

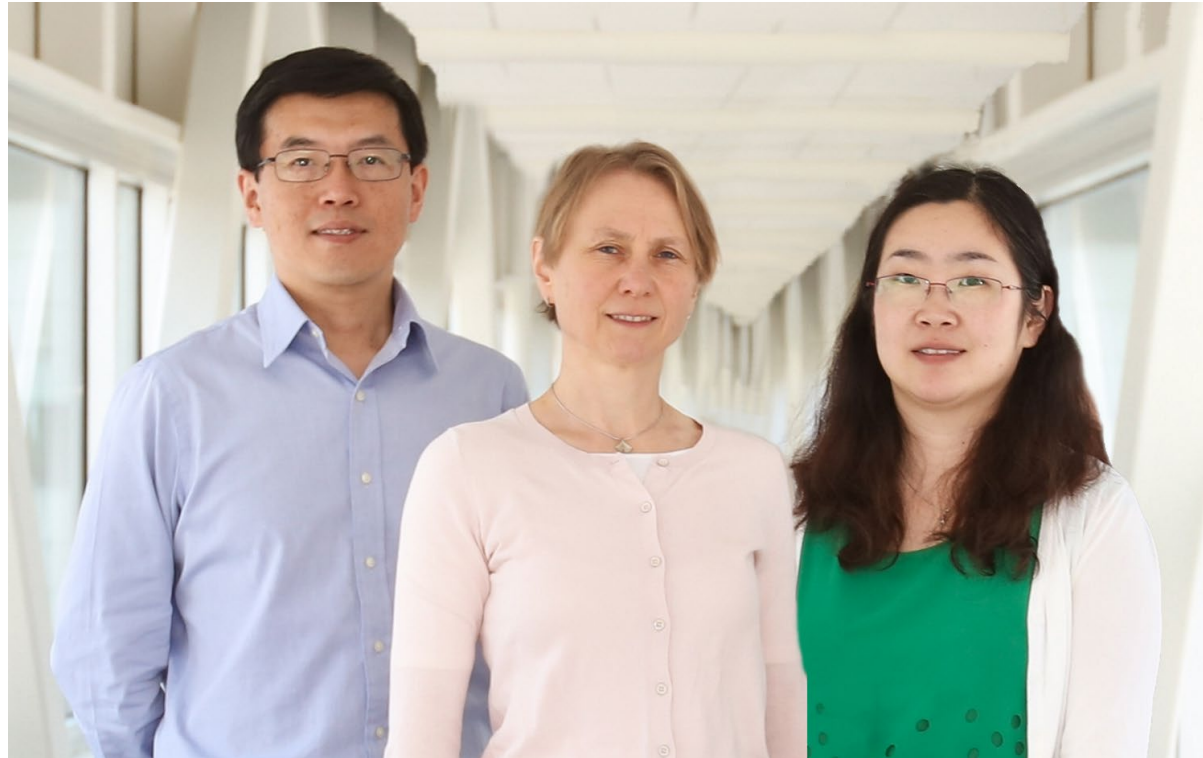
SNS BL-12 TOPAZ

Single crystal neutron
Time of Flight Laue diffractometer

Xiaoping Wang and Christina Hoffmann
Neutron scattering Division



TOPAZ Instrument Team

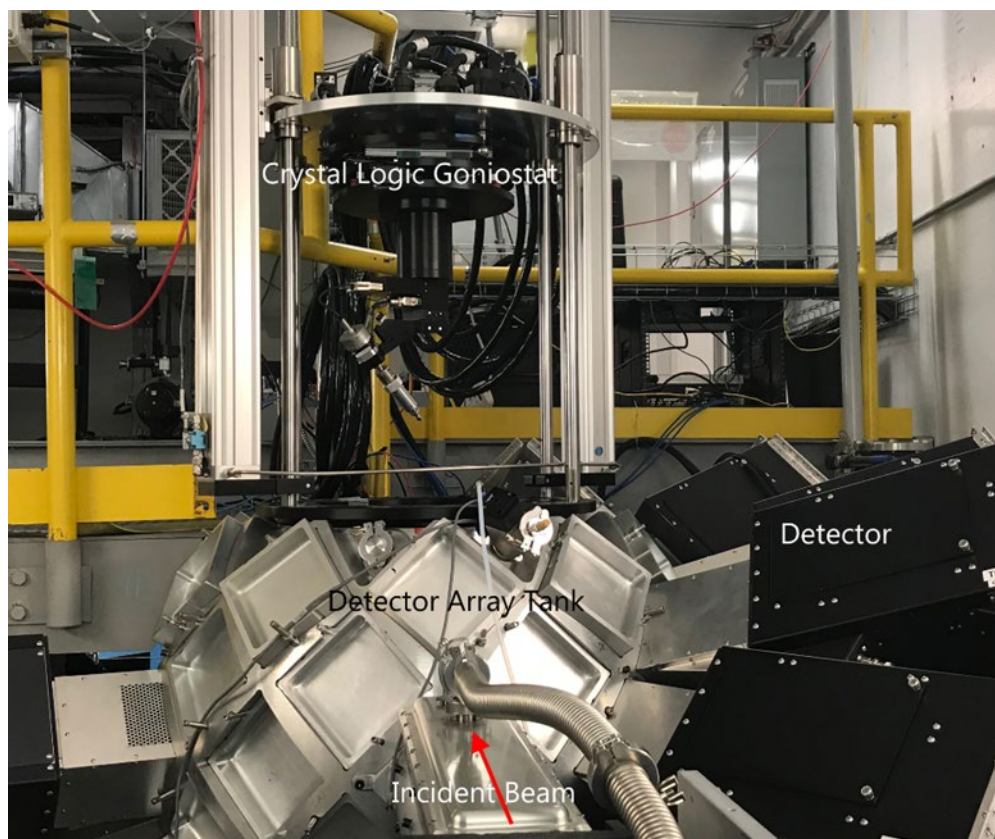


Xiaoping Wang, Christina Hoffmann, Helen He

TOPAZ

A high-resolution single crystal diffractometer

$$Q_{\max} = 25 \text{ \AA}^{-1} \quad d_{\min} = 0.25 \text{ \AA}$$



Sub-Millimeter Sized Crystals

Diameter: 0.10 – 4.0 mm, Volume: > 0.1 mm³

Multiple Area Detectors

Solid Angle Coverage: 3.2 ster.

Detector 2 θ Coverage: 13.5° - 160°

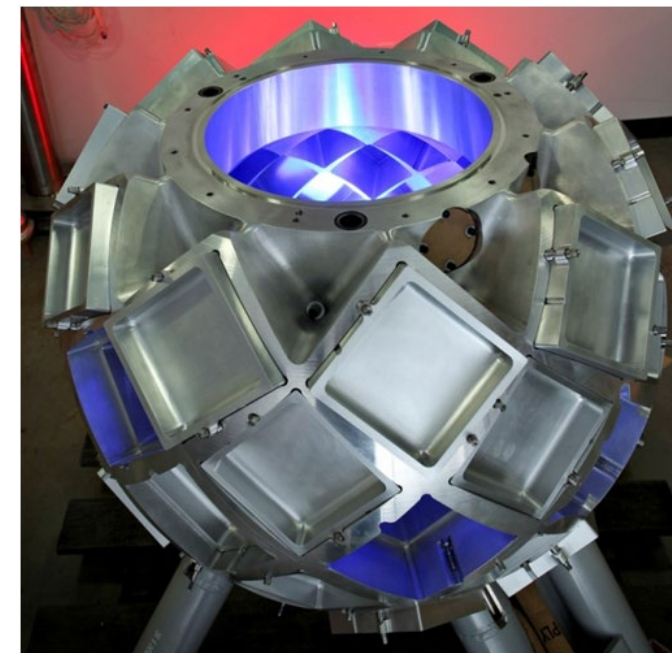
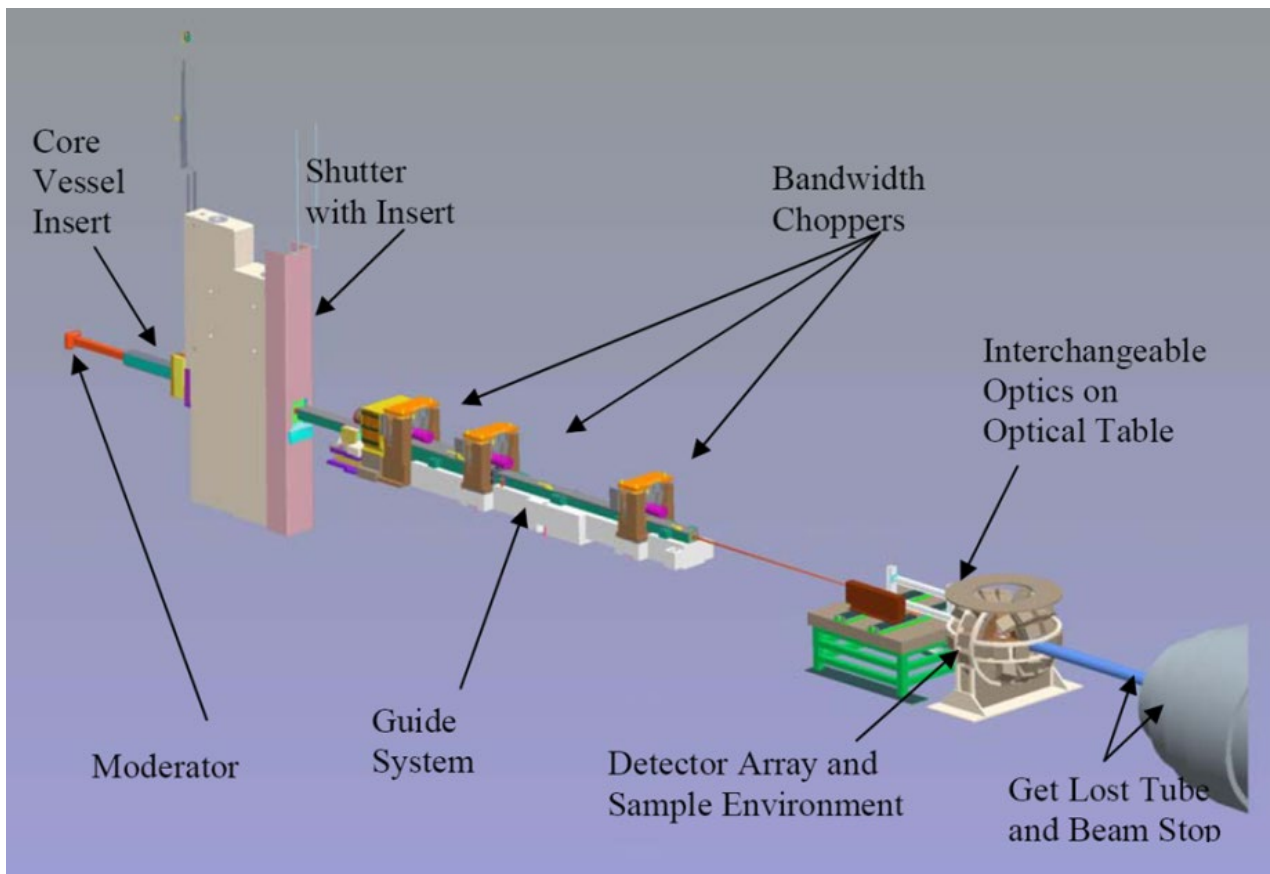
Sample Environment

CryoStream 700 Plus: 90K – 450K

Electric Field

Cryogenic goniometer 5K – 300 K (2020)

Original Design



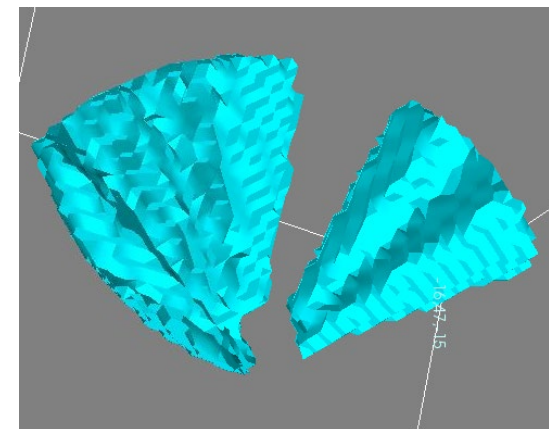
48

Detector modules to be installed on the Detector Array Tank (DAT)

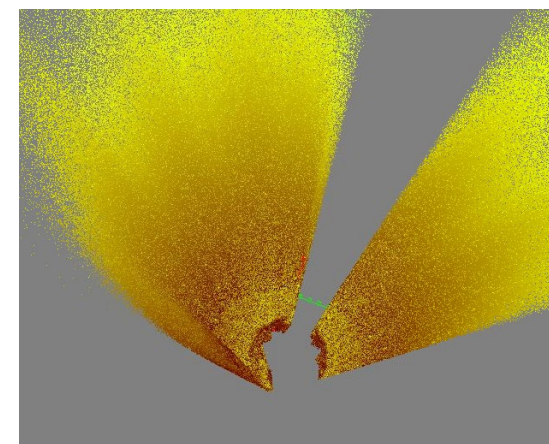
Data are recorded in neutron event mode

- 3D Continuous Q space mapping

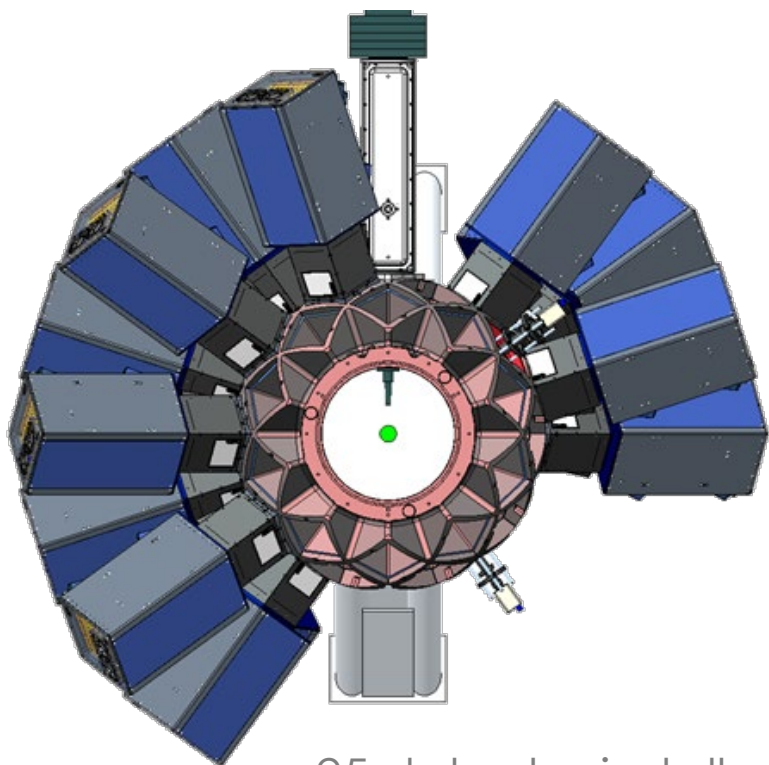
Anger Camera



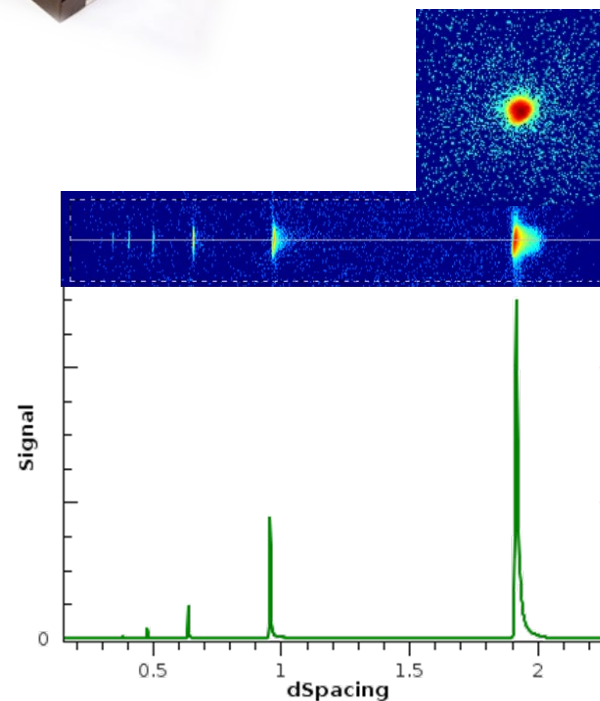
3D Q Coverage, simulated
CrystalPlan



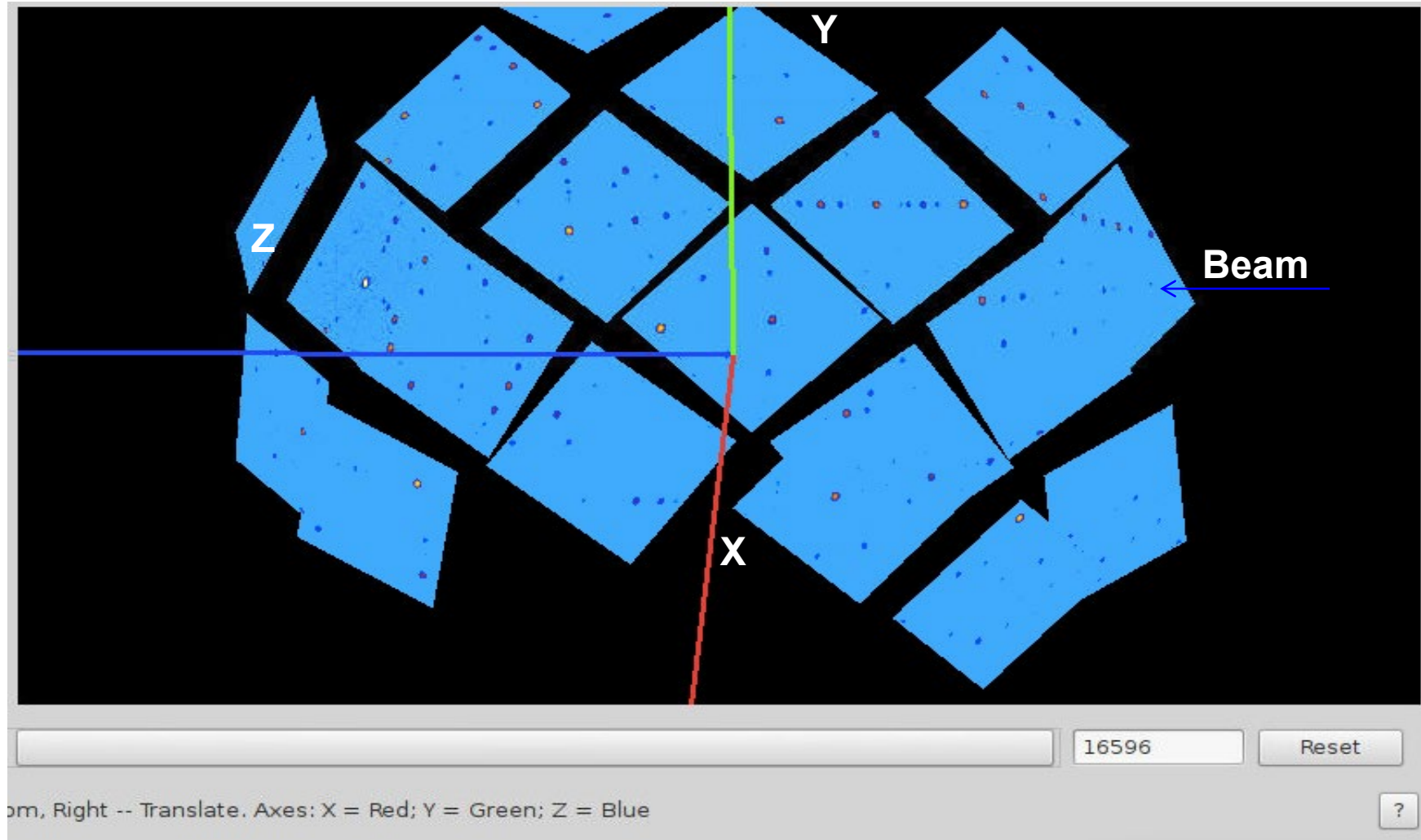
3D Q coverage, measured
ISAWEV



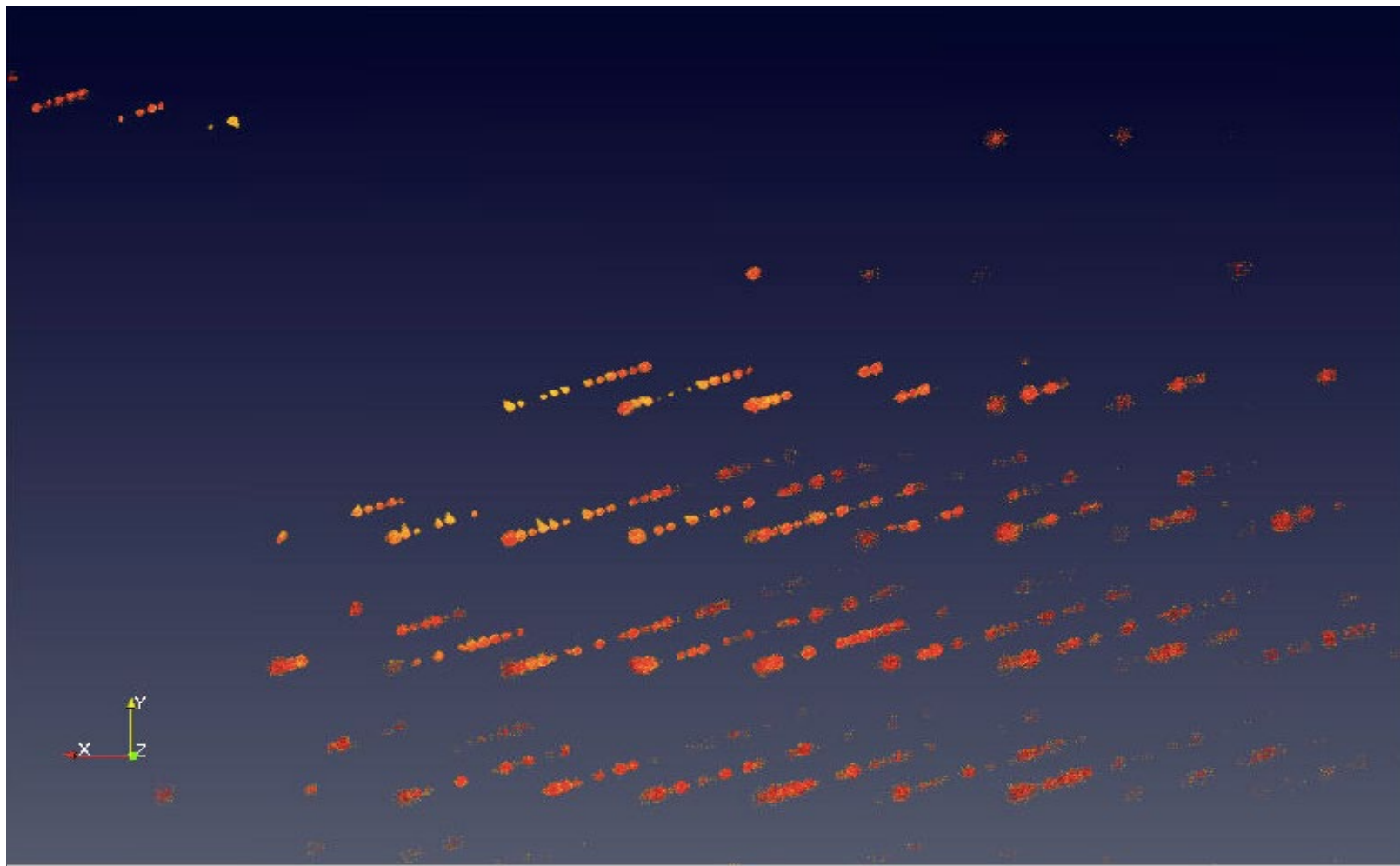
25 detector installed



Single crystal peaks on 2D detector space



Single crystal peaks in 3D Q space

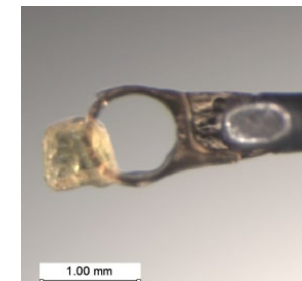


TOPAZ Ambient Goniometer

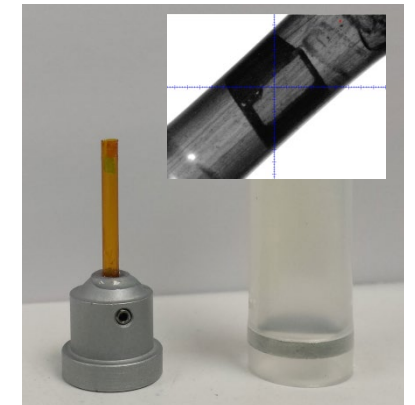


- A **two-axis** goniometer
 - Omega, phi with chi fixed at 135°.
 - Both omega and phi are fitted with sliprings that allow unlimited 360° rotational motion.
 - The omega and phi rotation axes are separated by 45 degrees.
 - Sample mount

MiTeGen loop (1 mm ϕ)



Glued or Coated with perfluorinated grease

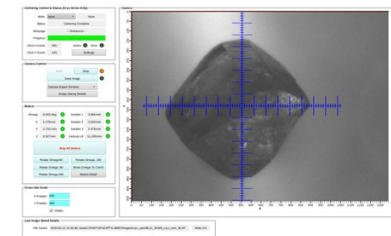
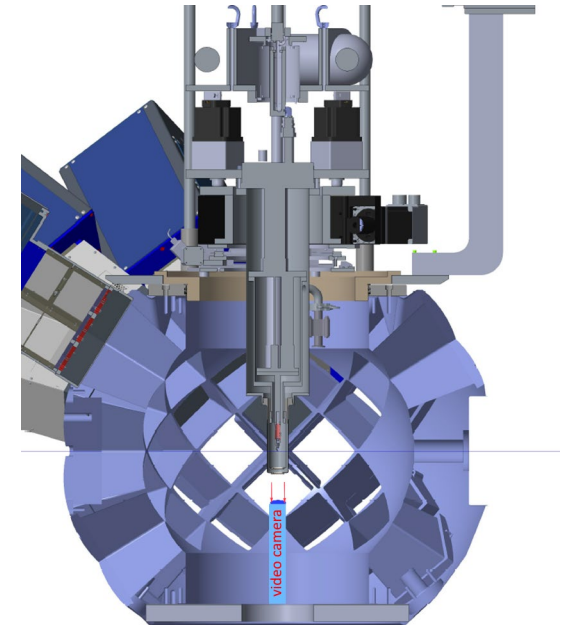
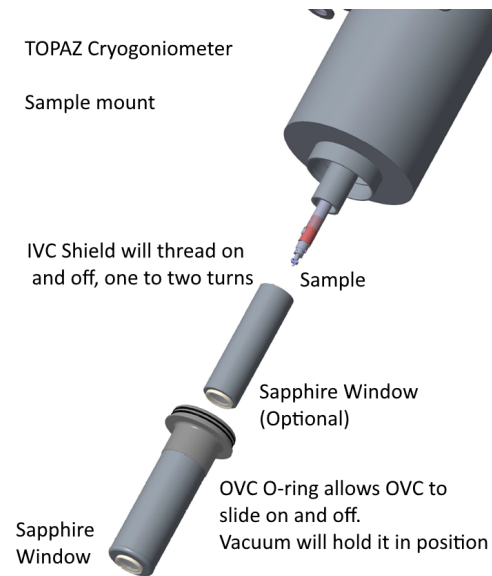
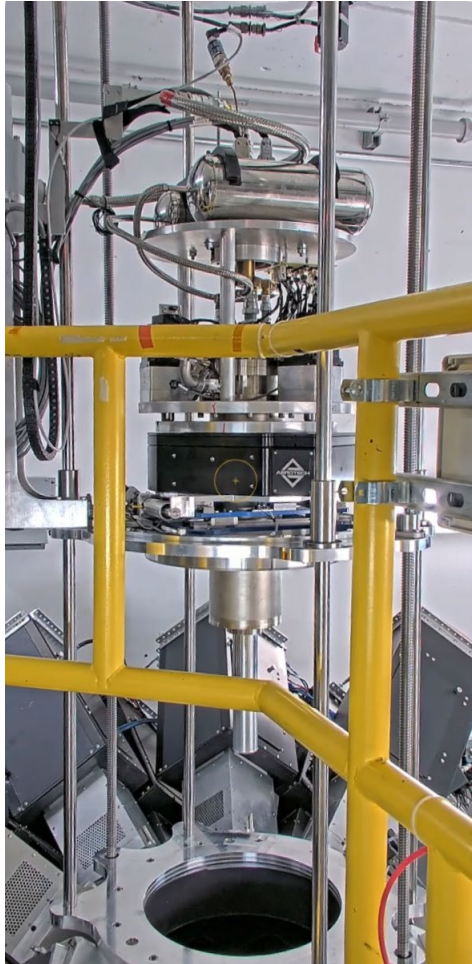


Glued onto the tip or inside a Kapton tube

TOPAZ Cryogenic Goniometer

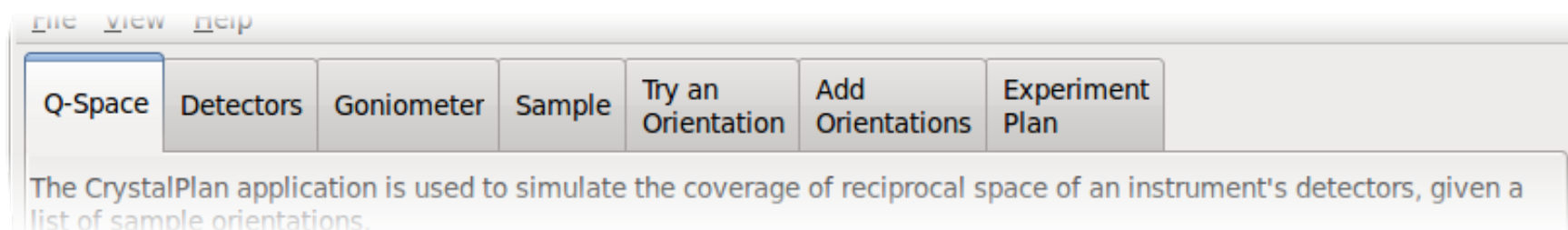
One axis of rotation (360°) with precision motor controls to center, orient, and hold sample in temperature range 5 K – 300 K.

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

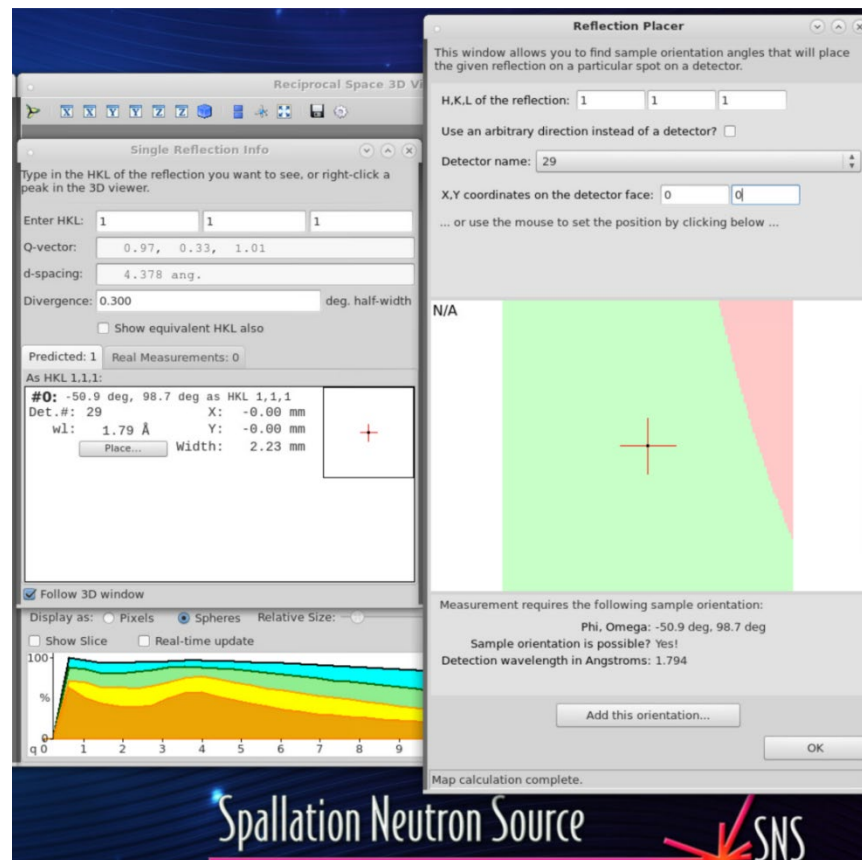


Crystal Plan

- An experiment planning tool for time-of-flight Laue experiment
- User friendly GUI Interfaces
 - Maximize the use of available beam time and productivity
 - Capable of placing an individual peak on selected detector position



CrystalPlan – Peak Prediction and Placement



Predict and place a single-crystal peak on selected detector location

Reflection Placer for Cryogoniometer

The screenshot displays the CrystalPlan 1.2 software interface. The main window contains a menu bar (File, View, Tools, Help) and a toolbar with buttons for Q-Space, Detectors, Goniometer, Sample, Try an Orientation, Add Orientations, and Experiment Plan. Below the toolbar is a text area with instructions: "Select the sample orientations you wish to use in the experiment, and the criterion for data acquisition at each orientation." There are checkboxes for "Use All" and "Highlighted Rows", and buttons for "Use", "Don't Use", and "Change Stopping Criteria".

The central part of the interface is a table with the following columns: Use?, Phi (deg), Stopping Criterion, Criterion Value, and Comment. The table lists 23 sample orientations, all with a "proton charge (pC)" stopping criterion and a "5.0" criterion value. The phi values range from 92.00 to -166.63 degrees.

At the bottom of the main window, there are buttons for "Delete All", "Delete Highlighted", "Delete Unused", and "Refresh List". Below these are text fields for "Estimated run time: 1.150e+02 pC proton charge, approx 00m 00s (at 1 MW accelerator power)." and "Calculating -166.6 deg...". There are also buttons for "Save to .CSV file" and "Automatic Coverage Optimizer...".

The "Reflection Placer" window is open, showing the H,K,L of the reflection as 2 0 -2. It includes a "Detector name" dropdown set to "18" and "X,Y coordinates on the detector face" set to 35.27 and -8.63. A 3D visualization shows a red rectangular detector face with a green line representing the reflection path. The coordinates -27.79, 71.02 are displayed. Below the visualization, it states: "Measurement requires the following sample orientation: Phi: -166.6 deg. Sample orientation is possible? Yes! Detection wavelength in Angstroms: 3.037". There is an "Add this orientation..." button and an "OK" button.

The "Single Reflection Info" window is also open, showing the H,K,L (0 -2 0), Q-vector (0.00, -1.74, 0.00), d-spacing (3.614 ang.), and Divergence (0.300 deg. half-width). It lists predicted and real measurements, and provides details for three reflections: #0 (92.0 deg), #1 (109.7 deg), and #2 (-39.3 deg), including detector numbers, wavelengths, and positions.

A "Coverage" window shows 3143 reflections with a coverage of 71.3%. A bar chart indicates that 52.2% of reflections were measured more than once. There is an "Auton" checkbox and a "3D Advanced Settings..." button.

CrystalPlan – Detector coverage

The screenshot displays the CrystalPlan 1.2 software interface, divided into two main windows: 'CrystalPlan 1.2 - Main Window' and 'Reciprocal Space 3D Viewer'.

CrystalPlan 1.2 - Main Window:

File View Tools Help

Q-Space Detectors Goniometer Sample Try an Orientation Add Orientations Experiment Plan

Select the sample orientations you wish to use in the experiment, and the criterion for data acquisition at each orientation.

Use All Highlighted Rows: Use Don't Use Change Stopping Criteria

	Use?	Phi (deg)	Omega (deg)	Stopping Criterion	Criterion Value	Comment
1	X	-168.65	137.01	proton charge (pC)	4e+12	
2	X	-114.41	-94.61	proton charge (pC)	4e+12	
3	X	-111.96	-144.47	proton charge (pC)	4e+12	
4	X	-99.78	69.31	proton charge (pC)	4e+12	
5	X	-71.25	-88.92	proton charge (pC)	4e+12	
6	X	-42.84	36.80	proton charge (pC)	4e+12	
7	X	13.79	-66.60	proton charge (pC)	4e+12	
8	X	70.89	94.66	proton charge (pC)	4e+12	
9	X	72.87	-50.16	proton charge (pC)	4e+12	
10	X	111.05	3.47	proton charge (pC)	4e+12	
11	X	20.49	87.19	proton charge (pC)	4e+12	020Det29
12	X	-50.92	98.66	proton charge (pC)	4e+12	111Det29
13	X	86.74	97.69	proton charge (pC)	4e+12	
14	X	34.13	115.12	proton charge (pC)	4e+12	
15	X	-20.43	76.44	proton charge (pC)	4e+12	
16	X	-3.00	11.50	proton charge (pC)	4e+12	
17	X	65.73	84.37	proton charge (pC)	4e+12	
18	X	83.66	127.29	proton charge (pC)	4e+12	

Delete All Delete Highlighted Delete Unused Refresh List

Estimated run time: 7.600e+13 pC proton charge, approx 20h 29m 48s (at 1 MW accelerator power).

Save to .CSV file Automatic Coverage Optimizer...

Calculating -93.9 deg, 36.4 deg...

Reciprocal Space 3D Viewer:

Volume Coverage View Reflections View

Mouse is over: hkl: -2,0,19

Show which peaks: Predicted reflections Use Symmetry?

Color by: Predicted Measured I/sigI threshold 0.0

Display as: Pixels Spheres Relative Size: Automatic

Show Slice Real-time update

Coverage:

19041 reflections

Coverage of 75.6%:

46.8% measured > once:

3D Advanced Settings...

Zikovsky J., Peterson P.F., Wang X.P., Frost M., Hoffmann C., "CrystalPlan: an experiment-planning tool for crystallography", *Journal of Applied Crystallography*, **44**, 418-423 (2011).

CrystalPlan – Detector coverage by symmetry

CrystalPlan 1.2 - Main Window

File View Tools Help

Q-Space Detectors Goniometer Sample Try an Orientation Add Orientations Experiment Plan

Select the sample orientations you wish to use in the experiment, and the criterion for data acquisition at each orientation.

Use All Highlighted Rows: Use Don't Use Change Stopping Criteria

	Use?	Phi (deg)	Omega (deg)	Stopping Criterion	Criterion Value	Comment
1	X	-168.65	137.01	proton charge (pC)	4e+12	
2	X	-114.41	-94.61	proton charge (pC)	4e+12	
3	X	-111.96	-144.47	proton charge (pC)	4e+12	
4	X	-99.78	69.31	proton charge (pC)	4e+12	
5	X	-71.25	-88.92	proton charge (pC)	4e+12	
6	X	-42.84	36.80	proton charge (pC)	4e+12	
7	X	13.79	-66.60	proton charge (pC)	4e+12	
8	X	70.89	94.66	proton charge (pC)	4e+12	
9	X	72.87	-50.16	proton charge (pC)	4e+12	
10	X	111.05	3.47	proton charge (pC)	4e+12	
11	X	20.49	87.19	proton charge (pC)	4e+12	020Det29
12	X	-50.92	98.66	proton charge (pC)	4e+12	111Det29
13	X	86.74	97.69	proton charge (pC)	4e+12	
14	X	34.13	115.12	proton charge (pC)	4e+12	
15	X	-20.43	76.44	proton charge (pC)	4e+12	
16	X	-3.00	11.50	proton charge (pC)	4e+12	
17	X	65.73	84.37	proton charge (pC)	4e+12	
18	X	83.66	127.29	proton charge (pC)	4e+12	

Delete All Delete Highlighted Delete Unused Refresh List

Estimated run time: 7.600e+13 pC proton charge, approx 20h 29m 48s (at 1 MW accelerator power).

Save to .CSV file Automatic Coverage Optimizer...

Calculating -93.9 deg, 36.4 deg...

Reciprocal Space 3D Viewer

Mouse is over: hkl: -2,36,6

Volume Coverage View Reflections View

Show which peaks: Predicted reflections Use Symmetry?

Color by: Predicted Measured I/sigI threshold 0.0

Display as: Pixels Spheres Relative Size: Automatic

Show Slice Real-time update

100%
%
q 0 1 2 3 4 5 6 7 8 9 10 11 12

Coverage (w/ symmetry)

4869 unique reflections
Coverage of 100.0%:

98.5% measured > once:

3D Advanced Settings...

Zikovsky J., Peterson P.F., Wang X.P., Frost M., Hoffmann C., "CrystalPlan: an experiment-planning tool for crystallography", *Journal of Applied Crystallography*, **44**, 418-423 (2011).

TOPAZ Live Instrument Data

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

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[Single-Crystal Diffractometer](#)
[TOPAZ](#) | [BL-12](#) | [SNS](#)

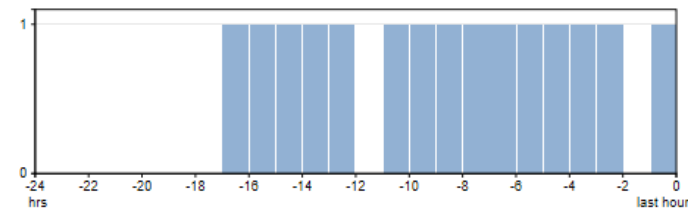
[Overview](#)
[Team](#)
[Capabilities](#)
[User Guidance](#)
[Live Instrument Data](#)
[Operating Status](#)
[Publications](#)
[Spec Sheet](#)
[Co](#)

<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

The screenshot displays the TOPAZ DAS OPI dashboard, which is a web-based interface for controlling the TOPAZ neutron source. The dashboard is organized into several main sections:

- Accelerator & Shutters:** Shows Power (1405 kW), Moderator Temp (19.60 K), and Primary Shutter status.
- Motors:** Lists various motor components like Guide 5, Guide 6, and Slit1, along with their overall status (Idle).
- Sample Environment:** Includes controls for LN2 Dewar Level, LN2 Plant, Cryostream, Sample LED, and HV Pulser.
- Detector Control:** Features buttons for nED, Detectors, and various ADnED modules.
- Experiment:** Contains user information (User: BL12_Dashboard.bob) and buttons for Range/Align, Table Scan, and Temperature Ramp.
- Main Reduction Params:** Displays current parameters such as omega (54.572 deg), phi (0.000 deg), chi (0.000 deg), sample_temp (189.541 K), and sample_ramp_rate (4.039 K/min).
- Choppers & Vacuum:** Shows the status of SKF Choppers and the Vacuum system.
- Software Status:** Includes IOC Status, Data / Reduction, and Archiver Status.
- Sample Centering & Cameras:** Features buttons for Ambient Gonio Cam, Cryo Gonio Cam, and Image Saving.
- Detector Plots:** Provides options for 2D and 1D detector plots.

The dashboard also includes a sidebar on the left for HFIR and various beamline stations (BL-1A USANS, BL-1B NOMAD, BL-2 BASI, BL-5 CNCS, BL-6 EQ-SANS, BL-7 VULCAN, BL-9 COR, BL-12 TOPAZ, BL-13 FNPB, BL-14B HYSPEC, BL-15 NSE) with their respective status indicators and control buttons.

TOPAZ Dashboard

← → ↻ 🏠 status.sns.ornl.gov/dbwr/view.jsp?display=https%3A//webopi.sns.gov/bl12/files/bl12/opi/BL12_Dashboard.bob¯os... 🔍 ☆ 📄 EN

Instrument Status

Beam Power (kW): 1404.98 kW

Primary Shutter: ●

Acquisition Software Status:

Data/Reduction Status: ●

Proposal Information

Proposal #: IPTS- 25682

Proposal Title: Phase competition in the exchange enhanced Stoner naramannet YCo2

Team Members: BGUELAND;BINGLI621;MC (XCAMS/UCAMS)

Run Information

Scan Status: Scanning

Run Status: Run Abort

Run Number: 38854

Run Time: 2130.2 s

Total Neutron Counts: 42588603

Count Rate (counts/s): 20100

Total Proton Charge: 2.9756 C

Beam Monitor 1 Counts: 5458183

Beam Monitor 2 Counts: 10231509

Chopper Information

Center Wavelength: 1.789 Å

Frequency: 60 Hz

3 BWs Phase-Locked: ●

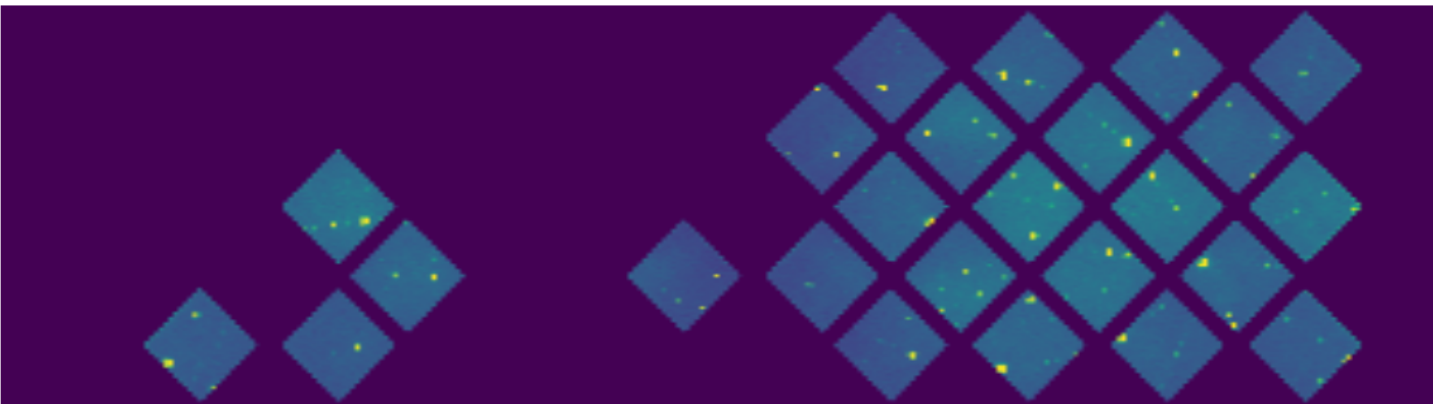
Motor Information

Aperture: 3,3

Phi: 0.000 deg

Omega: -87.997 deg

Main Detector X/Y Plot (4x4 Binned)



Min 0 Max autoscale

Detail Profiles Clear Array & Counts Reset ROI ROI

Min	Max	Mean	Total	Rate
409	591	488.789	175964	91 e/s

Show

Table: /home/controls/var/tmp/2020B/IPTS-25682/RT_YCO2_ARCS_sample_rev1.gnumeric {fileselector}

{table}

TOPAZ Sciences

- **Chemical crystallography**

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

- **Diffuse scattering**

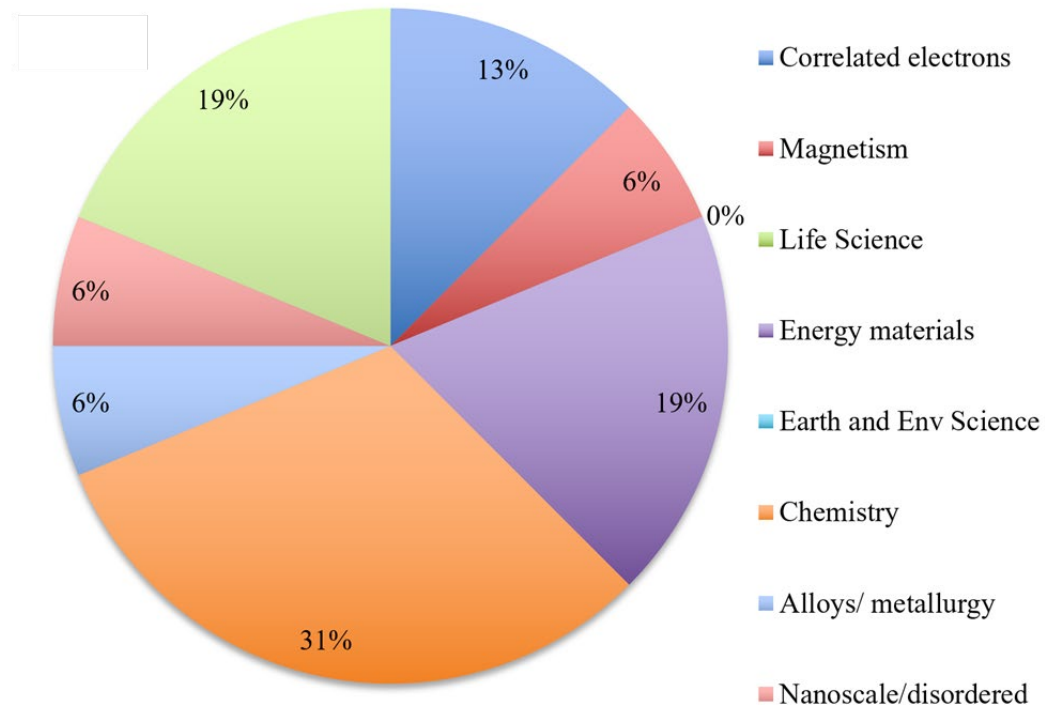
- 3D reciprocal space mapping

- **Magnetism**

- Solve & refine magnetic and nuclear structures

- **Event based parametric study**

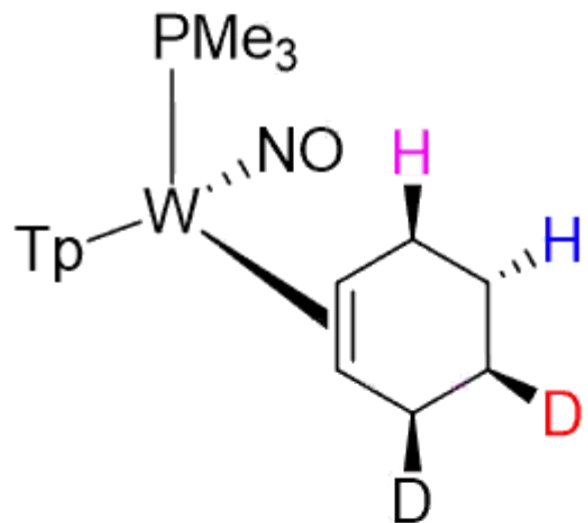
- Applied electric field
- Variable temperature
- Structural phase transitions
- Order parameters



Chemical crystallography

Transition metal-mediated dearomatization.

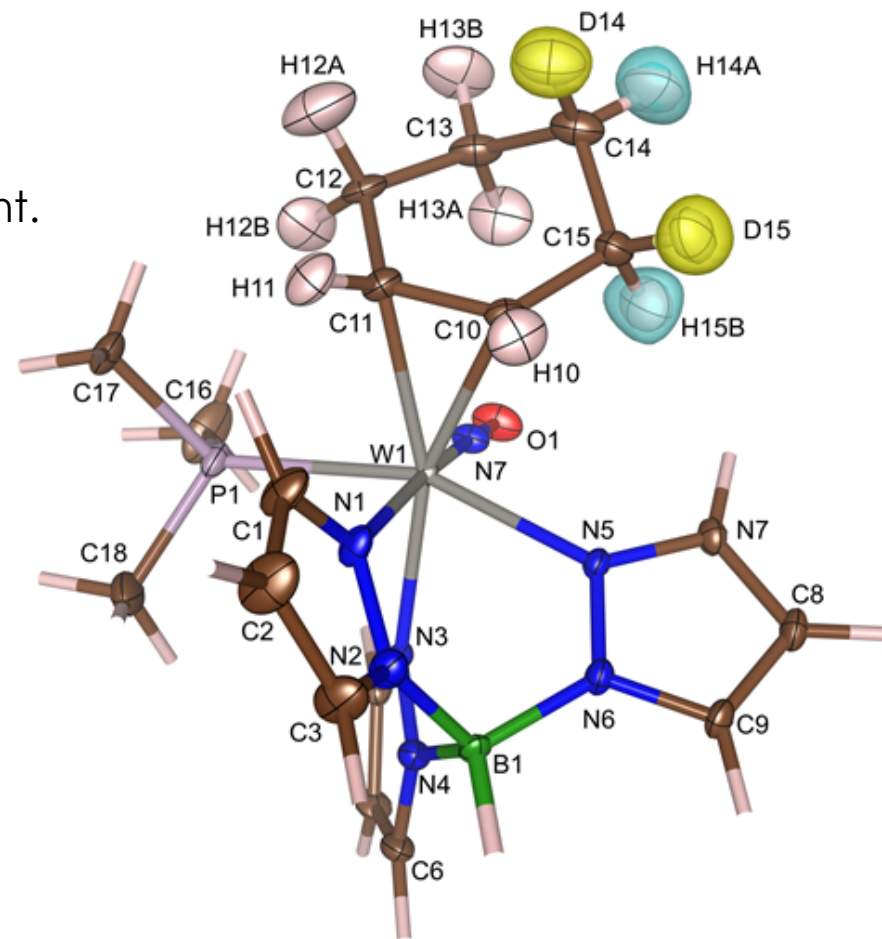
Opened pathways for a new generation of medicines and therapies that incorporate deuterium into the active pharmaceutical ingredient.



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



0.05 mm³



Neutron structure of a d_2 isotopologues of cyclohexene complex.

Single crystal neutron diffraction beyond three dimensions

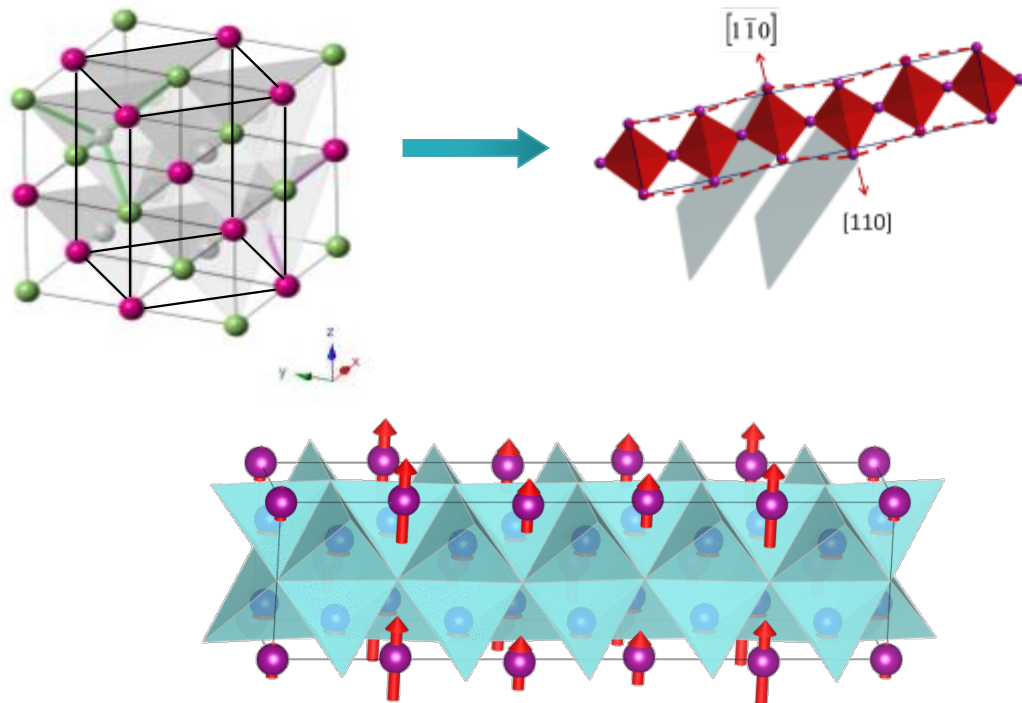
Modulated crystal structure

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

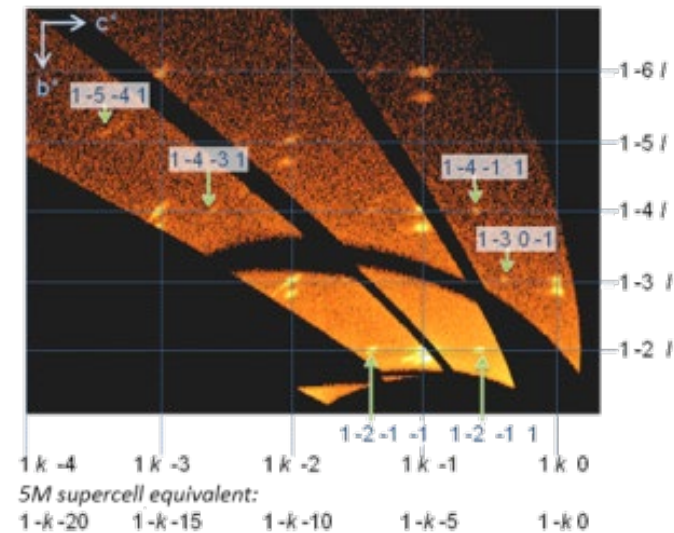


Local order and magnetic structural phase transition

- 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 $P2/m(a0r)00$



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

Single crystal neutron diffraction beyond three dimensions

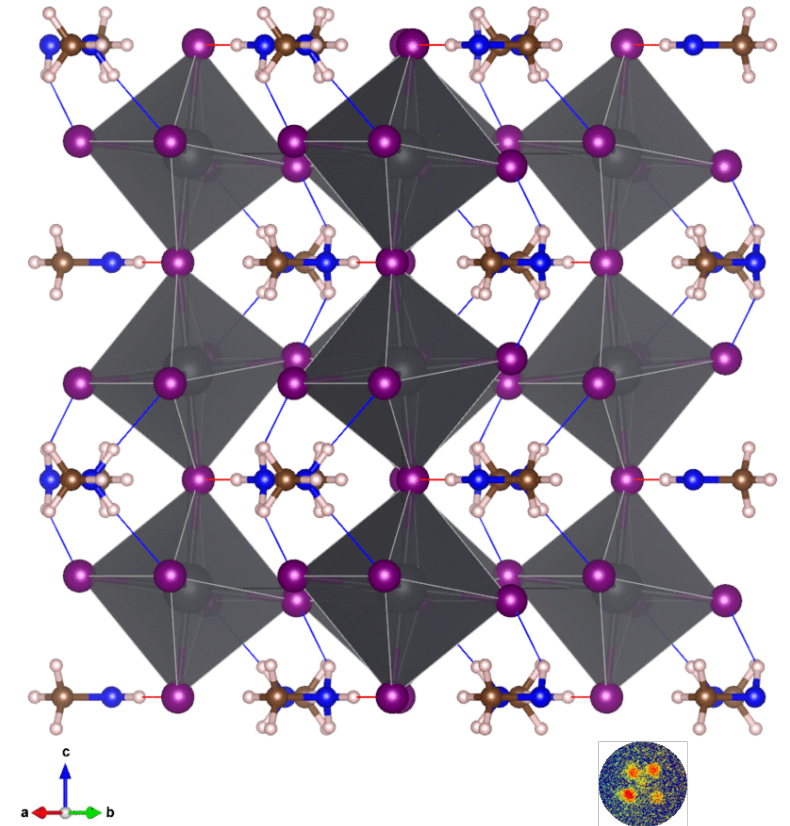
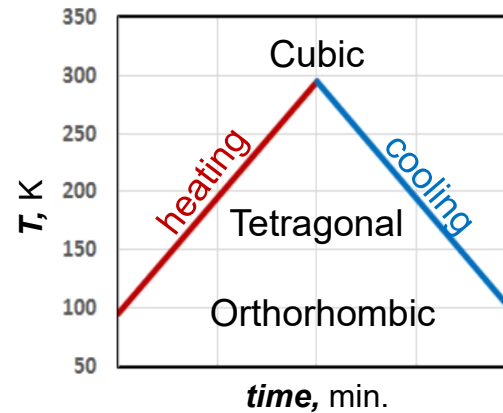
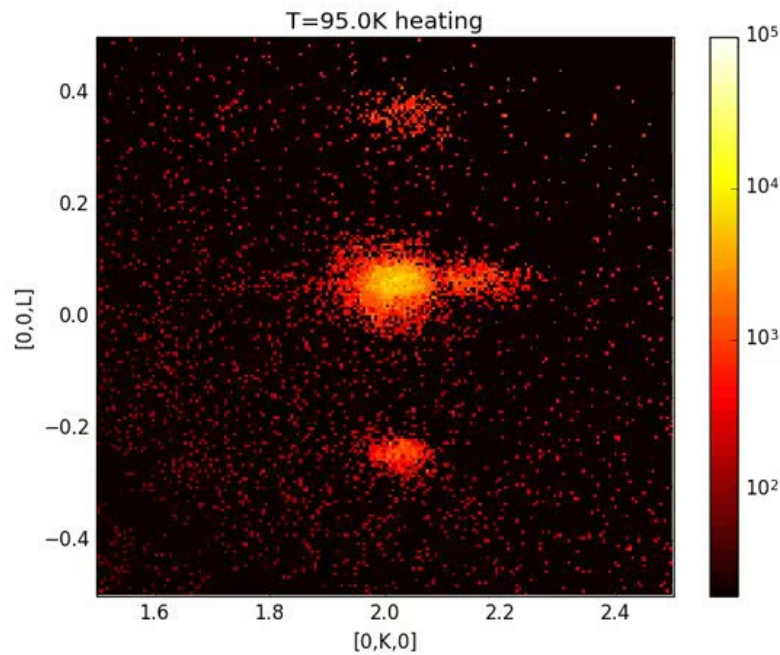
Modulated crystal structure

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

Multidimensional crystallography in *parameter space*

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

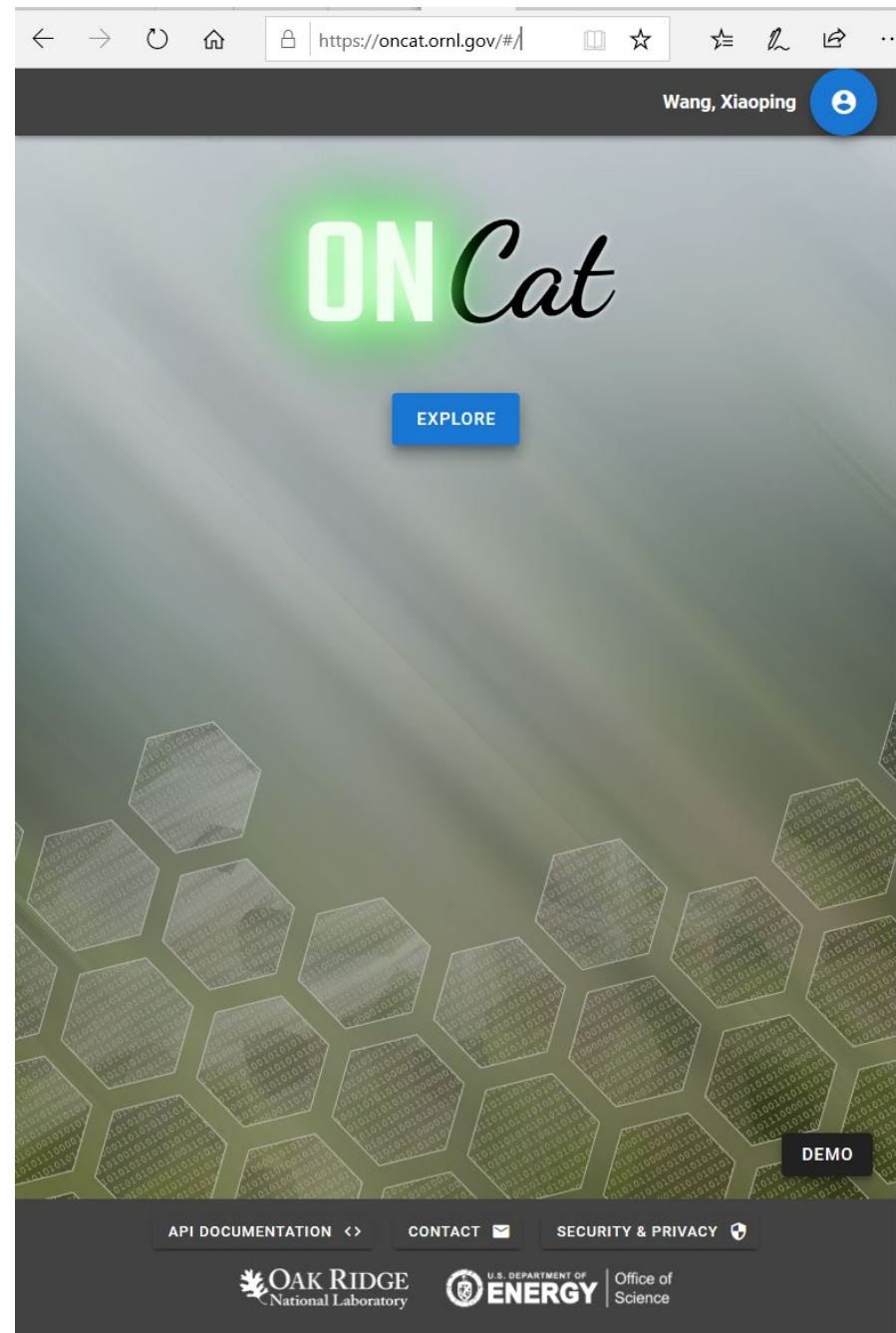
Reconstruction and visualization of real-time data



Temperature dependence of MAPbBr_3 $(2\ 0\ 0)_C$ peak

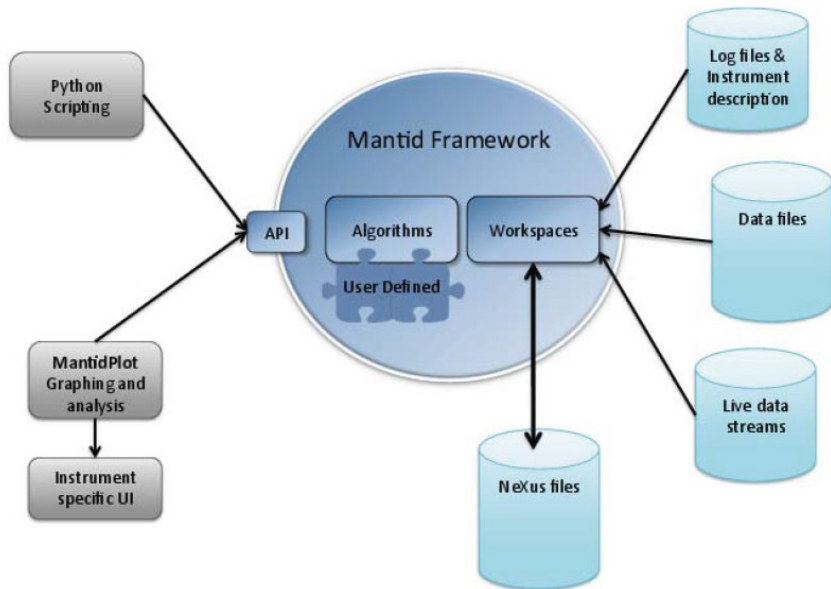
Experiment Log and Data storage

- 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>



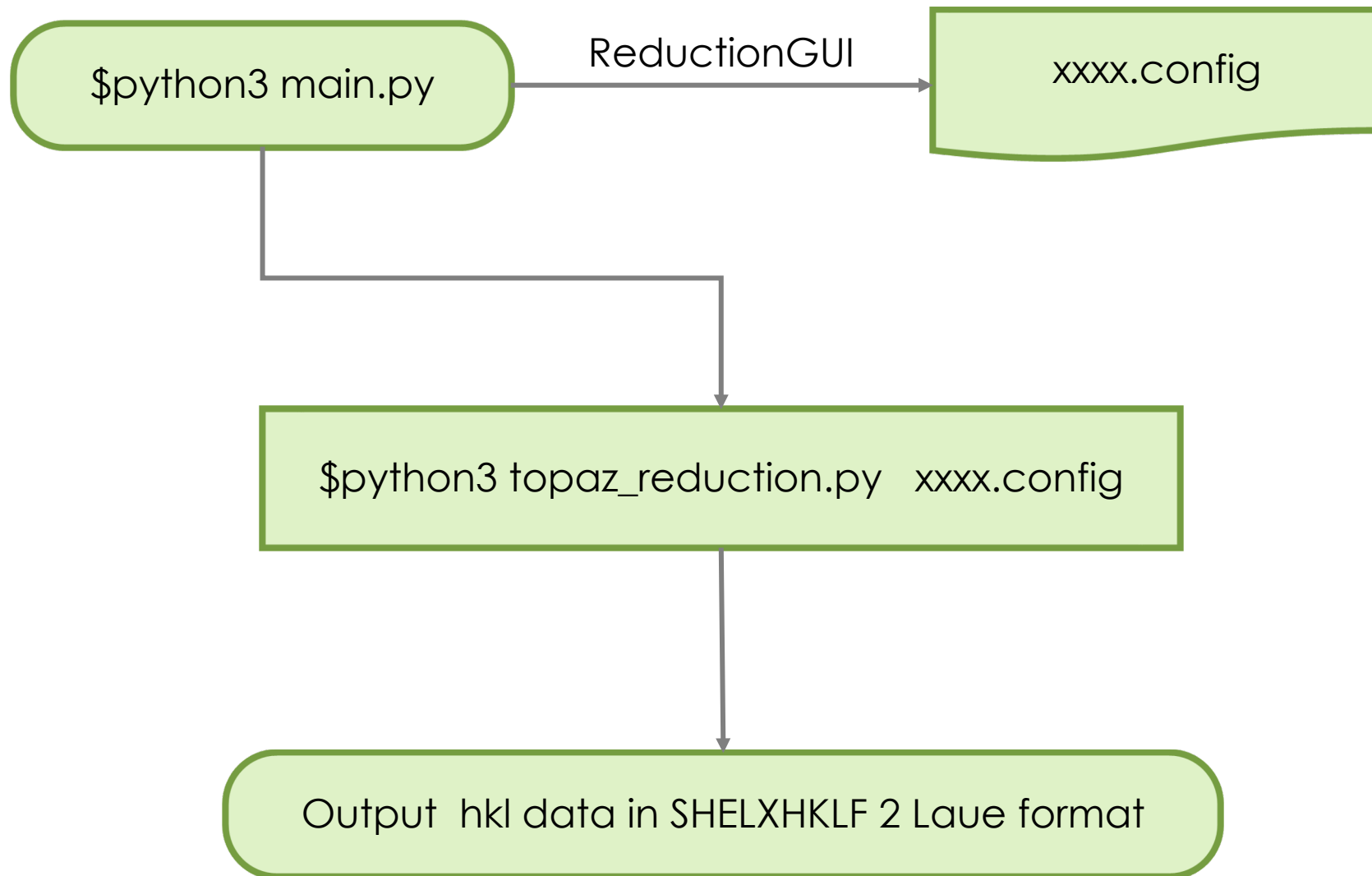
Data Reduction

<https://analysis.sns.gov>



The screenshot shows the "Remote Analysis Service" web client interface. At the top, there is a browser address bar with the URL <https://analysis.sns.gov/>. The page features the SNS logo and the HFIR logo. A prominent "Remote Analysis Service" banner is at the top right. Below it, a section titled "Remote Desktop Capabilities" contains the text: "As a Neutron Sciences user, you can view, analyze and download your data from anywhere. You will be on a machine just like one you use in our Instrument Hall or Target Building. You can work with your data and use the Data Analysis tools provided. To get started using our webclient click the "Launch Session" button below. For more information about different ways to access your data, please see the "Connection Options" section below." A red arrow labeled "Web client" points to the "Launch Session" button. Below this is a "Connection Options" section with a row of icons: a hand, a terminal, a laptop, a terminal with a penguin, a yellow duck, and a padlock. A red box highlights the terminal, penguin, and duck icons. A red arrow labeled "ThinLinc client" points to the terminal icon, and another red arrow labeled "SSH" points to the penguin icon. A red arrow labeled "FTP" points to the duck icon. Below the icons, the text reads: "Mouse over one of the icons above for more information". At the bottom, contact information is provided: "For assistance connecting to the Analysis servers or accessing your data, please contact Linux Support: linux@support.sns.gov or call [865-309-4649](tel:865-309-4649) for urgent requests."

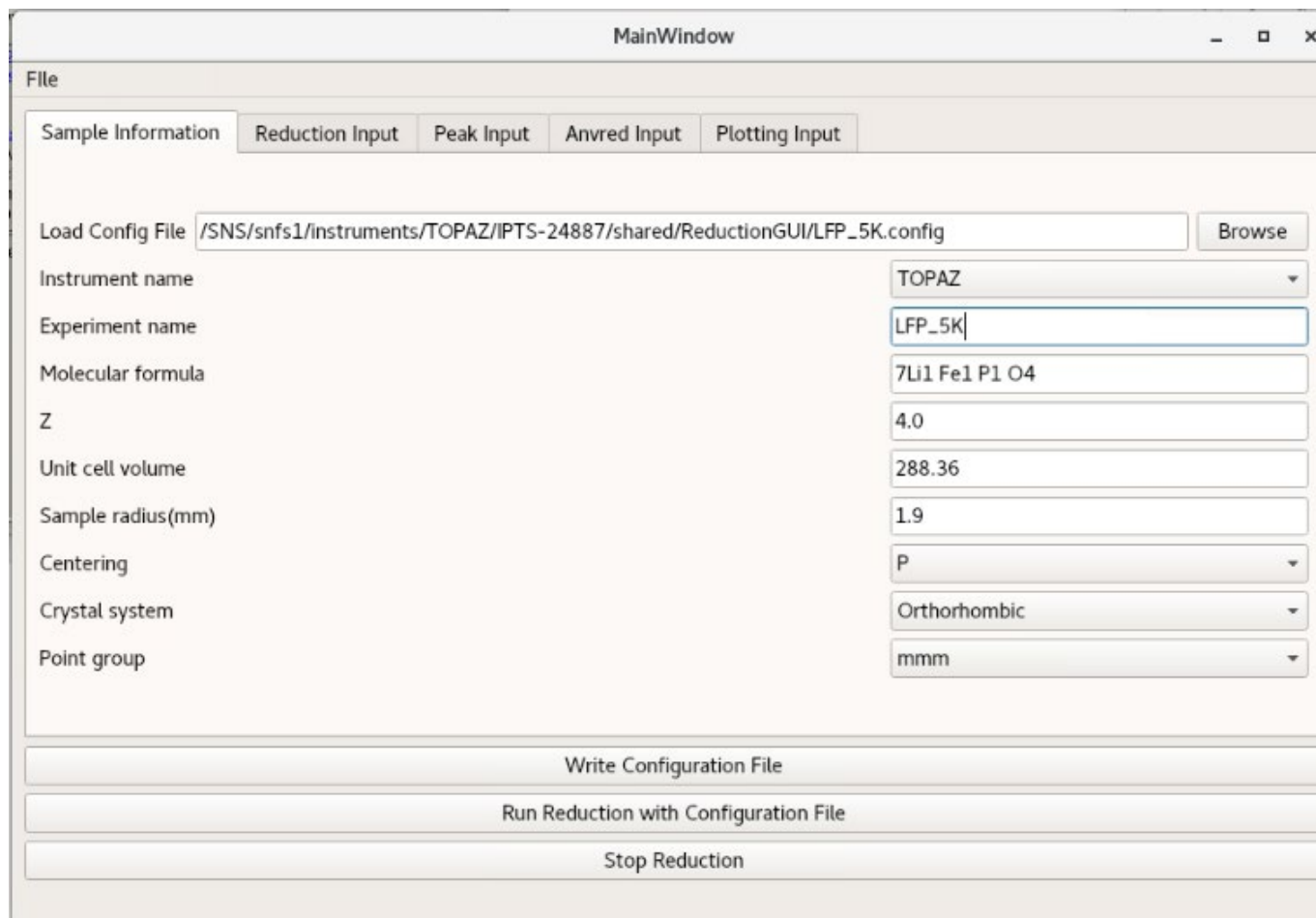
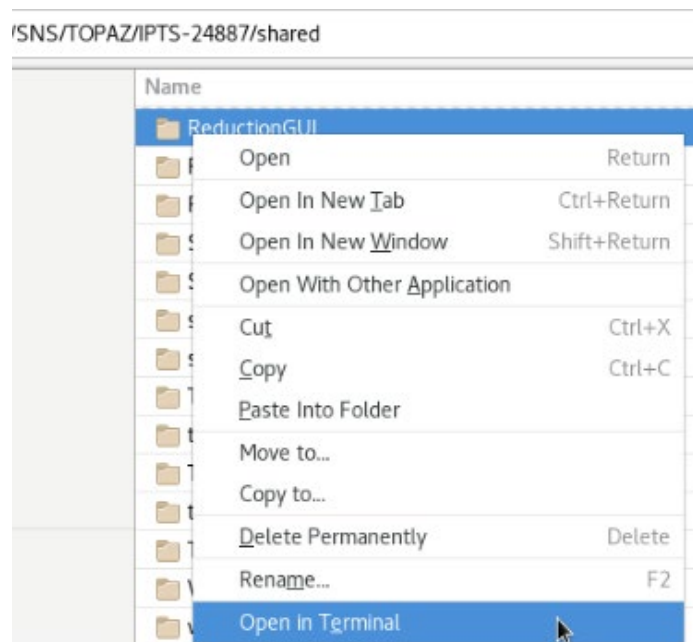
TOPAZ Data Reduction



TOPAZ Data Reduction GUI

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

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



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-xxxx **./ReductionGUI** subfolder:

```
$python3 topaz_reduction.py xxx.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:

```
$python3 topaz_reduction_combinefiles.py xxx.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:

```
$python3 topaz_reduction_anvred.py xxx.config
```

TOPAZ data analysis software

- ✓ **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>
- **FullProf** <https://www.ill.eu/sites/fullprof/>

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&
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- ✓ **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

[Workshop on Symmetry and Superspace Approach to Modulated Crystal Structures](#) (Oak Ridge 2019)

A Virtual Tour of TOPAZ at SNS

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

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Spallation Neutron Source (SNS)
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TOPAZ BL-12

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Thank you for viewing !

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