

Chemical Crystallography Laboratory	OUCB-CCL-6
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	April 10, 2017
Data Reduction and Processing	Page 1 of 7

Data Reduction and Processing

April 10, 2017

Chemical Crystallography Laboratory

Author: _____
Douglas R. Powell

Chemical Crystallography Laboratory	OUCB-CCL-6
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	April 10, 2017
Data Reduction and Processing	Page 2 of 7

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	6.001	Douglas R. Powell	Initial Release
1/30/2013	6.002	Douglas R. Powell	Minor revisions
4/10/2017	6.003	Douglas R. Powell	Software revisions

The following laboratory users have read this manual.

Name	Signature	Date

Table of Contents

A. Scope and Availability	4
B. Summary of Method	4
C. Responsibility	4
D. Safety and Training	4
E. Equipment and Supplies	4
F. Procedure	4
G. Records Management	5
H. Quality Control and Quality Assurance	5
I. References	5

Chemical Crystallography Laboratory	OUCB-CCL-6
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	April 10, 2017
Data Reduction and Processing	Page 3 of 7

A. Scope and Availability

This manual discusses data reduction and absorption correction using the stand-alone programs SAINT and SADABS or TWINABS from Bruker. A copy of this manual will be publicly available on the Laboratory's web site.

B. Summary of Method

Data reduction in SAINT includes the steps of integrating the data, correcting data for counting time and background, correcting the data for Lorentz and polarization effects, sorting and merging the data, and performing a least-squares fit on cell parameters. Absorption correction(s) and scaling are performed in SADABS or TWINABS.

C. Responsibility

The user or person in charge of collecting data should perform these tasks.

D. Safety and Training

Direct questions about this procedure to the lab manager.

E. Equipment and Supplies

- Computer with the software and the data files.

F. Procedure

1. Transfer files to the computer that will be used to integrate the data. If the integration computer is \\xray8 then use file explorer to transfer the files to the "frames3\$" share.
2. Log into the computer that will be used to integrate the data and start up the APEX program. Log into the program with the username / password of guest / guest.
3. Begin a new project with the Sample > New option. Set the current directory to the C:\frames\project\ directory.
4. Import the cell parameters and crystal information using the Sample > Import option. If the cell is acceptable, then skip the next step.
5. Improve or determine cell parameters by:
 - a. In the left-side panel Use the Evaluate > Determine Unit Cell and select Harvest Spots in the right-side panel to locate a set of spots. Harvest only from runs

Chemical Crystallography Laboratory	OUCB-CCL-6
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	April 10, 2017
Data Reduction and Processing	Page 4 of 7

collected with omega scans. General harvesting can be done from say the first 50 frames of the first 5 runs. If the sample is likely a split crystal or a non-merohedral twin, then use all frames from a single omega-scan run.

- b. Index data using the Index option. Accept the most likely cell. If the data do not index well (> 90% of the spots are indexed with a Score > 1.0), see below.
- c. Improve the cell parameters in the Refine option. In the first refinement run set the tolerance to about 1.0. Increase the tolerance to 1.5 and refine again. The tolerance may be increased further if desired. When finished, Accept the cell parameters.
- d. Check for possible higher metric symmetry (and reduce the cell) with the Reduce option. Be careful of accepting any cell with a figure of merit < 0.5.
- f. Improve the cell parameters with the Refine option. See part c, for how to use the tolerance.

If attempts to index the cell do not use > 90% of the data, then the cell is probably split or twinned. If the spots are relatively sharp, then the cell can be determined by the following steps.

- a. Harvest spots from all of frames in a single omega-scan run.
- b. Save the spots in a *.p4p file using the Sample > Export menu.
- c. Open a command prompt window and set the current directory to the C:\frames\project\ directory.
- d. Run the "cell_now" program using the *.p4p file created in step b. above. Select most of the defaults for other parameters. If you have some idea what the cell parameters could be, then be sure that the minimum and maximum cell lengths bracket the probably cell lengths. The first cell listed may not be the correct cell. Accept the spots that agree within 0.15 – 0.20 tolerance of the proposed cell.
- e. If the proposed lattice is primitive then perform the following tasks. Open a second command prompt window. Start the program XPREP with no data file. Enter <return> until asked for the cell. Enter the proposed cell parameters. Reduce the cell. If the new cell is different from the cell from cell_now, then in cell_now enter the "R" option to reorient the cell and enter the transformation matrix shown by XPREP. The transformation will look like (but not be) "1 0 0 0 1 0 0 0 1".
- f. Save the cell_now cell as the "temp1.p4p". Select the "S" option to look for more twin components.
- g. Use the same tolerance as was used in d above. Save the cell as "tempn.p4p", where "n" is a larger integer for each new twin component. Repeat this step until no more components can be indexed (the number of remaining spots is a small number).
- h. Close the cell_now program and the command prompt window. In the APEX program, read in the unit cells using Sample > Import.

Chemical Crystallography Laboratory	OUCB-CCL-6
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	April 10, 2017
Data Reduction and Processing	Page 5 of 7

6. In the APEX program open the Reduce Data >Integrate Images option. If the Resolution Limit is a larger value than 0.82 Å, then reset it to 0.82.
7. In the right-side option “Find Runs” move to the directory containing the frame data and select those all runs.
8. In the Refinement Options menu:
 - a. Uncheck the “Enable Orientation Refinement” and the “Enable Box Size Refinement” boxes.
 - a. Set the Periodic Refinement cell (on the left) to always “Triclinic” symmetry.
 - b. Set the symmetry in the right-side panel (the symmetry of the cell parameters during cell-parameter refinement) to the “Triclinic” for all but the last integration. Before the last integration, constrain the cell parameter to the value appropriate for the Laue symmetry of the sample.
 - c. Set the box size dimensions to roughly the size of anticipated for the spots. In particular, set the X and Y box dimensions to 0.95. Improve the box dimensions in later runs by setting these values to average values for X, Y, and Z accrued during a data reduction run.
9. In the Integration Options menu:
 - a. Set the “Active Image Queue Half-Width (Images) to at least “12”.
 - b. For twinned samples select “More Options” and change the overlap factor to “15.0”.
 - c. If the diffraction pattern is streaky or the spots are large, consider changing the Profile XYZ Half-Widths to a larger single integer, say “6” or “8”, and change the item in a. above to a value of “20” to “24”. Try different values to get the best results (largest <I> for the whole data set).
10. Integrate the data. When an integration is complete, read in the new orientation matrices with the Sample > Import option reading the “*project_0m.p4p*” file, on the Refinement Options menu update X, Y, and Z box dimensions and check the “Enable Box Size Refinement” box.
11. Repeat integration. When an integration is complete, read in the new orientation matrices with the Sample > Import option reading the “*project_0m.p4p*” file, check the “Enable Orientation Refinement” box. In the right-side panel, constrain the cell parameters as is needed by the Laue symmetry.
12. Repeat integration.

Chemical Crystallography Laboratory	OUCB-CCL-6
Department of Chemistry and Biochemistry	Version 003
University of Oklahoma	April 10, 2017
Data Reduction and Processing	Page 6 of 7

Scaling or Absorption Correction.

Run either the SADABS or the TWINABS program to scale and correct the data for absorption effects. SADABS is used for single crystal samples; TWINABS is required for twinned samples. In either case, save the listing file as the “*project.abs*” file. The input file is typically “*project_0m*”. On the first pass of scaling, set the symmetry to the Laue symmetry of the cell. When the space group is later determined and the point group of the space group is lower symmetry than the Laue symmetry, then before the final structure refinement, rerun scaling using the proper point group symmetry. If the calculated “g” value is > 0.07 then set the value to “0.07”. Be sure to save the resultant intensity data file to “*project.hkl*” for the hkl-4 file and “*project.hkl5*” for the hklf-5 file. In TWINABS the hkl-4 file is usually selected from the strongest domain, and the hkl-5 file is usually selected from all domains.

G. Records Management

Retain all data files for archiving.

H. Quality Control and Quality Assurance

The executing multiple runs of Saint improves the quality of the intensities and the values of the cell parameters.

I. References

SAINT Software Reference Manual. (2007) Bruker-AXS, Inc., Madison, Wisconsin