

Experimental Study of the Lattice Constant and Lattice Spacing Of Potassium Bromide by Laue and Debye Scherer Methods

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Abstract--- This paper presents the calculation of Lattice parameters of the structure of crystals of Crystalline and powder ionic salts of KBr by a x-rays LD apparatus. The crystals structure study is important to get knowledge about the different characteristics of materials just like by kinetic molecular theory. Due to analysis of materials on the basis of diffraction phenomena, the field has been made more growing and sophisticated. X-rays diffraction by points of the lattice provide radiograph on special Dentus films using X-ray machine. X-ray diffraction technique is an easy approach to crystallography as compared to electron and neutron diffraction due to its ease, availability and easily handling. By developing the film under darkroom conditions, the data is collected by indexing the pattern of spots and rings or the images and is tabulated for different parameters by calculation of the sample through Laue and Debye Scherer methods. The obtained results are much closed to the actual results. This research work will also be helpful in the crystallography by different techniques of different alkali halides samples.

Index Terms— scattering angle, count time, scattering rate, atomic number of aluminum, Debye Scherer Method, Lau method, X-ray

1. INTRODUCTION

Early civilizations have made materials of their level and just to fill their needs e.g Bronz, iron and Stone Age; it is because of their access to limited materials. With the passage of time, they investigated techniques for designing materials having characteristics better than the natural ones. They also discovered that characteristics can be changed by changing temperature of materials and making their mixtures (alloys) e.g N-type, P-type materials for designing diodes, transistors and amplifiers etc. By availing suitable materials and developing many technologies have made our life comfortable [1]. Material science spans physics, chemistry and engineering of matter and its utility in industry for manufacturing processes. Society is strongly dependent on the development and utility of advanced

materials now-a-days. e.g optical fibers for telecommunication, semiconductors for photocells and

silicon for microchips. It is the study about structure of matter, properties of matter, its mutual relation and the development of new improved materials [2]. Core electrons and nuclei approach describes the structure of metals, it is due the fact that electrons in metallic lattice are in a gas (Fermi electron gas model), according to which electronic orbitals in atoms is a macroscopic object and Pauli Exclusion Principle provides fundamental behavior of metal. What type of bonding may be, the said model is true for most of it. The atomic cores (nuclei) are assumed to be hard spheres which present the volume of the atom, because electrons occupy negligible volume as compared to the nucleus. These hard spheres results in a closed packed arrangement at which nearest neighbors are placed around the central sphere at six planes, where all spheres have same size. This atomic packing factor reveals the structure of material [3]. The study of materials enables us to select a material for use according to its performance and cost. Analysis of material developing techniques will guide towards limitation of materials and its characteristics with use. Due to material testing and structural observations, the

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clue for products of desirable properties can be determined. In industry, material study will be more helpful to choose more durable and economic samples for manufacturing, like hardware and software [4]. Solid matter exists in either crystalline or amorphous or combination of both. The constituents of crystalline solid are arranged in an orderly repeating pattern extending in all three spatial dimensions. Due to periodic arrangement it has long range atomic order [5-7]. Mostly metals and semi-conductors are crystalline solids. In amorphous solids there is no long order due to the position of their constituents (atoms or molecules). Due to this fact of constituent's randomness, amorphous solids have no definite and distinct shape, Such as glass or Plastic etc. Those materials whose crystallites are of varying size and orientation are known as polycrystalline materials, mostly ceramics and metals exist in poly crystalline form [8-10]. In the beginning of 20th century, it was realized that for the knowledge about non-intrusive structure of matter, x-rays can be used. This was the origin of modern Crystallography [11]. X-rays had been discovered by Roentgen in 1885 which are the beams of light not visible to naked eyes. When x-rays hit an object, the atoms of the object scatter the x-rays. For the structure of large and complex molecular structures, x-rays diffraction is the most efficient method. X-ray diffraction technique is a microscopic approach for getting most of biological structures, such as ribosomes and fatty acids [12]. Crystallographers discovered crystals because of their regular atomic structure. As x-rays scatters in different directions, by measuring these directions and intensity of the scattered beam, scientists are able to produce three dimensional picture of Crystal atomic structure. Crystals proved to be ideal for studying the structure of matter level. X-rays crystallography is used to deposit electromagnetic images in the same size as CCP4 format, which will contribute to increase the use of electromagnetic data. Crystal is associated with an array of discrete points, which

can be made by translational operation and was devised by ludwingseeber in 1840. In august 1850 Bravais suggested symmetries and derived all 14 lattice symmetries [1]. William H. Bragg and William L. Bragg, father and son solved the structure of several inorganic compounds with addition to diamond by x-rays diffraction. W.H Bragg while working on Laue formulations handled so many x-ray tubes and ionization chambers, which were extremely delicate instruments but evident tools for x-ray crystallography techniques. Bragg crystal structure technique was successful for the next fifty years i.e up to 1960. Later on this technique greatly improved with incorporation of computer technology. Before 1960 ,analysis of 20 atomic structures can be done in 1-2 days, but now-a-days modern crystallography can analyze 100-200 atomic structures in just 1-2 days. The structures of organic , inorganic , organometallic and biological structures can be studied through x-rays crystallography every year. Using x-rays diffraction technique, the electron density can also be determined. For rational drug design on functional studies to help in its development is possible in which molecular structural technique is inevitable. Crystallographers put their data in database, which is easily available to public. For example Cambridge structural database (CSD). For biological molecules, such as protein, the data is available in protein data bank (PDB). The data of structure of nucleic acid can be found in nucleic acid database (NDB) etc. As single molecules are neither possible to see nor handle. To know about various parameters of samples, its crystals are essential for looking at its three dimensional array [13]. The crystal structure study of metals and alloys helps to establish coordination number of atoms, concentration limits of different crystalline phases in alloys and the characteristics of atomic radii etc of metal atoms. In case of simple ionic salts, crystallography informs one about unit cell geometry, lattice parameters, and the sequence of array of points. For inorganic compounds , van der waals

bonding can also be investigated by this technique [14-15]. From the urine of multiple myeloma patient, the bence Jones proteins (in grams) were purified in Melbourne and its dry powder was deposited at room temperature before its ionization. For this purpose x-rays diffraction technique was used, whose biochemical investigation shown that it exhibit the properties of normal bence jones proteins.[15-16]. Those solvents in which the compounds produce super saturated solutions and gives rise to crystals of very small size (micro crystals) are not recommended for crystal formation. One method, which is difficult to achieve is the evaporation of compound (which is soluble in non polar solvents) will give crystals. Hydrogen bonding is also very successful in crystalizing solutions. This process provides energy to lattice for its better packing. Apart from it amides are more efficient solvents, which is favorable to take part in hydrogen bonding. For making x-ray quality crystals, benzene is better solvent [17]. In past crystallography has been limited to ground state structures only but now a days that tradition has been removed and in a recent review, a biochemical article has been presented by Moffat in 2001. This is a diffraction study of an organic molecule in a short time of picosecond. Laue polychromatic technique can be used for these macromolecular structure studies. This technique was applied for dissociation of carbon mono oxide from myoglobin. Also the enzyme substrate reaction was studied which occurs in iso citrate dehydrogenase [18]. In this research paper we have used two different techniques is used to determined and calculate the lattice spacing and lattice constant of potassium bromide crystal and powder sample by using Von Laue and Debye sheerer methods respectively. For indexing the pattern of spots or rings, manual method is used rather than software method.

2. MATERIALS AND METHODS

This experiment is performed in the LD Didactic 554800 X-Ray apparatus. This apparatus is fully featured, microprocessor and controlled x-ray device in which

molybdenum target is used for x-rays generation. It consists of an experimental chamber, which is radiation proof. It can be used to perform a variety of experiments. X-Ray apparatus 554800 is also fully assembled and basic device adjustable for all the tubes such as, Fe, Cu, Mo, Ag and W for x-ray generation. In apparatus there is also zirconium filter to make the x-ray beam monochromatic, which is incident on powder sample in debyes scherrer experiment .

In the present experiment, the molybdenum tube is used as an source to produce x-ray. This source produces characteristic x-ray of $K\alpha$ of 17.4 Kev having wavelength 71.1 pm and molybdenum $K\beta$ of 19.6 Kev having wavelength of 63.1pm. This system is designed in such a manner that x-rays generated only when the door of the system is closed for safety purpose. The tube voltage can be adjusted from minimum value of zero volts up to a maximum value of 35 Kv and tube current ranges between zero amperes and one milliampere for x-ray generations. This system can also be connect to a computer with the help of USB port (software included) for displaying the Laue diagram on computer screen but in the present experiment the Laue spot and Debye scherrer rings is formed on a x-ray film for diagnostic analysis of the sample.

In the present experiment the apparatus is set in a range of voltage to produce x-ray of $K\alpha$ of 17.4 kev having wavelength 71.1 pm as it is more energetic as compare to any other type of x-rays. The diagram of the x-ray apparatus is shown in figure 1.

554 801 X-ray apparatus



Fig 1: LD Didactic 554801 x-ray apparatus [19].

Inside LD x-ray apparatus, there is a film holder and experiment Rail. The film holder for x-ray apparatus 554800 possess a printed scale for defined positioning of film for transillumination by x-rays. On the experiment rail, the film holder is clamped. Experiment rail includes a millimeter scale and pin hole diaphragm. The x-ray films are used for the imaging of the samples. For processing in day light, high sensitivity sealed films for x-rays exposure are made along with developer and fixer chemicals. x-ray films are placed in light proof plastic foil for use in day light. Usually these films are of two types i.e. (1)Agfa dentus M2 comfort (2) Kodak ultra-speed dentus film. These imaging films have the following characteristics. These films are soft, flexible and sensitive. These films are highly contrasted having maximum density. These films are fully protected from moisture contamination and can be disinfected. These films support in diagnosis due to excellent grey graduations. These films help to reduce distortion of image due to their flexibility. The double sealed packets completely prevent the moisture access. These films have low base plus fog density. In the present experiment crystal and powder forms of potassium bromide is used as a sample for characteristics of Laue and Debye Scherrer diagrams.

We have used the potassium bromide as crystal and powder for Laue and scherrer diagrams due to the

following characteristics; It is a salt extensively used as an anticonvulsant. Due to presence of bromide it is more active. It is white crystalline powder under standard conditions. It is soluble in water. It has sweet taste in dilute aqueous solution. It is odorless powder. At high concentration it strongly irritates gastric and also causes sometimes vomiting.

In Laue experiment, potassium bromide crystal of the following dimension is attached with the pin hole of the diaphragm.

Dimension: 4.5 mm x 4.5 mm x 0.5 mm.

For structure study of potassium bromide by using Laue method, Potassium Bromide Crystal is attached to the pin hole diaphragm with adhesive transparent tape. The outside edges of the crystal made horizontal by slightly rotating collimator. Then film is clamped at the film holder exactly at the center outline made on it. Then film holder is clamped on experiment rail and mounted it into the experiment chamber of the x-rays apparatus. A spacer 11, 13, 15 millimeter long is made from paper board and shifted the film holder so that the distance between crystal and film is varied. By changing distance between crystal and the film, the area covered in the diagram is changed. The voltage of the x-ray tube is fixed 35 kv and the emission current is kept 1 mA. Then film is exposed to x-rays for 1800 second and started the exposure time was fixed with the SCAN Key. When exposure time is over the film holder with experiment rail is taken out of the experiment chamber. Now the film is carried to dark room for development.

The developing of film is done under dark room conditions. Mixture for developing and fixing chemical is prepared for developing. Three parts of water (300ml) and 1 part of developer(100 ml) is mixed in a plastic tray. For fixing three parts of water (300ml) and 1 part of developer

(100ml) is mixed in another plastic tray. The film is first placed in developer for approximately 2 minutes and 20 seconds. After that the film is washed under running water for 15 sec. Then the film is soaked into the fixer solution for two mints. Later on the film is again washed out under running water for four minutes. In order to obtain clear radiograph of crystal, in next step for analyzing the film, the film is scanned on Epson scanner and the size of the film was selected. The center of the co-ordinate system was set into the laue diagram and the X and Y co-ordinates were determined.

2.1 Theory/Calculations

The following procedure is used formulation of Laue diagram. For laue diagram the co-ordinate system is chosen so that its origin O corresponds to the point of the incoming X-ray on the film. The x-ray is perpendicular to the ray i.e it lies in the ray in the x-y plane as shown in figure 2, the orientation of the z-axis is opposite to the propagation direction of the x-ray. The x-ray penetrate the flat crystal at point k its undiffracted part impinges on the x-ray film at point o the part of x-ray which in scattered at k and which fulfills laue condition leaves the crystal with an angle θ relative to the direction of Primary ray and impinges on the film at point P as shown in figure 2.

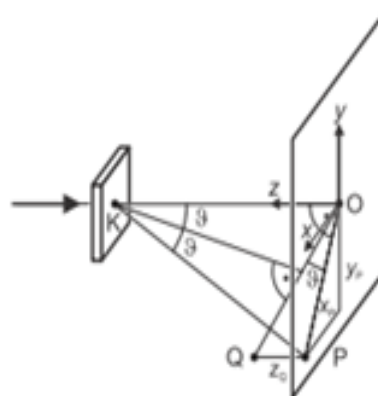
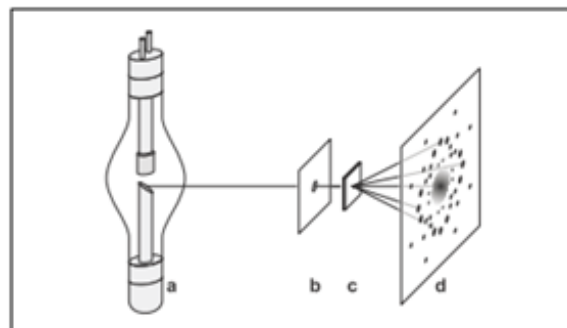


Figure.2: Penetration of x-rays in the crystal and formation of image at the films [19].

Now from the figure 2, we have,

$$\tan 2\theta = \sqrt{x_p^2 + y_p^2} / L$$

Where L is the distance between crystal and x-ray film.

The direction of set of planes (h k l) is given by the perpendicular bisectrix through point k.

Now vector OQ has co-ordinates (XQ , YQ , ZQ), and from diagram tan θ can be written as below,

$$\tan \theta = ZQ / \sqrt{(2XQ)^2 + 2YQ^2}$$

where $X_p = XQ$ and $Y_p = YQ$

Now form figure 2, we can write ZQ as,

$$ZQ = \sqrt{(2XQ)^2 + 2YQ^2 + 2L} - L$$

The crystal used in this experiment is KBr and is parallel to plan (100) so the crystal axis thus coincide with the laboratory co-ordinates system, therefore

$$h : k : l = XQ : YQ : ZQ$$

hkl are miller indices of smallest unmixed triple of integers.

The hkl can also be determined in terms of lattice spacing.

The Bragg angle Θ can be determined as,

$$\theta = \tan^{-1} / \sqrt{2h^2 + 2k^2 + 2l^2}$$

The above equation gives the scattering angle θ with known miller indices (hkl), from which we can also find the lattice constant a_0 and the wave length λ .

In second method, On the basis of Debye scherrer method, powdery sample of potassium Bromide is trans illuminated with mono chromatic x-rays as shown in figure 3. Dry Potassium Bromide is grinded in the mortar and embedded an approximately 0.4mm thick layer between two pieces of transparent adhesive tape. The sample is exposed to x-rays with an exposure time of 1000 s, 1080 s and 1440 s and with a varied distance between the film and x-rays source of 9 mm, 11 mm and 13 mm. In this method, the mono chromatic x-rays fall on a mass of tiny crystals in all orientations, and the diffracted beams of each order h, k, l form a cone. Arcs of the cones are intercepted by a film surrounding the specimen. The x-rays beam passes through a collimator and strikes the sample of powdered about 5-50um diameter of KBr crystals. A film strip which is placed along the camera wall is exposed by the scattered radiation and display the rings formed, after sufficient time and development, the diffraction pattern.

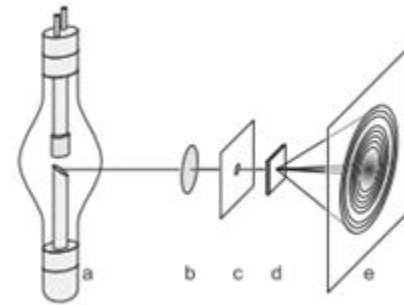


Fig 3: Diffraction of x-rays by potassium bromide powder sample and scattering angle 2Θ [19].

The interference pattern is frozen on an x-ray film. Gently the sample is attached to pinhole diaphragm with tape and is put onto the collimator.

Then x-ray film is clamped at the film holder so that it is centered on the marked area and the entire surface of film is made planer. The film holder is then clamped on experiment rail and mounted into the experiment chamber of x-ray apparatus. By varying the distance between sample and the film changed the area covered in the photograph. The tube voltage is fixed 35 KV and the emission current 1 mA as it is in the first case. After when exposure time is over the film holder with experiment rail is taken out of the experiment chamber. The film is removed from the film holder and is developed it accordingly as discussed in the first case. The complete diffraction patterns formed on the x-rays films is a concentric circle.

Formulation For Analysis of Powder Pattern of Debye Scherer Method

A set of lattice planes in a crystallite leads to a diffraction rejection on the film if it is aligned so that Bragg's law.

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

where n is order and λ is wavelength of x-ray beam, Θ is the Bragg angle relative to primary ray. The angle between diffracted ray and the center of the rings pattern is

2θ. Figure 4 shows the scattering angle 2θ of x-rays by potassium bromide powder sample.

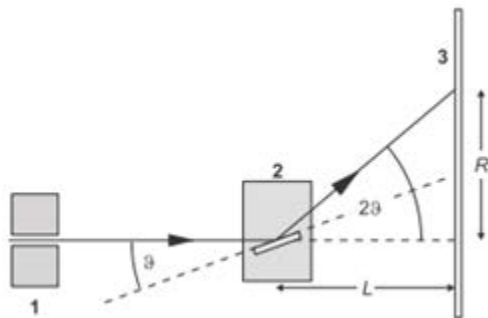


Figure 4: Scattering angle 2θ of x-rays by potassium bromide powder sample [19].

For diagram we can write,

$$\tan 2\theta = R/L$$

Or $R = L \tan 2\theta$

Where R is the distance between the central spot and the diffracted ray and L is the distance between film and the sample. As this is the case of cubic Crystals the lattice spacing can be expressed in the form.

$$d = a_0 / \sqrt{h^2 + k^2 + l^2}$$

where a_0 is lattice constant and hkl are the miller indices for the set of lattice planes we get

$$\sin 2\theta = (\lambda / 2 a_0)^2 [(nh)^2 + (nk)^2 + (nl)^2]$$

Let in the equation we have,

$$F = (\lambda / 2 a_0)^2$$

And $Z = (n.h)^2 + (n.k)^2 + (n.l)^2$

Then we have

$$\sin 2\theta = F.Z$$

Z is always an integer.

For cubic crystals every combination of integers n , h , k and l is allowed.

Diffraction reflection becomes weak for higher order n and greater miller indices hkl.

In case of ionic salt crystal structure there are four alkali and four halogenide atoms, due to which the structure is complex. Due to this fact every unit cell contains four halogenated atoms the co-ordinates for alkali atoms can be written as.

The coordinates of alkali atoms which turns into cubic lattice are given by

$$r_1 = (0, 0, 0) \quad r_2 = (a_0/2, a_0/2, 0)$$

$$r_3 = (a_0/2, 0, a_0/2) \quad r_4 = (0, a_0/2, a_0/2)$$

The co-ordinates of halogenide atoms can be written as,

$$r_5 = (a_0/2, 0, 0)$$

$$r_6 = (0, a_0/2, 0)$$

$$r_7 = (0, 0, a_0/2)$$

$$r_8 = (a_0/2, a_0/2, a_0/2)$$

According Laue Experimental results crystalline substance act as three dimensional diffraction grating for X-rays wavelengths similar to the spacing of planes in a crystal lattice. Typical crystal structure contains several thousand unique reflections whose spatial arrangement is referred to as diffraction pattern. Indices (h,k,l) may be assigned to each reflection, indicating its position within the diffraction pattern. This pattern has a reciprocal Fourier transform relationship to the crystalline lattice and the unit cell in real space. This step is referred to as the solution of crystal structure.

3. RESULTS AND DISCUSSION

In 1912, Max von Laue, a German physicist, discovered that x-rays could be diffracted, or scattered, in an orderly way by the orderly array of atoms in a crystal. That is, crystals can be used as three-dimensional 'diffraction gratings' for x-rays. The phenomenon of x-ray diffraction from crystals is used both to analyze x-rays of unknown wavelength using a crystal whose atomic structure is known, and to determine, using x-rays of known wavelength, the atomic structure of crystals. The atomic structure of crystals is deduced from the directions and intensities of the diffracted x-ray beams. A crystal is built of 'unit cells' repeated regularly in three dimensions. The directions of diffracted x-rays depend on the repeat distances of the unit cells. The strengths of the diffracted beams depend on the arrangement of atoms in each unit cell.

The X-rays have wavelength ranges ($\lambda=10^{-9}\sim 10^{-11}\text{m}$) and are electromagnetic in nature. In this experiment, the potassium bromide (KBr) crystal structure is being discussed using X-rays diffraction by two different techniques, i.e. Max von Laue method and Debye scherrer methods. In these methods, the KBr crystal diffraction pattern is obtained on a detector or x-rays film. After analysis of the diffraction pattern the lattice spacing d and lattice constant a_0 for the potassium bromide crystal are determined.

The diffraction of X-rays take place if the wavelength of X-rays is Comparable with the lattice spacing of the target and is based on the constructive interference and also in accordance to Bragg's law $n\lambda=2dsin\theta$. After the bombardment the X-rays can be scattered in a specular fashion by the atoms of the potassium bromide crystal and undergo in accordance with Laue's method. The diffraction pattern shows the exact features of the inter-planer, parameters, either the crystal structure is FCC or

BCC or SC. Figure 5 shows the diffraction patterns of diffracted rays by the atoms of the sample of KBr crystal sample on x-rays films and figure 6 also shows the diffracted rings form on the x-ray films diffracted by the sample KBr powder sample.

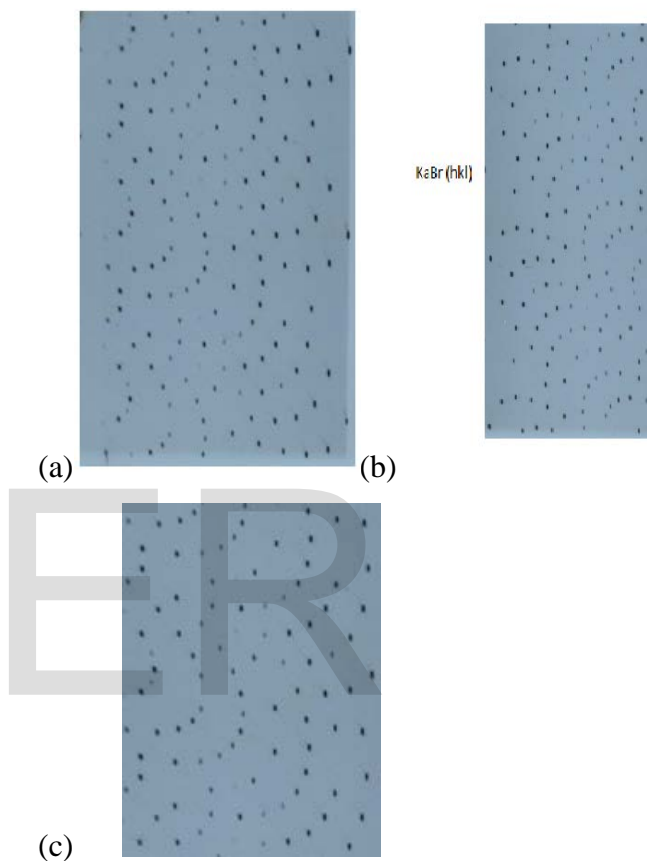
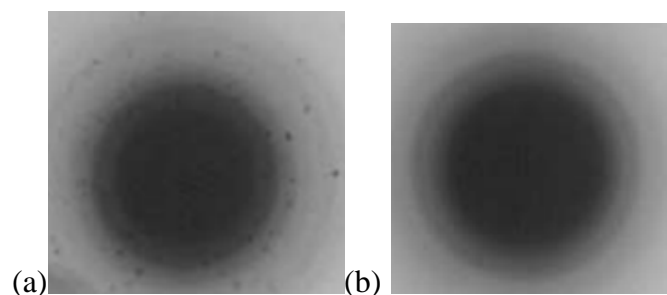


Figure.5: Formation of Laue spots of potassium bromide on x-rays films by the diffraction of monochromatic x-rays at different distances (a) 12 mm (b) 13 mm (c) 15 mm



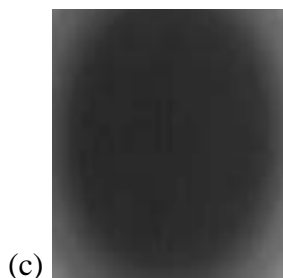


Figure.6: Formation of rings of potassium bromide on x-rays films by the diffraction of monochromatic x-rays at different distances of samples from film holder (a) 9 mm (b) 11 mm (c) 13mm

According Laue Experimental results crystalline substance act as three dimensional diffraction grating for X-rays wavelengths similar to the spacing of planes in a crystal lattice. Typical crystal structure contains several thousand unique reflections whose spatial arrangement is referred to as diffraction pattern. Indices (h,k,l) may be assigned to each reflection, indicating its position within the diffraction pattern. This pattern has a reciprocal Fourier transform relationship to the crystalline lattice and the unit cell in real space. This step is referred to as the solution of crystal structure.

The procedure for evaluating the Laue diagram for crystal sample is already described in experimental setup and procedure. The diffracted images of the KBr crystal is shown in figure 5. The following data tables 1.1 and 1.2 are calculated from the figures 5 (a), (b) and (c) with the following parameters of the LD x-ray apparatus by adjusting the parameters (1) Applied voltage=U=35kv (2) The emission current=I=1mA (3) Exposure time=Δt=1800sec and (4) The distance of the crystal from the film holder=L=12mm

The following data table is observed from the Laue diagram to find the simplest ratios XQ, YQ, ZQ of the coordinates of the Laue diagram for the calculations of Miller indices hkl as already discussed in the section experimental procedure.

No	$\frac{X_Q}{mm}$	$\frac{Y_Q}{mm}$	$\frac{Z_Q}{mm}$	h	k	l
1	6.23	5.96	2.77	3	3	1
	5.83	-6.62	2.86	3	-3	1
	-6.47	5.86	2.83	-3	3	1
	-7.04	3.1	3.1	-3	-3	1
2	16.18	9.06	10.08	4	2	2
	7.90	15.88	9.41	2	4	2
	-17.10	8.44	10.53	-4	2	2
	16.38	-8.64	10.06	4	-4	2
3	8.26	9.15	5.20	4	4	2
	7.88	-8.12	4.49	4	-4	2
	-8.17	8.73	4.93	-4	4	2
	-9.14	-8.24	5.18	-4	-4	2
4	12.34	0.27	6	0	2	2
	0.19	12.58	0	6	2	2
	-11.88	0.16	-6	0	2	2
	0.24	-10.62	0	-6	2	2
5	11.32	3.72	4.91	6	2	2
	4.09	12.73	5.966	2	6	2
	4.22	-13.20	6.33	2	-6	2
6	-11.59	4.18	5.19	-6	2	2
	8.73	5.13	3.70	6	4	2
	5.84	9.19	4.2	4	6	2
	-9.08	5.74	4.1	-6	4	2
6	8.71	-6.01	4	6	-4	2

TABLE 1.1 EXTENDED MILLER INDICIES BY LAUE DIAGRAM COORDINATES FOR SPACING L= 12 MM

To find the lattice spacing d, Bragg's angle φ and diffracted wavelength λ associated with the miller indices hkl calculated in table 1.1 for KBr by Laue Method.

TABLE 1.2 LATTICE PLANE SPACING D, ANGLE Θ AND DIFFRACTED WAVELENGTH λ FOR SPACING L= 12 MM

The following data table 1.3 and 1.4 is calculated from the figures 5 (b) with the following parameters of the LD x-ray apparatus with following parameters (1) Applied voltage= $U=35\text{kv}$ (2) The emission current= $I=1\text{mA}$ (3) Exposure time= $\Delta t=1800\text{sec}$ (4) The distance of the crystal from the film holder= $L=13\text{mm}$

The following data table is observed from the Laue diagram to find the simplest ratios XQ, YQ, ZQ of the coordinates of the Laue diagram for the Miller indices hkl already discussed in the section experimental procedure.

No	$\frac{x_{\theta}}{\text{mm}}$	$\frac{y_{\theta}}{\text{mm}}$	$\frac{z_{\theta}}{\text{mm}}$	H	K	L
1	3.83	3.25	0.936	3	3	1
	4.09	-3.67	1.11	-3	-3	1
	-3.57	3.80	1.006	-3	-3	1
	-4.18	-3.26	1.039	-3	-3	1
2	13.98	0.63	6.10	4	0	2
	-12.80	0.29	5.24	-4	0	2
	-0.13	-14.06	6.14	-0	-4	2
	0.17	13.77	5.93	0	4	2
3	11.22	6.08	5.21	4	2	2
	5.74.-	13.02	6.27	2	4	2
	7.15	12.84	6.62	-2	4	2
	-13.56	5.52	6.57	-4	2	2
4	5.84	5.18	2.16	4	4	2
	6.07	-5.45	2.34	4	-4	2
	-5.92	-5.78	2.40	-4	-4	2
	-4.86	5.21	1.82	-4	4	2
5	7.31	0.24	1.916	6	0	2
	0.16	6.54	1.55	0	6	2
	-0.29	-6.98	1.75	0	-6	2

No	H	K	L	$\frac{d}{p_m}$	Θ	$\frac{\lambda}{p_m}$
1	3	3	1	151.21 p_m	13.23°	69.2 p_m
2	4	2	2	134.5 p_m	25.5°	115.8 p_m
3	4	4	2	109.8 p_m	19.44°	73.08 p_m
4	6	0	2	14.2 p_m	18.26°	65.29 p_m
5	6	2	2	99.3 p_m	17.53°	59.81 p_m
6	6	4	2	88.10 P_M	15.48°	47.02 P_m
6	-5.66	0.29	1.18	-6	0	2
	7.18	2.42	2.04	6	2	2
	2.09	7.14	1.97	2	6	2
	-6.39	-1.94	1.61	-6	-2	2
	-5.84	2.32	1.44	-6	2	2

TABLE 1.3 EXTENDED MILLER INDICES BY LAUE DIAGRAM COORDINATES FOR SPACING L= 13 MM

Spacing of Lattice planes d, Bragg's angel and wavelength associated with set of Lattice planes of KBr

No	H	k	l	$\frac{d}{P_m}$	θ	$\frac{\gamma}{P_m}$
1	3	3	1	151.14 p_m	13.22°	69.1 p_m
2	4	0	2	147.4.2 p_m	26.56°	131.83 p_m
3	4	2	2	134.5 p_m	24.08	109.75 p_m
4	4	4	2	109.83 p_m	19.44°	73.10 p_m
5	6	0	2	104.20 p_m	18.26	65.29 p_m
6	6	2	2	99.35 p_m	17.5	59.75 p_m

TABLE 1.4 LATTICE PLANE SPACING D, ANGLE Θ AND DIFFRACTED WAVELENGTH λ FOR SPACING L= 13 MM

The following data table 1.5 and 1.6 is calculated from the figures 5 (b) with the following parameters of the LD x-ray apparatus with the following parameters, (1) Applied voltage= $U=35\text{kv}$ (2) The emission current= $I=1\text{mA}$ (3) Exposure time= $\Delta t=1800\text{sec}$

(4) The distance of the crystal from the film holder=L=15mm

The following data table is observed from the Laue diagram to find the simplest ratios XQ, YQ, ZQ of the coordinates of the laue diagram for the Miller indices hkl already discussed in the section experimental procedure.

No.	$\frac{XQ}{mm}$	$\frac{YQ}{mm}$	$\frac{ZQ}{mm}$	H	K	L
1	7.1	6.09	2.86	3	3	1
	-5.89	6.44	2.35	-3	3	1
	7.08	-5.78	2.56	3	-3	1
	-5.94	-7.85	2.94	-3	-3	1
2	16.61	9.05	9.41	4	2	2
	7.97	17.69	9.52	2	-4	2
	-9.23	15.86	8.70	-2	4	2
	-7.81	-17.47	9.31	-2	-4	2
3	5.97	1.42	12.06	5	1	1
	1.13	6.39	1.35	1	5	1
	-6.18	1.6	13.01	-5	1	1
	-1.33	6.8	1.52	-1	5	1
4	7.91	9.11	4.24	4	4	2
	9.19	-8.04	4.34	4	-4	2
	-8.28	-7.99	3.90	-4	-4	2
	-9.51	8.05	4.49	-4	4	2
5	11.12	0.3	4.93	6	0	2
	0.41	12.44	4.49	0	6	2

	0.24	-13.06	4.88	0	-6	2
	-	0.18	4.67	-6	0	2
	12.76					
6	11.9	3.81	4.52	6	2	2
	4.27	12.08	4.72	2	6	2
	-3.84	11.89	4.52	-2	6	2
	-	4.34	5.4	-6	2	2
	13.21					

TABLE 1.5 EXTENDED MILLER INDICES BY LAUE DIAGRAM COORDINATES FOR SPACING L= 15 MM

Spacing of lattice plane d, Braggs angle θ and diffracted wavelength λ associated with sett of lattice planes of KBr.

No.	H	K	l	$\frac{d}{mm}$	θ	$\frac{\lambda}{pm}$
1	3	3	1	151pm	13.22o	69.06p m
2	4	2	2	13.45pm	24.08o	109.74 pm
3	4	4	2	109.83pm	19.44o	7.31p m
4	5	1	1	12.68pm	11.08o	48.72p m
5	6	0	2	10.42pm	18.26o	65.28p m
6	6	2	2	99.3pm	17.53o	59.81p m

TABLE 1.6 LATTICE PLANE SPACING D, ANGLE θ AND DIFFRACTED WAVELENGTH λ FOR SPACING L= 15 MM

The Debye sheerer photograph is non-destructive analytical detailed information about the internal lattice of crystalline

subcutaneous including unit cell dimension, bond length, bound angles and details of site ordering.

The equation for reflection i.e. Bragg condition can be satisfied for any set of planes whose spacing is greater than half the wavelength of the x-rays used i.e if $d < \lambda/2$, then $\sin \theta > 1$, which is impossible. This condition sets a limit on how many orders of diffracted waves can be obtained from a given crystal using a x-ray beam of a given wavelength. Since the crystal pattern repeats in three dimensions, forming a three-dimensional diffraction grating, three integers, denoted h, k, l are required to describe the order of the diffracted waves. These three integers, the Miller indices used in crystallography, denote the orientation of the reflecting sheets with respect to the unit cell and the path difference in units of wavelength between identical reflecting sheets. The following table contains the first few allowed sets of h, k, l values:

The following data are taken for Debye scherrer Diagrams given in different tables as below.

No.	H	K	L	$\frac{d}{\text{pm}}$
1	2	0	0	329.83
2	2	0	2	233.26
3	4	0	0	164.9
4	4	2	0	147.5
5	4	2	2	134.67
6	6	0	0	109.94

The x-ray apparatus is adjusted as mentioned below.

(1) Applied voltage =U= 35kv (2) The emission current = I = 1x10⁻³A

(2) Exposure time = $\Delta t = 1000\text{sec}$ (4) The distance of sample from film holder L=13mm

The experimental data is given in the below table for calculation lattice constant a₀ and lattice spacing d.

The procedure for evaluating the debye scherrer diagram for powder sample is already described in experimental setup and procedure section.

The diameter D of the diffracted rings is calculated through a precise Vernier caliper mentioned in able below.

No.	$\frac{D}{\text{mm}}$	θ	Sin2 θ	n	h	k	l	Z	F
1	5.68	6.1 8 θ	0.01158	1	2	0	0	4	0.00290 43
2	841	8.9 7 θ	0.0243	1	2	0	2	8	0.00304
3	12	12. 33 θ	0.04560	1	4	0	0	1 6	0.00285 1
4	14.3 6	14. 46 θ	0.06151	1	4	2	0	2 0	0.00312 1
5	15.6 8	15. 55 θ	0.07186	1	4	2	2	2 4	0.00299 8
6	21.3 5	19. 70 θ	0.113633	1	4	0	0	3 6	0.00315 87

TABLE 1.7 SPLITTING OF SIN² θ VALUES INTO FACTORS F AND Z FOR FINDING LATTICE SPACING D AND LATTICE CONSTANT A.

TABLE 1.8: MILLER INDICES AND LATTICE PLANE SPACING FROM DEBYE SCHERER PHOTOGRAPH

Determination of Lattice constant:-

First of all we find out mean value of factor F ie.

$$F_{av} = 0.003012$$

After that the lattice constant is calculated by the formula as

$$a_0 = \frac{\lambda}{2\sqrt{F}} = \frac{71.1\text{Pm}}{2\sqrt{0.003012}}$$

$$a_0 = \frac{71.1\text{Pm}}{0.10976}$$

$$a_0 = 647.77\text{ pm}$$

The x-ray apparatus is adjusted as mentioned below.

(1) Applied voltage =U= 35kv (2) The emission current = I = 1x10-3A

(2) Exposure time = Δt = 1440sec (4) The distance of sample from film holder L=11mm

The experimental data is given in the below table for calculation lattice constant a0 and lattice spacing d.

The procedure for evaluating the Debye Scherrer diagram for powder sample is already described in experimental setup and procedure section.

The diameter D of the diffracted rings is calculated through a precise Vernier caliper mentioned in able below.

❖ The experimental data is given in the below table:

No.	$\frac{D}{\text{mm}}$	θ	$\frac{\text{Sin}2\theta}{\theta}$	n	h	k	l	z	F
1	7.07	8.91	0.02398	1	2	2	0	8	0.002999
2	8.48	10.47	0.03302	1	2	2	2	12	0.0027531
3	10.59	12.86	0.0495	1	4	0	0	16	0.0030972

4	12.13	14.44	0.062183	1	4	2	0	20	0.0031098
5	13.55	15.82	0.07431	1	4	2	2	24	0.0030998
6	15.66	17.72	0.09263	1	4	4	0	32	0.0028978

TABLE 1.9: SPLITTING OF SIN2 Θ VALUES INTO FACTORS F AND Z

No.	H	K	L	$\frac{d}{\text{pm}}$
1	2	0	0	329.83
2	2	0	2	233.26
3	4	0	0	164.9
4	4	2	0	147.5
5	4	2	2	134.67
6	6	0	0	109.94

TABLE 1.10: MILLER INDICES AND LATTICE PLANE SPACING FROM DEBYE SCHERRER PHOTOGRAPH

Determination of lattice constant:

First of all we find out mean value of factor F ie

$$F_{av} = 0.0029927$$

After the lattice constant is calculated by the formula as

$$a_0 = \frac{\lambda}{2\sqrt{F}} = \frac{71.1\text{Pm}}{2\sqrt{0.0029927}}$$

$$a_0 = \frac{71.1\text{Pm}}{0.10941}$$

$$a_0 = 649.84\text{ pm}$$

The x-ray apparatus is adjusted as mentioned below.

(1) Applied voltage =U= 35kv (2) The emission current = I = 1x10-3A

(2) Exposure time = Δt = 1080 sec (4) The distance of sample from film holder L=9mm

The experimental data is given in the below table for calculation lattice constant a₀ and lattice spacing d.

The procedure for evaluating the debye scherrer diagram for powder sample is already described in experimental setup and procedure section.

The diameter D of the diffracted rings is calculated through a precise Vernier caliper mentioned in table below.

The experimental data is given in the below table:

No.	$\frac{D}{mm}$	θ	$\sin^2 \theta$	N	H	K	L	Z	F
1	4.00 17	6.2 67°	0.0119 1	1	2	0	0	4	0.00297 96
2	5.87 6	9.0 27°	0.0244	1	2	0	2	8	0.00307 76
3	8.67 8	12. 87	0.0496 1	1	4	0	0	16	0.00310 23
4	9.51	13. 93	0.0579 5	1	4	2	0	20	0.00289 86
5	11.1 6	15. 90	0.0750 5	1	4	2	2	24	0.00312 97
6	12.8	17. 7	0.0924 3	1	4	4	0	32	0.00289 98

TABLE 1.11 SPLITTING OF SIN² Θ VALUES INTO FACTORS F AND Z

No.	H	K	L	$\frac{d}{pm}$
1	2	0	0	329.83
2	2	0	2	233.26
3	4	0	0	164.9
4	4	2	0	147.5
5	4	2	2	134.67
6	4	4	0	116.63

TABLE 1.12 MILLER INDICIES AND LATTICE PLANE SPACING FROM DEBYESCHERRER PHOTOGRAPH

Determination of lattice constant:

First of all we find out mean value of factor F ie

$$F_{av} = 0.0030141$$

After the lattice constant is calculated by the formula as

$$a_0 = \frac{\lambda}{2\sqrt{F}} = \frac{71.1Pm}{2\sqrt{0.0030141}}$$

$$a_0 = \frac{71.1Pm}{0.1098}$$

$$a_0 = 647.540 Pm$$

The goal of the present experiments was to determine the lattice constants "a" of monocrystal using by Laue Method and by Debye scherrer method for finding lattice spacig "a" and lattice constant "d" of potassium bromide.

In Laue method, the lattice spacing is findout by using equation 6 in section 3.5 of chapter 3 and experimental date in table 4.1 and 4.2.

Values for glancing angle have been measured as described in equation 13 of section 3.5 of chapter 3 and the values of

Miller indices are taken from table 4.1. Values for n are kept constant having value 1.

Three different Laue diagrams have been made which are shown in figure 3.11. The data is represented in table 4.1 up to 4.6: Only one variable i.e. L is varied. From the obtained results performed on KBr crystals, it has been determined that varying L while keeping exposure time Δt constant, does change the corresponding Miller indices, Bragg angle, wavelengths and the shape of Laue diagrams leading to different lattice spacing a . This actually makes sense because the simplest ratio of coordinates XQ , YQ and ZQ used in the equations for finding Miller indices have different length between film holder and crystal. This can be seen by looking at the values for certain Miller indices, and corresponding Bragg angle, wavelengths, in Tables 1.1 up to 1.6.

From the obtained results that have been performed on KBr crystals, it has been determined that varying L while keeping Δt constant, we get more clear and more spread out Laue spots, taken on the x-ray film. This actually makes sense because the equation that is used to calculate Miller indices does include L . When looking at the three Laue diagrams, it is easy to note that a high symmetric diagram is achieved at a distance of 12 mm.

Each Laue spot has its own symmetry. Some of the spots might not be showing. There are some errors. These errors might be due to not proper alignment of the x-ray film on the film holder. Some of the errors might be due to not long enough exposing the film to x-rays. Some of the future work can consist of performing Back-Reflection Laue method, instead of Transmission Laue method that has been used for this experiment.

In second experiment, Debye-Scherrer images of KBr powder are taken. The sample exposure time Δt and the spacing between sample film holder are varied for

calculation of lattice spacing " d " and lattice constant " a ". The Bragg angles for different h , k , l indices are recorded and compared to the calculated values.

The calculation of lattice spacing " d " and lattice constant " a " are done by using equations 4 of section 3.7 of chapter 3 and data tables are given in figure 4.7 and 4.8. The Debye Scherrer rings are shown in figure 3.15.

The results calculated by Debye Scherrer method are matching with the results of Laue Diagrams Methods.

5. CONCLUSIONS

The experiment consisted of determining the lattice constants of monocrystals using Laue and Debye Scherrer methods. The both experiments are performed to investigate the lattice structure of monocrystals. Using the Laue method the lattice spacing " d " for KBr is 151.21 pm, 155.14 pm and 134 pm for different sample distance $l = 15, 13, 12$ mm respectively and by Debye Scherrer Method, the lattice spacing " d " is 164.9 pm, 147.5 pm and 155 pm for different exposure time and spacing length and lattice constant " a " is 647.77 pm, 647.99 pm, and 649.84 pm for different sample lengths from the x-ray source up to the film holder at a fixed voltage of 35 kv. The calculated results are almost matching with theoretical results.

At the same time it has been noted that if Δt and L are being varied, the shape of the Laue diagrams is going to be changed, with different values for spacing of lattice planes, Bragg angle and wavelength.

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REFERENCES:

- [1] William, D., Callister, Jr. Materials Science and Engineering. An Introduction, 10, 1496, 2006.
- [2] Material science FJ 22, www.materials.ox.ac.uk.

- [3] Mitchell, Brian. S. Introduction to material engineering and science. ISBN: 978-0-471-43623-2 p-39, Canada, Dec 2003
- [4] Gisin, N., Ribordy, G., , W. and Zbinden, H. Quantum cryptography, *Rev. Mod. Phys.*, 74, 145, 2001.
- [5] Motte, L., Billoudet, F. and Pileni M. P. Self-Assembled Monolayer of Nanosized Particles , *J. Phys. Chem.*, 99, 17 1995.
- [6] Murray, C. B., Kagan, C. R. and Bawendi M. G. Self-Organization of CdSe Nanocrystallites into Three-Dimensional, *Science*, 16, 270 1995.
- [7] Brust, M., Schiffrin, D. J., Bethell, D. and Kiely C. Spontaneous ordering of bimodal ensembles of nanoscopic gold clusters, *J. Adv. Mater.*, 7 (1995) 795.
- [8] Niu, W., Zheng, S., Wang, D., Liu, X., Li, H., Han, S., Chen, J., Tang, Z. and Xu, G. Selective synthesis of single-crystalline rhombic dodecahedral, octahedral, and cubic gold nanocrystals, *J. Am. Chem. Soc.*, 131, 697 2008.
- [9] Zhang Q., Xie J., Yang J. and Lee J. Y., " Sequential Cation Exchange Generated Superlattice Nanowires Forming Multiple p-n Heterojunctions" *ACS Nano*, 3 (2008) 139.
- [10] Seo, D., Yoo, C. I., Chung, I. S., Park, S. M., Ryu, S. and Song, H., " Au@Ag Core-Shell Nanocubes with Finely Tuned and Well-Controlled Sizes, Shell Thicknesses, and Optical Properties., *J. Phys. Chem. C*, 112, 2469 2008.
- [11] Nozawa, J. et al., Magnetite 3D Colloidal Crystals Formed in the Early Solar System 4.6 Billion Years Ago., *J. Am. Chem. Soc.*, 133, 8782 2011.
- [12] Xiong, Yong., From electron microscopy to X-ray crystallography: molecular-replacement case studies, *Acta Cryst. D.*, 64, 76, 2008.
- [13] Portales, H., Goubet, N., Sirotki, S., Duval, E., Mermet, A., Albouy, P. A. and Pileni, M. P., Nano-supracrystallinity., *NanoLett.*, 12, 52 2012.
- [14] Goubet, N., Tempra, I., Yang, J., Soavi, G., Polli D., Cerullo, G. and Pileni, M. P., using of Solid Phase in Supracrystals Made of Silver Nanocrystals., *Nanoscale*, 7,32, 2015.
- [15] Martyn, D. Winn et. al., Overview of the CCP4 suite and current developments *Acta Crystallogr D Biol, Crystallography.*, 67, 23 2011.
- [16] Murshudov, G. N., Vagin, A. A. & Dodson, REFMAC5 for the refinement of macromolecular crystal structures., *E. J. Acta Cryst.*, D53, 24, 1997.
- [17] Lebedev, A. A., Vagin, A. A. & Murshudov, G. N., Rotational order-disorder structure of fluorescent protein FP480, *Acta Cryst. D*, 62, 8, 2006.
- [18] Philip, Coppens., Molecular Excited-State Structure by Time-Resolved Pump-Probe X-ray Diffraction. What Is New and What Are the Prospects for Further Progress?, *J. Phys. Chem. Lett.*, 2, 6, 2011.
- [19] www.LD-didactic.de