

X-Ray Diffraction

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I. INTRODUCTION

Prior to 1922, the concept of a photon was scarcely accepted, despite being introduced by Einstein in 1905. It was the scattering of X-rays from electrons that empirically proved the photon's existence, giving evidence of one of the most fundamental phenomena in physics, and perhaps piquing the interest of current academia in regards to X-ray crystallography.

X-ray diffraction can be seen empirically, and relatively noiselessly in various crystals. This process can thereby be used to ascertain various properties of the propagating material. In particular, due to their relatively simple structure, one can determine the separation between layers of atoms in face-centered cubic crystals through aforementioned diffraction. The ångström scale by which crystal layers are separated implies the need of high-frequency/short-wavelength radiation; particularly, the use of X-rays.

By comparison, one can take another method for determining this separation. Take NaCl (Fig. 1), the mass of a NaCl molecule is the molecular mass M *kg/mol* divided by the molecules per mole $N = 6.02 \times 10^{23}$. The molecules per unit volume are equivalent to the crystal density ρ (*kg/m³*) divided by the mass M/N *kg*. NaCl is diatomic, however, so there are two atoms per molecule. Reflecting from the crystal structure in Fig. 1, this implies one atom per cube; the side-length of which, will be defined by:

$$d^3 = \frac{1}{2\rho(N/M)} \Rightarrow d = \sqrt[3]{\frac{1}{2\rho(N/M)}} \quad (1)$$

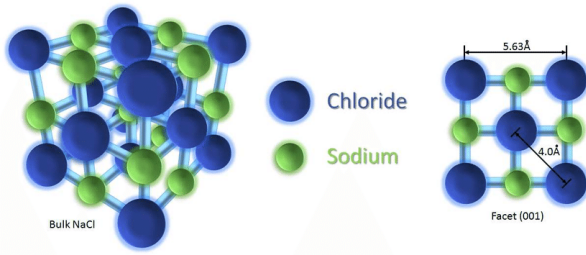


Figure 1: Crystal structure of NaCl¹. Structure is FCC (face-centered cubic), and two atoms correspond to a molecule.

With parameters $x = (M, \rho) = (58.46 \times 10^{-3}, 2.16 \times 10^3)$, the separation is easily calculated:

$$d(x) = \sqrt[3]{\frac{1}{2\rho(N/M)}} = 2.822 \times 10^{-10} \text{ m}$$

X-rays are generally emitted due to the excitation of electrons in atoms. A manufacturable way to achieve this, is by collisions of high-energy electrons with the atom to be excited (generally, a metal, and most notably copper, since copper gives the shortest wavelength above 1 Å). Atoms tend to minimal energies, leading to emission of energy in the way of electromagnetic radiation, to conserve energy when electrons from higher states transition to lower states. This phenomenon is illustrated in Fig. 2.

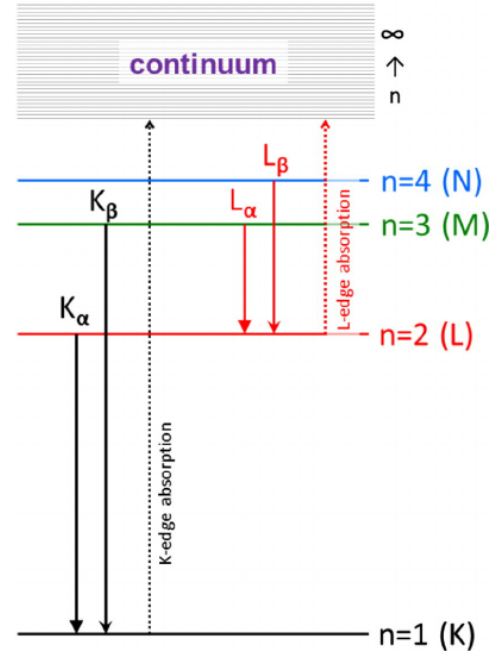


Figure 2: Electron transitions corresponding to X-rays (K lines)². Electrons upon excitation transition to higher shells, and release an X-ray to minimize atom energy, thereby returning to vacated lower levels.

Most collisions do not result in the emission of X-rays. However, continually colliding electrons with the target increases the target's temperature, and lets the target absorb longer wavelengths in the form of EM radiation. This continuum of wavelengths is termed "bremsstrahlung". The shortest of these wavelengths can be determined simply:

$$V\lambda_{min} = C \quad (2)$$

Where C is an arbitrary constant, and V is the tube (accelerating) voltage of the X-ray machine. Photon energy can be characterized simply by the photo-electric effect: $\omega = h\nu$, where ω is the photon energy, h is planck's constant, and ν is the photon frequency. When all of the

kinetic energy of an electron is converted to a single photon, the maximum frequency (or shortest wavelength) is achieved:

$$Ve = \frac{hc}{\lambda_{min}} = h\nu_{max} \quad (3)$$

In this report, two different uses of X-rays for ascertaining physical constants will be employed. X-ray counts, and calculations of corresponding wavelength values will be compared to calculated separation values for two crystals (NaCl, LiF). Additionally, observation of bremsstrahlung phenomena will be used in comparison to known equations to derive planck's constant.

II. METHODS

Figure 3 depicts the primary components of the apparatus used in our experiment. First, the X-ray source emits X-Rays towards the crystal sample, which will diffract at an angle θ . Simultaneously, the motor will rotate the detector twice as fast as it rotates the crystal in order to achieve the first Bragg reflection condition:

$$2\theta_{crystal} = \theta_{detector} \quad (4)$$

where $\theta_{crystal}$ is the x-ray's angle of incidence upon the crystal.

The x-rays are then diffracted towards the detector, which sends a signal to the software and records the signal as the number of photons detected as a function of the angle rotated by the detector.

As mentioned before, we will be using the data recorded by the detector as well as the calculated value of the separation d of the two crystals in order to find the values of λ and n . Moreover, we will verify our observations of the bremsstrahlung phenomena by using it to calculate Planck's constant h .

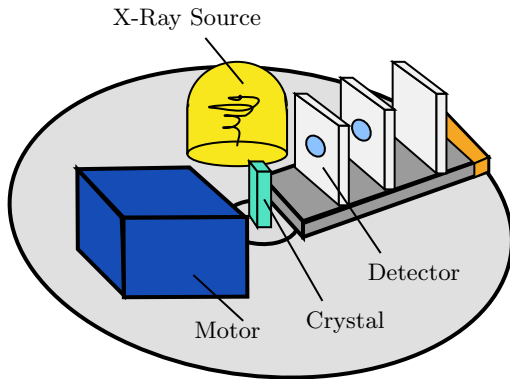


Figure 3: Primary Components of Apparatus

III. RESULTS AND ANALYSIS

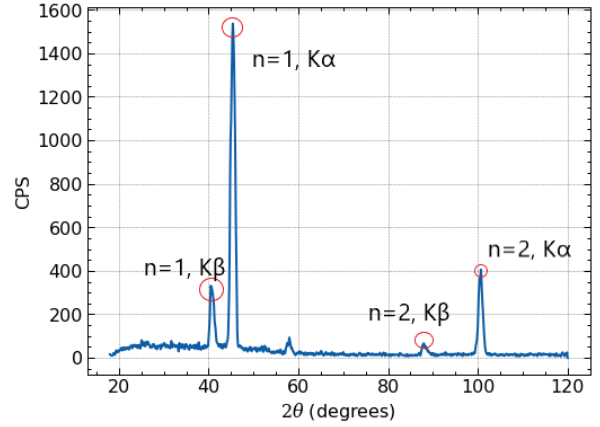


Figure 4: LiF Spectrum 20 kV

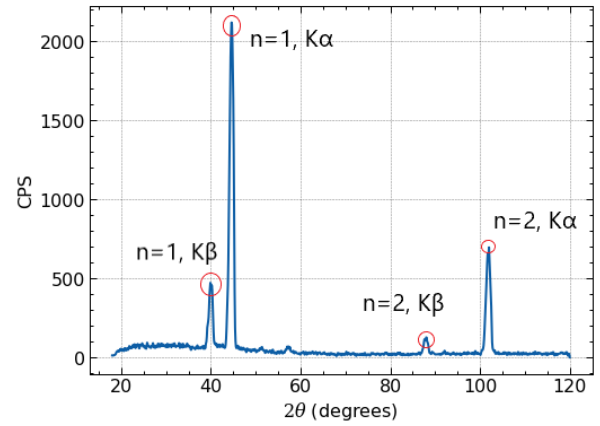


Figure 5: LiF Spectrum 30 kV

Voltage (kV)	θ (degrees)	n	λ (Å)
20	20.25	1	1.394
20	22.70	1	1.555
20	43.95	2	1.398
20	50.35	2	1.551
30	19.95	1	1.374
30	22.30	1	1.529
30	44.00	2	1.399
30	51.00	2	1.565

Table I: LiF Data

By noting that $M_{LiF} = 25.94 \text{ g/mol}$ and $\rho_{LiF} = 2635 \text{ kg/m}^3$, we can use equation 1 to evaluate the atomic lattice spacing for LiF: $d_{LiF} = 2.014 \times 10^{-10} \text{ m}$.

Figures 4 and 5 depict acquired emission lines for accelerating voltages of 20 kV and 30 kV respectively. The LiF sample yielded little noise for our measurements so the peaks were easy to discern. Overall, the θ values were mostly the same, where the difference between the two accelerating voltages was that 30 kV yielded higher peaks than for 20 kV.

In order to evaluate λ , the Bragg condition was used which is given by the following equation:

$$n\lambda = 2d \sin(\theta) \quad (5)$$

Values of n were determined based on using equation 5 to yield the closest value for λ to $\lambda = 1.38 \text{ pm}$ for the shorter peak (K_β) and to $\lambda = 1.54 \text{ pm}$ for the taller peak (K_α). All pertinent values for figures 4 and 5 are tabulated in table I.

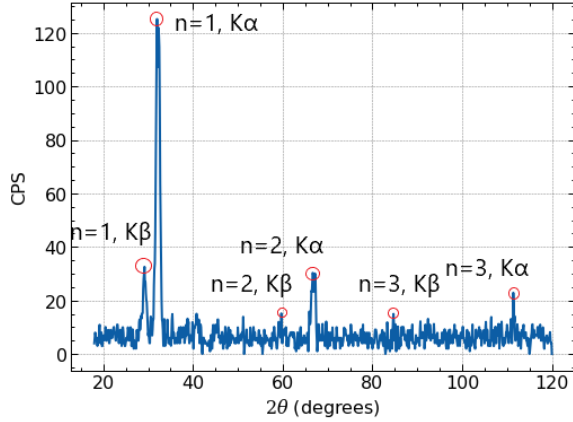


Figure 6: NaCl Spectrum 20 kV

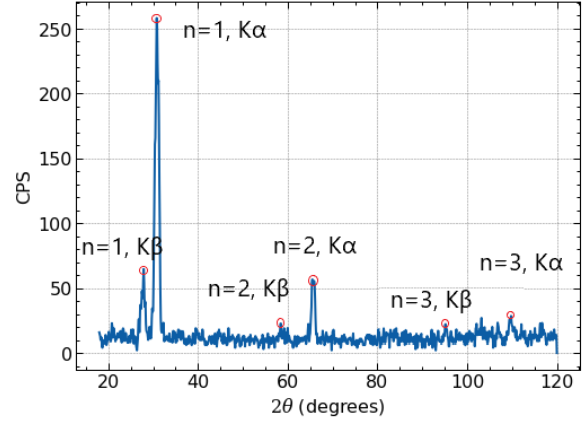


Figure 7: NaCl Spectrum 30 kV

Voltage (kV)	θ (degrees)	n	λ (Å)
20	14.60	1	1.423
20	16.00	1	1.556
20	29.85	2	1.404
20	33.35	2	1.551
20	42.35	3	1.267
20	55.70	3	1.554
30	13.95	1	1.360
30	15.40	1	1.499
30	30.00	2	1.411
30	32.75	2	1.526
30	47.60	3	1.389
30	54.90	3	1.539

Table II: NaCl Data

By noting that $M_{NaCl} = 58.46 \text{ g/mol}$ and $\rho_{NaCl} = 2160 \text{ kg/m}^3$, we can use equation 1 to evaluate the atomic lattice spacing for NaCl: $d_{NaCl} = 2.822 \times 10^{-10} \text{ m}$.

Unlike the LiF sample, the NaCl sample yielded three orders of diffraction; the spectrums for NaCl also tended to yield a lot more noise so discerning the peaks was pretty difficult. In this case, discerning the peaks was done by partitioning the 2θ values based on exploratory data analysis and finding the max value on those intervals. This is done for both LiF and NaCl and the corresponding maximums are indicated by a red circle. Values for λ and n were determined in the same way as for LiF using equation 5. All pertinent values for figures 6 and 7 are tabulated in table II

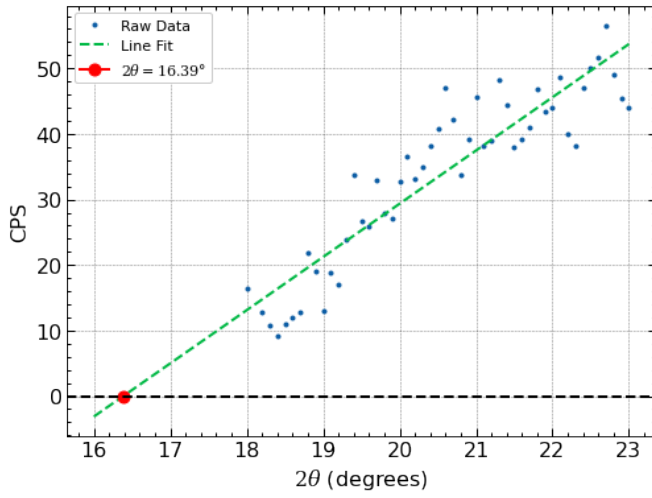


Figure 8: LiF Linear Bremsstrahlung Region 20 kV

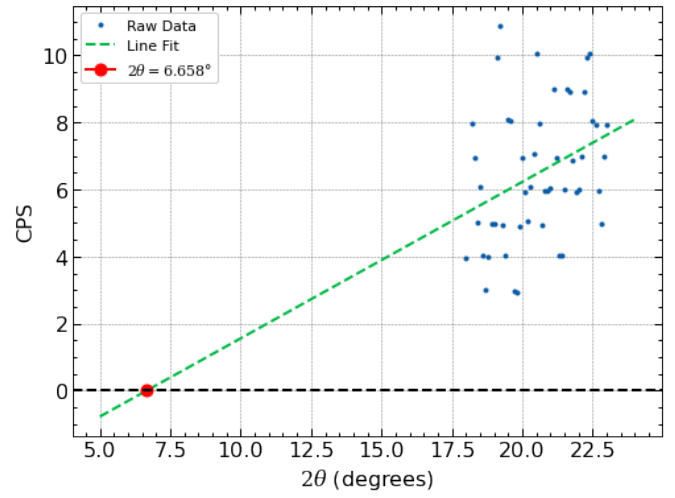


Figure 10: NaCl Linear Bremsstrahlung Region 20 kV

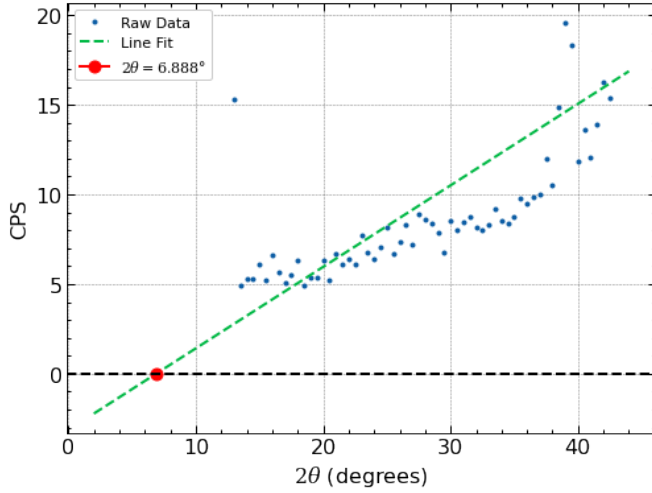


Figure 9: LiF Linear Bremsstrahlung Region 30 kV

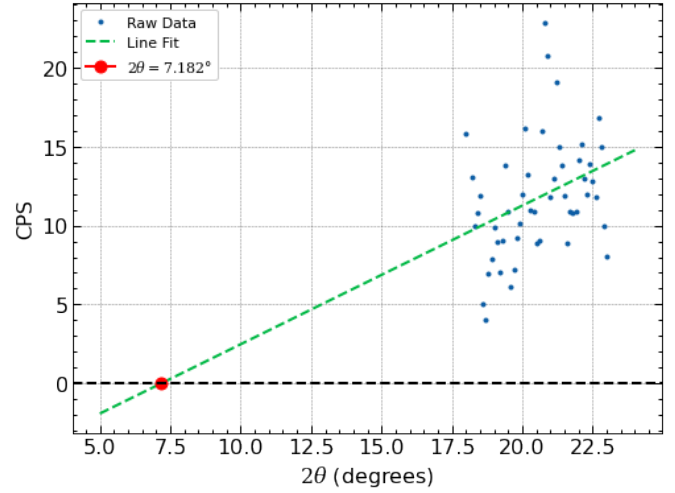


Figure 11: NaCl Linear Bremsstrahlung Region 30 kV

Figures 8, 9, 10, and 11 indicate plots of the linear portion of the Bremsstrahlung region for NaCl and LiF at accelerating voltages of 20 kV and 30 kV. Depicted in the plots is an extrapolation of these linear approximations of the raw data points up to a predicted root ($2\theta_{min}$ indicated by the red dot). Using equations 3 and 5, we can use this extrapolated root to find Planck's constant and the minimum wavelength (λ_{min}). Acquired values are tabulated in table III. Additionally, our acquired values for Planck's constant h_{exp} are compared against the official value³ of Planck's constant in the final column where $h = 6.626 \times 10^{-34} J \cdot s$. For the most part, our acquired values were off by almost a factor of two. Nevertheless, all of our calculated values for Planck's constant were on the expected order of magnitude.

Sample	V (kV)	θ_{min}	λ_{min} (pm)	$h_{exp} (J \cdot s)$	h/h_{exp}
LiF	20	8.193°	5.741	6.136×10^{-34}	1.080
LiF	30	3.444°	2.420	3.880×10^{-34}	1.708
NaCl	20	3.329°	3.277	3.503×10^{-34}	1.892
NaCl	30	3.591°	3.535	3.778×10^{-34}	1.754

Table III: Data: Planck's Constant

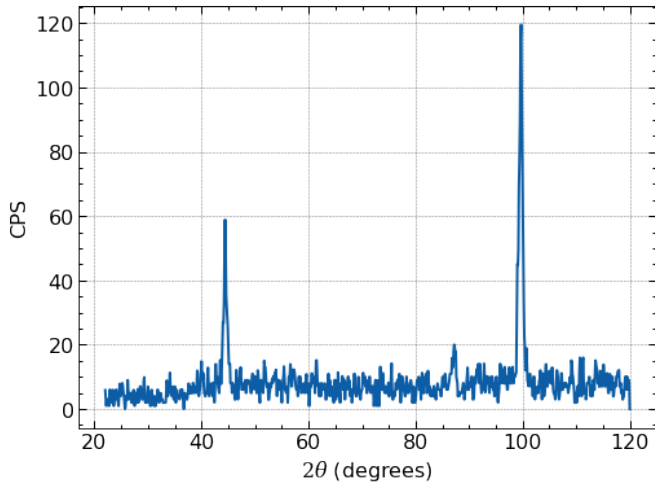


Figure 12: (Old Sample) LiF Spectrum 20 kV

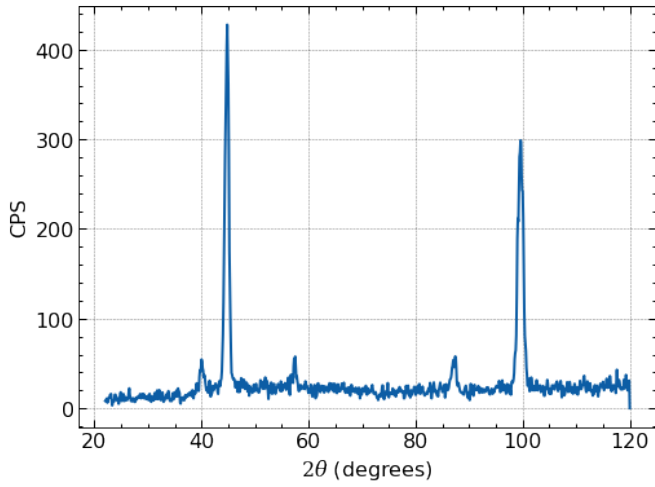


Figure 13: (Old Sample) LiF Spectrum 30 kV

Figures 12 and 13 indicate acquired spectra for an older sample of LiF. Qualitatively speaking, it appears that only the $K\alpha$ peaks are discernible. The $K\beta$ peaks are drowned out by the noise in figure 12, but they are somewhat apparent in figure 9. When Compared to our spectra for the newer samples, the peaks appear to be at the expected positions.

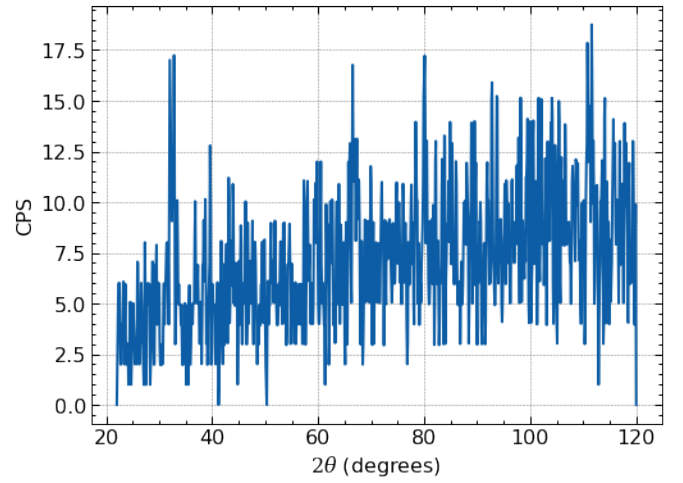


Figure 14: (Old Sample) NaCl Spectrum 20 kV

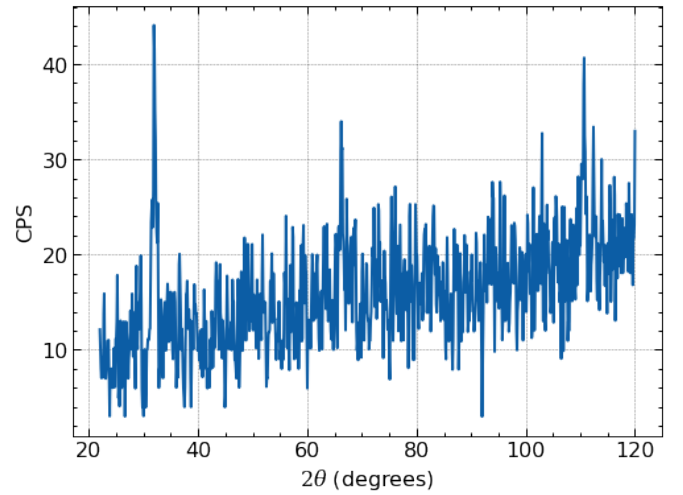


Figure 15: (Old Sample) NaCl Spectrum 30 kV

Figures 14 and 15 indicate acquired spectra for an older sample of NaCl. Qualitatively speaking, our acquired spectra was terribly infested by noise. This is likely due to deformities and acquired impurities in the crystal over time. Perhaps to an extent, this same line of reasoning also explains the weak $K\beta$ signals for the old LiF sample. Nonetheless, we can still somewhat discern $K\alpha$ peaks in figures 14 and 15 which also appear to be at the expected positions.

IV. CONCLUSIONS

As seen above, the values of λ and n were calculated according to the Bragg condition (Eq. 5) using the theoretical value of d as well as the collected data. After performing the experiment at both 20V and 30V for both crystals, we found that all of the calculated values of λ were extremely close to the theoretical values of

$\lambda_{Cu}(K_\alpha) = 1.54\text{\AA}$ and $\lambda_{Cu}(K_\beta) = 1.38\text{\AA}$. The λ value of the NaCl crystal had the highest error of 8.2% at 20V and $\theta = 42.35^\circ$, but that is still quite small.

Using the extrapolated root of the Bremsstrahlung region to find the minimum wavelength λ_{min} for both samples, we were able to calculate Planck's constant h using (Eq. 5). The LiF sample at 20V yielded the most accurate value of h with a % error of only 7.4% while the rest were off by a factor of almost 2.

The sources of error are difficult to pinpoint, but it is possible that the motor was not rotating the detector at exactly twice the angular speed of the crystal, leading to inaccurate θ values throughout the calculations. More-

over, the crystals themselves might not have been in ideal condition, with deformations and impurities affecting the diffraction of the x-rays.

V. AUTHOR CONTRIBUTIONS

Data was taken by all members. Nuno M. wrote the introduction. Lendel D. wrote the "Results and analysis" section and created all of the graphs. Bryant N. wrote the methods and conclusion.

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- [1] J. Hieulle, *Structures and Electronic Properties of Organic Self-Assembled Monolayers Characterized by STM and XPS*, Ph.D. thesis (2014).
 - [2] J. Kowalska and S. DeBeer, *Biochimica et biophysica acta* **1853**, 1406 (2015).
 - [3] "Kilogram: Mass and planck's constant," (2021).