Alignment and Calibration of the APEX Instrument

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Chemical Crystallography Laboratory

Author: ____________________________

Douglas R. Powell

Distribution

Douglas R. Powell, Laboratory Manager
Prof. George B. Richter-Addo, Chair, Faculty User's Committee
Laboratory copy maintained by Douglas R. Powell

Revision Record

Date	Version	Responsible Person	Description of Change
1/4/2013	2.001	Douglas R. Powell	Initial Release
1/30/2013	2.002	Douglas R. Powell	Minor revisions
3/30/2017	2.003	Douglas R. Powell	Software change

The following laboratory users have read this manual.

Name	Signature	Date

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A. Scope and Availability

This manual outlines the steps used to align and calibrate the Bruker APEX instrument in the Department's crystallography facility. This procedure is to be carried out semi-annually, and whenever any major change to the instrument (such as a tube replacement) occurs. A copy of this manual will be publicly available on the Laboratory's web site.

B. Summary of Method

Aligning the instrument directs the maximum peak intensity through the center of the three circles of the goniometer and onto the sample. The instrument calibration documents the general condition of the instrument and especially the X-ray tube.

C. Responsibility

The laboratory manager or a trained designate is responsible for performing these tasks.

D. Safety and Training

This instrument produces ionizing radiation that is potentially harmful to anyone near the instrument. Anyone that uses or maintains this instrument must be trained in the properties and safe use of X-ray radiation.

To produce the radiation, parts of the instrument operate at high voltages (20-50 kV). The internal components of the instrument should only be serviced by people trained to handle high voltages.

If the X-ray tube is removed, care must be taken with the windows of the tube. These windows are made of beryllium foil which is easily damaged. Beryllium metal is toxic if ingested.

All laboratory users must be trained annually in radiation safety as specified by the Radiation Safety Office (http://www.ouhsc.edu/rso/) at the University of Oklahoma. Further training in the safe use of this instrument will be provided by the lab manager.

E. Equipment and Supplies

- Bruker D8 goniometer with APEX detector.
- Ylid alignment crystal mounted on a goniometer head. Note that the ylid sample must only be analyzed at room temperature. This material undergoes a 2nd order phase transition which destroys the sample when it is warmed up from low temperature to room temperature (so do not cool below room temperature).
- Distilled (not deionized) water, if necessary.
- Ethylene glycol, if necessary.
- Glacial Acetic Acid, if necessary.
- Sodium Carbonate, anhydrous, if necessary.

F. Procedure

The specific steps in the instrument alignment and calibration will essentially follow the procedure listed in the "SMART APEX Installation Manual," M86-E00057. X-Ray tube changes that may need to be made are described in the Bruker-AXS "Changing X-ray Configurations" manual, M86-E00099.

Preliminary Steps

1. If the frame buffer computer is off, the X-ray generator is off, or power to the detector is off, consult APEX Instrument Operating Instructions to correct these issues.

2. Inspect the instrument for unsafe/unusual conditions.

3. Turn off the low temperature device and stop the flow of dry, room-temperature gas.

4. Check water level and water quality in the Haskris heat exchanger. If the level is low, then add tap water to fill the Haskris. If the water is turbid then drain and clean the water filter in the heat exchanger. Replace the water and power on the heat exchanger to circulate the water. Repeat water exchanges until the water is clear. With the last water fill add about 20g Na_2CO_3, anhydrous. Be sure to prime the water pump each time the water is replaced before starting the heat exchanger.

5. Check the water flow rate at the generator. If the flow rate is < 4 liter / min then clean the system using adding about 50 ml of glacial acetic acid to the water chiller while running. After about 30 minutes, drain and flush the water as described in the step above until the smell of acetic acid is no longer present. If the flow meter is still < 4 liter / min then check the flow meter for obstructions and check the screen in the X-ray tube. If the tube is removed from the tube housing, be sure to wipe any water

and fingerprints from the ceramic part of the tube and the plastic liner around the tube.

6. Check the fluid level and temperature of the Neslab cooler. If the level is too high, then transfer excess fluid to the hazardous waste container, add 10-20 ml of ethylene glycol and clean up the area.

7. Test the door part of the safety circuit by opening the shutter and then opening the enclosure doors. The shutter should close and the red light on the side panel should blink. If either of these events fails to happen, then replace the faulty safety circuit component(s) and retest.

8. Raise the nozzle of the low temperature device out of the way.

Optimize X-Ray Beam

Modern X-ray tube manufacturing of ceramic tubes consistently places the filaments at nearly the same position for each tube. Thus, from the factory, most new tubes need very little adjustment to align an instrument. This procedure assumes that the instrument is nearly aligned.

1. Power off the generator. Record instrument use in the log book, and record the date of the instrument alignment in the Maintenance log.

2. Remove the mono-capillary collimator and beam stop and replace it with the 0.3 mm collimator without a beam stop. Note that the flat “0.3 mm” marking should be horizontal and the end of the tube

3. Check for water leakage around the tube and housing.

4. Check for radiation leakage around the tube and housing.

5. Move the detector to 5 cm from the sample.

6. Log into the frame buffer computer next to the instrument.

7. Open the “Detector Test” program; click on the “Init” button. Click on the “Rate” button and set the Voltage to 30kV, the Current to 10mA, and hit the “Set” button. Set the Add time to 1 sec and the Repetitions to 99999.

8. Open the cabinet door, adjust the tube position, close the cabinet door and hit the “Start” button. After 3-4 seconds, hit the Abort button. Repeat the actions in this step adjusting the tube position to maximize the intensity from the tube. Maximize

the beam intensity by making adjustments in the following order, the Take-off angle adjustment, the Lateral adjustment, the Height adjustment and the Glancing angle adjustment. Repeat this process until the maximum intensity is reached, this should be > 1 x 10^6 cps. Record the maximum intensity in the Maintenance Log.

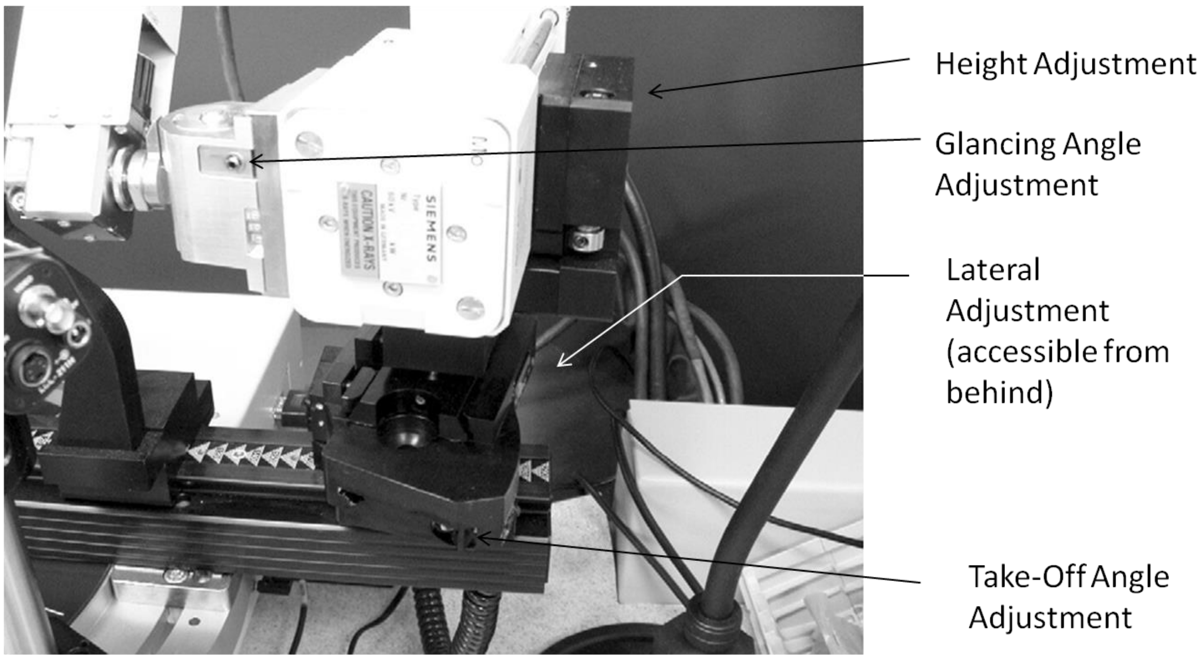

Figure 1. Alignment Adjustments. Adapted from Fig 2.17 of the SMART APEX Installation manual.

Determine an Orientation Matrix

1. Remove the 0.3 mm collimator, and install the mono-capillary collimator with the beam stop.

2. Open the D8Tools program and connect D8Tools to the instrument by selecting “On Line Status” then select the left icon in the lower row of icons. Select the X-ray Generator menu in the left side panel. At the top of the screen select Utilities > X-Ray > Set kV and mA. Set the kV to 50 and the mA to 30. When the screen shows this power level on the generator, exit D8Tools.

3. Open the BIS program, and when asked, confirm the sample-to-detector distance, and minimize the program.

4. Open the APEX3 program, connect to “localhost”, open a new project.

5. Mount goniometer head with the ylid sample. Center the crystal on the instrument using the instructions in the "Center the Sample on the Instrument" SOP.

6. Select Evaluate > Collect Data. Set the count time for 10 seconds per frame.

7. Harvest the spots, index the spots. Refine the cell as orthorhombic primitive.

8. The following items should be within the errors shown

X-Cen	Y-Cen	Dist	ω_o	χ_o
< 1 pixel	< 1 pixel	< 0.01 mm	< 0.03°	< 0.06°

9. If the values for X_{cent}, Y_{cent}, ω_o, or χ_o are greater than shown above, enter the correction(s) in the BPC program, and repeat the Orientation Matrix determination until ω_o and χ_o are within range.

Collect and Process a Hemisphere of Data

1. Use APEX3 to collect data on the ylid sample with the following sets of runs:

#	2θ	ω	ϕ	χ	Axis	Range	#	Time
1	-28	-28	0	54.8	2	-0.5	380	10
2	-28	-28	90	54.8	2	-0.5	380	10
3	-28	-28	180	54.8	2	-0.5	380	10
4	-28	-28	270	54.8	2	-0.5	380	10

2. When data collection is complete, remove the ylid sample from the instrument and secure it.

3. Reduce the data using the SAINT+ program, setting Crystal System in both the "Periodic orientation matrix updating" and the "Post-Integration Global Refinement" to triclinic.

4. When Saint completes running, copy the *_0m.p4p file from the Work directory to the directory with the data frames.

5. Run SADABS with the default selections except setting "Additional Spherical Absorption Correction" to 0.

6. Run XPREP to determine the space group ($P2_12_12_1$) with a composition of $C_{11}H_{10}O_2S$. Retain the <I/σ> value for all data.

G. Records Management

Record the date, operating hours of the X-ray generator, <I> for all data, the <I/σ> for all data, *a*, *b*, and *c* cell parameters and esds from the last integration run, and the R_1 and wR_2 from the final refinement in the Instrument Quality D8_APEX.xlsx spread sheet in the lab manager's office.

H. Quality Control and Quality Assurance

This operating procedure outlines a method to assure that the X-ray diffractometer with the APEX detector is operating at its peak efficiency. The resulting data from this study is used to quantify the state of the X-ray tube in the instrument.

I. References

http://www.ouhsc.edu/rso/
Web site for the Radiation Safety Office of the University of Oklahoma.

SMART APEX Installation Manual, M86-E00057, Bruker-AXS, Madison, WI.

Changing X-ray Configurations, M86-E00099, Bruker-AXS, Madison, WI.

APEX Instrument Operating Instructions, OUCB-CCL-1.002, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.

Center the Sample on the Instrument, OUCB-CCL-4.002, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.