

# **Single Crystal XRD: Data Acquisition and Structure Solving**

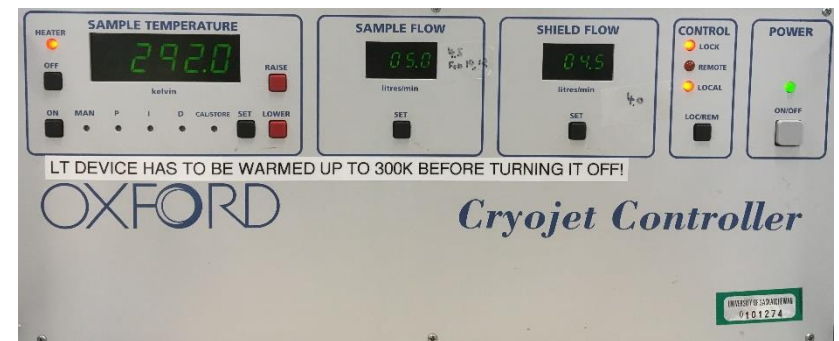
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**SSSC, University of Saskatchewan**

**June 2017**

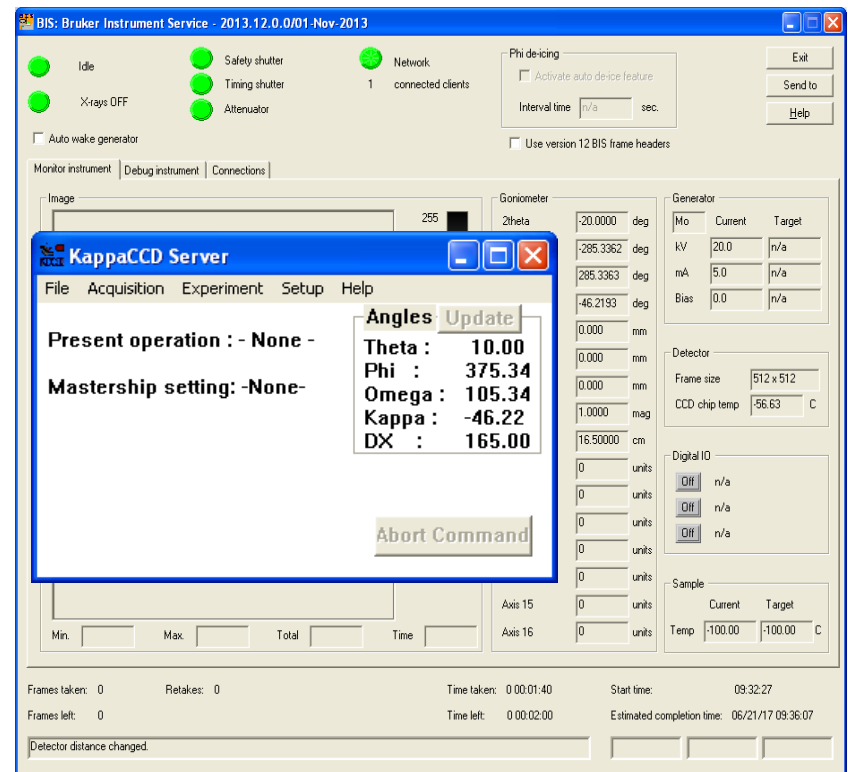
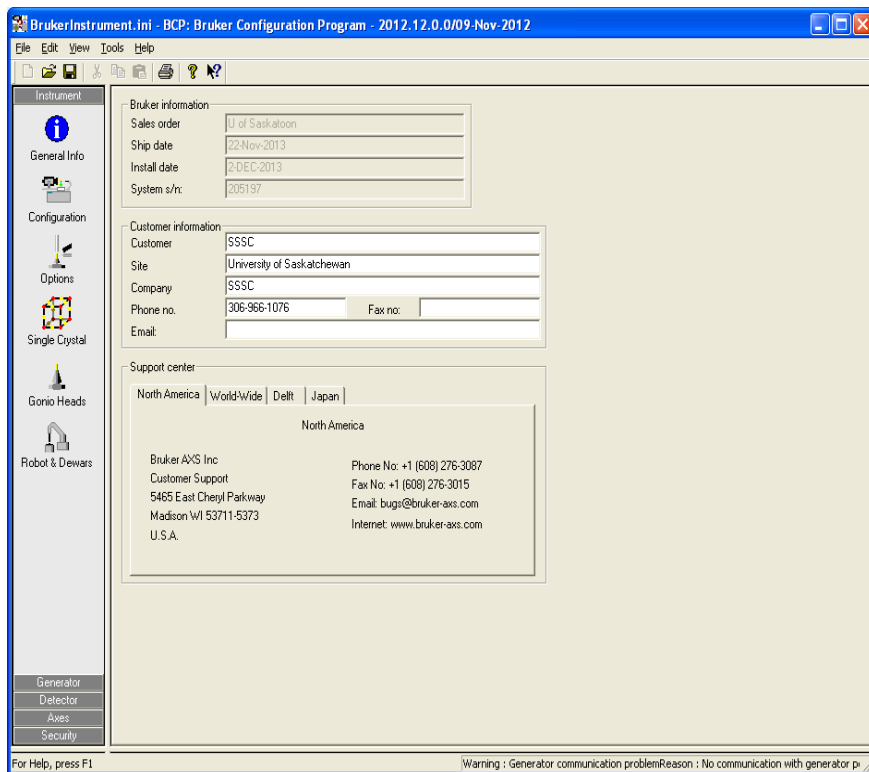
# 1. Turn on the Diffractometer

- 1) Turn on the cooling water for X-Ray tube, flow rate marked on the Gauge.
- 2) Turn on the Haskris and check flow.
- 3) Turn on X-Ray (key then 'ON' button).
- 4) Switch to Remote Control: 5 9 0 [Enter] → 9 → 1(hold) [Enter], should see '9 M 1' → Release both, then [Enter], should see '.....' → Enter.
- 5) Turn on the Cryojet Controller, and set the temperature to 173 K, Sample Flow to 5 L/min, and Shield Flow to 4.5 L/min.



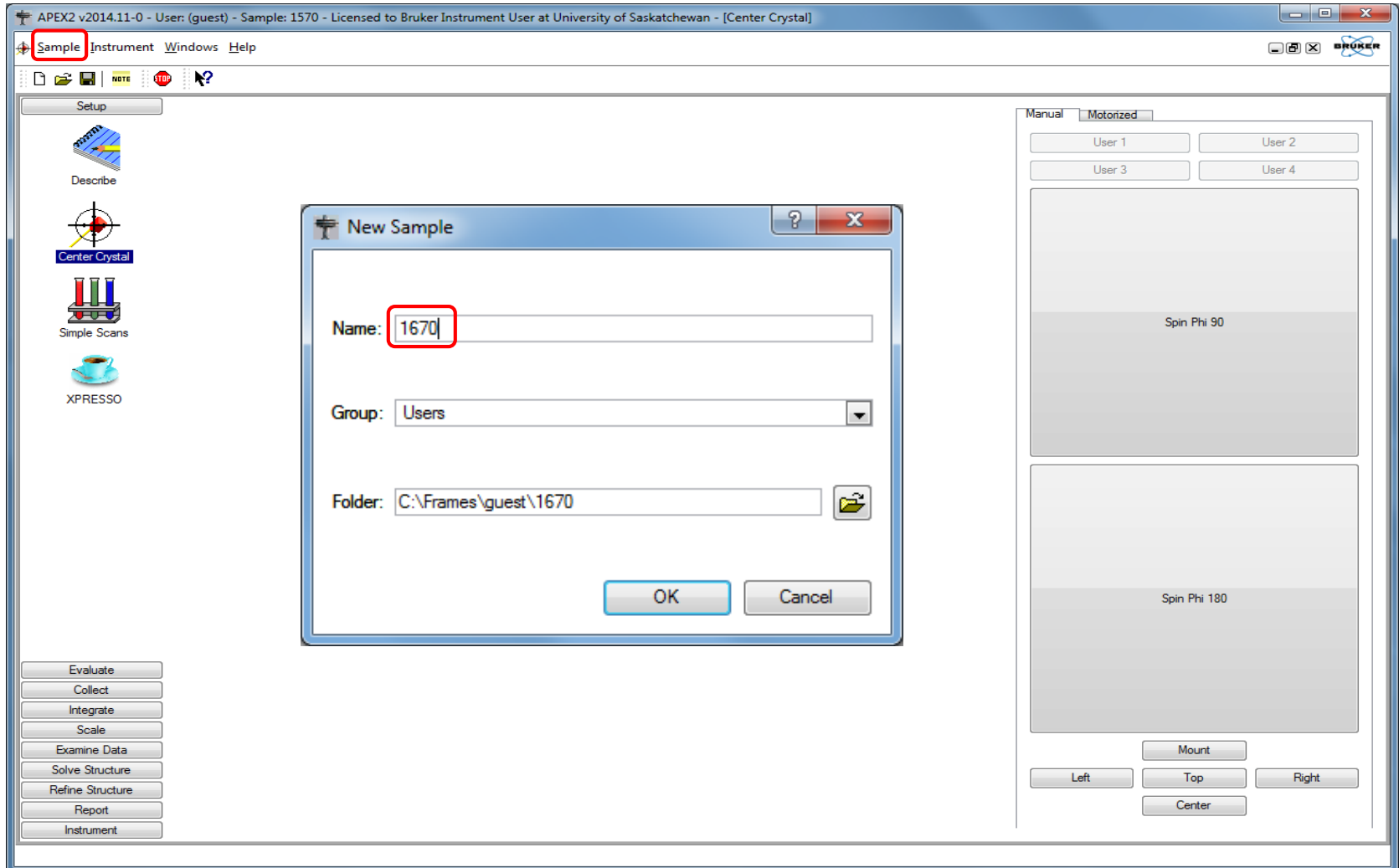
## 2. Start Softwares

- Start BCP (Bruker Configuration Program) at console PC (Bruker54).
- Start BIP (Bruker Instrument Service) at console PC (Bruker54).
- Start APEX2 at Workstation (Bruker55).



### 3. Create a New Dataset

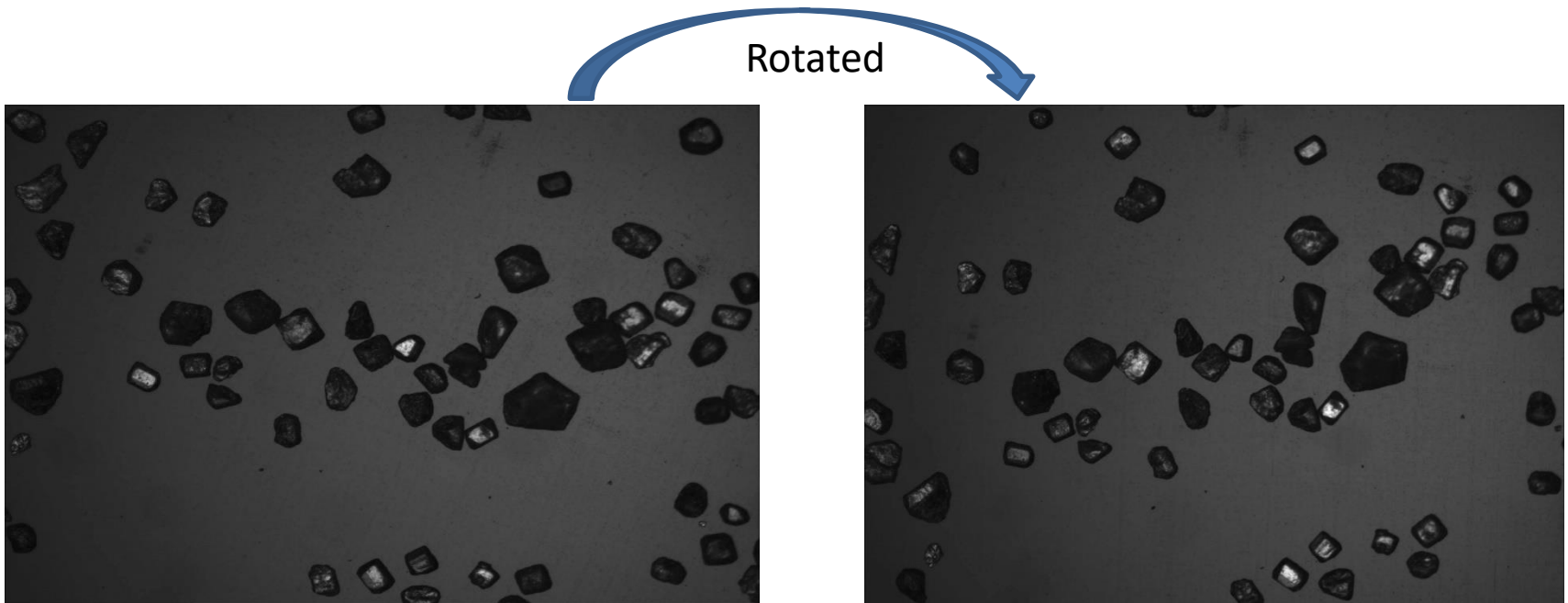
- Sample → New → Give a sample name (use sequential number).



## 4. Choose a Good Single Crystal

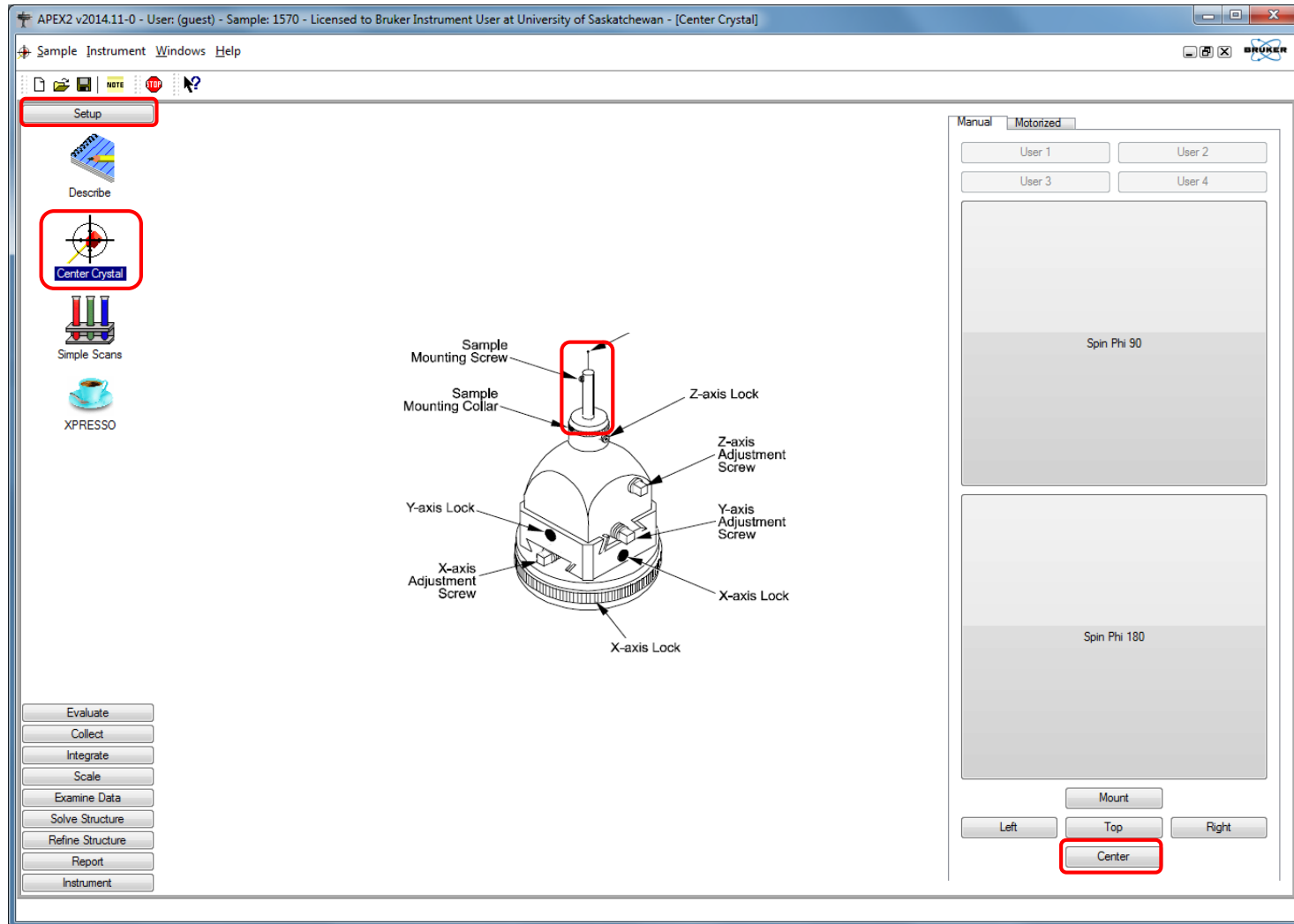
A “good” single crystal is:

- 0.1 - 0.4 mm in at least two dimensions;
- Will extinguish polarised light (check with Scope);
- Often shows regular faces and edges;
- No cracks or deformation in the crystal.



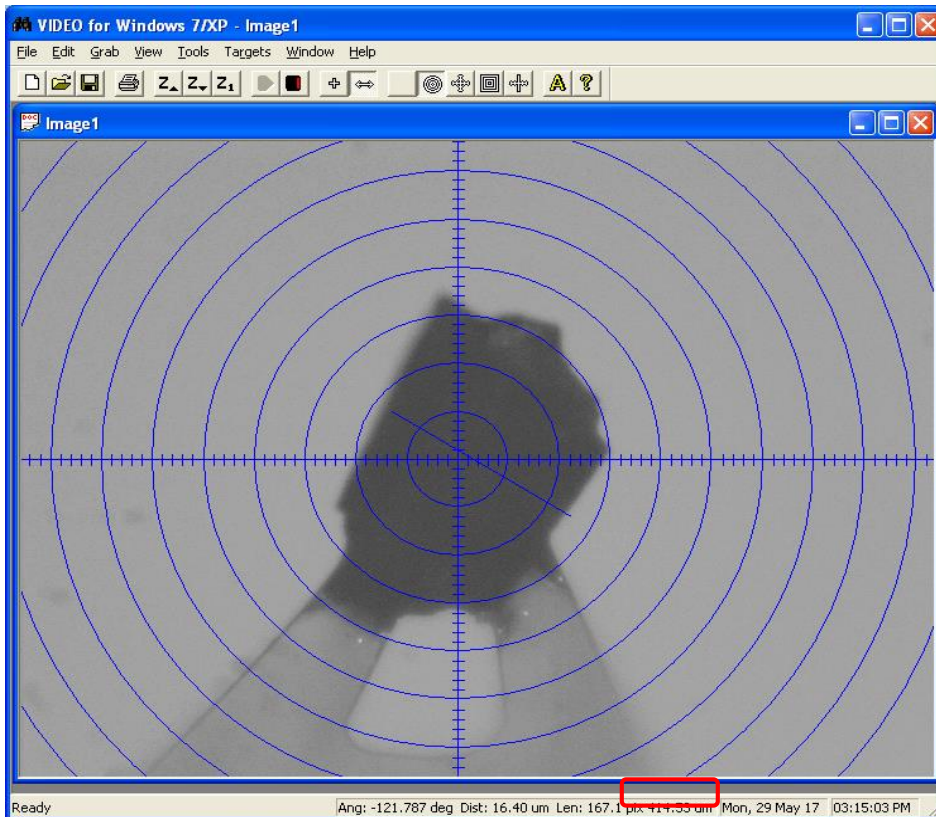
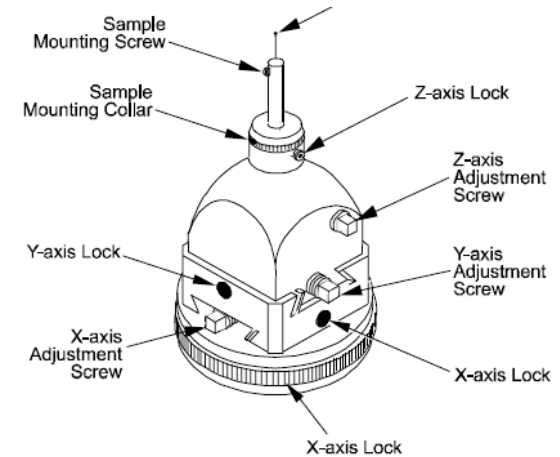
## 5. Mount the Crystal

- 1) Setup → Center Crystal → 'Center' (move Goniometer to position for Centering).
- 2) Take off the Pin → Pick a good crystal (on the tip) → load the Pin.



## 6. Center the Crystal

- 1) Adjust X to center the crystal.
- 2) 'Spin Phi 90' → Adjust Z (height) → Adjust Y.
- 3) 'Spin Phi 180' → Adjust Y → Repeat until centered.
- 4) 'Spin Phi 90' → Adjust X → 'Spin Phi 180' → Adjust X → ...
- 5) 'Spin Phi 90' → Check if still centered → ... → Close door.
- 6) Measure the crystal size (0.6 x pix).



# 7. Check Crystal Quality (1)

The screenshot shows the APEX2 software interface with a central text box containing a 7-step procedure for checking crystal quality. The interface includes a menu bar (Sample, Instrument, Windows, Help), a toolbar with various icons, and several control panels on the right. A vertical color scale on the right side ranges from 60 to 280. The bottom of the interface features a large horizontal scale from 0 to 999999 and a list of data points for a cursor position.

1) Setup → Simple Scans.  
2) Zero → set 'Distance to 50 mm'.  
3) Still → set 'Exposure Time to 5 or 10 s'.  
4) or '360 phi' → 'Exposure Time 60 s'.  
5) 'Drive + Scan' to start.  
6) Check image quality (intensities/lineshapes).  
7) If not good, try another crystal.

**Control Panels:**

- Preset Positions:** Zero, Current, Phi = 0, Phi + 90, User 1, User 2, User 3, User 4.
- Distance [mm]:** 50
- 2Theta:** 0
- Omega:** 0
- Phi:** 0
- Chi:** 0
- Drive** button
- Preset Scans:** Still, 360° Phi, Narrow (0.5), Wide (2.0)
- Scan Axis:** Phi (selected), Omega
- Scan Range:** 0.00
- Image Width:** 0.00
- Exposure Time:** 5 secs/image
- Correlate Exposures
- Dark Current Correction:** Existing dark image (selected), New dark image
- Drive + Scan** button

**Cursor Data:**

- Cursor
- Position [mm]
- Position [pixels]
- Intensity [counts]
- HKL index
- Resolution [Å]
- 2Theta [°]

**Bottom Panel:** Image Header, Top Editor, Cursor Position



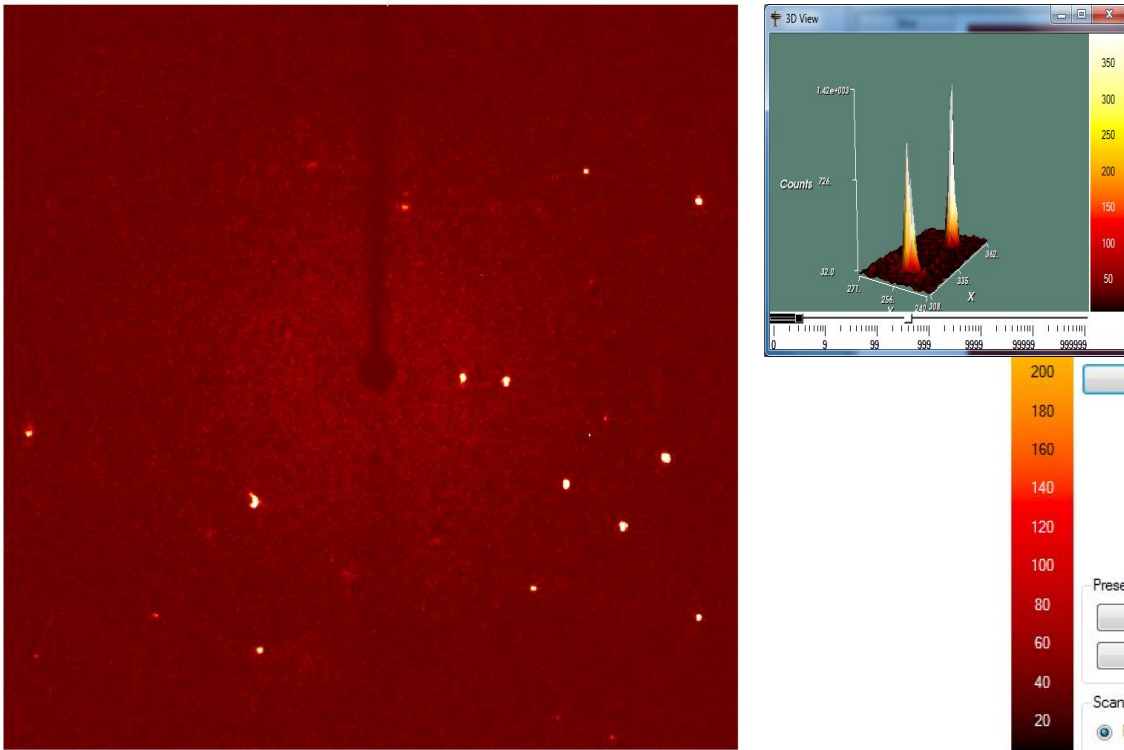
# 7. Check Crystal Quality (2)

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Simple Scans]

Sample Instrument Windows Help

C:\Frames\guest\1570\simple\_scans\1570\_01\_0001.sfm

Setup  
Describe  
Center Crystal  
Simple Scans  
XPRESSO



3D View

Positions

Zero Current  
Phi = 0 Phi + 90  
User 1 User 2  
User 3 User 4

Drive

Preset Scans

Still 360° Phi  
Narrow (0.5) Wide (2.0)

Scan Axis

Phi  Omega

Scan Range: 360.00  
Image Width: 360.00  
Exposure Time: 60.00 secs/image  
 Correlate Exposures

Dark Current Correction

Existing dark image  New dark image

Drive + Scan

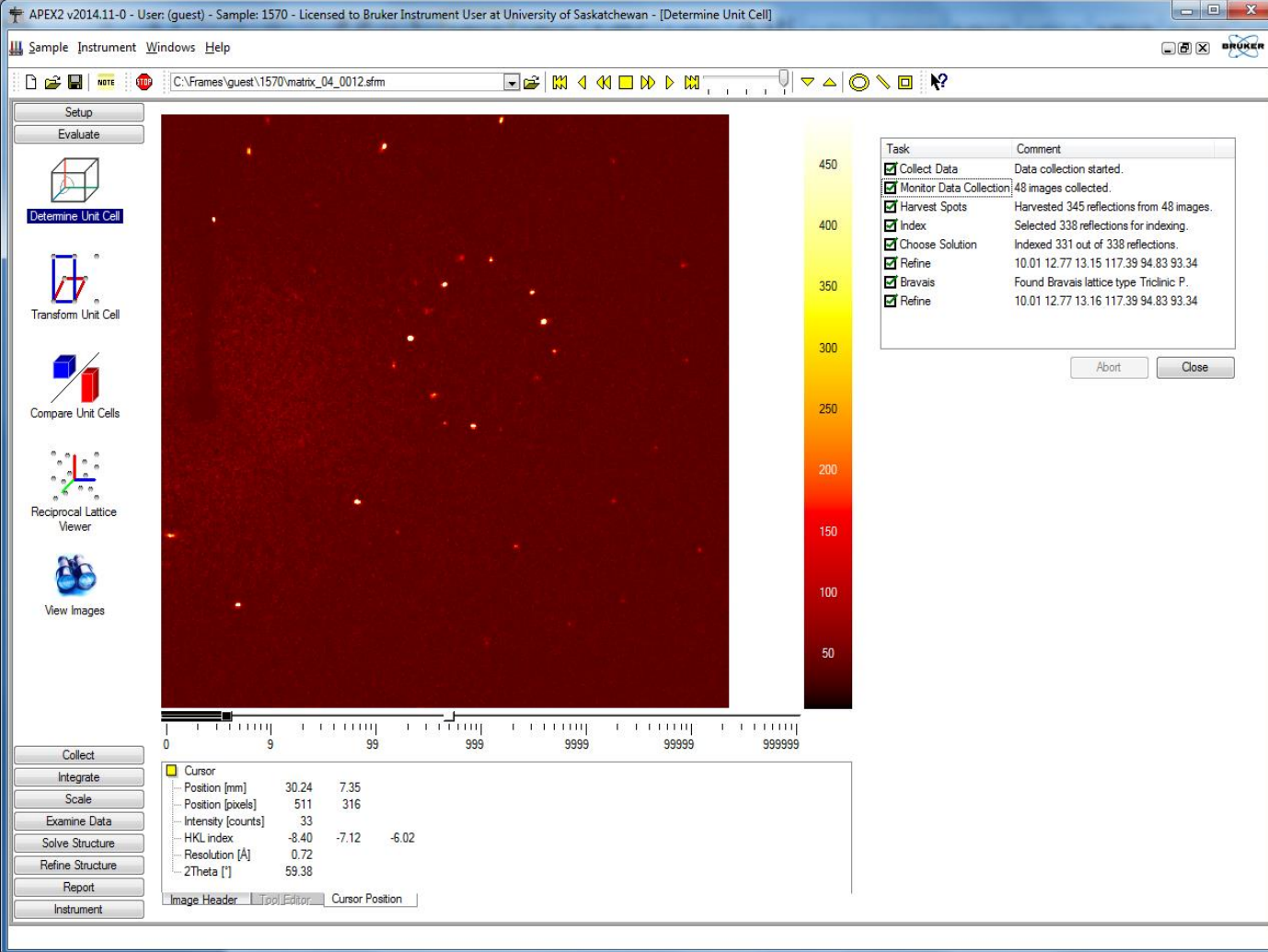
Evaluate  
Collect  
Integrate  
Scale  
Examine Data  
Solve Structure  
Refine Structure  
Report  
Instrument

Cursor			
Position [mm]	17.44	28.10	
Position [pixels]	403	491	
Intensity [counts]	37		
HKL index	-2.26	-3.99	6.52
Resolution [Å]	1.25		
2Theta [°]	32.92		

Image Header | [ppEditor](#) | Cursor Position

## 8. Determine Unit Cell

- 1) Click 'Evaluate' → 'Determine Unit Cell' → 'Run' Automatic Mode.
- 2) It will go through each module, until Unit Cell determined.



The screenshot shows the APEX2 v2014.11-0 software interface. The main window displays a diffraction pattern with a color scale on the right. The color scale ranges from 50 to 450, with 50 being dark red and 450 being yellow. The diffraction pattern shows a grid of spots, with a central spot at the origin. The software interface includes a menu bar (Sample, Instrument, Windows, Help), a toolbar with various icons, and a task list on the right. The task list shows the following tasks and comments:

Task	Comment
<input checked="" type="checkbox"/> Collect Data	Data collection started.
<input checked="" type="checkbox"/> Monitor Data Collection	48 images collected.
<input checked="" type="checkbox"/> Harvest Spots	Harvested 345 reflections from 48 images.
<input checked="" type="checkbox"/> Index	Selected 338 reflections for indexing.
<input checked="" type="checkbox"/> Choose Solution	Indexed 331 out of 338 reflections.
<input checked="" type="checkbox"/> Refine	10.01 12.77 13.15 117.39 94.83 93.34
<input checked="" type="checkbox"/> Bravais	Found Bravais lattice type Triclinic P.
<input checked="" type="checkbox"/> Refine	10.01 12.77 13.16 117.39 94.83 93.34

At the bottom left, a cursor position window shows the following data:

Cursor		
Position [mm]	30.24	7.35
Position [pixels]	511	316
Intensity [counts]	33	
HKL index	-8.40	-7.12 -6.02
Resolution [Å]	0.72	
2Theta [°]	59.38	

## 9. Refine Data Collection Strategy

- 1) Collect → 'Knight' Data Collection Strategy → Edit parameters.
- 2) Refine → Stop when 100% Completeness reached → Sort.

The screenshot displays the APEX2 v2014.11-0 software interface. The main window is titled "APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Data Collection Strategy]".

**Left Panel (Navigation):**

- Buttons: Setup, Evaluate, **Collect** (highlighted), Integrate, Scale, Examine Data, Solve Structure, Refine Structure, Report, Instrument.
- Icons: Knight (Data Collection Strategy), Horse (Data Collection Strategy), Test Tubes (Experiment), Grid (Oriented Scans).

**Top Plot (Resolution vs. Completeness/Redundancy):**

This plot shows the relationship between Resolution [Å] (x-axis, 1.6 to 0.8) and both Completeness [%] (left y-axis, 0 to 100) and Redundancy (right y-axis, 2 to 16). The blue line represents Completeness, which remains at 100% across the entire resolution range. The red line represents Redundancy, which starts at approximately 10.5 at 1.6 Å and decreases to about 2.5 at 0.8 Å.

**Right Panel (Parameters):**

- Distance [mm]: 40.10
- Mosaicity [°]: 0.63
- Image Width [°]: 0.50
- d [Å]: 0.77 (highlighted)
- 2Theta [°]: 54.97
- sinθ/λ [1/Å]: 0.65
- Unit Cell: a=10.03Å, b=12.80Å, c=13.18Å, α=117.44°, β=94.90°, γ=93.26°, Triclinic P
- Lave Class: -1
- Lattice Type: P
- Merge Bijvoet Pairs:
- Total Reflections: 12908
- Unique: 6852
- Runs: 8
- Images: 2446
- Req. Disk Space: 635 MB

**Bottom Plot (Time vs. Completeness/Redundancy):**

This plot shows the relationship between Time [h] (x-axis, 0 to 6) and both Completeness [%] (left y-axis, 0 to 100) and Redundancy (right y-axis, 2 to 10). The blue line represents Completeness, which rises sharply from 0% at 0h to 100% by approximately 3.5 hours. The red line represents Redundancy, which increases steadily from 0 at 0h to about 4.5 at 6 hours.

**Bottom Right Panel (Exposure Times and Targets):**

- Exposure Times [s] table:
 

Inf A	1.37Å	0.71Å	0.50Å	0.41Å	0.37Å	0.36Å
0°	30°	60°	90°	120°	150°	180°
0.00/Å	0.36/Å	0.70/Å	0.99/Å	1.22/Å	1.36/Å	1.41/Å
5	5	5	5	5	5	5
- Buttons: Same (highlighted), Extend, Reduce, Reset
- Completeness [%]: Current 100.00, Target 100.00, Priority 100
- Redundancy: Current 5.28, Target 50.00, Priority 5
- Even out redundancy:
- Time [h]: Current 6.00, Target 6.00, Priority 10
- Strategy: Best in 6 hours (highlighted)
- Execute: Refine (highlighted), Sort (highlighted)

# 10. Start Data Collection

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Experiment]

Sample Instrument Windows Help

Setup Experiment Monitor Experiment

Image location: C:\Frames\guest\1570  
Filename or prefix: 1570  
First run: 1

Exposures: automatic  
Retake if topped   
Generate new dark images   
Unwrap images

Default time: 10.000 [sec/image]  
Default width: 0.500 [degrees]  
Detector format: 512x512  
Deicing: off

Operation	Active	Distance [mm]	2Theta [deg]	Omega [deg]	Phi [deg]	Chi [deg]	Time [sec]	Width [deg]	Sweep [deg]	Direction
1 Phi Scan	Yes	40.100	-27.000	18.840	-10.440	-24.380	5.000	0.500	369.500	positive
2 Phi Scan	Yes	40.100	-27.000	-16.910	-230.300	-91.910	5.000	0.500	178.500	positive
3 Omega Scan	Yes	40.100	-12.000	-54.540	-65.380	61.020	5.000	0.500	48.500	positive
4 Phi Scan	Yes	40.100	-17.000	28.750	-122.260	-90.930	5.000	0.500	63.500	positive
5 Phi Scan	Yes	40.100	10.500	-14.380	-21.540	84.640	5.000	0.500	69.000	positive
6 Omega Scan	Yes	40.100	-7.000	-107.200	-64.730	39.980	5.000	0.500	106.500	positive
7 Phi Scan	Yes	40.100	-19.500	-34.010	50.090	84.640	5.000	0.500	167.000	positive
8 Phi Scan	Yes	40.100	0.500	4.920	-287.200	-44.690	5.000	0.500	218.500	positive
9 No Operation	Yes									
10 No Operation	Yes									
11 No Operation	Yes									
12 No Operation	Yes									
13 No Operation	Yes									
14 No Operation	Yes									
15 No Operation	Yes									
16 No Operation	Yes									
17 No Operation	Yes									
18 No Operation	Yes									
19 No Operation	Yes									
20 No Operation	Yes									
21 No Operation	Yes									
22 No Operation	Yes									
23 No Operation	Yes									
24 No Operation	Yes									
25 No Operation	Yes									
26 No Operation	Yes									
27 No Operation	Yes									
28 No Operation	Yes									

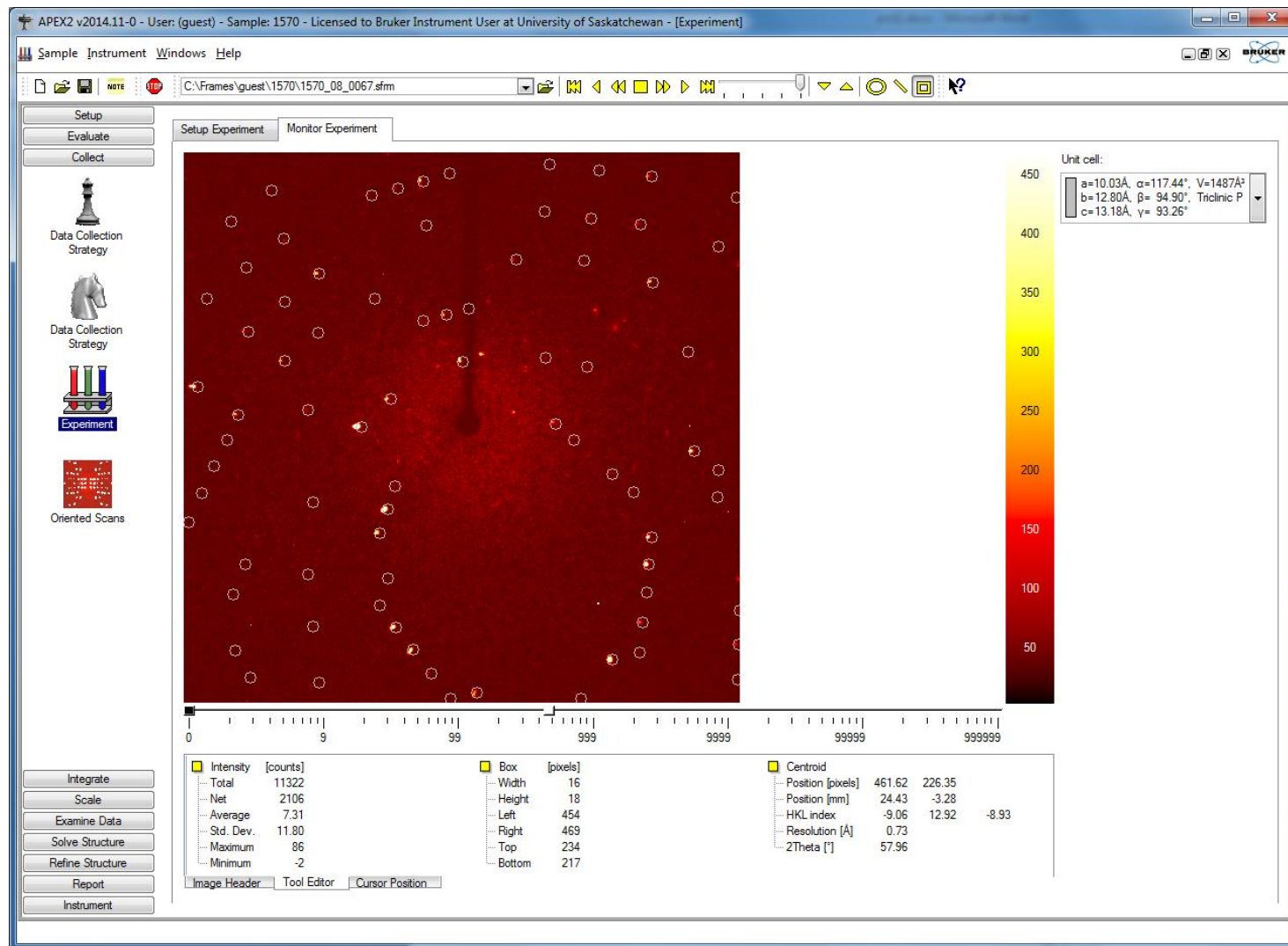
1) Collect → Experiment → Append Strategy.  
2) Validate → Execute (to start data collection).  
3) *To Stop:* Instrument → Abort.

Integrate Scale Examine Data Solve Structure Refine Structure Report Instrument

New Strategy... Append Strategy Append Matrix Strategy Load Table... Save Table... Validate Resume Execute

# 11. Monitor Data Collection

- Right click on Image → Select 'Show Overlay' (This shows how well the diffraction peaks are overlapped with the calculated ones.)



## 12. Fill Sample Info Page

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Describe]

Sample Instrument Windows Help

Setup

**Describe**

Center Crystal

Simple Scans

XPRESSO

Name: 1570

Compound:

Formula:

Appearance	Intensity	Primary Color	Secondary Color
Crystal Color: <input type="text" value="n/a"/>	<input type="text" value="n/a"/>	<input type="text" value="colourless"/>	<input type="text" value="n/a"/>

Crystal Dimensions:  x  x  [mm]

Crystal Shape:

Evaluate

Collect

Integrate

Scale

Examine Data

Solve Structure

Refine Structure

Report

Instrument

- 1) Setup → Describe.
- 2) Fill in crystal info such as Formula, Primary Colour, Crystal Dimensions, and Crystal Shape.
- 3) Go back to Collection → Experiment.

# 13. Integration (1)

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Integrate Images]

Sample Instrument Chart Windows Help

**Integrate → Integrate Images.**

Setup

Resolution Limit [Å]: 0.770

Unit Cells:  
  $a=10.03\text{Å}$ ,  $\alpha=117.44^\circ$ ,  $V=1487\text{Å}^3$   
 $b=12.80\text{Å}$ ,  $\beta=94.90^\circ$ , Triclinic P  
 $c=13.18\text{Å}$ ,  $\gamma=93.26^\circ$

Starting Image Filename	Images	Output Filename
1 C:\Frames\guest\1570\1570_01_0001.sfm	739	C:\Frames\guest\1570\work\1570_01.raw
2 C:\Frames\guest\1570\1570_02_0001.sfm	357	C:\Frames\guest\1570\work\1570_02.raw
3 C:\Frames\guest\1570\1570_03_0001.sfm	97	C:\Frames\guest\1570\work\1570_03.raw
4 C:\Frames\guest\1570\1570_04_0001.sfm	127	C:\Frames\guest\1570\work\1570_04.raw
5 C:\Frames\guest\1570\1570_05_0001.sfm	138	C:\Frames\guest\1570\work\1570_05.raw
6 C:\Frames\guest\1570\1570_06_0001.sfm	213	C:\Frames\guest\1570\work\1570_06.raw
7 C:\Frames\guest\1570\1570_07_0001.sfm	334	C:\Frames\guest\1570\work\1570_07.raw
8 C:\Frames\guest\1570\1570_08_0001.sfm	437	C:\Frames\guest\1570\work\1570_08.raw

**Integration Options**

Enable LS Profile Fitting  
 Blend Profiles from All Detector Regions

Intensity/Sigma Lower Limit for Model Profile Update: 10.000  
Fraction of Model Profile Maximum for Simple Sum Mask: 0.050  
Intensity/Sigma Upper Limit for LS Model Profile Fit: 8.000  
Lower Resolution Limit for LS Model Profile Fit [Å]: 9999.000  
Profile XYZ Half-Widths: 4 4 4

Background Update  
Background Update Scaling Factor: 1.000

Image Queue  
Active Image Queue Half-Width [Images]: 7

Beam Monitor  
 Enable Beam Monitor Normalization  
 Normalize each Run Separately

Active Mask  
 Generate Mask:  
Fractional Lower Limit of Average Intensity: 0.35  
 Use Pre-Existing Static Mask:  
Active Mask File:   
 Use Pre-Existing Dynamic Masks

Twin Overlap Determination  
Minimum Common Volume [%]: 4.000  
Separation Factor: 1.000  
Maximum Range: 1.300

Algorithm  
 Use Narrow Frame Algorithm  Use Wide Frame Algorithm

Modulated Structure Integration  
Maximum Satellite Index: 1

Monte Carlo Simulation  
Number of Monte Carlo Simulations: 32

Output / Diagnostic Files  
 Generate Diagnostic Plot Files  
 Keep Temporary Files  
 Append Listing Files  
 Hide Log Window  
Verbosity of Listing File: 2  
Snapshot Output Frequency [Images]: 100

Image Timeout  
 Wait for Images During Data Collection

Refinement Options...  
**Integration Options...**  
Find Runs...  
Import Runs from Experiment  
Start Integration...

Scale  
Examine Data  
Solve Structure  
Refine Structure  
Report  
Instrument

[06/21/2017 16:33:01] Generator is unstable - generator settings are fluctuating from target values

# 13. Integration (2)

APEX2 v2014.11.0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Integrate Images]

Sample Instrument Chart Windows Help

Setup Evaluate Collect Integrate Integrate Images

Setup Integration

Spot Shape Correlation

Average correlation coefficient

• Correlation coefficient

Average Spot Intensity and I/Sigma(I) Values

Average spot intensity

Average I/Sigma(I)

• Intensity ■ I/sigma

Spot Shape Profiles by Detector Region

Snapshot after image:

Average Difference Between Observed and Predicted X, Y, Z

Error [pixels]

• X ■ Y ▲ Z

Scan 1 - Integration

All Components

- Progress Statistics
  - Integration progress
- Per-Image Statistics
  - Spot shape correlation
  - Spot intensity and I/Sigma(I)
  - % Spot intensity < 2 Sigma(I)
  - Number of reflections harvested
  - Spot position overlay
  - X, Y, Z error
  - X, Y, Z RMS difference
  - X, Y, Z spot size
  - % Queue extent used
  - Primary beam intensity
- Periodic Statistics
  - Profile snapshots
  - A axis + ESD
  - B axis + ESD
  - C axis + ESD
  - Alpha angle + ESD
  - Beta angle + ESD
  - Gamma angle + ESD
  - Cell volume + ESD
  - LS goodness-of-fit
  - LS Residuals
  - X, Y, Z crystal translation
  - X, Y beam center
  - Distance correction
  - Pitch, Roll, Yaw correction
  - Angle zeros
- Coverage Statistics
  - Harvested reflections
  - Completeness and redundancy
  - Bijvoet pairs

Scale Examine Data Solve Structure Refine Structure Report Instrument

138_0139	15	-0.07	0.05	0.09	0.33	0.34	0.17	2901.7	15	27	0.80	31	1.30	1.35	0.87	1.000	
139_0140	18	-0.06	-0.07	-0.00	0.23	0.23	0.10	2338.5	13	17	0.80	24	1.30	1.35	0.87	1.000	
Background pixels updated = 93.41% Port, connections: 2001, 1																	
Integration of 1570																	
#	File	#Ref	ErrX	ErrY	ErrZ	RmsX	RmsY	RmsZ	Inorm	#Sig	%<2s	<Cor>	%Full	XSiz	YSiz	ZSiz	Beam
140_0141	19	0.02	0.16	0.01	0.42	0.68	0.23	888.63	7	32	0.77	27	1.30	1.35	0.87	1.000	
141_0142	16	-0.05	0.05	-0.01	0.39	0.25	0.29	418.98	4	44	0.67	26	1.30	1.35	0.87	1.000	
142_0143	12	0.04	0.05	-0.02	0.29	0.44	0.18	1034.7	11	33	0.77	32	1.30	1.35	0.87	1.000	

Connected:  Integration in Progress:

Stop Integration Close



# 14. Scale (1)

The screenshot shows the APEX2 v2014.11-0 software interface. The title bar indicates the user is 'User: (guest)' and the sample is 'Sample: 1570'. The main window is titled 'Scale' and contains several tabs: 'Setup', 'Numerical Absorption Correction', 'Parameter Refinement', 'Error Model', and 'Diagnostics'. The 'Setup' tab is active, showing options for 'Use Merged Batches or Individual Batches'. The 'Merged Batches' option is selected, and the file path is 'C:\Frames\guest\1570\work\1570\_0m.raw'. The 'Base' field is set to '1570'. The 'Output File Type' is 'Unmerged .hkl file', and the 'Output File Name' is '1570\_0m'. The 'Diagnostic Plots File Name' is '1570.eps', the 'Title of Diagnostic Plots' is '1570', and the 'Log File' is '1570.abs'. The 'Point Group' is set to '-1'. The 'Additional Spherical Absorption Correction' is checked, and the 'Mu\*r of Equivalent Sphere' is '0.2'. The 'Allow for crystal decomposition by B-value refinement' is set to 'None', and the 'Extra Linear Correction to be Applied to Each Reflection' is also 'None'. The 'Fast Scan Resolution Cutoff' is '0.1', and the 'Spatial display of  $I-\langle I \rangle / s_u$  greater than:' is '3.0'. The 'Absorption Correction Type' is set to 'Multiscan Absorption Correction'. The 'P4P File' is 'None'. The 'Next' and 'Finish' buttons are visible at the bottom right. A red box highlights the following instructions:

- Scale → Scale.
- Default setting → Next.
- Refine → Next.

# 14. Scale (2)

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Scale]

Sample Instrument Windows Help

Setup Evaluate Collect Integrate Scale

Crystal Faces Scale

Examine Data Solve Structure Refine Structure Report Instrument

Setup Numerical Absorption Correction Parameter Refinement Error Model Diagnostics

Initial Reflections  
 Total: 32954  
 Unique: 6763

Outlier Rejection  
 High resolution limit: 0.1  
 $|I| < I/su$  ratio for rejection: 4.0  
 g-value: 0.0400

Reflections after Outlier Rejection  
 Total: 32784  
 % Rejected: 0.5  
 Unique: 6763  
 % Rejected: 0.0

Include the following batches:

Batches	2-Theta	R(int)	Incid. factors	Dffr. factors	K	g	I/s(jin)
<input checked="" type="checkbox"/> 1	-27.0	0.0301	0.955 - 1.053	0.961 - 1.042	0.671	0.018	56.4
<input checked="" type="checkbox"/> 2	-27.0	0.0244	0.793 - 0.846	0.965 - 1.041	0.730	0.018	56.4
<input checked="" type="checkbox"/> 3	-12.0	0.0270	0.875 - 0.914	0.970 - 1.040	0.707	0.018	56.4
<input checked="" type="checkbox"/> 4	-17.0	0.0302	0.796 - 0.842	0.968 - 1.041	0.906	0.018	56.4
<input checked="" type="checkbox"/> 5	10.5	0.0230	0.809 - 0.833	0.964 - 1.042	0.684	0.018	56.4
<input checked="" type="checkbox"/> 6	-7.0	0.0274	0.928 - 0.979	0.970 - 1.044	0.675	0.018	56.4
<input checked="" type="checkbox"/> 7	-19.5	0.0226	0.785 - 0.882	0.968 - 1.042	0.664	0.018	56.4

Determine Error Model Repeat Parameter Refinement **Finish**

**R(int) [%]**

Batch	R(int) [%]
1	0.30
2	0.24
3	0.27
4	0.30
5	0.23
6	0.27
7	0.23
8	0.24

**Scale Factors**

Batch	Scale Factors
1	1.05
2	1.00
3	0.90
4	1.05
5	1.00
6	0.95
7	1.05
8	1.05

**Number of Reflections**

Batch	Number of Reflections
1	11000
2	4000
3	1500
4	1200
5	1800
6	3000
7	3500
8	6000

**K**

Batch	K
1	0.67
2	0.73
3	0.70
4	0.91
5	0.68
6	0.67
7	0.66
8	0.66

# 14. Scale (3)

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Scale]

Sample Instrument Windows Help

Setup Evaluate Collect Integrate Scale

Crystal Faces Scale

Setup Numerical Absorption Correction Parameter Refinement Error Model Diagnostics

Direction cosine  
Mean error 0.001  
Maximum error 0.004

Initial Reflections  
Total 32954  
Unique 6763

Transmission Data  
Corrected Reflections: 32784.0  
Replaced Reflections:  
Minimum Transmission: 0.6861  
Maximum Transmission: 0.7456  
Ratio of min/max apparent transmission: 0.9202

Data Statistics  
Maximum 2-Theta (degrees) 55.09  
Maximum Resolution (Angstroms) 0.77  
Approximate Wavelength (Angstroms) 0.71112  
Reflections 32954  
Unique 6763  
Data per frame 13.64  
Average Redundancy 4.87  
Approximate Unit Cell (from direction cosines)  
a b c  $\alpha$   $\beta$   $\gamma$   
10.012 12.770 13.152 117.332 94.955 93.266

wR2(int)  
Initial wR2(int) 0.0521  
Overall wR2(int) 0.0414  
(selected reflections only, after parameter refinement)

Reflections after Outlier Rejection  
Total 32784  
% Rejected 0.5  
Unique 6763  
% Rejected 0.0

Statistics Reflection Graphs Refinement Graph Error Model Graphs Scale Variations Intensity Statistics Chi-Squared Spatial Distribution

Examine Data Solve Structure Refine Structure Report Instrument

Start Over Exit AXScale

# 15. Determine Space Group (1)

The screenshot displays the APEX2 v2014.11-0 software interface. The main window shows a diffraction pattern with a color scale on the right ranging from 20 to 320. A dialog box titled "Select Files For XPrep" is open, prompting for P4P and HKL files. The P4P file path is `C:\Frames\guest\1570\work\1570_0m.p4p` and the HKL file path is `C:\Frames\guest\1570\work\1570_0m.hkl`. The "Examine Data" button in the left sidebar and the "Space Group Determination" icon are highlighted with red boxes. The unit cell parameters are listed as  $a=10.00\text{\AA}$ ,  $\alpha=117.57^\circ$ ,  $V=1478\text{\AA}^3$ ,  $b=12.78\text{\AA}$ ,  $\beta=94.96^\circ$ , Triclinic P,  $c=13.17\text{\AA}$ ,  $\gamma=93.18^\circ$ . The cursor position is shown as  $(-27.61, 12.39)$  in mm and  $(23, 359)$  in pixels, with an intensity of 50 counts. The HKL index is  $(-1.32, 3.78, 0.59)$ , the resolution is  $2.70\text{\AA}$ , and the  $2\theta$  is  $15.15^\circ$ .

Unit cell:

$a=10.00\text{\AA}$ ,  $\alpha=117.57^\circ$ ,  $V=1478\text{\AA}^3$   
 $b=12.78\text{\AA}$ ,  $\beta=94.96^\circ$ , Triclinic P  
 $c=13.17\text{\AA}$ ,  $\gamma=93.18^\circ$

Select Files For XPrep

P4P file: `C:\Frames\guest\1570\work\1570_0m.p4p`

HKL file: `C:\Frames\guest\1570\work\1570_0m.hkl`

OK Cancel

Cursor

Position [mm]	-27.61	12.39	
Position [pixels]	23	359	
Intensity [counts]	50		
HKL index	-1.32	3.78	0.59
Resolution [Å]	2.70		
2Theta [°]	15.15		

# 15. Determine Space Group (2)

```

XPREP Version 2014/2 for Windows Copyright(C) Bruker-AXS 2014
Current dataset: 1570_0m.hkl          Wavelength: 0.71073 Chiral: ?
Original cell:  9.986 12.744 13.128 117.37 94.93 93.28 Vol 1469.4
                Esds: 0.000 0.000 0.000 0.00 0.00 0.00 Lattice: P
Current cell:  9.986 12.744 13.128 117.37 94.93 93.28 Vol 1469.4
Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.0000
Crystal system: Triclinic Lattice: P

[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu or [Q] QUIT program

Select option [S]:

[A] Triclinic, [M] Monoclinic, [O] Orthorhombic, [T] Tetragonal,
[H] Trigonal/Hexagonal, [C] Cubic or [E] EXIT

Select option [A]:

Lattice exceptions: P A B C I F Obv Rev All
N (total) = 0 16320 16440 16436 16385 24598 21879 21859 32784
N (int>3sigma) = 0 12814 13247 13153 13065 19607 17604 17471 26227
Mean intensity = 0.0 9.0 13.3 12.9 13.6 11.7 12.2 13.5 13.4
Mean int/sigma = 0.0 14.1 15.6 15.2 15.5 14.9 15.3 15.4 15.4

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]: p

Mean |E*E-1| = 0.988 [expected .968 centrosym and .736 non-centrosym]

Systematic absences not required for triclinic

Identical indices and Friedel opposites combined before calculating R(sym)

Option Space Group No. Type Axes CSD R(sym) N(eq) Syst. Abs. CFOM
[A] P-1 # 2 centro 1 8646 0.000 0 0.0 / 15.4 0.70
[B] P1 # 1 chiral 1 700 0.000 0 0.0 / 15.4 7.16

Select option [A]:
  
```

```

XPREP Version 2014/2 for Windows Copyright(C) Bruker-AXS 2014
Resolution #Data #Theory %Complete Redundancy Mean I Mean I/s Rmerge Rsigma
Inf - 3.10 102 102 100.0 10.84 45.97 88.17 0.0196 0.0066
3.10 - 2.09 237 237 100.0 10.07 41.80 97.64 0.0170 0.0073
2.09 - 1.65 347 347 100.0 9.73 21.49 74.00 0.0192 0.0088
1.65 - 1.44 336 336 100.0 9.26 16.82 63.54 0.0230 0.0105
1.44 - 1.31 343 343 100.0 9.03 14.52 55.36 0.0264 0.0118
1.31 - 1.21 371 371 100.0 8.29 12.03 47.16 0.0300 0.0138
1.21 - 1.14 343 343 100.0 7.49 9.32 38.01 0.0352 0.0171
1.14 - 1.09 301 301 100.0 6.29 6.34 28.73 0.0391 0.0231
1.09 - 1.04 359 359 100.0 5.01 5.52 22.08 0.0441 0.0286
1.04 - 1.00 338 338 100.0 4.27 5.52 20.65 0.0480 0.0333
1.00 - 0.96 399 399 100.0 3.75 4.67 16.67 0.0556 0.0413
0.96 - 0.93 349 349 100.0 3.19 4.29 15.22 0.0557 0.0500
0.93 - 0.91 249 249 100.0 2.99 3.93 13.75 0.0551 0.0526
0.91 - 0.88 442 442 100.0 2.56 3.46 10.85 0.0622 0.0633
0.88 - 0.86 328 329 99.7 2.42 3.36 10.44 0.0577 0.0654
0.86 - 0.84 340 340 100.0 2.18 3.16 9.58 0.0570 0.0721
0.84 - 0.82 385 389 99.0 2.01 2.95 8.23 0.0639 0.0803
0.82 - 0.81 225 231 97.4 1.79 2.70 6.91 0.0775 0.0955
0.81 - 0.79 429 429 100.0 1.84 2.51 7.04 0.0790 0.0956
0.79 - 0.78 225 235 95.7 1.71 1.81 5.18 0.0831 0.1266
0.78 - 0.77 315 363 86.8 1.42 2.04 5.60 0.0882 0.1235

-----
0.87 - 0.77 2080 2149 96.8 1.88 2.62 7.51 0.0678 0.0906
Inf - 0.77 6763 6832 99.0 4.80 8.58 28.06 0.0261 0.0241

Merged [A], lowest resolution = 11.56 Angstroms

Graphical output: 1=<I/s>, 2=Rmerge, 3=Rsigma, <Enter>=none:
  
```

Choose the default options (Enter) all the way through.

# 16. Solve the Structure (1)

APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Structure Solution]

Sample Instrument Windows Help

Setup  
Evaluate  
Collect  
Integrate  
Scale  
Examine Data  
**Solve Structure**  
Auto-Structure  
**Structure Solution**  
View Molecule

Instructions Listing Results Structure View

```
TITL 1570_0m in P-1
CELL 0.71073 9.98620 12.74430 13.12770 117.3675 94.9275 93.2764
ZERR 4.00 0.00020 0.00030 0.00030 0.0008 0.0011 0.0011
LATT 1
SFAC C H O Cl
UNIT 80 120 16 8
TEMP -100.000
SIZE 0.13 0.18 0.31
HKLF 4
END
```

Instruction File 1570\_0m.ins  
Reflection File 1570\_0m.hkl  
Formula C<sub>20</sub>H<sub>30</sub>O<sub>4</sub>Cl<sub>2</sub> Z 4 Update  
 Chiral  Non-centrosymmetric  Any

Statistics

Reflections	32784	Unique	6763
Rejected	0	Observed	5724
R(int) [%]	2.6	% Observed	85
R(sigma) [%]	2.6	% Observed in 1.1-1.2Å Shell	96

Method

Intrinsic Phasing Intrinsic 1  
 Direct Direct 1  
 Dual Space Dual Space 1  
 Patterson Patterson 1 Expand

Intrinsic Phasing Solutions

R1 [%]  
alpha

**Solve Structure** Stop after Iteration  
Reset Exit

Refine Structure  
Report  
Instrument

- Solve Structure → Structure Solution.
- Choose 'Intrinsic Phasing'.
- Solve Structure.

# 16. Solve the Structure (2)

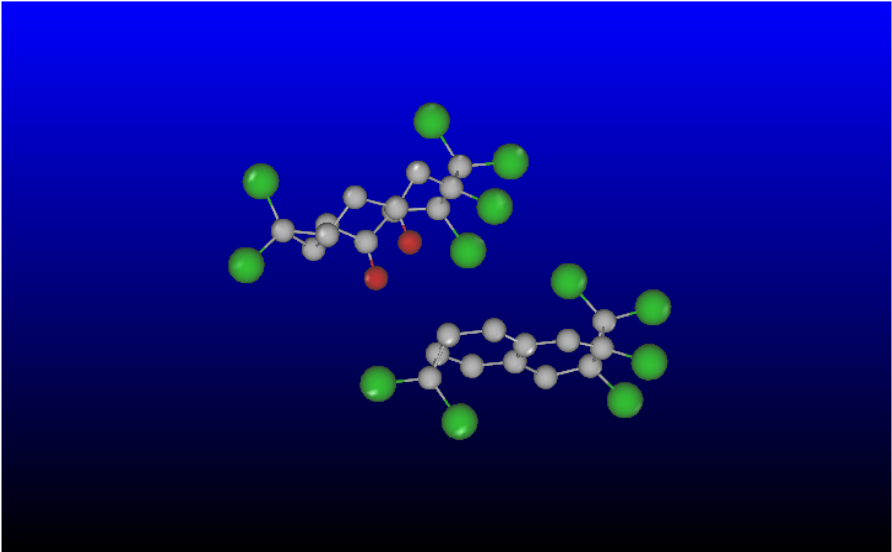
APEX2 v2014.11-0 - User: (guest) - Sample: 1570 - Licensed to Bruker Instrument User at University of Saskatchewan - [Structure Solution]

Sample Instrument Windows Help

Setup Evaluate Collect Integrate Scale Examine Data Solve Structure

Auto-Structure Structure Solution View Molecule

Instructions Listing Results Structure View



0 No. of QPeaks: 0 0 Zoom

Instruction File 1570\_0m.ins  
Reflection File 1570\_0m.hkl  
Formula C<sub>20</sub>H<sub>30</sub>O<sub>4</sub>Cl<sub>2</sub> Z 4 Update  
 Chiral  Non-centrosymmetric  Any

Statistics  
Reflections 32784 Unique 6763  
Rejected 0 Observed 5724  
R(int) [%] 2.6 % Observed 85  
R(sigma) [%] 2.6 % Observed in 1.1-1.2Å Shell 96

Method  
 Intrinsic Phasing Intrinsic 1  
 Direct Direct 1  
 Dual Space Dual Space 1  
 Patterson Patterson 1 Expand

Intrinsic Phasing Solutions  
[a] P-1  
R1 [%] 13.20  
alpha 0.030

Space group determination: 0.010 secs

R1	Rweak	Alpha	Orientation	Space group	Flack_x	File	Formula
0.132	0.005	0.030	as input	P-1		1570_0m_a	C <sub>24</sub> O <sub>2</sub> Cl <sub>12</sub>

Assign elements and isotropic refinement 1.566 secs

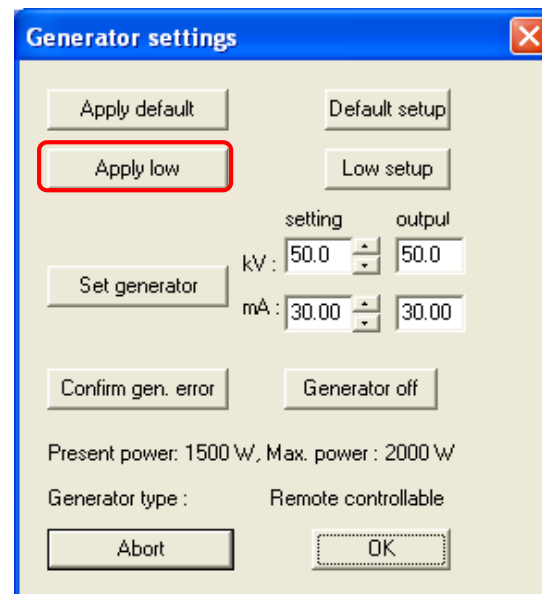
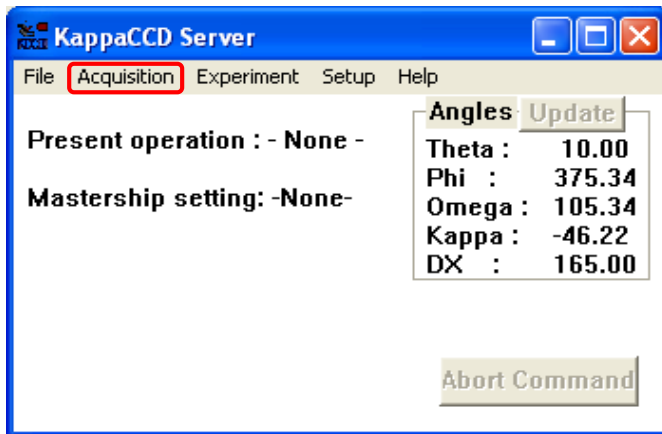
++++  
+ XT finished at 16:57:17 Total time: 3.389 secs +  
++++

Refine Structure Report Instrument

Solve Structure Stop after Iteration  
Reset Exit

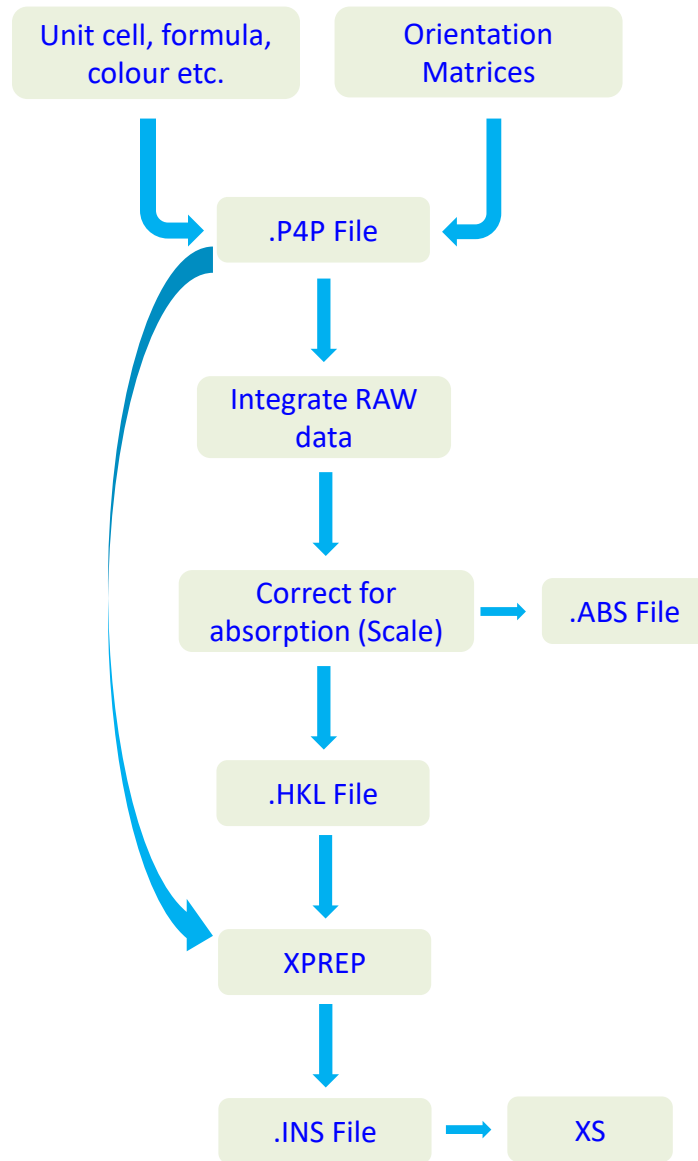
## 17. Turn off the Diffractometer

- 1) Remove the crystal from the pin.
- 2) Set Cryojet to RT, turned it off after returning to RT.
- 3) Turn off APEX2 and then BIS.
- 4) Lower X-Ray power under 'Acquisition → X-Ray Generator'.
- 5) Turn off the X-Ray tube. Keep the Haskris running for ~30 mins.
- 6) Turn off the Haskris and cooling water.





# Appendix 1. Data Processing Flow Chart



## Appendix 2. Four Circle Diffractometer



CCD Detector

Phi ( $\varphi$ )

Kappa ( $\kappa$ )

2-theta ( $2\theta$ )

Liquid N<sub>2</sub>

Microscope &  
video camera

X-ray Beam

Omega ( $\omega$ )