

Chemical Crystallography Laboratory	OUCB-CCL-4
Department of Chemistry and Biochemistry	Version 002
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

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The following laboratory users have read this manual.

Name	Signature	Date
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A. Scope and Availability

This procedure describes centering samples and measuring their dimensions as performed on a Bruker diffractometer with an APEX detector using the SMART and VIDEO programs. A copy of this manual will be publicly available on the Laboratory's web site.

B. Summary of Method

This procedure describes centering samples and measuring their dimensions.

C. Responsibility

Trained lab users may perform these tasks.

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.

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.

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.
- Sample mounted on a goniometer head.

F. Procedure

1. Carry the mounted sample and goniometer head to the instrument with the sample pointing down. Place the goniometer head on the instrument. If the sample is to be cooled to a low temperature, then raise the sample slowly into the gas cold stream.
2. Note that all goniometer heads are keyed where they attach to the goniometer. Place the goniometer head onto the goniometer and tighten the head "snugly" or "finger tight". Use no tools!

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3. If the instrument is not turned on, then follow the procedures in the APEX Instrument Operating Instructions manual to turn on the instrument. Log into the frame buffer computer, and open the APEX3 program. The username and password for the program are “guest” and “guest”.
4. Open a new *project* by opening the Sample > New menu. The name of the *project* can be any unique value. In this lab, it is common to use a number as the *project* name where the first two digits are the last two digits of the current year and the next three digits are the next serial number of projects. Check the log book if you are not sure what the next *project* should be. The directory should be set to c:\frames\project.
5.
 - a) In the Set Up > Center Crystal menu select “Right” to move the goniometer to a good position for adjusting the position of the crystal. If the Video
 - b) Move the crystal to the center of the crosshairs of the Video program screen.
 - c) Rotate the sample by 180° in phi.
 - d) If the center of the sample is not located at the rotation center, then move the sample ½ of the way between its current position and the rotation center.
 - e) Rotate the crystal by 180° in phi to confirm that the rotation center of the crystal remains unmoved. If the center of the crystal is not at the rotation center, return to d).
6.
 - a) Rotate the sample by 90° in phi.
 - b) If the center of the sample is not located at the rotation center, then move the sample ½ of the way between its current position and the rotation center.
 - c) Rotate the crystal by 180° in phi to confirm that the rotation center of the crystal remains unmoved. If the center of the crystal is not at the rotation center, return to b).

Check the Height Location

All previous adjustments have been made by centering the sample on a rotation axis that is inclined by about 54.7° from the horizontal plane.

1. Select the “Left” button to move the goniometer head by 180° in omega.
2. Move height of the crystal until the crystal is centered on the rotation axis of the instrument established in the steps above.
3. Move the goniometer head back to the “Right” position.

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Measure the Dimensions of the Sample

1. Select the Set Up > Screen Crystal. In the right-side Preset Position panel select Current and then Drive.
2. The crystal is moved by small adjustments of the Phi slider followed by hitting the Drive button. Repeat this step until a pair of faces appear as edges of the crystal.
3. Measure the shortest distance between the faces (that are now seen along their edges) by moving the cursor to one face and dragging it in a perpendicular path to the opposite face. The distance appears in the lower right corner of the VIDEO window with the number in units of microns (1 mm = 1000 micron). Retain this dimension in mm.
4. Repeat the last two steps for the other two directions of the sample. Retain these dimensions in mm.
5. Drive the instrument to 0° for 2theta, omega, and phi by selecting the Zero button in the Preset Position panel and hit Drive.

G. Records Management

Retain the dimensions of the sample for later reference.

H. Quality Control and Quality Assurance

Care with centering the sample produces better values for cell parameters with smaller standard uncertainties and produces better measured intensities.

I. References

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

Web site for the Radiation Safety Office of the University of Oklahoma.