Training for single crystal XRD

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The Team

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Internet: http://isic.epfl.ch/X-Ray

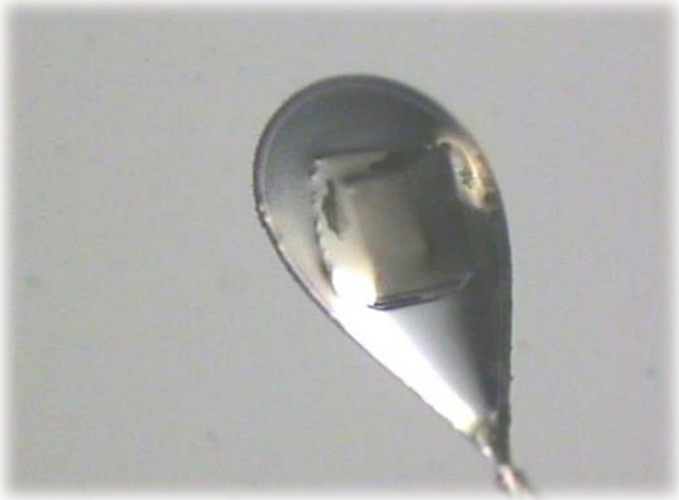
The Instrument

Rigaku Supernova
2 micro-sources, CCD detector,
kappa goniometer,
low temperature device

Single crystal (choose and mount)

- Crystals usually appear as unadulterated, homogenous and with ***well-defined geometric shapes*** (***habits***) when they are well-formed. However their external morphology is not sufficient to evaluate the crystallinity of a material.
- Crystals are mounted in loops
- Other techniques are also available

(capillaries, etc…)



Software

- CrysAlis(Pro)

This sofware is free of charge and can be dowloaded here:

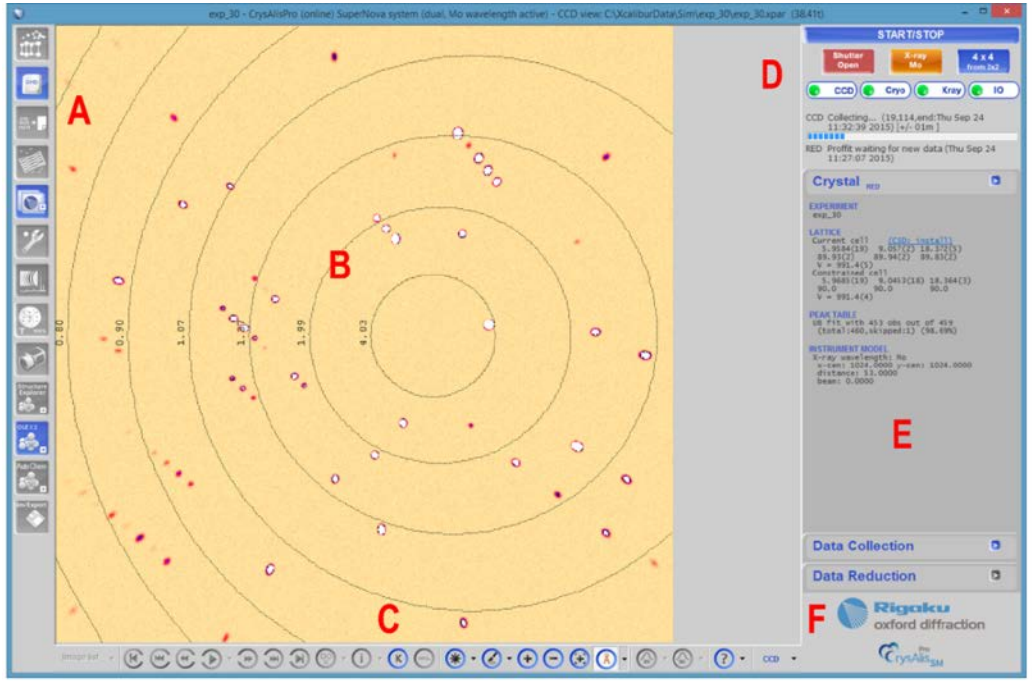
https://www.rigakuxrayforum.com/ (after registration)

It is divided into two main parts (Online and Offline)

Online takes care about the instrument and the data collection

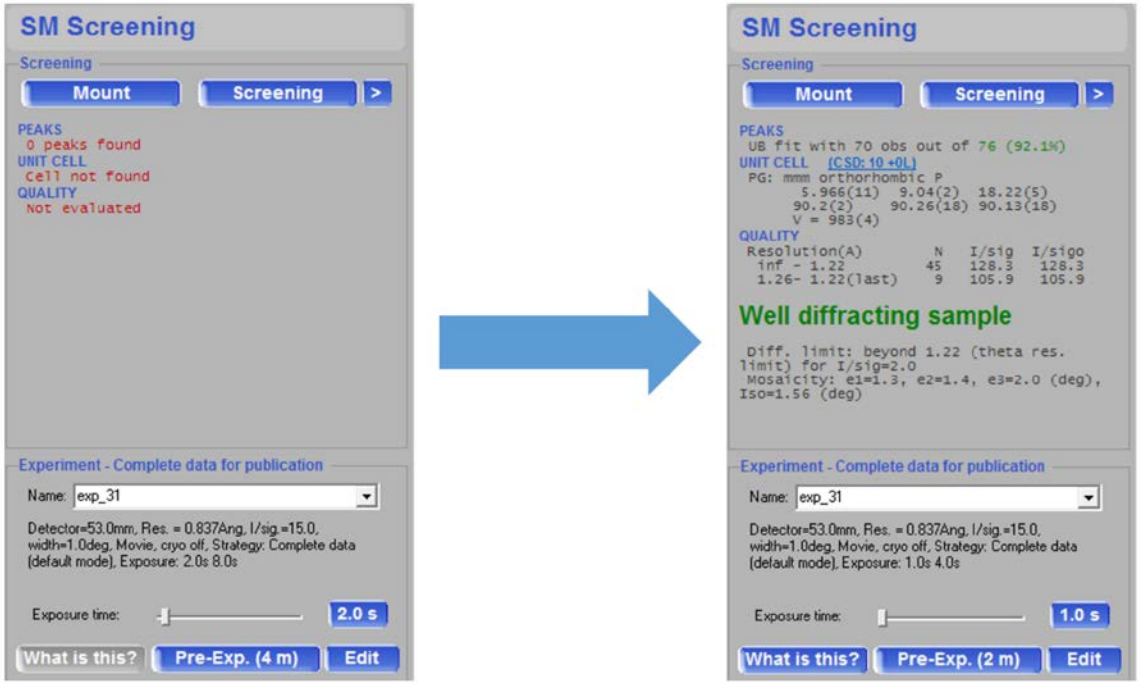
Offline (RED) allows the user to treat the data

Main Tools of the software



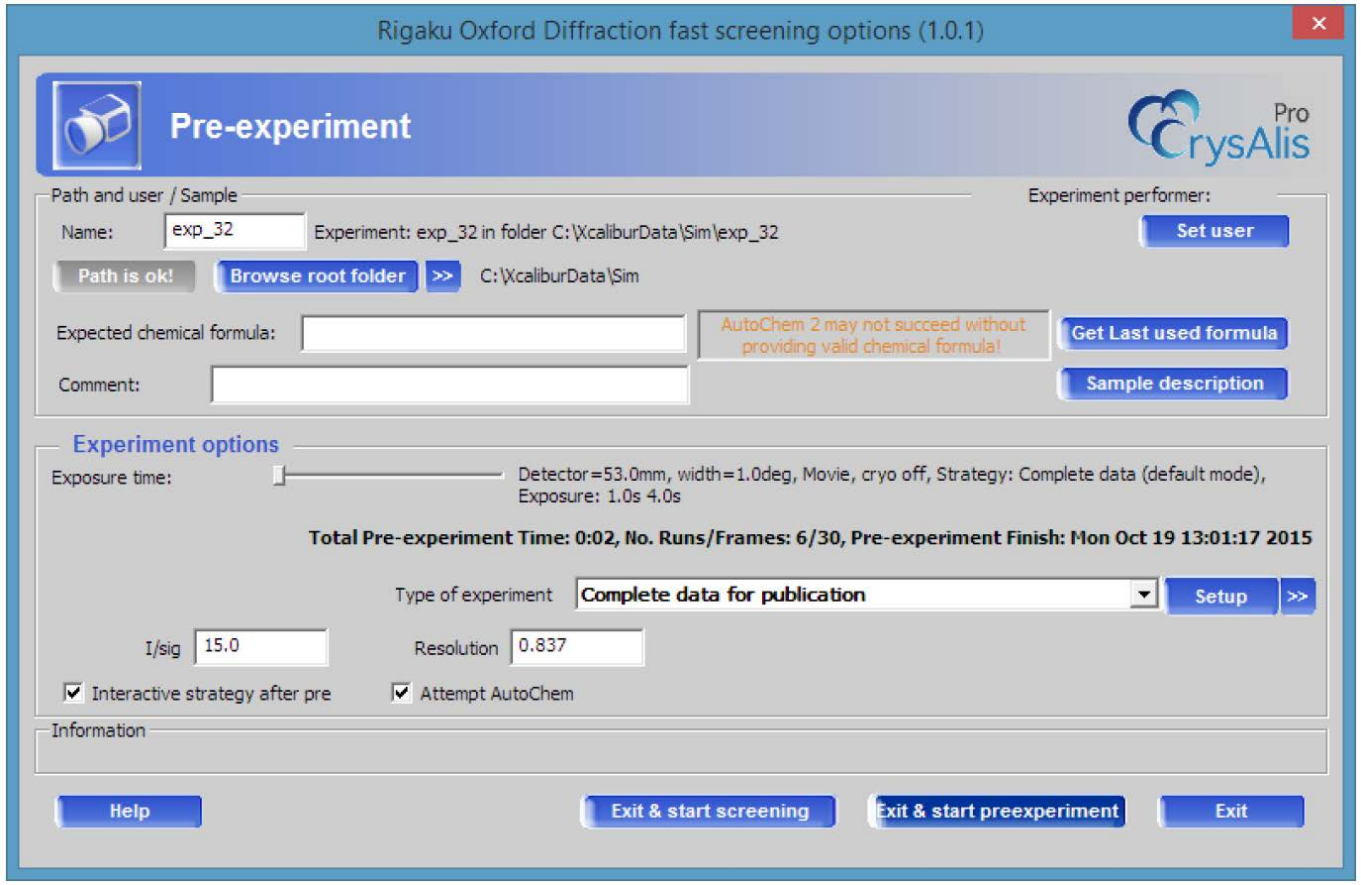
A: Power tools: access to the lattice wizard, command shell etc..
B: Main window: shows current frame in online mode, and selected frame in offline mode.
C: Layout and controls: allows user to scroll through frames, get peak information, change brightness, and use peak profile tools
D: Device control: Only shown in the online mode of the software, and shows status of the instrument
E: Provide information about the current unit cell, data collection progress, and a summary of the data reduction.
F: SM/PX button: allows the user to switch the software between small molecule and protein modes.

Screening a crystal



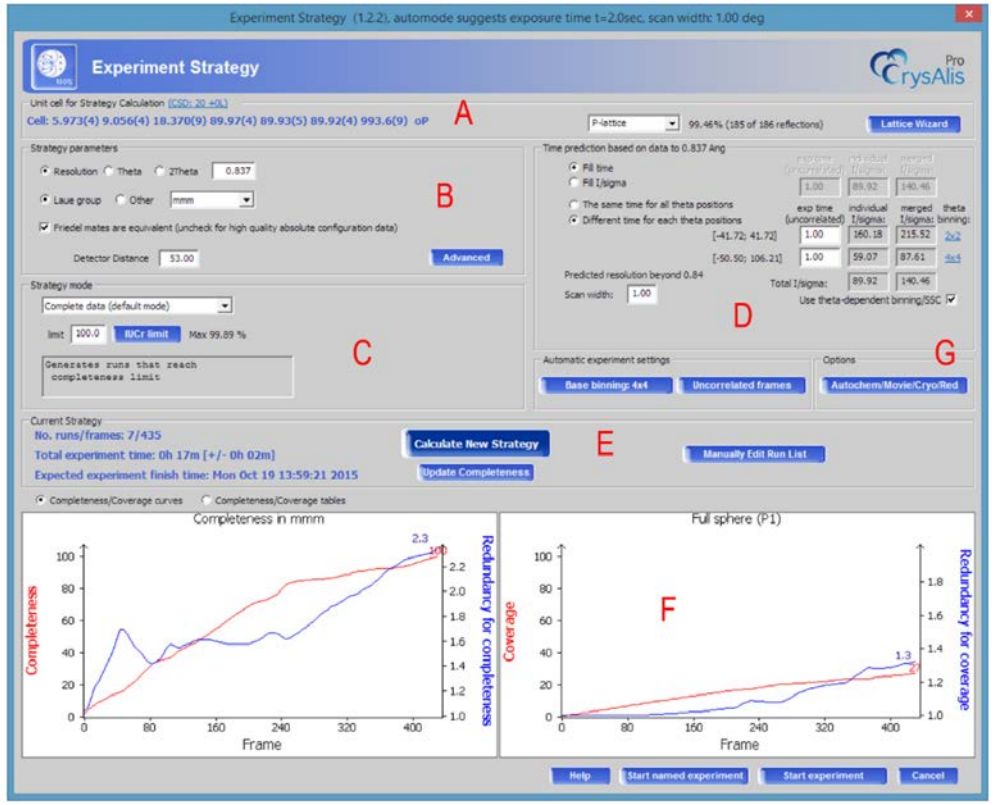
First start a new measurement on the online version, then mount and center your crystal.
The screening window will tell you how many peaks have been indexed, it will display the cell parameters and evaluate its quality (based on the intensity of the peaks). If the crystal is good then procede to the pre-experiment by clicking edit

Pre-experiment



This windows shows the details for the pre-experiment.
Insert the name of your sample (and the user), the chemical formula and the sample description. Modify the exposure time and the type of experiment according to your needs. The pre-experiment will collect 30 (Cu) or 15 (Mo) frames in order to get better cell parameters and a good strategy for the real data collection.

Strategy



A. Unit cell for Strategy Calculation: contains the proposed unit cell, calculated from the pre-experiment.
B. Strategy parameters: it is possible to set the maximum resolution of the data collection etc…
C. Strategy mode: includes options such as complete, redundant, and time-limited collections.
D. Time prediction: shows the predicted exposure times, based on the pre-experiment data and desired I/σ value (usually set to 15 for good quality data).
E. Current Strategy: details the number of frames and length of the experiment. If any changes are made to sections A, B, C or D the user must click on the Calculate New Strategy button to update the experiment details.
F. Coverage: graphs or tabulated views of the proposed data collection.
G. Settings/Options
Click on Start Experiment to begin

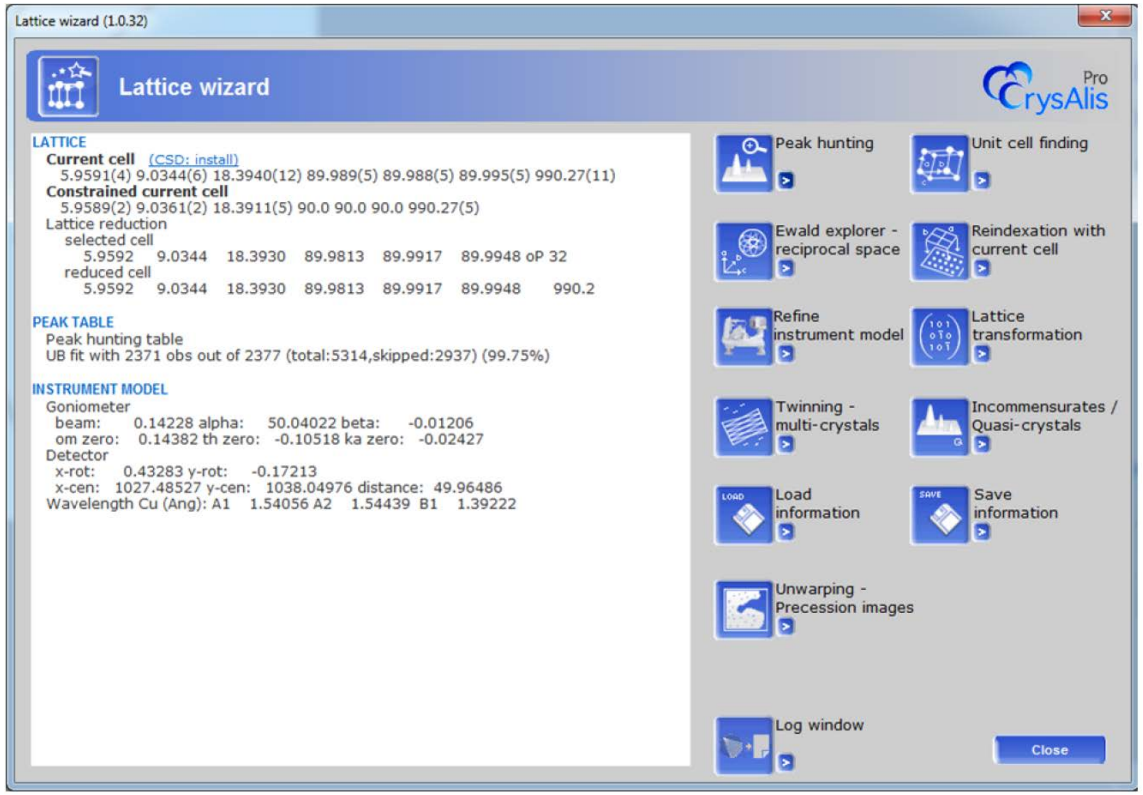
Data Collection and Reduction

- Data reduction starts automatically with the beginning of data collection and uses intelligent routines which tune the parameters to give the best data quality. Processed data are always available and accompanied by real time on-screen feedback of data quality and completeness.
- The measured data can be treated in two ways by the offline version of CrysAlis (Pro): Automatic or Manual (with options)
- Automatic treatment is good for very good samples (if not too absorbing)

Data reductions with options

- First of all, locate your par or run file in the folder containing the measured data
- Open it by clicking on it (it is loaded by Crysalis (Pro) Red (offline version)
- Select the Lattice Wizard from the menu at left

Data reductions with options



This tool will let you find peaks in the collected frames and find the unit cell and evaluate the quality of indexation and determine if twins or incommensurates are present.

Data reductions with options

- To change the settings used by the profile fitting data reduction, click on Start/Stop and select Data reduction with options. There are six windows, and the most commonly used settings are discussed below:
- Orientation Matrix: the user can choose which lattice extinctions to filter out. There are also options to choose unit cells from twin components or to use q-vectors determined for incommensurate structures.
- Experiment run list: by default the full run list is used during data reduction, but entire runs or individual frames can be removed using this window.

Data reductions with options

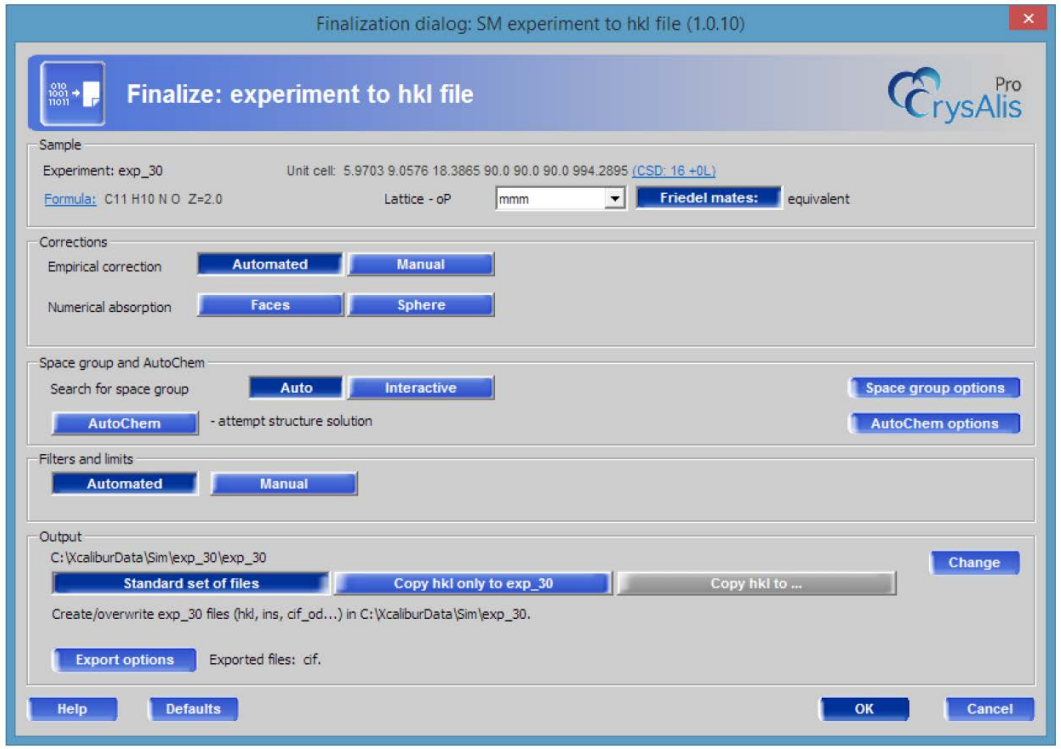
- Basic algorithm parameters: there are three levels for following crystal movement during a run: moderate sample wobble, significant sample wobble, and discontinuous jumps. This window also gives the user the option to remove any data generated from previous data reduction runs and also to edit several special sample movement parameters.
- Background evaluation: the method for determining the background correction can be changed from the standard “Average” method, to a “Smart” correction. For datasets in which the background is non-uniform, using the “Smart” method is often beneficial.

Data reductions with options

- Outlier rejection: the choice of outlier rejection is usually based on the chosen Laue group but can be altered.
- Output: details about the output, including:
- re-naming the output files
- the choice of automatic or manual space group determination via GRAL
- automatic generation of unwarp (precession) images

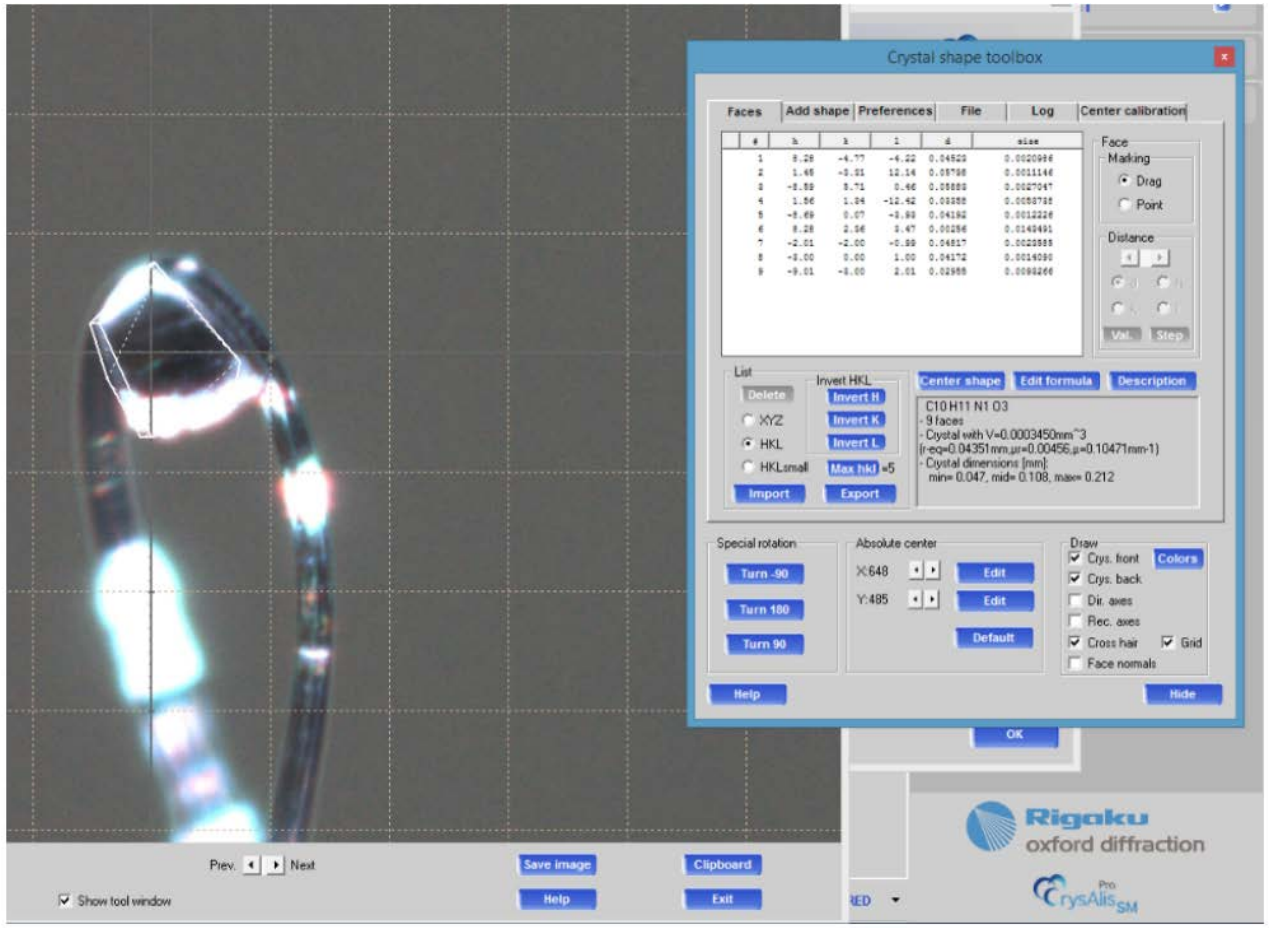
Data reductions with options

- Finalization of Data



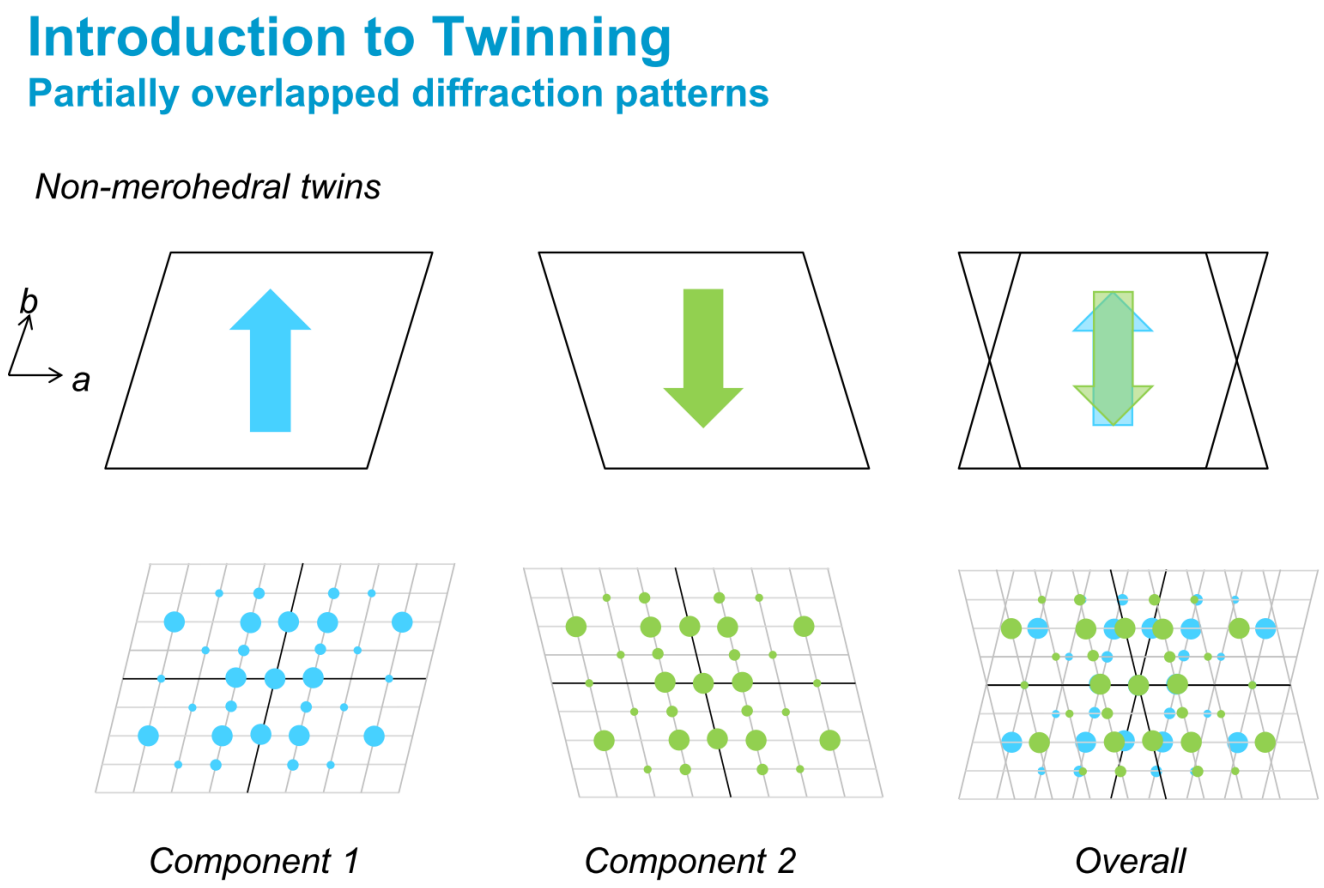
When data reduction is over, in order to get hkl file we need to apply corrections (based on symmetry, absorption, etc…), to scale the frames and try to determine space group.
The refinalize windows shows:
Sample (contains the cell parameters, the Laue group and the chemical formula)
Corrections (empirical absorption correction, frame-scaling and numerical absorption (based on faces))
Space group and Autochem (determination of the space group and try to solve the crystal structure)
Filters and limits: filters and limits (resolution for instance) may be applied
Output: hkl, ins, p4p, cif_od etc are created or overwritten

Crystal shape modelling (face indexing)



It uses the movie registered before starting the data collection and then by simple clicks allows the user to index the faces of the crystal, to determine exactly its size and to apply precise absorption corrections.

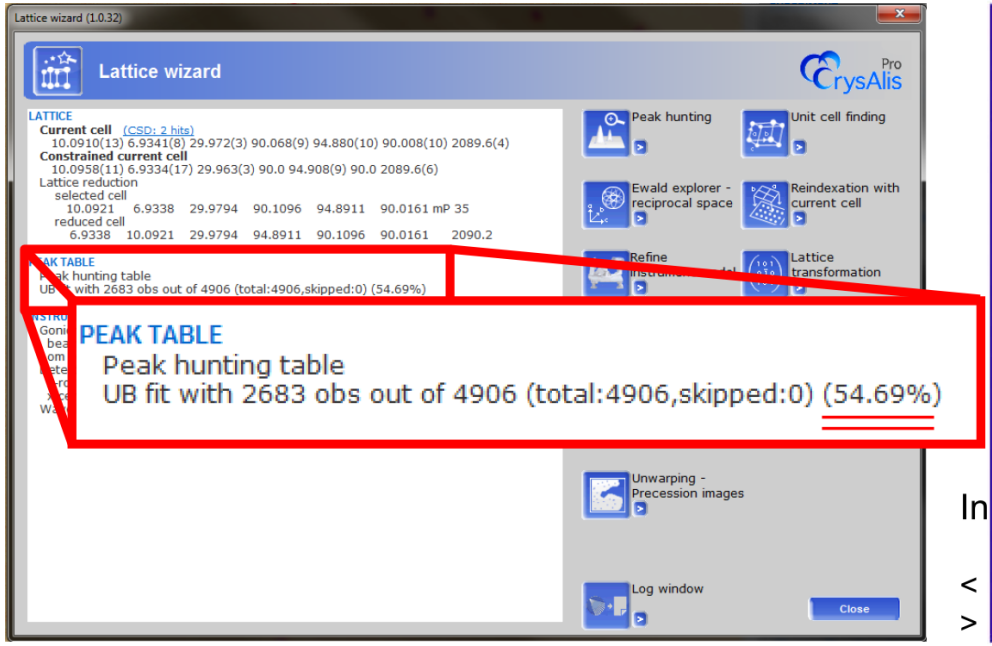
Twins



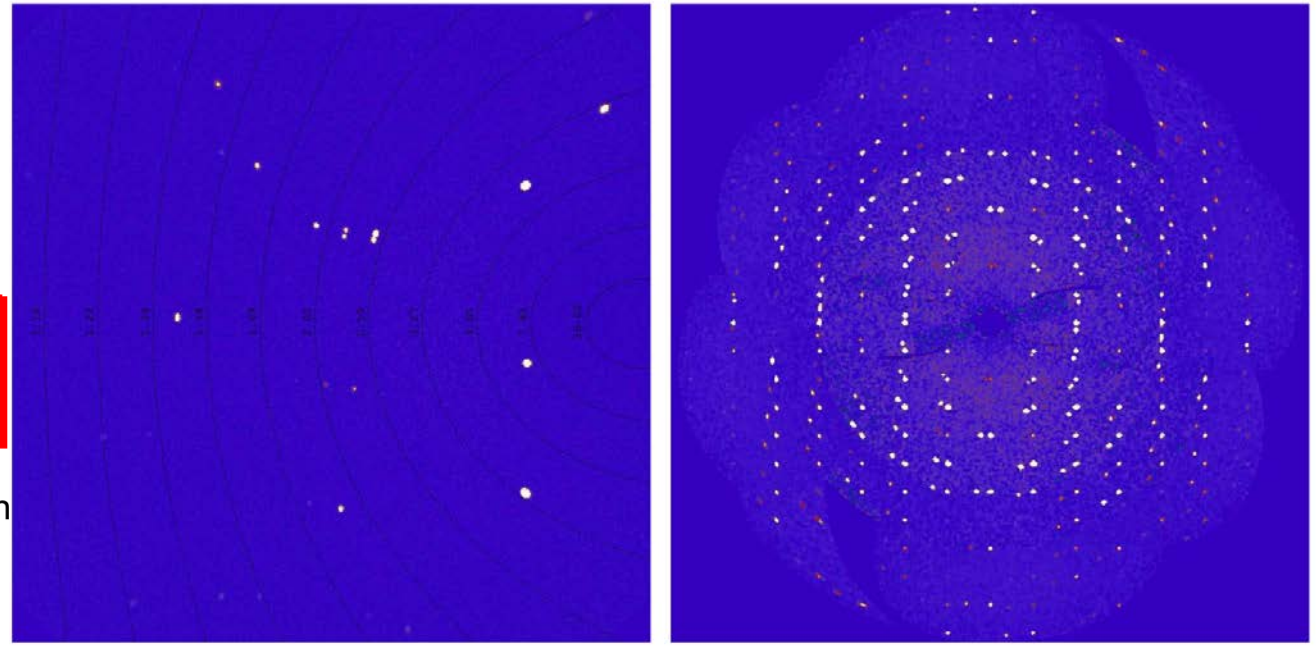
This type of twins can be treated during integration

Twins (diagnosis)

Lattice Wizard



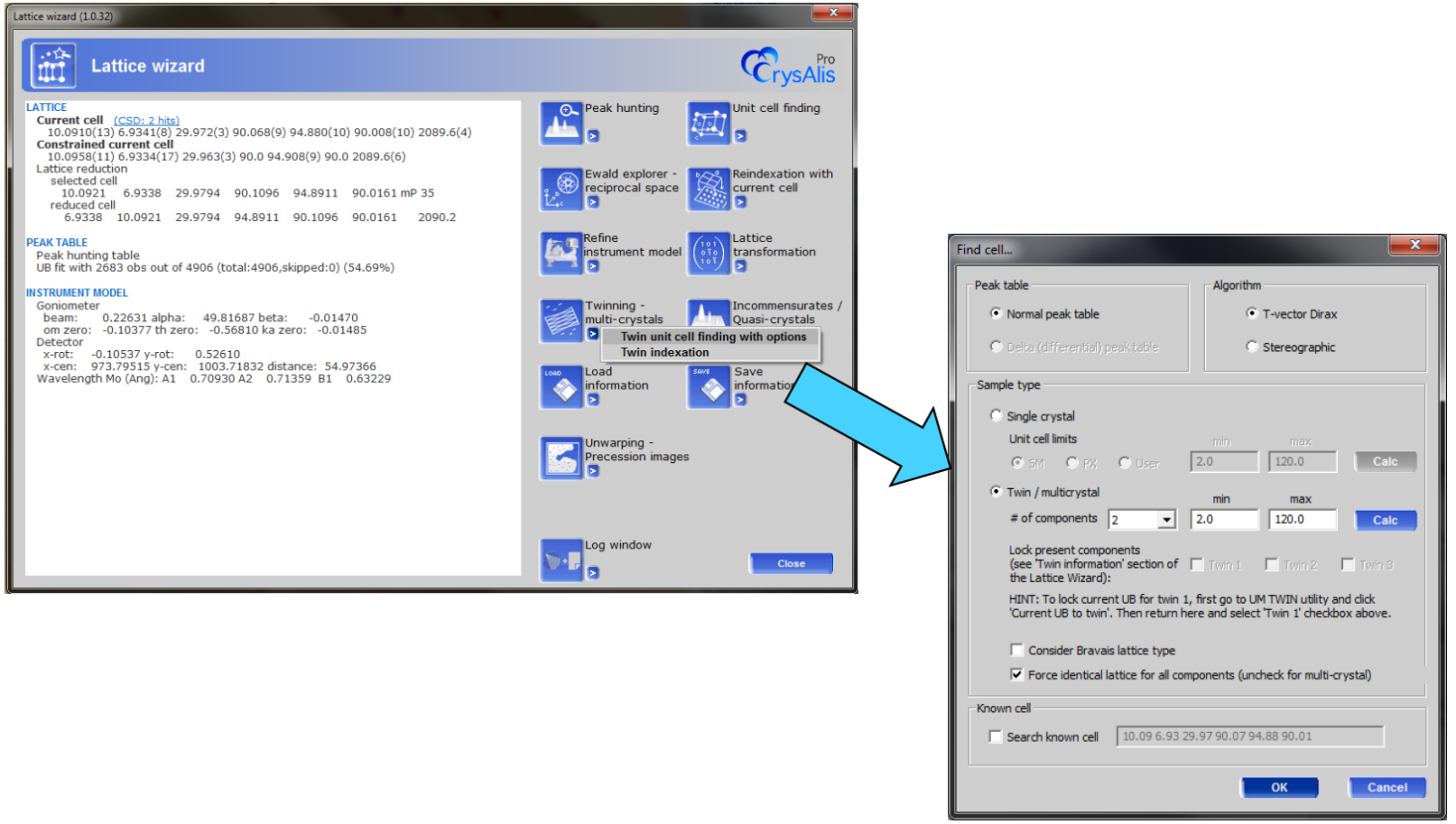
Diffraction and unwarped images



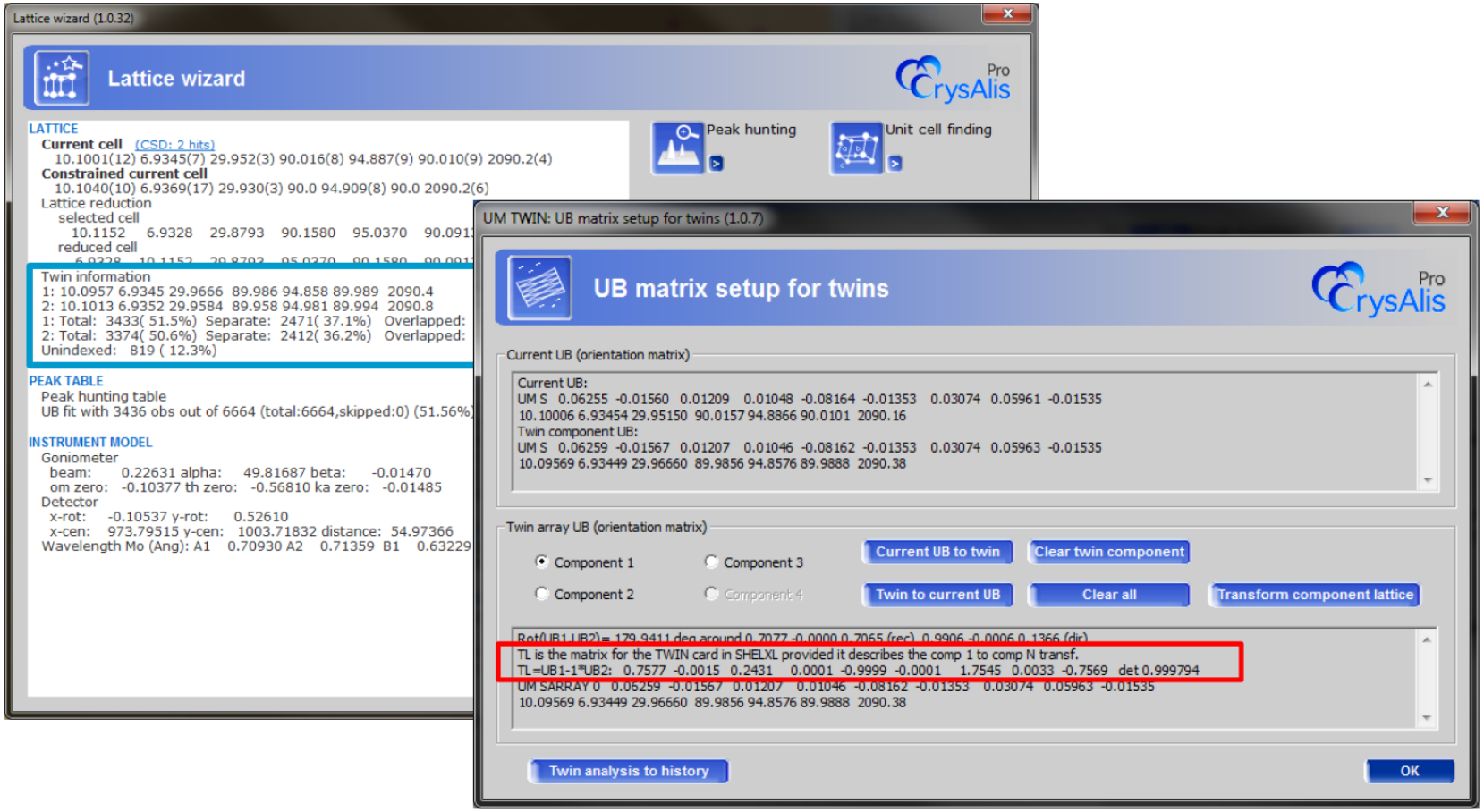
Twins (Automatic indexation)

- Automatic indexing will:
- 1. try to find an orientation matrix with the highest percentage
- 2. save it as first component
- 3. try to obtain another orientation of the cell which indexes the largest % of the remaining unindexed (wrong) peaks
- 4. save it as next unused component
- 5. Refine cells simultaneously

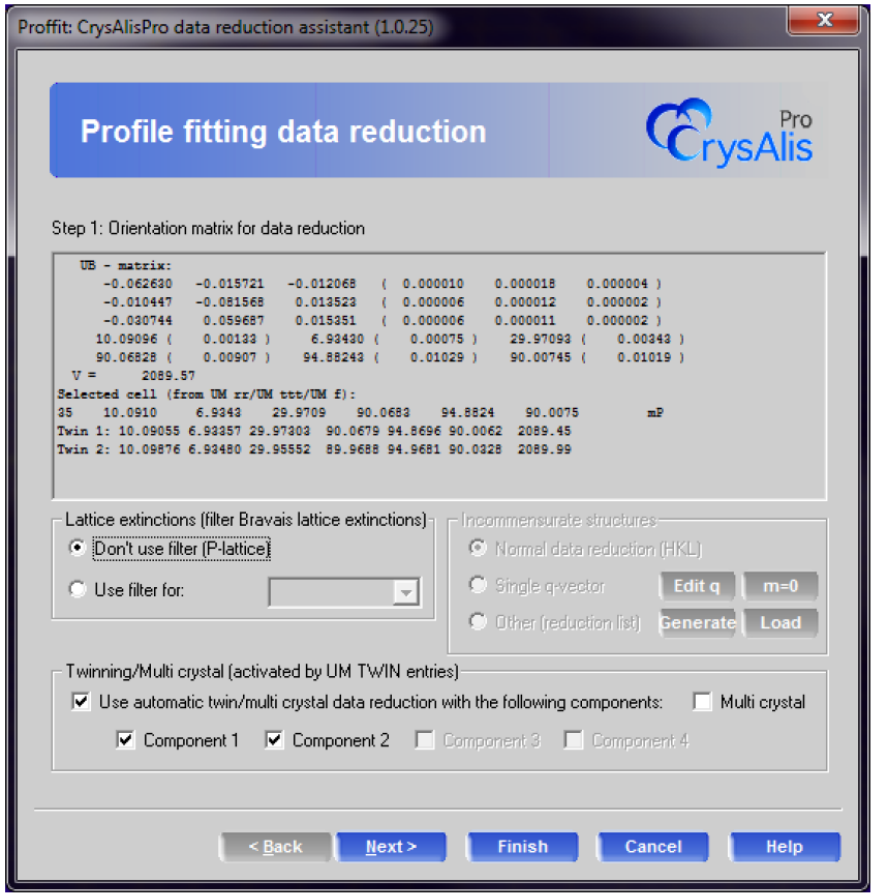
Twins (Automatic indexation)



Twins (Automatic indexation)



Twins (Data reduction)



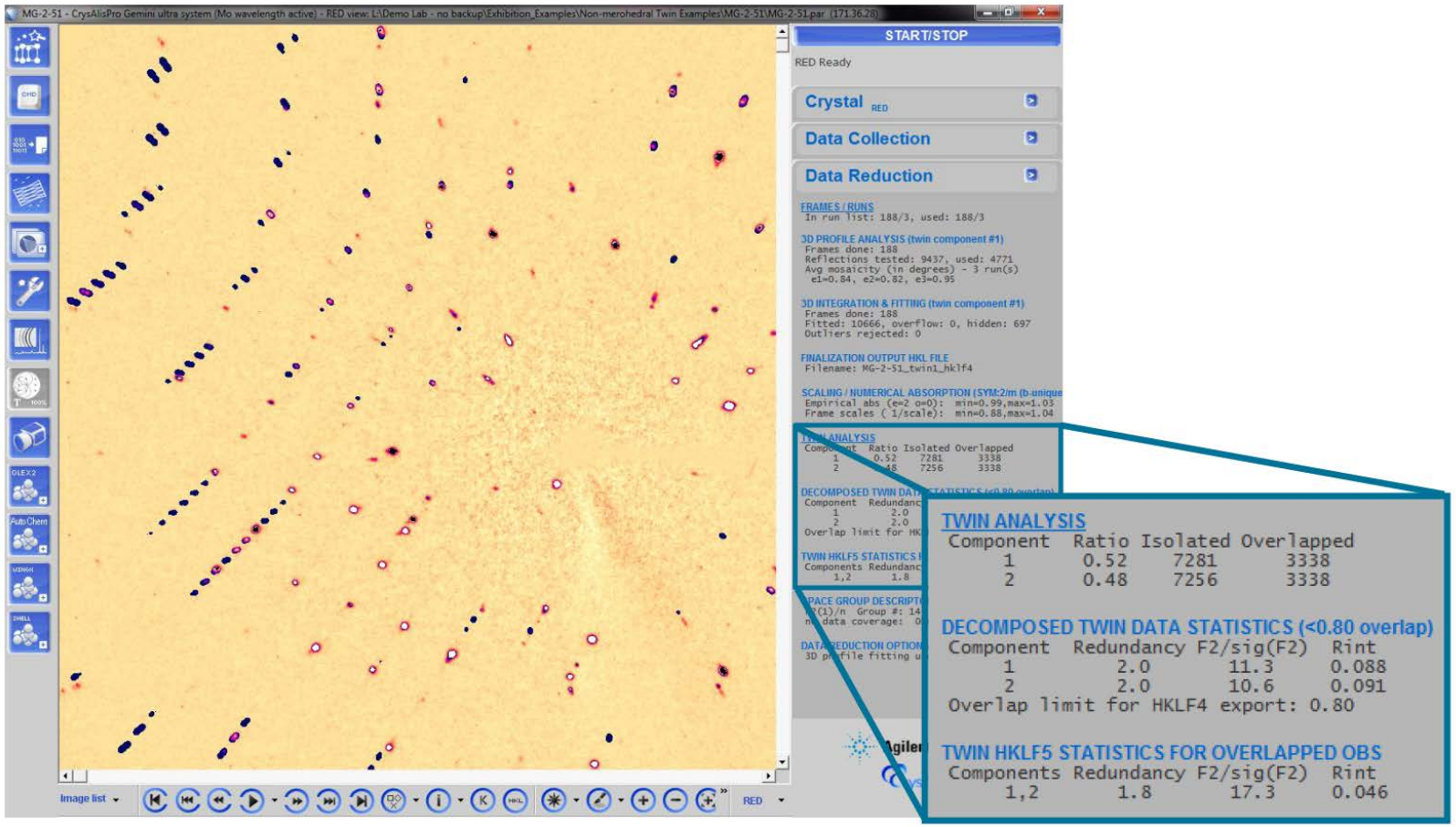
Difficulties

- Overlap prevent accurate determination of peak intensities
- Data completeness can be too low for structure solution

Goals

- Extract accurate intensities for all reflections for good refinement results
- Where possible, accurately deconvolute (de-twin) overlapped peaks to improve completeness

Twins (Data reduction)



Twins (Data reduction)