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INTRODUCTION

Use of this instrument is allowed only by qualified users after receiving training by a staff member. Do not run this instrument without approval from IMSERC staff. Failure to do so may cause damage to the instrument, produce invalid data, and result in additional fees and/or removal of all IMSERC privileges. This set of instructions is meant to serve as a guide for 'routine' data collection on the instrument. For custom experiments that are not covered in this user manual, contact a staff member. For the full list of modes, capabilities, and potential custom experiments that could be run on this instrument, please either contact a staff member or check the corresponding capabilities section at http://imserc.northwestern.edu/crystallography-instruments.html. Please read this user manual and acquaint yourself with the instrument.

A hard copy of this user manual can be found near the instrument. An electronic version of this user manual is linked to the desktop of the instrument computer and also available under the corresponding instrument section at http://imserc.northwestern.edu/crystallography-instruments.html by pressing on the 'User manual' button. If while using the system, something happens that you do not understand, please stop, and get help. In any event, be completely prepared to justify your actions. The cost of even minor repairs could be considerable.

Please remember to:

- 1. Leave the acquisition software open when you are done with the measurement
- End your reservation from NUcore when you are done with the experiment 2.
- 3. Leave lab tables clean and tools/accessories organized
- Report problems with the instrument (see troubleshooting section for more details) 4.

SAFETY

All users of IMSERC must review the general safety policies at http://imserc.northwestern.edu/aboutpolicies.html and the Crystallography specific policies at http://imserc.northwestern.edu/crystallographypolicies.html. To become an independent user of this instrument, you must have the following safety training and certificates under your LUMEN profile:

- Laboratory Safety
- Personal Protective Equipment
- X-Ray Safety



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You need the above certificates to be able to reserve time for this instrument on NUcore. Online classes and certification are offered at https://learn.northwestern.edu. Upon completion of the certificate, it will take an overnight to filter through the different systems and get into the files that NUcore uses. Additionally, familiarize yourself with the location of standard safety stations like eye wash and shower stations found in outside of room BG70. Protective eyewear is required in this room, and gloves should be removed when using the computer.

DATA MANAGEMENT

Your personal data folder is created during training. Please save data under your personal folder, which must be located under your supervisor's group folder, either wise you might not be able to access your data remotely. See a staff member if you do not have a personal folder on this instrument yet. For users that prefer to name their data folders using dates, use the order of YYYY-MM-DD or YYYYMMDD in the name, so that folders can be sorted chronologically by the operating system if needed.

Data from this instrument are copied in your supervisor's group folder on 'imsercdata.northwestern.edu' under 'xrd/X-Synergy' (where X is specific to the model, i.e., Cu, Mo, or DW) every 15 minutes. Please follow instructions at http://imserc.northwestern.edu/about-general-fag.html#data for details about data access.

SOFTWARE

Data reduction and analysis are performed with the 'CrysAlisPro' software package. Software is installed on the instrument computer. For offline analysis after your instrument reservation is complete, please use the following resources:

- For registered IMSERC users, software can be downloaded from 'imsercdata.northwestern.edu' under the folder 'public/Crystallography/CrysAlisPro'. Software is available for Windows only
- Software is installed on the communal computers located in the area outside room BG51 and in the computer lab room B190
- You have the option to use the instrument computer for analyses, but you must reserve instrument time through NUcore

For a detailed list of all available crystallographic software for IMSERC-registered users, please check at http://imserc.northwestern.edu/crystallography-resources.html#software.



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DEFAULT INSTRUMENT STATUS

In describing the steps involved in collecting a data set, several assumptions have been made regarding the status and conditions of the instrument. The default working condition of the diffractometer is listed below. Control panel is located at the bottom right side of the diffractometer

- 1. The HV ENABLE key should be "Enabled" (figure 1)
- 2. The following lights should be illuminated
 - a. Green ON (figure 2a)
 - b. HV ON/Start. If HV OFF is illuminated, press HV ON and wait ~1 min for the interlock status to update (figure 2b)
 - c. Shutters: Red (open) or Green (closed) are both OK (figure 2c)
 - d. Error = no light (figure 2d)
 - e. Doors OK: Red or none are both OK (figure 2e)

When you are done with your measurement, please remember to:

- 1. Leave the acquisition software open when you are done with the measurement
- 2. End your reservation from NUcore when you are done with the experiment
- 3. Leave lab tables clean and tools/accessories organized
- 4. Report problems with the instrument (see troubleshooting section for more details)

If there is an error or problem with the instrument which is not addressed under the troubleshooting section, please report the issue by following at least one of the steps below:

- 1. If you have already started your reservation using NUcore, please end your reservation and select the error reporting option with a brief description about the issue. Place the 'Stop' sign near the instrument computer to notify users immediately after you. 'Stop' signs are located on the shelf above the computers in BG51
- 2. If you have not started your reservation using NUcore, please report problems with the instrument at http://imserc.northwestern.edu/contact-issue.html and place the 'Stop' sign near the instrument computer
- 3. Contact a staff member for instructions





CRYSTAL MOUNTING UNDER A MICROSCOPE

This standard operating procedure is meant for training students/postdocs with the microscopes available at IMSERC. Do not run these microscopes without this training or approval from IMSERC staff. Failure to do so may cause damage to the instrument and result in additional fees and/or removal of all IMSERC privileges. This short set of instructions is meant to serve as a guide for 'routine' usage on the instrument.

A. OUR MICROSCOPES

IMSERC maintains two high resolution, polarized light microscopes available for student use in the Crystallography facility. Our Nikon SMZ1500 stereo-zoom microscope is equipped with a digital camera and video monitor for visualization of crystalline samples. Users can perform visual inspection of their samples with these instruments to assess crystal quality. High resolution photographs can be taken and used for publications or other presentations.





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B. TURNING ON THE MICROSCOPE AND ILLUMINATOR

Both microscopes have a timer by the back wall for both the underneath light and the goose neck lights. This timer lasts for 30 minutes.

C. IDENTIFYING A GOOD CRYSTAL AND PREPARATION

Crystals come in all shapes, sizes, colors and transparent, translucent, and opaque, air sensitive, solvent loss issues or stable. You will get used the types of crystals you usually grow and what tools and mounts you will usually need.

An optically 'good' crystal should:

- Extinguishes plane-polarized light
- Uniform color if does not extinguish light
- Smooth surfaces and sharp edges
- **Regular shape**
- Free of defects
- On rotation will go from light to dark uniformly
- For twin crystals, Cut with a razor blade if possible



You will learn to choose the types of tools that has the best fit for you

- Ease of use
- Matched to sample
- Pipette if needed in solvent
- Smaller tools for small specimens
- Slide for most mounting
- Watch glass if mounting solely from mother liquor
- Razor blade for cutting crystals









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Cut crystals if bigger than 0.5 mm

- Move crystal to open space on slide in oil .
- Use razor blade to make cut
- Brace tip of razor on slide to reduce motion
- Gently and smoothly press blade down to cut
- Crystal cleaves cleanly

By sweeping crystal through oil, small crystallites and other debris can be separated from the crystal. Do not crush, crystals can shatter









D. MOUNTING CRYSTALS ON AN APPROPRIATE MOUNT WITH ADHESIVE

Low temperature crystallography allows for easy handling of routine and air sensitive samples. The material you use to mount the crystal must harden at experimental temperature. Following are the adhesives depending on the temperature of data collection.

- Suitable compounds
 - Paratone-N 0
 - Grease 0
 - High-vacuum grease 0
 - Hydrocarbon oil 0
 - STP engine additive 0
 - Apiezon 0

For room temperature mounting you can glue your sample onto the glass fiber:

- Suitable adhesives
 - 0 Ероху
 - Cyanoacrylate (Super Glue) Ο
 - White glue 0
 - Rubber cement 0

Choosing a mount:

- Cryoloop
 - Typically mounted on end of tapered copper pin 0
 - Place crystal at center of 0
 - Affix with oil 0
 - Suspend in mother liquor 0
- Glass fiber
 - Inexpensive 0
 - Pulled from capillary on capillary puller 0
 - Some background scatters 0
 - Cut with stone Ο
 - Mount to copper pin with bee's wax 0



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- Affix crystal with small amount of grease or glue at end of fiber 0
- **MiTeGen** mounts
 - Almost no background 0
 - Easy to use 0
 - Affix with small amount of oil 0
 - Many different styles and sizes Ο



Mounting crystals:

- Select specimen to be mounted 1.
- Move to clean part of slide or edge of oil 2.
- 3. Slide under crystal with mount
- Push against crystal when using glass fiber 4.
- Center crystal on middle of loop or top of mount 5.
- Pick up crystal with minimum amount of adhesive 6.
 - a. Hard to see to center crystal
 - b. Hard to index faces
 - Crystal may slide in oil c.
 - d. Creates background amorphous scattering







STARTING A NEW MEASUREMENT

A. SETTING PARAMETERS FOR EXPERIMENT

- 1. Verify that the instrument is at a working state (see 'Default instrument status' session above)
- 2. In case the acquisition software (CrysAlisPro) is not running, double-click on the CrysAlisPro icon control on the desktop. The software will go through an initialization procedure, which takes a few seconds. Status of the initialization procedure is visible at the top right side of the CrysAlisPro window
- 3. Control menus of the various components, such as, low temperature attachment ('CRYO', figure 4b), X-ray source ('X-RAY', figure 4c), and lights ('STATUS', figure 4d) in the enclosure can be accessed by pressing the corresponding buttons at the top right corner of CrysAlisPro
- 4. Before starting the screening process of your crystal, select the appropriate settings:
 - When you are using the dual-wavelength system (DW-Synergy),
 press on the corresponding button (figure 4a) to change the

START/STOP Shutter Closed CAM CRYO X-RAY STATUS CCD Ready 4b 4c 4d

wavelength if needed. The Cu- and Mo-synergy systems do not have this option/button since they are single source systems

- b. Set collection temperature by pressing on the CRYO button (figure 4b)
 - Press on the 'Set' button (figure 4b-i) to change temperature. If button is not clickable, press on the 'Restart' button (label of the button toggles between 'Shut down' and 'Restart' depending on the state of the 'Set' button) and wait for a few seconds until the 'Set' button is activated





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At the set dialog window, depending on the current state of the cryostat and your target temperature, ii. ryostream 700 set dialog select (figure 4b-ii):

Mode operation

G

Plat Cool

C Purge C End

Information

Read values for active Mo far/offset lamp

Voltage [kV]

Set values

\ Status: De

20.0

Voltage [kV]

Current [mA]

Ramp rate [K/h] 360.00

Ramp at given rate to the specified temperature

Ramp Cryostream 700 at given rate to the specified temperture

Ramp specified time to specified temperature

Hold at current temperature indefinitely

Final temperature [K] 100.0

Set turbo mode on

Current [mA]

0.05

- 'Ramp at a given rate to the specific temperature' when you want to use a specific heating or cooling rate. Maximum rate is 360 K/h
- 'Cool' when you want to cool down as fast as possible without controlling the rate
- 'End' when you want to turn off the cryostat. Do not use the 'Stop' button on the controller of the Cryostream unless there is an emergency
- OK c. Current power settings of the X-ray source are shown at the top right corner in the diffraction frame window. If settings are different than 50 kV, and 1 mA, press on the X-RAY button (figure 4c)
 - i. On the generator window, press on the 'Set kV, mA, X-ray' button (figure 4c-i)
 - At the set dialog window, select 'Auto-ramp' ii. and press 'OK'
 - Wait until the power ramps up to 50kV and 1 iii. mA and then close the generator window
- d. Press on the STATUS button (figure 4d) in case you want to adjust the intensity of the light in the enclosure and/or the intensity of the light for the optical camera used for aligning the sample. Light in the enclosure will turn on when the main door of the enclosure is opened
- 5. To start a new experiment, press on the START/STOP button in the top right-hand corner (above the CRYO, X-RAY, and STATUS buttons). This will open up a new window. Press on 'Start New' option
- 6. The SM (Small Molecule) Screening window will now show up on the right-hand side. It is worth noting that if this is not a new instance of CrysAlisPro (i.e., you are working on a data set after someone else has been using the instrument and the software is still open), then you may see some unit cell information in the Screening section. Do not worry, once you start collecting your data, this will Experiment - Complete data for publication reflect your crystal
- 7. Set your data collection folder and information about your sample by pressing on the 'Edit' button at the bottom right hand corner of the SM Screening window (figure 7)



IS xgen flexgen 1.1.0.83 Final_rev656_PCB1

Power IW1

1.00

Ramping time [min] 632.0

4b-ii

Cancel

Filament current [A]

1.92







Rigaku Oxford Diffraction fast screening options (1.1.1)	×
Pre-experiment	CRYSALIS
Path and user / Sample Name: exp_101 Ex 7C cp_101 in folder D:\IMSERC\exp_101 Path is ok! Browse root folder >> 7b	Experiment performer: IMSERC
Expected chemical formula: AutoChem4 may not succeed without providing valid chemical formula!	Get Last used formul
Comment:	Sample description

- a. Press on the 'Set user' button (figure 7a) and select your group folder by selecting the name of your group and then selecting the option 'Set user'. Your group name should appear above the 'Set user' button and in the 'Experiment performer' label. In case your group folder does not exist in the list of groups, please talk to a staff to create the folder for you
- b. Press on the 'Browse root folder' button (figure 7b) and select your personal folder, e.g., 'D:\SupervisorLastname\LastName-FirstName'
- c. In the 'Name' text box (figure 7c), provide your experiment name. This experiment name will be also the name of the subfolder under your person folder
- d. Software will prompt for changing the default folder to your personal and project folder. Press on the 'No' button (figure 7d) since all other group members will not be saving under your personal folder

Change of	f default experiment folder	\times
×	Do you want to update experimental root for all future experiments?	k
	[7d 】 <u>№</u>	



B. MOUNTING YOUR CRYSTAL ON THE GONIOMETER

1. In the SM Screening panel, press on either the 'Mount' button (figure 1) or 'F12' on the keyboard. The crystal video window will open and the goniometer will move to the last mounting position. If the

goniometer does not move, then ensure that either the door lock button is 'enabled' or you are holding down the 'motion enabled' buttons located inside the enclosure. When the door lock button is 'disabled', anytime you need to move all except the phi axis, i.e., either the omega, 2theta, or chi axes, you need to hold down on both the 'motion enabled' buttons. This is an extra safety feature, so that your fingers and/or hand are not near the moving parts of the instrument

SM Screening

Mount

n = 5.0s >

Screening

2. On the crystal video window, press on the 'HOME' button (figure 2) once all motion stops (all buttons will be blue again) and open the door of the enclosure



- Mount the sample pin on the goniometer a.
- b. Using the monitor and mouse in the enclosure, press on the 'Lower' button (figure 2b) to adjust the left/right direction. Hold down on both the 'motion enabled' buttons to position the goniometer. Toggle between '0° Arrow Down' and '180° Arrow Up' button to adjust the X-direction
- c. Center the crystal such that it rotates about its center of mass. This is done by adjusting the adjustment screw pointing on the left or right as you face the mounted goniometer head
- d. In case you need to adjust the light intensity on the crystal, press on the 'Lights' button (figure 2d) at the bottom right corner of the video window
- Toggle between '0° Arrow Down' and '180° Arrow Up' button to adjust the up/down direction (X-direction) e.
- Finally, toggle between 'Upper' and 'Lower' to verify the Z-height f.
- Press on 'Home', so the instrument goes back to home at the end of data collection g.
- Exit the Crystal video window h.
- 3. Close the door of the enclosure and press the "Door OK" on the Safety System Control Panel







C. SCREENING YOUR CRYSTAL

- 1. You are ready to start the screening process, which is divided into two steps. First step is called 'Screen' and the second step is called 'Pre-Exp'. At the first step, you should be able to evaluate/screen crystal quality and depending on the number of reflections collected, obtain a unit cell. At the second step, you will collect more data for CrysAlisPro to calculate an accurate strategy
 - a. Press on the ">" button (figure 1a) just next to the 'Screen' button and select the exposure time that you feel would be appropriate for your sample. Last used exposure time is shown in the 'Screen' button as 'Screen = X.Xs'



- b. CrysAlisPro will collect a set of 20 images and try to determine a unit cell
- c. If a unit cell is found, a search against the Cambridge Structural Database (CSD) is run and possible hits are available through the CSD link next to the 'UNIT CELL' caption (figure 1c)



- d. A percent of reflections that fit the suggested unit cell is shown on the right panel along with intensity statistics for the exposure time used
- e. You need to press START/STOP and 'Start New' every time you mount a new sample
- f. Note that the screening frames are not stored in your directory, but frames are saved in a temporary folder that gets deleted before every screening collection
- 2. The 'Pre-exp' option (figure 2) will collect 30 images (Mo-radiation) or 60 images (Cu-radiation) and the software will try to determine how well the crystal diffracts and calculate a data strategy. The overall collection time of the pre-experiment will appear in parentheses on the button, e.g., 3 minutes as shown in figure 2. The minimum setting you need for the pre-experiment is the exposure time per frame:

-Experiment - Complete data for publication
Name: exp_7
User=Katz, Detector=34.0mm, Res. = 0.840Ang, I/sig.=10.0, width=0.5deg, Movie, cryo off, Strategy Complete data (default mode), Exposure: 2.0s 8.0s
Exposure time: 2.0 s
What is this? Pre-Exp. (3 m) Edit
Goniometer
Omega Theta Kappa Z i Dista 4 -0.0 0.0 -0.1 0.0 00.4





- a. A recommended exposure time for the 'Pre-Exp' will be estimated from the 'Screen' step and pre-selected for you. Exposure time appears as a label of the 'Exposure time' button (figure 3)
- b. (Optional) Adjust exposure time for the 'Pre-Exp' by either sliding the bar just above the 'Pre-Exp' button or pressing on the small button on the right of the sliding bar (figure 3). Exposure time depends on the size and quality of your crystal but in general:
 - Good scattering crystal: 1 s
 - Moderate scattering crystal: 5 s
 - Weakly scattering crystal: 30 s
- 3. (Optional) You have the option to re-edit crystal information (as described under the 'A. Setting parameters for experiment' section) by pressing on the 'Edit' button (figure 4)
 - a. Leave the 'Interactive strategy after pre' option checked for launching the strategy method right after the pre-experiment
 - b. Press on 'Exit & Start pre-experiment' and the software will start the pre-experiment collection
- 4. If you have skipped the optional step above, press on the 'Pre-Exp' button to start the pre-experiment measurement
- 5. As the images appear on the screen
 - a. Analyze spots for quality. Spots should be single and circular. If multiple spots are clumped together or the spots are very broad in one dimension, stop the pre-experiment by pressing on the 'START/STOP' button and selecting the 'Stop All' option, and screen more crystals
 - a. Turn on the resolution rings in case there are not visible by pressing on the 🙆 button under the frame window. Spots should be visible to around 0.80 Å resolution. If spots are not visible in this range, either re-screen with a longer exposure time or screen another crystal





D. COLLECTION STRATEGY

After the pre-exp ends, the 'Experiment Strategy' window will open if a unit cell has been found. A list of options and parameters are available for optimizing the collection strategy:

* Strategy (1.3.1), automode suggests exposure time t=4.5sec, scan width: 0.50 deg		×
Experiment Strategy		CRYSALIS ^{PRO}
Unit cell for Strategy Calculation - (CSD: install)		
Cell: 18.287(7) 18.298(0) 13.873(5) 90.00(3) 89.95(3) 120.02(4) 4020(3) 11K	R(obv)-lattice 91.47% (118 of 129 reflections)	Lattice Wizard
Strategy parameters	Time prediction based on data to 0.800 Ang exp time	individual merged
Resolution C Theta C 2Theta 0.800	Fill time Fill I/sigma	I/sigma: I/sigma:
● Laue group ○ Other -3 (hex-c)	The same time for all theta positions	15.00 29.54
Friedel mates are equivalent (uncheck for high quality absolute configuration data)	Different time for each theta positions	I/sigma: I/sigma:
• Thederinates are equivalent (analies to high quality absolute configuration data)	[-4.95; 5.34] 4.51	15.00 29.54
Detector Distance 34.00 Advanced	Scan width: 0.50	15.00 29.54
Strategy mode 🔽 Use run list sorting with scan inversion	Scanwiden: Jorso	
Complete data (default mode)		
limit 100.0 IUCr limit Max 99.96 %		
Generates runs that reach	Automatic experiment settings Optic	ons
completeness limit	Mode: 31bit Frequency: 0.44Hz	tochem/Movie/Cryo/Red
Current Strategy		
Total experiment time: 0h 22m	29Y Manually Edit Run List	
Expected experiment finish time: Thu Mar 21 09:45:13 2019 Update Completenes	s	
Completeness/Coverage curves Completeness/Coverage tables Completeness/Predicted resoluti	on	
Completeness in -3	Full sphere (P1)	
3.3 7 111 1 2	^	↑ -
	100 -	<u>e</u>
8 80 - 2.8 9	80 -	- 1.8
	60	- 1.6
		49 v fo
	40 -	- 1.4
	20 -	1 1.2
-1.2		
		240 280
Frame	Frame	
	Help Start named evneriment Start evneri	nent Cancel
	terp start named experiment	Cancer

- 1. Cell constants will appear at the top left corner of the strategy window. If there are any hits in the CSD that match these cell constants, you will be able to see these reported structured by pressing on the corresponding link. Select the lattice type that you want to collect under
- 2. 'Resolution': The higher the resolution the better. Typically, 0.84 Å⁻¹ is the lowest publishable value for samples without getting any alerts during checkCIF and publication. However, that does not mean that you should just collect to that resolution (for Mo-radiation typically to 0.70). If your sample diffracts well, then go higher. If your sample does not diffract well, then only collect it to where it needs to be collected. Inspect the images to see how well it collects the data. In case is needed, collect extra frames at high angles by taking

single exposures using the



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- 3. 'Laue group': If you are sure that the unit cell is correct and the point group is correct, then leave this unchanged. If you are worried that the actual sample will be of lower symmetry, then select other and lower the symmetry
- 4. 'Friedel mates': if you think the molecule is non-centrosymmetric (chiral or polar), then uncheck this option
- 5. 'Detector Distance': Minimum detector distance is 34 mm. If strategy gives a different value, accept the suggested value
- 6. 'Strategy mode': Typically use the 'Complete data' option for giving higher priority on completeness. Select one of the other options in case you want to optimize the strategy using additional priorities
- 7. 'Time prediction': Adjust the exposure time to achieve an estimated merged I/sigma greater than 15. As a rule, the ratio of the low and high angle exposure times should be about 1:4
- 8. Press on the 'Calculate New Strategy' button to (re-)calculate the list of runs that meet the criteria set above.
- 9. Adjust these values so the data collection ends at a reasonable time if necessary
- 10. Once you have finalized the strategy, press on the 'Start named experiment'. Type in the expected chemical formula and any comment you have about the sample in the corresponding fields
- 11. Press on the 'Sample Description' button and fill in the entries for Sample color and Sample Shape. Leave sample size as default values. Press on the 'OK' button
- 12. Once any of the start buttons is pressed, the software will take a movie of your crystal and automatically start the data collection





E. MEASURE CRYSTAL FACES AND REFINALIZE

If X-ray absorption is significant, a "face indexed" absorption correction must be performed. At minimum the size of the crystal must be provided for publication

1. Click Data Reduction and then

Inspect data reduction results



from the side bar. And then

Data reduction f	ile contents	Data ree	duction outp	ut	Red graphs	1	Data collection of	output	Devid	es log
1.14-1.04	674	238	235	98.7	2.9	1484.11	25.73	0.034	0.031	
L.04-0.96	621	236	235	99.6	2.6	1117.68	21.08	0.041	0.040	
0.96-0.90	525	243	235	96.7	2.2	796.22	15.73	0.049	0.050	
0.90-0.85	438	245	236	96.3	1.9	528.67	11.31	0.063	0.068	
0.85-0.82	405	249	235	94.4	1.7	437.49	9.54	0.072	0.085	
0.82-0.78	343	295	235	79.7	1.5	369.51	7.22	0.070	0.102	
0.78-0.70	256	897	235	26.2	1.1	255.87	4.77	0.087	0.145	
		3123	2351	75.2	2 2	2463 30	27.07	0.022	0 022	
inf-0.70	5137	0100	2001	13.3	6.6	2403.35	21.01	0.022	0.044	
inf-0.70 inf-0.80 Statistics resolu- tion(A)	5137 4709 vs resol # kept	2055 ution (tai # theory	1992 king redu # unique c	96.9 Indancy :	2.4 2.4 into account; average redundancy	2660.11 - Laue ç mean F2	29.02 group (anoma mean F2/sig(F2)	0.022 alous pa Rint	0.022 0.022 irs merged RsigmaB	l): Pmmm
inf-0.70 inf-0.80 Statistics resolu- tion(A) inf-1.73	5137 4709 vs resol # kept 514	2055 ution (ta) # theory 	1992 king redu # unique c 	96.9 96.9 indancy : * complete 	2.4 2.4 into account average redundancy 3.4	2660.11 - Laue (mean F2 10596.76	29.02 group (anoma mean F2/sig(F2) 	0.022 alous pa Rint 0.013	0.022 0.022 irs merged RsigmaB 0.010	l): Praman
inf-0.70 inf-0.80 Statistics resolu- tion(A) inf-1.73 1.73-1.33	5137 4709 vs resol # kept 514 664	2055 ution (tal # theory 151 150	1992 king redu # unique c 150 150	96.9 96.9 * * * * * * * * * * * * * *	2.4 2.4 into account; average redundancy 3.4 4.4	- Laue (mean F2 10596.76 3997.05	29.02 group (anome mean F2/sig(F2) 76.56 57.61	0.022 0.022 alous pa Rint 0.013 0.020	0.022 0.022 irs mergeo RsigmaB 0.010 0.015	l): Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 vs resol # kept 514 664 657	2055 ution (tai # theory 151 150 150	1992 king redu # unique c 150 150	96.9 indancy : * complete 99.3 100.0 100.0	2.4 2.4 into account average redundancy 3.4 4.4	- Laue (mean F2 10596.76 3997.05 2640.00	29.02 group (anoma mean F2/sig(F2) 76.56 57.61 44.50	0.022 alous pa Rint 0.013 0.020 0.029	0.022 0.022 irs merged RsigmaB 0.010 0.015 0.020	l): Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 vs resol # kept 514 664 657 726	2055 ution (tai # theory 151 150 150	1992 king redu # unique c 150 150 150	96.9 indancy : * complete 99.3 100.0 100.0 100.0	2.4 2.4 into account; average redundancy 3.4 4.4 4.4 4.8	- Laue (mean F2 10596.76 3997.05 2640.00 1529.26	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40	0.022 alous pa Rint 0.013 0.020 0.029 0.038	0.022 0.022 irs mergeo RsigmaB 0.010 0.015 0.020 0.025	l): Pranam
<pre>inf-0.70 inf-0.80 Statistics resolu- tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95</pre>	5137 4709 vs resol # kept 514 664 657 726 662	2055 ution (tai # theory 151 150 150 150 150	1992 king redu # unique c 150 150 150 150 150	96.9 indancy : * complete 99.3 100.0 100.0 100.0 100.0	2.4 2.4 into account average redundancy 3.4 4.4 4.4 4.8 4.4	- Laue (mean F2 10596.76 3997.05 2640.00 1529.26 1130.72	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07	0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047	0.022 0.022 irs merged RsigmaB 0.010 0.015 0.020 0.025 0.033	l): Pranam
<pre>inf-0.70 inf-0.80 Statistics resolu- tion(A)</pre>	5137 4709 # kept 514 664 657 726 662 545	2055 2055 # theory 151 150 150 150 150 150	1992 king redu # unique c 150 150 150 150 150 150	96.9 indancy : * omplete 99.3 100.0 100.0 100.0 100.0 100.0	2.2 2.4 into account; average redundancy 3.4 4.4 4.4 4.8 4.4 4.4 3.6	- Laue (mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15	0.012 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058	0.022 0.022 irs merged RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044	l): Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 # kept 514 664 657 726 662 545 464	2055 ution (tai # theory 151 150 150 150 150 150 150	1992 king redu # unique c 150 150 150 150 150 150	96.9 mdancy : * 99.3 100.0 100.0 100.0 100.0 100.0 100.0	2.2 2.4 into account; average redundancy 3.4 4.4 4.4 4.8 4.4 3.6 3.1	- Laue (mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03	0.022 alous pa Rint 0.013 0.029 0.038 0.047 0.058 0.047	0.022 0.022 irs merged RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062	l): Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 * * kept 514 664 657 726 662 545 465 465	2055 ution (tai # theory 151 150 150 150 150 150 150 150	1992 # unique c 150 150 150 150 150 150 150 150	96.9 indancy : * omplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0	2.4 2.4 into account; average redundancy 3.4 4.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7	- Laue (mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58	0.022 alous pa Rint 0.013 0.020 0.038 0.047 0.058 0.073 0.077	0.022 0.022 irs merged RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075	l): Pranana
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 * kept 514 664 667 726 662 545 464 405 299	2055 ution (ta: # theory 151 150 150 150 150 150 150 150 150	1992 king redu # 150 150 150 150 150 150 150 150 150 150	96.9 endancy : * 99.3 100.0 100.0 100.0 100.0 100.0 100.0 84.3	2.2 2.4 into account; average redundancy 3.4 4.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7 2.0	- Laue g mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93	0.022 alous pa Rint 0.013 0.020 0.038 0.047 0.058 0.047 0.053 0.073 0.073	0.022 0.022 irs mergec RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075 0.092	l): Pranato
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 * * kept 514 664 664 662 545 662 545 464 405 299 201	2055 ution (ta # theory 151 150 150 150 150 150 150 150 150 150	1992 king redu # 150 150 150 150 150 150 150 150 150 150	96.9 mdancy : * 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 34.3 34.6	2.2 2.4 into account; average redundancy 3.4 4.4 4.4 4.4 4.4 3.6 3.1 2.7 2.0 1.3	- Laue g mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01	0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.073 0.058 0.077 0.073 0.073 0.095	0.022 irs mergec RsigmaB 0.015 0.020 0.015 0.020 0.025 0.033 0.044 0.062 0.075 0.092 0.135	l) : Pratan
inf-0.70 inf-0.80 Statistics resolu- tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.03-0.95 0.95-0.89 0.89-0.84 0.84-0.81 0.81-0.77 0.77-0.70	5137 4709 vs resol # kept 514 667 726 662 545 464 405 299 201 5137	151 150 150 150 150 150 150 150	1992 king redu # unique c 1500 1500 1500 1500 1500 1500 1500 1500	96.9 mdancy : * somplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3 34.6 82.3	2.2 2.4 into account; average redundancy 3.4 4.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3 3.4	- Laue (mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02	29.02 group (anome mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01 33.93	0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.073 0.073 0.073 0.095 0.025	0.022 irs mergeo RsigmaB 0.015 0.020 0.025 0.033 0.044 0.062 0.075 0.092 0.155 0.092 0.155	l) : Pranan

go to crystal movie.

- 2. Index the faces of your crystal by the snap method
 - a. Select Snap
 - b. You can limit the number of HKL faces by clicking on "Max HKL"
 - c. Rotate your crystal until you see a face with perpendicular lines to it
 - d. Left click crosshairs along lines and face





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e. Right click and select "add face" you can also add mirror and we can correct size later

Crystal shape - add face X	Prior image Page up	ABS DISPLAY - play recorded sample movies (1.0.12) X
0.646 - 31.032 - 0.683	Next image Page down	
Custom hkl: 0-1 0 Custom hkl: 0-1 0 Integer hkl small (Snap mode): 0-1 0	Add face 2e	4
Distance selection Measured distance 0.02457 Custom distance: 0.02457	Define center Define scale	
Flags Fix distance (no refinement) Help Add face Cancel	Update H K L Update distance	0.6701.538.K134.0F-1280 #11 Peegs. Per € \$ Heat Mi 500.9500.0500 % 13417 7 7 Store tarlendow from tot

- Rotate crystal and do it again until all faces are measured f.
- g. You can now edit faces to match edge of crystal if used mirror

5	k	1	d	sise	Face
-0.00	-1.00	0.00	0.02457	0.0214247	- Marking
1.00	-0.00	-0.00	0.05117	0.0102874	O Drag
-1.00	0.00	0.00	0.05117	0.0102874	C Point
0.00	1.00	-0.00	0.02457	0.0214247	S Foint
0.00	-0.00	-1.00	0.10824	0.0050741	🕘 🔍 Snap
-0.00	0.00	1.00	0.09924	0.0050741	Distance
	5 -0.00 1.00 -1.00 0.00 -0.00	t k -0.00 -1.00 1.00 -0.00 -1.00 0.00 0.00 -0.00 -0.00 -0.00	k 1 -0.00 -1.00 0.00 1.00 -0.00 -0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00 -0.00 -1.00 0.00 -0.00 -1.00 -0.00 0.00 1.00	s k 1 d -0.00 -1.00 0.00 0.02457 1.00 -0.00 -0.00 0.05117 -1.00 0.00 0.00 0.05117 0.00 0.00 0.00 0.05117 0.00 -0.00 0.02457 0.00 0.00 -0.00 0.02457 0.00 -0.00 -0.02457 0.00 -0.00 -1.00 0.10224 -0.00 0.00 1.00 0.09924	k 1 d size -0.00 -1.00 0.00 0.02457 0.0214247 1.00 -0.00 -0.00 0.05117 0.0102874 -1.00 0.00 0.00117 0.0102874 0.00 1.00 -0.00 0.05457 0.0214247 0.00 -0.00 -0.0457 0.0102874 0.00 -0.00 -0.0457 0.0214247 0.00 -0.00 -1.00 0.0457 0.0214247 -0.00 -0.00 1.00 0.05524 0.0050741

- 3. Click on the "Refinalize" button.
- 4. Under the "Corrections" section, verify that "Empirical correction" is set to "Automated". This will apply the Blessing method of absorption correction to the reflections. You may also include faces. Click "OK".

on dialog: SM experiment to hkl file (1.0.15)	×
Finalize: experiment to hkl file	0
Sample	
Experiment: Ylid_20190827 Unit cell: 5.9691 9.0433 18.4007 90.1 90.1 90.0 993.2739 (CSD: 21 +0L)	
Set formula: Lattice - oP mmm Friedel mates: equivalent	
Corrections	
Empirical correction Automated Manual	
Numerical absorption Faces Sphere	
Space group and AutoChem	
Search for space group Auto Interactive Space group option	
Filters and limits Automated Manual	
Output	
Z:\Charlotte\Ylid_20190827\Ylid_20190827	
Standard set of files Copy hkl only to Yiid_20190827 Copy hkl to	
Create/overwrite Ylid_20190827 files (hkl, ins, cif_od) in Z:\Charlotte\Ylid_20190827.	
Export options Exported files: cif.	
Help Defaults OK Can	el



5. A new window will appear asking you to choose a space group. Choose the space group that is consistent with your previous choices. And press ok

on					
No.	Centro	CCDC	ICSD	R(int)	
19	-	20117	573	0.022	
18	-	1059	88	0.022	
NON		OSAWW	TRIC		
NOT	ACENTIN	03114141	LINC		
ormul	a				ОК
	No. 19 18 NON	No. Centro 19 - 18 - NON-CENTR	No. Centro CCDC 19 - 20117 18 - 1059	No. Centro CCDC ISD 19 - 20117 573 18 - 1059 88 NON-CENTROSYMMETRIC	No. Centro CCDC ICSD R(rrt) 19 - 20117 573 0.022 18 - 1059 88 0.022 NON-CENTROSYMMETRIC

6. Inspect the Rint and F2/sig(F2) to see how well the crystal diffracted vs resolution. Press OK

Data reduction	file contents	Data re	duction outp	ut	Red graphs	1	Data collection	output	Devices lo	g
1.31-1.14	630	238	235	98.7	2.7	2612.43	34.69	0.025	0.024	_
1.14-1.04	674	238	235	98.7	2.9	1484.11	25.73	0.034	0.031	
1.04-0.96	621	236	235	99.6	2.6	1117.68	21.08	0.041	0.040	
0.96-0.90	525	243	235	96.7	2.2	796.22	15.73	0.049	0.050	
0.90-0.85	438	245	236	96.3	1.9	528.67	11.31	0.063	0.068	
0.85-0.82	405	249	235	94.4	1.7	437.49	9.54	0.072	0.085	
0.82-0.78	343	295	235	79.7	1.5	369.51	7.22	0.070	0.102	
0.78-0.70	256	897	235	26.2	1.1	255.87	4.77	0.087	0.145	
inf-0.70	5137	3123	2351	75.3	2.2	2463.39	27.07	0.022	0.022	
inf-0.70 inf-0.80 Statistics resolu- tion(Å)	5137 4709 vs resol # kept	3123 2055 ution (ta # theory	2351 1992 king redu # unique c	75.3 96.9 ndancy % omplete	2.2 2.4 into account) average redundancy	2463.39 2660.11 - Laue g mean F2	27.07 29.02 group (anom mean F2/sig(F2)	0.022 0.022 alous pa Rint	0.022 0.022 irs merged): 3 RsigmaB	Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(A)	5137 4709 vs resol # kept	3123 2055 ution (ta # theory 	2351 1992 king redu # unique c	75.3 96.9 ndancy % omplete	2.2 2.4 into account) average redundancy	2463.39 2660.11 - Laue g mean F2	27.07 29.02 group (anom- mean F2/sig(F2)	0.022 0.022 alous pa Rint	0.022 0.022 irs merged): : RsigmaB	Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(Å) inf-1.73 1.73-1.33	5137 4709 vs resol # kept 514 664	3123 2055 ution (ta # theory 151 150	2351 1992 king redu # unique c 150 150	75.3 96.9 ndancy % omplete 99.3 100.0	2.2 2.4 into account) average redundancy 3.4 4.4	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05	27.07 29.02 group (anom- mean F2/sig(F2) 76.56 57.61	0.022 0.022 alous pa Rint 0.013 0.020	0.022 0.022 irs merged): : RsigmaB 0.010 0.015	Pmmm
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 514 664 657	3123 2055 ution (ta # theory 151 150 150	2351 1992 king redu # unique c 150 150 150	75.3 96.9 ndancy % omplete 99.3 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05 2640.00	27.07 29.02 group (anom- mean F2/sig(F2) 76.56 57.61 44.50	0.022 0.022 alous pa Rint 0.013 0.020 0.029	0.022 0.022 irs merged): : RsigmaB 0.010 0.015 0.020	Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(A) inf-1.73 1.73-1.33 1.33-1.15	5137 4709 vs resol # kept 514 664 657 726	3123 2055 ution (ta # theory 151 150 150	2351 1992 king redu # unique c 150 150 150	75.3 96.9 ndancy % omplete 99.3 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.8	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025	Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 514 664 657 726 662	3123 2055 ution (ta # theory 151 150 150 150	2351 1992 king redu # unique c 150 150 150 150	75.3 96.9 ndancy % omplete 99.3 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26 1130.72	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047	0.022 0.022 irs merged): 1 RsigmaB 0.010 0.015 0.020 0.025 0.033	Prana
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 514 664 657 726 662 545	3123 2055 ution (ta # theory 151 150 150 150 150	2351 1992 king redu # 150 150 150 150 150 150	75.3 96.9 ndancy * omplete 99.3 100.0 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.4 3.6	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058	0.022 0.022 irs merged): : RsigmaB 0.010 0.015 0.020 0.025 0.025 0.033 0.044	Pranan
<pre>inf=0.70 inf=0.80 Statistics resolu- tion(A)</pre>	5137 4709 vs resol # kept 514 664 657 726 662 545 464	3123 2055 ution (ta # theory 151 150 150 150 150 150	2351 1992 king redu # unique c 150 150 150 150 150 150 150	75.3 96.9 ndancy % omplete 99.3 100.0 100.0 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073	0.022 0.022 irs merged): : RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062	Pranan
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 514 664 657 726 662 545 464 405	3123 2055 ution (ta # theory 151 150 150 150 150 150 150	2351 1992 king redu # 150 150 150 150 150 150 150	75.3 96.9 mdancy % omplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19	27.07 29.02 group (anom mean F2/s1g(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58	0.022 0.022 alous pa Rint 0.013 0.020 0.038 0.047 0.058 0.047 0.058 0.073	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075	Pratan
<pre>inf-0.70 inf-0.80 Statistics resolu- tion(A)</pre>	5137 4709 vs resol # kept 514 664 657 726 662 545 464 405 299	3123 2055 ution (ta # theory 150 150 150 150 150 150 150 178	2351 1992 king redu # unique c 150 150 150 150 150 150 150 150	75.3 96.9 ndancy % omplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3	2.2 2.4 into account) average redundancy 3.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7 2.0	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.073 0.073	0.022 0.022 irs merged): : RsigmaB 0.010 0.015 0.025 0.025 0.025 0.025 0.023 0.044 0.062 0.075 0.092	Pranan
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 664 662 545 662 545 464 405 299 201	3123 2055 ution (ta # theory 150 150 150 150 150 150 150 150 178 451	2351 1992 # unique c 150 150 150 150 150 150 150 150 150 150	75.3 96.9 ndancy % omplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3 34.6	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.073 0.073	0.022 0.022 irs merged): : RsigmaB 0.010 0.015 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.022 0.025 0.033	Prana
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 ************************************	3123 2055 ution (ta # theory 	2351 1992 # unique c 150 150 150 150 150 150 150 150 150 156	75.3 96.9 mdancy % omplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 34.3 34.6 	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3 3.4	2463.39 2660.11 - Laue q mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 1529.26 1130.72 733.94 2463.39	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.073 0.073 0.073 0.073 0.095	0.022 0.022 irs merged): : RsigmaB 0.010 0.015 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.025 0.022	Ρπαιο

7. Click the OLEX2 button on the left toolbar to

will be in the struct folder under



start solution and refinement. All your files needed

olex2_XXXXX





PROCESSING YOUR DATA OFFLINE

A. DATA INTEGRATION

CAP automatically integrates data online, this section shows how to do it manually.

- Copy the parent directory (i.e. the whole folder) for your data from the X-ray-live drive 1.
- Double click the CrysAlisPro (CAP) RED 2.

shortcut icon on the desktop.

- 3. Double click the XXXXX.run file
- 4. Or in the Select Experiment menu, browse to your experiment. Select the XXXXX.par file.
- 5. Select the new "XXXXX" experiment file that has been created, and click "Open selected".

sAlis e	xperiment (1.0.43) - 2 experiments available -	(40.75a)			- D X
5 Select	experiment - standard list				
Name	Path		Created	Accessed	
cx1869a	Z:\Charlotte\cx1869a		Tue Apr 28 15:43:2	22 2020 running	
cx1812b	Z:\Charlotte\cx1812b		Wed Apr 15 13:17:	03 2020 running	
				Hide prev	Hide pre experiments
Diselected in fee					Hide screenings
Displaying infor	C values lass second back Dist and advance	C Public services	Contractor		st: Standard
• Standard	volume, laue, wavelength, Rint, redundancy	Protein screening	Custom columns		>> Delete Rename
Help	Multiple addition	Browse experiment		Delete experiment(s) from I	ist Open selected

6. Your diffraction image should appear as below. Now select the "Lattice wizard" 🕮 button.





7. Click the ">" button under "Peak hunting", and select "Peak hunting with user settings".

Lattice wizard (1.0.35)	×
Lattice wizard	
Description Control Contro Control Control	Peak hunting Unit cell finding Peak hunting with user settings Auto analyse unit cell Auto analyse unit cell Replace current peak table with a 'delta' Auto calibration Auto calibration soft Refine Incommensurates / Quasi-crystals Twinning - multi-crystals Incommensurates / Quasi-crystals Image: Load information Save information
	Ewald3D Ewald3D Log window Close

8. In the next window, select "Smart peak hunting" and press ok.



Peak hunting Run list, image type and image directory- Run list; Z:\Charlotte\Ylid_20190827\Ylid_20190827 Image dir: Z:\Charlotte\Ylid_20190827\Ylid_20190827 Image dir: Z:\Charlotte\Ylid_20190827\Yrames * type *stars end vish eng descent tappe phi 0 -14.00 -16.00 0.50 1.00 - 4.65 -10.00 -8.65 2 0 -14.00 1.00 - -4.65 95.00 - 2 0 -10.00 0.50 1.00 - - 4.65 95.00 - 2 0 -4.00 0.50 1.00 - - 4.65 95.00 - 3 0 - 0.50 1.00 - - - 0.00 - - 0.60 - 0.65 - 0.00 - - 0.60 - 0.60 - 0.60 - 0.60 - 0.60 - - 0.60 - -	start end 00 1, 50 00 1, 50 00 1, 100 00 1, 50	selected run	CRYSALIS ^{Po} .rodhypix .
Run list: L:: L:: <thl::< th=""> L:: <thl::< th=""> <thl::<< th=""><th>start end .00 1, 50 .00 1, 50 .00 1, 50 .00 1, 50 .00 1, 50</th><th>selected run</th><th>•.rodhypix •</th></thl::<<></thl::<></thl::<>	start end .00 1, 50 .00 1, 50 .00 1, 50 .00 1, 50 .00 1, 50	selected run	•.rodhypix •
Runge Gir. 2. (Charlotter (Hill 2019/027) (Faillies) 1 e -71.00 -60.00 .50 1.00 - 4.65 -10.00 -81.00 2 e - 14.00 11.00 .50 1.00 - 4.65 -10.00 -81.00 3 e12.00 27.00 0.50 1.00 - 4.65 +56.00 0.4 4 e - 14.00 25.00 0.50 1.00 - -6.65 57.00 -180. Run list modification By default the whole experiment will evaluated. Tedrit Tedrit C Automatic threshold and background detection (preferred) C Tradition	start end .00 1, 50 .00 1, 50 .00 1, 100 .00 1, 90 start num of s	sejected run	Edit end num of selected run
Applie Constraint Copy of the set of the s	start num of s	selected run	Edit end num of selected run
2 0 1.00 0.50 1.00 - 4.07 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 -95.00 - 1.00 - - - - 1.00 - - - - 1.00 - - - 1.00 - - - 1.00 - - 5.65 57.00 - 1.00 - - 5.65 57.00 - 1.00 - To To To To To To To To To	00 1, 50 00 1, 100 00 1, 90 start num of s	sejected run	Edit end num of selected run
a 32.00 27.00 0.50 1.00 - -4.65 -55.00 0.4 4 o -16.00 25.00 0.50 1.00 - -6.65 57.00 -180. Xun list modification	.00 1, 100 .00 1, 90 start num of s	selected run	Edit end num of selected run
tun list modification By default the whole experiment will evaluated. To modify this behaviour edit the run list > Automatic threshold and background detection (preferred)	start num of s	selected run	Edit end num of selected run
eak finding control Threshold: 1000 7x7 average: 20	nal pe 8	 Smart peak Overwrite existing Yes 	k hunting C 3D peak extraction g peak hunting table C No
- Use background subtraction			
Background evaluation control -> 50	Edit Re	50	Edit Fr
		© 1 – O	2 04
Reduce background accumulation to SHORT type (sa			
Resolution limits			
Skip peaks outside resolution limits			Edit res limits
Apply float correction n/a			
Demonstration Coursels Continues		Help	Cancel OK

- 9. Say yes to overwriting the peak table. A window will pop up, and images will rapidly start to sequence as the peak hunting algorithm searches for diffraction peaks. You should see the strongest peaks being marked with "+"
- 10. At the end of the process, a unit cell is displayed, but 0% of the reflections have been fit to the unit cell. This



is because the displayed unit cell is from a previous peak hunt

11. Click the ">" button under "Unit cell finding", and select "Unit cell finding with options"

Lattice wizard (1.0.35)	×
Lattice wizard	CRYSALIS ^{Ro}
LATICE 13.7977(11) 7.454(2) 19.604(3) 68.608(14) 72.616(10) 71.310(9) 4665.1(10) Lattce reduction selected cell 15.7976(11) 7.454(2) 19.7138 68.3164 72.0053 71.1193 aP 31 reduced cell 15.7996 17.4670 19.7138 68.3164 72.0053 71.1193 aP 31 reduced cell 15.7996 17.4670 19.7138 68.3164 72.0053 71.1193 4671.2 PEAK hunting table UB fit with 0 obs out of 27320 (total:27320,skipped:0) (0.00%) INTRUMENT MOCEL Goniometer beam: 0.06689 alpha: 50.09731 beta: 0.00412 om zero: -0.239337 tazero: Detector -0.01400 x-core: 388.13500 y-core: 381.95900 y-core: 361.96900 distance: 384.13500 y-core: 361.96900 distance: Wavelength Cu (Ang): A1 1.54056 A2 1.54439 B1 1.39222	Image: Select of the select
🕞 🕫 😦	indow Close



INTERCONNICTION Integrated Molecular Structure Education and Research Center Northwestern University



12. Ensure that "Normal peak table" and "T-vector Dirax" are selected. More importantly, "Single crystal" and "SM" should be selected in the Sample type filed. Here, you could also select "User" and set upper and lower bounds for unit cell parameters. For now, make sure your window looks like this image, and click "OK"

 Normal peak table 	Algorithm
C Delta (differential) peak table	• T-vector Dirax
Find center File semanics Fdit	C Stereographic
Sample type	
Single crystal	
Unit cell limits min	
ⓒ SM C PX C User 2.0	120.0 Cal
C Twin / multicrystal	max
# 0" components 2 💌 2.0	120.0 Ca
Lock present components (see Twin information' section of The Section of The Section of The Section Section of The Section Sec	Time 🗖 Time
HINT: To lock current UB for twin 1, first go to UM 'Current UB to twin'. Then return here and select T	TWIN utility and click win 1' checkbox above.
Consider Service Descentager	
🔽 Force Strengel Frankfor al concernent e Constr	
(nown cell	
Search known cell 5.96 9.03 18.38 90.01 89.98	90.00

Ewald explorer reciprocal space

- 13. Click Ewald explorer and look at your reciprocal space. You may look down the different axis. You may turn the overlay on and off. If the cell is indexed properly, then the grid lines should be passing through reflections. If there are a lot of reflections NOT passing through the grid, or if there are grid lines NOT passing through reflections, then something is wrong.
- 14. You can look for twins here too and look at the histograms





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15. From within the Ewald Explorer, check the predicting crystal system and lattice centering: Left click lattice and select "Modify lattice type". A window appears listing possible unit cells from highest to lowest.

Crystal	tion (1.0.6)	×
Log window LATINCT (CSR-20-00) 5-9556(4) 9-0121(5) 18-1849(11) 50.023(5) 90.020(5) 90.020(5) V = 988-97(11) 0 ² / ₂ , index: 100.00% Overlaw off Activate bala implificrential	Lattice reduction	CRYSALIS
Activate incommensurate peaks	Input cell: 5.96201 9.0327 18.39579 85.58885 50.00219 90.00219 vol:951.4 O.00010 0.00010 0.00010 0.00010 0.00010 0.00010 0.00010 Niggli form: 25.54558 81.70844 22.40494 0.02225 0.00420 0.00219 Reduced cell: 5.96201 9.02027 18.39579 85.58885 89.59671 89.59661 vol:991.4 Time: Thu Aug 29 12:24:46 2019 18.39579 85.58885 89.59671 89.59661 vol:991.4	Tolerance 0.01500
Datibution hetergame (2 vectors prejection 6-1-2) a" ans b" ans	Primitive to sel: UM C 1.000 0.000 0.000 0.000 -1.000 0.000 0.000 -1.000 Sel to primitive: UM C 1.000 0.000 0.000 0.000 -1.000 0.000 0.000 -1.000	Lattice as is Reduce cell Clear primitive UB
e" and	# IT code transformed cell (a,b,c,zl,be,gz,vol) 66 1 22 oP 5.96201 9.03927 18.39579 89.598855 89.59671 89.59661 591.39 0.	i proj dist 00311
	2 33 mP 5.54201 9.03527 18.35579 50.01115 50.00229 55.95661 591.35 0. 3 34 mP 5.54201 18.35579 5.03277 85.58658 50.00229 50.0025 591.35 0. 4 35 mP 9.03527 5.54201 18.35575 50.00229 50.01115 55.59661 591.35 0. 5 31 mP 5.54201 9.03527 18.35579 59.58285 59.59671 85.59661 591.39 0.	02251 02266 00706 00000
	Help To history Show C all Niggli cases	Skip indexation after closing

symmetry. The relative small figure of merit of 0.03 suggests that oP is correct. Click OK.

- 16. Close the Ewald viewer and Lattice Wizard.
- 17. Click overlay spot prediction and play through your frames with the 10-foward button. 🕑 Make sure the + marks are appearing on the peaks. Note: peaks marked ◊ and □ correspond to approaching and receding peaks, respectively. Click on 🙆 to toggle the frame information on and off





B. DATA REDUCTION

1. Click on "Data Reduction", (this time not the carrot next to it). Click on it again and select "Data reduction with options"



2. Ensure that don't use filter is checked if lattice is not known. If the lattice is centered (A, B, C, I, F, R), click use filter for: and select the correct centering. Make sure Normal data reduction is checked and then click "Next".

	Step 2: E	Experiment	t run list f	or data re	eduction								
ep 1: Orienta	Hun list.	2: \Lhano	de (rad_	2019082	A 1140_201	90827				* rodh	pix	•	
UB - matri	Image di	r: Z:\Char	lotte \Yid	_201908	27\frames								
0.0050	# 037pe	-74.00	end	widsh	exposure	onega	detector	kappa	phi .	-	end		
-0.0051	2.0	-14.00	11.00	0.50	1.00	-	4.87	-99.00	-90.00	1,	50		
5.96201	4 0	-22.00	27.00	0,50	1.00	2	-5.65	-99.00	-150.00	1,	90	- 1	
V = 9												- 1	
elected cell												- 1	
2 5.9620												- 1	
												- 1	
												- 1	
	172							-m					
Lattice extind	By defau	it the who	le experir	nent will	evaluated.	To mod	fy this	Edit	tart nun	n of se	lected	run	
Don't use	behaviou	r edit the	run list -	->			.,	Edit	end num	ofse	lected	run	
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O Use filter													m=0
				< <u>B</u> ack	Ne	d>	Fini	sh	Can	cel		Help	el Loar
								11 - 21		-			- Coar
Twinning/Multi	crystal	(activa	ated b	νим	TWIN	entries	a) —						
rwinning/ maia	ciyatui	(ucuvi	neu D	, 014			.					_	
												: L.	
Use autor	masne vri												

- 3. Check that CrysAlis^{Pro} has found all runs. This list will dictate which images are integrated. If there were bad frames, omit them here. To delete bad frames, click on the run '#'. Then select edit start [end] number of selected run, depending on whether the bad frames are at the beginning or the end of the run. Change the start [end] number to exclude the bad frames. To omit an entire run, enter 0 (zero) as the start number. Press next.
- 4. You can change some parameters here. You can correct for sample wobble or sudden movement. Clear data from previous run and Clear all data from tmp. Make sure you click yes in warning windows.





Profile fitting data reduction CressAcis**** Step 3: Basic algorithm parameters Reflection position prediction ✓ Auto select optimal prediction approach on run basis ✓ Follow model changes on frame by frame basis (moderate sample wobbling) ✓ Follow significant sample wobbling (2-cycle 3D peak analysis) Follow sudden (discontinuous) changes of sample orientation Drientation search range (max:10 deg) 2.00 Search steps/deg (max:10) 4 Edit special pars Data from previous run of 'dc profit' 3d profile information and/or integration results on the disk. Clear data from previous run	rysAlisPro data reduction assistant (1.0.29)	
Profile fitting data reduction CRYSALIS** Step 3: Basic algorithm parameters Reflection position prediction ✓ Auto select optimal prediction approach on run basis ✓ Follow model changes on frame by frame basis (moderate sample wobbling) ✓ Follow significant sample wobbling (2-cycle 3D peak analysis) Follow significant sample wobbling (2-cycle 3D peak analysis) ✓ Follow significant sample wobbling (2-cycle 3D peak analysis) ✓ Follow significant sample wobbling (2-cycle 3D peak analysis) ✓ Follow sudden (discontinuous) changes of sample orientation Drentation search range (max 10 deg) 2.00 Search steps/deg (max 10) 4 Edit special pars Scratch all tmp data of previous profit run 2d profile information and/or integration results on the disk Scratch all tmp data of previous profit run Clear data from previous run Clear all data from tmp		
Step 3: Basic algorithm parameters Reflection position prediction Auto select optimal prediction approach on run basis Follow model changes on frame by frame basis (moderate sample wobbling) Follow significant sample wobbling (2-cycle 3D peak analysis) Follow sugnificant sample wobbling (2-cycle 3D peak analysis) Follow sudden (discontinuous) changes of sample orientation Drientation search range (max:10 deg) 2.00 Search steps/deg (max:10) 4 Edit special pars Data from previous run of 'dc profit' Scratch all tmp data of previous profit run Bropfie information and/or integration results on the disk Scratch all tmp data of previous profit run Encider from all analysis files including background Clear all data from tmp	Profile fitting data reduction	CRYSALIS [™]
Reflection position prediction ✓ Auto select optimal prediction approach on run basis ✓ Follow model changes on frame by frame basis (moderate sample wobbling) ✓ Follow significant sample wobbling (2-cycle 3D peak analysis) ✓ Follow significant sample wobbling (2-cycle 3D peak analysis) ✓ Follow sudden (discontinuous) changes of sample orientation Orientation search range (max 10 deg) 2.00 Search steps/deg (max 10) 4 Edit special pars Scratch all tmp data of previous profit run 3d profile information and/or integration results on the disk Scratch all tmp data of previous profit run Clear data from previous run Clear all data from tmp	Step 3: Basic algorithm parameters	
Auto select optimal prediction approach on run basis Follow model changes on frame by frame basis (moderate sample wobbling) Follow significant sample wobbling (2-cycle 3D peak analysis) Follow sudden (discontinuous) changes of sample orientation Brientation search range (max 10 deg) 2.00 Search steps/deg (max 10) Edit special pars Data from previous run of 'dc profit' Scratch all tmp data of previous profit run Englise information and/or integration results on the disk. Clear data from previous run	Reflection position prediction	
 Follow model changes on frame by frame basis (moderate sample wobbling) Follow significant sample wobbling (2-cycle 3D peak analysis) Follow sudden (discontinuous) changes of sample orientation Brientation search range (max 10 deg) 2.00 Gearch steps/deg (max 10) Edit special pars Data from previous run of 'dc profit' Scratch all tmp data of previous profit run Brientation and/or integration results on the disk. Clear data from previous run	✓ Auto select optimal prediction approach on run basis	
Follow significant sample wobbling (2-cycle 3D peak analysis) Follow sudden (discontinuous) changes of sample orientation Brientation search range (max 10 deg) 2.00 Search steps/deg (max 10) Edit special pars Data from previous run of 'dc profit' Scratch all tmp data of previous profit run Entry tmp folder from all analysis files including background Clear data from previous run	Follow model changes on frame by frame basis (moderat	
Follow sudden (discontinuous) changes of sample orientation Grientation search range (max 10 deg) 2.00 Search steps/deg (max 10) Edit special pars Data from previous run of 'dc profit' Scratch all tmp data of previous profit run Bright run folder from all analysis files including background Clear all data from tmp	✓ Follow significant sample wobbling (2-cycle 3D peak analysis)	3)
Orientation search range (max/10/deg) 2.00 Search steps/deg (max/10/ 4 Edit special pars Edit special pars Scratch all tmp data of previous profit run Data from previous run of 'dc profit' Scratch all tmp data of previous profit run 3d profile information and/or integration results on the disk Encly tmp folder from all analysis files including background Clear data from previous run Clear all data from tmp	Follow sudden (discontinuous) changes of sample orientation	1
Edit special pars Data from previous run of 'dc proffit' 3d profile information and/or integration results on the disk Clear data from previous run Scratch all tmp data of previous profit run Empty tmp folder from all analysis files including background Clear all data from tmp	Orientation search range (max 10 deg) 2.00 Searc	h steps/deg (max 10) 4
	Edit special pars	
	Data from previous run of 'dc proffit' 3d profile information and/or integration results on the disk Clear data from previous run	mp data of previous proffit run older from all analysis files including Clear all data from tmp
	Data from previous run of 'dc profit' 3d profile information and/or integration results on the disk Clear data from previous run	mp data of previous proffit run older from all analysis files including Clear all data from tmp

- 5. Edit special pars "Use resolution limits". Click "Edit limits" will enable you to reset your resolution.
- 6. Click "Edit high limit", and enter "0.77" in the Editing high-resolution limit window. You should already have an idea on how far the crystal diffracts based on your unit cell analysis. Click "OK", and then click "OK" on the "Resolution limits" window.
- 7. Click "Next" to Background evaluation window.



- 8. You can change to "Smart background" option for weaker data. Your window should appear as below.
 - a. Re = # of frames used in background calculation
 - b. Fr = How often calculation restarts.
 - c. Make #s smaller if sudden changes between frames



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- d. Background for 3D integration for average
- e. Smart: weak data or Large variation w/in frame
- f. Click "Next" to proceed.
- 9. In the outlier rejection window, use the pull-down menu to choose the correct Laue group. Unless you know you have non-centrosymmetric space group make sure Use Friedel mates as equivalent is checked.
- 10. Change output name to something new, make sure that the Space group determination is set to "Manual". Check the formula and Z.

	Proffit: CrysAlisPro data reduction assistant (1.0.29)	\times
Proffit: CrysAlisPro data reduction assistant (1.0.29)	Profile fitting data reduction	
Profile fitting data reduction	Step 6: Output Tip: You may change the output name and directory to keep results of data reductions under different parameter sets (UB, supercells) Output file name:	
Step 5: Outlier rejection CCD data sets usually contain more than the unique data required for the structure determination. This redundant data can be used to check for measurement outliers. The rejection is based on R. Blessing (1997), J. Appl. Cryst. and additional CCD specific criteria.	Z:\Charlotte\Ylid_20190827\Ylid_20190827 Change output name Finalization options	
Outlier rejection © Use outlier rejection: mmm oP 5.96201 9.03927 18.39579 89.98885 89.99661	Space group determination Automatic Automatic Automatic Manual Automatic structure solation (AutoChem) Chemical formula not available Completeness computation:	
✓ Use Friedel mates as equivalent < Back Mext > Finish Cancel Help	Make unwarp pictures Mexioder (one for h, k, j): U Presolution: U.80 	

11. Click "Finish", Watch the integration and monitor the output in the tab. The software runs through the dataset twice. First, the software locates the peaks (marked "+") and develops a 3D peak profile. Second, the UB matrix and 3D profile are used to calculate the position and intensity of each reflection (3D integration & fitting). The peaks are marked by "integration masks" that give a visual idea of the size and possible overlap of the peaks.



12. Once the integration is finished, the GRAL window will pop-up (Like XPREP) and ask you to assign the space group. Click "Apply".

GRAL (vers.: 2.4.1) - YLID_20190827.HKL		? ×
Space group determination		
of Settings 📇 Load		
	Cell parameters	
TTTD_50130851.HKF (P143)	a: 5.96540 b: 9.04265	c: 18.40146
	α: 90.00580 β: 90.00080	γ: 90.00540
	Errors of cell parameters	
	a: 0.00017 b: 0.00025	c : 0.00047
	α: 0.00220 β: 0.00220	γ: 0.00230
Load Append HKL view	I Read parameters from file radia	12 J73
	Cancel	Apply Help

- 13. Investigate the centering absences. Recall that the first row corresponds to the total number of reflections you would expect to collect for the condition listed in the column. The second row indicates how many of the symmetry equivalent reflections that you collected violate that condition. This cell appears to be primitive. Click "Apply".
- 14. The Niggli cell test will look for any unit cell transformation matrices that produce a reduced cell. Click "Apply"



15. Pay close attention to the Rint value, recall that this value should be below 0.10 for good data. A low Rint value suggests that your integration strategy and unit cell assignments are correct. Click "Apply".



RAL (vers.: 2.4.1) - YLID_20190827.HKL	? ×
Space group determination	CRYSALIS ^{PRO}
😙 Settings 👜 Load 🙀 Centering 🛕 Niggli 🏂 Lattice	
Current cell a: 5.96540 b: 9.04265 c: 18.40146 α: 90.00580 β: 90.00080 γ: 90.00540	election tolerance Recalc
Transformation matrix from original cell 0.0000 <	
Option: (32) err= 0.018 ORTHOPHORETC P-lattice R(int) 0.023 [2673] Vol = 992.6 Option: [33] err= 0.018 MONOCLINIC P-lattice R(int) 0.023 [2473] Vol = 992.6 Option: [34] err= 0.017 MONOCLINIC P-lattice R(int) 0.023 [2443] Vol = 992.6 Option: [35] err= 0.005 MONOCLINIC P-lattice R(int) 0.023 [2645] Vol = 992.6 Option: [35] err= 0.005 MONOCLINIC P-lattice R(int) 0.023 [2645] Vol = 992.6 Option: [44] err= 0.000 TRICLINIC P-lattice R(int) 0.020 [1661] Vol = 992.6	^
Show C all crystal lattices C the best match	15 hes
Cancel	Apply Help

16. Now GRAL will search for higher metric symmetry and additional centering conditions. Click "Apply".

ŝ	Space grou	ıp d	eterm	inatio	on							CRYSALIS
Setti	ings 🖽 Load 🙀 Cente	ering j	, Niggli	<u>مُلْ</u> Lattic	:e 🐼 Ce	ntering						
	Lattice exceptions:	P	A	в	с	I	F	Obv	Rev	All		
	N (total) =		2569	2568	2563	2576	3850	3446	3432	5143		
	N (int>3sigma) =	ō	2335	2280	2333	2317	3474	3110	3087	4633		
	Mean intensity =	0.0	25.7	22.9	24.1	24.3	24.2	24.7	25.0	24.6		
	nean int/sigma -	0.0	10.4	10.0	10.4	16.3	16.1	16.3	16.5	10.4		
attice	e type											4.6
•	P CA		C	в		с с		01		C F	C R(obv)	
	Intensity divided by 100											

- 17. The |E2-1| analysis indicates a center symmetric structure. Look at the "Experimental" column, and the |E2-
 - 1 value. Click "Apply"

17	YLID_20	190827.	HKL											?
SI	pace	gro	up de	eterm	inatio	n							CRY	SALIS
Settings 📇 L	Load	Cent	terinc 🎍	Niggli	ġ, Lattic	e 🛃 Ce	entering	<u>⊊</u> <e2-1></e2-1>						
	Ехре	erimen	tal	Acen	tric	Cer	tric	Нур	ercentri	c [for n	=2 (*)]	 		
< E >		0.81	3	0.	886	< 0	.798>		0.718	1				
< E 2>		0.86	8	1.	000	1	.000		1.000					
< E 3>		1.09	6	<1.	329>	1	.596		1.916					
< 12 42 2 72 55		2.46	5	<2.	2225	3	202		4.500					
<1816>		4 22	4	<6	000>	19	000		37 500					
< E*E-1 >		0.71	7	<0.	736>		.968		1.145					
<(E*E-1)2>		0.82	9	<1.	000>	2	.000		3.500	(*)				
< E*E-1 3>		1.64	8	<2.	415>	8	.691		26.903					
*) number o	f asyr	metri	c units	in one	cell:									
	n=2	n=3	n=4	n=5	n=6	n=7	n=8	n=9	n=10	n=11	n=12			
<(E*E-1)2>	3.50	5.75	9.13	14.19	21.78	33.17	50.26	75.89	114.33	172.00	258.49			
C Centrosym	metric			ا ھ	Von-Centro	osymmetric								
												Cancel	Apply	Help



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18. Space groups are presented.

(1) - YLID_20190827.HKL	?
Space group determination	CRYSALIS
Settings 👜 Load 嵌 Centering 点, Niggli 点, Lattice 嵌 Centering 📐 <e2-1> 🕕 Space Group </e2-1>	
ystematic absence exceptions:	
21 b c n21acn21abn ▼ 4 311 255 298 8 201 195 198 17 76 80 78 ▼7>3s 0 271 242 245 0 161 153 132 0 68 70 62	
I> -0.0 33.0 31.6 38.6 0.0 54.3 53.7 28.3 -0.0 47.4 47.4 32.2 I/s> 0.2 18.6 17.5 18.3 0.1 20.4 20.8 13.7 0.1 21.1 21.6 19.1	
ctive filter: None Non-centro C Chiral	
Space Group No. C/A En. O.A. Pie. Pyr. CCDC ICSD R(int) N(eq)	
P2(1)2(1)2(1) (abc) 19 A Y Y Y N 20117 573 0.022 2790 P2(1)2(1)2(-cba) 18 A Y Y Y N 1059 88 0.022 2790	
how	
C all space groups C all solutions on a branch (like in IT pp 42-47, 55-67)	advanced space group selection
	Connect Another Hole

19. Now GRAL will produce your HKL and .ins files. Click "Apply".

Cremical formula: C2 H2004 52 Tritt Had SHORT'S BOOLD 110111 Import formula Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S BOOLD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S HAD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had SHORT'S HAD 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had 110111 Tritt Had SHORT'S BOOLD 110111 Formula Had 110111 T	19 Space	190827.нкі. group determination			? CRYSAL	× S ^{Pro}
NOTE: Unconstrained cell visible above will be afterwards replaced by a refined constrained one	Settings Change Load p Z Chenical formula: Z TTTL Tisk_Disket Settings Settings Total Settings Settings Settings Settings Total Settings Setting <th>Z Centerind J. Nigoli J. Lattice J. Centerind < < Z C22 H20 O4 52 Centering</th> <th>2-1> [1] Space Group [12] Ins-File [Import formula]</th> <th>Formula wt: 412.54 Mu(mm-1): 0.29 Density: 1.300 F(000): 432.00 A to:vo: 1.300 F(100): 432.00 A to:vo: 1.743 H all balanent(4): C-22.00(4.06%) B=20.00(4.06%) S=2.00(15.54%)</th> <th></th> <th>~</th>	Z Centerind J. Nigoli J. Lattice J. Centerind < < Z C22 H20 O4 52 Centering	2-1> [1] Space Group [12] Ins-File [Import formula]	Formula wt: 412.54 Mu(mm-1): 0.29 Density: 1.300 F(000): 432.00 A to:vo: 1.300 F(100): 432.00 A to:vo: 1.743 H all balanent(4): C-22.00(4.06%) B=20.00(4.06%) S=2.00(15.54%)		~



INTERC Integrated Molecular Structure Education and Research Center Northwestern University

20. Notice the side bar of CrysAlisPro: resolution, redundancy, intensity (F2/ σ (F2), Rint, and completeness values are listed for the reflection list. As is mosaicity values (e1,e2,e3), the min/max Empirical abs and Frame scales values. Mosaicity gives an idea of peak widths and crystal quality. For a good quality organic sample, values <1 are normal. The frame scaling and the empirical absorption correction correct for inconsistencies in the X-ray beam, absorption, and other anomalies. Ideally, both the min/max empirical absorption correction and the frame scaling should be close to 1. If a crystal is of poor quality, or a crystal absorbs X-rays strongly and a more accurate absorption correction based on crystal shape and size is necessary, then these values will deviate more substantially from 1.

Crystal RED
Data Collection
Data Reduction
FRAMES/RUNS In run list: 290/4, used: 290/4
3D PROFILE ANALYSIS Frames done: 290 Reflections tested: 5134, used: 3774 Avg mosaicity (in degrees) - 4 run(s) e1=0.44, e2=1.00, e3=0.58 Max incidence angle profile change(e2):1159
3D INTEGRATION & FITTING Frames done: 290 Fitted: 5173, overf/bad: 0, hidden: 396 Outliers rejected: 35
SCALING / NUMERICAL ABSORPTIONEmpirical abs (e=2 o=0): min=1.00,max=1.00scales (1/scale): min=0.96,max=1.00pairs treated as equivalent
RESULTS (290 frames) - SYM: Pmmm Resolution(A) Redundancy F2/sig(F2) Rint inf - 0.70 3.4 33.9 0.025 inf - 0.80 3.8 36.4 0.024 Completeness: 99.8% (0.80 ANG) Anom compl.: 96.8% (P222)
<pre>SPACE GROUP DESCRIPTOR P2(1)2(1)2(1) Group #: 19 (2 SG found) no data coverage: h00,</pre>
DATA REDUCTION OPTIONS Per-frame model refinement used 2-cycle 3D peak analysis used 3D profile fitting used





C. MEASURE CRYSTAL FACES AND REFINALIZE

If X-ray absorption is significant, a "face indexed" absorption correction must be performed. At minimum the size of the crystal must be provided for publication

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1. Click on from the side bar. And then go to crystal movie.

	utput	Data collection o		Red graphs	ıt	duction outpu	Data re	file contents	Data reduction f
034 0.031	0.034	25.73	1484.11	2.9	98.7	235	238	674	1.14-1.04
041 0.040	0.041	21.08	1117.68	2.6	99.6	235	236	621	1.04-0.96
049 0.050	0.049	15.73	796.22	2.2	96.7	235	243	525	0.96-0.90
063 0.068	0.063	11.31	528.67	1.9	96.3	236	245	438	0.90-0.85
072 0.085	0.072	9.54	437.49	1.7	94.4	235	249	405	0.85-0.82
070 0.102	0.070	7.22	369.51	1.5	79.7	235	295	343	0.82-0.78
087 0.145	0.087	4.77	255.87	1.1	26.2	235	897	256	0.78-0.70
022 0.022	0.022	27.07	2463.39	2.2	75.3	2351	3123	5137	inf-0.70
022 0.022	0.022	29.02	2660.11	2.4	96.9	1992	2055	4709	inf-0.80
lint RsigmaB	Rint	F2/sig(F2)	r 2	requiridancy	ompiece	antique o	cneory	repo	tion(A)
lint RsigmaB	Rint	F2/sig(F2)	F2						tion(A)
(1nt RsigmaB 013 0.010 020 0.015	0.013	F2/sig(F2) 76.56 57.61	10596.76	3.4	99.3	150	151	лерс 514 ссл	tion(A) inf-1.73
(1nt Rsigmaß 013 0.010 020 0.015 029 0.020	0.013 0.020	F2/sig(F2) 76.56 57.61 44 50	10596.76 3997.05 2640.00	3.4 4.4 4.4	99.3 100.0	150 150	151 150	514 664 657	tion(A) inf-1.73 1.73-1.33 1.33-1.15
(1nt RS1gmaB 013 0.010 020 0.015 029 0.020 038 0.025	Rint 0.013 0.020 0.029 0.038	F2/sig(F2) 76.56 57.61 44.50 33.40	10596.76 3997.05 2640.00	3.4 4.4 4.4 4.4	99.3 100.0 100.0	150 150 150 150	151 150 150	514 664 657 726	tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03
(1nt Rsigmab 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033	Rint 0.013 0.020 0.029 0.038 0.047	F2/s1g(F2) 76.56 57.61 44.50 33.40 27.07	F2 10596.76 3997.05 2640.00 1529.26 1130 72	3.4 4.4 4.4 4.8 4.4	99.3 100.0 100.0 100.0	150 150 150 150 150	151 150 150 150 150	514 664 657 726	inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03
(1nt Rs1gmab 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044	0.013 0.020 0.029 0.038 0.047 0.058	F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15	F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91	3.4 4.4 4.4 4.8 4.4 3.6	99.3 100.0 100.0 100.0 100.0 100.0	150 150 150 150 150 150 150	151 150 150 150 150 150	514 664 657 726 662 545	<pre>:ion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89</pre>
(11) K31gmaB 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044 073 0.062	0.013 0.020 0.029 0.038 0.047 0.058 0.073	F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03	10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67	3.4 4.4 4.4 4.8 4.4 3.6 3.1	99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0	150 150 150 150 150 150 150 150	151 150 150 150 150 150 150 150	514 664 657 726 662 545 464	<pre>inf-1.73 inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89 0.89-0.84</pre>
lint KsigmaB 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044 073 0.062 077 0.075	Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.073	F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58	10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19	3.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7	99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 100.0	150 150 150 150 150 150 150 150 150 150	151 150 150 150 150 150 150 150 150	514 664 657 726 662 545 464 405	tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89 0.89-0.84 0.89-0.81
Lint KsigmaB 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044 073 0.062 077 0.075 073 0.092	Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.077 0.073	F2/s1g(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93	F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28	3.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7 2.0	99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3	150 150 150 150 150 150 150 150 150 150	151 150 150 150 150 150 150 150 150 150	514 664 657 726 662 545 464 405 299	tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89 0.89-0.84 0.84-0.81 0.81-0.77
Lint KsigmaB 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044 073 0.062 077 0.075 073 0.092 095 0.135	Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.073 0.073 0.073 0.095	76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01	F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02	3.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3	99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3 34.6	150 150 150 150 150 150 150 150 150 150	151 150 150 150 150 150 150 150 150 178 451	514 664 657 726 662 545 464 405 299 201	tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89 0.89-0.84 0.84-0.81 0.81-0.77 0.77-0.70
11nt K=1 gmaB 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044 073 0.062 077 0.075 073 0.092 095 0.135	Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.077 0.073 0.073 0.095	F2/s1g(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01 33.93	10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02 2463.39	3.4 4.4 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3 3.4	99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3 34.6 82.3	150 150 150 150 150 150 150 150 150 156	151 150 150 150 150 150 150 150 150 178 451	514 664 657 726 662 545 464 405 299 201 5137	tion(A) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89 0.89-0.84 0.84-0.81 0.81-0.77 0.77-0.70 inf-0.70
lint k=1 gmaB 013 0.010 020 0.015 029 0.020 038 0.025 047 0.033 058 0.044 073 0.062 073 0.092 095 0.135	Rint 0.013 0.020 0.038 0.047 0.058 0.073 0.077 0.073 0.073 0.095	76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01 33.93 36.40	12 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02 2463.39 2600.11	3.4 4.4 4.4 4.6 4.4 3.6 3.1 2.7 2.0 1.3 3.4 3.8	99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3 34.6 82.3 99.9	150 150 150 150 150 150 150 150 150 156 1506 1230	151 150 150 150 150 150 150 150 150 178 451 1830 1231	514 664 657 726 662 545 464 405 299 201 5137 4709	Lion(A)

- 2. Index the faces of your crystal by the snap method
 - a. Select Snap
 - b. You can limit the number of HKL faces by clicking on "Max HKL"
 - c. Rotate your crystal until you see a face with perpendicular lines to it
 - d. Left click crosshairs along lines and face







e. Right click and select "add face" you can also add mirror and we can correct size later

Crystal shape - add face X	Prior image Page up	ABS DXRFLAY - play recorded sample movies (10.12)
0.646 -31.032 -0.683	Next image Page down	
	Add face 2e	
Distance selection	Define center	
C Custom distance: 0.02457	Define scale	
Figs Fix distance (no refinement)	Update H K L	
Help Add face Cancel	Update distance	0.57/01.530.07.534.0P.528.0 811 Ready. Pro: 4 a Heat Servirinage Capaboard. MI 500.07.500.07.500.00 % 134'Y 7 12' Slow tool window Refu

- f. Rotate crystal and do it again until all faces are measured
- g. You can now edit faces to match edge of crystal if used mirror

-						
#	h	k	1	d	sise	Face
1	-0.00	-1.00	0.00	0.02457	0.0214247	- Marking
2	1.00	-0.00	-0.00	0.05117	0.0102874	C Drag
3	-1.00	0.00	0.00	0.05117	0.0102874	C Point
4	0.00	1.00	-0.00	0.02457	0.0214247	C o
5	0.00	-0.00	-1.00	0.10824	0.0050741	💌 Snap
0	-0.00	0.00	1.00	0.09924	2g	Distance d h k l
						Val. Step

3. Click on the Refinalize button.

an dialog: SM experiment to hkl file (1.0.15)	×
Finalize: experiment to hkl file	SPRO
Sample	
Experiment: Ylid_20190827 Unit cell: 5.9691 9.0433 18.4007 90.1 90.1 90.0 993.2739 (CSD: 21 +0L)	
Set formula: Lattice - oP mmm Friedel mates: equivalent	
Corrections	
Empirical correction Automated Manual	
Numerical absorption Faces Sphere	
Space group and AutoChem	
Search for space group Auto Interactive Space group opt	ions
Filters and limits Automated Manual	
Output	
Z:\Charlotte\Ylid_20190827\Ylid_20190827 Cha	nge
Standard set of files Copy hkl only to Ylid_20190827 Copy hkl to	
Create/overwrite Ylid_20190827 files (hkl, ins, cif_od) in Z:\Charlotte\Ylid_20190827.	
Export options Exported files: cif.	
Help Defaults OK	ancel

4. Under the "Corrections" section, verify that "Empirical correction" is set to "Automated". This will apply the Blessing method of absorption correction to the reflections. You may also include faces. Click "OK".





5. A new window will appear asking you to choose a space group. Choose the space group that is consistent with your previous choices. And press ok



6. Inspect the Rint and F2/sig(F2) to see how well the crystal diffracted vs resolution. Press OK

Data reduction	file contents	Data re	duction outp	ut	Red graphs	1	Data collection	output	Devices	log
1.31-1.14	630	238	235	98.7	2.7	2612.43	34.69	0.025	0.024	
1.14-1.04	674	238	235	98.7	2.9	1484.11	25.73	0.034	0.031	
1.04-0.96	621	236	235	99.6	2.6	1117.68	21.08	0.041	0.040	
0.96-0.90	525	243	235	96.7	2.2	796.22	15.73	0.049	0.050	
0.90-0.85	438	245	236	96.3	1.9	528.67	11.31	0.063	0.068	
0.85-0.82	405	249	235	94.4	1.7	437.49	9.54	0.072	0.085	
0.82-0.78	343	295	235	79.7	1.5	369.51	7.22	0.070	0.102	
0.78-0.70	256	897	235	26.2	1.1	255.87	4.77	0.087	0.145	
inf-0.70	5137	3123	2351	75.3	2.2	2463.39	27.07	0.022	0.022	
inf-0.70 inf-0.80 Statistics resolu- tion(Å)	5137 4709 vs resol # kept	3123 2055 ution (ta # theory	2351 1992 king redu # unique c	96.9 ndancy	2.2 2.4 into account) average redundancy	2463.39 2660.11 - Laue g mean F2	27.07 29.02 group (anom mean F2/sig(F2)	0.022 0.022 alous pa Rint	0.022 0.022 irs merged): RsigmaB	Primi
inf-0.70 inf-0.80 Statistics resolu- tion(Å)	5137 4709 vs resol # kept	3123 2055 ution (ta # theory	2351 1992 king redu # unique c	96.9	2.2 2.4 into account) average redundancy	2463.39 2660.11 - Laue g mean F2	27.07 29.02 group (anom mean F2/sig(F2)	0.022 0.022 alous pa Rint	0.022 0.022 irs merged): RsigmaB	Pratan
inf-0.70 inf-0.80 Statistics resolu- tion(Å) inf-1.73 1 73-1 33	5137 4709 # kept 514 664	3123 2055 ution (ta # theory 151 150	2351 1992 king redu # unique c 150	75.3 96.9 indancy tomplete 99.3	2.2 2.4 into account) average redundancy 3.4 4.4	2463.39 2660.11 - Laue g mean F2 10596.76 3997 05	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57 61	0.022 0.022 alous pa Rint 0.013 0.020	0.022 0.022 irs merged): RsigmaB 0.010 0.015	Pmm
inf-0.70 inf-0.80 Statistics resolu- tion(Å) inf-1.73 1.73-1.33	5137 4709 vs resol # kept 514 664 657	3123 2055 # theory 151 150	2351 1992 king redu # unique c 150 150	75.3 96.9 mdancy = * omplete 99.3 100.0	2.2 2.4 into account) average redundancy 3.4 4.4	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05 2640.00	27.07 29.02 mroup (anom mean F2/sig(F2) 76.56 57.61 44 50	0.022 0.022 alous pa Rint 0.013 0.020 0.029	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020	Pranan
inf-0.70 inf-0.80 Statistics resolu- tion(Å) inf-1.73 1.73-1.33 1.33-1.15	5137 4709 ws resol # kept 514 667 726	3123 2055 # theory 151 150 150	2351 1992 king redu # unique c 150 150 150	75.3 96.9 mdancy : * omplete 99.3 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05 2640.00 1529.26	27.07 29.02 mean F2/sig(F2) 76.56 57.61 44.50 33.40	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025	Pranan
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 # kept 514 664 657 726 662	3123 2055 # theory 151 150 150 150	2351 1992 # unique c 150 150 150 150	75.3 96.9 * * * * * * * * * * * * * * * * * * *	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.4 4.8	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.025 0.025 0.033	Prana
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 1nf-1.73 1.73-1.33 1.73-1.33 1.33-1.15 1.35-1.03 1.03-0.95 0.95-0.89	5137 4709 vs resol # kept 514 664 657 726 662 545	3123 2055 # theory 151 150 150 150	2351 1992 # unique c 150 150 150 150 150	75.3 96.9 * * * * * * * * * * * * * * * * * * *	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.8 4.8 4.6 3.6	2463.39 2660.11 - Laue o mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91	27.07 29.02 froup (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.025 0.033 0.044	Pmm
inf-0.70 inf-0.80 Statistics resolu- tion(Å) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.95-0.89	5137 4709 vs resol # kept 514 664 657 726 662 545 464	3123 2055 # theory 151 150 150 150 150 150	2351 1992 king redu # unique c 150 150 150 150 150 150	75.3 96.9 * * 99.3 100.0 100.0 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.4 4.8 4.4 4.4 3.6 3.1	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.047 0.058 0.073	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062	Pmm
inf-0.70 inf-0.80 Statistics resolu- tion(A) 	5137 4709 # kept 514 664 657 726 662 545 464 405	3123 2055 ution (ta # theory 151 150 150 150 150 150 150	2351 1992 # unique c 150 150 150 150 150 150 150	75.3 96.9 somplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7	2463.39 2660.11 - Laue c mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58	0.022 0.022 alous pa Rint 0.013 0.020 0.038 0.047 0.058 0.073 0.073	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075	Pranam
inf-0.70 inf-0.80 Statistics resolu- tion(Å) inf-1.73 1.73-1.33 1.33-1.15 1.15-1.03 1.03-0.95 0.85-0.89 0.89-0.84 0.84-0.81	5137 4709 vs resol # kept 514 664 657 726 662 545 464 405 299	3123 2055 # theory 151 150 150 150 150 150 150 150 150	2351 1992 king redu # unique c 150 150 150 150 150 150 150	75.3 96.9 somplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3	2.2 2.4 into account) average redundancy 3.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7 2.0	2463.39 2660.11 - Laue c mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93	0.022 0.022 alous pa Rint 0.013 0.020 0.038 0.047 0.058 0.073 0.073 0.073	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075 0.072	Pranan
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 514 664 657 726 662 545 464 405 299 201	3123 2055 # theory 	2351 1992 # unique c 	75.3 96.9 * * * 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 100.0 84.3 34.6	2.2 2.4 into account) average redundancy 3.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3	2463.39 2660.11 - Laue c mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01	0.022 0.022 alous pa 0.013 0.020 0.029 0.038 0.047 0.058 0.073 0.073 0.073	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075 0.092 0.135	Prata
inf-0.70 inf-0.80 Statistics resolu- tion(Å) 	5137 4709 vs resol # kept 514 664 657 726 662 545 545 464 405 299 201 5137	3123 2055 ution (ta # theory 151 150 150 150 150 150 150 150 150 150	2351 1992 # unique c 150 150 150 150 150 150 150 150 150 156	75.3 96.9 mdancy * omplete 99.3 100.0 100.0 100.0 100.0 100.0 100.0 100.0 34.6 82.3	2.2 2.4 into account) average redundancy 3.4 4.4 4.4 4.4 4.8 4.4 4.8 4.4 3.6 3.1 2.7 2.0 1.3	2463.39 2660.11 - Laue g mean F2 10596.76 3997.05 2640.00 1529.26 1130.72 733.91 496.67 426.19 372.28 229.02	27.07 29.02 group (anom mean F2/sig(F2) 76.56 57.61 44.50 33.40 27.07 19.15 14.03 11.58 8.93 5.01	0.022 0.022 alous pa Rint 0.013 0.020 0.029 0.038 0.073 0.073 0.073 0.073 0.095	0.022 0.022 irs merged): RsigmaB 0.010 0.015 0.020 0.025 0.033 0.044 0.062 0.075 0.092 0.135 	Prata

7. Click the OLEX2 button on the left toolbar to

will be in the struct folder under



start solution and refinement. All your files needed

olex2_XXXXX





D. IMPORTANT FILES

- Frames directory has the images ٠
- XXX.par file has the experiment for the instrument. (this is the one you open when starting CAP) ٠
- XXX.rrprof is the data integration results •
- XXX.run has the scans table for the data collection ٠
- XXX.cif_od has all the experimental data for your cif file ٠
- Log directory has all current experiment log files ٠
- Quick overview of control buttons •

image list 👻	lmage list	(es.)	Find hkl
K	Previous run	*	Look up table
E	Jump back 10 frames	•	Colour table
	Previous image	(\cdot)	Zoom in
	Play/stop image movie	Θ	Zoom out
	Next image	(\cdot)	Zoom localiser window
	Jump forward 10 images	×	Resolution rings
	Next run		2D Peak profile (a line profile)
🛞 -	Predictions		3D Peak profile (rocking curve)
0	Pixel / area information	• (?)	Help
K	Image header information	CCD 🔻	View (CCD/RED/USER)



PUBLICATION

A. EXPERIMENTAL SECTION

Project-specific details and setup information are saved in the '*.cif od' file located under your project folder. Modify the text below according to the setup and conditions you used during the measurement:

"Intensity data of a (color and shape) single crystal of (project name) were collected at XXX(Y) K. A suitable single crystal with dimensions of X×Y×Z mm³ was mounted on a (loop | MiTeGen loop | glass fiber | etc.) with (paratone oil | qlue | grease | etc.) on an XtaLAB Synergy diffractometer equipped with a (micro-focus sealed X-ray tube PhotonJet (Mo) X-ray source | micro-focus sealed X-ray tube PhotonJet (Cu) X-ray source | micro-focus rotatinganode X-ray tube Rigaku (Cu/Mo) X-ray source) and a Hybrid Pixel Array Detector (HyPix) detector. Temperature of the crystal was controlled with an Oxford Cryosystems low-temperature device. Data reduction was performed with the CrysAlisPro software using an (empirical | numerical) absorption correction. The structure was solved with the (SheIXT | SheIXD | SheIXS | etc.) structure solution program using (the Intrinsic Phasing | direct methods | Patterson | Dual space | charge flipping) solution method and by using (Olex2 | Jana2006 | ShelXle | etc.) as the graphical interface. The model was refined with (SheIXL | Jana2006 | etc.) using least squares minimization."

B. ACKNOWLEDGEMENT

Use was made of the IMSERC X-ray Facility at Northwestern University, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-1542205); and Northwestern University.





TROUBLESHOOTING

- 1. If the goniometer does not move, then ensure that the door lock button is enabled or you are holding down the "motion enabled" buttons in the enclosure
- 2. Restart CAP to home the goniometer

REVISIONS

Reformatted according to the latest template. Sections about 'Safety', 'Da	ata
management', 'Software', 'Publication', and 'Troubleshooting' were added	
Section about 'Crystal mounting under a microscope' was added	
Data analysis section was expanded	
Release of original version of the user manual	
	Reformatted according to the latest template. Sections about 'Safety', 'Da management', 'Software', 'Publication', and 'Troubleshooting' were added Section about 'Crystal mounting under a microscope' was added Data analysis section was expanded Release of original version of the user manual

