Experiment 3: Simulating X-Ray Diffraction  
CH3500: Inorganic Chemistry, Plymouth State University  

References  
http://www.planewave.de/icp/atoms/atoms.sgml-3.html  

Introduction:  
Light can reflect off an atom or molecule, if the wavelength is on the order of the size of the particle. Atoms have radii in the range of ~70 – 200 pm (0.7 – 2 Å, or 0.7 – 2 × 10^{-10} m), which is in the range of X-rays. A number of X-ray diffraction techniques take advantage of this, and the fact that when two X-ray photons reflect off neighboring atoms, they will constructively or destructively interfere according to the angle of their reflection. Figure 1 shows two photons that have reflected at an angle such that the extra distance traveled by the bottom photon is equal to the wavelength of the light. Thus, the photons after reflection are in phase and will constructively interfere. The reflection angle (θ) needed for constructive interference is related to both the wavelength (λ) and the distance between the atoms by Bragg's Law (Equation 1):  

\[ n \lambda = 2d \sin \theta \]

'n' in the equation is any integer number, indicating that many discreet refraction angles are possible (though usually only the first one or two are considered). It follows then that an instrument capable of detecting the intensity of the reflected photons as a function of the reflection angle (see Figure 2) could provide data on the spacing between atoms. In order to perform such an experiment, the atoms in the material must be at a fixed distance from one another, so the critical angle does not change, and there must be many atoms at that distance, so the reflected beam is intense enough to be detected. In other words, the material must be a crystalline solid, wherein, by definition, the atoms are arranged in an orderly fashion in a regularly repeating pattern. The process by which light (or any wave) passes through a regularly repeating lattice, is reflected, and emerges on the other side in a pattern of constructive and destructive interference is known as “diffraction.” The process of using X-rays to determine structural information (i.e, the distances between atoms) about a material based on those diffraction patterns is known as “X-ray crystallography.”

A unit cell is the smallest arrangement of atoms that can be repeated in all three dimensions to describe the structure of a crystal. The simplest unit cell is a cube (a six-sided figure with equal sides and all 90° angles),
although a more general shape is the parallelepiped (a six-sided figure with sides 'a', 'b', and 'c' that may or may not be equal, and angles $\alpha$, $\beta$, and $\gamma$ that may or may not be equal, although opposite faces must be parallel). Generally, coordinates of atoms in a unit cell are given as fractions of the axes 'a', 'b', and 'c', and the actual distances of the atoms are stated in angstroms (Å). Thus, an atom at the very center of a unit cell would be at coordinates ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$) regardless of whether the length of the 'a' axis is 5 Å or 100 Å, or whether $\alpha = 90^\circ$ or 63°.

Although there are an infinite number of unit cells possible with different values of $a$, $b$, $c$, $\alpha$, $\beta$, and $\gamma$, all of these would fit into one of fourteen Bravais lattices (arrays of infinitely repeated points in three dimensions) (Figure 3). These lattices arise from the combination of seven possible crystal/lattice systems (cubic, orthorhombic, tetragonal, hexagonal, trigonal (rhombohedral), monoclinic, and triclinic) and five centering types (see Table 1).

![Figure 3: The 14 Bravais lattices](image)

Table 1: Lattice centering Types

<table>
<thead>
<tr>
<th>Type</th>
<th>Description</th>
<th>Symbol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Primitive</td>
<td>Lattice points on corners only.</td>
<td>P</td>
</tr>
</tbody>
</table>
| Face / Base Centered | Lattice points on corners and centered on 2 faces.          | A (face between b, c axes)  
                  |                                  | B (face between a, c axes)  
                  |                                  | C (face between a, b axes)  |
| All-Face Centered   | Lattice points on corners and centered in all faces.        | F        |
| Body-Centered       | Lattice points on corners and in the center of the unit     | I        |
| Rhombohedral        | Lattice points on corners and two interior points equally spaced along a diagonal | R        |

It is possible for the components of a unit cell (i.e., atoms and molecules) to have various symmetry elements (e.g., rotation, reflection). The combination of potential symmetries in the unit cell with the Bravais lattices results in 230 unique "space groups." A common system for designating three dimensional space groups is the Hermann-Maguin system, which uses two to four symbols to specify...
the properties of the space group. The first symbol is a single letter (P, I, R, F, A, B or C) referring to
the lattice type (Table 1), and the remaining symbols describe the rotational and/or mirror symmetries
of the space group. In addition, all the possible space groups have been given a unique number
between 1 and 230, inclusive. Table 2 lists a few common structure types, their Hermann-Maguin
designation, and their space group number. (The detailed method of defining a space group and its
symmetry elements is beyond the scope of this introduction, and Table 2 is meant to introduce a few
symbols only). The determination of a crystal's space group is one of the first steps in the process of
solving a structure in crystallography, because once the space group is known, all of the symmetry
elements of the crystal are also known. This, in turn, is necessary to interpret a diffraction pattern and
solve the structure of a crystal.

<table>
<thead>
<tr>
<th>Structure</th>
<th>Space Group (Hermann-Maguin notation)</th>
<th>Space Group Number</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>cubic (primitive)</td>
<td>P m 3 m</td>
<td>221</td>
<td>cesium chloride, perovskite</td>
</tr>
<tr>
<td>body-centered cubic</td>
<td>I m 3 m</td>
<td>229</td>
<td>Metals: Li, Na, V, Cr, Fe</td>
</tr>
<tr>
<td>face-centered cubic</td>
<td>F m 3 m</td>
<td>225</td>
<td>halite (NaCl)</td>
</tr>
<tr>
<td>zincblende</td>
<td>F -4 3 m</td>
<td>216</td>
<td>ZnS</td>
</tr>
<tr>
<td>hexagonal close pack</td>
<td>P 6/m m c</td>
<td>194</td>
<td>graphite</td>
</tr>
</tbody>
</table>

Because X-rays diffract off many atoms in a crystal, it is convenient to consider not just individual
atoms, but planes of atoms, chosen such that the distance between parallel planes is the same as that
between atoms (‘d’ from Bragg’s law, as discussed above). These planes are described by their
intersection points along the axes of the unit cell. For reasons beyond the scope of this introduction,
planes are labeled with Miller indices using ‘h k l’ coordinates, wherein ‘h’, ‘k’, and ‘l’ are the inverse
of the intersection points on ‘a’, ‘b’, and ‘c’ respectively (see Figure 4). Some tips for determining the
Miller indices:

1. Note the position of the primary axes (a, b, c). The intersection point is often the back, left,
   bottom corner of the unit cell. Determine the fraction of the axis where the plane intersects it,
   starting at this primary intersection.
2. If the plane appears to intersect a given axis, but on the opposite side of the unit cell, it is given
   a negative (-) value.
3. If the plane never (or always) intersects an axis (i.e., it runs parallel), it will have a '0' Miller
   index.
4. Miller indices must be integers, so if the process of inverting the axis intersection coordinates
   leaves fractional Miller indices, they are multiplied by the common
denominator (symmetry says there
will be another plane at every integer
multiple of a plane, so this works out).

Figure 4: Cleavage planes in a Unit Cell with their Miller indices
With a little trigonometry and incorporating the concepts of Miller indices just described, it can be shown that distances between planes of atoms in a crystal with any unit cell are given by Equation 2:

\[
\frac{1}{d^2} = \frac{1}{V^2} (S_{11} h^2 + S_{22} k^2 + S_{33} l^2 + 2S_{12} hl + 2S_{23} kl + 2S_{13} hl)
\]

where 'V' is the volume and the 'S' terms are given by Equations 3-8:

Eqn 3: \( S_{11} = b^2 c^2 \sin^2 \alpha \)
Eqn 4: \( S_{22} = a^2 c^2 \sin^2 \beta \)
Eqn 5: \( S_{33} = a^2 b^2 \sin^2 \gamma \)
Eqn 6: \( S_{12} = abc (\cos \alpha \cos \beta - \cos \gamma) \)
Eqn 7: \( S_{23} = a^2 bc (\cos \beta \cos \gamma - \cos \alpha) \)
Eqn 8: \( S_{13} = ab^2 c (\cos \gamma \cos \alpha - \cos \beta) \)

The 'd' in Equation 2 is the same 'd' as in Bragg's law (Equation 1). Thus, by consulting Figure 2, you will see we have just discussed the equations that describe the x-axis positions (angle of reflection, 2\( \theta \)) of the peaks in an X-ray diffraction pattern. However, we have not yet described the y-axis: the intensities of peaks (I), which are given by Equation 9:

\[
I_{hkl} = |F_{hkl}|^2
\]

where F is the "structure factor" for the hkl plane of atoms. The structure factor equation is given by Equation 10:

\[
F_{hkl} = \sum_{j=1}^{N} f_j e^{2\pi i (hx_j + ky_j + lz_j)}
\]

where 'N' is the total number of atoms in plane hkl, \( j \) refers to an individual atom at location xyz, and \( f \) is the atomic scattering factor (an empirically derived value proportional to the number of electrons in an atom). A detailed analysis of this equation is beyond the scope of this lab, but suffice it to say that the location and intensity of a specific peak in an X-ray diffraction pattern depends upon three things:

1. The distance between atoms.
2. The number of electrons in an atom (more electrons means more X-rays will scatter).
3. The number of equivalent atoms in a crystal.

Thus, if the structure of a crystalline material is known, its theoretical diffraction pattern can be calculated. Conversely, if a diffraction pattern is measured experimentally, it should be possible to determine the structure of the material. However, the process of solving a structure from the diffraction data is considerably more complicated. Consider, for example, the peaks in Figure 2. Knowing the x-coordinate (2\( \theta \)) for a peak can directly yield 'd' from Equation 1, but when that is related to Equation 2, there are still 9 unknown variables: \( a, b, c, \alpha, \beta, \gamma, h, k, \) and \( l \). Further, the y-axis yields intensity (I), but when that is related to Equation 9, there are 7 unknown variables: \( h, k, l, x, y, z, \) and \( f \). In other words, it is possible, but it requires a lot of data and computational power to relate many equations with many unknowns!

Although the process of determining a compound's structure from X-ray data can be complex and time consuming, it is much simplified in situations of simple ionic compounds (salts) with highly symmetric unit cells. In cubic systems, \( a = b = c \), and \( \alpha = \beta = \gamma \), bringing the number of unknowns in
Equation 2 down to 5. With just a bit of analysis, it thus becomes possible to determine the size of a unit cell by hand.

In the determination of cubic structures from diffraction data, there is just one more aspect that must be discussed: symmetry. Consider for a moment a body-centered cubic system, which is higher symmetry than a primitive cubic system by virtue of the fact that it has another symmetry-related atom in the unit cell. These body-centered atoms would create planes exactly half-way between the planes of the corner atoms, but the atoms are offset by half the cell axis. This means X-ray deflected by the body-centered atom plane would be exactly out of phase with those deflected by the corner atom planes, and the peak that would have appeared in the diffraction pattern of the simple cubic system is “systematically absent” in the body-centered system. Thus, by identifying the systematic absences in a diffraction pattern, the lattice type of the unit cell can be determined. Table 1 below shows the $hkl$ planes of the peaks that appear in the diffraction patterns of the three cubic systems.

<table>
<thead>
<tr>
<th>Simple cubic $(hkl)$</th>
<th>Body-Centered Cubic $(hkl)$</th>
<th>Face-Centered Cubic $(hkl)$</th>
<th>Sum of the squares $(h^2 + k^2 + l^2)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>110</td>
<td>110</td>
<td></td>
<td>2</td>
</tr>
<tr>
<td>111</td>
<td></td>
<td>111</td>
<td>3</td>
</tr>
<tr>
<td>200</td>
<td>200</td>
<td>200</td>
<td>4</td>
</tr>
<tr>
<td>210</td>
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<td>220</td>
<td>220</td>
<td>8</td>
</tr>
<tr>
<td>211,300</td>
<td></td>
<td></td>
<td>9</td>
</tr>
<tr>
<td>310</td>
<td>310</td>
<td></td>
<td>10</td>
</tr>
<tr>
<td>311</td>
<td></td>
<td>311</td>
<td>11</td>
</tr>
<tr>
<td>222</td>
<td>222</td>
<td>222</td>
<td>12</td>
</tr>
</tbody>
</table>

In this lab, you will develop a spreadsheet that predicts the positions of peaks (diffraction angle) for cubic, orthorhombic, and tetragonal lattice crystals. You will then use a computer program that can predict both diffraction angles and peak intensities for any Bravais lattice to check your spreadsheet and to further explore diffraction patterns. Finally, you will determine the Bravais lattices and dimensions of a unit cell from diffraction data.

**Safety Considerations:**
- This lab does not use any chemicals or other hazardous materials.

**Pre-Lab**

Note: You must perform this part of the procedure in your lab notebook before coming to lab.

1. Derive a simplified version of Equation 2 for systems when $\alpha = \beta = \gamma = 90^\circ$
2. If you did not already do so for your report, set up .cif files for the structures of CsCl, CaF$_2$, and ZnS from Learning Lab: Modeling the Solid State
   a) Look up the radii of the ions. These are available in your book, Resource Section 1: Selected Ionic Radii.
   b) Using the ionic radii and your data on the nearest neighboring atoms to calculate the dimensions of the unit cells. These should include the lengths of the unit cell axes and the total volume. Collect these results in a table.

3. Sketch the cleavage planes from Figure 5 in your notebook. Identify their Miller indices ($hkl$).

4. Make a list of all possible Miller indices with $hkl$ values 0 and/or 1. (Note: 0 0 1 is the first one; 0 0 0 does not exist).

5. To familiarize yourself with the Bragg's law, check out the "Bragg's Law Applet" at http://www.eserc.stonybrook.edu/projectjava/bragg/
   a) Click the "details" button to show a simulated detector.
   b) Using the arrows, adjust Theta to smaller angles. Notice what happens to the plots of Xray1 and Xray2 waves and the Detector output as you do this. Continue decreasing Theta to the smallest number possible. What value(s) of Theta maximize the Detector output?
   c) Change the values of Lambda and Distance to see how this affects the values of Theta where the detector signal is maximized.

Procedure

A. Develop your own diffraction pattern predictor

1. Boot up and login to a computer. You may use your own computer. If you have a spreadsheet program and have installed "Mercury", see the instructions on Moodle for obtaining Mercury.

2. From Moodle or the website, download the file "03-X-rayDiffractionSpreadsheet.xls" and open it. Make sure you are in the first tab: "Prediction".

3. A few lines down on the left you will see the columns 'h', 'k', and 'l'. In the cells under these, enter the $hkl$ values for all the planes using values from 0 to 3. Notes:
   a) There is no 0 0 0 plane.
   b) There are $4^3 - 1 = 63$ possibilities. Take a moment to think about how you can arrange these so you do not miss any, do not duplicate any, and do not take hours to fill them in!

4. Using your simplified version of Equation 2 (Part A2), enter the formula to find $1/d^2$ in the appropriate cell.

5. With reference to the cell you used above, enter a formula to find $d$.

6. With reference to the cell you used above, and assuming $n = 1$, use Bragg's law to enter a formula for "2theta." Notes:
   a) This should be 2 times the $\theta$ value in Bragg's law.
b) The built-in spreadsheet formula for arcsine is "asin()".

c) The "asin()" function outputs an angle in terms of radians. Convert it to degrees with the function "degrees()". Thus, a portion of your equation will be "degrees(asin(XXX)).

7. Copy and paste the formulas you just entered down the columns next to all your values of \( hkl \).

8. In the "Cell Parameters" area:
   a) Enter a formula to calculate volume in the cell next to "V"
   b) Enter the value for the wavelength of Cu K-\( \alpha \) X-rays in the cell next to "lambda"
   c) Enter the cell parameters you calculated (Part A3) for CsCl structure from Lab 03.

9. \text{SAVE YOUR SPREADSHEET!}

10. At this point, you should see a pattern appear in the spreadsheet's pre-existing figure. Make a table with headings as shown below in your notebook to record the peaks between \( 2\theta = 5^\circ \) and \( 60^\circ \). Some notes:
   a) Due to the high symmetry of the cubic system, many of the hkl planes result in the same \( 2\theta \) value. Rather than write down each plane resulting in the same diffraction angle, note the plane with the \textbf{smallest values of hkl}. \textbf{It may be helpful to copy the \( 2\theta \) and hkl values to a new area and use the “sort” function.}
   b) You may see that some of these hkl planes result in a \( 2\theta \) value "#VALUE!". These planes would result in a diffraction angle greater than \( 180^\circ \), which is nonsensical.

<table>
<thead>
<tr>
<th>Structure</th>
<th>My Diffraction Prediction</th>
<th>Mercury prediction</th>
</tr>
</thead>
<tbody>
<tr>
<td>hkl plane</td>
<td>( 2\theta ) angles (deg)</td>
<td>( 2\theta ) angles (deg)</td>
</tr>
</tbody>
</table>

\textbf{B. Check your pattern predictor}

1. Run the program “Mercury”
   a) When the program starts, a pop-up window may ask whether you have purchased a license to CCDC. Just say “no.” Don't worry--you'll still be able to use everything in the program you need.

2. Open the .cif file for your CsCl structure. DON'T PANIC! Your structure will probably not look right just yet. Make sure the following settings are accurate:
   a) Style (top left): either “Spacefill” or “Ball and Stick”
   b) Manage Styles (top center): “Work”
   c) Packing (bottom left): check the “Packing” box

3. Display the predicted powder pattern: “Calculate” \( \rightarrow \) “Powder pattern”

4. In the window that pops up, click “Customize”.
   a) Ensure that the wavelength is set to that of Cu-K\( \alpha \) (1.54 Å).
   b) Set the “Stop” angle to 60 (may be set to 50 by default)

5. Using the mouse to hover over the peaks, record the values of \( 2\theta \) and hkl for the peaks in the spectrum.
   a) If the Mercury values for \( 2\theta \) and the number of peaks do not closely resemble yours, go back and try to fix the problem. Are the formulas in your prediction correct? Are both predictors using the same \( \lambda \) value for the X-ray? Are the unit cell parameters the same? Are you looking just in the \( 2\theta \) range 5-60\(^\circ\)?
b) Once your two predictions are the same, record the values for \( hkl \) planes, \( 2\theta \), and intensity from the Mercury prediction. Do NOT record any planes with any \( hkl \) values that are negative or greater than 4. Observe similarities and differences between your predicted pattern and Mercury's

**C. Investigate effects of changing unit cells on diffraction patterns**

1. For this part, select either the fluorite or zinc blende structure for which you previously created a .cif file. If you were a complete slacker and did not create the proper .cif files before coming to lab, you may download .cif files for the minerals:
   a) In a web browser, navigate to [http://rruff.geo.arizona.edu/AMS/amcsd.php](http://rruff.geo.arizona.edu/AMS/amcsd.php)
   b) Search for the minerals "fluorite" and "sphalerite" (zinc blende) and download their CIF data files.

2. Back in your X-ray Diffraction Spreadsheet, make a copy of the “Prediction” tab. Rename the original tab “CsCl” and the new tab by your selected structure's name.

3. In the new, copied tab, enter the cell parameters for your selected structure (fluorite or zinc blende). In your table, record the diffraction angle and lowest \( hkl \) values of the peaks in the \( 2\theta \) range 5-60\(^\circ\). *It may be helpful to copy the \( 2\theta \) and \( hkl \) values to a new area and use the “sort” function.*

4. Back in Mercury, load the .cif file for your structure. Display the powder prediction and record the diffraction angle with \( hkl \) values of the peaks in the \( 2\theta \) range 5-60\(^\circ\).

5. Make note of the differences between your prediction and Mercury's, particularly peaks that are missing in one or the other. Fluorite and zinc blende are face centered cubic structures, which means there are more symmetry-related atoms in a unit cell than in the primitive cubic cell of CsCl. What is the effect of increasing the symmetry of a unit cell on its X-ray diffraction pattern?

6. Back in your X-ray Diffraction Spreadsheet, investigate the effects of changing the unit cell on diffraction patterns in the following way:
   a) In your spreadsheet, make two copies of the CsCl tab. Label them "Larger" and "Tetragonal"
   b) In the Larger tab, increase all three cell axes by 1 Å. Count the number of peaks in the \( 2\theta \) range 5-60\(^\circ\). Are there more or fewer peaks than in the regular CsCl pattern? What is the effect of increasing the size of a unit cell on its X-ray diffraction pattern?
   c) In the Tetragonal tab, increase the length of the ‘b’ axis by 1. Count the number of peaks in the \( 2\theta \) range 5-60\(^\circ\). Are there more or fewer peaks than in the regular CsCl pattern? What is the effect of decreasing the symmetry of a unit cell on its X-ray diffraction pattern?

7. SAVE YOUR SPREADSHEET!

**D. Determining the Unit Cell from Powder Data**

1. From Moodle, download and open the file “03-Unknown-PowderDiffractionData.pdf”

2. Back in your X-ray Diffraction Spreadsheet, go to the “DetermineStructure” tab. You will be given data for two structures. Follow the steps below for both structures.

3. Enter the \( 2\theta \) values from the data into the column A.
4. Calculate \( \theta \) in column B. (yes, it is as easy as dividing by 2).
5. Using the function “radians()”, calculate the angle \( \theta \) in radians in column C.
6. In column D, calculate \( \sin^2(\theta) \).
7. In order to determine the unit cell parameters from \( \theta \), we must use a form of Bragg's law simplified for cubic systems that relates the diffraction angle (\( \theta \)) to the length of the unit cell (a) and the sum of the squares of \( hkl \) (Equation 10):

\[
\text{Eqn 10: } \sin^2(\theta) = \left(\frac{\lambda^2}{4a^2}\right)(h^2+k^2+l^2)
\]

Note that the term \( \left(\frac{\lambda^2}{4a^2}\right) \) is a constant and \( (h^2+k^2+l^2) \) will always be an integer, so all the values of \( \sin^2(\theta) \) should be integer values of one another. Thus, in column E, divide all the \( \sin^2(\theta) \) values by the smallest \( \sin^2(\theta) \) value (from cell D2).
8. If the cell is primitive cubic, all values of \( hkl \) will show reflections, and the answers in column E will be integers (or close to it, within experimental error). However, face-centered and body-centered cubic systems have missing reflections (systematic absences), so the answers in column E will not be integers. Multiply by 2 or 3 to get all integer numbers in column G.
9. Based on the pattern of systematic absences, and with reference to Table 1, determine the Bravais lattice of the compound.
10. Using Equation 10, solve for the length of the unit cell, 'a'.

The Lab Report

- **Focus**: Analysis and Conclusion
- Your lab report must be typed in a modern word processor and submitted electronically to Moodle by the start of class next Wednesday. A hard copy of the report, which includes the duplicate “yellow” sheets from your lab, is due within 48 hours thereafter.
- You will NOT write a full, formal report for this lab. Instead, your report will contain the following:
  - **Purpose**: In a few sentences, describe why you did this lab. This should be well thought-out, complete, and succinct. Although this section appears first in your report, it is recommended that you write it LAST. Working through the results and analysis will give a better understanding of the main things you were expected to learn.
  - **Results**: Report the numerical results from lab, formatted into tables to the extent possible (if you saved your spreadsheets, this should largely involve cutting and pasting). This section should include:
    - Tables that meet the ACS Style Guide rules for “formal tables” (see relevant section of ACS Style Guide in “Misc Documents” section on Moodle). A few quick rules—Tables should:
      - Have a title in the first row that begins with “Title” followed by its number and then a brief, non-sentence, descriptive title
      - Have headings at the top of every column that include units where appropriate
      - Not break across pages, or if it is larger than one page, repeats the title and heading rows
- A table containing the $2\theta$ angles in the range 5-60° and respective $hkl$ planes (do not include duplicate planes; note that by convention, for example, if 2 0 0 and 0 0 2 planes are equivalent, the 2 0 0 is reported; that is, the “lowest” $hkl$ plane has the lower value of $l$ then $k$ then $h$) from both your prediction spreadsheet and Mercury's prediction, for CsCl, the second structure of your choice (fluorite or zinc blende), the “Larger” cell, and the “Tetragonal” cell (part B and C). Format these tables to allow easy comparison of your predictions with Mercury's. In these tables, also include the total number of unique reflections in the range 5-60° for all your structures using both prediction methods.

- A table showing all the data and calculated values for determining the cell dimensions of the unknowns (part D)

- Narrative text that refers to all the tables by number and briefly describes them or what data will be found in them. This text is more appropriately written as one single paragraph in the beginning of the section, rather than individual sentences spaced between tables.

  - **Analysis and Conclusions:**
    - For the prediction part of the lab, discuss / answer the questions “What is the effect on the X-ray powder diffraction pattern when the symmetry of a unit cell is increased or decreased? When the size of the unit cell is increased or decreased?” In answering these questions, you should refer to the results from all of the structures (CsCl, fluorite / zinc blende, “Larger”, and “Tetragonal”). This should be a detailed discussion considering both theoretical and experimental arguments. For the “experimental” portion, cite specific, numerical data from the “Results” section and refer to data in tables by the table's number in the “Results” section (e.g., “See Table 1”). For the “theoretical” part, discuss relevant equations (e.g., “according to Equation 1, increasing 'x' will result in smaller 'y' value”). These equations should be numbered, labeled, and written somewhere in this section.

    - For the structure determination portion, answer the questions “What is the lattice setting of the Unknown? What are the cell dimensions of the Unknown” for both unknowns. Discuss briefly the process you went through to determine these values—this should NOT be a detailed step-by-step “x was divided by y”, but rather a general explanation of the process focusing more on why specific steps were taken. Look at your values for the unit cell dimensions and consider for a moment whether they make sense (for example, would $a = -3.25\text{Å}$ or $a = 0.000345 \text{Å}$ make sense?).

- Grade Scheme: Your grade for this lab will be based on both your duplicate notebook pages AND your written report, according to the following scheme:

<table>
<thead>
<tr>
<th>Grade Category</th>
<th>Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organized Lab Notebook with Observations and Data</td>
<td>2</td>
</tr>
<tr>
<td>Purpose</td>
<td>4</td>
</tr>
<tr>
<td>Results</td>
<td>5</td>
</tr>
<tr>
<td>Analysis and Conclusions</td>
<td>10</td>
</tr>
<tr>
<td>Overall (Grammar, style, formatting)</td>
<td>4</td>
</tr>
</tbody>
</table>