ELECTRON PARAMAGNETIC RESONANCE

Phys 1560 & 2010 – Brown University – November 2010

Purpose

- Use microwaves to induce and detect electron paramagnetic resonance
- Become familiar with a waveguide spectrometer and phase-sensitive detection
- Measure the gyromagnetic ratio and transverse relaxation time of the nearly-free electrons in DPPH

Theory

An atom has a net magnetic moment $\boldsymbol{\mu}$ due to the spin and orbital magnetic moments of unpaired electrons, with the exception that electrons in $^1S_0$ state do not contribute to a net magnetic moment. When this net magnetic moment is in an external magnetic field $\boldsymbol{H}$, the orientational potential energy is

$$E(H) - E(0) = -\boldsymbol{\mu} \cdot \boldsymbol{H} = -\mu_H H$$

where μ_H is the component of $\boldsymbol{\mu}$ along $\boldsymbol{H}$. In a magnetic field each orbital energy level will be split into several energy levels corresponding to the several possible values of μ_H.

For simplicity we will consider the case of an atom with a single unpaired electron and zero orbital angular momentum. In a magnetic field the spin has two possible energy states given by

$$E = \pm\frac{1}{2} g\mu_B H.$$

The split in energy levels due to the magnetic field is called the Zeeman interaction. The Landé g factor for a free electron is approximately g =2. 0023. The Bohr magneton is $\mu_B = \frac{e\hbar}{2m_e c}$ in cgs units, or $\mu_B = \frac{e\hbar}{2m_e} = 9.\ 27 \times 10^{-24} J/T$ (in SI units). If the electron absorbs a photon of energy $E = h\nu_R$ (e.g. from a microwave field,) where

$$h\nu_R \equiv g\mu_B H \quad (1)$$

then the electron will transition from the lower energy state to the higher energy state. The frequency ν_R at which absorption occurs is known as the resonant frequency due to similarities the system shares with classical resonant systems. The magnetic field used in this experiment dictates that ν_R fall in the microwave portion of the spectrum. The microwave power absorbed when the electrons transition to a higher energy state can be detected and measured with the bridge circuit to quantify the absorption of photons from the electromagnetic field.

We will use the unpaired electrons in the free radical diphenyl-picryl-hydrazil (DPPH) — $(C_6H_5)_2N - NC_6H_2(NO_2)_3$. The molecular structure of DPPH is shown in Figure 1. This organic salt has one free spin per molecule; the free electron behavior arises in one of the nitrogen atoms.

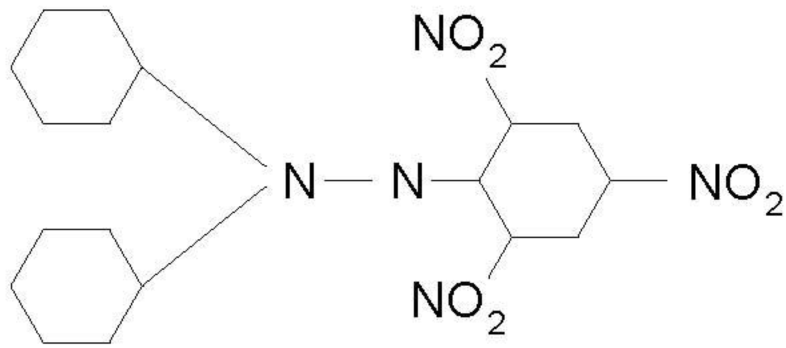


Figure 1: Molecular structure of DPPH. Unmarked sites are assumed to be Carbon.

For DPPH (as in most cases), the distribution of electrons in the two energy states is given by Maxwell-Boltzmann statistics. Let N_1 be the number of electrons in the lower energy state, and N_2 the number of electrons in the upper energy state. If ΔE is the difference in energy between the two states, then $N_2/N_1 = \exp -\Delta E/k_B T$, where k_B is Boltzmann's constant and T the temperature. Greater sensitivity is achieved for $N_2 \ll N_1$, which means working at a high frequencies and correspondingly large $g\mu_B H$, or by working at low temperatures.

For the resonant absorption to continue there must be some way for the electrons in the higher energy state to lose energy and drop back to the lower state. If this were not so, N_1 and N_2 would equalize and no further net absorption would occur - a state called saturation. Electrons can exchange energy with their surroundings through what are known as relaxation processes, which are described in terms of a relaxation time. The longitudinal or spin-lattice relaxation time T_1 is a measure of how rapidly an out-of-equilibrium spin distribution returns to thermal equilibrium. If the spins absorb energy from the microwave field at a rate slow compared to the rate at which they can exchange energy with their surroundings, saturation does not occur.

Realistically, the absorption of photons occurs over a narrow range of frequencies. The width δv of this distribution can have a number of causes:

- Slight variations of the magnetic field from spin to spin due to inhomogeneity of the applied field or due to internal fields in the sample ($h\delta v = g\mu_B \delta H$).
- Variation of the effective g value from site to site ($h\delta v = \delta g \mu_B H$).
- If the spin lattice relaxation time is sufficiently short, δv may be affected by energy uncertainty from the finite lifetime in a state ($h\delta v = \delta E = h/T_1$).

The linewidth δv is inversely proportional to what is called the spin-spin or transverse relaxation time, T_2. The line-shape function $f(v)$ represents the shape of the absorption line; $f(v)$ as a maximum at $\nu = \nu_0$ and can usually be approximated by a Lorentzian distribution (Pake, 1962). A Lorentzian has the form

$$f(v) = \frac{1}{1 + \left(2\pi T_2 (v - v_0)\right)^2}$$

The full width at half maximum (FWHM) for a Lorentzian is $\delta v_{1/2} = 2/(2\pi)T_2$.

Apparatus

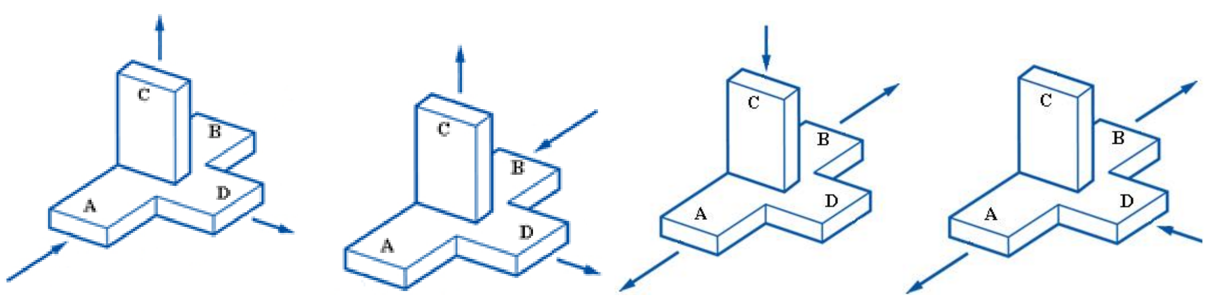
Main Magnet

A large electromagnet is needed to generate a magnetic field constant over the entire sample to break the degeneracy of the spin states and align the electron's spins. Due to the sensitivity required to get accurate measurements in this experiment, the power supply for the main magnet is extremely stable and demonstrates almost no measurable fluctuations at a given setting. The currents necessary to power the magnet could be achieved using a smaller, less expensive power supply, but these would not provide the required stability.

Waveguides

In this experiment microwaves will be used to measure the energy absorbed by electrons during transitions between spin states. Microwave power is generally expressed decibel-milliwatts (dBm, dBmW). Measurements in decibels (dB) express the base ten logarithm of a ratio between two quantities. Measurements of power in dBm $\equiv \log_{10}(P/1\text{mW})$, where P is the power in mW. As an example 0 dBm corresponds to a power of 1 mW.

The microwaves used in the experiment will be directed and controlled using rectangular waveguides. The waveguides you will use are hollow metal tubes whose conducting walls create a boundary condition dictating that the transverse portion of the wave's electric field drops to zero at the walls. Applying this boundary condition to Maxwell's equations shows that the waveguide will only allow propagation of waves for which the dimensions of the guide are half-integer multiples of the wavelength. The waveguide's mode is thus uniquely specified by two integers M and N. M refers to the number of field maxima (number of half-wavelengths) along the narrow side of the waveguide, and N to the number of field maxima along the broad side. For the X–band (8 to 12 GHz) waveguides used in this experiment, N=1, and M=0, i.e. in the TE_{10} mode, where TE stands for transverse electric, meaning the electric field has no component in the direction of wave propagation ($\vec{E} \cdot \vec{k} = 0.$) In contrast, since a coaxial cable has a central conductor as well as an outer conducting surface, it can support modes in which both the electric and magnetic fields lie in the transverse plane.



MW into A MW into B MW into C MW into D

Figure 2A: The Magic T. - Behavior of microwaves entering each of the four arms

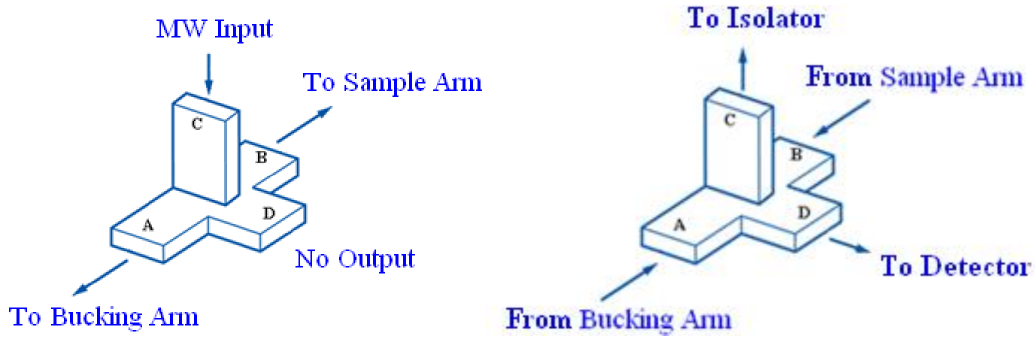


Figure 3B: The Magic T. - How the T in this experiment before and after absorption

Magic-T

A waveguide spectrometer (bridge) will be used to study the properties of the sample. Microwaves travel through a coaxial cable from the microwave generator to the bridge, where they are transmitted into the waveguide. The microwaves first pass through an isolator that allows the microwaves to travel forward into the wave guide system but not back into the cable. Next, the microwaves enter the heart of the bridge - the magic-T.

The magic T and its function are pictured above in figure 2. Microwaves entering the T through either port D or C are divided into two equal parts and exit via ports A and B, while microwaves entering through either A or B are divided equally between C and D. This behavior is not inherent to the geometry of the T, but is generated by small impedance matching devices placed inside the T. However, due to the relative orientation of the arms, waves incident on arm D (sum or Σ arm) will lead to the waves in A and B being in phase, whereas waves incident on C (difference or Δ arm) will undergo a 180 degree phase shift between waves in arms A and B. In the second scenario, the waves undergo destructive interference, allowing one to directly observe the difference in returning signals from the two arms; you will exploit this property of the T in this experiment to measure the power loss due to resonant absorption of microwave photons. In the apparatus you will use, you have the added luxury of the adjustable short in the reference arm, allowing you to tune the interference of the waves precisely.

For more information on the workings of a magic T, see the Wikipedia page at http://en.wikipedia.org/wiki/Magic_tee, or for an animated representation, see http://www.feko.info/knowledge-base/application-notes/waveguide-magic-tee/modelling-of-a-magic-t-waveguide-coupler and click on the “Animate” links under the pictures. Pay close attention to the “Magic” section of the Wikipedia article and the animated vector representations on the other page for an understanding of how the polarity of the waves interacts with the changing dimensions of the T. The “Magic” section also provides an explanation of the importance of the T’s internal matching structure.

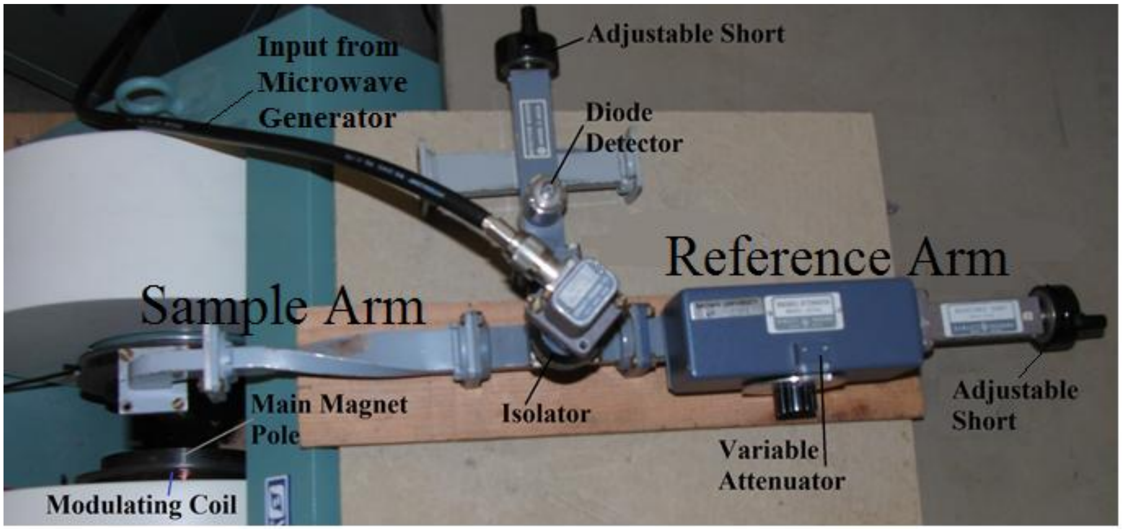


Figure 4: The Spectrometer

Waveguide Spectrometer (Bridge)

By adjusting microwave components in the reference arm we can vary the magnitude and phase of signal at the detector arm. We use an attenuator to vary the amplitude and an adjustable short to vary the phase in the detector arm. To detect the signal, a crystal detector (a diode, see Ginzton Sections 2. 1-2. 3) is placed in the waveguide at a position of maximum electric field along the length of the waveguide. The diode detector rectifies the microwaves producing a signal that can be displayed on an oscilloscope or fed into an amplifier. The diode is at a fixed position inside the waveguide, adjust the short at the end of the waveguide to an odd multiple of $\lambda_g/4$ from the diode, where λ_g is the guide wavelength.

The microwave spectrometer is illustrated in Figures 3 and 4. You should familiarize yourself with the different components by reading manuals, etc. The preliminary work will be done with the oscilloscope. The key to understanding much of how the spectrometer works is to understand how the magic T functions. Remember that the power coming down the waveguide is split into two, with half going down two separate arms – the sample arm and the reference (sometimes called the bucking) arm. Whatever is reflected from those two arms adds to go to the fourth arm. The observed output from the detector diode will be a DC signal on the order of a few hundred millivolts, whereas the absorption signal will only cause a microvolt-order change in the signal. As such, in order to see the absorption you will have to make the waves from the reference arm destructively interfere with the waves from the sample arm so that only the difference in the two waveforms caused by the absorption will be picked up by the detector. Devices with this function are referred to as bridges in conventional circuitry.

When two sinusoidal signals of the same frequency combine, one must not only consider magnitude, but relative phase as well. Let the electric field incident on the detector coming from the reference arm be

$$E_r(t) = E_{r,0} \cos(\omega t + \phi).$$

The magnitude $E_{r,0}$ and the phase ϕ with respect to the wave coming from the sample arm can be adjusted separately. Take the field from the sample arm at the detector to be

$$E_s(t) = E_{s,0} \cos \omega t + E_{s,0}(a \cos \omega t + b \sin \omega t).$$

The second term in the right side represents the effect of the wave due to the absorption (term containing $a \cos \omega t$) and dispersion (term containing $b \sin \omega t$) of the spins near resonance. The coefficients "a" and "b" are functions of magnetic field. The output voltage of the detector is generally proportional to the microwave power incident on it (a square law detector).

$$V_D \sim \langle |E_s(t) + E_r(t)|^2 \rangle_{ave.}$$

Suppose the reference arm is adjusted so that E_r and E_s are 180^o out of phase, i. e. $\phi = 180^O$, and their difference in magnitude are written as $E_{s,0} - E_{r,0} = \Delta E$. Then

$$E_s(t) + E_r(t) = (\Delta E + E_{s,0}a) \cos \omega t + E_{s,0} b sin\omega t.$$

Since a and b are small quantities, that is a, $b \ll \Delta E/E_{s,0}$, only first order terms in these parameters are included in calculating the detector response.

$$V_D \sim (\Delta E)^2 \left(1 + 2\frac{E_{s,0}}{\Delta E}\right)$$

Hence in this particular bridge balance configuration the output is sensitive to the absorption of the spins but not the dispersion.

On the other hand, the reference arm can be adjusted so that $E_{r,0} = E_{s,0}$ and $\phi = 180 - \Delta\phi$. With $\Delta\phi$ small but $\sin\Delta\phi \approx \Delta\phi$ still large compared to a and b, then

$$E_s(t) + E_r(t) = E_{s,0}(\Delta\phi + b) \sin \omega t + Es_{,0}a \cos \omega t.$$

In this case the detector output is proportional to

$$V_D \sim (E_{s,0}\Delta\phi)^2 \left(1 + 2\frac{b}{\Delta\phi}\right)$$

and is a measure of the dispersion rather than the absorption.

Lock-in Amplification

In this experiment, the absorption signal that occurs at resonance is very small, making it difficult to distinguish from background noise. To overcome this difficulty, you will employ techniques of phase-sensitive detection, otherwise known as lock-in amplification. A lock-in amplifier is basically a voltmeter that employs sophisticated filtering techniques to pick out a weak signal from noise. It uses an internally or externally produced reference frequency at a particular phase which is run through the experiment and becomes a portion of the experiment's output, which we'll call the return signal. In addition to the desired signal, which has been modulated at the reference frequency, the return signal will include noise distributed across all Fourier modes. The return signal is fed back into the lock-in, which isolates the desired signal from this noise.

The critical stage of the lock-in amplification process involves combining the reference and return signals such that the combined waveform is given by the product of the two initial waveforms. If the reference and return signals are given by

$$V_{ref} = V_r \sin(\omega_r t + \theta_r)$$

$$V_{sig} = V_s \sin(\omega_s t + \theta_s),$$

Then the combined signal is given by

$$V_t = V_r V_s \sin(\omega_r t + \theta_r) \sin(\omega_s t + \theta_s)$$

$$V_t = \tfrac{1}{2} V_r V_s \{\cos[(\omega_r - \omega_s)\, t + \theta_s - \theta_r] - \cos[(\omega_r + \omega_s)\, t + \theta_s + \theta_r]\}$$

The combined signal is sent through a low-pass filter with a bandwidth $\Delta f = \frac{1}{4T}$, where T is the time constant of the lock-in. As such, only portions with little time dependence, i.e. only portions with $\omega_r - \Delta f < \omega_s < \omega_r + \Delta f$, will appear in the final measurement. In the ideal case of infinite T, only the portion of the signal with $\omega_s = \omega_r$ is retained (no time dependence at all,) and the measured voltage will be a DC signal given by

$$V_{measured} = \tfrac{1}{2} V_r V_s \cos(\theta_s - \theta_r)$$

The magnitude of this DC signal is proportional to the amplitude of the signal which has been modulated at the reference frequency. The time constant of the lock-in used in this experiment can go as high as 100 seconds, giving the low pass filter a bandwidth of 0.0025 Hz. However, increasing the time constant comes at the cost of losing sensitivity to rapid changes in the signal.

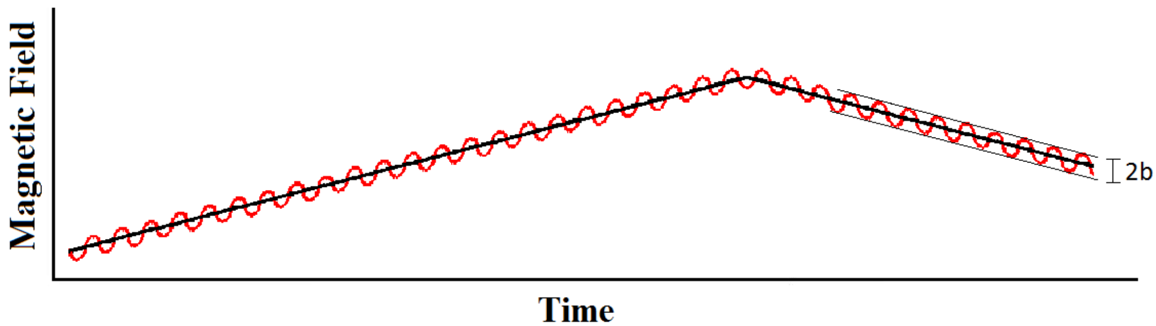


Figure 4: Effect of modulation on magnetic field strength: The black line represents the field of the main magnet as it is ramped with a triangle wave. The red curve represents the combined field strength of the main magnet and the modulation coils. Note: Not drawn to scale.

Remember, the modulating coils only provide a small alternating component of the magnetic field. Figure 4 depicts how the modulating coils affect the overall magnetic field. The function generator connected to the main magnets produces the extremely low-frequency wave (triangle or sawtooth) that ramps the magnetic field through a range including the value required for resonance. However, since $\omega_{ref} \gg \omega_{ramp}$ and $B_{mod} \ll B_{main}$ we can treat the total magnetic field as a DC signal which varies slowly with time plus a small oscillating perturbation. Since the return signal depends on the total magnetic field, we write the amplitude *A* of the return signal as

$$A = A(B + \Delta B)$$

Where *B* is the field strength of the main magnets and $\Delta B = bcos(\omega_r t)$ is the perturbation provided by the modulating coils. If we Taylor expand *A* around *B*, we find

$$A(B + \Delta B) = A(B) + \frac{dA}{dB}\Delta B + \frac{1}{2}\frac{d^2A}{dB^2}(\Delta B)^2 + \cdots$$

$$A(B + \Delta B) = A(B) + \frac{dA}{dB} b\cos(\omega_r t) + \frac{1}{2}\frac{d^2A}{dB^2}(1 + \cos(2\omega_r t)) + \cdots$$

We can apply the reasoning used above to demonstrate that only the part of the signal which is periodic with frequency ω_r emerges in the final measurements ($\cos(\omega_r t) = \sin\left(\omega_r t + {}^{\pi}/_{2}\right)$.) As such, the signal you will observe on the oscilloscope is the derivative of the absorption resonance with respect to the (time-varying) magnetic field and not the resonance signal itself.

See figure 5 for a conceptual picture of why the lock-in yields the derivative of the absorption signal. As you sweep the main magnet through resonance, the modulating coils move the total value of the magnetic field back and forth with an amplitude *b* as described above ($\Delta B = bcos(\omega_r t)$.) As the total magnetic field oscillates, a portion of the output signal $A(B + \Delta B)$ oscillates at the same frequency with amplitude determined by the derivative of the absorption signal at that value of the magnetic field. In essence, the modulation makes the output signal *A* slide up and down the absorption signal—the Lorentzian in figure 5—at the reference frequency, with an amplitude proportional to the local slope of the curve. This amplitude is denoted as ΔA in the figure and is given by the second term in the Taylor expansion. When the lock-in amplifier isolates the portion of the return signal at the reference frequency, the DC signal registered on the oscilloscope is the amplitude of that oscillation, which is proportional to the derivative of the absorption signal. The phase change in the return signal on opposite sides of the peak tells the lock-in that the slope of the absorption curve has become negative.

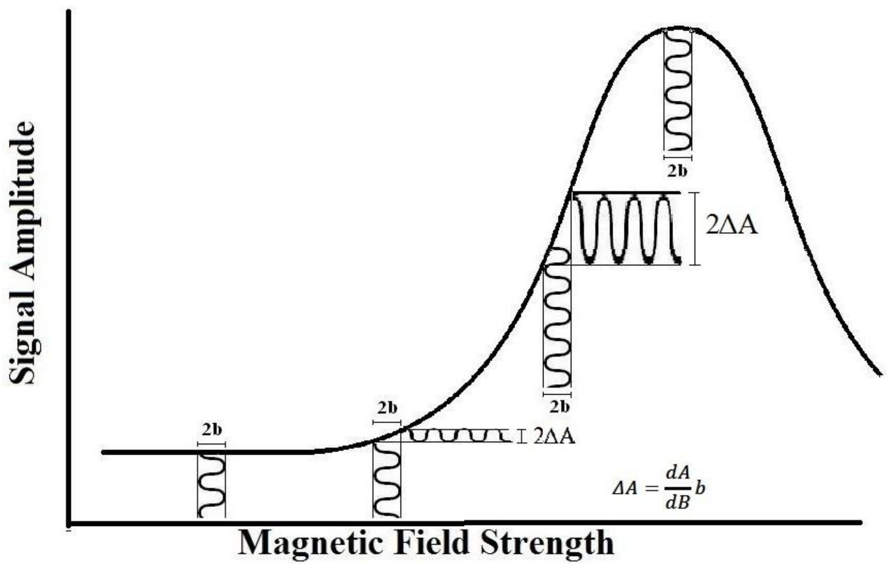


Figure 5: Physical explanation of why the lock-in gives the derivative of the absorption signal. The oscillations which appear to be moving upwards in the image represent the field modulation at constant amplitude, and those which appear to be moving rightwards represent the corresponding change in signal amplitude. As the magnetic field is modulated with constant amplitude at the reference frequency, there is a response in the output voltage at the same frequency whose amplitude is proportional to the slope of the absorption curve. Take note of how the response amplitude goes to zero on the far left and at the top of the absorption peak, i.e. where the derivative goes to zero. Amplitudes not drawn to scale.

For more information on lock-in amplification, see Appendix A of Preston & Dietz (p 367) and the pamphlet titled "About Lock-In Amplifiers" by Stanford Research Systems. The former should be in the reference library in the lab, and the latter should be with the manuals for the equipment used in this experiment, and can also be found on the course's wiki page at https://wiki.brown.edu/confluence/download/attachments/29406/About+LIAs.pdf?version=1.

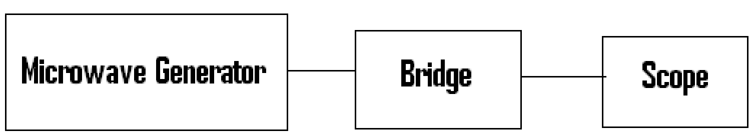


Figure 6: Initial Setup

Procedure **Note:** The magnet used in this experiment is water cooled. Be sure to follow all of the instructions regarding the water cooling system.

A. Set up and power up the apparatus as pictured in figure 6. The output of the microwave generator is already connected to the bridge. Connect the oscilloscope to the bridge detector. Set the variable attenuator to ~20 dB - see fig 3. Set the microwave output power to 10 dBm (see "Power" section), the central frequency "CF" to 9 GHz, the sweep width to 1GHz "Δf" (see Frequency Section) and the sweep time to 7.0 second (see "Frequency/Time" section.) Set the oscilloscope to observe the structure in the signal. The peaks and valleys are due to the movement of standing wave maxima as the frequency varies. Note the changes in the signal when you adjust the width of the sweep or the adjustable short on the detector arm. Why is the signal asymmetric? To better observe the signal, connect the "sweep output" (below "Data Entry") of the microwave generator to an unused channel on the scope and use it as the trigger.

B. Set the microwave frequency to 9 GHz continuous wave "CW" and the output power to 10 dBm. There are two adjustable shorts used in this experiment, one in the reference arm and one behind the detector. An adjustable short allows you to vary the length of the microwave cavity, changing the intensity profile of the standing wave which develops—there is an adjustable short sitting on the bench for you to look at. Vary the adjustable short behind the crystal detector so that you get the maximum signal; this amounts to moving the local maxima around until you place one right on the detector. Record voltage observed on the oscilloscope. Determine the output voltage of the detector versus the input microwave power so that you can relate the signal voltage to changes in power reflected in the sample arm. Plot the log of output voltage versus log of incident power from -5 dBm to 20 dBm and comment on the results. This will be used to calculate power loss from resonant absorption during the experiment.

C. Balance the bridge: Reset the microwave output power to 10 dBm. The sample sits in a small vial capped with black putty near the end of the sample arm. Make sure the sample is properly centered between the main magnets. Use the adjustable shorts in the detector arm to find the maximum signal from the detector. Use the adjustable short in the s Explore the use of the attenuators and and use them to cancel the signal from sample arm with that from the reference arm. This will null the voltage out of the bridge diode detector. Record the minimum voltage achieved at balance.

D. Procedure to operate the magnet:
   1. The magnet and power supply are water cooled. You should have been shown how to operate the water cooling system during the start up procedure. As a reminder, the cooling water is controlled by two valves - labeled *"EPR"* - located under the bench next to the NMR experiment and two switches that control the circulation pumps located on the wall above the bench. Both valves must be open and both switches must be on to operate the magnet. There are two additional valves - "*MAIN IN"*& "*MAIN OUT"* - that must also be open but these valves are usually open during the semester particularly if the evaporator is running.
   2. After turning off the magnet power supply, wait at least 15 min then turn off both circulation pumps (assuming no other equipment is using them) and both water valves labeled EPR. Except in an emergency, Do Not close the "*MAIN IN"*& "*MAIN OUT"* valves if the evaporator or other equipment is using cooling water.
   3. Turn on the power supply for the main magnet. A yellow light indicating that there is sufficient water flow should be on. The light is a little flaky, and may need to be gently tapped to come on. If the light does not turn on, turn off power and seek assistance.
   4. Turn on the D.C. power. Note: The digital display on the power supply displays the percentage of maximum current (50 amps).

5. The magnet supply can be controlled with an external voltage. This control voltage must be between -10V & 10V though you should not need anything like this in the experiment. The current in the magnet coils is changed by roughly 10% with the application of 1.0 V.

E. Calibrate the dependence of the magnetic field on voltage applied to the external input of the magnet power supply. Do not trust the voltage reading on the function generator, as it is only the machine's best guess of what the voltage should be were it connected with the correct impedance load. Measure with a multimeter or the oscilloscope instead in order to get an accurate calibration. You will need to use this data to calculate the exact resonant field strength.

F. Using a gauss meter (see TA for access and instruction,) plot magnetic field vs. current. Perform this exercise by taking measurements at ascending then descending amperages to explore the effects of hysteresis in current vs. B field.

G. Next, feed a series of triangle waves at varying frequencies from the function generator into the power supply. Plug the function generator output and the gaussmeter output into the oscilloscope and observe the two signals. Note how the magnet's impedance changes in magnitude and phase when you vary the frequency (for an inductor, $Z = R + i\omega L$ where L is the inductance).

H. Using the controls on the front of the magnet's power supply, slowly sweep the magnetic field through the value you calculated for the position of resonance. Due to the extremely narrow range of magnetic field associated with resonance and because the power supply's controls only provide crude control, it is very difficult – but possible - to observe resonance using this technique. Are you able to observe the small change in DC output from the detector that indicates resonance?

I. Given the difficulty inherent in the previous method, we will use a function generator to slowly scan the output of the magnet power supply. Set the function generator on triangle wave at a slow frequency and small P-P amplitude. Think about what this signal is controlling and select the frequency and amplitude accordingly. Connect the output to the variable resistor and then plug it into the front of the magnet power supply. Note the ground side of the BNC to banana adapter and connect it accordingly.

J. Not only does the method suggested in step "H" make it exceedingly difficult to hit the resonant field strength, but if the resonant field were found, the absorption signal would be difficult to distinguish from the noise. Since the signal is DC, 1/f noise is of particular concern. To overcome this difficulty, the field of the main magnet will not only be ramped, as described in the step "I", but will also be modulated with a reference signal. The reference signal is feed to the modulation coils – the smaller coils attached to the main magnet's pole caps. This process inserts a signature or reference frequency into the absorption signal and allows the lock-in amplifier to distinguish the signal from the noise.

K. The modulation has to be large enough to generate a signal the lock-in amplifier can detect. However, if the signal is too large, the audio amplifier will blowout. Set the "master volume" of the audio amplifier to just below 1/3 of its total range - Do Not turn it beyond 1/3 of its total range! Now, use the "Phono/Aux CD" knob to adjust the amplitude of the modulation. When adjusting the audio amplifier 's output to the modulation coils, always simultaneously monitor this output on scope. Immediately shut off the amplifier or reduce its gain if the waveform starts to distort or become nonlinear. If the output of the amplifier becomes distorted, the amplifier will blowout almost immediately

L. Set up the apparatus pictured in Figure 7. Connect the sine out on the back of the lock-in to the audio amplifier and the output of the audio amplifier to the modulation coils. Connect the reference out on the back of the lock-in to the reference input on the front of the lock-in. Connect the output of the lock-in to channel 1 on the oscilloscope, and the diode detector to one of the lock-in's inputs. Use a BNC T to Split the output of the function generator between the external input of the magnet power supply and the channel 2 input of the oscilloscope.

M. Use the tips included below to create the best possible absorption signal. See figure 8 for a picture of what you should be aiming for. Remember that the output will be the derivative of a Lorentzian, meaning it will rise, hit a maximum, fall below zero, hit a minimum, and return to zero. The curve should have equal areas above and below zero.

N. Once a good signal has been observed, hit the Run/Stop button on the scope to get a snapshot of the data. You can then either connect a USB drive to the scope or use the scope's connection to the computer and the OpenChoice Desktop program to collect the data. Make sure to capture the channel 2 input as well as channel 1. The output voltage of the function generator at the time when the absorption signal goes through zero can be used to determine the precise resonant value of the magnetic field strength. This will be necessary to calculate the electron's gyromagnetic ratio.

O. Use your acquired data and the equations in the Theory section of this manual to calculate the electron's gyromagnetic ratio and the transverse relaxation time of this system.

P. Again, after turning off the magnet power supply, wait at least 15 min then turn off both circulation pumps (assuming no other equipment is using them) and both water valves labeled EPR. Except in an emergency, Do Not close the "*MAIN IN"*& "*MAIN OUT"* valves if the evaporator or other equipment is using cooling water.

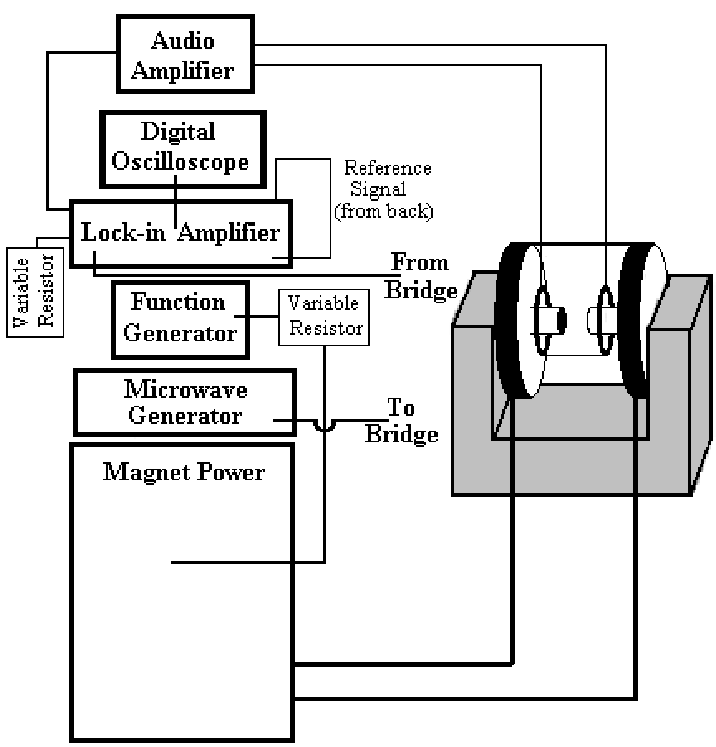


Figure 7: Setup used to observe resonant absorption

Tips for Getting a Good Signal

- The function generator is used to sweep the magnetic field with a triangle wave. A high amplitude wave spends more time away from resonance, a high frequency wave moves through resonance more quickly, a low frequency wave takes longer to sweep, plus what was learned in part G; what is a reasonable frequency and amplitude for this wave?
- In the section on lock-in amplification, remember the assumptions made in the Taylor expansion. How should the frequency of the ramp compare to the frequency of modulation?
- The "Time Constants" on the lock-in are inversely proportional to the bandwidth of the low-pass filter applied after the recombination of the reference and return signals ($\Delta f = \frac{1}{4T}$.) The setting of the "Time Constants" determines how much the signal is "smoothed out". Higher settings give greater noise reduction but decrease the amplifier's responsiveness.
- The "Sensitivity" setting on the lock-in determines the voltage range to be measured - just like on a multimeter. A sensitivity in the mV range will give less noise but requires a stronger input signal. A sensitivity in the nV range will measure a much weaker signal but will likely be noisier.
- The phase of the reference signal heavily influences the lock-in's output signal. Remember $V_{measured} = \frac{1}{2}V_r V_s \cos(\theta_s - \theta_r) = \frac{1}{2}V_r V_s [\cos(\theta_s)\cos(\theta_r) + \sin(\theta_s)\sin(\theta_r)]$, where θ_r is the phase of the reference signal. After attaining an absorption signal, adjust the phase controls to null the lock-in output. Next, shift the phase by ninety degrees. This will give a maximized signal; however, it may be inverted. How can an inverted signal be corrected?

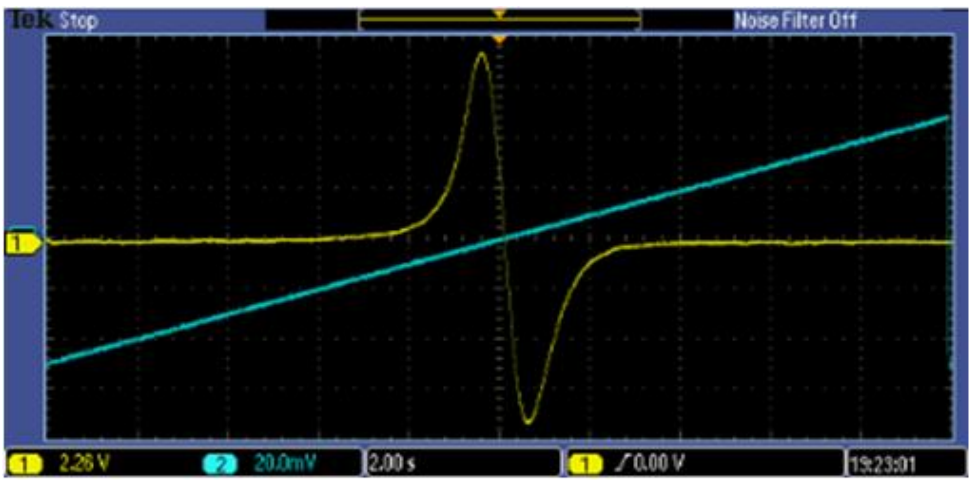


Figure 5: A very nice looking signal. Note the antisymmetry; the signal should ideally have equal weight and width above and below the horizontal axis. The blue line is the function generator sweeping the magnetic field through resonance.

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