

X-Ray Diffraction

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Experimental

In this X-ray diffraction (XRD) experiment, we first prepared two samples: NaCl and Fe powders. The samples were mounted on glass plates, ensuring a uniform distribution of the powders on the plates. The glass plates were used because they lack a crystalline structure, which prevents any interference with the diffractogram. Next, we placed each sample inside the Discover D8 Bruker diffractometer for measurement. We initiated the XRD scans for each sample using the provided scan parameters. For the NaCl sample, we used a start angle of 26 degrees, a stop angle of 86 degrees, an increment of 0.03 degrees, a scan time of 1 second per step, and a locked couple scan type. For the Fe sample, we used a start angle of 43 degrees, a stop angle of 85 degrees, an increment of 0.03 degrees, a scan time of 1 second per step, and a locked couple scan type. Once the scans were complete, we saved the raw data and converted it into ascii text files using the File Exchange software. We then plotted the XRD data, determined the positions of the peaks in 2θ , and analyzed the data to determine the Miller indices (h, k, l), interplanar spacing (d), and lattice parameters (a) for each sample.

Results

NaCl

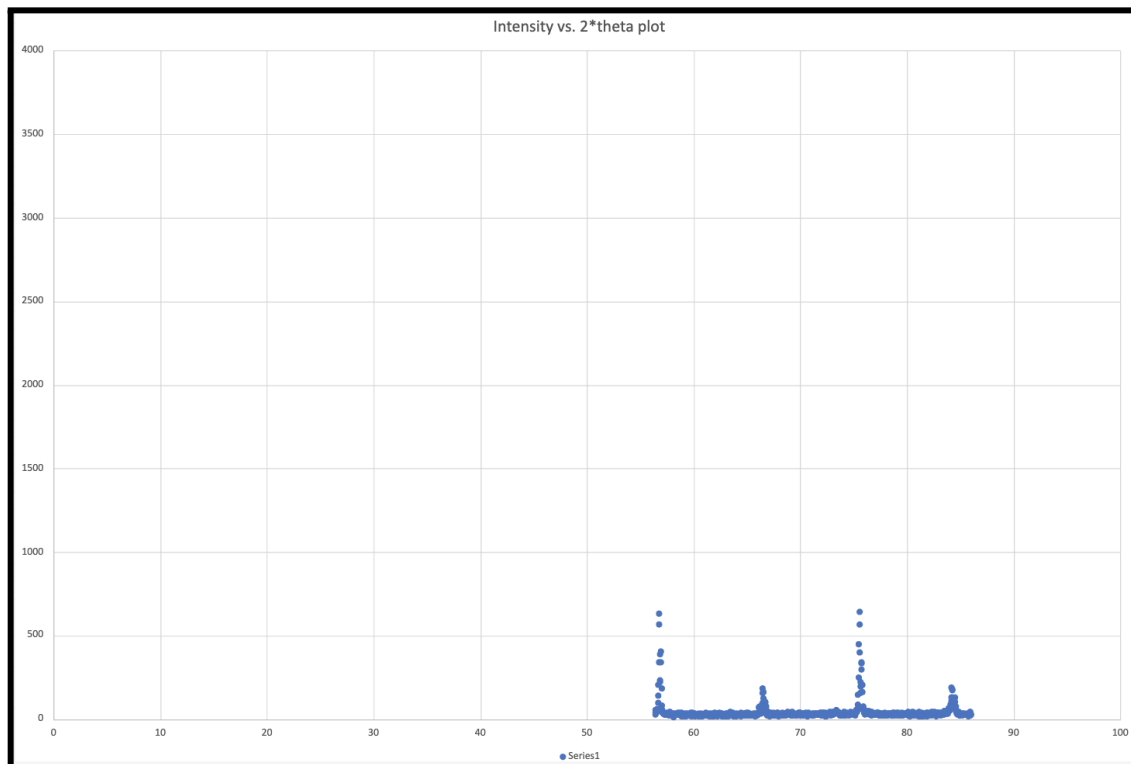


Figure 1: Intensity vs. 2θ plot of data measured from the NaCl sample

2theta	theta	(hkl)	d	a
31.9601	15.98005	200	2.796922019	3.955445053
45.7657	22.88285	220	1.980207187	3.960414374
56.804	28.402	222	1.618820655	3.96528459
66.5044	33.2522	400	1.404275789	2.808551579
75.5358	37.7679	420	1.257216191	3.079538165
84.2023	42.10115	422	1.148496904	3.248439796

Table 1: Indexing of NaCl XRD spectrum

Q1. What is the mean lattice constant 'a' value (and error) that you calculate for NaCl? How does this compare to the known value?

Iron (Fe)

Based on the Table 1 shown above, we can calculate the mean lattice constant 'a' value and its error for the NaCl sample.

First, let's find the mean lattice constant 'a':

A Mean = $(3.955445053 + 3.960414374 + 3.96528459 + 2.808551579 + 3.079538165 + 3.248439796) / 6 = \mathbf{3.502945593}$

Next, let's calculate the standard deviation (SD):

Find the differences between each 'a' value and the mean 'a' value:

1. A difference = $[0.45249946, 0.457468781, 0.462338997, -0.694393014, -0.423407428, -0.254505797]$

Square each difference and find the mean of these squared differences:

Difference squared = $[0.20475234, 0.209230285, 0.213707228, 0.481981758, 0.179278769, 0.064772501]$

2. Variance = $\text{sum}(\text{Difference Squared}) / 6 = 0.223953814$

Take the square root of the variance to get the SD:

3. Standard deviation = $\text{sqrt}(0.223953814) = 0.473021149$

Now, let's calculate the standard error (SE):

SE = SD / $\text{sqrt}(6) = 0.473021149 / \text{sqrt}(6) = \mathbf{0.193107003}$

So, the mean lattice constant 'a' value for the NaCl sample is **3.502945593 Å**, and the error is **±0.193107003 Å**. The known value for the NaCl lattice constant 'a' is approximately **5.640 Å**. The calculated value is significantly lower than the known value, which suggests that there may be some discrepancies in the data or the experimental setup. It is important to investigate potential sources of error in the experiment and verify the correctness of the data and calculations.

Fe

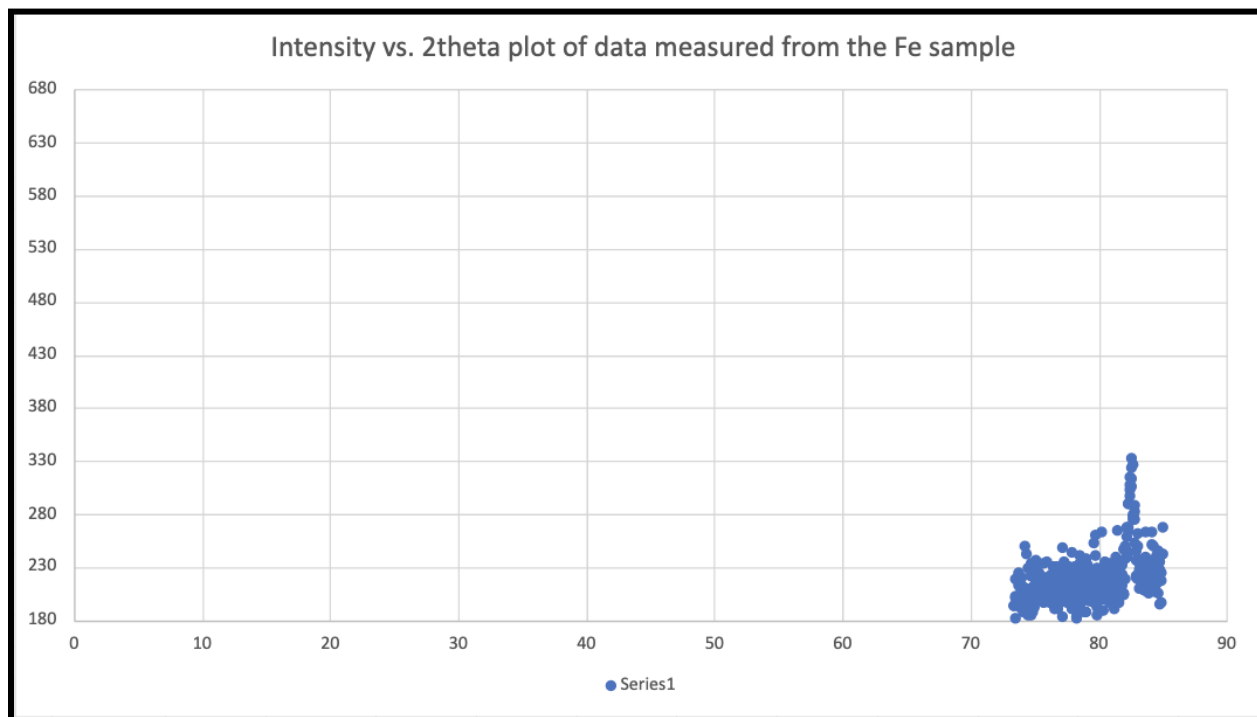


Figure 2. Intensity vs. 2*theta plot of data measured from the Fe sample

2theta	theta	(hkl)	d	a
44.8549	22.42745	110	2.018278389	2.85427667
65.1375	32.56875	200	1.430399319	2.022890116
82.5921	41.29605	211	1.16675528	2.33351056

Table 2. Indexing of Fe XRD spectrum

Q2. What is the mean lattice constant 'a' value (and error) that you calculate for Fe? How does this compare to the known value?

Based on the provided data, we can calculate the mean lattice constant 'a' value and its error for the Fe sample.

First, let's find the mean lattice constant 'a':

A Mean= $(2.85427667 + 2.022890116 + 2.33351056) / 3 = 2.403559115 \text{ \AA}$

Next, let's calculate the standard deviation (SD):

Find the differences between each 'a' value and the mean 'a' value:

1. A Difference = $[0.450717555, -0.380668999, -0.070048835]$

Square each difference and find the mean of these squared differences:

A Difference Squared= $[0.203146336, 0.144908719, 0.004906846]$

2. $\text{variance} = \text{sum}(\text{A Difference Squared}) / 3 = 0.117653967$

Take the square root of the variance to get the SD:

3. $\text{SD} = \sqrt{0.117653967} = 0.343002896$

Now, let's calculate the standard error (SE):

$\text{SE} = \text{SD} / \sqrt{3} = 0.343002896 / \sqrt{3} = \mathbf{0.197959879}$

So, the mean lattice constant 'a' value for the Fe sample is **2.403559115 Å**, and the error is **±0.197959879 Å**. The known value for the Fe lattice constant 'a' is approximately 2.866 Å for body-centered cubic (BCC) structure. The calculated value is slightly lower than the known value, which suggests that there may be some discrepancies in the data or the experimental setup. It is important to investigate potential sources of error in the experiment and verify the correctness of the data and calculations.

Q3. Molybdenum (Mo) metal and Fe metal both have the BCC structure. Assuming all else being equal (and both are measured using the same diffractometer), how do you expect the intensity of the XRD pattern signal to compare for Mo vs that for Fe and why?

The intensity of the XRD pattern signal depends on several factors, including the atomic scattering factor, the type of X-ray source used, the atomic structure, and the concentration of the sample. When comparing Mo and Fe, which both have a BCC structure, the intensities of their XRD patterns will be influenced by their atomic scattering factors, as other factors like the diffractometer and the structure are the same in this case. The atomic scattering factor is affected by the atomic number (Z) and the electron distribution of the element. Molybdenum has an atomic number of 42, while iron has an atomic number of 26. The higher atomic number of Mo means it has more electrons surrounding its nucleus, which leads to stronger scattering of X-rays and, consequently, higher intensity peaks in the XRD pattern. Therefore, assuming all else being equal, and both Mo and Fe samples are measured using the same diffractometer, we would expect the intensity of the XRD pattern signal for Mo to be higher than that for Fe, primarily due to the difference in their atomic scattering factors resulting from their differing atomic numbers.