OAK RIDGE National Laboratory

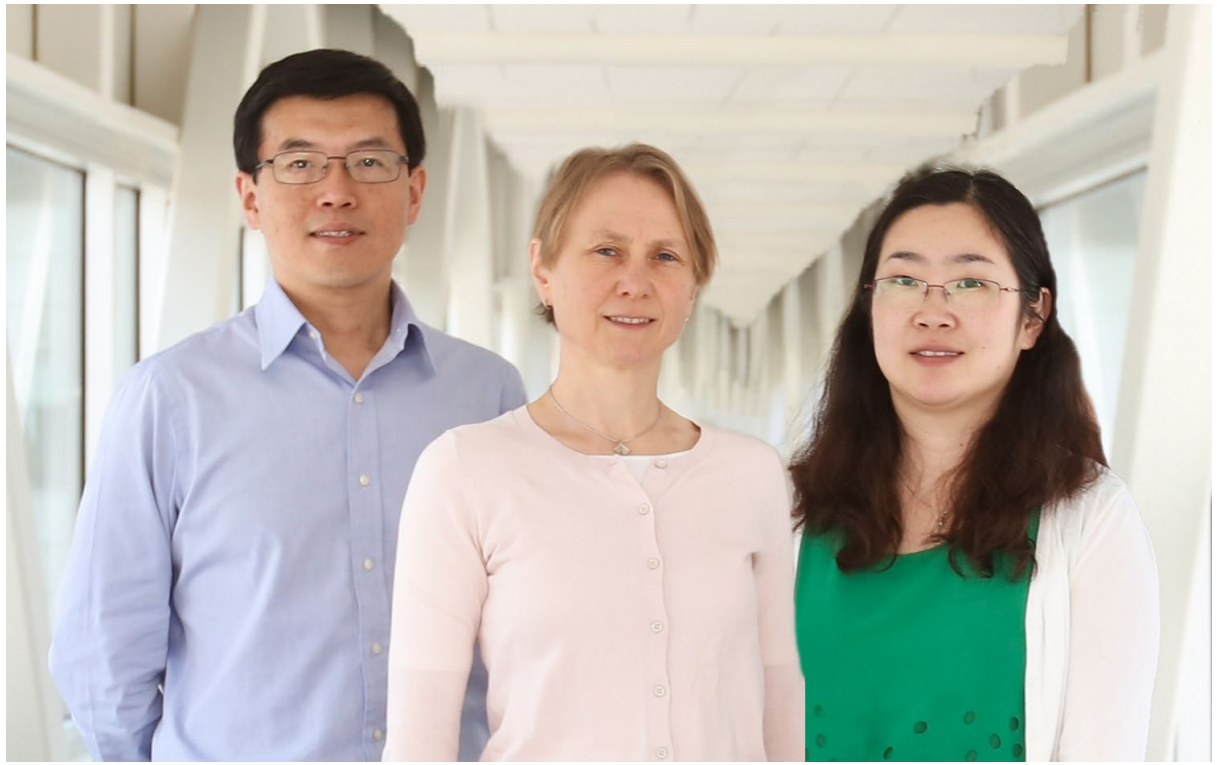
SNS BL-12 TOPAZ

Single crystal neutron Time of Flight Laue diffractometer

Xiaoping Wang and Christina Hoffmann

Neutron scattering Division

TOPAZ Instrument Team



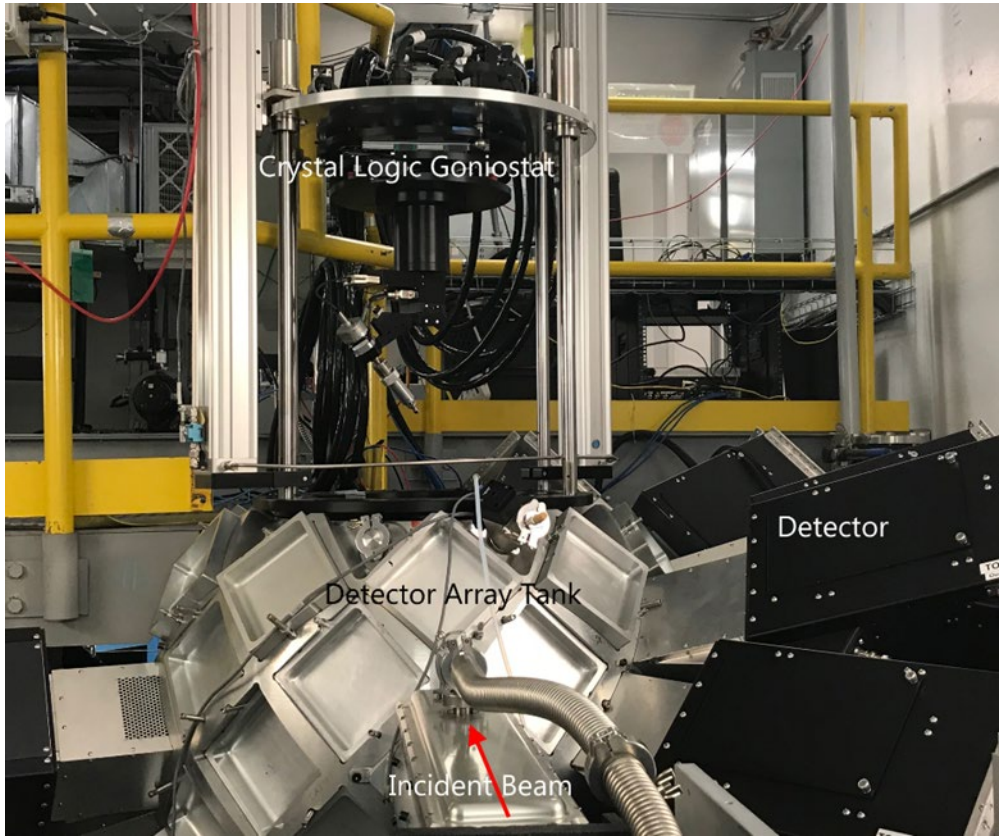
Xiaoping Wang, Christina Hoffmann, Helen He

https://neutrons.ornl.gov/topaz

TOPAZ

A high-resolution single crystal diffractometer

Q_{max} = 25 Å^{-1} d_{min} = 0.25 Å



Sub-Millimeter Sized Crystals

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

Multiple Area Detectors

Solid Angle Coverage: 3.2 ster.

Detector 2θ Coverage: 13.5° - 160°

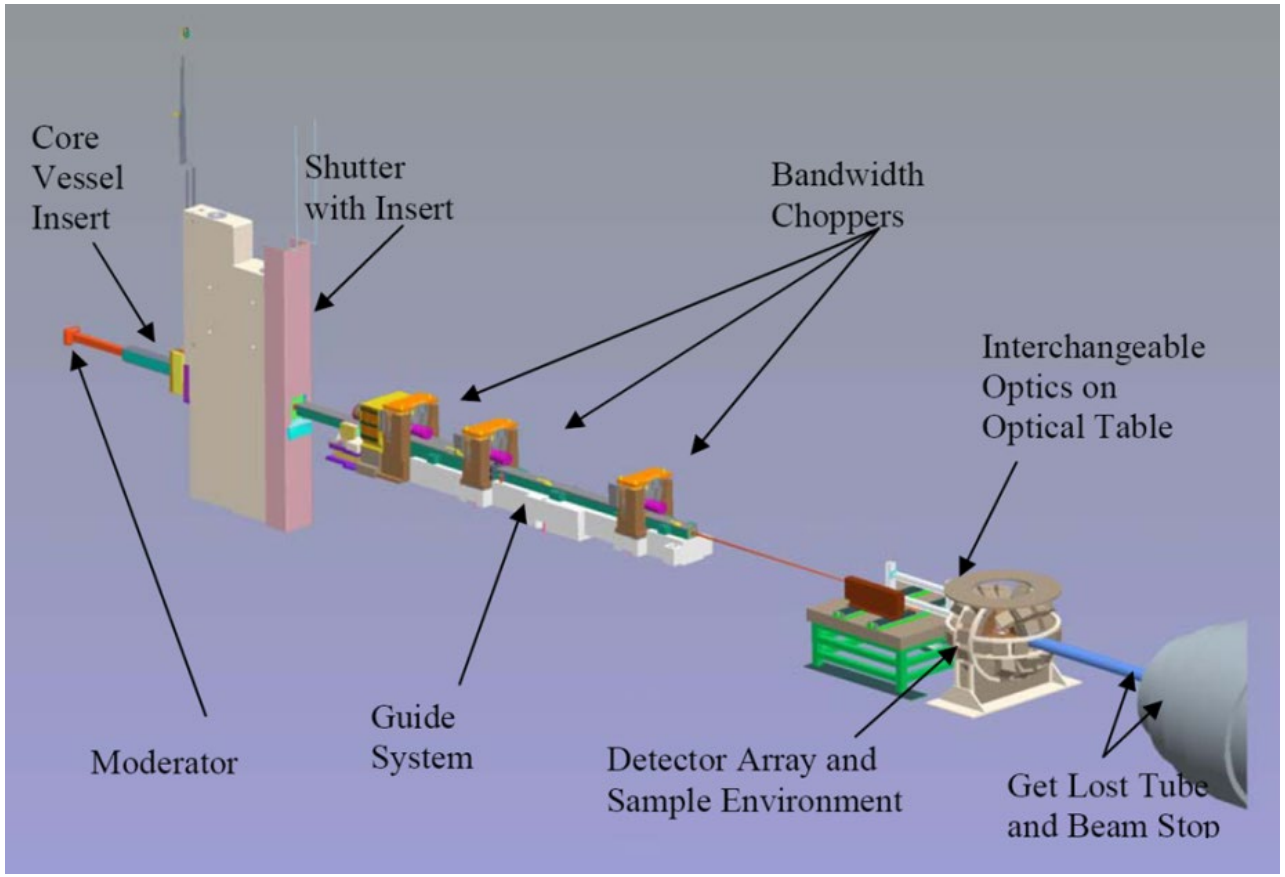
Sample Environment

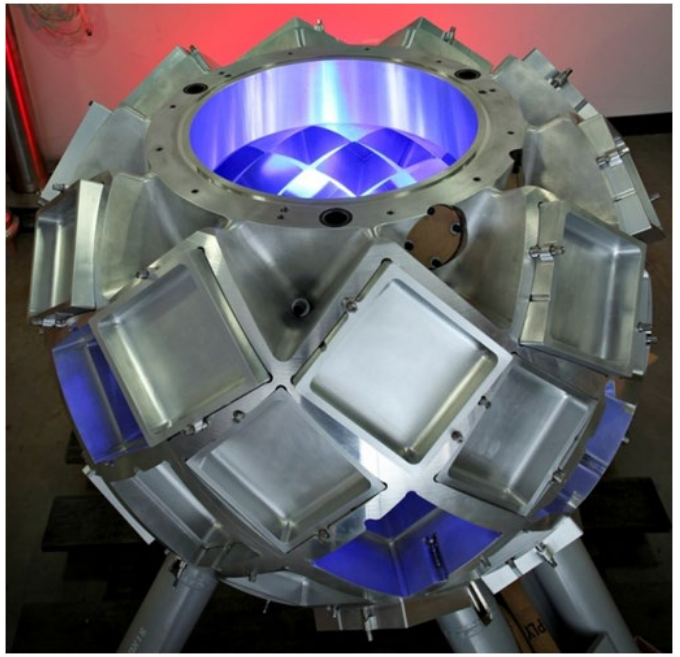
CryoStream 700 Plus: 90K – 450K

Electric Field

Cryogenic goniometer 5K – 300 K (2020)

Original Design



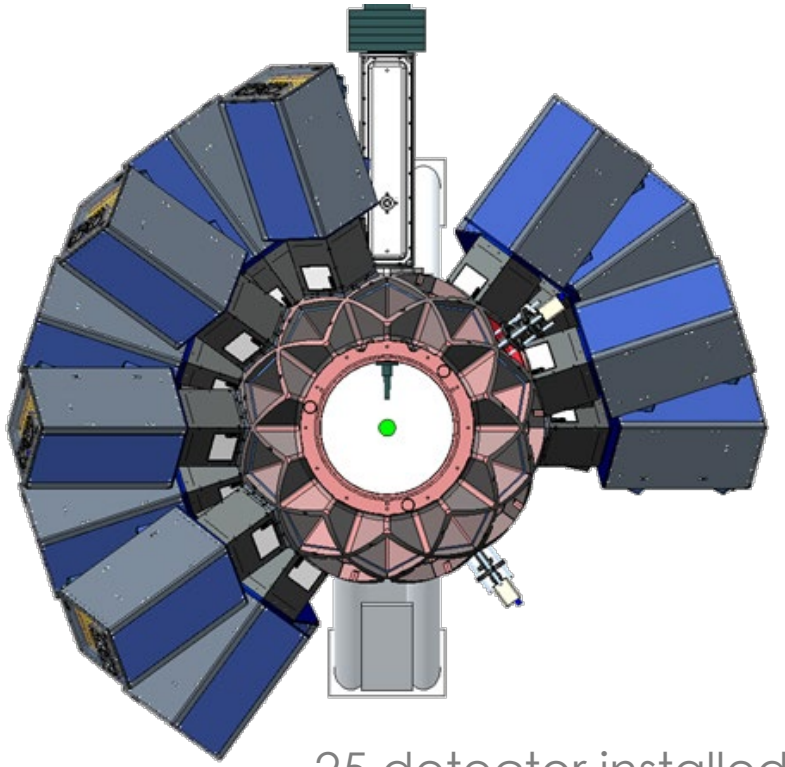


48

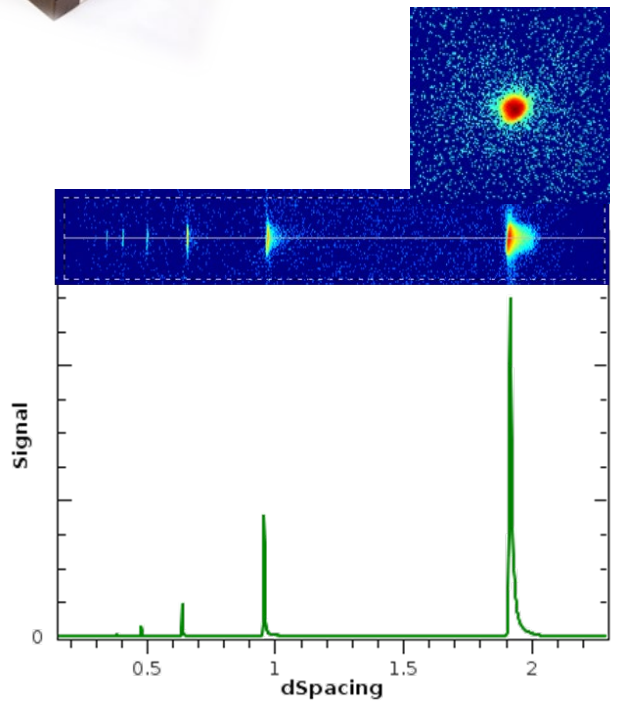
Detector modules to be installed on the Detector Array Tank (DAT)

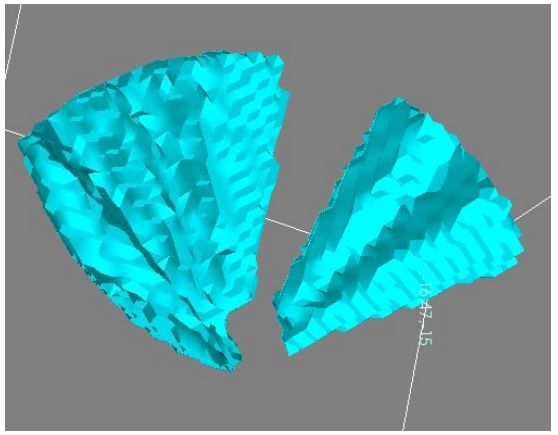
Data are recorded in neutron event mode

- 3*D* Continues *Q* space mapping

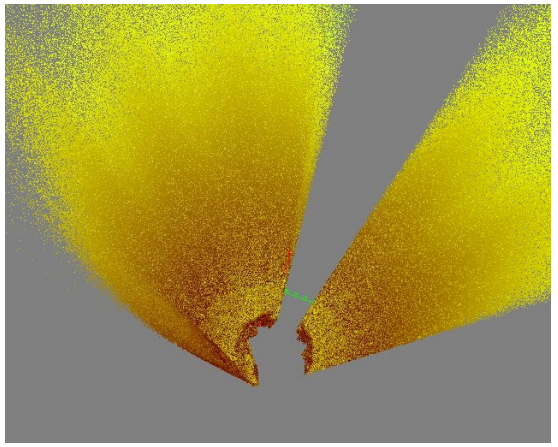


25 detector installed





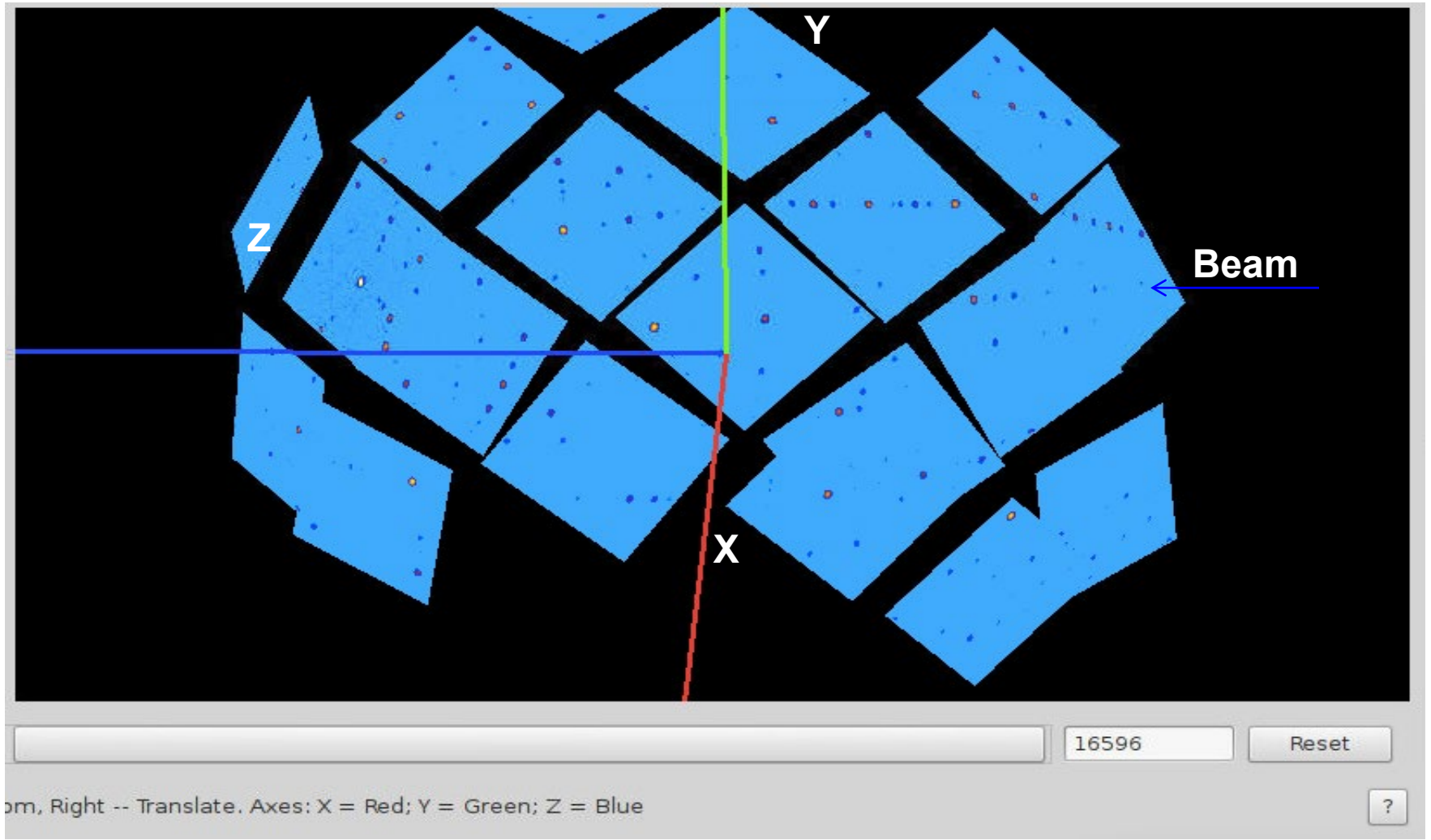
3D Q Coverage, simulated CrystalPlan



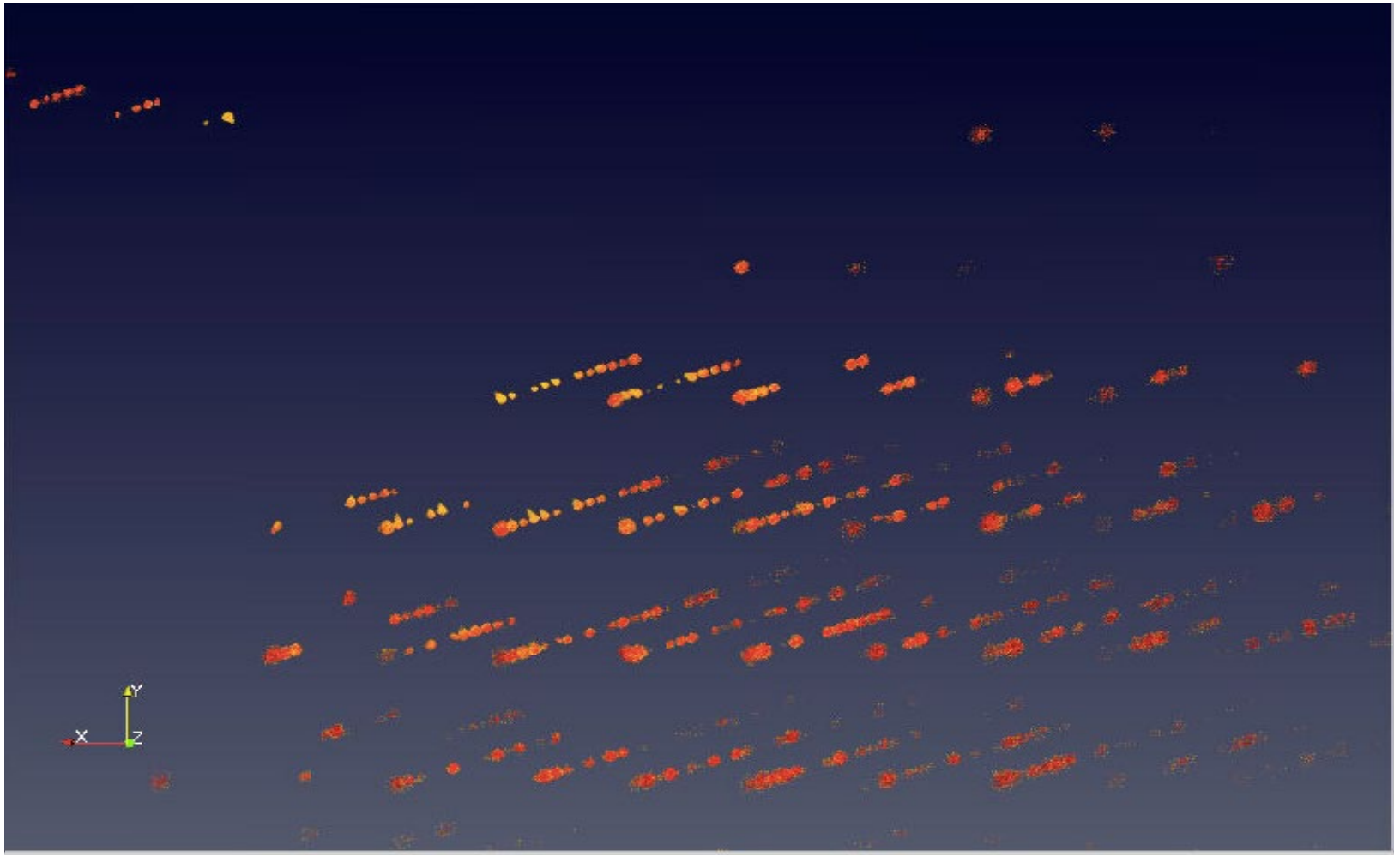
3D Q coverage, measured ISAWEV

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

Single crystal peaks on 2D detector space



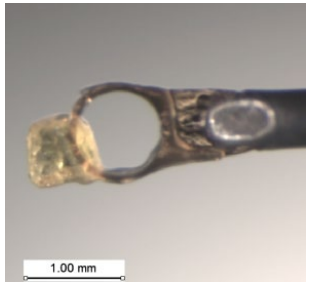
Single crystal peaks in 3D Q space



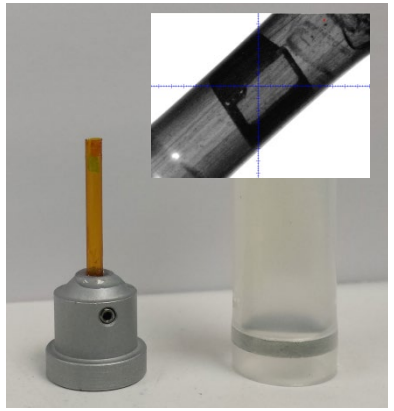
TOPAZ Ambient Goniometer

- 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

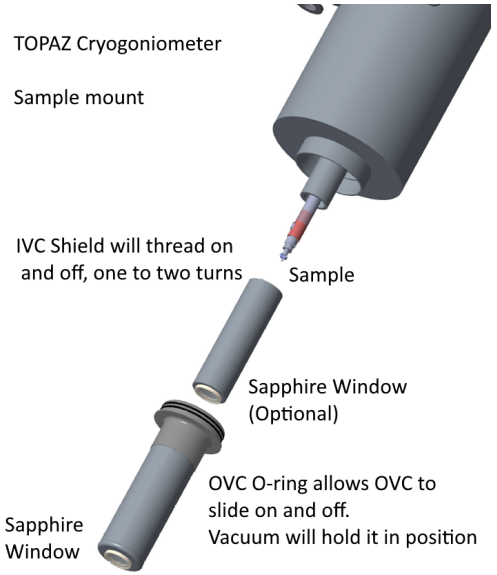


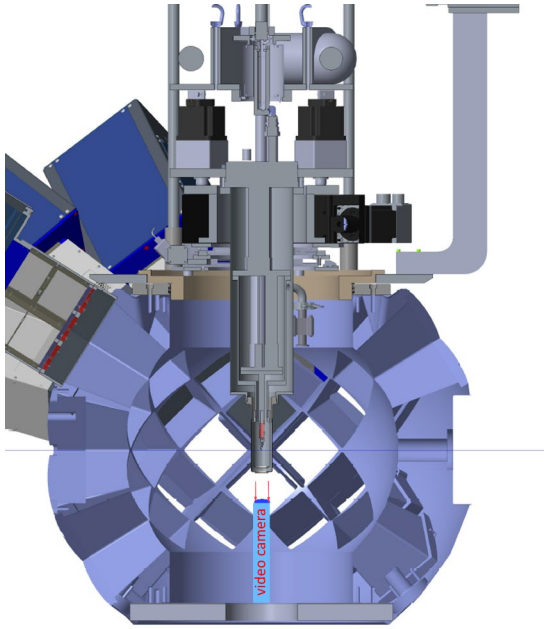
Glued onto the tip or inside a Kapton tube

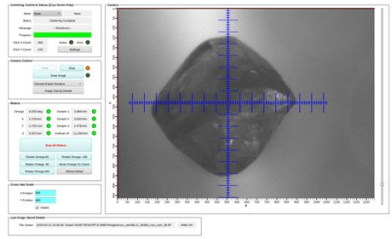
TOPAZ Cryogenic Goniometer

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

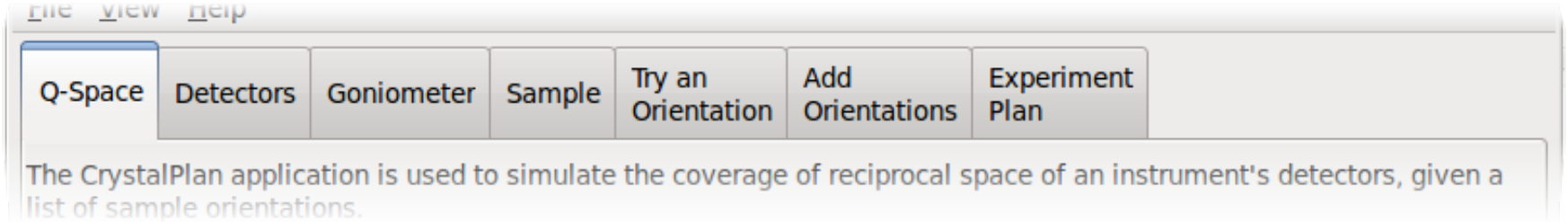






Crystal Plan

- 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

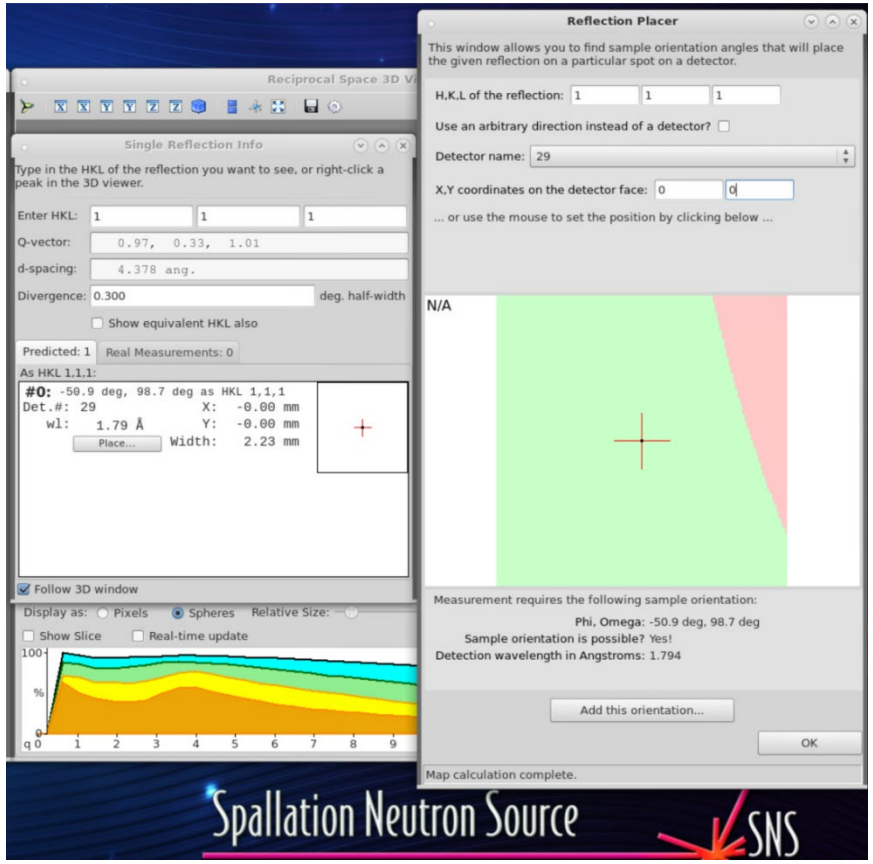


OAK RIDGE National Laboratory | HIGH FLUX ISOTOPE REACTOR | SPALLATION NEUTRON SOURCE

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).

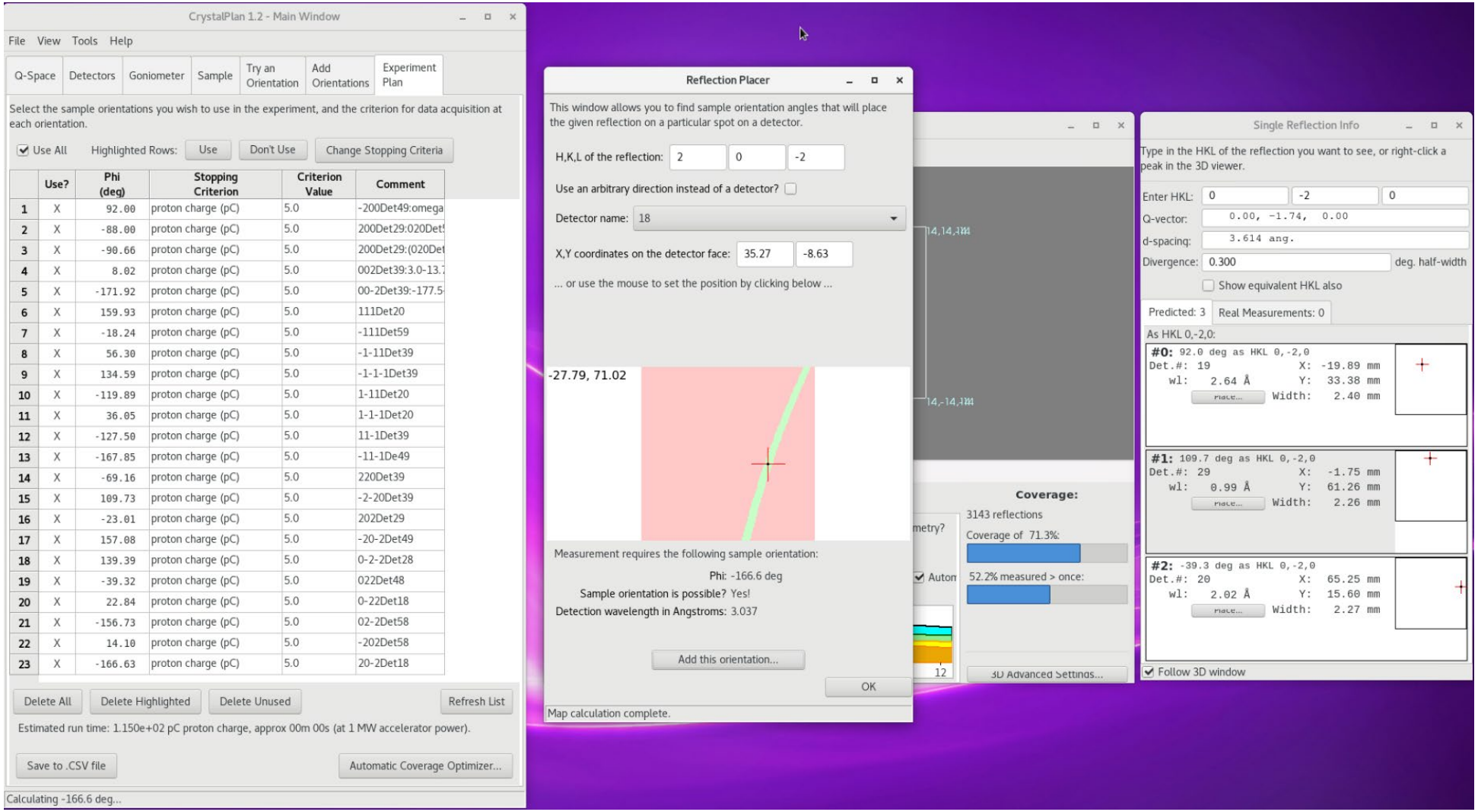
Appl Cryst JAC JOURNAL OF APPLIED CRYSTALLOGRAPHY

CrystalPlan – Peak Prediction and Placement

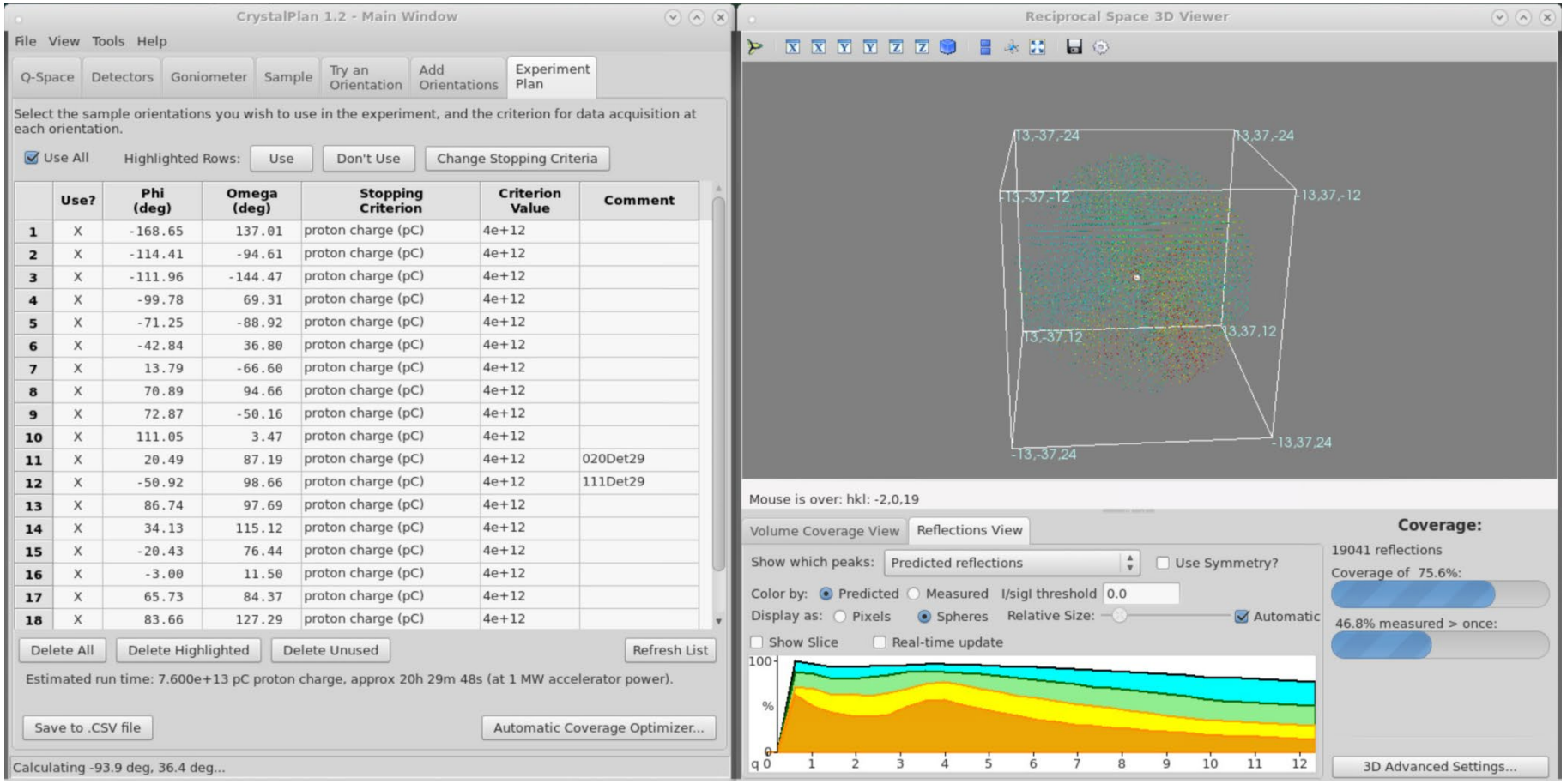


Predict and place a single-crystal peak on selected detector location

Reflection Placer for Cryogoniometer

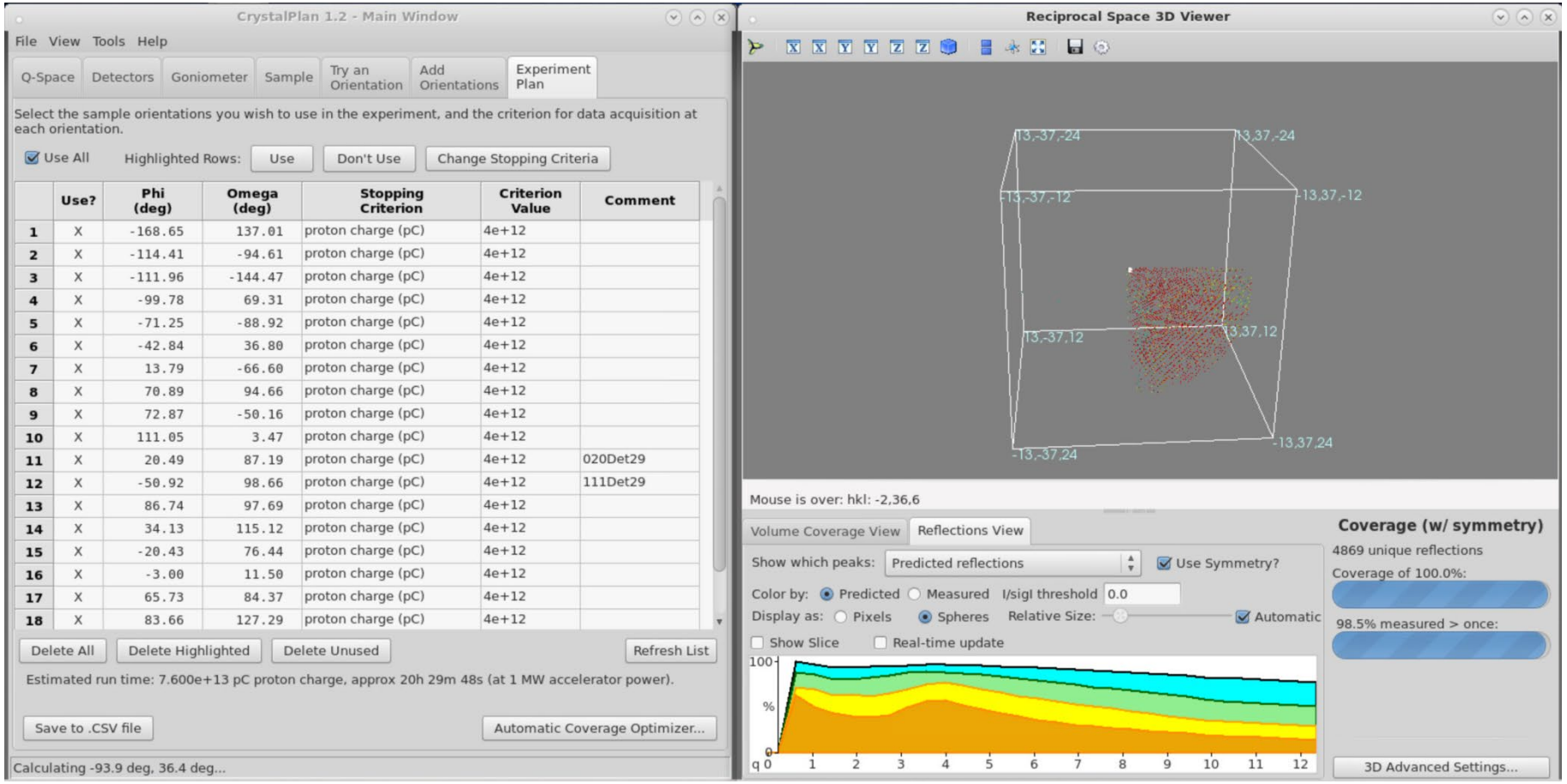


CrystalPlan – Detector coverage



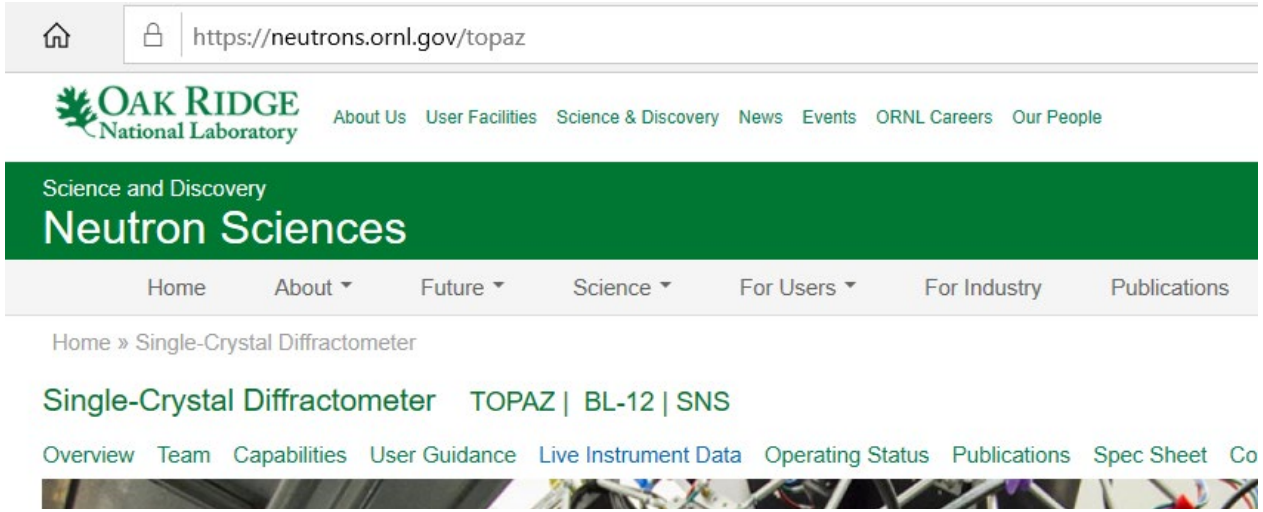
Zikovsky J., Peterson P.F., Wang X.P., Frost M., Hoffmann C., "CrystalPlan: an experiment-planning tool for crystallography", *Journal of Applied Crystallography,* **44,** 418-423 (2011).

CrystalPlan – Detector coverage by symmetry

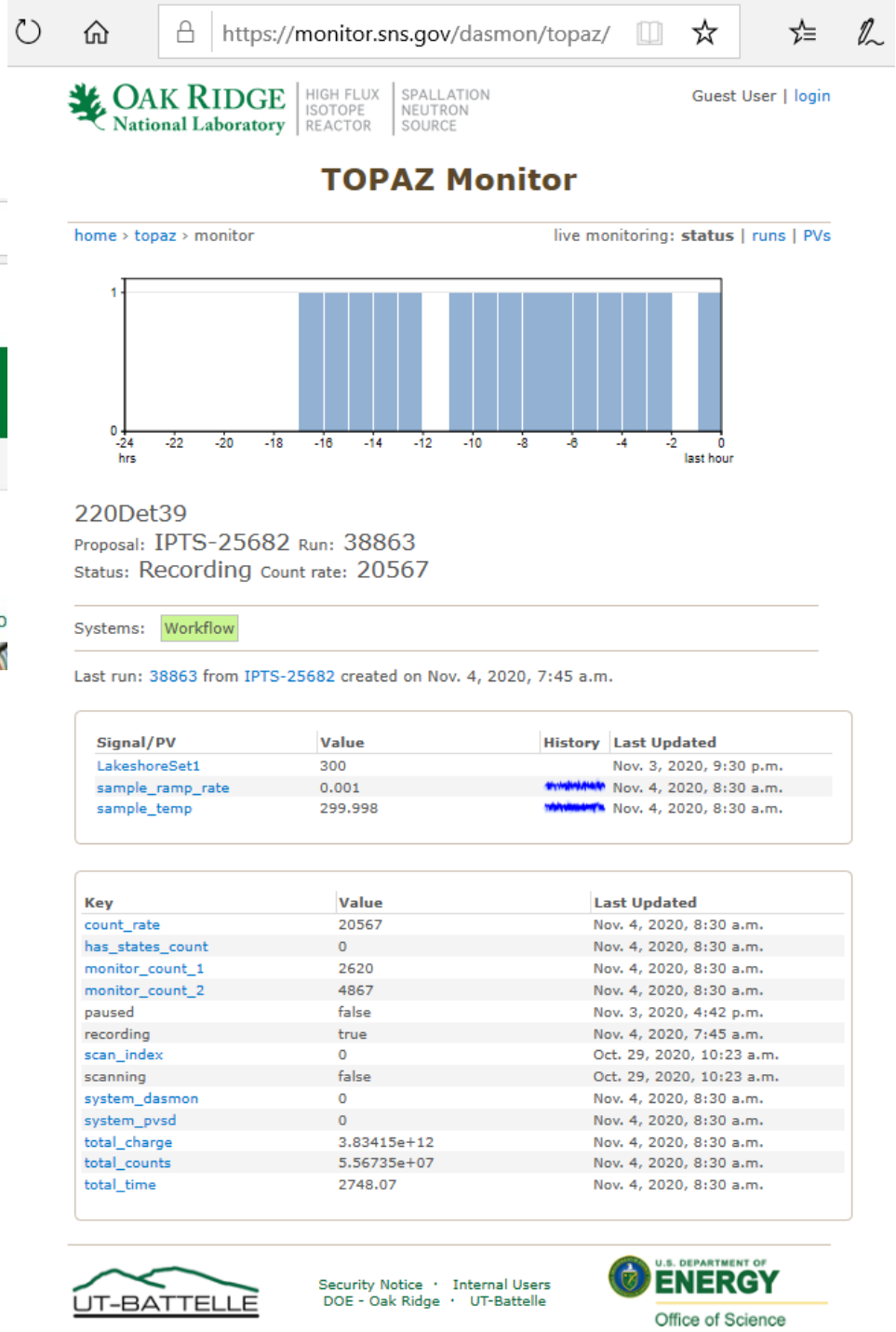


Zikovsky J., Peterson P.F., Wang X.P., Frost M., Hoffmann C., "CrystalPlan: an experiment-planning tool for crystallography", *Journal of Applied Crystallography,* **44,** 418-423 (2011).

TOPAZ Live Instrument Data

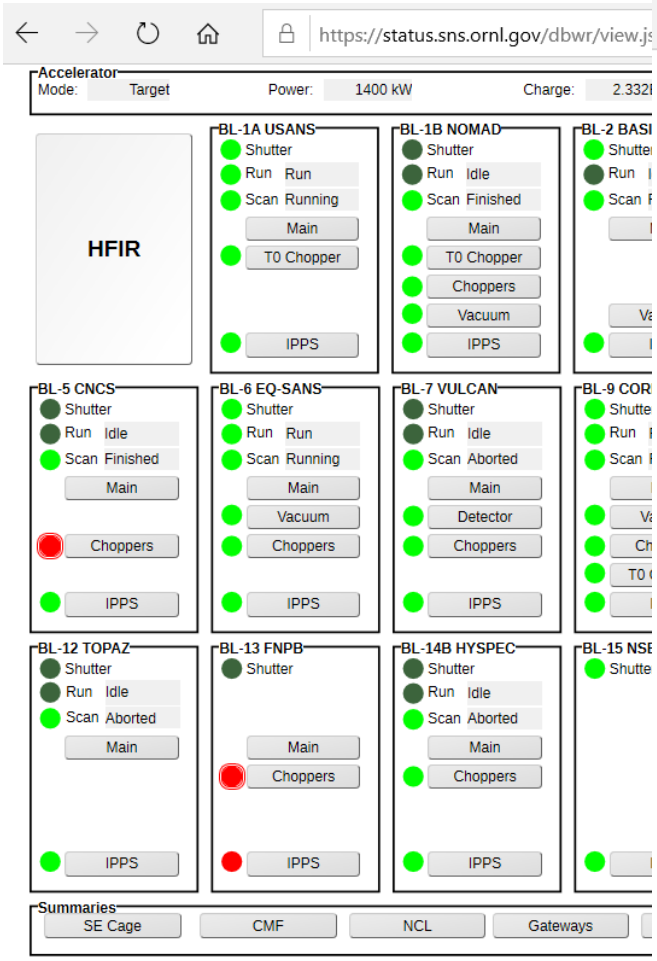


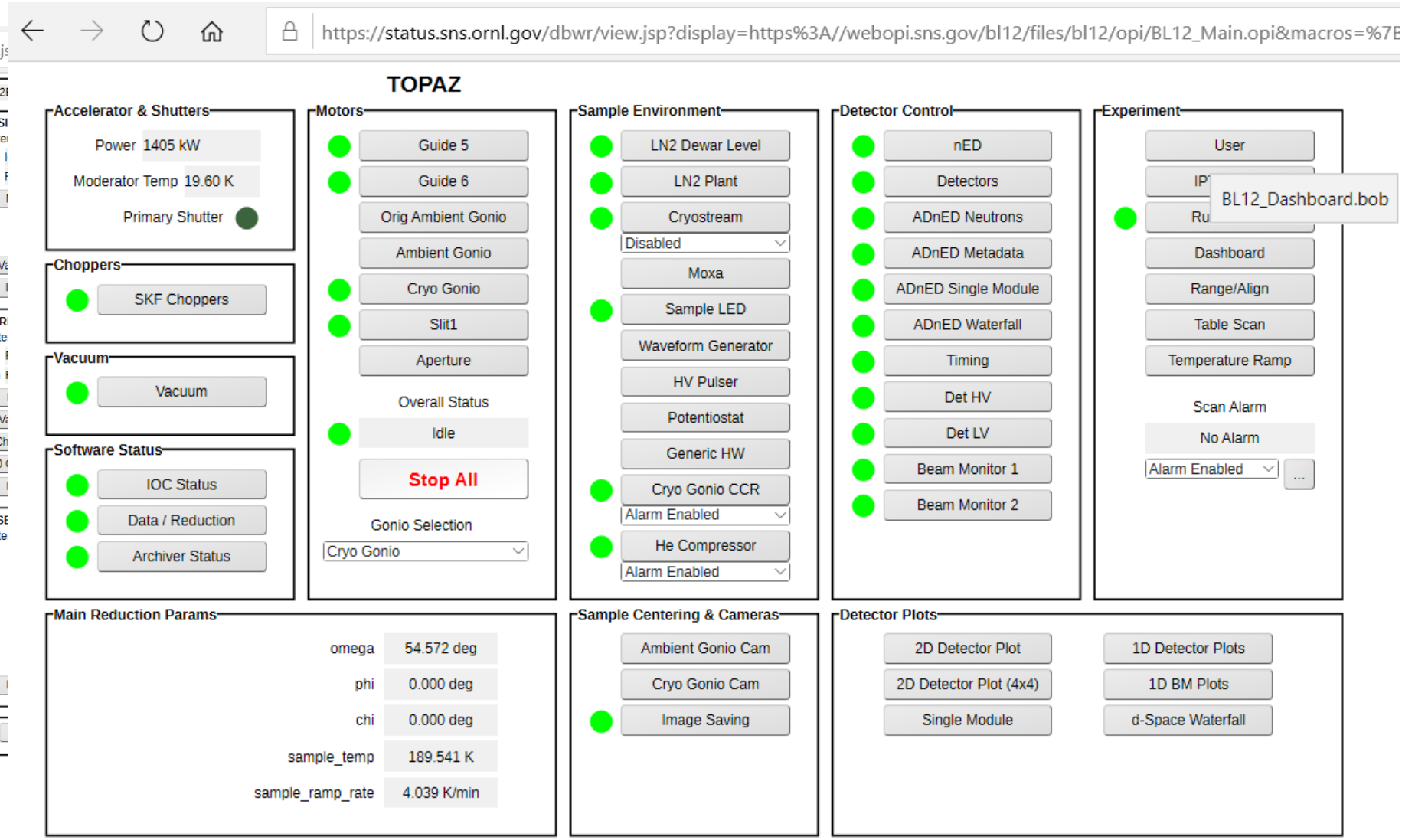
https://neutrons.ornl.gov/topaz



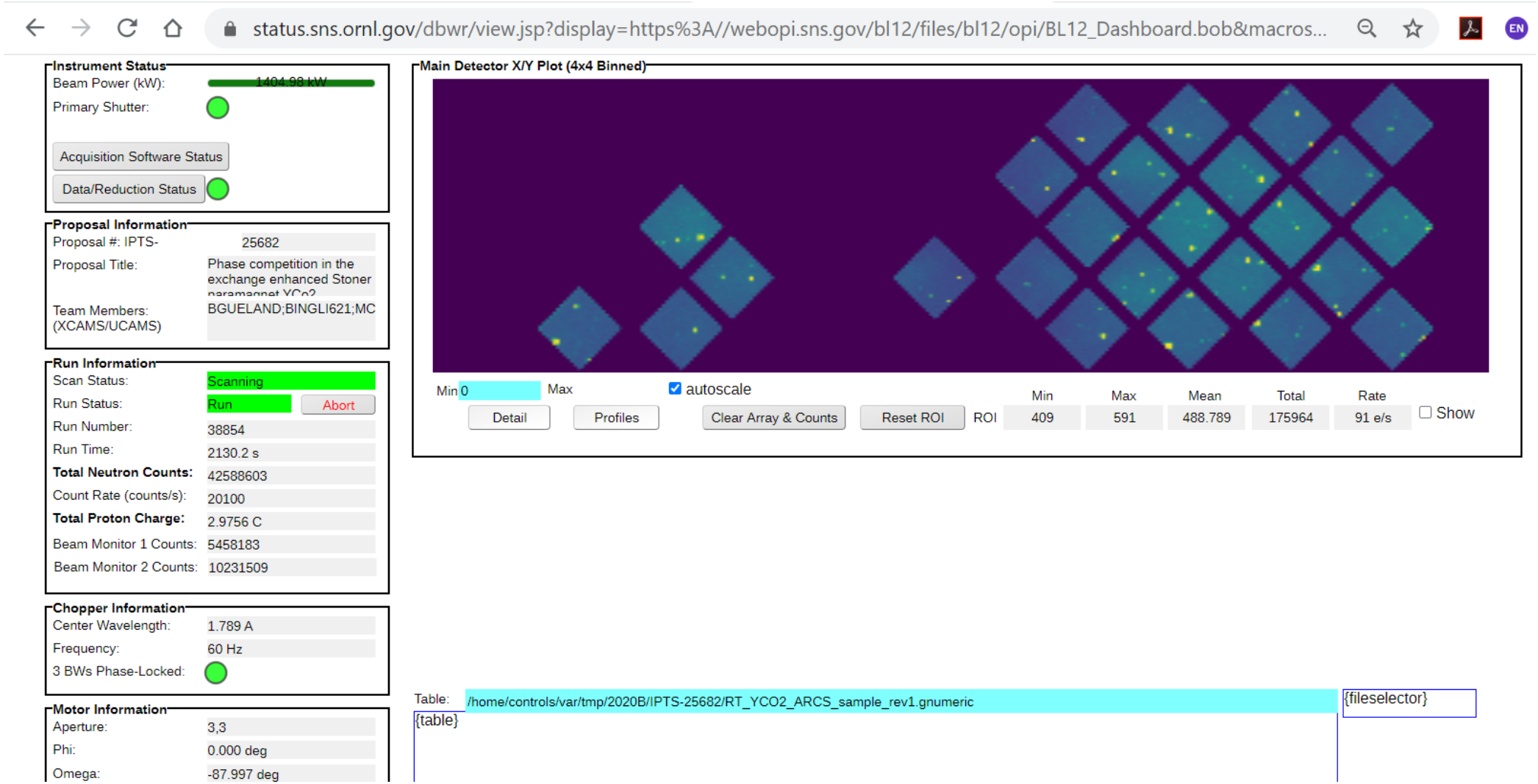
TOPAZ DAS OPI

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





TOPAZ Dashboard

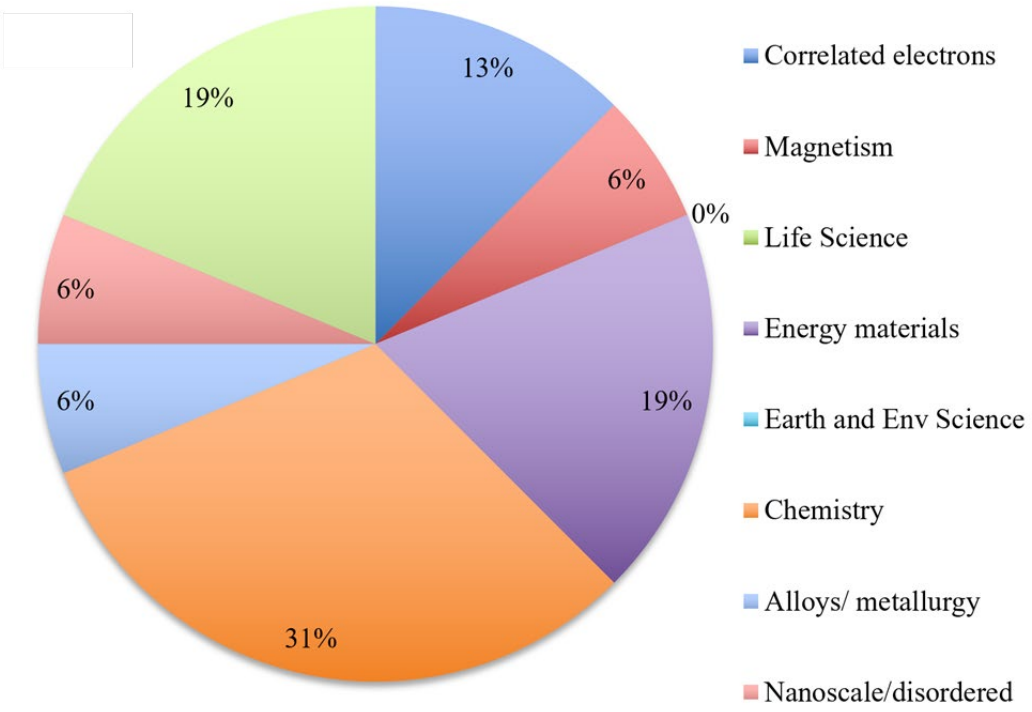


TOPAZ Sciences

- **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

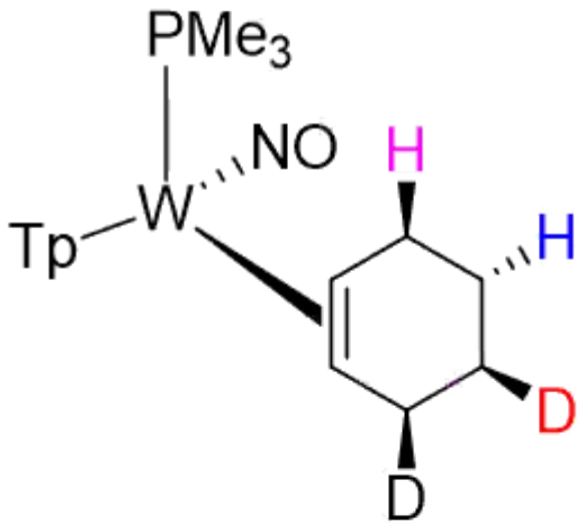
    Order parameters



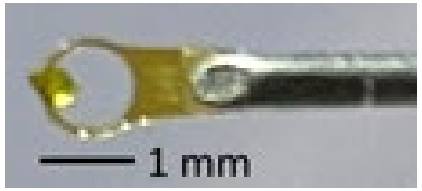
Chemical crystallography

Transition metal-mediated dearomatization.

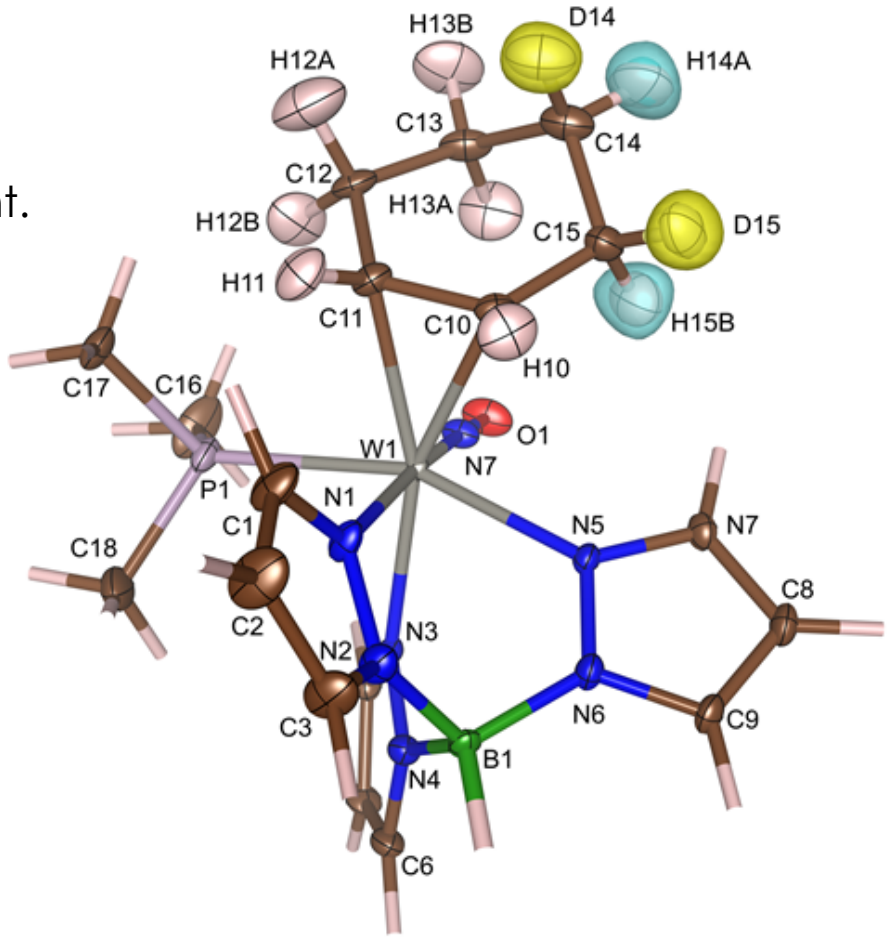
Opened pathways for a new generation of medicines and therapies that incorporate deuterium into the active pharmaceutical ingredient.



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



0.05 mm^3



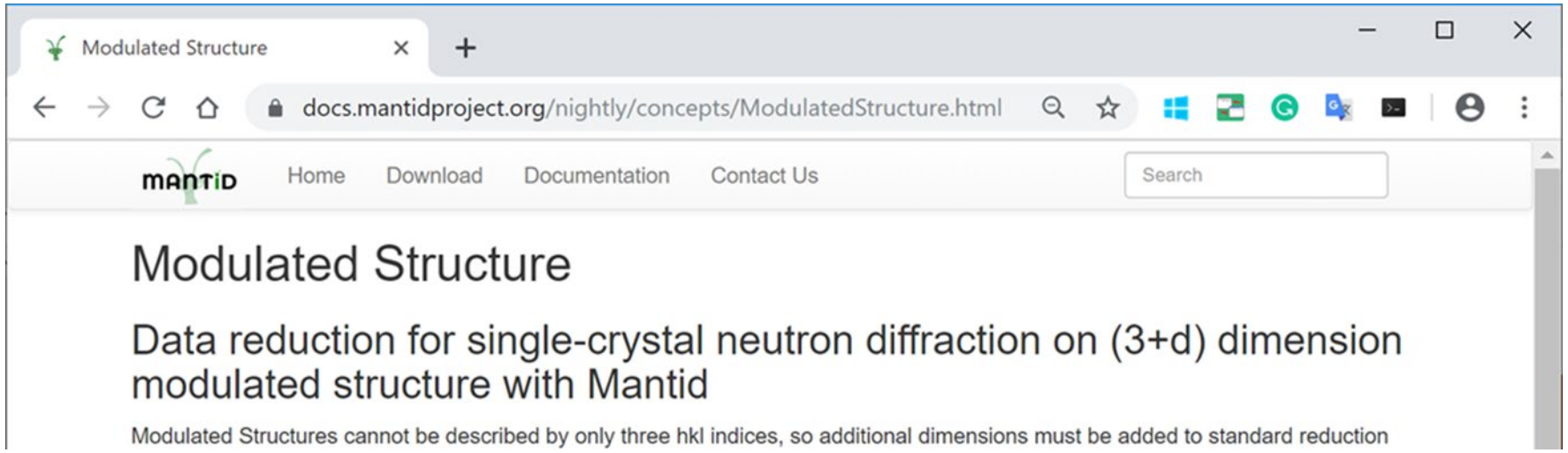
Neutron structure of a d_2 isotopologues of cyclohexene complex.

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

Single crystal neutron diffraction beyond three dimensions

Modulated crystal structure

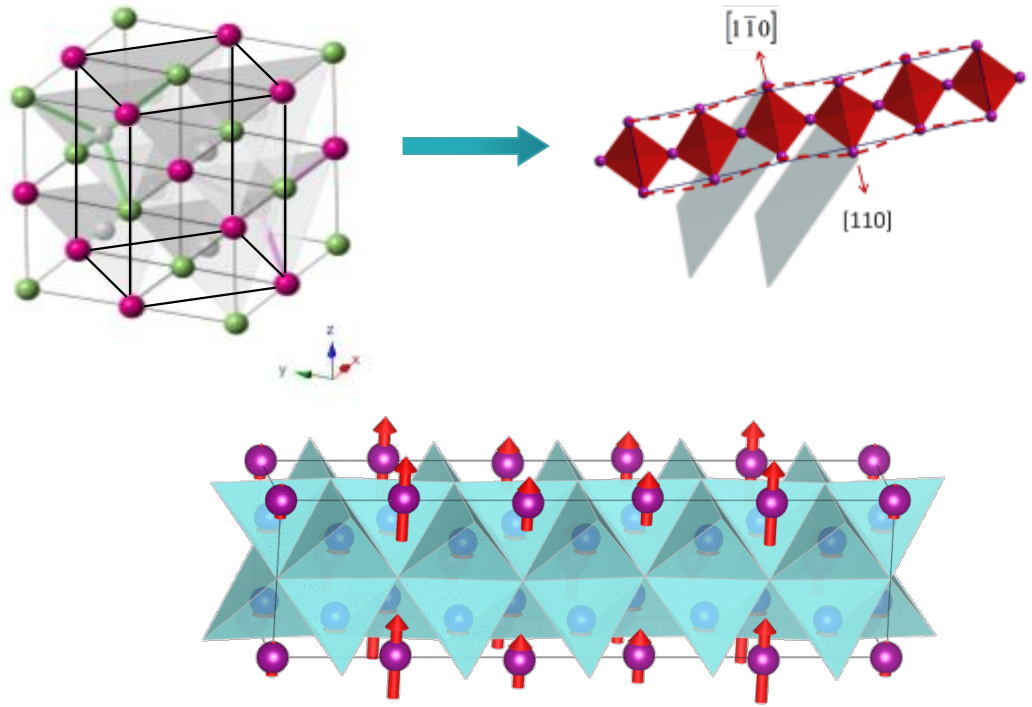
$$\boldsymbol{Q} = 2\pi(h\boldsymbol{a}^* + k\boldsymbol{b}^* + l\boldsymbol{c}^* + m\boldsymbol{q}_1 + n\boldsymbol{q}_2 + p\boldsymbol{q}_3)$$



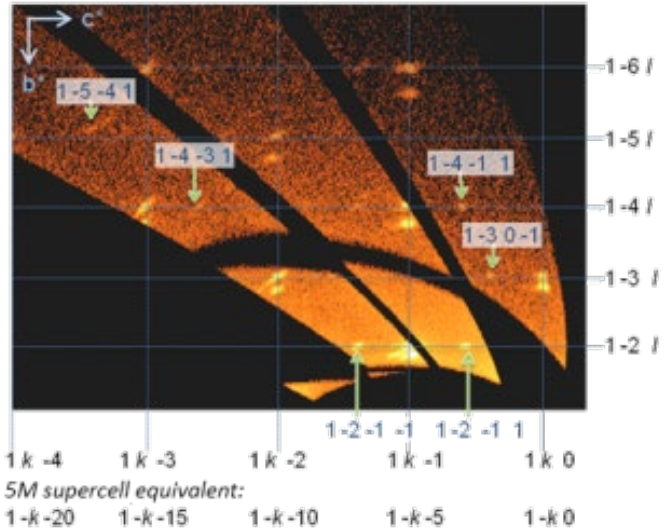
https://docs.mantidproject.org/nightly/concepts/ModulatedStructure.html

Local order and magnetic structural phase transition

- Data measured on TOPAZ are used to describe simultaneous structural and magnetic modulations of $Ni_2Mn_{1.16}Ga_{0.84}$, a Magnetic Shape Memory Alloy.
- Both nuclear and magnetic structures can be refined in JANA2006



JANA2006 / 5M *P2/m(a0γ)00*



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

Single crystal neutron diffraction beyond three dimensions

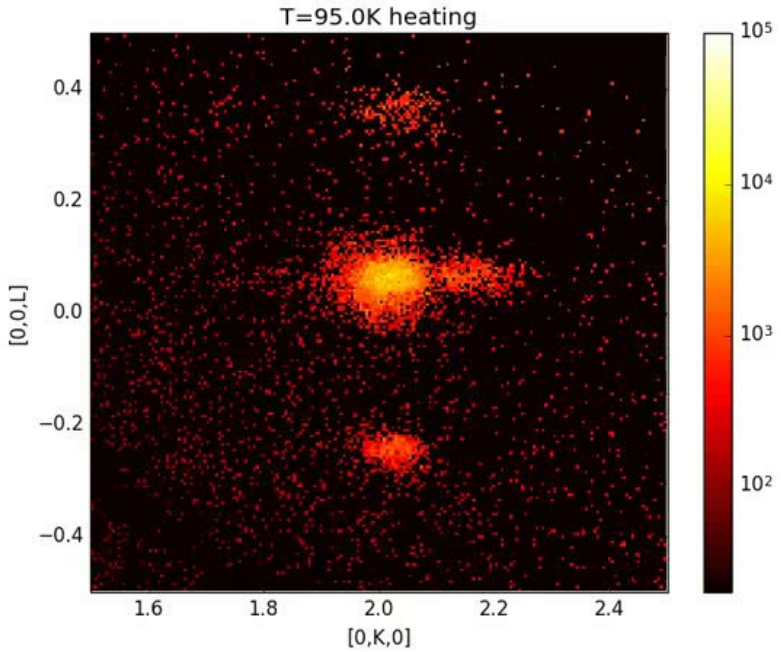
Modulated crystal structure

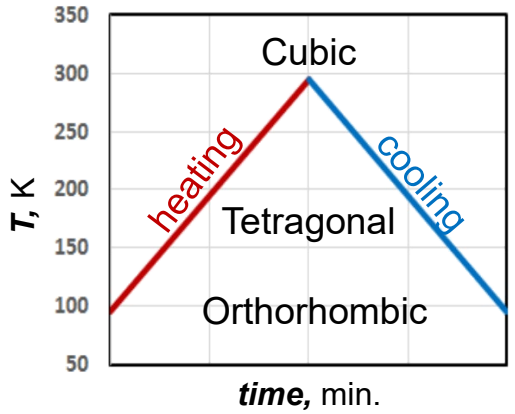
$$\boldsymbol{Q} = 2\pi(h\boldsymbol{a}^* + k\boldsymbol{b}^* + l\boldsymbol{c}^* + m\boldsymbol{q}_1 + n\boldsymbol{q}_2 + p\boldsymbol{q}_3)$$

Multidimensional crystallography in *parameter space*

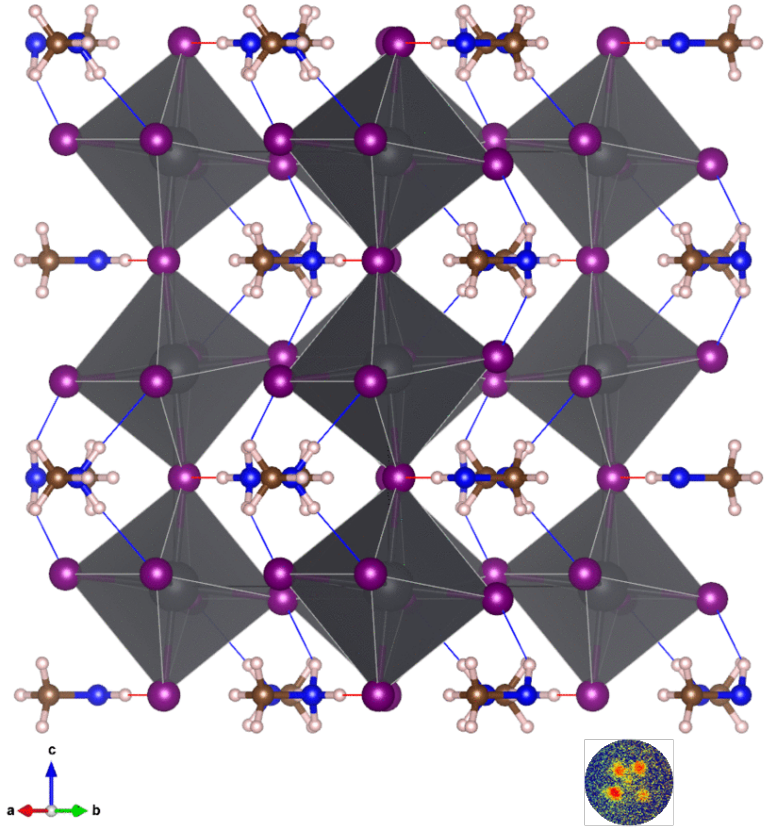
$$\boldsymbol{Q} + T, E, P, B$$

Reconstruction and visualization of real-time data





Ramping rate 1K per min.



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

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

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

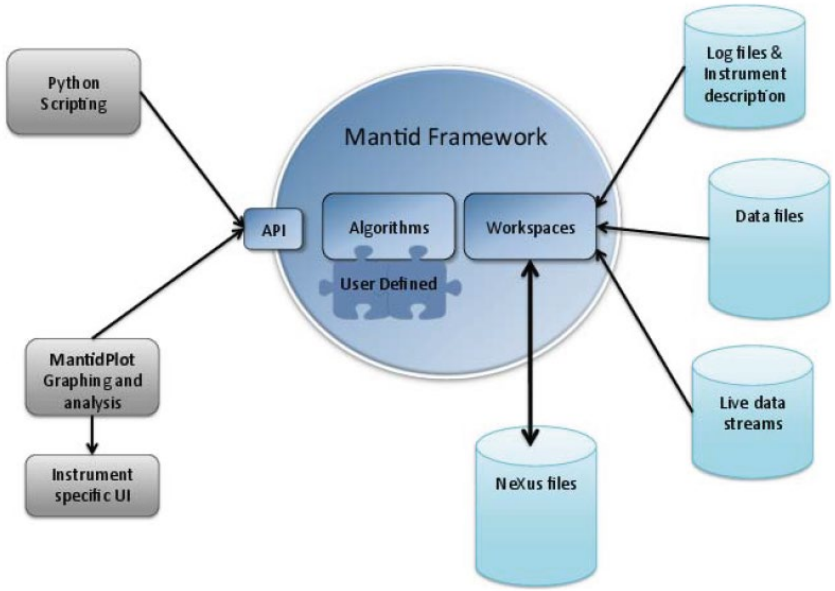
  https://analysis.sns.gov

http://www.nexusformat.org

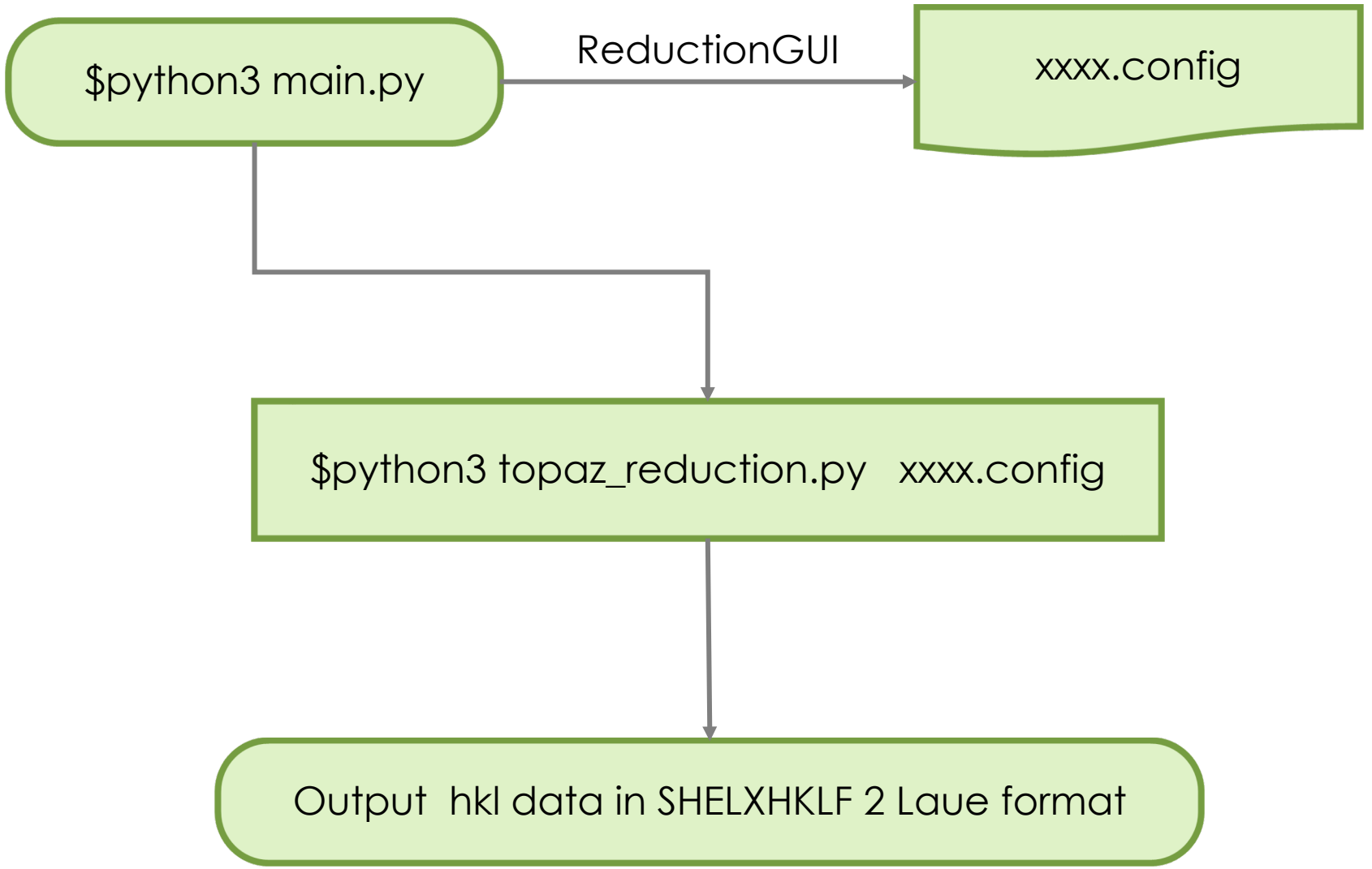
http://docs.mantidproject.org/nightly/concepts/NexusFile.html

Data Reduction

https://analysis.sns.gov



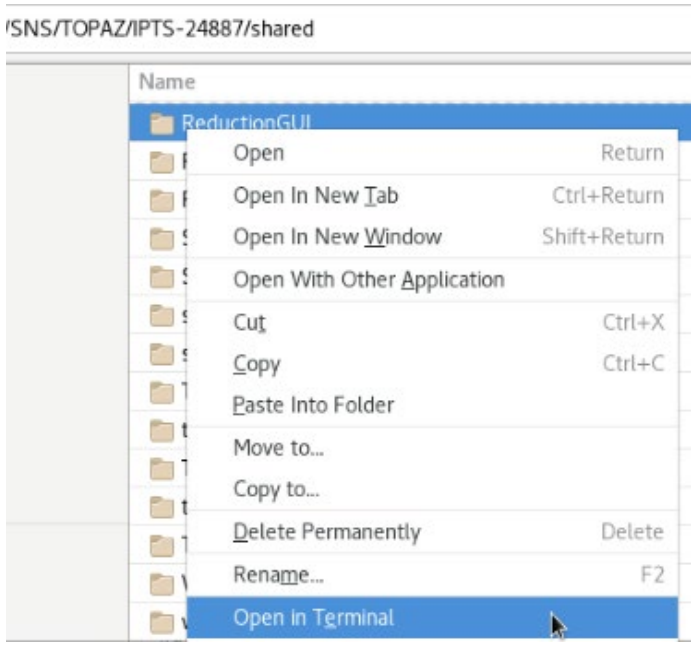
TOPAZ Data Reduction

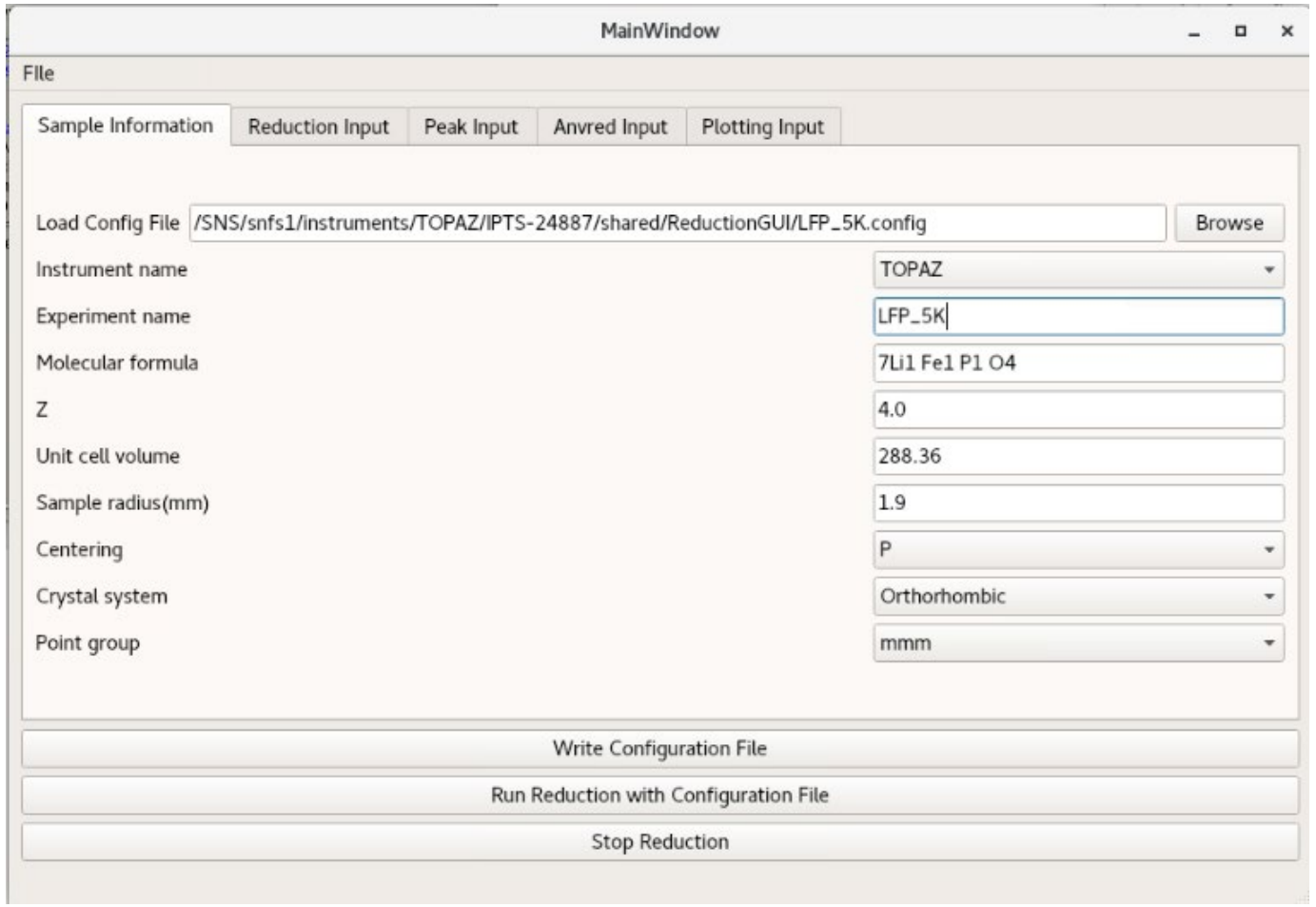


TOPAZ Data Reduction GUI

- 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-xxxxx **./ReductionGUI** subfolder:

$python3 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:

$python3 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:

$python3 topaz_reduction_anvred.py xxxx.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
- o **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)

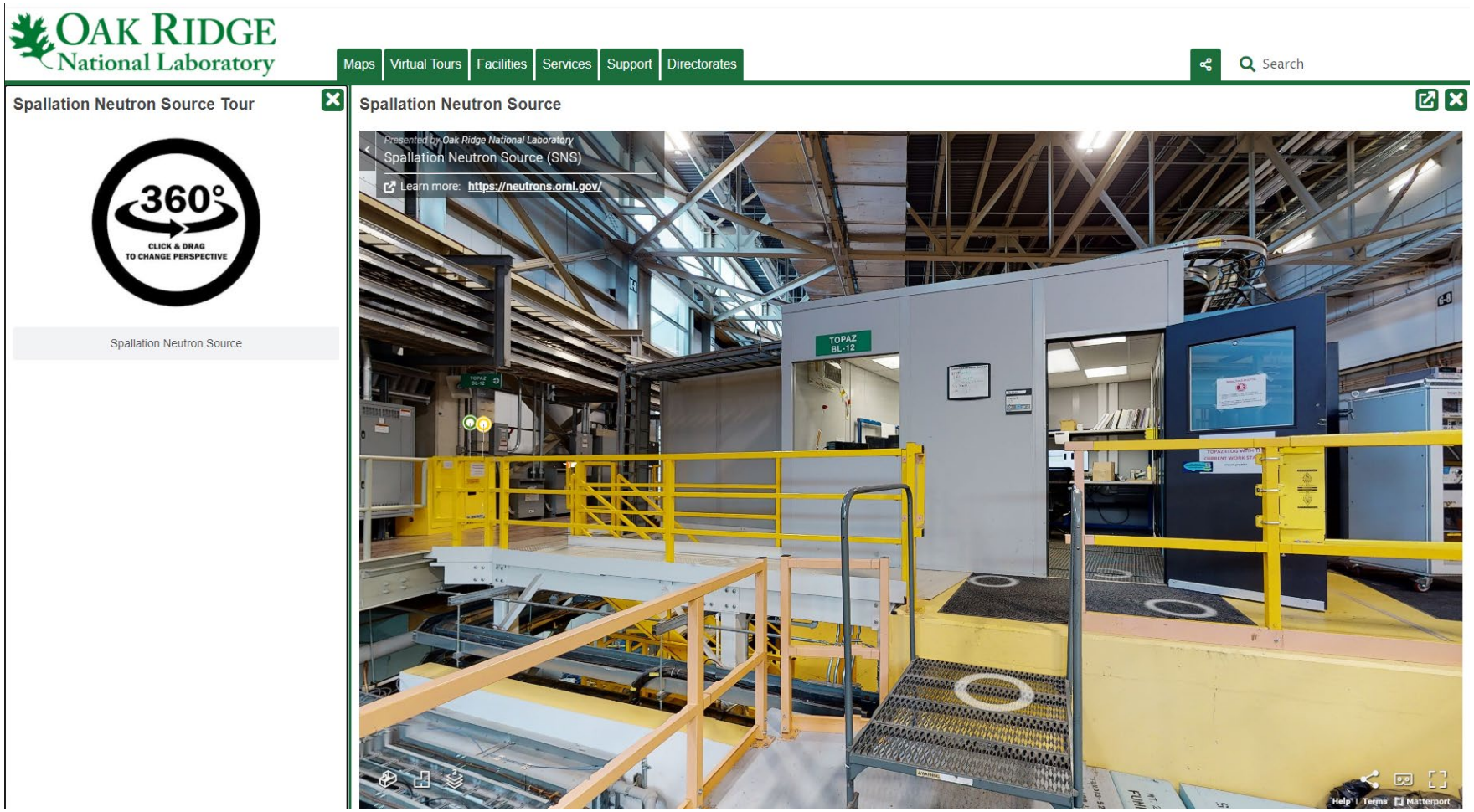
  Robert Von Dreele: Single crystal structure refinement with TOF data in GSAS-II (Argonne, 2016)

- **Tutorials**

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

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