

Synchrotron Powder Diffraction Simplified: Mail-In and In-Situ Data Collection at 11-BM

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11-BM Staff: Structural Science Group (XSD)

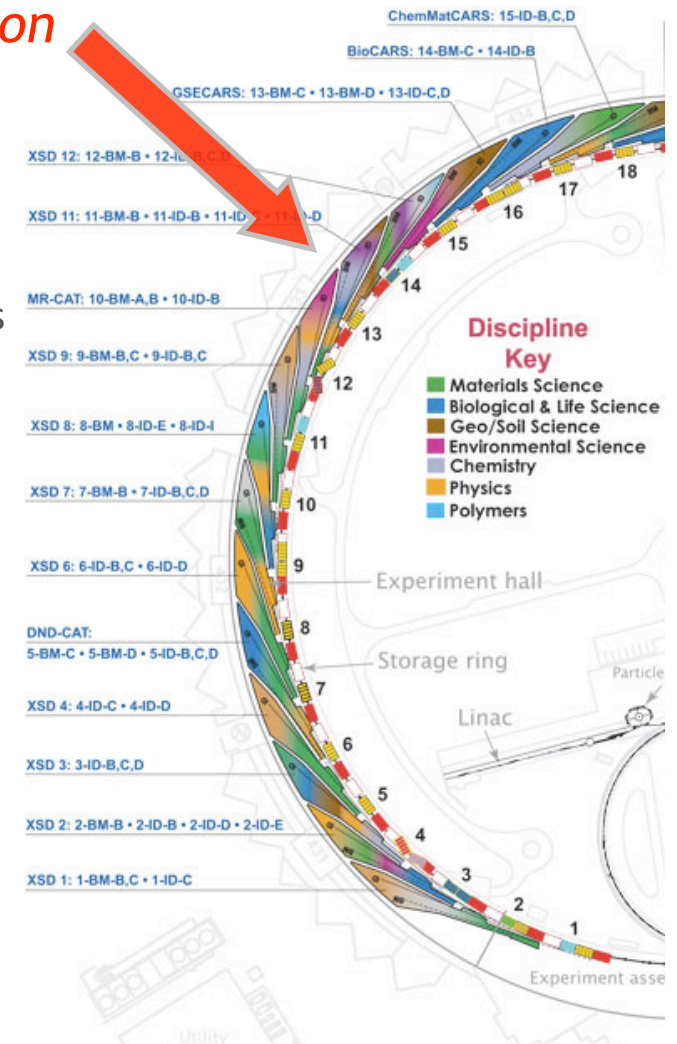
Brian Toby, and Robert Von Dreele

Distinguished Alumni: now Theory & Software Group (XSD)

APS Beamline 11-BM

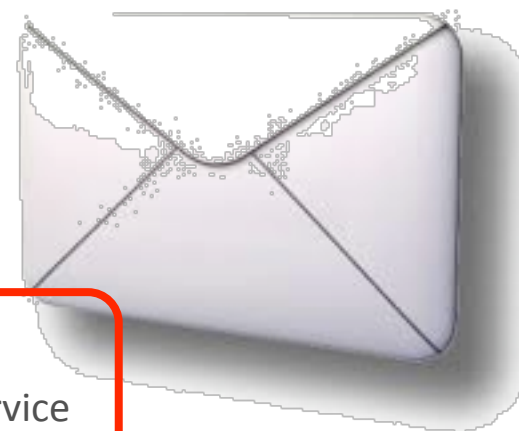
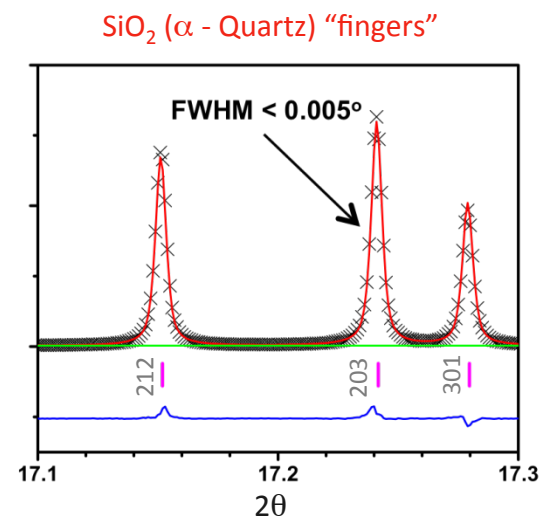
Dedicated to High Resolution Powder Diffraction

- Why Synchrotron Powder Diffraction ?
 - Established Technique, Widely Used & Understood
 - Offers Many Advantages Over Lab-Based Measurements
 - Provides Essential Structural Information for New Materials
 - In-Situ Probe: Real Materials - Real Time
- Beamline Mission
 - World-Class Performance, User-Friendly Operation
 - Rapid-Access Measurements via Mail-In Program (50%)
 - Support High Resolution On-Site Experiments (50%)
- Brief History
 - DOE Proposal: 2003
 - Begin Construction: 2005
 - First Beam in Hutch: 2007
 - Start Mail-In Program: 2008
 - Launch On-Site Experiments: 2010



11-BM Beamline Overview

- Energy Range (BM): 10 - 35 keV
 - mail-in service optimized @ 30 keV ($\sim 0.4 \text{ \AA}$)
- High Resolution: $\Delta d/d$ ($\Delta Q/Q$) $\approx 2 \times 10^{-4}$
 - Nearly Equivalent to Best-In-World (ESRF, Diamond, etc.)
 - Highest resolution powder diffraction in the Americas
- Beam Focusing & Flux
 - Sagittal Crystal & Mirrors \rightarrow 500 x 200 μm at sample
 - Typical BM Flux: 5×10^{11} phs/sec @ 30 keV
- Supported Sample Environments
 - Cryostream (90 K - 450 K), Hot Gas Blower ($< 1000 \text{ }^\circ\text{C}$)
 - Helium Cryostat ($> 5 \text{ K}$), In-Situ Pressure & Gas Cells
- Robotic Arm \rightarrow Automated Sample Loading
 - Robot + Database + Software \rightarrow High Throughput Mail-In Service



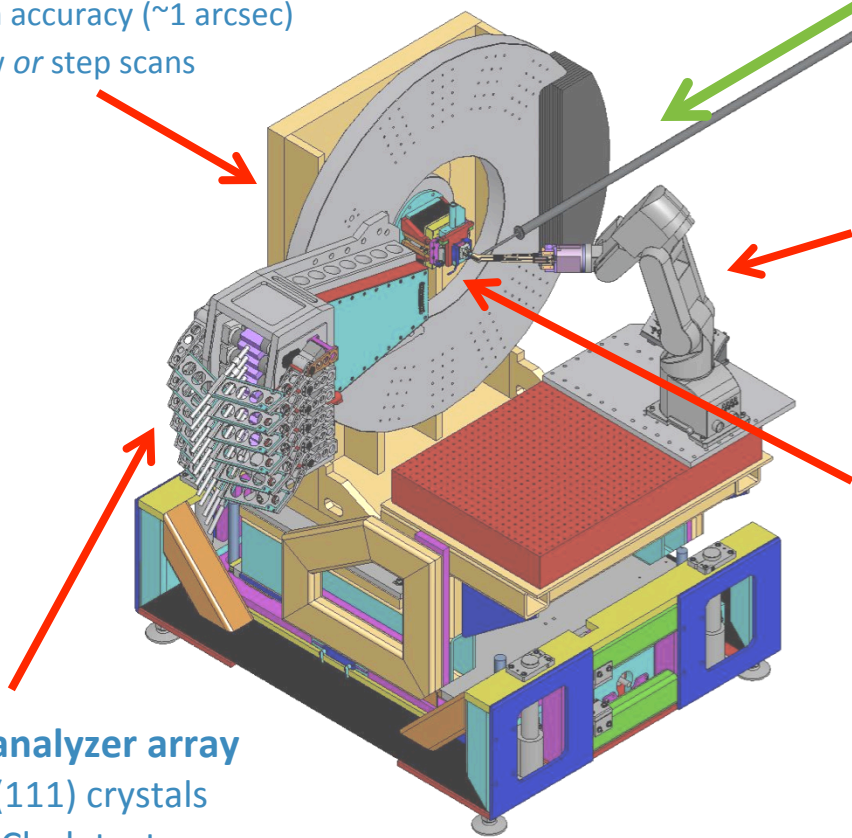
11-BM Diffractometer

Huber 480 rotation stage:

high precision (~ 0.35 arcsec)

high accuracy (~ 1 arcsec)

slew or step scans

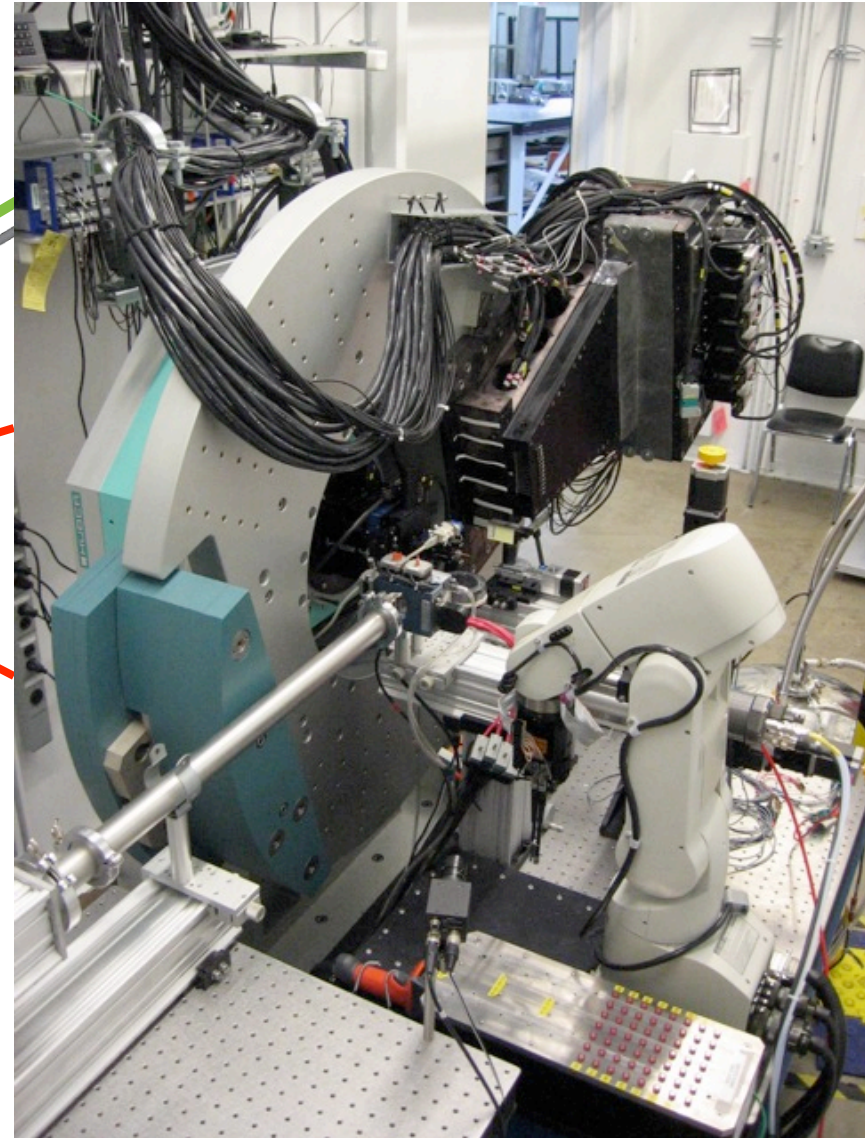


12 analyzer array

Si(111) crystals

LaCl₃ detectors

2° apart in 2 θ

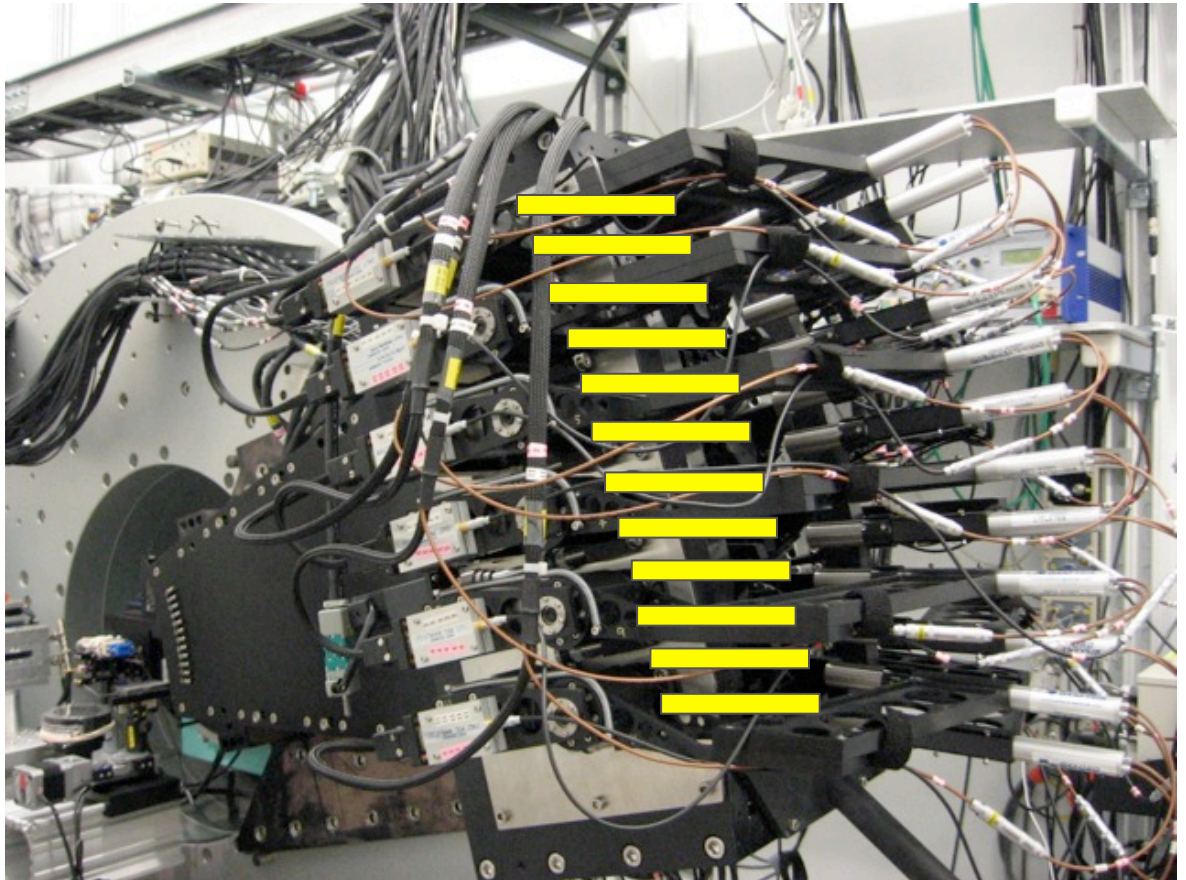


Multi-Crystal Detector Assembly

- 12 crystals/detectors
 - Individually adjusted
(*unique - theta & chi angles*)
 - 2° separation
 - Total 2θ range = 22°
- Si(111) crystals
 - excellent discrimination
 - low background (& yield!)
 - LaCl₃ Scintillators

Multiple Observations

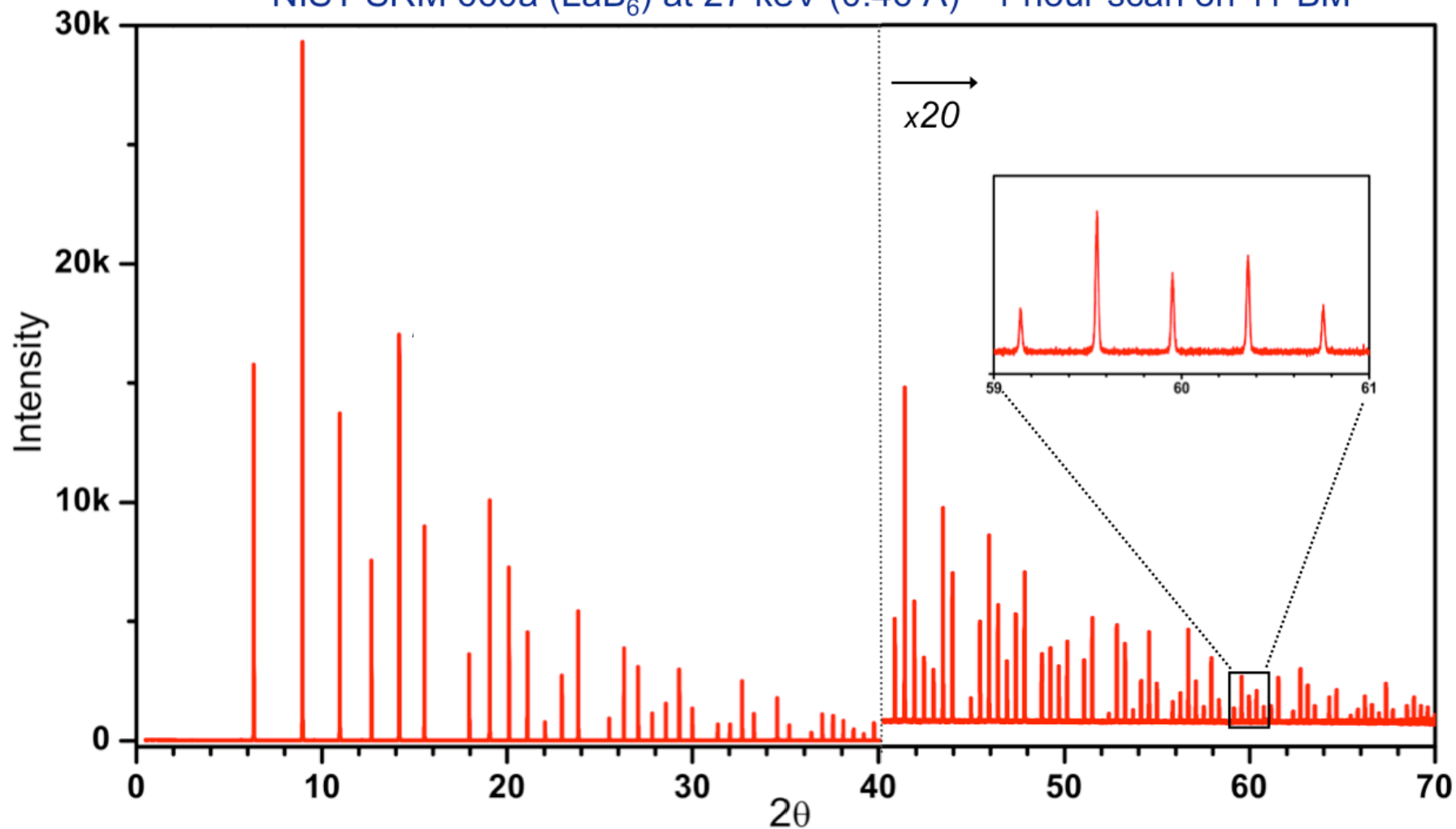
- Reduce Collection Time
- Improve Statistics
- Time Resolution



AES Design: Deming Shu & Curt Preissner

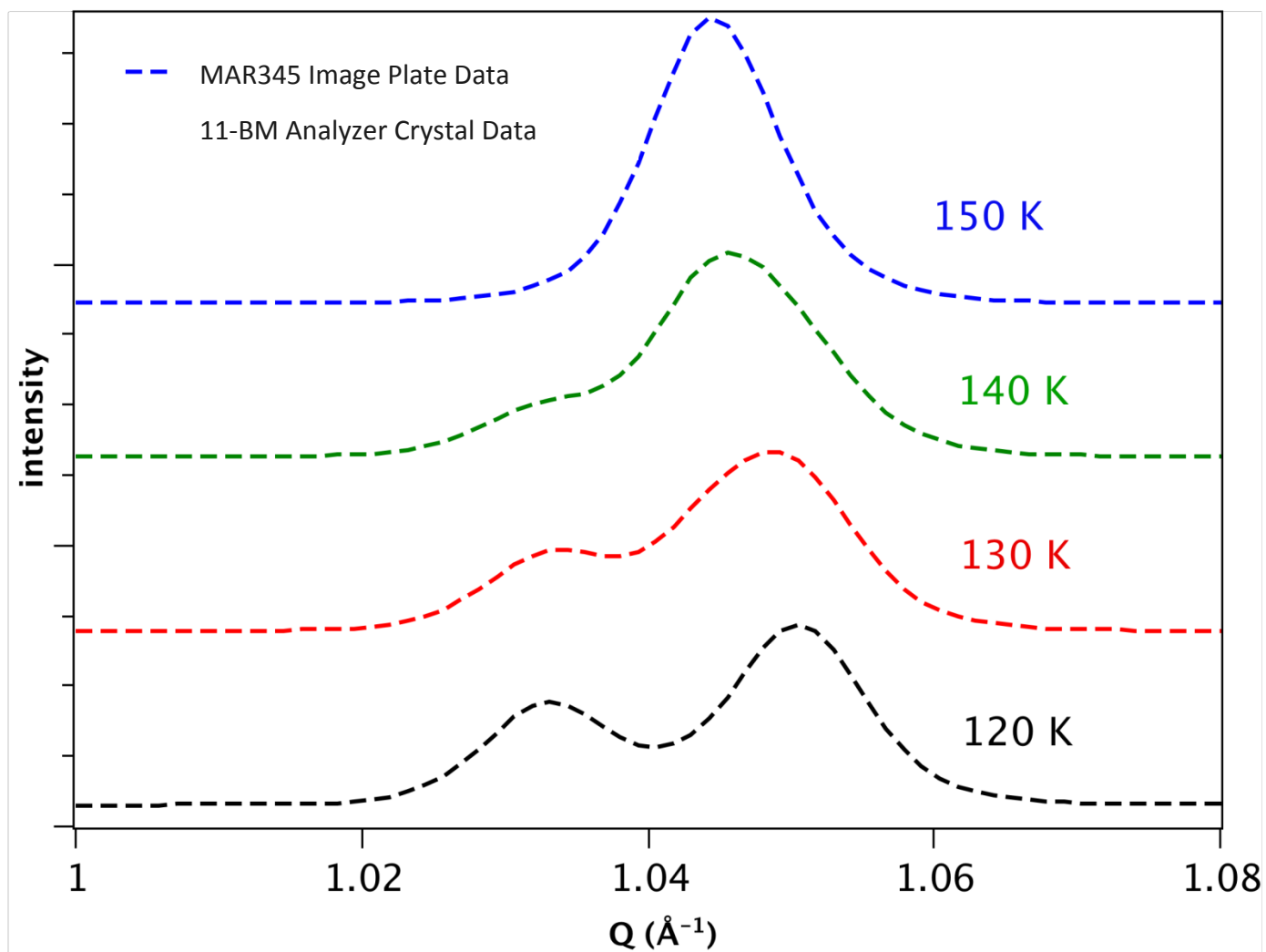
High Resolution Performance

NIST SRM 660a (LaB₆) at 27 keV (0.46 Å) - 1 hour scan on 11-BM



Analyzer Crystals vs. Image Plate

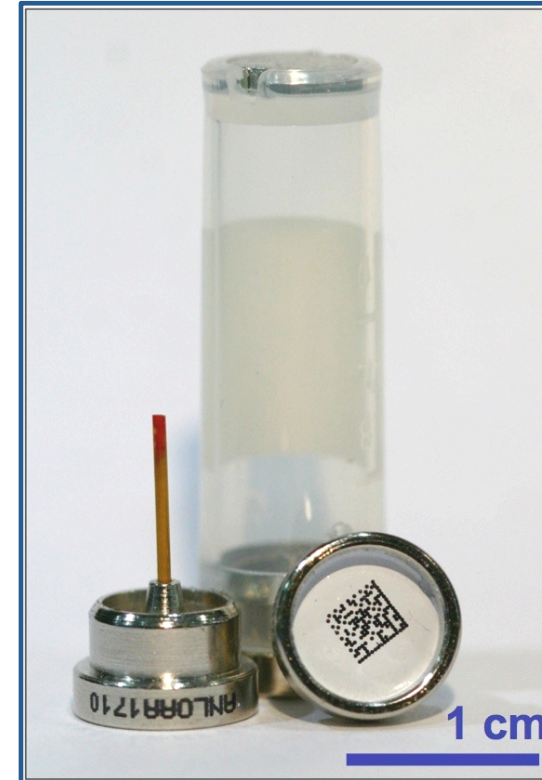
Phase Transitions Impact Gas Storage in Metal Organic Frameworks (MOFs)



Secrets of a Mail-In Service

High-Throughput with Minimal Effort

- Robotic Sample Loading Arm
 - 120+ Sample Capacity
 - Works Nights & Weekends
 - Optical Barcode Reader
- Bar Coded Sample Bases
 - Custom Hampton CrystalCap HT Bases
 - Magnetic Vial Cap
 - Kapton Capillary Tube (0.8 mm)
 - Unique Barcode for Each Sample
- Software & Databases (*kudos to Brian Toby*)
 - Automates Repetitive Tasks
 - Python, PHP/MySQL, HTML
 - Sample Tracking: Proposal to Disposal
request -> receipt -> data -> disposal -> nagging for publications



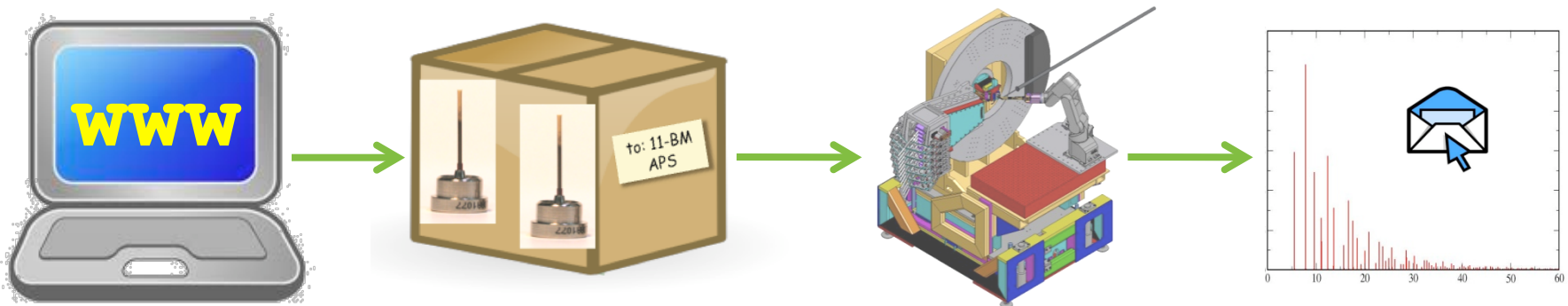
Robot Action



How it Works - the User Perspective

1. Submit Rapid Access Proposal for Beamline
2. Request, Receive, and Prepare Sample Kits
3. Register Sample Info and Desired Scan
4. Ship Sample to APS Beamline
5. Download Data when Notified
6. Publish Data and Report Citations

- free service for all non-proprietary users
- open to international users



The Advanced Photon Source is an Office of Science User Facility operated for the U.S. Department of Energy Office of Science by Argonne National Laboratory



User Perspective: Mail-In Workflow Features

- Convenient Web & Email Based-Workflow
 - Minimize Waiting for Staff Reply and Action
- User Responsible for Data Entry
 - No Repetitive Data Entry
 - User Responsible for Safety Information
- Metadata Access Limited by E-Mail Address(s)
 - Sample & Data Security without Passwords
- Rapid Service - No Travel
 - 3 weeks from Proposal to World-Class Data w/o Leaving the Lab
- Data Available Now (& *Later*)
 - Archived Storage for Data Retrieval Anytime



User Perspective: Requesting Sample Bases

- Received Notice of Accepted Rapid Access Proposal by E-Mail
- Web and E-Mail Instructions Guide User Step-by-Step
- User Data Entry Limited to **Red Text** Fields

11-BM Sample Activities

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

Select an action for 11-BM Samples

Step 1: Obtain sample mounting kit for an accepted proposal

Request sample bases for proposal #:

e-mail address:

On-site pickup at the APS?

SUBMIT

User Perspective: Confirm Requests by E-mail

- E-mail Confirmation Step Provides Security for Users & Staff

11-BM Mail-In Service Notice — Inbox

Delete Junk Reply Reply All Forward

From: 11-BM@aps.anl.gov
Subject: Request Confirmation

Dear 11-BM User,

Please confirm your contact information

Dr. Jones
State University
273 Smith Lane
Springfield 60501, IL

Click on the link below to have request

<http://11bm.xor.aps.anl.gov/user.ph>

User Information pulled from APS Database

Mail-In Sample Kit

- Vial of Kapton Tubes
- Magnetic Cap
- Mounting Base
- Kapton Capillary Tube

User Perspective: Enter Sample & Scan Info

11-BM Sample Activities

http://11bm.xor.aps.anl.gov/ Google

11-BM Sample Registration

*sample bases to register for email: **user@univ.gov***

Barcode	Scan	Formula	Name	APS Proposal
ANL01235	100 K	Al₂O₃	Alumina	23452
ANL01236	other	C₆H₁₂O₆	Glucose	23452
ANL01237				23452

Required Safety Information: Check Appropriate Material Hazard(s): ([Definitions](#))

None Toxic Flammable Corrosive Oxidizer

Radioactive Nano Bio Other:

SUBMIT

User Perspective: Safety Issues

- Required **E-mail Confirmation** Provides E-Signature
- Must Wait for “OK” Before Shipping Sample(s) to APS
- Automatic ESAF Submission Based on Supplied Information

11-BM Mail-In Service Notice — Inbox

Delete Junk Reply Reply All Forward Print To Do

From: 11-BM@aps.anl.gov
Subject: **Sample Registration Approval**

Registration for the following sample(s) has been approved:

Barcode	Scan	Formula	Name	Proposal	Hazards
ANL01235	100 K	Al ₂ O ₃	Alumina	23452	None
ANL01236	other	C ₆ H ₁₂ O ₆	Glucose	23452	None

The [ESAF](#) will now be automatically generated for your experiment.

You may now send your sample(s) to the APS for data collection.

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User Perspective: Obtaining Data

11-BM Sample Activities

http://11bm.xor.aps.anl.gov/ Google

11-BM Collected Data

*collected data for user with email: **user@univ.gov***

	Barcode	Temp (K)	File number	Date	Wavelength
<input checked="" type="checkbox"/>	ANL01235	100	apr11/11bmb_3691	2011-04-14	0.42133
<input checked="" type="checkbox"/>	ANL01236	295	apr11/11bmb_3692	2011-04-14	0.42133
<input type="checkbox"/>	ANL01137	200	jan11/11bmb_1286	2011-01-28	0.84121

select data format ([Definitions](#))

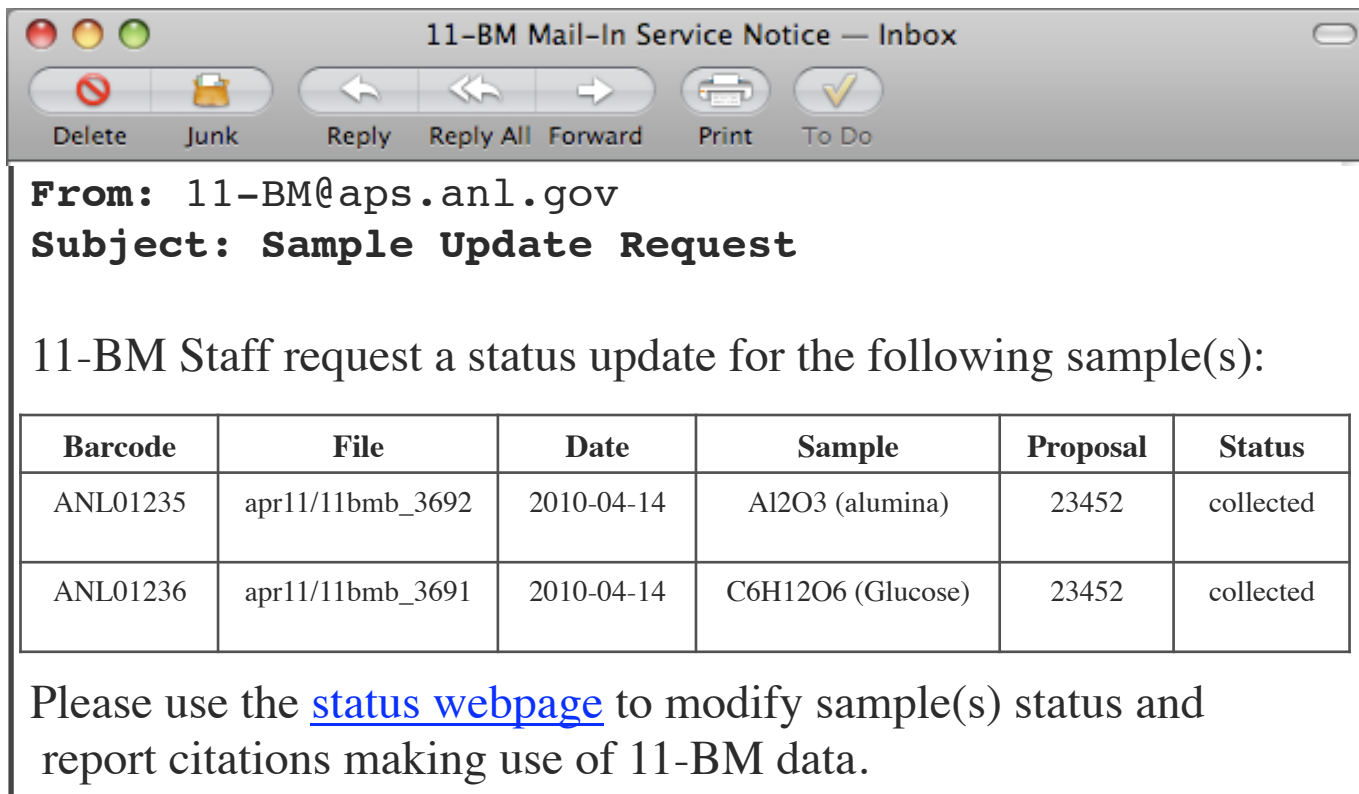
.fxye (GSAS) .xye (Topas) .xy (FullProf) .csv (ASCII Text)

send data by email (or) *post to ftp site*

SUBMIT

User Perspective: Reporting Citations

- Automatic Follow Up – i.e. *Periodic Nagging Emails*
- Request to Update Sample Status (Analyzing, **Published**, **Abandoned**, etc)
- Response Tracked, Impacts Future Instrument Access



From: 11-BM@aps.anl.gov
Subject: **Sample Update Request**

11-BM Staff request a status update for the following sample(s):

Barcode	File	Date	Sample	Proposal	Status
ANL01235	apr11/11bmb_3692	2010-04-14	Al2O3 (alumina)	23452	collected
ANL01236	apr11/11bmb_3691	2010-04-14	C6H12O6 (Glucose)	23452	collected

Please use the [status webpage](#) to modify sample(s) status and report citations making use of 11-BM data.

Behind the Curtains - the Staff Perspective

- Staff Web Interface to Sample Database
- Extensive Use of Handheld Bar-Code Scanners
minimize typing (& typos!) for common tasks - issuing bases, receiving & disposing samples

11-BM Staff Sample Activities

http://11bm.xor.aps.anl.gov/staff.php

Database Query & Editing

Wild-card search/edit for entries. Display entries starting with entry .

Sample barcode number: * **ANL03668**

AND e-mail: *

AND chemical name: *

AND chemical formula: *

AND sample name: *

AND sample status is

AND Proposal number between and

AND ESAF number between and

AND storage location is / is not

AND sample return is requested

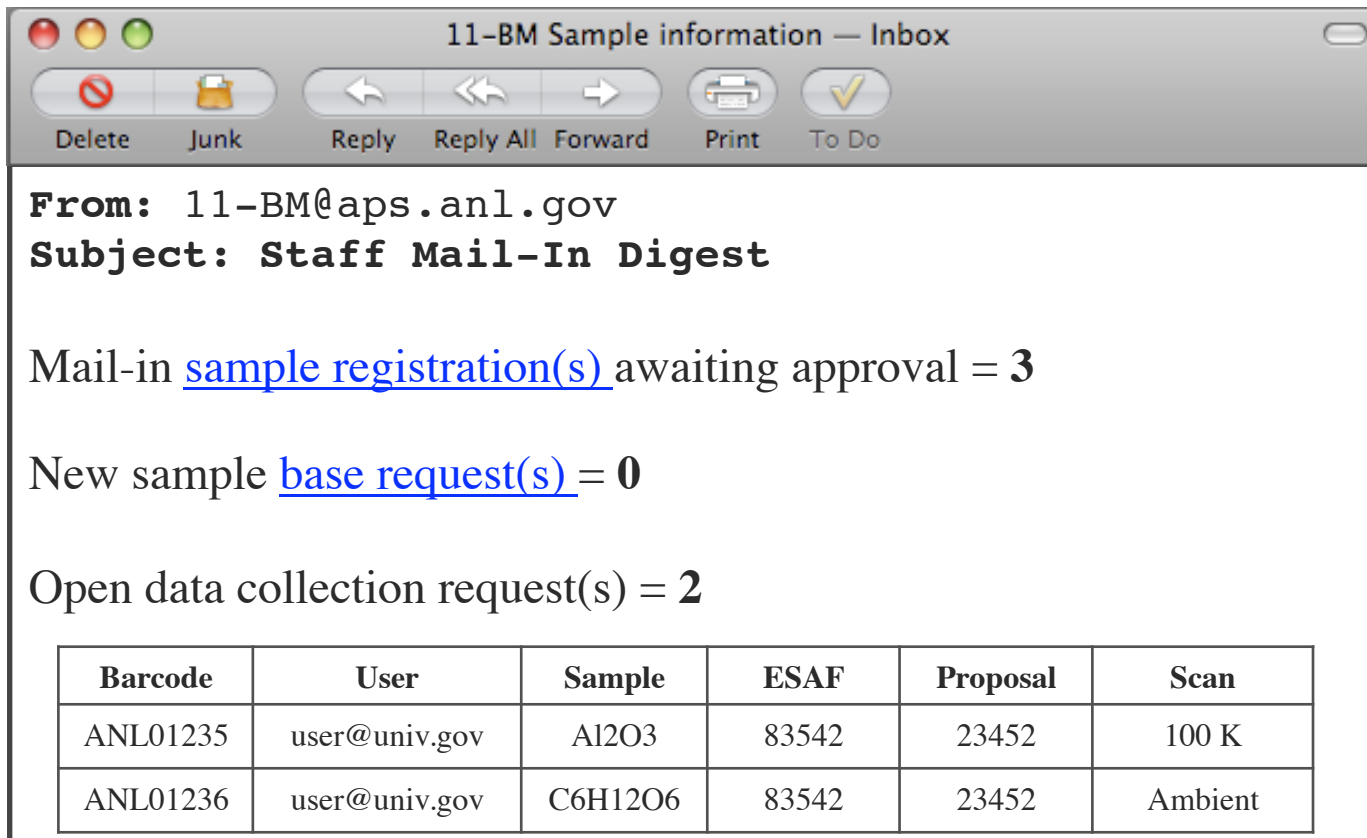
Sample Information

Run request Information



Staff Perspective - Simplify Repetitive Tasks

- Email Notifications for Mail-In Workflow Events
- Pre-Approved Packages Eases Sample Base Shipping
 - No transfer order required to ship out sample base kits



From: 11-BM@aps.anl.gov
Subject: Staff Mail-In Digest

Mail-in [sample registration\(s\)](#) awaiting approval = **3**

New sample [base request\(s\)](#) = **0**

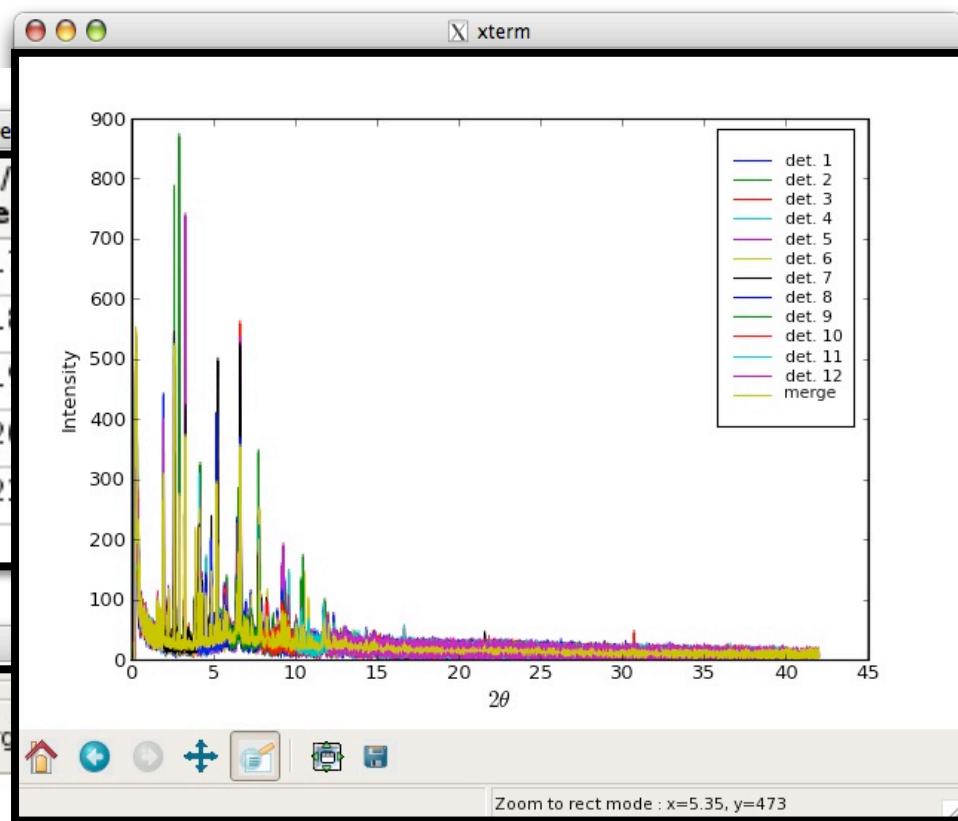
Open data collection request(s) = **2**

Barcode	User	Sample	ESAF	Proposal	Scan
ANL01235	user@univ.gov	Al2O3	83542	23452	100 K
ANL01236	user@univ.gov	C6H12O6	83542	23452	Ambient

Staff Perspective - Convenient Data Reduction

- Quick Visual Inspection of Data for “Quality Control”

Barcode ID	Data file	Date/colle
ANLOAA1220	oct09/11bmb_3735.mda	Oct 12 1
ANLOAA1220	oct09/11bmb_3736.mda	Oct 12 1
ANLOAA1220	oct09/11bmb_3737.mda	Oct 12 1
ANLOAA1220	oct09/11bmb_3738.mda	Oct 12 2
ANLOAA1220	oct09/11bmb_3739.mda	Oct 12 2



Post-merge options

Choose an option for processing this merge

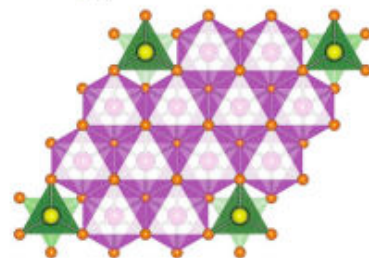
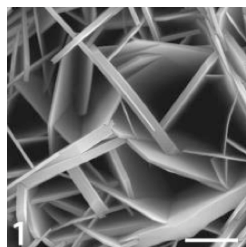
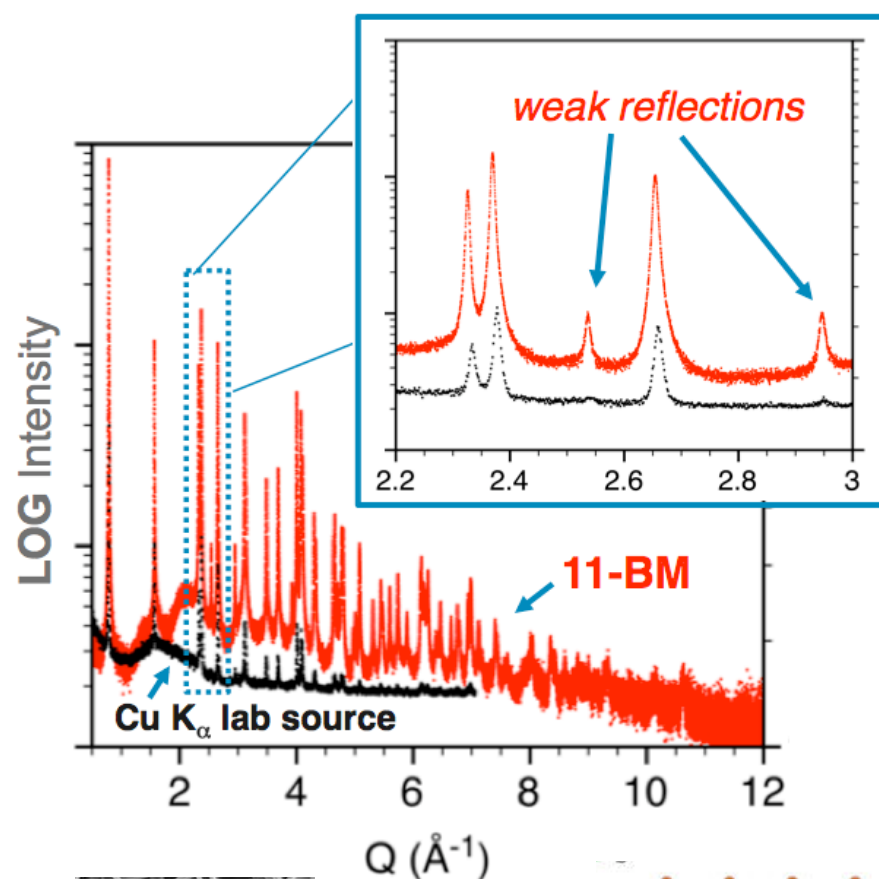
Comment to User

Data look Great!

Skip/Reprocess Recollect **Accept Merge**



Mail-In User Science Example



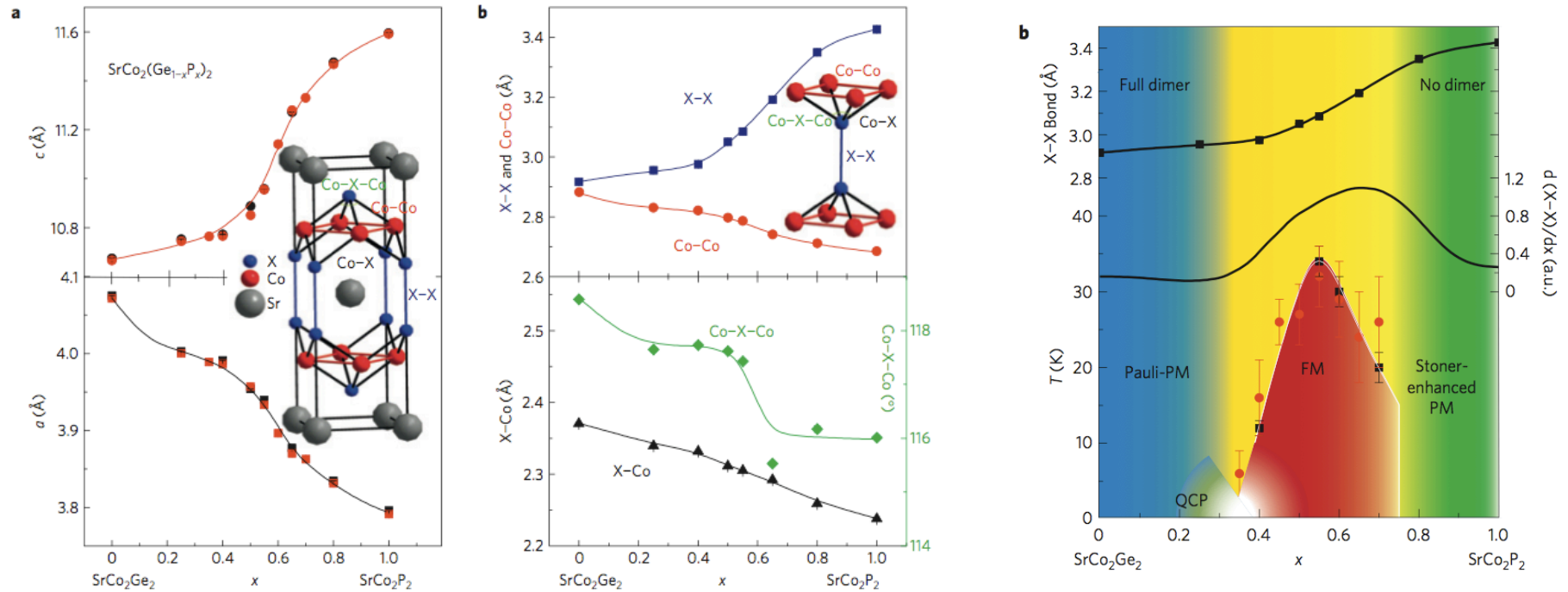
11-BM Provides Enhanced Structural Detail in Bio-Inspired $\text{Co}(\text{OH})_2$

- Aqueous synthesis, inspired by biomineralization processes
- Promising ion exchange applications
- Brucite Structure: $\alpha\text{-Co}(\text{OH})_2$
 - disordered defects
- Compare 11-BM vs lab-based XRD
 - short wavelength avoids edges
 - superior counting statistics
 - greater resolution & observations

J. Neilson, *et al. Inorg. Chem.* **2009**, *48*, 11017-11023

Mail-In User Science Example

Unique Rapid Access Mail-In Program at APS beamline 11-BM Enables High Impact Discovery of Quantum Critical Point Control by Chemical Tuning



Structural and bonding trends, obtained from high-resolution diffraction at the APS, show the collapse of tetragonal distortion in the layered intermetallic $\text{SrCo}_2\text{Ge}_{2-x}\text{P}_x$ system, driving ferromagnetic order and a quantum critical point transition.

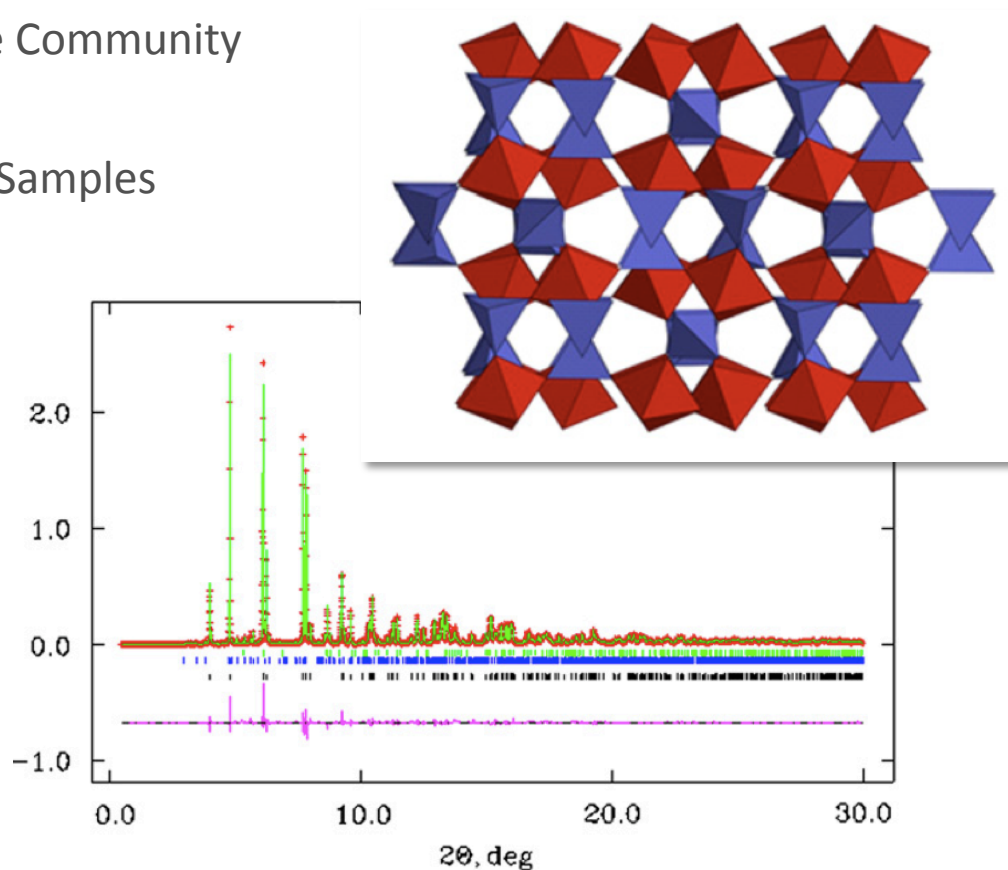
Jia *et al.*, *Nature Physics* (2011) v. 7 p. 207

ANL “Drop-Off” User Example

- “Drop Off” Service for Argonne Community
- Extensive Use by MSE Division
- Ideal for Small or Air Sensitive Samples

Structural model and refinement of $Sc_{0.67}WO_4$ - new NTE material synthesized under high-pressure & high temperature methods

Measurement on < 100 mg of powder

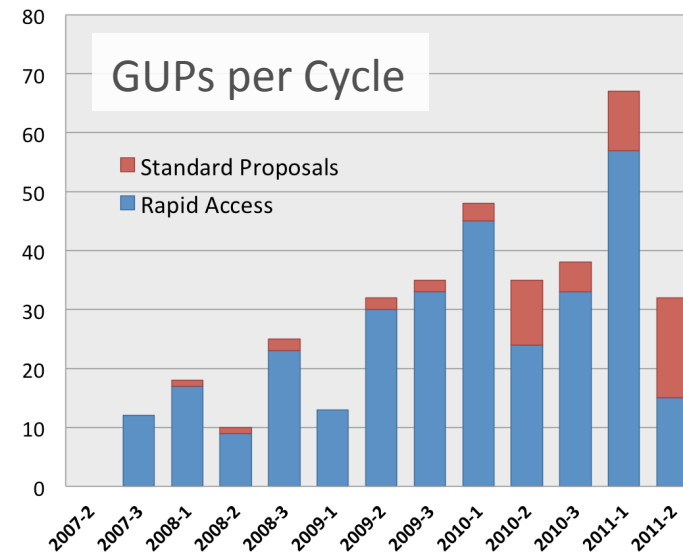
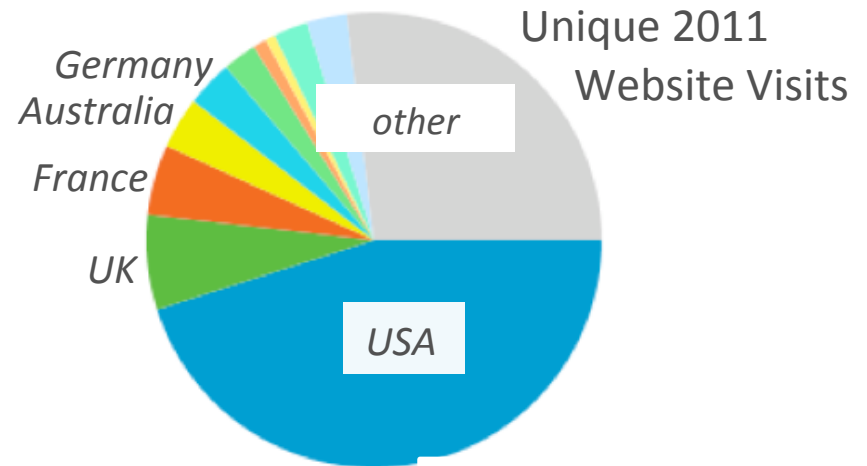


Varga, Mitchell et al, JSSC , 2010 vol. 183 pp. 1567

Demand for a Mail-In Service?

- Plenty, and not just Domestically

- Web Page Traffic
~ 150 hits/week
- Rapid Access Proposals
~ 50 requests per cycle
> 15 already for 2011-2
- Mail-In Measurements
~ 2,500 samples, > 7,500 files emailed
- Publications
> 60 Reported (since 2007)
~ 10 in 2011 (to date)
- Industrial Users
several per cycle, growing interest



But Wait, There's More!

- Also Supporting On-Site User Experiments (50% of shifts)
- Ideal In-Situ Probe: Real Materials - Real Time
- User Friendly Interface with GUI-based Controls (*R. Von Dreele*)

The screenshot displays the '11BM-B User Control' software interface. The main window has a menu bar with 'Run', 'Tasks', and 'Utilities'. The 'Tasks' menu is open, showing options like 'Load Sample', 'Unload Sample', 'Slew scan', 'Un/Load Cryostream', 'Cryo Temperature', 'Hot air blower parameters', 'Wait time', and 'Z translate'. A red arrow points from the 'Slew scan' menu item to a 'Cryostream' dialog box in the foreground. The dialog box contains the following fields and options:

- Set Point Temp, K: 0.0
- Ramp Rate, K/min: 5.0
- Temp. window, K: 0.0
- Delay after T reached, m: 2.0
- Wait for set point?
- Buttons: Ok, Cancel

In the background, the 'Task List' panel shows five tasks:

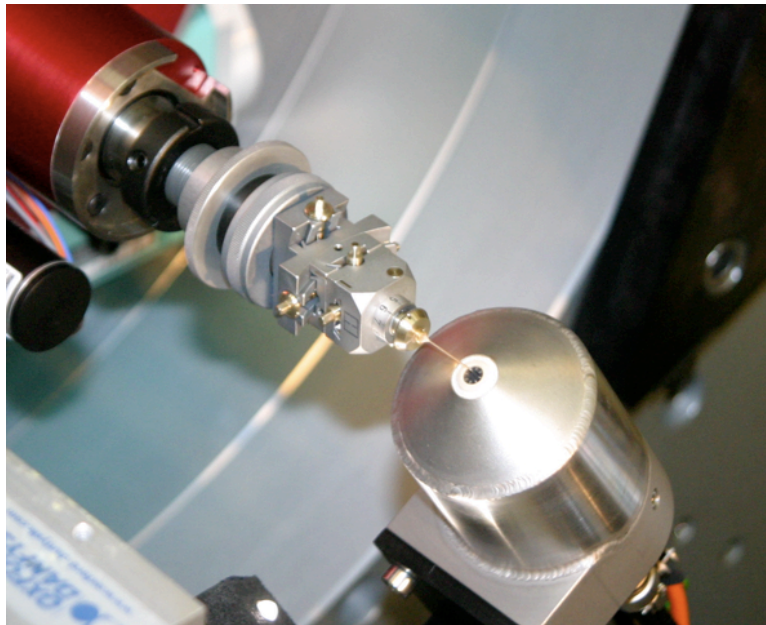
- Task #1: Cryostream: Setpoint: 100.0 Ramprate: 5.0
- Task #2: Slew scan: -6.0 to 24.0 step: 0.001 step tin
- Task #3: Wait: 5 min
- Task #4: Cryostream: Setpoint: 150.0 Ramprate: 5.0
- Task #5: Spinner: on

The main interface also shows input fields for 'Sample ID: sample', 'Sample name: sample name', 'Composition: ABO3', and 'Comment: Manual Data Collection'.

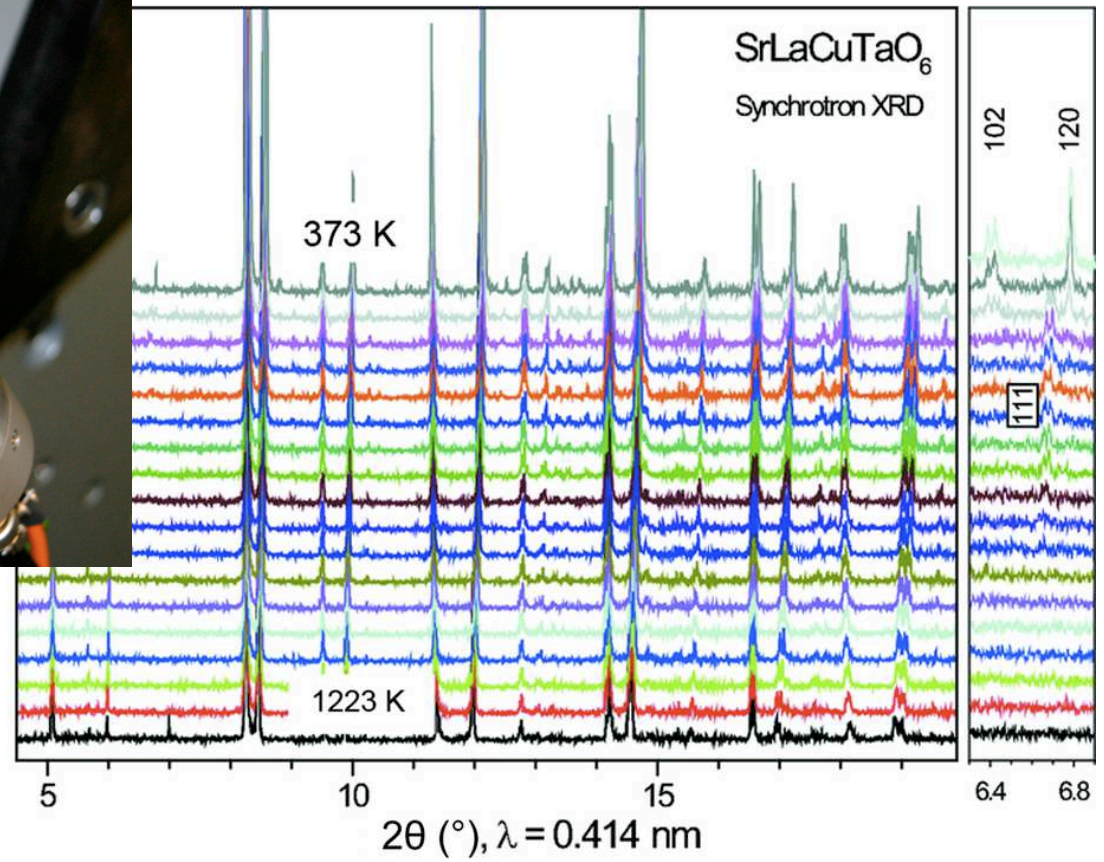


Heating Samples

- Gas Blower Heats Capillaries to 950 °C
- Intermetallic and Ceramic Phase Transitions



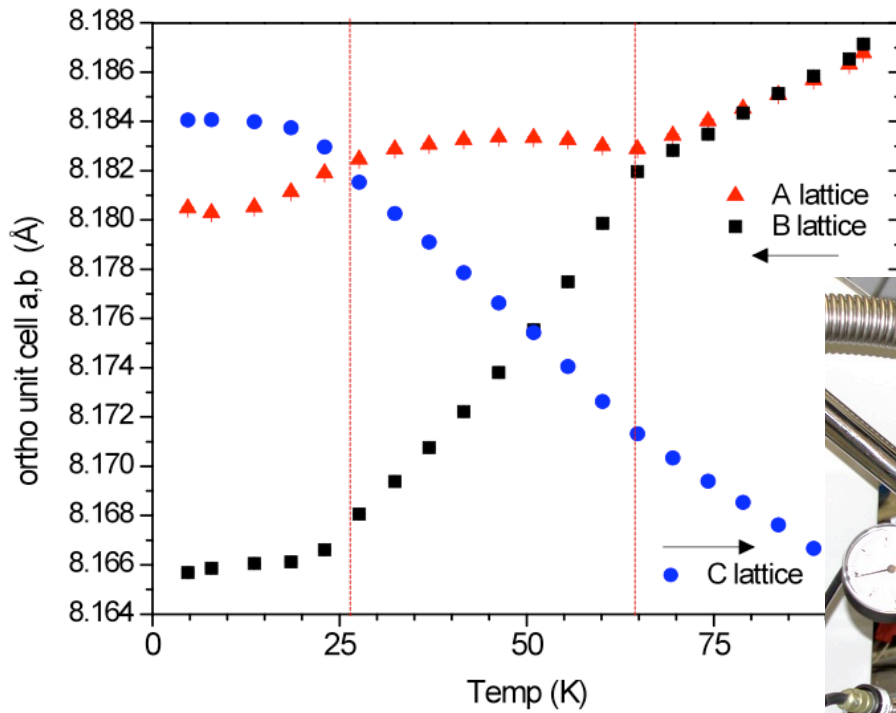
superstructure peaks show tilt symmetry transition from triclinic to monoclinic



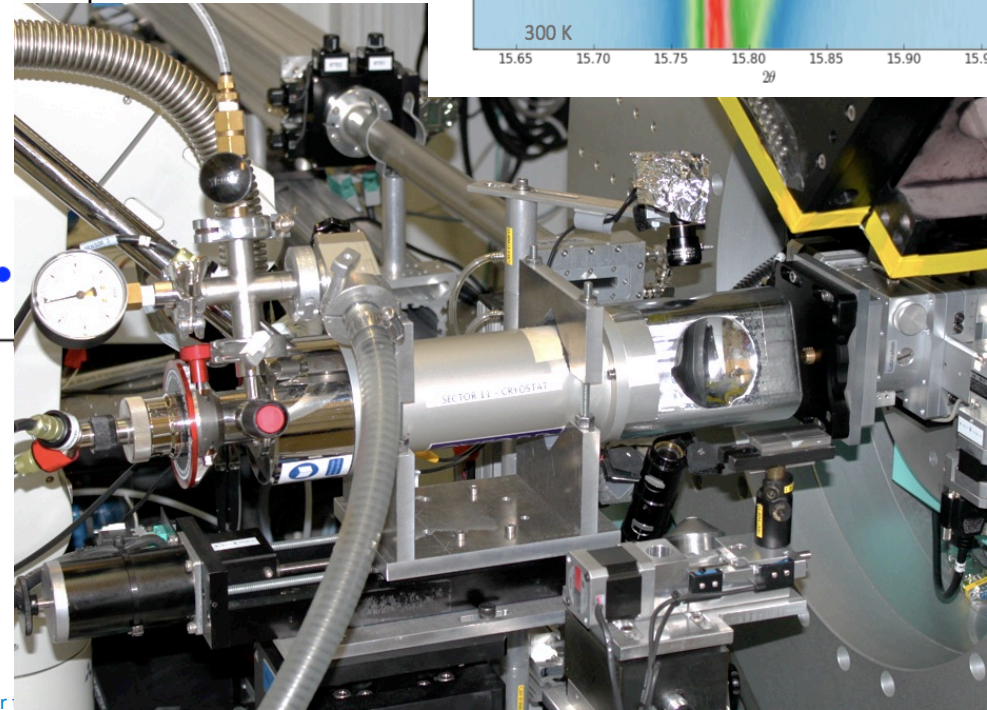
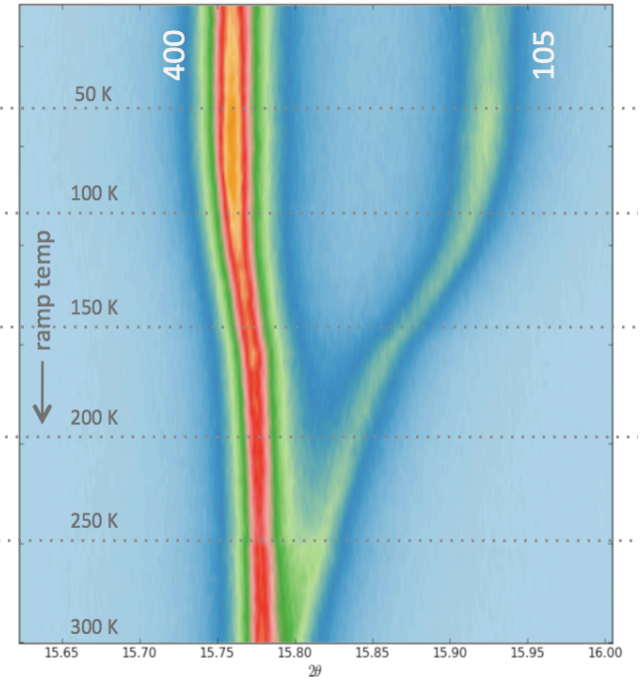
West D, J App Cryst (2011) v 44 p 595

Cooling Samples

- Closed-Flow Helium Cryostat (> 5 K)
- Structural Studies in Magnetism & Superconductivity



tracking lattice values in CuCr_2O_4

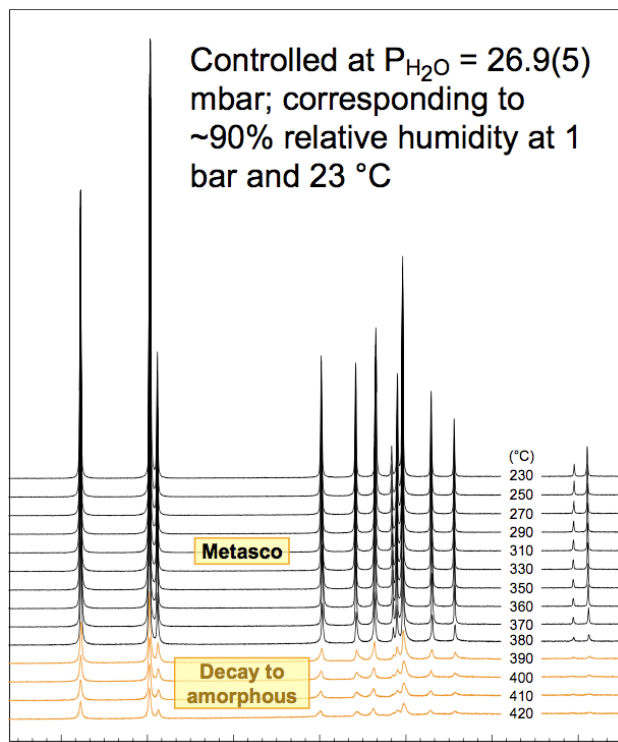


Seshadri *et al*

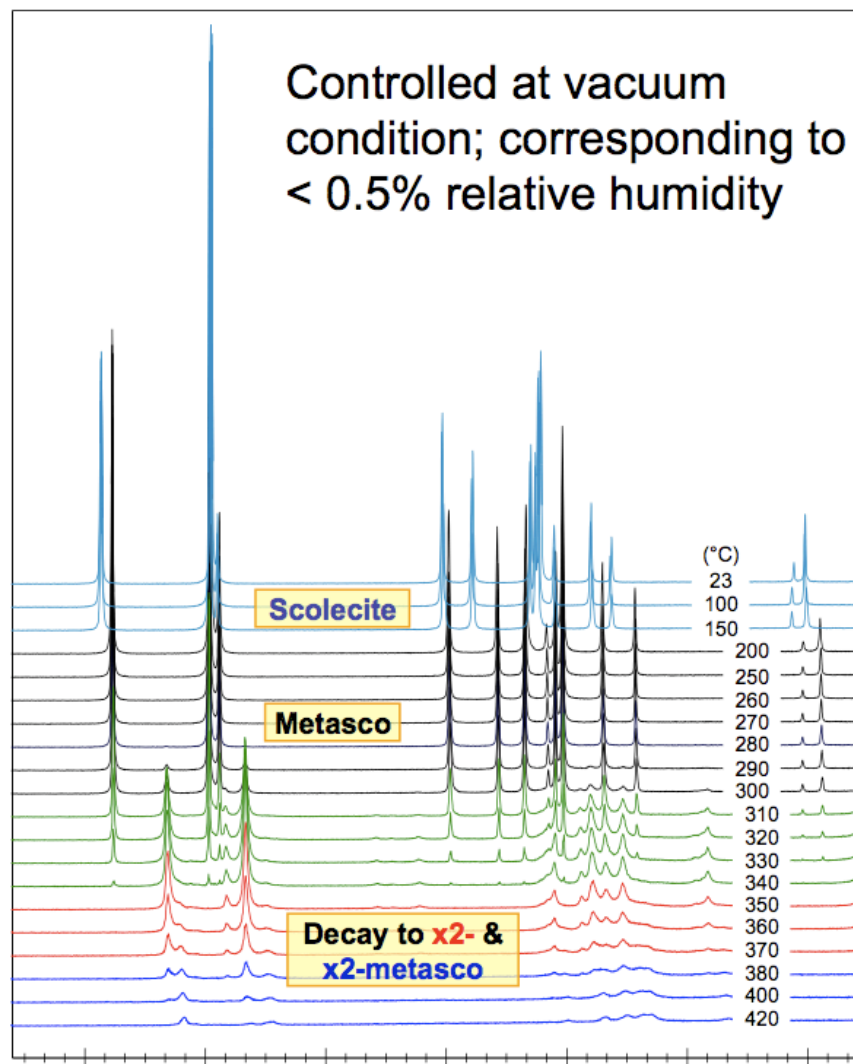


and Atmosphere Control

- Reaction Cells & Gas Flow Meters
- Gas Reduction or Humidity Studies



Bish Group , IU-Bloomington



Zeolite degradation path varies with P_{H_2O} conditions

Accomplishments & Future Plans

Unique Rapid Access Mail-In Program Established

- Offer World Class Data; Selection of Scan Parameters & Temps (100-450K)
- > 250 Unique Users, > 60 Publications to Date
- Future Growth Expected, but Approaching Capacity
- Challenges: Diverse User Background & Training (*Sample Prep, Data Analysis*)

Expanding On-Site Experiment Capabilities

launched summer 2010

- User Friendly GUI-based Control Software Nearing Completion
- New In-Situ Sample Environments:
Cryostream, Gas Blower, Cryostat (> 5 K), In-Situ Pressure & Gas Cells

What's Next?

- Expand Working Energy Range & Simplify Changing Energy
- Anomalous & Resonant Power Diffraction
- High temperature (> 1000 C) sample furnace
- Increased support for structure solution from powder

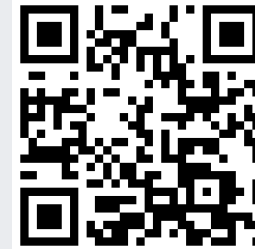


More Information

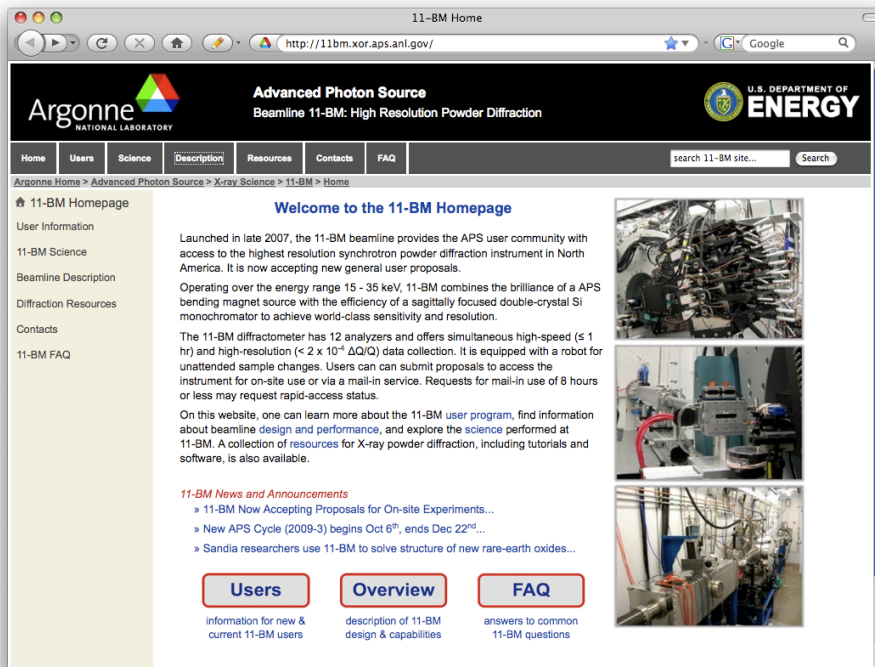


11-BM webpage: <http://11bm.xor.aps.anl.gov/>

email contact: 11BM@aps.anl.gov



11-BM Website



11-BM Acknowledgements

11-BM staff (past & present): Lynn Ribaud, Brian Toby, Bob Von Dreele, Jennifer Doebbler, Jun Wang & Sytle Antao.

APS Support: Peter Lee, Mohan Ramanathan, Chuck Kurtz, Curt Preissner, Xuesong Jiao and many many others...

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