X Ray Fluorescence Spectroscopy

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Outline

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  - Bremsstrahlung Rays
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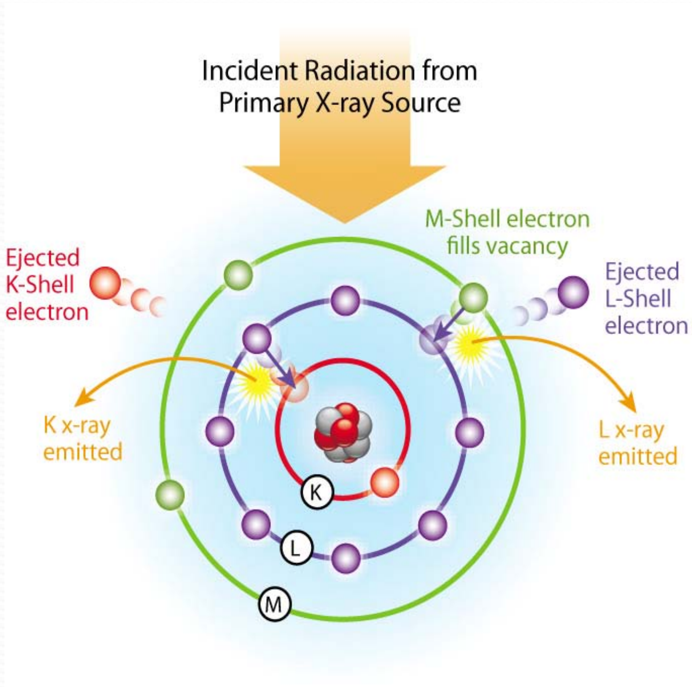
X Ray Fluorescence

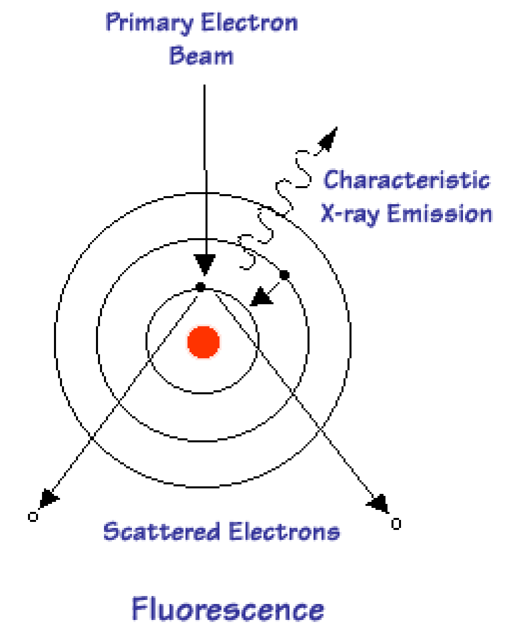
- X-Ray Fluorescence: *Emission of characteristic secondary X rays from a material that has been excited by bombarding with high energy X-Rays.*
- Characteristic X-Rays:

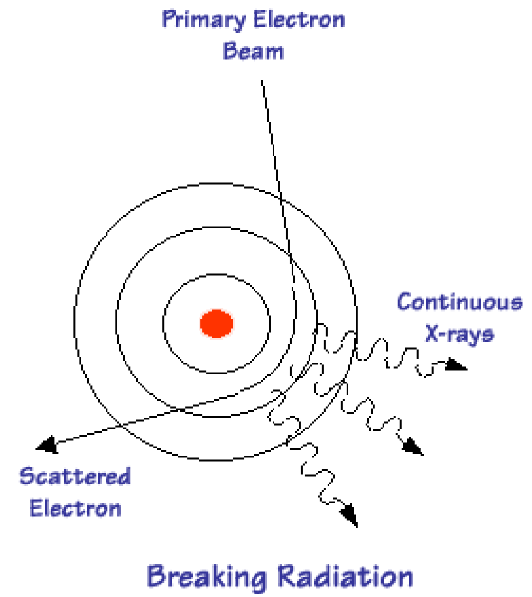
X-Rays emitted from heavy elements when their electrons make transitions from a higher to a lower atomic energy level.

- *Characteristic X Rays* are unique for each element as energy emitted is proportional to the binding energy of the element.

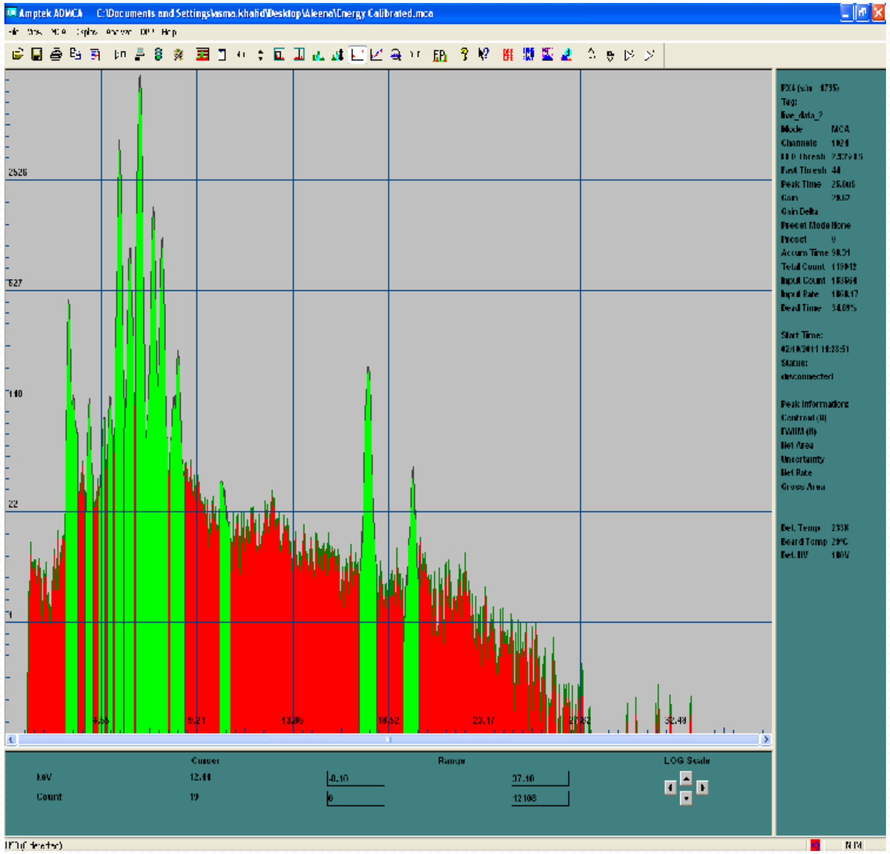


- Bremsstrahlung Radiation: *The electromagnetic radiation produced due to deceleration of a charged particle (electron) when deflected by another charged particle (the atomic nucleus).*
- Bremsstrahlung radiations have a continuous spectrum where as intense peaks of the characteristic x rays can be seen on the spectrum.





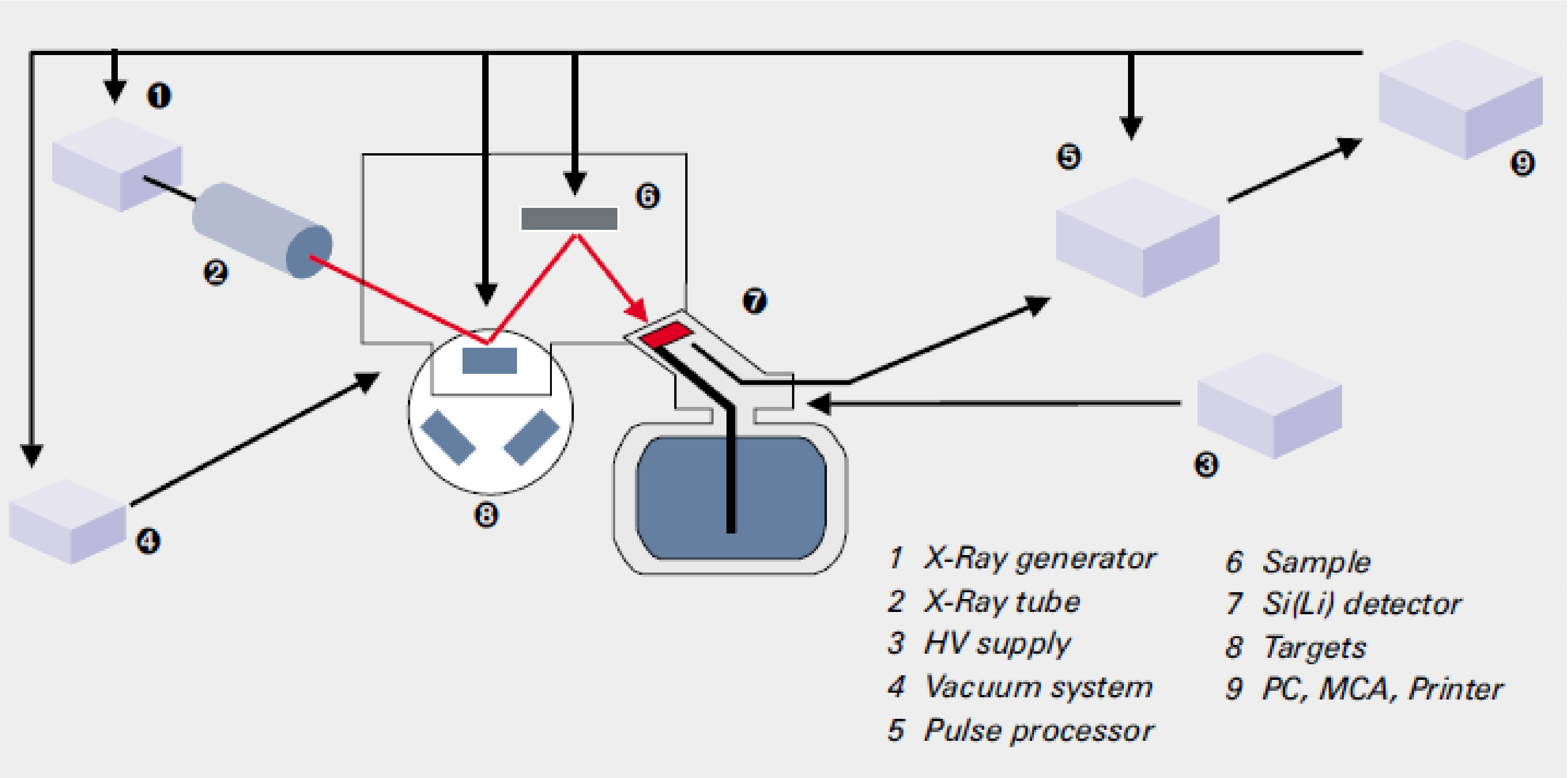
Bremsstrahlung and Characteristic X Rays - Spectrums



XRF Analysis

- XRF Analysis is of two types
  - Energy Dispersive XRF (EDXRF)
    - *Uses a solid state detector and distinguishes each peak according to its energy*
  - Wave Dispersive XRF (WDXRF)
    - *Uses the scanning crystal as the dispersive element and distinguishes each peak according to its wavelength*
- We have used the EDXRF spectrometer.

EDXRF Spectrometer Working Principle



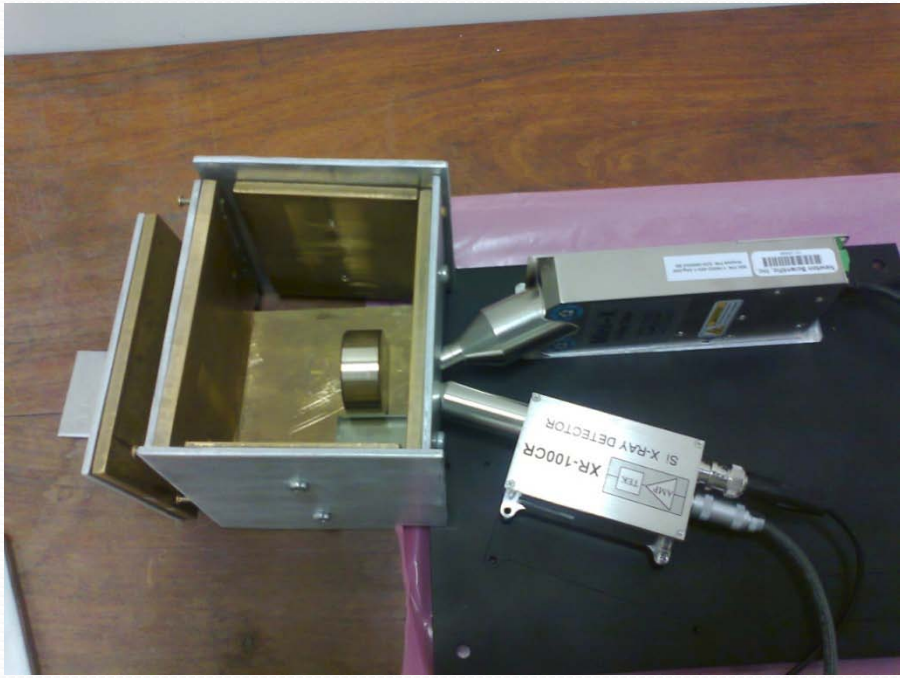
Sample Analysis

Sample Analysis

- Obtaining A Spectrum
- Calibration of Curve for Energy
- XRF-FP Corrections
- Sample Analysis
  - Calibration using Standards – Intensities obtained
  - Calculation of Calibration Coefficients – Concentrations Obtained

Obtaining A Spectrum

- We use ADMCA along with the PX4 MCA to obtain the spectrum for the sample
- To Calibrate: We identify at least two peaks in the spectrum and enter the characteristic energy value for each peak. Using the "Calibrate" function of ADMCA we calibrate the spectrum in terms of energy



- This Energy calibration is saved in the MCA and loaded automatically each time a new spectrum needs to be obtained.

XRF-FP Analysis

- The XRF-FP software loads the spectrum obtained by ADMCA and using the initial parameters etc, corrects the spectrum.
- Three steps for correction:
  - Corrections for escape peaks, sum peaks, background continuum, background peaks etc.
  - Deconvolution
  - Accounting for attenuation and matrix effects
- Using the corrected spectrum, it then calculates the relative intensities and concentrations of each element in the sample.

XRF-FP Analysis

- The following tables show the intensities and concentrations of various elements in a Steel Sample.

Element	Concentration (wt (%))	Proposed Concentration (wt(%))
Cr	19.055	18.45
Mn	1.281	1.63
Fe	65.939	65.19
Ni	8.67	12.18
Cu	0.294	0.169
Mo	2.902	2.38

Moseley's Law

Moseley’s Law

- **Moseley's law** is an empirical law concerning the characteristic x-rays that are emitted by atoms.
- Moseley was able to show that the frequencies of characteristic X-rays emitted from chemical elements are proportional to the square of the element's atomic number

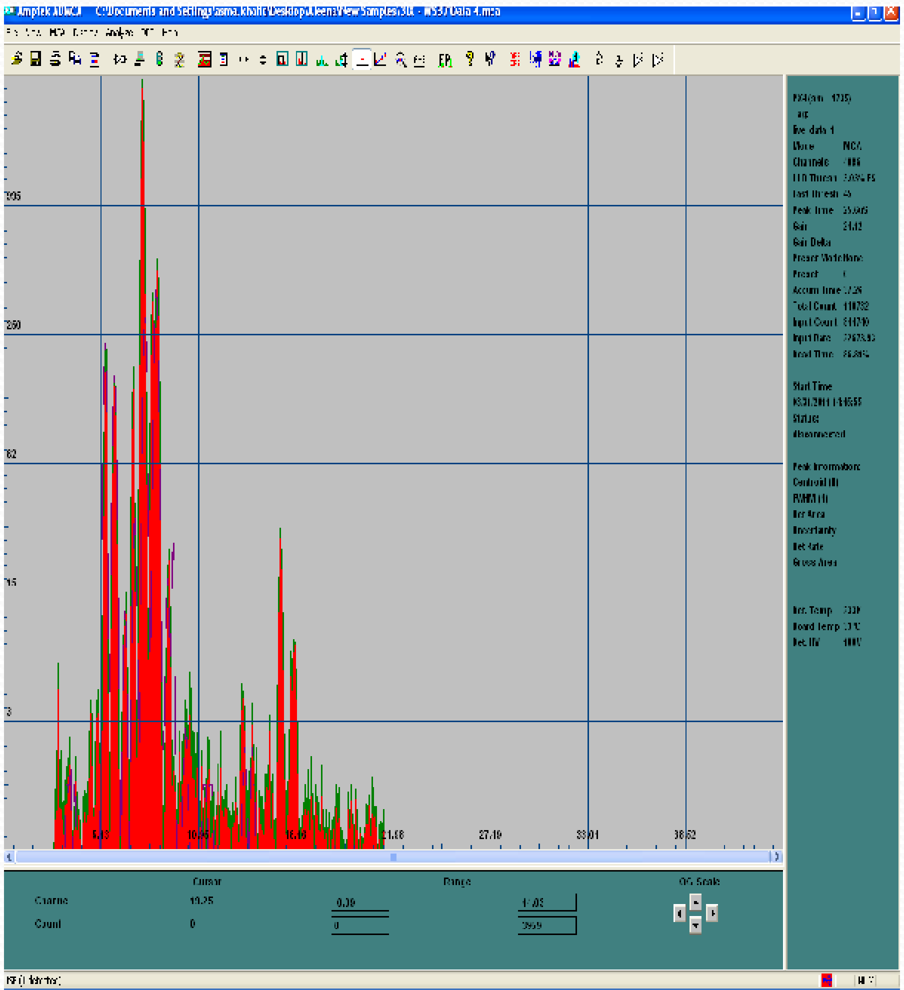
$$E_{k\alpha} = \frac{3R_{\infty}(Z-1)^2}{4}$$

- A finding which supported Bohr's model of the atom in which the atomic number is the same as the number of positive charges in the nucleus of the atom.

Verification of Results

- Two different samples used for the verification of Moseley's Law:
  - Steel
  - Silicon-Brass
- A spectrum of the characteristic X Rays was obtained for each sample and calibrated. Using these energies, a graph between square root of energy and atomic number of the element was plotted.
- The slope of the resulting graph gave the Rydberg Constant.

Silicon – Brass



Element	Atomic Number	Energy (keV)
Chromium	24	5.41
Manganese	25	5.90
Iron	26	6.40
Cobalt	27	6.93
Nickel	28	7.48
Copper	29	8.05
Zinc	30	8.64
Antimony	51	26.36
Lead	82	74.95
Bismuth	83	77.1

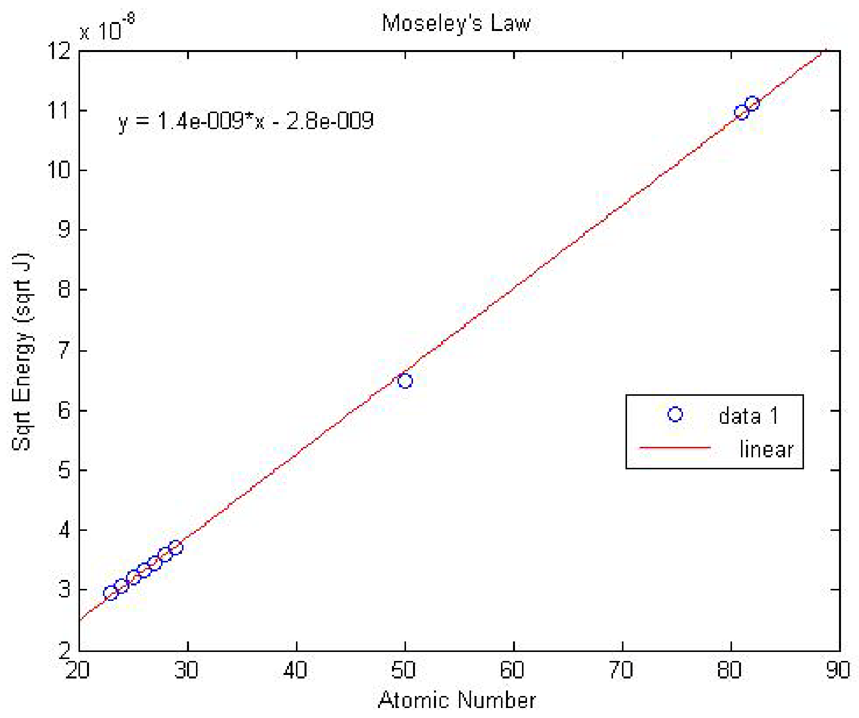
Silicon - Brass

$$E_{k\alpha} = \frac{3R_\infty (Z-1)^2}{4}$$

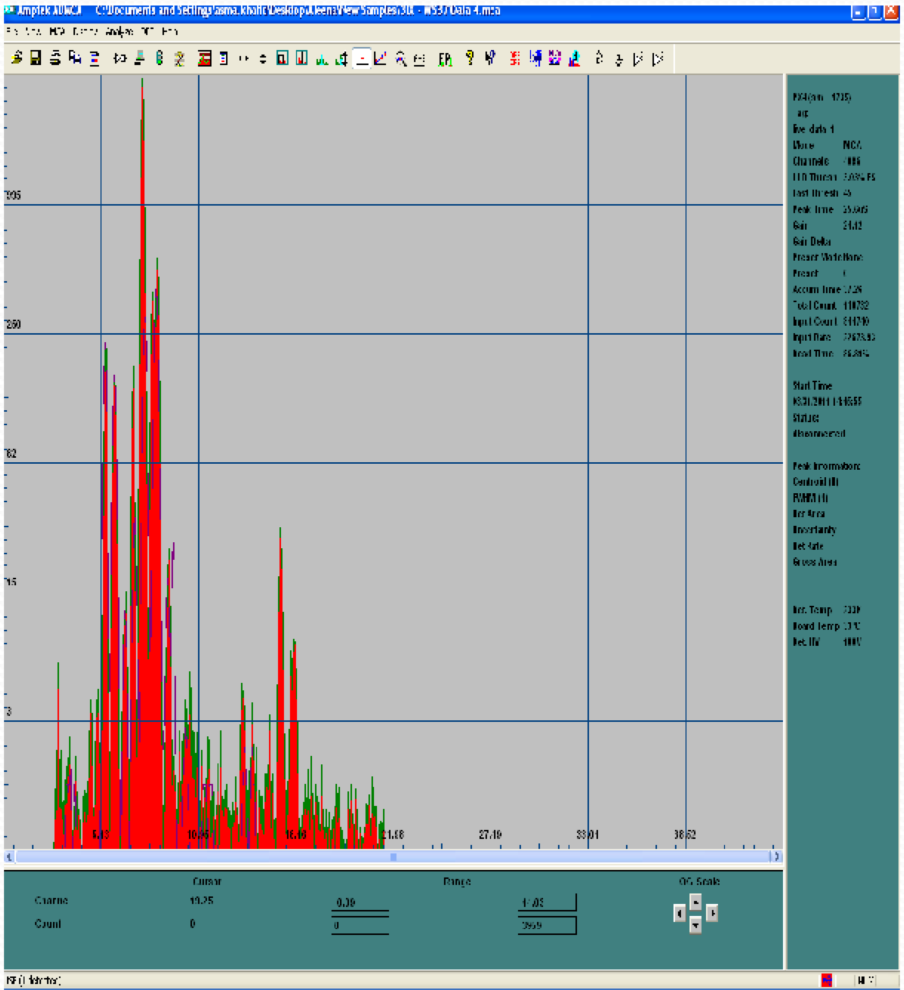
and thus we get: slope as:

$$2.16 \times 10^{-18} J \approx R_\infty$$

$$R_\infty = 2.11 \times 10^{-18} J$$



Steel



Element	Atomic Number	Energy (keV)
Cr	24	5.41
Mn	25	5.90
Fe	26	6.40
Ni	28	7.48
Cu	29	8.05
Mo	42	17.48

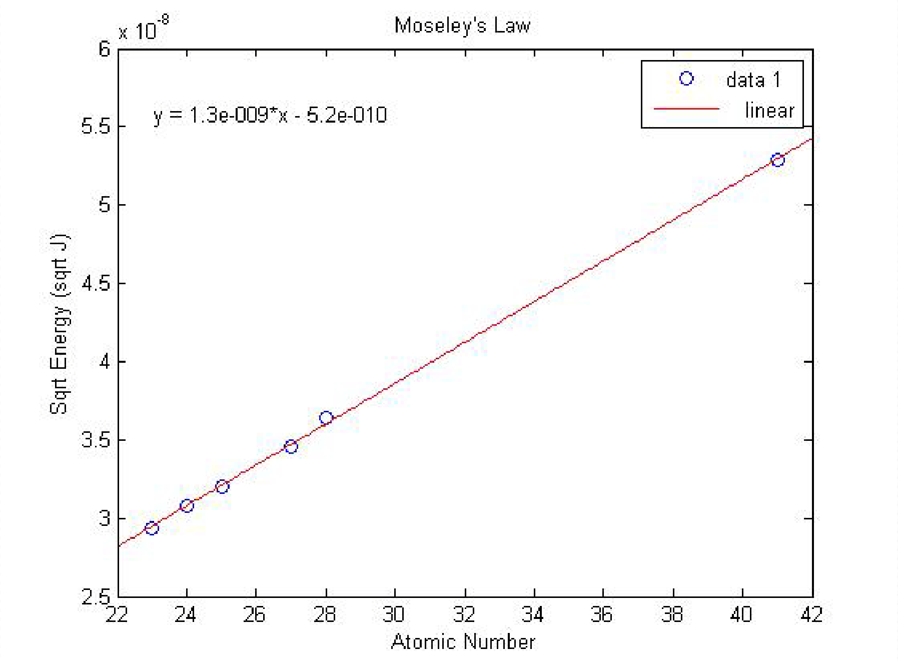
Steel Sample

$$E_{k\alpha} = \frac{3R_\infty (Z-1)^2}{4}$$

and thus we get: slope as:

$$2.253 \times 10^{-18} J \approx R_\infty$$

$$R_\infty = 2.11 \times 10^{-18} J$$



Comtpon Scattering

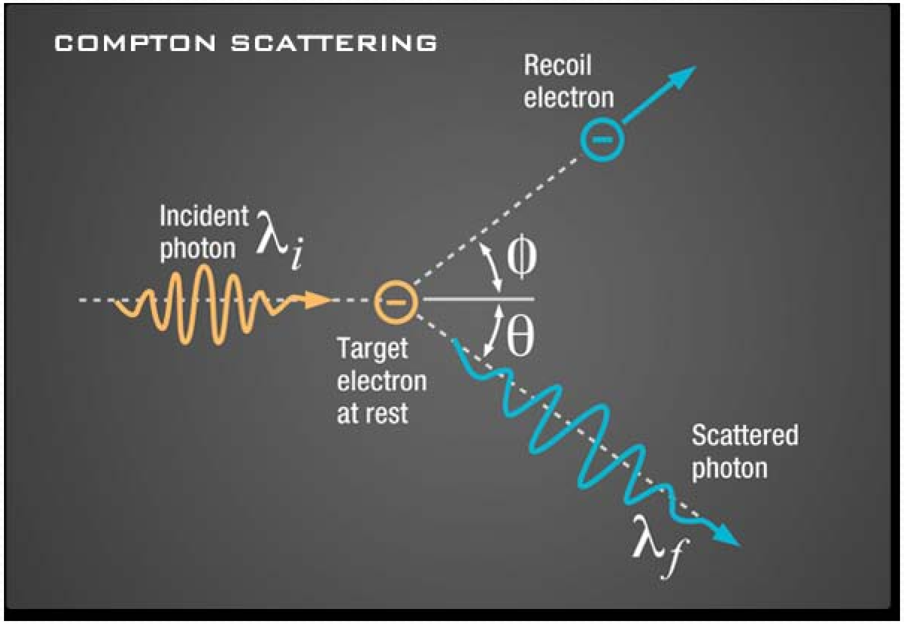
Compton Scattering

- Compton scattering is the interaction of a high energy photon with an electron, and the resulting "scattered" photon which has a reduced frequency, and therefore reduced energy introducing a shift in its wavelength.

$$\lambda' - \lambda = \frac{h}{m_e c}(1 - cos\theta)$$

$$= \lambda_c \, (1 - cos\theta)$$

$$\lambda_c = 2.426 \times 10^{-12} \text{ m}$$

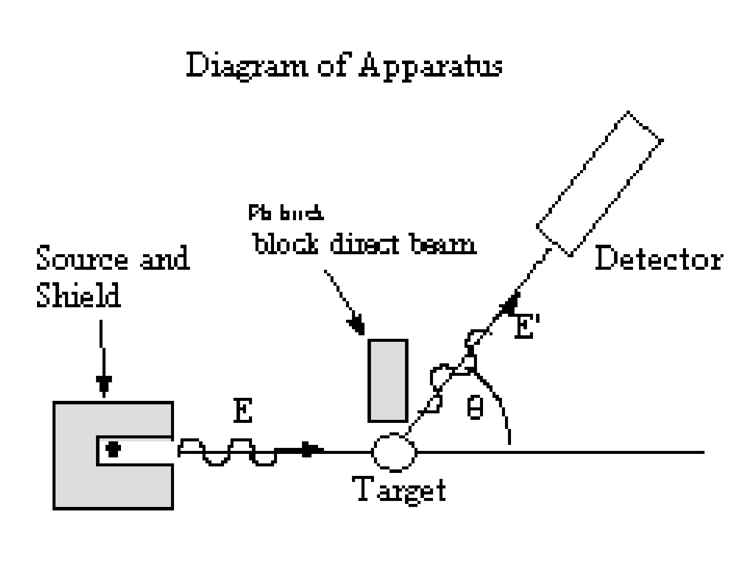


Experimental Setup

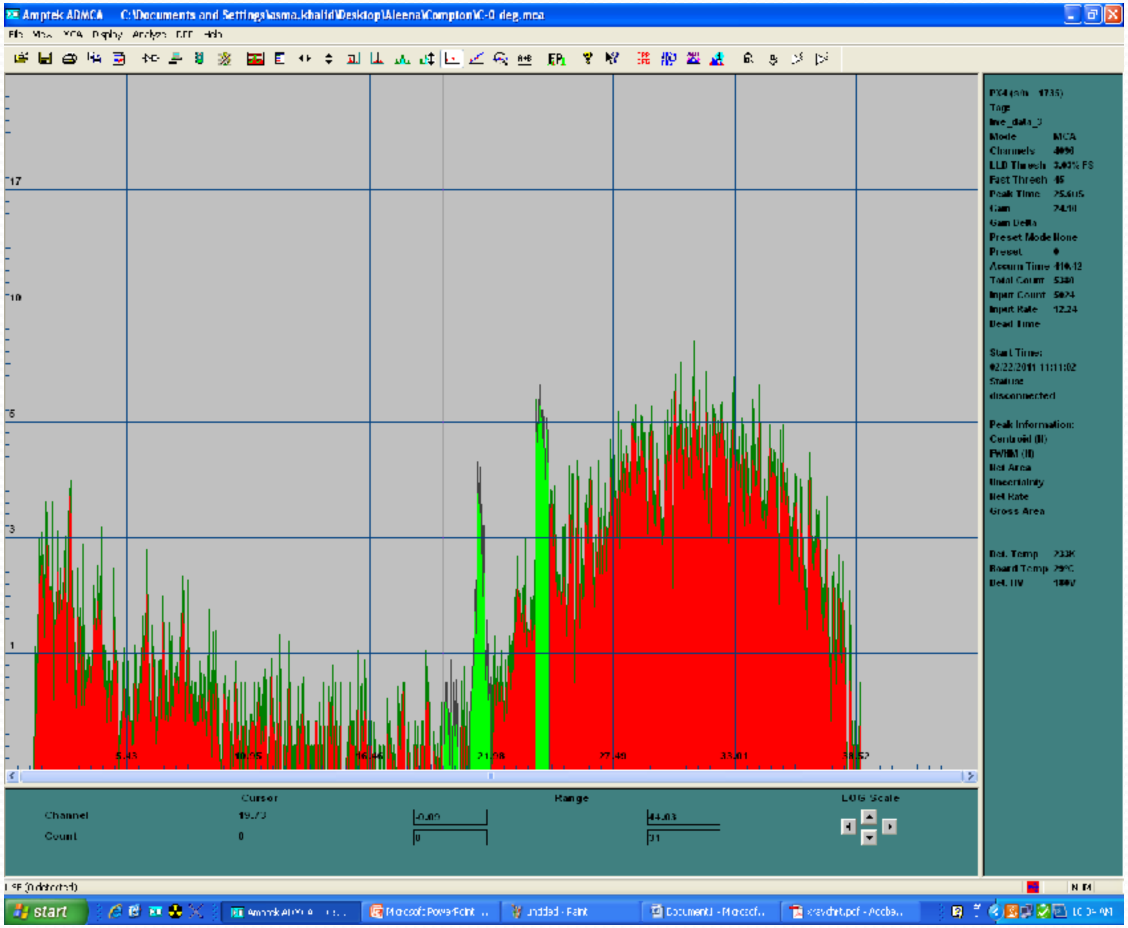
Experimental Setup

- Scattering of X Rays was observed by varying the angle at which the detector was placed. The distance between x-ray tube and the detector was kept larger than 25cm for the detector's safety.

An appreciable shift in wavelengths was seen when the angle was changed.



Results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Mode MCA
Fast Thresh 45
Peak Time
Gain
Gain Delta
Preset Mode None
Total Count
Input Count
Input Rate 12.24
Dead Time
Start Time:
02/22/2011 11:11:02
Status
disconnected
Peak Information:
Net Area
Uncertainty
Net Rate
Gross Area
Det. Temp
Board Temp
Det. HV
Channel
Count
Cursor
Range
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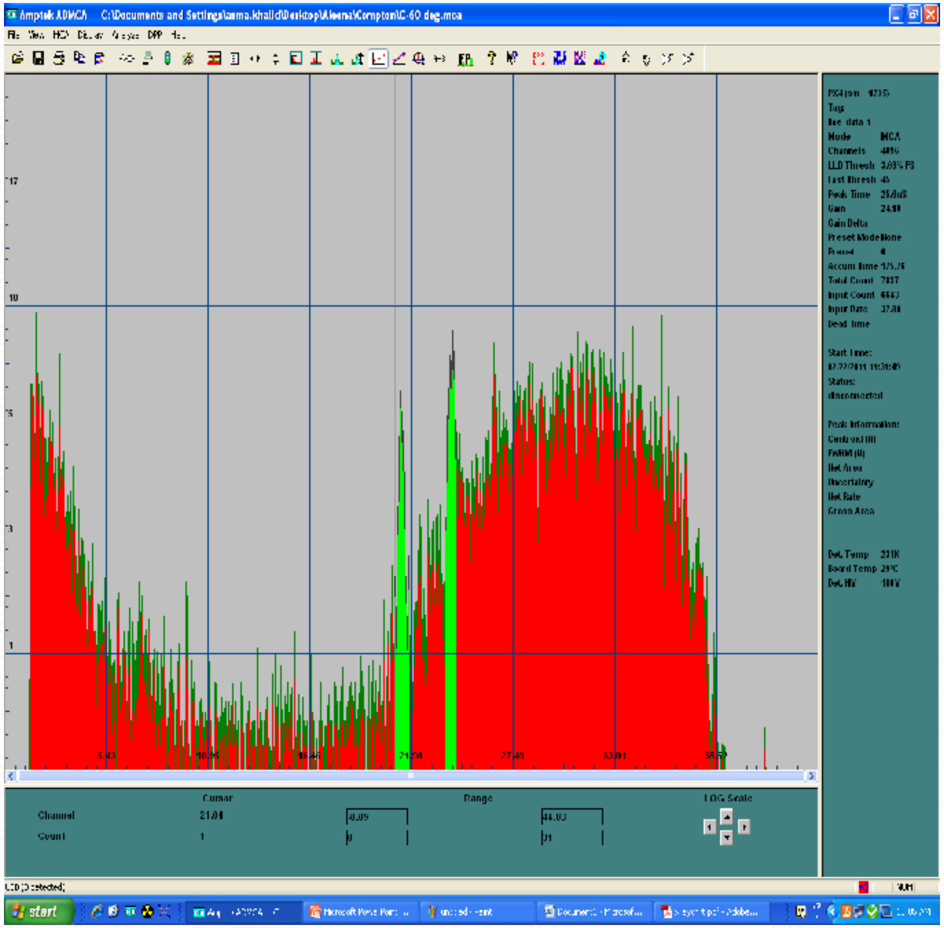
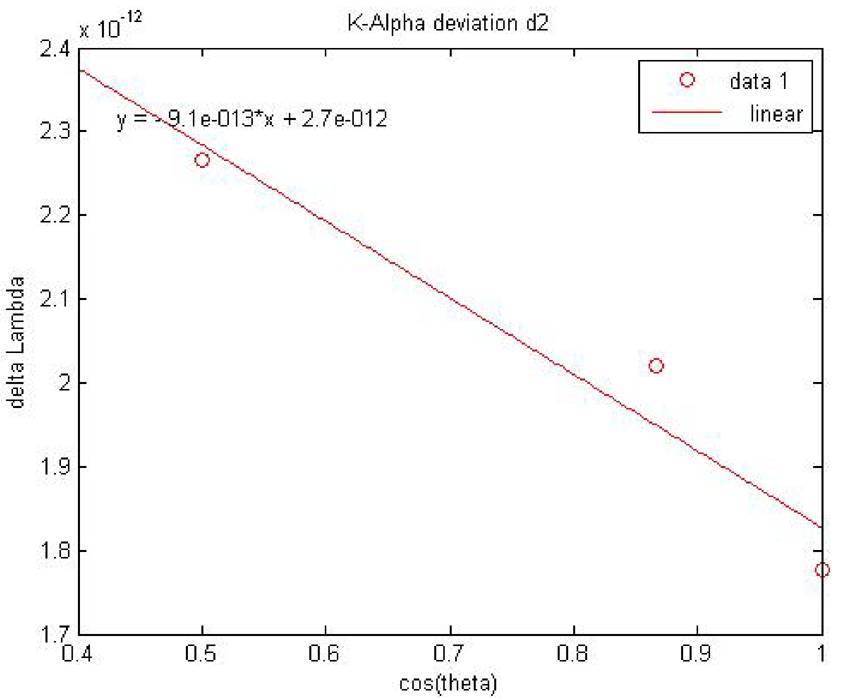
start

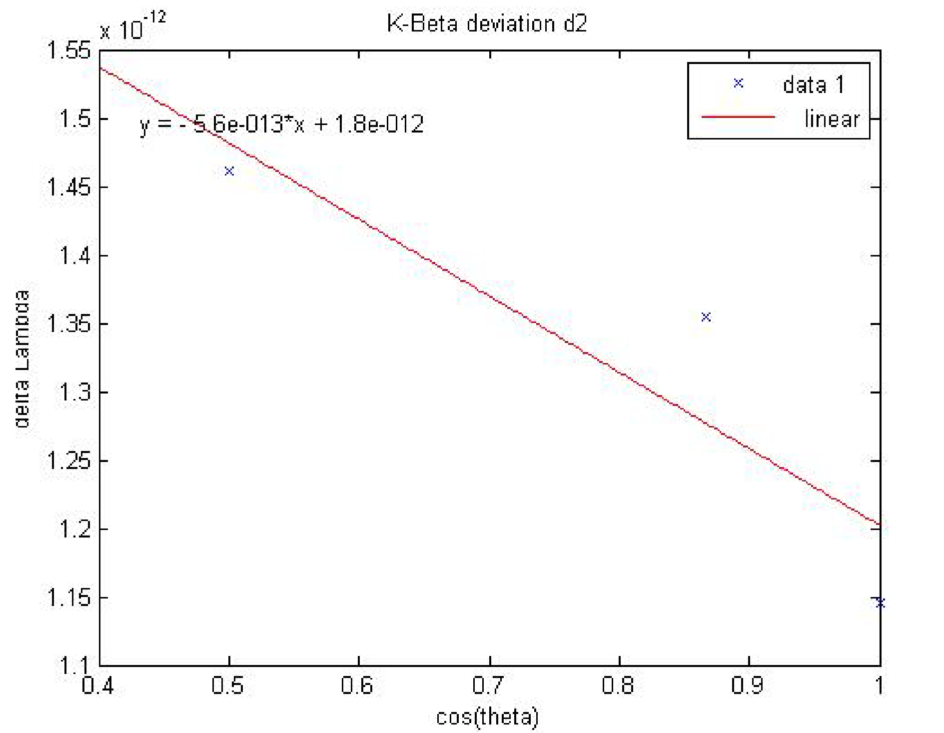
Results



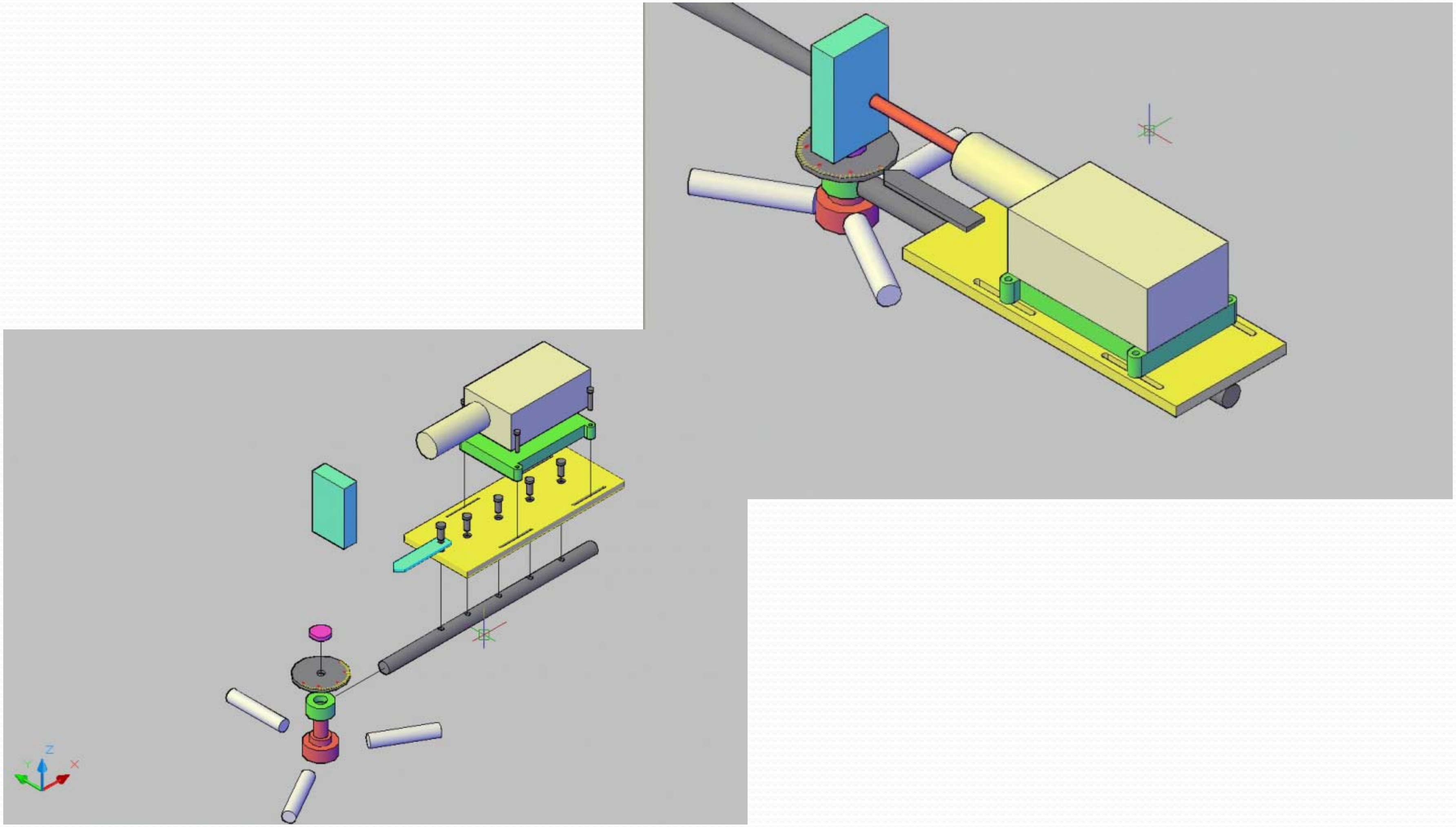
Analysis

- A graph between shift in wavelength (delta lambda) and cosine of angle was plotted.





Proposed Assembly







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