

SNS BL-12 TOPAZ

Introduction to data collection, reduction and analysis

Xiaoping Wang and Christina Hoffmann

Neutron scattering Division

wangx@ornl.gov; choffmann@ornl.gov



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U.S. DEPARTMENT OF
ENERGY

Outline

Neutron Time of Flight (TOF) Laue diffraction

- Event-base data collection
- 3D reciprocal space mapping

TOPAZ experiment workflow

- Sample mount → Experiment planning → Data collection

TOPAZ data reduction

- Data Reduction GUI
- Data normalization and absorption correction

Data analysis, Structure solution and refinement

- JANA2020 [Example](#)

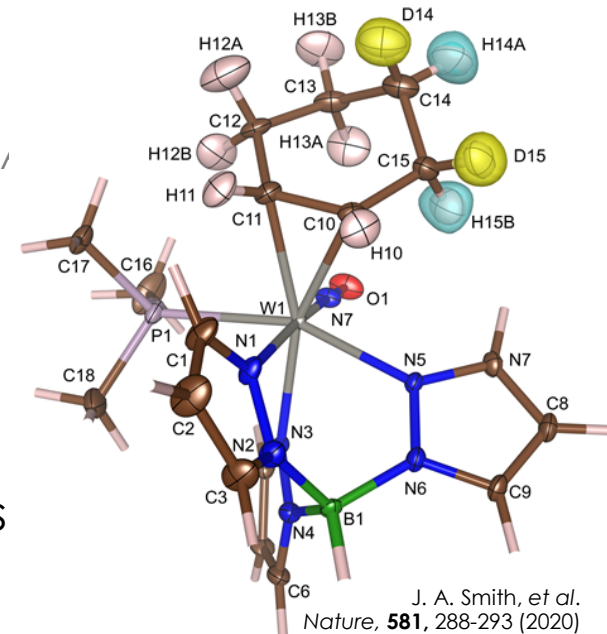
Neutron single crystal diffraction

✚ **Neutron diffraction** *Weak* neutron $\sim$ nuclear interaction E 81.8 meV at 1.0 $\text{\AA}$

- Scattering length b :
 - Fourier transform of the nuclear density of an atom
 - b is isotope specific
 - H -3.74 fm, D 6.67 fm
 - A number that is independent of scattering angle and wavelengths

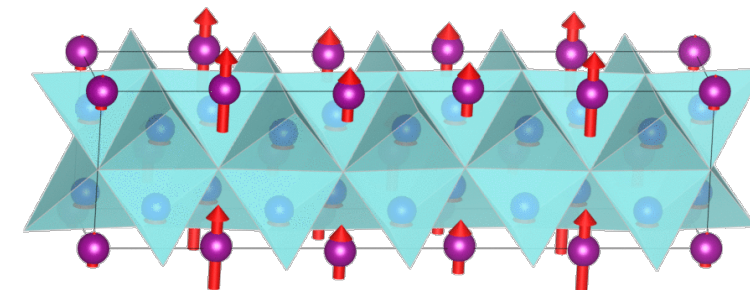
Magnetic scattering

Neutron ($spin \pm$) $\sim$ (out-shell) electron interaction, strong Q dependence



✚ **X-ray diffraction** Photon $\sim$ electron interaction, $E_{Mo\ K\alpha}$ 17.5 keV, $\lambda = 0.71\ \text{\AA}$

- Form factor f :
 - Fourier transform of the atomic electron density of an atom
- $f \propto$ Atomic Z
 - Decay as Q increases



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

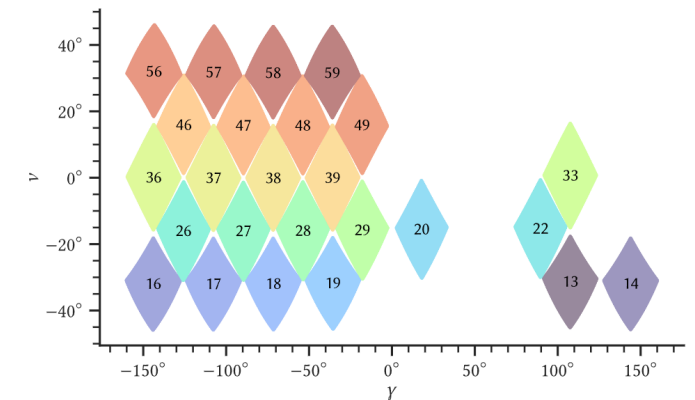
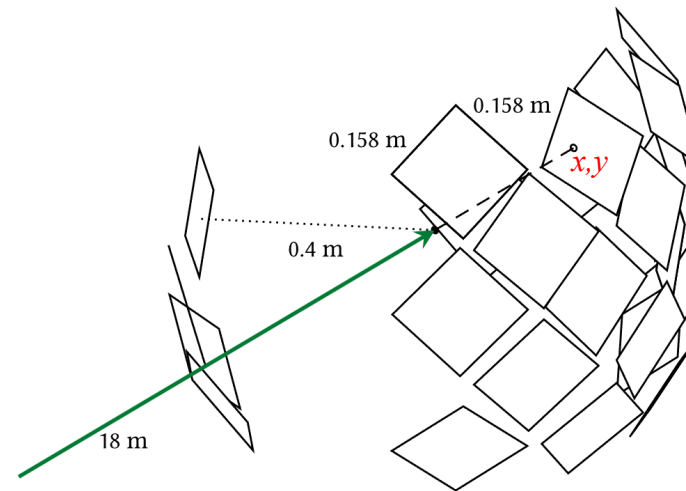
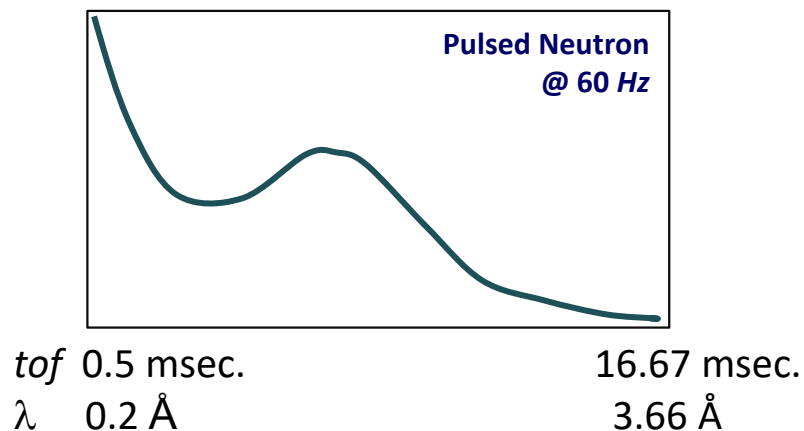
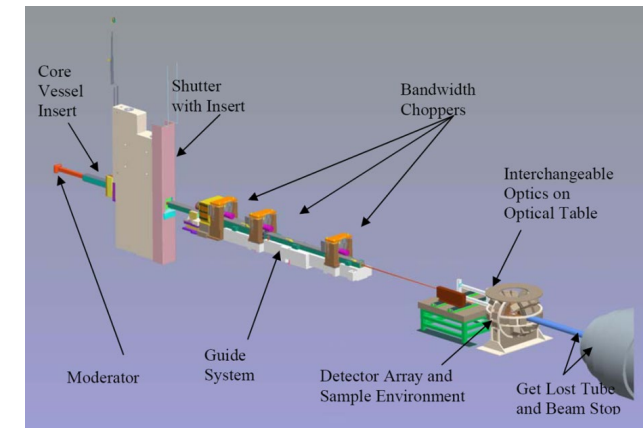
Neutron Time of Flight Laue diffraction

Neutron Time of Flight: Event-based neutron detection technique

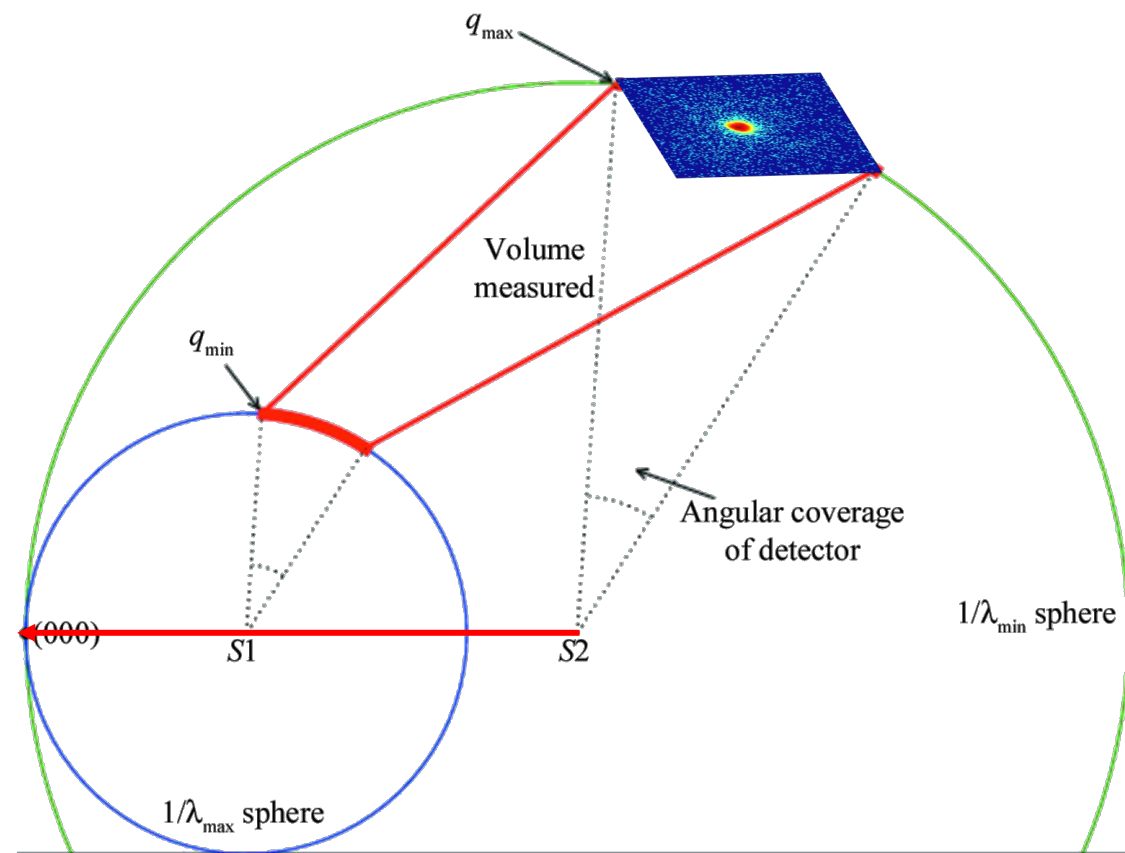
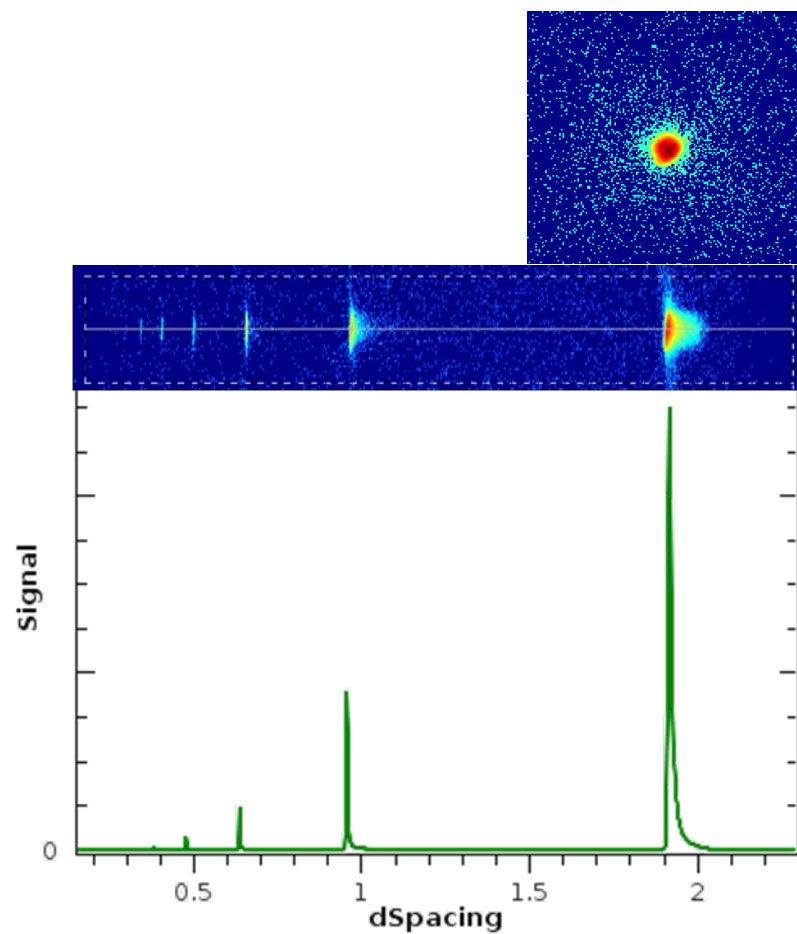
- de Broglie equation relates neutron wavelength to its momentum:

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

- 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.



Neutron Time of Flight (TOF) Laue diffraction



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

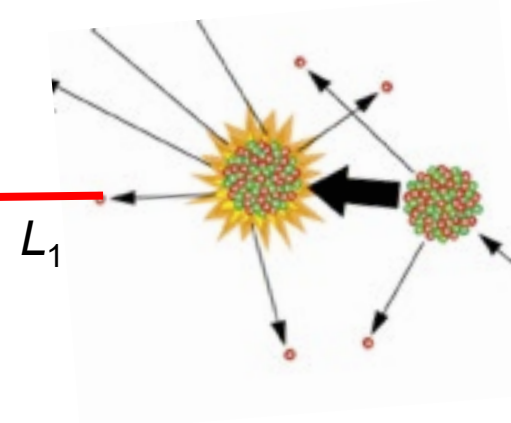
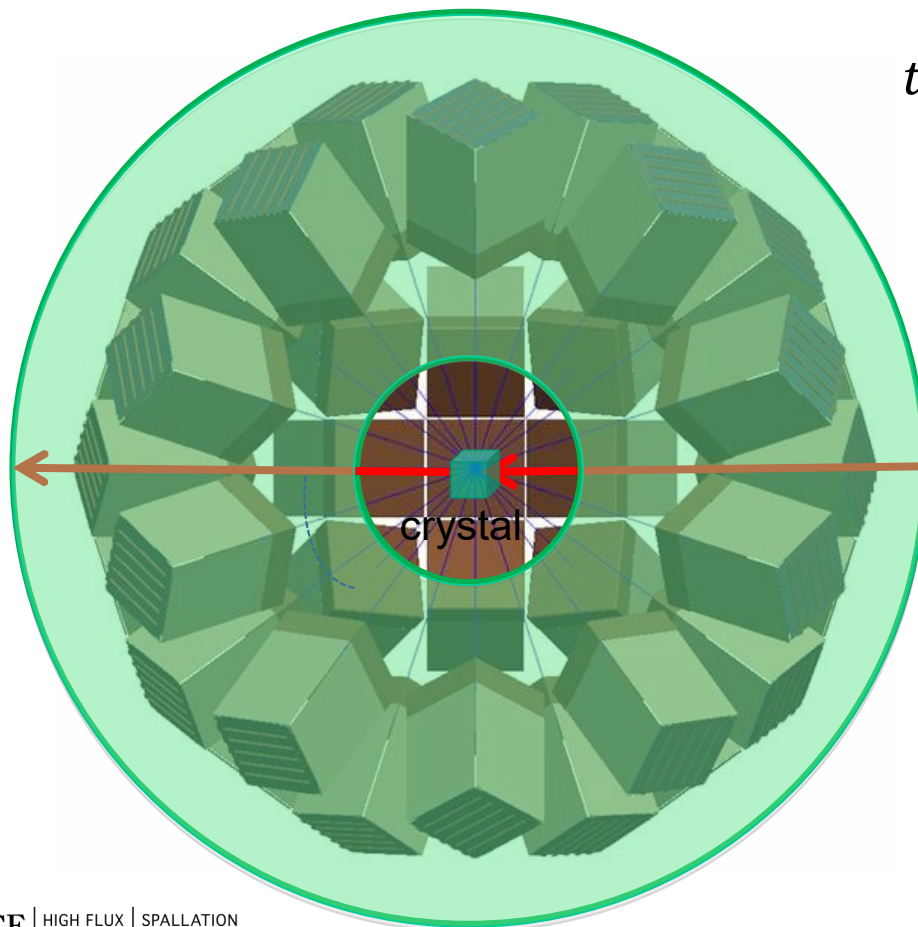
Neutron Wavelength-resolved Laue

Combine de Broglie's equation and Bragg's law

de Broglie Equation $\lambda = \frac{h}{mv} = \frac{ht}{m(L_1 + l_2)}$

$$t = \frac{m}{h} (L_1 + l_2) \times 2d \sin \theta_{x,y}$$

$$L_1 = 18 \text{ m}$$
$$l_2 = 0.39 \text{ to } 0.46 \text{ m}$$



Pulsed Neutron
60 Hz

Neutron Time-of-flight Laue
Wavelength-resolved Laue

Laue Condition

$$\mathbf{k}_f - \mathbf{k}_i = \mathbf{G}$$

Square both sides and apply the trigonometric Identity

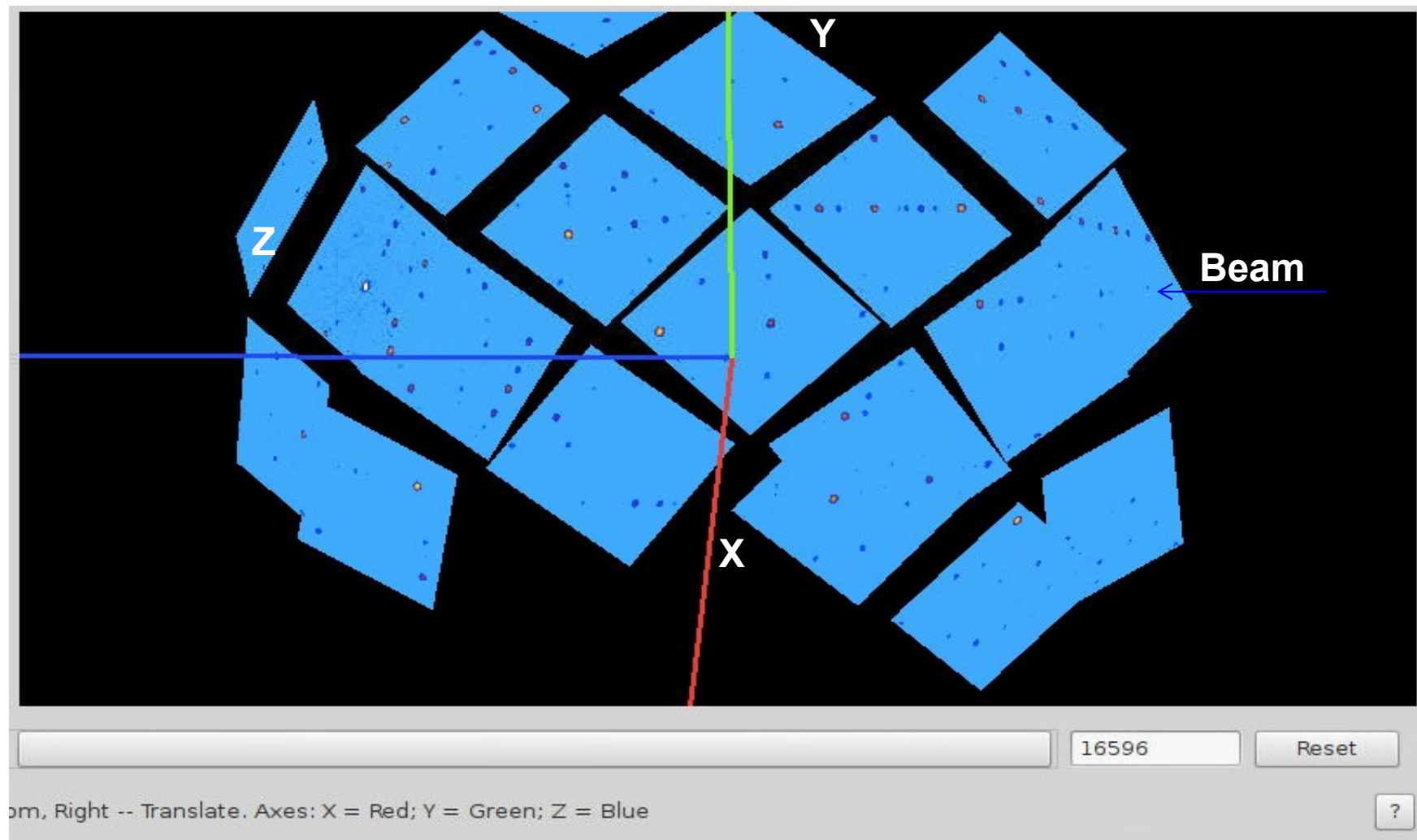
$$1 - \cos 2\theta = 2\sin^2\theta$$

$$|\mathbf{k}_f| = |\mathbf{k}_i| = \frac{2\pi}{\lambda} \text{ and } |\mathbf{G}| = \frac{2\pi}{d}$$

$$\lambda = 2d \sin \theta_{x,y}$$

Bragg's Law

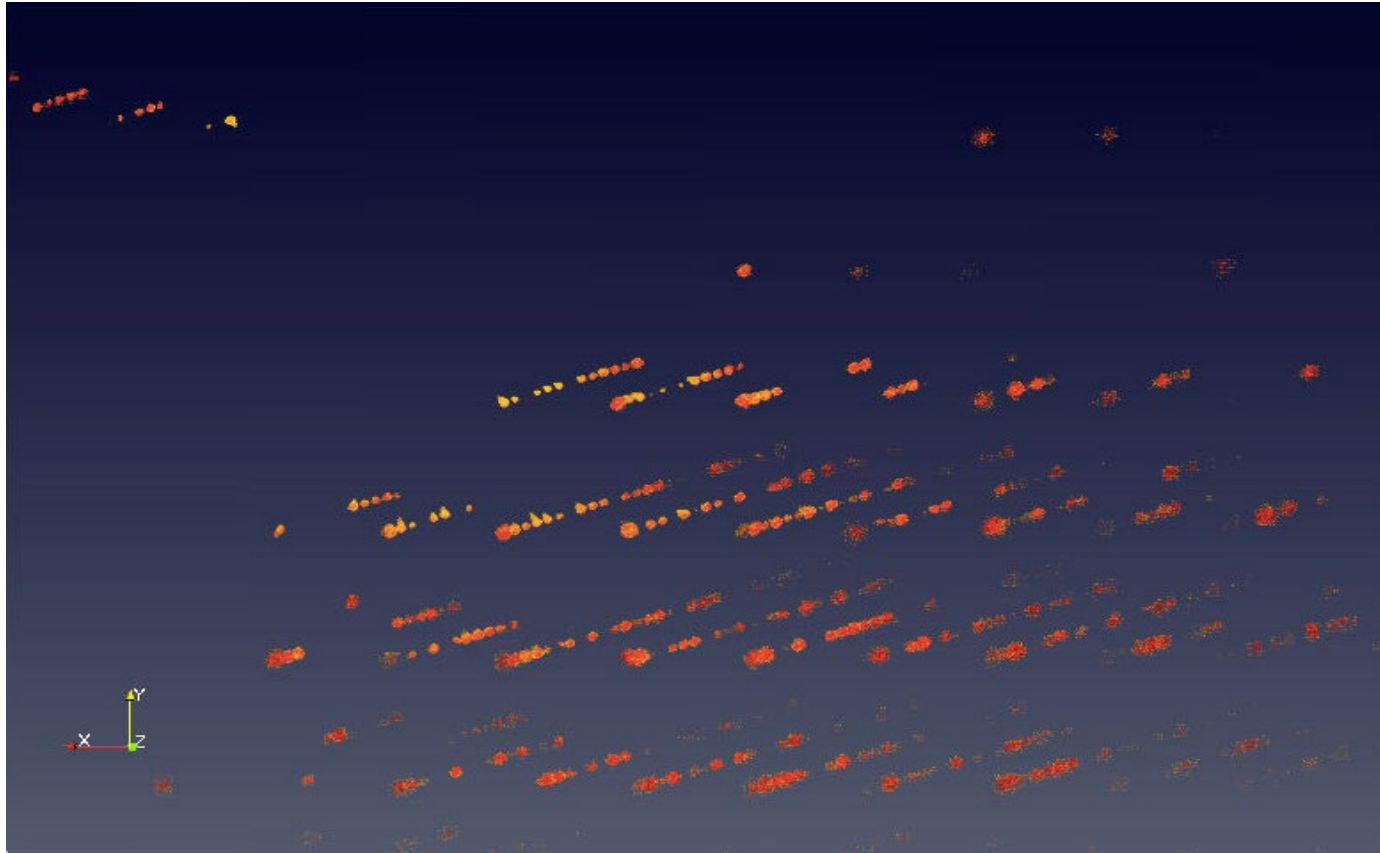
Single crystal peaks on 2D detector space



Single crystal peaks in 3D Q space

Sample continuous reciprocal Q space

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



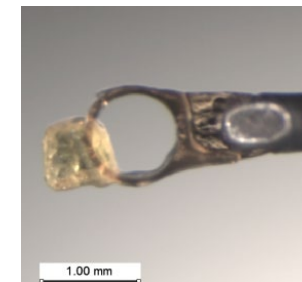
$$\frac{\pi}{4} \frac{(\lambda_{\max} - \lambda_{\min}) d_{\max}^{*4}}{V^*}$$

TOPAZ Ambient Goniometer

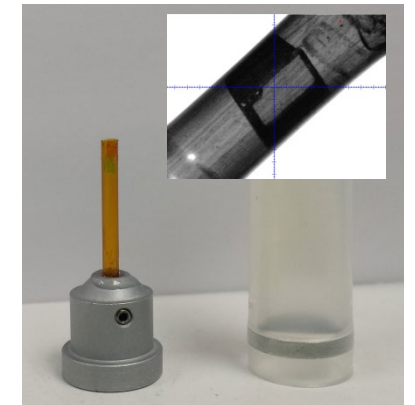


- A **two-axis** goniometer
 - Omega, phi with chi fixed at 135°.
 - Both omega and phi are fitted with sliprings that allow unlimited 360° rotational motion.
 - The omega and phi rotation axes are separated by 45 degrees.
 - Sample mount

MiTeGen loop (1 mm ϕ)



Glued or Coated with perfluorinated grease

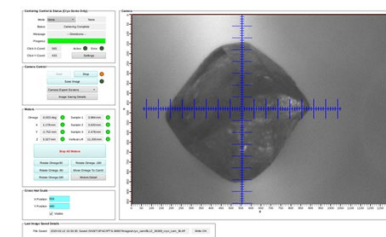
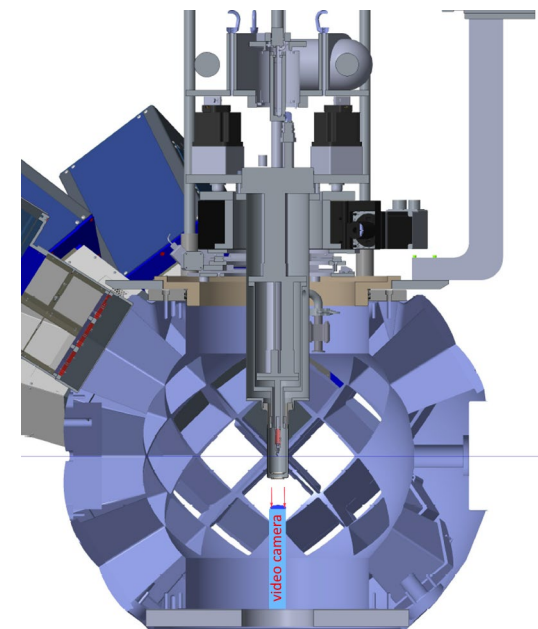
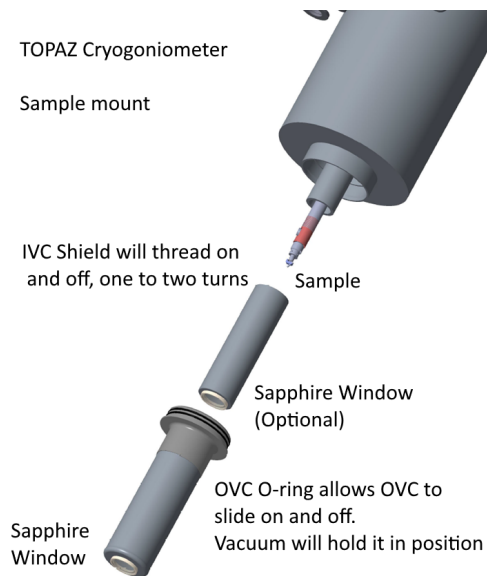


TOPAZ Cryogenic Goniometer

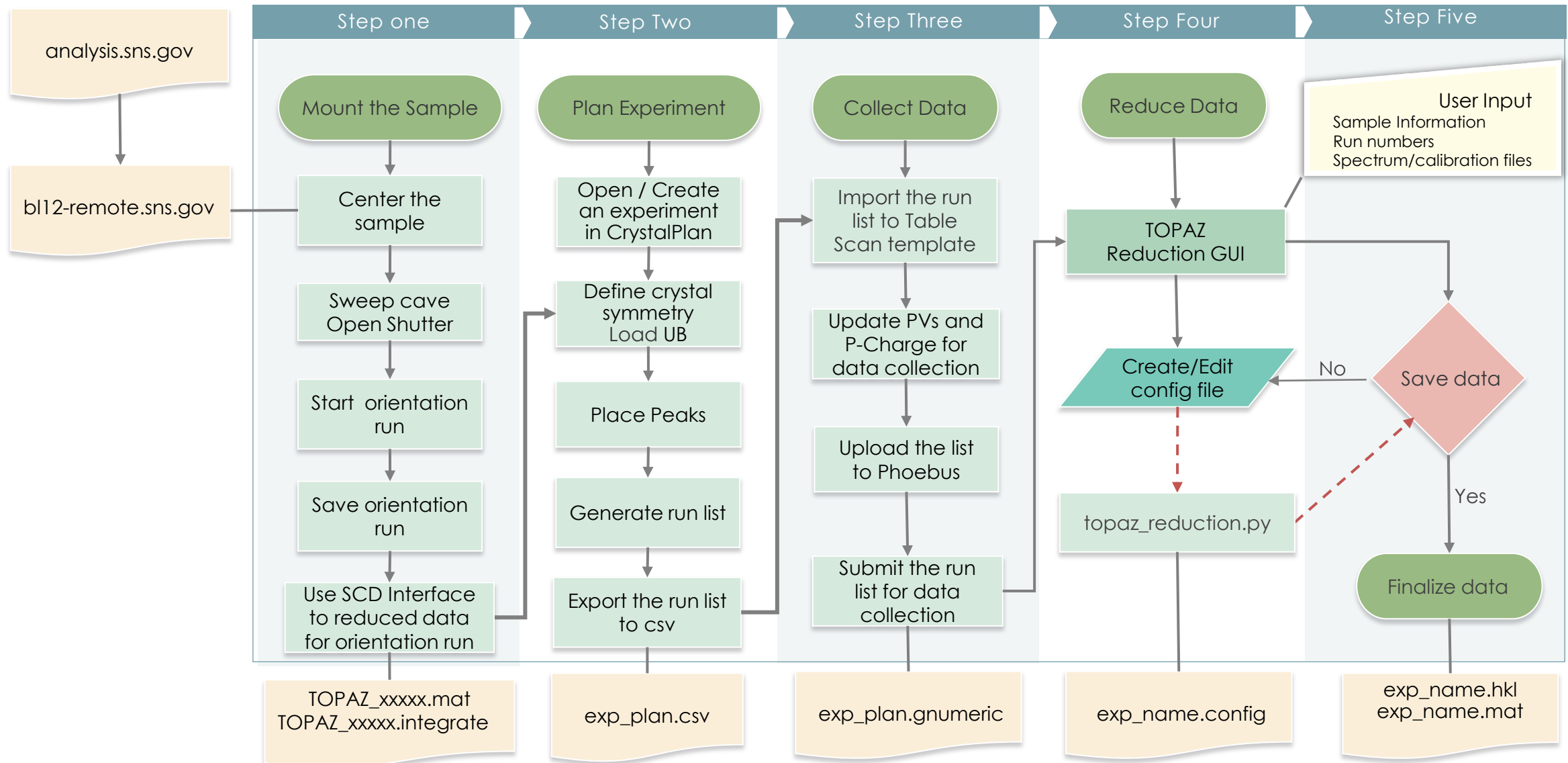
One axis of rotation (360°) with precision motor controls to center, orient, and hold sample in temperature range 5 K – 300 K.



- ✎ A video camera is mounted on the base of the DAT
- ✎ Click-to-center of single crystal sample



TOPAZ Experiment Workflow



TOPAZ Live Instrument Data

https://neutrons.ornl.gov/topaz

OAK RIDGE National Laboratory

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Science and Discovery
Neutron Sciences

Home About Future Science For Users For Industry Publications

Home » Single-Crystal Diffractometer

Single-Crystal Diffractometer TOPAZ | BL-12 | SNS

Overview Team Capabilities User Guidance Live Instrument Data Operating Status Publications Spec Sheet Co

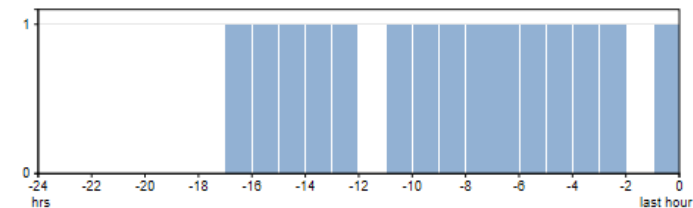


<https://neutrons.ornl.gov/topaz>

TOPAZ Monitor

[home](#) > [topaz](#) > monitor

live monitoring: **status** | [runs](#) | [PVs](#)



220Det39

Proposal: IPTS-25682 Run: 38863

Status: Recording Count rate: 20567

Systems: **Workflow**

Last run: 38863 from IPTS-25682 created on Nov. 4, 2020, 7:45 a.m.

Signal/PV	Value	History	Last Updated
LakeshoreSet1	300		Nov. 3, 2020, 9:30 p.m.
sample_ramp_rate	0.001		Nov. 4, 2020, 8:30 a.m.
sample_temp	299.998		Nov. 4, 2020, 8:30 a.m.

Key	Value	Last Updated
count_rate	20567	Nov. 4, 2020, 8:30 a.m.
has_states_count	0	Nov. 4, 2020, 8:30 a.m.
monitor_count_1	2620	Nov. 4, 2020, 8:30 a.m.
monitor_count_2	4867	Nov. 4, 2020, 8:30 a.m.
paused	false	Nov. 3, 2020, 4:42 p.m.
recording	true	Nov. 4, 2020, 7:45 a.m.
scan_index	0	Oct. 29, 2020, 10:23 a.m.
scanning	false	Oct. 29, 2020, 10:23 a.m.
system_dasmon	0	Nov. 4, 2020, 8:30 a.m.
system_pvsd	0	Nov. 4, 2020, 8:30 a.m.
total_charge	3.83415e+12	Nov. 4, 2020, 8:30 a.m.
total_counts	5.56735e+07	Nov. 4, 2020, 8:30 a.m.
total_time	2748.07	Nov. 4, 2020, 8:30 a.m.

TOPAZ DAS OPI

https://status.sns.ornl.gov/dbwr/view.jsp?display=https%3A//webopi.sns.gov/bl12/files/bl12/opi/BL12_Main.opi

← → ↻ 🏠 🔒 https://status.sns.ornl.gov/dbwr/view.jsp?display=https%3A//webopi.sns.gov/bl12/files/bl12/opi/BL12_Main.opi¯os=%7E

Accelerator
Mode: Target Power: 1400 kW Charge: 2.332I

HFIR

BL-1A USANS
Shutter Run Run
Scan Running
Main
T0 Chopper
IPPS

BL-1B NOMAD
Shutter Run Idle
Scan Finished
Main
T0 Chopper
Choppers
Vacuum
IPPS

BL-2 BASIS
Shutter Run Idle
Scan Finished
Main
T0 Chopper
Choppers
Vacuum
IPPS

BL-5 CNCS
Shutter Run Idle
Scan Finished
Main
Choppers
IPPS

BL-6 EQ-SANS
Shutter Run Run
Scan Running
Main
Vacuum
Choppers
IPPS

BL-7 VULCAN
Shutter Run Idle
Scan Aborted
Main
Detector
Choppers
IPPS

BL-9 CORAL
Shutter Run Idle
Scan Finished
Main
Choppers
IPPS

BL-12 TOPAZ
Shutter Run Idle
Scan Aborted
Main
IPPS

BL-13 FNPB
Shutter Run Idle
Scan Aborted
Main
Choppers
IPPS

BL-14B HYSPEC
Shutter Run Idle
Scan Aborted
Main
Choppers
IPPS

BL-15 NSLS-II
Shutter Run Idle
Scan Aborted
Main
Choppers
IPPS

Summaries
SE Cage CMF NCL Gateways

TOPAZ

Accelerator & Shutters

Power 1405 kW
Moderator Temp 19.60 K
Primary Shutter

Choppers

SKF Choppers

Vacuum

Vacuum

Software Status

IOC Status
Data / Reduction
Archiver Status

Motors

Guide 5
Guide 6
Orig Ambient Gonio
Ambient Gonio
Cryo Gonio
Slit1
Aperture
Overall Status
Idle
Gonio Selection
Cryo Gonio

Stop All

Sample Environment

LN2 Dewar Level
LN2 Plant
Cryostream
Disabled
Moxa
Sample LED
Waveform Generator
HV Pulser
Potentiostat
Generic HW
Cryo Gonio CCR
Alarm Enabled
He Compressor
Alarm Enabled

Detector Control

nED
Detectors
ADnED Neutrons
ADnED Metadata
ADnED Single Module
ADnED Waterfall
Timing
Det HV
Det LV
Beam Monitor 1
Beam Monitor 2

Experiment

User
IP
BL12_Dashboard.bob
Run
Dashboard
Range/Align
Table Scan
Temperature Ramp
Scan Alarm
No Alarm
Alarm Enabled

Main Reduction Params

omega	54.572 deg
phi	0.000 deg
chi	0.000 deg
sample_temp	189.541 K
sample_ramp_rate	4.039 K/min

Sample Centering & Cameras

Ambient Gonio Cam
Cryo Gonio Cam
Image Saving

Detector Plots

2D Detector Plot
2D Detector Plot (4x4)
Single Module
1D Detector Plots
1D BM Plots
d-Space Waterfall

Data Collection – EPICS Phoebus Interface

Instrument Status

Beam Power (kW): 1378.06 kW

Primary Shutter:

Acquisition Software Status

Data/Reduction Status

Proposal Information

Proposal #: IPTS-12132

Proposal Title: NX School: High-resolution single crystal structure

Team Members: Xiaoping Wang, Justin Paloni, Ashley Cardenal, (XCAMS/UCAMS)

Run Information

Scan Status: Scanning

Run Status: Run Abort

Run Number: 40731

Run Time: 7.3 s

Total Neutron Counts: 835132

Count Rate (counts/s): 121473

Total Proton Charge: 0.0102 C

Beam Monitor 1 Counts: 18959

Beam Monitor 2 Counts: 17957

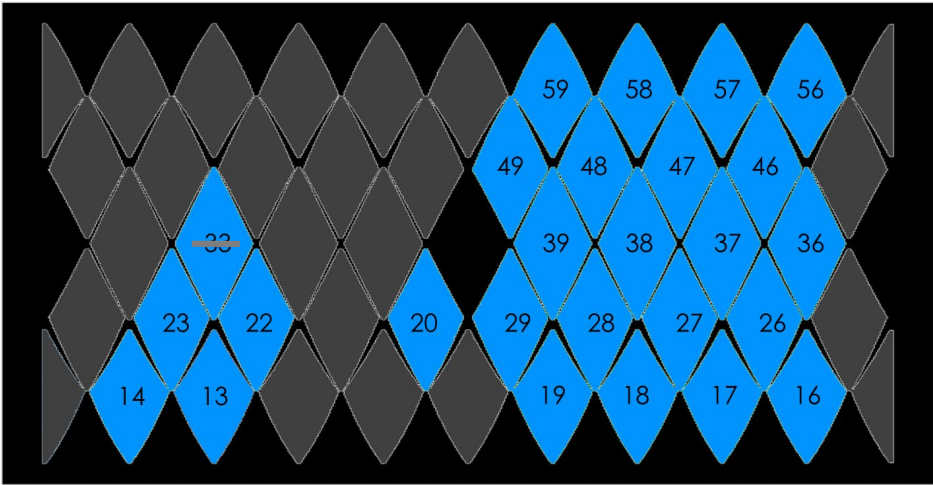


Detectors	Pixels / Det	Total Pixels
25	256 x256	1.6384 Million
Meas. Time	Pulse Rate	File Size
One Pulse	@ 60 Hz	26.7 GB
1 Sec.	@ 60 Hz	1.60 TB
1 Sec. meas.	121473 n	0.97 MB
1 hour meas.	4.373E+8 n	3.5 GB

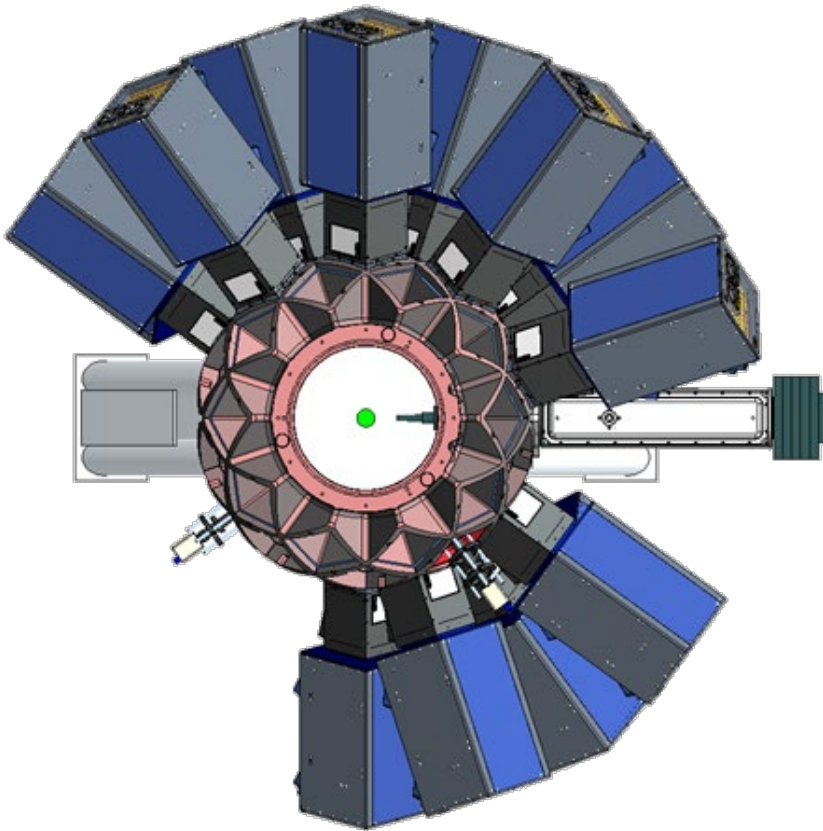
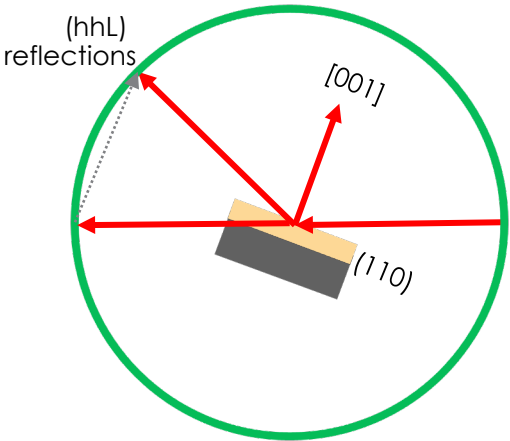
} Histogram data

} Event data

TOPAZ Detector Layout – 2026 A



25 detector installed



Det. No.	Theta, °	Chi, °	L2, cm
13	108.0	32.0	45.5
14	144.0	32.0	45.5
16	-144.0	32.0	45.5
17	-108.0	32.0	45.5
18	-72.0	32.0	45.5
19	-36.0	32.0	45.5
20	18.0	16.0	42.5
22	90.0	16.0	42.5
23	126.0	16.0	42.5
26	-126.0	16.0	42.5
27	-90.0	16.0	42.5
28	-54.0	16.0	42.5
29	-18.0	16.0	42.5
33	108.0	0.0	39.5
36	-144.0	0.0	39.5
37	-108.0	0.0	39.5
38	-72.0	0.0	39.5
39	-36.0	0.0	39.5
46	-126.0	-16.0	42.5
47	-90.0	-16.0	42.5
48	-54.0	-16.0	42.5
49	-18.0	-16.0	42.5
56	-144.0	-32.0	45.5
57	-108.0	-32.0	45.5
58	-72.0	-32.0	45.5
59	-36.0	-32.0	45.5

TOPAZ Sciences

✦ Chemical crystallography

- Structures of metal hydrides; Hydrogen bonding
- Discern the ordering and positions of neighboring elements

✦ Diffuse scattering

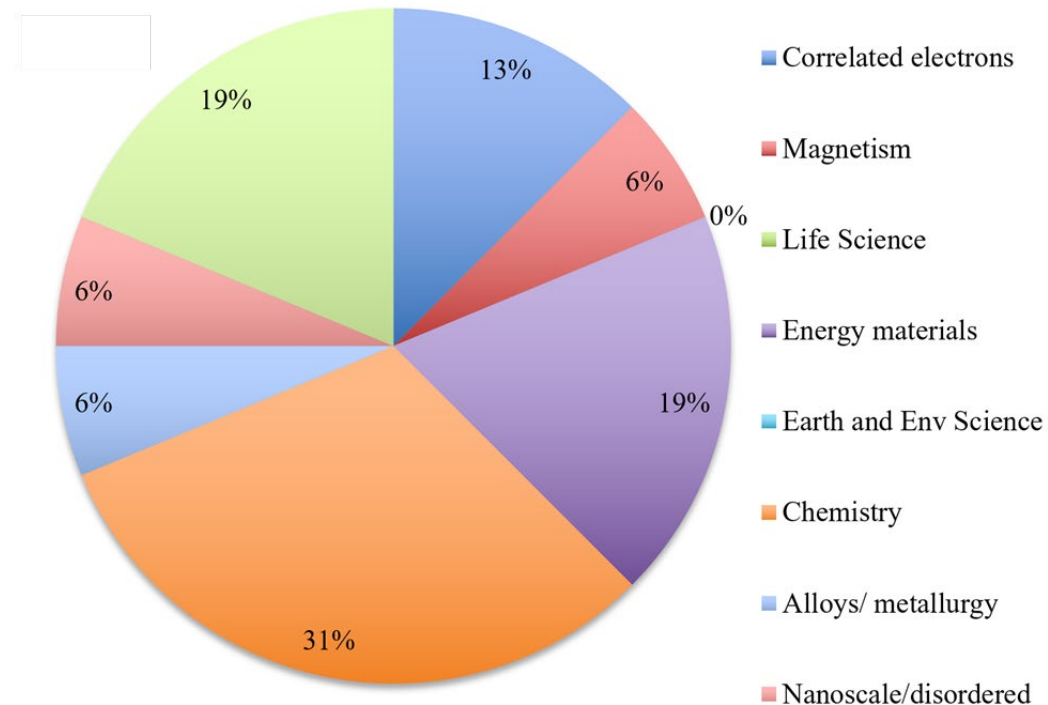
- 3D reciprocal space mapping

✦ Magnetism

- Solve & refine magnetic and nuclear structures

✦ Event based parametric study

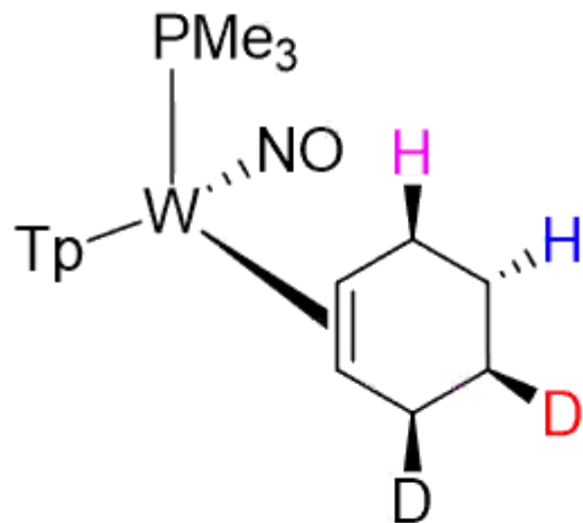
- Applied electric field
- Variable temperature
- Structural phase transitions
- Order parameters



Chemical crystallography

Transition metal-mediated dearomatization.

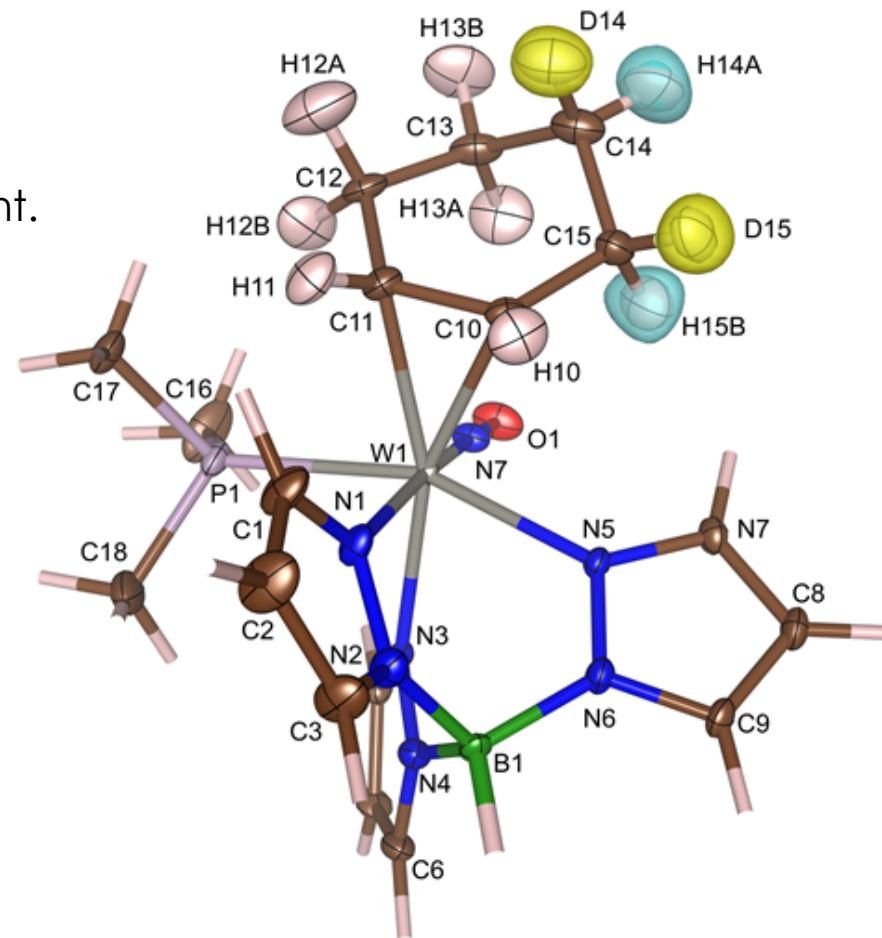
Opened pathways for a new generation of medicines and therapies that incorporate deuterium into the active pharmaceutical ingredient.



Isotope	Scattering lengths
Hydrogen ¹ H	-3.74 fm
Deuterium D (² H)	6.67 fm

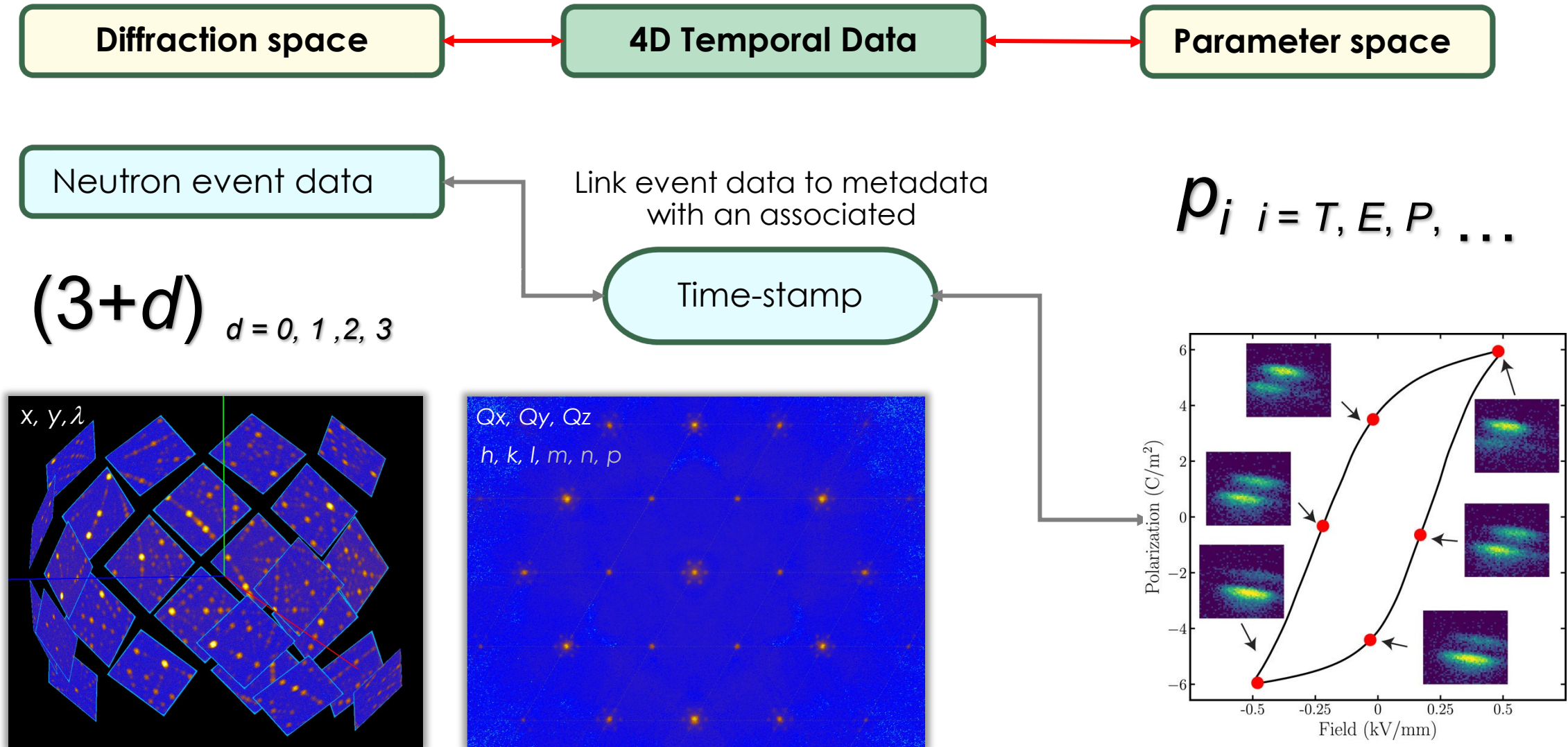


0.05 mm³



Neutron structure of a d₂ isotopologues of cyclohexene complex.

Single Crystal Neutron Diffraction Beyond Three Dimensions



Single crystal neutron diffraction beyond three dimensions

Modulated crystal structure

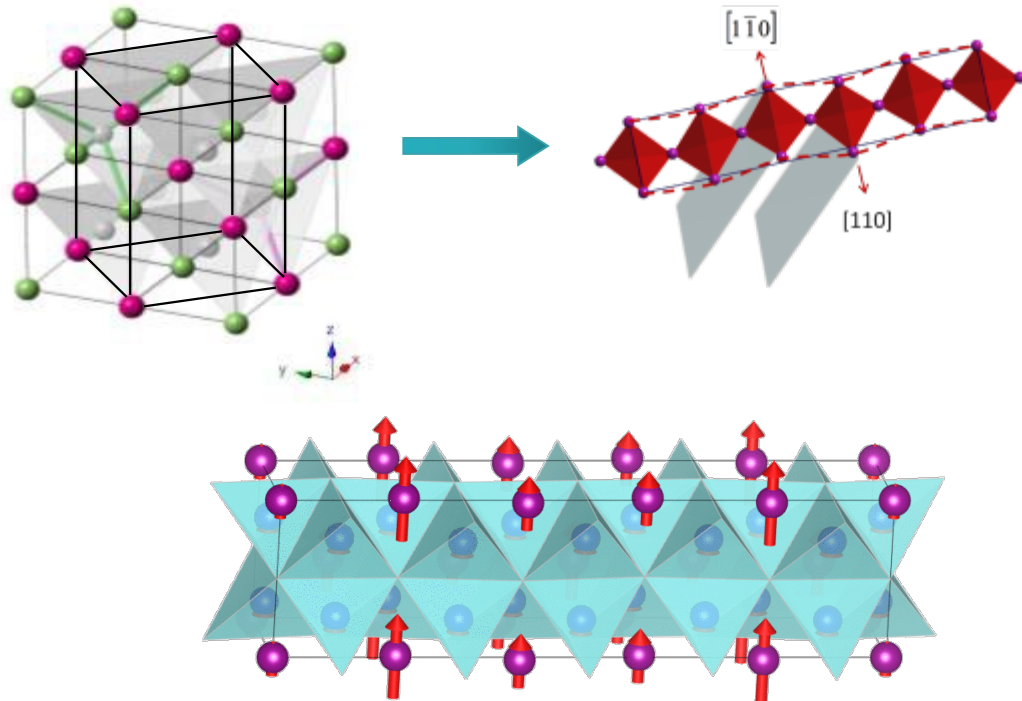
$$\mathbf{Q} = 2\pi(h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^* + m\mathbf{q}_1 + n\mathbf{q}_2 + p\mathbf{q}_3)$$



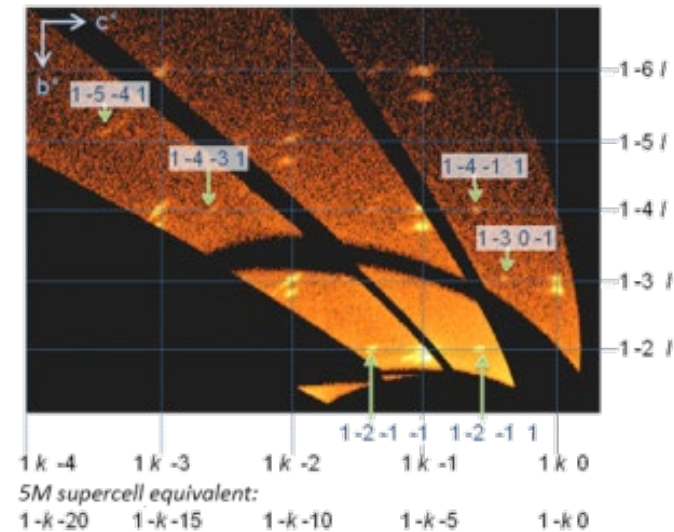
<https://docs.mantidproject.org/nightly/concepts/ModulatedStructure.html>

Local order and magnetic structural phase transition

- 🔧 Data measured on TOPAZ are used to describe simultaneous structural and magnetic modulations of $\text{Ni}_2\text{Mn}_{1.16}\text{Ga}_{0.84}$, a Magnetic Shape Memory Alloy.
- 🔧 Both nuclear and magnetic structures can be refined in JANA2006



JANA2006 / 5M $P2/m(a0r)00$



Single crystal neutron diffraction beyond three dimensions

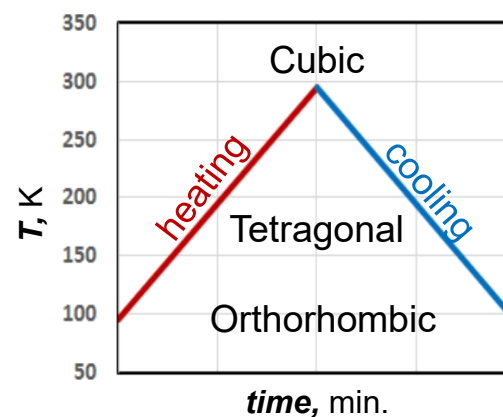
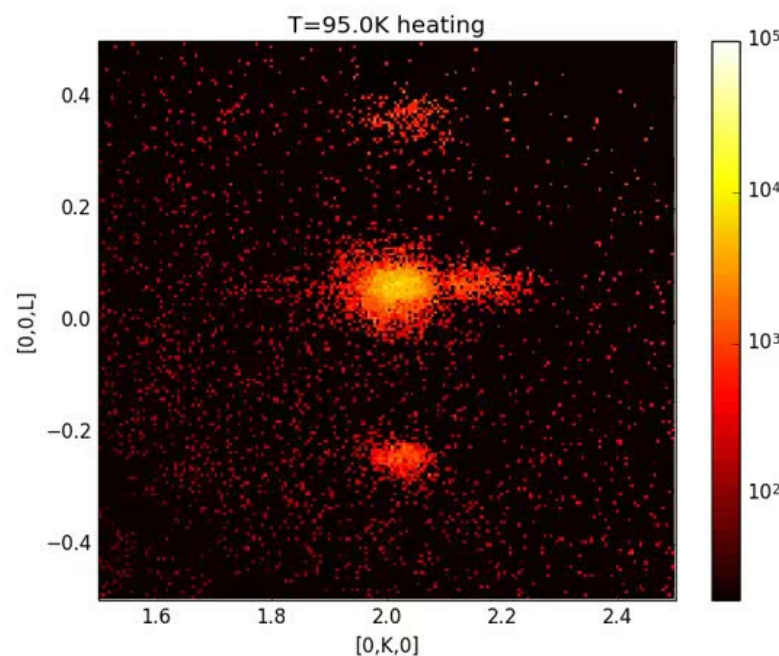
Modulated crystal structure

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

Multidimensional crystallography in *parameter space*

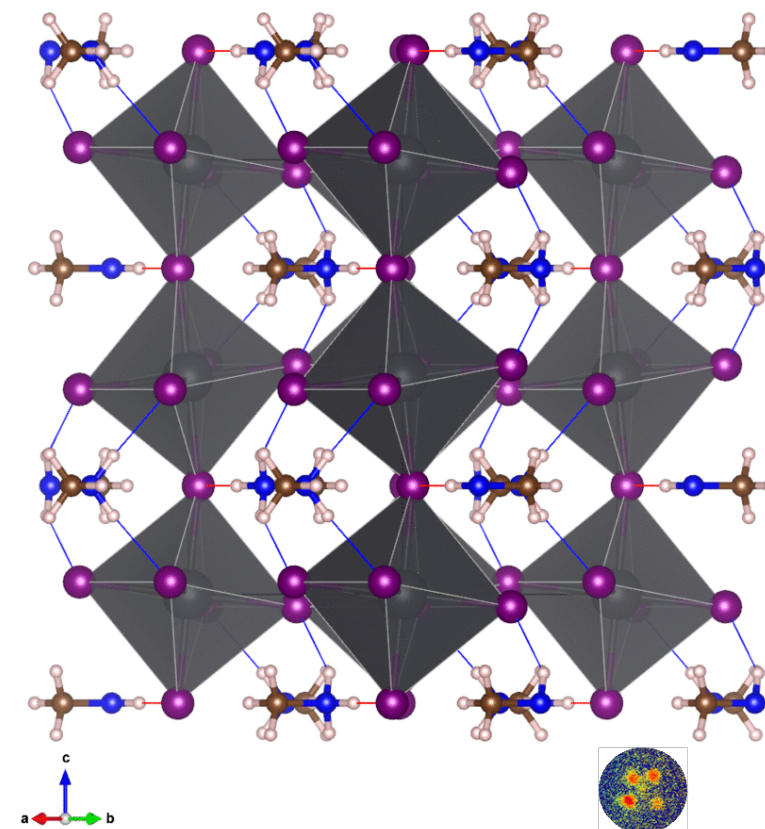
$$\mathbf{Q} + T, E, P, B$$

Reconstruction and visualization of real-time data



Ramping rate 1K per min.

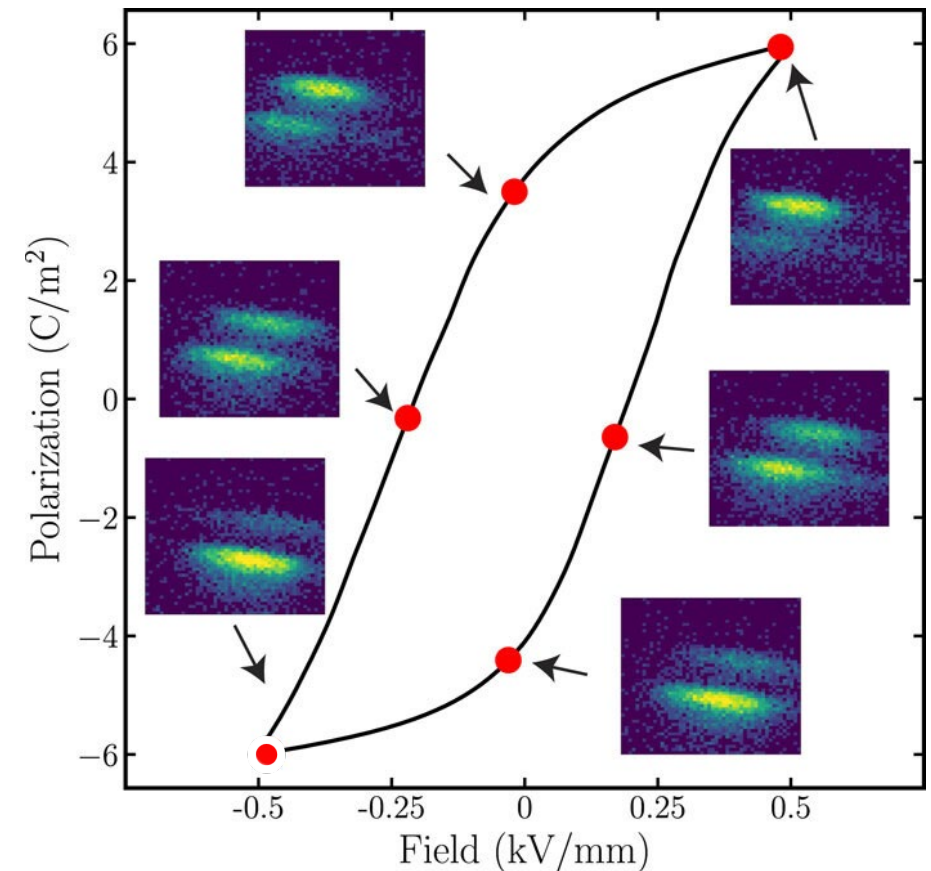
Temperature dependence of MAPbBr_3 (2 0 0)_C peak



Ferroelastic domain switching in KDP

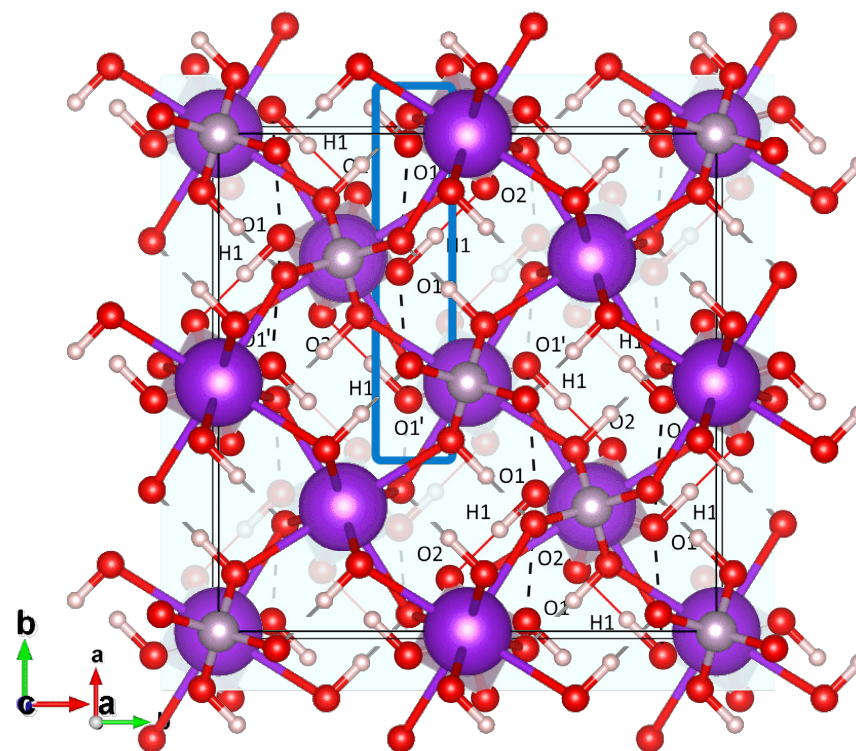
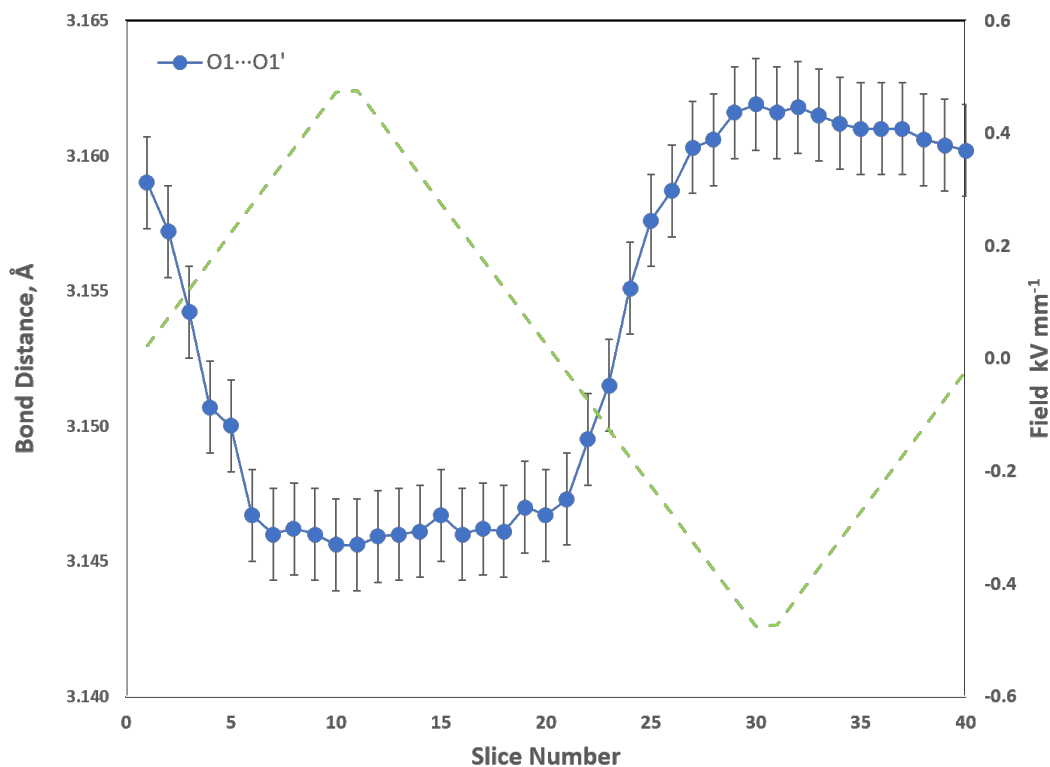
Simultaneous measurement of both current and diffraction data

- Split peaks at 95 K $\rightarrow$ Twin domains
- Inverted intensities for E-field at ± 0.5 kV/mm
 - Positive states $\rightarrow$ 171 peak
 - Negative states $\rightarrow$ 711 peaks
- Change polarization includes a lattice rotation
- Ferroelastic effects are driven through a 180° DWM



Changes in the bond distances at the polarization switch

O1...O1'



Outline

✚ The aim of the TOPAZ experiment is to use the high-resolution TOPAZ data to locate water hydrogen in the *nat* framework channels of scolecite.

✚ Introduction to TOPAZ and TOF Laue Diffraction

✚ Reduce one run using Mantid SCD Interfaces

✚ Reduce TOPAZ data using Reduction GUI

✚ **Structure Analysis**

The structure will be refined using the SHELXL program interfaced with ShelXle.

Locate missing hydrogen atoms from difference Fourier map

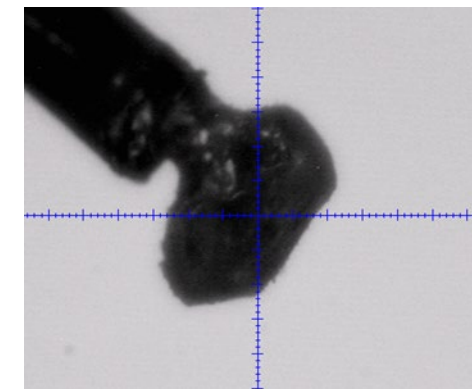
✚ Option to refine neutron structure using GSAS, JANA2020 and Olex2

Experiment Details

Scolecite is a member of *nat* zeolites with a porous aluminosilicate framework analogous to natrolite but a different placement of the calcium cation and water molecules in the zeolite channels.

A block-shaped crystal with the dimension of $0.62 \times 1.19 \times 1.63 \text{ mm}^3$ was cut from a prismatic scolecite crystal originated from India, and mounted with cyanoacrylate glue on the tip of a polyamide capillary to minimize the scattering from the mount. The initial orientation matrix was obtained in mantid at 100 K. Using this orientation matrix, an optimized data collection strategy was calculated in CrystalPlan [1]. Data were collected using 39 crystal orientations for an estimated 99.7% coverage of symmetry-equivalent reflections to $d_{\min}$ of $0.5 \text{ \AA}$. An estimated 90% of the measured reflections would be measured more than once. Each orientation was measured for approximately 1.5 h.

[1] Zikovsky J., Peterson P. F., Wang X. P., Frost M., Hoffmann C., "CrystalPlan: an experiment-planning tool for crystallography", *J. Appl. Cryst.* **44**, 418-423 (2011)



1.63 x 1.19 x 0.62 mm³

Scolecite	$\text{CaAl}_2\text{Si}_3\text{O}_{10} \cdot 3\text{H}_2\text{O}$
Temperature	100(2) K
Radiation source	TOF neutron
wavelengths	0.4 – 3.2 Å
Crystal system	Monoclinic
Space group	Cc
a, Å	6.5120(1)
b, Å	18.9545(4)
c, Å	9.7582(2)
β, °	108.921(1)
V, Å³	1139.39(4)
Z	4

Data

Single crystal data are saved in /SNS/TOPAZ/IPTS-33715/data

- Event NeXus

TOPAZ_#####_event.nxs

- Histogrammed NeXus

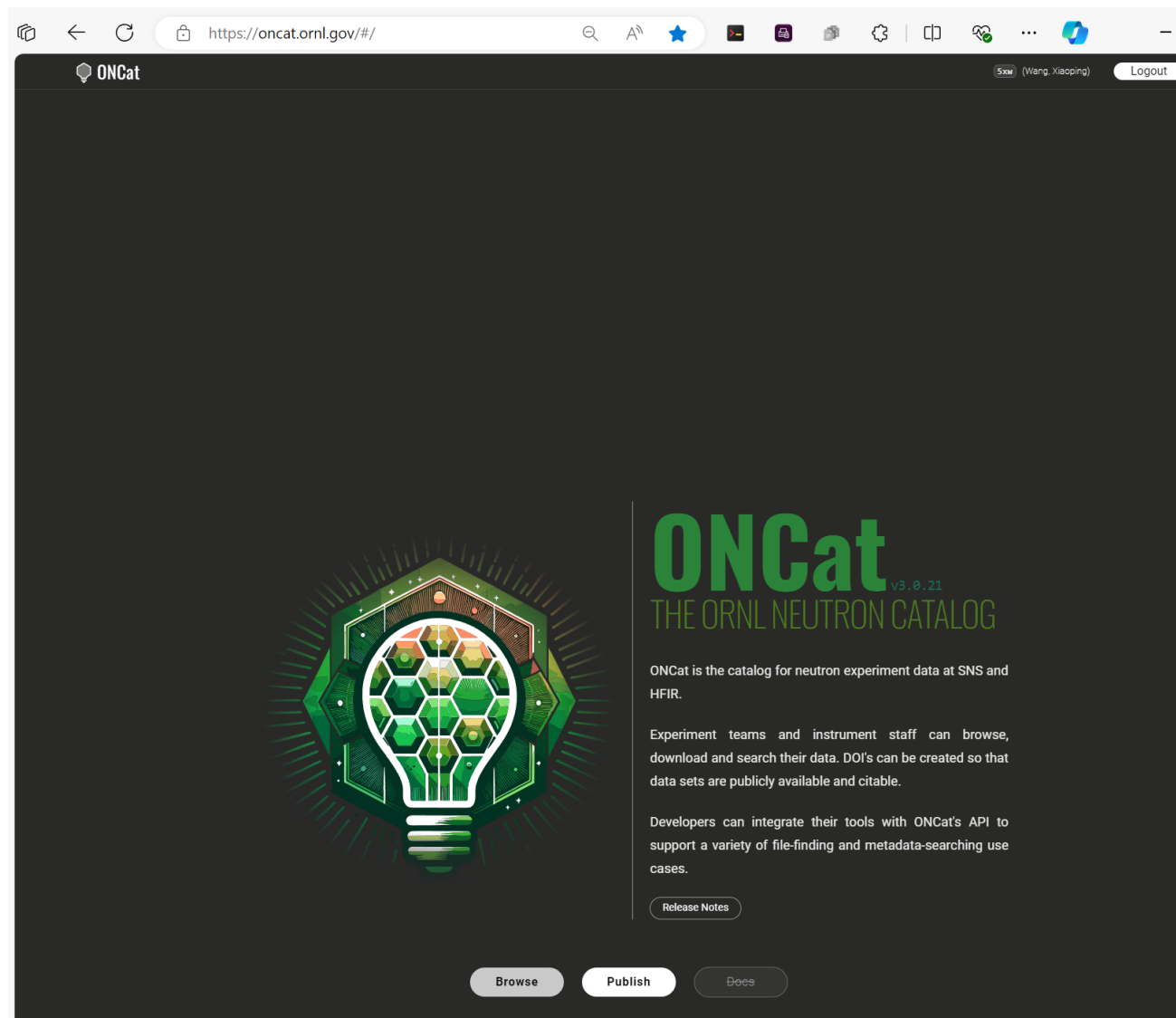
TOPAZ_#####_histo.nxs

100 K Data Set

- Run numbers 7450:7488

295 K Data Set

- Run numbers 10074:10079,10081:10084



Experiment Log and Data storage

- Experiment information is available online

Login with your user id and password:

<https://oncat.ornl.gov>

- NeXus file format

- A common data exchange format for neutron, X-ray, and muon experiments.

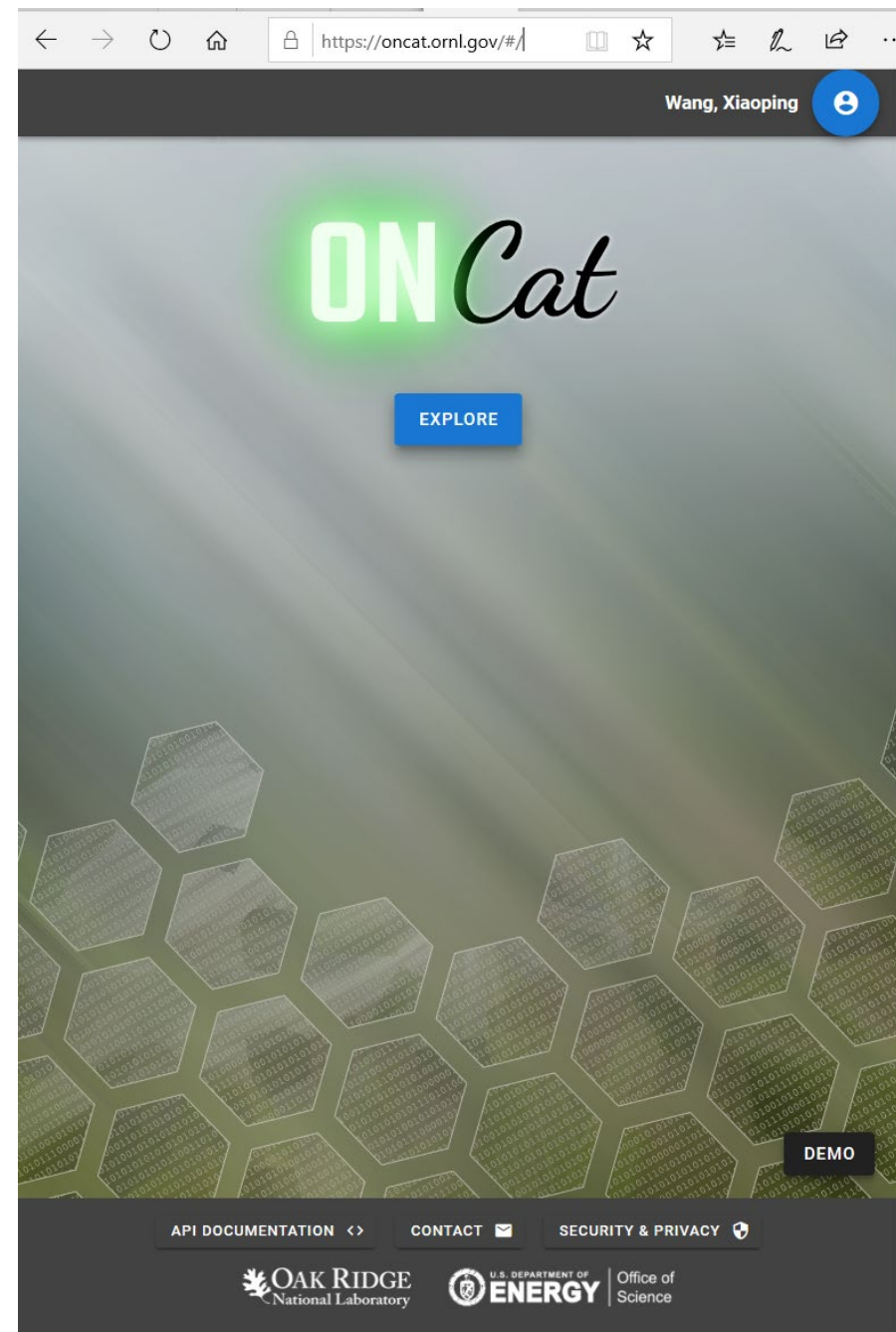
HDF5 format with domain-specific field names

Can be used to store raw data and processed data

- SNS data are saved in event NeXus mode

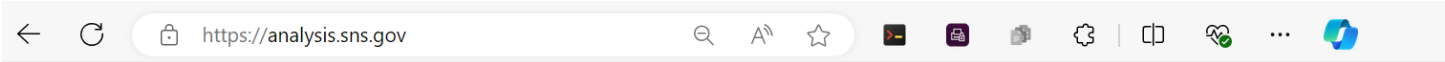
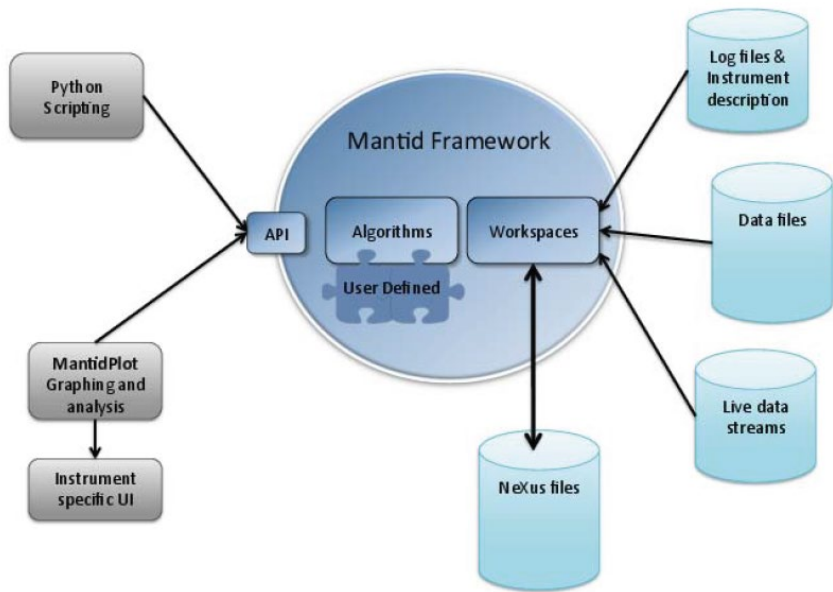
- All experiment data are saved on a data server at a remote location, and available online at

<https://analysis.sns.gov>



Data Reduction

<https://analysis.sns.gov>



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

Remote Desktop Analysis

Neutron Science users can view, analyze, and download their data from anywhere. Accessing a virtual session, you will be able to work on a computer just like those used in our Instrument Hall or Target Building. You will have access to your data as well as data analysis tools. To get started, click the "Launch Session" button below.

For assistance connecting to the analysis servers or accessing your data, please contact Linux Support at linux@support.sns.gov or call 865-309-4649 for urgent requests.

Launch Session

Single Crystal Data Reduction Using Mantidworkbench

- 👉 Logon to `userid@analysis.sns.gov`
- 👉 Open a terminal window
[aka command line window \$]

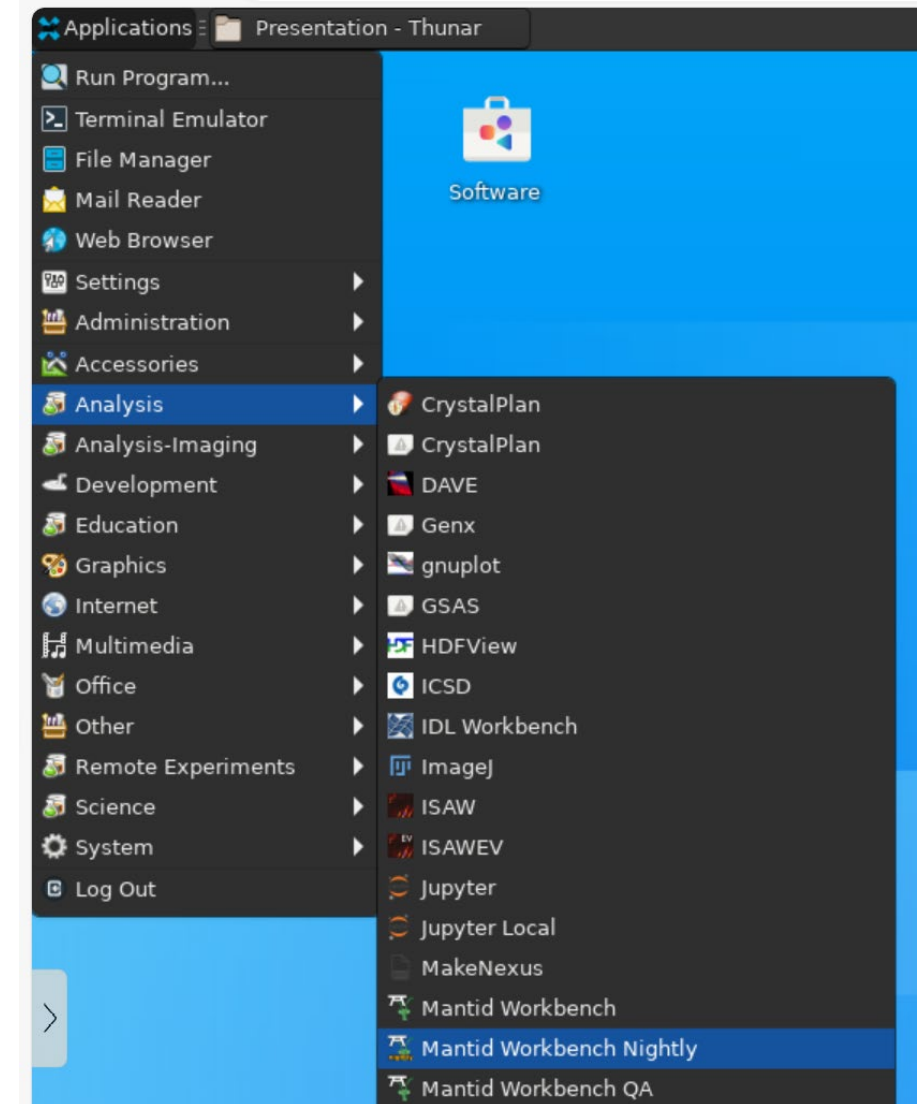
Reduce one run using MantidWorkbench

<https://single-crystal.ornl.gov/guide/mantid/index.html#id10>

Reduce multiple runs using python script

- 👉 CD to reduction folder

`$cd /SNS/TOPAZ/IPTS-33715/shared/ReductionGUI`
- 👉 Run the mantid python script



Single Crystal Data Reduction Using NeuXtalViz

(1) Launch Remote Session on analysis computer

<https://analysis.sns.gov/>



Remote Desktop Analysis

Neutron Science users can view, analyze, and download their data from anywhere. Accessing a virtual session, you will be able to work on a computer just like those used in our Instrument Hall or Target Building. You will have access to your data as well as data analysis tools. To get started, click the "Launch Session" button below.

For assistance connecting to the analysis servers or accessing your data, please contact Linux Support at linux@support.sns.gov or call 865-309-4649 for urgent requests.

Launch Session



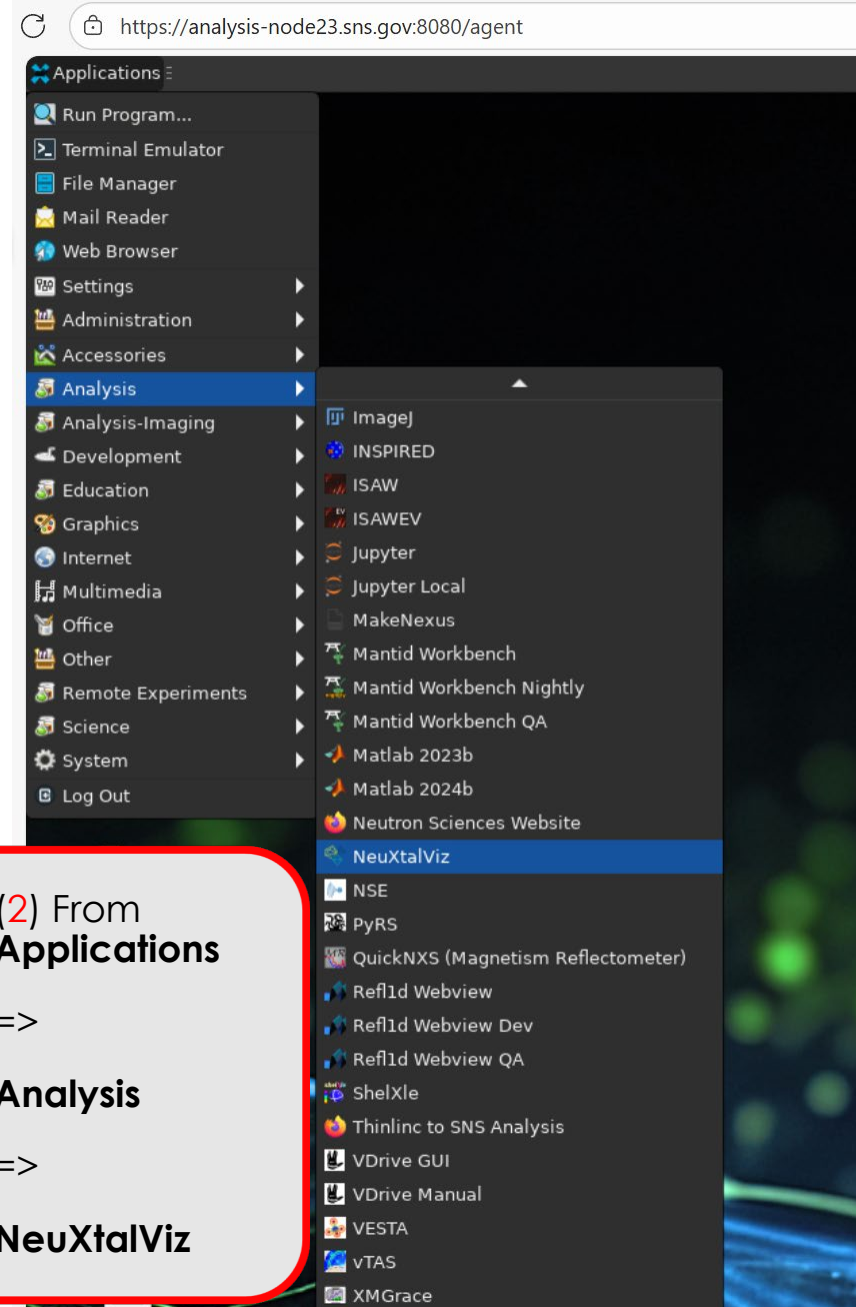
[Security Notice](#)

[ORNL](#)

[Account Resources](#)

[Connection Options](#)

[Troubleshooting](#)



(2) From Applications

=>

Analysis

=>

NeuXtalViz

Experiment Planning - NeuXtalViz tutorials

UB Matrix Determination

UB Tools with Silicon Data

- Step 1: Select Instrument and Data
- Step 2: Find Peaks
- Step 3: Primitive Cell Calculation
- Step 4: Select Conventional Cell
- Step 5: Refine UB Matrix
- Step 6: View Peak
- Step 7: Slice View
- Step 8: Save UB

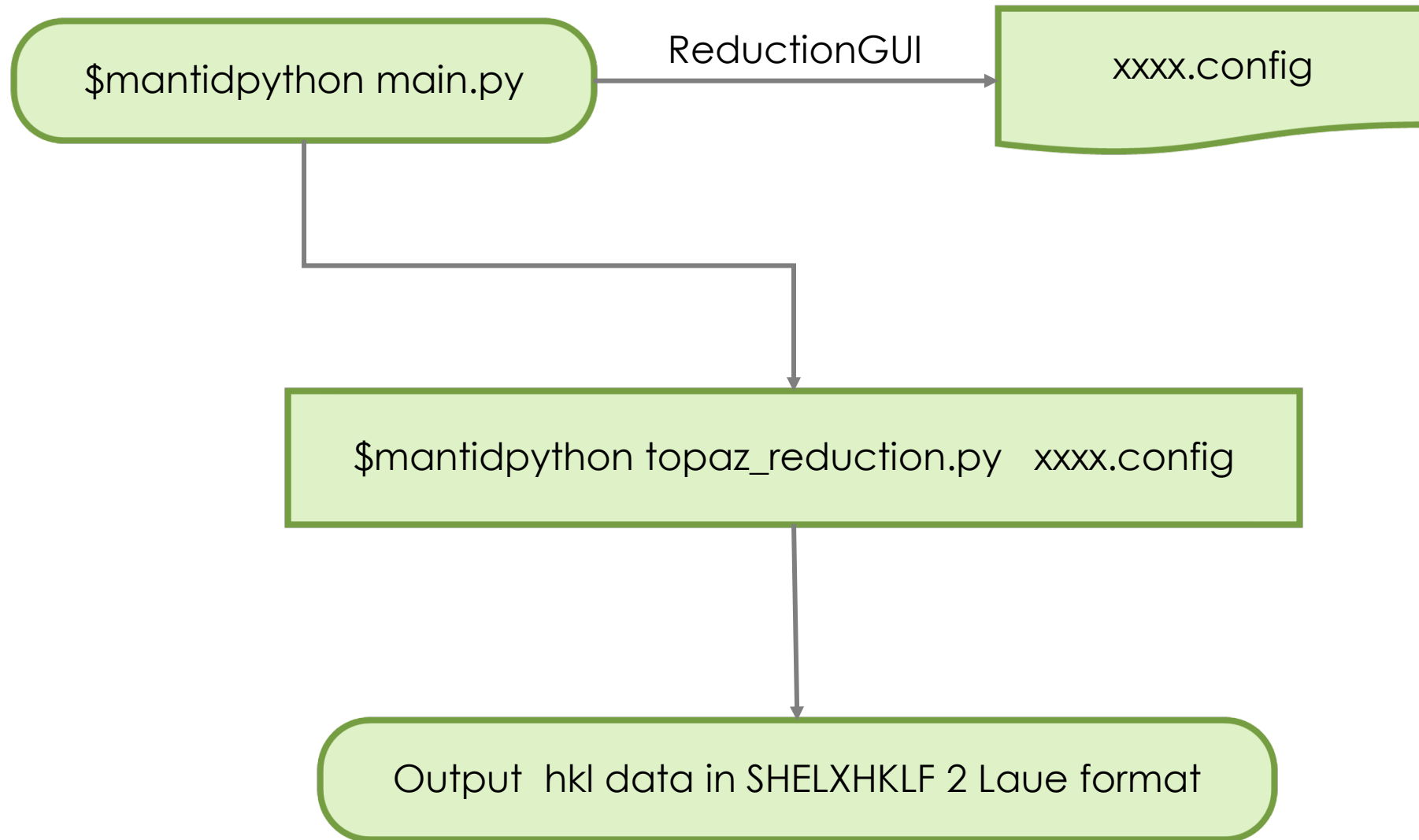
Generate Experiment Plan

Experiment Plan with Silicon Data

- Step 1: Initialize the Experiment Planner
- Step 2: Update Goniometer Angles
- Step 3: Calculate Peaks
- Step 4: Add Orientation
- Step 5: Optimize Coverage
- Step 6: Update and Review
- Step 7: Save and Export

<https://zjmorgan.github.io/NeuXtalViz-tools/source/NeuXtalViz.tutorials.html>

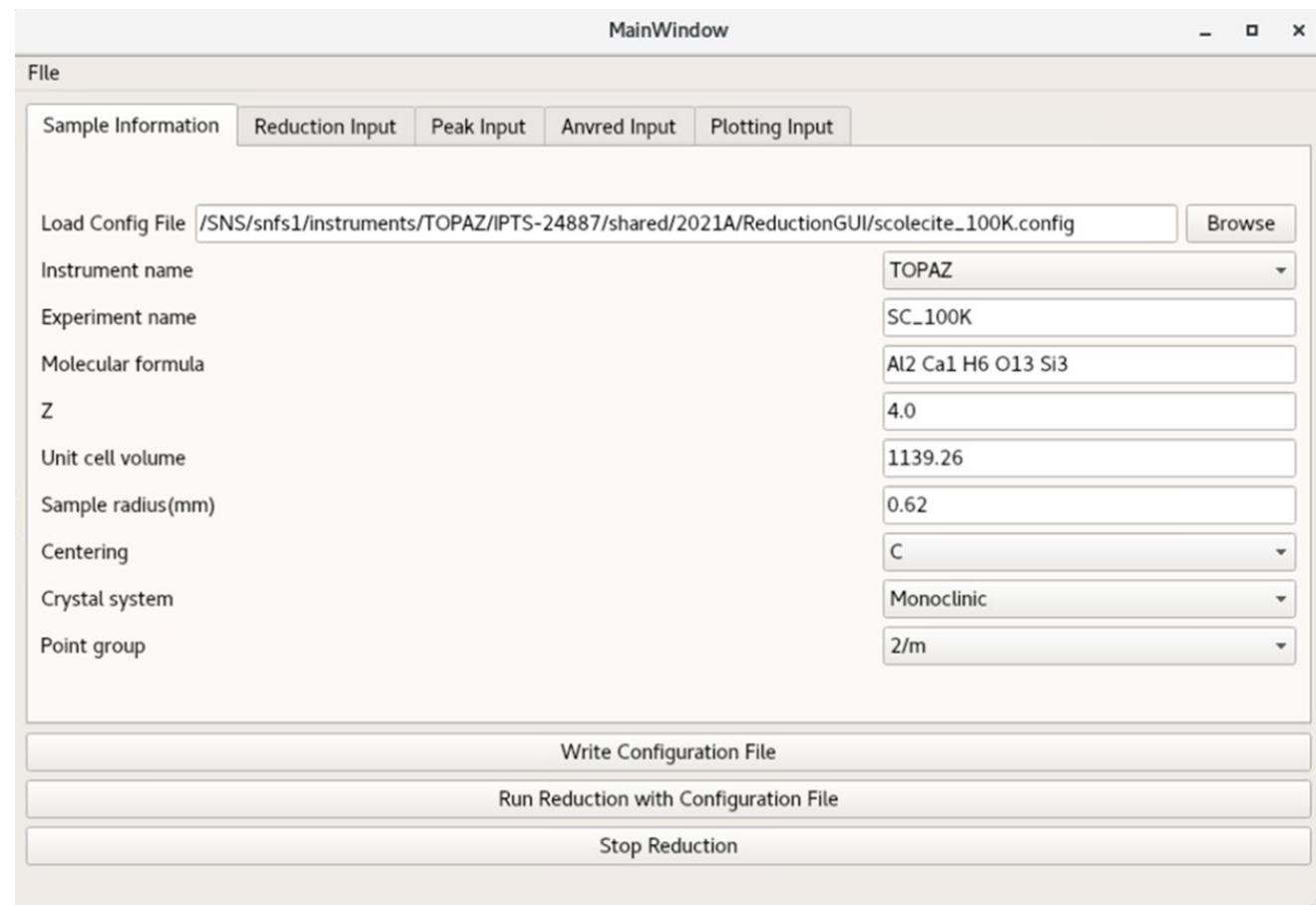
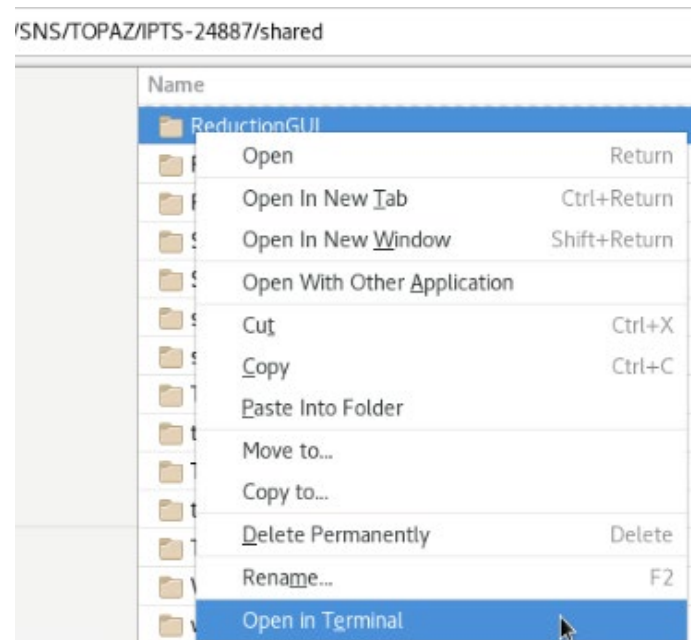
TOPAZ Data Reduction



TOPAZ Data Reduction GUI

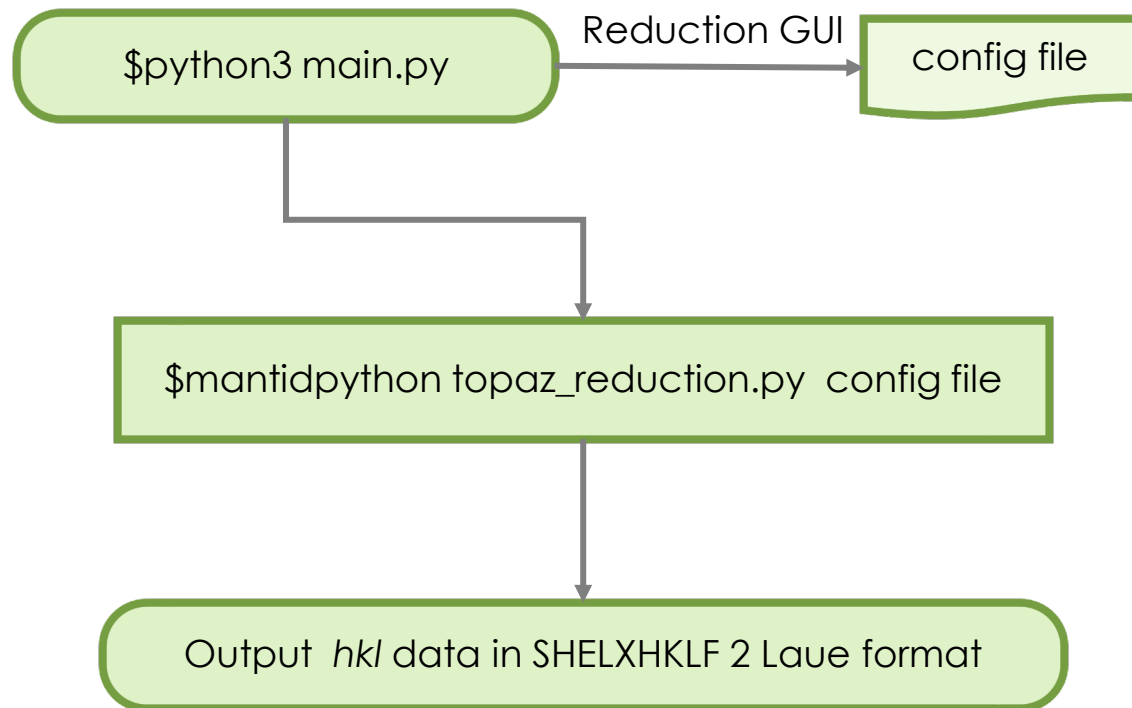
- ✎ The data reduction GUI will generate the configure file for TOPAZ data reduction

Open in Terminal window and run `$python3 main.py`



TOPAZ Data Reduction

Flowchart



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

Result

Crystal symmetry

Point Group: 2/m
Lattice System: Monoclinic
Lattice Centering: C-face centred

Peak Statistics

Number of Peaks: 31805
Multiplicity: 3.85
Data Completeness: 86.4%
Resolution Min: 0.5
Resolution Max: 9.47
No. of Unique Reflections: 8265
Mean ((I)/sd(I)): 13.75
Rmerge: 7.6%
Rpim: 4.07%

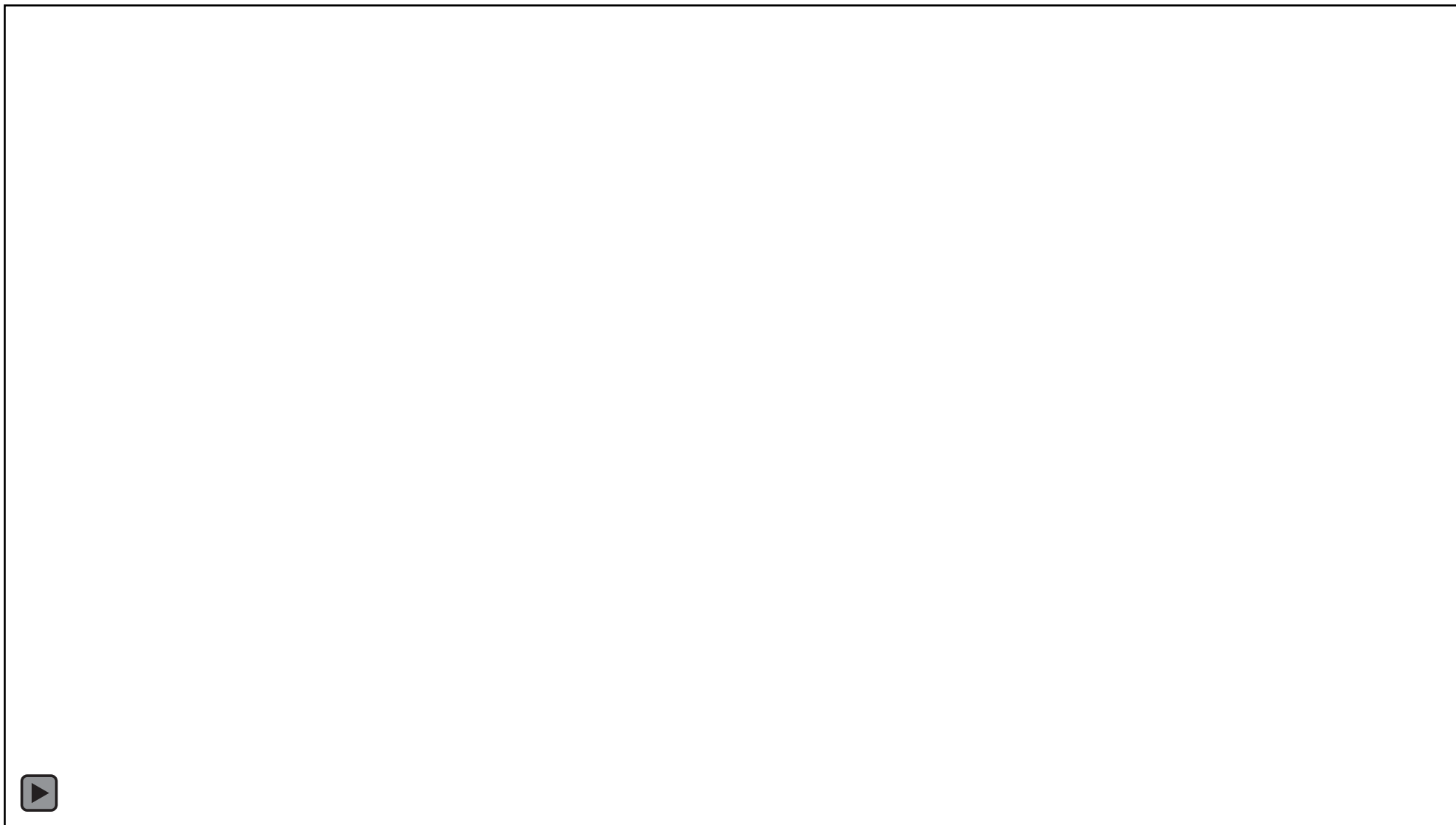
Number of peaks after outlier removal: 31805

Saving result ...

/SNS/TOPAZ/IPTS-24887/shared/SC_100K/SC100K_Monoclinic_C.hkl
/SNS/TOPAZ/IPTS-24887/shared/SC_100K/SC100K_Monoclinic_C_symm.hkl

```
*****  
***** All DONE *****  
*****
```

TOPAZ Data reduction demo



Data Reduction and Structure Analysis Programs on NDIP

Structure Analysis Programs

Crystal/Nuclear Structure

🔧 SHELX

🔧 Olex2

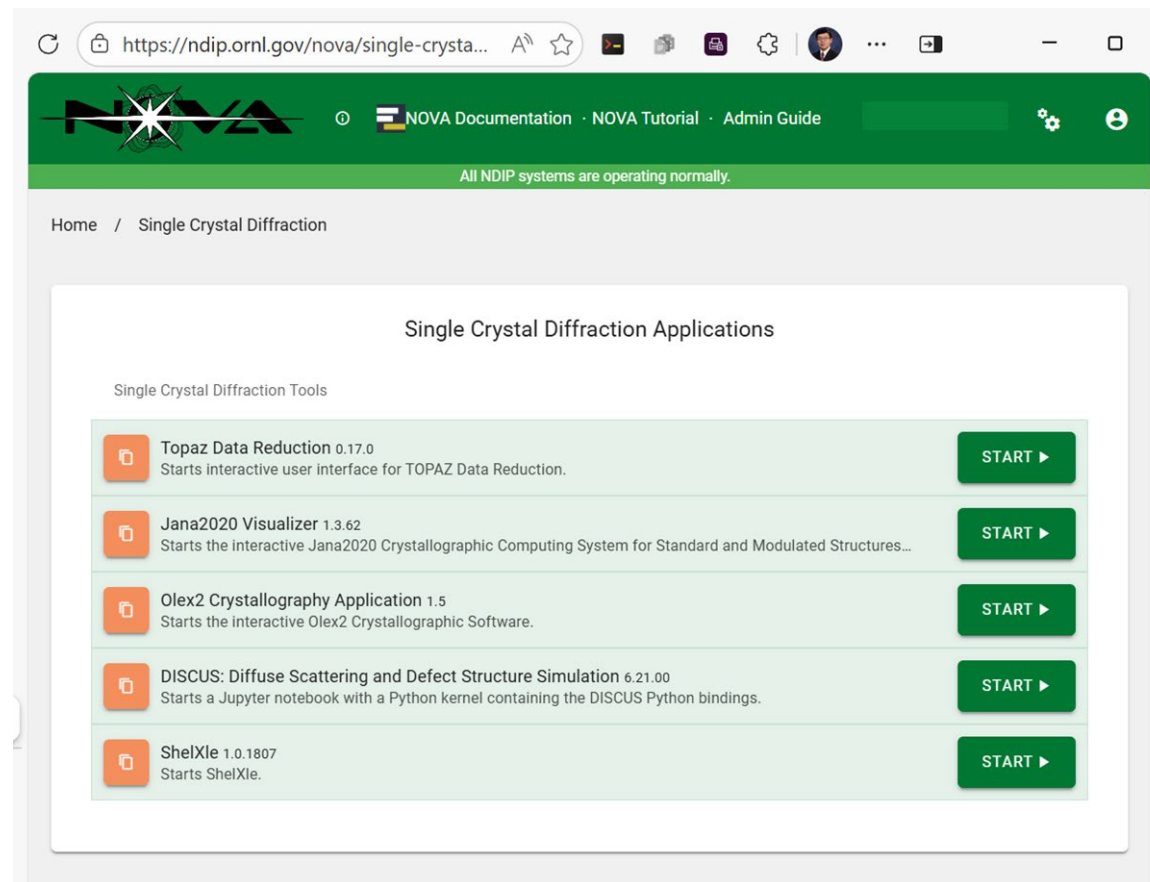
Nuclear /Magnetic Structure

🔧 JANA2020

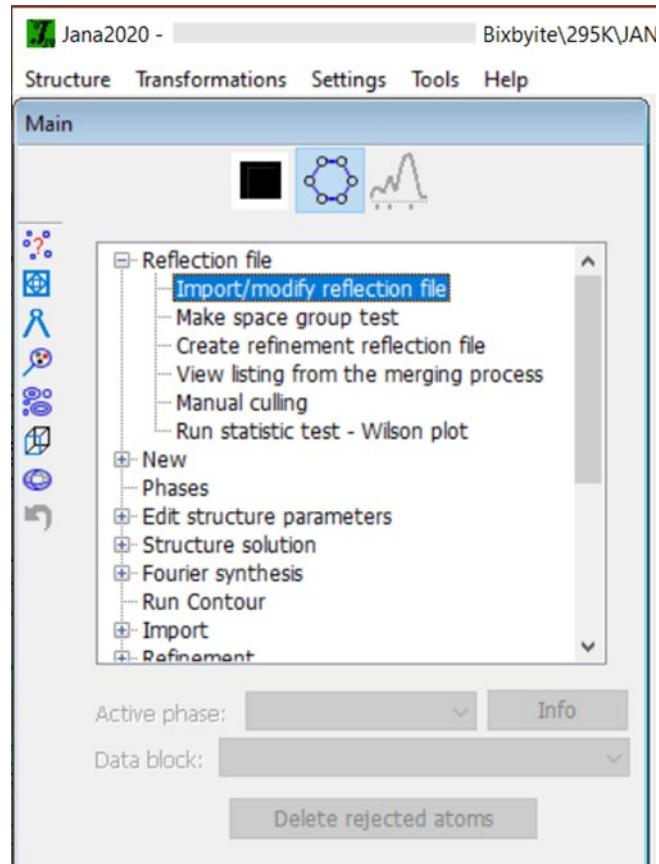
Diffuse Scattering

- DISCUS

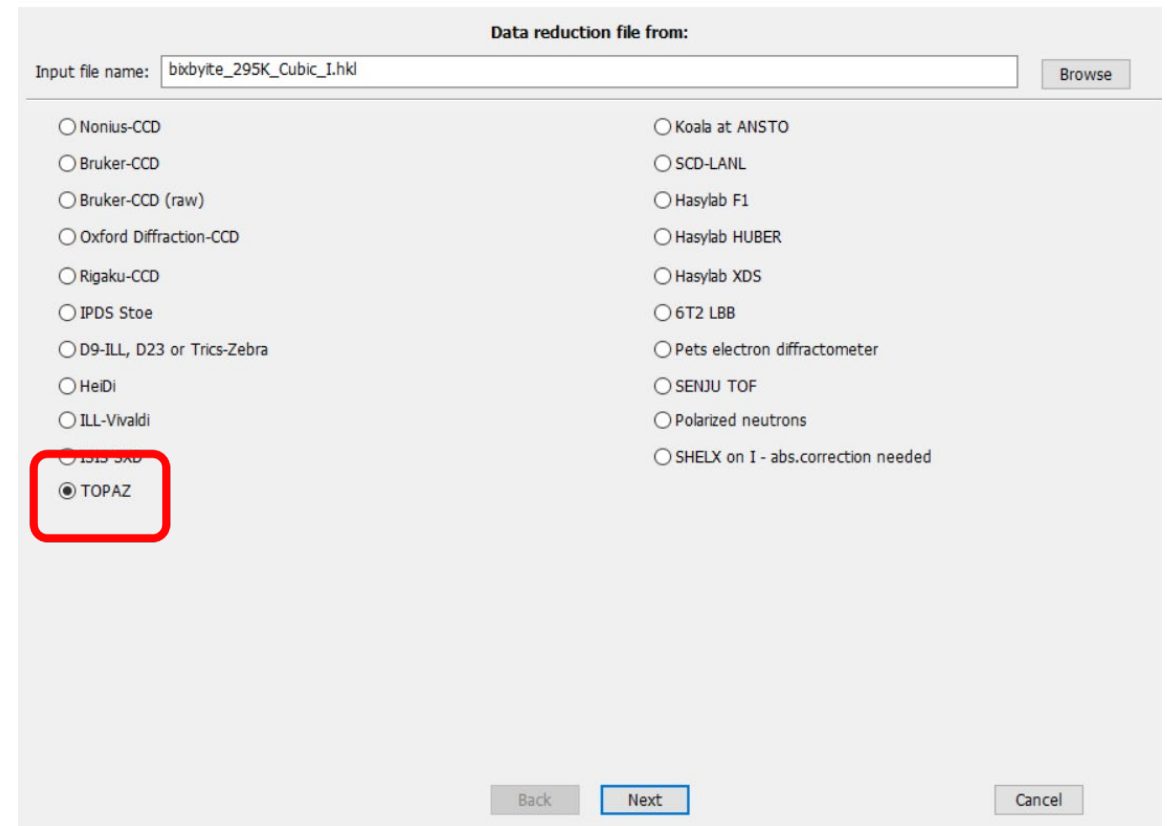
<https://ndip.ornl.gov/nova/single-crystal-diffraction>



Import TOPAZ data



[Link to bixbyte tutorial example](#)



TOPAZ Data Reduction Program

There are three Python scripts for TOPAZ data reduction

topaz_reduction.py; *topaz_reduction_combinefiles.py*; and *topaz_reduction_anvred.py*

The python script for reducing multiple runs from scratch is *topaz_reduction.py*. The reduction script needs to run from the IPTS-xxxxx **./ReductionGUI** subfolder:

```
$mantidpython topaz_reduction.py xxxx.config
```

The reduction will perform all corrections, including tof spectrum, Lorentz, and absorption corrections

The reduction script generates two hkl files, the one label *_symm.hkl* has outliers removed based on the Z-scores specified

The python script *topaz_combinefiles.py* is for recombining individual *.integrate* files:

```
$mantidpython topaz_reduction_combinefiles.py xxxx.config
```

The python script *topaz_anvred.py* is for absorption correction if one wants to modify the sample radius, or change the absorption type from spherical to polyhedral:

```
$mantidpython topaz_reduction_anvred.py xxxx.config
```

Workshop Tutorial Single Crystal Neutron Diffraction Data Reduction and Analysis (ORNL 2024)

HKLF 2 Laue data format

✚ **HKLF 2** ! + batch number (BN) and wavelength λ for individual reflections

✚ **Data Format** 3I4, 2F8.2,I4,F8.4

✚ 1234123412341234567812345678123412345678

✚ h k l Fo^2 $\sigma(Fo^2)$ 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

Data Reduction and Normalization

- ✦ Convert raw integrated intensities, I_{hkl} , into relative structure factor amplitudes, F_{hkl}^2 .

$$I_{hkl} = \phi(\lambda)\varepsilon(\lambda, \vec{r})A(\lambda)y(\lambda) \frac{V_s}{V_c} \frac{|F_{hkl}|^2}{V_c} \frac{\lambda^4}{2 \sin^2 \theta}$$

$f(l)$ = incident flux spectrum, obtained by measuring the incoherent scattering from a vanadium sample

$e(l, \mathbf{r})$ = detector efficiency calculated as a function of wavelength l and position $\mathbf{r}$ on the detector for each Bragg peak since the slant path through the flat ^6Li glass varies with $\mathbf{r}$

$A(l)$ = sample absorption; includes the wavelength dependence of the linear absorption coefficients

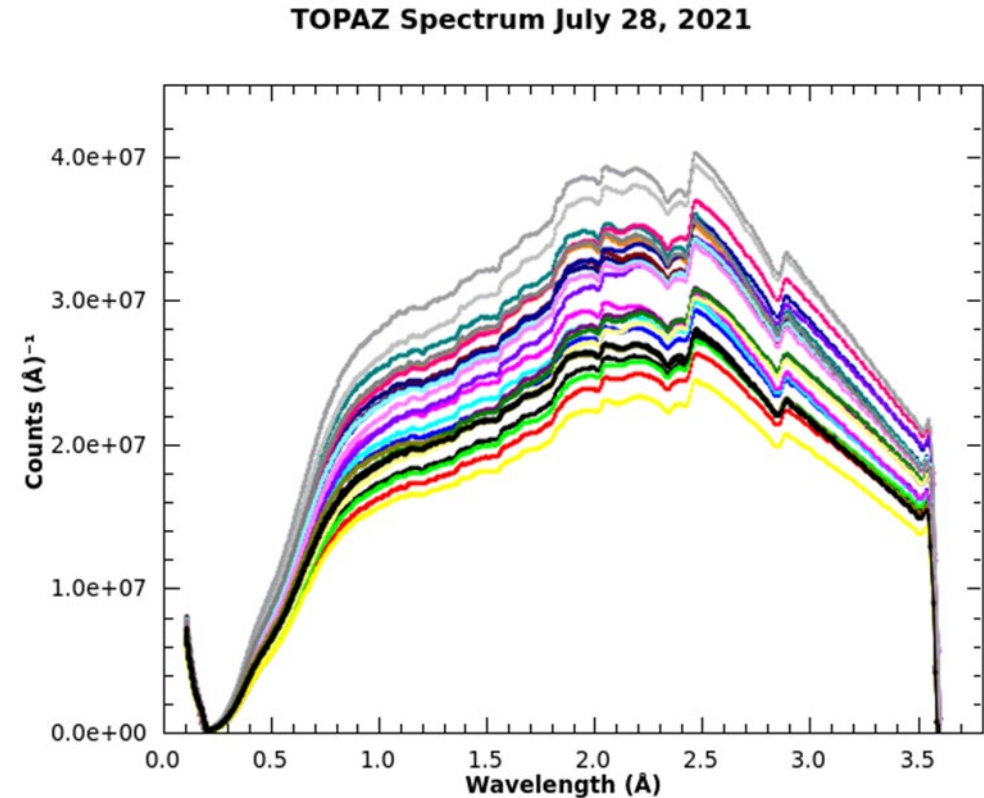
$y(l)$ = extinction correction is evaluated during the least-squares refinement of the structure

V_s = sample volume

V_c = unit cell volume

TOPAZ Spectrum correction

- ✚ Divide the TOF range into several hundred channels.
- ✚ Sum the the counts over the entire detector face for vanadium and for background.
- ✚ Subtract background from vanadium.
- ✚ Corrected for vanadium absorption



HKLF 2 Instruction for Laue data

✚ **HKLF 2** ! + batch number (BN) and wavelength λ for individual reflections

✚ **Data Format** 3I4, 2F8.2,I4,F8.4

✚ 1234123412341234567812345678123412345678

✚ h k l Fo^2 $\sigma(Fo^2)$ 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

✚ **TWIN** instruction cannot be used for refinement against data in **HKLF 2** format

- ** ILLEGAL TWIN/HKLF COMBINATION **

SHELXL instructions for neutron data

name.ins

CELL 0.600 6.5120 18.9545 9.7582 90.0 108.921 90.0

Nominal wavelength

ZERR 4 0.0001 0.0004 0.002 0.0 0.001 0.00

...

NEUT ! Placed before the first SFAC instruction with element names

SFAC Ca Si Al O H

ACTA

FMAP -2

...

...

EXTI

FVAR

....

HKLF 2

data from Laue source

TOPAZ Data Format SHELX HKLF2 Laue + TOF

```
hklFile.write( 3*'%4d' % (h, k, l)
+ 2*'%8.2f' % (fsq, sigfsq)
+ '%4d' % hstnum
+ 2*'%8.5f' % (wl, tbar)
+ 6*'%9.5f' % (dir_cos_1[0], dir_cos_2[0], dir_cos_1[1], dir_cos_2[1], dir_cos_1[2], dir_cos_2[2])
+ '%6d' % (curhst)
+ '%7d' % (seqnum)
+ '%7.4f%4d%9.5f%8.4f%7.2f%7.2f\n' % (transmission, dn, twoth, dsp, col, row) )
```

Where

h, k, l	peak indices	curhst:	the actual run number
fsq	peak intensity	seqnum:	a number from 1 to the number of peaks
sigfsq	sigma	transmission:	the transmission after absorption
hstnum:	a number from 1 to the number of crystal settings	dn:	the detector number
wl:	neutron wavelength	twoth:	2-theta angle
tbar:	the absorption weighted path length	dsp:	d-spacing
dir_cos_1:	the three direction cosines of a*, b* and c* with the reverse incident beam	col:	detector column pixel number
dir_cos_2:	the three direction cosines of a*, b* and c* with the scattered beam	row:	detector row number

Spherical Absorption Correction

- Applied to correct measured spectrum using the V/Nb sphere.
- Applied to correct individual hkl peaks from the sample.
- Spherical absorption correction A^* obtained from table of μR vs. θ .
- C. W. Dwiggin, Jr., Acta Cryst. A31, 395 (1975).
- For each theta, fit polynomial to the μR dependence. Interpolate between θ values.

Table 1. Values of A^* for spheres

μR	$\theta=0^\circ$	$\theta=5^\circ$	$\theta=10^\circ$	$\theta=15^\circ$	$\theta=20^\circ$	$\theta=25^\circ$	$\theta=30^\circ$	$\theta=35^\circ$	$\theta=40^\circ$	$\theta=45^\circ$
0.0	1	1	1	1	1	1	1	1	1	1
0.1	1.1609	1.1609	1.1609	1.1607	1.1606	1.1603	1.1600	1.1597	1.1593	1.1589
0.2	1.3457	1.3456	1.3452	1.3447	1.3439	1.3428	1.3415	1.3400	1.3383	1.3366
0.3	1.5574	1.5571	1.5561	1.5546	1.5525	1.5497	1.5463	1.5426	1.5383	1.5339
0.4	1.7994	1.7988	1.7968	1.7935	1.7891	1.7833	1.7765	1.7689	1.7604	1.7515
0.5	2.0755	2.0743	2.0706	2.0647	2.0565	2.0462	2.0340	2.0204	2.0056	1.9901
0.6	2.3897	2.3877	2.3816	2.3715	2.3578	2.3406	2.3206	2.2984	2.2746	2.2500
0.7	2.7467	2.7434	2.7336	2.7177	2.6959	2.6691	2.6382	2.6042	2.5683	2.5316
0.8	3.1511	3.1461	3.1312	3.1069	3.0740	3.0339	2.9882	2.9386	2.8869	2.8347
0.9	3.6082	3.6009	3.5789	3.5431	3.4952	3.4374	3.3723	3.3026	3.2308	3.1592
1.0	4.1237	4.1131	4.0815	4.0304	3.9625	3.8816	3.7917	3.6966	3.6001	3.5048
1.1	4.7035	4.6886	4.6442	4.5729	4.4790	4.3686	4.2474	4.1211	3.9945	3.8710
1.2	5.3542	5.3335	5.2722	5.1747	5.0476	4.9001	4.7404	4.5761	4.4137	4.2571
1.3	6.082	6.054	5.9710	5.8399	5.6710	5.4776	5.2711	5.0617	4.8573	4.6625
1.4	6.895	6.857	6.746	6.573	6.352	6.102	5.8400	5.5774	5.3244	5.0862
1.5	7.801	7.750	7.604	7.377	7.092	6.775	6.447	6.123	5.8143	5.5273
1.6	8.806	8.740	8.549	8.256	7.894	7.497	7.092	6.697	6.326	5.9849
1.7	9.920	9.834	9.587	9.214	8.759	8.268	7.774	7.299	6.859	6.458
1.8	11.151	11.040	10.725	10.254	9.689	9.088	8.492	7.928	7.411	6.946
1.9	12.507	12.366	11.967	11.380	10.685	9.957	9.246	8.583	7.982	7.447
2.0	13.998	13.819	13.320	12.593	11.746	10.873	10.034	9.262	8.570	7.961
2.1	15.632	15.408	14.788	13.895	12.874	11.837	10.855	9.964	9.175	8.486
2.2	17.419	17.141	16.376	15.290	14.067	12.847	11.708	10.688	9.795	9.023
2.3	19.369	19.025	18.089	16.778	15.327	13.902	12.592	11.433	10.429	9.569
2.4	21.489	21.069	19.931	18.361	16.652	15.000	13.504	12.198	11.077	10.125
2.5	23.791	23.280	21.907	20.040	18.041	16.142	14.445	12.982	11.738	10.690

Polyhedral Absorption Correction

```
C      Program      : GAUSSIAN      C
C      Source file   : gaussian.f    C
C      Description   : Gaussian absorption correction C
C      Author(s)     : K.W. MUIR (1985) mods L. J. Farrugia (2001) C
C      Last modified  : 09/09/2001   C
```

P. Coppens in *Crystallographic Computing*, Ed F.R. Ahmed, S.R. Hall and C.P. Huber, Copenhagen, Munksgaard, (1970) pp 255-270

```
TITL  Rh137T295K 295 K Tetragonal
CELL  5.4877  5.4877  25.7634  90.0  90.0  90.0
FACE   1  0  0  0.565
FACE  -1  0  0  0.565
FACE   0  1  0  0.45
FACE   0 -1  0  0.45
FACE   0  0  1  0.215
FACE   0  0 -1  0.215
MUAB  0.0432  0.3994
```

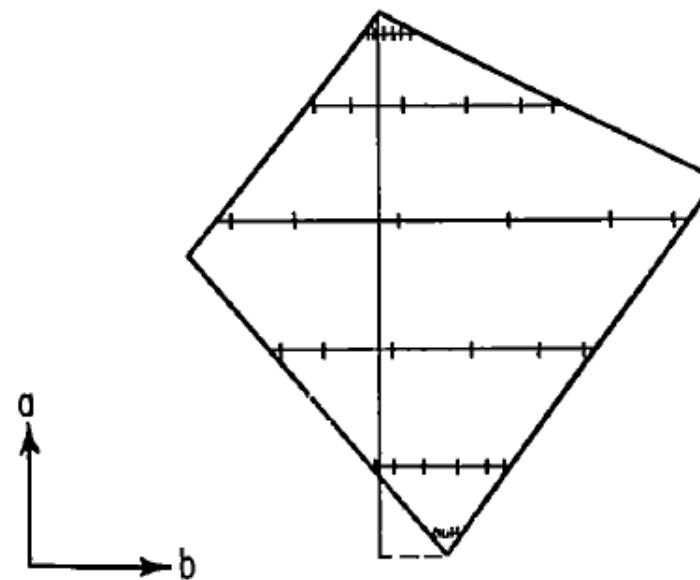


Fig.2. A 6×6 Gaussian grid in a two-dimensional crystal.

$\text{Sr}_2\text{Ir}_{0.8}\text{Rh}_{0.2}\text{O}_4$ linear absorption coefficients

- ✎ In general, μ is equal to the sum of cross sections for all atoms in the unit cell, divided by the unit cell volume:

$$\mu = \Sigma \sigma_i / V_c$$

- ✎ For each peak, read the wavelength and calculate the linear absorption coefficient:

$$\mu(\lambda) = 0.431 + (3.893/1.8)\lambda$$

Output from topaz_reduction.py script:

Chemical formula: Ir0.82 Rh0.18 Sr2 O4

Number of formula units in the unit cell (Z): 8.000

Unit cell volume (Å³): 775.00

Cross sections in units of barns (1 barn = 1E-24 cm²)

Absorption cross section for 2200 m/s neutrons (wavelength = 1.8 Å)

For further information and references, see ...\\ISAW\\Databases\\NIST_cross-sections.dat

Atom	ScatXs	AbsXs
----	-----	-----
Ir	14.00000	425.00000
Rh	4.60000	144.80000
Sr	6.25000	1.28000
O	4.23200	0.00019

The linear absorption coefficient for total scattering is 0.431 cm⁻¹

The linear absorption coefficient for true absorption at 1.8 Å is 3.893 cm⁻¹

The calculated density is 7.120 grams/cm³

TOPAZ data analysis software

- ✓ **JANA2020** <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>
 - **FullProf** <https://www.ill.eu/sites/fullprof/>
- } **nuclear
&
magnetic**
-
- ✓ **SHELX-2018** <http://shelx.uni-ac.gwdg.de/SHELX/download.php>
 - User GUI
 - ShelXle** <https://www.shelxle.org/shelx/eingabe.php>
 - Olex² Crystallography Software** <http://www.olexsys.org/Software>
- } **nuclear**

Workshop Talks

George Sheldrick: [SHELXL for neutrons](#) (TOPAZ, Oak Ridge 2015)

Xiaoping Wang: [Refinement of small molecules against neutron data](#) (ACA, 2016)

Robert Von Dreele: [Single crystal structure refinement with TOF data in GSAS-II](#) (Argonne, 2016)

Tutorials

[Single Crystal Neutron Diffraction Data Reduction and Analysis](#) (ORNL 2024)

NDIP - <https://ndip.ornl.gov/nova/>

Home / Single Crystal Diffraction

Single Crystal Diffraction Applications

Single Crystal Diffraction Tools

	Topaz Data Reduction 0.17.2 Starts interactive user interface for TOPAZ Data Reduction.	START ►
	Jana2020 Visualizer 1.3.62 Starts the interactive Jana2020 Crystallographic Computing System for Standard and Modulated...	OPEN STOP ■
	Olex2 Crystallography Application 1.5 Starts the interactive Olex2 Crystallographic Software.	START ►
	DISCUS: Diffuse Scattering and Defect Structure Simulation 6.21.00 Starts a Jupyter notebook with a Python kernel containing the DISCUS Python bindings.	START ►
	ShelXle 1.0.1807 Starts ShelXle.	START ►
	NeuXtalViz 0.1.0.dev122 Starts the noVNC version of NeuXtalViz.	START ►

SHELX Wiki and Links

 <https://shelx.uni-goettingen.de/wikis.php>

Registration
Downloads
Tutorials & talks
Wikis & manuals
Open access papers
SM GUIs etc.
MM GUIs etc.
SHELX workshops
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SHELX workshops

SHELX Workshops at the ACA Meeting 2016

The following talks were presented at the SHELX workshops at the ACA Meeting in Denver Colorado USA on July 22nd 2016, chair George Sheldrick, Göttingen and co-chair: Jens Lübben, Düsseldorf. [P] refers to versions with four slides per page suitable for printing.

Small molecule and solid state chemistry

George Sheldrick (Göttingen): [Introduction to SHELX, structure solution with SHELXT](#) [P]

Peter Mueller (MIT): [Refinement of disorder with SHELXL](#) [P]

Bill Clegg (Newcastle): [Twinning and absolute structure](#) [P]

Claudia Wandtke (Göttingen): [shelXle - a GUI for structure determination with SHELXL](#) [P]

Daniel Kratzert (Freiburg): [DSR - disordered structure refinement](#) [P]

Xiaoping Wang (ORNL): [Refinement of small molecules against neutron data](#) [P]

Ton Spek (Utrecht): [\(check\)CIF, SHELXL-2014, SQUEEZE](#) [P]

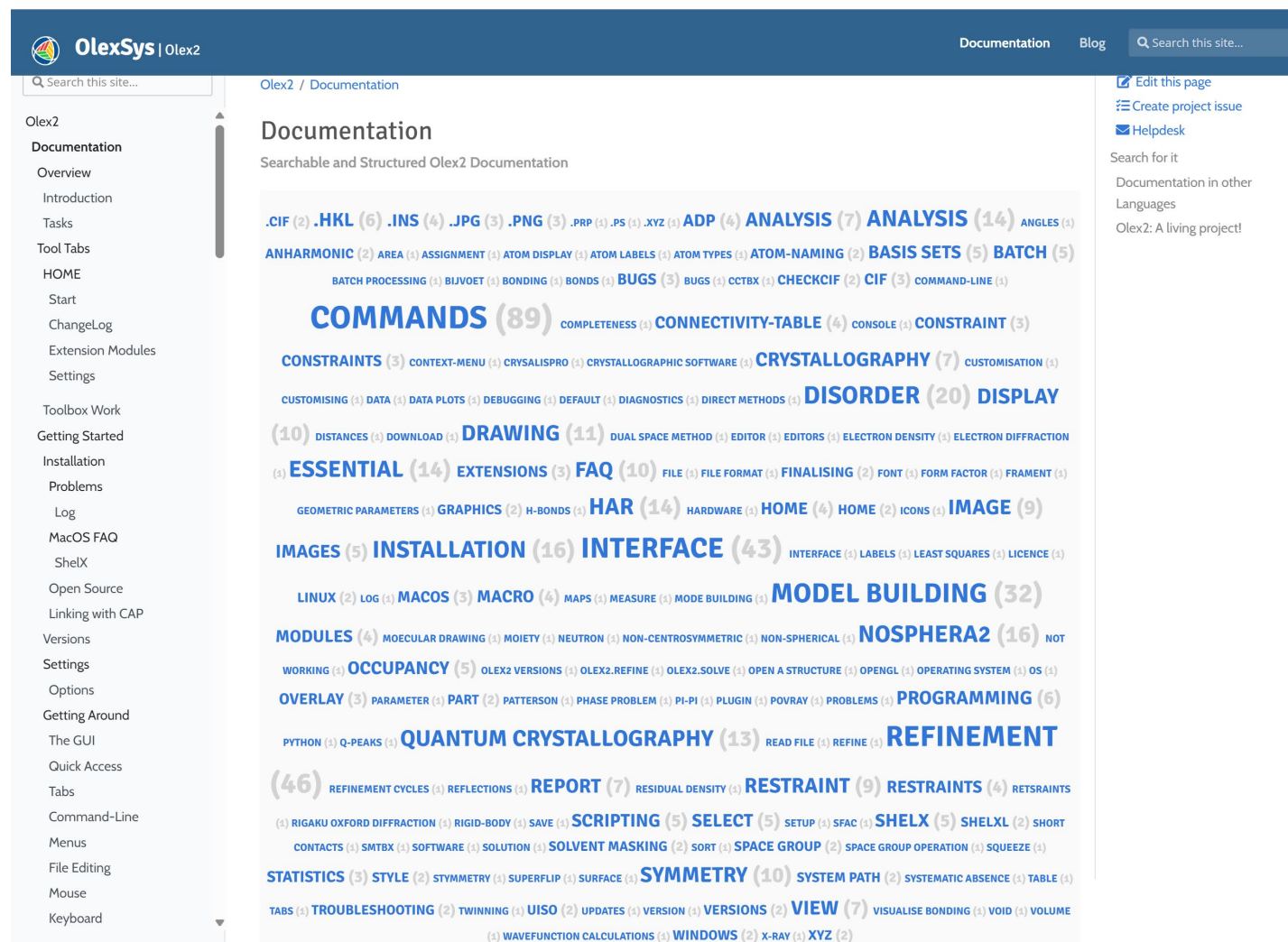


Olex2 Links

 <https://www.olexsys.org/olex2/>

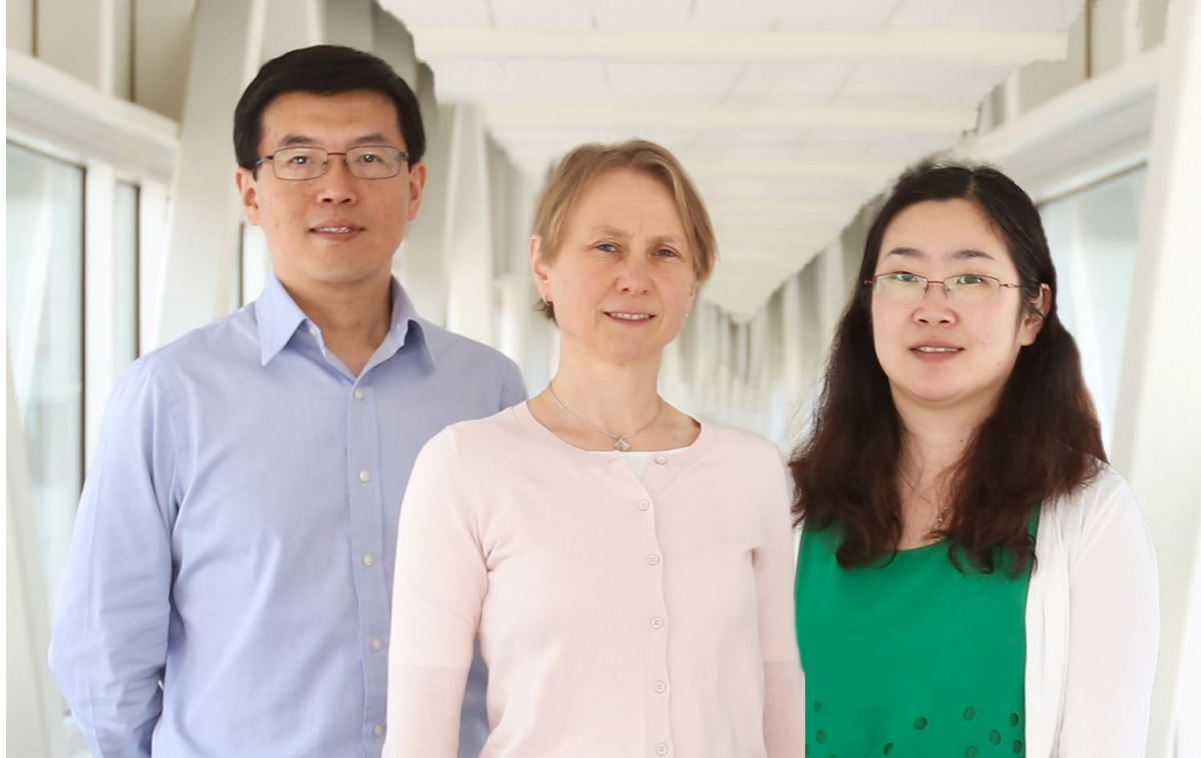
 [Olex2 User Guide](#)

 [Olex2 Cheat Sheet](#)



The screenshot shows the Olex2 Documentation website. The header is dark blue with the OlexSys logo and 'Olex2' text. Navigation links include 'Documentation' and 'Blog'. A search bar is present. The left sidebar lists various categories like 'Olex2', 'Documentation', 'Overview', 'Tasks', 'Tool Tabs', 'HOME', 'Start', 'ChangeLog', 'Extension Modules', 'Settings', 'Toolbox Work', 'Getting Started', 'Installation', 'Problems', 'Log', 'MacOS FAQ', 'ShelX', 'Open Source', 'Linking with CAP', 'Versions', 'Settings', 'Options', 'Getting Around', 'The GUI', 'Quick Access', 'Tabs', 'Command-Line', 'Menus', 'File Editing', 'Mouse', and 'Keyboard'. The main content area is titled 'Documentation' and 'Searchable and Structured Olex2 Documentation'. It features a large list of links with counts in parentheses, such as '.CIF (2)', '.HKL (6)', '.INS (4)', '.JPG (3)', '.PNG (3)', '.PRP (1)', '.PS (1)', '.XYZ (1)', 'ADP (4)', 'ANALYSIS (7)', 'ANALYSIS (14)', 'ANGLES (1)', 'ANHARMONIC (2)', 'AREA (1)', 'ASSIGNMENT (1)', 'ATOM DISPLAY (1)', 'ATOM LABELS (1)', 'ATOM TYPES (1)', 'ATOM-NAMING (2)', 'BASIS SETS (5)', 'BATCH (5)', 'BATCH PROCESSING (1)', 'BIJVOET (1)', 'BONDING (1)', 'BONDS (1)', 'BUGS (3)', 'BUGS (1)', 'CCTBX (1)', 'CHECKCIF (2)', 'CIF (3)', 'COMMAND-LINE (1)', 'COMPLETENESS (1)', 'CONNECTIVITY-TABLE (4)', 'CONSOLE (1)', 'CONSTRAINT (3)', 'CONSTRAINTS (3)', 'CONTEXT-MENU (1)', 'CRYALISPRO (1)', 'CRYSTALLOGRAPHIC SOFTWARE (1)', 'CRYSTALLOGRAPHY (7)', 'CUSTOMISATION (1)', 'CUSTOMISING (1)', 'DATA (1)', 'DATA PLOTS (1)', 'DEBUGGING (1)', 'DEFAULT (1)', 'DIAGNOSTICS (1)', 'DIRECT METHODS (1)', 'DISORDER (20)', 'DISPLAY (10)', 'DISTANCES (1)', 'DOWNLOAD (1)', 'DRAWING (11)', 'DUAL SPACE METHOD (1)', 'EDITOR (1)', 'EDITORS (1)', 'ELECTRON DENSITY (1)', 'ELECTRON DIFFRACTION (1)', 'ESSENTIAL (14)', 'EXTENSIONS (3)', 'FAQ (10)', 'FILE (1)', 'FILE FORMAT (1)', 'FINALISING (2)', 'FONT (1)', 'FORM FACTOR (1)', 'FRAGMENT (1)', 'GEOMETRIC PARAMETERS (1)', 'GRAPHICS (2)', 'H-BONDS (1)', 'HAR (14)', 'HARDWARE (1)', 'HOME (4)', 'HOME (2)', 'ICONS (1)', 'IMAGE (9)', 'IMAGES (5)', 'INSTALLATION (16)', 'INTERFACE (43)', 'INTERFACE (1)', 'LABELS (1)', 'LEAST SQUARES (1)', 'LICENCE (1)', 'LINUX (2)', 'LOG (1)', 'MACOS (3)', 'MACRO (4)', 'MAPS (1)', 'MEASURE (1)', 'MODE BUILDING (1)', 'MODEL BUILDING (32)', 'MODULES (4)', 'MOLECULAR DRAWING (1)', 'MOIETY (1)', 'NEUTRON (1)', 'NON-CENTROSYMMETRIC (1)', 'NON-SPHERICAL (1)', 'NOSPHERA2 (16)', 'NOT WORKING (1)', 'OCCUPANCY (5)', 'OLEX2 VERSIONS (1)', 'OLEX2.REFINE (1)', 'OLEX2.SOLVE (1)', 'OPEN A STRUCTURE (1)', 'OPENGL (1)', 'OPERATING SYSTEM (1)', 'OS (1)', 'OVERLAY (3)', 'PARAMETER (1)', 'PART (2)', 'PATTERSON (1)', 'PHASE PROBLEM (1)', 'PI-PI (1)', 'PLUGIN (1)', 'POVRAY (1)', 'PROBLEMS (1)', 'PROGRAMMING (6)', 'PYTHON (1)', 'Q-PEAKS (1)', 'QUANTUM CRYSTALLOGRAPHY (13)', 'READ FILE (1)', 'REFINE (1)', 'REFINEMENT (46)', 'REFINEMENT CYCLES (1)', 'REFLECTIONS (1)', 'REPORT (7)', 'RESIDUAL DENSITY (1)', 'RESTRAINT (9)', 'RESTRAINTS (4)', 'RETSRAINTS (1)', 'RIGAKU OXFORD DIFFRACTION (1)', 'RIGID-BODY (1)', 'SAVE (1)', 'SCRIPTING (5)', 'SELECT (5)', 'SETUP (1)', 'SFAC (1)', 'SHELX (5)', 'SHELXL (2)', 'SHORT CONTACTS (1)', 'SMTBX (1)', 'SOFTWARE (1)', 'SOLUTION (1)', 'SOLVENT MASKING (2)', 'SORT (1)', 'SPACE GROUP (2)', 'SPACE GROUP OPERATION (1)', 'SQUEEZE (1)', 'STATISTICS (3)', 'STYLE (2)', 'SYMMETRY (1)', 'SUPERFLIP (1)', 'SURFACE (1)', 'SYMMETRY (10)', 'SYSTEM PATH (2)', 'SYSTEMATIC ABSENCE (1)', 'TABLE (1)', 'TABS (1)', 'TROUBLESHOOTING (2)', 'TWINNING (1)', 'UIISO (2)', 'UPDATES (1)', 'VERSION (1)', 'VERSIONS (2)', 'VIEW (7)', 'VISUALISE BONDING (1)', 'VOID (1)', 'VOLUME (1)', 'WAVEFUNCTION CALCULATIONS (1)', 'WINDOWS (2)', 'X-RAY (1)', 'XYZ (2)'.

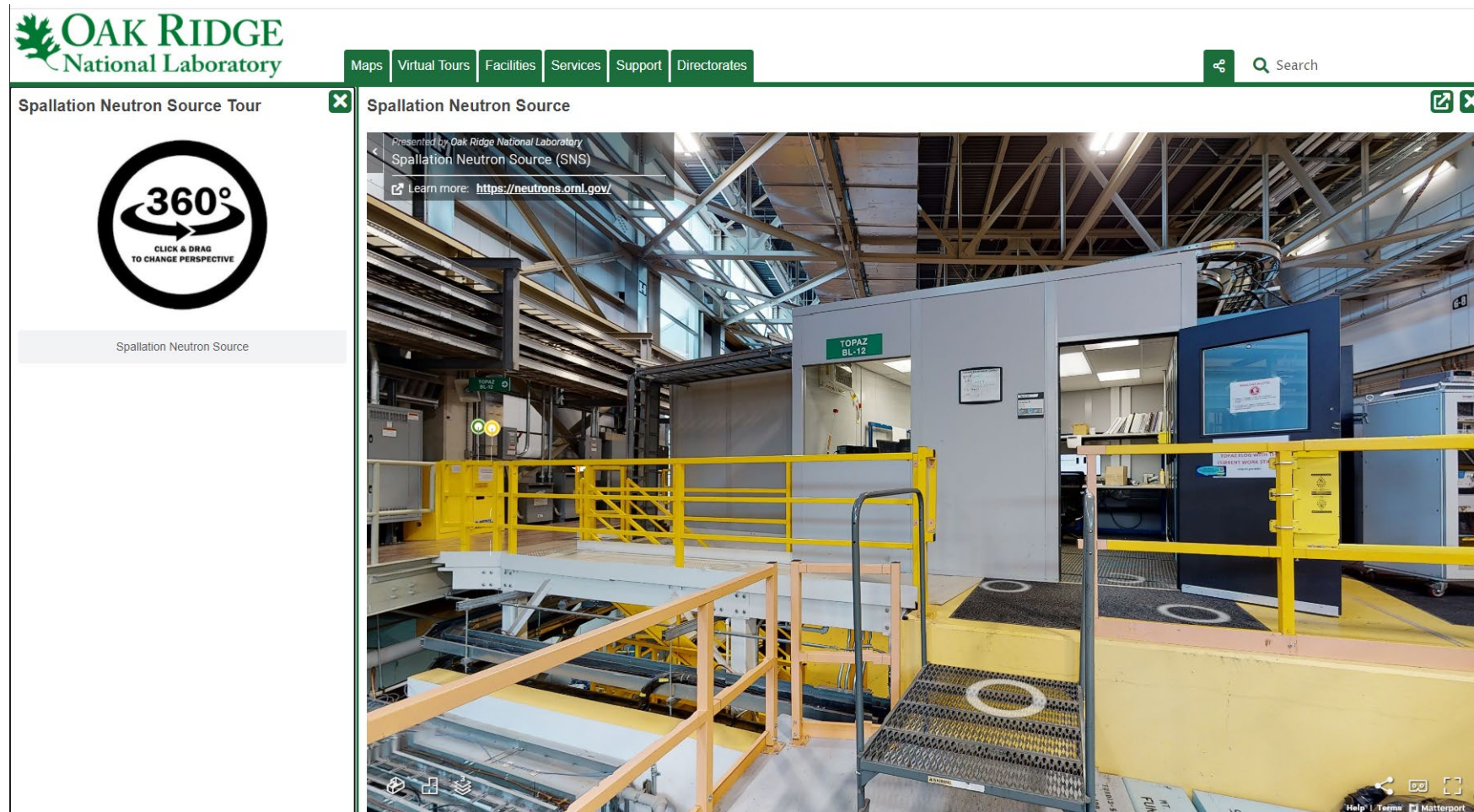
TOPAZ Instrument Team



Xiaoping Wang, Christina Hoffmann, Helen He

A Virtual Tour of TOPAZ at SNS

<https://neutrons.ornl.gov/virtual-tour>



Thank you for viewing !

Xiaoping Wang wangx@ornl.gov

Christina Hoffmann choffmann@ornl.gov

Information for TOPAZ are available online at <https://neutrons.ornl.gov/topaz>