8. Examine Data

In this step, the space group for the sample is determined and optional simulated precession photographs are calculated to further evaluate the overall quality of the data. APEX2 provides two tools for this: XPREP for space group determination and Precession Images for looking at undistorted slices of reciprocal space. This is the final step before beginning the structure solution and refinement process.

8.1 XPREP

1. Click on XPREP (Space Group Determination) under Examine in the task bar.
2. Check that the two files in the pop-up window are correct (see Figure 8.1) and click OK.

![Select Files For XPREP](image)

Figure 8.1 - Select files for XPREP input

**NOTE:** In this example, the scaling process has created two files: ylid_manual_0m.p4p containing the final unit cell parameters from integration and ylid_manual_0m.hkl containing the corrected intensities. Typically, these are the files to use for determining space groups, but you can browse to choose other files.
8.2 Space Group Determination

1. XPREP evaluates the data and looks at the mean intensities and the mean int/sigma. Since these are large for all groups except P, XPREP suggests that the lattice is P (see Figure 8.2). Press Enter to accept.

   Figure 8.2 - Lattice statistics

2. XPREP determines the reduced cell based on the lattice entered above. Since the lattice was primitive and the magnitudes of the cell dimensions were proper (a<b<c), the original and reduced cells are the same (see Figure 8.3). Press Enter to search for a higher symmetry cell.

   Figure 8.3 - Reduced cell

For the YLID, no higher symmetry cell is found. The program has determined that the YLID crystal has an orthorhombic primitive lattice (see Figure 8.4). Press Enter to accept.

   Figure 8.4 - Higher symmetry cells
3. The next logical step is to determine the space group. XPREP suggests this (see Figure 8.5). Press Enter to determine the space group.

4. If the space group is known or if the compound is known to be chiral, enter that information (see Figure 8.6). Generally, it is sufficient to press Enter to start the space group determination.

5. XPREP has chosen the crystal system [O] (see Figure 8.7). Press Enter to accept.

6. XPREP has chosen the crystal lattice [P] (see Figure 8.8). Press Enter to accept.

7. XPREP evaluates the data and looks at the systematic absences for all possible glide planes and screw axes (see Figure 8.9). These are displayed across the middle of the figure. By examining the number of reflections with I>3 sigma(I), the mean intensities, and the mean int/sigma, which should all be very small for a systematic absence, XPREP derives a suggested space group, P2(1)2(1)2(1). Press Enter to accept.
### 8.3 Reflection Statistics

1. XPREP returns to the general menu seen previously (see Figure 8.5). This time D is chosen to evaluate the data set. Press Enter to accept.

2. There are multiple choices for data manipulation (see Figure 8.10). “S” chooses a display of statistics.

3. The data can be merged in several ways. Choose the “Merge ALL equivalents including Friedel opposites” option [A] (see Figure 8.11). Press Enter to accept.

**NOTE:** This merge will not average the reflections in the final data file. It is only for the calculation of statistics.

4. A table of statistics appears (see Figure 8.12). Examine the data presented. Is the completion near 100%? Is the redundancy good? Are Rint and Rsigma small and increasing smoothly from top to bottom? In particular, look at the last two lines which compare all of the data with the high-resolution data. The completion should be near 100% for both the high-resolution shell and the complete data set. The redundancy and Rint should be similar for the two. The Mean Intensity and the Mean I/sigma(I) will usually be quite different. The Mean I/sigma(I) for the high-resolution data should be greater than 3.0.
For this data set, when integrated to 0.67 Å resolution, the difference between the Rint for the high-resolution data and the complete data set is slightly larger than expected. Press Enter to continue.

1. Since the difference between the Rint for the high-resolution data and the complete data set is slightly larger than expected, it is reasonable to apply a high-resolution cutoff using the H option in the data manipulation menu. (see Figure 8.13). Type H and press Enter.

2. Enter a high resolution limit of 0.75 (see Figure 8.14) and press Enter to accept the low resolution cutoff of infinity.

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8.4 Applying High Resolution Cutoff

**NOTE**: For the YLID crystal, if the resolution limit for integration was changed to 0.75 as suggested on Page 7-3, then there will be no need to apply a High Resolution Cutoff at this point.
3. Applying the cutoff removes approximately 3000 reflections from the data set (see Figure 8.15). The next step is to see if the cutoff has improved the statistics. Type S and press Enter.

4. As in step 3 of Section 8.3, enter [A] to merge all equivalents.

5. After the cutoff, all of the statistics look better (see Figure 8.16). Press Enter to continue.

6. This response will return to the data manipulation menu (see Figure 8.10, Figure 8.13 or Figure 8.15). The default answer should be E for Exit. Press Enter to accept this default and return to the general menu.
8.5 Preparing an Output File

1. In the general menu, chose C to define the unit cell contents.

2. A window will open displaying the current formula, \( Z \), the density, and the atomic volume (see Figure 8.17). In this example, the formula is incorrect and \( Z \) has been set to six to try to achieve a reasonable density and atomic volume. Since this formula is incorrect, it must be modified now. The correct chemical formula for the YLID crystal is \( \text{C}_{11}\text{H}_{10}\text{O}_{2}\text{S} \).

2.1 At Select Option, do not accept the default answer of E. Type in F to enter a new formula.

2.2 In response to the question “Enter Formula,” type the correct formula \( \text{C}_{11}\text{H}_{10}\text{O}_{2}\text{S} \) and press Enter.

2.3 Check that the information is correct. Check that \( Z \) seems reasonable for the space group, that the density is as expected (1.1 to 1.4 for organic molecules, higher for inorganic compounds), and that the atomic volume is around 17 or 18. Significant variation from the expected values may indicate an incorrect molecular formula or missing counter ions or solvates. The values at the bottom of the window look fine for the YLID. Press Enter to accept.

3. The next default action for XPREP is to write out the files necessary for the structure solution process (see Figure 8.18). Press Enter to accept.
4. The program asks for an output file name. Since this data has been cut off at a resolution of 0.75Å, it makes sense to change the output file name to ylid_res75 to reflect the cutoff (see Figure 8.19). Press Enter to accept.

![Figure 8.19 - Changing the file name](image)

5. After entering the file name (or hitting Enter to accept the default file name) an input file for the structure solution module is created and displayed on the screen, and the program asks, “Do you wish to (over)write the intensity data file ylid_res75.hkl?” Since the file name has been changed, this question must be answered with a y (see Figure 8.20).

![Figure 8.20 - The input file for structure solution and a final question](image)

6. Exit XPREP (see Figure 8.21). Press Enter to exit the program.

![Figure 8.21 - Exit XPREP from the general menu](image)

**NOTE:** There are many other features in XPREP that can be accessed from the general menu; reciprocal space plots, simulated powder patterns and a test for merohedral twinning are very useful tools.