### University of Arizona

# Materials Science and Engineering

## MSE 110: Solid State Chemistry

##### Interpretation of an X-ray diffraction pattern

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#### Interpretation of an X-ray diffraction pattern

**Introduction**

The purpose of the experiment was to determine the X-ray pattern of an unknown compound using a given intensity plot by identifying the d-spacing for each peak and using comparison with the given JCPDS cards to identify the unknown compound. We then calculate the Miller indices of the diffraction plane for each identified peak and use that to calculate the unit cell parameter.

We use our understanding of d-spacing and Miller indices to identify unknown compounds as we understand that each crystalline structure is associated with a different metallic compound. By examining intensity data, we are able to determine from the graph and by performing certain calculation, the Miller indices of an unknown compound and eventually the unit cell parameter. By understanding this process, we are able to properly identify the crystalline structures and unit cell parameters of any kind of compound so long we are able to properly examine and differ from a graph and by comparison with known data.

###### Experimental Procedure

Materials used:

* Set of XRD patterns obtained from unknown substances.
* Set of JCPDS cards listing the d-spacing and corresponding peak intensities of known crystalline compounds.
* Table listing the cell parameters of known crystalline compounds.

Procedure:

1. Using the given XRD pattern provided, examine the 2θ values by identifying the peak on the pattern and then calculate the θ by dividing the original value by 2.
2. To find the d-spacing, using Bragg’s law and rearranging the equation to solve for d. Use the given wavelength to help with the calculation, therefore making d the only unknown.
3. Compare the d-spacing values with the JCPDS cards provided to identify the compound that matches the d-spacing calculated.
4. Using the cell parameter equation, identify the Miller indices by rearranging the cell parameter equation so that h^2, k^2, and l^2 are equal to (a/d) ^2.
5. Then from there, using the found h, k, and l values as well as the calculated d-spacing values, calculate the actual a value for each respective h, k, l, and d-spacing.
6. Calculate the average experimental a value and compare to the given a value.

###### Experimental Results

Table: Data for compound Potassium Iodine (KI) λ = 1.5405 Å, Cell parameter for KI, a = 7.066 Å.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 2θ (degrees) | θ (degrees) | d (Å) | h | k | l | a (Å) |
| 22 | 11 | 4.037 | 1 | 1 | 1 | 6.992 |
| 25.5 | 12.75 | 3.49 | 2 | 0 | 0 | 6.98 |
| 36 | 18 | 2.493 | 2 | 2 | 0 | 7.051 |
| 42.5 | 21.25 | 2.125 | 3 | 1 | 1 | 7.048 |
| 44.5 | 22.25 | 2.034 | 2 | 2 | 2 | 7.046 |
| 52 | 26 | 1.757 | 4 | 0 | 0 | 7.028 |
| 57 | 28.5 | 1.614 | 3 | 3 | 1 | 7.035 |
| 58.5 | 29.25 | 1.576 | 4 | 2 | 0 | 7.048 |
| 65 | 32.5 | 1.434 | 4 | 2 | 2 | 7.025 |
| 69 | 34.5 | 1.36 | 5 | 1 | 1 | 7.067 |

To find the d-spacing, rearrange equation (1) into (2):

Given our known d-spacing equation and rearranging for (5), we can then find the respective h, k, and l values.

Our average calculated a = 7.032 Å

Given a = 7.066 Å

###### Discussion and Conclusions

For our identified compound, based on the d-spacing values calculated, we were given the X-ray graph of Potassium iodine (KI). The given value of a = 7.066 Å and our calculated average experimental a = 7.032 Å were identified and compared, allowing us to examine the percent error and giving way to identifying possible errors in the calculations, methods of analyzing the data, etc.

Comparing our actual calculated with the given a value, we get an error of 0.48%. From this value, we can determine that our data was not too far off from the given value, therefore, making our data fairly accurate. Possible discrepancies could arise from analyzing the given graph, not making 100% accurate identifications of the 2theta values. As seen from the table, all the values were either whole numbers or rounded to something.5. This will cause some of the data to not be exactly at the desired value as seen through comparison of the given d-spacing values and our calculated values. However, from our calculated percent error, we had some pretty close values and there wasn’t too much of a variation comparing both data sets.

To reduce possible errors, perhaps having more accurate lines on the graph will allow for proper lining up with a ruler to identify the values as accurate as possible, preferably to 2 decimal places. This will inevitably reduce the amount of errors as our calculations will be a lot more accurate compared to 1 decimal place, rounded up to 0.5 or 0.0.

Works Cited:

MSE 110 Lab Manual