

SNS BL-12 TOPAZ

Single crystal neutron
Time of Flight Laue diffractometer

Xiaoping Wang

Neutron scattering Division

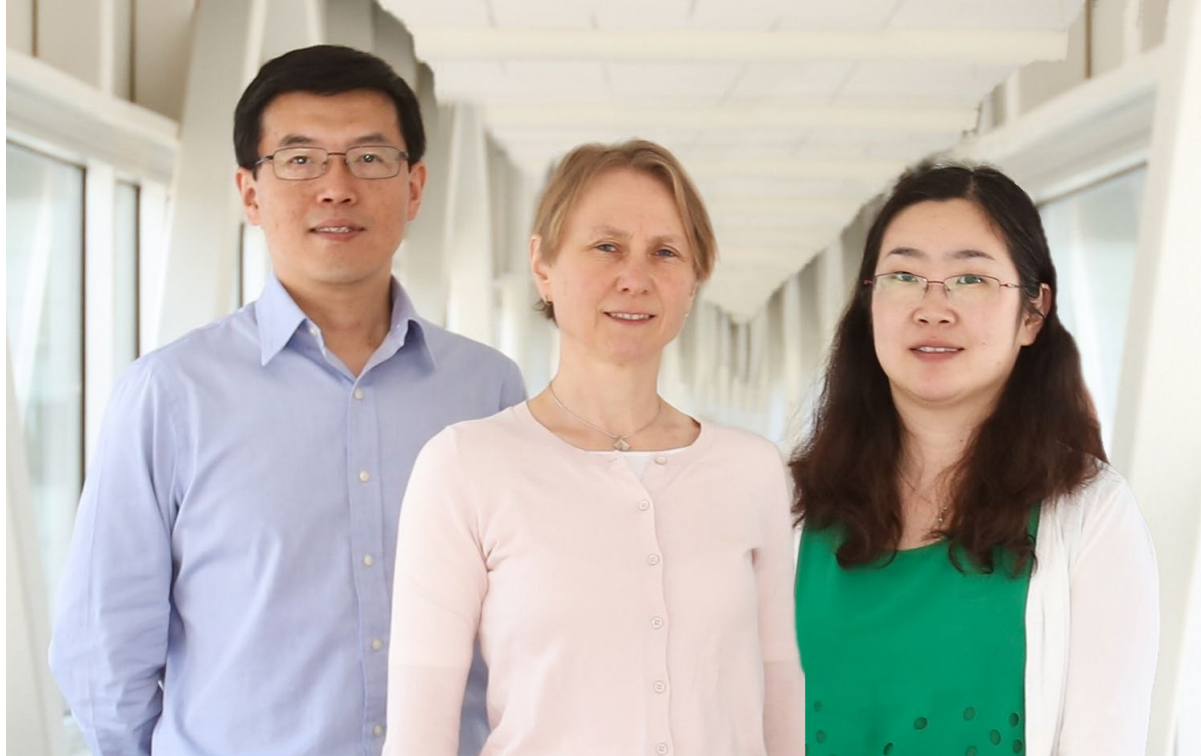


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U.S. DEPARTMENT OF
ENERGY

TOPAZ Instrument Team

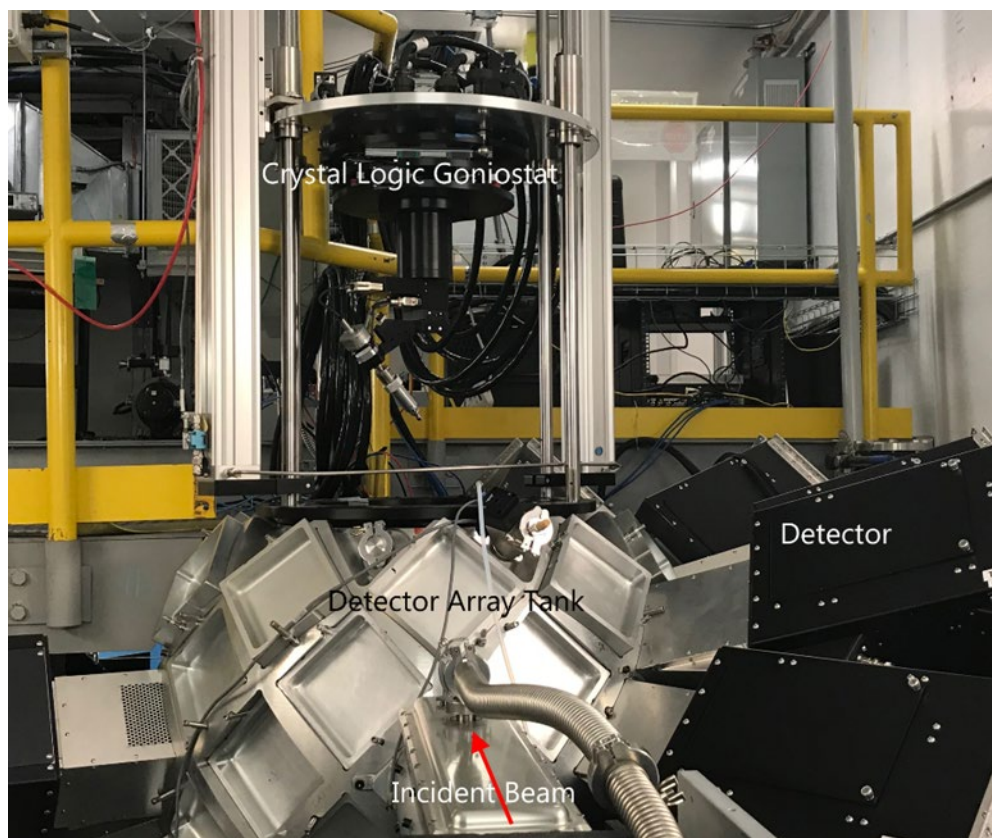


Xiaoping Wang, Christina Hoffmann, Helen He

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°

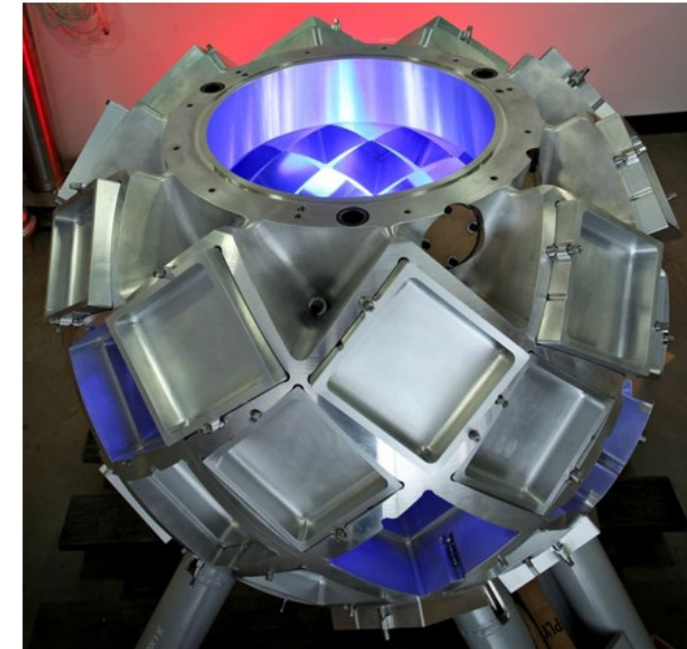
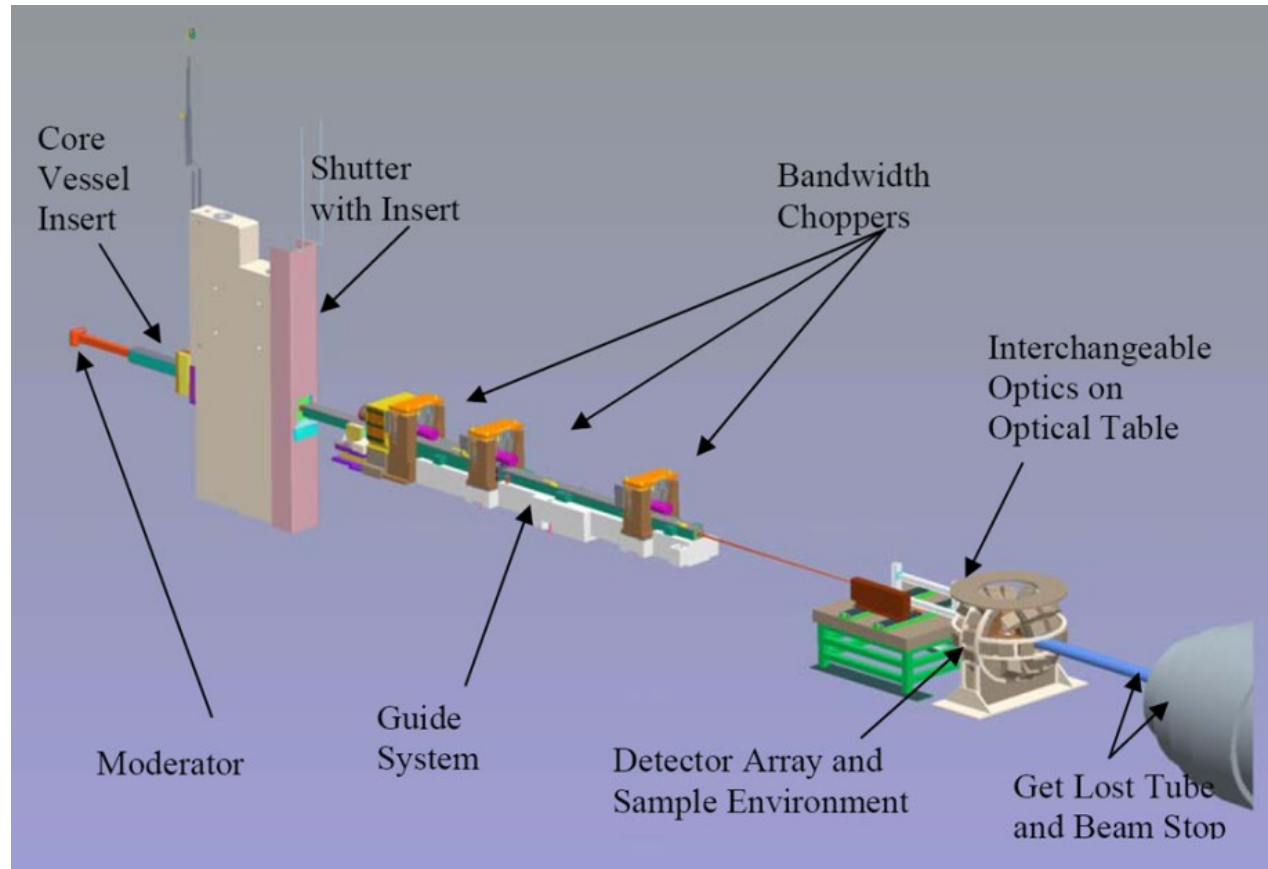
Sample Environment

CryoStream 700 Plus: 90K – 450K

Electric Field

Cryogenic goniometer 5K – 300 K (2020)

Original Design



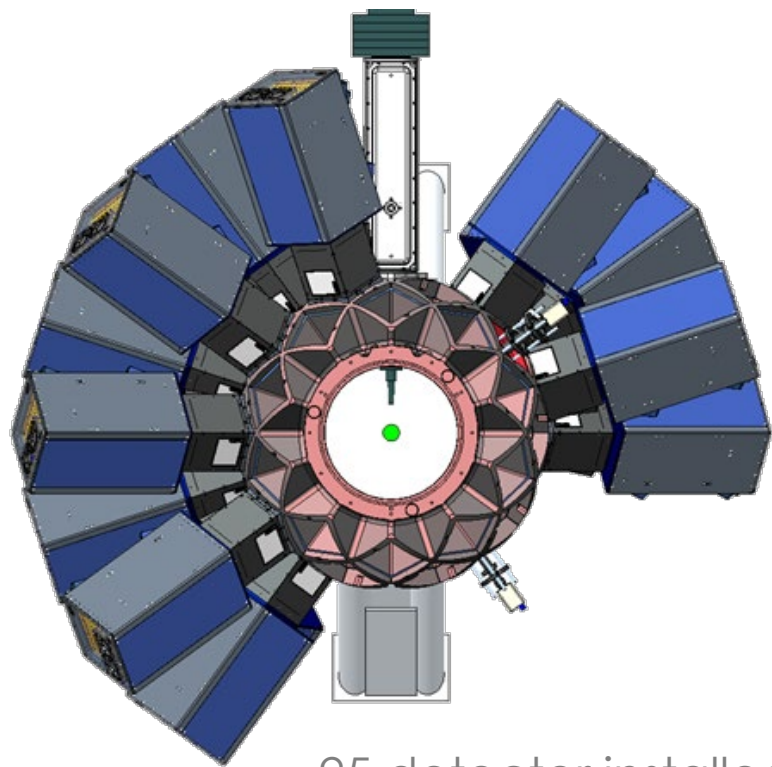
48

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

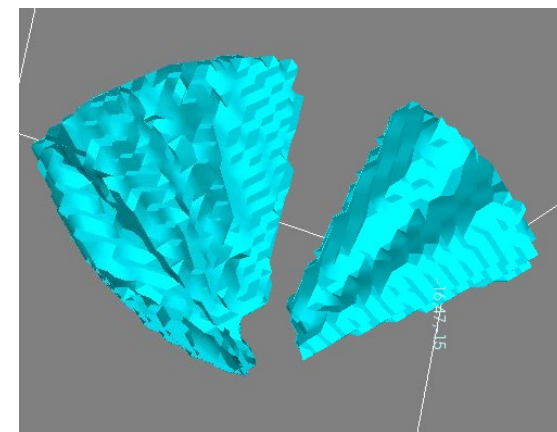
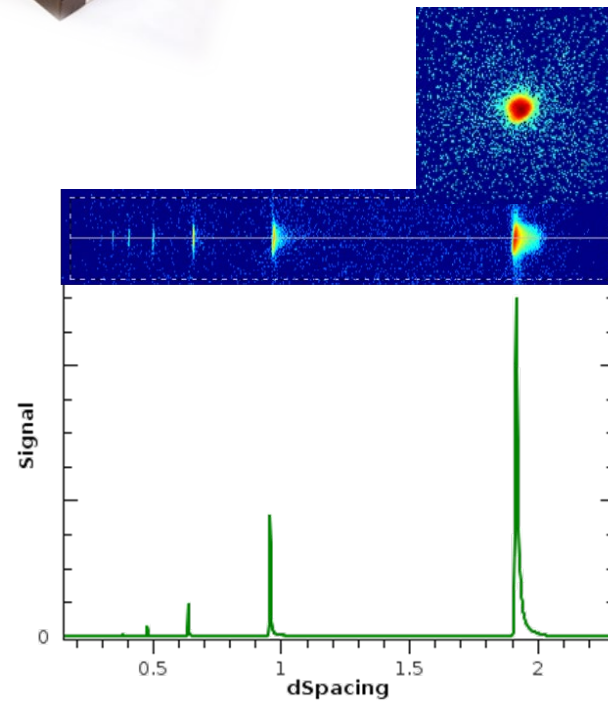
Data are recorded in neutron event mode

- 3D Continuous Q space mapping

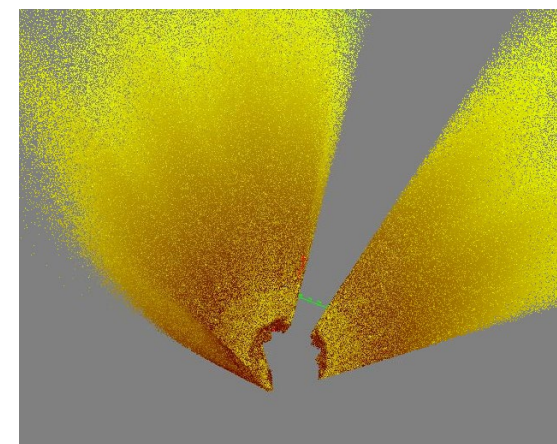
Anger Camera



25 detector installed

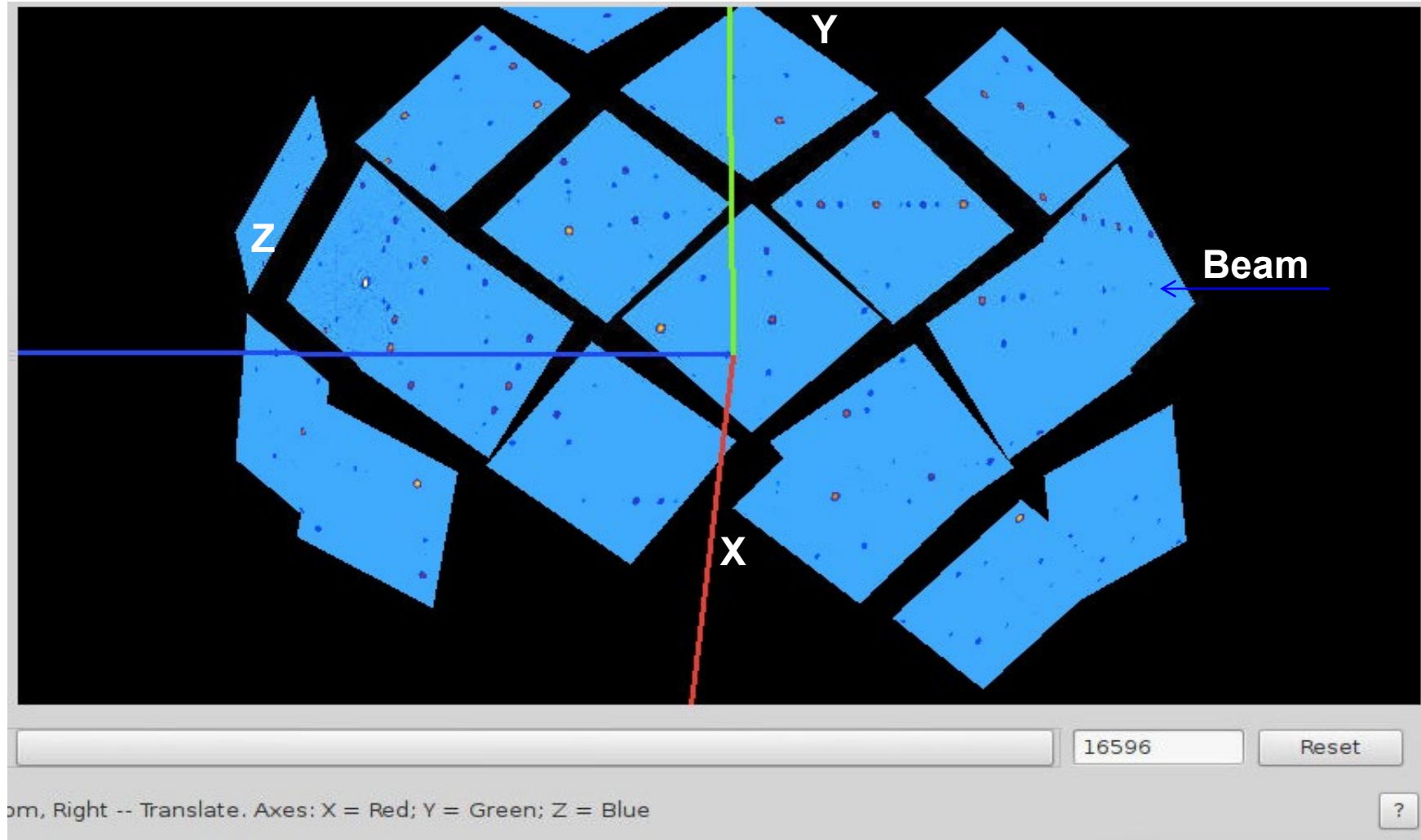


3D Q Coverage, simulated
CrystalPlan

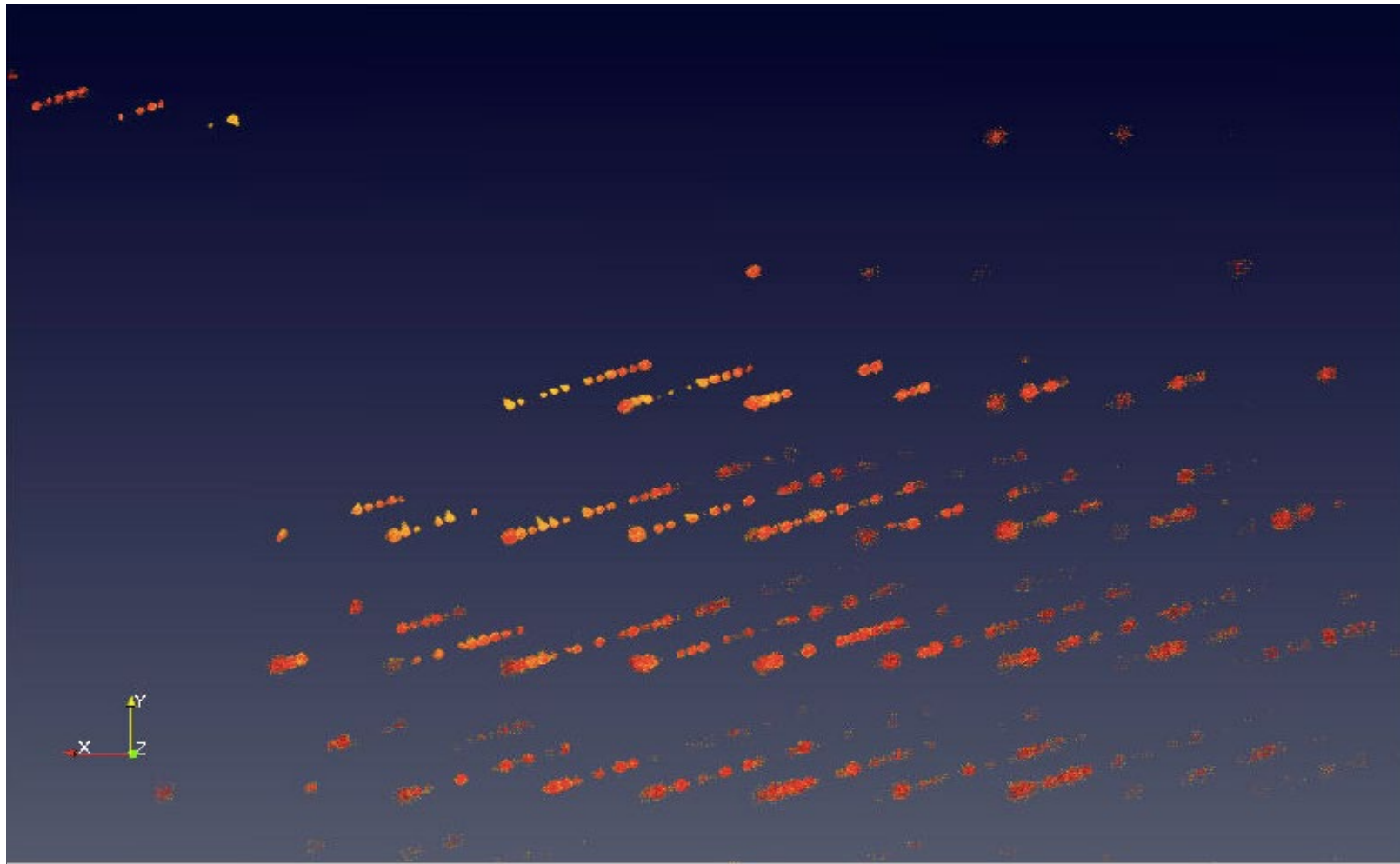


3D Q coverage, measured
ISAWEV

Single crystal peaks on 2D detector space



Single crystal peaks in 3D Q space

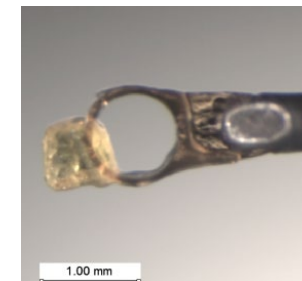


TOPAZ Ambient Goniometer

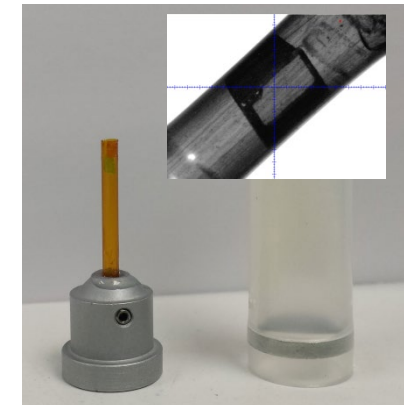


- A **two-axis** goniometer
 - Omega, phi with chi fixed at 135°.
 - Both omega and phi are fitted with sliprings that allow unlimited 360° rotational motion.
 - The omega and phi rotation axes are separated by 45 degrees.
 - Sample mount

MiTeGen loop (1 mm ϕ)



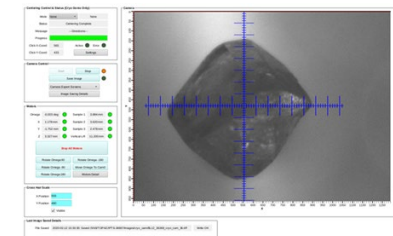
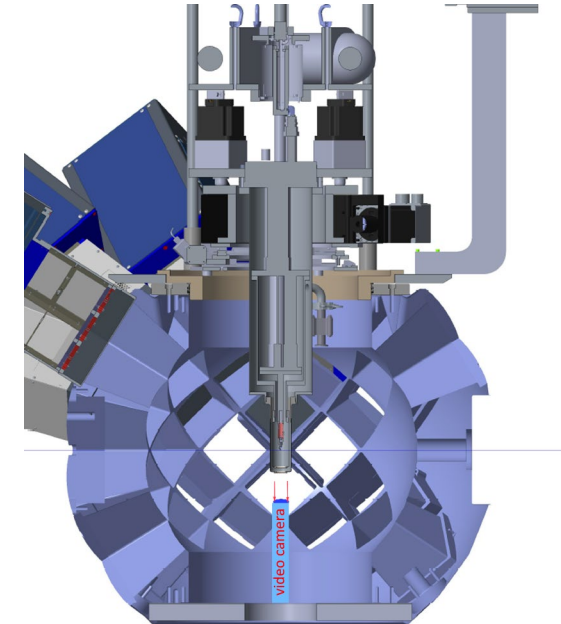
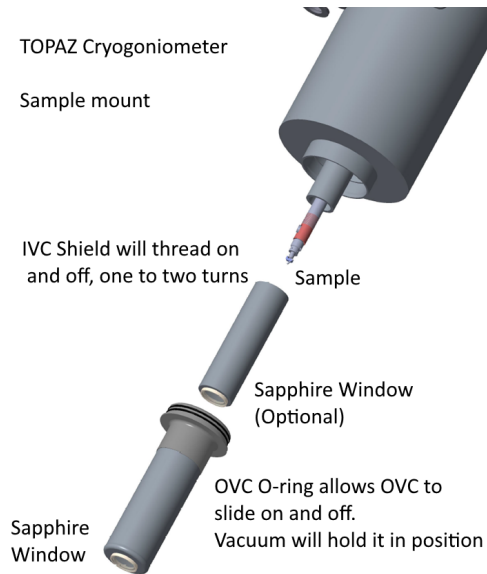
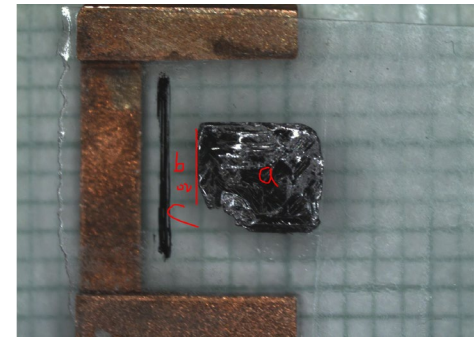
Glued or Coated with perfluorinated grease



TOPAZ Cryogenic Goniometer

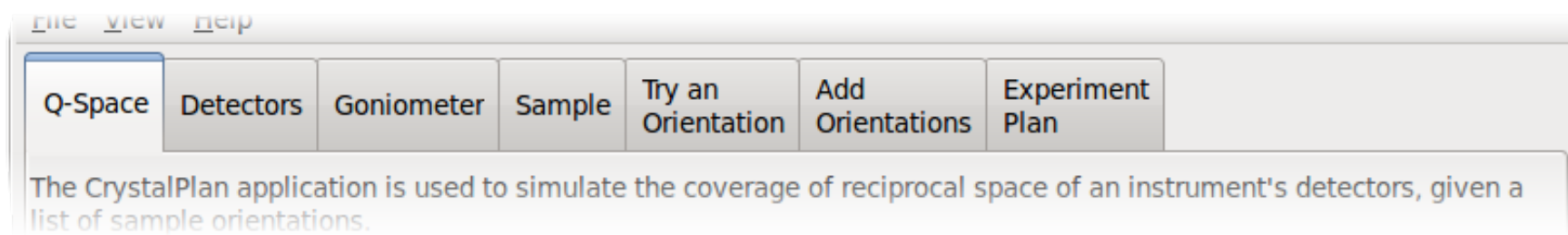
One axis of rotation (360°) with precision motor controls to center, orient, and hold sample in temperature range 5 K – 300 K.

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

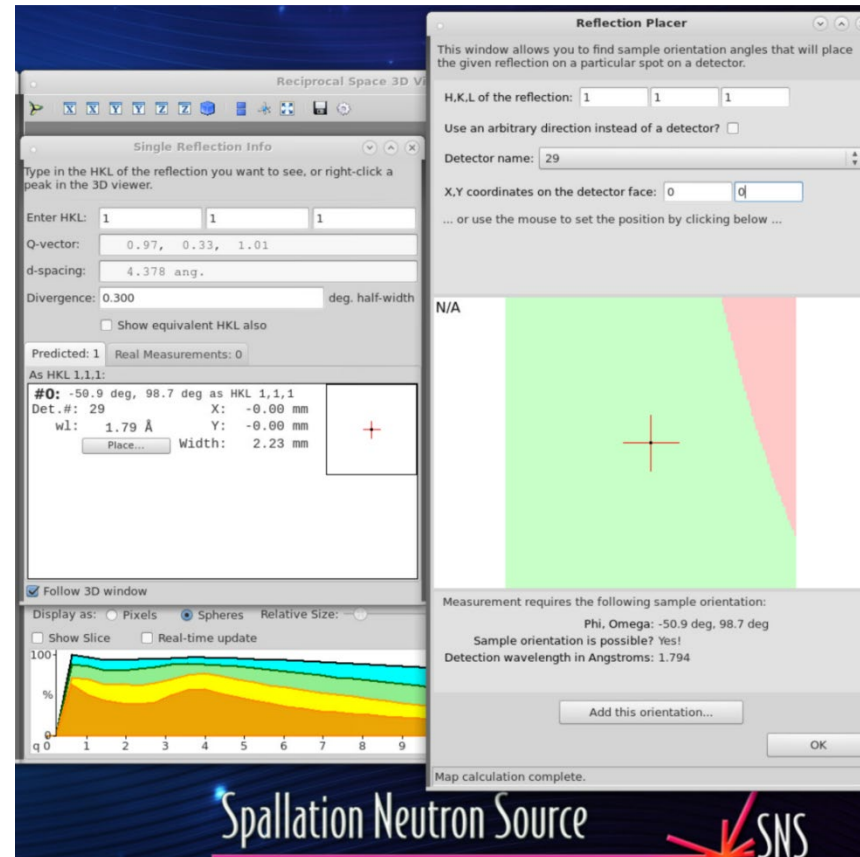


Crystal Plan

- 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 Main Window with the Reflection Placer dialog box open. The Reflection Placer window shows the H,K,L of the reflection as 2, 0, -2 and the detector name as 18. It also displays the X,Y coordinates on the detector face as 35.27, -8.63. The Single Reflection Info window shows the predicted and real measurements for the reflection, including the H,K,L, detector number, wavelength, and width.

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?	Phi (deg)	Stopping Criterion	Criterion Value	Comment
1	X	92.00	proton charge (pC)	5.0	-200Det49:omega
2	X	-88.00	proton charge (pC)	5.0	200Det29:020Det
3	X	-90.66	proton charge (pC)	5.0	200Det29:020Det
4	X	8.02	proton charge (pC)	5.0	002Det39:3.0-13.7
5	X	-171.92	proton charge (pC)	5.0	00-2Det39:-177.5
6	X	159.93	proton charge (pC)	5.0	111Det20
7	X	-18.24	proton charge (pC)	5.0	-111Det59
8	X	56.30	proton charge (pC)	5.0	-1-11Det39
9	X	134.59	proton charge (pC)	5.0	-1-1-1Det39
10	X	-119.89	proton charge (pC)	5.0	1-11Det20
11	X	36.05	proton charge (pC)	5.0	1-1-1Det20
12	X	-127.50	proton charge (pC)	5.0	11-1Det39
13	X	-167.85	proton charge (pC)	5.0	-11-1De49
14	X	-69.16	proton charge (pC)	5.0	220Det39
15	X	109.73	proton charge (pC)	5.0	-2-20Det39
16	X	-23.01	proton charge (pC)	5.0	202Det29
17	X	157.08	proton charge (pC)	5.0	-20-2Det49
18	X	139.39	proton charge (pC)	5.0	0-2-2Det28
19	X	-39.32	proton charge (pC)	5.0	022Det48
20	X	22.84	proton charge (pC)	5.0	0-22Det18
21	X	-156.73	proton charge (pC)	5.0	02-2Det58
22	X	14.10	proton charge (pC)	5.0	-202Det58
23	X	-166.63	proton charge (pC)	5.0	20-2Det18

Estimated run time: 1.150e+02 pC proton charge, approx 00m 00s (at 1 MW accelerator power).

Reflection Placer

This window allows you to find sample orientation angles that will place the given reflection on a particular spot on a detector.

H,K,L of the reflection: 2 0 -2

Use an arbitrary direction instead of a detector? ☐

Detector name: 18

X,Y coordinates on the detector face: 35.27 -8.63

... or use the mouse to set the position by clicking below ...

-27.79, 71.02

Measurement requires the following sample orientation:
Phi: -166.6 deg
Sample orientation is possible? Yes!
Detection wavelength in Angstroms: 3.037

Map calculation complete.

Single Reflection Info

Type in the HKL of the reflection you want to see, or right-click a peak in the 3D viewer.

Enter HKL: 0 -2 0

Q-vector: 0.00, -1.74, 0.00

d-spacing: 3.614 ang.

Divergence: 0.300 deg. half-width

☐ Show equivalent HKL also

Predicted: 3 Real Measurements: 0

As HKL 0,-2,0:

#0: 92.0 deg as HKL 0,-2,0
Det.#: 19 X: -19.89 mm Y: 33.38 mm
w1: 2.64 Å Width: 2.40 mm

#1: 109.7 deg as HKL 0,-2,0
Det.#: 29 X: -1.75 mm Y: 61.26 mm
w1: 0.99 Å Width: 2.26 mm

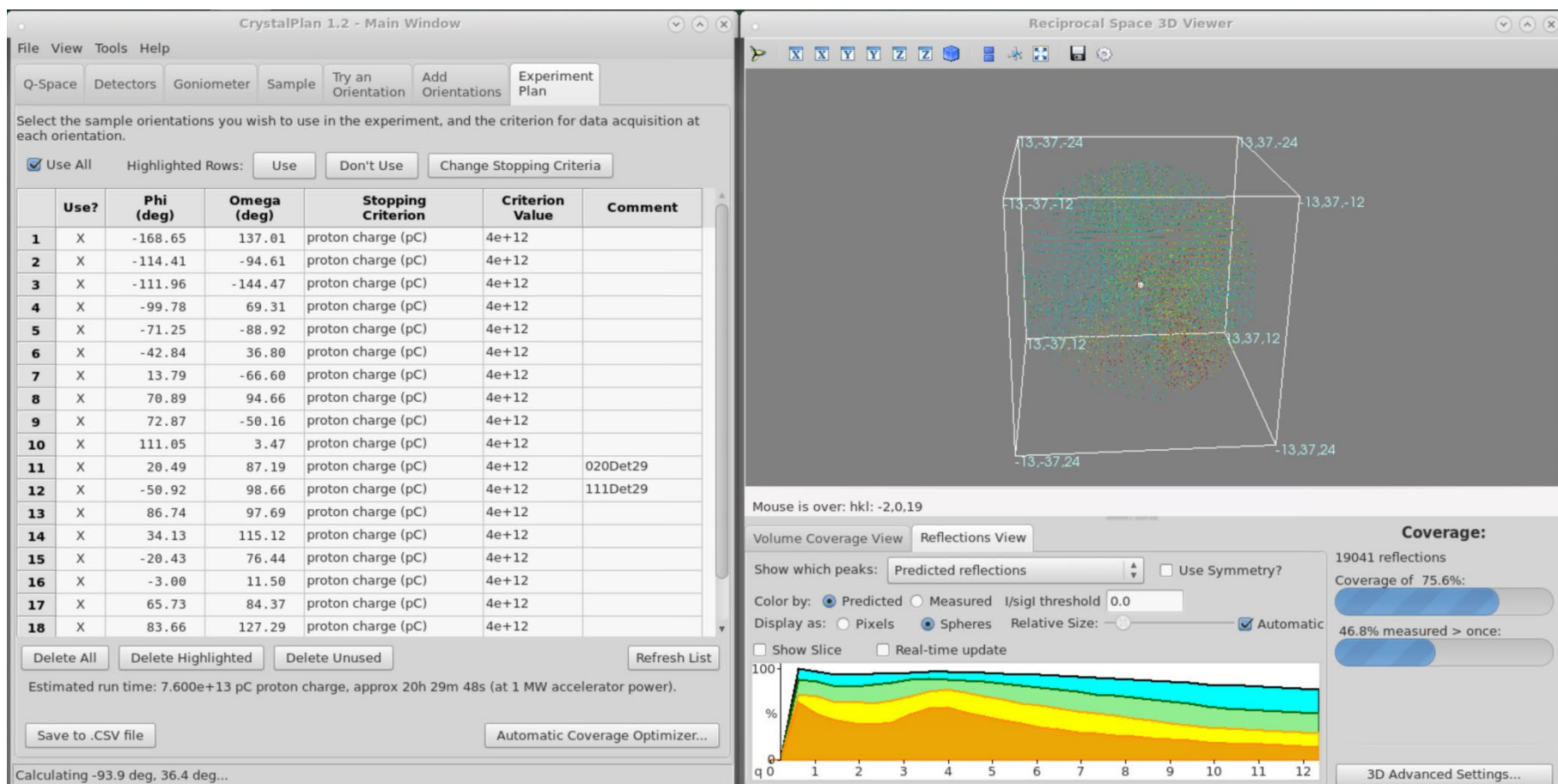
#2: -39.3 deg as HKL 0,-2,0
Det.#: 20 X: 65.25 mm Y: 15.60 mm
w1: 2.02 Å Width: 2.27 mm

☒ Follow 3D window

Coverage:

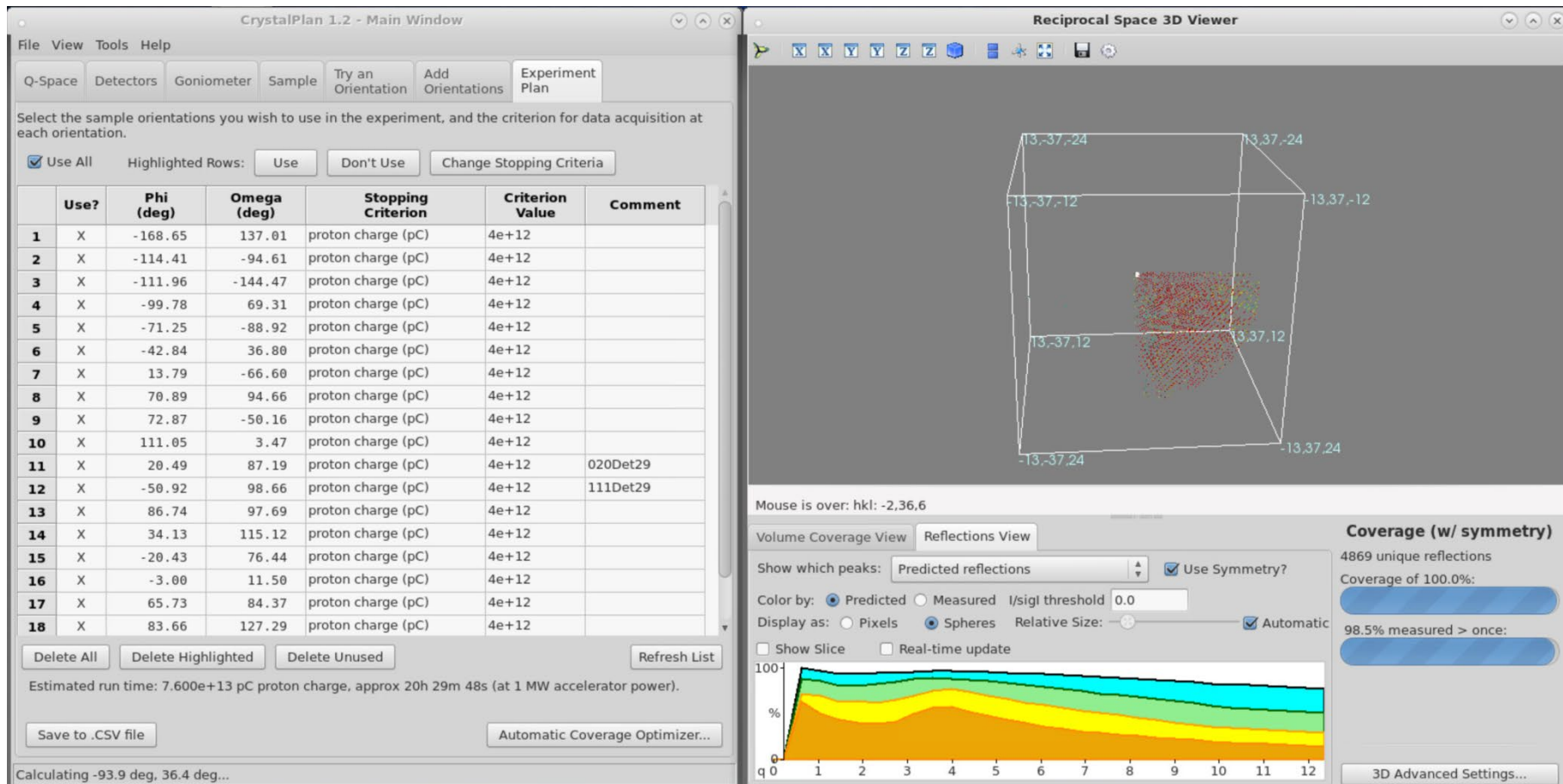
3143 reflections
Coverage of 71.3%
52.2% measured > once:

CrystalPlan – Detector coverage



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



Zikovskiy 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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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

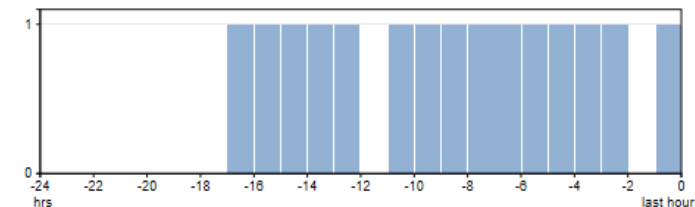


<https://neutrons.ornl.gov/topaz>

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

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

TOPAZ Dashboard



TOPAZ Sciences

- **Chemical crystallography**

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

- **Diffuse scattering**

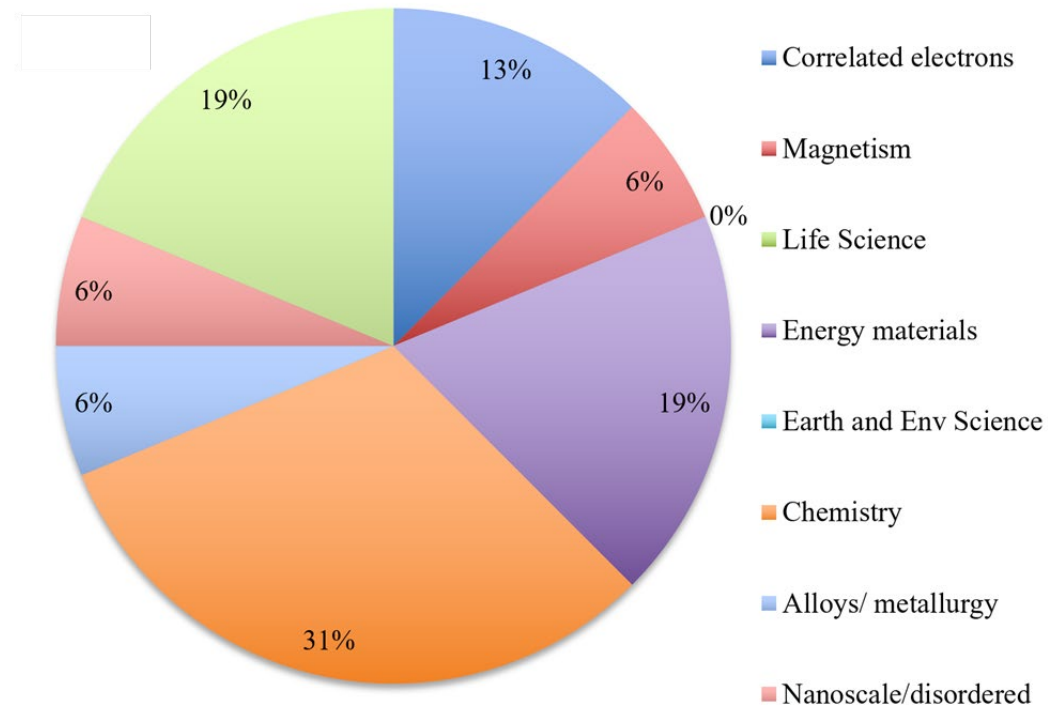
- 3D reciprocal space mapping

- **Magnetism**

- Solve & refine magnetic and nuclear structures

- **Event based parametric study**

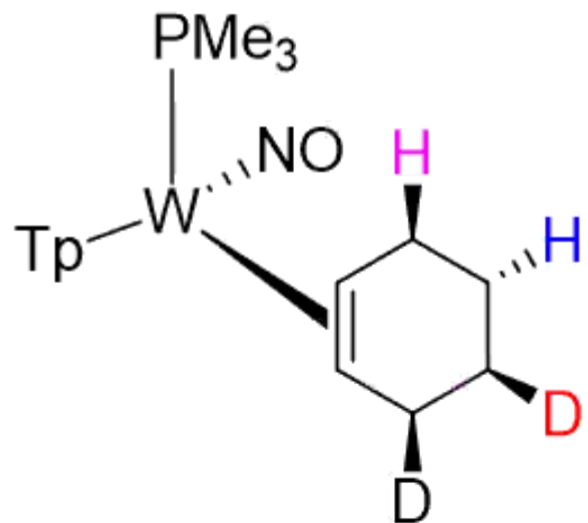
- Applied electric field
- Variable temperature
- Structural phase transitions
- Order parameters



Chemical crystallography

Transition metal-mediated dearomatization.

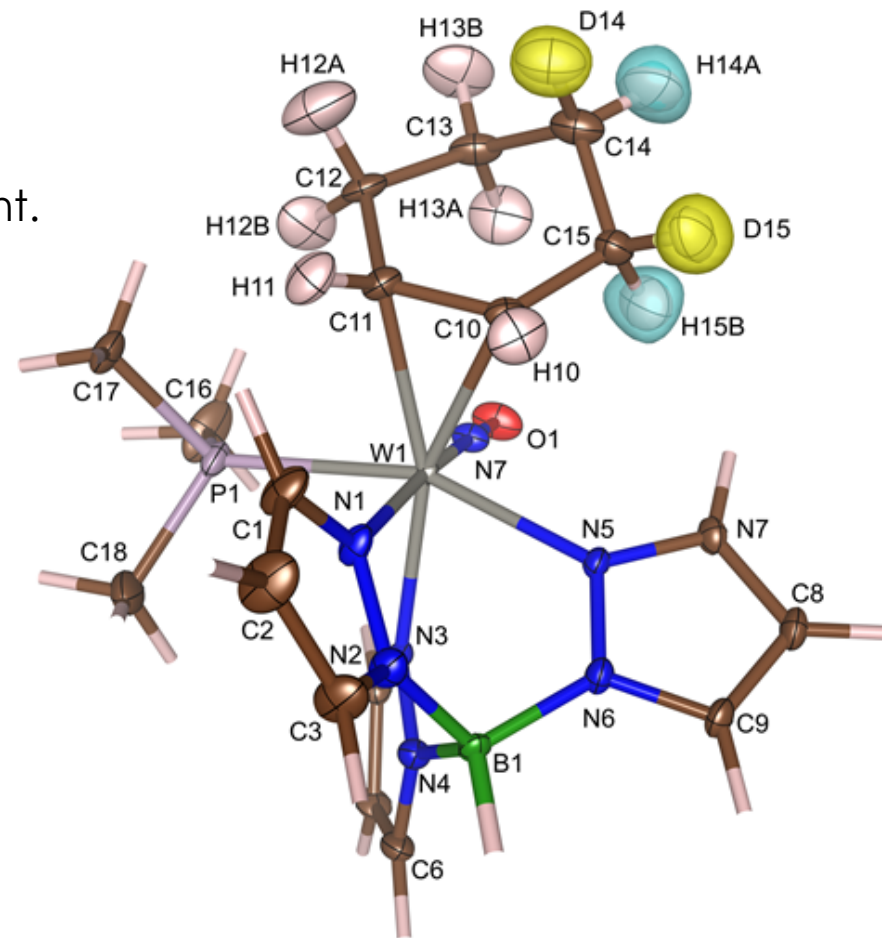
Opened pathways for a new generation of medicines and therapies that incorporate deuterium into the active pharmaceutical ingredient.



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



0.05 mm³



Neutron structure of a d₂ isotopologues of cyclohexene complex.

Single crystal neutron diffraction beyond three dimensions

Modulated crystal structure

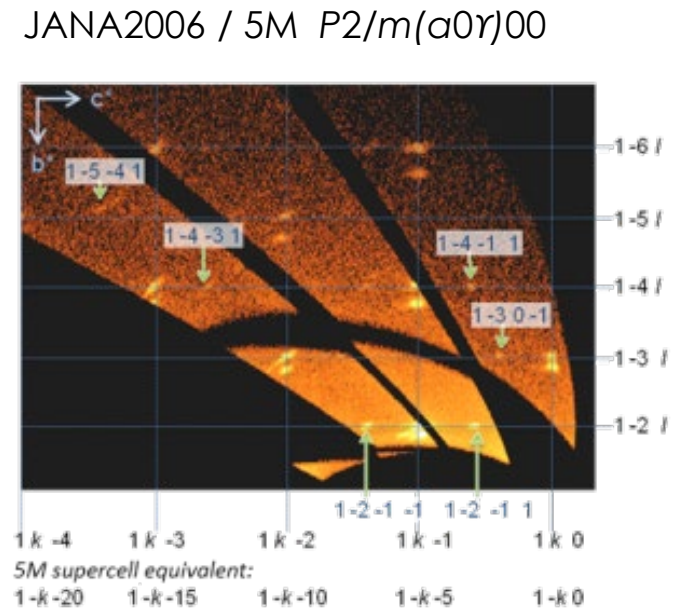
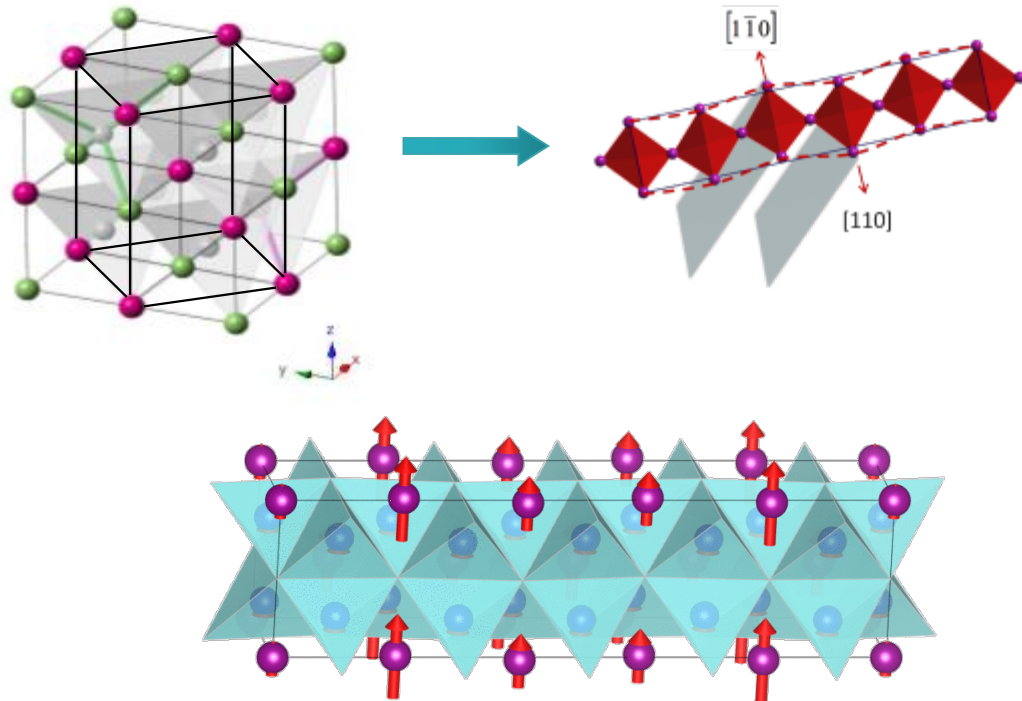
$$\mathbf{Q} = 2\pi(h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^* + m\mathbf{q}_1 + n\mathbf{q}_2 + p\mathbf{q}_3)$$



<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



Single crystal neutron diffraction beyond three dimensions

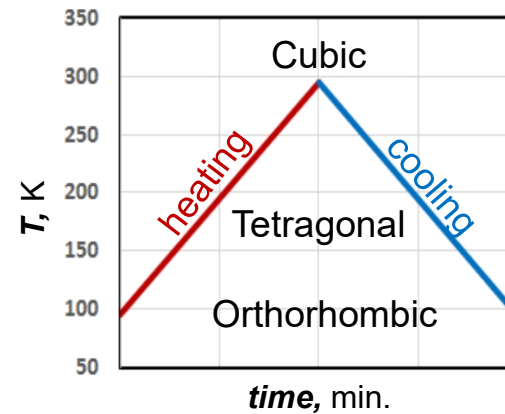
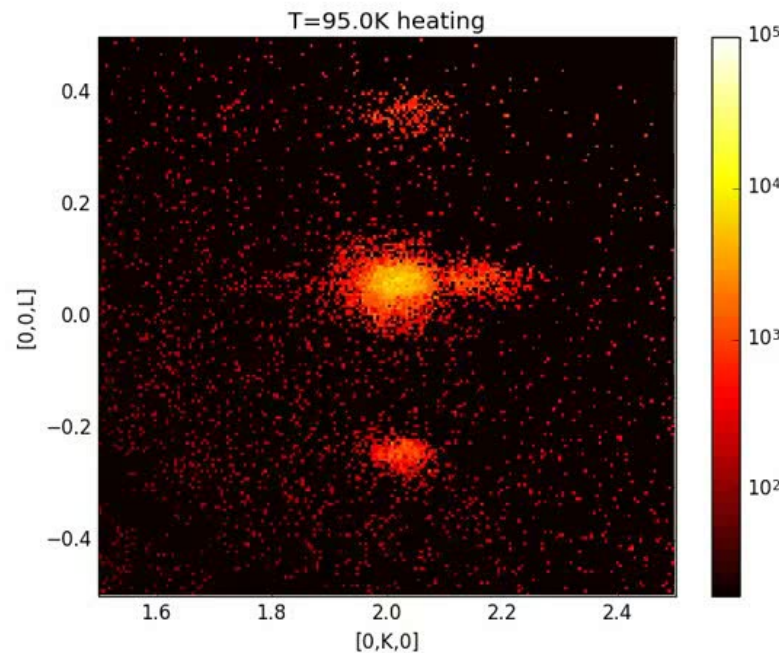
Modulated crystal structure

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

Multidimensional crystallography in *parameter space*

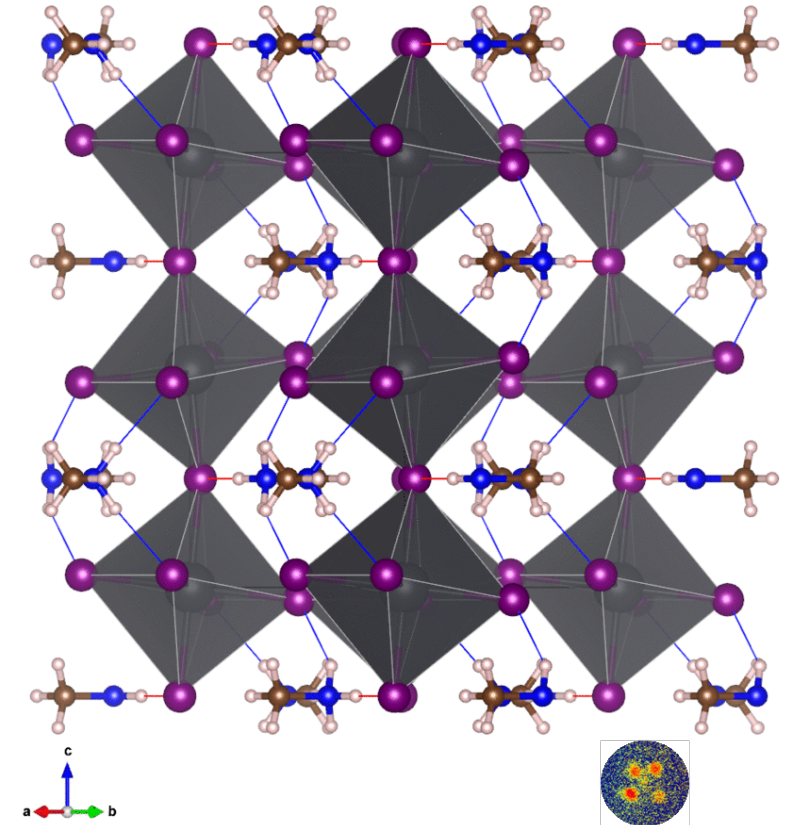
$$\mathbf{Q} + T, E, P, B$$

Reconstruction and visualization of real-time data



Ramping rate 1K per min.

Temperature dependence of MAPbBr_3 (2 0 0)_c peak



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/>

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)

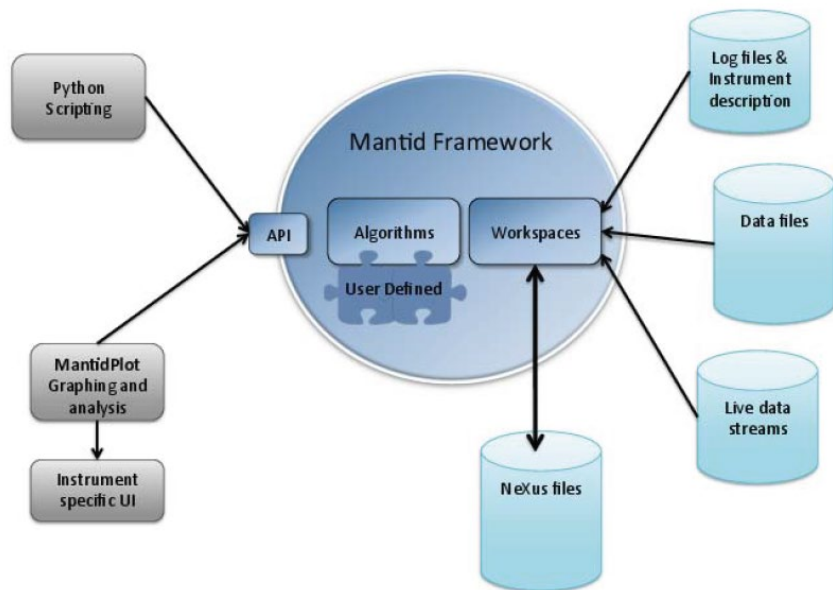
Robert Von Dreele: [Single crystal structure refinement with TOF data in GSAS-II](#) (Argonne, 2016)

• Tutorials

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

Data Reduction

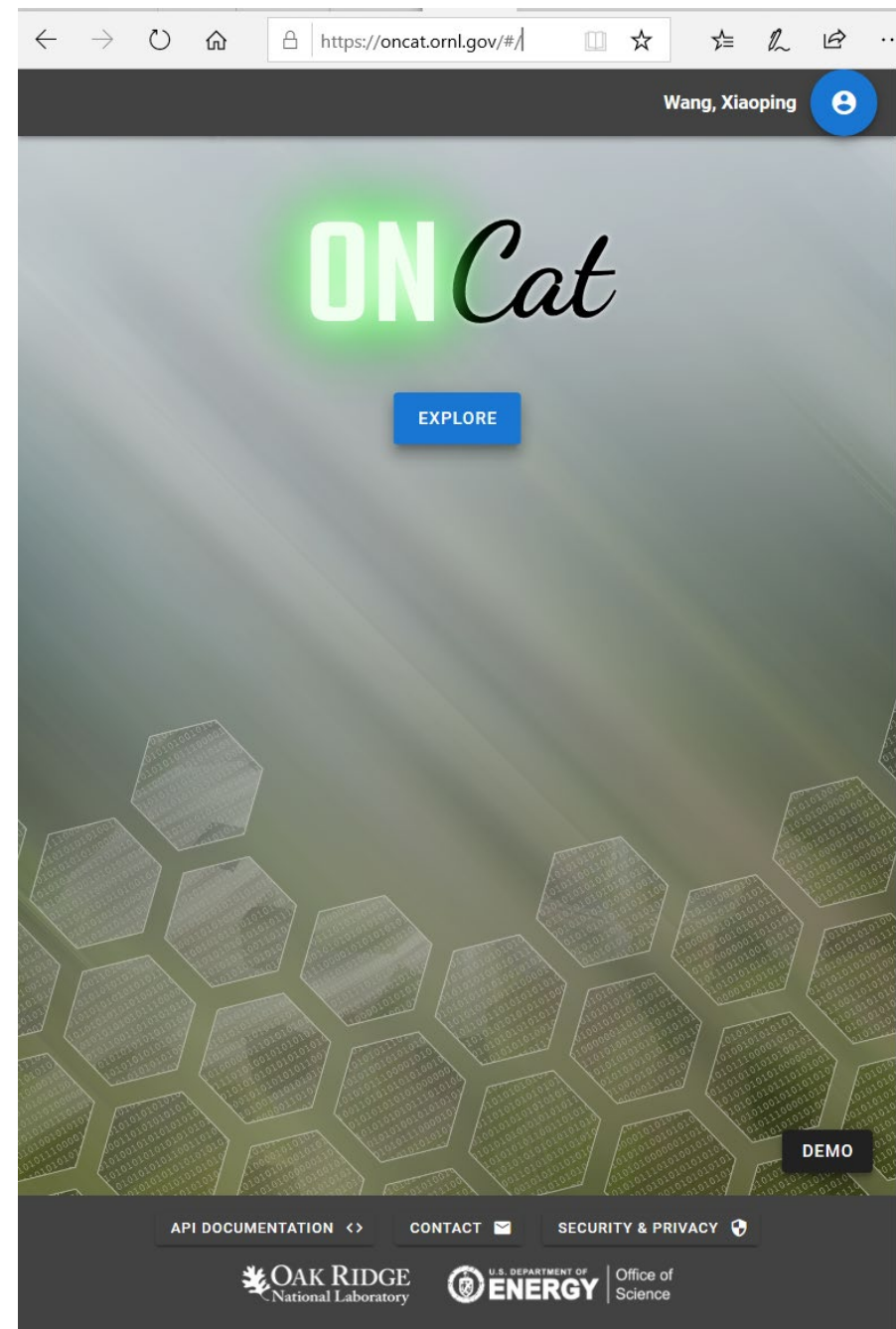
<https://analysis.sns.gov>



A screenshot of the "Remote Analysis Service" web client interface. The browser address bar shows <https://analysis.sns.gov/>. The page features the SNS logo and the HFIR logo. A section titled "Remote Desktop Capabilities" contains text about viewing, analyzing, and downloading data from anywhere. Below this is a "Launch Session" button. A "Connection Options" section displays a row of icons: a hand cursor, a terminal window, a puzzle piece, a terminal window, a penguin (Linux), a duck (Windows), and a padlock (FTP). Red arrows point from the text "Web client", "ThinLinc client", "SSH", and "FTP" to their respective icons. Below the icons is the text "Mouse over one of the icons above for more information". At the bottom, contact information for Linux Support is provided: linux@support.sns.gov or call 865-309-4649 for urgent requests.

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>



Thank you for viewing !

wangx@ornl.gov

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