RESEARCH INSTRUMENTATION STANDARD OPERATING PROCEDURE FOR BRUKER D8 VENTURE SINGLE CRYSTAL X-RAY DIFFRACTOMETER (OPSCXRD-1) AT NUCS FULMER

REVISION 0.0, 7/1/2022

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OPERATING PROCEDURE OPSCXRD-1: OPERATION OF THE BRUKER D8 VENTURE SINGLE CRYSTAL X-RAY DIFFRACTOMETER

1 Background

Single crystal X-ray diffraction is a non-destructive scientific technique that uses X-ray diffraction on a crystalline sample to obtain structural information on chemical compound/material. An instrument dedicated to performing such measurements is called a single crystal X-ray diffractometer. Single crystal X-ray diffraction is a non-destructive analytical technique which provides detailed information about the internal lattice of crystalline substances, including unit cell dimensions, bond-lengths, bond-angles, and details of site-ordering.

X-ray diffractometers consist of three basic elements, an X-ray tube, a sample holder, and an X-ray detector. X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and impact of the electrons with the target material. When electrons have enough energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. These spectra consist of several components, the most common being K_α and K_β. K_α consists, in part, of $K_{\alpha1}$ and $K_{\alpha2}$. $K_{\alpha1}$ has a slightly shorter wavelength and twice the intensity as $K_{\alpha2}$. The specific wavelengths are characteristic of the target material. Filtering, by foils or crystal monochrometers, is required to produce monochromatic X-rays needed for diffraction. $K_{\alpha1}$and $K_{\alpha2}$ are sufficiently close in wavelength such that a weighted average of the two is used. Molybdenum is the most common target material for single-crystal diffraction, with Mo K_α radiation = 0.7107Å. These X-rays are collimated and directed onto the sample. When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, constructive interference occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor.

Single-crystal diffractometers use either 3- or 4-circle goniometers. These circles refer to the four angles (2θ, χ, φ, and Ω) that define the relationship between the crystal lattice, the incident ray and detector. Samples are mounted on thin glass fibers which are attached to brass pins and mounted onto goniometer heads. Adjustment of the X, Y and Z orthogonal directions allows centering of the crystal within the X-ray beam.

X-rays leave the collimator and are directed at the crystal. Rays are either transmitted through the crystal, reflected off the surface, or diffracted by the crystal lattice. A beam stop is located directly opposite the collimator to block transmitted rays and prevent burn-out of the detector. Reflected rays are not picked up by the detector due to the angles involved. Diffracted rays at the correct orientation for the configuration are then collected by the detector. Modern single-crystal diffractometers use CCD (charge-coupled device) technology to transform the X-ray photons into an electrical signal which are then sent to a computer for processing.

Single-crystal X-ray diffraction is most commonly used for the precise determination of a unit cell or a molecular structure, which includes the determination of cell dimensions and the positions of atoms within the lattice. Bond-lengths and angles are directly related to the atomic positions. The crystal structure of a mineral or a chemical compound is a characteristic property that is the basis for understanding many of the properties of each mineral or chemical compound. Specific

applications of single-crystal diffraction include: new mineral or chemical compound identification, crystal solution and refinement, determination of unit cell, bond-lengths, bond-angles and site-ordering, characterization of cation-anion coordination, variations in crystal lattice with chemistry, with specialized chambers, structures of high pressure and/or temperature phases can be determined, and determination of crystal-chemical vs. environmental control on mineral and molecular chemistry.

1.1 Monthly Calibrations/QA/QC Checks

Monthly calibrations are undertaken to verify the functionality of the instrument and to head off any problems or safety concerns before they become larger problems that could result in instrument failure. A calibration standard is used in the monthly calibration (see Section 3).

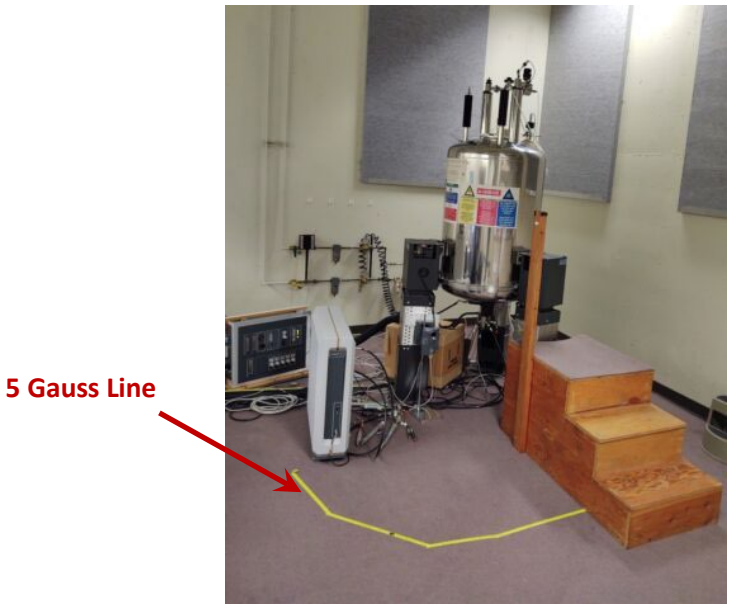
2 Safety Requirements

Single crystal X-ray diffraction is a scientific technique that utilizes X-rays to obtain structural information on crystalline samples. X-rays are ionizing radiation and X-ray photons carry enough energy to ionize atoms and disrupt molecular bonds, which makes X-rays harmful to living tissue. A very high radiation dose over a short period of time will cause radiation sickness, while lower doses will give an increased risk of radiation-induced cancer. The Bruker D8 Venture contains radiation protective interlocks (the main door needs to be closed for operation) to prevent a user from being exposed to the X-ray radiation during the use of the single crystal X-ray diffractometer. The doors to the instrument are locked when voltage is applied to the X-ray tube. The yellow lights on the side of the X-ray diffractometer and the red lights (highlighted in red) on the X-ray tube are illuminated when voltage is applied to the X-ray tube, X-rays are being generated, and the shutter is open.

All users are required to provide the NUCS Core Facility Staff with records of completion of the WSU Radiation Safety Office Training Courses #1-7 & 10 (https://rso.wsu.edu/wsu-radiation-

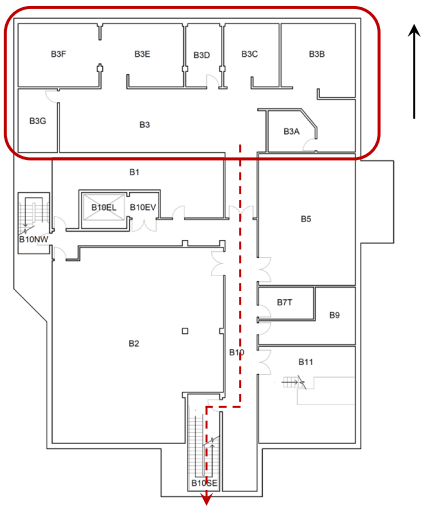
safety-training/), *prior* to being trained on the use of the Bruker D8 Venture Single Crystal X-ray Diffractometer.

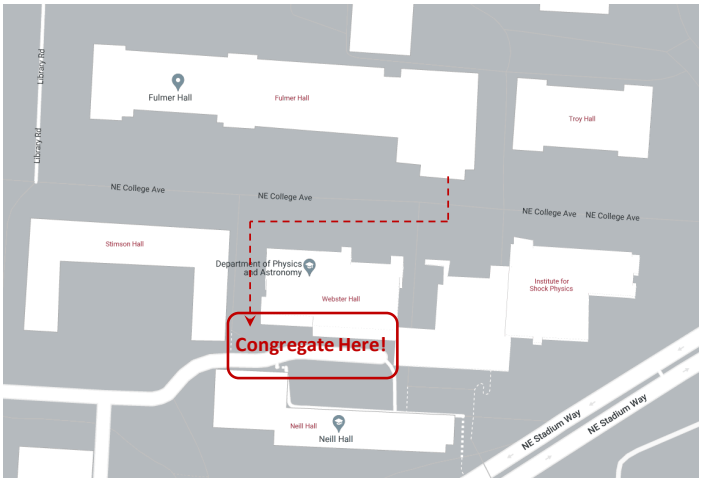
The Bruker D8 Venture Single Crystal X-ray Diffractometer is located in the Nuclear and Chemical Science (NUCS) Core Facility, which contains strong magnets for nuclear magnetic resonance (NMR) spectroscopy. Electronic, electrical, or mechanical medical implants may be affected or even stopped in the presence of a static or changing magnetic field. For your own safety, if you have a pacemaker or other medical implant that could be adversely affected by strong magnetic fields, do NOT enter the NUCS Core Facility. Magnets can exert large attractive forces on equipment or other ferromagnetic objects when brought. Please do not approach the magnets beyond the 5-gauss line (outlined with yellow tape) or within 10 feet of the magnets.



All of the NMR spectrometer magnets are superconducting, which means they are kept in a cryostat filled with liquid helium. A concentric dewar of liquid nitrogen is placed around the helium cryostat in order to keep the helium boil-off rate low. Additionally, liquid nitrogen may be kept in portable dewars around the facility. Cryogens can pose several risks including: asphyxiation, frostbite, and chemical explosions. In the event of a magnet quench (see picture below), the superconducting wire inside the instrument transitions to a normal conducting state. This would boil off all of the liquid helium very quickly. The rapid expansion of helium as it vaporizes can displace the oxygen in the NUCS Core Facility and cause asphyxiation. The facility contains an oxygen meter, which causes an audible alarm if the oxygen concentration drops below 20%. If you observe a sudden exhaust of gas from a magnet (and NUCS staff are not performing a cryogen fill) or hear an audible alarm due to low oxygen levels, exit the NUCS Core Facility immediately following the same procedure as the occurrence of a fire alarm.

In the event of an audible alarm, due to a fire alarm or an air alarm from low oxygen levels, or an instrument quench, please exit the facility through the main door to the NUCS Fulmer Core Facility and go up the stairs at south end of the building (see left picture below). The building exit at the south stairs will lead to College Avenue. Please congregate behind Webster until the fire alarm has ended (see right picture below).





The same principles of research safety apply in instrumentation laboratories when you are handling samples. Research samples, glassware, chemical storage, spills, and waste disposal

must be properly handled. You must wear long pants (or equivalent) and closed-toed shoes. No food or beverages are allowed in the NUCS Core Facility. The NUCS Core Facility is not a wet lab. All sample prep should be done in your lab. Do not prep samples at spectrometers. Do not bring your lab coat or gloves into the NUCS Core Facility. Keeping a shared lab clean requires the cooperation of everyone. Please do not leave KimWipes, paper towels, etc. laying around. If you believe any sample may have spilled into or onto one of the instruments, please notify the NUCS Core Facility staff immediately. Place a written note on the keyboard to inform the next user.

Some users are approved to analyze radioactive samples in the NUCS Fulmer Core Facility. If an instrument is blocked off with a barrier (see below picture) for the analysis of a radioactive sample, do not cross this barrier and enter the instrument bay until the barrier has been removed (indicating that the instrument bay is free of radioactive material).

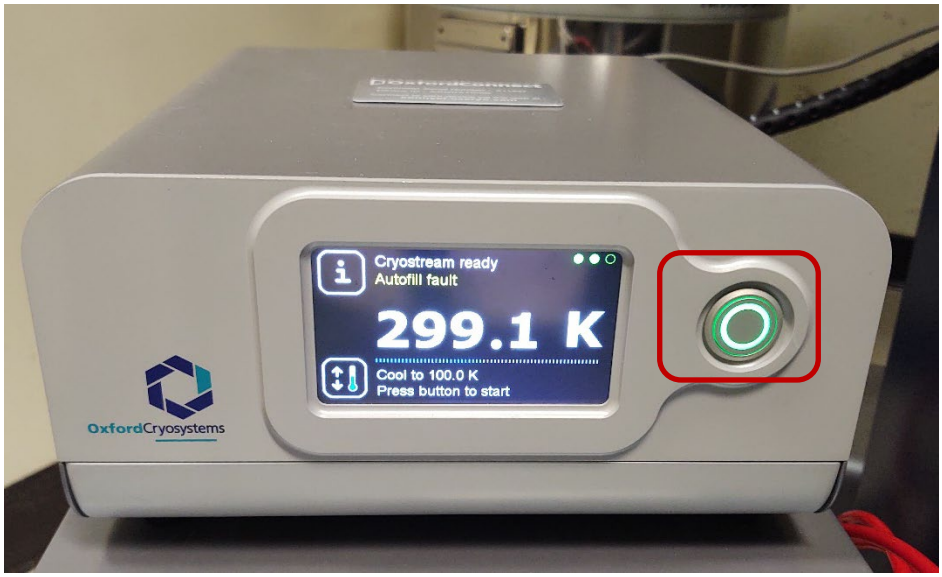
3 Calibration Procedure

Materials: Sucrose sample mounted on a cryoloop, Bruker D8 Venture Single Crystal X-ray Diffractometer, Oxford Cryostream

3.1 Before starting a calibration, reserve at least two hours of instrument time on iLab (see Steps 6.1 and 6.2).

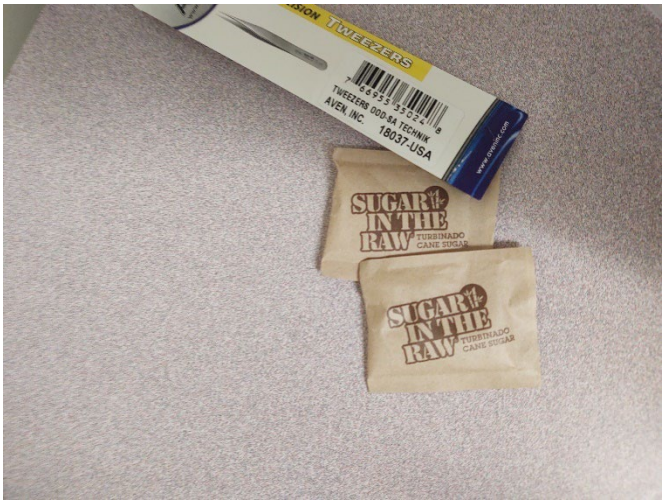
3.2 Check the amount of nitrogen in the liquid nitrogen dewar, it will need to be at least 25%. The liquid nitrogen level in the picture below is 15.1%. If the level is below 25%, it will need to be filled to at least 25%. Follow Steps 7.7 to 7.14 to add liquid nitrogen to the dewar, if needed.

3.3 Start to cool the instrument to 100K by pressing the green button (highlighted in red) on the front of the cryostream controller (see Steps 7.15 – 7.20 for more information). It will take about 15 minutes to cool to 100K.

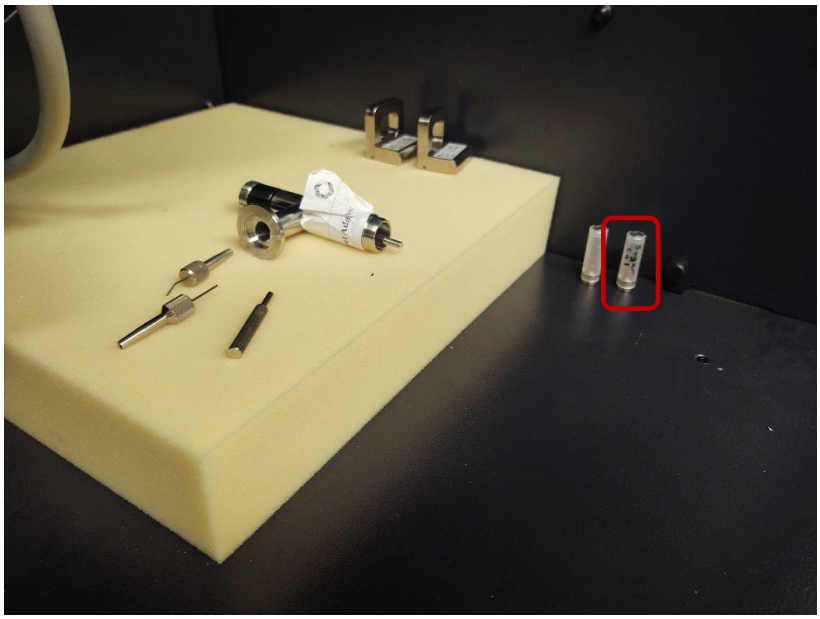


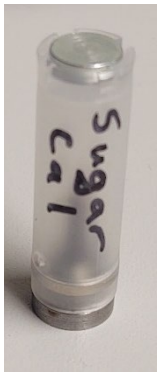
3.4 Open the doors to the diffractometer as indicated in Step 6.13.

3.5 The calibration sample is obtained from sugar crystals in the Raw Sugar Packets.



3.6 Find the sucrose calibration sample. It can be found inside the instrument on the right side (highlighted in red). It is labeled Sugar Cal (see picture on right).

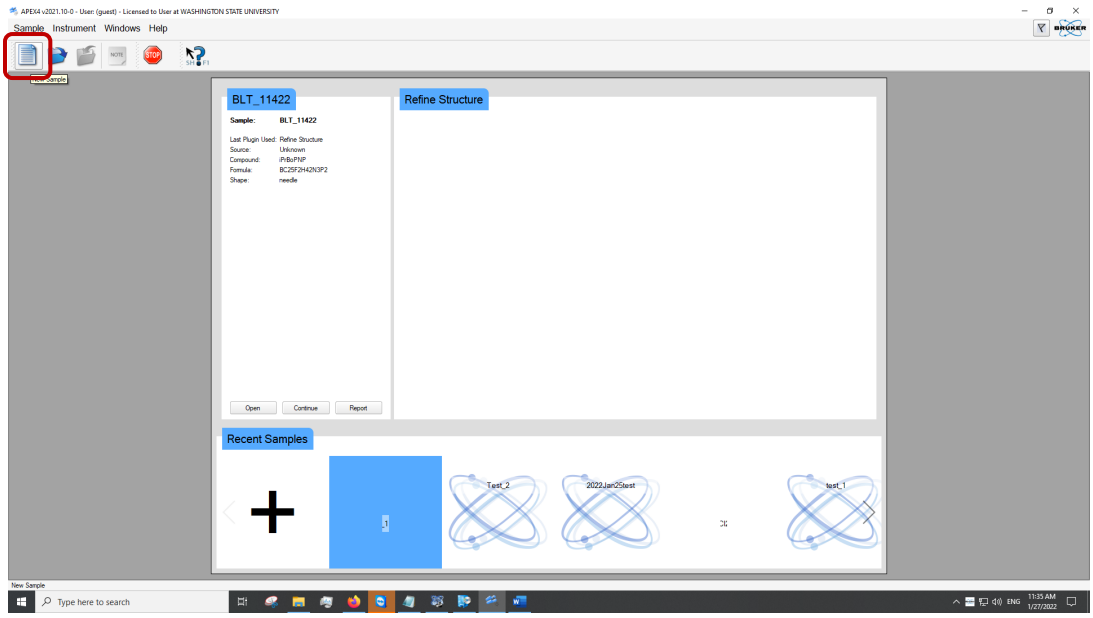




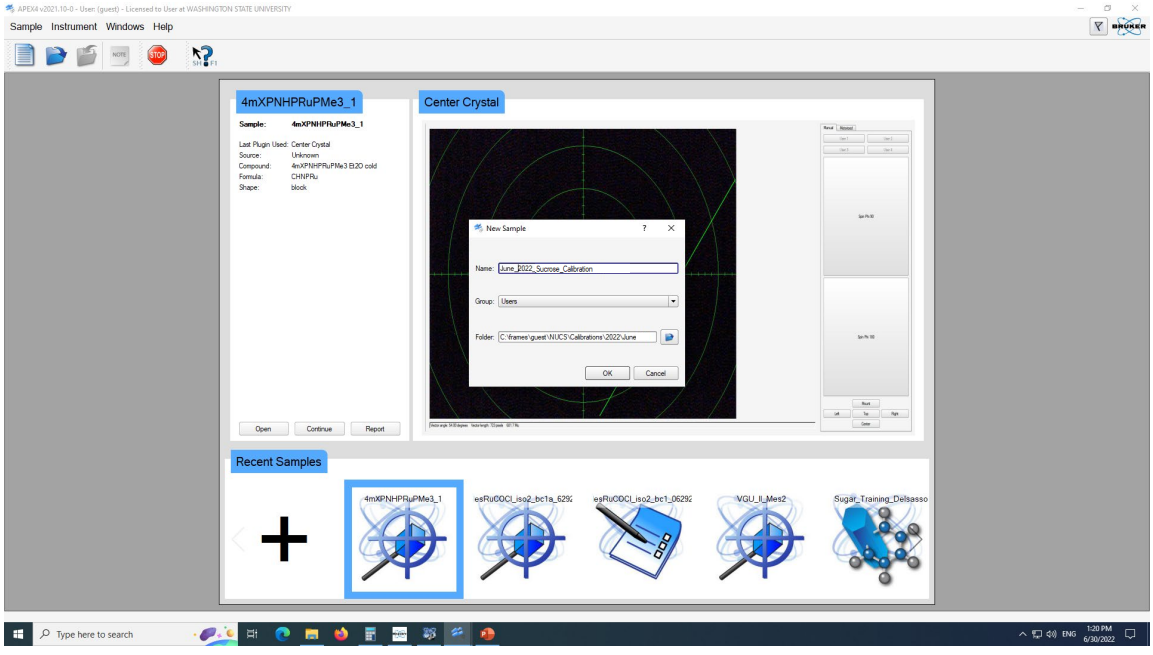
3.7 Carefully remove the cover by lifting it straight up. If the covers rubs against the tip of the goniometer head it will remove the sucrose sample from the tip.

3.8 Place the place the sucrose sample (labeled Sugar Cal) in the sample holder indicated in step 6.20 of the operating procedure.

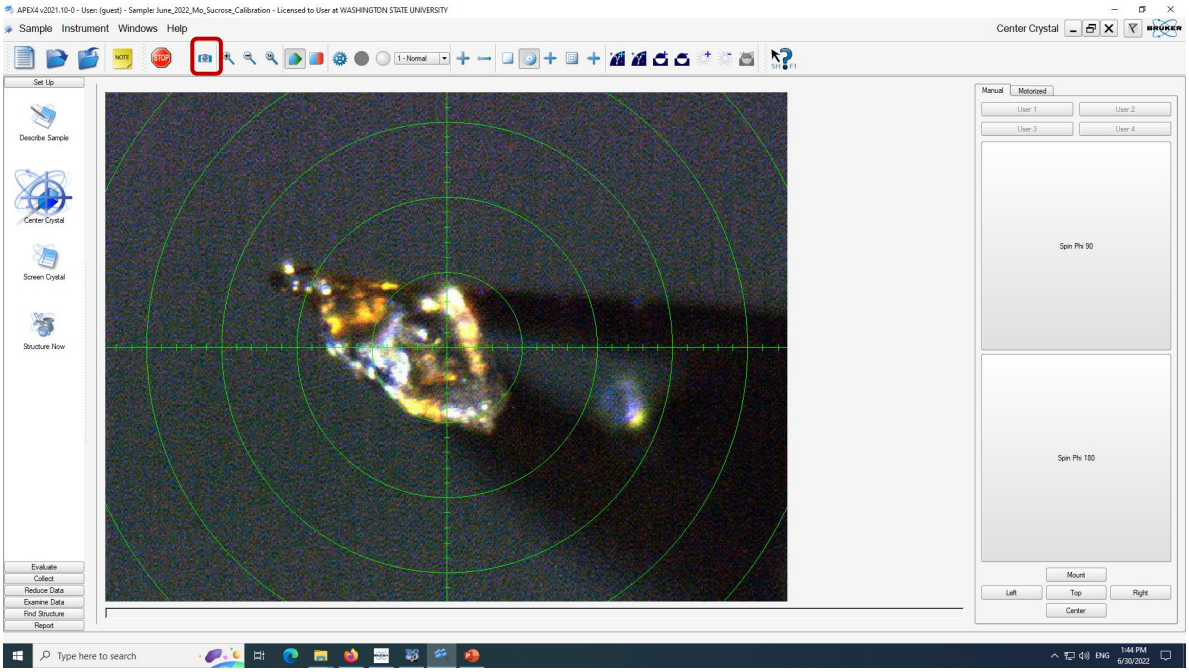
3.9 To get started on the data collection, click on the New Sample Icon (highlighted in red).



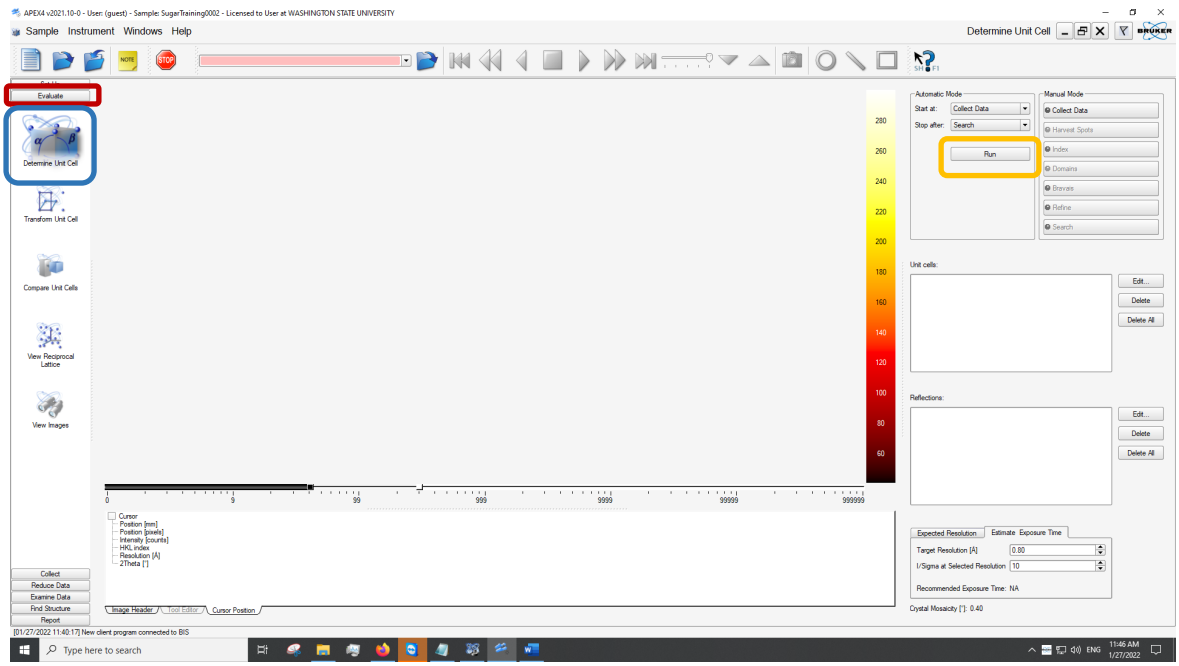
3.10 Fill in the information on the New Sample, The Name should be Month Year Sucrose Calibration (e.g. June_2022_Sucrose_Calibration) and the Folder should be: C:\frames\guest\NUCS\Calibrations\YYYY\Month (e.g. C:\frames\guest\NUCS\Calibrations\2022\June for the calibration data set collected in June of 2022). Note the folder will need to be created before it can be selected.



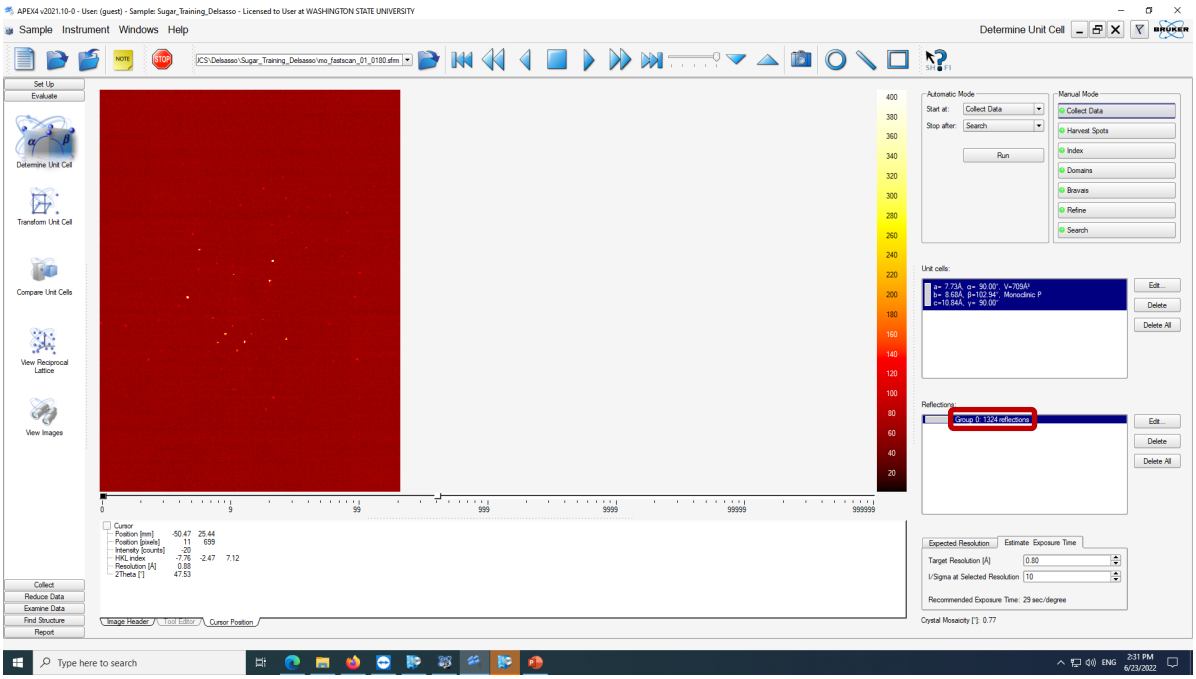
3.11 Follow Steps 6.17 to 6.26 to center the crystal in the goniometer. Before moving on, take a snapshot of the crystal by clicking on the camera icon (highlighted in red) and save the picture as: Month Year Sucrose Calibration.jpg.



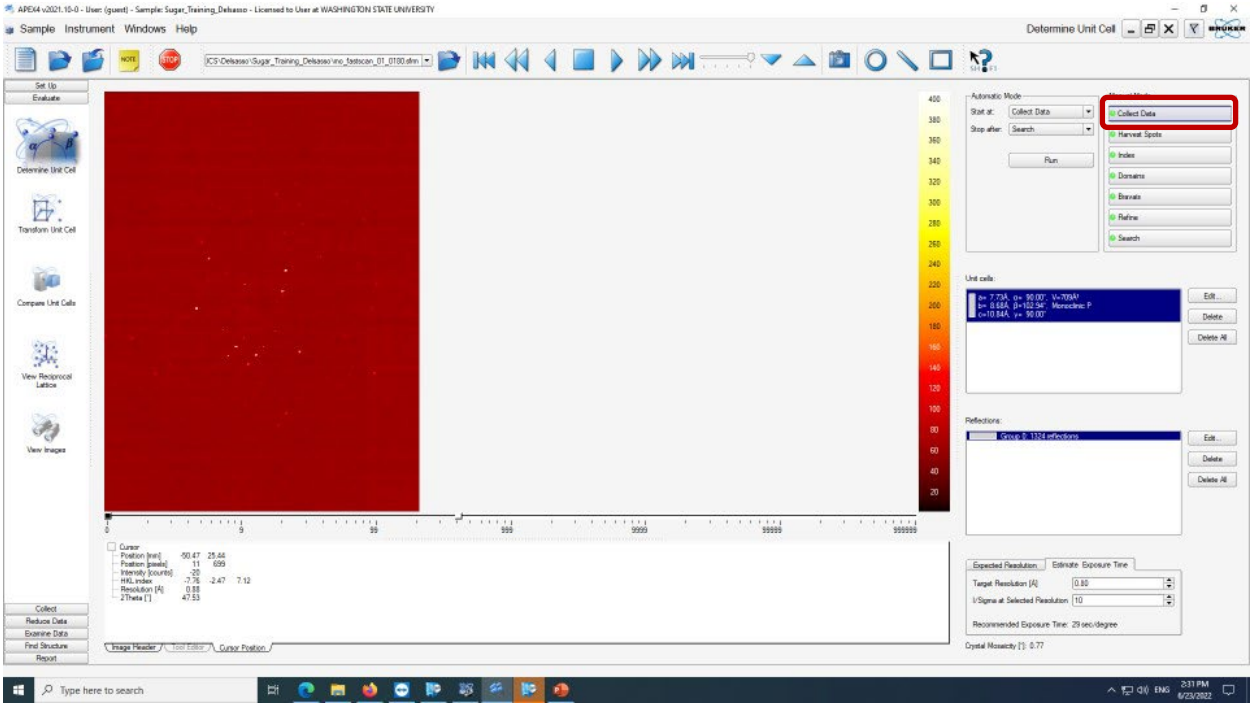
3.12 Start by doing an automatic unit cell collection, by clicking on the Evaluate button (highlighted in red), followed by the Determine Unit Cell icon (highlighted in blue). This will bring up the data collection option. To collect a unit cell using the molybdenum X-ray tube, click on the Run button in automatic mode (highlighted in orange).



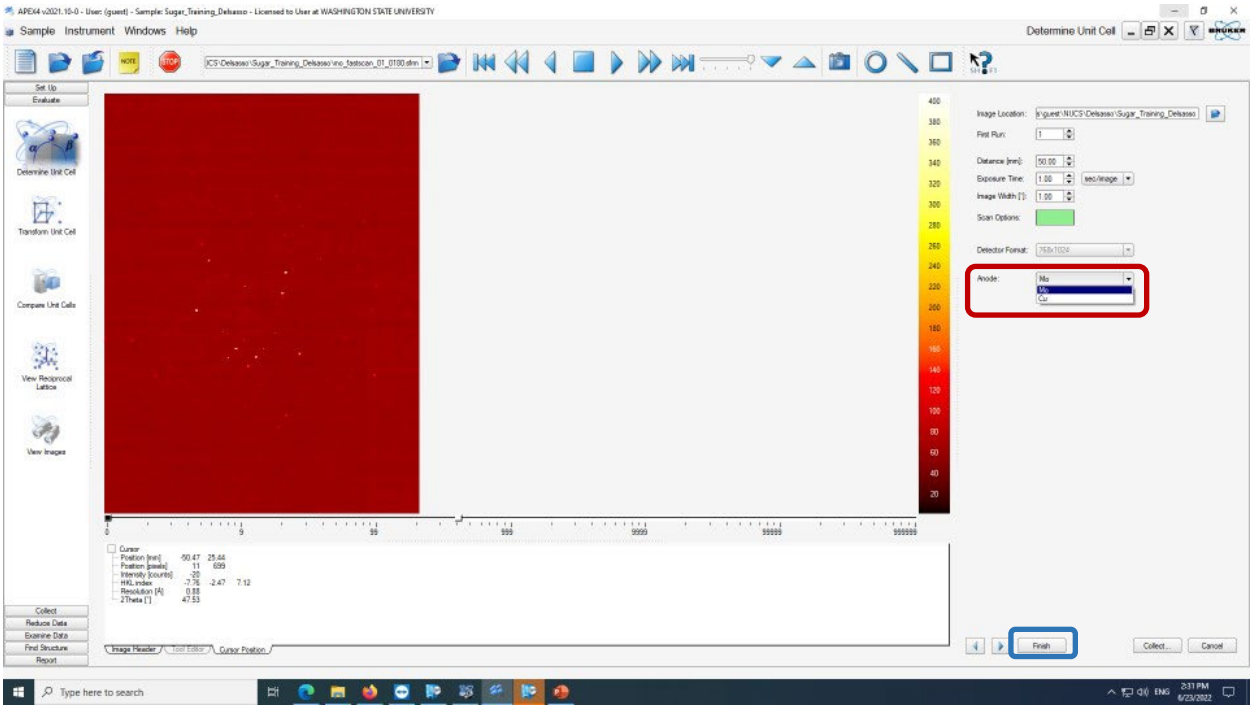
3.13 After the unit cell data collection has completed, write down the number of reflections for the unit cell data collection (highlighted in red) on the calibrations form.



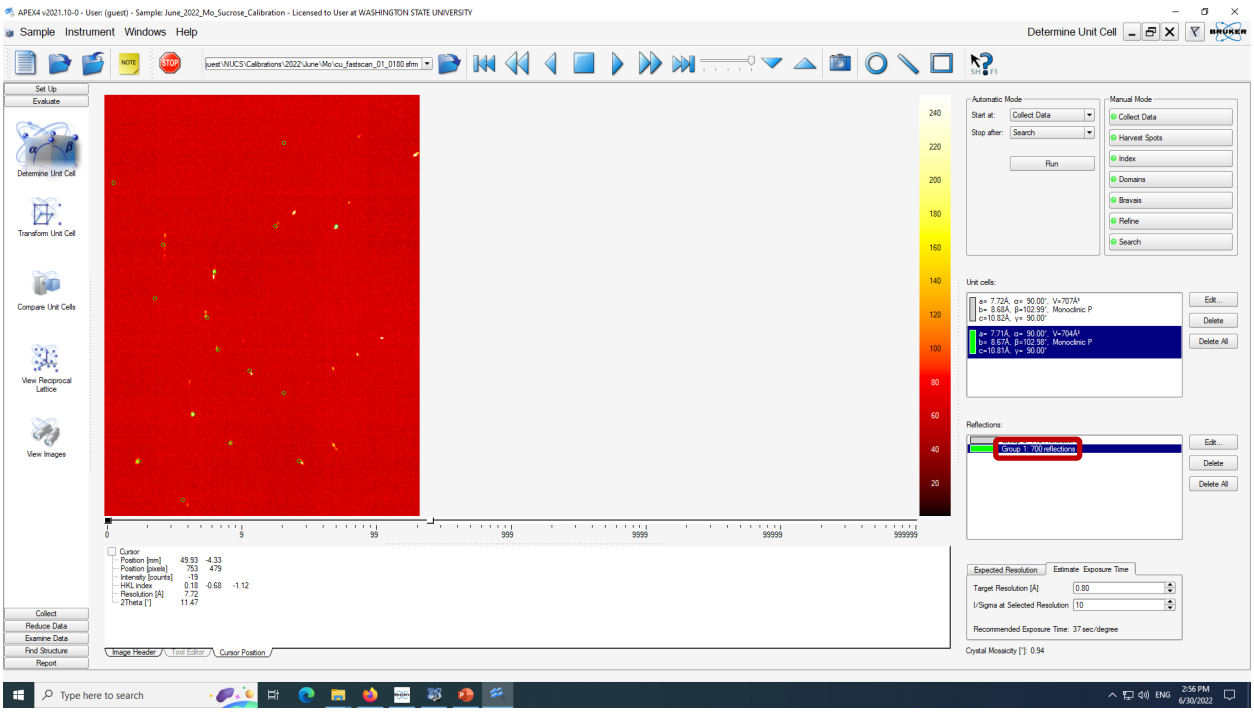
3.14 Click on the Collect Data button under the Manual Mode (highlighted in red) top set up a unit cell collection with the copper anode.



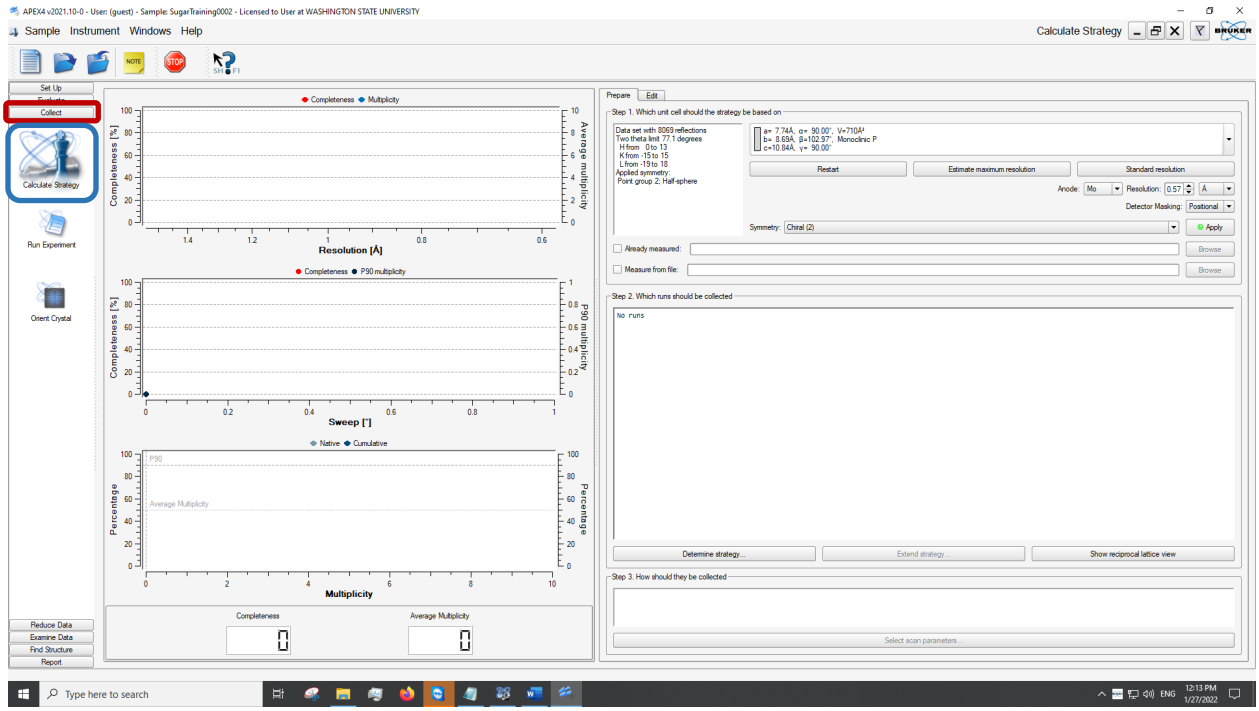
3.15 Change the anode from Mo to Cu to change to the copper X-ray tube (highlighted in red), and then click on the Finish button (highlighted in blue) to collect a unit cell.



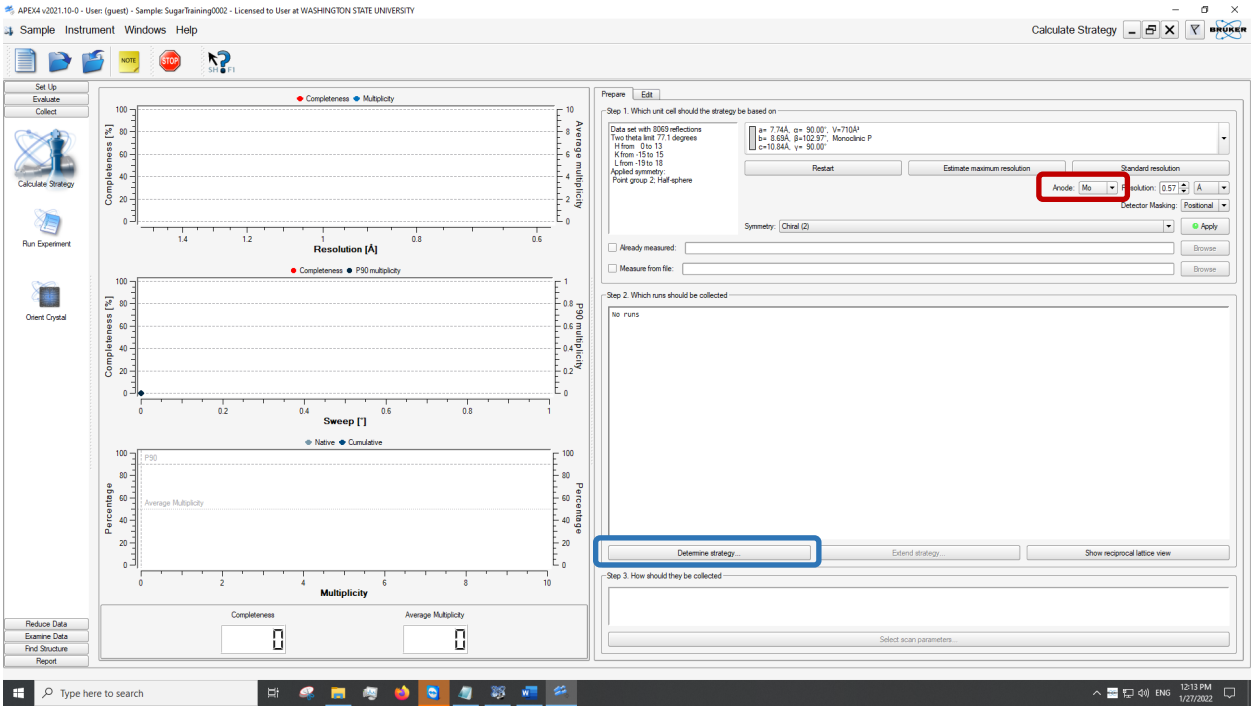
3.16 After the unit cell data collection has completed, write down the number of reflections for the unit cell data collection (highlighted in red) on the calibrations form for the copper anode. The first unit cell was measured with the molybdenum anode and the second one was measured with the copper anode.



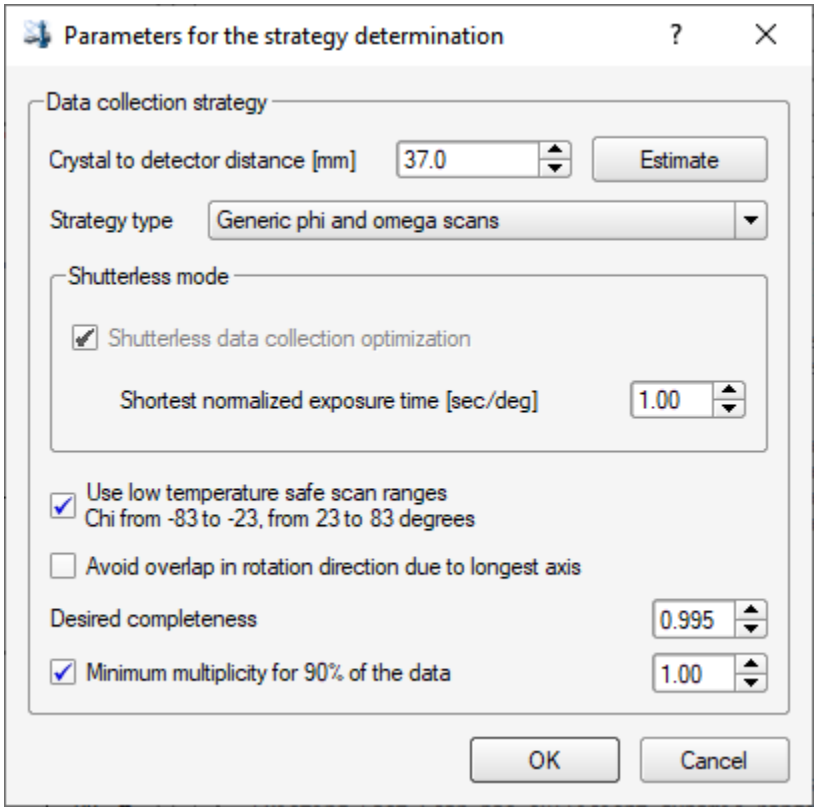
3.17 Click on Collect (highlighted in red) and then Calculate Strategy (highlighted in blue).



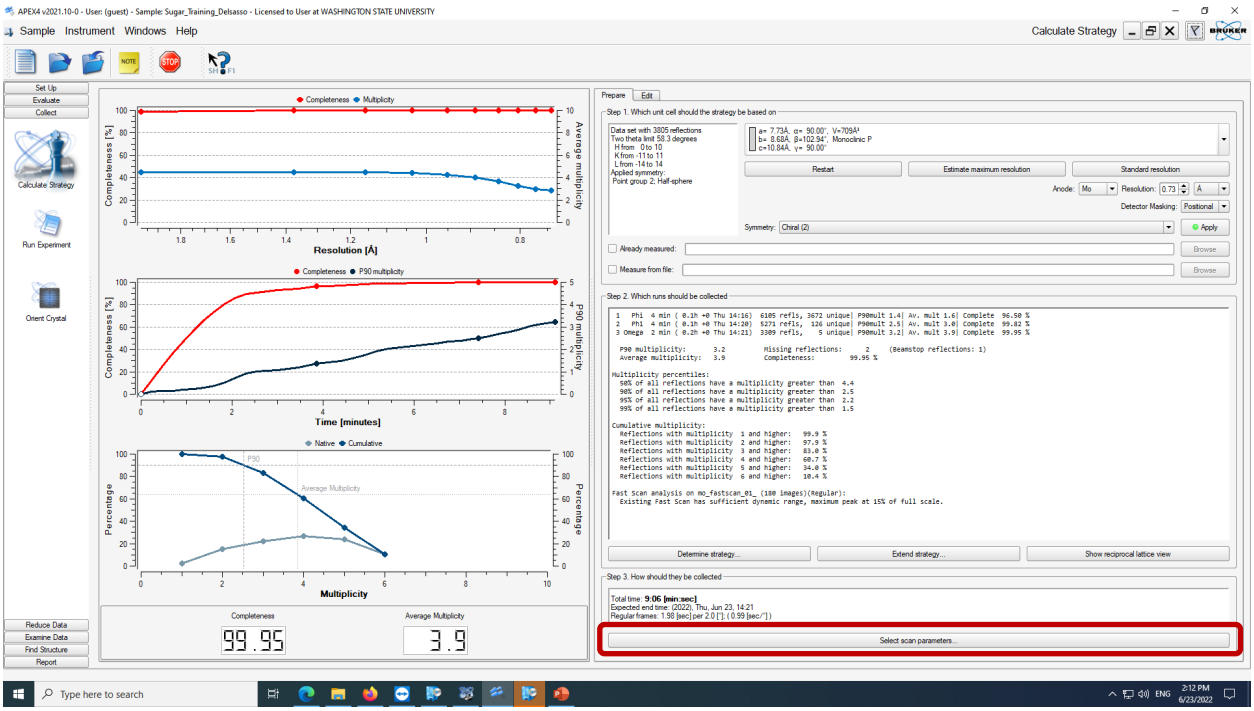
3.18 Check the anode (highlighted in red). It should be Mo for the first data collection and Cu for the second data collection. Click on the Determine Strategy button (highlighted in red).



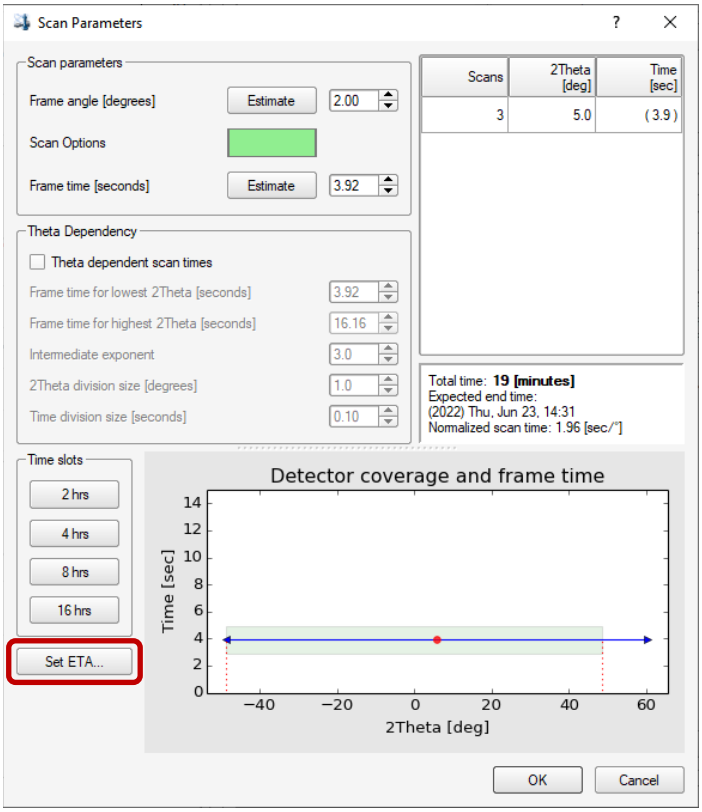
3.19 Accept the default values and click the OK button on the window that pops up.



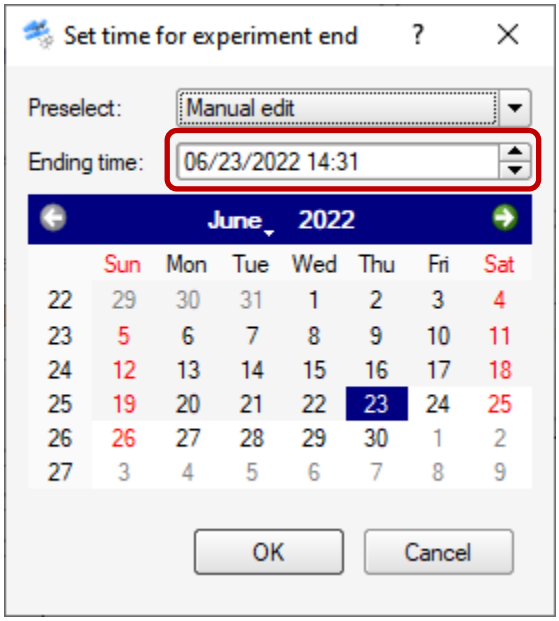
3.20 Upon clicking okay the computer will take about one minute to calculate a strategy and it will look similar to the following screen. Click on the Select scan parameters button (highlighted in red).



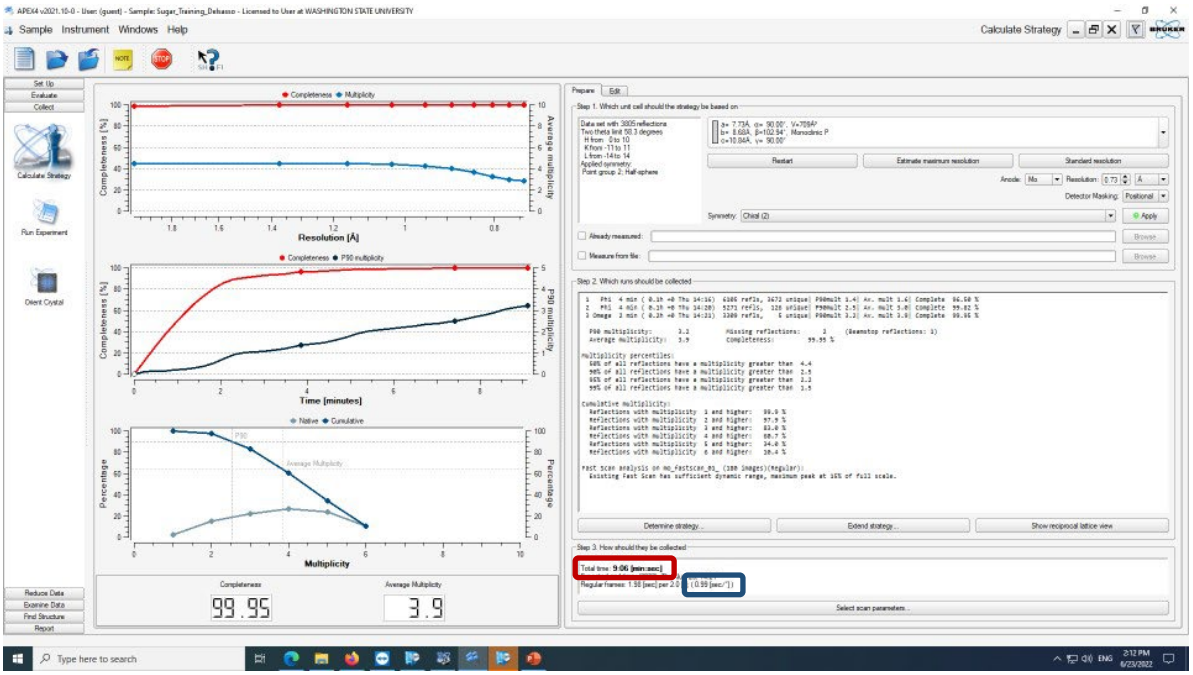
3.21 In the window that pops up, click on the Set ETA button (highlighted in red).



3.22 Select an ending time that is 9 to 10 minutes after the current time by typing it into the Ending time section and then clicking the OK button.

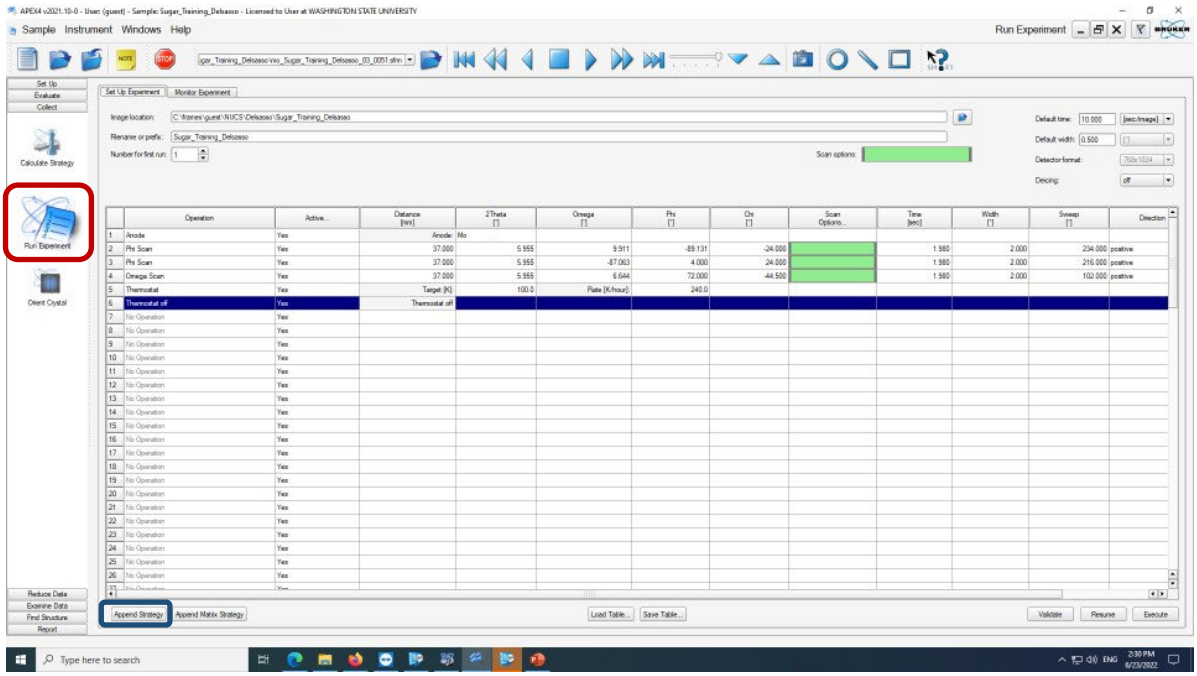


3.23 The resulting strategy should look like the following picture. The Total time should be between 9 and 10 minutes (highlighted in red).

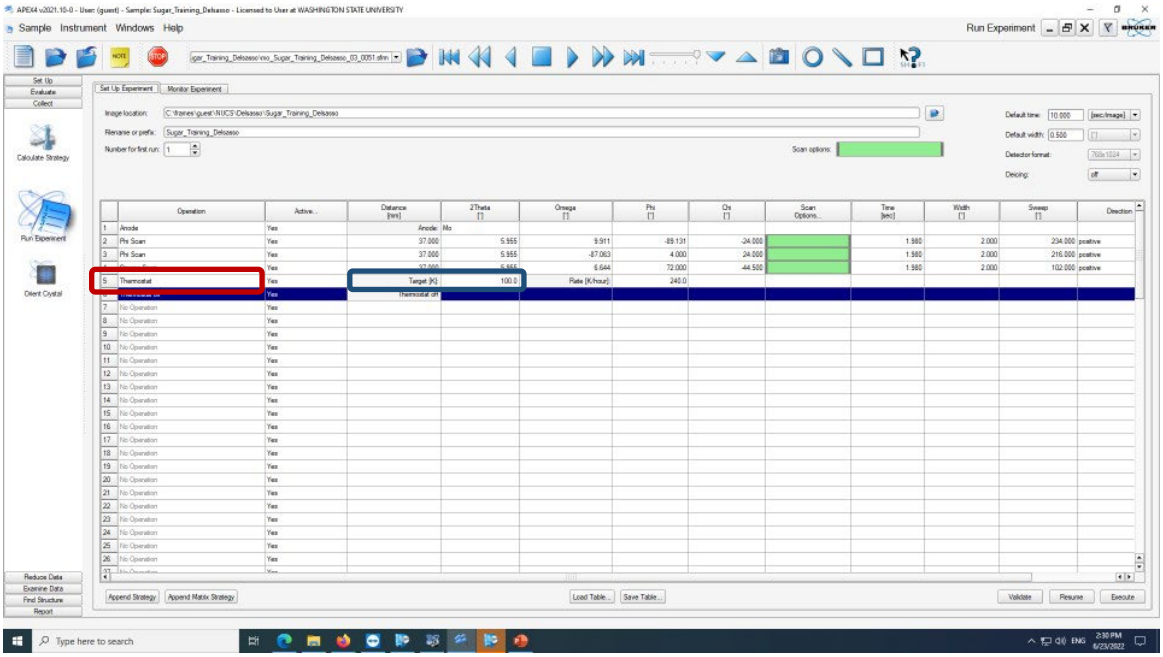


3.24 Before moving onto the next step, write down the frame time per frame width (sec/°) (highlighted in blue in the previous picture).

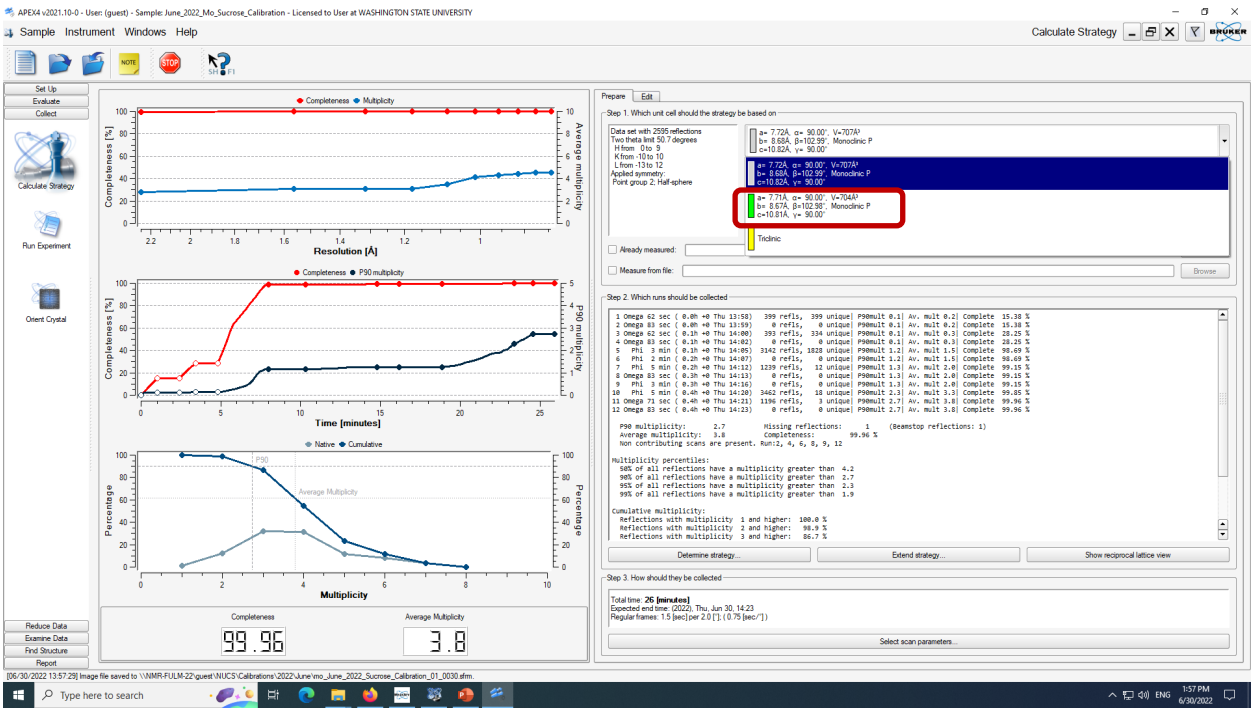
3.25 Click on the Run Experiment icon (highlighted in red) and then the Append Strategy button (highlighted in blue) to add the strategy from Steps 3.12 to 3.17.



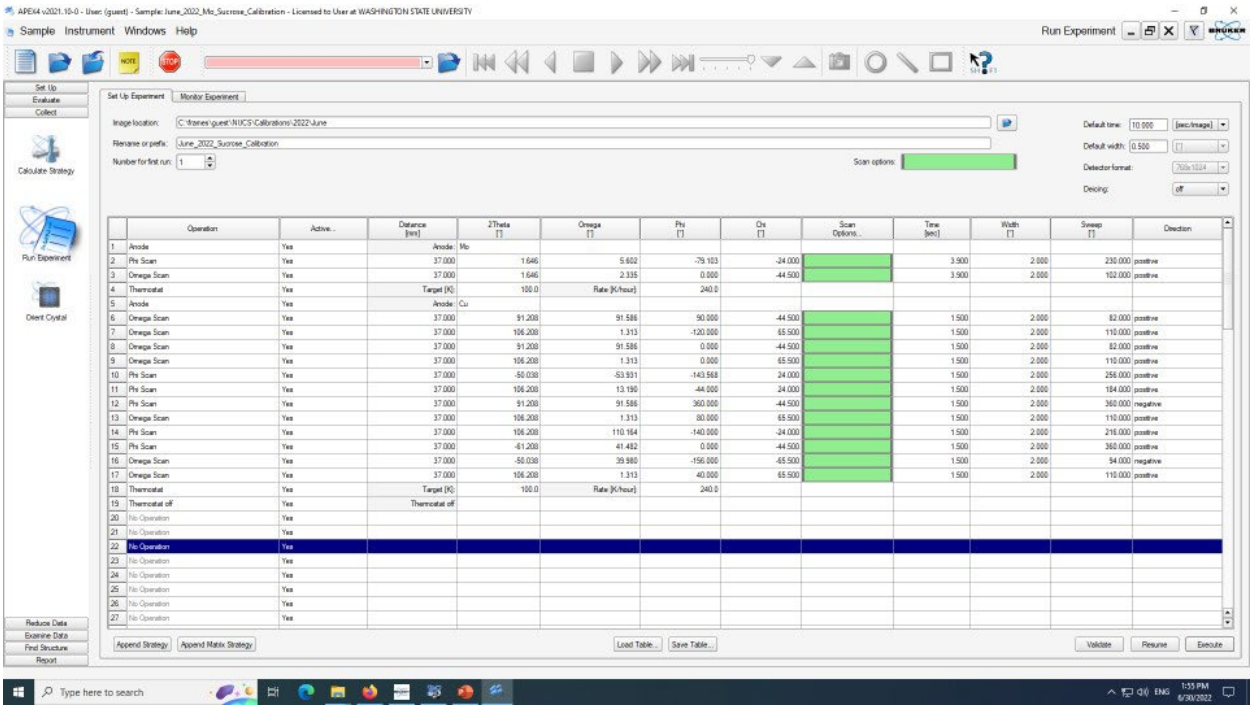
3.26 To run the data collection at 100K the temperature needs to be set in the experiment strategy before it is executed. To set the data collection temperature, click on the box in the Operation column directly below the last row of the strategy and select the option of Thermostat (highlighted in red). Then set the target temperature to 100K (highlighted in blue).



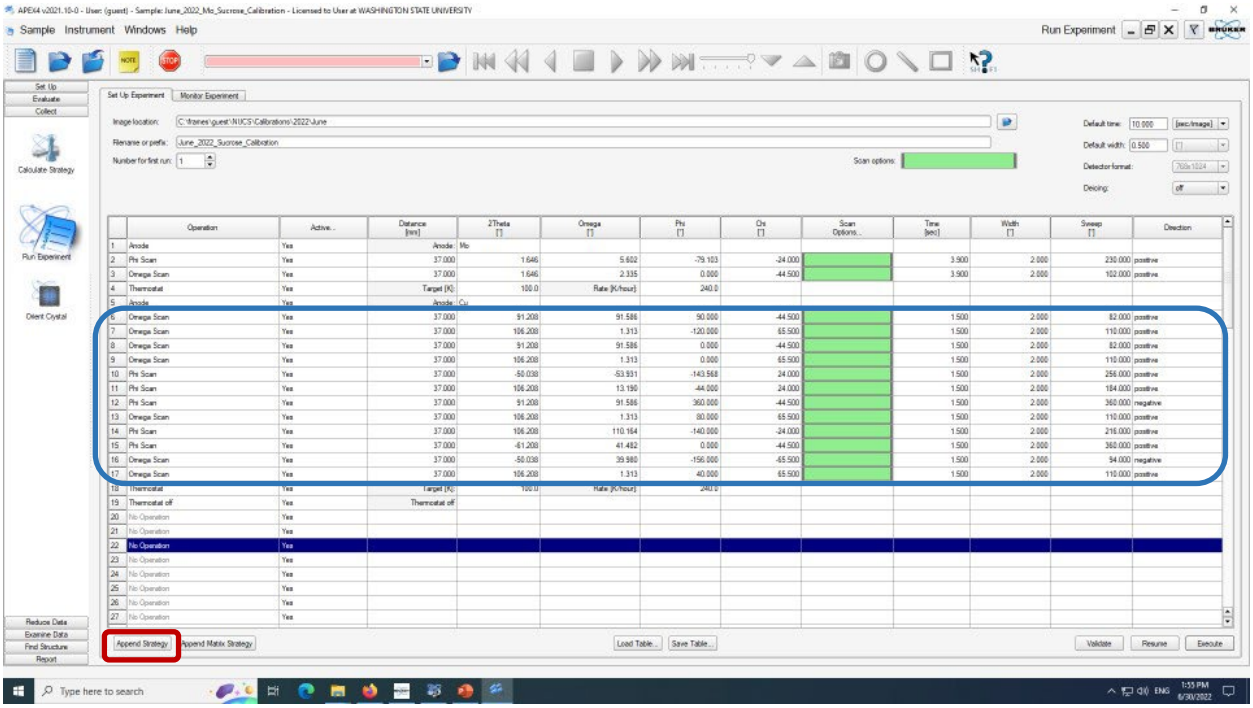
3.27 Return to Step 3.18 and select the second unit cell collected (with the Cu anode, highlighted in red) and set the anode to Cu. Repeat steps 3.19 to 3.24 for copper. The copper data collection will be a little longer (about 30 minutes).



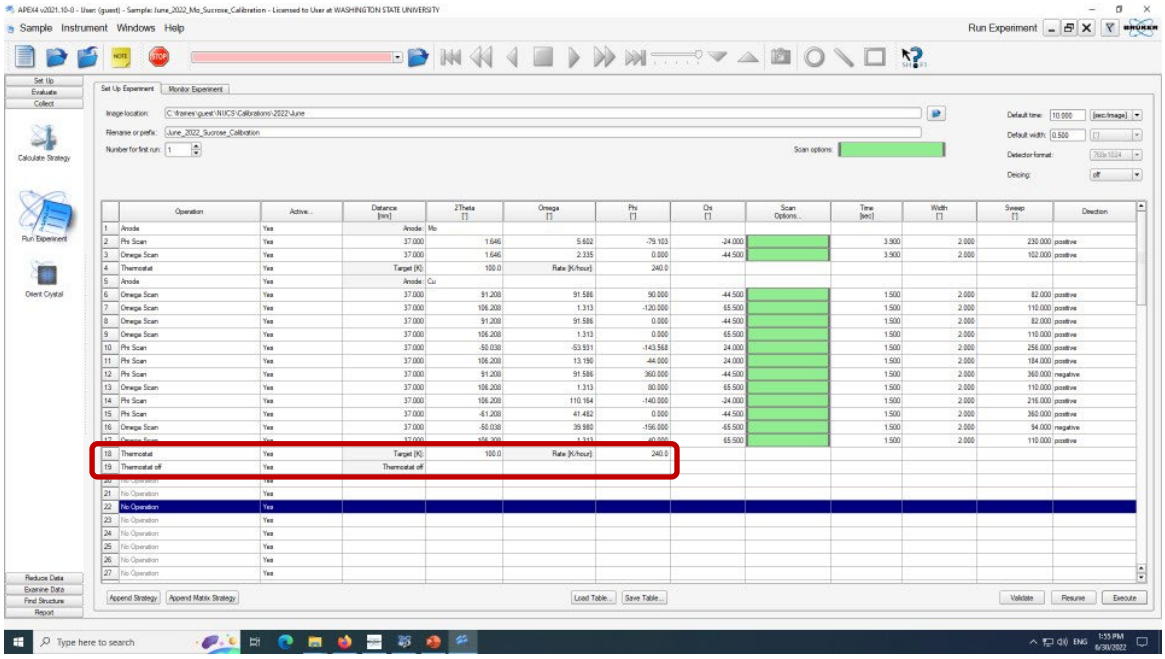
3.28 Click on the Run experiment Icon and select Anode in the Operation column and choose the Cu anode (highlighted in red).



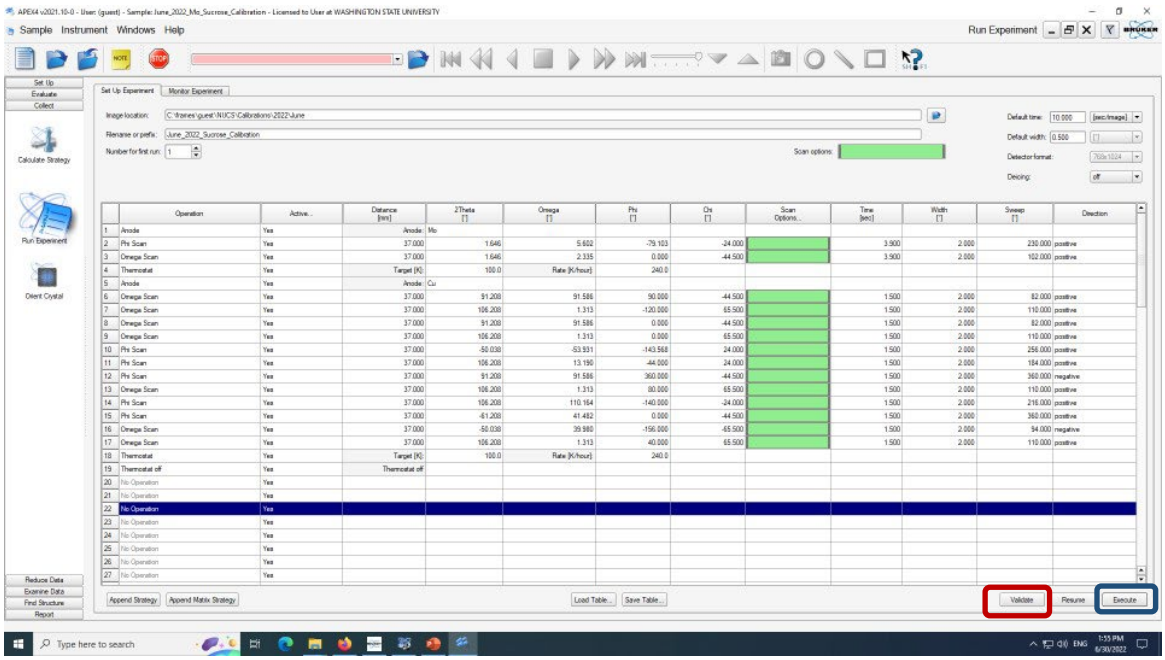
3.29 Then click the Append Strategy button (highlighted in red) to add the strategy for the Cu anode (highlighted in blue).



3.30 Then, add the thermostat option (as seen in 3.26) to collect data at 100K (highlighted in red). Also, add an entry to the next row, by selecting Thermostat off under the Operation column (highlighted in red) to have the instrument automatically turn of the cold stream when the data collection has completed. This step should only be added after the second data collection with the Cu anode, otherwise the instrument will need to be cooled down again for the second data set.

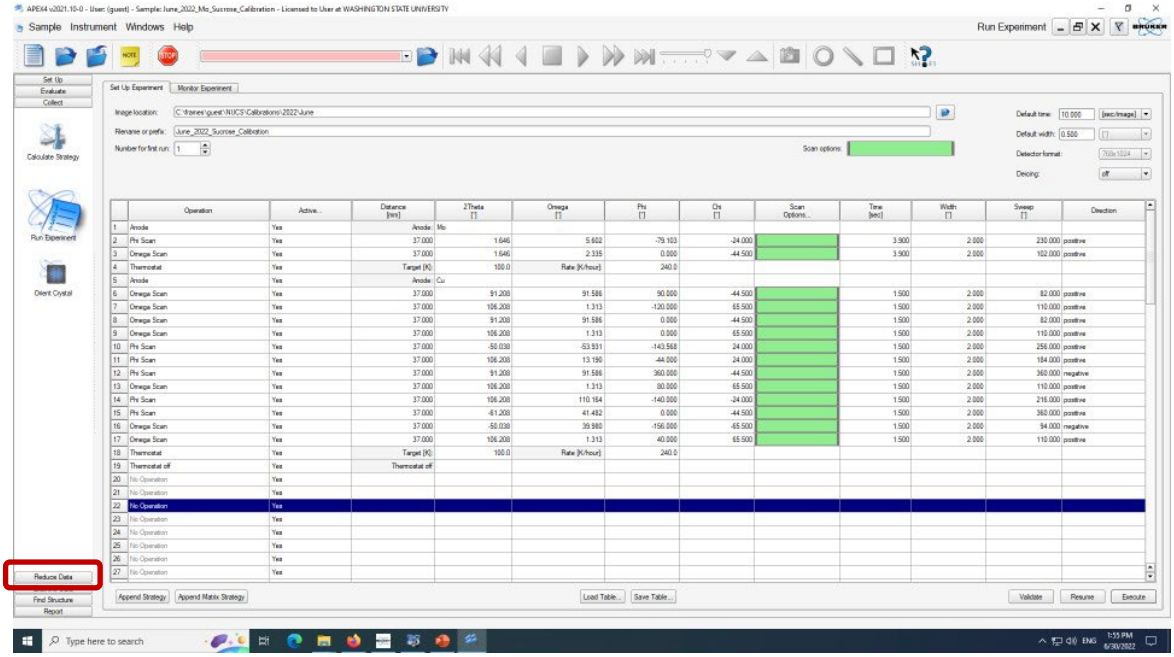


3.31 To execute the data collection, click on the Validate button (highlighted in red) and if all of the tasks are valid then the Execute button (highlighted in blue) can be clicked. Data will be collected with the Mo anode first followed by a data collection with the Cu anode.

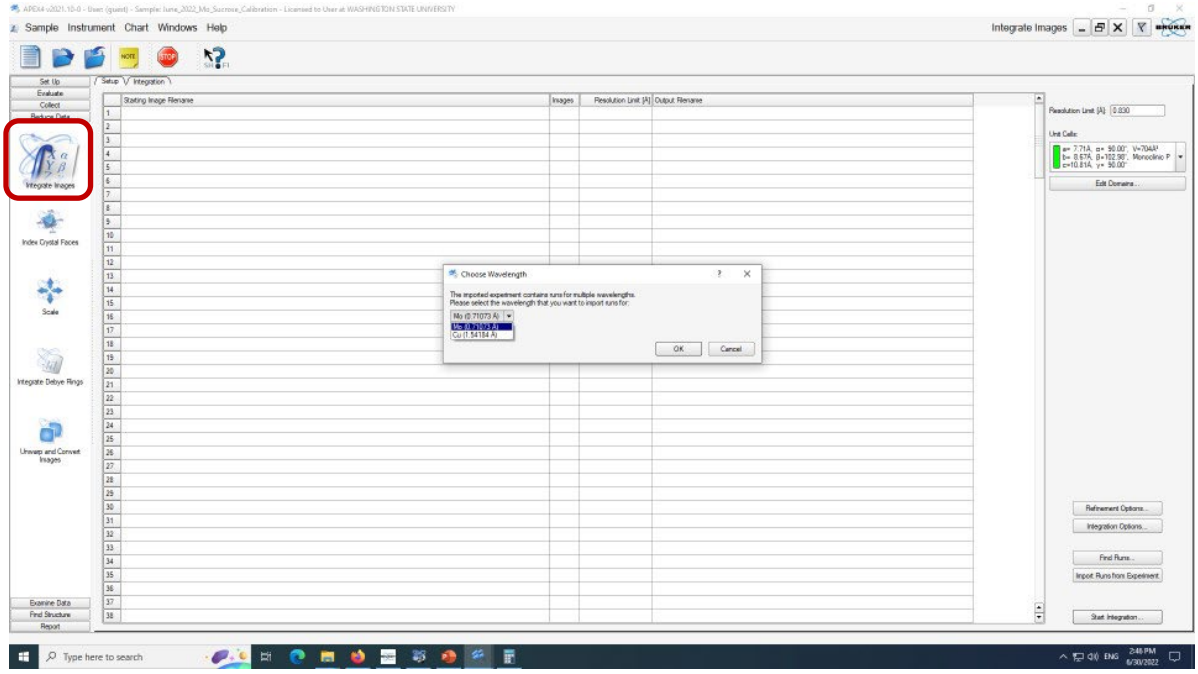


3.32 When the data collection begins, it will look similar to the screen seen during the unit cell data collection.

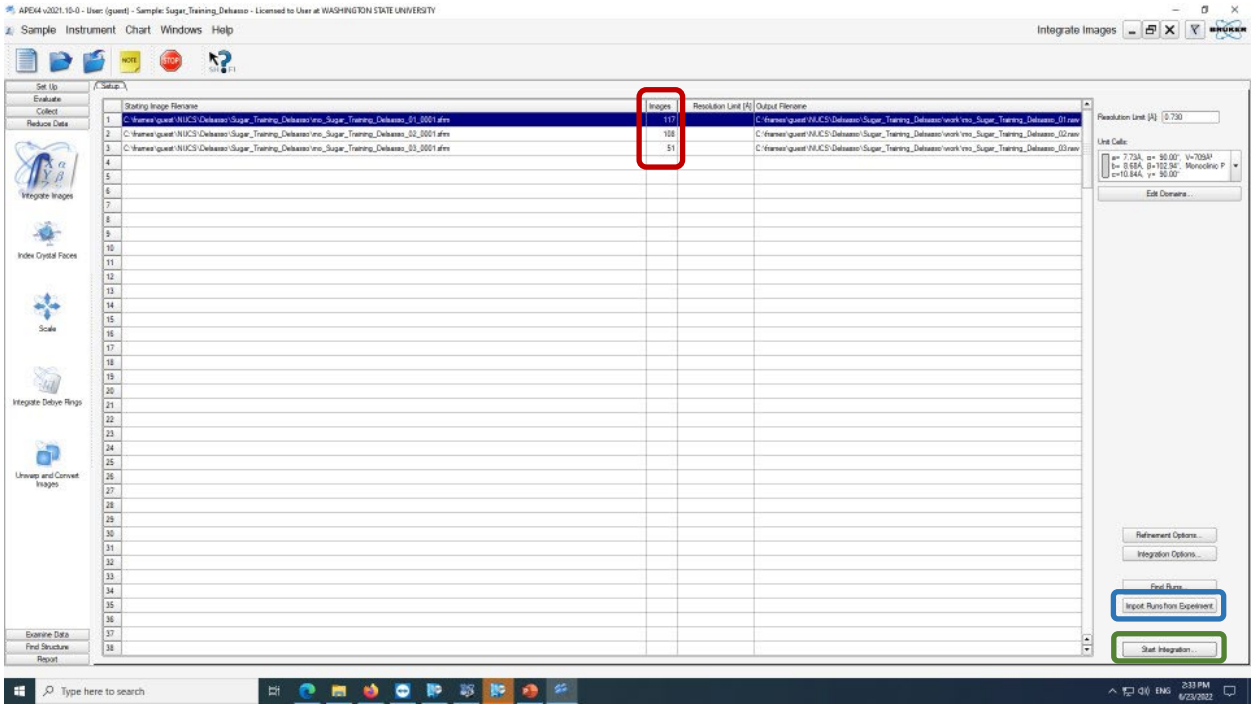
3.33 When the data collection has completed, Click on the Reduce Data Button on the left side of the screen (highlighted in red).



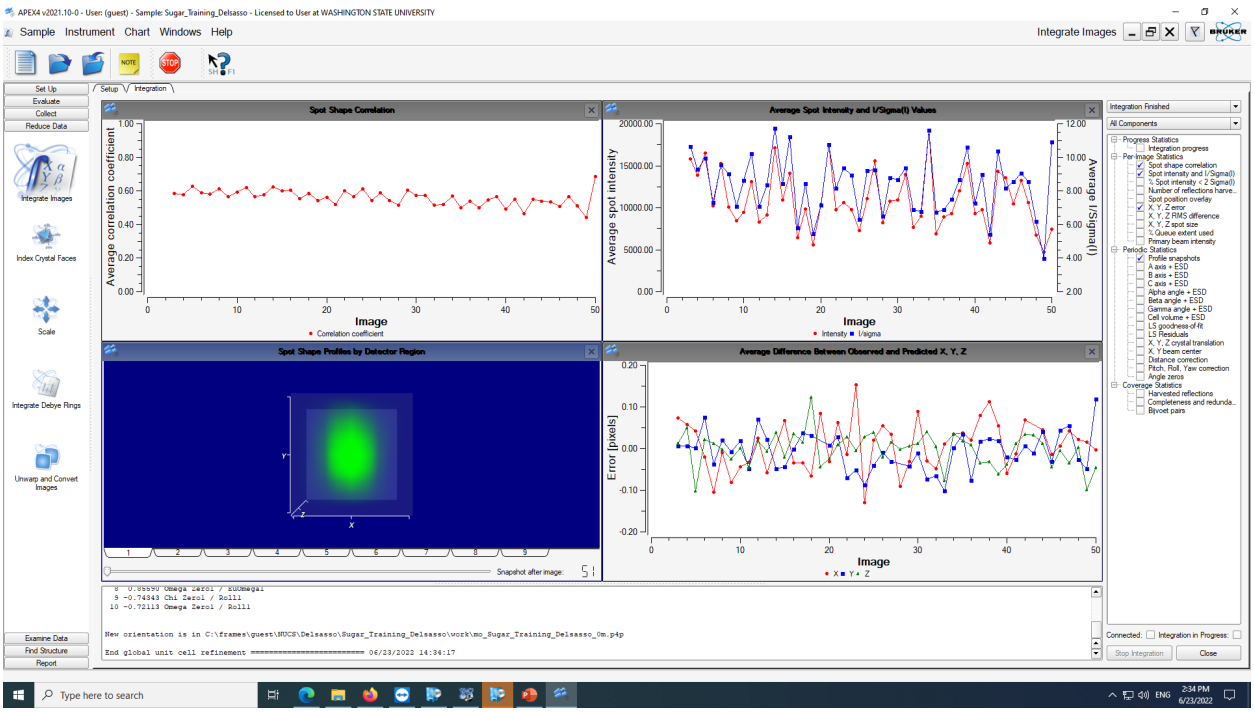
3.34 Click on the Integrate Images Icon (highlighted in red), a window showing the choice between the Mo and Cu data sets will appear. Choose the Mo option, and click the OK button.



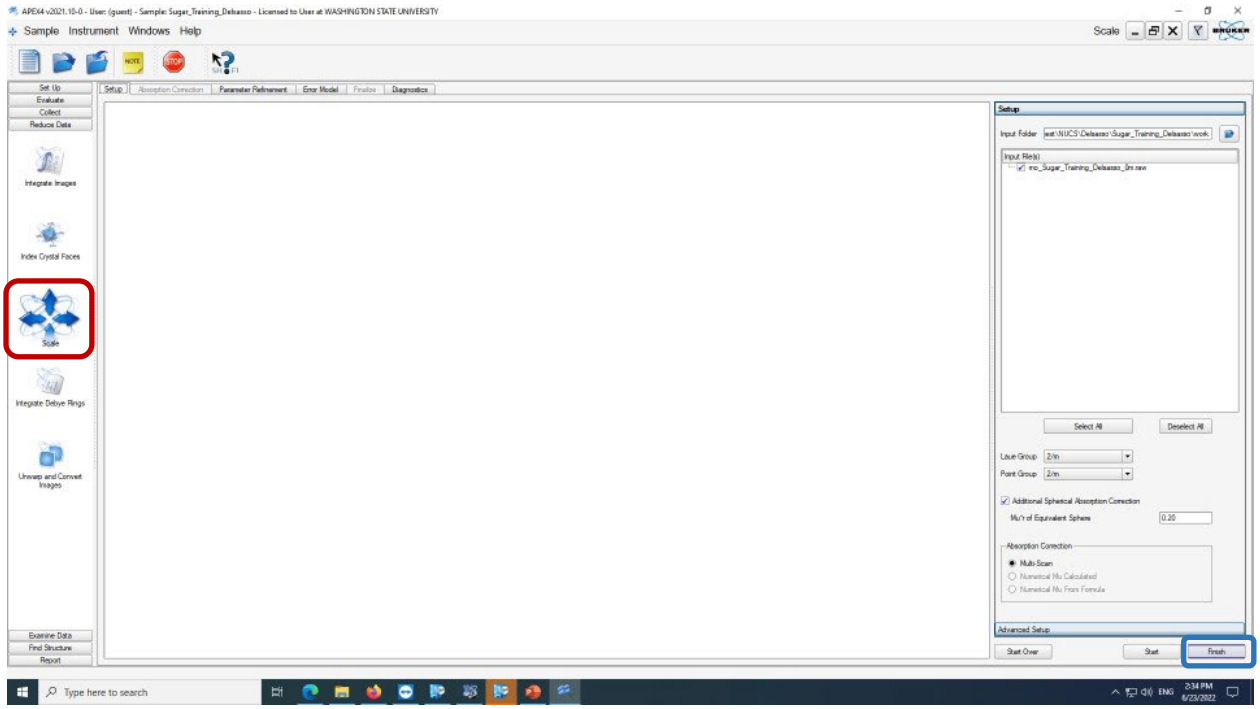
3.35 If the runs do not immediately show up in the Setup window, click on the Import Runs from Experiment button (highlighted in blue). Once the images are added to the Integration window, sum up the total number of images (highlighted in red) and place the total on the calibration form. Start the integration by clicking on the Integration button (highlighted in green).



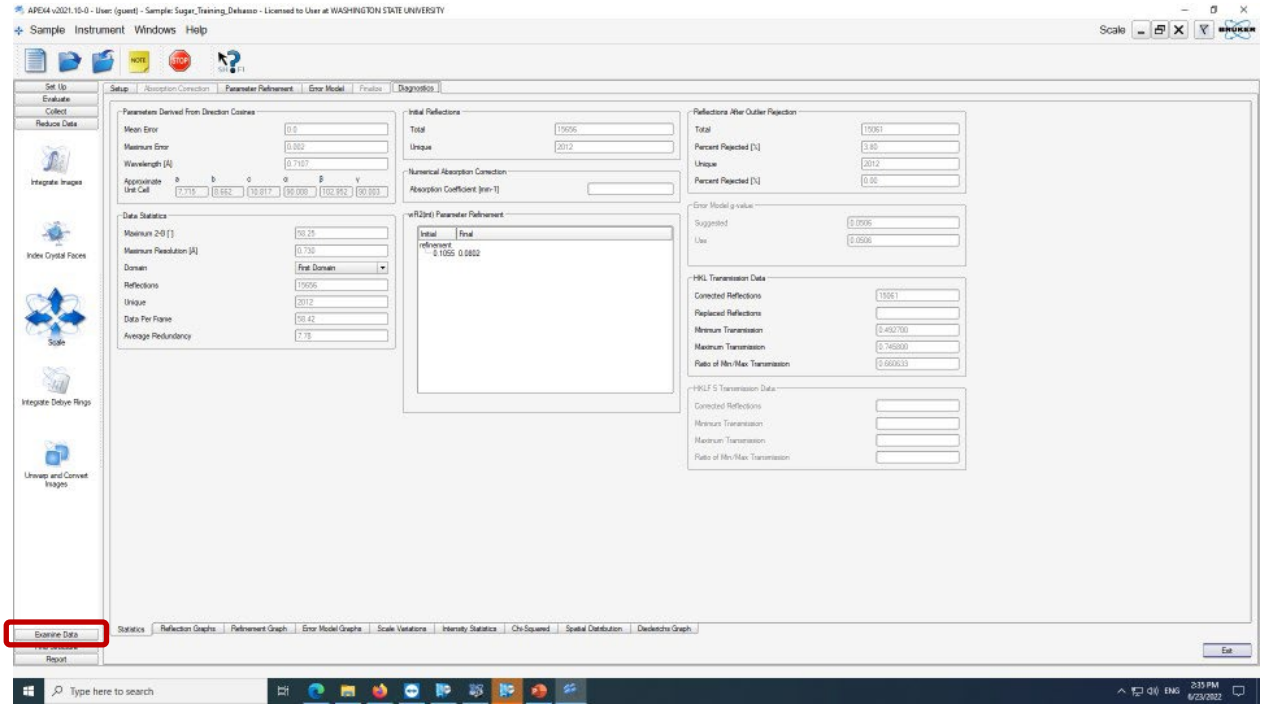
3.36 It will take roughly a minute to complete the integration. The following screen will result when the integration has completed.



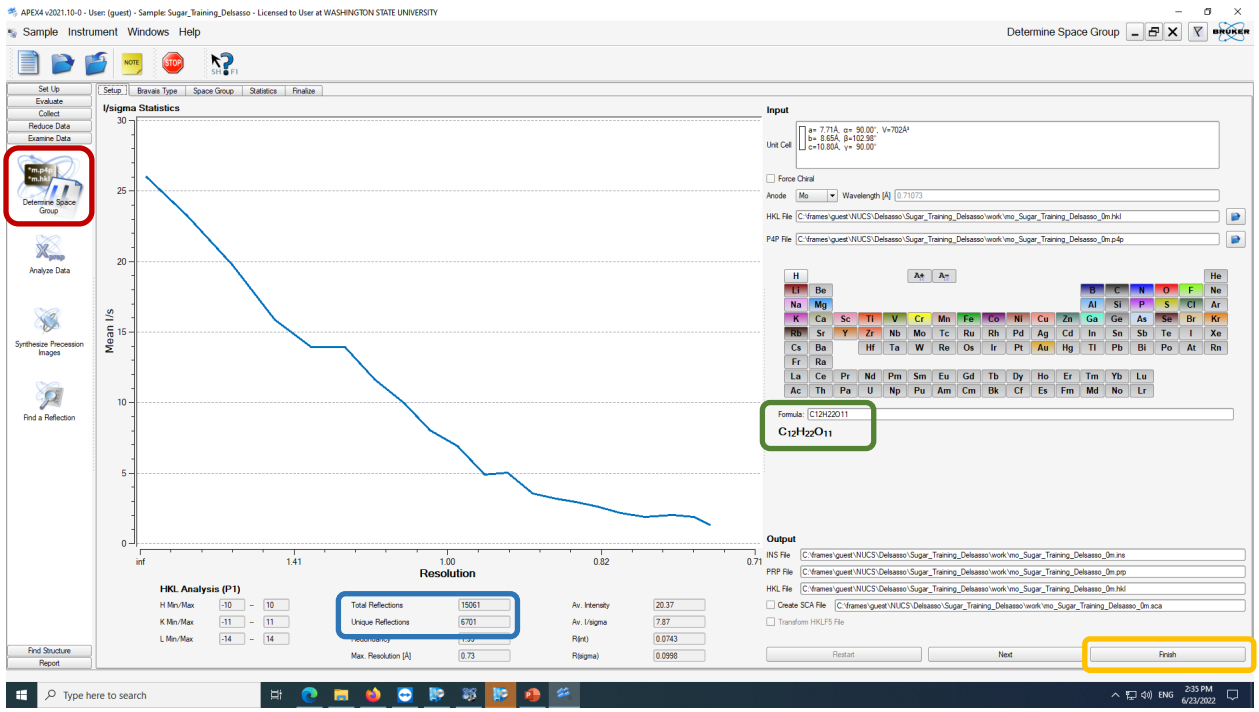
3.37 Then click on the Scale icon (highlighted in red) followed by the Finish button (highlighted in blue).



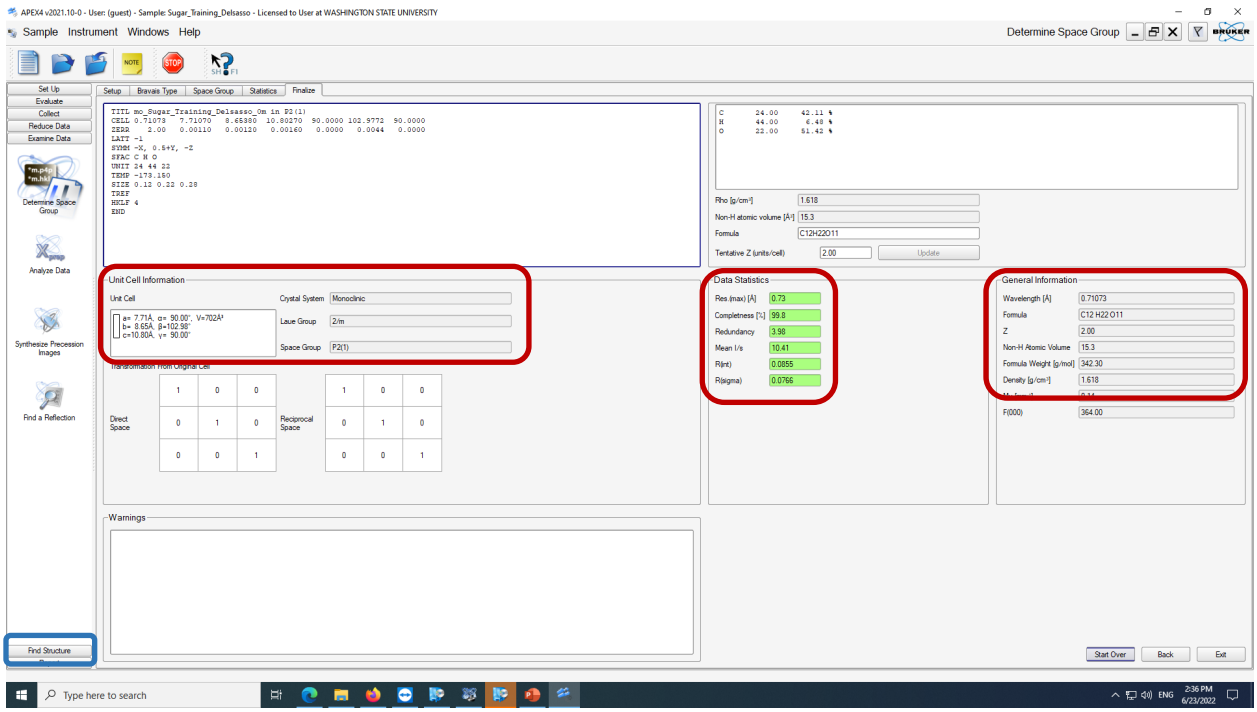
3.38 Scaling of the X-ray diffraction data has completed when the following screen appears. Click on the Examine Data button (highlighted in red) to continue.



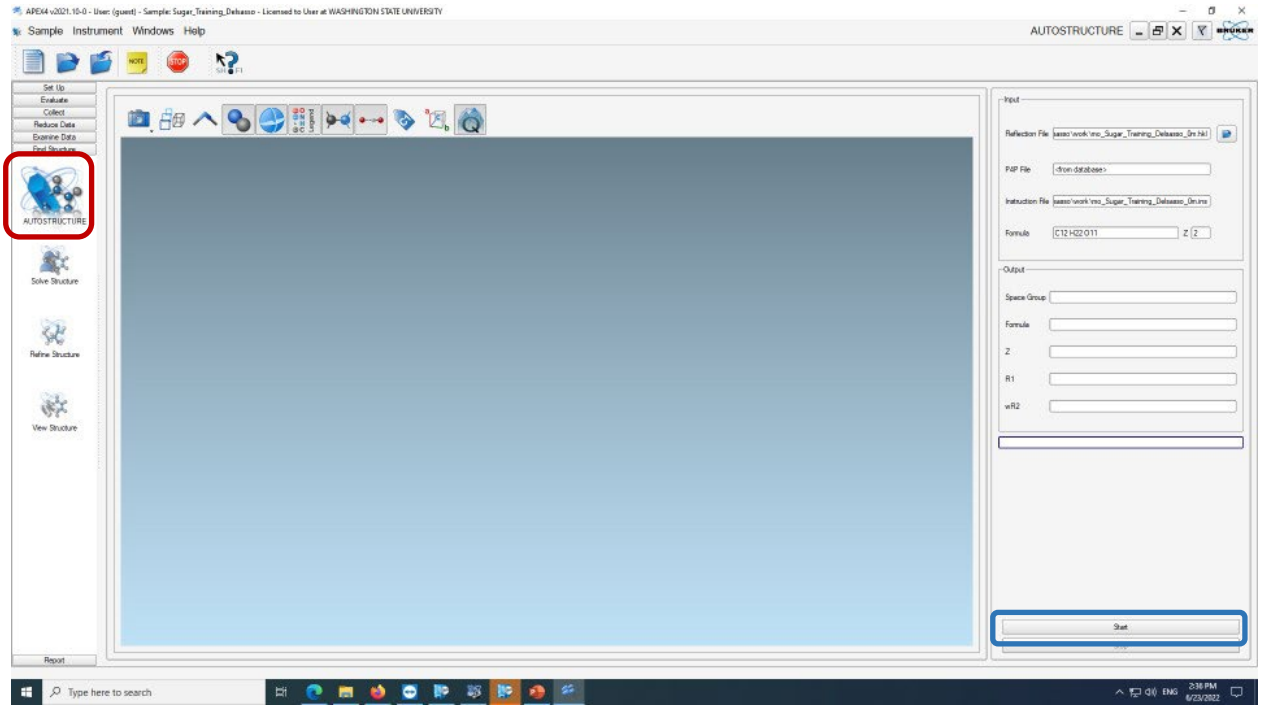
3.39 Click on the Determine Space Group Icon (highlighted in red) and the following screen will result. Write down the Total Reflections and number of Unique Reflections (highlighted in blue) on the calibration form. Check to make sure the formula for sucrose is entered ($C_{12}H_{22}O_{11}$, highlighted in green) before clicking on the Finish button (highlighted in orange).



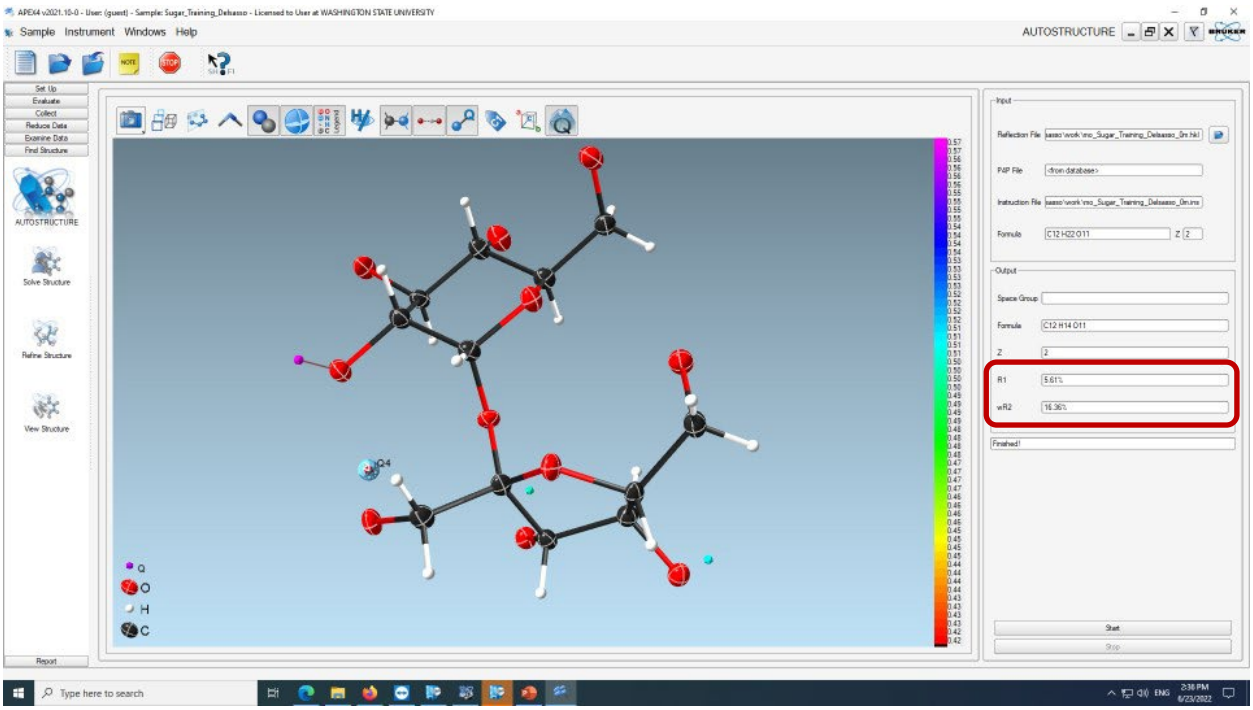
3.40 The following screen will result. Write down the information highlighted in red on the calibration form and when finished, click on the Find Structure button (highlighted in blue).



3.41 Click on the AUTOSTRUCTURE icon (highlighted in red), followed by the Start button (highlighted in blue).

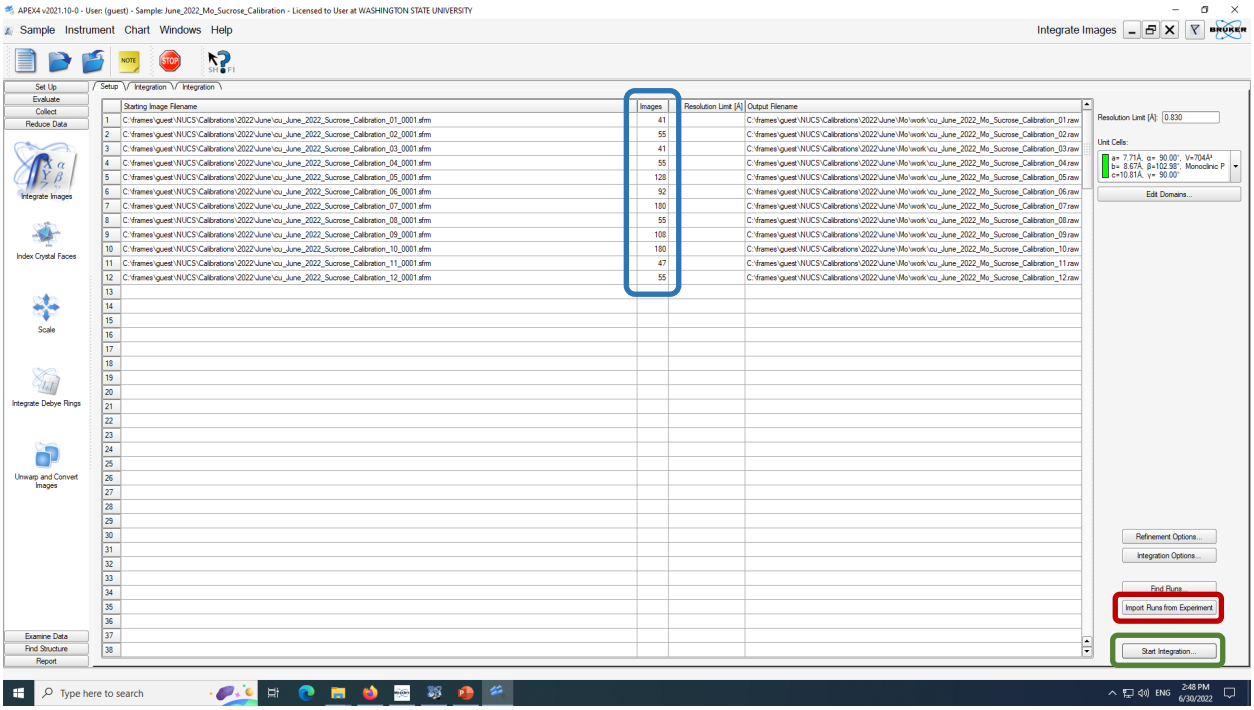


3.42 After about 10 seconds or less the following screen will result. Enter the R1 and wR2 values (highlighted in red) seen in the calibration report. Make a screenshot of the window (using the Print Screen button) and save as Month Year Sucrose Calibration Anode Structure.png (e.g. June 2022 Sucrose Calibration Mo Structure.png). Save the picture of the structure at the following location: Z:\Bruker D8\NUCS\Monthly_Calibrations\Year\Month Year



3.43 After the data collection for the molybdenum X-ray tube has been completed, return to Step 3.34 and select Cu anode.

3.44 Delete all of the images for the Mo anode by right clicking on the images under the Starting Image Filename column and select Delete. Then, click on the Import Runs from Experiment button (highlighted in red). Once the images are added to the Integration window, sum up the total number of images (highlighted in blue) and place the total on the calibration form. Start the integration by clicking on the Integration button (highlighted in green).



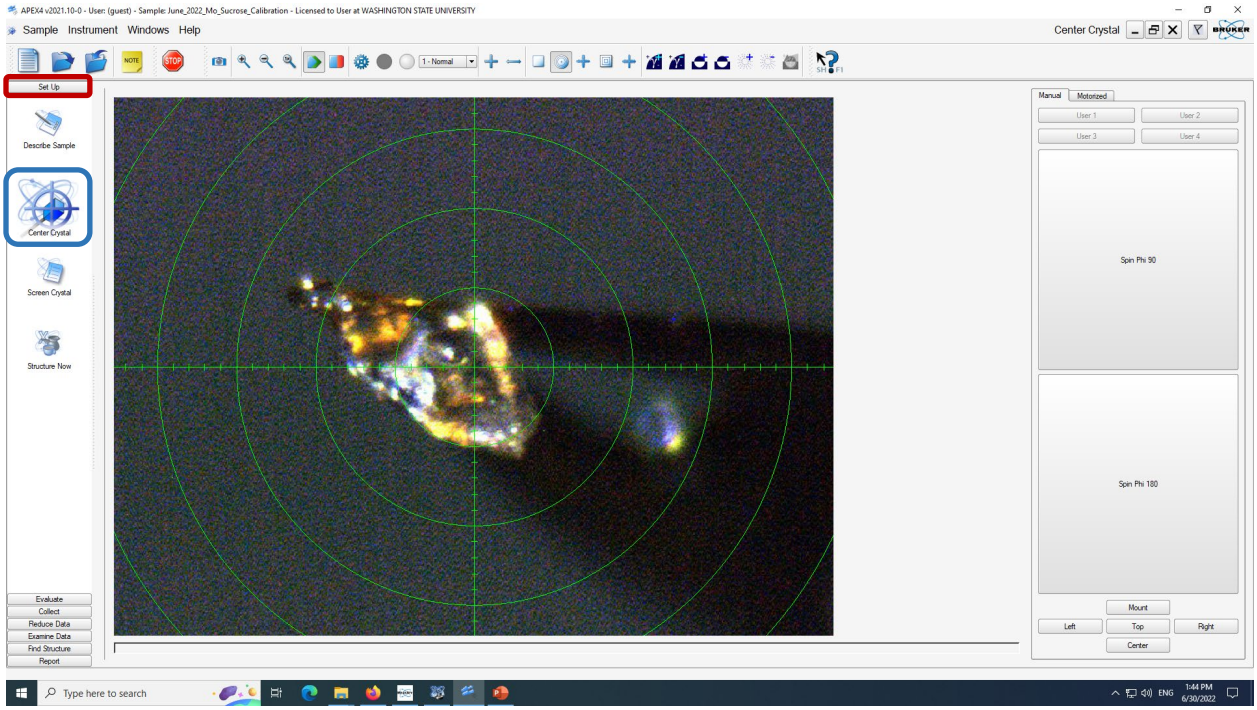
3.45 Change the anode from Mo to Cu to change to the copper X-ray tube (highlighted in red), and then click on the Collect button (highlighted in blue).

3.46 After the unit cell data collection has completed, write down the number of reflections for the unit cell data collection (highlighted in red) on the calibrations form.

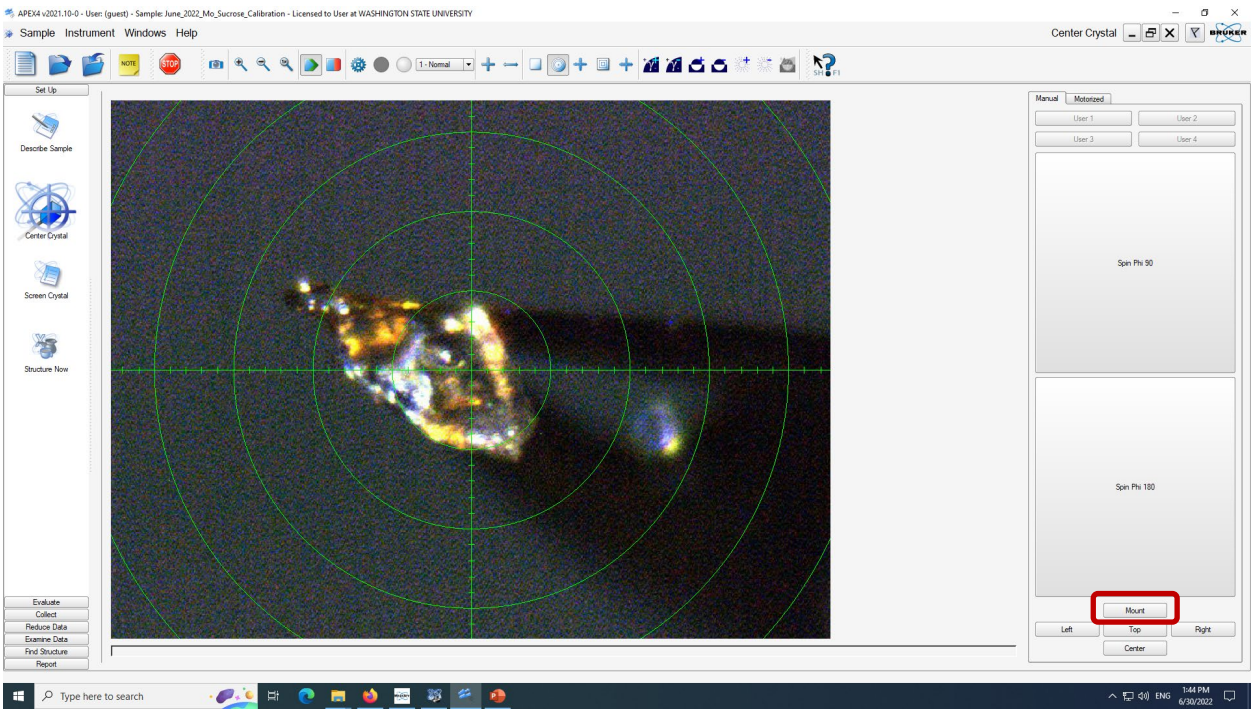
3.47 Repeat steps 3.36 to 3.42 for the copper anode.

3.48 When the data collection has completed, the monthly calibration can be completed, see Section 4.

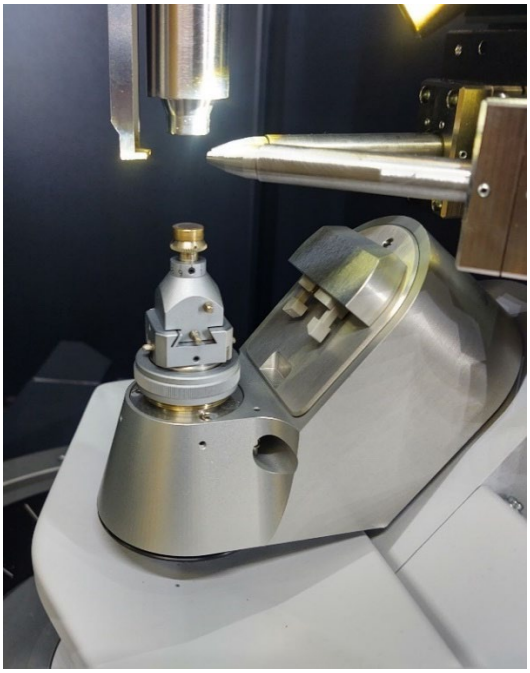
3.49 Once you have completed your data analysis and the monthly report can been completed, click on the Set Up button (highlighted in red) and then on Center Crystal icon (highlighted in blue).



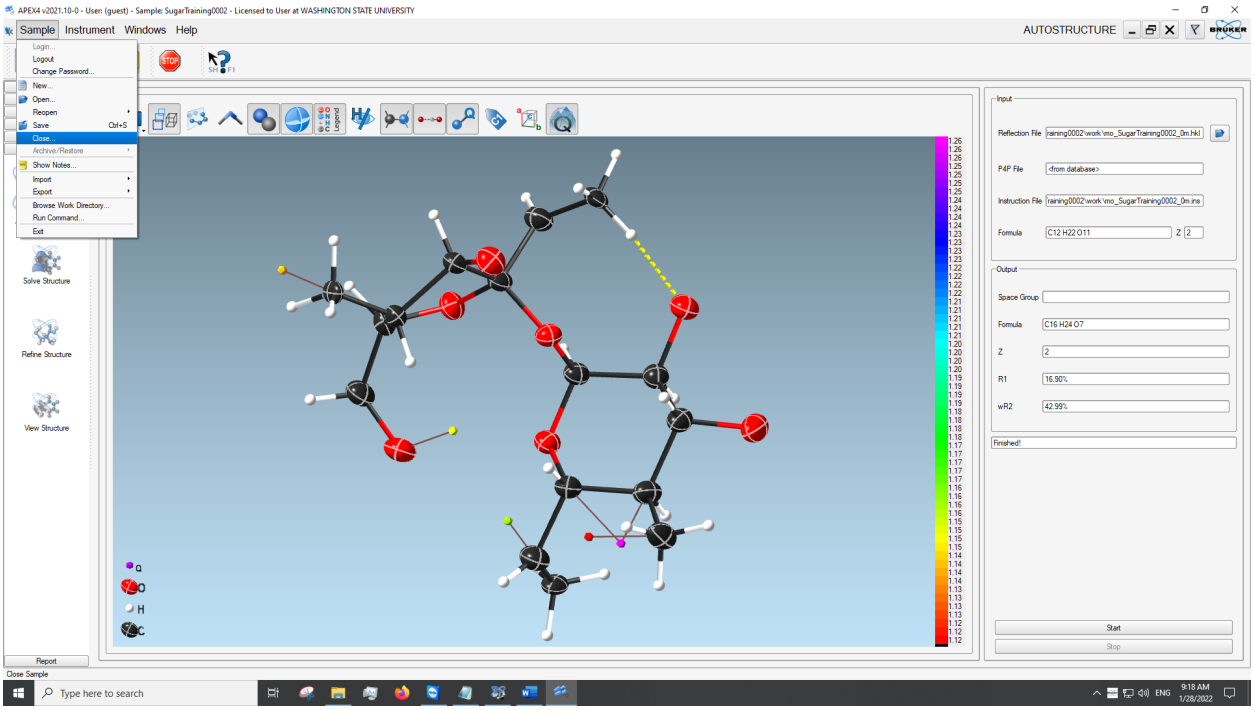
3.50 Then click on the Mount button (highlighted in red) to move the goniometer in a position where the calibration standard can be removed.



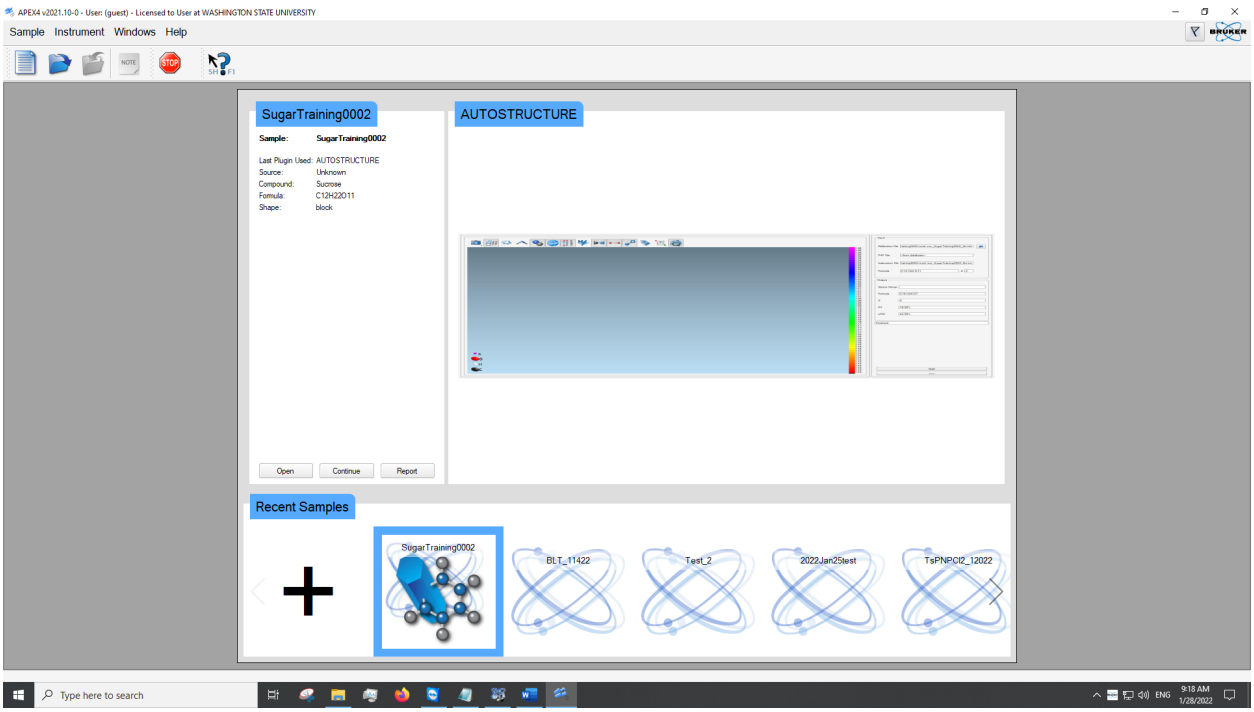
3.51 Then open the doors and remove your sample from the goniometer. The goniometer should look like the following picture when finished removing the sample.



3.52 Once you have removed your sample form the diffractometer, please go to Sample-> Close and close your data collection so that the instrument is available for the next user.

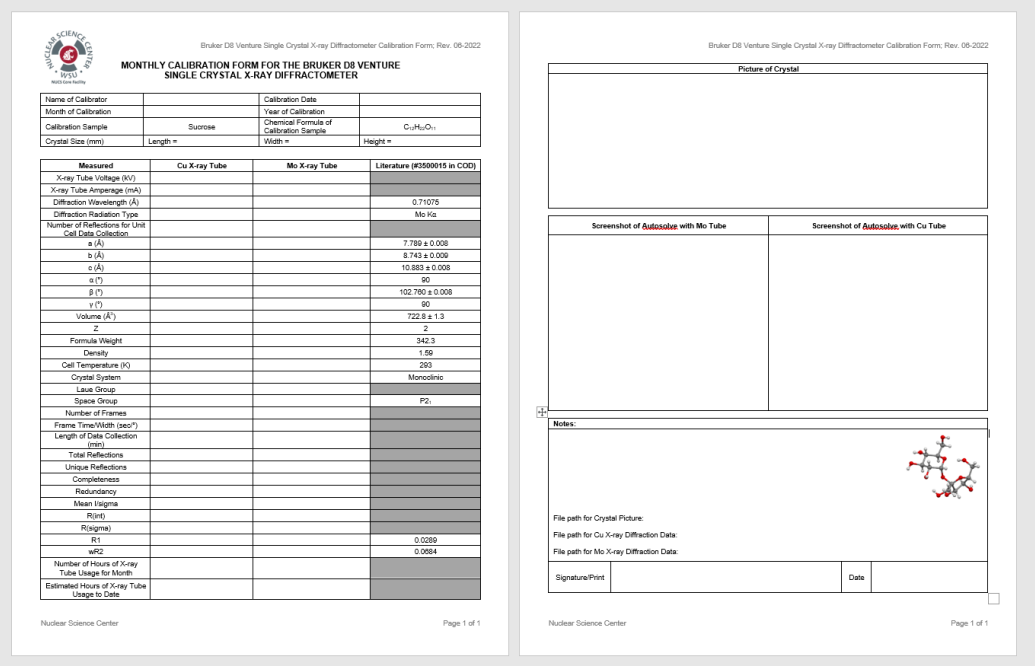


3.53 The following window should be visible when you have properly closed out of your sample and it is available for the next user.



4 Reporting Monthly Calibration

4.1 The form for the monthly calibrations report can be found at the following location: Z:\Bruker D8\NUCS\Monthly_Calibrations. An example of a blank form can be seen below.



Bruker D8 Venture Single Crystal X-ray Diffractometer Calibration Form; Rev. 08-2022

MONTHLY CALIBRATION FORM FOR THE BRUKER D8 VENTURE SINGLE CRYSTAL X-RAY DIFFRACTOMETER

Name of Calibrator		Calibration Date	
Month of Calibration		Year of Calibration	
Calibration Sample	Sucrose	Chemical Formula of Calibration Sample	$C_{12}H_{22}O_{11}$
Crystal Size (mm)	Length =	Width =	Height =

Measured	Cu X-ray Tube	Mo X-ray Tube	Literature (#3500015 in COD)
X-ray Tube Voltage (kV)			
X-ray Tube Amperage (mA)			
Diffraction Wavelength (Å)			0.71075
Diffraction Radiation Type			Mo Kα
Number of Reflections for Unit Cell Data Collection			
a (Å)			7.789 ± 0.008
b (Å)			8.743 ± 0.009
c (Å)			10.883 ± 0.008
α (°)			90
β (°)			102.760 ± 0.008
γ (°)			90
Volume (Å^3)			722.8 ± 1.3
Z			2
Formula Weight			342.3
Density			1.59
Cell Temperature (K)			293
Crystal System			Monoclinic
Laue Group			
Space Group			$P2_1$
Number of Frames			
Frame Time/Width (sec/°)			
Length of Data Collection (min)			
Total Reflections			
Unique Reflections			
Completeness			
Redundancy			
Mean I/sigma			
R(int)			
R(sigma)			
R1			0.0289
wR2			0.0684
Number of Hours of X-ray Tube Usage for Month			
Estimated Hours of X-ray Tube Usage to Date			

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Bruker D8 Venture Single Crystal X-ray Diffractometer Calibration Form; Rev. 08-2022

Picture of Crystal

Screenshot of Autosolve with Mo Tube	Screenshot of Autosolve with Cu Tube

Notes:

File path for Crystal Picture:

File path for Cu X-ray Diffraction Data:

File path for Mo X-ray Diffraction Data:

Signature/Print		Date	

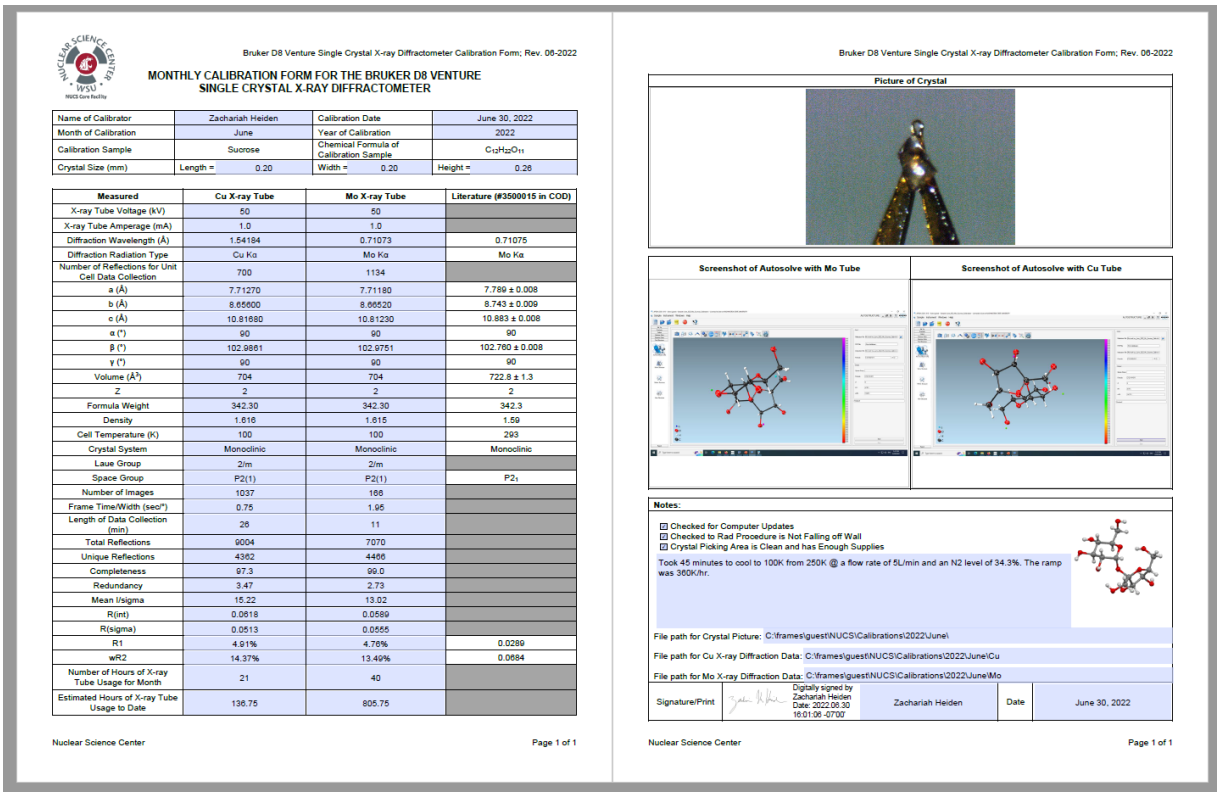
Nuclear Science Center Page 1 of 1

4.2 Enter the respective data, as described in Steps 3.10, 3.12, 3.19, 3.20, 3.31, 3.32, 3.34, and 3.39.

4.3 The number of estimated Hours of X-ray Tube Usage to date can be determined by adding the number of hours over the last month to the value on the previous calibration form.

4.4 If the number of reflections are below 1000, the R1 value is above 0.10, or the estimated number of hours of an X-ray tube to date is greater than 9000 hours, then there is a reason to be concerned and inform NUCS Core Facility staff.

4.5 A completed form should like the following and be saved here: Z:\Bruker D8\NUCS\Monthly_Calibrations\Year\Month Year (e.g. Z:\Bruker D8\NUCS\Monthly_Calibrations\2022\June 2022) as Bruker D8 Venture Calibration Form for Month Year.pdf (e.g. Bruker D8 Venture Calibration Form for June 2022.pdf).



Bruker D8 Venture Single Crystal X-ray Diffractometer Calibration Form; Rev. 06-2022

MONTHLY CALIBRATION FORM FOR THE BRUKER D8 VENTURE SINGLE CRYSTAL X-RAY DIFFRACTOMETER

Name of Calibrator	Zachariah Heiden	Calibration Date	June 30, 2022
Month of Calibration	June	Year of Calibration	2022
Calibration Sample	Sucrose	Chemical Formula of Calibration Sample	$C_{12}H_{22}O_{11}$
Crystal Size (mm)	Length = 0.20	Width = 0.20	Height = 0.26

Measured	Cu X-ray Tube	Mo X-ray Tube	Literature (#3500015 in COD)
X-ray Tube Voltage (kV)	50	50	
X-ray Tube Amperage (mA)	1.0	1.0	
Diffraction Wavelength (Å)	1.54184	0.71073	0.71075
Diffraction Radiation Type	Cu Kα	Mo Kα	Mo Kα
Number of Reflections for Unit Cell Data Collection	700	1134	
a (Å)	7.71270	7.71180	7.789 ± 0.008
b (Å)	8.65600	8.66520	8.743 ± 0.009
c (Å)	10.81680	10.81230	10.883 ± 0.008
α (°)	90	90	90
β (°)	102.9861	102.9751	102.760 ± 0.008
γ (°)	90	90	90
Volume (Å^3)	704	704	722.8 ± 1.3
Z	2	2	2
Formula Weight	342.30	342.30	342.3
Density	1.616	1.615	1.59
Cell Temperature (K)	100	100	293
Crystal System	Monoclinic	Monoclinic	Monoclinic
Laue Group	2/m	2/m	
Space Group	P2(1)	P2(1)	$P2_1$
Number of Images	1037	166	
Frame Time/Width (sec/°)	0.75	1.95	
Length of Data Collection (min)	26	11	
Total Reflections	9004	7070	
Unique Reflections	4362	4466	
Completeness	97.3	99.0	
Redundancy	3.47	2.73	
Mean I/sigma	15.22	13.02	
R(int)	0.0618	0.0589	
R(sigma)	0.0513	0.0555	
R1	4.91%	4.76%	0.0289
wR2	14.37%	13.49%	0.0684
Number of Hours of X-ray Tube Usage for Month	21	40	
Estimated Hours of X-ray Tube Usage to Date	136.75	805.75	

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Bruker D8 Venture Single Crystal X-ray Diffractometer Calibration Form; Rev. 06-2022

Picture of Crystal

Screenshot of Autosolve with Mo Tube

Screenshot of Autosolve with Cu Tube

Notes:

☑ Checked for Computer Updates
☑ Checked to Rad Procedure is Not Falling off Wall
☑ Crystal Picking Area is Clean and has Enough Supplies

Took 45 minutes to cool to 100K from 250K @ a flow rate of 5L/min and an N2 level of 34.3%. The ramp was 360K/hr.

File path for Crystal Picture: C:\frames\guest\NUCS\Calibrations\2022\June\

File path for Cu X-ray Diffraction Data: C:\frames\guest\NUCS\Calibrations\2022\June\Cu

File path for Mo X-ray Diffraction Data: C:\frames\guest\NUCS\Calibrations\2022\June\Mo

Signature/Print	Digitally signed by Zachariah Heiden Date: 2022.06.30 16:01:06 -07'00'	Zachariah Heiden	Date	June 30, 2022

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5 Sample Preparation

5.1 The NUCS Core Facility has a crystal picking station that can be used to select crystals of non-radiative material.

5.2 If a user is inexperienced in picking crystals, this can take several hours to find a suitable crystal for X-ray diffraction.

5.3 Samples for single-crystal diffraction should be selected from unfractured, optically clear crystals. This can be determined by viewing the samples under crossed polarized light on a microscope.

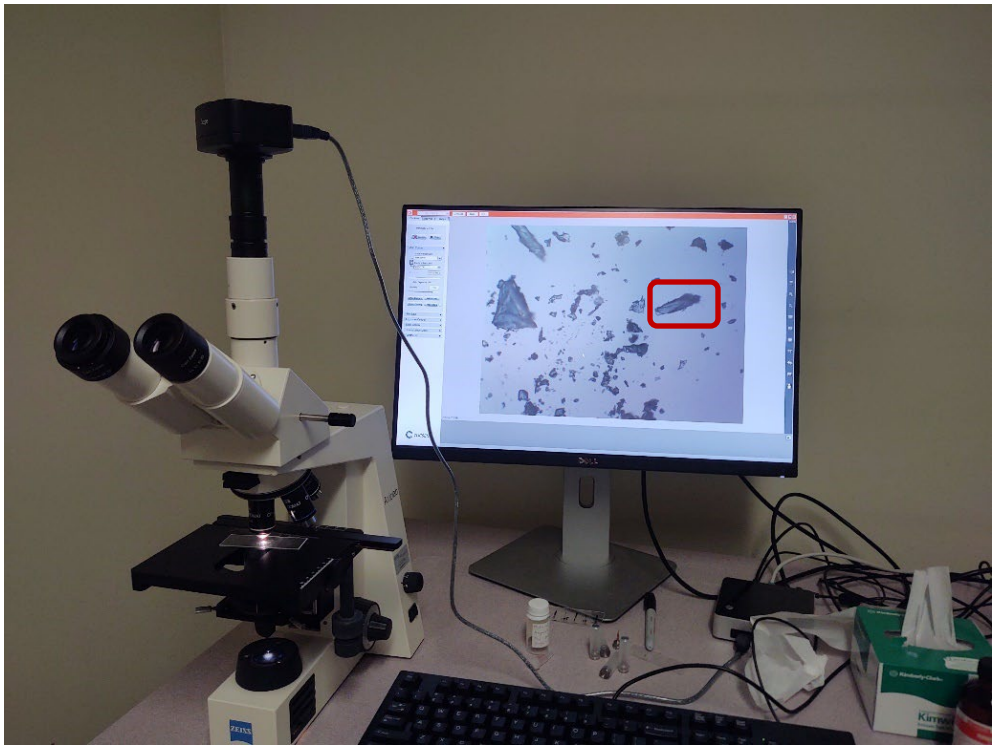
5.4 Crystals (if already dry) can be placed on a clean microscope slide for analysis and crystal picking.

5.5 If the crystals are suspected to have solvent in their lattice, a drop of immersion oil can be added to the microscope slide and the crystal can be placed into the oil to prevent desolvation and collapse of the crystalline lattice.

5.6 Crystal mounted on the single crystal X-ray diffractometer should be between 30 and 300 microns, with ideal crystals averaging 150-250 microns in size. Crystals can be broken off a larger sample and the best fragment selected, but it is preferred to not cut or break a larger crystal, if possible.

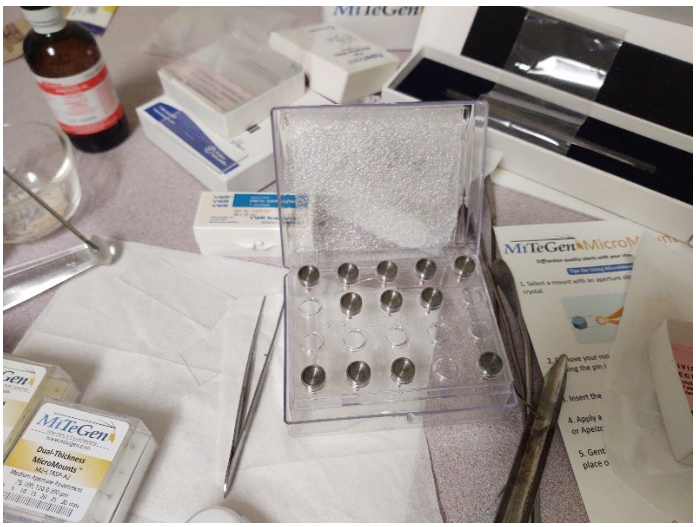
5.7 When crystal picking, to get an idea of the size of a crystal a calibration slide is available (highlighted in red).

5.8 When a suitable crystal has been identified, a needle or razor blade can be used to move the other crystals away for the chosen crystal to make it easier to pick up with the cryo loop. This should be done prior to trying to scoop up the crystal. If the other crystals are not moved away, the cryo loop will also pick them up, resulting in conflicting X-ray patterns and data that will not be usable.

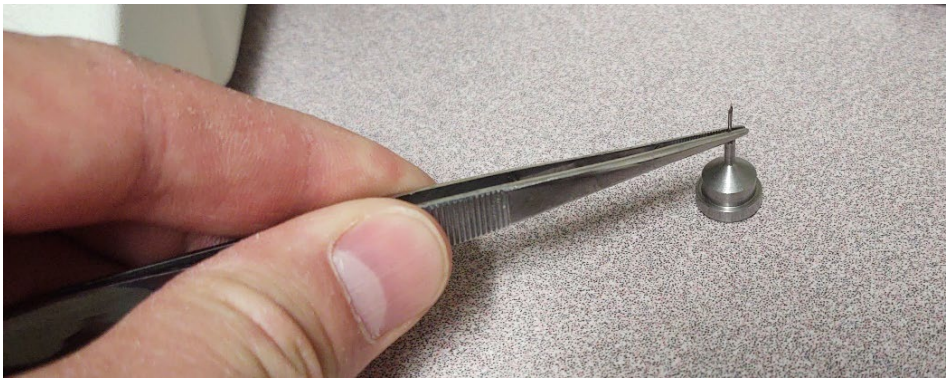


5.9 Once a crystal has been identified, a MicroMount (cryo loop) that is of similar size to the crystal is chosen. It should be slightly smaller than the crystal so that the crystal sits on the loop. Loops with diameters between 10 and 200 µm are available.

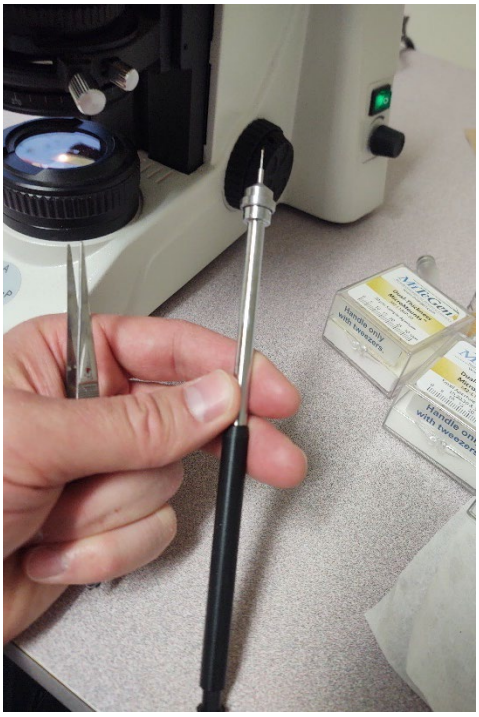
5.10 Before removing the cryo loop from the holder, pick a goniometer base out of the holder with your hand and place it on the table.



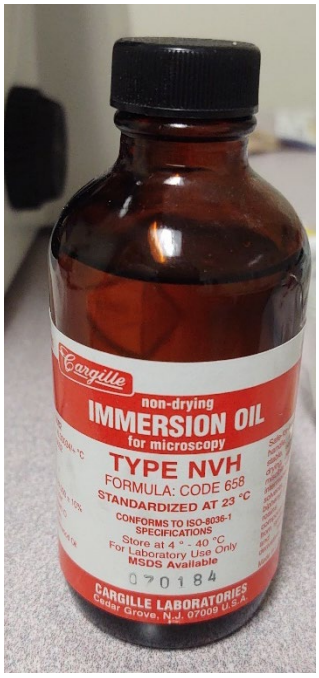
5.11 Cryo loops are only handled with a tweezers (do not touch with your hand). Use a tweezers and remove the cryo loop from the cryo loop container and insert it into the goniometer base until it stops.



5.12 Place the goniometer base on the end of the magnetic tool to help with the mounting of the crystal.



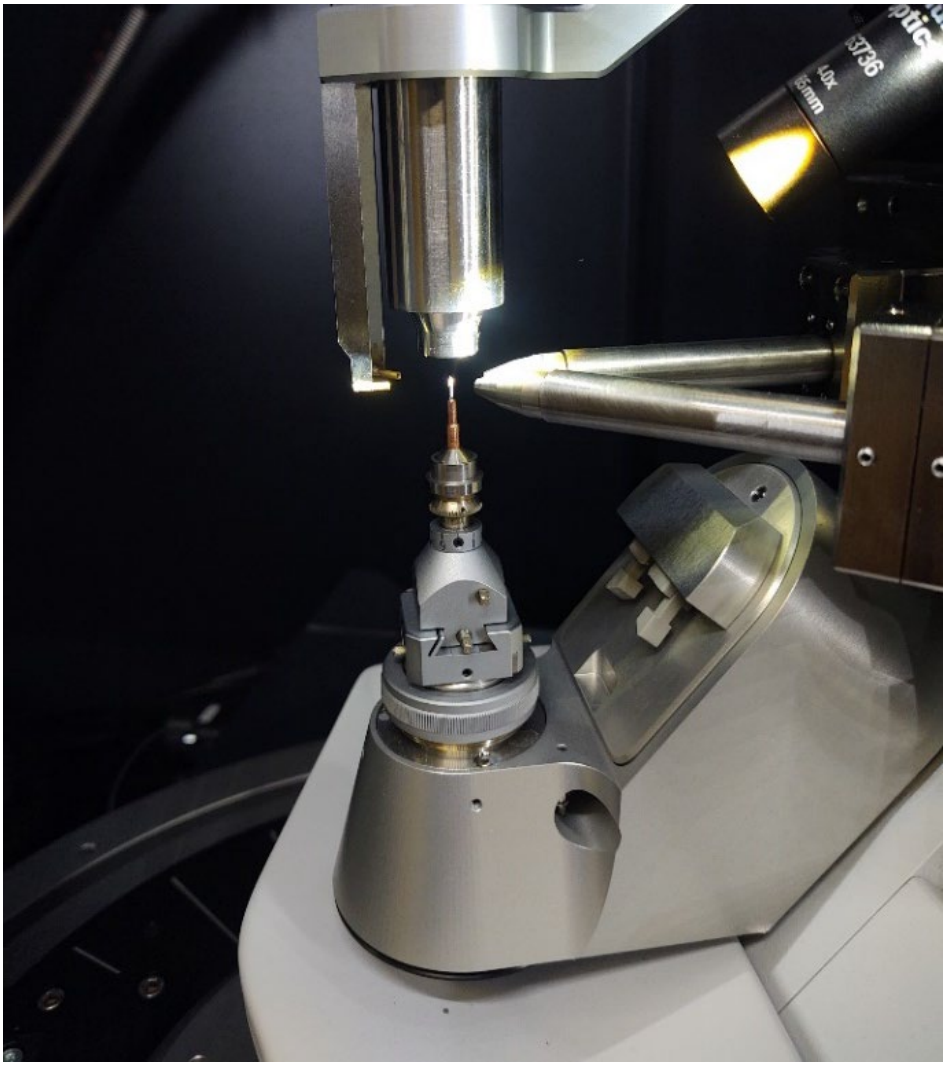
5.13 Dip the tip of the cyro loop into the cryo oil, immersion oil, or Paratone. The oil helps the crystal stay on the loop. If the crystals have solvent in their lattice, the crystals can be placed in the immersion oil prior to their analysis on the microscope.



5.14 Use the microscope to find the crystal and scoop up the selected crystal onto the cryo loop.

5.15 Make sure that only the selected crystal is present on the loop. If multiple crystals are mounted a conflicting X-ray pattern will result and the data will be unusable. If this occurs, use a Kimwipe to wipe off the crystal and oil and repeat Steps 5.13 & 5.14.

5.16 When the crystal is on the cryo loop, the goniometer base is ready to be placed into the instrument (see Steps 6.17 – 6.20 for how to mount the goniometer base onto the goniometer) and the goniometer should look like the picture below when the goniometer head has been placed on the goniometer.



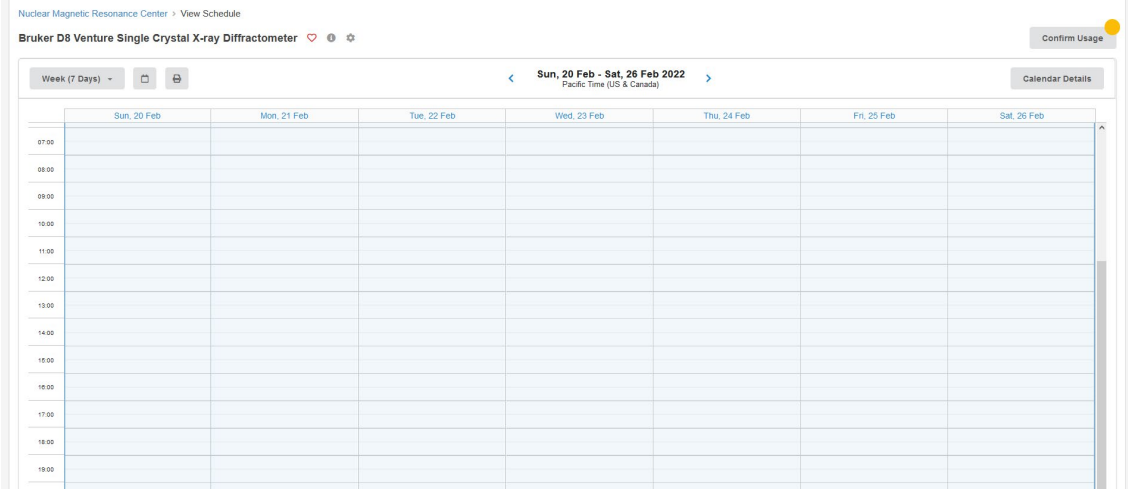
5.17 See Section 6 for information on data collection.

6 Operation of the Bruker D8 Venture Single Crystal X-ray Diffractometer

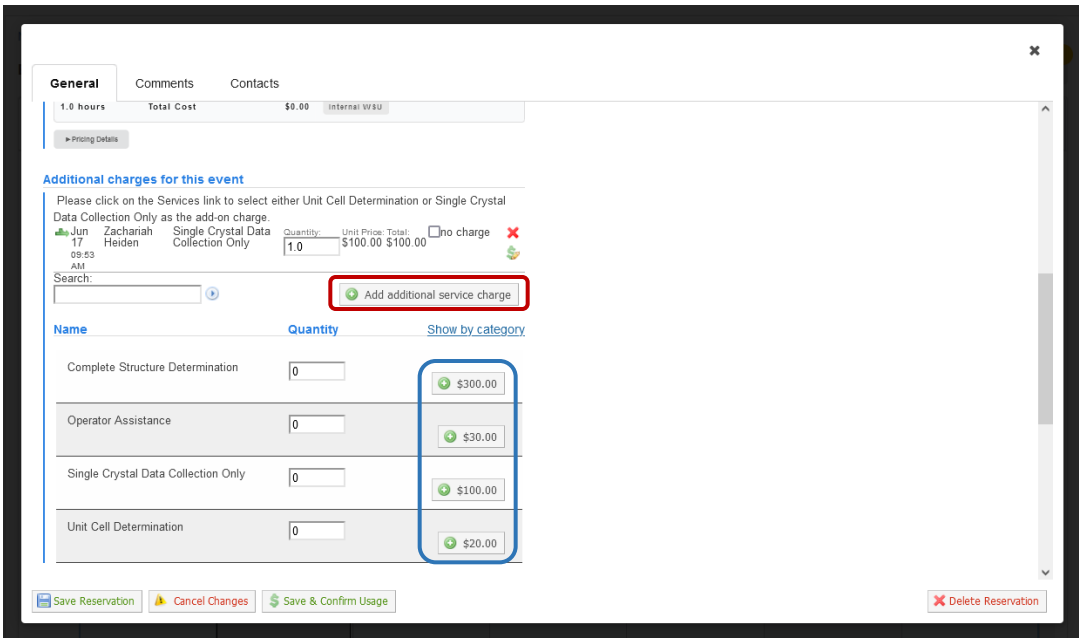
This section is to be done after a monthly calibration has been performed.

Materials: Single Crystal X-ray Diffraction Sample mounted on a cryo loop, Bruker D8 Venture Single Crystal X-ray Diffractometer

6.1 Instrument time on the Bruker D8 Venture Single Crystal X-ray diffractometer are made on iLab (https://wsu.corefacilities.org/), at least one hour prior to the user's instrument time. Reservations are based on service, not instrument time and are reserved in 15 minute increments, with a minimum reservation time of one hour. Please reserve the desired instrument time on iLab. A reservation can be made by clicking on an available time and saving the reservation. If you have questions regarding booking instrument time, please ask the NUCS Core Facility Staff.



6.2 When making your reservation, make sure to click on the correct additional charges for the reservation. The add-on charges correspond to the service received. The add-on charges are: single crystal data collection only (use this charge if you are collecting a full data set on a crystal), unit cell check (use this charge if you are only collecting data on a unit cell, and complete structure determination (this option should only be used for NUCS Core Facilty Staff if data collection and asolved structure is supplied to the customer). The default selection is a single crystal data colleciton only. To add additional charges or replace the single datra collection only with a different charge, click on the add additional service charge button (highlighted in red). Change the quantity and click on the +dollar amount button (highlighted in blue) to add the new charge. Please make sure the quantities are correct before saving your reservation so that you are not overcharged for your reservation.

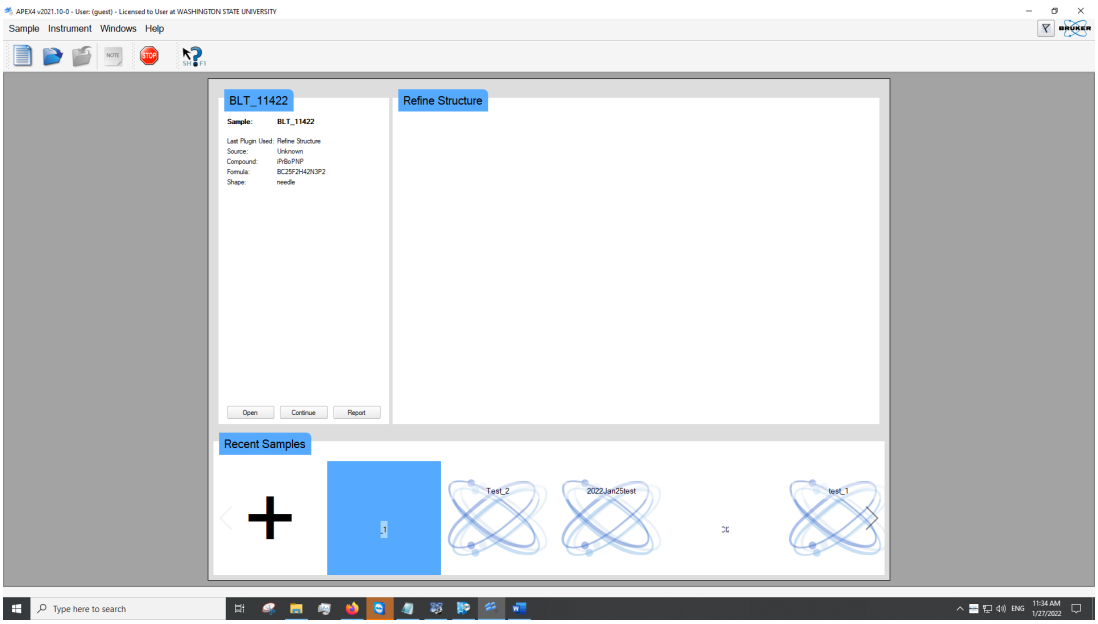


6.3 When you show up for your reserved instrument time, start by checking to see if the Bruker D8 Venture single crystal X-ray diffractometer is on. The default state is to have the system powered up with the lights on the side of the instrument indicating that the X-ray are on. Although the lights on the instrument suggest that the X-rays are on, they are not actually on. The X-ray tubes are warmed up and in a standby state, which allows them to be initiated on demand. Both X-ray tubes should be ready to use.

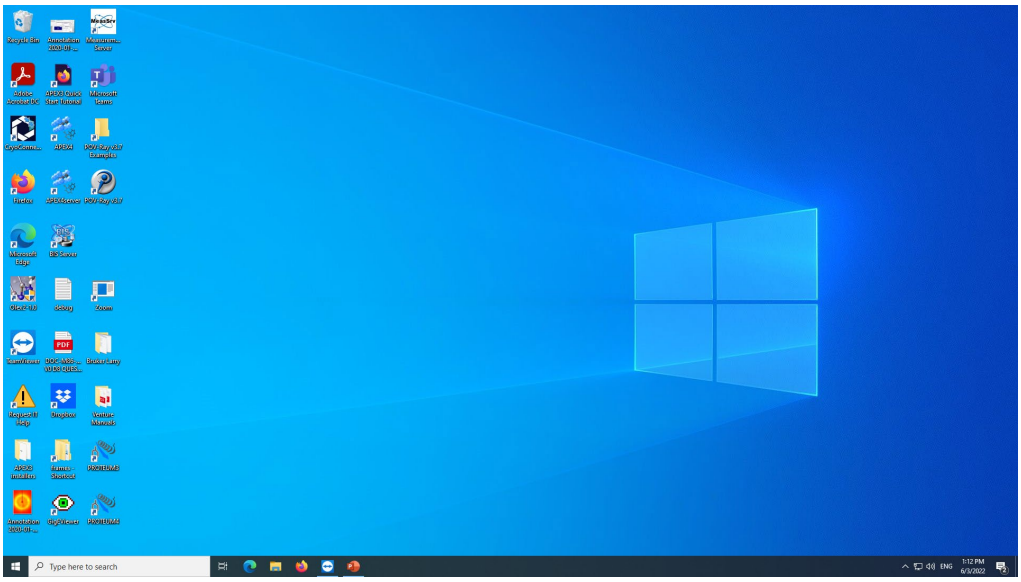
Picture of the D8 Venture when not in use.

6.4 If you want data at 100K, start the cryostream now (see Section 7) and wait 15-30 minutes to cool down to 100K before putting on the crystal. If you have not already picked a crystal, this is a good time to pick a crystal.

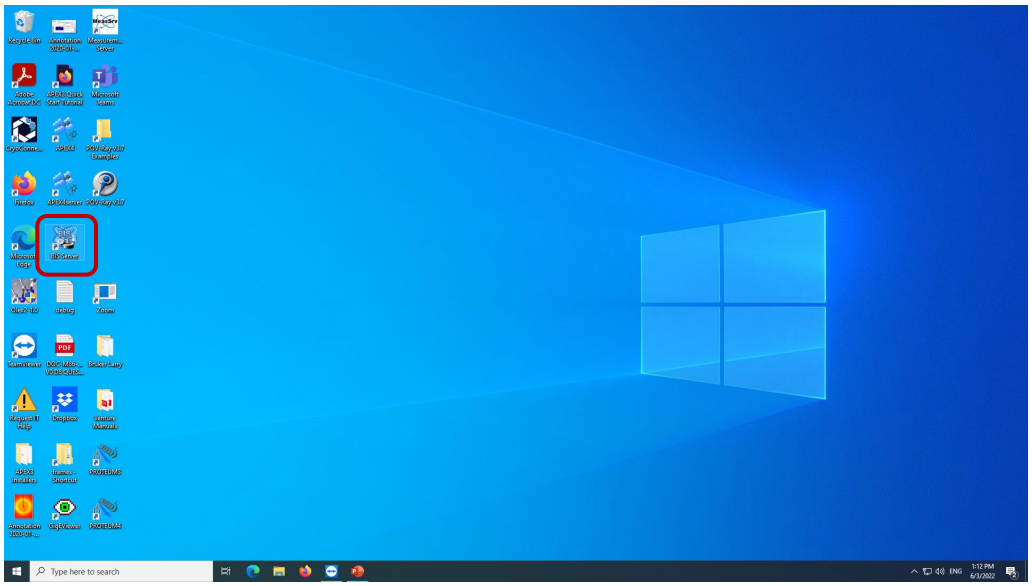
6.5 When you sit down at the diffractometer, Apex 4 should be already open. If so, it will look like the following image, and you can skip to Step 6.11.



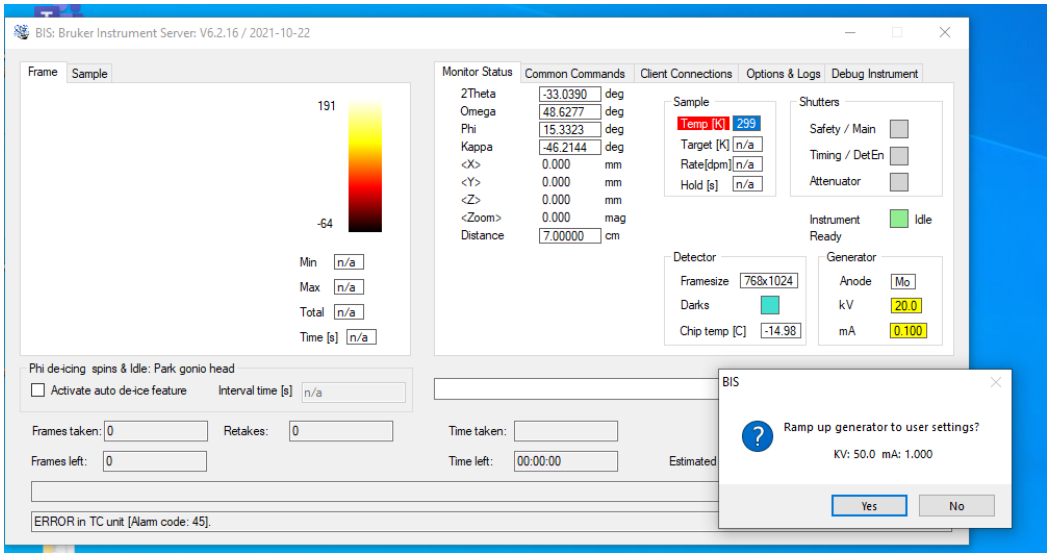
6.6 If the screen looks like the following, Apex 4 and the instrument server will need to be opened.



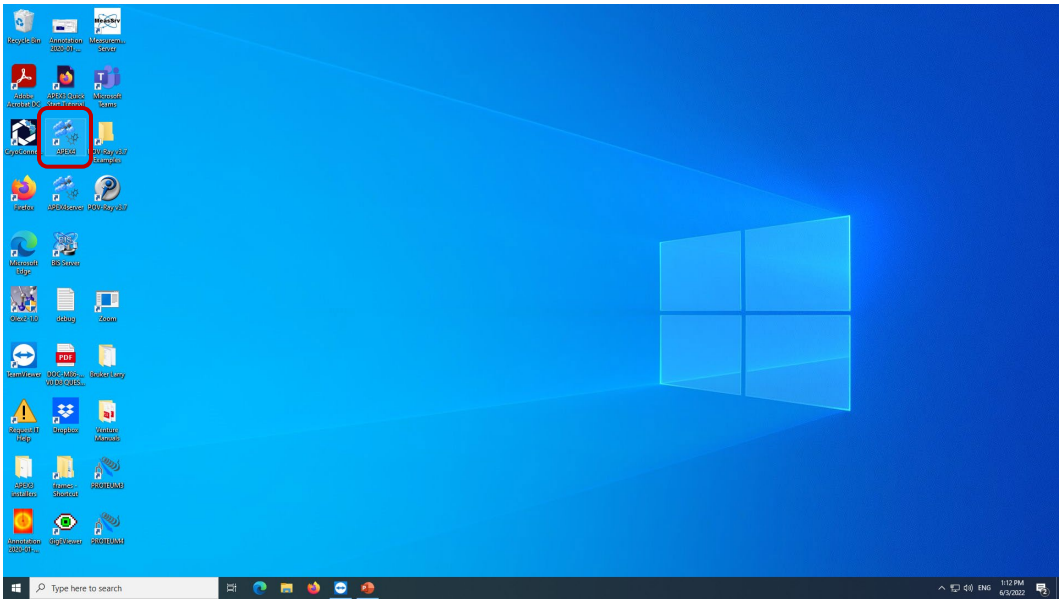
6.7 If no programs are open, you will need to open the BIS Server first to allow for the computer to connect to the instrument. Double click on the BIS Server icon on the desktop (highlighted in red).



6.8 When the BIS Server is running you will see the following window and be asked to ramp up the generator for the X-ray tubes. Click the Yes button to provide power to the X-ray tubes.

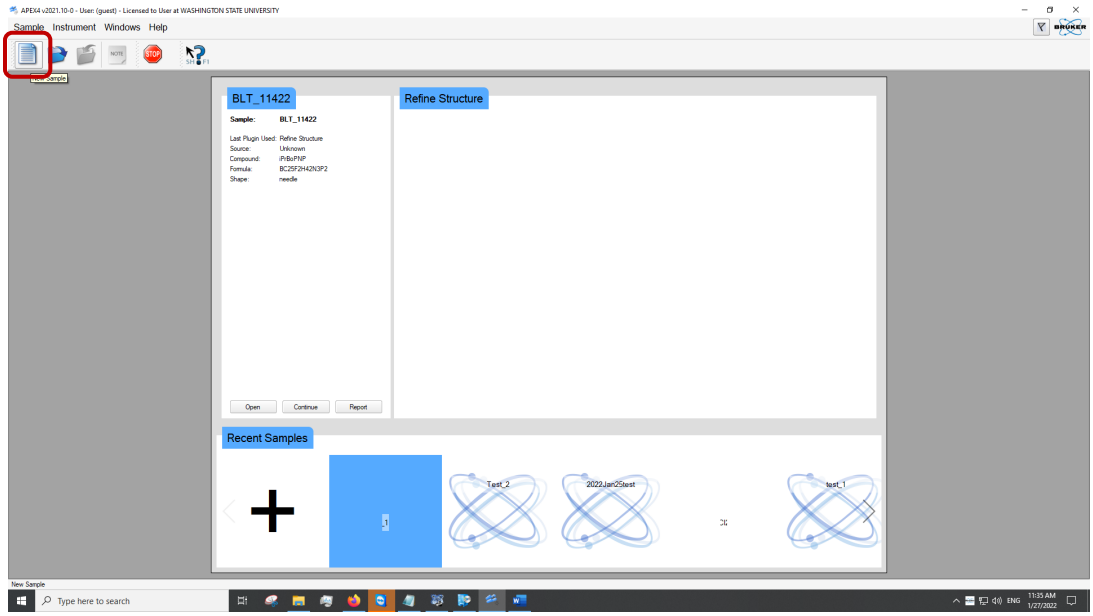


6.9 After the BIS Server is opened, you need to open Apex 4. The icon to open Apex 4 can be found on the Desktop and is highlighted in red.

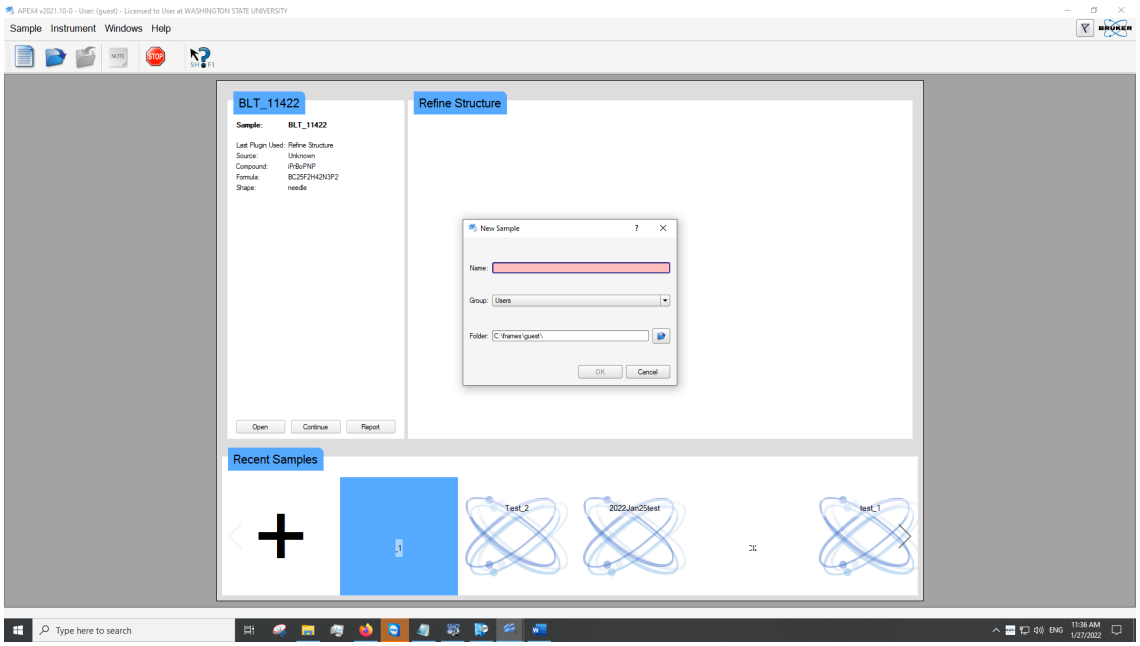


6.10 If a password is requested when Apex 4 opens, the group is guest and the password is guest.

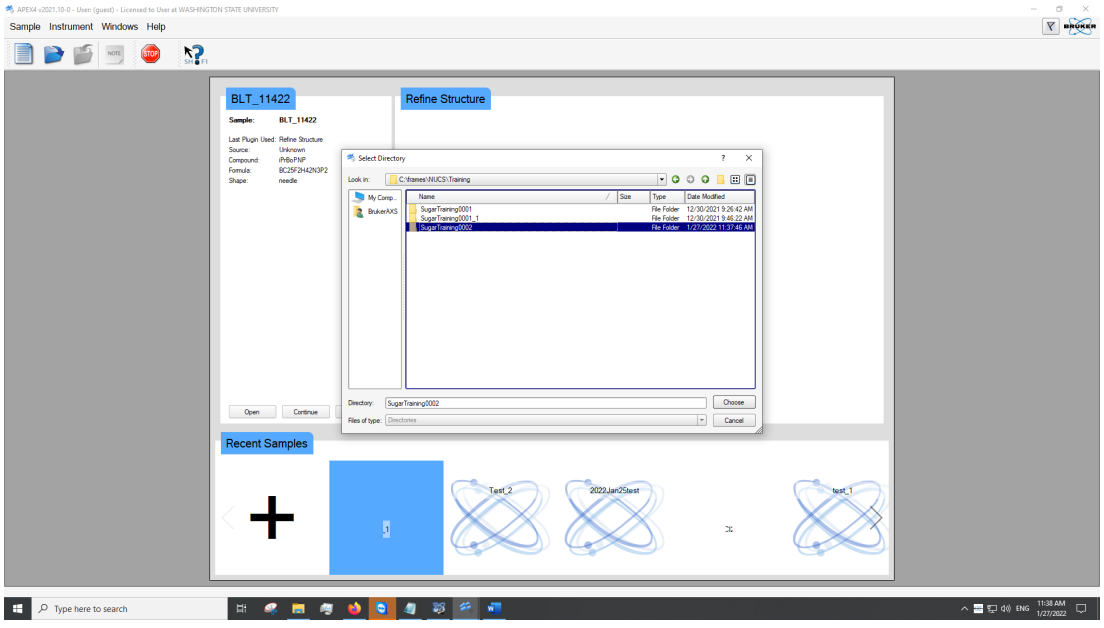
6.11 To get started on the data collection, click on the New Sample Icon (highlighted in red).



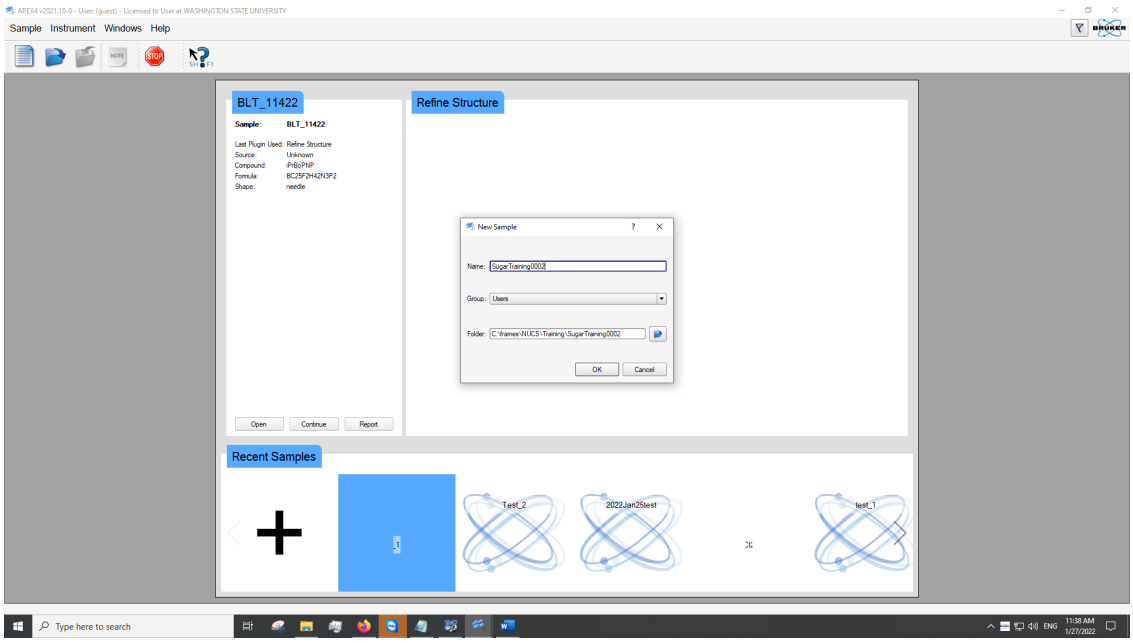
6.12 Fill in the information on the New Sample



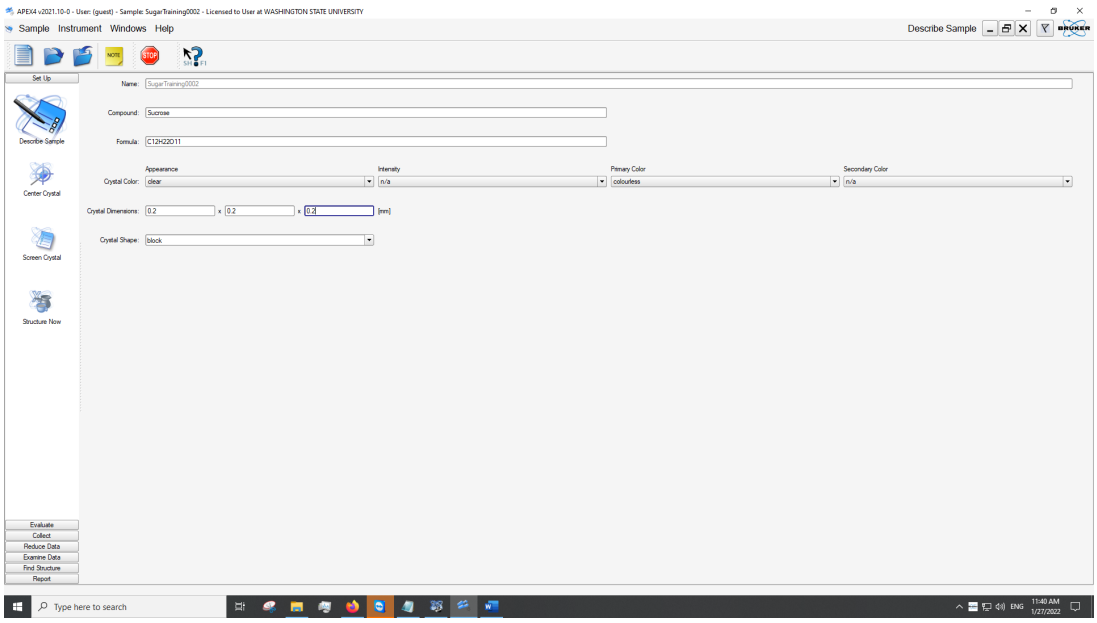
6.13 To have the data automatically transfer, make sure that your data is in the following location: C:\frames\guest\ResearchGroup\UserLastName. For example, if John Doe was in the Heiden research group, the file location should be: C:\frames\guest\Heiden\Doe.



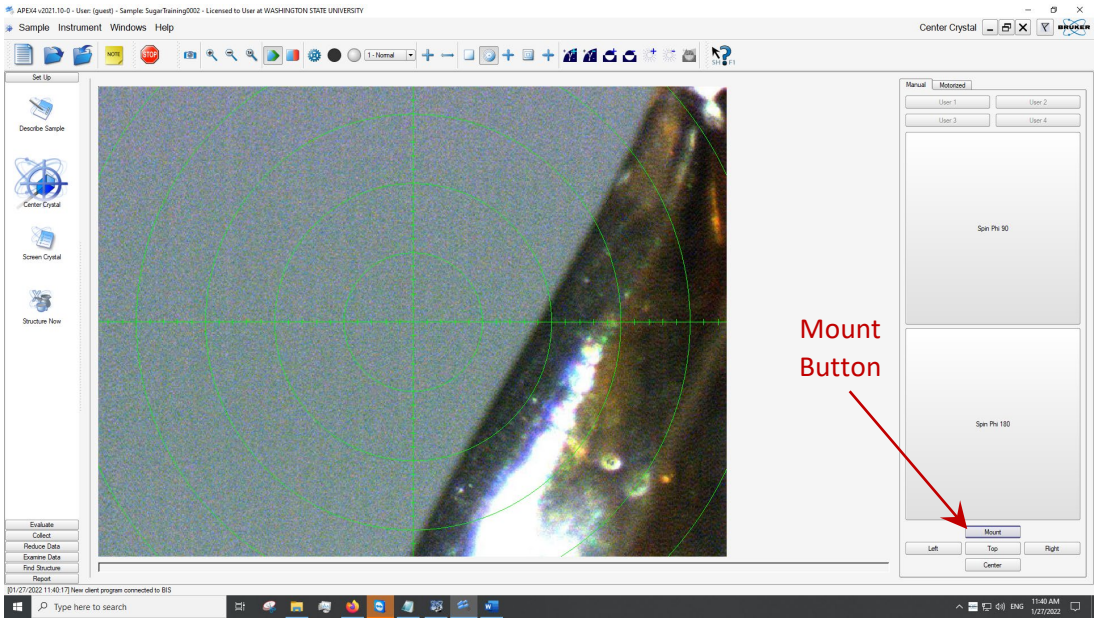
6.14 For a training session, create a folder with the name: SugarTraining_LastName. For example, John Doe would create a folder with the name: SugarTraining_Doe.



6.15 To get ready for a data collection, click on describe sample and fill out the information. The crystal dimensions can be measured in the center crystal stage (Step 6.16) and entered by clicking back on the Describe Sample tab.

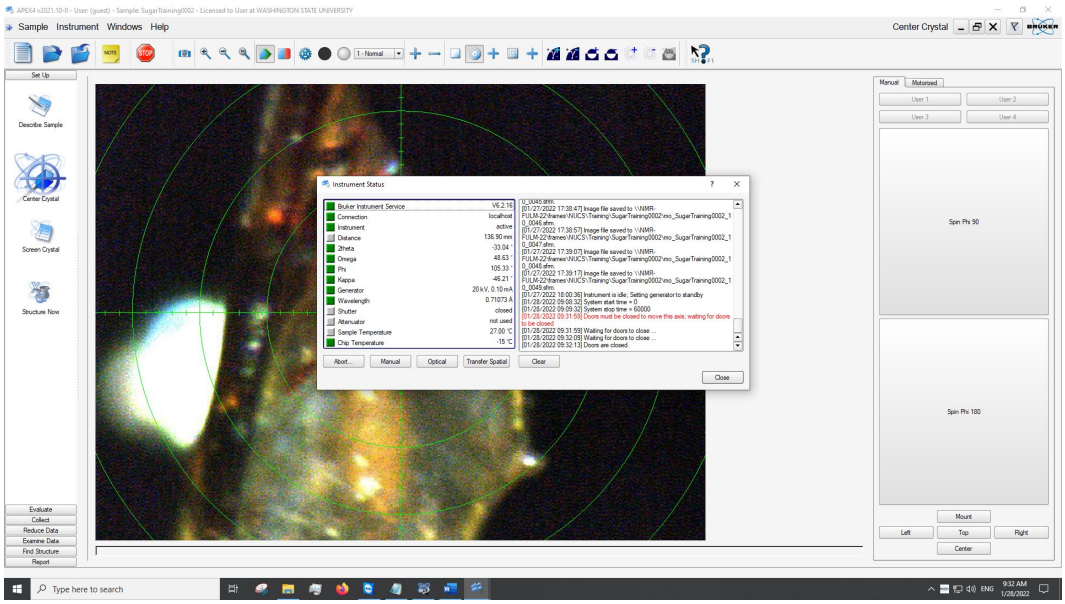


6.16 Click on the Center Crystal icon and click on the Mount button to move the instrument to the crystal mounting position.

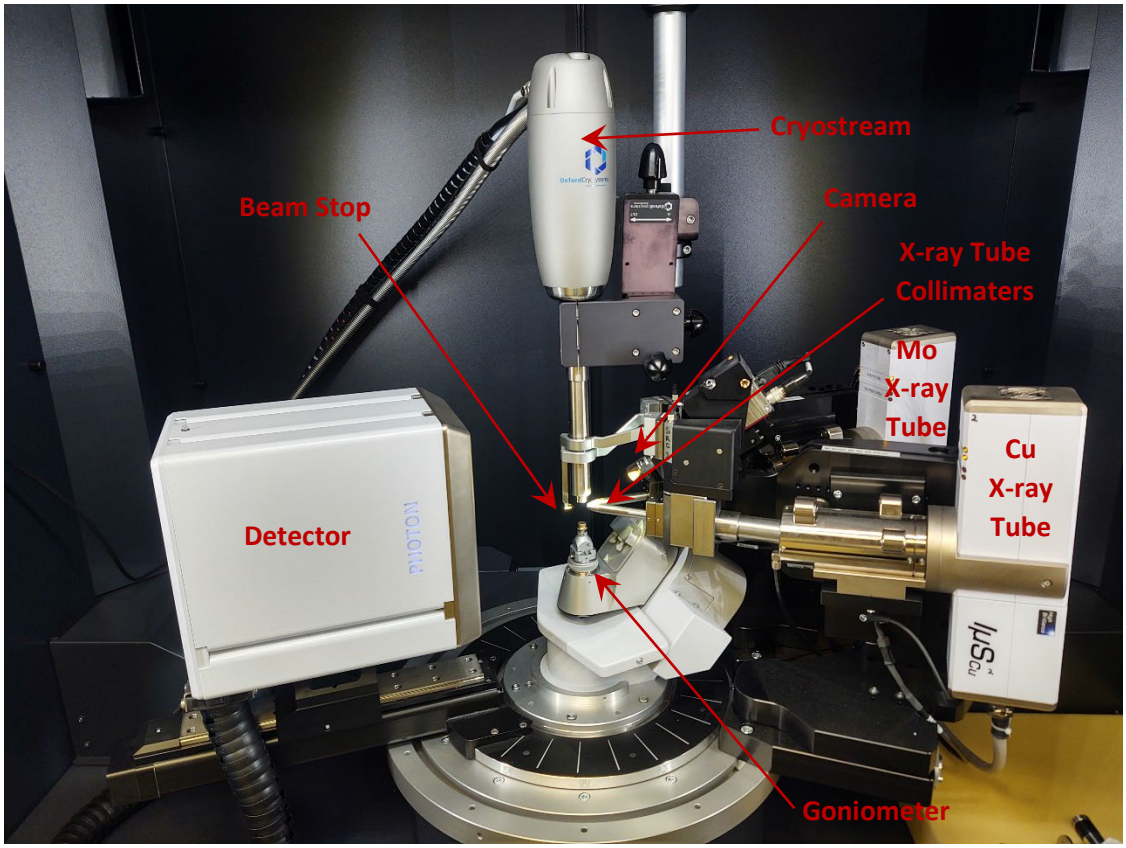


6.17 open the doors by pushing on the button on the inside of the door handle when the detector has stopped moving. When the interlocks release, the doors will be able to slide to the left or to the right.

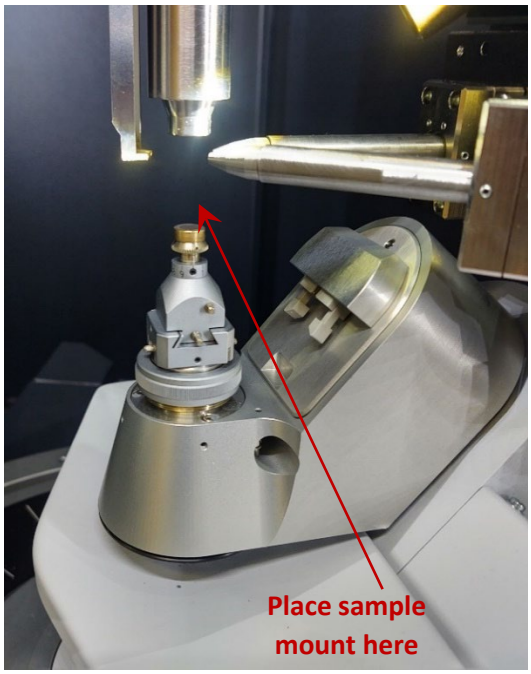
6.18 If you try to open the doors while the detector is moving then you will receive the following error message.

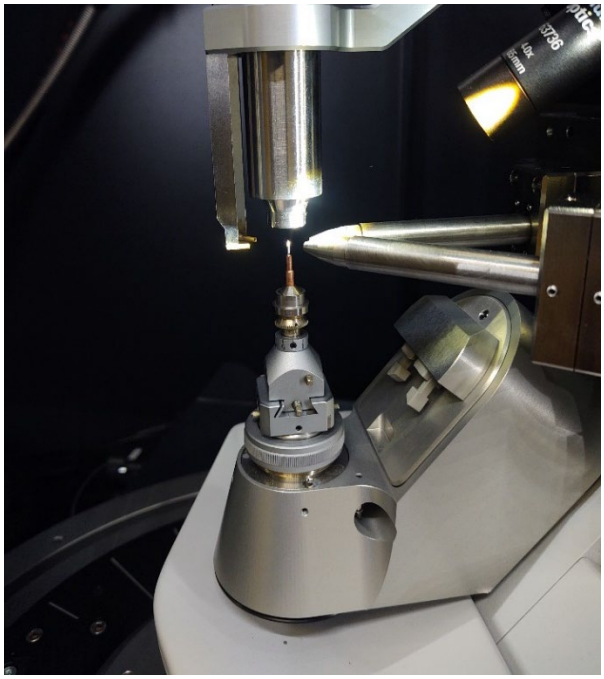

Sample Instrument Windows Help
Center Crystal
Set Up
Describe Sample
Center Crystal
Screen Crystal
Structure Now
Evaluate
Collect
Reduce Data
Examine Data
Find Structure
Report
Instrument Status
Bruker Instrument Service
Connection
Instrument
Distance
2theta
Omega
Phi
Kappa
Generator
Wavelength
Shutter
Attenuator
Sample Temperature
Chip Temperature
Abort...
Manual
Optical
Transfer Spatial
Clear
Close
Manual
Motorized
Spin Phi 90
Spin Phi 180
Mount
Left
Top
Right
Center
Type here to search

6.19 The diffractometer will look like the setup in the following picture. The individual components are labeled.



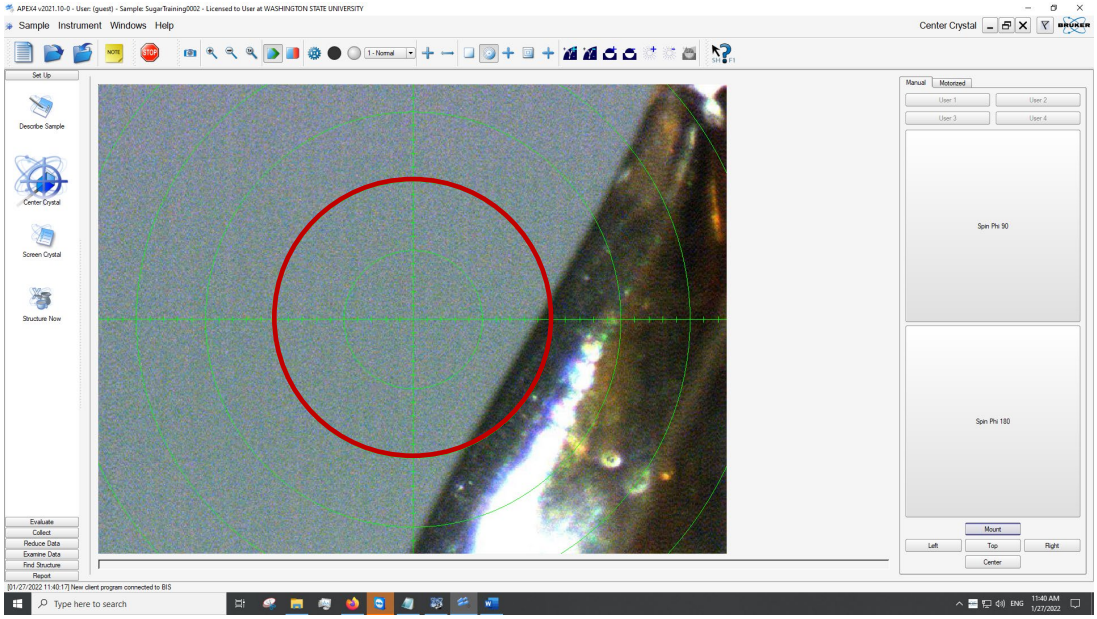
6.20 The mounted crystal is then placed on the goniometer head, as seen in the pictures below. Note that the mount is magnetic and it clicks on top of the goniometer head.



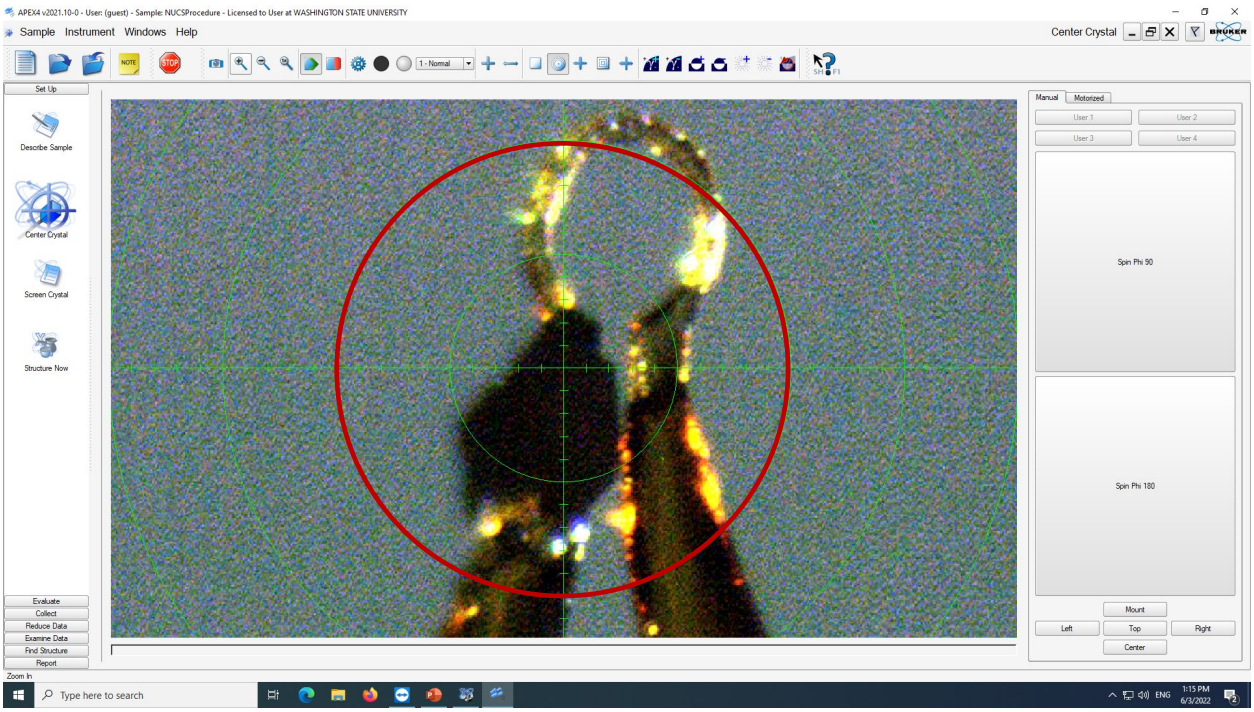


6.21 When looking at the camera image, the crystal will probably be on the side of the screen and need to be moved into the target.

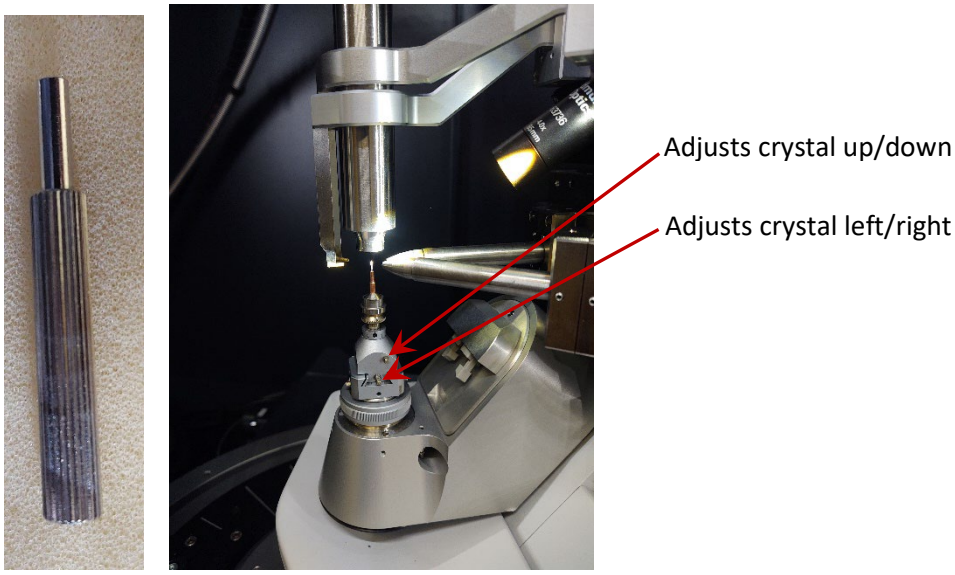
6.22 Click on the mount button and put on your crystal, make sure the temperature is already 100K, unless want a room temperature data set.



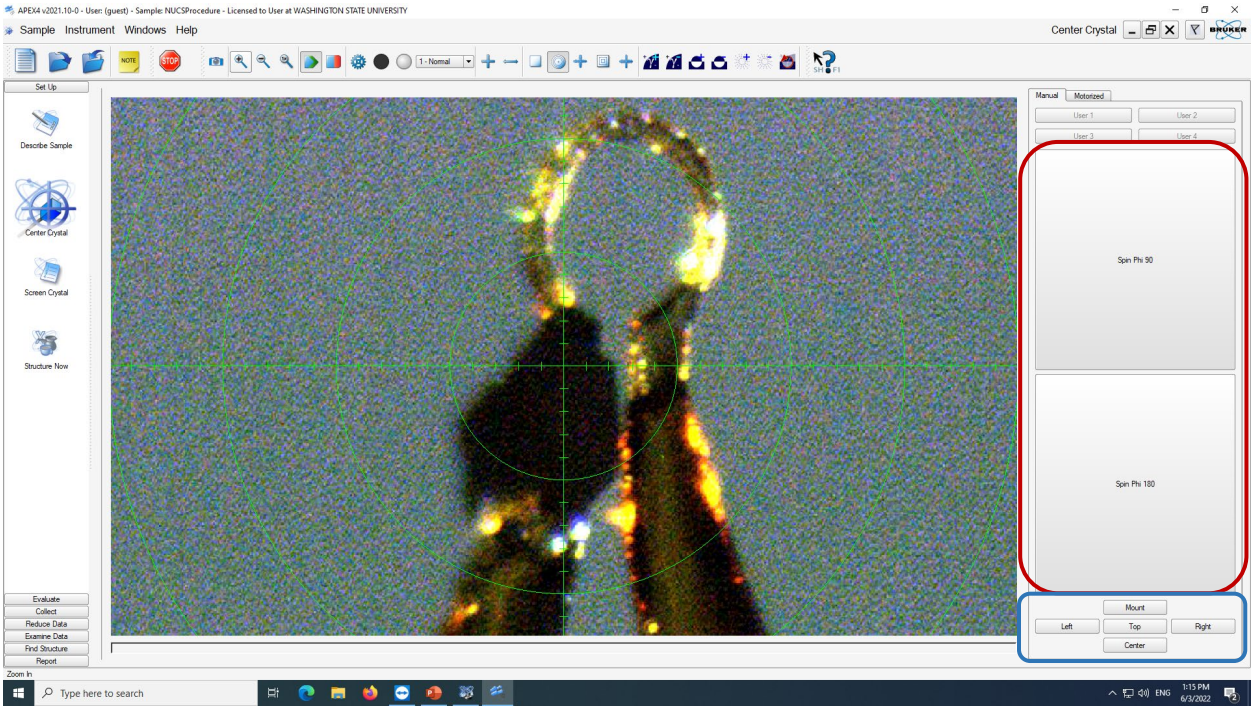
6.23 You will want to move the crystal into the center of the crosshairs to guarantee that the crystal is always hit with X-rays during the experiment.



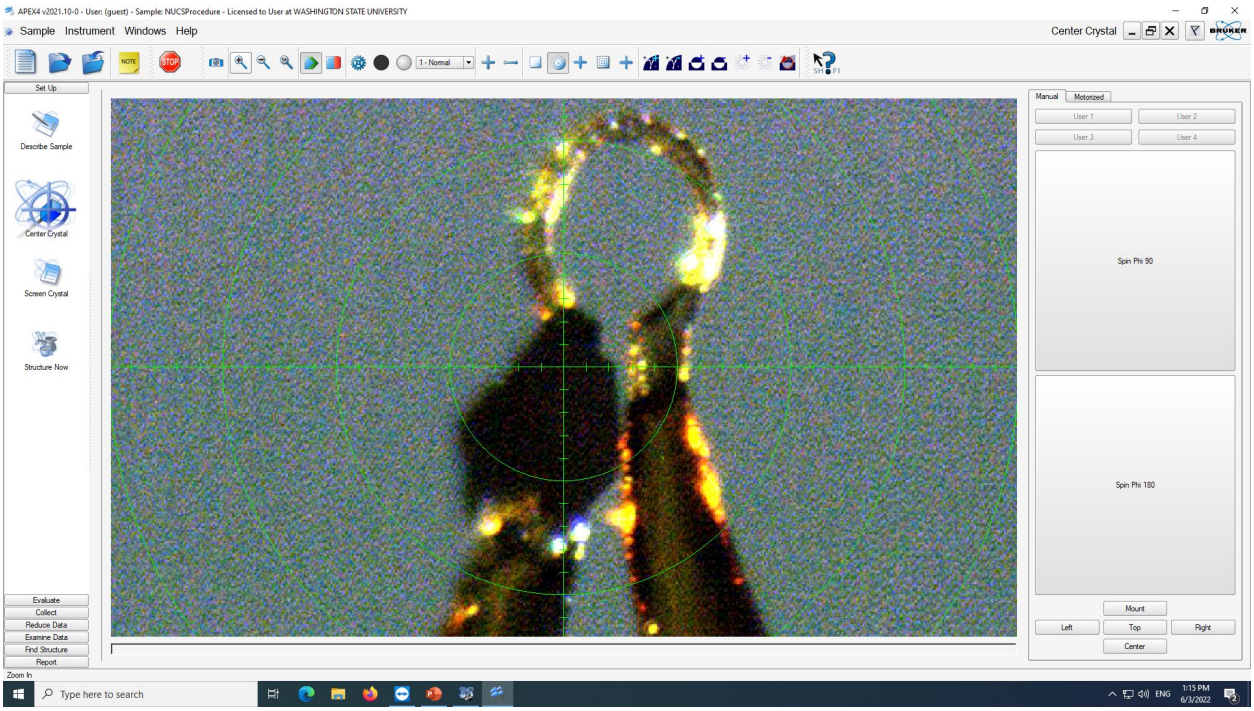
6.24 To center the crystal, use the tool provided and adjust the square screws that are pointing towards you to move the crystal in the crosshairs. Only adjust the screws that are pointing towards you.



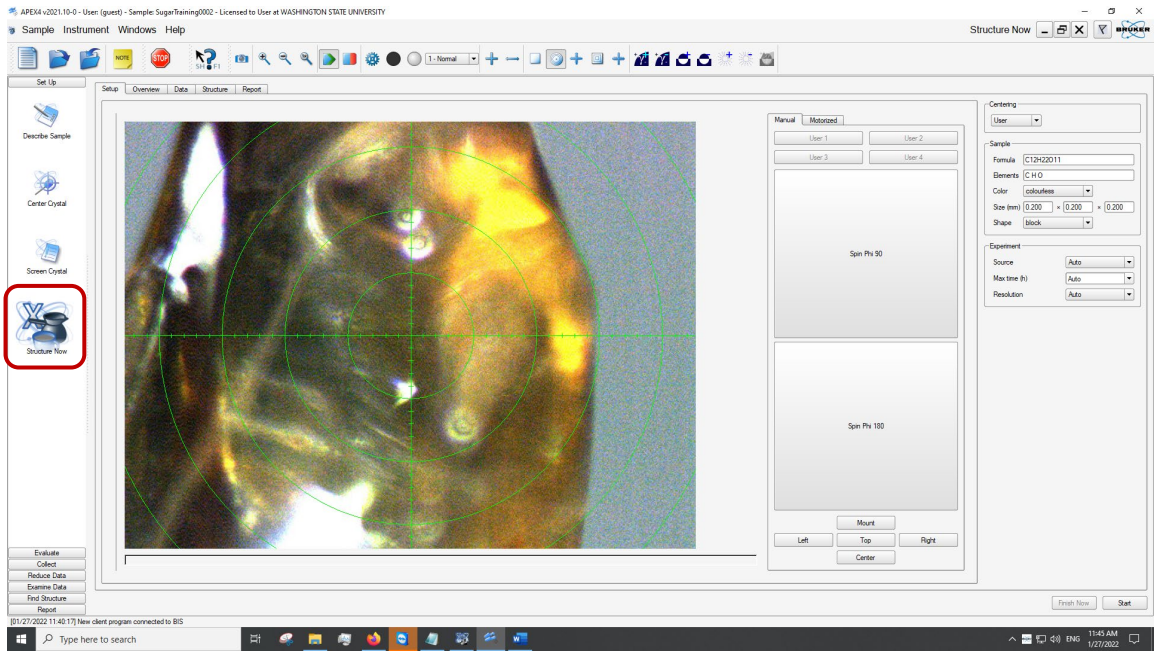
6.25 As you try to center your crystal, the rotation of the crystal that occurs by clicking on the large Phi buttons (highlighted in red) can be completed with the doors open. The doors need to be closed for the operations that occur from the smaller buttons (highlighted in blue)



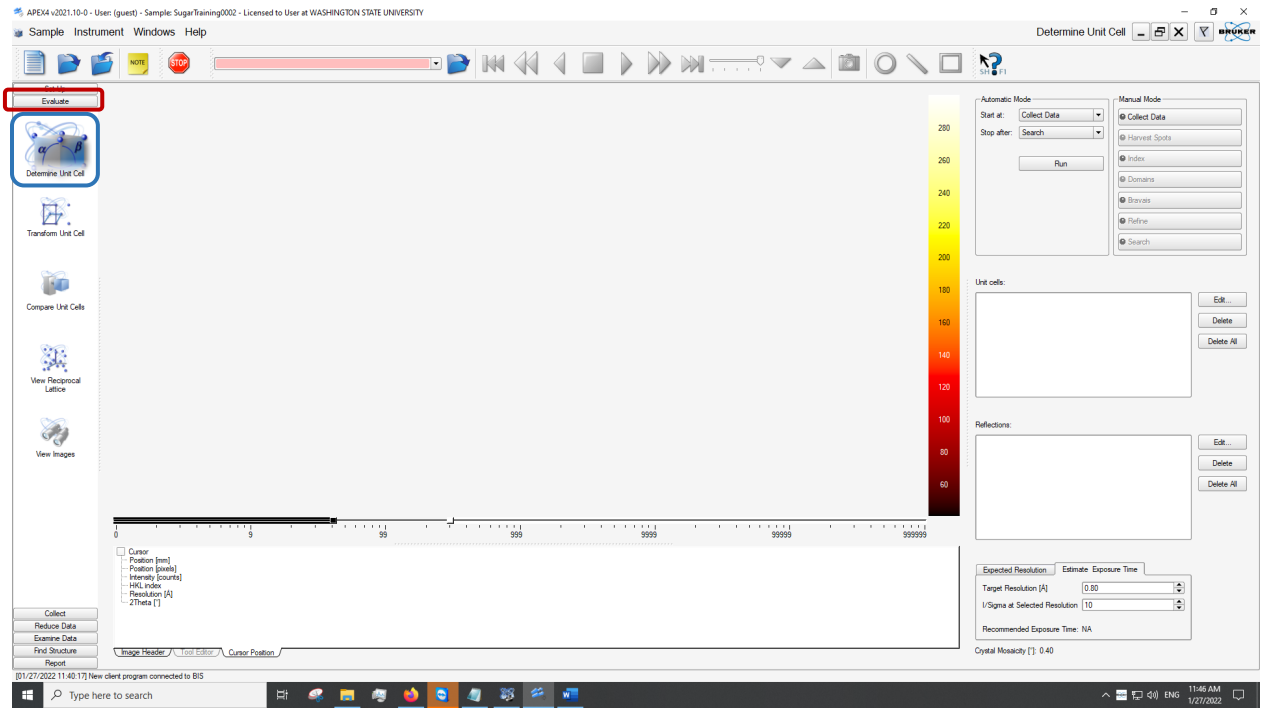
6.26 When it is in the center of the crosshairs with multiple movements in phi, then you are ready to collect a unit cell.



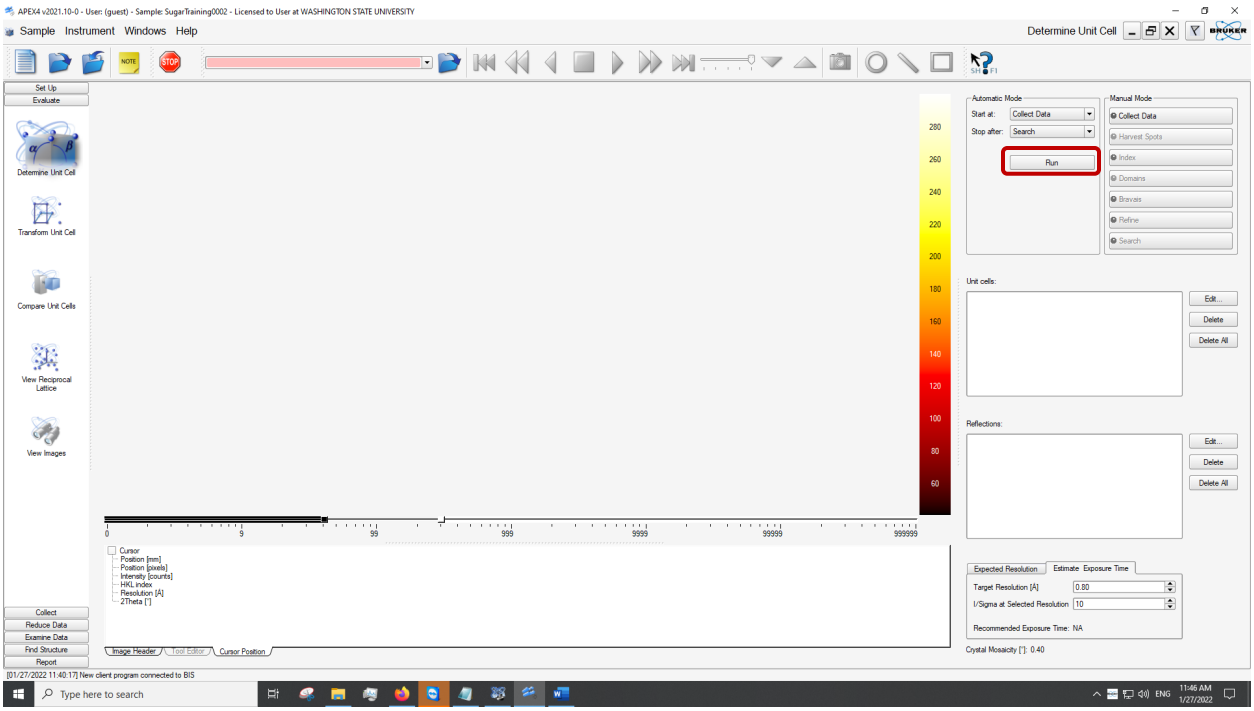
6.27 There is also the structure now option (highlighted in red), which has everything automated, but the use of this software often does not give satisfactory results. Note: the crystal in the picture is too big, and a smaller one should be chosen.



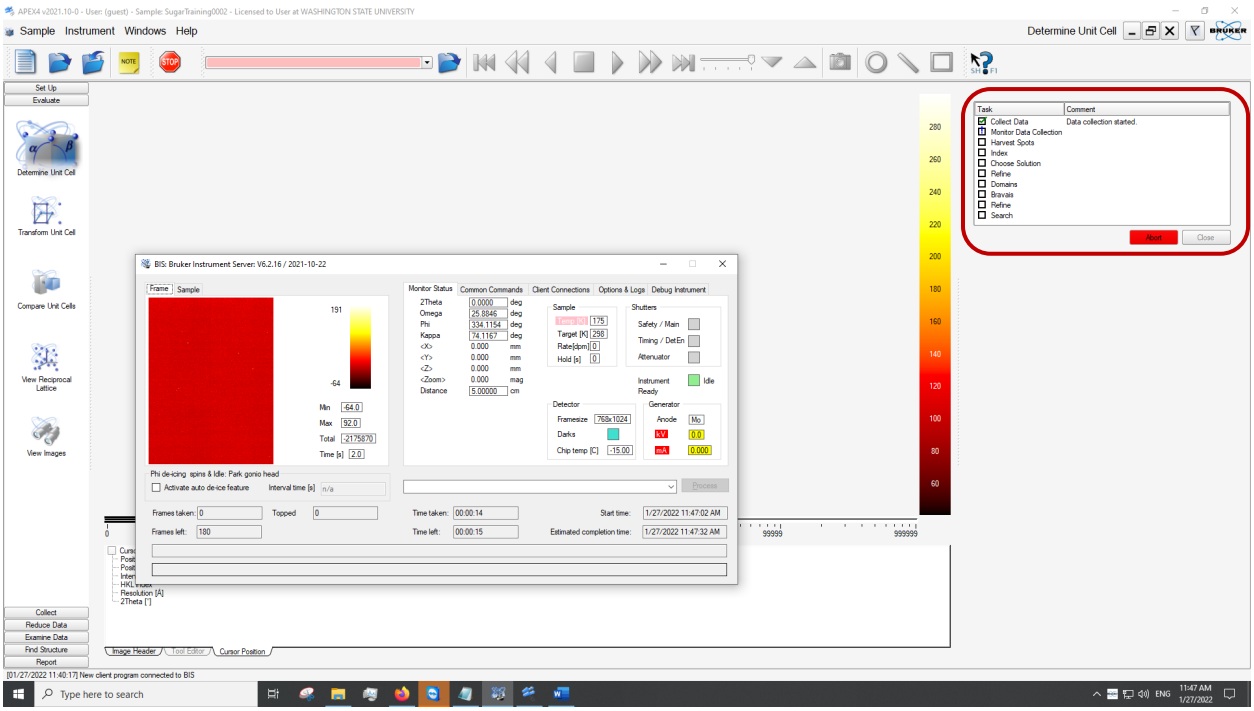
6.28 Then click on the Evaluate button (highlighted in red), followed by the Determine Unit Cell icon (highlighted in blue), which will result in the following window. Skip to Step 6.29 for using the automatic mode for unit cell data collection. If the manual mode is preferred, skip to Step 6.35.



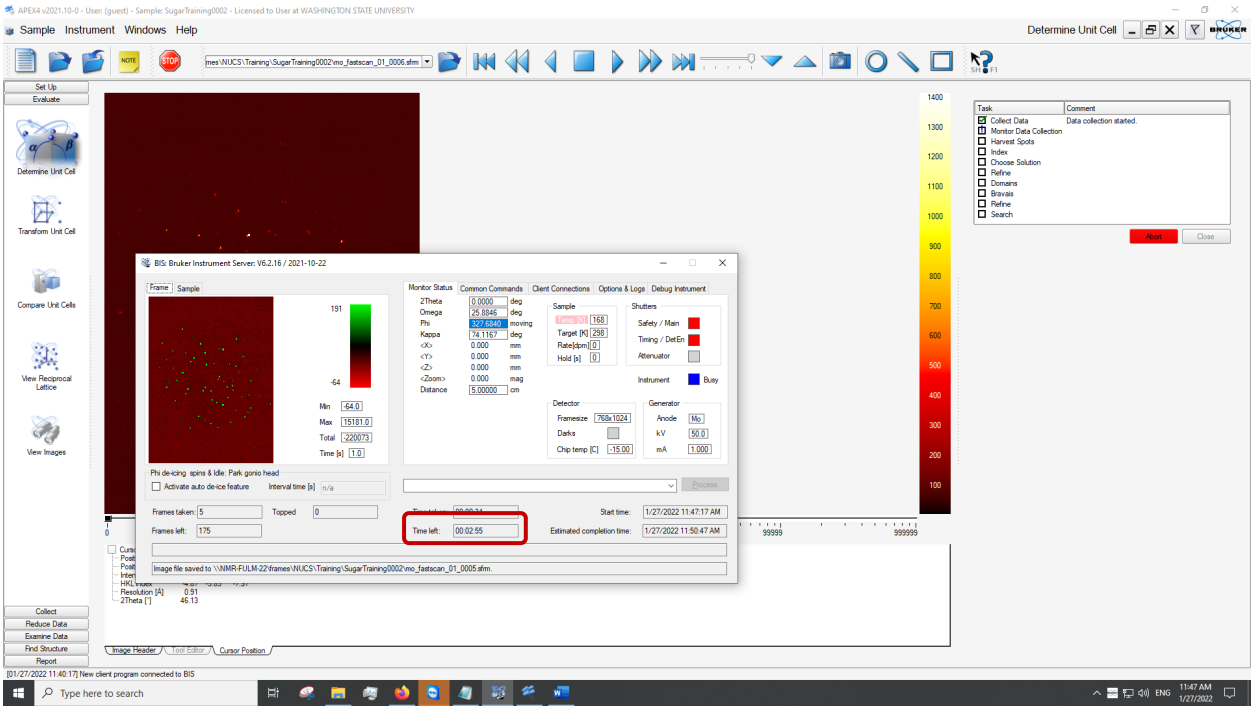
6.29 To collect a unit cell through the automatic mode, which uses the molybdenum X-ray tube, Click on the Run button (highlighted in red) under the automatic mode.



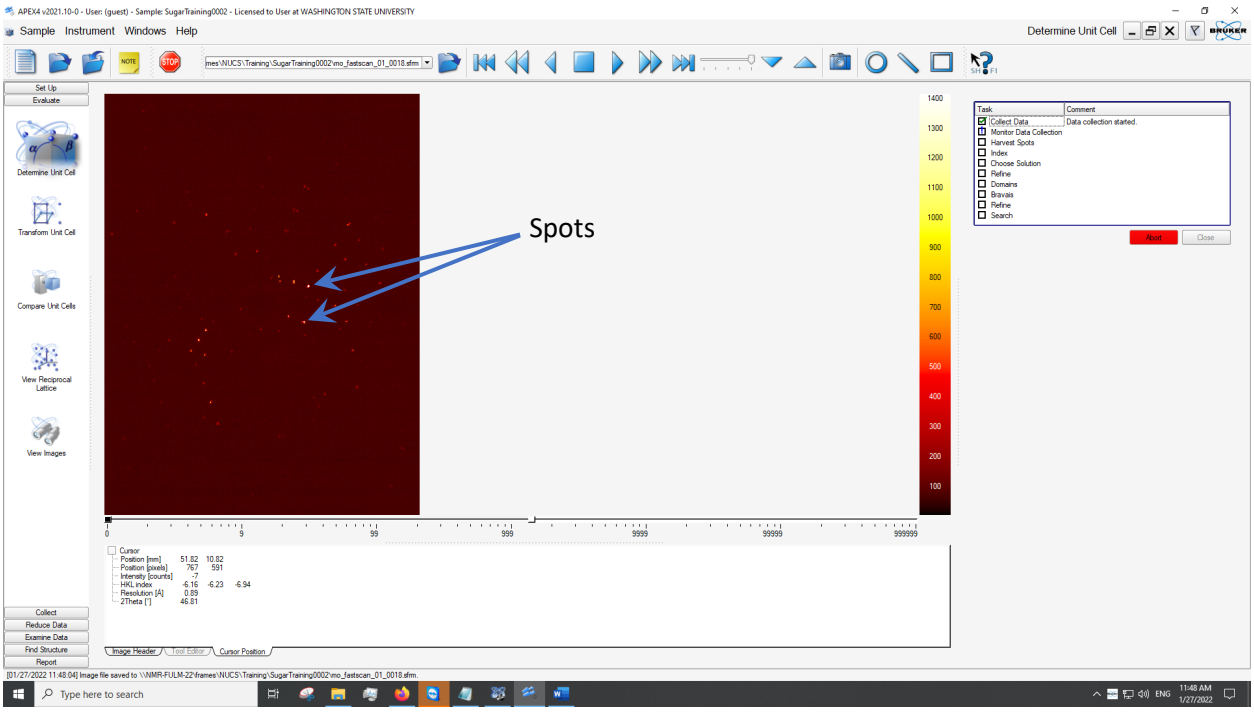
6.30 The following window will result at the beginning of the unit cell data collection. The progress of the unit cell data collection tasks can be seen in the region highlighted in red.



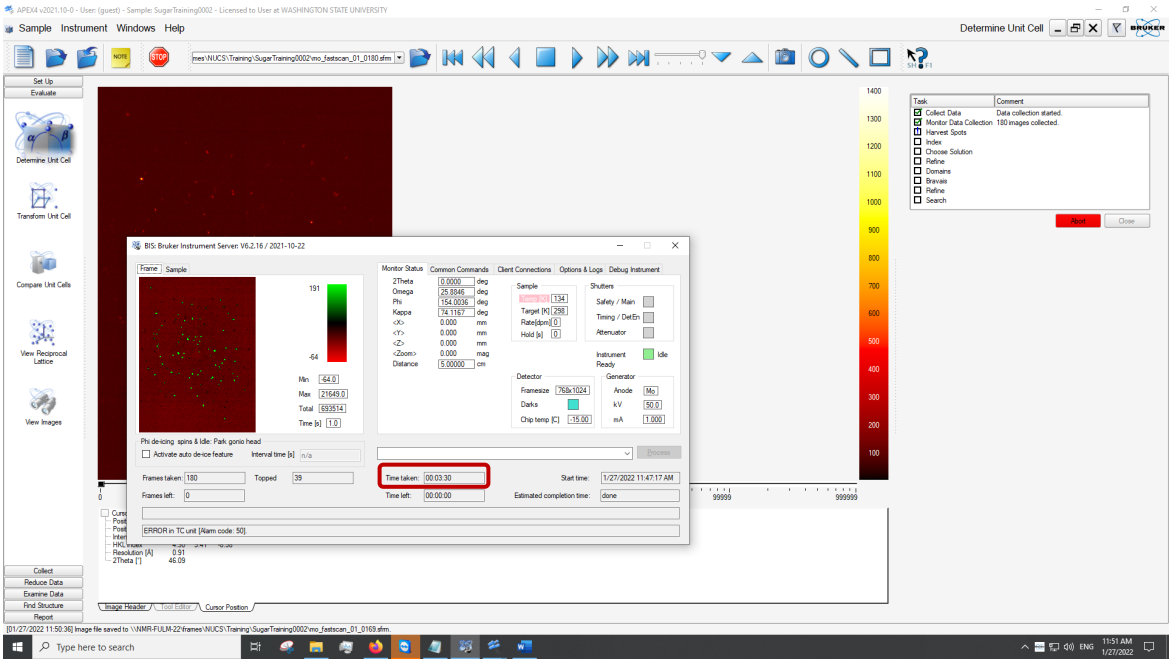
6.31 Clicking on the BIS Measurement Server window will show the time left on the unit cell data collection (highlighted in red). The unit cell data collection should take about 3 minutes.



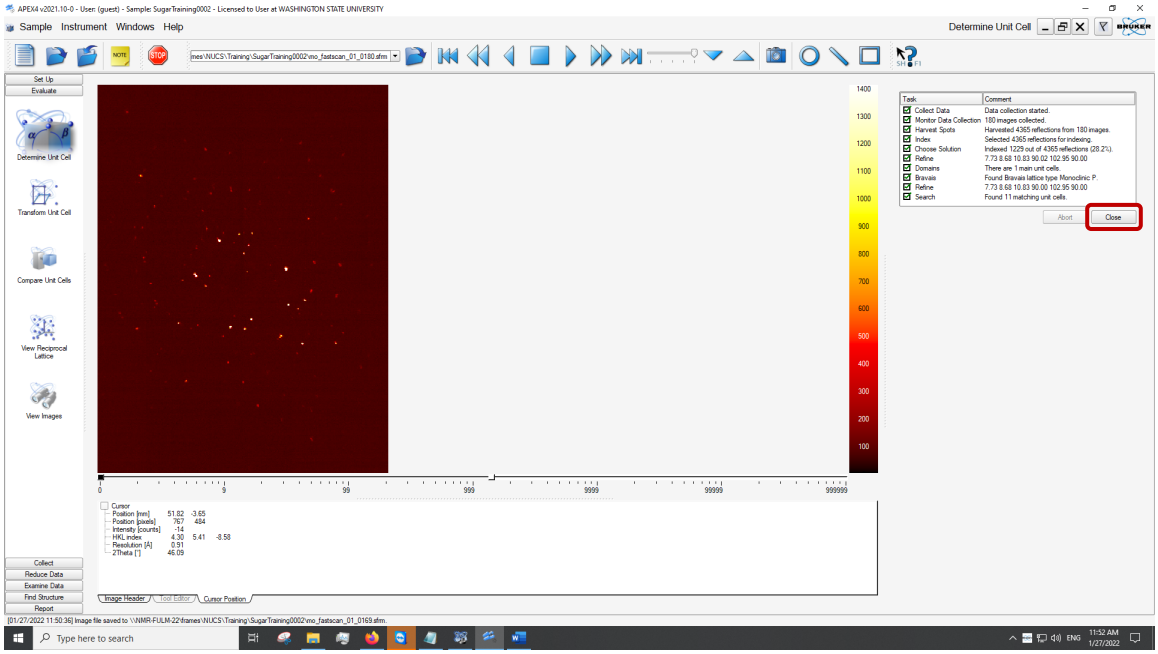
6.32 Within a few seconds you should be seeing spots. If you do not see any spots, then the mounted crystal does not diffract X-rays and a different one should be chosen.



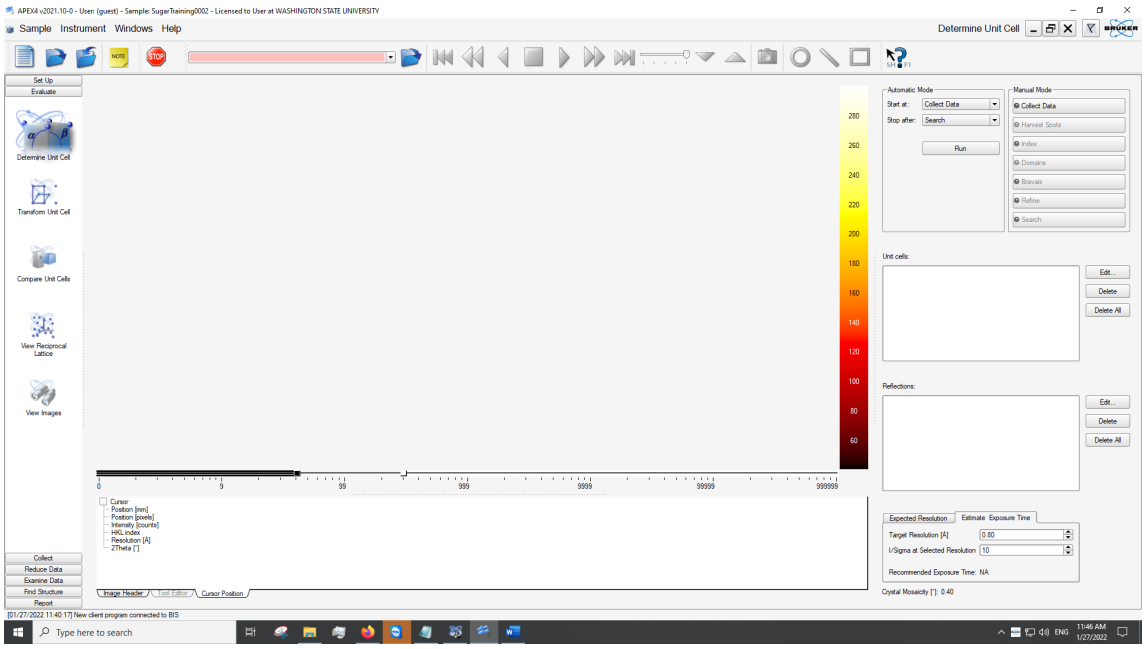
6.33 The unit cell data collection will be completed when the time left on the Bis Server (highlighted in red) says zero. A typical unit cell data collection will last about 3-5 minutes per unit cell.



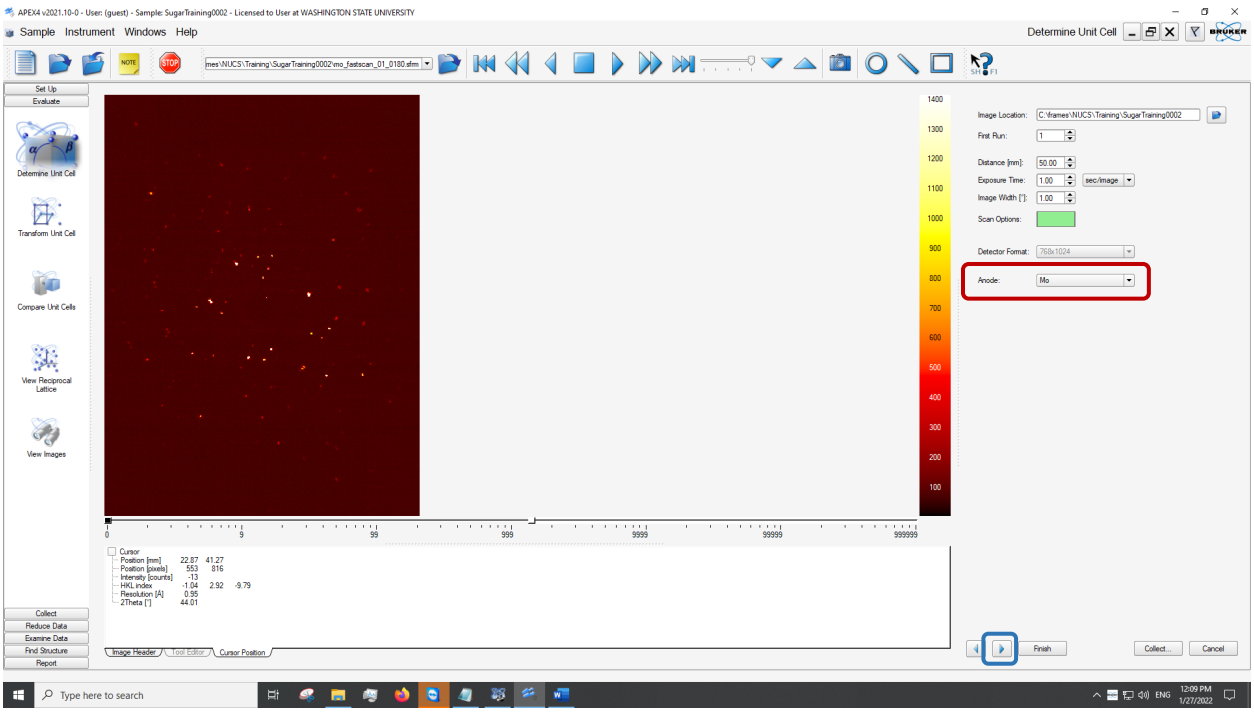
6.34 Upon completion of the unit cell data collection, the spots will be harvested, indexed, a solution will be found, the unit cell will be refined, the software will look for twin domains, determine a Bravais lattice, refine the unit cell further, and look to see if the unit cell matches any previously reported compounds. The search examines the open crystallography database, CCDC (if have a subscription), and the structures solved with the diffractometer. Can click the Close button (highlighted in red) to see the results of the search.



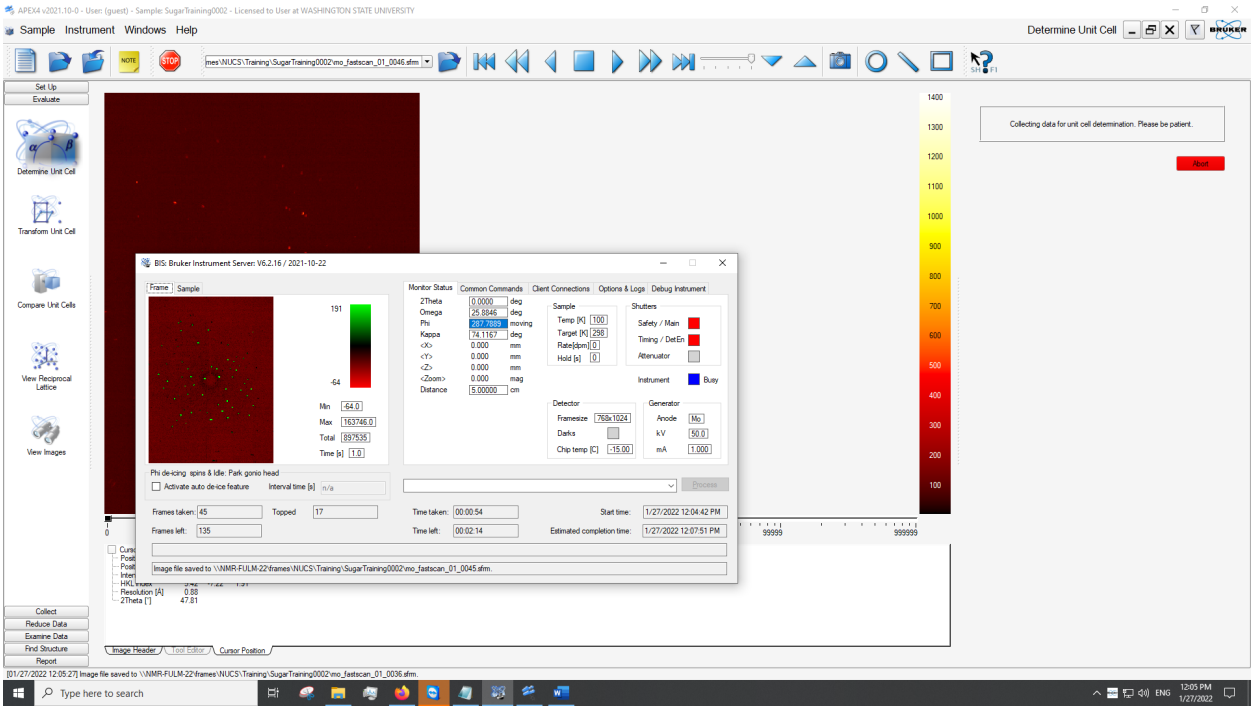
6.35 For manual unit cell data acquisition, click on the individual buttons of the Manual Mode window.



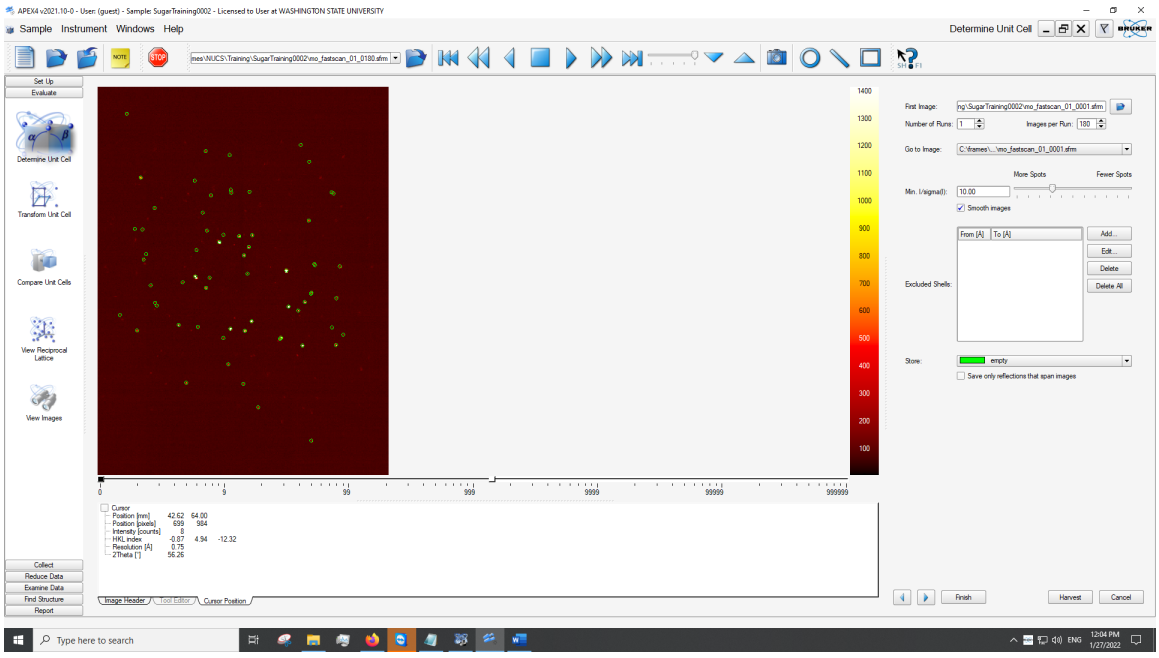
6.36 Click on Collect data to set the parameters and begin data collection on a unit cell. Here the instrument can be switched between using the molybdenum and the copper X-ray tubes (highlighted in red). Adjust the parameters as necessary and click on Collect.



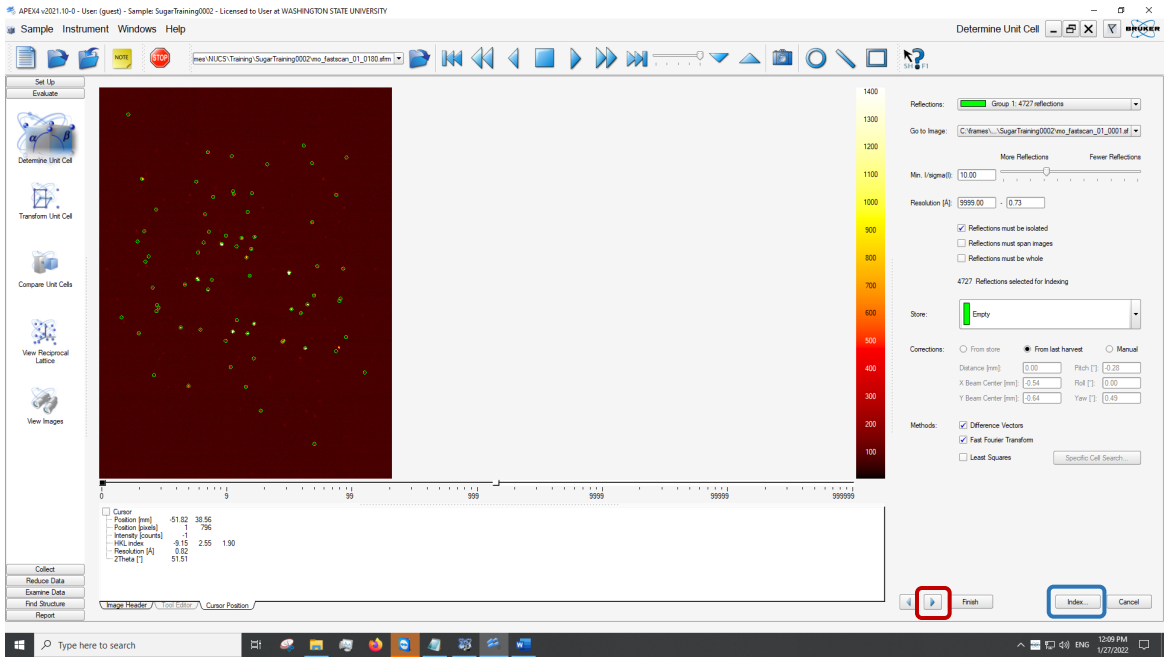
6.37 After the data collection, you can click on the right arrow (highlighted in blue in the previous picture) to harvest the spots. Do not click on the back arrow, or it will collect another unit cell.



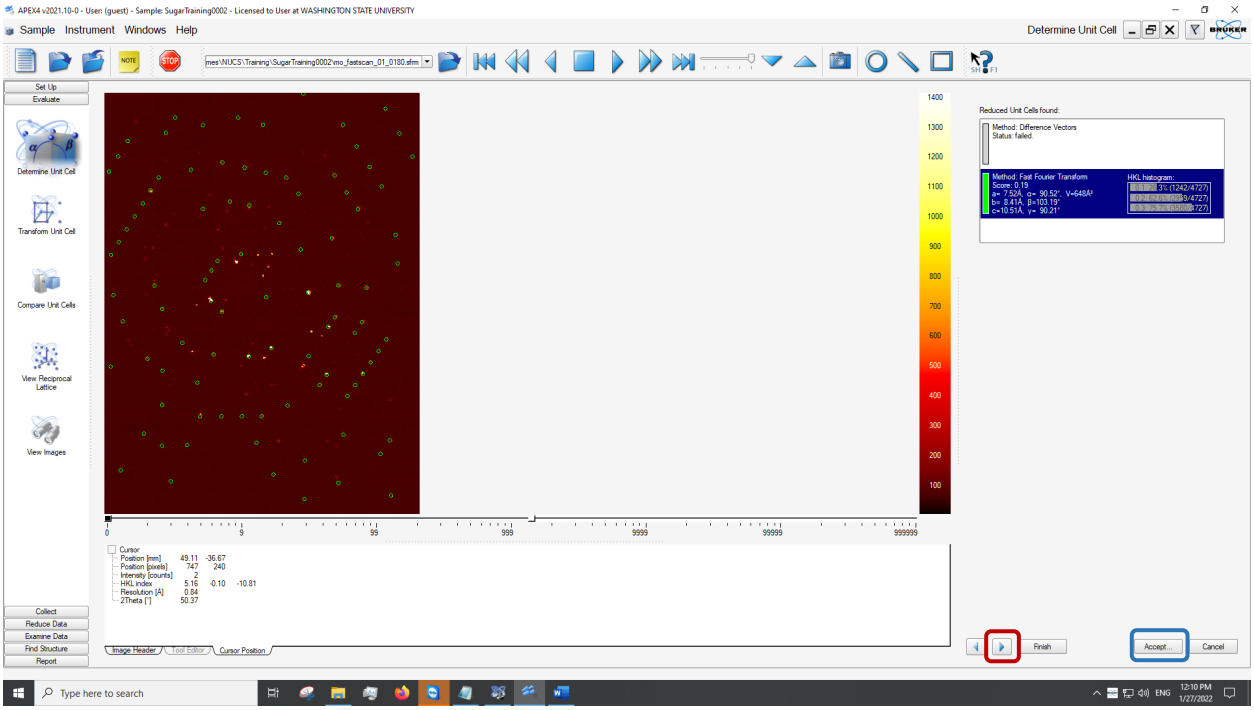
6.38 The manual mode will ask for the locations of the images and the resolution. I/sigma is a measure of the signal to noise ratio. Resolution of the data set is determined by a combination of statistics pertaining to the last shell (high resolution shell) of the data set. At the end of the data collection the I/sigma of the highest resolution shell be greater than 2.0 and the Rsym be less than 50%.



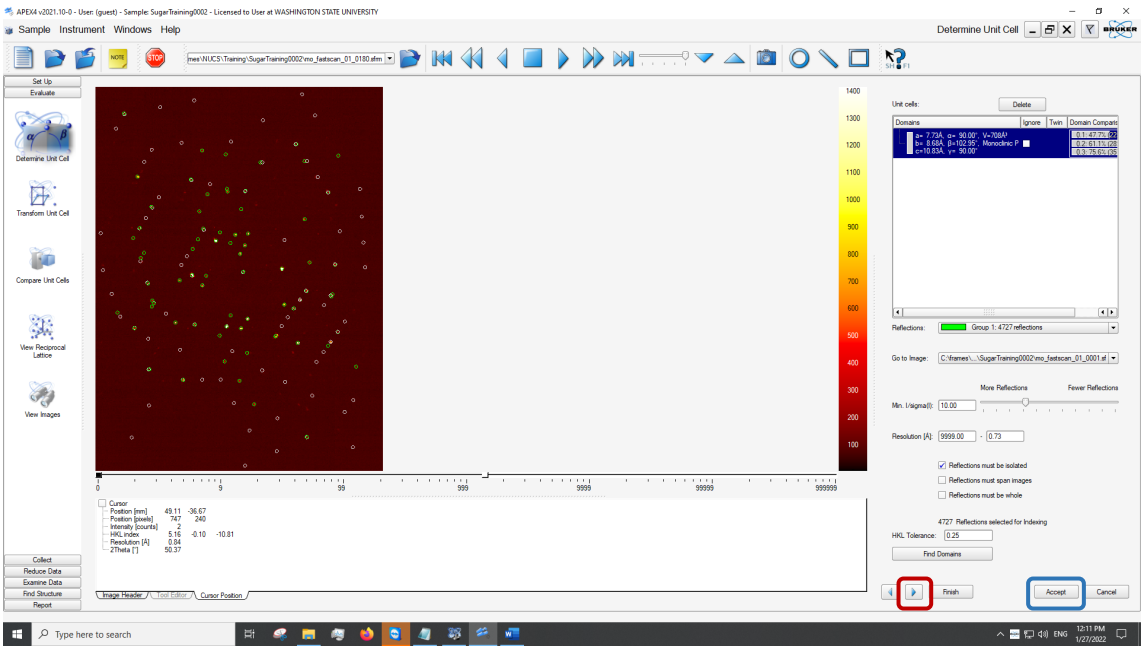
6.39 Clicking the right arrow (highlighted in red) or the Index button (highlighted in blue) will move on to the indexing step.



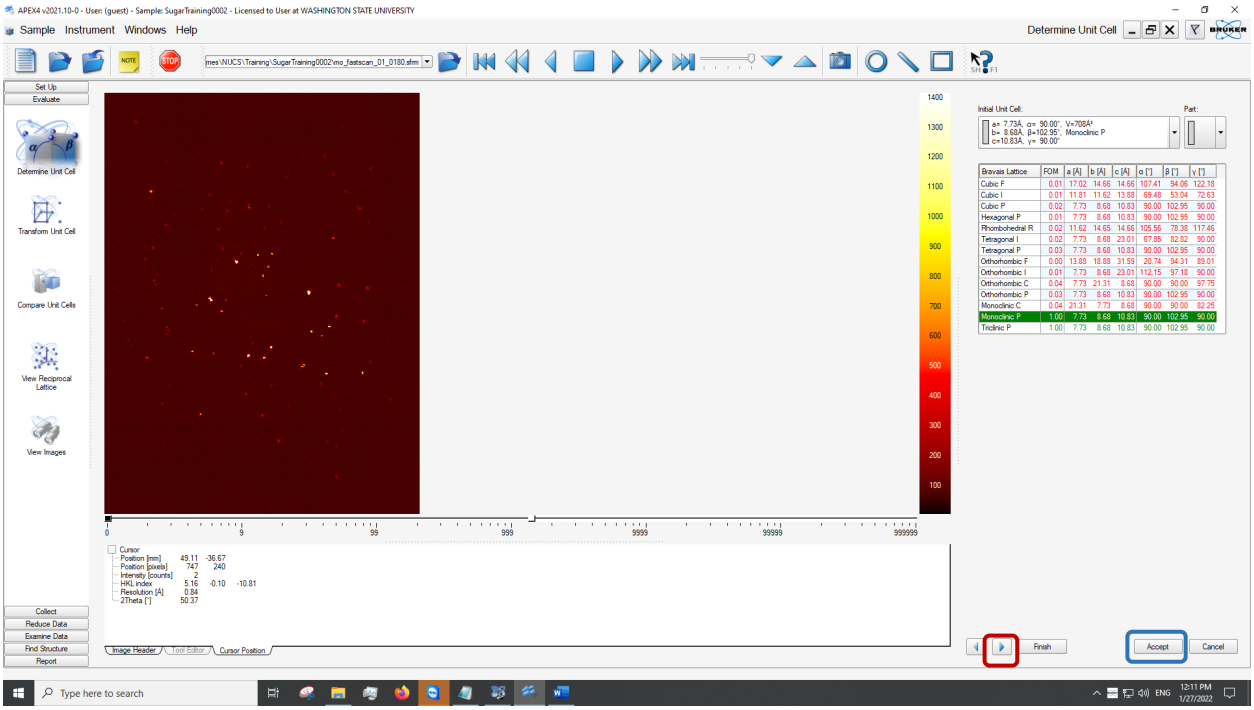
6.40 After indexing the unit cell, the unit cell will need to be reduced to see if a smaller unit cell can be generated. Select one of the reduced unit cells by highlighting it and clicking on the right arrow (highlighted in red) or the Accept button (highlighted in blue) will move on to the next step to look for twin domains.



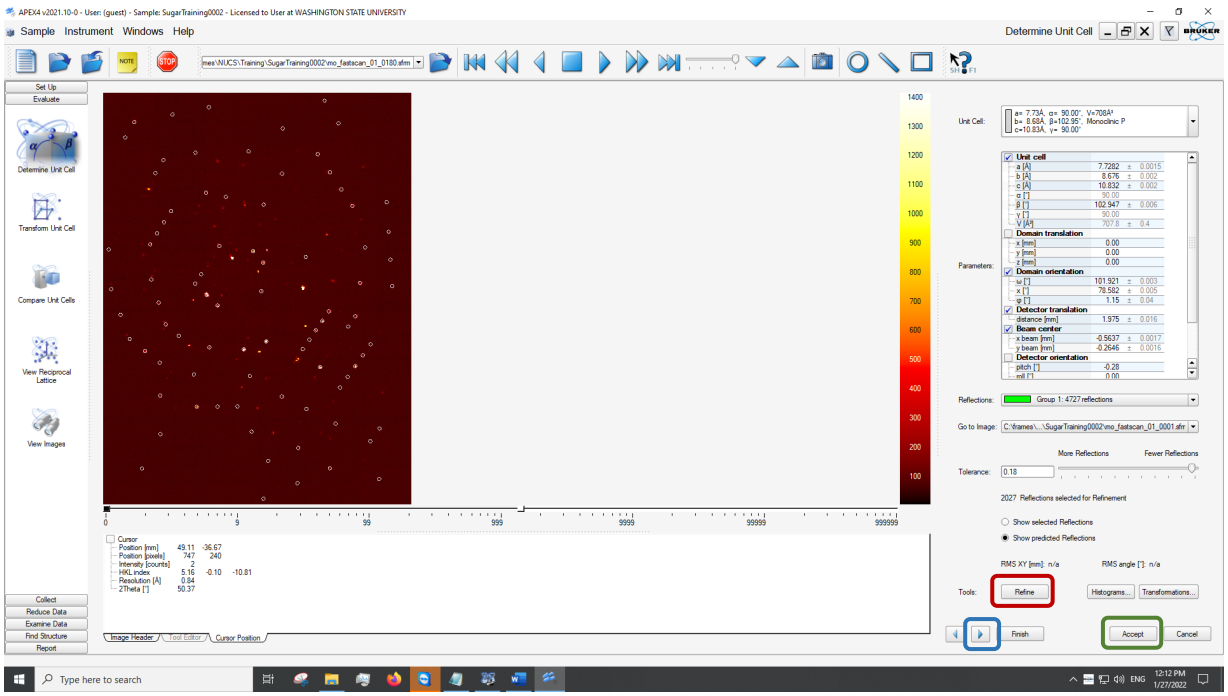
6.41 The manual software then searched for twin domains in the crystal. If more than one option is available select one of the options that seems the most plausible (this option is usually highlighted). Then, click on the right arrow (highlighted in red) or the Accept button (highlighted in blue) will move on to the next step to pick a Bravais lattice.



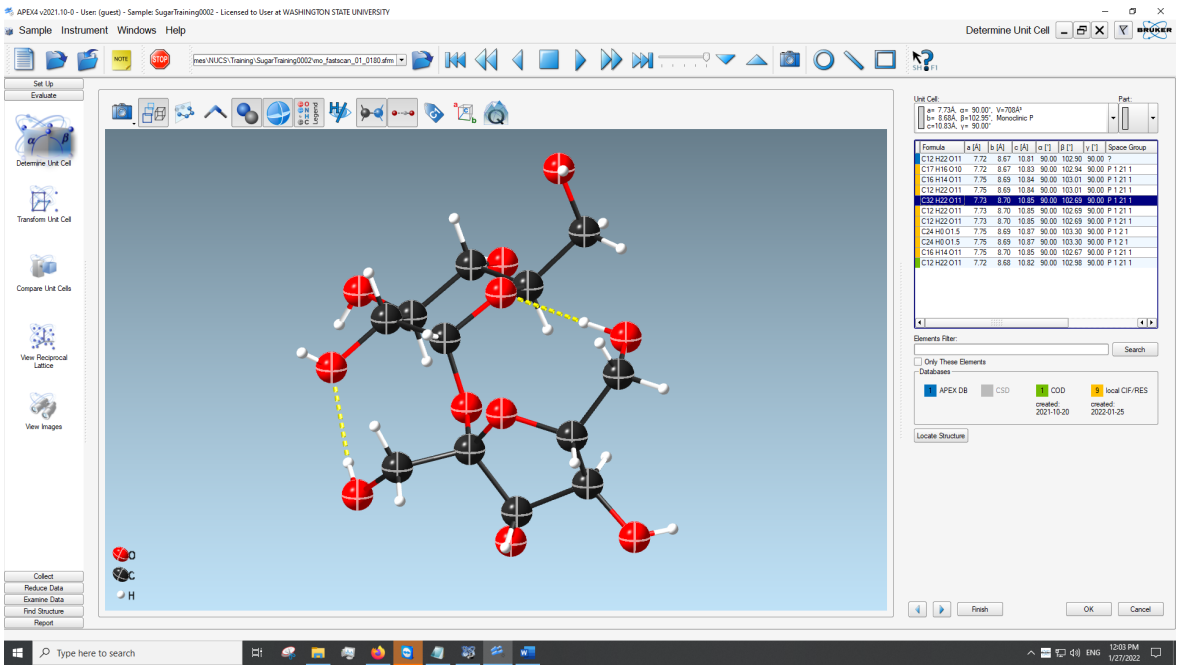
6.42 After looking for twin domains, the software determines the Bravais lattice of the crystal. The default value is often correct, but if another lattice is desired it can be chosen by clicking on it to select it. Then, click on the right arrow (highlighted in red) or the Accept button (highlighted in blue) will move on to the next step to pick a Bravais lattice.



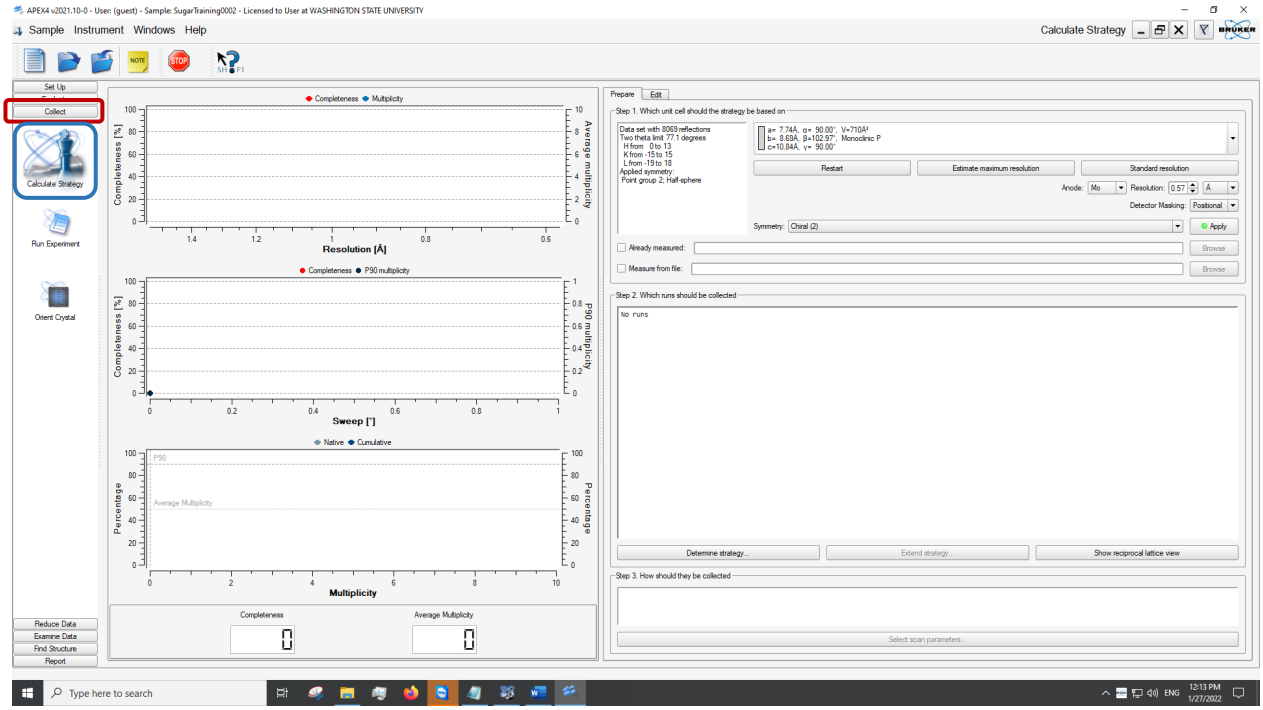
6.43 The software will then refine the lattice and the unit cell based on the lattice and space group. Click on the Refine button (highlighted in red), followed by the right arrow (highlighted in blue) or the Accept button (highlighted in green) to move on to the next step to search the crystallographic databases to see if it is a known structure.



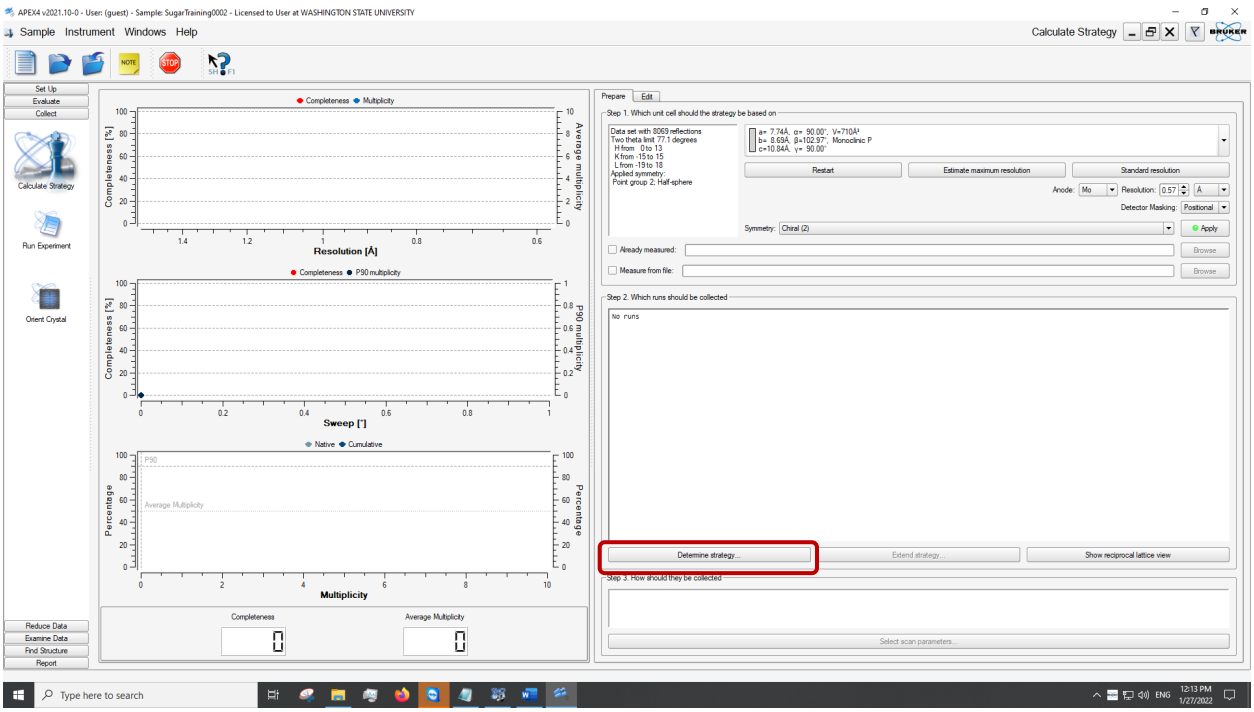
6.44 The unit cell determination will then search to see if the unit cell is known in the CSD, COD, and local data base of structures. The results seen below can be clicked on to see if it is possible that the structure is already known instead of collecting a full structure on a known compound.



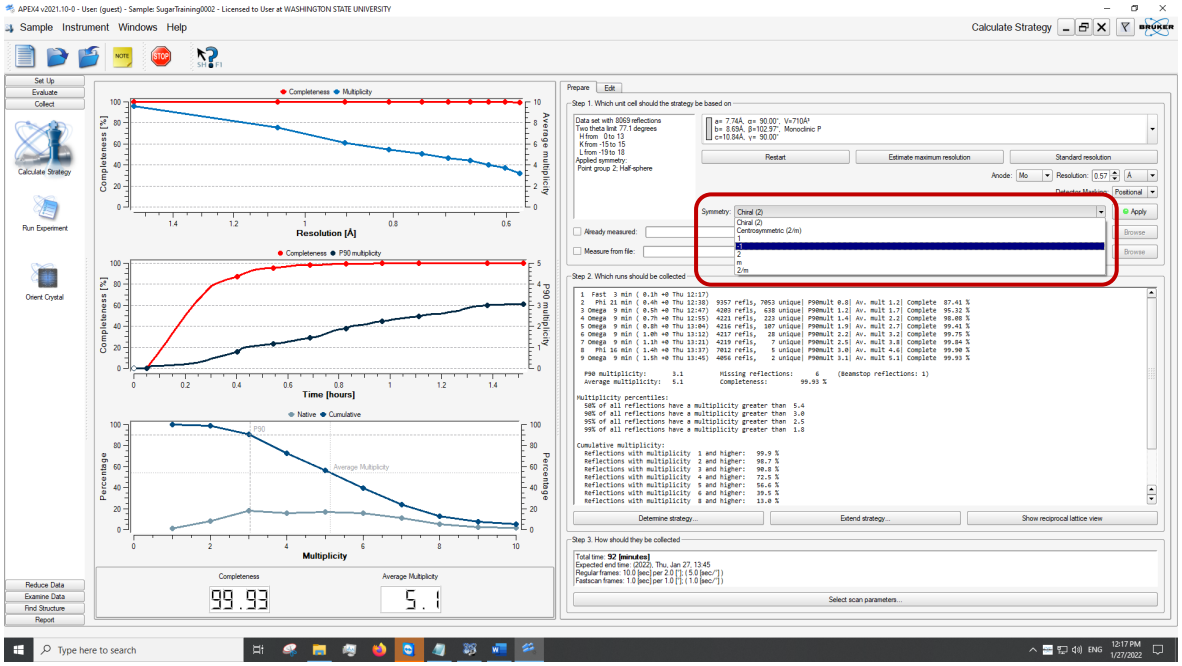
6.45 After it is known that a full data set is desired and the unit cell has been completed, click on the Collect button on the left side of the screen (highlighted in red), followed by the Calculate Strategy icon (highlighted in blue).



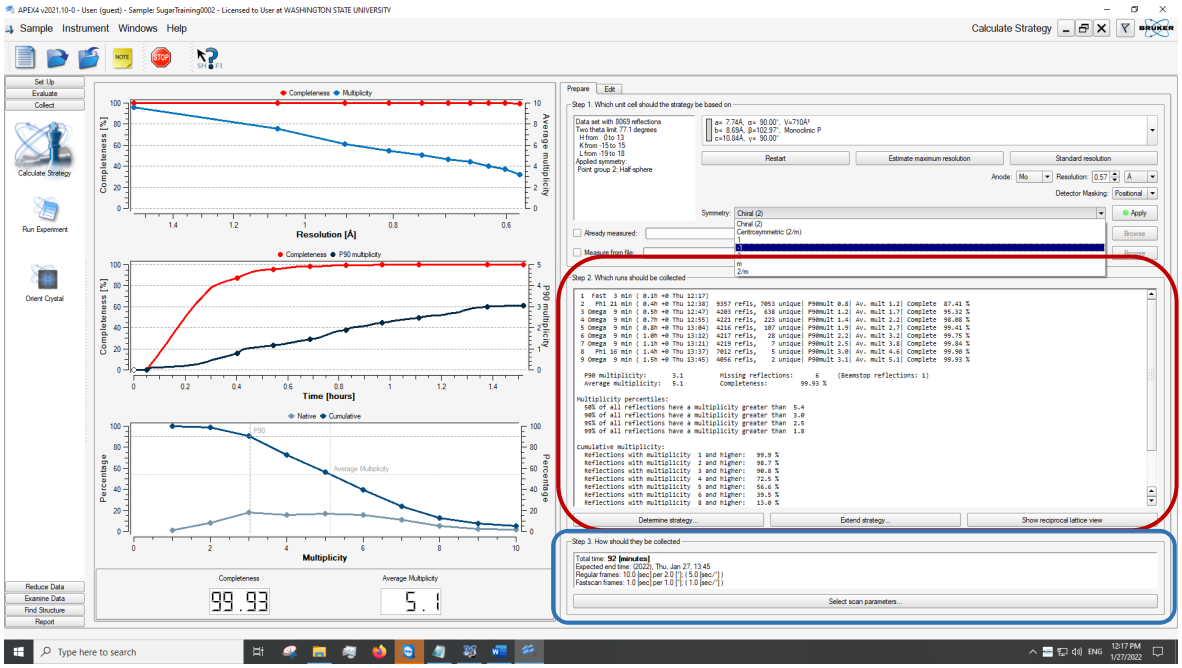
6.46 To determine the length of the data collection, click on the Determine strategy (highlighted in red) and the software will run some calculations for a little while to determine the best strategy for the data collection.



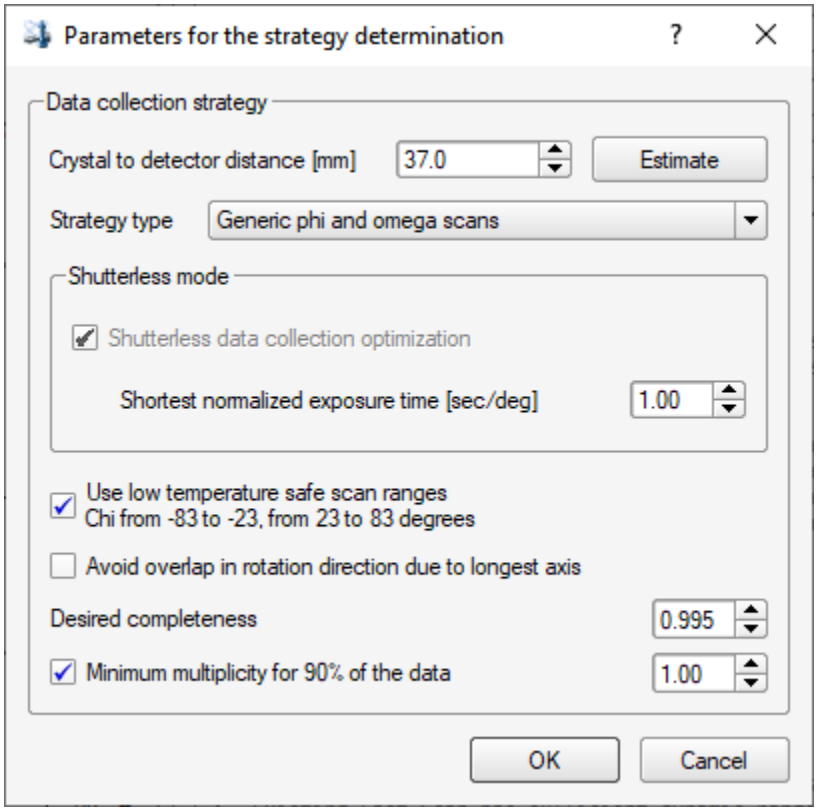
6.47 Sometimes there is a problem with collecting data in a higher space group if the crystal ends up being in a lower space group, which will result in unsolvable data. To avoid this possibility, click on the Symmetry drop down menu (highlighted in red) and collect the data in P1 or P-1. The data collection will be longer, but it will usually not need to be recollected. If the default value is changed, make sure to click on the Apply button before moving on.



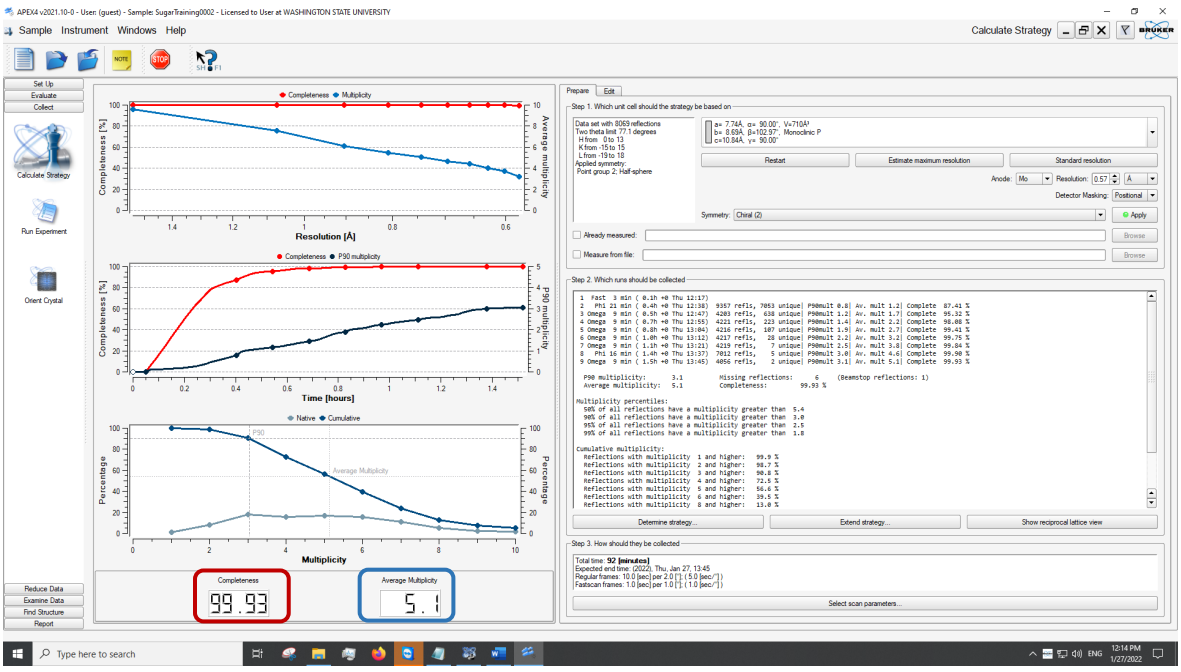
6.48 Step 2 (highlighted in red) will tell you the parameters for the run, the multiplicity. Step 3 (highlighted in blue) will tell you the frame rates and the total time for the data collection.



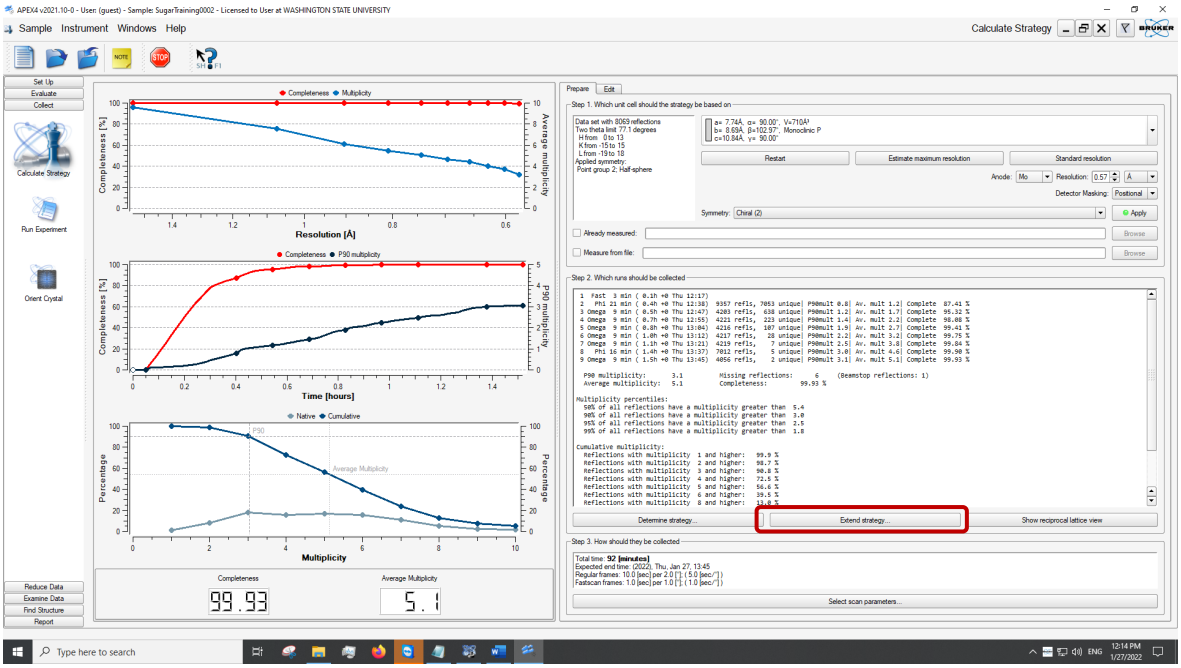
6.49 Click on the Determine strategy button, which will open a window. The default values are acceptable and the OK button can be clicked.



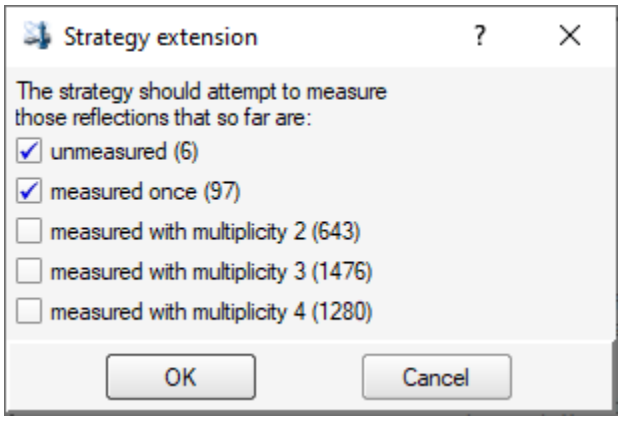
6.50 Upon clicking OK, it will take a minute or two to determine the strategy. The completeness plot should be near 100%, at least greater than 98% (highlighted in red). The multiplicity should be at least 4 (highlighted in blue).



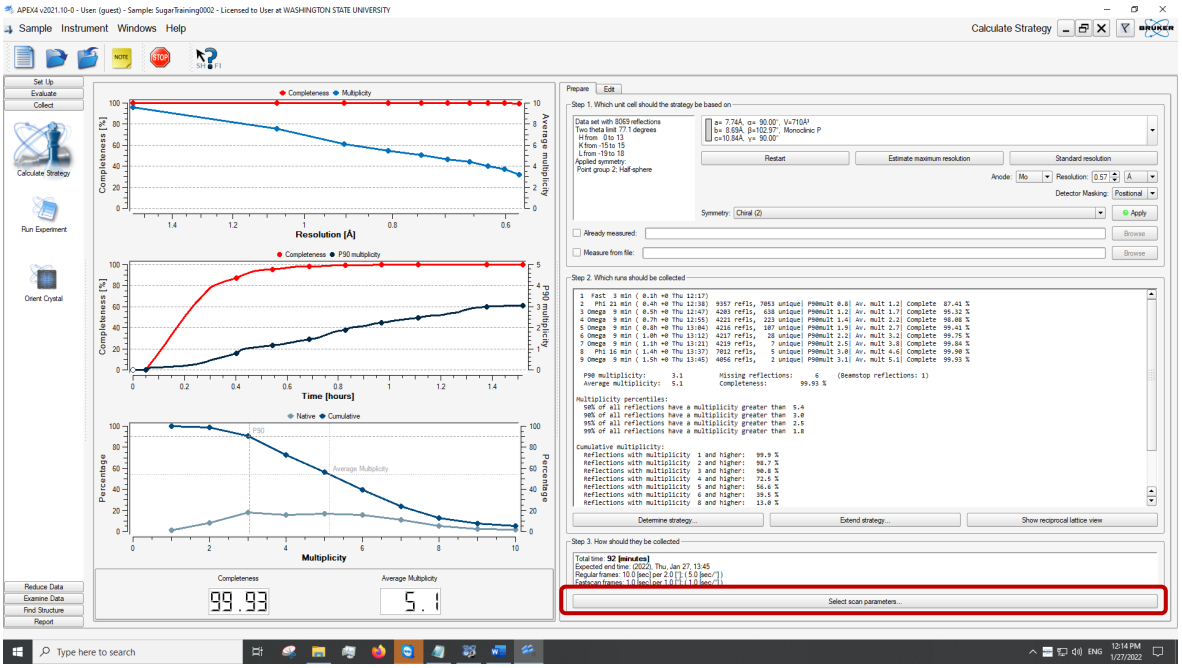
6.51 Before moving on, the parameters need to be checked to see if additional data can be collected will minimal time increase by clicking on the Extend strategy button (highlighted in red).



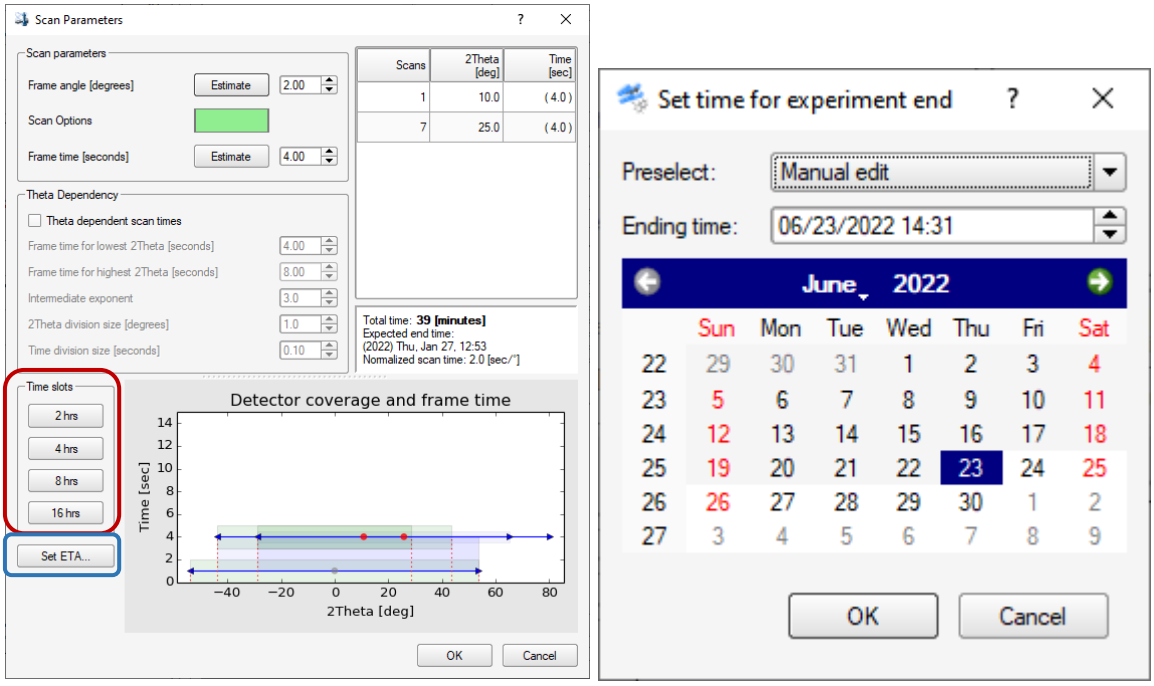
6.52 A window will appear allowing to see how long the data collection would be if extra reflections that will not be easily seen are included in the data collection. Start by checking all of the boxes to see if they can be picked up. If they cannot, repeat steps 6.51 & 6.52 with one or two less boxes checked. If no message shows up when clicking the OK button the Strategy extension was successful.



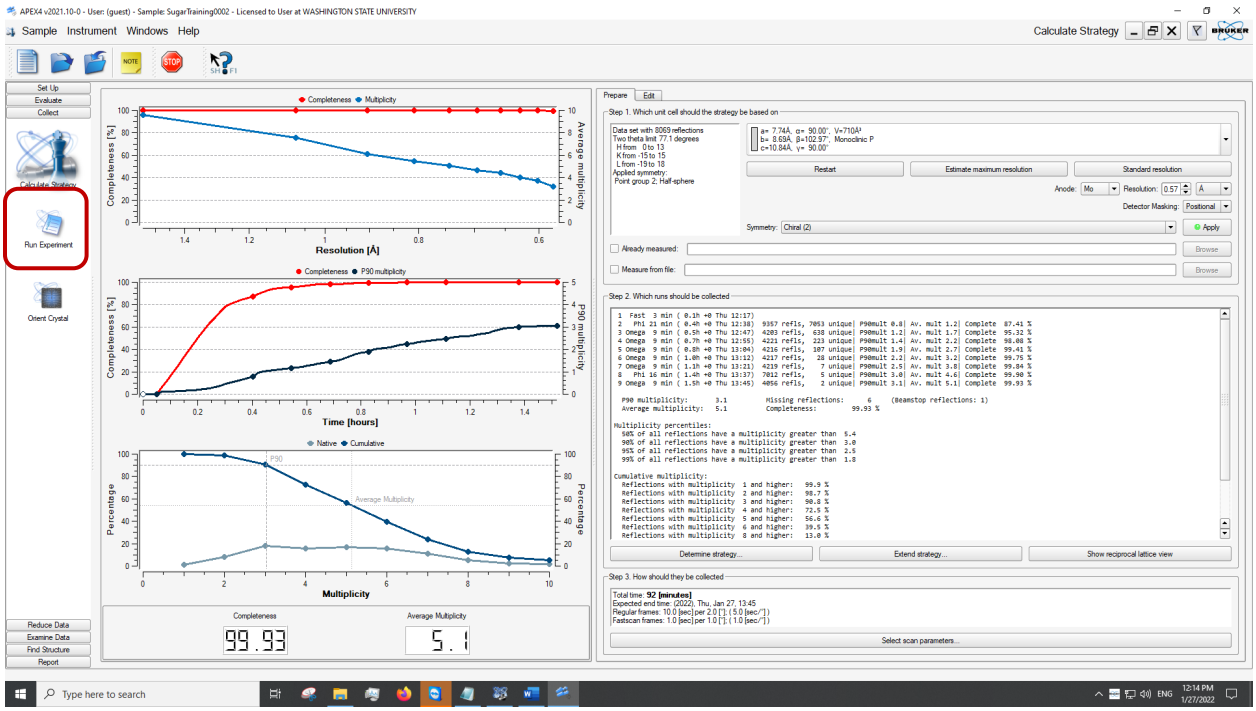
6.53 At this point the data can be collected (skip to Step 6.56), or the time and parameters of the data collection can be adjusted to run for a specific length of time. To run the experiment for a specific length of time, click on Select scan parameters button (highlighted in red).



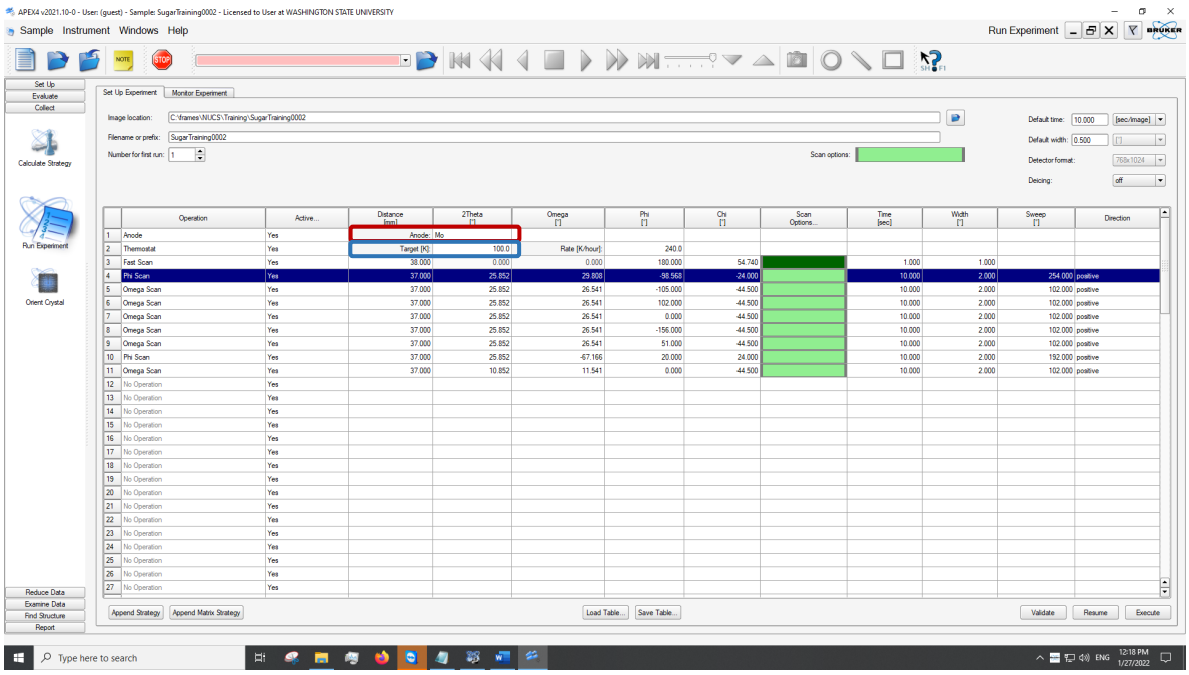
6.54 The following window will result, allowing for the data collection to run for a certain number of hours (highlighted in red) or a custom length (highlighted in blue). Clicking on the Set ETA button opens up a separate window that allows for the ability to set a custom experiment end time.



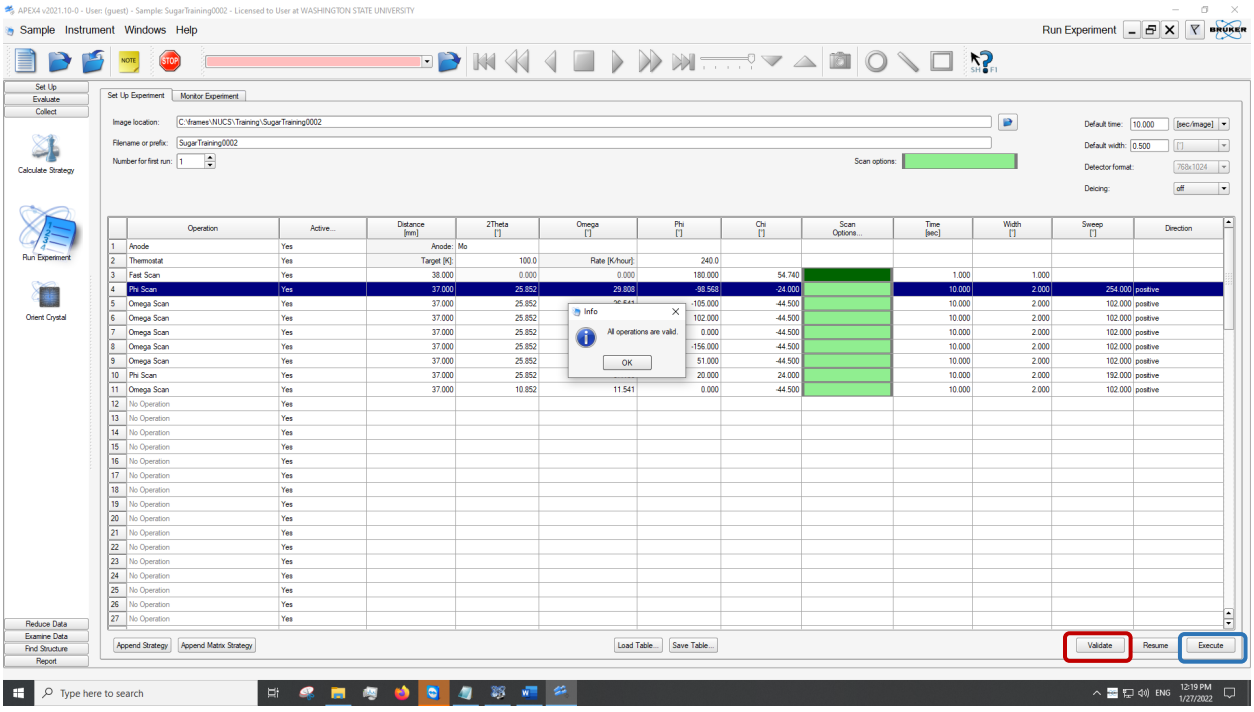
6.55 To run the experiment, click on the Run Experiment icon (highlighted in red).



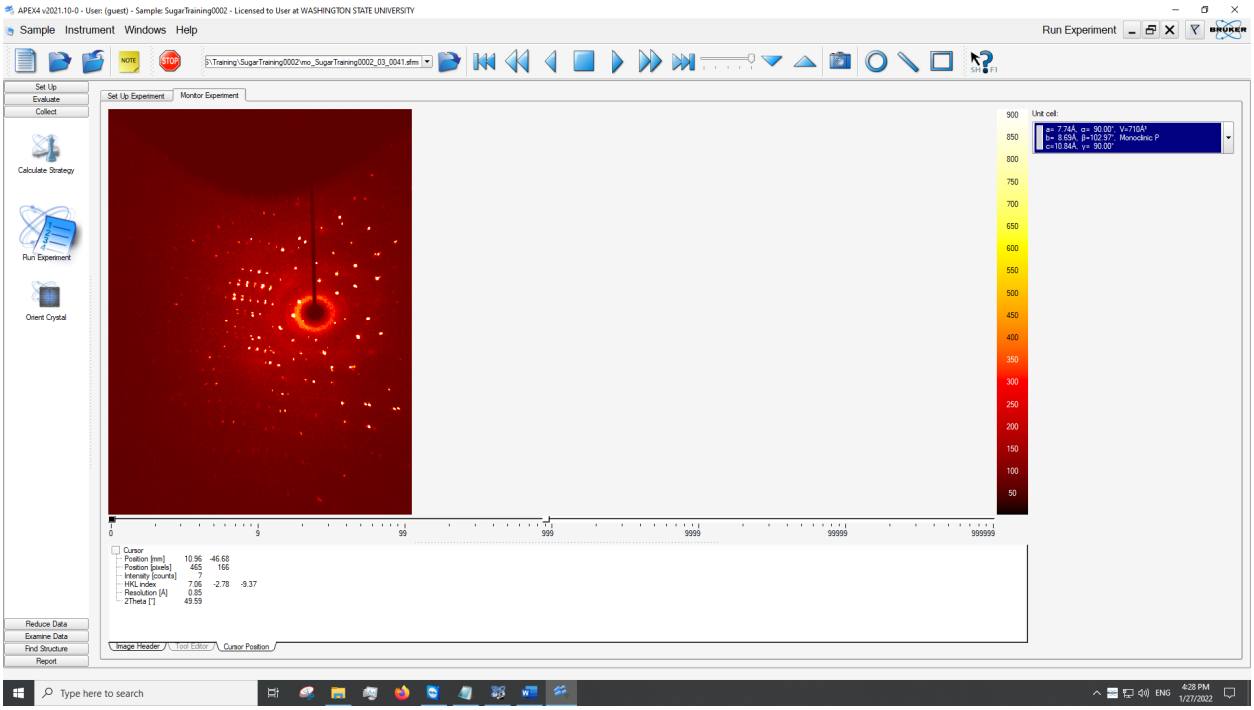
6.56 Clicking on Run Experiment will bring up a window that looks like a blank spreadsheet. This part of the experiment brings up the data collection strategy and any other parameters that are to be added to the data collection strategy. Make sure that the anode is correct (Mo vs. Cu) (highlighted in red), that if the data collection is done at room temp, nothing needs to be done, but if the data collection is to be done at 100 K, the thermostat option with a target temperature of 100K needs to be entered in the parameters (highlighted in blue). Then, to add the data collection parameters from the previous screen, click on the Append Strategy button. If you want the cryocool to end after the data collection, then the Thermostat option off will need to be at the end of the strategy. This option is important for overnight runs to conserve liquid nitrogen.



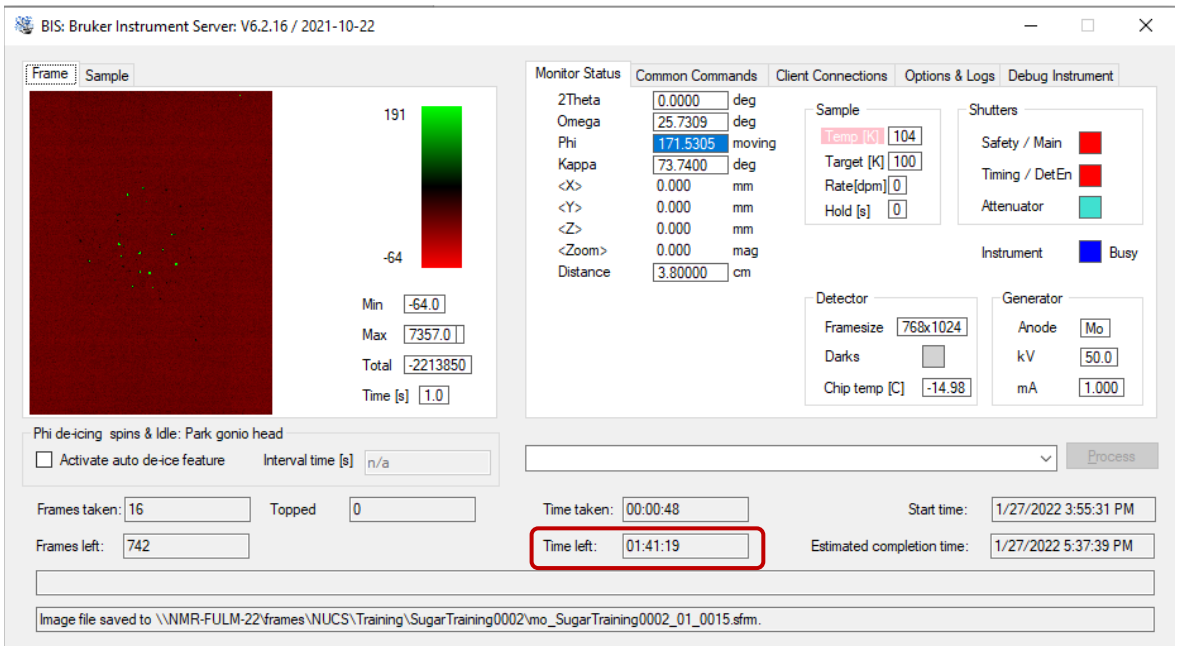
6.57 Before the data collection can be started, the Validate button (highlighted in red) needs to be clicked to make sure that there are no errors in the strategy that could cause the instrument to error out during the data collection.



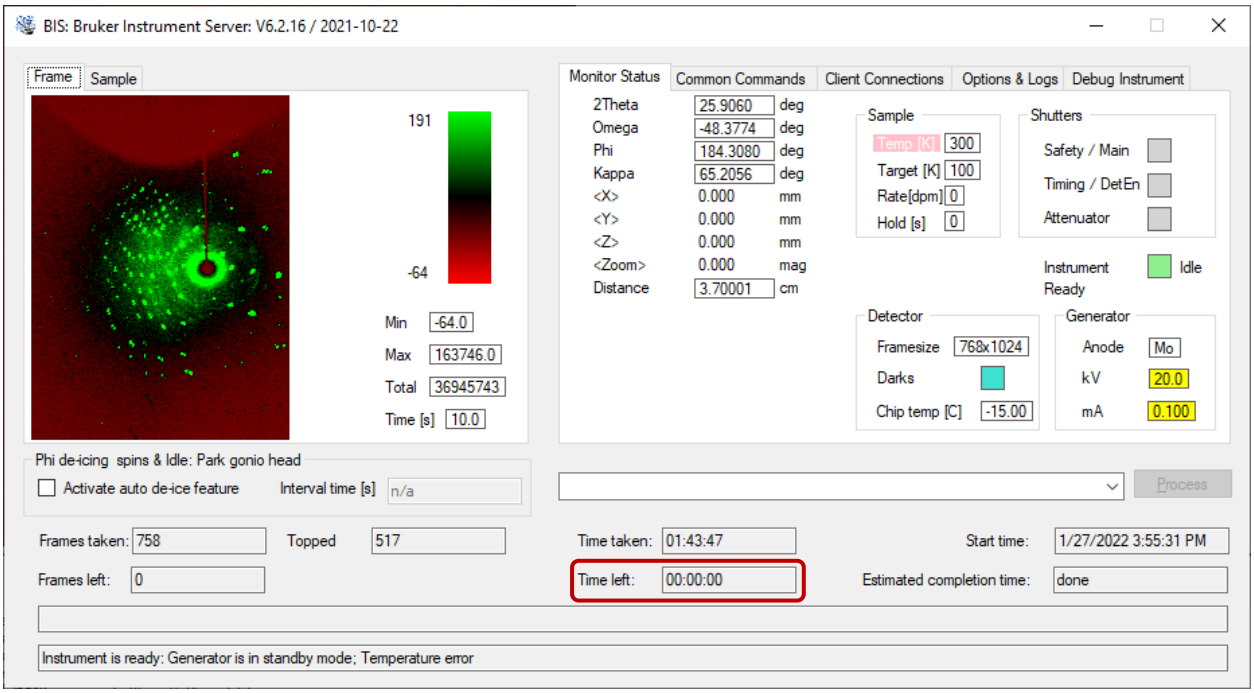
6.58 One the strategy has been validated (as seen in the picture above), The Execute button (highlighted in blue in the above picture) can be clicked to begin the data collection. After a short amount of time (15-60 seconds) a window showing a diffraction pattern will result.



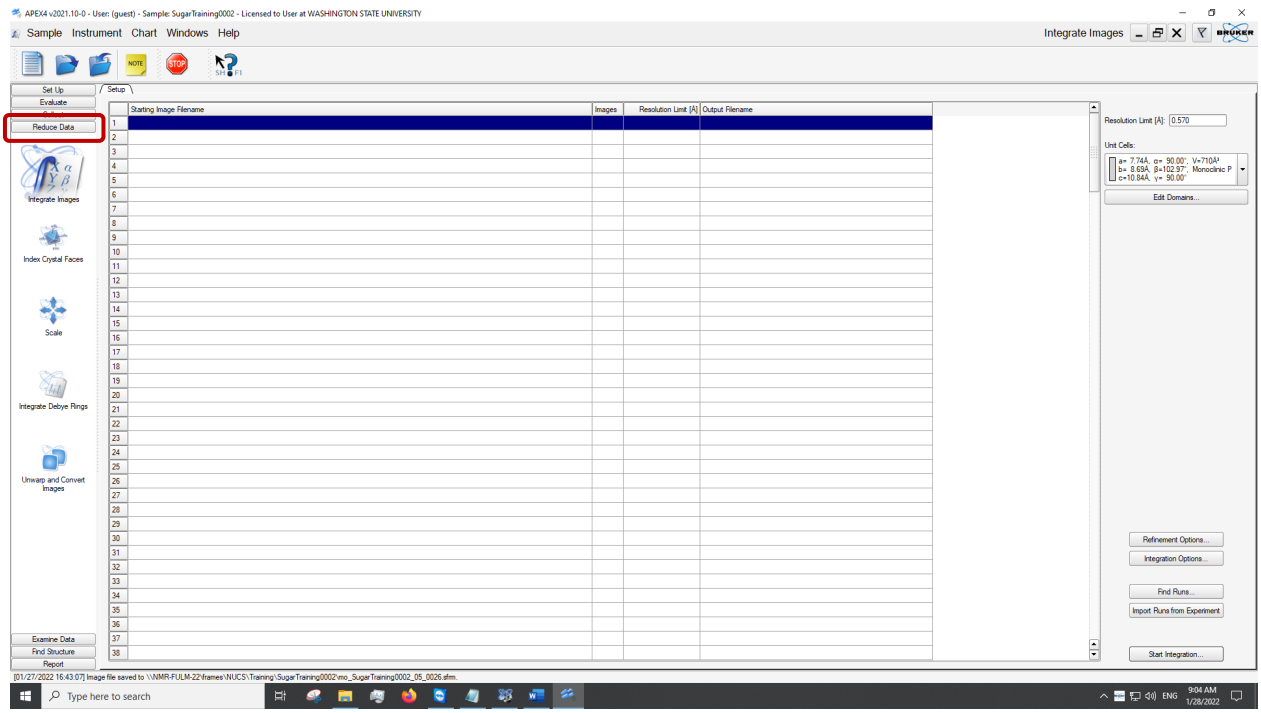
6.59 To see the time remaining on the data collection, the BIS Measurement Server can be maximized and the time remaining in the data collection can be seen in the Time left section (highlighted in red).



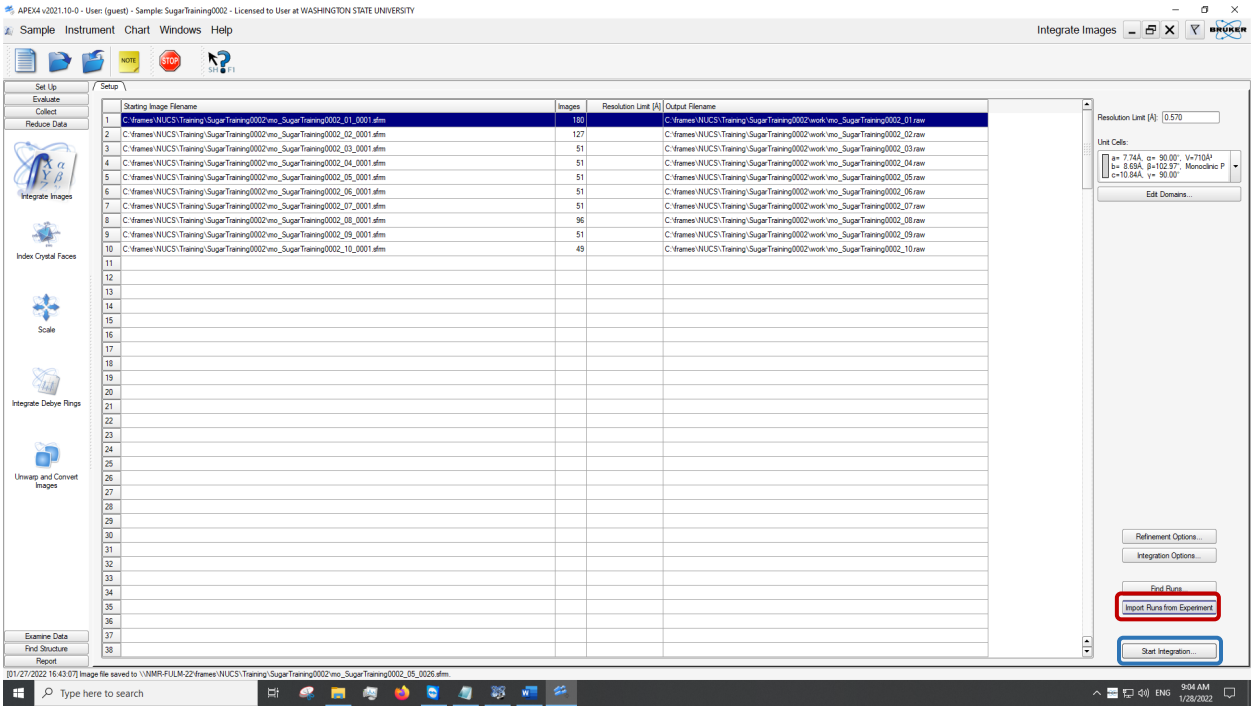
6.60 When the BIS server says that there is no time left in the Time Left section (highlighted in red), then the data collection has completed. If the thermostat was set to off after the experiment has completed then the temperature will be higher than the target temperature.



6.61 To workup the collected data, click on the Reduce Data button (highlighted in red), which will bring up the following screen allowing for the integration of the images.

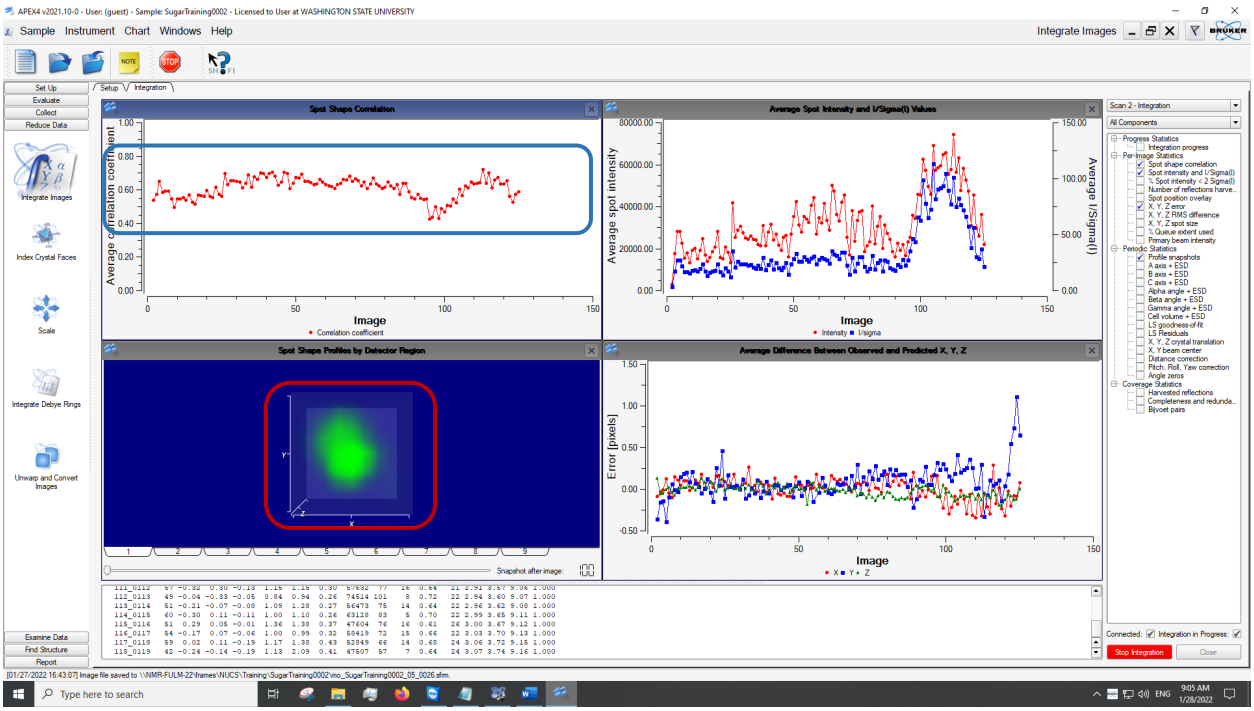


6.62 To integrate the images, the images need to be imported by clicking on the on the Import Runs from Experiment button (highlighted in red). The set of images should appear in the screen as seen below.

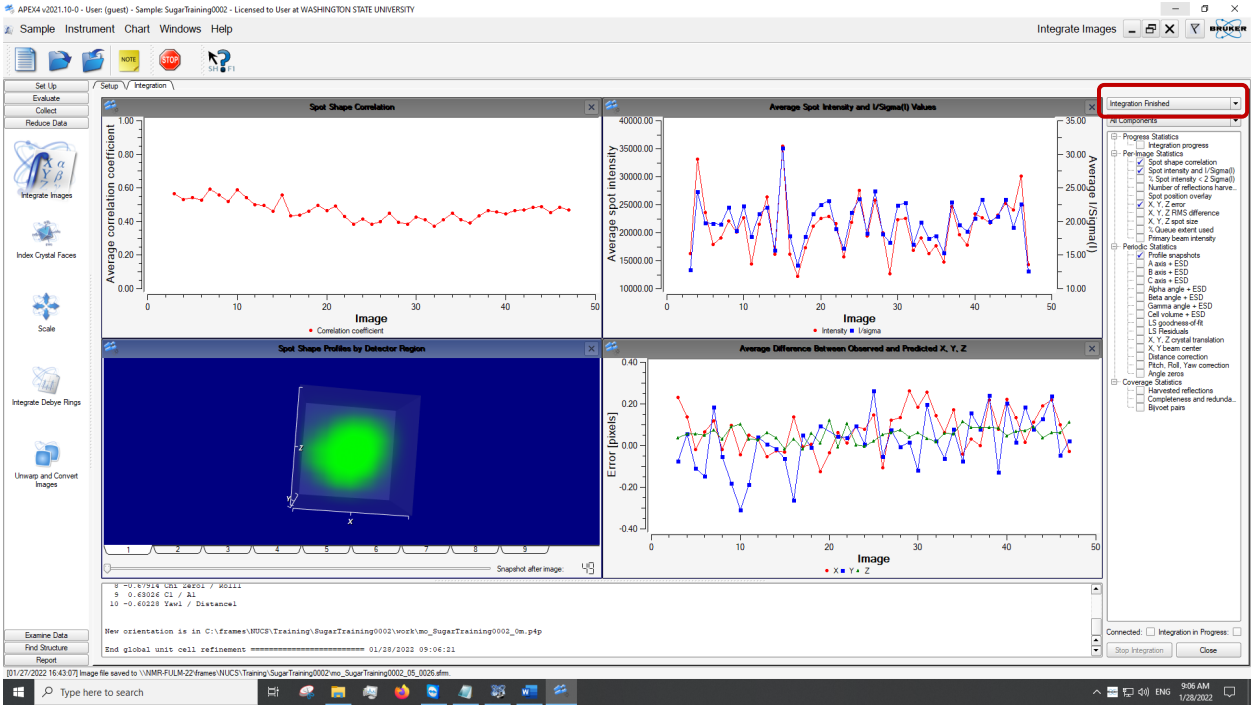


6.63 Click on the Start Integration button (highlighted in blue in the previous picture) to begin the integration of the imported images.

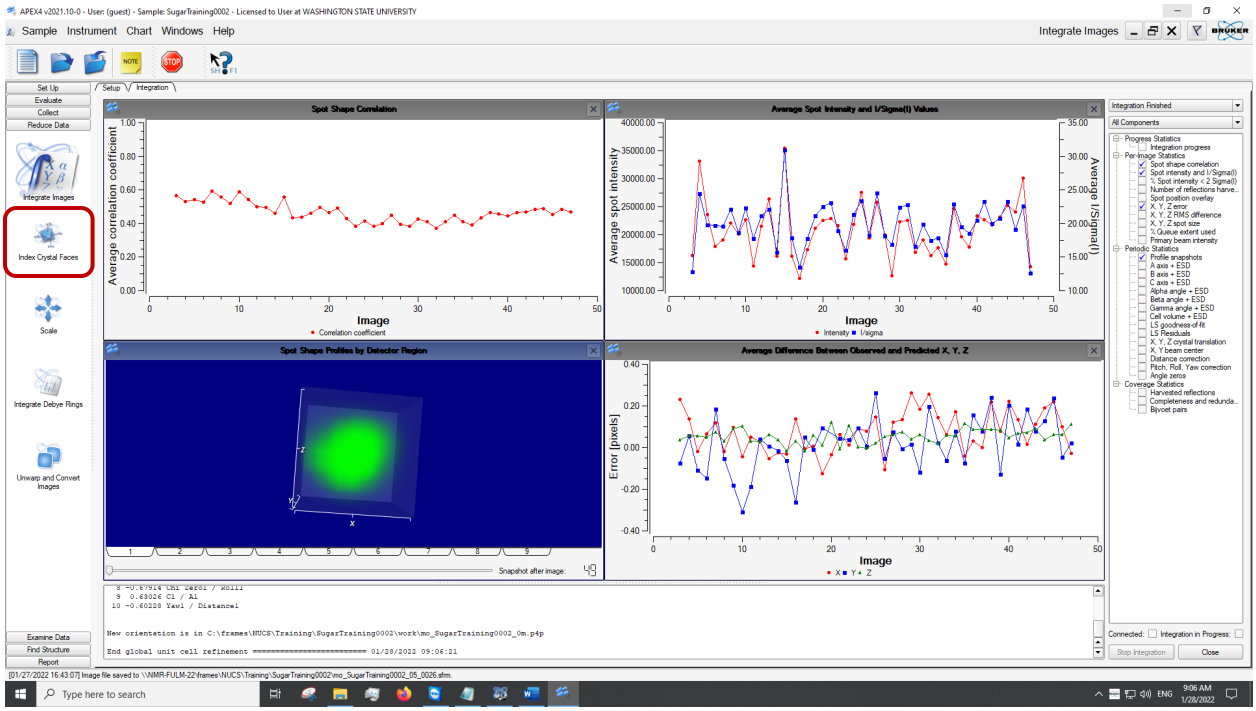
6.64 The frames will be integrated. This will take a while (5-10 minutes). The spot shape profile should be close to a sphere (highlighted in red) for good data. The spot shape correlation (highlighted in blue) should be around 0.60 to 0.80 for good data.



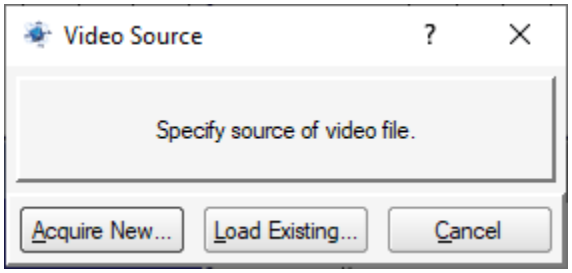
6.65 When the window states the Integration has finished in the top right (highlighted in red), the integration has completed.



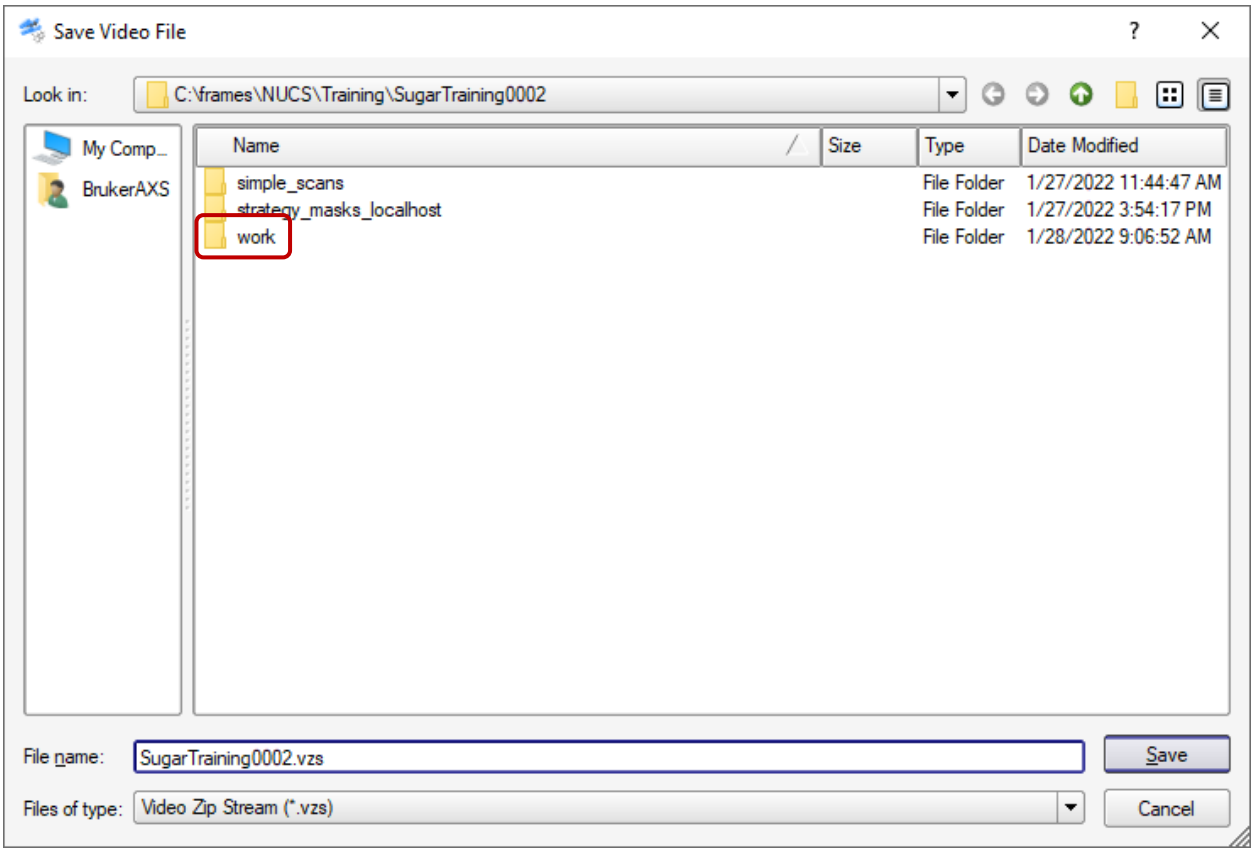
6.66 After the integration has completed, the faces of the crystal can be indexed. Indexing of the crystal faces takes about 5 minutes to collect the video and only works about 30% of the time. If indexing of the crystal faces is not desired, skip to Step 6.72. To start to index the crystal faces click on Index Crystal Faces icon on the left side of the Apex 4 window (highlighted in red).



6.67 After clicking on the Index Crystal Faces icon, a window asking for the video source will show up. Click on the Acquire New button.

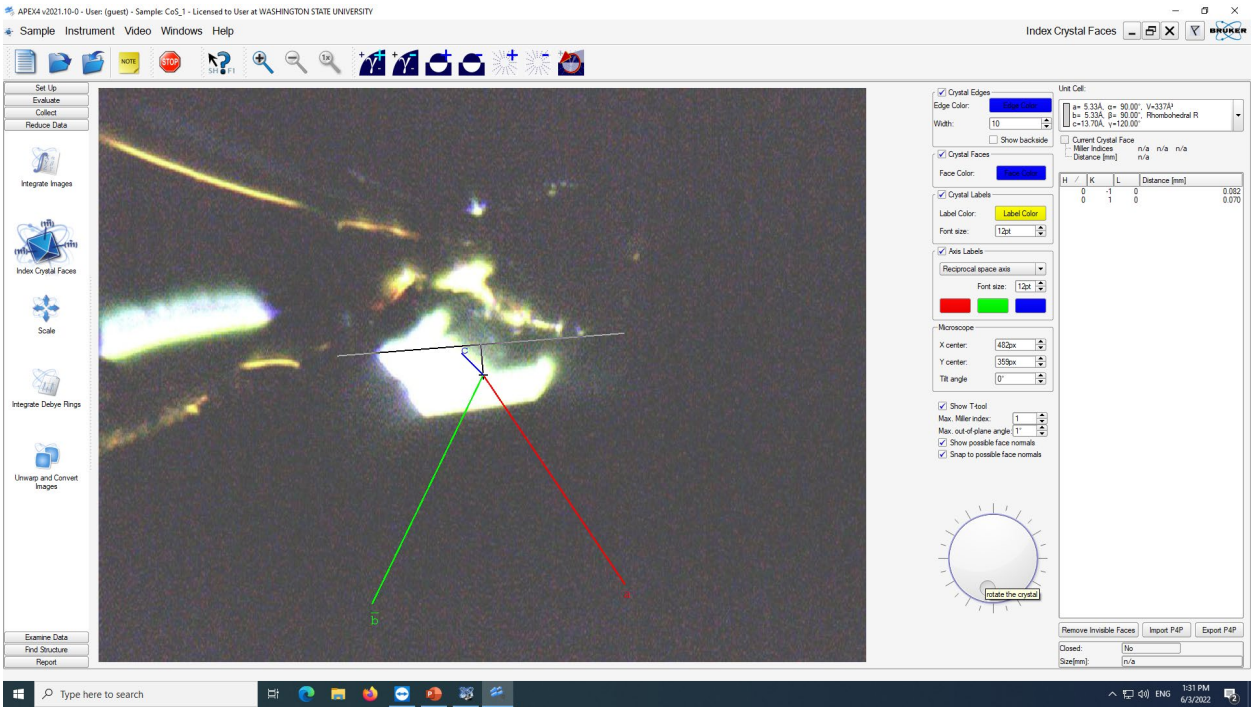


6.68 A window asking for a location to save the crystal face file will appear. Please save the file in the work folder (highlighted in red) of the respective data set.



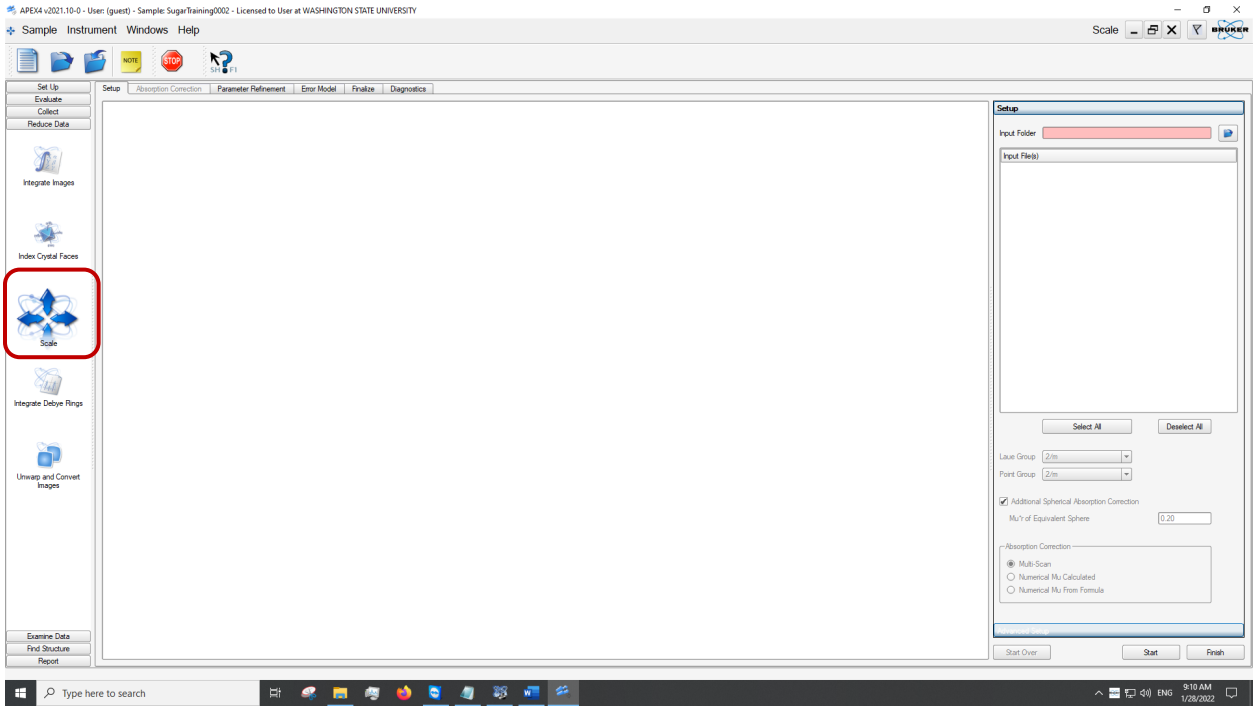
6.69 After clicking on save, a window saying that the goniometer is being driven followed by a window saying video is being acquired. This step takes about 5 minutes and works about 25 – 30% of the time.

6.70 Upon completion of the video acquisition, a picture of the crystal will appear, and the software can be used to add crystal faces.

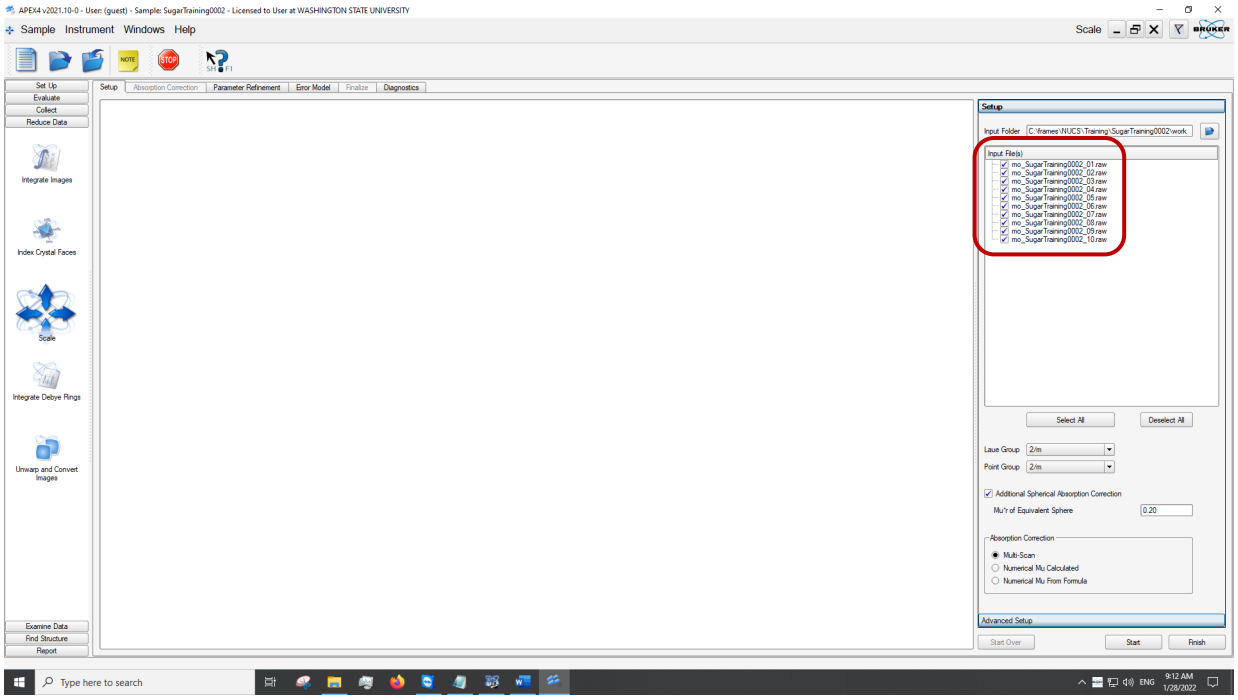


6.71 Crystal faces are added by left clicking with a mouse on the edges of the crystal. The software is not easy to use and does not often give satisfactory results.

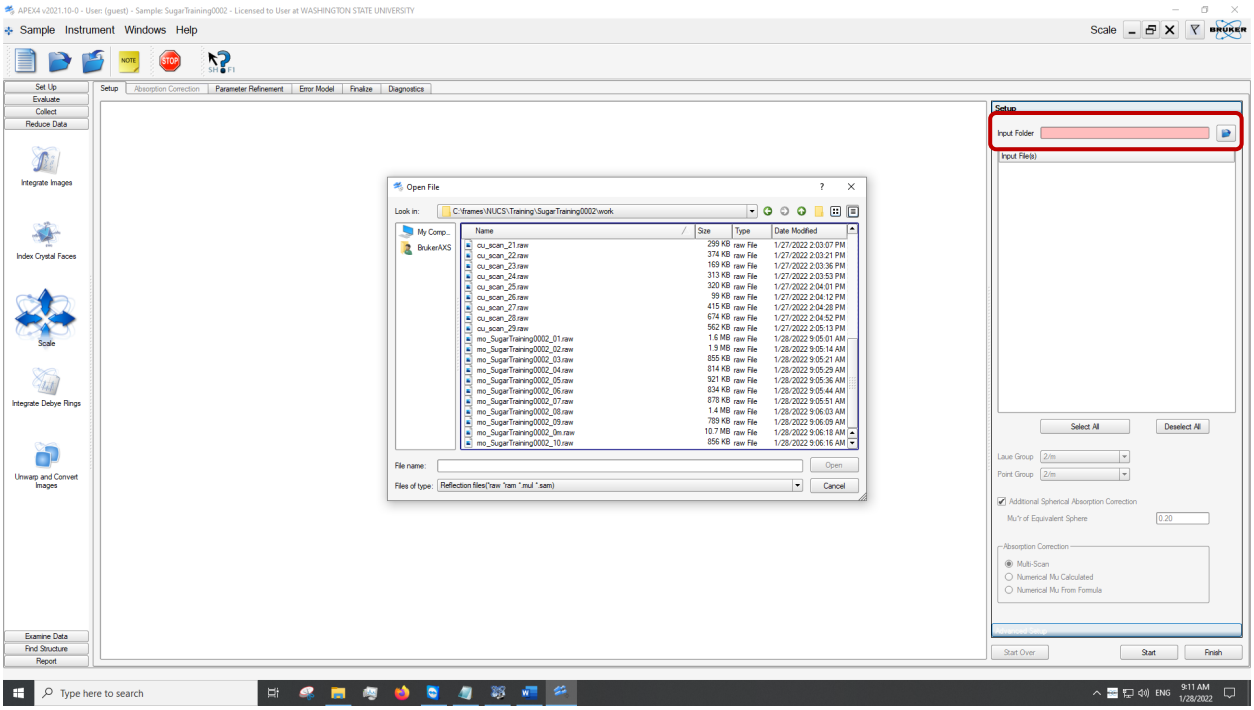
6.72 To continue analyzing the data, the data needs to be scaled. To scale the data, click on the Scale data icon on the left side of the Apex 4 window (highlighted in red).



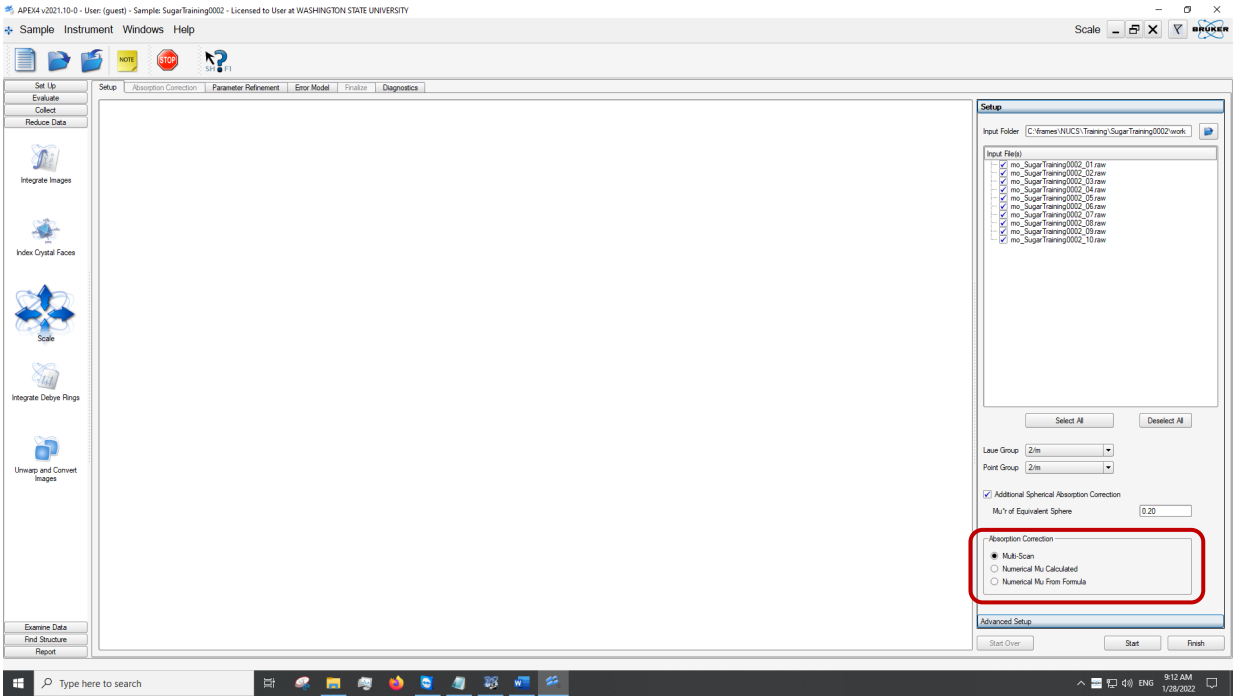
6.73 The data for the data collection should be already imported into the Scale window (highlighted in red). If the data imported correctly, skip to Step 6.75, if the data did not import correctly go to Step 6.74.



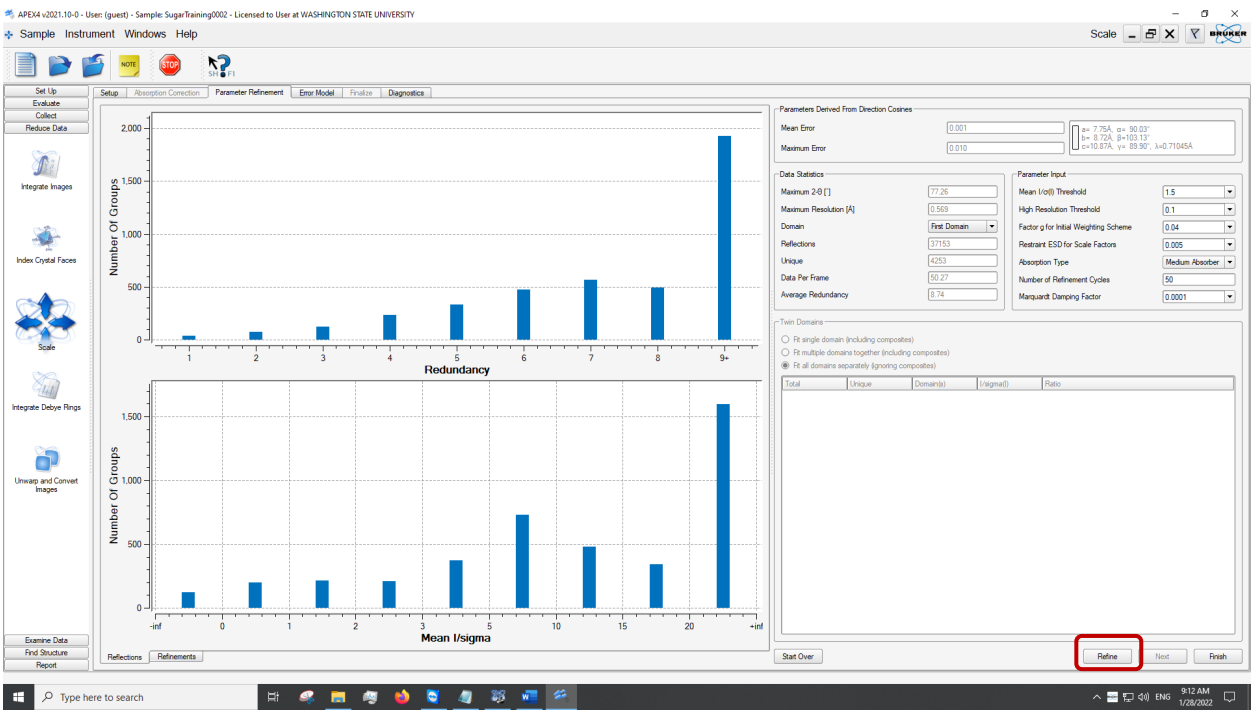
6.74 To import data, click on the folder icon (highlighted in red) and open the designated folder. Click on one file and then on open and all of the corresponding frames will be added.



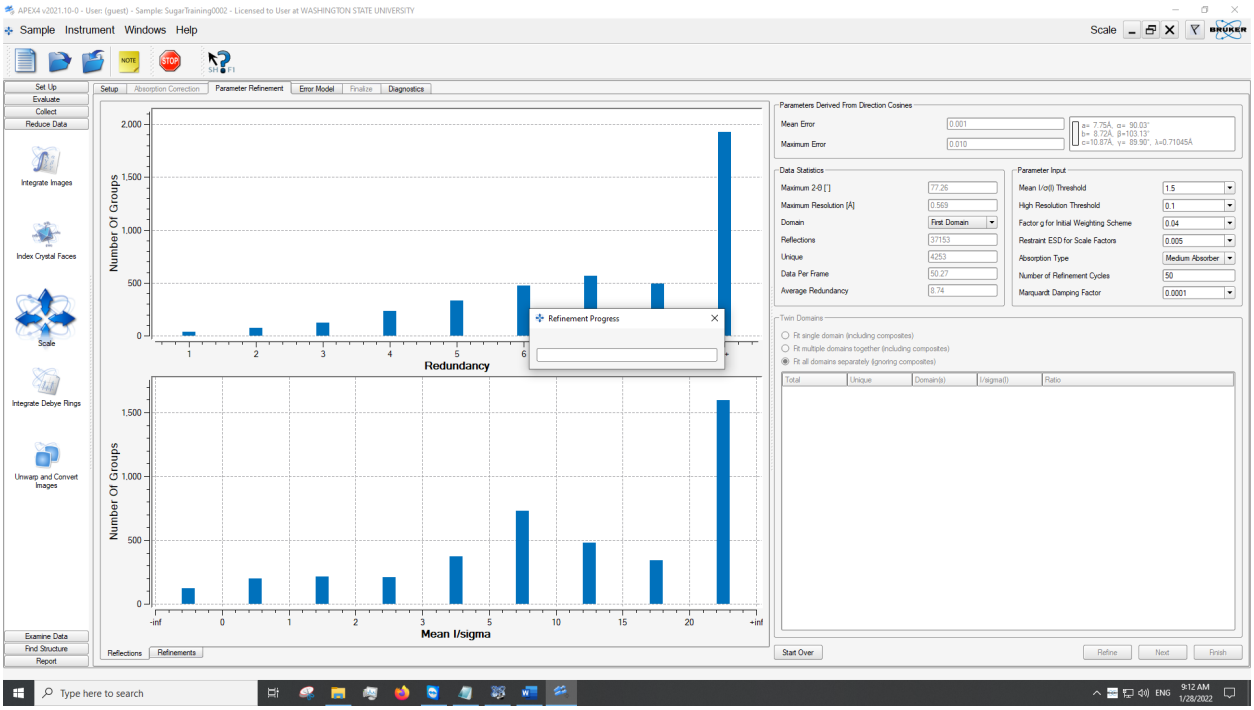
6.75 Once the frames have been added, make sure that all of the frames have been checked to have them scaled. Check the Laue groups and Point groups. Also select Multi-scan for the absorption correction (highlighted in red). Click on the Start button to walk through the steps or the Finish button to go through all of the steps automatically (if chosen, skip to Step 6.81).



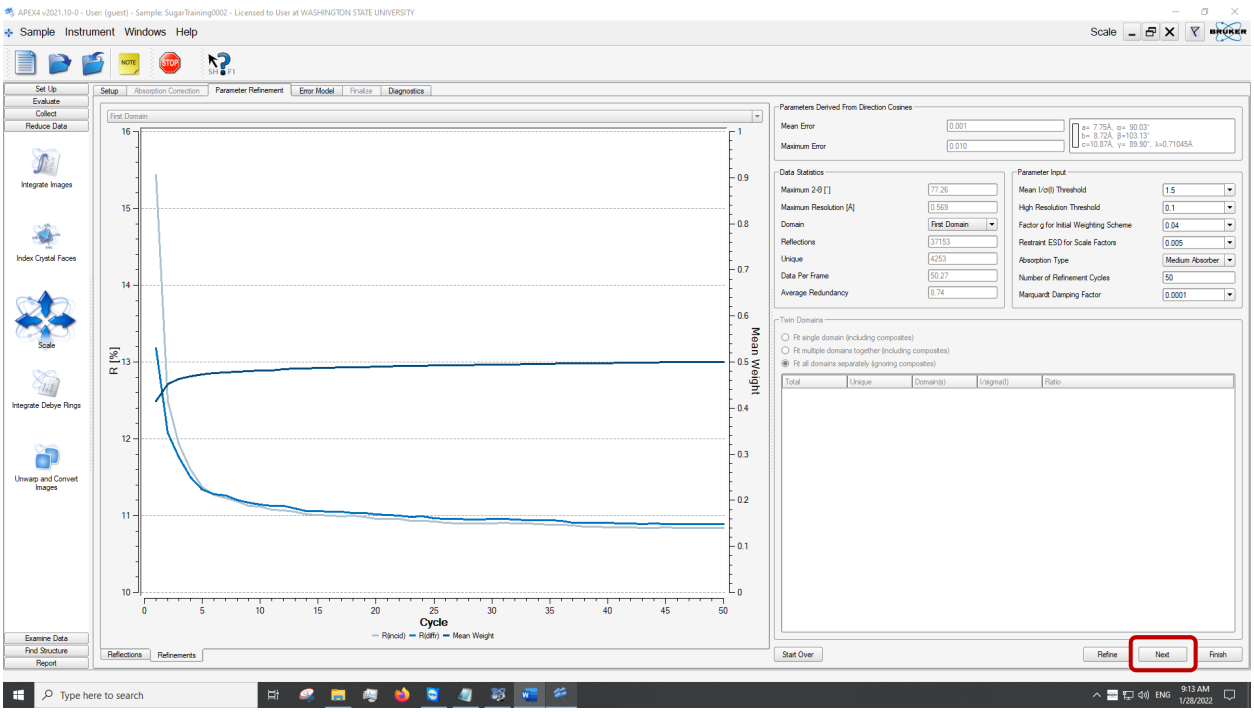
6.76 Upon clicking the Start button, a window showing the Redundancy and the Mean I/sigma will be shown. The bars should be the largest on the right side. Click on the Refine button.



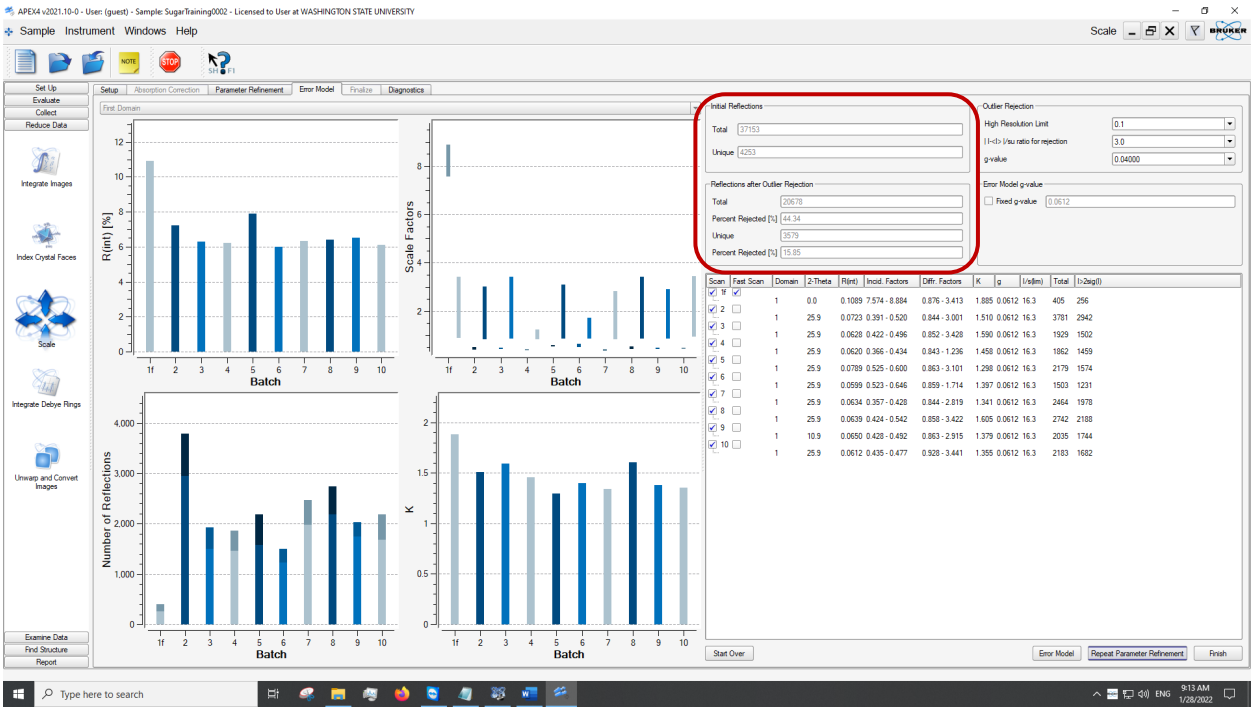
6.77 Upon clicking the Refine button, a window showing the Refinement process is occurring will show up. The refinement will take 1-5 minutes, depending on how much data was collected.



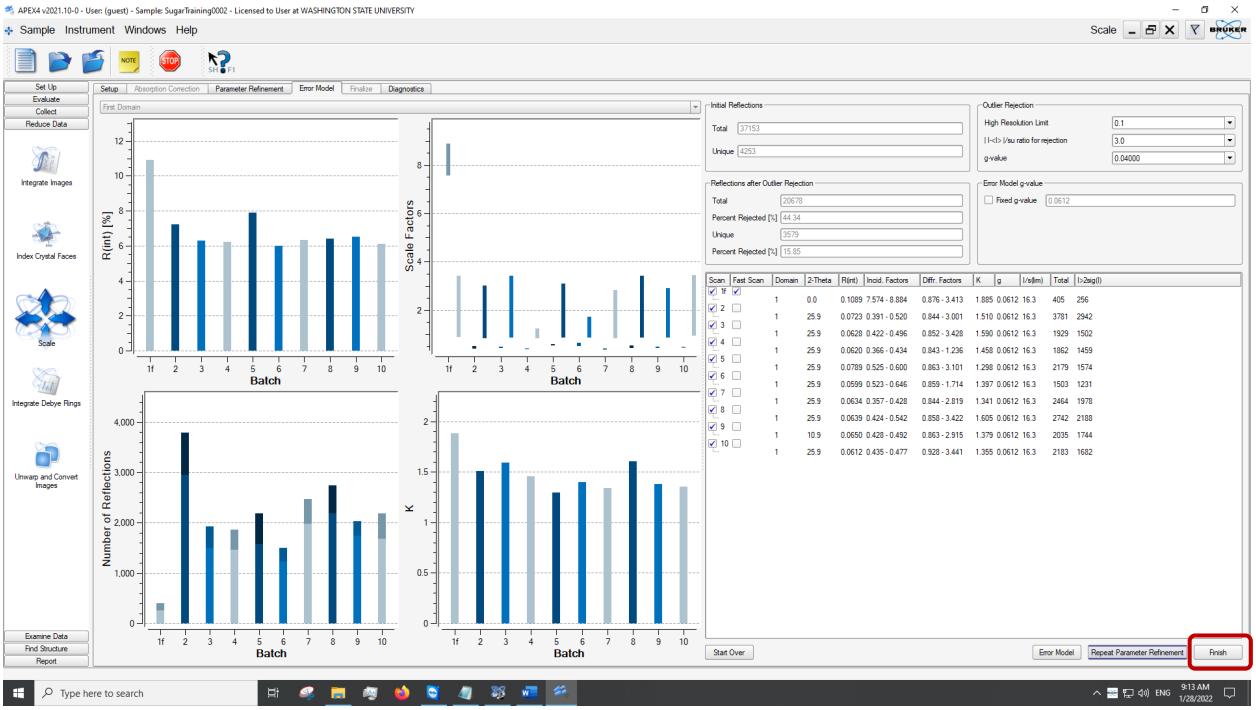
6.78 After the refinement has completed a plot of R% and Mean Weights will be displayed. A line that has an asymptote with a lower R[%] is better. A preferred value is below 10%. Click on the Next button to examine the error model.



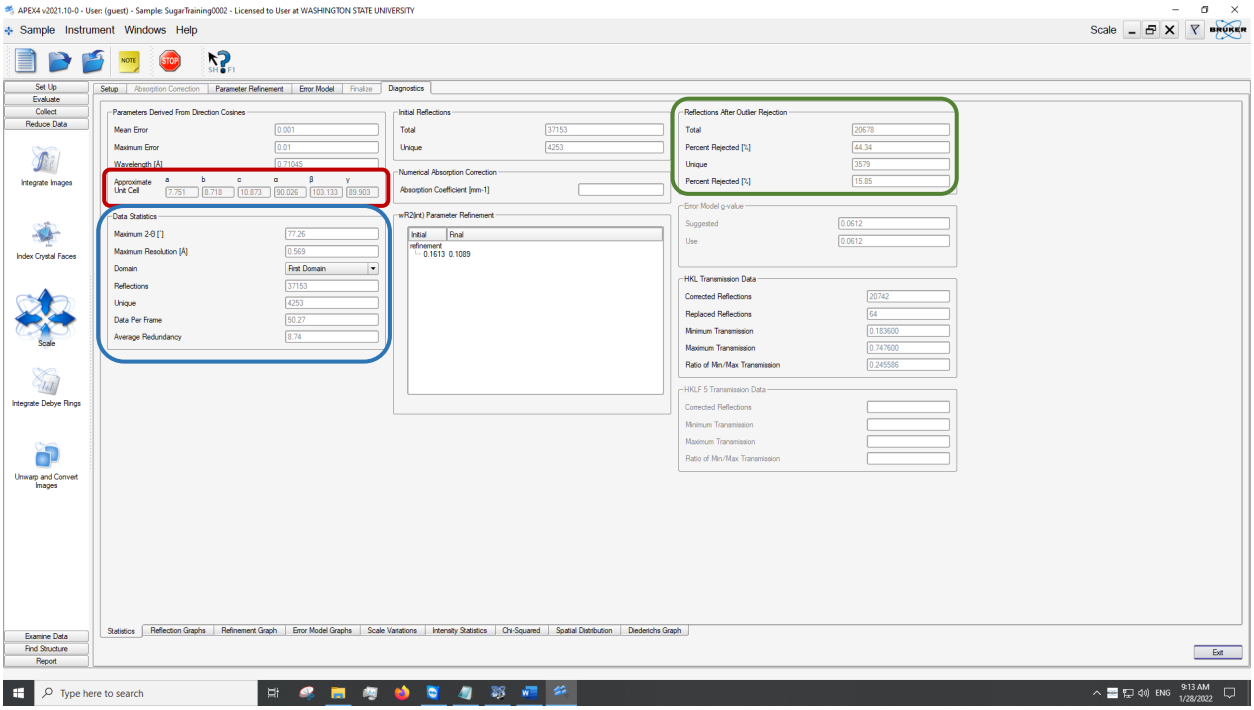
6.79 An example of the data obtained from an error model can be seen below. The total number of reflections, unique reflections, and rejected reflections can be seen (highlighted in red).



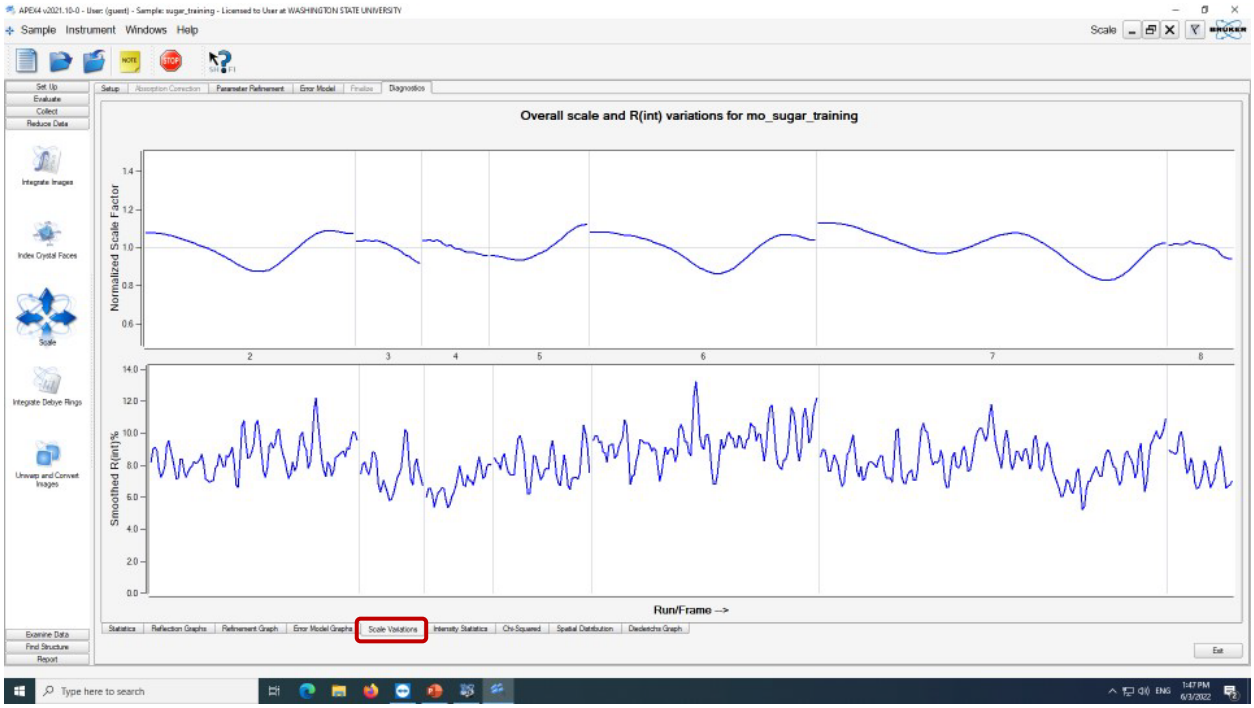
6.80 To finish the scaling procedure, click on the Finish button on the bottom right to generate the Diagnostics plots on the data analysis.



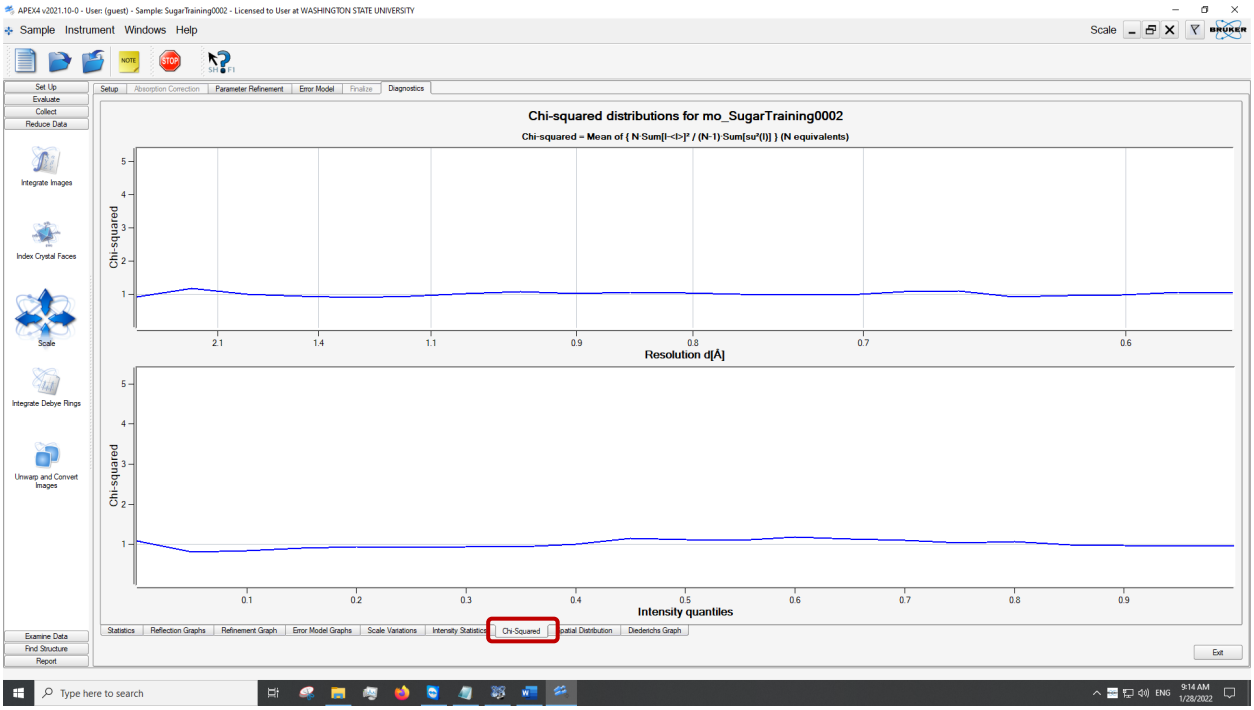
6.81 The Diagnostics window shows the approximate unit cell (highlighted in red), the total number of reflections and number of unique reflections (highlighted in blue), and the number of rejected reflections (highlighted in green).



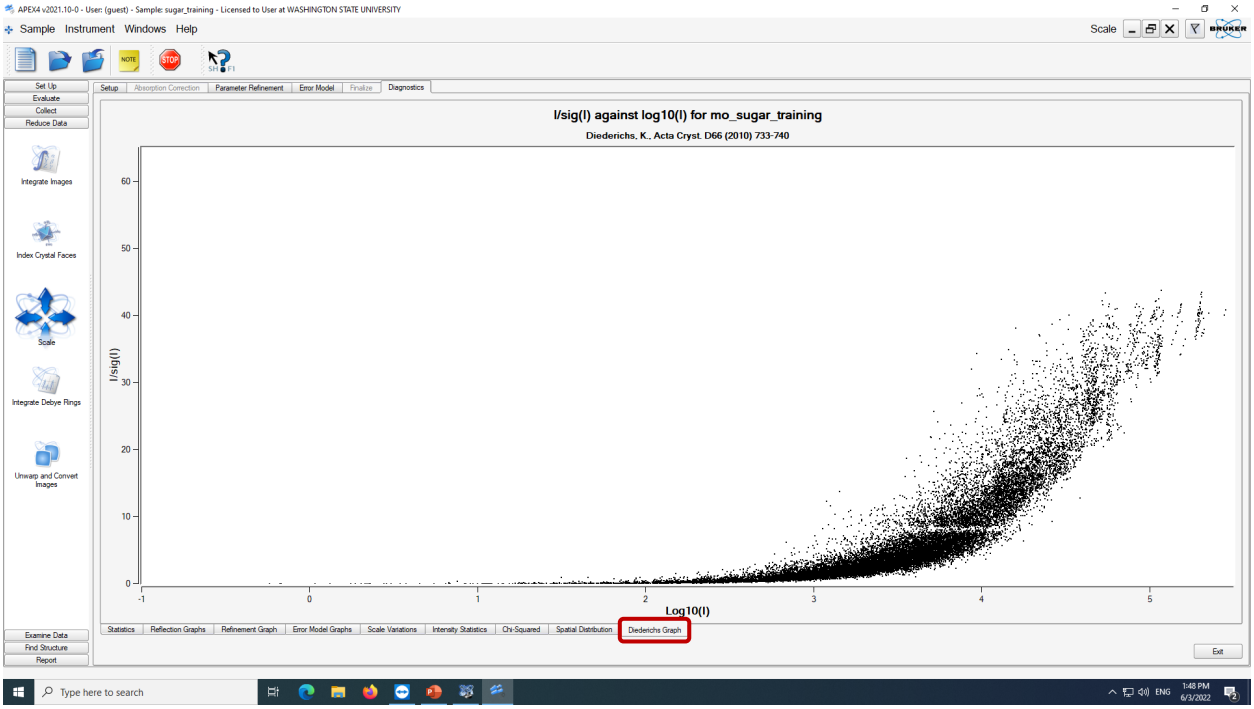
6.82 Before moving on the Scale Variations tab (highlighted in red) should be examined. There should not be large variations in the Normalized Scale Factor (see picture below).



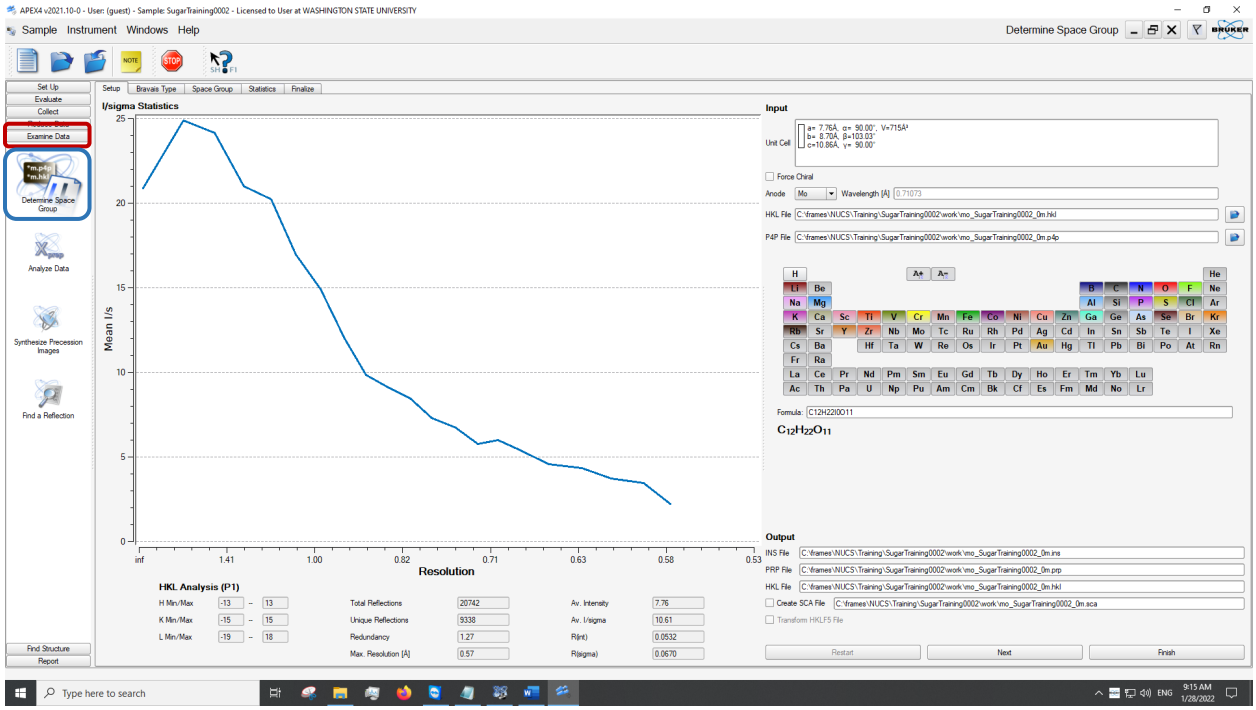
6.83 The Chi-Squared plot (highlighted in red) should also be examined. The values in the plot should be near one and not vary much, deviations away from one indicate a problem with the data.



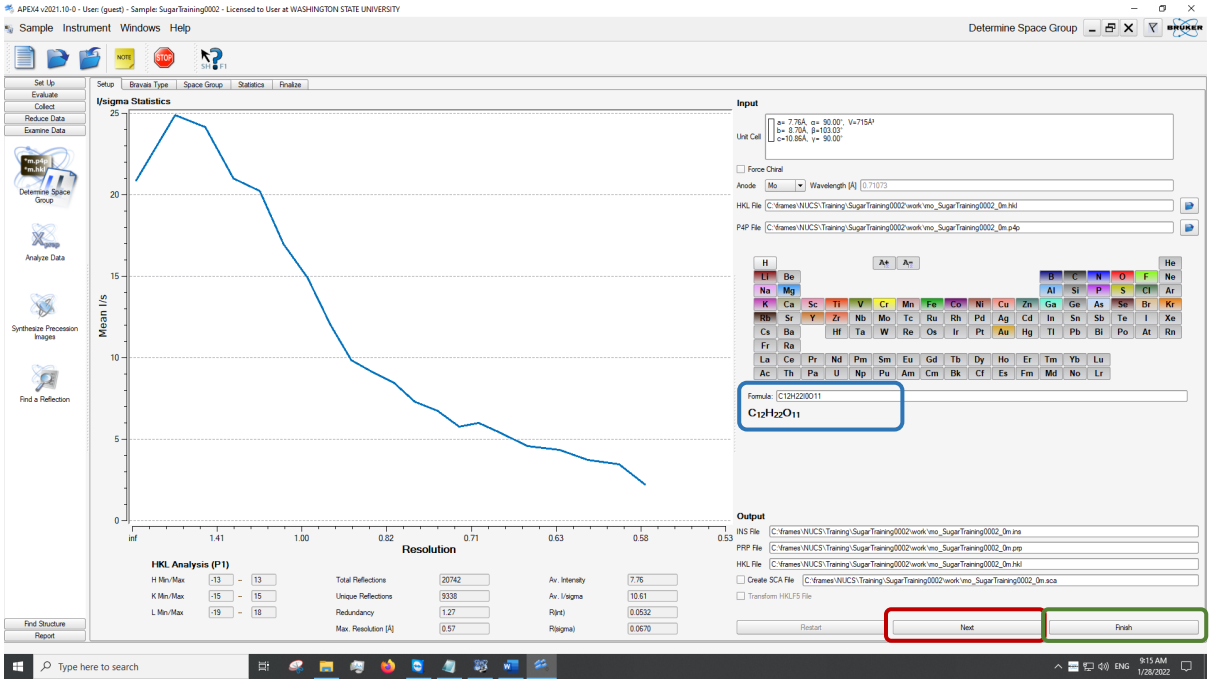
6.84 The last tab, labeled as the Diedrichs Graph (highlighted in red), should be examined. The plot should look sigmoidal in shape.



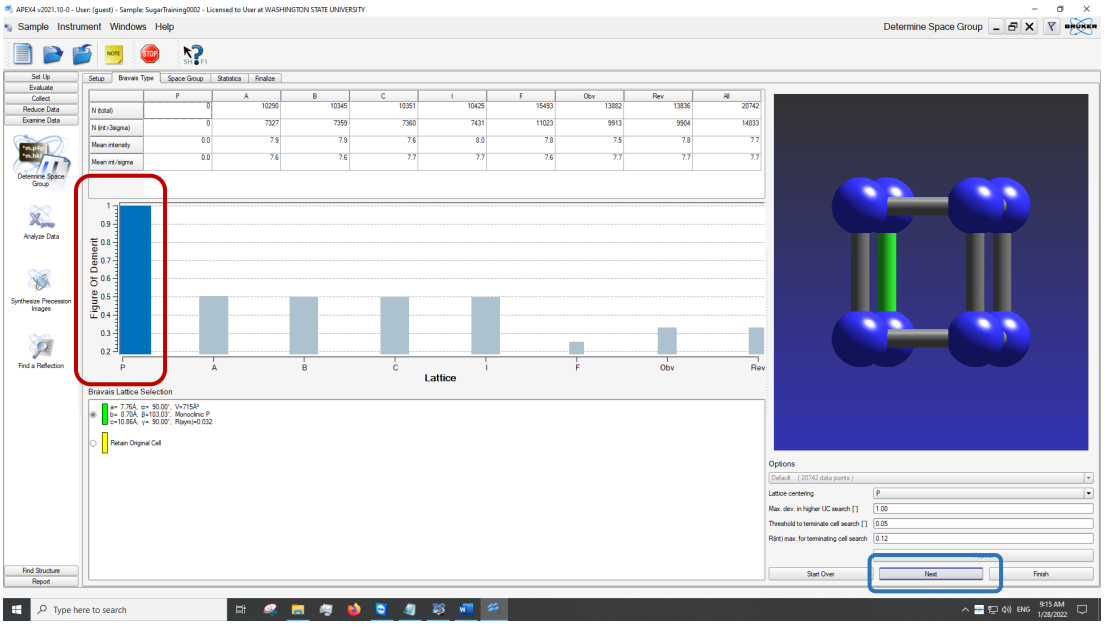
6.85 After scaling the data, the data will need to be examined if it is solvable. Click on the Examine Data button on the left side of the Apex 4 window (highlighted in red), followed by the Determine Space Group icon (highlighted in blue).



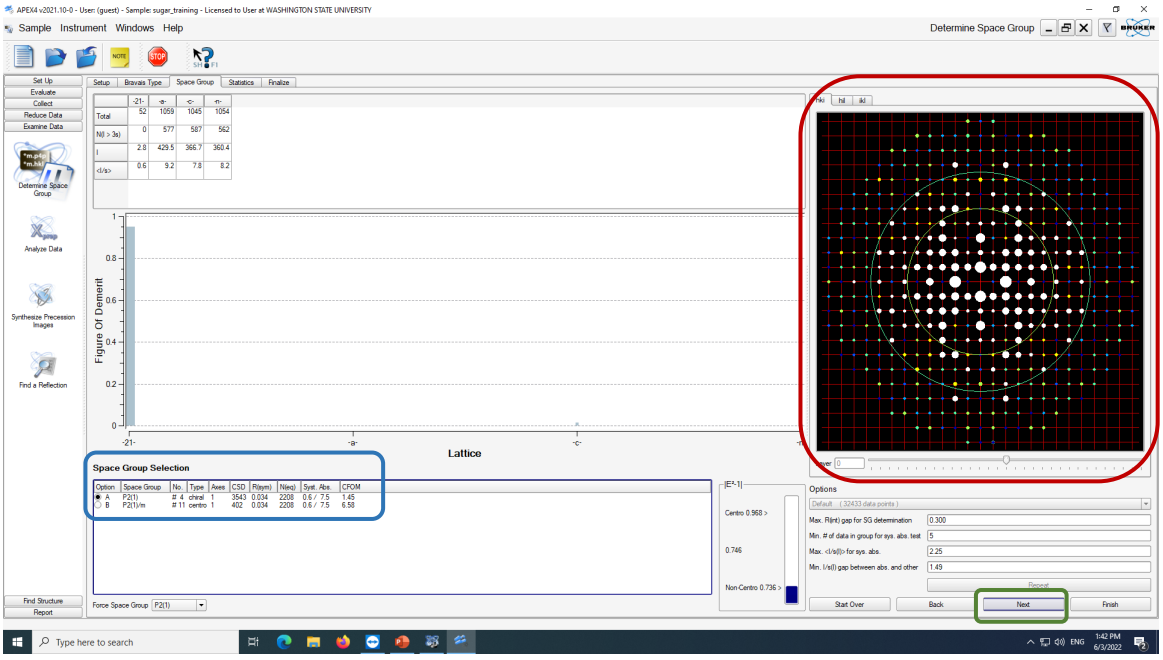
6.86 Before clicking on the Next button (highlighted in red) double check the chemical formula (highlighted in blue). It is not critical to have the correct formula at this time, but the elements that are present need to be in the formula and the closer the entered formula is to the formula of the final structure the easier it will be to solve the structure of the crystal. The Finish button (highlighted in green) can also be clicked to skip to Step 6.90.



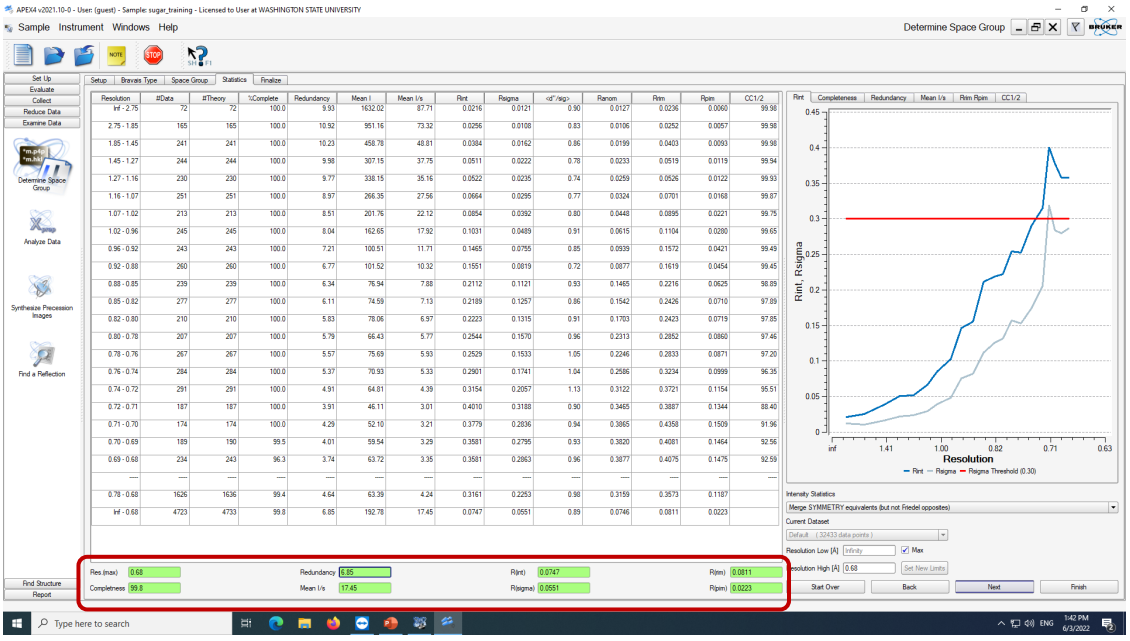
6.87 Click on the Next button to determine the Bravais type. The Bravais lattice chosen by the Apex4 software will be highlighted in dark blue (highlighted in red). If the default values of the Apex4 software are acceptable, the Next button can be clicked (highlighted in blue).



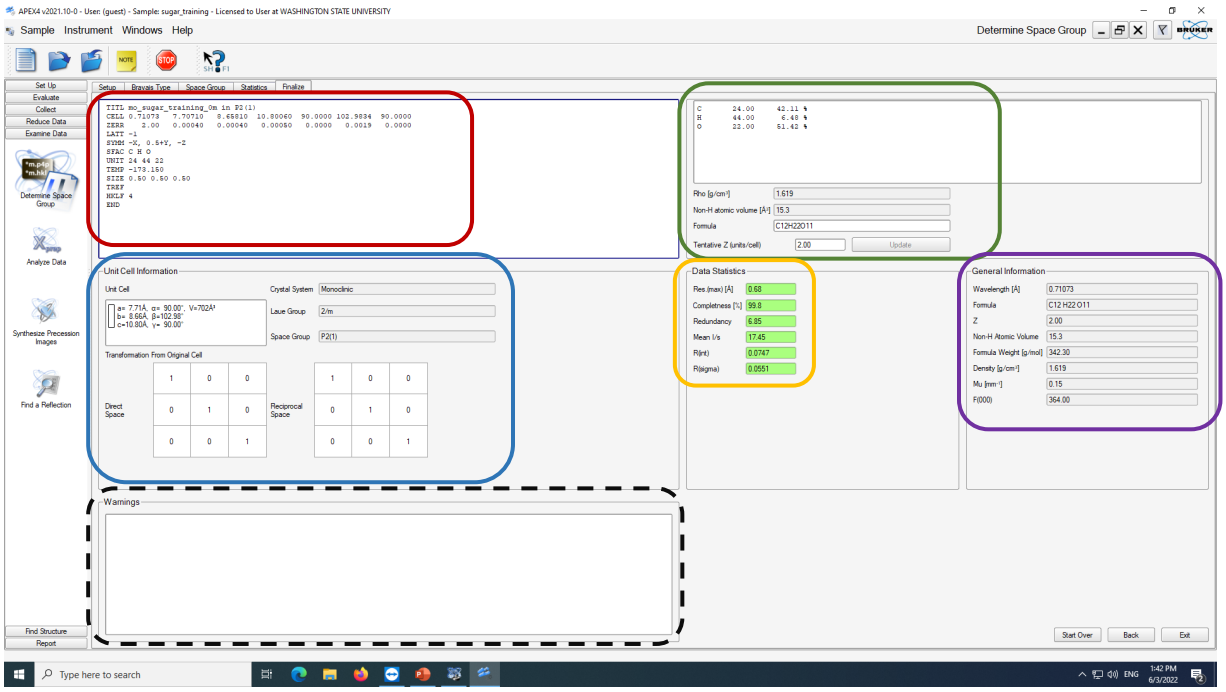
6.88 The next step determines the space group. The reciprocal lattice is shown in the top right picture (highlighted in red). If any of the dots are not at the intersection of the red lines, then a twinned crystal is suspected. To determine which space group is most correct, use the CFOM column (highlighted in blue). The lower CFOM (C Figure of Merit) is the more correct space group, so in this case, P2(1) is selected. The $|E^2-1|$ value also gives insight on whether the structure is centrosymmetric (contains an inversion center) ($|E^2-1|$ greater than 0.968) or non-centrosymmetric ($|E^2-1|$ around 0.736). When the desired space group has been selected, the Next button can be clicked (highlighted in green).



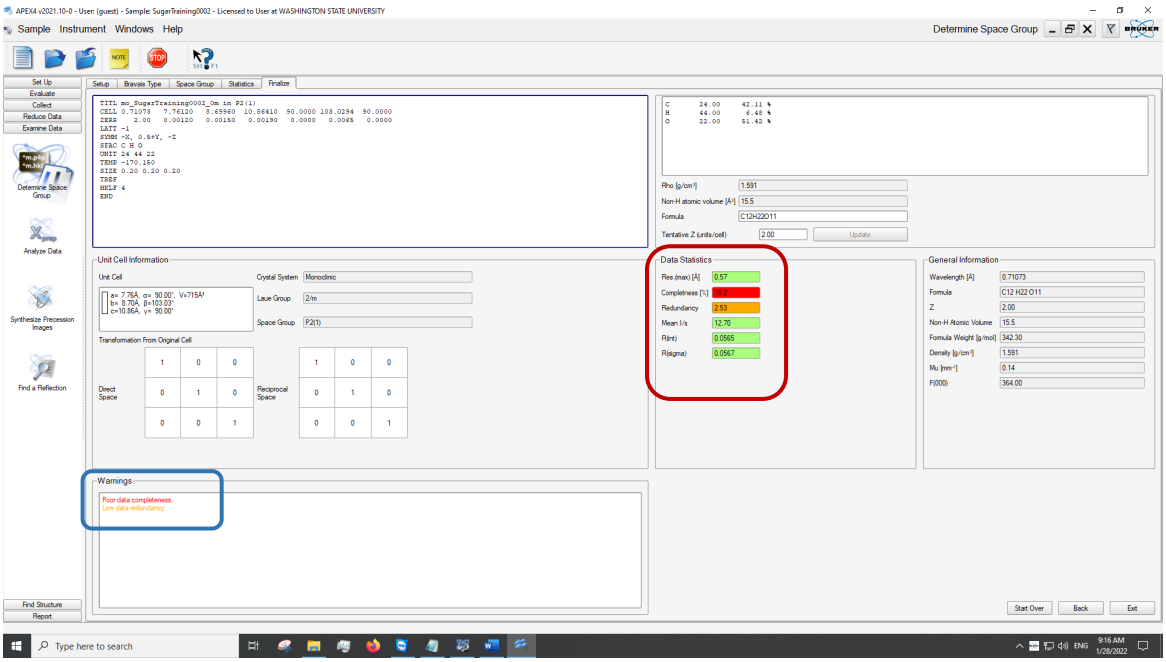
6.89 Upon clicking the Next button, the Statistics tab is displayed. The eight values (highlighted in red) should be highlighted in green. If they are highlighted in red or orange, there is a problem with the data. Click on the Next or Finish buttons to Finalize the data.



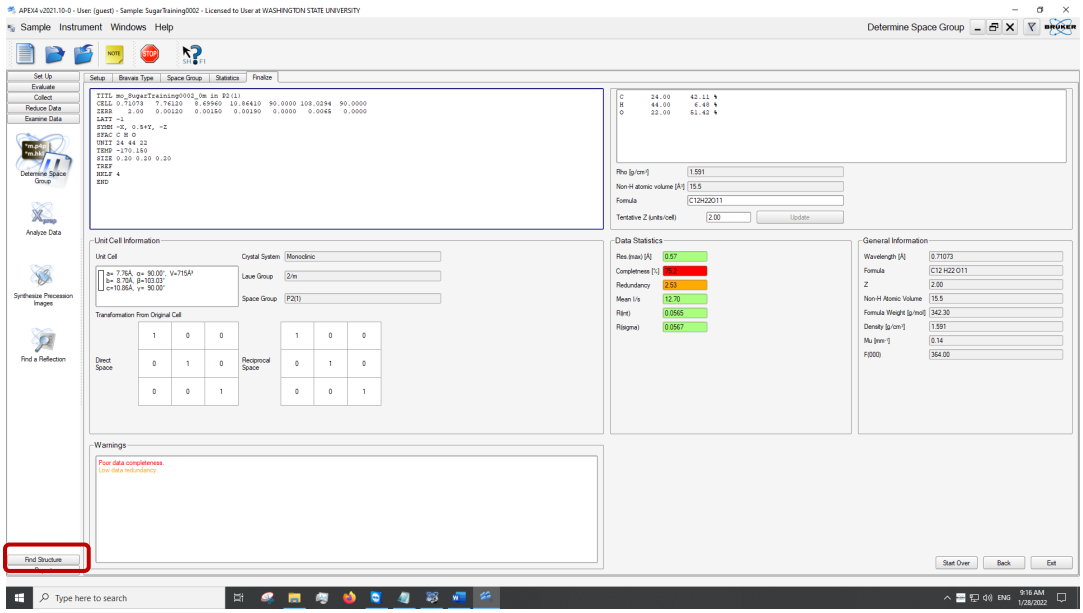
6.90 The Finalize tab lists the input file used to solve the structure (highlighted in red), the unit cell information (highlighted in blue), the respective formula and density (highlighted in green), the general information on the formula and formula weight (highlighted in purple), the statistics on the data (highlighted in orange), and whether there are any warnings (highlighted in dashed black).



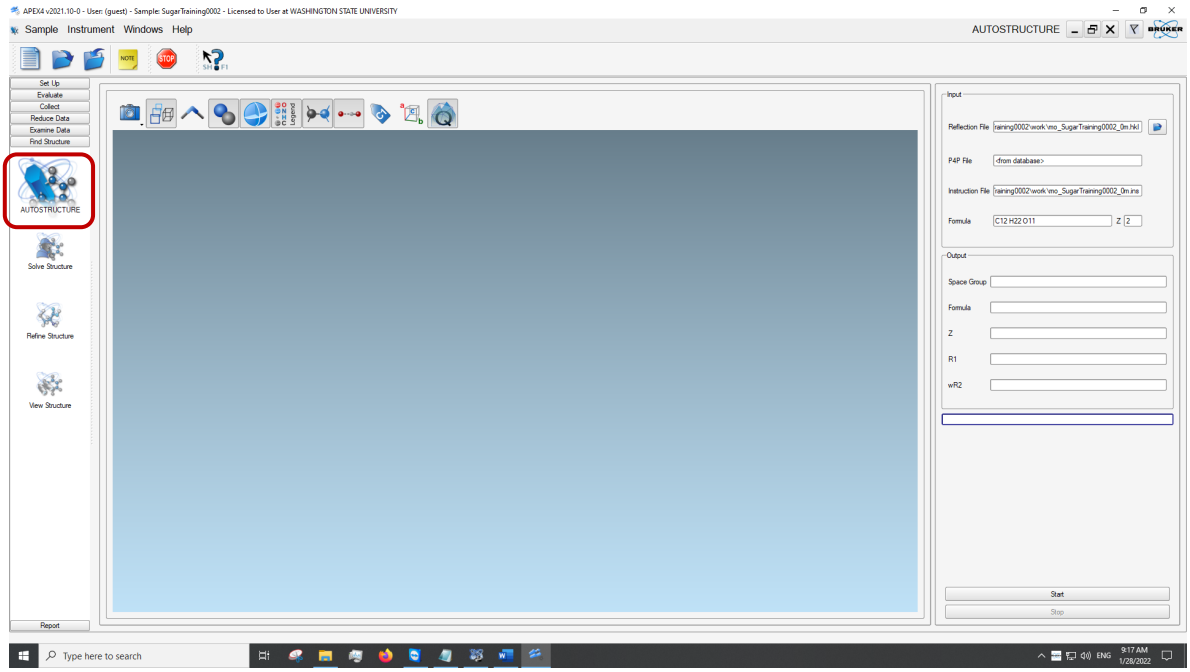
6.91 The Data Statistics section should all be highlighted green. If any of the entries are highlighted in orange or red, there is a problem with the data and another data collection is needed. An example of a problematic data set is seen below. In the example below, there is a poor completeness of the data and a poor redundancy. A possible solution is to pick a better crystal (this crystal was too big) and to collect the data at lower symmetry (see Step 6.47). All of the entries under the Data Statistics section need to be highlighted green to be able to generate a publishable structure.



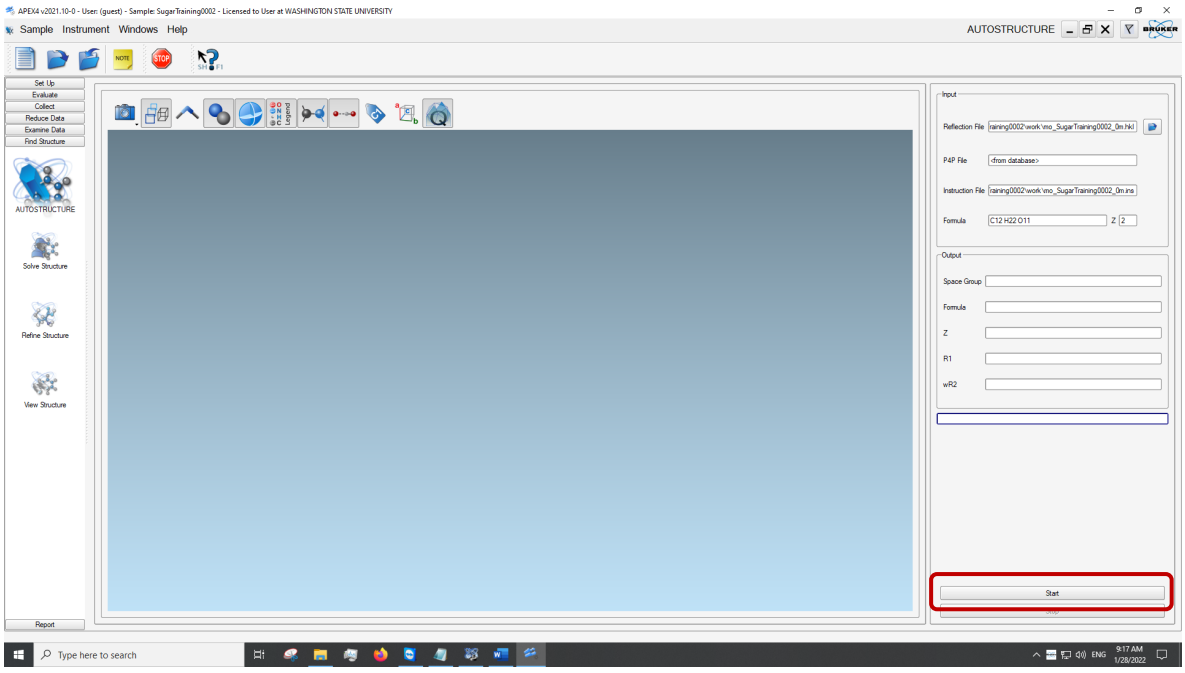
6.92 After the files have been set up, the Find structure button option on the right side of the Apex 4 window (highlighted in red) can be clicked to access the autosolve program or the data files in the Work folder of the data set can be opened in a structure solving program, such as Olex2.



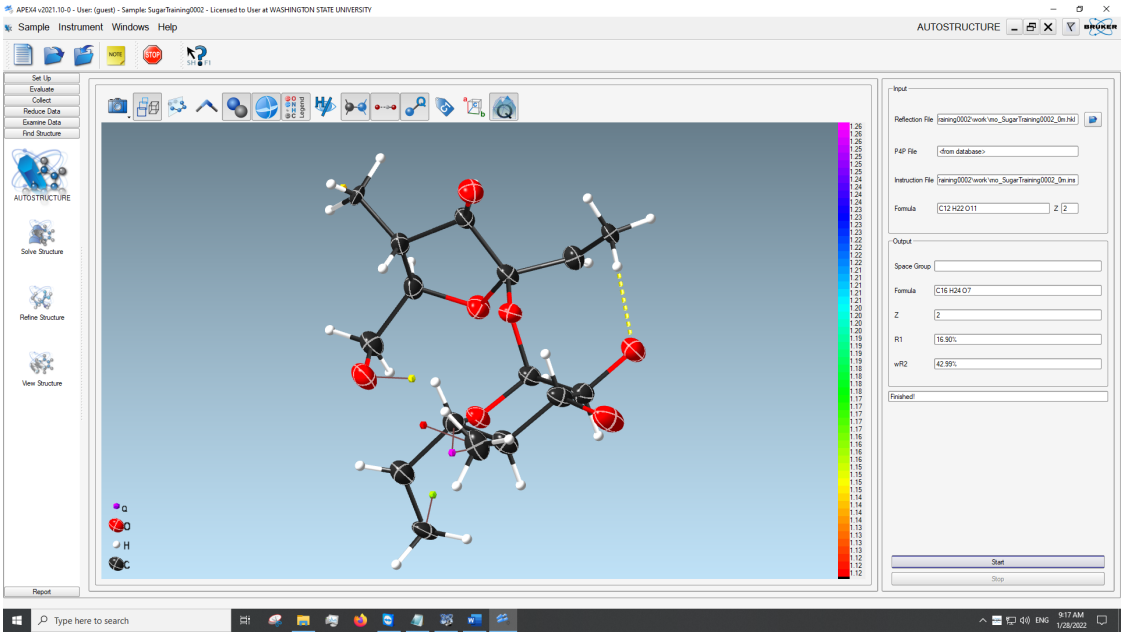
6.93 To solve the structure with the autosolve program, AUTOSTRUCTURE icon (highlighted in red), which will bring of the following window.



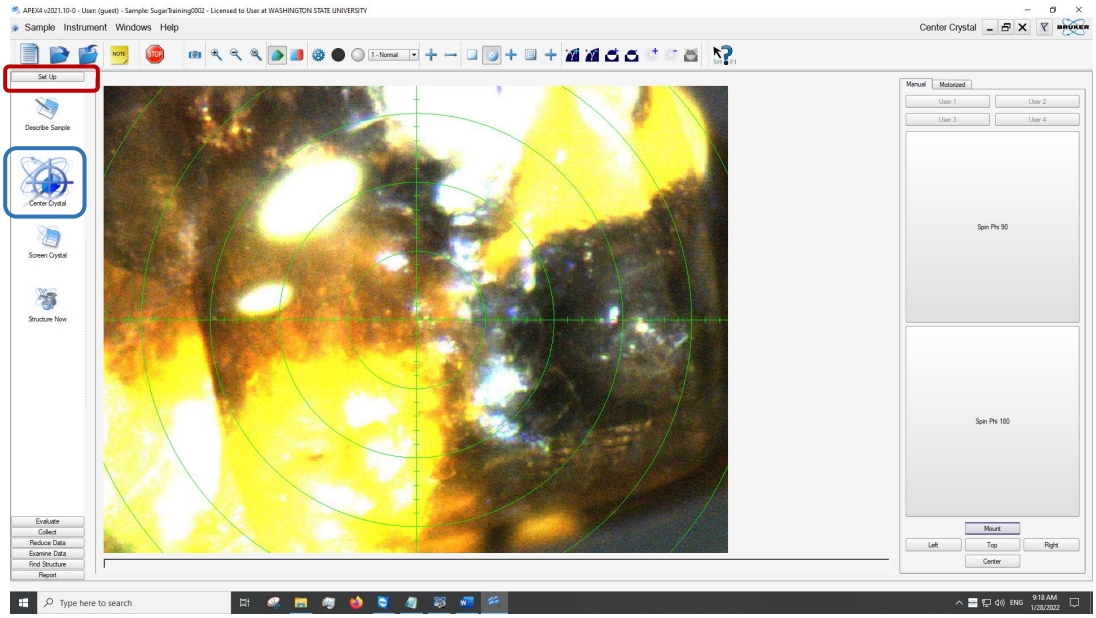
6.94 To start the autosolve program, click on the Start button (highlighted in red).



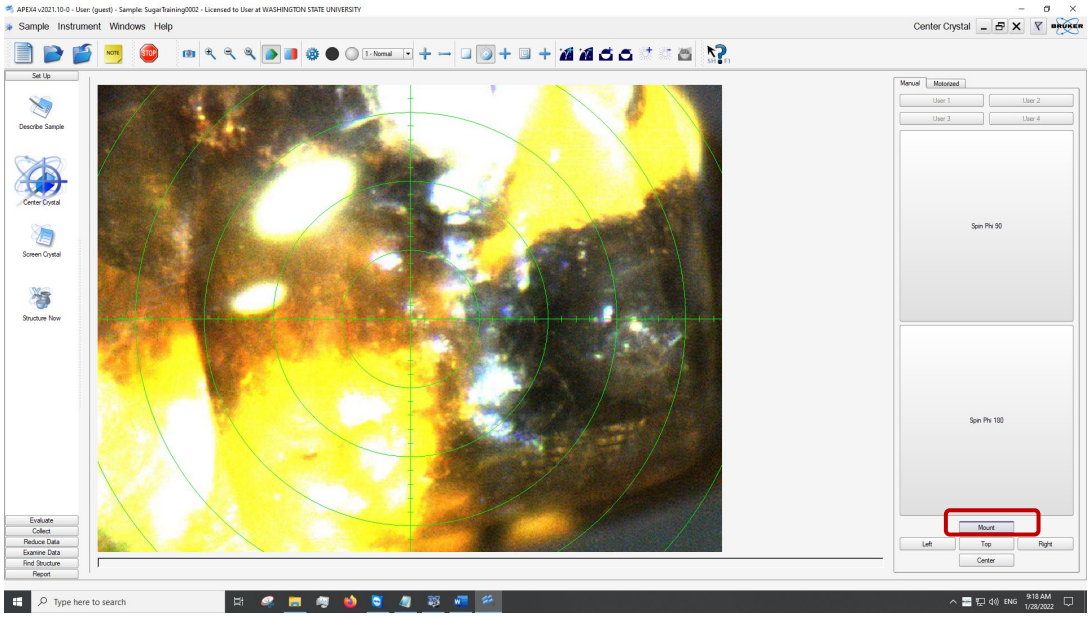
6.95 When the structure has been solved, a picture of the structure will show up in the central window with the resulting model parameters shown in the window. The R1 value will need to be below 8% to be publishable in most journals. Some journals will want an R1 value below 5 %. The lower the R1 value the better, as the value indicates how well the structure models the data. The autosolve program can misassign heteroatoms if there are several in a structure, so do not always believe the output. The autosolve program does a good job with simple organic compounds and it often used as a starting point to solve a structure in an external program (e.g. Olex2).

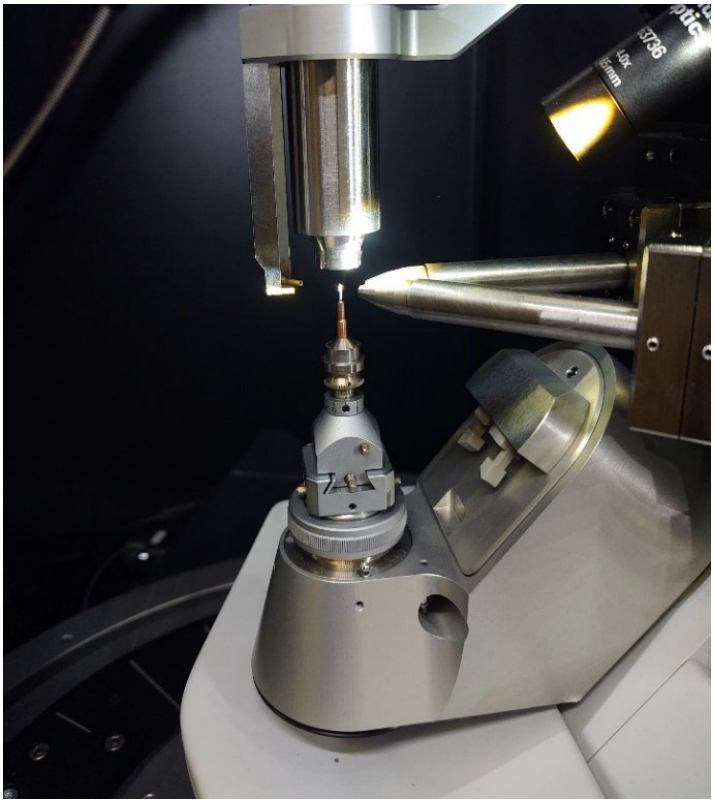


6.96 Once a solvable data set has been obtained and not further data collections are needed, click on the Set Up button (highlighted in red), followed by the Center Crystal icon (highlighted in blue).



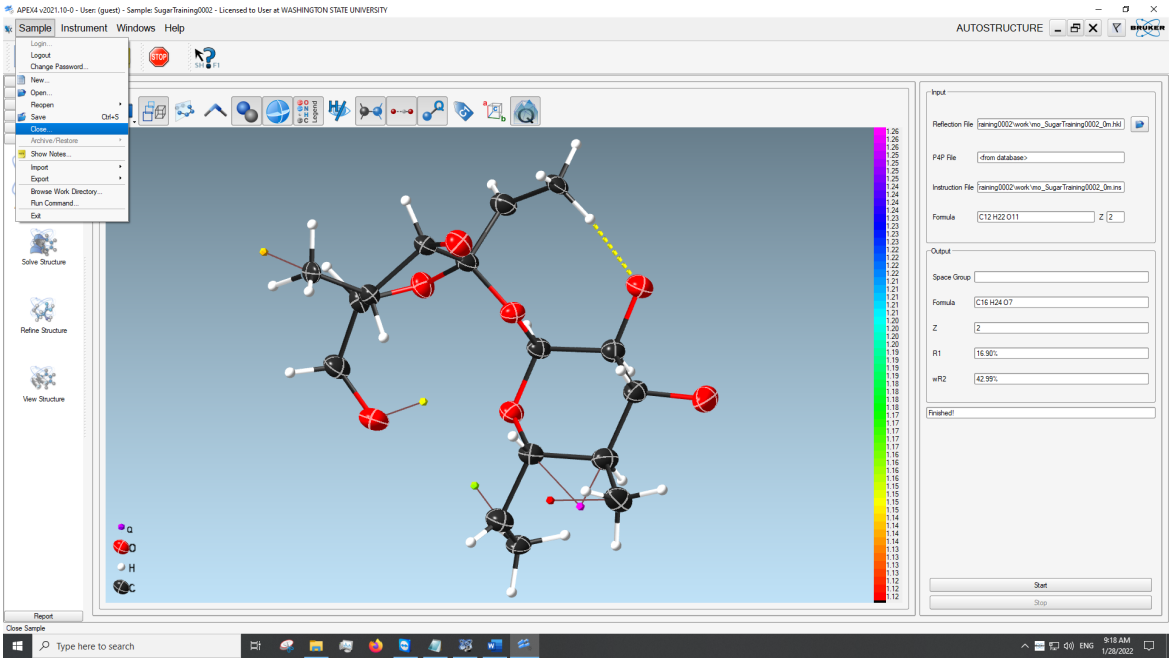
6.97 To move the crystal to a location so that the goniometer head and sample can be removed, click on the Mount button (highlighted in red) and wait until the detector is no longer moving and the goniometer looks like the picture seen below.



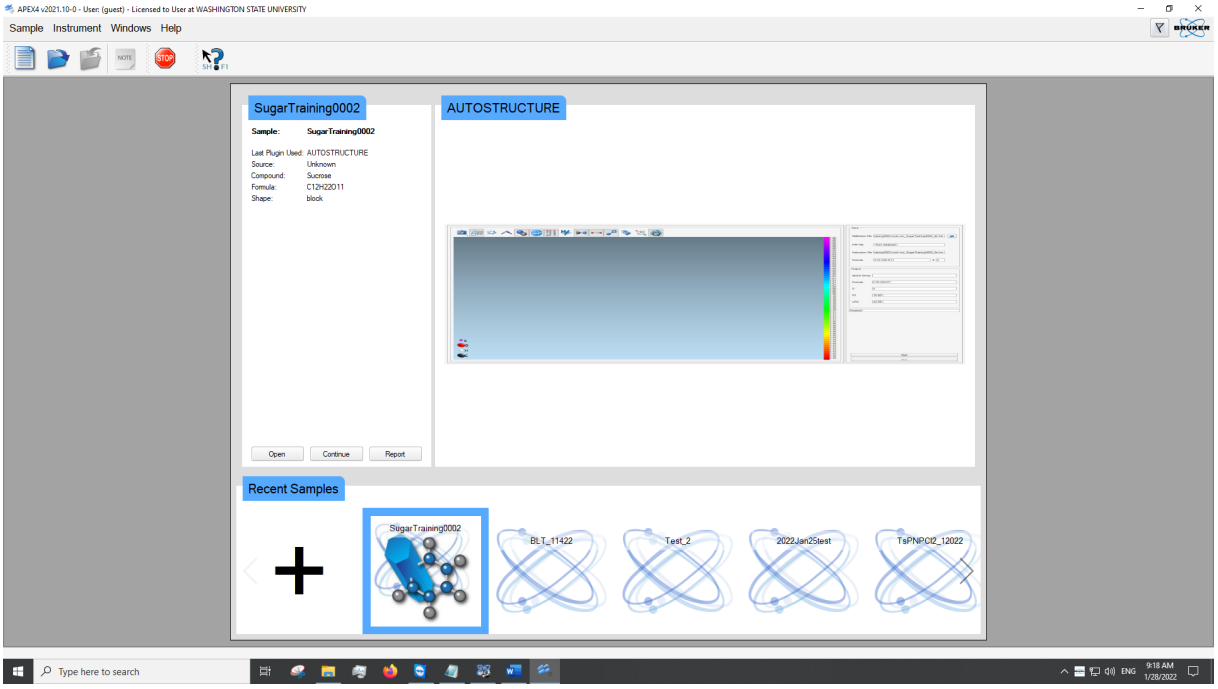


6.98 Open the doors to the diffractometer (see Step 6.17 for more info) and remove the sample from the goniometer.

6.99 Once the sample has been removed from the diffractometer, please go to Sample-> Close and close the data collection so that the instrument is available for the next user. The software will ask if the data is to be saved before closing, select Yes.



6.100 The following window should be visible when the data collection for the sample has been properly closed and the instrument is available for the next user.



7 Operation of the Oxford System Cryostream

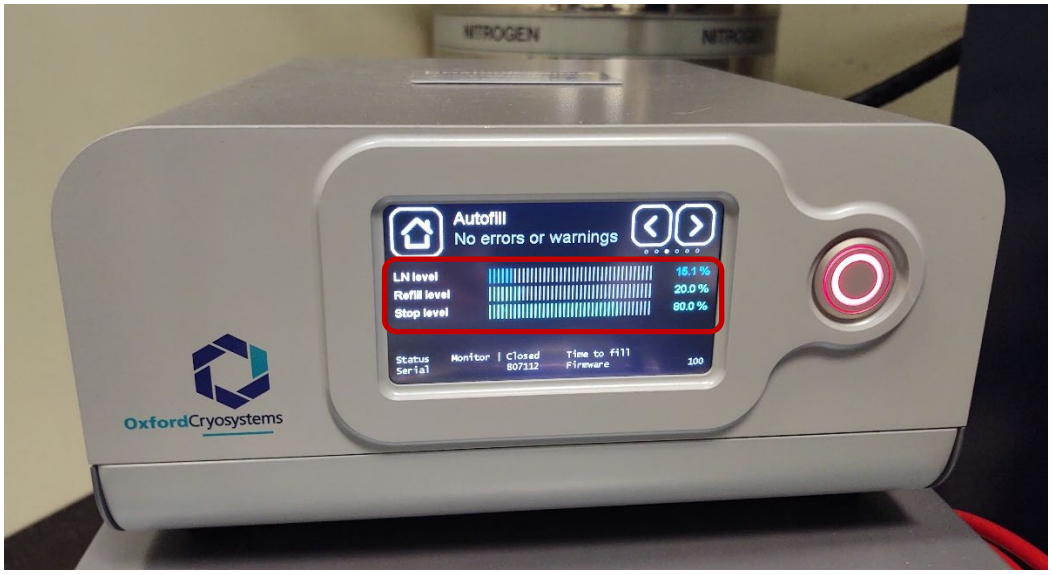
7.1 To collect data on a crystal below room temperature, the Oxford Cryostream is used (highlighted in red).

7.2 Before starting the cryostream, the level in the liquid nitrogen dewar (highlighted in red) needs to be checked.

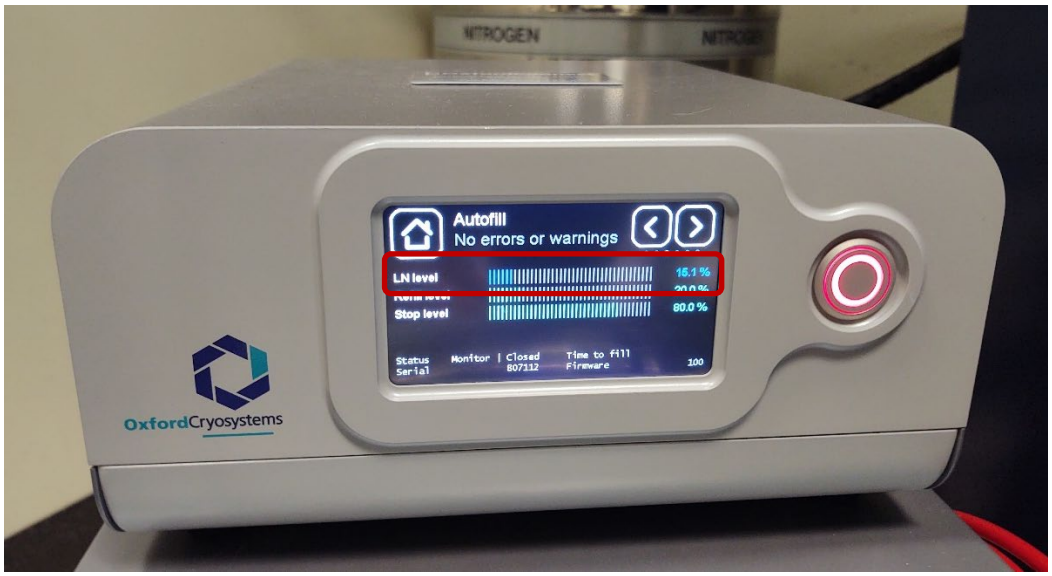
7.3 The liquid nitrogen level is checked by scrolling through the options on the touch screen user interface (highlighted in red) on top of the air dryer.

7.4 To scroll through the screens, press the icon in the top left corner (highlighted in red) of the touch screen.

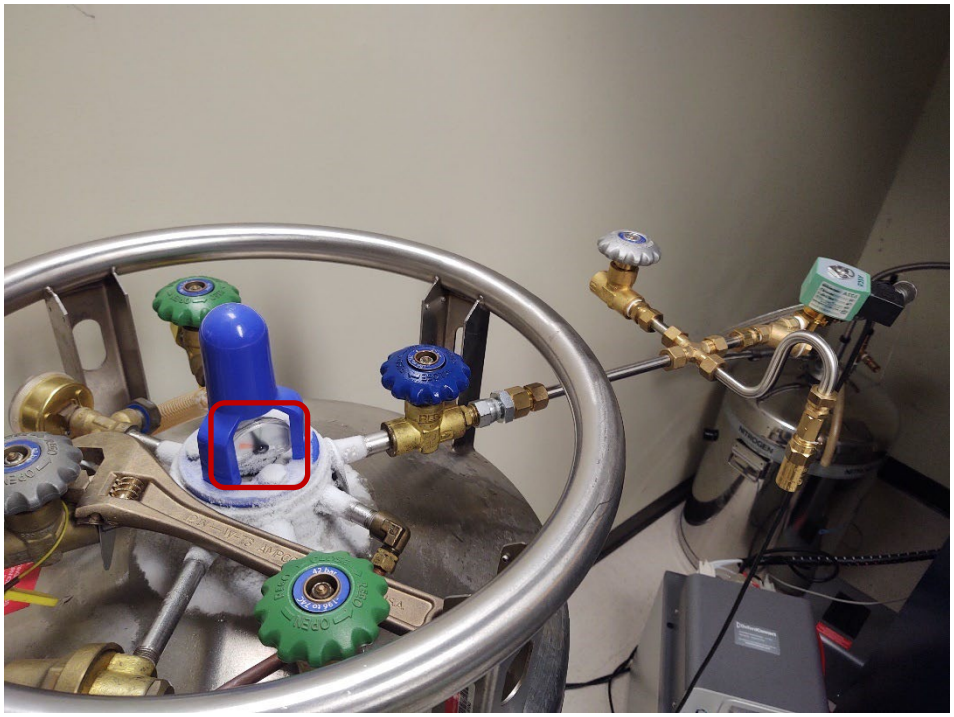
7.5 The screen will change to a screen describing the device information. Press on of the arrows in the top right corner (highlighted in red) until the screen reads Autofill and shows the level of the liquid nitrogen in the cryostream dewar (highlighted in red in the bottom picture).



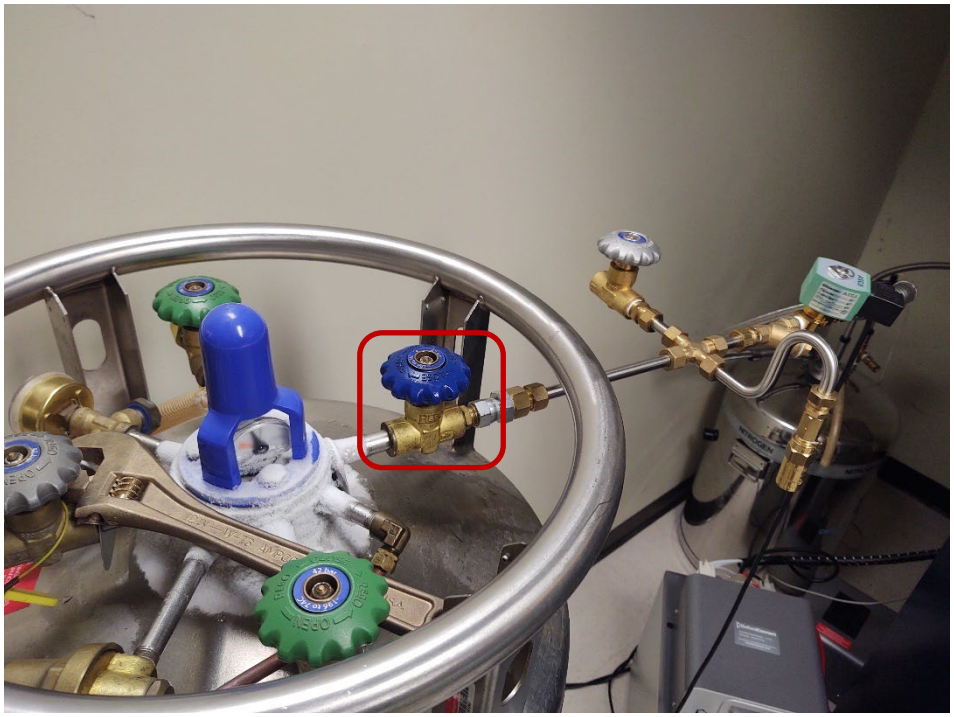
7.6 If the liquid nitrogen level is lower than 20%, then liquid nitrogen should be added to the dewar. A 15 hour run will require about 20% of the dewar, and the dewar should be at least 40% before the data collection is started. The liquid nitrogen level shown in the picture below is 15.1 %. If liquid nitrogen does not need to be added, skip to Step 7.15.



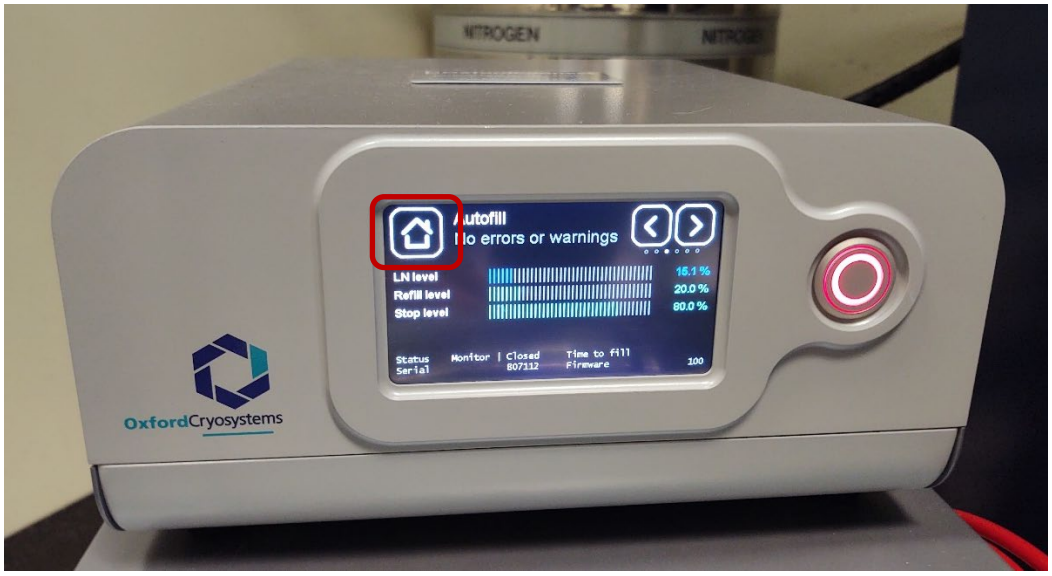
7.7 To add liquid nitrogen to the dewar, start by checking the amount of liquid nitrogen in the liquid nitrogen dewar connected to the Oxford Cryostream. If the dewar is empty, please inform NUCS Core Facility staff.



7.8 To fill the cryostream dewar, start by opening the valve of the liquid nitrogen dewar (highlighted in red).



7.9 Then, move to the cryostream control, and press the icon in the top left of the touch screen (highlighted in red).



7.10 Toggle through the screens by pressing the arrows in the top right corner until the screen reads: Set Autofill mode (see below picture).

7.11 To add liquid nitrogen to the cryostream dewar, press the Fill button on the touch screen (highlighted in red). The sound of valve opening can be heard and the cryostream dewar will start to be filled.

7.12 As the cryostream dear is filled, check to make sure the vent (highlighted in red) is not aimed at the electronics and aim it to point at the wall. The picture below depicts what the set up will look like at the beginning of the fill, showing the vent tube pointing down. It will need to be turned to the side to avoid getting ice on the electronics behind the diffractometer.

7.13 The cryostream is then filled until the dewar has achieved the desired nitrogen level. Please do not fill the cryostream dewar completely unless a data collection over several days is planned. To see the liquid nitrogen level of the cryostream dewar, please see Steps 7.5 & 7.6.

7.14 When the cryostream dewar has been filled to the desired level, press the Fill/Stop button to stop the fill. A sound closing a valve will be heard.

7.15 To start to cool the instrument, press the arrows in the top right of the touch screen to toggle to the Set Temperature window.

7.16 Most low temperature data collections are collected at 100K. If 100K is the desired temperature, skip to Step 7.18.

7.17 To adjust the temperature of the data collection, the temperature can be changed using the up or down arrows, or the toggle. Do not set the temperature below 100K.

7.18 To begin the cryostream, click the OK button when the desired temperature has been chosen.

7.19 A home screen showing the current temperature will be displayed. If this screen is not seen, press the icon in the top left corner.

7.20 To start the cryostream, press the green button on the front of the cryostream control. The cryostream will start. It will take about 15 minutes to cool from room temperature to 100K. While the cryostream is cooling, this is a good time to pick out a crystal if one is not already chosen.

8 Training

All users are required to provide the NUCS Core Facility Staff with records of completion of the WSU Radiation Safety Office Training Courses #1-7 & 10 (https://rso.wsu.edu/wsu-radiation-safety-training/), *prior* to being trained on the use of the Bruker D8 Venture Single Crystal X-ray Diffractometer.

Instrument Trainers

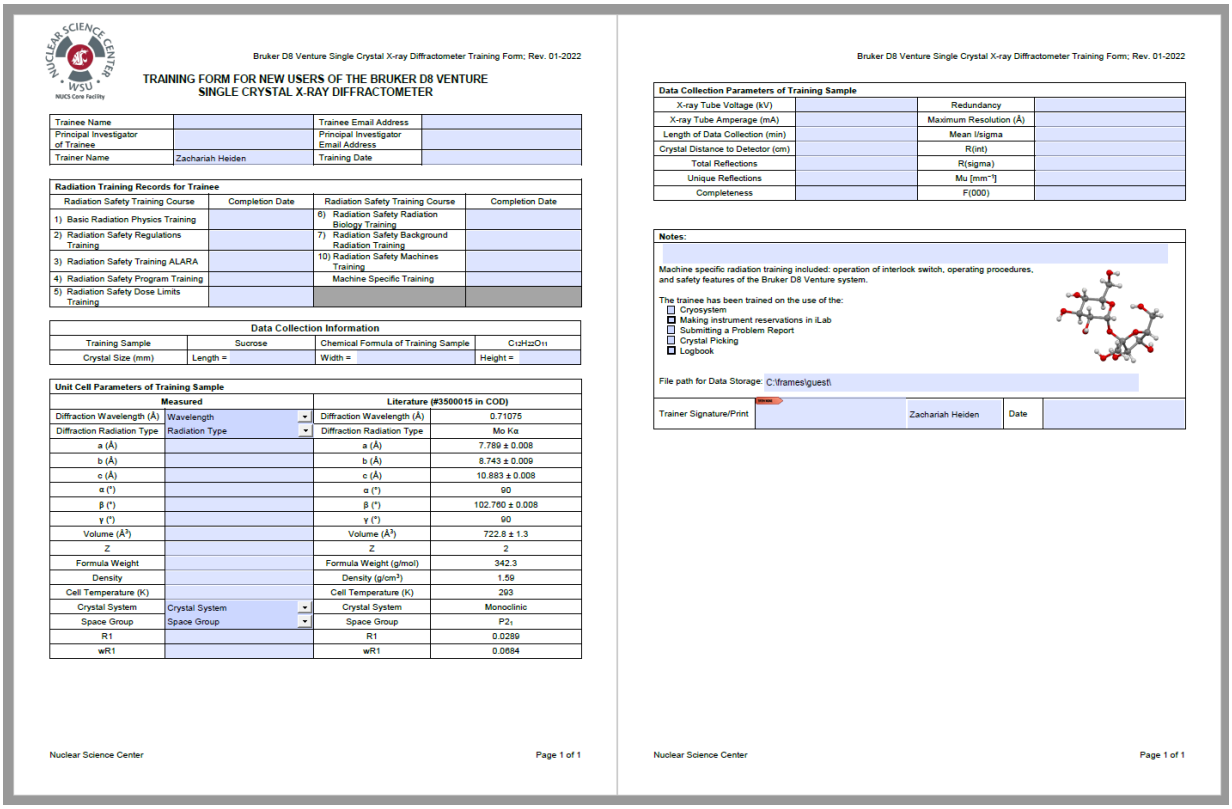
Zach Heiden, NUCS Fulmer, 509-335-0936

Bill Hiscox, NUCS Fulmer, 509-335-8259

Nuclear Science Center Emergency Line: 509-335-0004

Training on the Bruker D8 Venture Single Crystal X-ray Diffractometer consists of the safe use of the instrument, crystal picking of a sucrose sample, collection of a single dataset on a sucrose sample, using the Oxford Cryostream for low temperature data collections, data analysis using the Apex 4 software, discussion of the interlock system and safety features of the Bruker D8 Venture Single Crystal X-ray Diffractometer, making instrument reservations in iLab, submitting an instrument problem report, keeping records of use in the instrument logbook, and data transfer/access.

The form that is completed during a training session is seen below:



Bruker D8 Venture Single Crystal X-ray Diffractometer Training Form; Rev. 01-2022

TRAINING FORM FOR NEW USERS OF THE BRUKER D8 VENTURE SINGLE CRYSTAL X-RAY DIFFRACTOMETER

Trainee Name		Trainee Email Address	
Principal Investigator of Trainee		Principal Investigator Email Address	
Trainer Name	Zachariah Heiden	Training Date	

Radiation Training Records for Trainee			
Radiation Safety Training Course	Completion Date	Radiation Safety Training Course	Completion Date
1) Basic Radiation Physics Training		6) Radiation Safety Radiation Biology Training	
2) Radiation Safety Regulations Training		7) Radiation Safety Background Radiation Training	
3) Radiation Safety Training ALARA		10) Radiation Safety Machines Training	
4) Radiation Safety Program Training		Machine Specific Training	
5) Radiation Safety Dose Limits Training			

Data Collection Information			
Training Sample	Sucrose	Chemical Formula of Training Sample	$C_{12}H_{22}O_{11}$
Crystal Size (mm)	Length =	Width =	Height =

Unit Cell Parameters of Training Sample			
Measured		Literature (#3500015 in COD)	
Diffraction Wavelength (Å)	Wavelength	Diffraction Wavelength (Å)	0.71075
Diffraction Radiation Type	Radiation Type	Diffraction Radiation Type	Mo Kα
a (Å)		a (Å)	7.789 ± 0.008
b (Å)		b (Å)	8.743 ± 0.009
c (Å)		c (Å)	10.883 ± 0.008
α (°)		α (°)	90
β (°)		β (°)	102.760 ± 0.008
γ (°)		γ (°)	90
Volume (Å³)		Volume (Å³)	722.8 ± 1.3
Z		Z	2
Formula Weight		Formula Weight (g/mol)	342.3
Density		Density (g/cm³)	1.59
Cell Temperature (K)		Cell Temperature (K)	293
Crystal System	Crystal System	Crystal System	Monoclinic
Space Group	Space Group	Space Group	$P2_1$
R1		R1	0.0289
wR1		wR1	0.0684

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Bruker D8 Venture Single Crystal X-ray Diffractometer Training Form; Rev. 01-2022

Data Collection Parameters of Training Sample			
X-ray Tube Voltage (kV)		Redundancy	
X-ray Tube Amperage (mA)		Maximum Resolution (Å)	
Length of Data Collection (min)		Mean I/sigma	
Crystal Distance to Detector (cm)		R(int)	
Total Reflections		R(sigma)	
Unique Reflections		Mu [mm⁻¹]	
Completeness		F(000)	

Notes:

Machine specific radiation training included: operation of interlock switch, operating procedures, and safety features of the Bruker D8 Venture system.

The trainee has been trained on the use of the:
- ☐ Cryosystem
- ☐ Making instrument reservations in iLab
- ☐ Submitting a Problem Report
- ☐ Crystal Picking
- ☐ Logbook

File path for Data Storage: C:\frames\guest\

Trainer Signature/Print		Zachariah Heiden	Date

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