

# The Mössbauer Effect

## 1 Background

Any bit of matter (say, an atom or molecule) emitting any type of radiation must recoil in order to conserve energy and momentum. Both the recoil energy and the energy of the emitted photon must come from the energy difference between the initial and final state energy levels of the emitting source ( $h\nu = E_i - E_f$ ); thus the energy of the emitted photon ( $h\nu'$ ) must be *smaller* than  $h\nu$ . Likewise, an atom or molecule absorbing radiation (absorber) must recoil in order to conserve energy and momentum. The energy of the photon to be absorbed ( $h\nu''$ ) must be *larger* than  $h\nu$  for absorption to happen. Notice that emission and absorption work in opposite directions.

An approximate calculation gives the difference between the energy needed for absorption and the energy needed for emission involving a particular transition:

$$\Delta(h\nu) \approx \frac{(h\nu)^2}{mc^2}, \quad (1)$$

where  $m$  is the mass of the source (and absorber) and  $c$  is the speed of light [1, p. 265]. If we compare a transition involving an optical photon (say, around 2 eV) and a nuclear transition involving a soft gamma-ray photon (say, around 10 keV) in a free iron (Fe) atom ( $mc^2 \approx 5 \times 10^{10}$  eV) we get energy differences due to recoil of approximately  $8 \times 10^{-11}$  eV for the optical transition, and  $2 \times 10^{-3}$  eV for the nuclear one.

Is it possible for our iron atom to absorb a photon emitted by another iron atom? To answer this we must consider another aspect of radiative transitions: they are resonant phenomena, and like any resonant phenomenon, there are a range of frequencies around the central frequency which can participate in the energy transfer. That is, emission and absorption spectra exhibit characteristic line widths. Thus, the resonant emission-absorption process can occur if the difference  $\Delta(h\nu)$  in the above equation is smaller than the relevant linewidth.

Many physical process can act to broaden the linewidth of a radiative transition. Examples you may encounter in experiments in the advanced labs are pressure broadening (important in microwave spectra) and Doppler broadening (evident in visible-light spectra). But even if all of these effects are minimized, there is still one irreducible cause of a linewidth: the Heisenberg Uncertainty Principle. This is the source of the so called “natural” linewidth. For atomic, molecular, and nuclear radiation, the minimum possible spread in frequencies emitted from a particular transition, or natural linewidth ( $\Delta E$ ), is obtained from the uncertainty relation

$$\Delta E \Delta t \approx \hbar, \quad (2)$$

where  $\Delta t$  is the lifetime ( $\tau$ ) of the excited state. For an excited state with  $\tau \approx 10^{-7}$  s, the natural linewidth is  $\approx 4 \times 10^{-8}$  eV. So, in our example, the natural linewidth for a transition from a state with  $\tau \approx 10^{-7}$  s would allow for an overlap of energies between the radiation emitted by the source and radiation needed for absorption in the case of the optical transition, while the energy difference in the case of the gamma-ray is several orders of magnitude larger than the natural linewidth and *no resonant emission and absorption should be observed*.

So, the physics community was quite surprised when, in 1958, Rudolf Mössbauer discovered that for a substantial fraction of  $^{191}\text{Ir}$  atoms in a solid source, nuclear resonant emission and absorption was possible. Mössbauer was able to explain this unexpected effect by invoking the quantum mechanical

nature of a solid. It is possible to show that the energy states of a crystal lattice (normal modes) are quantized, thus only a discrete number of energy states is available for the solid to be in. A radiating atom may not be able to transfer its recoil energy to an excited state of the lattice. When this occurs, the *entire crystal* recoils as a whole (translates), and the energy change between  $h\nu'$  and  $h\nu$  is given instead by  $(h\nu)^2/Mc^2$ , where  $M$ , the mass of the crystal, has replaced  $m$ , the mass of an atom. Since  $M/m$  is usually larger than  $10^{24}$ , the emitted radiation has  $h\nu' = h\nu$ . The same process applies to a solid absorber. Thus the Mössbauer effect is often referred to as the “recoilless emission and absorption of nuclear radiation”. For his discovery and subsequent explanation of the effect, Mössbauer was awarded the Nobel Prize in Physics in 1961. Detailed explanations of the Mössbauer effect and its uses in a wide variety of experimental investigations are treated in a number of books [2, 3, 4, 5].

## 2 Mössbauer Spectroscopy

The recoilless emission and absorption allows for the study of very subtle effects in sources and absorbers which produce nuclear energy shifts of the order of the natural linewidth. For a nuclear transition at 14.4 keV, and  $\tau \approx 10^{-7}$ s, this amounts to effects of  $\Delta(h\nu)/h\nu \approx 3 \times 10^{-12}$ . In a typical Mössbauer experiment a source and absorber with the same isotope of an atom are used,  $^{57}\text{Fe}$  in our case. The emitting  $^{57}\text{Fe}$  results from electron capture in a  $^{57}\text{Co}$  radioactive source, as shown in Fig. 1. Our source is embedded in (diffused into) a Rh matrix. For the  $^{57}\text{Fe}$  nuclei in this experiment, the nuclear radiation has  $E = 14.4$  keV, with wavelength  $0.84\text{\AA}$  (in the x-ray region of the electromagnetic spectrum).

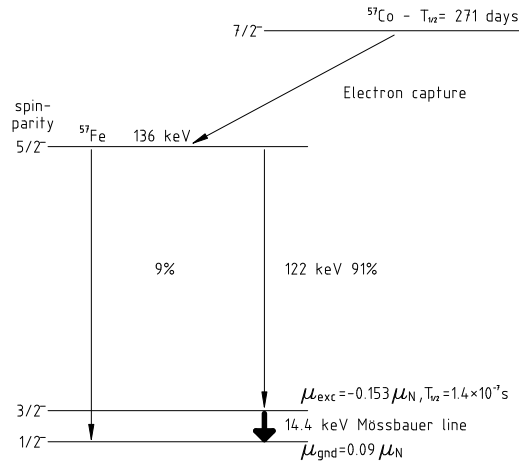


Figure 1: Decay scheme of  $^{57}\text{Co}$ . Note that Co becomes Fe after the electron capture reaction.

The nuclei of  $^{57}\text{Fe}$  forming different compounds or in pure iron experience different electric and magnetic fields depending on the host lattice. These electric and magnetic fields interact with the nuclei, producing different level splittings and shifts. Some effects which can be investigated:

*A. MAGNETIC SPLITTING.* The ground state of  $^{57}\text{Fe}$  in a magnetic field  $B_z$  is split into two energy levels,  $E_g(I, M_I) = \mu_g B_z (M_I/I)$ , with  $M_I = \pm 1/2$ , and  $I = 1/2$ . The 14.4 keV excited state is split into four levels given by  $E_e(I, M_I) = \mu_e B_z (M_I/I)$  with  $M_I = \pm 3/2, \pm 1/2$ , and  $I = 3/2$ . Here,  $\mu_g = +0.0903 \mu_n$  where  $\mu_n = 3.1525 \times 10^{-8}$  eV/Tesla is the nuclear magneton. The internal magnetic field  $B_z$  in pure (natural) iron is about 30 Tesla. The splittings are of the order of  $10^{-7}$  eV,

much smaller than the 14.4 keV energy difference between the ground state and the first excited state. Transitions allowed between these levels are indicated in Fig. 2.

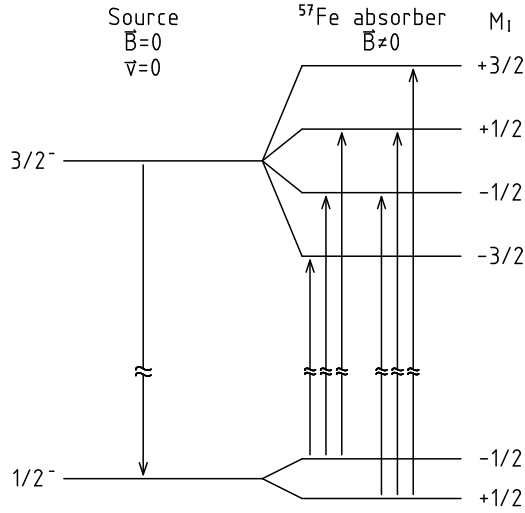


Figure 2: Allowed transitions in  $^{57}\text{Fe}$ , following the selection rule  $\Delta M_I = 0, \pm 1$ .

*B. ISOMER SHIFT (or CHEMICAL SHIFT).* The  $S$  electrons from the atom have a significant probability of being at the nucleus (see any modern physics or chemistry textbook). Other electrons may affect the  $S$  electrons, so they indirectly will have an effect on the nucleus. A nucleus in the excited state is larger than a nucleus in the ground state, so the nucleus in the excited state will have a larger overlap with the  $S$  electron than when in the ground state. This will decrease the energy of the excited state relative to the ground state. If the source and absorber have different values of  $|\psi(r=0)|^2$  (i.e., different electron probability densities at the site of respective nuclei), there will be a shift in the resonance energy of one relative to the other, see Fig. 3(a). These shifts will vary from zero to energies of the order of  $10^{-7}$  eV. Note: although Fig. 3(a) shows the isomer shift as a shift in only the excited state, the ground state (and any other excited state) may also show a shift; moreover, shifts occur in the states of both the source and target, but the spectrometer can only show a difference between the energy levels between the source and target. Iron in non-magnetic stainless steel does not have a (strong) magnetic field at the site of the nucleus (and thus no visible magnetic splitting), but has a different shift in energy levels than iron in the Rh-matrix of the source.

*C. ELECTRIC QUADRUPOLE SPLITTING.* This splitting arises from the interaction between the gradient of the electric field at the site of the nucleus of the source and/or absorber and the quadrupole moment of the source or absorbing nucleus. The energy shift  $\varepsilon$ , in simple cases, is given by

$$\varepsilon = eQV_{zz} \frac{3m_I^2 - I(I+1)}{4I(2I-1)}. \quad (3)$$

This equation gives the energy of the quadrupole interaction for an axially symmetric electric field [6, 7]. The quantity  $V_{zz}$  is the electric field gradient,  $e$  is the charge of an electron and  $Q$  is the quadrupole moment. The overall energy scale is set by  $eQV_{zz}$  with the various levels given by different values of  $I$  and  $m_I$ . You should be able to show that  $\varepsilon$  has two values, above and below zero, for  $I = 3/2$ . Note that for  $I = 1/2$ ,  $\varepsilon = 0$  since the quadrupole moment  $Q$  vanishes.

The splitting produced in  $^{57}\text{Fe}^{++}$  in  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  is shown in Fig. 3(b). The two transition lines

resulting in this case have energy differences in the  $10^{-7}$  eV range.

Although the difference in energy between the different transitions is larger than the natural linewidth of the radiation emitted by the source, it is possible to observe and measure these differences in various absorbers by Doppler shifting the radiation emitted by the source. The Doppler shift, to first order, requires source velocities given by

$$\Delta h\nu/h\nu = v/c, \quad (4)$$

with  $v$  the velocity of the source. For the magnetic splittings given above,

$$v \approx \frac{10^{-7}\text{eV} \times 3 \times 10^8\text{m/s}}{1.44 \times 10^4\text{eV}} \approx 2 \times 10^{-3}\text{m/s}. \quad (5)$$

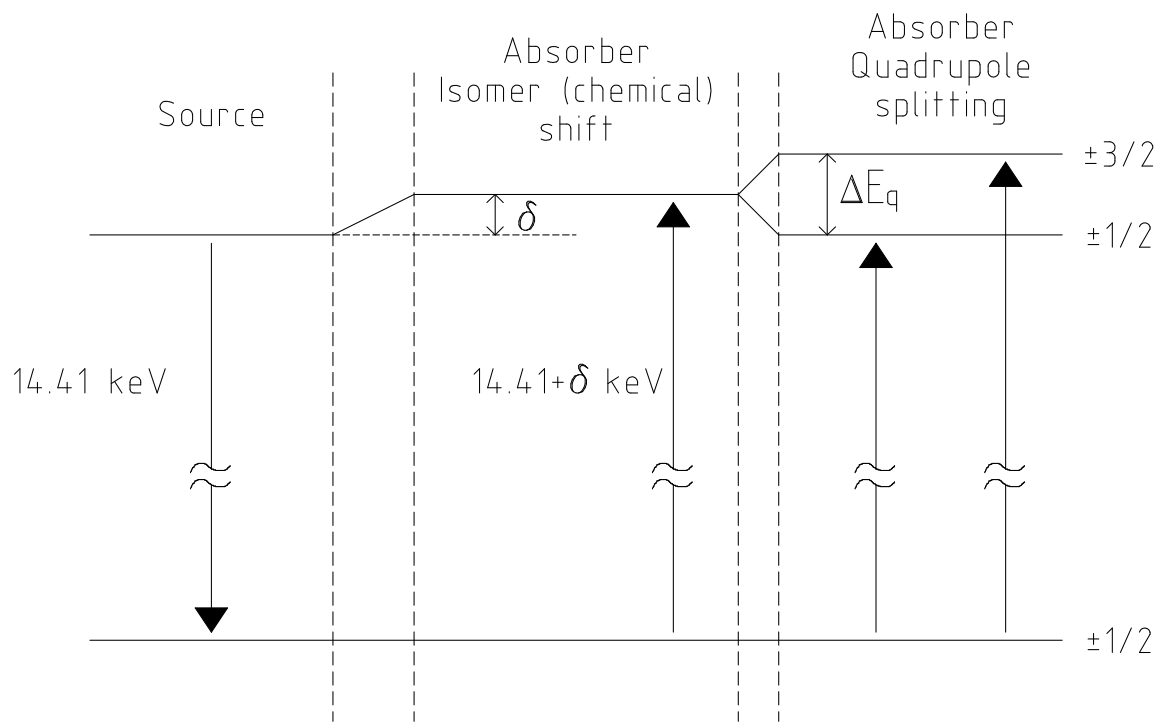
Thus the typical Mössbauer spectrometer has a source mounted on a drive which can move at different velocities up to several millimeters/second, and a fixed absorber, see Fig. 4. Our source moves at constant acceleration, thus the velocity changes linearly with time, from moving forward (towards the absorber) to moving backwards (away from the absorber). The velocity waveform is triangular, not sinusoidal! A radiation counter is placed behind the absorber. When radiation from the source is not absorbed, the counter gives a certain number of counts/second. When the source moves and Doppler shifting of the radiated energy occurs, absorption at the absorber decreases the number of counts/second at the counter (the excited nuclei in the absorber re-radiate within  $\approx 10^{-7}$  s, but in all directions).

### 3 *The Mössbauer Experiment*

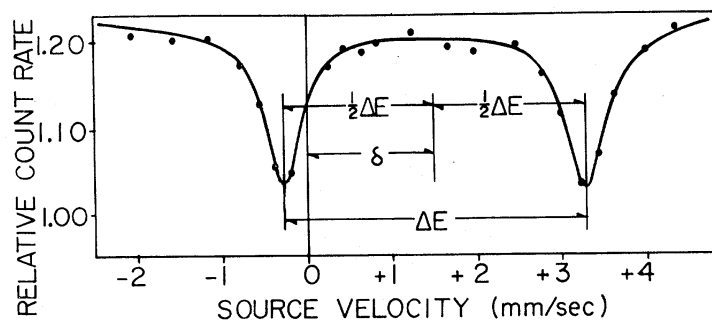
The spectrometer used in this experiment is a research quality instrument and should be treated with great care. Please refer to Fig. 4 for a schematic diagram of our experiment. Inside the lead-lined wooden box are several components of the spectrometer system. The large cylindrical object resting in the aluminum cradle is the drive unit with the source attached to one end of the drive shaft and the velocity transducer attached to the other end. The absorber being studied is placed between the source and the “proportional-tube” detector, the device which detects the photons coming from the source. The detector is the smaller cylindrical object at the right side of the box.

#### **IMPORTANT PRECAUTIONS!!!**

- The source is moderately strong (current activity posted on the lead-lined box), and one should exercise caution to minimize exposure to the source. The radioactive material is on a foil that is located in the recessed center area of the source. Do not touch the source as the foil is fragile and can easily be damaged. The only reason to open the box is to check that the drive unit is operating properly, or to change absorbers. Keep the lid closed unless necessary to open it for one of these reasons. Neither of these operations takes very long, so there is no reason to have the lid open for prolonged periods of time. With the lid closed, radiation exposure in the vicinity of the box is very small due to the lead lining.
- The proportional-tube window is made of beryllium (Be) and it is very thin. Do not touch the window under any circumstances as it is easily damaged, and you may contaminate your hand with BeO, which is toxic in sufficient quantities.



(a)



(b)

Figure 3: (a) Energy level diagram showing isomer (chemical) shift and electric quadrupole splitting in  $^{57}\text{Fe}$  nuclei. (b) Figure 1 from DeBenedetti, Lang and Ingalls [8]. Mössbauer spectrum of the  $\text{Fe}^{++}$  ion in an absorber of  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  at liquid nitrogen temperature using a room temperature stainless steel source. The pattern exhibits the chemical shift  $\delta$  and the electric quadrupole splitting  $\Delta E$  of the excited state of the  $^{57}\text{Fe}$  nucleus. The velocity is positive for the source approaching the absorber.

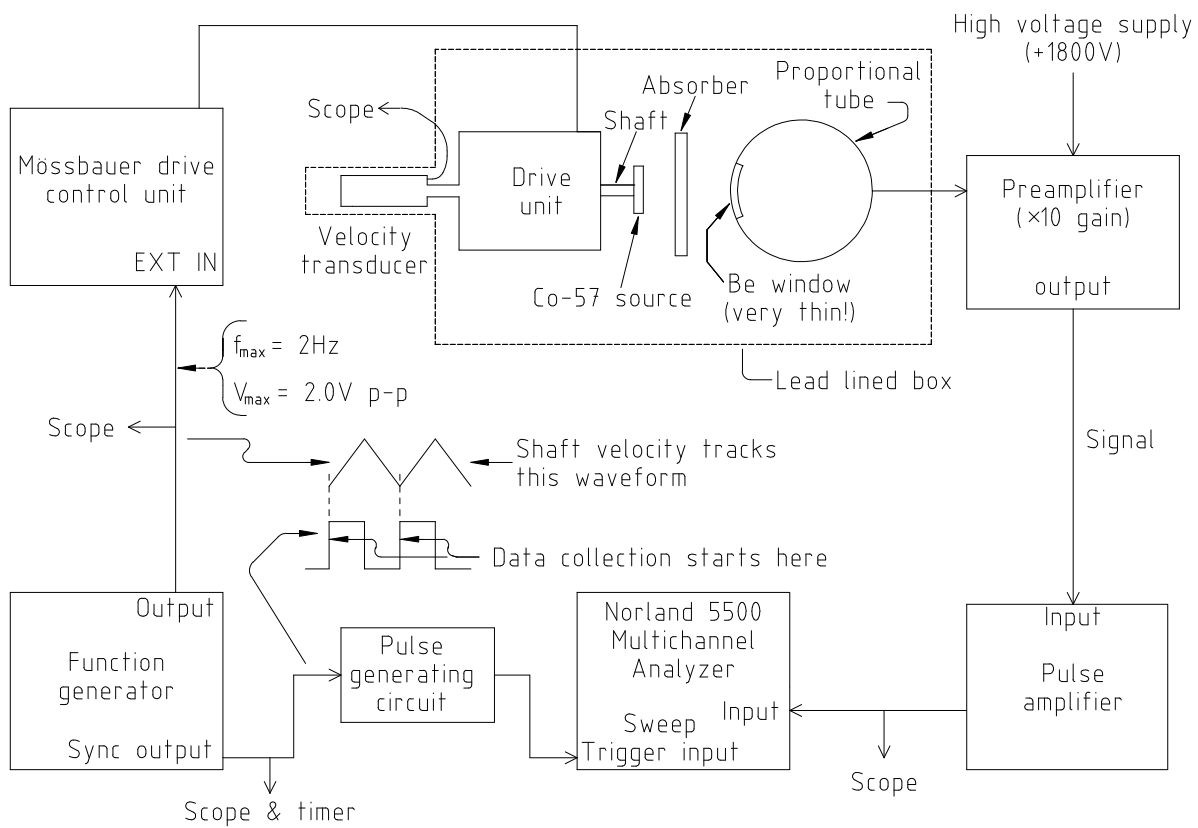


Figure 4: Diagram of the apparatus used in the Mössbauer Effect experiment.

- The proportional-tube requires a high voltage bias of +1800 volts; the power supply for this tube can source enough current to make it dangerous. Do not connect or disconnect any of the high voltage cables with the high voltage on.

### 3.1 Setup of the Mössbauer Apparatus

The Mössbauer apparatus may be understood as consisting of two main portions:

1. The detector channel consisting of the detector, high-voltage supply, amplifiers and discriminator electronics.
2. The velocity-drive channel consisting of the velocity drive unit and its control box, the velocity transducer, the triangle-wave generator, the MCS start-pulse generator, and the MCS sweep electronics.

Proceed with the set-up of these portions as follows to insure good-quality data.

#### 3.1.1 Velocity drive setup

The velocity of the drive shaft tracks the input waveform to the drive control unit. With a triangular wave input, the shaft velocity changes uniformly in time, that is, the acceleration is constant. For our setup, this drive waveform should have a frequency of about 1 Hz, and the amplitude should never exceed 2 volts peak-to-peak. *The amplitude of the signal from the velocity transducer should not exceed 100 mV peak-to-peak.*

1. Direct your attention to the the Elscint Mössbauer Driving Unit. Make sure the “TRANSDUCER” switch is OFF, and the “ATTENUATOR” knob is fully counterclockwise (CCW). Then turn the power switch to ON.
2. Turn on the power to the Wavetek 29 digital function generator, the digital oscilloscope, and the 5 volt power supply connected to the homemade pulse generating circuit.
3. Use the oscilloscope to look at the waveform from the function generator. You want to produce a waveform with the following characteristics: triangle-wave, amplitude of 1.00 volts peak-to-peak, frequency equal to 1.00 hertz. The “sync” signal (AUX OUT) should have its rising edge at the trough of the triangle wave. To set these characteristics, either use the setup memory recall on the Wavetek 29, or follow these operations:
  - (a) Among the FUNCTION keys, select the one marked TRIANGLE.
  - (b) Push the FREQ/PER button, and then use the DIGIT buttons to position the cursor on the numerical part of the frequency reading.
  - (c) Use the keypad to enter ‘1’, and then the unit key ‘Hz ms dBm’ to set the frequency to 1.00 Hz.
  - (d) Use the FIELD buttons to position the cursor on the numerical part of the amplitude field.
  - (e) Use the numerical keypad to enter ‘1’, and then the unit key ‘MHz ns V’ to set the amplitude to 1.00 V<sub>pp</sub> (volts peak-to-peak).

- (f) Push the EDIT (dark blue) and then the TRIG buttons.
  - (g) Use the FIELD buttons to move the cursor to the numerical part of the phase field.
  - (h) Use the keypad and ENTER keys to set the phase. For example, push ‘9’, ‘0’, ‘+/-’ and ENTER to set the phase of the AUX OUT signal to  $-90^\circ$  relative to the MAIN OUT.
  - (i) Confirm that the phase reads ‘-090’ degrees, then press the ‘ESC’ key to return to the main window.
4. On the Wavetek 29, turn the MAIN OUT to ON, and make sure that waveform conforms to the limits of less than 2.0 volts peak-to-peak with a frequency less than 2 Hz.
  5. Make sure that the cable from the velocity transducer is plugged in to Channel 2 of the digital oscilloscope, and that a trace from this source is visible. (The scope should be running on “auto trigger” or “roll”, and the triangle wave from the signal generator should be visible on Channel 1.)
  6. Turn the TRANSDUCER switch on, and wait about 15 seconds for the transducer trace to settle.
  7. *SLOWLY* turn the ATTENUATOR knob clockwise until you see the oscillating signal from the velocity transducer appear. Adjust the scope controls so that you can comfortably view a waveform of 100 mV peak-to-peak, and set the attenuator to give a waveform of about 50 mV peak-to-peak. This is good to start with.
  8. Finally, open the lid to the lead-lined box, and note the back and forth motion of the source.

When the Mössbauer data are collected, you will need to know as accurately as possible the peak-to-peak amplitude of the waveform from the velocity transducer in order to calibrate the energy scale of the spectrum. Since the velocity transducer is sensitive to extraneous electromagnetic fields, the signal is typically very noisy. Once you have started a data collection run, you should use the waveform averaging feature of the oscilloscope to reduce the noise on the transducer waveform. Then you may use the “Measure” feature of the scope and/or the screen cursors to accurately obtain the peak-to-peak amplitude.

### *3.1.2 Counter setup*

The proportional-tube is a radiation detector operated in such a manner that the size of the outgoing pulse is proportional to the energy of the incident particle. The gas filled tube (Kr and Xe) consists of a center wire kept at a high positive voltage (+1800 volts) and the outer shell, which is kept at ground potential. An incident photon produces electrons and ionized atoms. As the electrons move toward the center wire, they are accelerated and produce additional electron-ion pairs. The high voltage is set so that the total amount of charge produced is proportional to the energy of the incident particle. (Can you surmise why this proportionality exists?)

The charge pulse leaving the detector is shaped and amplified by the preamplifier (just behind the wooden box) and the shaping amplifier in the NIM crate. The height (maximum voltage) of the output pulse from the shaping amplifier is proportional to the energy of the incident particle.

The MCA can operate in two different modes. In the Pulse Height Analysis (PHA) mode, it displays the number of pulses (vertical axis) as a function of pulse height (horizontal axis). The



source emits photons of several different energies so the lower peak height pulses from the lower energy photons will be toward the left side of the MCA screen, and the higher peak height pulses from the more energetic photons will be further to the right. Most MCA's accept pulses with peak height between 0 and 10 volts, with a pulse of 10 volts height putting a count in the highest channel available, 1023 for our MCA.

In the Multichannel Scaling (MCS) mode, the MCA displays the total number of counts on the vertical axis versus time on the horizontal axis. The MCA is operated in this mode for the Mössbauer experiment, as time on the horizontal axis can be correlated with source velocity, the parameter of interest in analyzing the position and separation of the various absorption lines. In the MCS mode, the MCA stops, or dwells, at each channel for a preset length of time (called the dwell time—same for all channels), increments the number of counts in that channel by the number of counts received during that time, and then moves on to the next channel. The time sweep starts at channel 0, finishes at channel 1023, and is initiated when a proper trigger signal is received by the MCA. There is a notebook in the lab with a brief outline of MCA operation; a detailed manual with a complete description is also available from the lab manager.

For the Mössbauer measurements, we are only interested in the pulses corresponding to the 14.4 keV photons. The Norland MCA has the capability to discriminate against (nearly) all other pulse heights. To make this setting, we must first run the MCA in pulse height analysis (PHA) mode. Then we will use the output of the on-board discriminator to send counts to the multichannel scaling (MCS) input.

First, check out the counter operation:

1. Turn on the power to the analog oscilloscope, the Norland 5500 MCA, the NIM bin and the high-voltage power supply.
2. Set the high voltage to +1800 volts.
3. Check that the Ortec 575A shaping amplifier is set to a coarse gain of 4 and a fine gain of 5.00 (this means “5” showing in the outer ring of the ten-turn knob with the inner dial set to “0”).
4. At this point, you should see positive pulses on the screen of the analog scope. If nothing is in the path between the  $^{57}\text{Co}$  source and the detector, you should see two bright pulse lines of about 0.5 V and 0.2 V peak height. The lower (and brighter) pulses are produced by 6.4 keV *K*-shell x-rays from the electrons in the Fe atoms. The upper pulses are the 14.4 keV “Mössbauer line”.

To set the discriminators, take pulse height spectra as follows:

1. Turn the LLD (lower-level discriminator) knob fully CCW.
2. Use the screwdriver to set ULD (upper-level discriminator) a few turns CW (from the minimum possible value). The specific setting is not important at this step; the ULD simply needs to be high enough to pass the important 14.4 keV peak.
3. Take a pulse height spectrum: Press “RUN” then the “PHA” softkey. If previous data are present, press “0”, “GOES INTO ==>”, and then the “ALL” softkey to clear the data first. You should quickly see a spectrum similar to that shown in Fig. 5a. Note the high peaks corresponding to the 6.4 keV and 14.4 keV energies.

4. Confirm that the lower bright line comes from the 6.4 keV x-rays by placing a packet of aluminum foil in front of the detector and take another PHA spectrum—the line should diminish in intensity greatly, but the 14.4 keV line will remain. You should also see a smaller peak, even further to the right, due to 20.5 keV x-rays from the rhodium matrix in which the  $^{57}\text{Co}$  is embedded.
5. Once you have identified the 14.4 keV peak for certain, take repeated PHA spectra while adjusting first the LLD knob and then the ULD screw adjustment to narrow down the discriminators to pass only that peak. Your final spectrum should look something like Fig. 5b. Ideally, the discriminators should be set to pass the peak at its full width at half maximum (FWHM).

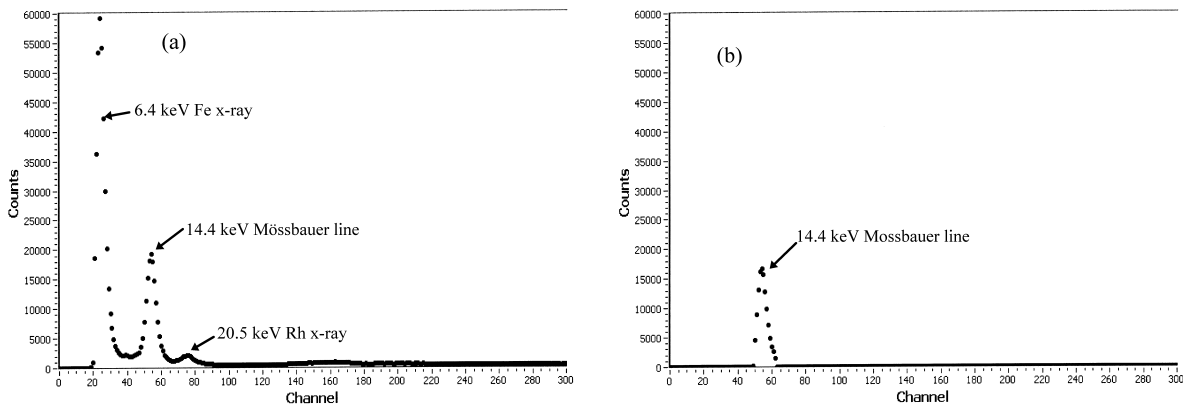


Figure 5: Pulse height analysis spectra seen in the setup of the counter discriminators. (a) Full spectrum from the proportional counter. Note the peaks corresponding to the Fe (6.4 keV) and Rh (20.5 keV) x-rays along with the 14.4 keV Mössbauer line. (b) Spectrum taken with upper- and lower-level discriminators set to pass only the 14.4 keV line.

Once the discriminators are set, they will not need to be adjusted further. Now take a moment to look at the back of the Norland MCA. Notice that we have connected a line called “SCA out” to the “MCS input”. This sends the logic pulses produced by the discriminator circuit to the multichannel scaling input.

### 3.2 Taking Mössbauer spectra

Start your data taking with the enriched  $^{57}\text{Fe}$  absorber; this gives the quickest data set and will reveal any problems with your setup. This particular target is in a small brass holder with rubber feet and is simply placed in front of the detector. All of the other targets are mounted in 1/8”-thick brass squares that fit into the slot cut into the aluminum base in front of the detector.

Check that the source is oscillating back and forth, and that the velocity transducer is producing a triangle wave of 50 mV peak-to-peak at 1 Hz.

Check to make sure that the “MCS Trigger Control” box switch is set to “Trigger Enable”.

Set the MCS parameters on the Norland MCA:

1. Press “SET UP” and then the “MCS” softkey.

2. Move through the highlighted fields at the bottom of the screen with the “CONT” key.
3. Highlight the number of sweeps field “SW=”, and key in the number of sweeps you want. Note: “9999” is the maximum and a good choice to start; you can stop the run at any time by pressing “TERM”.
4. Highlight the “T/CH=” field and set the dwell time per channel to 500  $\mu$ s by pressing “5” and then the appropriate “ $\times 10$ ” or “ $\div 10$ ” softkey.
5. Finally press “TERM”.

You are ready to go! Start the MCS run as usual: “RUN” followed by “MCS”. You should see the counts accumulate by apparent sweeps, and the runs will be counting down from their maximum as shown at the top of the MCA screen. In a few moments, you should also begin to see absorption peaks in the spectra which indicate absorption of the gamma rays by the target at specific energies. These peaks correspond to absorption transitions indicated in Fig. 2.

If you see these peaks, everything is working fine, and you are ready to take data in earnest. You may want to adjust the “ATTENUATOR” knob on the Mössbauer Driving Unit to optimize the peak pattern so that you see the complete pattern with good resolution.

Once data collection has begun, when to terminate it is something of a judgment call: some targets like the enriched  $^{57}\text{Fe}$  will produce a usable spectrum in 10-20 minutes; others, like the sodium nitroprusside, will require an overnight run. Please work with the TA and lab management when you start a very long run; they will insure that your data collection is left undisturbed.

As implied above, in the MCS mode, the MCA will execute, at maximum, 9999 sweeps and then stop. At an effective rate of approximately 1-sweep per second, 9999 sweeps requires slightly less than three hours of data accumulation. In order to have the MCA run longer (i.e., overnight), one can program it to repeat the 9999 sweeps:

1. With the MCA in the idle mode (READY annunciator visible at lower left corner of screen) press the PROG key and the prompt “SELECT PROGRAM NUMBER” will appear.
2. Press any key between 1 and 9. This will be the number of your program.
3. Press the “EDIT” key.
4. Press the “RUN” key and then the “MCS” key. The program line RUN MCS need appear only one time.
5. Press the “EXIT EDIT” key.

To run the program, press the “RUN” key, the number key(s) for the number of times you want the program to execute (can be up to 999), then the “PROG” key, and finally the number of your program. The program will now begin execution. To stop it at any time, press the “TERM” key twice. *Note: Although the data memory for the MCA is non-volatile, the programs are lost when the MCA is turned off.*

When you have finished accumulating data for a particular sample, the data can be transferred from the MCA to a computer for graphing and analysis.

To transfer data to the computer, turn on the computer and login according to the TA or manager’s instructions (it may already be running). Open the LabVIEW application “Norland Interface”

which should be available on the desktop. When the application is loaded, start the program by clicking on the “arrow” button at the top left corner.

When the program starts, it will show the most recent data set that was loaded from the MCA. To transfer your data to the computer:

1. If the MCS or PHA run is still going, terminate it by pressing “TERM” a couple of times, and make sure that the READY annunciator is visible at the lower left corner of the MCA screen.
2. Click the green “Read data from MCA” button.
3. Press “RUN” followed by the softkey “I/O” on the MCA.

You will see the “I/O” indicator light up on the MCA—this indicates that data is being sent over the serial line to the computer. As soon as the I/O indicator goes out (about 15 seconds), you will see your data appear on the screen.

After your data has loaded, you can save it to a file, analyze the data with the peak-fitting routine, print the data and comments on the lab printer, and load old data sets for examination and analysis. Instructions for the program are available from the “SHOW INSTRUCTIONS” button. You should make sure to save your data to a floppy disk or other off-computer storage device (i.e., email, memory stick, etc.); we cannot guarantee that your data will remain on the computer.

### 3.3 Energy calibration

After you begin a data collection run there should be plenty of time to work out your energy calibration calculations. You need to bring together a few facts:

As mentioned above, the time sweep is synchronized with the motion of the source, and for our setup, the time sweep is triggered when the source is moving *toward the absorber with the highest velocity* (the output of the velocity transducer is *negative* when the source is moving towards the absorber and the proportional-tube). To calibrate the horizontal axis of the MCA, one needs to know the dwell time of the MCA (typically 500  $\mu\text{s}$  per channel, but check to be sure), the period of the triangular waveform, and the peak-to-peak voltage output from the velocity transducer. The conversion factor (voltage to velocity) for the velocity transducer is

$$0.295 \pm 0.002 \frac{\text{mm/s}}{\text{mV}} .$$

This calibration has been deduced by direct observation of the source displacement as a function of transducer output along with comparisons of data to standard Mössbauer spectra. A data sheet for this device is available in the lab, which shows its construction and nominal output.

It is helpful to perform the energy calibration in steps: First, determine the velocity-versus-channel conversion factor. When you fit your peaks to find the best-fit peak position, convert these numbers to velocity shifts in millimeters per second—these are the units usually reported in the literature, and make for easiest comparison. Then determine the energy-versus-velocity conversion factor; re: Eq. (4). The product of these two conversion factors yield your calibration, which should be  $< 1.2 \text{ neV/channel}$ . Hint: the cursors on the digital scope are very useful in performing the energy calibration.

## 4 Student Experiments

Following the topics discussed in Section 2, a few standard experiments are possible. You should perform at least 3 of these.

### Magnetic splitting

Measure the magnetic splitting in an absorber of enriched  $^{57}\text{Fe}$ . Since the absorber has been enriched with the right isotope of Fe, it is easy to see the six absorption lines after half hour of data collection. As little as 5 minutes of collection time yields a visible spectrum, though 30 minutes will give more than adequate statistics. If you do not see the six lines on the MCA there is some problem which needs to be fixed. Given Figs. 1 and 2, and your calibration of the velocities of the source, you can determine two quantities:

1. The magnitude of the magnetic field at the site of the iron nucleus.
2. The magnetic moment of the excited state at 14.41 keV.

Doing just this part well takes considerable care. You will need to identify which absorption line comes from which transition. Then you can determine how the differences between levels in Fig. 2 correspond to differences between certain peaks in the spectrum. The energies of the ground and excited state are each altered by the magnetic field according to  $E \rightarrow E - \vec{\mu} \cdot \vec{B}$  (so the lower energy state corresponds to the moment  $\vec{\mu}$  aligned with  $\vec{B}$ ). The energy of absorption by the target is given by the *difference* between the ground and excited states:

$$E_{\text{abs}} = E_e - E_g = \Delta E_0 - \mu_e B \frac{M_{I,e}}{I_e} + \mu_g B \frac{M_{I,g}}{I_g}, \quad (6)$$

where the subscripts  $g$  and  $e$  refer to the ground and excited states, and  $\Delta E_0$  is the energy of the unsplit or zero-field transition (which may include an isomer shift). By considering different values of  $M_I$  for the transitions, you can derive equations to give you  $\mu_e$  and  $B$ , using the known value of  $\mu_g = +0.0903\mu_n$  [9].

It is interesting to compare the enriched sample to the natural iron samples. The proportion of  $^{57}\text{Fe}$  is much less, so you will need to count for a longer period to get a good spectrum.

You can also prove that the sign of  $\mu_e$  is negative if you know that the intensity of the absorption lines in natural, unmagnetized, iron is greatest for the transitions involving the  $|\frac{1}{2}, +\frac{1}{2}\rangle$  to  $|\frac{3}{2}, +\frac{3}{2}\rangle$  and  $|\frac{1}{2}, -\frac{1}{2}\rangle$  to  $|\frac{3}{2}, -\frac{3}{2}\rangle$  states, and weakest for the transitions involving the  $|\frac{1}{2}, +\frac{1}{2}\rangle$  to  $|\frac{3}{2}, -\frac{1}{2}\rangle$  and  $|\frac{1}{2}, -\frac{1}{2}\rangle$  to  $|\frac{3}{2}, +\frac{1}{2}\rangle$  states. See the paper by Preston, Hanna and Heberle [10] for details and other useful information.

### Isomer shift

Measure the isomer shift in an absorber of stainless steel. This absorber has only the natural abundance of  $^{57}\text{Fe}$ , 3% in nature, thus you need roughly 33 times the counting time you used for enriched iron to get data of quality comparable to the one for magnetic splitting. Experience has shown that for this sample, the shift is quite small.

In some runs, you can see what looks like two overlapping peaks. This is apparently an artifact of the data collection system. Look closely at the output of the velocity transducer. For small

amplitudes (i.e., 15 mV peak-to-peak), there is a distortion near the zero crossing point, indicating that the driver pauses momentarily near zero. This momentary pause would cause a few MCS channels to have the same number of counts. If this happens between peaks, one would not see any feature, but if it happens within a peak, it creates a shoulder.

You should also calculate the isomer shift of your other samples relative to the  $^{57}\text{Co}/\text{Rh}$  source by locating the center of the spectrum in each case.

## Quadrupole splitting

Measure the quadrupole splitting in an absorber of sodium nitroprusside,  $\text{Na}_2\text{Fe}(\text{CN})_5\text{NO}\cdot 2\text{H}_2\text{O}$ . This sample has some enriched  $^{57}\text{Fe}$ , but the number of iron atoms in the compound is small. Thus you also need to have the spectrometer running for a considerable time to acquire good data. (This target is the weakest absorber in the collection.)

Also calculate the isomer shift of this sample. Compare your results to the published values of 1.7048 mm/s for the splitting and -0.257 mm/s for the isomer shift relative to  $^{57}\text{Fe}$  [11]. (Your data will be relative to the  $^{57}\text{Co}/\text{Rh}$  source; how can you convert?)

## Temperature shift

As an optional experiment (the other three must be done first, if you wish to do this one), you may wish to explore the effect of temperature on the absorber. Because of the extremely fine resolution available with the Mössbauer technique, experiments have been performed which are sensitive to *relativistic* effects in the Doppler shift associated with gamma ray absorption. Such effects also go by the name of “second-order Doppler shift” effects, since the important shift is proportional to  $v^2/c^2$ . The temperature shift comes about because the mean-square vibration of the iron ions increases as the temperature increases. A simple calculation gives the result for the shift of frequency of absorption  $\delta\nu$  as a function of temperature change  $\delta T$  as

$$\delta\nu = \left(\frac{\partial\nu}{\partial T}\right)\delta T = -\left(\frac{\nu C_p}{2Mc^2}\right)\delta T, \quad (7)$$

where  $C_p$  is the specific heat and  $M$  is the molar mass of the absorber’s lattice. Details of the theory concerning this effect are given in the paper by Pound and Rebka [12].

We have a small liquid nitrogen dewar with its own enriched iron absorbed inside. With it you can repeat the experiment on magnetic splittings above and measure the direction and magnitude of the shift of the peaks. Compare the results with the predictions from the theory given in the references. You must have the lab manager set the apparatus up for you if you wish to do this measurement.

You will see a couple of effects as you cool the sample: a change in the peak splittings as well as a shift in the centroid of the pattern. Can you figure out what is going on?

## Magnetic and Quadrupole effects combined

the Mössbauer spectrum of hematite, or  $\alpha\text{-Fe}_2\text{O}_3$ , shows effects of magnetic splitting and quadrupole splitting combined. The internal magnetic field is about 1.5 times stronger than in natural iron, so

you will need to increase the maximum velocity of the source correspondingly in order to see the full spectrum.

The quadrupole splitting is seen as an energy shift  $\varepsilon$  which is added to the energy shift from the magnetic interaction. Thus, the absorption energy Eq. (6) gets another term:

$$E_{\text{abs}} = \Delta E_0 + \mu_g B \frac{M_{I,g}}{I_g} - \mu_e B \frac{M_{I,e}}{I_e} + \varepsilon, \quad (8)$$

where  $\varepsilon$  is given by Eq. (3). The effect of the additional energy  $\varepsilon$  on the Mössbauer spectrum is to shift each absorption peak to the right or left of where it would be if only magnetic interactions were present.

For this experiment, you should plan an overnight run (at least) since the absorber is fairly weak. Then analyze the resulting spectrum to find the peak positions. Estimate the internal magnetic field strength from the average of the appropriate peak positions, and then calculate the expected peak positions assuming magnetic effects only. Finally, calculate an estimate for  $\varepsilon$  using the difference between the expected peak positions and the actual peak positions. Compare your results with those give in the paper by Kistner and Sunyar [6].

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