

# Measuring the Polarization of a $^3\text{He}$ Target Cell Using Electron Paramagnetic Resonance

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## Abstract

Researchers at the Thomas Jefferson National Accelerator Facility perform electron scattering experiments in order to gain insight into the substructure of the neutron. For these experiments, polarized  $^3\text{He}$  is used to provide a target of polarized neutrons. Although a  $^3\text{He}$  nucleus consists of two protons and one neutron, the anti-alignment of the protons' spins leaves the nucleus with the effective spin of the neutron. The College of William and Mary has a lab specifically designed to fill the cells and to measure polarization of  $^3\text{He}$  which is contained in glass target cells. A relative polarization measurement can be obtained using a nuclear magnetic resonance (NMR) technique known as adiabatic fast passage (AFP). This year an electron paramagnetic resonance (EPR) system was developed to make precision measurements of absolute polarization. EPR utilizes the fact that there is rubidium in the target cells in addition to  $^3\text{He}$ , which is used to help polarize the  $^3\text{He}$ . EPR polarimetry measures the shift in the rubidium Zeeman resonance which is caused by the presence of the polarized  $^3\text{He}$ . This shift is directly proportional to the polarization of the cell. Therefore by measuring the frequency shift of rubidium EPR frequency, the absolute polarization of the cell can be known.

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# 1 Introduction

To learn more about the substructure of the neutron, researchers at the Thomas Jefferson National Accelerator Facility conduct electron scattering experiments on neutron targets. However, the instability of the neutron itself limits its use in experiments. Free neutrons decay in around 10.61 minutes, but polarized  $^3\text{He}$  has been found to be a feasible alternative for these experiments.

A ground state  $^3\text{He}$  nucleus consists of a neutron and two protons which are predominantly in the S-state. In this configuration, the spins of the protons essentially cancel, effectively leaving the nucleus with the spin of the neutron. This result makes  $^3\text{He}$  ideal for experiments involving long-term study of the neutron. More importantly, however, in order to produce accurate experimental results, the spins of the neutrons need to be pointing in the same direction. In other words, the nuclei of the  $^3\text{He}$  must be polarized.

The College of William and Mary is able to polarize glass cells filled with  $^3\text{He}$  and rubidium (Rb). The cell is placed in a hot oven, which vaporizes the Rb and allows for interaction between the  $^3\text{He}$  gas and the Rb. Next, circularly polarized laser light is shined onto the cell, polarizing the rubidium through optical pumping. Once the rubidium is polarized, it polarizes the  $^3\text{He}$  through spin exchange [3].

In order for the cells to be useful, they must reach high polarization, and they must be able to retain this polarization for extended periods of time. After a cell is filled and polarized, experimenters must know exactly how many spins are pointing in one direction as opposed to the other, a quantity defined as polarization. The current nuclear magnetic resonance (NMR) system provides a method for measuring relative and absolute polarizations, but the absolute measurements require a separate and difficult water calibration procedure [4, 5]. This year an Electron Paramagnetic Resonance (EPR) system, which allows for direct measurements of polarization, was added to the target setup at William and Mary. EPR shifts the focus of attention onto the rubidium in the cell, which is directly affected by the presence of the aligned  $^3\text{He}$  nuclei [1, 2]. By studying the shift of the rubidium Zeeman frequency due to the presence of polarized  $^3\text{He}$ , the precise and absolute polarization of the cell can be found.

## 2 Polarization

If the magnetic moment,  $\mu$ , of the nucleus is placed in an external magnetic field, it aligns itself either parallel or anti-parallel to that field. Polarization is defined by the following equation,

$$P = \frac{N^\uparrow - N^\downarrow}{N^\uparrow + N^\downarrow}, \quad (1)$$

where  $N^\uparrow$  is the number of spins aligned parallel to the external field and  $N^\downarrow$  is the number of spins anti-aligned. Therefore the polarization will equal zero when the spins are equally distributed in the two quantum states.

Figure 1 depicts a typical target cell used in the polarization process. The cells are made of aluminosilicate glass and are hand-blown by a glass blower. Polarization occurs in the pumping chamber, while the target chamber is ideal for electron scattering experiments. The cells are filled with  $^3\text{He}$  at a pressure of approximately 10 atm. Rubidium and trace amounts of nitrogen are also added to the cell to help with the polarization process.

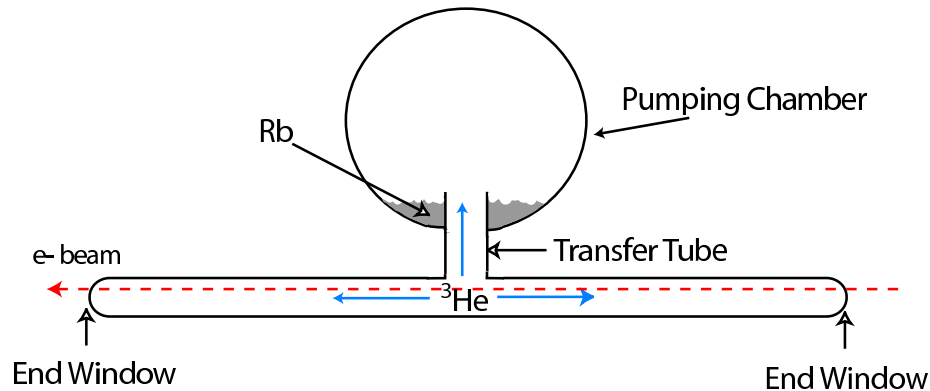


Figure 1: Polarization occurs in the pumping chamber when polarized rubidium transfers its spin to the  $^3\text{He}$  nuclei. The bottom target chamber is used during electron scattering experiments.

### 2.1 Optical Pumping

In order to measure the polarization of a cell, it must first be polarized using the setup shown in Figure 2. To polarize the  $^3\text{He}$  nuclei, lasers first polarize the rubidium vapor in

the pumping chamber of the cell. The upper chamber of the cell is placed in a 170 °C oven to vaporize the Rb. The Rb gas remains trapped in the warmer pumping chamber, while the  $^3\text{He}$  gas is free to move about the entire cell. The presence of an external magnetic field created by two large Helmholtz coils then induces Zeeman splitting in the atomic states of the Rb gas. This effect splits both the  $5S_{\frac{1}{2}}$  ground state and  $5P_{\frac{1}{2}}$  first excited state into two states,  $m = \frac{1}{2}$  and  $m = -\frac{1}{2}$ , each with a slightly different energy [1].

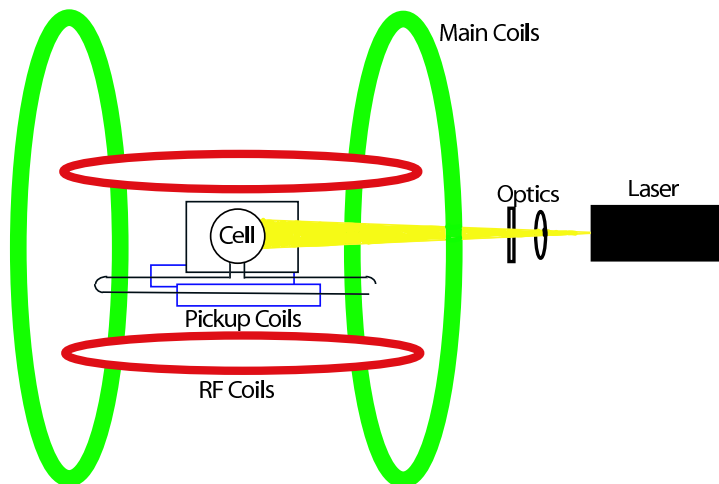


Figure 2: To polarize the  $^3\text{He}$ , the cell is placed in an oven to vaporize the rubidium. Two large Helmholtz coils, shown in green, provide an external magnetic field used to induce Zeeman splitting in the Rb energy levels as well as to align the  $^3\text{He}$  nuclei parallel or anti-parallel to the field. Polarization of the cell begins when right circularly polarized laser light is projected on the cell. RF coils, shown in red, flip the direction of the spins when at resonance during NMR. The blue Pickup Coils measure the induced EMF from this magnetic flux.

Polarizing the rubidium involves getting all of the atoms into the  $5S_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  state, a process depicted in Figure 3. To achieve this distribution, two diode lasers (795 nm) shine circularly polarized light onto the pumping chamber. This light excites the electrons in the  $5S_{\frac{1}{2}}$ ,  $m = -\frac{1}{2}$  ground state to the  $5P_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  excited state. From here, the electrons eventually fall to the ground state. If they fall to the  $5S_{\frac{1}{2}}$ ,  $m = -\frac{1}{2}$  state, they are optically pumped back up, but if they fall to the  $5S_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  state, they remain in place. In time, the majority of the electrons end up in the  $5S_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  ground state [1, 3].

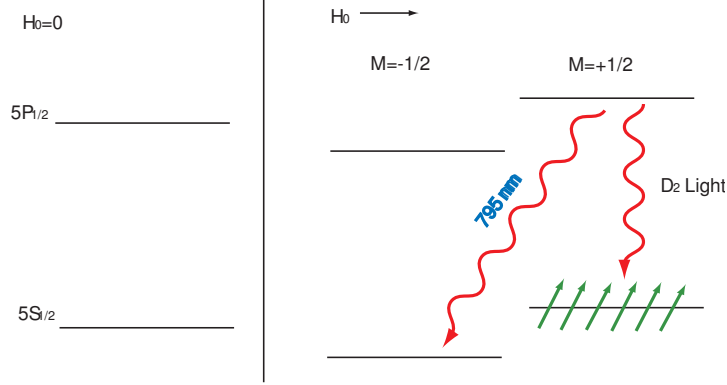


Figure 3: Electrons initially in the  $5S_{\frac{1}{2}}$ ,  $m = -\frac{1}{2}$  state are excited by 795 nm laser light into the  $5P_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  level. From there, they either fall back to the  $5S_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  or  $m = -\frac{1}{2}$  level. If they drop to the  $5S_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  level, they are then optically pumped back to the  $5P_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  state. Eventually all the electrons end up in the  $5S_{\frac{1}{2}}$ ,  $m = \frac{1}{2}$  level, and the rubidium is polarized.

## 2.2 Spin Exchange

The polarization of the Rb electrons allows for the  $^3\text{He}$  nuclei to become polarized. When the rubidium and  $^3\text{He}$  collide within the pumping chamber, a hyperfine-like interaction occurs between the outermost electron in the rubidium and the  $^3\text{He}$  nuclei. This interaction causes the  $^3\text{He}$  spin to align itself to that of the rubidium. In this process the rubidium becomes depolarized; however, the diode laser optically pumps it back to the correct state.

Polarization of  $^3\text{He}$  depends on the spin exchange rate between the rubidium electrons and  $^3\text{He}$  nuclei, the average polarization of the rubidium, and the rate at which the  $^3\text{He}$  depolarizes the cell. The following equation governs the polarization of the cell,

$$P_{He}(t) = \frac{\gamma_{SE}\langle P_{Rb} \rangle (1 - e^{-(\gamma_{SE} + \Gamma)t})}{(\gamma_{SE} + \Gamma)}. \quad (2)$$

In this equation  $\gamma_{SE}$  is the Rb-He spin exchange rate,  $\langle P_{Rb} \rangle$  is the average polarization of the rubidium, and  $\Gamma$  is the depolarization rate of  $^3\text{He}$ . A cell will reach a maximum polarization described by the equation [3],

$$P_{He}(t \rightarrow \infty) = \frac{\gamma_{SE}\langle P_{Rb} \rangle}{(\gamma_{SE} + \Gamma)}. \quad (3)$$

## 2.3 NMR Polarimetry

An easy way to measure the relative polarization of a cell uses a nuclear magnetic resonance (NMR) technique known as adiabatic fast passage (AFP). This technique utilizes the principle that moving magnetic moments induce a voltage in a surrounding coil of wire called pick-up coils. The signal induced in the pick-up coils is proportional to the polarization of the cell.

NMR polarimetry utilizes the 25 G external magnetic field created by two large Helmholtz coils (see Figure 2). In addition, a RF field applied at 91 kHz which is perpendicular to the direction of the holding field is added to the system. At a holding field of 28.6 G and an RF field of 91 kHz, the spins will flip direction because they are at the Larmor frequency, given by  $\nu = \frac{2\mu B}{h}$ . Therefore, the RF frequency is held constant, while the holding field is slowly ramped through resonance. Once the spins flip, the holding field is the ramped back down to 25 G, causing the spins to return to their original orientation [4].

Each time the spins flip, they induce a voltage in the pick-up coils which are next to the target chamber of the cell. This voltage is read by a lock-in amplifier and sent to a computer to analyze the results. The peak height of the signal is proportional to the polarization of the cell. However, unless a water calibration is done on the system using a target cell filled with water, the constant of proportionality is unknown. Because water calibrations are subject to large error, typically only a relative measurement is found using NMR, and this is done by comparing peak height signals from cell to cell. When the polarization of a water ( $10^{-9}$ ) is compared to the polarization of  $^3\text{He}$  ( $10^1$ ), it is obvious to see why the task of a water calibration is so difficult.

As mentioned previously, EPR provides an easier, more precise way to measure polarization. It still utilizes the adiabatic fast passage technique to flip the nuclear spins. For EPR though, it is important that the main holding field be held constant. In order to do this and still take an AFP measurement, the RF frequency sweeps through resonance (91 kHz) while the holding field stays constant (28.6 G).



## 3 EPR Polarimetry

### 3.1 Theory

EPR measures the shift in the rubidium energy levels while in the presence of a magnetic field. The holding field created by the Helmholtz coils is not the only field present in this system. The  $^3\text{He}$  nuclei which are aligned in one direction also create an additional field. By measuring the shift in Rb energy levels due solely to the  $^3\text{He}$ , the polarization of the cell can be found.

The EPR procedure starts by looking at a more accurate depiction of the energy level splitting of Rb as seen in Figure 3. This picture is obtained when the hyperfine interaction between the outermost Rb electron and the nucleus of the Rb is taken into account. This effect splits the rubidium into energy levels, two of which,  $F = 3$  and  $F = 2$ , are relevant to this experiment. With the addition of an external magnetic field, the  $F = 3$  state of Rb splits into seven sublevels  $M_F = -3, -2, \dots, 2, 3$ , and the  $F = 2$  state splits into five sublevels [1]  $M_F = -2, -1, 0, 1, 2$ . The Zeeman splitting between the  $F = 3, M_F = -3$  and  $M_F = -2$  describes the Electron Paramagnetic Resonance (EPR) frequency. This frequency is affected not only by the large external field created by the Helmholtz coils mentioned earlier, but also by the Rb- $^3\text{He}$  spin exchange interaction, and by the local field of the polarized  $^3\text{He}$  [1]. In order to isolate the effect of the polarized  $^3\text{He}$  on the frequency shift, the  $^3\text{He}$  spins are flipped during an AFP NMR measurement. This reversal cancels the effects due to the holding field,  $B_0$ , allowing the frequency shift due purely to the polarized  $^3\text{He}$  to be measured.

Once the rubidium in the cell is polarized by the diode laser, most of the Rb electrons are in the  $F = 3, M_F = 3$  or  $-3$  state. The EPR resonance frequency describes the frequency at which the transition occurs between the  $F = 3, M_F = -3$  and  $M_F = -2$  states (or the  $F = 3, M_F = 3$  and  $M_F = 2$  states depending on the orientation of the external field). Using an EPR coil which induces transitions between neighboring  $M$  sublevels, the EPR resonance frequency can be applied to the system [1]. Once at resonance, those electrons which reside in the  $F = 3, M_F = -3$  level are then driven between the  $M_F = -2$  and  $M_F = -3$  levels. Upon repolarization through optical pumping, 3-5% of the electrons emit either  $D_1$  or  $D_2$  light, while the remaining excited electrons are non-radiatively quenched to the ground state by the nitrogen in the cell. Since the laser light has the same frequency as

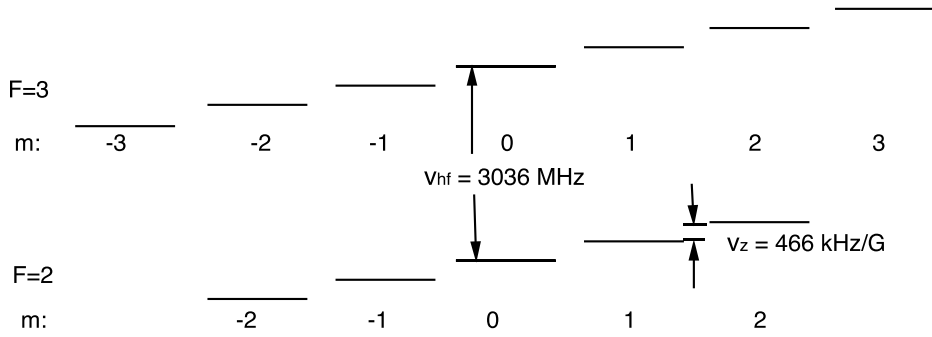


Figure 4: A more accurate description of the splitting of rubidium energy levels due to the hyperfine splitting caused by the interaction between the Rb electron and nucleus, as well as the Zeeman effect caused by the external field, the Rb- $^3\text{He}$  spin exchange, and the polarization of  $^3\text{He}$ . The hyperfine splitting is much larger than the Zeeman splitting.

the  $D_1$  light, it is the  $D_2$  light that is measured by placing a  $D_1$  light filter over a photodiode. The highest intensity of  $D_2$  light measured by the photodiode signifies resonance, meaning that the number of electrons making the transition is at a maximum [7].

To find the equation for the EPR resonance frequency, it is useful to look at the Hamiltonian for Rb in a magnetic field. The Hamiltonian will consist of three parts, one due to the hyperfine interaction between the electron and the nucleus, one due to the Zeeman splitting of the electron energy levels, and one due to the Zeeman splitting of the nucleus. Therefore, the Hamiltonian will take the following form:

$$\mathcal{H} = 2\pi\hbar A \vec{I} \cdot \vec{S} + \vec{S} \cdot \vec{B} + \vec{I} \cdot \vec{B} \quad (4)$$

Here  $A$  is the Rb hyperfine splitting frequency which is measured to be 1012 MHz,  $\vec{I}$  is the nuclear spin of Rb,  $\vec{S}$  is the electron spin, and  $\vec{B}$  is the total magnetic field.

In this experiment, the magnetic field does not come solely from the holding field. There is also a contribution from the magnetization of the surrounding polarized  $^3\text{He}$ . In addition, the spin exchange interaction between the Rb and  $^3\text{He}$  causes a frequency shift corresponding to a effective field shift. Therefore  $B$  can be written as

$$B = B_0 + B_{He} + B_{SE}, \quad (5)$$

where  $B_0$  is the contribution from the external field. It is important to note that the additional fields created by the polarized  $^3\text{He}$  and from the spin exchange interaction are directly

dependent on the amount of polarization of the cell. Since the Hamiltonian, and in turn the energy of the atomic states, is proportional to the magnetic field, it must also be proportional to the polarization.

The solution to this Hamiltonian is given by the Breit-Rabi formula [1]:

$$E_{F=I\pm 1/2, M} = -\frac{A(I+1/2)}{2(2I+1)} - g_I\mu_N BM \pm \frac{A(I+1/2)}{2} \left(1 + \frac{4M}{2I+1}x + x^2\right)^{1/2}, \quad (6)$$

where  $x = \frac{\omega}{2\pi A(I+1/2)}$ , and  $\omega = \frac{2\mu B}{\hbar}$ .

The goal of this derivation will be to find  $\Delta\nu_{EPR}(B)$ , which is proportional to the polarization of the cell. Because the variation of  $\nu_{EPR}$  with respect to  $B$  is small for all purposes used in this experiment,  $\Delta\nu_{EPR}$  will be approximated by the following equation:

$$\Delta\nu_{EPR} = \frac{d\nu}{dB} \Delta B \quad (7)$$

The EPR resonance frequency  $\nu_{EPR}$  is given by the energy difference between the  $F=3$ ,  $M_F=-3$  and  $F=3$ ,  $M_F=-2$  states divided by  $h$ ,

$$\nu_{EPR} = \frac{E_{F=3, M_F=-2} - E_{F=3, M_F=-3}}{h} \quad (8)$$

Because  $x$  is small,  $(1 - ax + x^2)^{1/2}$  can be approximated using a binomial expansion as  $(1 - \frac{a}{2}x)$ , and

$$\nu_{EPR} = \frac{-g_I\mu_N B + \frac{A(I+1/2)}{2} \left(1 - \frac{4}{2I+1}x\right) - \frac{A(I+1/2)}{2} \left(1 - \frac{6}{2I+1}x\right)}{h} \quad (9)$$

$$= \frac{-g_I\mu_N B + \frac{A}{2}x}{h} \quad (10)$$

In this case, the term due to the Zeeman splitting of the nucleus,  $g_I\mu_N B$  can be ignored because it is small when compared to the other terms. The derivative of  $\nu_{EPR}$  with respect to  $B$  is,

$$\frac{d\nu_{EPR}}{dB} = \frac{\mu}{\pi h(2I+1)}. \quad (11)$$

The next step to determine Equation 7 is to find  $\Delta B$ . In this experiment, it is important to note that the shift in EPR frequency,  $\Delta\nu_{EPR}$ , is determined by three factors:

$$\Delta\nu_{EPR} = \Delta\nu_{SE} + \Delta\nu_{He} + \Delta\nu_{B_0}, \quad (12)$$

where  $\Delta\nu_{SE}$  is the contribution from the spin exchange interaction,  $\Delta\nu_{He}$  is the contribution from the polarized  $^3\text{He}$  and  $\Delta\nu_{B_0}$  is the contribution from the holding field. Since  $\Delta\nu_{EPR}$  will be found by changing the direction of the magnetic moments of  $^3\text{He}$  using the frequency sweep NMR technique discussed previously, the effect from the holding field will cancel, and only the parts due to the spin exchange interaction and the polarized  $^3\text{He}$  will remain.

The frequency shift from the spin-exchange interaction and the polarized  $^3\text{He}$  can be found in the same way that  $\Delta\nu_{EPR}$  was:

$$\Delta\nu_{SE} = \frac{d\nu}{dB} \Delta B_{SE} \quad (13)$$

$$\Delta\nu_{He} = \frac{d\nu}{dB} \Delta B_{He} \quad (14)$$

In reference [2], Romalis shows that  $\Delta B_{SE} = (2K_{He}\hbar/T_{He}g_e\mu_B)\langle\vec{K}\rangle$ . Here  $K_{He}$  is a frequency shift parameter,  $1/T_{He}$  is the Rb-He spin exchange rate per Rb atom,  $g_e = 2.00232$ ,  $\mu_B = 5.7884 \times 10^{-11}$  MeV/T, and  $\langle\vec{K}\rangle$  is the average  $^3\text{He}$  nuclear spin. Romalis also states that the contribution from the  $^3\text{He}$  is  $\Delta B_{He} = C\eta_p\mu_{He}\langle\vec{K}\rangle/K$ , where  $C$  is a dimensionless factor that depends on the geometry of the cell,  $\eta_p$  is the density of  $^3\text{He}$  in the pumping chamber,  $\mu_{He} = 6.706984 \times 10^{-14}$  MeV/T and  $\langle\vec{K}\rangle/K$  is the polarization of the cell, denoted by  $P_p$  [2].

By combining these two effects for a sample of a given shape, the following equation governs the EPR frequency shift due to the polarization of the cell [5]:

$$\Delta\nu_{EPR} = \frac{2\mu_0}{3} \frac{d\nu_{EPR}}{dB} \kappa\mu_{He}\eta_p P_p. \quad (15)$$

In this equation,  $\kappa$  is a flux factor which depends on the temperature of the cell. It has been experimentally found to be  $4.52 + 0.00934 T_p(^{\circ}\text{C})$  [2]. Hence, in order to determine the polarization of the pumping chamber of the cell, the EPR frequency shift  $\Delta\nu_{EPR}$  must be known. In addition, the temperature of the pumping chamber  $T_p$  needs to be known in order to determine  $\kappa$ .  $T_p$  is also needed for the measurement of  $\eta_p$ , which will be discussed later. Once these measurements are made, polarization can be found.

### 3.2 Setup

To find  $\Delta\nu_{EPR}$ , the resonance frequency is found while the spins are aligned parallel to the holding field, as well as when they are anti-parallel, and subtracted. In order to determine EPR resonance frequency, a coil of wire is placed close to the pumping chamber of the cell, creating what is called the EPR RF field (see Figure 5). This field is used to induce the transition between the  $M_F = -3$  and  $M_F = -2$  states.

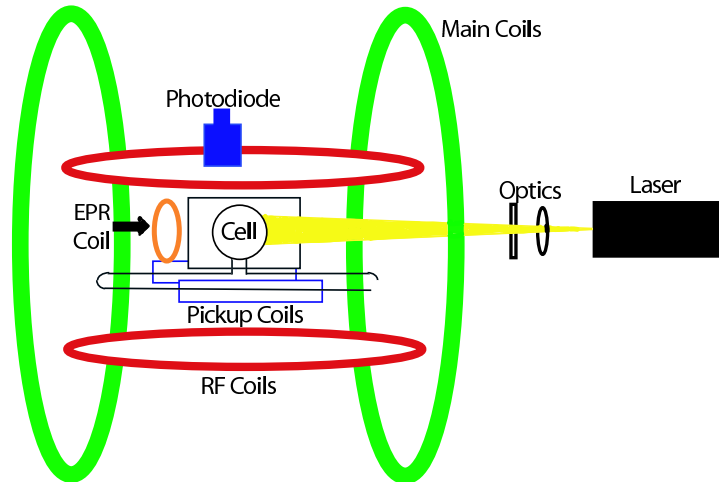


Figure 5: To measure polarization of a cell using EPR, the EPR coils lock on to resonance and the photodiode measures intensity of  $D_2$  light.

A  $D_2$  light photodiode filter, specially designed to detect  $D_2$  light emitted from the pumping chamber, measures the intensity of the signal from the cell when the EPR coil is inducing transitions. As mentioned earlier, EPR resonance corresponds to a peak in the signal height of  $D_2$  light. Therefore, in order to find the resonance frequency, or in terms of signal measured, the highest intensity of  $D_2$  light, the frequency of the EPR RF field can be changed using a Voltage Controlled Oscillator (VCO) Wavetek function generator.

To find resonance, a particular method known as frequency modulation is employed. In this way, the frequency sent to the EPR coil is modulated, while the amplitude of the signal remains constant. This process measures the derivative of the EPR frequency line-shape. Appendix 1 shows how to make a manual FM measurement. The theory behind this measurement is given below.

A schematic diagram of the EPR equipment setup is shown in Figure 6. The mod-

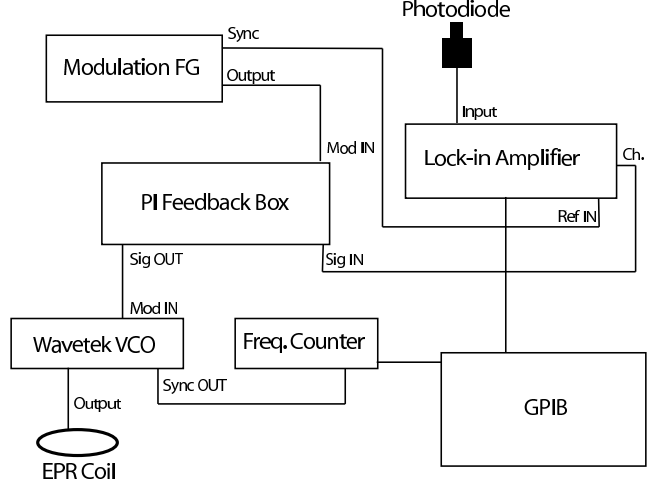


Figure 6: A Wavetek VCO, modulation function generator, proportional-integrator feedback box, and a lock-in amplifier are connected to a computer to measure EPR results.

ulation function generator sends a modulating signal to the input of the PI box,  $V_1 = V_{mod} \sin(2\pi f_{mod}t + \phi_0)$ , and this signal is then sent out to the Wavetek. Here  $V_{mod}$  and  $f_{mod}$  are the modulation amplitude and frequency, and  $\phi_0$  is an arbitrary phase factor. The Wavetek then converts this input voltage into an output frequency of the form  $f = f_0 + c \cdot V_1$  which then corresponds to an output voltage of  $V = V_0 \sin(2\pi ft + \phi_1)$ . In this case,  $f_0$  is the carrier frequency,  $c$  is the voltage to frequency conversion coefficient of the Wavetek,  $V_0$  is the Wavetek (RF) output voltage amplitude, and  $\phi_1$  is a phase factor [7]. It is important to note that  $f$  is a function of time, since it depends on  $V_1$ :

$$f = f_0 + c \cdot V_{mod} \sin(2\pi f_{mod}t + \phi_0). \quad (16)$$

The intensity of  $D_2$  light measured by the photodiode,  $I_s$ , is a function of the frequency of the Wavetek.  $I_s$  is defined as  $I(f) + I_0$  where  $I(f)$  is the intensity of  $D_2$  as a function of frequency and  $I_0$  encompasses all background noise. The strength of the  $D_2$  signal is sent to the lock-in amplifier, which then gives the following output:

$$\bar{x} \propto \frac{\int_0^{\Delta t} [I_s \sin(2\pi f_m t + \phi_0)] dt}{\Delta t}, \quad (17)$$

where  $\Delta t$  is the time constant of the lock-in. It can be shown that  $\int \sin(2\pi f_m t + \phi_0) dt = \frac{\cos(2\pi f_m t + \phi_0)}{2\pi f_m}$ , so the equation for the lock-in output becomes:

$$\bar{x} \propto \frac{-1}{\Delta t} \int_0^{\Delta t} [I(f) + I_0] \frac{d(\cos(2\pi f_m t + \phi_0))}{2\pi f_m} \quad (18)$$

After integrating by parts, the new equation for  $\bar{x}$  becomes:

$$\bar{x} \propto \frac{-1}{2\pi f_m \Delta t} [I(f) + I_0] \cos(2\pi f_m t + \phi_0) \Big|_0^{\Delta t} + \int_0^{\Delta t} \frac{\cos(2\pi f_m t + \phi_0)}{2\pi f_m \Delta t} dI(f) \quad (19)$$

Because the first term is evaluated over the cosine function's period,  $\Delta t$ , it vanishes.

$$\bar{x} \propto \frac{1}{2\pi f_m \Delta t} \int_0^{\Delta t} \cos(2\pi f_m t + \phi_0) \frac{dI}{df} \frac{df}{dt} dt. \quad (20)$$

From the previous equation for  $f$ , we can calculate  $\frac{df}{dt}$  and substitute it into this equation,

$$\bar{x} \propto \frac{c \cdot V_m}{\Delta t} \frac{dI}{df} \int_0^{\Delta t} \cos(2\pi f_m t + \phi_0) \cos(2\pi f_m t + \phi_1) dt, \quad (21)$$

$$\bar{x} \propto c \cdot V_m \cos(\phi_0 - \phi_1) \frac{dI}{df}. \quad (22)$$

Hence, the signal measured by the lock-in amplifier is proportional to the modulation amplitude, the phase change, and the derivative of the lineshape of EPR resonance [7]. A frequency modulation measurement used to find resonance is shown in Figure 6. Resonance is marked by the point near the middle where the lock-in signal is zero.

Once resonance is found, it is necessary to use a PI feedback box which is built to lock on to the particular resonance frequency (see Figure 8). The goal of the PI box is to read the photodiode derivative from the lock-in. If a non-zero signal appears due to a shift in the resonance frequency, the PI Box will apply the correct voltage to the Wavetek VCO to adjust the central frequency. In this way, the system remains at resonance, even when conditions in the system change.

The PI box is given a voltage corresponding to the EPR frequency, which it uses to adjust the output of the Wavetek to keep the lock-in output signal locked on resonance. The box splits the input signal from the lock-in,  $V_{IN}$ , into two, and provides one output which is proportional to  $V_{IN}$ , and another which is proportional to the integral of  $V_{IN}$ . Looking at the diagram of the PI box in Figure 8, point 1 provides a signal which is equal to  $G_1 \times V_{IN}$ , while point 2 provides a signal of  $G_2 \int V_{IN}$ . These two signals are then added together with a modulation signal,  $V_1$ , which comes from the modulation function generator [8]. Therefore the final output signal,  $V_{out}$  equals the sum of the proportional signal from the circuit, the

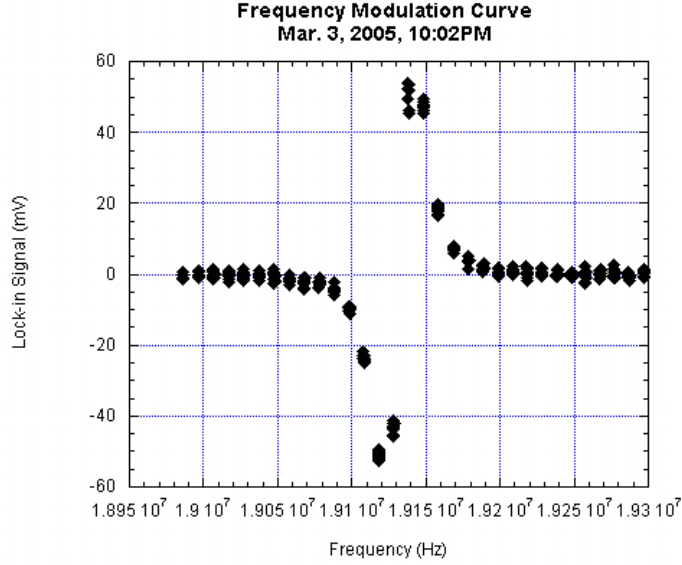


Figure 7: A graph of Frequency Modulation used to measure the derivative of the EPR resonance lineshape. Resonance occurs when the signal from the lock-in passes through zero near the middle of the graph.

integral signal, and the modulation.

$$V_{out} = V_1 + G_3(G_1 V_{IN} + G_2 \int V_{IN}) \quad (23)$$

### 3.3 Results

The lab at the College of William and Mary gathered EPR polarimetry data on three cells in the past year. A brief write up of AFP sweep operating procedure can be found in Appendix 1. Figure 9 demonstrates various results achieved using the EPR\_AFPSweep2004.vi program. The noise in the graphs was reduced by adjusting the absolute gain in the PI feedback box, while the relative gain helped to control the slope of the drop in the graph. The slope is also controlled by the amplitude of the Wavetek and modulation function generator.

To measure polarization, Equation 15 is used:

$$P_p = \frac{3\Delta\nu_{EPR}}{4\mu_0 \frac{d\nu_{EPR}}{dB} \kappa \mu_H \epsilon \eta_p}. \quad (24)$$



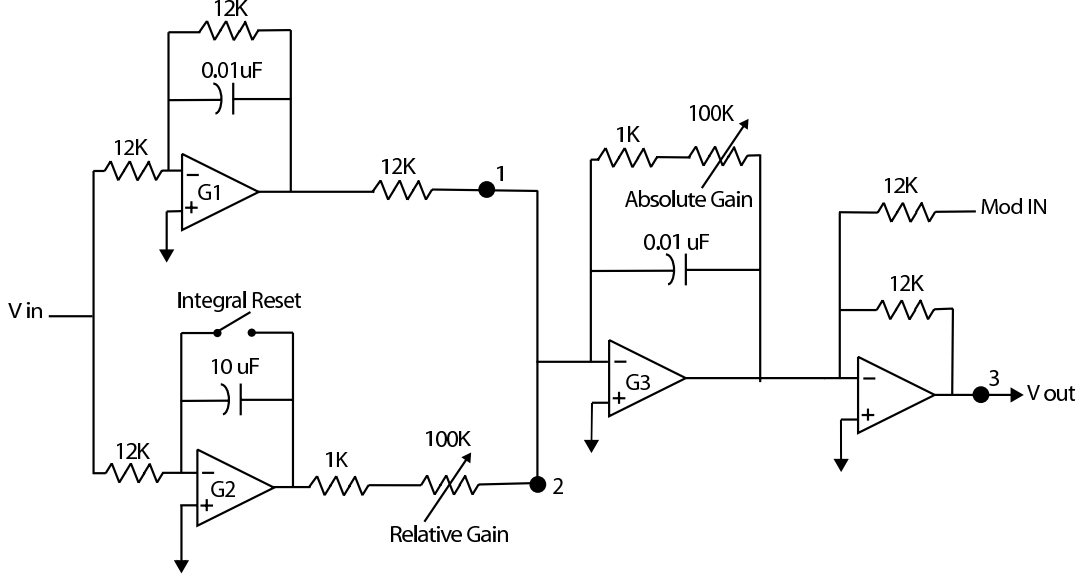


Figure 8: A diagram for the PI feedback box. The upper section produces a signal which is proportional to the output from the lock-in amplifier. The lower section gives a signal proportional to the integral of the lock-in output. Together, these two branches are added together and then an additional modulation is added in to modulate the Wavetek. The box is intended to lock the EPR coil on the resonance frequency of the system [8].

In this equation,  $\mu_0 = 4\pi \times 10^7 \text{ N/A}^2$ ,  $\frac{d\nu_{EPR}}{dB}$  is approximately  $4.67 \times 10^3 \text{ MHz/T}$  [5], and  $\mu_{He} = 1.07 \times 10^{-26} \text{ J/T}$ .  $\kappa = 4.52 + .00934T_p$ , where  $T_p$  is the temperature of the pumping chamber measured in degrees celsius [5]. The cell's geometry and gas properties come into the equation for  $\eta_p$ , the density of the  $^3\text{He}$  in the pumping chamber.

$$\eta_p = \frac{\eta_0}{1 + \frac{V_p}{V_T} \left( \frac{T_p}{T_t} - 1 \right)}. \quad (25)$$

In this equation,  $\eta_0$  is the cell density measured in amagats,  $T_p$  and  $T_t$  are the temperatures of the pumping and target chambers respectively, and  $\frac{V_p}{V_T}$  is the ratio of the volume of the pumping chamber to that of the entire cell. With the cells used at William and Mary, it is safe to approximate this ratio as 1/2, therefore the equation for  $\eta_p$  becomes:

$$\eta_p = \frac{2\eta_0}{1 + \frac{T_p}{T_t}}. \quad (26)$$

When all the constants are plugged in to the polarization equation, and units are converted such that the final answer is unit-less (since polarization is measured in a percentage),

the following equation remains:

$$P_p = 222.01 \frac{\Delta\nu_{EPR} \left(1 + \frac{T_p(K)}{T_t(K)}\right)}{\eta_0 (4.52 + .00934 T_p(^{\circ}C))}. \quad (27)$$

In order to use this equation,  $\Delta\nu_{EPR}$  must be measured in MHz, and  $\eta_0$  should be in amagats. Under these conditions, the polarization of the pumping chamber should be a unitless fraction corresponding to a percentage of polarization.

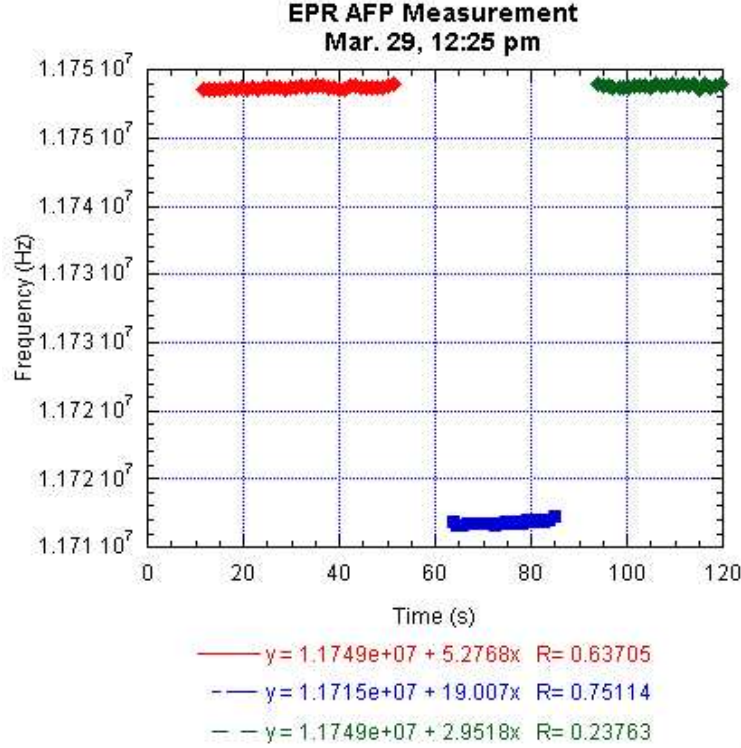


Figure 9: The difference in frequency is proportional to the magnitude of the polarization of the  $^3\text{He}$  in the cell. For this cell, at 12:25 pm on March 29, 2005,  $\Delta\nu_{EPR} = .0328$  MHz,  $T_p = 236^{\circ}\text{C}$ ,  $T_t = 61^{\circ}\text{C}$ , and  $\eta_0 = 8.597$  amagats. These values made the percentage of polarization of  $^3\text{He}$  in the pumping chamber 31.8%.

While three target cells were tested this year, only one was tested with working temperature sensors (which enables an exact measurement of polarization). The EPR signal measured from this cell is shown in Figure 8. The calculated shift in EPR frequency was 0.03289 MHz. The cell density,  $\eta_0$ , was 8.597 amagats, and at the time of the measurement, the temperature of the pumping and target chambers were  $236^{\circ}\text{C}$  and  $61^{\circ}\text{C}$  respectively. Therefore, this cell reached a maximum polarization of 31.8%

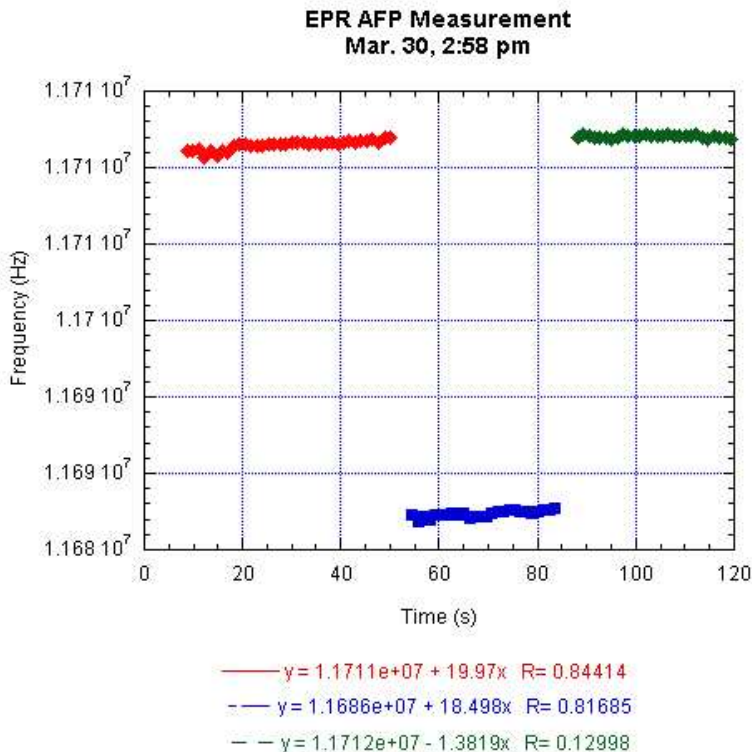


Figure 10: Another measurement made on the cell shown in Figure 9. At this time, at 2:58pm on March 30, some of the  $^3\text{He}$  had depolarized, making  $\Delta\nu_{EPR} = 0.0247$  MHz. This change in frequency shift altered the percentage of polarization of  $^3\text{He}$  in the pumping chamber to 23.9%.

Polarization data was also attained from the other two cells even though the exact temperature of the cell was not known. As will be shown in Table 1, the uncertainty in  $P_p$  due to the pumping and target chamber temperatures was minimal.

It is important to note that one of the cells tested was a hybrid cell. Hybrid cells use potassium in addition to rubidium to obtain higher levels of polarization levels [3]. Because there is less rubidium in these cells, it was thought that performing EPR measurements would be extremely difficult, if not impossible. However, it was proven that EPR can be done on hybrid cells as shown in Figure 11 using the much higher frequency (20 MHz) of potassium EPR.

In order to measure the temperature of the cell in both the target and pumping chambers, 2 RTD temperature sensors were attached to the cell. Every time an EPR measurement was

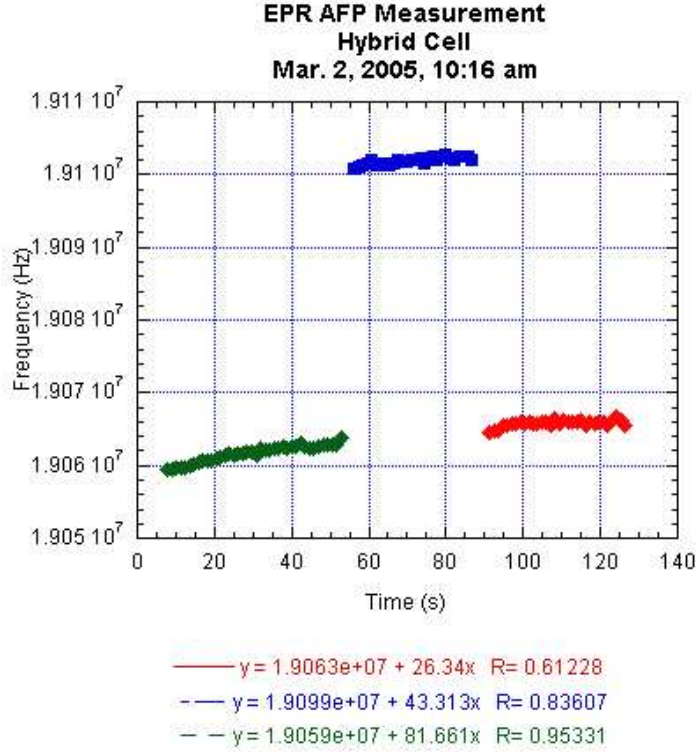


Figure 11: EPR measurement of a hybrid cell, which contains potassium as well as rubidium. This cell reached a polarization of 34.53 %.

taken, the temperature of the cell was recorded. Unfortunately, while the target chamber RTD provided stable measurements ( $\pm 2^\circ C$ ), the pumping chamber RTD fluctuated greatly ( $\pm 10^\circ C$ ). To combat this problem with uncertainty in temperature, a Variac power source was used to stabilize the heater in the oven system. However, even with this stabilizing source, it was still challenging to keep the temperature constant.

The main source of uncertainty in polarization comes from the fact that  $\nu_{EPR}$  is not constant. Typically, the frequency would increase as time went on (this can be seen in the positive slopes of all the curve fits in Figure 9). This slope could be caused if the polarization of  $^3\text{He}$  is not saturated, or if the external holding field,  $B_0$  is not stable. This uncertainty in frequency led to uncertainty when calculating  $\Delta\nu_{EPR}$ , and contributed the largest amount of uncertainty in the measurements of  $P_p$ , as seen in Table 1

The PI feedback box also seemed to have caused problems with some of the EPR measurements that were taken. In some measurements, the resonant frequency would not change

Variable	Value	Uncertainty	Absolute Uncertainty in $P_p$
$\Delta\nu_{EPR}$	0.0329	0.5 kHz	0.0048
$T_t$	334 K	2 K	0.0011
$T_p$	509 K	10 K	0.0006
$\eta_0$	8.597 amg	0.43 amg	0.0155
Total Uncertainty			0.0163

Table 1: Uncertainty Table for Polarization Measurement. This table shows that the polarization for this cell can be known to  $\pm 0.0163$ , or in other words, with a 5 % relative error.

automatically, rather it would gradually drop down to the new value (see Figure 12). The only parameter that affected the slope of this line was the relative gain of the PI box. However, because the box was not specifically designed for the system at William and Mary, the relative gain did not go as high as it needed to. Therefore attempting to increase the slope of the graph was not possible.

## 4 Conclusions

To date, an electron paramagnetic resonance system has been successfully built and tested at the College of William and Mary. Data shows a distinct shift in the EPR frequency, corresponding to the polarization of the target cell. The theory behind the EPR system has also been extensively studied in order to understand what information is needed to analyze results. Measurements of specific cell parameters have been made so that an exact value for cell polarization can be calculated. It was concluded that the last cell tested at William and Mary reached polarization levels of around  $(31 \pm 1.63\%)$ .

There is opportunity for much improvement on the system. Because the dramatic fluctuations in temperature contribute to the large uncertainty in  $\eta_0$ , improvements in the temperature sensor system need to be made. It is not determined why the RTDs measure a different temperature than the oven controller or why both temperatures have such large fluctuations.

In addition, because of the troubles had when measurements were taken at some times,

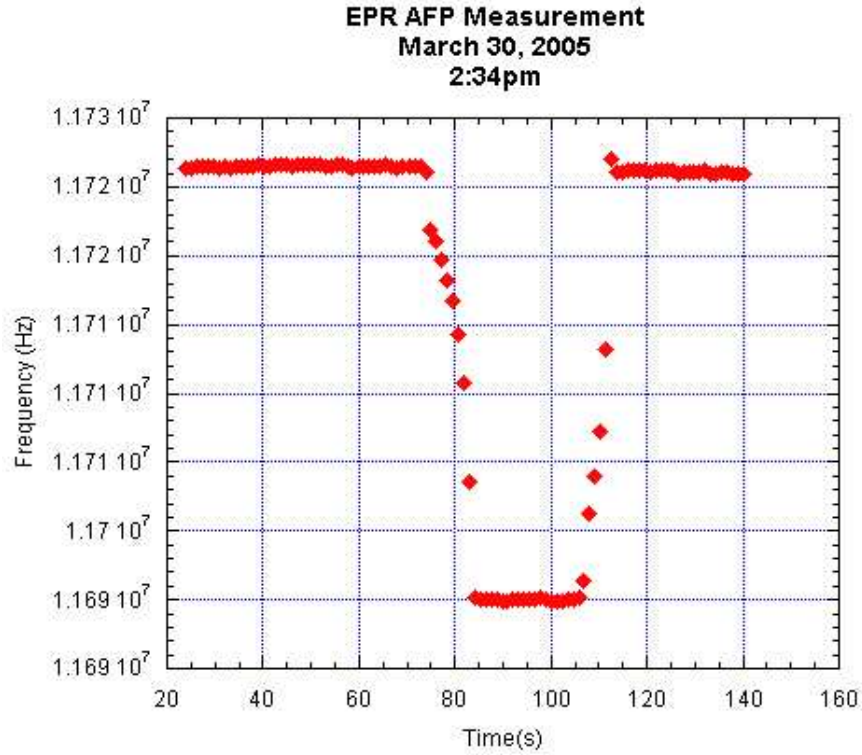


Figure 12: Because of glitches in the PI box, sometimes the resonant frequency did not fall straight to its new value after the spins flipped. Instead, it would gradually drop down to the correct value. Because the PI box was not designed specifically for the system at William and Mary, it seems that the time constant of the circuit was just too large.

it seems that the PI Box should be redesigned in order to optimize the setup at William and Mary. The setup used currently was originally designed by Schaefer with parameters optimized for their system [8]. The reasons for all the settings used need to be re-examined.

## 5 Appendix

### EPR Measurement

#### Device Settings:

##### *Lock-in Amplifier*

AC Gain	50dB
Input Limit	10 mV
Sensitivity	1 mV
Time Constant	100 ms
DR	16
OSC	0 Hz

##### *PI Box:*

Attenuator set to "In" Position

Integrator "Off."

##### *Wavetek:*

Sine Wave

VCO Mode

Frequency approximately 11 MHz

Amplitude around 5 V.

##### *EPR Modulation Function Generator:*

Triangle Wave

Frequency = 200 Hz

Amplitude = 1 V<sub>pp</sub>

Output Impedance = High Z

Offset = 0 V

### Finding Resonance

- With integrator off, adjust the Wavetek frequency by hand in steps of 10 KHz. Resonance can usually be found around 11.5 MHz for a Rb cell and 19.3 for a hybrid cell, and it is

signified by a jump in the signal read by the lock-in amplifier. Note that although the Wavetek may read one value for the frequency, the counter may be measuring a slightly different value.

- Once resonance is found, the value of the peaks near resonance need to be around  $\pm 30$  mV as measured by the lock-in. This step is so that the PI Box will not reach saturation. In order to change this signal, adjust the Wavetek and/or the EPR Modulation FG amplitude.
- Move signal to peak and autophase on lock-in so the majority of the data is in the X-channel. -When the signal is down to a reasonable height, adjust the Wavetek frequency to 10-20 kHz. now it is time to adjust the relative gain knob in order to control the speed at which the signal goes to zero when the integrator switch is turned on. The goal is to get the signal to go to zero as fast as possible (usually within 3 steps).
- Put the integrator in the "On" position.
- Run the EPR\_AFPSweep2004.vi program. Hit the "download parameters" button, and then put the switch to the "run" position. Wait thirty seconds to get a baseline measurement, and then hit "Start Sweep."
- Make sure you let the program run until the "Start Sweep" button reads "Waiting for sweep signal." This ensures that the spins are flipped back to their original position.
- If the signal is noisy, adjust the absolute gain knob.



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