## Using CrysAlisPro to process Saturn and Nonius Data sets

## Open CrysAlisPro

)pen CrysAlis experim	ent (1.0.41) - 11 experiments available		- 🗆 X		
Select exp	eriment - standard list		CrysAlis		
Name	Path	Created	Accessed		
FNU1 sh18034 tested_crystal_1 FNU-2a FNU-2a tested_crystal_1 sh18040 pre_sh18040 pre_sh18040 HQ-why-TPE-3 JHL_155_thianMn	E:/FNT-82018/SWTMedCtr/FNU1 E:/FNT-8-2018/Humphrey/sh18034P/sh18034 E:\4gilent-Yild-Test-Crystal-8-9-2018/tested_crystal_1 E:/FNT-8-2018/SWTMedCtr/Agilent-Data/FNU-2a\mages E:\FNT-8-2018/SWTMedCtr/Agilent-Data/FNU-2a\mages E:\/Yild-Test-Xtal-8-16-2018/tested_crystal_1 E:\FNT-8-2018/Humphrey\sh18040 E:\FNT-8-2018/Humphrey\sh18040 E:\FNT-8-2018\Humphrey\sh18040 E:\FNT-8-2018\Humphrey\sh18040-part E:\FNT-8-2018\Rose\UHL_155_thianMn\Images	Thu Aug 02 20:44:10 2018 Wed Aug 08 15:47:50 2018 Fri Aug 18 07:47:37 2017 Fri Aug 10 14:06:18 2018 Mon Aug 13 10:05:30 2018 Fri Aug 18 07:47:37 2017 Sun Aug 19 18:34:38 2018 Sun Aug 19 18:14:48 2018 Sun Aug 19 18:14:48 2018 Fri Jun 22 15:21:42 2018 Wed Aug 22 11:19:56 2018	Wed Aug 22 11:15:30 2018 Wed Aug 08 15:47:55 2018 Fri Aug 10 14:05:39 2018 Mon Aug 13 10:04:39 2018 Mon Aug 13 10:05:34 2018 Thu Aug 16 15:38:02 2018 Mon Aug 20 11:52:05 2018 Mon Aug 20 10:39:49 2018 Mon Aug 20 10:47:03 2018 Mon Aug 20 16:24:34 2018 Wed Aug 22 11:45:59 2018		
			Hide preview 🔽 Hide pre experiments ↓		
Hide screenings					
Help	Multiple addition Browse experime	ent Delete exp	eriment(s) from list Open selected		

Select an experiment from the list. It doesn't matter which one you choose.



Select the command icon

Dtrek createrunlist for Saturn data or saxi createrunlist for Nonius data followed by enter

Shell command window (Ctrl - interrupts)				
Command shell				
RED	Force auto scroll 📃 Transparent			
! SETUP WARNING: The image format currently used is unknown (2084x2084)! Goniometer type: KM4 (km4gonio.xdll) Switching to new KM4 collision model (Sapphire (large Be window), rail: Star Collision model: Xcalibur2, standard collimator, FIP31 camera Sapphire, Beam	ndard,1.1.20.) nstop standard			
<pre>FIP60 CCD INTERFACE Reading tabbin file: "E:\FNT-8-2018\Rose\JHL_155_thianMn\Images\JHL_155_thia UB - matrix:</pre>	anMn_peakhunt" 13 ) 14 ) 15 ) 01 ) 01 ) 01 )			
unit cell: 14.164(6) 15.530(5) 17.545(8) 67.88(4) 78.04(3) 77.02(3) V = 3452(2)	~			
Options RED	Close			

A menu will open where you select the data frames you want to use with the Browse button.

Run list and aliases file generator for DTREK data collections (1.0.2)	×			
This dialog allows you to quickly generate a *.run file and aliases file for the data reduction of a DTREK data set!				
<ol> <li>Select an image, by clicking 'Browse' button in group box First dc DTREK dc file, e.g. name1001.img</li> <li>Choose whether instrument model will be loaded from selected image header or selected par file</li> </ol>				
3. Terminator format is automatically set, but you can change it if necessary				
4. Also the last image will be automatically found, but you can change it if necessary (if it is not automatically found) Note: It is assumed that all frames between these two are available				
<sup>1</sup> 5. Save the file				
- You will be prompted for entering some critical parameters (usually default values are OK, as they are taken from image headers)				
- Finally a new CrysAlisPro instance will be launched with the DTREK data set added to the experiment list				
NOTE: Using CrysAlisPro you can process only DTREK images from selected Rigaku detectors! R-AXIS format is not supported!				
1. First dc DTREK dc file (*1001.img)				
Browse n/a				
2. Instrument model Use par file information instead of image header				
Browse n/a				
Par file information: n/a				
3. Terminator format	5			
Frun digits 🔽 0 Separator None 🔍 Frame digits 🖵 3				
nameFFF.img				
4. Last de DTREK de file				
Browse n/a				
Help Run TC Save run file				

Select Browse for the first image in the first frameset and Browse to select the last image in the last frameset. Using Saturn data, you can ignore the initial screen data. For Nonius data, select the first frameset even if it is the initial screening data. You can deselect frames later. Select

Save run file save run file to read in the data. It may take a few seconds to read in all the data. When successful, a window will appear with the header information for the detector.



Select OK.

A new experiment will be created and highlighted for the Saturn/Nonius data set. Open the selected dataset.

A new CrysAlisPro menu will appear and will show the first image of the Saturn data. One thing you will notice immediately is that the Saturn frames are noisier than the Agilent frames. Some of this is due to the Atlas detector and some due to the lack of dezingering of the Saturn images (usually).

Basically, you proceed as you would for any Agilent data set. Select the Lattice Wizard



icon to begin peak selection and unit cell finding. Select Peak hunting



followed by unit cell finding to get a

starting unit cell. Left click the > symbol by the Peak hunting icon and select 'Peak hunting with user settings'.

Peak hunting wizard (1.0.15)

Peak hunting	
Run list, image type and image directory Run list: E:\FNT-8-2018\Rose\JHL_155_thianMn\Images\JHL_155_thianMn	*.dtrek
Image dir: E:\FNT-8-2018\Rose\JHL_155_thianMn\Images	
# type start end width exposure omega detector kappa phi start end	
2 o -110.00 24.00 0.50 60.0020.00 -60.00 0.00 1, 268	
3 o -110.00 24.00 0.50 60.0020.00 -60.00 120.00 1, 268	
4 o -67.00 22.00 0.50 60.0020.00 -60.00 240.00 1, 178	
Run list modification	
By default the whole experiment will evaluated. Edit start num of selected run	lit end num of selected run
To modify this behaviour edit the run list>	
Automatic threshold and background     detection (preferred)     O Traditional peak hunting     O Smart peak	hunting O 3D peak extraction
Peak finding control Overwrite existing	peak hunting table
Threshold: 1000 7x7 average: 20 • Yes	O No
Use background subtraction	
Background evaluation control -> 50 Edit Re 50	Edit Fr
Binning for background evaluation:	2 • 4
Reduce background accumulation to SHORT type (saves memory)	
Resolution limits	
Skip peaks outside resolution limits d-value (Ang): inf- 0.68 2theta (deg): 0.00- 62.74	Edit res limits
Apply float correction n/a	
Remove spikes • weak • strong Help	Cancel OK

The frame data that was read in will be listed. I usually do two things at first. I will select one frameset and ignore the others when doing a peak search to save time. To bypass a frame set, select a frame set and then select 'Edit start num of selected run' and enter 0. Do this for all the frame sets you wish to bypass.

Secondly, I check the box 'Skip peaks outside resolution limits'. By default, CrysAlisPro will search the entire image which has resolution limits exceeding the useful range of the data scattering. Check the box and then select 'Edit res limits'.

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Parameter to enter:	d-value (Ang) 💌	
	Edit low limit	Edit high limit
d-value (Ang):	inf	0.681
Theta (deg):	0.000	31.372
2 Theta (deg):	0.000	62.745

I usually select both low and high limits. For low limits, I enter 10 for the d-value. For the high limit, I will typically select 0.9 or something like that. These values will vary with the crystal and the data. This is only for the peak search and unit cell determination. For data integration, I use all the frames and use at least 0.83 or 0.77 for the high resolution limit. To use these values, select OK to get back to the Peak hunting menu.

To begin the peak search, select OK in the Peak hunting menu. CrysAlisPro will go through the selected frames and generate a peak list just like CrystalClear. Because the frames are noisier, sometimes you have to work at getting a cell. If you have a good cell from CrystalClear and just want to take advantage of the superior data integration program in CrysAlisPro, you can enter the known cell in the 'Unit cell finding' menu. Left click the > symbol by the



icon, and select 'Unit cell finding with options'. At the bottom of the menu check the 'Known cell' box and enter the known unit cell.

Find cell	×			
Peak table Normal peak table Delta (differential) peak table Find center Use search box Edit Use previous algorithm	Algorithm T-vector Dirax Stereographic			
Sample type     Single crystal     Unit cell limits     min				
SM C PX C User 2.0      Twin / multicrystal	120.0 Calc			
# of components 2 2.0 Lock present components (see 'Twin information' section of Twin 1	120.0 Calc			
the Lattice Wizard): HINT: To lock current UB for twin 1, first go to UM TWIN utility and click 'Current UB to twin'. Then return here and select 'Twin 1' checkbox above.				
Consider Bravais lattice type Force identical lattice for all components (uncheck for multi-crystal)				
Known cell Search known cell 8.08 10.63 22.79 96.85 95.10 92.20				
	OK Cancel			

And select OK. The Lattice wizard menu will appear with the unit cell. Look carefully at the results and check to see if the known cell is the current cell. Also, check the Peak Table and look at the percentage of reflections that match the cell. In this case, the value is 72%, which is good. If this number is much lower, sometimes the cell parameters need a little tweaking to come into agreement. Select Refine instrument model to refine the cell and instrument parameters to improve the fit. Sometimes an initial fit of 10% will improve to 30% and then 50% and higher with additional refinements. And sometimes not. Check the instrument model values listed on the right side of the CrysAlisPro menu. The easiest number to check is the crystal to detector distance.

## Crystal RED EXPERIMENT FNU-2a **CHEMICAL FORMULA** C20H21N202C1 Z=4.0 Current cell (CSD: install) 8.0734(13) 10.624(2) 22.784(3) 96.859(14) 95.105(13) 92.195(14) V = 1930.2(5) Constrained coll LATTICE Current cell Constrained cell unknown 8.0734(13) 10.624(2) 22.784(3) 96.859(14) 95.105(13) 92.195(14) V = 1930.2(5) Symmetry Laue class: 1bar P-lattice AVERAGE UNIT CELL FROM PROFFIT Constrained cell (1122 obs) 8.0728(7) 10.6160(9) 22.776(2) 96.869(7) 95.070(7) 92.162(7) V = 1928.0(3) PEAK TABLE UB fit with 1022 obs out of 1418 (total:1418,skipped:0) (72.07%) **INSTRUMENT MODEL** X-ray wavelength: Mo x-cen: 1009.9056 y-cen: 1052.1517 distance: 44.9868 beam: -0.3243

For the Saturn, the default distance is 45.0, in the menu above it is 44.9868 mm. If the number is significantly different from the distance set for data collection, you will have to start over with the original values. The only way I know how to do this is to delete the file, filename\_cracker.par, which contains the current information for the experiment and where filename is the name of the data set. Then copy filename.par to filename\_cracker.par. These file will be located in the Image directory.

At this point, you have to reselect the experiment

and start over.



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Lattice wizard (1.0.35)

Lattice wizard		
LATTICE Current cell (CSD: install) 8.0763(13) 10.627(2) 22.792(3) 96.854(14) 95.100(13) 92.196(14) 1932.1(5) Lattice reduction	Peak hunting	Unit cell finding
selected cell 8.0763 10.6271 22.7920 96.8539 95.0998 92.1956 aP 44 reduced cell 8.0763 10.6271 22.7920 96.8539 95.0998 92.1956 1932.1	Ewald explorer - reciprocal space	Reindexation with current cell
PEAK TABLE Peak hunting table UB fit with 1022 obs out of 1418 (total:1418,skipped:0) (72.07%) INSTRUMENT MODEL	Refine instrument model	Lattice transformation
Goniometer beam: -0.31826 alpha: 50.00000 beta: 0.00000 om zero: 0.00877 th zero: -0.04382 ka zero: 0.00000 Detector x-rot: -0.04081 y-rot: 0.01944 x-cen: 1009.87941 y-cen: 1052.12532 distance: 45.00772 Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229	Twinning - multi-crystals	Incommensurates / Quasi-crystals
Refinement res: 0.010487, da=0.008042, sx=0.000879, sy=0.001493 h=0.004302, k=0.003651, l=0.003235 #ref: 1018 (skipped: 2)	Load information	Save information
	Ewald3D	Unwarping - Precession images
	Log window	Close

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