Neutron Single Crystal Diffraction: Principle and Applications in Chemistry and Materials Science

Xiaoping Wang

TOPAZ Instrument Scientist
Chemical and Engineering Materials Division
Neutron Science Directorate
Oak Ridge National Laboratory
Tennessee, USA

Dept. Chemistry & Chemical Biology
Harvard University
April 6, 2017
Oak Ridge National Laboratory
Outline

- Single crystal diffraction
- Spallation Neutron Source at Oak Ridge National Laboratory
- Neutron time-of-flight and wavelength-resolved Laue technique
- TOPAZ TOF Laue single crystal diffractometer
  - Instrument configuration
  - TOPAZ Sample Environment
  - 3D Reciprocal space mapping
  - Data collection strategy
  - Data reduction procedures
  - Data analysis and structure refinement using TOPAZ data
- Applications
### Crystallography milestones

http://www.nature.com/milestones/crystallography

<table>
<thead>
<tr>
<th>Year</th>
<th>Milestone Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre 1912</td>
<td>Periodicity (Milestone 1)</td>
</tr>
<tr>
<td>1912</td>
<td>von Laue experiment (Milestone 2)</td>
</tr>
<tr>
<td>1913</td>
<td>Bragg’s Law (Milestone 3)</td>
</tr>
<tr>
<td>1913 to 1916</td>
<td>X-ray spectrometer and power diffraction (Milestone 4)</td>
</tr>
<tr>
<td>1923 to 1928</td>
<td>First organic molecules (Milestone 5)</td>
</tr>
<tr>
<td>1930</td>
<td>Minerals and silicates (Milestone 6)</td>
</tr>
<tr>
<td>1934</td>
<td>Direct methods (Milestone 7)</td>
</tr>
<tr>
<td>1949</td>
<td>Neutron diffraction (Milestone 8)</td>
</tr>
<tr>
<td>1951</td>
<td>Absolute configuration of tartaric acid (Milestone 9)</td>
</tr>
<tr>
<td>1951 to 1953</td>
<td>Ferrocene (Milestone 10)</td>
</tr>
</tbody>
</table>
Single crystal diffraction

• Nobel Prizes Associated with Crystallography
  – 13 Nobel Prizes in chemistry and physiology or medicine awarded for work in the field of crystallography from 1956 to 2009.
  – Almost one in four chemistry prizes since 1956 have been for structure work. M. Seringhaus and M. Gerstein, *Science*, 2007, 315, 412.

A critical stance is essential to science. Proving other people wrong is a favorite private and public satisfaction—there is nothing some scientists like better. But, excess zeal discounted, doubt serves as a powerful impulse to the advancement of knowledge. We document it here with the discovery of the structure of ferrocene,

Accurate structure determination - molecular structure of ferrocene

Fe(C₅H₅)₂


*The Nobel Prize in Chemistry 1973*

"for their pioneering work, performed independently, on the chemistry of the organometallic, so called sandwich compounds"

*Bis-cyclopentadienyl Iron, a Molecular Sandwich*

Ferrocene: Ironclad History or Rashomon Tale?**

Pierre Laszlo and Roald Hoffmann*


Peter Pauson, private communication, recalls that he had crystals of ferrocene in the fall of 1951. He was trying to decide whether to ask Jack Dunitz, or the senior organic crystallographer of the time, J. Monteath Robertson (real or adopted Scotsmen all). Pauson decided on Robertson. Who, according to Pauson, took the crystals to...Cornell, where he asked Lynn Hoard if someone could look at them. The crystals were assigned to a beginning graduate student, who failed to solve the structure.
History of single crystal diffraction at ORNL

• 1951 Refinement of single crystal structures using neutron data
• 1952 Determination of hydrogen position in potassium bifluoride

THE JOURNAL OF CHEMICAL PHYSICS VOLUME 20, NUMBER 4 APRIL, 1952

A Single Crystal Neutron Diffraction Determination of the Hydrogen Position in Potassium Bifluoride*

S. W. Peterson and Henri A. Levy
Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee
(Received December 10, 1951)

Neutron diffraction measurements on KHF₂ single crystals show that the hydrogen atom occupies the central position, within 0.1Å, in the linear F—H—F ion. The data also indicate asymmetry in thermal motion, which suggests that the bifluoride ion undergoes rotatory oscillation with appreciable amplitude. The study demonstrates the usefulness of single crystal neutron diffraction data for crystal structure determination.
History of single crystal diffraction at ORNL


Angle Calculations for 3- and 4- Circle X-ray and Neutron Diffractometers*

BY WILLIAM R. BUSING AND HENRI A. LEVY

Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830, U.S.A.

(Received 13 June 1966)

Methods are derived for calculations useful in the operation of 3- and 4-circle X-ray or neutron single-crystal diffractometers. These include: (1) establishing the sample orientation from the cell parameters and the observed angles for two reflections, or from the observed angles for three reflections only, (2) calculating the angles for observing a given reflection either in a special setting or at a specified azimuthal angle, (3) obtaining the vectors needed for calculating absorption corrections, and (4) using observations of several reflections to refine cell and orientation parameters by the method of least squares.

1962 ORFLS W.R. Busing et. al.
A Fortran Crystallographic Least-Squares Program

1965 ORTEP C. Johnson
The Oak Ridge Thermal Ellipsoid Plot for drawing crystal structure illustrations

Henri Levy at the keyboard of the four-circle Picker X-ray diffractometer
http://www.amercrystalassn.org/h-busing_memoir
Data acquisition for single crystal diffraction

Monochromatic beam

- Serial detector [*old fashioned*]
  - Record photon/neutron counts using a point counter
  - 1 D Peak integration using peak profile from step scans

- Area detector [*Currently the state-of-the-art for X-rays*]
  - record photon/neutron counts at 2D (x,y) pixel positions
  - 3 D Peak integration possible by combining 2D frame images from step scans
  - Excellent spatial resolution
  - Time resolution is limited > $10^{-3}$ second
Ewald construction
Monochromatic source

\[ 2 \theta_{\text{max}} = \theta_d + \tan^{-1}\left(\frac{a}{2D}\right) \]
\[ 2 \theta_{\text{min}} = \theta_d - \tan^{-1}\left(\frac{a}{2D}\right) \]

Image from Nonius FAST in 1998. X-ray Mo Kα
Laboratory for Molecular Structure and Bonding, TAMU
Established in 1965, the CSD has grown rapidly, reaching 50,000 entries in 1983, 250,000 in 2001, 500,000 as recently as 2009, and 750,000 in 2014, 875,000 in 2016.

ccdc.cam.ac.uk
The underlying concept and theory is the same for X-rays and neutrons:
- **X-ray** → electron density distribution
- **Neutron** → nuclear / magnetic density distribution

**X-ray** Scattering power $\propto$ Atomic No.
Hydrogen is difficult to locate

**Neutron** Scattering powder is Isotope specific
H -3.74 fm, D 6.67 fm
Comparable for N and Pb

Neutron elastic scattering amplitude, known as the scattering length ($fm$), can be positive or negative, depending on whether the neutron-nuclear interaction is attractive or repulsive.

www.ncnr.nist.gov/resources/n-lengths/
Neutron single crystal instruments at ORNL

HIFR

• HB-3A  Four-Circle Diffractometer
• CG-4D Imagine Laue Diffractometer

SNS

• BL-3 SNAP  Spallation Neutrons and Pressure Diffractometer
• BL-9 CORELLI  Elastic Diffuse Scattering Spectrometer
• BL-11B ManDi  Macromolecular Neutron Diffractometer
• BL-12 TOPAZ  Single-Crystal Diffractometer
The Spallation Neutron Source (SNS)

- The SNS is an accelerator-based neutron source facility that provides the most intense pulsed neutron beams in the world.
The Spallation Neutron Source

Accelerator based
Neutron production at the SNS

- The spallation process releases neutrons by bombarding heavy metal atoms (Hg) with energetic protons

\[ p + Hg = 30 \ n + \text{fragments} \]

1GeV
<table>
<thead>
<tr>
<th>Instruments by techniques</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Diffraction</strong></td>
</tr>
<tr>
<td>• Single Crystal:</td>
</tr>
<tr>
<td>• TOPAZ</td>
</tr>
<tr>
<td>• MANDI</td>
</tr>
<tr>
<td>• CORELLI</td>
</tr>
<tr>
<td>• IMAGINE</td>
</tr>
<tr>
<td>• HB3A</td>
</tr>
<tr>
<td>• Powder &amp; Total Scattering:</td>
</tr>
<tr>
<td>• POWGEN</td>
</tr>
<tr>
<td>• NOMAD</td>
</tr>
<tr>
<td>• SNAP</td>
</tr>
<tr>
<td>• HB-2A (Powder)</td>
</tr>
<tr>
<td>• HB-2C (WAND)</td>
</tr>
<tr>
<td>• RAPID (proposed)</td>
</tr>
<tr>
<td>• Engineering:</td>
</tr>
<tr>
<td>• VULCAN</td>
</tr>
<tr>
<td>• HB-2B (NRSF2)</td>
</tr>
<tr>
<td><strong>Spectroscopy</strong></td>
</tr>
<tr>
<td>• Triple Axis</td>
</tr>
<tr>
<td>• CG-4C (CTAX)</td>
</tr>
<tr>
<td>• HB-1 (PTAX)</td>
</tr>
<tr>
<td>• HB-1A (FIE-TAX)</td>
</tr>
<tr>
<td>• HB-3 (TAX)</td>
</tr>
<tr>
<td>• Direct Geometry</td>
</tr>
<tr>
<td>• CNCS</td>
</tr>
<tr>
<td>• SEQUOIA</td>
</tr>
<tr>
<td>• ARCS</td>
</tr>
<tr>
<td>• HYSPEC</td>
</tr>
<tr>
<td>• Indirect Geometry</td>
</tr>
<tr>
<td>• BASIS</td>
</tr>
<tr>
<td>• VISION</td>
</tr>
<tr>
<td><strong>Other</strong></td>
</tr>
<tr>
<td>• Small Angle:</td>
</tr>
<tr>
<td>• EQ-SANS</td>
</tr>
<tr>
<td>• USANS</td>
</tr>
<tr>
<td>• GP-SANS</td>
</tr>
<tr>
<td>• BIO-SANS</td>
</tr>
<tr>
<td>• Reflectometers:</td>
</tr>
<tr>
<td>• Liquids Reflectometer</td>
</tr>
<tr>
<td>• Magnetism Reflectometer</td>
</tr>
<tr>
<td>• Imaging:</td>
</tr>
<tr>
<td>• CG-1D</td>
</tr>
<tr>
<td>• VENUS (proposed)</td>
</tr>
<tr>
<td>• Fundamental Physics</td>
</tr>
</tbody>
</table>

Chemical and Engineering Materials Division
Quantum Condensed Matter Division
Biology and Soft Matter Division
Data acquisition for single crystal diffraction

Monochromatic beam

• Serial detector [*old fashioned*]
  – Record photon/neutron counts using a point counter
  – 1 D Peak integration using peak profile from step scans

• Area detector [*Currently the state-of-the-art for X-rays*]
  – record photon/neutron counts at 2D \((x,y)\) pixel positions
  – 3 D Peak integration possible by combining 2D frame images from step scans
  – Excellent spatial resolution
  – Time resolution is limited > 10^{-3} second

• Polychromatic beam
  – Laue diffraction
  – Use all wavelengths available from a radiation source
  – 3D Diffraction is possible if individual wavelengths is resolved
Neutron Time-of-Flight, TOF

- **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 (aka **time of flight**), its velocity, and consequently its wavelength can be measured.

<table>
<thead>
<tr>
<th>tof</th>
<th>(0.5) msec.</th>
<th>(16.67) msec.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\lambda)</td>
<td>(0.2) Å</td>
<td>(3.66) Å</td>
</tr>
</tbody>
</table>
Neutron Time-of-Flight Laue

- Combine de Broglie’s equation with Bragg’s law

\[ \lambda = \frac{h}{mv} = \frac{ht}{m(L_1 + l_2)} \]

\[ \lambda = 2d \sin \theta \]

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

Neutron Time-of-flight Laue
(Wavelength-resolved Laue)

3-D Reciprocal Space Mapping

\[ L_1 \text{ – Source to sample distance} \]
\[ l_2 \text{ – Sample to detector distance} \]

Pulsed Neutron 60 Hz
The SNS TOPAZ instrument
The SNS TOPAZ instrument

Neutron wavelength-resolved Laue
3D Reciprocal space mapping

\[ d_{\text{min}} = 0.25 \text{ Å} \]
\[ Q_{\text{max}} \approx 25 \text{ Å}^{-1} \]
Sample mount

Single Crystal Samples

Coated with perfluorinated grease and attached onto a MiTeGen loop for low temperature exp.

Glued onto MiTeGen loop

Inside a Kapton tube

Glued onto the tip of a Kapton tube

Sub-Millimeter Sized Crystals
Diameter: 0.10 – 5.0 mm, Volume: > 0.1 mm³

Multiple Area Detectors
Solid Angle Coverage: 2.4 ster.
Detector 2θ Coverage: 13.5° - 160°

Controlled Sample Environment
CryoStream 700 Plus: 90K – 450K
Laue pattern in detector space
Ewald Sphere
Time-of-Flight Laue technique

Reflections at one scattering angle (90°) resolved at different TOF's

Arthur J. Schultz, TOPAZ Workshop, 2015
Laue pattern in detector space
Laue pattern in detector space
Laue pattern in detector space – Cylindrical view
Single crystal peaks in Q space
Q space views of single crystal peaks
Experiment planning

CrystalPlan

• 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

CrystalPlan – Detector coverage

## Instrument parameters

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Moderator</strong></td>
<td>decoupled poisoned H₂</td>
</tr>
<tr>
<td><strong>Source to sample L₁</strong></td>
<td>18 m</td>
</tr>
<tr>
<td><strong>Sample to detector l₂</strong></td>
<td>0.395-0.460 m</td>
</tr>
<tr>
<td><strong>Detector angular coverage</strong></td>
<td>13.5°&lt; 2Q &lt;160°</td>
</tr>
<tr>
<td><strong>Detector solid angle coverage</strong></td>
<td>2.4 ster.</td>
</tr>
<tr>
<td><strong>Bandwidth</strong></td>
<td>~3.6 Å</td>
</tr>
<tr>
<td><strong>Frames 1 &amp; 2 (λ range at 60Hz)</strong></td>
<td>0.40 – 3.50 Å; 3.75 – 7.0 Å</td>
</tr>
<tr>
<td><strong>Sample size: Diameter, Volume</strong></td>
<td>0.1 – 4.0 mm &amp; &gt; 0.10 mm³</td>
</tr>
<tr>
<td><strong>Unit cell length</strong></td>
<td>&lt;70 Å,</td>
</tr>
<tr>
<td><strong>Resolution, d_{min}, Q_{max}</strong></td>
<td>d_{min}&gt;= 0.30 Å, 20.9 Å⁻¹</td>
</tr>
</tbody>
</table>
TOPAZ data collection

Sample Evaluation
- **ISAW EventViewer** → Live data
- **Mantidpot SCD Interface** → Saved short run
- Index peaks and find UB for data collection strategy

Data Collection Strategy
- **CrystalPlan**
  - Optimize sample orientations by crystal symmetry
  - Balance coverage, redundancy and collection time
  - Save sample orientations to .csv

Data Collection
- **SNS PyDas and DcomClient**
  - Load crystal settings from CrystalPlan
  - Setup data collection parameters:
    - $\lambda$, $T$, stopping criteria …
  - Run as table scan in PyDas
ISAW - Event data viewer

Live data stream is plotted into 3D $Q$-space
Peaks are searched and indexed
Cell parameters and UB-matrix are refined
ISAW - Event Data Viewer

Live data stream is plotted into 3D Q-space
Peaks are searched and indexed
Cell parameters and UB-matrix are refined
Data Collection Strategy

CrystalPlan
Optimize sample orientations by crystal symmetry
Balance coverage and redundancy
Save sample orientations to .csv
Data storage

• 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

http://www.nexusformat.org
http://docs.mantidproject.org/nightly/concepts/NexusFile.html
Data reduction
TOPAZ peak integration

Get peak profile
- **Stats_vs_radius.py** ➔ Find peak integration radius
- Index peaks and save UB and integrate file
- The integration radius is in Q Å⁻¹ (Q = 2π/d) unit

Setup integration parameters
- **ReduceSCD.config** ➔ Select proper peak integration parameters
- Sphere, ellipse, 2D, 1D...

Perform peak integration
- **ReduceSCD_Parallel.py**
- Generates individual .integrate and .mat files
- Combine Niggli .integrate and .mat files into one integrate and mat file for all runs in a data set
- Performs symmetry transform if cell_type is specified
Peak integrations in $Q$ space

- Bragg peak integration using three-dimensional ellipsoids in Mantid.

TOPAZ peak integration

Stats_vs_radius.py ➔ Find optimal peak integration radius
Python scripts for integration of multiple runs

- ReduceSCD_xxx.config
- ReduceSCD_Parallel.py
- ReduceSCD_OneRun.py

<table>
<thead>
<tr>
<th>DETNUM</th>
<th>NRUNS</th>
<th>NCOLS</th>
<th>WIDTH</th>
<th>HEIGHT</th>
<th>DEPTH</th>
<th>DETD</th>
<th>CenterX</th>
<th>CenterY</th>
<th>CenterZ</th>
<th>BaseX</th>
<th>BaseY</th>
<th>BaseZ</th>
<th>UpX</th>
<th>UpY</th>
<th>UpZ</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>16</td>
<td>256</td>
<td>256</td>
<td>15.8190</td>
<td>15.9600</td>
<td>0.2000</td>
<td>-22.9051</td>
<td>-24.6398</td>
<td>-31.7036</td>
<td>0.35194</td>
<td>0.60487</td>
<td>-0.71434</td>
<td>-0.80273</td>
<td>0.58755</td>
<td>0.10202</td>
</tr>
</tbody>
</table>

.. | NRUN | DETNUM | CHI | PHI | OMEGA | MONCNT |
.. | 10074 | 16    | 135.00 | -3.44  | -110.24 | 2222759 |
Data normalization

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

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

$k$ = scale factor based on total incident beam monitor counts

$\phi(\lambda)$ = incident flux spectrum, obtained by measuring the incoherent scattering from a vanadium sample

$\varepsilon(\lambda, \vec{r})$ = detector efficiency calculated as a function of wavelength $\lambda$ and position $\vec{r}$ on the detector for each Bragg peak since the slant path through the flat $^6$Li glass varies with $\vec{r}$, or it is measured.

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

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

$V_s$ = sample volume

$V_c$ = unit cell volume

Correction to Bragg peaks

- For each hkl, the raw integrated intensity, wavelength and two-theta angle are read from the integrate file.

- Intensity is normalized to 1 Å using the absorption-correction vanadium spectrum $\phi(\lambda)\varepsilon(\lambda, r)$.

- Lorentz factor: correct for $\frac{\lambda^4}{\sin^2 \theta}$

- Calculate wavelength dependent $\mu$ for sample crystal. Apply spherical or gaussian absorption correction.

- Scale the based on total protons-on-target or total incident beam monitor counts.

- The extinction correction $y(\lambda)$ is evaluated during the least-squares refinement of the structure.

- $V_s$ and $V_c$ are constants for the sample, and are not applied. Scale factors to convert the relative $|F_{hkl}|^2$ to absolute $|F_{hkl}|^2$ are applied in the least-squares refinement.
Absorption correction

- Absorption derived from tables of $\mu R$ vs. $\theta$, where $\mu$ is the wavelength dependent linear absorption coefficient for the sample, $R$ is the sphere or rod radius, and $\theta$ is half of the scattering angle.

  \[ \mu = 0.367 + (0.366/1.8) \lambda \] for vanadium.

- For the rod, which is assumed to be vertical, an additional path length and absorption is evaluated for detectors not in the horizontal plane.

- Numerical absorption correction
  - Face indexed single crystal sample
    
    **Python script**  anvred3.py
    
    **Input file**  anvred3.inp

Absorption Correction

Output from ISAW  lin_abs_coef.py:

Chemical formula: Ca1 Al2 Si3 O13 H6
Number of formula units in the unit cell (Z): 4.000
Unit cell volume (Å^3): 1137.85

Cross sections in units of barns (1 barn = 1E-24 cm^2)
Absorption cross section for 2200 m/s neutrons (wavelength = 1.8 Å)
For further information and references, see
...\ISAW\Databases\NIST_cross-sections.dat

....
The linear absorption coefficient for total scattering is 0.671 cm^-1
The linear absorption coefficient for true absorption at 1.8 Å is 0.734 cm^-1

The calculated density is 2.290 grams/cm^3

For a weight of 2.710 mg, the crystal volume is 1.183 mm^3
The crystal radius is 0.656 mm, or 0.0656 cm
### Face indexing

<table>
<thead>
<tr>
<th>TITL Cu32H20 Cluster</th>
<th>CELL</th>
<th>16.2664</th>
<th>17.6384</th>
<th>17.8248</th>
<th>108.126</th>
<th>103.296</th>
<th>111.151</th>
</tr>
</thead>
<tbody>
<tr>
<td>FACE</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>0.1250</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>0</td>
<td>0</td>
<td>-1</td>
<td>0.1250</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>0.8100</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>0</td>
<td>-1</td>
<td>0</td>
<td>0.8100</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>0.5500</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>-1</td>
<td>0</td>
<td>0</td>
<td>0.5500</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>1</td>
<td>-1</td>
<td>0</td>
<td>1.2000</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>-1</td>
<td>1</td>
<td>0</td>
<td>1.2500</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>2</td>
<td>1</td>
<td>0</td>
<td>0.5700</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FACE</td>
<td>-1</td>
<td>-1</td>
<td>0</td>
<td>0.7500</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MUAB</td>
<td>0.1194</td>
<td>0.1712</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Example
********** nod = 19
Reading spectrum for Bank 1 DetNum 16
.....
Reading spectrum for Bank 17 DetNum 48
Reading spectrum for Bank 18 DetNum 49
Reading spectrum for Bank 19 DetNum 58

Minimum and maximum transmission = 0.8070, 0.9301
_diffrn_reflns_theta_min 7.525
_diffrn_reflns_theta_max 78.045
min d-spacing : 0.5000
max d-spacing : 9.4767

Save hkl files by runs and by detector number

scolecite295K_Monoclinic_C.hkl
scolecite295K_Monoclinic_C.hkl_dn
**TOPAZ reduced data format**

- SHELX HKLF 2 with direction cosines

<p>| | | | | | | | | | | | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>1580.63</td>
<td>35.70</td>
<td>1.267106</td>
<td>0.09649</td>
<td>-0.01594</td>
<td>-0.71045</td>
<td>-0.18348</td>
<td>-0.24735</td>
<td>0.92343</td>
<td>0.38974</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-3</td>
<td>-3</td>
<td>6</td>
<td>2556.38</td>
<td>55.46</td>
<td>1.251639</td>
<td>0.09632</td>
<td>-0.01594</td>
<td>-0.66630</td>
<td>-0.18348</td>
<td>-0.48860</td>
<td>0.92343</td>
<td>0.31393</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-4</td>
<td>-4</td>
<td>8</td>
<td>170.26</td>
<td>10.48</td>
<td>1.200330</td>
<td>0.09699</td>
<td>-0.01594</td>
<td>-0.71045</td>
<td>-0.18348</td>
<td>-0.24735</td>
<td>0.92343</td>
<td>0.38974</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-4</td>
<td>-4</td>
<td>8</td>
<td>186.80</td>
<td>11.71</td>
<td>1.922225</td>
<td>0.09684</td>
<td>-0.01594</td>
<td>-0.67953</td>
<td>-0.18348</td>
<td>-0.45277</td>
<td>0.92343</td>
<td>0.33710</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-5</td>
<td>-5</td>
<td>9</td>
<td>2753.24</td>
<td>65.94</td>
<td>1.658760</td>
<td>0.09685</td>
<td>-0.01594</td>
<td>-0.79522</td>
<td>-0.18348</td>
<td>-0.28209</td>
<td>0.92343</td>
<td>0.28045</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-5</td>
<td>-5</td>
<td>11</td>
<td>3257.70</td>
<td>80.33</td>
<td>1.53488</td>
<td>0.09791</td>
<td>-0.01594</td>
<td>-0.62499</td>
<td>-0.18348</td>
<td>-0.23003</td>
<td>0.92343</td>
<td>0.50055</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-5</td>
<td>-5</td>
<td>11</td>
<td>1644.98</td>
<td>47.54</td>
<td>1.49418</td>
<td>0.09783</td>
<td>-0.01594</td>
<td>-0.60686</td>
<td>-0.18348</td>
<td>-0.37869</td>
<td>0.92343</td>
<td>0.46234</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- OAK RIDGE National Laboratory
- HIGH FLUX ISOTOPIC REACTOR
- SPALLATION NEUTRON SOURCE
## Programs for Single Crystal Structure Analysis

<table>
<thead>
<tr>
<th>Program</th>
<th>Description</th>
<th>Website</th>
</tr>
</thead>
<tbody>
<tr>
<td>SHELX-2013</td>
<td>Structure solution and refinement</td>
<td><a href="http://shelx.uni-ac.gwdg.de/SHELX/index.php">http://shelx.uni-ac.gwdg.de/SHELX/index.php</a></td>
</tr>
<tr>
<td>JANA2006</td>
<td>Modulated and magnetic structures</td>
<td><a href="http://jana.fzu.cz/">http://jana.fzu.cz/</a></td>
</tr>
<tr>
<td>GSAS</td>
<td>Single crystal and powder data</td>
<td><a href="http://www.aps.anl.gov/Xray_Science_Division/Powder_Diffraction_Crystallography/GSASEXPGUI.html">http://www.aps.anl.gov/Xray_Science_Division/Powder_Diffraction_Crystallography/GSASEXPGUI.html</a></td>
</tr>
<tr>
<td>WinGX</td>
<td>Windows system of programs for solving, refining and analysing single crystal X-ray diffraction data for small molecules.</td>
<td><a href="http://www.chem.gla.ac.uk/~louis/software/wingx/">http://www.chem.gla.ac.uk/~louis/software/wingx/</a></td>
</tr>
</tbody>
</table>
Applications of Single Crystal Diffraction

• **Small molecule in Chemistry and Materials Science**
  A neutral or ionic compound of synthetic or biological origin but it is not a polymer, protein or nucleic acid:
  – Inorganic and Organic Compounds
  – Catalysts
  – Natural Products
  – Pharmaceuticals
  – Synthetic Chemicals

• **Chemical Crystallography**
  – Use single crystal X-ray/Neutron diffraction methods to determine the three dimensional structure of a *small molecule* at *atomic resolution*.
    – Study the relationship between molecular structure and chemical, biochemical or biological properties.
Applications of Neutron TOF Laue diffraction

- **TOPAZ** is a high resolution time-of-flight Laue diffractometer.
  - It is capable of measuring a 3D volume of reciprocal space from a stationary single crystal using an array of state-of-the-art neutron area detectors with microsecond readout time for individual neutron events.

- **Chemical crystallography** complementary to X-rays
  - The nature of the hydrogen bond
  - The structures of metal hydrides
  - Discern the ordering and positions of neighboring elements

- **Magnetic structure**

- **Diffuse scattering**
  - Total scattering sensitive to light elements

- **Parametric study** of nuclear and magnetic structural phase transitions
  - Neutron event-based technique
TOPAZ science areas

- Correlated electrons: 19%
- Magnetism: 6%
- Life Science: 6%
- Energy materials: 13%
- Earth and Env Science: 6%
- Chemistry: 31%
- Alloys/metalurgy: 0%
- Nanoscale/disordered: 0%
Accurate atomic displacement parameters from time-of-flight neutron-diffraction data at TOPAZ

Mads R. V. Jørgensen, a Venkatesha R. Hathwar, a Mattia Sist, a Xiaoping Wang, b Christina M. Hoffmann, b Alejandro L. Briseno, c Jacob Overgaard, a* and Bo B. Iversen, a*

a Center for Materials Crystallography, Department of Chemistry and iNano, Aarhus University, Langelandsgade 140, Aarhus C, DK-8000, Denmark, b Chemical and Engineering Materials Division, BL-12 TOPAZ, Oak Ridge National Laboratory, PO Box 2008 - MS 6475, Oak Ridge, TN 37831, USA, and c Department of Polymer Science and Engineering, University of Massachusetts, 120 Governors Drive, Amherst, MA 01003, USA. Correspondence e-mail: jacob@chem.au.dk, bo@chem.au.dk

Accurate atomic displacement parameters (ADPs) are a good indication of high-quality diffraction data. Results from the newly commissioned time-of-flight Laue diffractometer TOPAZ at the SNS are presented. Excellent agreement is found between ADPs derived independently from the neutron and X-ray data emphasizing the high quality of the data from the time-of-flight Laue diffractometer.
Resolving H atom positions

Difference Fourier map at 0.5 Å resolution, TOPAZ data

Blue: Nuclear density of H atoms

Rubrene, C_{42}H_{28}

\(R_w(F), R(F^2)\) 0.046, 0.088
\(\Delta \rho, \text{fmÅ}^{-3}\) -0.31, 0.23

[Cu_{32}(H)_{20}\{S_2P(O)iPr\}_2]_{12}

The largest number of hydrides recorded in a molecular nanocluster by single crystal neutron diffraction

The induction of chirality in metal hydrides is of added value because of the importance of such chiral complexes for asymmetric catalysis.

Crystal Structure of Cu$_{20}$H$_{11}$\{Se$_2$P(OiPr)$_2$\}$_9$ (Cu cyan, Hydride red)

C$_{81}$H$_{172}$Cu$_{20}$O$_{18}$P$_9$Se$_{18}$
Space group $R$-3
$a = 18.3282(2)$ Å
$c = 74.752(2)$ Å
$V = 21746.6(6)$ Å$^3$
$R_1$ (obs) = 0.0672

Hydrogenated sample
54% Hydrogen contents by atom.
Well resolved hydrogen atom positions
10 Hydrides as capping $\mu_3$-H ligands
1 Hydride as a $\mu_5$-H ligand in trigonal-bipyramidal cavity
No-classical bonding of a side-on $\text{H}_2$ ligand

- Open-shell Co-H2 moiety in (TPB)Co($\text{H}_2$)
  - Putative catalytic intermediates in Co-catalyzed proton reduction reactions
- Single crystal neutron diffraction structure of Co–$\text{H}_2$
  - A side-on $\text{H}_2$ ligand with H–H distance of 0.834(6) Å.

Neutron structure validates predictions that distortions of the complex in the solid state quenches free-rotor behavior of the $\text{H}_2$ ligand.

Neutron structure of Co–$\text{H}_2$ showing the disordered $\text{H}_2$ ligand in the solid state

Hydrogen Fuel Cell

Converts chemical energy into electricity

\[ \text{Anode: } 2H_2 \rightarrow 4H^+ + 4e \]

\[ \text{Cathode: } O_2 + 4H^+ + 4e \rightarrow 2H_2O \]

\[ 2H_2 + O_2 \rightarrow 2H_2O + \text{Electricity} \]

Pt catalyst 0.125 – 0.30 mg/cm²

Energy stored in the form of H–H bond

en.wikipedia.org/wiki/Fuel_cell
transportevolved.com
Hydrogenase

Catalyzes the reversible oxidation of molecular hydrogen

The cleavage of $\text{H--H}$ bonds cannot be readily observed by X-rays

Ogata, H.; Nishikawa, K.; Lubitz, W. Nature 2015, 520, 571
H-bond transformation in electrocatalyst

• **Hydrogen fuel cell** Convert chemical bond to electricity
  – Need a viable electrocatalyst (not Pt)
  – Iron-based molecular electrocatalyst

  *Electrocatalyst from earth abundant elements*

• **Neutron structural study of an iron-based electrocatalyst**
  – Hydrogen bonding related to H₂ oxidation
    Heterolytic cleavage of H–H bond
  – Hydrogen bonding related to H₂ production
    Heterocoupling of a proton and a hydride

**High resolution neutron single crystal diffraction**
Electrocatalysts Using Earth Abundant Elements

The Elements According to Relative Abundance

A Periodic Chart by Prof. Wm. F. Sheehan, University of Santa Clara, CA 95053

Roughly, the size of an element’s own niche (“I almost wrote square”) is proportioned to its abundance on Earth’s surface, and in addition, certain chemical similarities (e.g., Be and Al, or B and Si) are suggested by the positioning of neighbors. The chart emphasizes that in real life a chemist will probably meet O, Si, Al, . . . and that he better do something about it. Periodic tables based upon elemental abundance would, of course, vary from planet to planet . . . W.F.S.

NOTE: TO ACCOMMODATE ALL ELEMENTS SOME DISTORTIONS WERE NECESSARY. FOR EXAMPLE SOME ELEMENTS DO NOT OCCUR NATURALLY.
Hydrogen Oxidation Reaction

• Three steps
  – Reaction of H₂ with the metal complex
  – Heterolytic cleavage of H₂ into a proton and a hydride ion
  – Release of the two protons and two electrons

\[ H_2 \rightarrow (H^+ + H^-) \rightarrow 2H^+ + 2e \]

Hydrogen Oxidation
(Heterolytic Cleavage of H–H Bond)
Pendant amine as proton relay

Mimic the second coordination sphere at the enzyme active site

\[ [\text{CpFeN}_2\text{P}_2](\text{BArF}) \] (1)
Design a Functional Electrocatalyst

Facilitate proton-coupled electron transfer

\[ C_T = \left[ H^+ + H^- \right]^{\ddagger} \]

\[ V = \nu[C_T] \times 10^{-\left( \frac{E^\ddagger}{2.3RT} \right)} \]

The postulated “dihydrogen” bond

- Non-covalent interaction between hydrogen atoms with partial negative and positive charges

\[ M-H^\delta-\cdots H^\delta+ - X \]

- Hydride as proton acceptor

Neutron single crystal diffraction

- Capture the structure details of the electrocatalyst in action
  - The electrocatalyst is at its intermediate state
  - Hydrogenated sample (39% H by atom)
  - Locate hydrogen atoms at sub-atomic resolution
Locate hydrides and protons from single-crystal neutron diffraction

Sub-atomic resolution

$H^{\delta+}\cdots H^{\delta-} \quad 1.489(10) \, \text{Å}$
Neutron structure – Dihydrogen bond

Distance, Å  [FeH−HN]⁺
Fe1−H1B  1.544(7)
N1−H1A  1.079(6)
Fe1−P1  2.157(3)
Fe1−P2  2.160(3)

{Fe(II)−H⁻} acts as the H-bond acceptor
The shortest H···H distance observed between the hydridic Fe−H⁻ and protic N−H⁺ site

H⁺···H⁻  1.489(10) Å

C₆₈H₆₇BF₂₉FeN₃P₂
Monoclinic, P2₁/c
a = 13.9202 (3) Å  \( R[F^2 > 2\sigma(F^2)] = 0.067 \)
b = 36.0546 (13) Å  \( wR(F^2) = 0.150 \)
c = 19.5234 (4) Å  \( S = 1.07 \)
\( \beta = 133.6892 (13)° \)  25230 reflections
V = 7085.3 (3) Å³  1628 parameters
Z = 4  110 restraints

0.32 x 0.90 x 1.95
Transfer of H atoms in the solid state

H₂O
Transfer of H atoms in SCS reaction

$H_2$
Locating Hydrogen atoms in $[1-\text{Fe(OH}_2\text{)}\text{N}]^+$

Difference map
Showing nuclear densities from missing H atoms
Locating Hydrogen atoms in \([1-\text{Fe(OH}_2\text{)N}]^+\)

- Highest peak: 0.71 at 0.0393 0.8773 0.4887 [0.14 Å from C10]
- Deepest hole: -0.71 at 0.1124 0.8352 0.2988 [0.42 Å from H9A]

\[
\begin{align*}
\text{wR}2 & = 0.1245 \text{ before cycle 8 for 13638 data and 0 / 1569 parameters} \\
\text{GooF} & = S = 1.022; \text{ Restrained GooF} = 1.024 \text{ for 109 restraints} \\
R1 & = 0.0721 \text{ for 11858 Fo > 4\text{sig}(Fo)} \text{ and 0.0777 for all 13638 data} \\
\text{wR2} & = 0.1245, \text{ GooF} = S = 1.022, \text{ Restrained GooF} = 1.024 \text{ for all data} \\
R1 & = 0.0717 \text{ for 5831 unique reflections after merging for Fourier}
\end{align*}
\]
Comparison of Neutron & X-ray structures

<table>
<thead>
<tr>
<th></th>
<th>X-ray</th>
<th>Neutron</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-O1</td>
<td>2.022</td>
<td>2.032</td>
</tr>
<tr>
<td>H1a-O1</td>
<td>0.852</td>
<td>0.995</td>
</tr>
<tr>
<td>H1b-O1</td>
<td>1.693</td>
<td>1.096</td>
</tr>
<tr>
<td>Hb-N1</td>
<td>0.930</td>
<td>1.503</td>
</tr>
<tr>
<td>Fe-P</td>
<td>2.208</td>
<td>2.208</td>
</tr>
<tr>
<td>O1-Hb-N1</td>
<td>162.74</td>
<td>162.3</td>
</tr>
<tr>
<td>P1-Fe-P2</td>
<td>81.88</td>
<td>82.25</td>
</tr>
</tbody>
</table>

X-ray structure
Tianbiao Liu
Reaction pathway for $\text{H}_2$ production

Spontaneous combination of a proton with a hydride

$$\text{H}_2\text{O} + [\text{1-FeH---HN}]^+ \rightarrow \text{H}_2 + [\text{1-Fe(OH}_2\text{)N}]^+$$

Hetero-coupling of a proton and a hydride

$$\text{H}^+ + \text{H}^- \rightarrow \text{H}_2$$
Heterolytic Cleavage of Hydrogen by an Iron Hydrogenase Model: An Fe-H···H-N Dihydrogen Bond Characterized by Neutron Diffraction**

Tianbiao Liu,* Xiaoping Wang, Christina Hoffmann, Daniel L. DuBois, and R. Morris Bullock*
Hybrid organic—inorganic perovskites (HOIPs)

Methylammonium Lead Halide(s)

MAPbI$_3$ Electronic structure

- Direct bandgap semiconductor
- High symmetry Brillouin zone boundary points
  - R, M, X
  - Optical bandgap is located at the R point in the cubic phase

- High optical absorption coefficient
  - Thin films harvest light effectively

Hybrid organic—inorganic perovskites (HOIPs)

Methylammonium Lead Halide(s) \( \text{MAPbX}_3 \)

Perovskite: \( \text{ABX}_3 \)
Space group: \( \text{Pm}-3m \)

Positions of MA is difficult to Locate using X-rays
Neutron diffraction study of MAPbI$_3$

**Neutron** scattering lengths

H -3.74 fm

Comparable for N and Pb
Cubic-tetragonal phase transition in MAPbI₃

Crystal Growth & Design 2016, 16, 2945-2951

Orientation of Organic Cations in Hybrid Inorganic–Organic Perovskite CH₃NH₃PbI₃ from Subatomic Resolution Single Crystal Neutron Diffraction Structural Studies

Yixin Ren,† Iain W. H. Oswald,† Xiaoping Wang,*‡ Gregory T. McCandless,† and Julia Y. Chan*†

†Department of Chemistry and Biochemistry, The University of Texas at Dallas, Richardson, Texas 75080, United States
‡Chemical and Engineering Materials Division, Neutron Sciences Directorate, Oak Ridge National Laboratory, Oak Ridge, Tennessee 39831, United States
Single crystal growth

- MAPbI$_3$

As-grown well-faceted single crystals from a supersaturated solution at room temperature.
Hydrogen bonding in hybrid perovskite CH$_3$NH$_3$PbI$_3$

- Contains heavy elements Pb and I
  - High X-ray absorption $\mu = 526.82$ cm$^{-1}$
  - **Transparent to neutrons** $\mu = 0.654 + 0.508\lambda$ cm$^{-1}$

- Effect of H-bonding on structural phase transitions

  - Cubic
  - Tetragonal
  - Orthorhombic
Positive nuclear density has a larger value at the four exterior lobes indicating the presence of nitrogen (shown in silver). The central disordered area has a lower scattering factor due to the presence of carbon (shown in brown) that is disordered to generate a total of eight positions.
3D difference Fourier maps from single crystal neutron diffraction data for CH$_3$NH$_3$PbI$_3$, (a) showing the region of positive (yellow) nuclear density indicates the disorder of the CH$_3$NH$_3^+$ cation, (b) showing the region of negative (blue) nuclear indicates a highly disordered arrangement of the hydrogen atoms.
Cubic, Pm-3m

3D nuclear difference Fourier maps for CH$_3$NH$_3^+$ at 350 K. (a) Region of positive (yellow) nuclear density indicates disorder of C and N atoms in CH$_3$NH$_3^+$. (b) Location of disordered CN groups (N blue, C brown) and H atoms from refinement of the neutron structure. (c) View of the structure of CH$_3$NH$_3$PbI$_3$ showing the region of negative (blue) nuclear and a highly disordered arrangement of the hydrogen atoms. (d) Location of CH$_3$NH$_3^+$ with the CN bond aligned along the [011] direction.
Structure of a Magnetic Shape Memory Alloy
Harness the power of wavelength-resolved Laue for 3D Q-space mapping

- Data measured on TOPAZ are used to describe simultaneous structural, compositional and magnetic modulations of Ni$_2$Mn$_{1.16}$Ga$_{0.84}$.
- Both nuclear and magnetic structures were successfully refined in JANA2006 using 3D+1 superspace approach.

Neutron Crystal Structure of Ni$_2$Mn$_{1.16}$Ga$_{0.84}$

Structure refinement in JANA2006 using 3D+1 superspace approach, 5M $\rightarrow$ $q \approx 0.4c$

Displacement of Mn atoms in the 5M structure
Expanding Lorentz and spectrum corrections to large volumes of reciprocal space for single-crystal time-of-flight neutron diffraction

T. M. Michels-Clark, A. T. Savici, V. E. Lynch, X. P. Wang and C. M. Hoffmann

Extraction of structure factor amplitudes from total scattering (Bragg + diffuse diffraction) data for local structure modeling remains a significant challenge, especially when considering time-of-flight measurements. Individual intensity contributions carry different statistical weights, with associated distinctive effects on data summation and correction. A comprehensive, statistically improved data analysis approach to correct and scale the complete volume of reciprocal space data in one step is described.
Event data collection for parametric study

- VT study the structural phase transition of a ferroelectric material

- Place preselected Bragg peak on selected detector position
  - CrystalPlan

- Measure while cooling
  - Stationary single crystal
  - Neutron recorded in event mode
    - Detector pixel position (x, y)
    - Neutron time of flight (λ)
  - Sample temperature (T, K)
    - Link to event data with a time stamp

- Data saved as event nexus file
  - Stores (3 + n) D information
    - \( n = 1 \) for VT experiment

- Data analysis uses Mantid software
Neutron structure analysis using SHELX program

- **SHELX-2013**
  
  [http://shelx.uni-ac.gwdg.de/SHELX/download.php](http://shelx.uni-ac.gwdg.de/SHELX/download.php)

- **GUI**
  
  - **ShelXle**
    
    [https://www.shelxle.org/shelx/eingabe.php](https://www.shelxle.org/shelx/eingabe.php)
  
  - **WinGX Program System**
    
    [http://www.chem.gla.ac.uk/~louis/software/](http://www.chem.gla.ac.uk/~louis/software/)
  
  - **Olex² Crystallography Software**
    
    [http://www.olexsys.org/Software](http://www.olexsys.org/Software)

- **Workshop Talks**

  - **George Sheldrick: SHELXL for neutrons (TOPAZ Oak Ridge 2015)**
    
    [http://shelx.uni-ac.gwdg.de/SHELX/shelxl_for_neutrons.pdf](http://shelx.uni-ac.gwdg.de/SHELX/shelxl_for_neutrons.pdf)

  - **Xiaoping Wang: Refinement of small molecules against neutron data (ACA, 2016)**
    
    [http://shelx.uni-ac.gwdg.de/SHELX/neutrons_SM_ACA2016.pdf](http://shelx.uni-ac.gwdg.de/SHELX/neutrons_SM_ACA2016.pdf)
Become a user

http://neutrons.ornl.gov/users

Neutron Sciences Call for Proposals
Due April 12, 2017

Proposals for beam time at Oak Ridge National Laboratory’s High Flux Isotope Reactor (HFIR) and Spallation Neutron Source (SNS) will be accepted via the web-based proposal system until 11:59 a.m. (EDT), Wednesday, April 12, 2017. This call is for experiments anticipated to run from July–December 2017.

Information and instructions
To learn more about submitting a proposal for beam time, go to neutrons.ornl.gov/users or directly to the proposal system at www.ornl.gov/sci/rums/ppts/. Previously submitted proposals may be used as the basis for new proposals. All proposals will be reviewed for feasibility, safety, and the potential for high-impact science. Before beginning approved experiments, users must complete access and training requirements and ensure that the appropriate user agreements are in place.

Available Instruments for General Users

<table>
<thead>
<tr>
<th>HFIR</th>
<th>SNS</th>
</tr>
</thead>
<tbody>
<tr>
<td>• HB-1 Polarized Triple-Axis Spectrometer</td>
<td>• BL-1A Ultra-Small-Angle Neutron Scattering Instrument (USANS)</td>
</tr>
<tr>
<td>• HB-1A Fixed-incident-Energy Triple-Axis Spectrometer</td>
<td>• BL-1B Nanoscale-Ordered Materials Diffractometer (NOMAD)</td>
</tr>
<tr>
<td>• HB-2A Neutron Powder Diffractometer</td>
<td>• BL-2 Backscattering Spectrometer (BASIS)</td>
</tr>
<tr>
<td>• HB-2B Neutron Residual Stress Mapping Facility</td>
<td>• BL-3 Spallation Neutrons and Pressure Diffractometer (SNAP)</td>
</tr>
<tr>
<td>• HB-2C US/Japan Wide-Angle Neutron Diffractometer (WAND)</td>
<td>• BL-4A Magnetism Reflectometer (MR)</td>
</tr>
<tr>
<td>• HB-3 Triple-Axis Spectrometer</td>
<td>• BL-4B Liquids Reflectometer (LR)</td>
</tr>
<tr>
<td>• HB-3A Four-Circle Diffractometer</td>
<td>• BL-5 Cold Neutron Chopper Spectrometer (CNCS)</td>
</tr>
<tr>
<td>• CG-1D Neutron Imaging</td>
<td>• BL-6 Extended Q-Range SANS (EQSANS)</td>
</tr>
<tr>
<td>• CG-2 General-Purpose SANS</td>
<td>• BL-7 Engineering Materials Diffractometer (VULCAN)</td>
</tr>
<tr>
<td>• CG-3 Bio-SANS</td>
<td>• BL-8 Elastic Diffuse Scattering Spectrometer (CORELLI)</td>
</tr>
<tr>
<td>• CG-4C Cold Neutron Triple-Axis Spectrometer</td>
<td>• BL-9A Powder Diffractometer (POWGEN)</td>
</tr>
<tr>
<td>• CG-4D Image-Plate Single-Crystal Diffractometer (IMAGINE)</td>
<td>• BL-11B Macromolecular Neutron Diffractometer (MaNDI)</td>
</tr>
</tbody>
</table>

For more information on any of these instruments go to neutrons.ornl.gov, or contact the Neutron Sciences User Office, neutronusers@ornl.gov or (865) 574-4600.
The neutron single crystal study at ORNL’s Spallation Neutron Source TOPAZ instrument was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, US Department of Energy.

http://neutrons.ornl.gov/topaz/