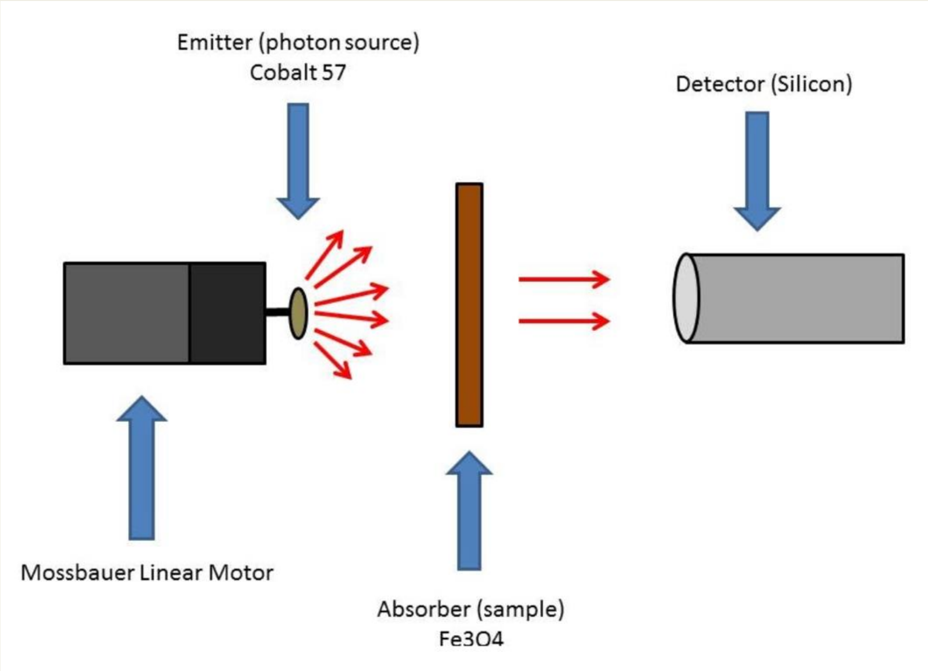
Characterization of Hyperfine Interactions in Magnetite Using Mössbauer Spectroscopy

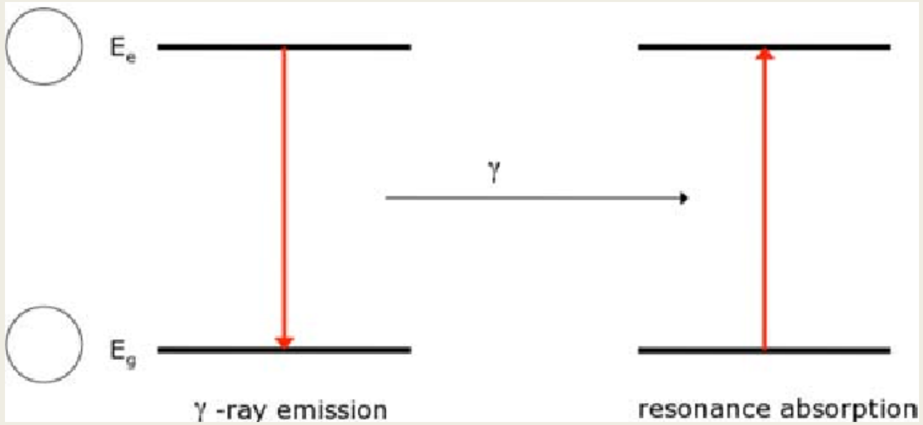
Objectives and Outline

- Goal: Characterize Three Hyperfine Interactions
  - *Isomer Shifts*
  - *Quadrupole Splitting*
  - *Magnetic Hyperfine Splitting*
- Talk Outline
  - *Overview of Mössbauer spectroscopy*
  - *What are the Hyperfine Interactions?*
  - *Experimental Design*
  - *Data Analysis*
  - *Schedule*



Mössbauer Spectroscopy – *Overview*

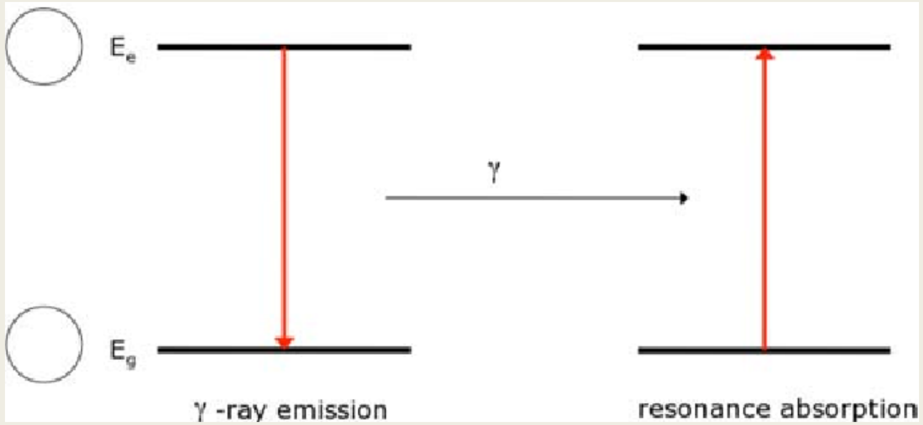
- What is Mössbauer Spectroscopy?
  - *Recoil free emission and absorption of gamma rays*



Kurian, R. (2011). First principles theoretical modeling of the isomer shift of Mossbauer spectra.

Mössbauer Spectroscopy – *Overview*

- What is Mössbauer Spectroscopy?
  - *Recoil free emission and absorption of gamma rays*
- What are hyperfine interactions?
  - *Very small energy shifts in the atomic energy levels of a material*
- What do they tell us?
  - *Oxidation states*
  - *Electron density*
  - *Electron symmetry*
  - *Spin states*
  - *Magnetic properties*



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Mössbauer Spectroscopy – *Source and Target*

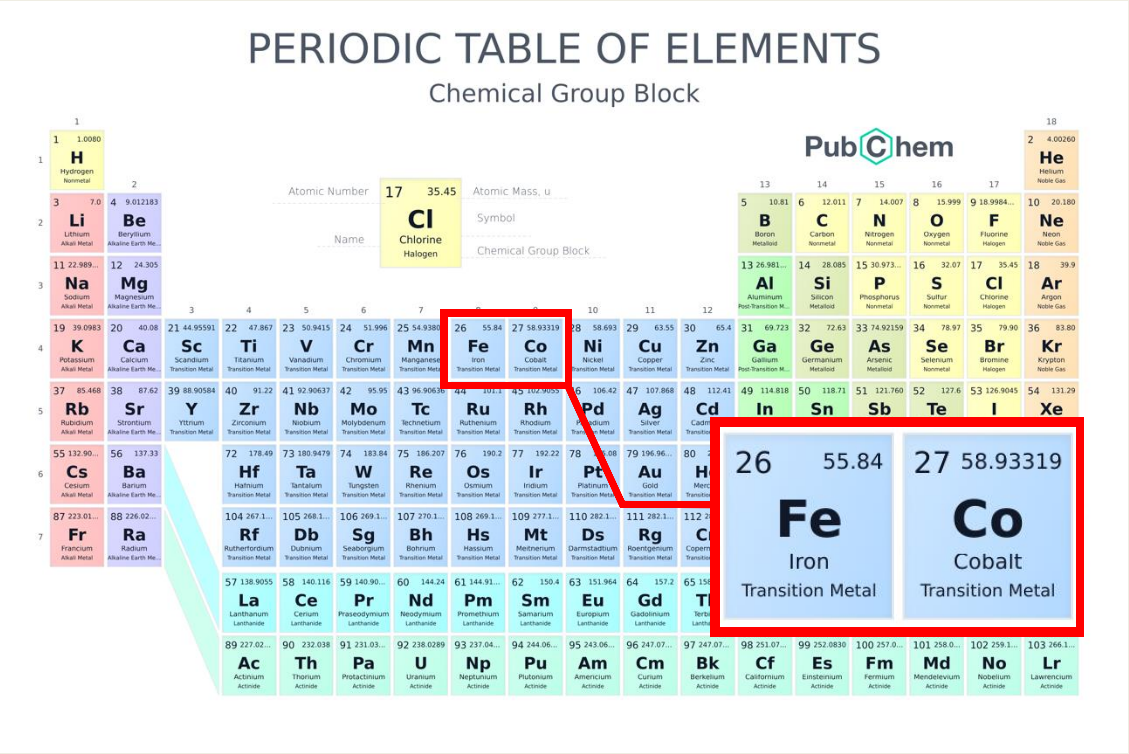
- Why Magnetite (Fe_3O_4)?
  - *Contains Iron 57*
  - *Lattice structure*
  - Fe^{2+} *and* Fe^{3+} *Ions → interesting hyperfine interactions*

- Why Cobalt 57?

→ Because it decays into Iron 57!

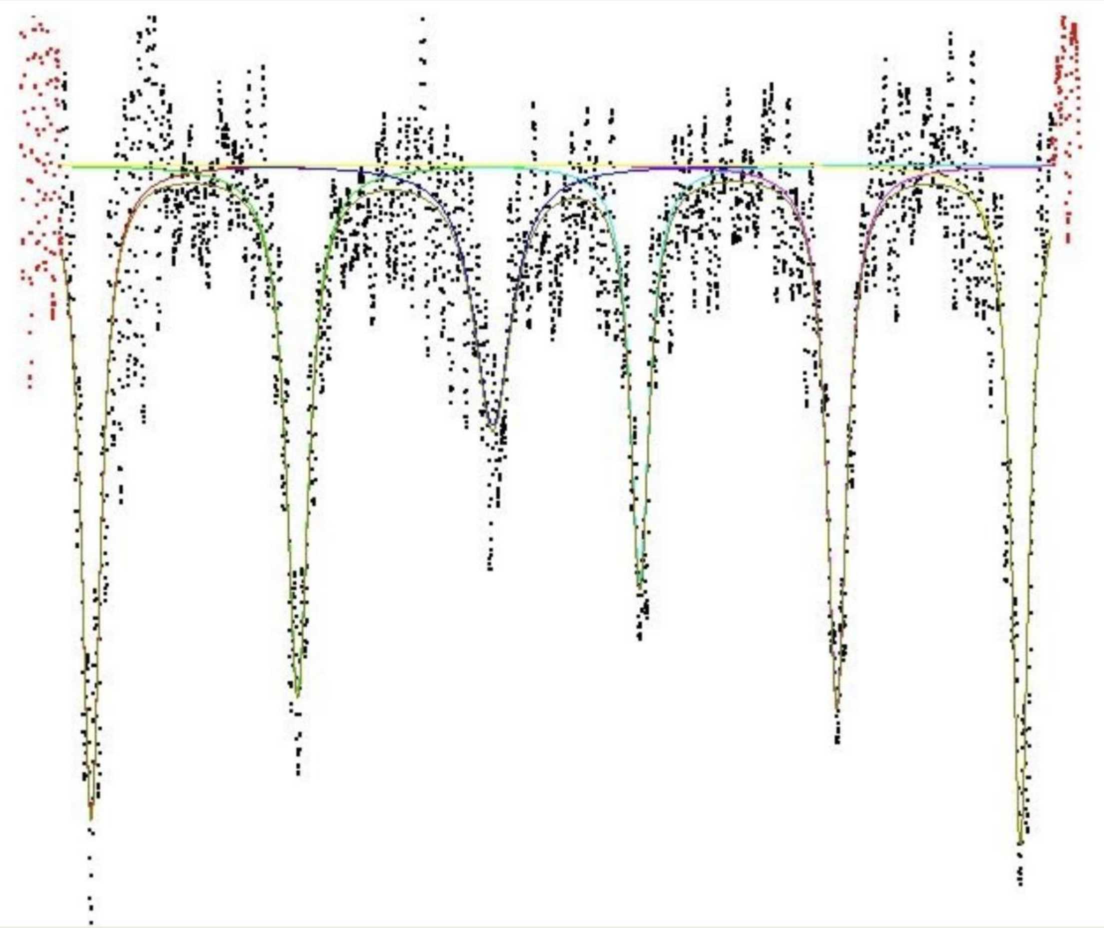
$$^{57}Co \rightarrow ^{57}Fe^{*} + \beta^{+} + \nu_e$$
$$^{57}Fe^{*} \rightarrow ^{57}Fe + \gamma\,(14.4\text{keV}).$$

* Excited State



U.S. National Library of Medicine. (n.d.). Periodic Table of elements

Mössbauer Spectroscopy – The Data



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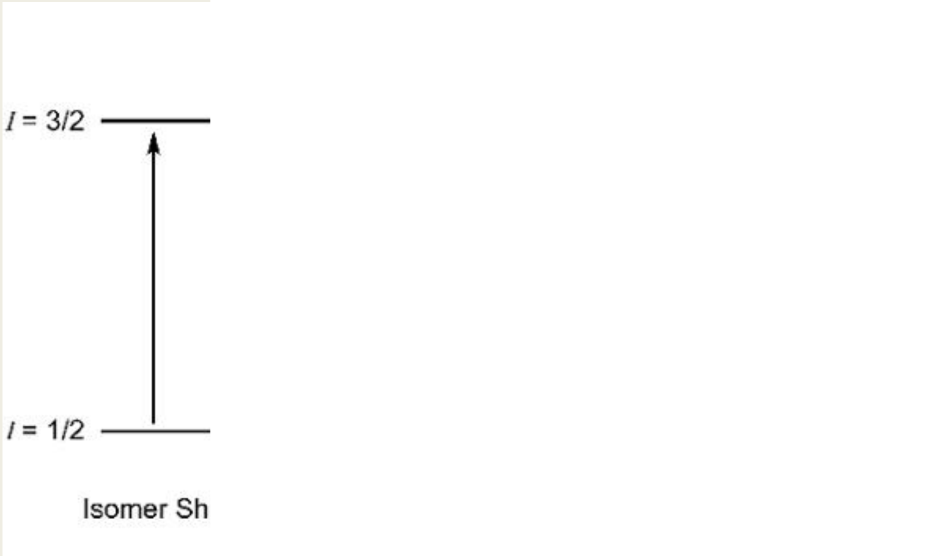
Isomer Shift



- A measure of nuclear charge distribution due to electron density at the nucleus
  - *Distinguish between Fe^{2+} and Fe^{3+} ions*
  - *Fe^{2+} → higher oxidation state → larger shift*
- How does it affect our data?
  - *Shifts peaks*

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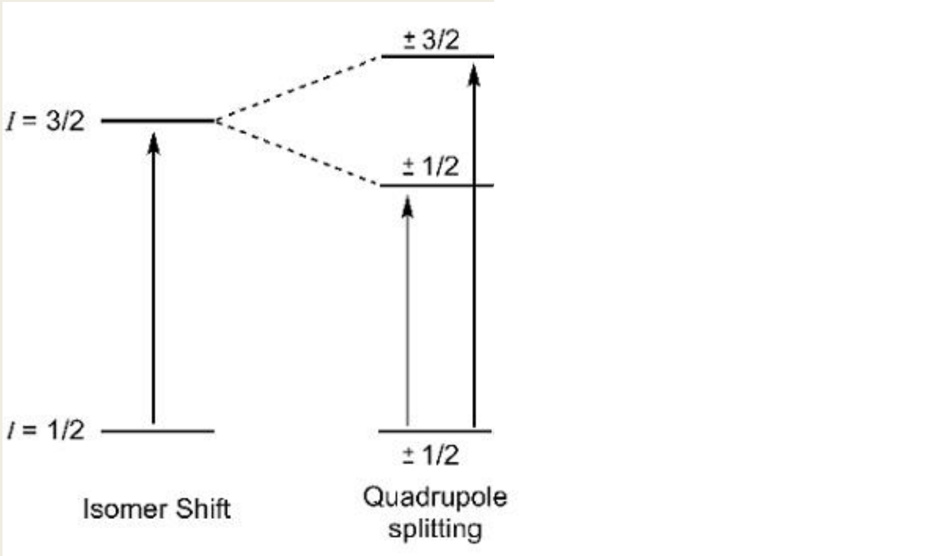
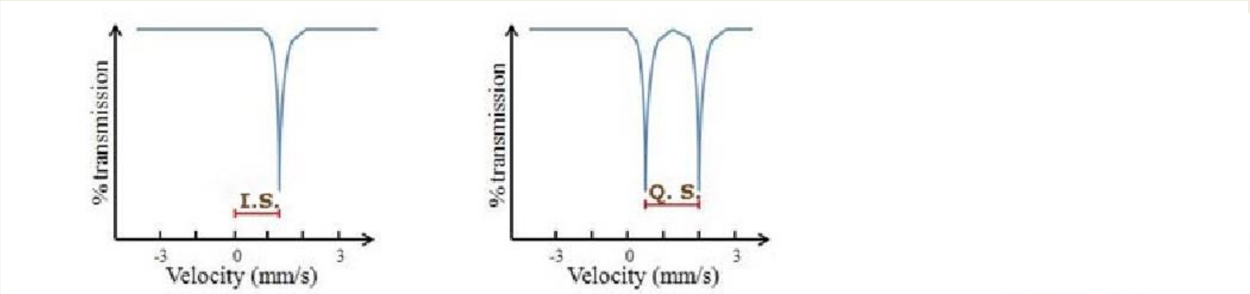
$$\delta E = K[\Psi(0)_s]^2 R^2$$

Quadrupole Splitting



- A radially asymmetric nucleus interacts with a surrounding electric field gradient (EFG)
  - *Quadrupole moment*
  - *Excited state splits into two levels*
  - *A measure of nuclear asymmetry*
- How does it affect our data?
  - *Splitting of peaks*



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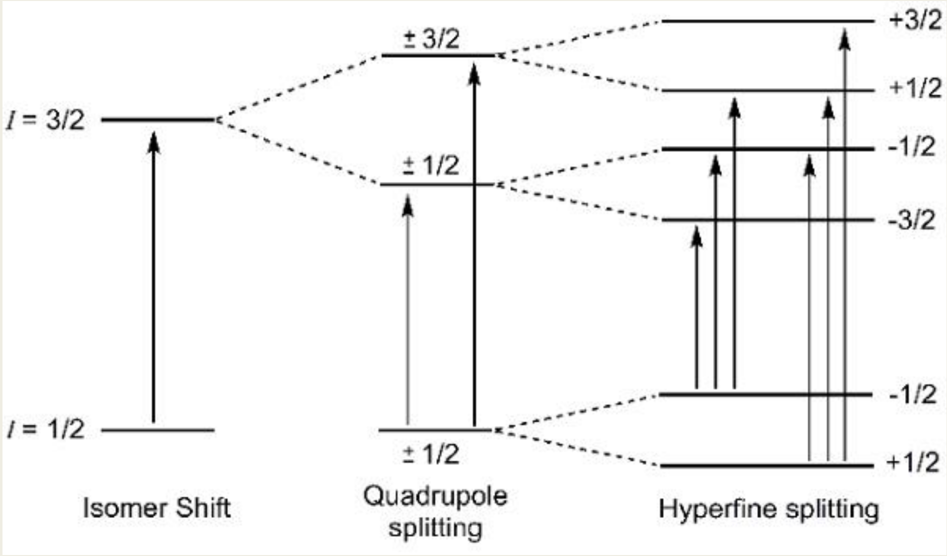
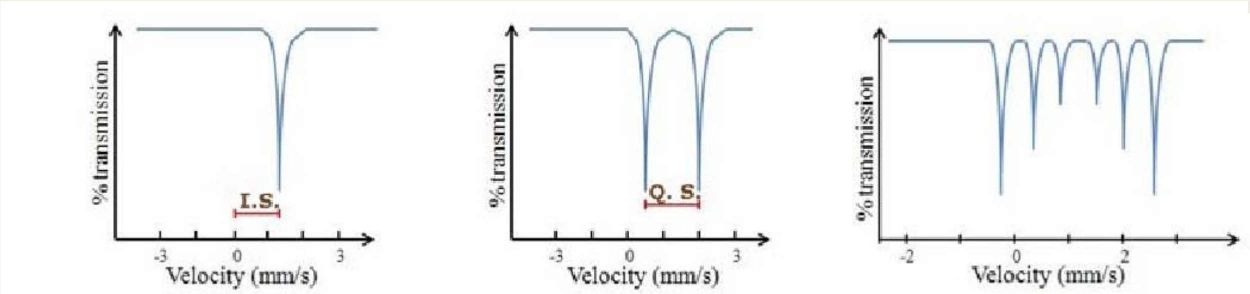
Magnetic Hyperfine Splitting

a.k.a. Zeeman Splitting or MHS





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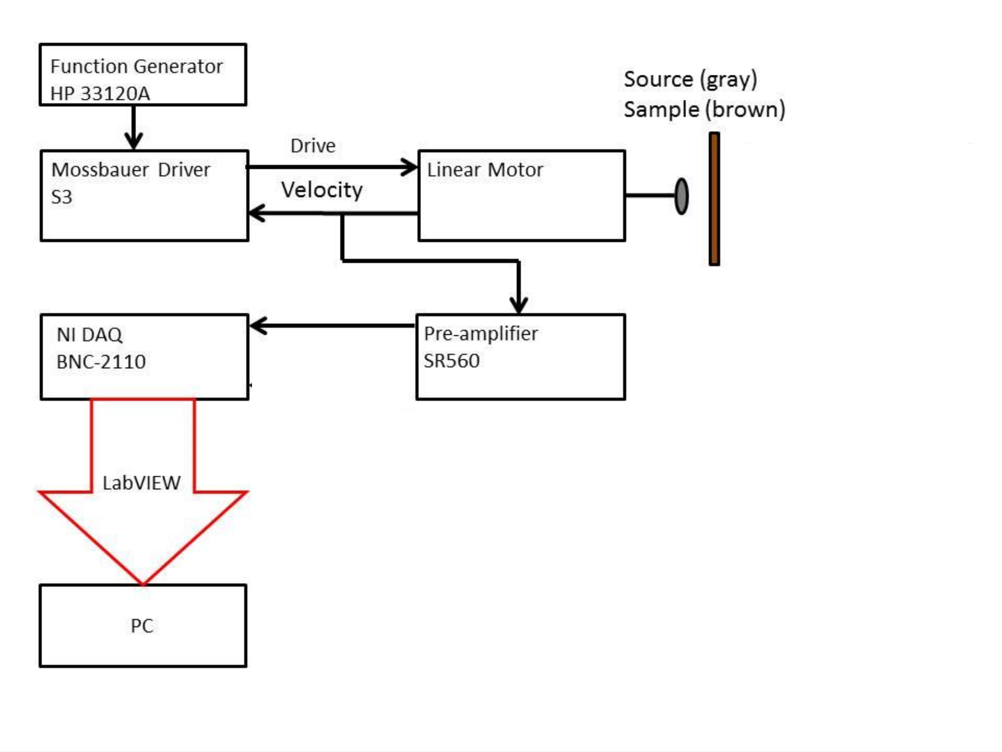
- Interaction of the nuclear magnetic dipole moment with an internal magnetic field.
  - *Magnetic ordering*
  - *Spin states*
- How does it affect our data?
  - *Characteristic splitting of peaks*

Experimental Setup – *Driver*



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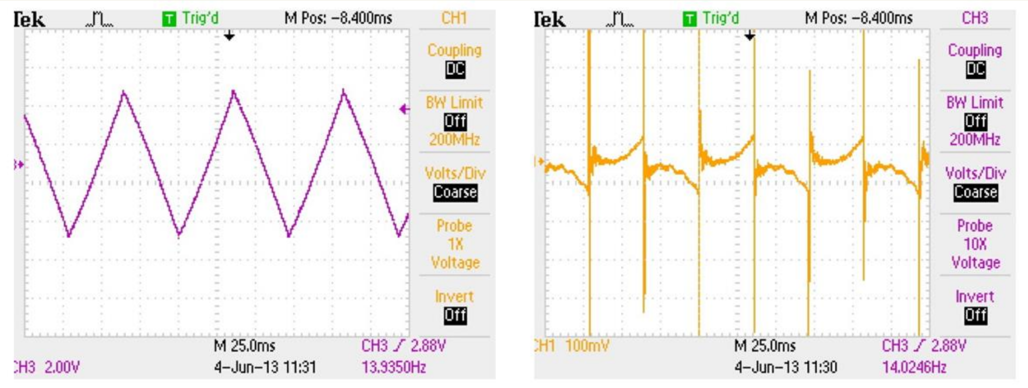
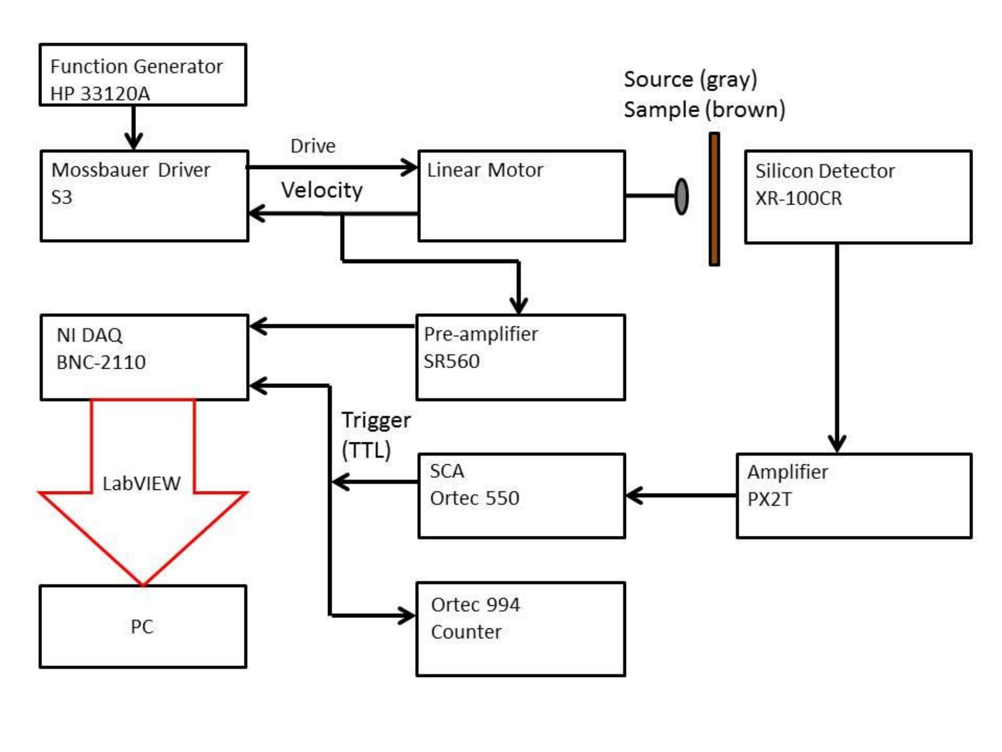
- Source is attached to a linear motor to exploit the doppler effect
  - *Driven by a 14 Hz signal passed through a driver apparatus*
- Feedback loop between motor and driver
  - *Drive = square wave trigger signal*
  - *Velocity = triangle wave*
    - Uniform in shape



Experimental Setup – *Detector*



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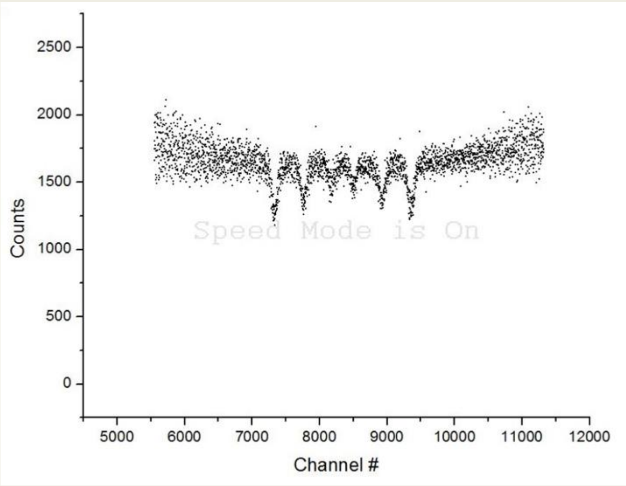
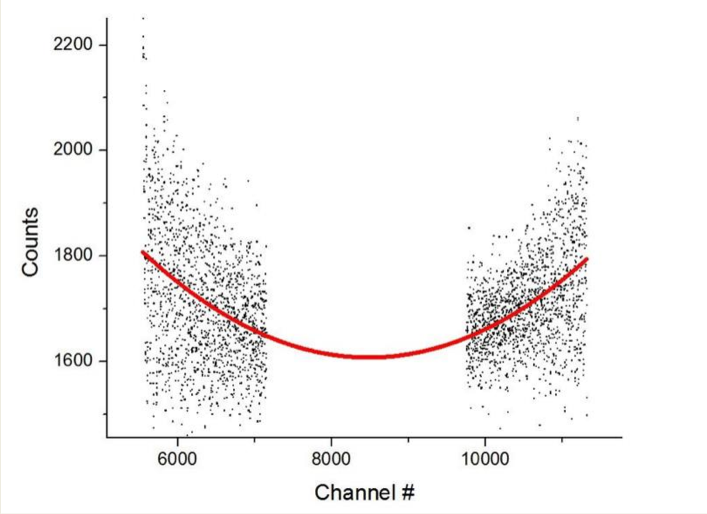
- Silicon detector
  - *Very efficient in desired range*
  - *Photoelectric effect dominates*

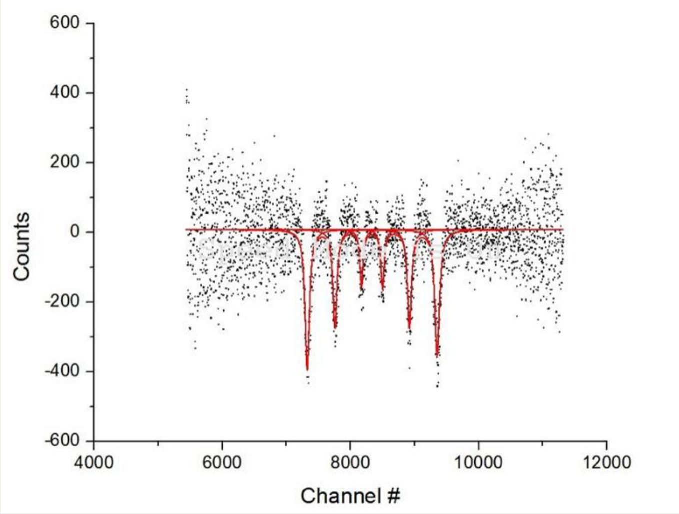
    photon → photoelectron
  - *Very small pules emitted, then amplified*
- Pass to Single Channel Amplifier (SCA)
  - *Acts as a “window”*
  - *If criteria is met, emits small square wave pulse*
- Ortec counter → crude approximator

Data Analysis – *Background Subtraction*

- Edges data are trimmed to focus on important data
- Any impurities in the Velocity signal create a quadratic trend in the data
  - *Removed by fitting data without peaks*
- Convert to physical units – <u>10.657 mm/s</u>
  - *Associate each channel number with a velocity and thus gamma ray energy*







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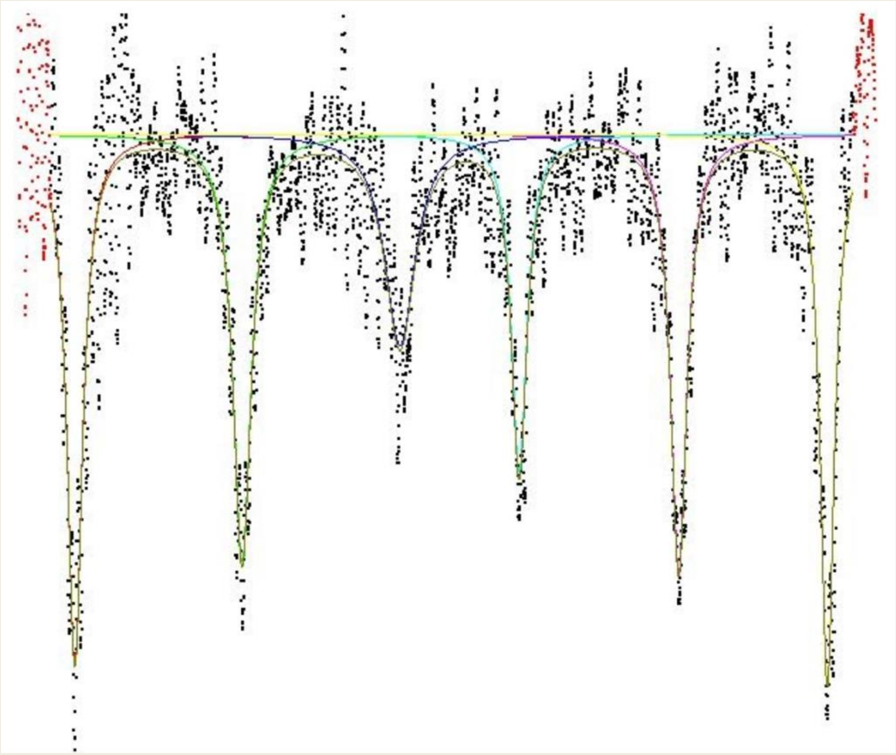
Data Analysis – Multiple Peak Fitting

- Each peak can be represented by a Lorentzian distribution
- Use Origin to:
  - *Fit peaks*
  - *Extract peak width and x axis position*
  - *Extract uncertainties*



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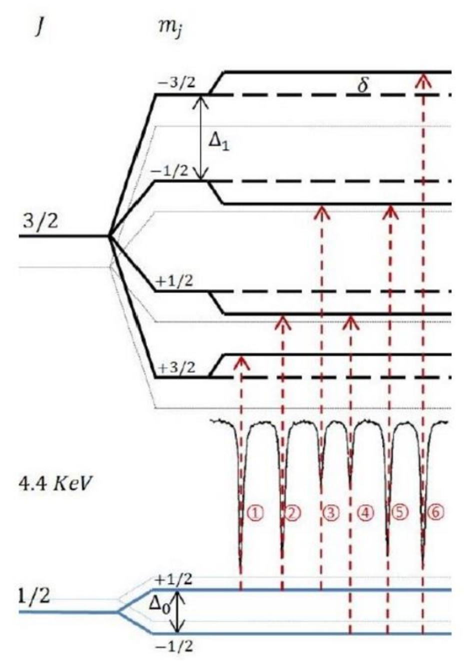
Data Analysis – Extraction of Parameters

- Peak centers tell us about hyperfine interactions!
  - ***Isomer shift:*** *Compare with True Center*
    - True Center: obtained from calibration data (iron foil target which has no isomer shift)



$$\Delta E_1 = \frac{3\Delta_1}{2} - \frac{\Delta_0}{2} + \delta + \epsilon$$

$$\Delta E_2 = \frac{\Delta_1}{2} - \frac{\Delta_0}{2} + \delta - \epsilon$$

$$\Delta E_3 = -\frac{\Delta_1}{2} - \frac{\Delta_0}{2} + \delta - \epsilon$$

$$\Delta E_4 = \frac{\Delta_1}{2} + \frac{\Delta_0}{2} + \delta - \epsilon$$

$$\Delta E_5 = -\frac{\Delta_1}{2} + \frac{\Delta_0}{2} + \delta - \epsilon$$

$$\Delta E_6 = -\frac{3\Delta_1}{2} + \frac{\Delta_0}{2} + \delta + \epsilon$$

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- Peak centers tell us about hyperfine interactions!
  - ***<u>Isomer shift:</u>*** *Compare with True Center*
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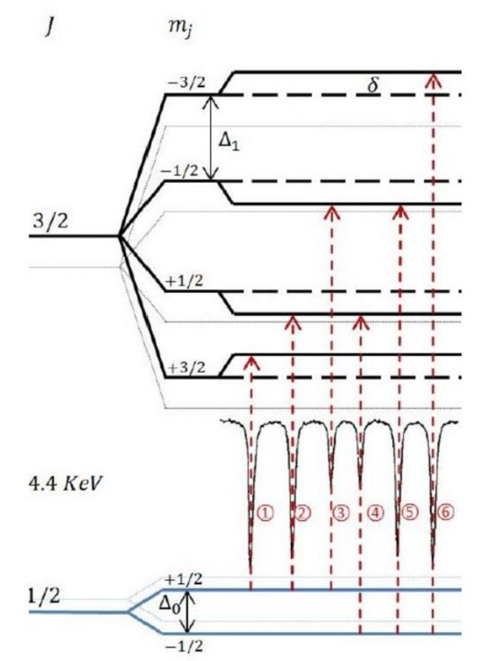
  - ***<u>Quadrupole splitting:</u>*** *Subtract Peak 5 from 6 and 1 from 2*



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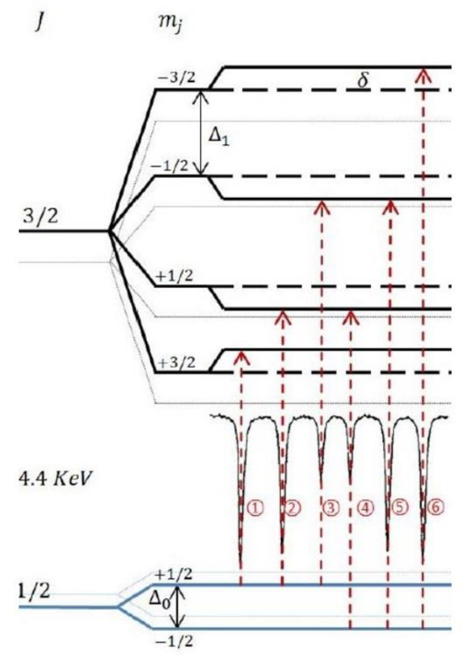
Data Analysis – Extraction of Parameters

- Peak centers tell us about hyperfine interactions!
  - ***<u>Isomer shift:</u>*** *Compare with True Center*
    - True Center: obtained from calibration data (iron foil target which has no isomer shift)
  - ***<u>Quadrupole splitting:</u>*** *Subtract Peak 5 from 6 and 1 from 2*
  - ***<u>MHS:</u>*** *Subtract peak 3 from peak five to get Δ_o and peak 4 from peak 5 to get Δ_1*



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Schedule

- Literature Review ✓
- Initial calibration of detector and SCA using MPL americium source ✓
- Calibration using weak cobalt source ✓
- Data collection (one week)
- Data analysis
- Presentation of results

References

- U.S. National Library of Medicine. (n.d.). *Periodic Table of elements - pubchem*. National Center for Biotechnology Information. PubChem Compound Database. https://pubchem.ncbi.nlm.nih.gov/periodic-table/
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