

Kratzert Bruker Apex2 twin

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Context

- Dr. Daniel Kratzert reduced a Bruker Apex2 twin with CAP and Saint and gets significantly better results with saint and TwinAbs
- This presentation gives the workflow for importing and reducing Bruker data and handling twinning
- For the evaluation the forum version 38.43 was used.
- The results will be compare in table form.

Data set import

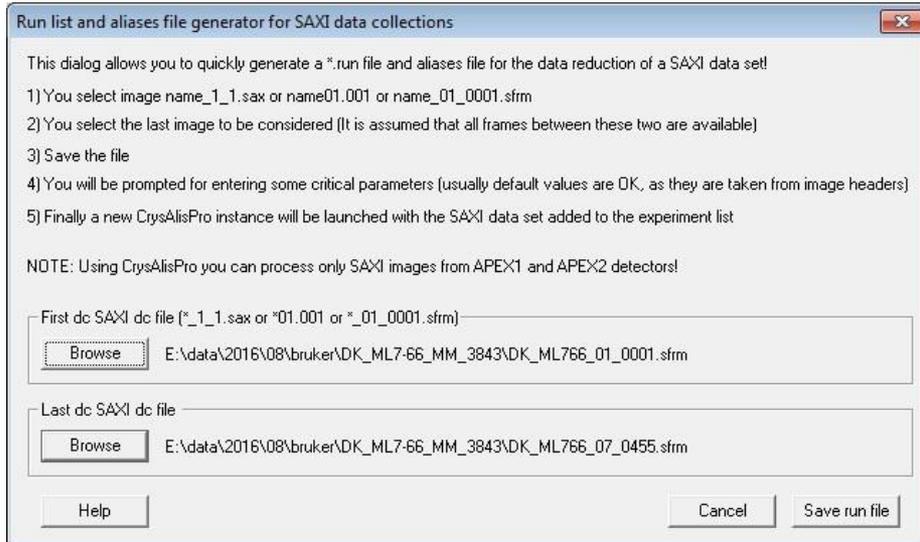
The screenshot shows a file explorer window with a list of files. The files are named with a pattern: DK_ML766_01_0001 through DK_ML766_01_0035. The columns are Name, Ext, Size, and Date. The files are sorted by Name. The file size is in sfrm format. The date is in YYYY-MM-DD format. The file extension is sfrm. The file size is in k. The date is in YYYY-MM-DD format.

Overlaid on the file explorer are two dialog boxes. The first is titled "Image import/export options (1.0.1)". It has a "CRYSTALIS Pro" logo and a "Im/Export" icon. It has two radio buttons: "Export" and "Import". The "Import" radio button is selected. Below the radio buttons is a section titled "Import options" with two radio buttons: "Known image format (with valid image headers)" and "Transform unknown image format to esperanto". The "Known image format" radio button is selected. There are "Help", "Cancel", and "Ok" buttons at the bottom.

The second dialog box is titled "Known format selector (1.0.1)". It has a section titled "Known formats with valid headers" with four radio buttons: "Dtrek (Rigaku)", "MAR/Rayonix", "Saxi (Bruker)", and "Dectris". The "Saxi (Bruker)" radio button is selected. There are "Help", "Cancel", and "Ok" buttons at the bottom. A note at the bottom says: "Note: Known formats with unsupported headers may be transformed to Esperanto format with an Esperanto importer." There is an "Esp. importer" button next to the note.

- Open any existing experiment
- Import button

Data set import



- Open any existing experiment
- Import button

Data set facts

- Mo, $I_{\mu\text{s}}$, no Si filter showing 3λ effect
- Frame width: 0.4, correlated ? Frames

CrysAlisPro run list data

Data collection mode: correlated

Total number of frames:2889 (scan: 2889, reference: 0)

Disk space: 763.79Mb

Approximate data collection time (h:min): 10:55

#	t	start	end	width	exposure	speed-rat	omega	theta	kappa	phi	# to do	# done	
1	o	1.386	184.186	0.400	5.000+	5.000	0.000	-	30.000	73.926	-0.814	456	456
2	o	-11.114	171.686	0.400	3.000+	3.000	0.000	-	17.500	73.926	4.186	456	456
3	o	21.376	163.775	0.400	3.000+	3.000	0.000	-	10.000	73.926	86.686	356	356
4	o	21.786	174.186	0.400	3.000+	3.000	0.000	-	20.000	73.926	-35.816	380	380
5	o	-28.114	126.686	0.400	5.000+	5.000	0.000	-	-27.500	73.926	11.685	386	386
6	o	18.386	179.186	0.400	4.000+	4.000	0.000	-	25.000	73.926	9.184	401	401
7	o	-7.815	174.186	0.400	3.000+	3.000	0.000	-	20.000	73.926	-60.814	454	454

First opening of the data set

- Select SM/PX nature

Open CrysAlis experiment (1.0.39) - 25 experiments available

Select experiment - standard list

Name	Path	Created	Accessed	Chemical
mrp160014	E:\data\2016\04\Newcastle Face Indexing\mrp160014_copy	Fri Feb 26 12:07:29 2016	Wed May 25 17:59:06 2016	C6 H8 O6
exp_66	E:\data\2016\04\warrick_movie_issue\exp_66_copy	Wed Apr 13 06:32:20 2016	Tue Jun 14 14:10:08 2016	???
jw2new	E:\data\2016\04\warrick_movie_issue\jw2new_copy	Sun Apr 10 08:13:28 2016	Thu Jun 16 18:40:29 2016	C20 H20 N
exp_48	C:\XcaliburData\Mathias_PX_screens\exp_48	Thu May 19 13:09:32 2016	Mon May 23 08:34:47 2016	???
150716_CudppaO2dppm	E:\data\2016\05\isaturn_fixedch\FixedChi-Saturn	Mon May 30 11:07:06 2016	Mon May 30 11:07:09 2016	???
150716_CudppaO2dppm	E:\data\2016\05\isaturn_fixedch\full	Mon May 30 11:20:22 2016	Mon May 30 11:20:26 2016	C300 H256
Cu_Near48mm	E:\data\2016\05\biberach\calib_XtaLAB_Mon-May-30-18-10-13-...	Mon May 30 16:11:22 2016	Tue May 31 09:57:45 2016	???
13ICR_L35_260x_2p25...	E:\data\2016\05\20160510_p300k_fix\unpack	Tue Jul 21 11:28:37 2015	Tue May 31 10:43:43 2016	C616 H963
pre_exp_5	E:\data\2016\05\biberach\email1\exp_5	Mon May 30 16:03:24 2016	Tue May 31 11:51:33 2016	???
test	E:\data\2016\05\biberach\Pierre	Tue May 31 08:51:38 2016	Tue May 31 13:33:58 2016	???
test2	E:\data\2016\05\biberach\BL_SecondDataCollection	Tue May 31 10:51:16 2016	Tue May 31 13:51:08 2016	???
data	E:\data\2016\06\Japan_Bruker_data\data	Fri Dec 18 05:03:58 2015	Fri Jul 15 10:38:45 2016	C27 H39
pre_exp_49	C:\XcaliburData\Mathias_PX_screens\exp_49	Thu Jun 16 09:12:03 2016	running	???
pre_exp_50	C:\XcaliburData\Mathias_PX_screens\exp_50	Thu Jun 16 09:16:36 2016	Wed Jul 13 17:59:30 2016	???
pre_exp_51	C:\XcaliburData\Mathias_PX_screens\exp_51	Thu Jun 16 16:06:51 2016	Thu Jun 16 16:06:51 2016	???
pre_exp_52	C:\XcaliburData\Mathias_PX_screens\exp_52	Thu Jun 16 17:49:31 2016	Mon Sep 05 12:48:04 2016	???
IBR-co-PHO A-0193-07...	E:\data\2016\07\dusek_twin\IBR-co-PHO A-0193-0721_6	Wed Jun 29 19:01:46 2016	Fri Jul 15 16:42:02 2016	C2 H2 N2
Cy_20160713_2_PAF0...	E:\data\2016\07\hypix3000\XLM_ILMages	Thu Jul 14 14:40:07 2016	Fri Jul 15 17:29:02 2016	C36 H52
MJR1918_cystiene_30...	E:\data\2016\08\george_white\MJR1918_cystiene_300_2	Thu Aug 18 14:32:15 2016	Fri Aug 19 17:47:47 2016	C11 H10 N
DK_ML766	E:\data\2016\08\bruker\DK_ML7-66_MM_3843	Tue Sep 06 17:23:54 2016	Tue Sep 06 17:23:54 2016	???

Hide pre experiments
Hide screenings

Displaying information
 Standard Volume, laue, wavelength, Rint, redundancy Protein screening Custom columns [Change columns](#)

List: Standard [>>](#) [Delete](#) [Rename](#)

[Help](#) [Multiple addition](#) [Browse experiment](#) [Delete experiment\(s\)](#) [Open selected](#)

CrysAlis RED program options (1.1.4)

SMIPX

Small Molecule Protein

Lattice finding / Data reduction
Min lattice size: 2, max lattice size: 120
Use pre-exp SM indexing [400 ref., 5.0s]
Used options: completeness after data red, 2nd cycle in 3D peak analysis, SG determination (GRAL) after data red, concurrent data red used, min frames for dc red=25

Data collection / Strategy
Max automode exposure time (sec): 400, default completeness: 100.0%, I/sig for max. res. prediction: 2.0
Used options: overlap computation type: complex

DC JETSHADOW (to visualize beforehand use 'beamstop mask')
DC JETSHADOW not used

[Edit options](#)

[OK](#) [Cancel](#) [Help](#)

First peak hunting

- Use default

The screenshot displays the Lattice wizard (1.0.33) software interface. The main window shows a diffraction image with a grid overlay. A blue arrow points to the 'Peak hunting' button in the left-hand toolbar. A 'Peak hunting' dialog box is open, showing the current cell parameters and a 'Stop' button. The interface includes several panels:

- LATTICE**: Current cell (CSD: not done) 7.0930 7.0930 7.0930 90.0 90.0 356.8534. Lattice reduction: No reduced unit cell present!
- PEAK TABLE**: User-loaded table, 0 obs out of 0 (total:0,skipped:0)
- INSTRUMENT MODEL**: Goniometer beam: -0.01932 alpha: 50.05613 beta: 0.00000. Detector x-rot: 0.48390 y-rot: 0.13904. Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229
- Toolbars**: Peak hunting, Unit cell finding, Ewald explorer - reciprocal space, Refine instrument model, Twinning - multi-crystals, Load information, Unwarping - Precession images, Log window.
- START/STOP**: Crystal RED, LATTICE Current cell (CSD: not done), PEAK TABLE 0 obs out of 0 (total:0,skipped:0), INSTRUMENT MODEL.
- Data Collection** and **Data Reduction** buttons.
- Rigaku oxford diffraction** logo and **CRYSTALIS™ SM** logo.

Automatic unit cell finding

- Use default, 66% indexed, in spite of slightly off model

Lattice wizard (1.0.33)

LATTICE

Current cell (CSD: not done)
8.1341(7) 9.4250(10) 11.6001(13) 79.290(9) 82.878(8) 79.555(8) 855.58(15)

Lattice reduction
selected cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 aP 31
reduced cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 855.4

PEAK TABLE

Peak hunting table
UB fit with 8408 obs out of 12728 (total:12728,skipped:0) (66.06%)

INSTRUMENT MODEL

Goniometer
beam: -0.01932 alpha: 50.05613 beta: 0.00000
om zero: 0.04303 th zero: -0.32229 ka zero: 0.00000
Detector
x-rot: 0.48390 y-rot: 0.13904
x-cen: 515.30000 y-cen: 510.30000 distance: 41.00000
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Peak hunting Unit cell finding
Ewald explorer - reciprocal space Reindexation with current cell
Refine instrument model Lattice transformation

Compare data with CSD (1.1.3)

Check CSD and local database

Current cell (#1) (aP; V= 858.5; 8.15 9.43 11.60; 79.4 82.8 79.6; 1 ht)

Current cell (#1)
Lattice centering: aP
8.1479 9.4331 11.6046 79.4352 82.7719 79.6405 858.5259

Data sent to CSD
Lattice centering: aP
8.1479 9.4331 11.6046 79.4352 82.7719 79.6405 858.5259

Start search Stop search Toler.: 1.50% Options

No.	Cell ma...	Volume	a	b	c	alpha	beta	gamma	Formula	Symm	Origin: name	Prim. V	More info.
1	100.0%	858.7	8.15	9.43	11.62	79.4	82.7	79.6	C23H21N1O1	P-1	CSD: VAVXQH	858.7	CCDCCheckCell

Information
Searching completed.

CSD tools
Select all Deselect all View in Mercury Go To WebCSD (license required) Close

First instrument model refinement

- The header info is not precise. Refine on full data.
- Use default

Lattice wizard (1.0.33)

Lattice wizard

LATTICE
Current cell (CSD: not done)
8.1341(7) 9.4250(10) 11.6001(13) 79.290(9) 82.878(8) 79.555(8) 855.58(15)
Lattice reduction
selected cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 aP 31
reduced cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 855.4

PEAK TABLE
Peak hunting table
UB fit with 8408 obs out of 12728 (total:12728,skipped:0) (66.06%)

INSTRUMENT MODEL
Goniometer
beam: -0.01932 alpha: 50.05613 beta: 0.00000
om zero: 0.04303 th zero: -0.32229 ka zero: 0.00000
Detector
x-rot: 0.48390 y-rot: 0.13904
x-cen: 515.30000 y-cen: 510.30000 distance: 41.00000
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Lattice wizard (1.0.33)

Lattice wizard

LATTICE
Current cell (CSD: not done)
8.1480(3) 9.4339(6) 11.6071(4) 79.434(4) 82.776(3) 79.625(4) 858.76(7)
Lattice reduction
selected cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 aP 31
reduced cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 855.4

PEAK TABLE
Peak hunting table
UB fit with 8602 obs out of 12728 (total:12728,skipped:0) (67.58%)

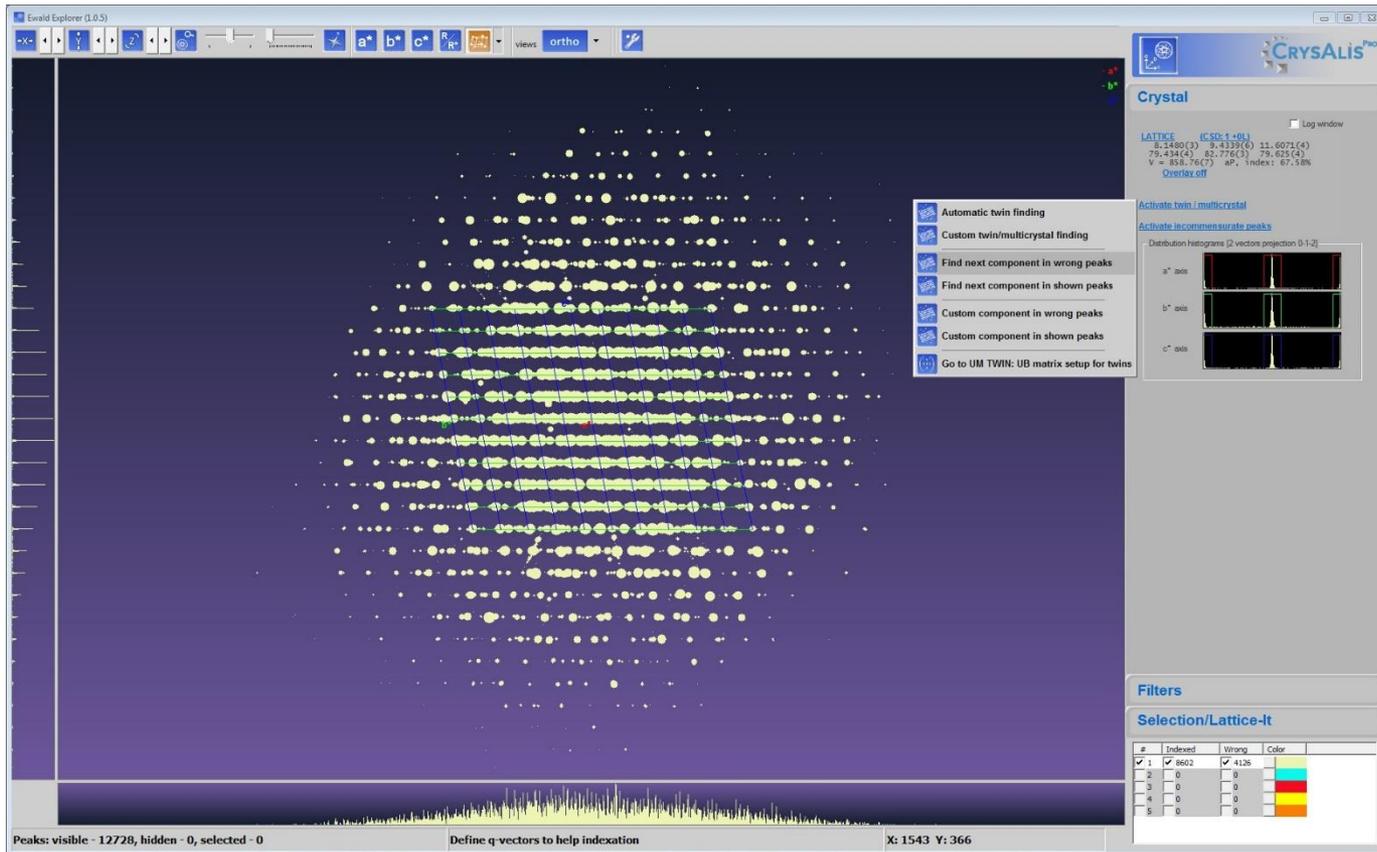
INSTRUMENT MODEL
Goniometer
beam: 0.12847 alpha: 50.05613 beta: 0.00000
om zero: -0.01105 th zero: 0.10058 ka zero: 0.00000
Detector
x-rot: 0.05131 y-rot: 0.24013
x-cen: 520.21123 y-cen: 507.31092 distance: 41.12325
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Refinement
res: 0.010877, da=0.006531, sx=0.002067, sy=0.002304
h=0.003394, k=0.006851, l=0.002762
#ref: 8565 (skipped: 34)

Peak hunting Unit cell finding
Ewald explorer - reciprocal space Reindexation with current cell
Refine instrument model Lattice transformation
Twinning - multi-crystals Incommensurates / Quasi-crystals
Load information Save information
Unwarping - Precession images
Log window Close

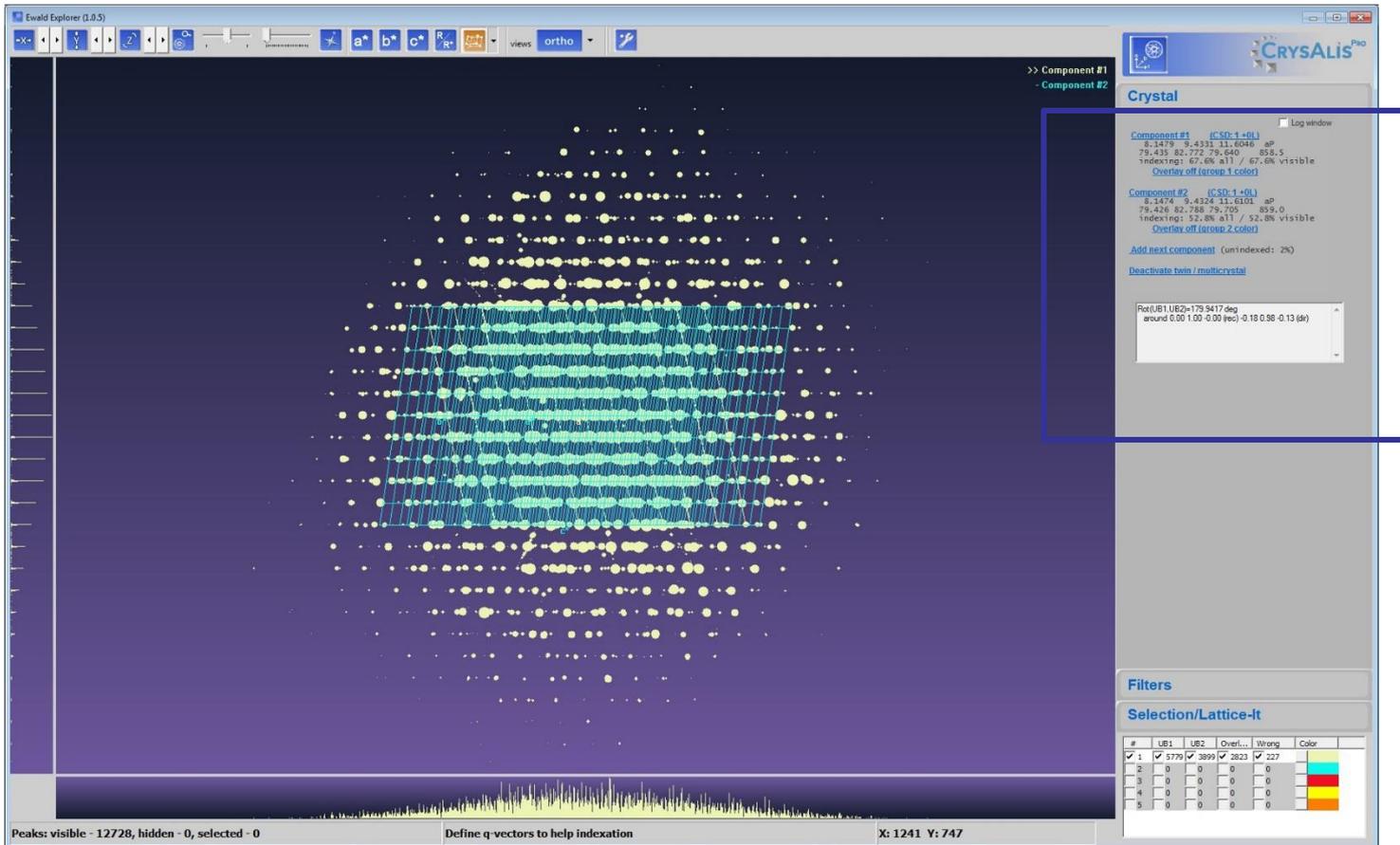
Use EwaldPro to find the twin

- Find next component in wrong peaks



Use EwaldPro to find the twin

- Easy: 180 deg rotation twin



Use EwaldPro to find the twin

- Back to lattice wizard...

Lattice wizard (1.0.33)

Lattice wizard

LATTICE

Current cell (CSD: 1 +0L)
8.1480(3) 9.4341(6) 11.6071(4) 79.433(4) 82.776(3) 79.626(4) 858.78(7)

Lattice reduction
selected cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 aP 31
reduced cell
8.1340 9.4225 11.6005 79.2946 82.9556 79.5359 855.4

Twin information
1: 8.1479 9.4331 11.6046 79.435 82.772 79.640 858.5
2: 8.1474 9.4324 11.6101 79.426 82.788 79.705 859.0
1: Total: 8602(67.6%) Separate: 5779(45.4%) Overlapped: 2823(22.2%)
2: Total: 6722(52.8%) Separate: 3899(30.6%) Overlapped: 2823(22.2%)
Unindexed: 227 (1.8%)

PEAK TABLE

Peak hunting table
UB fit with 8603 obs out of 12728 (total:12728,skipped:0) (67.59%)

INSTRUMENT MODEL

Goniometer
beam: 0.12847 alpha: 50.05613 beta: 0.00000
om zero: -0.01105 th zero: 0.10058 ka zero: 0.00000

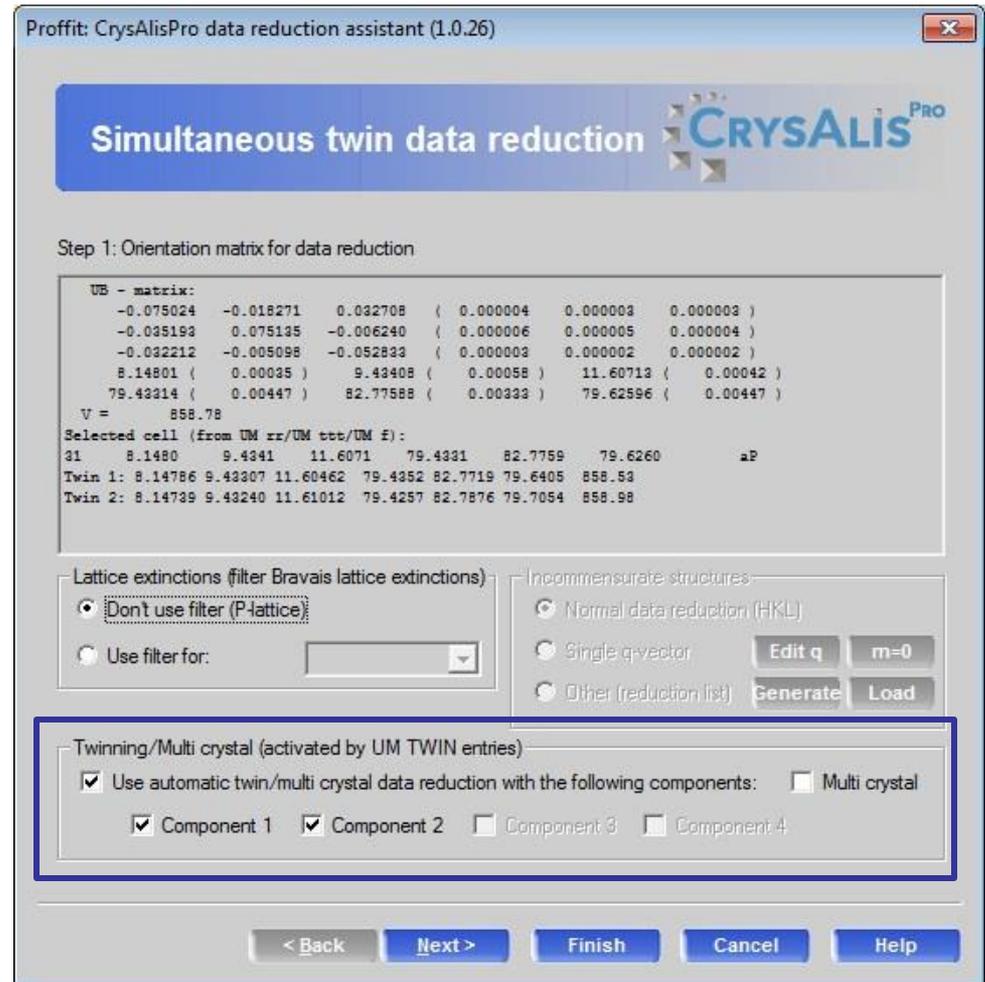
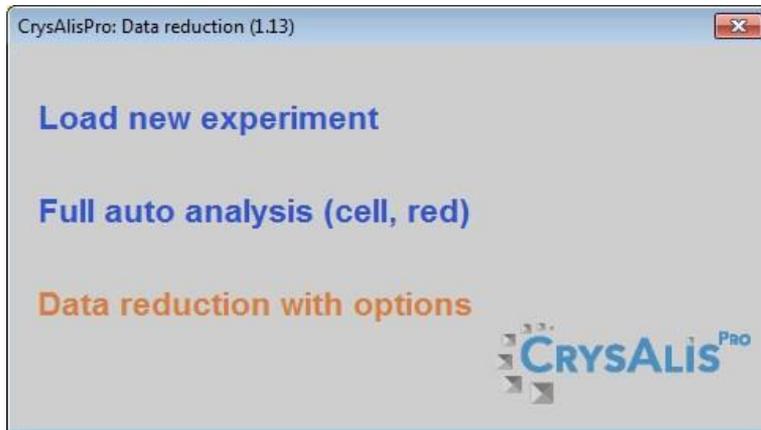
Detector
x-rot: 0.05131 y-rot: 0.24013
x-cen: 520.21123 y-cen: 507.31092 distance: 41.12325
Wavelength Mo (Ang): A1 0.70930 A2 0.71359 B1 0.63229

Refinement
res: 0.010877, da=0.006531, sx=0.002067, sy=0.002304
h=0.003394, k=0.006851, l=0.002762
#ref: 8565 (skipped: 34)

Peak hunting
Unit cell finding
Ewald explorer - reciprocal space
Reindexation with current cell
Refine instrument model
Lattice transformation
Twinning - multi-crystals
Incommensurates / Quasi-crystals
Load information
Save information
Unwarping - Precession images
Log window
Close

Do twin data reduction

- Run the data reduction wizard. Twin reduction auto set...



Do twin data reduction

- Accept the run list

Proffit: CrysAlisPro data reduction assistant (1.0.26)

Simultaneous twin data reduction

CRYALIS^{Pro}

Step 2: Experiment run list for data reduction

Run list: E:\data\2016\08\bruker\DK_ML7-66_MM_3843\DK_ML766

*.sfm

Image dir: E:\data\2016\08\bruker\DK_ML7-66_MM_3843

#	type	start	end	width	exposure	omega	detector	kappa	phi	start	end
1	o	1.39	184.19	0.40	5.00	-	30.00	73.93	-0.81	1,	456
2	o	-11.11	171.69	0.40	3.00	-	17.50	73.93	4.19	1,	456
3	o	21.38	163.78	0.40	3.00	-	10.00	73.93	86.69	1,	356
4	o	21.79	174.19	0.40	3.00	-	20.00	73.93	-35.82	1,	380
5	o	-28.11	126.69	0.40	5.00	-	-27.50	73.93	11.69	1,	386
6	o	18.39	179.19	0.40	4.00	-	25.00	73.93	9.18	1,	401
7	o	-7.81	174.19	0.40	3.00	-	20.00	73.93	-60.81	1,	454

By default the whole experiment will be evaluated. To modify this behaviour edit the run list -->

Edit start num of selected run

Edit end num of selected run

< Back Next > Finish Cancel Help

Do twin data reduction

- Special pars: All default; Limit the data to 0.75Å (as done in the B set)

The screenshot displays the 'Proffit: CrysAlisPro data reduction assistant (1.0.26)' window. The main window is titled 'Simultaneous twin data reduction' and shows 'Step 3: Basic algorithm parameters'. A 'Resolution limits' dialog box is open, showing the following parameters:

Parameter to enter:	d-value (Å)	Theta (deg)	2 Theta (deg)
	inf	0.000	0.000
	0.750	28.221	56.441

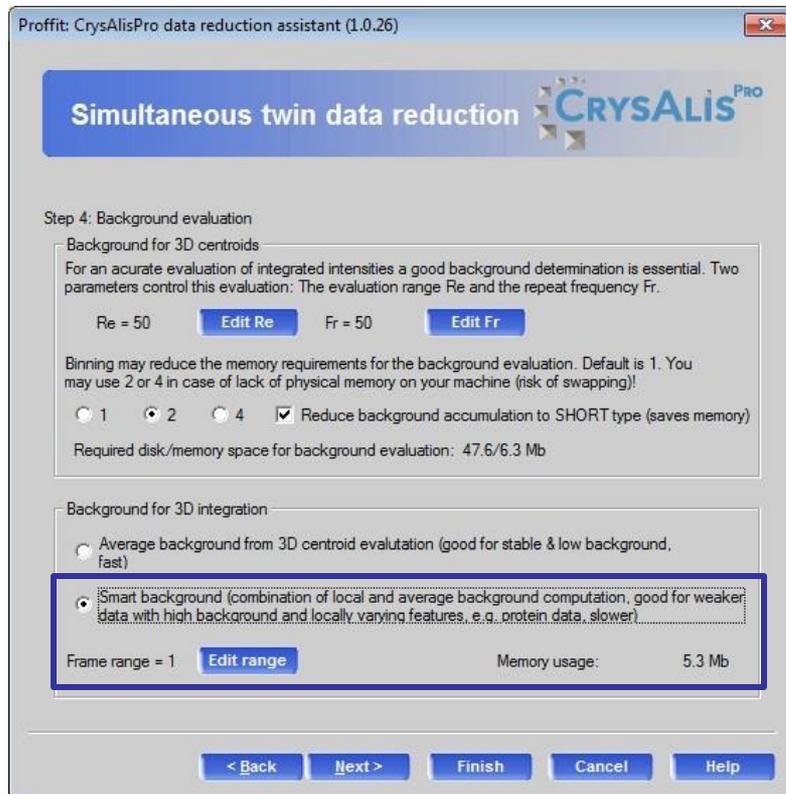
The 'Resolution limits' dialog box also includes buttons for 'Edit low limit' and 'Edit high limit'. The main window shows the 'Proffit special parameters' dialog box with the following settings:

- Use resolution limits (Edit limits)
- d-value (Å): inf-0.75
- 2theta (deg): 0.00-56.44
- Reject reflections with bad profiles (e.g. for HP data)
- I/sig > 10 & Profile agreement < 0.8

The 'Resolution limits' dialog box also includes 'OK' and 'Cancel' buttons. The main window has a 'Clear data from previous run' button and navigation buttons: '< Back', 'Next >', 'Finish', 'Cancel', and 'Help'.

Do twin data reduction

- Background: Smart background



This is the most critical for the data set: It seems that the Apex detector has unstable background, thus introducing a bias on the average background method. Such behavior is also seen with other detectors where CAP uses by default Smart background.

Do twin data reduction

- Automatic outlier rejection: default

Proffit: CrysAlisPro data reduction assistant (1.0.26)

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

Use outlier rejection

1

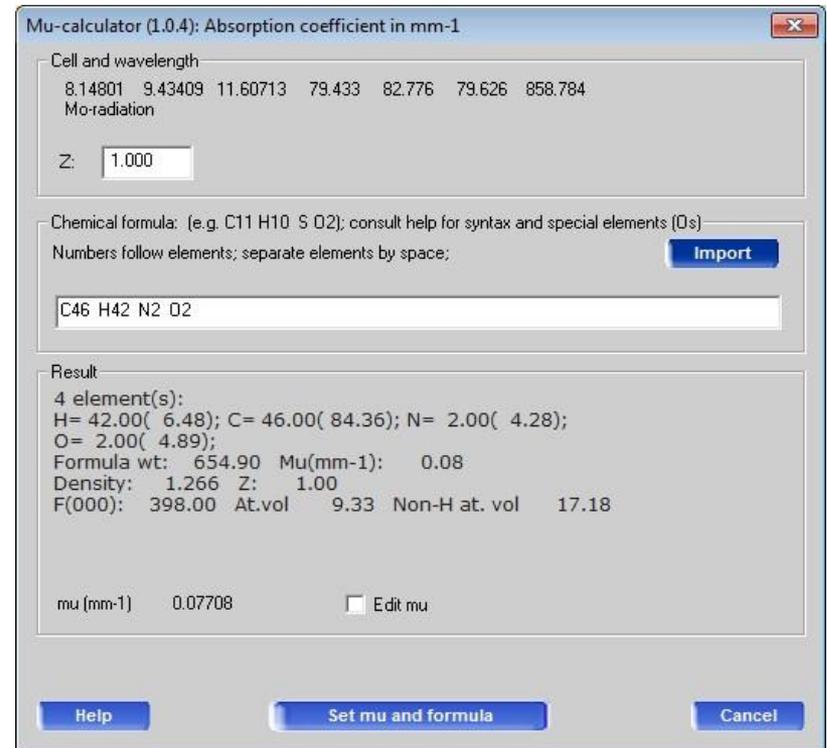
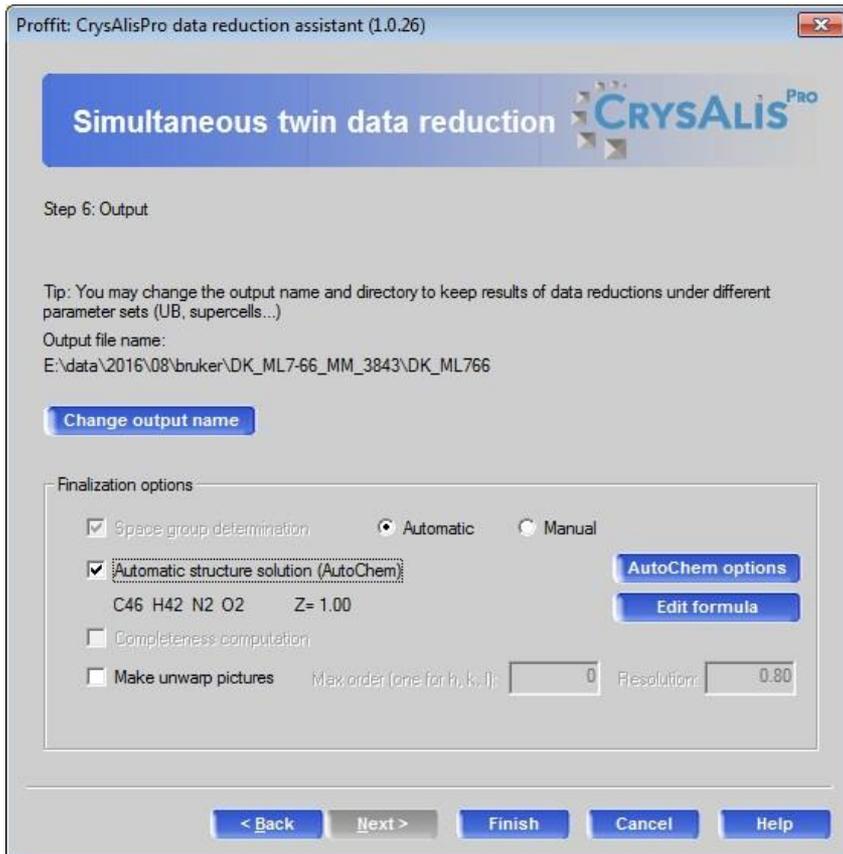
aP 8.14801 9.43408 11.60713 79.43314 82.77588 79.62596

Use Friedel mates as equivalent

< Back Next > Finish Cancel Help

Do twin data reduction

- Chem. formula imported for AutoChem



Automatic result

- Auto: based on all hklf4 data.

The screenshot displays the Olex2 software interface. The main window shows a diffraction pattern with several peaks highlighted in red. The right-hand panel contains the following information:

- START/STOP** button
- RED Ready** status
- Crystal** (RED) dropdown menu
- Data Collection** dropdown menu
- Data Reduction** dropdown menu
- AutoChem** dropdown menu
- Olex2** and **Restart AutoChem** buttons
- REFINEMENT STATISTICS** section:
 - Chemical formula: C46H42N2O2
 - Space Group: P-1 #2
 - Formula weight: 654.90
 - R1: 6.28%
 - wR2: 15.56%
 - GOOF: 1.18
 - Absorption: 0.077 mm⁻¹
 - Peak and Hole: 0.377 and -0.258
- STRUCTURE FILE** section:
 - Filename: DK_ML766_twin1_hk1f4
- Rigaku oxford diffraction** logo

At the bottom left of the main window, the following image information is displayed:

IMAGE: DK_ML766_01_0458.stm (run 0 frame 1)
Omega: 24.93 Tilt: 10.00 Kappa: 78.93 Phi: 88.69 Distance: 41.91

Tuning your result

- Tuning means what kind of scaling is used and what reflection classes are in the final hklf4 and 5 files

Scale3 abspack (1.0.7) - Refinement of scales, emp. absorption, sensitivity and sample d...
hkFile: E:\data\2016\08\bruker\DK_ML7-66_MM_3843\DK_ML766_twin1.hkl
Output dir: E:\data\2016\08\bruker\DK_ML7-66_MM_3843

Symmetry settings
 Use Friedel mates as equivalent even for noncentrosymmetric SG

LS refinement control
SigCut: 7.00
 Exclude 15 strongest unique reflections (along with all symmetric equivalents)

Frame scaling
 Automatic frame scale assignment Apply frame scaling
How many frames have a common scale? 2 frames = 1 scale
Variation restraint (ESD): 0.20000
 Reject frame scales < 0.20 & > 5.00

B-factor/A-factor refinement
 Refine B-factors
 Refine A-factors
10 frames = 1 factor
Restraint 0.20000

Empirical absorption correction
 Automatic parameter selection for absorption correction
Max even order: 8 Max odd order: 7 80 parameters
 Absorption correction before frame scaling (recommended for strong absorbers)

Detector area scaling
How many detector area regions? 4x4 Apply detector correction
Variation restraint (ESD): 0.20000

Help OK Cancel

DC PROFFITRRP TWIN (1.1.2)
Twin data finalization

rrrprof files for twin finalization
Browse DK_ML766.rprtwin
Component #1 DK_ML766_twin1.rrrprof aP 8.143 9.430 11.603 79.46 82.79 79.63
Component #2 DK_ML766_twin2.rrrprof aP 8.144 9.425 11.609 79.42 82.80 79.71
Component #3 None LATTICE #
Component #4 None LATTICE #
Twin finalization log file (from previous run): DK_ML766.twinlog View log

Chemical formula
C46 H42 N2 O2 Z= 1.00
Edit formula

Lattice symmetry
Laue class: 1 Use Friedel mates as equivalent

Corrections
Twin decomposition
Overlap threshold for scaling & space-group determination: 0.400 Edit threshold
 Ensure min completeness in HKL F4 file (for struct solution): 80.0% Edit comp

scale3 abspack: scaling, uniformity, empirical absorption
 Use ABSPACK Edit ABSPACK
 Common scales for all twin components
 Separate scales for all twin components

Numeric and spherical absorption correction
 Apply absorption correction No face information available!
n/a
Spherical abs (μr)= 0.031

Space-group and structure
 Space group determination Options
 Structure solution (AutoChem) Options

Filters and lattice extinction filters
 Resolution d-value (Å): inf. θ-cut: 2θmax (Deg): 0.00-82.83 dit res limit
 No filter use filter for: P-lattice
Filters 0 active filters

Export options
Data items in *.hkl files (HKL F4/HKL F5)
 Beam path information (for absorption correction)/special formats:
CIF
 Export CIF file Add hkl data to CIF (HKL F4 only)

HKL file options
 Use reflections from component 1 only
 Output multiple HKL F4 files containing data from twin 1 and twin
 #2 #3 #4
Invert HKLs of twin:
 #2 #3 #4

Merging (HKL F5 only)
 Merge data using:
 the same Laue class Use Friedel mates as equivalent
 different Laue class 1
 Remove outliers
Merging options

Output
 Standard set of files Copy hkl to DK_ML766_twin...
Change output base name DK_ML766

Create/overwrite DK_ML766 files (hkl, ins, cif, od) in E:\data\2016\08\bruker\DK_ML7-66_MM_3843.
Copy DK_ML766.hkl and DK_ML766.cif files also to \struct\plex2_DK_ML766_twin1_hklf4

Help OK Cancel

HKLF4 result

Space group selection

Select space group:

Space Group	No.	Centro	CCDC	ICSD	R(int)
P1	1	-	2112	239	0.034
P-1	2	+	47868	2604	0.037

<E2-1>

CENTROSYMMETRIC

Test chemical formula OK

bruker\DK_ML7-66_MM_3843\DK_ML766.par (38.43)

START/STOP

RED Refinement finished

Assign Crystal RED

Data Collection

Data Reduction

Structure Explorer

SG: P-1
R1: 5.93%

Peak and hole: 0.4 / 0.2

Rint: 3.74% GOOF: 1.177
Rtail/wR2: 6.45% / 10.95%

C₂₃H₂₁N₁O₁

Auto update weights: ON
Weight: 0.024 1.164
Shift (max): 0.000
Absorption: 0.077 mm⁻¹

Unit cell:
a: 3413(3) 9.4289(4) 11.6009(3)
b: 4459(4) 82.786(3) 79.830(3)
v: 857.26

Structure solved (res file):
...IDK_ML766_twin1_hklf4.res
...IDK_ML766_twin1_hklf4.hkl

Experiment Autochem/Solve Refinement Publish/Share

Refine Complete AutoAssign (ATA) History .ins Cmd Twin DK_ML766_twin1_hklf4.hkl

Rigaku oxford diffraction

CRYALISSM

HKLF5 result

The screenshot displays the CrysAlisPro interface during a refinement process. A dialog box titled "Twin refinement options" is open, showing the following settings:

- Twin refinement method:
 - Refine structure on whole data set (HKLF5)
 - Refine structure on first component only (HKLF4)
 - Refine structure on second component only (HKLF4)
 - Refine structure on third component only (HKLF4)
 - Refine structure on fourth component only (HKLF4)
- Information:

INS file: ...plex2_DK_ML766_twin1_hklf4\DK_ML766_twin1_hklf4.ins,
HKL file: ...plex2_DK_ML766_twin1_hklf4\DK_ML766_twin1_hklf5.hkl
INS file will be modified with BASF and HKLF 5 instructions.
- Refine on exit

The main window shows a 3D ball-and-stick model of a complex organic molecule with a blue nitrogen atom and a red oxygen atom. The right-hand panel displays the following data:

START/STOP
RED Refinement finished

Assign
Crystal RED
Data Collection
Data Reduction
Structure Explorer

SG: P-1
R1: 5.18%

Peak and hole: 0.4 / 0.3
Rint: - GOOF: 1.154
R1all/wR2: 5.46% / 10.25%

C₂₃H₂₁N₁O₁
Auto update weights: ON
Weight: 0.027 0.827
Shift (max): 0.000
Twin ratio: 0.302
Absorption: 0.077 mm⁻¹

Unit cell:
8.1413(3) 9.4289(4) 11.6009(3)
79.469(4) 82.786(3) 79.630(3)
V01 = 857.26

Structure solved (res file):
...DK_ML766_twin1_hklf4.res
...DK_ML766_twin1_hklf5.hkl

At the bottom, the status bar shows the current command: `Twin DK_ML766_twin1_hklf5.hkl`

Compare to Saint/TwinAbs

- The Saint/TwinAbs processing and structure was provided by Dr. Kratzert.
- AC3.0 and Olex2 used automatically a different style of hydrogen adding, so the result was refined using Daniel's result.

Compare to Saint/TwinAbs hklf4

	Saint/TwinAbs	CrysAlisPro 38.43 AC2.1
wR2 refined quantity	0.1581	0.1429
R1all	0.0759	0.0645
R1 (I>4sig) #>4sig	0.0604 (3431)	0.0593 (3101)
#reflections	4240	3395
#par	227	289
I/sig	23.1	28.8

Compare to Saint/TwinAbs hklf5

	Saint/TwinAbs	CrysAlisPro 38.43 AC2.1
wR2 refined quantity	0.1342	0.1233
R1all	0.0546	0.0546
R1 (I>4sig) #>4sig	0.0472 (3582)	0.0518 (4229)
#reflections	4022	4453
#par	228	290
Twin ratio	0.29389	0.30184
I/sig	13.9	50.7

Compare to Saint/TwinAbs

- Now one more time the results refined against Daniel's res file, which carries less parameters
- Generally the results are similar

Compare to Saint/TwinAbs hklf4

	Saint/TwinAbs	CrysAlisPro 38.43 Daniel res
wR2 refined quantity	0.1581	0.1471
R1all	0.0759	0.0655
R1 (I>4sig) #>4sig	0.0604 (3431)	0.0603 (3101)
#reflections	4240	3395
#par	227	227
I/sig	23.1	26.6

Compare to Saint/TwinAbs hklf5

	Saint/TwinAbs	CrysAlisPro 38.43 Daniel res
wR2 refined quantity	0.1342	0.1257
R1all	0.0546	0.0555
R1 ($I > 4\sigma$) $\# > 4\sigma$	0.0472 (3582)	0.0527 (4229)
#reflections	4022	4453
#par	228	228
Twin ratio	0.29389	0.30205
I/sig	13.9	50.7

Compare to Saint/TwinAbs gain 150

- It is clear from the previous table that the agreement factor results are very similar, but the sigma scheme is significantly different. Especially the hklf5 TwinAbs reduction significantly damps the I/σ in spite of the additional observations mapped in.
- To see the effect of such 'pessimistic' view, I conducted a data reduction as in the previous using a system gain of 150.
- This was done with 39.8f.

Compare to Saint/TwinAbs hklf4

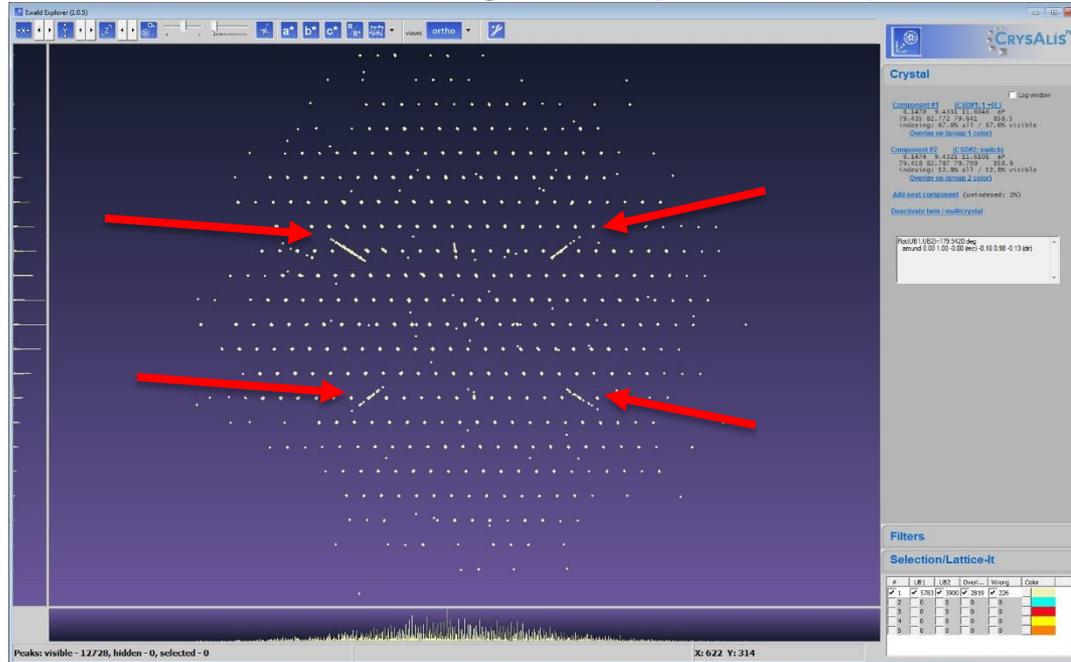
	Saint/TwinAbs	CrysAlisPro 39.8f Daniel res
wR2 refined quantity	0.1581	0.1366
R1all	0.0759	0.0650
R1 (I>4sig) #>4sig	0.0604 (3431)	0.0527 (2814)
#reflections	4240	3435
#par	227	227
I/sig	23.1	20.6

Compare to Saint/TwinAbs hklf5

	Saint/TwinAbs	CrysAlisPro 39.8f Daniel res
wR2 refined quantity	0.1342	0.1238
R1all	0.0546	0.0560
R1 ($I > 4\sigma$) $\# > 4\sigma$	0.0472 (3582)	0.0476 (3898)
#reflections	4022	4531
#par	228	228
Twin ratio	0.29389	0.30447
I/sig	13.9	17.5

Extra mention 3I effect

- Ewald with large steak. Missing Si filter. This will mess up SG extinctions on higher symmetry SGs.



J. Appl. Cryst. (2011). 44, 763-771, doi:10.1107/S0021889811016232

Low-energy contamination of Mo microsource X-ray radiation: analysis and solution of the problem

P. Macchi, H.-B. Bürgi, A. S. Chimpri, J. Hauser and Z. Gál

Theory: Reflection de-convolution

- Twin law
 - Reflection overlap
 - Overlap ratio
-
- The following slides are adapted from a 2013 webinar
 - It introduces some of the CrysAlis^{Pro} twinning concepts

Twining*: Challenges for the crystallographer

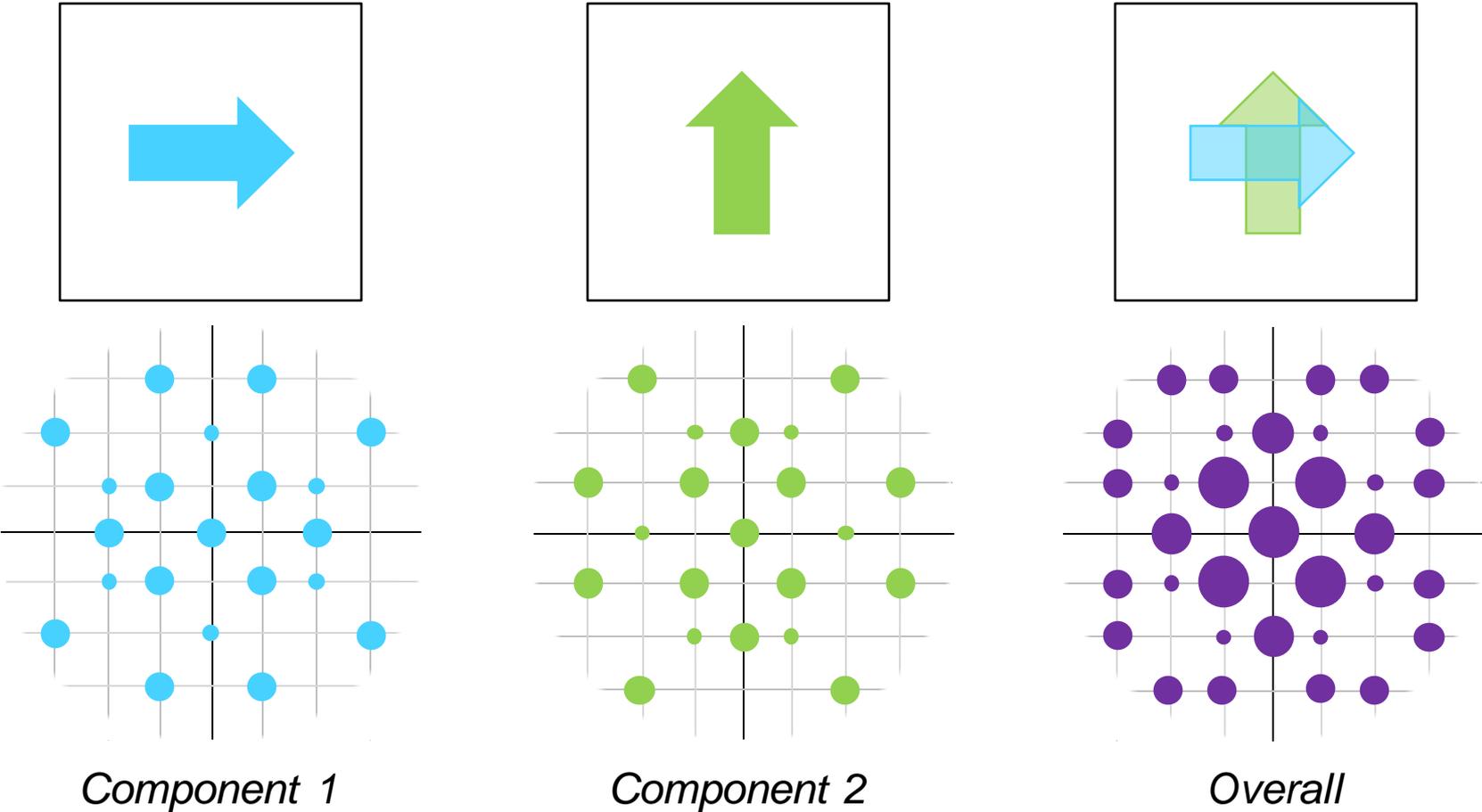
- Identify 'proper' unit cell(s); if possible at the screening/pre-experiment stage
- Reduce overlapping data
- De-convolute and correct data
- Solve the structure
- Refine in best possible way

**non-merohedric*

Introduction to Twinning

Fully overlapped diffraction patterns

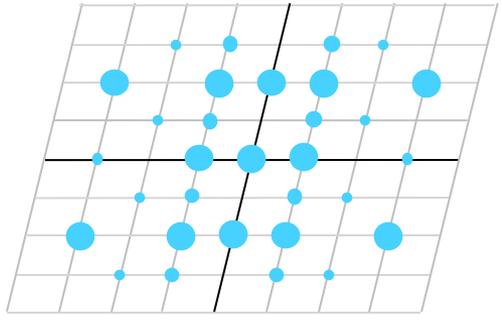
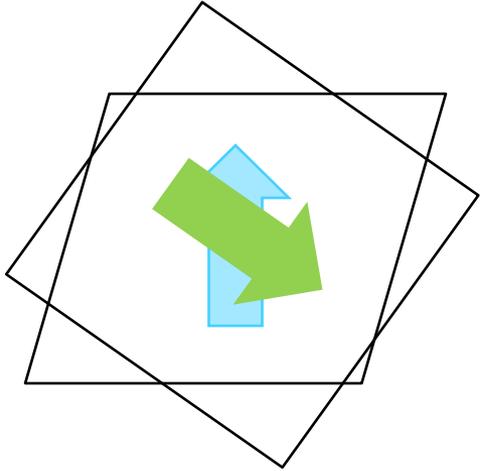
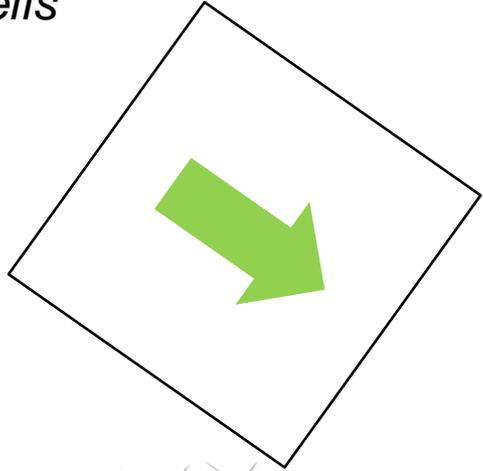
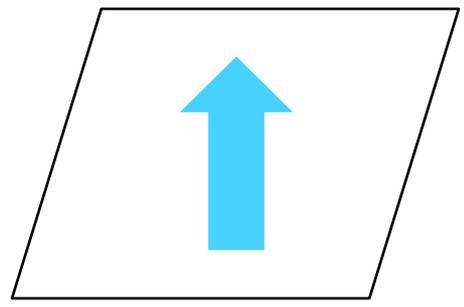
Merohedral twins



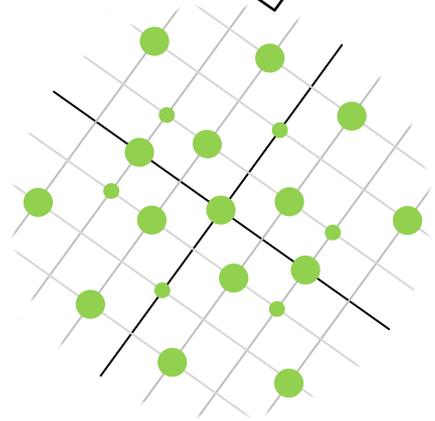
Introduction to Twinning

Partially overlapped diffraction patterns

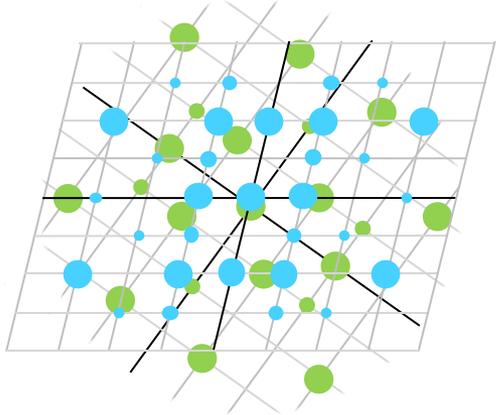
Multi-crystals – different unit cells



Component 1

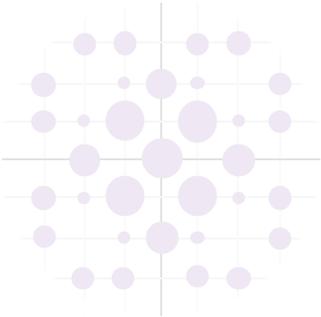
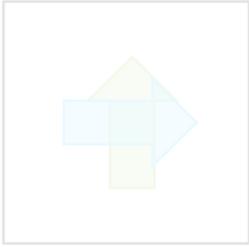


Component 2

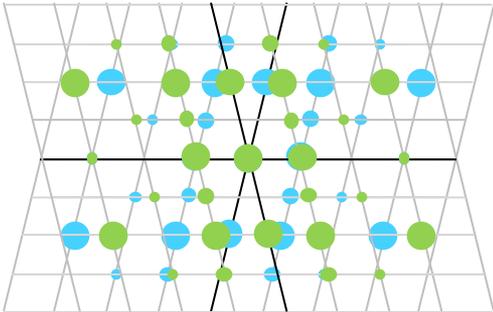
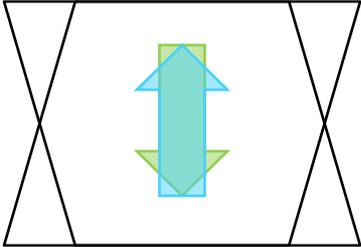


Overall

Introduction to Twinning



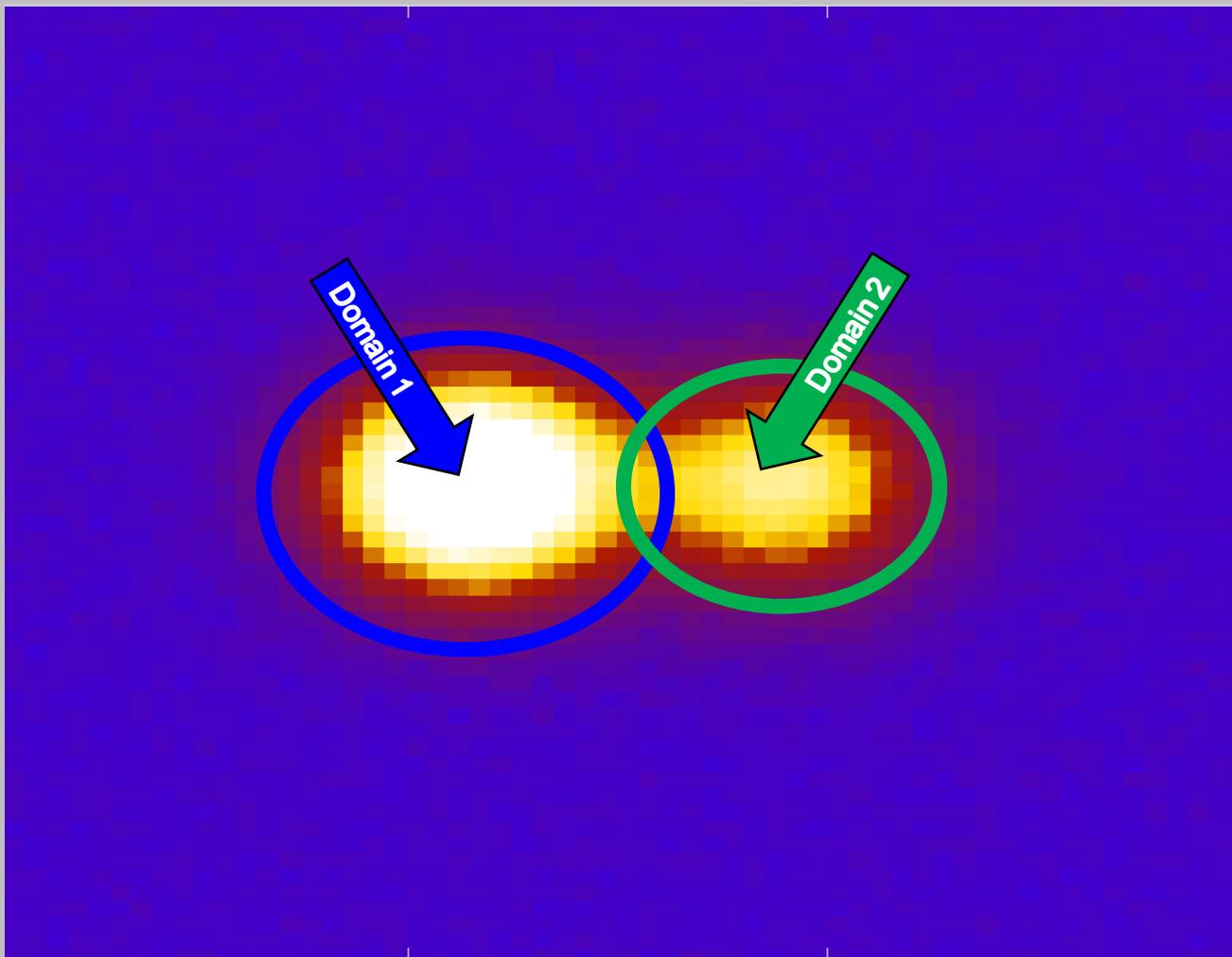
Fully overlapped diffraction patterns can't be separated at integration



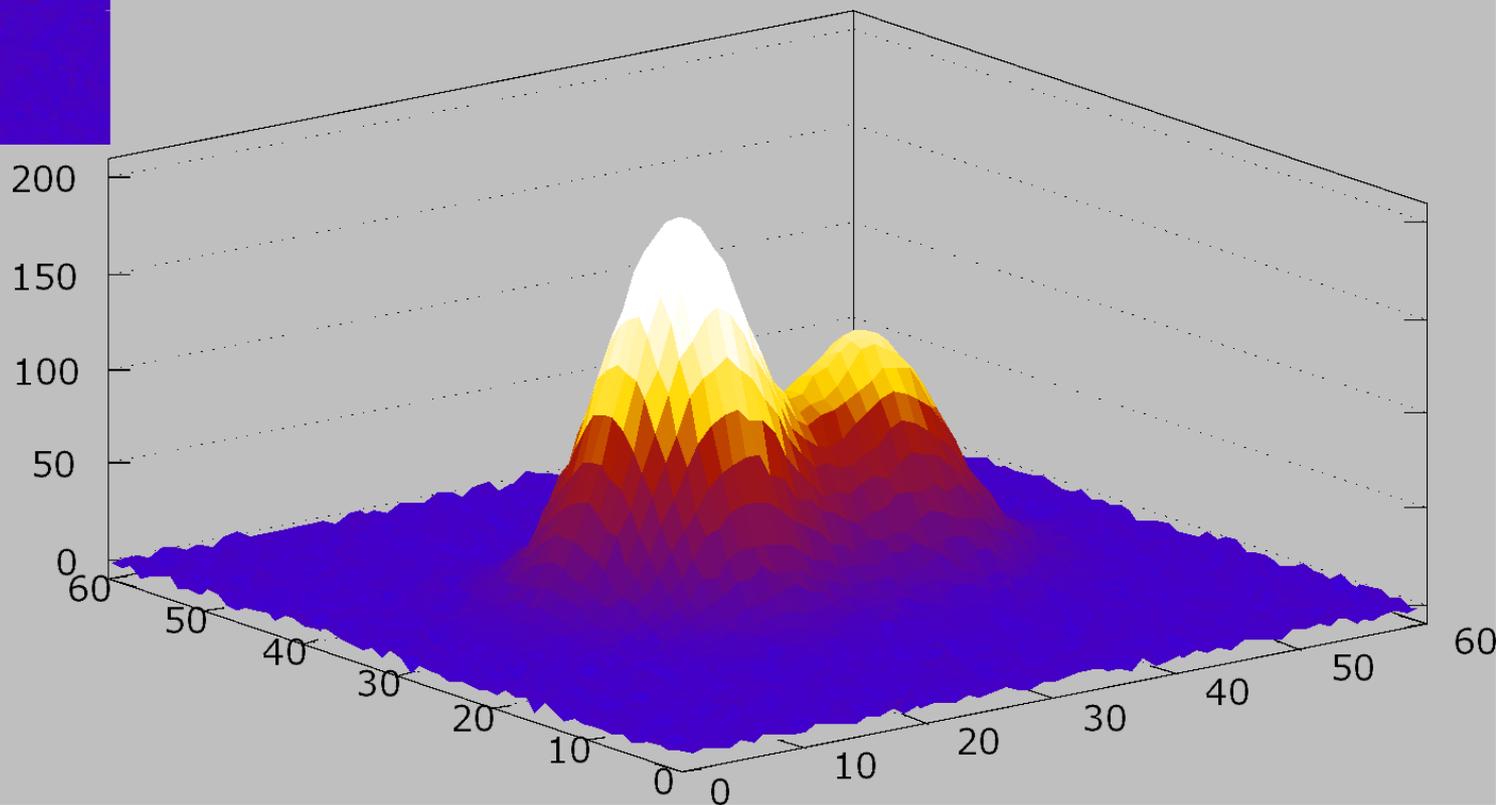
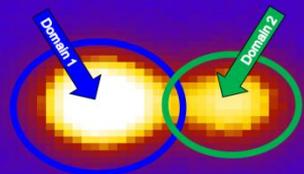
Partially overlapped reflections can be treated during integration

Twinning

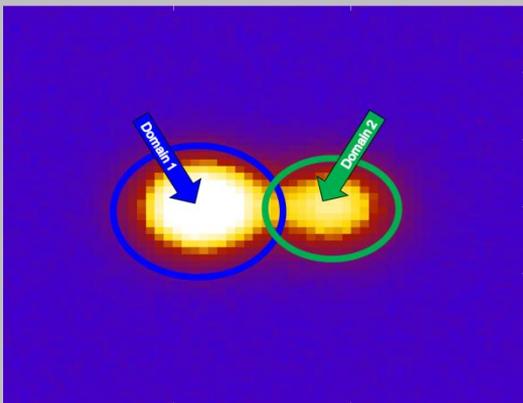
Integration



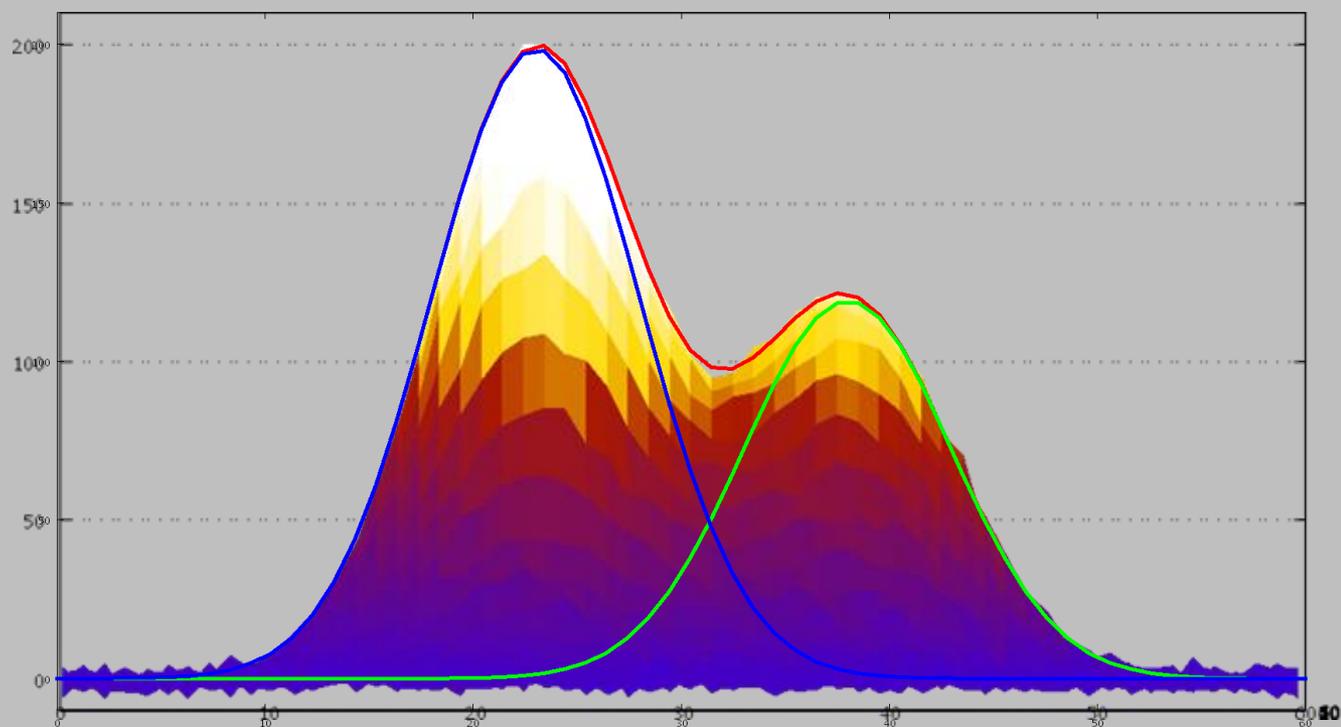
Twinning Integration



Twinning Integration



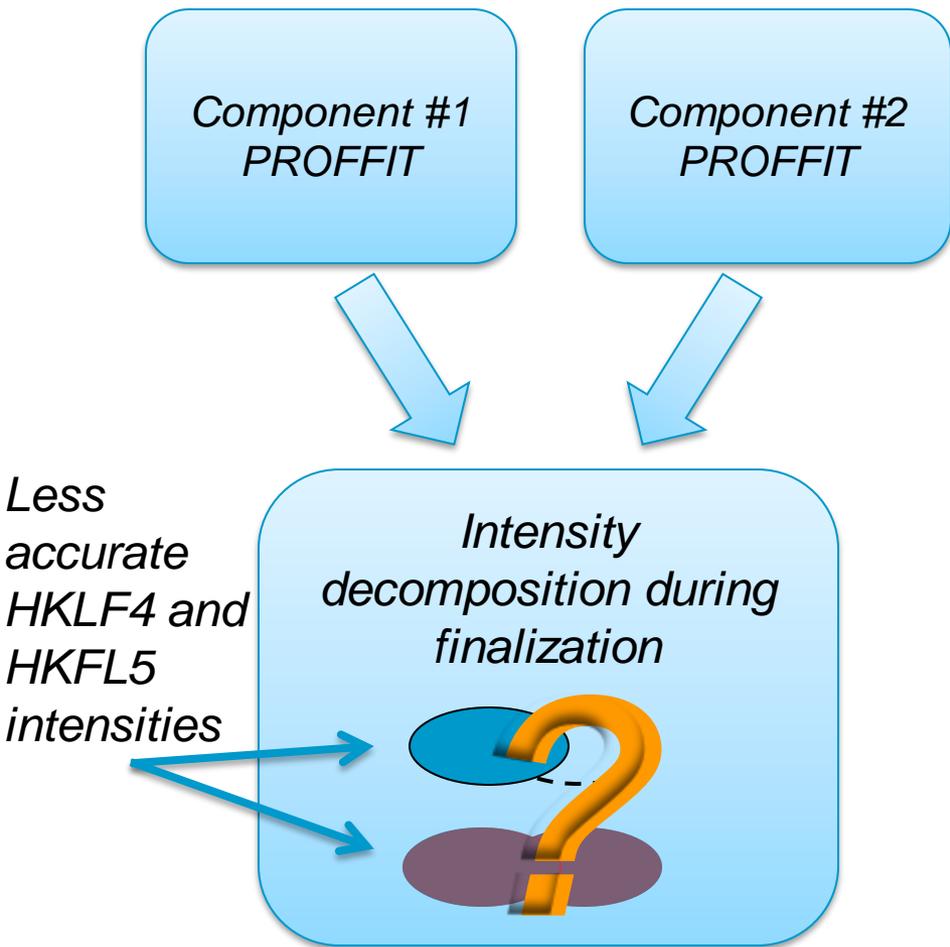
- Component 1 contribution
- Component 2 contribution
- Overall profile



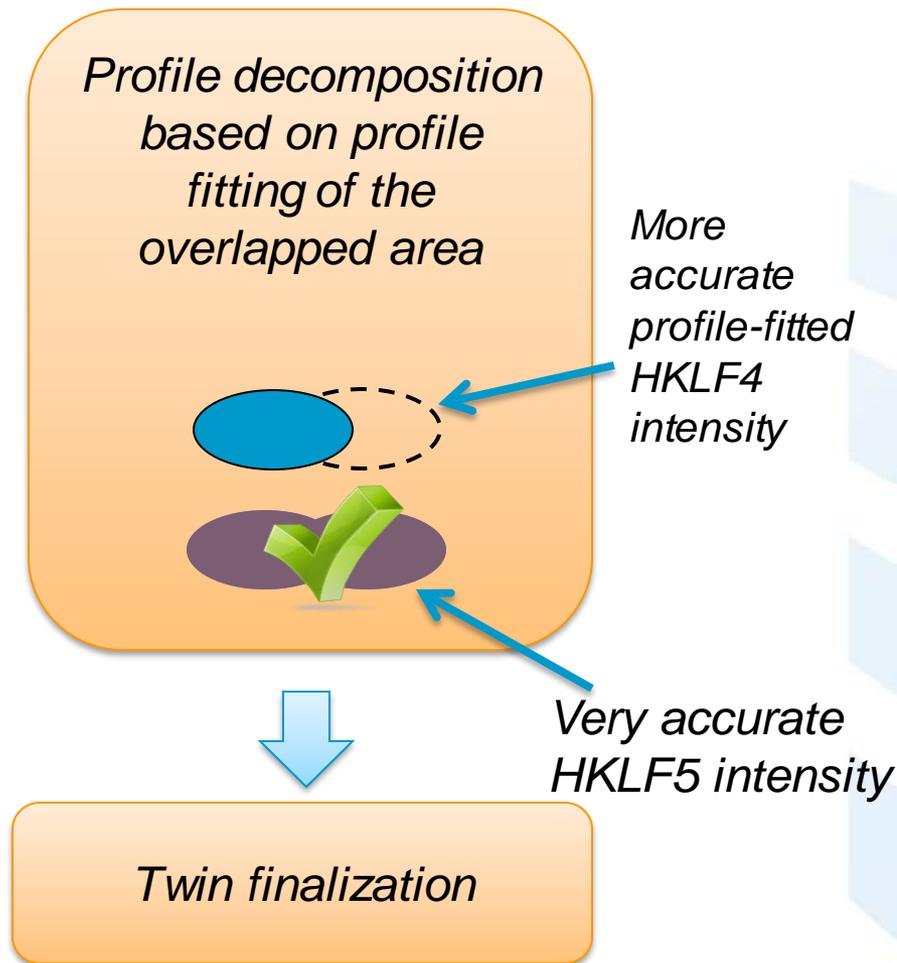
Current vs. new approach



Current twin integration (36)



New simultaneous integration (37)



New features

- Profile fitting
- Smart background (combination of local and average background)
- Bad-profile rejection filter
- Completeness-based reflection selection for HKLF4 file:
 - User does not have to adjust thresholds to obtain reasonable completeness
 - If the completeness of isolated reflections is below the requirement:
 - Reflections are sorted by overlapping factor
 - Starting from least overlapping there are added to the file until minimum required completeness reached (default 80% - enough to solve the structure in majority of cases)

Twinning

Integration output

Exp_1_twinN_HKLF4.hkl

Use for structure solution

Contains:

- Domain **N** only reflections
- Domain **N** portion of reflections below full overlap threshold (i.e. detwinned data)

Exp_1_twin1_HKLF5.hkl

Use for structure refinement

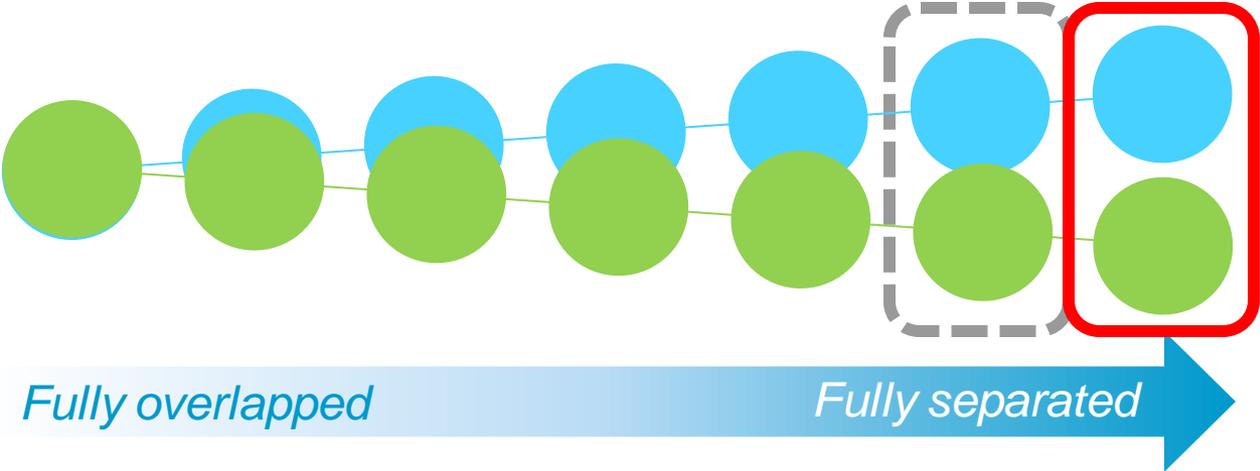
Contains:

- All reflections
- Any overlapped reflections are not detwinned

The Twin Challenge

Integration treatments – profile size

With typical masks, in this example, few reflections are fully separated...



Component 1 profile mask



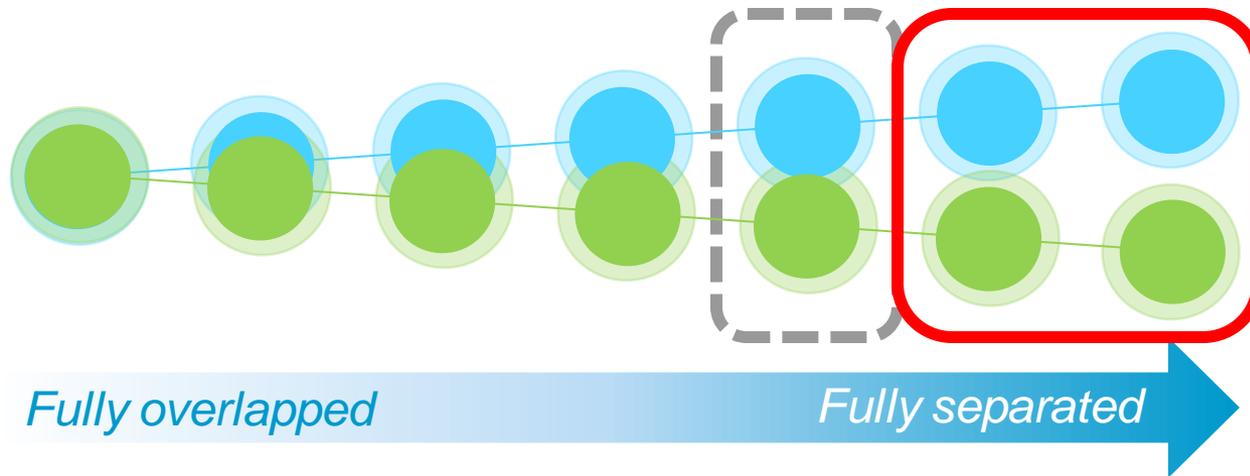
Component 2 profile mask



Twinning

Integration treatments – profile size

Using smaller masks can reduce overlap meaning more separate reflections



Component 1 profile mask



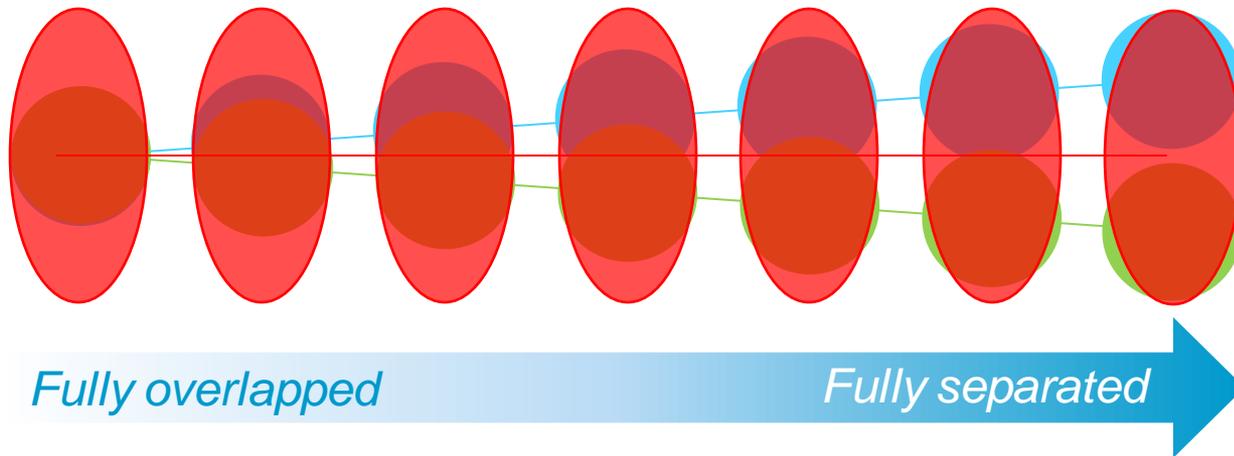
Component 2 profile mask



Twinning

Integration treatments – profile size

Alternatively, if separation is small enough, and depending on the nature of the twin, twinning can sometimes be ignored entirely by *increasing* profile mask sizes and treating as a single crystal

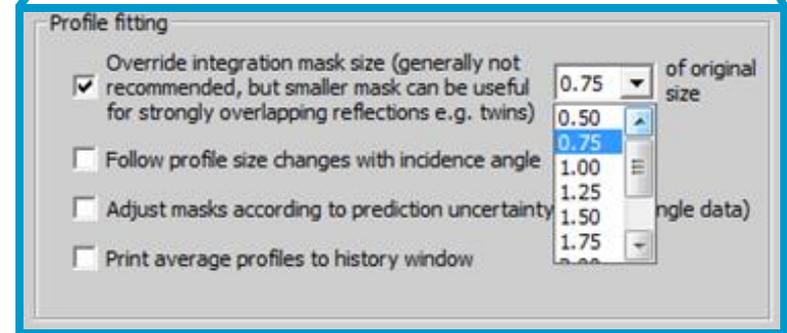
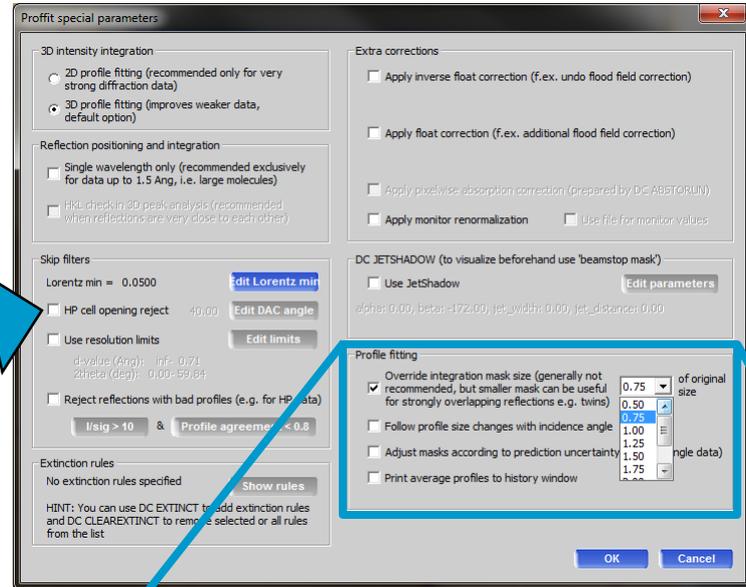
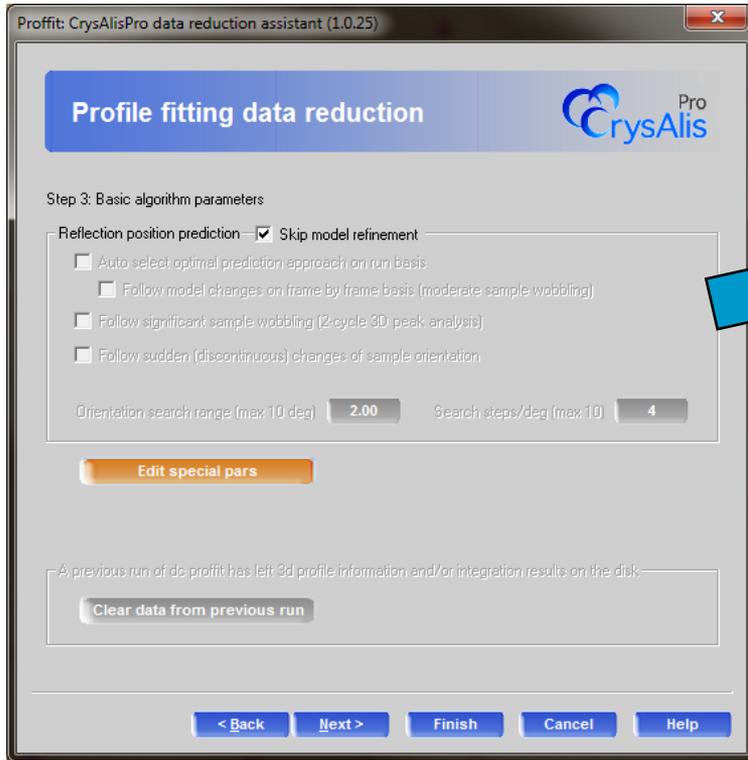


- Increase Profile 
- Component 1 
- Component 2 

Twinning

Integration treatments – profile size

Altering profile mask sizes in CrysAlis^{Pro}

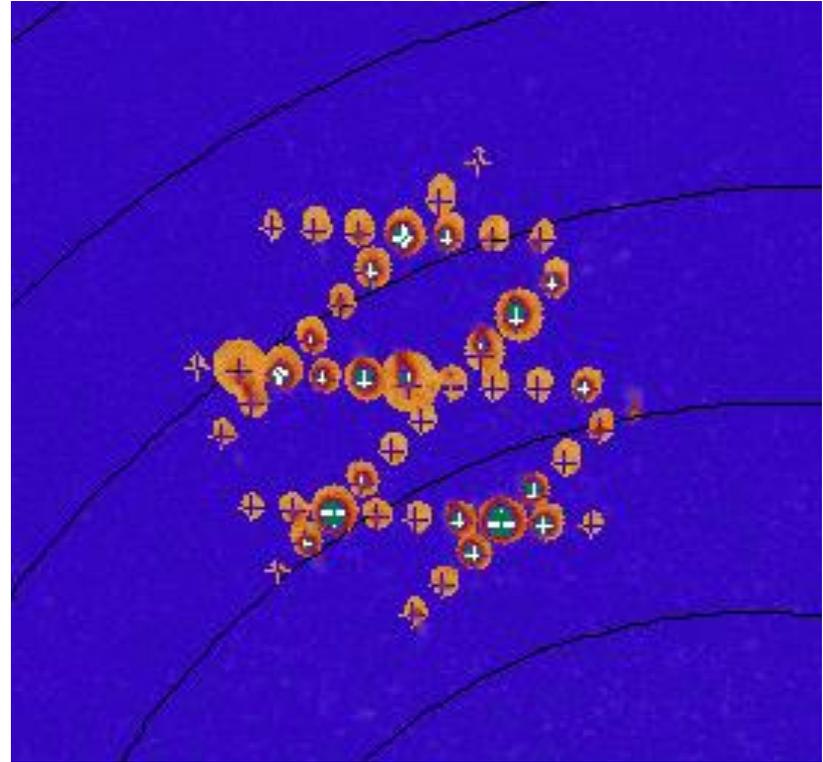


Twinning

Integration treatments – smart background

For datasets with uneven backgrounds, smart background can help.

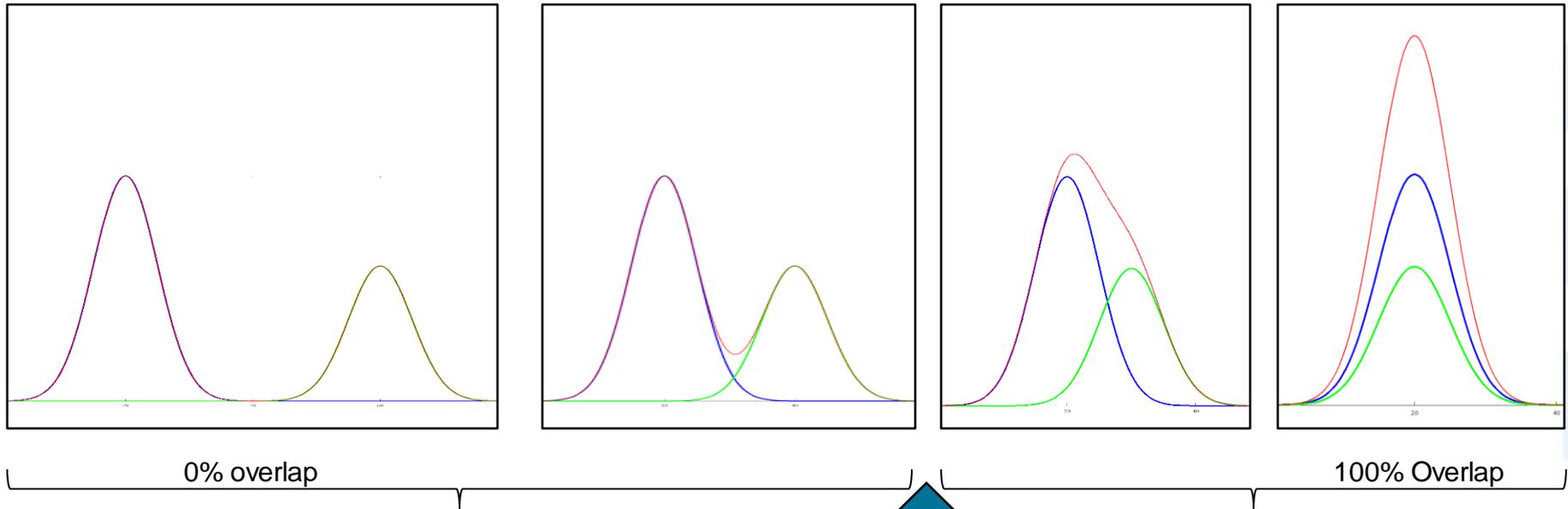
From v37 upwards, smart background is now twin enabled



Twinning

Post Integration treatments – overlap threshold

Increasingly difficult deconvolution



0% overlap

100% Overlap

*Detwinning attempted
for structure solution*

Changeable
threshold

*Detwinning not attempted
default: 80%*

Twinning

Post Integration treatments – overlap threshold

Version 36 or lower

Twin decomposition

Full overlap threshold: 0.800 [Edit threshold](#)

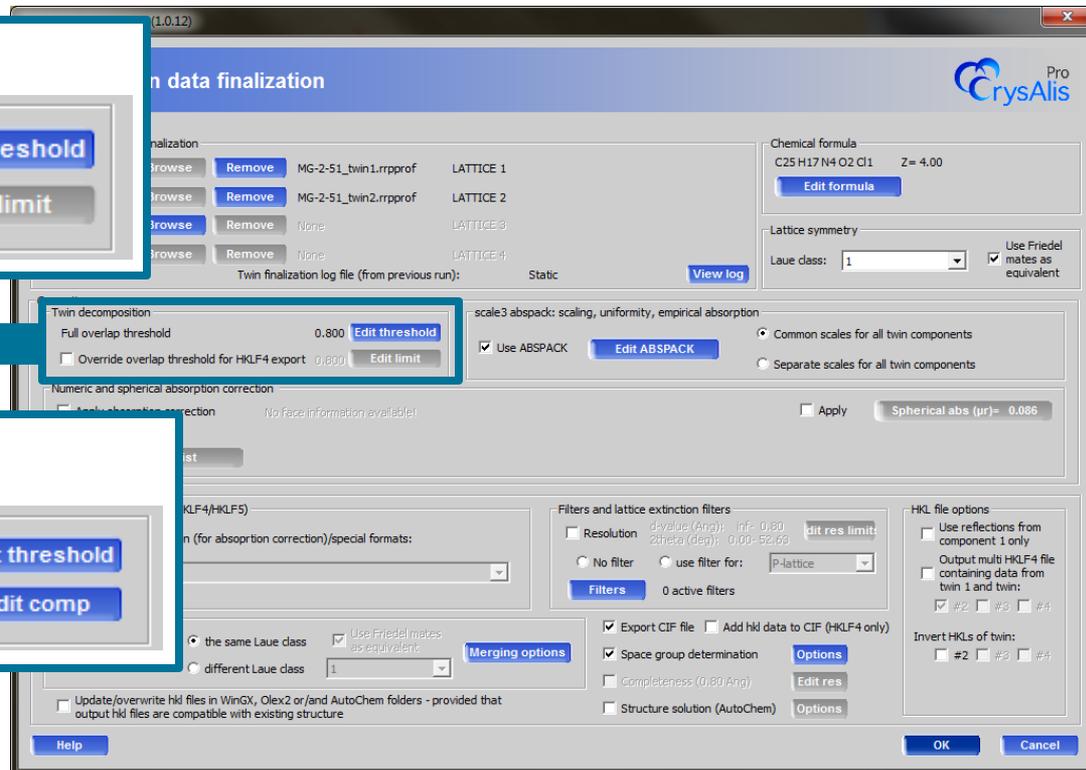
Override overlap threshold for HKLF4 export: 0.800 [Edit limit](#)

Version 37 or higher

Twin decomposition

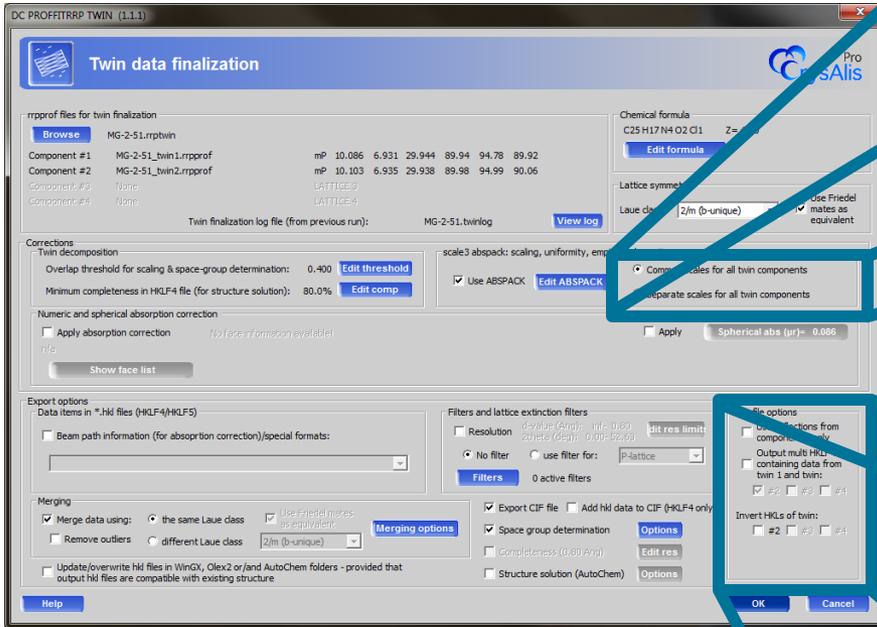
Overlap threshold for scaling & space-group determination: 0.400 [Edit threshold](#)

Minimum completeness in HKLF4 file (for structure solution): 80.0% [Edit comp](#)



Twinning

Additional options



Common scales for all twin components
 Separate scales for all twin components

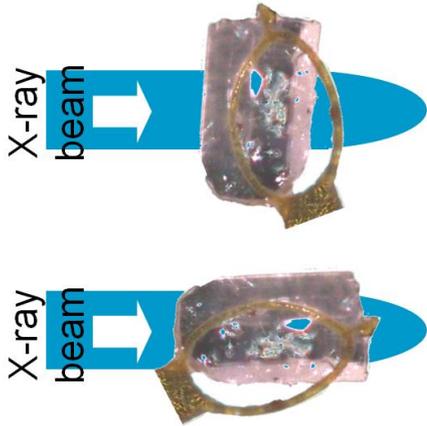
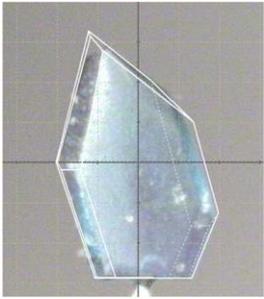
Useful for cases where secondary domain(s) are weak

HKL file options
 Use reflections from component 1 only
 Output multi HKLF4 file containing data from twin 1 and twin:
 #2 #3 #4
Invert HKLs of twin:
 #2 #3 #4

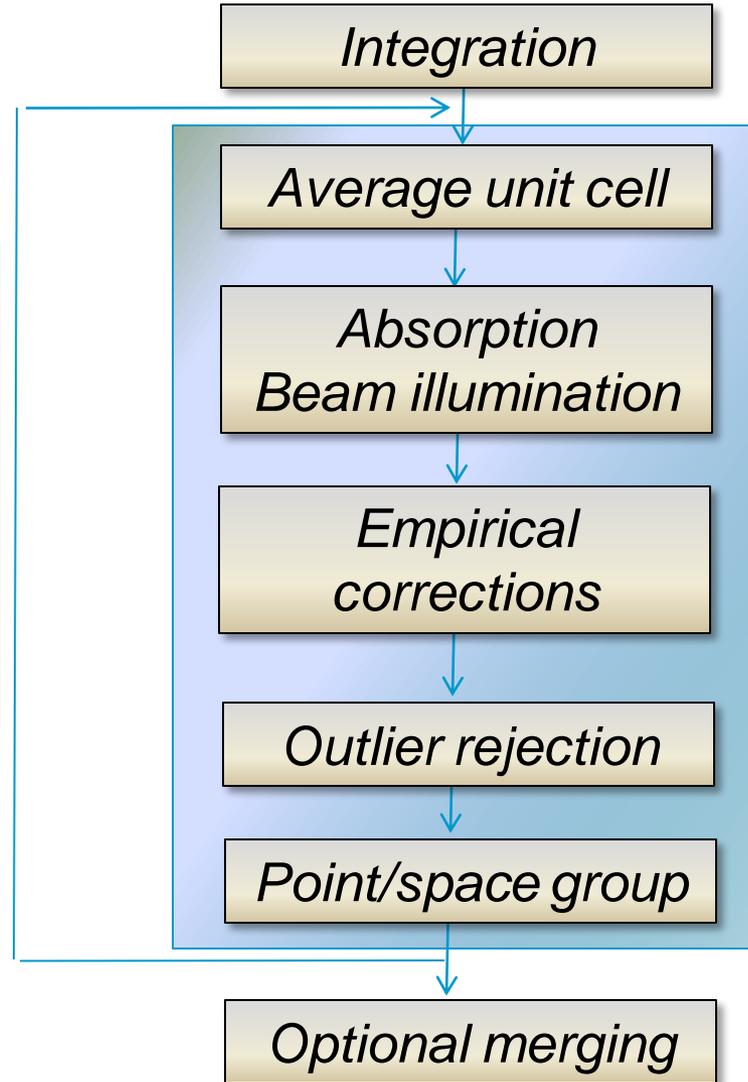
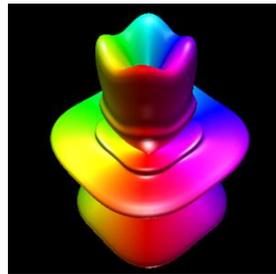
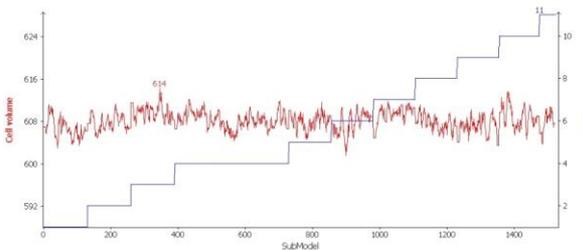
Useful for cases where structure solution is difficult due to low completeness of separate reflections

Special case... Needs some explanation.

Good data quality through full post corrections



PG changed?



Thank you for listening!

Find out more at

www.rigaku.com