**X-Ray Diffraction**

**Physical Chemistry II Lab**

**CHM4111L**

**Dr. Clark**

**1. Relation to Lecture**

The physics of diffraction, especially when it comes to X-rays, has many connections to the quantum mechanical principles that underlie chemistry. Diffraction is fundamentally a phenomenon governed by constructive and destructive interference of waves, and it can be connected to the famous “double-slit” experiment by Young by considering a periodic array of slits. Connections to CHM4411 can best be drawn with the following realization: you can do this type of diffraction experiment with electrons instead of X-rays! Just like the Davison and Germer experiment, the wave-like properties of electrons can also lead to electron interference when impinging on a periodic lattice. Electron diffraction is currently a state-of-the-art technique for determining the structures for difficult to crystallize biomolecules like enzymes caught in the act of chemistry.

**2. Introduction / Theory**

*Part I – Simple Diffraction*

In this experiment, we explore the principles of diffraction using laser pointers and a diffraction grating, which are foundational to understanding the wave-like behavior of light. Diffraction occurs when light encounters an obstacle or slit, causing the light to spread out and form a pattern of bright and dark spots. This phenomenon can be analyzed through quantum mechanics, which describes light as exhibiting both particle-like and wave-like properties. The wave nature of light, where photons (the particle aspect) exhibit interference patterns due to their wave properties, is central to diffraction. By carefully measuring the positions of these bright spots, or diffraction maxima, we can gain insight into the properties of the light and the grating through which it passes.

The procedure involves directing a laser beam perpendicular to a diffraction grating and measuring the distance between the diffraction maxima on a nearby screen. These measurements are repeated at different distances between the grating and the screen to obtain more data. With this information, the d-spacing, or the distance between the slits in the diffraction grating, can be calculated. This d-spacing is then used to determine the wavelength of a second laser pointer by performing a similar series of measurements. The relationship between these parameters is described by the diffraction equation:

Where:

* 𝑛 is an integer (the order of reflection)
* 𝜆 is the wavelength of the laser
* 𝑑 is the interplanar spacing between adjacent atomic planes in the crystal
* 𝜃 is the angle of incidence at which constructive interference occurs.

This experiment not only allows us to calculate physical properties such as the wavelength of light but also provides a deeper understanding of the wave nature of light through the analysis of diffraction patterns, which align with quantum mechanics' description of photons as quantum objects exhibiting interference due to their wave-like nature.

*Part II – X-Ray Diffraction*

X-ray powder diffraction (XRD) is a powerful technique that exploits the wave-particle duality of X-rays to investigate the atomic structure of crystalline materials. In quantum mechanics, particles such as photons, which make up X-rays, exhibit both particle-like and wave-like behavior. When a beam of X-rays interacts with the periodic arrangement of atoms in a crystalline solid, the wave-like nature of X-rays results in constructive and destructive interference, forming a diffraction pattern. This pattern is directly related to the arrangement and spacing of atoms in the crystal lattice.

The principles behind XRD can be understood using Bragg's Law, which describes how constructive interference occurs when X-rays are scattered by the atomic planes in a crystal. According to quantum mechanics, the energy and wavelength of X-rays are quantized, and these X-rays will only diffract at specific angles where the path difference between waves scattered by adjacent planes is an integer multiple of the wavelength. Bragg's Law is given by:

Where:

* 𝑛 is an integer (the order of reflection)
* 𝜆 is the wavelength of the X-rays
* 𝑑 is the interplanar spacing between adjacent atomic planes in the crystal
* 𝜃 is the angle of incidence at which constructive interference occurs.

X-ray powder diffraction leverages this principle to probe the crystal structure. As the X-rays interact with the electron clouds surrounding the atoms, they scatter in various directions. When a crystalline powder is exposed to X-rays, the random orientation of the crystals results in diffraction from multiple planes at different angles, forming a pattern of rings that can be detected. Each diffraction peak corresponds to a specific d-spacing, providing information about the crystal's lattice dimensions, symmetry, and atomic arrangement.

By analyzing the diffraction pattern using quantum principles, scientists can determine the material's crystal structure, phase composition, and even defects within the lattice. XRD serves as a crucial tool for understanding the quantum-scale properties of materials and how they relate to macroscopic physical and chemical behavior. In this experiment, students will use X-ray powder diffraction (XRD) to analyze known salts and identify the components of an unknown salt mixture. By comparing the diffraction patterns of the unknown sample to those of known salts, students will determine the composition of the mixture. Additionally, students will calculate the percentage of each salt present in the mixture using peak intensity ratios from the diffraction data. This experiment highlights the power of quantum mechanics in explaining the behavior of light and X-rays and their practical applications in material science.

**3. Materials and Equipment**

**Part I**

* Laser Pointers
* Diffraction Grating
* Clip to hold grating
* Tape Measure
* Goggles

**Part II**

* X-ray powder diffractometer
* Known salts (e.g., NaCl, KCl, CaCO₃, MgSO₄)
* Unknown salt mixture (containing a combination of the known salts)
* Sample holders
* Mortar and pestle (for grinding powders

**4. Safety Precautions**

* **Caution:** Never look directly at the laser beam. When making measurements on the diffraction maxima, avoid having your eyes level with the laser.
* **Hazard:** X-ray radiation can be harmful, leading to tissue damage or radiation burns.

**5. Experimental Procedure**

**Procedure:**

*Part I – Simple Diffraction*

Set up the laser pointer (650nm) to shine its beam perpendicular onto the diffraction grating provided, so that a diffraction pattern is generated on a nearby wall or screen. Measure the distance from the wall to the grating (*L*). Measure the distance from the central bright spot and the first-order maximum (*n=1*) on one side. This distance is *x*. Perform your measurements at three different distances between screen and grating. You may wish to dim the lights in the room to aid in your measurements. Calculate the d-spacing between the slits in the grating. First you will need to find θ. **\*HINT\* Be careful about your units in these calculations.**

Set up the other laser pointer to shine its beam perpendicularly onto the grating. Measure the distance from the screen to the grating as well as the distance between diffraction maxima as before.

Using the d-spacing from the first measurements, calculate the wavelength (λ2) of the new laser pointer in nm.

*Part II – X-Ray Diffraction*

**Calibration and Analysis of Known Salts in Unknown Mixtures**

1. **Sample Preparation**:
   * Take a small amount (~ 0.5-1 g) of each unknown sample and grind into a fine powder using a mortar and pestle. IMPORTANT: If you do not grind the sample finely, it will not stay in the sample holder and you will not get any data.
   * Place a small amount of each sample into individually labeled centrifuge tubes.
   * You will now be taken to CSL to conduct your sample analysis.
2. **XRD Measurement**:
   * Gently load a small amount of each sample into the XRD sample holder, ensuring an even and smooth surface for the XRD measurement.
   * Place the sample holders in the XRD instrument. Record in your notebook the numbered spot each sample is in.
   * Set up the X-ray powder diffractometer with appropriate parameters:
     + Start: 10
     + Stop: 80
     + Step: 0.01
     + Speed: 8/min
     + Option → 2theta: check ; beta: 1 ; speed: 10
   * Make sure the sample is labeled correctly in the software for each sample holder and will save to your folder, Data → Clark → CHM4411L → “YourName”.
   * Perform the XRD scan for each salt mixture and record the diffraction patterns.
3. **Data Analysis**:
   * Open the data in the XRD software.
   * Export the data as a CSV and save it to your USB.

**7. Calculations and Analysis**

*Part I – Simple Diffraction*

1. Report the average value of *d*, the grating spacing (µm), along with the standard deviation in your measurement.
2. Report the average value of λ2 (nm) along with the standard deviation of your measurement.
3. List two sources of error in your measurement and how they can be minimized.

*Part II – X-Ray Diffraction*

1. Graph all the salts and the unknown mixtures (intensity vs 2θ). Label all the major peaks for each salt and your unknown. Find the hkl indices for each peak. Put this data into a table and upload each graph here.
2. Peak Intensity Ratios

* Identify the two salts in your mixture.
* Explain how you know you have identified the correct composition.

1. Calculation of Percent Composition

Use the ratio of peak intensities to estimate the relative amounts of each salt in the mixture. The percentage of each salt can be calculated using the following formula

Where:

* is the intensity of the major peak for salt
* is the sum of intensities for all the major peaks corresponding to each salt in the mixture

1. Report the composition

* Using the calculated intensities, determine the percent composition of each salt in the unknown mixture
* Report the identity of the salts and their respective percentages in the lab report

1. How does XRD provide information about the crystal structure of a material?
2. Why is it important to use the same scanning parameters for both known salts and the unknown mixture?
3. Discuss the potential sources of error in the XRD analysis and how they might affect the calculated percent composition of the salts.