X- ray Crystallography: Overview

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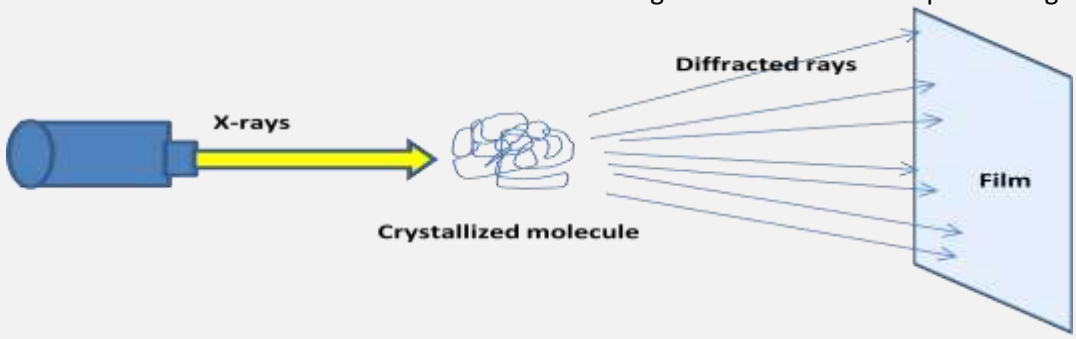
Summary

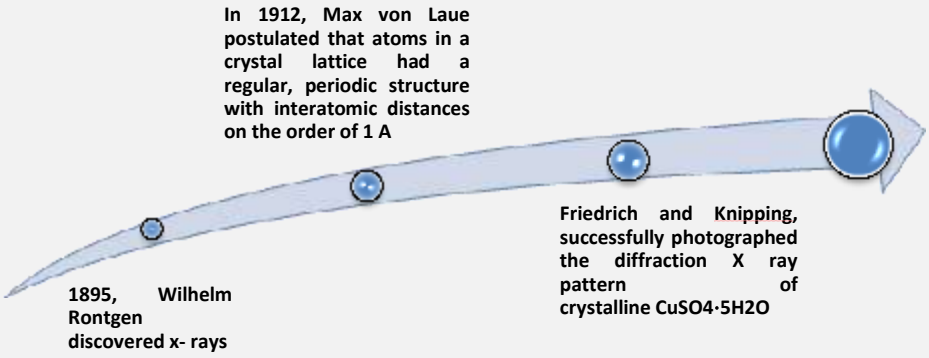
X-ray Crystallography is a scientific method used to determine how the atoms are arranged in a crystalline solid in three dimensional spaces. X- rays are used in the crystallography has wavelength of 0.02 to 100 Å. In X-ray crystallography the crystalline atom cause diffraction of incident X-rays in to different path. This article highlights the overview and application of X-ray crystallography in brief.

Introduction:

What is X-ray Crystallography? X-ray crystallography is scientific technique that relies on the diffracted X-rays passing on crystals. It is not an imaging technique. On the basis of diffraction pattern obtained from X-ray scattering off the periodic assembly of molecules or atoms in the crystal, the electron density can be restructure. Using electron density, the mean positions of the atoms in the crystal, their bonding nature and other information can be determined. The goniometer is used for positioning of the crystal.



History



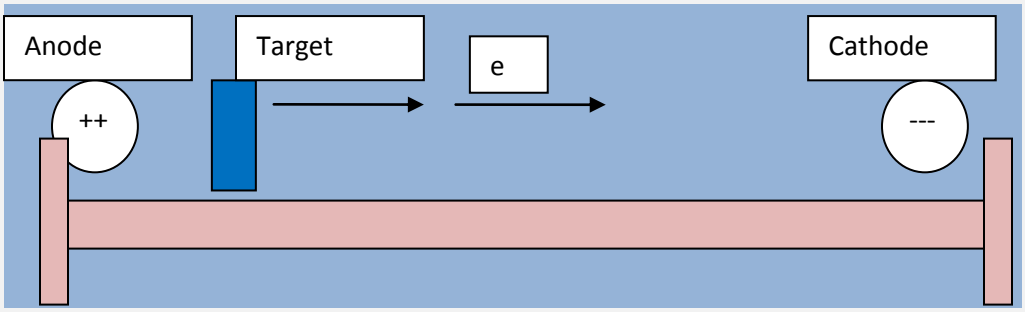
X- rays: X-rays are a form of electromagnetic radiations ranges from 0.01 to 10 nanometers and frequencies range from 30 petahertz to 30 exahertz (3×10^{16} Hz to 3×10^{19} Hz) and energies in the range

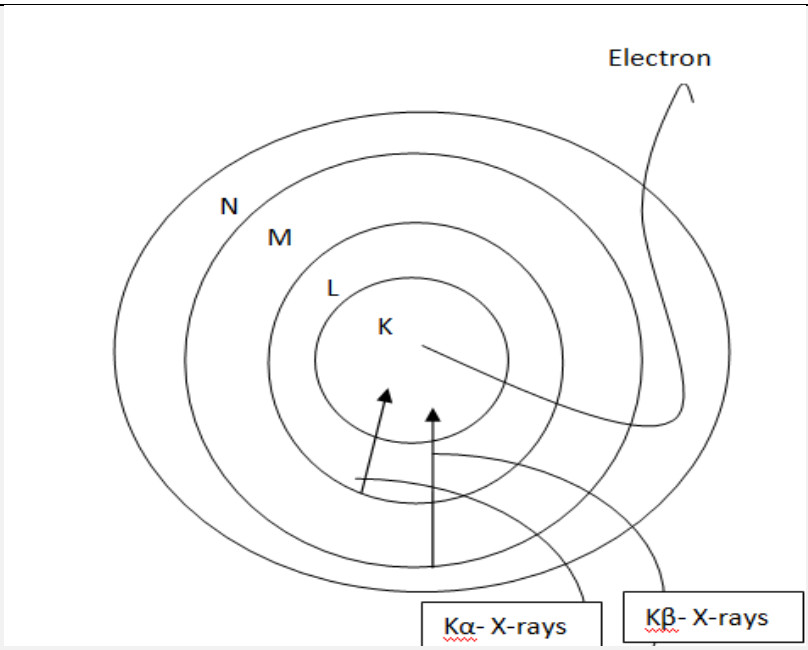
of 100 eV to 100 keV. X-ray wavelengths are shorter and longer than UV rays and gamma rays respectively. High energy X-rays have ability to ionize atoms. This ionizing potential of X-rays can be exploit in cancer treatment to kill malignant cells using radiation therapy.
X-rays are of two types. (1) *hard X-rays* of photon energies (above 5–10 keV, below 0.2–0.1 nm and (2) *soft X-rays* with lower energy.

X-rays have following properties:

- X-rays travel in straight lines path.
- X-rays travel at the speed of light electromagnetic radiation
- X-rays cannot be deflected by electric or magnetic field.
- X-rays are electrically neutral and are Polyenergetic and heterogeneous
- X-rays are highly penetrating, invisible rays.
- X-rays cannot be focused by lens. Can be used to block the Photographic film
- Fluorescent materials glow when X-rays are directed at them.
- Generate chemical and biologic changes by ionization and excitation.
- Release minute amounts of energies while passing through matter.
- X-rays interact with matter bring into being photoelectric and Compton Effect.

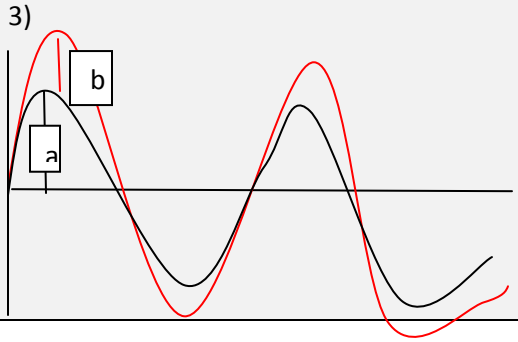
Why Crystallography? Crystallography can unfailingly offer the answer to many questions, related with detailed structure of atoms like bond length, position of atom in the crystal, and distance between atoms etc. Unlike NMR, which is an indirect spectroscopic method, there is no size limitation exists for the molecule or complex to be studied. X ray diffraction discloses the relative positions of the atoms in space to determine the stereochemistry. Bond length and distance between atoms can also be calculated by this technique. X- ray diffraction only locates an atom in space and gives an idea of the structure of crystal, but it cannot make known kind of elements are present. For example crystallographic analysis of the Structure of DNA via X-Ray Diffraction shows that, DNA form helix, twist every 34 angstrom, 10 bases per twist, DNA structure is double helical in which phosphate are on the outside





A high voltage difference is placed between cathode and anode causing electron to move at high velocity from filament to the anode target. Upon striking on the atom in the target, electron removed from the inner shell (K –Shell) resulting in to the jumping of electron from high energy shell (L and M) to the lower energy shell. This electronic transition result in to the generation of X- rays. Kα X-rays have higher intensity than Kβ X- rays. Values of different kinds of transition energies like Kα, Kβ, Lα, Lβ and so on for different elements and electronic transition. Kα emission lines obtained when an electron transitions occurs to the innermost "K" shell (principal quantum number 1) from a 2p orbital of the second or "L" shell (with principal quantum number 2). The line is truly a doublet, with a little different energies depending on spin-orbit interaction energy between the electron spin and the orbital momentum of the 2p orbital. K-alpha is typically by far the strongest X-ray spectral line for an element bombarded with energy enough to source maximally intense X-ray emission.
X- rays when interact with another X- rays such interaction ids called as interference. Interference are of two types.

1) Constructive interference: When both X- rays are in same phase the amplitude of resulting X-ray wave increases.
2) Destructive interference: When two X-rays are in different phase the amplitude of resulting wave decreases.
3)



(a+b) Constructive interference

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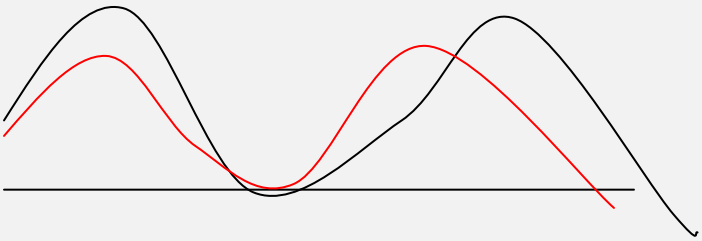
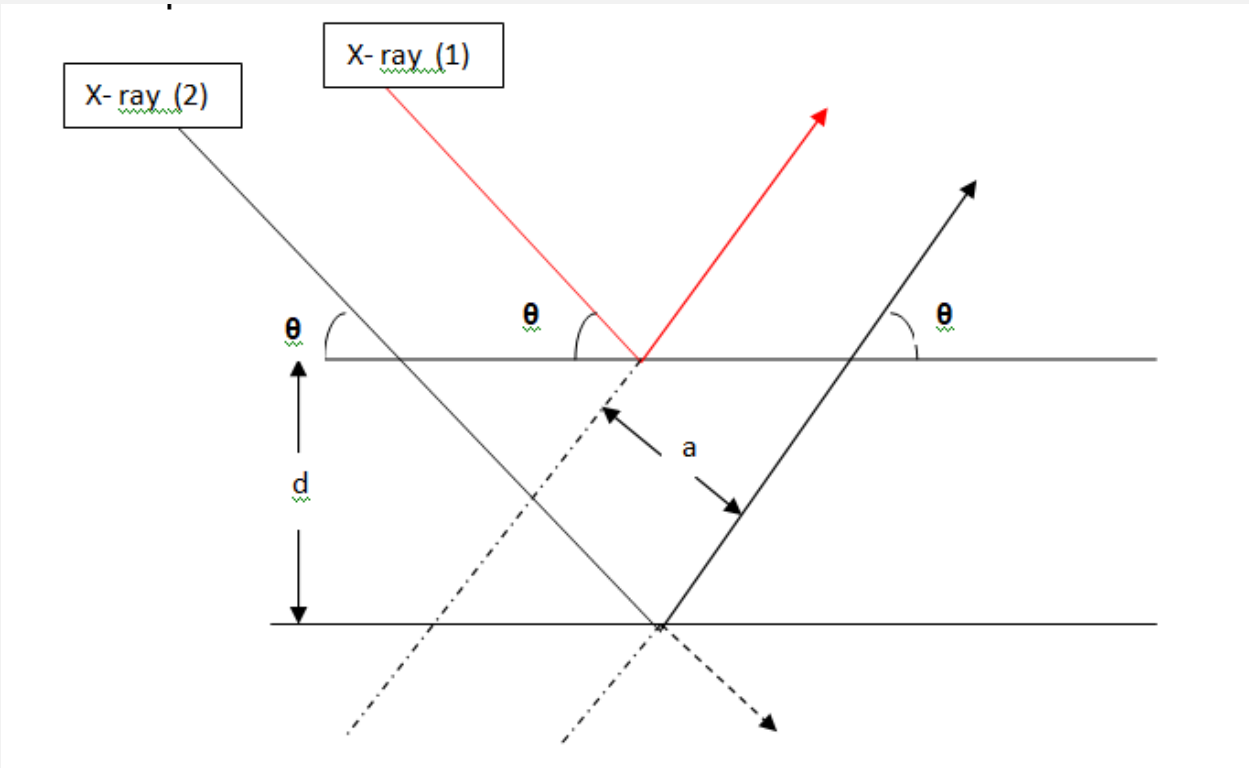




Atom in the crystal interacts with the X-ray in a way that it produces interferences. When two X- rays, X-rays 1 and 2 pass to the atomic plane present at a distance D, X-ray 1 reflect from the upper atomic plane at angel θ which is equal to the angel of incident θ. Similarly X- ray 2 reflect from lower atomic plane at angel θ that means X-ray two travel a distance of 2a more than the X –ray 1. If this distance is equal to the integral no of nλ then X- ray 1 and 2 will be in the same phase and constructive interference will occur.

After diffraction of two waves, phase difference is created in two waves. These waves interact and produce diffraction pattern.

For a constructive interference

$n\lambda = 2a$

$A = d. \sin \theta$

$\text{Sin } \theta = a/d$

This is known as Bragg's equation. $$n\lambda = 2d \sin \theta$$

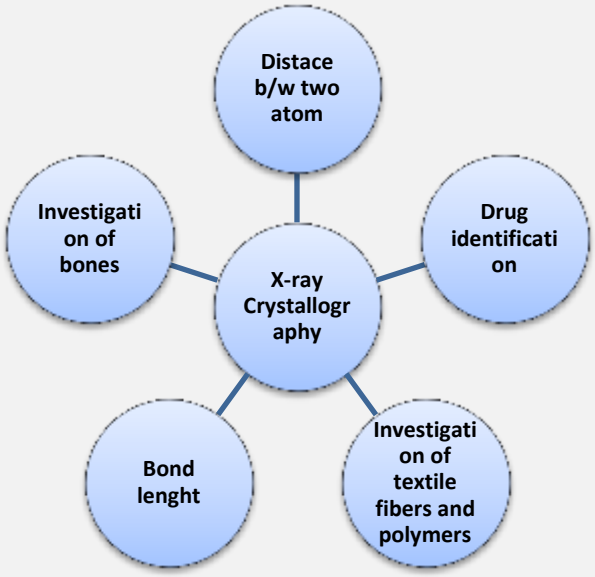
If λ of X- ray going to the crystal is known and angle **θ of diffracted X- ray coming out of the crystal is measured then spacing between atomic planes is calculated by**

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

Steps involved in X- ray crystallography

1. Preparation of the crystal (hanging- drop method)
2. Diffraction pattern is generated
3. Diffraction pattern is converted in to image (furrier transformation)
4. Generation of models of bio molecules
5. Error is removed by refinement

Application



References:

https://www.slideshare.net/dbc9427/basics-of-radiation-and-production-of-x-rays

http://ib.bioninja.com.au/higher-level/topic-7-nucleic-acids/71-dna-structure-and-replic/structure-of-dna.html

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