Collect Intensity Data – Photon II

General Steps in Collecting Intensity Data

Note that the steps outlined below are generally followed when using all modern automated diffractometers, regardless of manufacturer.

1. Mount crystal using a microscope.
2. The mounted crystal is *centered* on the different axes of the goniometer. This also places the sample in the path of the incident X-ray beam.
3. One or more preliminary images of the diffraction pattern of the sample are taken to verify the sample's crystallinity, mosaicity, and scattering power.
4. A preliminary unit cell is determined.
5. A data collection strategy is determined. Intensity data are collected.
6. Auxiliary information about the absorption correction or cell parameters may be collected.

The following specific instructions are for collecting intensity data using the department's Bruker Quest Kappa-geometry goniometer with a Photon II cmos (complementary metal-oxide semiconductor) detector using the APEX3 software.

Mount the Crystal

The crystal should be selected and mounted using instructions in the "Microscope.pdf" handout. As a reminder, the crystal should generally be mounted on the end of a plastic loop, plastic support, or glass fiber using an appropriate adhesive. Crystals with all dimensions ≤ 0.3 mm are preferred. Crystals should have no dimensions > 0.5 mm.

Specific Instrument Steps

1. Log into the computer next to the instrument using the xtal account. If the BIS and APEX3 programs are not running, then first start BIS. Starting BIS may also start the Measurement Server program. The MEASUREMENT SERVER program will show a diagram of the instrument; minimize this image. While the BIS program is loading, it will show a green progress bar along the bottom of the BIS window; minimize BIS when done. Open the APEX3 program. If the program asks for a username and password enter "guest" and "guest". Connect the APEX3 program to BIS by selecting Instrument > Connect and enter the value "bruker3".

2. In APEX3, open a new *project* by selecting the Sample > New menu. The name of the *project* can be any unique value. In this lab, it is common to use a 5-digit number as the "*project*" 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 for that year. Check the log book if you are not sure what the next "*project*" should be. The directory should be set to c:\frames*project*.

3. The crystal is centered on the instrument using the *Set Up > Center Crystal* option. Select *Center* if the instrument is not already in a good position for adjusting the goniometer head (reminder: the instrument door must be closed to move most of the instrument motions). Select the "video" icon, a blue five-sided object with a green ">", and then select the round grid icon. Open the cabinet by clicking the lower button on the right side of the upper-front instrument panel. Mount the goniometer head with crystal onto the instrument. Move the crystal horizontally and vertically to the center of the screen by adjusting the translational motions of the goniometer head. Rotate the sample 180° in φ. If the sample is not centered, then move the sample until it is ½ of the distance between its current position and the current "centered" position. Rotate the sample by 180° in φ to verify that the crystal is now centered on this new spot. If the crystal is not centered then again translate the crystal ½ of the distance between its current position and the current "centered" position, and repeat until the center of mass of the crystal does not move with a 180° rotation in φ. The center of mass of the crystal is the new "centering point". Then rotate the sample by 90° in φ to adjust the other translation of the goniometer head to the new "centering point".

Make measurements of the three crystal dimensions by first selecting the double-headed arrow icon. Drag the mouse between the two faces to measure the shortest distance between those faces. The distance appears just below the crystal display in the right side in units of micrometers. Multiply this distance by 1000 to convert to the value to units of millimeters. Repeat this step for the other two approximately perpendicular directions of the crystal.
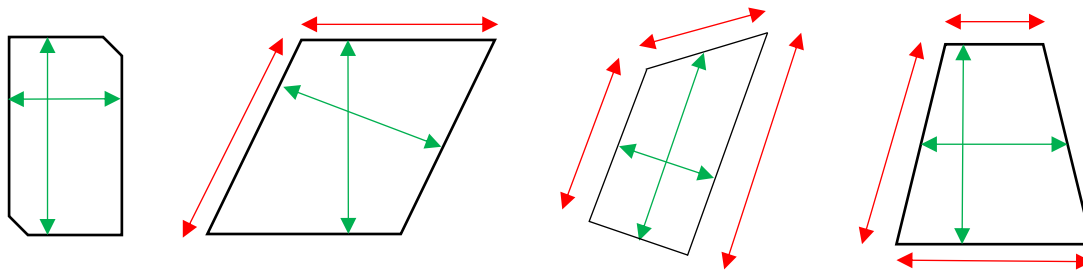

The images above show how crystals should be measured. The green (inside) lines show where measurements should be made; the red (outside) lines show incorrect (large) measurements. When opposite faces are not parallel, select an average distance between the faces. Record these distances on a piece of paper or on the notepad that can be opened by clicking the yellow notepad in the upper right corner of the APEX3 program. Close the instrument door.

4. In the *Set Up > Screen Crystal* routine, select the *Screen* button in the upper right menu of the screen and hit *Drive*. After the goniometer has stopped, then in the lower right panel, select 360° *Phi* and then *Drive & Scan* to record a rotation image. After about 1 minute, an image should appear on the screen. The resulting image should show two distinct mirror planes of symmetry in the spots. In the example below left, note that the mirrors are offset from the horizon by about 40°. The shadow appearing on the center of the image and extending to the upper edge is the shadow of the beam stop and its support arm. The mirror planes have nothing to do with the symmetry of the sample and are an

artifact of the type of image collected (full-rotation image). Note that if the mirror planes are not observed in this rotation image, then either the sample is not correctly centered on the instrument or the instrument itself is not correctly aligned. Some spots may be overlapped, but many should be isolated. Remember, the sharper the spots then the smaller the mosaicity of the crystal (usually a good thing). If the pattern is OK, continue to step 5 below; if not mount another crystal and return to step 3 above.

Two rotation images are shown below. The image on the left shows a good quality crystal. The image on the right shows the diffraction rings of a polycrystalline material (with preferred orientation—few large crystallites).
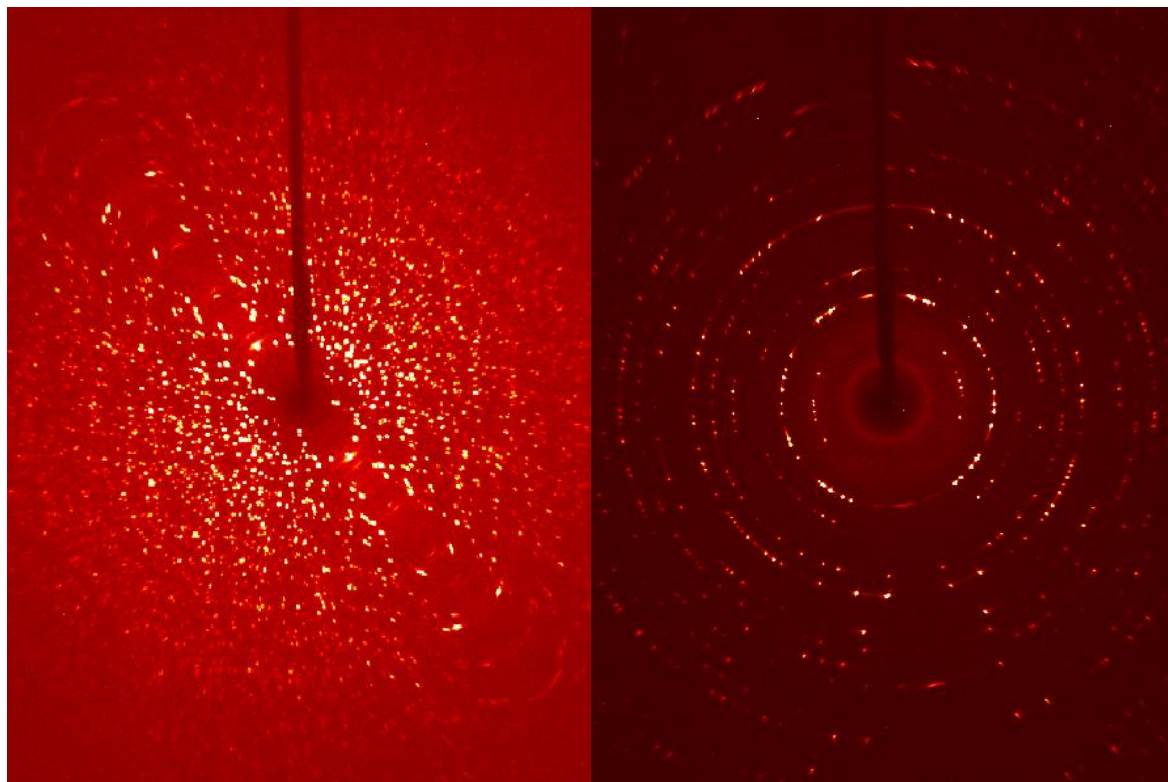
5. In the *Set Up > Describe Sample* menu, enter your sample name (usually your lab notebook reference), the chemical composition (without spaces), the primary crystal color, the crystal dimensions in millimeters determined above, and a brief description of the crystal (often "needle", "plate", or "block" without quotes).

Data-Collection Strategy and Collect Data

Most data-collection strategies require that you first determine a preliminary unit cell. Intensity data are collected and peaks are "harvested" to determine their relative positions and intensities. Then the peaks are "indexed" to assign (*hkl*) indices to each spot and to determine an orientation matrix and initial, primitive (reduced) cell parameters. The cell is then checked for higher metric (Bravais lattice) symmetry and refined again with possibly new cell constraints. The orientation matrix is then passed to the strategy program to select an optimized set of runs to collect data. The Laue symmetry is presumed from the cell parameters, but remember, the true symmetry of the sample can always be *lower* than the symmetry expected from the cell parameters alone.

There are three main methods to determine a data collection strategy. The method directly below is usually preferred. The alternative The 1st alternative method can give cell parameters but cannot be used to verify the structure. The 2nd alternative method works best if you have very little set-up time, but plenty of time to collect data.

1. In the *Collect > Run Experiment* menu, place the mouse over the first *No Operation* window. Use the right mouse button to pull down a menu of operations and select a *Fast Scan*. Hit the *Execute* button. If spots are close together, stop data collection, increase the crystal-to-detector distance, and restart data collection. If spots do not extend to the edge of the image, consider increasing the count times and restart data collection. When data collection is finished note the number of Topped spots as shown in the BIS window.

2. After the data are collected, go to the *Evaluate > Determine Cell* menu and select *Harvest Spots*. Set the number of frames to 180 or the number of frames you collected and select *Harvest*. When sufficient spots are harvested, select "Index" at the top of the screen and then "Index" at the bottom of the next screen. A unit cell is usually acceptable if the "Score" for a given cell is > 1.0 and $> 90\%$ of the peaks are indexed. If no acceptable cell can be determined, then see the notes below about handling a twin by non-merohedry. Accept the cell and refine the cell parameters. In the first refinement pass, refine with a tolerance level of about 1.0. Hit the refine button several times until the volume stops changing. Follow this by refinements at a tolerance level of about 1.5. Accept the results, and in the next menu select the *Bravais* routine that only looks for possible higher-metric-symmetry cells. Accept the cell with the highest score (> 0.5). The program then directs you to refine the cell with constraints on the cell parameters.

3. The data collection strategy is determined from the *Collect > Calculate Strategy* menu. Select the required resolution ($0.45 \leq 0.82$ Å) of the data and the non-centrosymmetric crystal symmetry and click *Apply*. Click the *Determine Strategy* button, set the sample-to-detector distance to ≥ 37.0 mm (larger values for a cell axis > 25 Å or a twinned sample). A good rule of thumb is to set the sample-to-detector distance (in mm) to at least 2 times the longest axis (in Angstroms). Set the *Redundancy* to ≥ 2 and click *OK*. The program will calculate an optimal strategy for collecting the data. Then use *Scan Parameters* menu to select the scan width ($\leq 1.0°$) and count time (≥ 10 seconds) and click *OK*.

4. On the *Collect > Run Experiment* menu, select *Append Strategy* to upload the strategy determined earlier. If the number of topped peaks in the Fast Scan run was > 5, add another Fast Scan run with either ½ of the time of the original Fast Scan or twice the scan width of the original Fast Scan. Add a *Position* task to the run list and set the 2Theta value for the position task to "0.000".

5. Select the *Resume* button to start data collection.

6. After data collection has begun, integrate, scale, solve the structure using the Fast Scan data, and roughly refine to verify the Laue symmetry and composition.

1st Alternative Data Collection Path

1. In the *Evaluate > Determine Unit Cell* menu select *Collect Data* on the right then click *Collect*. If the spots appear in rows that are too close to each other, cancel data collection, increase the sample-to-detector distance and restart data collection. After the data are collected, *Harvest Spots* the spots and select "Index" at the top of the screen and then "Index" at the bottom of the next screen. A unit cell is usually acceptable if the "Score" for a given cell is > 1.0 and > 90% if the peaks are indexed. If no acceptable cell can be determined, then see the notes below about handling a twin by non-merohedry. Accept the cell and refine the cell parameters. In the first refinement pass, refine with a tolerance level of about 1.0. Hit the refine button several times until the volume stops changing. Follow this by refinements at a tolerance level of about 1.5. Accept the results, and in the next menu select the Bravais routine that only looks for possible higher-metric-symmetry cells. Accept the cell with the highest score (> 0.6). The program then directs you to refine the cell with constraints on the cell parameters.

2. Go to *Collect > Determine Strategy* to determine an optimum strategy. Select the appropriate point group symmetry and resolution (≤ 0.82 Å) and click *Apply*. Then select *Determine Strategy* and select the appropriate sample-to-detector distance (≥ 37.0 mm). Larger distances should be selected if one or more cell lengths > 25 Å or if the crystal is twinned. A good rule of thumb is to set the distance (in mm) to at least 2 times the longest axis (in Angstroms). Select redundancy (≥ 2) and click *OK*. Set the count time to 10 seconds or less and set the scan width to ≥ 0.5 °.

3. On the *Collect > Run Experiment* menu, select *Append Strategy* to upload the strategy determined earlier. Add a *Fast Scan* run to the run list. Add a *Position* task to the run list and set the 2Theta value for the position task to "0.000".

4. Select the *Resume* button to start data collection.

5. After data collection has begun, integrate, scale, solve the structure, and roughly refine to verify the Laue symmetry and composition.

2nd *Alternative Data Collection Path*

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1. In *Collect > Run Experiment* hit the *Load Table* button and select the file "c:\frames\stand-tric.exp". Adjust the count times from the rotation image. If spots are too close, increase the sample-to-detector distance.

2. Select the *Execute* button to start data collection.

Indexing a Twinned Lattice

If attempts to index the cell do not use > 90% of the data, then the crystal is probably split or twinned. If the spots are relatively sharp, then perform the following steps to determine the cell. First be sure that most, if not all, of the data are collected. If spots were harvested earlier, delete those harvested spots. Harvest spots from a single, omega-scan run (usually all frames in that run). Save those spots in a *project*.p4p file using the Sample > Export menu. Open a command prompt window. Change the working directory to the directory that contains the *.p4p file (usually "c:\frames*project*\work"). Run the CELL_NOW program. Enter the name of the *.p4p file when prompted. Take the defaults for other variables. If you have some idea what the cell parameters could be, then be sure that the minimum and maximum cell lengths bracket the likely cell lengths.

Several cells will usually be given with a hint as to the Bravais lattice type in the far right column. Set the tolerance to 0.15, and select the most likely cell. Check that the cell is the best cell by the following. (1) open another command prompt window, (2) start the XPREP program without a project name, hit enter when prompted for the data file name, (3) enter the cell parameters and presumed lattice type given by CELL_NOW, (4) reduce the cell, check for higher metric (Bravais) symmetry. If the cell from XPREP is the same as the cell from CELL_NOW, then simply proceed in CELL_NOW to find other orientations of this cell in the intensity data. If the cells are different, then in CELL_NOW select the first cell, then enter "R" to reorient the cell and type in the transformation matrix from XPREP. The transformation matrix looks like: "1 0 0 0 1 0 0 0 1" but will not be the values shown here.

The cell is saved to a new file such as "temp1.p4p". Then select "S" to search for that cell in another orientation. Use the same tolerance of 0.15 for all other cells. Other cell orientations are saved to files such as "temp2.p4p" for the second cell, "temp3.p4p" for the third cell, etc. When the "remaining spots" are a very small number or when the "uniquely indexed" spots are a small number compared to the "remaining spots" then exit CELL_NOW. Copy the final cells (orientation matrices) to the APEX3 program using the Sample > Import menu and selecting the last "temp*n*.p4p" file.