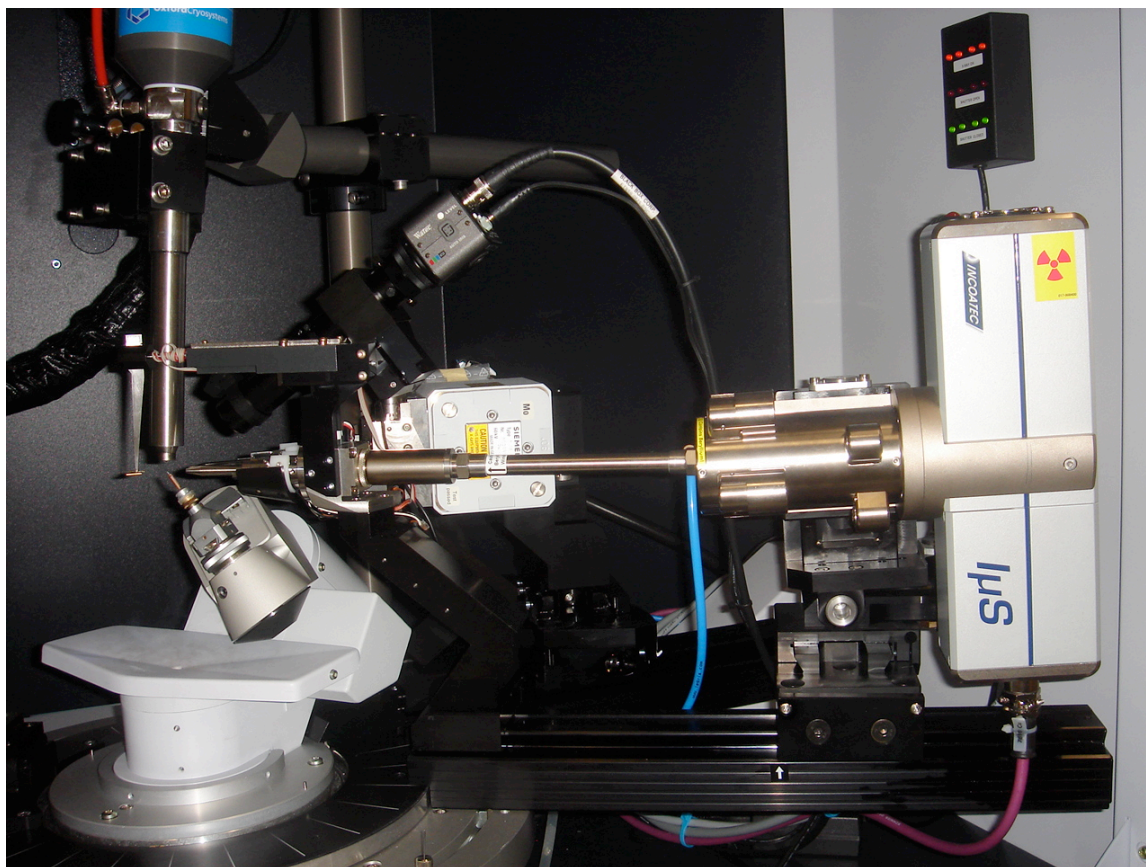# User Guide

Bruker KAPPA APEX DUO Diffractometer
With IμS Cu source

Dr. Bo Li
Director, X-ray Crystallographic Facility
Department of Chemistry
Boston College

This document is a simple guide for data collection using the Bruker KAPPA APEX DUO located at X-ray crystallographic facility, Department of Chemistry in Boston College. At this point, you should already have crystals of decent quality ready and it is best to follow this procedure.

Diffractometer Status Check:

1) On the front posts (Figure 1), check orange display marked 'Ready' and green display marked 'On' is lit. If not, see facility director for help.

Figure 1

2) On the front posts (Figure 1), check yellow 'Busy' light is off; on right panel inside enclosure (cover figure), check green 'shutter close' light is on. Otherwise, you need to wait as the instrument is in use.
3) Check Lytron cooling system for detector. It's on the right side of the diffractometer and it should display -5°C. Check to make sure solution level is between the high and low level marks.
4) Check Haskris chiller used for X-Ray tube cooling. Water temperature is set to 61°F, and the full level lamp is on. The unit is located in room 210. Skip this step is you do not have access.

Oxford Cryostream 700 Low Temperature System

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.
5) On the Cryostream controller (Figure 2), the upper LCD shows the current coldhead temperature, the lower LCD gives the status. The temperature is usually kept at 173 or 100 K but you can change it to your desired temperature. Temperature deviation should be 0.1-0.2 K. If not, please inform the facility director.
6) The LCD on the autorefill controller (Figure 2) shows the amount of liq N_2 (in L) in the storage dewar (60 L volume). Make sure the 'auto' button is turned on for

refill option. Check the weight and display of liquid N_2 tank (220 L volume) to make sure there is enough N_2, replace it with a new tank if needed.

Figure 2

7) To adjust the temperature of Oxford Cryostream system, open the Cryopad program (Figure 3) on the desktop of computer right next to the diffractometer. The current setting and status are showed on the 'Display' menu. Click the 'Command' menu (Figure 4), use the 'Ramp' or 'Cool' option under Phase to set desired temperature. The program will automatically hold the temperature.
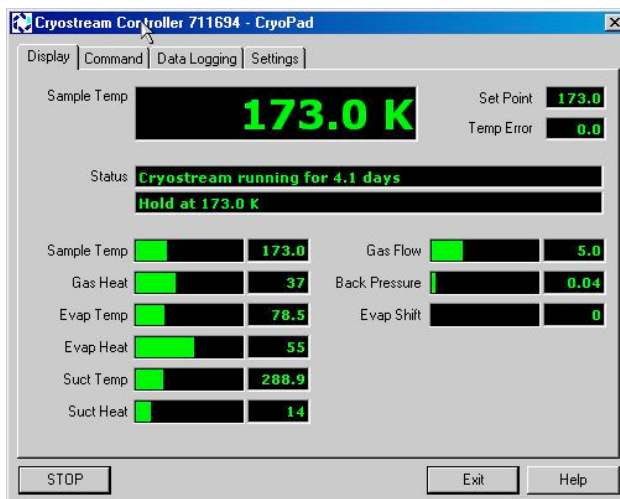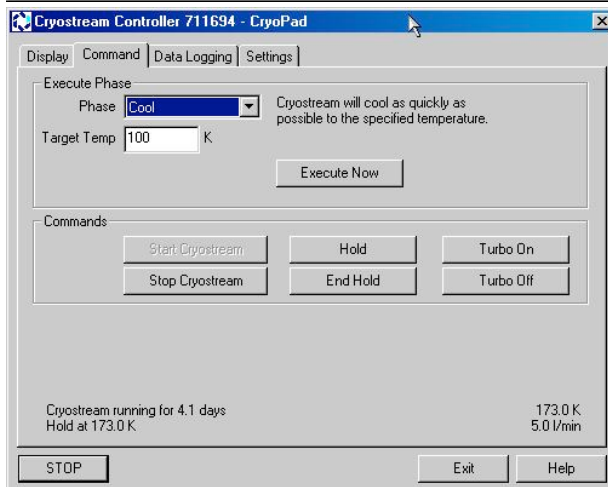

Figure 3

Figure 4

8) At the back of diffractometer check the Oxford dry air unit (Figure 5). The air pressure reading should be 10-15 to prevent ice and condensation on crystal.

Figure 5

Activate BIS Program:

9) Click icon BIS on the desktop and BIS should start without any errors (Figure 6) and Video window will come up too. If not, see facility director for help.
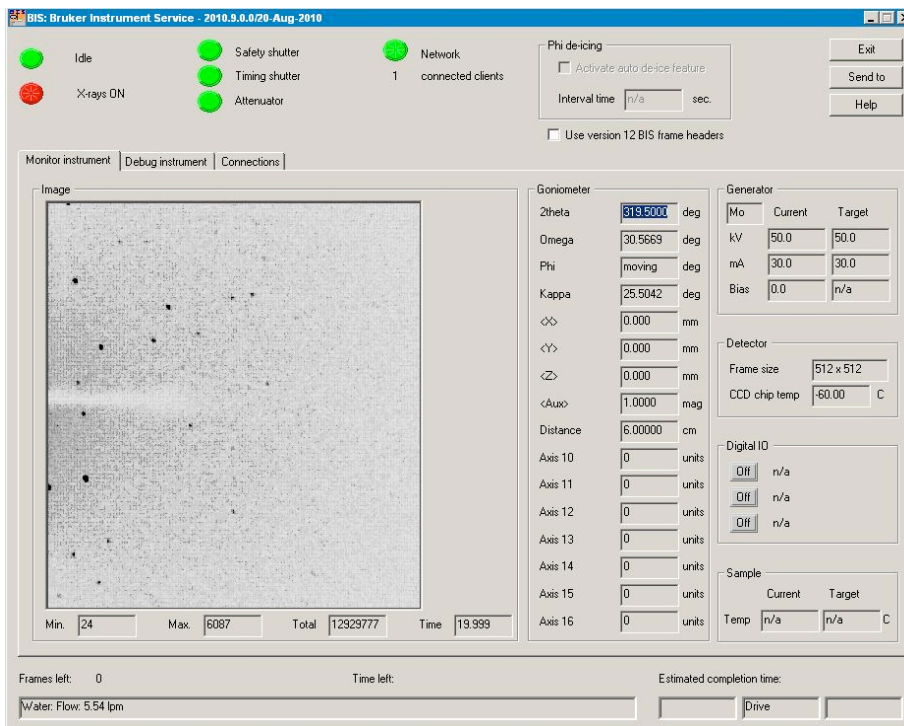

Figure 6

Start APEX2 Program:

10) Click icon APEX2 on the desktop and select **Sample > New**, a window will pop up (Figure 7).
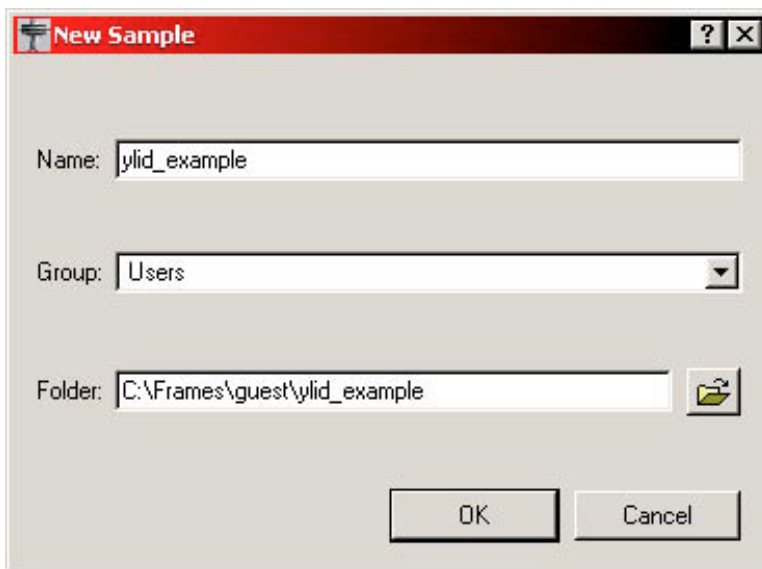

Figure 7

In window for new project (Figure 8), you need to fill some information:

Name – you need to give a name of your sample. Usually we use your initials followed by two or three digit number

Folder – each research group has its own subdirectory under C:\Frame\Guest\. The name of the directory is initials of the faculty. Under this directory, use the folder with the crystal name.

11) Click OK to create new folders. The Task Bar appears with the Setup section, then click **Center Crystal** (Figure 8).
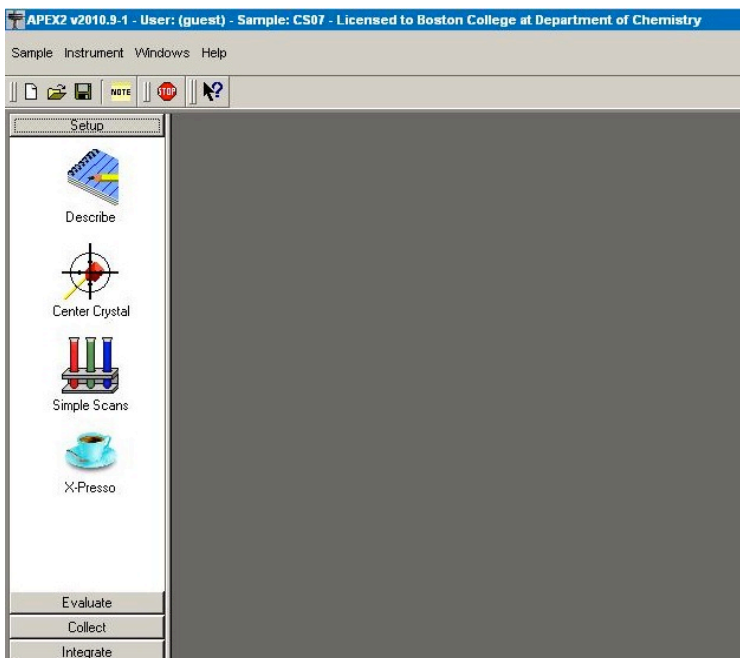

Figure 8

12) On the window coming up (Figure 9), click **Center** button at the bottom. Goniometer will drive into a position easy for mounting crystals.
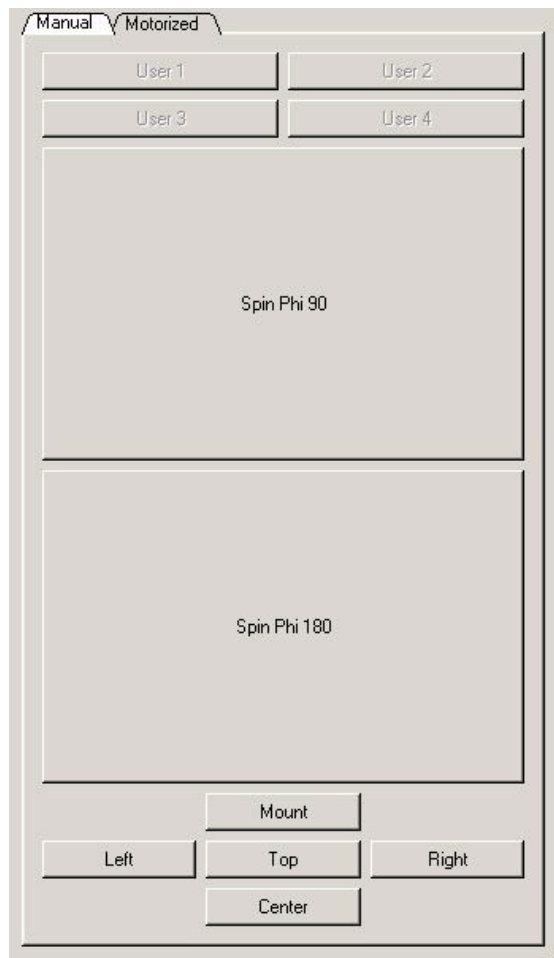
Manual	Motorized	
User 1		User 2
User 3		User 4

Spin Phi 90

Spin Phi 180

	Mount	
Left	Top	Right
	Center	

Figure 9

Crystal Mounting and Centering:

13) On a clean microscope slide, spread evenly small amount of paratone oil. Transfer some crystals from your container into the oil. Select a good-looking crystal (shining, transparent, regular shape, no crack, no satellite) of reasonable size (no larger than $0.4 \times 0.4 \times 0.4$ mm^3, the diffractometer is equipped with a 0.6 mm collimator) under microscope and attach it to a cryoloop with minimum amount of paratone oil.

14) Push the 'Open Door' button on either side of front posts (Figure 1), then slide to open the doors of enclosure and mount the pin onto the magnetic base of goniometer head.

15) Use the Video window (Figure 10) to help centering crystals.

Be careful not to touch the collision protection plates, collimators, and beamstop while adjusting goniometer screws.

Figure 10

16) Use the goniometer wrench to quickly adjust Y and Z screws (Figure 11) on the goniometer head so the crystal is roughly sit in the center of beam. Click **Spin Phi 180** (Figure 9) to make sure crystal remains same position after rotation. If not, adjust screws for half of the difference. Repeat the previous steps until you are satisfied.

17) Click **Spin Phi 90** (Figure 9) to adjust X-axis screw (Figure 11). Click **Spin Phi 180** (Figure 9) to make sure crystal remains same position after rotation. If not, adjust screws for half of the difference. Repeat the previous steps until you are satisfied.

18) Click **Left** (Figure 9) and then **Right** to check the height. If not the same adjust Z-axis screw for half of the difference (Figure 11). Click **Spin Phi 180** (Figure 9) and repeat the previous steps until you are satisfied.

19) Once crystal stays at same position for all rotation, click **Center** to drive goniometer back. Close the sliding lead glass doors; make sure you hear the clinking sound and red blinking alarm light on front posts disappear.
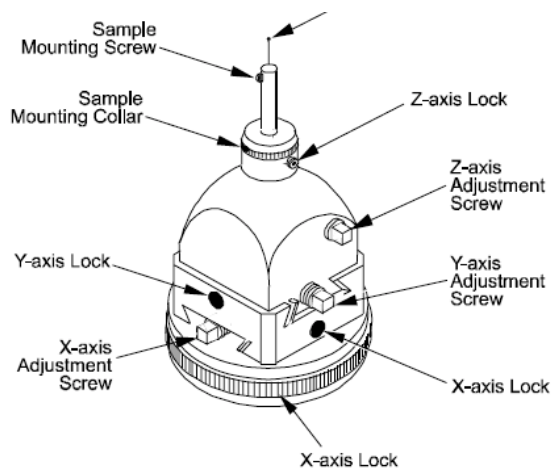
Figure 11

Unit Cell Determination:

20) To screen crystals, click **Simple Scans** in APEX2 window (Figure 8) and the following menu (Figure 12) comes up.
Set distance to 60 mm, then click **Zero**, **Still**, **Drive+Scan** to obtain a still image; or click **Zero**, **360 Phi**, **Drive+Scan** to obtain a rotation image. If the crystal is of poor quality, remove it and mount a new one. Repeat steps in Crystal Mounting and Centering session, and then obtain still images or rotation images.
Once you are satisfied with the quality of selected crystal, move to next step for unit cell determination.
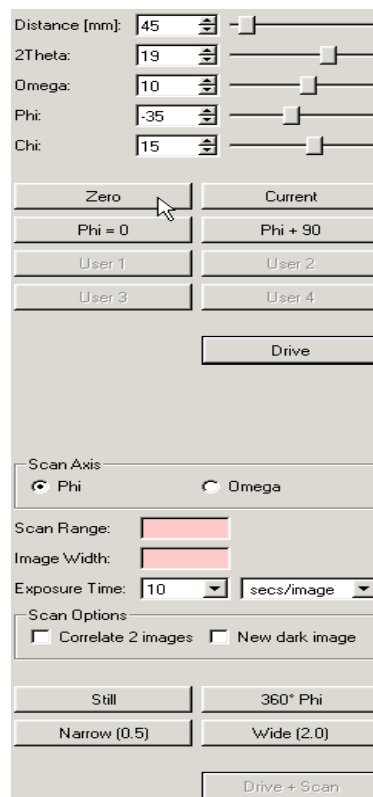
Figure 12

21) In the task menu bar, Click **Evaluate** and then **Determine Unit Cell**. A new module appears (Figure 13). Click **Run** for automatic mode.
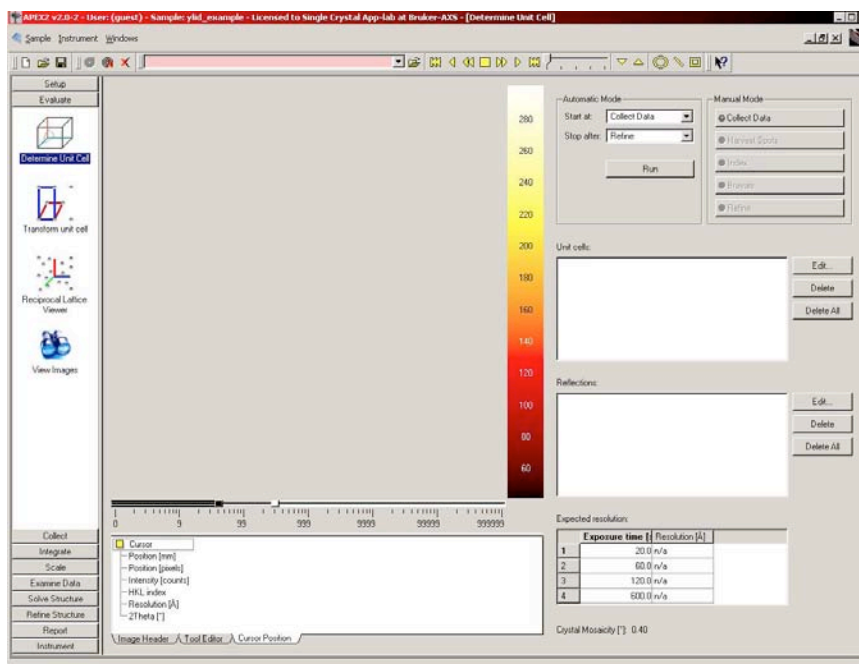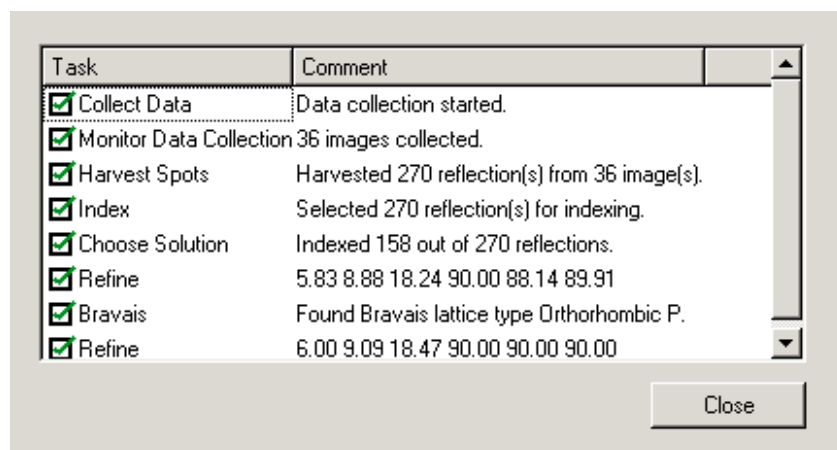
Figure 13

The progress list window (Figure 14) will show a check mark and comments for each completed step. Ideally, all the harvested reflections should be selected and indexed.

Figure 14

Click **Close** button to close the list and go back to main unit cell window and you can perform additional operation using steps in Manual Mode.

22) If there are problems with the unit cell determination, you may want to try to look for a better crystal. Drive goniometer to **center** position and repeat from crystal mounting and centering session.

CSD search:

23) It's important to check if this crystal is already known. Cambridge Structural Database can make a search based on predefined lattice parameters by comparing them with already known. See CSD help page to learn how to use the database.

Data collection:

24) Before starting data collection, fill in information in the **Describe** window (Figure 15) in Setup under task bar menu.
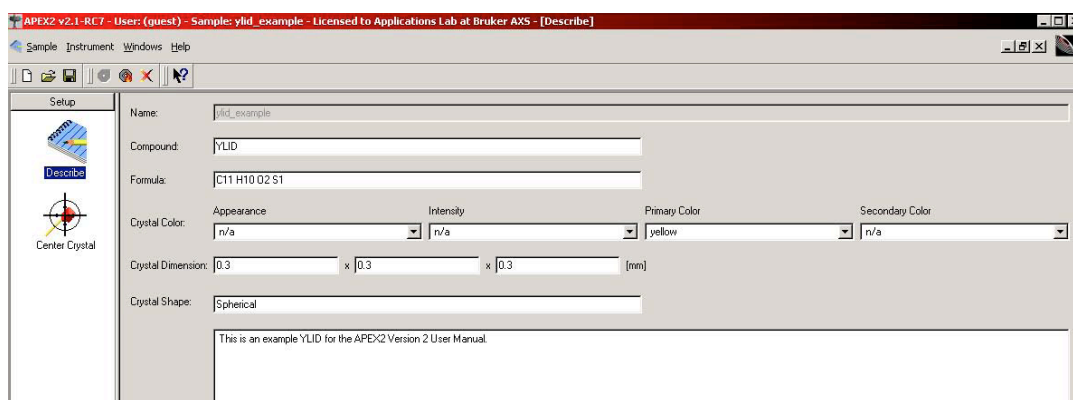

Figure 15

25) Under **Collect** in task bar, click **Data Collection Strategy**. Adjust necessary parameters in the new window (figure 16). Click **Refine** button to refine strategy. Stop the refinement when it is 30 to 50% finished and click **Sort**.

Typical parameters setting is:

Distance – 60 for both Mo and Cu;

d – 0.75 for Mo and 0.81 for Cu;

Image width – 0.50 for both Mo and Cu;

Anode – Mo or Cu

Exposure time – 5/10/20/30, depending on the diffraction intensities;

Strategy – Best in 12 hours or Best in 24 hours.
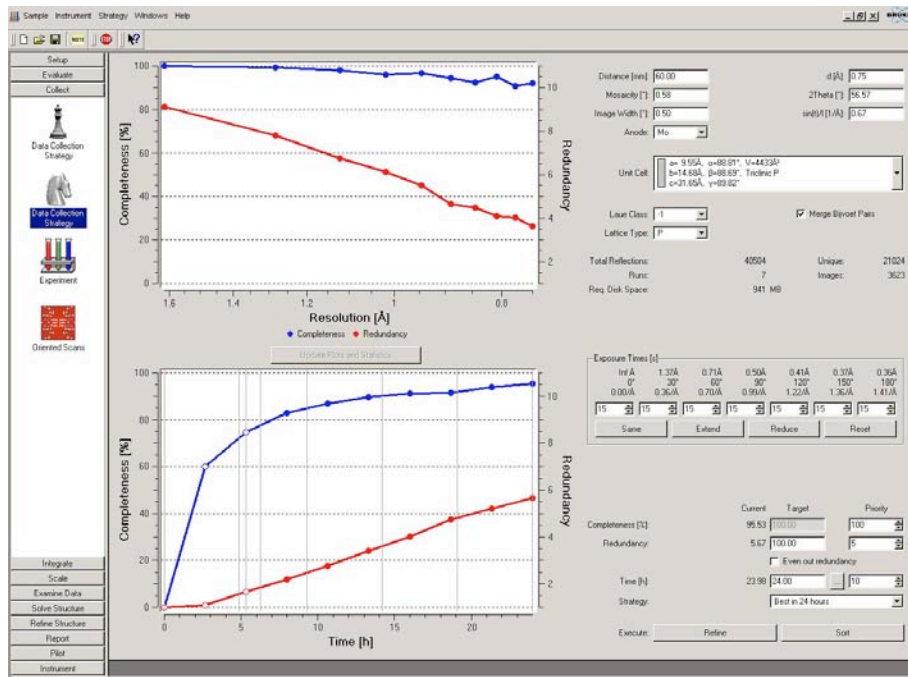
Figure 16

26) Click **Experiment** (Figure 17) under **Collect** in task bar. Click **Append Strategy** and the run list appear. You now have a complete data set to work with.

Figure 17

Click **Validate** to make sure all runs are valid, and then click **Execute**. Date collection will start and you can monitor the progress in this window (Figure 18).
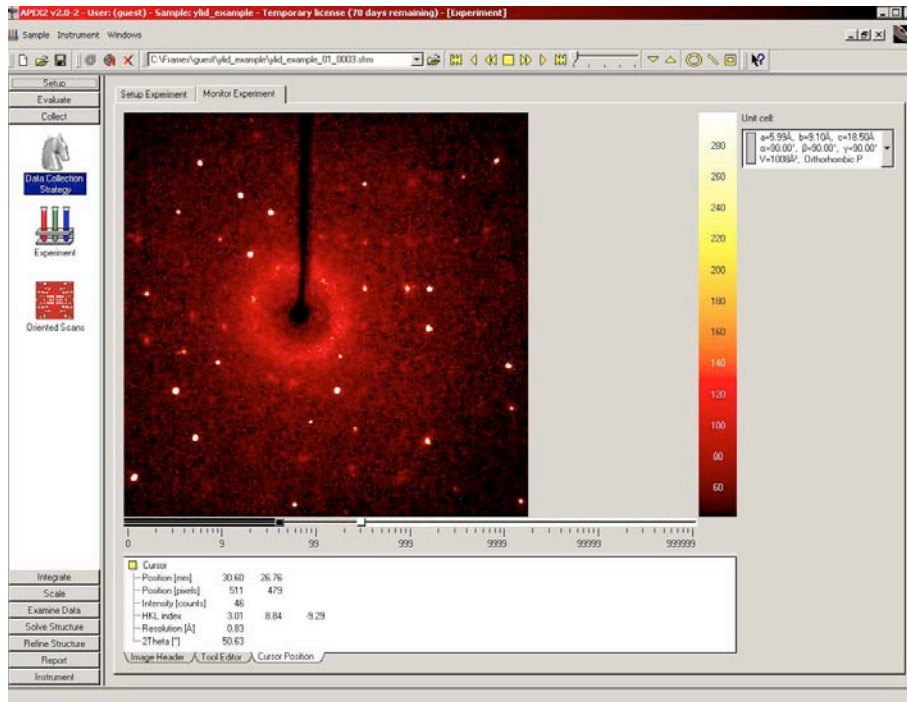

Figure 18

Switching Radiation Source:

If you need to switch between Mo and Cu sources, check what is the current radiation source first. You can do so in BIS window (Figure 6), or instrument status (Figure 19) in APEX2 (**Instrument**>**Show Status**). Wavelength is 0.71073 for Mo and 1.54184 for Cu.
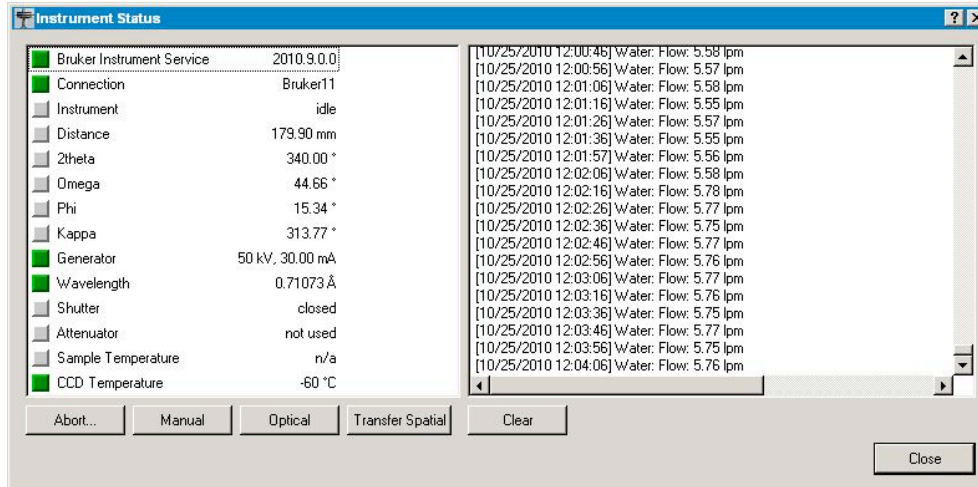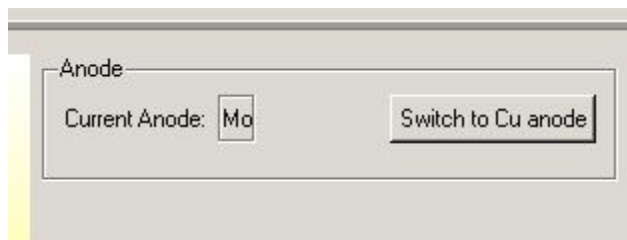

Figure 19

Another way to check is to see physically green light on which source is on.
To change wavelengths, go to **Simple Scans** in setup section under task bar. Click the **Switch to Cu (or Mo) anode** button and wait a minute or so for the switch to complete. Confirm the right source is present using the method mentioned above.

Additional Information:

For more information about operation on KAPPA APEX DUO, read Apex Duo manual.

For information about data processing, structure solutions, and refinement, register to attend the small molecule crystallography course offered annually. Read Apex2 user manual and Shelxtl manual.

For information about Oxford Cryostream 700 LT system, read the operation manual.

All the manuals are located in x-ray facility, room 209c and might be available the facility website.