NUCLEAR MAGNETIC RESONANCE AND MAGNETIC FIELD MEASUREMENTS

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Purpose

Become familiar with the principles and detection techniques of Nuclear Magnetic Resonance (NMR), examine the relationship between current and magnetic field in an electromagnet, and gain experience in the use of magnetic field measurement techniques.

Theory

NMR

Consult the appendices located in lab #203 at the NMR workstation, or the listed references located in the sciences library and Mr. Hudek's office, B&H, Room 218.

General detection technique

 The NMR spectrometer consists of a sample coil, tuning capacitors, a Robinson Oscillator, a frequency counter, a lock-in amplifier, an oscilloscope, a function generator, a 10 kG electromagnet, an audio amplifier, modulation coils, and associated power supplies. See Fig. 1 for a block diagram. The Robinson Oscillator is the heart of this spectrometer. The sample coil and tuning capacitors form a tank circuit, which sets the resonance frequency of the oscillator. A sample is placed in the sample coil and the coil is placed between the poles of the electromagnet. The field of the electromagnet, controlled by the function generator, is slowly swept. Modulation coils driven by the audio amplifier are attached to the poles and modulate the magnetic field. The magnetic field modulation appears in the output of the Robinson Oscillator and allows the signal to be detected by the lock-in amplifier. When the sample resonates it absorbs energy from the coil which reduces the oscillator output.

Robinson oscillator

(See Figs. 7 & 8 of Appendix B): The radio frequency (RF) of the tank circuit is fed into a low noise FET amplifier (transistors T_1 and T_2) loosely coupled to the tank circuit through capacitor $C_1^{'}$ (usually 1 pf). The amplified RF signal is then fed into a combination RF limiter-detector (T_3 and T_4). The limiter reduces the RF to a constant level independent of the RF level in the tank circuit. By changing the value of C_2 (12-75 pf) and C_1 (usually 2 pf) a small amount of the limited RF is fed back to the tank circuit to produce the desired level of oscillation. The limiting of the RF in the oscillator

prevents the oscillator from saturating. Thus any change in the level of the RF in the tank circuit is due to changes in the tank circuit only and is not compensated for by the nonlinear effects found when an oscillator saturates. The collector of T_3 provides a rectified RF signal whose amplitude is proportional to the RF amplitude in the tank circuit. This signal is filtered to remove the RF and amplified in transistors T_6 and T_7 to give the output signal. Transistor T_5 is a buffered amplifier whose output is fed into a frequency counter. When the feedback through C_1 is too low, oscillation will cease. This can usually be noticed by an increase in the noise of the signal output and a dramatic lowering of the frequency. See Appendix B for more details.
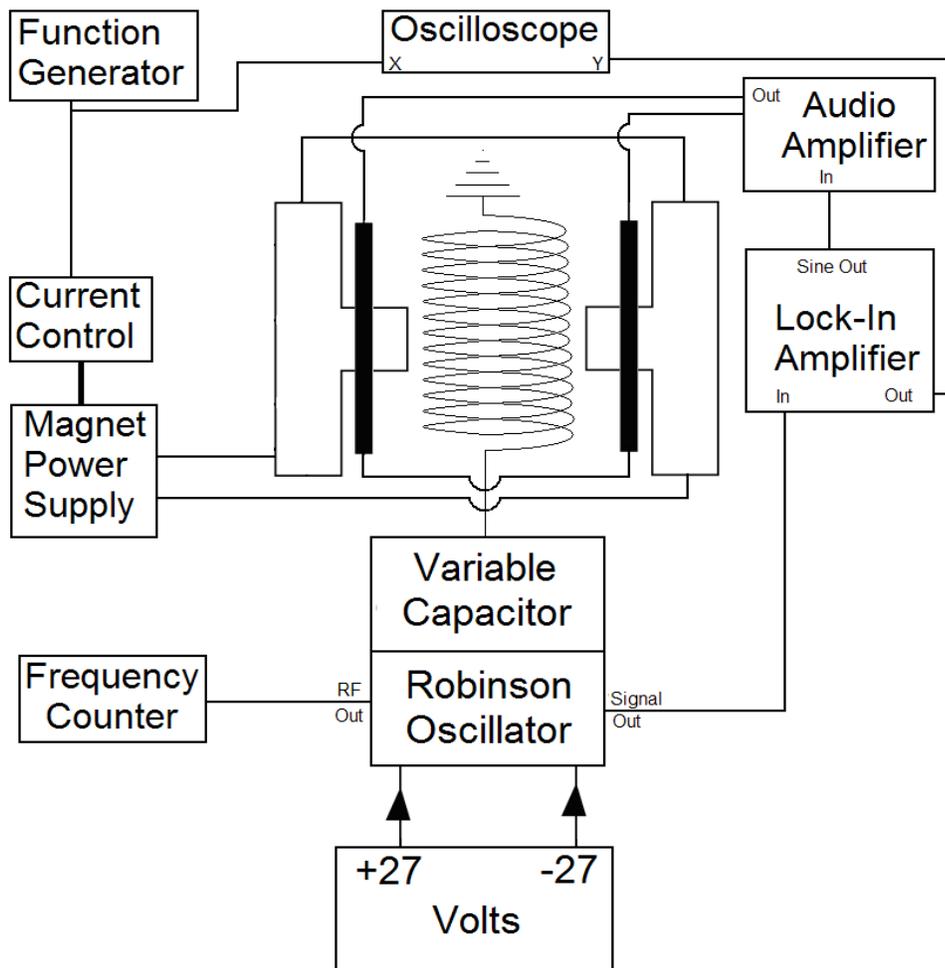
Figure 1. Block diagram of the NMR experiment.

Coil

The coil used in the tank circuit determines the frequency range of the oscillator. The oscillator you will be using has been tested in the frequency range 10-30 MHz. With the provided coil and variable capacitance the oscillator can be tuned from 13 to 26 MHz. A

new coil of different inductance could be made to expand this range. As mentioned in Appendix B, the Q of this coil needs to be as high as possible. A high Q coil ($Q > 150$) is about twice as long as it is wide and the space between the windings equals a little less than the width of the windings. For a coil of length ℓ cm, width w cm and a number of windings N the inductance is:

$$L = \frac{N^2 w}{101(\ell / w + 0.45)}$$

and $Q = \omega L/R$ where R = resistance. Smaller coils provide better field homogeneity over the volume of the sample and reduce the broadening of the NMR line due to the inhomogeneity of the field. However, they also decreases signal strength.

Lock-in detection

Lock-in amplification is used to improve the signal to noise ratio (S/N) of the broad, weak signal obtained from resonating nuclei. This signal is usually too weak to be seen directly on an oscilloscope. The lock-in amplifier works by only amplifying signals with a user selected frequency signature. In this experiment the modulation of the magnetic field provides the needed signature. All other signals are ignored. The output of the lock-in is the derivative of the resonance curve. See Appendix D for more details.

Sample doping

Doping a water sample with less than 0.1 mol/liter of $MnCl_2$ allows the spin system to relax faster and absorb more energy but also broadens the resonance line. If pure water were used the line would be narrower but harder to detect because a lower RF level would be required to avoid saturation.

Hall probe

Refer to Halliday and Resnick for a discussion of the Hall effect.

Equipment

NOTE: Following are special considerations required for some of the equipment. In general, refer to the appropriate manual to learn how to operate a piece of equipment. Manuals are located in lab #203 at the workstation. If a needed manual is not supplied, request one from Mr. Hudek.

Hall probe

Warning: Hall probe is very delicate. **DO NOT** bend or clamp the thin aluminum sheathed portion of the probe. When it is not in use keep the probe in its storage box.

Magnet

For the power supply to become fully stabilized, the "Power" switch must be on for at least one hour prior to normal operation. The magnet current meter reads out in % maximum current; 35 amps is maximum current.

Warning: Before turning on the Walker power supply be sure to completely open the valve to the magnet's cooling water and the valve to the power supply's cooling water. Make sure water is flowing freely through both cooling systems. This water is required to keep the magnet and power supply from overheating and possibly being destroyed.

Audio amplifier

A signal of 20 Volts Peak to Peak will provide adequate modulation. The amplifier will go up to 40 Volts Peak to Peak without significant distortion. Monitor the amplifier output on the oscilloscope.

Warning: Turning the amplifier too high will distort its output. Do NOT use the amplifier with significantly distorted output, this will damage the amplifier.

Robinson oscillator and variable capacitance

The Robinson oscillator requires +27 V and -27 V regulated power. This oscillator has been tested in the frequency range 10-30 MHz. With the supplied coil and capacitance it can be tuned in the range 13-26 MHz. Use the variable capacitance to set the frequency of the oscillator. The RF level of the oscillator tank circuit cannot be measured directly with an oscilloscope probe due to the tank circuit's high Q. Touching a probe to the tank circuit will either severely change the RF oscillation or, as in most cases, stop oscillation completely. Section 7 of Appendix B gives a method for measuring the RF level if necessary.

Sample coil probe

To open the probe and put a sample in the coil, remove the cover with two screws, not the cover with four screws. Isolate the probe from the magnet pole faces or any other source of vibration. Mechanical vibrations experienced by the probe, the capacitors, or the oscillator will be detected by the oscillator. The inductance of the given coil is approximately 10 µH and the Q at 25 MHz is approximately 190. The inductance of the coil, and frequency of the oscillator, can be varied by gently expanding or compressing the distance between the individual windings of the coil.

Lock-in amplifier

To use the lock-in, connect the pot in a box to the "VCO in," connect the "REF out" to the Reference "input" and set the reference signal of about 90 Hz. Feel free to try other frequencies but avoid 60 Hz or its multiples (i.e., 120 Hz) as noise will be picked up from the AC power. The maximum reference phase with a liquid sample is usually zero. When setting up with a liquid sample use a sensitivity of 5 to 10 mV and a time constant of 1

sec. The lock-in signal will have a small DC component due to the oscillator detecting the modulation directly. This is zeroed out using the "REL" or "Offset" functions.

Experimental Procedures

1. Plot the magnetic field (H) versus coil current (I) of the electromagnet using theoretical calculation. There are 800 turns in the magnet's coils.

2. With the Hall probe gaussmeter plot H versus I of the electromagnet at the center of a pole face. For a given I, plot H versus radial distance across the pole face from circumference to circumference.

3. Use the NMR to observe proton resonance in distilled water. See Fig. 1 for a diagram of the NMR spectrometer. Position the probe at the center of the pole gap and set the oscillator frequency near 25 MHz. Modulate the Modulation coils as much as possible without vibrating the coils or significantly distorting the amplifier output, (20 V peak to peak is adequate). Sweep a wide band of the magnetic field with the function generator and find the resonance peak. When the sample resonates, what happens in the coil that allows detection? Does the frequency of the Robinson Oscillator change at resonance? Why?

4. Use the proton resonance of water to plot H versus I of the electromagnet at the center of the pole gap. Compare this plot with the plot you made with the Hall probe. Identify a relationship between frequency of sample resonance and magnetic field.

5. Using the same modulation amplitude used in (3), find the proton resonance of the water sample containing $MnCl_2$. Compare the peak-to-peak line width of the $MnCl_2$ sample with the distilled sample. Plot the peak-to-peak line width of resonance versus modulation amplitude for the $MnCl_2$ sample. Using extrapolation, determine the fundamental line width.

6. With the sample coil at the center of the poles, find the resonance of distilled water with the maximum modulation amplitude that doesn't broaden the line width. Record the line width. Using the same modulation amplitude note the resonance line width with the sample coil half way between the center and the edge of the poles. Do this again with the coil at the edge of the poles. What happens to the line width as the coil is moved toward the edge of the poles? What causes this?

7. Observe the proton resonance in gypsum ($CaSO_4 \cdot 2H_2O$). It may be necessary to adjust the modulation and lock-in sensitivity. Use a longer time constant and slower sweep rate to improve the S/N. Compare your spectrum to Fig. 6 of Appendix E. Carefully measure the peak-to-peak width of the gypsum resonance. Calculate the distance between protons in a gypsum molecule.

References

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