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**Science Engineering & Education Co.**

**SEE Co Model MS6S  
Student Lab Mössbauer Spectroscopy System  
User Manual**

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## 1. Overview

The SEE Co Model MS6S is a student lab grade system of hardware and software for the acquisition and analysis of Mössbauer Spectra. This manual contains a brief introduction to Mössbauer Spectroscopy plus the design and use of the MS6S system.

## 2. Introduction to Mössbauer Spectroscopy

### 2.1 Mössbauer Spectroscopy is Resonant Gamma-ray Spectroscopy (RGS)

All nuclei possess excited states, some of which are accessible from the ground state by photon absorption\*. Often the excited states of the absorber are long-lived and the range of photon energies which will resonantly excite the absorption is extremely narrow. If strong absorption is to be observed, a significant fraction of the energy of the source radiation must be within this range. Such a source may obviously consist of excited nuclei of the same isotope as the absorber. The excited nuclei may be decay products of appropriate parent nuclei. It was once thought that conservation of momentum requires the recoil of the emitting nucleus and that the photon would not have the full transition energy and hence would not resonantly excite the absorber. If the nucleus is free, the recoil momentum and energy are taken by the nucleus itself. In a solid the momentum and energy go into lattice vibrations, i.e., phonons. The temperature dependence of the absorption cross-section of Ir191 led Mössbauer (1958) to be the first to realize that a photon could be emitted with the entire solid recoiling as one rigid mass. The energy lost to the recoil in this situation is negligible and the emitted photon may resonantly excite the absorber.

Resonant Gamma-ray Spectroscopy is commonly called Mössbauer Spectroscopy in honor of its discoverer, Prof. Rudolf Ludwig Mössbauer. In 1961, at the age of 32, Prof. Mössbauer was awarded the Nobel Prize in Physics "for his researches concerning the resonance absorption of gamma radiation and his discovery in this connection of the effect which bears his name"

See [http://www.nobelprize.org/nobel\\_prizes/physics/laureates/1961/mossbauer-facts.html](http://www.nobelprize.org/nobel_prizes/physics/laureates/1961/mossbauer-facts.html)

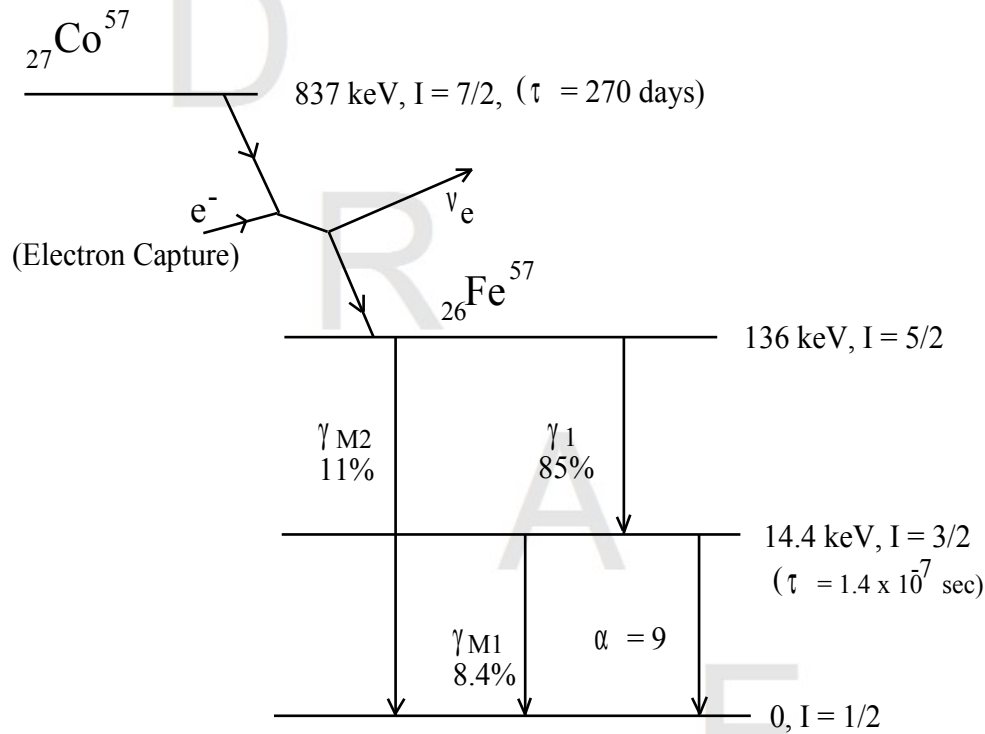
### 2.2 Nuclear Physics of Fe57

The isotope with the strongest recoilless resonant absorption is Iron-57, Fe57. Since the vast majority of the work reported in the Mössbauer literature is for iron, this discussion is limited to that isotope. **Figure 1** shows the decay of Fe57 from its parent Co57. Of all the excited Fe57 nuclei, about 10% will emit a 14.4 keV gamma ray via a magnetic dipole transition from the metastable  $I = 3/2$  state to the  $I = 1/2$  ground state ( $I$  is the nuclear spin). The ratio of recoil-free 14.4 keV photons to all the 14.4 keV photons emitted is  $f$ , the recoil-free fraction of the source.  $f$  varies with the properties of the solid and decreases monotonically with increasing temperature. The line width of the emitted radiation is limited in theory by  $\tau$ , the mean life of the  $I = 3/2$  state. In Fe57,  $\tau = 0.14$  micro sec, and the energy distribution is given by a Lorentzian with a full width at half maximum of

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\*Historically, photons that are generated by electronic transitions are called "X-rays" and photons generated by nuclear transitions are called "Gamma-rays". Both X-rays and Gamma-rays have enough energy to ionize atoms, a clear distinction from visible light photons.

$\Gamma_{\text{nat}} = 4.7 \times 10^{-9}$  eV (Lang, 1970)\*. The intensity per unit energy of the Mössbauer radiation is many orders of magnitude greater than the background radiation, and we shall henceforth refer to the gamma beam as if it were 100% Mössbauer radiation unless we specifically note otherwise. We shall also assume that the nuclear levels of the source are not split, and the energy distribution of the beam is given by a single Lorentzian.



**Figure 2.1** Energy level scheme of  $\text{Fe}^{57}$  nucleus. Mössbauer spectroscopy involves the 14.4 keV transition. Intensities are given in % of decays. “I” specifies the nuclear spin of the various level. Each level is a multiplet with  $2 \cdot I + 1$  sub-levels, i.e. the  $I = 1/2$  level is a doublet and the  $I = 3/2$  level is a quartet.

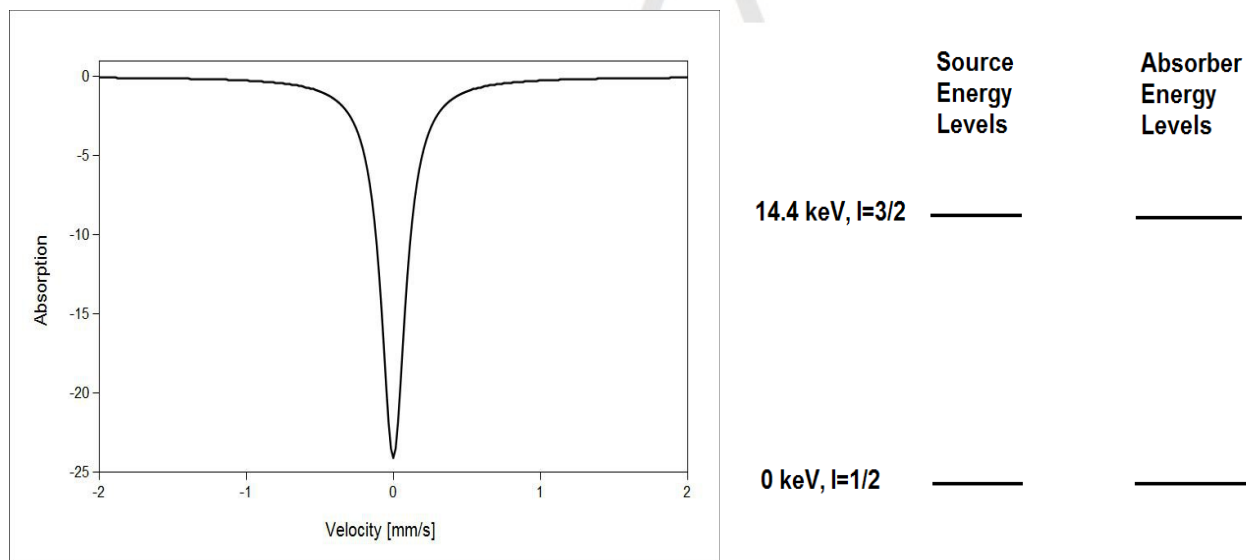
To use our Mössbauer source as a spectroscopic tool we must be able to vary its energy over a significant range. This is accomplished by Doppler shifting the energy of the gamma beam. Moving the source at a velocity of 1 mm/sec toward the sample will increase the energy of the photons by  $14.4 \text{ keV} (v/c) = 4.8 \times 10^{-8}$  eV or ten natural linewidths. The “mm/sec” is a convenient Mössbauer unit and is equal to  $4.8 \times 10^{-8}$  eV for  $\text{Fe}^{57}$ . A Mössbauer spectrometer consists of a source which may be moved relative to the sample and a counter to monitor the intensity of the beam after it has passed through the sample. The Mössbauer spectrum is a plot of the counting rate

\* The ratio of transition energy  $E_0$  to the line width  $\Gamma$  of the resonance to its is  $E_0 / \Gamma = (14.4 \times 10^3 \text{ eV} / 4.7 \times 10^{-9} \text{ eV}) = 3 \times 10^{12}$ . To put this ratio into perspective, consider that the distance from the Earth to the Moon is  $384,400 \text{ km} = 4 \times 10^{11} \text{ mm}$ . If the distance to the Moon represented  $E_0$  then  $\Gamma$  would be 0.1 mm, the thickness of a sheet of paper!

against the source velocity, i.e., the beam energy. If the sample nuclear levels are not split and the  $I = 3/2$  to  $I = 1/2$  transition energy equals that of the source, then the effective cross-section for absorption is a function of gamma energy as given by Fraunfelder (1963)

$$\sigma_{\text{eff}} = \frac{2 I^* + 1}{2 I + 1} \frac{\lambda^2}{2\pi} \frac{f'}{2(1+\alpha)} \frac{\Gamma_{\text{nat}}^2}{(E-E_\gamma)^2 + \Gamma_{\text{nat}}^2} \quad (\text{eq 1})$$

where  $I = 1/2$  and  $I^* = 3/2$  are the ground and excited nuclear spins,  $\alpha = 9.0$  is the internal conversion coefficient (ratio of betas to gammas in the 14.4 keV decay),  $\lambda = 8.61 \times 10^{-9}$  cm is the wavelength of the radiation,  $(E-E_\gamma)$  is the difference between mean incident and resonant gamma energy,  $\Gamma_{\text{nat}}$  is the energy width of the excited nucleus, and  $f'$  is the probability of recoilless absorption. The spectrum will be a single Lorentzian\* centered at  $v = 0$  with a linewidth (FWHM, Full Width at Half Maximum) of  $2\Gamma_{\text{nat}} = 0.19$  mm/sec. The linewidth is  $2\Gamma_{\text{nat}}$  since the observed line arises from the convolution of the source energy distribution and absorber cross section. As discussed below, the hyperfine interactions will split the nuclear levels of the sample and complicate the Mössbauer spectrum.



**Figure 2.2** Fe-57 Absorption Cross-section vs. Gamma-ray Energy in the case of no Hyperfine Interactions.  $E_0 = 14.41$  keV. Linewidth = 0.2 mm/s FWHM. The Doppler Shift caused by a source velocity (relative to the absorber) of [1/mm/s] is  $4.8 \times 10^{-8}$  eV

\* The Lorentzian line shape is not specific to Mössbauer Spectroscopy. This line shape is characteristic of an exponentially decaying oscillator, i.e. the Fourier Transform of the an exponentially decaying oscillator in the time domain is a Lorentzian in the frequency domain. Lorentzian line shapes are observed in many types of spectroscopy. See [http://en.wikipedia.org/wiki/Spectral\\_line\\_shape](http://en.wikipedia.org/wiki/Spectral_line_shape)

## 2.3 Hyperfine Interactions

### 2.3.1 Isomer Shift

The nucleus and its electrons interact in several ways, the most obvious being the electrostatic attraction. If the Fe<sup>57</sup> nuclear charge distribution were the same for the I = 1/2 ground state and the I = 3/2 excited state, then the electrostatic energy of the system of electrons plus nucleus would be the same for both states. In fact the excited Fe<sup>57</sup> nucleus is 0.1% smaller in radius than the ground state nucleus, which causes the Mössbauer transition energy to depend on the electron density at the nucleus. This effect produces the so-called isomer shift of the Mössbauer spectrum, which may be written as

$$\delta = K \{ \Sigma_s |\psi(0)|^2 - \Sigma_a |\psi(0)|^2 \} \quad (\text{eq 2})$$

where  $\delta$  is the isomer shift, K is a positive constant depending on the change in the nuclear radius,  $\Sigma_s |\psi(0)|^2$  is the electron density at the source nucleus, and  $\Sigma_a |\psi(0)|^2$  is the electron density at the absorber nucleus.\*

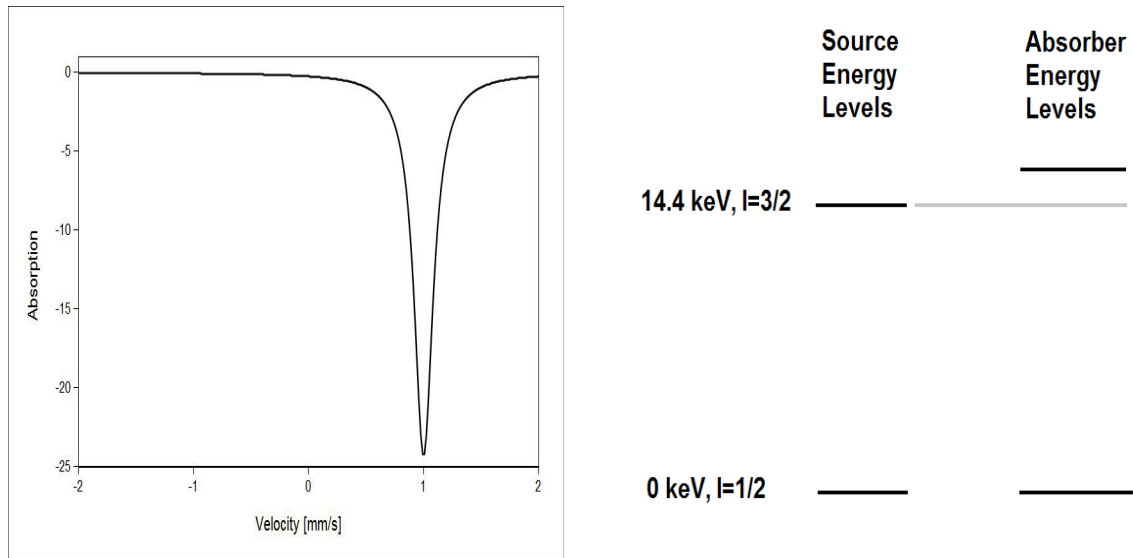
To allow isomer shifts to be measured independently of the nature of the source, the centroid of the room temperature spectrum of metallic iron has been adopted as the zero of energy and all shifts are measured relative to it. This convention yields

$$\delta = K_0 - K \Sigma_a |\psi(0)|^2 \quad (\text{eq 3})$$

where  $K_0$  is a constant. Calculations of  $\delta$  are difficult to perform, hence its most valuable application is classifying various compounds according to oxidation state, degree of covalency, and coordination number. The 1s and 2s electron density at the nucleus is independent of the chemical environment of the Fe ion. However, the 3d electrons shield the 3s electrons and cause a decrease in the electron density at the nucleus. The more delocalized the 3d electrons, the smaller the isomer shift. However, this picture is complicated by the presence of Fe 4s character in the bonding orbitals. The 4s character also reduces the isomer shift, and it is not always clear which of the two effects is dominant (Greenwood and Gibb, 1971). The electronic contribution to the isomer shift is essentially temperature independent and the observed decrease at high temperatures is due to the relativistic second order Doppler shift. This effect decreases the isomer shift approximately  $6 \cdot 10^{-4}$  mm/sec for a 1 K increase in T when the temperature is above the Debye temperature (Lang and Marshall, 1966).

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\* Only the 1s, 2s and the 3s electrons of the iron atom have a non-zero valued wave function at the nucleus. The 2p, 3p and 3d electron wave functions have a node at the nucleus.



**Figure 2.3** Single line Mössbauer spectrum with Isomer Shift = 1.0 mm/s and sketch of Fe-57 nuclear energy levels.

### 2.3.2 Electric Quadrupole Interaction

The excited ( $I = 3/2$ ) state of the  $\text{Fe}^{57}$  nucleus possesses an electric quadrupole moment, and the presence of a low symmetry electric field will tend to orient the nuclear spin.\* The interaction may be written as

$$H^Q = - (Q/6) \underline{I} \cdot \underline{V} \cdot \underline{I} \quad (\text{eq A4})$$

where  $Q$  is the nuclear quadrupole moment (a scalar),  $\underline{I}$  is the nuclear spin vector, and  $\underline{V}$  is the electric field gradient (efg) tensor. The elements of the efg tensor are

$$V_{ij} = \delta^2 V / \delta X_i \delta X_j \quad i,j = x,y,z \quad (\text{eq A5})$$

where  $V$  is the potential due to all charges outside the nucleus and the derivatives are evaluated at the nuclear site. The  $V_{ij}$  are the coefficients of the quadratic terms of a power series expansion of the potential about the nuclear site and are given in their principal axis system by

$$V_{zz} = \int \rho(\underline{r}) (3z^2 - r^2) r^{-5} d\underline{r} \quad (\text{eq A6})$$

and similar expressions.  $\rho(\underline{r})$  is the total charge density and the integral is taken over all space

\* As a classical analogy, the charge distribution of the  $I=1/2$  ground state  $\text{Fe}^{57}$  nucleus may be thought of as a sphere and that of the  $I=3/2$  excited state as a prolate ellipsoid, e.g. an American football.

outside the nucleus. Also,  $\underline{V}$  is real, symmetric, and traceless. Hence in the frame in which  $\underline{V}$  is diagonal,

$$H^Q = (Q/4)V_{zz}[I_z^2 - 5/4 + (\eta/3)(I_x^2 - I_y^2)] \quad (\text{eq A7})$$

$$\eta = (V_{xx} - V_{yy})/V_{zz}$$

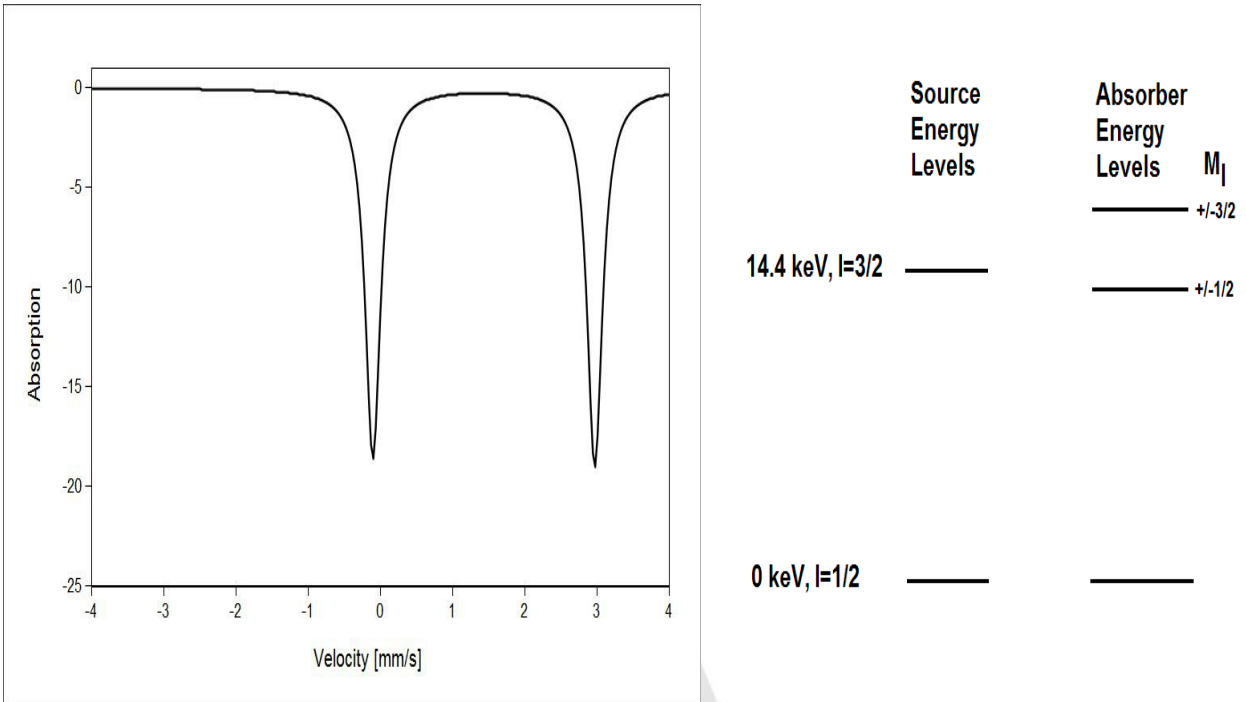
where  $\underline{x}$ ,  $\underline{y}$ , and  $\underline{z}$  denote the principal axes of the efg and  $\eta$  is called the asymmetry parameter. By convention,  $\underline{x}$ ,  $\underline{y}$ , and  $\underline{z}$  are chosen so that  $|V_{zz}| \geq |V_{yy}| \geq |V_{xx}|$ , which forces  $0 \leq \eta \leq 1$ . Note,  $\underline{V}$  has five independent elements. Two of these are  $V_{zz}$  and  $\eta$  and the other three are the Euler angles locating the efg principal axis system relative to some experimental frame.

Reversal of the sign of the nuclear spin will not change the nuclear charge distribution; therefore the efg will not completely lift the fourfold degeneracy of the  $I = 3/2$  states. The quartet will be split into two doublets, while the  $I = 1/2$  states will remain degenerate. Since two transition energies are now possible, two absorption peaks will appear in the Mössbauer spectrum. Diagonalizing  $H^Q$ , we find that the difference in energy of the two peaks to be

$$\Delta E_Q = (Q/2) V_{zz} (1 + \eta^2/3)^{1/2} \quad (\text{eq A8})$$

If the recoil-free fraction does not depend on the orientation of the  $\gamma$ -beam relative to the Fe site, a sample with randomly oriented sites in zero applied field will produce a symmetric quadrupole pair. Since the sign of  $\Delta E$  can not be determined from such a spectrum, the absolute value of  $\Delta E_Q$  is usually called "the quadrupole splitting." The sign of  $\Delta E$  is usually referred to as "the sign of  $V_{zz}$ " and is more difficult to measure than the quadrupole splitting.





**Figure 2.4** Mössbauer spectrum showing one Quadrupole Pair with Isomer Shift  $\delta = 1.4$  mm/s and Quadrupole Splitting  $\Delta E_Q = 3.0$  mm/s plus sketch of Fe-57 nuclear energy levels.

### 2.3.3 Magnetic Hyperfine Interaction

The  $Fe^{57}$  nucleus possesses a magnetic moment and its energy levels will be perturbed by the local magnetic field. In the presence of a field  $\underline{H}$ , the interaction is

$$H^M = -g_N \beta_N \underline{I} \cdot \underline{H} \quad (\text{eq A9})$$

where  $\beta_N$  is the nuclear magneton and  $g_N$  is the nuclear gyromagnetic ratio.  $g_N$  has the values  $0.1806 \pm 0.0014$  and  $-0.1033 \pm 0.0008$  in the ground and excited states, respectively (Lang, 1970).  $\underline{H}$  will be the sum of an applied field with the internal field of the unpaired electrons. Depending on the magnetic properties of the sample, the internal field may or may not equal zero in the absence of the applied field. A more explicit expression of the magnetic interaction is

$$H^M = P \sum_k \{ \underline{l}_k \cdot \underline{I} + 3(\underline{r}_k \cdot \underline{s}_k)(\underline{r}_k \cdot \underline{I}) - (\underline{s}_k \cdot \underline{I}) - \kappa(\underline{s}_k \cdot \underline{I}) \} - g_N \beta_N \underline{I} \cdot \underline{H}^{\text{app}}$$

$$P = 2 g_N \beta \beta_N \langle r^{-3} \rangle_{\text{eff}} \quad (\text{eq A10})$$

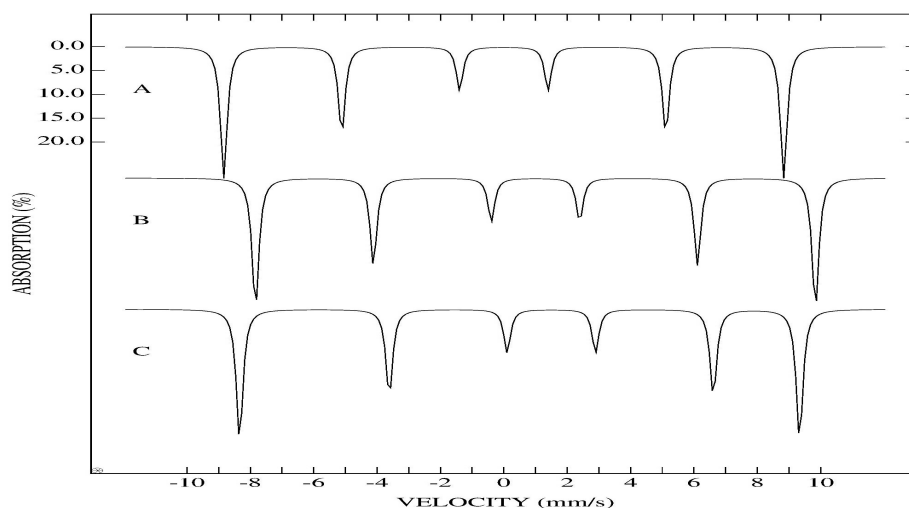
where the sum is over the unpaired electrons,  $\underline{l}_k$  is the orbital angular momentum on the  $k^{\text{th}}$  electron

and  $\beta$  is the electron magneton. It is convenient to think of  $H^M$  in terms of an effective field acting on the nuclear moment. The effective field is

$$\begin{aligned} \underline{H}^{\text{eff}} &= \underline{H}^{\text{app}} + (p/g_N\beta_N)\Sigma\{ \underline{L}_k + 3(\underline{r}_k \cdot \underline{s}_k)\underline{r}_k - \underline{s}_k - \kappa \underline{s}_k \} \\ &= \underline{H}^{\text{app}} + \underline{H}^{\text{int}} \end{aligned} \quad (\text{eq A11})$$

The first term of  $\underline{H}^{\text{eff}}$  is the applied field. The second is the field at the nucleus due to the orbital motion of the electrons. The third and fourth describe the field due to the dipole moment of the electrons. The last term is the Fermi contact term which accounts for any net polarization of the electronic spin at the nucleus. The polarization of s electron spin at the nucleus stems from the exchange interaction between the 3d electrons and the s electrons. Any admixture of 4s character into the 3d wave functions would also contribute to the contact field. To a good approximation the net spin at the nucleus will be proportional to  $\Sigma_k \langle \underline{s}_k \rangle$ , the sum being taken over 3d electrons.

P and  $\kappa$  depend on the radial part of the wave functions and on shielding effects which are difficult to calculate. Values have been determined empirically for many complexes. Lang and Marshall (1966) quoted values of  $P/g_N\beta_N = -620$  kG and  $\kappa = 0.35$  for high- and low-spin ferric heme complexes.  $P\kappa/g_N\beta_N$  is the effective field per unit spin due to the contact term. For the ferric heme complexes it would be -220 kG. For a variety of high-spin ferrous inorganic compounds the survey of Varret (1976) shows  $P\kappa/g_N\beta_N$  to be approximately -250 kG. Edwards et al. (1967) found  $P\kappa/g_N\beta_N$  for high-spin ferrous tetrahedral compounds to be reduced to -160 kG by covalency effects.



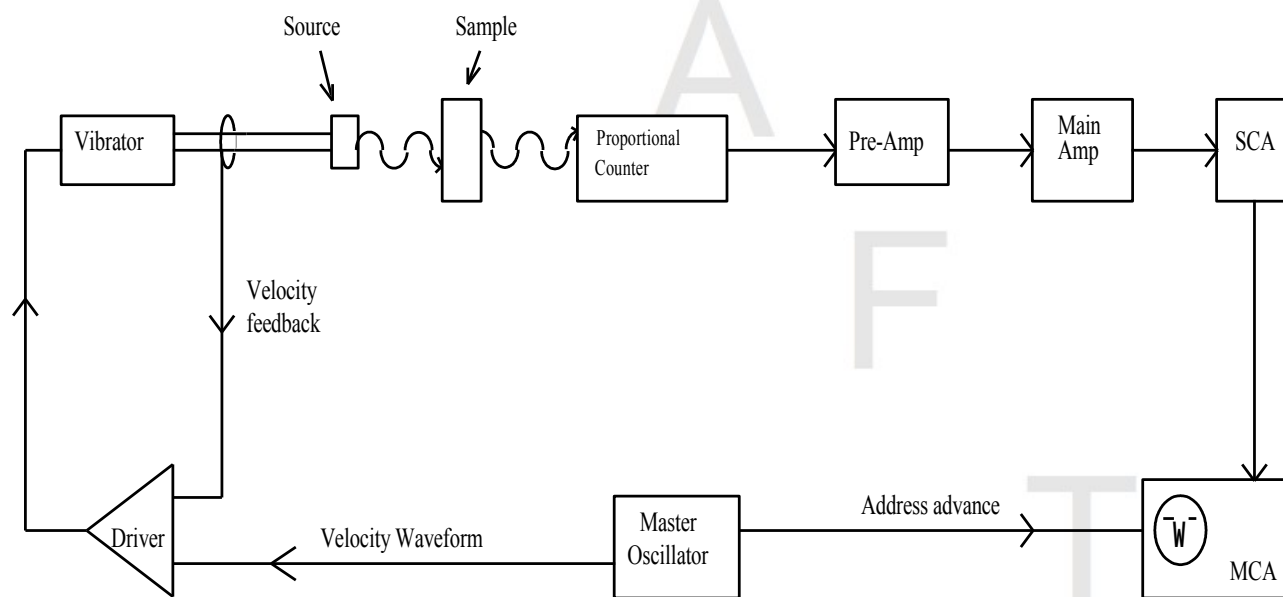
**Figure 2.5** Mössbauer spectra with  $H^{\text{int}} = 55$  T, typical for Iron oxides. A)  $\delta = 0$  mm/s and  $\Delta E_Q = 0$  mm/s, B)  $\delta = 1.0$  mm/s and  $\Delta E_Q = 0$  mm/s, C)  $\delta = 1.0$  mm/s and  $\Delta E_Q = -1.0$  mm/s,

### 3. Experimental Setup and Operation

#### 3.1 Overview

The Mössbauer spectra are usually recorded in the transmission geometry using a constant acceleration spectrometer operated in connection with a multichannel analyzer in the time scale mode. The source is kept at room temperature and consists of approximately 20 mCi of  $\text{Co}^{57}$  diffused into rhodium or palladium foil. The spectrometer is calibrated against a metallic iron foil and zero velocity is taken as the centroid of its room temperature Mössbauer spectrum. In such calibration spectra, line widths of about 0.23 mm/s were normally observed. The duration of a typical run is a few hours for non-biological compounds and 24 hours for proteins. A typical spectrometer is depicted schematically in Figure 6.

As shown in Figure A2, both the source velocity and the address of the active channel of the multichannel analyzer (MCA) are controlled by the master oscillator. The oscillator synchronizes the source acceleration and the sweep of the memory registers, causing the active channel address to be a linear function of the velocity. In other words, the pulses counted while the source is at a particular velocity are always stored in a particular register.



**Figure 3.1** Schematic diagram of the Mössbauer spectrometer.

The master oscillator output is a rounded sawtooth or triangular wave and is applied to one of the driver inputs. The driver is essentially a difference amplifier with its output applied to the vibrator. A pick-up coil on the vibrator supplies a voltage proportional to the velocity of the source and is connected to the second input of the driver. The driver and vibrator form a tightly coupled, electromechanical, negative feedback loop, forcing the source velocity to be directly proportional to the master oscillator signal. The desired velocity range is selected by scaling the master oscillator signal before it is applied to the driver.

The gas proportional counter contains primarily krypton or argon plus a small amount of methane quench gas. A 14.4 keV photon entering the chamber through the Mylar window ionizes some of

the Ar or Kr atoms, the creation of each ion/electron pair requiring about 30 eV. The positive ions drift to the outer wall and the electrons to the center wire. The accelerating electrons ionize more atoms, the effective gas gain being approximately 1000. The methane suppresses the random motion of the electrons, thus shortening the travel time to the center wire and the rise time of the signal pulse. The overall efficiency of a Harwell Argon counter is 60% at 14.4 keV, and a 14.4 keV photon will cause about  $10^6$  ion pairs to be created. The 122 keV and 136 keV photons entering the counter Compton scatter and produce a broad background of lower energy signals. The single channel analyzer (SCA) discriminates against most of these, but about 20% of the pulses falling in the 14.4 keV window of the SCA are from this Compton background.

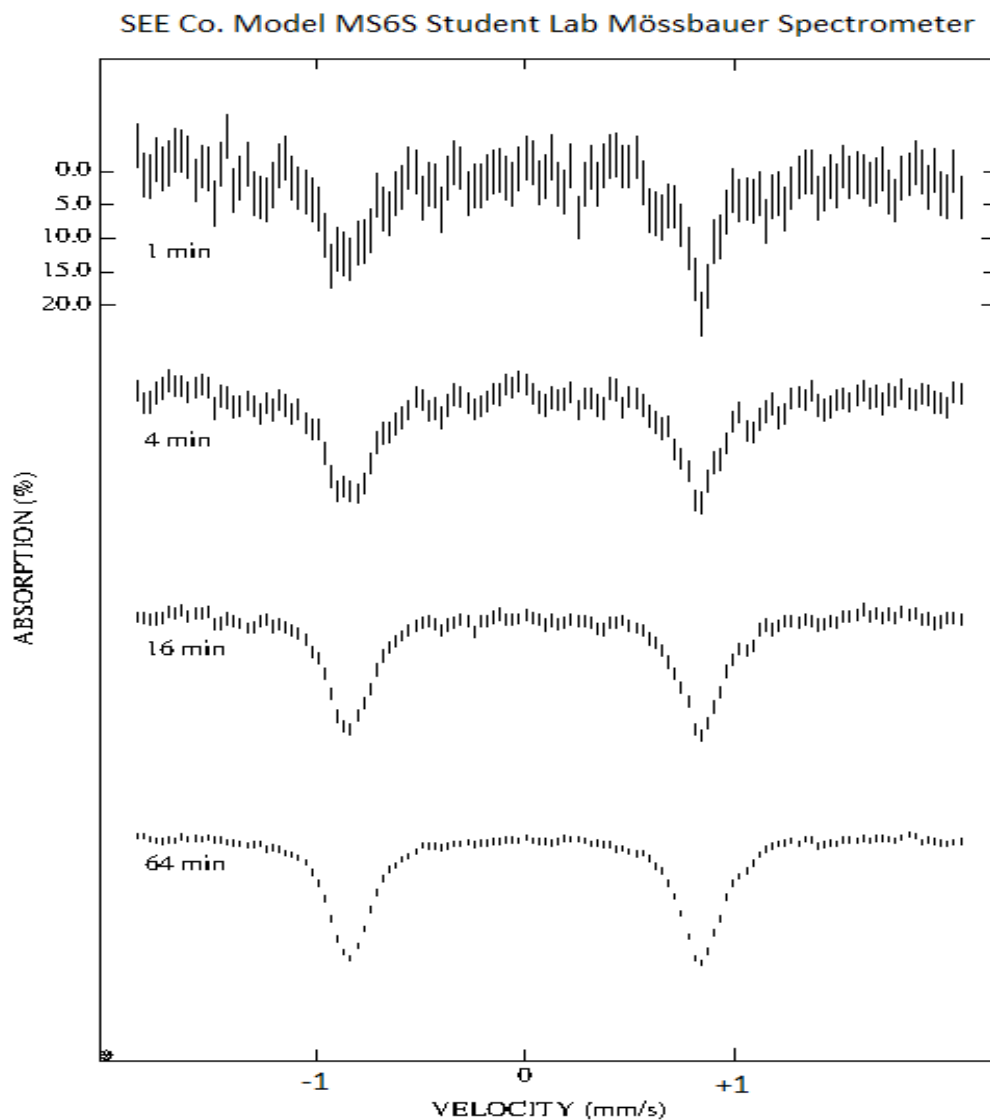
The charge pulse from the counter is amplified and differentiated by the preamp which is mounted as close to the counter as possible. Further amplification of the pulse is provided by the main amplifier and its output is applied to a single channel analyzer. The SCA is set to discriminate against the non-14.4 keV signals. The signals accepted by the SCA are added to the current channel of the MCA.

The observed linewidths are larger than the intrinsic width of 0.19 mm/s since some broadening is always introduced by inhomogeneity of the environment of the iron in the source and sample. Additional broadening arises from imperfections in the source driving mechanism. Even so, typical observed linewidths obtainable in the lab are 0.23 mm/s which is close to the minimum predicted by the uncertainty principle.

We now consider the time required to record a Mössbauer spectrum. A typical sample will have 1 mg/cm<sup>2</sup> of iron in the gamma-ray beam. For purposes of illustration, we assume the spectrum has two absorption peaks. If the Fe<sup>57</sup> is present in its natural 2% abundance, there will be  $2 \times 10^{17}$  Fe<sup>57</sup> nuclei/cm<sup>2</sup>. The peak resonant cross section is approximately  $2 \times 10^{-18}$  cm<sup>2</sup> (Frauenfelder, 1963). We divide this value by 8 because of the finite source linewidth, the two absorption peaks of the spectrum, and the estimated product of the recoil free fraction of the source and absorber. The observed intensity will be 5%. The spectrometer will typically divide the velocity scale into 256 channels. For a 20mCi source the total counting rate is usually about 20,000 counts/s of which about 85% will be 14.4 keV radiation. The spectrometer duty cycle is about 66% and r, the 14.4 keV counting rate per channel, is typically 44 counts/s/channel. The signal-to-noise ratio, S/N, is given by

$$S/N = a_p [r T]^{1/2} \quad (\text{eq A12})$$

where  $a_p$  is the peak absorption intensity and T is the running time. If we want a S/N of 40, then, for  $a_p = 0.05$ , T will be 14,500 s or 4 hours. **For a given S/N, the run time is inversely proportional to the square of the peak absorption.**

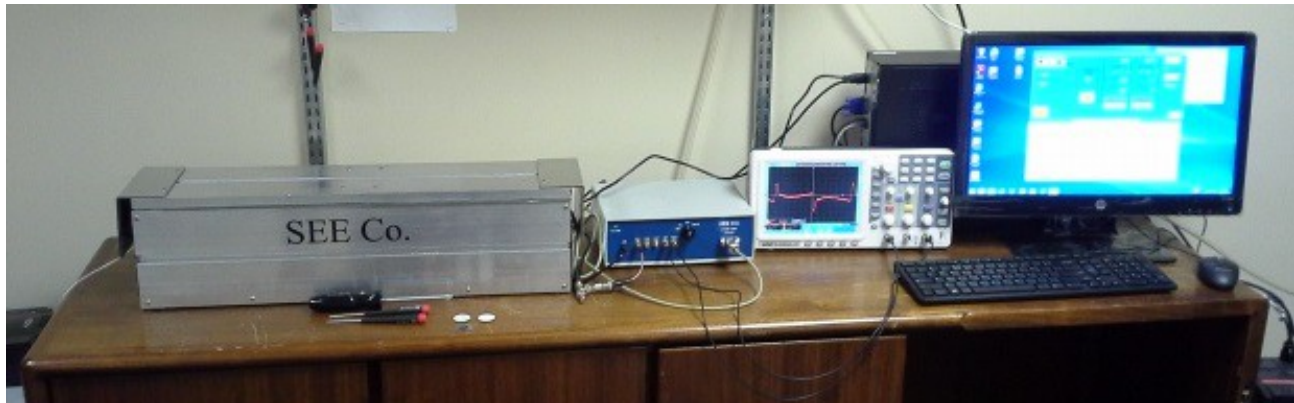


Mössbauer Spectra of innermost lines of Iron Metal  
with Acquisition Time specified.

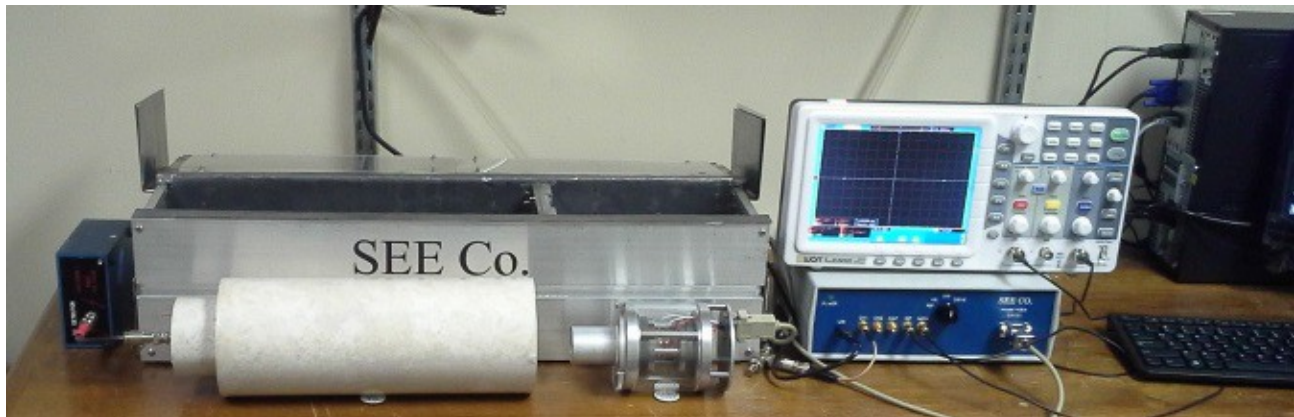
Source activity 0.8 mCi. Sample 27  $\mu\text{m}$  thick Fe metal foil.

**Figure 3.2** Mössbauer spectra showing lines 3 and 4 of the sextet of a 27  $\mu\text{m}$  thick natural Iron foil at room temperature. Data acquisition times are noted. The Co-57 source activity was 0.8 mCi. The sample had diameter 18 mm and was 35 mm from the source.

Figure 3.3 shows a complete MS6S spectrometer.



A



B

### Figure 3.3

- A) SEE Co. MS6S Student Lab Mössbauer Spectrometer recording a spectrum with the sample at room temperature. The gas counter and velocity transducer are inside the Pb-lined Al case.
- B) Components shown are (left to right) W202S Gamma-ray Spectrometer, Argon/Methane Gas Flow Proportional Counter, VT6S Velocity Transducer, W304S Resonant Gamma-ray Spectrometer and 30 MHz Oscilloscope. The Co-57/Rh Mössbauer gamma-ray source is mounted on the end of the VT6S shaft inside the radiation shield between the VT6S and the counter. The MS Windows PC controls the W202S and the W304S via the USB interface.

Figure 3.4 shows a sketch and diagram of the MS6S spectrometer and also shows a block diagram of the internal components of the W202S and the W304S.

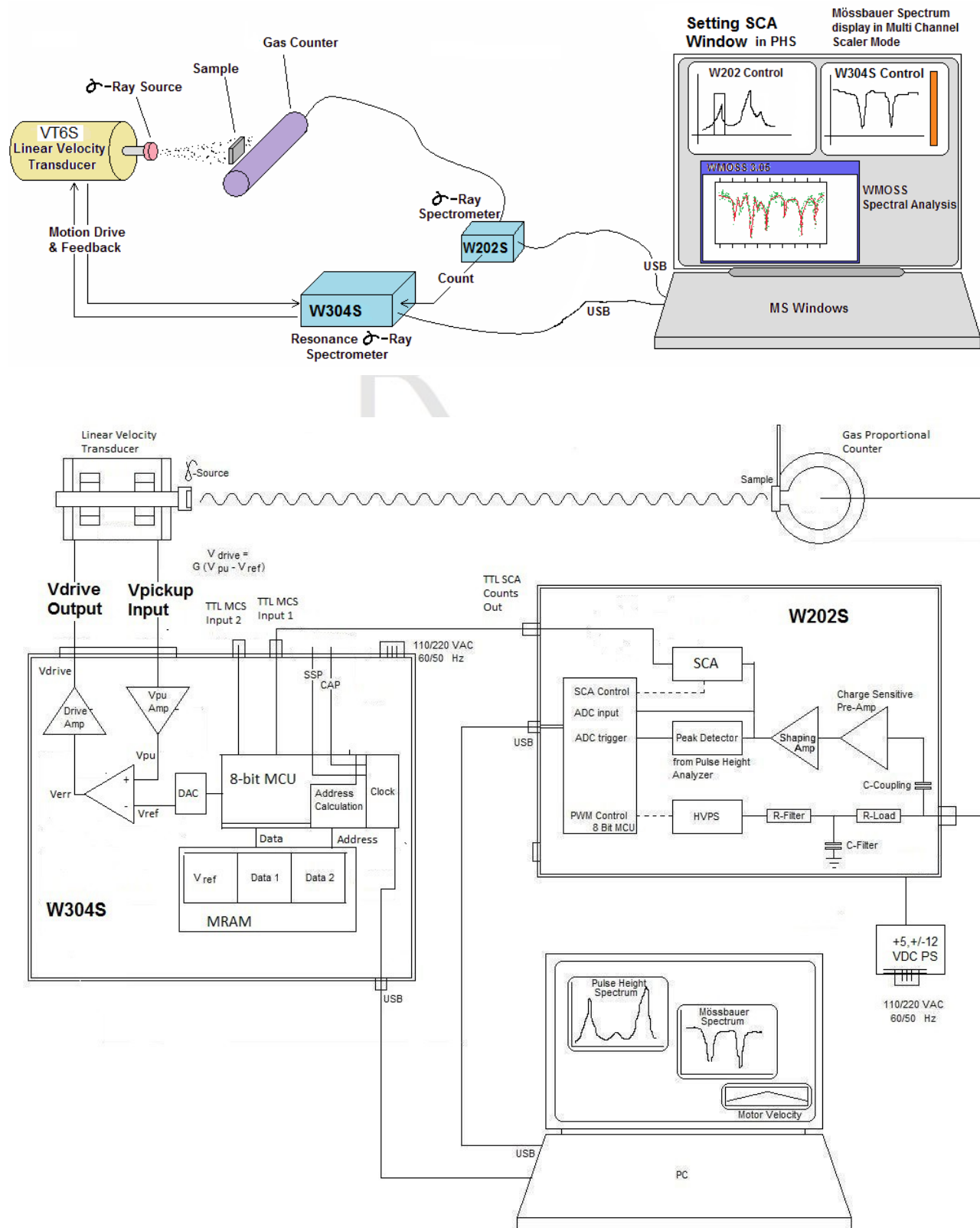


Figure 3.4 Sketch and Diagram of the SEE Co. MS6S Mössbauer Spectroscopy System



<b>Cables shown in Figure 9</b>
W304SS Power 100 – 240 VAC, 50 – 60 Hz
W202S Power +5, +/-12 VDC from external PS
2X USB Type Mini B / Type A
Velocity Transducer Cable
Counts In from W202S SCA Output to W304SS Channel 1 MCS Input
SHV-MHV coax cable from W202S to gas counter



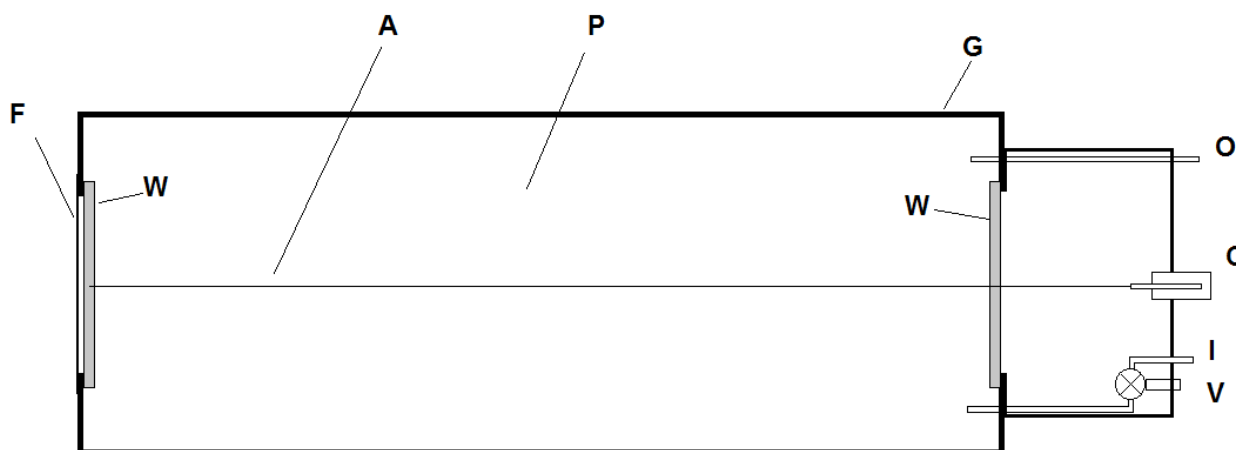
### I. 3.2 Gas Flow Proportional Counter Gamma-ray Detector

Gas proportional counters have been used as gamma-ray detectors for over 100 years.<sup>+</sup> A detailed review is the classic paper “Energy Measurements with Proportional Counters” by D. West (Prog Nucl Phys 3, pp 18-62, 1950). Although many solid state gamma detectors have since been developed, gas counters are still widely used and their development continues.\*

A cylindrical, single-wire, proportional gas flow counter is a deceptively simple device. While such a gamma-ray detector can be constructed from materials found at any hardware store, its function involves much Physics, including

- Photo-electric events
- Compton Scattering
- Electrostatics
- X-ray emission
- Conversion electron emission
- Elastic and inelastic scattering of electrons
- Ionization by electron impact and the statistics of energetic charged particle motion in a gas.

**Proper use of the SEE Co MS6S Mössbauer spectrometer requires a basic understanding of the gas proportional counter.**

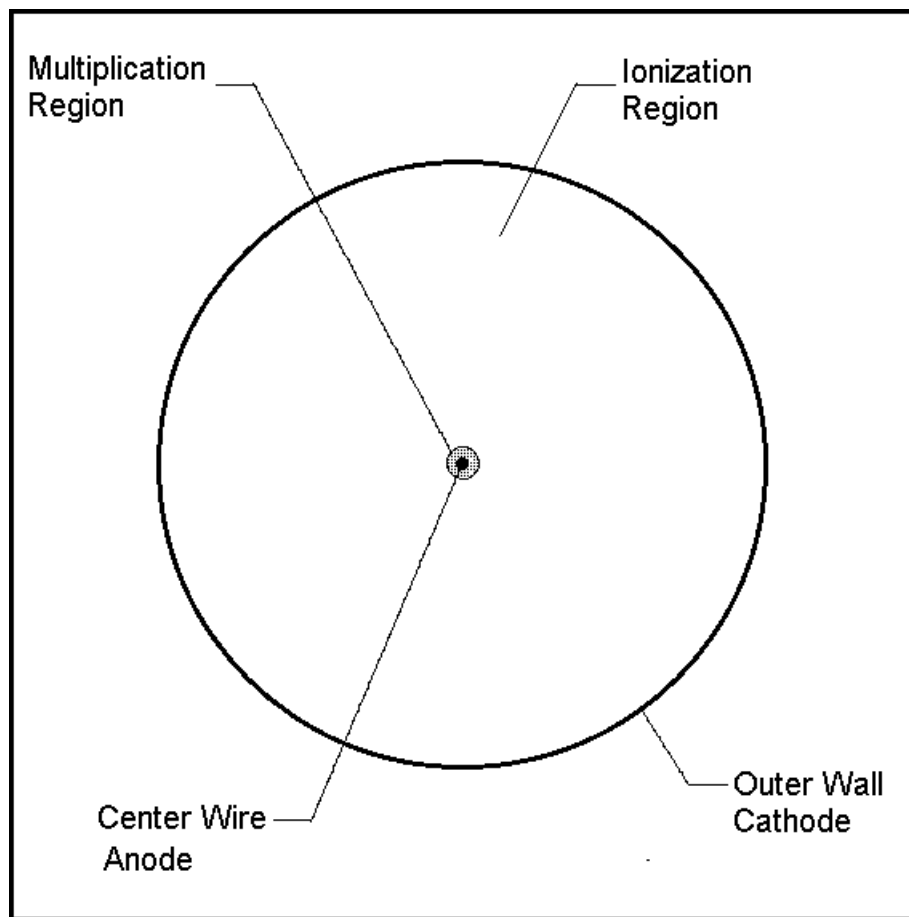


**Figure 3.5** Sketch of SEE Co Model GFC6S End-Window Gas Flow Proportion Counter

- A. Center wire anode, 50 microns diameter
- C. Electrical Connector for High Voltage Power Supply and Charge Sensitive Pre-Amplifier
- G. Grounded metal case cathode, 100 mm diameter x 300 mm length
- F. Aluminized Mylar Front Cover over entrance window.
- W. Plastic Window, Insulator
- P. P10 Gas, 90% Argon + 10% Methane, ~ 1 atms pressure.
- I. Gas Inlet
- O. Gas Outlet
- V. Flow rate control valve

<sup>+</sup> <http://www.science.uwaterloo.ca/~cchieh/cact/nuctek/interactdetector.html> ,  
[http://en.wikipedia.org/wiki/X-ray\\_astronomy\\_detector](http://en.wikipedia.org/wiki/X-ray_astronomy_detector) and  
<http://ebooks.cambridge.org/chapter.jsf?bid=CBO9780511735554&cid=CBO9780511735554A009>

\* <http://cerncourier.com/cws/article/cern/35458>



**Figure 3.6** Radial Geometry of a single-wire proportional counter.

The geometry of the GFC6S counter is illustrated in Figures 3.5 and 3.6. The interior of the counter is filled with an inert gas (He, Ne, Ar, Kr or Xe) plus a small amount of a “quench gas” such as CO<sub>2</sub> or Methane. An inert gas is required since free electrons and positive ions must travel throughout the volume of the counter without unwanted chemical reactions taking place. The SEE Co GFC6S counter is designed to use “P10” gas, a 90% Argon and 10% Methane mixture. Argon is chosen because it is inexpensive and, as discussed below, has gamma absorption properties well matched to the radiation from the Co-57 Mössbauer gamma source.

The counter's function is dominated by:

- 1) the voltage applied between the case and the center wire and
- 2) the mean free path of an electron moving in the gas.

Given the geometry of the counter, the applied voltage will determine the E-field within the counter. The electron mean free path determines how much work is done on the electron by the electric field before the electron collides with a gas atom or molecule.

Ignoring end effects and applying Gauss's Law we can solve for the E-field inside a cylindrical capacitor.

$$E(r) = V / ( r \ln(b/a) )$$

where

- V = voltage difference between the center wire and the outer wall
- r = radial distance from the center of the cylinder
- b = radius of the outer wall
- a = radius of the center wire

For  $V = 2,000$  Volts,  $a = 25$  microns and  $b = 50$  mm, the E-field values near the outer wall and the center wire are:

$$E(b) = 2,000V / ( 50 \text{ mm} * \ln(50 / 0.025) ) = 5.2 \text{ V / mm}$$

$$E(a) = 2,000V / ( 0.025 \text{ mm} * \ln(50 / 0.025) ) = 10,500 \text{ V / mm}$$

The minimum kinetic energy required for an electron impact to ionize an Argon atom is 15 eV. The mean free path of an electron in Argon gas at STP is approximately 3 microns = 0.003 mm. The minimum E-field required to do 15 eV of work over a distance of 0.003 mm is 5,000 V/mm. The E-field in the GFC6S counter with 2,000 V applied will be larger than 5,000 V/mm in the region where  $r < 2a$ , i.e. within 25 microns of the surface of the center wire.

As depicted in Figure 3.6, the proportional gas counter has two distinct regions: the large Ionization Region with  $r > 2a$  and the very small Multiplication Region with  $r < 2a$ .

A gamma-ray entering the gas may be absorbed via a photoelectric event transferring all its energy to an electron of an Argon atom. That electron will impact other Ar atoms causing more ion pairs to be created. If the gamma-ray energy is 14,400 eV and the average energy to ionize an Ar atom is 30 eV then approximately 500 ion pairs will be created. The free electrons will move quickly toward the positively charged center wire and the  $\text{Ar}^+$  ions will move much more slowly toward the negatively charged outer wall.

The role of the Methane gas is to reduce the travel time of the electrons. If the gas were pure Argon, the free electrons in the Ionization Region would experience elastic collisions causing their progress toward the center wire to be much like random diffusion. The presence of the Methane provides inelastic scattering via excitation of low energy molecular vibrations. The Methane is called a “quench gas” since it quenches the random motion of the free electrons. The result is that all the primary electrons arrive at the Multiplication Region as a better defined group and the output pulse will have a much sharper rise time.

Once the primary electrons reach the Multiplication Region, they will gain enough kinetic energy to create secondary ion pairs. The secondary electrons will create tertiary electrons and so on. The result of this chain reaction is the total amount of ion pairs created in the Multiplication Region will be on the order of 1000X the number of primary ion pairs created in the Ionization Region. This factor of 1000X is called the “Gas Gain” and enables the detection of low energy ( $< 1$  MeV) gamma-rays and X-rays using inexpensive electronics.

If the applied voltage was lowered so that there was no Gas Gain then the output pulse of the counter would be too small to be observed given the noise level of the electronics. Conversely, if the applied voltage was raised too much the Gas Gain would become non-linear and the counter output pulse would be very large but not proportional to the incident gamma-ray energy. Such a non-linear gas counter is called a “Geiger Counter”.

For Mössbauer spectroscopy, it is important that the Gas Gain is linear and the counter output pulse is proportional to the energy of the incident gamma-ray. The Co-57 source emits several different energy gamma-rays and X-rays. A proportional counter resolves the different energies allowing selection of the gamma-ray of interest. This selectivity greatly improves the signal-to-noise of the gamma-ray counting and reduces the data acquisition time.

Due to the random fluctuations of the charged particles' motion in the gas, the theoretical minimum energy resolution of a gas counter is ~10%. The line widths of the peaks in the Pulse Height Spectrum affect the signal to noise ratio of the gamma counting, but has nothing to do with the line shapes in the Mössbauer spectrum.

### 3.3 W202S Gamma-ray Spectrometer

The SEE Co. Model W202S Gamma-ray Spectrometer\* is a compact, highly integrated system providing both Pulse-Height Analysis (PHA) and dual Single-Channel Analysis (SCA). The user interface is provided via a USB link to a Windows PC. The SCA TTL pulse output may be routed to the input of a separate Multi Channel Scalar (MCS), such as the SEE Co W304S. The discussion below assumes that the W202 is to be used as a part of a Mössbauer Spectrometer with a gas proportional counter. Many other applications of the W202 are possible.



**Figure 3.7** Front and Rear views of the W202S Gamma-ray Spectrometer. Case dimensions are 5 cm x 10 cm x 12 cm.

#### Block Diagram and External Connections

See Figure 3.7 on next page.

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\*A student should clearly understand the difference between a “Gamma-ray Spectrometer” and a “Resonant Gamma-ray Spectrometer”.

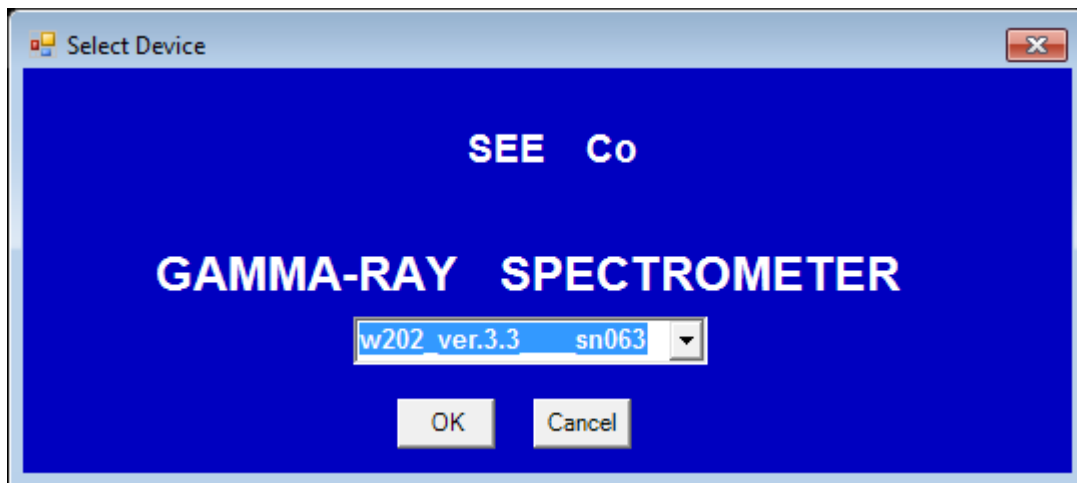


## Setup & Operation

Before plugging power supply into 120-240 VAC, connect all cables as illustrated in Fig. 3.8. Power should be disconnected anytime a cable is inserted or removed from the W202 unit.

With all cables connected, plug power supply into 120-240VAC source. Often this would be a battery backed-up UPS. The W202 has no on/off switch.

If a computer was purchase with the W202, the W202 user interface software will be already installed on that MS Windoes PC. If not, see the W202 PC software documentation for installation instructions. After the PC software is properly installed, execute the program W202.exe by double clicking on the W202 icon on the PC desktop. If all cable connections are correct, you will see the dialog box shown in Fig. 3.9. The drop down list will show all W202 units that are connected to the PC. Select the unit you require and then click OK. Clicking Cancel will terminate the W202 program.



**Figure 3.9** Initial “Select Device” dialog box . Clicking the down arrow will display a list of all W202 units connected to the PC.

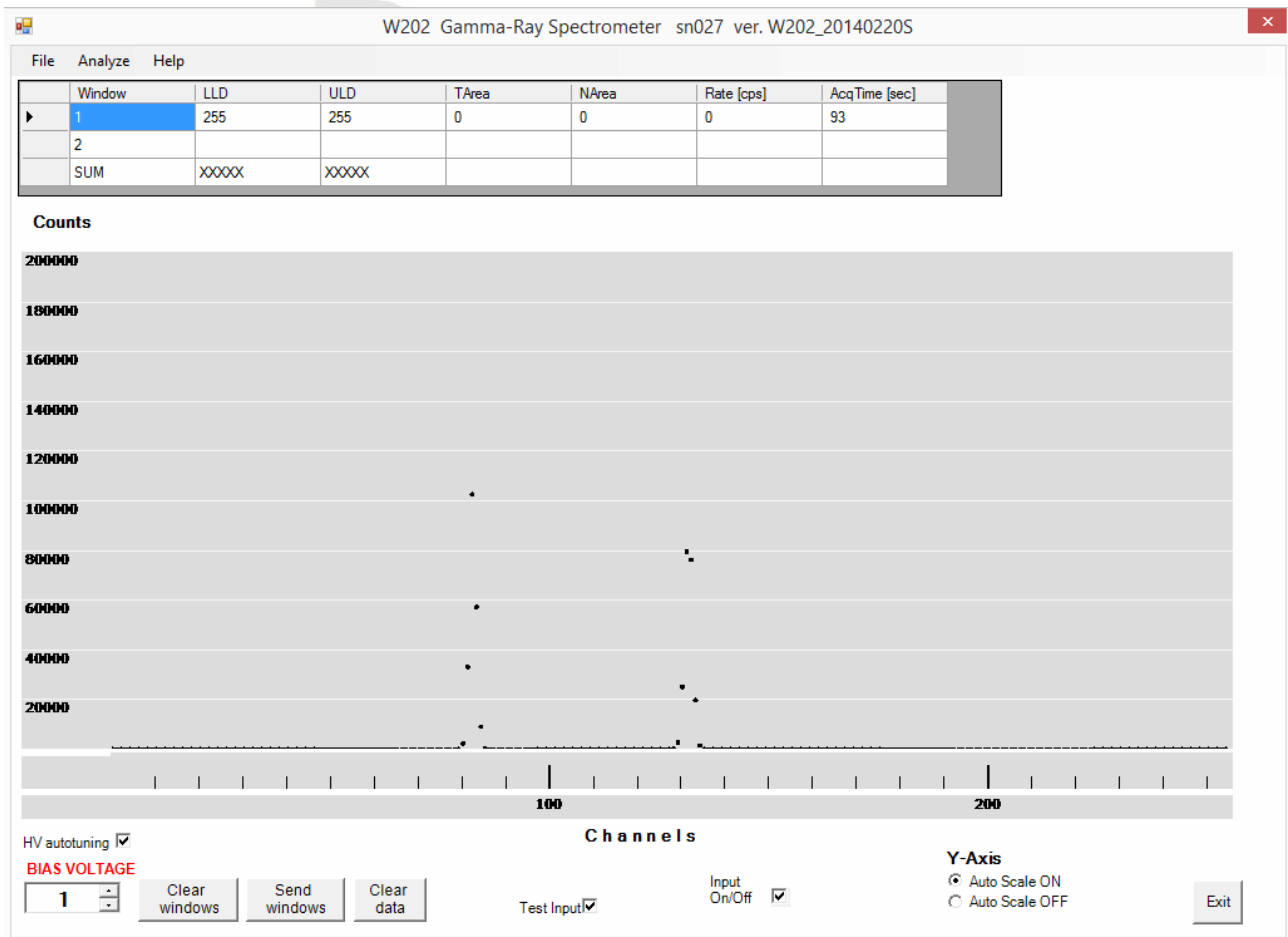
After the Select Device form closes, the Main Form of the W202 PC software will appear as shown in Fig. 13. The central area is used for the plot of the pulse height spectrum. The horizontal axis units of the PHA plot has units of Channels. The range of 256 channels corresponds to the 8-bit ADC used to convert the peak voltage value. Channel 256 corresponds to the 12 V reference voltage.

On the lower left is a display/edit box for the DC voltage applied to the detector. Units are Volts. The voltage is controlled by clicking on the arrows or entering a number via the keyboard. **After the user has changed the voltage value, several minutes are required for the voltage at the detector to stabilize. An accurate pulse height spectrum will not be generated until the voltage is stable.** Use the Clear button to remove any data collected during the time of unstable voltage.



**Figure 3.10** W202 PC Software main form.

The input to the Peak detector is selected by the “Test Input” and “Detector Input” buttons. Clicking the Test Input button causes internally generated pulsed of two fixed heights to be routed to the pre-amp input. See Figure 3.11. This function is used to verify the W202 is working properly independent of the detector. The positions of the test pulses on the PHA plot are independent of the High Voltage setting.



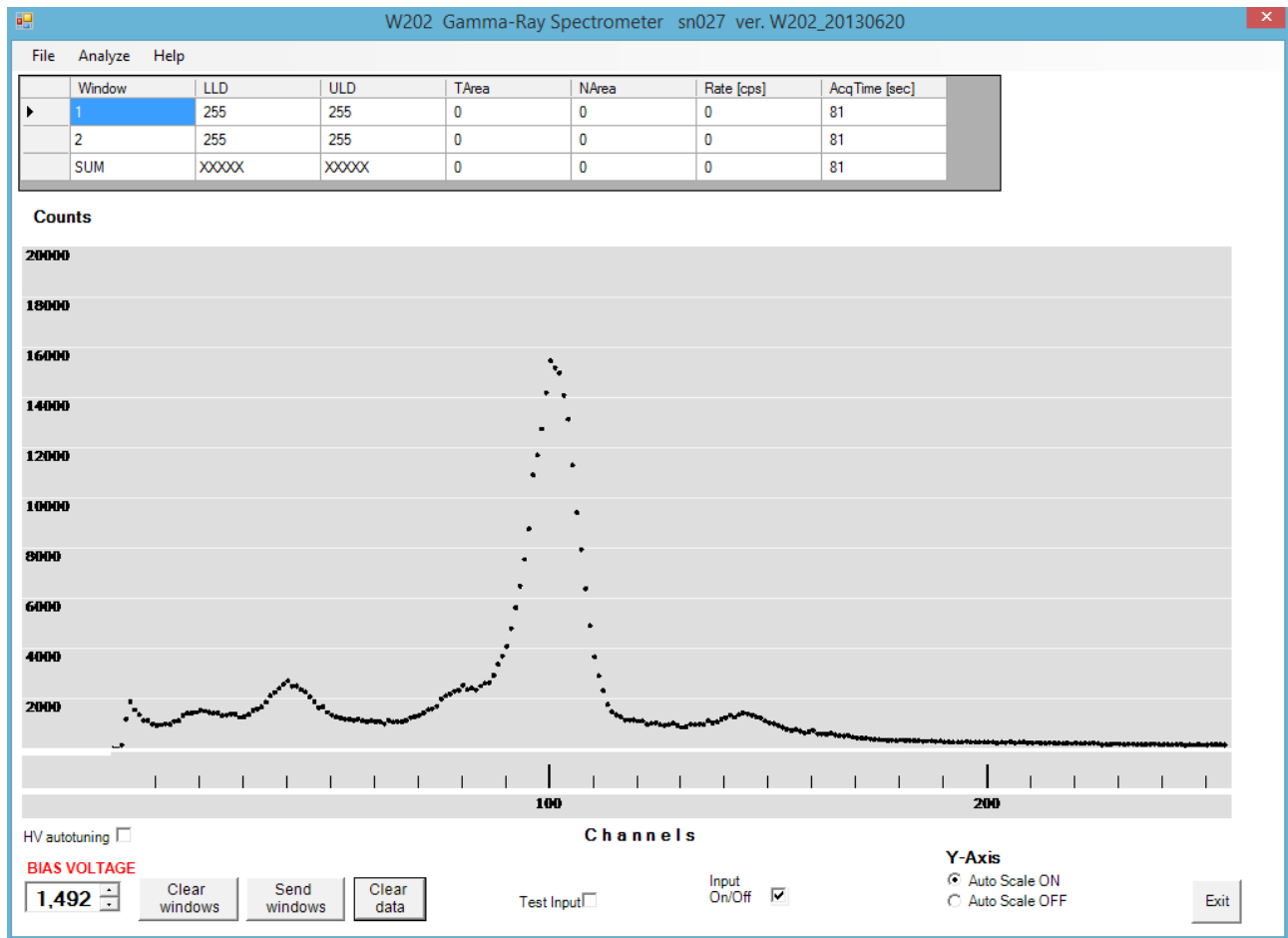
**Figure 3.11** Display of Pulse Height Spectrum of Test Input pulses.

The input from the detector is always active.

### Setting the Bias Voltage

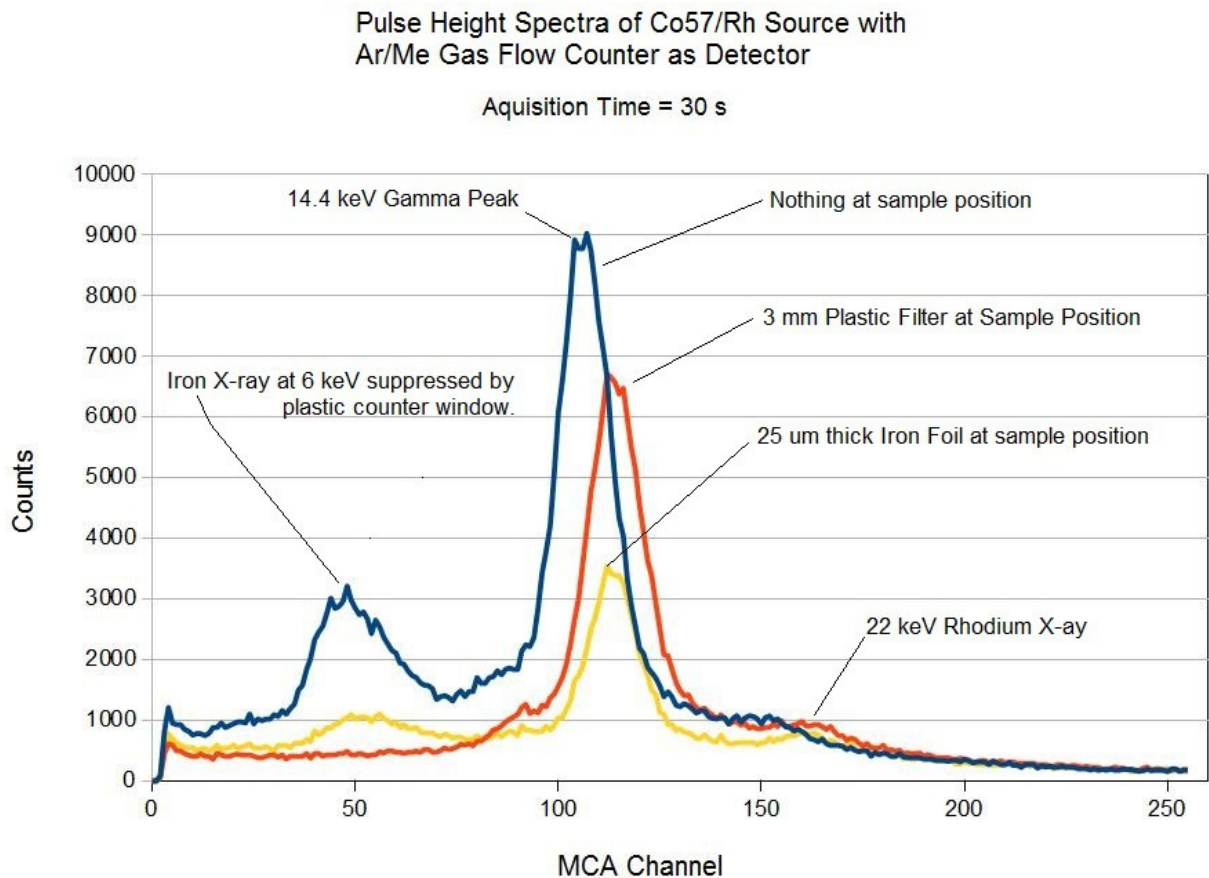
If the W202 is connected to a gas counter (detector), use the arrow buttons in the lower left corner to increase the High Voltage to until the 14 keV gamma peak is at approximately channel 100. The desired voltage value can also be typed into the Bias Voltage test box. After the HV has been changed, it can take as long as 20 s for the voltage at the detector to stabilize. Once the HV is stable and if you have a Co-57 gamma-source shining on the detector window, you will see the pulse height spectrum growing in amplitude as shown in Fig. 3.12. The actual HV required depends of the particular detector and the gain and filter settings in the W202. Normally, for a Ar/Me gas counter with 1 atmosphere gas pressure and a 50 micron diameter center wire, the HV will be in the range of 1,700 V to 2,000 V.





**Figure 3.12** The pulse height spectrum of a 5 mCi Co-57 Mossbauer source with a Rh matrix. The source to detector distance was 65 mm. A Pb shield with a 17 mm diameter aperture was in front of the Kr gas counter.

The the 6 to 7 keV Iron X-rays, the 14.4 keV gamma and the 22 to 24 keV Rhodium X-rays. For Fe-57 Mossbauer spectroscopy the 14 keV Gamma peak is counted. The 6 keV X-rays are unwanted. At higher count rates the 6 keV absorption events can generate pulses in the detector that overlap in time with the pulses due to the gamma and reduces the gamma counting efficiency. Often non-resonant absorption by the sample in a transmission mode experiment will reduce the 6 keV rate so that the X-rays are not a problem. If not, then a high pass filter can be placed in the beam to absorb the 6 keV X-rays before they enter the detector. Fig. 3.13 shows the effect as placing various material in front of the detector window.



**Figure 3.13** Several Pulse Height Spectra showing effects of different non-resonant absorbers being placed in the Gamma-ray beam. The data were saved to ASCII text files via the W202 program File/Save As procedure and then imported into a spreadsheet program to generate the plot.

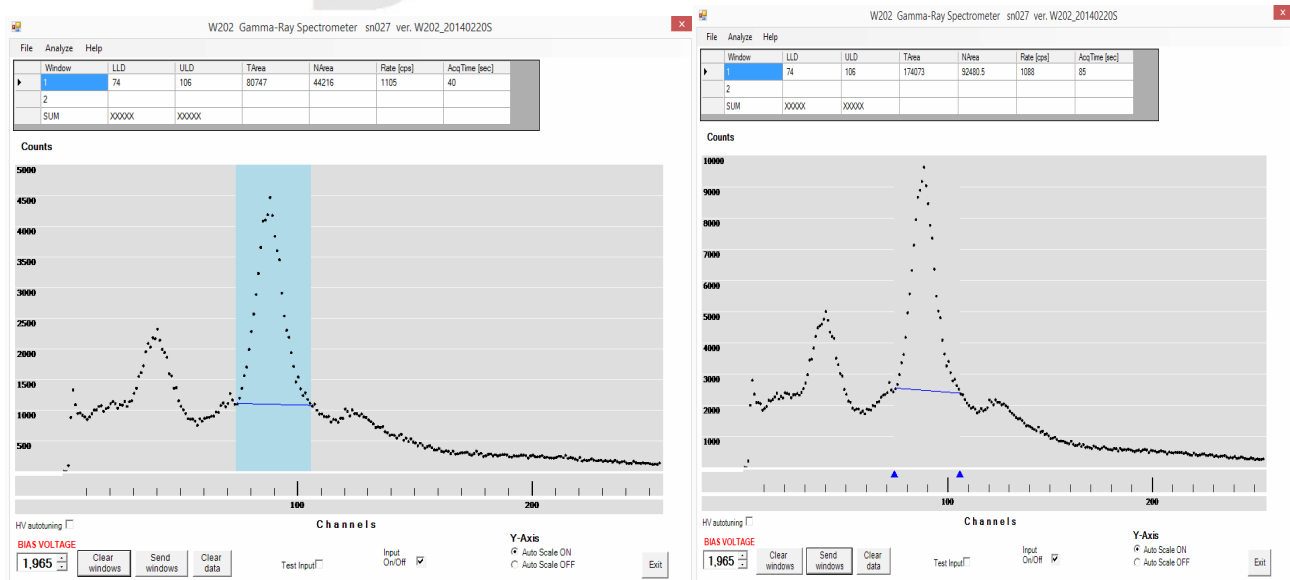
Placing a sample in the beam will reduce the 14 keV peak height relative to the background generated by the 122 keV gammas Compton scattering off electrons in the Kr gas. Figure 8 shows the pulse height spectrum of the Co-57 source after a 27um thick natural Iron foil is placed in the beam in addition to the 3 mm plastic filter .

Once the beam geometry is defined in terms of solid angle and absorbers in the beam, it is time to set the Single Channel Analyzers (SCA) windows. There are two independent SCA's in the W202 hardware. Two SCA's are provided to optimize the system for counting the 14 keV Gammas detected by a Kr gas counter. Approximately one third to one half of the 14 keV gammas absorbed in a Kr gas counter generate a 12 keV Kr X-ray that leaves ("escapes") the counter without interacting with the gas. For those escape events only a net energy of 2 keV is deposited in the detector by the 14 keV Gamma. Thus the peak at 2 keV in the PHA spectrum.

Set the window of SCA #1 by slowly dragging the mouse cursor over the the region of interest. This is done be holding down the left mouse button while moving the mouse. A blue background will appear on the PHA plot indicating the range of SCA #1's window. The range of the window is

defined by the horizontal position of the mouse cursor. The left edge of the window defines the voltage level for the Low Level Detector for SCA #1. The right edge defines the voltage level for the High Level Detector for SCA #1. Once SCA #1's window is defined, clicking and dragging the mouse again will define SCA #2's window, denoted by the pink background color. See Figure 8.

**After both windows have been set, click on Send Windows to transfer the window settings from the PC to the dual SCA's in the W202 hardware.** This action will also clear the windows from the screen plot. See Figure 9. The windows settings are stored in the memory of four digital potentiometers in the W202.



**Figure 3.14** W202 PHS display. (Left) SCA Window defined by mouse click and drag. (Right) After “Send Windows” button has been clicked.

The “Counts Out” signal will now be a stream of TTL pulses generated by detector events that have a peak voltage height that falls within the SCA Window. These counts are routed to the SEE Co W304S Resonant Gamma-ray Spectrometer “Counts-1 In” or “Counts-2 In” when collecting a Mossbauer spectrum.

You can now terminate the W202 PC program by clicking on the Exit Command button or on the “X” at the upper right. The W202 hardware will continue to generate the SCA output pulses until its power is removed or its settings are changed via the W202 PC program. **The W202 PC program does not need to be running to collect a Mossbauer spectrum. It is only used to set the dual SCA windows.**

### 3.4 Velocity Transducer SEE Co Model VT6S

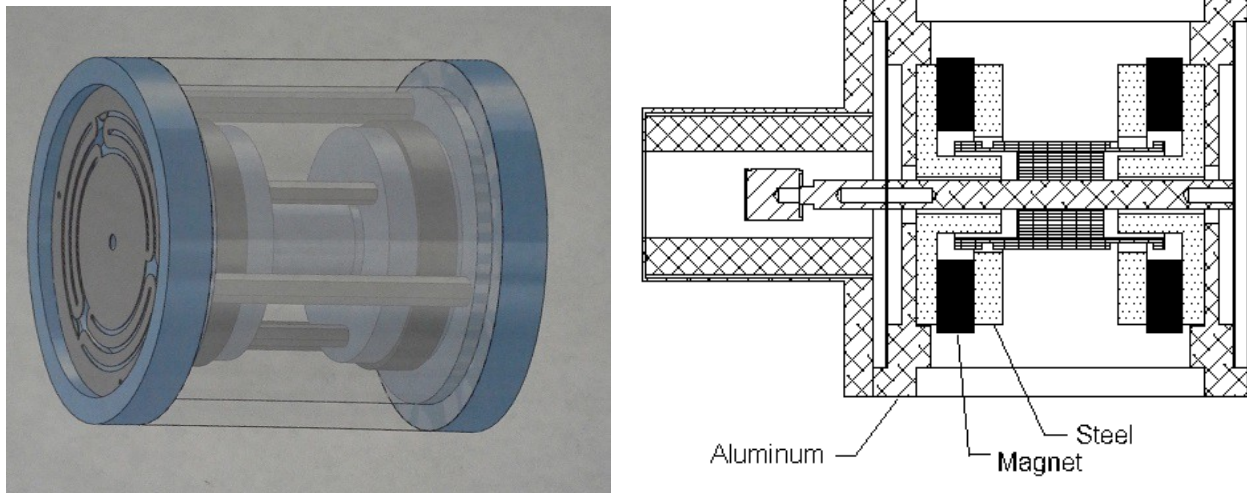
The SEE Co Model VT6S Linear Velocity Transducer is shown in Figures 3.15 and 3.16. The transducer has a double voice coil geometry. The magnet assemblies and the coil form are visible through the transparent case. The table below lists the technical specifications.

SEE Co Model VT6S Specifications

Natural Frequency	11 Hz
$T_{1/2}$ Decay Time	200 ms
Drive Coil Resistance	25 Ohms
Velocity Pick-up Coil Resistance	3,200 Ohms
Shaft Thread	Male M4-0.7
9-pin male sub-D Connector	Pins 2-4 Velocity Pick-up Coil Pins 7-8 Drive Coil
Velocity Pick-up Coil Sensitivity	28 mV / mm/s



**Figure 3.15** VT6S Velocity Transducer showing (left) source shield off and gamma source mounted on motor shaft and (right) source shield mounted on transducer. Visible through the transparent case are the magnets for the drive coil and the velocity pick-up coil.



**Figure 3.16** (Left) Drawing showing one of two flexure plates that support the motor shaft. (Right) Cross-section showing double “Voice-Coil” magnet assemblies. The Garolite coil form is shown with a narrow groove on the left for the velocity pick-up coil and a wide groove on the right for the drive coil. The steel pole pieces concentrate the magnetic fields at the coils.



### 3.5 W304S Resonant Gamma-ray Spectrometer

This section describes the configuration and operation of the SEE Co. Model W304S Resonant Gamma-ray Spectrometer. The W304S incorporates a dual-input Multi Channel Scaler (MCS), a waveform generator, an analog servo amplifier, a USB interface and user-interface PC software. The hardware components are housed in a single, compact box and may be used for many applications. However, the W304S was designed primarily to be the core of the SEE Co. MS6S Student Lab Resonant Gamma-ray Spectroscopy (RGS) System.



**Figure 3.17** W304S Resonant Gamma-ray Spectrometer Front View. From left to right are:

- A. Power Indicator LED
- B. Mini B USB connector
- C. “Ch1” Counts Input SMA jack for TTL input pulses for MCS 1
- D. “Ch2” Counts Input SMA jack for TTL input pulses for MCS 2
- E. “CAP” – Channel Advance Pulse output, 3.3V, 7 microseconds TTL, SMA connector
- F. “SSP” – Start Sweep Pulse output, 3.3V, 7 microseconds TTL, SMA connector
- G. “Mon” – Analog Monitor SMA connector.
- H. Selector switch to route one of the four analog signals to the “Mon” output connector. The signals are:
  - “REF” Velocity Reference Waveform
  - “VEL” Amplified Velocity Pick-up Voltage
  - “ERR” Velocity Error signal = “VEL” - “REF”
  - “DRIVE” Voltage applied to Drive Coil of Velocity Transducer
- I. 9-pin sub-D female connector for the Velocity Transducer.
  - Pin outs:
    - 2 Pick-up coil V+
    - 4 Pick-up coil V-
    - 7 Drive coil V+
    - 8 Drive coil V-



**Figure 3.18** W304S Rear View showing the 100 – 240 VAC, 50 – 60 Hz line power connector with ON/OFF rocker switch.

A

F

T

### W304S Operation as part of a SEE Co. MS4 Mössbauer Spectrometer

When used as part of the MS4 system, the W304S is very simple to install. With the power off, connect all cables as shown in Figure 3.2. Before turning on the W304S, the user interface program must be installed on the PC along with the driver for the USB interface. If the PC was supplied with the MS4 then the W304S program and the USB drivers will have been installed and tested at SEE Co. If you do need to install the USB drivers and W304S program on you PC, follow the instructions in the README.TXT file bundled with the installation package.



Figure 3.19. Initial “Splash” Window of W304S Program

When the W304S program is executed, the first window to appear is shown in Figure 5. This splash screen displays a drop-down list box with the serial numbers of all the W304S boxes connected to the PC. Usually, there is only one. The displayed serial number was sent from the W304S to the PC and demonstrates that the W304S is powered on and communicating properly. Choose the W304S you wish to use and click with the left mouse button on “OK” to continue. The splash screen will close and the main window of the W304S program will open as shown in Figure 6.

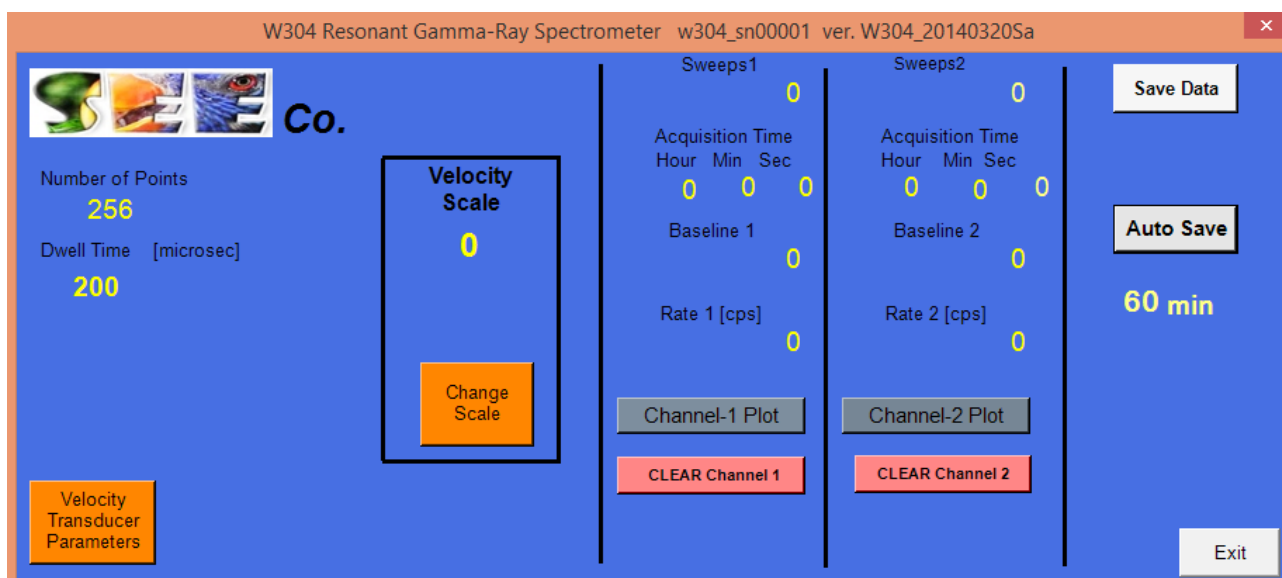


Figure 3.20. Main window of W304S PC program.



The main window is divided into four parts. On the left are displayed the number of points in the time scan of the MCS and two Command Buttons. In the middle are information and Command Buttons regarding the two spectra stored in the dual MCS. On the right are the Save Data to disk button, Auto Save button and the Exit button. Table 1 lists the displayed parameters and Command Buttons.

Name	Symbol	Description
Number of Points	Nt	Total number of channels in the MCS time scan
Velocity Scale	Vs	Label that indicates scaling of Vref by factor of (Vs/12). If Vpu Amplifier Gain is set properly then Vs will equal the Amplitude of Velocity Scan, e.g. Vs = 12 would indicate a velocity scan from -12 mm/s to +12 mm/s.
Change Scale Command Button		Makes the change velocity scale buttons visible.
Velocity Transducer Parameters Command Button		Makes the servo amp parameters and dwell time parameter visible for editing. The servo parameters are shown Table 2 and discussed in Section IV.
Sweeps1, Sweeps2		The number of MCS sweeps since the data was last cleared.
Acquisition Time		The time since the data was last cleared.
Baseline		The value of the first channel in the MCS data.
Rate		The MCS Input count rate [counts / second ]
Channel 1 Plot, Channel 2 Plot Command Buttons		Makes the MCS data plot (the Mössbauer spectrum) visible.
Clear Channel 1, Clear Channel 2 Command Buttons		Set the MCS data to zero.
Save Data Command Button		Immediately writes the MCS data to the disks files c:\W304S_data\W304S_1.txt and c:\W304S_data\W304S_2.txt
Auto Save Command Button		Display Auto Save Options box. See below
Exit Command Button		Closes the W304S program on the PC. Note, the W304S box will continue to collect data until it is turned off. If the W304S PC program is run again, the current data in the W304S box will be sent to the PC.

Table 1. Parameters and Command Buttons on the Main Form.

When the main window first opens, a few seconds will pass as the W304S box is interrogated regarding its status. The W304S RAM memory is non-volatile MRAM. When the W304S is first turned on, items such as the number of channels and the Velocity Reference Waveform are set to zero. If that is the case then the W304S PC program will download the required information to the W304S box. The default values are stored as ASCII files in the C:\W304S\W304\_SNxxxxx folder on the PC disk drive, where xxxxx is the W304S box serial number. These files are listed in Table 2.

ASCII File Name	Content
Nt_dt_POTs.txt	<p>Nt = total number of channels in MCS time scan ( fixed at 256 for the current version of the W304S embedded software.)</p> <p>dt = MCS dwell time [ micro seconds ]</p> <p>The current values of the eight 10-bit 100 kohm digital potentiometers on the W304S circuit board. The pot values shown on the Servo Parameter Windows are in units of kohms while the pot values stored in the N_dt_POTs.txt file are integers with range 0 to 1023. See below for discussion of the Servo Amp.</p>
Vref.txt	<p>Vref(i) where <math>i=1, Nt</math>. The Velocity Reference Waveform is stored as a single column of integers with range -32,000 to +32,000. A velocity of 0 mm/s corresponds to Vref value of 0. This waveform is loaded into the RAM in the W304S box when the “Send Vref” command button is activated. The default waveform is triangular and is the one displayed in this manual. The user may define any waveform and copy it to Vref.txt.</p> <p><b>Note, the average velocity over one cycle must be zero which means the average value of Vref must be 0.</b> Otherwise, the average position of the motor shaft will shifted and may move until the shaft hits its mechanical stop.</p>

Table 2. ASCII Files used to define the default values of the Velocity Waveform and Servo Amp settings.

Figure 3.21 shows the PC screen after the Channel 1 Plot Button has been activated. The MCS data for Channel 1 are scaled automatically to span the Plot Window.

The Velocity Transducer Monitor output is selected by a rotary switch on the W304S front panel. The analog signals are the Velocity Reference Waveform, the Velocity Pick-up Coil signal or the Velocity Error signal and the Drive Coil Voltage. The Vref voltage is generated by a 16-bit DAC. Hence, the Vref waveform is stored as integers within the range -32,768 to +32767 which corresponds to a Voltage range of -12 V to +12 V.

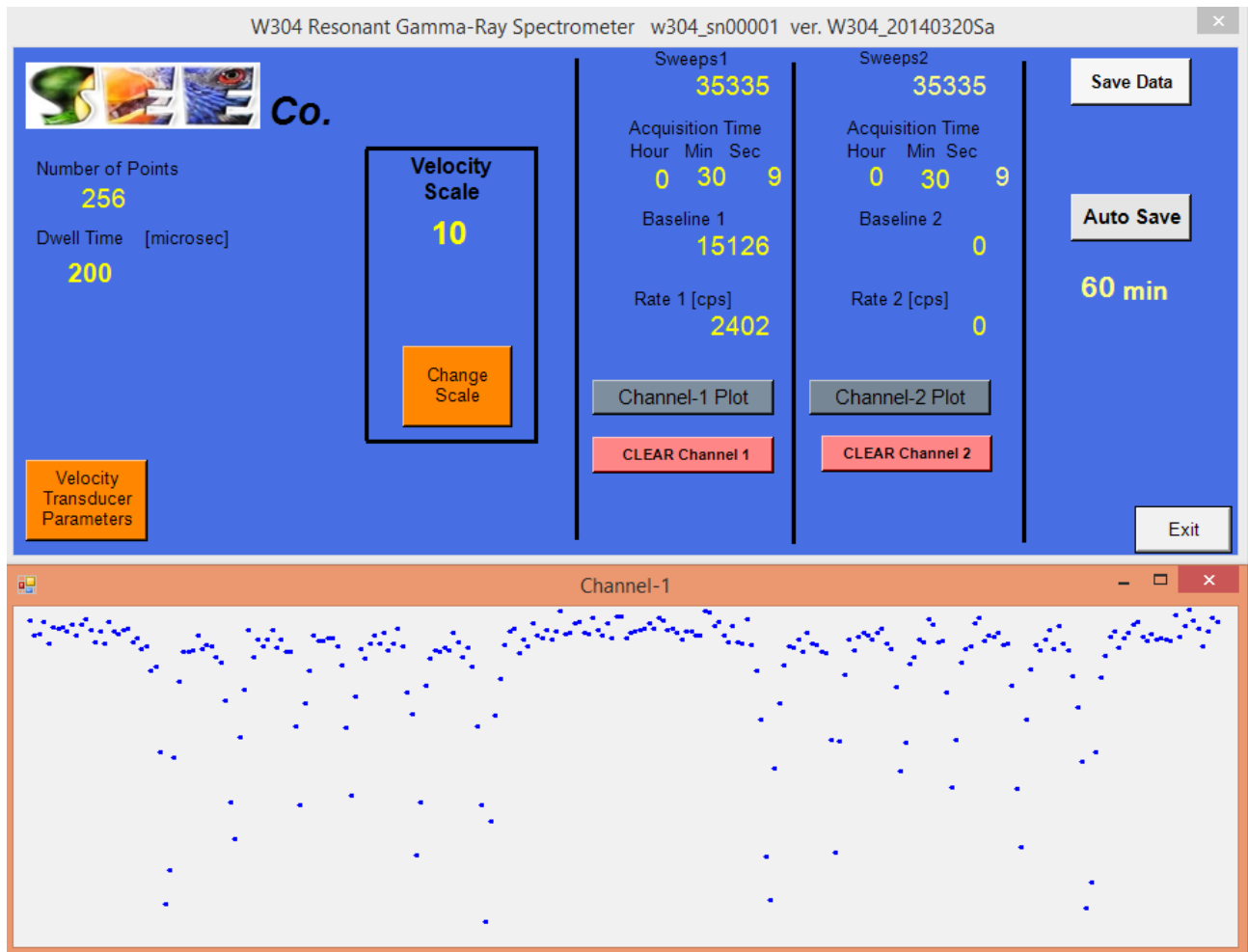


Figure 3.21. Screen with Main Window, Channel 1 Data Plot Window and Monitor Window.

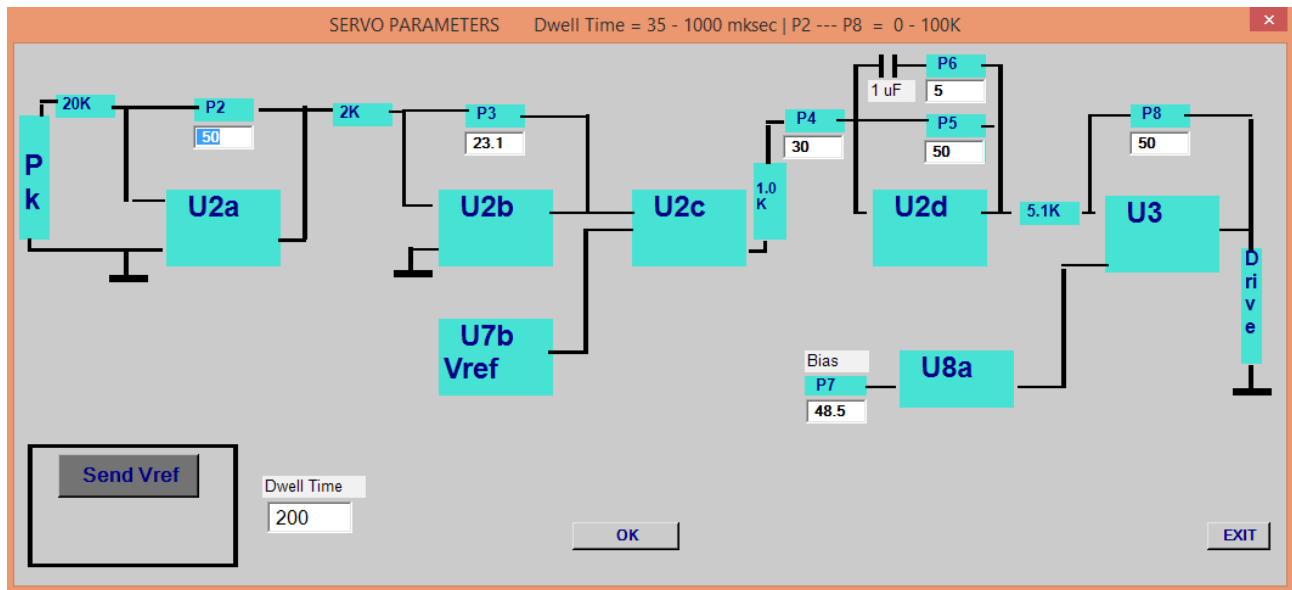


Figure 3.22. Servo Amp Parameters Displayed

Clicking the Velocity Transducer Parameters button will cause the servo amp parameters to be displayed as shown in Figure 3.22. Also displayed is the MCS dwell time “dt” in units of microseconds. The value in the white boxes may be edited. Clicking “EXIT” will cause the Servo Parameters display to close. The servo amp parameters values and the dt value will be updated in the W304S RAM via the USB connection, the MCS data cleared and the data collection started. The values stored in the disk file Nt\_Ndata\_dt\_K.txt are also updated.

When the W304S is used with a SEE Co VT6S Velocity Transducer, the servo parameters stored in the Arr\_Sc.txt file will have been optimized at SEE Co. No changes should be required. The servo parameters are discussed in detail in Section 4.

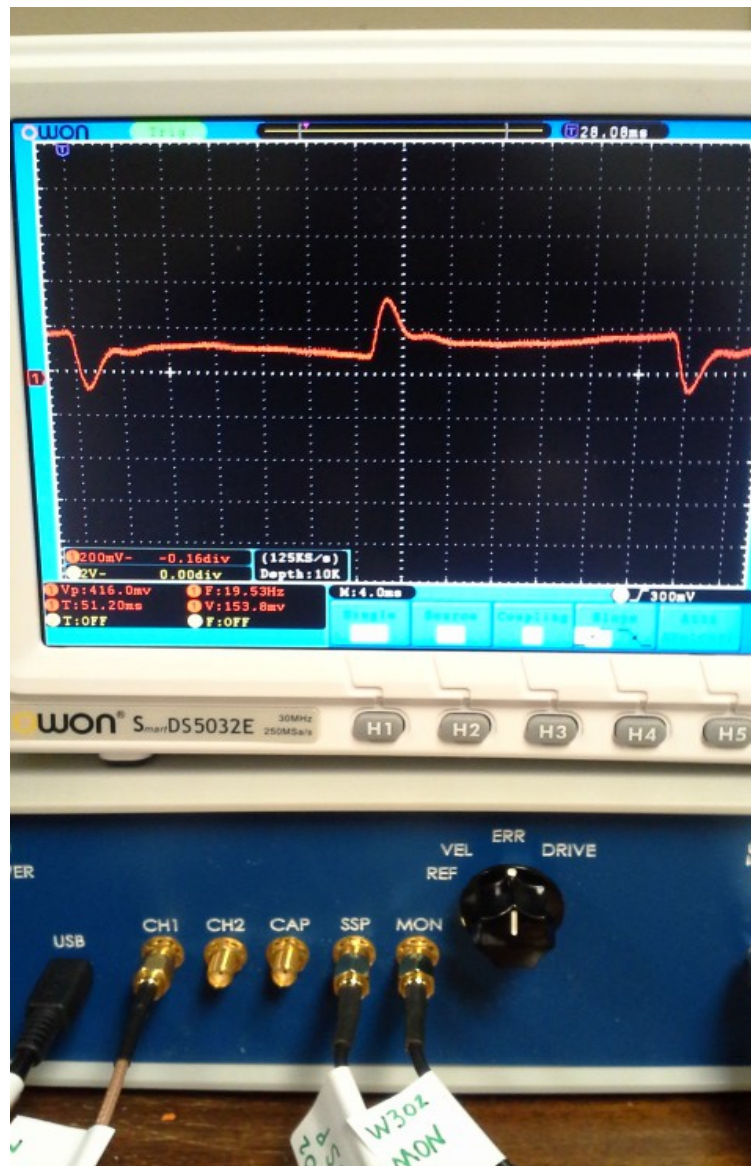


Figure 3.23. Velocity Transducer Error Signal

Figure 3.23 shows the Velocity Error signal “Verr”. In this example, the peak to peak amplitude is approximately 80 mV if the ringing that occurs at the corners of  $V_{ref}$  is ignored.

The range of the velocity scan may be changed by clicking on the “Change Scale” button. Left and Right arrows and an “OK” button will appear. Click on the arrows to choose the desired range and then click “OK”. After 2 or 3 seconds, the data in both MCS 1 and MCS 2 will be cleared and the data acquisition started.

## 4. Discussion of the Servo Loop

### 4.1 Servo Amplifier Circuit

As mentioned above, when the W304S is used with the SEE Co. VT6S velocity transducer the parameters in the Nt\_dt\_K\_POTs.txt file will have factory set defaults that cause the servo amp to drive the VT6S over the nominal velocity range of +/- Vs. Situations arise that require custom settings of the servo parameters. Examples are the need for a larger than +/- 12 mm/s scan range or the use of a different velocity transducer with the W304S. Here the servo loop design and adjustment of the parameters are discussed in detail.

A linear velocity transducer consists of a drive device and a velocity sensor, both mounted on the same motor shaft. The drive device is usually a “drive coil” that is in a constant magnetic field. When the drive amplifier causes current to flow in the drive coil, a force proportional to that current will act on the motor shaft. At low frequencies,  $\sim < 1,000$  Hz, the motor shaft is rigid and the Velocity Pick-up Coil (VPC) moves in phase with the Drive Coil. The VPC is also in a magnetic field created by a second magnet separate from the drive coil magnet. The Voltage across the VPC will be proportional to the product of the velocity of the coil perpendicular to the field and the total magnetic flux through the coil. A perfect velocity sensor will have the value of the total flux independent of the position of the VPC. Such a sensor is linear. A real velocity sensor will have flux that depends slightly on the VPC position. This flux variation will contribute to the non-linearity of the velocity sensor. To avoid distortion of Mössbauer line shapes the overall non-linearity of the velocity scan should be less than 0.1%.

Depending on the shaft material and shape plus the amount of attached mass, at some higher frequency, usually more than 10 kHz, the shaft is not rigid and can oscillate in a “breathing” mode. At that frequency, the motion of the VPC is out of phase with the Drive Coil. This situation could cause oscillation in the servo loop at that frequency and should be avoided. See Section 4.3.

The W304S servo amp is the analog circuit depicted in Figure 3.23.

The drive coil Voltage,  $V_{DR}$ , is given by

$$V_{DR} = G_3 ( G_{2d} V_{ERR} + V_{bias} )$$

where

$$V_{ERR} = \text{error Voltage} = G_{PU} V_{PU} - V_{REF}$$

$$G_{2d} = - P5 / P4 \quad (\text{gain of op amp } U_{2d} \text{ at low frequencies})$$

$$G_3 = - P8 / 5.1 \text{ kohm} \quad (\text{gain of op amp } U_3)$$

$$V_{PU} = \text{Velocity Pick-up Coil Voltage (shown as “Pk” in Figure 3.22)}$$

$$G_{PU} = G_{2a} G_{2b} \quad (\text{Pick-up Gain})$$

$$G_{2a} = - P2 / 20 \text{ kohm} \quad (\text{gain of op amp } U_{2a})$$

$$G_{2b} = - P3 / 2 \text{ kohm} \quad (\text{gain of op amp } U_{2b})$$

The op amps of U2 have open-loop bandwidth gain product on the order of 1 MHz. Since the Drive Coil and the VPC are mounted on the same shaft, the configuration shown in Figure 11 is a negative feedback loop or “servo” loop.

There are 7 digital 100 k Ohm potentiometers in the W304S servo amplifier. Digital pots function the same as mechanical pots except the “wiper” connection is controlled by integer valued number loaded into an 10 bit register via a serial interface. (There is an 8<sup>th</sup> pot on the W304S circuit board. It is denoted p1 and is not used at this time.) The digital pots are labeled P2, P3...P8 in Figure 3.22. Their values may be entered in units of kohms with a range of 0.1 kohm to 100.0 kohm. The W304S program converts the resistance value to 10 bit integers and sends those values to the embedded MCU in the W304S box.

#### 4.2 Setting the Range of the Velocity Scan

A given velocity transducer will have a fixed velocity sensor (pick-up coil) sensitivity, S, given in mV per mm/s of the motor shaft velocity. Typically, S will have a value on the order of 20 mV/mm/s. In the W304S RGS unit, the velocity reference waveform is stored in MRAM as an array of integers with a range of -32,768 to +32767. This range corresponds the +/-12V output range of the 16-bit Digital-to-analog converter (DAC) that converts the integers to an analog velocity signal.

The velocity reference waveform, Vref, normally has a peak-to-peak amplitude that provides some margin relative to the full range of the DAC, +/-12V. If that margin is 20%, then the range of Vref will be from -26,000 to +26,000 and correspond to an analog range of  $0.8 * 24V = 19.2 V$ . The velocity scan range, R<sub>v</sub>, will be equal to this 19.2 V divided by G<sub>PU</sub> S.

$$R_v = 19.2 V / (G_v S)$$

For example, if the desired velocity scan range is +/- 10 mm/s and S = 20 mV/mm/s then

$$\begin{aligned} R_v = 20 \text{ mm/s} &= ( 19.2 V / 0.02 V/\text{mm/s} ) / G_v \\ &= 960 \text{ mm/s} / G_v \end{aligned}$$

and

$$G_v = 48 = (P_2 / 20 \text{ kohm} ) (P_3 / 2 \text{ kohm} )$$

If a range of +/- 50 mm/s ( R<sub>v</sub> = 100 mm/s ) was required, P<sub>2</sub> and P<sub>3</sub> would be changed so that G<sub>v</sub> = 9.6 .

For the SEE Co MS6S Spectrometer, the values of P<sub>2</sub> and P<sub>3</sub> are usually set so that the Velocity Scale Index indicates R<sub>v</sub> / 2 in [mm/s].

### 4.3 Servo Loop Gain and Bandwidth

As given above, the magnitude of  $V_{err}$  is

$$|V_{ERR}| = |V_{DR} / (G_{2d} G_3)| = |V_{dr} / G_D|$$

If all the components in the servo loop were perfectly ideal with no time delay between input and output then  $|V_{ERR}|$  could be forced to an arbitrarily small value by increasing  $G_D$ . However, all electronic and mechanical components have some finite delay in their response. For a given frequency sinusoidal excitation that time delay causes a phase shift of the output relative to the input. A servo loop's performance depends critically on the frequency dependence of the gain and phase shift around the loop. If for a particular frequency the total unwanted phase shift is 180 degrees and the loop gain is greater than or equal to one then the servo mechanism will oscillate at that frequency without regard for control signal.

Positive feedback – PA system howl: First heard in 1915 by Jensen and Pridham (ref. [http://en.wikipedia.org/wiki/Public\\_address](http://en.wikipedia.org/wiki/Public_address) )



## 5. Using the Iron Metal Spectrum to Calibrate the Velocity Scale

The spectrum of an iron metal foil **at room temperature** is the working standard used to calibrate the velocity scale; the energy (velocity) corresponding to each line is known. We wish to establish the connection between channel number and velocity, and so will want to know the MCS channel location of each line.

Figure 5.1 shows the room temperature spectrum of a 6 micron iron metal foil, plotted against channel number. Instructions for doing a least squares fit of 6 Lorentzians lines to this spectrum are given in the first example of Chapter 3 of the WMOSS Software Manual. The results are line positions 46.30, 80.10, 114.24, 139.37, 173.56, 207.49. We now consider the calibration problem.

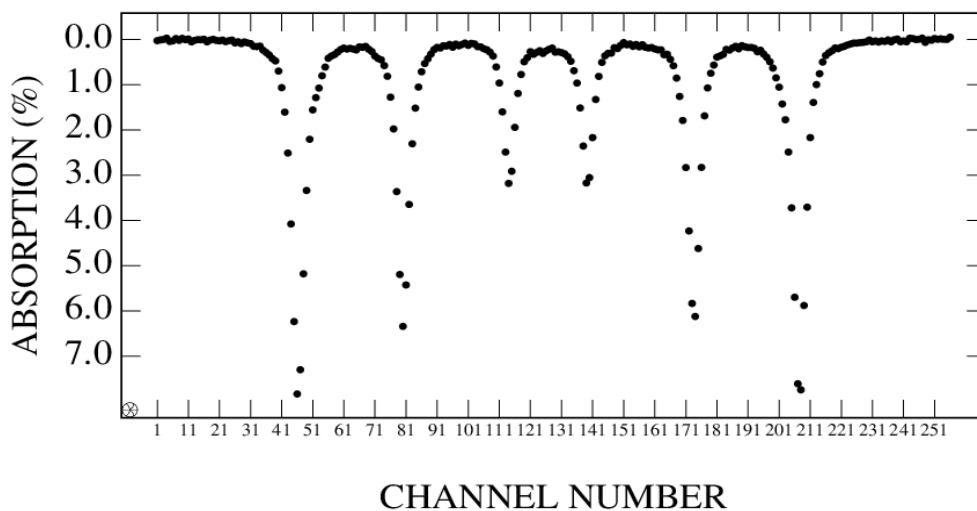
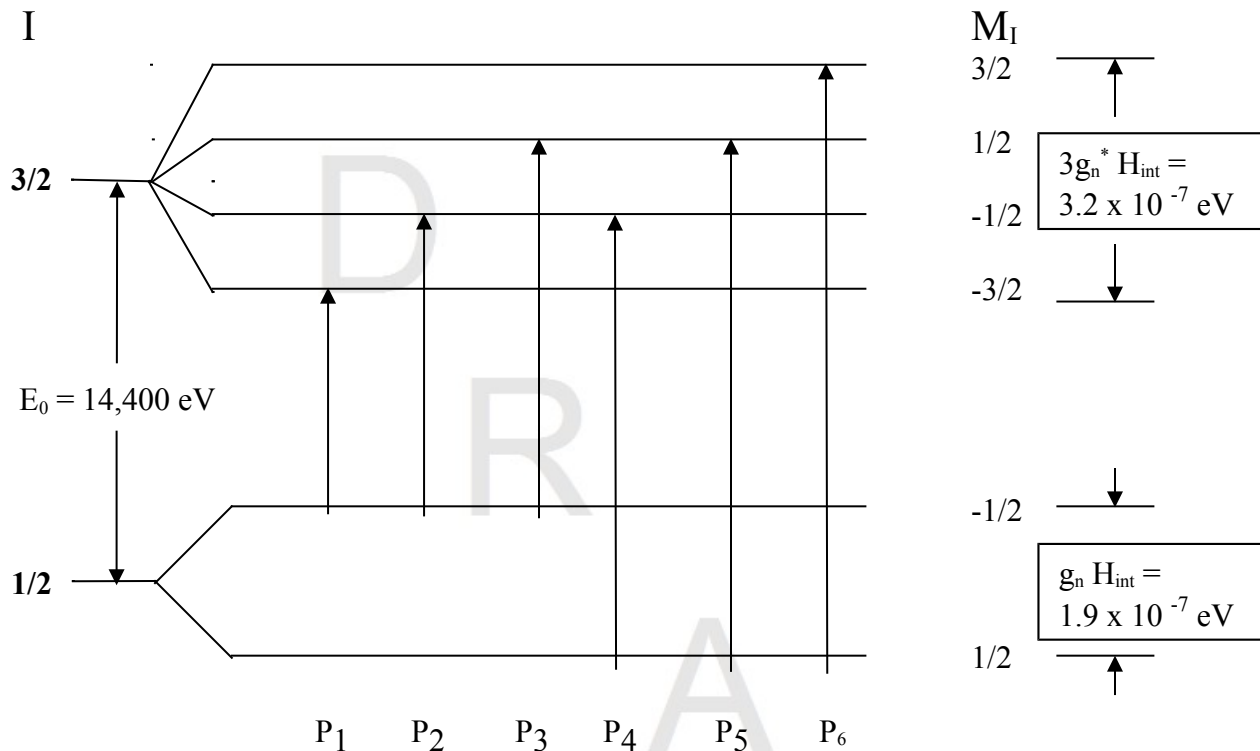
As discussed by Preston, *et al.* (1962)\*, there is no significant electric field gradient at the Fe nucleus in iron metal and the hyperfine splittings of the  $I = 1/2$  doublet and the  $I = 3/2$  quartet are simply proportional to the effective internal magnetic field  $H^{\text{int}}$ . They reported

$$H^{\text{int}}(T=293\text{K}) = 330 \text{ kG}$$

$$\Delta H^{\text{int}} / \Delta T \Big|_{T=293\text{K}} = -0.06 \text{ kG / K}$$

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\* Preston, R. S. , Hanna, S. S. and Heberle, J. (1962) "Mössbauer Effect in Metallic Iron\*", Phys. Rev. 128, 2207-2218.



$$P_6 - P_1 = (3g_n^* + g_n) \beta_n H_{\text{int}} = 10.66 \text{ mm/s}$$

$$P_5 - P_2 = (g_n^* + g_n) \beta_n H_{\text{int}} = 6.17 \text{ mm/s}$$

$$P_4 - P_3 = (-g_n^* + g_n) \beta_n H_{\text{int}} = 1.68 \text{ mm/s}$$

$$1 \text{ mm/s} = h\nu(1 \text{ mm/s})/c = 14.4 \text{ keV} / 3 \times 10^{11} = 4.8 \times 10^{-8} \text{ eV}$$

Figure 5.1 Iron Metal  $^{57}\text{Fe}$  hyperfine splitting and Mössbauer spectrum

Over a range of 5 K,  $H^{\text{int}}$  changes by 0.1 %, an insignificant amount for routine measurements. However, Preston, *et al.* also detected a 0.5% variation in  $H^{\text{int}}$  among different iron foils.

Therefore, if iron metal and the value of  $H^{\text{int}}$  quoted above are used as the calibration standard, the experimental precision can be no better than 0.5% unless the foil was prepared in the manner given by Preston, *et al.*

We assume that the spectrometer is operated in the constant acceleration mode and that any non-linearities in the electromechanical velocity transducer response are small. The source velocity as a function of time is

$$V(t) = a t + V_0$$

where  $a$  and  $V_0$  are constants. The source velocity at the midpoint of the dwell time for the  $i^{\text{th}}$  channel of the scan is then

$$V(i) = \Delta_V (i - C_0)$$

where

$i$  is the number of the active memory channel,

$V(i)$  is the source velocity at the midpoint of the dwell time for the  $i^{\text{th}}$  channel ,

$\Delta_V$  is the acceleration of the source given in mm/s/channel

$C_0$  is the channel number at which the velocity is defined to be zero.

Given the peak positions  $C_i$  from the fit and the published values of the  $P_i$ , we can calculate  $\Delta_V$  and  $C_0$ . By convention, the zero of the velocity scale is the centroid of the room temperature iron metal spectrum. This choice permits comparison of Mössbauer spectra without reference to the isomer shift of the source. The centroid of the spectrum is

$$C_0 = \sum_{i=1,6} a_i C_i / \sum_{i=1,6} a_i$$

where the  $a_i$  are the area fractions. The step,  $\Delta_V$ , is

$$\Delta_V = \sum_{i=1,3} \{ (a_i + a_{7-i}) (P_{7-i} - P_i) / (C_{7-i} - C_i) \} / \sum_{i=1,6} a_i$$

For spectrum FEMETAL6.B, the calibration constants are:

$$C_0 = 126.80$$

$$\Delta_V = 0.0662 \text{ mm/s/chan}$$

**According to Preston, *et al.*, the uncertainty in  $\Delta_V$  is 0.5%.**

Once  $\Delta_v$  and  $C_0$  are known for a particular velocity scan range setting on the spectrometer, they can be entered into the WMOSS Data Header of spectra recorded on that scale. Many spectrometers suffer from long term (~months) drift. Therefore, an iron metal calibration spectrum should be recorded periodically.

D  
R  
A  
F  
T

## 6. References

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