HACETTEPE UNIVERTSTY CHEMICAL ENGINEERING DEPARTMENT

X-RAY DIFFRACTION

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Presentation includes;

- Introduction
- What is XRD
- History
- Istrumentation
- How Does It Work
- Aplications
- Summary and Conclusions
- Referances

Introduction

Motivation:

- X-ray diffraction is used to obtain structural information about crystalline solids.
- Useful in biochemistry to solve the 3D structures of complex biomolecules.
- Bridge the gaps between physics, chemistry, and biology.

X-ray diffraction is important for:

- Solid-state physics
- Biophysics
- Medical physics
- Chemistry and Biochemistry

Figure 1: X-ray Diffractometer

X - Ray

X-rays on Electromagnetic Spectrum

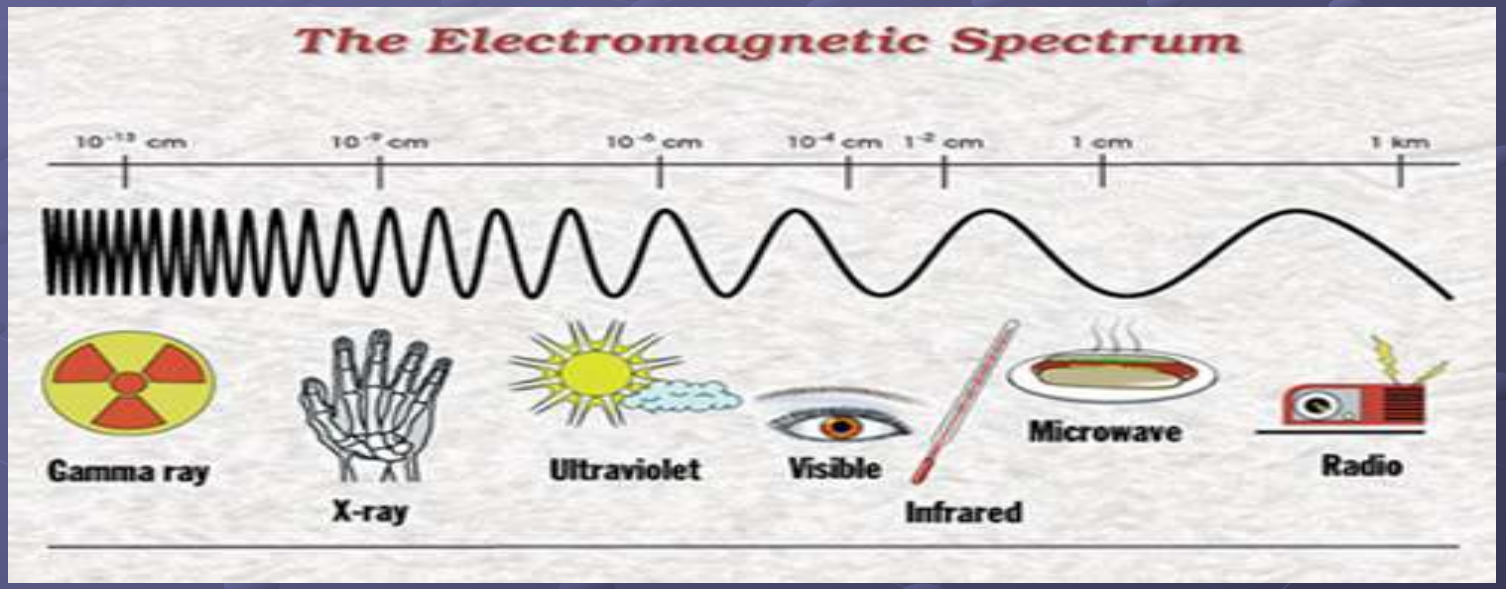


Figure 2: Electromagnetic Spectrum

- Wawelengts range : 0.01 – 10 nm
- Energy : 10^3 – 10^5 eV

What is XRD ?

- Non-destructive analytical technique for identification and quantitative determination of the various crystalline forms, known as ‘phases’.
- Identification is achieved by comparing the X-ray diffraction samples

History of X-Ray Diffraction

- X-rays were discovered in 1895 by Wilhelm Conrad Roentgen (1845-1923) who was a Professor at Wuerzburg University in Germany
- In June, 1896 X-rays were being used by battlefield physicians to locate bullets in wounded soldiers.
- In 1912 X-rays were used medicine and dentistry
- In 1913 when the high vacuum X-ray tubes designed by Coolidge, X-rays go in to the industry
- In 1914 the first diffraction pattern of a crystal made by Knipping and von Laue
- In 1915 Theory to determine crystal structure from diffraction pattern developed by Bragg
- In 1953 DNA structure solved by Watson and Crick
- Today we use XRD to determine atomic structures of materials

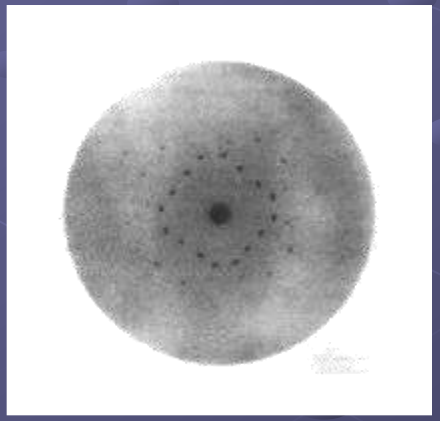
Figure 3:

W.C. ROENTGEN

Figure 4:

Roentgen's wife's hand
(The first x-ray photo)

Max von Laue (1897-1960)



- The first kind of scatter process to be recognised was discovered by Max *von* Laue who was awarded the Nobel prize for physics in 1914 *"for his discovery of the diffraction of X-rays by crystals".* His collaborators Walter Friedrich and Paul Knipping took the picture on the bottom left in 1912. It shows how a beam of X-rays is scattered into a characteristic pattern by a crystal. In this case it is copper sulphate.

- The X-ray diffraction pattern of a pure substance is like a fingerprint of the substance. The powder diffraction method is thus ideally suited for characterization and identification of polycrystalline phases.

PHOTOS OF X-RAY AND X-RAY DİFFRACTION

X-ray of a pregnant woman

Figure 5

Photo of X-ray Diffraction Pattern

- The array of spots is called a *Laue* pattern
- The crystal structure is determined by analyzing the positions and intensities of the various spots
- This is for NaCl

Figure 6

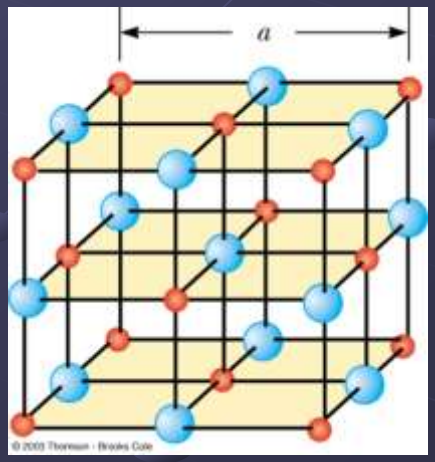


Figure 7

Basic Component Of XRD Machine

XRD machine will consist of three basic component.

- Monochromatic X-ray source
- Sample-finely powdered or polished surface-may be rotated against the center – (goniometer).
- Dedector - such as film, strip chart or magnetic medium/storage.

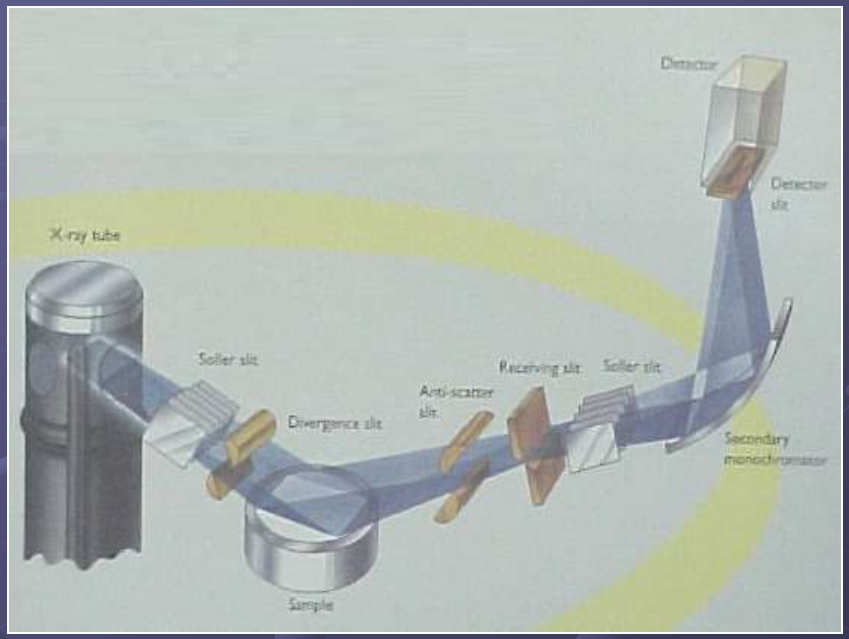


Figure 8: equipment of xrd

How Does XRD Work

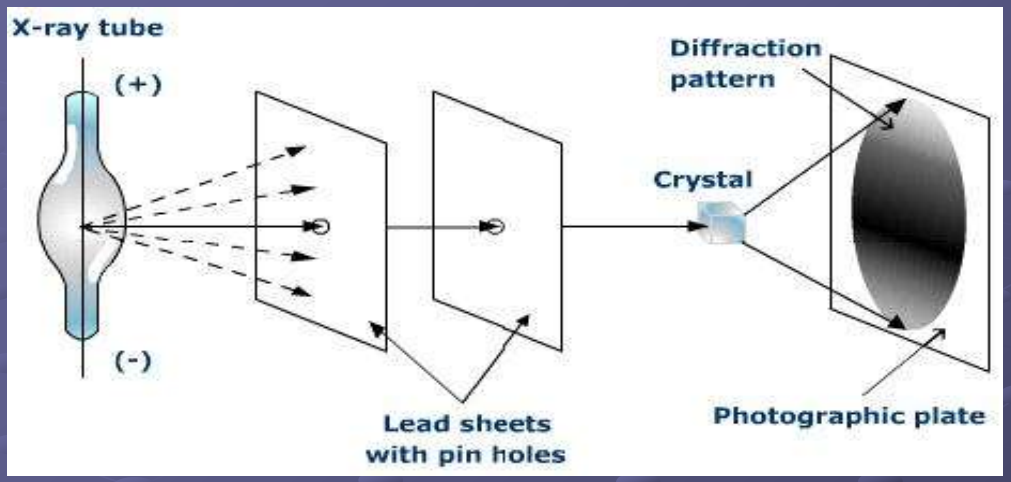


Figure 11:

- X-rays are generated in a cathode ray tube
- Electrons bombarding target material with electrons
- When the electrons have enought energy to get out inner shell electrons of the target material, characteristic X-ray spectra are produced.
- X- ray spectras create photographic film

How does Xrd Diffraction work?

In powder XRD method, a sample is ground to a powder (±10μm) in order to expose all possible orientations to the X-ray beam of the crystal values of λ, *d* and θ for diffraction are achieved as follows:

1. λ is kept constant by using filtered X-radiation that is approximately monochromatic.
2. *d* may have value consistent with the crystal structure.
3. θ is the variable parameters, in terms of which the diffraction peaks are measured.

How Diffraction Works: Schematic

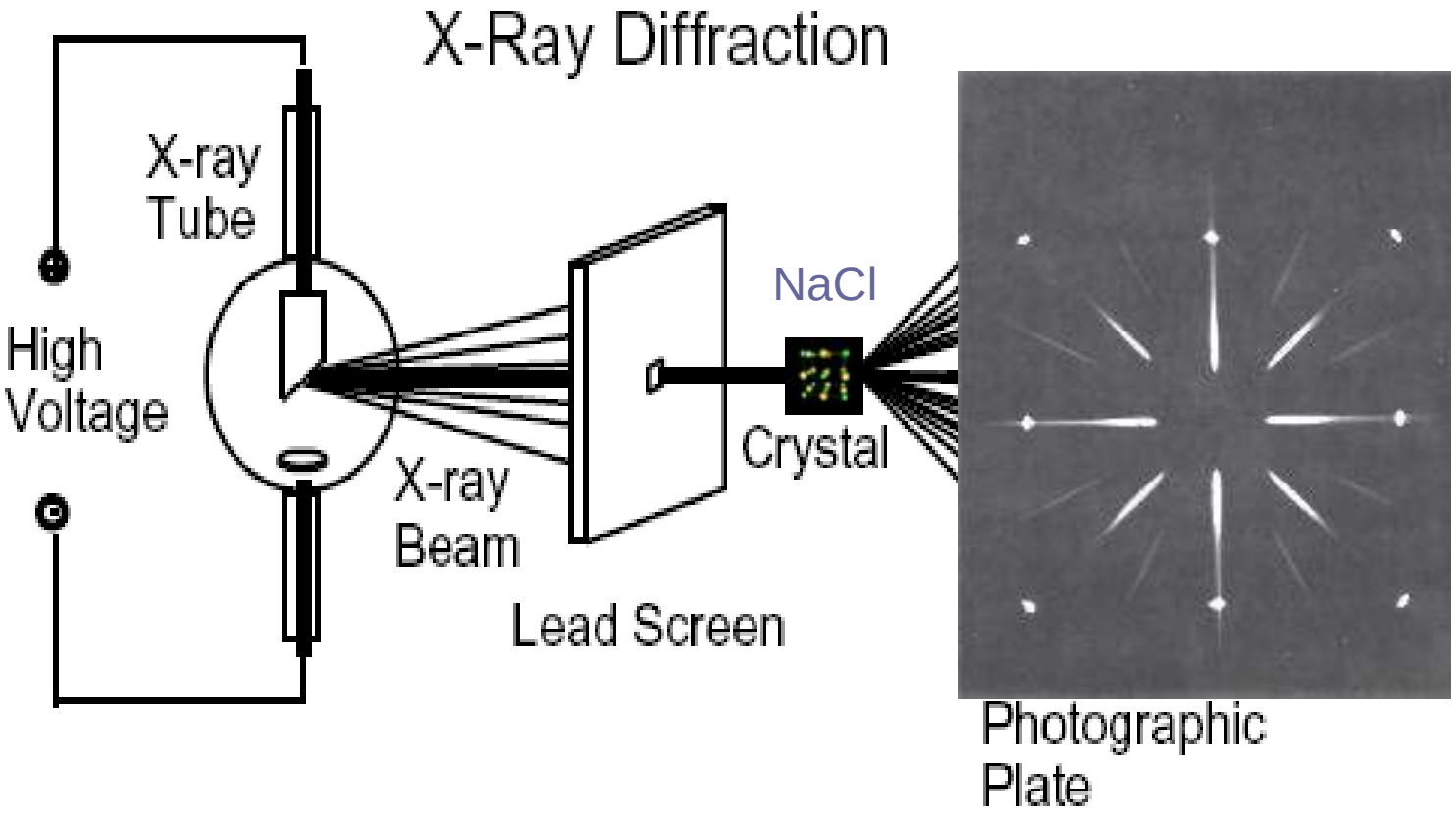


Figure 2. A schematic of X-ray diffraction.

Figure 9:

http://mrsec.wisc.edu/edetc/modules/xray/X-raystm.pdf

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How Diffraction Works: Bragg's Law

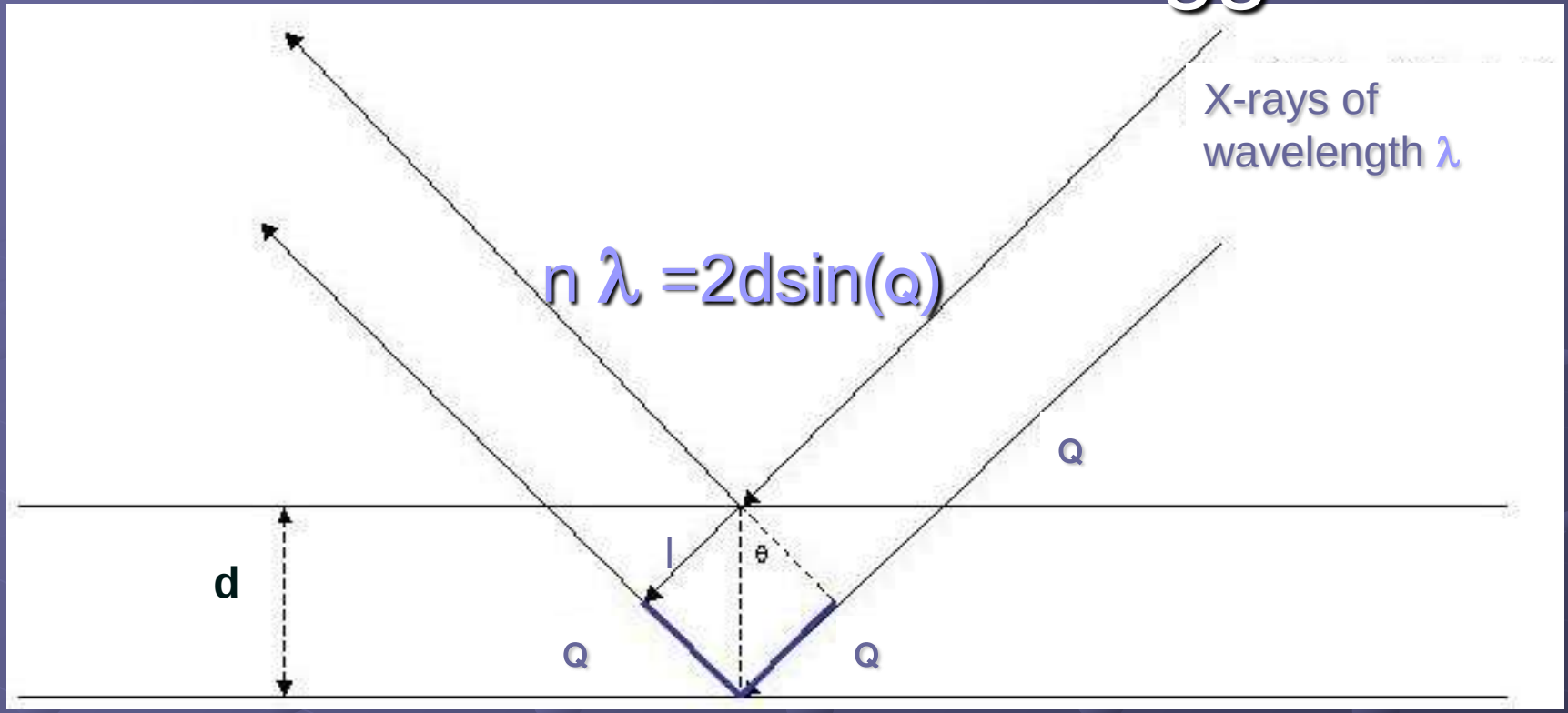

Figure 10:

- Similar principle to multiple slit experiments
- Constructive and destructive interference patterns depend on lattice spacing (d) and wavelength of radiation (λ)
- By varying wavelength and observing diffraction patterns, information about lattice spacing is obtained

Example of Diffraction Patterns

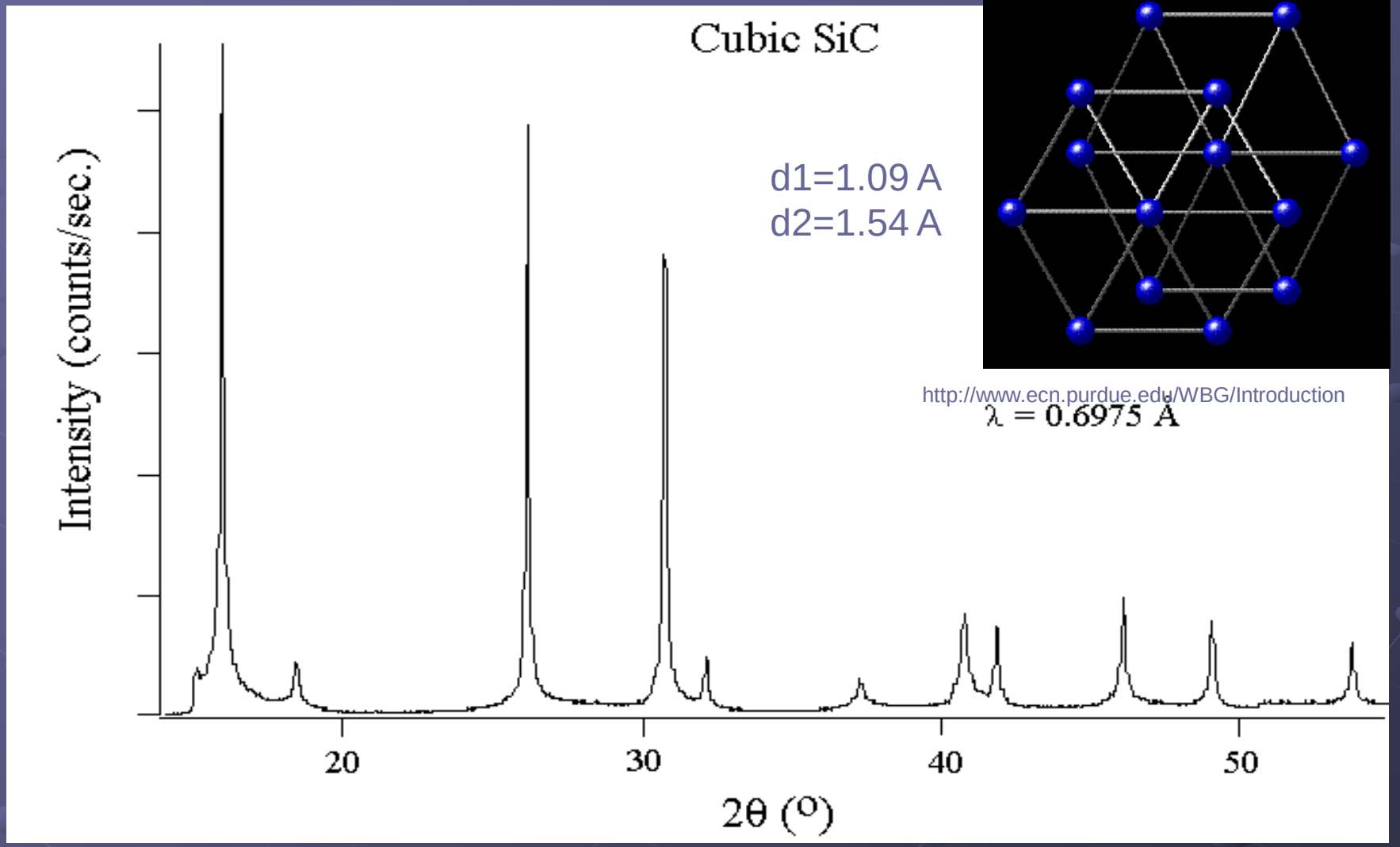

Figure 12:

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http://www.eserc.stonybrook.edu/ProjectJava/Bragg/

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

Applications

- New mineral identification, crystal solution
- Determination of unit cell, bond-lengths, bond-angles and site-ordering
- Characterization of cation-anion coordination
- Variations in crystal lattice with chemistry
- Determination of crystal-chemical vs. environmental control on mineral chemistry
- Powder patterns can also be derived from single-crystals by use of specialized cameras (Gandolfi)

Summary and Conclusions

- X-ray diffraction is a technique for analyzing structures of molecules
- X-ray beam hits a crystal, scattering the beam in a manner characterized by the atomic structure
- Even complex structures can be analyzed by x-ray diffraction, such as DNA and proteins
- This will provide useful in the future for combining knowledge from physics, chemistry, and biology

Referances

- http://www.ndt-ed.org/EducationResources/CommunityCollege/Radiography/Introduction/history.htm
- http://spectroscopyonline.findanalytichem.com/spectroscopy/article/articleDetail.jsp?id=443484
- http://techcenter.jefferson.kctcs.edu/RS/topics/properties/Pages/EM.aspx
- http://serc.carleton.edu/research_education/geochemsheets/techniques/XRD.html
- http://www.chem.wisc.edu/~newtrad/CurrRef/BDGTopic/BDGtext/BDGGraph.html
- www.nhn.ou.edu/~johnson/Education/Juniorlab/Presentations/2004-X-RayDiffraction-DayRoss.ppt+x+ray+diffraction+history&hl=tr&gl=tr&pid=bl&srcid=ADGEESicFwFC4Osd {a slide, written by Day and Ross }

Please forgive me if i made a mistake

Thanks for your attention