Quality Assurance Plan

March 30, 2017

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	001	Douglas R. Powell	Initial Release
1/30/2013	002	Douglas R. Powell	Minor revisions
3/30/2017	003	Douglas R. Powell	Minor revisions

Table of Contents

A. Project Summary

Provide a single crystal chemical analysis service of small-molecule compounds to the students and researchers at the University of Oklahoma and more broadly to the citizens of the state of Oklahoma. A chemical analysis of a single crystal provides a geometrical description of the molecular and ionic species found in the crystal. Crystals are made up of repeating blocks of molecules. Because of the nature of the interaction between the radiation source and the atoms in these molecules, a model of the molecules from an "average" unit cell is produced.

This laboratory strives to produce the best models of the molecular geometry available from the crystals provided and the department's instrument. This plan describes the activities carried out to assure that the best models are produced.

B. Project Description

The diffraction experiment involves shooting a beam of nearly monochromatic X-rays at a crystalline sample, measuring the relative intensities of the resulting diffraction pattern, and analyzing the diffraction data to determine the location and types of atoms (the model). From these atom locations, the bonding geometry between the atoms is determined.

Our lab follows the guidelines for collecting and processing data, and refining chemical models that are set forth by the International Union of Crystallographers, IUCr, as described in the structure verification program checkcif (http://checkcif.iucr.org/). This verification program is utilized by the editors of all major journals to evaluate the quality of a small-molecule crystal structure that is included in any submitted manuscript.

Small-molecule crystal structure analyses typically produce more data than is needed to uniquely determine the model, often producing 10-20 times as many unique data as there are variables in the model. These highly over-determined models nearly always lead to an unambiguous result. A statistical analysis of the data set is carried out as a part of each analysis. Standard uncertainties for the intensity data, the cell parameters, the atomic parameters, and the derived geometry objects are always reported.

The crystal structure determinations conducted in this lab utilize a Bruker APEX ccd detector mounted on a 3-circle D8 goniometer with a sealed tube X-ray source, and a mono-capillary collimator. The samples are routinely cooled to 100 K using a nitrogen-gas streaming, Oxford Cryostream700 low-temperature device. This instrument was installed at the University of Oklahoma in 2002.

C. Quality Objectives and Criteria for Measurement of Data

Crystals are selected based upon their size and appearance. Single crystals are much easier to analyze than twinned crystals. Larger crystals tend to produce stronger (better) data sets. Experimental conditions such as counting time, thickness of slices of diffraction space, and sample-to-detector distance are optimized to produce the best data for the selected crystal. Typically, several crystals are screened to find the crystal with the strongest diffraction pattern for any sample.

When possible, the counting time per image is adjusted to produce observable intensites at the highest scattering angles. Using this criteria to select the counting time, data will typically produce an average $I/\sigma(I)$ ratio of > 10 (the prefered level of precision used in this lab). Unfortunately, not all crystals diffract to IUCr minimum limits of $\sin \theta_{max} / \lambda > 0.6$ Å^{-1}. Data are still collected on these samples presuming that some description of the crystal structure is better than no description of the crystal structure.

The solid angle thickness of slices is routinely set at 0.5° so that each diffraction maximum appears on 2-3 consecutive slices (images) of diffraction space. When the samples scatter poorly producing large spots, the thickness of each image is adjusted to make 2-3 slices through each spot.

In most cases, leaving the sample-to-detector distance at 5 cm is sufficient. However, when a unit cell axial length in the sample is > 25 Å, the diffraction maxima appear to blend together forming diffraction lines. The sample-to-detector distance is increased to produce diffraction patterns with discrete spots. Note that when this distance is > 7 cm, then data must be collected with at least two swing-angle detector settings to collect sufficient data for the IUCr minimum standards.

To attain the best quality measurements of our diffraction intensities, multiple measurements of the symmetry-unique data are made. For systems with monoclinic or higher symmetry, our lab tries to achieve an average data redundancy of at least 4. For triclinic systems, our lab tries to achieve an average data redundancy of at least 3.

D. Special Training and Certification

All users of the facility must have annual training in radiation safety from the University's Radiation Safety Office (http://www.ouhsc.edu/rso/), as well as annual training from the University's Environmental Health and Safety Office in chemical hazards, chemical waste disposal, and fire safety. The lab manager should have training from the manufacturer on the operation and maintenance of the instrument. Other users of the facility will be trained by the lab manager in the use of the instrument and computer software.

E. Documents and Records

Records of the radiation safety training will be maintained by the University's Radiation Safety Office. Records of the chemical and fire safety training will be maintained by the University's Environmental Health and Safety Office. Records of lab user's training will be maintained by the CCL laboratory manager.

F. Quality Control

Users are to follow the procedures outlined in the series of standard operating procedures prepared for the Chemical Crystallography Laboratory.

APEX Instrument Operating Instructions	OUCB-CCL-1.003	3/30/2017
Alignment and Calibration of the APEX Instrument	OUCB-CCL-2.003	3/30/2017
Mounting Samples	OUCB-CCL-3.003	3/30/2017
Center the Sample on the Instrument	OUCB-CCL-4.003	3/30/2017
Data Collection Using APEX3	OUCB-CCL-5.003	3/30/2017
Data Reduction and Processing	OUCB-CCL-6.003	4/10/2017
Chemical Waste Disposal from Detector Chiller	OUCB-CCL-7.002	3/30/2017

All crystal structures determined in this lab will be evaluated using the checkcif program developed by the International Union of Crystallographers (http://checkcif.iucr.org/). Serious issues such as A and B level alerts must be addressed in a lab report describing the structure.

G. Instrument Testing, Inspection, and Maintenance

Weekly checks of the general laboratory conditions will be made to assure that all pieces of equipment are working as specified. Problem issues will be noted and addressed as soon as possible.

The precision of the instrument will be assessed by the results of a semi-annual analysis of a standard sample, provided by the manufacturer. For these analyses, the instrument will first be aligned according to the procedures in Alignment and Calibration of the APEX instrument. Then intensity data will be collected and processed by a standard procedure, and the values of average I, average $I/\sigma(I)$, cell parameters, and refined R_1 and wR_2 kept by the lab manager as a permanent lab record for this instrument.

Annually these preventive maintenance steps will be performed: check and clean all screens in the water cooling system for the generator, replace the cooling water in the Haskris and Neslab

water coolers, clean and lubricate the gears of the omega and 2theta drives, empty, drain, and dry the 60 liter supply Dewar for the LT device, check all voltages in the D8 controller using the D8Tools program.

Assessment and Reporting

The checkcif report for each crystal structure will be included with data files for each project. External assessment of crystal structures submitted for publication is provided by reviewers of manuscripts that include the crystal structures.

Users are encouraged to discuss ideas for improving lab activities with either the lab manager or the faculty supervisor chairing the lab faculty committee. Annual surveys will be sent out to all users asking for additional input. All suggestions will be strongly considered and, when possible, will be implemented.

An annual report of the productivity of the facility is presented to both the chair of the faculty oversight committee and the Department's assistant chairman.

References

http://checkcif.iucr.org/
Web site to submit crystal structure model and data to verify and validate one or more crystal structures.

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

APEX Instrument Operating Instructions. OUCB-CCL-1.003, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, 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.

Mounting Samples, OUCB-CCL-3.003, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.

Center the Sample on the Instrument, OUCB-CCL-4.003, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.

Data Collection Using APEX3, OUCB-CCL-5.004, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.

Data Reduction and Processing, OUCB-CCL-6.003, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.

Chemical Waste Disposal from Detector Chiller, OUCB-CCL-7.002, Chemical Crystallography Laboratory, Department of Chemistry and Biochemistry, University of Oklahoma.