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Ready or Not: The Transition to Mail-in Synchrotron Powder Diffraction

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Contributors

- Don Dohan (databases)
- Yu Huang (ESAF & GUP interactions)

- Xuesong Jiao & Tim Mooney (Instrument automation)
- Curt Preissner & David Kline (Robotics)

- Jun Wang, Sytle Antao & Lynn Ribaud (critiques & testing)

- Mark Beno, Peter Lee & Mohan Ramanathan for too much to list

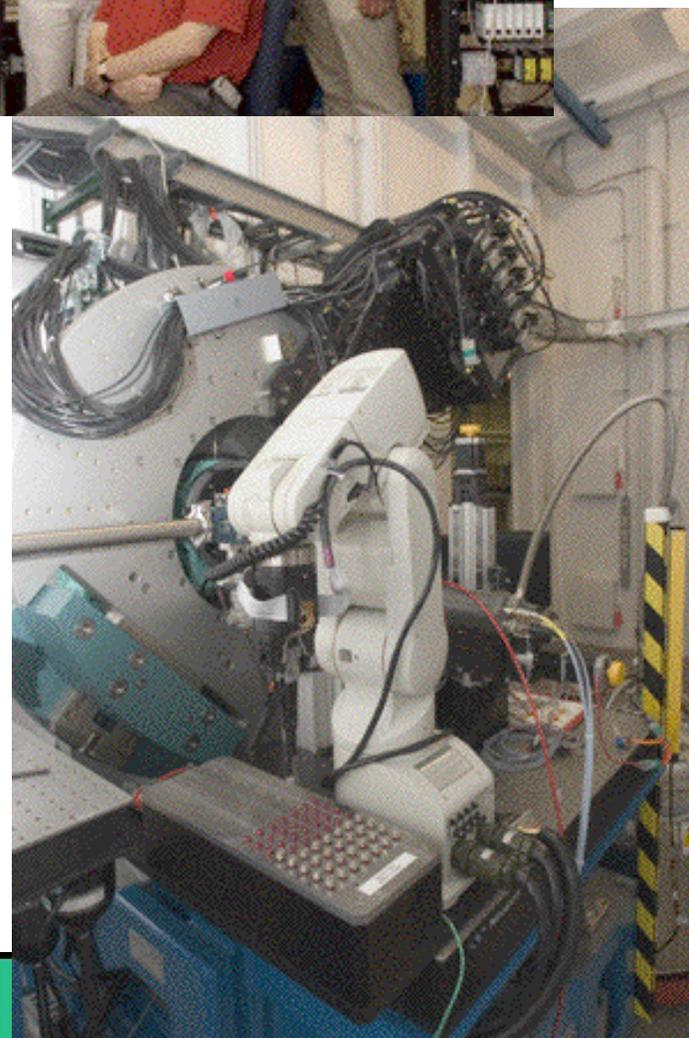
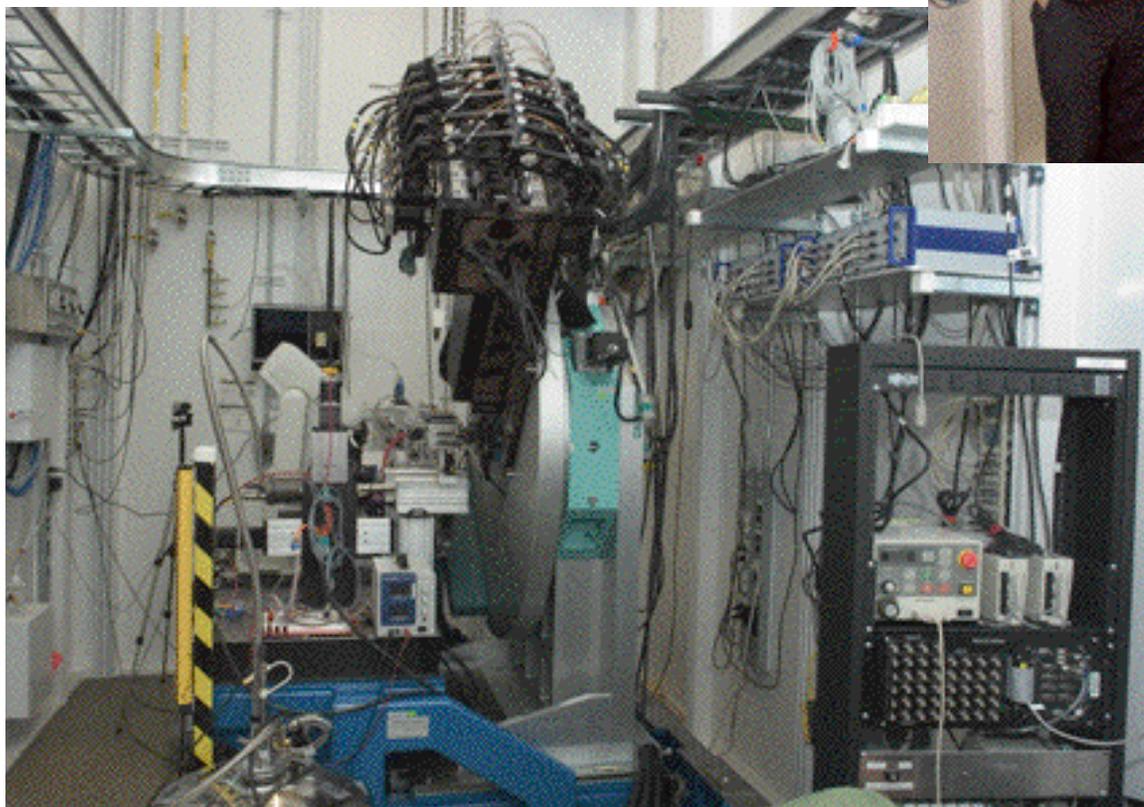
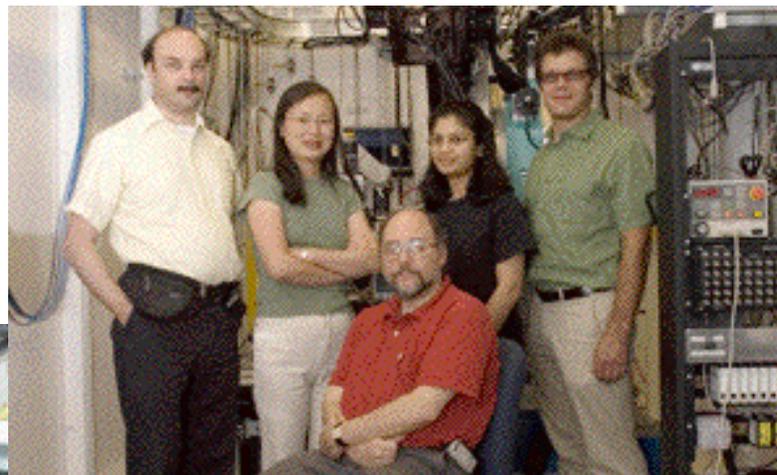
- John Mitchell & Ray Orbach for \$\$\$ (Helen Kerch for urgency)

Talk Outline

1. Update on 11-BM
2. Challenges of High-Throughput Diffraction
3. User needs
4. New Science

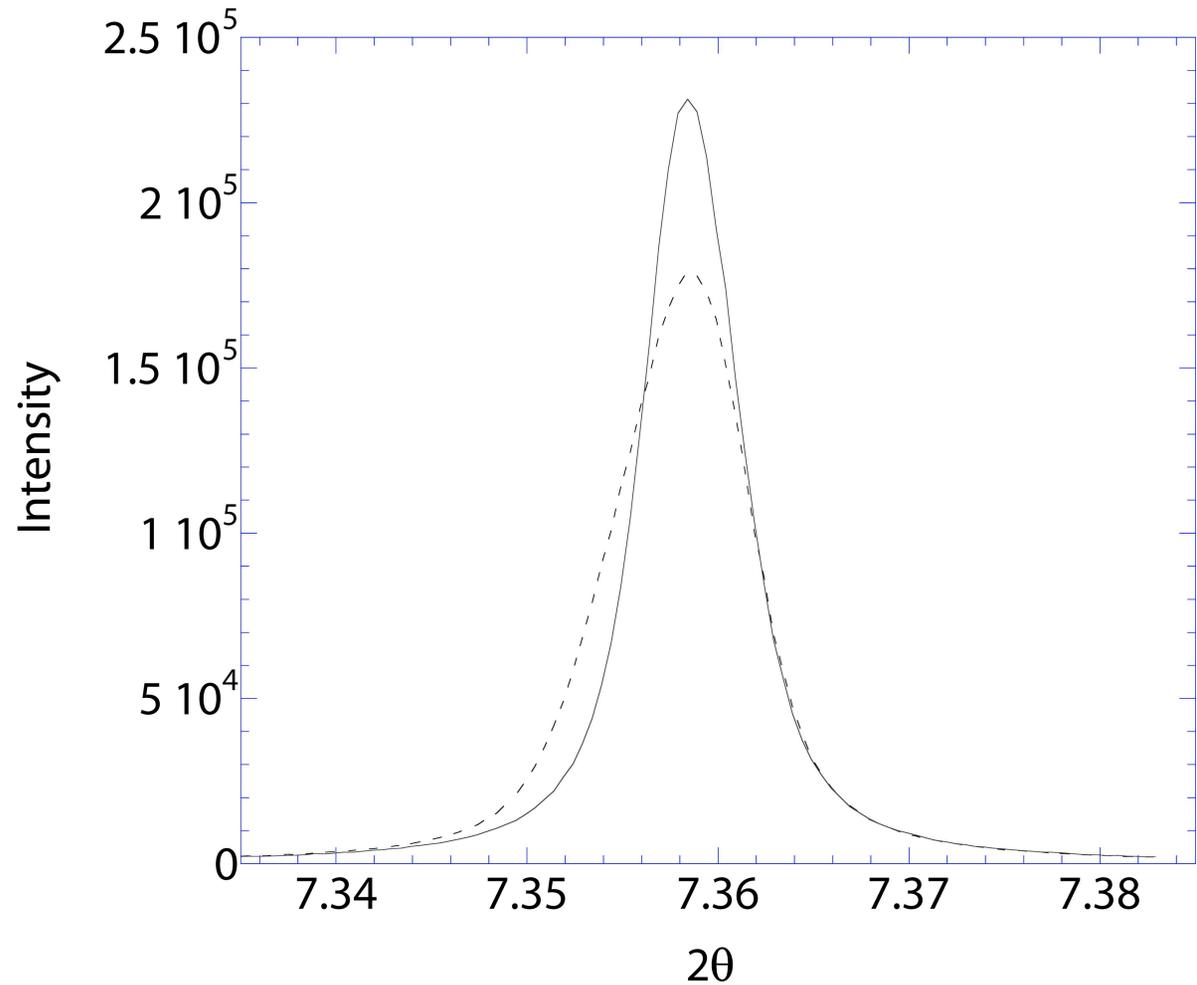
11-BM Status report

1) 11-BM update



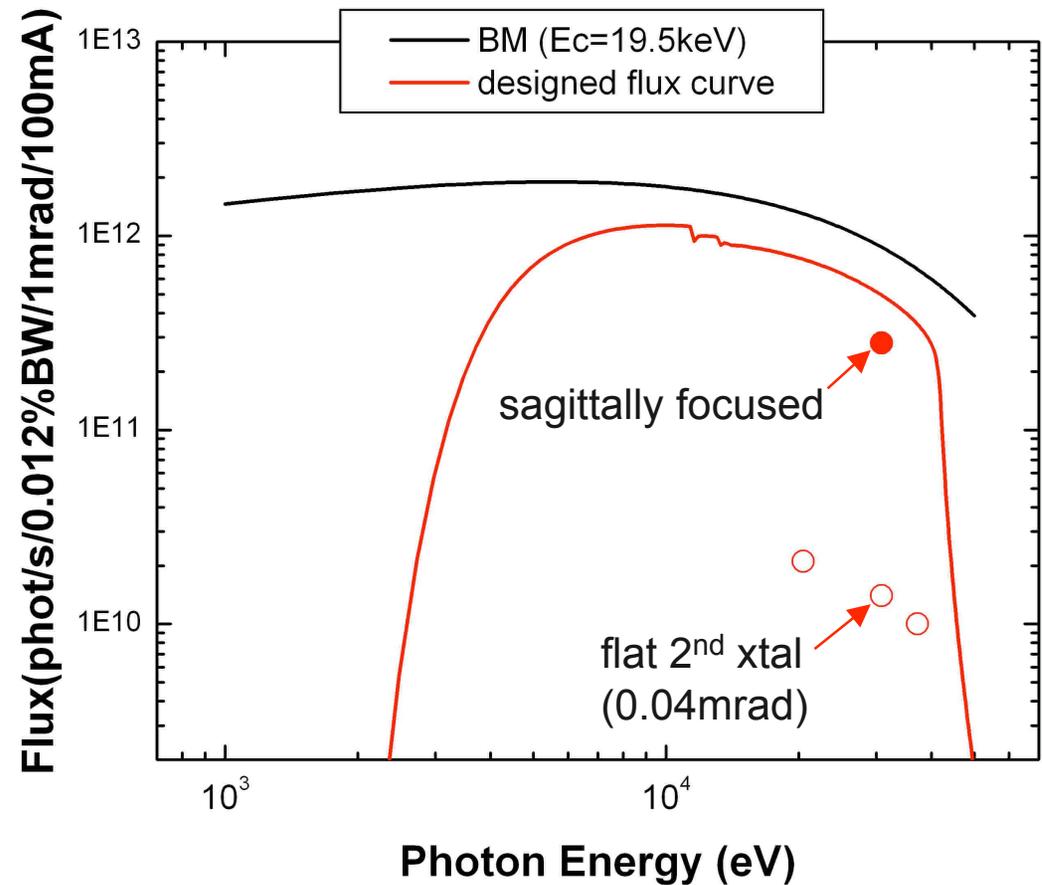
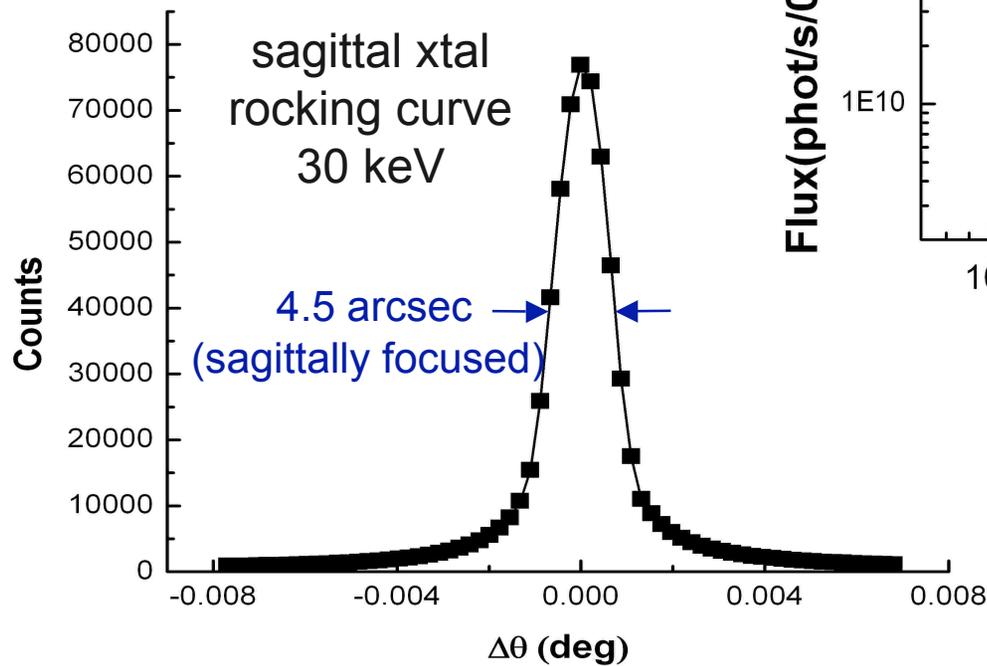
Optical alignment is not sufficient to align χ

Diffracted beam profile before (dash) and after (solid) in x-ray beam χ alignment



Incident x-ray flux & mono rocking curve

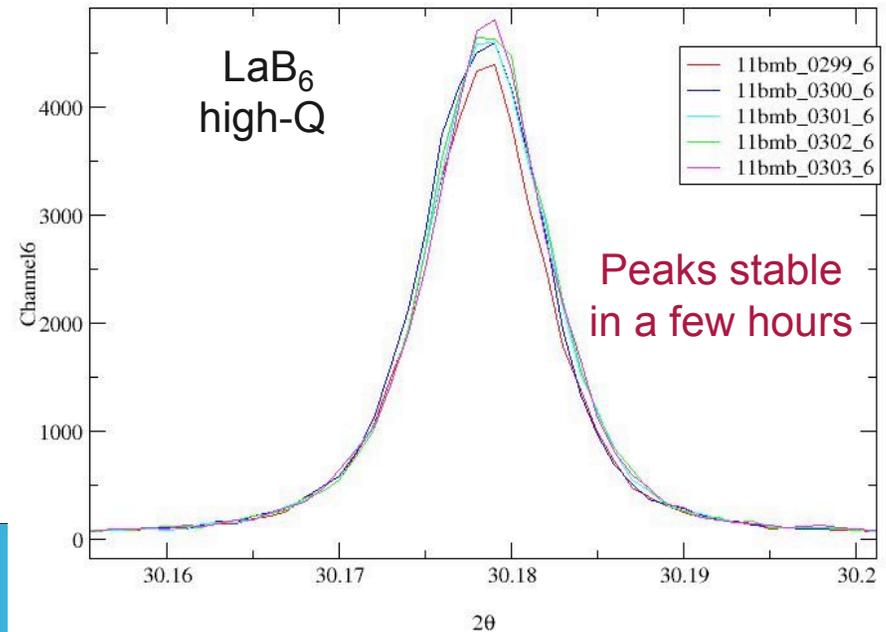
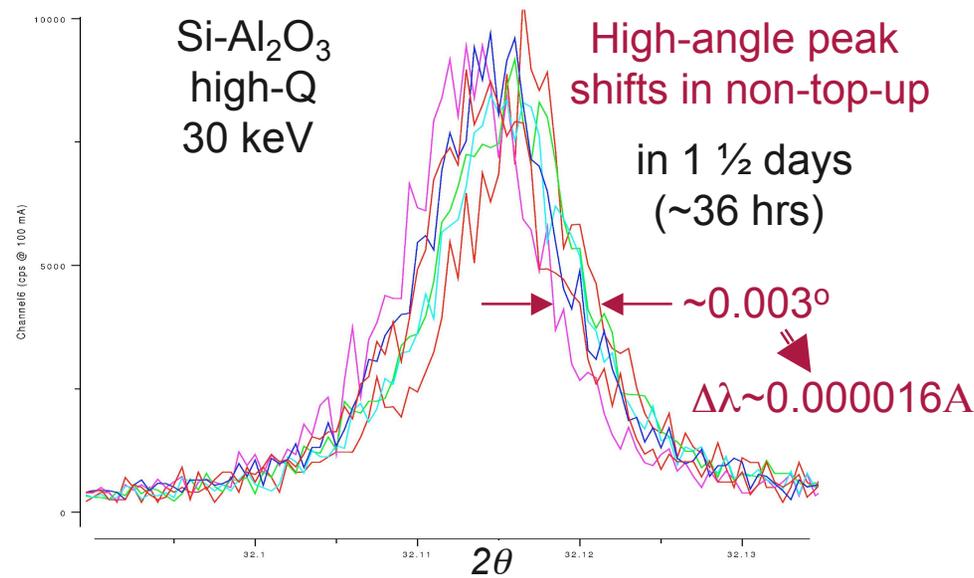
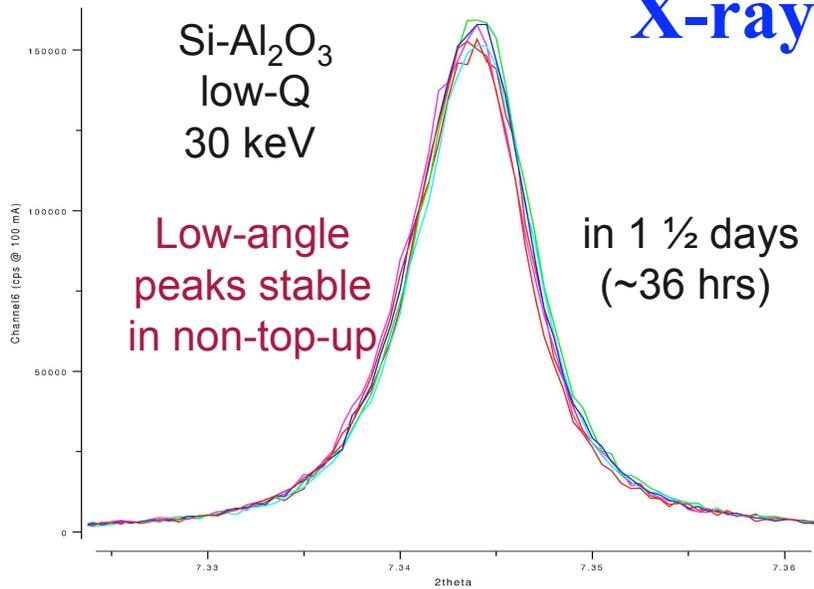
- Measured x-ray flux:
 2.8×10^{11} phs/sec @ 30keV
- Doubly focused beam:
0.35mm (H) x 0.2mm (V)



- Mono rocking curve @ 30keV:
measured FWHM = 4.5 arcsec
(when sagittally focused)
theory (flat xtal) \Rightarrow 2.7 arcsec.

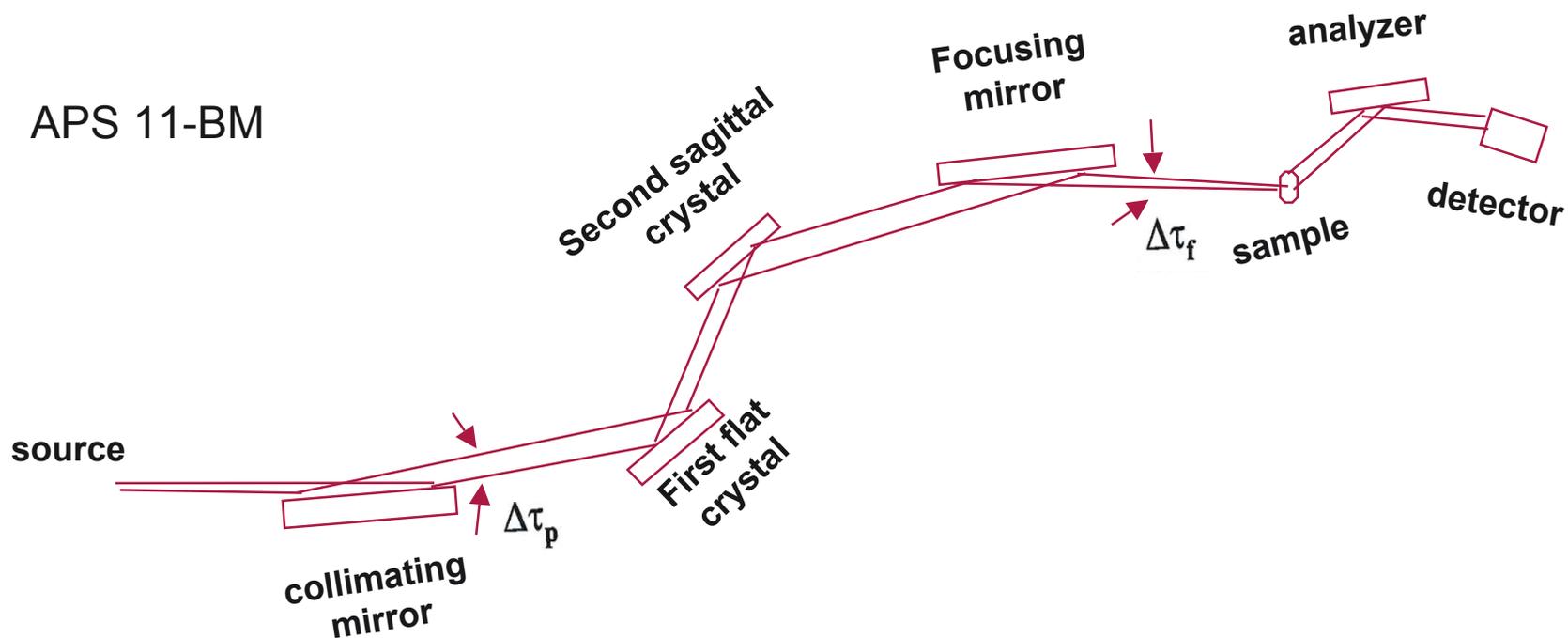
X-ray Beam and Instrument Stability

- Goal: long-term stability in automated operation without staff intervention.
- Preliminary measurements show slow drift in peak position over couple days. May be caused by wavelength drift on the order of 0.004% or beam angle drift $2.5\mu\text{rad}$ on mono.
- Slow drift can be completely corrected for by calibration using standards.



Powder diffraction instrument resolution function

APS 11-BM



$$\Delta^2(2\theta) = (\Delta\tau_p^2 + \Delta_m^2/2) \left(\frac{\tan\theta_a}{\tan\theta_m} - 2\frac{\tan\theta}{\tan\theta_m} \right)^2 + \Delta_a^2 + \Delta\tau_f^2$$

$\Delta\tau_p$: residual source divergence

Δ_m : monochromator crystal Darwin width

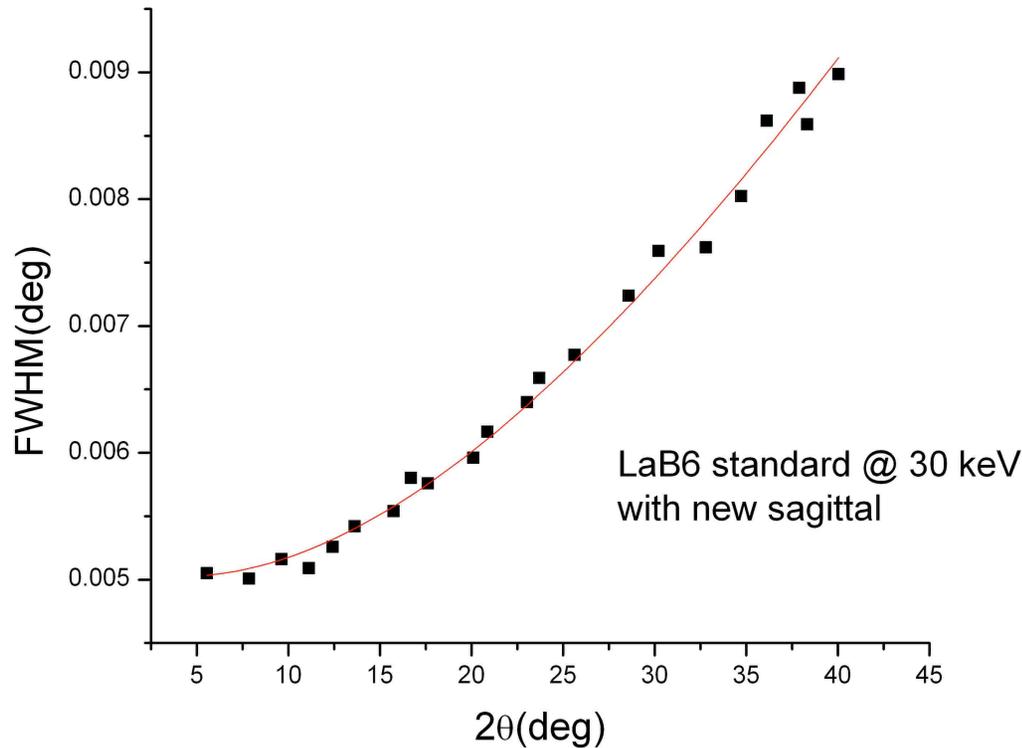
Δ_a : analyzer crystal Darwin width

$\Delta\tau_f$: focusing beam divergence

Reference: Gozzo et al., *J. Appl. Cryst.* 39, 347 (2006)

Resolution function with standard

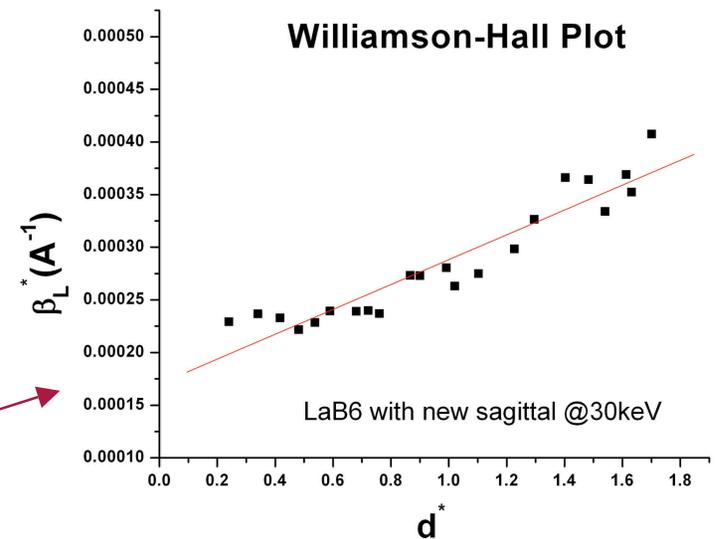
Instrument Resolution Function



Residual divergence: 13 urad
Focusing beam divergence: 90 urad
Particle size: 0.5 um

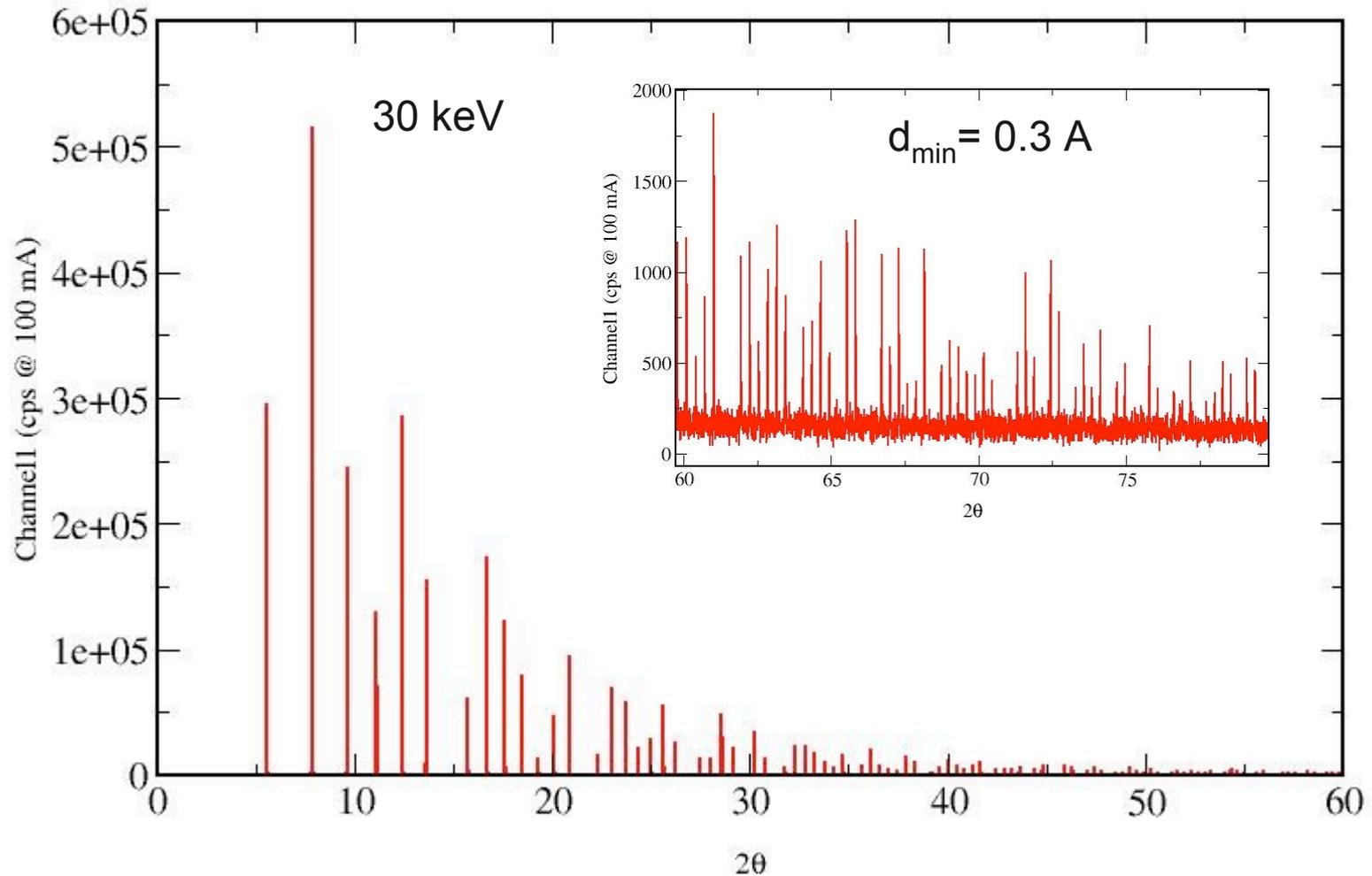
$$\Delta Q/Q = 2 \times 10^{-4} \text{ (on spec)}$$

particle size = 0.58um

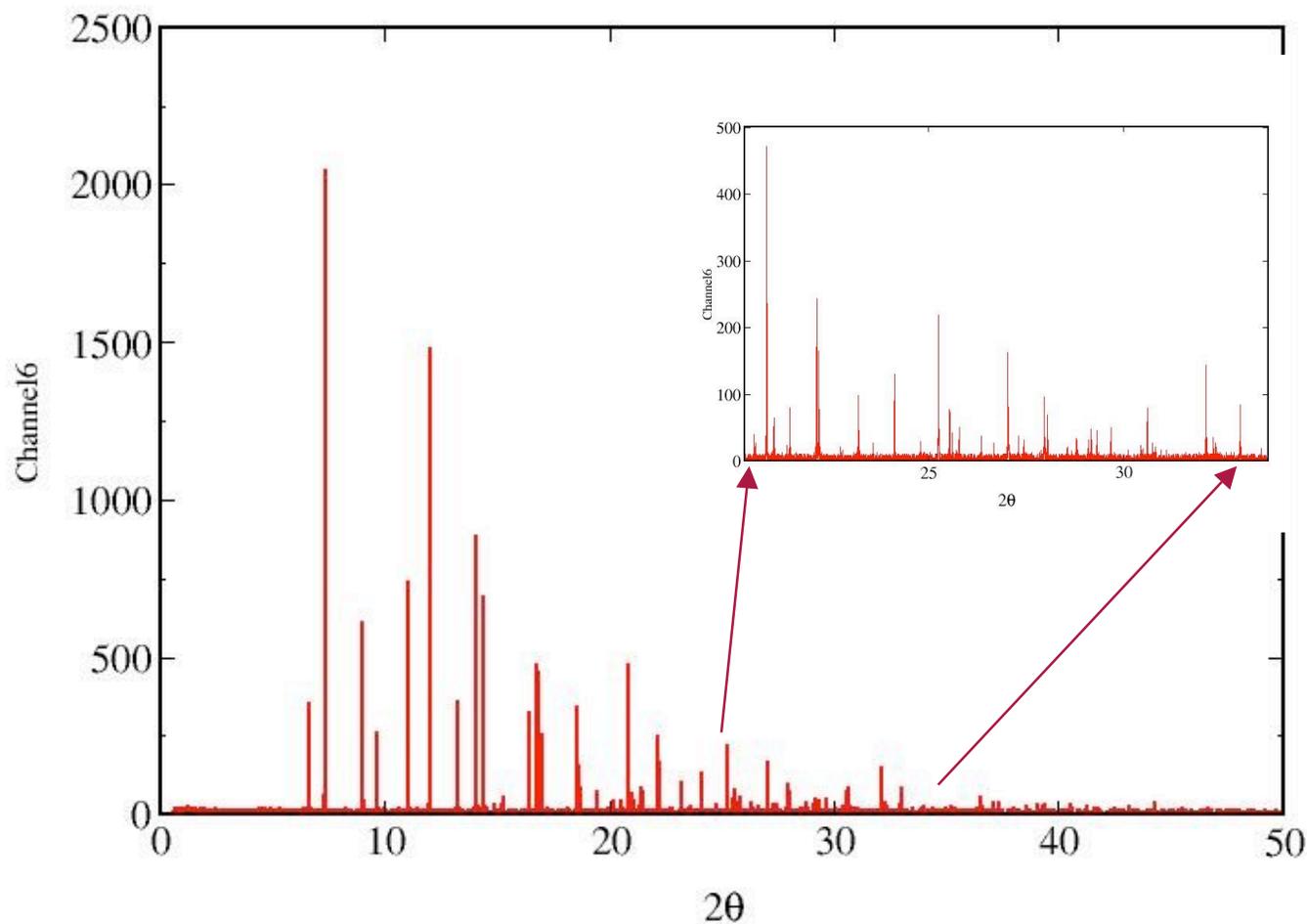


11-BM: Exquisite data for the most complex problems

NIST SRM 660a LaB_6



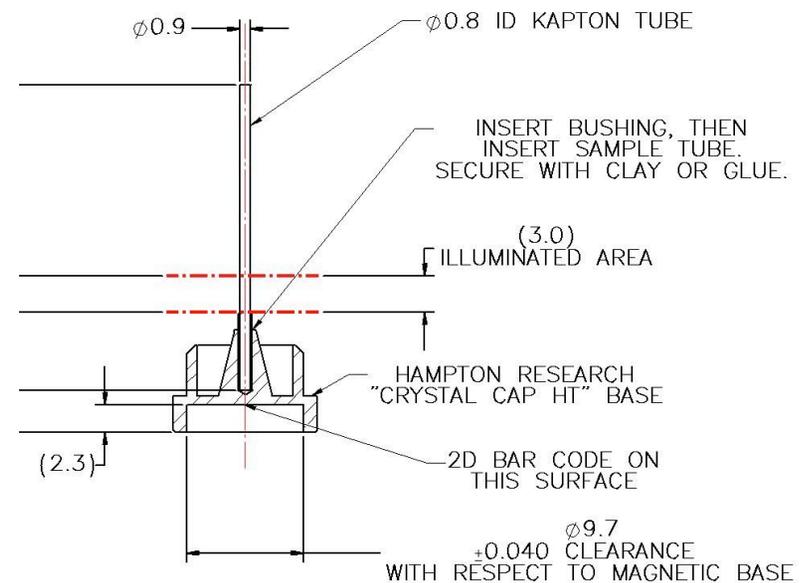
10 min data collection on NIST standard mixture Si + Al₂O₃



A full pattern with
ultra high
resolution
collected in only
10 minutes

Mail-in operation

- Run ~24 samples per day @ 6 days/week
- Stage 1 Operations for 11-BM:
 - Mail-in only
 - 100 K & RT only
 - fixed energy (30 keV) operation only
- All mail-in work through General User Proposal System
- Expect most beamtime requests to have few (1-10) samples
- Work needs to be supported by 1 Scientific Associate



Mail-in Powder Diffraction: Pros & Cons

■ Pro

- Need to open up user community to the widest possible range of scientists
- Modern high-throughput instruments are pushing the time per measurement way down
- Airports/airplanes seem less pleasant every year
- Grouping measurements for the next synchrotron trip limits productivity

■ Con

- On-site interaction has traditionally been where users have learned many aspects of data analysis
- We have always recruited beamline scientists from the experimental floor
- Staffing demands of a high throughput beamline are beyond the means of the DOE to fund

Like it or not, users are expecting mail-in access and it is our job to make it work

Challenges of High Throughput/Mail-in Work

Sample tracking: design principles

- Users do bulk of data entry
 - Document usage, materials & experiment safety info
- Force data entry on samples prior to shipment
 - Check if samples are entered before package is opened
- Preference: avoid passwords, but must protect information from snooping
- Tie sample to user (& optionally advisor) via e-mail address
- System extensible to track status of user analysis
 - Capture publication information

11-BM Work flow: the 11-BM 12 step procedure (Synchrotrons as an addiction?)

1. User enters rapid access General User Proposal (GUP)
2. Proposal is reviewed & accepted; 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
5. ESAF 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 iff 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. Nag users for publications

Complete process must be completed with less 15 minutes effort per sample total to require less than 1 FTE

Other, less routine, tasks in 11-BM workflow

By Users

- Obtain reports on samples in system with status
- *Request duplicate copy of data*
- Request that a sample be returned (rather than disposed)

By Staff

- Edit sample metadata
- Update e-mail addresses
- Change parameters for data collection for an individual sample
- *Report GUP usage stats*

The Robot/Data Collection 12-step

1. The beam is blocked with an absorber
2. The diffractometer is moved to the scan starting position
3. The cryostream is moved out of the sample position
4. The sample is translated to to the robot home position and the sample spinner is stopped, if running
5. Previously loaded sample is removed from the diffractometer, if needed
6. A sample is loaded on the diffractometer, confirming the barcode matches what is expected
7. The sample spinner is started and the translation is changed to the data collection position
8. The cryostream device is returned to the sample position and a short delay is initiated to allow the sample to cool
9. It is 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.



Educating our users

(If you build it, will they come?)

- Few universities are teaching practical crystallography; even fewer teach powder diffraction crystallography.
- With potentially 5+ users per *day*, one can not collaborate or even keep up with the e-mailed questions.
- Workshops are great: but need to be more comprehensive (longer) and need to be regional.
- Need outreach that works between workshops and reaches people who will not get to workshops.

Possible solutions

- Require the next generation of synchrotron users all be trained at Stony Brook
- Commission a modern rewrite of Klug & Alexander (Stephens, Cox & Parise?) that includes Rietveld analysis

Brian's goals:

- Develop a two-part instructional workshop. Part 1 should travel around the country; part 2 is best as hands-on.
- Develop web accessible instructional materials
- (Internal ~biweekly powder diffraction crystallography study group)

***Data analysis needs
(If they collect it, can they use it?)***

The next generation of data-fitting software

- Three data analysis codes (GSAS, Topas & FullProf). Two old, one commercial. All are basically single-person efforts.
- All are written by experts for use by experts
- Scope limited to diffraction

- GSAS-II workshop (May 2007). Report in 11/2007 IUCr Computing Comm. Newsletter (www.iucr.org/iucr-top/comm/ccom/newsletters/2007nov/)
 - Build a flexible & modern new code from ground up
 - Fit to all types of data that can be quantitatively predicted from an atomistic structural model
 - Keep GSAS alive until GSAS-II can replace it

- Hope for inclusion and funding as part of new Argonne Institute: ASI²

GSAS goes to Mars in 2009



Unsolved questions

- How well can non-diffraction data be fit with atomistic models?
- If most users never put hands on a synchrotron (or neutron) diffractometer, who will run them when we retire?
- What defines an experiment that should be hands-on vs. mail-in?

Why bother?

New Science!

Understanding the M1/M2 Propane Ammoxidation Catalyst

- Why: ammoxidation makes acrylonitrile
 - world use of acrylonitrile is 10^{10} lbs/year
 - Currently all is made from propene
 - Would rather use propane (much cheaper)
- Propane amoxidation catalyst has been known for >10 years
 - multi-phase mixture of two very complex layered (Mo,V,Te,Nb) oxides
 - Structure & mechanism a puzzle to academic & industrial groups.
- Buttrey *et al* breakthrough simultaneous use of:
 - Combinatorial synthesis: enhanced concentration of each phase.
 - TEM
 - X-ray powder diffraction (X7A)
 - Neutron powder diffraction (NIST)

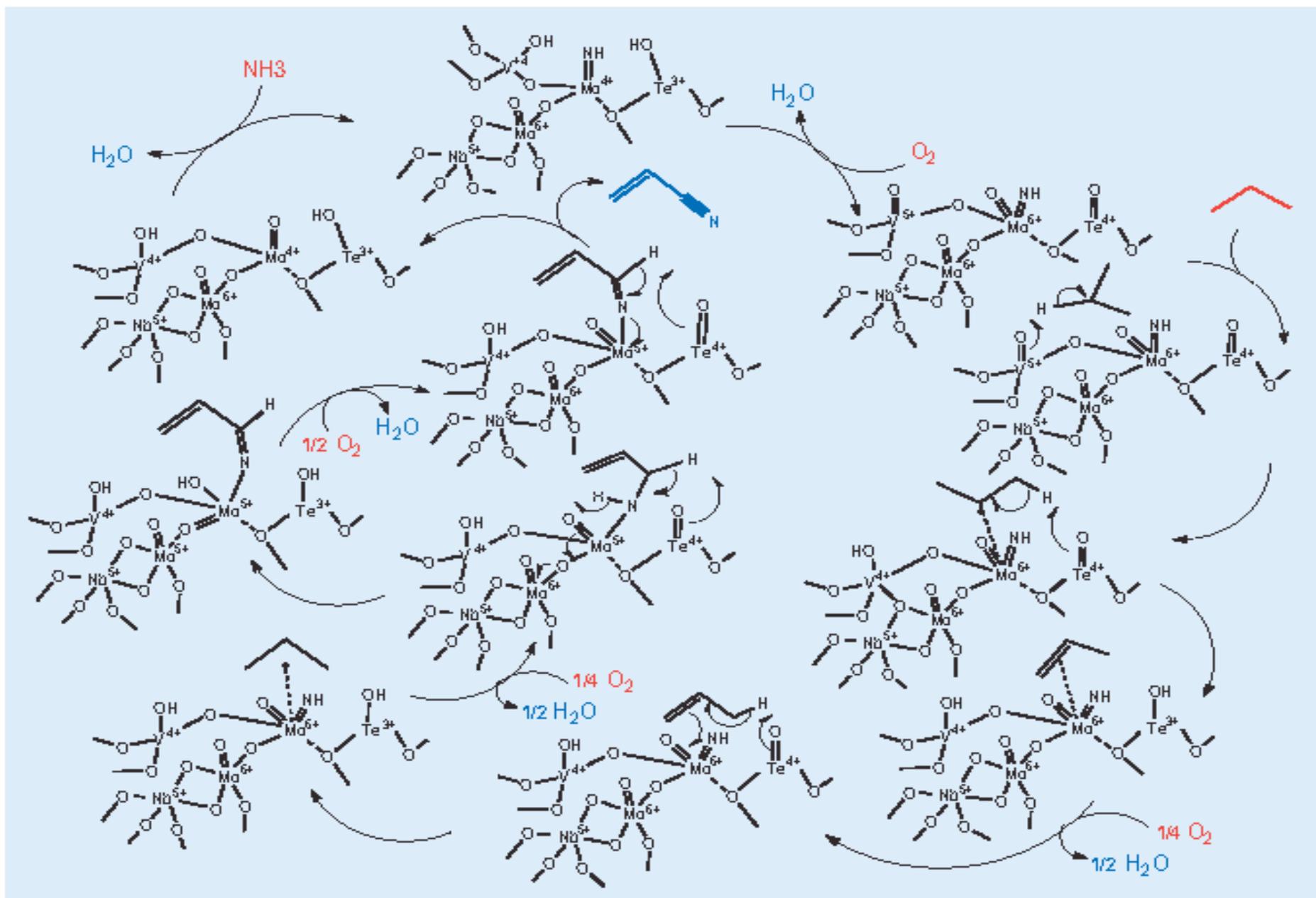


Fig. 2. A schematic mechanism for propane ammoxidation by the M1 catalyst that details the cation centers believed to be responsible for reactive process [3].

Take home messages

- We do powder diffraction crystallography because it allows us to learn unique information about real materials (perhaps in operating conditions) and understand their function and properties.
- Synchrotrons have incredible advantages over lab instruments
- Next-generation high-throughput synchrotron instruments can put this power into everyone's lab
- Considerable work-flow planning and automation is needed to allow these instruments to be utilized effectively
- Developments in software and instruction are needed to allow the data to be better exploited by non-experts
- Experiments at powder/single crystal boundary are also extremely important, but are even less accessible to general user population