Reciprocal Workshop: Rigaku Oxford Diffraction

Handling twins

Some notes on corrections

Rigaku oxford diffraction

Takashi SATOw

Overview

- Twin/Multi-crystal
- New twin processing for better accuracy
- Step-by-step processing on easy twin and lattice faker
- Twin example on Crystalline Sponge

  (Vacant [cyclohexane x 5] but twined, guideline for the worst condition before soaking)

  Rint < 3.0%, R1 < 7.0% @ redundancy > 2.5
- Time permitting: Some notes on corrections: Empirical and numeric corrections

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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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Question:
Twin vs. multi-crystal – what is what?

*non-merohedric

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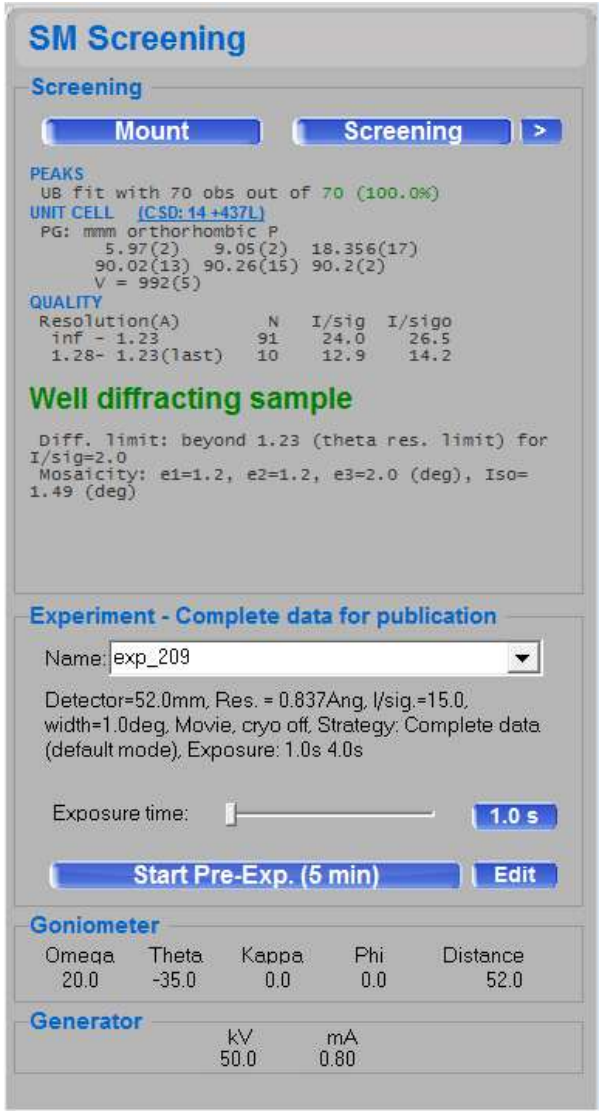
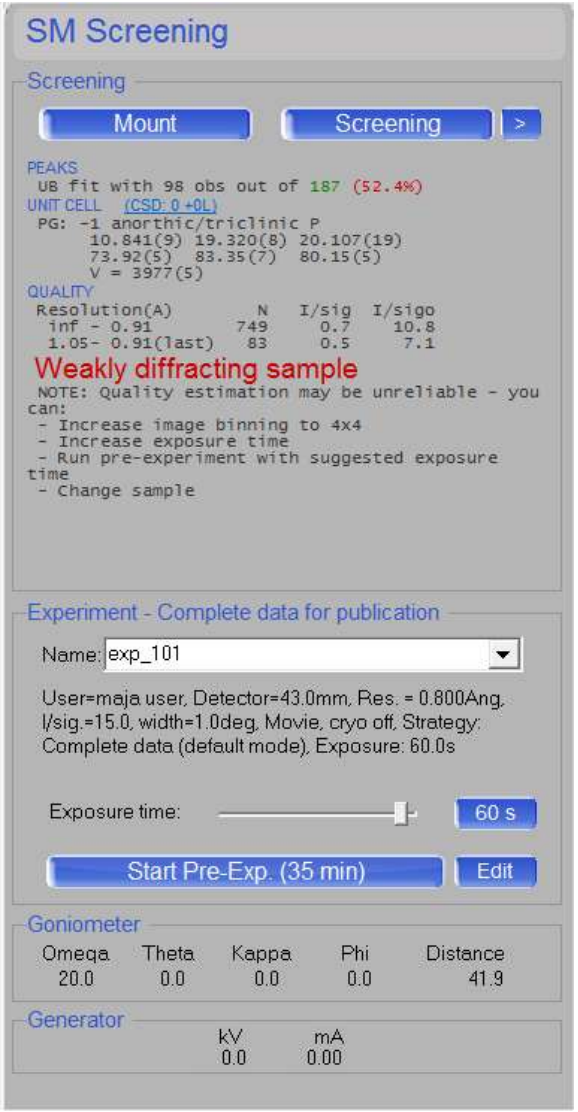
Screening tool for quickly judging sample quality





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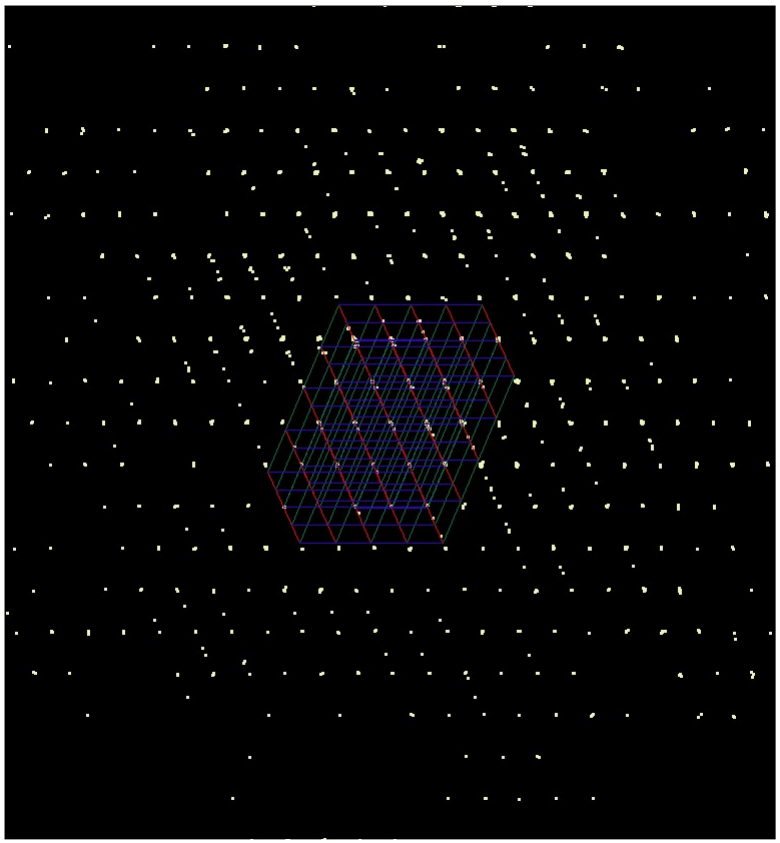
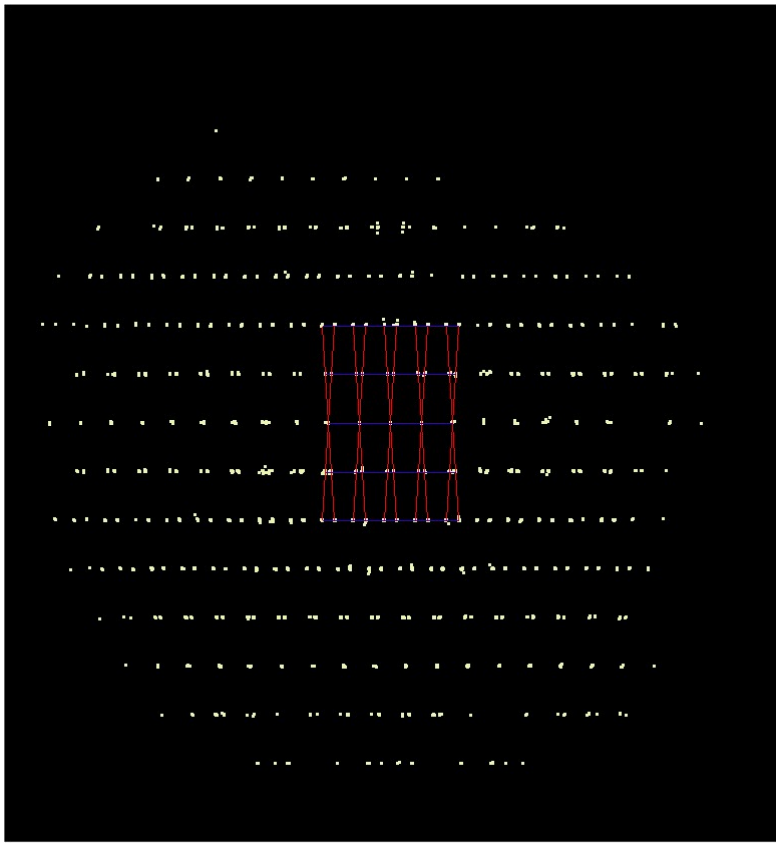
Twin types



- Easy twin



- Super lattice “faker”

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Data sets for these examples

- Pol411, pol426
- CCD data; kindly provided by Prof. Maria Gdaniec (Poznan, Poland)
- You can try to apply the concepts presented here to these data sets.

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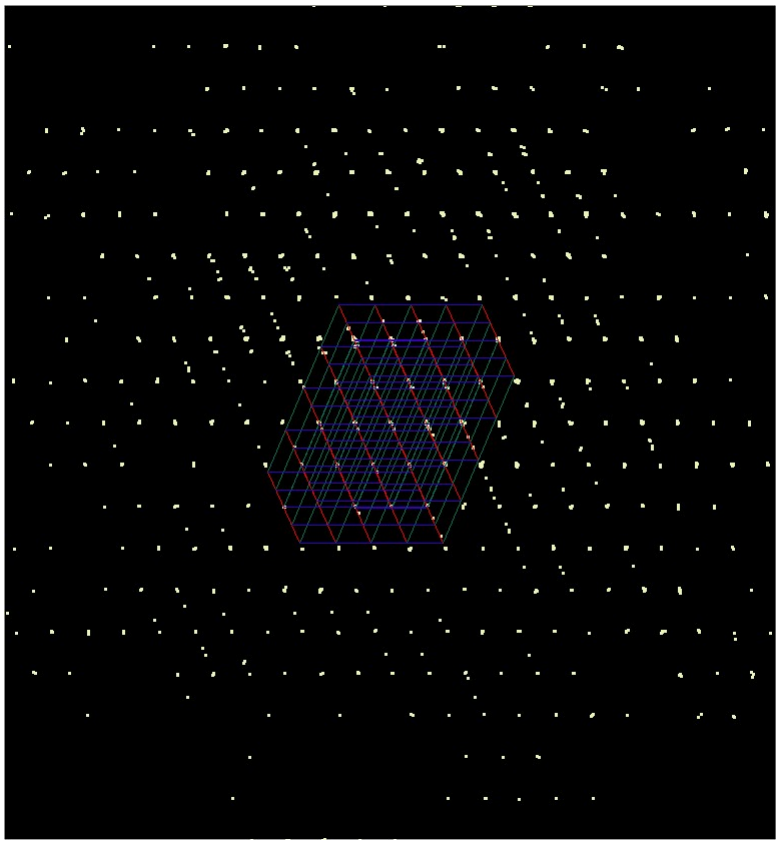
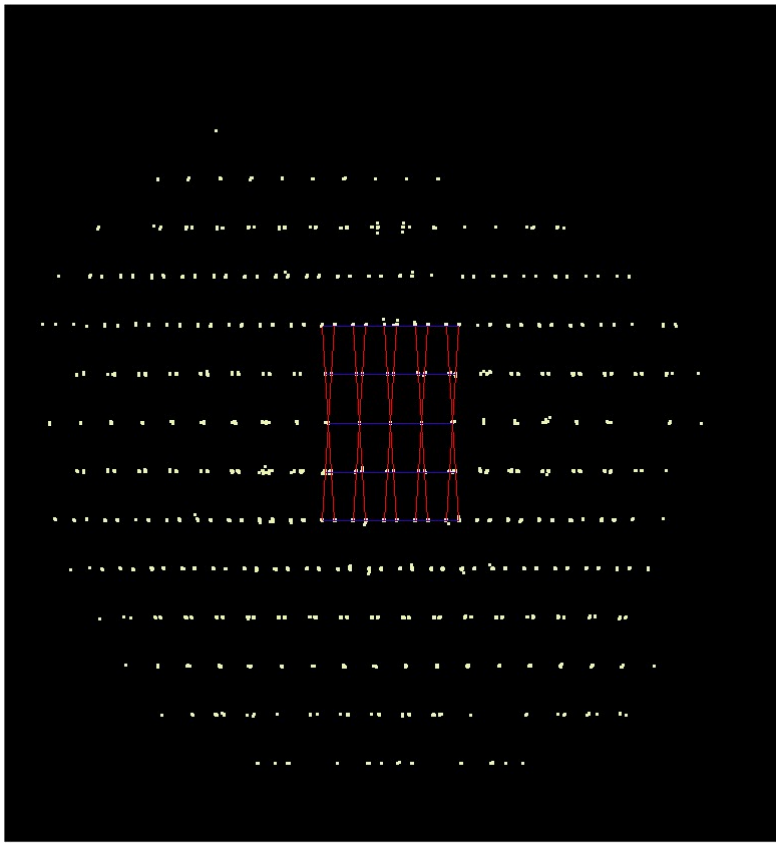
Twin types



- Easy twin (pol426)



- Super lattice “faker” (pol411)

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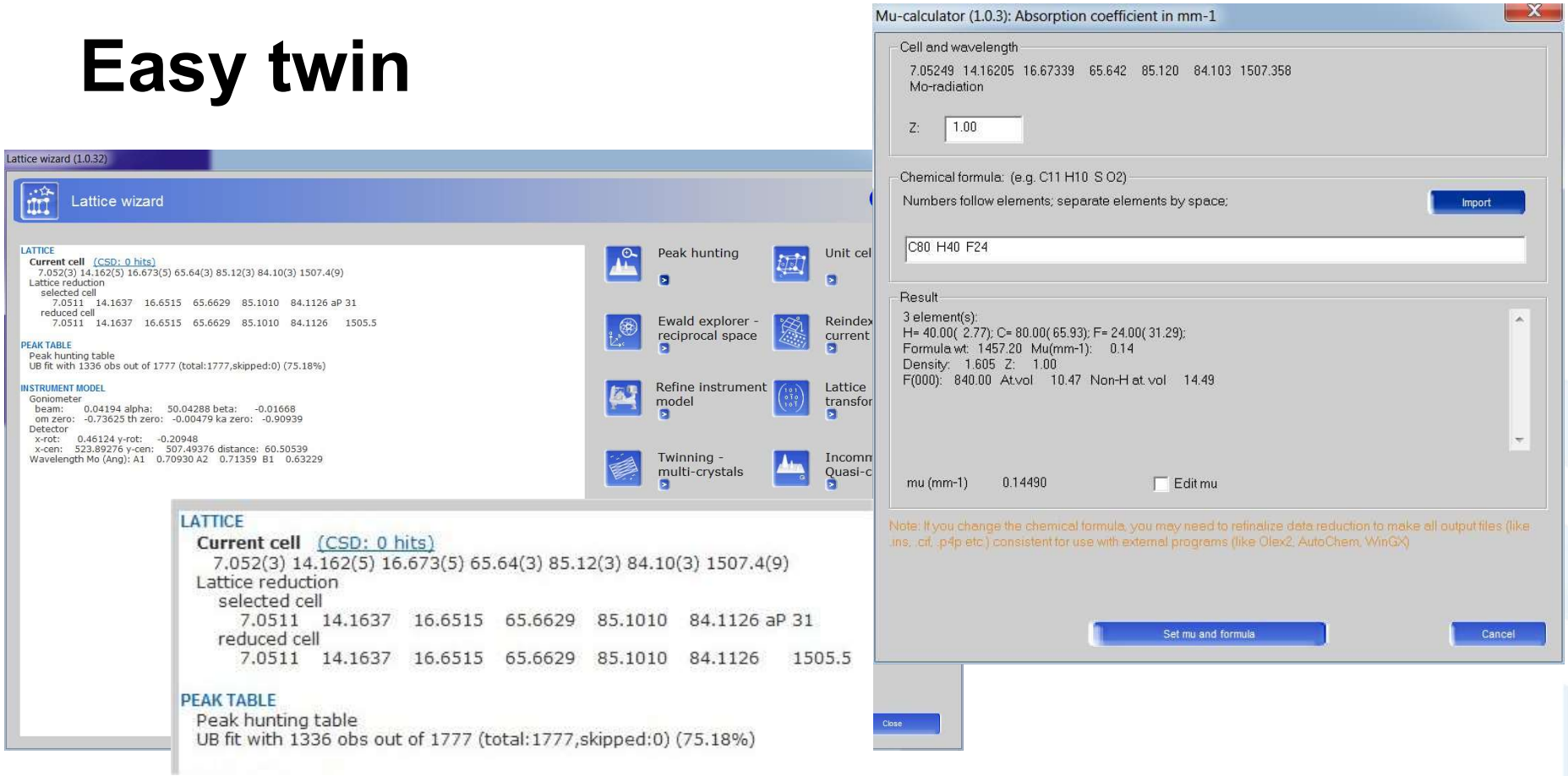
Easy twin



We do peak hunting and auto-indexing gives:

- Indexation <90%
- Chemical content consistent

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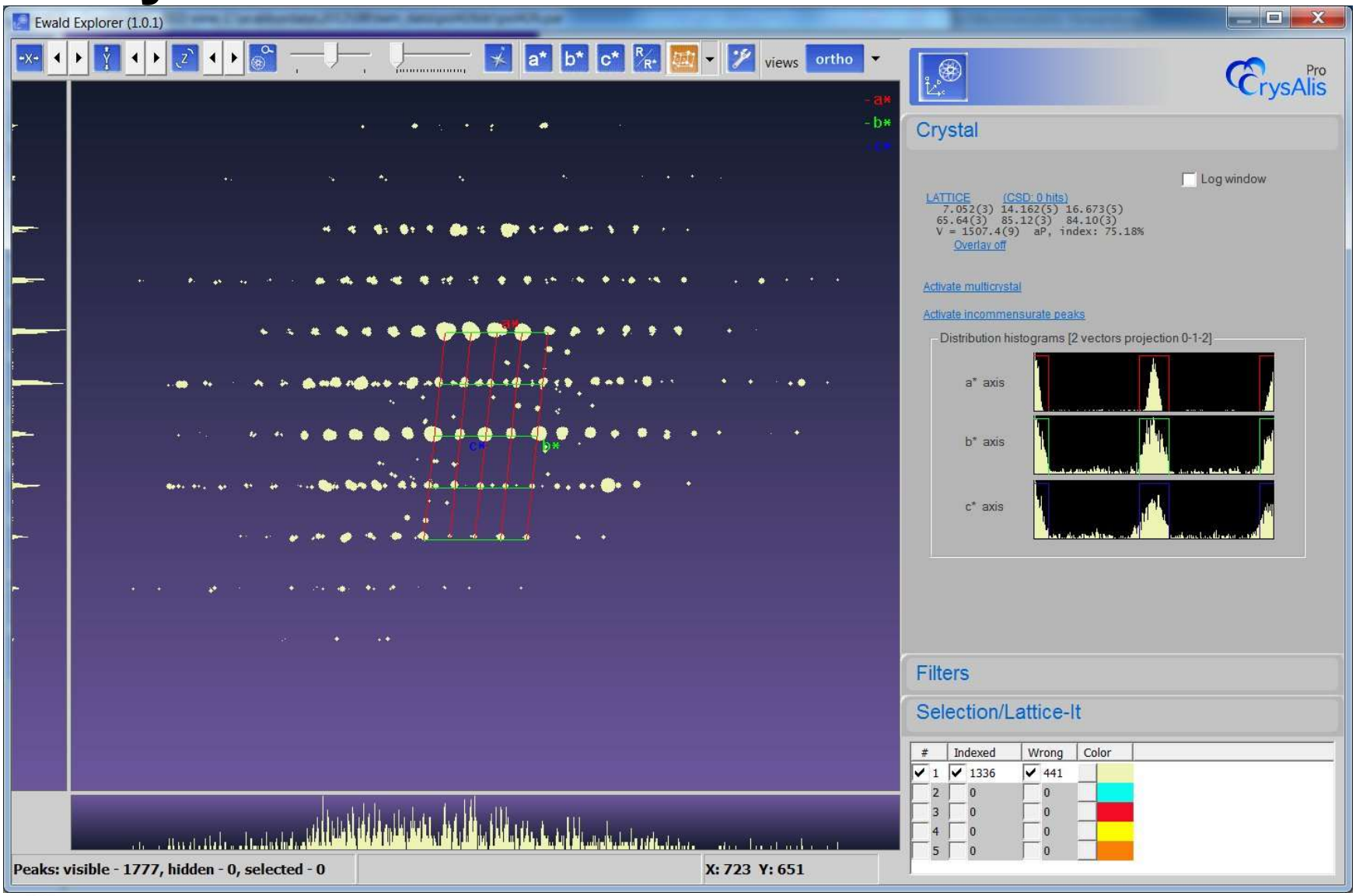
Easy twin in Ewald^{Pro}



Rigaku oxford diffraction

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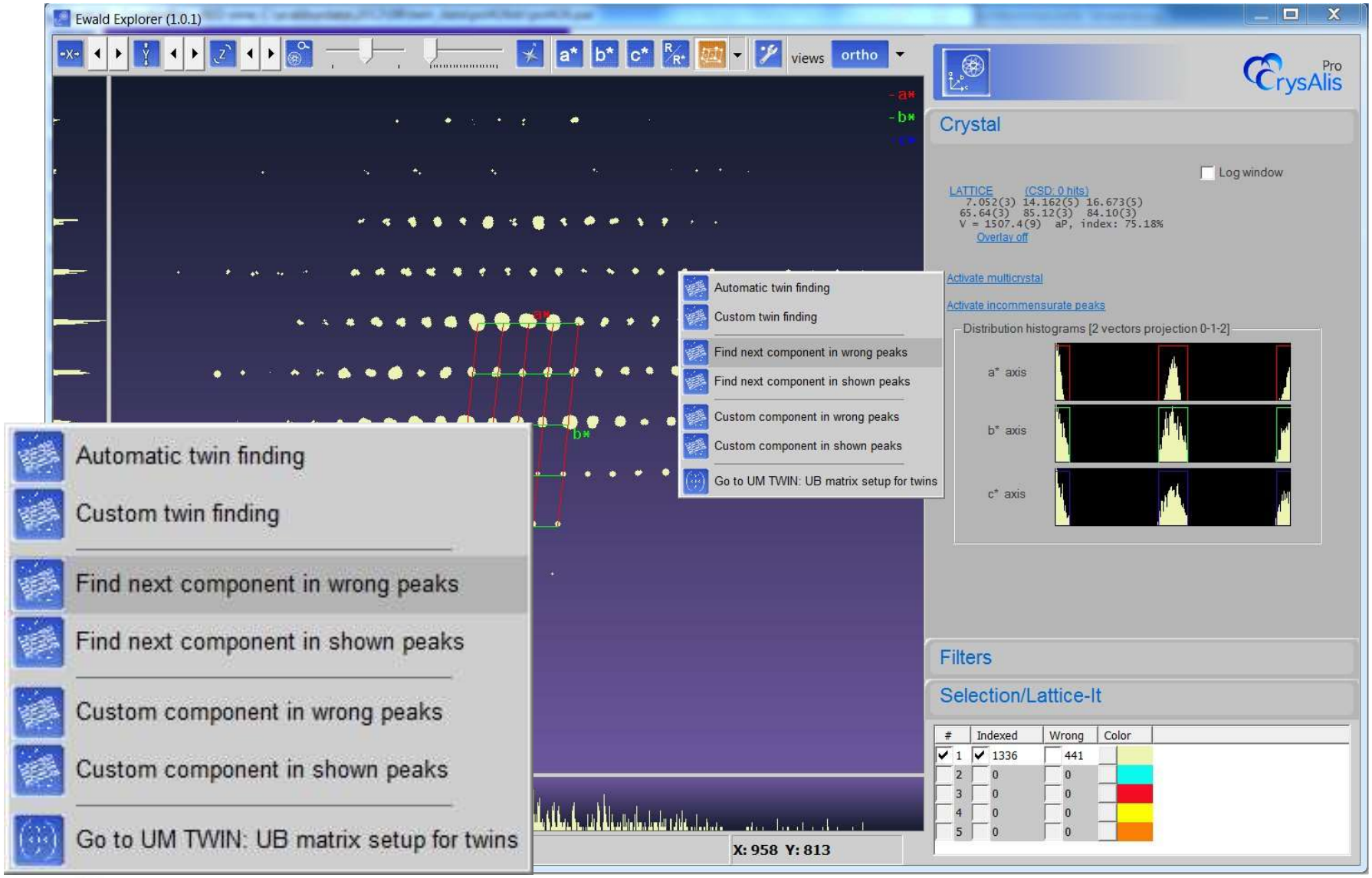

Rigaku oxford diffraction

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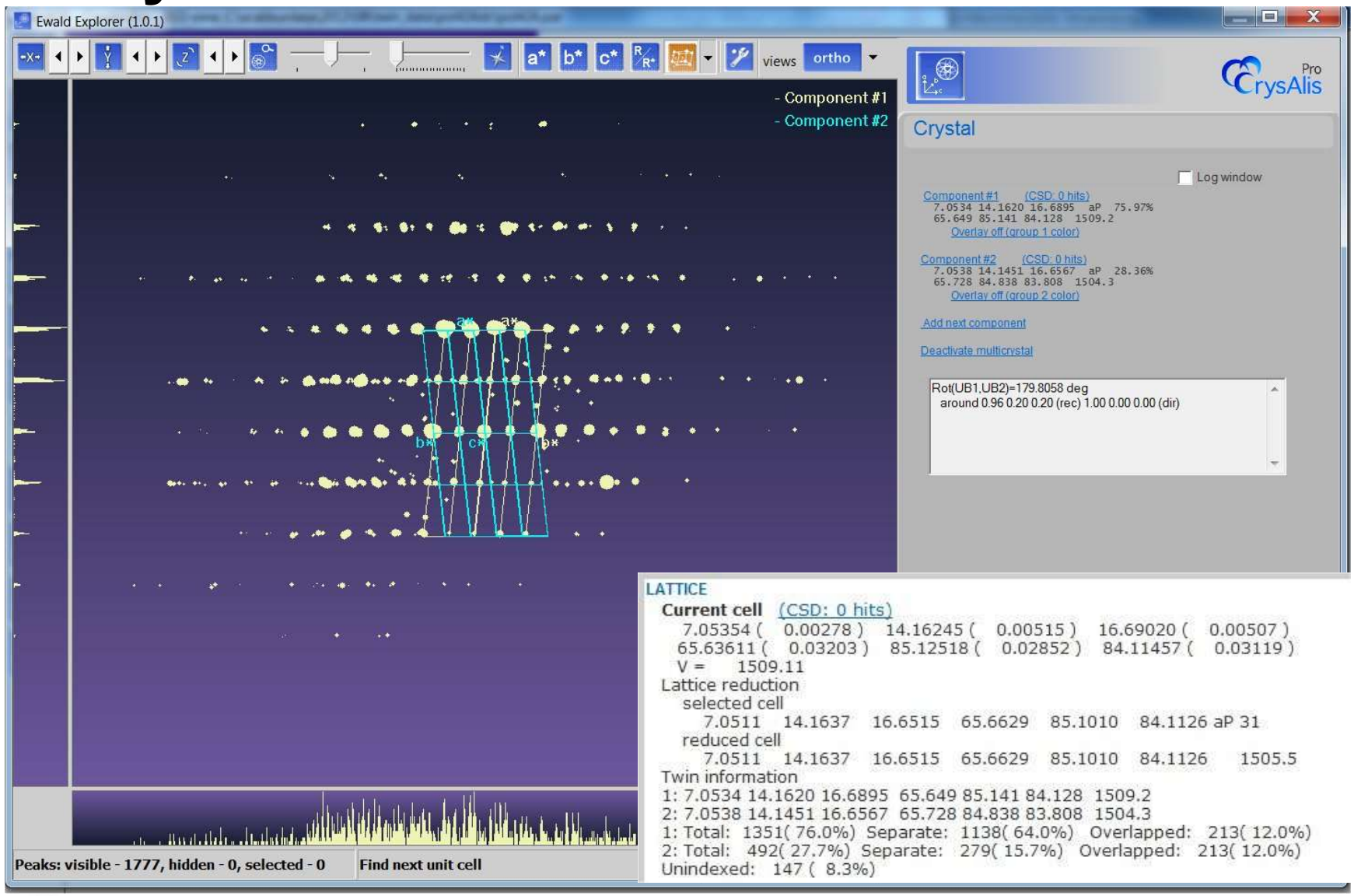

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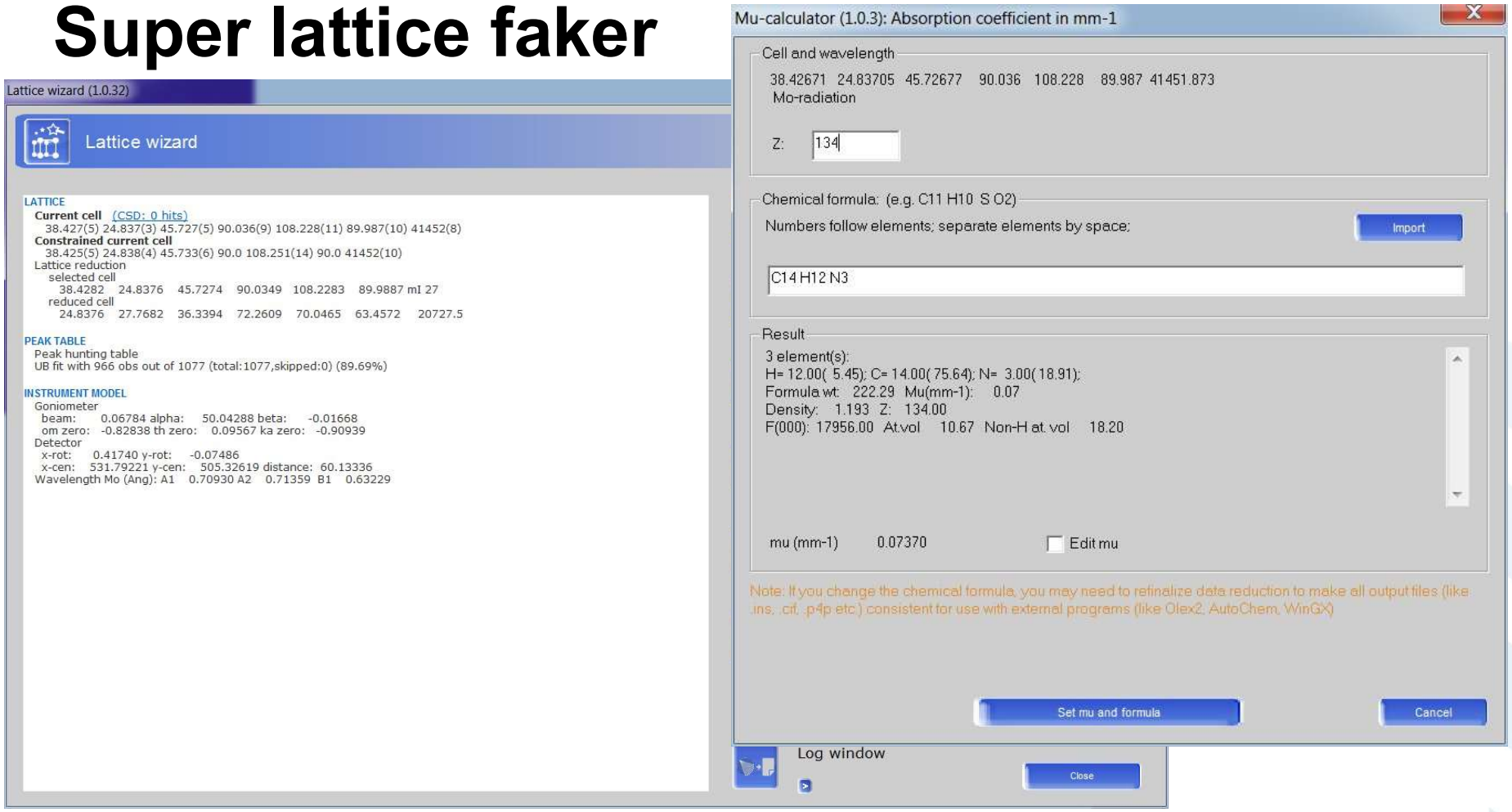
Super lattice faker



- Indexation good/high
- Chemical content inconsistent (here Z=134)

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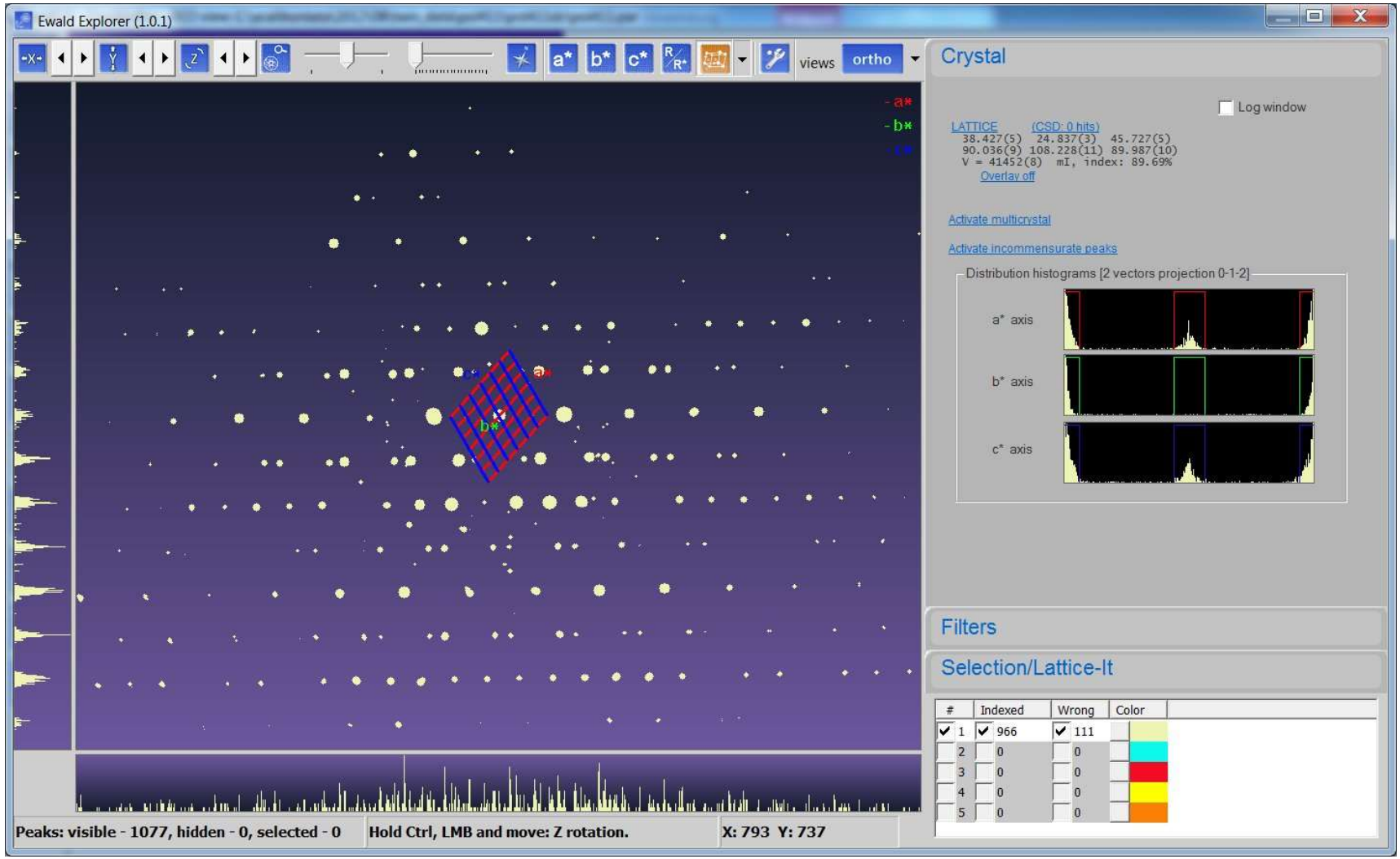
Super lattice faker in Ewald^{Pro}



Rigaku oxford diffraction

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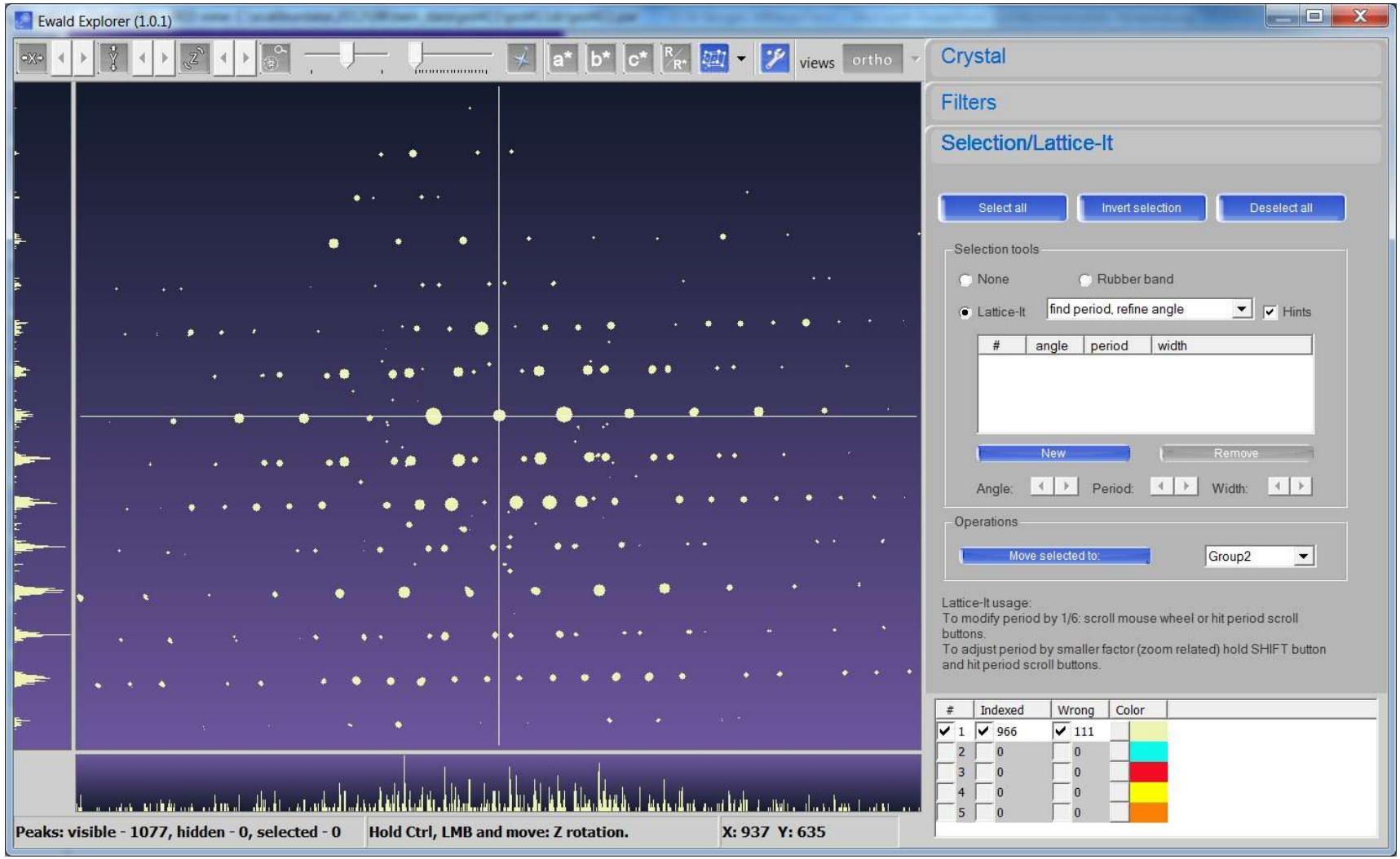

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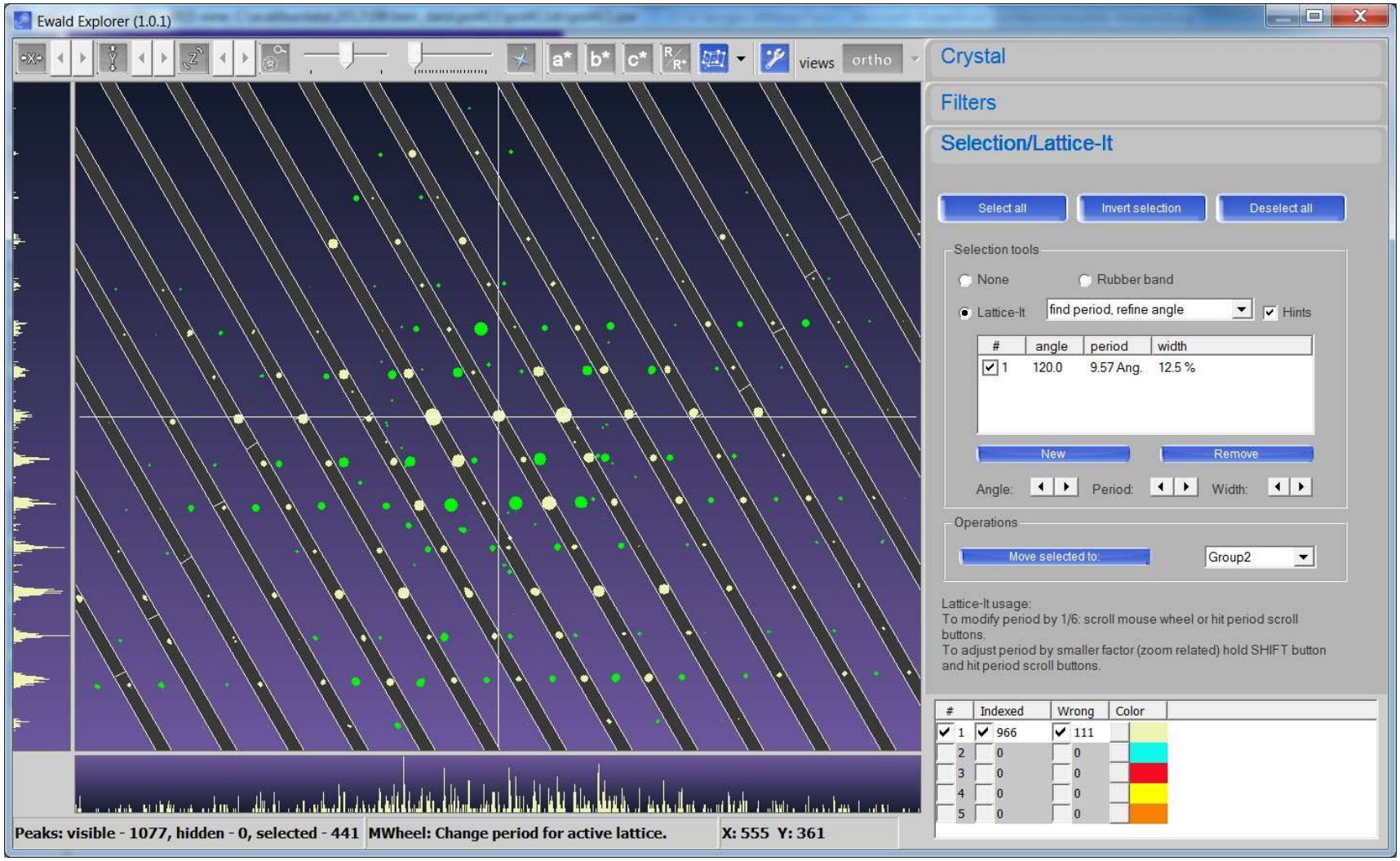
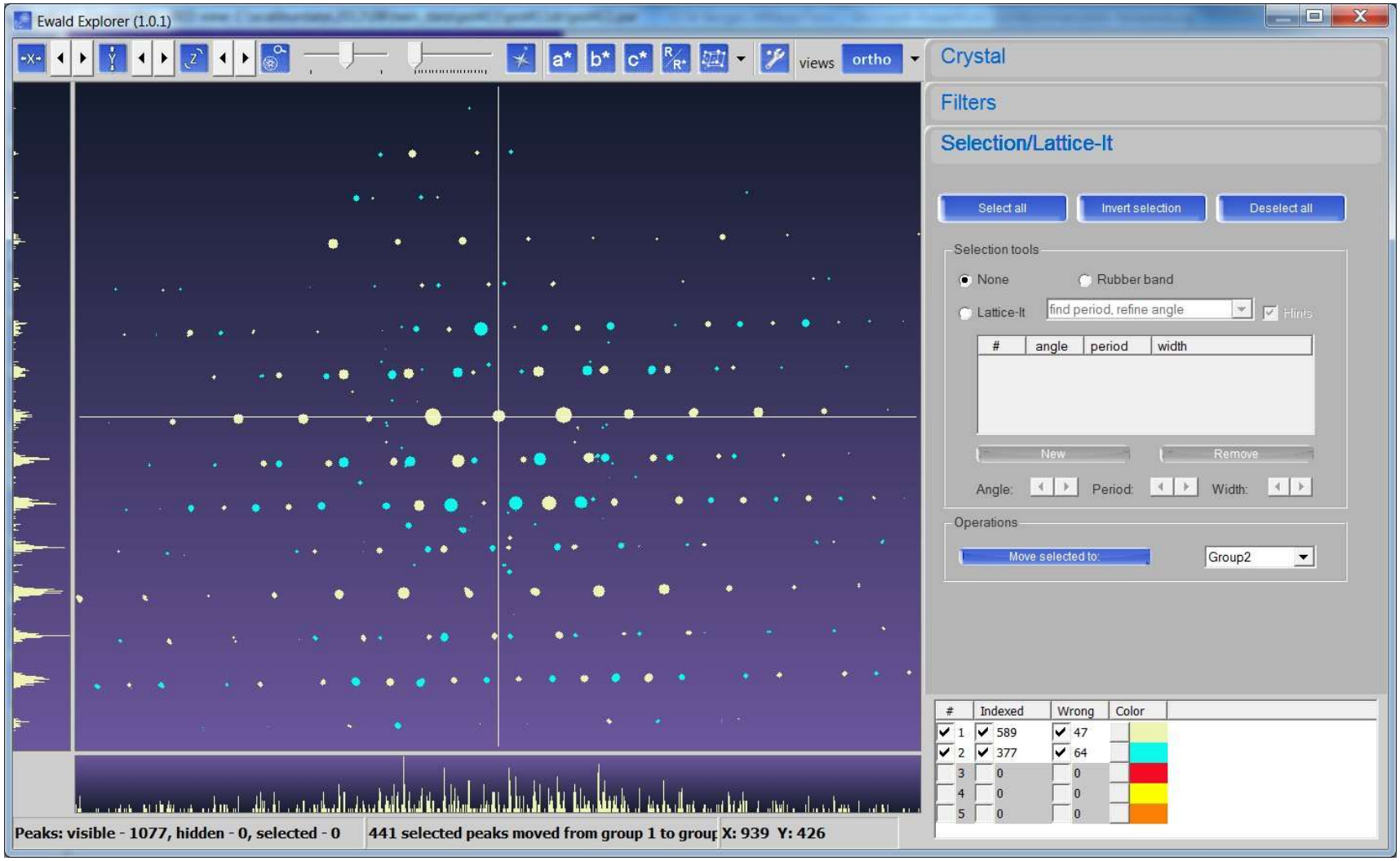
Super lattice faker in Ewald^{Pro}



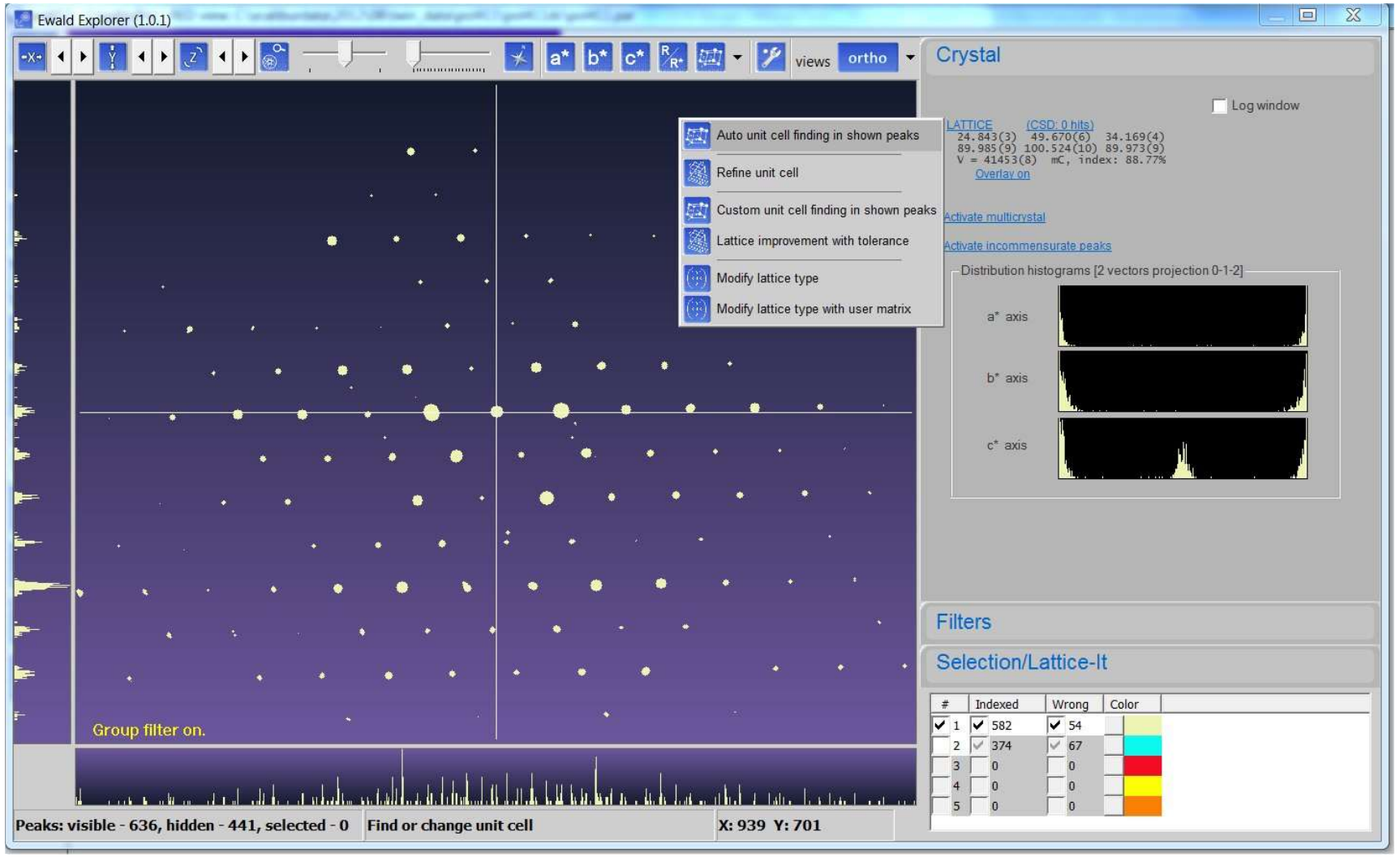
Rigaku oxford diffraction

Super lattice faker in Ewald^{Pro}



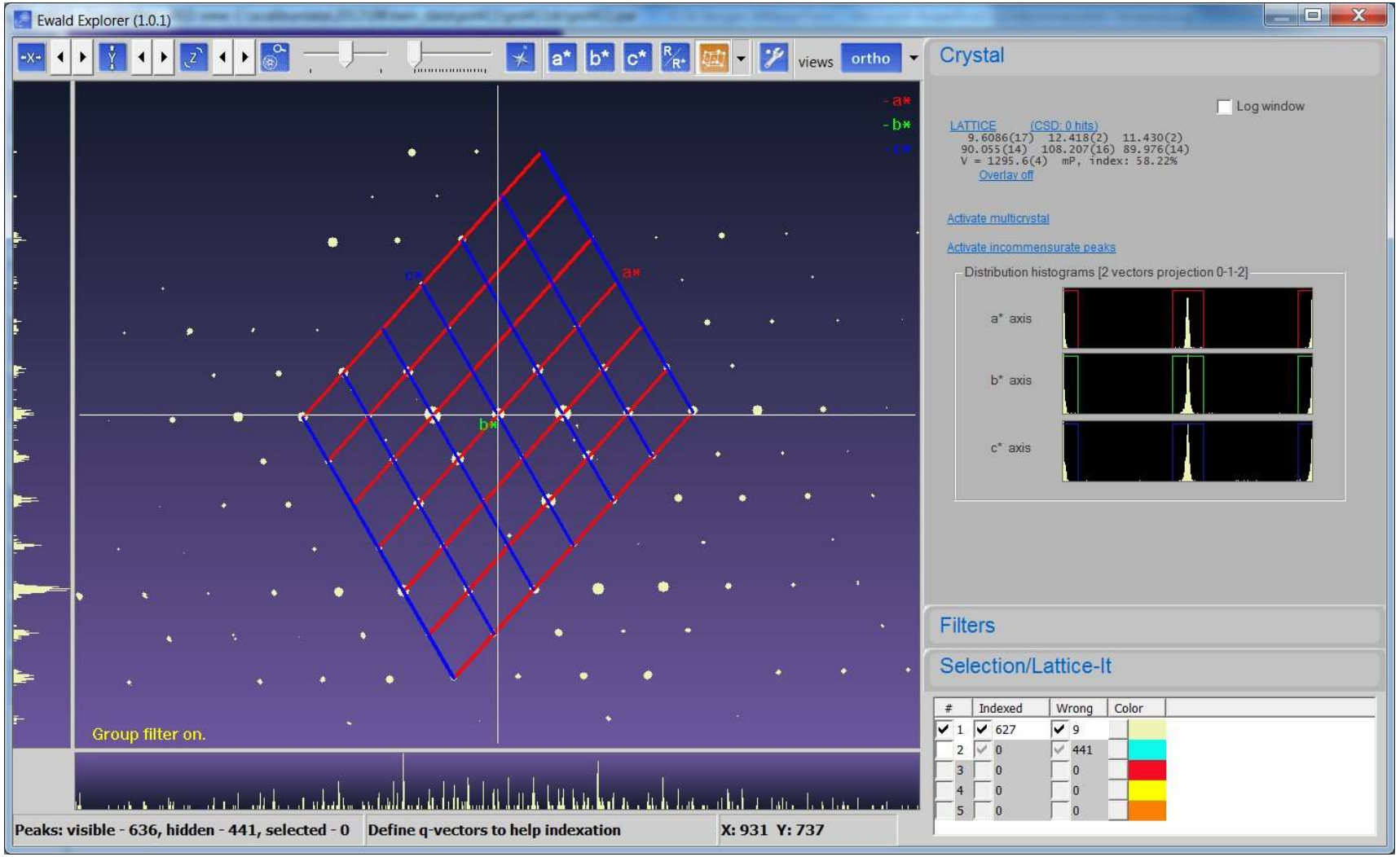
Rigaku oxford diffraction

Super lattice faker in Ewald^{Pro}



Rigaku oxford diffraction

Super lattice faker in Ewald^{Pro}



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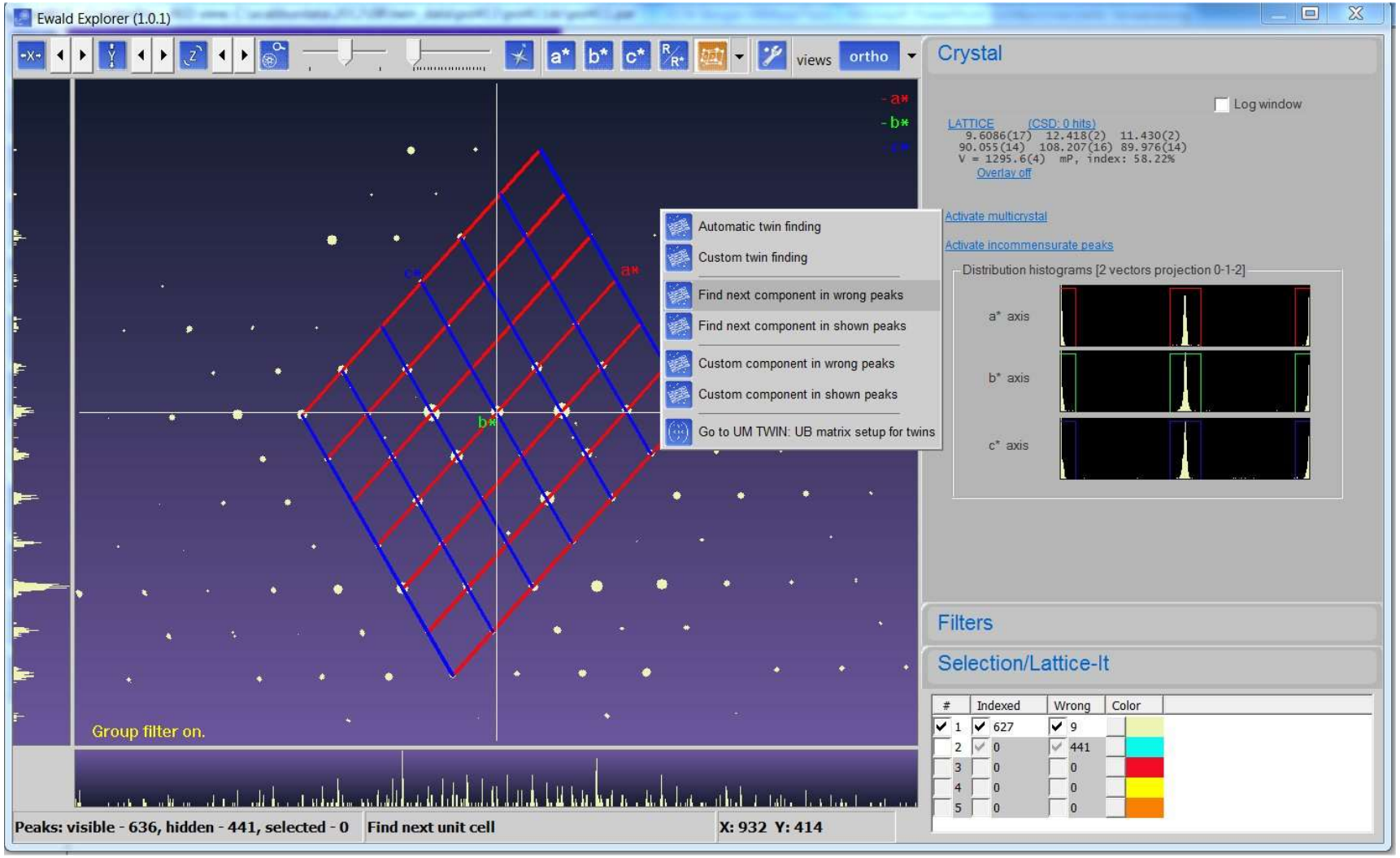
Super lattice faker in Ewald^{Pro}



Rigaku oxford diffraction

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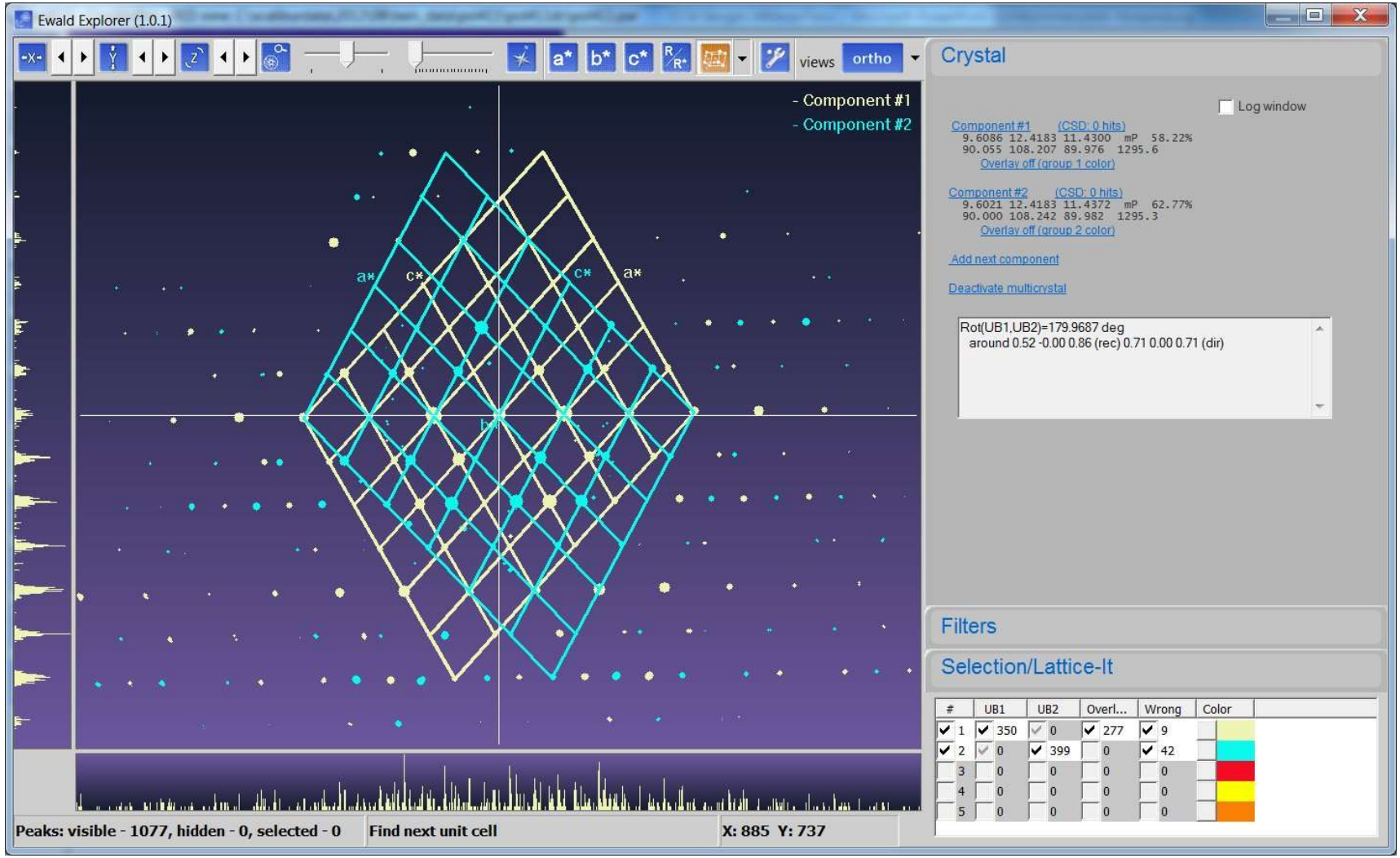

Rigaku oxford diffraction

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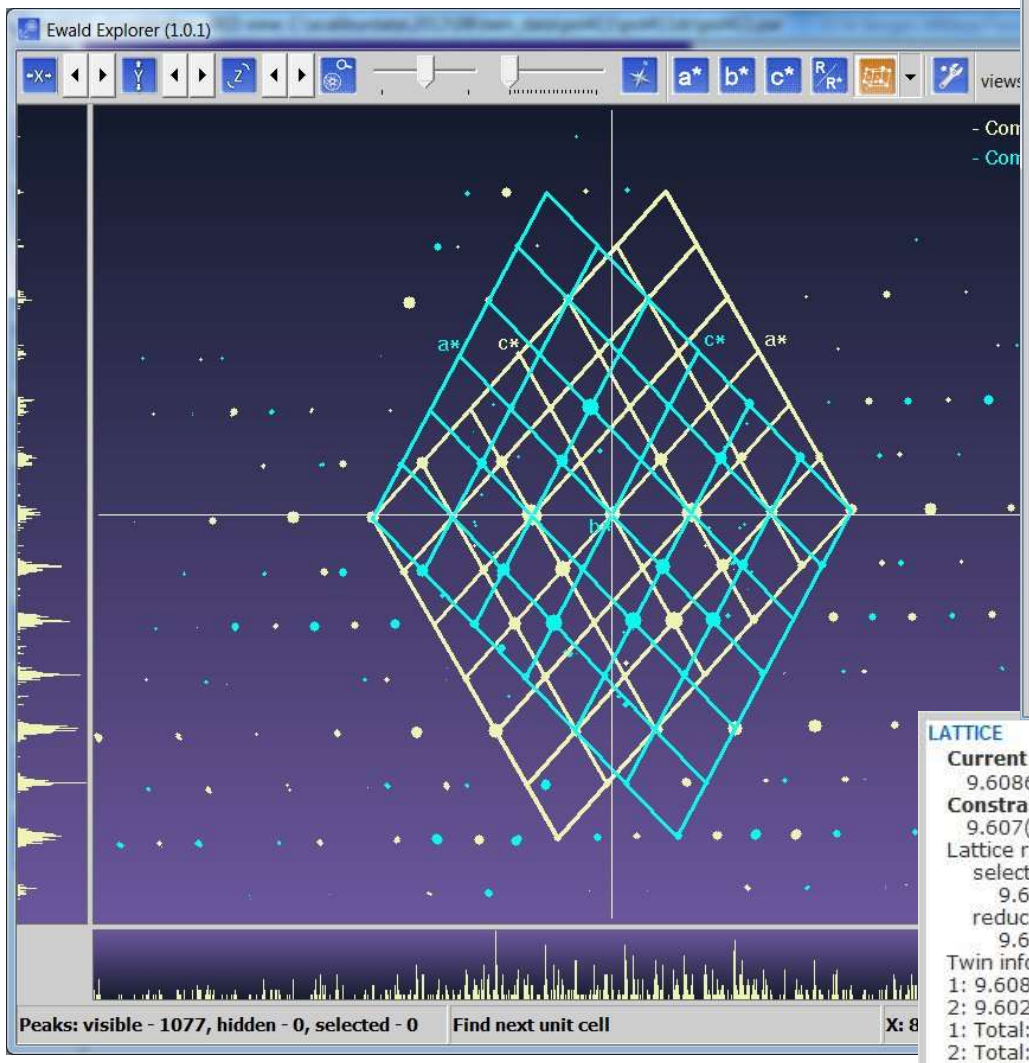

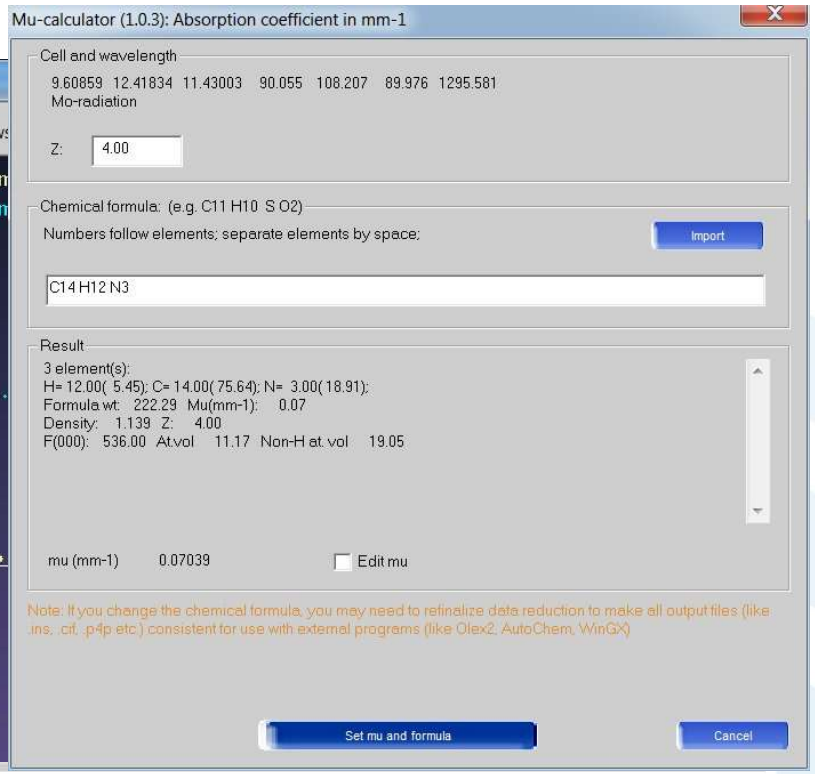

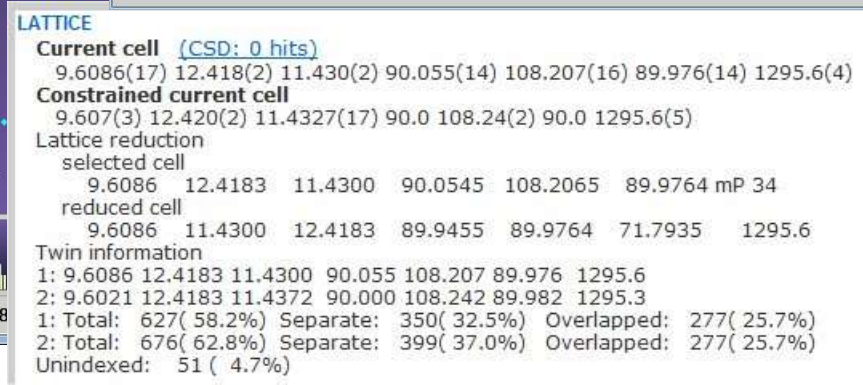

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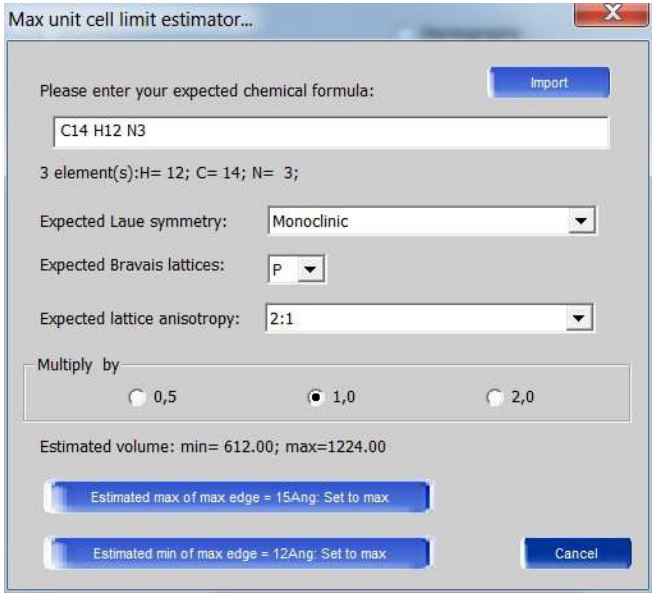
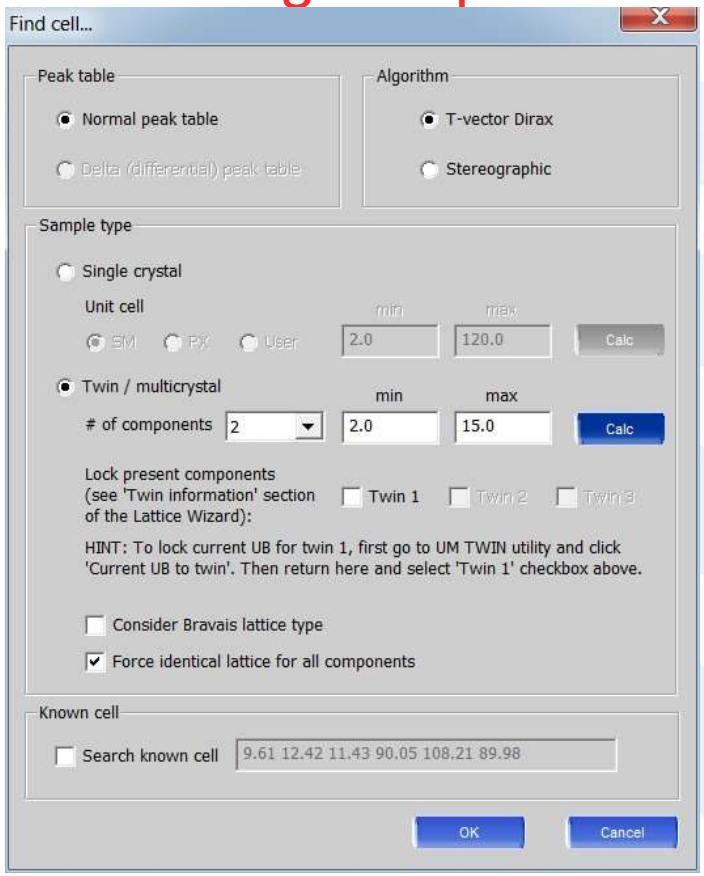
Other tools in Ewald^{Pro} to support twin handling

- Reflection grouping (up to 20 groups)
- Filters (intensity, d-value, runs [mistake a moving sample as twin])
- Custom unit cell finding





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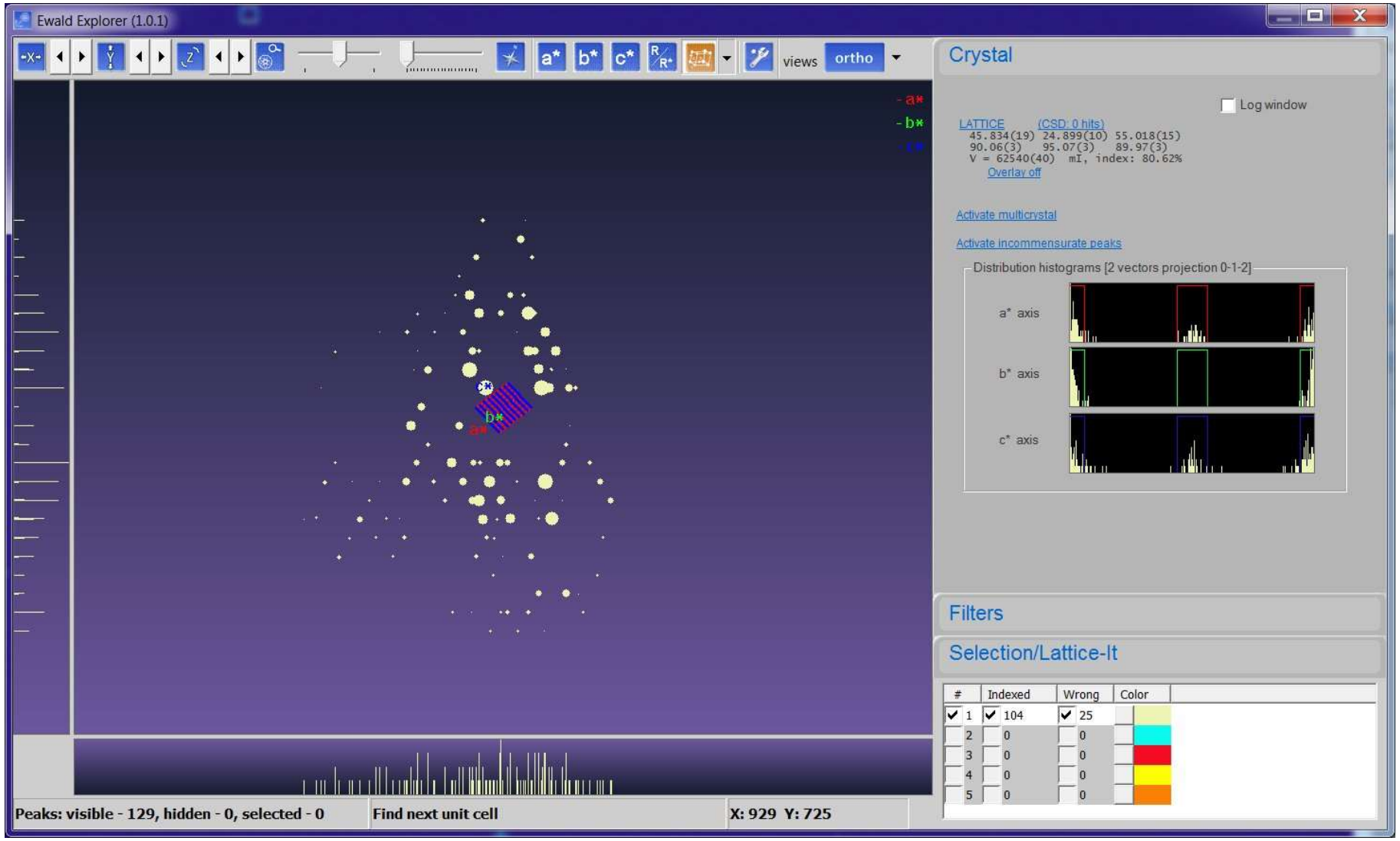
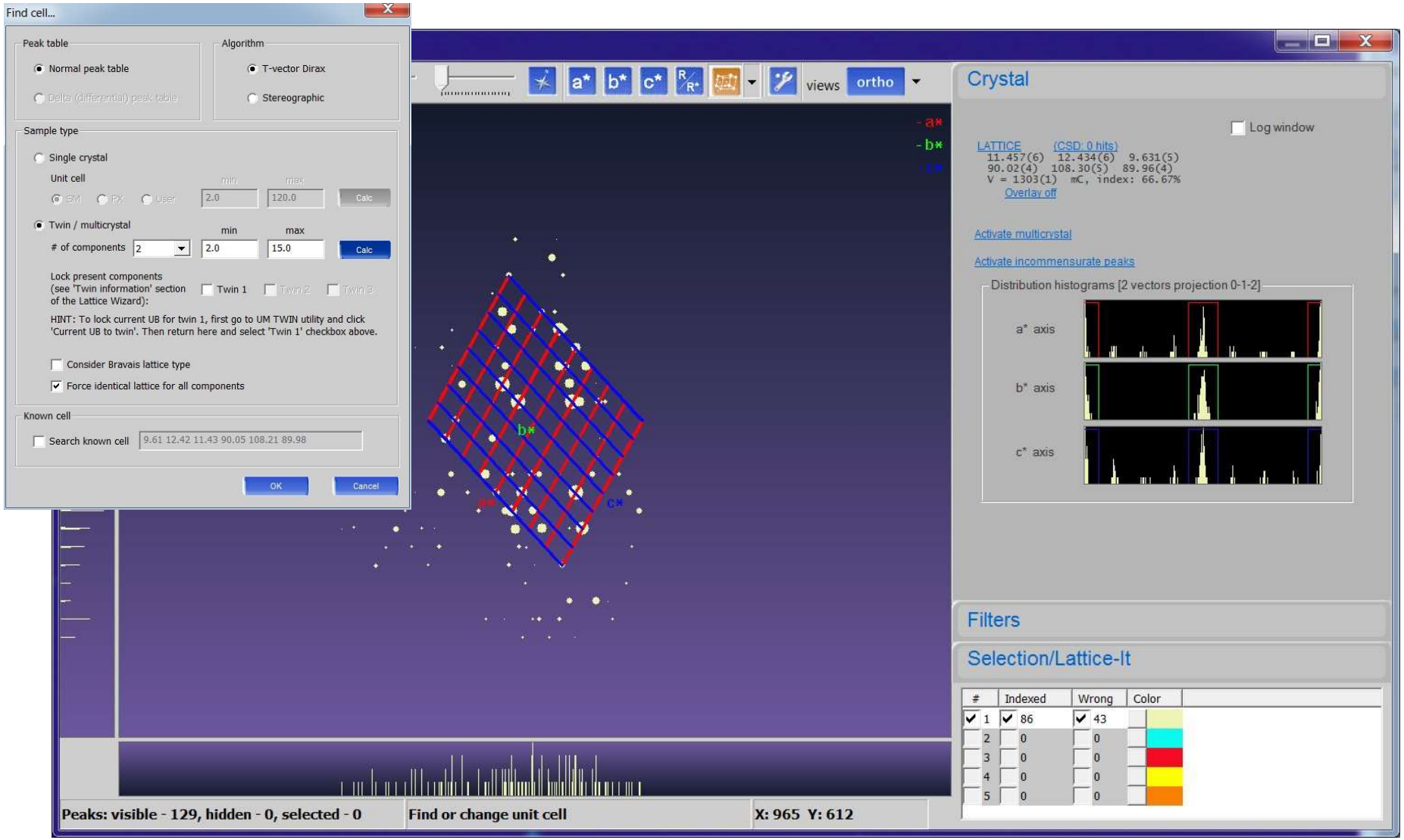
Judging a twin from few reflections can be tricky…



Rigaku oxford diffraction

Use the custom unit cell tool…



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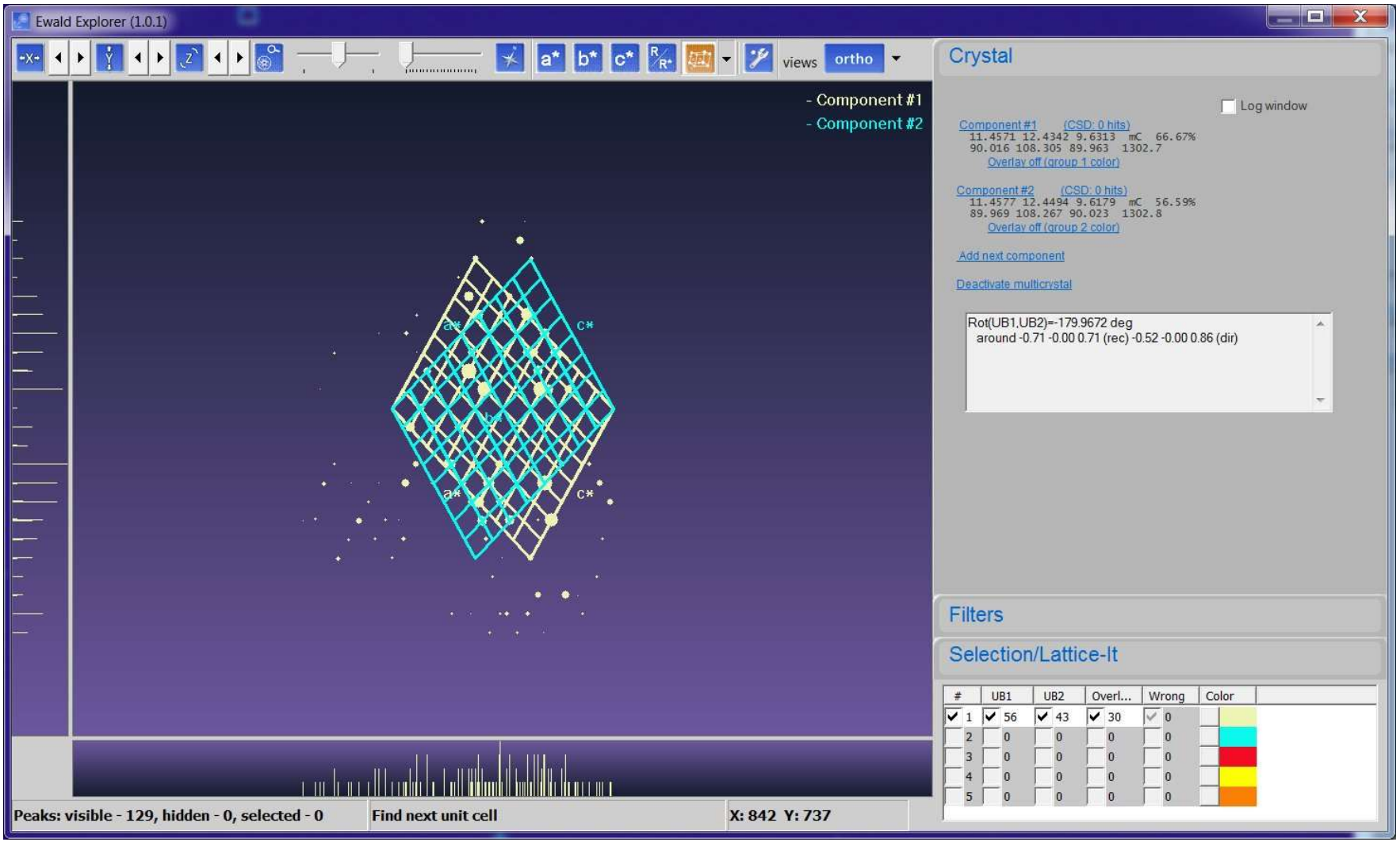
Find the twin from the wrong peaks…



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Strategy with known twin law

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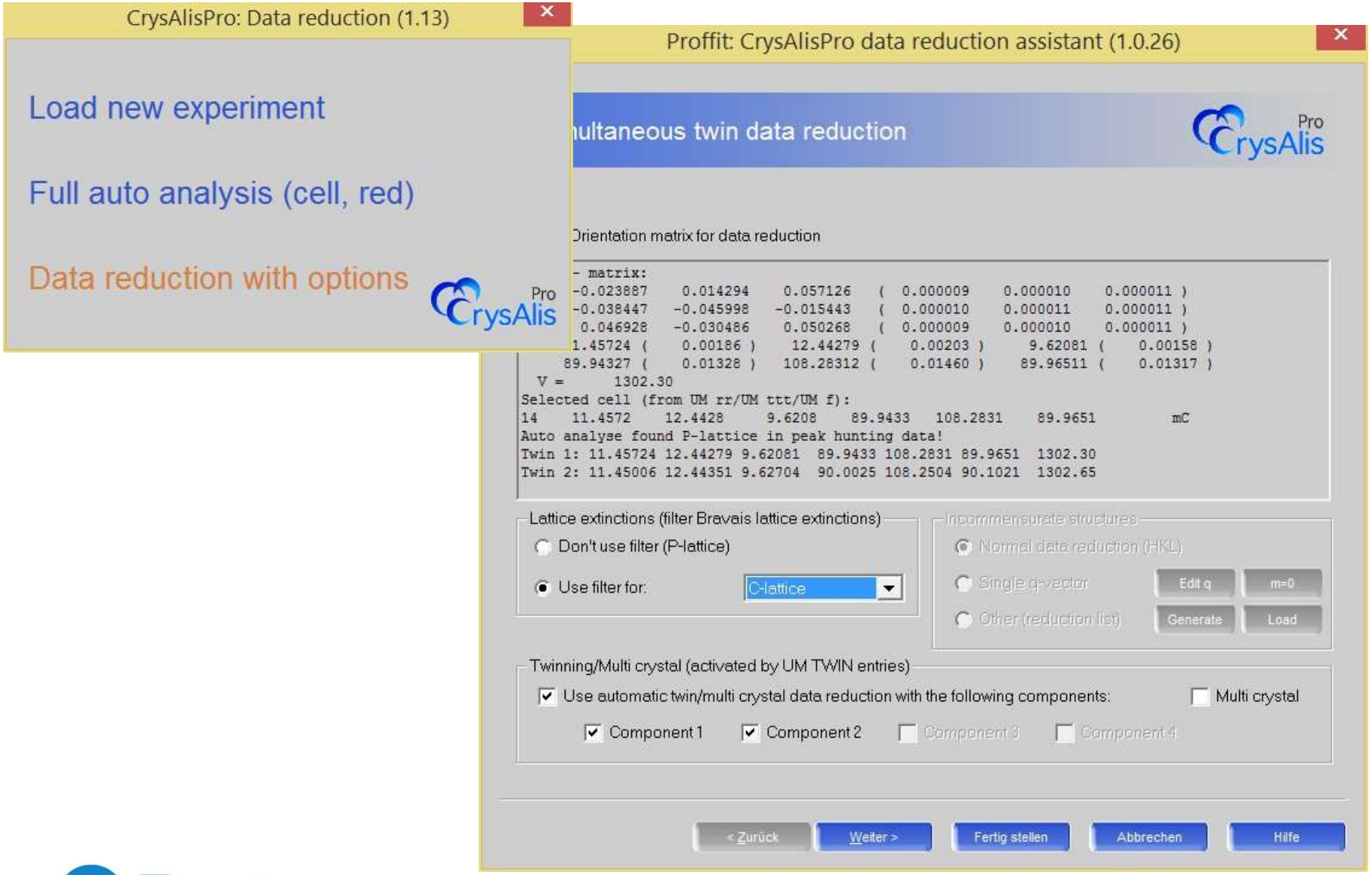
Data reduction



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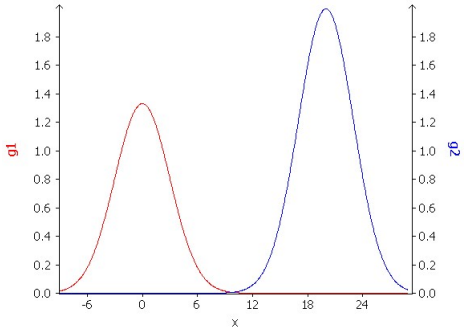
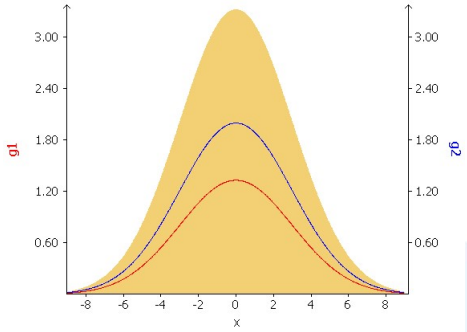
Twin profile data



Fully separated



80% - 100%
Overlap data

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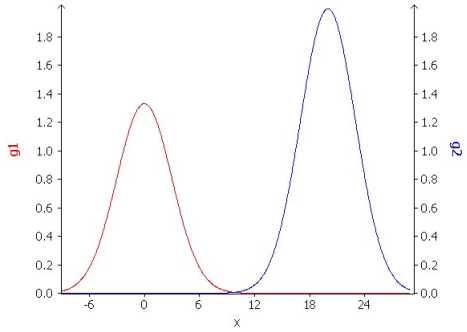
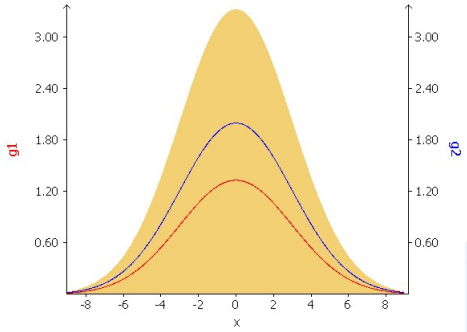
Twin profile data



Fully separated



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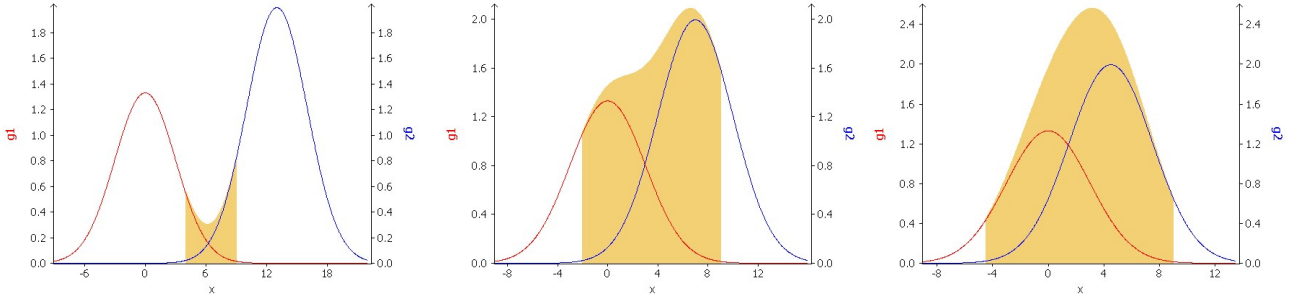


0%

Deconvolution possible

80%

Pre-37 vs. new 37 approach

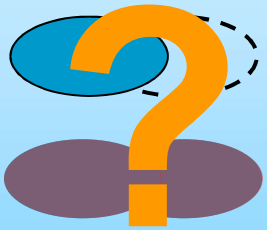
Pre-37 twin integration

Component #1
PROFFIT

Component #2
PROFFIT

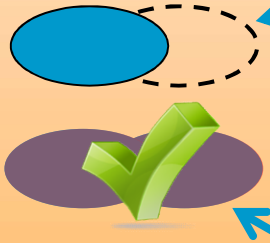
Less accurate HKLF4 and HKFL5 intensities

Intensity decomposition during finalization



New simultaneous integration

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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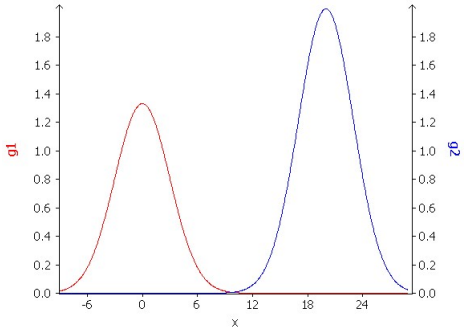
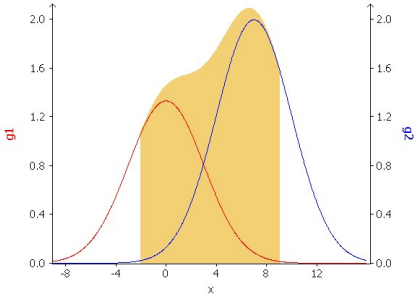
HKLF4 and HKLF5 play modes

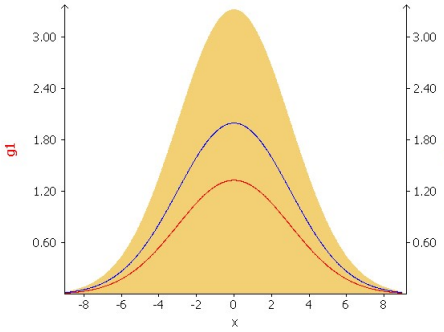
Fully separated

Partially overlapped

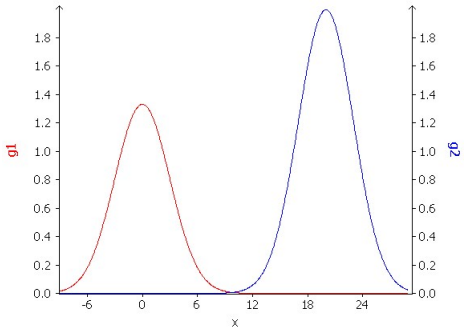
‘Full’ overlap

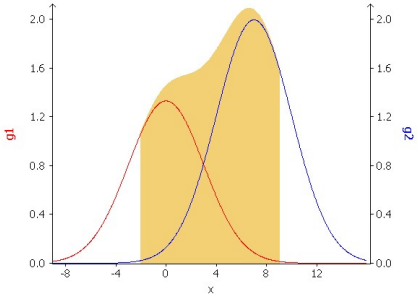


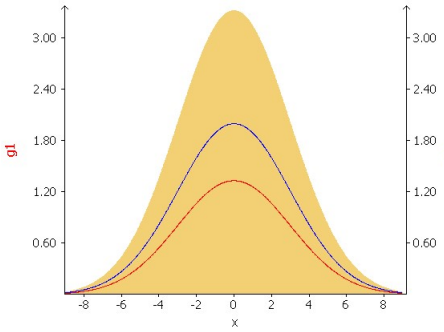




Component 1







Component 2

…

Component n

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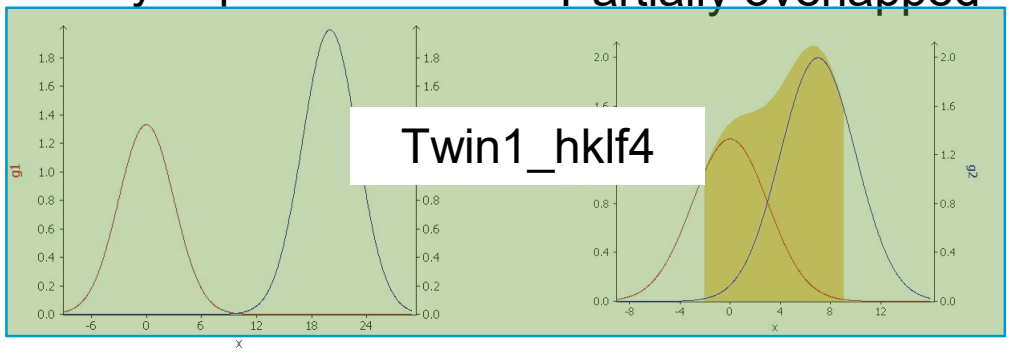
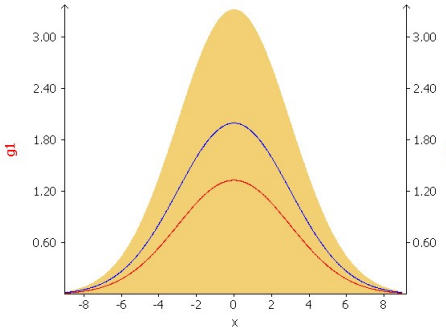
HKLF4 and HKLF5 play modes: 1st trial

Fully separated

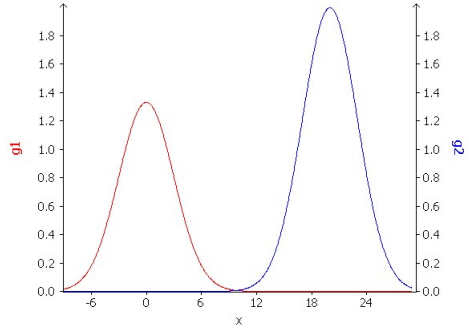
Partially overlapped

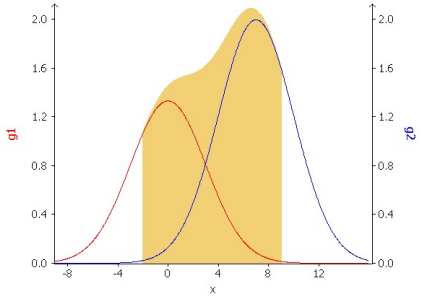
'Full' overlap

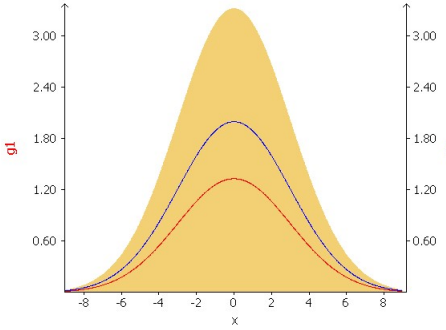




Component 1







Component 2

…

Component n

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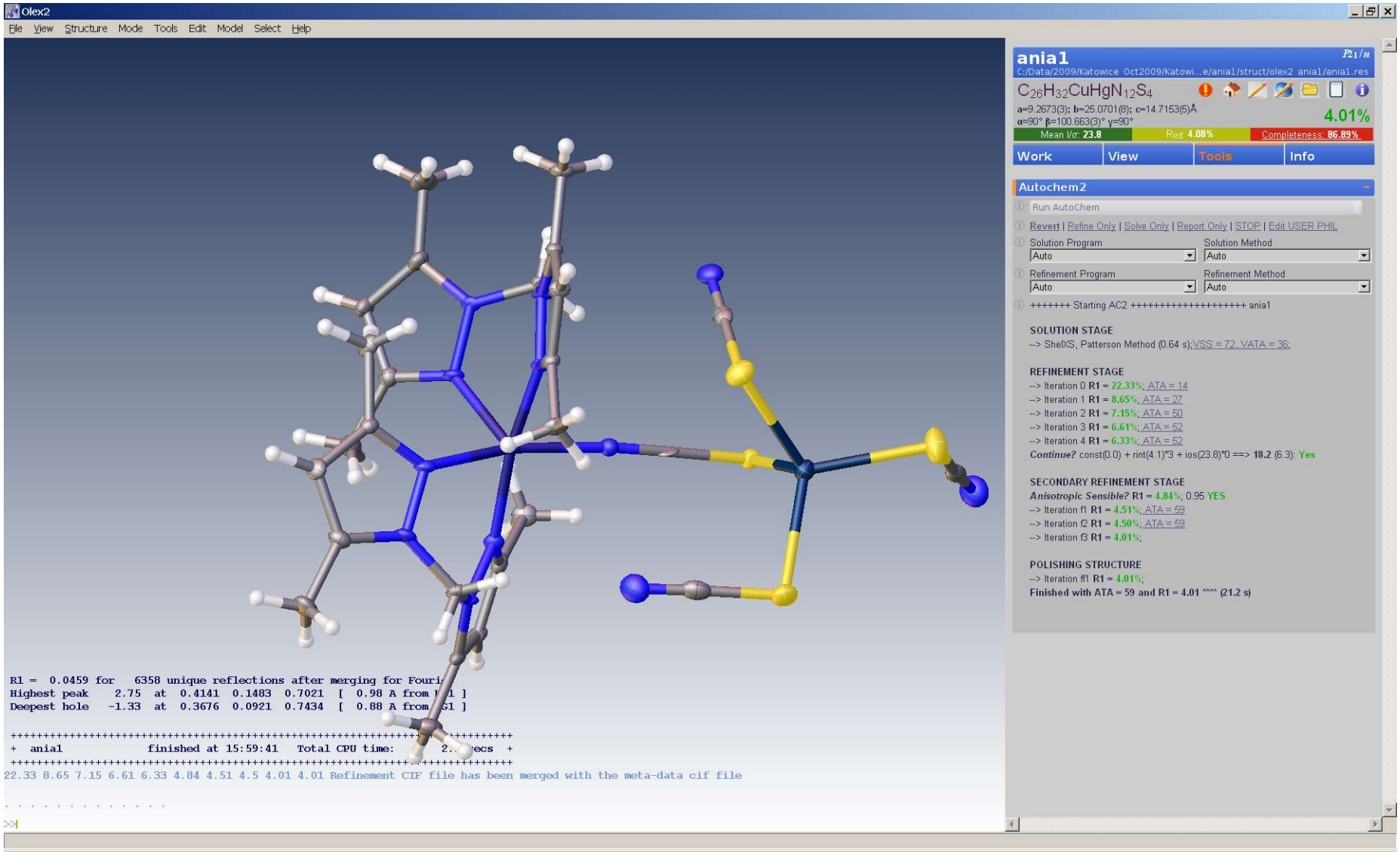
Structure solve and refine using AutoChem$^{2.0}$



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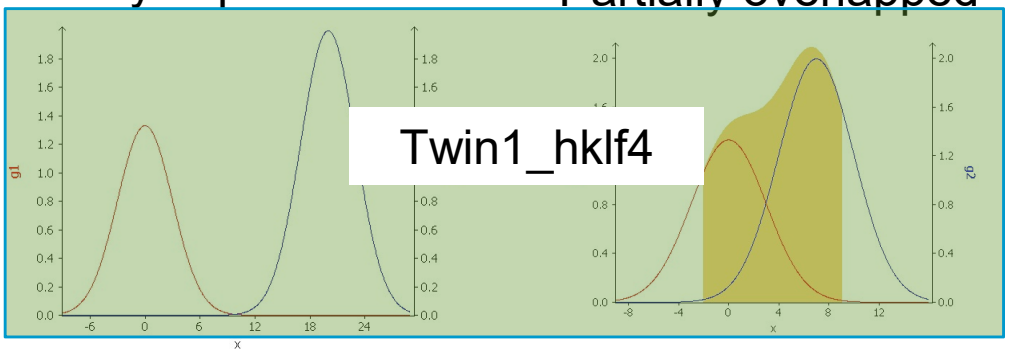
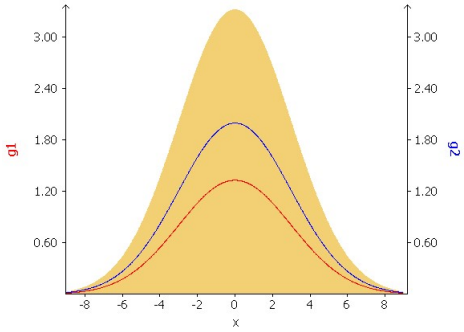
Get extra data in to solve the structure…

Fully separated

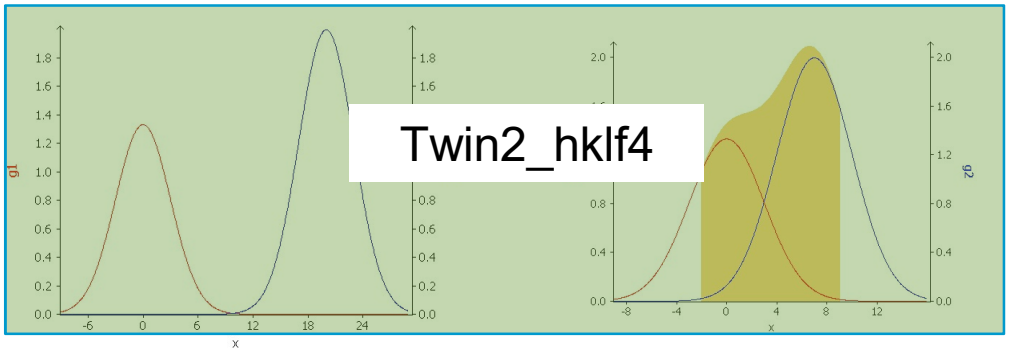
Partially overlapped

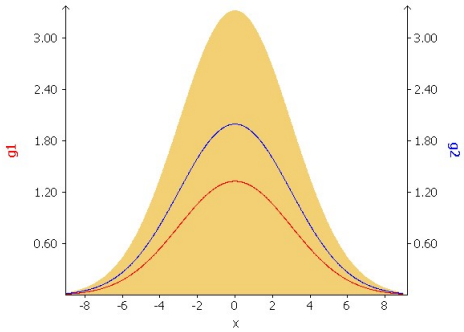
‘Full’ overlap





Component 1





Component 2

…

Component n

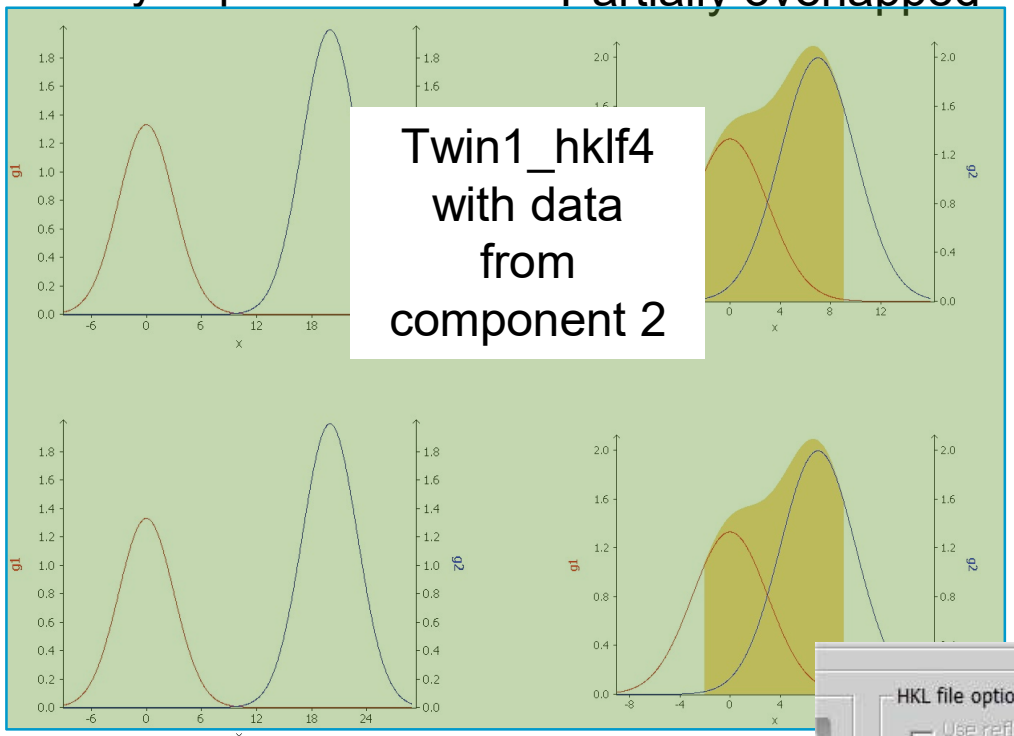
Rigaku oxford diffraction

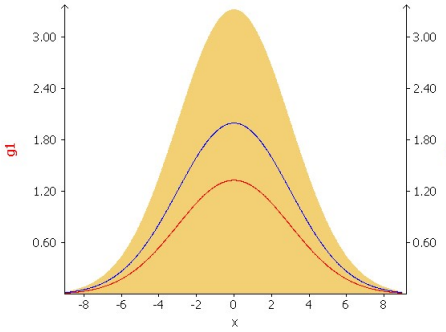
Get extra data in to solve the structure…

Fully separated

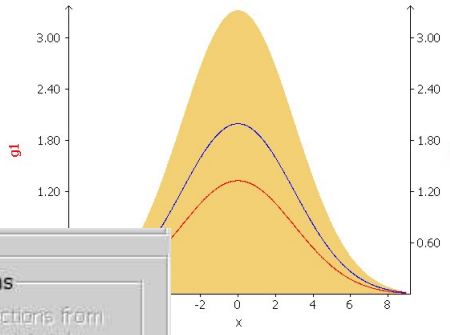
Partially overlapped

'Full' overlap

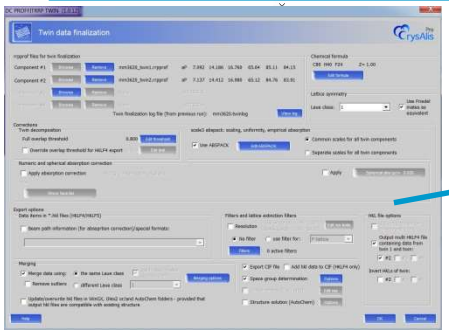


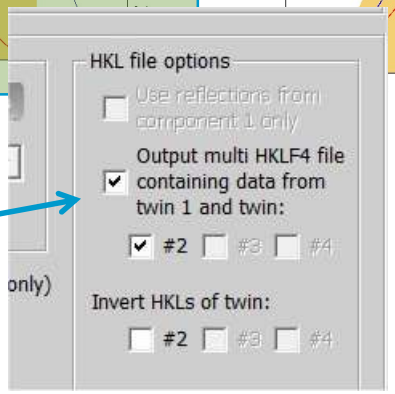


Component 1



Component 2





…

Component n

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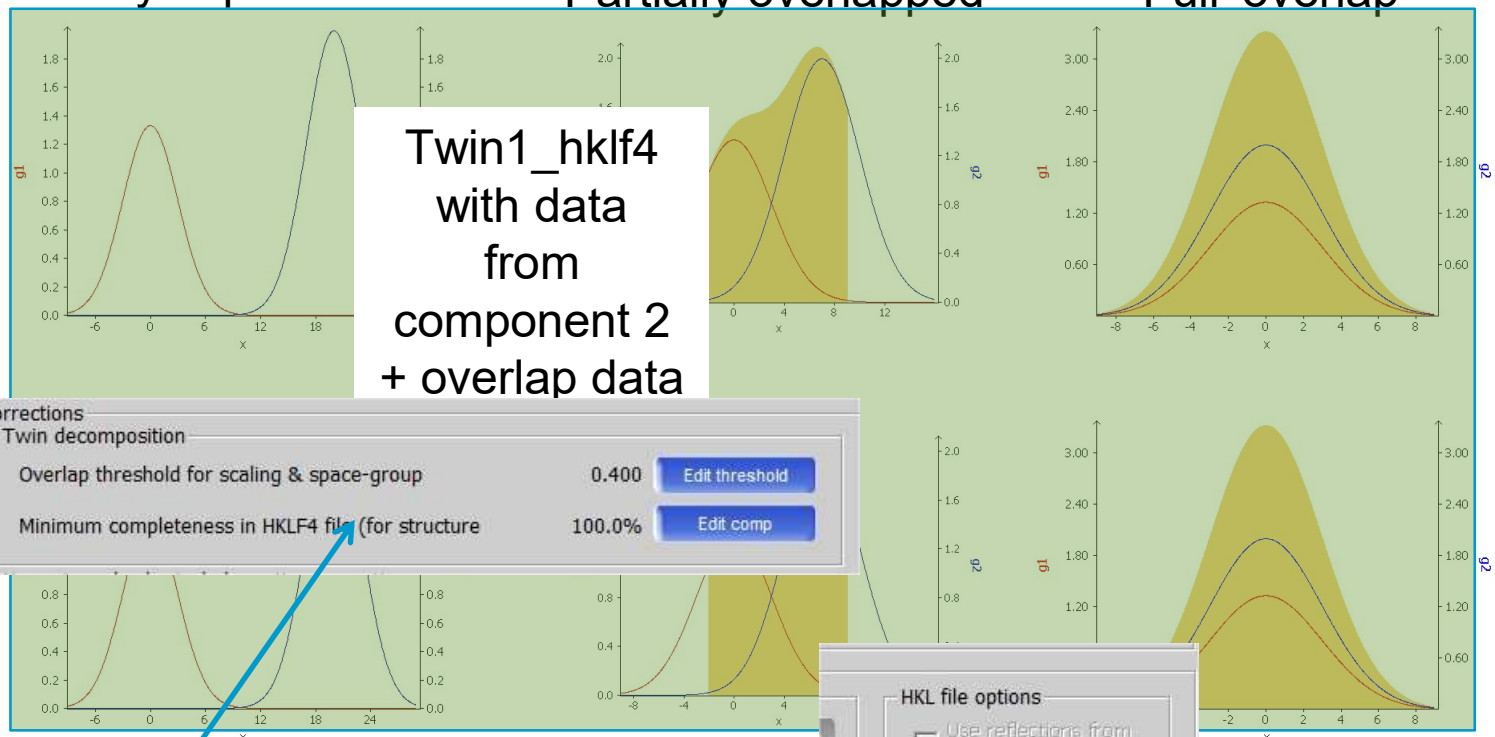
Not enough yet: Add full overlap data…

Fully separated

Partially overlapped

'Full' overlap



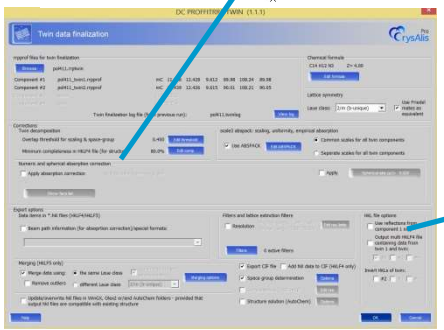
Twin1_hklf4 with data from component 2 + overlap data

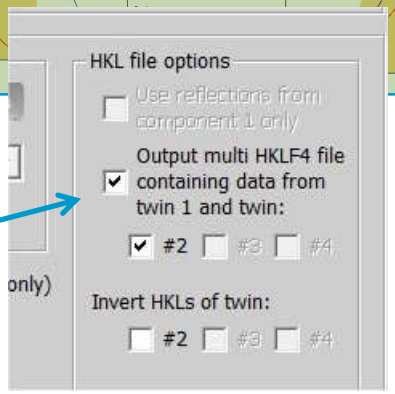
Component 1

Component 2

…

Component n





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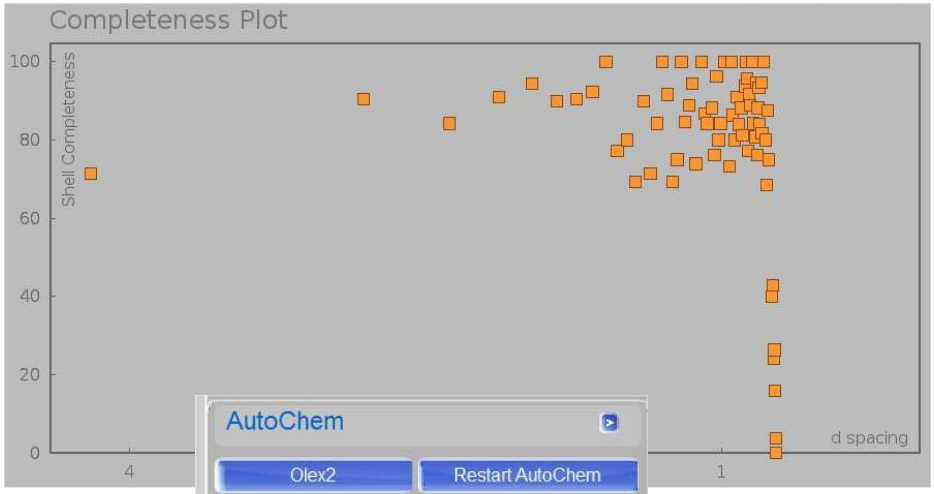
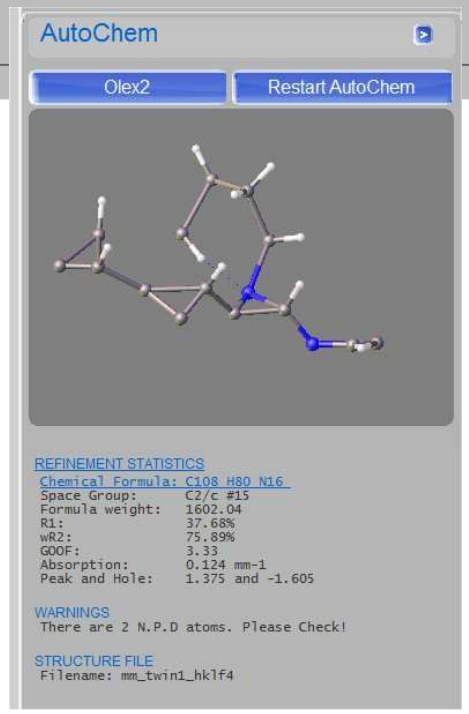
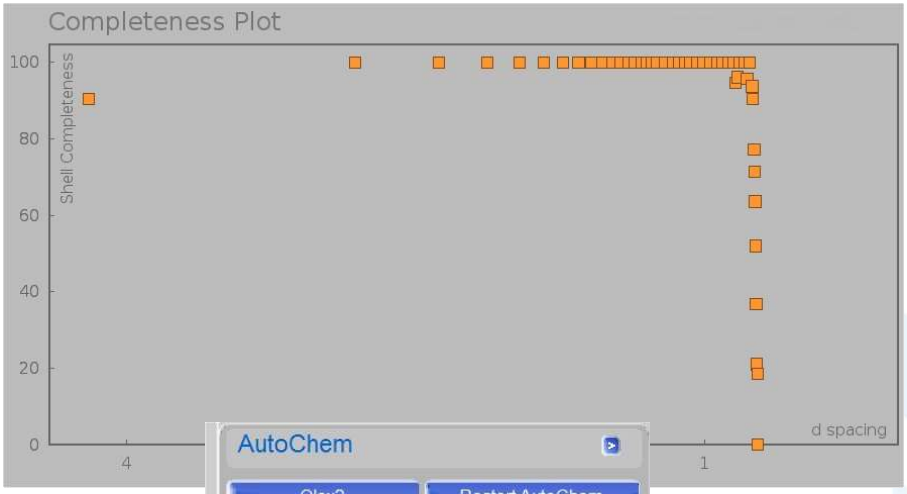
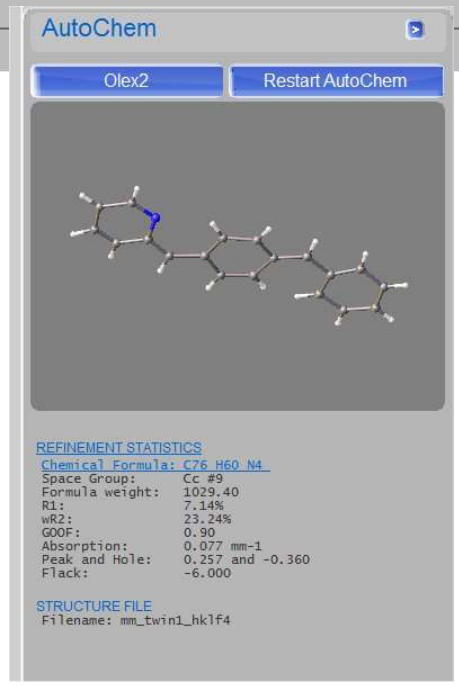
Why?









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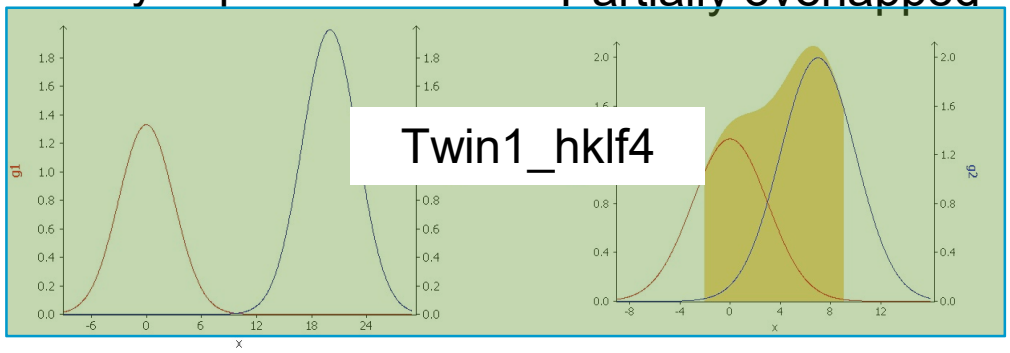
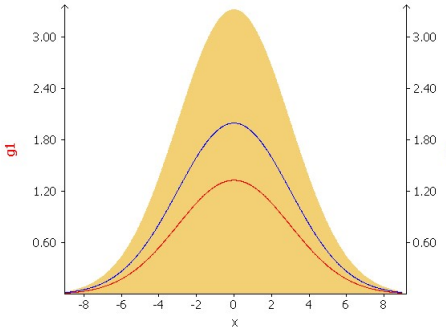
Solve done: Refine on good HKLF4

Fully separated

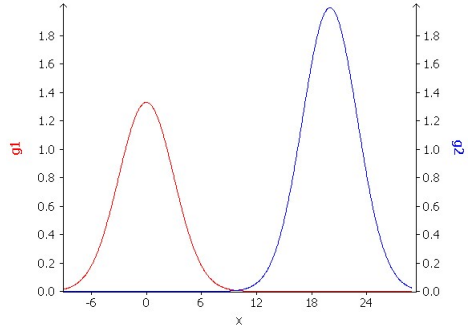
Partially overlapped

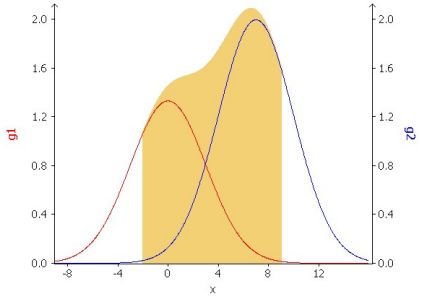
‘Full’ overlap

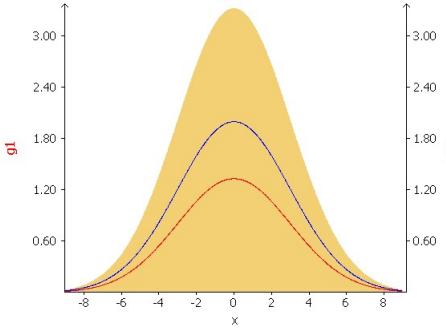




Component 1







Component 2

…

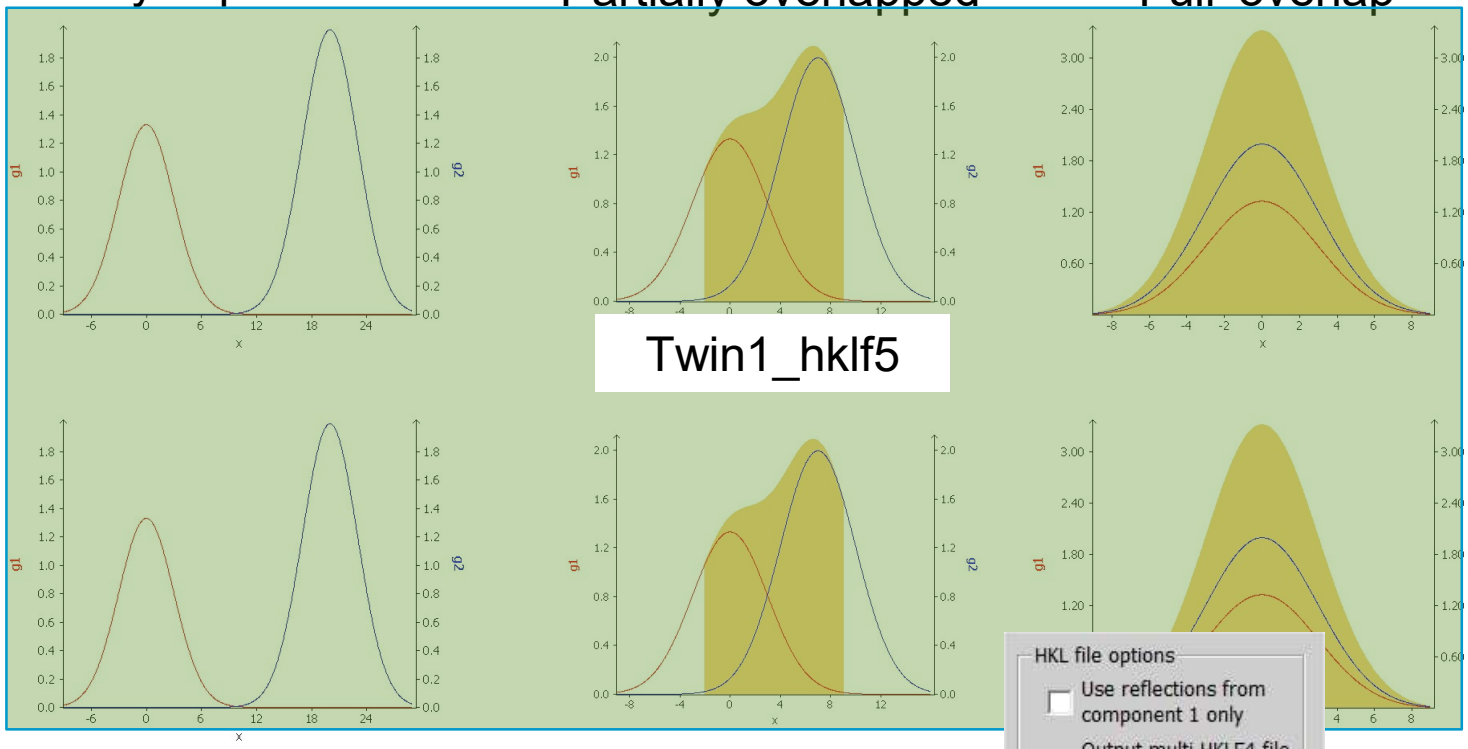
Component n

Solve done: Refine on good HKLF5

Fully separated

Partially overlapped

'Full' overlap

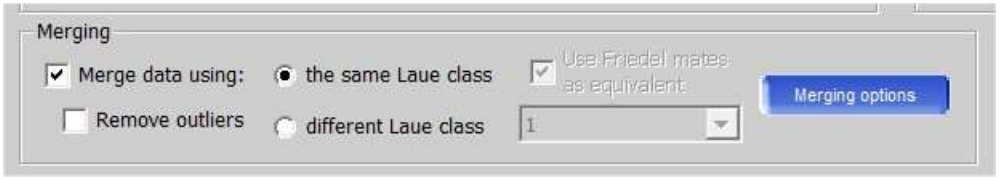


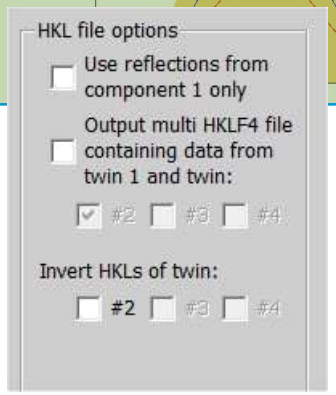
Component 1

Component 2

…

Component n





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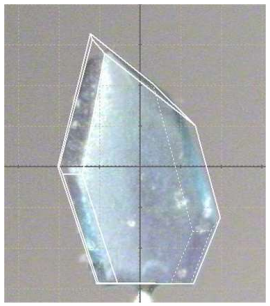
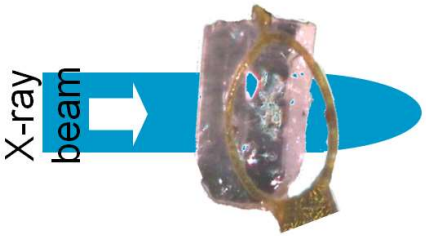
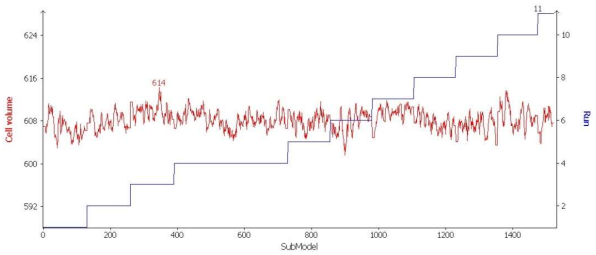
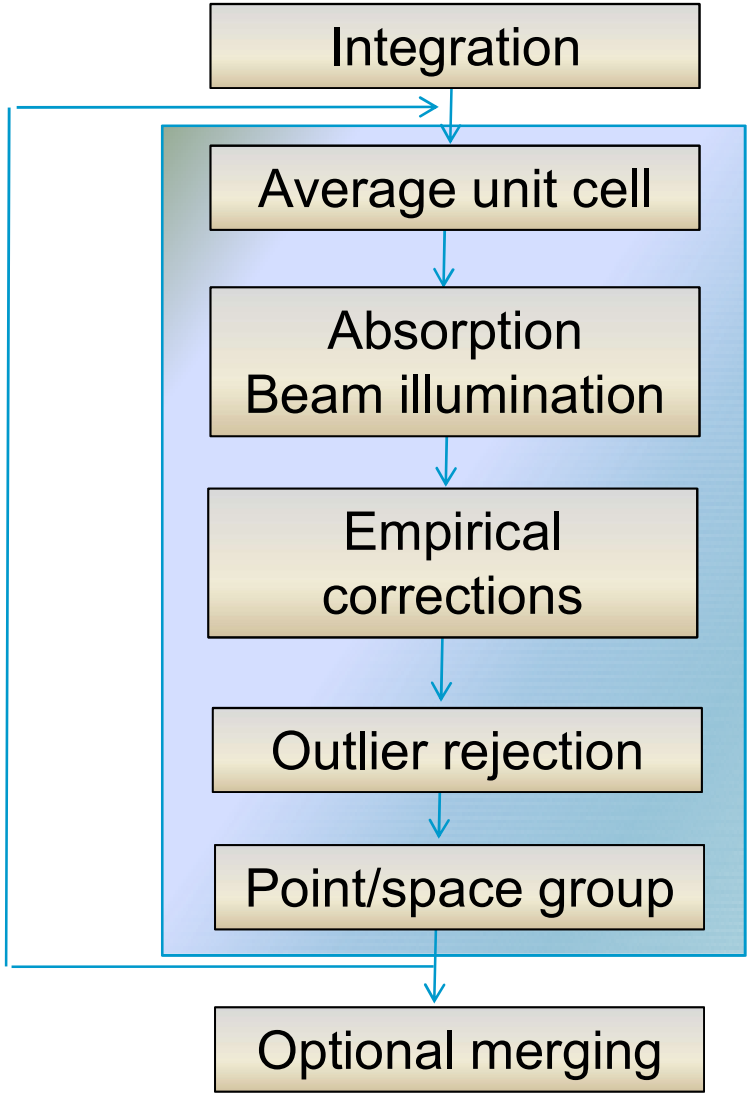
Good data quality through full post corrections









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Summary - twin

- Twins can be recognized early on during screening/pre-experiment
- With the graphic and computational tools in Ewald^{Pro} twin assignment is easy.
- De-convolution of overlap data gives good HKLF4 files.
- Sometimes solution boot-strapping requires different play modes
- HKLF4 and 5 files can be easily conditioned for top data quality with absorption, beam illumination and empirical corrections.

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Step by step guide on easy twin and super lattice faker

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Typical data reduction sequence

Unit cell finding

↓

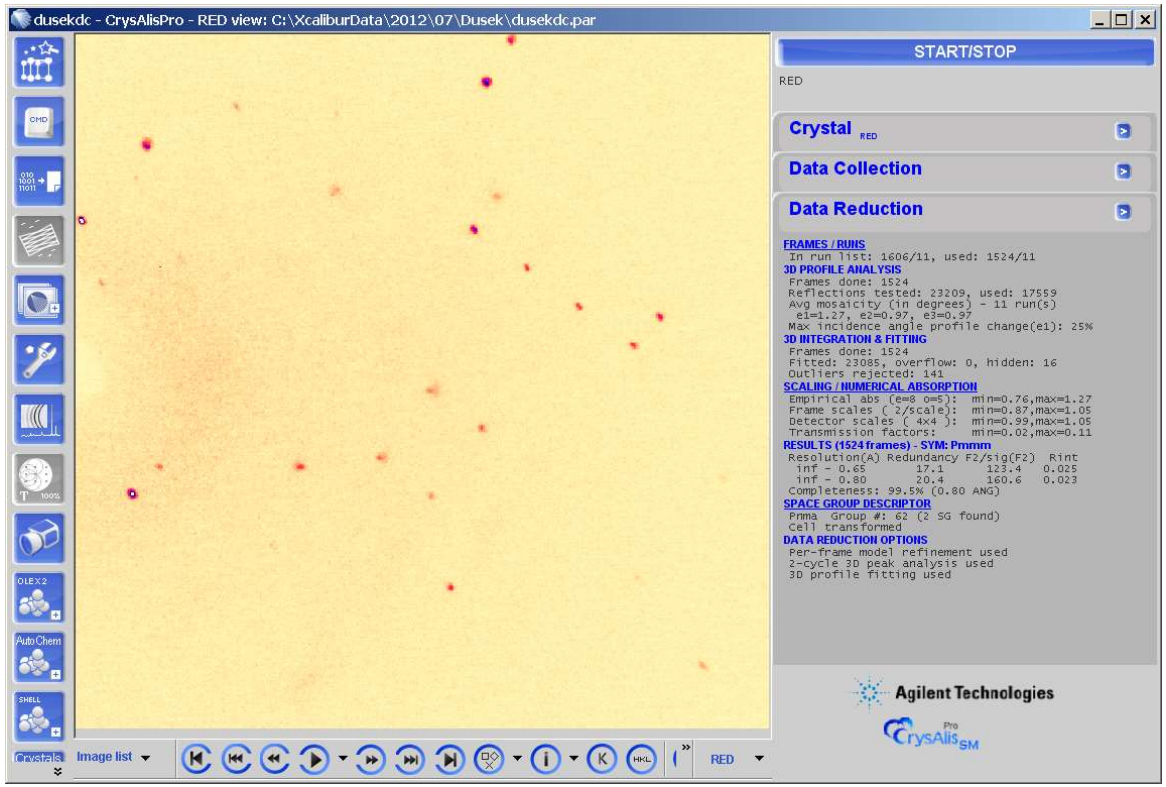
Profile learning

↓

Integration

↓

Post correction



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Post correction motivation

- Frame information to HKL file information
- Improve I/σ of redundant information
- Reduce 'systematic effects'

Note:
Post corrections can only correct observed data!

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