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.

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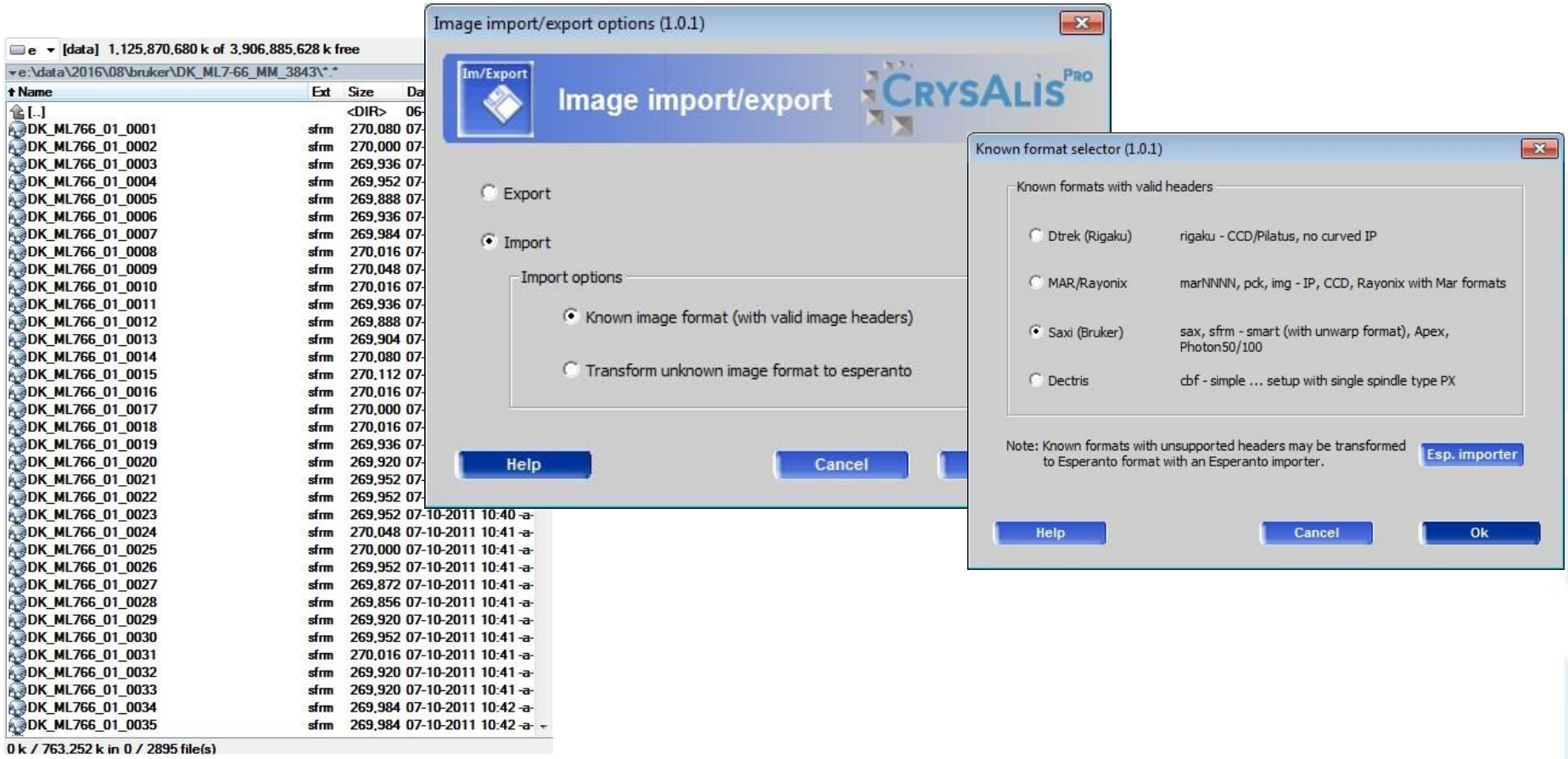
Data set import



- Open any existing experiment
- Import button

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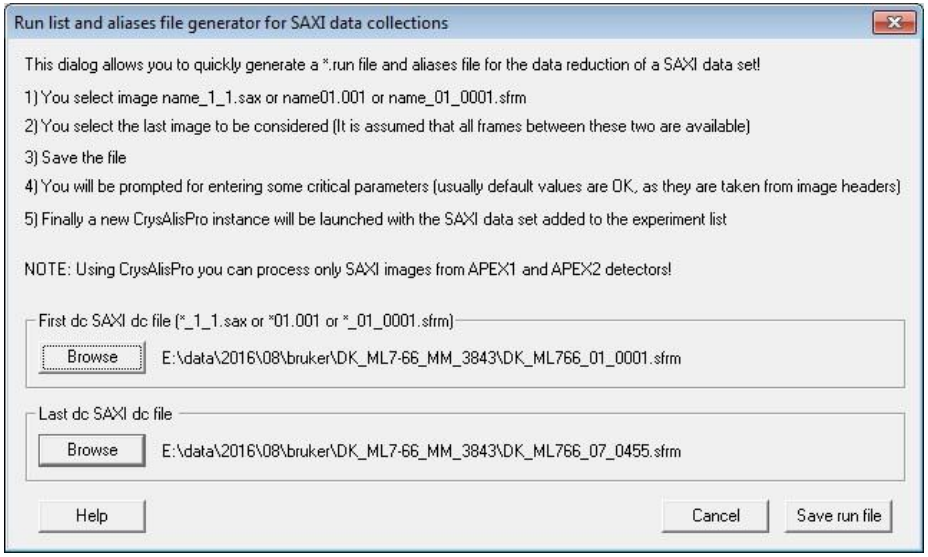
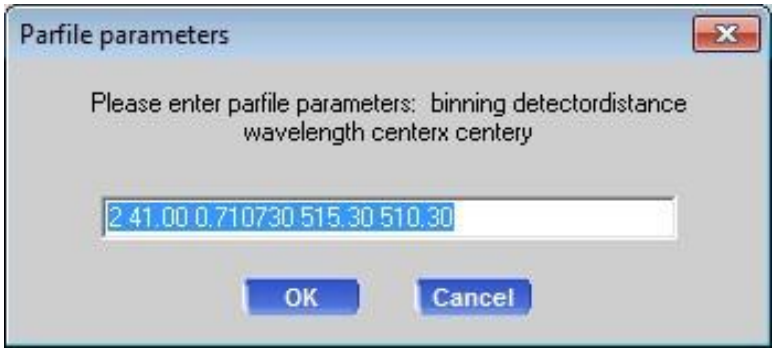
Data set import





- Open any existing experiment
- Import button

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Data set facts

- Mo, Iμs, no Si filter showoing 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

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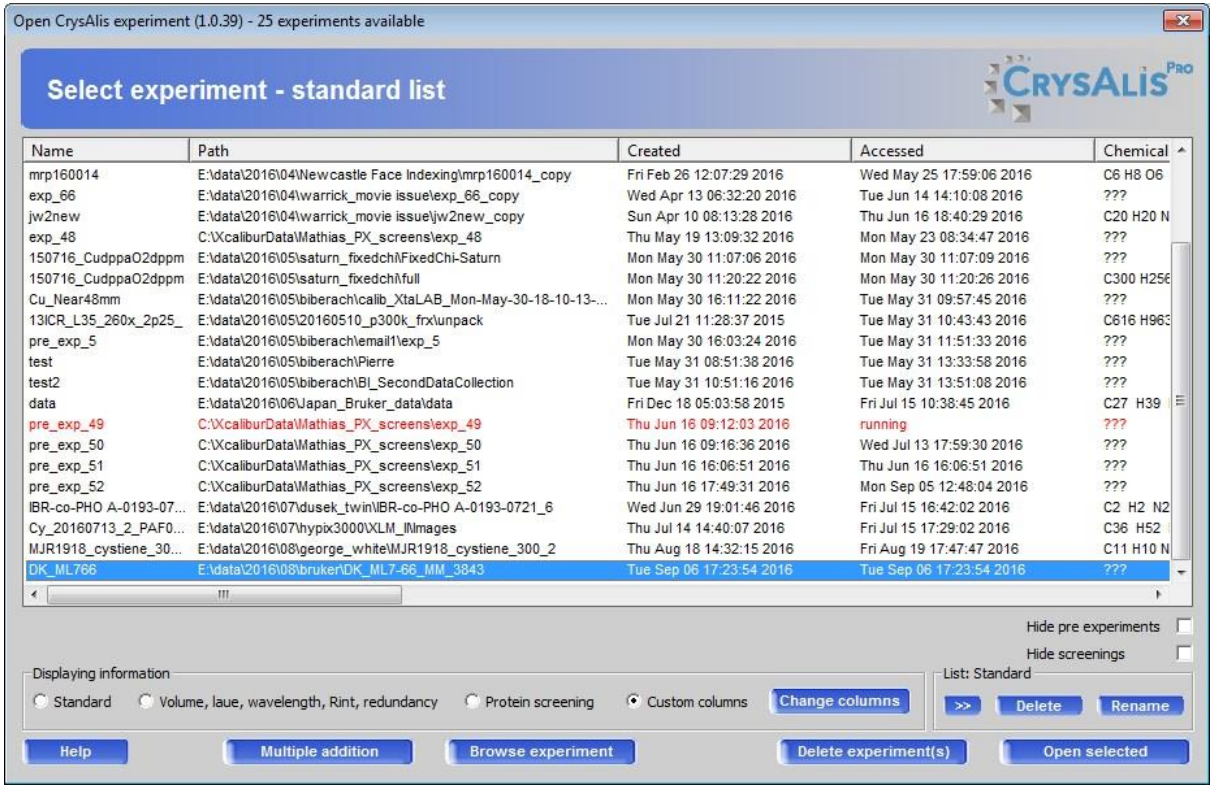
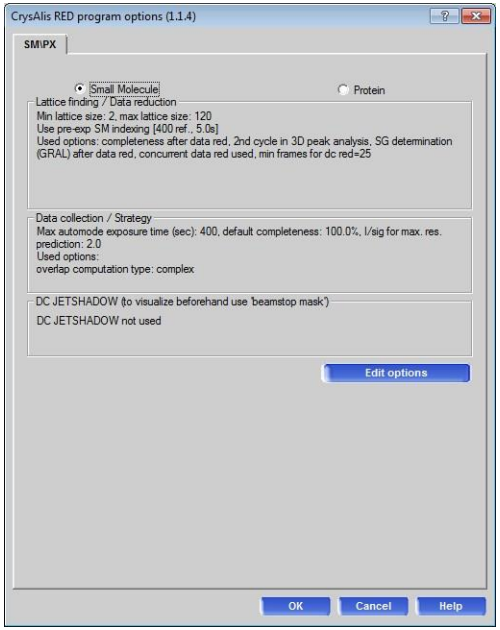
First opening of the data set

- Select SM/PX nature





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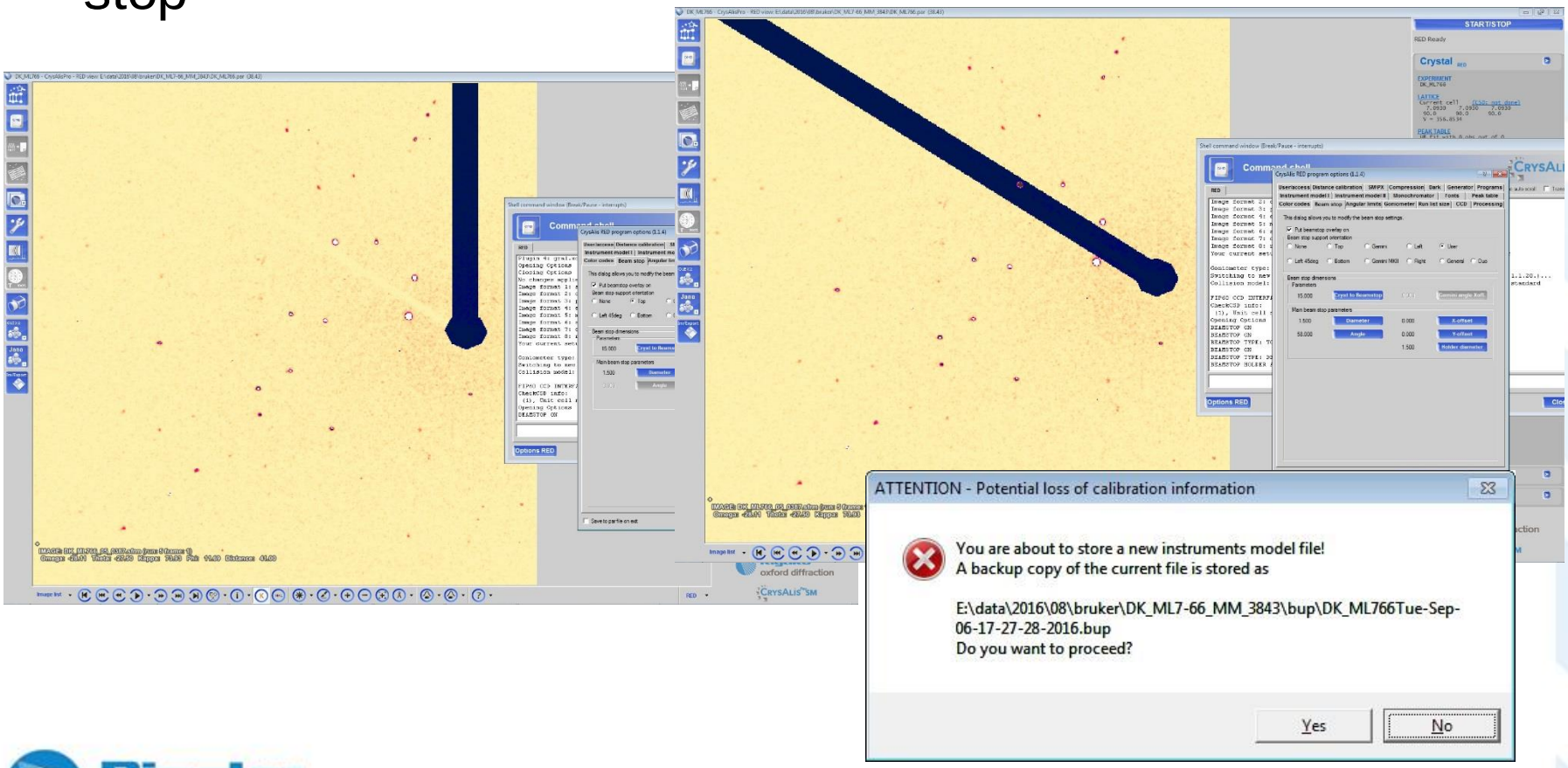
Set the beam stop

- Apex system sometimes use a user angle for the beam stop



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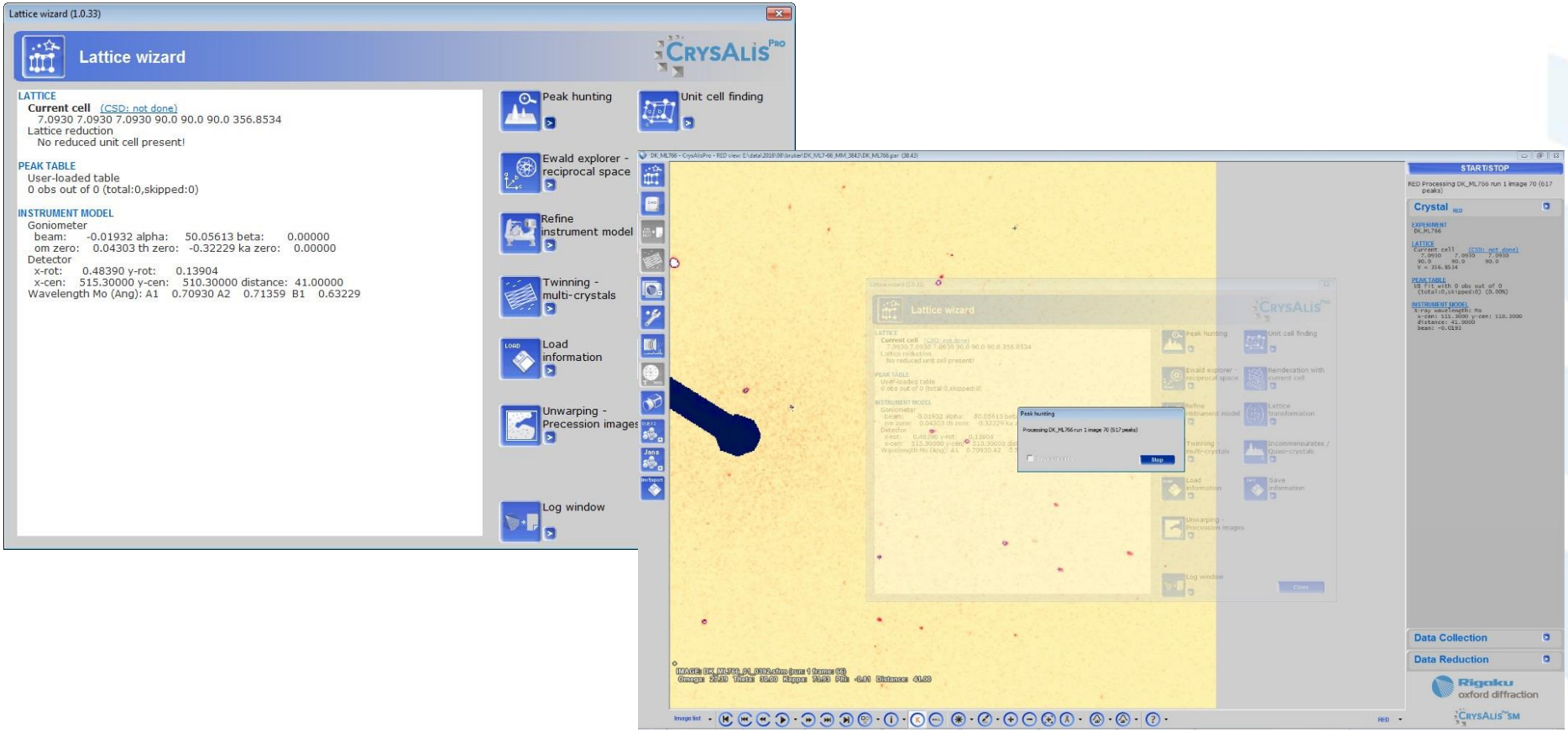
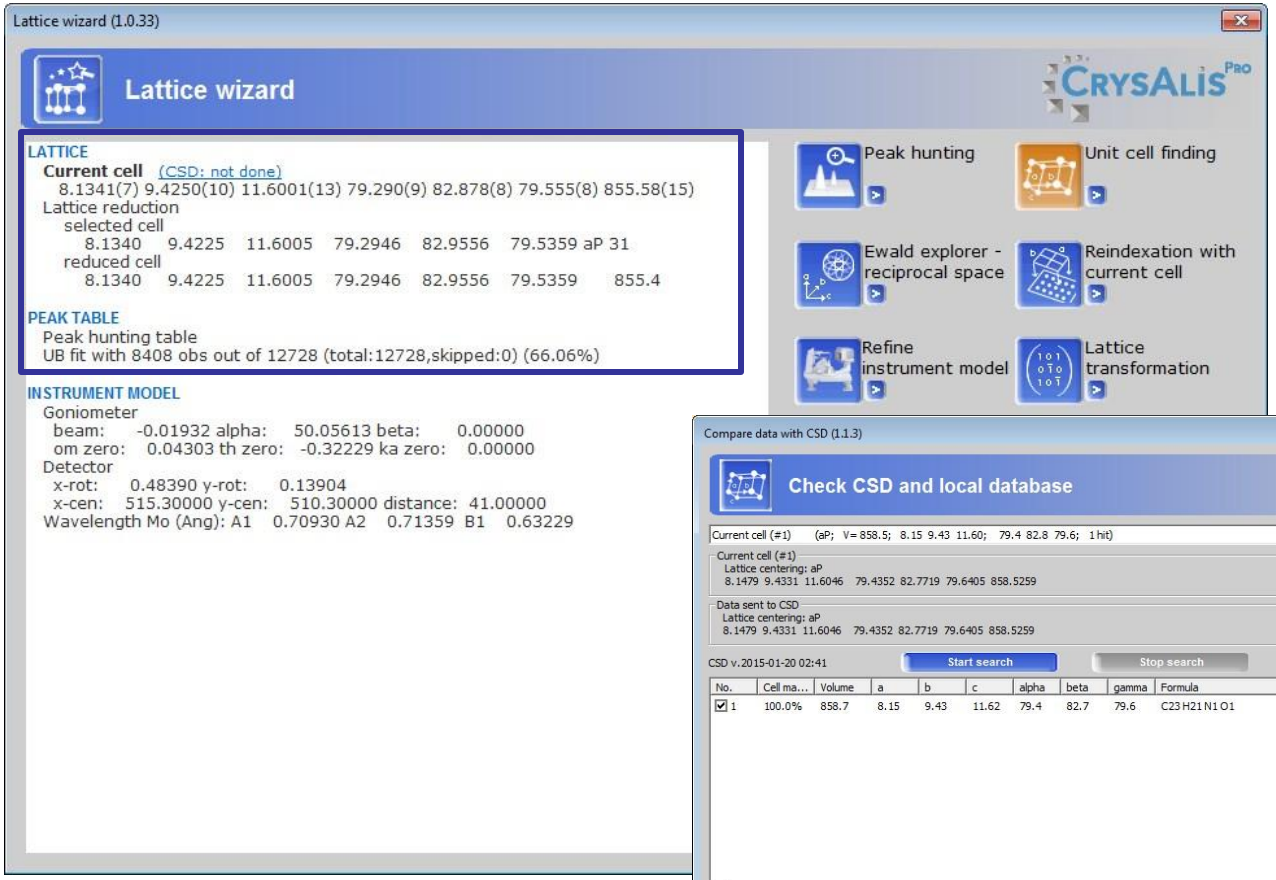
First peak hunting

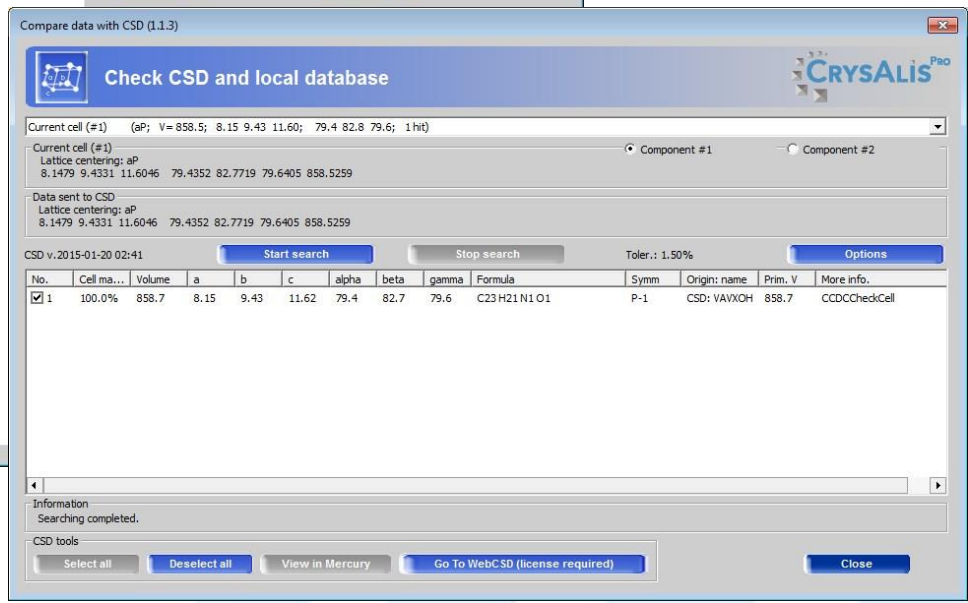
- Use default



Automatic unit cell finding

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





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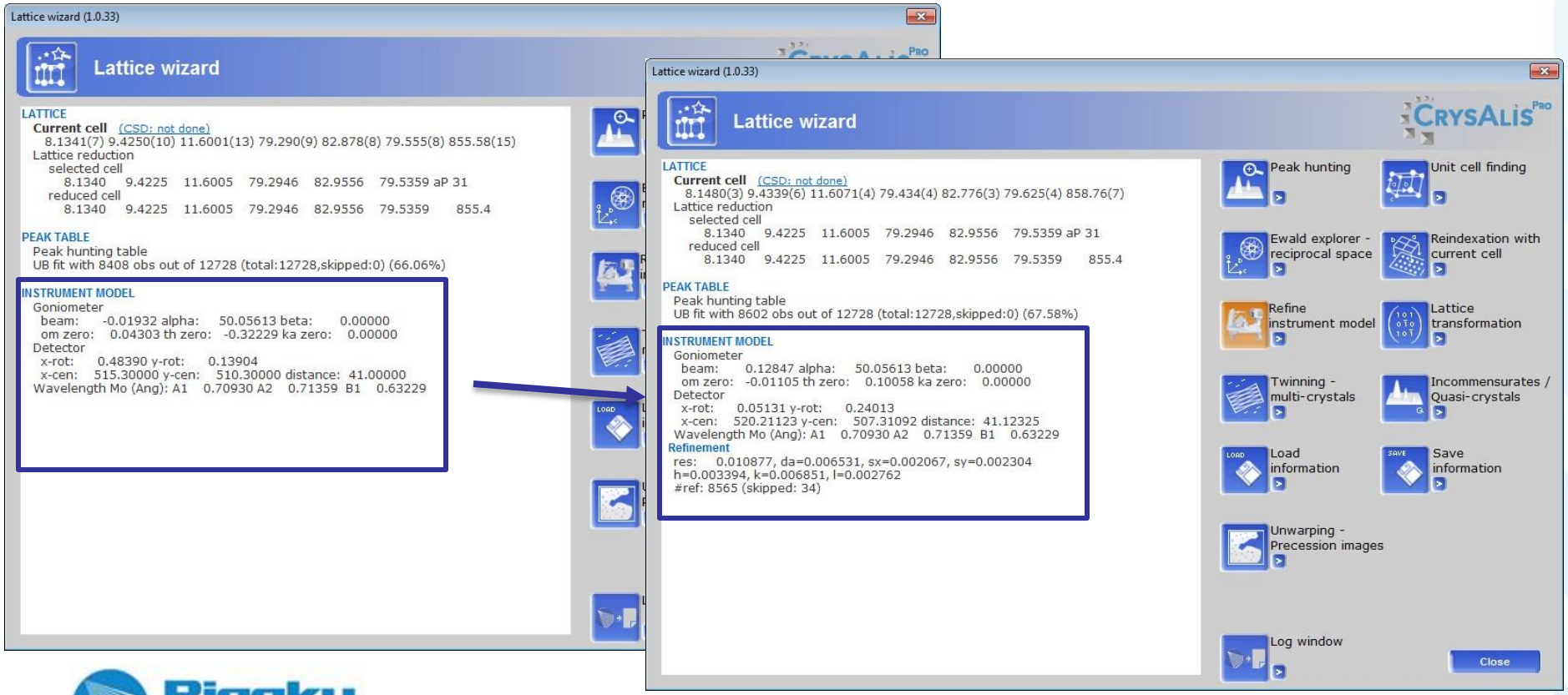
First instrument model refinement

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



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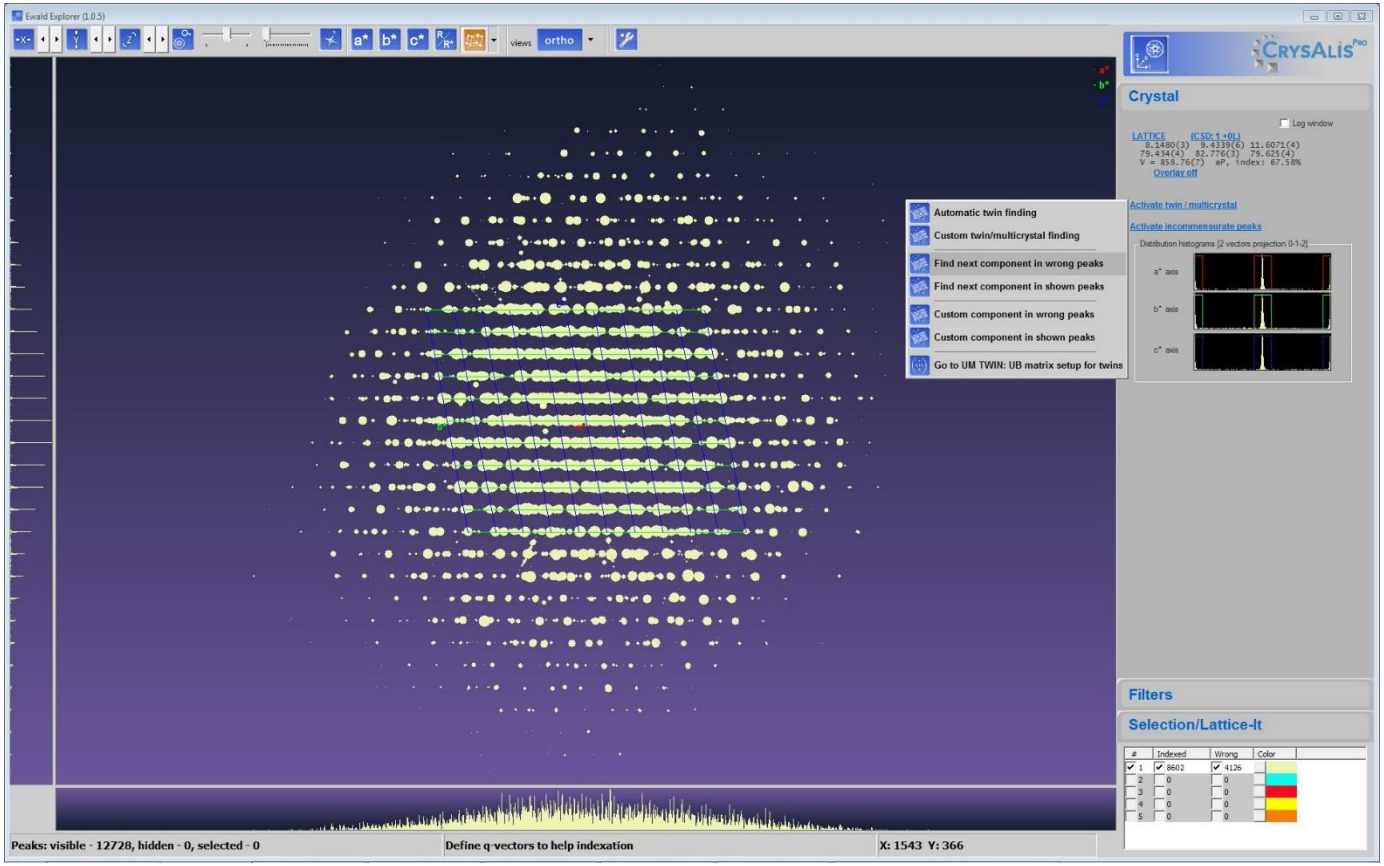
Use EwaldPro to find the twin

- Find next component in wrong peaks



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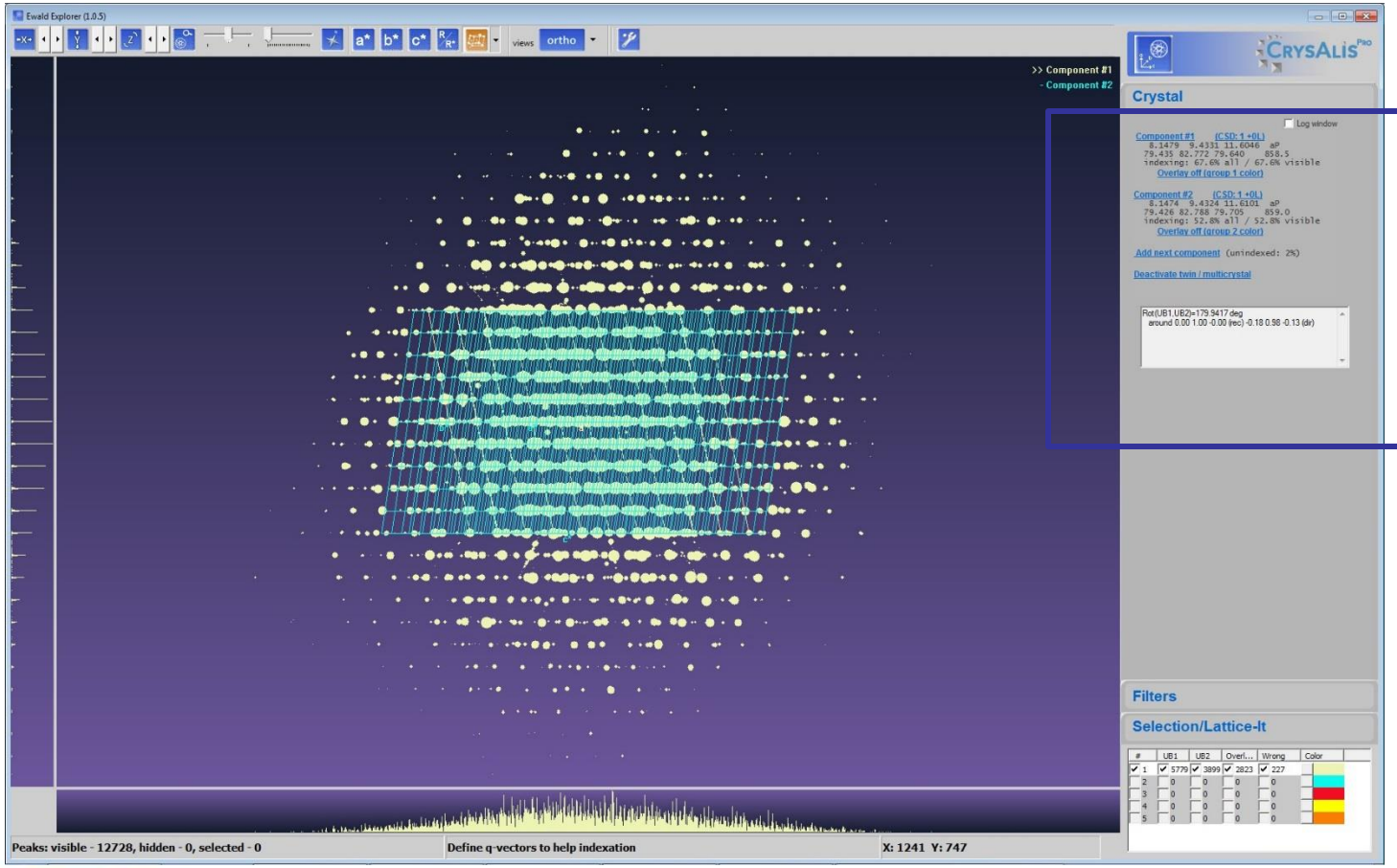
Use EwaldPro to find the twin

- Easy: 180 deg rotation twin



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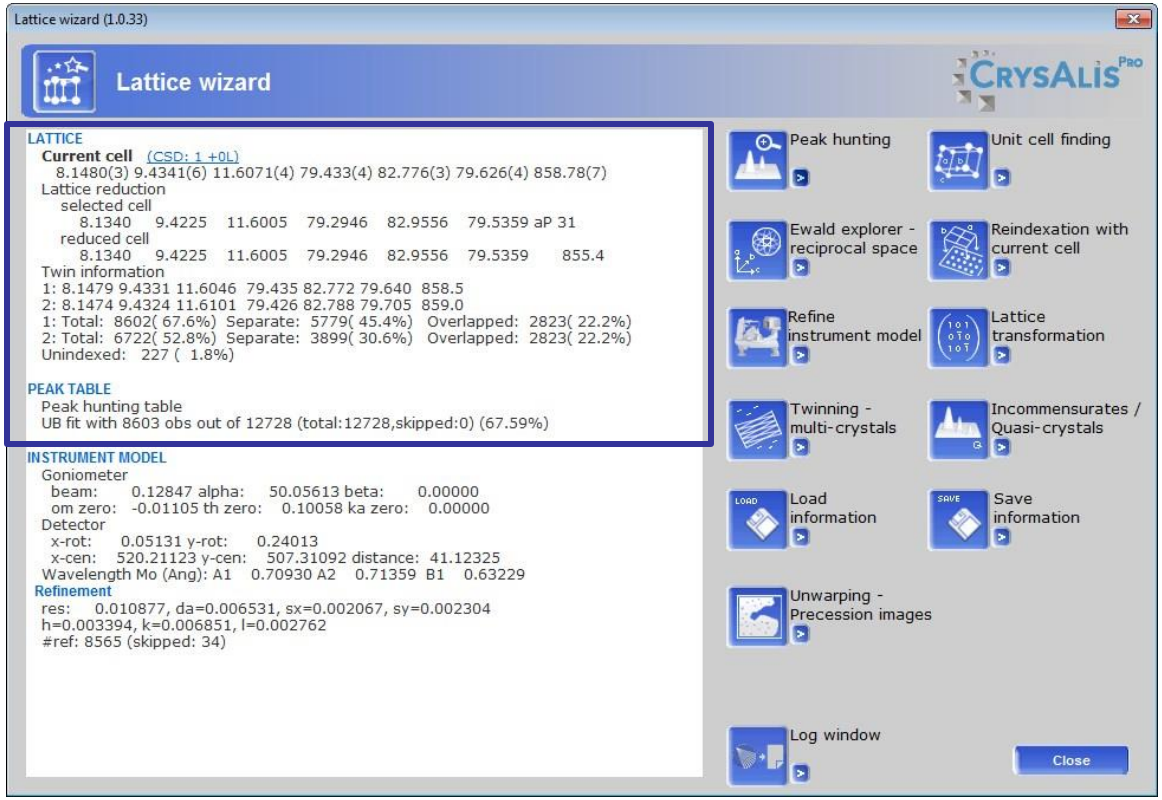
Use EwaldPro to find the twin

- Back to lattice wizard…



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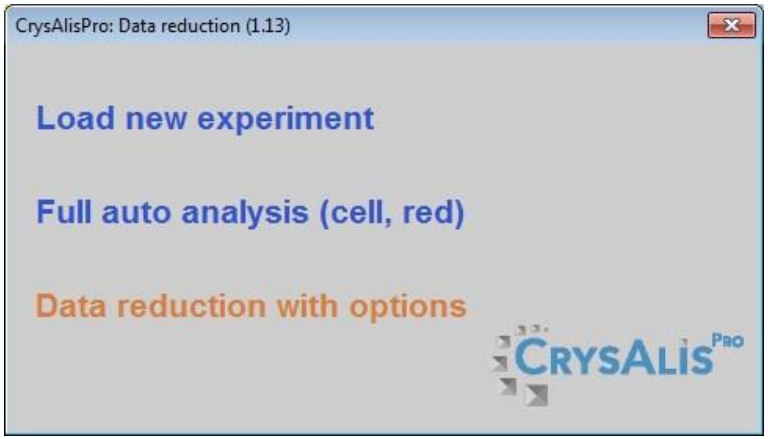
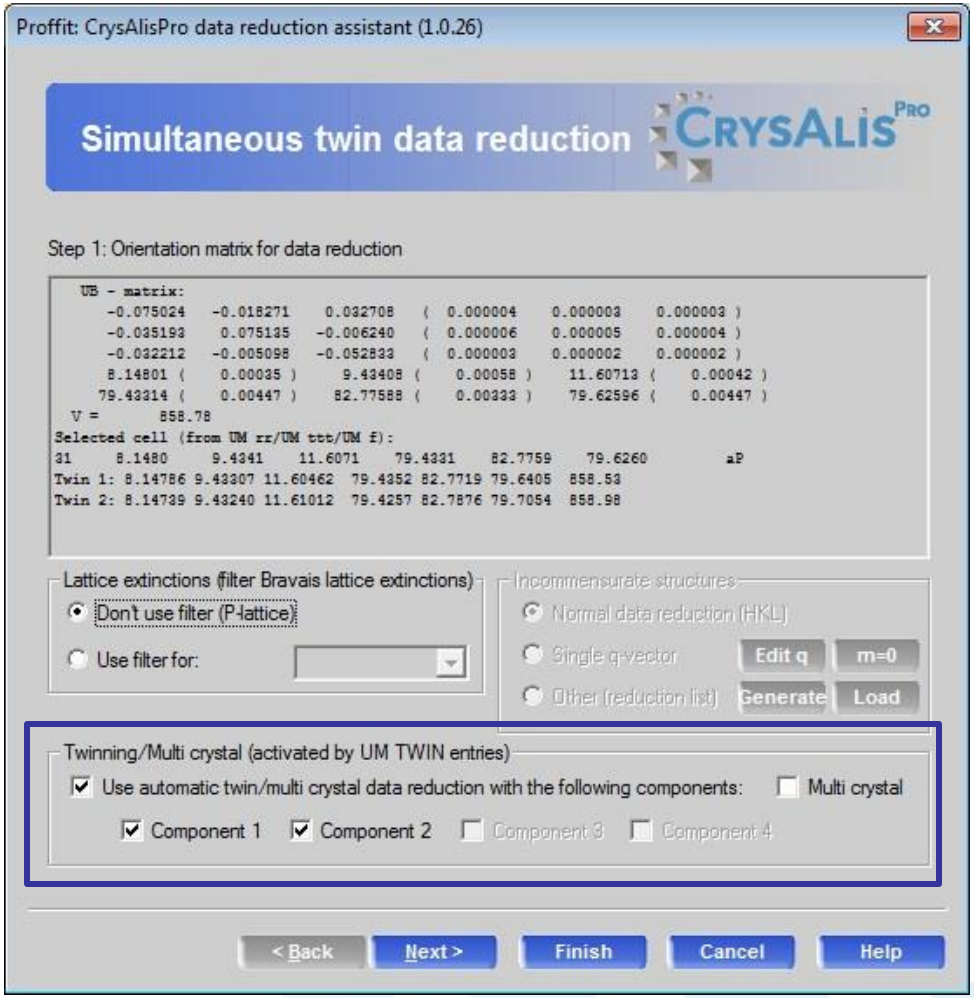
Do twin data reduction

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





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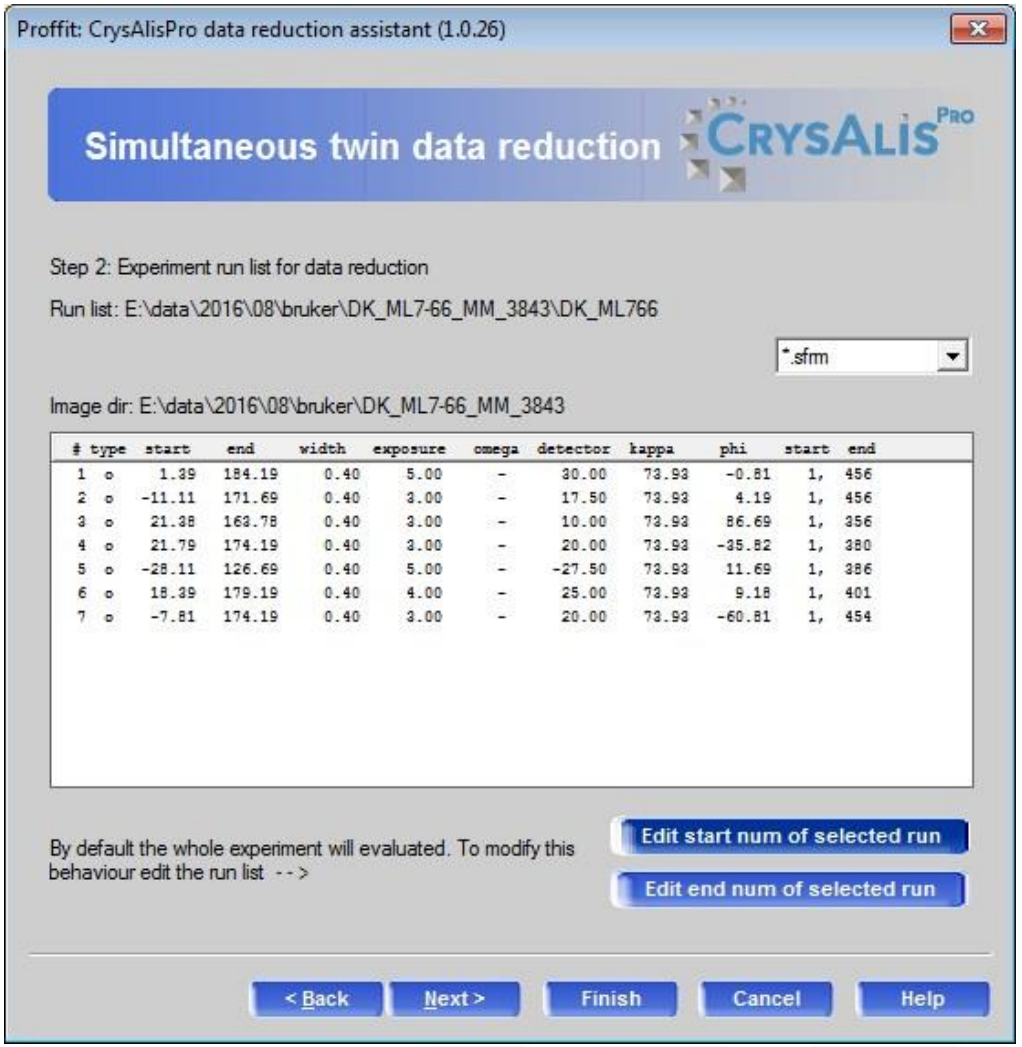
Do twin data reduction

- Accept the run list



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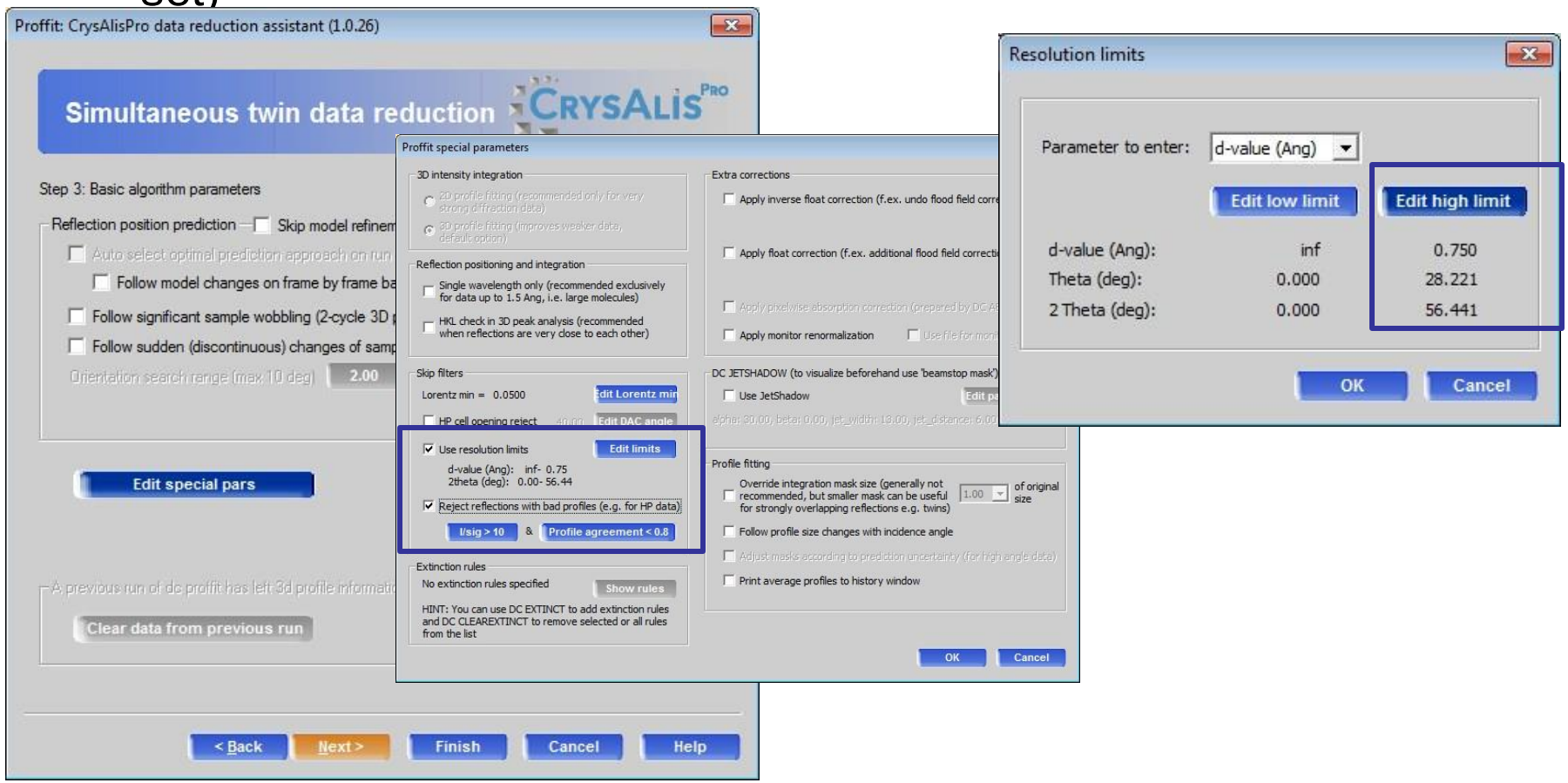
Do twin data reduction

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



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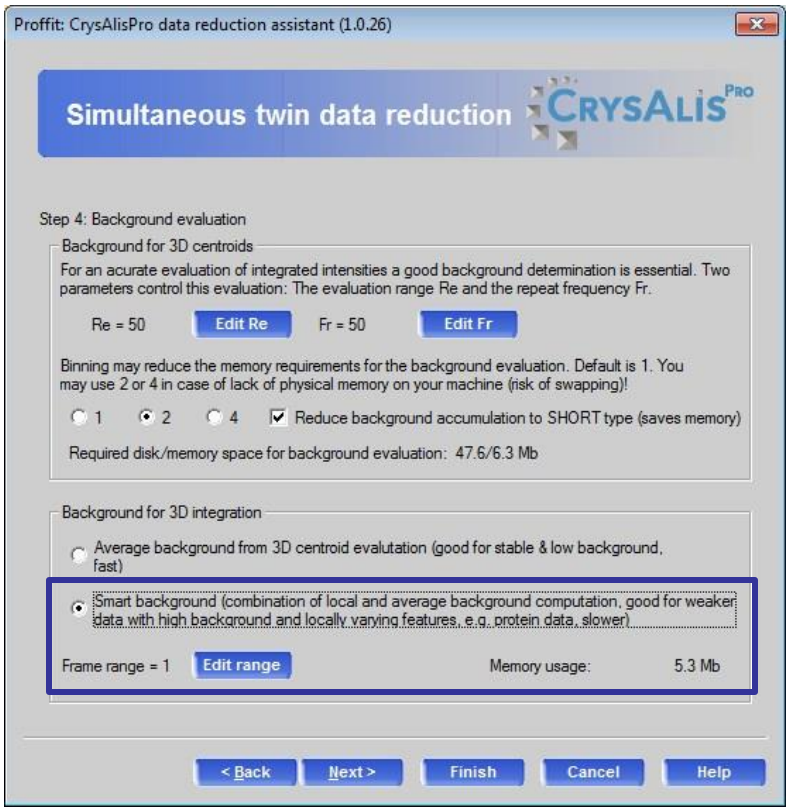
Do twin data reduction

- 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.

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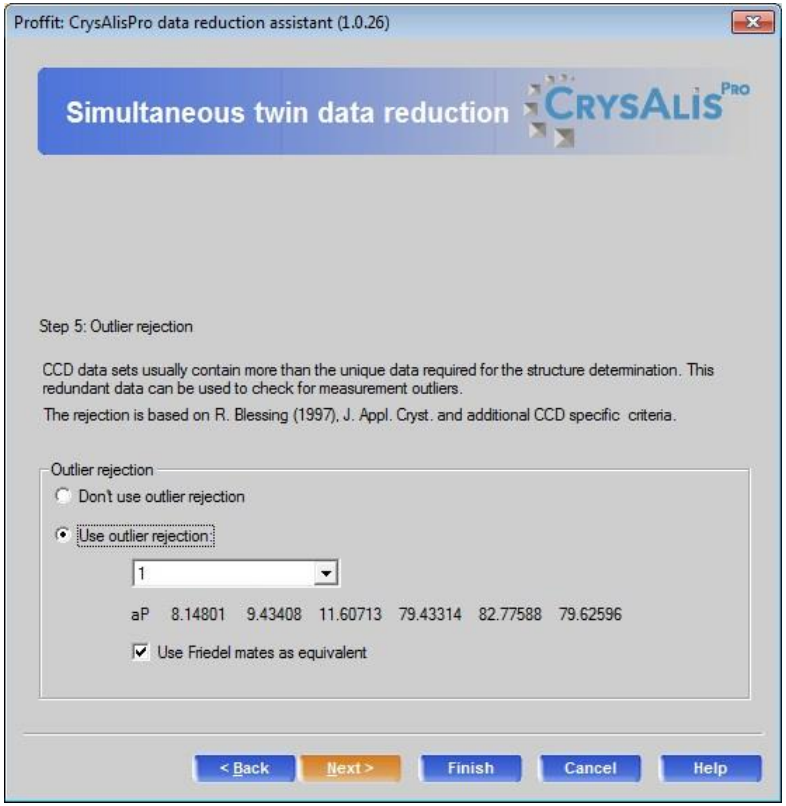
Do twin data reduction

- Automatic outlier rejection: default



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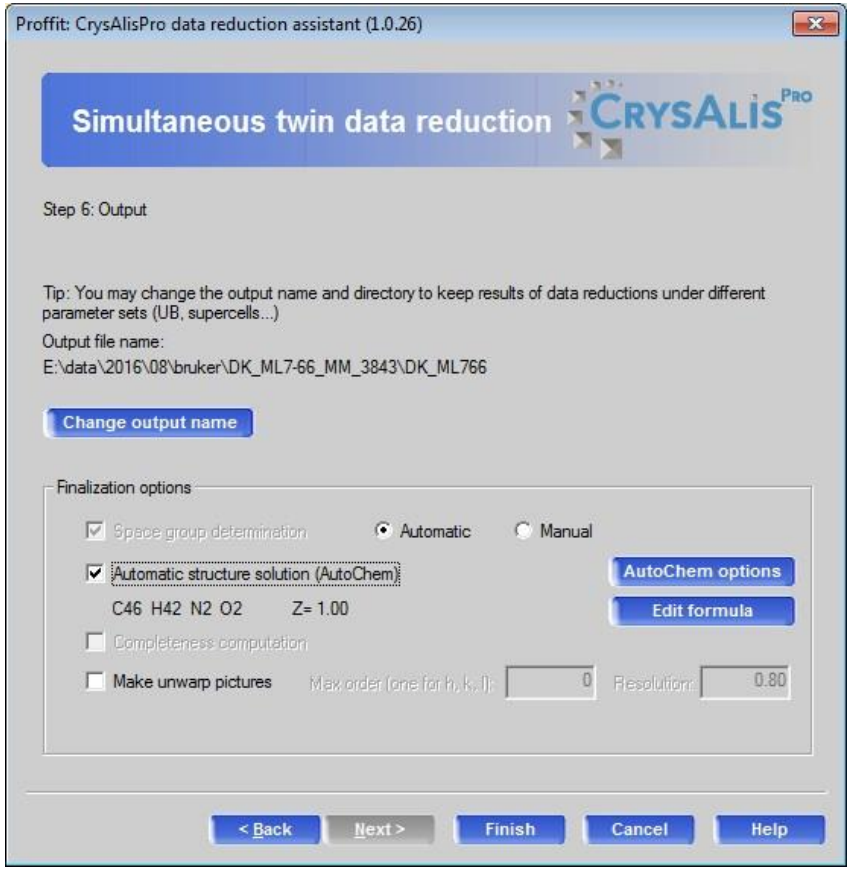
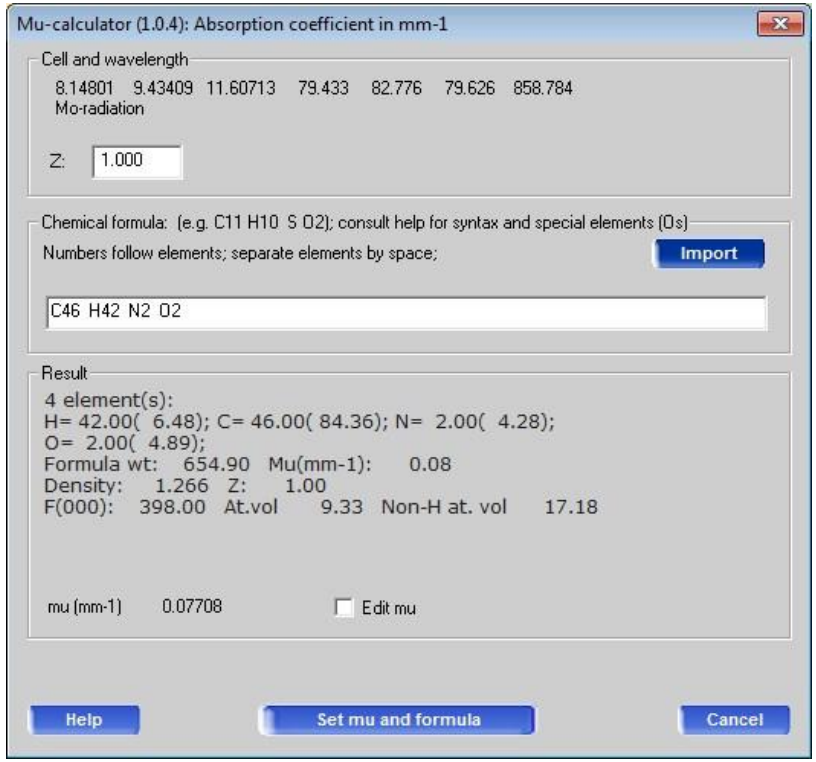
Do twin data reduction

- Chem. formula imported for AutoChem





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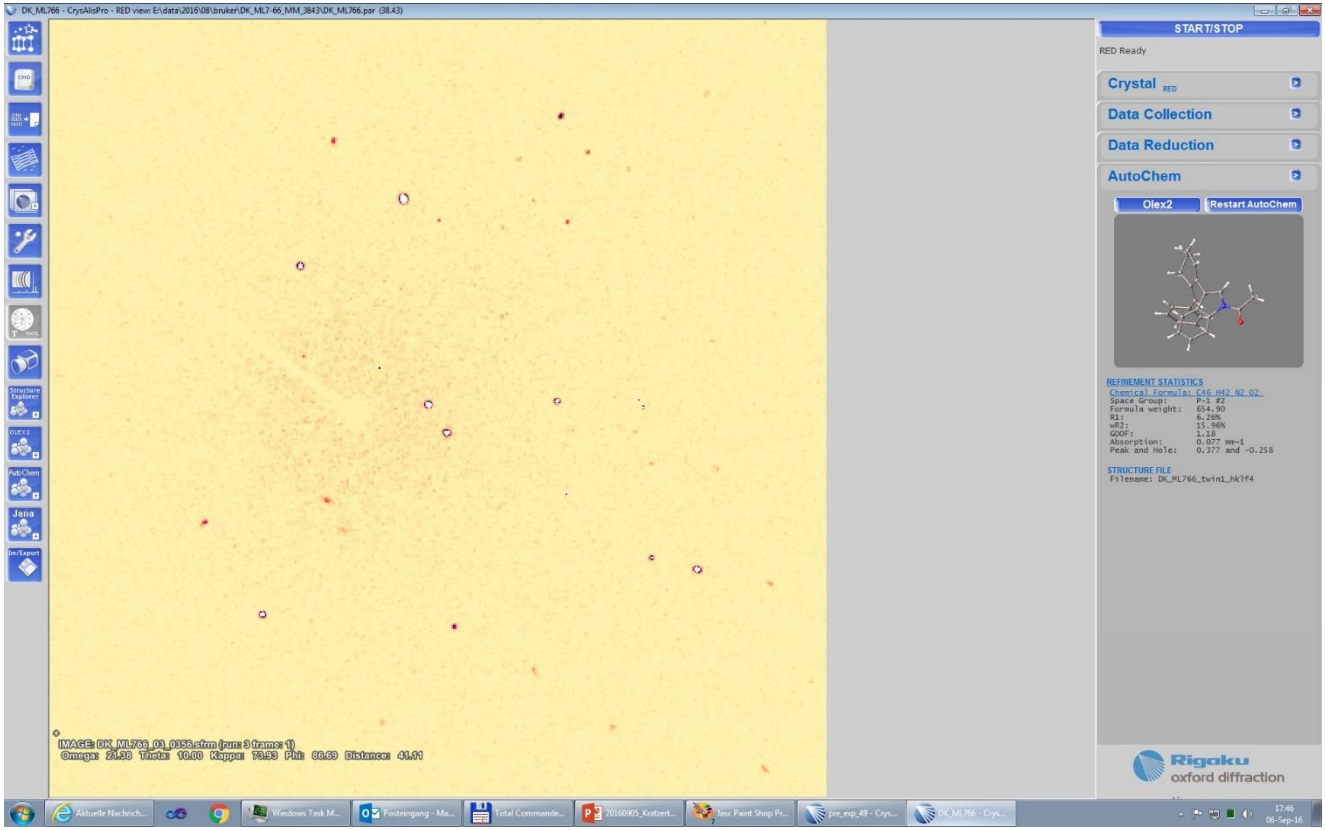
Automatic result

- Auto: based on all hklf4 data.



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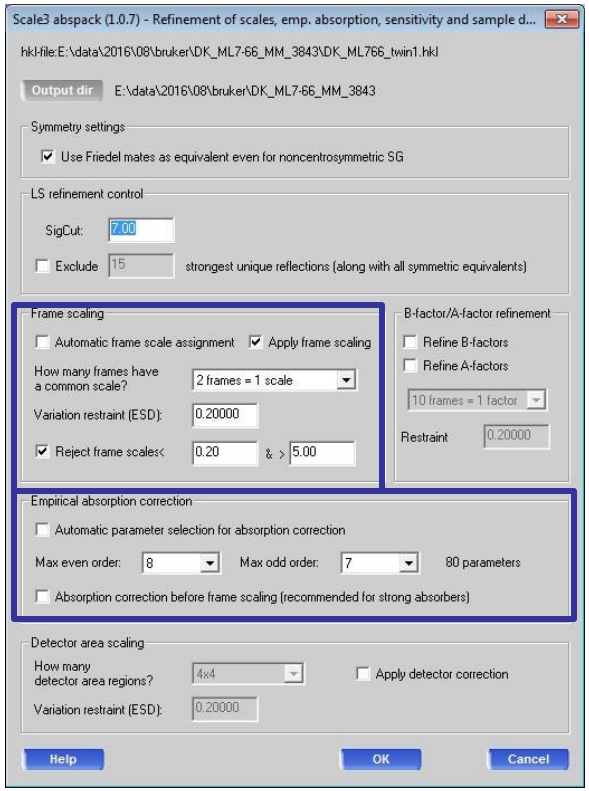
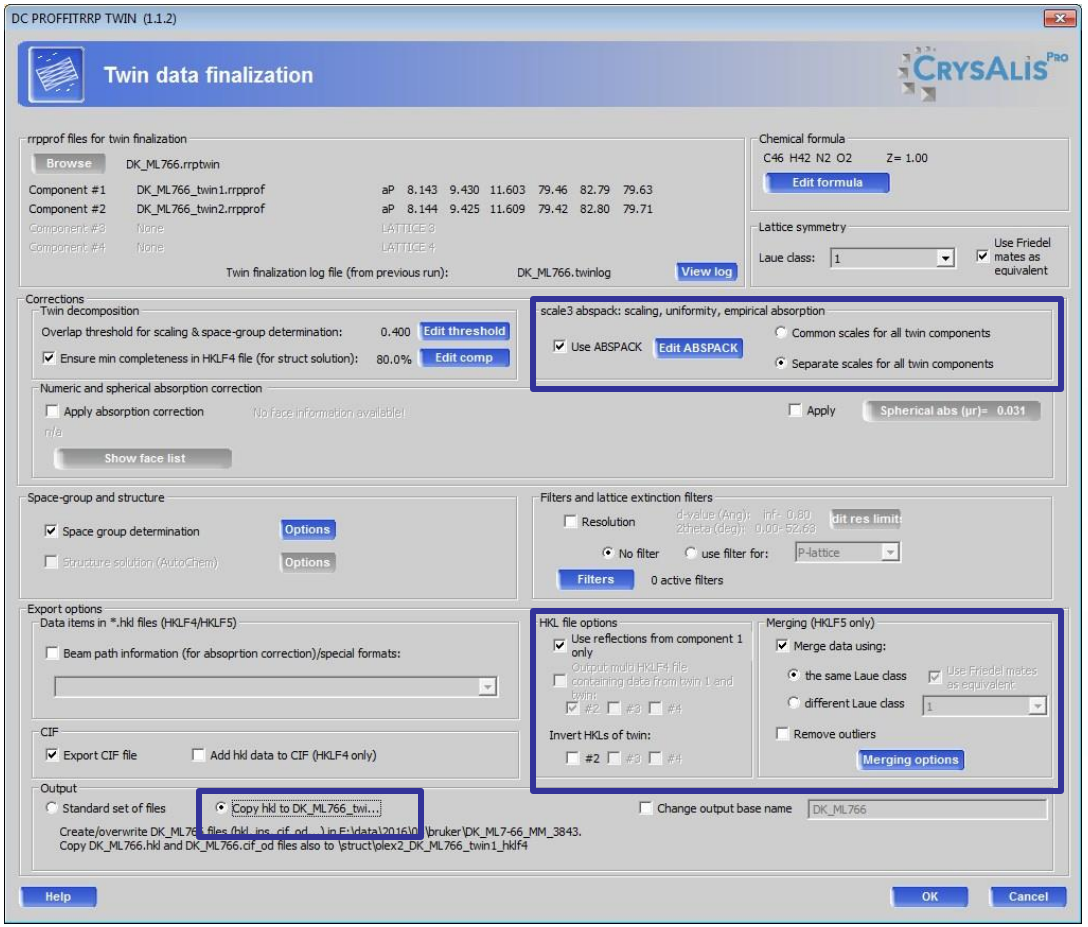
Tuning your result

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





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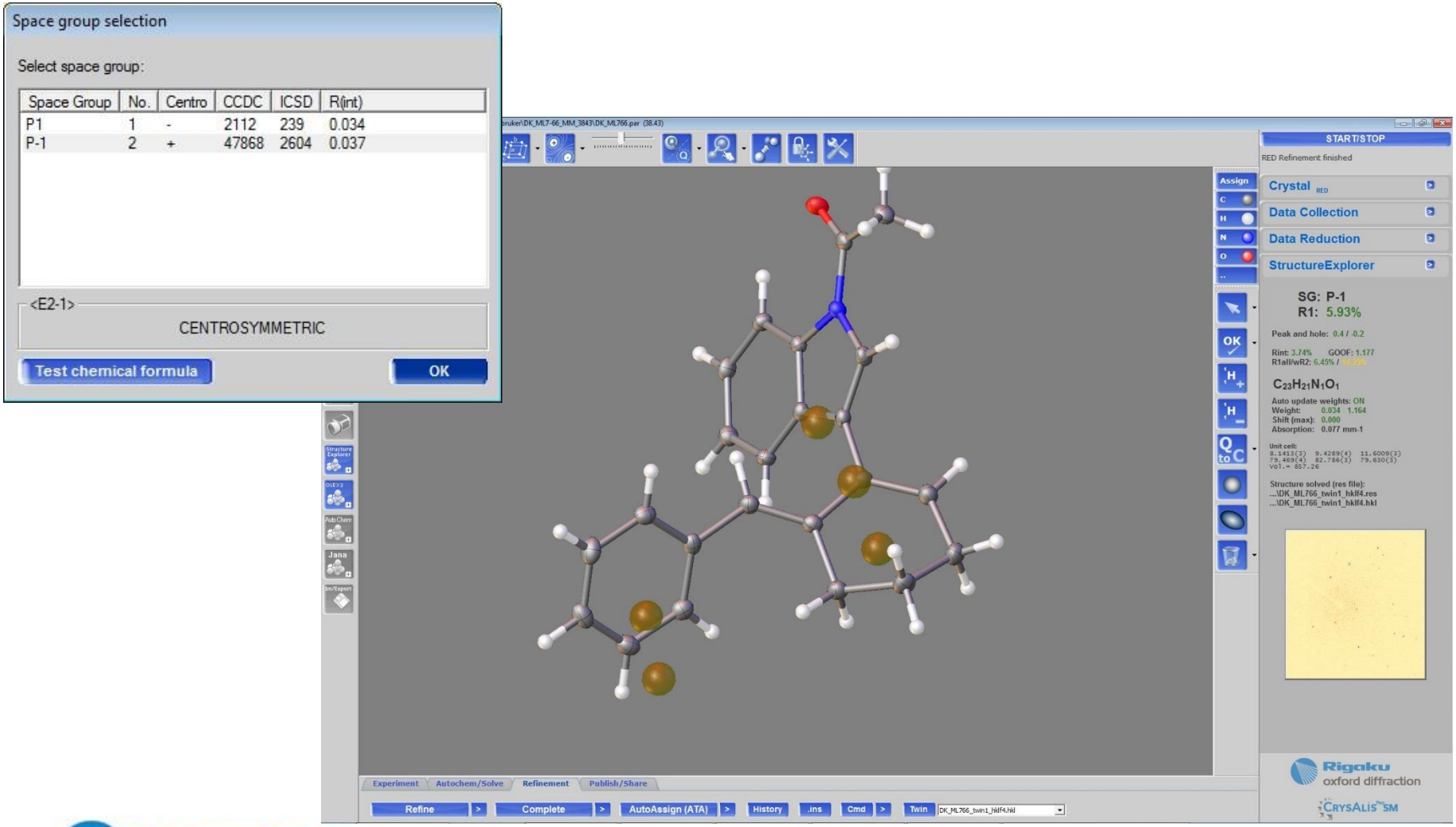
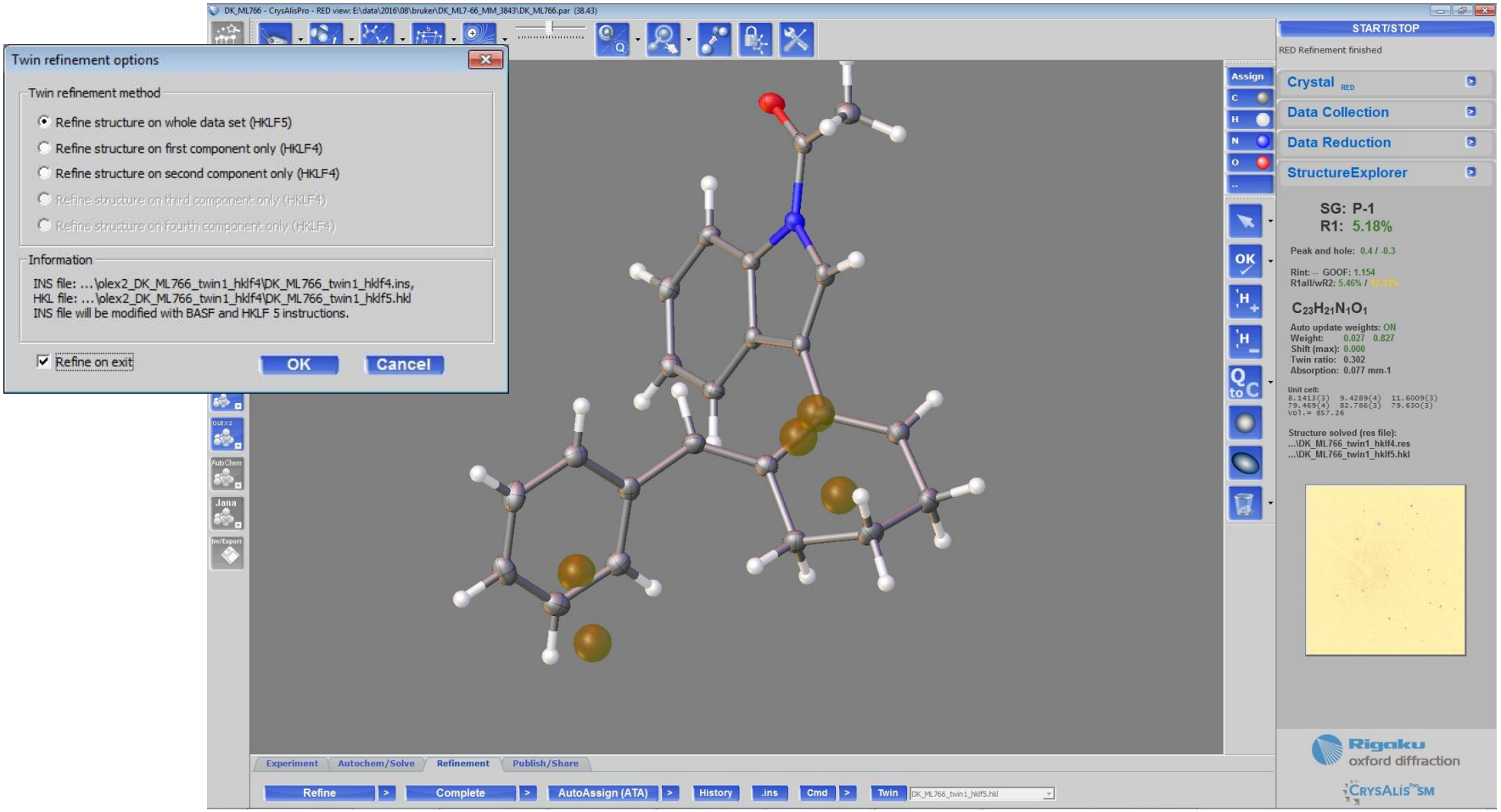
HKLF4 result



HKLF5 result



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 hydrogren adding, so the result was refined using Daniel's result.

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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

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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

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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

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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

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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>4sig) #>4sig	0.0472 (3582)	0.0527 (4229)
#reflections	4022	4453
#par	228	228
Twin ratio	0.29389	0.30205
I/sig	13.9	50.7

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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/sig 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.

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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

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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>4sig) #>4sig	0.0472 (3582)	0.0476 (3898)
#reflections	4022	4531
#par	228	228
Twin ratio	0.29389	0.30447
I/sig	13.9	17.5

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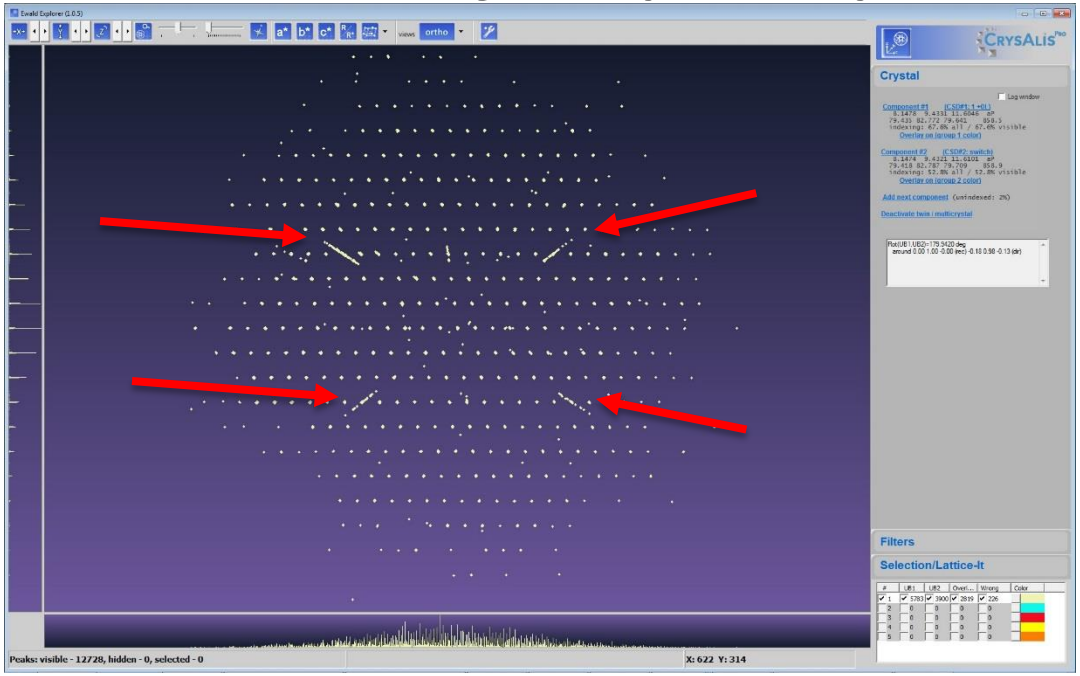
Extra mention 3l effect

- 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

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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

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Twinning*: 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*

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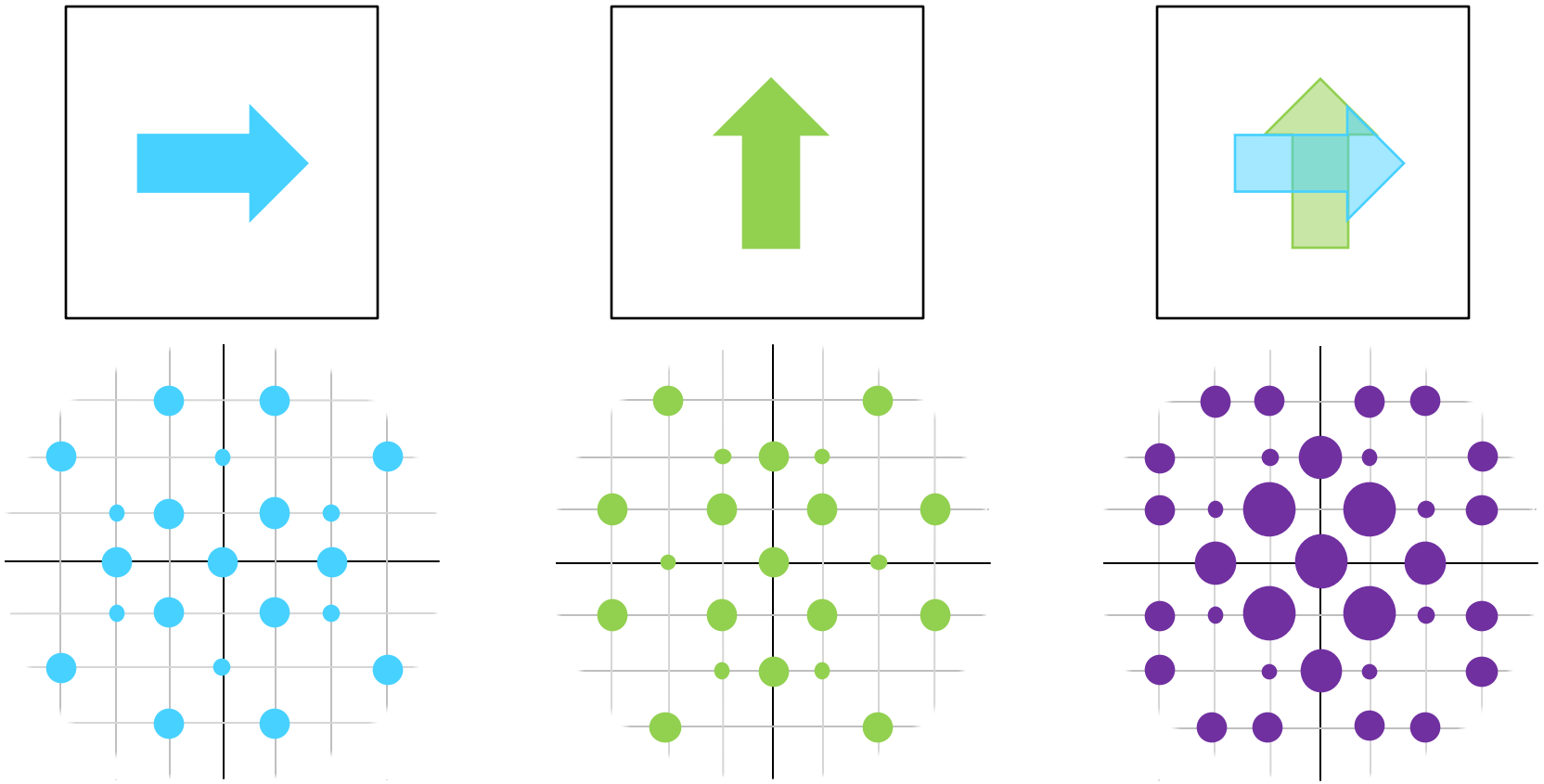
Introduction to Twinning

Fully overlapped diffraction patterns

Merohedral twins

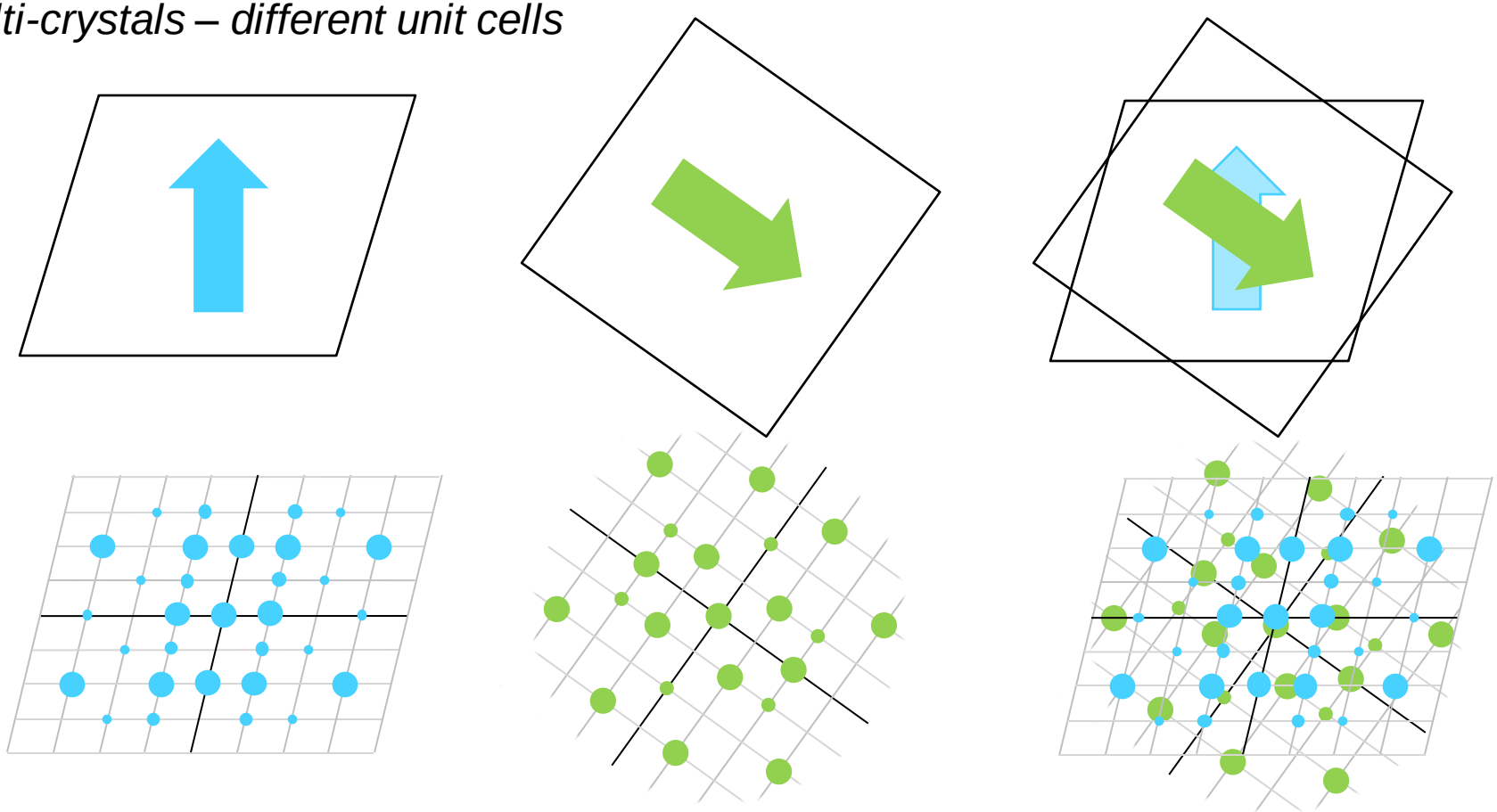


Component 1 *Component 2* *Overall*

Introduction to Twinning

Partially overlapped diffraction patterns

Multi-crystals – different unit cells



Component 1 *Component 2* *Overall*

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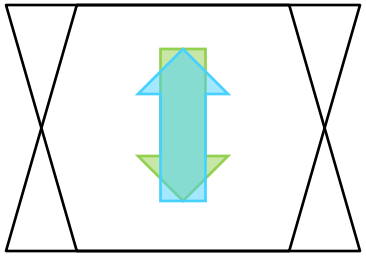
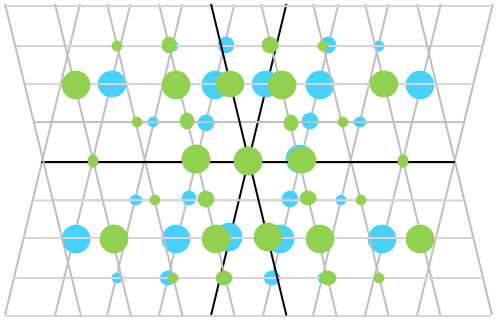
Introduction to Twinning





Fully overlapped diffraction patterns can't be separated at integration

Partially overlapped reflections can be treated during integration

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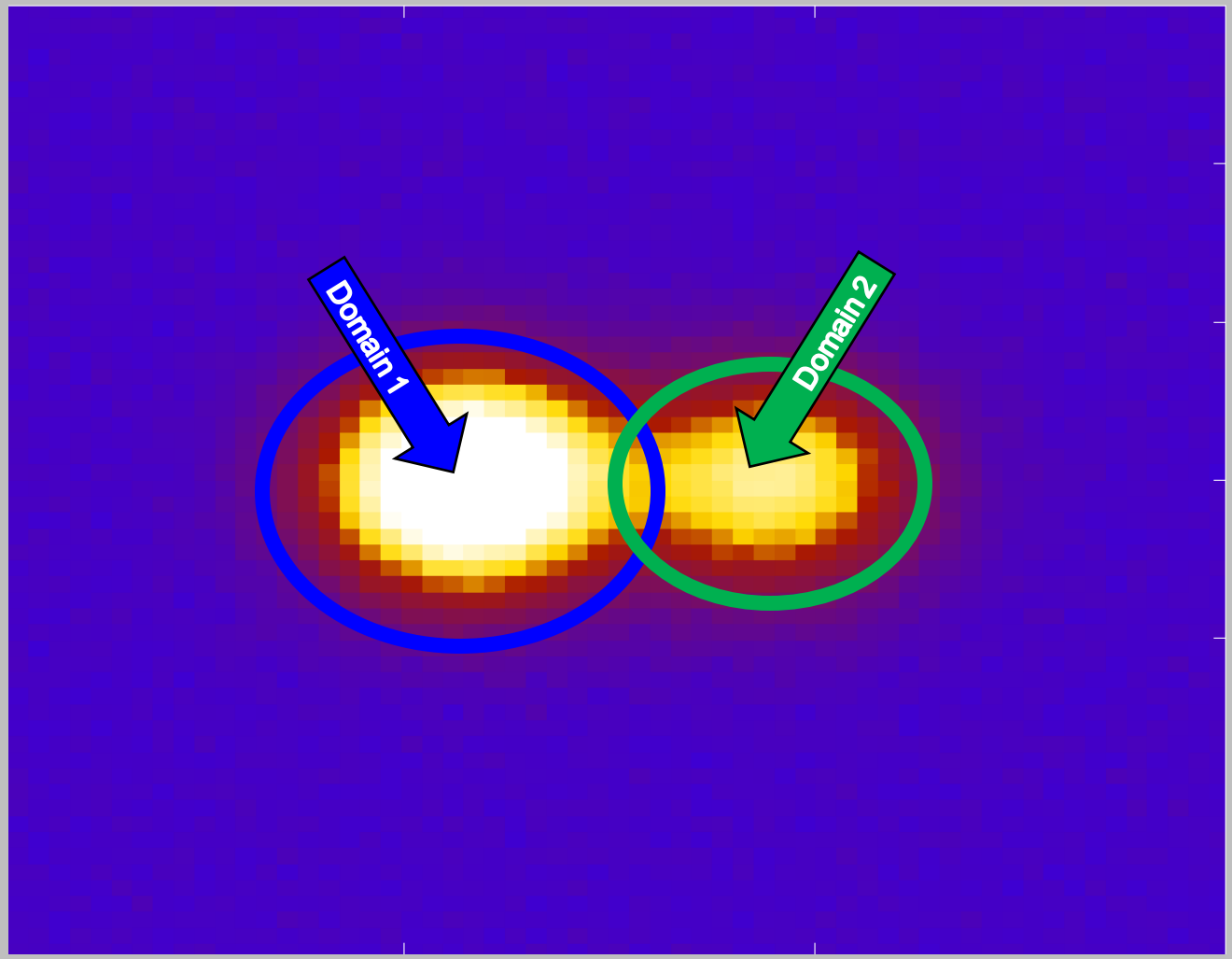
Twinning
Integration



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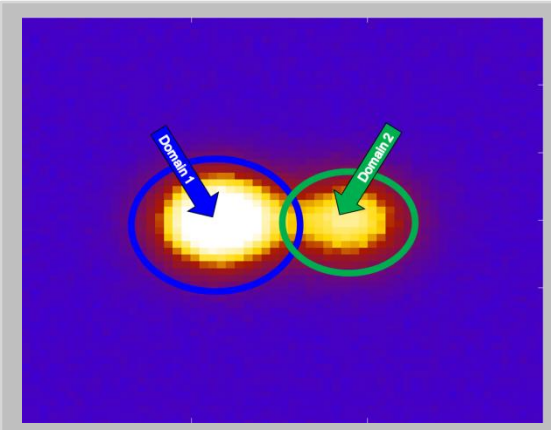
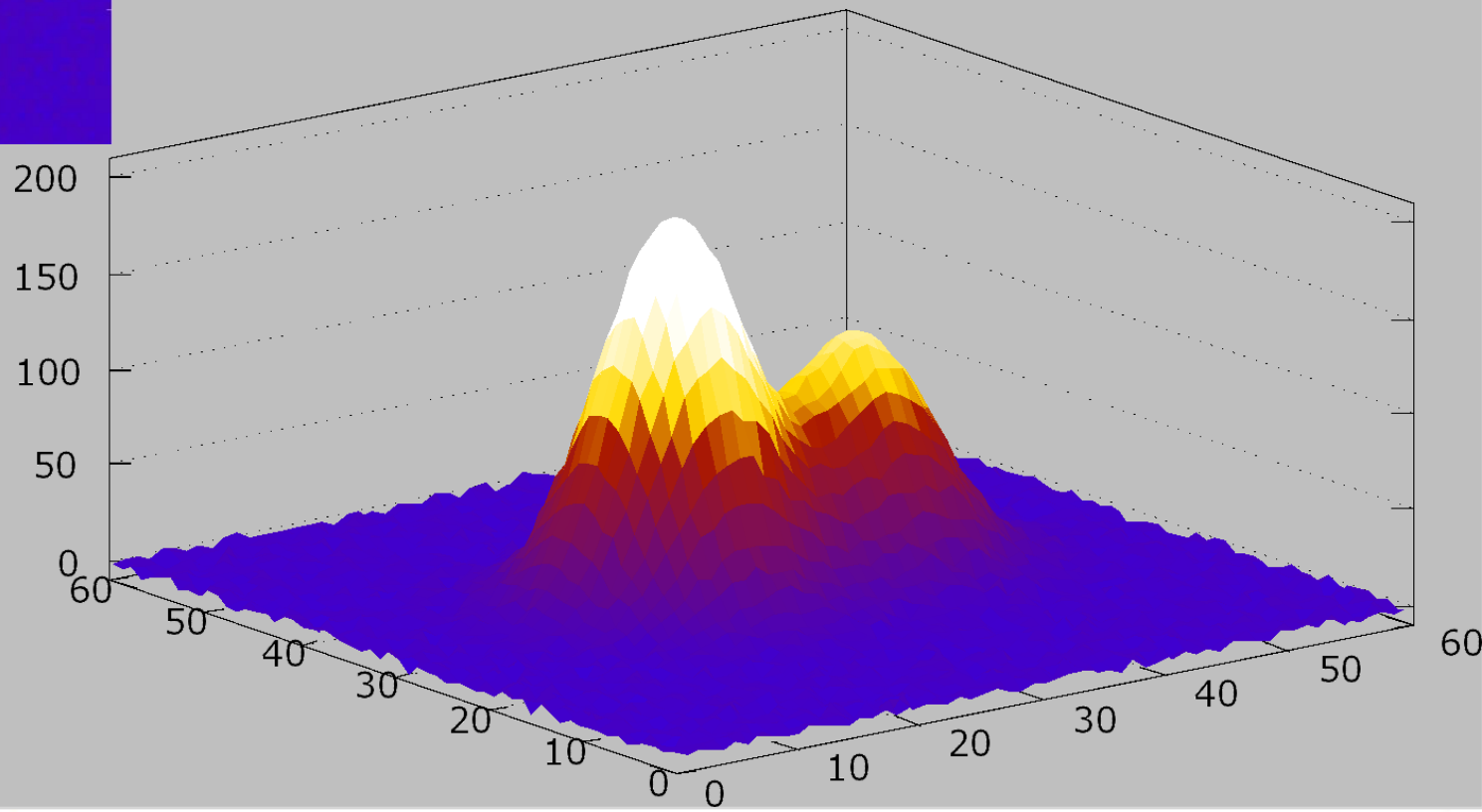
Twinning
Integration





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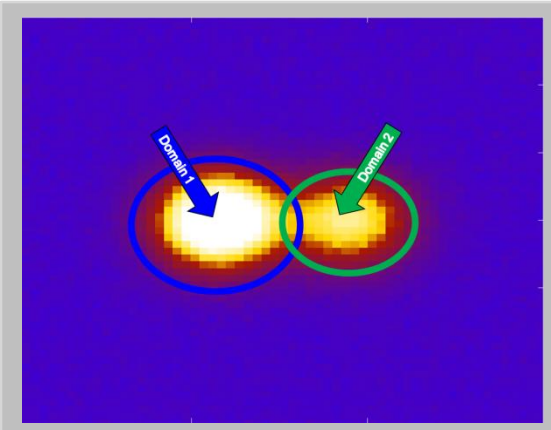
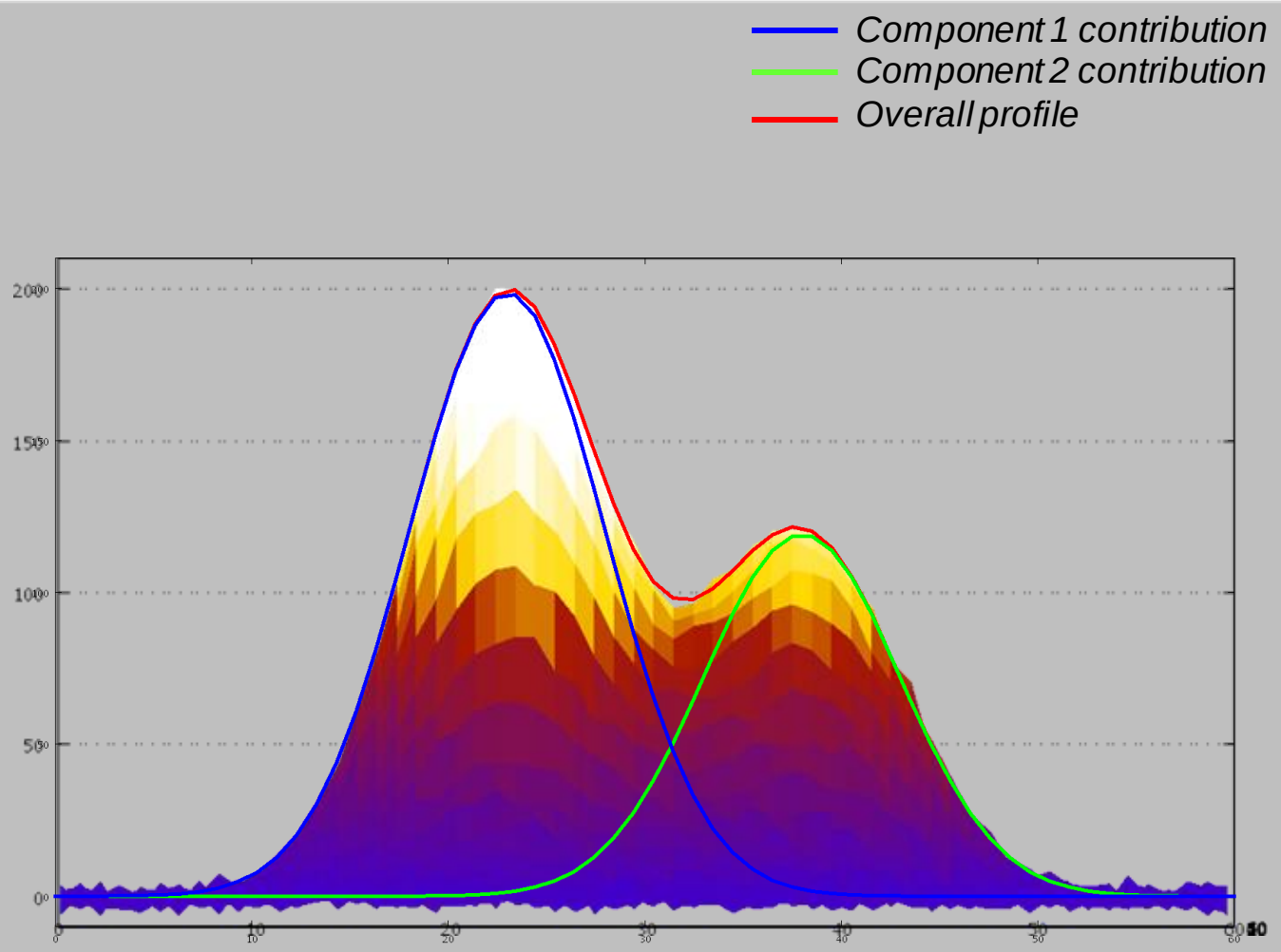
Twinning
Integration





Current vs. new approach

Current twin integration (36)

Component #1 PROFFIT

Component #2 PROFFIT

Less accurate HKLF4 and HKFL5 intensities

Intensity decomposition during finalization

New simultaneous integration (37)

Profile decomposition based on profile fitting of the overlapped area

More accurate profile-fitted HKLF4 intensity

Very accurate HKLF5 intensity

Twin finalization

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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)

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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

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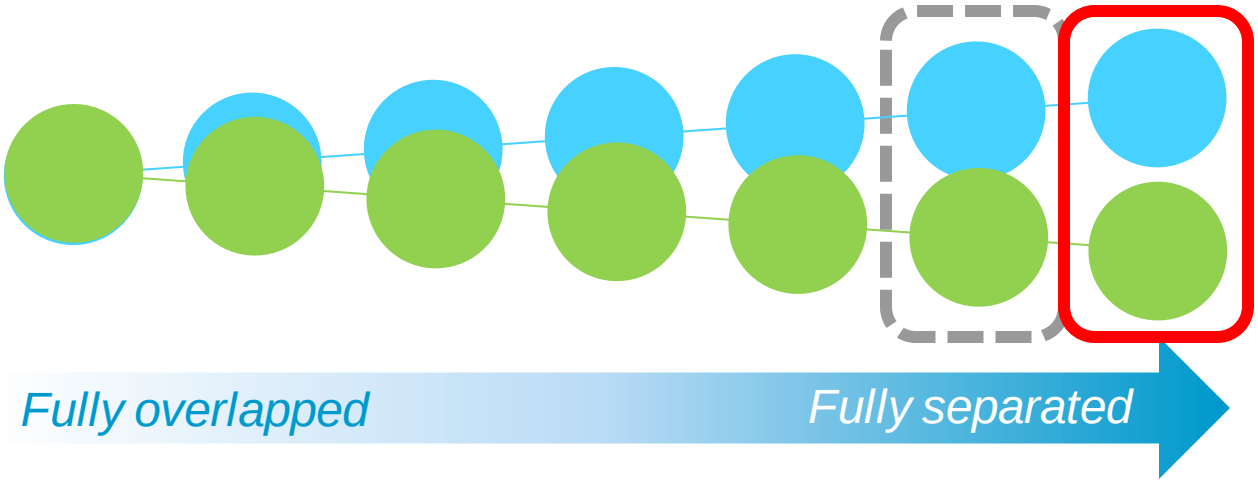
The Twin Challenge

Integration treatments – profile size

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



Component 1 profile mask

Component 2 profile mask

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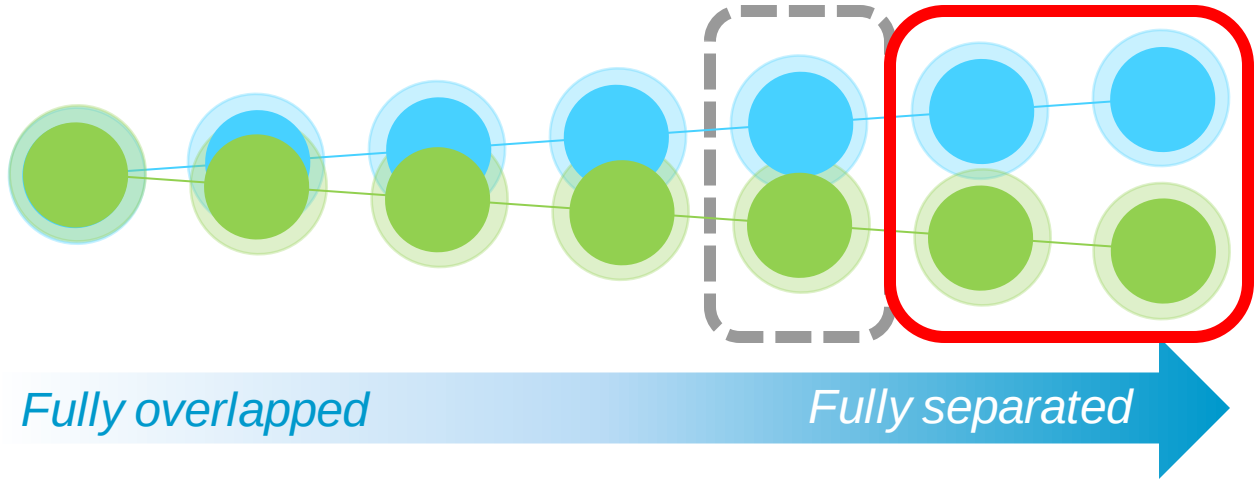
Twinning

Integration treatments – profile size

Using smaller masks can reduce overlap meaning more separate reflections



Component 1 profile mask

Component 2 profile mask

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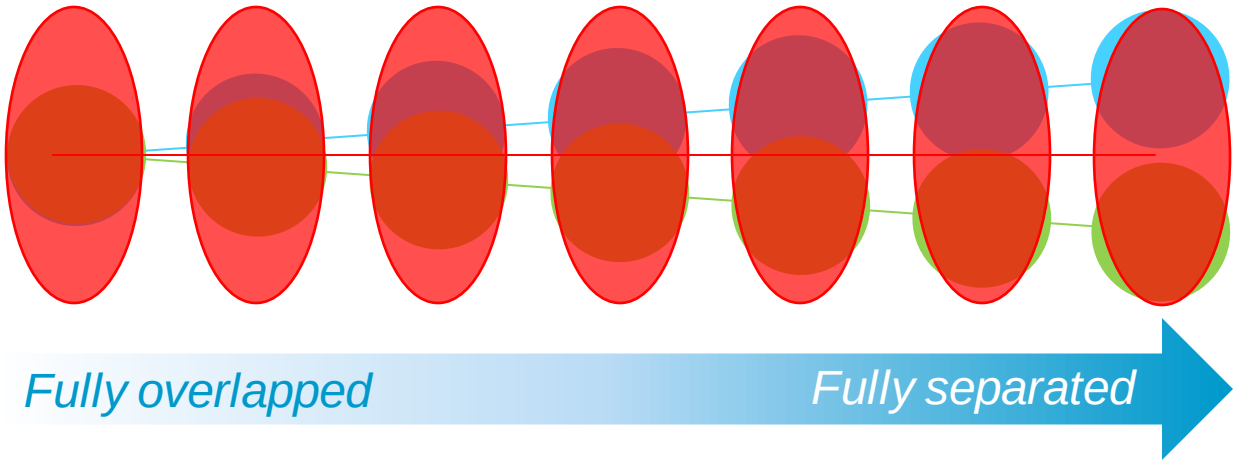
Twinning

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

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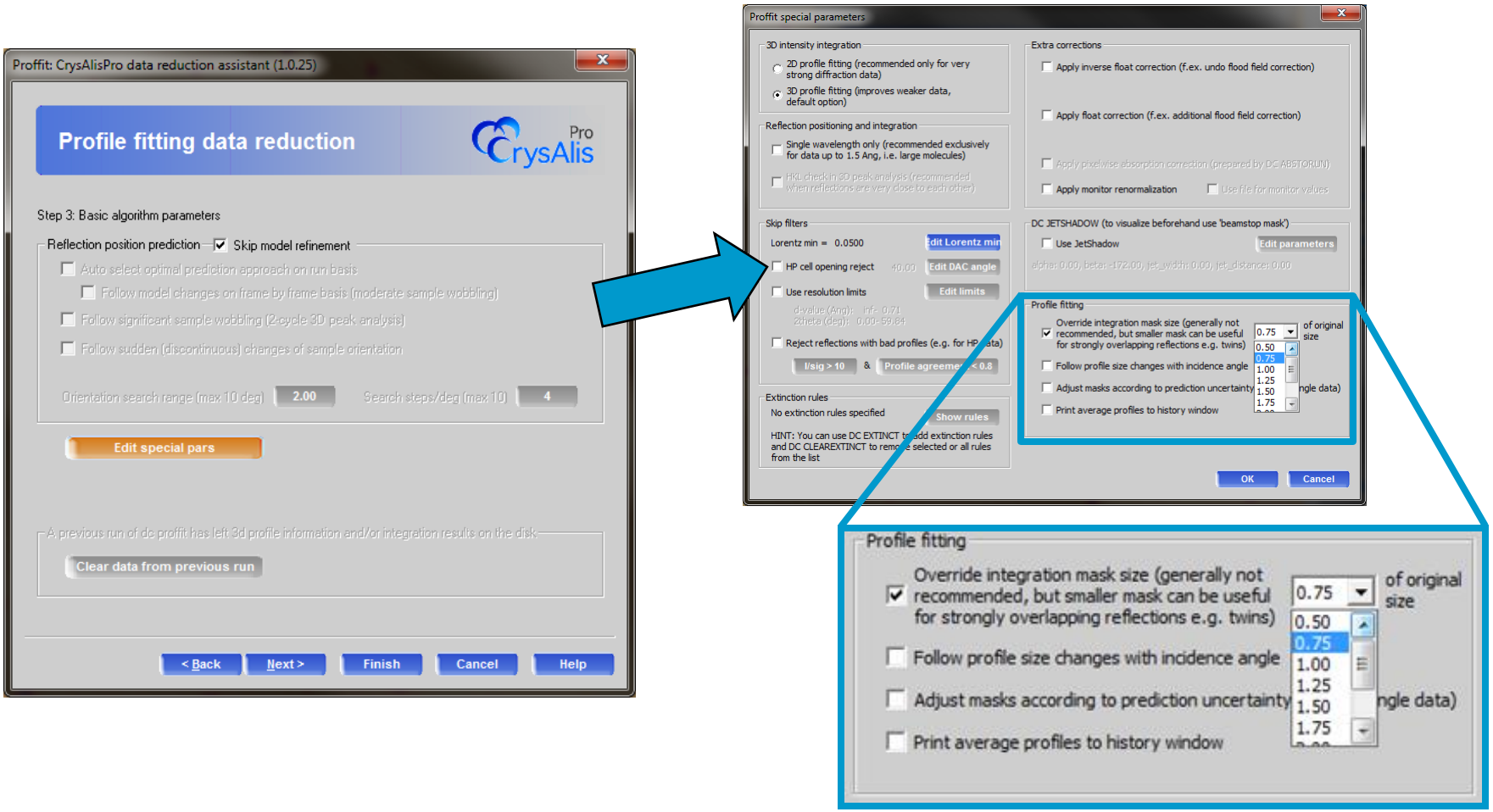
Twinning

Integration treatments – profile size

Altering profile mask sizes in CrysAlis^{Pro}



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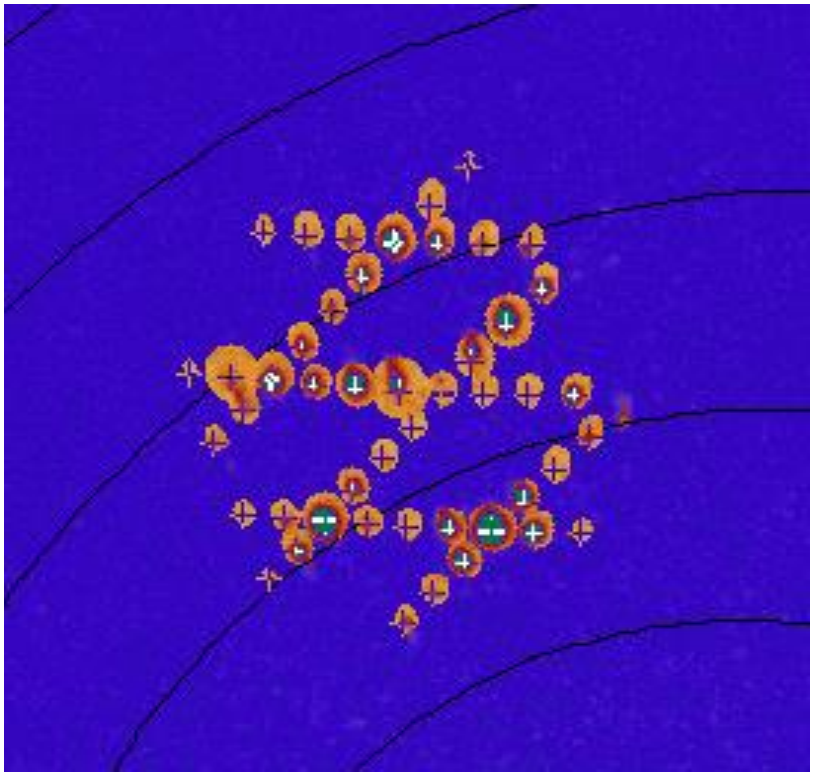
Twinning

Integration treatments – smart background

For datasets with uneven backgrounds, smart background can help.

From v37 upwards, smart background is now twin enabled



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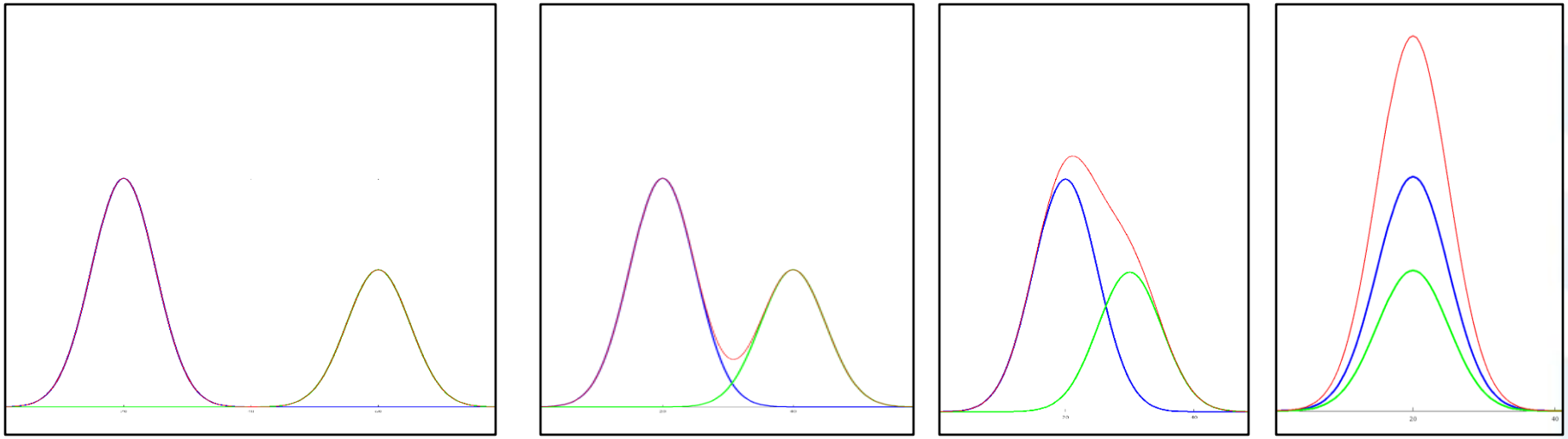
Twinning

Post Integration treatments – overlap threshold

Increasingly difficult deconvolution



0% overlap

100% Overlap

Detwinning attempted for structure solution

Changeable threshold

Detwinning not attempted default: 80%

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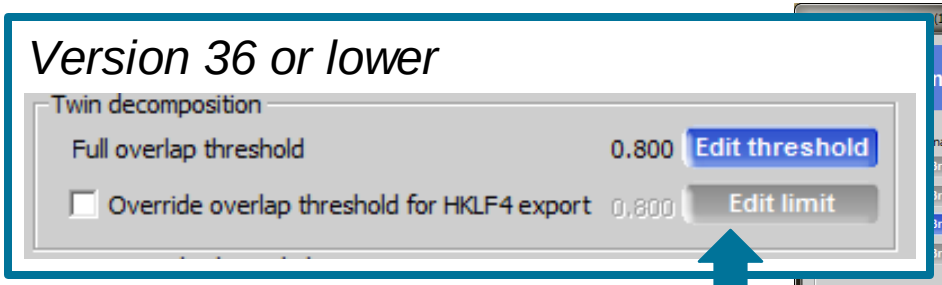
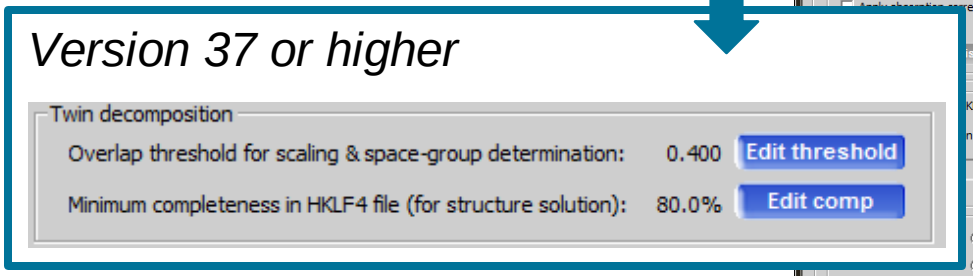
Twinning

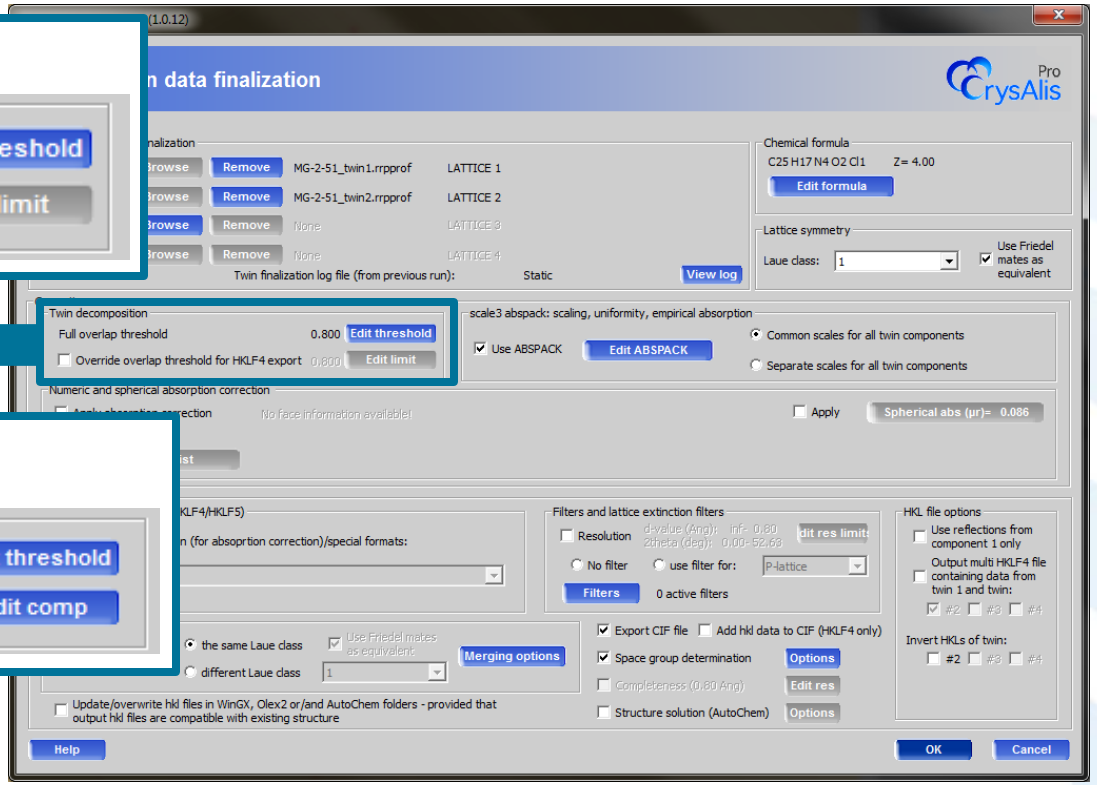
Post Integration treatments – overlap threshold

Version 36 or lower



Version 37 or higher





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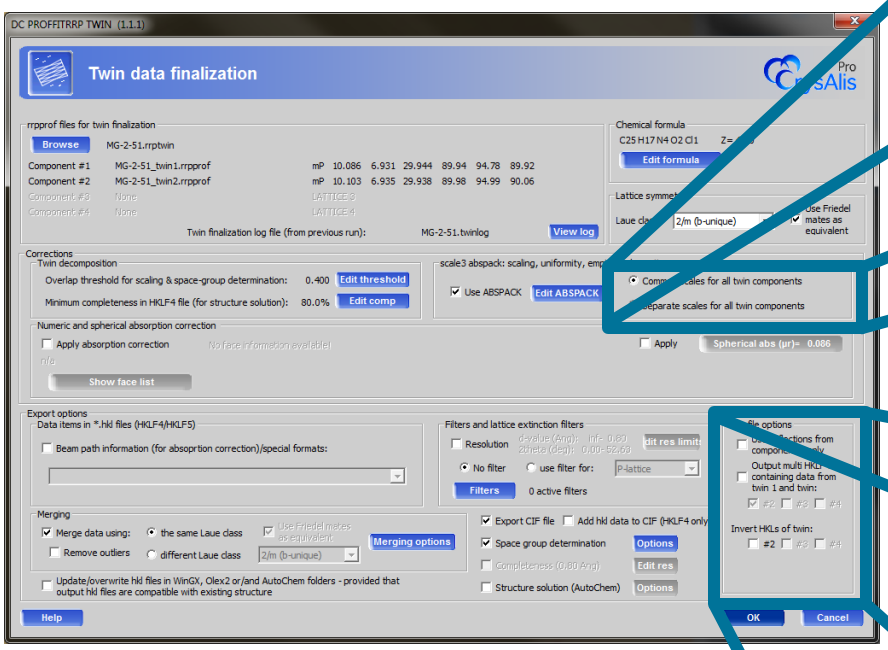
Twinning

Additional options



Useful for cases where secondary domain(s) are weak

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

Special case… Needs some explanation.

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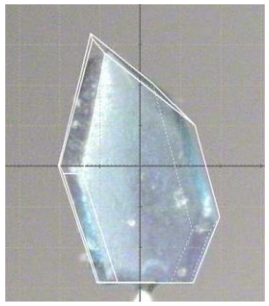
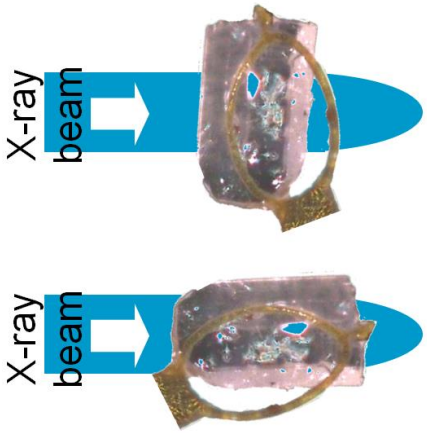
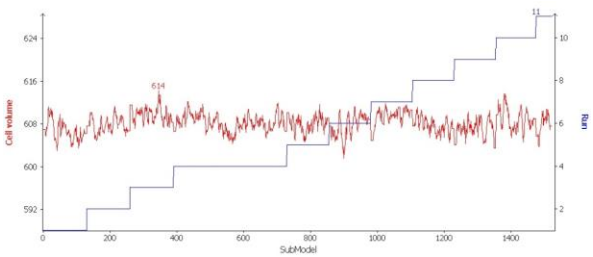
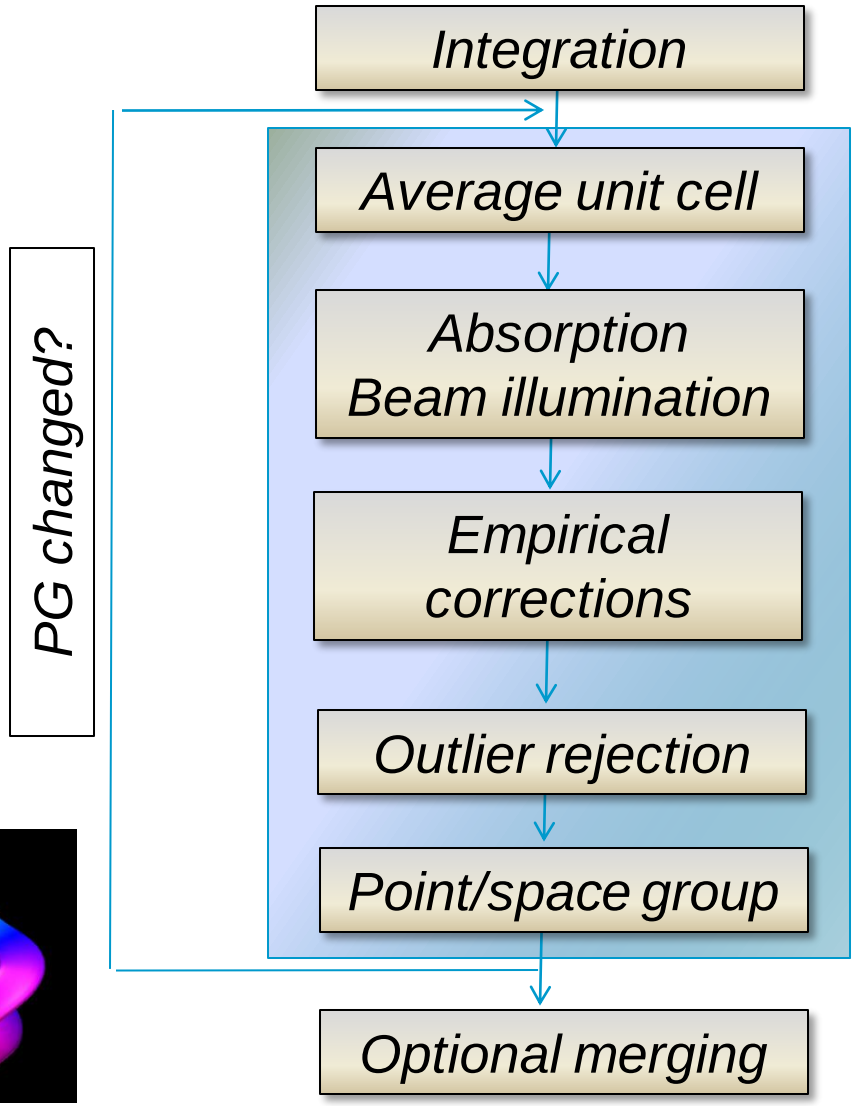
Good data quality through full post corrections









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Thank you for listening!

Find out more at

www.rigaku.com

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