International Journal of Research
Available at https://edupediapublications.org/journals
e-ISSN: 2348-6848
p-ISSN: 2348-795X
Volume 05 Issue 7
March 2018

Single X ray diffraction: A technique to elucidate the structure of crystal

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Introduction

X-ray crystallography is a technique that enable us to determine the atomic and molecular structure of a crystal. As we are acquainted with the fact that all the substances which surround us are made up of atoms and the three dimensional arrangement of an atom define structure of substance. Various measurement techniques which can elucidate the structure of any material are Nuclear magnetic resonance (NMR), infrared spectroscopy (IR), Mass spectroscopy (MS). But from these spectroscopic techniques only the partial structure can be identified. So Single crystal X-ray diffraction is a technique that can deduce the atomic and molecular structure in precise manner. This technique has enabled the determination of arrangement of atoms within the lattice including unit cell dimensions, bond angle and bond length which cannot be determined by the conventional techniques NMR,IR and Mass spectroscopy.

Fundaments of X- ray diffraction

X-rays were discovered by Wilhelm Conrad Rontgen in 1895[1]. Max von Laue, in 1912, discovered the first X- ray diffraction pattern [2]. Laue used a crystal of copper sulfate as the diffraction grating and awarded Nobel Prize in 1914. The evolution in X-ray crystallography after 1912 had fulfilled earlier expectations as it has not only revealed the way atoms are arranged in matter but can also explain the nature of forces between the atoms and different properties of matter. Further in 1914, William Henry Bragg and William Lawrence Bragg refined the crystal structure analysis with x-ray diffraction and determined atomic structure of a common inorganic substance sodium chloride. In 1915, they were jointly awarded the Nobel Prize.

Need of X-rays

Electromagnetic radiation wavelength ranging from 400 to 700 nm i.e. visible light cannot determine submicroscopic objects. Thus in order to determine molecular structure in atomic level, shorter wavelength is required. Internal structure of crystal

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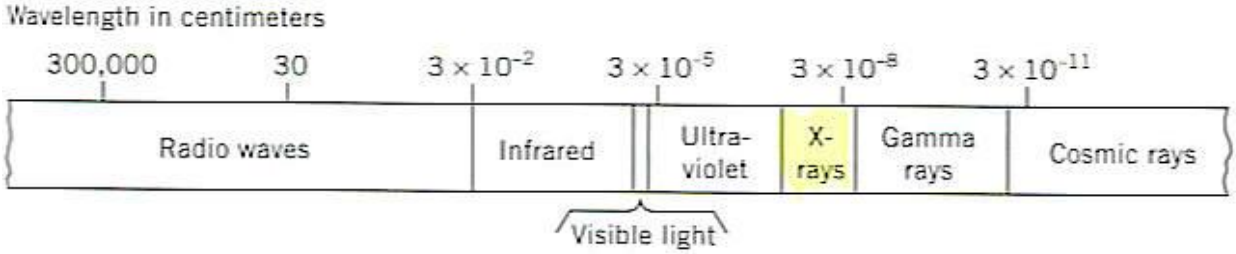
can be explored with radiation whose wavelength is comparable to inter-planar spacing. Most molecules usually have dimensions in the range 1 to 10 Å and it is far too small to be visible in the visible light. The wavelength of the incident light has to be on the same order as the spacing of the atoms. X-rays and neutrons have wave properties and crystal acts as a diffraction grating producing constructive and destructive interference.



Thus X rays in electromagnetic spectrum are in order of the covalent bonds and radius of atoms and this expalains why these are used in crystal structure elucidation.

Crystal Requirement and its preperation

A crystal is a solid material in which atoms are packed in a regular pattern, chacteristic orientation and are characterized by its unit cell. It is three dimensional array of molecules which are held together by various chemical bonds. There is requirement of pure crystal which have high regularity in order to solve even the complicated structure.

In order to adequately model the crystal,quality of crystal should be good. Quality of crystal is characterized by maximum diffraction angle (θ) and is also expressed in "resolution" (Å). The larger the diffraction angle, the greater will be the resolution and the greater number of data is obtained. Crystallization genarally involves two steps: nucleation of a crystallite, followed by growth of that crystallite, ideally to a diffraction-quality crystal [6]. Good quality crystal is grown only from pure compound. Crystal grow by deposition of solute molecules onto surface of pre-existing crystal and are faciliated by environment changing slowly. Several techniques involved in crystal growth

areslow evaporation, layering and vapor diffusion.Crystal growth is affected by solvent, nucleation, time and growth vessel which is to be kept away from mechanical agitation. Always recystallized is used when setting crystal growing attemps since recystallization minimizes the presence of foreign insoluble material . The below figure shows the ordered array of molecules with defined planes.

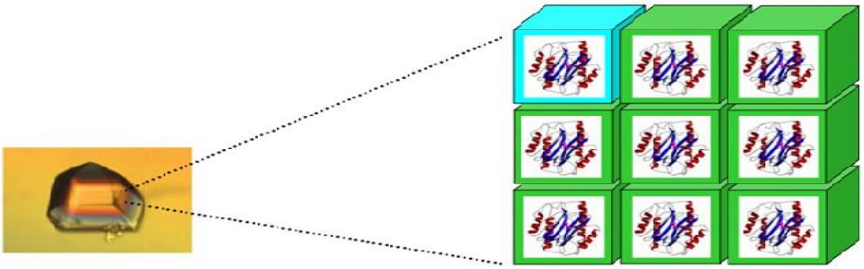
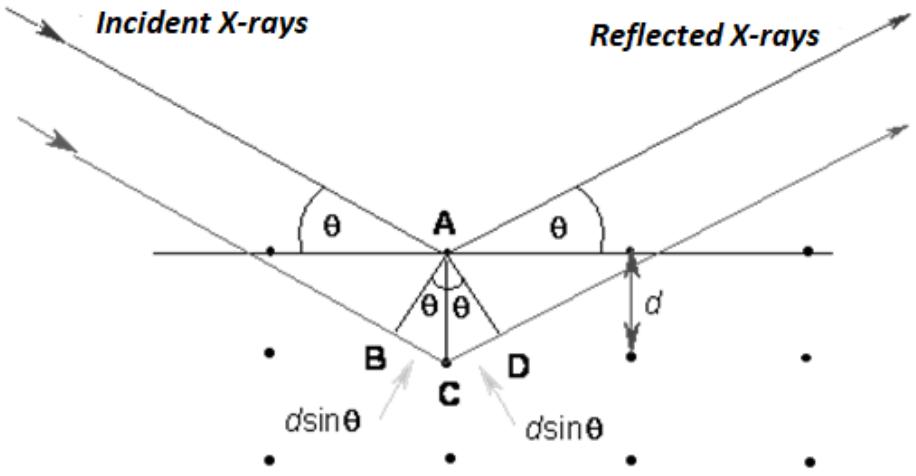


Fig 1: Defined planes of atoms in crysal

Bragg's Law

This law states, if the spacing between the reflecting planes of atoms is 'd' and the glancing angle of the incident X-ray beam is 'Ɵ', then the path difference for waves reflected by successive planes is '2dsinƟ'[3].



Difference in path length = BC + CD

$BC = CD = d_{hkl} \sin\theta_{hkl}$

Difference in path length = $2d_{hkl} \sin\theta_{hkl}$

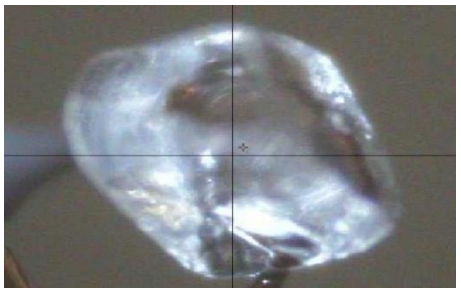
$n\lambda = 2d_{hkl} \sin\theta_{hkl}$ (n = 1, 2, 3, …)

$\lambda = 2d_{hkl} \sin\theta$ **Bragg's Equation**

Diffraction will occur only when Bragg's Law is satisfied Condition for constructive interference. Peak will not be observed if there is destructive interfernce.

Procedure involved

Initially an good quality crystal with adequate size is obtained by several crystallization techniques in order to obtain good results. Then from that sample is selected for diffraction which is optically clear.The crystal is then mounted into the goniometer head. The sample and goniometer head are then affixed to the diffractometer. Then sample centering is done by viewing the sample under an attached microscope or camera and adjusting the axis until the sample is centered under the cross-hairs for all crystal orientations as shown in the figure. After data is collected,the third step is to solve the structure computationally by various software. Atomic coordinates are provided by the final model, from which bond distances and bond angles can be easily calculated. Crystallographic information can be exchanged in CIF format.



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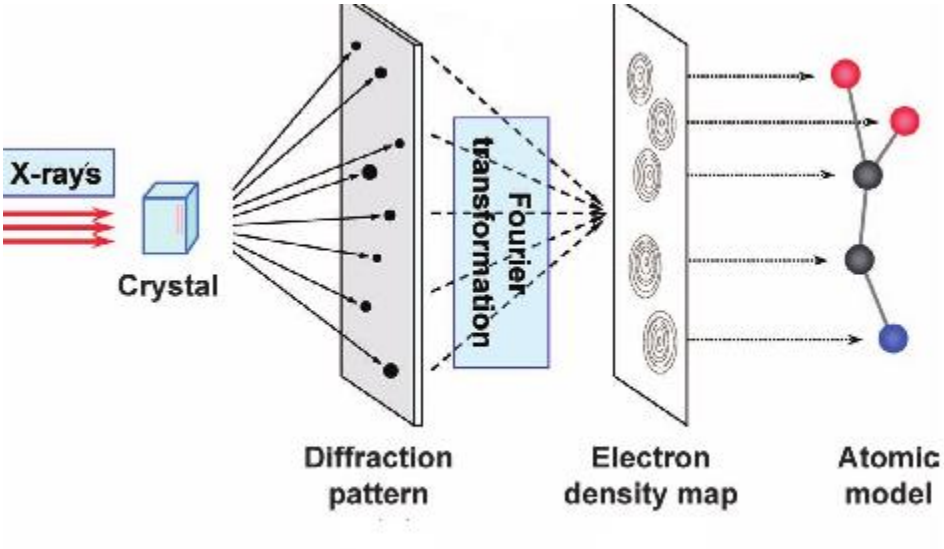

Fig 2: Schematic diagram of technique[7]

Conclusion

Single crystal X-ray Diffraction is an analytical technique which uses interference and diffraction of X- rays and provides detailed information about the internal lattice of crystalline and non-crystalline, at the atomic and molecular level. It also give information of unit cell dimensions, bond lengths and bond angles. Initially it was used just to investigate the structure of minerals but now the technique was expanded to investigate structure of metals, alloys, organic and inorganic substances. Prior to its discovery, crystallographers had no means to measure the internal positions of atoms within crystals but now X-ray diffraction allow crystallographers to demonstrate internal structure of crystal in a very precise manner.

References:

1. W.C. Rontgen: *Nature*, **53**(1896), 274-276

2. W.Friedrich, P. knipping and M. von Laue: *Sitzber. Math.-Phys. Klasse Bayer. Akad. Wiss. Munchen*, (1912), 303

3. Lawerence Bragg, *Scientific American*, Vol 219, No. 1(July 1968)

4. Clegg, William, ed. *Crystal Structure: Principles and Practice.* New York: Oxford Uniersity Press,2001

5. H. Sato and A. Yamano: *The Rigaku journal* ,27(2011), No. 2,22-23

6. Chernov AA (2003)."Protein crystals and their growth". *J. Struct. Biol.* 142 (1):3 21.

7. Kimiko Hasegawa ; *The Rigaku journal*, 28(1),2012

8. Elements of X-ray Diffraction, 3rd Ed., by B.D. Cullity, Addison-Wesley, 2001

9. Y. Mizobe, N. Tohnai, M. Miyata and Y. Hasegawa: *Chem. Commun.*, (2005), 1839-1841

10. X-ray diffraction crystallography." A Dictionary of Earth Sciences. . Encyclopedia.com. (November 17, 2018).