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X-Ray Crystallography-A Review

Sakshi Bisen¹, Mrudali Kathane², Manisha Bisen³, Devendra Borkar⁴

Dr. T.P. Nimbekar

Shri Laxmanrao Mankar Institute of Pharmacy Amgaon, Gondia. 441902, Maharashtra, India

Abstract:

X-ray crystallography is a widely used scientific technique that helps researchers understand the structure of molecules at the atomic level. The process begins with the creation of a crystal from the substance being studied, which can be a protein, mineral, or any other chemical compound. Crystals are necessary because they allow the X-rays to be diffracted in a regular pattern, which is essential for accurate analysis. Once the crystal is formed, it is exposed to a beam of X-rays. As the X-rays pass through the crystal, they hit the atoms inside and scatter in various directions. This scattering creates a unique pattern of spots on a detector, known as a diffraction pattern. Each spot corresponds to a specific arrangement of atoms in the crystal. To interpret this pattern, scientists use mathematical equations and models to reconstruct the three-dimensional structure of the molecule. This involves complex calculations to determine the position of each atom based on how the X-rays were scattered. The final result is a detailed image of the molecular structure, showing how the atoms are arranged and how they interact with each other. X-ray crystallography has played a critical role in many scientific advancements. For example, it has been essential in drug development by allowing scientists to design medications that can fit precisely into the target molecules, such as enzymes or receptors. It has also contributed to our understanding of biological processes by revealing the structures of important proteins, such as antibodies and hormones.

Key Words: X-Ray, crystal, detectors, diffraction, lattice, bragg's law.

Introduction:

- X-ray crystallography is an analytical method in which the crystalline atoms cause a beam of X-rays to diffract into numerous distinct directions. This method is used to determine the atomic and molecular structure of a crystal.
 - **Terminologies**
 - ❑ **Crystal:** A solid material whose atoms are arranged in a highly ordered, repeating pattern extending in all three spatial dimensions.
 - ❑ **Diffraction:** The bending of waves around obstacles and openings, which in X-ray crystallography refers to the pattern created when X-rays interact with the crystal lattice.[1]
 - ❑ **Bragg's Law:** A fundamental equation that relates the angle of incidence and the wavelength of X-rays to the distance between crystal planes. It is expressed as $n\lambda = 2d \sin(\theta)$, where n is an integer, λ is the wavelength of the X-ray, d is the distance between crystal planes, and θ is the angle of diffraction.
 - ❑ **Unit Cell:** The smallest repeating unit of a crystal lattice that shows the full symmetry of the structure. It defines the dimensions and angles of the crystal lattice.

- ❑ **Lattice:** A three-dimensional arrangement of points representing the positions of atoms, ions, or molecules in a crystal.[2]

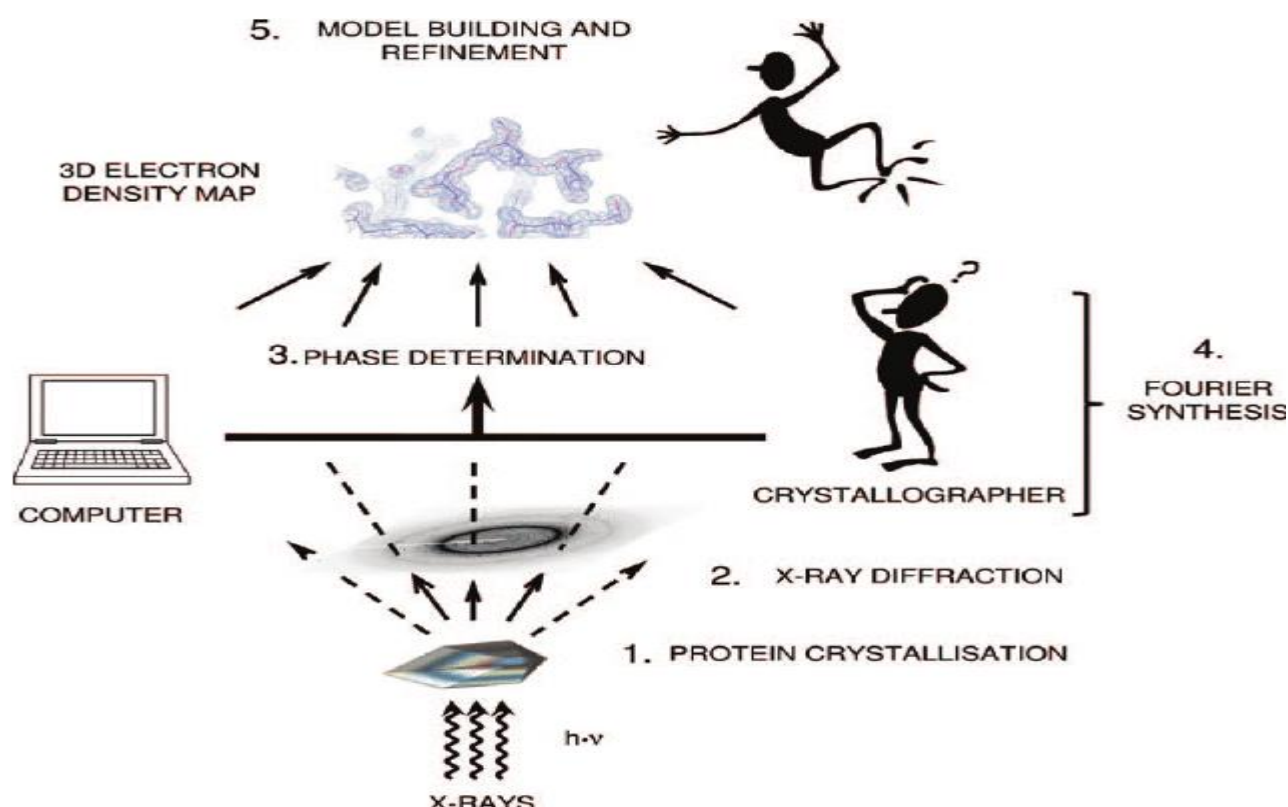


Fig. Representation of X-ray crystallography

History:

- Although crystals have long been admired for their regularity and symmetry, they were not studied scientifically until the 17th century. In his work "Hexagonal Snow as a New Year's Gift" (1611), Johannes Kepler proposed that the hexagonal symmetry of snowflake crystals was due to the volume of spherical water.
- Danish scientist Nikolaus Steno (1669) was the first to investigate the symmetry of crystals. Steno showed that the angles of the faces were the same in all specimens of a given type of crystal. René Just Haüy (1784) discovered that all the faces of a crystal could be described by simple stacking patterns of blocks of the same shape and size. Therefore, in 1839, William H. Miller gave each crystal face a three-digit number, the Miller Index; this number is still used today to describe crystal faces.
- atoms and molecules (Bravais lattice); in the 19th century, Johann Hessel [3], August Bravais, Yevgraf Fedorov, Arthur Schenck and William Barlow (1894). "First there may be a spectral line." He began to write them starting with the letter "K".
- Barlow prepared several crystal structures in the 1880s that were later described by X-ray crystallography; but there was too little data available in the 1880s to consider his models complete. Wilhelm Roentgen discovered X-rays in 1895. Physicists are uncertain of the nature of X-rays, but they think they are waves of electromagnetic radiation. Maxwell's theory of electromagnetic radiation became widely accepted, and Charles Glover Barkley's experiments showed that X-rays exhibit phenomena associated with electromagnetic waves, including transmission lines and spectral lines.[4] There are not only waves, but also particles, so Sommerfeld named the continuous spectrum produced by the disintegration of radioactive particles "bremsstrahlung".

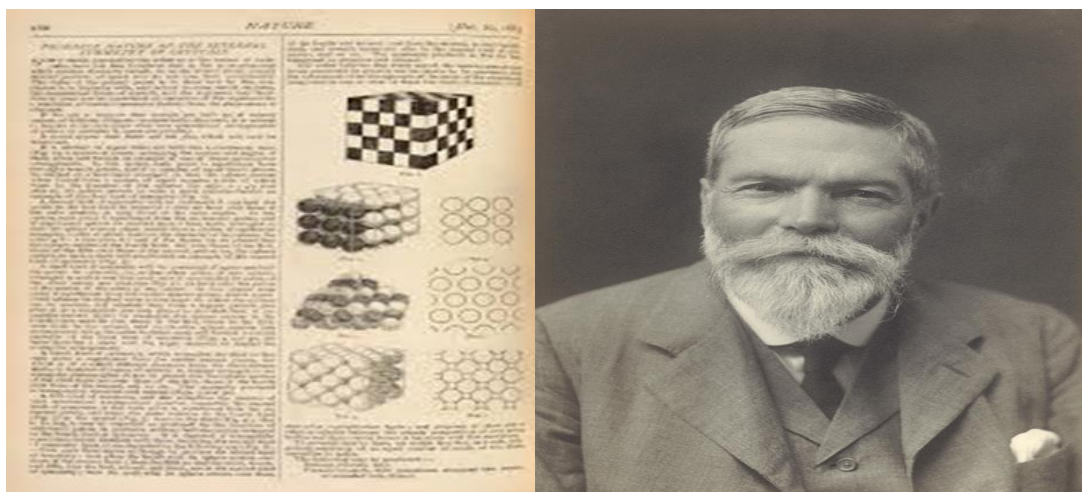


Fig. William Barlow (1894). And his 1st original research paper

- Albert Einstein introduced the concept of the photon in 1905,^[11] but it did not become widely accepted until Arthur Compton confirmed the concept of the photon from electron X-ray scattering in 1922.^[12] The properties of X-rays, such as their ability to ionize gases, led William Henry Bragg to propose in 1907 that X-rays were not electromagnetic radiation. Bragg's hypothesis was disproved and in 1912 Max von Laue's observations of X-ray diffraction confirmed that X-rays were a form of radiation. [5]

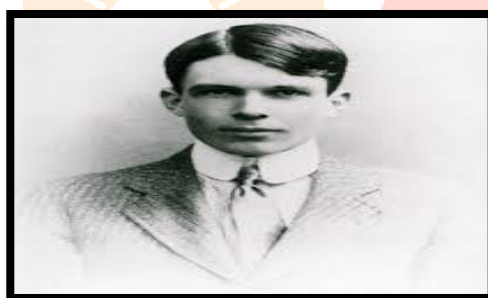


Fig. Lawrence Bragg remains the youngest ever winner of a Nobel prize, aged just 25.

Instrumentation:

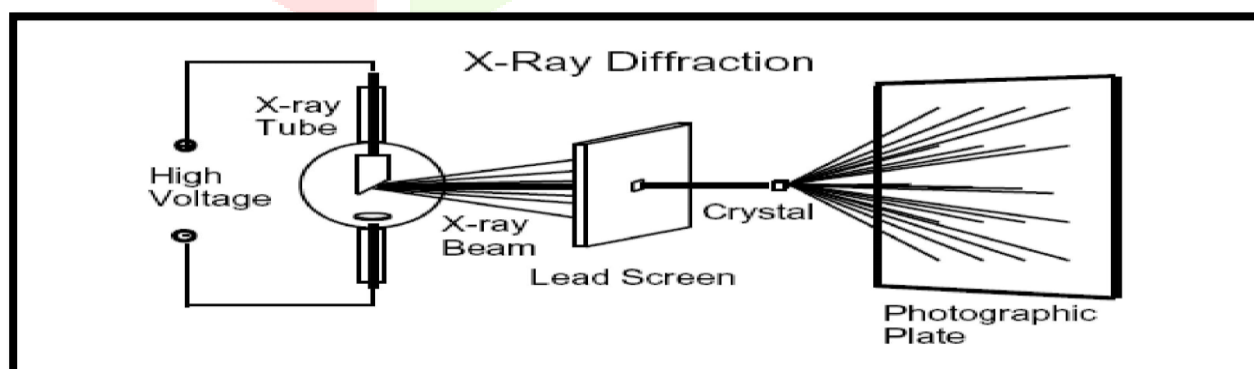


Fig. Instrumentation of XRC

Instrumentation of x ray crystallography involves following components and they are:

- ♣ Source of X-ray (production of X-ray)
- ♣ Collimator
- ♣ Monochromator
- ♣ Detectors

- ♣ **Source of X ray:** X-rays are produced inside the X-ray tube when high energy projectile electrons from the filament interact with the atoms of the anode.

- ✓ Conditions necessary:
 - ☐ Source of electrons
 - ☐ Target (anode)
 - ☐ High potential difference
 - ☐ Cooling facility

There is a cathode which is a filament of tungsten metal heated by a battery to emit the thermionic electrons.

This beam of electrons moves towards anode and attain the kinetic energy and 99% of energy is converted into heat via collision and remaining 0.5- 1% is converted to X-rays via strong coulomb interactions (Bremsstrahlung process).

Generally, the target gets extremely hot in use. This problem has been solved to some extent by cooling the tube with water[6]

Types of X-ray sources are:

- ✓ **Crookes tube (cold cathode tube):**
 - ✓ **Sealed Tubes:**
- PROBLEM:
- A) overheating
 - B) unable to produce high energy

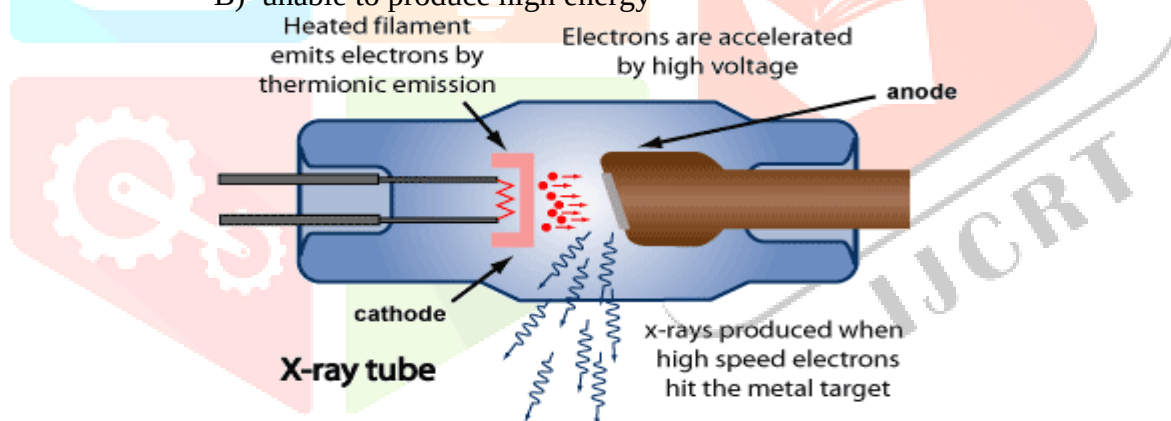


Fig. Representation of X-Ray tube

Besides these two X-Ray tubes there are several more X-ray tubes employed in XRC and they are:

- rotating anode
- synchrotrons
- wigglers
- modern sealed X-Ray tubes[7]

- ♣ **Collimator:** Collimator is a device that narrows a beam of particle or waves.
 - This is to cause the directions of motion to become more aligned to a specific direction.
 - In order to get a narrow, focused beam of x-rays, the x-rays generated by the target material are allowed to pass through a closely packed metal plates separated by a small gap.^[21]
 - The collimator absorbs all the x-rays except the narrow beam that passes between the gaps. Collimators are important because they:
- ✓ Reduce patient exposure:

- ✓ Protect X-ray mirrors

♣ Monochromator:

→ A monochromator is an optical device that transmits a mechanically selectable narrow band of wavelengths chosen from a wider range of wavelengths available at the input.

→ The name is from the Greek root's mono-, "single", and chroma, "color", and the Latin suffix-ator, denoting an agent.

→ Monochromator crystals partially polarize an unpolarized light[8]

Monochromatization can be broadly divided into two classes,

1. Interference Filters

2. Crystal Monochromator: Crystal Monochromators can be again divided into two,

i) Flat crystal Monochromator

ii) curved crystal Monochromator

1. Interference filter:

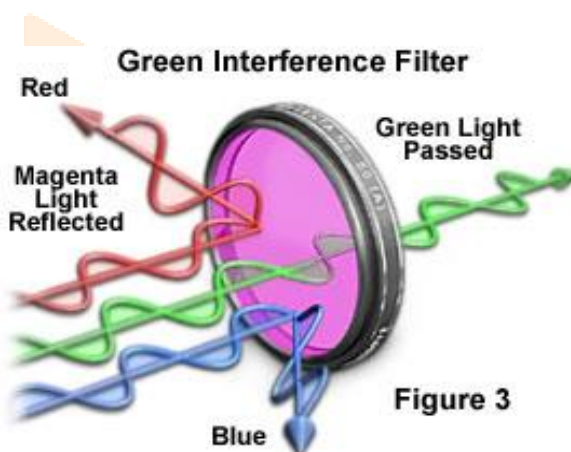


Figure 3

Fig. Green Interference Filter

2. Crystal Monochromator:

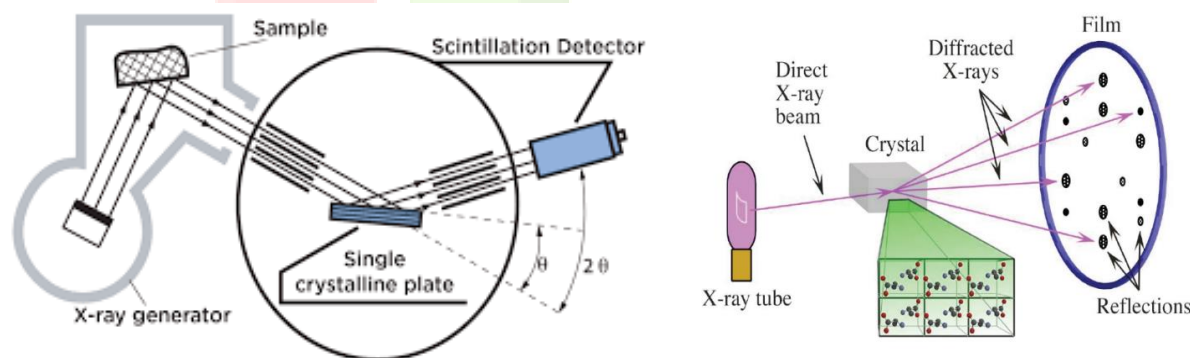


Fig. Crystal Monochromator

♣ Detectors:

- Detectors can be divided into two major categories:

A) Imaging detectors, such as photographic plates and X-ray film (photographic film) now mostly replaced by various digitizing devices like image plates or flat panel detectors.

B) dose measurement devices called counter methods.[9]

- The X-ray intensities can be measured and recorded either by,

1) Photographic methods

2) Counter methods

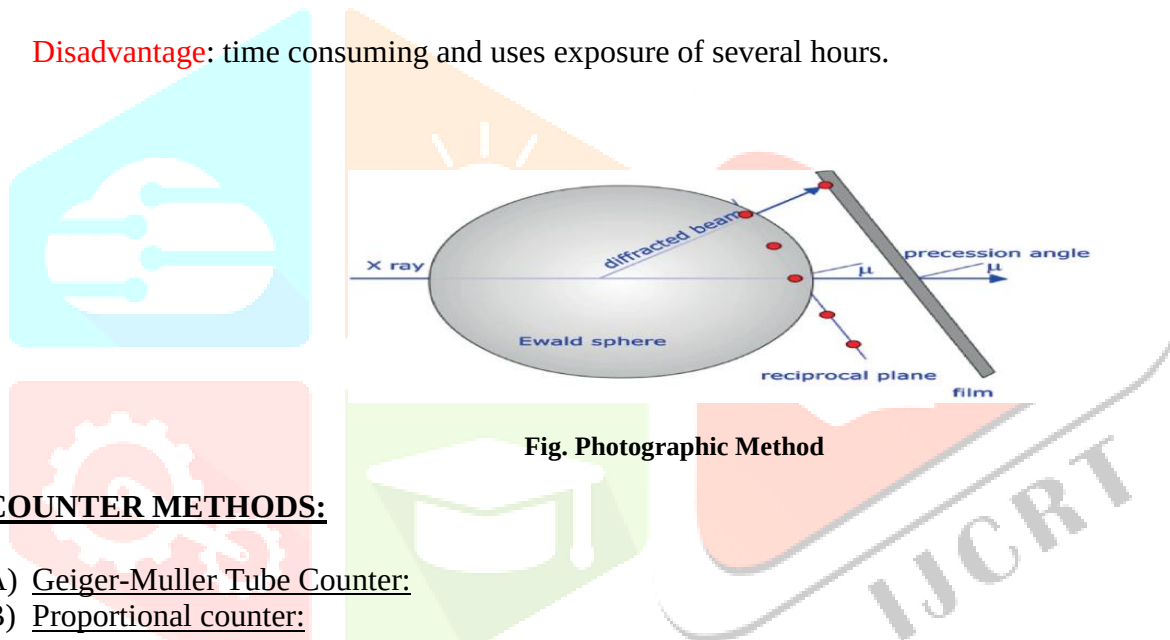
- Geiger Muller tube counter
- Proportional counter
- Scintillation detector
- Semiconductor detectors

Both these types of methods depend upon ability of x-rays to ionize matter and differ only in the subsequent fate of electrons produced by the ionizing process. [10]

1) PHOTOGRAPHIC METHOD:

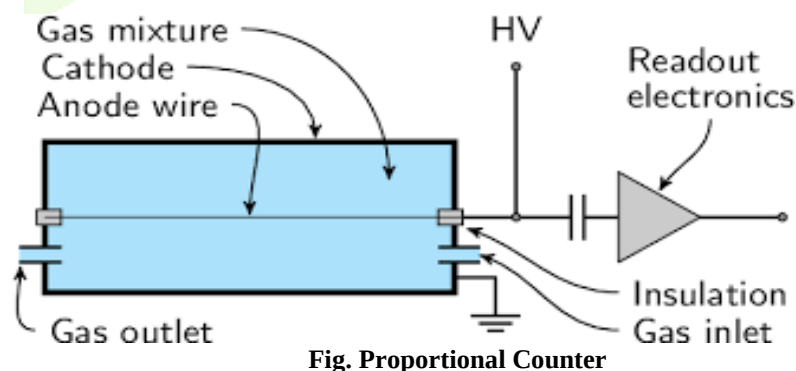
- To record the position and intensity of x-ray beam a plane or cylindrical film is used.

Disadvantage: time consuming and uses exposure of several hours.



2) COUNTER METHODS:

- Geiger-Muller Tube Counter:
- Proportional counter:



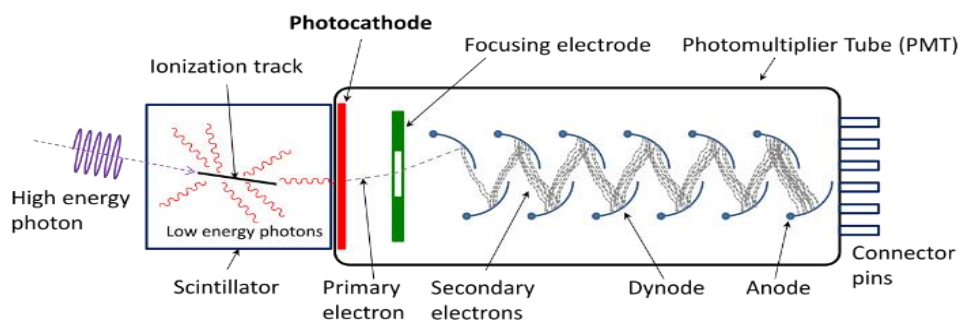
C) Scintillation detector:

Fig. Scintillation Detector

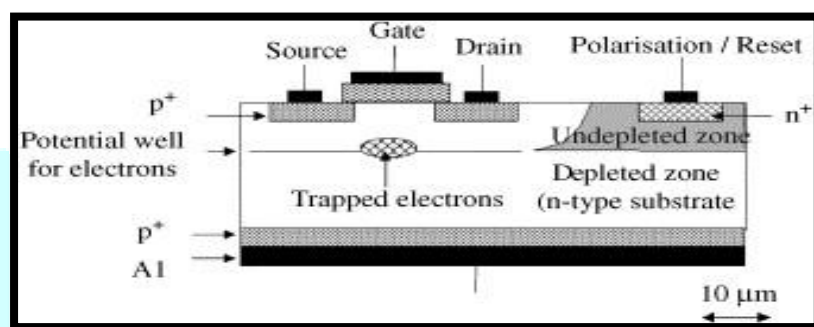
D) Semi-conductor detector:

Fig. Semi-conductor Detector [11]

Applications:

1. **Drug Design:** Understanding drug binding mechanisms to optimize and design pharmaceuticals that target specific biological molecules, such as enzymes and receptors.
2. **Protein Structure Determination:** Reveals atomic-level details of proteins, aiding in comprehending their function and interaction with other molecules.
3. **Material Science:** Investigates the atomic arrangement in materials, helping to develop new materials with enhanced properties, like superconductors or catalysts.
4. **Molecular Biology:** Determines the structure of macromolecules, providing insights into molecular processes such as DNA replication and enzyme action.
5. **Nanotechnology:** Helps to characterize the crystal structure of nanoparticles, supporting the design of nanoscale devices. [12]
6. **Environmental Science:** Analyzes pollutants or contaminants at the molecular level, providing insights for environmental remediation strategies.
7. **Forensic Science:** Examines crystalline substances like drugs or explosives in forensic investigations to identify unknown samples.
8. **Chemistry:** Identifies the structure of organic and inorganic compounds, enabling chemists to determine molecular arrangements and bonding.
9. **Crystallography in Geology:** Studies minerals to understand their crystalline structure and formation processes.
10. **Agriculture:** Supports the development of pesticides by identifying active sites in target pests and pathogens.[13]

Conclusion:

X-ray crystallography is a method used to find out the arrangement of atoms in a crystal. Here's a detailed conclusion in simple language. When scientists want to know what a molecule looks like, they can use X-ray crystallography. They take a crystal of the substance and shine X-rays on it. The X-rays bounce off the atoms in the crystal and create a pattern. By studying this pattern, scientists can figure out where each atom is located in the molecule. This technique is very important because it helps us understand how molecules work and interact with each other. For example, knowing the structure of proteins can help in drug design, as scientists can see how a drug might fit into a protein and affect its function. In summary, X-ray crystallography is a key tool that allows scientists to visualize the tiny building blocks of matter, leading to discoveries in medicine, biology, and materials science. It helps us unlock the secrets of how life works at the molecular level.

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