X-Ray Diffraction Analysis of Cubic LiF Crystals *and* Investigation of Geiger Counter Background Noise

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X-rays are a higher energy form of radiation than visible light, allowing them the ability to partially penetrate through a material. Rather than being reflected off the surface of an object, x-rays will actually reflect off lower layers of atomic sheets. Because the path length between the atomic layers can differ by an integer number of wavelengths, a diffraction pattern is observed in the reflected x-rays. By directing x-rays at a crystal lattice and using a revolving Geiger counter to determine the angles of constructive interference, the spacing between atomic layers of the crystal can be determined. Unfortunately, the x-ray source was not properly functioning during this experiment, but the data collected using two separate Geiger counters during this time remains useful. This data will be presented to provide a reference for the background noise of Geiger counters used in the lab room. Data from an identically performed experiment by Cottingham will be analysed and used to solve for the spacing between atomic layers of a LiF crystal [3]. This data gives the distance between atomic layers to be $d = 0.1995\,\text{nm}$, which has a difference of $0.94\,\%$ from the reference value.

Introduction

X-rays are a relatively high energy form of electromagnetic radiation with wavelengths shorter than visible and ultraviolet light, ranging from 0.01 nm to 10 nm. A common way to produce x-rays for research purposes is by accelerating electrons with a high voltage potential and colliding them into a metal target. These "bullet" electrons knock electrons from the inner shell of the metal. As an electron falls from a higher orbital to fill the vacancy, an x-ray is emitted to conserve energy. This results in sharply defined characteristic x-rays. A second form of x-ray is called *brehmsstrahlung*, or "breaking radiation" [8]. These x-rays are emitted as a result of the deceleration of the incident electrons when they are fired at the metal. Any accelerating charged particle gives off electromagnetic energy, and assuming the energy of the particle is high enough, the emitted radiation can be in the x-ray region of the electromagnetic spectrum.

In the x-ray source and measuring device used to perform the lab, a Tel-X-Ometer, electrons are accelerated in a vacuum tube from a 30 kV potential between a heated cathode and a copper anode. It is easy to see that as the electrons accelerate, they gain 30 keV of energy. This is more than enough energy to knock inner shell electrons out of the atom. As previously explained, the x-ray is produced as an electron from an upper orbital fills the gap in the lower shell. If it is an electron from the next shell that fills the vacancy, a k-α x-ray photon is emitted. If an electron from two shells out happens to be closer than an electron in the nearest shell, it may fill the vacancy instead, resulting in k-β x-ray emission [2].

Unlike visible light which reflects off the surface of objects, x-rays are of short enough wavelength to pass in between individual atoms or molecules. If the x-ray collides with an atom in a subsurface sheet, it is then reflected outwards. A diffraction pattern is formed when the path length between surface and subsurface reflections vary by an integer number of wavelengths, as in Figure 1. This causes the reflected light to either constructively or destructively inter-

*The author would like to thank [redacted] for assistance in performing the experiments.

fere with itself. The goal of this experiment was to measure the spacing between atomic layers in a cubic LiF crystal using the Bragg diffraction equation and data from Cottingham [3].

The detector used in this experiment was a Geiger

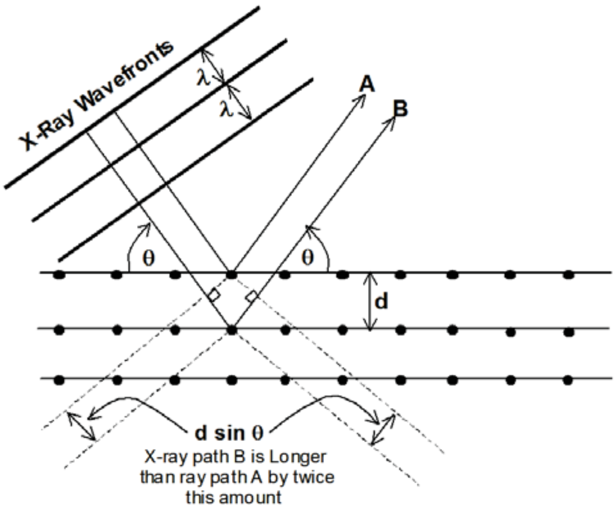

Figure 1: Some x-rays are reflected off the surface of an object, while others reflect off of atoms beneath the surface. The difference in path length $d \sin\theta$ can cause a diffraction pattern in the reflection [2].

counter, the active part of which is known as a Geiger-Müller tube. This tube is filled with argon gas and has a positive several-hundred-volt wire suspended in the middle. When a particle enters the tube, it ejects an electron from the argon which then moves to the positive wire. As the electron rushes towards the wire it knocks many other electrons free from the argon causing an "avalanche" [4]. The electrons arriving at the wire create an electric pulse which can be amplified and counted by the other components of the Geiger counter.

Procedure

A Tel-X-Ometer x-ray source and Bragg diffractometer was used to perform the experiment. After noting that the alignment of the LiF crystal was offset from the angular measure scale by about 2.5°, the protective lid was closed and the crystal was set to 12.5° according to the rotating arm angular measure scale. The Tel-X-Ometer had previously been operated for more than 30 minutes to allow the vacuum tube to warm up and dry out the device. The Geiger counter used was set to 460 V and an integration time of 10 seconds was used. The tube current was set to about 58 µA and the counts were recorded over two 10 s periods for each 0.5° increment. Angles between 12.5 and 28.5° were swept out before the counter was replaced with a second Geiger counter set to 450 V. This time a 20 s integration time was used for the remainder of measurements, up through 60° at 0.5° increments.

Determination of spacing between atomic layers of LiF crystal

Using the data from Cottingham, we were able to calculate the spacing between uniform atomic layers of a LiF crystal using the Bragg diffraction equation. The Bragg diffraction equation is

$$m\lambda = 2d\sin\theta \qquad m = 1, 2, 3, \ldots \tag{1}$$

where m is the diffraction order, λ is the wavelength of the x-rays, θ is the angle of diffraction measured from the surface (unlike measuring from normal with the crystal surface, as is common in most visible optics problems), and d is the distance between the atomic sheets.

Bragg diffraction theory matches the observed results and can be demonstrated geometrically as in Figure 1. The x-ray wavefronts approach the surface of the crystal at some angle θ and of course each peak is a wavelength λ apart. Some part of the wavefront will reflect off of an atom on the surface of the crystal, but another part of the x-ray wavefront will pass between atoms on the surface and reflect off of an atom in the next sheet down. The path length for x-rays that bounce off the subsurface atomic sheet travel an extra $d\sin\theta$ distance. When that distance is equal to an integer wavelength ($m\lambda$), constructive interference of the reflected x-rays results. These are seen in the data as large spikes in the number of detected x-rays.

It is obvious looking at Figure 2 that the counts are much higher for the α than β peaks of the same order m. This is because the reactions that cause the k-α emissions are much more likely, and therefore more common than the reactions which cause the k-β emissions. More of these x-rays are detected than the k-β rays. In addition, the number of detected x-rays at order $m = 2$ are less than the number of detections at $m = 1$. This decrease in intensity with distance from $m = 0$ is the same Fraunhofer diffraction effect observed in single slit diffraction patterns with visible light. Using Equation 1, each diffraction spike observed in the Cottingham data was used to solve for d. The results are summarized in Table 1. Assuming the data is purely a function of the random decay events and independent of the measurement, the uncertainty of a single measurement is approximately

$$\sigma_S = \sqrt{S}. \tag{2}$$

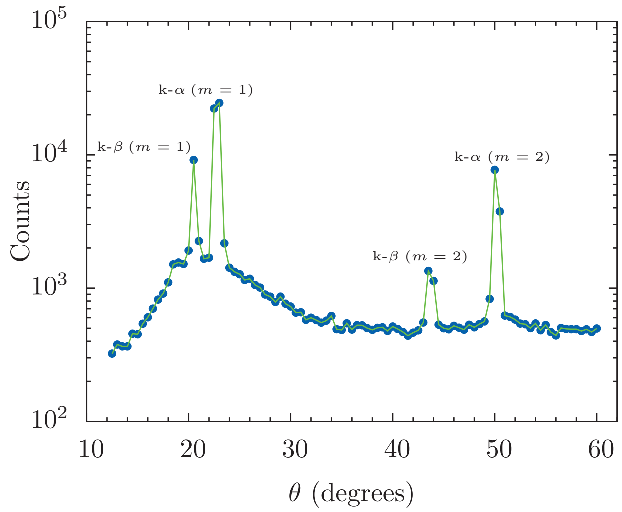

Figure 2: The angles of diffraction for k-α and k-β are obvious for both $m = 1$ and $m = 2$. Using these angles, the distance d between the atomic sheets of LiF crystal can be calculated [3].

θ (°)	d (nm)	λ (nm)	m	Counts	$\pm\sigma_S$
20.5	0.1970	0.138	1	9164	95.7
23.0	0.1971	0.154	1	24 530	156.6
43.5	0.1801	0.138	2	1346	36.7
50.0	0.2237	0.154	2	7718	87.9

Table 1: A summary of the x-ray diffraction spikes used to determine d.

Therefore, the relative uncertainty of a measurement is given by

$$\sigma_{S\mathrm{rel}} = \pm\frac{1}{\sqrt{S}} \tag{3}$$

where S is the number of counts over a single integration time [7]. Real measurements might have meaningful detector error, and, more importantly, the brehmsstrahlung radiation and other external sources of radiation are ignored in Equation 2 and Equation 3. Broadband radiation has a component of the counts in the x-ray peaks. It should also be remembered that the counts do not directly represent a number of detected photons. The Geiger counter exhibits some gain, and information on the original number of photons is lost in the avalanche stage. In addition, some photons may not be detected during the dead time in the Geiger-Müller tube until the ions are neutralized [7].

Comparing the calculated values for d to the reference value, $d = 0.2014$ nm, there is very little difference (see Table 2) [2]. The average measurement for the distance between atomic sheets in LiF is 0.1995 nm, which has a percent difference of only 0.94 %. That said, this average is not a good indicator of the experimental results because it is biased upwards by d_{meas} at 50°. It is unlikely a diffraction spike between 12.5 and 60° was missed because the 0.5° sampling was good enough to observe at least part of the spike. However, it is not guaranteed that the true peak is at one of the angles which were measured. This appears to be particularly true in the case of $m = 2$ where the error is much higher in our calculated d (see Table 2). From Equation 1, the uncertainty Δd can be calculated by standard methods as

θ (°)	d_{meas} (nm)	% difference	m	Counts
20.5	0.1970	2.2	1	9164
23.0	0.1971	2.1	1	24 530
43.5	0.1801	10.6	2	1346
50.0	0.2237	11.1	2	7718

Table 2: The percent difference between the measured and reference value for $d = 0.2014$ nm is given for each observed diffraction peak. The error is probably higher at $m = 2$ because the true peak may not have been at one of the measured angles.

$$\frac{\Delta d}{d} \approx \frac{\cos\theta}{\sin^2\theta}\Delta\theta, \tag{4}$$

which is about 5 % for $\Delta\theta = 0.5°$ when $\theta = 23°$. This is within the percent difference for $m = 1$ in Table 2. This drops to about 1 % at 46°. This is far from the 11 % difference which was observed at $m = 2$. An 11 % difference would mean an $11/1 = 11°$ error in the measurement, which is unlikely given the strength of the signal as shown in Figure 2.

Looking at Figure 2, there are multiple observed elevated points on the spike "curves". This may mean that the true peak is in-between or nearby those points at an angle other than the 0.5° increments we were measuring at. More measurements taken at smaller increments through those spike ranges would need to be taken to approach the true angle of maximum reflection. Individual Gaussian curves could also be placed over each observed peak to infer the angle of each true peak. Lastly, the offset between the crystal angle and measurement scale angle might not be constant for the entire range of angles tested. The values of d_{meas} for both emission types at $m = 1$ in Table 2 are very consistent with each other which suggests simply offsetting the angle θ for both might give the reference value exactly.

Analysis of Geiger Counter Background Noise

The Tel-X-Ometer x-ray source originally used for this lab had several issues during data collection. Throughout the experiment both the power and x-ray emission indicator lights were on, although some x-rays were observed by the Geiger counter regardless of its angle. In case it was a Geiger counter problem, the counter was switched out for a different model at angle $\theta = 58°$ and the experiment was finished with this second counter despite the fact no noticeable difference in counts was observed. During the lab, a 1-σ detection test was devised to declare what we considered to be a significant number of observed counts – and perhaps a diffraction spike. At the time we thought that the low number of counts might be due to a badly collimated source or the fact the tube current was slightly less than the recommended 60 µA. Looking back, a 1-σ detection is rather foolish, as by definition we would expect 16% of our observed data to fall above this level. At the end of the experiment, several steps were taken to check the functioning of the Tel-X-Ometer. First, a piece of lead was placed between the source and the counter. This resulted in no change of counts. Second, with the Tel-X-Ometer powered on, the angles which satisfied the 1-σ test were re-examined and they did not produce detections a second time. Third, measurements were taken with all Tel-X-Ometers in the room powered off and the observed counts remained the same. In addition, a third Geiger counter was tested with still no noticeable increase in the number of observed counts. At one point during testing the power indicator lamp failed to light but the vacuum tube filament continued to glow. The fuses were checked and it was found the extra high tension (EHT) fuse was blown. The fuse was later replaced only to blow almost immediately again. According to the Tel-X-Ometer manual, a fault in which the "POWER ON" lamp fails to operate, but the tube filament is illuminated indicates either an issue with the indicator lamp or EHT circuitry. We had confirmed that the EHT fuse was blown and because it also blew upon replacement, the manual suggests checking the EHT stabiliser and also looking for shorts [10]. This leads the author to conclude with very high certainty that all of the measurements taken with the Geiger counters represent the noise internal to the counters and the background radiation of the room. There are no known x-ray sources in the area and weak sources should not penetrate through the scatter shield lid of the Tel-X-Ometer, so most observed radiation is probably in the form of high energy particles caused by cosmic rays entering Earth's atmosphere.

Although it is unclear when exactly the failure took place (both indicator lights were on during data collection), looking at the data suggests the Tel-X-Ometer was not emitting x-rays for any of the experiment. Looking at the observed number of counts in the peaks from the Cottingham data, they are clearly above the background radiation and noise levels. See Figure 3 for a plot of the original raw data with the faulty Tel-X-Ometer. It is clear there are no obvious spikes. A histogram of this data shows it is approximately normally distributed (see Figure 4). There are two histograms because two Geiger counters were used to collect data.

Assuming all observed counts are from random noise, the background source count rate and the uncertainty is

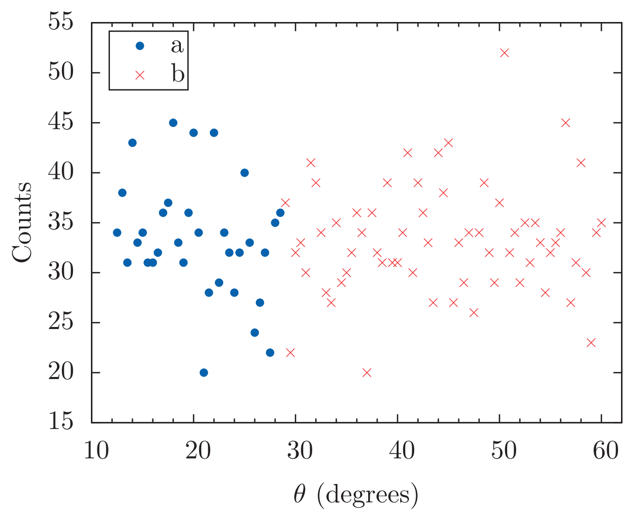

Figure 3: A plot of the original raw data with the failed x-ray source. No diffraction spikes are observed and the data appears to be random.

$$r_B = \frac{S_B}{t} \tag{5}$$

$$\sigma_{r_B} = \frac{\sqrt{S_B}}{t} \tag{6}$$

where r_B is the rate of backgrounds counts (counts/sec), S_B is the number of counts, and t is the integration time in seconds [9]. For Geiger counters **a** and **b** (from Figure 4), we have

$$r_{B,a} = 1.665\,\text{counts/s} \quad \sigma r_{B,a} = 0.408\,\text{counts/s}$$
$$r_{B,b} = 1.664\,\text{counts/s} \quad \sigma r_{B,b} = 0.288\,\text{counts/s}.$$

Despite the fact these results come from two entirely different Geiger counters, they are fairly con-

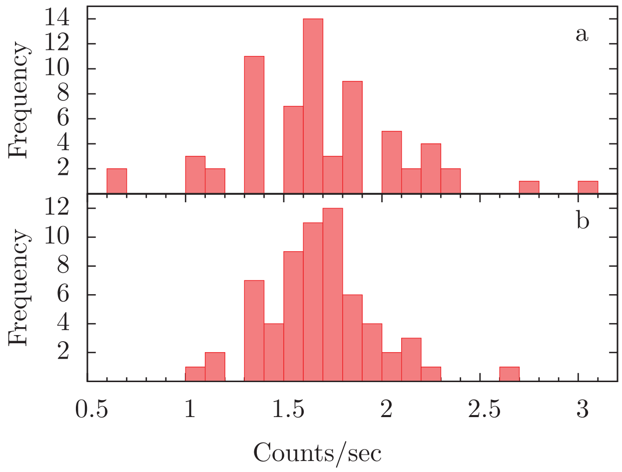

Figure 4: Histograms of the raw data from the two Geiger counters used to collect data during the lab. The x-ray source had failed so this represents the noise from the counters and background noise in the rooms. A Gaussian curve is over-plotted on both to show they are approximately normally distributed.

sistent with each other. This leads the author to believe that these results can be expanded to any of the Geiger counters used in the lab room, LB 372, under normal conditions. Cosmic rays come from a variety of galactic and extra-galactic sources, but the shower of secondary particles which are detected by the Geiger counter distributes the point source over an area between hundreds of square meters and several square kilometers [1]. Despite the fact that a diurnal variation in cosmic ray flux has been measured, it is on the magnitude of one-fifth of a percent of the peak flux [6]. In addition, cosmic ray flux is known to vary with pressure, at a rate of about 1% every 3 mmHg, and also with temperature [5]. Therefore, the author feels it is worthwhile to note that the atmospheric pressure during the data collection was 760 mmHg and the temperature at data collection altitude, sea level, was 77 °F [12]. Using these known background radiation values, a more accurate determination of the counts observed from the x-ray source can be obtained. Even if future data is collected during a weather extreme, the number of background counts is expected to shift no more than 20 %. This is small enough so that when applying a background radiation correction to the total number of detected counts, the difference will be sufficiently small compared to the total number of detected counts to safely ignore meteorological effects. The true source rate and error in that rate is given by

$$r_S = r_{tot} - r_B \tag{7}$$

$$\delta r_S = \pm\sqrt{(\sigma_{r_{tot}})^2 + (\sigma_{r_B})^2} \tag{8}$$

where r_{tot} is the total rate of observed counts and r_B is the rate of background counts, which is estimated to be about 1.665 counts/s. The uncertainty in the source rate is similar to how the propagation of error is calculated. Taking the data from Cottingham as an example, for the weakest diffraction spike (at k-β and $m = 2$) we have

$$\begin{aligned} r_S &= \frac{1346 \text{ counts}}{20\,\text{s}} - 1.664 \text{ counts/s} \\ &= 67.3 \text{ counts/s} - 1.664 \text{ counts/s} \\ &= 65.636 \text{ counts/s} \end{aligned}$$

and

$$\begin{aligned} \delta r_S &= \sqrt{\left(\frac{\sqrt{1346 \text{ counts}}}{20 \text{ s}}\right)^2 + (0.288 \text{ counts/s})^2} \\ \delta r_S &= \sqrt{(1.834 \text{ counts/s})^2 + (0.288 \text{ counts/s})^2} \\ &= 1.856 \text{ counts/s}. \end{aligned}$$

This can be transformed from the rate to number of counts domain by multiplying by the integration time. This background-corrected result for the counts in the lowest signal to noise ratio diffraction spike gives approximately 1313 ± 37 counts. This confirms it is a real diffraction spike. I would like to also add that in the case of $S_{\text{tot}} >> S_B$, the signal to noise ratio is approximated by $\sqrt{S_{\text{tot}}}$. For this spikes, that gives $S/N = 36.7$.

Conclusions

This lab used x-ray diffraction to determine the spacing between atomic sheets of a LiF crystal. Although the x-ray source was not properly functioning, the Geiger counter data could be used to characterize the background noise in the lab room. Using data obtained by Cottingham, the spacing between atomic sheets of LiF crystal was determined by the Bragg diffraction equation to be $d = 0.1995$ nm, which has a percent difference of only 0.94 % from the reference value. In addition, the background noise of the Geiger counters used in the laboratory was estimated to be about 1.664 counts/s.

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