Argonne NATIONAL LABORATORY

... for a brighter future

High-throughput powder diffraction on 11-BM:

Design and Execution

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July 2008

U.S. Department of Energy

THE UNIVERSITY OF CHICAGO

Office of Science
U.S. DEPARTMENT OF ENERGY

A U.S. Department of Energy laboratory managed by The University of Chicago

What do we need in a powder diffractometer?

Most powder diffraction studies are information starved -- the detail we can learn is limited by the detail in the experimental measurements. True for most powder diffraction problems.

High-resolution diffraction allows peaks to be resolved: essential for indexing and providing structural detail by providing many observations

High-sensitivity diffraction allows small peaks to be seen above background: essential for structural details (also more observations)

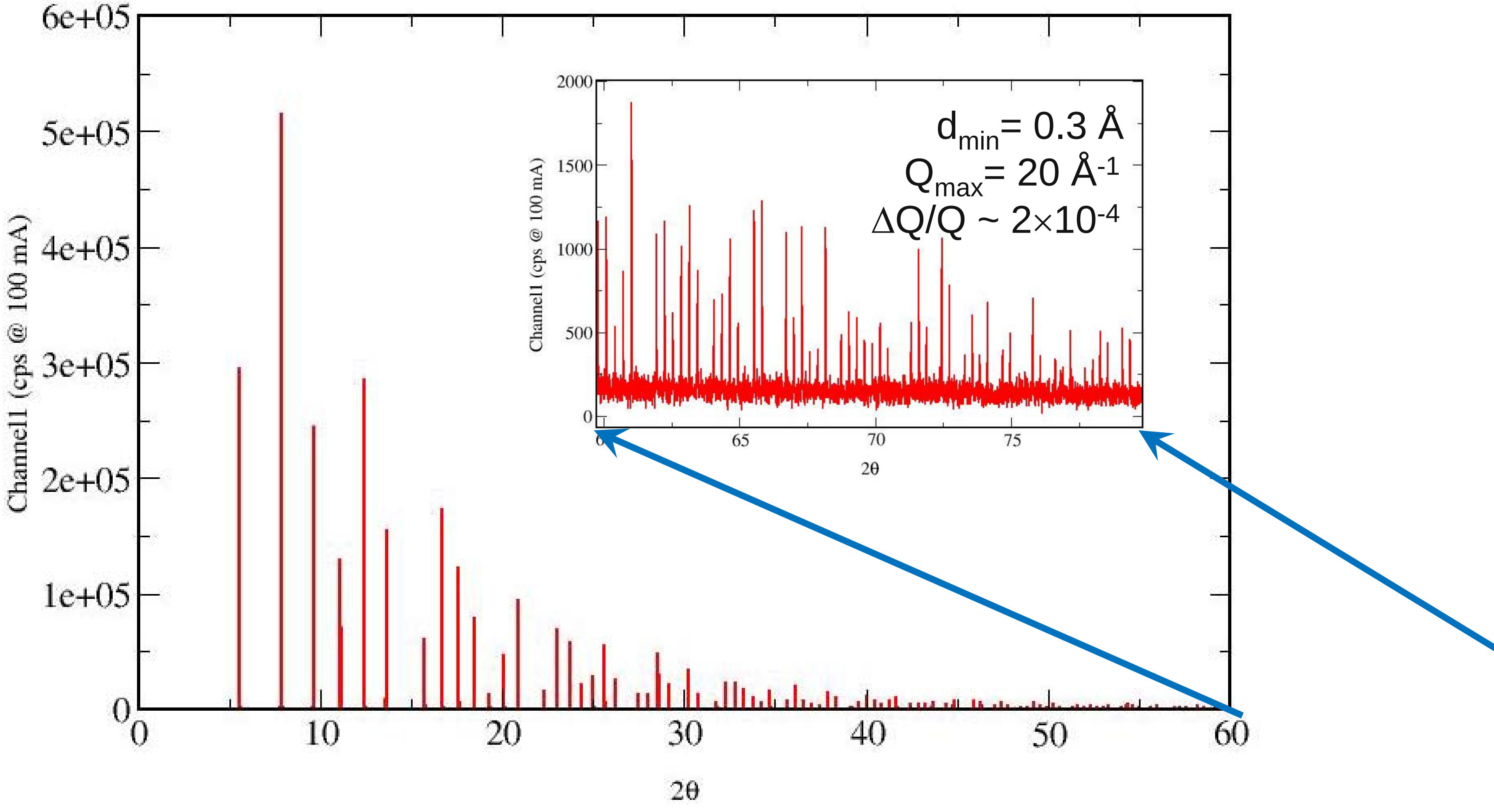
High-Energy diffraction provides more accurate data and provides a wider Q range energy (more observations)

High-Throughput diffraction allows these capabilities to be made available to the appropriate research communities in chemistry, materials, condensed matter physics, geosciences, pharmaceutical science, structural biology…

Users want rapid access and they do not want to travel to the APS (mail-in) for routine types of measurements.

11-BM: Exquisite data for the most complex problems

NIST SRM 660a (LaB_6) @30 keV (0.4Å)



11-BM Project history & design specifications

History of APS 11-BM Project

DOE grant proposal (2003) by ***J.F. Mitchell, J.D. Jorgensen, R.B. Von Dreele, P.L. Lee, M.A. Beno:***

Build a dedicated high-resolution powder diffraction beamline at APS => 11-BM (bending magnet)

Goals:

- World-class instrument with state-of-the-art powder diffraction capability to further the rapid growth of that user community at the APS.

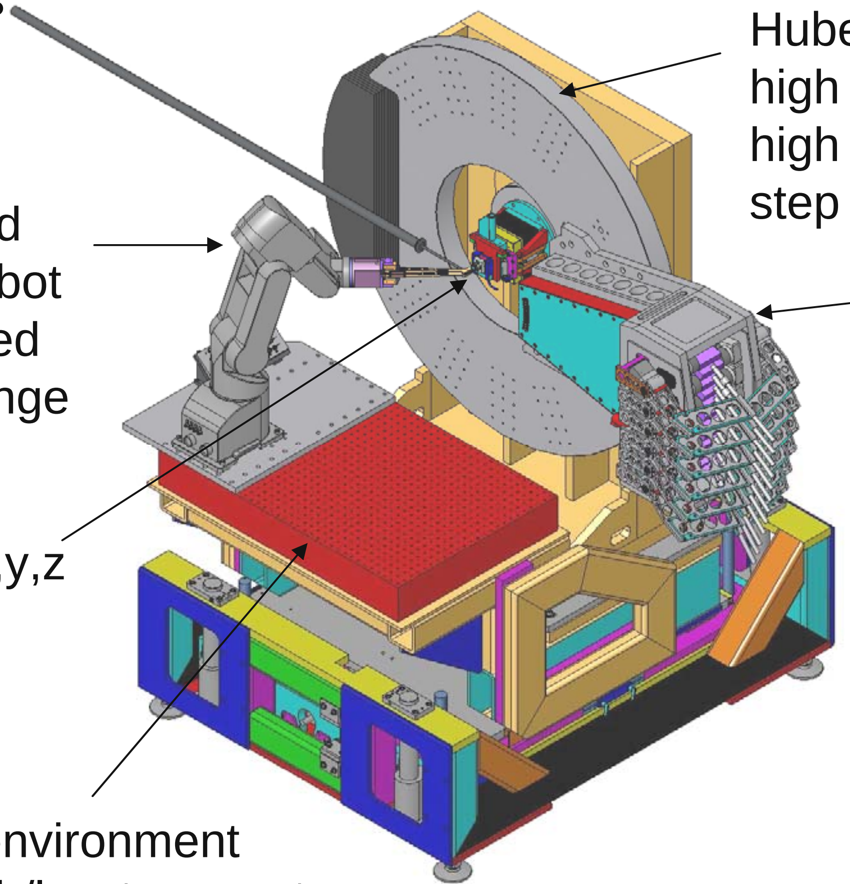
- User-friendly, high-resolution, high-throughput instrument for leading structural science in fields ranging from condensed matter physics and materials chemistry to pharmaceutical and biological sciences.

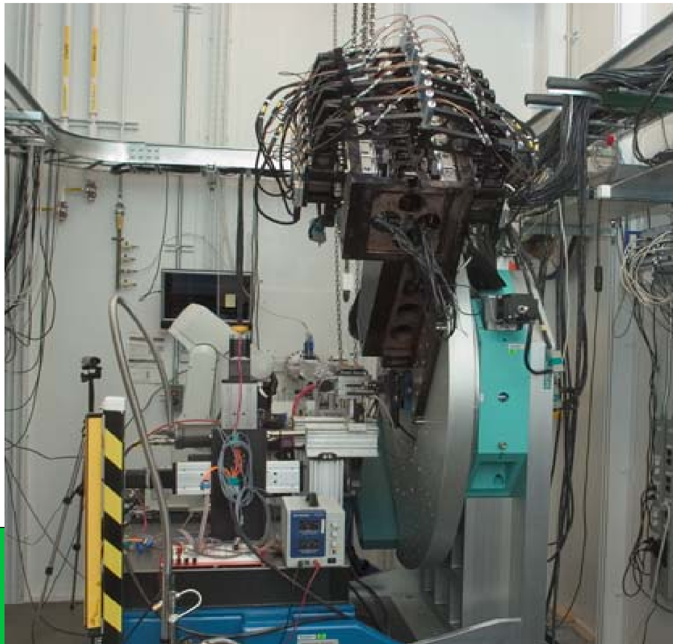
11-BM Proposed & Implemented Technical Specs

green: as initially deployed
red: differed for lack of funds/staff

- Energy range – 5.5-39keV (2.5 – 0.3Å)
    - Current operation: 30 keV (0.3Å) fixed
- Energy resolution – $\Delta E/E \sim 10^{-4}$
- Diffraction resolution
    - high resolution ($\Delta d/d \sim 2 \times 10^{-4}$),
      <1 hr scan length
    - Image plate/CCD measurements ($\Delta d/d \sim 10^{-3}$)
      "scan time" of a few minutes or less
    - Current operation: high res. only
- Robotic sample change for automation and high throughput
- Parametric experiment control for T (4-1500K), pO_2 or other gas (vacuum – 10 atm)
    - Current operation: 100 K & 300 K only

High resolution powder diffractometer with 12 channel analyzer system





12 Analyzer/Detector System

Deming Shu & Curt Preissner

⇒ 12 analyzers with independent motion controls for θ, χ

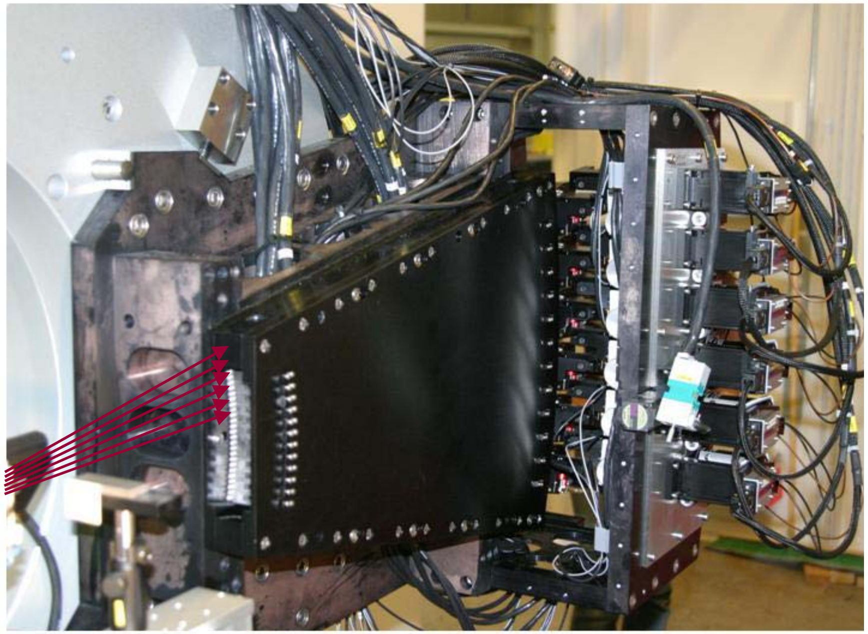
⇒ Wide θ angle range (0-24.5^{o}) for each analyzer to cover the energy range

⇒ Additional piezo control for fine adjustment

⇒ Sufficient χ adjustment without compromising stability of the analyzer

⇒ All analyzers have to fit into a confined space (2^{o} apart)

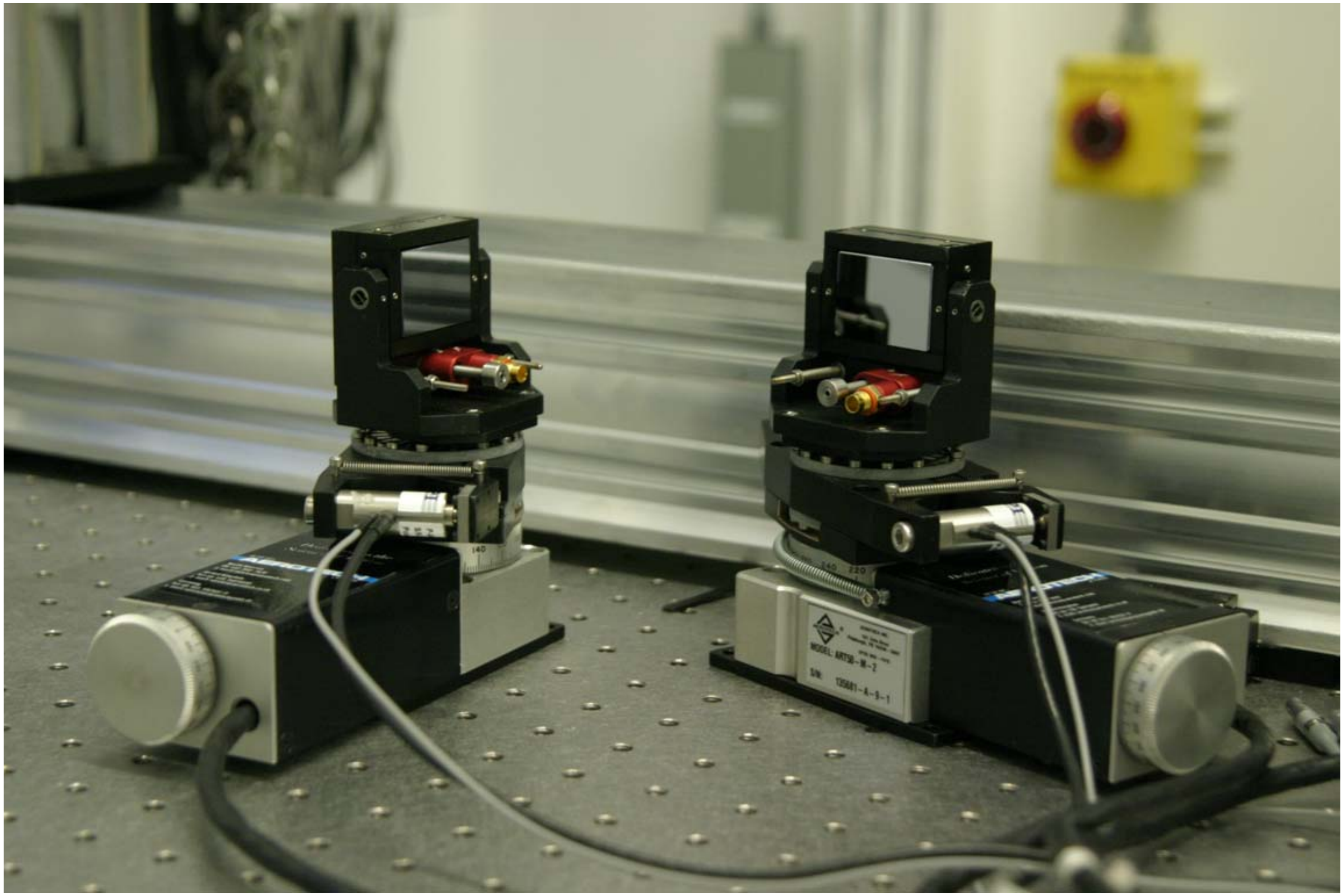
⇒ All analyzers are rigid and stable with respect to analyzer orientation during 2θ scan



- Greatly reduce the data collecting time
- Improve data accuracy by increasing the data redundancy
- Facilitate time-resolved experiment at high resolution

12 Analyzer/Detector System:

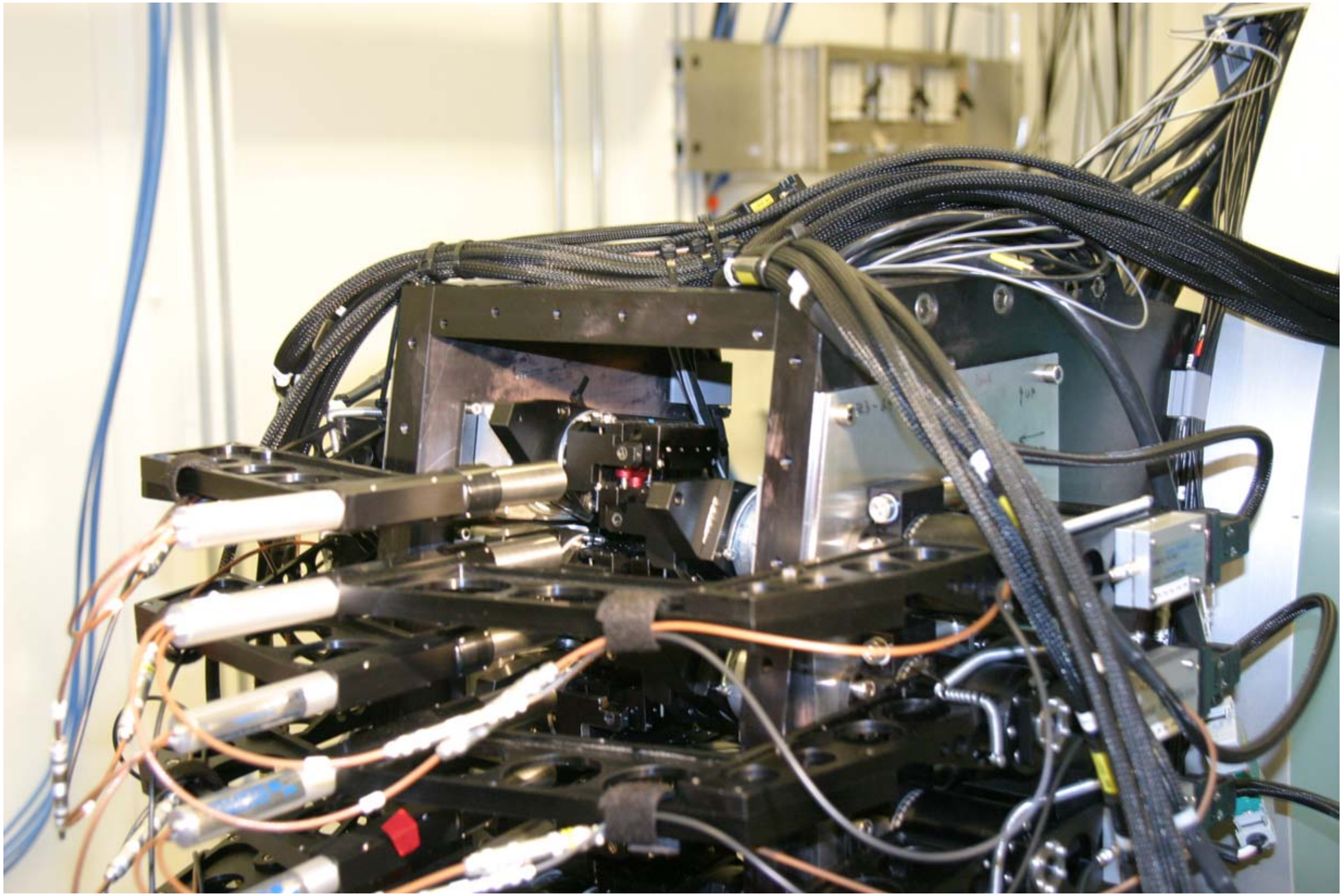
Two-axis positioning analyzers with weak-link fine ω adjustment



Deming Shu & Curt Preissner

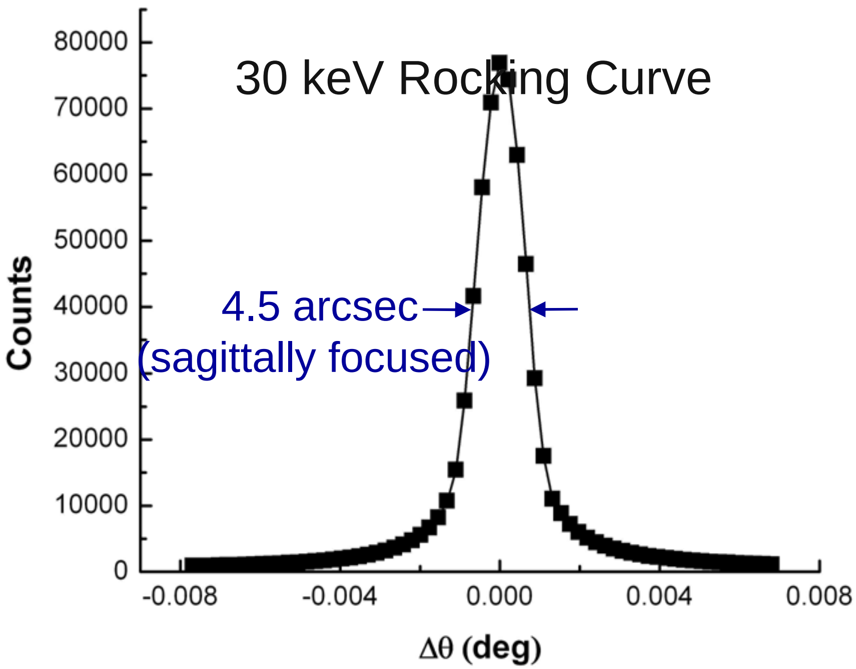
12 Analyzer/Detector System

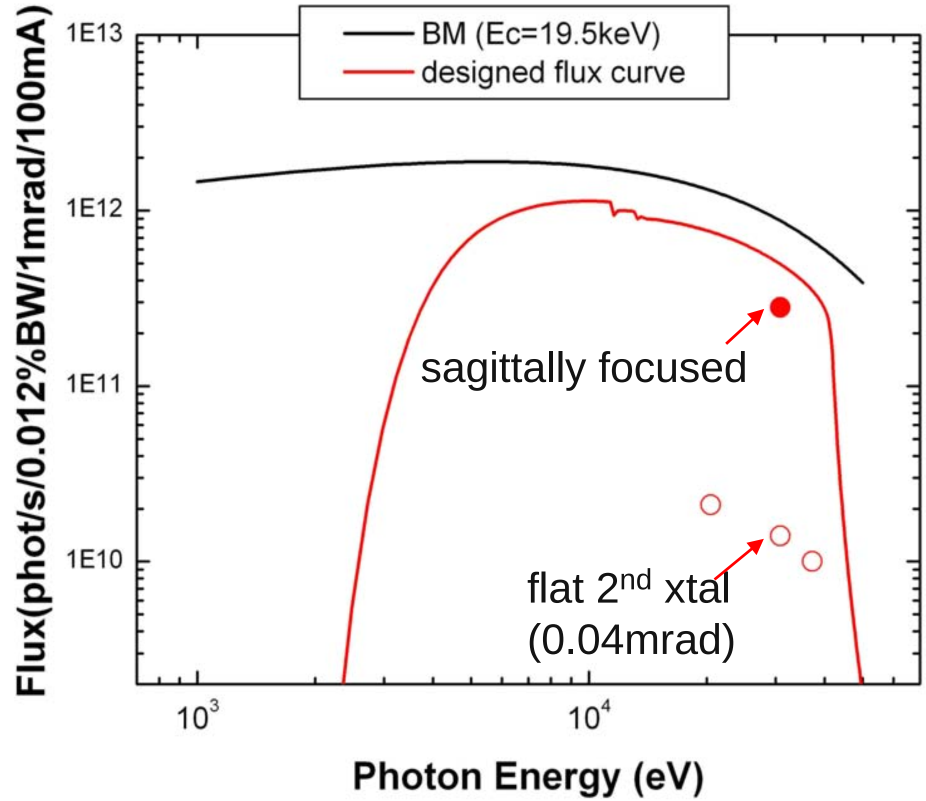
As assembled



11-BM Actual performance

Optics: Actual versus Theoretical Performance





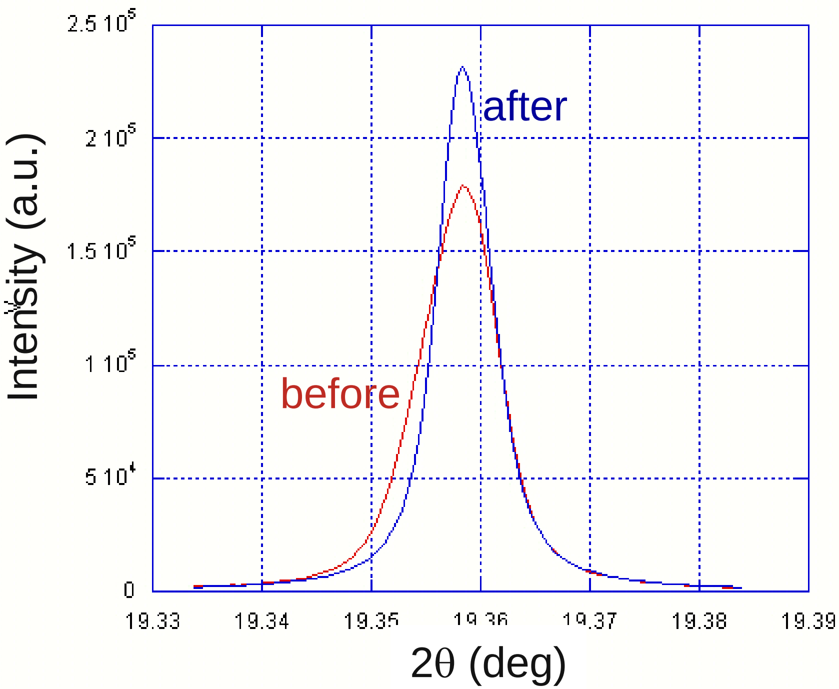
- Theory: Flat Si(111): 2.7 arcsec
- Actual: 4.5 arcsec *with* sagittal focusing (excellent!)

- Measured x-ray flux: 2.8×10^{11} phs/sec @ 30keV (within factor of 2 of ideal: excellent!)
- Doubly focused beam size: 0.35mm (H) x 0.2mm (V) FWHM

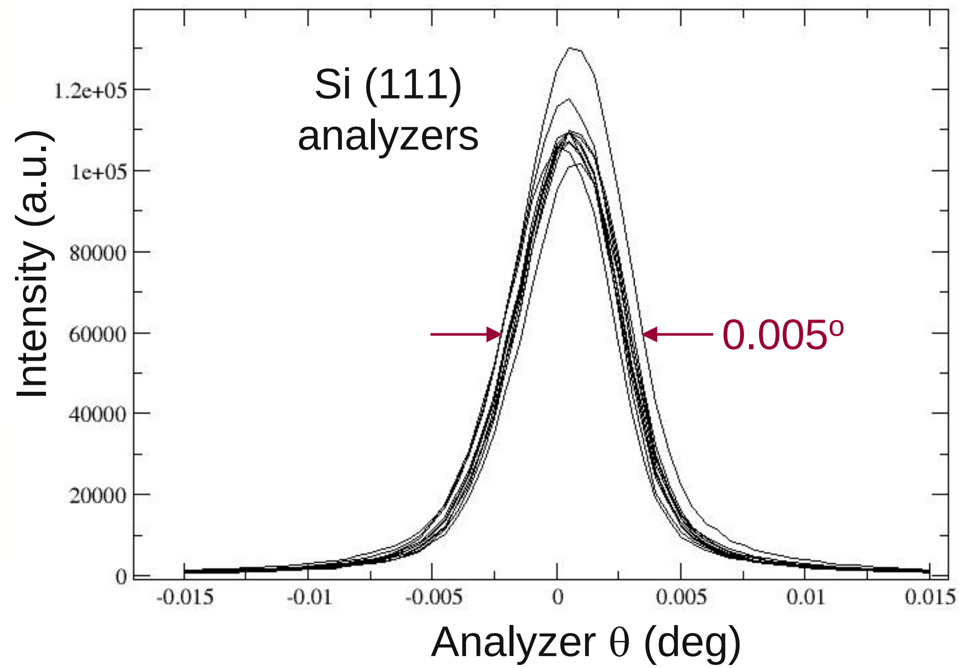
Analyzer System: 2 axis alignment is critical for both uniform and optimum peak shapes

- As seen at COMCAT, optical alignment of crystals is not sufficient

- After two-axis alignment, all 12 detectors provide equivalent resolution with symmetric peaks



Si (111) powder peak before and after χ adjustment

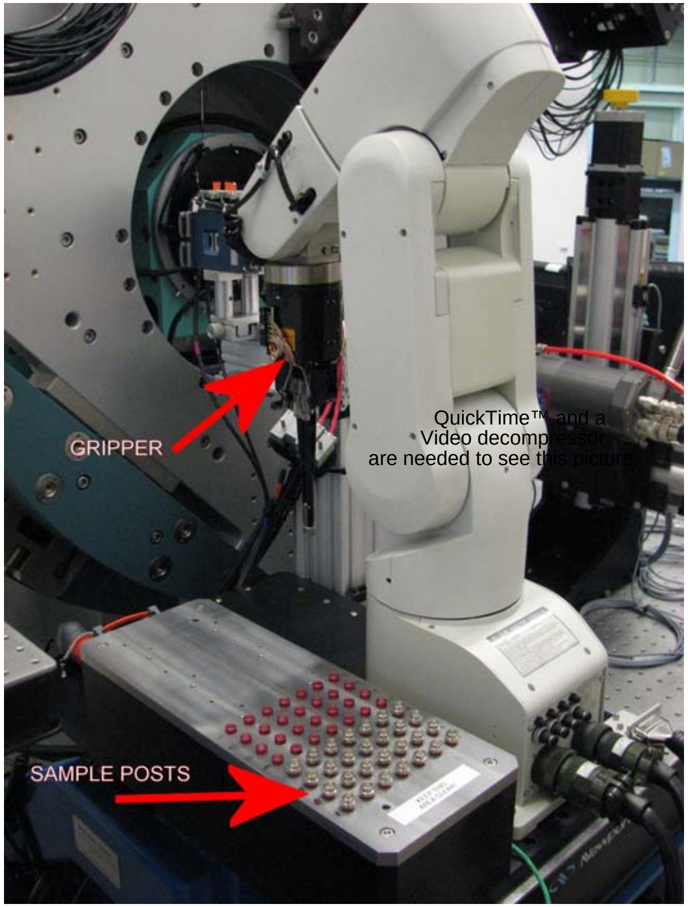


12 analyzer rocking curves at $2\theta = 0$ with direct beam

Accommodating high throughput at 11-BM

Robotic sample changer

Curt Preissner & David Kline



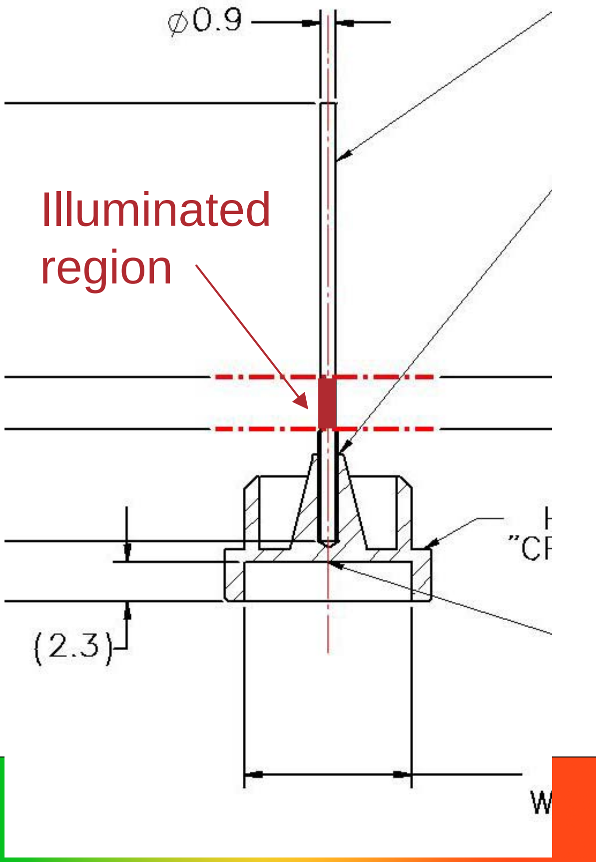
- ⇒ Capacity: 100+ samples
- ⇒ Small footprint and easy sample access
- ⇒ Safe operation: inhibit when hutch open
- ⇒ Integrated into EPICS

11-BM User program

- Until staffing grows, 11-BM will concentrate on a single mission (at present):
  - mail-in measurements
  - 100 K or ambient data collection temperature
  - 1 hour scans, fixed data range
  - Standard sample mounts (supplied by APS) & standard positioning (no alignment)

- As staffing, experience & demand grows we expect to add more data collection options (protocols for beam-sensitive samples…, more temperatures, user-selected data collection parameters, in-house users, area detection…)

- Beamline Advisory Group: 1 shift (typically 8 samples) or less: rapid access



Mail-in data collection requires high-levels of automation

Tasks to be done for each sample/experiment:

- Data entry
- Safety forms
- Loading/running instrument
- Data reduction
- Validate data
- Getting data to users
- Sample receipt, storage & return/disposal
- Tracking instrument use

All of the above must be completed in less than 15 minutes per sample (total!) to be completed by 1 FTE

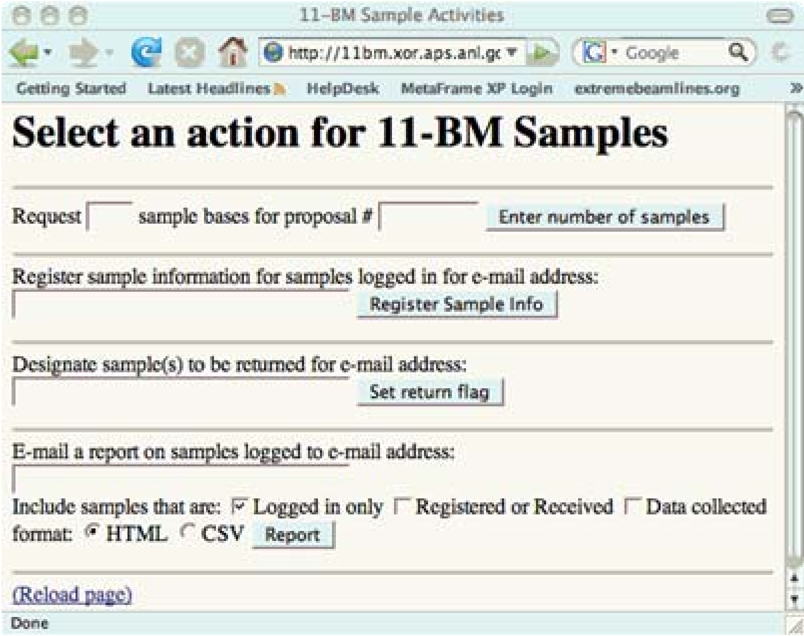
(24 samples/day @ 6 days/week * 15 min = 36 hours per week)

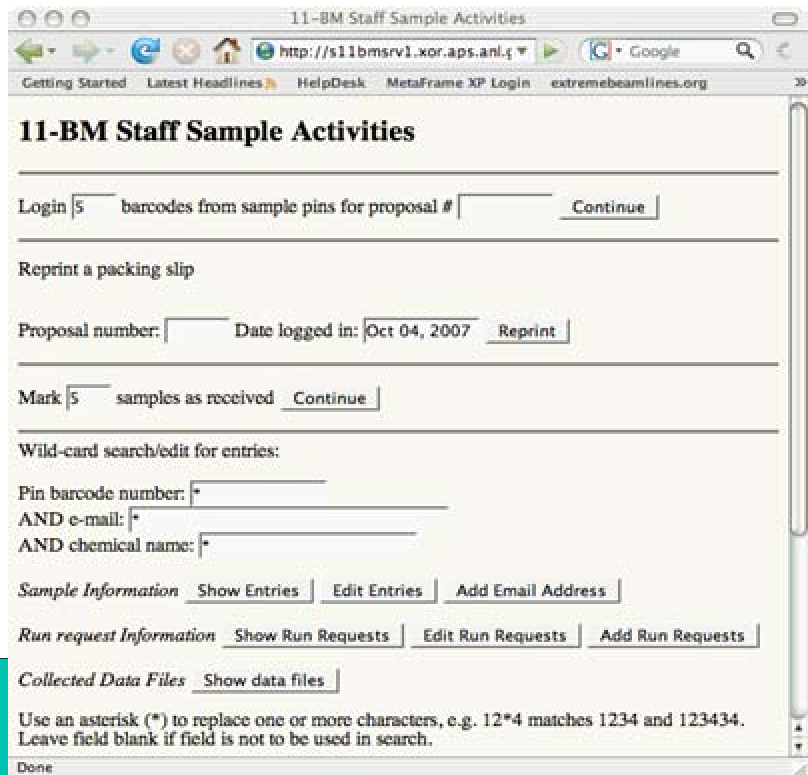
Automated Sample Handling

12 steps from reviewing proposals to tracking user's publications
http://11bm.xor.aps.anl.gov

1. General User Proposal submitted by user.
2. APS or BAC review; accepted to beamline; user prompted to specify # of samples
3. Staff logs sample bases to e-mail address & GUP #; sends bases to user
4. User enters sample description & hazard info for samples
5. ESAF (safety form) automatically generated from sample info
6. User ships samples to APS
7. Staff receive samples, scan bar codes, *store by hazard category*
8. Samples loaded on diffractometer; data collected if ESAF approved
9. Calibrate against standard, *screen data for glitches. Reduce & e-mail data (+ calibrated wavelength)*
10. *Store samples sorted by hazard category*
11. *Dispose of samples: segregate & catalog by disposal class*
12. *Automated nagging of users for publications*

(green: not yet implemented, cyan: in progress)





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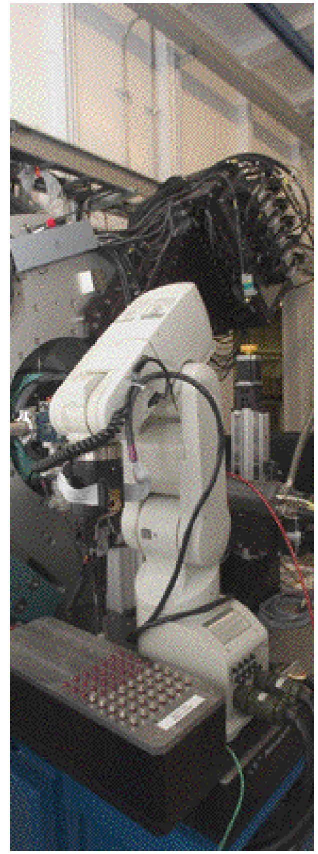
The Robot/Instrument/Database Automation 12-step

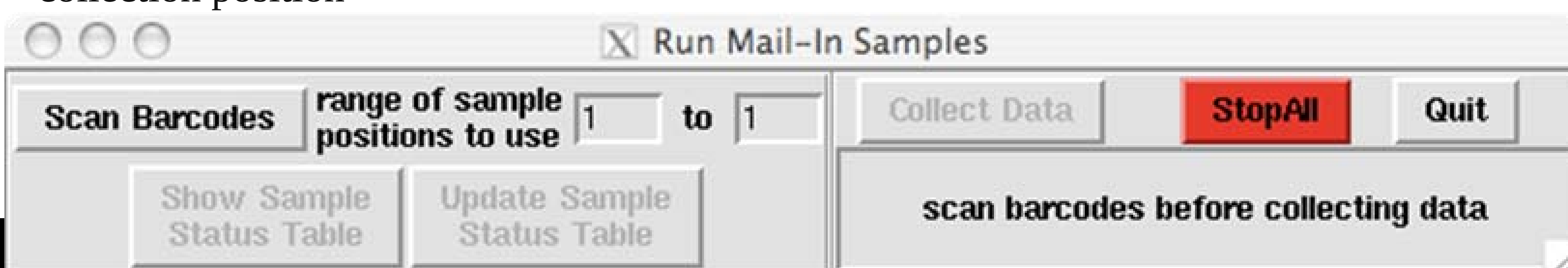
0. Read barcodes for all samples; warn on samples that can't be run

Loop:

1. Beam is blocked with an absorber
2. Diffractometer moved to the scan start
3. The cryostream is moved out of the cooling position
4. Sample stage is translated to robot home; spinner is stopped, if running
5. Previously loaded sample is removed from the diffractometer, if needed
6. Sample is loaded on the diffractometer, confirming the barcode matches previous
7. The sample spinner is started and the translation is changed to the data collection position



8. The cryostream device is returned to the cooling position with a short delay to reach T
9. Confirmed that the beam has been up for a minimum period to ensure thermal stability of the beamline optics, if not a delay is initiated
10. The absorber is removed and data collection is initiated
11. Confirm there has been no beam dump
12. Queue updates to the Run Data & Run Request database entries; queue data reduction request



Acknowledgements

- Lynn Ribaud, Jennifer Doebbler, Bob Von Dreele (**Jun Wang** & Sytle Antao) (11-BM staff)
- Mark Beno, Peter Lee, Mohan Ramanathan & Chuck Kurtz for too much to list
- Deming Shu, Curt Preissner, David Kline (multi-analyzer/detector and robotics)
- Mark Engbretson (EPS)
- Xuesong Jiao & Tim Mooney (Instrument control)
- Bill Sheehan & Dave Cyl (IT)
- Don Dohan (databases)
- Yu Huang (ESAF & GUP interactions)
- David Carroll (DB/Web programming)
- John Mitchell for launching the project
  - Ray Orbach for listening to him

QuickTime™ and a
TIFF (LZW) decompressor
are needed to see this picture.

11-BM staff, engineer & BCDA programmer, now safe