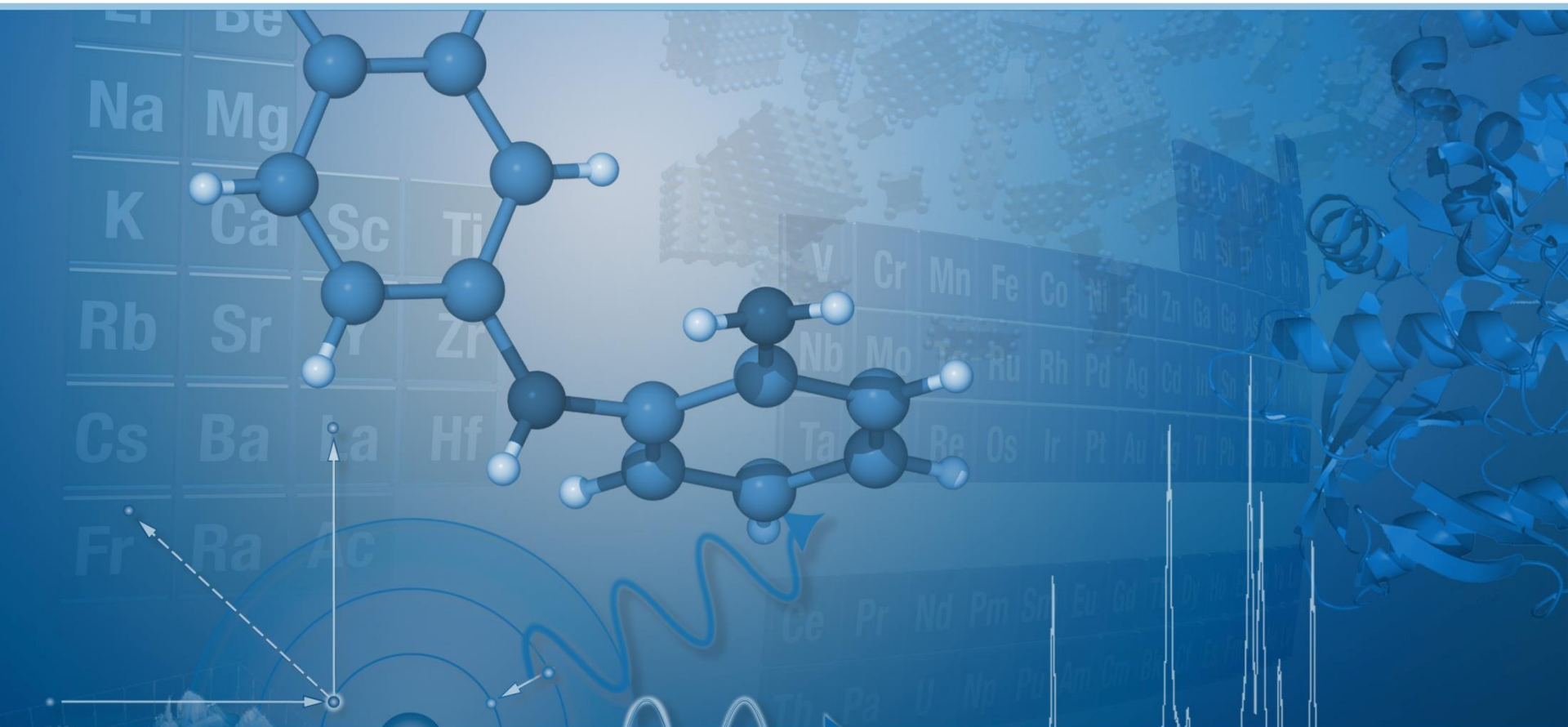


Kappa Geometry and Data Collection Strategy

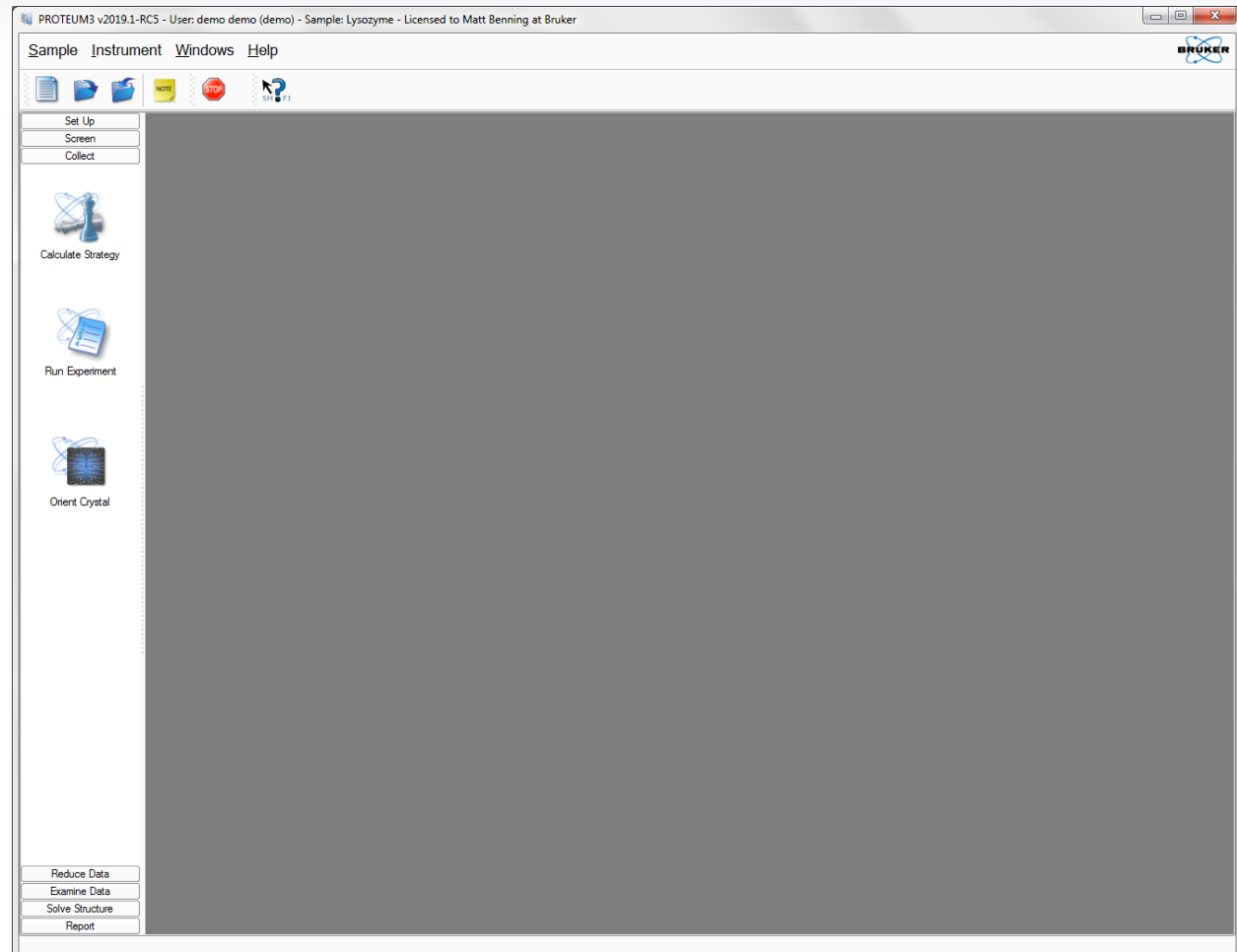


Data collection strategy

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

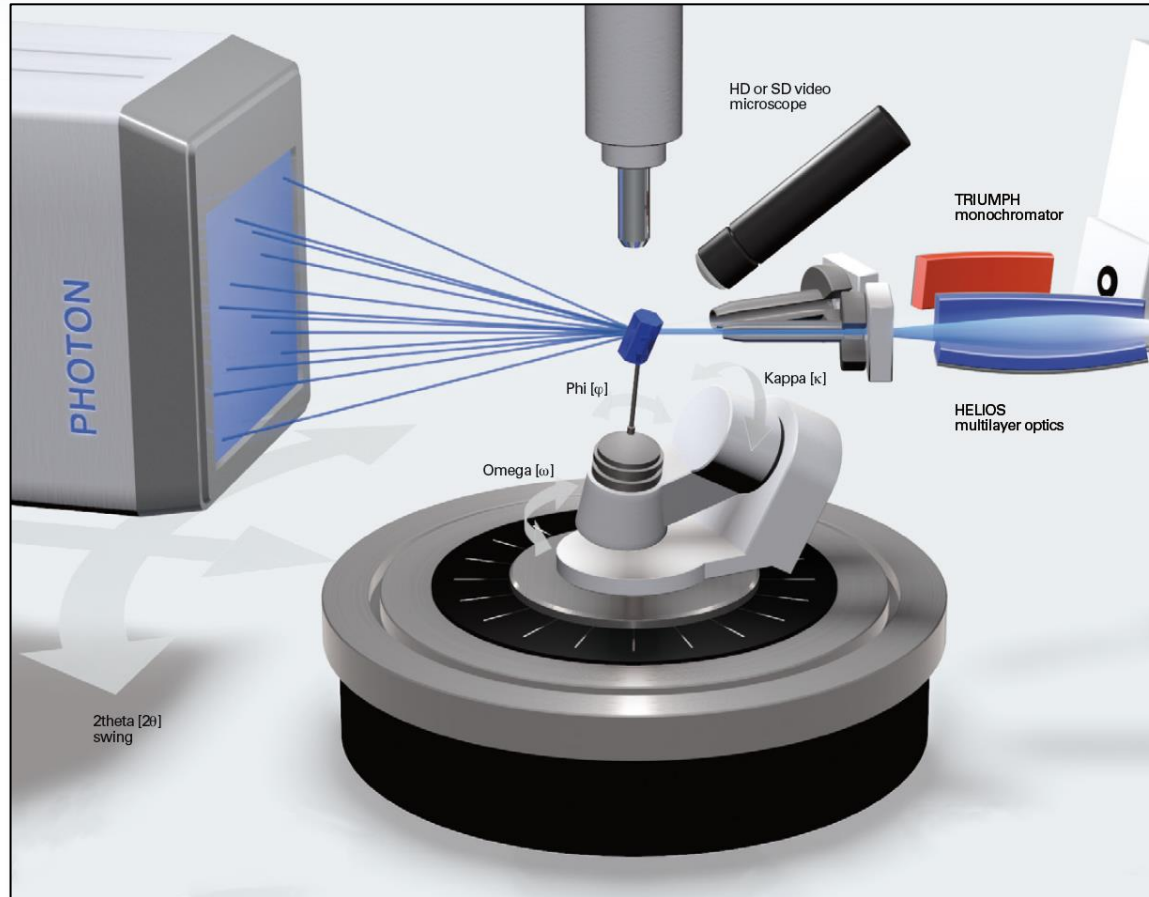
Why do you need a data collection strategy

- 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

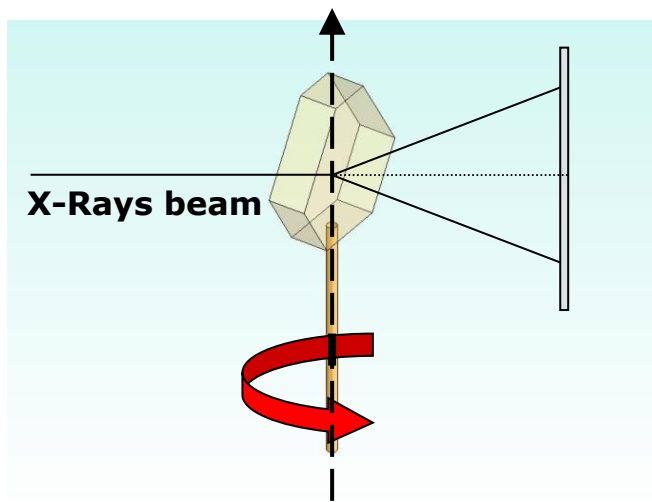
The Kappa Geometry



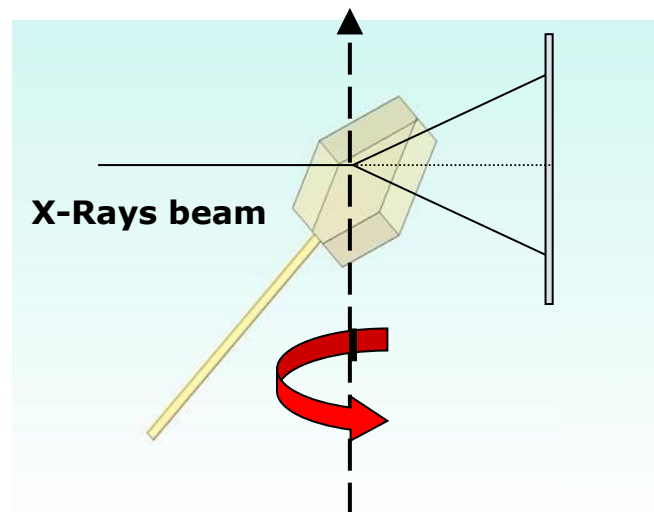
The Kappa Goniometer

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

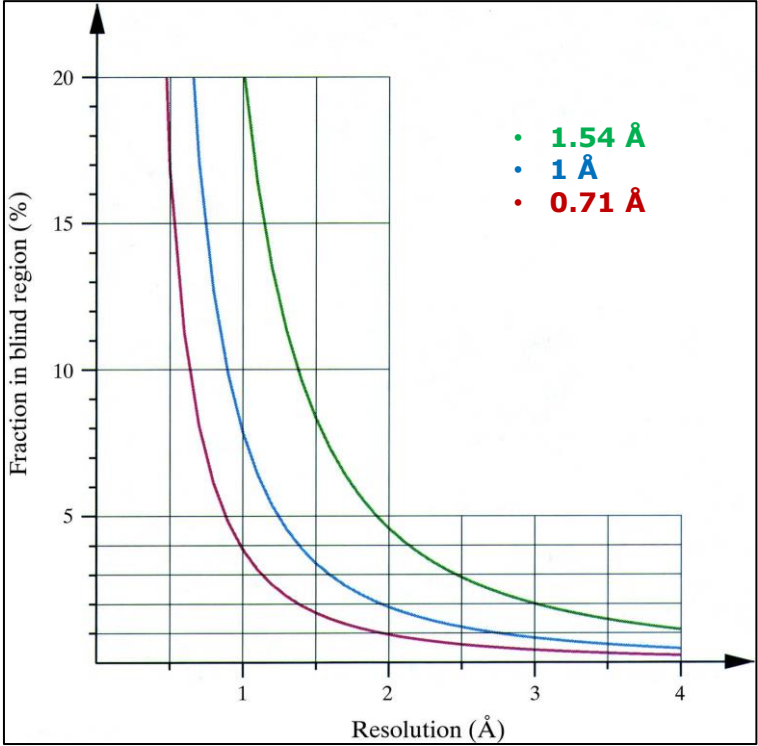
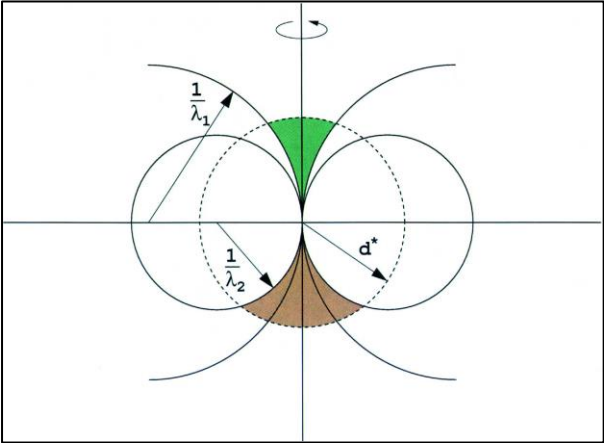


True multiplicity

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 collection
 - For a hexagonal 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

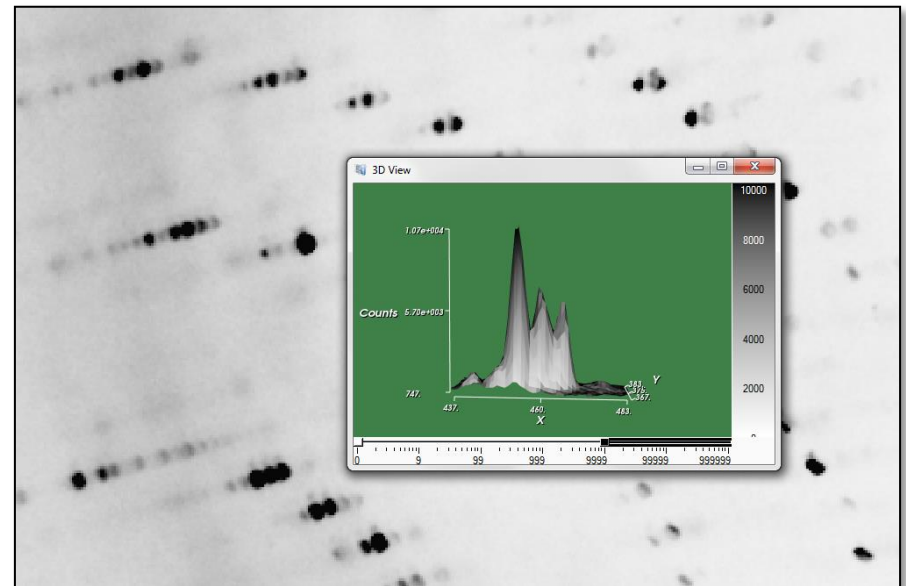
Data Collection

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

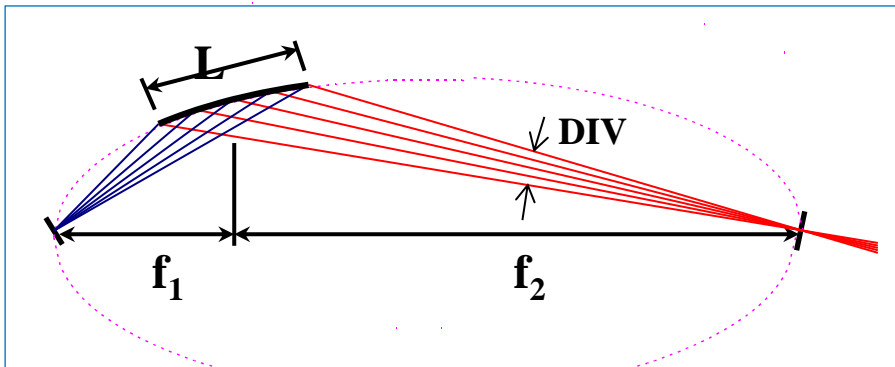
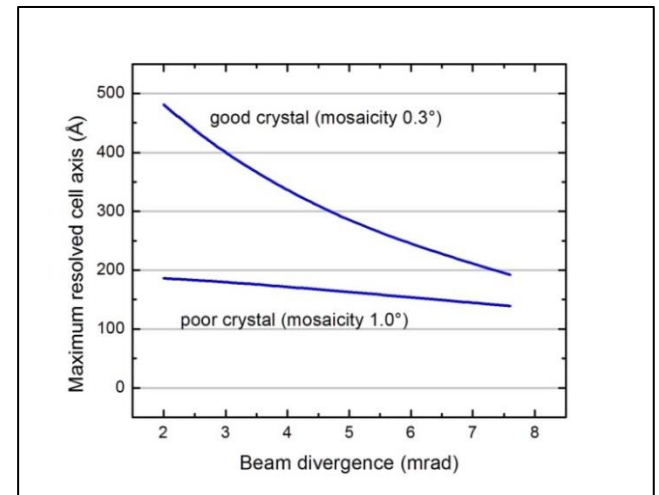
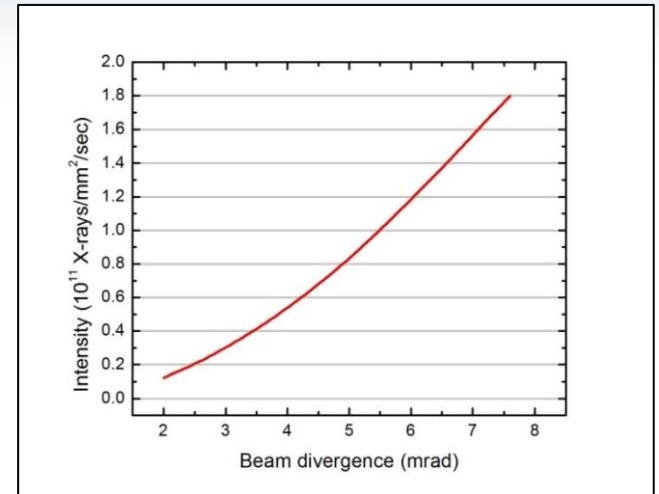


Data Collection

Resolving reflections



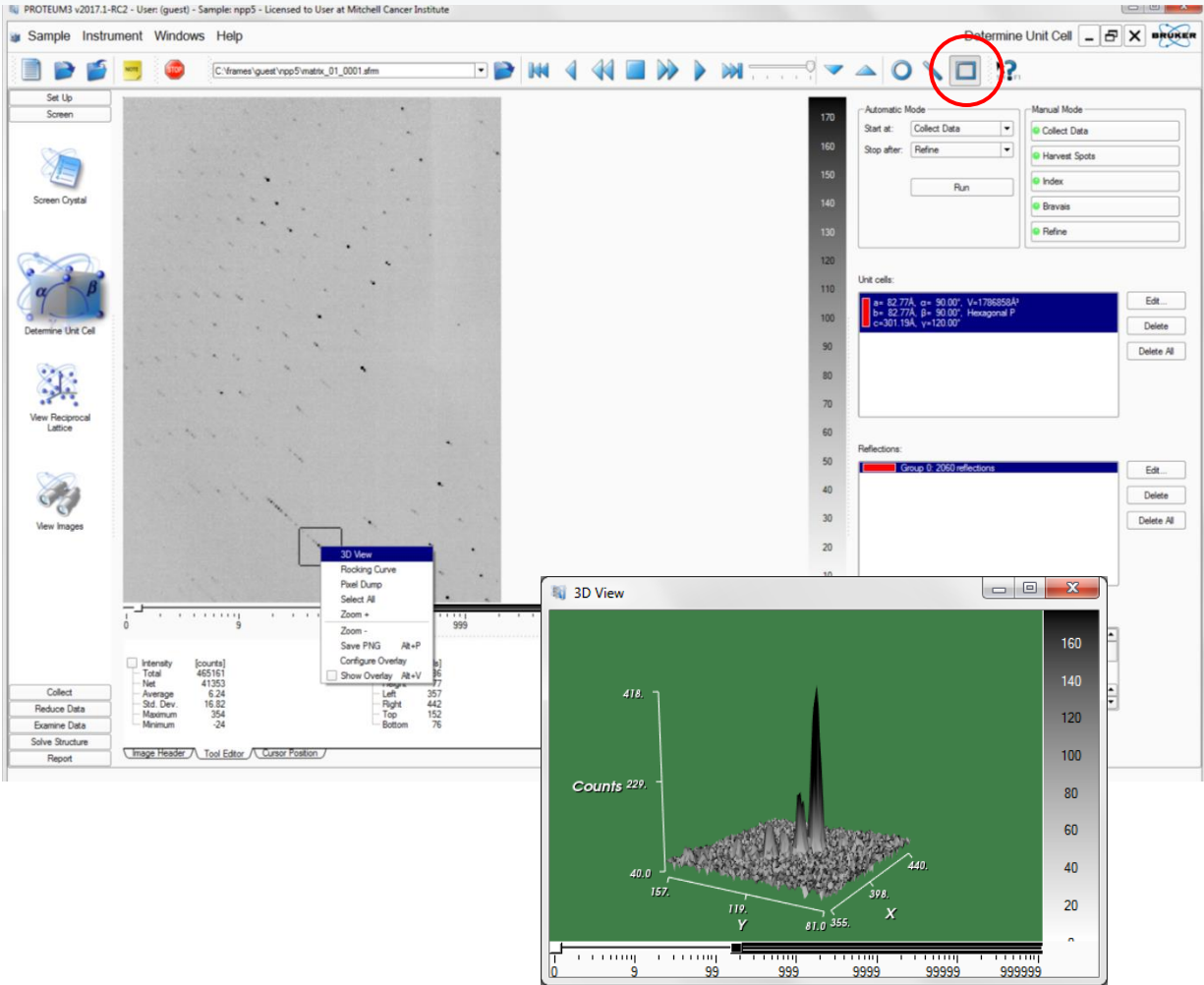
- In-house systems use a focusing optic to increase the intensity of the beam
- The divergence or crossfire angle determines the increase in intensity
- However, the larger the divergence the bigger the spots
- To resolve the reflections for larger unit cells, you have to lower the divergence or move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

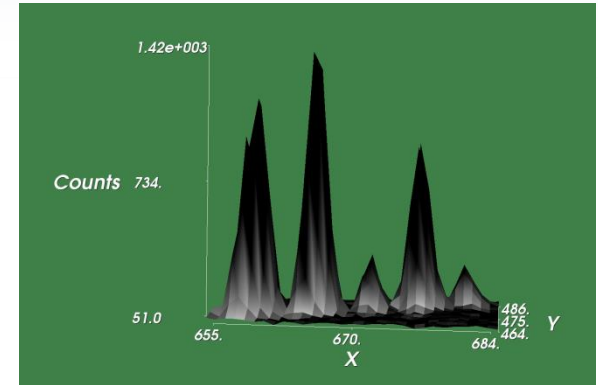
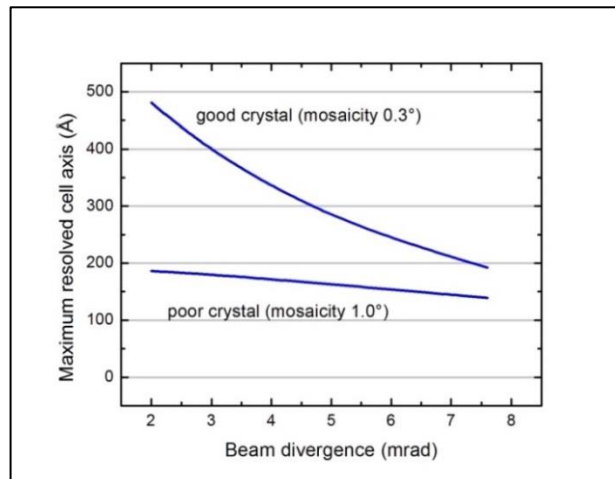
Collimator	Divergence	Beam size (μm)	Relative Intensity	Longest Unit cell Axis (\AA)
Full	7.6	70	1.00	~ 180
0.3 x 17	~ 7.0	70	0.85	~ 220
0.3 x 10	~ 5.0	70	0.48	~ 300
0.2 x 6	~ 3.0	70	0.23	~ 370

Data Collection

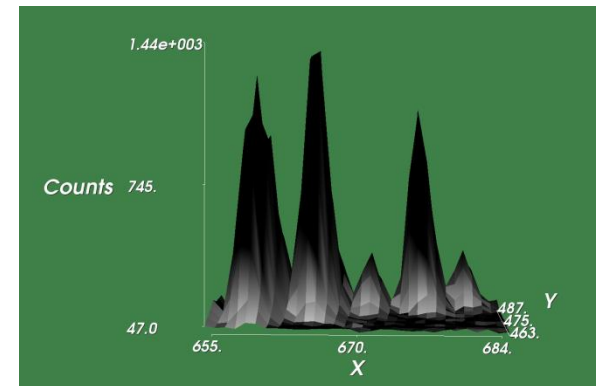
Divergence



- 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

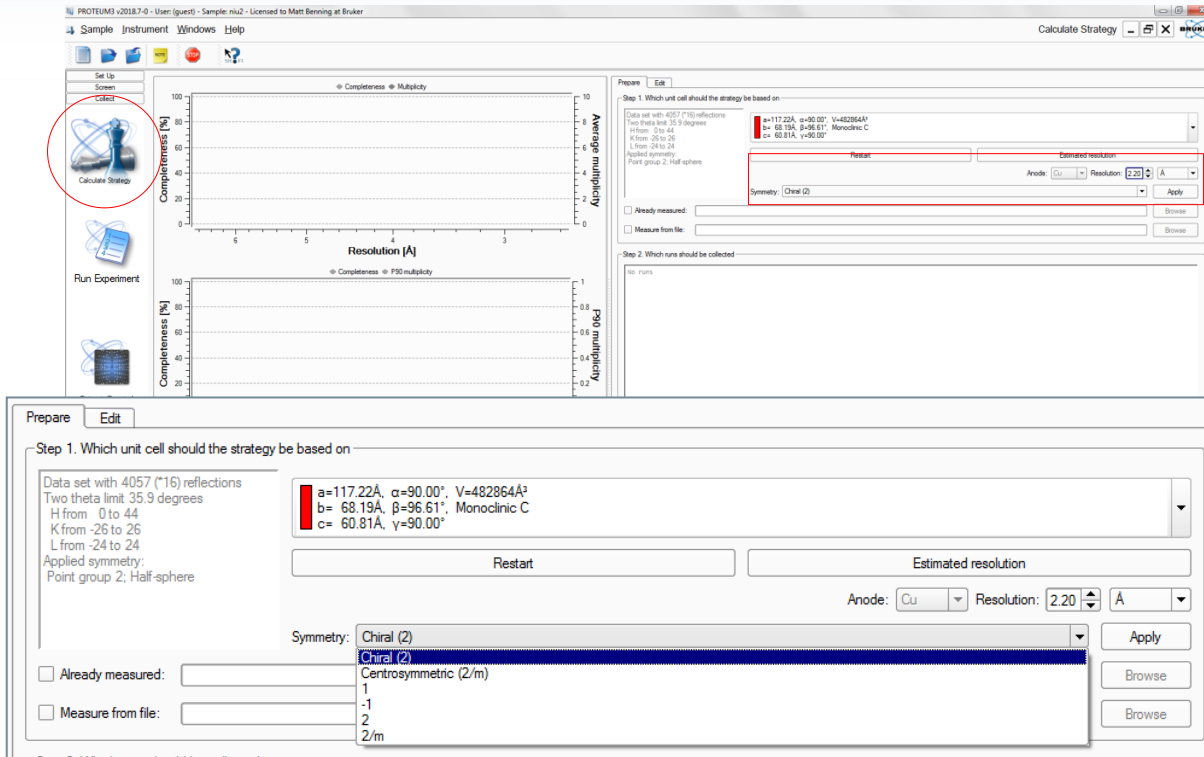
Experimental parameters to be set

- Sample to detector distance
- Exposure time
- Scan width

Parameters against which the runs are optimized

- Multiplicity
- Completeness
- Total exposure time

Data Collection Strategy



- 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)

Multiple crystal data collection

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

Data Collection Strategy

Determine strategy

A screenshot of a software dialog box titled "Parameters for the strategy determination". The dialog has a standard Windows-style title bar with a question mark icon and a close button. The main content area is organized into several sections. At the top, there's a "Data collection strategy" section with a "Crystal to detector distance (mm)" field set to "60" and a "Reset" button. Below that is the "Strategy type" section, which features a dropdown menu currently showing "Generic phi and omega scans" and a list of options: "Generic phi and omega scans" (highlighted), "Generic omega scans", "Friedel pairs in same frame", and "Two 360 degree phi scans in reverse beam geometry". To the left of this dropdown is a "Shutterless mode" section with a checked checkbox for "Shutterless data collection". Below the dropdown is a "Shortest normalized exposure time [sec/deg]" field set to "1.00". The bottom section contains several checkboxes: "Use low temperature safe scan ranges" (checked), "Avoid overlap due to longest axis" (unchecked), and "Strict efficiency theta limitations" (checked). Below these are two more fields: "Desired completeness" set to "0.995" and "Minimum multiplicity for 90% of the data" set to "1.00". At the bottom of the dialog are "OK" and "Cancel" buttons.

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 screenshot displays the Proteomics v2018.7.0 software interface. The main window is titled 'Calculate Strategy' and shows a 'Protein' tab with various parameters. On the left side, there are two graphs: 'Completeness (%)' vs 'Resolution [Å]' and 'Completeness (%)' vs 'P90 multiplicity'. The bottom of the window shows a summary of the data collection strategy and results.

Summary of Data Collection Strategy:

- 1_P 52 min (0.9h +0 Wed 12:15) 9681 refls, 3124 unique; P90mult 2.2; Av. mult 3.1; Complete 99.94 %
- 2_0 21 min (1.2h +0 Wed 12:36) 5360 refls, 2 unique; P90mult 3.6; Av. mult 4.8; Complete 100.00 %

Multiplicity Statistics:

- P90 multiplicity: 3.6
- Average multiplicity: 4.8
- Missing reflections: 0
- Completeness: 100.00 %

Multiplicity percentiles:

- 1% of all reflections have a multiplicity greater than 8.0
- 10% of all reflections have a multiplicity greater than 6.9
- 50% of all reflections have a multiplicity greater than 5.3
- 90% of all reflections have a multiplicity greater than 3.6
- 99% of all reflections have a multiplicity greater than 2.1

Cumulative multiplicity:

- Reflections with multiplicity 1 and higher: 100.0 %
- Reflections with multiplicity 2 and higher: 99.7 %
- Reflections with multiplicity 3 and higher: 94.4 %
- Reflections with multiplicity 4 and higher: 86.5 %
- Reflections with multiplicity 5 and higher: 59.2 %
- Reflections with multiplicity 6 and higher: 31.5 %

- 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



Scan Parameters

Scan parameters

Frame angle [degrees] 0.50

Frame time [seconds] 10.00

Theta Dependency

Theta dependent scan times

Frame time for lowest 2Theta [seconds] 10.00

Frame time for highest 2Theta [seconds] 20.00

Intermediate exponent 3.0

2Theta division size [degrees] 1.0

Time division size [seconds] 0.10

Scans	2Theta [deg]	Time [sec]
1	1.0	(10.0)

Normalized scan time: 20.00 [sec/deg]
Total time: 01:41 [hrs:min]
Expected end time: (2019) Wed, Jan 09, 14:43

Time slots

2 hrs

4 hrs

8 hrs

16 hrs

Set ETA...

Detector coverage and frame time

OK Cancel

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

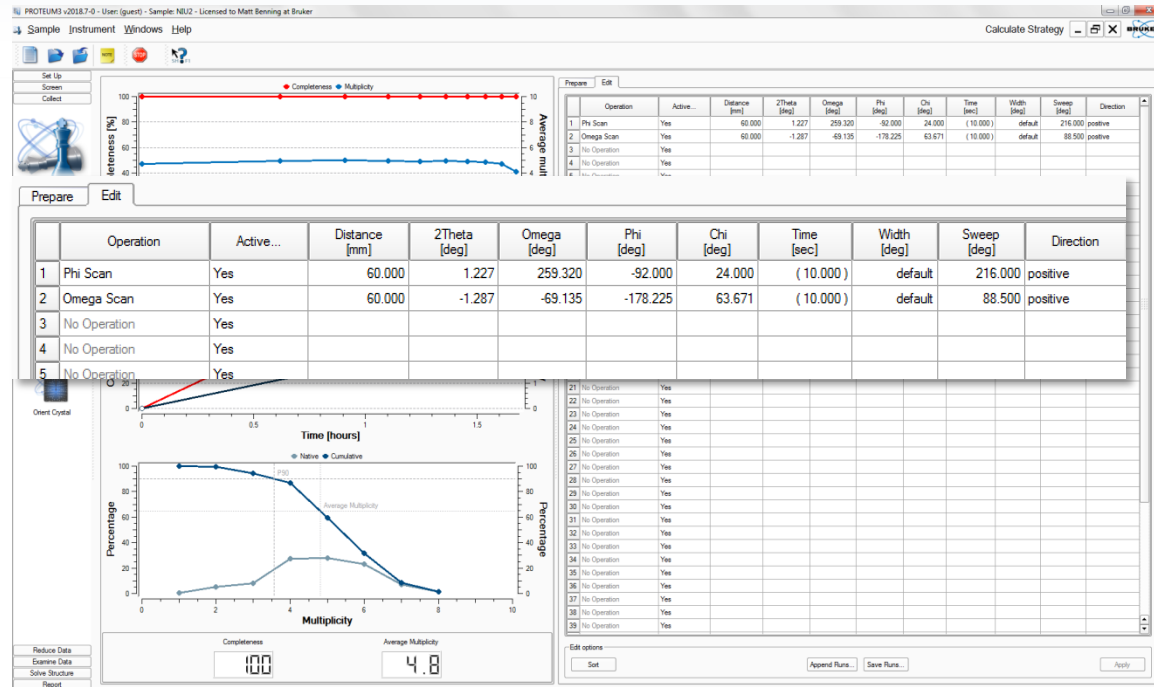
- Set time allows you input the actual time you want the experiment to be finished
- The program does this by adjusting the frame time

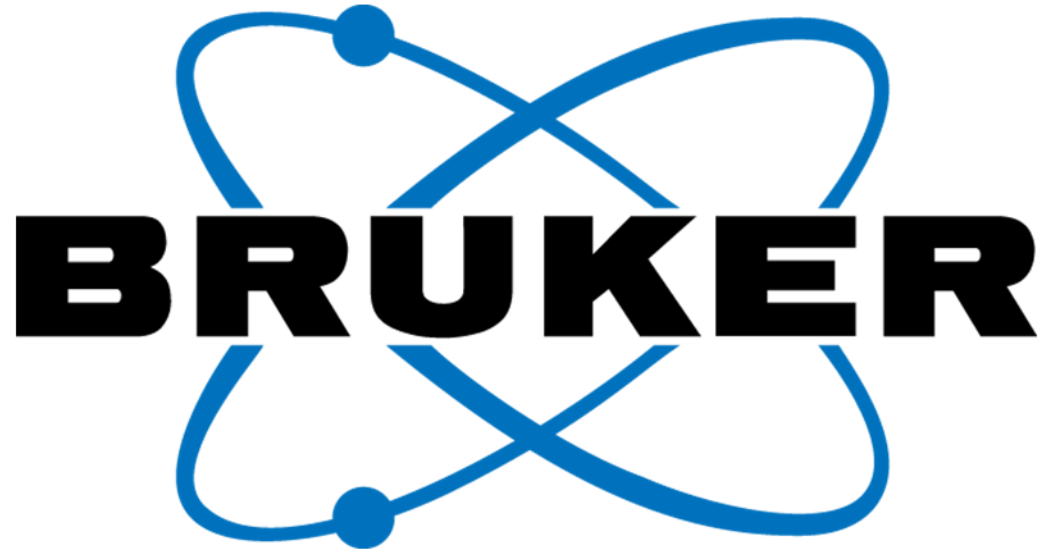
Data Collection Strategy

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





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