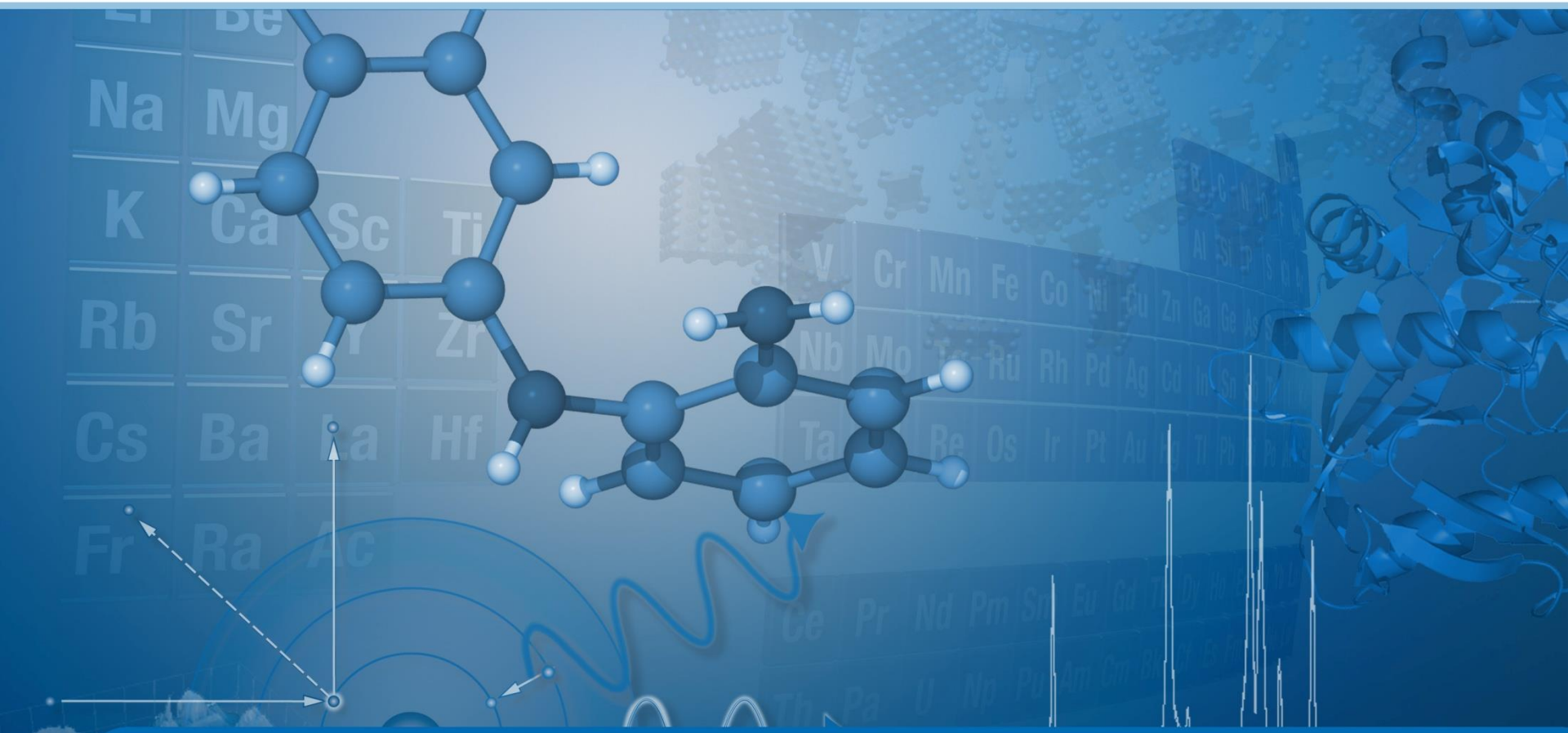
Crystal Indexing in PROTEUM3

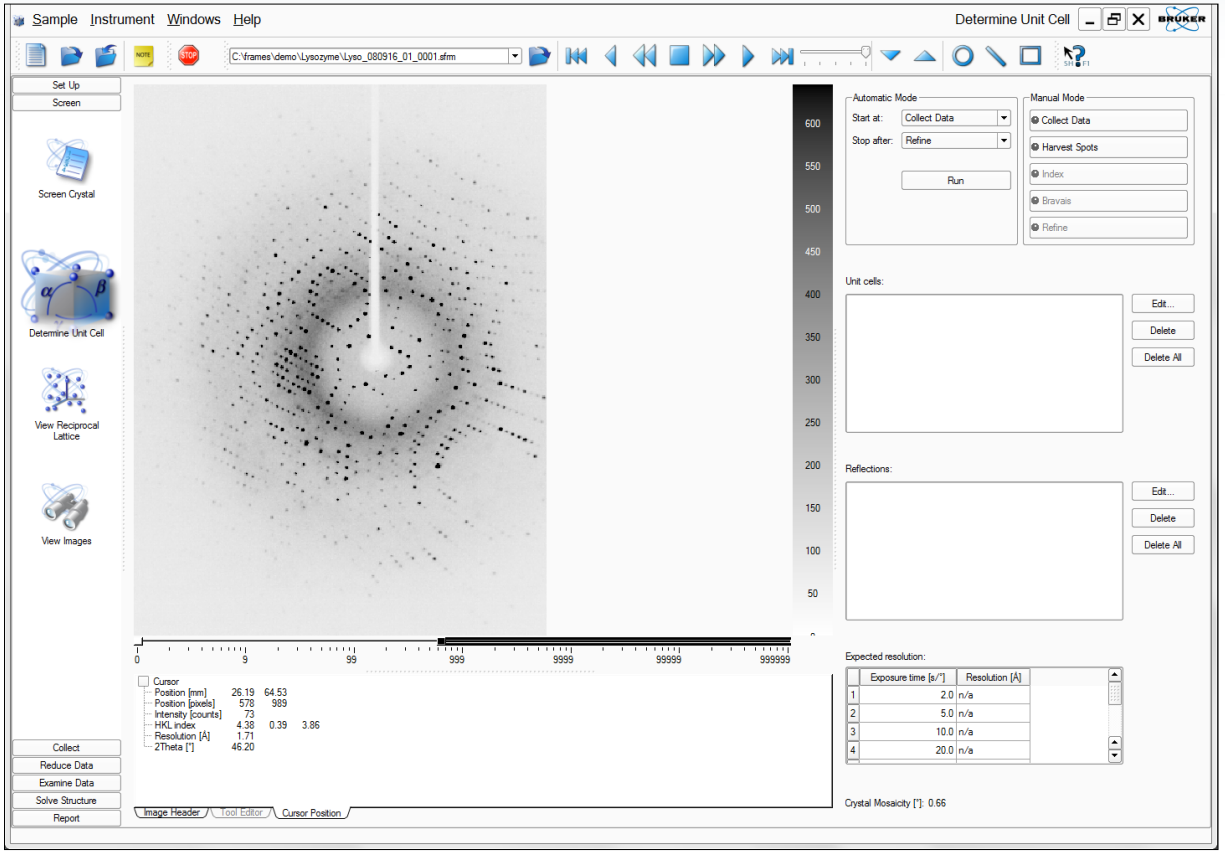


Indexing
Screen

- Determine Unit Cell

  Finding the orientation of the crystal lattice relative to the instrumental setup

- Automatic
  - Can be run from any step
- Manual
  - Collect Data
  - Harvest spots
  - Index
  - Refine
  - Bravais
  - Refine
- Methods
  - Difference vector
  - FFT
  - Least squares (Cell_now)



Determine Unit Cell
Collect data

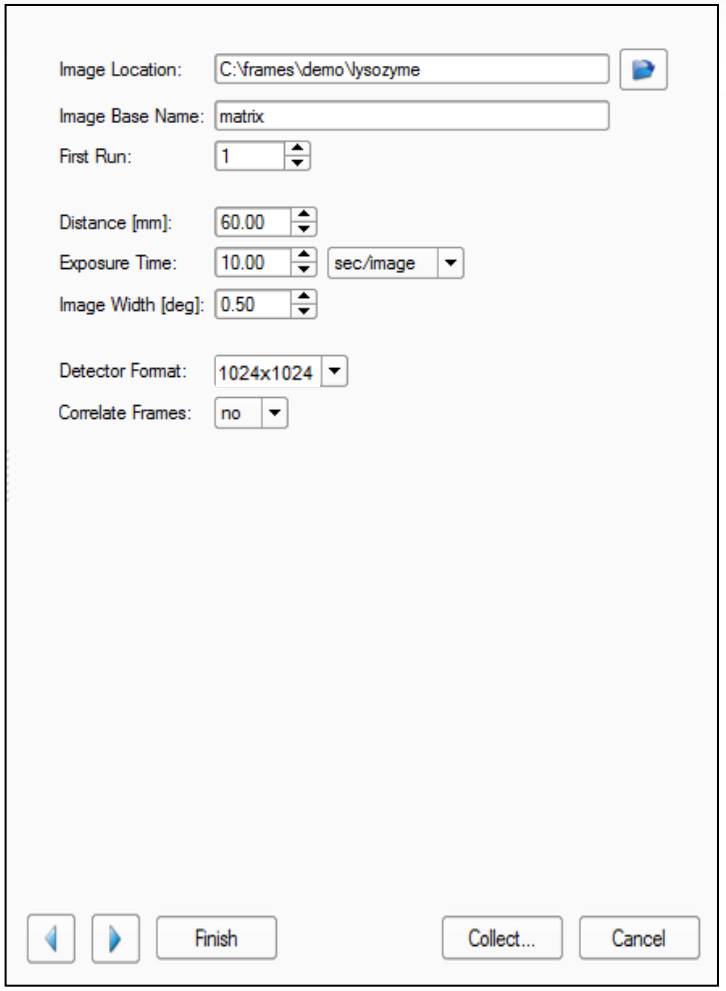
You can collect images for indexing based on preset runs in the bn-config.py file. Since every crystal diffracts differently, you to change critical parameters to match the sample.

- Distance
- Exposure time
- Image width

Standard setting for CMOS and CCD detectors

- Detector Format always 1024x1024
- Correlate Frames always "no" for CMOS
- Runs or scans are defined as angular sweeps in different crystal orientations. They can be setup in the bn-config file or from the *run experiment* menu
  - 2 runs of 3° separated by 90° in reciprocal space
  - *Filename*_01_0001.sfrm

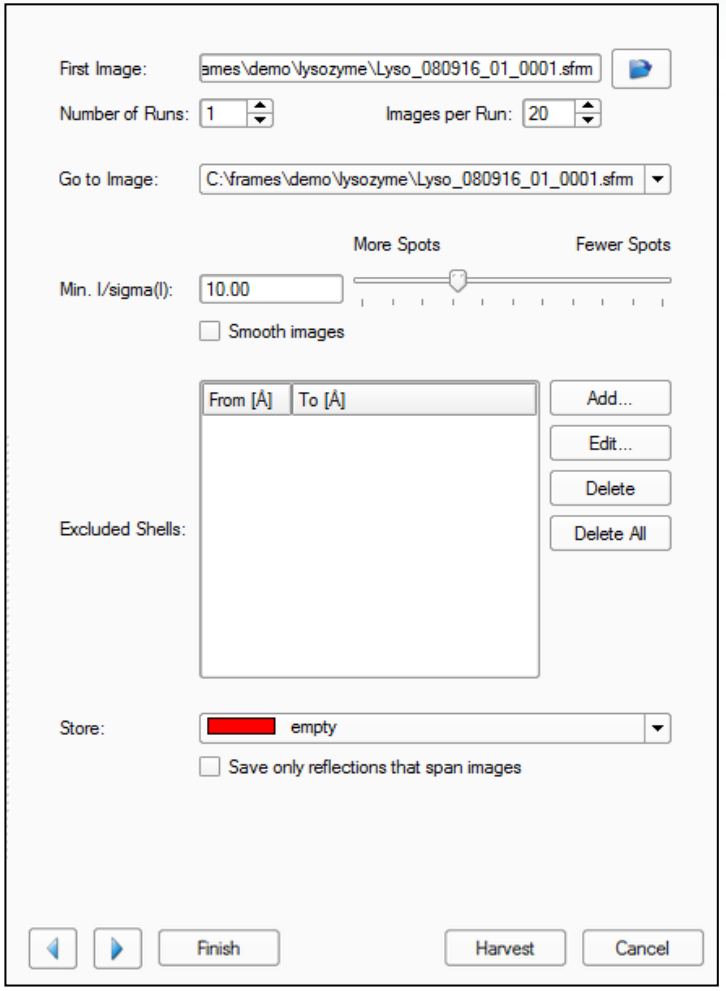
Run # **Frame #**



Determine Unit Cell
Harvest spots

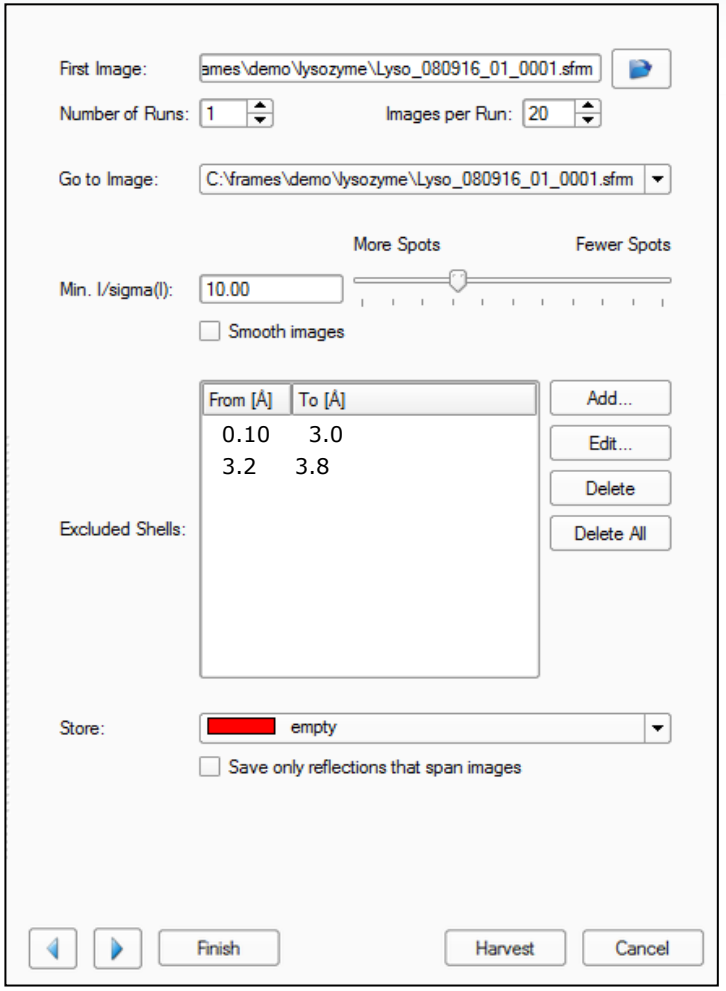
Find reflections for the indexing. Can harvest from anywhere in the run or from more than one run. Can be in different regions of reciprocal space.

- First image can be from anywhere in the data
- Number of runs
  - Will start at the beginning of all runs after run 1
- Go to Image – what image to display
- Min I/sigma(I)
  - Signal to noise cutoff for finding reflections
  - Smooth images reduces image resolution
- Smooth images
  - Reduces the resolution of the images which removes weak reflections and combines split reflections
- Excluded Shells
  - Remove resolution shell from harvesting
- Store
  - Stores reflections by color scheme
  - Save only reflections that span images



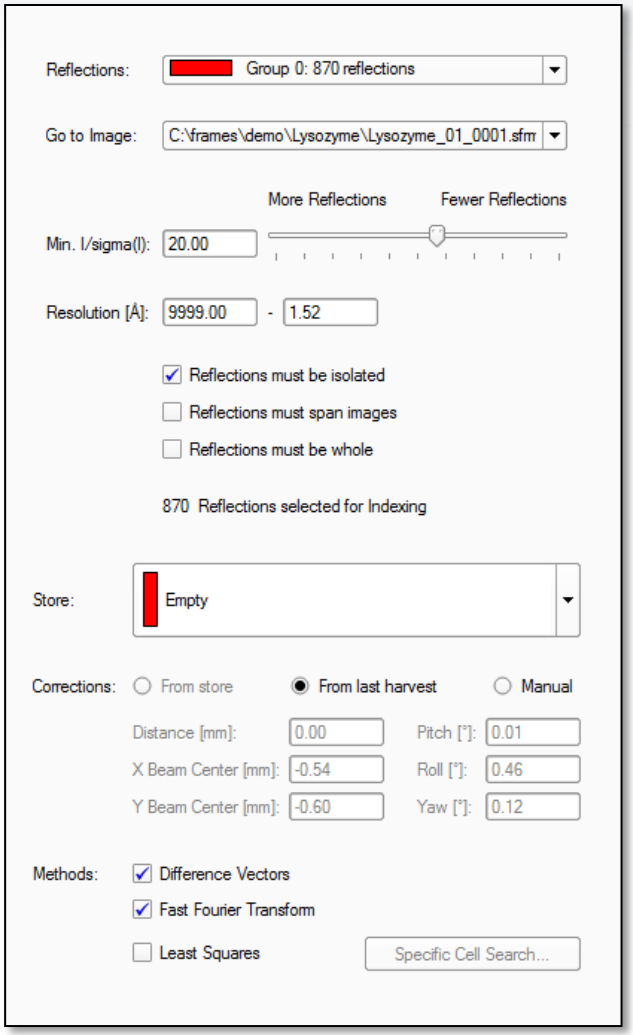
Determine Unit Cell
Navigating through the indexing steps

- Arrows move to the previous or next step
  - Harvest ⟶ Index
- Finish
  - Runs auto mode from current step
- Harvest
  - Performs function and returns to the main menu
- Cancel returns to the main menu



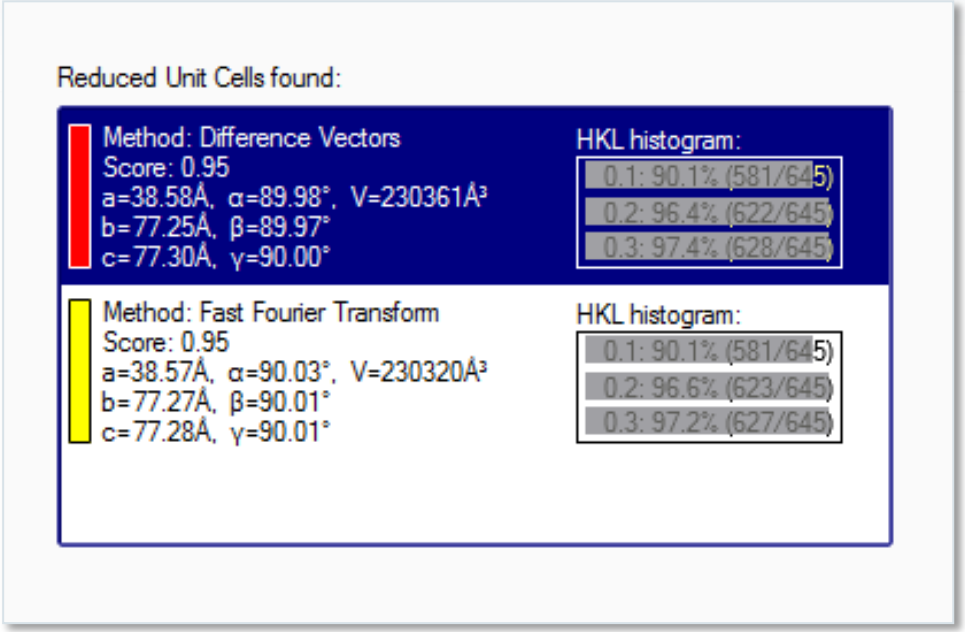
Determine Unit Cell
Indexing

- Reflections shows the number of spots harvested and color code
- Go to Image shows the image displayed
- Min I/sigma(I)
  - Signal to noise cutoff for finding reflections
- Resolution
- Filters
- Corrections
  - Offsets from the frame headers
  - Clicking manual allows them to be edited
- Methods
  - Difference Vector
  - Fast Fourier Transform
  - Least Squares (Cell_now)



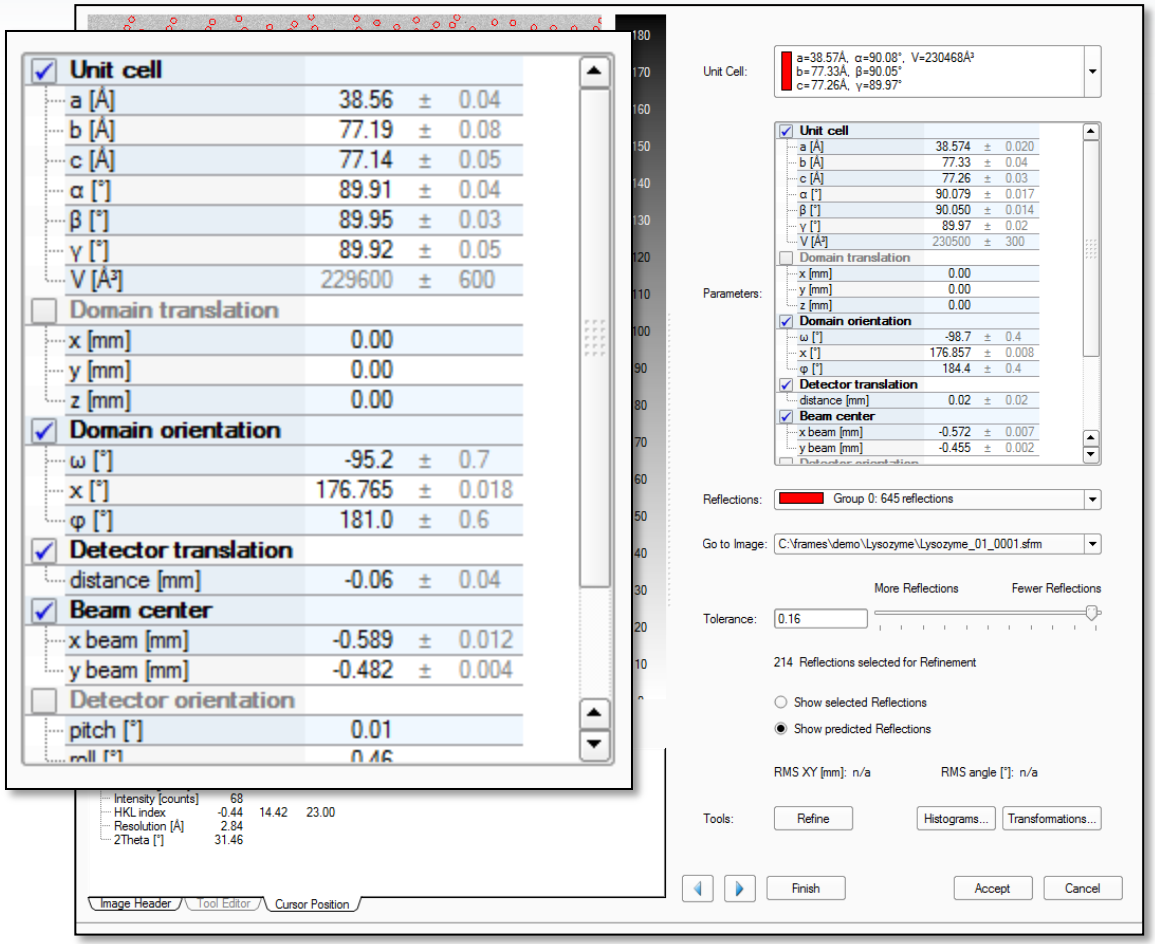
Determine Unit Cell
Indexing

- Indexing results gives the unit cell found from all methods selected
- You can choose any cell to continue
- HKL histrogram
  - Percentage of harvested reflections that match the predicted spots based on the orientation matrix



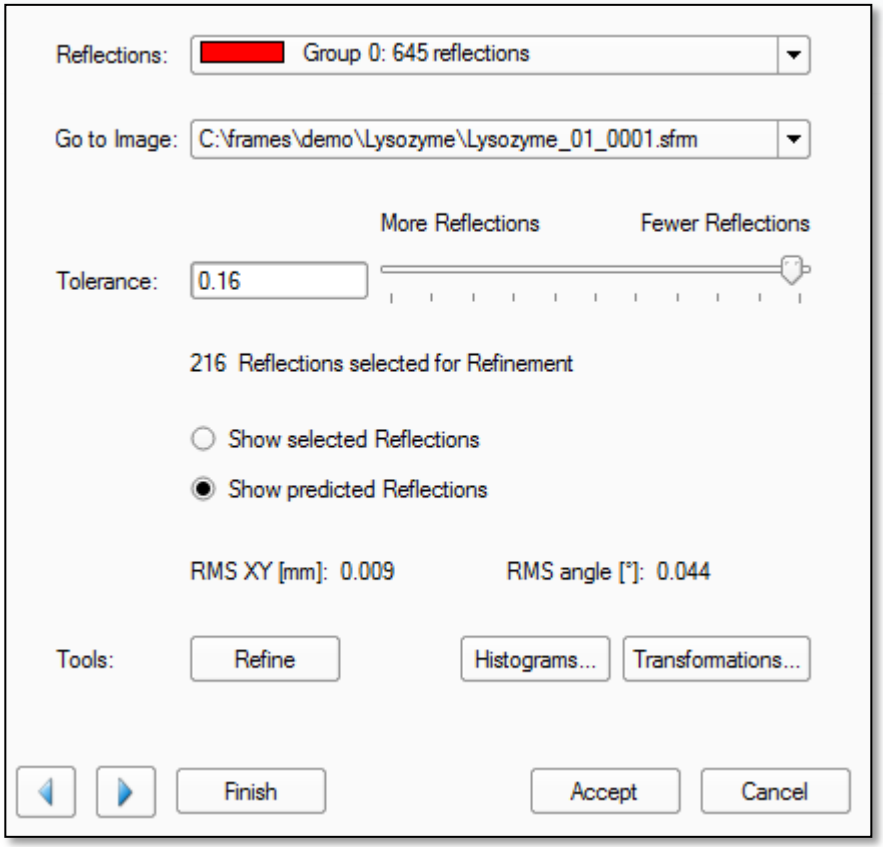
Determine Unit Cell
LS refinement reduced triclinic cell

- Refine the unit cell constants and hardware parameters to improve the orientation matrix
- Makes it easier to search for higher symmetry
- If you only index from a small angular range, the program only allows you to refine the Domain orientation, beam center and crystal to detector distance (DX)



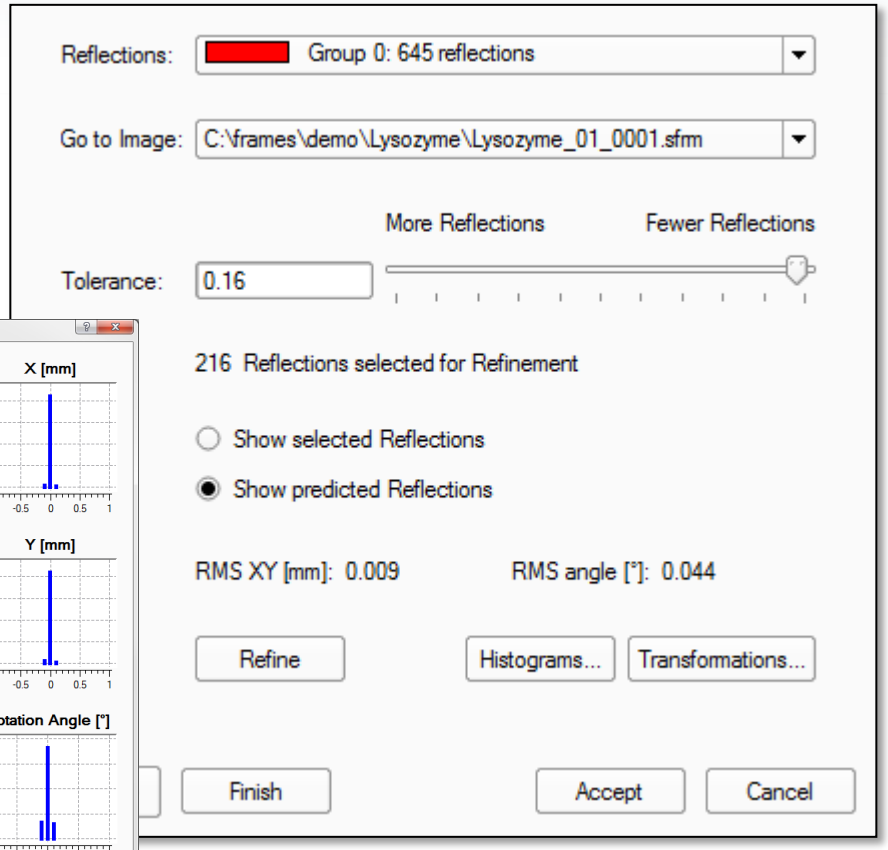
Determine Unit Cell
LS refinement reduced triclinic cell

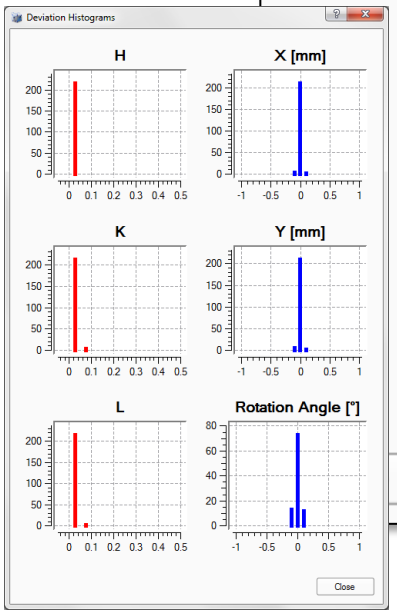
- Tolerance
  - How far the position of the observed reflection differs from the predicted
  - Moving the slider bar increases the accepted tolerance and add more reflections
  - Start with suggested reflection set and then add more as the cell improves
- RMS XY, RMS angle
  - How far off are the predicted spots
  - RMS XY should be < 0.3
  - RMS angle should be < 1 deg

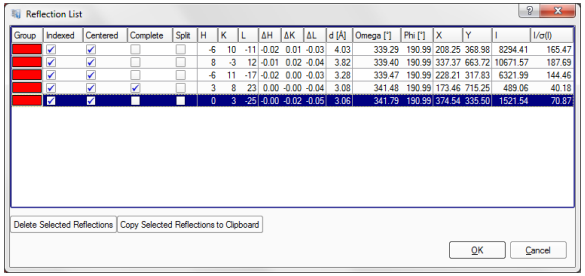


Determine Unit Cell
LS refinement reduced triclinic cell

- Histograms
  - Error distribution for all of the reflections
  - Shows how well the observed spot matches the predicted indices and detector position
  - Can display and remove reflections by clicking on the error bar

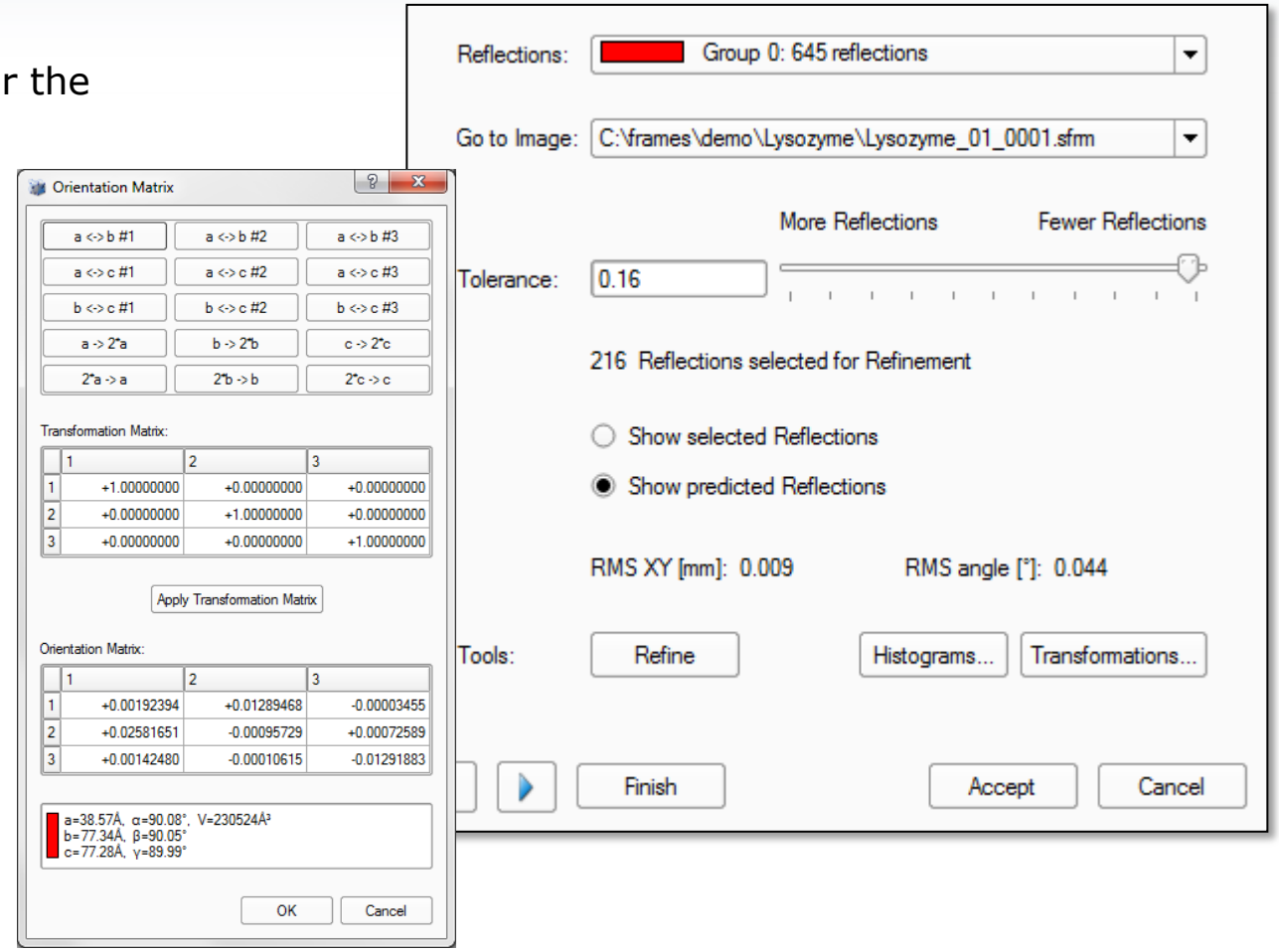






Determine Unit Cell
LS refinement reduced triclinic cell

- Transformations
  - Allows you to transfer the orientation matrix



Determine Unit Cell
Bravais search

- Higher symmetry search
- Transforms the reduced triclinic cell into all possible choices
- Rates them on how closely they match the Laue constraints for that point group symmetry and lists the FOM from 0 ⟹ 1, the higher value being the better score
- Cells shown in green are those the program thinks are acceptable, red not realistic
- Any cell with a FOM 0.3 or higher is shown in green, anything lower shown in red
- You can select any cell shown and the program will transform the reduce cell to that setting

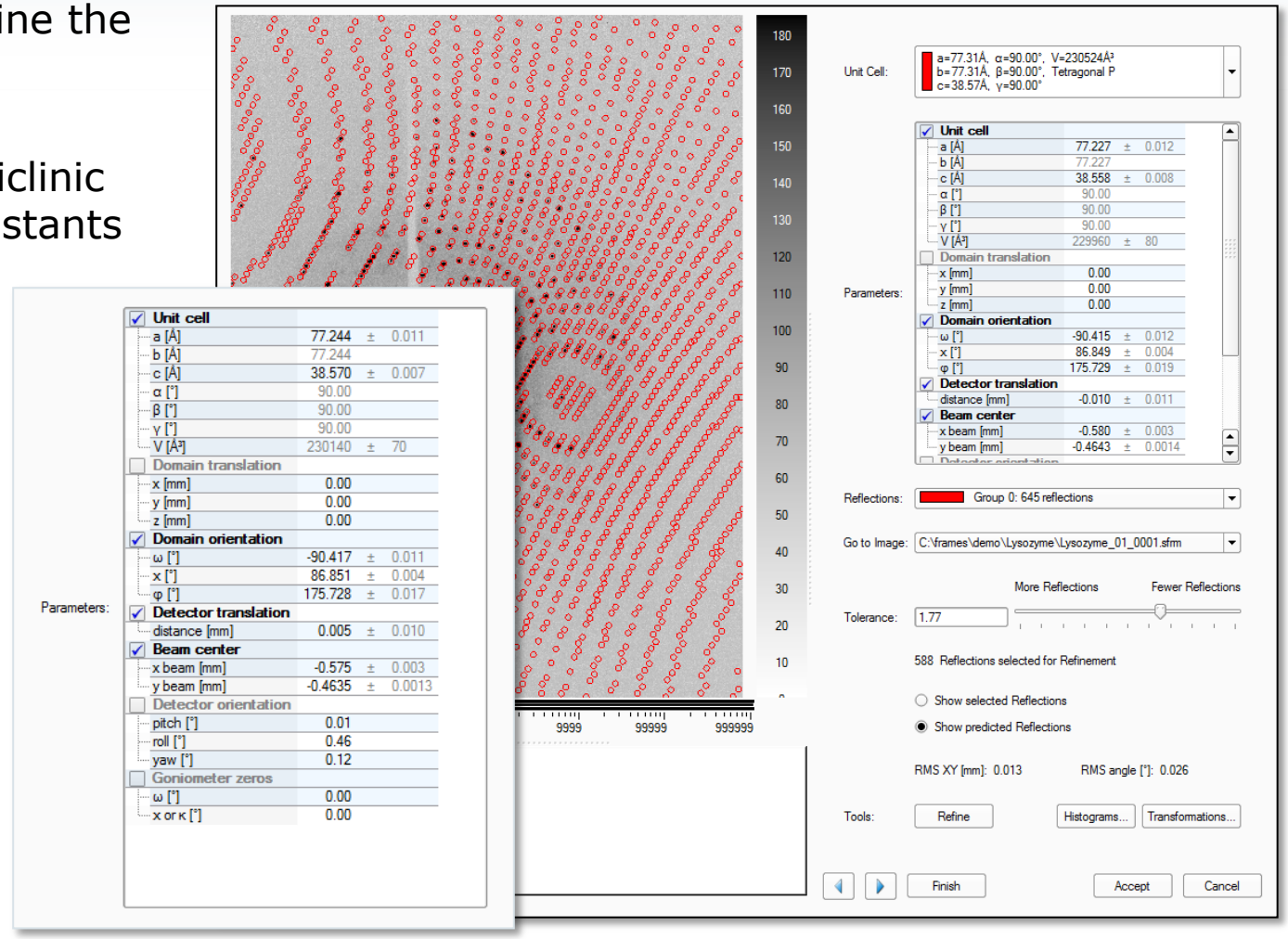
Initial Unit Cell:

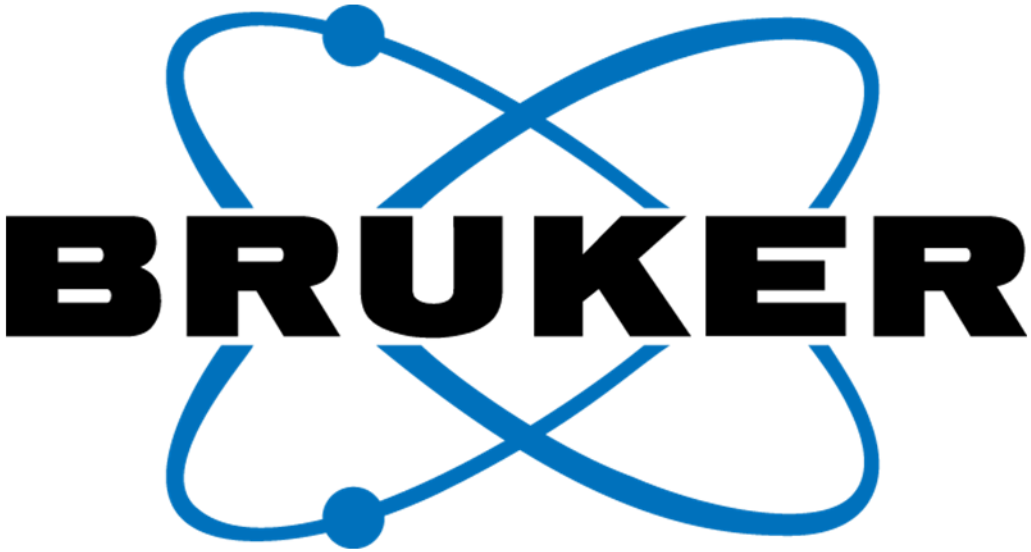
a=38.57Å, α=90.08°, V=230468Å³
b=77.33Å, β=90.05°
c=77.26Å, γ=89.97°

Bravais Lattice	FOM	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
Cubic F	0.01	115.84	115.86	115.95	96.39	96.29	141.10
Cubic I	0.01	86.44	86.33	109.24	50.80	50.78	78.57
Cubic P	0.01	38.57	77.26	77.33	89.92	89.97	89.95
Hexagonal P	0.01	77.26	77.33	38.57	90.03	89.95	90.08
Rhombohedral R	0.01	86.33	86.40	116.03	116.66	63.38	101.54
Tetragonal I	0.02	109.24	115.95	38.57	70.63	90.01	90.05
Tetragonal P	0.79	77.26	77.33	38.57	89.97	89.95	89.92
Orthorhombic F	0.02	38.57	159.24	159.37	86.58	103.97	103.97
Orthorhombic I	0.02	38.57	109.24	115.95	89.95	70.63	89.99
Orthorhombic C	0.82	109.24	109.39	38.57	90.06	89.99	89.95
Orthorhombic P	0.78	38.57	77.26	77.33	89.92	89.97	89.95
Monoclinic C	0.85	109.39	109.24	38.57	90.01	90.06	90.05
Monoclinic P	0.82	77.26	38.57	77.33	90.03	90.08	89.95
Triclinic P	1.00	38.57	77.26	77.33	89.92	89.97	89.95

Determine Unit Cell
LS refinement of the final unit cell

- Final step is to refine the cell with the Laue constrains applied
- For the reduced triclinic cell all the cell constants are refined





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