

## VL-M3: DETERMINATION OF THE LATTICE CONSTANTS OF NaCl CRYSTAL BY AN X-RAY DIFFRACTION SIMULATOR

### Objectives:

- To understand the Bragg's law.
- To determine the crystal structure of NaCl by using X-ray diffraction.

### System requirements:

Computer (Desktop/Laptop), Operating systems: Windows, **Bragg Diffraction\_vle (zipped) file.**

### Advise:

Students are advised to follow the **procedures written in this manual very strictly** while performing the experiment. **Do not try to explore anything else in the experiment.**

### Theory:

When X-rays encounter a crystalline material, they are diffracted by the plane of atoms or ions (Bragg plane) within the crystal.

Consider the parallel planes of atoms in the Fig.1 from which X-rays are diffracted. The waves may be reflected from the surface atom H or H'. However, X-rays are reflected not only from the surface plane, but also from the adjacent subsurface plane e.g., H''. If these diffracted rays are to remain in phase and be coherent, the distance MH''P is the path difference between these two diffracted waves, must be equal to integer multiples of the incident wavelength,  $\lambda$ , of X-rays.

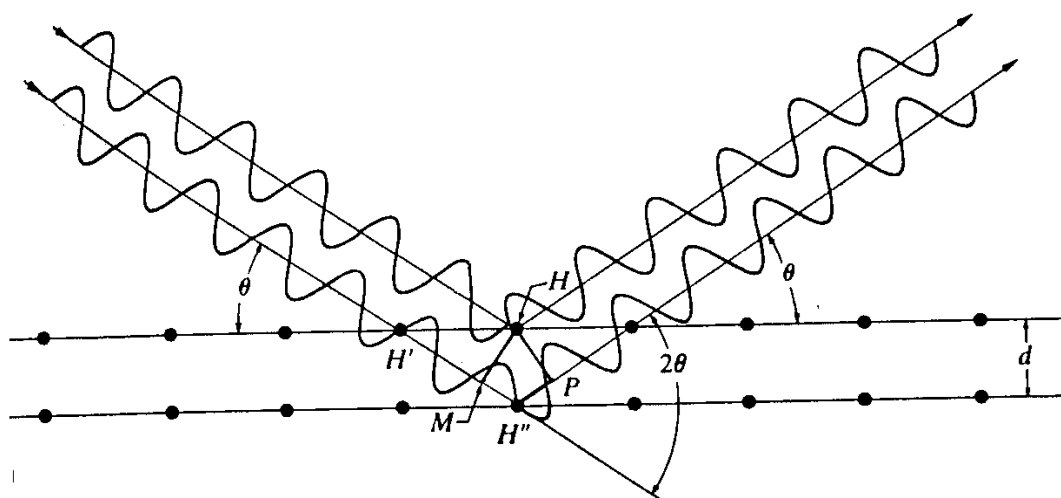
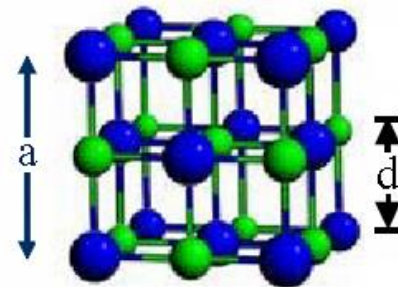


Fig. 1:  $MH''P$  is the path difference between the two diffracted rays from the two parallel planes of the crystal.

From the Fig. 1, the path difference is



**Fig. 2:** Schematic diagram of the NaCl crystal structure.

$$\begin{aligned} MH''P &= MH'' + PH'' \\ &= d \sin \theta + d \sin \theta = 2d \sin \theta \end{aligned}$$

Where,  $\theta$  is the glancing angle and  $d$  is the interplanar distance between the planes.

For constructive interference,

$$2d \sin \theta = n\lambda \dots \dots \dots (1)$$

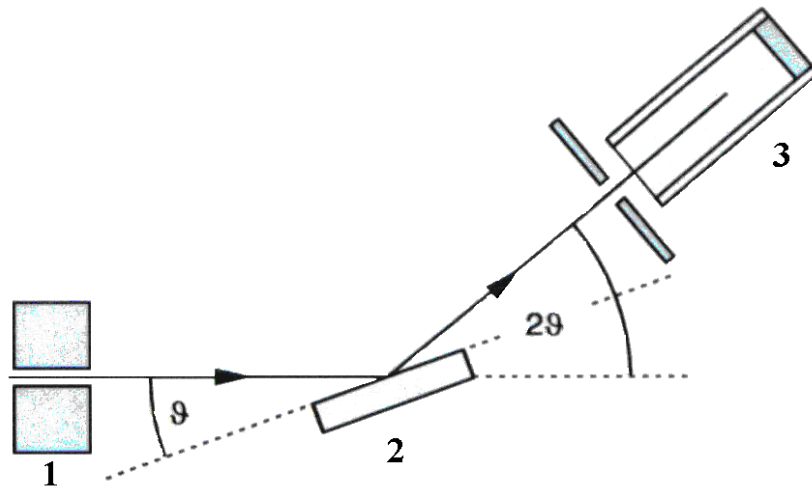
Fig. 2 shows a typical unit cell of NaCl crystal. Sodium Chloride is an fcc crystal structure. The basis is two ions, a sodium cation and a chlorine anion. The lattice planes run parallel to the surface of the crystal's unit cells. The interplanar spacing corresponds to one half of the lattice constant,  $a$ , i.e.

$$d = \frac{a}{2} \dots \dots \dots (2)$$

Substituting value of  $d$  in Eq. 1, the Bragg's condition becomes,

$$\begin{aligned} 2\left(\frac{a}{2}\right) \sin \theta &= n\lambda \\ \Rightarrow a \sin \theta &= n\lambda \\ \Rightarrow a &= \frac{n\lambda}{\sin \theta} \dots \dots \dots (3) \end{aligned}$$

From, Eq. 3 the lattice constant can be determined.

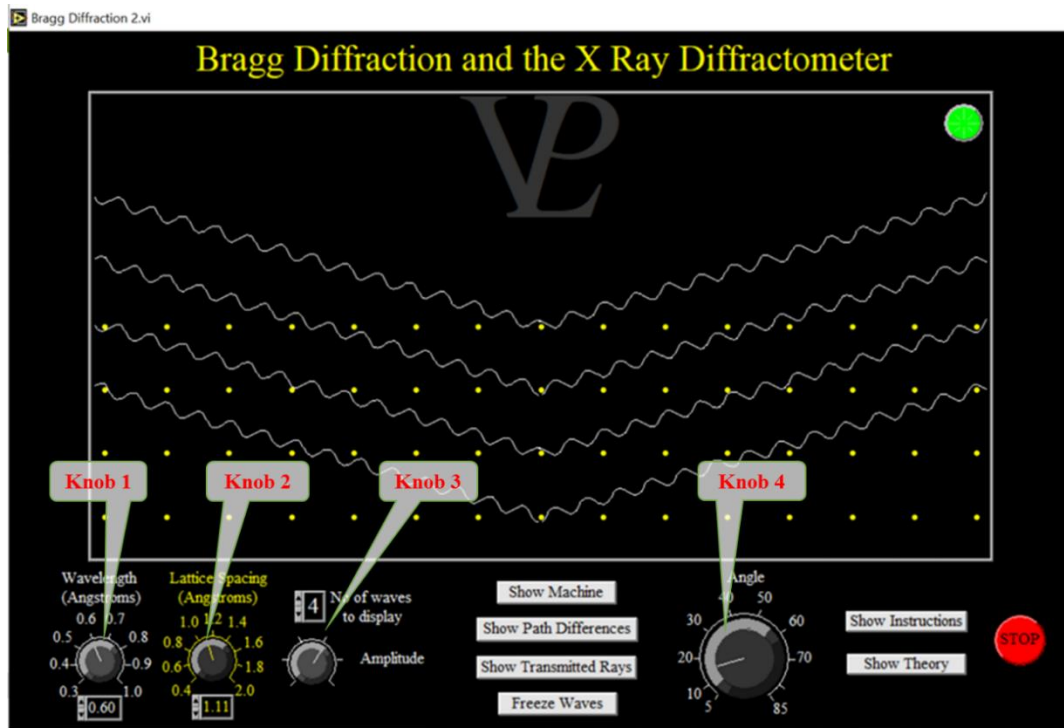


**Fig. 3:** Schematic diagram of diffraction of X-rays from a crystal coupling between counter-tube (detector) and incident X-rays. **1** Collimator, **2** Crystal, **3** counter tube.

Fig. 3 shows the schematic diagram of diffraction of X-rays from a crystal,  $2\theta$  coupling between counter tube angle and glancing angle. This method is more precise when the glancing angles are measured in higher diffraction order.

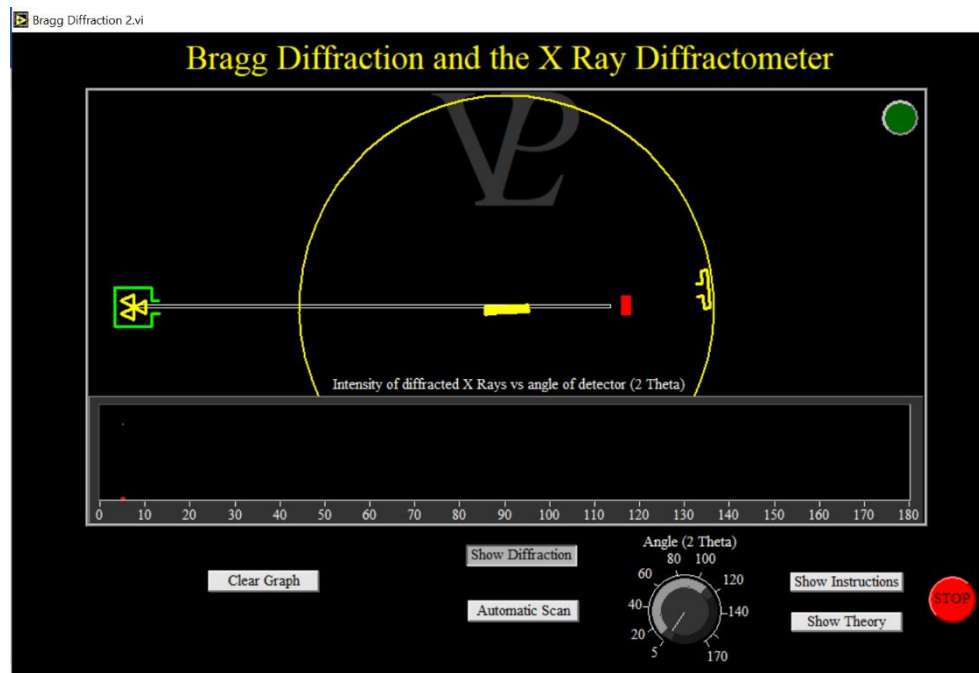
#### Procedures:

1. At first unzip “**Bragg Diffraction\_vle (zipped)**” File. Click on the executable file “**Bragg Diffraction\_vle**” then a window will open as shown in Fig. 4.
2. You can vary the wavelength of the X-rays using the Knob 1 on the left, the lattice spacing using Knob 2 in the middle, amplitude of X-ray using Knob 3, and the glancing angle of X-rays using Knob 4 on the right.



**Fig. 4:** Photograph of the simulated experimental set up.

3. Set the wavelength of the X-ray as  $1.54 \text{ \AA}$ , lattice spacing as  $2.81 \text{ \AA}$ . Set desirable amplitude of X-ray using Knob 3.
4. Press 'Show Machine' button, you will see the typical X-ray diffractometer as shown in Fig.
- 5.



**Fig. 5:** X-Ray diffractometer.

- Press 'Automatic scan' button to scan through the range automatically, you will see an X-ray diffraction pattern (Intensity versus  $2\theta$  plot) as shown in Fig. 6.

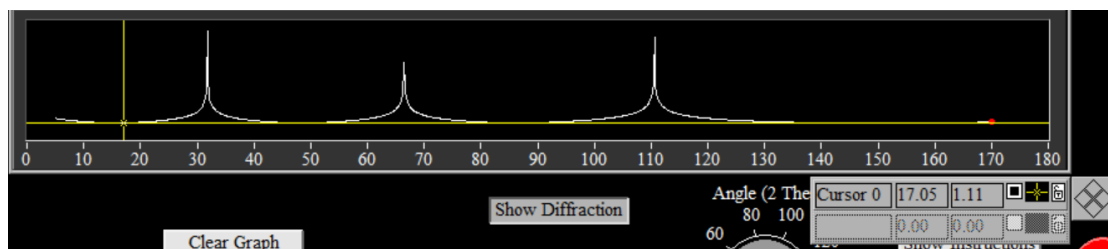


Fig. 6: The intensity of diffracted X-rays versus  $2\theta$ .

- Construct a data Table (as shown in Table 1) of Intensity of diffracted X-rays versus  $2\theta$ , and take data at least  $3^\circ$  interval, however around the peak interval should be  $0.2^\circ$ . To get the experimental data from the graph, right click on the graph and select 'show' and then the 'cursor display' option. It will display two cursors (as shown in Fig.6). If they are locked, unlock any cursor by clicking on the locked button and you will see two cursor pointers (two yellow lines) along X- and Y-axes. Drag the cursor pointers to select data points from the graph.
- Draw the Intensity versus  $2\theta$  graph, you will see three diffraction peaks, which satisfy Bragg's conditions ( $n = 1, 2, 3$ ).
- Construct another Table (Table 2) to obtain  $2\theta$  values for corresponding Bragg's conditions. and calculate the corresponding  $n\lambda$  values.
- Plot  $n\lambda$  vs  $\sin\theta$ , which will be a straight line passing through origin as shown in Fig. 8. The slope of the line will give the lattice constant of the NaCl crystal (Eq. 3).

**Data Collections:**

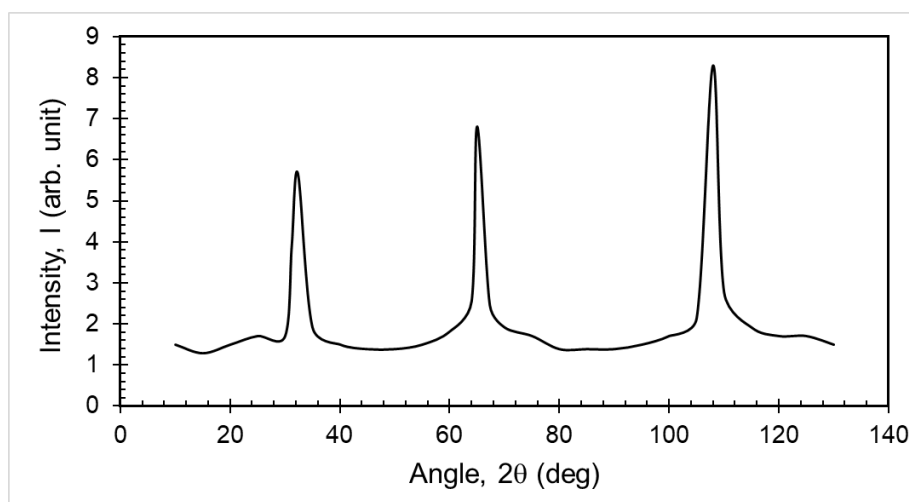
Wavelength of the characteristics X-ray radiation,  $\lambda = 1.54 \text{ \AA} = 1.54 \times 10^{-10} \text{ m}$

**Table 1: The intensity of diffracted X-rays versus  $2\theta$  data.**

Sl.	$2\theta$ (deg)	Intensity of diffracted X-rays (arb. unit)	Sl.	$2\theta$ (deg)	Intensity of diffracted X-rays (arb. unit)
1			16		
2			17		
3			18		
4			19		
5			20		
6			21		
7			22		
8			23		
9			24		
10			25		
11			.		
12			.		
13			.		
14			.		
15			.		

**Calculations:**

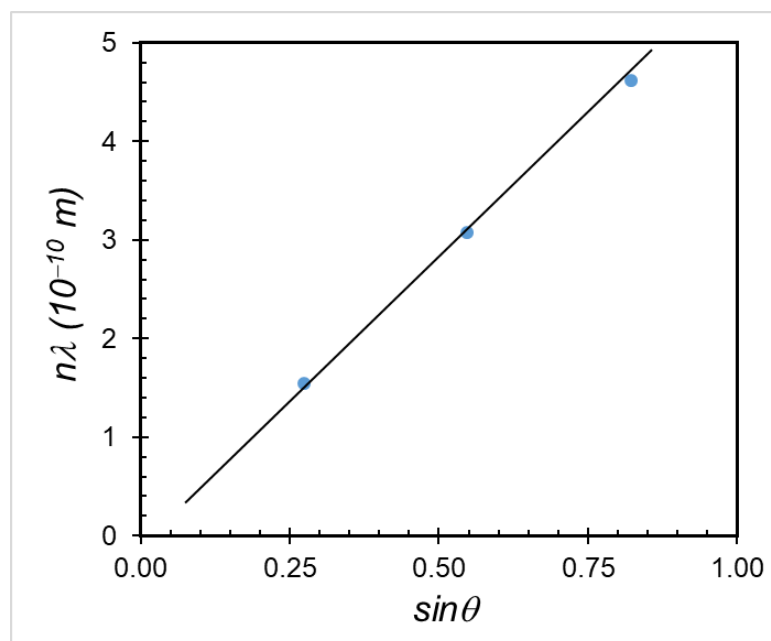
Draw a graph of intensity of diffracted X-rays versus  $2\theta$  data as shown below.



**Fig. 7:** Intensity of diffracted X-rays versus  $2\theta$  data.

**Table 2:** Data for  $\sin\theta$  and  $n\lambda$

$2\theta^\circ$	$\sin\theta$	Diffraction order ( $n$ )	$n\lambda$ (m)
		1	
		2	
		3	



**Fig. 8:** Plot  $n\lambda$  vs  $\sin\theta$

**Result:** The value of the lattice constant of NaCl crystal using an X-ray diffractometer is.....m

**Error analysis:**

**Discussion:**

Based on your understanding from this experiment, answer the following questions:

1. What is a unit cell? Discuss the lattice parameters of a unit cell.
2. Briefly discuss about crystal planes and Miller indices.
3. If you decrease wavelength ( $< 1.5 \text{ \AA}$ ) of the X-rays, what will happen to the Bragg angle for a particular crystal plane?
4. If you increase the interplanar distance ( $> 2.81 \text{ \AA}$ ) of the crystal keeping the wavelength of X-rays fixed, what will happen to the Bragg angle?
5. Can you perform this experiment using visible light, justify your answer?