X-ray Diffraction

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This study aimed to utilize an x-ray apparatus to determine the crystal structures and lattice constants of sodium chloride and lithium fluoride [1]. The sodium chloride lattice constants of the K_β peaks for n=1 and n=2 and the K_α peaks for n=1 and n=2 were (596.3±10.8) pm, (574.3±4.5) pm, (595.8±9.7) pm, and (574.0±3.9) pm, respectively. The correlating errors compared to the actual value of 564.0 pm, in order, were 3.0σ, 2.3σ, 3.3σ, and 1.0σ. The lithium fluoride lattice constants of the K_β peaks for n=1 and n=2 and the K_α peak for n=1 were (407.0±4.9) pm, (404.4±1.9) pm, (411.6±4.4) pm, respectively. The correlating errors compared to the actual value of 403.0 pm, in order, were 0.8σ, 0.7σ, and 1.9σ.

Background and Significance

X-ray diffraction occurs when x-rays deflect off of structures within a crystal. The deflection maxima carry information about the arrangement of the crystalline structure in question. At 22 years old, William Bragg used x-rays to determine spacing of atoms within solid crystals. The discovery led to the practice of x-ray crystallography, and in 1953 the method was used to reveal the structure of DNA [2]. X-ray diffraction allows for researchers to study the micro-structures of crystals with precision.

Apparatus

The study utilized an x-ray emitting apparatus seen in figure 2. The x-ray source was supplied with a high voltage source (HV 1), outputting 20 kV. The electronics in figure 1 included a second high voltage source (HV 2), outputting 0.384 kV. HV 2 was supplied to the Geiger-Muller (GM) counter. Output of the GM counter is amplified by a preamplifier and fed to a scaler. A terminated oscilloscope was also connected to the scaler to determine potential noise pulses. Apparatus components, including the target sample, between the x-ray apparatus and GM counter in figure 1 are illustrated in figure 2.

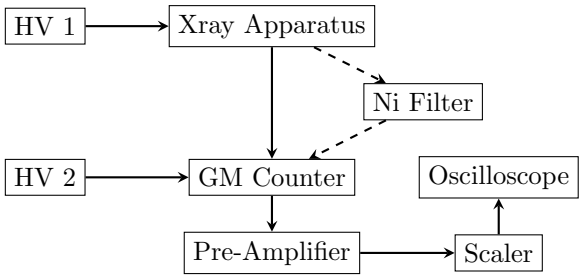

Figure 1. A schematic diagram for the electronics used in this experiment. Dashed lines represent the inclusion of a nickel filter, which was placed between the x-ray source and GM counter for certain procedures.

The x-ray apparatus consisted of a copper target x-ray tube, power supply, and a photon detecting Geiger-Muller tube [1]. Two crystalline samples of sodium chloride (NaCl) and lithium fluoride (LiF) were used for the experiment, which were individually holstered between the x-ray source and detector. While the x-ray tube remained stationary, the detector arm was manipulated by set angles, mainly of a single degree. A sample is rotated half as much as the detector arm. A Nickel (Ni) filter was used to absorb Copper (Cu) K_β radiation, which allows for determination of wavelengths needed for analysis [1].

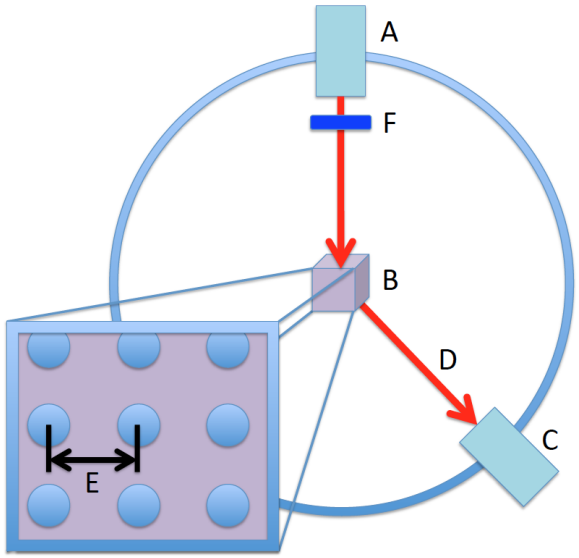

Figure 2. A conceptual illustration of the x-ray apparatus and the individual components, also including a portion of the lattice structure. A) Copper target x-ray tube [x-ray source] B) Crystalline sample (NaCl/LiF) C) Photon detecting Geiger-Muller tube [detector] D) Source beam E) Lattice constant/spacing F) Nickel filter

Procedure and Relevant Equations

Before measurements were conducted on the crystalline samples, backgrounds for each sample were evaluated. Considerable background was generated from scattered x-rays, so the added counts were subtracted from the crystal sample measurements. Estimates of maximum count angles were predetermined, and only certain angle ranges were swept for the background analysis. Isolating the angle in equation 1, the predicted values for the NaCl K_β peaks of n=1 and n=2 were approximately 14.0°and 29.0°. The NaCl K_α peaks of n=1 and n=2 were about 16.0°and 33.0°. Predicted values of the LiF K_β peaks of n=1 and n=2 were 20.0°and 44.0°. The LiF K_α peaks of n=1 and n=2 were about 22.0°and 49.0°.

For NaCl, background angles measured for n=1 were 12.5°–17.0°, and the n=2 angles were 28.0°–34.5°. The angles for the LiF background for n=1 and n=2 were 17.0°–23.5°and 41.5°–44.5°, respectively. Counts were recorded for 45 seconds for the NaCl angles, and 180 seconds for the LiF angles. The LiF sample time interval was significantly longer due to excessive noise background when applying the Ni filter. The exact preceding procedure was replicated while placing the crystal sample of NaCl and LiF in the center of the apparatus for the correlating time intervals. The Ni filter was inserted into the system past the x-ray source to distinguish the K_α and K_β lines for analysis.

$$a_0 = \frac{n\lambda}{\sin(\theta)} \tag{1}$$

Equation 1 was derived from the Bragg condition, but the lattice constant of the sample, a_0, was isolated for computation. The x-ray wavelength, λ, corresponds to the angle between the emitter and detector, θ, and integer values, n.

Calculation of Results and Errors

Fit Justification

Peaks generally consisted of three points, therefore instead of fitting a Gaussian relation, lines were fit between every point, and more importantly, the highest points were considered the peaks.

NaCl Sample

Figure 3 yielded the n=1 maximum count peaks at 13.5°±0.3° for K_β, and 15.0°±0.3° for K_α. The NaCl n=2 maximum count peaks in figure 4 for K_β was 29.0°±0.3°, and 32.5°±0.3° for the K_α peak.

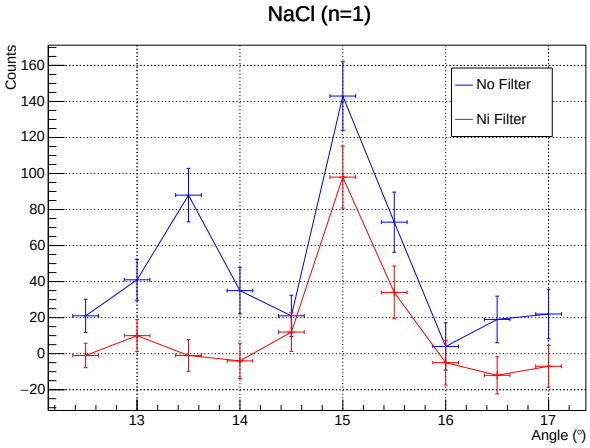

Figure 3. NaCl maxima peaks for K_β and K_α correlating to n=1. The K_β peak (left peak) is identified due to the extinguished peak when a Ni filter is introduced.

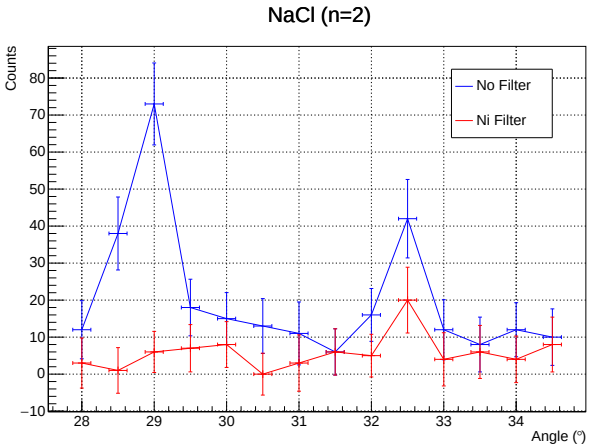

Figure 4. NaCl maxima peaks for K_β and K_α correlating to n=2. The K_β peak (left peak) is identified due to the extinguished peak when a Ni filter is introduced.

LiF Sample

Figure 5 yielded the n=1 maximum count peaks at 20.0°±0.3° for K_β, and 22.0°±0.3° for K_α. The NaCl n=2 maximum count peaks in figure 6 for K_β was 43.5°±0.3°. The K_α peak, for n=2, was predetermined to be measured at approximately 50.0°, but due to the apparatus angular limitations, this angle wasn't able to be achieved.

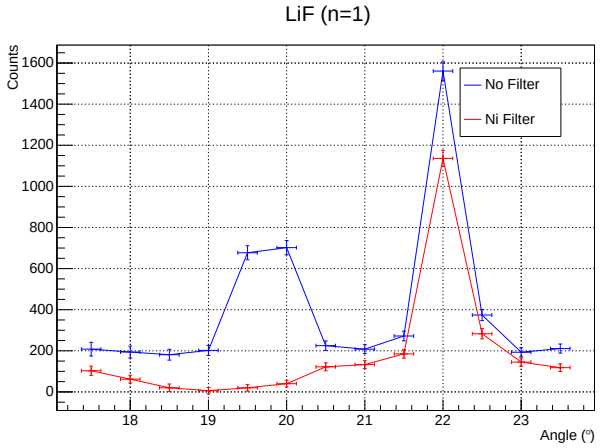

Figure 5. LiF maxima peaks for K_β and K_α correlating to n=1. The K_β peak (left peak) is identified due to the extinguished peak when a Ni filter is introduced.

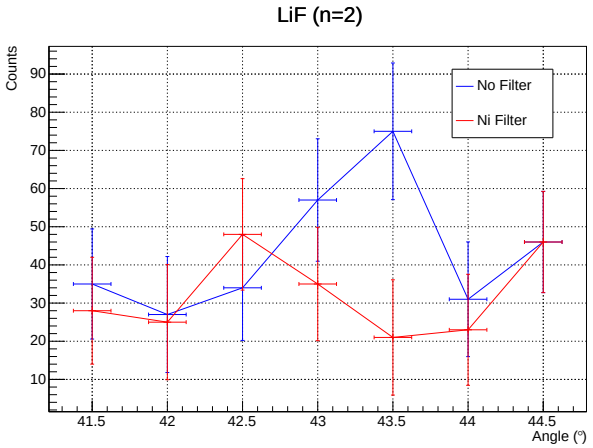

Figure 6. LiF maxima peaks for K_β and K_α correlating to n=2. Only the K_β peak is present in this plot, which is seen at 43.5°.

Lattice Constant Results

Table I. Experimental lattice constant and literature values of crystal samples of K_β peaks

Crystal Sample	Experimental Lattice Constant (pm)	Actual Lattice Constant (pm)	Error
NaCl (n=1)	596.3±10.8	564.0	3.0σ
NaCl (n=2)	574.3±4.5	564.0	2.3σ
LiF (n=1)	407.0±4.9	403.0	0.8σ
LiF (n=2)	404.4±1.9	403.0	0.8σ

Table II. Experimental lattice constant and literature values of crystal samples of K_α peaks

Crystal Sample	Experimental Lattice Constant (pm)	Actual Lattice Constant (pm)	Error
NaCl (n=1)	595.8±9.7	564.0	3.3σ
NaCl (n=2)	574.0±3.9	564.0	1.0σ
LiF (n=1)	411.6±4.4	403.0	1.9σ
LiF (n=2)	-	-	-

Discussion and Conclusion

This study led to experimental lattice constants, which were generated from the peak maxima values, resulting in K_β values in table I and K_α values in table II. The NaCl K_β for n=1 and n=2 and K_α peaks for n=1 and n=2 were (596.3±10.8) pm, (574.3±4.5) pm, (595.8±9.7) pm, and (574.0±3.9) pm, respectively. The correlating errors compared to the actual value of 564.0 pm, in order, were 3.0σ, 2.3σ, 3.3σ, and 1.0σ.

The LiF K_β for n=1 and n=2 and the K_α peaks for n=1 were (407.0±4.9) pm, (404.4±1.9) pm, (411.6±4.4) pm, respectively. The correlating errors compared to the actual value of 403.0 pm, in order, were 0.8σ, 0.8σ, and 1.9σ.

Low sigma results showed that the experimental LiF measurements were quite accurate in determining the lattice constant. Conversely, the NaCl sigma errors ranged from 2.3σ to 3.3σ, which showed more deviation.

The n=2 peak for the LiF crystal is not included since the apparatus cannot reach the predetermined angle estimation of 49.0°. An amplifier was originally between the pre-amplifier and scaler, in figure 1, but was removed. Low HV voltages, below 0.400 kV, generated excessive pulses and produced too much noise when including the amplifier. Figure 6 exhibited a small peak-like structure, but the counts are consistent to a "flat" line, which meant that the no-filter peak was in fact the K_β peak. This assumption leads to the conclusion that the statistical errors result mainly from the lack of angle measurements and the Poisson error of the counts.

[1] http://www.phys.hawaii.edu/~shige/phys481L/XRay.txt
[2] http://www.outreach.phy.cam.ac.uk/camphy/xraydiffraction/xraydiffractionindex.htm