang X.P., Mikkelson R.L., Mikkelson D.J., Lynch V.E., Peterson P.F., "Integration of neutron time-of-flight single-crystal Bragg peaks in reciprocal space", *Journal of Applied Crystallography*, **47,** 915-921 (2014).

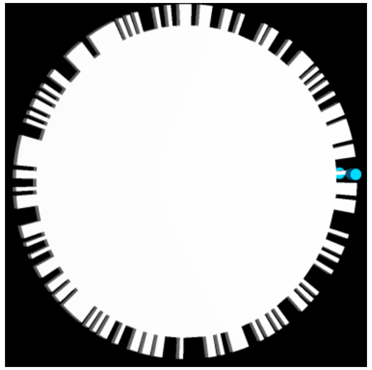
Courtesy of Zach Morgan

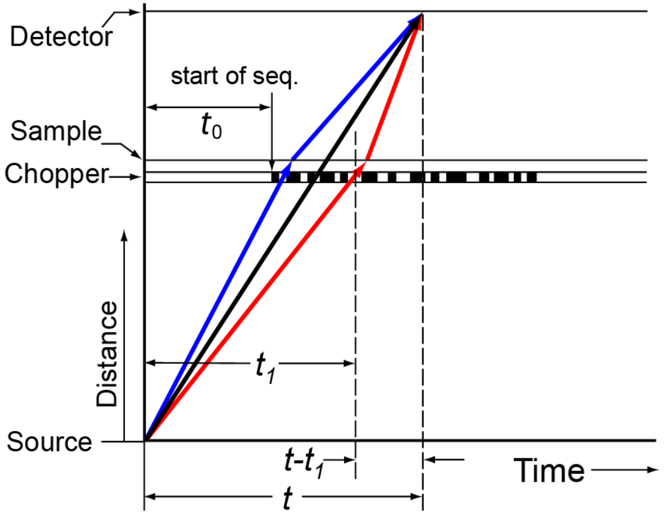
CORELLI BL-9 SNS

Key Component

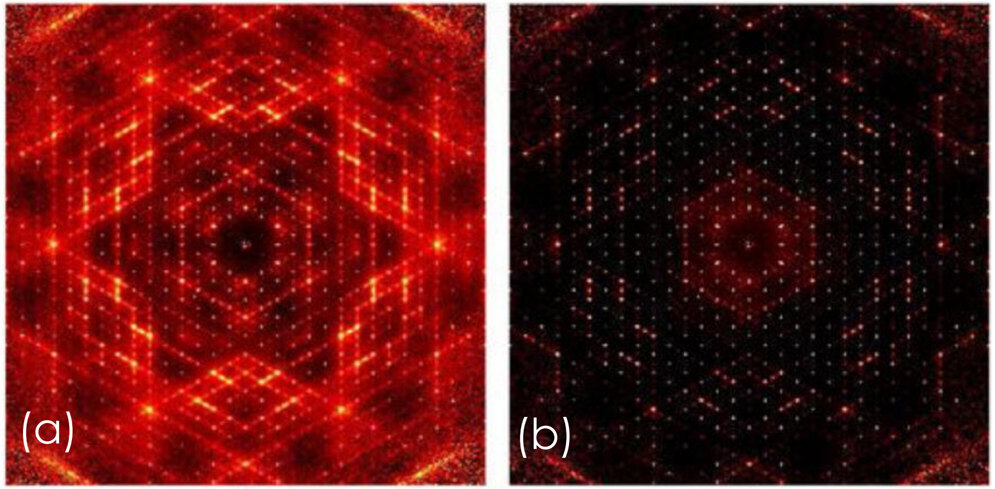
Correlation Chopper







The combination of correlation chopper and white beam Laue diffraction allows reconstruction of the elastic signal.



Diffuse scattering of benzil. (a) Total scattering (elastic plus inelastic) recorded at 100 K; (b) Elastic scattering only at 100 K

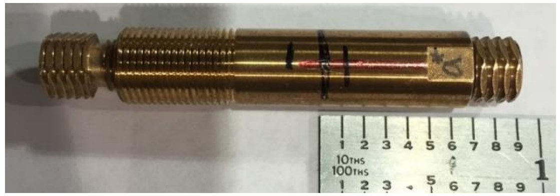
Quantum Beam Science, **2**, 2 (2018); *J. Appl. Cryst.* **51**, 315 (2018)

CORELLI BL-9 SNS Sample Environment

Low background top loading CCR
6 K – 750 K

MICAS Furnace
RT - 1400 °C

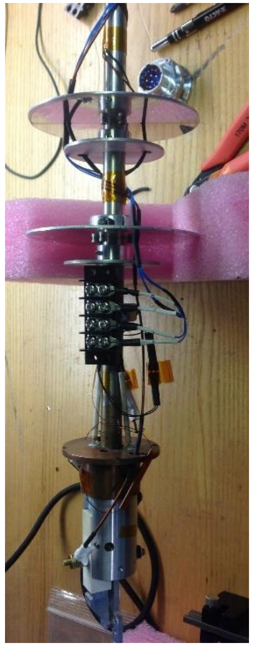
Courtesy of Feng Ye



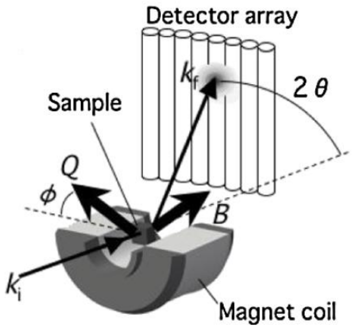
Pressure Cell, P_{max} = 1.8 GPa

^{3}He insert
300 mK – 300K
or
Dilfridge insert
100 mK – 300K

Static Vertical Magnetic Field,
B_{max} = 5 T



High Voltage

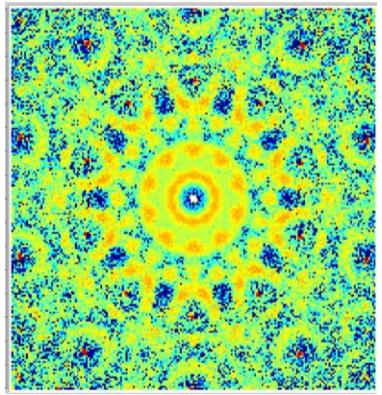


Pulsed Magnetic Field
B_{peak} = 30 T

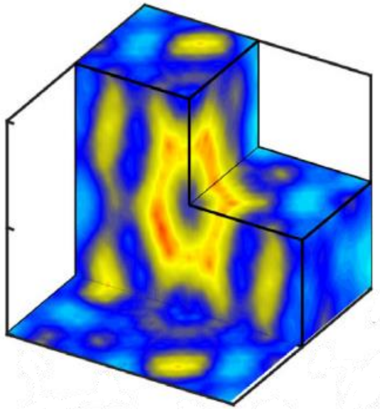
DAC, P_{max} ~ 8 GPa

Collection of diffuse scattering studies @ CORELLI

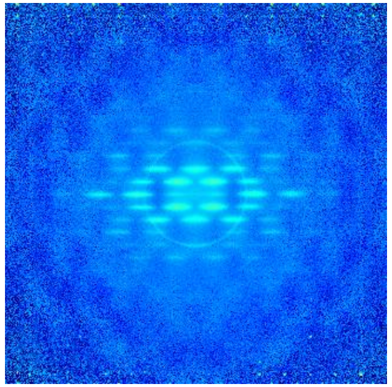
Courtesy of Feng Ye



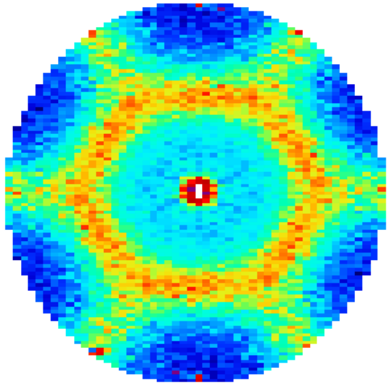
Quasi-crystal
i-Tb-Cd
(PRB 2023)



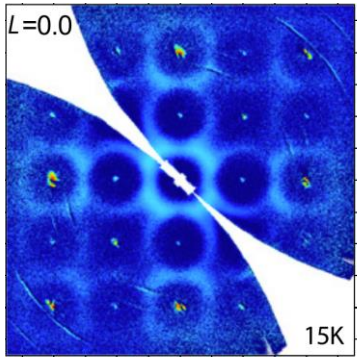
Spin ice $Dy_2Ti_2O_7$
(Nat. comm. 2020)



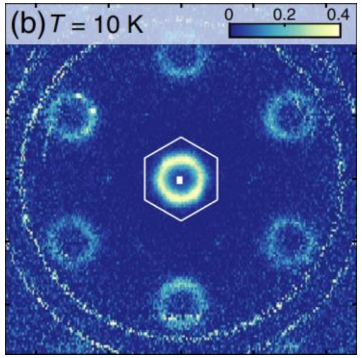
Spin Glass Fe_2TiO_5
(PRB 2021)



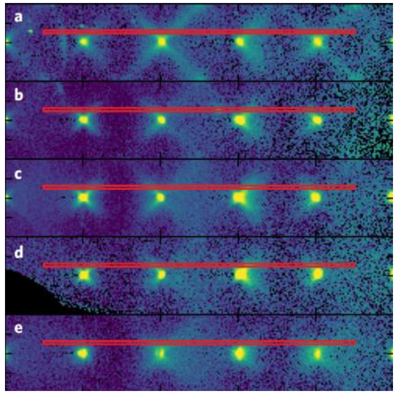
"Quantum spin Liquid"
$YbMgGaO_4$ (PRR 2021)



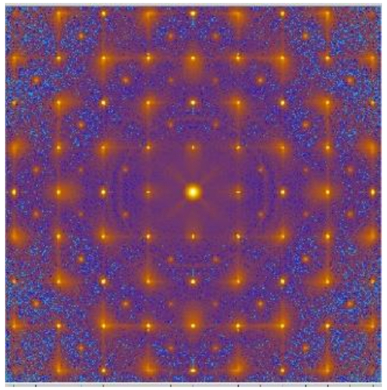
Spinel $ZnFe_2O_4$
(PNAS 2022)



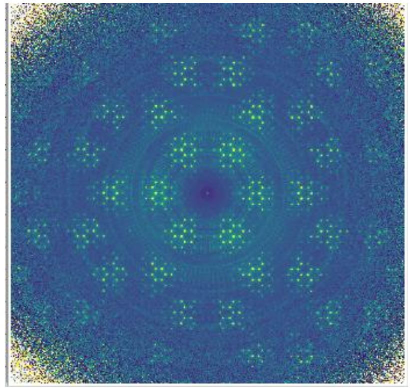
Spiral Spin Liquid
$FeCl_3$ (PRL 2022)



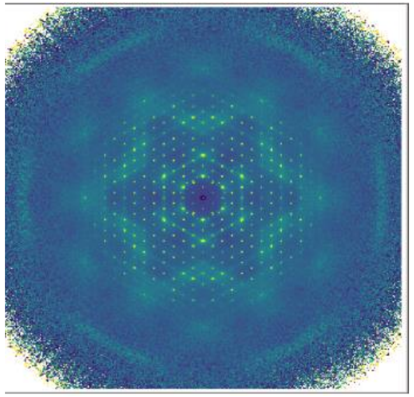
Relaxor ferroelectrics
PMN-*x*PT
(Nat. Mat. 2018)



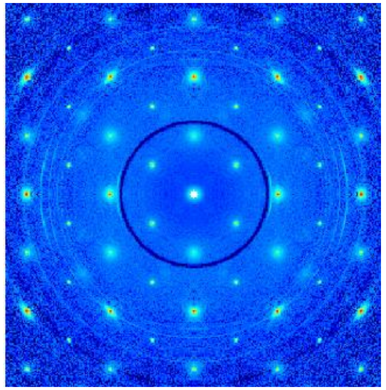
Relaxor ferroelectrics
$Bi_{0.5}Na_{0.5}TiO_3$



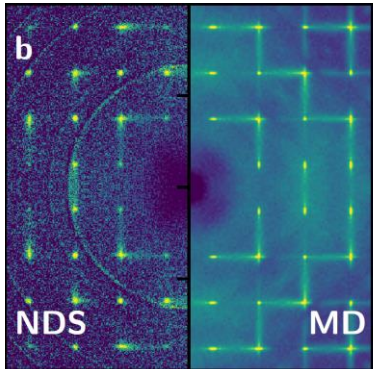
Channeled carbonate
$Ba_3Co_2O_6(CO_3)_{0.7}$
(J. of Appl. Crys. 2021)



Molecule Benzil
($C_{14}D_{10}O_2$)

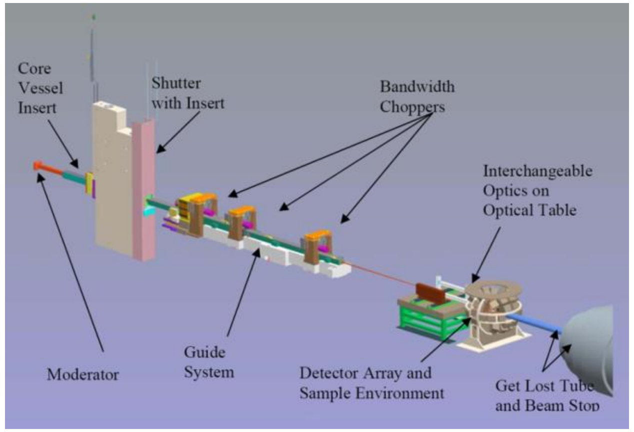


superelasticity
NiCoFeGa
(Nat. Mat. 2020)



Hybrid lead halide
$MAPbI_3$
(Joule 2023)

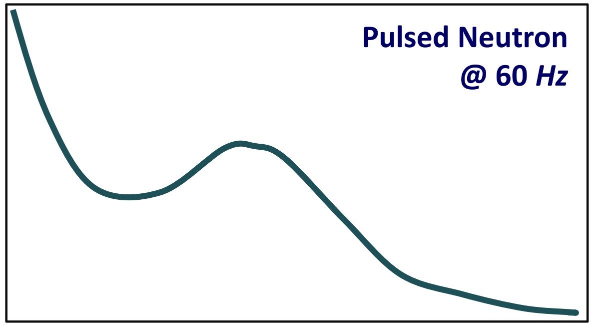
Neutron Time of Flight Laue diffraction



- **Neutron Time of Flight**: Event-based neutron detection technique
  - de Broglie equation relates neutron wavelength to its momentum:

$$\lambda = \frac{h}{mv} = \frac{h}{m}\frac{t}{L} = \frac{h}{m}\frac{t}{(L_1 + l_2)}$$

  - By recording the time of a neutron arrives over a fix path length from source to detector (aka **time of flight**), its velocity, and consequently its wavelength can be measured.



tof 0.5 msec. 16.67 msec.

λ 0.2 Å 3.66 Å

Anger Camera

<1 μ sec readout time

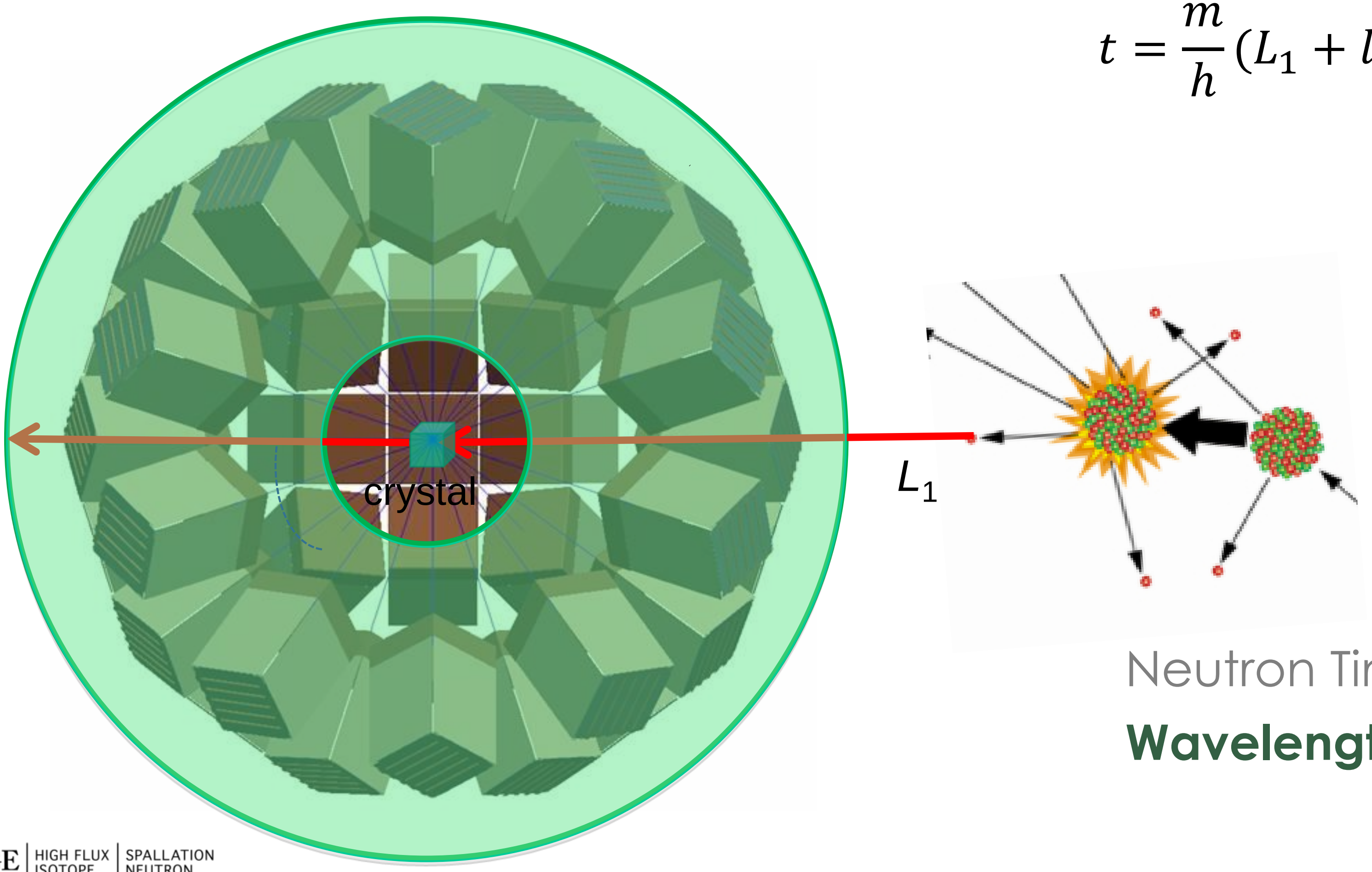
Neutron Wavelength-resolved Laue

Combine de Broglie's equation and Bragg's law

$$\lambda = \frac{h}{mv} = \frac{ht}{m(L_1 + l_2)} \qquad \lambda = 2d\sin\theta$$

$$t = \frac{m}{h}(L_1 + l_2) \times 2d\sin\theta$$

L_1 – 18 m
l_2 – 0.39 to 0.46 m



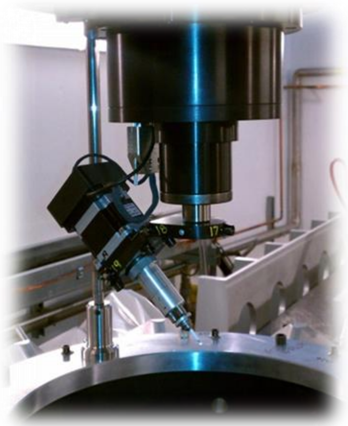
Pulsed Neutron
60 *Hz*

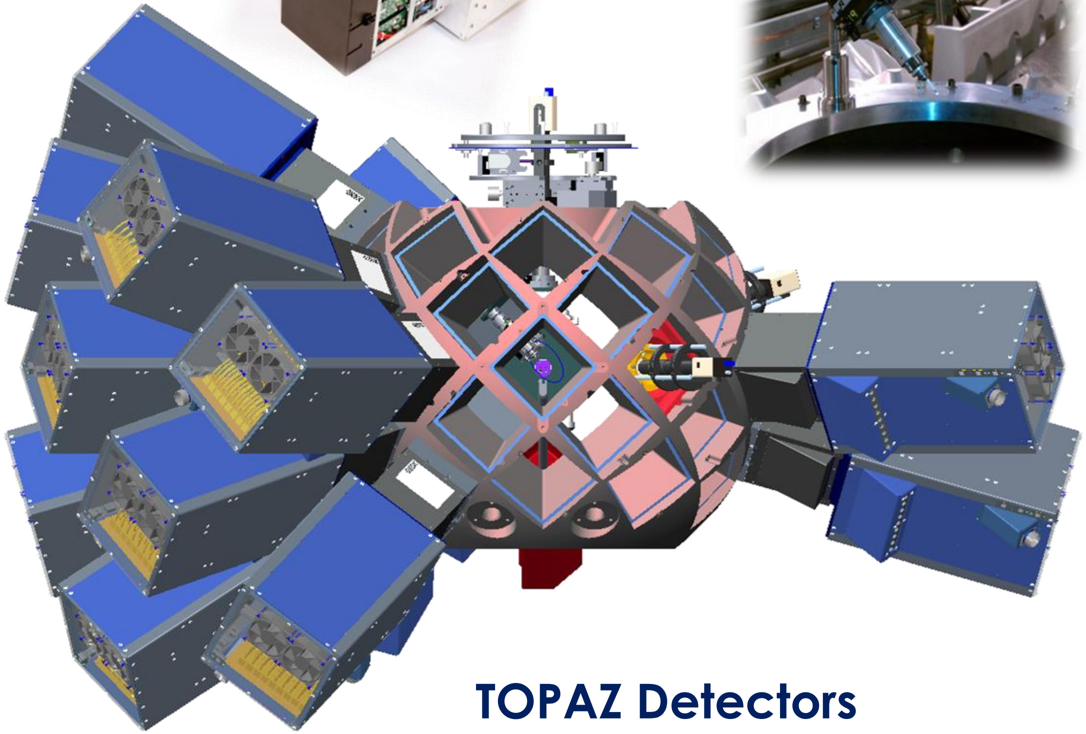
Neutron Time-of-flight Laue

Wavelength-resolved Laue

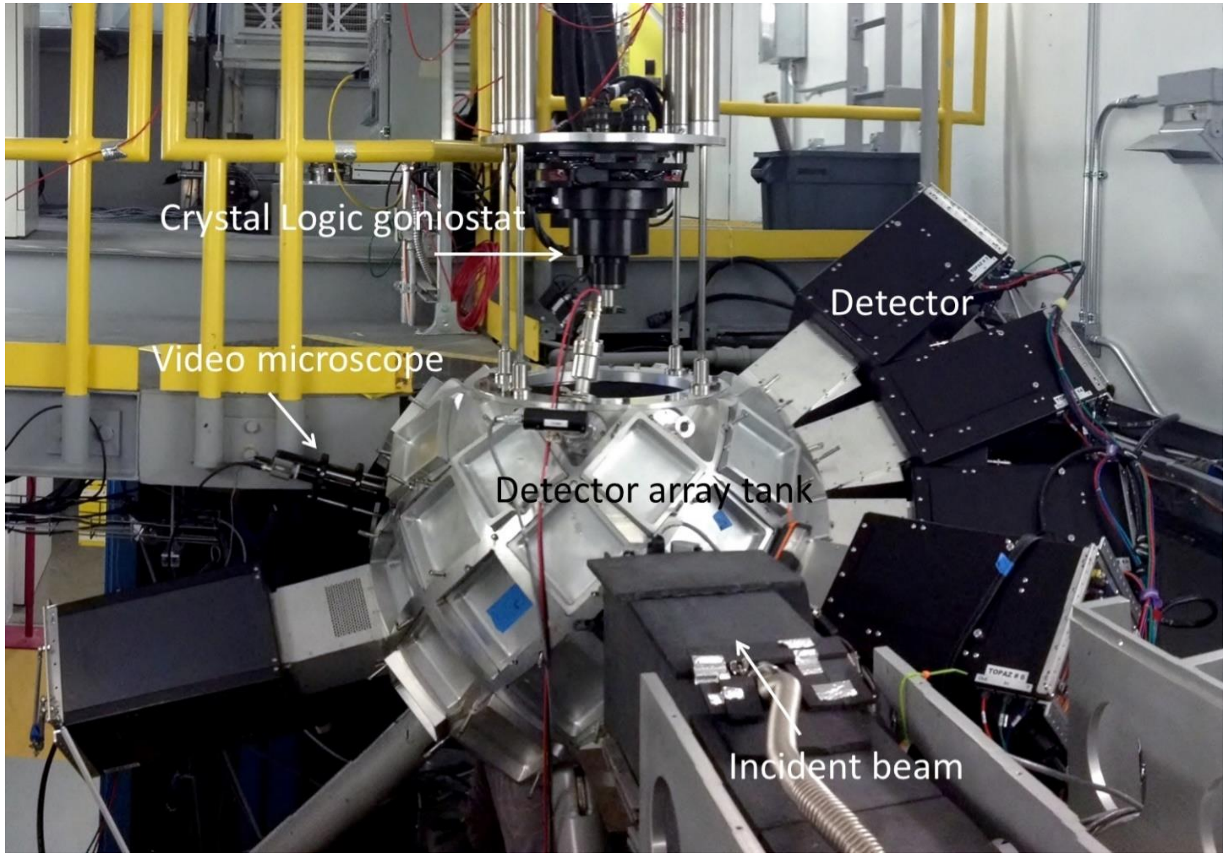
TOPAZ Single Crystal Diffractometer BL-12 SNS

Anger Camera





TOPAZ Detectors



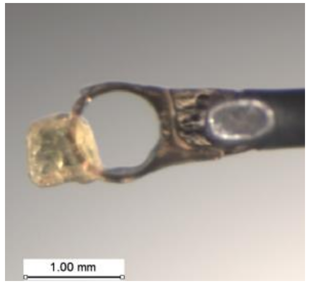
J. Appl. Cryst. **47**, 915 (2014)

https://neutrons.ornl.gov/topaz

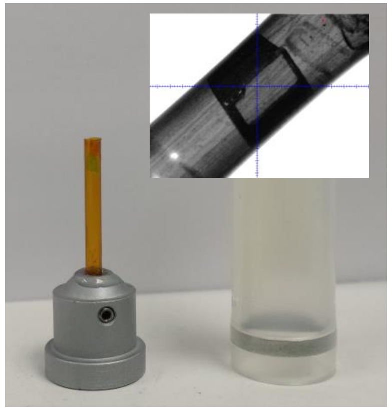
TOPAZ Ambient Goniometer

- A **two-axis** goniometer
  - Omega, phi with chi fixed at 135°.
  - Both omega and phi are fitted with sliprings that allow unlimited 360° rotational motion.
  - The omega and phi rotation axes are separated by 45 degrees.
  - Sample mount

MiTeGen loop **(1 mm ϕ)**



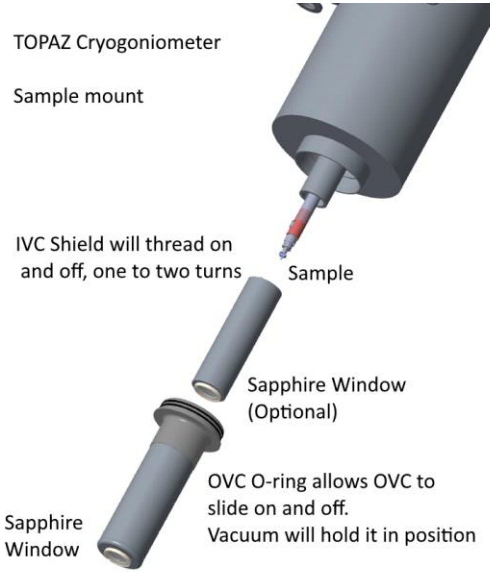
Glued or Coated with perfluorinated grease

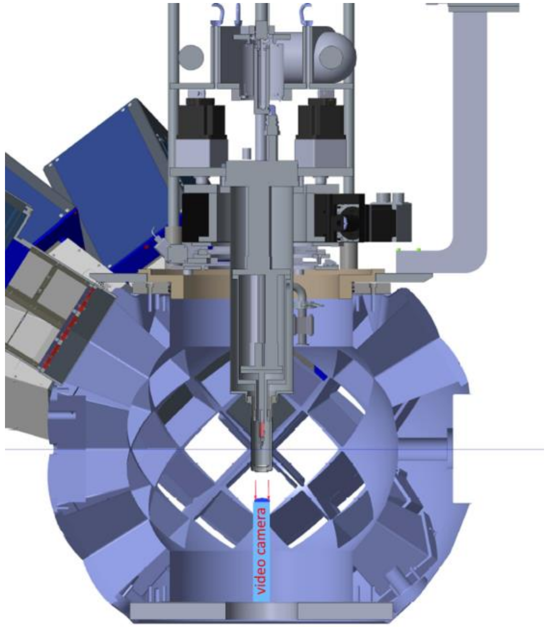


Glued onto the tip or inside a Kapton tube

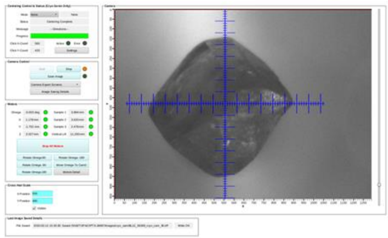
TOPAZ Cryogenic Goniometer

- One axis of rotation (360°) with precision motor controls to center, orient, and hold the sample in the temperature range 5 K – 300 K.
- A video camera is mounted on the base of the DAT

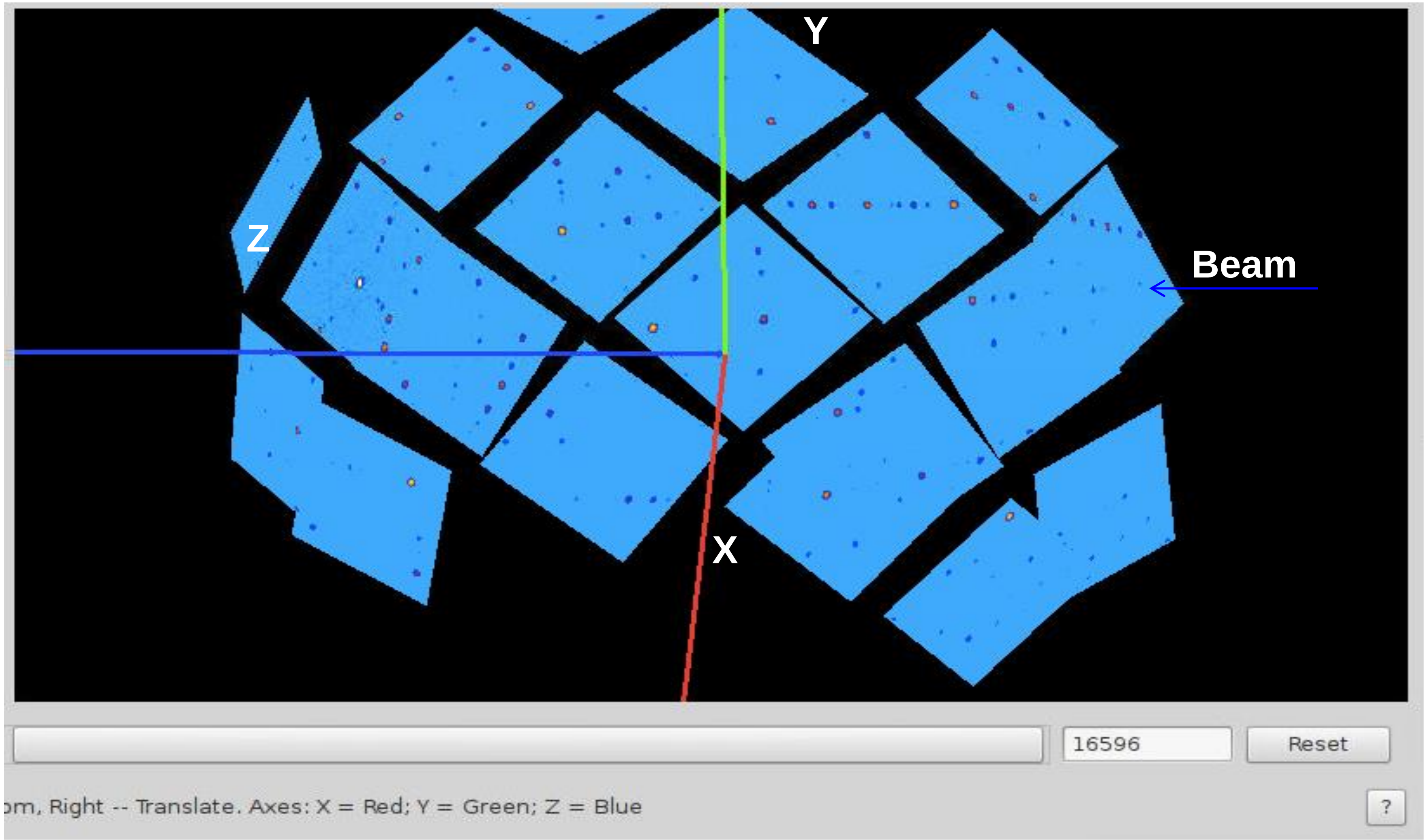




- Click-to-center of a single crystal sample



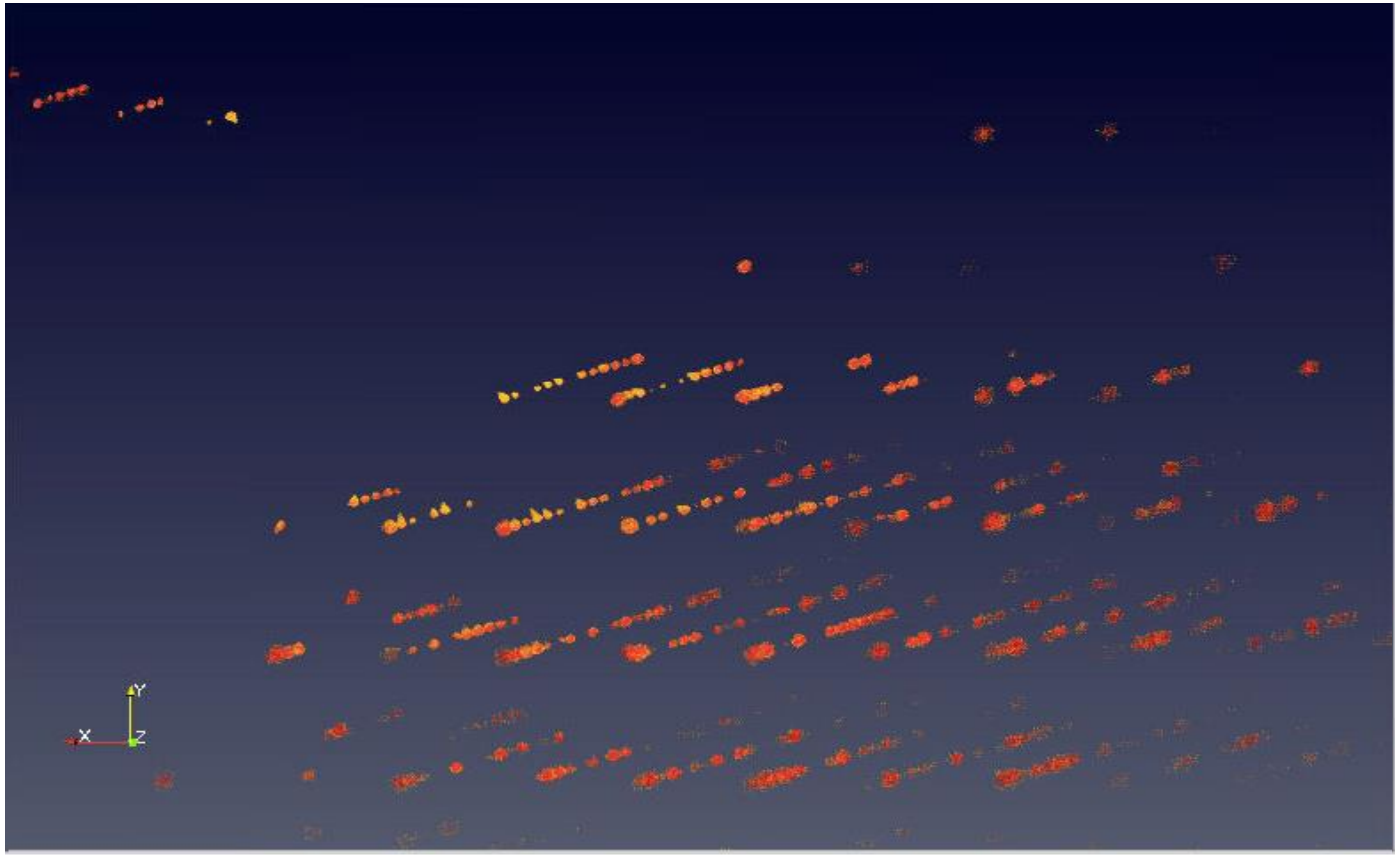
Single crystal peaks on 2D detector space



Single crystal peaks in 3D Q space

Sample continuous reciprocal Q space

Cover a large number of reflections stimulated at a ***stationary*** crystal



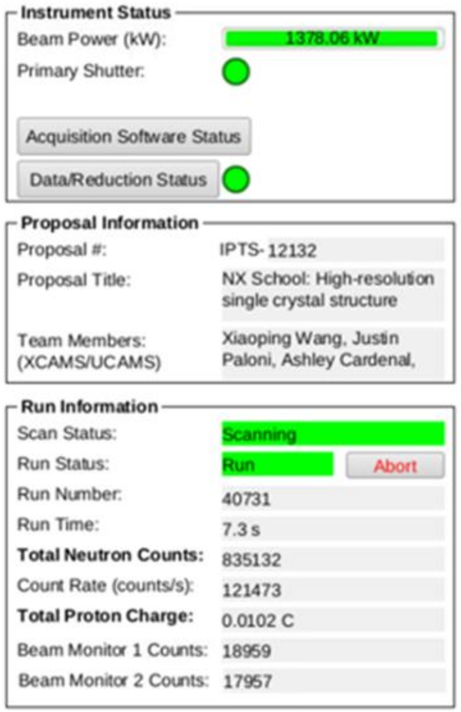
$$\mathbf{k_f} - \mathbf{k_i} = \mathbf{G}$$

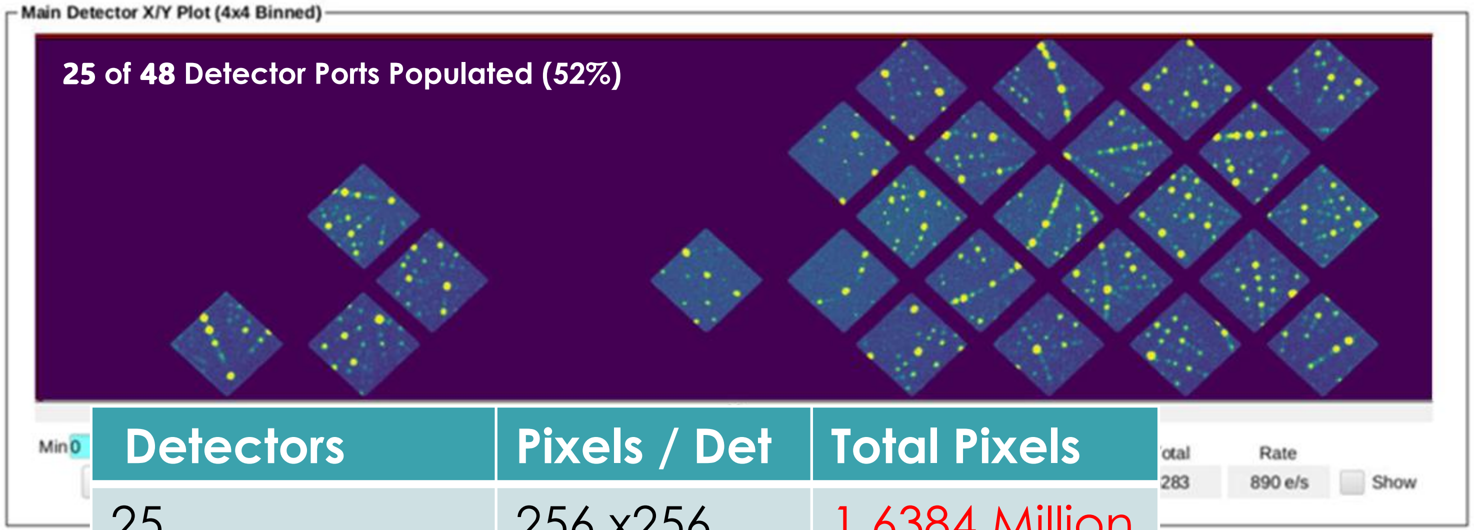
Laue condition

$$\frac{\pi}{4}\,\frac{(\lambda_{max} - \lambda_{min})d_{max}^{*4}}{V^*}$$

Number of reflections

TOPAZ Data Collection





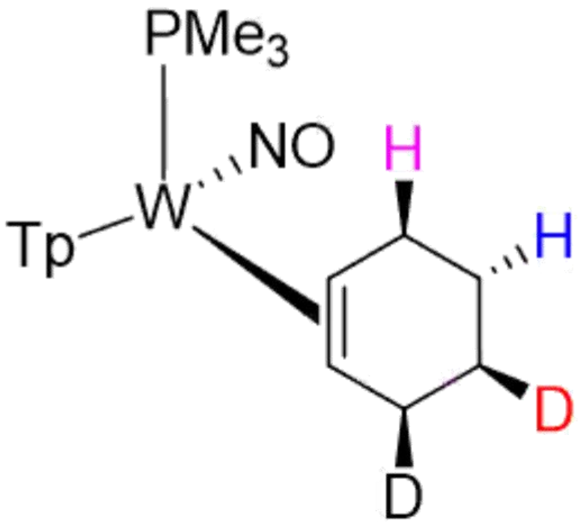
Detectors	Pixels / Det	Total Pixels
25	256 x256	1.6384 Million
Meas. Time	Pulse Rate	File Size
One Pulse	@ 60 Hz	27.3 GB
1 Sec.	@ 60 Hz	1.60 TB
1 Sec. meas.	121473 n	0.97 MB
1 hour meas.	4.373E+8 n	3.5 GB

https://neutrons.ornl.gov/topaz

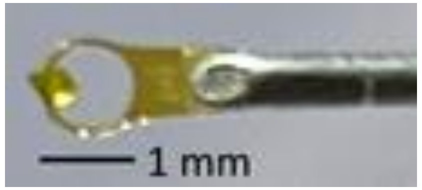
Site Specific Deuteration of a Cyclohexene Complex

Transition metal-mediated dearomatization.

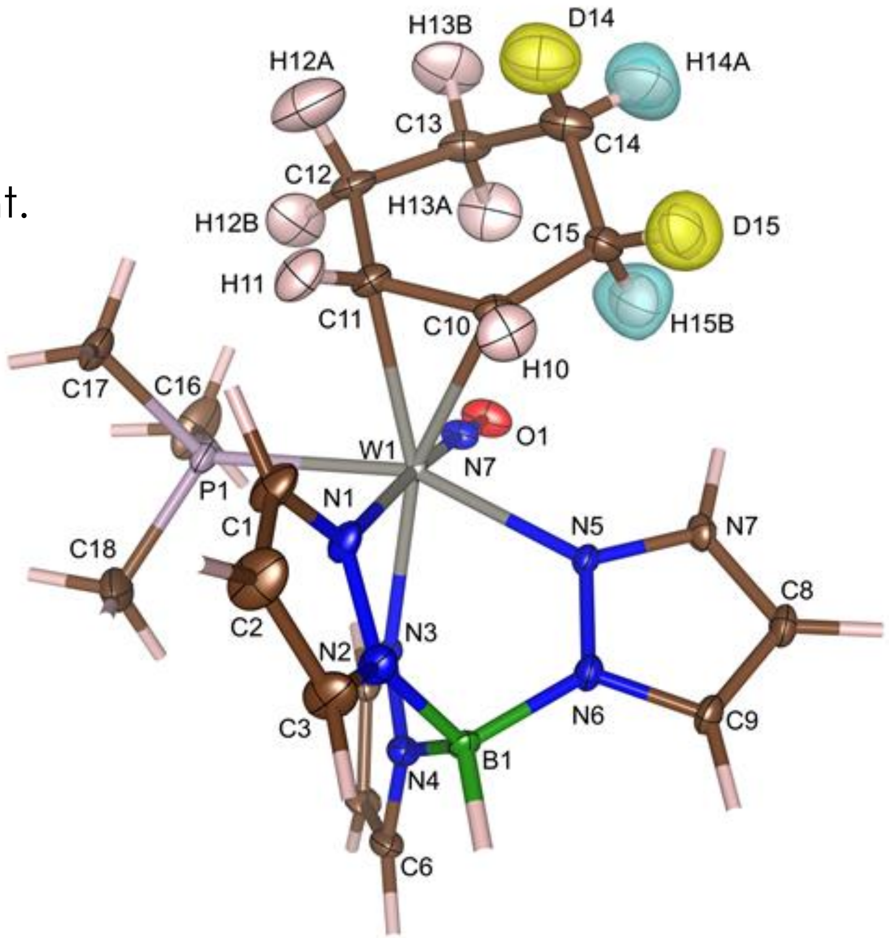
Opened pathways for a new generation of medicines and therapies that incorporate deuterium into the active pharmaceutical ingredient.



Isotope	Scattering lengths
Hydrogen **¹H**	**-3.74** fm
Deuterium **D** (^{2}H)	**6.67** fm



0.05 mm^3



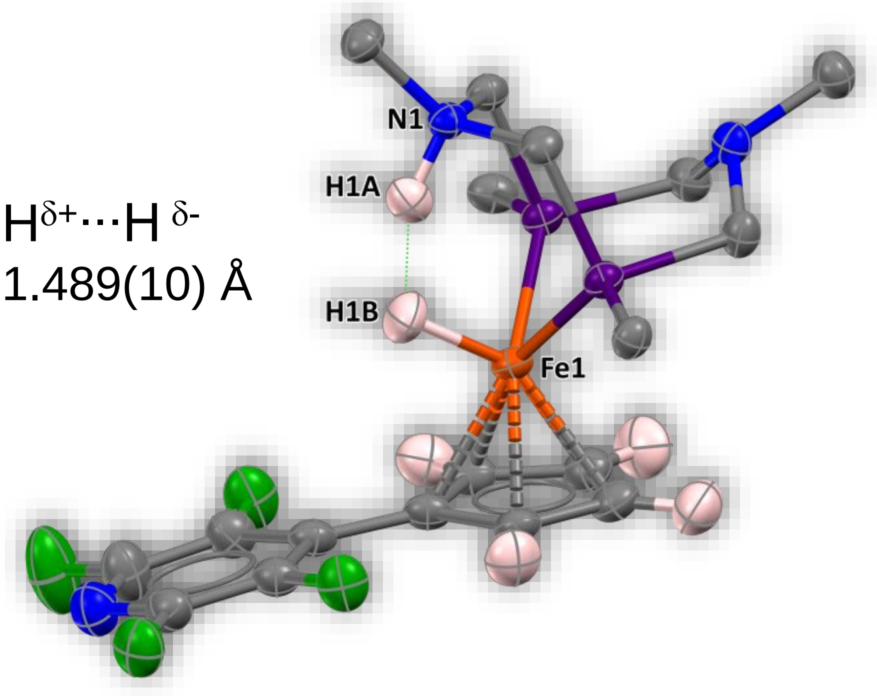
Neutron structure of a d_2 isotopologues of cyclohexene complex.

J. A. Smith, *et al*. *Nature*, **581,** 288-293 (2020)

Chemical crystallography

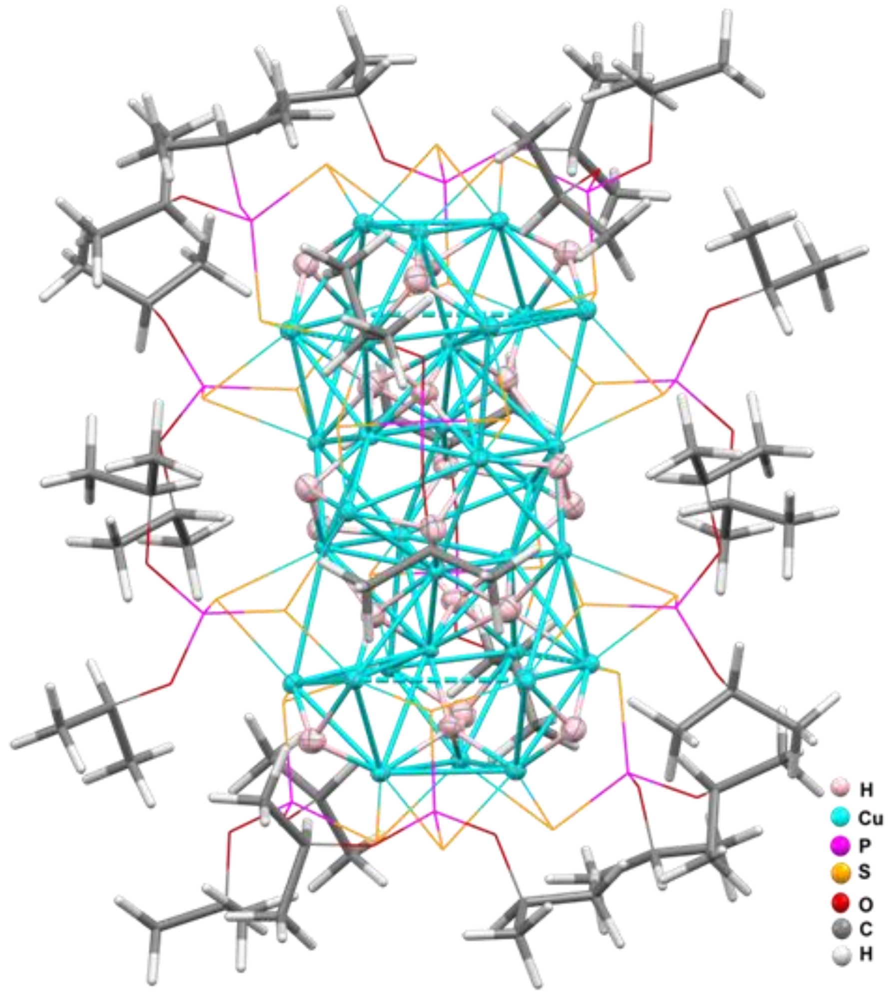
- Locate Hydrogen Atoms in a Crystal Structure

$H^{\delta+}\cdots H^{\delta-}$
1.489(10) Å



0.32 x 0.90 x 1.95 mm^3

T. Liu *et. al. Angew. Chem. International Edition,* **53,** 21, 5300-5304 (2014)



0.25 x 1.10 x 1.62 mm^3

R. S. Dhayal, *et. al. Chemistry - A European Journal,* **21**, 8369 (2015)

Single Crystal Neutron Diffraction beyond three dimensions

Diffraction space ↔ **4D Temporal Data** ↔ **Parameter space**

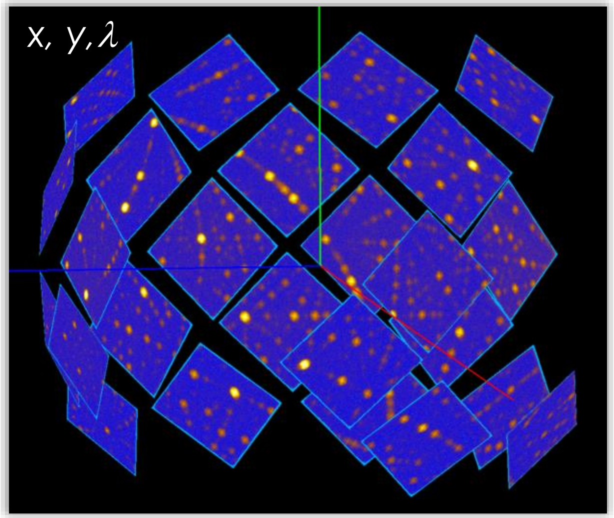
Neutron event data

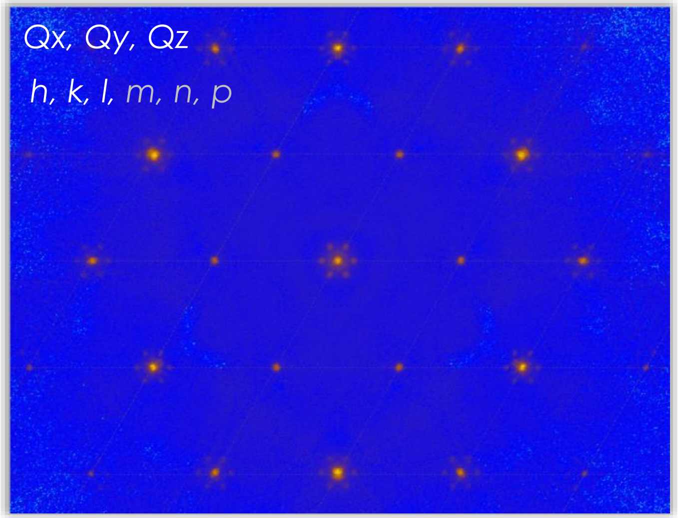
Link event data to metadata with an associated

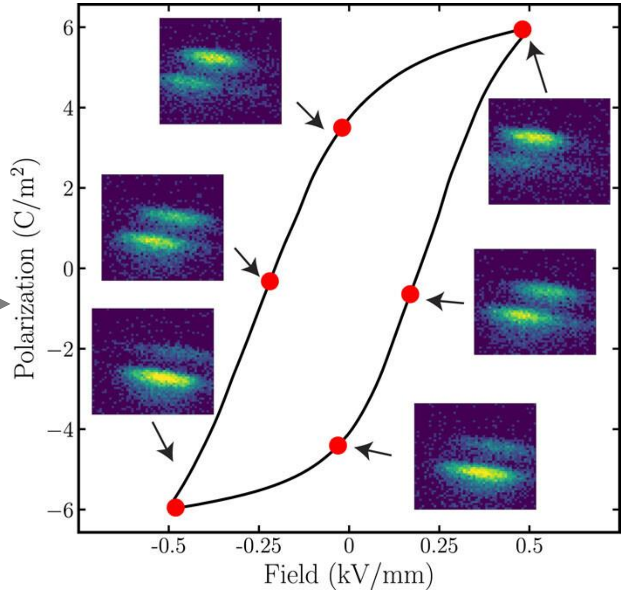
Time-stamp

p_i $i = T, E, P, \ldots$

(3+*d*) $d = 0, 1, 2, 3$

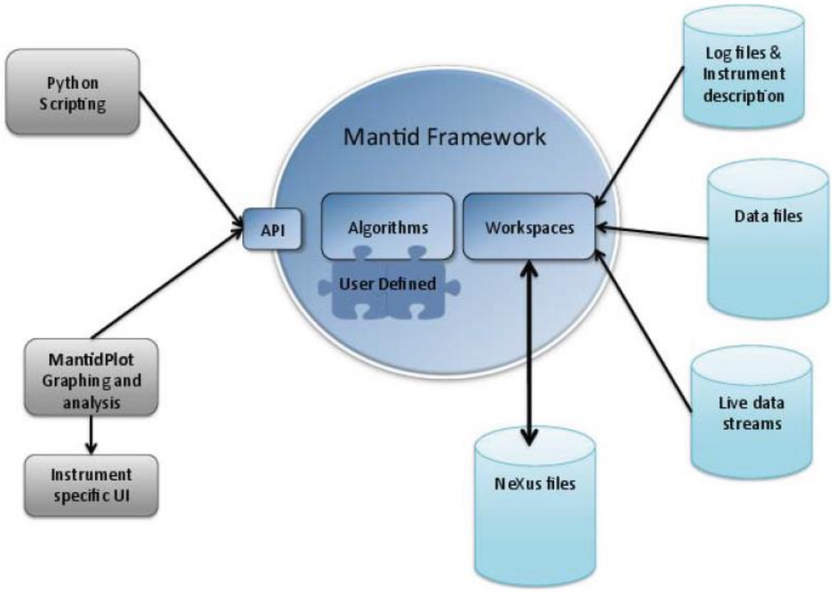


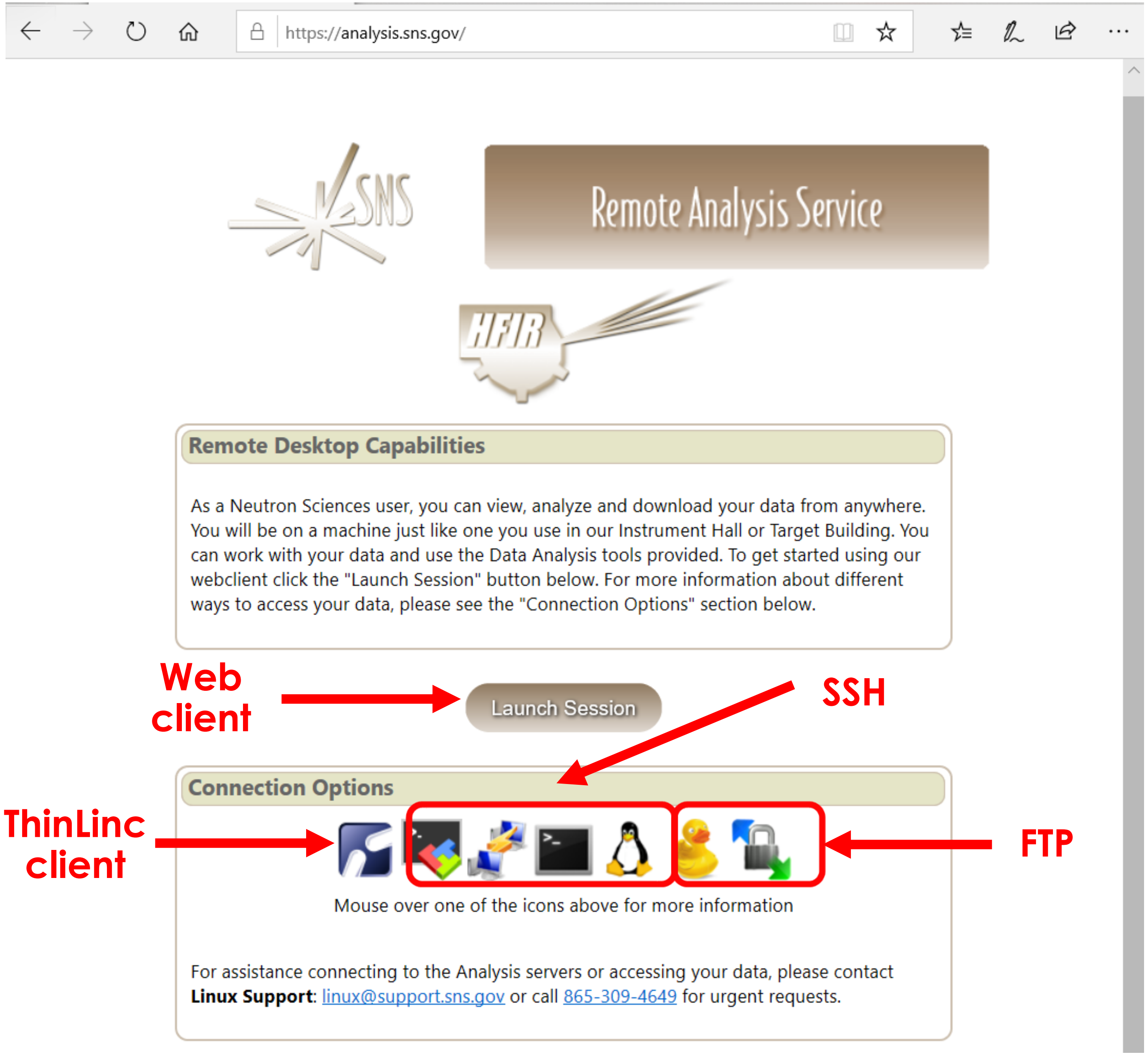




Remote Controlled Experiment

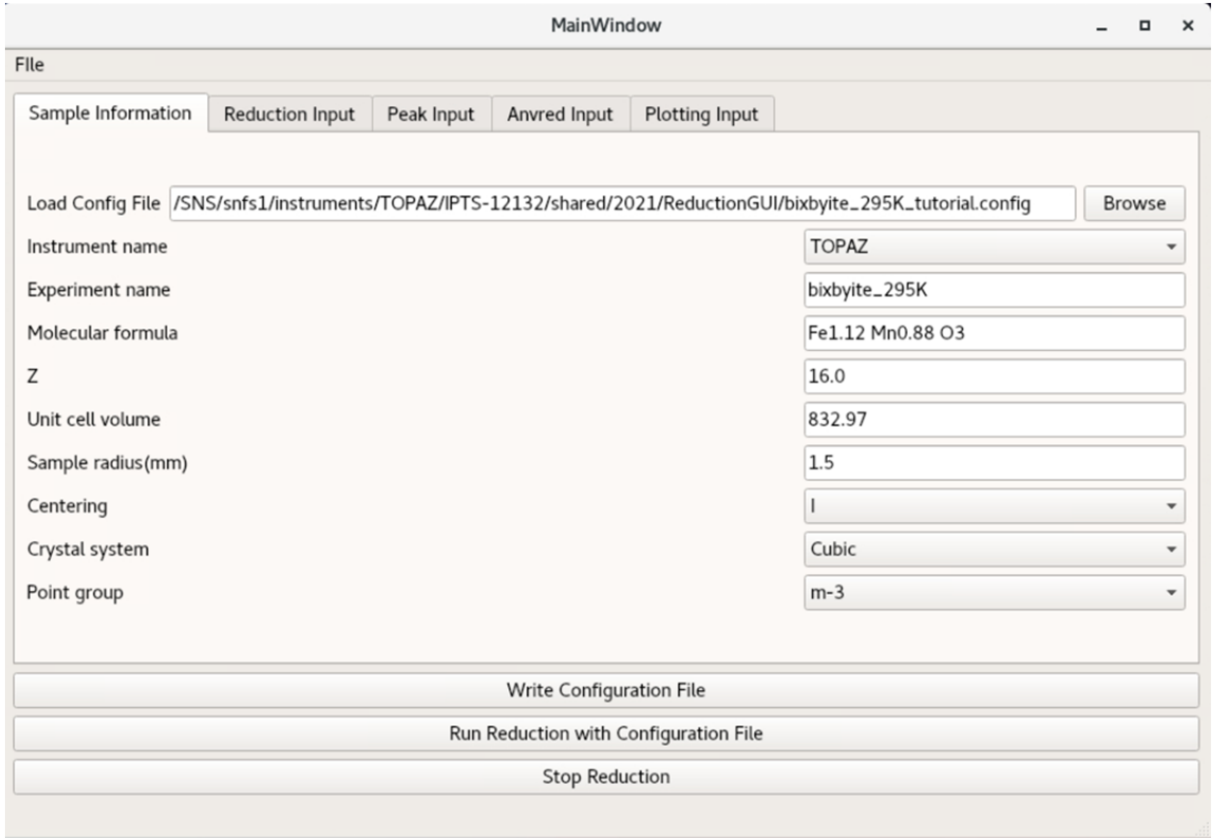
https://analysis.sns.gov



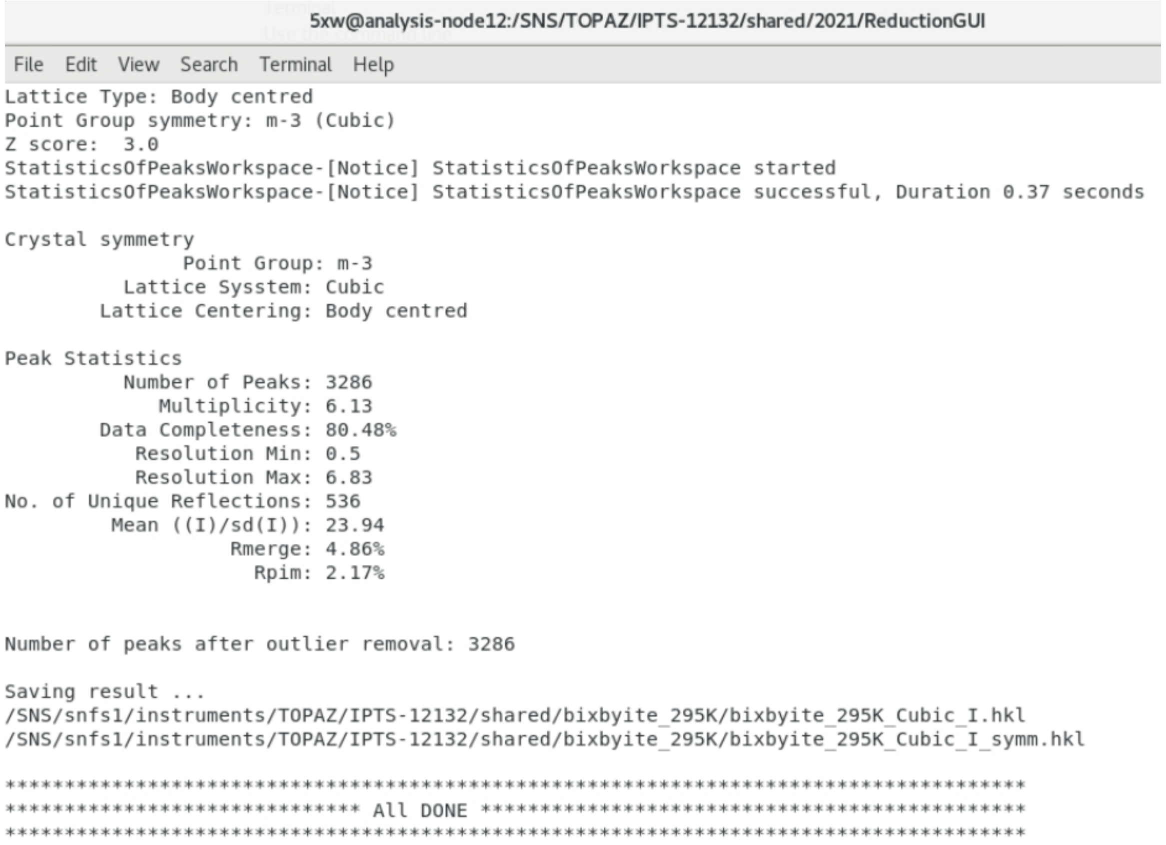


TOPAZ data reduction interface

$ python3 main.py

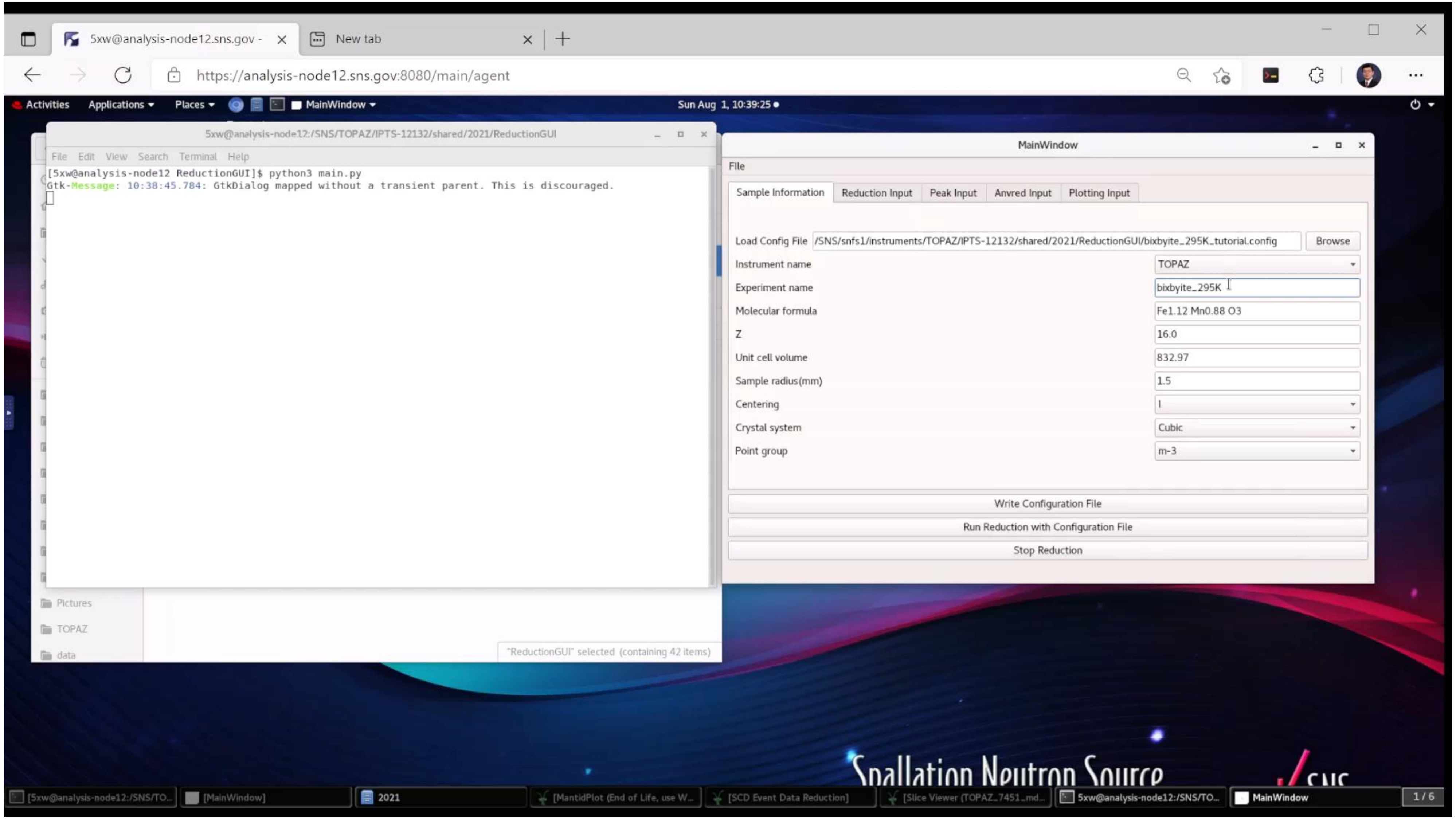


Terminal output



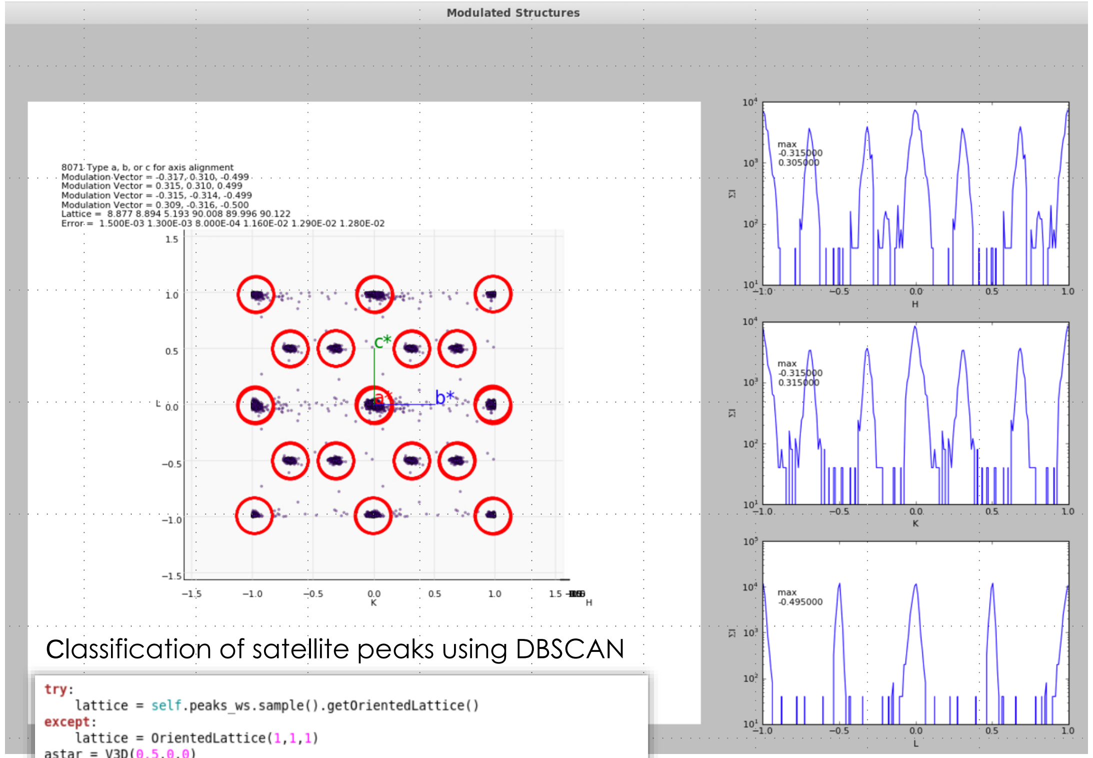
OAK RIDGE National Laboratory | SPALLATION NEUTRON SOURCE

TOPAZ data reduction demo



Machine Learning for single crystal neutron diffraction

Satellite Peak Index Product of ORNL GO! Student Project

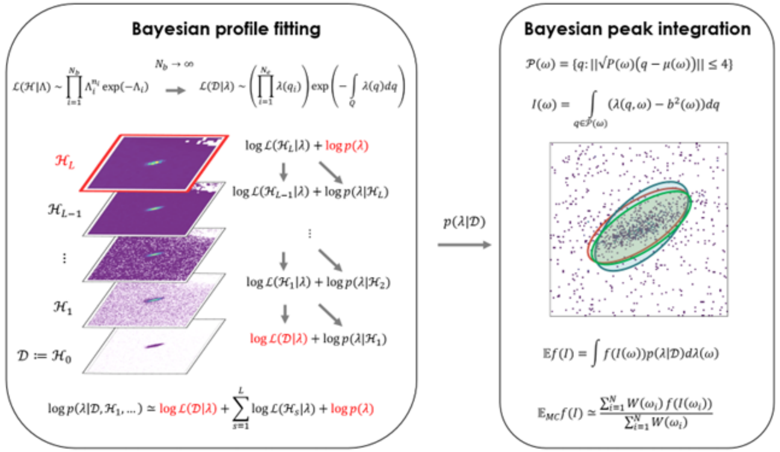


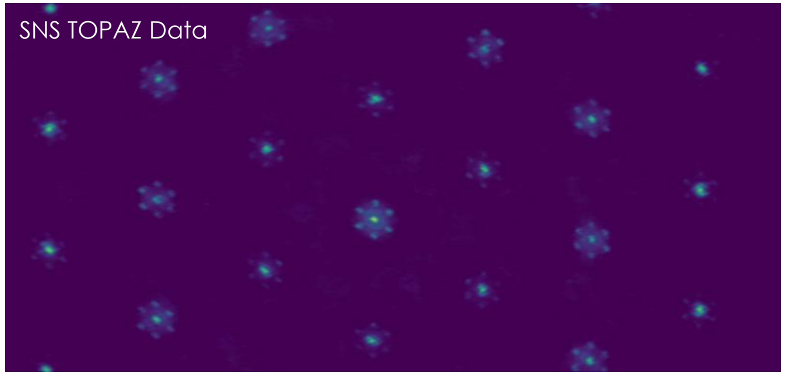

```
try:
    lattice = self.peaks_ws.sample().getOrientedLattice()
except:
    lattice = OrientedLattice(1,1,1)
astar = V3D(0.5,0,0)
bstar = V3D(0,0.5,0)
cstar = V3D(0,0,0.5)
hkls = np.array([np.array([x, y, z]) for x,y,z in zip(self.x,self.y,self.z)])
labels, centroids = self.dbscan(hkls, eps=.0425, min_points=6)

i = 0
modTest = str(self.peaks_ws.getPeak(0).getRunNumber())
lastRun = str(self.peaks_ws.getPeak(npeaksTotal-1).getRunNumber())
if lastRun != modTest:
    modTest = modTest + "-" +lastRun
modTest = modTest + " Type a, b, or c for axis alignment"
```

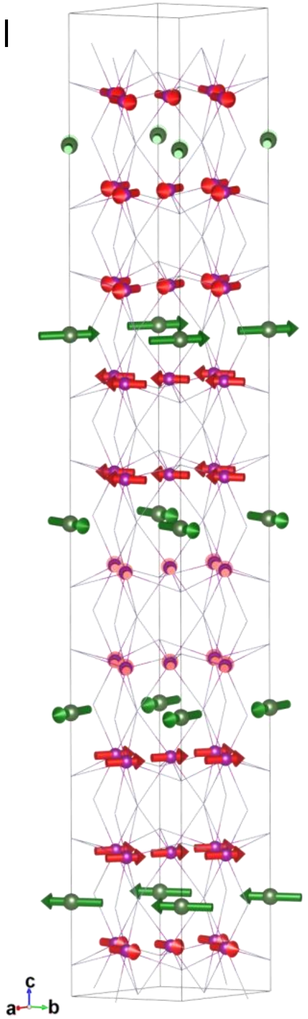
Mantid Algorithms
Available to users

Peak Integration Recent progress

- Multiresolution Bayesian optimization with global prior from ML/AI for integrating weak magnetic peaks near strong Bragg reflections







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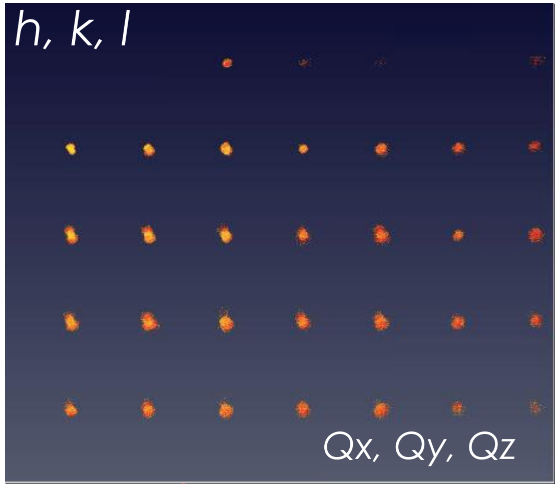
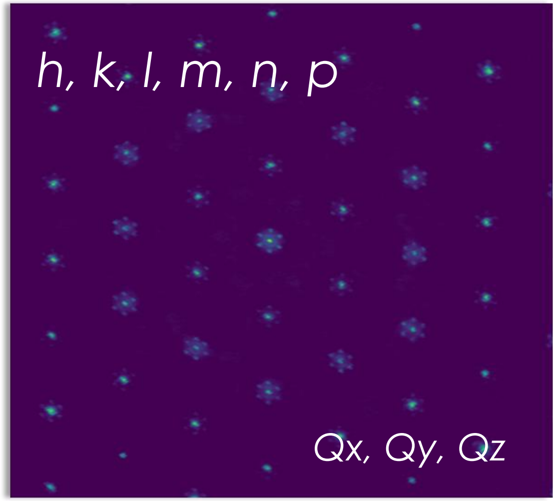
[Modulated Structure (mantidproject.org)](https://mantidproject.org)

TOPAZ Data reduction for modulated crystal





$$\boldsymbol{Q} = 2\pi(h\boldsymbol{a}^* + k\boldsymbol{b}^* + l\boldsymbol{c}^* + m\boldsymbol{q}_1 + n\boldsymbol{q}_2 + p\boldsymbol{q}_3)$$

$$\boldsymbol{Q}\begin{pmatrix} x_i \\ y_i \\ z_i \end{pmatrix} = \left(\underbrace{\begin{matrix} \mathrm{UB}_{11} & \mathrm{UB}_{12} & \mathrm{UB}_{13} \\ \mathrm{UB}_{21} & \mathrm{UB}_{22} & \mathrm{UB}_{23} \\ \mathrm{UB}_{31} & \mathrm{UB}_{32} & \mathrm{UB}_{33} \end{matrix}}_{\textbf{UB}} \; \underbrace{\begin{matrix} \mathrm{MV}_{11} & \mathrm{MV}_{12} & \mathrm{MV}_{13} \\ \mathrm{MV}_{21} & \mathrm{MV}_{22} & \mathrm{MV}_{23} \\ \mathrm{MV}_{31} & \mathrm{MV}_{32} & \mathrm{MV}_{33} \end{matrix}}_{\textbf{ModUB}} \right) \times \begin{pmatrix} h_i \\ k_i \\ l_i \\ m_i \\ n_i \\ p_i \end{pmatrix}$$

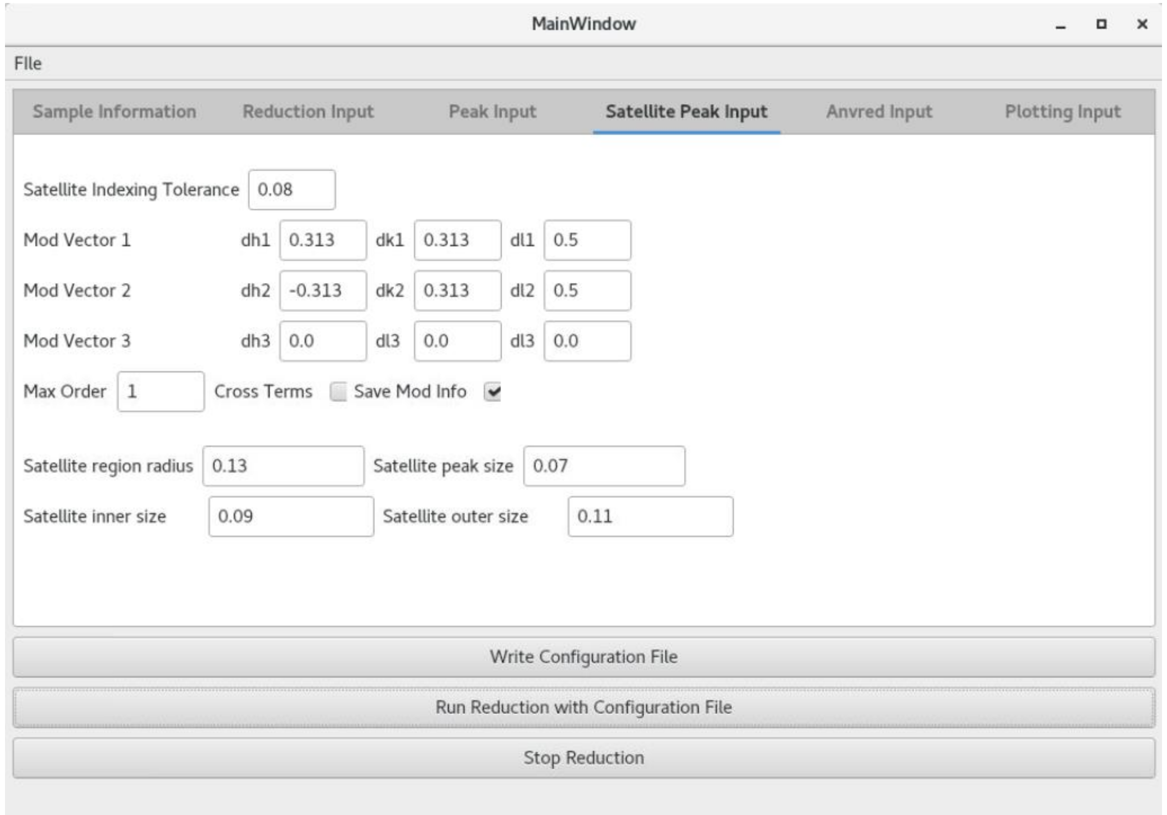
Shiyun Jin
U Wisconsin

https://docs.mantidproject.org/nightly/concepts/ModulatedStructure.html

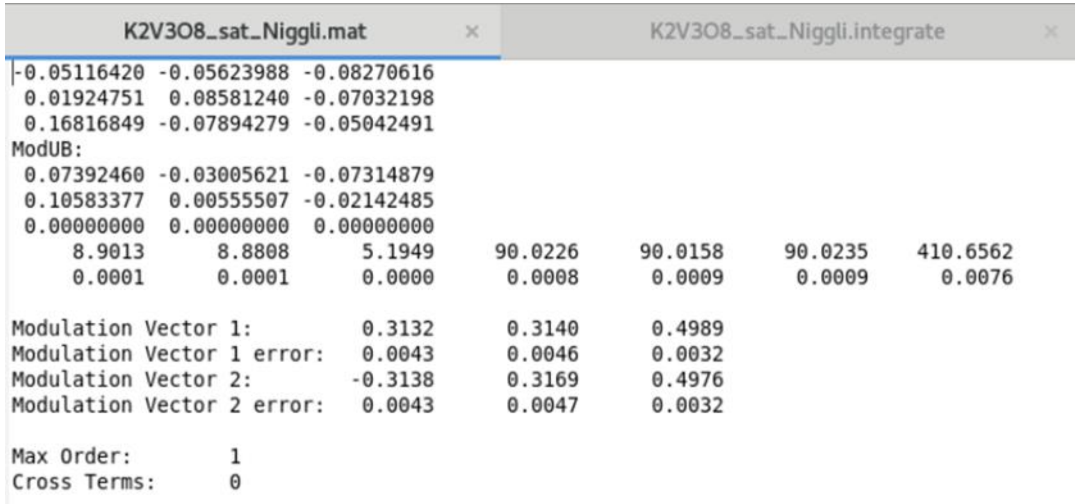
TOPAZ data reduction GUI for modulated crystal

TOPAZ Data Reduction Tutorial – Modulated Crystal

$ python3 main.py



Mod UB Matrix with q-vectors

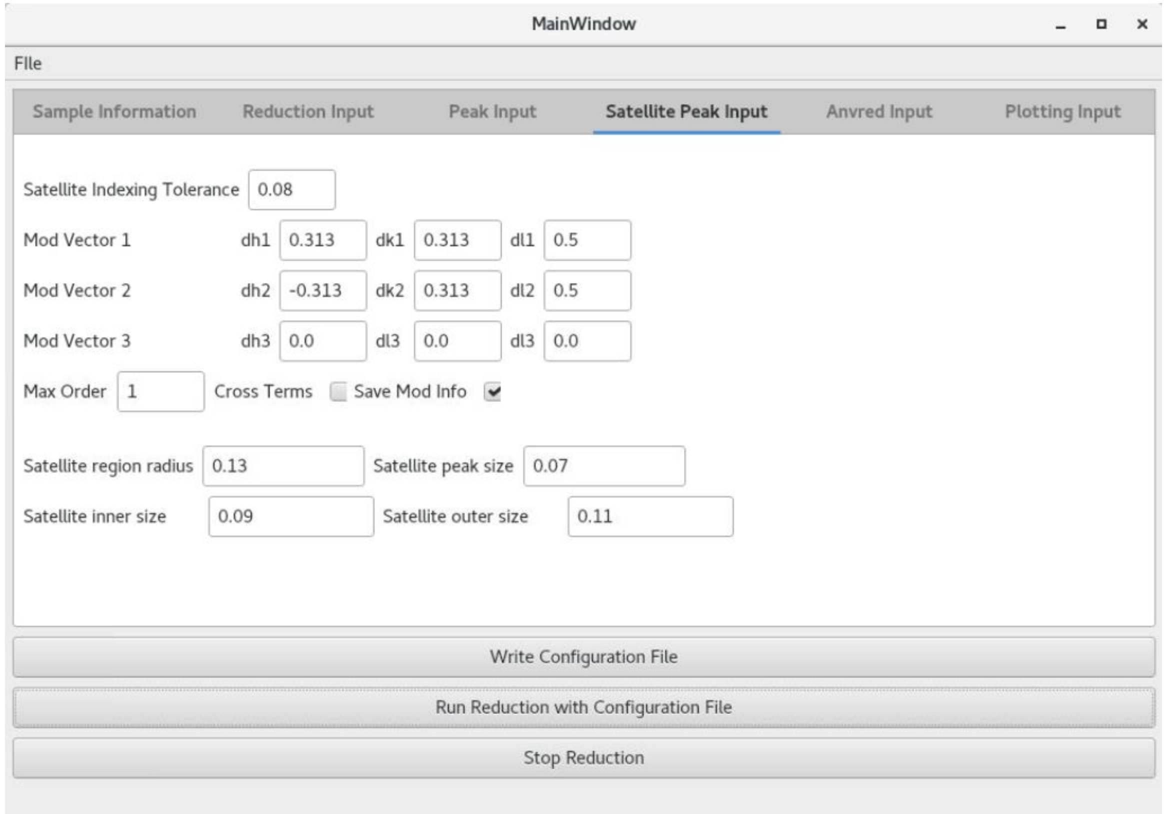


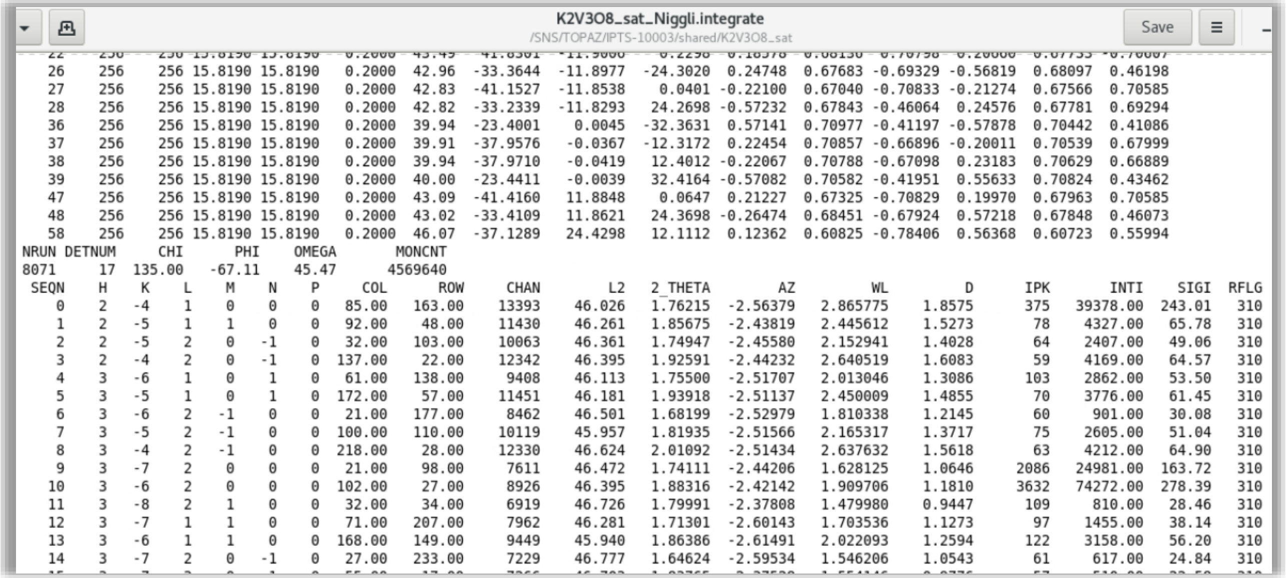

```
-0.05116420 -0.05623988 -0.08270616
 0.01924751  0.08581240 -0.07032198
 0.16816849 -0.07894279 -0.05042491
ModUB:
 0.07392460 -0.03005621 -0.07314879
 0.10583377  0.00555507 -0.02142485
 0.00000000  0.00000000  0.00000000
     8.9013      8.8808      5.1949      90.0226      90.0158      90.0235      410.6562
     0.0001      0.0001      0.0000       0.0008       0.0009       0.0009        0.0076

Modulation Vector 1:          0.3132      0.3140      0.4989
Modulation Vector 1 error:    0.0043      0.0046      0.0032
Modulation Vector 2:         -0.3138      0.3169      0.4976
Modulation Vector 2 error:    0.0043      0.0047      0.0032

Max Order:        1
Cross Terms:      0
```

TOPAZ data reduction GUI for modulated crystal

$ python3 main.py

→ *6-D* Miller Indices *h*, *k*, *l*, *m*, *n*, *p*






```
  26    256    256 15.8190 15.8190   0.2000  42.96  -33.3644  -11.8977  -24.3020  0.24748  0.67683 -0.69329 -0.56819  0.68097  0.46198
  27    256    256 15.8190 15.8190   0.2000  42.83  -41.1527  -11.8538    0.0401 -0.22100  0.67040 -0.70833 -0.21274  0.67566  0.70585
  28    256    256 15.8190 15.8190   0.2000  42.82  -33.2339  -11.8293   24.2698 -0.57232  0.67843 -0.46064  0.24576  0.67781  0.69294
  36    256    256 15.8190 15.8190   0.2000  39.94  -23.4001    0.0045  -32.3631  0.57141  0.70977 -0.41197 -0.57878  0.70442  0.41086
  37    256    256 15.8190 15.8190   0.2000  39.91  -37.9576   -0.0367  -12.3172  0.22454  0.70857 -0.66896 -0.20011  0.70539  0.67999
  38    256    256 15.8190 15.8190   0.2000  39.94  -37.9710   -0.0419   12.4012 -0.22067  0.70788 -0.67098  0.23183  0.70629  0.66889
  39    256    256 15.8190 15.8190   0.2000  40.00  -23.4411   -0.0039   32.4164 -0.57082  0.70582 -0.41951  0.55633  0.70824  0.43462
  47    256    256 15.8190 15.8190   0.2000  43.09  -41.4160   11.8848    0.0647  0.21227  0.67325 -0.70829  0.19970  0.67963  0.70585
  48    256    256 15.8190 15.8190   0.2000  43.02  -33.4109   11.8621   24.3698 -0.26474  0.68451 -0.67924  0.57218  0.67848  0.46073
  58    256    256 15.8190 15.8190   0.2000  46.07  -37.1289   24.4298   12.1112  0.12362  0.60825 -0.78406  0.56368  0.60723  0.55994
NRUN DETNUM     CHI      PHI    OMEGA       MONCNT
8071     17  135.00   -67.11    45.47      4569640
 SEQN     H    K    L    M    N    P     COL      ROW      CHAN       L2  2_THETA       AZ        WL        D      IPK      INTI    SIGI RFLG
    0     2   -4    1    0    0    0   85.00   163.00     13393   46.026  1.76215 -2.56379  2.865775   1.8575      375  39378.00  243.01  310
    1     2   -5    1    1    0    0   92.00    48.00     11430   46.261  1.85675 -2.43819  2.445612   1.5273       78   4327.00   65.78  310
    2     2   -5    2    0   -1    0   32.00   103.00     10063   46.361  1.74947 -2.45580  2.152941   1.4028       64   2407.00   49.06  310
    3     2   -4    2    0   -1    0  137.00    22.00     12342   46.395  1.92591 -2.44232  2.640519   1.6083       59   4169.00   64.57  310
    4     3   -6    1    0    1    0   61.00   138.00      9408   46.113  1.75500 -2.51707  2.013046   1.3086      103   2862.00   53.50  310
    5     3   -5    1    0    1    0  172.00    57.00     11451   46.181  1.93918 -2.51137  2.450009   1.4855       70   3776.00   61.45  310
    6     3   -6    2   -1    0    0   21.00   177.00      8462   46.501  1.68199 -2.52979  1.810338   1.2145       60    901.00   30.08  310
    7     3   -5    2   -1    0    0  100.00   110.00     10119   45.957  1.81935 -2.51566  2.165317   1.3717       75   2605.00   51.04  310
    8     3   -4    2   -1    0    0  218.00    28.00     12330   46.624  2.01092 -2.51434  2.637632   1.5618       63   4212.00   64.90  310
    9     3   -7    2    0    0    0   21.00    98.00      7611   46.472  1.74111 -2.44206  1.628125   1.0646     2086  24981.00  163.72  310
   10     3   -6    2    0    0    0  102.00    27.00      8926   46.395  1.88316 -2.42142  1.909706   1.1810     3632  74272.00  278.39  310
   11     3   -8    2    1    0    0   32.00    34.00      6919   46.726  1.79991 -2.37808  1.479980   0.9447      109    810.00   28.46  310
   12     3   -7    1    1    0    0   71.00   207.00      7962   46.281  1.71301 -2.60143  1.703536   1.1273       97   1455.00   38.14  310
   13     3   -6    1    1    0    0  168.00   149.00      9449   45.940  1.86386 -2.61491  2.022093   1.2594      122   3158.00   56.20  310
   14     3   -7    2    0   -1    0   27.00   233.00      7229   46.777  1.64624 -2.59534  1.546206   1.0543       61    617.00   24.84  310
```


OAK RIDGE National Laboratory | SPALLATION NEUTRON SOURCE

Data format for modulated crystal

- Extended SHELX HKFL 2 Laue format with six indices *h, k, l, m, n, p*

h	*k*	*l*	*m*	*n*	*p*	*I*	$\sigma(I)$	*bn*	λ	*t-bar*	<			*direction cosines*		>	*run*	*seq*
1	-3	-4	0	0	0	7302	178	2	1.89287	0.14303	0.68718	-0.46953	-0.17725	-0.45864	-0.70434	-0.75488	8072	4906
1	-3	-3	0	0	0	3498	85	2	2.37125	0.14307	0.68718	-0.41664	-0.17725	-0.61983	-0.70434	-0.66552	8072	4907
1	-7	-7	1	0	0	16	6	2	1.03874	0.14032	0.68718	-0.53077	-0.17725	-0.60149	-0.70434	-0.59767	8072	4915
1	-6	-8	1	0	0	45	10	2	0.98173	0.14142	0.68718	-0.53707	-0.17725	-0.44885	-0.70434	-0.71467	8072	4916
1	-6	-7	1	0	0	26	6	2	1.10068	0.14136	0.68718	-0.51996	-0.17725	-0.52377	-0.70434	-0.67527	8072	4917
1	-6	-6	1	0	0	22	5	2	1.24005	0.14104	0.68718	-0.50024	-0.17725	-0.61446	-0.70434	-0.61067	8072	4918
1	-5	-5	1	0	0	15	3	2	1.53722	0.14199	0.68718	-0.45615	-0.17725	-0.63150	-0.70434	-0.62757	8072	4921
1	-4	-6	1	0	0	24	3	2	1.40411	0.14320	0.68718	-0.47362	-0.17725	-0.40265	-0.70434	-0.78369	8072	4922
1	-4	-5	1	0	0	9	2	2	1.66864	0.14353	0.68718	-0.43555	-0.17725	-0.51087	-0.70434	-0.74159	8072	4923
1	-7	-8	0	-1	0	36	13	2	0.83093	0.14039	0.68718	-0.56147	-0.17725	-0.50414	-0.70434	-0.65673	8072	4932
1	-7	-7	0	-1	0	42	8	2	0.91129	0.14013	0.68718	-0.54893	-0.17725	-0.57005	-0.70434	-0.61191	8072	4933
1	-5	-5	0	-1	0	22	4	2	1.27571	0.14150	0.68718	-0.49500	-0.17725	-0.58308	-0.70434	-0.64475	8072	4939
1	-3	-4	0	-1	0	17	2	2	1.71003	0.14399	0.68718	-0.42858	-0.17725	-0.45904	-0.70434	-0.77861	8072	4943
1	-3	-3	0	-1	0	8	1	2	2.09457	0.14423	0.68718	-0.37306	-0.17725	-0.60221	-0.70434	-0.70628	8072	4944
2	-8	-8	0	1	0	25	7	2	0.90866	0.14079	0.68718	-0.51303	-0.17725	-0.60434	-0.70434	-0.61015	8072	4953
2	-7	-7	0	1	0	12	6	2	1.05860	0.14144	0.68718	-0.48368	-0.17725	-0.61649	-0.70434	-0.62185	8072	4957
2	-5	-7	0	1	0	10	4	2	1.17683	0.14339	0.68718	-0.45928	-0.17725	-0.43942	-0.70434	-0.77240	8072	4963
2	-5	-6	0	1	0	9	4	2	1.35646	0.14361	0.68718	-0.42626	-0.17725	-0.53288	-0.70434	-0.73144	8072	4964
2	-4	-5	0	1	0	9	2	2	1.70479	0.14502	0.68718	-0.35999	-0.17725	-0.52485	-0.70434	-0.77173	8072	4966
2	-7	-8	-1	0	0	30	10	2	0.84506	0.14132	0.68718	-0.52441	-0.17725	-0.51348	-0.70434	-0.67972	8072	4979
2	-6	-7	-1	0	0	21	7	2	0.97402	0.14198	0.68718	-0.49954	-0.17725	-0.51027	-0.70434	-0.70055	8072	4983
2	-6	-6	-1	0	0	52	7	2	1.08204	0.14185	0.68718	-0.47900	-0.17725	-0.58895	-0.70434	-0.65146	8072	4984
2	-6	-5	-1	0	0	33	5	2	1.21030	0.14145	0.68718	-0.45477	-0.17725	-0.67755	-0.70434	-0.57863	8072	4985
2	-5	-6	-1	0	0	11	5	2	1.14255	0.14285	0.68718	-0.46607	-0.17725	-0.50501	-0.70434	-0.72692	8072	4987
2	-5	-5	-1	0	0	28	5	2	1.29798	0.14283	0.68718	-0.43648	-0.17725	-0.59704	-0.70434	-0.67359	8072	4988
2	-4	-5	-1	0	0	8	3	2	1.38444	0.14399	0.68718	-0.42153	-0.17725	-0.49183	-0.70434	-0.76227	8072	4990

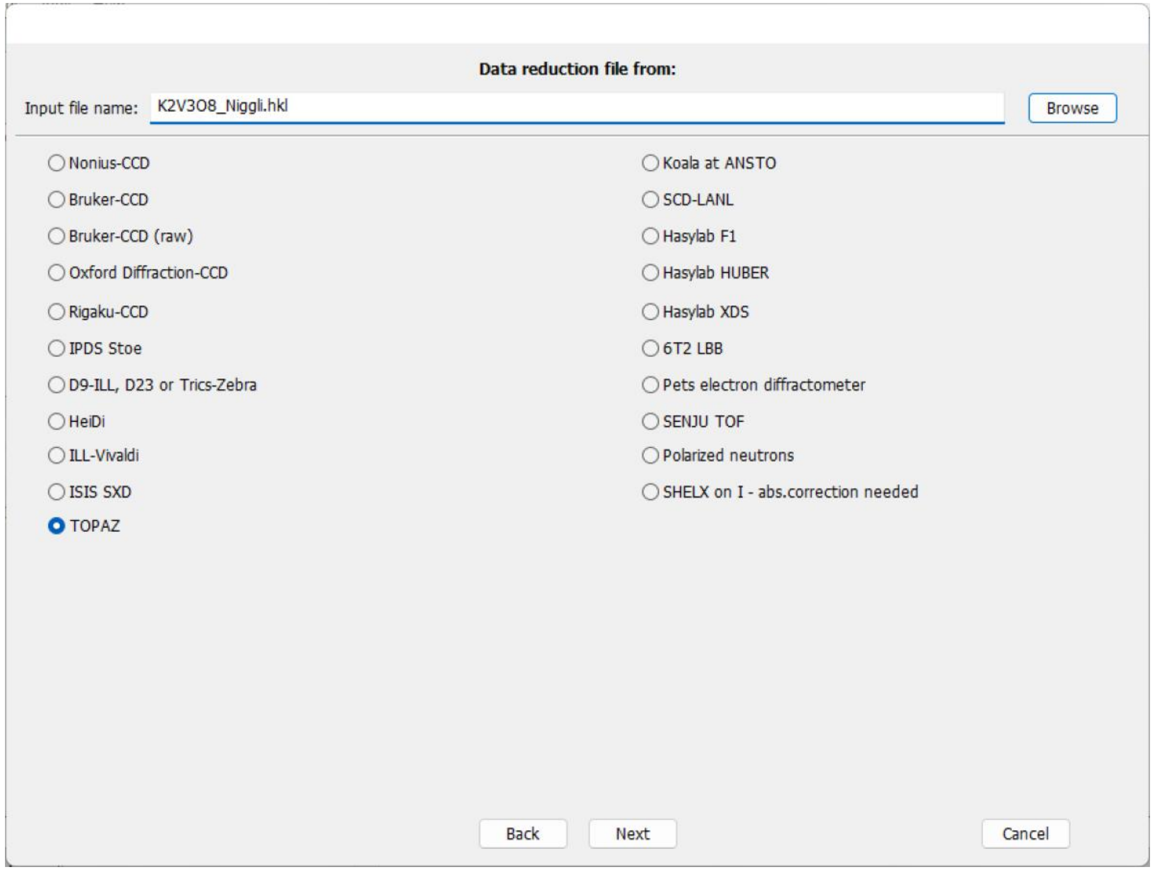
TOPAZ Data format: SHELX HKLF 2 Laue

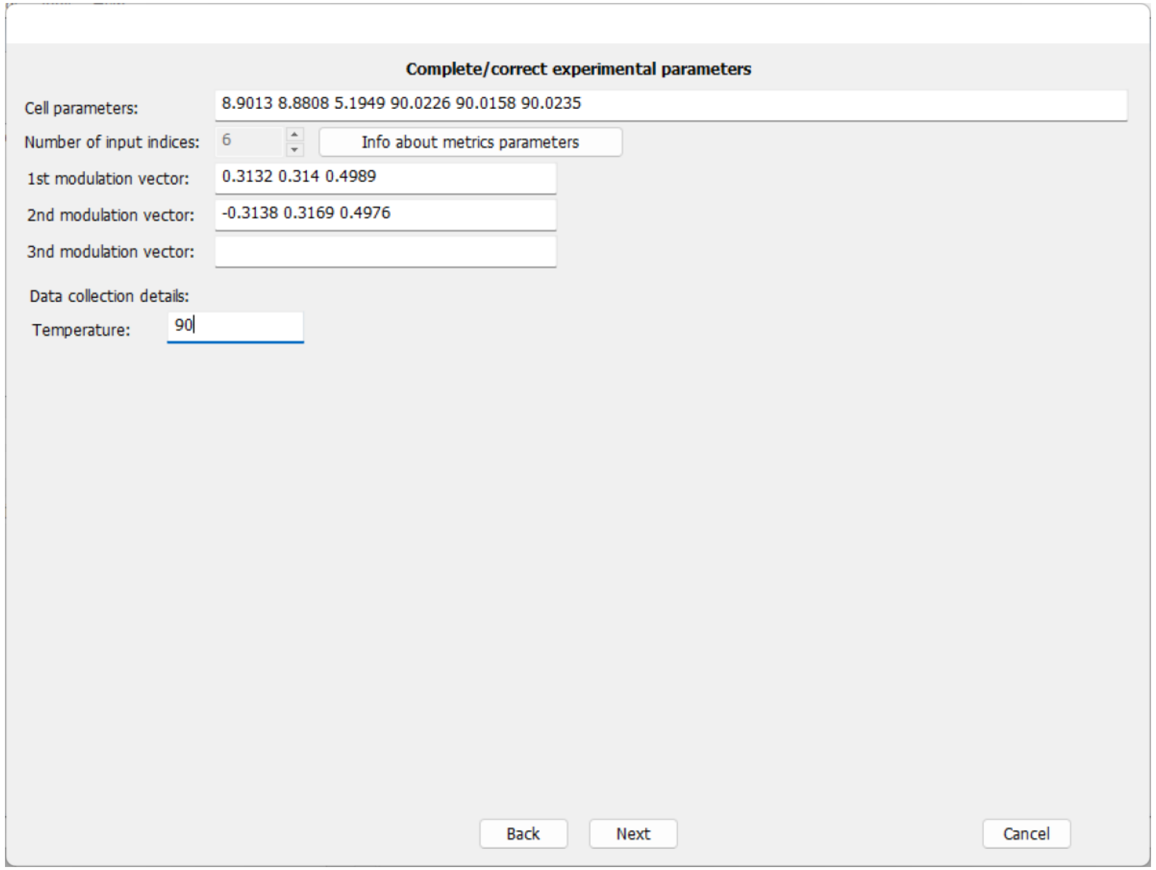
- **HKLF 2** ! + batch number (BN) and wavelength **λ** for individual reflections
- **Data Format** 3I4, 2F8.2,I4,F8.4
- 12341234123412345678123456781234123456 78
- *h* *k* *l* *Fo²* *σ(Fo²)* BN **λ**
- **Neutron Time of Flight Laue**
  - Reflections are measure on a stationary single crystal sample.
  - Integrated intensities are corrected for intensity distributions by neutron wavelengths, Lorentz and sample absorption.
- It is possible to refine the scale factors BN for different sets of reflections measured at different sample orientations / or on different detectors

The neutron wavelength for each reflection was recorded separately. No symmetry average is applied to individual reflections due to wavelength- dependent sample extinction

JANA2020 – Neutron structure solution and refinement

JANA2020 Data import





Software

Oak Ridge National Laboratory | High Flux Isotope Reactor | Spallation Neutron Source   Single Crystal Diffraction   Instrument Suite   Getting Started

> Software

Software

- Resources
  - Data reduction
  - Structure databases
  - Web applications
  - Structure refinement
  - Diffuse scattering analysis
- Reduction
  - Loading data
  - Converting data to Q-sample
  - Determining the UB-matrix
  - Refining the UB-matrix
  - Integrating peaks
  - Normalizing data

Zach Morgan

https://single-crystal.ornl.gov/software/

Welcome to ORNL!

Thank you!