Neutron and X-ray Sources
Producing neutrons

**Fission**
Nuclear reactor

**Spallation**
Particle accelerator

Moderators → Cold-Thermal

Moderators → Cold-Epithermal
Neutrons by reactor fission

High flux isotope reactor - ORNL

NIST
High Flux Isotope Reactor at Oak Ridge National Laboratory

The United States’ highest flux reactor-based source of neutrons for condensed matter research

**Fixed-incident-Energy Triple-Axis Spectrometer • HB-1A**
- Low-energy excitations, magnetism, structural transitions
  - Jerrel Zaretsky • 865.574.6951
  - zaretsky@ornl.gov

**Triple-Axis Spectrometer • HB-1**
- Polarized neutron studies of magnetic materials, low-energy excitations, structural transitions
  - Andrey Zholudev • 865.241.0318
  - zholudev@ornl.gov

**Neutron Powder Diffractometer • HB-2A (2008)**
- Structural studies, magnetic structures, texture and phase analysis
  - Ovidiu Gardea • 865.574.3041
  - gardea@ornl.gov

**US/Japan WAND • HB-2C**
- Diffuse-scattering studies of single crystals and time-resolved phase transitions
  - Jaime Fernandez-Duque • 865.574.3659
  - fernandezduque@ornl.gov

**Neutron Residual Stress Mapping Facility • HB-2B**
- Strain and phase mapping in engineering materials
  - Camden Hubbard • 865.574.4472 • hubbardc@ornl.gov

**Future Development • HB-2D**

**Future Development • CG-1**

**General-Purpose SANS • CG-2**
- Polymer blends, flux lattices in high-Tc materials, soft materials processing and structure
  - Ken Liboff • 865.574.4535 • liboffk@ornl.gov

**Bio-SANS • CG-3**
- Proteins and complexes, pharmaceuticals, biomaterials
  - Walter Urban • 865.574.2078
  - urbanw@ornl.gov

**Future Development • CG-4D**

**Future Development • CG-4**

**Future Development • CG-4C (2009)**
- High-resolution inelastic scattering at cold neutron energies
  - Barry Wise • 865.241.0082
  - wiserb@ornl.gov

**Future Development • CG-4A**
- Small unit-cell crystal structural studies, particularly H-bonding
  - Bjorn Christiansen • 865.574.3320
  - chansk@bnl.gov

**Future Development • CG-4B**
- Medium- and high-resolution inelastic scattering at thermal energies
  - Mark Lovern • 865.241.0060
  - lovernm@ornl.gov

**Future Development • CG-3**

**Future Development • CG-2**

**Future Development • CG-1**

**Cold Neutron Source**

*Scheduled commissioning date.*
Neutrons by pulsed spallation

Spallation Neutron Source (ORNL)
Target-moderator system

SNS liquid Hg target

Fig. 3. Horizontal cross-section of the flux-trap moderators.
Spallation Neutron Source at Oak Ridge National Laboratory

The world’s most intense pulsed, accelerator-based neutron source

**Backscattering Spectrometer (BASIS) - BL-2**
- Dynamics of macromolecules, constrained molecular systems, polymers, biology, chemistry, materials science
  - Eugene Mamonov - 865.372.3009 - mamontova@ornl.gov

**Nanoscale-Ordered Materials Diffractometer (NOMAD) - BL-1B (2010)**
- Liquids, solutions, glasses, polymers, nanocrystalline and partially ordered complex materials
  - Joerg Neumeier - 865.341.1638 - neumeierj@ornl.gov

**Wide Angular-Range Chopper Spectrometer (ARCS) - BL-1B**
- Atomic-level dynamics in materials science, chemistry, condensed matter sciences
  - Doug Abernathy - 865.372.5185 - abernathyd@ornl.gov

**Fine-Resolution Fermi Chopper Spectrometer (SEQUOIA) - BL-17 (2008)**
- Dynamics of complex fluids, quantum fluids, magnetism, condensed matter, materials science
  - Garrett Granroth - 865.576.0900 - granrothg@ornl.gov

**Ultra-Small-Angle Neutron Scattering Instrument (TOF-USANS) - BL-1A (2012)**
- Life sciences, polymers, materials science, earth and environmental sciences
  - Michael Agozzino - 865.372.4004 - agozzinm@ornl.gov

**Chemical Spectrometer (VISION) - BL-16B (2011)**
- Vibrational dynamics in molecular systems, chemistry
  - Christoph Wildgruber - 865.374.6376 - wildgruber@ornl.gov

**Neutron Spin Echo Spectrometer (NSE) - BL-15 (2009)**
- High-resolution dynamics of slow processes, polymers, biological macromolecules
  - Michael Cain - 865.371.8499 - acain@ornl.gov

**Cold Neutron Chopper Spectrometer (CNCS) - BL-5**
- Condensed matter physics, materials science, chemistry, biology, environmental science
  - Georg Ehlers - 865.376.3811 - ehlersg@ornl.gov

**Magnetism Reflectometer - BL-4A**
- Chemistry, magnetism of layered systems and interfaces
  - Valeria Lauter - 865.372.5380 - lauterv@ornl.gov

**Liquids Reflectometer - BL-4B**
- Interfaces in complex fluids, polymers, chemistry
  - John Ankerer - 865.372.5182 - ankererj@ornl.gov

**Extended Q-Range Small-Angle Neutron Scattering Diffractometer (EQ-SANS) - BL-6 (2008)**
- Life science, polymer, and colloidal systems, materials science, earth and environmental sciences
  - Jinzhu Zhao - 865.372.0411 - zhaoj@ornl.gov

**Elastic Diffuse Scattering Spectrometer (CORELLI) - BL-9 (2013)**
- Detailed studies of disorder in crystalline materials
  - Fang Wu - 865.372.0921 - yufli@ornl.gov

**Macromolecular Neutron Diffractometer (MaNDI) - BL-11B (2012)**
- Atomic-level structures of membrane proteins, drug complexes, DNA
  - Leighann Costas - 865.576.8103 - leighannc@ornl.gov

**Powder Diffractometer (POWGEN) - BL-11A (2008)**
- Atomic-level structures in magnetism, chemistry, materials sciences
  - Jason Hodges - 865.372.7024 - hodgesj@ornl.gov

**Fundamental Neutron Physics Beam Line - BL-13 (2008)**
- Fundamental properties of neutrons
  - Geoffrey Greene - 865.372.4490 - greene@ornl.gov

**Single-Crystal Diffractometer (TOPAZ) - BL-12 (2009)**
- Atomic-level structures in chemistry, biology, earth science, materials science, condensed matter physics
  - Christine Hoffmann - 865.372.3317 - hoffmannc@ornl.gov

**Hybrid Spectrometer (HYSPEC) - BL-14B (2011)**
- Atomic-level dynamics in single crystals, magnetism, condensed matter sciences
  - Mark Hegwood - 865.341.8703 - hegwoodm@ornl.gov

**Engineering Materials Diffractometer (VULCAN) - BL-7 (2008)**
- Mechanical behaviors, materials science, materials processing
  - Xunli Wang - 865.372.0164 - wangxl@ornl.gov
Reactor vs. Spallation

• Reactors:
  - Continuous source
  - Limited high energy flux (except for hot sources)
  - Licensing issues

• Spallation Sources
  - High peak flux (but lower average flux)
  - Time structure (Time-of-flight techniques)
  - Complex data reduction and analysis, but a very good way to see all regions of Q and E.
Production of x-rays

Characteristic x-rays

X-rays from a molybdenum target at 35 kV

Relative intensity

Kα

Kβ

Bremssstrahlung continuum

Wavelength (nm)
Synchrotron radiation

- Static charge ---- Electric field
- Charge moving at constant $v$ ---- Magnetic field
- Accelerating charge --- Electromagnetic radiation

Synchrotron radiation is
- highly collimated
- highly linearly polarized
- highly brilliant
- continuous wavelength distribution (beyond Cu, Mo, etc..)
Advanced Photon Source

Synchrotron
Insertion Devices

High Energy Undulator Flux

Flux in 1x1mm² pinhole @ 30m (ph/0.1%bw)

- UA
  - λ=3.3cm
  - gap=10.5cm
  - K=2.62
- HEX-1
  - λ=2.25cm
  - gap=8.5mm
  - K=1.26

Energy (keV)
Main Undulator Line
Places to go
Single crystal x-ray and neutron diffraction
Periodic crystals

7 crystal systems (cubic, tetragonal, orthorhombic, monoclinic, trigonal, hexagonal)
14 Bravais lattices (above + centering (body, base, face))
230 periodic space groups (14 Bravais lattices + 32 crystallographic point groups)
Diffraction... Bragg’s Law

\[ n\lambda = 2dsin\theta \]
Lost in reciprocal space

For an infinite 3D lattice defined by primitive vectors \( \mathbf{a}_1, \mathbf{a}_2, \mathbf{a}_3 \) we can define a reciprocal lattice generated by:

\[
\begin{align*}
\mathbf{b}_1 &= 2\pi \frac{\mathbf{a}_2 \times \mathbf{a}_3}{\mathbf{a}_1 \cdot (\mathbf{a}_2 \times \mathbf{a}_3)} \\
\mathbf{b}_2 &= 2\pi \frac{\mathbf{a}_3 \times \mathbf{a}_1}{\mathbf{a}_2 \cdot (\mathbf{a}_3 \times \mathbf{a}_1)} \\
\mathbf{b}_3 &= 2\pi \frac{\mathbf{a}_1 \times \mathbf{a}_2}{\mathbf{a}_3 \cdot (\mathbf{a}_1 \times \mathbf{a}_2)}
\end{align*}
\]

For \( \mathbf{R} = m_1 \mathbf{a}_1 + m_2 \mathbf{a}_2 + m_3 \mathbf{a}_3 \) and \( \mathbf{G} = m_1 \mathbf{b}_1 + m_2 \mathbf{b}_2 + m_3 \mathbf{b}_3 \)

\[ e^{i\mathbf{G} \cdot \mathbf{R}} = 1; \quad \mathbf{G} \cdot \mathbf{R} = 2\pi \times \text{integer} \]

\( \mathbf{G} \) is normal to sets of planes of atoms spaced \( 2\pi / G \) apart

Each point \((hkl)\) in the reciprocal lattice corresponds to a set of planes \((hkl)\) in the real space lattice.
Miller indices and reciprocal space

(100) Reflection = diffraction from planes of atoms spaced $2\pi/a$ apart
(200) Reflection = diffraction from planes of atoms spaced $2\pi/2a$ apart
Reciprocal space and diffraction

\[ n\lambda = 2d_{hkl}\sin\theta \leftrightarrow k - k_0 = G; \quad k = k_0 = \frac{2\pi}{\lambda} \]

Ewald sphere; Radius = \(\frac{2\pi}{\lambda}\)

For single crystal diffraction – both the detector angle and the sample orientation matter
Courtesy of the CSIC (Spanish National Research Council).
Intensities (Neutrons)

In General:

\[
\lim_{\Omega_{f},E_{f} \to 0} \frac{d^{2}\sigma}{d\Omega_{f}dE_{f}}|_{coh} = N \frac{k_{f}}{k_{i}} \frac{\sigma_{coh}}{4\pi} S(Q, \omega).
\]

\[
S(Q, \omega) = \frac{1}{2\pi \hbar N} \sum_{ll'} \int_{-\infty}^{\infty} dt \langle e^{-iQ \cdot \tau_{l'}(0)} e^{iQ \cdot \tau_{l}(t)} \rangle e^{-i\omega t}
\]

For elastic Bragg Scattering:

\[
\lim_{\Omega_{f},E_{f} \to 0} \frac{d\sigma}{d\Omega}|_{coh}^{el} = N \frac{(2\pi)^{3}}{v_{0}} \sum_{G} \delta(Q - G) |F_{N}(G)|^{2}
\]

\[
F_{N}(G) = \sum_{j} b_{j} e^{iG \cdot d_{j}} e^{-W_{j}}
\]
Measuring peak shapes

θ-2θ Step Scan

Longitudinal Scan

Omega Step Scan

Transverse Scan

- Detector stationary at 2θ angle.
- Crystal is rotated about θ by +/- ω.
- FWHM is the mosaic spread.
Why do single crystal diffraction (vs. powder diffraction)?

- Smaller samples – 1-10 mg vs 500-5000 mg
- Larger molecules and unit cells
- Hydrogen is ok – generally does not need to be deuterated
- Less absorption
- Fourier coefficients are more accurate – based on integrating well-resolved peaks
- Uniquely characterize non-standard scattering – superlattice and satellite peaks (commensurate and incommensurate), diffuse scattering (rods, planes, etc.)

**But:**
- Need to grow a single crystal
- Data collection can be more time consuming
CaFe$_2$As$_2$...an example
Temperature dependent studies

(1 1 10) reflection

Counts/s

Temperature $T$ in K:
- 2400 K
- 2000 K
- 1600 K
- 171 K
- 170.5 K
- 170 K
- 169.5 K
- 169 K
- 168.5 K
- 168 K
- 150 K
- 100 K
- 10 K

$\xi$ in $(\xi \xi 10)$

Intensity (arb. unit)

Lattice parameter ($\AA$)

Temperature $T$ (K)

$T_{LT}$

$T_{HT}$

$C_{LT}$

$C_{HT}$

$\bar{a}_{LT}$

$\bar{a}_{HT}$

$\bar{b}_{LT}$

$\bar{b}_{HT}$

$\bar{c}_{LT}$

$\bar{c}_{HT}$