X-Ray Diffraction

X-Ray Diffraction (XRD): Theory

What is X-Ray Diffraction?

- X-ray diffraction (XRD) is a powerful analytical technique used to determine the crystalline structure of materials.
- It works by directing X-rays at a material and measuring how these rays are scattered or diffracted by the atoms within the crystal lattice.
- When X-rays interact with a periodic arrangement of atoms in a solid, they can undergo constructive interference at specific angles, creating a diffraction pattern.
- These patterns are characteristic of the atomic arrangement within the material.

Nature of X-Rays:

- X-rays are a form of electromagnetic radiation with wavelengths typically in the range of 0.01 to 10 nanometers.
- Their wavelengths are on the order of interatomic distances (about 0.1–0.3 nm), making them suitable for probing the arrangement of atoms in a crystal.

Basic Principle

- When X-rays strike a crystal:
    1. They are **scattered by the electrons** of the atoms in the material.
    2. In certain directions, these scattered waves **interfere constructively**, forming observable diffraction maxima.
    3. These directions correspond to specific **atomic planes** within the crystal that satisfy **Bragg's Law**.
    4. The angles and intensities of the diffracted beams provide a **map of the atomic structure**.

Basic Theory: Bragg's Law

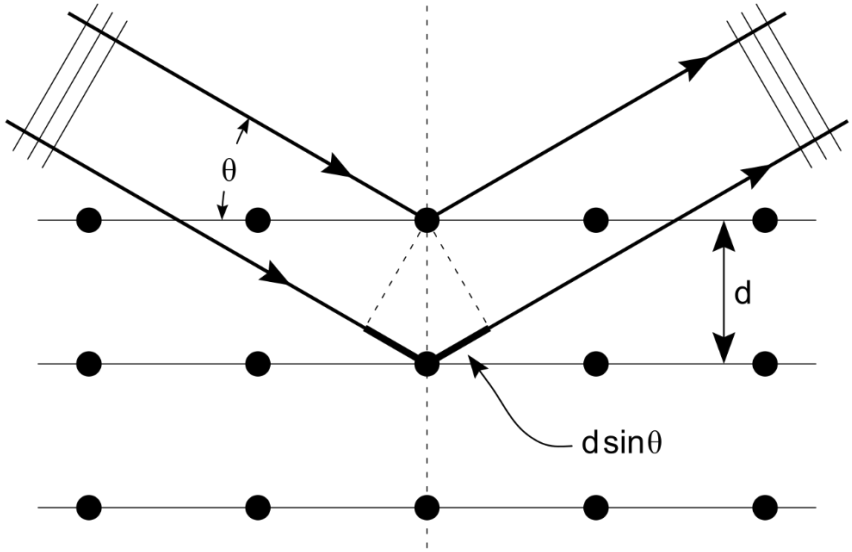
- The fundamental principle of XRD is given by Bragg's Law, which describes the condition for constructive interference of X-rays diffracted by crystal planes.

Bragg's Law:

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

- Where:
    - n= order of reflection (usually 1),
    - λ= wavelength of incident X-ray,
    - d= interplanar spacing (distance between crystal planes),

- θ= angle of incidence (glancing angle),
- $2d\sin\ \theta$= path difference between reflected waves.



Explanation:

- When X-rays strike a crystal, some are reflected from the surface, while others penetrate and are reflected from deeper planes.
- If the path difference between these waves is an integer multiple of the wavelength, they interfere constructively and form a diffraction peak.
- This condition (Bragg's Law) allows the calculation of the spacing between crystal planes, d, if $\lambda$and $\theta$are known.

X-Ray Interaction with Crystals:

- Crystals are made up of a **periodic arrangement of atoms** forming **lattices**.
- X-rays interact with the **electron clouds** of atoms.
- As they scatter from multiple planes of atoms, the interference between these scattered waves gives rise to **diffracted beams**.
- Only certain orientations and spacings lead to detectable diffracted beams, depending on Bragg's condition.

X-Ray Diffraction: Applications

A. Polymorphism Identification

- Crucial in preformulation studies.
- Helps detect different crystalline forms which may vary in therapeutic effectiveness.

B. Crystallinity and Amorphous Content

- Determines the degree of crystallinity in formulations.
- Assesses amorphous content, which affects solubility and dissolution rate.

C. Stability Studies

- Monitors solid-state stability during stress testing (e.g., heat, humidity).
- Detects phase transitions or degradation.

D. Quality Control

- Ensures batch-to-batch consistency.
- Detects contaminants or adulterants.

E. API and Excipient Characterization

- Confirms the identity of Active Pharmaceutical Ingredients (APIs).
- Evaluates excipient compatibility and purity.

F. Particle Size Estimation

- Using peak broadening (Scherrer Equation), nanocrystalline domains or small particle size can be estimated.

Reciprocal Lattice Concept

Introduction:

- The reciprocal lattice is a mathematical construct used to simplify and understand diffraction phenomena in crystallography.
- It is not a real lattice in physical space but an abstract lattice used to represent the periodicity of a crystal in reciprocal (or momentum) space.
- The concept of the reciprocal lattice is central to the theory of diffraction, including X-ray diffraction, electron diffraction, and neutron diffraction.

Real vs. Reciprocal Lattice:

- The **real lattice** describes the periodic arrangement of atoms in real 3D space using vectors $\vec{a}, \vec{b}, \vec{c}$.
- The **reciprocal lattice** is defined by vectors $\vec{a}^*, \vec{b}^*, \vec{c}^*$, which are mathematically related to the real lattice vectors.

Definition:

- The reciprocal lattice vectors are defined as:

$$\vec{a}^* = \frac{\vec{b} \times \vec{c}}{V}, \vec{b}^* = \frac{\vec{c} \times \vec{a}}{V}, \vec{c}^* = \frac{\vec{a} \times \vec{b}}{V}$$

- Where V is the volume of the unit cell:

$$V = \vec{a} \cdot (\vec{b} \times \vec{c})$$

- Each point in the reciprocal lattice corresponds to a set of crystal planes in real space, identified by **Miller indices (hkl)**.

Importance in X-Ray Diffraction:

- X-ray diffraction is most easily explained using reciprocal lattice theory.
- The condition for diffraction is that the difference between the incident and scattered wavevectors must equal a reciprocal lattice vector.
- This condition is called the Laue condition, and is equivalent to Bragg's Law.

Ewald Sphere Construction:

- The Ewald construction is a geometric way to visualize diffraction conditions in reciprocal space.
- It represents the incident X-ray wavevector and the reciprocal lattice.
- When a reciprocal lattice point lies on the surface of the Ewald sphere, the Bragg condition is satisfied, and diffraction occurs.

Advantages of Reciprocal Lattice Concept:

- Simplifies complex diffraction geometry into vector equations.
- Allows clear visualization of diffraction conditions.
- Facilitates indexing of diffraction patterns.
- Essential for understanding diffraction in 2D and 3D crystals, electron microscopy, and surface science.

Diffraction Patterns

What is a Diffraction Pattern?

- A diffraction pattern is the result of X-rays interacting with the periodic atomic arrangement in a crystal. When constructive interference occurs (according to Bragg's law or Laue conditions), a set of bright spots or rings appears on a detector.
- These patterns are a map of the reciprocal lattice and directly relate to the internal structure of the material.

Types of Diffraction Patterns

1. Single-Crystal Diffraction Pattern

- Shows discrete **spots** corresponding to reciprocal lattice points.
- Each spot represents a specific set of crystal planes.
- Used to determine full 3D atomic structure of a material.
- Requires a well-oriented single crystal.

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