CrysAlis^{Pro}



Getting Started

1. Launch the online version of CrysAlis^{Pro} by pushing the blue CrysAlis^{Pro} icon. - 0 START/STOP Shutter X-ray Cu Closed 2. Wait for the instrument to initialize and show 'Ready'. X-RAY STATUS CRYO CAM)) CCD Ready RED Ready 3. You should be in Small Molecule mode. Rigaku Click once on this icon if it shows 'PX'. oxford diffraction CRYSALIS 4. Click on 'START/STOP' Start new Start new (no pre-experiment) Resume all / pre-experiment; recalculate strategy Resume data collection only 5. Click on 'Start new'. Append data collection Other experiments / de-icing Rigaku Copyright © 2015 — Rigaku Corporation and its Global Subsidiaries. All Rights Reserved. oxford diffraction

The Crystal Screening Panel Opens ...

START/STOP
Closed X-ray Mo 4 x 4
SM Screening Screening Mount Screen = 5.0s
Experiment - Complete data for publication
Name: exp_1 Detector=53.0mm, Res. = 0.800Ang, I/sig =15.0, width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 5.0s
Exposure time: 5.0 s
What is this? Pre-Exp. (2 m) Edit
Goniometer Omega Theta Kappa Phi Distance -21.5 -21.5 0.0 0.0 60.0



Click on Edit

First, Setup User Info !!

	Pre-experiment	CRYSALIS ^{Pro}	
1. Open File	Path and user / Sample	Experiment performer:	
Explorer	Name: exp_1 Experiment: exp_1 in folder C:\kcaliburData\exp_1 Path is ok! Browse root folder >> C:\kcaliburData	Setuser	
	Expected chemical formula: AutoChem 4 may not suc providing valid chemic	cceed without cal formula! Get Last used formula	
	Comment:	Sample description	
	Experiment options Exposure time:	ie, αγο off, Strategy: Complete data (default mode), , Pre-experiment Finish: Thu Dec 13 17:34:42 2018	
	Type of experiment Complete data for publication	on 💌 Setup >>	
	I/sig 15.0 Resolution 0.837		
	✓ Interactive strategy after pre ✓ Attempt AutoChem	ntal)	3. Click Exit
	Help Exit & start screening	Exit & start preexperiment Exit	

2. Browse to an existing folder or create new folders to something as shown below.







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Second, Mount & Center a Crystal

1. Click 'Mount sample' to open the mounting & centering window.



2. Mount the crystal & center to the crosshairs in several Phi orientations. Click on these buttons or use arrow keys shortcuts to switch between 0, 90, 180 and 270° Phi orientations.

The crystal must be centered as well as possible. This picture shows an example of a needle that is centered exactly to the crosshair.

3. Click 'Exit' or press 'ESC" on the keyboard.





Third, Enter Your Screening Parameters

START/STOP
Shutter X-ray 4 x 4
CAM) (CRYO) (X-RAY) (STATUS)
265 h I
SM Screening
Screening
Mount Screen = 5.0s
Experiment - Complete data for publication
Detector E2.0mm Ros = 0.0004mg Main =15.0
width=1.0deg, Movie, cryo off, Strategy: Complete data (default mode), Exposure: 5.0s
Exposure time: 5.0 s
What is this? Pre-Exp. (2 m) Edit
Goniometer
-21.5 -21.5 0.0 0.0 60.0



- If you click directly on 'Screen', 10 frames will be
 collected in a single crystal orientation and using the displayed exposure time.
- 2. To change the parameters, click here. Then change the exposure time and # frames in the next dialog.

Approx. theta	а	35.Q N	ax res.: 0.553	
hi offset to current position:	● 0	C 20	C 40	C 60
ican axis:	C Phi	Omega		
etector distance:	Close	C User	52.0	mm
	Use the	same distance	for pre-experin	nent
Exposure time				
Exposure time			5.0 Prope	r dark is not existing
eatures of screening		+		
Frames used		— 10 frame	:(s)	
🗖 Reset unit cell	F Use 'ur finding	m ttť instead o I	f 'Fast UB sear	ch' during unit cell
lardware settings				
		Generator		
information				
			the second se	the second se

3. Click here to start screening.

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Examine the frames

Criteria for a good diffraction pattern:

- 1. <u>Well-defined, single spots.</u> If some spots appear split, you may have a cracked or twinned crystal.
- 2. <u>Ordered, non-intertwined array of</u> <u>spots.</u> Rows of spots crossing each other (intertwined) are indicative of multiple diffraction patterns. You may have mounted more than 1 crystal.
- 3. <u>No rings of diffraction.</u> If there are, then you have either mounted a polycrystalline sample or there is ice on the crystal.
- 4. <u>Strong enough diffraction.</u> Spots should be visible to 0.8-0.9 Å.

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If 1.-3. are not satisfied, click 'Start new' again and screen another crystal.
 If 4. is not satisfied, increase the exposure time.

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Use the Toolbar Functions





Check the Auto-Indexing Results





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Run 'What is this?' experiment or 'Pre-experiment'



Choose **What is this?** if the aim is to obtain a quick structure (quality control, filtering out starting materials ...). This features will collect a partial data set (60-70% completeness) with a target $< I/\sigma(I) > of 5$ to 1 Å. Choose **Pre-experiment** if you plan to collect a full data set to IUCr standards. For this option, go to Page 12.



Setting up a 'What is this' experiment?



Pre_experiment

The 'Screening' step is really a way to have a quick X-ray snapshot of the crystal diffraction (quality, intensity, preliminary unit cell).

A more sophisticated screening is done via 'Pre_experiment', whereby a few orthogonal scans, each of 10 frames, are collected. This will yield a better indexing, orientation matrix and $<I/\sigma(I)>$ estimate, leading to a more accurate data collection strategy.

Name:	exp_1					-	
Detecti width=" (default	or=53.0mm, 1.0deg, Mov t mode), Exp	Res. /ie, crj posure	= 0.837/ yo off, Si : 1.0s	Ang, I/sig trategy: C	.=15.0, omplete	data	
Exdos	ure time:	4-		-	-	1.0 s)

- This exposure time is selected by CrysAlis^{Pro} using the <I/σ(I)> estimated upon screening. Click on the button to change the exposure time. Note that this number is a rate per degree. Since the frame width is 0.5°, an exposure time of 0.5 second per frame would actually be used.
- Click 'Pre_Exp'. In parenthesis, the estimated time for the pre_experiment is shown. Concurrent indexing takes place during the pre_experiment. Check the indexing results in the right hand side panel, as described on Page 9.



The Strategy Module Opens ...

And a strategy is automatically calculated, but you may want to change some of the parameters ...

Outcold of using (Sublading Contracting) Control to the optical (Sublading Control to the optical optica	Experiment Strategy		CRYSALIS
Strategy parameters © Resolution C Theta C Ztheta 0.337 © Laue group C Other mmm • © Frield mates are equivalent (uncheck for high quality absolute configuration data) Detector Distance 53.00 Strategy mode Complete data (default mode) • Int 100.0 UCClimit Max 99.9 % Generates runs that reach complete mest frames: 4/178 Total experiment finish time: Fri Dec 14 14:06:18 2018 © Completeness/Coverage tables Completeness in mmm 3.9 3.6 3.2 3.0 0 0 0 0 0 0 0 0 0 0 0 0 0	Cell: 5.971(2) 9.042(3) 18.365(10) 90.06(4) 90.09(4) 90.13(3) 991.6(7) oP	P-lattice 98.59% (210 of 213 reflections)	Lattice Wizard
Imit 100.0 Identified Max 99.9 % Generates runs that reach completeness limit Automatic experiment settings Options Current Strategy No. runs/frames: 4/178 Total experiment time: 0h 06m Expected experiment finish time: Fri Dec 14 14:06:18 2018 Calculate New Strategy Update Completeness Manually Edit Run List * Completeness/Coverage curves * Completeness/Coverage tables Full sphere (P1) 100 100 3.9 3.0 100 100 100 3.2 70 100 100 100 3.4 0.2 0.0 0.0 100 3.4 0.0 0.0 0.0 100 100 100 100 100 100 100 2.8 0.0 0.0 0.0 0.0	Strategy parameters	Time prediction based on data to 0.837 Ang exp time • Fill time • Fill I/sigma • Different time for each theta positions • Different time for each theta positions • Predicted resolution beyond 0.84 • Scan width: 1.00 • Use theta-de • Otherapy the second of	ndividual merged T/cirma: 17.12: 17.12: 17.12: 17.12: 17.12: 17.12: 15.36 4x4 35.36 ependent binning/SSC V
Completeness/Coverage curves Completeness/Coverage tables Completeness in mmm 100 - 100 -	Generates runs that reach completeness limit Current Strategy No. runs/frames: 4/178 Total experiment time: 0h 06m Expected experiment finish time: Fri Dec 14 14:06:18 2018 Update Completenes	Automatic experiment settings Base binning: 4x4 Uncorrelated frames Automatic experiment settings Automatic experiment settings Manually Edit Run List	s chem/Movie/Cnyo/Red
$\begin{array}{c} 1.4 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ $	Completeness/Coverage aurves Completeness/Coverage tables Completeness/in mmm 3.9 3.6 3.2 2.8 5 5 5 5 5 5 5 5 5 5 5 5 5	Full sphere (P1)	Redundancy for coverage



Open the Lattice Wizard to Check/Improve Indexing

Unless 90-100% reflections are already indexed, it is recommended to re-index the diffraction pattern manually. Open the Lattice Wizard from the strategy interface by clicking on **Lattice Wizard** at the top right corner of the interface.





Peak Search with User Settings

- **Traditional peak hunting**: Each frame pixel is scanned and compared to the 'Threshold'. If above the 7x7 average is checked to be higher. If yes, some further 2D peak shape tests are made and the pixel is accepted if passed. These are the 2D peak locations. In a further algorithm step, these 2D locations are assembled to 3D peaks. This method works well when no background is present (for high background. use the background subtraction). Its benefit is speed, but the 3D assembled peaks are not as accurate as the 3D profiles.

- **Automatic background and threshold detection**: This is an extension of the 'Traditional peak hunting' in that it finds automatically the approximate background and according to the findings adjusts the thresholds. For speed, some shortcuts are made.

- **Smart peak hunting**: This method was first developed for proteins and is useful for weaker data. It uses local background to cope with the situation of small peaks on strongly varying background. Slower than traditional and automatic peak hunting.

- **3D peak extraction**: This method is based on the dc proffit 3D profile learning routines. It locates potential peaks and then extracts 3D profiles from it to compute an accurate 3D centroid. The slowest method, but excellent profile position information and better at extracting reflections from weakest data.



Lattice Wizard Manual Functions

(Big button = auto function, small arrow button = options)



Peak search



Find the best unit cell compatible with current peak table



Refine parameters and instrument model to improve the fit



Index with current cell to improve the fit



Potential fits to other Bravais lattices from the same primitive unit cell



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Access to a reciprocal space viewer with some additional features



Adjust the Strategy

1. When closing the Lattice Wizard, you are asked whether an updated strategy should be calculated, Click on YES.

u ategy parame	ers				
Resolution	C Theta C 2	ZTheta 🔽 🤇	0.837		
• Laue group	C Other	mmm	•		
Friedel mat	es are equivalent	t (uncheck for	high quality abso	olute configuration	data
Detect	or Distance	53.00			
trategy mode –					
Complete dat	a (default mode)]		
limit 100.0	UCr limit	Max 99.9	%		

3. Optional: select the desired Laue group if different from CrysAlis^{Pro} solution. You can also select a sphere, hemisphere or quadrant from the 'Other' pull-down menu in the option.

C Laue group	• Other	hemisphere	-	
		sphere		
		hemisphere		
		quadrant		

4. Check/uncheck to make the Friedel mates equivalent/merged.

Complete data (default mode)	-
Complete data (default mode)	
Complete redundant data	
Total time constrained data	
Complete total time constrained data	- 1
Absorption correction	
Coverage data	
Complete data for twins	_

5. Optional: change the Strategy mode according to the type of experiment planned. An explanation of the strategy type selected is given in the panel underneath.



Adjust the Strategy (continued)

6. Optional: change the rotation width if you want finer or wider slicing. Don't forget to change the exposure time accordingly to keep the same rate per degree.

Strategy parameters	Time prediction based on data to 0.837 Ang	
Resolution C Theta C 2Theta 0.837	Fill time concretated) Tristmer Tristmer (Incorrelated) Tristmer Trist	
 ✓ Laue group ✓ Other mmm ✓ Friedel mates are equivalent (uncheck for high quality absolute configuration data) 	The same time for all theta positions exp time individual merged th Different time for each theta positions (uncorrelated) 1/sigma. 1/sigma	eta nina:
Detector Distance 53.00 Advanced	I-8.39: 8.551 I.00 I7.12 35.36 45 Predicted resolution beyond 0.84 Total I/siama: 17.12 35.36 45 Scan width: 1.00 Use theta-dependent binning/SSC Use theta-dependent binning/SSC Image: State Sta	(4 7
Strategy mode		
Complete data (default mode)		
limit 100.0 UCr limit Max 99.9 %		
Generates runs that reach	Automatic experiment settings Options	
completeness limit	Base binning bx4 Uncorrelated frames Autochem/Movie/Cryo/Re	J
Current Strategy		
No. runs/frames: 4/178		2
Total experiment time: Oh O6m	Manually Edit Run List	
Expected experiment finish time: Fri Dec 14 14:06:18 2018		

- 7. Optional: the exposure time was calculated by CrysAlis^{Pro} based upon a user-input target $<I/\sigma()>$ (15 is a good target for medium to well-diffracting crystals). You can change it at will, and the data collection time will be updated automatically.
- Time prediction based on data to 0.837 And 8. Optional: click on 'Fill I/Sigma' and C Fill time Fill I/sigma enter a new $<I/\sigma(I)>$ target. The 1.00 17.12 35.36 C The same time for all theta positions individual exp time merged theta exposure time in the box on the left Different time for each theta (uncorrelated) I/sigma I/sigma: hinning 30 61.96 3.07 [-8.39: 8.55] 2x2 will automatically be updated. Predicted resolution beyond 0.84 29.99 61.94 Total I/sigma: 1.00 Scan width: Use theta-dependent binning/SSC 🔽 Rigaku

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View the Predicted Completeness/Redundancy

View as a graph



View as a table

		Complet	teness (un	der Laue	symmetry)	C	overage	(under P	1)		
Res		#Data	#Theory	8	Redundancy	#Total	#Data	#Theory	% Re	dundancy	#Tota
18.36-	1.94	100	107	93.46%	4.1	406	424	706	60.06%	1.2	516
1.94-	1.49	107	107	100.00%	4.5	482	444	706	62.89%	1.2	523
1.49-	1.30	107	107	100.00%	5.3	571	505	706	71.53%	1.2	610
1.29-	1.17	107	107	100.00%	5.2	552	439	706	62.18%	1.2	507
1.16-	1.08	107	107	100.00%	4.7	498	389	706	55.10%	1.2	474
1.08-	1.00	107	107	100.00%	3.9	420	339	706	48.02%	1.2	398
1.00-	0.96	107	107	100.00%	3.7	394	313	706	44.33%	1.2	362
0.95-	0.91	107	107	100.00%	2.8	296	260	706	36.83%	1.1	276
0.91-	0.87	107	107	100.00%	2.6	276	252	706	35.69%	1.0	260
0.87-	0.84	113	113	100.00%	2.6	291	240	706	33.99%	1.1	260
18.36-	0.84	1069	1076	99.35%	3.9	4186	3605	7060	51.06%	1.2	4186



Calculate a New Strategy and review the Scans

1. Once you have adjusted all the parameters, click 'Calculate New Strategy'.

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Parameters for Automatic Structure Solution



Start Data Collection

- 5. Check the predicted finish time for the experiment.
- Current Strategy No. runs/frames: 4/178 Total experiment time: 0h 06m Expected experiment finish time: Fri Dec 14 14:06:18 2018

6. Click on 'Start Named Experiment to access the 'Special collection' dialog.



	Special collection	SALIS
7. Enter a name for the experiment.	File name and path Name: exp_1 Experiment: exp_1 in folder C:\XcaliburData\YourLab\YourName\exp_1 Clear folder Browse root folder SC:\XcaliburData\YourLab\YourName	Welcome –
 Enter the chemical formula or chemical content, if known. 	Expected chemical formula: Comment: Information The experiment name in folder: C: \\CaliburData\YourLab\YourName\exp_1\exp_1 is already existing but only pre/scr was performed (1 180/0) Cancel	formula cription frames all/done

9. Click 'Start'. 60 pictures, one every 6° along phi, will first be taken of the crystal. Then, concurrent data reduction and structure solution will take place during data collection.



Data Processing Flow Chart





End of Data Collection



At the end of data collection, the interface should look like the above:

- The last diffraction frame is displayed in the main panel
- The current structure along with the current refinement statistics are displayed in the right hand side panel.
- A summary of data reduction statistics can be viewed by clicking on the 'Data Reduction' tab (see Page 26).



Relying on the Automatic Processing from CrysAlis^{Pro}



Data Processing Results





Data Inspection Module

In the Power Tools panel on the left to open the Data Inspection Module.

2. Check statistics tables

1. Click

3. Check statistics graphs (see next 2 pages)

 Click Refinalize to open the scaling options dialog and apply different types of absorption correction. –



	Inspec	ct data co	llectio	n and ı	reductio	n results				CrysAlis
Data reduction file	contents D	ata reduction outpu	ut	Red graphs) Data	collection output	Devices	log		
inf 1.73	602	212	201	94.8	3.0	613941.56	78.97	0.012	0.010	^
1.73-1.38	640	202	202	100.0	3.2	239196.36	38.22	0.024	0.022	
1.38-1.19	685	202	202	100.0	3.4	141150.30	27.98	0.034	0.032	
1.19-1.09	534	201	201	100.0	2.7	110467.96	20.21	0.038	0.040	
1.09-1.01	488	201	201	100.0	2.4	61954.57	15.18	0.056	0.056	
1.01-0.95	432	202	201	99.5	2.1	53465.93	11.84	0.057	0.069	
0.95-0.90	321	201	201	100.0	1.6	34738.53	7.07	0.087	0.101	
0.90-0.87	307	206	201	97.6	1.5	25069.78	5.77	0.095	0.125	
0.87-0.23	317	217	202	93.1	1.6	20882.40	5.16	0.099	0.136	
0.83 0.76	289	535	205	38.3	1.4	15015.75	3.63	0.175	0.182	
inf-0.76	4615	2381	2017	84.7	2.3	165004.16	26.26	0.025	0.027	
inf-0.84	4237	1782	1758	98.7	2.4	178268.14	28.25	0.024	0.025	
Statistics resolu- tion(A)	vs resol # kent	ution (taki # theory u	ng redu # unique c	ndancy i % omplete	nto accour average redundanc	nt) - Laue (mean :v F2	group (anoma mean F2/sig(F2)	alous pa Rint	irs merge RsigmaB	ed): Pmmm
inf-1.81	509	130	121	93.1	4.2	646522.19	104.76	0.013	0.008	
1.81-1.42	593	121	121	100.0	4.9	299231.35	54.24	0.022	0.015	
1.42-1.23	707	121	121	100.0	5.8	134075.97	38.29	0.039	0.026	
1.23-1.11	532	121	121	100.0	4.4	122903.60	27.93	0.039	0.032	
1.11-1.02	540	121	121	100.0	4.5	70957.51	22.59	0.054	0.044	
1.02-0.96	437	121	121	100.0	3.6	52718.54	15.41	0.066	0.060	
0.96-0.91	346	121	121	100.0	2.9	38593.07	11.07	0.085	0.084	
0.91-0.87	317	121	121	100.0	2.6	26743.80	7.74	0.108	0.110	
0.87-0.83	326	125	121	96.8	2.7	21721.65	6.99	0.103	0.128	
0.83-0.76	308	310	128	41.3	2.4	14696.40	4.87	0.176	U.162	
inf-0.76	4615	1412	1217	86.2	3.8	165004.16	33.89	0.028	0.025	
inf-0.84	4237	1075	1066	99.2	4.0	178268.14	36.44	0.027	0.024	
Refinalize	Crystal mov	vie								ок

Data Inspection Module – Scale Factors per Frame

- The 1st useful plot to look at is that of the scale factors per frame. It should always be smooth, i.e. not jittery as scale factors (dependent on the average diffraction intensity per frame) should remain almost identical for consecutive frames.
- 2. The values are likely to change for different runs, as different orientations of the crystal will vary the average intensity per frame and thus the scale factors.



3. Scale factors between runs will vary more for anisotropic crystals (needles, rods, plates) than for isotropic crystals (blocs, cubes, rounder crystals), as different amounts of crystalline matter are in the beam for different crystal orientations. For the same reason, for anisotropic crystals, scale factors may slowly vary over wide ranges of rotation ... but remember, this variation must remain smooth.



Data Inspection Module – R_{int} per Frame

- The 2nd plot to look at is that of the R_{int} per frame. Open the pulldown menu at the bottom of the interface, scroll down and select 'Rint'.
- This plot is always jittery, as shown on the picture. You only want to identify obvious outliers, i.e. single frames whose R_{int} will be grossly greater than its neighbors'. In this example, the 3 frames within the red circle may barely be considered outliers.



3. If single frames or parts or full runs are identified as bad according to their scale factor or R_{int} values, you may reject them by filtering them out (see page 31). However, ensure that data completeness does not go below the minimum required by the IUCr for publications as a result.



Refinalize Module - Crystal Data

Experiment: Ylid Unit cell: 5.9665 9.0	475 18.3739 90.0 90.0 90.0 991.8518 (CSD: install)	
. Enter chemical formula and Z, if not already done.	 Ensure the correct Laue group is shown. 	



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Refinalize Module – Filters and limits



<- Add

Check

 In the filters dialog, click '< Add' and ∠ expand the list of available filters.

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OK

Cancel

Refinalize Module – Filters and limits (2)

- 5. The most commonly used filters are:
 - 'Run filter': removes entire runs from scaling.
 - 'Run frame filter': allows to define a range of frame within a run to be removed from scaling.
 - 'Rint-frame filter': single frames with R_{int} > threshold are removed from scaling.

For instance, this picture shows how single frames with $R_{int} > 50\%$ would be removed:

- 'Rint-frame filter' selected.
- Condition is 'greater than'
- Threshold is 0.5, i.e. 50%. The threshold must be entered as a fraction.
 Upon clicking 'OK', that filter is added to the list. Several filters may be used at once.



Please	enter filter type, rejection relation and	thresho	old value
Type of filter	'Rint-frame' filter		•
Condition:	> (greater than, GT)	•	0.5
	RINT GT 0.50000		
		Add and	other condition
Help		Cancel	ОК

6. Run Refinalize by clicking 'OK' at the bottom of the dialog and check for best R_{int} and $<I/\sigma(I)>$.



Refinalize Module – Absorption Correction

1. Click here to access the empirical absorption correction options.

Empirical correction	Automated	Manual				
Frame scaling:	Auto C Set:	4 frames = 1 scale	Absorption harmonics:	C Auto	🙆 Set:	8 🕶 0 💌
🗂 Sample decay		C	Detector correction	4x4		Advanced
Numerical absorption	Faces	Sphere	1			

- Optional for mediocre data sets: check this box and vary the array dividing up the detector face into smaller areas for local scaling. <u>Check for best R_{int} and <I/σ(I)>.</u>
- Optional for mediocre data sets: select 'Set' and incrementally increase the coefficients for the even & odd orders of the spherical harmonics. <u>Check for</u> <u>best Rint and <l/σ(l)>.</u>



Refinalize Module – Absorption Correction (2)

 Click here to apply a face-based numerical absorption correction. However, you must first create a 3D model of the crystal via face indexing, provided crystal pictures were taken by the collection program. If so, see next 4 pages.

If not, skip numerical absorption correction.

2. You may run an Analytical or a Gaussian correction. <u>Check for best R_{int} and $<I/\sigma(I)>$ </u>. For Gaussian, also check this box. For crystals with at least one dimension larger than the X-ray beam, CrysAlis^{Pro} will take into account that only part of the crystal is bathed in the beam at once.



 If a 3D model of the crystal has been created and the crystal is fairly isotropic, click here to run a spherical absorption correction instead of face-based. <u>Check for best R_{int} and <I/σ(I)></u> to determine the best absorption correction procedure.

Face Indexing (1)

1. This can be done only if the collection program has taken visible pictures of the crystal.

in the Power Tools panel on the left to open the Data Inspection Module

2. At the bottom of the Data Inspection module, Refinalize Crystal movie click 'Crystal movie'. Add shape Preferences Center calibration File Log Faces # h k 1 d sise Face Marking C Drag C Point Snap Distance 3. In the 'Crystal shape' window, ensure 'Snap' Þ 6 d Ch is selected. O k C I Val. Step Show possible face normals - Settings ('Snap' mode only) =2 🔽 Snap to face normal Max. out of plane angle =3.5 Max. HKL

Face Indexing (2)

- 4. Select a crystal view that shows normal directions to crystal faces (doted lines). Different crystal orientations may be displayed using the mouse's wheel or the Prev/Next buttons underneath the frame. The crystal view can also be rotated by +/-90° or 180° at once using the corresponding buttons on the right of the picture.
- 5. Left-click near the crosshair and drag along a dotted line until the mark (in yellow on this picture) coincides with a crystal face.
- 6. Right-click on the picture and select 'Add face'.
- 7. In the new dialog, ensure this option is selected and click 'Add face' to validate the face.

Index selection	-0.222 -11.098 -5.097
C Integer hkl:	0-11-5
C Integer hkl small:	0 -5 -2
C Custom hkl:	0-2-1
 Integer hkl small (Sr 	nap mode): 0 -2 -1
Distance selection Measured distance	0.06476
C Custom distance:	0.06476

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Face Indexing (3)

- The new face is added to the list in the right hand side panel, along with its miller indices. A face highlighted in blue in the list is shown in red on the crystal picture.
- A good practice is to select a face view of the crystal first and create as many faces as possible. Then rotate by 90° and add more faces.

10. Once a few faces have been created, CrysAlis^{Pro} will come up with an idealized shape. However, it is likely to be imperfect at this point.

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Face Indexing (4)

11. Add faces from intermediate crystal orientations to carve out the shape so it fits best to the crystal. Also, you can move an existing face towards or away from the crystal to make it flush with a crystal edge by selecting it in the list and using the 'Distance' arrows.

<u>Note:</u> A * symbol preceding a face in the list means this face has become too small and should be deleted.

12. As the shape is being adjusted, the 3 dimensions of the crystal are automatically calculated by CrysAlis^{Pro}. If the correct chemical formula and Z have been previously entered, the crystal density and absorption coefficient μ are also automatically calculated by CrysAlis^{Pro}.

Refinalize Module – Manual Space Group Search

Space group and AutoChem			
Search for space group	Auto	Interactive	Space group options
AutoChem	- attempt structure soluti	on	AutoChem options

1. Click here to open the space group search dialog.

 Click here to switch from running GRAL (space group search algorithm) in silent mode (automatic) to interactive mode (manual).

 Click 'OK'. At the end of the next 'Refinalize' run, the space group search dialog will open. Note: GRAL is the equivalent of XPREP.

Re-processing Data Manually in an Offline Version of CrysAlis^{Pro}

Reprocess the Data Offline

Previously, we have refinalized data that have been <u>automatically integrated</u> by CrysAlis^{Pro}. The next, and final, section shows how to re-integrate data manually in CrysAlis^{Pro}, using user-input parameters, should you wish to do so.

 You must open a processing-only instance of CrysAlis^{Pro}. Manual data integration cannot be performed in an online instance. The easiest is to open File Explorer and browse to the desired CrysAlis^{Pro} experiment directory. Then double-click on the experiment_name.par file. CrysAlis^{Pro} will open and display the current status of the experiment.

2. Open the Lattice Wizard and perform manual indexing, if desired (as described on Pages 14-16). Then close the Lattice Wizard.

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Step 1: Lattice Review

oxford diffraction

Step 2. Frame Range Selection

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Step 3. Special Parameters

Proffit: CrysAlisPro data reduction assistant (1.0.28)	_	
Profile fitting data reduction		
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		
Orientation search range (max 10 deg) 1.00 Search steps/deg (max 10) 8		
	1	. Click 'Edit special pars' (next
Edit special pars		
		page).
	2	. Click these buttons to delete all
3d profile information and/or integration results on Empty trup folder from all analysis files including		info from provious propossing
the disk background		into non previous processing
Clear data from previous run		runs and start afresh.
	່ ວ	Click (Novt
< <u>Back Next> Cancel</u> Help	3	. UIICK INEXL.

Special Parameters Options

1. To cut off the data set, check this box and enter a resolution limit.

2. To reject the worst shaped reflections or reflections overlapping with ice rings, check this box.

3D intensity integration	Extra corrections
C 2D profile fitting (recommended only for very strong diffraction data)	Apply inverse float correction (f.ex. undo flood field correction)
 3D profile fitting (improves weaker data, default option) 	
Reflection positioning and integration	Apply hoat correction (r.ex. additional flood field correction)
Single wavelength only (recommended exclusively for data up to 1.5 Ang, i.e. large molecules) HKL check in 3D peak analysis (recommended	Apply pixelwise absorption correction (prepared by DC ABSTORUN)
when reflections are very close to each other)	Apply monitor renormalization
Skip filters	DC JETSHADOW (to visualize beforehand use 'beamstop mask')
Lorentz min = 0.0500 Edit Lorentz min	Use JetShadow Edit parameters
HP cell opening reject 0,00 Edit DAC angle	alr na: 30.00, beta: 0.00, jet_width: 13.00, jet_distance: 6.00
Use resolution limits Edit limits	
d-value (Ang): inf- 0.74	Profile fitting
	(generally not recommended, but 1.00 right)
Reject reflections with bad profiles (e.g. for HP data)	overlapping reflections e.g. twins)
I/sig > 10 & Profile agreement < 0.8	Follow profile size changes with incidence angle
Extinction rules	Adjust masks according to prediction uncertainty (for high angle data)
No extinction rules specified Show rules	Print average profiles to history window
HINT: You can use DC EXTINCT to add extinction rules and DC CLEAREXTINCT to remove selected or all rules from the list	
	OK Cancel

3. Check this box so the detector area covered by the cryo-nozzle shadow is removed from reduction. This must be done only if Cu radiation was used for data collection

4

Click OK.

Step 4. Background Subtraction

Proffit: CrysAlisPro data reduction assistant (1.0.28)

Profile fitting data reduction

Step 4: Background evaluation

- Background for 3D centroids

For an acurate evaluation of integrated intensities a good background determination is essential. Two parameters control this evaluation: The evaluation range Re and the repeat frequency Fr.

 Re = 50
 Edit Re
 Fr = 50
 Edit Fr

 Binning may reduce the memory requirements for the background evaluation. Default is 1. You may use 2 or 4 in case of lack of physical memory on your machine (risk of swapping)!

Required disk/memory space for background evaluation: 163.4/104.6 Mb

 Background for 3D integration

 Average background from 3D centroid evalutation (good for stable & low background, fast)

 Smart background (combination of local and average background computation, good for weaker data with high background and locally varying features, e.g. protein data, slower)

 Frame range = 1
 Edit range

 Memory usage:
 22.0 Mb

 Select 'Average background' if the X-ray background is low
 throughout the data set.

Select 'Smart background' if the Xray background is high and/or irregular throughout the data set.

If in doubt, run processing twice using one and then the other background option. Then check for best Rint and $< I/\sigma(I) >$.

2. Click 'Next'.

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Step 5. Outlier Rejection

Simultaneous twin data reduction	
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. Outlier rejection © Don't use outlier rejection 1 aP 7.16293 7.78051 11.20846 106.39120 90.56476 97.52915	 1. Ensure this box is checked and that the Laue group and
Use Friedel mates as equivalent Sack Next> Finish Cancel Help	correct.

Step 6. Output

 You must change the name of the final reflection file or else the reflection file created previously by CrysAlis^{Pro} upon automatic processing will be **overwritten**. It is recommended to keep the reflection file from automatic processing as a backup.

Click here and input a filename of your liking for the reflection file in the next dialog.

2. Enter chemical formula and Z, if not already done.

	Ouput	
Tip: Yo	u may change the output name and directory to keep results of da	ata reductions under different
parame Output	ter sets (UB, supercells) file name:	
C:\C\E)ata\Single_Crystal\Training-Agendas\Examples_Data\Twin_TTF	ET_TCNB_RT_02272014_r
Cha	nge output name	
- Finali	zation options	
Finali	zation options	
Finali	zation options Z Space group determination Automatic Ma	inual
Finali	zation options Space group determination Automatic Ma Automatic structure solution (AutoChem)	nual AutoChem option
Finali	zation options Space group determination Automatic Ma Automatic structure solution (AutoChem) Chemical formula not available	nual AutoChern option Edit formula
Finali F F	zation options Image: Space group determination Image: Automatic Image: Mage: Space group determination Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (AutoChem) Image: Automatic structure solution (Automatic structure solutio	AutoChern option

3. Click 'Finish' to exit the dialog and start processing. CrysAlis^{Pro} will reduce the data, determine the space group and scale the reduced data at once.

Re-finalize and Compare Statistics from Automatic and Manual Processing

- 1. Refer to pages 26-39 to refinalize the data manually integrated.
- 2. Click to open the Data Inspection Module. From the bottom pull-down menu, you can select any of the processing runs that have been completed and compare the statistics.

Data reduction file conte 1.73-1.38 1.37-1.19 1.19-1.09 1.09-1.01 1.01-0.95 0.95-0.90 0.90-0.87 0.87-0.83 0.83-0.76 inf-0.76 inf-0.84	Data reduction of 639 202 665 202 535 201 488 201 431 202 322 201 306 206 315 216 291 531	202 10 202 10 202 10 201 10 201 10 201 9 201 10	d graphs D 00.0 3.2 00.0 3.4 00.0 2.7 00.0 2.4	ata collection output 237867.26 141213.65 111038.66	Devices Io, 39.59 29.07	0.023	D.021	^
1.73-1.38 1.37-1.19 1.19-1.09 1.09-1.01 1.01-0.95 0.95-0.90 0.90-0.87 0.87-0.83 0.83-0.76 	639 202 685 202 535 201 488 201 431 202 322 201 306 206 315 216 291 531	202 10 202 10 201 10 201 10 201 10 201 9 201 10	DO.0 3.2 DO.0 3.4 DO.0 2.7 DO.0 2.4	237867.26 141213.65 111038.66	39.59 29.07	0.023	0.021	
1.37-1.19 1.19-1.09 1.09-1.01 1.01-0.95 0.95-0.90 0.90-0.87 0.87-0.83 0.83-0.76 	685 202 535 201 488 201 431 202 322 201 306 206 315 216 291 531	202 10 201 10 201 10 201 9 201 9	DO.O 3.4 DO.O 2.7 DO.O 2.4	141213.65 111038.66	29.07	0.034	_	
1.19-1.09 1.09-1.01 1.01-0.95 0.95-0.90 0.90-0.87 0.87-0.83 0.83-0.76 	535 201 488 201 431 202 322 201 306 206 315 216 291 531	201 10 201 10 201 9 201 10	00.0 2.7 00.0 2.4	111038.66		0.001	0.031	
1.09-1.01 1.01-0.95 0.95-0.90 0.90-0.87 0.87-0.83 0.83-0.76 	488 201 431 202 322 201 306 206 315 216 291 531	201 10 201 9 201 10	0.0 2.4	111000.00	21.02	0.036	0.038	
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0.95-0.90 0.90-0.87 0.87-0.83 0.83-0.76 	322 201 306 206 315 216 291 531	201 10	99.5 2.1	53472.75	12.20	0.055	0.067	
0.90-0.87 0.87-0.83 0.83-0.76 inf-0.76 inf-0.84	306 206 315 216 291 531		0.0 1.6	34826.36	7.28	0.086	0.099	
0.87-0.83 0.83-0.76 inf-0.76 inf-0.84	315 216 291 531	201 9	97.6 1.5	25012.46	5.85	0.096	0.12	
0.83-0.76 inf-0.76 inf-0.84	291 531	201 9	93.1 1.6	20999.12	5.25	0.098	0.14	
inf-0.76 inf-0.84		205 3	38.6 1.4	14981.31	3.65	0.184	0.186	
inf-0.84	4615 2374	2016 8	34.9 2.3	165038.04	27.27	0.023	0 026	
	4237 1782	1758 9	98.7 2.4	178303.74	29.34	0.023	.025	
Statistics vs	resolution (ts	aking redunda	ancy into acco	unt) - Laue g	roup (anomal	ous pairs	merged):	Pmmm
resolu- #	# #	# *	averag	e mean	mean			
tion(A) kep	pt theory	unique comp	plete redunda	ncy F2	F2/sig(F2)	Rint Rs	igmaB	
inf-1.81	510 130	121 9	93.1 4.2	647022.23	109.20	0.017	0.008	
1.81-1.42	592 121	121 10	0.0 4.9	297739.13	56.28	0.020	0.014	
1.42-1.23	707 121	121 10	5.8	134104.24	39.80	0.0/8 0	0.025	
1.23-1.11	533 121	121 10	0.0 4.4	123482.83	29.03	0. 37 1	0.031	
1.11-1.02	540 121	121 10	00.0 4.5	70953.14	23.39	0,052	0.042	
1.02-0.96	437 121	121 10	0.0 3.6	52697.76	15.89	0.063 0	0.059	
0.96-0.91	345 121	121 10	2.9	38655.39	11.37	.083 1	0.081	
0.91-0.87	317 121	121 10	2.6	26727.84	7.88	0.107 0	0.109	
0.87-0.83	326 125	121 9	96.8 2.7	21738.16	7.11	0.103	0.127	
0.83-0.76	308 306	127 4	41.5 2.4	14741.96	4.90	0.179	0.164	
inf-0.76	4615 1408	1216 8	36.4 3.8	165038.04	35.20	0.027	0.025	
inf-0.84	4237 1075	1066 9	99.2 4.0	178303.74	37.86	0.026	0.023	
Data reduction	n ended at Mon	Dec 17 17:55	5:35 2018					~
Refinalize								
·	Crystal movie		Ylid_Ma	nual				ок

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Where Are My Files?

Go to the experiment data directory:

≪ XcaliburData → YourLab → YourName → LY_20180508a →

- The raw diffraction frames are in the 'frames' directory.
- The visible crystal pictures are in the 'movie' directory.
- All log files for the current experiment are in the 'log' directory.
- The reflections files are named 'Experiment_name.hkl' (from automatic processing) or 'Your_Filename.hkl' (from your manual processing) and are located in the experiment directory. The input files for structure solution have the same filenames, but with the extension '.ins'.

10H93

- For structure solution, click select the desired .ins file in the next dialog. Upon opening a file for the 1st time, Olex2:
 - creates a 'struct' directory in the experiment data directory (alongside 'frames', 'movies' and 'log'), followed by a sub-folder name 'Olex2_ExperimentName'.
 - $\,\circ\,$ transfers the corresponding .hkl and .ins files to the Olex2 sub-folder.

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Typical SMX Workflow: Relying on automatic results

Typical SMX Workflow: after data collection, manual data reduction

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