Determination of Inter-planar Spacing of NaCl Crystal in Presence of Benzene Micro-layer by X-ray Diffraction Method

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A crystal diffraction method is used for the determination of the interplanar spacing of sodium chloride with and without a micro layer of benzene. The apparent displacement of crystal planes is studied using the X-ray diffraction method and by using a device called goniometer (contact angle type) along with an X-ray source and a receiver. A sealed X-ray source is used in a high quality precision goniometer to obtain the direct measurement of diffracted angle in a single determination. The intensity of the diffracted beam without a micro layer of benzene and with different layer of benzene is compared.

1. Introduction

The crystallographic planes [1], [2] are fictitious planes linking nodes. Some directions and planes have a higher density of nodes; these dense planes have an influence on the behavior of the crystal. The smallest distance between two crystallographic planes is known as inter-planar spacing. Inter planar spacing is determined by a goniometer in which a sealed X-ray source [3], called transmitter, is fixed on one arm of the goniometer and a receiver, which receives the diffracted X-ray and measure the intensity of diffracted beam in terms of current (mA), is fixed on the other arm at an equal distance from the centre of the gonoimeter (contact angle type). The inter-planar spacing of sodium chloride crystal is measured by targeting X-ray on (100) plane. A micro-layer of benzene is deposited on the crystal by means a pressure adjusted atomizer and the thickness of the layer is measured correctly by electron microscope, and then the inter-planar spacing is measured. Surprisingly, it was observed that an apparent displacement of the crystal plane is taking place, which do not coincide with the apparent displacement due to diffraction. Benzene is selected due to its high refractive index. The relation, which satisfactorily held for the apparent displacement, is formulated.

2. Technique of X-ray Diffraction

The technique of single-crystal X-ray crystallography has two basic steps.

Step-1
The first, and often most difficult step, is to obtain an adequate crystal of the material under study. The crystal should be sufficiently large (typically larger than 1000 microns in all dimensions) and of pure composition and regular structure with no significant internal imperfections such as cracks or twinning. A small or irregular crystal will give fewer and less reliable data from which it may be impossible to determine the atomic arrangement.

Step-2
In the second step, the crystal is placed in an intense beam of X-rays, usually of a single wavelength (monochromatic X-rays), producing the regular pattern of reflections. As the crystal is gradually rotated, previous reflections disappear and new ones appear, the intensity of every spot is recorded at every orientation of the crystal.

3. Bragg's Law

The law, which was derived by physicist Sir William Lawrence Bragg in 1912 and first presented on 11 November 1912 to the Cambridge Philosophical Society, predicts the conditions under which diffracted X-ray beams from a crystal are possible. In deriving the law, we ignore the structure of the unit cell, which is related only to the intensities of these beams. The dashed sloping lines in Fig. 1 represent the intersection with the plane of the figure of an arbitrary set of planes passing through the elementary diffracting centers. The perpendicular distance between adjacent planes is d (inter-planar spacing). Many other such families of planes, with different inter-planar spacing, can be defined. Fig. 1 shows an incident wave striking the family of planes with the incident rays making an angle θ with the plane. For a single plane, mirror-like "reflection" occurs for any value of θ. To have a constructive interference in the beam diffracted from the entire family of planes in

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the direction θ, the rays from the separate planes must reinforce each other, which is the condition for maxima. This means that the path difference for rays from adjacent planes must be an integral multiple of wavelengths, which is nothing but Bragg's Law.

Mathematically, Bragg's Law can be written as an equation:

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

Where, λ is the wave length [4] of incident X-ray,
n is integer called order of diffraction,
θ is the glancing angle,
d is the inter planar spacing and 2d sinθ is the path difference for rays from adjacent planes.

4. Derivation of Bragg's Law

The incoming beam (coming from upper left) causes each scatterer to re-radiate a small portion of its energy as a spherical wave. If scatterers are arranged symmetrically with a separation d, these spherical waves will be in synch (add constructively) only in directions where their path difference 2d sin θ equals an integer multiple of the wavelength λ. In that case, part of the incoming beam is deflected by an angle 2θ, producing a reflection spot in the diffraction [5] pattern.

Crystals are regular arrays of atoms, and X-rays are the waves of electromagnetic radiation. Atoms scatter X-ray waves, primarily through the atoms' electrons. Just as an ocean wave striking a lighthouse produces secondary circular waves emanating from the lighthouse, so an X-ray striking an electron produces secondary spherical waves emanating from the electron. This phenomenon is known as scattering, and the electron (or lighthouse) is known as the scatterer. A regular array of scatterer produces a regular array of spherical waves. Although these waves cancel one another out in most directions (destructive interference) they add constructively in a few specific directions, determined by Bragg's Law

Bragg's Law can easily be derived by considering the conditions necessary to make the phases of the beams coincide, when the incident angle equals a reflecting angle. The rays of the incident beam are always in phase and parallel up to the point at which the top beam strikes the top layer at atom z (Fig. 1). The second beam continues to the next layer, where it is scattered by atom B. The second beam must travel the extra distance AB + BC if the two beams are to continue traveling adjacent and parallel. This extra distance must be an integral (n) multiple of the wavelength (λ) for the phases of the two beams to be the same i.e.

$$n\lambda = AB + BC \quad (2)$$

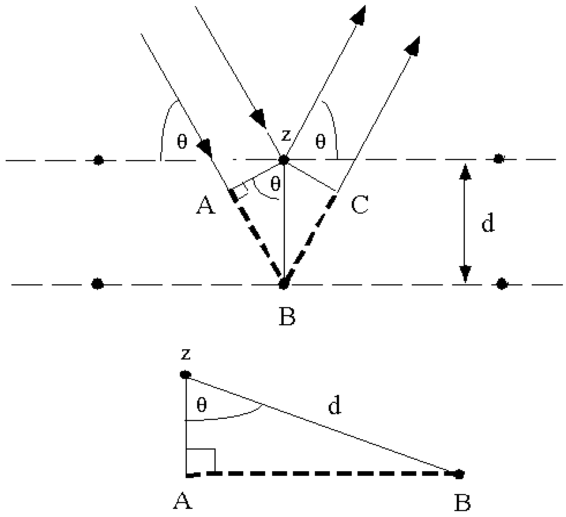

Fig.1: Bragg's Law.

The lower beam must travel the extra distance (AB + BC) to continue traveling parallel and adjacent to the top beam.

Let BZ = d, then from figure

$$AB = d \sin\theta \quad (3)$$

Since AB = BC, then Eqn.2 becomes,

$$n\lambda = 2AB \quad (4)$$

Substituting Eqn.3 in Eqn.4 we have,

$$n\lambda = 2\,d \sin\theta \quad \text{or} \quad 2\,d \sin\theta = n\lambda \quad (5)$$

Hence Bragg's Law is derived.

Diffracted waves from different atoms can interfere with each other and the resultant intensity distribution is strongly modulated by this interaction. If the atoms are arranged in a periodic fashion in crystals, the diffracted waves will consist of sharp interference maxima (peaks) with the same symmetry as in the distribution of atoms.

5. Procedure

Here, the object is to plot [6] a graph between intensity vs. grazing angle [7] for two different Bragg's plane and then placing a micro layer of benzene by a pressure adjusted atomizer on it and repeating the same procedure to plot the graphs.

1. Straighten the goniometer arms such that Transmitter and receiver face each other directly then the reading will be at 0 and 180 degrees.
2. Rotating table is kept on the goniometer and is rotated such that the angle indicator reads 0 degrees. Here grazing angle is noted not the angle of incidence. Grazing angle is the angle between the plane surface and the incident ray. This is the complement of incident angle.
3. Now NaCl crystal is kept on the rotating table in the center of the goniometer such that the Bragg's plane (100) is parallel to both the analyzer and source.
4. Transmitter AC adapter is connected and receiver is on. Multiplier and fine-tuning knobs are adjusted on the receiver. One should so aware that nothing (arms, head, books etc.) was close to the apparatus while making the reading to minimize the reflections which may cause interference and give false measurements.
5. Rotating table was turned and hence the crystal by two degrees clockwise and the movable arm of goniometer through four degrees clockwise. Here, we maintain the incident angle the same as the reflected angle, a condition necessary for Bragg's diffraction. Reading was noted and recorded. The procedure was repeated each time rotating table two degrees (four degrees for movable arm) to take new reading. The multiplier setting was changed (30X, 10X, 3X) [8] to get the meter needle with in the mid range. It works like a multi-meter with selectable ranges, switching to the range that gives the most significant digits in the measurement, which improve the precision of data.
6. The data was plotted and peaks in the graph noted. Each peak corresponds to a maxima in the 2d sinθ =n λ equation. The higher order maxima have greater angles. From the data and labeled frequency on the transmitter, the experimental value of inter planar spacing between consecutive Bragg's plane is calculated.
7. Then, a micro-layer of benzene is sprayed by a pressure adjustable atomizer on the surface of NaCl crystal. The thickness of the layer was measured by means of a micrometer fitted with an electron microscope.
8. The above procedure is again repeated for different thickness of benzene layer and the corresponding inter planar spacing is calculated from the graph and is compared with inter planar spacing with out the micro layer of Benzene.

(1) Bragg's diffraction without a micro layer of benzene

Table 1: Measurement of intensity of diffracted ray in terms of current through (100) plane

100 Bragg Plane			100 Bragg Plane	
Degrees	Intensity (μA)		Degrees	Intensity (μA)
0	100.12			
2	91.23		52	67.53
4	88.01		54	62.69
6	73.01		56	52.72
8	67.13		58	51.91
10	58.24		60	54.71
12	51.27		62	54.24
14	50.18		64	51.78
16	375.29		66	256.42
18	57.52		68	51.28
20	52.63		70	77.35
22	53.28		72	62.46
24	110.79		74	50.95
26	53.92		76	52.39
28	51.83		78	55.56
30	56.39		80	51.43
32	304.98		82	53.83
34	54.67		84	52.69
36	82.01		86	238.52
38	49.29		88	54.35
40	98.38		90	50.21
42	51.93		92	48.91
44	53.82		94	52.27
46	51.37		96	49.35
48	379.76		98	50.79
50	49.24		100	51.32

In Table 1, the values obtained from the experiment are noted.

Calculation

Wave length of X- Ray = λ = 0.1542 nm

For n= 1, $2\theta = 16^0 \Rightarrow \theta = 8^0$

So,

$$d_1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 8^0} = \frac{0.1542}{2 \text{ x } 0.13973} = 0.552 \text{ nm}$$

For n = 2, $2\theta = 32^0 \Rightarrow \theta = 16^0$

$$\text{So, } d_2 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 16} = \frac{0.1542}{0.27564} = 0.559 \text{ nm}$$

For n = 3, $2\theta = 48^0 \Rightarrow \theta = 24^0$

So, d_3 =

$$\frac{n\lambda}{2\sin\theta} = \frac{3 \text{ x } 0.1542}{2\sin 24^0} = \frac{1.5 \text{ x } 0.1542}{0.4067} = 0.5687 \text{ nm}$$

For n = 4, $2\theta = 66^0 \Rightarrow \theta = 33^0$

So,

$$d_4 = \frac{n\lambda}{2\sin\theta} = \frac{4 \text{ x } 0.1542}{2\sin 33^0} = \frac{2 \text{ x } 0.1542}{0.5446} = 0.566 \text{ nm}$$

For n = 5, $2\theta = 86^0 \Rightarrow \theta = 43^0$

So, d_5 =

$$\frac{n\lambda}{2\sin\theta} = \frac{5 \text{ x } 0.1542}{2\sin 33^0} = \frac{2.5 \text{ x } 0.1542}{0.6820} = 0.565 \text{ nm}$$

But $d = (d_1 + d_2 + d_3 + d_4 + d_5) / 5$
= (0.552 +0.559 +0.5687 + 0.566 + 0.565) /5
= 0.56214 nm

So, d = 0.56214 nm (6)

If we draw a graph by taking 2θ along X-axis and the corresponding intensity (μA) of the diffracted beam along Y-axis, the nature of the graph will be like Fig. 2 and the peaks in the graph is noted. Each peak corresponds to a maxima in the 2d sinθ = nλ equation. The higher order maxim has greater angles. From the figure, it is clear that maximum intensity occur at nearly equal interval but with varying magnitude of intensity. The intensity monotonically decreases and further monotonically increases.

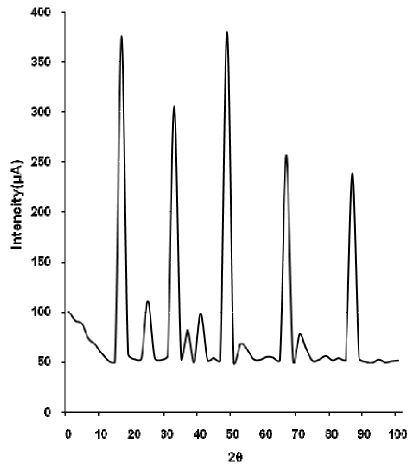

Fig.2: Intensity distribution curve through (100) plane without micro layer of benzene.

(2) Bragg's diffraction with a micro layer of benzene t = 10μm

Table 2: Measurement of intensity of diffracted ray in terms of current through (100) plane, t= 10μm.

100 Bragg Plane t = 10μm			100 Bragg Plane t = 10μm	
Degrees	Intensity (μA)		Degrees	Intensity (μA)
0	88.35			
2	76.87		52	45.24
4	72.79		54	44.36
6	69.26		56	42.08
8	67.56		58	46.12
10	65.39		60	42.54
12	61.39		62	41.33
14	58.91		64	87.98
16	53.46		66	42.81
18	49.27		68	41.71
20	44.58		70	45.38
22	45.14		72	46.22
24	346.72		74	43.57
26	47.07		76	304.77
28	45.15		78	42.09
30	78.83		80	42.41
32	42.31		82	78.26
34	43.94		84	43.21
36	45.59		86	41.11
38	89.23		88	39.94
40	45.73		90	38.75
42	44.04		92	40.36
44	46.19		94	42.62
46	50.26		96	41.68
48	48.95		98	43.09
50	352.17		100	41.13

In Table 2, the tabulated values obtained from the experiment when a micro layer of thickness 10μm of benzene is spread on the surface of the sodium chloride crystal.

Calculation

Wave length of X- Ray = λ = 0.1542 nm

For n= 1, $2\theta = 24^0 \Rightarrow \theta = 12^0$

So,

$$d_1^1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 12^0} = \frac{0.1542}{2 \text{ x } 0.2079} = 0.3709 \text{ nm}$$

For n = 2, $2\theta = 50^0 \Rightarrow \theta = 25^0$

$$\text{So, } d_2^1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 25} = \frac{0.1542}{0.4226} = 0.3649 \text{ nm}$$

For n = 3, $2\theta = 76^0 \Rightarrow \theta = 38^0$

So, d_3^1 =

$$\frac{n\lambda}{2\sin\theta} = \frac{3 \text{ x } 0.1542}{2\sin 38^0} = \frac{1.5 \text{ x } 0.1542}{0.6157} = 0.3757 \text{ nm}$$

But $d^1 = (d_1^1 + d_2^1 + d_3^1) / 3$

$= (0.3709 + 0.3649 + 0.3757) / 3$

$$\text{So, } d^1 = 0.3719 \text{ nm} \quad (7)$$

$$\text{For } t = 10 \text{ μm}, \quad d^1 = d\,\frac{e^t}{\mu} = \frac{0.56214 \text{ x } e^{10^{-5}}}{1.51}$$

$= 0.372278$ nm

The above value coincides with the value obtained from the experiment. This concludes that there exist apparent displacements.

The graph between 2θ vs intensity (μA) is shown in Fig. 3.

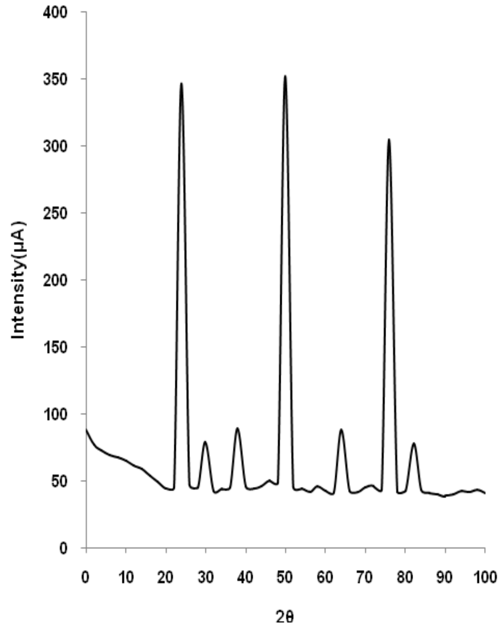

Fig.3: Intensity distribution curve through (100) plane with a micro layer of benzene t= 10μm.

Fig. 4 shows the comparison between the intensities and order of diffraction for the experiment carried with out and with a micro layer of t =10μm of benzene.

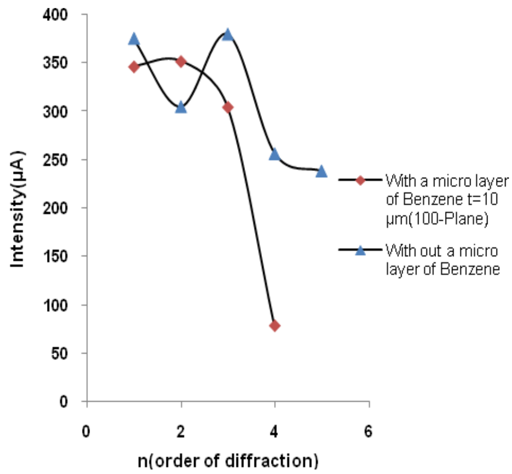

Fig.4

(3) Bragg's diffraction with a micro layer of benzene t = 100μm

Table 3: Measurement of intensity of diffracted ray in terms of current through (100) plane, t= 100μm.

100 Bragg Plane t = 100μm		100 Bragg Plane t = 100μm	
Degrees	Intensity (μA)	Degrees	Intensity (μA)
0	71.72		
2	70.15	52	49.39
4	67.12	54	52.32
6	62.09	56	51.86
8	61.67	58	50.91
10	57.35	60	68.60
12	46.25	62	46.51
14	78.26	64	42.50
16	51.91	66	45.38
18	54.15	68	59.35
20	52.29	70	46.75
22	53.34	72	47.75
24	269.68	74	45.42
26	50.19	76	48.24
28	51.53	78	240.59
30	53.55	80	48.13
32	52.62	82	49.74
34	54.47	84	49.48
36	51.23	86	47.49
38	78.48	88	46.28
40	49.95	90	50.30
42	48.54	92	46.35
44	47.57	94	45.46
46	51.45	96	46.75
48	246.36	98	44.23
50	50.18	100	45.11

In Table 3 the tabulated values obtained from the experiment, when a micro layer of thickness 100μm of benzene is spread on the surface of the Sodium chloride crystal is noted.

Calculation

Wave length of X- Ray = λ = 0.1542 nm

For n= 1, $2\theta = 24^0 \Rightarrow \theta = 12^0$

So,

$$d_1^1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 12^0} = \frac{0.1542}{2 \text{ x } 0.2079} = 0.3709\text{ nm}$$

For n = 2, $2\theta = 48^0 \Rightarrow \theta = 24^0$

$$\text{So, } d_2^1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{\sin 24} = \frac{0.1542}{0.4067} = 0.3791\text{ nm}$$

For n = 3, $2\theta = 78^0 \Rightarrow \theta = 39^0$

So, d_3^1 =

$$\frac{n\lambda}{2\sin\theta} = \frac{3 \text{ x } 0.1542}{2\sin 39^0} = \frac{1.5 \text{ x } 0.1542}{0.6293} = 0.3675\text{ nm}$$

Hence $d^1 = (d_1^1 + d_2^1 + d_3^1) / 3$

$= (0.3709 + 0.3791 + 0.3675) / 3$

So, $d^1 = 0.3725$ nm - (8)

For

$$t = 100\ \mu m,\ d^1 = d\frac{e^t}{\mu} = \frac{0.562 \text{ x } e^{10^{-4}}}{1.51}$$

$= 0.37222$ nm

The above value coincides with the value obtained from the experiment. This concludes that there exists an apparent displacement.

The graph between 2θ vs intensity (μA) is shown in Fig. 5.

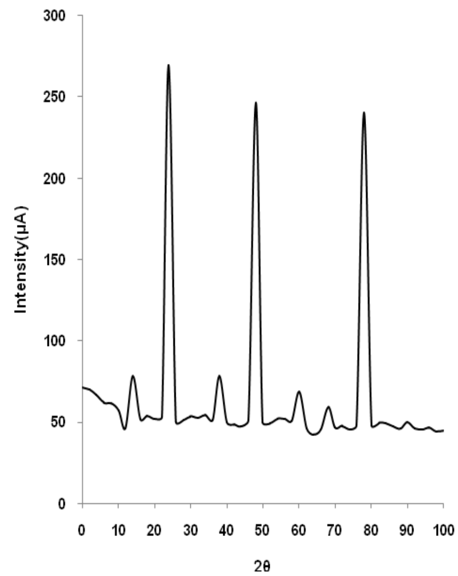

Fig.5: Intensity distribution curve through (100) plane with a micro layer of benzene t= 100μm.

Fig. 6 shows the comparison between the intensities and order of diffraction for the experiment carried with out and with a micro layer of t =100μm of benzene.

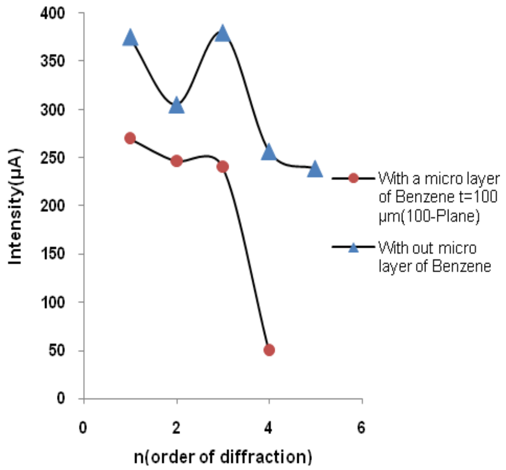

Fig.6

(4) Bragg's Diffraction (with a micro layer of benzene t = 1000 µm)

Table 4: Measurement of intensity of diffracted ray in terms of current through (100) plane, t= 1000µm.

100 Bragg Plane t = 100µm			100 Bragg Plane t = 100µm	
Degrees	Intensity (µA)		Degrees	Intensity (µA)
0	67.97			
2	65.38		52	39.36
4	61.54		54	40.78
6	59.33		56	41.39
8	58.25		58	39.07
10	54.45		60	42.47
12	62.53		62	67.38
14	45.67		64	38.52
16	44.07		66	37.51
18	43.73		68	41.49
20	44.98		70	42.12
22	45.15		72	57.94
24	246.65		74	37.20
26	44.43		76	38.47
28	41.28		78	238.49
30	40.87		80	40.30
32	41.93		82	41.25
34	38.91		84	42.29
36	62.80		86	39.57
38	41.23		88	42.82
40	38.57		90	41.80
42	39.13		92	41.83
44	40.20		94	40.92
46	41.15		96	41.37
48	234.26		98	42.75
50	38.09		100	42.02

In Table 4, the tabulated values obtained from the experiment when a micro layer of thickness 1000µm of the benzene is spread on the surface of the sodium chloride crystal is noted.

Calculation

Wave length of X- Ray = λ = 0.1542 nm

For n= 1, $2\theta = 24^0 \Rightarrow \theta = 12^0$

So,

$$d_1^1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 12^0} = \frac{0.1542}{2 \text{ x } 0.2079} = 0.3709 \text{ nm}$$

For n = 2, $2\theta = 48^0 \Rightarrow \theta = 24^0$

So, $$d_2^1 = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{\sin 24} = \frac{0.1542}{0.4067} = 0.3791 \text{ nm}$$

For n = 3, $2\theta = 78^0 \Rightarrow \theta = 39^0$

So, d_3^1 =

$$\frac{n\lambda}{2\sin\theta} = \frac{3 \text{ x } 0.1542}{2\sin 39^0} = \frac{1.5 \text{ x } 0.1542}{0.6293} = 0.3675 \text{ nm}$$

But $d^1 = (d_1^1 + d_2^1 + d_3^1)/3$
$= (0.3709 + 0.3791 + 0.3675)/3$
$= 0.3725$ nm

So, $d^1 = 0.3725$ nm (9)

For t = 1000 µm, $$d^1 = d\frac{e^t}{\mu} = \frac{0.562 \text{ x } e^{10^{-3}}}{1.51}$$
$= 0.372557$ nm

This coincides with the experimental value.

The graph between 2θ vs intensity (µA) is shown in Fig. 7.

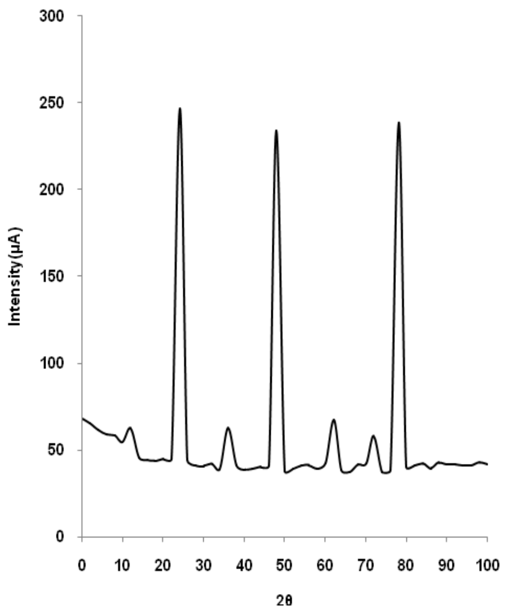

Fig.7: Intensity distribution curve through (100) plane with a micro layer of benzene t= 10000µm.

Fig. 8 shows the comparison between the intensities and order of diffraction for the experiment carried with out and with a micro layer of t =1000µm of benzene.

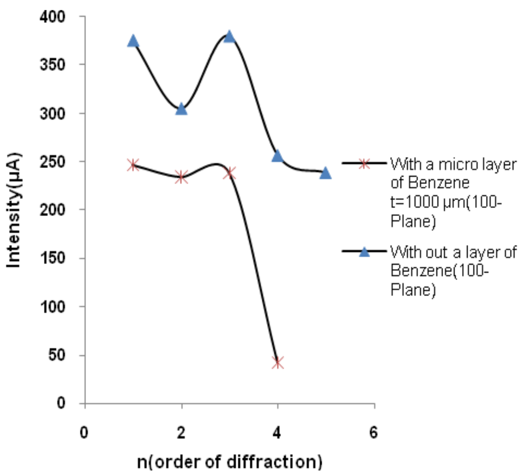

Fig.8

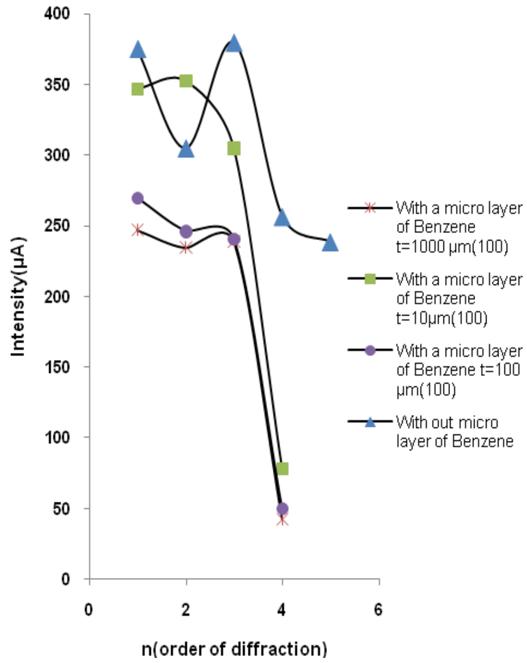

Fig.9

Fig. 9 shows the common comparison between the intensities and order of diffraction for the experiment carried with and without a different micro layer of benzene. The thickness of the Benzene layer is t=10µm, t=100µm and t=1000µm. From the figure, it is clear that the maximum intensity of the diffracted beam is more with out any layer of benzene and maximum intensity decreases as the layer of benzene increases due to absorption.

6. Conclusion

On the basis of the structure of solids, especially for sodium chloride using a scaled-up version of x-ray diffraction, the variation of inter planar spacing is studied when a different fine layer of interfering materials (e.g., benzene) is present on the crystal surface. More precaution has been taken to find the thickness of benzene layer because it is little volatile in nature. Constant reading of electron microscope has been taken to find the exact thickness of benzene layer. In this experiment, the wavelength from a sealed x-ray source of rotating anode generator having wave length 0.1542 nm is used and the apparent spacing successfully determined. Inter planar spacing between two Bragg planes is obtained by the formula $d^1 = d\frac{e^t}{\mu}$, where d is inter planar spacing with out any layer of benzene on the surface of the sodium chloride crystal, t is the thick ness of the micro layer and μ is the refractive index of benzene. The inter planar spacing remains nearly same as the thickness of the layer sprayed on the sodium chloride crystal increases, but is less than the actual inter planar spacing. However, the intensity of diffracted ray decreases with the increase in the thickness of the benzene layer due to absorption factor.

This is a simple experiment, which can be fabricated and installed at low cost in laboratory for crystallographic research and it is also a convenient method to determine the refractive index of liquids.

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Received: 20 April, 2011
Accepted: 11 July, 2011