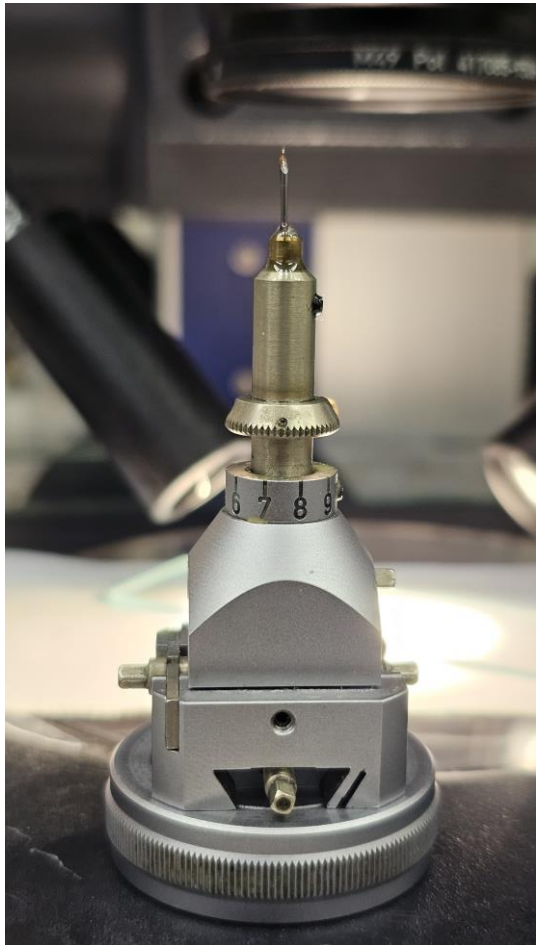
Single Crystal X-ray Diffractometer

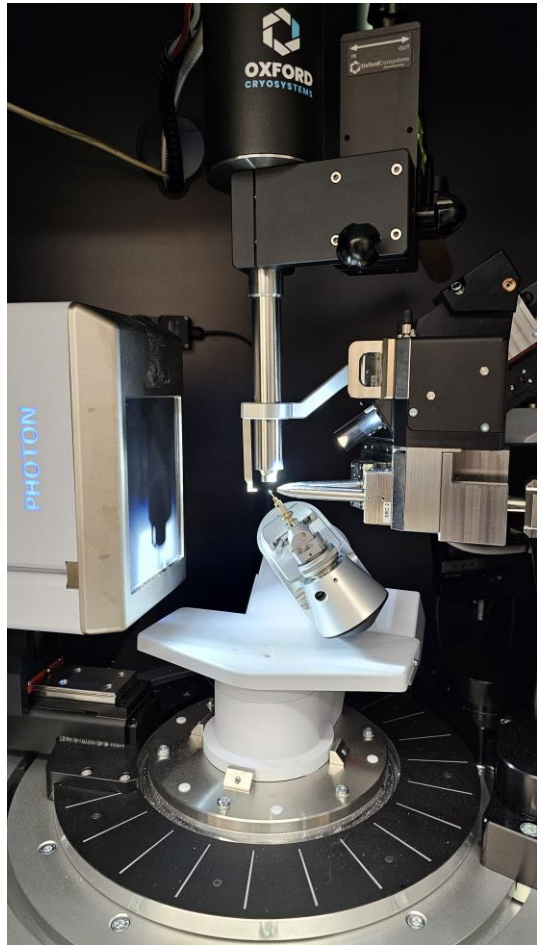
Procedure



- Find a good quality single crystal.
- Mount it on to the goniometer loop.
- Attach the goniometer to the SCXRD instrument.



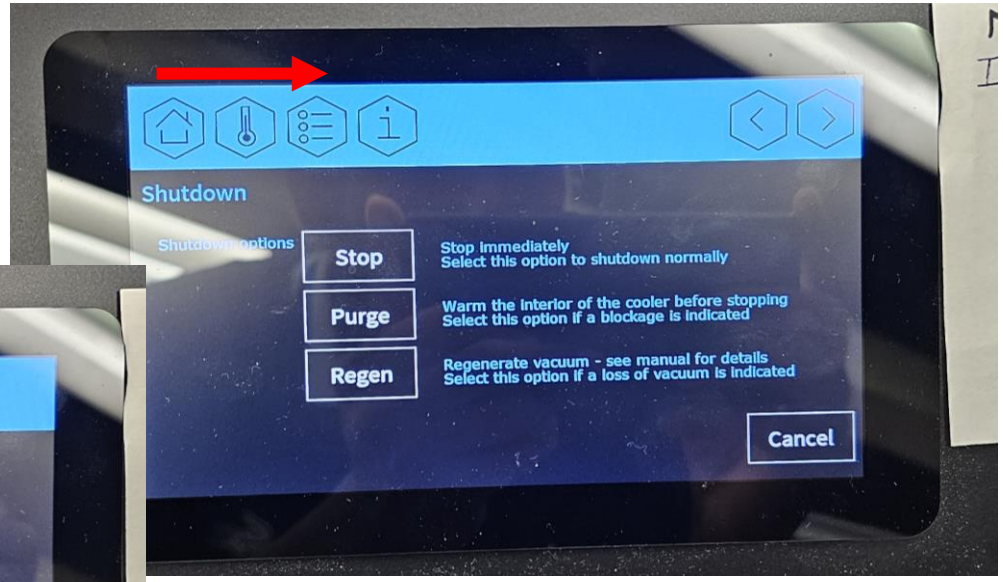


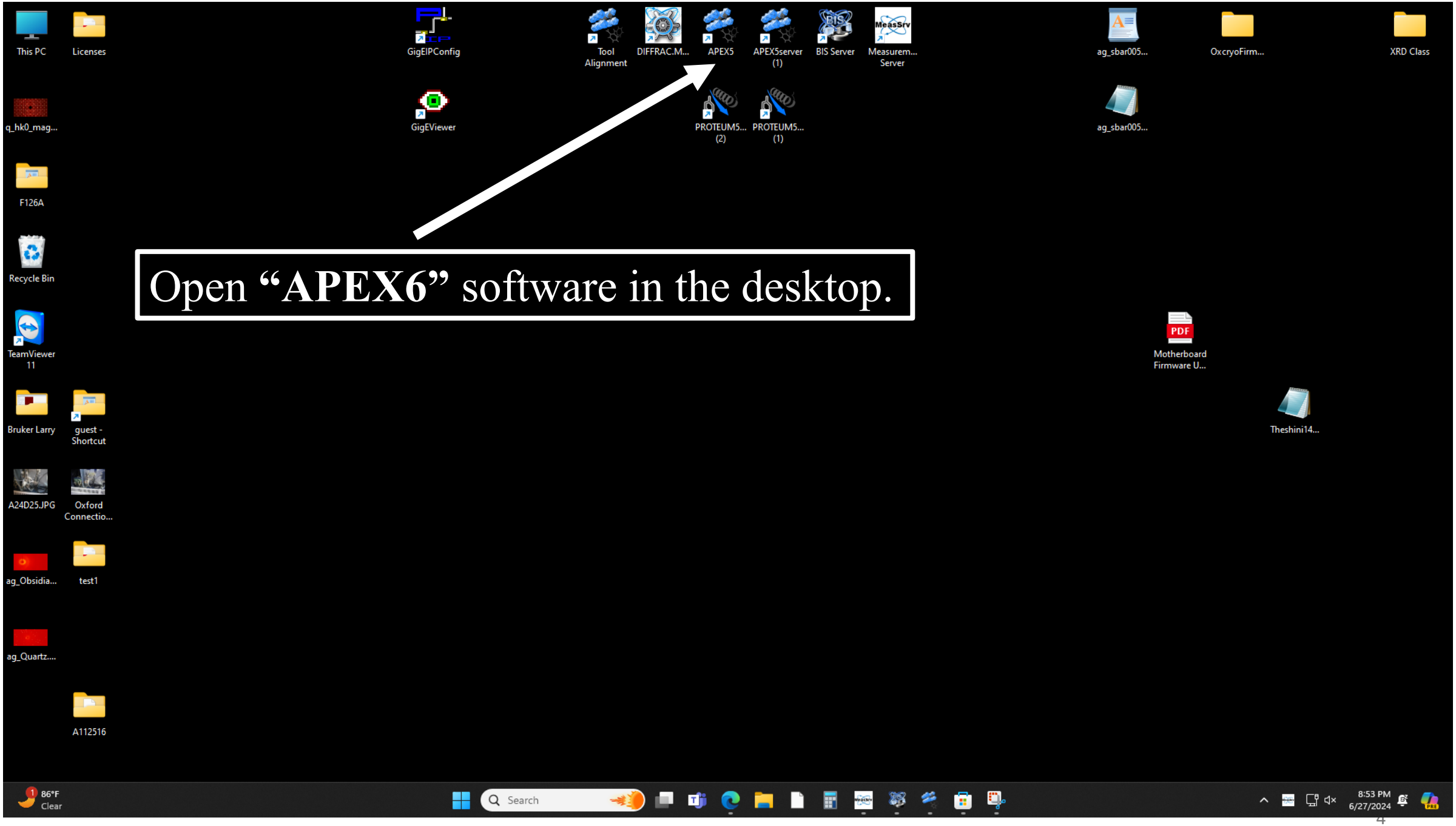


- Check the liquid nitrogen dewar and make sure it contains enough nitrogen to run the experiment
- To switch on the cryosystem click ***"start"***, set the desired temperature for the measurement & then click ***"ok"***.
- To switch off click ***"Stop"***.

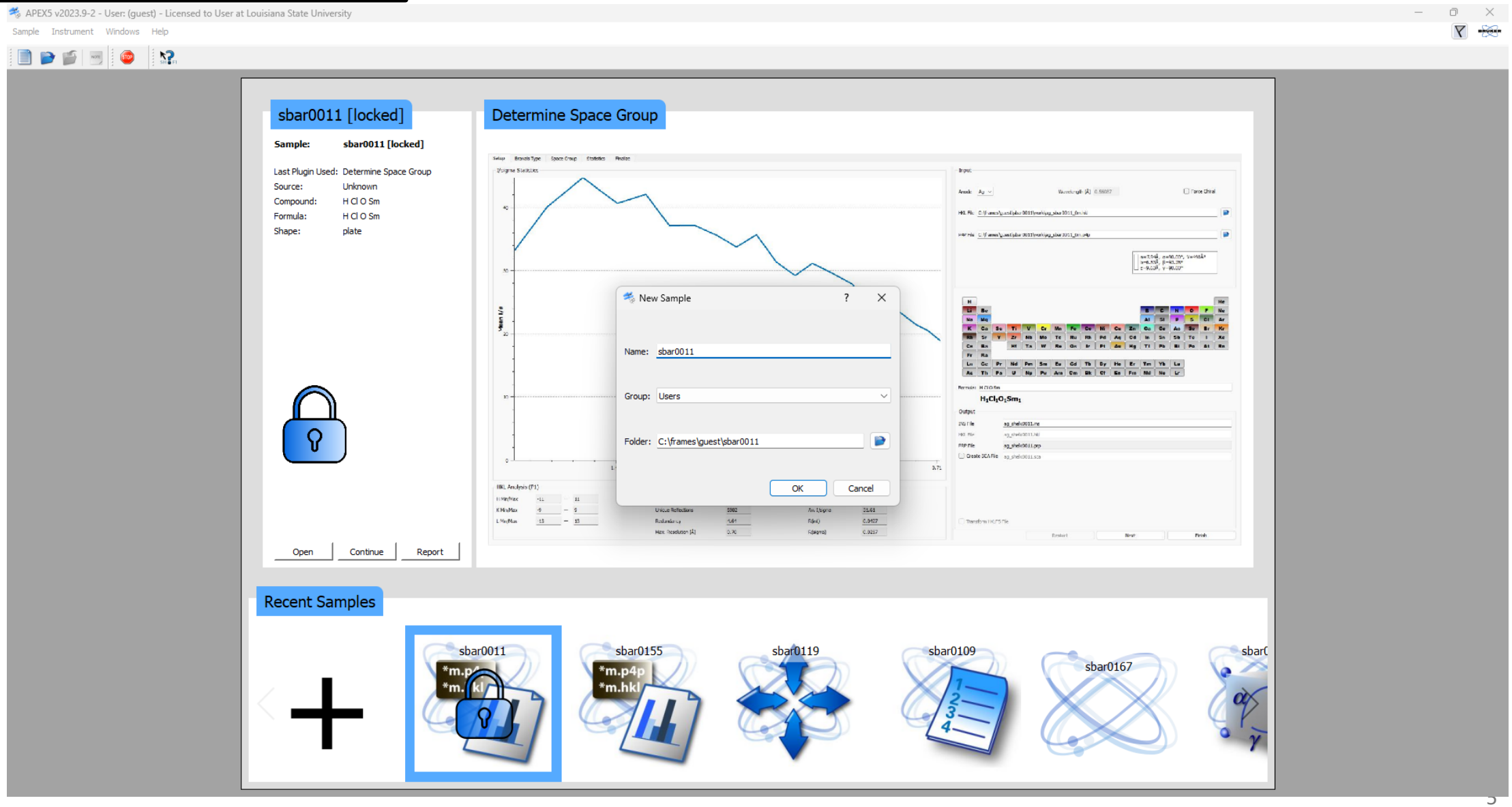




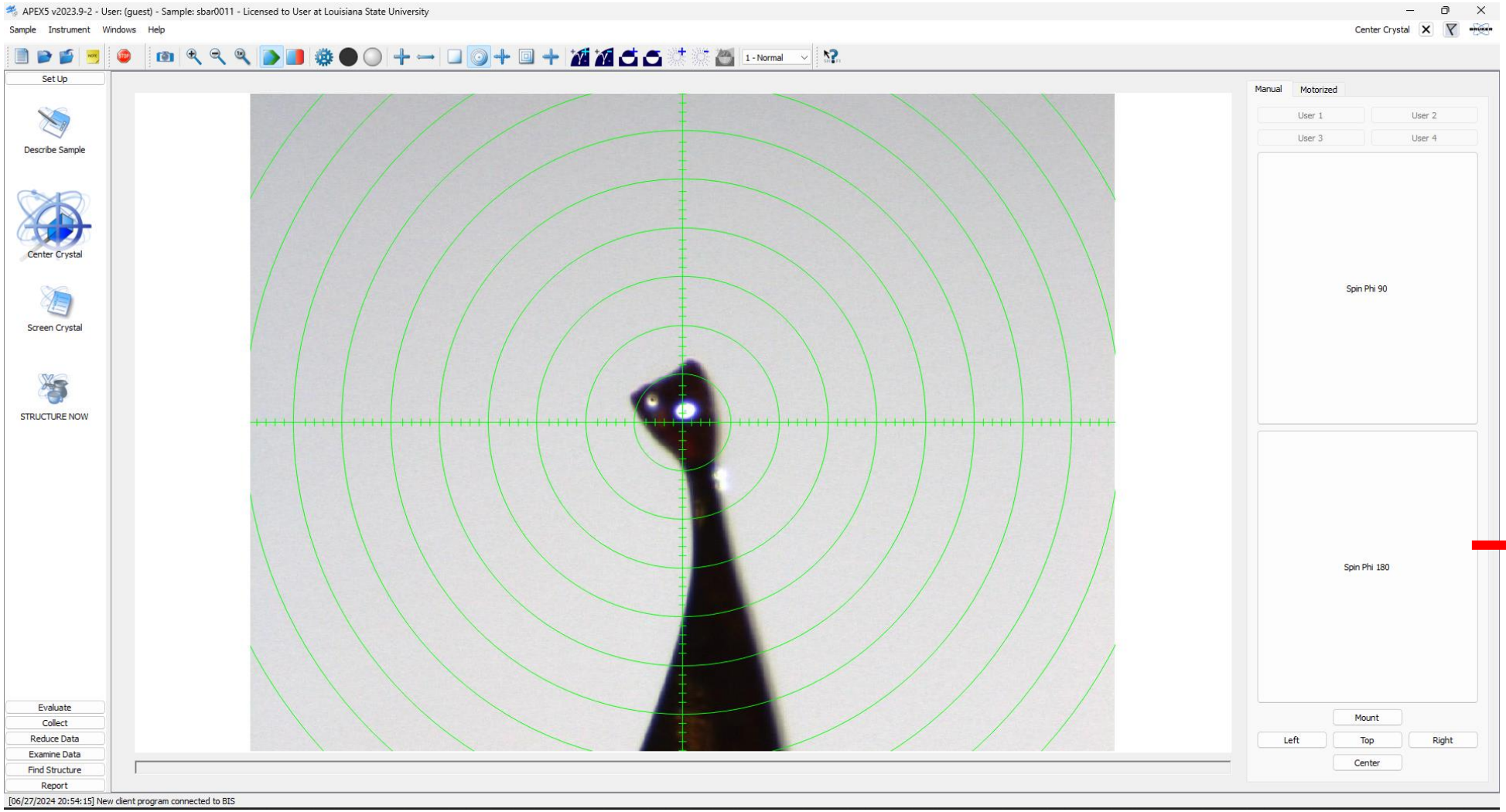


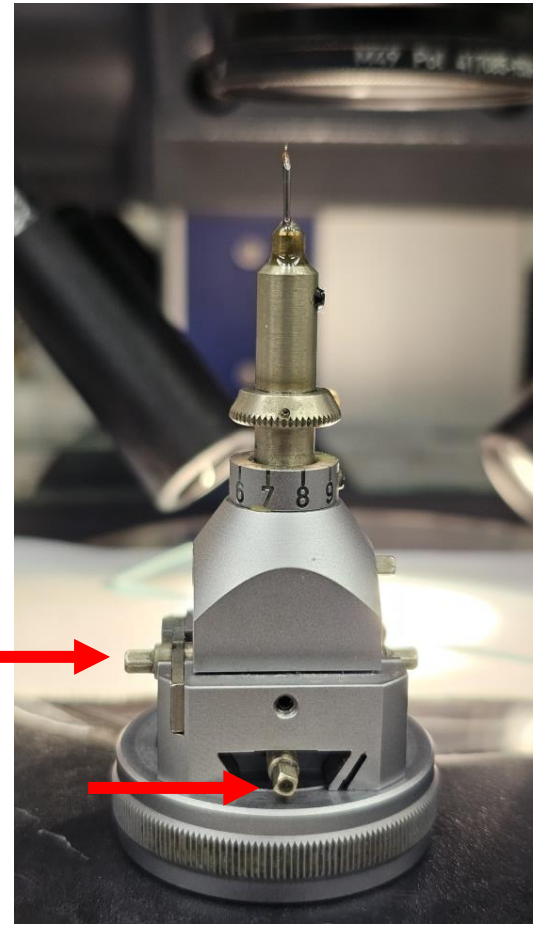

This PC
Licenses
GigEIPConfig
Tool Alignment
DIFFRAC.M...
APEX5
APEX5server (1)
BIS Server
Measurem... Server
ag_sbar005...
OxcryoFirm...
XRD Class
q_hk0_mag...
GigEViewer
PROTEUM5... (2)
PROTEUM5... (1)
ag_sbar005...
F126A
Recycle Bin
Open "APEX6" software in the desktop.
TeamViewer 11
Motherboard Firmware U...
Bruker Larry
guest - Shortcut
Theshini14...
A24D25.JPG
Oxford Connectio...
ag_Obsidia...
test1
ag_Quartz....
A112516
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Search
8:53 PM 6/27/2024

Create a new project



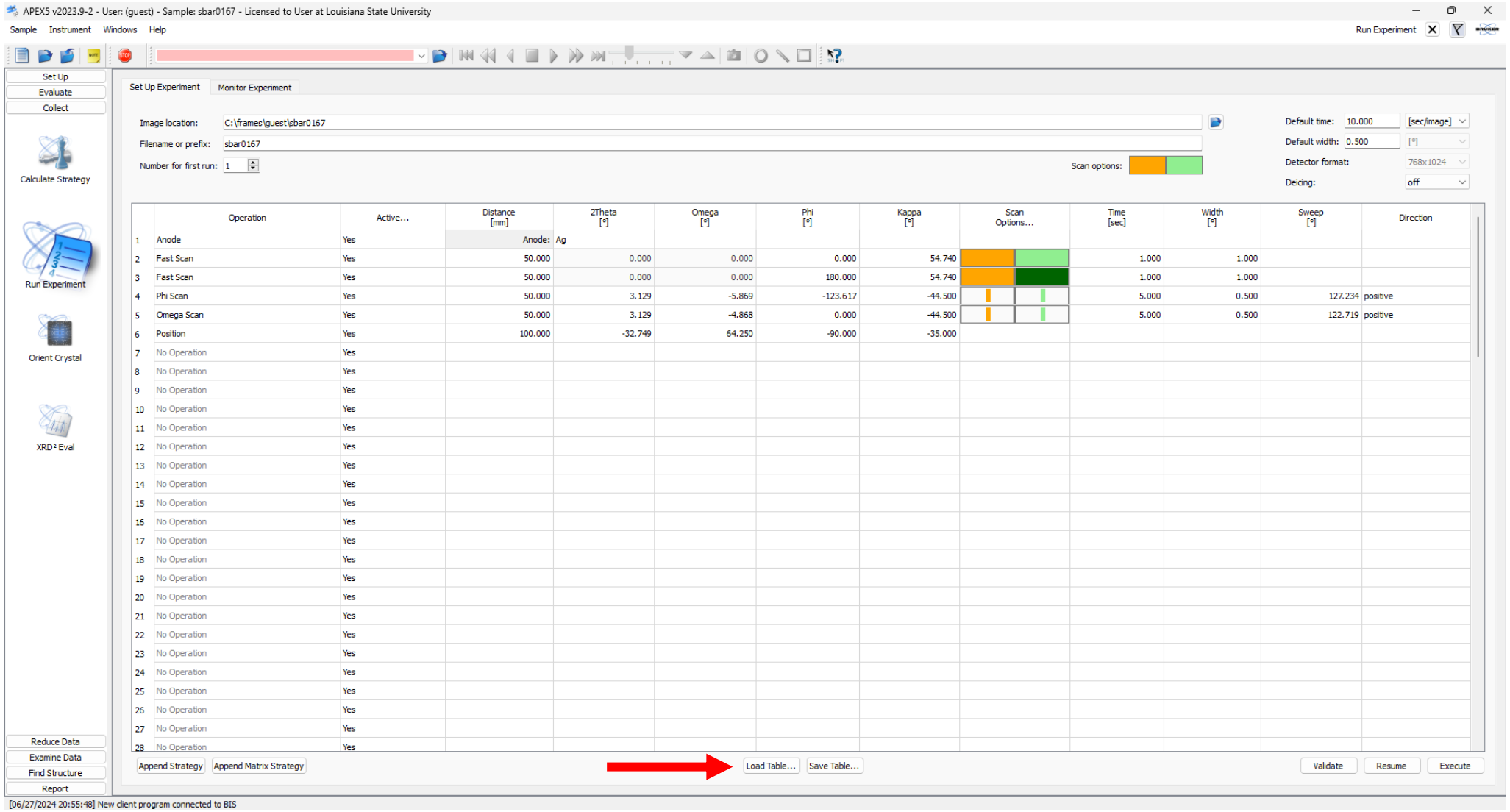
- Go to ***"set up"*** then ***"center crystal".***
- Center the crystal using Screws in goniometer. Rotate the crystal and try to align it in different directions.





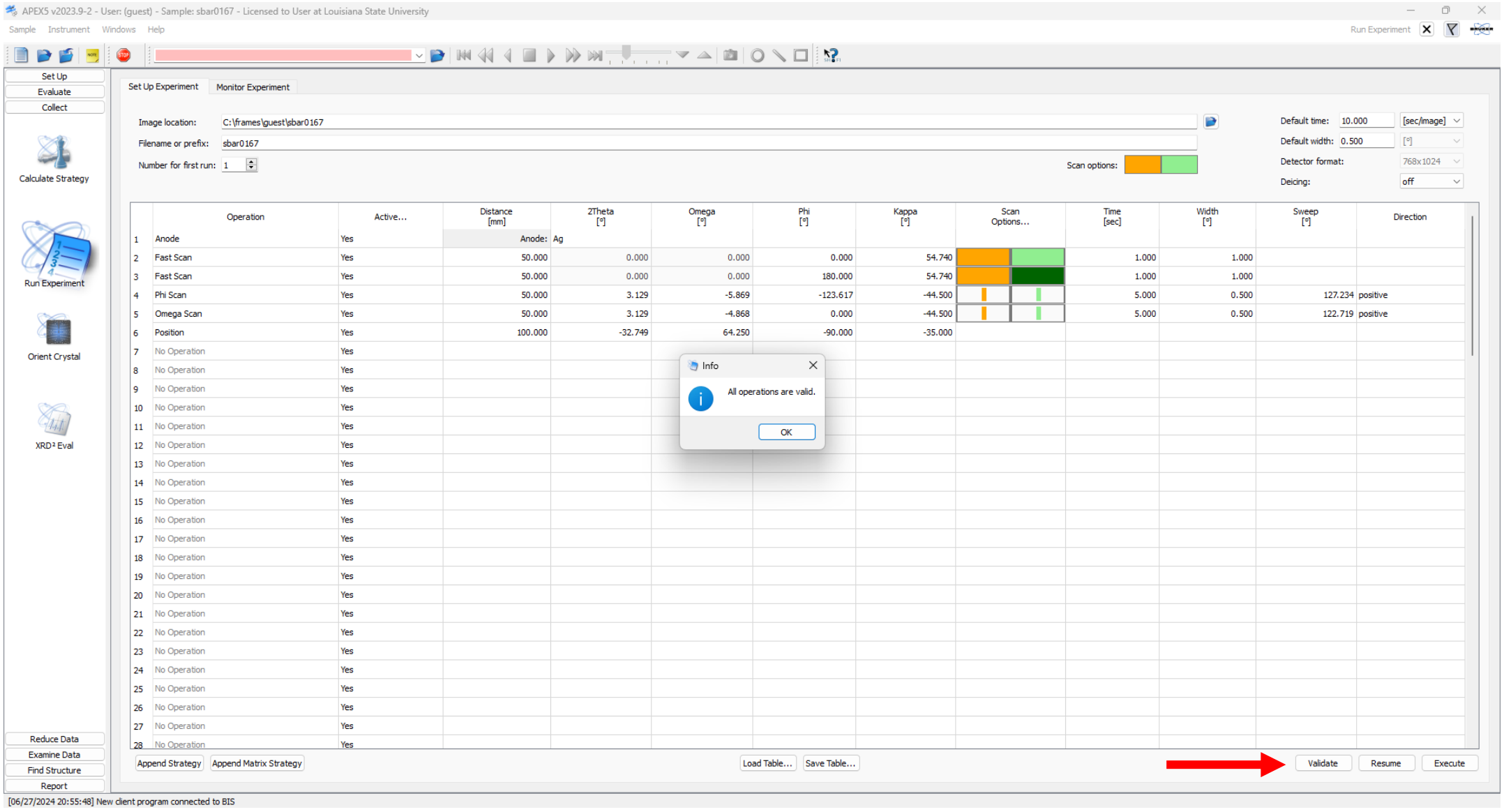
How to Determine the Unit Cell of a Crystal

- Go to ***"Collect"*** then ***"Run Experiment"***, after that go to ***"Set Up Experiment"***.
- Load the table ***"matrix strategy"***.



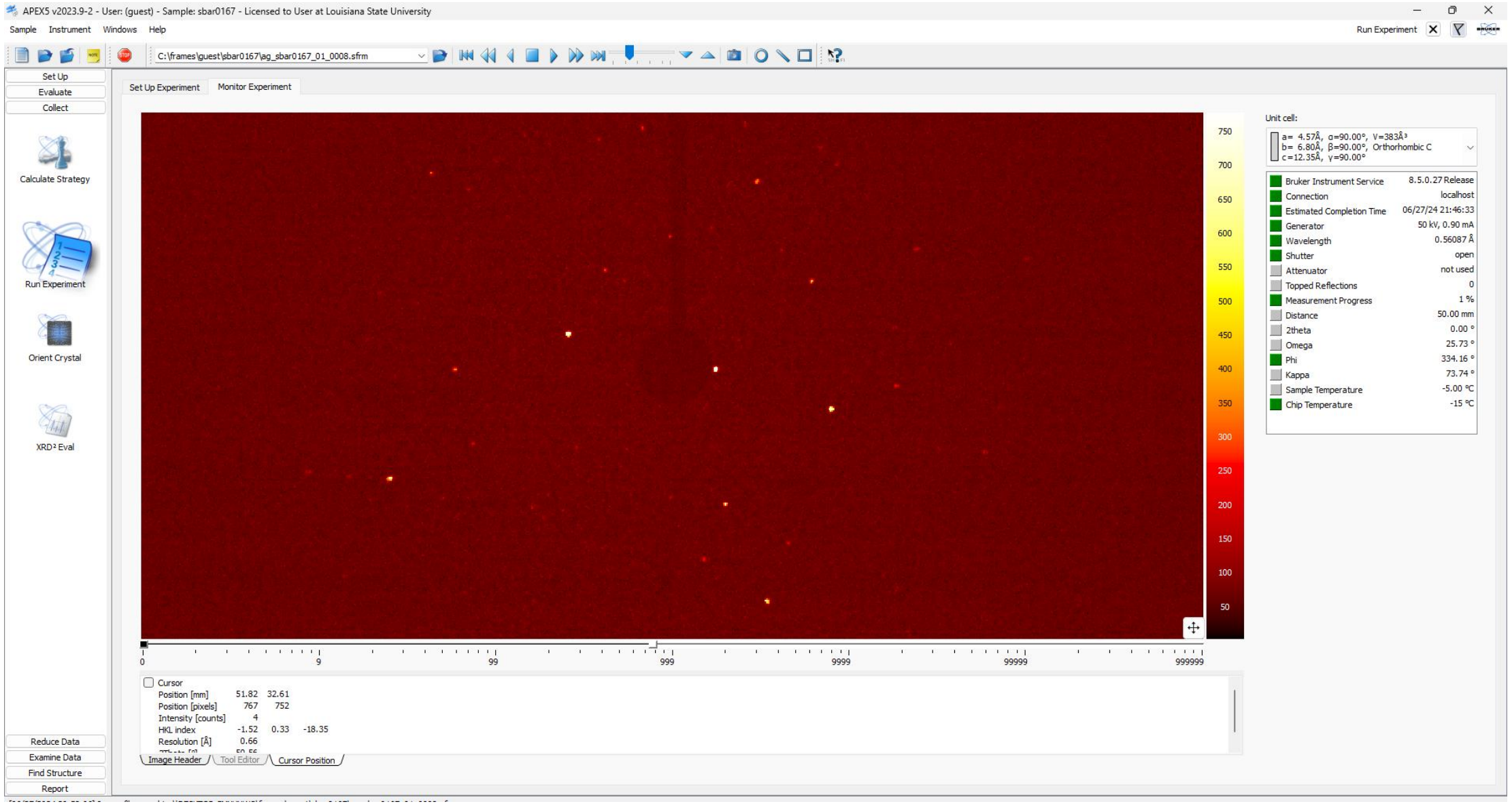
	Operation	Active...	Distance [mm]	2Theta [°]	Omega [°]	Phi [°]	Kappa [°]	Scan Options...	Time [sec]	Width [°]	Sweep [°]	Direction
1	Anode	Yes	Anode:	Ag								
2	Fast Scan	Yes	50.000	0.000	0.000	0.000	54.740		1.000	1.000		
3	Fast Scan	Yes	50.000	0.000	0.000	180.000	54.740		1.000	1.000		
4	Phi Scan	Yes	50.000	3.129	-5.869	-123.617	-44.500		5.000	0.500	127.234	positive
5	Omega Scan	Yes	50.000	3.129	-4.868	0.000	-44.500		5.000	0.500	122.719	positive
6	Position	Yes	100.000	-32.749	64.250	-90.000	-35.000					
7	No Operation	Yes										
8	No Operation	Yes										
9	No Operation	Yes										
10	No Operation	Yes										
11	No Operation	Yes										
12	No Operation	Yes										
13	No Operation	Yes										
14	No Operation	Yes										
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23	No Operation	Yes										
24	No Operation	Yes										
25	No Operation	Yes										
26	No Operation	Yes										
27	No Operation	Yes										
28	No Operation	Yes										

➢Validate & Execute.

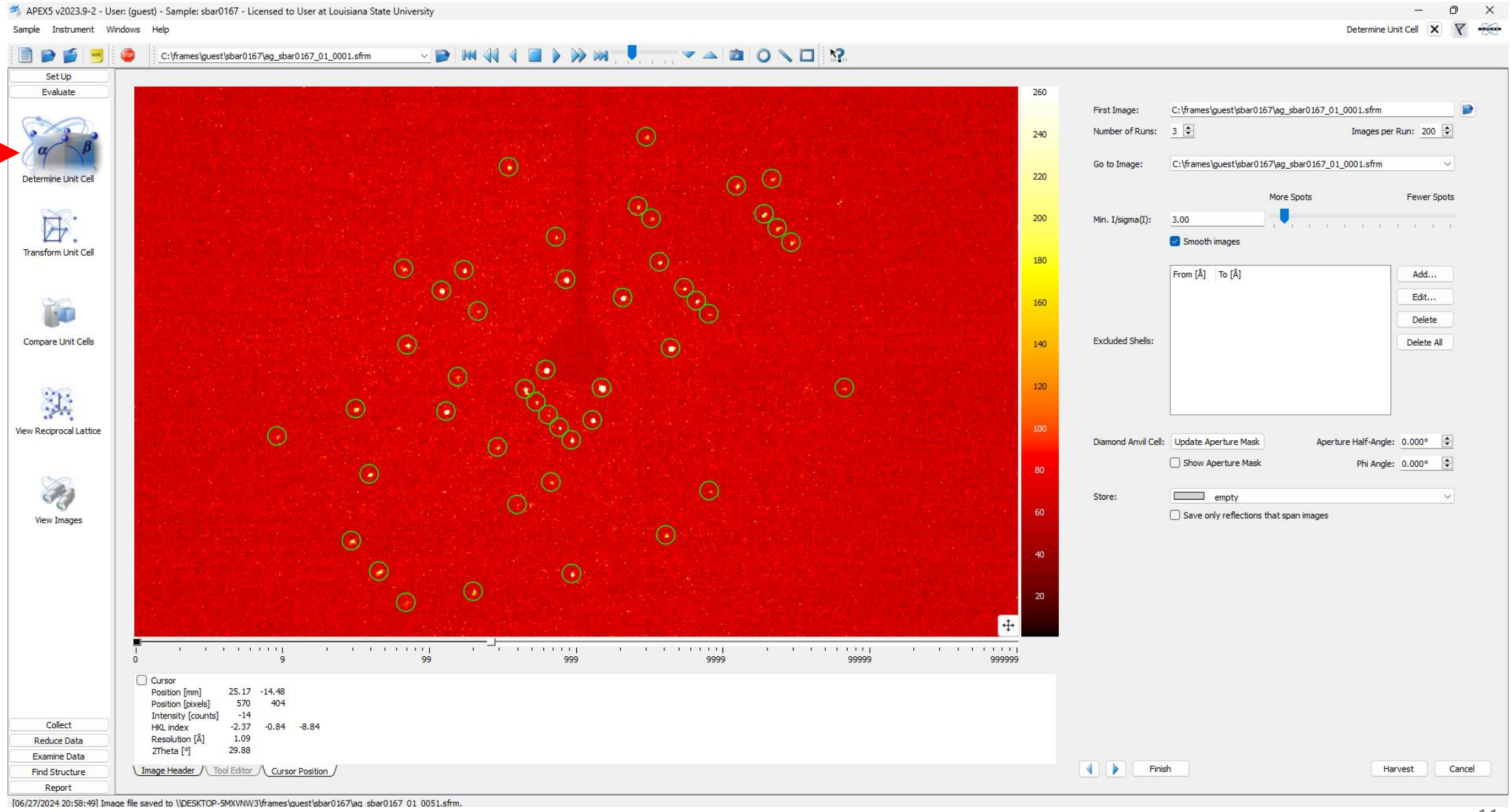


	Operation	Active...	Distance [mm]	2Theta [°]	Omega [°]	Phi [°]	Kappa [°]	Scan Options...	Time [sec]	Width [°]	Sweep [°]	Direction
1	Anode	Yes	Anode:	Ag								
2	Fast Scan	Yes	50.000	0.000	0.000	0.000	54.740		1.000	1.000		
3	Fast Scan	Yes	50.000	0.000	0.000	180.000	54.740		1.000	1.000		
4	Phi Scan	Yes	50.000	3.129	-5.869	-123.617	-44.500		5.000	0.500	127.234	positive
5	Omega Scan	Yes	50.000	3.129	-4.868	0.000	-44.500		5.000	0.500	122.719	positive
6	Position	Yes	100.000	-32.749	64.250	-90.000	-35.000					
7	No Operation	Yes										
8	No Operation	Yes										
9	No Operation	Yes										
10	No Operation	Yes										
11	No Operation	Yes										
12	No Operation	Yes										
13	No Operation	Yes										
14	No Operation	Yes										
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28	No Operation	Yes										

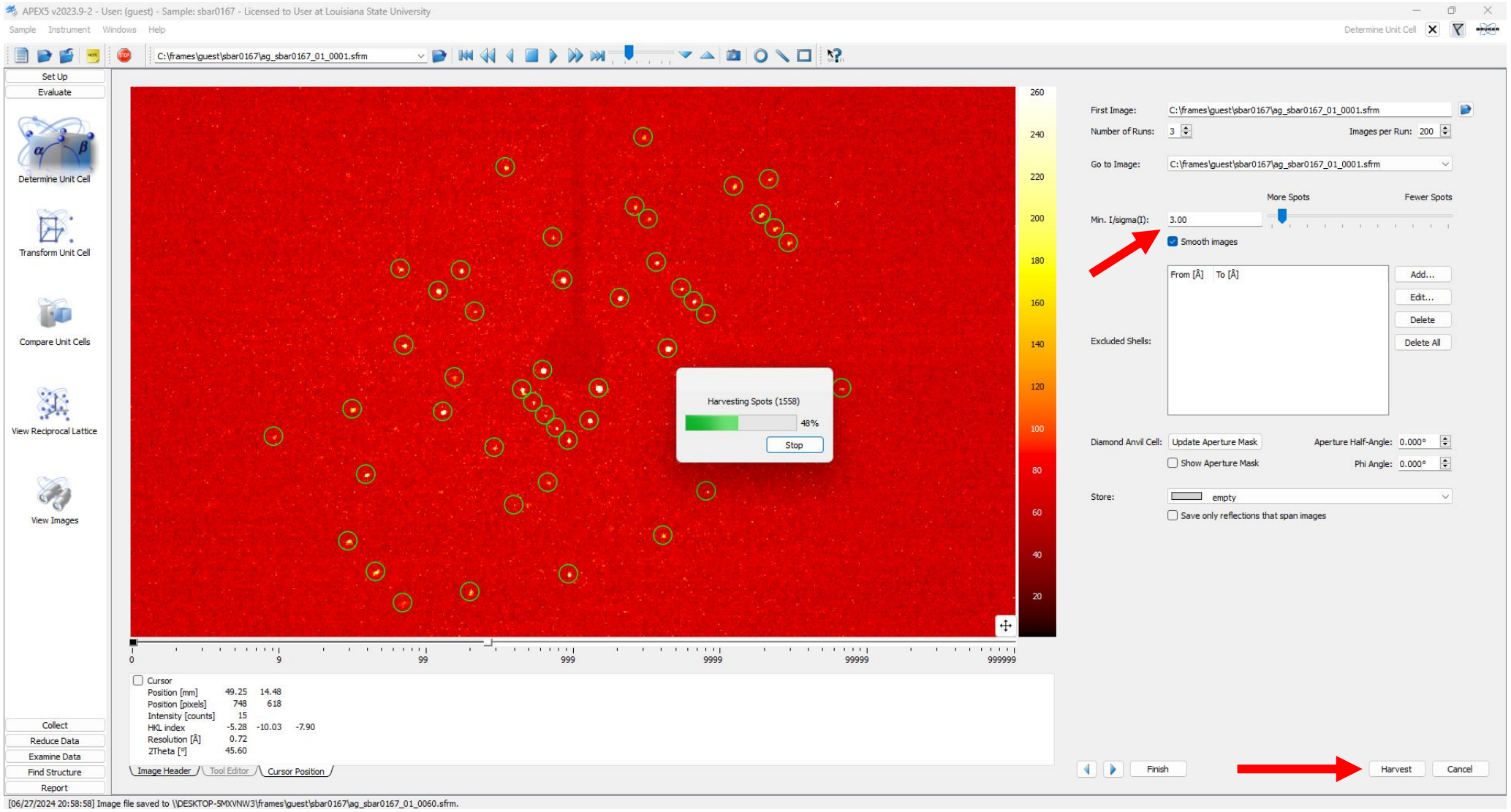
- Matrix frames are collecting now.



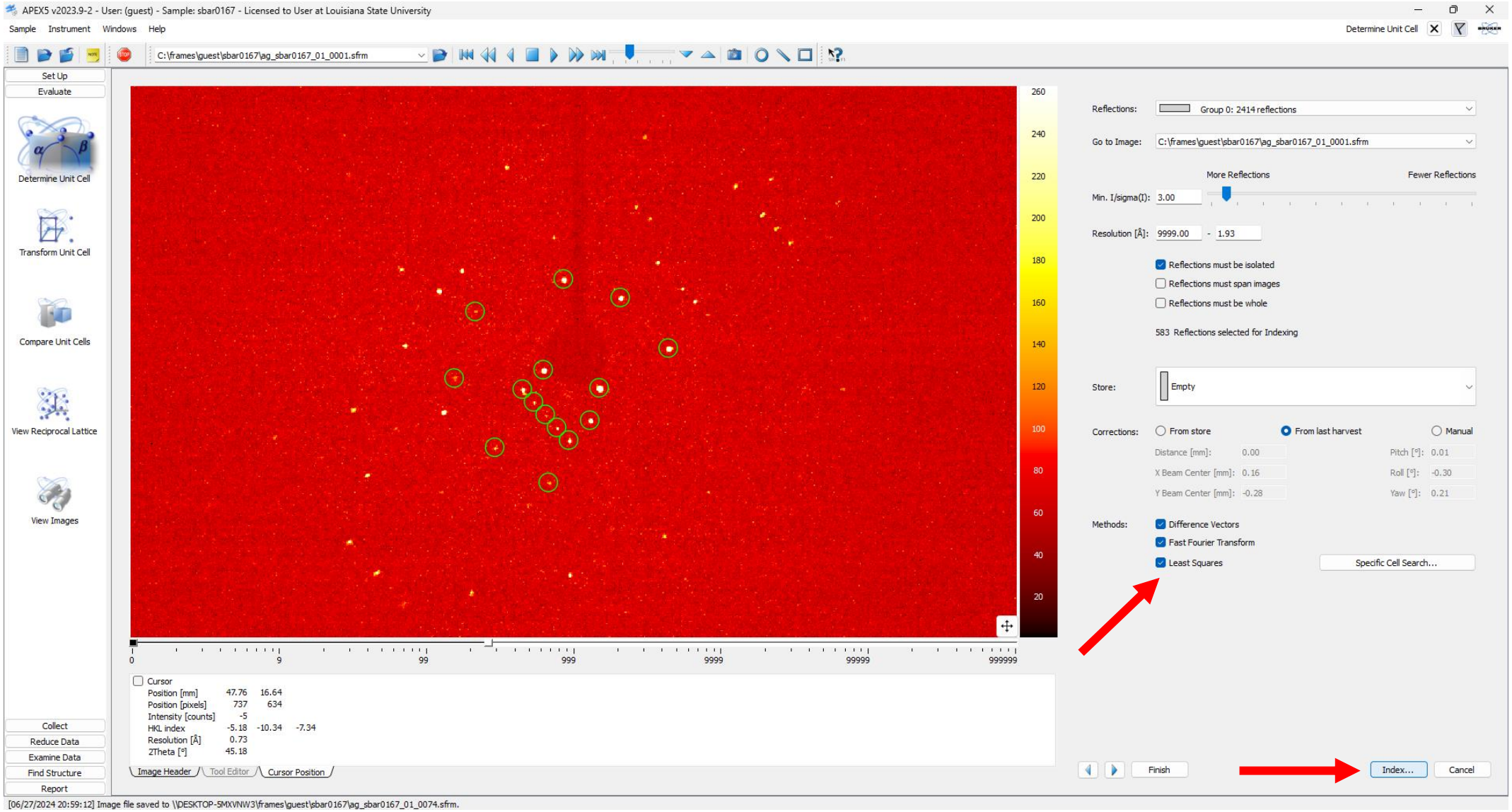
➢ Go to ***"Evaluate"*** then ***"Determine Unit Cell"*** and go to ***"Harvest Spots"***



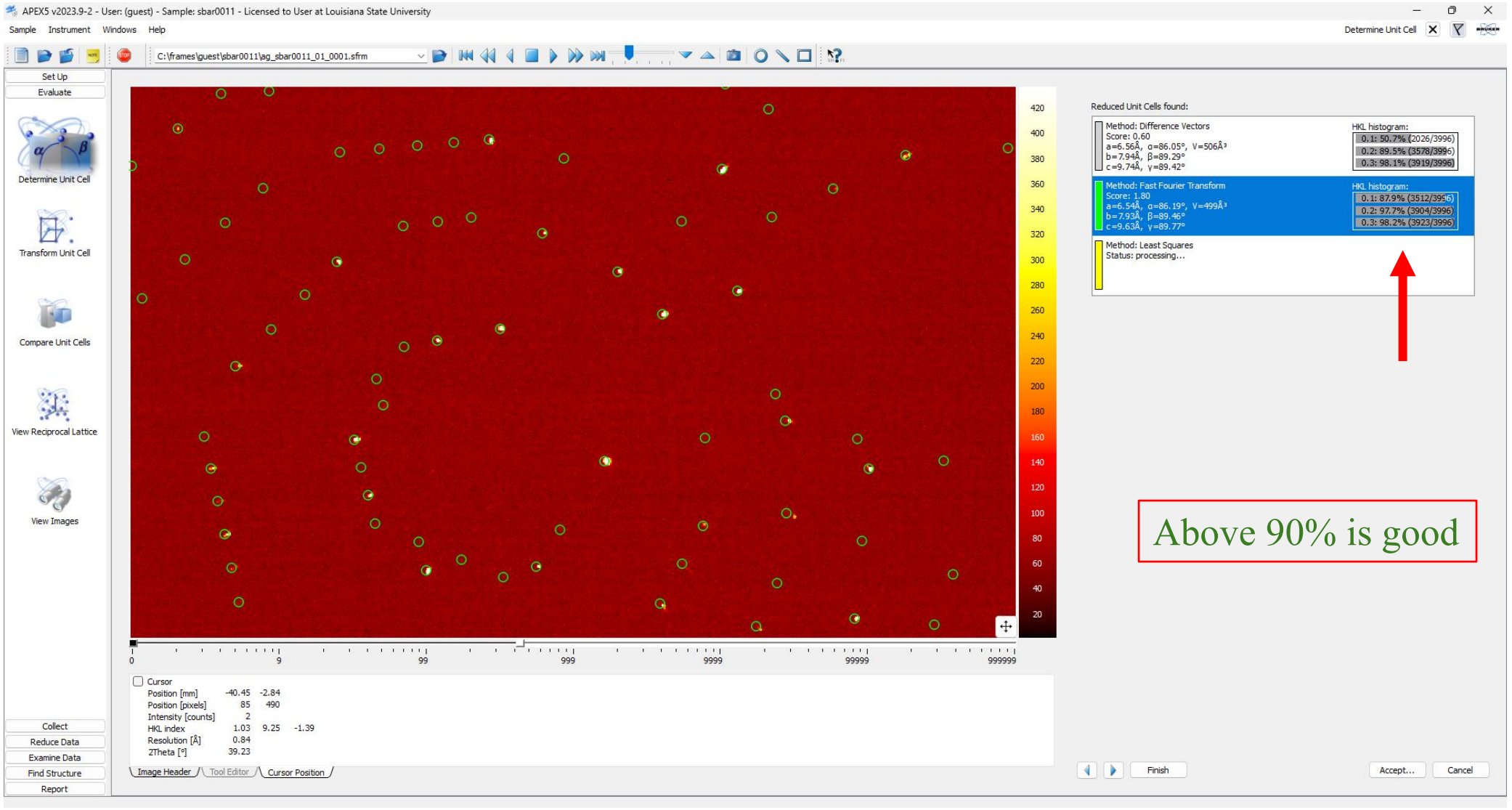
➢Select proper *No. of runs*, *Images per run & I/Sigma*. Then click ***"harvest"***.



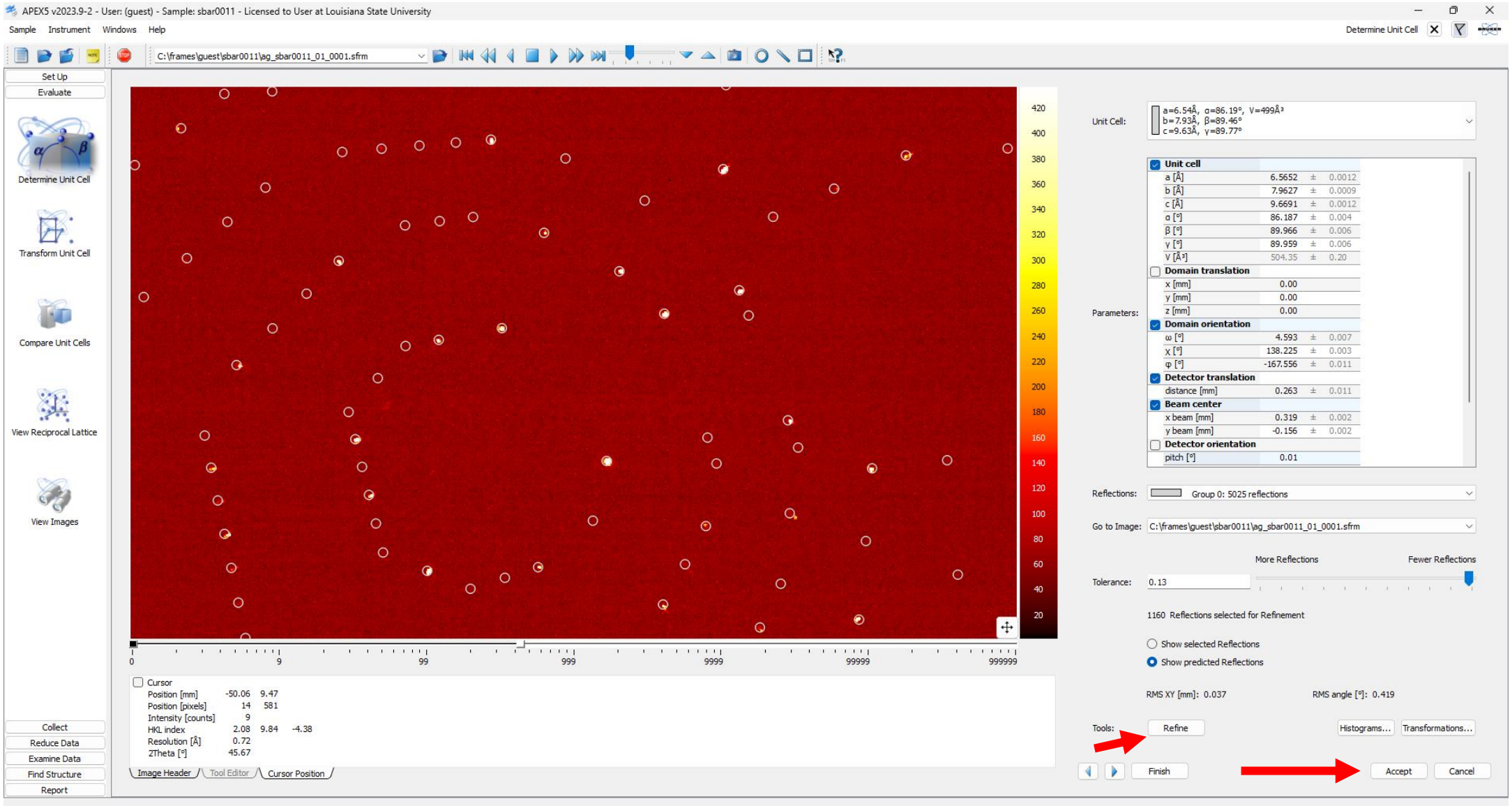
➢ Go to Index select all the methods and click ***"Index"***.



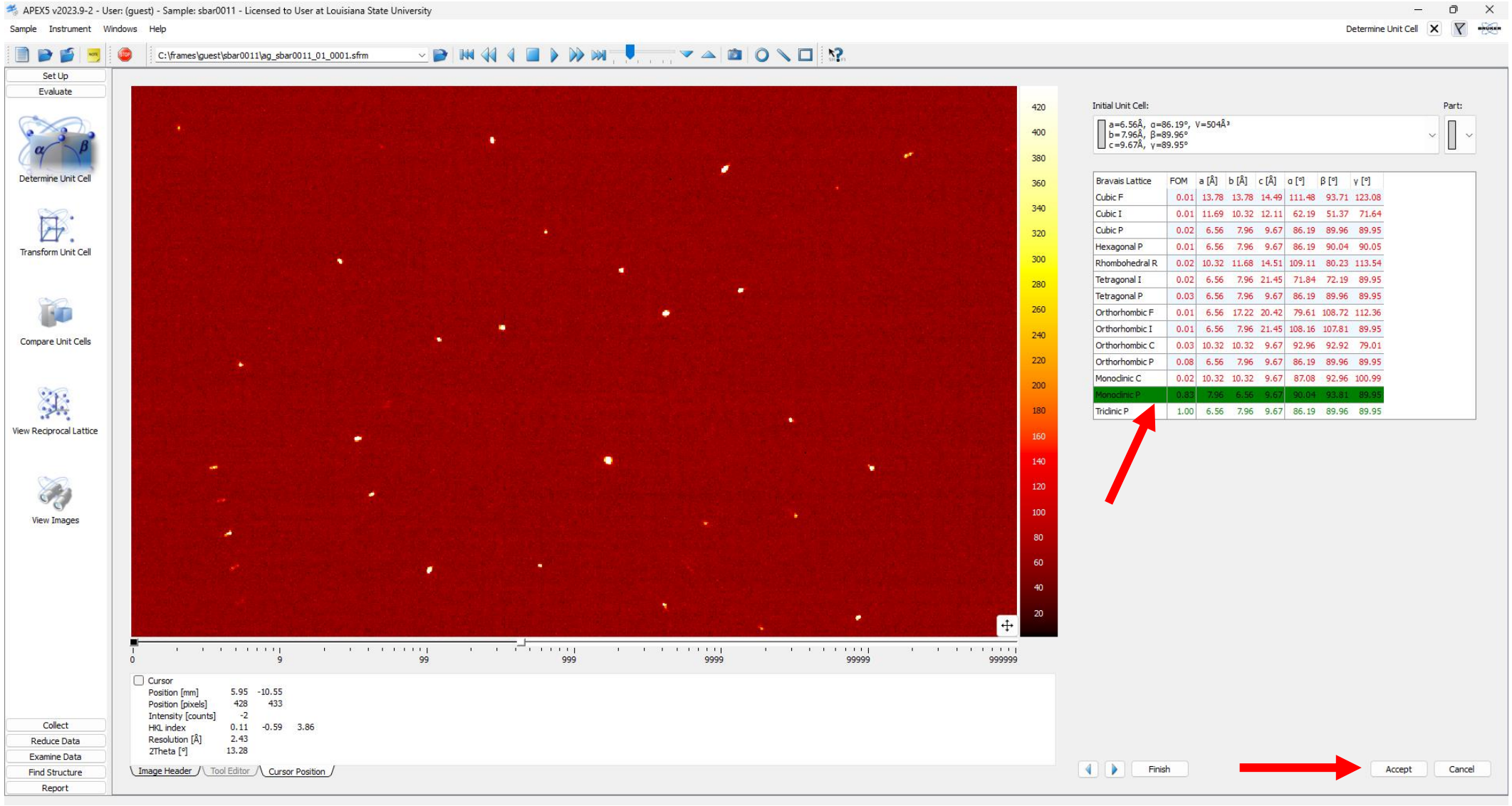
➢ Select the best method according to HKL statistics and click *"Accept"*.



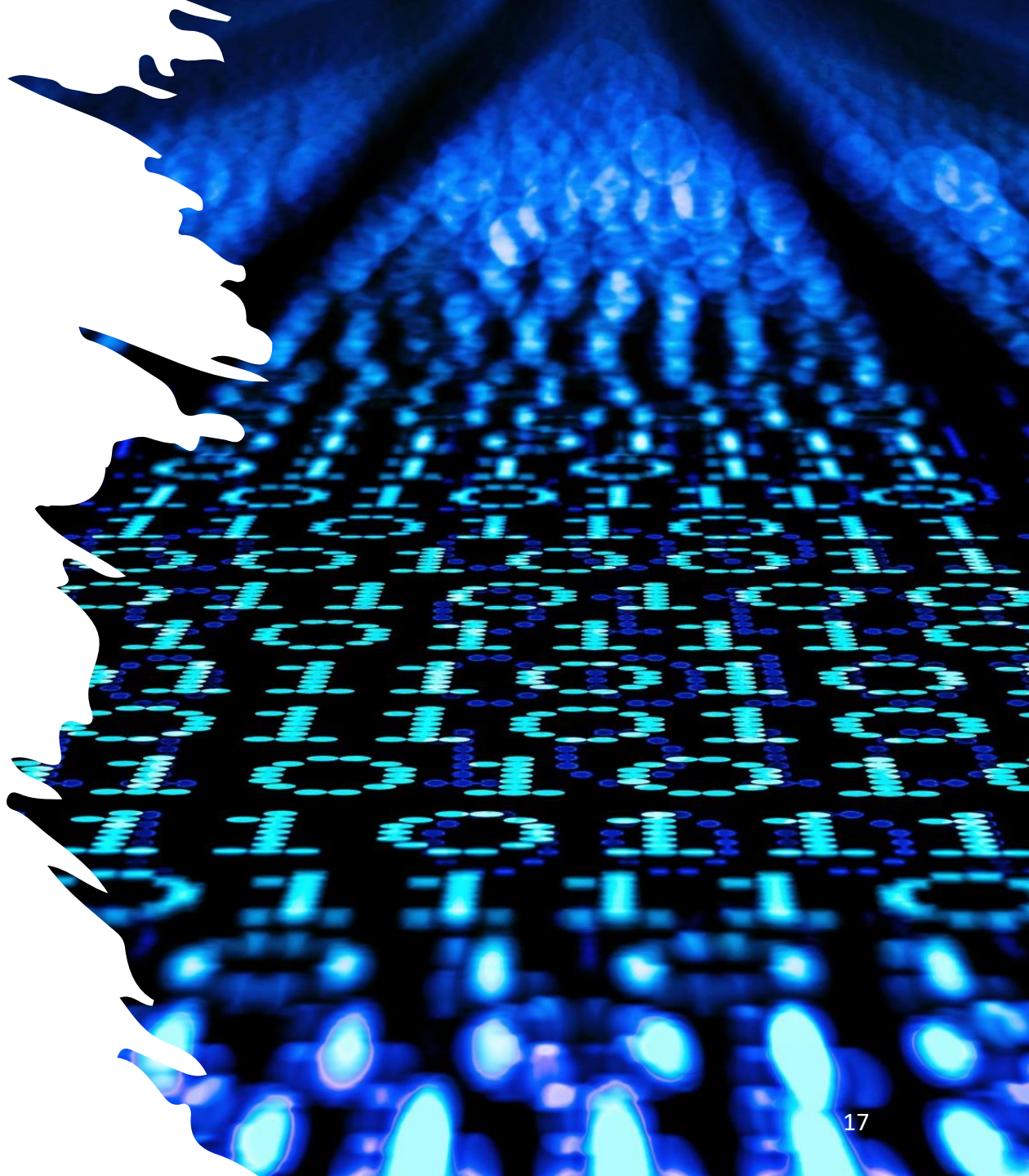
➢Then go to **Refine** and ***"Refine"*** it several times and click ***"Accept"***.



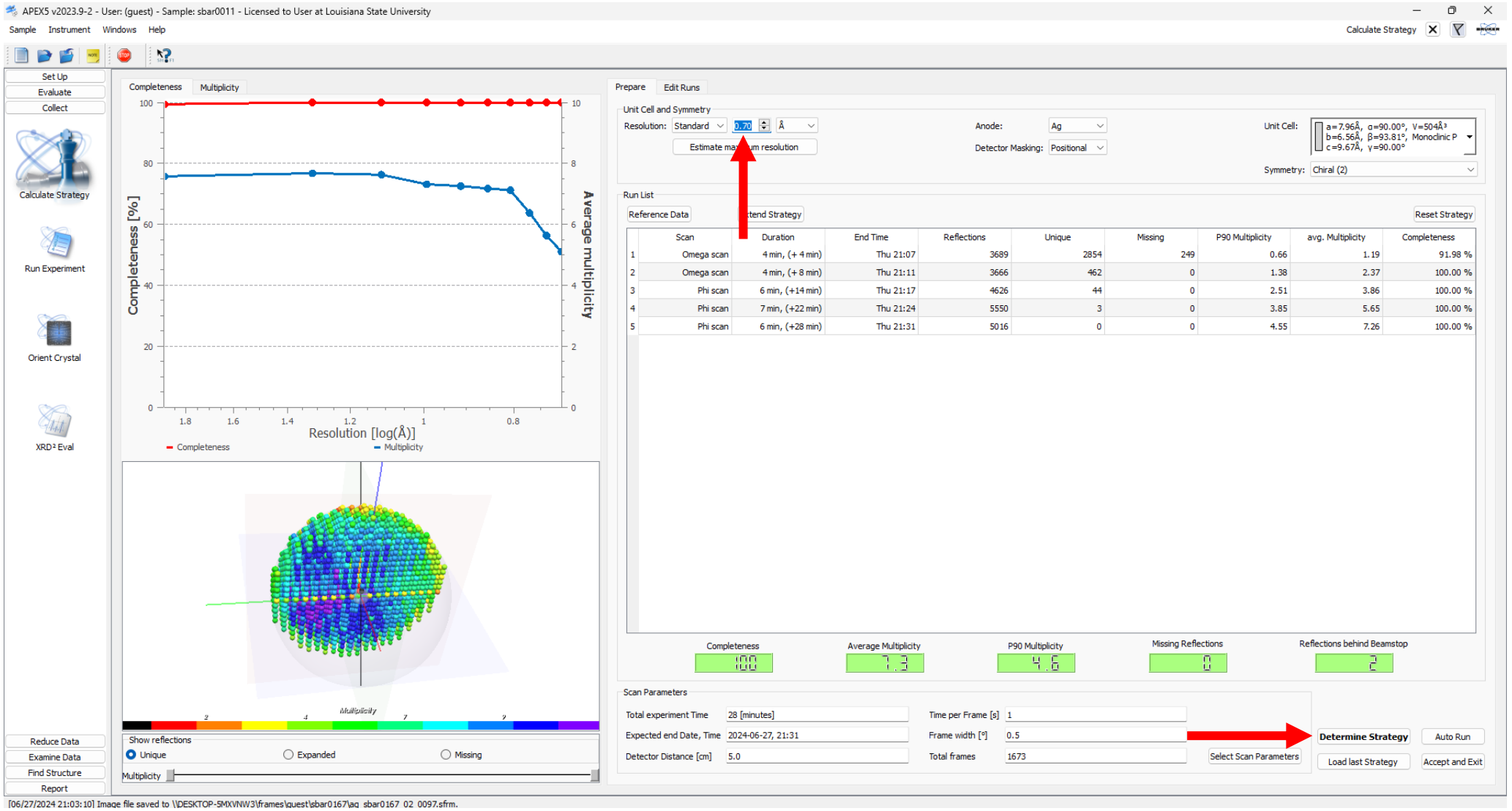
➢Then go to **Bravia** & select the correct **crystal lattice** and click ***"Accept"***.



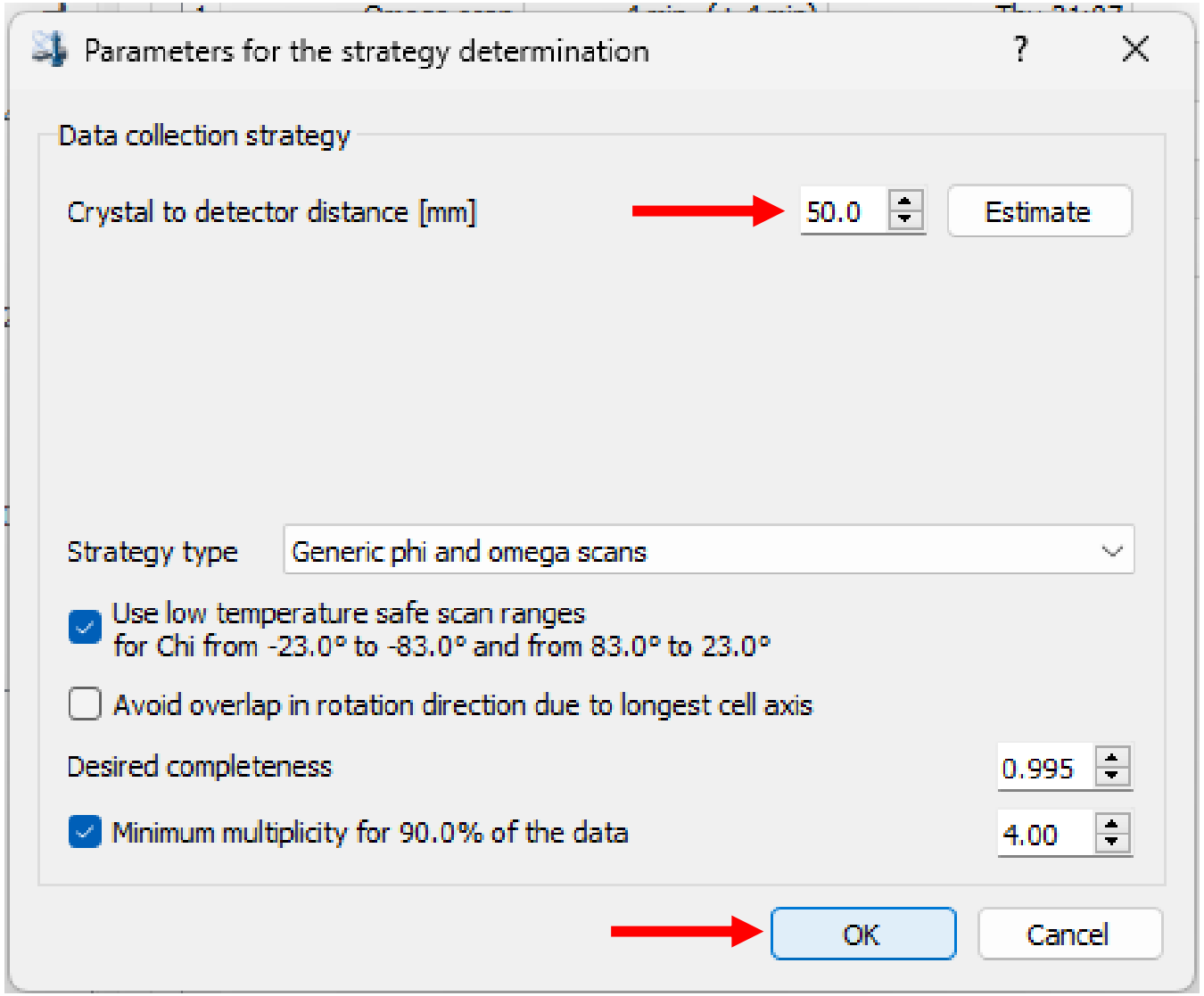
How to Set Up a Full Data Collection



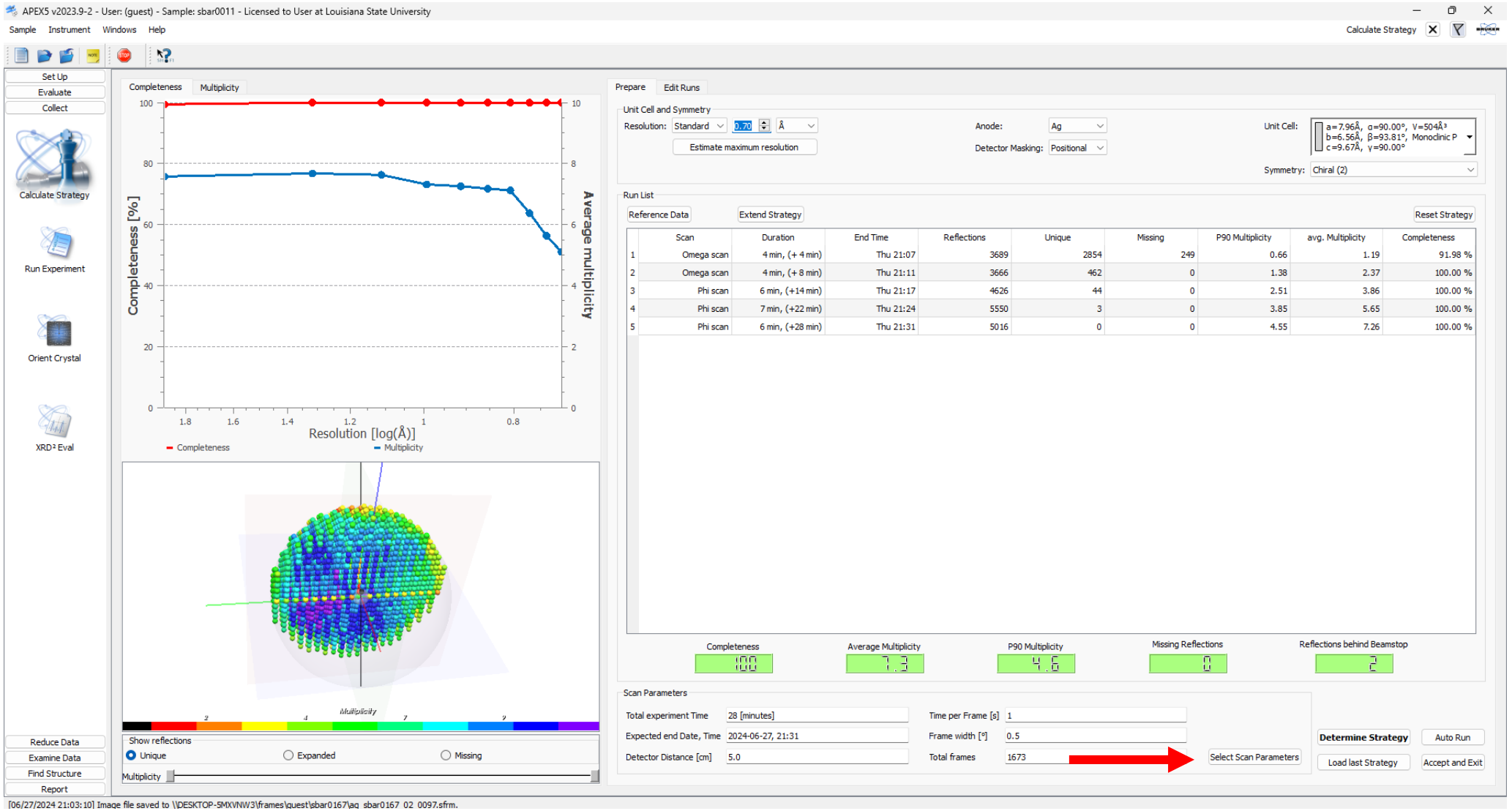
- After determined the unit cell, go to ***"Collect"*** & then go to **"Calculate Strategy"**. Change the **resolution to 0.70**. Then click ***"Determine Strategy"*.**



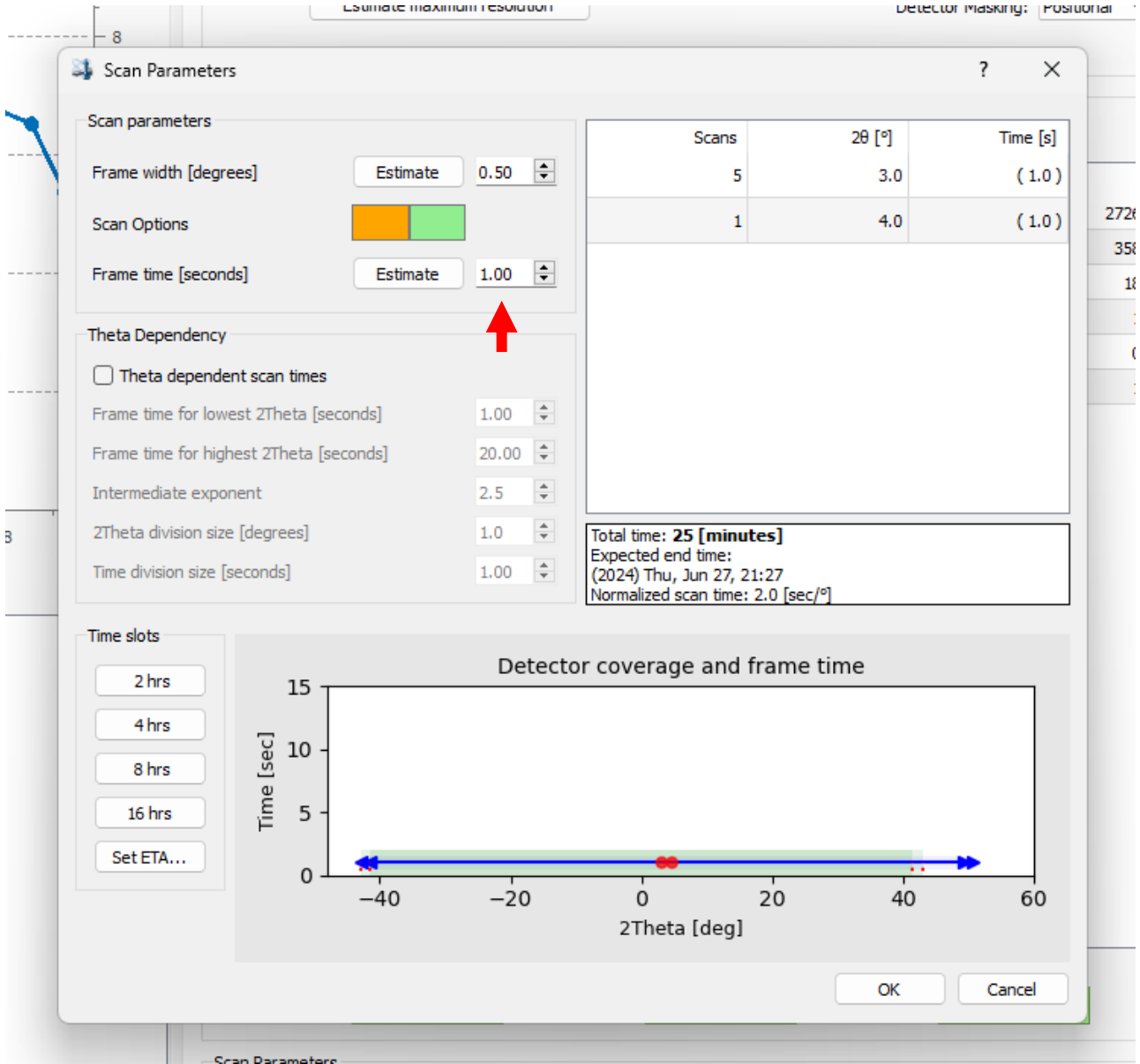
➢Change the crystal to **detector distance** to **50.0 mm** (If the unit cell is large 60.0) and click ***"OK"***.



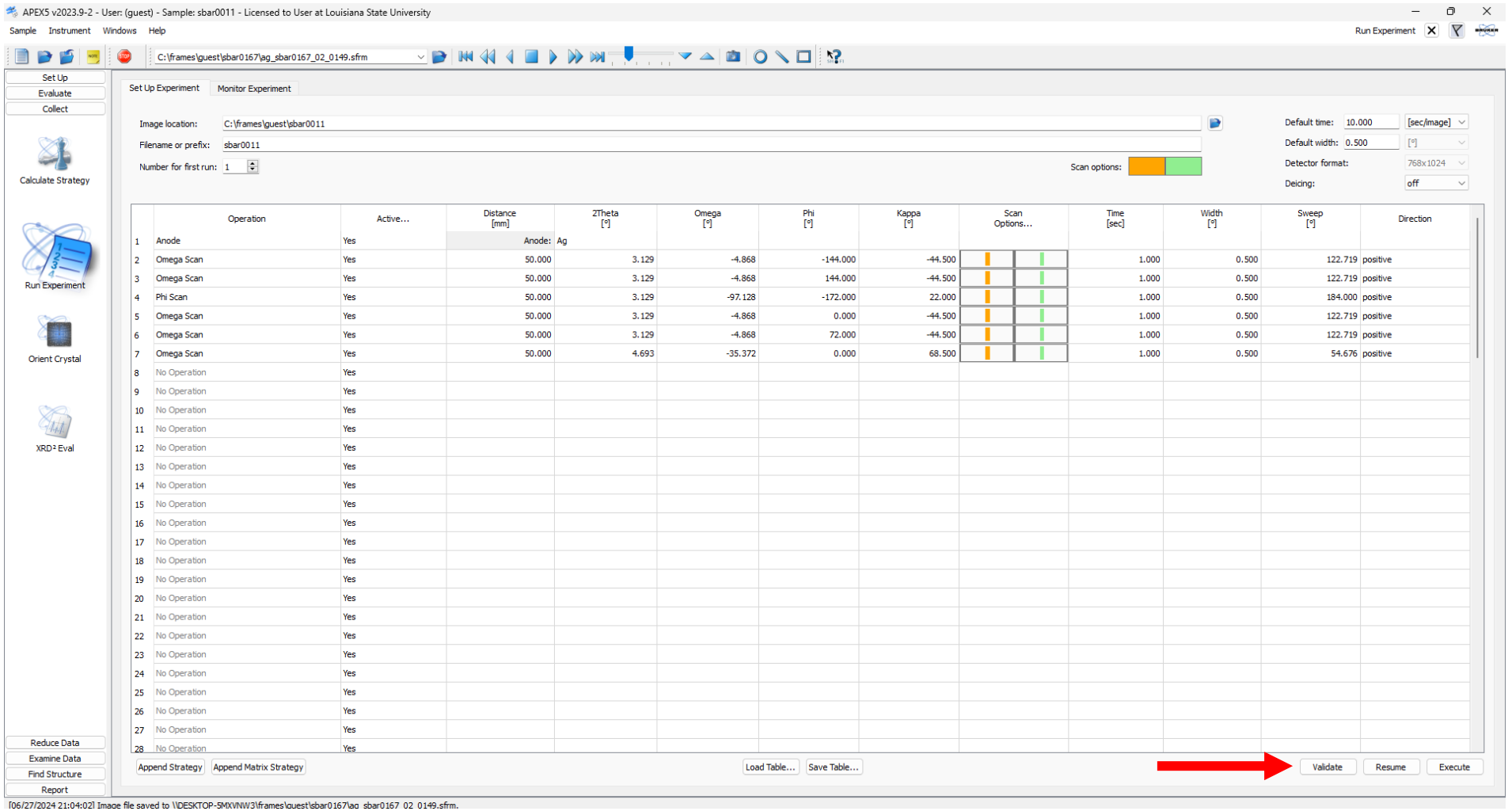
➢ Then click ***"Select Scan Parameters"***.



- Here we can Adjust the **frame time, Scan Options.**
- Then click ***"Ok"***

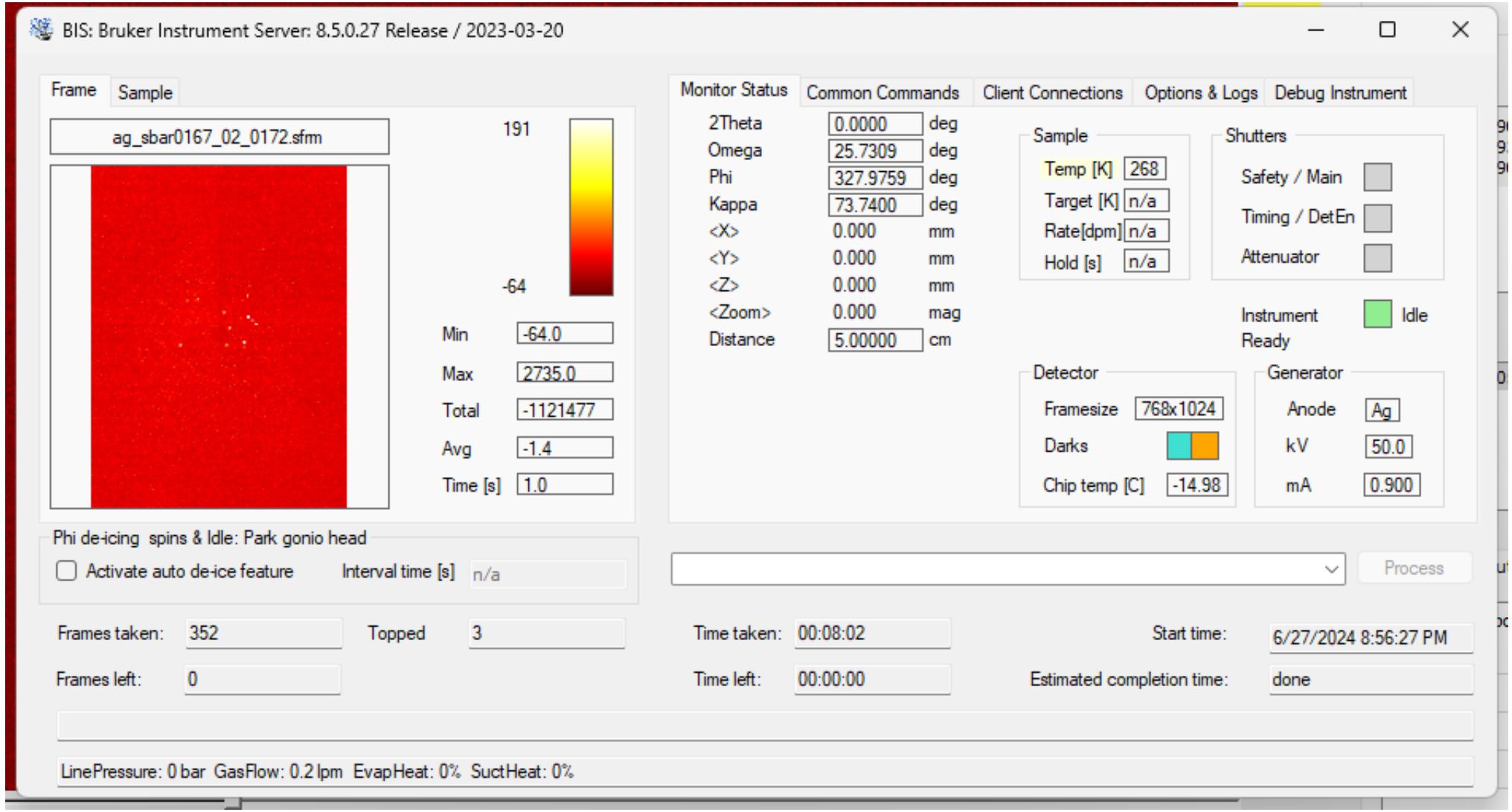


- Next go to **Run Experiment** again and delete the matrix strategy & click ***"Append Strategy"***. After that add more operations **Crystal video**, **Thermostat off** and **Position**.
- Then ***"Validate"*** & ***"Execute"***.

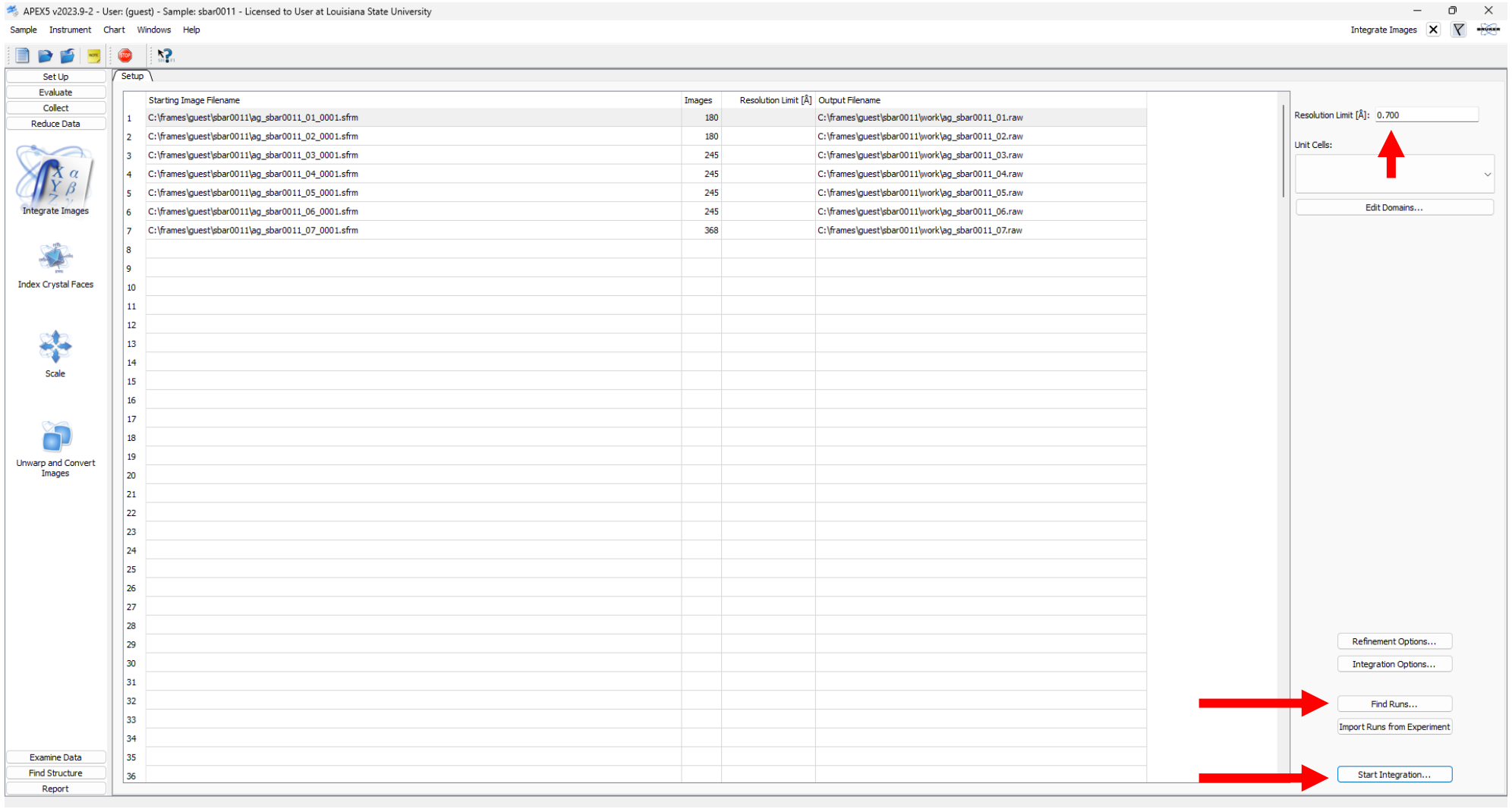


	Operation	Active...	Distance [mm]	2Theta [°]	Omega [°]	Phi [°]	Kappa [°]	Scan Options...	Time [sec]	Width [°]	Sweep [°]	Direction
1	Anode	Yes	Anode:	Ag								
2	Omega Scan	Yes	50.000	3.129	-4.868	-144.000	-44.500		1.000	0.500	122.719	positive
3	Omega Scan	Yes	50.000	3.129	-4.868	144.000	-44.500		1.000	0.500	122.719	positive
4	Phi Scan	Yes	50.000	3.129	-97.128	-172.000	22.000		1.000	0.500	184.000	positive
5	Omega Scan	Yes	50.000	3.129	-4.868	0.000	-44.500		1.000	0.500	122.719	positive
6	Omega Scan	Yes	50.000	3.129	-4.868	72.000	-44.500		1.000	0.500	122.719	positive
7	Omega Scan	Yes	50.000	4.693	-35.372	0.000	68.500		1.000	0.500	54.676	positive
8	No Operation	Yes										
9	No Operation	Yes										
10	No Operation	Yes										
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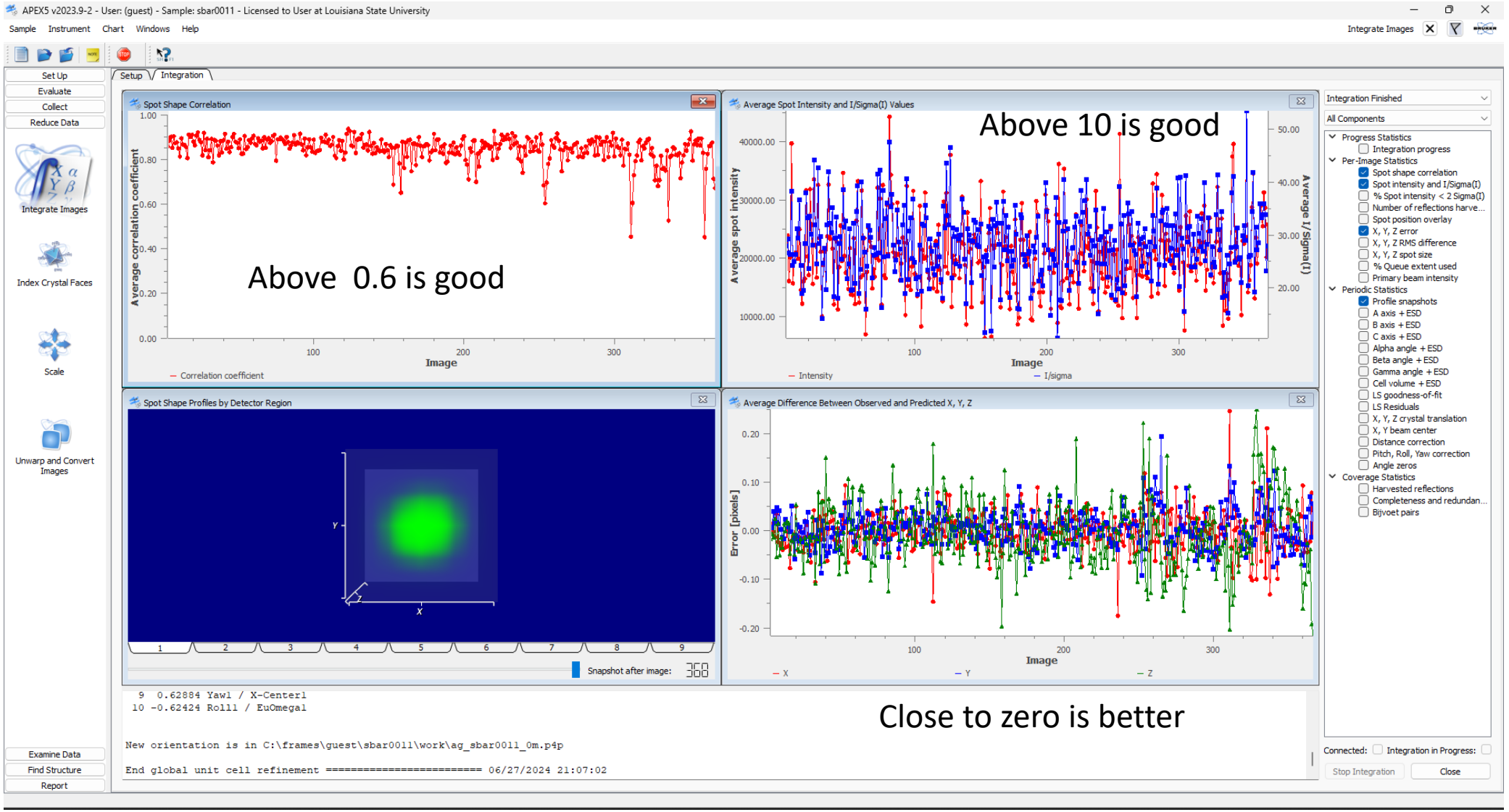
- Check the completion time in **BIS** software & after the data collection is over, we can start the integration.



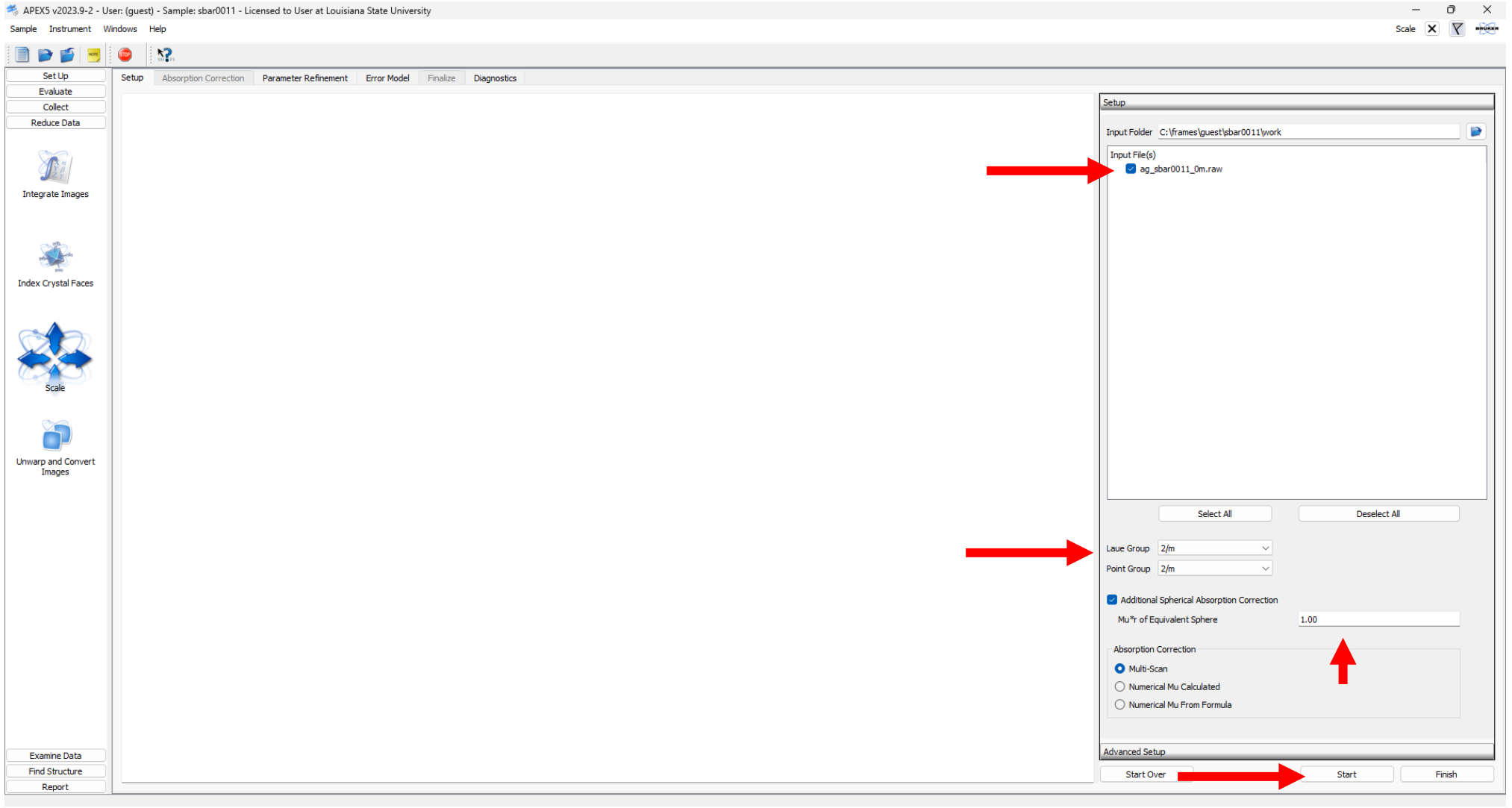
- To integrate data, go to ***"Reduce Data"*** & then ***"Integrate Images"***. Set the resolution to **0.700** and click ***"Find Runs"***. Then select the runs and press ***"OK"***. Finally click ***"Start Integration"***.



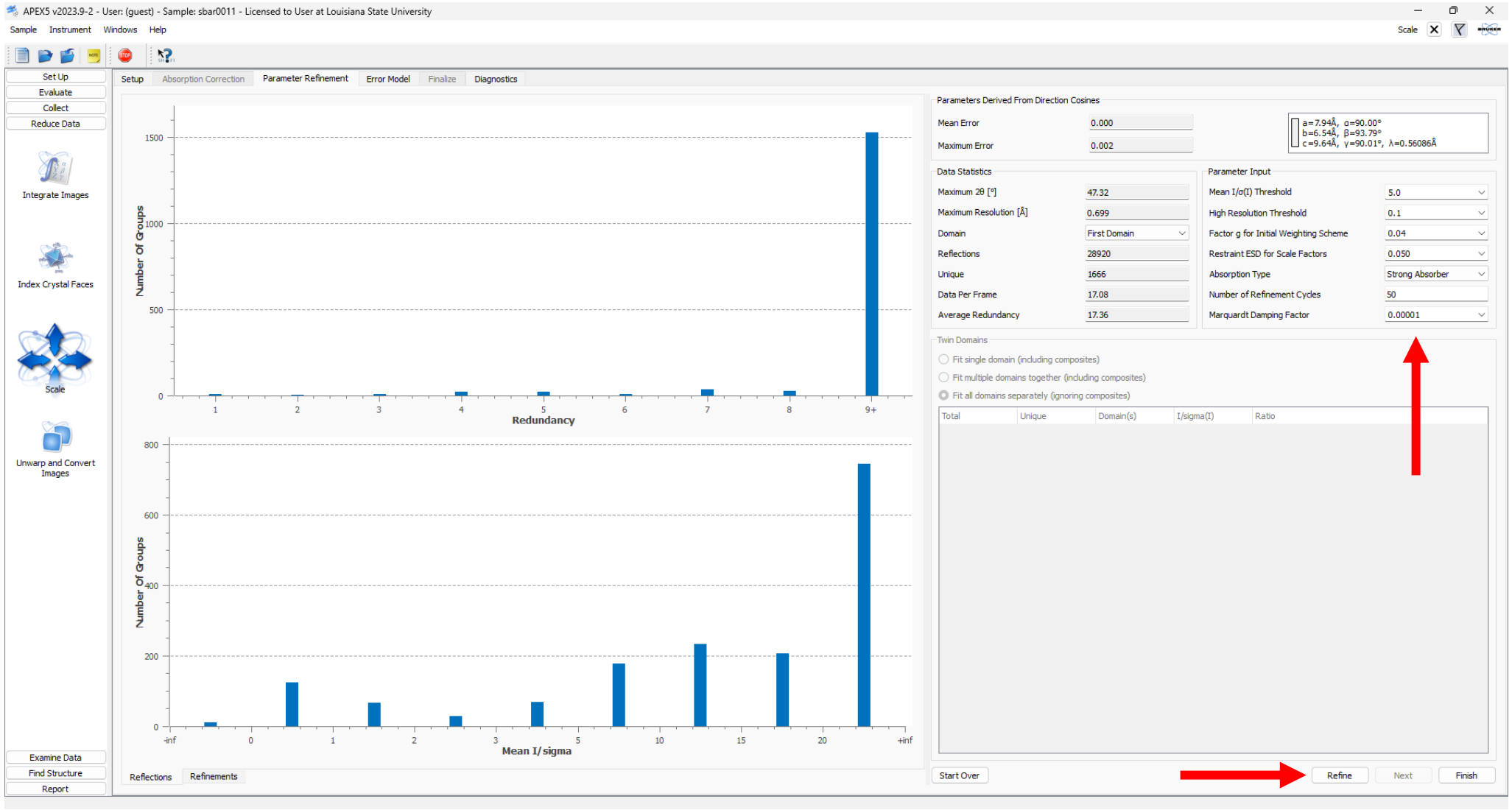
Integration



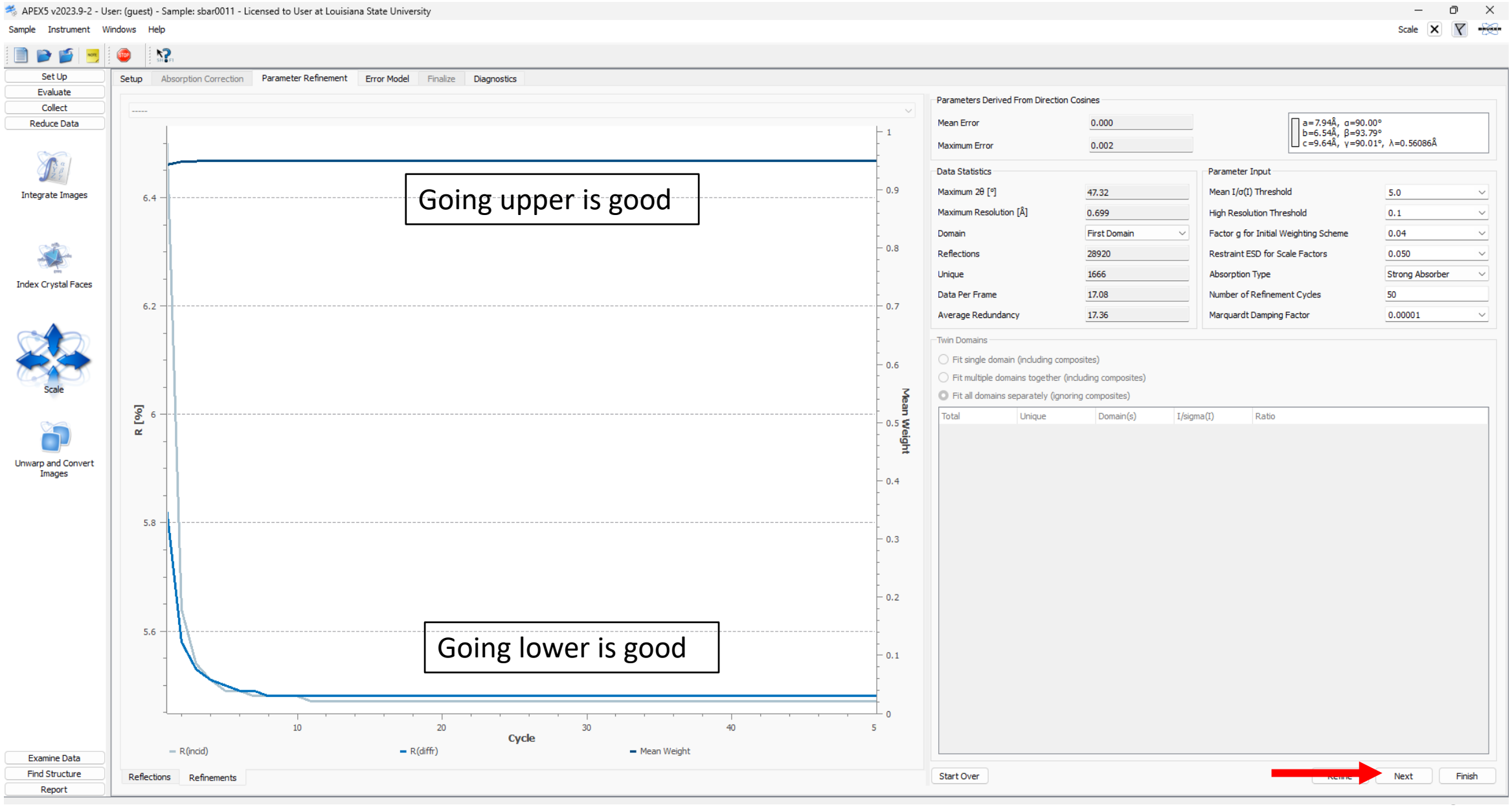
➢To do **Absorption Correction** go to **"Scale"** select the **merged raw file** as the input file & Select the correct **Laue group**, next change the **Mu** value and click ***"Start"***.



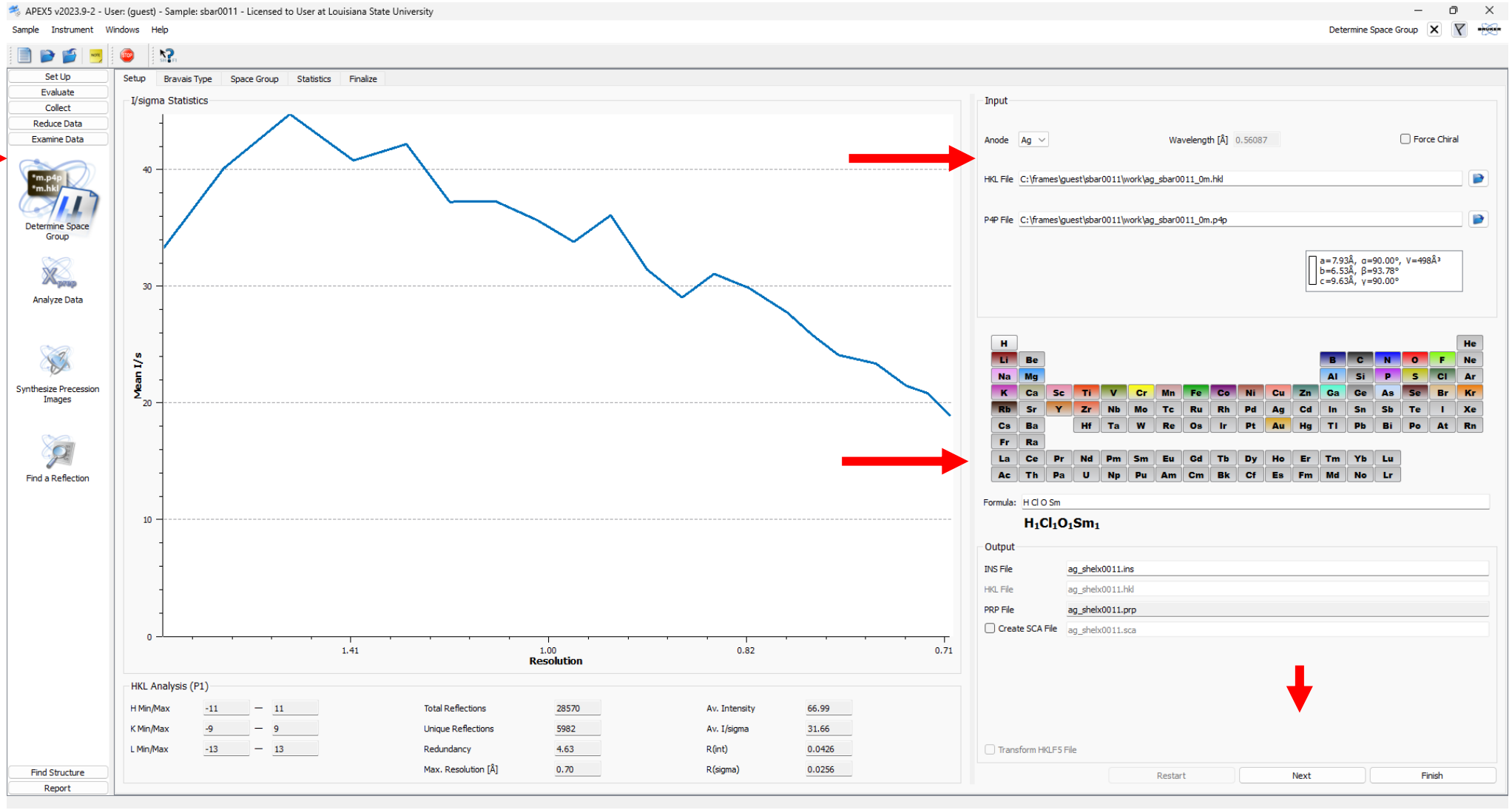
➢Next change the input parameters as in the picture and click ***"Refine"***.



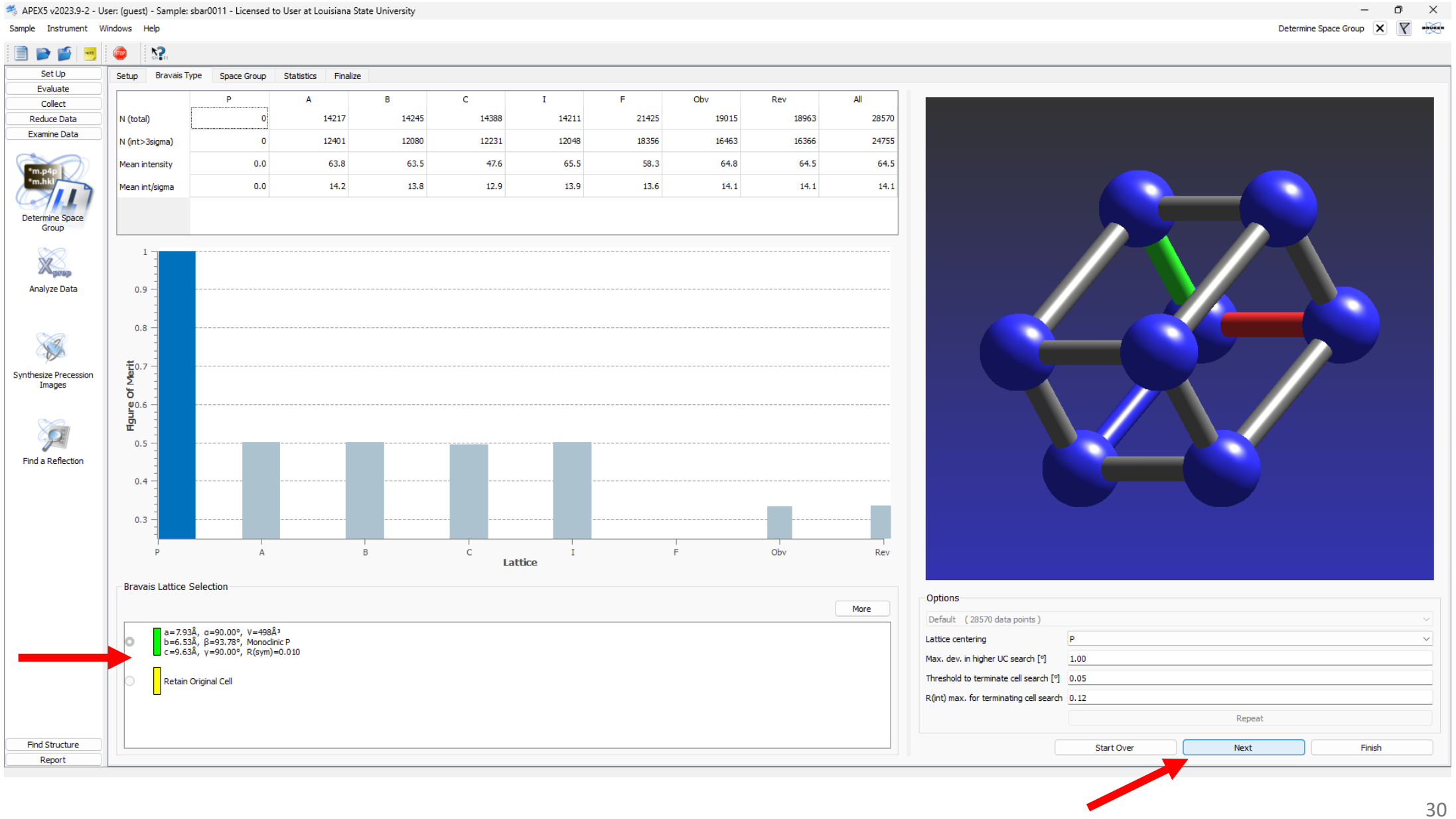
➢ Then click *"Next"* and finally *"Finish"*.



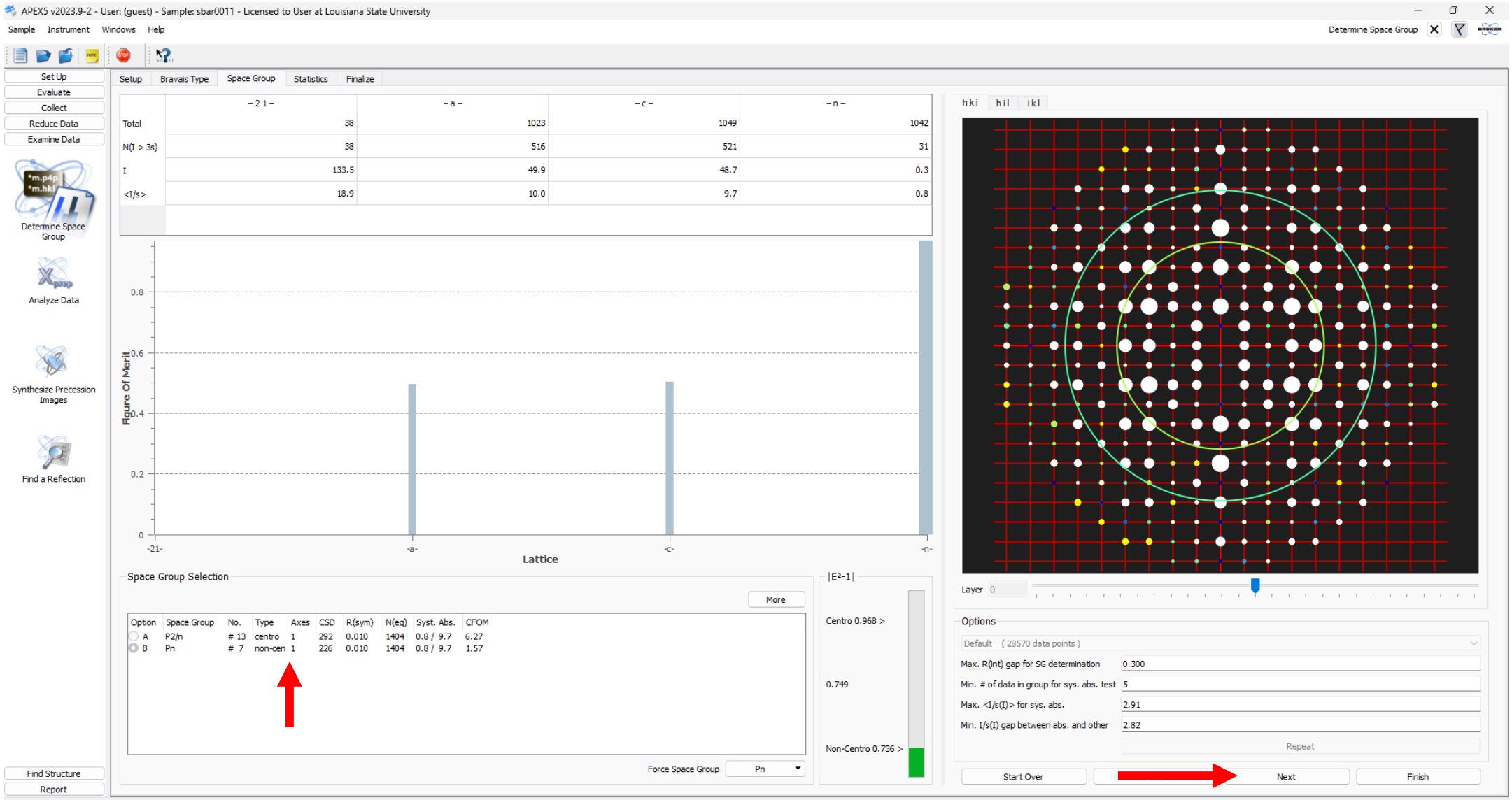
➢To determine the space group, go to ***"Examine Data"*** then go to ***"Determine Space Group".*** Select the correct **HKL File** & **P4P File**, then add the formula & click ***"Next"***.



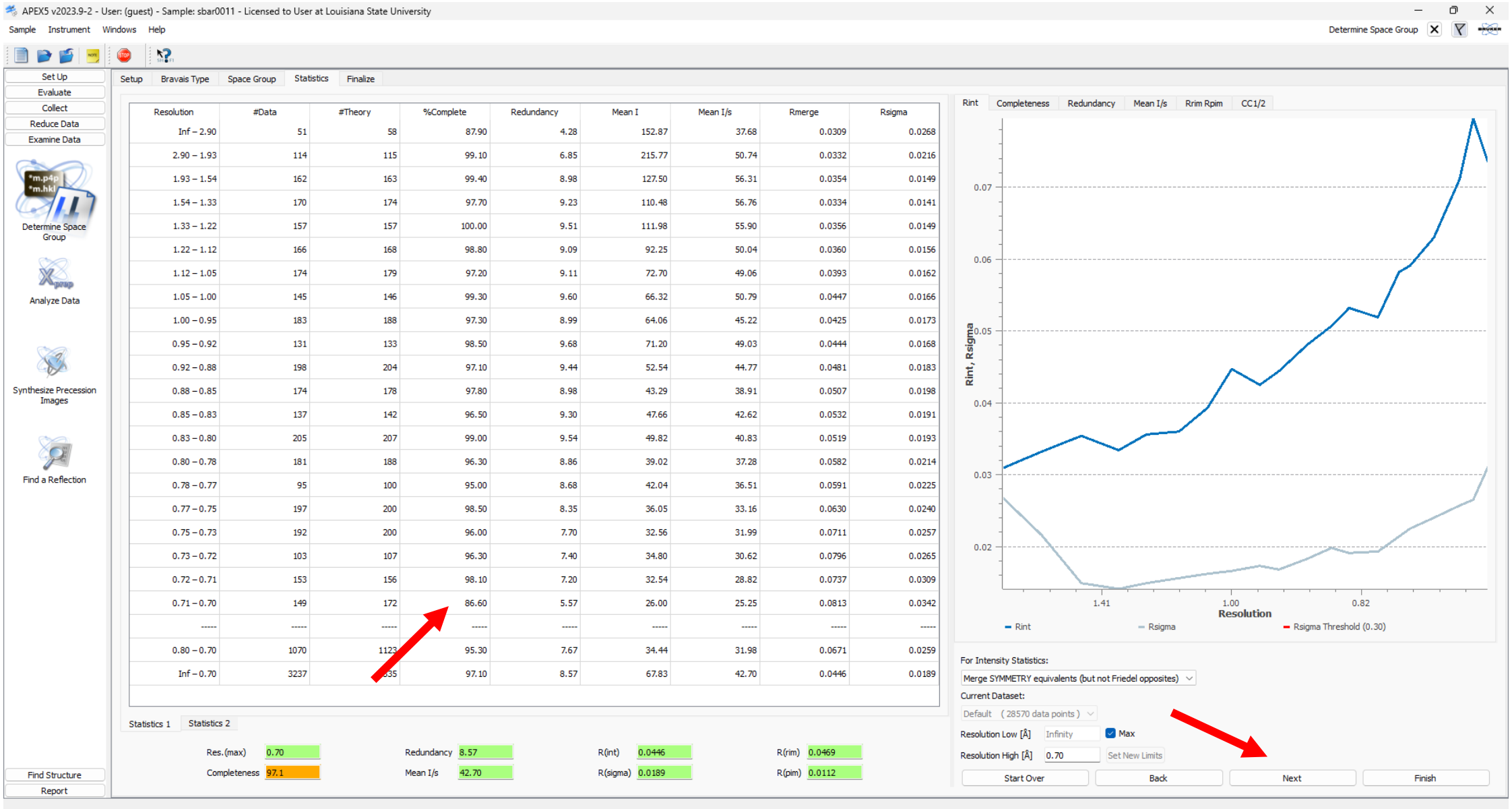
➢Select the **correct unit cell** & click *"Next"*.



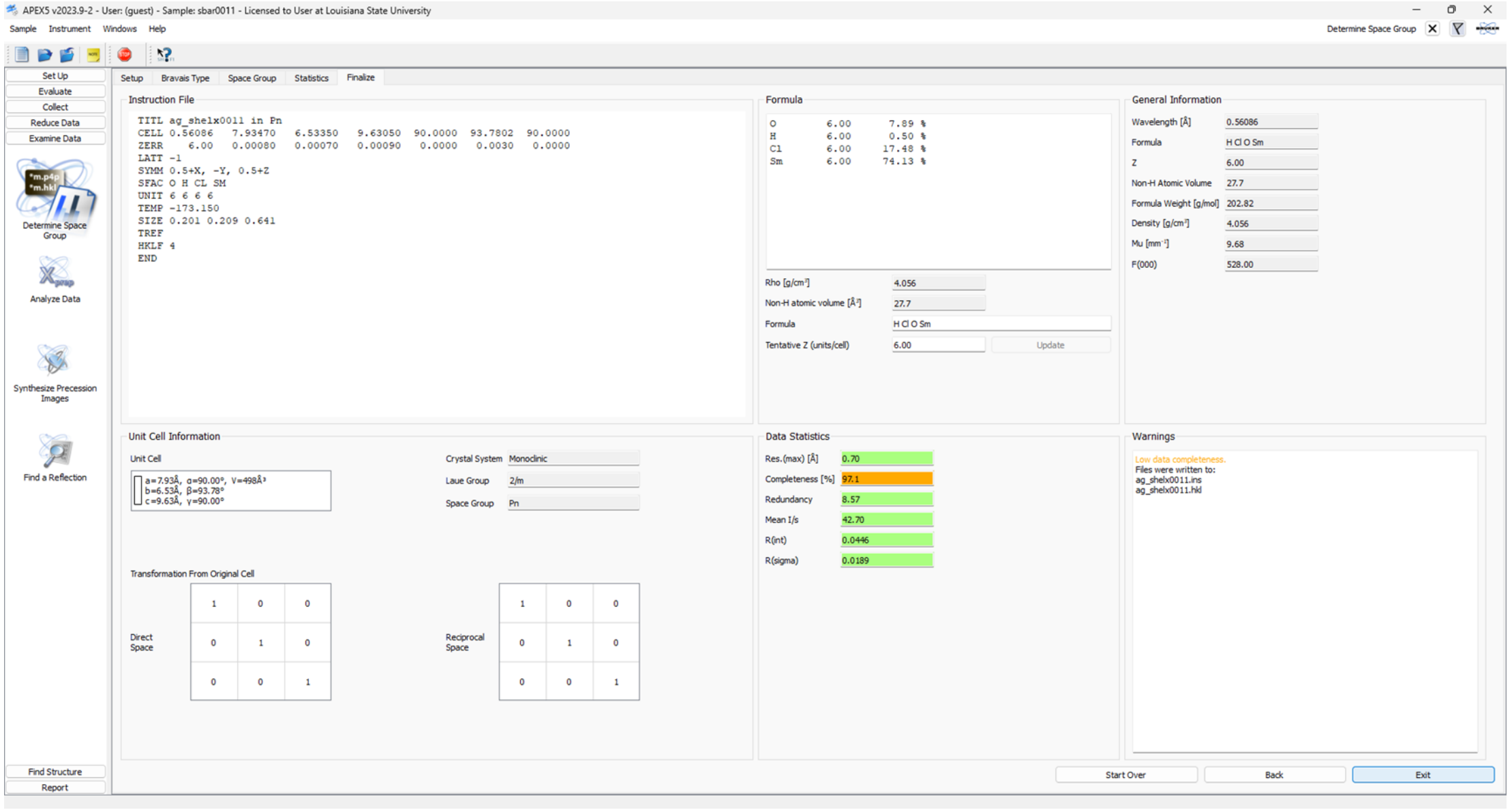
➢Select the most probable **Space Group** & click ***"Next"***.



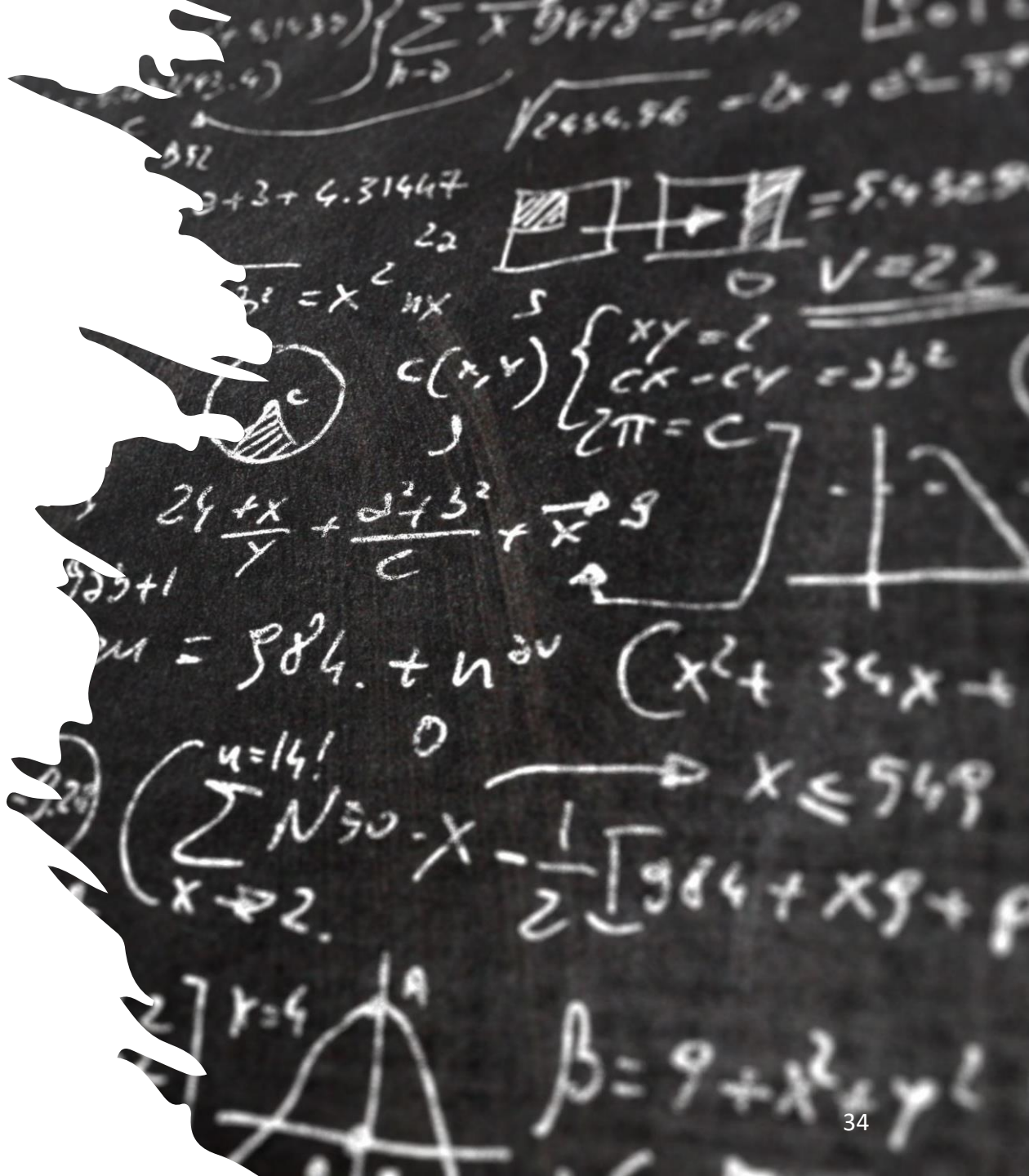
➢Check the **Completeness** of the data & click *"Next"*.

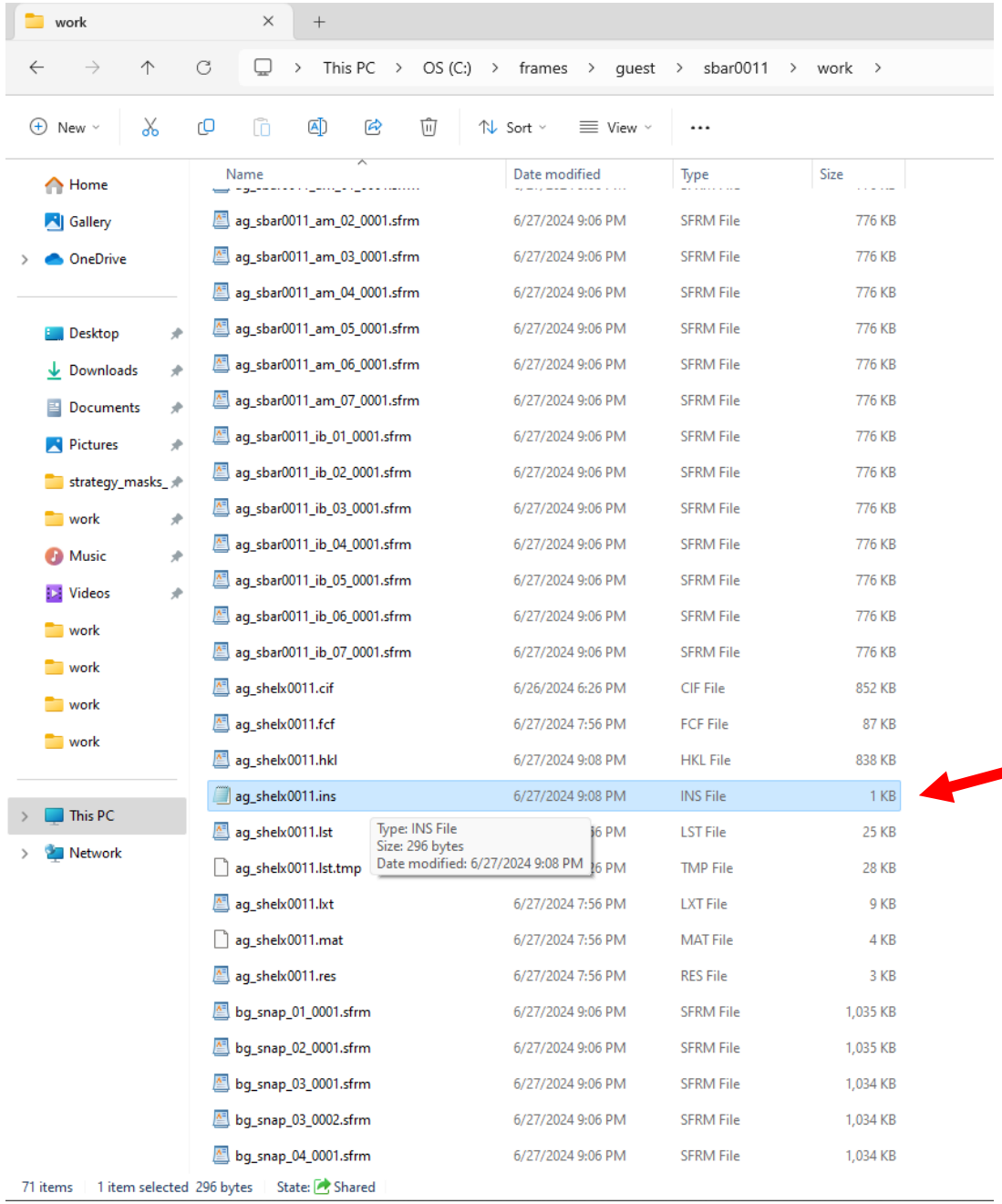


➢Check the **Data Statistics** & click ***"Exit"***.



How to solve the structure

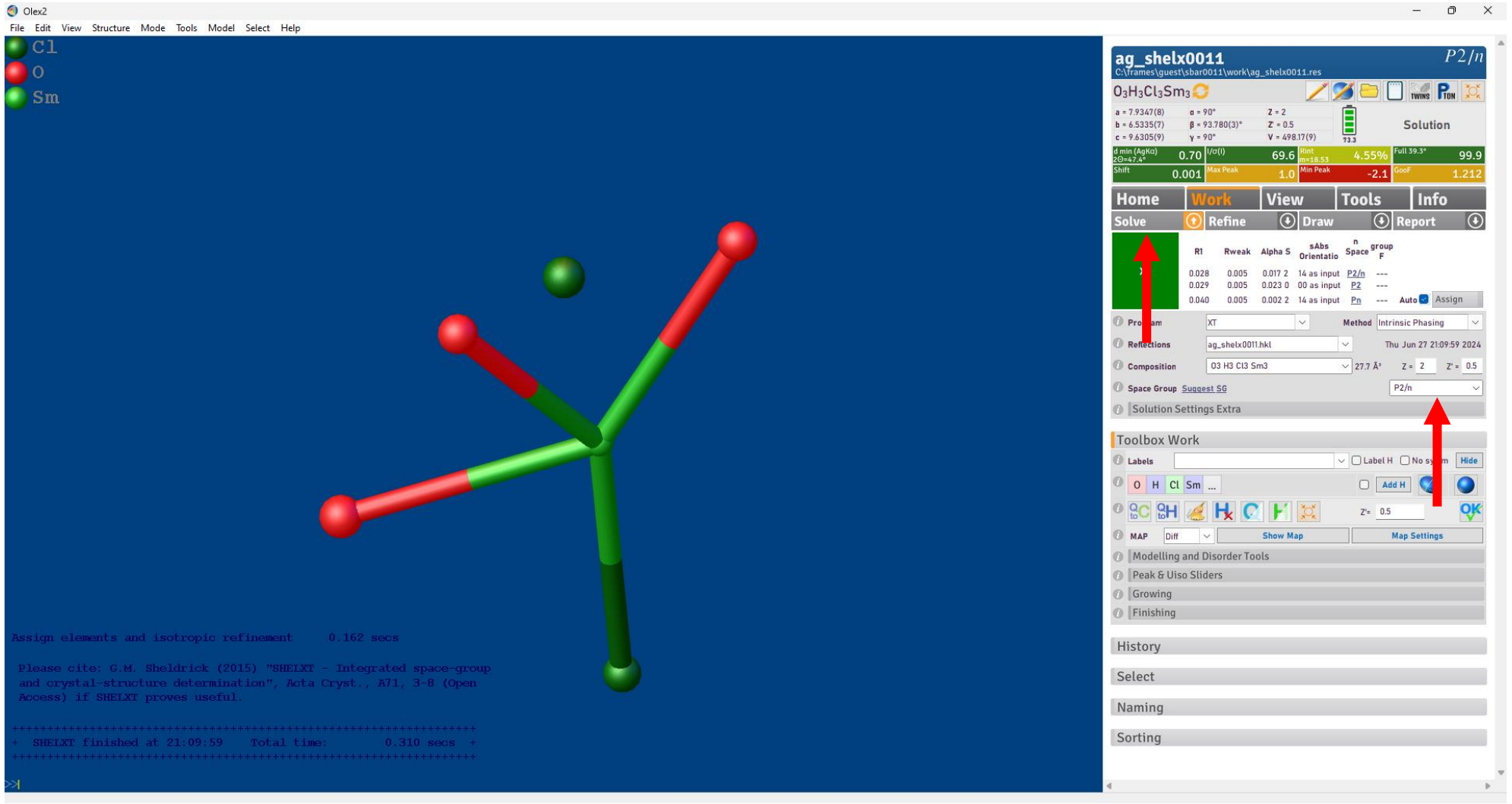




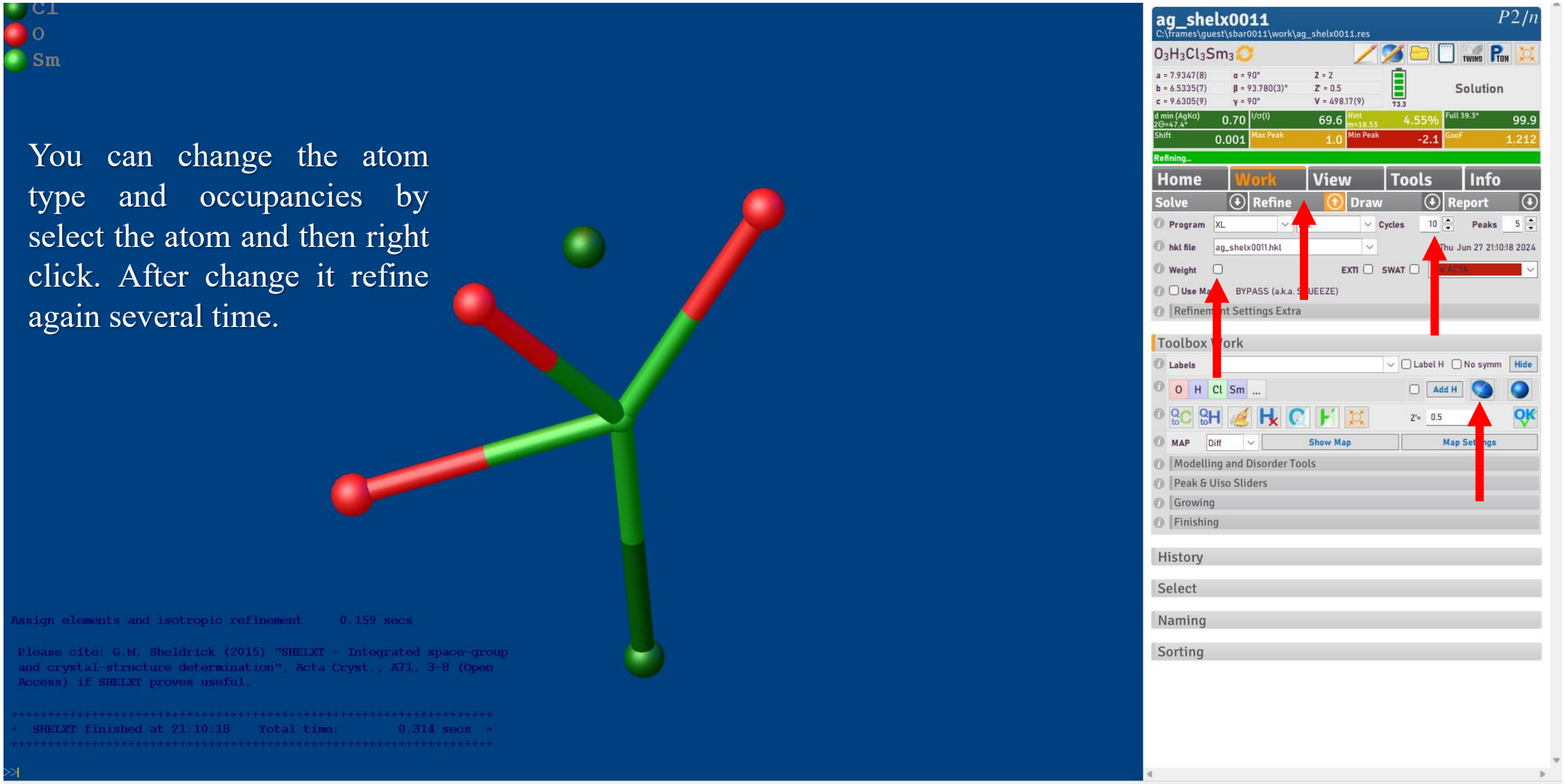
- Data files are in the relevant folder. Go to the folder, copy data and paste it wherever you want. Make sure to have backup.

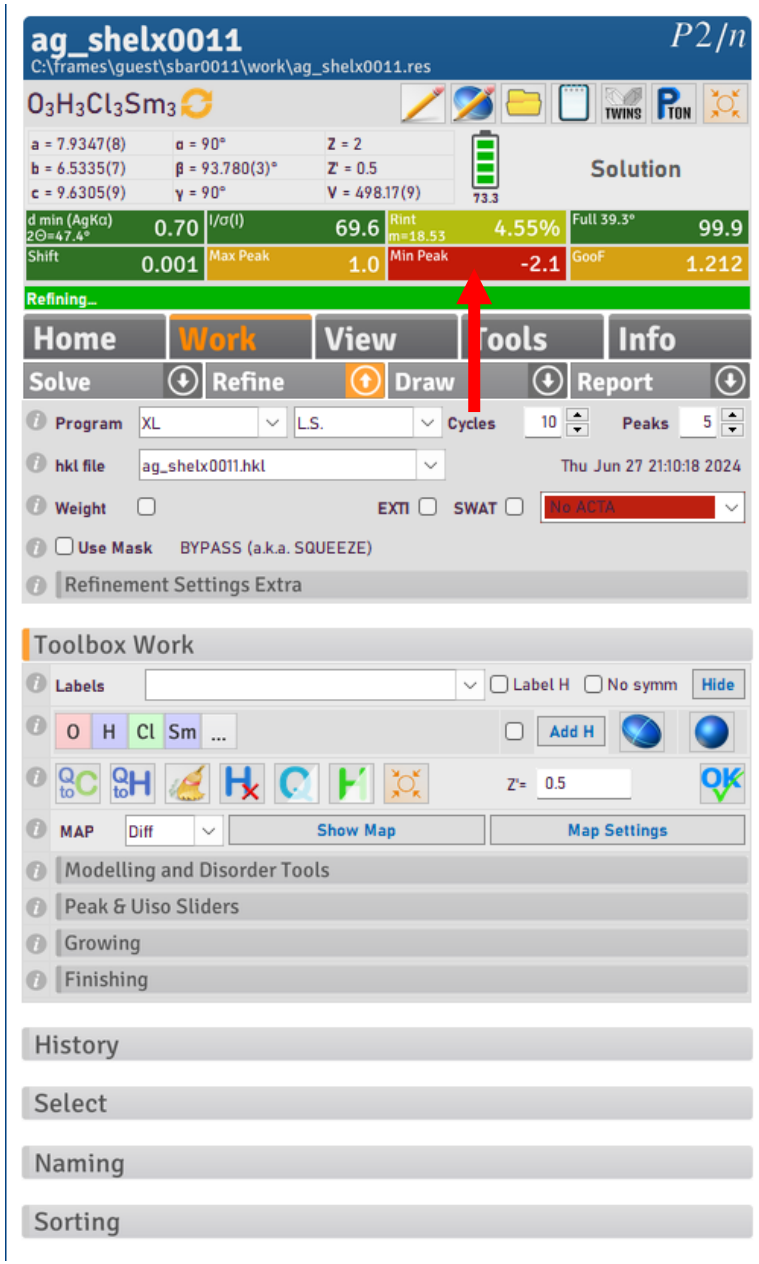
- To solve the structure, open the **INS** File in **Olex2**

- Go to **Solve** then select the correct **Space Group** and click ***"Solve"***.

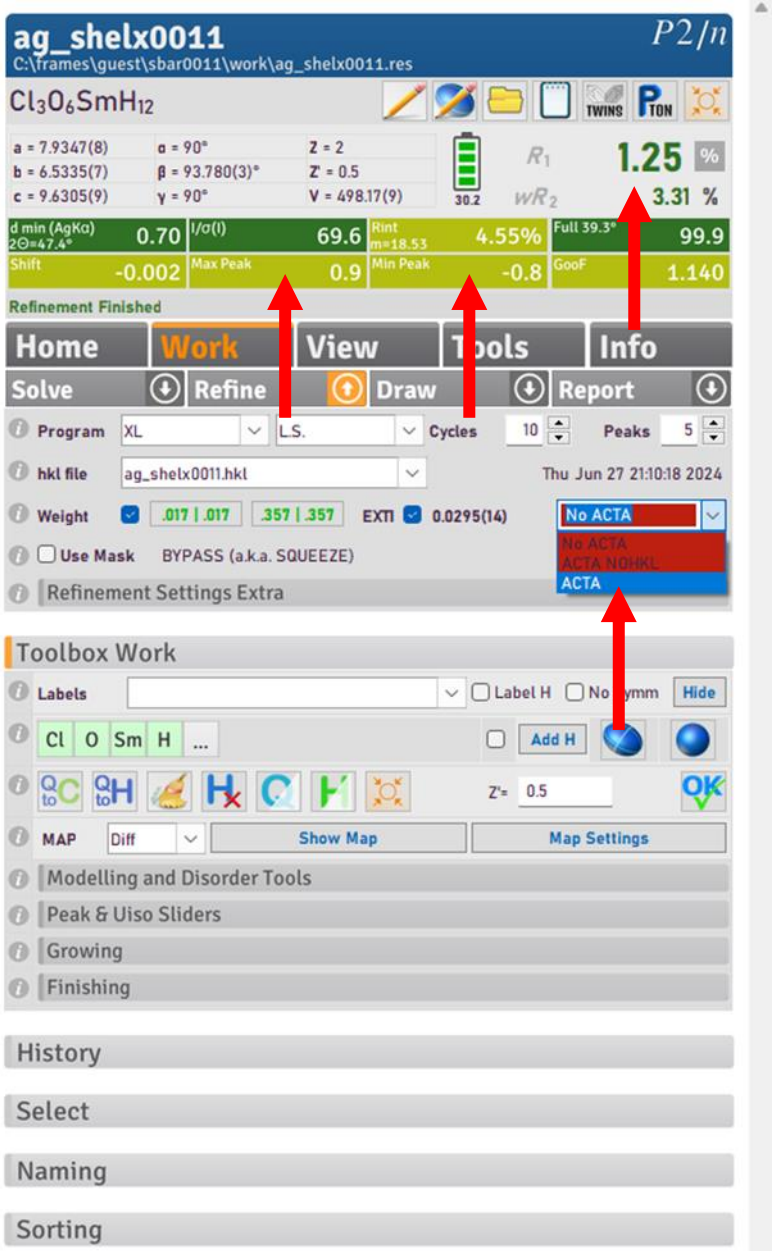


➢Go to **Refine** & click on it. Then add **Weight Factor** and ***"Refine"***, next add **ADP** and ***"Refine"***. Try to solve the structure by changing **Atom types** and **Occupancies**





- During this process ***R1, wR2,*** **max & min peaks** should go down (close to zero is good).
- To create a CIF File, select **ACTA** and ***"Refine"*.**
- **CIF** contains the crystal information, which also can use to visualize structures



- Using **PLATON** we can check the Symmetry, Twin laws, Standard coordinates and etc.

