Notes from the Chat on May 22, 2020

Keep in mind that Shelx/Olex/Crystals have no idea about chemistry. It doesn't know that it was a BF2. So it may choose to make an atom a carbon even though it should be a B. So you can always rename an atom as something else.

Louise: In reviewing the following, please review the SDS of any solvent that you might consider using, especially benzene.

Paul maintains a list of crystal growing guides here: http://xray.chem.uwo.ca/Guides.html

What about if we're only using benzene within the glovebox? I very often layer when growing and use benzene as my 'poor solvent'

make sure you're taking precautions on bringing out benzene waste out of boxes...straight into a fumehood and aired out for a while

As the group's crystallographer I smell pretty every solvent when transferring to the microscope so avoiding benzene at all stages is great

Trouble with cyclohexane and benzene is you can't put them in the freezer to recrystallize

Any recommendations for a less poison 'poor' solvent to use?

toluene

Hannah sometimes chlorinated solvents make great crystallization solvents but unfortunately not compatible with glove boxes

What do you suggest is an appropriate amount of material to use in a crystallization if we're making fairly low yields of about 50mg?

50 mg is a good number, but only for one crystallization attempt

I've had 1 mg in about 1 ml with good results

Hexane is way better than hexaneS. At least if it shows up in a Lattice e you know exactly what thew molecule is

Pentane is probably my favorite solvent for compounds I work with, but anything polar makes it relatively useless

You could really scale your crystallization experiments where I've only had a like 10 needles in the entire vessel but the crystals were good quality. The amount you need really depends on how well it crystallizes. You may get 99% bad crystals in a batch but that 1% will allow you to get a structure. Statistically you need to increase the mass to increase that 1% if you know what I mean

Slow cooling favors larger scale as well

could you expand on dewar use for slow cooling (like using liquid Nitrogen?) if that is the case is there a danger that crystals could dissolve when lifted out of liquid-N?

I think it's dewar to keep things from cooling down too quickly. Like putting it in a thermos

Or wrapping it in Styrofoam

Be careful leaving vials with solvents in glovebox freezer. make sure they are closed tightly. The labels of all vials in the freezer could be washed off.

Parafilm dissolves in lots of solvents!

For small vials or nmr tubes you can take a block of styrofoam and stick the nmr tubes into small holes you poked with a pencil

Andreas, styrofoam on top?

Especially with vials you cannot put them in the fridge by themselves. They will cool down too quickly. You can put them into a styrofoam container or into a block that has holes in it to fit the vial. That way they cannot flip over as well

I find that NMR tubes lose their solvent slow enough to grow crystals with some tender loving neglect

I found the polished surface of NMR tubes provided an excellent for nucleation.

Yea vapour transport makes very clean crystals in my experience. Usually just not he crystals I was trying to make

we used to slow evaporate by taking the vial caps off in the wetbox and check on it a week later...once it was discovered the box filter was a lovely shade of dark purple we put a stop to that :)

PLATON: Another Canadian shout-out: ADDSYM was written by Yvon LePage, NRC, Ottawa.

There are great tools for preparing Acta Cryst manuscripts here:
https://journals.iucr.org/services/wordstyle.html
I also use these templates for making nice tables for other publications and theses.

This is enCIFer. It is a free tool from the CCDC, which you would have gotten with your CSD software download. It is invaluable for handling syntax errors in your .cif:
https://www.ccdc.cam.ac.uk/Community/csd-community/encifer/

ShredCIF is among the shelx executables that you downloaded, to extract the various components of a .cif.

OLEX2 will also offer to extract the .res and .hkl for any modern cif that contains that information, if you open your .cif in OLEX.

This is actually a great way to generate teaching examples, for anyone who is looking for examples for a class.

If you use Encifer, you can select and item and hit on the “book w the questions mark” to get the swarm info

I just emailed instructions from Amy to launch shredCIF from within OLEX2.
Joe also checked with Horst (OLEXSYS), who says "If you want to use ShredCif, just make sure it's on the

path (which it probably will be, if you installed ShelX in the recommended way), then you can type 'shell' and then 'shredcif filename.cif'."

For checkCIFvrf replies in .cif: where in the cif do you paste it in, does it matter?

Usually at the end to ensure you do not break a “loop” or a set section already there.

If I ran CheckCIF in PLATON, should I worry that some updates to the tests have occurred that PLATON wouldn’t contain?

not sure, the online version is always updated and works great

If you look in the CIF dictionary, there are a few entries _exptl_crystal_colour which can be used to characterize the colour. See: https://www.iucr.org/__data/iucr/cifdic_html/1/cif_core.dic/index.html and used your browser's search function to find 'colour'

For standard colours, see:
https://www.iucr.org/__data/iucr/cifdic_html/1/cif_core.dic/Iexptl_crystal_colour_primary.html

You can just Mercury to make publication quality graphics. Export as PNG. You may need to tweak the image size get a dpi value to get a publication quality image.

Mike Katz has been working on a Miller plane generator that is an excellent teaching tool:
http://katzresearchgroup.com/Miller.html

https://xtallography.ca/wp-content/uploads/2018/05/Olex2-Cheat-Sheet.pdf

This should fix your space group when using XT

-s"Name" space group (replace "/" by "_" e.g. -s"P2(1)_c") [off].

This is a .pdf with the chapter Amy is showing on twinning in shelx:
http://www.csb.yale.edu/userguides/datamanip/shelx/97/ch_6.pdf
The full manual in .pdf format: http://shelx.uni-goettingen.de/shelx97.pdf
All tutorials and talks: http://shelx.uni-goettingen.de/tutorials.php

And specific to twinning: http://shelx.uni-ac.gwdg.de/~rherbst/twin.html

This is where you can find documentation and resources for CSD products:
https://www.ccdc.cam.ac.uk/support-and-resources/CCDCResources/

An introduction to Mogul tutorial: https://www.ccdc.cam.ac.uk/support-and-resources/ccdcresources/HG-Mogul.pdf

The CCDC has educational resources here:
https://www.ccdc.cam.ac.uk/Community/educationalresources/

These resources include excellent "How To" videos!

Here's a definition of the +/- for torsion angles:

If the chain is viewed along the line BC, the torsion angle is positive if the bond AB would have to be rotated in a clockwise sense (less than 180°) to eclipse (i.e. align with) the bond CD. If the rotation of AB has to be in an anticlockwise sense, the torsion angle would be negative.

This is the CCDC on twitter: @ccdc_cambridge

https://www.ccdc.cam.ac.uk/Community/blog/2018-12-20-4th-annual-csd3dprint-contest-winner-announced/