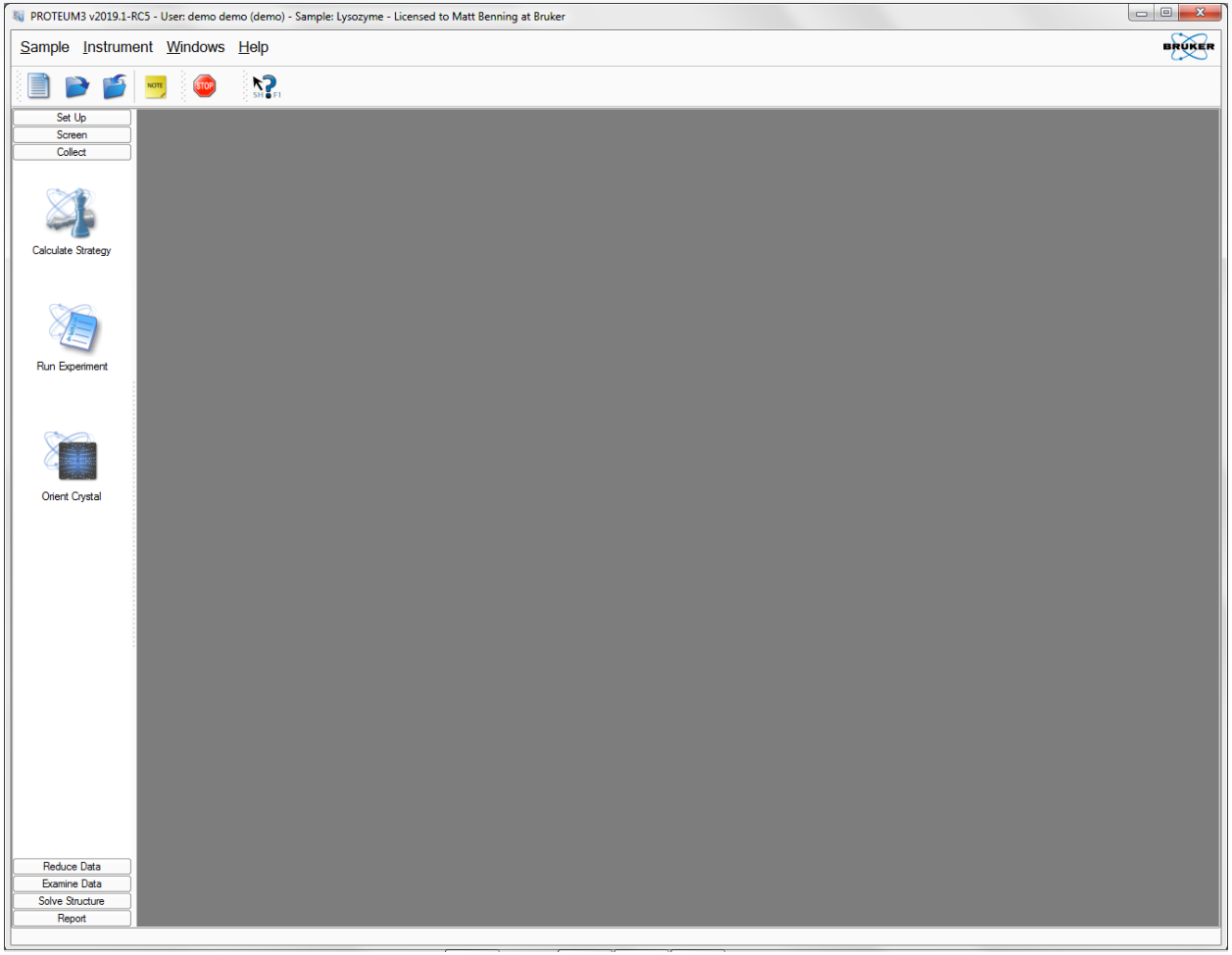
Kappa Geometry and Data Collection Strategy

Data collection strategy
Collect

- Collect strategy
  - Does not need an predetermine hardware profile
  - Hardware configuration and mapping is determined using OpCo
  - Finds strategy from requested maximum resolution and unit cell
  - Special strategies such as Friedel's pairs in the same image and inverse beam are implemented
  - Redundancy is 90% median multiplicity



Data collection strategy

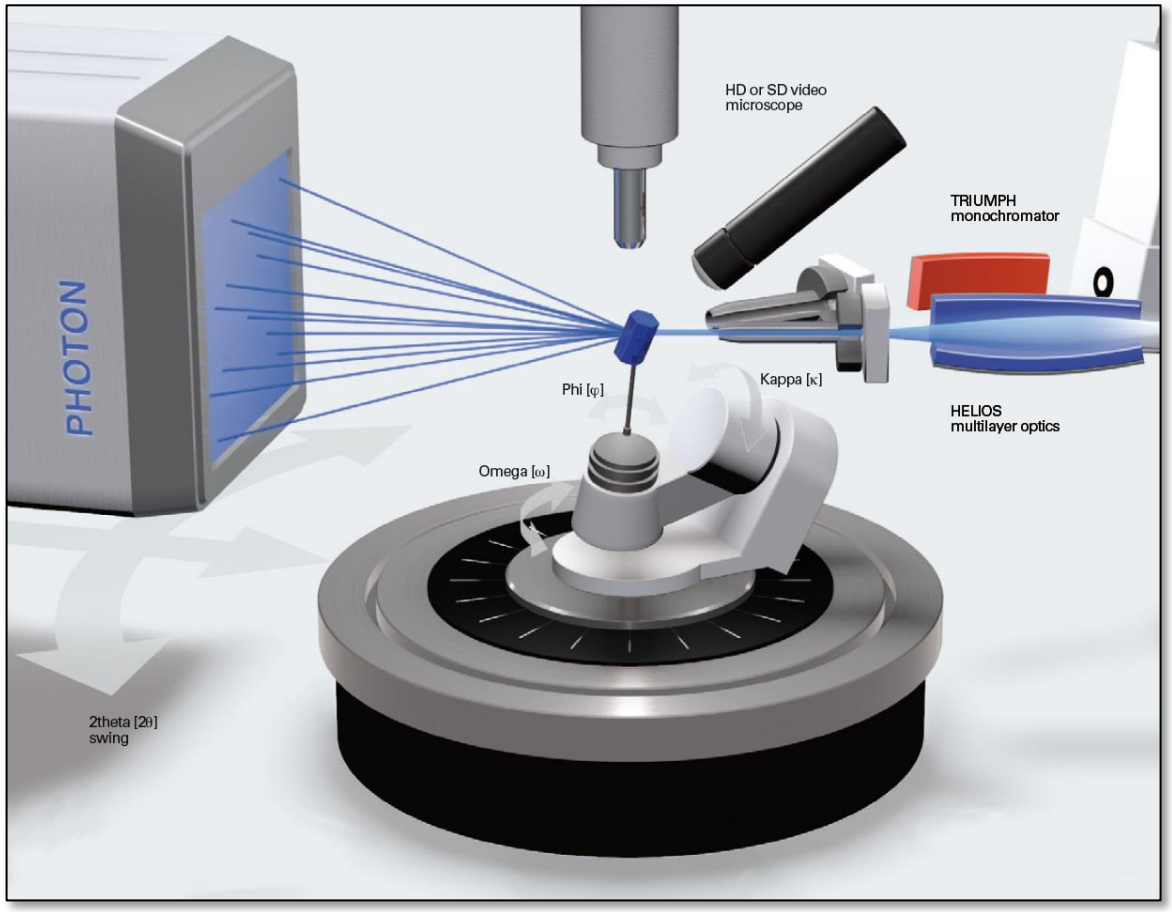
Why do you need a data collection strategy

- Exploit the benefits of the kappa geometry
- Collect the best data for your purpose
- Minimize data collection time

What do you need

- Unit cell
- Resolution limit of your sample
- Experimental parameters
  - Rotation angle
  - Exposure time
  - Detector distance
- Purpose of the experiment
  - Native data set
  - SAD phasing

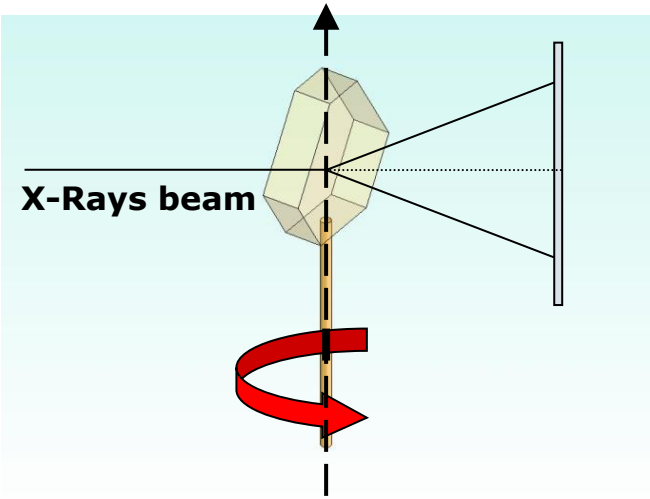
The Kappa Geometry



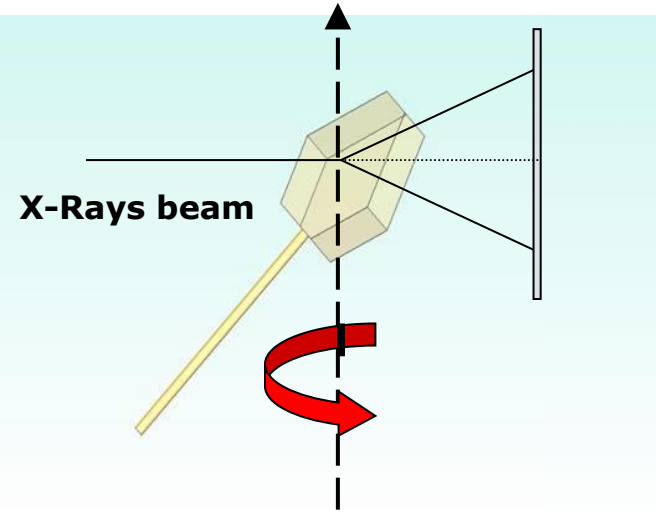
The Kappa Goniometer

Advantages

- High random multiplicity data can be collected
- Data is collected with different crystal orientations
- Better model for scaling data together
- Easier to handle flash-cooling crystals



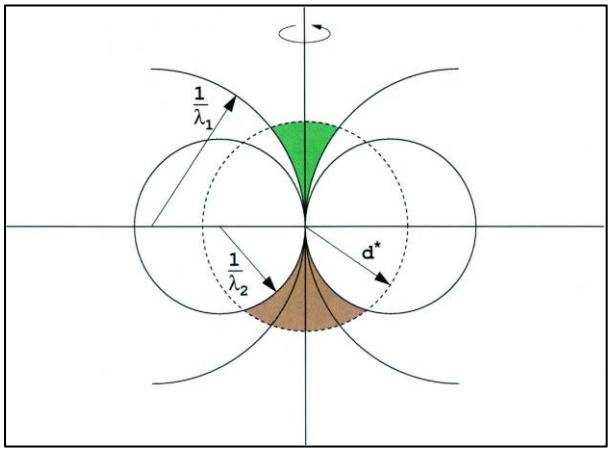
Serendipitous multiplicity

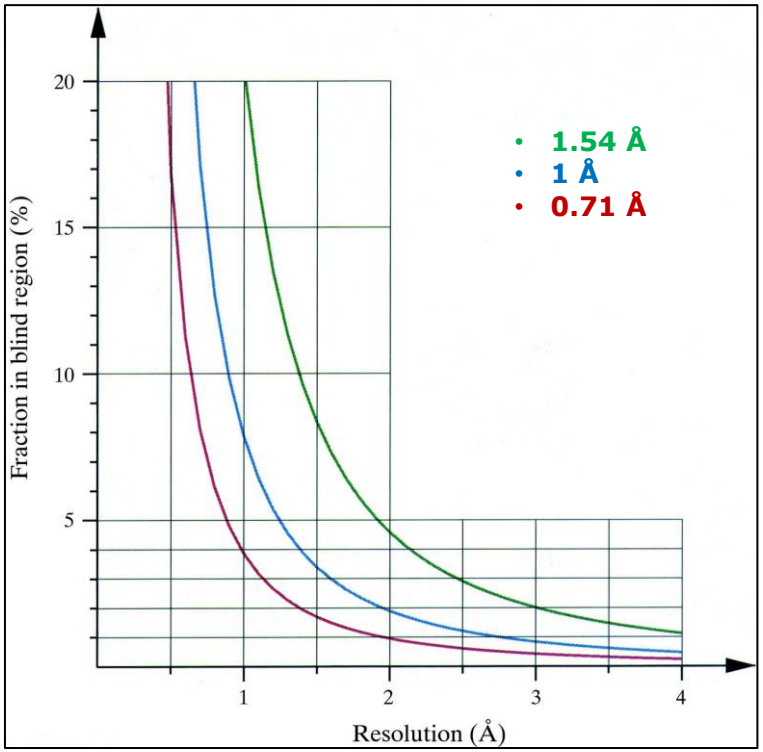


True multiplicity

The Kappa Goniometer
Blind region

- Using only a fixed rotation axis creates a blind region during data collection
- More significant at higher resolution, longer wavelengths and lower symmetry





*Dauter, 1999

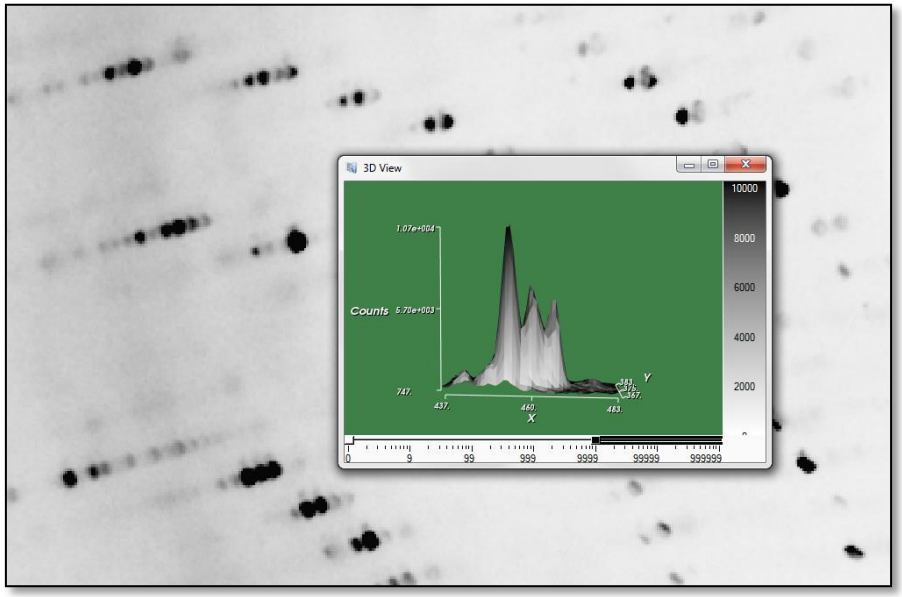
The Kappa Goniometer
Crystal orientation

- Can limit spot overlap involving a long axis by positioning the axis parallel to the rotation axis
  - If the long axis is parallel to the x-ray beam, it's difficult to separate the Bragg reflections
- Optimize data colleciton
  - For a hexangonal 622 cell, rotation around the c axis requires only 30° of data, but if rotated around a vector in the ab plane 90° are necessary

Data Collection
Resolving reflections

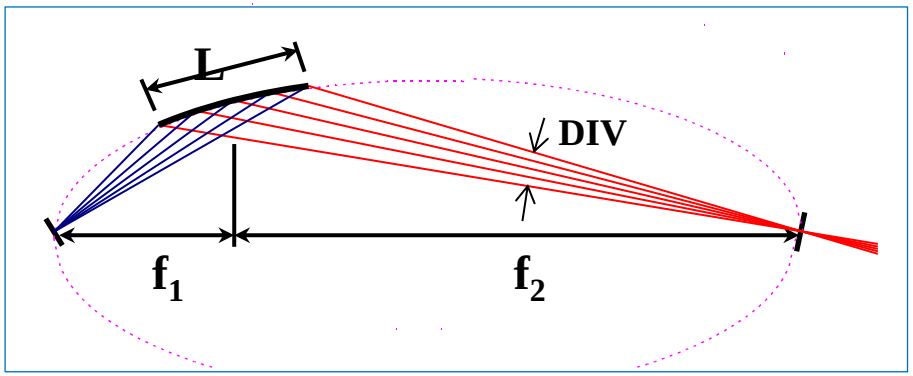
To improve the separation between the reflections:

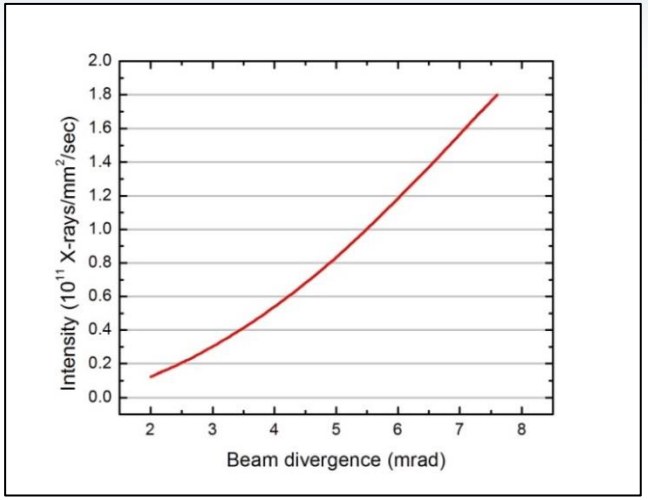
- Increase the detector to sample distance (DX)
- Collect data with a smaller rotation angle
- Decrease the beam divergence

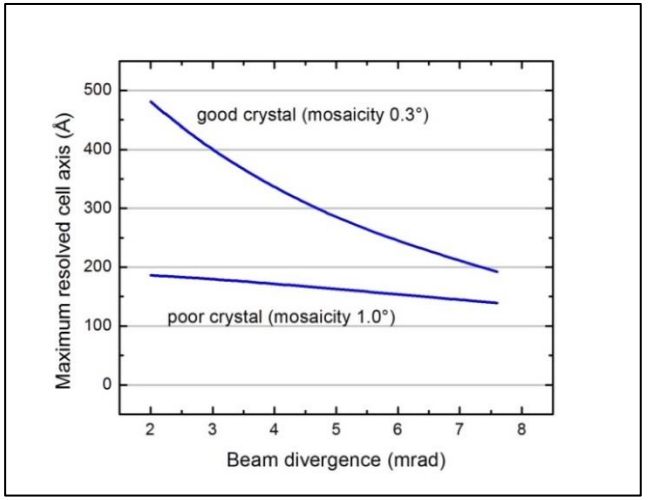


Data Collection
Resolving reflections

- In-house systems use a focusing optic to increase the intensity of the beam
- The divergence or crossfire angle determines the increase in intensity
- However, the larger the divergence the bigger the spots
- To resolve the reflections for larger unit cells, you have to lower the divergence or move the detector away from the sample

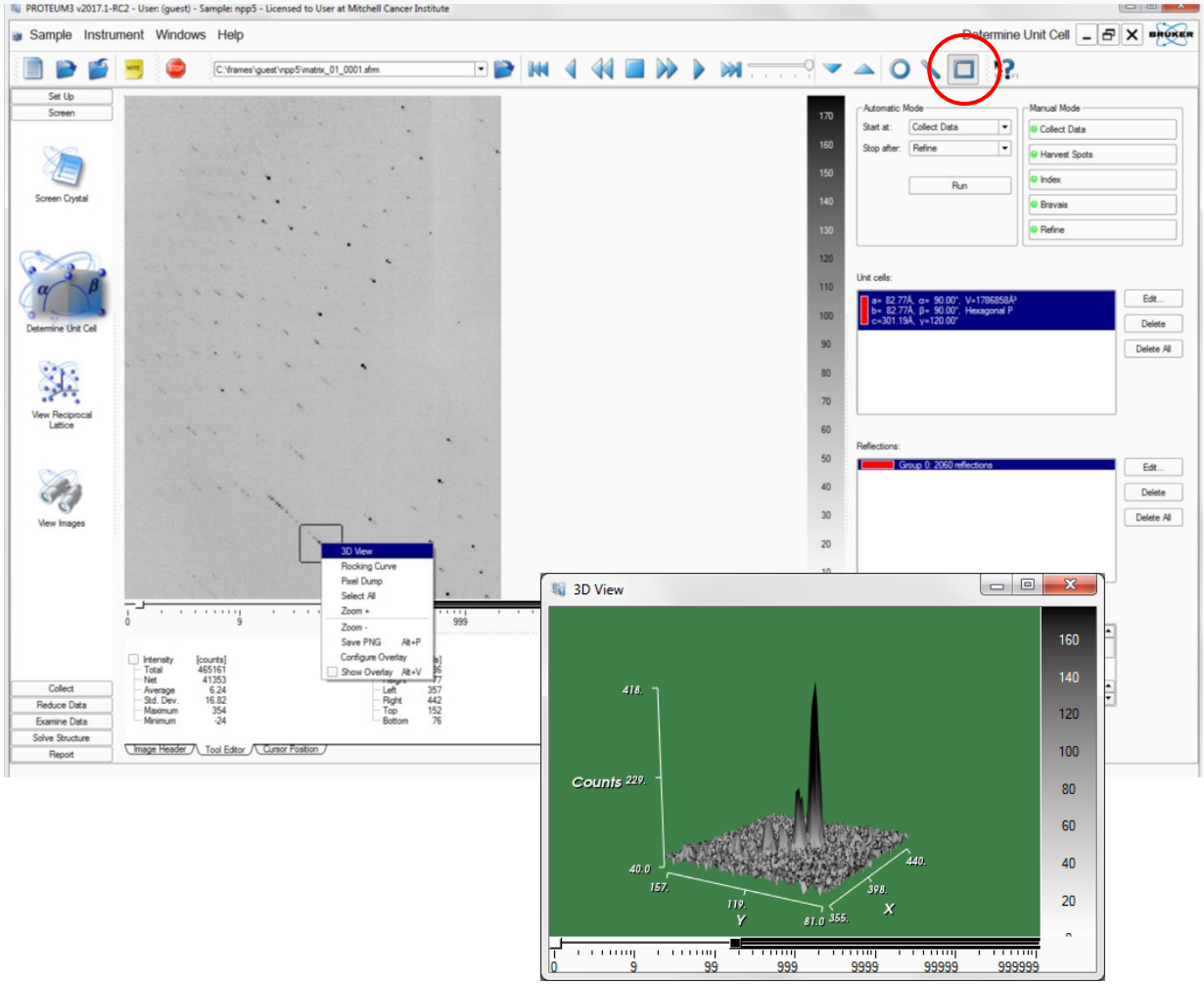






Using the 3D View to Look at Peak Separation

- Select the box cursor in the upper right
- Draw a box around some spots showing the long axis by holding the left mouse button down and extending the box around the spots
- Click the right button and select 3d view, you can rotate the view around by holding down the left mouse button
- This shows the peaks relative to each other and the background. They can be overlapped at the base but not too far up the peaks



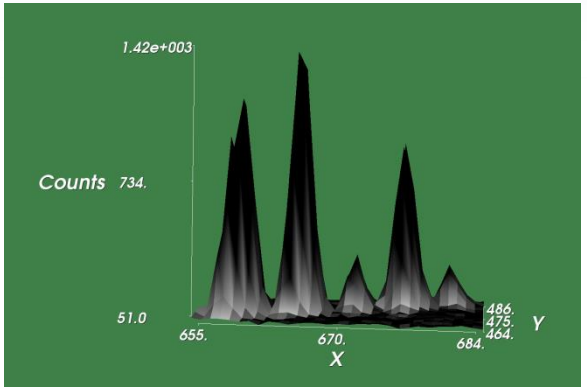
Data Collection
Divergence

- Full divergence is 7.6 mrad

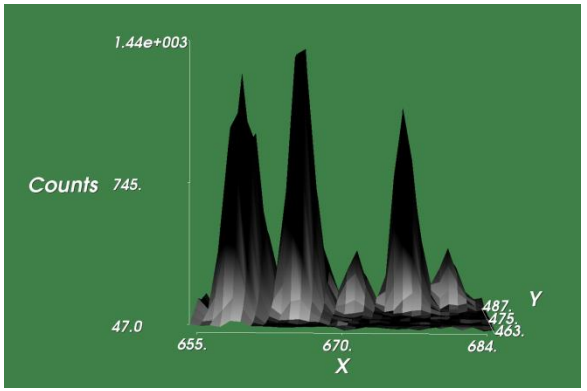
Collimator	Divergence	**Beam size** (μm)	**Relative Intensity**	**Longest Unit cell Axis** (Å)
Full	7.6	70	1.00	~180
0.3 x 17	~7.0	70	0.85	~220
0.3 x 10	~5.0	70	0.48	~300
0.2 x 6	~3.0	70	0.23	~370

Data Collection
Divergence

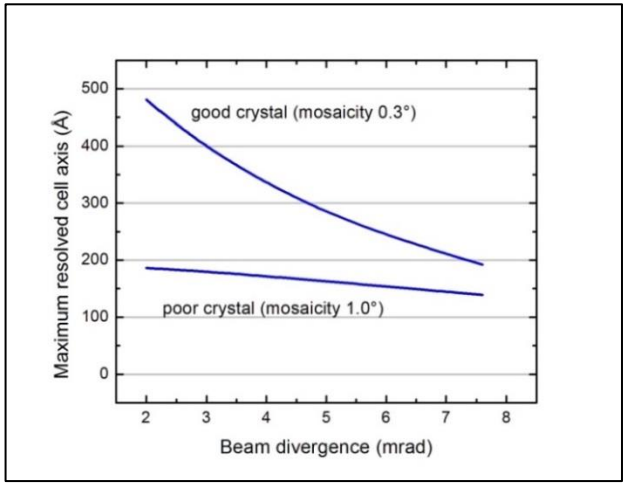
- Lowering divergence reduces the rocking curve of the reflections
- Also lowers the peak intensity
- Some of the peak width is due to the peak intensity so crystals with a high mosaic spread will be more difficult to resolve regardless of the chosen divergence.



0.3 x 10



0.3 x 17



Data Collection Strategy

Information needed

- Unit cell and Laue group
- Maximum resolution
- Mosaicity

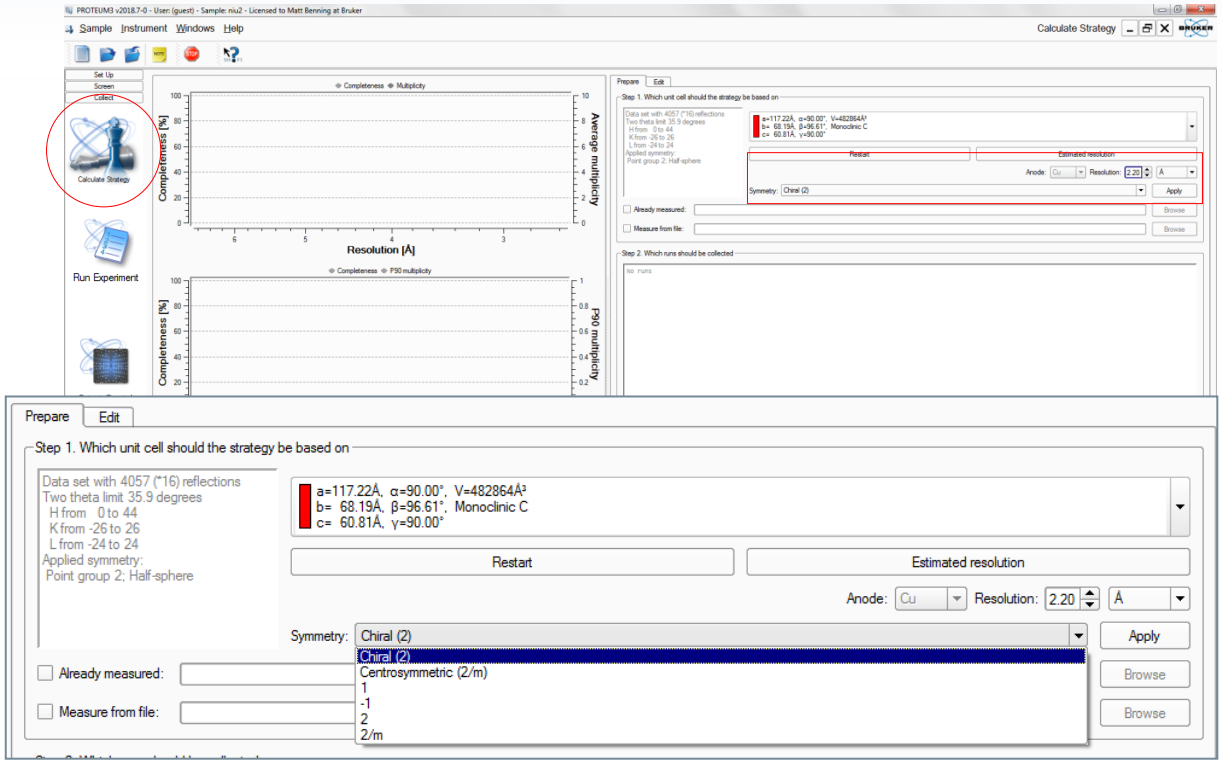
Experimental parameters to be set

- Sample to detector distance
- Exposure time
- Scan width

Parameters against which the runs are optimized

- Multiplicity
- Completeness
- Total exposure time

Data Collection Strategy

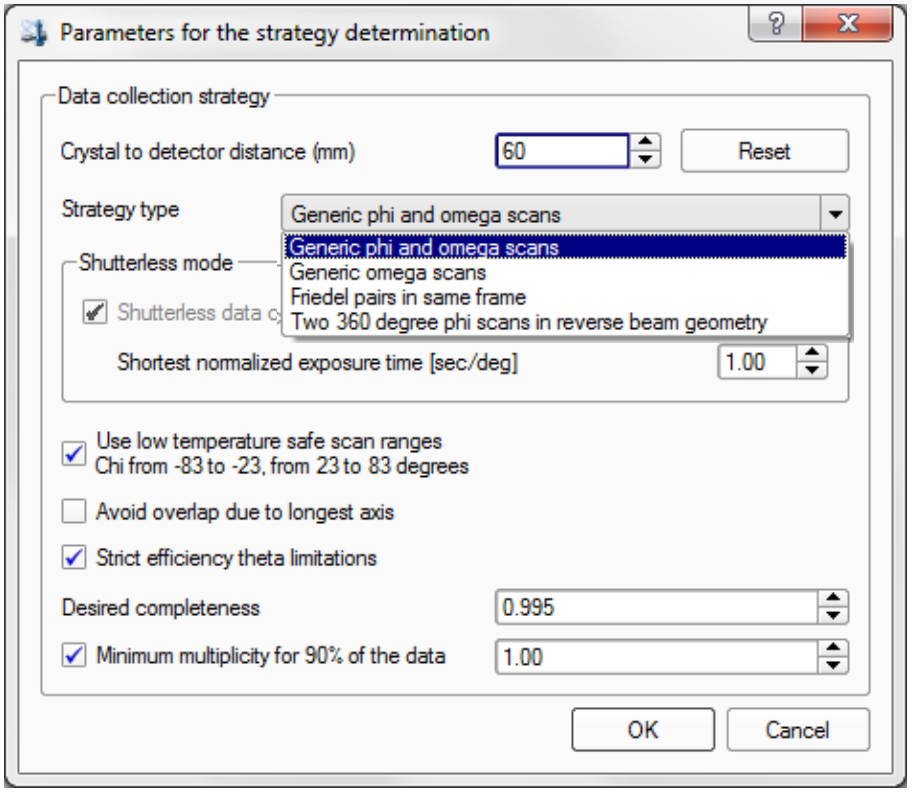


Multiple crystal data collection

- Already measured – Add unique data to incomplete data set
- Measure from file – Collect identical regions of reciprocal space

- To open the strategy window, click on the Data collection Strategy icon (Queen) under the collect menu
- Input the maximum resolution
- Assign the point group symmetry
  - The program assigns the lowest symmetry for the crystal
  - To keep I |HKL| and I |-H-K-L| separate, use the Chiral or non-centrosymmetric point group (1, 2, 222, 4, 422, 6, 622, 23, 423)

Data Collection Strategy
Determine strategy



Strategy type
- Generic omega & Phi scans
- Generic omega
- Friedel pairs in the same image
- Two 360° phi scan in reverse beam

Shutterless mode
- No dead time between frames
- Shortest exposure time

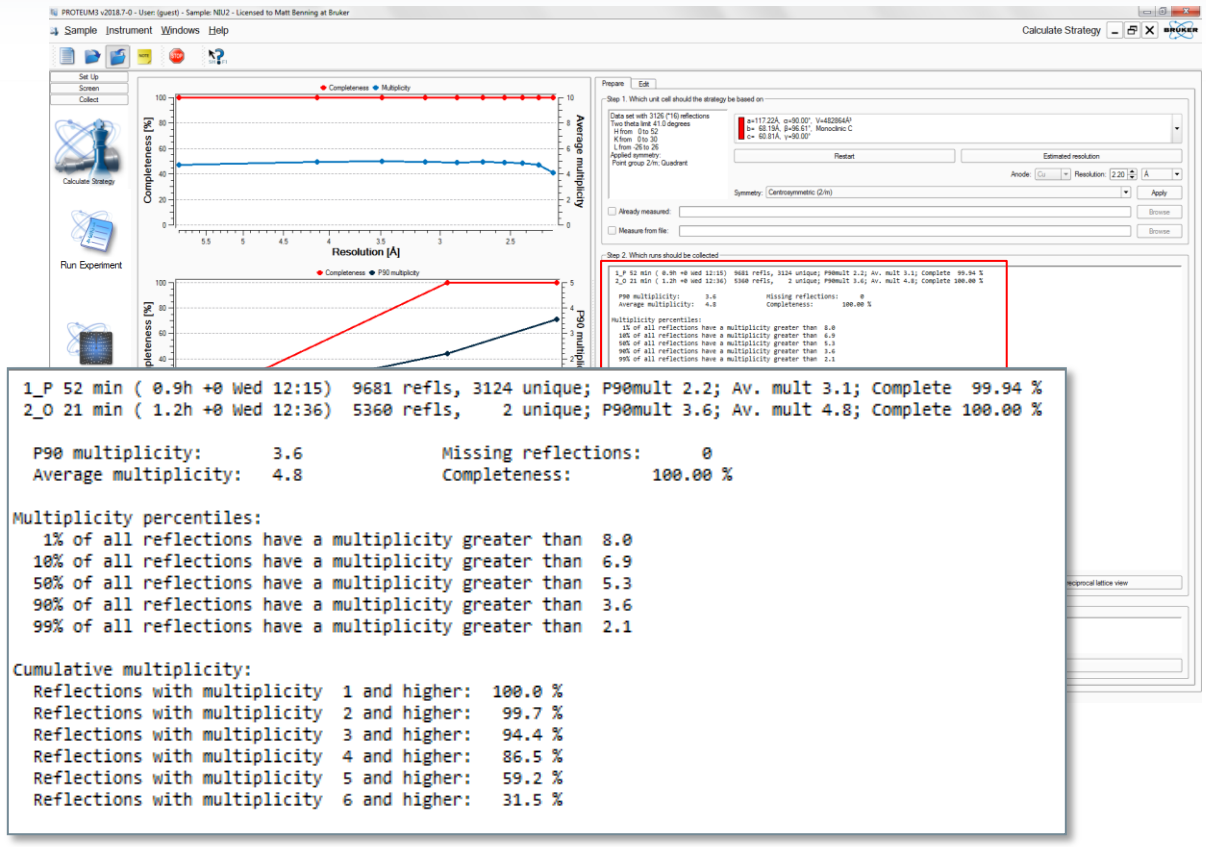
Low temp safe scan
- Keeps goniometer head from sitting directly below LT during data collection

Avoid overlap due to long axis
- Aligns long axis along the rotation axis

Strict efficiency theta limitation
- Limits 2Theta angle to max resolution

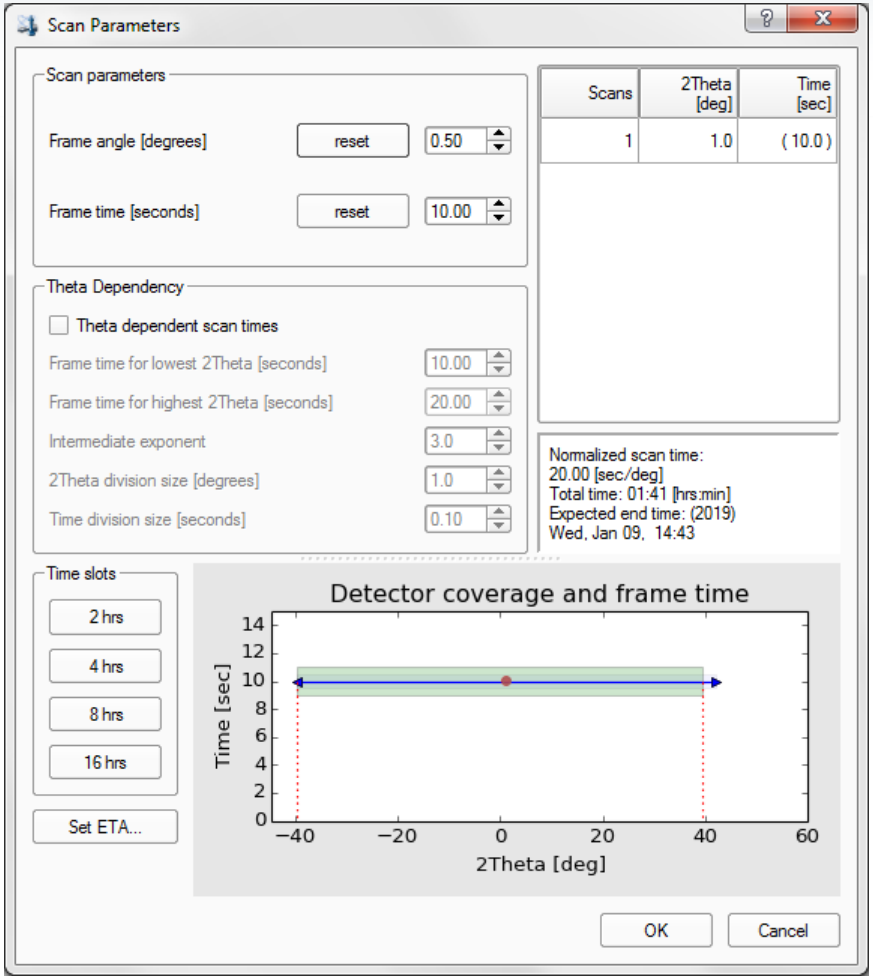
Minimum multiplicity for 90%
- Typically lower angle data will have higher multiplicity, this assures that the higher angle data will be similar

Data Collection Strategy
Determine strategy



- The display window shows the extent of each run plus the completeness and multiplicity
- This information is displayed in graphical form on the left side
- There is no information concerning the time for each scan because we haven't input the exposure time yet, that's next
- Scan listed in degrees

Data Collection Strategy
Select scan parameters



Frame angle

- Rotation angle for each frame, how finely you wish to slice

Frame time

- Exposure time for each frame

Shutterless scans

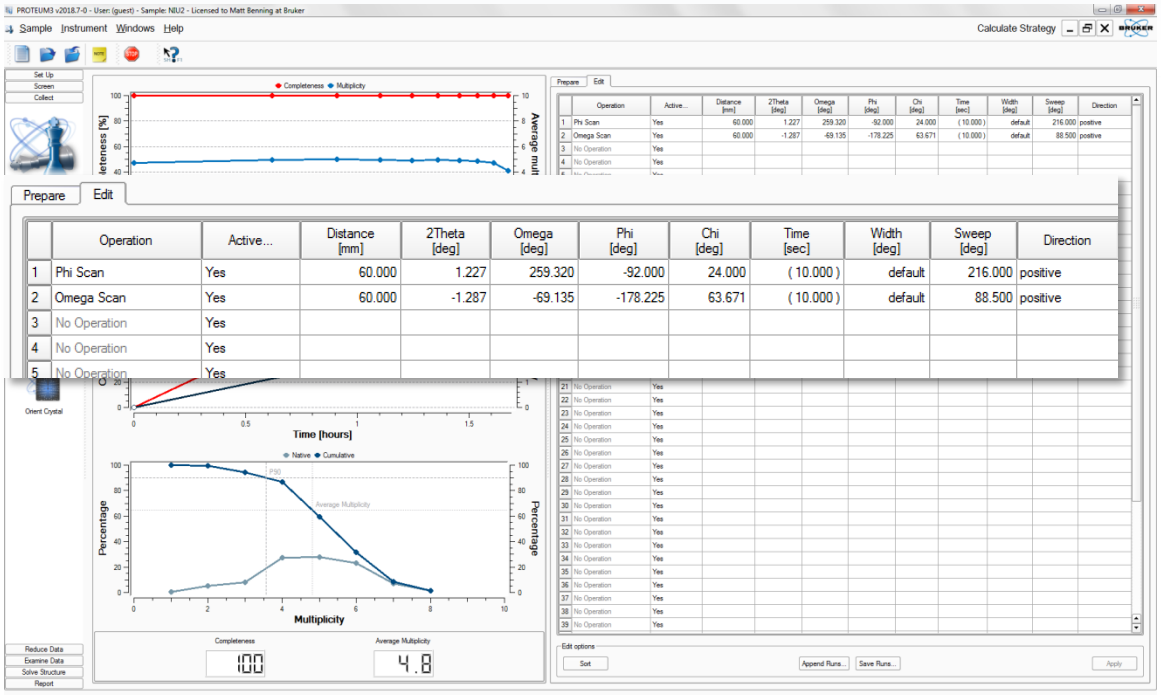
- Removes frame to frame dead time from time calculation

At the bottom is the time/deg and the expected end time for the experiment

- Set time allows you input the actual time you want the experiment to be finished
- The program does this by adjusting the frame time

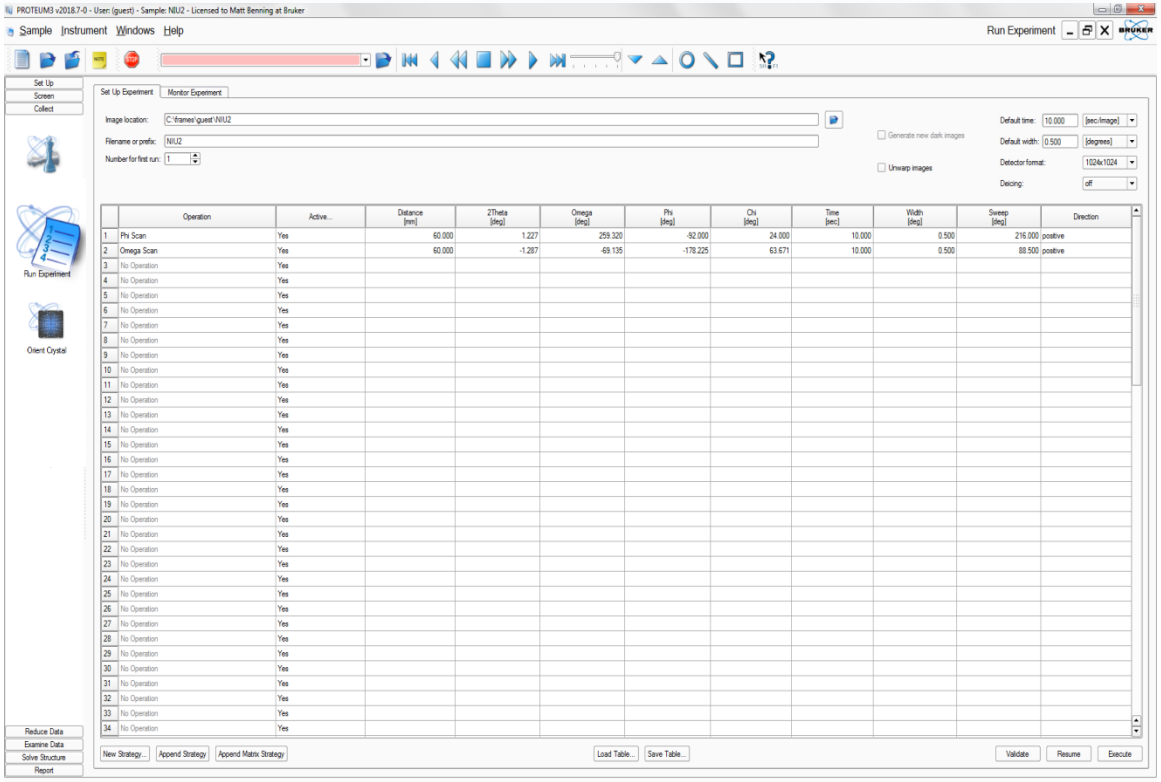
Data Collection Strategy
Edit tab

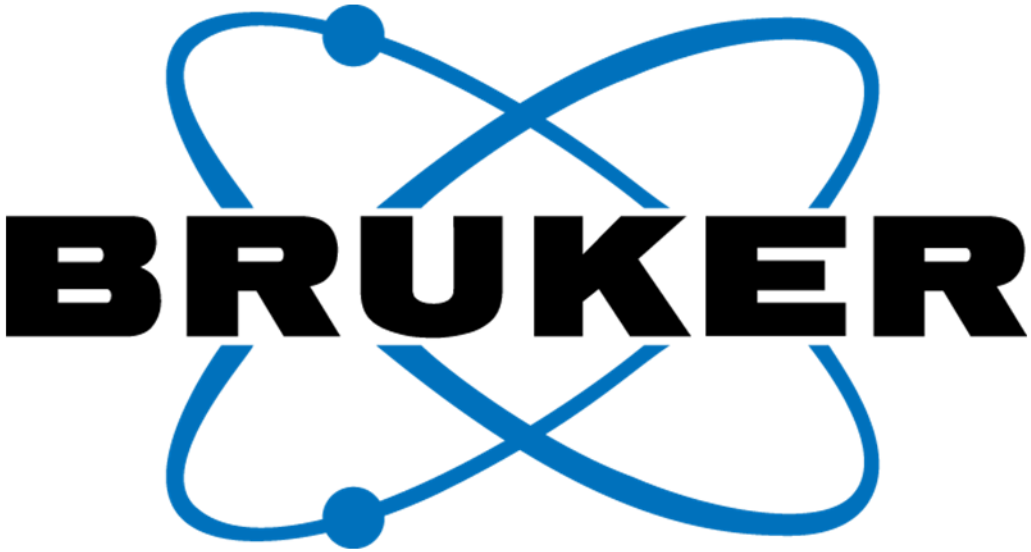
- The edit tab shows the runs in the strategy
- These can be edited just as in the experiment plugin
- Runs can be added or removed
- Once a change has been made, the apply button in the lower right corner becomes active
- If any of the changes is not allowed based on the hardware configuration, a warning message will appear



Data Collection Strategy
Creating an experiment

- To open the experiment window, click the Experiment icon under the Collect menu
- To load the strategy, click the Append Strategy button
- The runs can be edited by clicking once in the box to replace the whole value or twice to edit
- Hitting the execute button will start the experiment





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