Bruker AXS

Your Questions Answered

from SC-XRD Webinar: *Finding That Diamond in the Rough*

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Q: What App are you using? I am using crystal Pro. How can I get this app... is it downloadable online?

A: The demonstration was done using APEX3 software controlling a D8 QUEST diffractometer with a PHOTON III detector, Mo sealed tube and TRIUMPH monochromator, and a D8 VENTURE dual IμS microfocus source system with a PHOTON II. The software can be downloaded from www. brukersupport.com. We do provide demo licenses also.

Q: What if you are doing a sample that is totally unknown? How will you know that you are having the right space group?

A: We provide software within the APEX3 software that helps you determine the space group by examination of the Laue symmetry, |E| statistics, and systematic absences. Structure solution can also determine the space group from phases. All it needs as input is the Laue class.

Q: At what point would you want or need to deal with the additional lattices and/or overlap?

A: If the only crystal you have has more than one domain, you would determine those domains and carry multiple orientation matrices forward. Data reduction (integration and scaling) would then deal with the multiple matrices and provide files for structure solution and refinement ---, for structure solution: a file that has the de-convoluted information and for refinement: a file

that has information on all domains that were identified. The ratio of the domains is then determined during structure refinement.

Q: How would you know you have the "correct" cell if you did not know what it should be, like you did with sucrose?

A: In the end this is what crystallography is all about: finding the unit cell, the unit that repeats over and over. There are quite a few checks in the software during indexing (unit cell determination) that help you to do that. If you have multiple domains that might not be easy. Looking at reciprocal space and using the tools in the plugin provide you with a visual check. You can also see whether the predicted overlay of reflections matches the diffraction pattern. If you have more than one independent molecule in the asymmetric unit during refinement, that should raise a red flag. Tools like PLATON will check for you if an axis should be halved or doubled.

Q: You just treat the twin crystal as a single crystal, right?

A: This is what we did. If there is not a lot of overlap, then you might get away with this. It's always better to look for a single crystal. If you don't have one, you should treat it with multiple matrices.

Q: Is the procedure demonstrated for fast scan limited to an instrument that is set up for shutterless? I have previously done similar methods of examining crystals just using the experiment plugin.

A: You did the right thing! The method is not limited to a shutterless instrument with a large detector that can reach atomic resolution with Mo at 2Theta at zero. You can do this with a SMART 1000 or an APEX II system if you choose an offset to be able to reach the necessary resolution.

Q: Is Photon III detector in the market already?

A: The large PHOTON III, 20 cm x 14 cm, is available.

Q: With these very well diffracting crystals you can easily get overloads at one second, making the fast scan not "fast" enough. Can you insert the attenuator from the data collection strategy for only the fast scan?

A: The current release of APEX3 lets you choose the attenuator from the experiment plugin. So yes, you can easily do a fast scan with the attenuator activated.

Q: What about screening crystals of large molecules such as metal complexes, where twinning may be involved and unit cell determination is not straightforward?

A: You would use the same procedure. You might have to adjust your exposure time and/or width. Maybe 1s/1deg is a good start.

Q: While integrating the fast scan data for missing low angle diffraction, the Rint for fast scan is too high. So how we can incorporate the fast scan in routine data?

A: The fast scans can be processed in such a way that only reflections that are missing in the

"regular data collection" are used for the final hkl file. This is done during scaling. Because the weaker, less well-determined reflections are discarded, this will lower Rint for the fast scan.

Q: On integration, I wondered if you had a word/opinion on wide frame vs narrow frame algorithm for integration. When is it better to use which?

A: We start using the wide scan option for widths larger than 1.5 deg. But really, there is not a huge difference.

Q: Are you able to upgrade an Apex 2 instrument to Photon yet?

A: Yes, we are offering upgrades, and plan on delivering a couple of upgrades by the end of the month.

Q: Can we do the quick scan with old style Mo sealed tube?

A: Yes, you can, and you should try it!

Q: If we cannot easily separate frames in reciprocal lattice, how can we resolve the twinning problem?

A: We provide a program called cell_now that can be used for indexing twins. Bruce and I did a webinar about twinning also. Check it out. Our webinars are available at: https://www.bruker.com/service/education-training/webinars/sc-xrd/sc-xrd-archive.html.

Q: With a dual source can you use fast scan Mo data to fill missing data from a full Cu scan?

A: Unfortunately, Mo and Cu data cannot be combined.

Q: Along the lines of larger unit cells and MOFs determining scan parameters for larger unit cells, would you stay at 2 deg scan width and just adjust exposure (Mo radiation), or just provide some general comments?

A: As stated above, we would probably start with 1s/1deg. If this doesn't give enough reflections, try 2s, and so on.

Q: For absolute structure determination of light atom structure with Cu, do you have any collection strategy recommendations / tips to get a good dataset with low uncertainty in the Flack parameter?

A: Collect with as much multiplicity as you can tolerate (it takes time to collect). Low temperature is a must. Let's say it is monoclinic: Use the strategy planner and determine a strategy using 2(Chiral) to 0.8A and choose a multiplicity of 1 (the default). Start that data collection. Now go back to the strategy planner and determine a strategy using 1(chiral) to 0.8 and choose a higher multiplicity, like 3. Maybe also choose longer exposure times. Append that strategy and click resume. Now you can periodically check how good your Flack parameter is with the data you have already collected. When you are happy, stop the data collection.

Q: How do we make sure that the reticle center is the actual center?

A: This really depends on the instrument. Some require adjusting the video microscope and some can be adjusted in the software. Either consult a manual or contact customer service.

Q: I am running a Duo with an APEX II detector. Uncorrelated collection takes an extra 2-sec/frame, correlated 5-sec/frame above whatever the exposure time is. This can more than double the time needed for the experiment. How can I speed this up?
A: Unfortunately CCD detectors cannot be run shutterless. The shutter needs to be closed to be able to read them out. There is nothing you can do to speed this up.

Q: Will you go over how to replace the "topped" reflections during the scaling process? How intense do the reflections need to be from the fast scan to be able to replace those topped/overly intense reflections from a full data collection?
A: I think we did show this during scaling of the Cu dataset.

Q: Where/how did you open fast scan page?
A: The fast scan is available in the experiment plugin from the pulldown menu when you select a scan type. Matrix collection can also be set up to do a fast scan, instead of the traditional three orthogonal sets of thin data slices.

Q: I have 15 runs for high resolution data (0.45 A) and let's say in 5 runs it is diffracting only to 0.8 A, so how to integrate those runs and finally add with the other 10 high resolution runs?
A: You can integrate all together or you can integrate them in groups to different resolution limits and then combine individual runs in the scaling plugin.

Q: How short can your exposure time get? Is this depending on the instrument?
A: It is 0.5 second for PHOTON II, PHOTON III and APEX II, and 1s for PHOTON 100.

Q: Is APEX3 v2018.1-0 available publicly?
A: Registered users can download it from www.brukersupport.com. Demo licenses are available from your salesperson.

Q: I obtained data for a crystal that gave a clean reciprocal lattice. When one 360-degree scan was performed the Rint was 3.57 and the I/sigma was 80.7. When the number of scans was increased, the Rint= 9.3 and I/sigma was 36.7. Is this normal?
A: Yes, this is normal. The Rint will increase if you merge more data. Also, it looks like you are adding weaker (higher resolution) data to your 360-deg scan (judging from the I/sigma(I)) which would also increase the Rint (counting statistics).

Q: I would like to ask you about xprep and if this is an obligatory step in solution's procedure.
A: It is good practice to use XPREP, but not necessary if you use intrinsic phasing (XT) for structure solution. XT requires the Laue class and will determine the space group from the phases.

Q: I think fast scan will work only on very good crystals or organic molecule. Am I right? Will it work with coordination polymers or MOFs?
A: Yes, it does. It might require longer exposure times.

Q: Is there any advantage to choosing phi and omega scan technique over only omega scan?
A: It is more efficient to combine the two to generate complete and redundant data sets.

Q: You skipped XPREP every time, will it work every time?
A: It is good practice to use XPREP, but not necessary if you use intrinsic phasing (XT) for structure solution. XT requires the Laue class and will determine the space group from the phases.

Q: What is correlated vs uncorrelated scan?
A: For longer exposure times CCD detectors collect a 30-sec exposure as two 15s frames. The two frames were then compared to remove spurious events like zingers from cosmic rays.

Q: Why do you not change the point group in SCALE (or did I miss the explanation)?
A: Even for chiral compounds you typically scale in the centrosymmetric Laue class.

Q: For the shutterless, how to choose when to use shutterless vs normal mode?
A: Shutterless is the new normal. ☺

Q: Is there any way to predict which type of twinning is present while processing the data?
A: The easiest way to check for non-merohedral twins (where not all reflections overlap) is to look at the reflection array in the reciprocal space viewer. There are other indicators and I would suggest that you watch one of our webinars about twinning.

Q: Can you quickly review the reciprocal lattice viewer to deconvolute a twin?
A: We did in the beginning of the presentation. This is shown in depth in our twinning webinar: https://www.bruker.com/service/education-training/webinars/sc-xrd/sc-xrd-archive.html.

Q: When you are 'cleaning up' the reciprocal space view, does this just remove from unit cell indexing or does it remove those reflections from the dataset?
A: Using one domain only will “remove” all the reflections that do not overlap. In many cases this gives an okay result. Treating it as a multiple domain compound is typically better.

Q: Hello! Thank you for the broadcast. One of the students once had a crystal with a very large unit cell parameter and the reflections overlapped to a certain degree. The integration looked well. But the structure won't solve. The crystal is gone now (decomposed). What settings for integration would you suggest? Can another program (other than SAINT) be used to tackle this?
A: Unfortunately this could be many things. You can always contact us directly and we will do our best to help you out.

Q: Can we download this webinar video?
A: We will send a link to the recording to all registrants. Webinars are also accessible through the Bruker website at: https://www.bruker.com/service/education-training/webinars/sc-xrd.html