

Chemical Crystallography Laboratory	OUCB-CCL-1
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	March 30, 2017
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Chemical Crystallography Laboratory

Author: _____
Douglas R. Powell

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Distribution

Douglas R. Powell, Laboratory Manager
 Prof. George 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	1.001	Douglas R. Powell	Initial Release
1/30/2013	1.002	Douglas R. Powell	Minor revisions
3/30/2017	1.003	Douglas R. Powell	Minor revisions + software

The following laboratory users have read this manual.

Name	Signature	Date
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A. Scope and Availability

This manual describes general tasks related to starting and stopping the APEX instrument in the Chemical Crystallography Laboratory at the University of Oklahoma. These tasks include emergency instrument shutdown, starting the instrument, shutting down the instrument, starting the low-temperature device, filling the liquid nitrogen dewar, and shutting down the low-temperature device. This manual is publicly available from the Laboratory's web site.

B. Summary of the Method

Steps necessary to perform the activities listed above are given.

C. Responsibility

All users of the APEX instrument are required to follow the steps in these procedures to complete these tasks. Any problems with performing these steps should be immediately reported to the Laboratory manager.

D. Safety and Training

The diffractometer 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 of X-ray radiation provided by the lab manager or from personal reading.

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.

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 Instrument with an APEX detector.
- Oxford Cryosystems Cryostream 700 Low-temperature device.

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F. Procedures

Emergency Instrument Shut Down

1. Hit the red *Emergency Stop* button on either rounded front corner of the instrument cabinet or on the front of the X-ray generator. X Rays are no longer being generated, but the storage capacitors in the generator will continue to hold an electrical charge for at least 15 minutes.
2. Turn off power to other parts of the instrument by depressing the “0” button on the rounded right-hand corner of the instrument cabinet.
3. Turn off power to the Haskris water chiller in room 2803. Close the water valves to the chilled recirculating water.
4. Turn off power to the Neslab chiller on the left side of the instrument.

Normal Instrument Start-up

1. Be sure that power to the D8 goniometer and the detector are turned off. Reset the X-ray generator by twisting the red *Emergency Stop* buttons clockwise on both rounded corners of the instrument cabinet and the front of the X-ray generator.
2. If the frame buffer computer is not running, then boot the frame buffer computer.
3. After the frame buffer computer has been booted, turn on power to the D8 instrument cabinet by depressing the “1” button on the rounded right-hand corner of the cabinet. The D8 controller takes a few minutes to reboot. While it is rebooting, a yellow Busy lamp is displayed on the two front rounded cabinet corners.
4. Turn on the detector by completing the following instructions.
 - Turn on power to the Neslab water chiller located on the left side of the instrument. The Neslab cooler takes about 10 minutes to achieve its running temperature of about 0 °C. Often the Neslab cooler will be left running.
 - Turn on power to the detector by activating the detector power supply that is located in the top-left-side of the cabinet behind the lower front panel. Note that the detector must always be rebooted after the frame buffer computer.

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5. Turn on the X-ray generator by completing the following instructions. If trouble is encountered at any step, contact the lab manager for assistance.
 - Check that the water valves on the lines between the water chiller and the chilled-water supply and return are opened (at a 45° angle to the pipes).
 - Turn on power to the Haskris water chiller in room 2803. The pump motor sounds should be audible.
 - Check that both doors to the upper instrument enclosure are closed and locked.
 - Turn the key on the X-ray generator control panel to the “I” position. The generator is located in the middle-left-side behind the lower front panel of the instrument cabinet.
 - On the generator control panel, depress and hold the *Heater* button for at least 5 seconds or until the green lamp on the button is lit. If the green lamp remains lit, then wait for 5 minutes. The heater button is the black button between the *Off* and *On* buttons. If the green lamp is not lit, depress the *Clear Alarm* button 2-3 times. Each time this button is depressed a relay should be heard. The Clear Alarm button is located on the upper left corner behind the lower front panel of the cabinet. Depress the Heater button again and wait 5 minutes to warm up the filament of the tube. If the relay is not heard when the Clear Alarm button is pushed, then there must be a part of the safety circuit that is incorrectly set – contact the lab manager.
 - Push the *On* button on the generator control panel. Note that the LCD display should now read “kV = 20, mA = 5” and the 4 white X-RAY ON lamps on the generator should be lit.
6. Log onto the frame buffer computer. 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.
7. Open the BIS program, and when asked, confirm the sample-to-detector distance, and minimize the program.
8. Open the APEX3 program, connect to “localhost”.

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Normal Instrument Shut Down

1. Exit the APEX3 program, saving changes to the current project. Close the BIS program and the Video program.
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 20 and the mA to 5. When the screen shows this power level on the generator, exit D8Tools. Wait 15 minutes for the X-ray tube to cool.
3. Shut down the D8 controller by depressing the "0" button on the right side of the rounded corner of the cabinet. This will also turn off power to the generator and the detector.
4. Turn off the Neslab water chiller on the left side of the instrument.
5. Turn off the power to the Haskris water chiller in room 2803 that is closest to the instrument. Close the water valves that connect the Haskris to the building's chilled water loop.

Starting the Low-Temperature Device

1. Turn on the flow of dry, room temperature gas to the instrument either by opening the valve to the stopcock on the nitrogen supply line behind the instrument or by turning on power to the AD41 air compressor in room 2803. Be sure that the tube with this dry, room-temperature gas is connected to the lower connection of the Oxford cold head.
2. Fill the 60 liter Dewar behind the instrument with liquid nitrogen (see instructions below).
3. Turn on power to the low-temperature controller. The power switch is located on the back right side of the device. Wait for the controller to initialize. If the lamp above the *Display* button is lit, depress the *Display* button to turn off the lamp.
4. Depress the blue switch labeled *Start*. The controller is set to automatically cool to 100 K and hold this temperature setting. Cooling from room temperature to 100 K takes about 20 minutes.

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Filling the Liquid Nitrogen Dewar

1. If it is not already attached, connect the flexible metal tubing from the 60 liter Dewar to the *Liquid* connection of the large 180 liter supply Dewar.
2. Open the *Liquid* valve of the large supply Dewar just enough to hear a repeating surge sound. Wait until this surging sound stops (usually 1-2 minutes) then open the valve fully.
3. When small amounts of liquid nitrogen begin to splash out of the top of the 60 liter Dewar, close the *Liquid* valve on the supply Dewar.
4. If a whistling sound is heard from the 180 liter Dewar, the Dewar is empty.

Low-Temperature Device Shut Down

Stopping the controller using Cryopad

1. Close the BIS program.
2. Open the Cryopad program on the frame buffer computer. The program should open to the Display panel showing the current condition of the controller. If the connection to the controller is not working properly, be sure that the program is using the COM2 port, in the Settings panel, to communicate with the controller.
3. In the Command panel set the option to End, and hit the Execute button. If a message from the controller does not show that the End program is being executed after about 10 seconds, then hit the Cancel button and repeat this step until the controller acknowledges the command.

Stopping the controller using the controller itself

1. On the controller depress the *Program* button.
2. Rotate the round knob until the word *End* is displayed. Add this item to the phase table by hitting the *Enter* button twice. If the *Program* is still lit, depress it to make the light turn off. Depress the *Hold* button to release the hold state.

Final steps

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4. When the low-temperature device pump turns off in 20-30 minutes, turn off power to the controller.
5. Stop the flow of dry, room-temperature gas to the cooling head by either closing the stopcock to the house nitrogen gas or turning off the AD41 air dryer in room 2803.

Disposal of Sample Material

Disposal of sample material is the responsibility of the user that supplies the sample. Samples that are not pyrophoric are removed from glass slides and tools by wiping with a KimWipe and placed in the solid waste container near the microscope.

G. Records Management

Users are to log all experiments in the log book that is kept on the table with the frame buffer computer. Equipment malfunctions are recorded in a Maintenance Log book kept by the lab manager.

H. Quality Control / Quality Assurance

The instrument is periodically realigned and checked for proper functioning of the safety equipment and optimum data collection. See Alignment and Calibration of the APEX Instrument.

I. References

<http://www.ouhsc.edu/rso/>

Web address of the Radiation Safety Office of the University of Oklahoma.

Alignment and Calibration of the APEX Instrument, OUCB-CCL-2.003, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.