Student Manual

TABLE OF CONTENTS

INTRODUCTION TO NMR
IN THE EARTH'S MAGNETIC FIELD

Gyromagnetic Ratio

Consider a particle of mass m and charge $+q$ rotating at constant angular velocity ω in a circular orbit of radius r. The angular momentum of the particle is

$$L = I\omega = mr^2(2\pi f) .$$

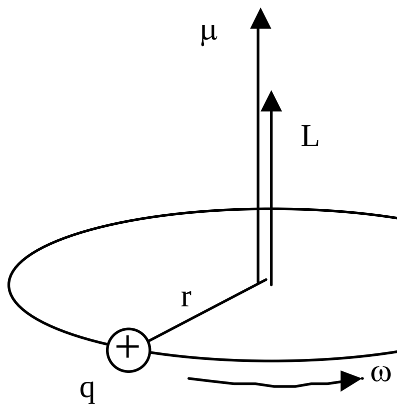


Fig. 1. Magnetic moment and angular momentum vectors for a charged particle rotating at constant angular velocity in a circle of radius *r*.

Associated with the moving charge is an average current $I = qf$ in the loop, and a magnetic moment

$$\mu = IA = qf\left(\pi r^2\right) .$$

Eliminating r^2 from the last two equations yields

$$\vec{\mu} = \gamma\vec{L} , \tag{1a}$$

where

$$\gamma = \frac{q}{2m} . \tag{1b}$$

Vector symbols have been included in Eq. (1a) to indicate that the magnetic moment and angular momentum vectors point in the same direction, as shown in Fig. 1. The constant γ is known as the *gyromagnetic ratio*. (Perhaps "*magnetogyric*" ratio would have been a better name.) Although derived here for just one very special case, Eqs. (1a) and (1b) can be shown to hold equally well for other cases, like spinning charged rings, balls, and spherical shells.

Equation (1a)—but not (1b)—even holds for atomic nuclei and orbital electrons, as has been verified by both experiment and application of the quantum theory. For protons, for example, calculating the gyromagnetic ratio using Eq. (1b) gives $\gamma = q/2m$ = (1.602 x 10^{-19} C)/(2 x 1.672 x 10^{-27} kg) = 4.79 x 10^7 C/kg. The measured value is $\gamma = \vec{\mu}/\vec{L} = 2.675 \times 10^8$ C/kg, which is larger than the classically-computed value by a factor of 5.58. Still, considering the simplicity of the classical model, the agreement between the measured and classically-computed gyromagnetic ratios for protons is surprisingly good.

The Curie Law

Figure 2 shows an idealized model of a proton, which we imagine classically to be a ball of spinning positive charge. It has spin angular momentum $\vec{L}$ and magnetic moment $\vec{\mu}$, both of which point in the same direction along the axis of rotation.

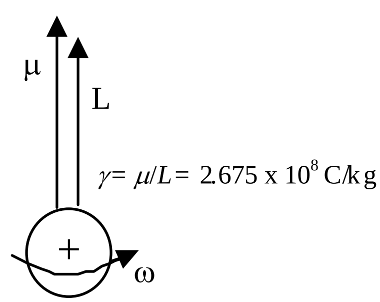


Fig. 2. Classical model of a proton. The magnetic moment and spin angular momentum vectors point in the same direction along the axis of rotation.

Consider the water sample shown schematically in Fig 3. Each water molecule contains two hydrogen atoms, and each hydrogen atom has a nucleus consisting of a single proton. In the absence of a magnetic field, the proton magnetic moments in water are, according to the classical model, randomly oriented in space. If, however, the water sample is placed in an external field $\vec{B}$, the proton magnetic moments will tend to align themselves with the field. At absolute zero the alignment would be perfect, and the net nuclear magnetization of the sample, defined as the magnetic moment per unit volume, would be $M = N\mu$, where μ is the magnetic moment of a single proton, and N is the number of magnetic moments (hydrogen nuclei) per unit volume. (For water, N is just twice the number of water molecules per unit volume.) At temperatures above

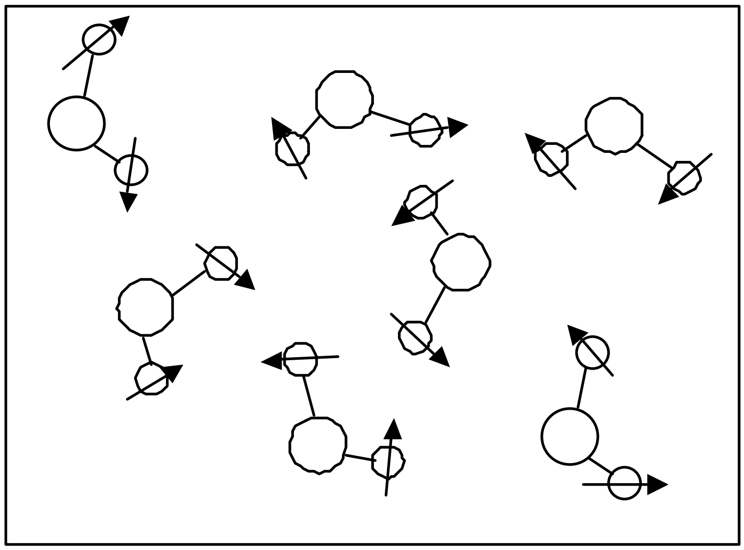


Fig. 3. Schematic representation of molecules of water, H_20. In the absence of an external magnetic field, the magnetic moment of each proton (hydrogen nucleus) is randomly oriented in space.

absolute zero, thermal motions disturb the alignment, so that a typical magnetic moment makes some angle θ with the external field. The magnetization of the sample is $M = N\mu\overline{\cos\theta}$, where $\overline{\cos\theta}$ represents the average value of $\cos\theta$ for all magnetic moments in the sample. Calculation of $\overline{\cos\theta}$ using classical thermodynamics gives the familiar result known as the Curie law:

$$M_o = \frac{N\mu^2 B}{3kT}, \tag{2}$$

where M_o is the equilibrium magnetization in the field B, k is Boltzmann's constant, and T is the temperature on the Kelvin scale. The derivation of the Curie law can also be done using quantum mechanics. The end result can be written in exactly the same form as Eq. (2) if one makes the substitution

$$\mu = \gamma\hbar\sqrt{I(I+1)} \ , \tag{3}$$

where I is the nuclear spin quantum number, equal to 1/2 for protons.

The Curie law predicts a magnetization proportional to the field B and inversely proportional to the temperature in kelvins. It is valid so long as $\mu B/kT << 1$. Because of the small size of nuclear magnetic moments, the Curie law holds at virtually all obtainable temperatures.

Spin-Lattice Relaxation Time

When an external field B is applied to a sample, the magnetization M does not assume the value predicted by the Curie law instantaneously, but approaches the equilibrium magnetization in a manner that is, in most cases, exponential. Representative graphs of B and M versus time are shown in Fig. 4.

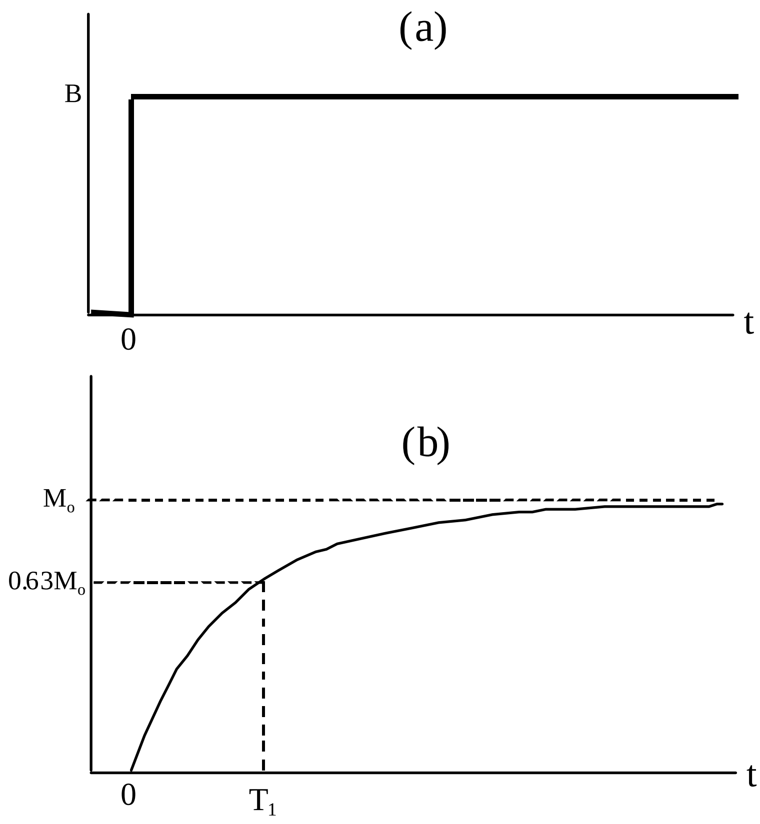


Fig. 4. (a) A constant magnetic field B is switched on at time $t = 0$.
(b) The magnetization grows exponentially with time constant T_1 toward the equilibrium Curie value M_o.

The graph of M versus time in Fig. 4(b) is described by the equation

$$\mathrm{M}(t) = M_o\left(1 - e^{\frac{-t}{T_1}}\right), \qquad (4)$$

where M_o represents the equilibrium magnetization given by Eq. (3). The time constant T_1, known as the spin-lattice relaxation time, is the time it takes for the magnetization to rise to $(1 - e^{-1})$, or about 63%, of the equilibrium Curie magnetization M_o. After two time constants $\mathrm{M}(2T_1) = 0.86M_o$. After five time constants $\mathrm{M}(5T_1) = 0.99M_o$. In water, for example, for which the relaxation time is about 2.5 s at room temperature, the magnetization will have reached slightly more than 99% of its equilibrium value within 12.5 s after the field is turned on.

Larmor Precession

When a proton is placed in an external magnetic field $\vec{\mathrm{B}}$, it experiences a magnetic torque $\vec{\tau} = \vec{\mu} \times \vec{\mathrm{B}}$ that tends to align the proton magnetic moment with the field. However, because of its spin angular momentum $\vec{L}$, the proton's motion is a precession about the field $\vec{B}$ at angular frequency $\omega = \gamma B$. (See Appendix A for a derivation of this expression.) In an earth's field of approximately 0.5 Gauss = 0.05 mT, the precession frequency is

$$f = \omega/2\pi = \gamma B_e/2\pi = (2.675\times10^8\ \mathrm{s^{-1}T^{-1}})(0.05 \times 10^{-3}\mathrm{T})/2\pi = 2.1\ \mathrm{kHz}\ .$$

It is interesting to note that, in a uniform field, all protons within a sample precess at the same frequency independent of cone angle θ. Furthermore, since f is independent of θ, magnetization $\vec{M}$ precess at the same frequency f also.

The Earth's-Field Free Precession Technique

In the earth's-field free precession technique of studying magnetic moments and relaxation times, the sample is placed inside a coil oriented with its axis perpendicular to the earth's field $\vec{B}_e$ as in Fig. 5. A current I_p in the coil produces a polarizing field $\vec{B}_p$ perpendicular to $\vec{B}_e$, and nuclear magnetization $\vec{M}$ builds up with time constant T_1 toward the equilibrium Curie value in the resultant field $\vec{B}$, the vector sum of $\vec{B}_p$ and $\vec{B}_e$. We designate the angle between $\vec{B}$ and the earth's field as α. Since the polarizing field is normally several hundred times the earth's field, the initial value of this angle (α_o) is usually very close to 90°.

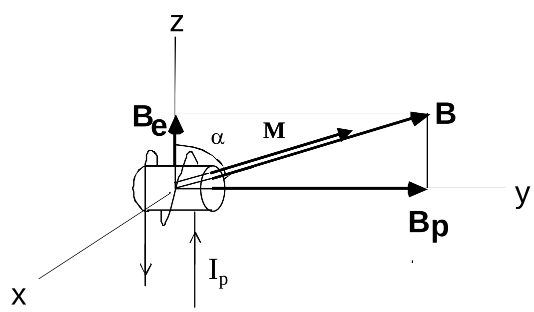


Fig. 5. A schematic representation of the geometry for the earth's-field free precession technique.

When the coil current is reduced to zero, the resultant field $\vec{B}$ shrinks in magnitude and rotates through angle α_o. If the polarizing field is reduced quickly (sudden passage), the magnetization is "left behind" and ends up precessing about $\vec{B}_e$ at frequency $\omega = \gamma B_e$ in a cone of angle $\theta_f \approx \alpha_o \approx 90°$. The precessing magnetization produces a changing magnetic field in the coil, which induces a signal in the coil, with signal amplitude proportional to $M \sin\theta_f$, the component of $\vec{M}$ perpendicular to $\vec{B}_e$. For maximum signal, the polarizing field must be reduced to zero quickly in order to achieve the desired sudden-passage condition $\theta_f \approx \alpha_o \approx 90^o$.

A simplified block diagram of the apparatus required for detecting the free precession signal is shown in Fig. 6. A switching circuit (represented by a relay in Fig. 6) connects the coil to a dc power supply for polarizing the sample, or to a high-gain, narrow-bandwidth, tuned amplifier for detecting the free precession signal. When the user presses the START button, the switching circuit connects the coil to the power supply, and turns the polarizing current on in the coil. Magnetization M grows exponentially with time constant T_1 toward the equilibrium Curie value in the polarizing field. Graphs of B_p and M versus time are shown in Fig. 7. When the polarizing field is switched off suddenly at the end of the polarizing time t_p, the magnetization is left behind, and begins precessing about the earth's magnetic field at frequency $f = \omega/2\pi = (\gamma/2\pi)B_e$. The precession frequency is typically in the range 2.0 - 2.5 kHz.

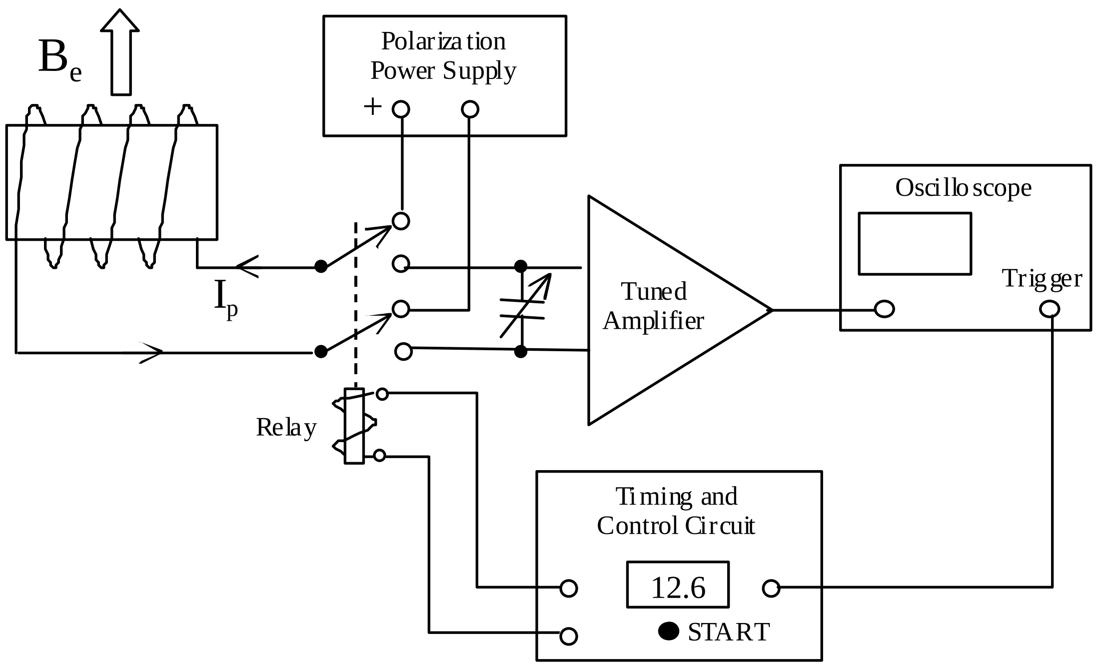


Fig. 6. Simplified block diagram of the apparatus required for the earth's-field free precession technique.

The precessing magnetization induces a sinusoidal signal in the coil at the precession frequency (see Fig. 7(b)) whose amplitude is proportional to the magnetization M(t_p). The switching circuit quickly disconnects the coil from the power supply, and connects the coil to the tuned amplifier, which allows the user to view the free precession signal on an oscilloscope screen. The signal does not persist indefinitely. Magnetization $\vec{M}$ relaxes toward its new, and much smaller, equilibrium value along the direction of the earth's magnetic field. The free precession signal decays toward zero with time constant T_2, the spin-spin relaxation time in the earth's field B_e. By measuring the initial amplitude of the free precession signal as a function of polarizing time t_p, the user can trace out the magnetization curve in Fig. 7(b) and determine the spin-lattice relaxation time T_1 in the field B_p. One of the most attractive features of the technique is that the earth provides—for free—a stable uniform magnetic field in which to detect the free precession.

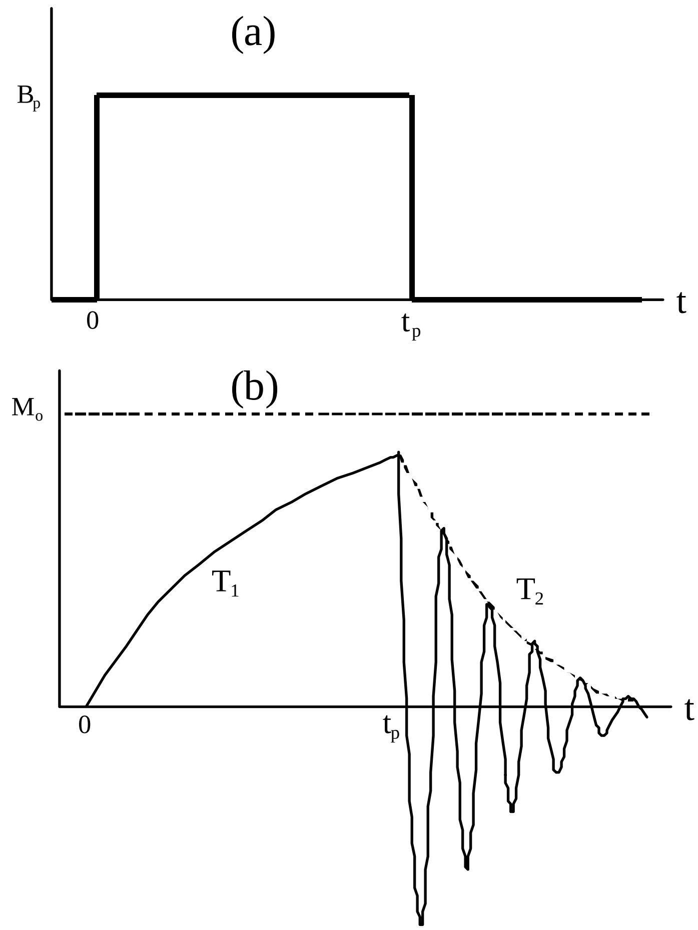


Fig. 7. Variation of polarizing field B_p and magnetization M with time in the earth's-field free precession technique. (a) The polarizing field B_p is switched on at time $t = 0$, and switched off at the end of the polarizing time t_p. (b) When the field is first turned on, magnetization rises exponentially toward the equilibrium value with time constant T_1. When the polarizing field is reduced suddenly to zero at time t_p, the magnetization precesses about the earth's field and induces a signal in the Sample Coil having initial amplitude proportional to $M(t_p)$. The magnetization and signal decay toward zero with time constant T_2.

THE EARTH'S-FIELD NMR INSTRUMENT

Block Diagram and General Description

A more detailed block diagram of the apparatus required for the earth's-field free precession technique is shown in Fig. 8. It consists of 4 major elements:

1) Sample and Bucking Coils,
2) Digital Timing and Control Circuit,
3) Current-Switching and Relay Circuits,
4) Preamplifier, Bandpass Amplifier, and Amplitude Detector.

The two Bucking Coils are included to compensate for any stray signals that may be picked up by the Sample Coil due to fluorescent lights and other sources of electrical noise. During signal detection, the Sample and Bucking Coils are connected in series. The Bucking Coils together have the same total number of area-turns as the Sample Coil (number of turns times the average surface area of one turn), but the turns on the Bucking Coils are wound in the opposite direction. Therefore, the majority of the pickup noise in the Sample Coil is cancelled by an equal and opposite emf induced in the Bucking Coils.

The Signal and Current Relays in Fig. 8 are both double-pole. They are shown in Fig. 8 in their quiescent (unenergized) states, with the Current Relay open, which disconnects the coils from the Polarization Power Supply and Current-Switching Circuit. In the quiescent state, the Signal Relay connects the Sample and Bucking Coils to the Preamplifier for signal detection. Pressing the MANUAL START switch initiates the following ordered sequence of events:

1) The Signal Relay is energized, which disconnects the coils from the Preamplifier and grounds the Preamplifier input.
2) The Current Relay is energized, which connects the Sample Coil to the Current-Switching Circuit and Polarization Power Supply.
3) The Digital Timing and Control Circuit sends a pulse to the Current-Switching Circuit, which turns current on in the coil. A counter begins counting from the preset polarizing time down to zero.

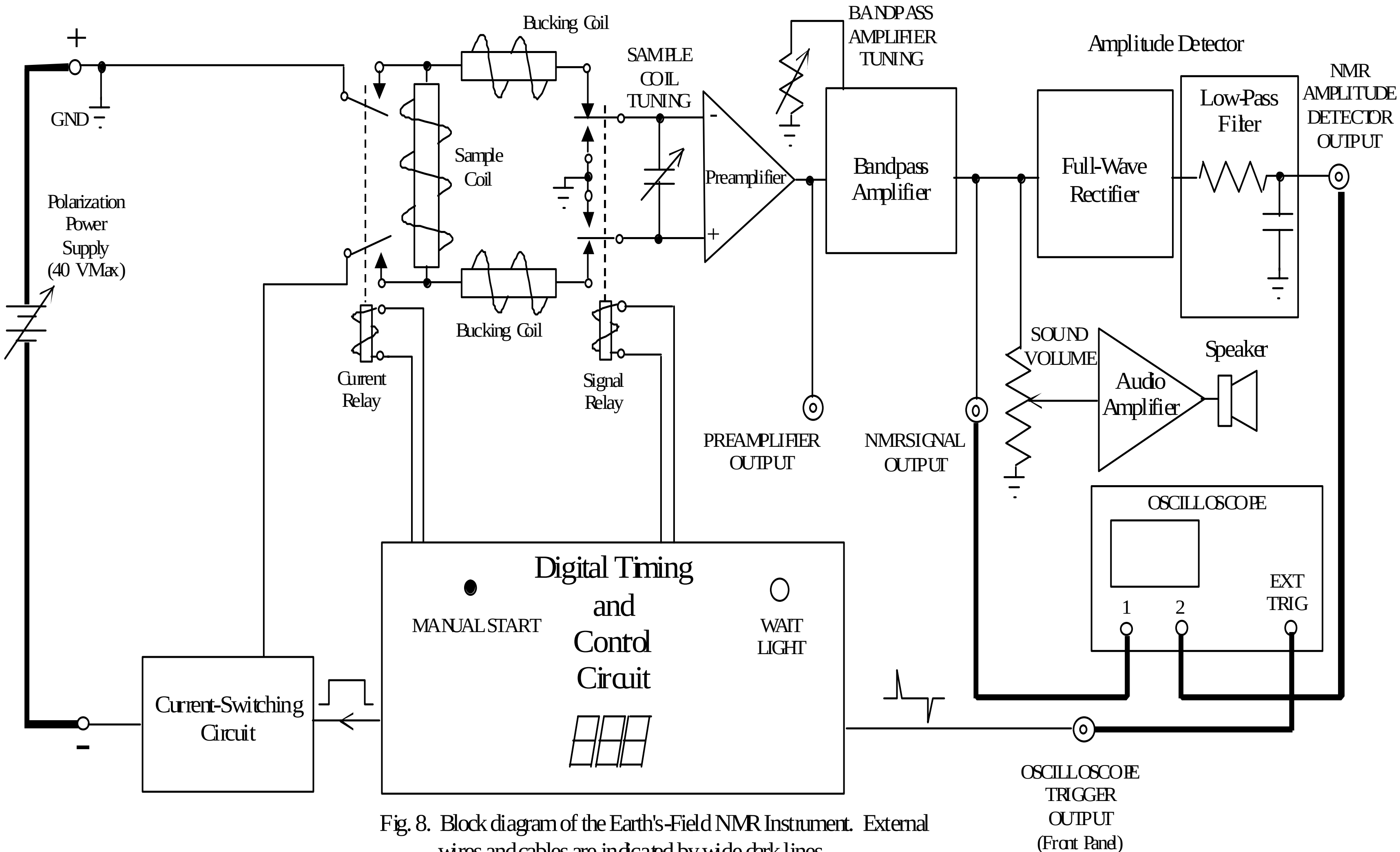


Fig. 8. Block diagram of the Earth's-Field NMR Instrument. External wires and cables are indicated by wide dark lines.

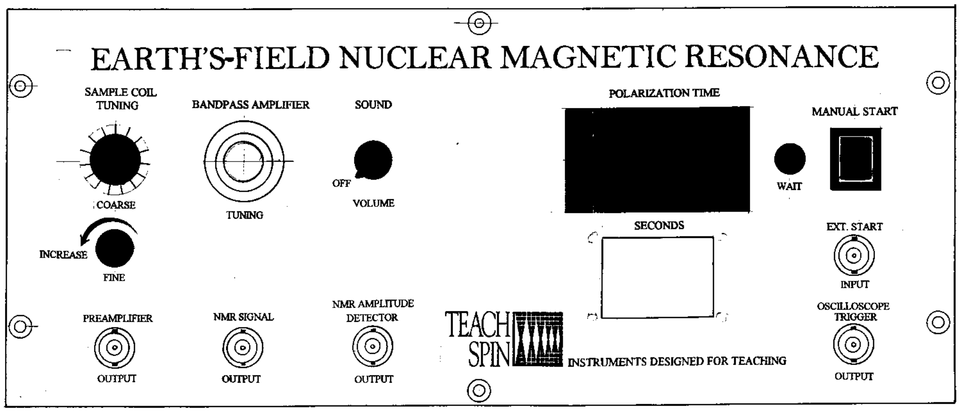
On the count of zero, counting stops, and the Current-Switching Circuit turns off the current in the Sample Coil within a few milliseconds, which is sufficiently fast to meet the condition for sudden passage.

4) After a short delay, to insure that the current has been reduced to zero, the Current Relay is de-energized, which opens the relay and disconnects the Sample Coil from the Current-Switching Circuit and Polarization Power Supply.
5) After another short delay, to insure that the Current Relay has opened, the Signal Relay is de-energized, which connects the Sample and Bucking Coils to the Preamplifier. At the same time, the control circuit provides a pulse to trigger the oscilloscope sweep for signal detection. On the count of zero, the WAIT light comes on. The digital counter remains on the count of zero for 5.0 s, and then counts back up to the original polarizing time. These two time delays are included to prevent rapid cycling of the instrument that might cause overheating of the Sample Coil and Current-Switching Circuit.

The free precession signal is normally viewed by connecting an oscilloscope to the NMR SIGNAL OUTPUT. The PREAMPLIFIER OUTPUT is provided as an aid in adjusting the input capacitors to tune the series LC resonant circuit to the precession frequency. The NMR AMPLITUDE DETECTOR OUTPUT is useful for samples for which the signal is small; multiple signals can be collected and averaged to improve signal-to-noise.

Controls and Connectors

Front Panel



THUMBWHEEL SWITCHES and DIGITAL DISPLAY

Users set the polarizing time in the range 0.1 to 99.9 seconds by means of thumbwheel switches on the front panel. Switch settings are automatically loaded into the count register of an internal presettable down counter. Contents of the count register are continuously displayed on the seven-segment digital displays during both loading and counting operations.

MANUAL START

Pressing the MANUAL START button initiates the automatic sequence necessary for acquiring a free precession signal. The polarization time t_p is the time set via the thumbwheel switches and displayed on the seven-segment displays.

EXTernal START INPUT

A contact closure to ground on this input (or a negative TTL pulse) performs the same function as pressing the MANUAL START button.

WAIT light

To prevent rapid cycling of the apparatus that would lead to overheating the Sample Coil and Current-Switching Circuit, the apparatus automatically delays 5.0 s on the count of zero. There is an additional delay (equal to the polarization time) as the counter counts from zero back up to the time that was set on the thumbwheel switches when the

MANUAL START button was pressed. The WAIT light is turned on during both of these delays.

SAMPLE COIL TUNING (COARSE and FINE)

The input to the Preamplifier (see Fig. 8) is the voltage across a capacitor that is connected in series with the Sample and Bucking Coils. The resonant frequency of this series circuit, $\omega_o = (LC)^{-1/2}$, can be adjusted to coincide with the Larmor precession frequency by rotating the COARSE and FINE switches to vary the capacitance. Rotating either switch counterclockwise decreases the capacitance and increases the resonant frequency

PREAMPLIFIER OUTPUT

This output is provided for monitoring the output of the Preamplifier while adjusting the COARSE and FINE controls to tune the resonant frequency of the input circuit to the Larmor precession frequency. When the input circuit is properly tuned to the frequency of the free precession signal, the amplitude of the signal at the output of the Preamplifier will be a maximum.

BANDPASS AMPLIFIER TUNING

The Preamplifier is followed by a Bandpass Amplifier that is included to block high and low frequency noise from the Preamplifier. The center frequency of the Bandpass Amplifier can be adjusted by means of this ten-turn potentiometer.

NMR SIGNAL OUTPUT

The output of the Bandpass Amplifier is connected to the NMR SIGNAL OUTPUT, which is normally connected to an oscilloscope for observing the free precession signal.

SOUND VOLUME

The NMR SIGNAL OUTPUT is connected internally to an audio power amplifier that drives an internal speaker. This allows users to hear the free precession signal, as well as view it on an oscilloscope.

NMR AMPLITUDE DETECTOR OUTPUT

The signal available at the NMR SIGNAL OUTPUT is also connected internally to an Amplitude Detector, which consists of a Full-Wave Rectifier followed by a Low-Pass Filter. The output of the filter is the average value of the full-wave rectified free precession signal. For a full-wave rectified sinusoidal signal, the average value is $2/\pi$ of the peak value. Thus, the output of the NMR AMPLITUDE DETECTOR has essentially the same shape as the envelope of the free-precession signal, but the amplitude is about 2/3 as large

OSCILLOSCOPE TRIGGER OUTPUT

When using an oscilloscope to view the free precession signal at any one of the three outputs on the front panel (PREAMPLIFIER OUTPUT, NMR SIGNAL OUTPUT, OR NMR AMPLITUDE DETECTOR OUTPUT), connect the OSCILLOSCOPE TRIGGER OUTPUT on the front panel to the EXTernal TRIGger input on the oscilloscope. The signal at the OSCILLOSCOPE TRIGGER OUTPUT is two narrow pulses similar to those shown in Fig. 9. The leading (positive) pulse occurs at the instant the Signal Relay is deactivated, which connects the amplifier to the Sample and Bucking Coils. The trailing (negative) pulse occurs after a fixed delay of 80 ms, which is sufficient time to allow switching transients to die away before triggering the scope to view the precession signal.

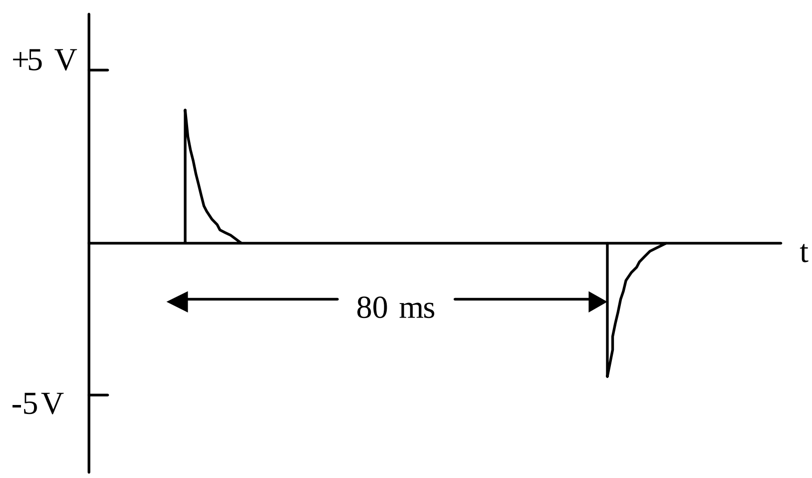
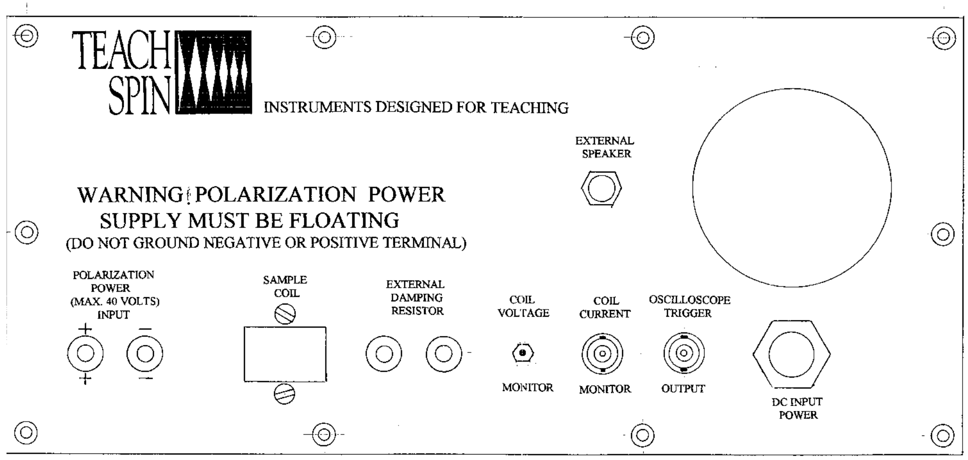


Fig. 9. Pulse sequence at the front-panel OSCILLOSCOPE TRIGGER OUTPUT.

Back Panel



POLARIZATION POWER INPUT (MAX 40 VOLTS)

The external power supply for providing polarizing current is connected to these two banana plug sockets. In order to obtain maximum signal, the power supply should be capable of providing 3.0 A at 36 volts. Both terminals of the power supply should be floating, i.e., neither terminal should be connected to ground. The positive terminal is connected to ground inside the Earth's-Field NMR instrument.

SAMPLE COIL

The four-wire shielded cable that connects to the Sample and Bucking Coils plugs into this socket.

EXTERNAL DAMPING RESISTOR

COIL VOLTAGE MONITOR

COIL CURRENT MONITOR

OSCILLOSCOPE TRIGGER OUTPUT

EXTERNAL SPEAKER

These connections are provided for testing and for special applications. Their use is covered in the Instructor's Manual

INITIAL SETUP

Positioning the Coils

If the sample is located in a region where the earth's magnetic field B_e is perfectly homogeneous, the free precession signal decays with time constant T_2, the spin-spin relaxation time that is characteristic of the sample. For water, T_2 is equal to T_1 in the range of magnetic fields accessible to the earth's-field free precession technique; both are about 2.5 seconds at room temperature. In practice, however, one finds that the earth's magnetic field is not perfectly homogeneous, even outdoors, and even over relatively small sample volumes of 100 ml. Because of the earth's-field inhomogeneity, spins in different parts of the sample precess at slightly different frequencies $\omega_e = \gamma B_e$. As the spins precess they gradually lose phase coherence with each other, which causes the signal to decay toward zero with time constant T_2* that is less than T_2. If there are magnets or magnetic materials nearby that significantly distort the earth's magnetic field, T_2* can be so short that the signal dies away before the switching transient, which persists for times on the order of 50 ms. It is, therefore, desirable to position the coils in an area where the earth's magnetic field is as homogeneous as possible. That usually means near the open center of a room, about four or five feet above the floor.

Ideally, the coils should also be placed in an area as far away as possible from sources of radiated electrical noise. Fluorescent lights in the same room as the coils should be turned off.

Once you have positioned the coils, orient the coils with their axes pointing east-west, perpendicular to magnetic north. Now you are ready to tune the instrument in order to detect a free precession signal.

Tuning the Instrument to Obtain Maximum Signal

1) To avoid distorting the earth's magnetic field, and to minimize possible pickup noise, position the Earth's-Field NMR instrument as far as practical away from the coils. About 3 meters (10 feet) is usually sufficient. Connect the coils to the instrument by inserting the plug on the coil cable into the socket on the back panel
2) Fill a 125-ml plastic bottle with tap water, and place it in the center of the Sample Coil.

3) Make sure the power supply you intend to use for providing the polarizing current does not exceed 40 volts. Also, make certain that both outputs are floating, i.e., neither terminal is connected to ground. With the power supply turned off, connect the + and - terminals on the power supply to the corresponding terminals on the back panel. The + terminal of the power supply is connected to ground inside the instrument. DON'T TURN THE POLARIZING POWER SUPPLY ON JUST YET. WAIT UNTIL INSTRUCTED TO DO SO IN PROCEDURE 7.
4) Connect the low-voltage "brick on a rope" power supply to the DC INPUT POWER connector on the back panel. The instrument should now be on, and the seven-segment displays on the front panel should display whatever polarizing time happens to be set on the thumbwheel switches.
5) In order to detect a free precession signal, connect the PREAMPLIFIER OUTPUT to one of the input channels of an oscilloscope. Superimposed on the signal is a dc offset of up to ±500 mV. To eliminate the offset, set the oscilloscope COUPLING to AC. Set the vertical sensitivity to 50 mV/Div, and the sweep speed to 2 ms/Div.
6) Connect the OSCILLOSCOPE TRIGGER OUTPUT on the front panel to the EXTernal TRIGger input on the oscilloscope. Set the oscilloscope to trigger on EXTernal, DC COUPLING with HF (High Frequency) REJECT, -SLOPE, and -LEVEL (≈ -1 V). This will insert an 80-ms delay between the time the Signal Relay is de-energized (which connects the coils to the Preamplifier) and the time the oscilloscope begins its sweep. Thus, the switching transient will not be visible since it will have died away before the oscilloscope sweep begins.
7) Set the COARSE and FINE SAMPLE COIL TUNING switches to the middle of their ranges. Turn on the Polarization Power Supply; set the voltage at about 34 volts. If it is a current-limiting supply; set the current limit at maximum. Set the polarizing time to about two times T_1 (about 5.0 s for water); then press the MANUAL START button. If the precession frequency is near the resonant frequency of the tuned circuit, you will see a free precession signal.
8) Turn the COARSE SAMPLE COIL TUNING switch clockwise one position, and try again.

If the signal is larger than before, you are searching in the right direction. Turn the COARSE switch clockwise one additional position, and repeat the process.

If the signal is smaller than before, you have gone in the wrong direction. Rotate the COARSE switch counterclockwise two positions, and try again.

If you still haven't seen any signal at all it may be because the resonant frequency of the tuned circuit is still too far away from the precession frequency. Turn the COARSE switch clockwise one additional notch, and try again. Keep rotating the switch clockwise until you detect the signal or come to the end of the range. If you come to the end of the range and still haven't detected the signal, set the COARSE switch back to the center of its range, and begin searching for the signal as you rotate the COARSE switch counterclockwise one step a time.

9) Once you have found the setting of the COARSE switch that gives maximum signal, begin adjusting the FINE switch to obtain the largest signal possible. When you have finished, the resonant frequency of the tuned circuit coincides with the free precession frequency.

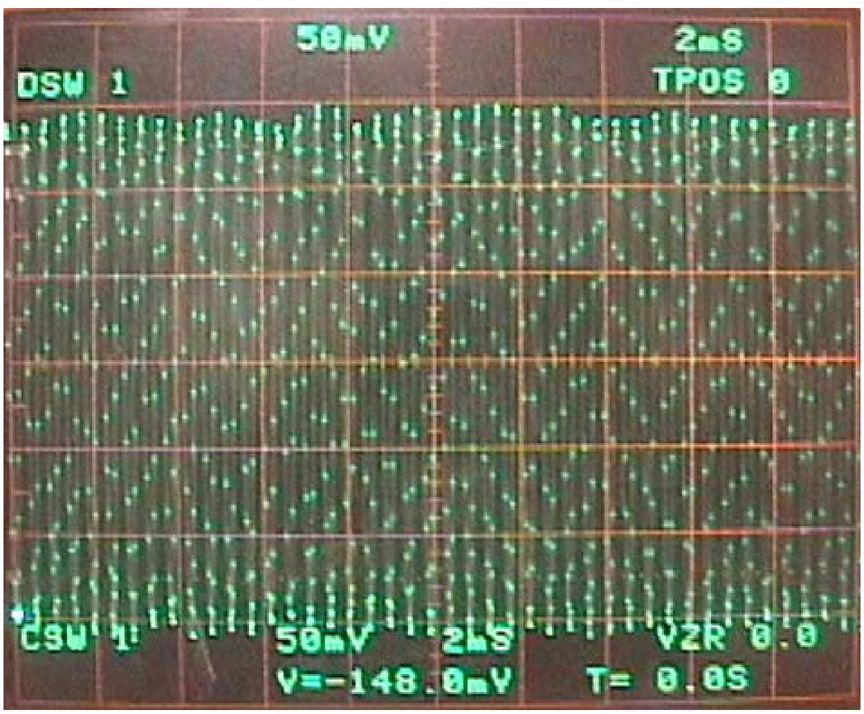


Fig. 10. Free precession signal at the PREAMPLIFIER OUTPUT. The oscilloscope is set at 50 mV/Div and 2 ms/Div. The sample frequency is 100 samples/Div for all oscilloscope waveforms shown in this manual.

Adjusting the Center (Resonant) Frequency of the Bandpass Amplifier

In addition to supplying more signal amplification, the Bandpass Amplifier prevents electrical noise at frequencies far above and below the free precession frequency from reaching the NMR SIGNAL OUTPUT. By adjusting the ten-turn BANDPASS AMPLIFIER TUNING potentiometer, it is possible to adjust the center frequency of the Bandpass Amplifier to coincide with the frequency of the free precession signal. When that occurs, the amplitude of the free precession signal at the NMR SIGNAL OUTPUT will be a maximum. You will accomplish that in the steps that follow:

10) Leaving the PREAMPLIFIER OUTPUT connected to the oscilloscope, connect the NMR SIGNAL OUTPUT to the unused oscilloscope input channel. Adjust the controls on the oscilloscope to view *only* the channel connected to the NMR SIGNAL OUTPUT. (Since the NMR SIGNAL OUTPUT has no DC offset, you may set the oscilloscope COUPLING to either AC or DC.) Set the vertical sensitivity to 1 Volt/Div. As a first approximation, set the ten-turn BANDPASS AMPLIFIER TUNING potentiometer to the center of its range. Without changing the settings of the COARSE and FINE SAMPLE COIL TUNING switches, cycle the instrument; as you do so, adjust the potentiometer to obtain a signal of maximum amplitude at the NMR SIGNAL OUTPUT. (Adjusting the potentiometer should have little or no effect on the amplitude of the signal at the PREAMPLIFIER OUTPUT.) As you adjust the potentiometer you can start by making relatively coarse adjustments in one- or one-half-turn increments. As the center bandpass frequency gets nearer the free precession frequency, you will need to make finer adjustments.

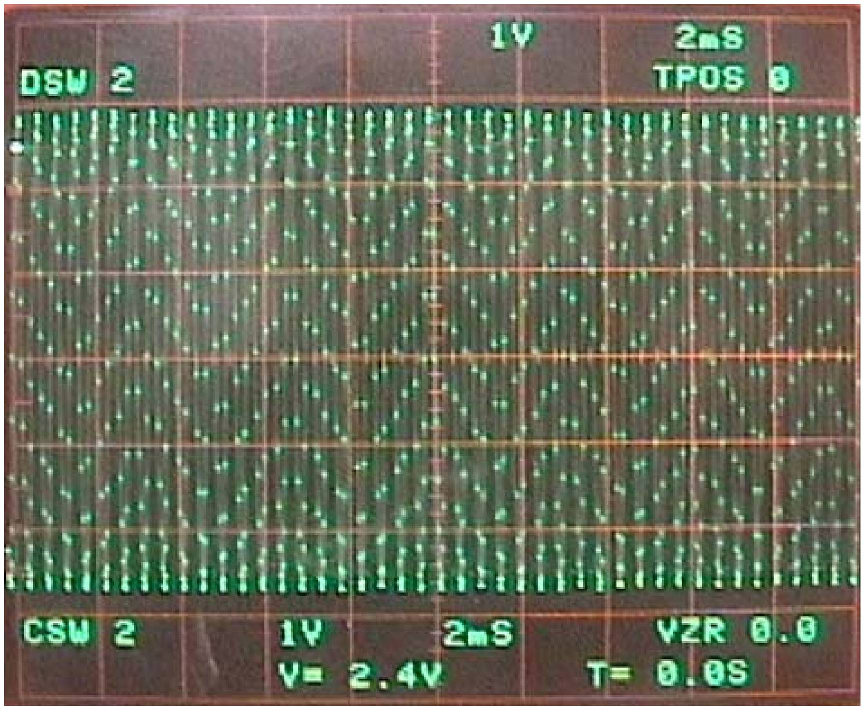


Fig. 11. Free precession signal at the NMR SIGNAL OUTPUT. The oscilloscope is set at 1 Volt/Div and 2 ms/Div.

Figure 12 shows a free precession signal at the NMR SIGNAL OUTPUT obtained with the oscilloscope sweep speed reduced to 50 ms/Div. (The free precession frequency was 2088 Hz; yet the digitized waveform appears to have a much lower frequency of only 88 Hz. A discussion of the source of this problem—called aliasing—and what to do about it may be found in *Appendix B* and in the section on the *NMR Amplitude Detector*.)

The signal in Fig. 12 exhibits the Gaussian-shaped decay that is commonly observed where the earth's field indoors is relatively inhomogeneous. Refer to Fig. 13 on page 22 for an example of the longer decays that are observed in more homogeneous earth's magnetic fields.

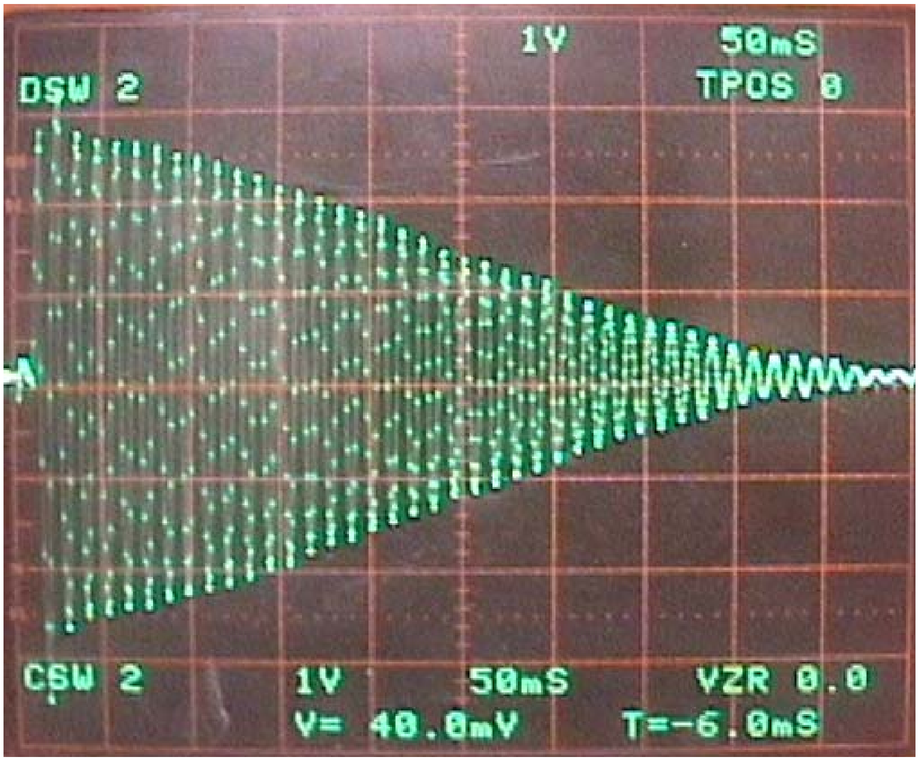


Fig. 12. Water free precession signal at the NMR SIGNAL OUTPUT. Note the Gaussian-shaped decay caused by a relatively inhomogeneous earth's magnetic field. The oscilloscope is set at 1 V/Div and 50 ms/Div.

NMR AMPLITUDE DETECTOR

Figure 13 shows the NMR SIGNAL from a water sample viewed simultaneously with the signal at the NMR AMPLITUDE DETECTOR OUTPUT. As discussed in the section on *Front Panel Controls and Connectors*, the output of the Amplitude Detector has essentially the same shape as the envelope of the free precession signal, but an amplitude that is $2/\pi$, or about 2/3, as large. The signals in Fig. 13 were obtained with the 80-ms oscilloscope sweep delay disabled. Therefore, we can see the switching transient and initial growth of the signal, followed by the exponential decay. The same signals as in Fig. 13 are shown in Fig. 14, but there the sweep speed has been increased from 50 ms/Div to 20 ms/Div. Note in Fig. 14 the apparent beats in the amplitude of the free precession signal at the NMR SIGNAL OUTPUT. These beats are not real; they are an artifact caused by the fact that the digital storage oscilloscope samples and digitizes the input waveforms at a frequency that depends on the oscilloscope sweep speed. Significant distortions can occur when the sample frequency is less than, or on the order of, the frequency of the input signal. Since the output of the NMR AMPLITUDE DETECTOR is a relatively slowly varying signal, it is relatively immune to problems associated with reducing the oscilloscope sample rate. Refer to *Appendix B* for a more complete discussion.

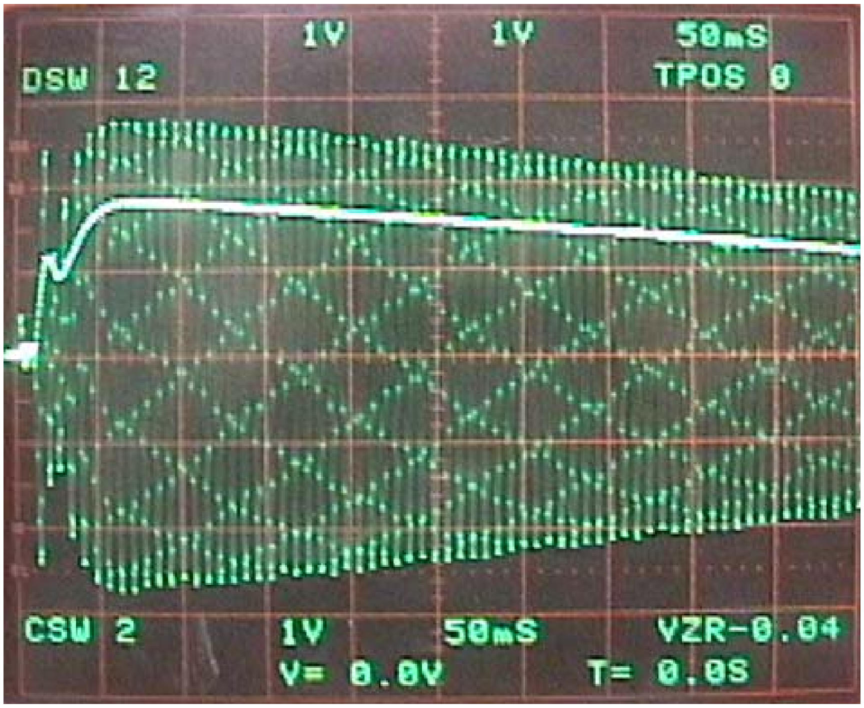


Fig. 13. Water NMR SIGNAL shown along with the output of the NMR AMPLITUDE DETECTOR. Both oscilloscope channels are set at 1V/Div and 50 ms/Div.

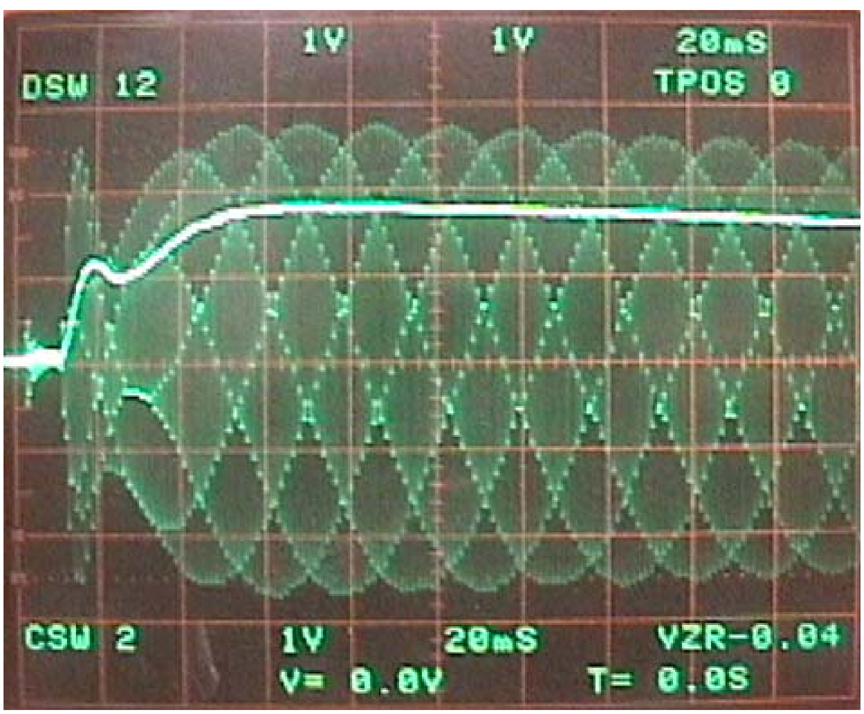


Fig. 14. Same signals as in Fig. 13, but with the oscilloscope sweep speed increased to 20 ms/Div. Note the appearance of "beats" in the NMR SIGNAL OUTPUT caused by problems associated with aliasing.

APPENDIX A: Larmor Precession

Figure A1 shows a positively charged nucleus with its magnetic moment oriented at angle θ with respect to a magnetic field $\vec{B}$ pointing along the +z axis. The spin angular momentum vector $\vec{L}$ is not shown in the figure, but it points in the same direction as $\vec{\mu}$. We wish to prove that at the magnetic moment $\vec{\mu}$ will precess about $\vec{B}$ at constant angular velocity $\omega = \gamma B$. That is, the tip of $\vec{\mu}$ will follow the circular dotted path shown in the figure.

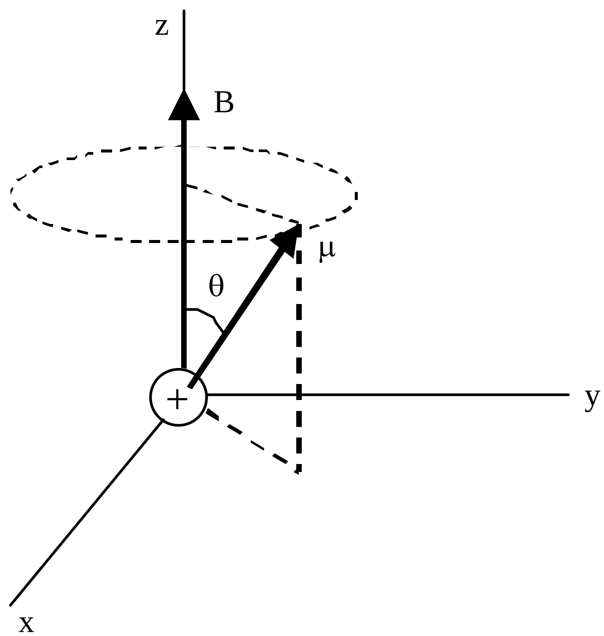


Fig. A1. A positively charged nucleus with its magnetic moment $\vec{\mu}$ oriented at angle θ with respect to a magnetic field $\vec{B}$ pointing along the +z axis. The angular momentum vector $\vec{L}$, not shown, points in the same direction as $\vec{\mu}$.

We begin by writing Newton's 2nd law in the form

$$\vec{\tau} = \frac{d\vec{L}}{dt} \quad . \tag{A1}$$

The magnetic torque tending to align $\vec{\mu}$ with $\vec{B}$ is given by

$$\vec{\tau} = \vec{\mu} \times \vec{B} \quad . \tag{A2}$$

By combining Eqs. (A1) and (A2), and making the substitution $\vec{\mu} = \gamma\vec{L}$, we obtain

$$\frac{d\vec{u}}{dt} = \gamma\, \vec{\mu} \times \vec{B} \quad . \tag{A3}$$

By making the substitutions $\vec{B} = \hat{k}\, B$ and $\vec{\mu} = \hat{i}\, \mu_x + \hat{j}\, \mu_y + \hat{k}\, \mu_z$ the vector equation (A3) can be written as three separate scalar equations, one for each of the components:

$$\frac{d\mu_x}{dt} = \gamma B \mu_y , \tag{A4}$$

$$\frac{d\mu_y}{dt} = -\gamma B \mu_x , \tag{A5}$$

and

$$\frac{d\mu_z}{dt} = 0 . \tag{A6}$$

Solving Eq. (A6) yields $\mu_z(t) = \mu_z(0) = \text{constant}$ By multiplying Eq. (A5) by $i = \sqrt{-1}$ and adding the result to Eq. (A4) we obtain

$$\frac{d}{dt}\left(\mu_x + i\mu_y\right) = -i\gamma B\left(\mu_x + i\mu_y\right) .$$

By making the substitution $\mu_+ = \mu_x + i\mu_y$ this equation may be simplified to

$$\frac{d\mu_+}{dt} = -i\gamma B \mu_+ ,$$

which has as its solution

$$\mu_+(t) = \mu_+(0) e^{-i\gamma Bt} .$$

Expanding by using Euler's identity, and substituting $\mu_+ = \mu_x + i\mu_y$, yields

$$\mu_x(t) + i\,\mu_y(t) = \left(\mu_x(0) + i\,\mu_y(0)\right)\left(\cos\gamma Bt - i \sin\gamma Bt\right).$$

By choice of axes let $\mu_x(0) = 0$. Then, by equating the real and imaginary parts of both sides of the previous equation, we obtain

$$\mu_x(t) = \mu_y(0) \sin\gamma Bt$$

and

$$\mu_y(t) = \mu_y(0) \cos\gamma Bt ,$$

which shows that the angular velocity of precession is $\omega = \gamma B$. It is interesting, perhaps surprising, to note that the angular frequency of precession is independent of cone angle θ.

APPENDIX B: Aliasing

A typical digital oscilloscope samples the input waveform at fixed time intervals, and then displays the digitized samples on the oscilloscope screen. These samples are normally connected by straight line segments in order to give, at least roughly, the appearance of a smooth waveform. Figure B1 shows a 2200 Hz sinusoidal waveform and, for comparison, the sampled approximation. The samples are shown as

Fig. B1. The rough approximation that results when a 2.2 kHz sinusoidal waveform is sampled at 10 kHz

squares, and the squares are shown connected by straight line segments. In constructing Fig. B1 the time between samples was assumed to be 0.1 ms, which corresponds to a sampling frequency of 10 kHz, which is only 4.5 times the frequency of the input signal. Thus, on average, each cycle of the input signal is approximated by only about 5 points. Furthermore, the points are joined by straight line segments rather than a smooth curve. The distortion is obvious. Some of the cycles appear to have "missing peaks" since the oscilloscope digitizer did not happen to sample the input waveform when it was at a maximum.

A similar graph is shown in Fig. B2, but in this case the sampling frequency has been increased to 20 kHz, which is almost ten times the frequency of the input waveform. The digitized approximation now appears to have about the same shape as the input waveform. Now each cycle is approximated by ten straight line segments, rather than just five as before. However, there are still some obvious distortions. Note, for example, the 4th peak from the left, which occurs near time t = 1.5 ms.

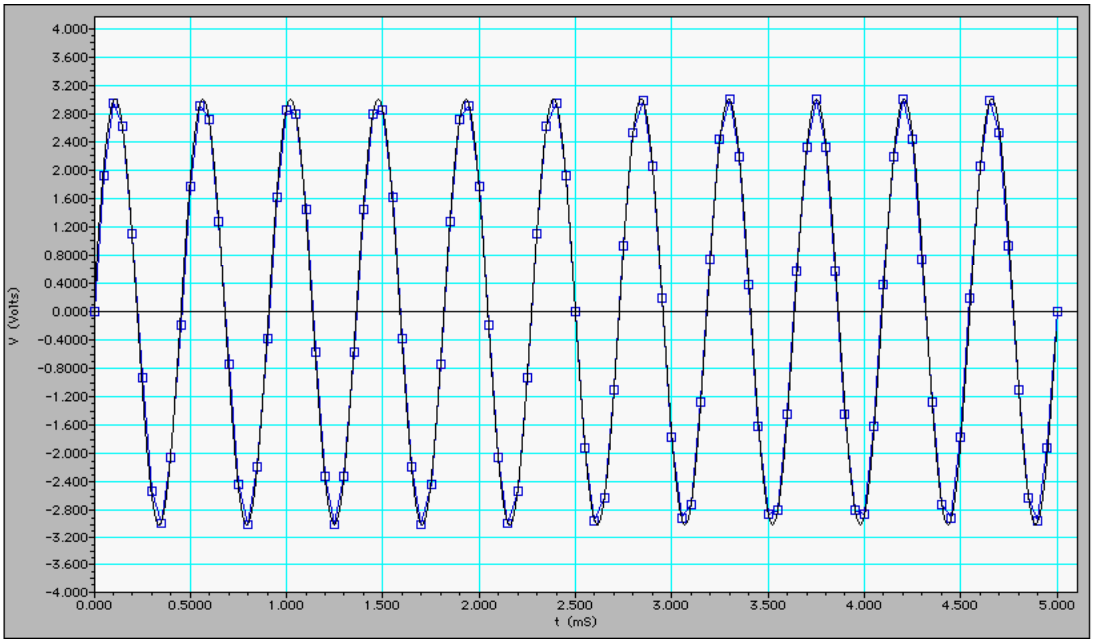


Fig. B2. The relatively smooth waveform that results when a 2.2 kHz sinusoidal waveform sampled at 20 kHz

The oscilloscope happened to sample the waveform on either side of the peak, but not at the peak itself. Therefore, when viewed on the oscilloscope screen, the peak will appear to be "flattened off" and slightly reduced in size relative to other peaks of the waveform. Thus, in order to obtain a waveform that is displayed smoothly on the screen, it is necessary to have an oscilloscope sampling frequency that is *more* than ten times the frequency of the signal.

At low sampling frequencies it is easy to be completely misled by the digitized waveform displayed on the oscilloscope screen. Figure B3 shows the same 2200 Hz signal as before, but the sampling frequency has been reduced to 2 kHz. Note that the input waveform is sampled only about once each cycle, and the digitized waveform

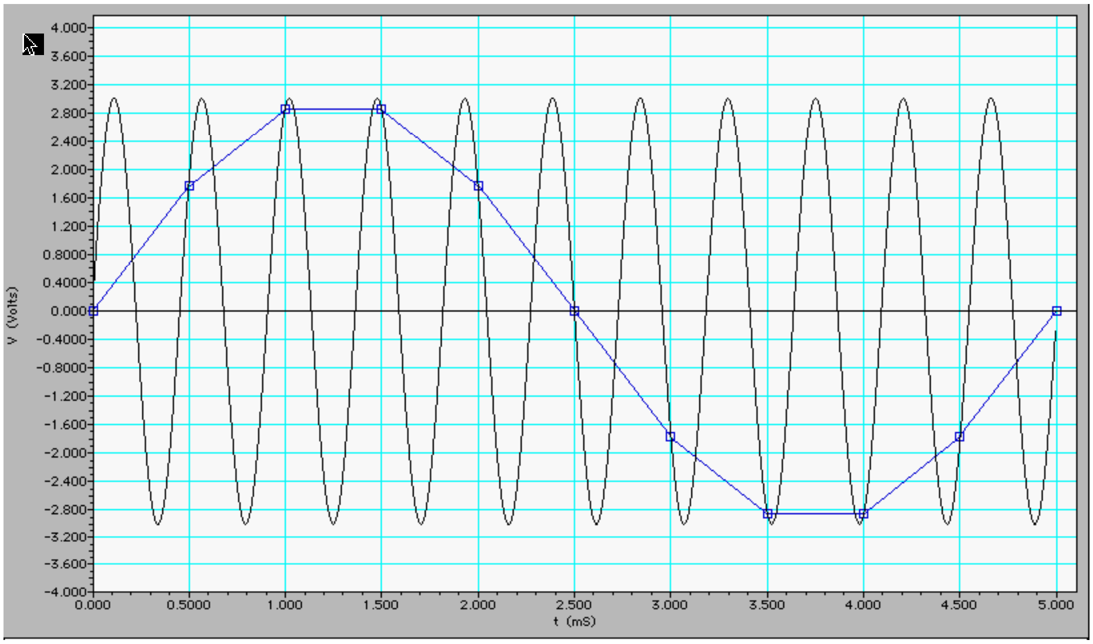


Fig. B3. Missing cycles that result when a 2.2 kHz sinusoidal waveform is sampled at 2 kHz, which does not meet the Nyquist criterion.

appears to be, at least approximately, a sinusoidal signal of much lower frequency than the input waveform! To avoid these kinds of errors, the sampling frequency must meet the requirement of the Nyquist theorem, which states that the sampling frequency must be at least twice the signal frequency. For comparison, Fig. B4 shows the 2200 Hz input waveform sampled at 5 kHz (time between samples is 0.2 ms). This sampling frequency meets the Nyquist criterion, which effectively means that the sampled waveform has no "missing cycles." However, it still looks greatly distorted. Based on the appearance of the sampled waveform in Fig. B4 one might be tempted to conclude, quite incorrectly, that the input waveform exhibits beats. (For an example of apparent beats in a free precession signal, refer to Fig. 14 on p. 22.)

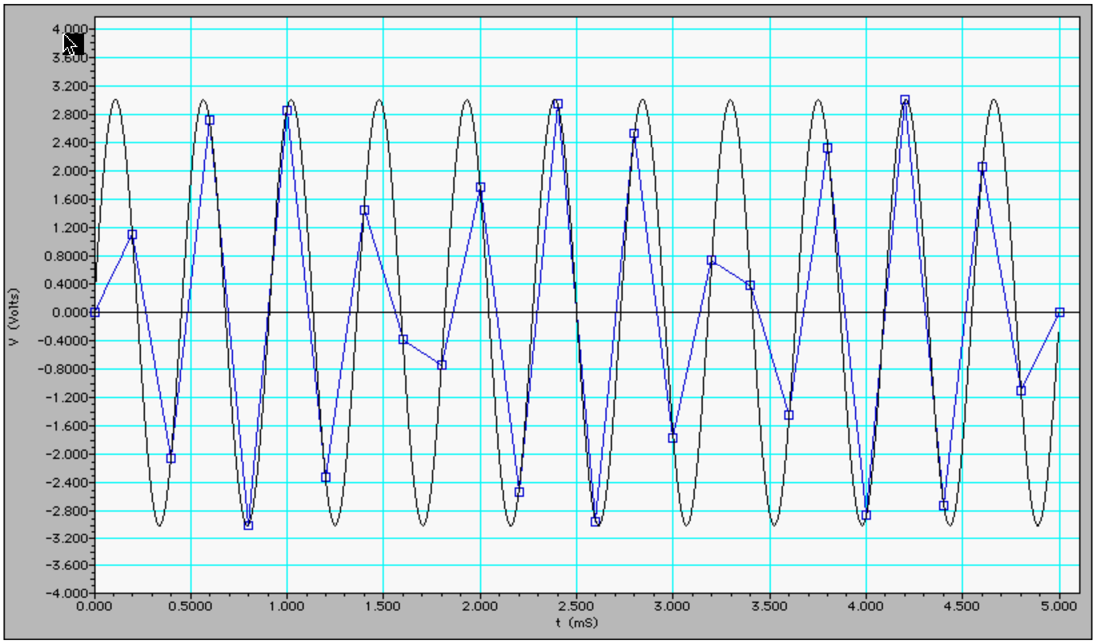


Fig. B4. A 2.2 kHz sinusoidal waveform sampled at 5.0 kHz. There is significant distortion even though the sampling frequency meets the Nyquist criterion.

So, what does all this mean with regard to using digital storage oscilloscopes for capturing free precession signals? First of all, it is obviously desirable to have a sampling frequency that is at least ten times the frequency of the free precession signal. For signals at 2.1 kHz, corresponding to an earth's field of 0.05 mT, the sample frequency should be of the order of 20 kHz or more. Specifications for digital oscilloscopes may claim maximum sampling frequencies of hundreds of thousands, or even millions, of samples per second. But those specifications may be misleading. Digital oscilloscopes vary, but, for most commonly-used oscilloscopes, the oscilloscope takes only 500 or 1000 samples during one horizontal sweep. Assuming the screen is 10 divisions wide, that corresponds to as few as 50 samples per division. At a sweep speed of 0.2 ms/Div, 50 samples per division corresponds to a time interval of 0.004 ms between each sample, which is equivalent to a sample frequency of only 25 kHz, barely sufficient to produce a smooth digitized waveform. At 50 samples per division and a sweep speed of 20 ms/Div, the time between samples is 0.4 ms. The corresponding sample frequency is only 2.5 kHz, which does not even meet the Nyquist criterion. Therefore, severe sampling errors like that shown in Fig. B3 can be expected when the oscilloscope sweep speed is 20 ms/Div or slower. The following examples will illustrate this principle.

Figure B5 shows a photograph of an oscilloscope waveform obtained from a 125-ml sample of water. The frequency of the free precession signal was 2.088 kHz. The oscilloscope sweep speed was 50 ms/Div. The waveform appears to be relatively

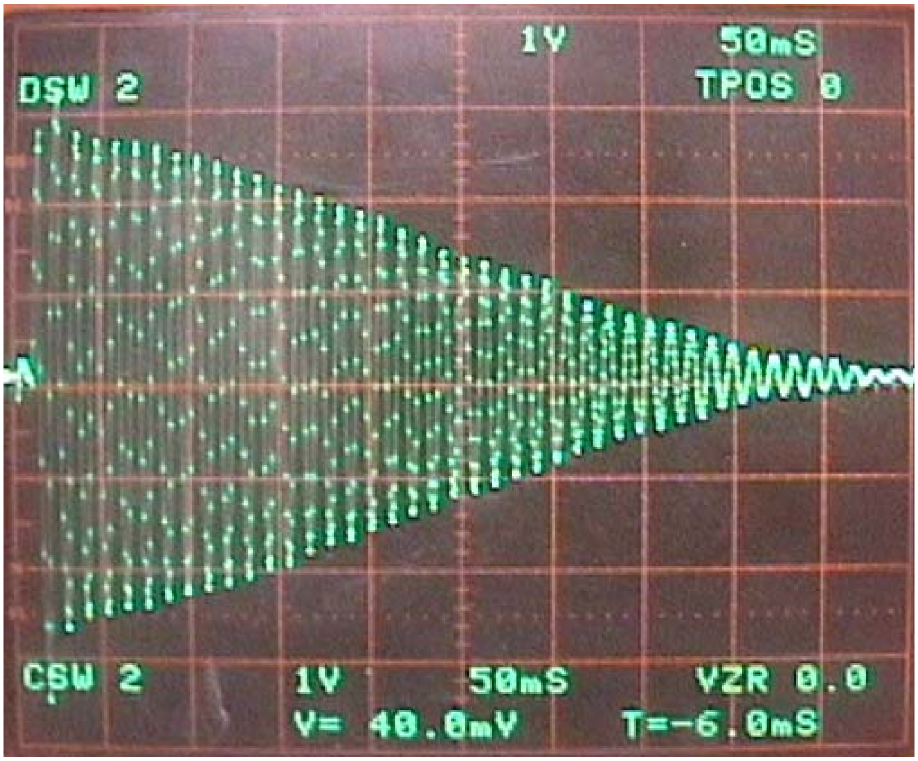


Fig. B5. Apparently smooth free precession signal at a frequency of 2.088 kHz. The sample frequency was 2 kHz.

smooth, with multiple samples per cycle. However, that is not the case. The oscilloscope was sampling at 100 samples/division, which corresponds to a time interval between samples of 0.5 ms and a sample frequency of 2 kHz. Thus, the sample frequency is just slightly less than the frequency of the signal. The situation is similar to that in Fig. B3. What appears in Fig. B5 to be many closely-spaced samples on the same cycle are actually single samples taken on many successive cycles. For a signal frequency of 2.088 kHz and a sample frequency of 2.000 kHz, the difference in frequencies (or beat frequency) is 88 Hz, which is the apparent frequency of the digitized waveform in Fig. B5.

The waveform in Fig. B6 shows an even more extreme example of aliasing The 2.007 kHz signal was obtained from fluorine nuclei in a 25-gram sample of

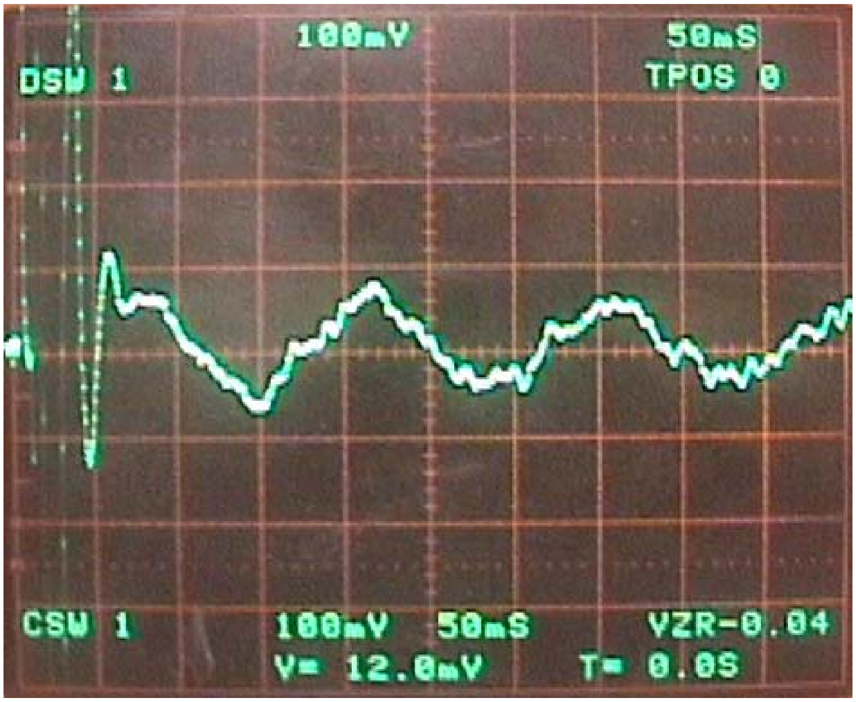


Fig. B6. 2.007 kHz fluorine signal from a 25-gram sample of C_6F_6. The oscilloscope sample frequency was 2.000 kHz. The digitized waveform appears to have a frequency equal to the difference, 7 Hz.

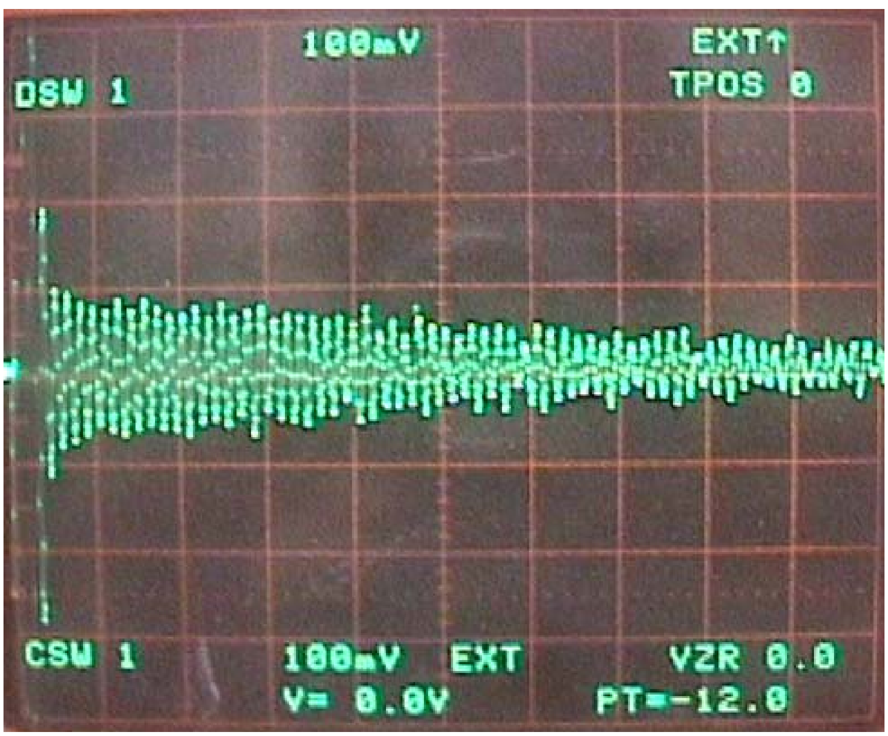


Fig. B7. Same as Fig. B6, except the oscilloscope sample frequency was reduced to 1.040 kHz. Since the oscilloscope displays 100 samples per division, the equivalent sweep speed is 96 ms/Div.

hexafluorobenzene, C_6F_6. The oscilloscope sweep speed was 50 ms/Div, and the oscilloscope sample frequency was 2 kHz. Here, the signal and sample frequencies differ by only 7 Hz, which is identical to the apparent frequency of the sampled waveform observed on the oscilloscope screen. The same fluorine signal is shown in Fig. B7, but there the sample frequency was reduced to 1.040 kHz. The time between samples was 0.96 ms, which is almost twice the period. The situation is similar to that shown in Fig. B3, except the sample frequency was so low that the sampling process skipped whole cycles. Yet, the sampled waveform appears surprisingly smooth.

EXPERIMENTS

PRIOR TO PERFORMING ANY OF THESE EXPERIMENTS, THE APPARATUS MUST BE POSITIONED AND TUNED AS DESCRIBED IN THE SECTION ON **INITIAL SETUP**.

EXPERIMENT 1: Measurement of the Proton Spin-Lattice Relaxation Time in Water

Objectives

The objective of this experiment is to measure the proton spin-lattice relaxation time T_1 in water at room temperature.

Equipment

Earth's-Field NMR instrument

Polarization power supply (floating outputs, 40 volts maximum)

Oscilloscope

125-ml sample bottle

Theory

According to the Curie law, the equilibrium magnetization of a sample containing magnetic moments μ is

$$M_o = \frac{N\mu^2}{3kT} B \; , \tag{1}$$

where μ is the magnetic moment of each spin; N is the number of magnetic moments per unit volume; B is the magnetic field; k is Boltzmann's constant; and T is the temperature on the Kelvin scale. The magnetization of the sample does not assume the equilibrium value instantaneously, but, rather, rises exponentially toward the Curie value with time constant T_1, the spin-lattice relaxation time. The growth of M(t) toward M_o is described by the equation

$$\mathrm{M}(t) = M_o\left(1 - e^{-\frac{t}{T_1}}\right) \; , \tag{2}$$

where M_o is the equilibrium Curie magnetization, and M(t) is the magnetization at time t. By rearranging Eq. (2) we obtain

$$M_o - \mathrm{M}(t) = M_o e^{-\frac{t}{T_1}} \; . \tag{3}$$

By taking the natural logarithm of both sides, Eq. (3) can be rewritten

$$\ln\left(M_o - \mathrm{M}(t)\right) = \ln\left(M_o\right) - \frac{1}{T_1}t \,. \tag{4}$$

From Eq. (4) we see that a plot of ln(M_o-M(t)) versus t should be a straight line having intercept ln(M_o) and slope equal to -1/T_1. This provides a straightforward graphical method of determining the spin-lattice relaxation time.

Procedure

1. Fill a 125-ml sample bottle with tap water, and place it in the center of the Sample Coil.
2. Connect the NMR SIGNAL OUTPUT and NMR AMPLITUDE DETECTOR OUTPUT to oscilloscope channels 1 and 2 (or A and B), respectively. Adjust the oscilloscope controls so you can view both channels simultaneously. Set the vertical sensitivities of both channels to 1 V/Div and the COUPLING to DC. Set the horizontal sweep speed to 2 ms/Div.
3. Connect the OSCILLOSCOPE TRIGGER OUTPUT on the front panel of the instrument to the EXTernal TRIGger input on the oscilloscope. Set the oscilloscope to trigger on EXTernal, DC COUPLING with HF REJECT, -SLOPE, and -LEVEL (≈ -1 V). These settings will cause the oscilloscope to delay its sweep 80 ms, which is sufficiently long to allow switching transients to die away before start of the sweep.
4. If the power supply is a variable voltage supply, set it at about 36 volts. If the power supply has variable voltage and current limiting, set the voltage on 36 volts, and set the current limit knob at maximum.
5. Set the polarizing time to 13.0 s.
6. Press the MANUAL START button. When the current switches on, make the following adjustments depending on your type of power supply:

   Variable Voltage but No Current Limiting

   If the polarization power supply has variable voltage, but not current limiting, reduce the power supply voltage to give a polarizing current of 3.0 A. Note the voltage that is required. At 3.0 A the power dissipated in the Sample Coil is roughly 100 watts.

As time goes by, the Sample Coil will get warm; its resistance will increase, and it will be necessary to increase the voltage slightly in order to maintain the polarizing current constant.

Variable Voltage With Current Limiting

If the polarization power supply has both variable voltage and current limiting, wait until the current switches on. Then turn the current limit knob counterclockwise until the polarizing current drops to 3.0 A. Note the power supply voltage. You will find that the current-limiting power supply has automatically reduced the output voltage in order to provide the desired current of 3.0 A. At 3.0 A the power dissipated in the Sample Coil is roughly 100 watts. As time goes by, the Sample Coil will get warm; its resistance will increase. But the power supply will automatically increase the voltage as necessary in order to maintain the current fixed at 3.0 A. Ideally, the initial power supply voltage, which was set before the current was switched on, should be about 2 to 3 volts greater than the voltage actually required to provide the desired current. A difference of 2 to 3 volts is sufficient to allow the power supply to compensate for the coil's rise in temperature. If the voltage difference is too large, there may be problems associated with voltage transients that are invariably produced when the power supply current is suddenly switched from zero to 3.0 A.

7. Once the power supply has been properly adjusted to deliver 3.0 A, you are ready to measure the relaxation time by measuring the amplitude of the free precession signal as a function of polarizing time. The amplified free precession signal is available on the NMR SIGNAL OUTPUT. The output of the NMR AMPLITUDE DETECTOR is the NMR SIGNAL after it has been full-wave rectified and filtered. It has the same shape as the envelope of the free precession signal, but its amplitude is $2/\pi$, or about 2/3, as large.

   Pick a convenient reference point on the oscilloscope screen, say 1.0 division (which corresponds to 2.0 ms) after start of the sweep. Measure the zero-to-peak amplitude of the free-precession signal. Also measure the amplitude of the signal at the NMR AMPLITUDE DETECTOR OUTPUT. Do this for polarizing times of 13.0, 5.0, 4.0, 3.0, 2.0, 1.0, and 0.5 seconds. As a general rule, for maximum accuracy in measuring signal amplitudes, the oscilloscope vertical sensitivity should always be adjusted so that waveforms fill as much of the screen as possible. For low-level signals, you will need to increase the vertical sensitivity of both channels to 500 or 200 mV/Div.

8. Trace out the magnetization curve by plotting the amplitude of the free precession signal versus polarizing time. On the same sheet, graph the output of the NMR AMPLITUDE DETECTOR versus polarizing time. Both curves should have the same shape.
9. The relaxation time T_1 can be determined, at least in principle, by fitting Eq. (2), with M_o and T_1 as adjustable parameters, to either of the data sets plotted in Procedure 8. Alternatively, one can assume that M_o is approximately equal to the amplitude of the signal at the longest polarizing time (13.0 s in this case), and plot M_o-M(t) versus t on semi-log graph paper. Equation (4) shows that the slope of this graph is $-1/T_1$. A third alternative is to use a spreadsheet, or similar program, to graph ln(M_o-M(t)) versus t. A linear least-squares fit of the straight line obtained will give the slope, which can then be used to calculate T_1. Whichever method you choose, determine two values for T_1, one for each data set. These values of T_1 should agree to within experimental error.

EXPERIMENT 2: The Curie Law

Equipment

Earth's-Field NMR instrument

Polarization power supply (floating outputs, 40 volts maximum)

Oscilloscope

125-ml sample bottle

Theory

According to the Curie law, the equilibrium magnetization of a sample containing magnetic moments μ is

$$M_o = \frac{N\mu^2}{3kT} B \ , \qquad (1)$$

where μ is the magnetic moment of each spin; N is the number of magnetic moments per unit volume; B is the magnetic field; k is Boltzmann's constant; and T is the temperature on the Kelvin scale. According to the Curie law, for a given sample at constant temperature, the equilibrium magnetization M_o should be proportional to B. Because of the small size of the earth's magnetic field, the net field B is approximately equal to the polarizing field B_p of the coil. (See Fig. 5 in the section on *The Earth's-Field Free Precession Technique*.) Furthermore, B_p is proportional to the coil current I_p. Thus, we expect the equilibrium Curie magnetization M_o to be proportional to the polarizing current I_p in the Sample Coil.

The magnetization of the sample does not assume the equilibrium value instantaneously, but, rather, rises exponentially toward the Curie value with time constant T_1, the spin-lattice relaxation time. The growth of M(t) toward M_o is described by the equation

$$\mathrm{M}(t) = M_o\left(1 - e^{-\frac{t}{T_1}}\right) \ , \qquad (2)$$

where M_o is the equilibrium Curie magnetization, and M(t) is the magnetization at time t. For polarizing times equal to 5 times the T_1 or longer, the exponential term in Eq. (2) is less that 0.01, and M(t) is approximately equal to M_o, to within an error of less than 1%. When the polarizing current is reduced suddenly to zero at the end of the polarizing time t_p, the ensuing free-precession signal dies away with time constant T_2 (actually, T_2* since the earth's field is not perfectly homogeneous). In any case, the amplitude of the free precession signal is proportional to M_o. (Refer to Fig. 7(b) in the section on *The Earth's-Field Free Precession Technique*.) Thus, we expect the amplitude of the precession signal to be proportional to the polarizing current I_p. That is, a graph of initial amplitude of the free precession signal versus I_p should be a straight line.

Procedure

1. Fill a 125-ml plastic sample bottle with tap water, and place it in the center of the Sample Coil.
2. Connect the NMR SIGNAL OUTPUT and NMR AMPLITUDE DETECTOR OUTPUT to oscilloscope channels 1 and 2 (or A and B), respectively. Adjust the oscilloscope controls so you can view both channels simultaneously. Set the vertical sensitivities of both channels to 1 V/Div and the COUPLING to DC. Set the horizontal sweep speed to 2 ms/Div.
3. Connect the OSCILLOSCOPE TRIGGER OUTPUT on the front panel of the instrument to the EXTernal TRIGger input on the oscilloscope. Set the oscilloscope to trigger on EXTernal, DC COUPLING with HF REJECT, -SLOPE, and -LEVEL (≈ -1 V). These settings will cause the oscilloscope to delay its sweep 80 ms, which is sufficiently long to allow switching transients to die away before start of the sweep.
4. If the power supply is a variable voltage supply, set it at about 36 volts. If the power supply has variable voltage and current limiting, set the voltage on 36 volts, and set the current limit knob at maximum.
5. Set the polarizing time to 13.0 s, which is about five time the spin-lattice relaxation time in water at room temperature.
6. Press the MANUAL START button. When the current switches on, make the following adjustments depending on your type of power supply:

   Variable Voltage but No Current Limiting

   If the polarization power supply has variable voltage, but not current limiting, reduce the power supply voltage to give a polarizing current of about 3.0 A. Note the voltage that is required. At 3.0 A the power dissipated in the Sample Coil is roughly

100 watts. As time goes by, the coil will get warm; its resistance will increase, and it will be necessary to increase the voltage slightly in order to maintain the polarizing current constant.

<u>Variable Voltage With Current Limiting</u>

If the polarization power supply has both variable voltage and current limiting, wait until the current switches on. Then turn the current limit knob counterclockwise until the polarizing current drops to 3.0 A. Note the power supply voltage. You will find that the current-limiting power supply has automatically reduced the output voltage in order to provide the desired current of 3.0 A. At 3.0 A the power dissipated in the Sample Coil is roughly 100 watts. As time goes by, the Sample Coil will get warm, and its resistance will increase. But the power supply will automatically increase the voltage as necessary in order to maintain the current fixed at 3.0 A. Ideally, the initial power supply voltage, which was set before the current was switched on, should be about 2 to 3 volts greater than the voltage actually required to provide the desired current. A difference of 2 to 3 volts is sufficient to allow the power supply to compensate for the coil's rise in temperature. If the voltage difference is too large, there may be problems associated with voltage transients that are invariably produced when the power supply current is suddenly switched from zero to 3.0 A.

7. Once the power supply has been properly adjusted to deliver 3.0 A, you are ready to measure the amplitude of the free precession signal. The amplified free precession signal is available at the NMR SIGNAL OUTPUT. The output of the NMR AMPLITUDE DETECTOR is the NMR SIGNAL after it has been full-wave rectified and filtered. It has the same shape as the envelope of the free precession signal, but its amplitude is $2/\pi$, or about 2/3, as large.
8. Pick a convenient reference point on the oscilloscope screen, say 1.0 division (which corresponds to 2.0 ms) after start of the sweep. With the polarizing time kept fixed at 13.0 s, measure the zero-to-peak amplitude of the free precession signal. Also measure the amplitude of the signal at the NMR AMPLITUDE DETECTOR OUTPUT.

9. Repeat Procedures 4-8 for polarizing currents of 2.5, 2.0, 1.5, 1.0, and 0.5 A. The dc resistance of the Sample Coil and connecting cable is on the order of 10-11 ohms. Therefore, reducing the current in 0.5-A steps will require reducing the power supply voltage in steps on the order of 5.0-5.5 volts. **If you are using a current-limiting power supply, be sure to heed the warning given in the next paragraph**. As the polarizing current is reduced, the amplitude of the free precession signal drops as well. As a general rule, for maximum accuracy in measuring signal amplitudes, the oscilloscope vertical sensitivity should always be adjusted so that waveforms fill as much of the screen as possible. For low-level signals, you will need to increase the vertical sensitivity of both channels to 500 or 200 mV/Div.

> **CAUTION: When using a variable voltage supply with current limiting, don't reduce the current limit without simultaneously reducing the power supply voltage limit as well. Ideally, the voltage limit should be set no more than 2-3 volts higher than that required to deliver the desired current.** Otherwise, unacceptably large power supply voltage transients may result. If, for example, the current limit is reduced to 0.5 A while the voltage limit is left at or near the maximum of 36 V, when the current is switched on, the output voltage from the current-limiting power supply will drop suddenly from 36 V toward a steady-state value of 6 V or less, depending on the resistance of the Sample Coil and cable. During switching, the output voltage of a typical current-limiting power supply is an underdamped transient that oscillates as it decays with a time constant on the order of 20 ms or even longer. During large amplitude transients, the power supply output voltage can undershoot so far that it reverses polarity. If that occurs, the switching circuit will turn the current off in the coil until the power supply voltage assumes its normal polarity.

10. Plot the amplitude of the free precession signal versus polarizing current. On the same graph, plot the amplitude of the signal from the NMR AMPLITUDE DETECTOR versus polarizing current. Both graphs should be straight lines having the same slope. This experiment serves as a sensitive test of both the Curie law and for proper operation of the instrument.