Beamline 11-BM at the Advanced Photon Source:
Synchrotron Powder Diffraction Simplified…

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Argonne NATIONAL LABORATORY

What is 11-BM ?

11-BM is a dedicated high resolution synchrotron powder diffraction instrument at the Advanced Photon Source (APS)

Beamline 11-BM provides its users with access to the highest resolution synchrotron powder diffractometer available in North America. Our mission is operate an exceptional powder diffraction program for the diverse multi-disciplinary APS user community, and to enable superior crystallography with convenient and timely access to world-class powder diffraction data.

Why use a synchrotron for powder diffraction?

Many powder diffraction studies are information starved - - the structural detail one can extract is limited by the observable details of the measurement.

- **High-Resolution diffraction:** allows peaks to be resolved, essential for indexing, improves structure solution and provides optimal detail in crystal structures
- **High-Sensitivity diffraction:** allows weak peaks to be seen above background, essential for structural details and increases the number of observations
- **High-Energy diffraction:** provides more accurate data and a wider Q range energy (even more observations), less sample absorption for high Z samples
- **High-Throughput diffraction:** ensures these capabilities are widely available to a diverse range of research communities, including chemistry, materials, physics, geosciences, pharmaceutical science, structural biology, and beyond

Accessing Beamtime

Users may access the instrument via a mail-in service or on-site as a user at the APS. Use of the rapid access mail-in program is encouraged for new users and for projects requiring < 1 beamtime shift (8 hrs) of data collection.

11-BM is a free service for all non-proprietary users

Your 11-BM Experiment: Mail-In or On-Site?

Rapid Access Mail-In Proposal

- **Fast Review and Approval:** no deadlines, rapid access proposals accepted anytime, award decision typically within 5 days
- **Economical and Convenient:** no travel expenses, data typically available within 4 weeks of proposal submission
- **Limited in Duration & Scope:** limited to one shift per rapid access proposal, limited scan parameters and temperatures available

11-BM Mail-In sample base with barcode and capillary

Standard User Proposal

- **Proposal Review Deadlines:** 3 deadlines per year for committee review of standard proposals, time awarded depends on score and beamline subscription
- **Unique Scan Conditions:** user has greater control of scan parameters, full suite of samples environments and experimental setups available
- **On-Site or Mail-In:** required for proposed on-site user experiment, or for mail-in users requesting > 1 shift of beamtime per APS cycle

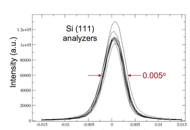
ADDITIONAL INFORMATION AVAILABLE

webpage: **http://11bm.xor.aps.anl.gov**

email: **11BM@aps.anl.gov**

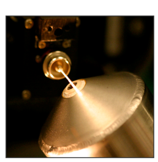
Instrument Specifications

- **Energy range:** 10 – 36 keV (1.3 – 0.3 Å)
  Mail-in service optimized @ 30 keV (~ 0.41 Å)
- **Angular scan range:** 0.3° – 150° 2θ
- **Q coverage:** $Q_{max} \approx 25$ Å^{-1}
  1 hour mail-in scan: 2θ range 0.5° → 50° (d ≥ 0.5 Å)
- **Resolution:** Δd/d (ΔQ/Q) ≈ 2×10^{-4}
  Equivalent to best-in-world (ESRF, Diamond etc.)
  Highest Resolution Powder Diffraction in Americas



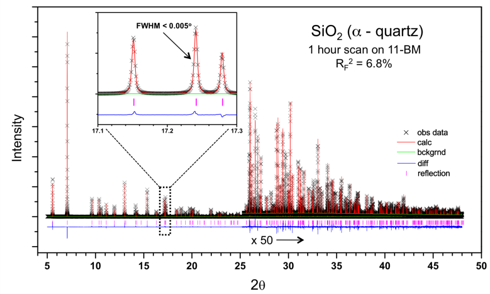
Overlaid rocking curves from all 12 of the Si(111) analyzer crystals at 11-BM

- **Beam Size:** 0.5 mm (vertical) x 1 mm (horizontal)
- **Sample Environments:**
  Oxford Cryostream 700+ (100 K - 450 K)
  Cyberstar Hot Gas Blower (25° C → 1000 °C)
  Helium Cryostat (295 K → 10 K) *coming fall 2010*
  In-Situ Gas Flow Reaction Cell *summer 2010*
  Diamond Anvil Pressure Cell *fall 2010*
- **Robotic Sample Changer:** automated loading of 100 + samples



Hot Gas Blower shown heating a spinning quartz capillary sample

High-Resolution Powder Diffraction at 11-BM

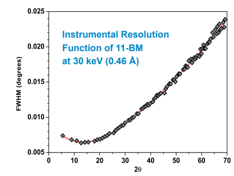


Exceptional Resolution and Count Sensitivity

Δd/d ≈ 2×10^{-4}

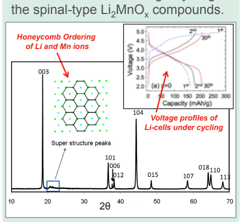
2θ FWHM ≈ 0.005°

count statistics ≈ 10^5



Chemical Ordering in Li-ion Battery Materials

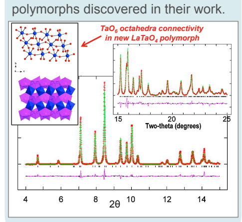
Lithium-ion batteries are key to addressing global demands for lightweight portable energy storage. High resolution powder diffraction measurements at 11-BM have been used to better understand the structural and ordering transitions which occur under voltage cycling in the spinal-type Li_2MnO_x compounds.



Clare P. Grey *et al*
J. Electrochem. Soc., **2009**, *156*, A730-A736
Chemistry of Materials, **2009**, *21*, 2733–2745

Synthetic Routes to New Photocatalyst Oxides

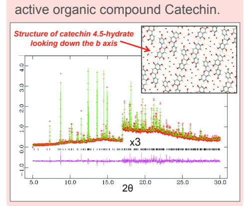
Sandia researchers are discovering new photocatalysis materials through their studies of novel low temperature synthesis routes for La-Ta and La-Nb based oxides. Powder diffraction data obtained from the 11-BM mail-in program were key to determining the chemical structures of the new polymorphs discovered in their work.



May Nyman, Mark A. Rodriguez *et al*
Chemistry of Materials, **2009**, *21*, 2201–2208
Chemistry of Materials, **2009**, *21*, 4731–4737

Complex Structures of Organic Compounds

High resolution powder diffraction is increasingly being used to solve the complex structures of organic materials which are inaccessible by single crystal methods. Recent analyses combining solid-state NMR and powder diffraction data from 11-BM have been used to finally reveal the extended structure of the bio-active organic compound Catechin.

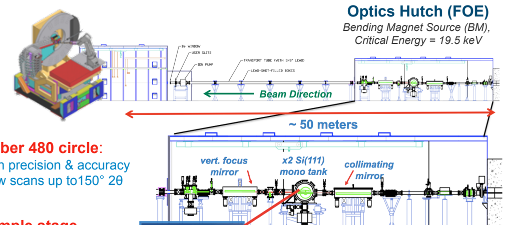


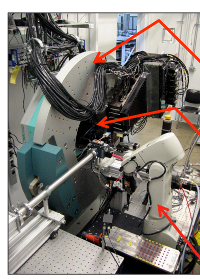
James K. Harper, Jennifer A. Doebbler *et al*
J. Am. Chem. Soc., **2010**, *132*, 2928–2937

Overview of Beamline

Experimental Hutch
Diffractometer, Multi-Crystal Detector Assembly and Auxiliary Equipment

Optics Hutch (FOE)
Bending Magnet Source (BM), Critical Energy = 19.5 keV

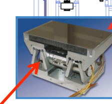




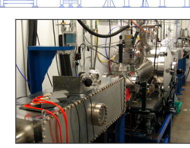
Huber 480 circle:
high precision & accuracy
slew scans up to150° 2θ

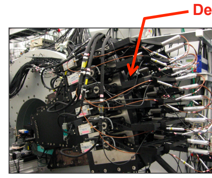
Sample stage
XYZ translation
spinner > 90 Hz
mounting for in-situ
pressure & gas cells

Robotic Sample Loading Arm
automatically mounts and runs
capillary samples, capacity > 100



Sagittally-Bent Crystal
Sagittally focusing Si(111)
2nd monochromator crystal





Detector Assembly
12 analyzer array
Si(111) crystals
Θ + X adjustment
2Θ spacing of 2°
$LaCl_3$ detectors

Beamline 11-BM is located at a bending magnet (E_c = 19.5 keV) on the 7 GeV APS storage ring. 11-BM operates over the energy range 10 keV - 36 keV. The optics hutch contains all beamline optics, including a Si/Pt collimating mirror, double-crystal Si (111) monochromator (27.6 m from source) with sagittal horizontal focusing, and a 1 meter vertical focusing mirror.

An experimental hutch (~ 50 m from source) contains a high-precision Huber two-circle diffractometer mounted on a large optical table. The table also supports sample environmental cells, a robotic sample changing arm, and other accessories. The diffractometer has a unique multi-analyzer detection assembly, consisting of 12 independent Si (111) crystal analyzers and $LaCl_3$ scintillation detectors. This enables simultaneous high-speed (~1 hour) and high-resolution (ΔQ/Q ≈ 2×10^{-4}) data collection.

Acknowledgements: We acknowledge all those at the APS who shared in the design, construction and commissioning of 11-BM, especially Mohan Ramanathan, Yeldez Amer, Deming Shu, Jun Wang, Peter Lee, Sytle Antao, Jennifer Doebler, Charles Kurtz, Mark Engbretson, Curt Preissner, Xuesong Jiao, David Cline & Tim Mooney, Yu Huang, Bill Sheehan, Dave Cyl, Mark Beno, Gabrielle Long, George Srajer. We also thank John Mitchell, the late James Jorgenson and Helen Kerch for their work.