Data Collection and Reduction with APEX6

Ashley (Weiland) Schmidt, PhD

Applications Scientist, SC-XRD

ashley.schmidt@bruker.com

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Ashley (Weiland) Schmidt – Applications Scientist SC-XRD

Awards

- ACS Division of Inorganic Chemistry Young Investigator Awardee
- Eugene McDermott Graduate Fellow
- DOE Office of Science Graduate Student Research Awardee
- Excellence in Education Foundation Scholar
- Duquesne University Bayer Scholar

Experience

- Bruker Applications Scientist (Nov '21)
- Director's Postdoctoral Fellow – Los Alamos National Laboratory
  - Mapped crystallographic deviations in samples of unconventional superconductor, UTe_2, exhibiting varying superconducting transition temperatures

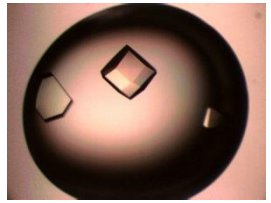
Education

- PhD Chemistry (Advisor: Dr. Julia Chan)
  - Elucidated complex crystal structures of intermetallics
- SCGSR Awardee (Argonne National Laboratory - Advisor: Dr. Saul Lapidus)
  - Designed and built experimental setups for high temperature in situ synchrotron powder X-ray diffraction characterization
- B.S. Environmental Chemistry (Research Advisor: Dr. Jen Aitken)
  - Synthesis and characterization of Li_2-II-IV-S_4 semiconductors

Publications

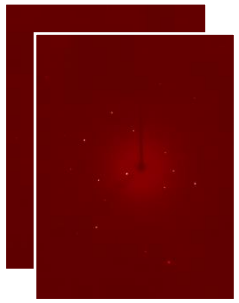
- 20+ publications in peer reviewed scientific journals, over 500 citations

SCD Workflow



Sample

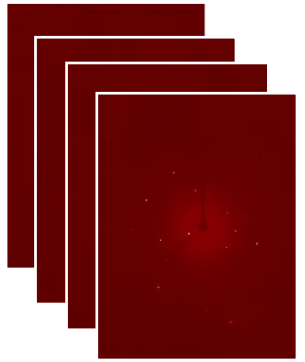
Screening/
Indexing



Diffraction
Frames

Data
Collection

5 – 15 GB



Full set of Diffraction
Frames

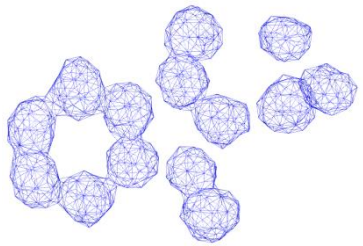
Data
Reduction

1	0	0	1.0000	0.3000
1	0	0	1.1000	0.2000
-1	0	0	2.1000	0.5000
2	0	0	4919.51	199.78
-2	0	0	4931.71	191.98
3	0	0	0.1000	0.3000
3	0	0	0.3000	0.3000
-3	0	0	1.2000	0.3000
4	0	0	837.52	21.50
-4	0	0	856.61	16.60
5	0	0	1.3000	1.3000
6	0	0	1230.28	32.20
7	0	0	-0.4000	1.5000
0	-1	0	0.2000	0.1000
0	-1	0	0.4000	0.1000
0	-1	0	0.3000	0.1000
1	1	0	170.48	4.40
1	-1	0	161.58	4.70
-1	-1	0	170.28	4.40
-1	-1	0	175.58	4.10
1	-1	0	178.88	4.50
1	-1	0	171.38	4.40
1	1	0	170.58	4.10

HKL Intensity
Data

5 – 50 MB

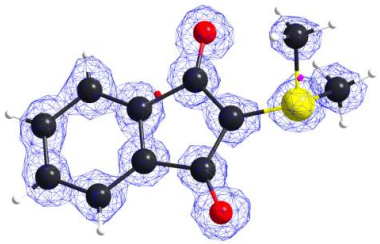
Structure
Solution



Electron
Density Map

Structure
Refinement

< 1 MB



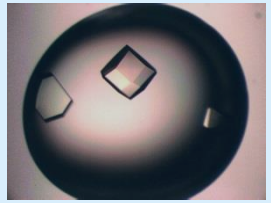
Atomic Model

Validation

Publication

SCD Workflow

5 – 15 GB



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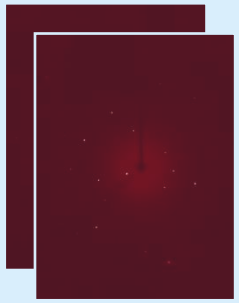
Screening/
Indexing



Diffraction
Frames

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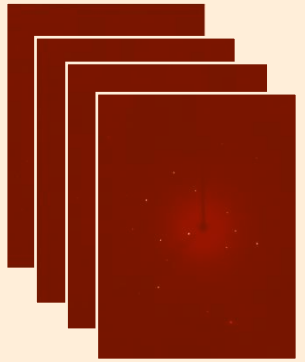
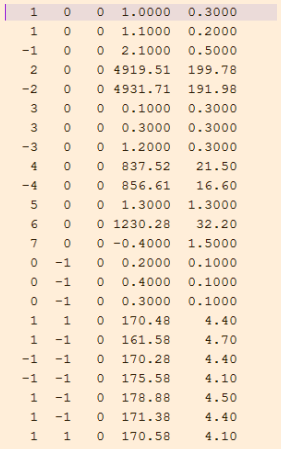
Collection



Full set of Diffraction
Frames

Data
Reduction

PC



HKL Intensity
Data

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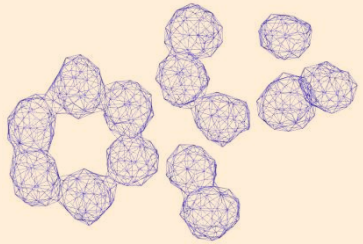
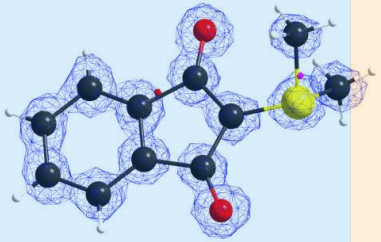
Structure
Solution



Electron
Density Map

Structure
Refinement

< 1 MB



Atomic Model

Validation

Publication

Human

DATA COLLECTION AND REDUCTION WITH APEX6

Unit Cell Determination in APEX6

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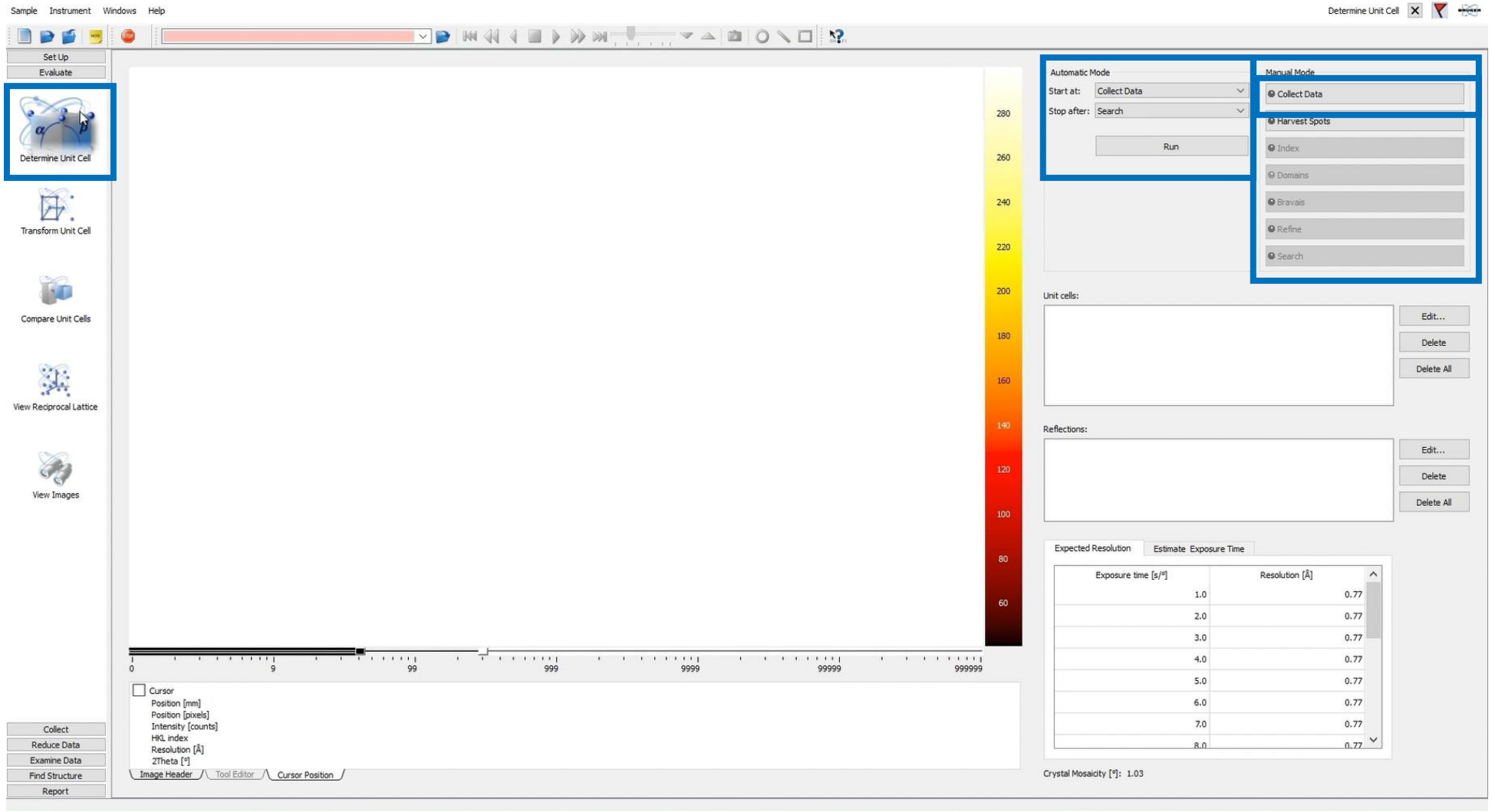
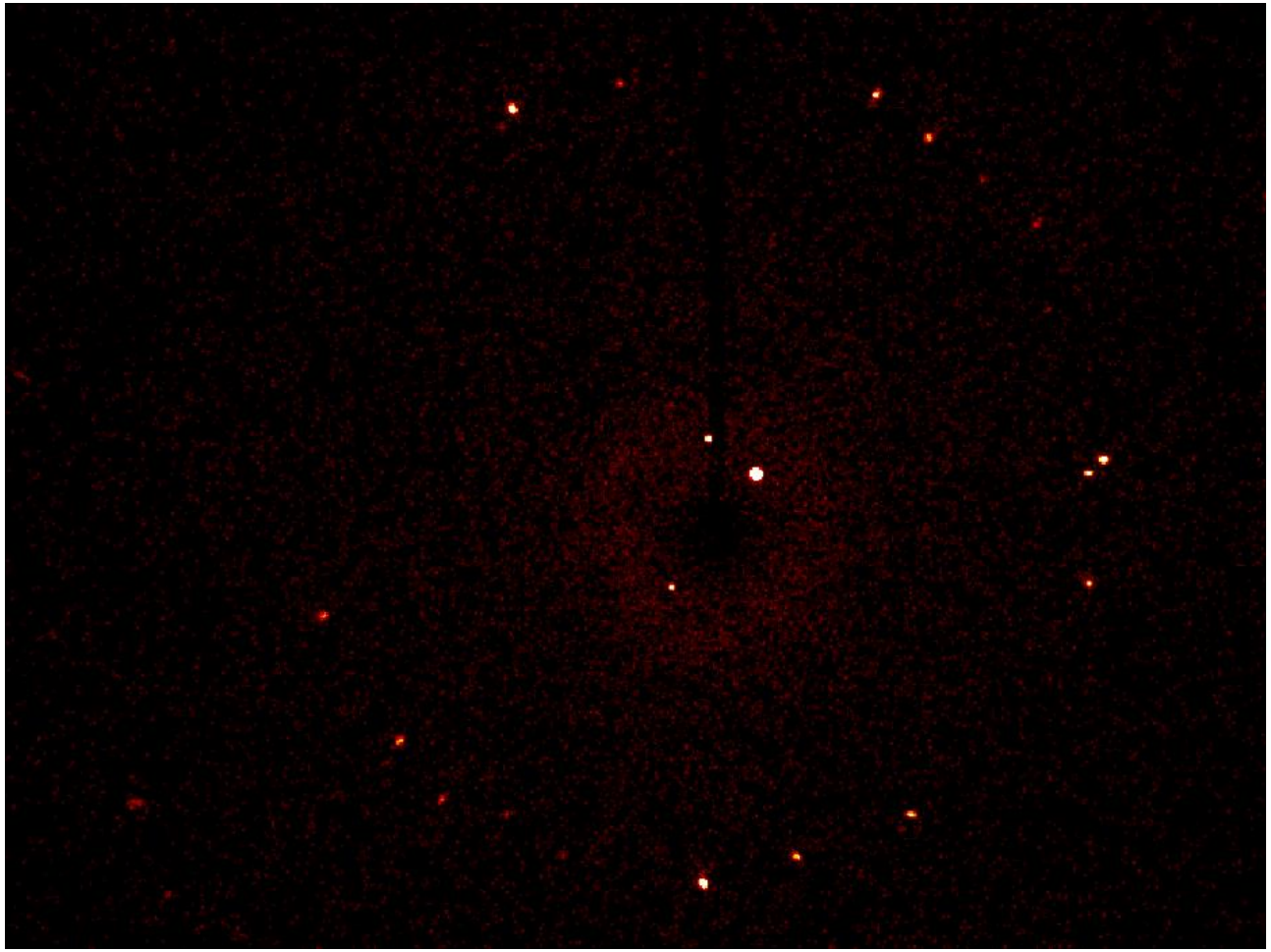
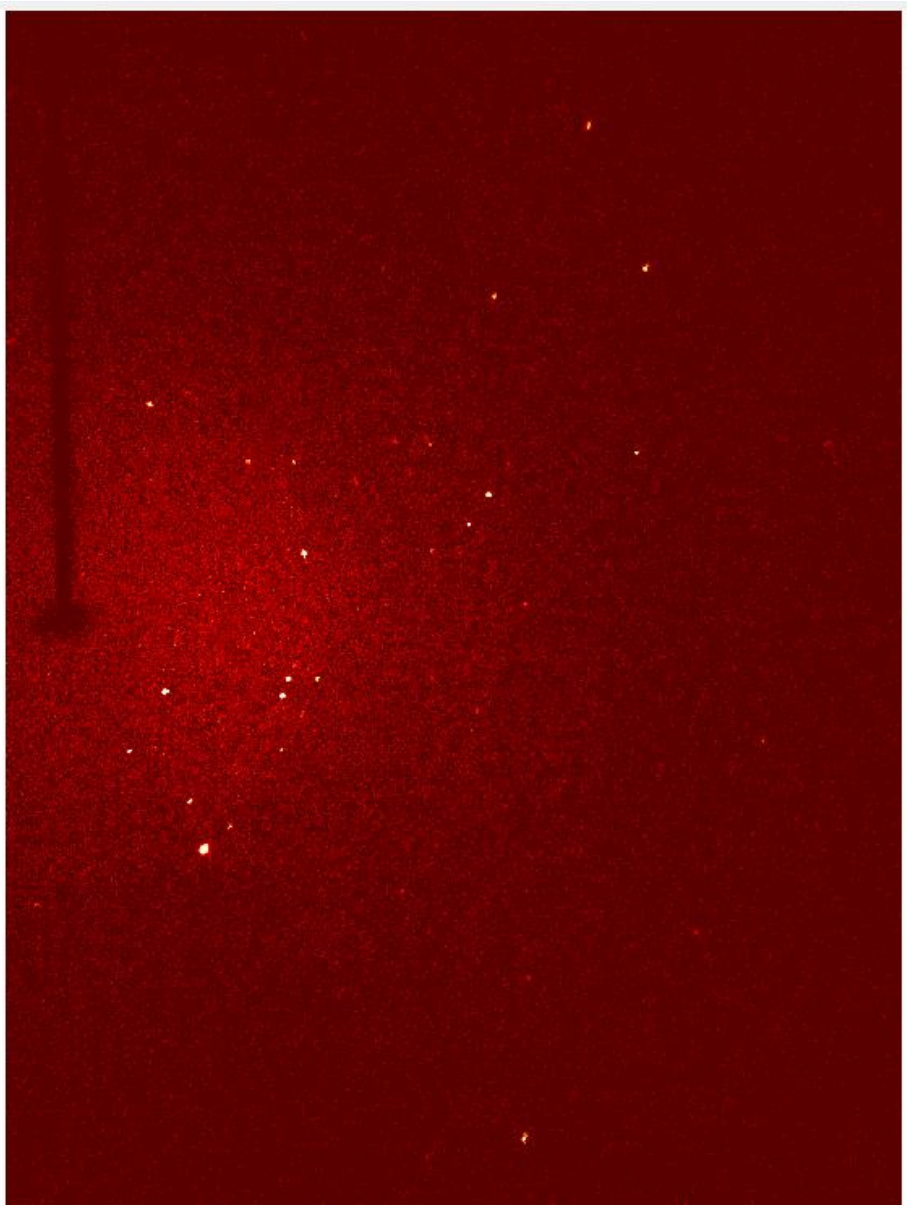
Ashley.Schmidt@bruker.com

Determine Unit Cell – Collect Data

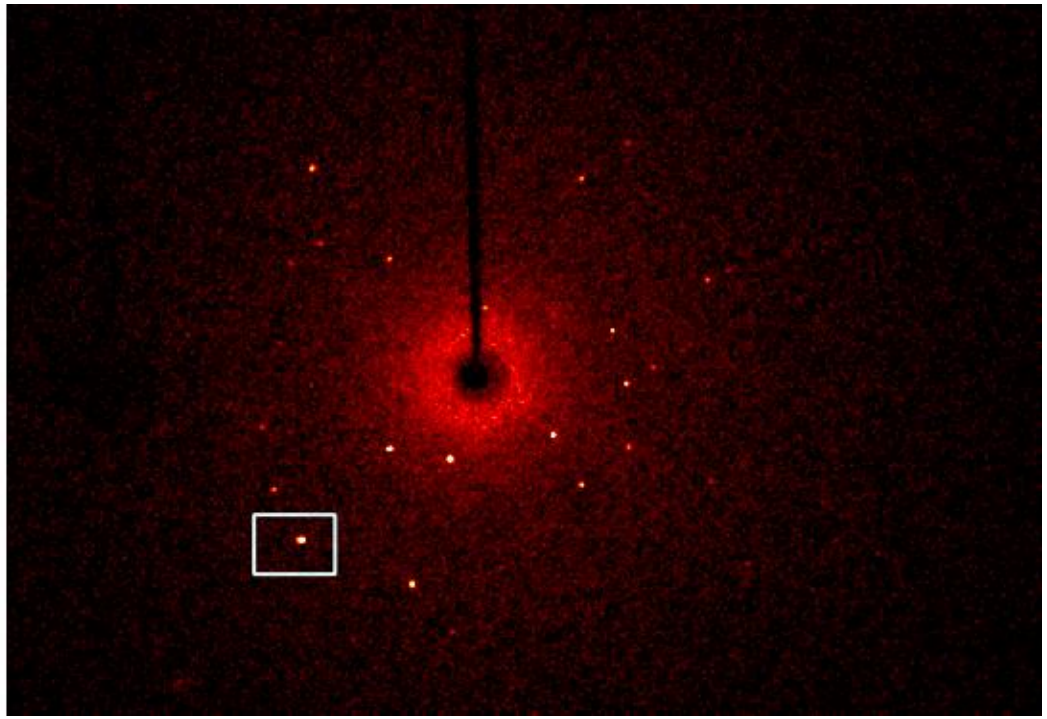


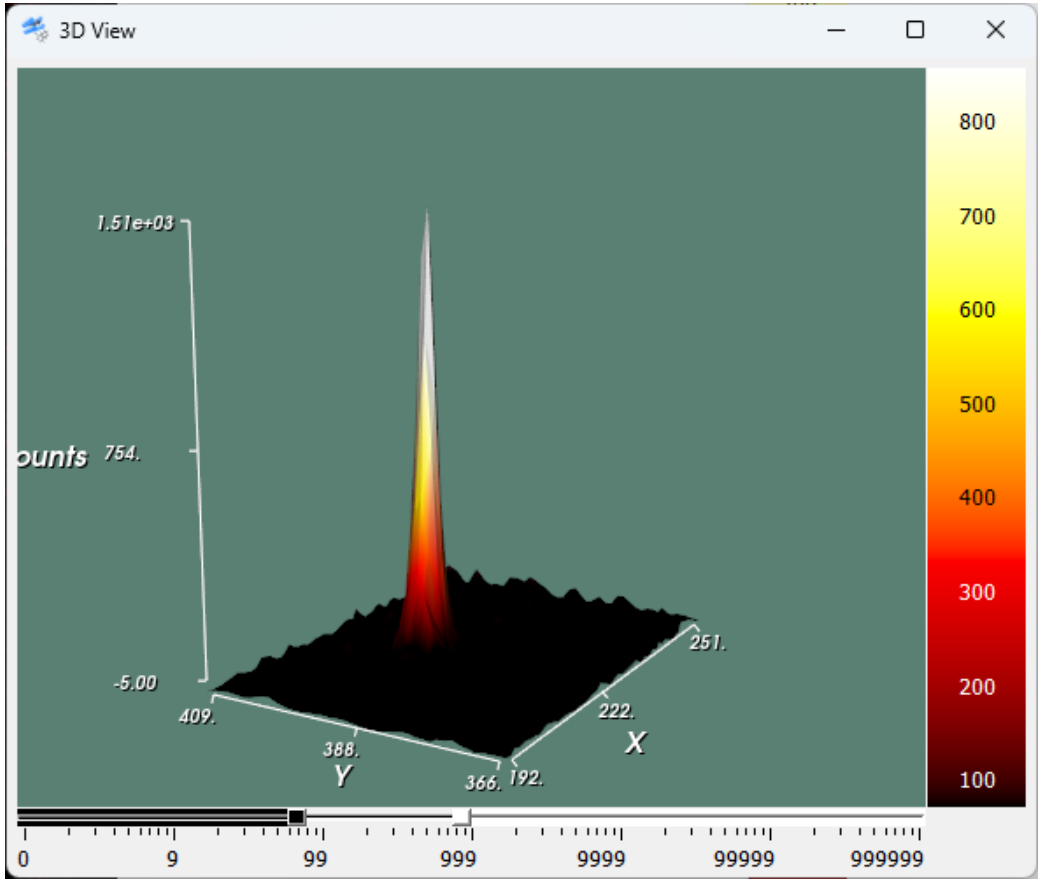
What Do Data Frames Look Like?



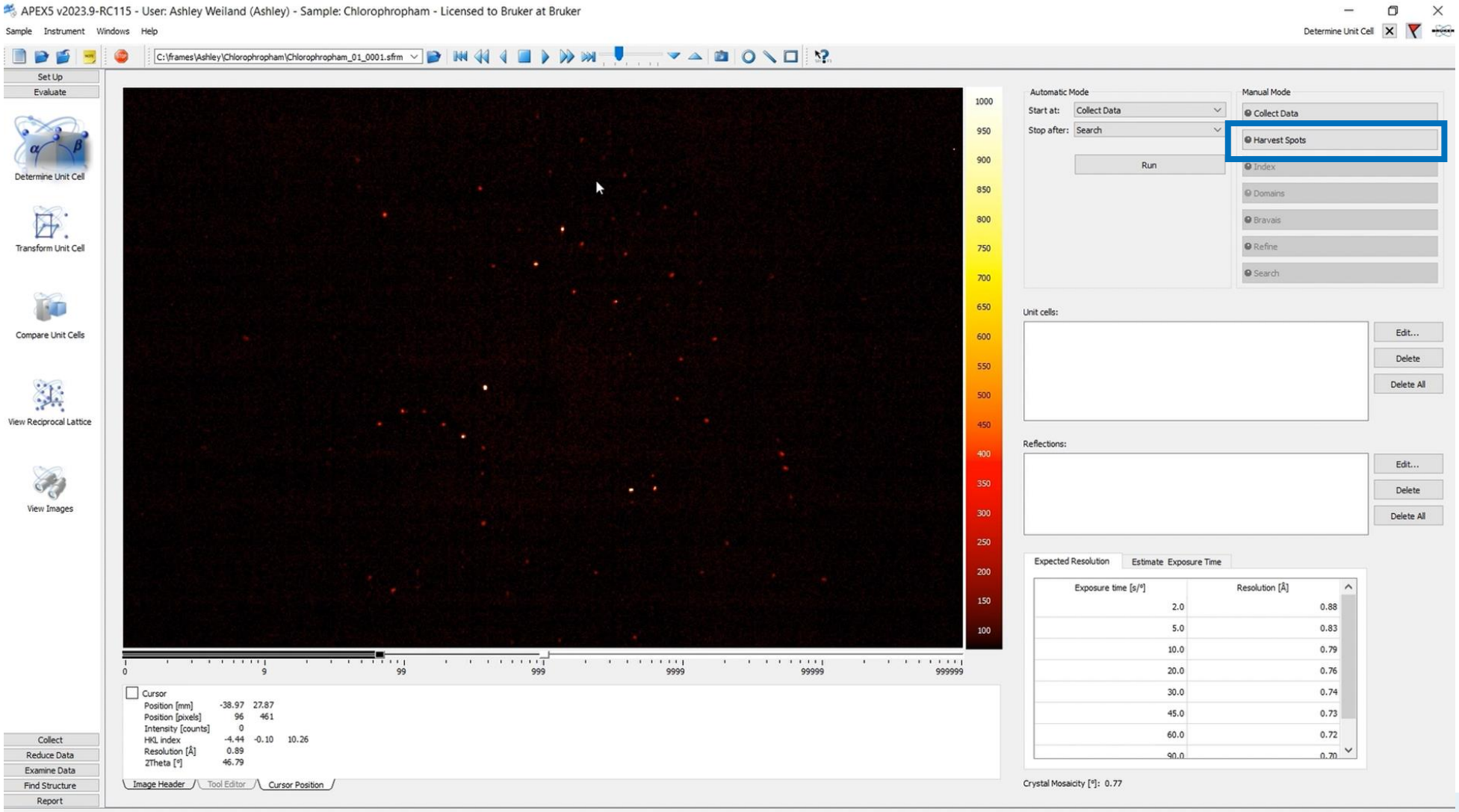


3D View of a Reflection

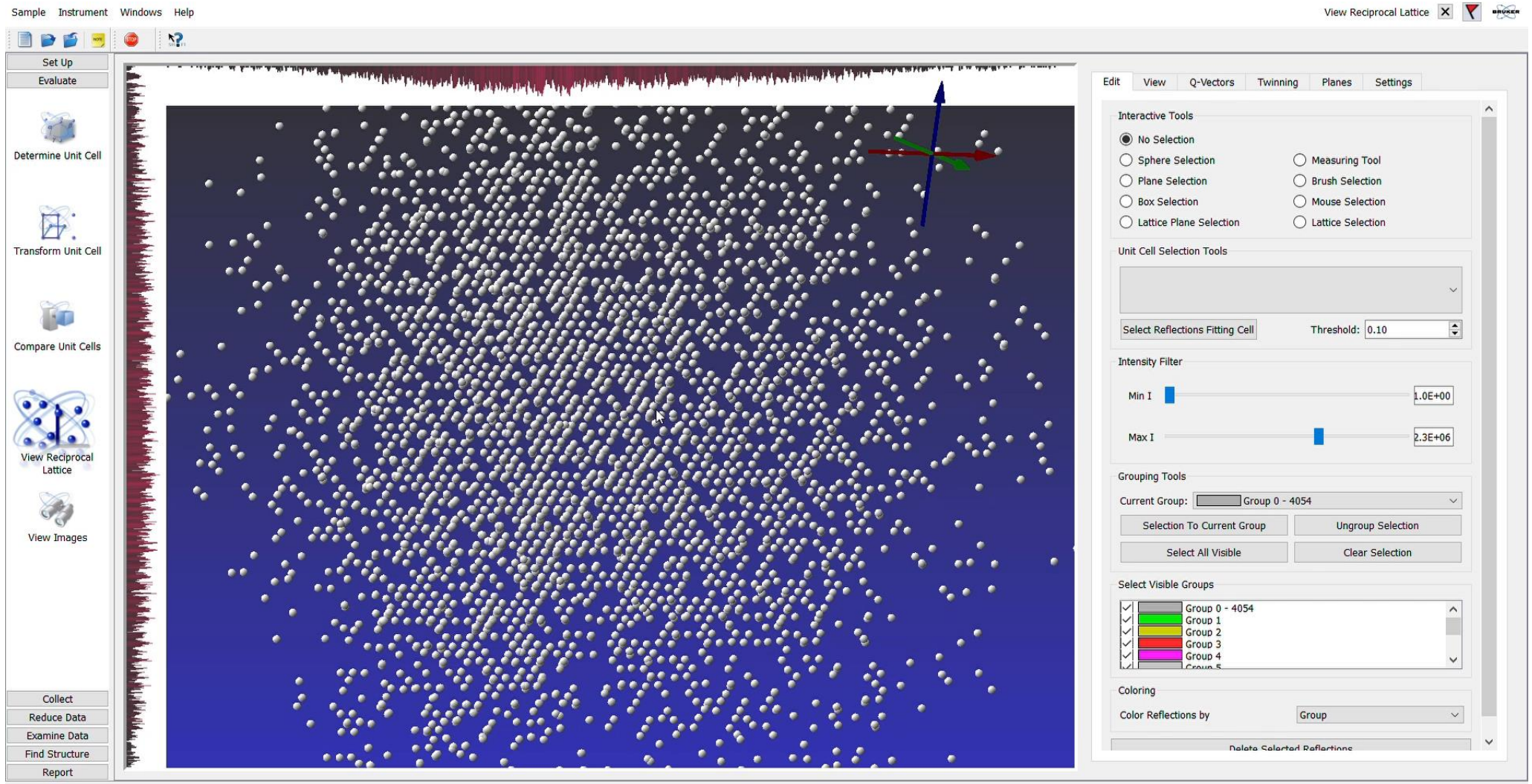




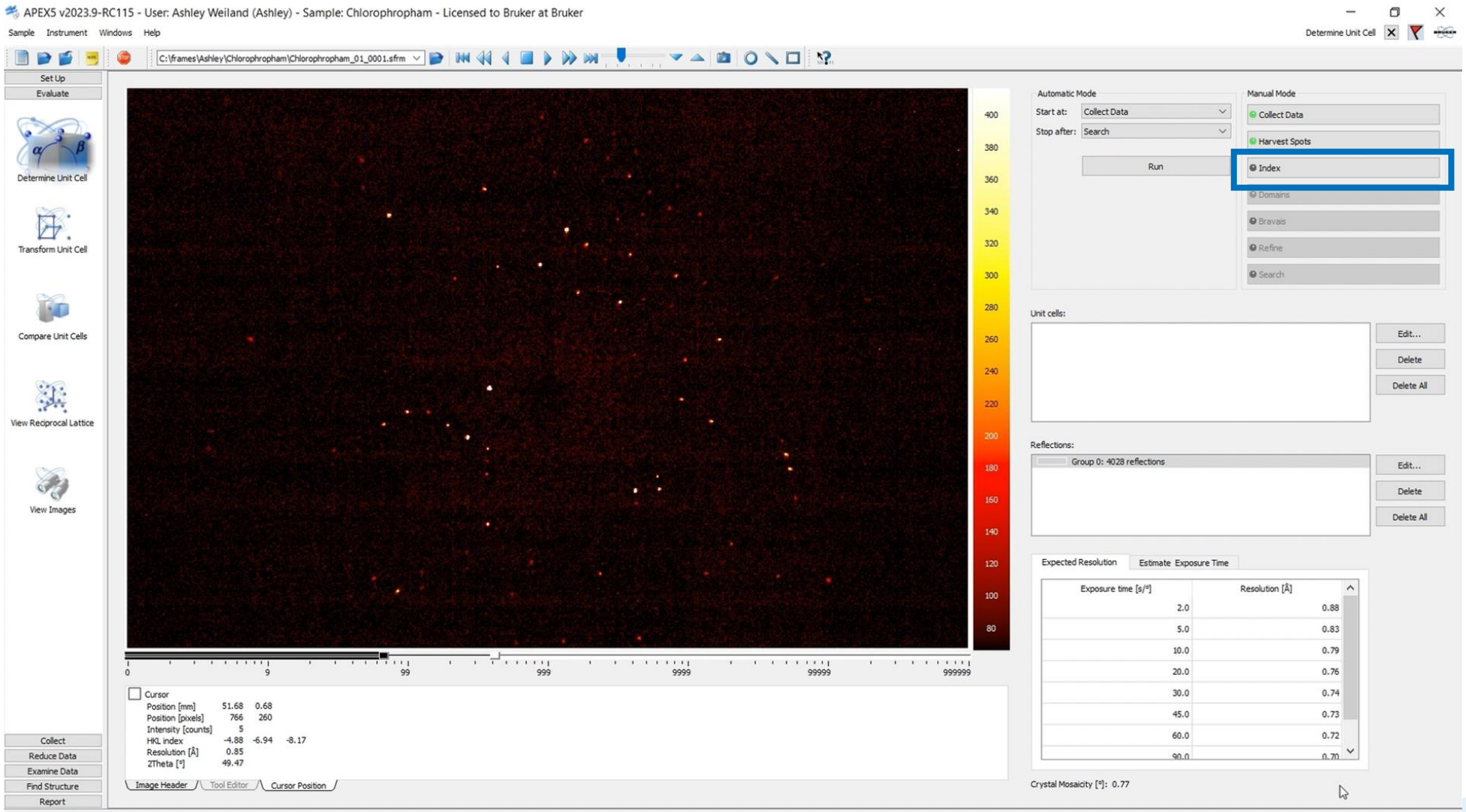
Determine Unit Cell – Harvest Spots



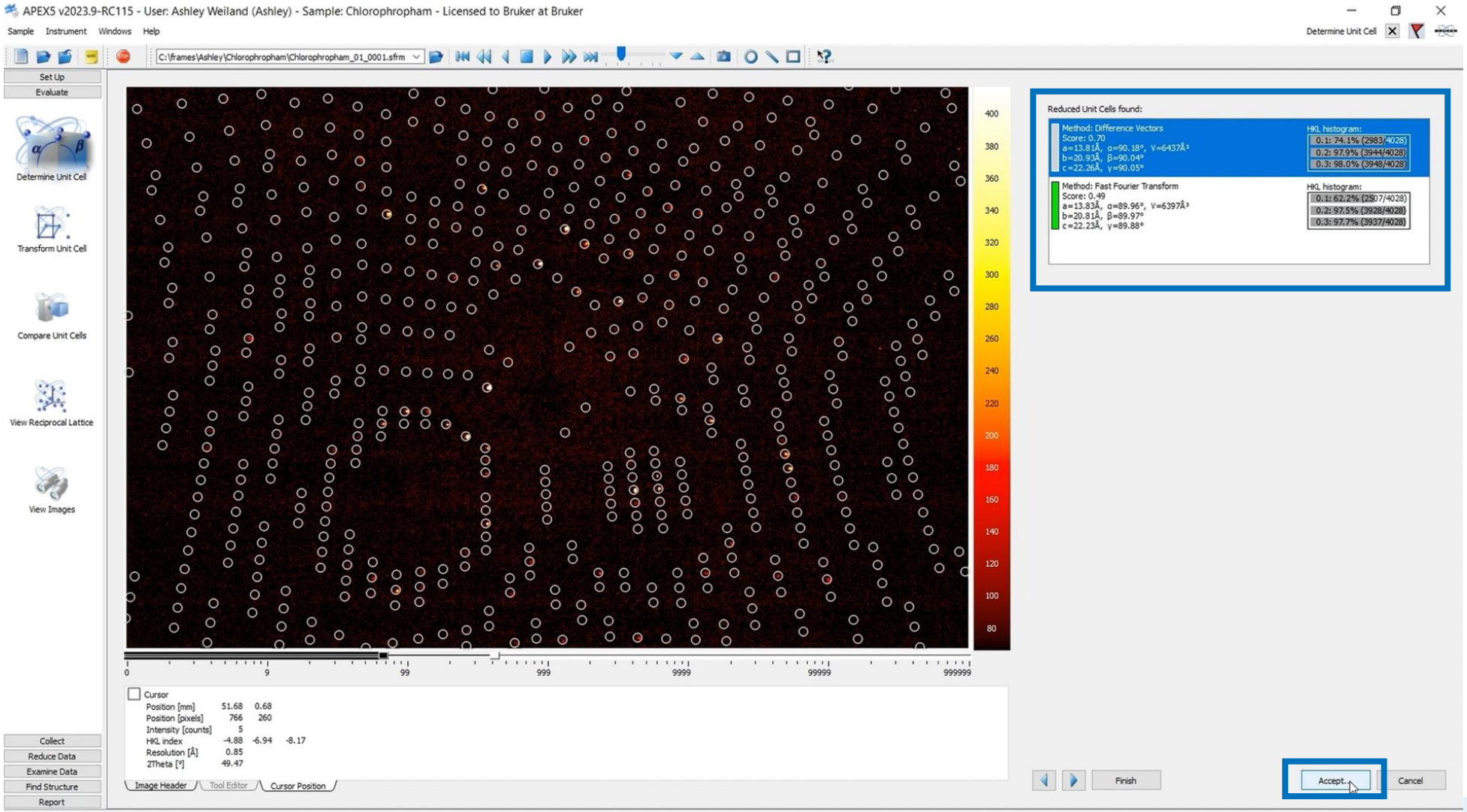
Seeing Symmetry (video)



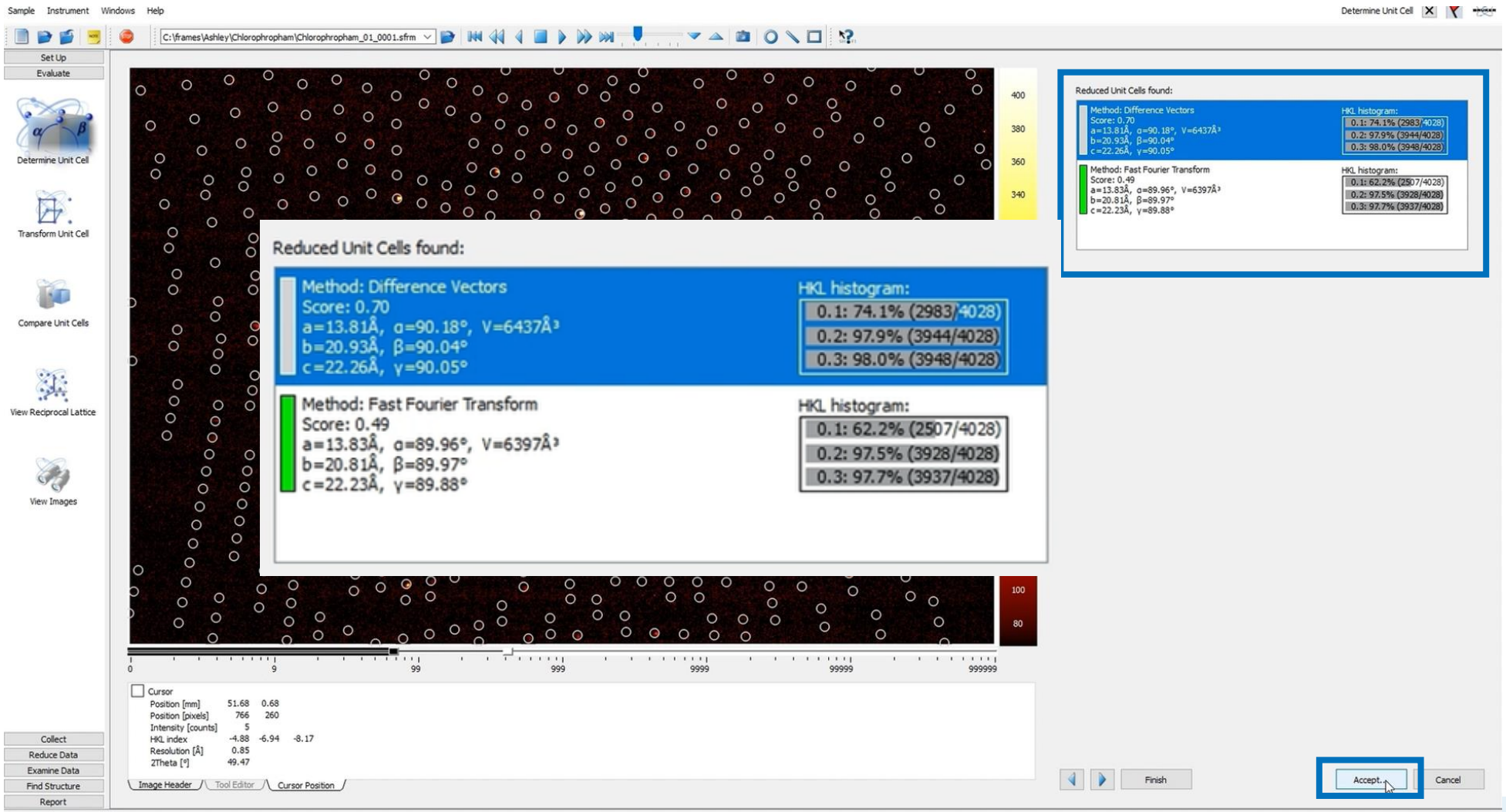
Determine Unit Cell – Index



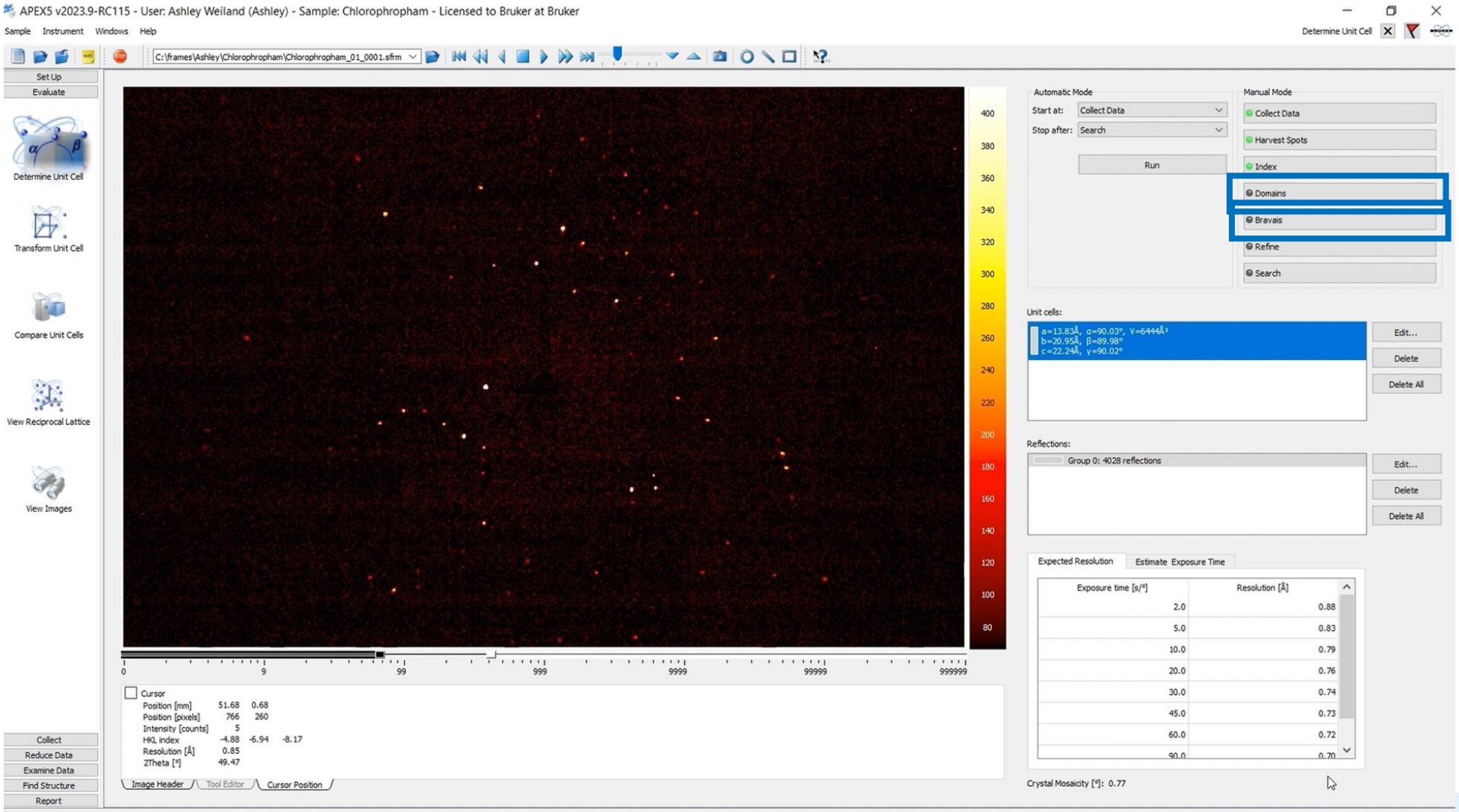
Determine Unit Cell – Index



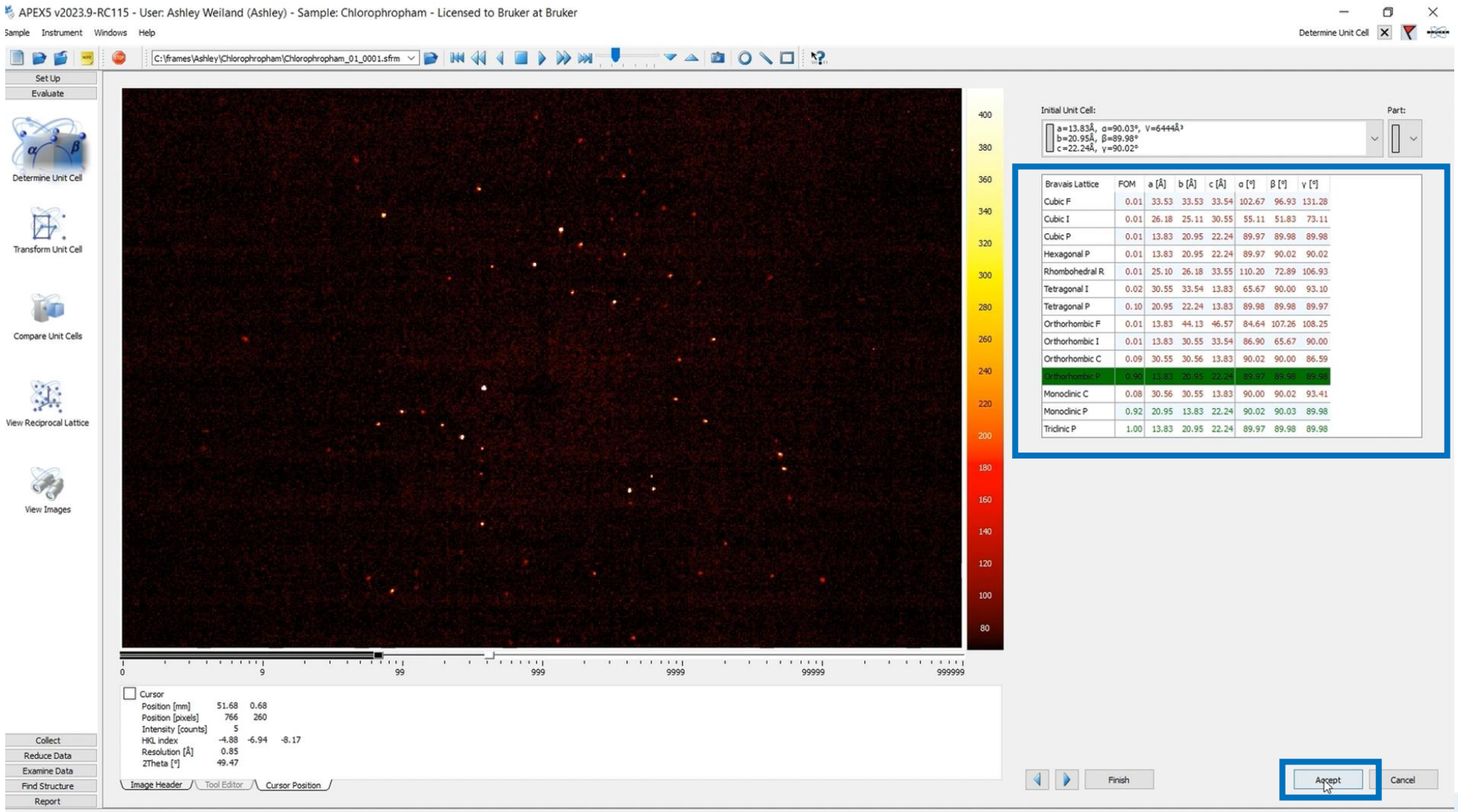
Determine Unit Cell – Index



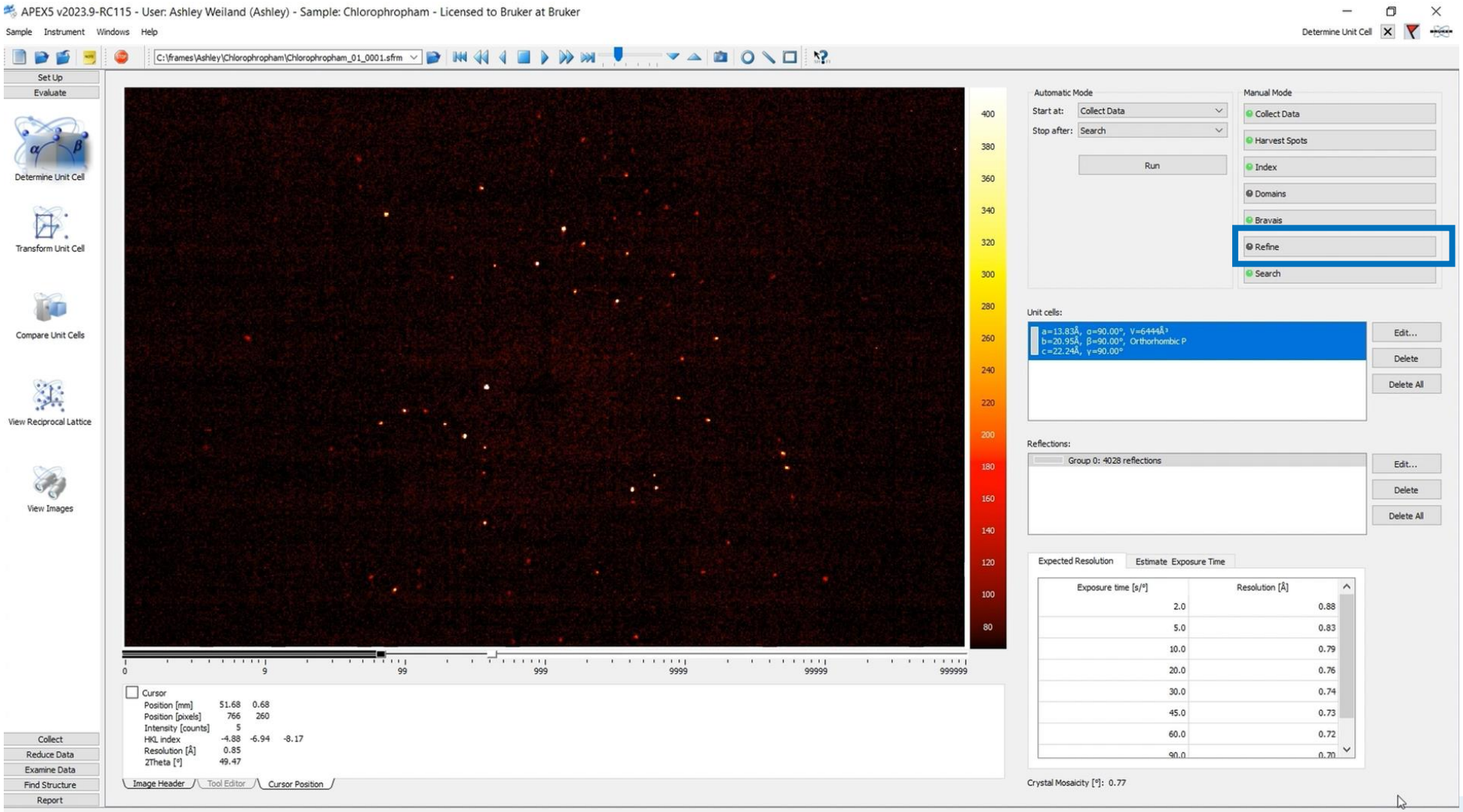
Determine Unit Cell – Bravais



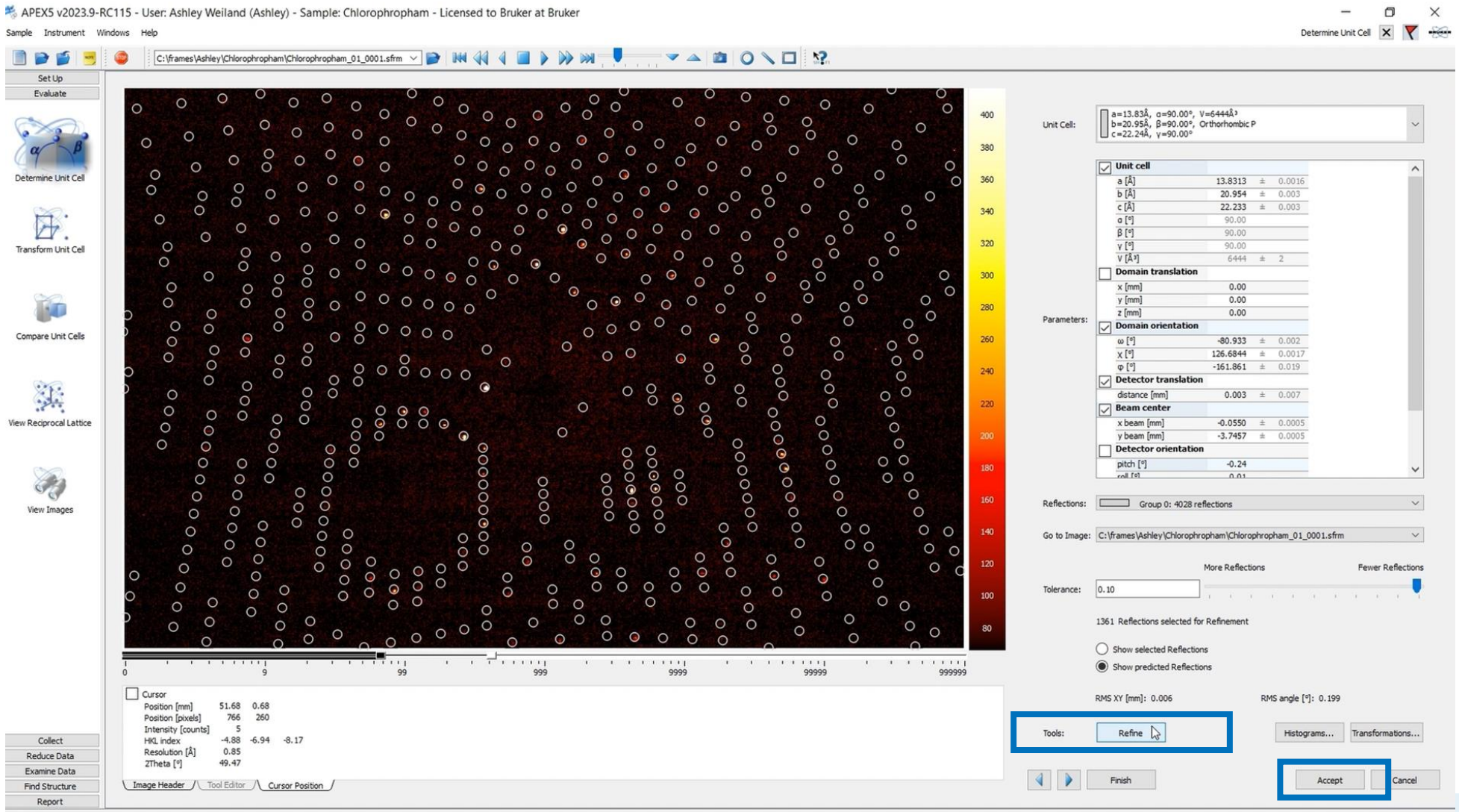
Determine Unit Cell – Bravais



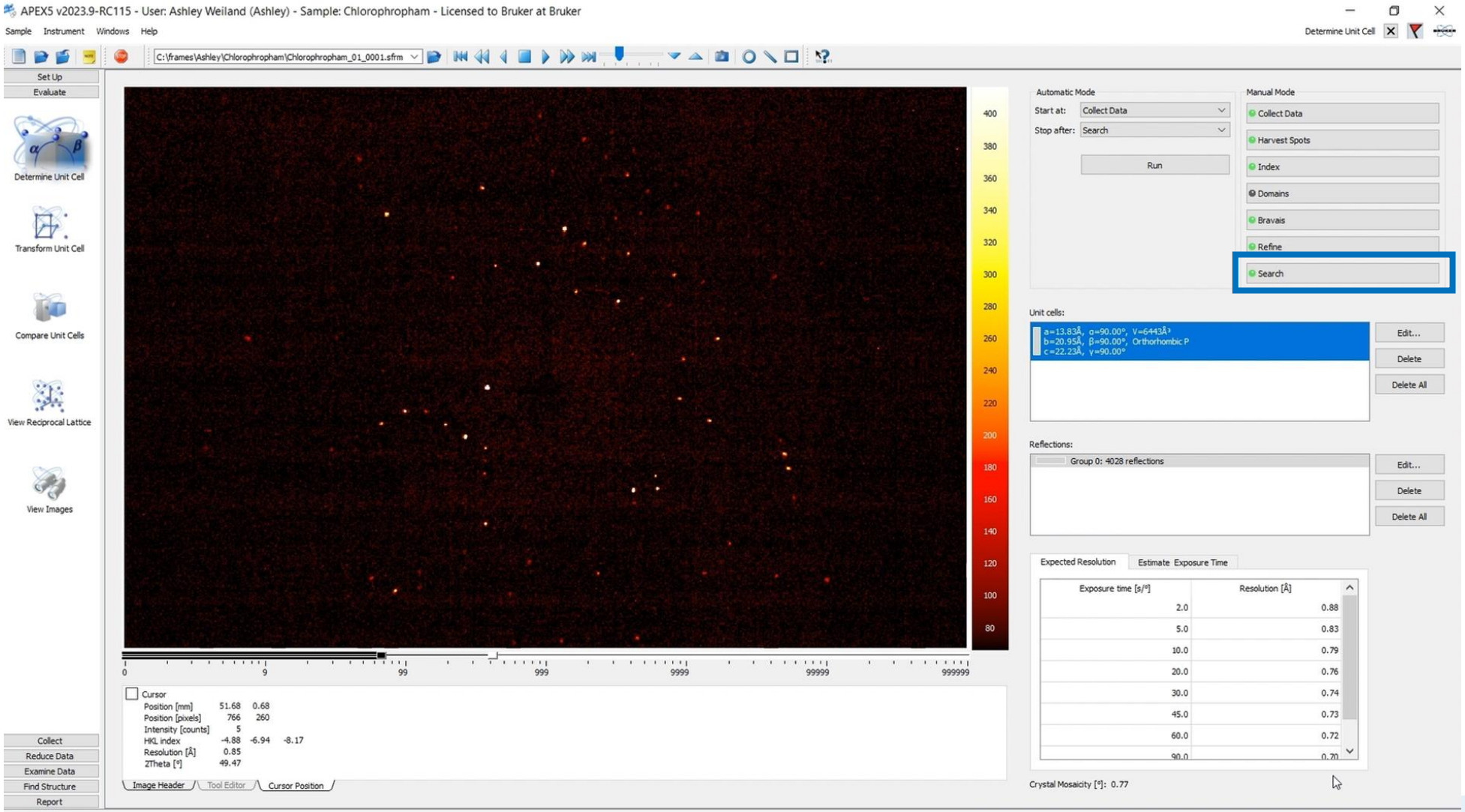
Determine Unit Cell – Refine



Determine Unit Cell – Refine



Determine Unit Cell – Search



DATA COLLECTION AND REDUCTION WITH APEX6

Strategy Determination in APEX6

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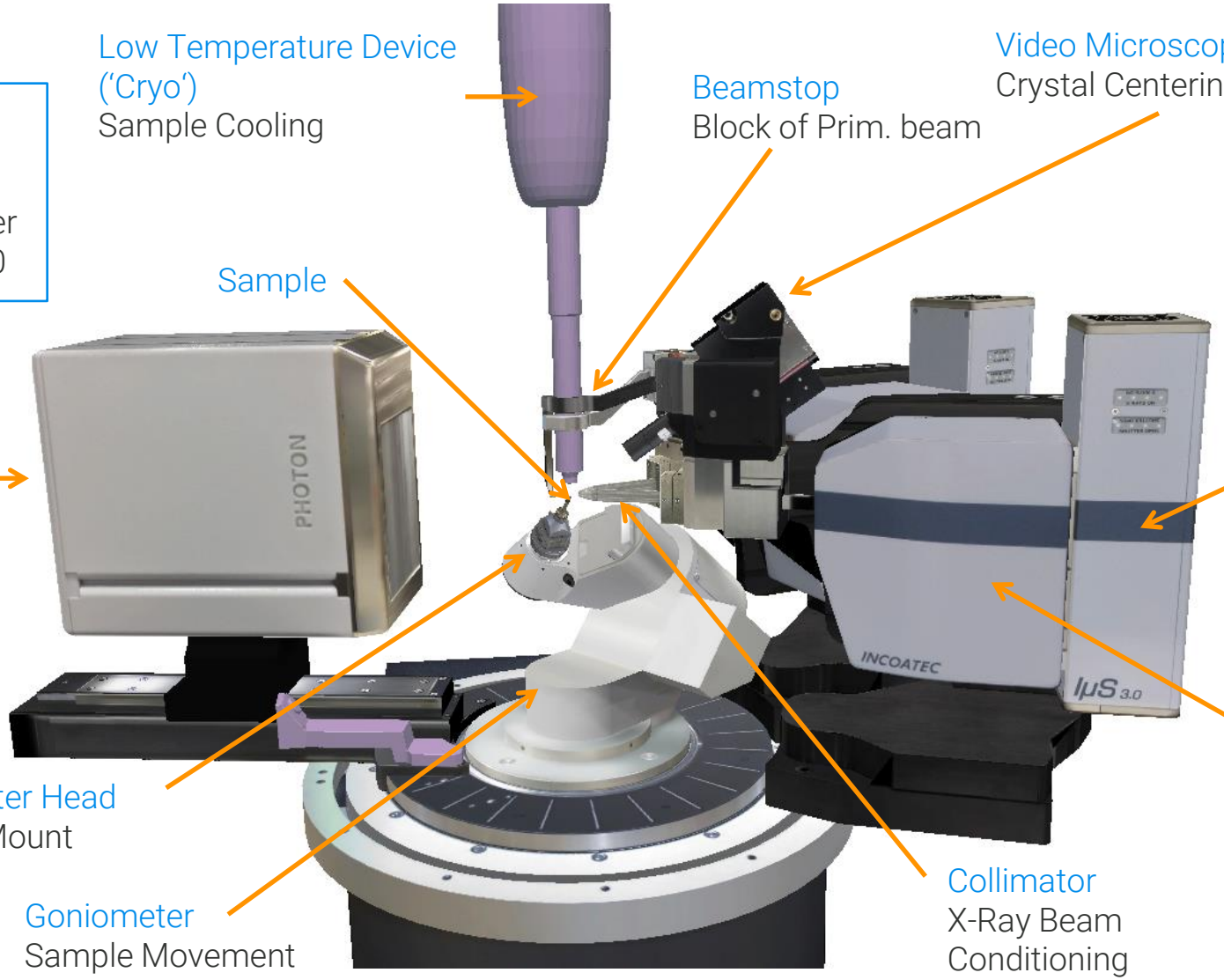
The Anatomy of SC-XRD

D8 VENTURE
- Dual Microfocus Source
- PHOTON III Detector
- 4-Axis KAPPA Goniometer
- Oxford Cryo Stream 1000



Low Temperature Device ('Cryo')
Sample Cooling

Beamstop
Block of Prim. beam

Video Microscope
Crystal Centering

Sample

X-Ray Detector
Data Recording

X-Ray Source
Generation of X-rays

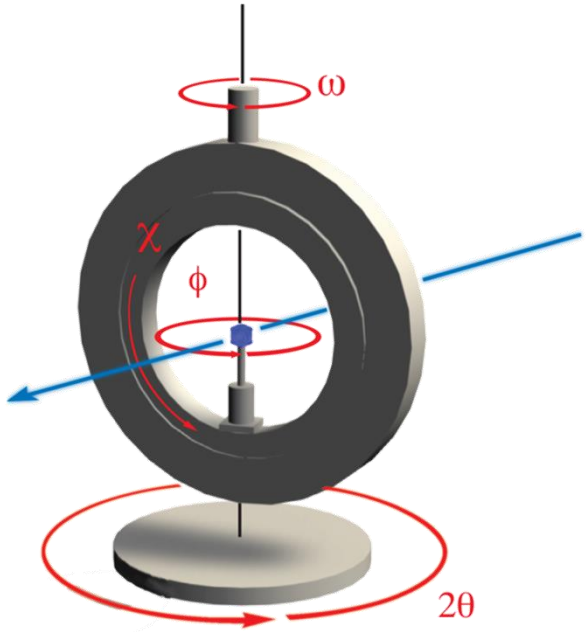
Goniometer Head
Sample Mount

X-Ray Optics
Monochromatization

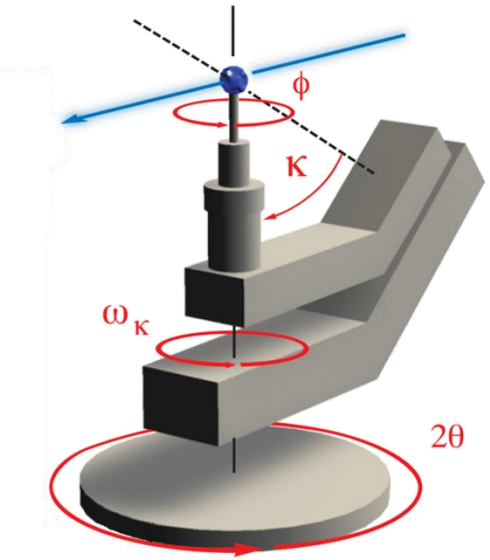
Goniometer
Sample Movement

Collimator
X-Ray Beam
Conditioning

Goniometer Geometry

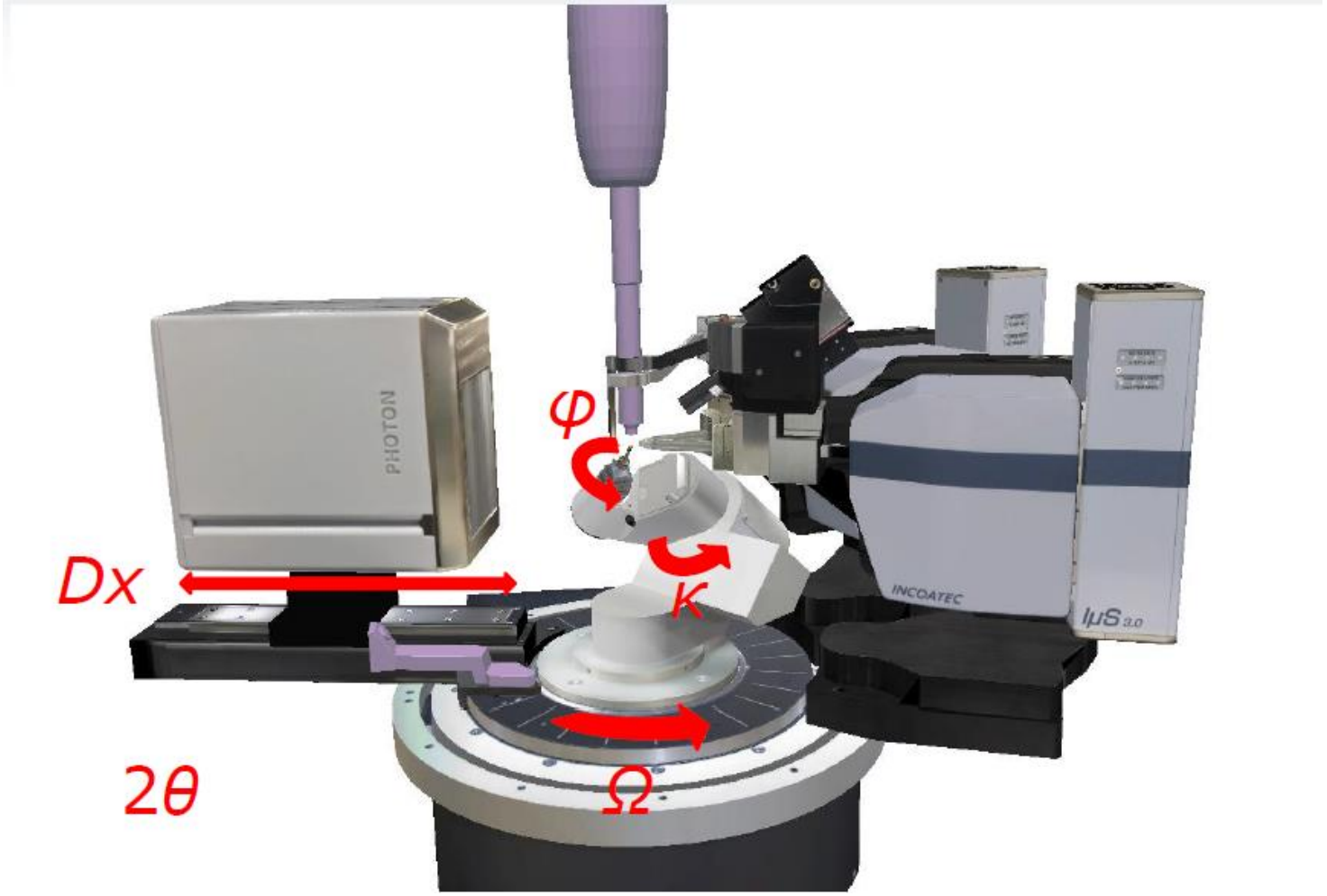


Eulerian Geometry

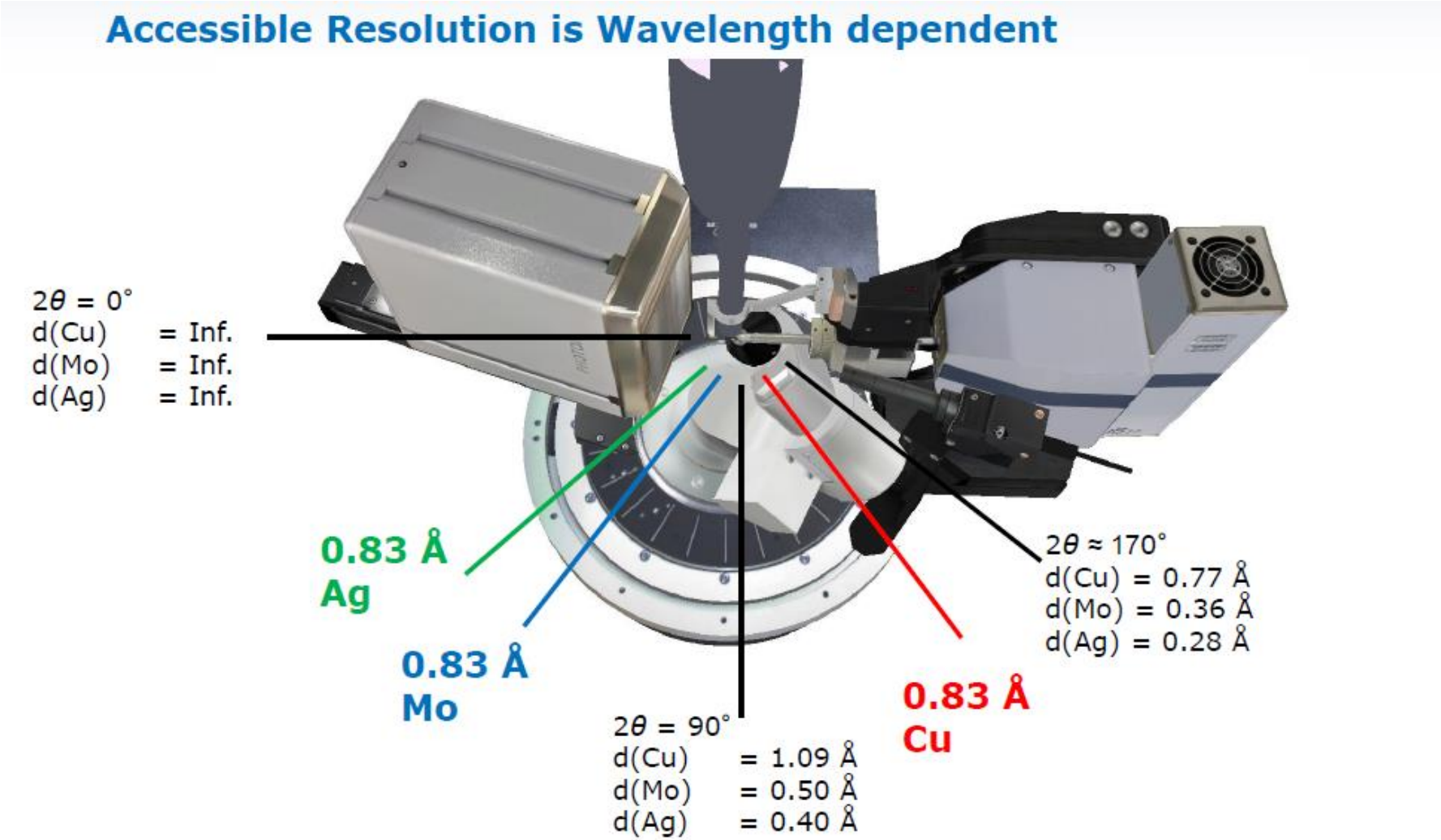


KAPPA Geometry

The Angles of SC-XRD

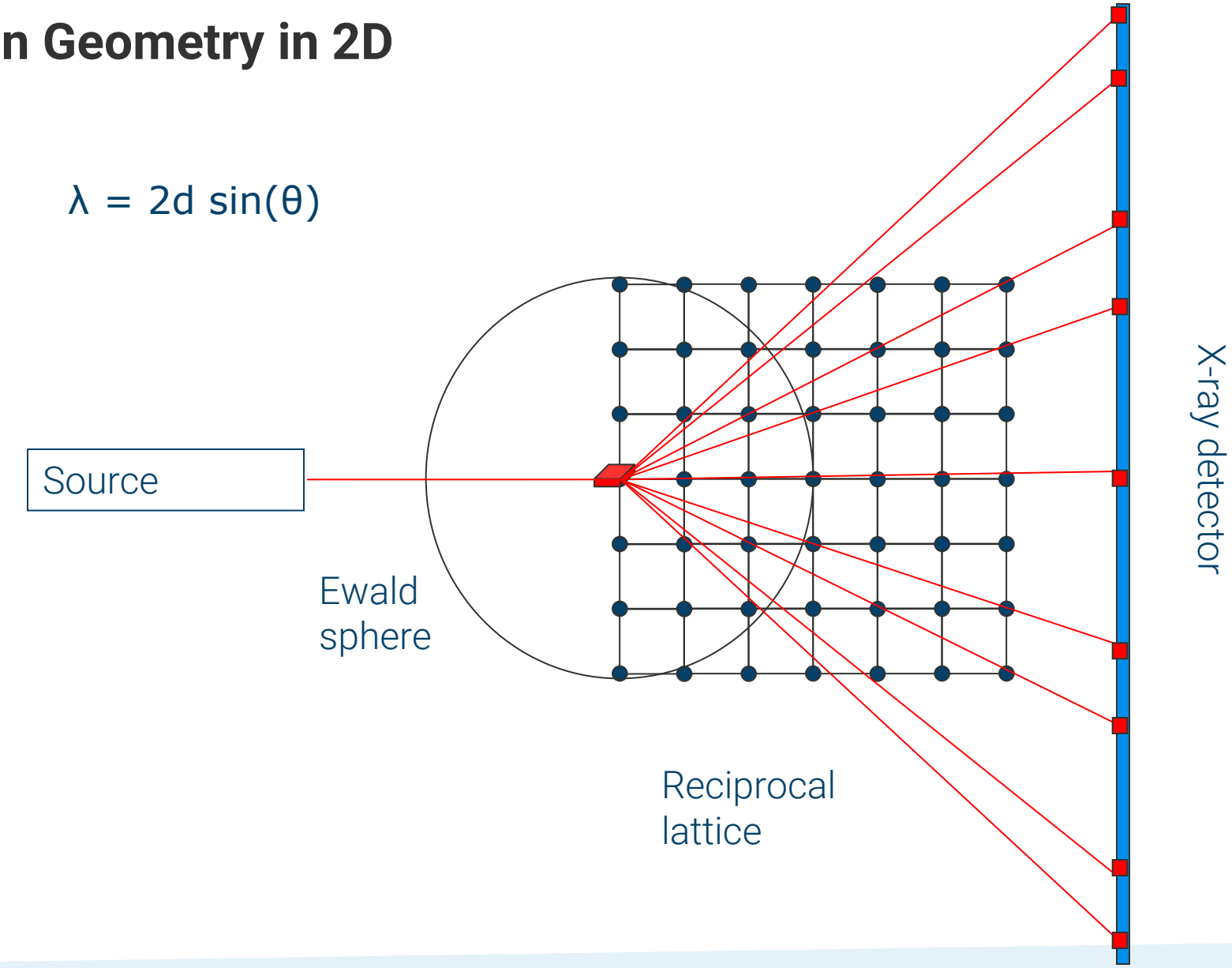


Wavelength and Source

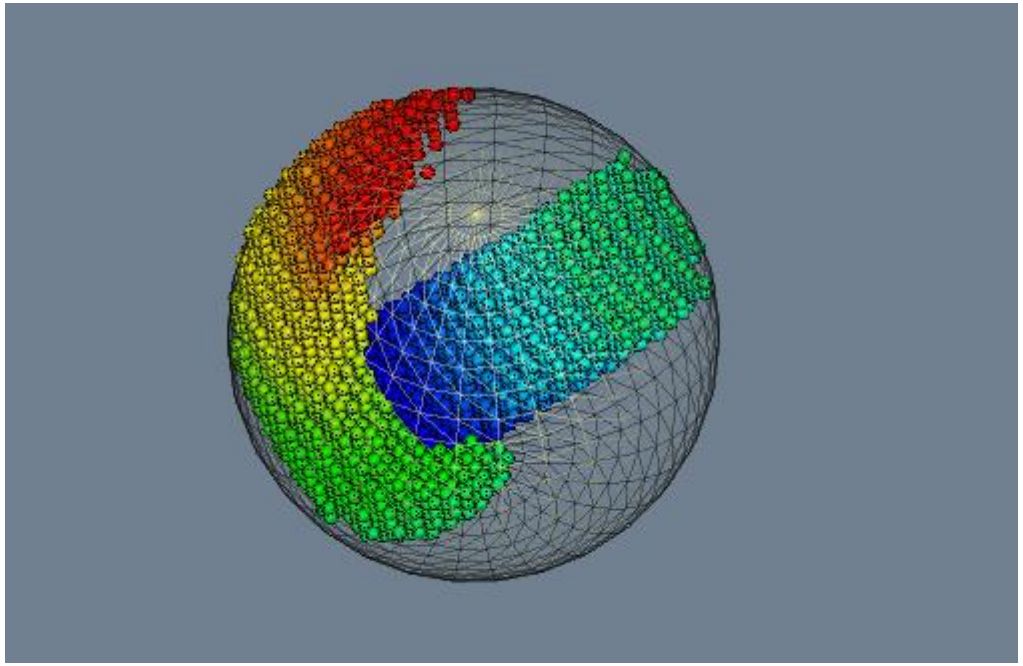


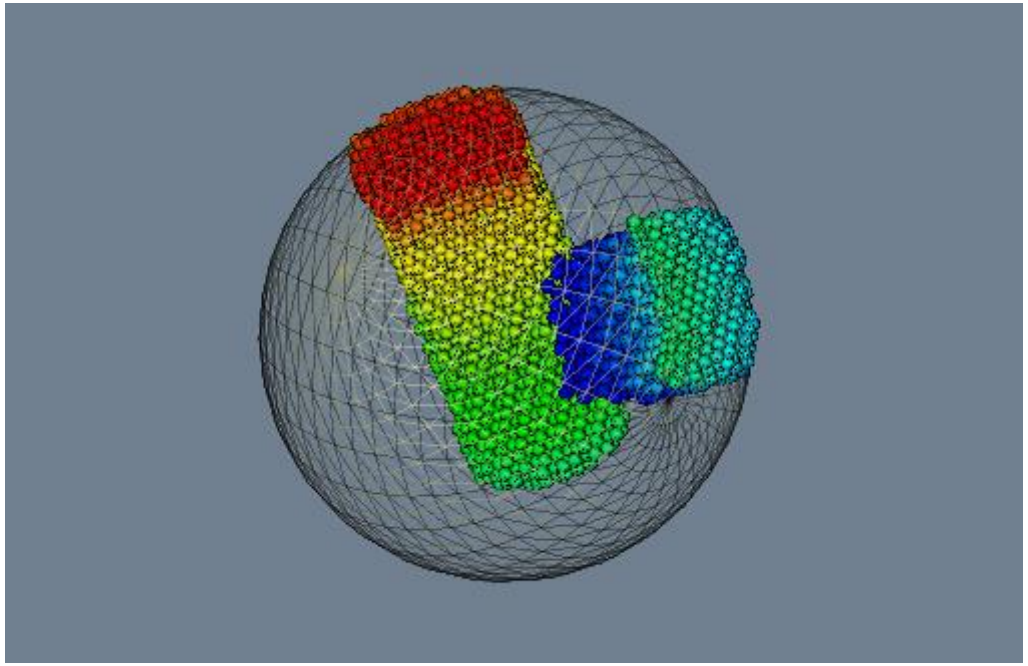
Diffraction Geometry in 2D

$$\lambda = 2d \sin(\theta)$$

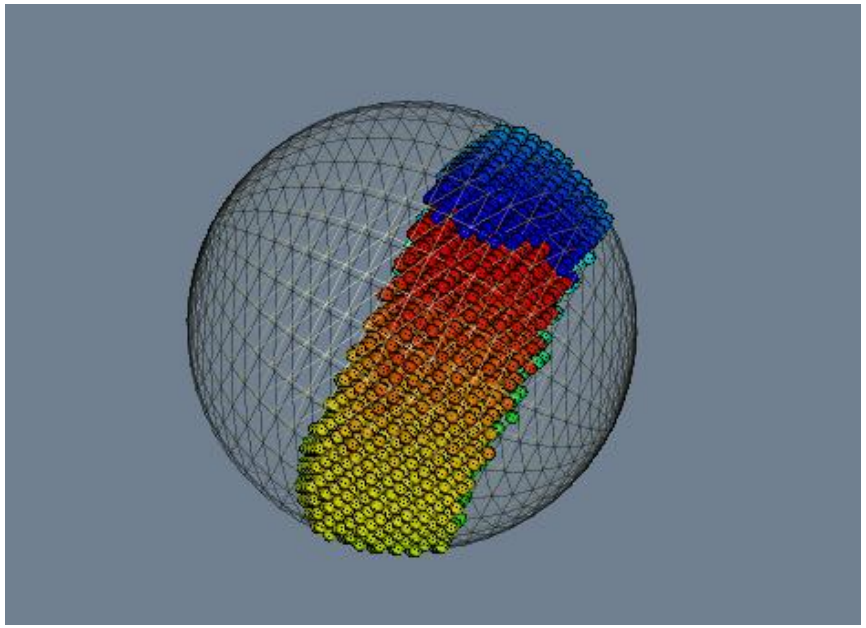


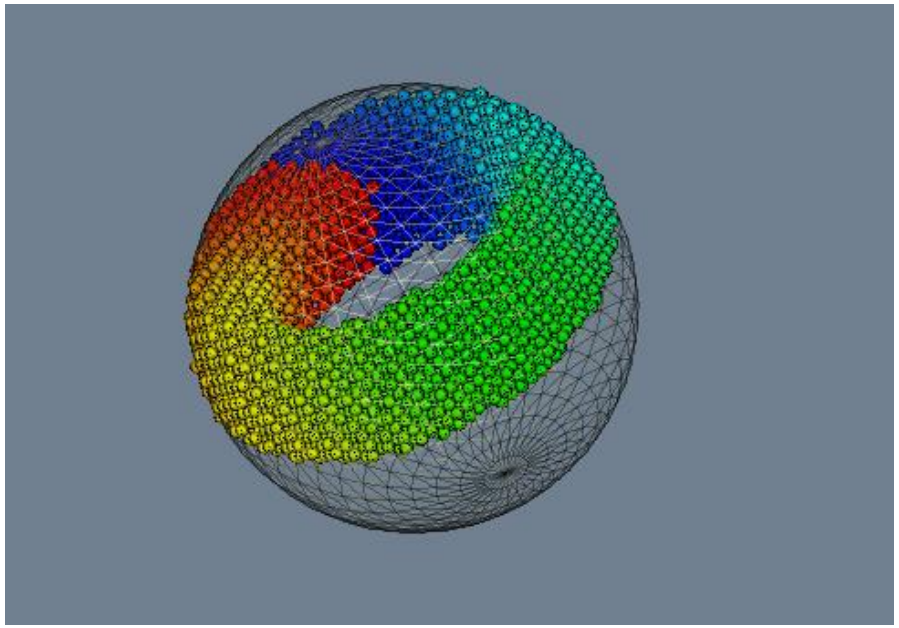
Omega scans





Phi Scan





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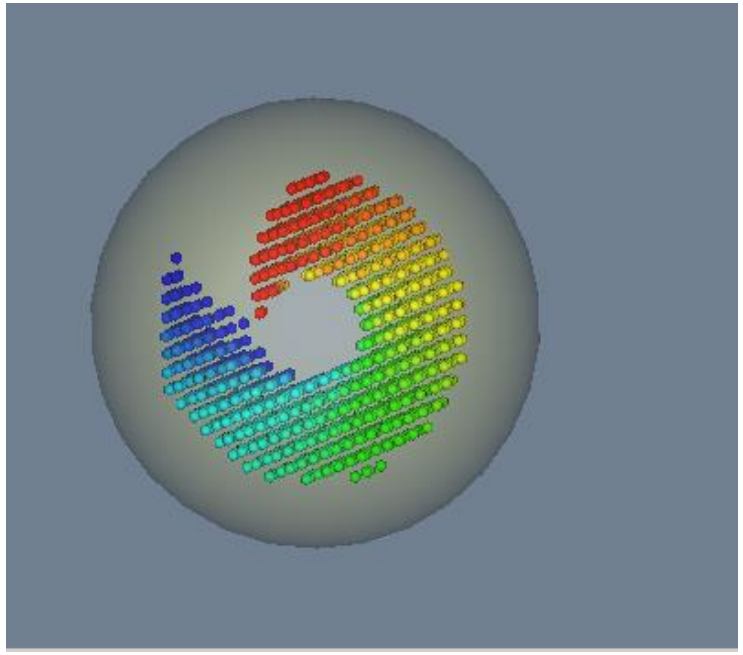
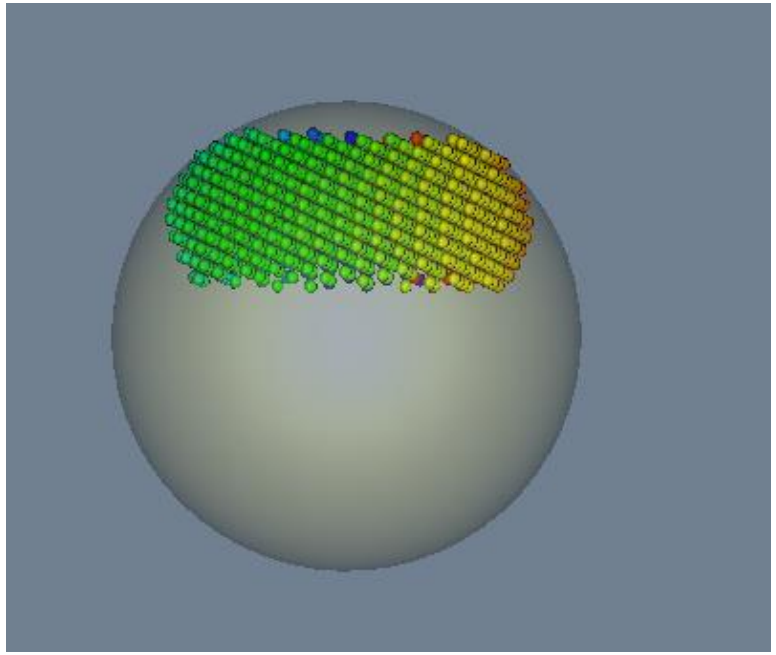
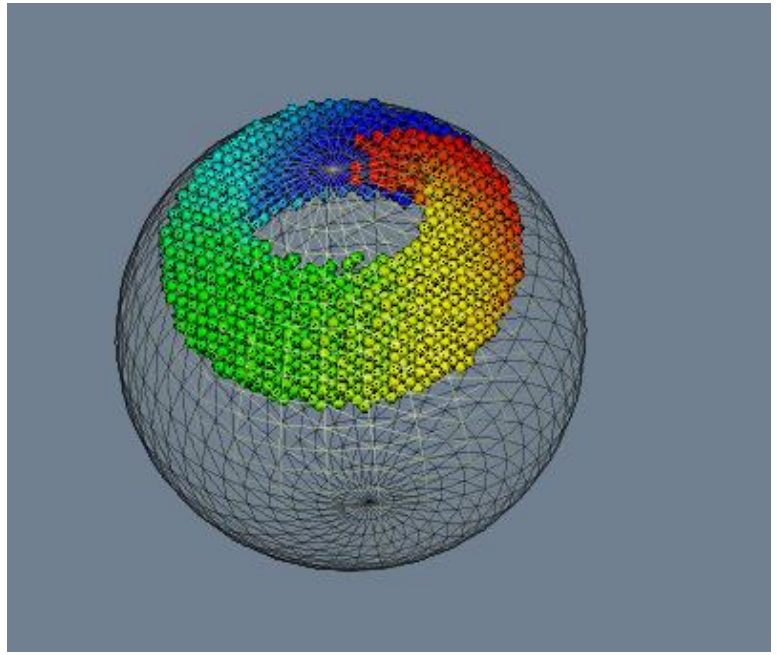
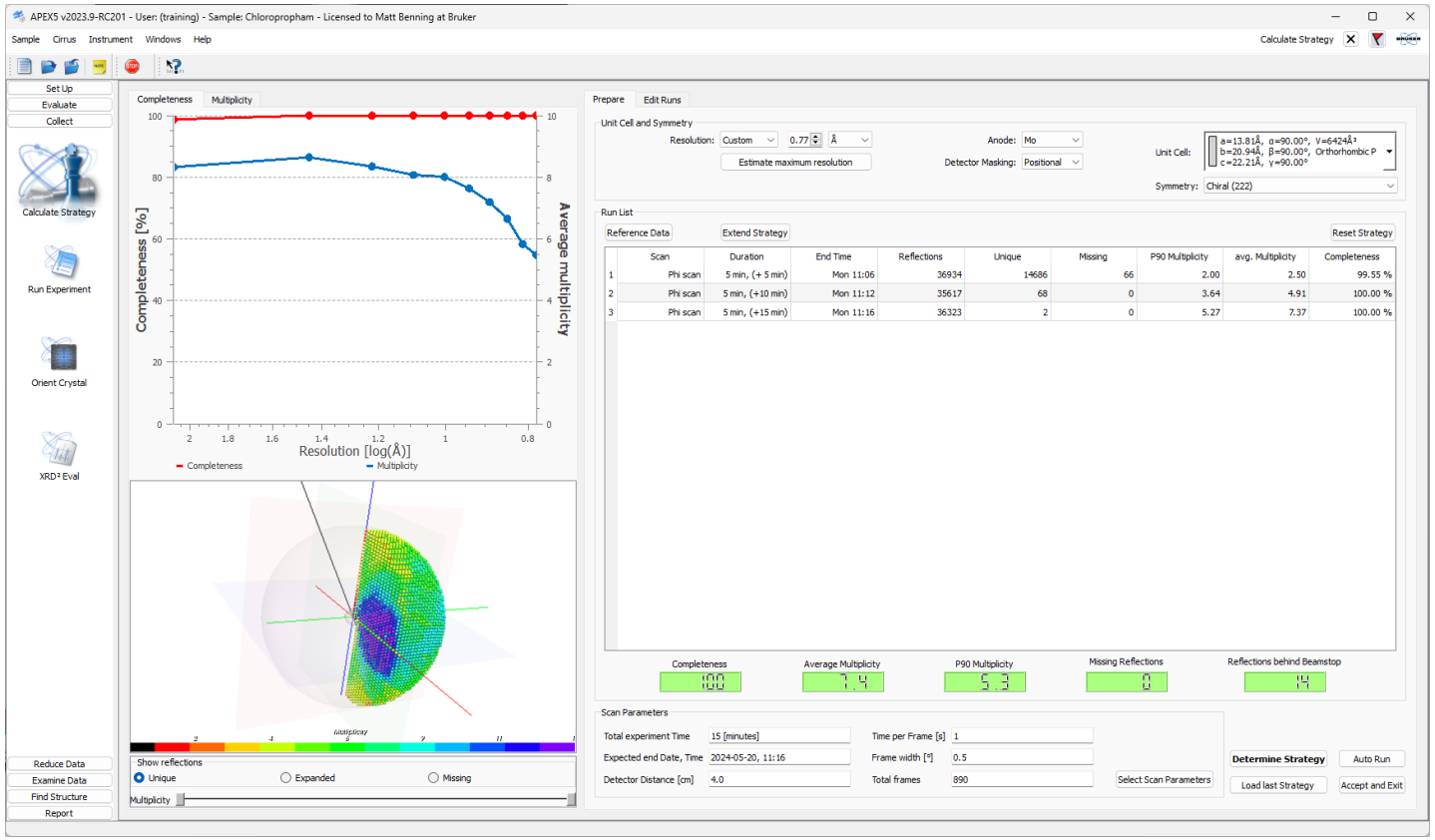






Automated Strategy Determination

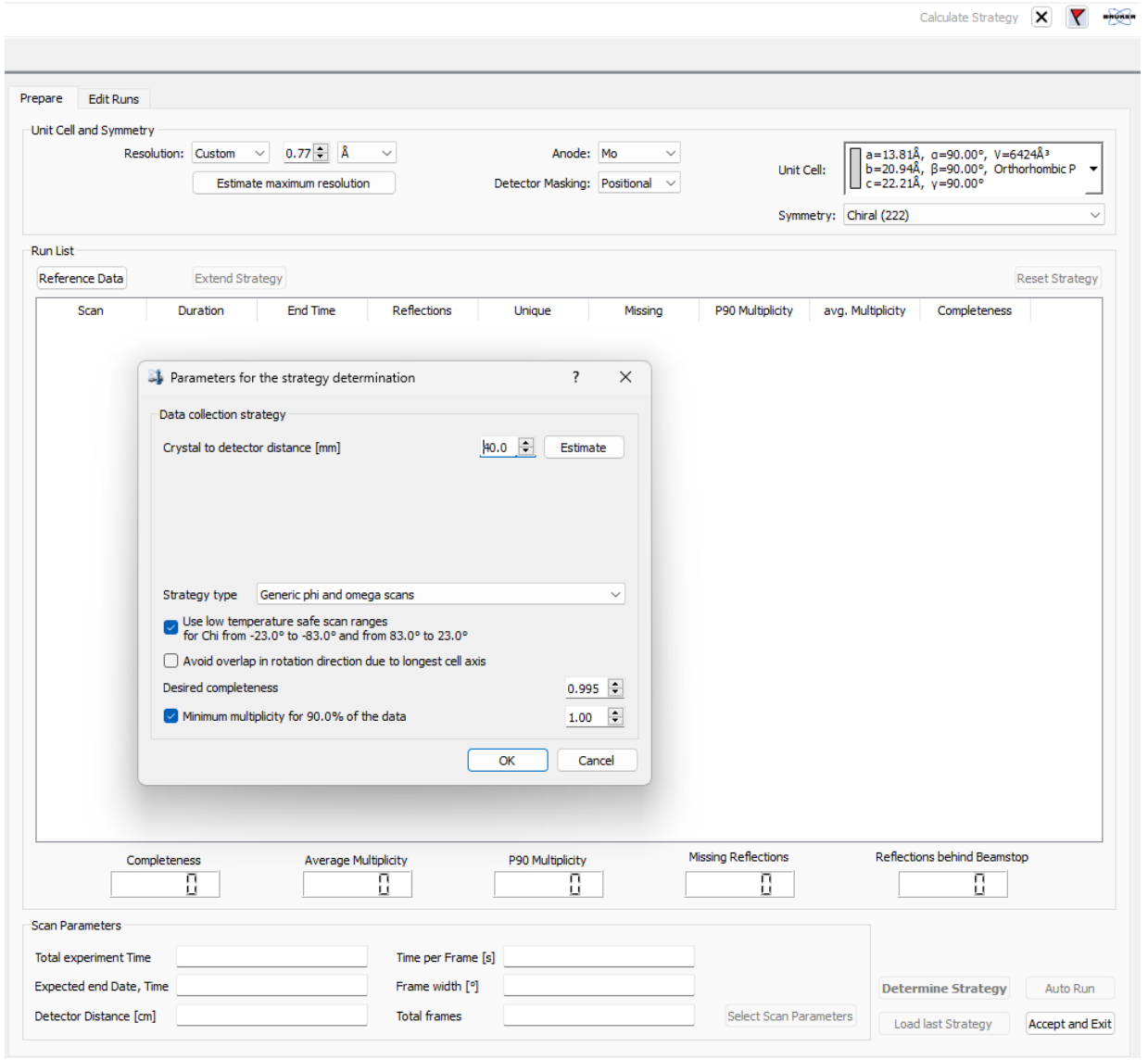


- Completeness
- Multiplicity
- Speed
- ETA

- What do you want from strategy?

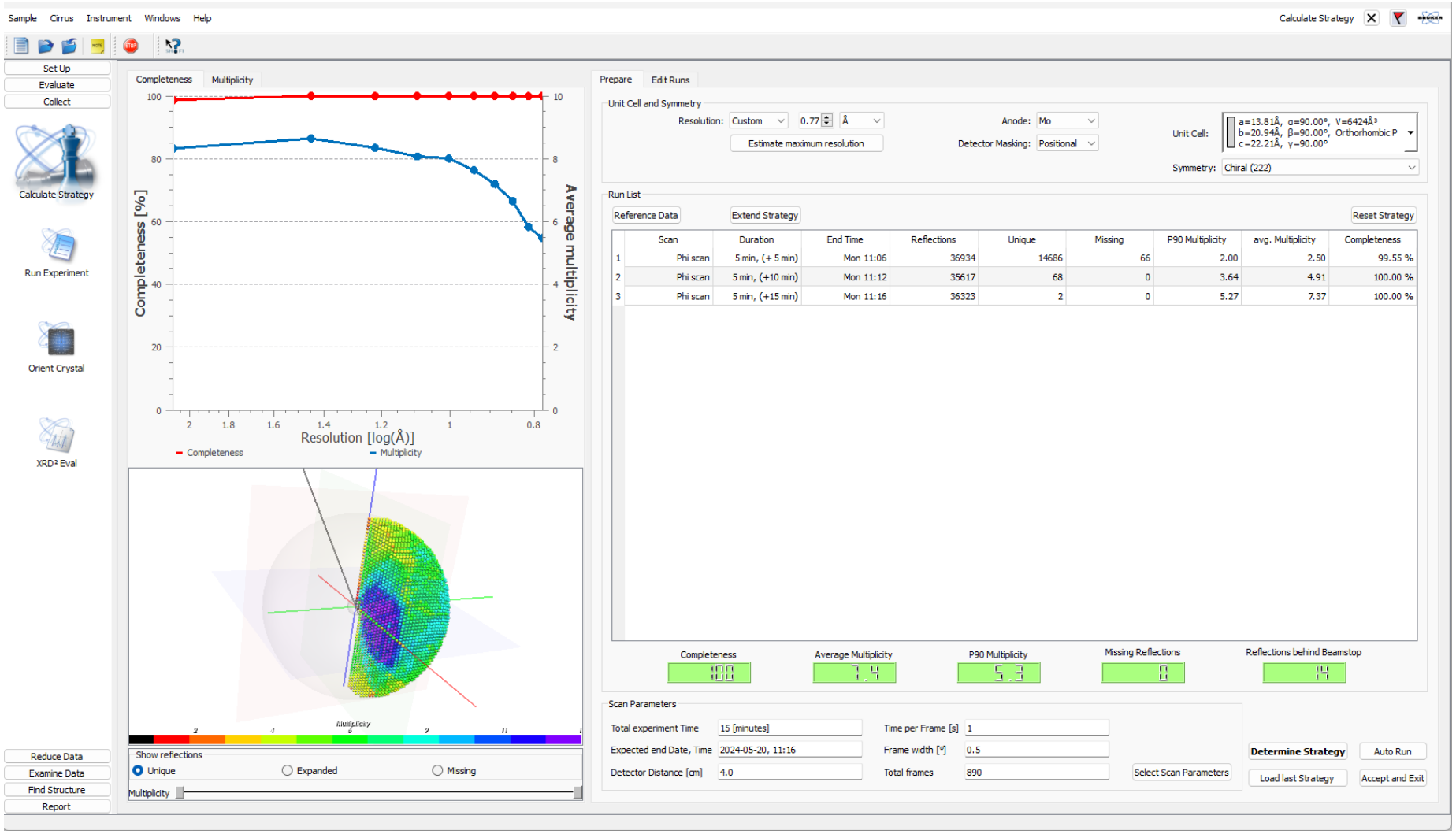
Data Collection Strategy

- Software reads in the cell and sets symmetry to lowest point group in crystal system
- Suggests wavelengths and resolution
- You can choose between different measurement types
- Distance is chosen taking unit cell and mosaicity into account
- You can set target completeness and multiplicity
- Use safe angles for low temp data
- Intelligent defaults work well



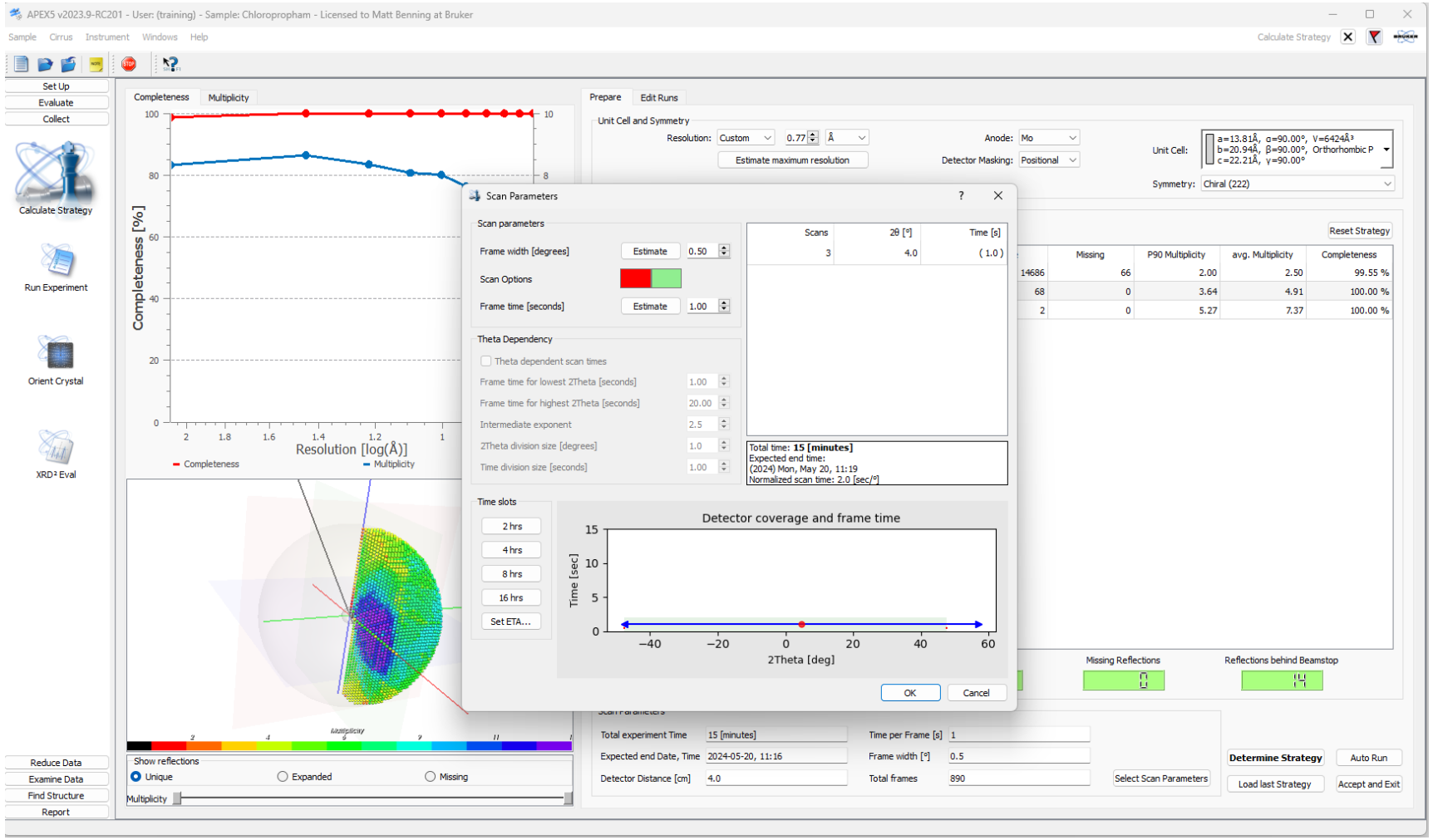
Data Collection Strategy

- Run calculation
- Statistics



Data Collection Strategy

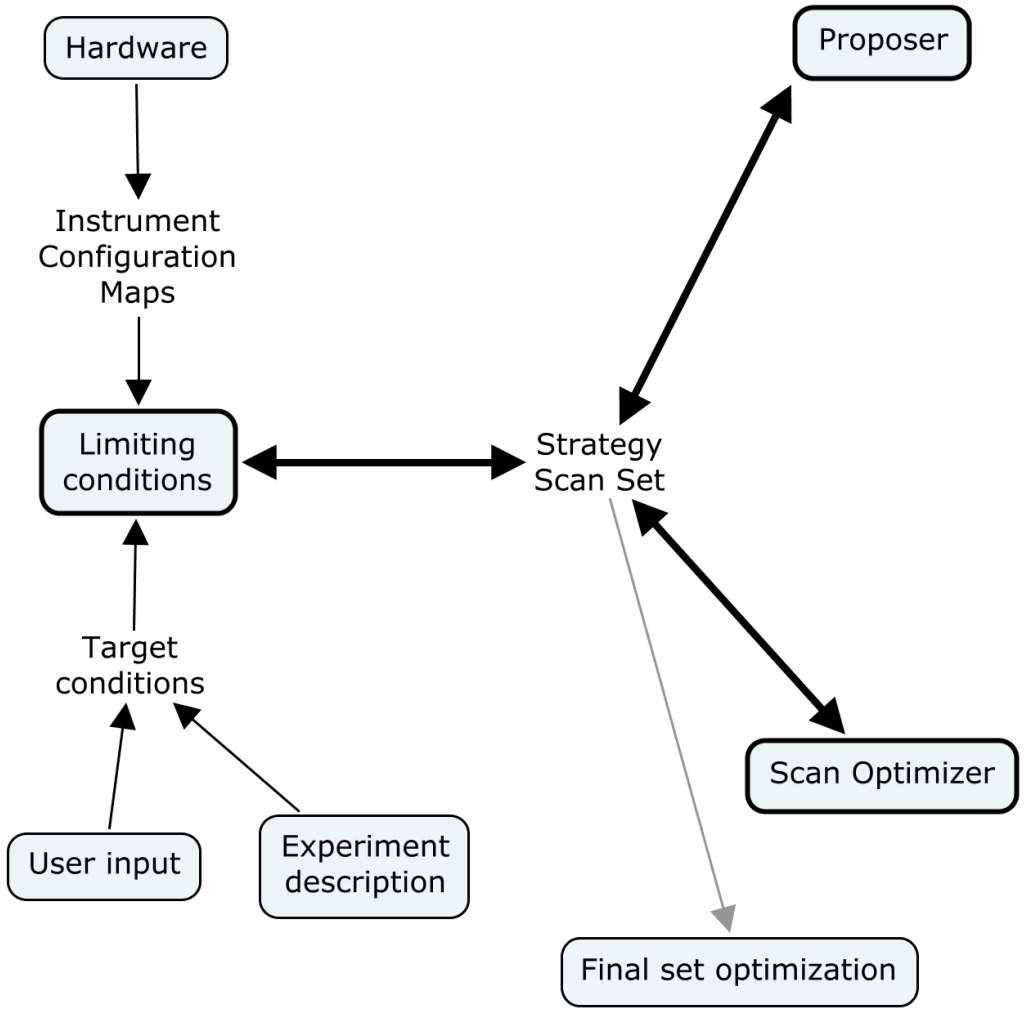
- Scan parameter selection
- Software will estimate
- Frame angle
- Frame time
- 2θ dependent data collection
- Time slots
- Set ETA



Challenging Cases

- Data Set Completeness
  - Crystals with Low Symmetry (Triclinic or Monoclinic)
  - Large Unit Cells (i.e. Large Detector Distances)
  - Unfavorably Mounted Crystals (Spindle || Reciprocal Axis)
  - Blind Detector Regions (Beam Stop, Cryo Nozzle, DAC, …)
- Data Set Redundancy
  - Absorption Correction
  - Anomalous Data (MAD, SAS, SIR, …)
- Data Collection Time
  - Crystal Decay
  - Strict Time Limit
  - Data collection slots

Scan generation
How does it work?



- Form clusters of missing data.
- Turn the clusters into Omega and Phi scans.
- Select the most efficient scan, i.e. the one that covers the largest number of reflections per degree of rotation.
- Repeat

DATA COLLECTION AND REDUCTION WITH APEX6

Data Integration with APEX6

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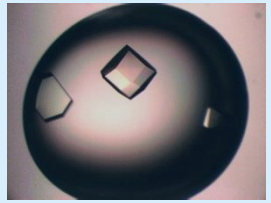
SCD Workflow

5 – 15 GB



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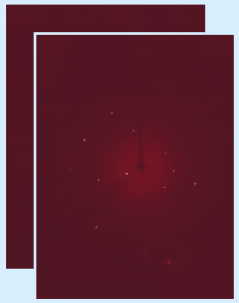

Diffraction
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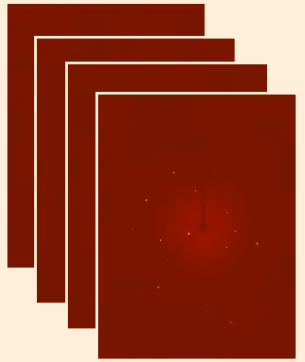

Full set of Diffraction
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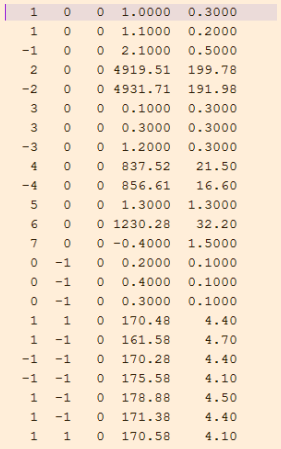
Reduction

PC



HKL Intensity
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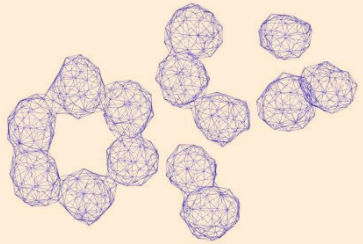
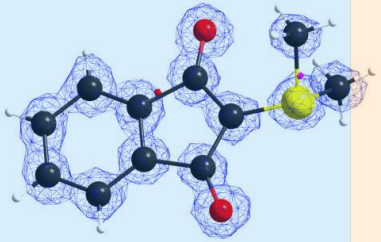
Structure
Solution



Electron
Density Map

Structure
Refinement

< 1 MB



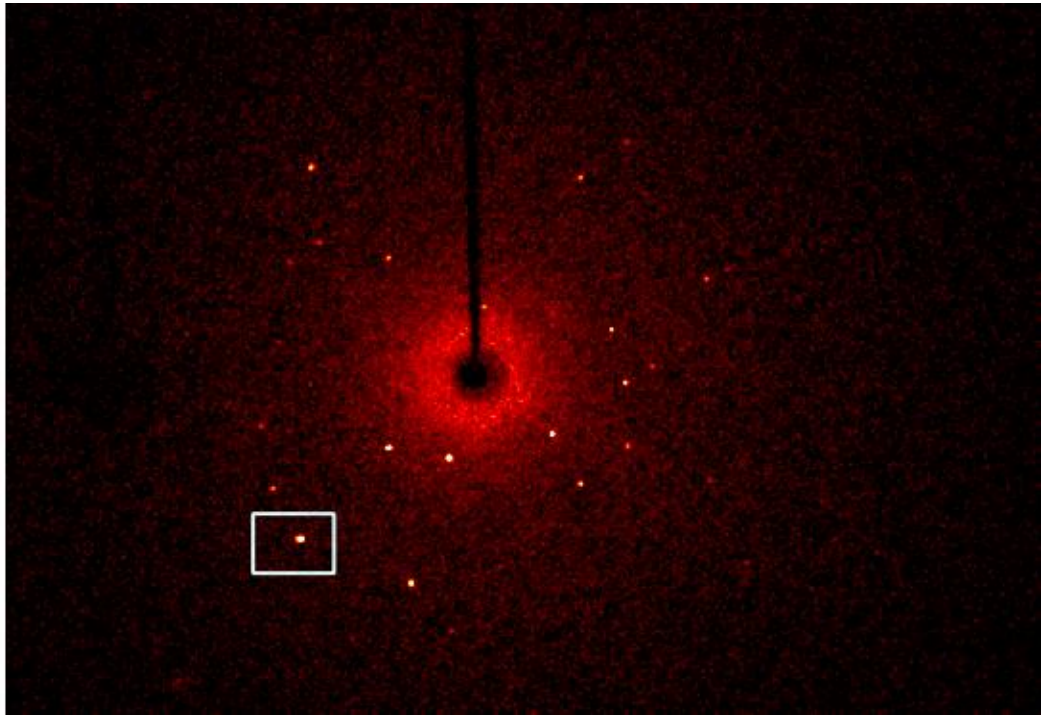
Atomic Model

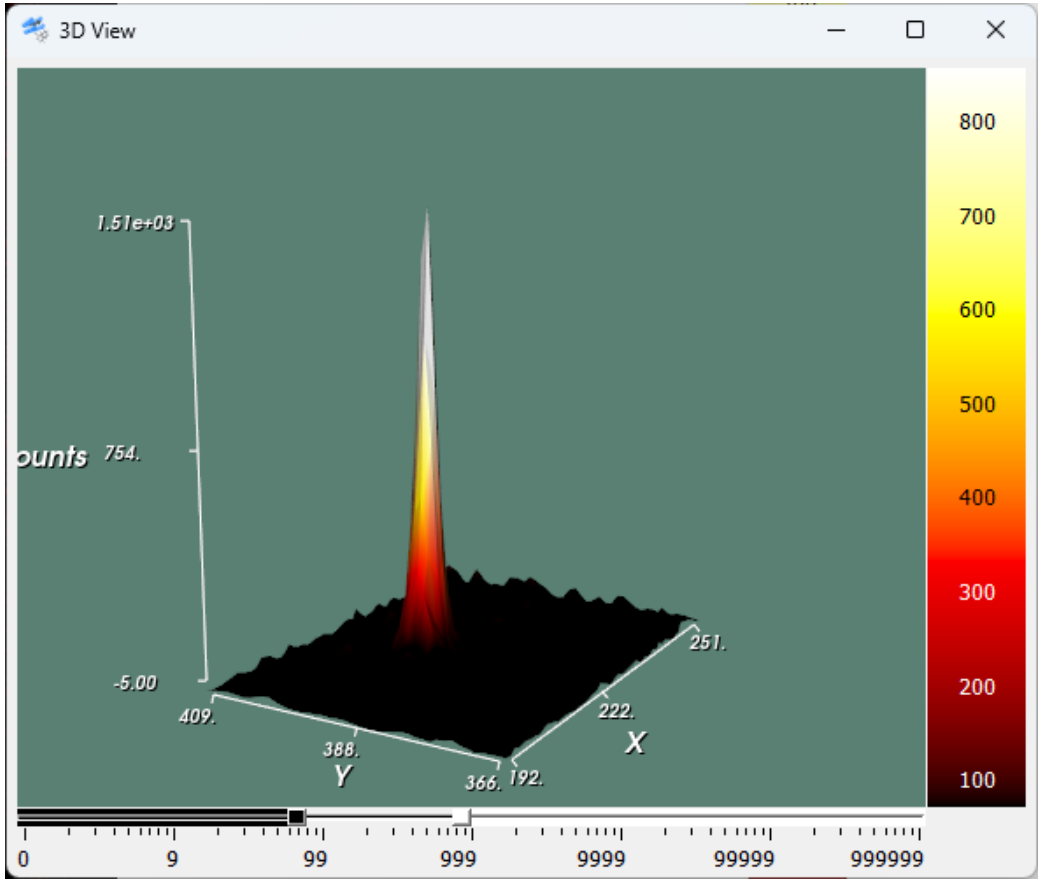
Validation

Publication

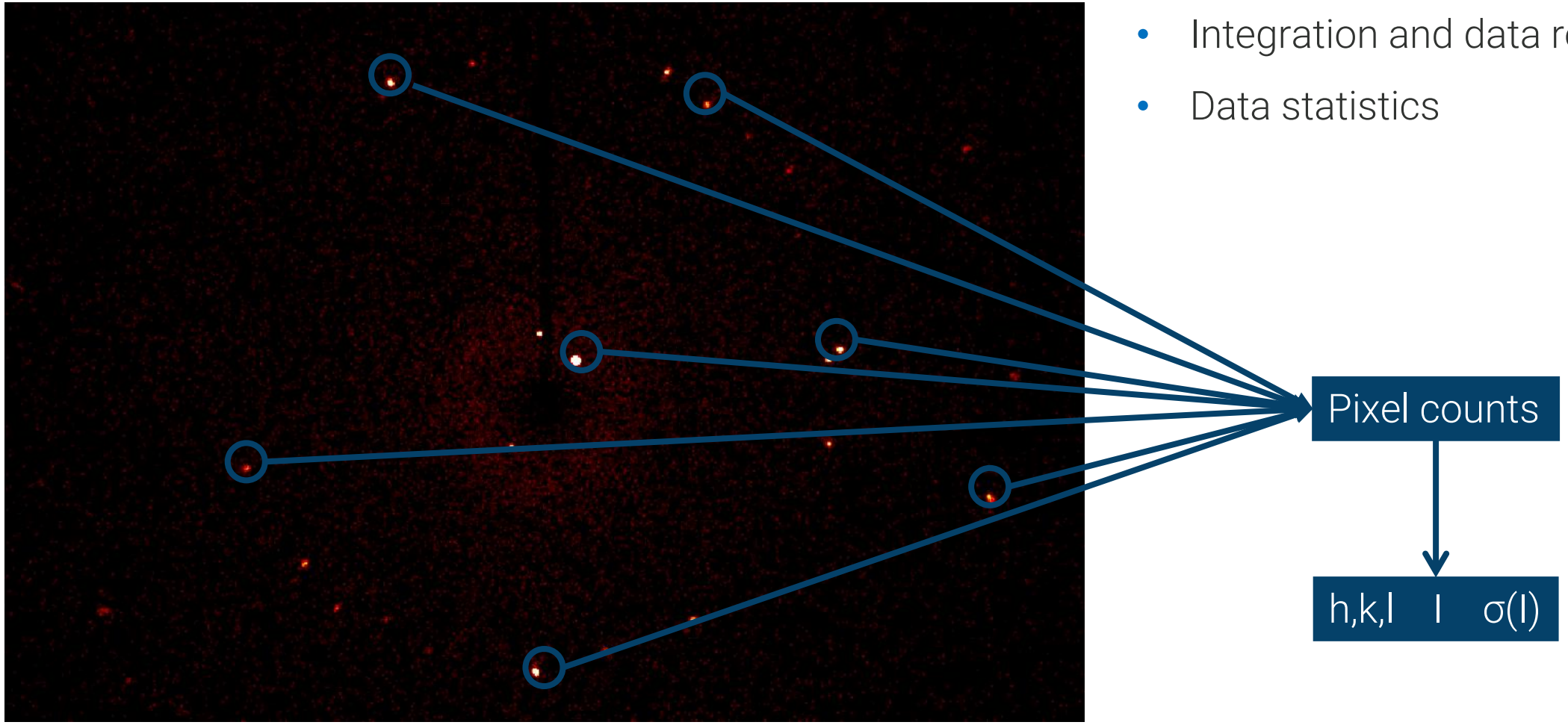
Human

3D View of a Reflection





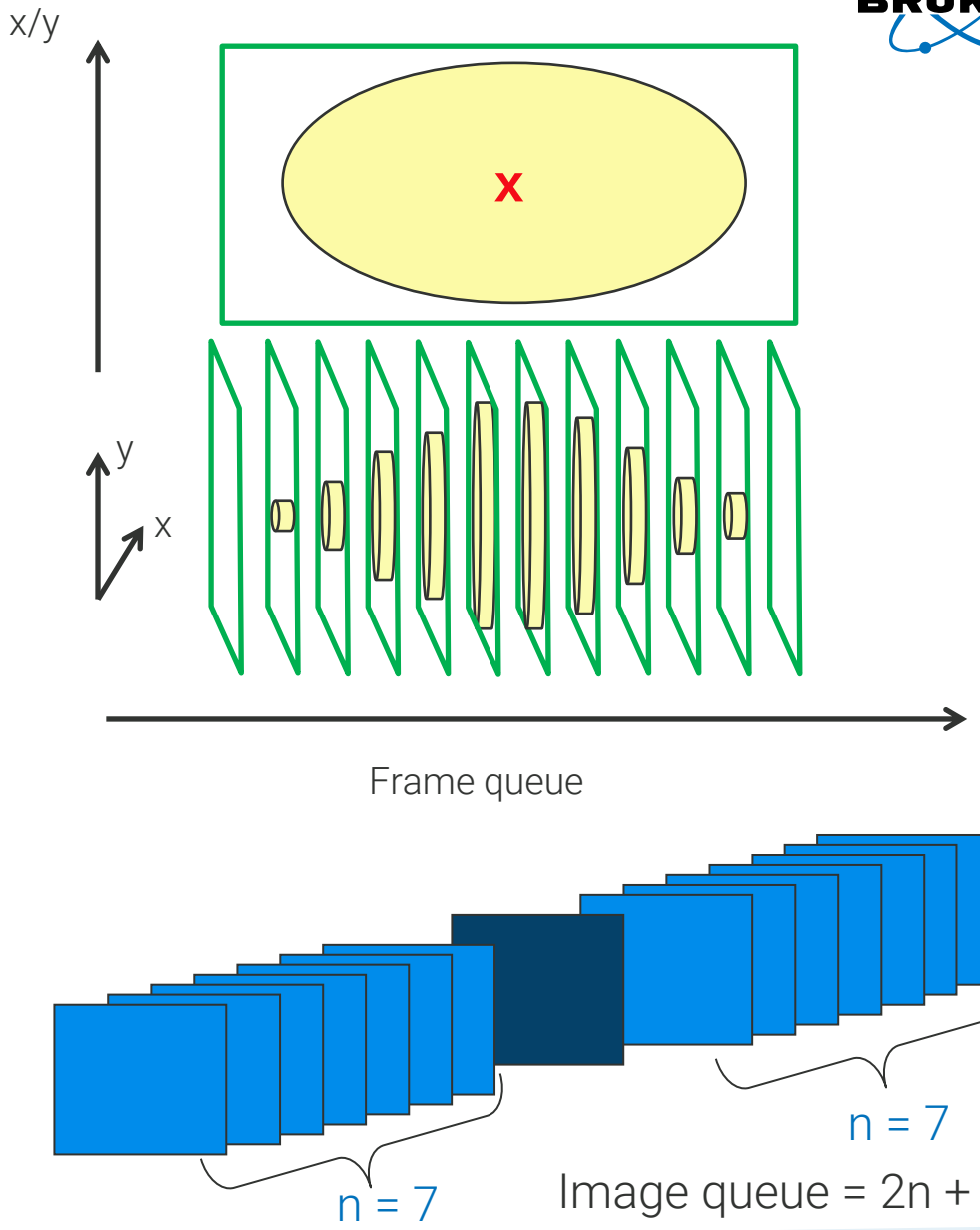
What Does SAINT (Integration Plug-In) Do?



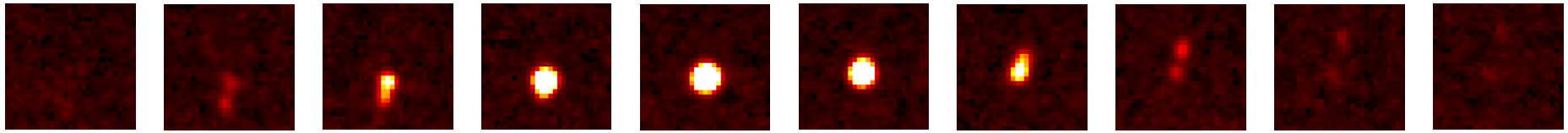
- Integration and data reduction
- Data statistics

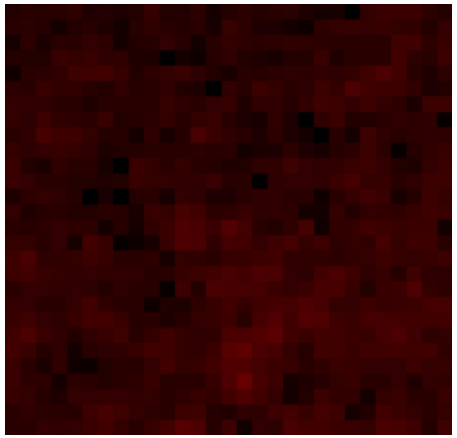
Data Integration – Steps in Integration

- Which Steps are involved?
  - Determining the background
  - Find out which frames belong to the reflection
  - Is the information complete?
  - Load all required frames in the buffer
  - “Center” the reflection, refine unit cell
  - Determine the best integration box size
  - Analyze the reflection profile
  - Determine the intensity
  - Determine the standard deviation
  - (Re-)Refine the Orientation Matrix



Spot Integration

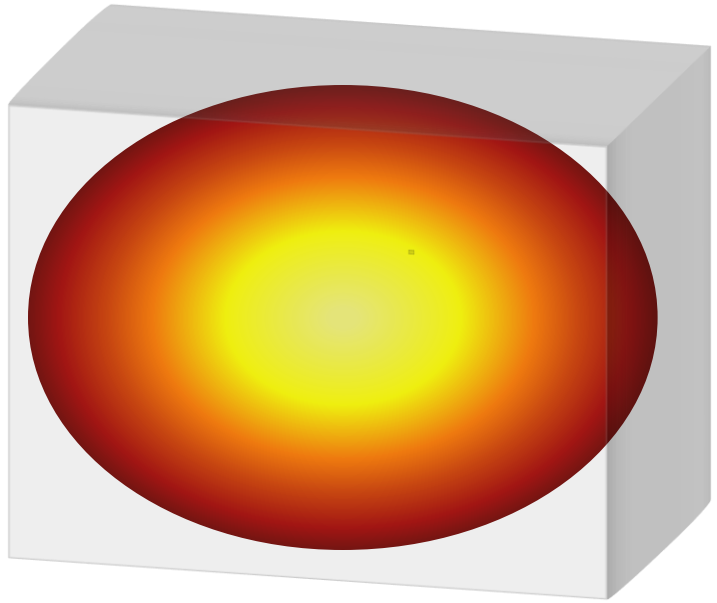




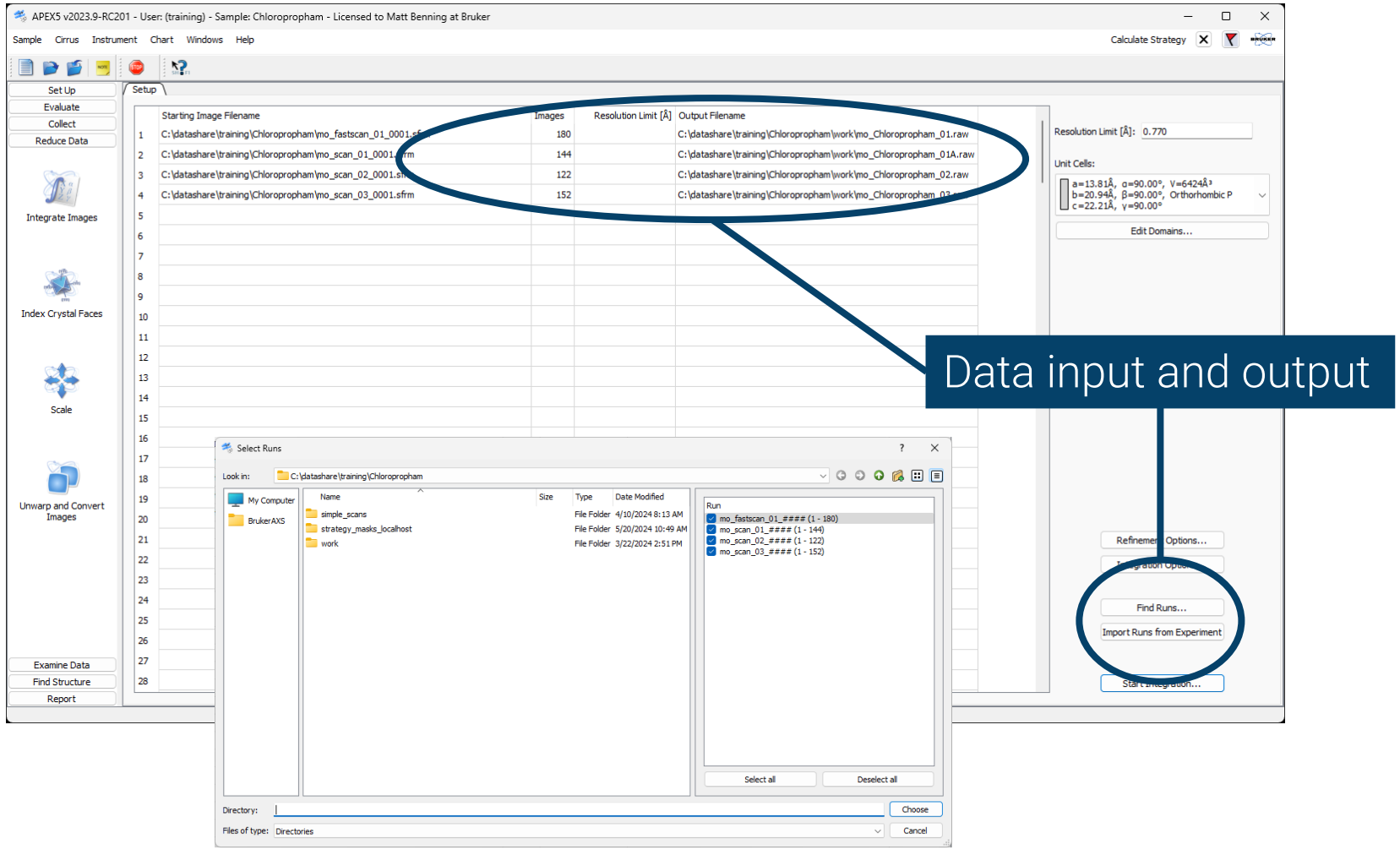
H	K	L	Intensity	σ(intensity)
−1	−5	4	247774.	6934.

Learned Profiles

- Integration box
- Profile
- Model profiles are built as averages of strong, well-behaved reflections

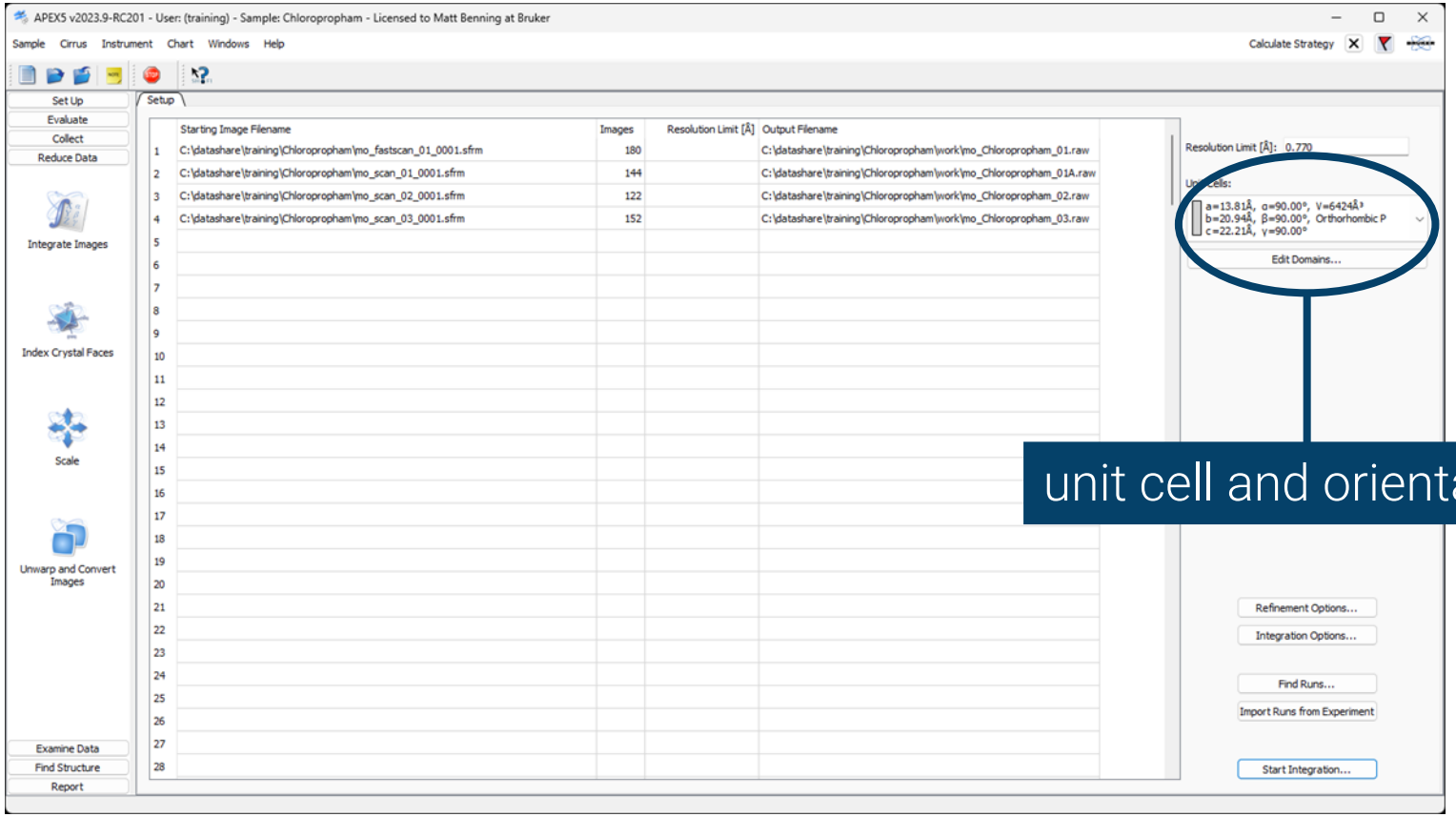


SAINT GUI – Run Selection



- Easy to use
- Supports twinning
- Supports incommensurates
- Intelligent defaults

SAINT GUI – Run Selection



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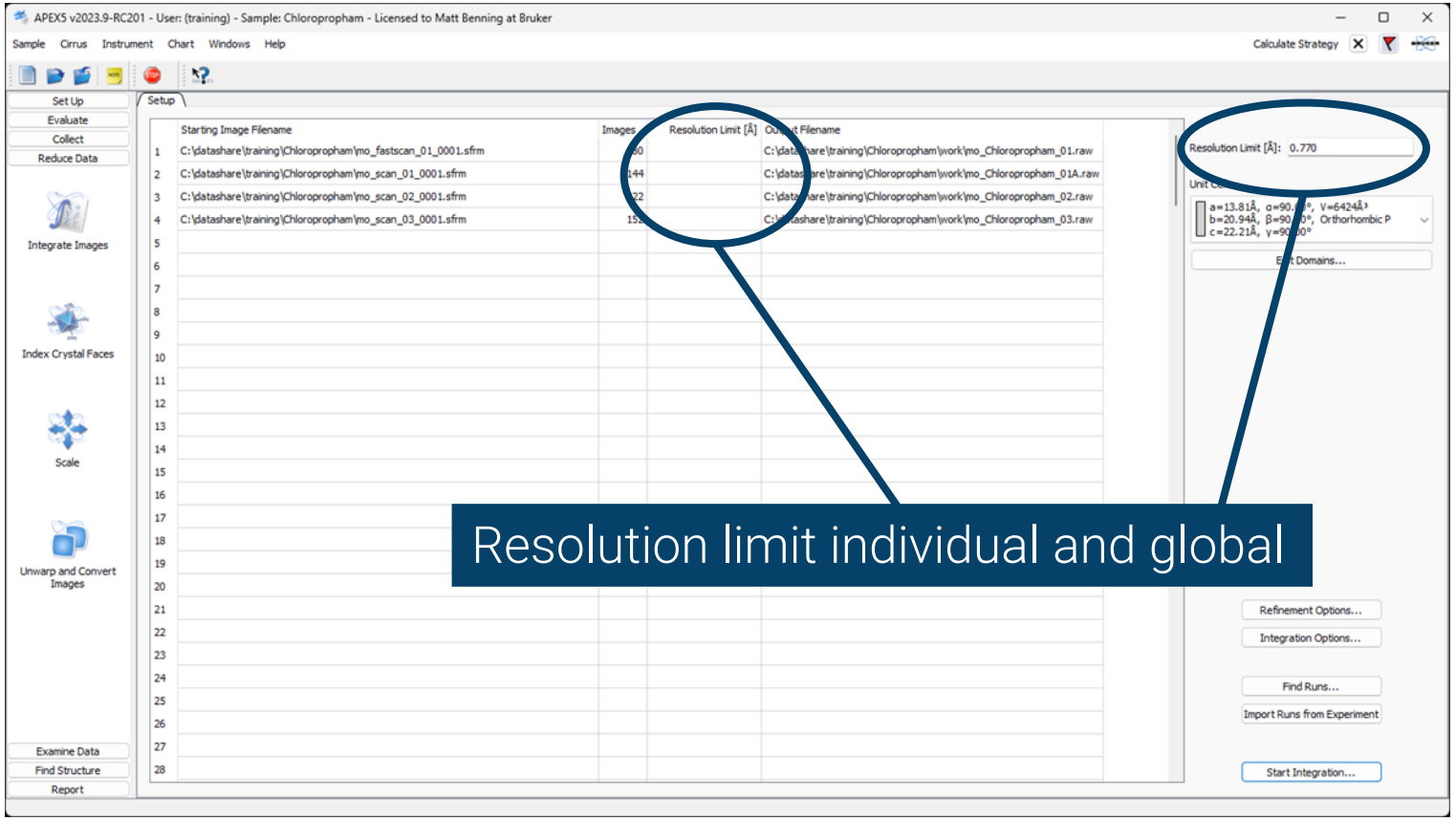
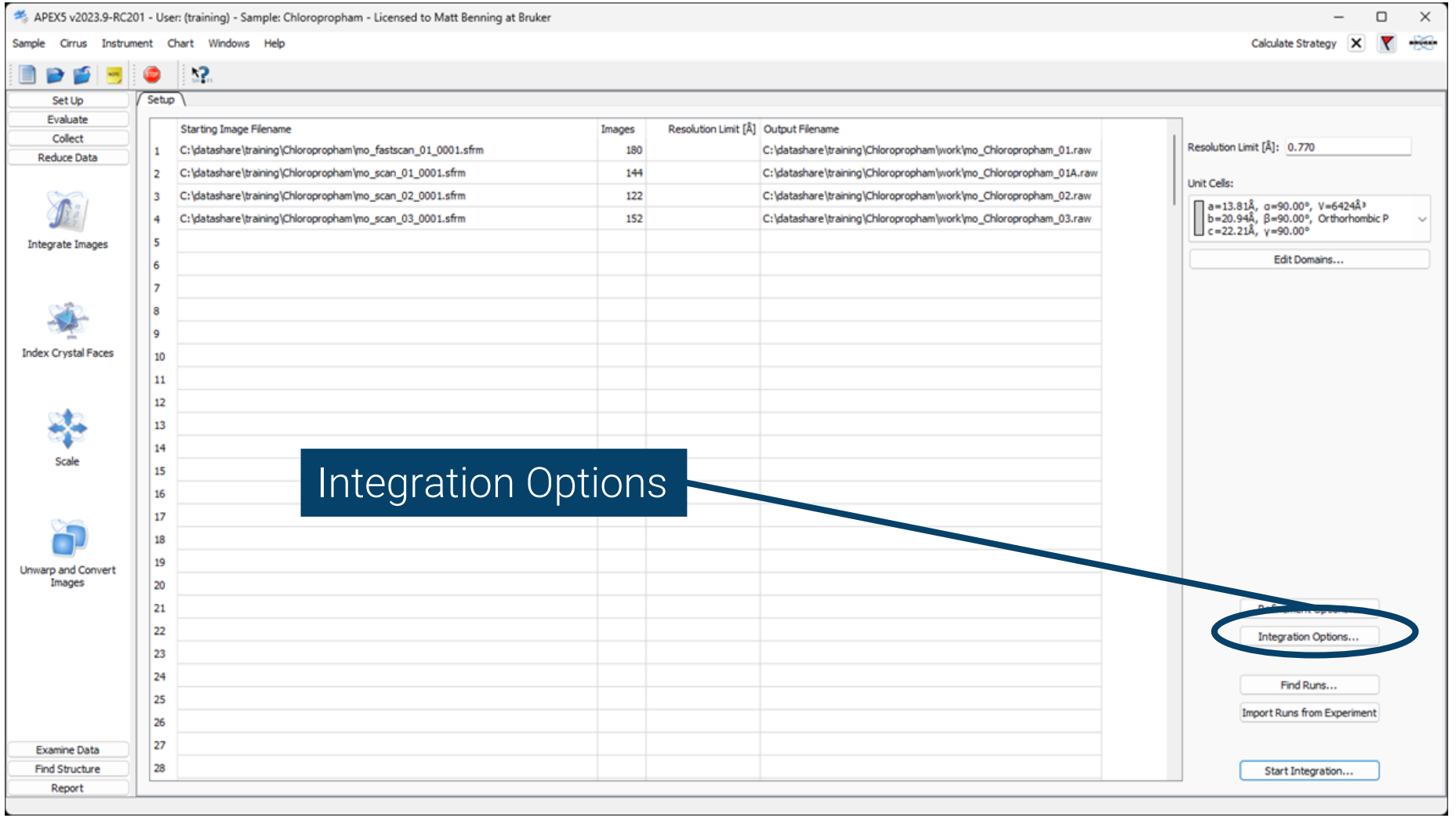
SAINT GUI – Run Selection

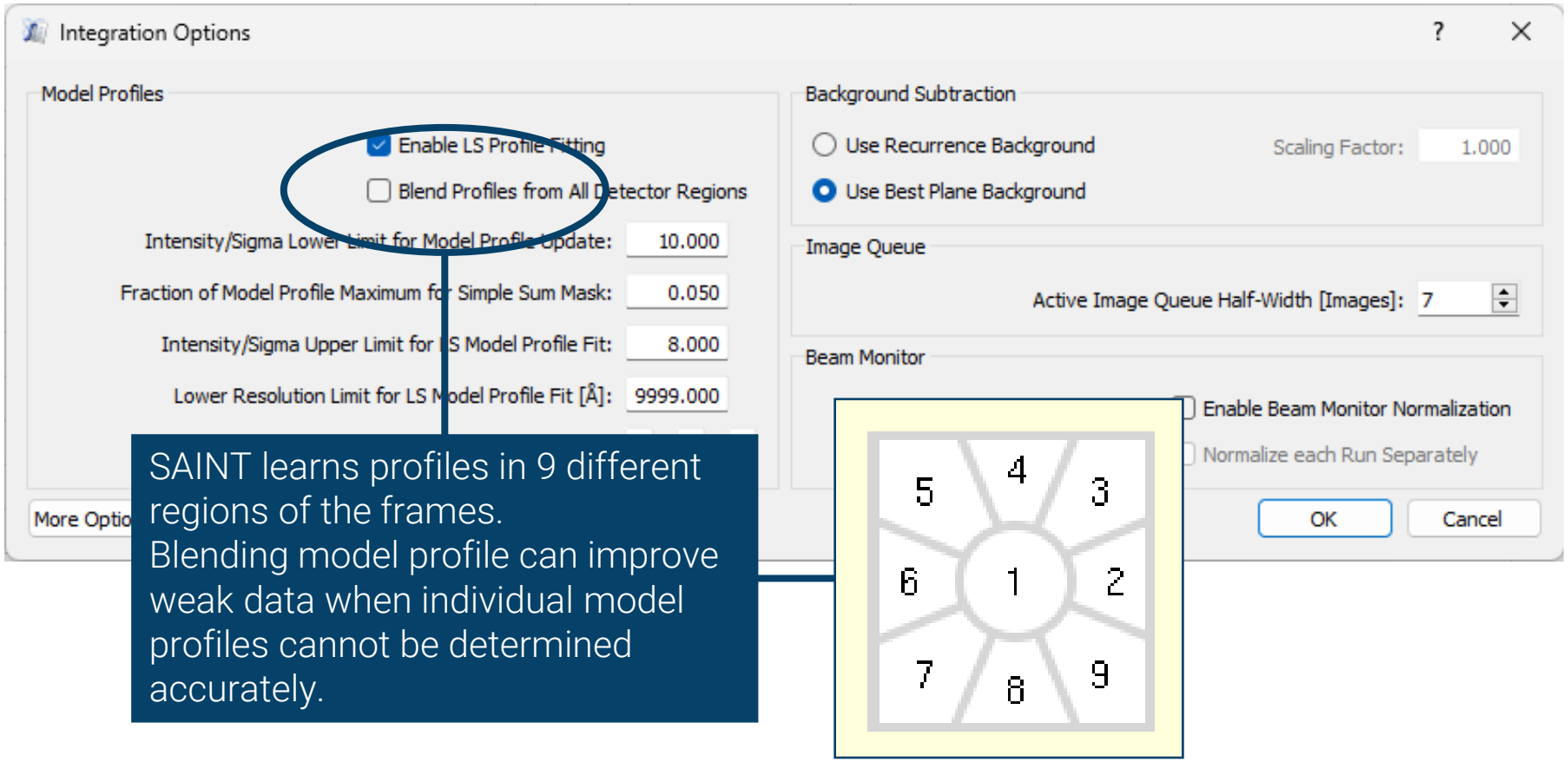


- Choose a reasonable resolution limit!
- Repeat after looking at data statistics
- Recycle the orientation matrix (import *m.p4p)
- Don’t integrate noise

SAINT – Integration Options

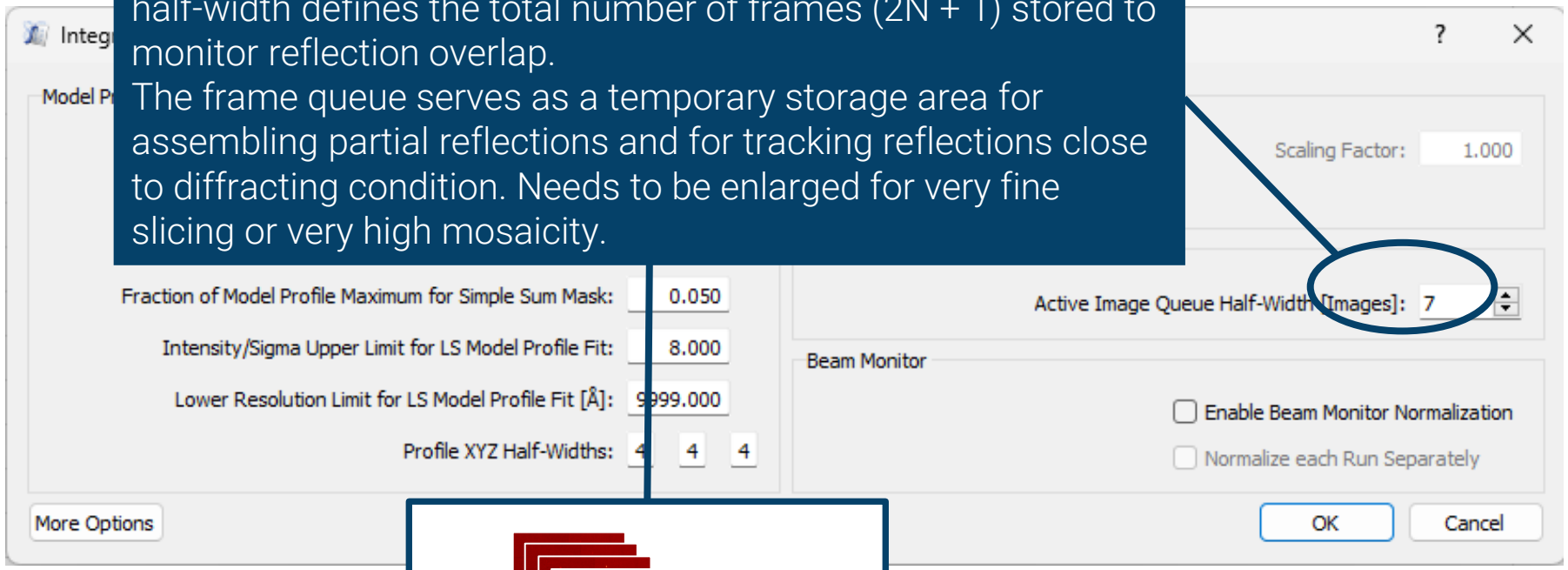


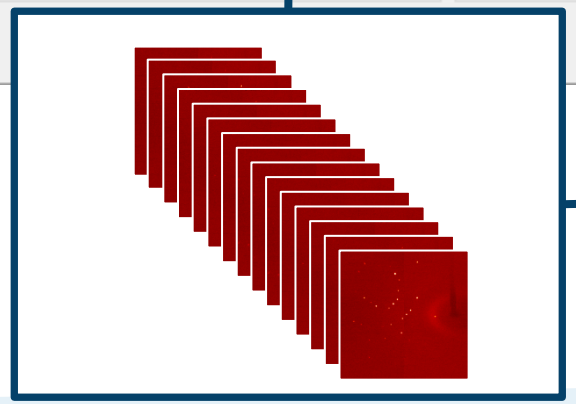
SAINT – Integration Options



SAINT – Integration Options

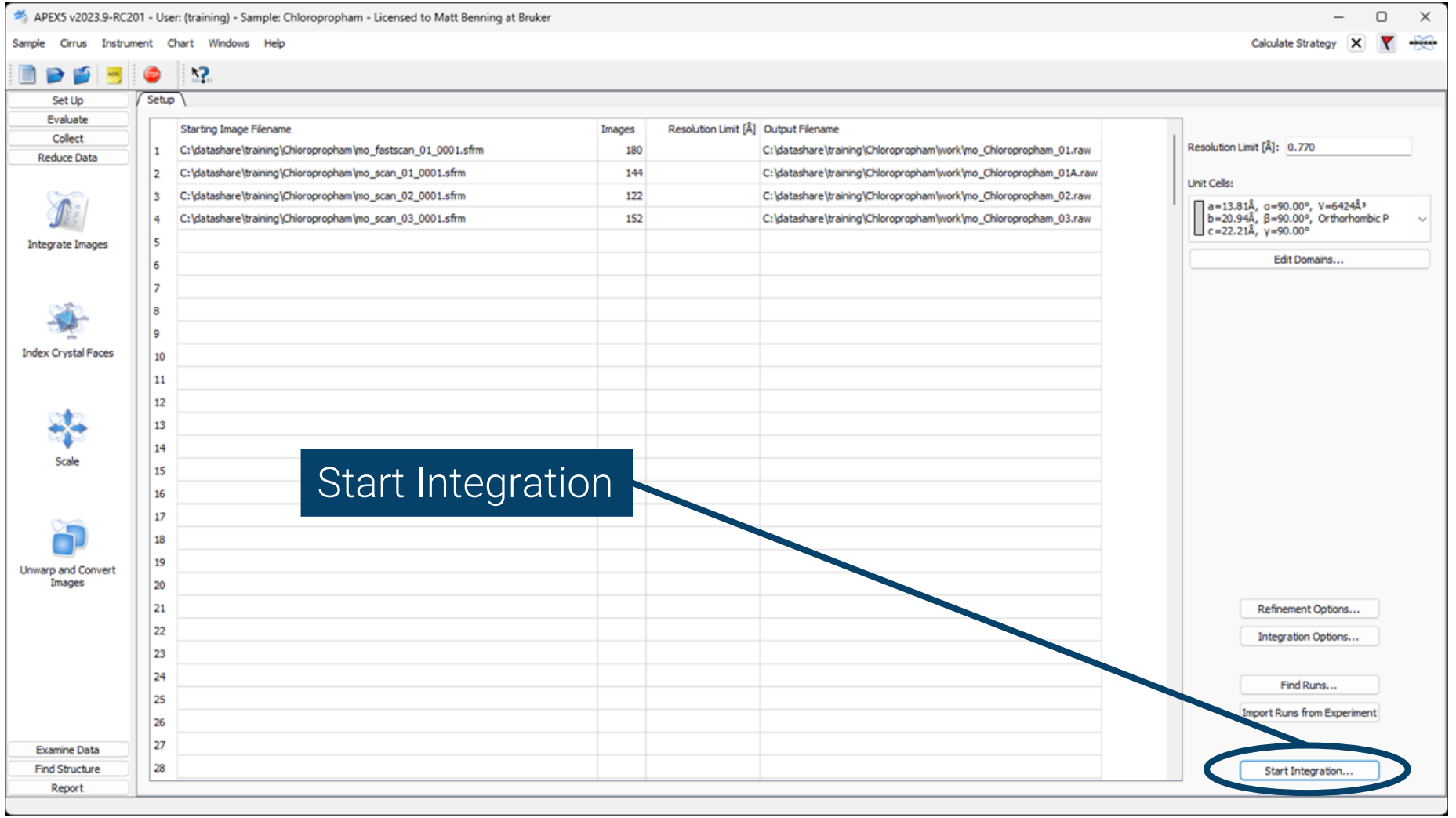
Size of the frame queue stored in memory. Active frame queue half-width defines the total number of frames (2N + 1) stored to monitor reflection overlap.
The frame queue serves as a temporary storage area for assembling partial reflections and for tracking reflections close to diffracting condition. Needs to be enlarged for very fine slicing or very high mosaicity.



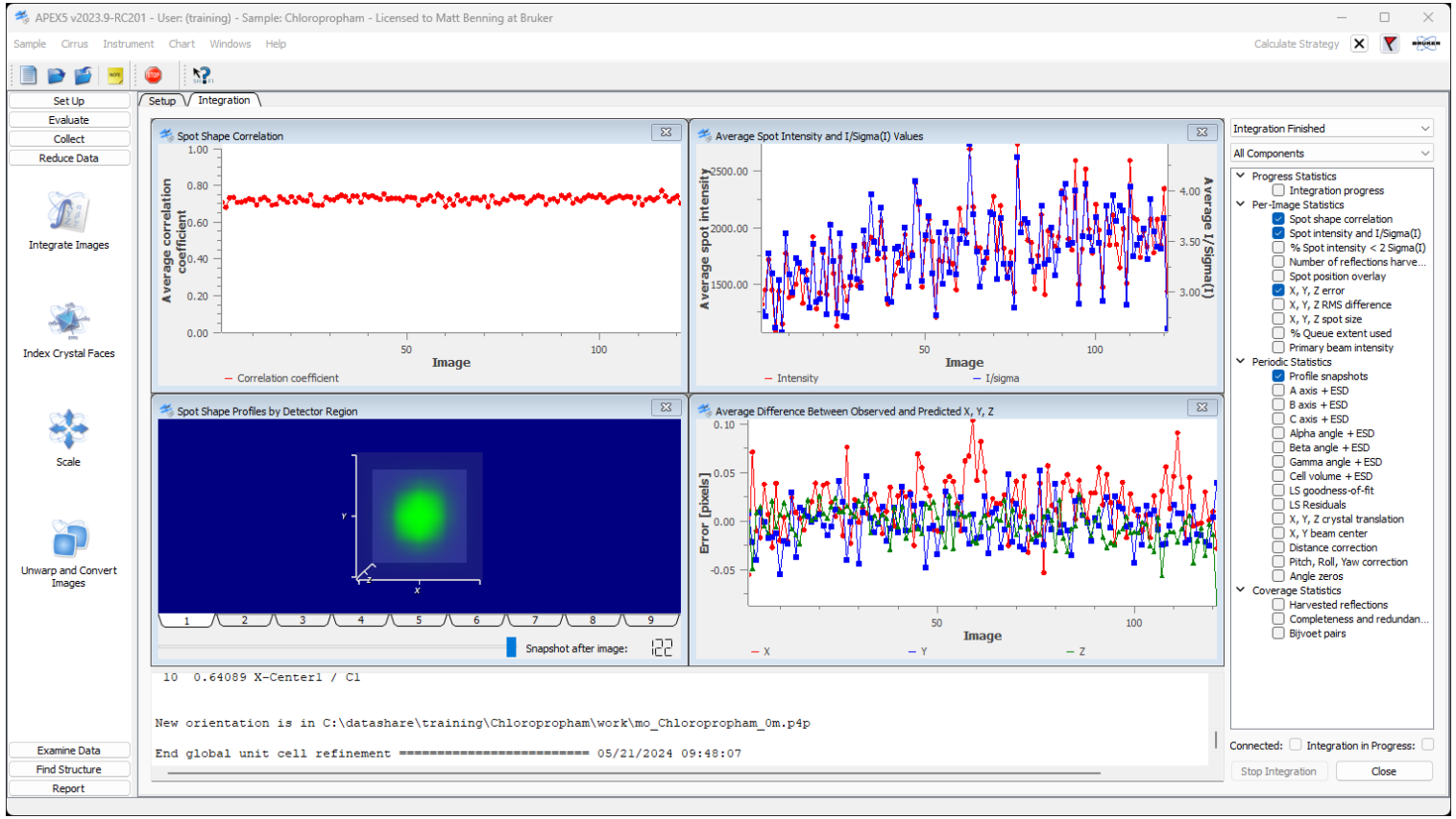


Frame queue 2N + 1 Half-width = 15 frames

SAINT – Let‘s Integrate

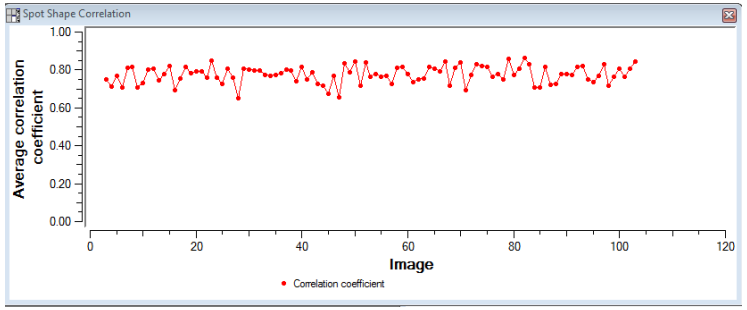


SAINT CHART

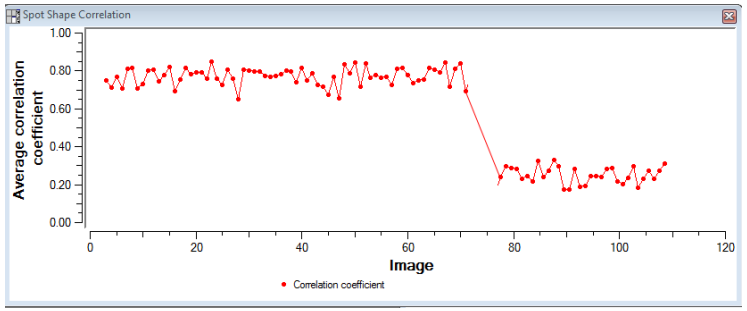


- Most important parameters at a glance
- Spot correlation should be high and not change much during the scan
- If model profiles are white a default profile is used – consider blending
- X-Y-Z errors should not drift

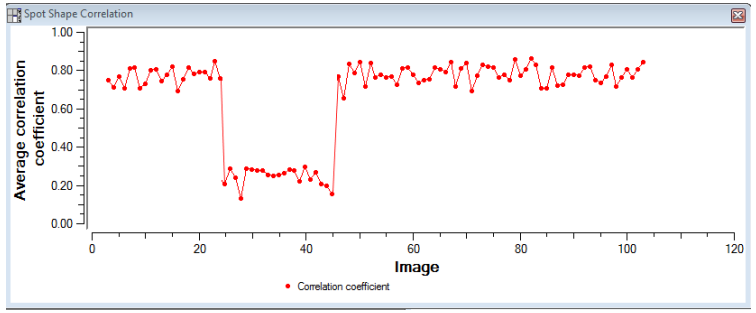
Correlation Coefficient



- Good run



- Reduce number of images to be integrated (click on spot for image number)



- Create two runs for integration and exclude bad part

Data Integration – The *_0m._ls File

```
Spots with topped/rescanned pixels:          762
Total topped/rescanned pixels:              6071
Minimum pixel ceiling(frame):             163809
Maximum pixel ceiling(frame):             163809

Total spots predicted:                     27750
Spots with absolute HKL > 511:                 0*
Spots outside resolution limit:                0*
Spots outside active detector area:         5200*
Outside frame limits:                        769*
Spots exceeding frame queue size:             32*
Collisions(same XY, adjacent frame):           0*
Spots exceeding dynamic range:               656*
Spots with too many spot components:           0*
Spots with large Lorentz factor:               2*
Spots with too much missing I:                 0*
Spots with too few BG pixels:                  0*
Spots below I/sigma threshold:                77*
Spots left in Write-Behind Cache:              0*
Spots with unwanted components:                0*
Total unwanted components:                     0*
Good spots written:                        20742
Total spot components written:             20742
Twin overlaps integrated:                      0
Partial, strong, full overlaps:                0
Full overlaps collapsed to singlet:            0
Singlets (used in stats below):            20742
Twin overlaps written:                         0

Average Intensity:                    316182.5
Average I/sigma:                         62.669
% over "strong" threshold:                74.51
% less than 2 sigma:                       9.42
% which spanned more than 1 frame:        38.08
```

→ Spots too close to restricted areas (beamstop, …)

→ Spots incomplete due to beginning / end of run

→ Spots exceeding frame queue

→ Overlapped spots

→ Overflowed reflections (detector saturation)

Optimizing Data Integration

- Recycle the orientation matrix
  - Import the *m.p4p and repeat the integration
- Carefully look at the correlation coefficient
- Adjust the resolution limit for the data integration to the diffraction limit of the crystal
- Try to activate "blend profiles“ for very weak data
- Increase the box size
- Try fixing the box size
- Increase "queue half width" slightly

SAINT – Output

- Log and statistics
  - Listing files: *._ls
  - Parameter files: *.p4p

- Intensity files:
  - single crystal: *.raw
  - twin: *.mul
  - Modulated *.ram

- Diagnostic output frame files
  - initial background: *._ib
  - active pixel mask: *._am,
  - background snapshots: bg_snap*.sfrm

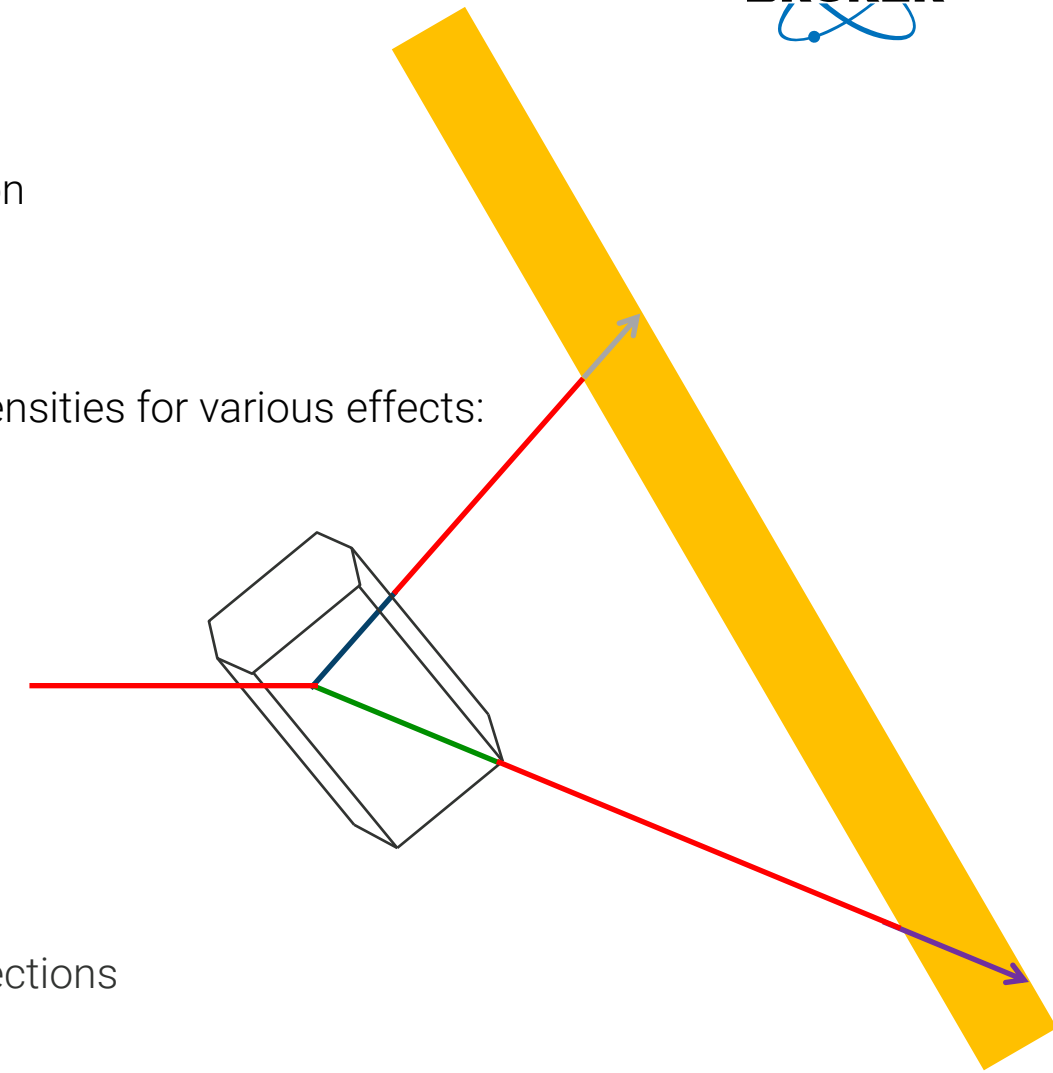
DATA COLLECTION AND REDUCTION WITH APEX6

Scaling in APEX6

Ashley (Weiland) Schmidt
Ashley.Schmidt@bruker.com

SADABS - Data scaling

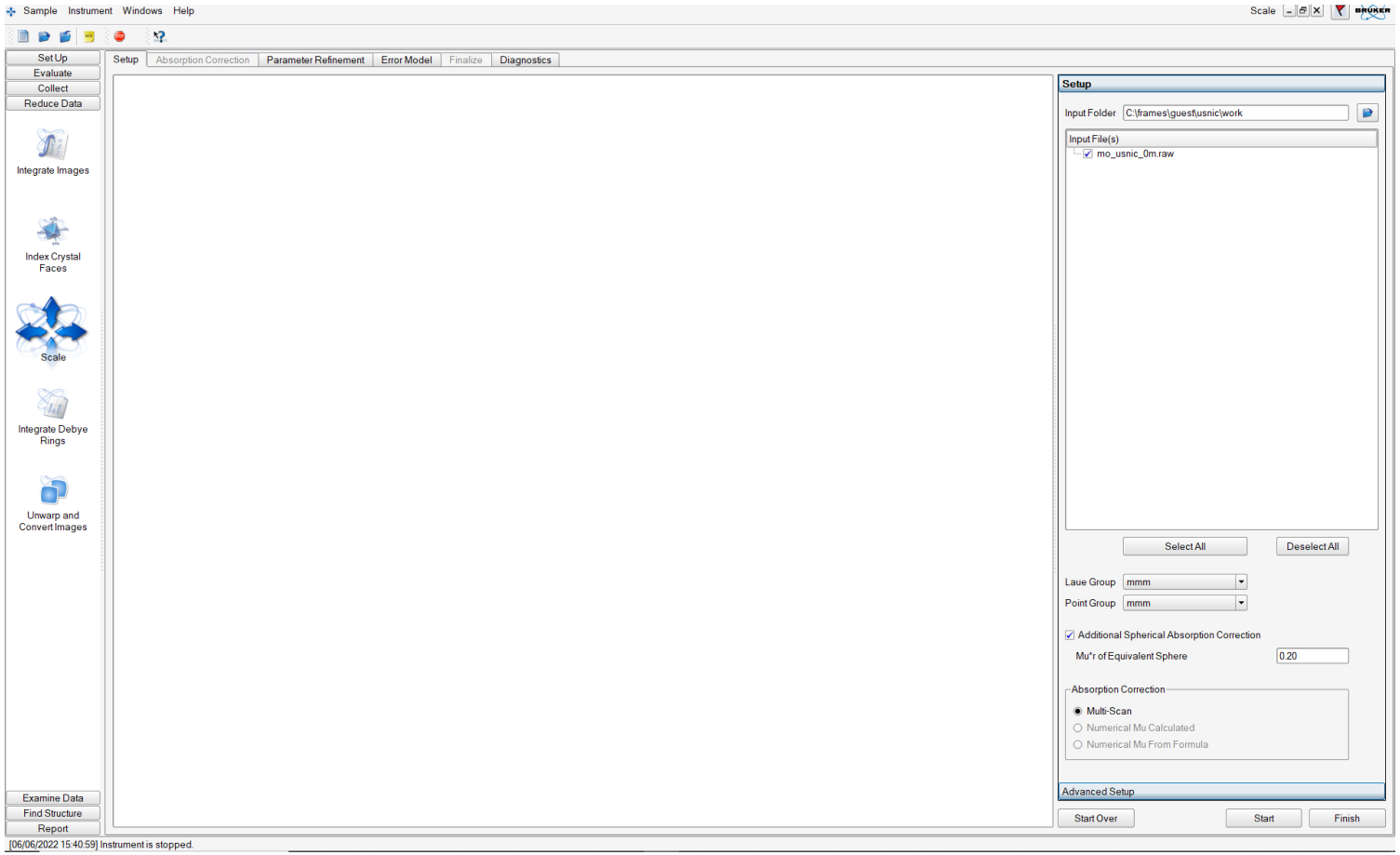
- Data set consists of multiple observations for each unique Bragg reflection
  - Duplicate observations collected at different crystal orientations
  - Symmetry-related reflections
- SADABS exploits multiplicity of observations for correcting integrated intensities for various effects:
  - Absorption due to different path length through crystal
  - Different incident angle on detector
  - Change of irradiated volume
  - Inhomogeneity of the incident beam
  - Crystal miscentering, crystal decay,…



To do so

1. Determination of the measured intensity I_{obs} of a set of equivalent reflections
2. Determination of their standard uncertainties
3. Put data set on a common scale
4. Remove outliers

SCALING

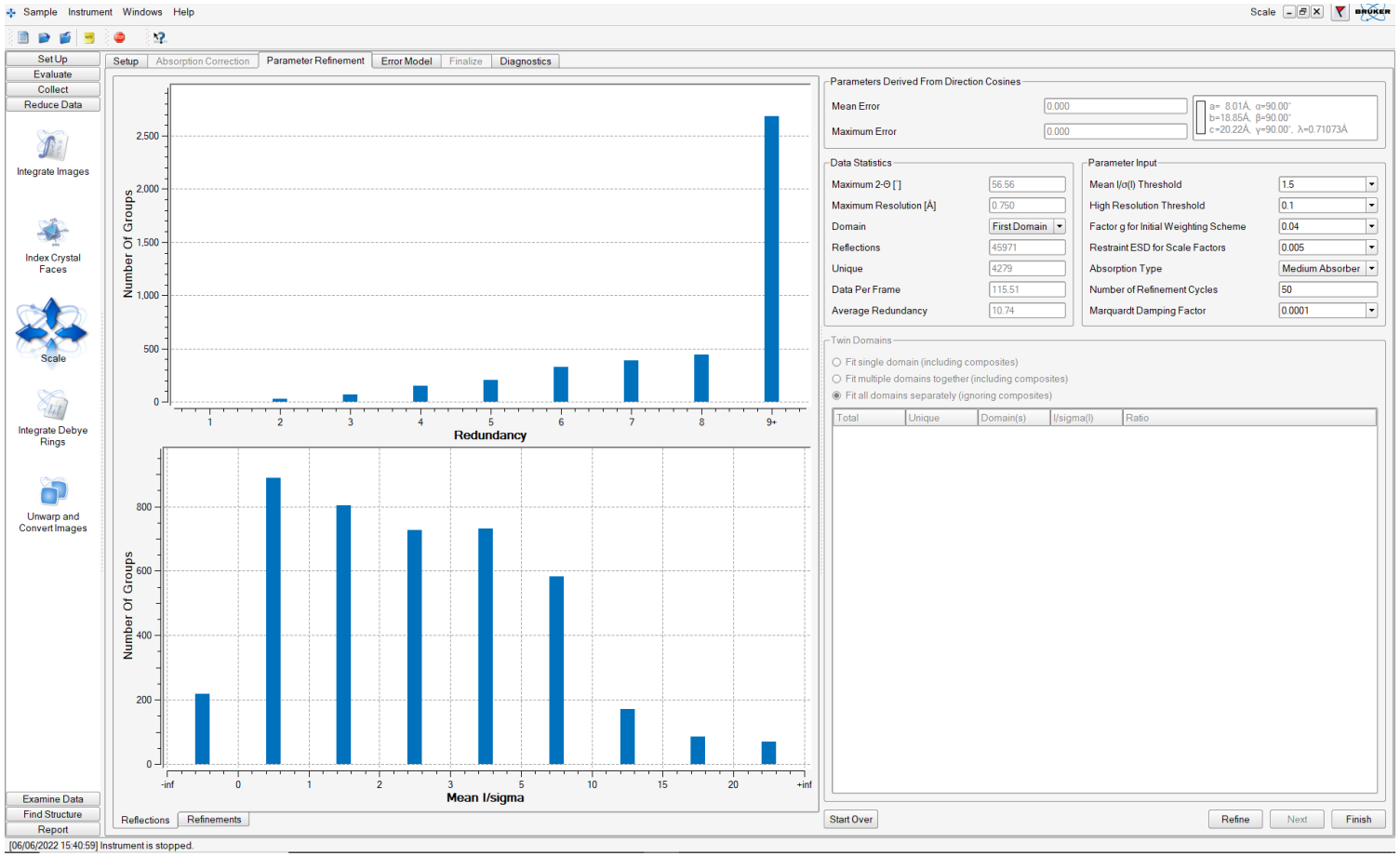


- APEX6 pre-fills important information
  - Input files
  - Output files
  - Symmetry
- Choose combined output from SAINT (default) or individual files
- Adjust μ × r if needed
- Numerical absorption correction is an option too

Laue Groups and Point Groups

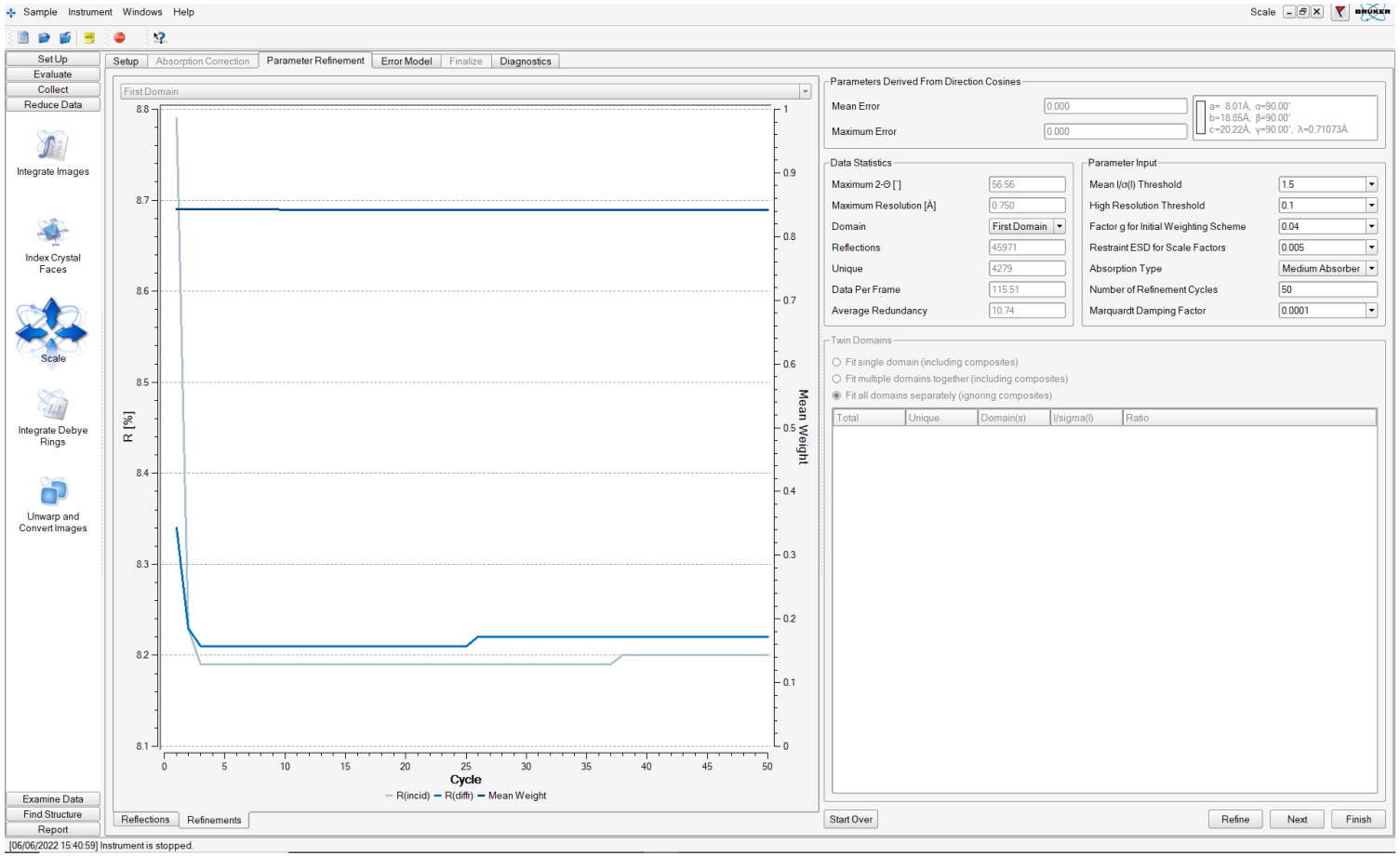
Crystal System	Laue Group	Point Group
Triclinic	-1	1, -1,
Monoclinic	2/m	2, m, 2/m
Orthorhombic	mmm	222, mm2, mmm
Tetragonal	4/m	4, -4, 4/m
	4/mmm	422, 4mm, -42m, 4/mmm
Trigonal	-3	3, -3
	-3m	32, 3m, -3m
Hexagonal	6/m	6, -6, 6/m
	6/mmm	622, 6mm, -62m, 6/mmm
Cubic	m-3	23, m-3
	m-3m	432, -43m, m-3m

SCALING – Parameter Refinement



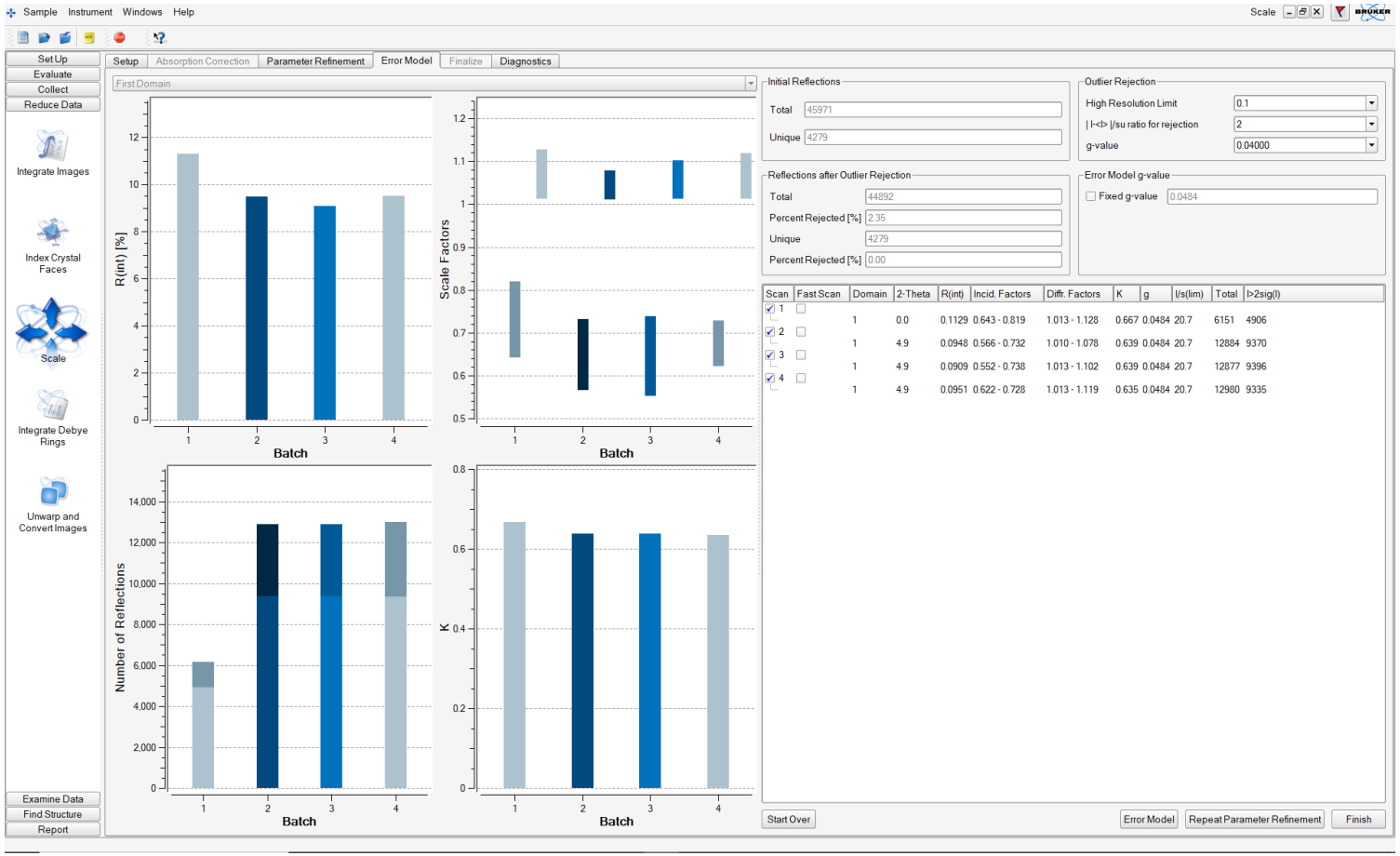
- Parameter input for scaling parameter and absorption correction parameter refinement
- Absorption type
- Restraint esd for scale parameter
  - Increasing allows a faster frame to frame change of scale parameters

SCALING – Parameter Refinement



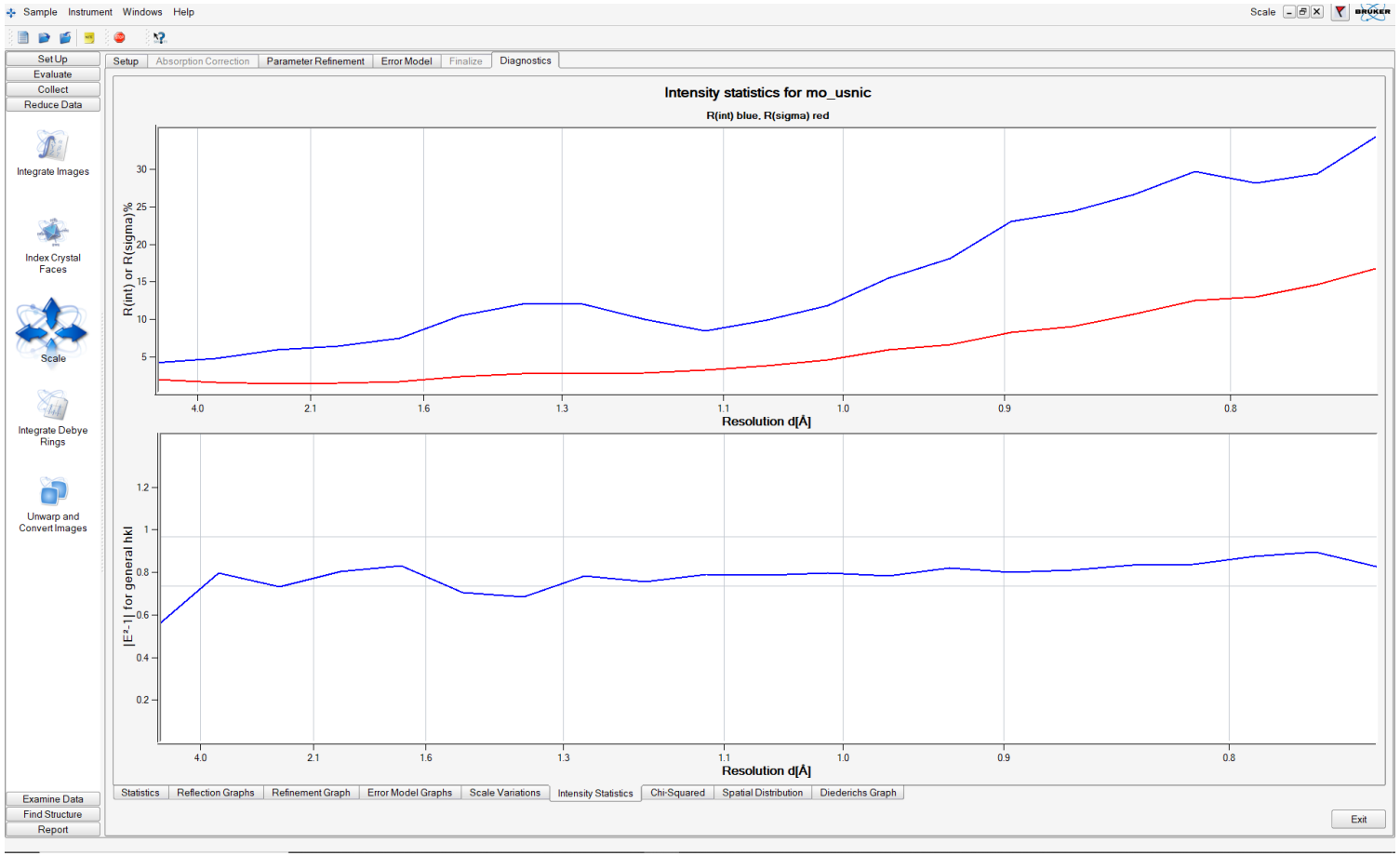
- Run at least 50 cycles
- Mean weight should be above 75%
  - If it is around 60% the chosen symmetry is probably too high
- R values need to converge
  - If you see “ringing” increase the restraint esd for scale parameters

SCALING – Error Model



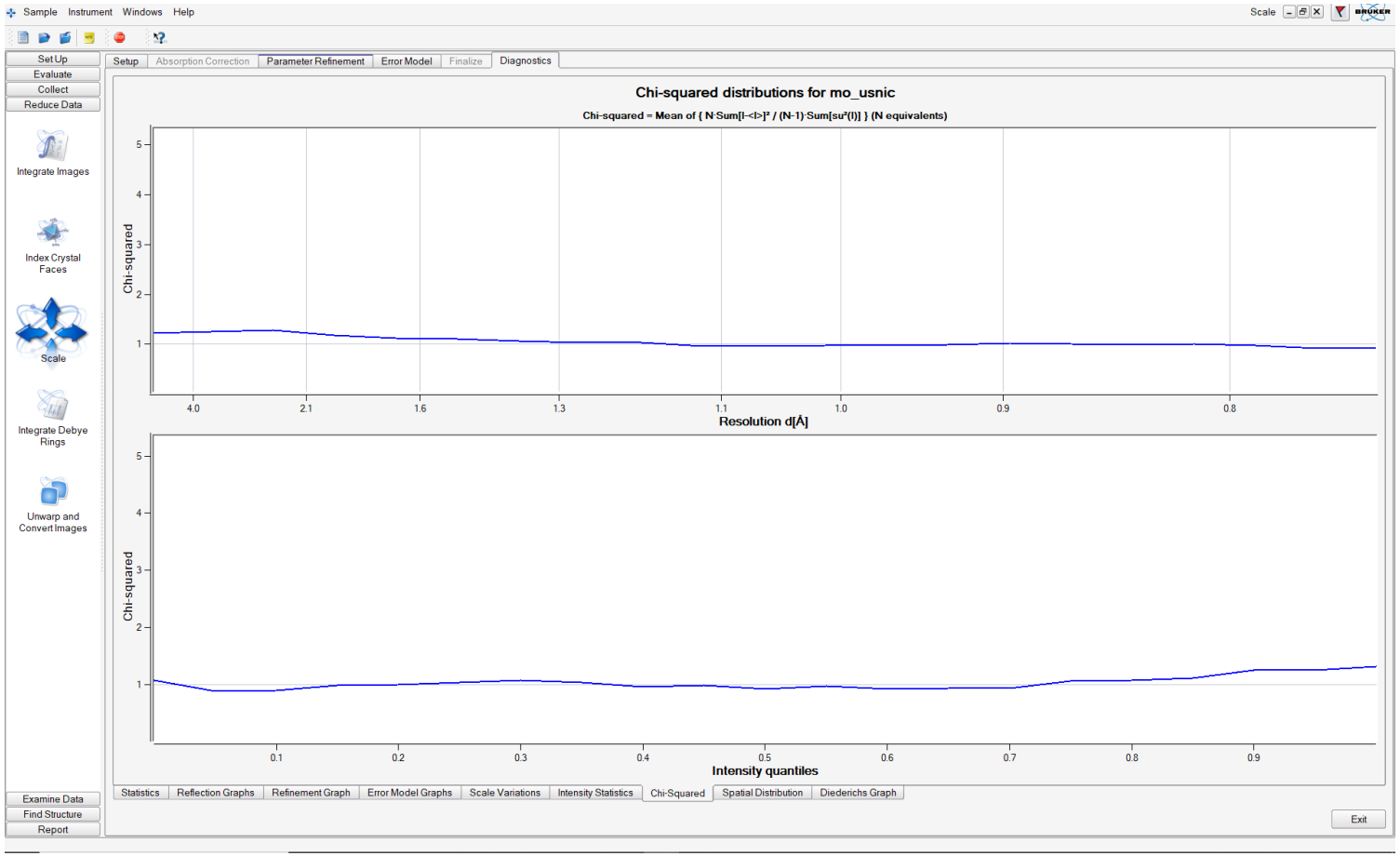
- g-value should be small
  - 0.02 to 0.04
- Only a very small number of reflections should be rejected
- Look for consistency
- If a run sticks out, remove it and start over
  - Provided you have enough redundancy

SCALING – Diagnostic Plots



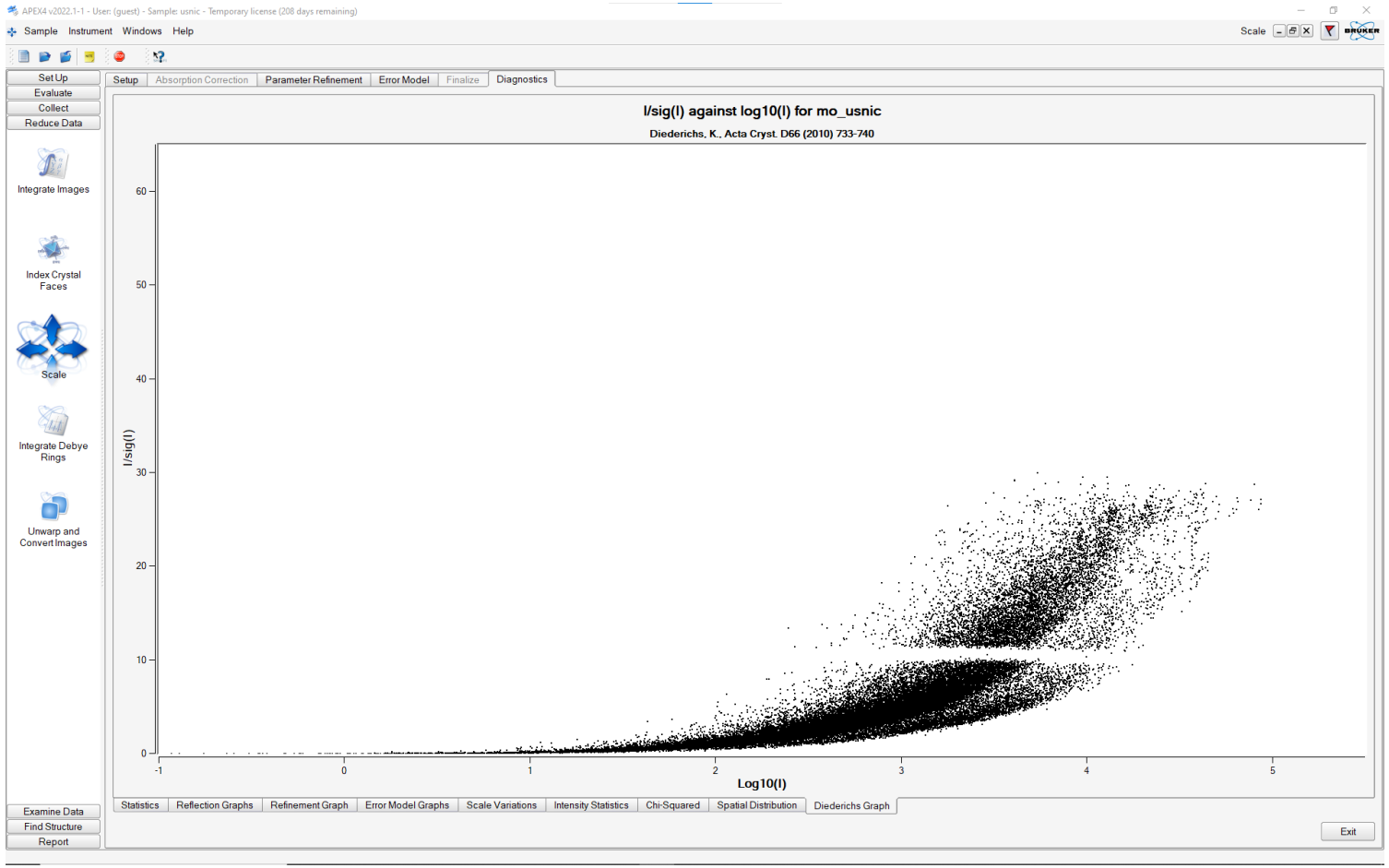
- Rint and Rsigma plot
- For a routine experiment we consider data up to Rsigma of 30% usable

SCALING – Diagnostic Plots



- The chi-squared distribution tells you how “gaussian” the distribution of your ESDs is vs intensities

SCALING – Diagnostic Plots



- Diederichs plot
- I/sig(I) vs log I
- Needs to be an S shaped distribution
- Look for a mean and lean cobra!
- The earlier the cobra rises out of the dust, the better!
- If it is lean, the Rsigma is low
- If it stands up high the error model g value is low

Thank you!

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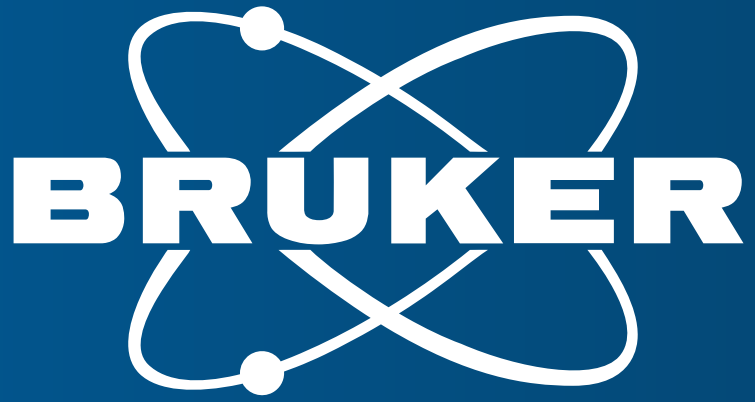
Ashley.Schmidt@bruker.com



Innovation with Integrity