Oklahoma State University
College of Arts and Science

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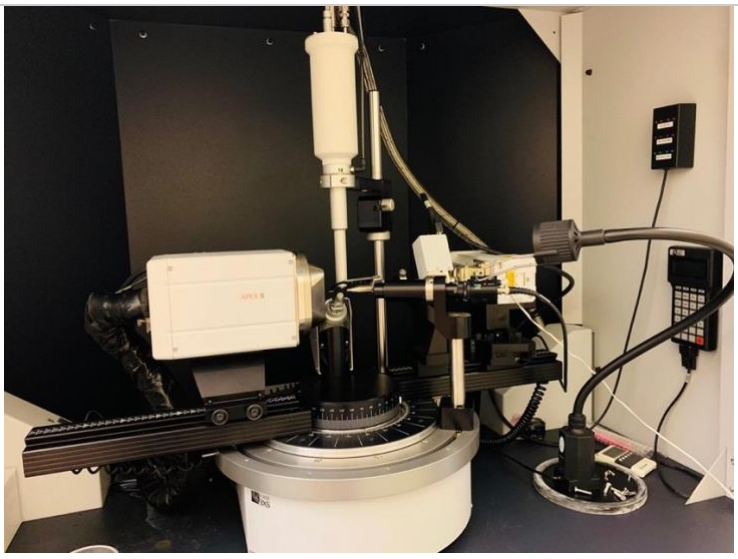
Bruker AXS Smart Apex II:
Single Crystal Diffractometer



Safety and Operation for the Bruker SMART APEX II system:

Locate the EMERGENCY STOP BUTTONS on either side of the machine. If you feel that something is not going correctly, press one of these buttons and the power will be turned off, thus disabling the X-rays. Inform **Dr. Reza Latifi** Immediately.

Safety Interlocks.

- The SMART APEX II system has an enclosure around the X-ray beam to prevent exposure.
- The enclosure is equipped with magnetic interlocks. If the interlocks are not engaged, the lead shutter will close, and no X-ray beam will be emitted. **NEVER** disable the interlocks! The interlocks are for your protection, but always be conscious of the path of the X-ray beam and avoid crossing it even when the shutter is closed.

Radiation Safety Training:

Supporting Documents:

- Radiation Safety Training Request Form (Start Here)
- Training Requirements
- Radiation Safety Training Documents
- URC Assistant Login

General Safety rules:

1. Closed-toe shoes and ankle-length clothes (outerwear) must be worn at all times in the laboratory (no sandals, crocs, shorts, etc.).
2. No eating or drinking in the laboratory. All consumable goods, including wrappers and containers, must be left outside of the laboratory.
3. Dosimetry (ring badge) is to be worn at all times when working within the X-ray room.
4. All users must have current record of laboratory safety class (or laboratory safety refresher) and Radiation Safety Training taken prior to be allowed to use X-ray facility.

5. If a laboratory accident occurs, respond to all health and safety concerns first.
6. Spills must be cleaned up properly. Inform Dr. Latifi of all accidents as soon as possible (Office: 405-744-4330, Cell phone: 857-399-5676; e-mail: reza.latifi@okstate.edu).
7. Waste produced from sample examination will be removed by the user from the X-ray room and properly disposed of in their home laboratory.
8. Highly toxic substances (toxicity grade 4) and samples which are explosive and/or flammable when in contact with air or moisture must be prepared for examination prior to entry to the X-ray room.
9. No laboratory items are to be taken into or disposed of in the X-ray room area.
10. User is not allowed to move or disconnect any major instrumentation in X-ray room. If there is a potential problem the user will inform the director.
11. Cryo-protective gloves and face-shield must be worn when transferring liquid nitrogen to the buffer Dewars from the mobile 180 L Dewar.
12. Radioactive or potentially radioactive samples are not permitted in the X-ray room.
13. Training and authorization by the X-ray director are required before use of any X-ray instrument. Users are not to train other people. Undergraduate students are not allowed to work alone without laboratory manager or authorized user supervision.
14. No repairs or modifications of instruments or computers will be performed by anyone but the X-ray director. This includes the installation of any personal software or games on X-ray room computers. Users must communicate to the X-ray director any errors or problems with instrumentation as soon as the problems occur.
15. It is the user's responsibility to backup data. The X-ray director will perform regular data removal from instrument and is not required to contact the user prior to data removal.
16. All instrumentation must be left clean and orderly. It is mandatory to remove all personal items and to clean up when finished with sample analysis. Storage of any items in the X-ray room is not allowed without permission.
17. Laboratory doors must be securely closed when departing (even if temporarily).
18. If a user's negligence or impropriety causes any damage to the X-ray room, access to the X-ray facility for that user will be restricted.
19. Failure to follow all required safety policies may result in restricting or banning user access to X-ray facility.

Radiation Dosimeter:

⇒ Wear your radiation badge all the time when you work in the X-ray lab.

To obtain the whole-body badge, please contact Brandi Simmons:

Brandi Simmons

Radiation Safety Officer
Oklahoma State University
University Research Compliance
215 Scott Hall
Stillwater, OK 74078
Phone: 405.744.7890
Fax: 405.744.4335
E-mail: brandi.simmons@okstate.edu

Link of **Radiation Safety Office:**

https://research.okstate.edu/compliance/rso/rso-contacts.html

Starting the Apex II system:

1. Make sure the Haskris water chiller is on and the building chilled water supply is flowing. Allow the temperature to stabilize (65-69 °F). The chiller is located in **150LC** room.

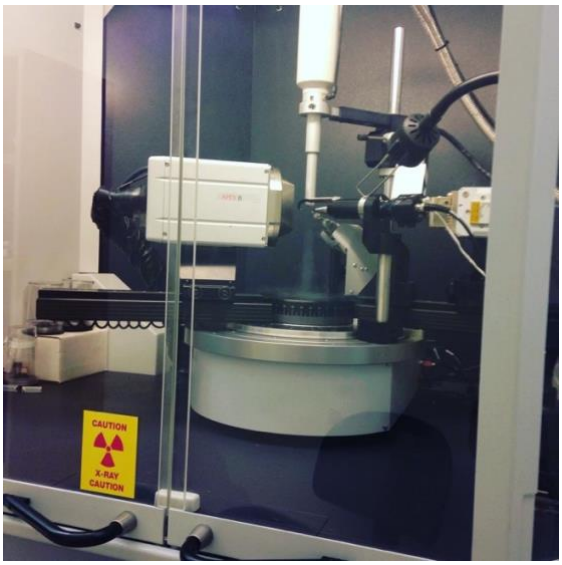
2. Turn on Lytron chiller and allow temperature to stabilize (between -6 to -9 °C). The chiller is located in **150LB** room.

3. Start computer and let it boot up (you must complete the x-ray machine training to receive the password).
4. Turn on the detector. The switch is behind lower front cover of enclosure (upper left)

5. Push the on button on the right column (circular green button labeled "I")- All 4 status lights will light.

6. All 4 lights will go out and then the ON light will light (and also the alarm light).

7. Check that the glass doors of the instrument are fully closed! Otherwise the next step will fail.



8. Flip the "High Voltage" switch by rotating it clockwise and holding until the X-ray light stops flashing.

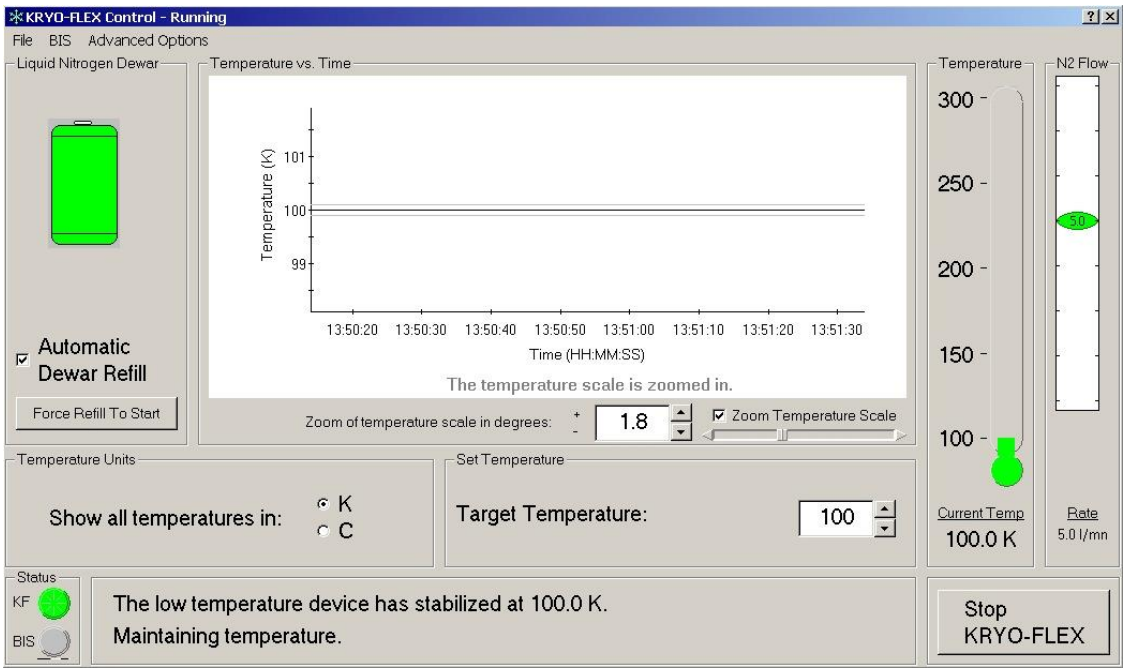
9. Confirm by noting that the X-ray light is lit at the upper corners of the machine.

On the computer:

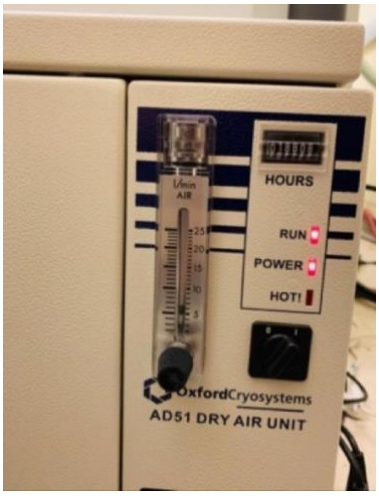
1. All but very high melting samples should be measured at lower temperature to avoid extensive thermal motion of the atom cores and to minimize radiation damage of the sample.

⇒ With the Smart Apex machine, the cooling unit is controlled by the program **KRYOFLEX** installed on the client computer. The sample is embedded in a stream of cold nitrogen gas supplied by a low pressure liquid nitrogen dewar.

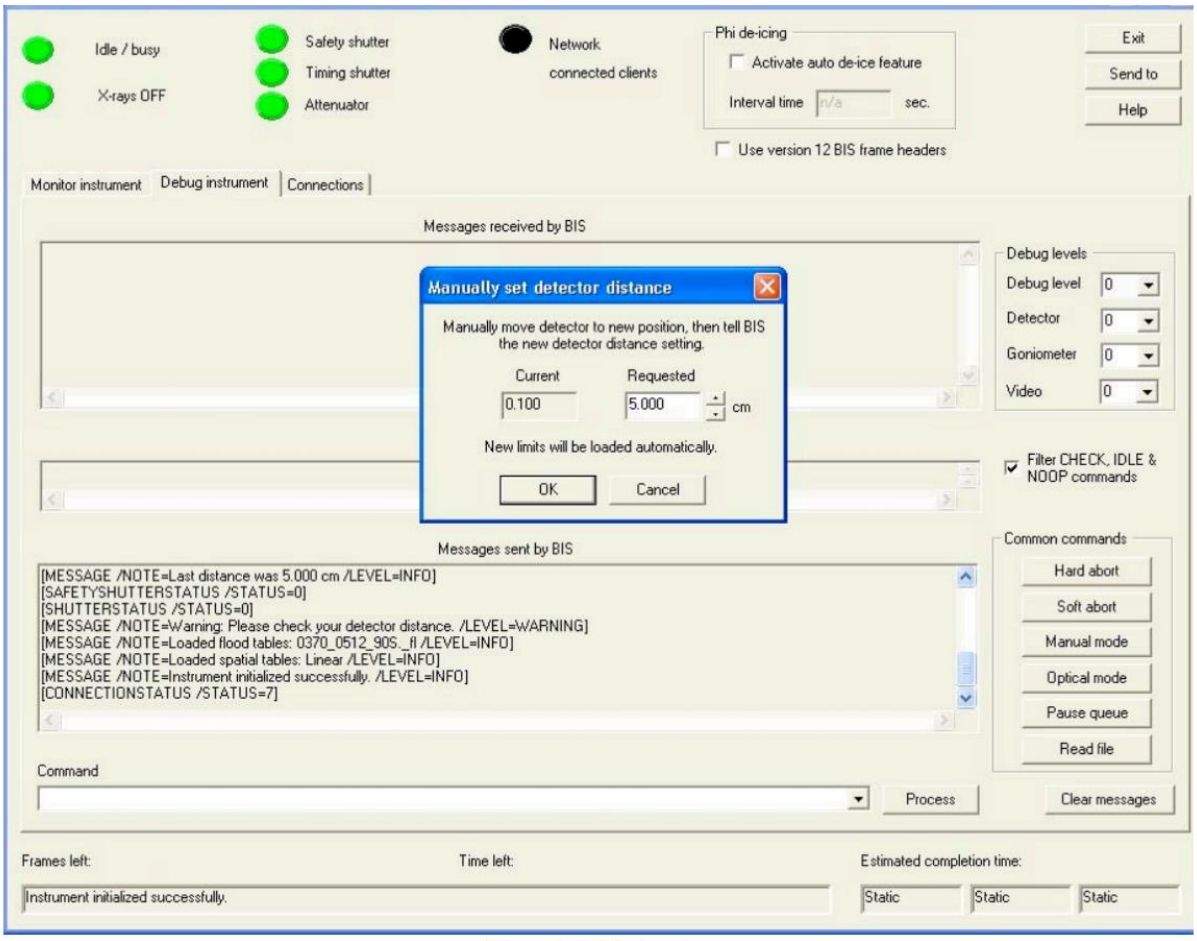
⇒ On the client computer open the **KRYOFLEX** program, wait some time until ready. Make sure, the valve of the liquid nitrogen tank is open and nitrogen is flowing.



2. To avoid buildup of ice, the sample is insulated from ambient humidity by an outer layer of warm nitrogen supplied by dry air unit. Make sure to turn it ON!



3. Start BIS - confirm detector distance is correct. If the detector distance is changed, BIS has to be restarted and the correct distance entered. {BIS has to be running but can be minimized)



To start BIS, choose **Start** > **Programs** > **Bruker AXS** > **Administration** > **BIS** or double-click BIS icon on the **Desktop**. After initialization, BIS asks for the detector distance as shown in the above figure. Click OK.

4. **Start APEX II**

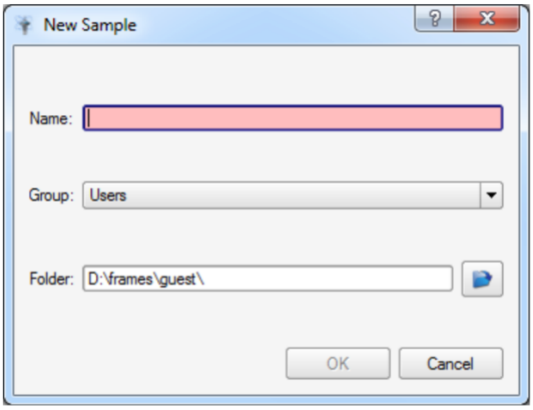
   a. Make sure connection is established (server is called APEX II)

   b. Project names are named after the principle investigators initials followed by the sample number (e.g. lms00l)

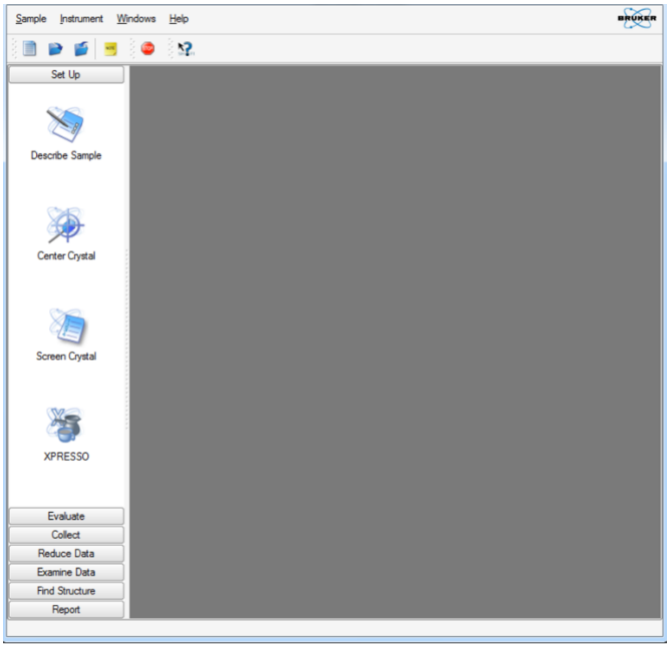
   To start APEX2, choose **Start > Programs > Bruker AXS > APEX2** or double-click APEX2 icon on the **Desktop**. Once APEX-II has launched, select the **Instrument menu** and click on **Connect**.

5. To create a new sample, choose **Sample > New** from the Menu Bar or click the **New icon**.

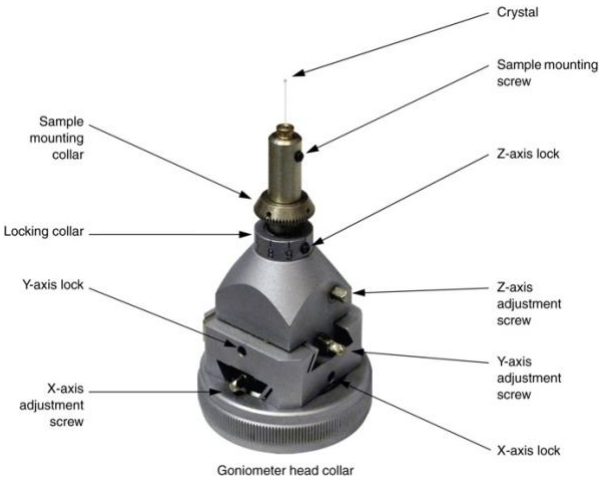
   Give a sample name under the pop-up window. Give it a project name. As a suggestion, make it short (five or six characters) as you will be typing it out later. Using your initials and a reference number is a good idea.

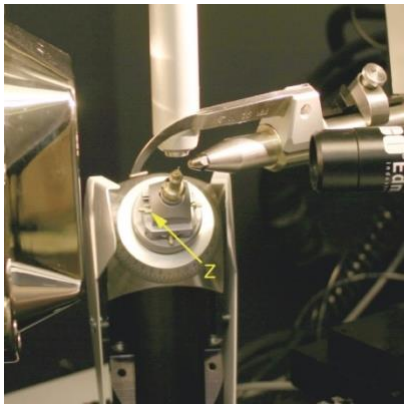


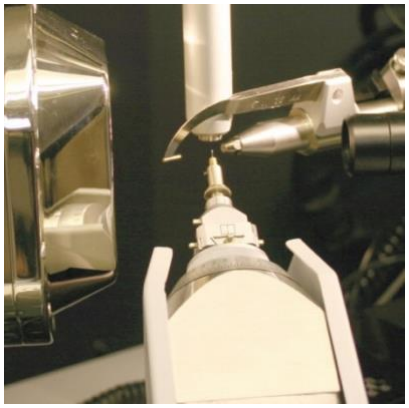
6. Turn on the enclosure lights by pressing the switch marked with a lightbulb on the front left panel.
7. Once the project is set up, click on the Set-up tab (left-hand panel) and click on the Center Crystal option. This will launch the centering routine applet. The video window should also be restored.



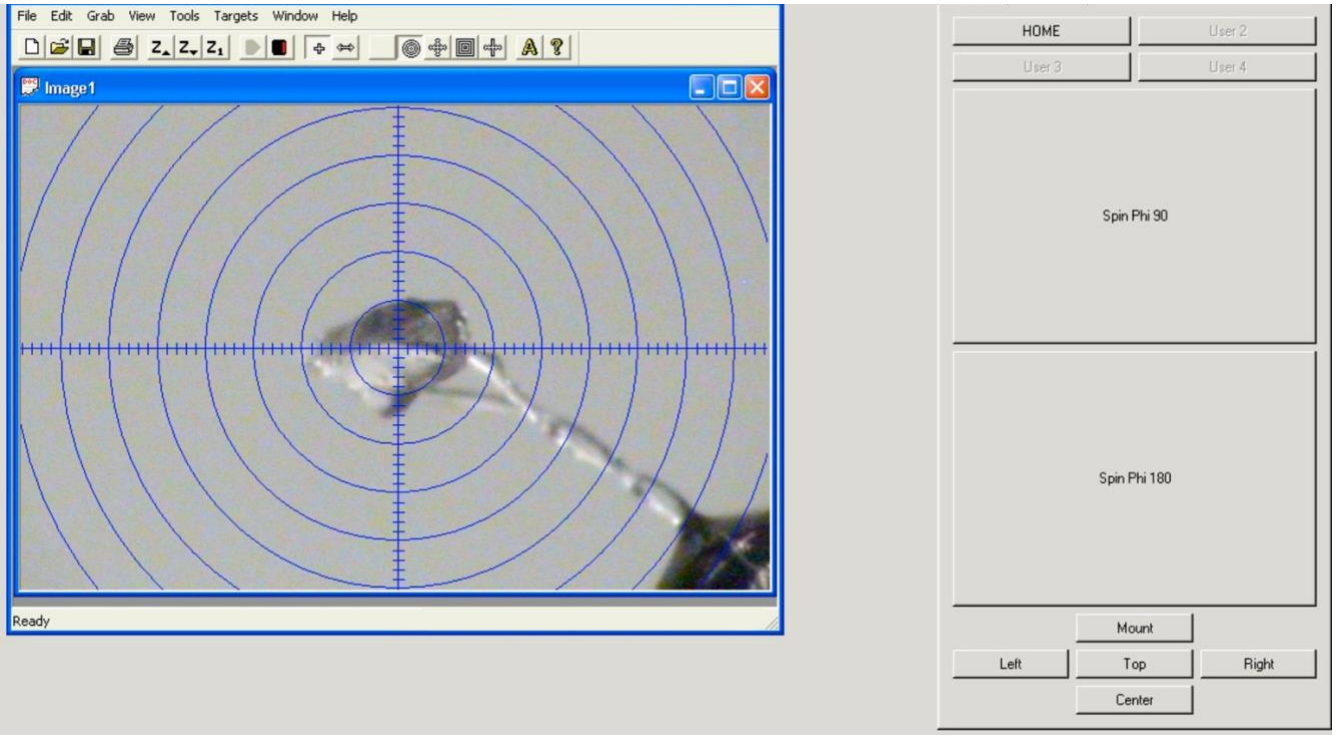
8. Drive the goniometer to a Mount position to mount your crystal on the magnetic head of the goniometer.





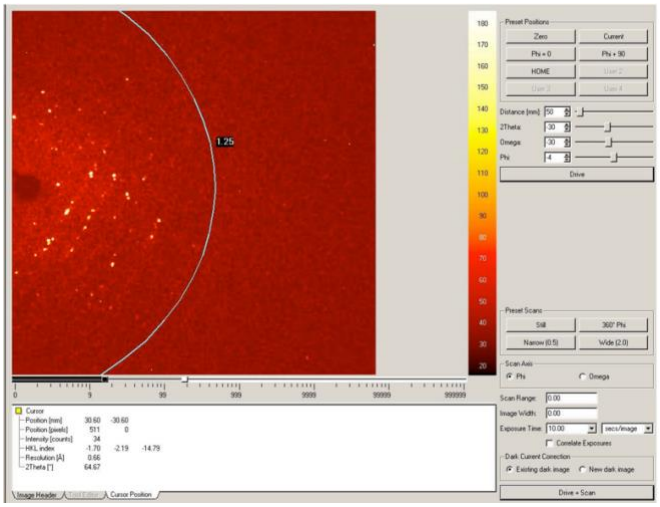


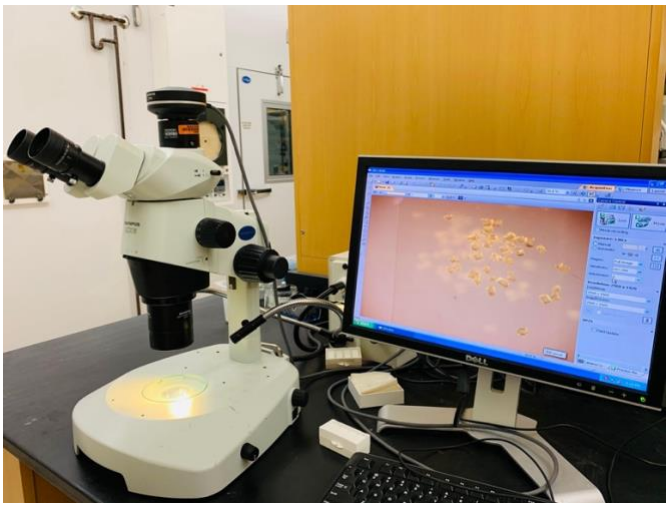
9. Now drive the goniometer to the “Left” and by using a combination of phi-90 and phi-180 rotations (large buttons on the right-hand panel) as well as manipulating the sleds on the goniometer head align the crystal such that it does not shift as you rotate it around.



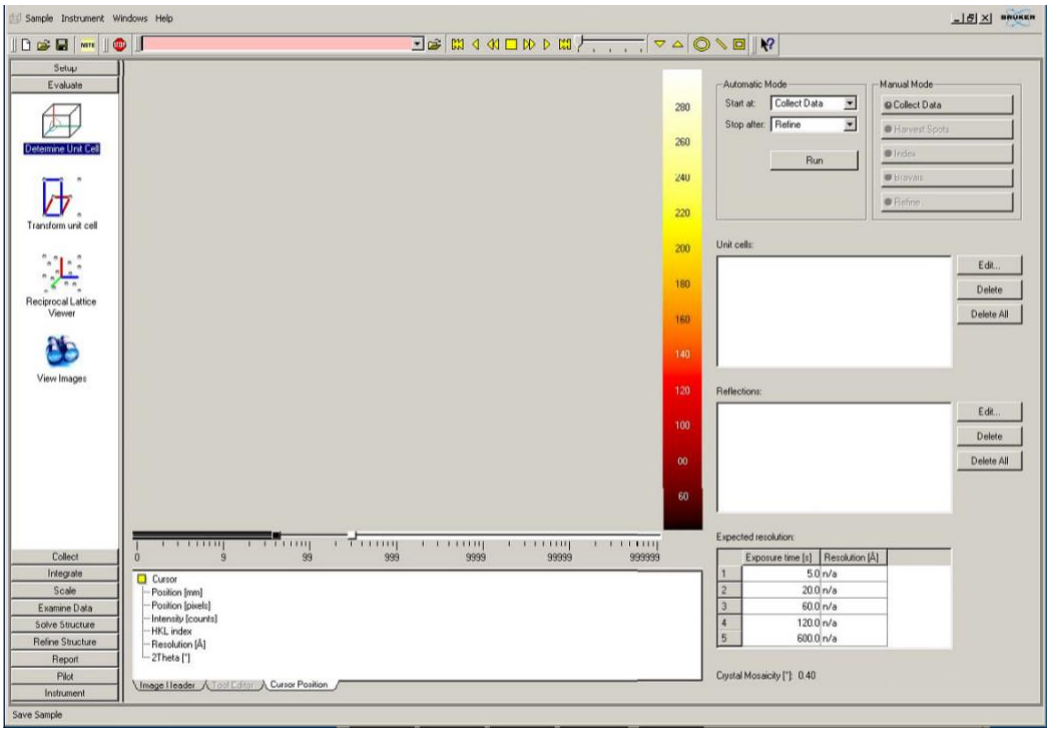
10. Once you are finishing centering the crystal close the doors.

11. Select Simple Scans module. Click Still, and Drive + Scan. After 10 seconds, an image collected is displayed on the screen. If you are not satisfied with the image, find another crystal under microscope and try again. If the crystal diffracts well and gives nice spot distribution, go ahead to collect matrix runs and determine the unit cell.





12. In the task menu bar, Click Evaluate and then Determine Unit Cell. A new module appears.



Two procedures are available, Automatic Mode or Manual Mode. Click Run for automatic mode.

⇒ **Users of the OSU are encouraged to use the manual procedure.**

a) Click Collect Data, this will open the Unit Cell Data Collection Window.

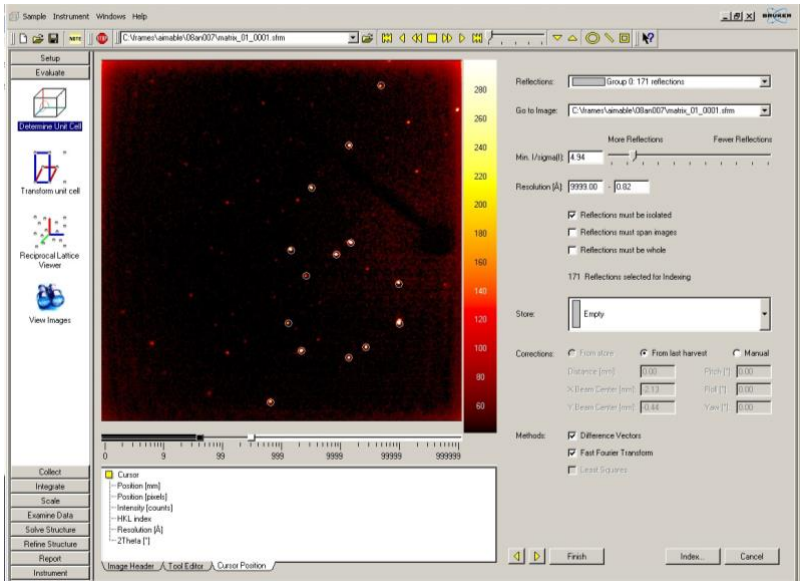
b) Choose an exposure time (default is 10 seconds) and click Collect.

c) Three times 12 frames will be collected which will be displayed in the frame window.

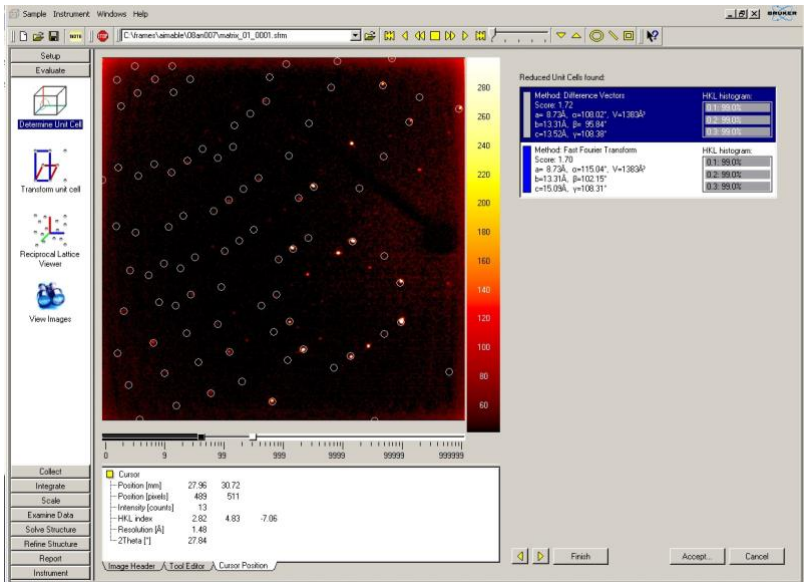
d) When finished proceed to Harvest Spots, select an I/sigma cutoff value for the diffraction spots to be used (see the frame to the left for the spots that will be used, a useful default value is 20.0) and click on Harvest. Reading in of the spots may take several minutes.

e) Proceed to Index, use all default values and click on Index.

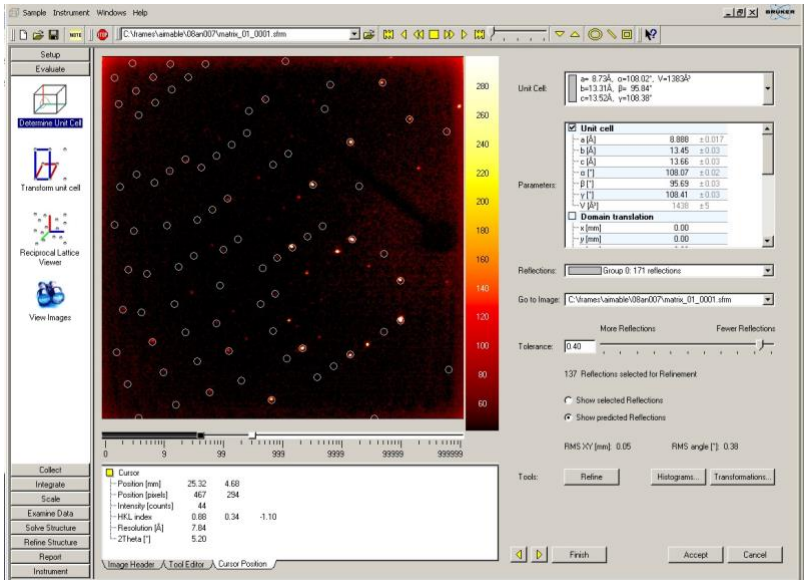
f) If the unit cell looks reasonable proceed to Refine, take all defaults and click on Refine, then Accept.



Unit Cell Indexing View



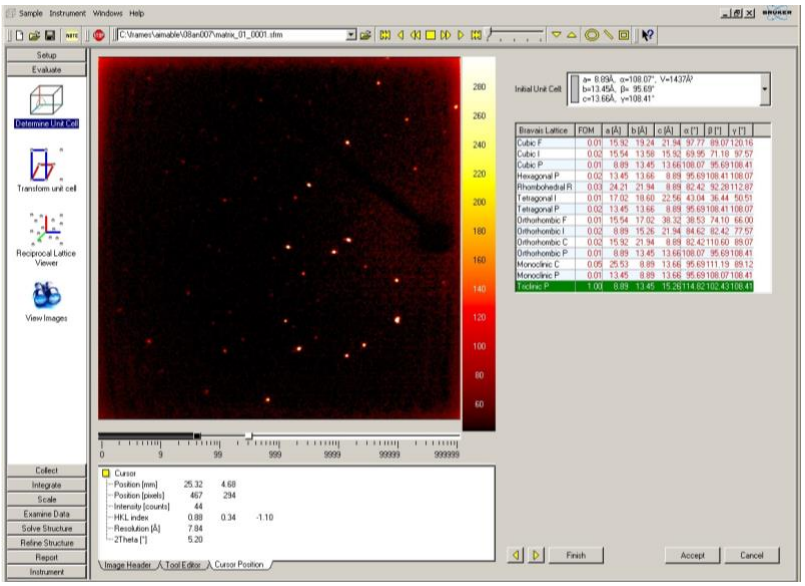
Unit Cell Indexing Result View



Unit Cell Refinement View

g) The next step is to see if there is higher **Bravais symmetry** for your cell. Select the **Bravais button** and see if any alternative unit cells come up. ***The highest symmetry will be highlighted in green***. This may not be the real symmetry; the **Laue symmetry** of the diffraction pattern dictates the true symmetry of the crystal system. Usually the option selected is correct. If you wish to err on the side of caution choose the triclinic setting. Click on **Accept** once you have made your decision.

h) Finally, **Refine** the unit cell parameters. In the Parameters window check the Domain Translation, Detector Orientation and Goniometer Zeroes check boxes. This will allow a better refinement of the crystal orientation matrix. You can introduce more reflections to the matrix by adjusting the Tolerance slider to allow More Reflections. Click on **Refine** to improve your unit cell data.



i) Save your orientation matrix information at this stage by clicking on the Sample menu and Exporting the **P4P file**. The default filenames (based on your project) are acceptable.

Data Collection:

13. I suggest recording a series of images of your sample at this stage. This can be useful for applying a **face-indexed absorption correction**. It is not necessary, but is a good record of the sample on which data were collected.

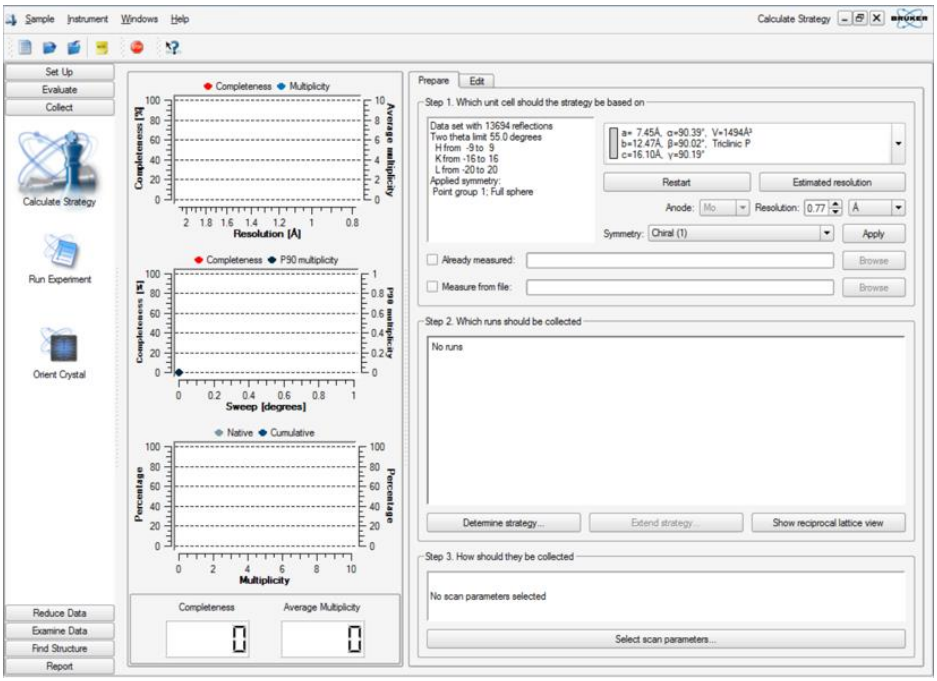
⇒ To perform this operation, select the **Reduce Data Tab** and click on the **Index Crystal Faces** applet. Create a new image file and when the pop-up to save the file appears choose the work directory and save the image into this directory.

⇒ The software will drive the goniometer and you will see the crystal rotate on the video window, which will have been restored during this operation. Once the crystal has stopped rotation minimize the video window and using a combination of the mouse-scroll wheel and good judgment select the faces of the crystal.

⇒ Once you have mapped out the surface of the crystal you will find in the bottom right-hand corner the size of your crystal (note this down).

⇒ You should also save the referenced faces to a file. To do so, select the **Sample** menu and Export the **P4P file**, the same as you did above at the end of the unit cell determination.

14. Under Collect in task bar, click **Data Collection Strategy**. Adjust necessary parameters in the new window. In general, 0.8 to 0.75 Å resolution is suitable for a "good" crystal structure. Click **Determine Strategy** to build a strategy.



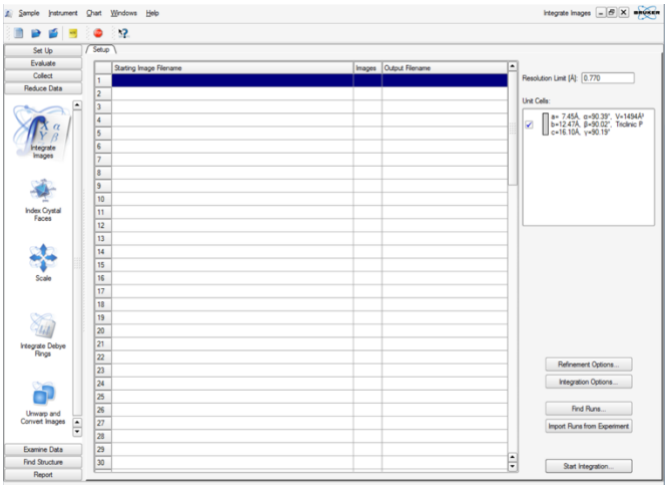
⇒ This step allows you to modify the runs, in particular the frame time and frame width; click on Select **scan parameters** to change them. You should check these values. A typical small molecule data collection uses frame widths of 0.3 or 0.5 deg (the software attempts to do some calculations to determine a suitable frame width … do not trust the computer, you will not get suitable coverage if the frame-width is 1.9 deg (or some other relatively large value). You can enter the frame time in the box below (this is the counting time per frame recorded); a suitably diffracting crystal will typically record data with a 10 s frame, weaker diffracting crystals will require longer times. Accept the frame width and time and the program will update the runs appropriately.

15. Click **Run Experiment** under Collect in task bar. Click **Append Strategy** and the run list appear. You now have a complete data set to work with.

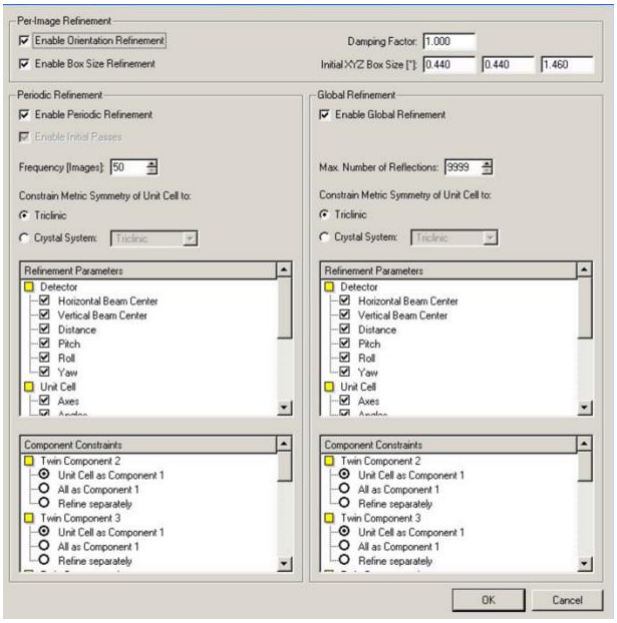
16. Check **Generate New Dark Images**, then proceed to **Execute**. The instrument will now collect your dataset. Open **Check Status** from the Instrument drop down menu to check variables and completion time. This will open the **Instrument Status** window.

17. Fill out the user's log!

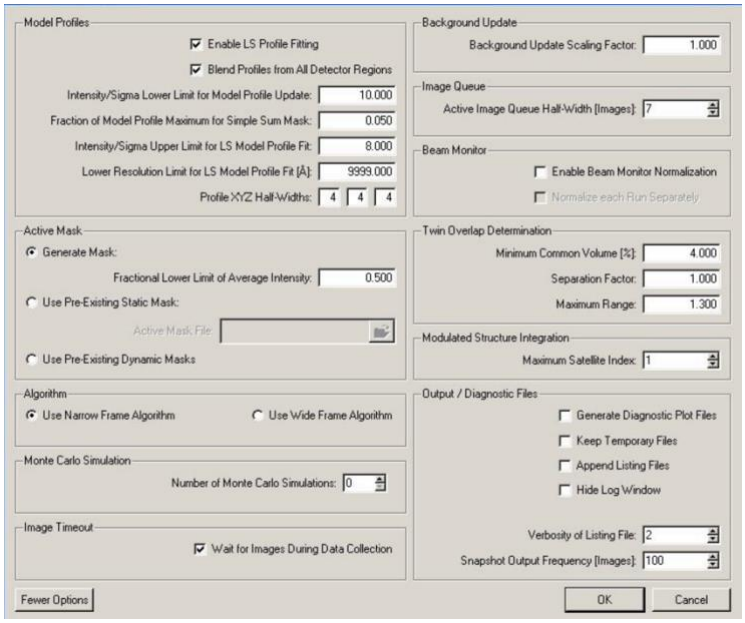
18. Under **Integrate** open **Integrate Images**. Change the resolution limit to 0.75 or lower. Click on **Find Runs**, check the runs you would like to integrate (leave out the Matrix runs).



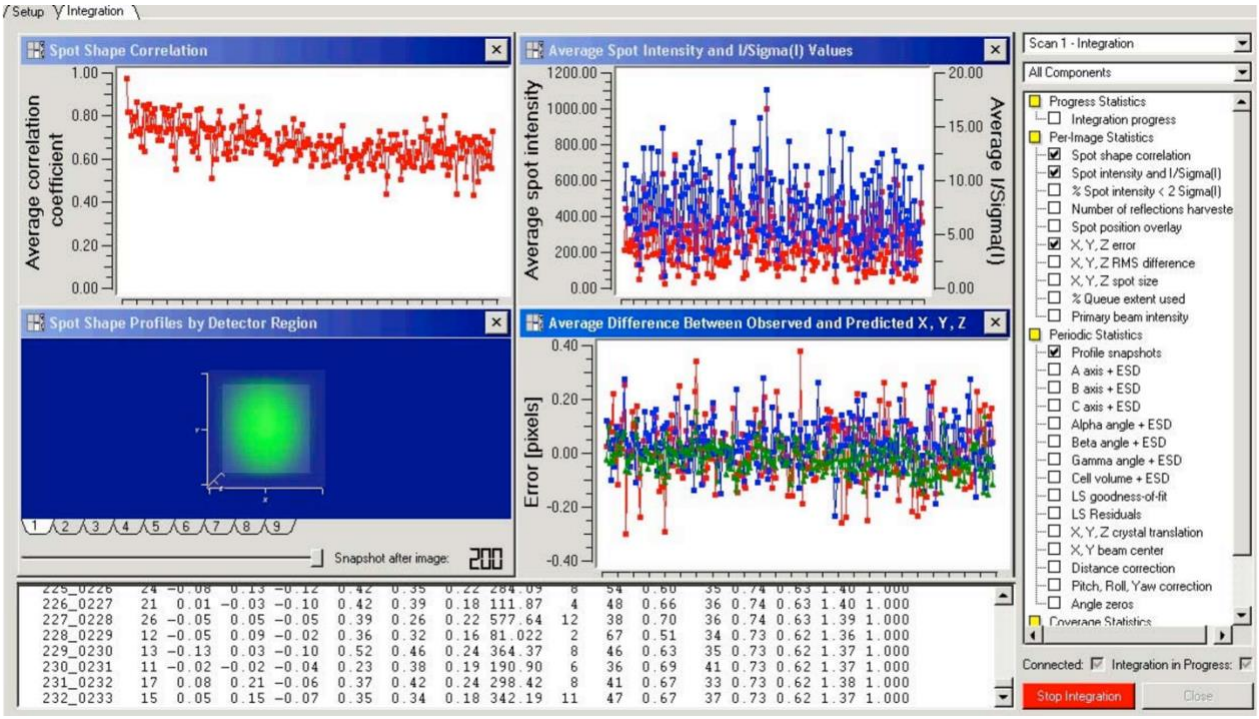
19. Click **Refinement Options** button. In the pop-up window, **enable the options** shown below. Click OK.



20. Click **Integrate Options** button. In the pop-up window, click **More Options** to extend the window. Enable the **options** and **use 0.500 for the Generate Mask option**. Click OK.



21. Click on **Start Integration**. This will start the integration. Wait for it to finish, this might take several minutes.



22. During the integration, the **XYZ box size** is re-calculated and updated. Write down **these numbers**. When the integration finishes, select **Sample** > **import** > **P4P/SPIN** file, load the ***m.p4p** file and import all the information from this file. Change the **Initial XYZ Box Size** with the updated ones under **Reflection Options**, re-run the integration. Doing so usually gives better results.

23. Select Scale module and click scale icon. In most cases the default settings are good enough.

⇒ If the space group of the structure is **noncentrosymmetric**, you need to select the corresponding **non-centrosymmetric** point group.

⇒ Click Next to open the Parameter Refinement window. Select the proper **Absorption Type** for your crystal. Click Refine. A graph appeared shows the intensity statistics.

⇒ Click Next to continue. In the Error Model window click **Determine Error Model**. If you are happy with the results, click Finish.

⇒ In the next window click on the tabs at the bottom of the screen to view diagnostic data. If the results are good, click Exit Scale to exit the Scale.

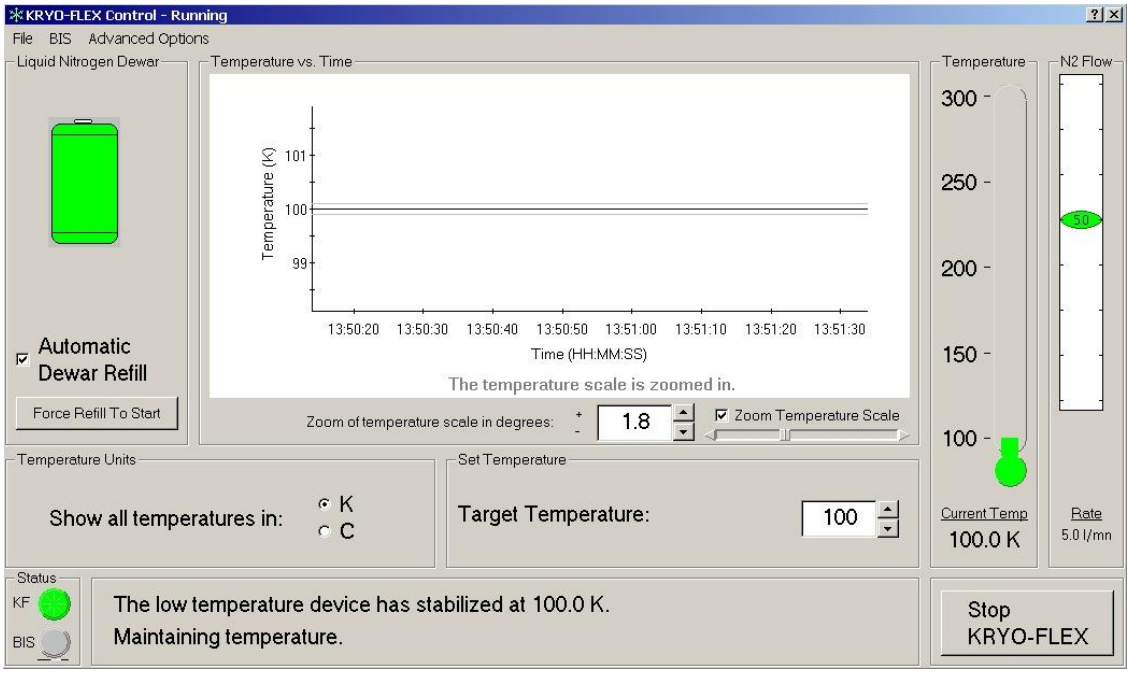
24. Use the obtained *m.p4p and *m.hkl files as input files for ***space group*** determination (**XPREP**), ***structure solution*** (**SHELTL xs**), and ***structure refinement*** (**SHELTL xl**).

At this point you are now ready to solve and refine the structure; a different set of skills and instructions are required for this. Please let the next user or Dr. Latifi know that you are finished.

More information on data collection - see APEX2 User Manual.

25. To shut down the Low Temperature Device:

In order to save LN_2, hit the "**Stop Kyro-Flex**" button to stop N_2 flow. You should see the temperature begin to rise.



If you are unsure of any aspect of operating the X-ray Instrument – See Dr. Reza Latifi.

Facility Director:
Dr. Reza Latifi
Tel: 405-744-4330
reza.latifi@okstate.edu
https://chemistry.okstate.edu/bios/367-faculty-member-bio-latifi

Sample Ship-to Address:
Dr. Reza Latifi
Department of Chemistry
107 Physical Science Building
Oklahoma State University
Stillwater, OK, 74078

X-ray Lab Location:
HBRC Building, Rm. 150L (150 LB)
Tel: 405-744-4330

RADIATION SAFETY TRAINING REQUEST

RETURN TO: **RADIATION SAFETY**
radsafe@okstate.edu OR
223 Scott Hall

OFFICE USE ONLY:

Entered By: _____ Training Date: _____

M/LST Training Date: _____ Email Sent: _____

FULL NAME *(first, middle, last)*:_____

DATE:_____ ☐ MALE ☐ FEMALE

CAMPUS WIDE ID (CWID):_____DATE OF BIRTH: _____

LAB OR OFFICE PHONE: _____HOME PHONE: _____

CAMPUS E-MAIL ADDRESS: _____

DEPARTMENT: _____

PLEASE LIST THE ISOTOPE(S) AND/OR X-RAY MACHINES YOU WILL BE USING:

_____

Please check which of the following training module(s) you will need to take (ask your PI if you are unsure):

- ☐ Radioactive Materials – Sealed Sources Training
- ☐ Radioactive Materials – Unsealed Sources Training
- ☐ X-ray Machine Training

The person in charge of the RAM lab and/or X-ray instrument you will use is the permit holder. This form will not be processed without the permit holder's signature or email authorization.

AUTHORIZED USER/PERMIT HOLDER (PRINT):_____

AUTHORIZED USER/PERMIT HOLDER (SIGN): _____

If submitting electronically, this form must either be signed by the RAM/X-ray Permit Holder or come from his/her e-mail account. If submitting a printed copy via campus mail, the Permit Holder's signature is required.

THERE IS A SEPARATE FORM REQUIRED FOR ISSUING DOSIMETERS. IF YOUR WORK REQUIRES YOU TO WEAR DOSIMETERS PLEASE FILL OUT THE **BADGE REQUEST FORM** (SEE THE FORMS PAGE ON THE RADIATION SAFETY OFFICE WEBSITE) AND SUBMIT IT TO OUR OFFICE ONCE YOU'VE COMPLETED YOUR TRAINING.

ALLOW 2-3 DAYS FOR PROCESSING AFTER RECEIPT OF THIS FORM. ONCE PROCESSED, AN EMAIL WILL BE SENT TO YOU CONTAINING YOUR TRAINING REQUIREMENTS AND DUE DATES, AS WELL AS INSTRUCTIONS FOR LOGGING INTO THE SYSTEM.

PLEASE DIRECT ANY QUESTIONS TO THE RADIATION SAFETY OFFICE STAFF AT 744-7890 OR 744-3474

(Revision 08/2018)

Revised August, 2018

DOSIMETRY MONITORING REQUEST

FULL NAME:_____ ☐ MALE ☐ FEMALE
(First Middle Last)

OSU Campus Wide ID (CWID): _____ DATE OF BIRTH:_____

CHECK ALL THAT YOU WILL BE WORKING WITH, AND ENTER ALL REQUESTED INFORMATION:

☐ RAM – SEALED SOURCES

- Isotopes: _____
- Name of RAM Permit Holder/Principal Investigator: _____

☐ RAM – UNSEALED SOURCES

- Isotopes: _____
- Name of RAM Permit Holder/Principal Investigator: _____

☐ X-RAY

- X-ray Instrument(s): _____
- Name of X-ray Permit Holder/Principal Investigator: _____

Fees and Other Information:

All fees are determined by contract with Landauer and are subject to change.

Required Radiation Safety online training must be completed before dosimeter(s) will be issued.

Faculty Supervisors (PIs) are responsible for returning all dosimeters to the RSO at the end of each quarter by the due date.

The PI who signs this document will be responsible for all monthly fees in addition to any late or unreturned badge fees charged by Landauer for the applicant's badge. See the RSO Dosimeter Billing Policy for more information.

The applicant is responsible for submitting a schedule of RAM/X-ray work for any quarter for which he/she does not turn in one or both badges so that a dose estimate can be determined by the RSO and added to his/her occupation exposure record.

_____ _____
Faculty Supervisor Printed Name (this person will be billed for badge) Date

FACULTY SUPERVISOR SIGNATURE _____
If submitting electronically, this form does not require a signature but must be submitted from the Faculty Supervisor's e-mail account. If submitting a printed copy via campus mail, the Faculty Supervisor's signature is required.

Please allow 5 business days after receipt/completion of required training for processing of badge request.

RETURN COMPLETED FORM TO THE RADIATION SAFETY OFFICE
223 SCOTT HALL
OR
radsafe@okstate.edu