

# X-Ray Crystallography

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In this experiment the lattice constants of NaCl and LiF were determined using x-ray crystallography. The experimentally determined values  $a_{NaCl} = (0.5881 \pm 0.0136)nm$  and  $a_{LiF} = (0.7696 \pm 0.0577)nm$  were found to lie 1.78 and 6.63 standard deviations from their tabulated values, respectively.

## INTRODUCTION

Crystal structures can be deduced from the interference patterns of electromagnetic waves, most commonly using a method known as x-ray crystallography. This method relies on a phenomenon known as Bragg diffraction (Fig.1), in which waves reflecting from adjacent crystalline planes interfere, producing a diffraction pattern. The positions of maximum and minimum intensity in this pattern are used to characterize the spacing and orientation of planes within the crystal.

Bragg's Law:

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

where

$\lambda$  is the wavelength of the rays

$\theta$  is the angle between the incident rays and the surface of the crystal

$d$  is the spacing between layers of atoms

and constructive interference occurs when  $n$  is an integer (whole number)

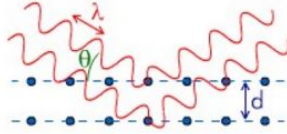


FIG. 1. Bragg diffraction [1]. As waves reflect from adjacent planes, their difference in optical path length " $2d\sin(\theta)$ " results in an interference pattern. By measuring the intensity at various angles, the interplanar spacing " $d$ " can be calculated from Bragg's law.

The orientation of crystal planes follows a convention known as Miller indices. These indices ascribe to each plane a number representing the coefficient scaling the reciprocal lattice vector of that plane. For the purposes of this experiment, only cubic lattices are considered, for which the reciprocal lattice is the same as the lattice itself. Using planar geometry the spacing of the planes  $d$  can be shown to relate to the so called lattice constant  $a$  as follows:

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (1)$$

where  $(h, k, l)$  are the Miller indices. For a cubic

crystal, this reduces to the simple relation  $d = 2a$ .

## EXPERIMENT

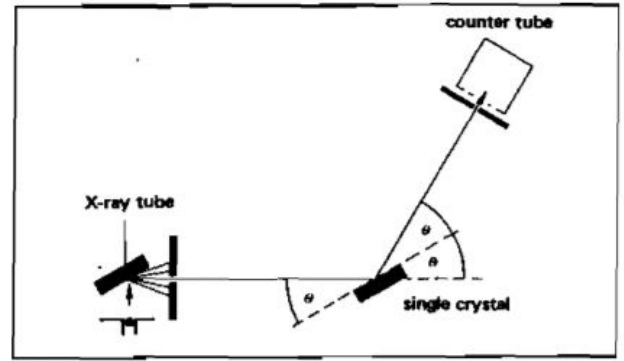


FIG. 2. Experimental apparatus [2]. The angle measured relative to the counter tube is  $2\theta$  where  $\theta$  is the true angle of incidence used in Bragg's law.

X-rays in the 0.1392nm-0.1544nm range were generated using a 40kV potential supplied across the circuit formed by a heated cathode displaced in vacuum from a copper anode. The copper target produced x-rays having wavelengths 0.1540nm, 0.1544nm, and 0.1392nm, corresponding to  $2p_1 \rightarrow 1s$ ,  $2p_2 \rightarrow 1s$ , and  $3p \rightarrow 1s$  transitions, respectively.

These rays were directed toward a mounted NaCl or LiF sample enclosed within a lead-glass shield containing a Geiger-Muller counter. The crystal was rotated using a dial marked in degree increments.

The counter output was connected to a pre-amplifier and subsequent amplifier. The negative output signals from the amplifier were then connected to a discriminator. Discriminated signals were passed to a scaler, used to read the number of counts per time interval.

A nickel filter with an absorption peak of 0.1488nm was used to discern between the  $2p$  and  $3p$

emissions via a series of A/B tests, in which NaCl and LiF crystals were measured with and without the filter for a total of four data sets.

## RESULTS

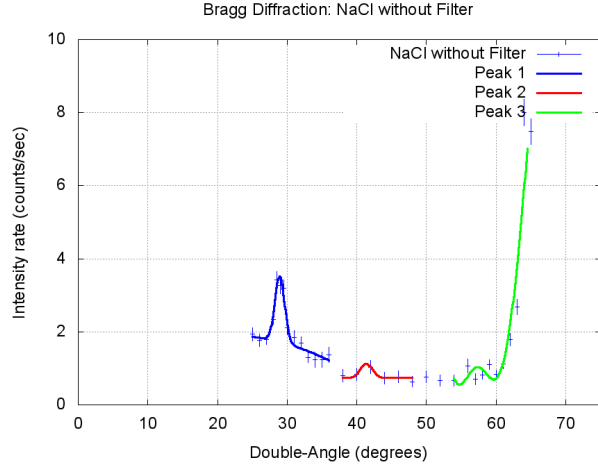


FIG. 3. *NaCl without filter. Peak 1:  $28.95 \pm 1.79$ . Peak 2:  $41.39 \pm 3.02$ . Peak 3:  $57.28 \pm 4.74$ .*

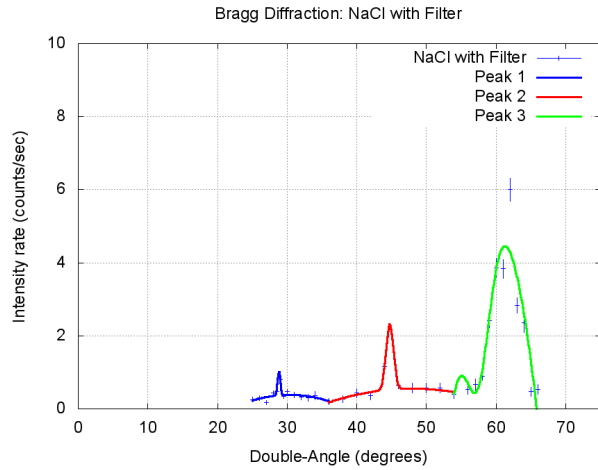


FIG. 4. *NaCl with filter. Peak 1:  $28.83 \pm 1.92$ . Peak 2:  $44.75 \pm 5.86$ . Peak 3:  $57.48 \pm 1.55$ .*

The location of each peak was determined by fitting the surrounding distribution to a Gaussian with polynomial offset up to order 2. Using Bragg's law

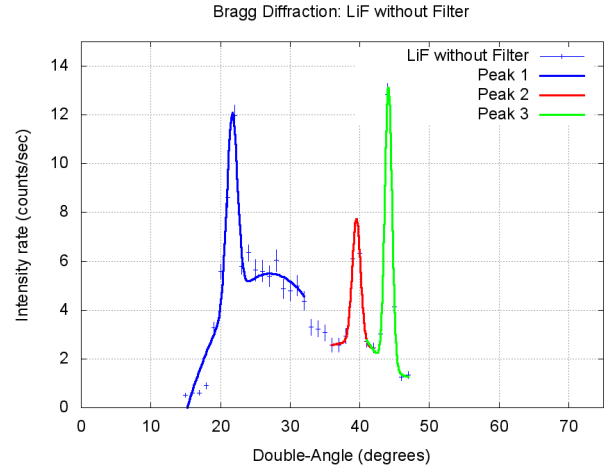


FIG. 5. *LiF without filter. Peak 1:  $21.68 \pm 7.73$ . Peak 2:  $39.52 \pm 5.1$ . Peak 3:  $44.13 \pm 11.44$ .*

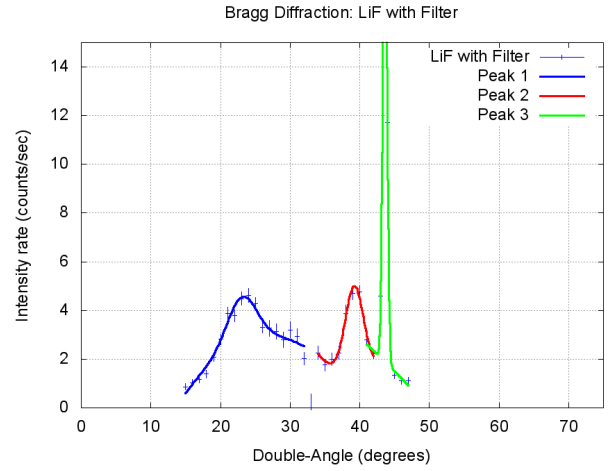


FIG. 6. *LiF with filter. Peak 1:  $23.08 \pm 1.86$ . Peak 2:  $39.23 \pm 3.61$ . Peak 3:  $43.62 \pm 21.30$ .*

and taking a weighted average the lattice constants were determined to be

- $a_{NaCl} = (0.5881 \pm 0.0136)nm$
- $a_{LiF} = (0.7696 \pm 0.0577)nm$

## DISCUSSION

The lattice constant for NaCl is known to be  $0.5640nm$  [3], giving a z-score of 1.78. The lattice

constant for LiF is known to be  $0.4026nm$  [4], giving a z-score of 6.36.

Probable sources of error include the behavior of the detector. After prolonged use the Geiger-Muller counter began to dramatically overcount, as can be seen in the third peak of Fig. 6. This was possibly a result of internal heating.

## CONCLUSION

In summary the lattice constants of NaCl and LiF were determined using x-ray crystallography and found to lie within 1.78 and 6.26 standard deviations from the mean, respectively.

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## REFERENCES

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- [2] Thomas Browder, "*Spectra Diffraction, Fig. 15.5*" (Accessed 2017).  
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