**Experiment 14: X-Ray Diffraction**

**Scope:**
A powder sample of a cubic crystal will be exposed to copper K\(\alpha\) x-rays in a diffractometer. The diffraction pattern will be used to determine the crystal structure, to calculate the spacing between layers in the crystal, and determine the lattice constant \(a\).

**Introduction:**

See Brown 3.1-3


The diffractometer consists of a powder sample on a glass slide with a detector that can be varied in angle relative to the sample plane.

**Procedure:**

**USE EXTREME CARE IN WORKING WITH X-RAYS.**
The instructor will insert and remove the samples.

For operation of the x-ray machine, see detailed instructions on the front of the x-ray machine and on the computer which is used to control the x-ray machine, obtain, record and save data.

Electrons are accelerated across a large potential difference and then strike a copper target producing the x-rays. Our x-rays are from the copper K\(\alpha\) lines. It turns out that there are two very closely spaced wavelengths: \(\lambda_{K\alpha_1} = 1.5405\ \text{Å}, \lambda_{K\alpha_2} = 1.5443\ \text{Å}\). If you are not sufficiently resolved to see the two distinct peaks it is appropriate to use the intensity-weighted average for the wavelength (see reference):

\[
\bar{\lambda}_{K\alpha} = \frac{(2\lambda_{K\alpha_1} + \lambda_{K\alpha_2})}{3} = 1.5418\ \text{Å}.
\]

The sample holder and the detector are mechanically connected and they both rotate so the angles are as indicated in the figure. The sample plane is at angle \(\theta\) with respect to the incident beam. The detector is at angle \(\theta\) with respect to the sample plane. The quantity that is recorded is the angle between the detector and the incident beam, \(2\theta\).
1. Get an x-ray diffraction spectrum for Al powder. After identifying a strong peak, take another spectrum while zooming in on a single peak until you can see the two peaks corresponding to the two wavelengths.
2. Repeat for Aluminum foil.
3. Repeat the above for Cr powder and other samples.
4. Now get a spectrum for an unknown. (Ask instructor.)
5. Ask for another sample if you want to see more.

Report

• Determine the Bragg angles of the diffraction peaks and calculate the lattice plane spacings \( d \) that would produce them. From the pattern of Bragg angles, determine whether each of your samples is a face-centered cubic or body-centered cubic structure, and calculate the lattice constant, \( a \). (See example in the Brown's text.) Compare your results with accepted values.
• Identify any similarities or differences between Al powder and Al foil.
• Use a table of crystallographic data for the elements (see link on web page) to try to identify your unknown sample. Such tables may be found at the link on the course webpage or in the American Institute of Physics Handbook or in some editions (with small page size) of the Handbook of Chemistry and Physics or in Cullity, Elements of X-Ray Diffraction.

Reference
Stout and Jensen, X-ray structure and Determination, McMillan, 1968