Crystallography Software Development and Data Management

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Microcrystallography at ChemMatCARS 2000 - 2002

- Bruker Mosaic CCD detector was mounted on a Bruker Kappa goniometer.
- This instrument offered a rapid coverage of reciprocal space to a resolution of 0.50 Å with a combination of ω and φ scans.
- Nitrogen cryostat provided routine data collections down to 90 K.
- The detector did not live up to expectations for a number of factors.
Target Data Collection Parameters for the Kappa Mosaic

- The Mosaic detector provided a large solid angle for data collection.
- Remote positioning of the detector allowed one to make the best use of the diffraction available.
- Four CEUs allowed frame readout in 1K mode in ~3 seconds – very slow!
- The insertion device provided a high flux of photons at short wavelengths without \( \lambda/n \) contamination.
- The phosphor was optimized for long wavelengths so transparency was an immediate problem.
Microcrystallography at ChemMatCARS
Crystallographer Jamboree

• In June 2002 we had 10 service crystallographers who arrived at ChemMatCARS to collect micro-crystallography data during 6 days. About 40 projects were worked on during this time.

• Crystallographers from Indiana, Wisconsin, Iowa, Minnesota, Chicago, UMSL, SUNYSB, and Cincinnati visited.
Time-Resolved Photocrystallography at ChemMatCARS 2002-2003

• Bruker SMART 6000 CCD detector is mounted on the Huber 6-circle goniometer.
• This goniometer only provided one axis of rotation so it had limited usefulness for high-throughput microcrystallography.
Microcrystallography at ChemMatCARS 2004

- Microcrystallography instrument in use in fall 2003.
- Oxford Cryojet cryostat is in place.
- Image at left is 1 of 4 web-based images from the “Xport” web page. This provided remote user access to the beamline.
Lessons learned in pursuit of SCD at ChemMatCARS

• The beam is highly polarized: \( p \sim 0.99 \).
• Air scatter of beam contributes significantly to background – helium purge reduces effect.
• Software was added to track beam drift to make minute corrections in mirror positioning.
• Bruker SMART 6000 and APEX-II detectors were easily “topped” so usage of an attenuator was necessary: lost beam intensity that could not be used.
• Some commercially-available detector phosphor materials were sufficiently transparent to beam with \( \lambda \sim 0.50 \) Å that a geometrical correction was necessary: Coppens group OBLIQUE program provided a correction to this.
• Mounting microcrystallography specimens on glass fibers pulled to a point is better than using MiTeGen dual-thickness Microloops™ for both positioning and background.
• Every sample mounted costs at least 5 minutes of beam time for manual mounting and opening/securing PSS.
• Microcrystallography-sized specimens usually harbor serious crystallographic problems: pseudo-symmetry, twinning, and disorder.
May 2019 collaboration with P. Kaszynski: Crystals are found as flexible, branching filaments ~0.5-1.5 µm thick and ~3-5 µm wide for single specimens.
Microcrystallography:
A paramagnetic nano-graphene compound

- Highly persistent, planar radicals based on the 1,4-dihydro[1,2,4]triazin-4-yl unit
- Space group is $P2_1$ with $Z' = 2$
- Very good pseudo-symmetry for screw axis parallel to $c$-axis with $\Delta Z \sim \frac{1}{3} c$
- Also detected twinning as 180° rotation about $c$-axis ($\beta \sim 90°$)
- Resolution maximum 0.9Å with 5 sec. scans 0.6° width.
- $R1 = 16\%$, $wR^2 = 36\%$

P. Kaszynski (private communication)
Two specimens examined
Dectris *.cbf* format files were converted to Bruker *.sfrm* format for integration
Played with wider frames, but settled on 0.6° frames (5 sec. each) for refinement.
Total space ~15Gb!
APS 15-ID Layout

A Second, Independent Beamline on NSF’s ChemMatCARS Sector
2 independent beams to serve 2 concurrent experiments
Dectris Pilatus Detectors

Pilatus (CdTe) 1M
500 Hz readout
frame size 4M
`cbf` format

Pilatus (Si1mm) 2M
250 Hz readout
frame size 8M
`cbf` format

These detectors may be used separately for different experiments or together in the same.
The utility program *PilatusTIFF* performs a real-time conversion of the Pilatus *.cbf* format files to Bruker APEX-II readable *.sfrm* files. It includes background subtraction and concatenation of runs with common orientation. The majority of users work comfortably with the Bruker APEX-II software for structure solution and refinement.
Experimental Setup
Robotic Automation

Many beamlines at APS have installed robots to load and change samples as part of high-throughput data collection. Pictured here is the robot for the 11-BM powder diffraction station. Beam time usage will be more efficient by employing robotic automation, however no funding for this item has been acquired at present for the new 15-ID station.
11-BM User Program

User proposals for beamtime may request on-site experimental time or rapid-access via a mail-in program. Proposals from industrial users are also accepted.

The unique 11-BM mail-in service allows users to submit a short proposal and receive world-class powder diffraction data for their experiment typically in less than one month. This is a free service for all non-proprietary users.

To maintain a swift turnaround for all users, rapid-access allocations are limited to a single APS shift (8 hours, or 8 samples) per proposal. Experiments requiring more beamtime must submit a standard proposal via the normal APS review process (see deadlines).

Mail-in data collection is performed at a fixed energy (~ 30 keV), with limited collection at 27 keV for Sn containing samples, and users are offered a section of scans at a select number of temperatures. On-site experiments are required for more specialized measurements or for samples not acceptable for the mail-in program.

All beamtime requests must be submitted through the Advanced Photon Source General User Program. More information about 11-BM capabilities and our unique rapid-access mail-in program can be found throughout this website and on the 11-BM FAQ page.

More information is available for new and current users.

More information is available for industrial users.

New measurement protocols are often added or considered; please feel free to contact 11-BM staff with any questions or comments, or for more information about on-site experimental capabilities.
**Instructions for 11-BM Mail-in Users**

For 11-ID-B and 17-BM mail-in users, instructions will be uploaded shortly and for now please communicate with the beamline scientists at the corresponding beamline.

**General Notes**

Carefully read and follow these instructions to ensure a successful mail-in experiment. Contact 11-BM staff with questions at anytime.

Please note the APS run [Schedule](http://example.com) and proposal [Calendar](http://example.com) dates.

Samples arriving during or just a shutdown will be saved and run in the following cycle. Contact staff for details.

Proprietary industrial users should consult additional instructions about performing experiments at the APS.

Please note additional important APS safety guidelines. Although many apply only to on-site users, Mail-In users should review notes for Experiment Hazard Classes and restrictions on the Shipping of Samples.

**Submit a Proposal**

To obtain experimental time at 11-BM, all users must submit a General User Proposal (GUP). Proposals for the rapid access mail-in service are limited to 1 shift (~8 hours or 8 samples) per proposal, but may be submitted at anytime. Standard proposals must be submitted prior to the run cycle deadlines (three times per year). Check the GUP calendar for exact dates.

See our detailed [step-by-step instructions](http://example.com) describing the General User Proposal (GUP) system, plus how to complete and submit a rapid access mail-in GUP. Note: at least one member of your research team must be a registered APS user. A valid APS user badge number and password is required to access the system and submit a proposal for beamtime; this can be obtained by registering with the APS User Office. For 11-BM mail-in users, an example registration is available [here](http://example.com) (PDF document).

**Safety Notice**

The mail-in service **cannot** accept any of the following sample types:

- Biohazards
- Human-Derived Materials
- Radioactive Materials
- Non-Sterilized Regulated Soils
- Explosives or Unstable Materials
Example: 11-BM Mail-In Kits

Request Sample Base Kits
Rapid access proposals for the mail-in service are normally reviewed within 1 week. You will receive email notification if your proposal is accepted. This email will contain instructions (and web links) describing how to request sample base fits for your mail-in experiment. Follow the email links, or navigate to:


At the above link, users enter a GUP proposal number, the associated e-mail address, and requested number of sample base kits. Argonne or APS based users may also specify that sample bases be held on-site (not mailed) for local pickup. Note: rapid access proposals are limited to 1 shift (~8 hours or 8 samples) per proposal.

Once bases are requested, a confirmation email message will be sent. Please double check that the user name(s) and shipping address (listed in the email message) are correct. You MUST click on the link embedded in the email to confirm your request. Bases will not be sent without your confirmation.

Receive Sample Base Package
Sample base kits are shipped to your confirmed address. In this package, you will find your requested mail-in sample base kit(s) and a pre-printed return shipping label. For each requested sample base, the package contains the following: one sample mounting base, one magnetic cap, and Kapton sample capillary tubes (inside a plastic vial). Additional Kapton tubes are provided for your convenience. Requests are normally processed within a few days, and packages are sent via the US Mail.

Each part of the sample base kit is described below and shown in the image on the right. Also see our image gallery for more sample base pictures.

**Mounting Base:** Holds one sample capillary tube and is integrated with the robotic arm used for automated data collection. The unique 10 digit barcode (e.g. ANL00AA1234) printed on the side, also encoded in a 2D barcode on the bottom, tracks the sample at all steps of our mail-in service. Note: The barcode number is required when registering each sample online.

*Do NOT return any materials to the APS until your samples have been registered, verified, and approved by beamline staff.*

**Magnetic Cap:** A magnetic ring secures caps on mounting bases, and provides some protection for sample capillary tubes during shipment. Please avoid writing on the caps, as they are re-used and cannot be used to identify your sample.

*Note:* all samples must fit completely inside the caps when placed on mounting bases; longer samples may be destroyed by the 11-BM robotic sample loader.

**Capillary Tubes:** The supplied Kapton tubes have an inner diameter of 0.80 mm, and an outer diameter that fits snugly inside 11-BM mounting bases. Kapton is stable over a wide temperature range and adds a negligible contribution to the diffraction scan background. *Note:* All sample powders must be fully contained and secured within these capillary tubes.
Example: 11-BM Data Retrieval

Select an action for 11-BM Samples

*Step 4: Download diffraction data for completed samples*

Search for samples registered by e-mail address: 

that were collected since 

Display entries starting with entry 1

Get File List
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