

EARTH'S FIELD NUCLEAR MAGNETIC RESONANCE

INSTRUCTOR'S MANUAL STUDENT MANUALS

A PRODUCT OF TEACHSPIN, INC.

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Instruments Designed for Teaching

MEMORANDUM

TO: Instructor

FROM: Dr. Jonathan Reichert
President, TeachSpin, Inc.

RE: Teaching Earth's Field Nuclear Magnetic Resonance

Congratulations on your purchase of our EFNMR-1A apparatus. We at TeachSpin are extremely proud of this apparatus and believe it will make a significant contribution to the teaching of magnetic resonance phenomena at both the introductory and advanced level.

With this apparatus, you now have new ways to introduce an "old" but important subject in physics (as well in other sciences). We only make the instrument, you introduce it to your students. However, because this instrument presents a new paradigm in magnetic resonance instruction, we have developed three different sets of instructions, an Instructor's Manual and two very different student manuals.

The instructor's manual goes into almost every detail we could think of. It is for you. It will help you to become a real "expert" with the apparatus and can serve as a detailed reference guide. The two student manuals are quite different.

The first manual requires students to do some simple derivations and calculations and to carefully read the instructions before they are allowed to perform the experiment. It reviews some basic concepts, introduces important definitions and gives a conceptual description of the apparatus. To be most effective, the pre-lab written assignment and introduction to NMR should be a graded, or even in-class tested, homework assignment.

The manual is written so as to focus the student on the central physics issues but to let them discover the Curie Law and the exponential relationship in the measurement of spin-lattice relaxation. It requires students to develop an experimental strategy, set the controls properly, plot the data as they take it, and generally take ownership of the experiment. It also includes suggestions for extensions they can pursue.

The second manual is in a traditional style. It takes the students through the basics of magnetic resonance theory and asks them to verify that theory. It discusses both the Curie Law and spin lattice relaxation, gives the equations, with some derivation, and tells the students, in detail, how to make the measurements.

This apparatus might appear too complicated for sophomore or junior physics (or chemistry) students if they are confronted with all of its "bells and whistles." We strongly urge that you not do this. We have provided a student manual which will focus their attention on the basic functions of the apparatus and allow them to make most essential measurements. They need not examine every aspect of the instrument.

For example, the bucking coils' function is explained in detail (and is essential for the reduction of pickup noise) but its operation does not need to be studied or deeply understood by the beginning student. Another example is the study of the damping of the polarizing magnetic field as it is switched off. This is certainly not an appropriate part of the NMR experiment, even for the sophomore physics major.

Obviously you will have to choose what experiments are required and in what sequence. These student manuals are only offered as helpful suggestions. We urge you to read all of the "manuals" very carefully before drawing up your own manual for your students. You are free to use any or all parts of these documents. We can provide you with a floppy disk with text so you can easily edit or modify the manual to suite your particular needs. If you do make significant changes to the manual, we would greatly appreciate your sending us a copy. The insights you gain working with your students will help us to improve our version!

Teaching, is much like raising children, we all have our own "wrong" way of doing it. (just kidding). But we really do want to know how you are using the Earth's Field NMR. It will help us to understand the possibilities and limitations of both our instrument and our student manuals.

We hope you and your students enjoy exploring physics with this instrument.

Sincerely,

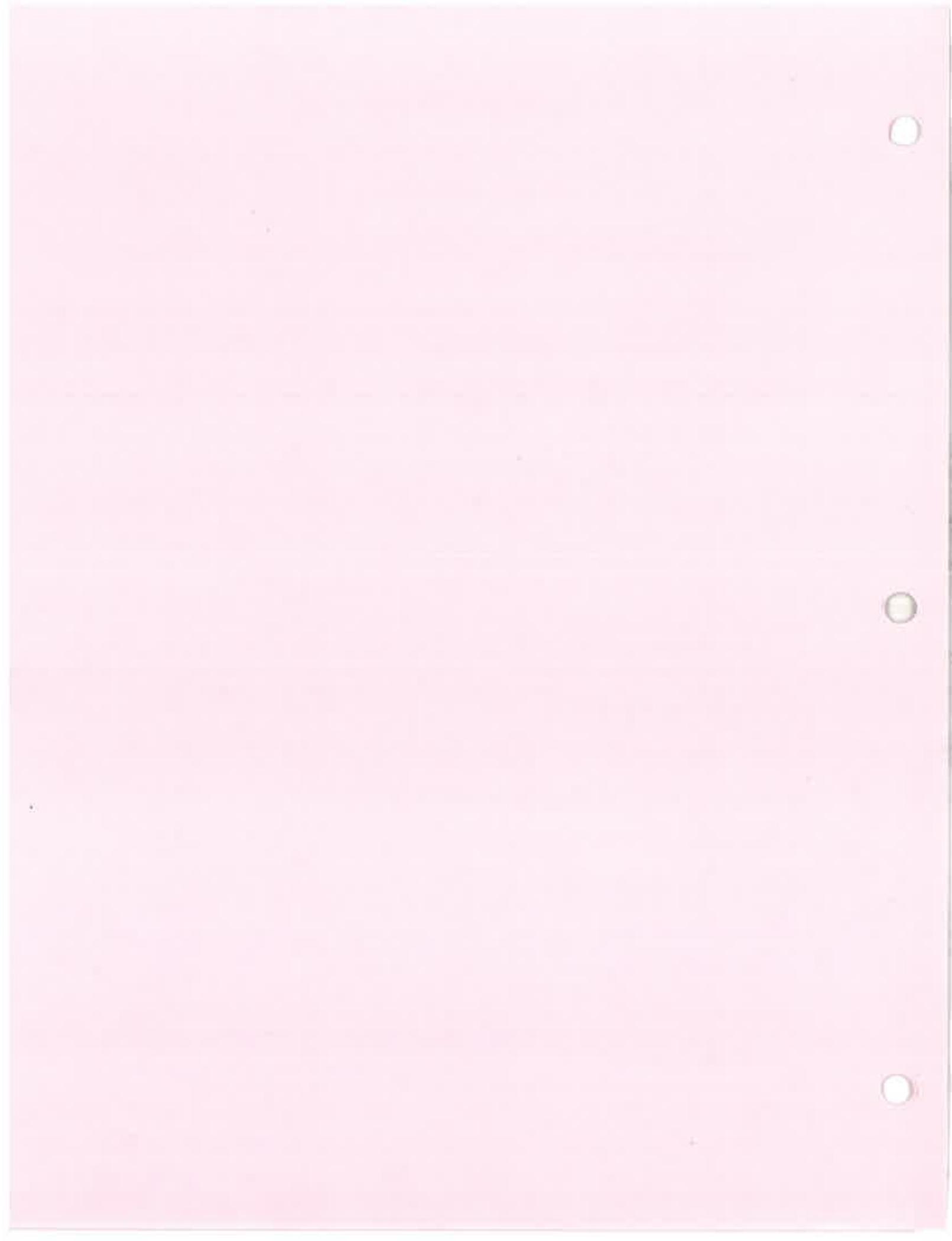


Jonathan F. Reichert

Instructor's Manual

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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

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

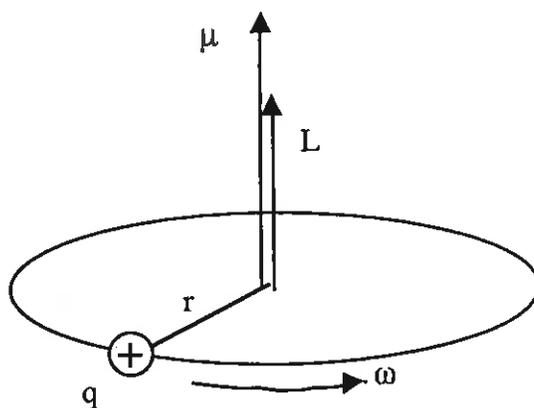


Fig. 1. Magnetic moment and angular momentum vectors for a charged particle rotating at constant angular velocity in a circle of radius r .

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

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

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 $\gamma = q/2m = (1.602 \times 10^{-19} \text{ C})/(1.672 \times 10^{-27} \text{ kg}) = 9.58 \times 10^7 \text{ C/kg}$. The measured value is $\gamma = \bar{\mu} / \bar{L} = 2.675 \times 10^8 \text{ C/kg}$, which is larger than the classically-computed value by a factor of 2.79. 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 \bar{L} and magnetic moment $\bar{\mu}$, both of which point in the same direction along the axis of rotation.

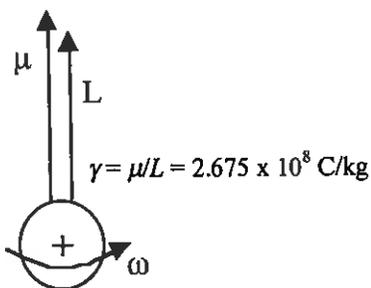


Fig. 2. Classical model of a proton. The magnetic moment and spin angular momentum vectors point in the same direction along the axis of rotation.

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 \bar{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

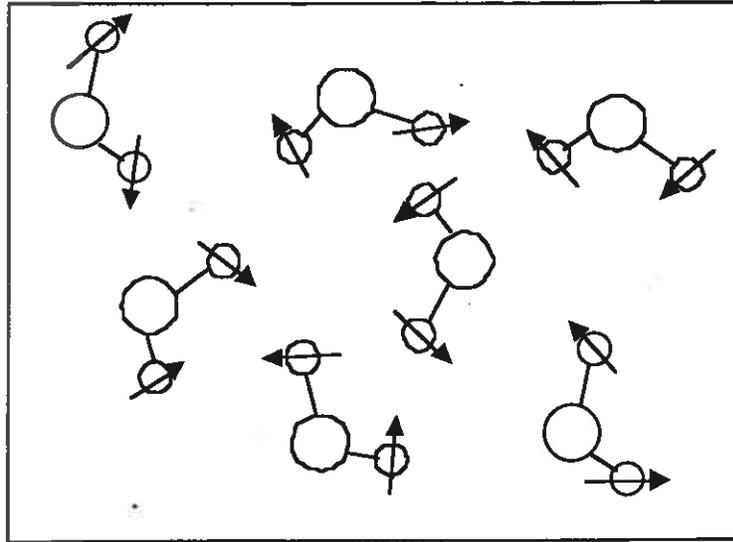


Fig. 3. Schematic representation of molecules of water, H_2O . In the absence of an external magnetic field, the magnetic moment of each proton (hydrogen nucleus) is randomly oriented in space.

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:

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

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.

Actually, a quantum-mechanical correction to the classical expression in Eq. (2) must be made. The corrected result is

$$M_o = \left(\frac{I+1}{I} \right) \frac{N\mu^2 B}{3kT}, \quad (3)$$

where I is the nuclear spin quantum number, equal to $1/2$ for protons. Equation (3) predicts a magnetization proportional to the field B and inversely proportional to the

temperature in kelvins. It is valid so long as $\mu B/kT \ll 1$. 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.

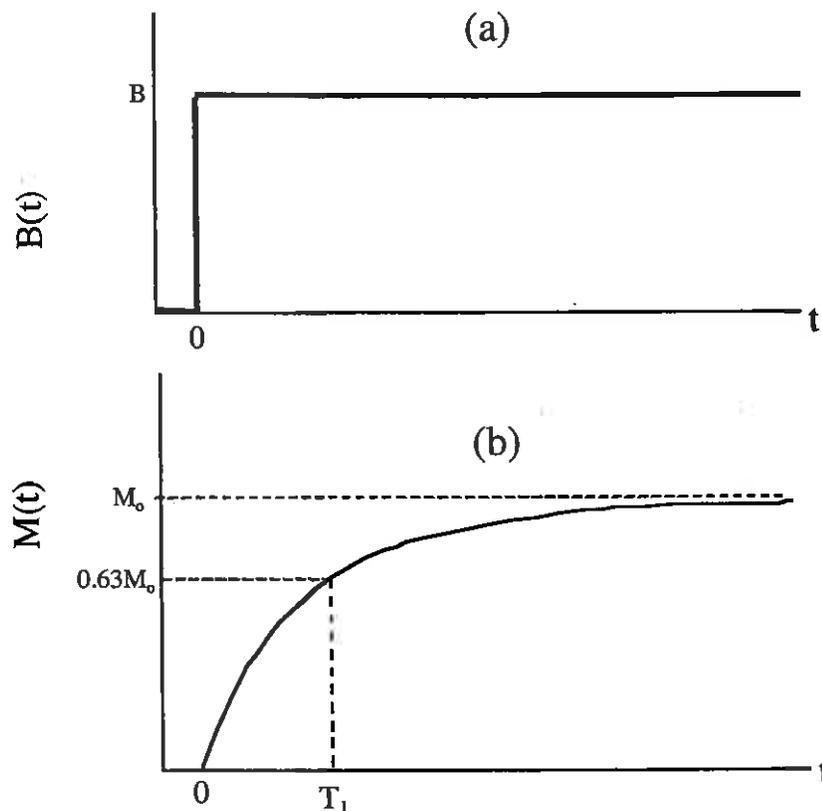


Fig. 4. (a) A constant magnetic field B is switched on at time $t = 0$.
(b) The magnetization grows exponentially with time constant T_1 toward the equilibrium Curie value M_0 .

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

$$M(t) = M_0 \left(1 - e^{-\frac{t}{T_1}} \right), \quad (4)$$

where M_0 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_0 . After two time constants $M(2T_1) = 0.86M_0$. After five time constants $M(5T_1) = 0.99M_0$. 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.

When the field is first turned on, the potential energy of each magnetic moment in the magnetic field is given by

$$U = -\vec{\mu} \cdot \vec{B} = -\mu B \cos \theta .$$

As the proton magnetic moments turn over and line up with the external magnetic field, their magnetic potential energy decreases. Where does the energy go? The answer, at least for water, is that the magnetic potential energy is given up to other degrees of freedom, such as random thermal motion of molecules, within the liquid lattice. Because two neighboring magnetic dipoles exert forces on each other, each proton magnetic moment experiences forces, and in turn exerts forces on, the magnetic dipole moments of protons on the same and on neighboring water molecules. This dipole-dipole interaction provides a mechanism whereby the nuclear spin system is coupled to the lattice and tends to be in thermal equilibrium with it. Where coupling between the spin system and lattice is strong, the spin system gives up its energy to the lattice quickly, and the proton spin-lattice relaxation time is short. Where coupling is weak, spin-lattice relaxation times are long. In chloroform (CHCl_3), for example, proton spin-lattice relaxation times due to magnetic dipole-dipole interactions are very long, on the order of 80 seconds at room temperature. The major reason is that chloroform has only one hydrogen atom per molecule. The nearest hydrogen atoms are relatively far away on neighboring molecules. Each hydrogen nucleus on a chloroform molecule does experience magnetic forces due to the magnetic moments of the three chlorine nuclei on the same molecule, but chlorine nuclei have much smaller magnetic moments than protons, and have correspondingly weaker effects on proton relaxation times. Other factors influence proton relaxation times as well

Larmor Precession

When a proton is placed in an external magnetic field \vec{B} , it experiences a magnetic torque $\vec{\tau} = \vec{\mu} \times \vec{B}$ 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

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

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

After the magnetization has reached its final equilibrium value along the direction of \vec{B} , "What happens to \vec{M} if the direction of \vec{B} is changed?" It is useful to think of \vec{M} as precessing about \vec{B} at angular frequency ω , with its tip tracing out a circle of infinitesimally small radius. If \vec{B} is rotated through angle α very slowly, such that $d\alpha/dt \ll \omega$, the condition for adiabatic passage is satisfied, and \vec{M} rotates with \vec{B} . If, however, \vec{B} is turned quickly such that $d\alpha/dt \gg \omega$, \vec{M} will be left behind pointing in the original direction. The magnetization vector \vec{M} will then precess about the new field \vec{B} at its characteristic angular frequency ω .

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_0) is usually very close to 90° .

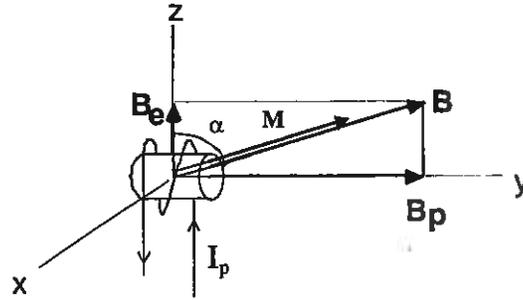


Fig. 5. A schematic representation of the geometry for the earth's-field free precession technique.

When the coil current is reduced to zero, the resultant field \vec{B} shrinks in magnitude and rotates through angle α_0 . 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 $\theta_f \approx \alpha_0 \approx 90^\circ$. 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 $\theta_f \approx \alpha_0 \approx 90^\circ$.

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.

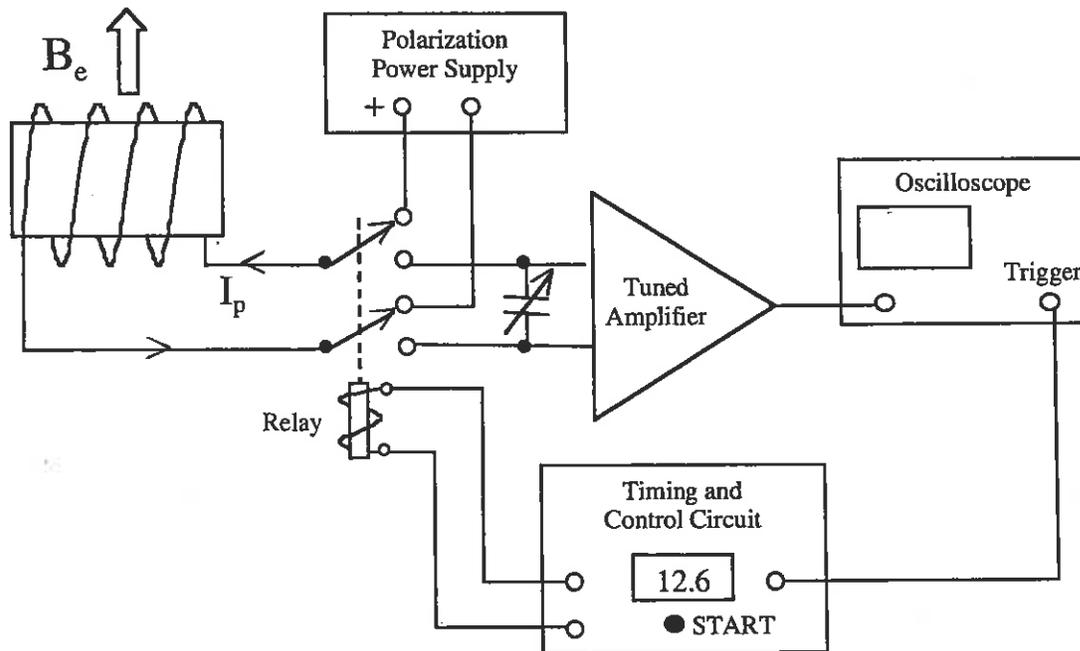


Fig. 6. Simplified block diagram of the apparatus required for the earth's-field free precession technique.

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 \bar{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.

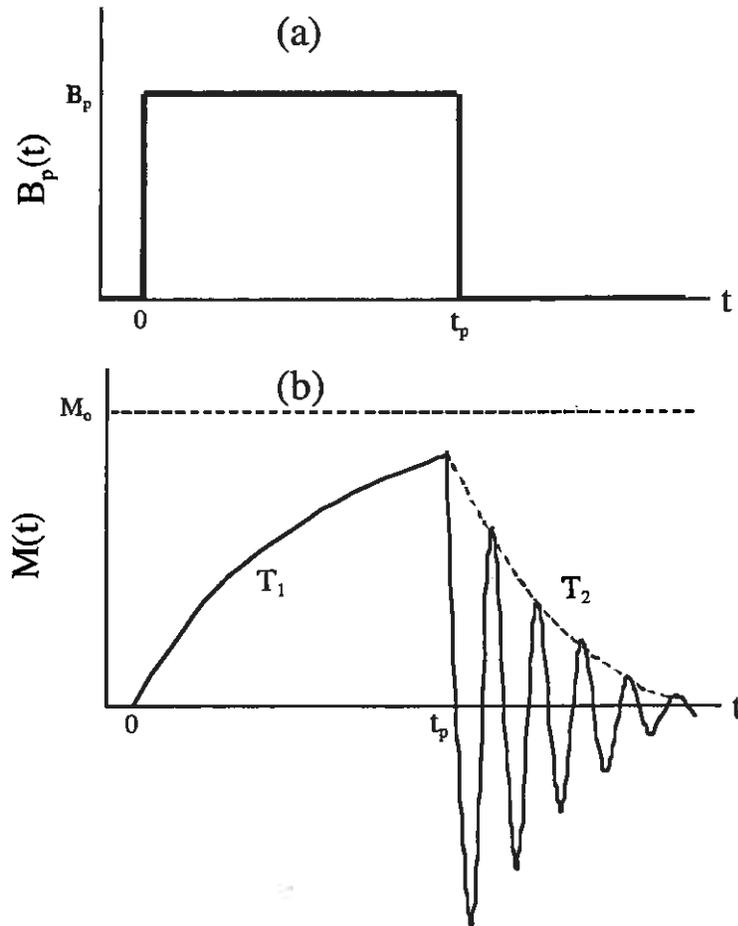


Fig. 7. Variation of polarizing field B_p and magnetization M with time in the earth's-field free precession technique. (a) The polarizing field B_p is switched on at time $t = 0$, and switched off at the end of the polarizing time t_p . (b) When the field is first turned on, magnetization rises exponentially 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. Thus, in the ideal case, the pickup noise is effectively cancelled, and the majority of the noise at the input to the Preamplifier is thermal noise from the combined resistance of the Sample Coil, Bucking Coils, and connecting cable.

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.

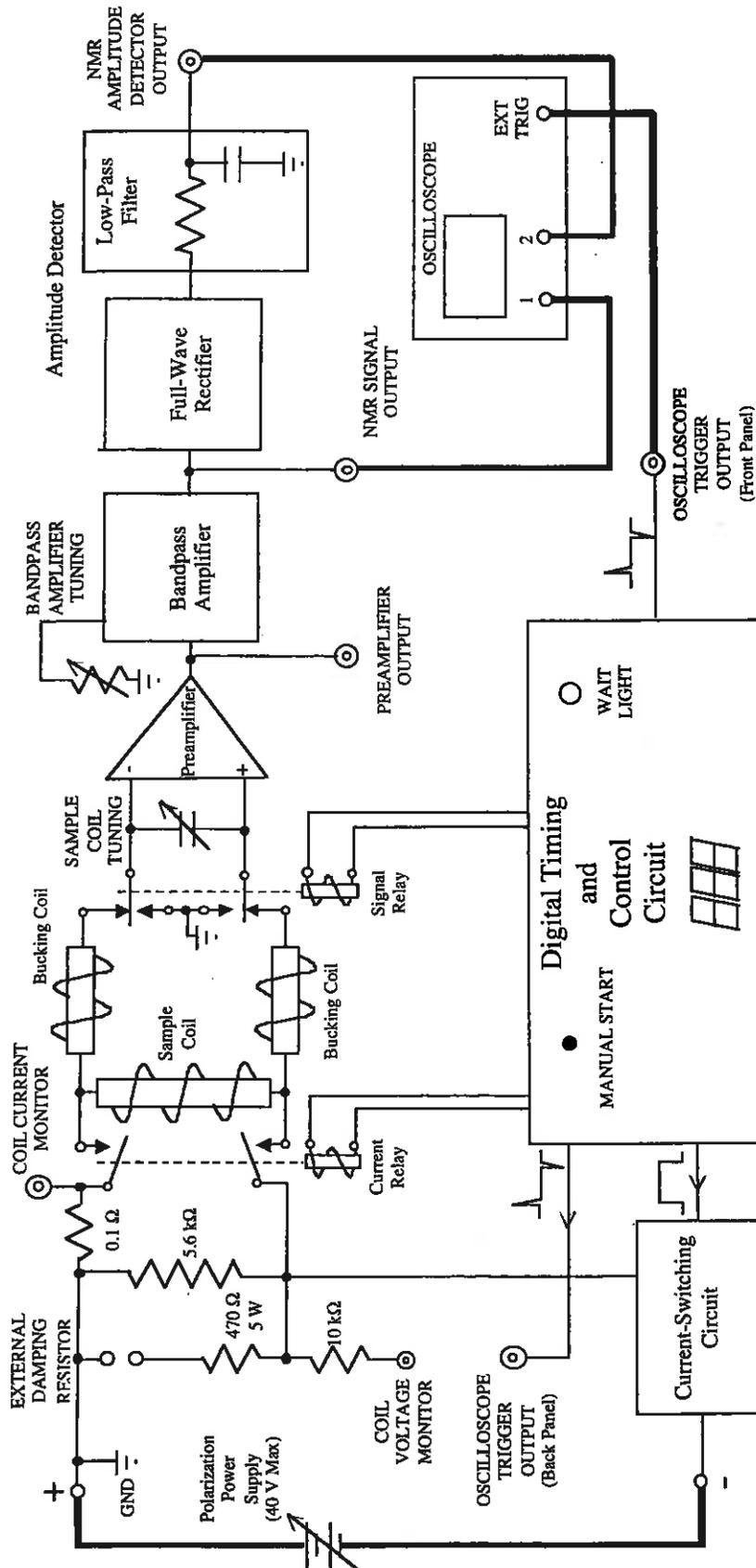


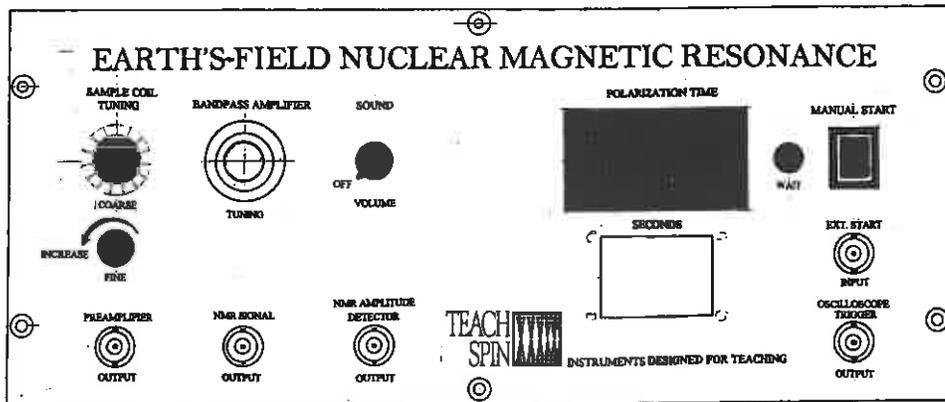
Fig. 8. Block diagram of the Earth's-Field NMR Instrument. External wires and cables are indicated by wide dark lines.

- 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.
- 4) 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.
- 5) 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.
- 6) 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, $\omega_0 = (LC)^{-1/2}$, can be adjusted to coincide with the Larmor precession frequency by rotating the COARSE and FINE switches to vary the capacitance. The 16-position COARSE switch varies the capacitance in approximately equal increments of 5000 pf. The FINE switch (also 16-position) changes the capacitance in increments of 500 pf. Rotating either switch counterclockwise decreases the capacitance and increases the resonant frequency. At 2100 Hz, corresponding to an earth's magnetic field of 0.05 mT, changing the capacitance in increments of 500 pf by rotating the FINE switch one position at a time changes the resonant frequency by about 6 Hz for each step, which is small compared to the approximately 30-Hz bandwidth of the tuned input circuit

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. The gain of the Preamplifier is approximately 1000. 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 bandwidth is approximately 100 Hz, which is large compared to the approximately 30-Hz bandwidth of the tuned input circuit. The center frequency of the Bandpass Amplifier can be adjusted by means of this ten-turn potentiometer. The gain of the Bandpass Amplifier at the center frequency varies somewhat with frequency, but is typically in the range 15-20.

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.

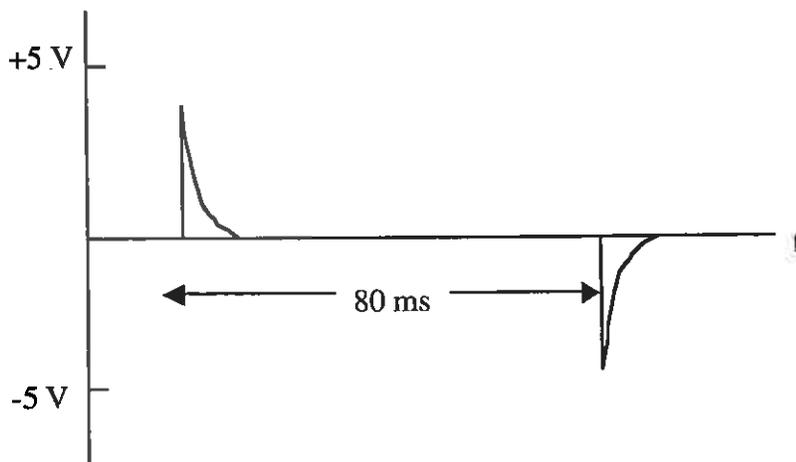
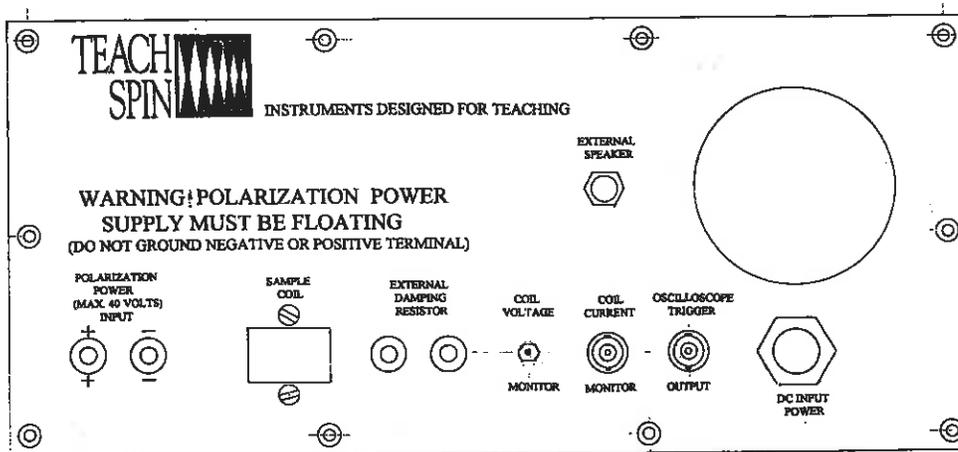


Fig. 9. Pulse sequence at the front-panel OSCILLOSCOPE TRIGGER OUTPUT.

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. A constant current power supply is the best choice. It can maintain the coil current and polarizing field constant as the coil temperature rises, which necessarily occurs during fast repetition of moderate to long polarizing times. 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

When the current is switched off in the Sample Coil, the switching transient is damped by a 5.6-k Ω resistor that is connected, via the Current Relay, in parallel with the Sample Coil (see Fig. 8). This damping resistor causes the switching transient to be slightly underdamped. The damping resistance can be reduced by adding an external damping resistor in parallel with the internal 5.6 k Ω damping resistor. Therefore, by decreasing the size of the external damping resistor the damping can be varied from underdamped, to critically damped, to overdamped.

COIL VOLTAGE MONITOR

This test point allows monitoring the voltage across the Sample Coil. During the time that the current is being switched off in the Sample Coil, the coil voltage rises to approximately 60 volts during the few milliseconds that the coil current is being switched off. Use a 10 \times oscilloscope probe to monitor the coil voltage. **BE CAREFUL NOT TO TOUCH THE TEST POINT WITH YOUR FINGERS.** A 10-k Ω resistor in series with the test point has been included for safety, but you still might feel a mild shock if you happen to touch the test point while the current is being switched off in the coil.

COIL CURRENT MONITOR

This test point allows monitoring the Sample Coil current by monitoring the voltage across an $r = 0.10 \Omega$ resistor connected in series with the coil. While polarizing current is on, the potential at the test point is $V = -I_p r$ with respect to ground. When the coil current is 2.5 A, for example, the potential measured at the test point will be $V = -I_p r = -(2.5 \text{ A})(0.10 \Omega) = -0.25 \text{ V}$.

OSCILLOSCOPE TRIGGER OUTPUT

While using an oscilloscope to monitor COIL VOLTAGE or COIL CURRENT, connect the OSCILLOSCOPE TRIGGER OUTPUT on the back panel to the EXTERNAL TRIGGER input on the oscilloscope. The output of the OSCILLOSCOPE TRIGGER is two narrow pulses similar to those shown in Fig. 10. The leading (positive) pulse occurs at the instant the current is switched on. The trailing (negative) pulse occurs when the current is switched off. The time interval between the pulses is the polarizing time t_p . For monitoring COIL VOLTAGE and/or COIL CURRENT during the entire polarizing time, the oscilloscope should be set to trigger on the leading edge of the positive pulse. In order to monitor COIL VOLTAGE and/or COIL CURRENT during the switching transient that occurs at the end of the polarizing time, set the oscilloscope to trigger on the leading edge of the negative pulse.

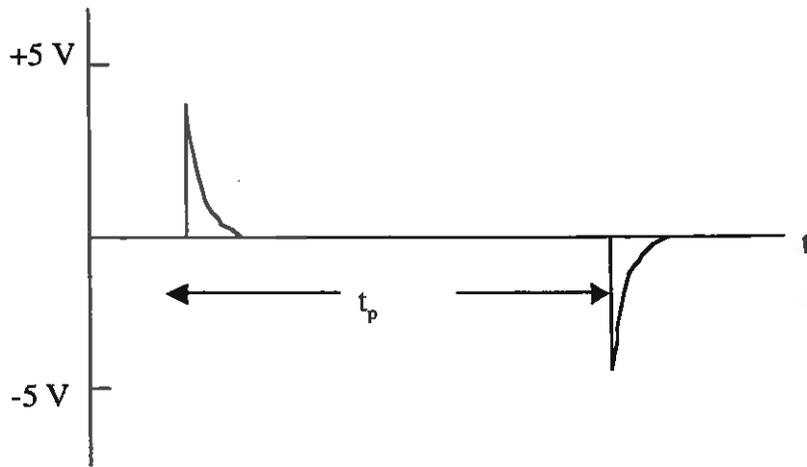


Fig. 10. Pulse sequence at the back-panel OSCILLOSCOPE TRIGGER OUTPUT.

EXTERNAL SPEAKER

This phono-plug socket is provided for connecting an external 8- Ω speaker, in place of the internal speaker, for listening to the free precession signal.

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. Near concrete floors the earth's magnetic field is distorted by steel reinforcing wire or rods. Typical ceilings have metal light fixtures and/or metal grids that hold ceiling tile. It is usually not possible to set the coils on or near a lab stool or table since they are usually fastened together by screws, bolts, and brackets made of magnetic materials. (TeachSpin, Inc., offers a non-magnetic support stand that simplifies the process of searching for the location in the room where the earth's magnetic field is most homogeneous.)

Once you have selected what appears to be a reasonably good location, use a small compass to examine the direction of the earth's magnetic field there and at other places in the room. Noticeable variations in the direction of the earth's field indicate the presence of nearby magnetic materials like iron water pipes, or steel beams that support the ceiling or floor. Such regions must be avoided.

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. Refer to the section on MINIMIZING PICKUP NOISE for more information.

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 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 10.
- 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 5 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 ($\approx +1$ V).

7) Rotate the thumbwheel switches to obtain some convenient polarizing time, say 5.0 s. Press the MANUAL START button. You should hear a faint audible click when the Current and Signal Relays are energized, and the counter should start counting down toward zero. On the count of zero you should hear a faint click when the Current and Signal Relays are de-energized. The oscilloscope should start its sweep almost simultaneously. A few milliseconds after start of the sweep, the Signal Relay will make contact with the coils, and you should see a switching transient on the scope similar to the one shown in Fig. 11. The transient dies away within about 50 ms.

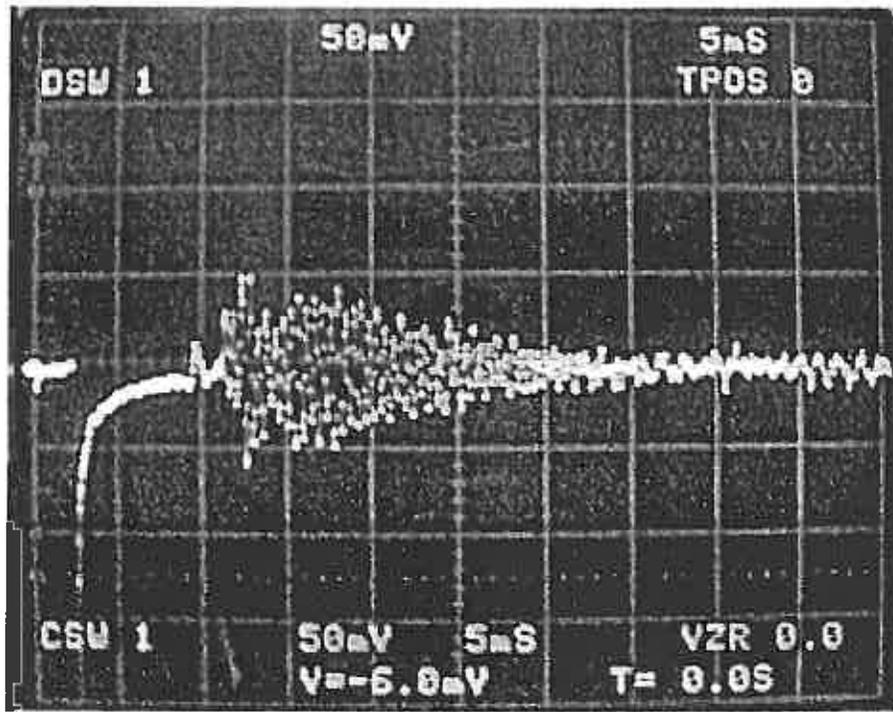


Fig. 11. Switching transient at the PREAMPLIFIER OUTPUT. The oscilloscope vertical sensitivity and sweep speed are set at 50 mV/Div and 5 ms/Div. Oscilloscope COUPLING is set on AC, which blocks any dc offset.

- 8) On the count of zero the WAIT light comes on. After a delay of 5.0 s on the count of zero, the counter counts from zero back up to the preset polarizing time. When that has occurred, the WAIT light will go off, and the instrument will be ready for another cycle.
- 9) Increase the oscilloscope sweep speed to 2 ms/Div. Cycle the instrument again, but this time set the oscilloscope to trigger on -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.

If everything seems to be working properly, you are ready to tune the instrument to detect the free precession signal.

10) 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.

11) 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.

12) 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.

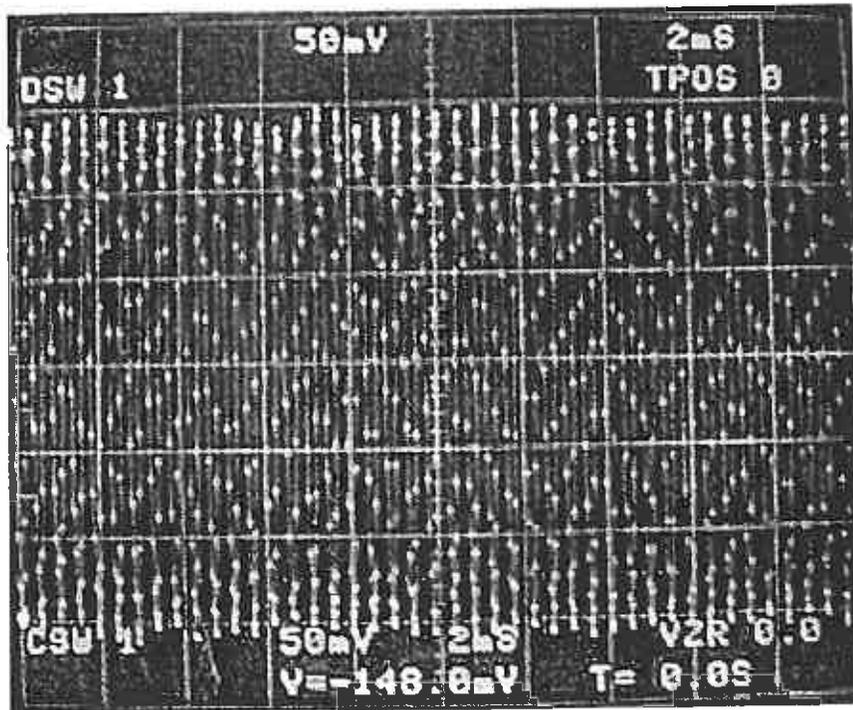


Fig. 12. Free precession signal at the PREAMPLIFIER OUTPUT. The oscilloscope is set at 50 mV/Div and 2 ms/Div. The sample frequency is 100 samples/Div for all oscilloscope waveforms shown in this manual.

Adjusting the Center (Resonant) Frequency of the Bandpass Amplifier

At 2100 Hz the Bandpass Amplifier has a gain of about 20 and a bandwidth of approximately 100 Hz, which is large compared to the approximately 30-Hz bandwidth of the tuned input circuit. 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:

- 13) 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 and 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.

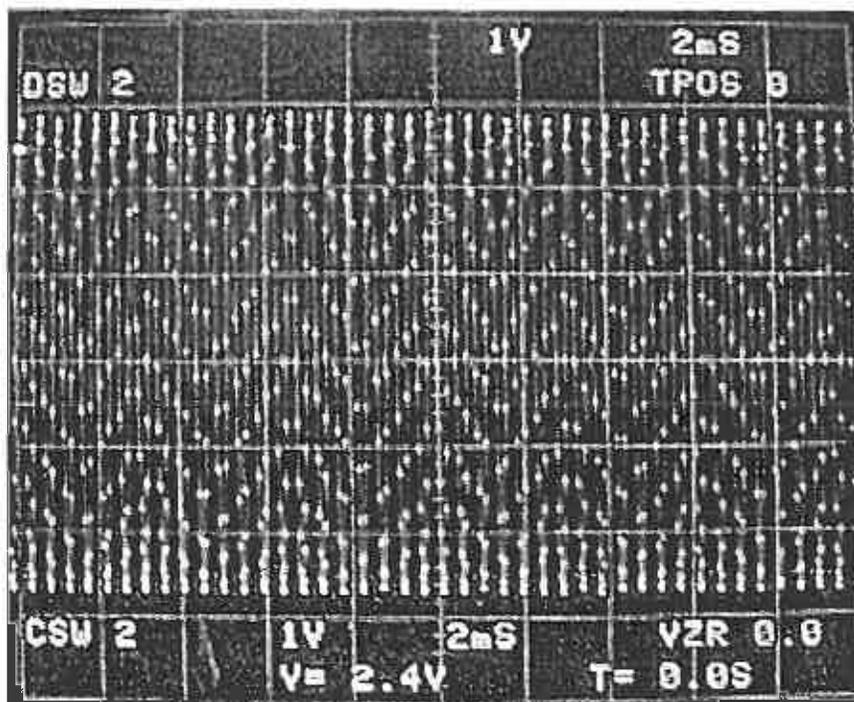


Fig. 13. Free precession signal at the NMR SIGNAL OUTPUT. The oscilloscope is set at 1 Volt/Div and 2 ms/Div.

14) After the Bandpass Amplifier has been properly adjusted for maximum output signal, as in Fig. 13, the Bandpass Amplifier input and output signals will be 180° out of phase. This actually provides the most sensitive test for proper tuning. To check the phase difference, you need to be able to see individual cycles of the free precession signal. Increase the oscilloscope sweep speed to 0.2 ms = 200 μs per division. As you cycle the instrument, adjust the BANDPASS AMPLIFIER TUNING potentiometer in fairly fine (1/4 turn or less) increments until the PREAMPLIFIER OUTPUT and NMR SIGNAL OUTPUT are 180° out of phase as shown in Fig. 14. You should observe that when the phase difference is 180°, the NMR SIGNAL OUTPUT is a maximum.

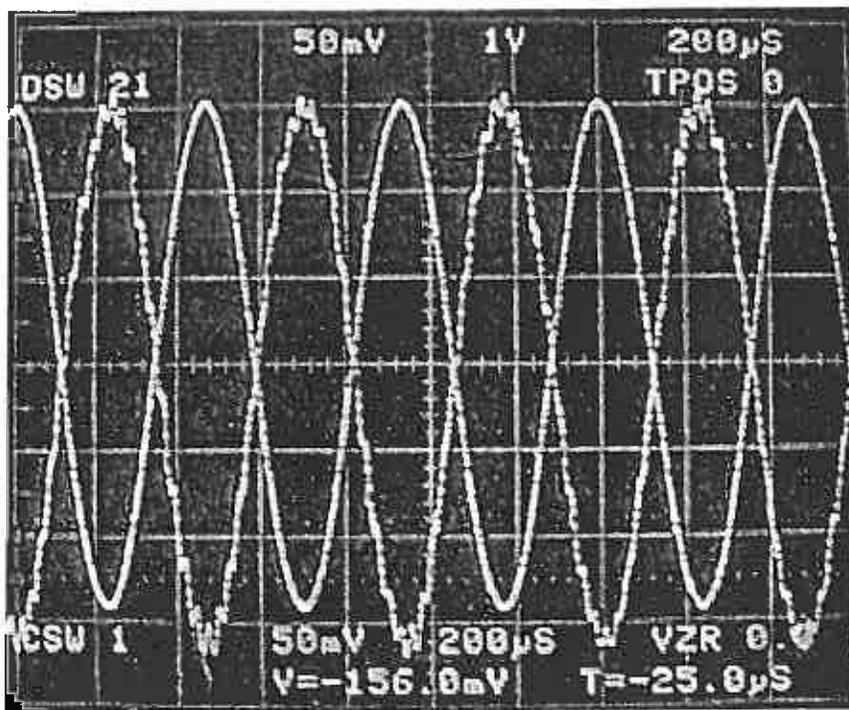


Fig. 14. When the BANDPASS AMPLIFIER is properly tuned, the free precession signals at the PREAMPLIFIER OUTPUT (oscilloscope channel 1, 50 mV/Div) and NMR SIGNAL OUTPUT (oscilloscope channel 2, 1 V/Div) are 180° out of phase. The oscilloscope sweep speed is 200 µs/Div. Note the approximately 20× larger signal, and improved signal-to-noise ratio, at the NMR SIGNAL OUTPUT.

Figure 15 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. 15 exhibits the Gaussian-shaped decay that is commonly observed where the earth's field indoors is relatively inhomogeneous. Refer to Fig. 17 on page 30 for an example of the longer decays that are observed in more homogeneous earth's magnetic fields.

By carefully positioning the coils, it is usually possible to find a location indoors where T_2^* for water at room temperature is in the range 500-800 ms, which is sufficiently long that the decay of the free precession signal appears to be—at least approximately—exponential. The signal in Fig. 16 was acquired with the oscilloscope sweep speed set at a relatively slow 200 ms/Div.

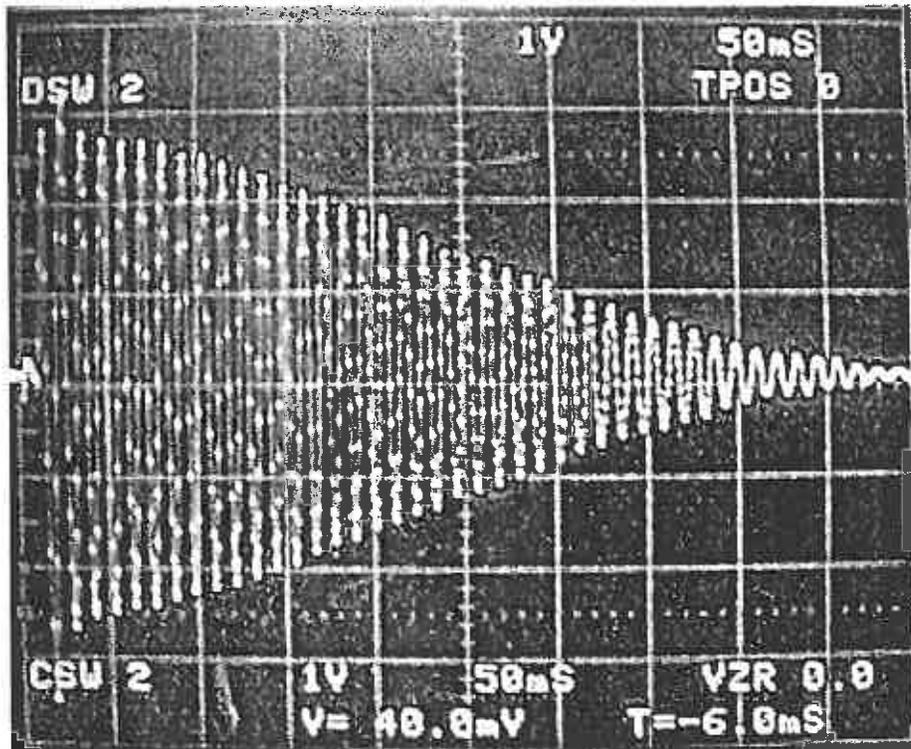


Fig. 15. Water free precession signal at the NMR SIGNAL OUTPUT. Note the Gaussian-shaped decay caused by a relatively inhomogeneous earth's magnetic field. The oscilloscope is set at 1 V/Div and 50 ms/Div.

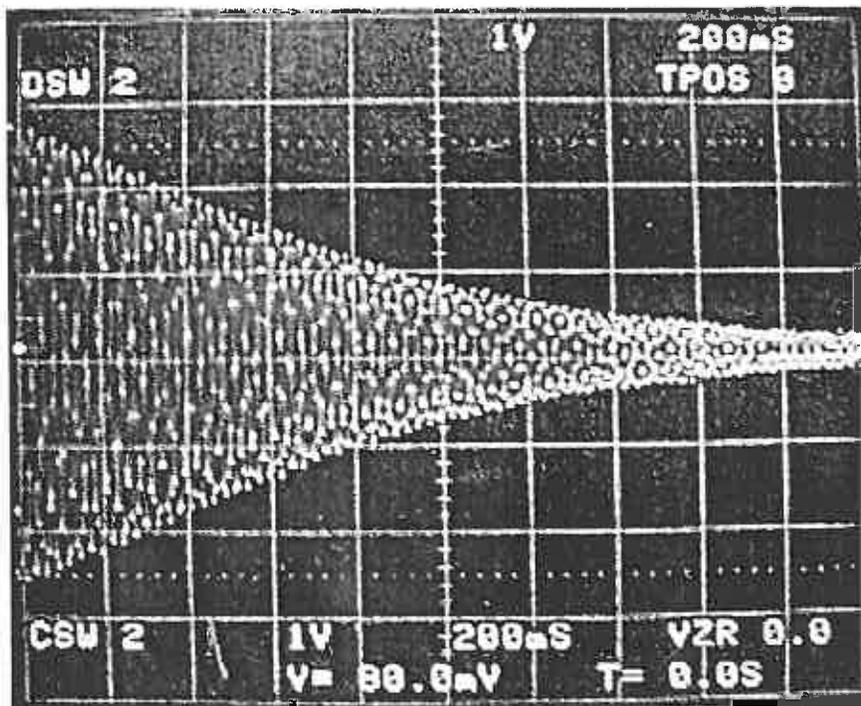


Fig. 16. Water free precession signal at the NMR SIGNAL OUTPUT. The oscilloscope is set at 1 V/Div and 200 ms/Div. This signal was obtained with the coils positioned indoors in a region where the earth's magnetic field was relatively homogeneous.

It is difficult to predict with certainty exactly where in the room the earth's magnetic field is most homogeneous. The signal in Fig. 15, for example, was taken with the coils positioned near the open center of an 18 x 22-foot laboratory. The much longer decay in Fig. 16 was obtained with the coils moved off to one side, only a few feet away from a long wooden lab bench permanently attached to the wall! Moving the coils only 6 inches (15 cm) or so can sometimes make a big difference in the observed value of T_2^* .

MINIMIZING PICKUP NOISE

Reorienting the Coils

As mentioned briefly in the section on INITIAL SETUP, the coils should be placed in an open area as far as possible away from sources of radiated electrical noise. Fluorescent lamps, switching-mode power supplies (like those in most desktop computers), electrical motors, electric fans and blowers, power panels, etc., are all examples of potentially strong sources of radiated electrical noise. Incandescent lights in the same room do not usually pose a problem unless they are controlled by dimmer switches. Fluorescent lights in the same room as the coils should be turned off if at all possible.

Perform the following test: With the instrument on and tuned to the free precession frequency, use an ac voltmeter to monitor the rms pickup noise at the NMR SIGNAL OUTPUT as you slowly rotate the base of the Sample and Bucking Coils 90°, first clockwise and then counterclockwise, away from east-west. Find the orientation where pickup noise is a minimum.

Rotating the coil axes away from east-west may have the undesirable effect of reducing the amplitude of the free precession signal. As described earlier in the section on *The Earth's-Field Free Precession Technique*, rotating the Sample Coil axis away from east-west will make the angle α_0 between the polarizing field and the earth's field less than 90°. Assuming a perfect sudden cutoff of the polarizing field, the ensuing free precession signal will have amplitude proportional to $M \sin \alpha_0$. Thus, reducing α_0 from 90 to 60° will reduce the signal by 13%. However, this relatively small drop in signal may be more than compensated for by an even greater reduction in pickup noise. Actually, because of the relatively large dip angle of the earth's magnetic field at latitudes far from the equator, it may be possible to rotate the coil axis by as much as $\pm 90^\circ$ away

from east-west without seeing an unacceptably large drop in amplitude of the free precession signal. If that is the case, the optimum orientation will likely be the one that minimizes pickup noise. Otherwise, it will be necessary to choose an orientation that is a compromise between maximizing the signal and reducing the pickup noise.

Aligning the Axes of the Coils

Another variable that affects pickup noise is the efficiency of the Bucking Coils in canceling pickup noise in the Sample Coil. The Bucking Coils have a much larger surface area than the Sample Coil, but the Bucking Coils have many fewer turns. The Sample and Bucking Coils are designed to have the same number of area-turns. Therefore, a distant noise source will produce the same total magnetic flux Φ through the two different sets of coils. From Faraday's law, the noise source will induce the same emf $d\Phi/dt$ in each. Since the Sample and Bucking Coils are electrically connected in series, but are wound in opposite directions, the emf's in the coils due to pickup noise tend to cancel.

In addition to surface area and number of turns, another important variable that affects the efficiency of the Bucking Coils is the orientation of the axis of the Sample Coil relative to the axis of the Bucking Coils. In order for pickup noise to be a minimum, those two axes should coincide. Otherwise, the pickup noise flux Φ through the Sample and Bucking Coils may not be the same, even though they have the same number of area-turns. The Bucking Coil is permanently fixed to the base, but the Sample Coil axis can be rotated in a horizontal plane relative to the Bucking Coils by loosening the nylon screws, located under the base, that attach the base to the Sample Coil supports. In addition, the axis of the Sample Coil can tilted up or down by means of nylon leveling screws in one of the Sample Coil supports.

Loosen the screws, and monitor the pickup noise as you change the orientation of the Sample Coil axis relative to the Bucking Coil. You will find that the pickup noise is very sensitive to even small changes in the orientation of the axis of the Sample Coil. Once you have found the position that minimizes pickup noise, retighten the nylon screws that hold the Sample Coil firmly in place. Then try rotating the base of the Sample and Bucking Coils to make sure the orientation that gives minimum noise has not changed.

For a 125-ml water sample at room temperature, and a polarizing current of 3.0 A, signal-to-noise ratios as high as 700:1 (peak signal to average rms noise) are obtainable under ideal conditions, typically during nights and weekends when electrical pickup noise in the building is diminished. Signal-to-noise ratios of 500:1 are common during normal weekday work hours. Even during worst-case conditions, the signal-to-noise ratio should be 100:1 or more.

NMR AMPLITUDE DETECTOR

Figure 17 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. 17 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. 17 are shown in Fig. 18, but there the sweep speed has been increased from 50 ms/Div to 20 ms/Div. Note in Fig. 18 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.

A second advantage of using the Amplitude Detector has to do with the fact that the free precession signal is rectified and passed through a low-pass filter before being sent to the NMR AMPLITUDE DETECTOR OUTPUT. The time constant of the filter is about 5 ms. Thus, any rapid fluctuations in signal amplitude, caused by thermal or pickup noise, that are less than a few milliseconds wide are effectively blocked by the low-pass filter. This improves signal-to-noise.

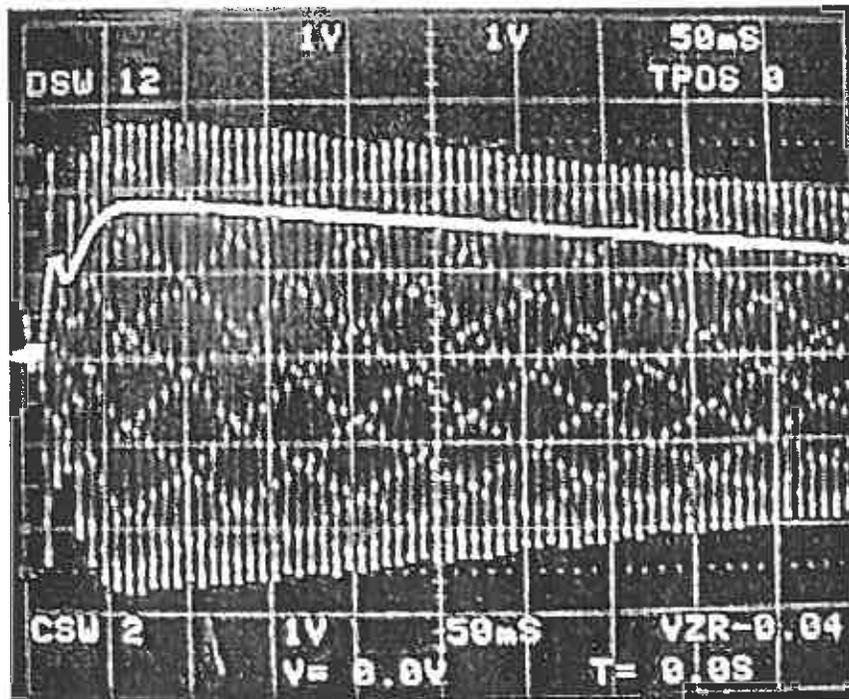


Fig. 17. Water NMR SIGNAL shown along with the output of the NMR AMPLITUDE DETECTOR. Both oscilloscope channels are set at 1V/Div and 50 ms/Div.

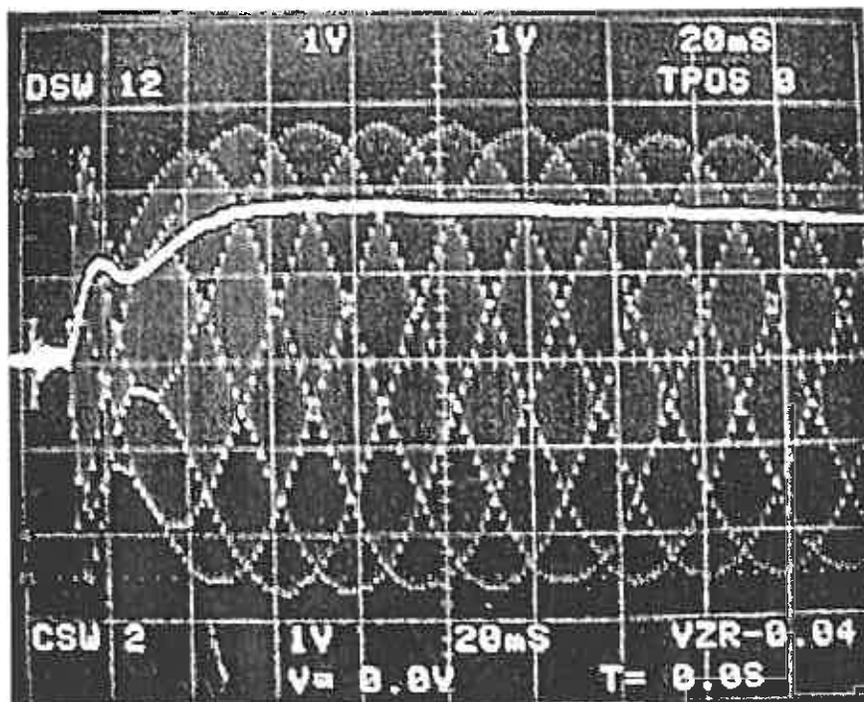


Fig. 18. Same signals as in Fig. 17, but with the oscilloscope sweep speed increased to 20 ms/Div. Note the appearance of "beats" in the NMR SIGNAL OUTPUT caused by problems associated with aliasing.

A third advantage of the Amplitude Detector has to do with its use in signal averaging to improve signal-to-noise. That topic is covered in the next section.

SIGNAL AVERAGING

For small samples (and for even large samples that have short T_2 s) the signal-to-noise ratio can be very small. Most digital oscilloscopes allow successive traces to be averaged, which can greatly improve signal-to-noise. Averaging N signals will improve the signal-to-noise ratio by a factor of $N^{1/2}$, which is about 3.2 for $N = 10$.

The effect of averaging, for a small 10-ml water sample, is shown in Figs. 19 and 20. Figure 19 shows the signals at the NMR SIGNAL OUTPUT and NMR AMPLITUDE DETECTOR OUTPUT after a single trace. The signals are relatively small, so the oscilloscope vertical sensitivity has been increased to 100 mV/Div for both channels. As discussed in the previous section, the output of the NMR AMPLITUDE DETECTOR is less susceptible to noise due to the approximately 5-ms time constant of the RC circuit used to filter the full-wave-rectified NMR SIGNAL.

Figure 20 shows the waveforms displayed on the oscilloscope after averaging 10 traces. As expected, the signal-to-noise ratio at the output of the NMR AMPLITUDE DETECTOR is greatly improved. However, the averaged waveform at the NMR SIGNAL OUTPUT is greatly diminished in amplitude. This occurs because there is slight jitter in the digital timing and analog oscilloscope trigger circuits, which translates, at low oscilloscope sweep speeds, into significant jitter in the phase of successive traces. For a very large number of traces, the signal at the NMR SIGNAL OUTPUT is effectively averaged to zero. The problem of phase jitter can be overcome by increasing the oscilloscope sweep speed to 500 μ s/Div or faster. At these higher sweep speeds, a ± 20 μ s jitter in the timing or trigger circuits translates into negligibly small jitter in the phase of signals acquired in successive traces. In most cases, however, the envelope of the free precession signal is all that is of interest. In those situations, the averaged output from the NMR AMPLITUDE DETECTOR is all that is required.

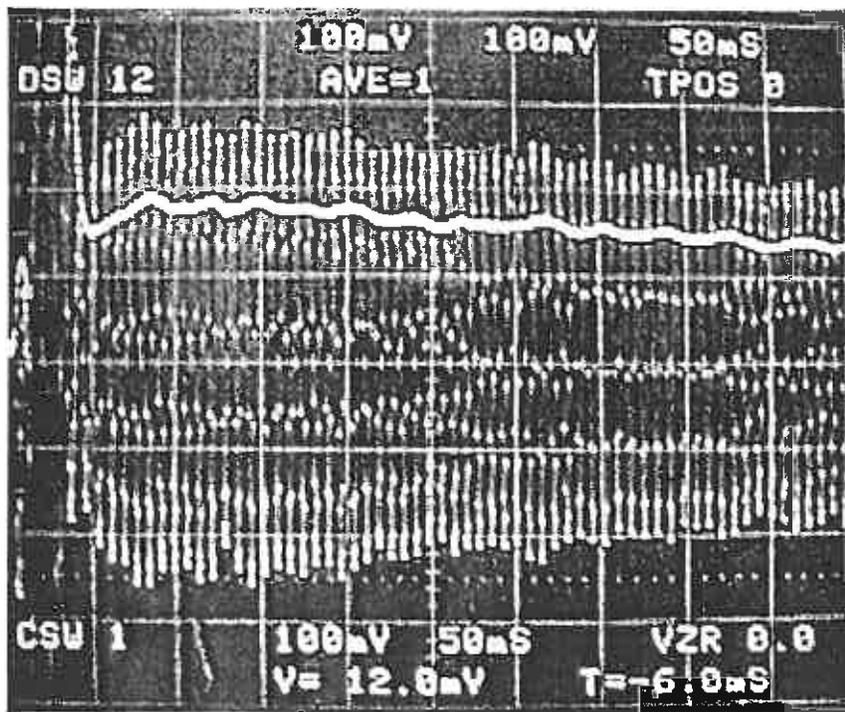


Fig. 19. Signals at the NMR SIGNAL OUTPUT and NMR AMPLITUDE DETECTOR OUTPUT for a 10-ml sample of water. The oscilloscope is set at 100 mV/Div and 50 ms/Div.

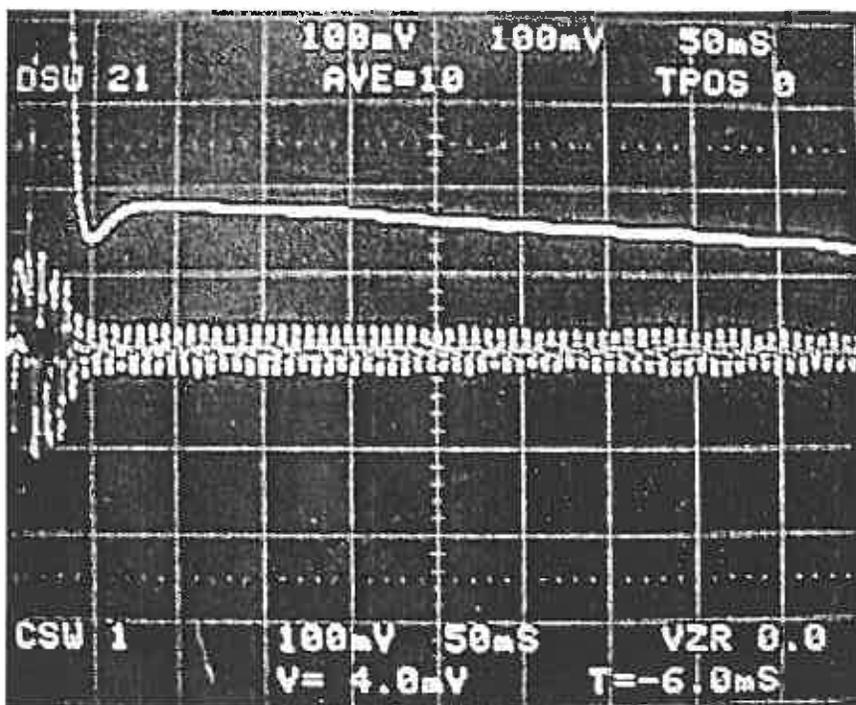


Fig. 20. Same sample as in Fig. 19, but this figure shows the effect of using the oscilloscope to average 10 traces.

MONITORING COIL VOLTAGE AND COIL CURRENT

Referring to the block diagram in Fig. 8, the Digital Timing and Control Circuit first activates the Signal Relay, which disconnects the coils from the Preamplifier. Next, the Control Circuit activates the Current Relay, which connects the Sample Coil to the Polarization Power Supply and Current-Switching Circuit. The Control Circuit then sends a TTL-level pulse, having width equal to the polarizing time t_p , to the Current-Switching Circuit. The same pulse is also differentiated by the Control Circuit before being sent to the OSCILLOSCOPE TRIGGER OUTPUT connector on the back panel. These two pulses are shown, not to scale, in Figs. 21(a) and 21(b). The positive edge of the positive pulse in Fig. 21(b) occurs at the instant coil current is switched on at the beginning of the polarizing time. It is used for triggering the oscilloscope when monitoring the coil current and coil voltage waveforms described later.

The TTL pulse in Fig. 21(a) causes the Current-Switching Circuit to switch into its low impedance conducting state for the duration of the polarizing time. The COIL CURRENT MONITOR test point allows an oscilloscope to be used for monitoring current through the Sample Coil by monitoring the voltage across an $r = 0.10 \Omega$ resistor in series with the coil. While polarizing current is on, the potential at the test point is $V = -I_p r$ with respect to ground. By using the channel invert feature, most oscilloscopes can be set to display $V = +I_p r$. The important features of the $V = +I_p r$ waveform are shown in Fig. 21(c). The polarizing current I_p rises exponentially toward its steady-state value with time constant $L/R \approx 6$ ms. When the coil current reaches its steady-state value, say 3.0 A, the magnitude of the voltage measured at the test point is

$$\underline{V = +I_p r = +(3.0 \text{ A})(0.10 \Omega) = +0.30 \text{ V.}}$$

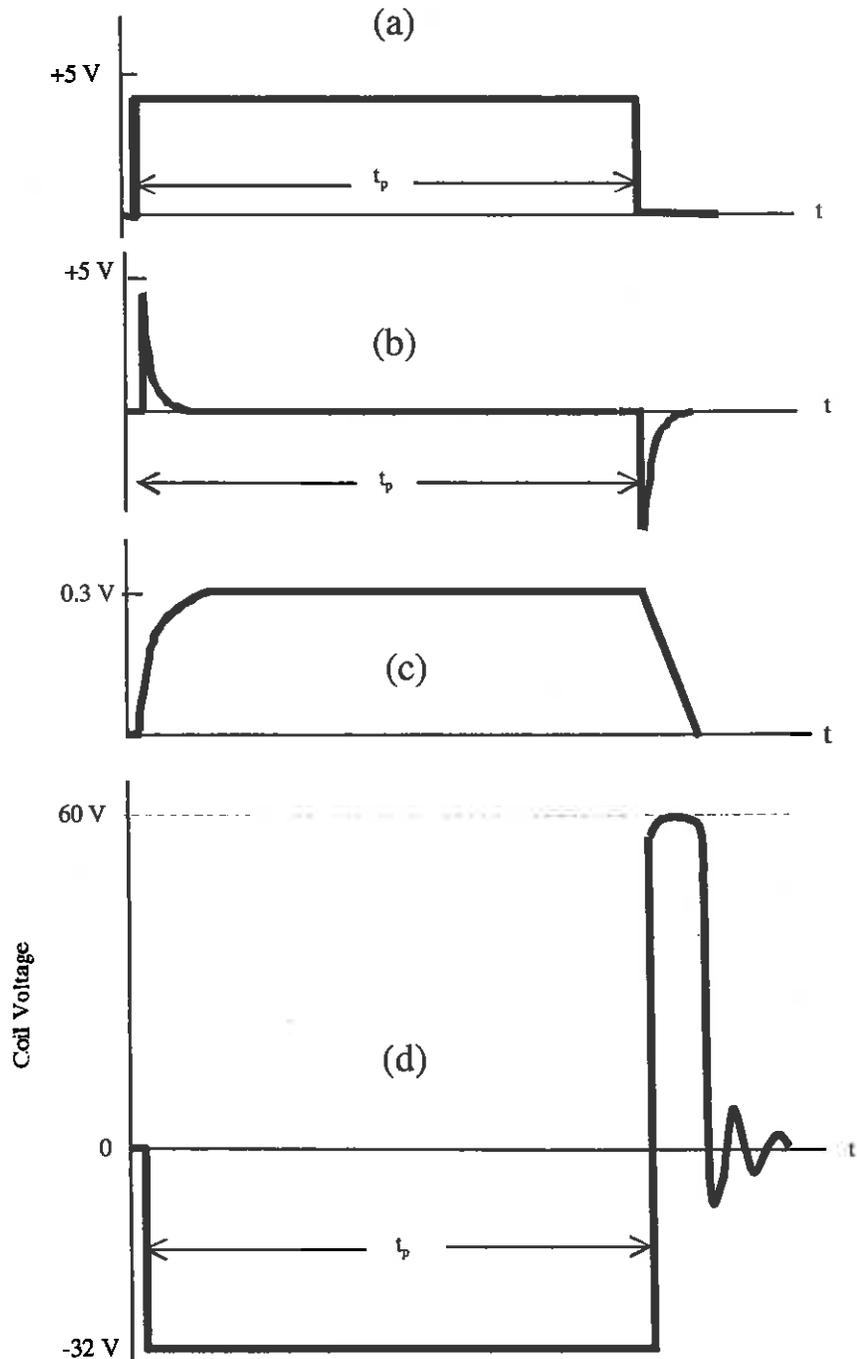


Fig. 21. Diagrams, not to scale, showing the voltage waveforms at various points in the block diagram of Fig. 8. (a) The TTL pulse that is sent from the Control Circuit to the Current-Switching Circuit. The width of the pulse is the polarizing time t_p . (b) The same pulse is differentiated by the Control Circuit before being sent to the OSCILLOSCOPE TRIGGER OUTPUT on the back panel. (c) The waveform viewed on an oscilloscope connected to the COIL CURRENT MONITOR. (d) The waveform viewed on an oscilloscope connected to the COIL VOLTAGE MONITOR.

The voltage across the Sample Coil can be monitored by connecting an oscilloscope to the COIL VOLTAGE MONITOR test point on the back panel. The principal features of the coil voltage waveform are shown in Fig. 21(d). When the Current-Switching Circuit switches into its low impedance conducting state at the beginning of the polarizing time, almost all of the Polarizing Power Supply voltage, assumed here to be 34 V, appears across the Sample Coil, with only 1-2 volts across the Current-Switching Circuit, and much smaller voltages across the Current Relay contacts and 0.10-Ω series resistor used for monitoring current in the coil. Therefore, as shown in Fig. 21(d), the potential at the COIL VOLTAGE MONITOR test point drops to about -32 V with respect to ground for the duration of the polarizing time.

When the voltage of the control pulse in Fig. 21(a) drops back to zero at the end of the polarizing time, the Current-Switching Circuit switches back into its high impedance non-conducting state. The polarizing current in the Sample Coil starts dropping quickly toward zero, and the Sample Coil voltage $V_L = -L \, dI_p/dt$ increases rapidly. A zener diode, not shown, that is part of the Current-Switching Circuit in Fig. 8 limits the maximum coil voltage to approximately 60 V. During the time the coil voltage is held constant by the zener diode, the polarizing current I_p in the coil drops at a constant rate

$$\underline{dI_p/dt = -V_L/L .} \quad (5)$$

Most of the energy in the magnetic field of the Sample Coil is dissipated as heat in the zener diode. The linear cutoff of the current is shown, not to scale, in Fig. 21(c). The minimum polarizing time t_p is 100 ms; the cutoff time is less than 4 ms. Actually, the cutoff is not exactly linear, primarily due to voltage drops across the resistances of the Sample Coil and connecting cable, which were ignored in obtaining Eq. (5).

The ringing transient shown in the coil voltage waveform in Fig. 21(d) occurs after the zener diode stops conducting, which occurs after the coil current has dropped below a few milliamperes. The ringing frequency depends on the inductance of the Sample Coil, and the stray capacitance of the Sample Coil and connecting cable. The transient is damped by a 5.6-kΩ resistor that is connected in parallel with the Sample Coil while the Signal Relay is closed during polarizing and current-switching times. This resistor causes the switching transient to be slightly underdamped. It is normally not necessary to change the damping. But the damping can be increased, if desired, by

connecting an external resistor between the EXTERNAL DAMPING RESISTOR banana plug sockets on the back panel. Referring to the block diagram in Fig. 8, the external damping resistor is connected in series with a fixed 470- Ω resistor inside the instrument, and the combination is in parallel with the internal damping resistor of 5.6 k Ω . This arrangement prevents shorting the EXTERNAL DAMPING RESISTOR sockets from short-circuiting the Sample Coil. **USE CAUTION WHEN SELECTING AND CONNECTING EXTERNAL DAMPING RESISTORS. DEPENDING ON THE SIZE OF THE RESISTOR, THE VOLTAGE ACROSS THE EXTERNAL DAMPING RESISTOR DURING CUTOFF CAN GO AS HIGH AS 60 V, AND THE CONTINUOUS POWER DISSIPATED DURING POLARIZING CAN BE AS HIGH AS 1 WATT.** A detailed discussion of the underdamped ringing transient, and the effect it has on the dynamical motion of the magnetization vector during cutoff, may be found in Ref. 1.

REFERENCES

¹B. F. Melton and V. L. Pollak, *Optimizing Sudden Passage in the Earth's-Field NMR Technique*, Journal of Magnetic Resonance, Series A, **122**, 42 - 49 (1996).

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.

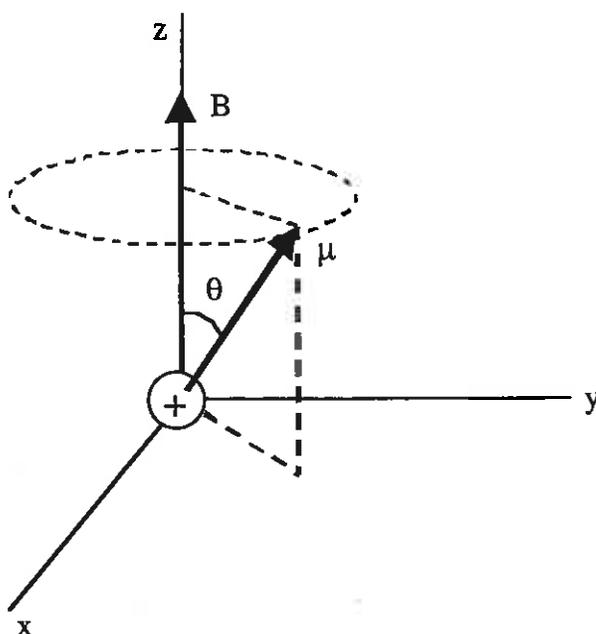


Fig. A1. A positively charged nucleus with its magnetic moment $\vec{\mu}$ oriented at angle θ with respect to a magnetic field \vec{B} pointing along the $+z$ axis. The angular momentum vector \vec{L} , not shown, points in the same direction as $\vec{\mu}$.

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

$$\vec{\tau} = \frac{d\vec{L}}{dt} \quad (\text{A1})$$

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

$$\vec{\tau} = \vec{\mu} \times \vec{B} \quad (\text{A2})$$

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

$$\frac{d\vec{\mu}}{dt} = \gamma \vec{\mu} \times \vec{B} \quad (\text{A3})$$

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:

$$\frac{d\mu_x}{dt} = \gamma B \mu_y, \quad (\text{A4})$$

$$\frac{d\mu_y}{dt} = -\gamma B \mu_x, \quad (\text{A5})$$

and

$$\frac{d\mu_z}{dt} = 0. \quad (\text{A6})$$

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

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

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

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

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

$$\mu_x(t) + i\mu_y(t) = (\mu_x(0) + i\mu_y(0))(\cos \gamma B t - i \sin \gamma B t).$$

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

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

and

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

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 1A shows a 2200 Hz sinusoidal waveform and, for comparison, the sampled approximation. The samples are shown as

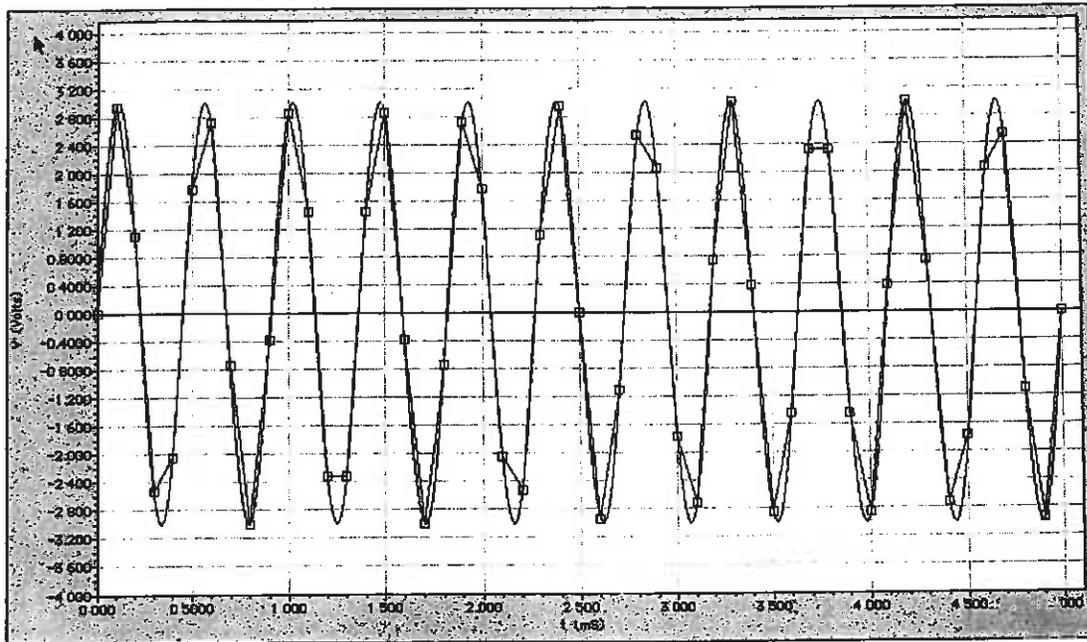


Fig. B1. The rough approximation that results when a 2.2 kHz sinusoidal waveform is sampled at 10 kHz

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.

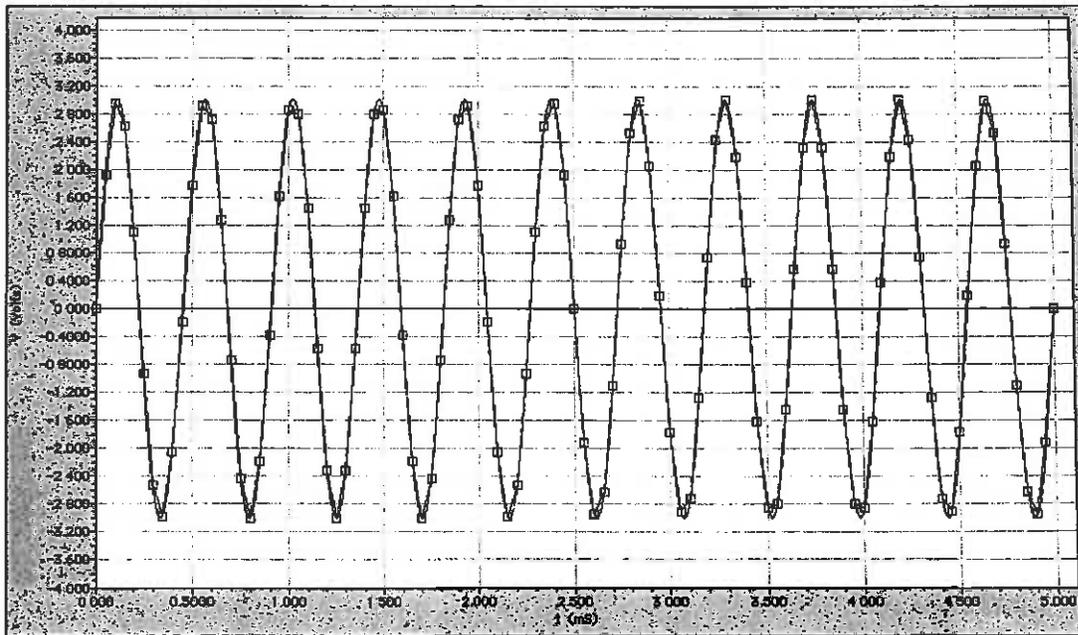


Fig. B2. The relatively smooth waveform that results when a 2.2 kHz sinusoidal waveform sampled at 20 kHz

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

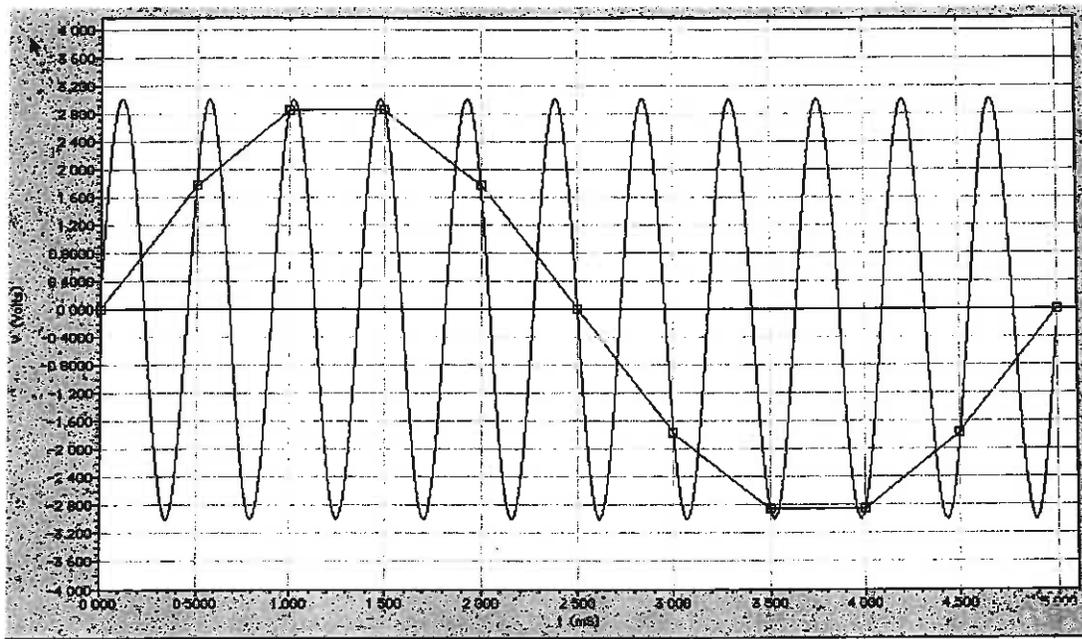


Fig. B3. Missing cycles that result when a 2.2 kHz sinusoidal waveform is sampled at 2 kHz, which does not meet the Nyquist criterion.

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. 18 on p. 30.)

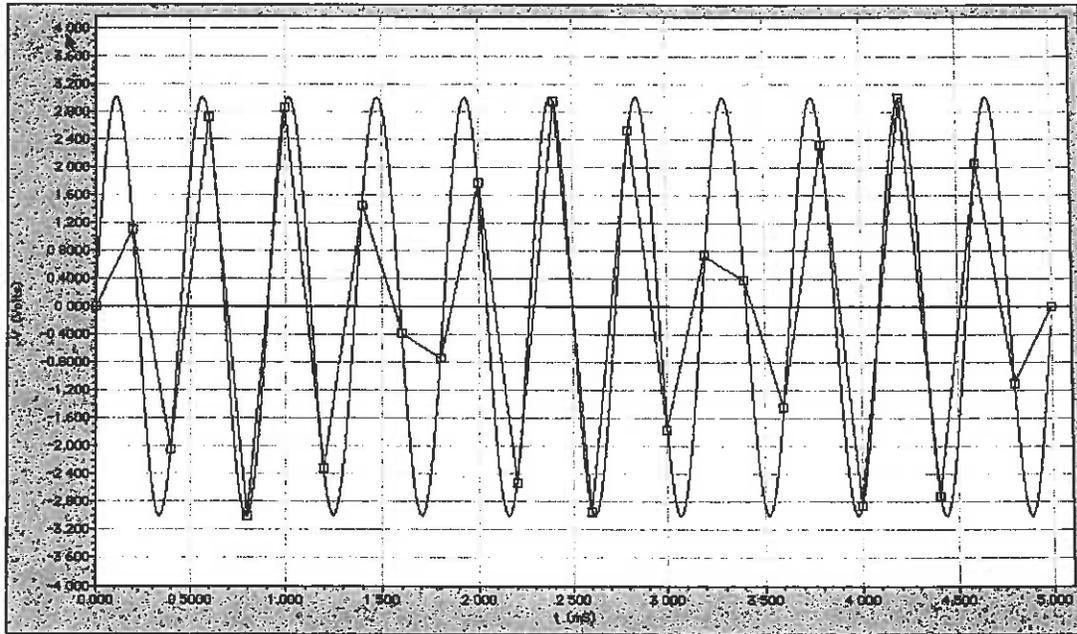


Fig. B4. A 2.2 kHz sinusoidal waveform sampled at 5.0 kHz. There is significant distortion even though the sampling frequency meets the Nyquist criterion.

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

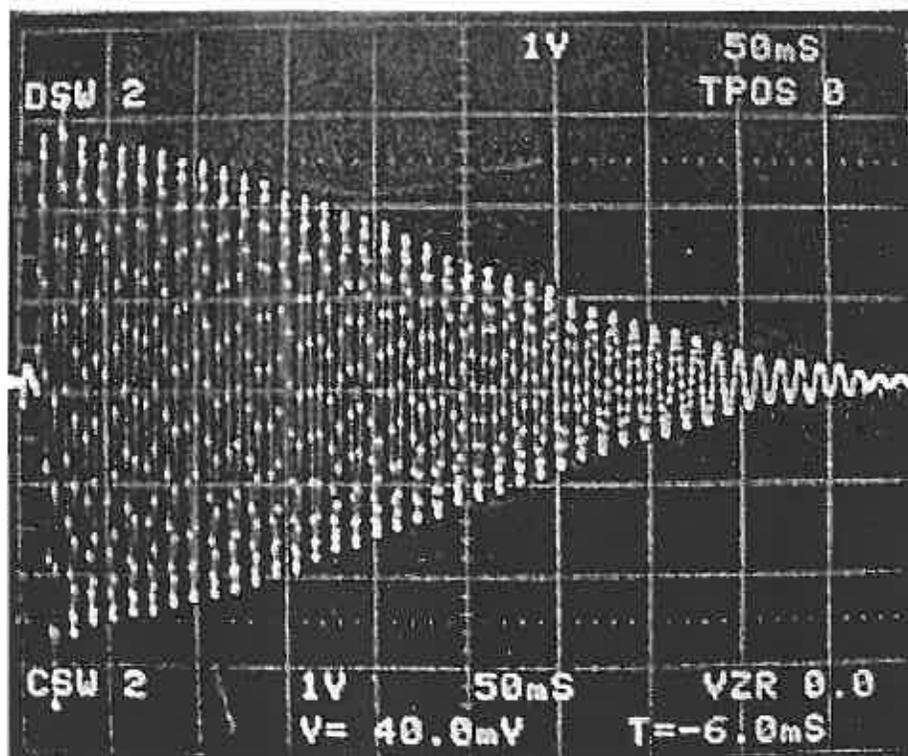


Fig. B5. Apparently smooth free precession signal at a frequency of 2.088 kHz. The sample frequency was 2 kHz.

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

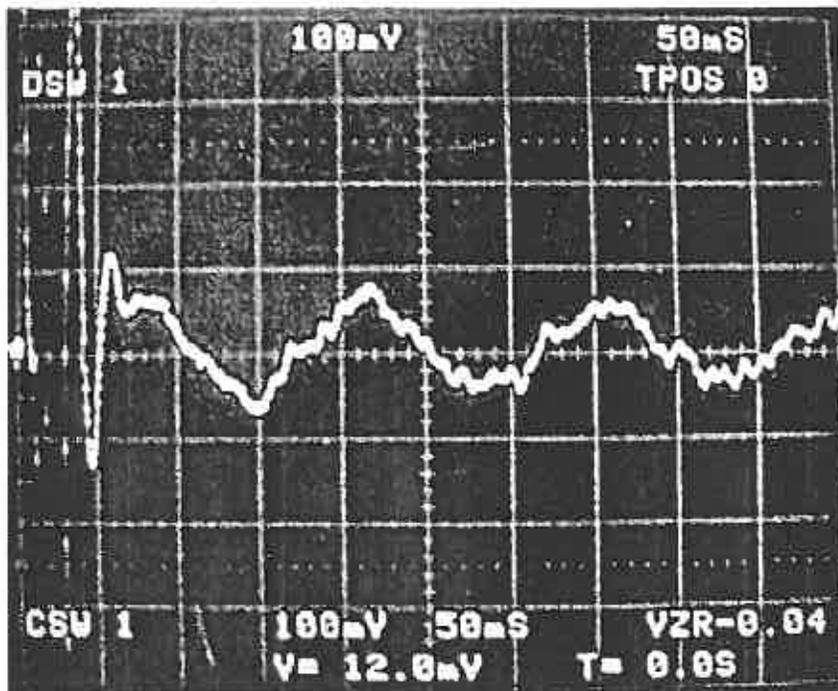


Fig. B6. 2.007 kHz fluorine signal from a 25-gram sample of C_6F_6 . The oscilloscope sample frequency was 2.000 kHz. The digitized waveform appears to have a frequency equal to the difference, 7 Hz.

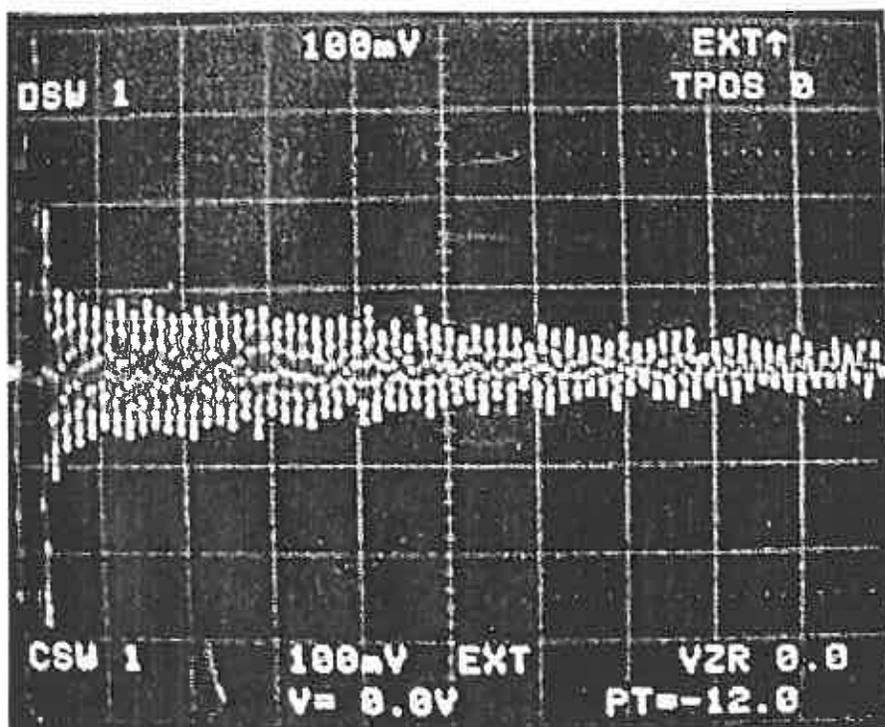


Fig. B7. Same as Fig. B6, except the oscilloscope sample frequency was reduced to 1.040 kHz. Since the oscilloscope displays 100 samples per division, the equivalent sweep speed is 96 ms/Div.

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.

APPENDIX C: Specifications

Accessible Nuclei:

${}^1_1\text{H}$ (protons)

${}^{19}_9\text{F}$ (the abundant isotope of fluorine)

Frequency Range: 1.9 - 2.7 kHz, which corresponds, for protons, to earth's magnetic fields in the range 0.045 - 0.063 mT

Bandwidth: 33 Hz at 2.1 kHz

Sample Coil:

Inside Diameter:	5.1 cm (2.0 in)
Length:	7.0 cm (2.75 in)
DC Resistance:	9.5 Ω
Field-to-Current Ratio:	
At Center:	15.4 mT/A
At End:	9.9 mT/A

Nominal Sample Volume: 125 ml (4 oz)

Range of Polarizing Currents: 0.5 - 3.0 A

Maximum Polarizing Power Supply Voltage: 40 V

Polarizing Times: 0.1 - 99.9 s in increments of 0.1 s

Time Base: Internal, Crystal-Controlled

Range of Measurable Spin-Lattice Relaxation Times, T_1 : 0.1 s to 20 s

Dead Time : 100 ms (includes 20 ms to turn off the polarizing current and connect the coils to the amplifier, plus an additional 80 ms for the transient to decay)

Signal-to-Noise Ratio: Depends on the sample and ambient electrical pickup noise. For a 125-ml water sample, the signal-to-noise ratio (peak signal divided by rms noise) is normally greater than 100:1. Signal-to-noise ratios of 700:1 are achievable in moderately quiet environments indoors.

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

$$M_o = N \left(\frac{I+1}{I} \right) \frac{\mu^2}{3kT} B, \quad (1)$$

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

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

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

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

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

$$\ln(M_0 - M(t)) = \ln(M_0) - \frac{1}{T_1} t. \quad (4)$$

From Eq. (4) we see that a plot of $\ln(M_0 - M(t))$ versus t should be a straight line having intercept $\ln(M_0)$ 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_0 and T_1 as adjustable parameters, to either of the data sets plotted in Procedure 8. Alternatively, one can assume that M_0 is approximately equal to the amplitude of the signal at the longest polarizing time (13.0 s in this case), and plot $M_0 - M(t)$ 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 $\ln(M_0 - M(t))$ 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

$$M_o = N \left(\frac{I+1}{I} \right) \frac{\mu^2}{3kT} B, \quad (1)$$

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

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

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 than 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_0 . (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 times 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.

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, 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, 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.

NUCLEAR MAGNETIC RESONANCE IN THE EARTH'S MAGNETIC FIELD

STUDENT INSTRUCTION MANUAL # 1

Careful reading of this manual and the completion of the pre-lab written assignment will prepare you for making your first magnetic resonance measurements. It is essential that you come to the lab prepared, having reviewed the basic physics ideas of the pre-lab, learned some new definitions, and understood the basic workings of the apparatus.

I. INTRODUCTION

A. The People

On a snowy December 15, in 1945, a group of three young Harvard physicists, fresh from working on a secret radar system at the MIT Radiation Laboratory that greatly helped the allies win World War II, adjusted the controls on a crude electromagnet borrowed from Professor Curry Street's comic ray experiments. They became excited when they observed a repeatable deflection on the detector output meter. It was the first measurement of nuclear magnetic resonance in condensed matter. The material used for these first experiments was paraffin wax. The experimenters were Edward Purcell, Robert Pound and Henry Torrey.¹

Ed Purcell, who led the group and received the Nobel Prize for this discovery, was born and raised in Taylorville Illinois, population 11,133. His father ran a local telephone office and Edward went off to Purdue University to study electrical engineering. How he ended up at Harvard and become one of the great scientist and teachers of the twentieth century is a fascinating story.

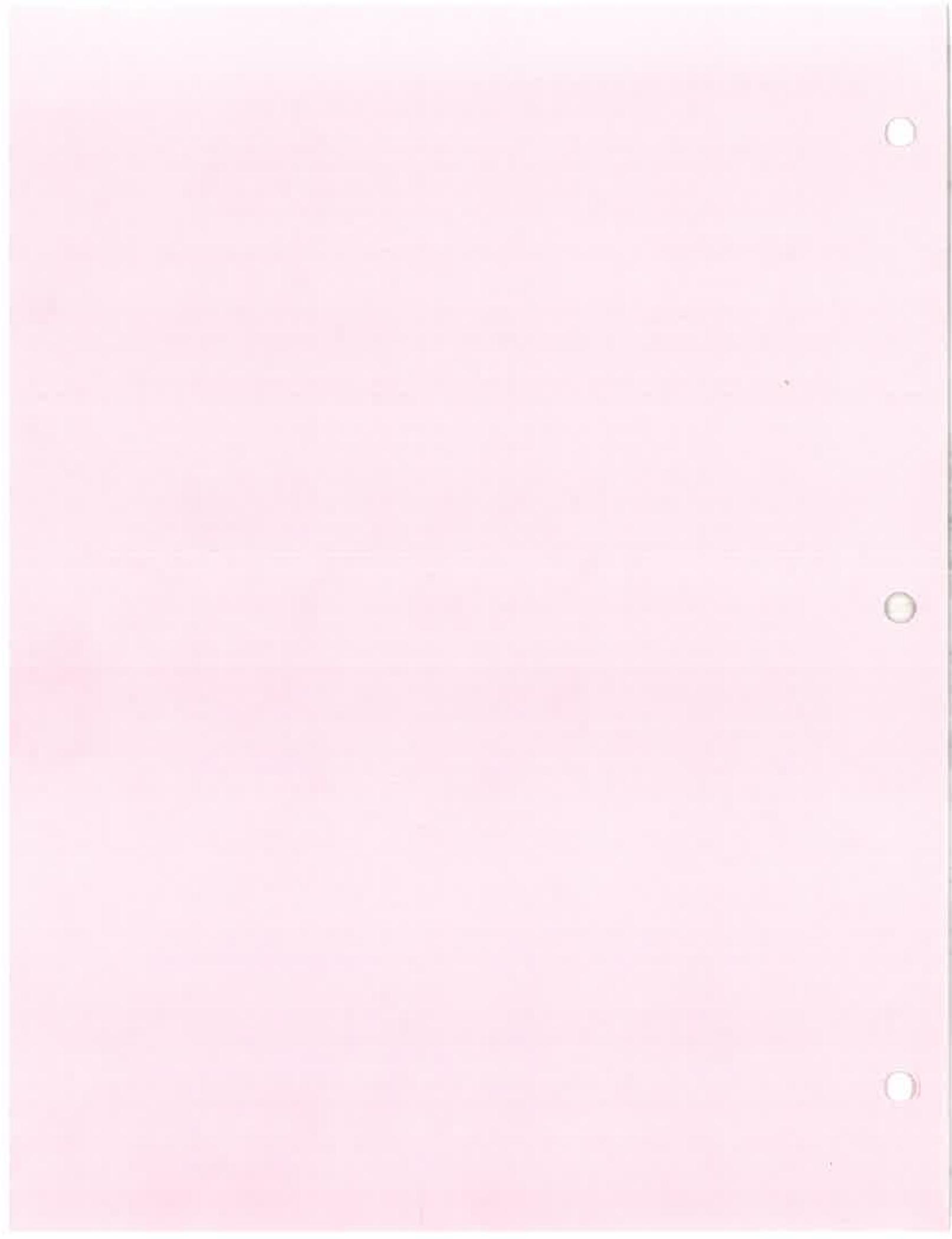
On the west coast, at Stanford University, a group headed by a European Jew who had fled Nazi oppression, had their quite different apparatus almost assembled. Their intent was also to observe signals from magnetic nuclei in condensed matter. Felix Bloch, already a world-renowned solid-state theoretical physicist, born and educated in Zurich, Switzerland, had devised an apparatus to observe "nuclear induction". His work with W.W. Hansen and M. Packard published shortly after Purcell's, is in many ways close to the experiments you will be doing in this laboratory.²

Neither of these two giants of twentieth century physics had any idea that their discoveries would change physics, chemistry, biology, and medicine in truly profound ways. Physicists seem to be very good at making fundamental breakthroughs in science but rather poor at predicting the impact of their discoveries. Mattson and Simon have written a very readable account of history of NMR and the major players in this drama.³

B. The Phenomenon

What kind of atomic size particles in condensed matter exhibit magnetic resonance? Magnetic resonance signals can only be observed from condensed matter whose atomic constituency has two properties: **magnetic moment** and **angular momentum**.

Both are required. Both are absolutely essential. And both act along a common axis, either parallel or anti-parallel. Nature has provided us with many examples, such as a single electron, certain collections of electrons, protons, neutrons, the nuclei of He^3 , Li^7 , Be^9 , C^{13} , N^{14} , O^{17} , F^{19} , Na^{23} , Al^{27} , P^{31} , and many, many more. Magnetic resonance refers to the characteristic precession of these particles in the presence of an external magnetic field.



There is a fundamental relationship between the magnetic moment of a nucleus and its angular momentum. This is written as:

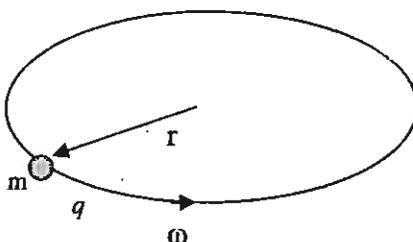
$$\vec{\mu} = \gamma \hbar \vec{I}$$

where I is the nuclear spin, μ is the magnetic moment, \hbar is Planck's constant divided by 2π , and γ is the gyromagnetic ratio. The product $\hbar \vec{I}$ is the angular momentum of the nucleus.

In these experiments we will concern ourselves with only two nuclei, the proton which is hydrogen's nucleus and possibly with the fluorine nucleus, which is in special samples of fluorinated oils. Mostly, these experiments will examine the signals from the hydrogen nuclei, the protons, in ordinary water.

II. PRE-LAB WRITTEN ASSIGNMENT

A. The diagram below shows a point charge executing uniform circular motion..



1. Derive the relationship between the magnetic moment μ and the angular momentum L for this point charge q with mass m which is rotating at a constant angular velocity ω around a circle of radius r . This is a non-relativistic classical physics calculation.
 2. How does this relationship depend on the speed of the rotation?
- B. Show that the same relationship holds for a solid sphere of charge spinning about a fixed axis. Assume a total charge, q , is uniformly distributed throughout the sphere of mass m .
- C. The ratio of the magnitude of the magnetic moment μ to the magnitude of the angular momentum L is defined as the gyromagnetic ratio, gamma, γ :

$$\frac{|\vec{\mu}|}{|\vec{L}|} \equiv \gamma$$

Calculate the numerical value of the ratio for an electron and for a proton.

- D. All of your calculations assume that both the electron and proton are classical particles. They are not! In fact, calculating gyromagnetic ratios for these particles is one of the important theoretical physics problems. Our interest is in the hydrogen nucleus (the proton) and the fluorine nucleus. Your answer for the proton should be off by a factor of 2(2.79). The experimentally measured values are:

$$\gamma_{\text{proton}} = 2.675 \times 10^8 \text{ rad/sec tesla}$$

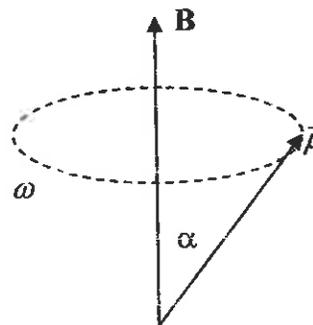
$$\gamma_{\text{fluorine}} = 2.517 \times 10^8 \text{ rad/sec tesla}$$

These are the numbers you should use in any future calculations.

- E. The diagram to the right shows the orientations of the axis and the magnetic moment μ of a spinning sphere of charge with respect to a uniform magnetic field B .

Start with the following two fundamental relationships:

$$\vec{\tau} = \frac{d\vec{L}}{dt} \quad \text{and} \quad \vec{\tau} = \vec{\mu} \times \vec{B}$$



1. Show that a spinning sphere of charge will execute precessional motion when placed in a uniform magnetic field B with its axis of rotation at an angle α with respect to that field.
2. Show that the precession frequency (radians/sec), called the Larmor precession frequency, is independent of angle and is given by the relationship:

$$\omega_{\text{precession}} = \gamma B$$

- F. Review Faradays' Law of electromagnetic induction. Be sure you understand it!

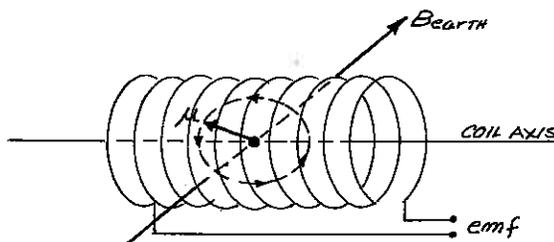
III. MAGNETIC RESONANCE

It probably would be more accurate to call these experiments: "Studies of Free Precession of Nuclear Moments in the Earth's Magnetic Field." This instrument detects the collective precession of the nuclear magnetic moments in the sample. How do we create this "collective" precession? How do we detect this precession once it is created?

A. Detecting the Precession

1. Generating *emf*

Let's start with the question of how to detect the precession of a magnetic moment. Suppose we place our sample in a coil of wire whose axis is perpendicular to the axis of precession of the nuclear moments. This is shown on the diagram. As the magnetic moments precesses in the magnetic field B , the nuclear moments produce a time varying flux of their magnetic field through a plane defined by a turn on the coil. This time varying flux produces an *emf* in the coil. The collective nuclei produce a collective *emf* through all the turns in this coil, which results in a time varying voltage at the coil's terminals. It is this voltage which is amplified and measured in these experiments.



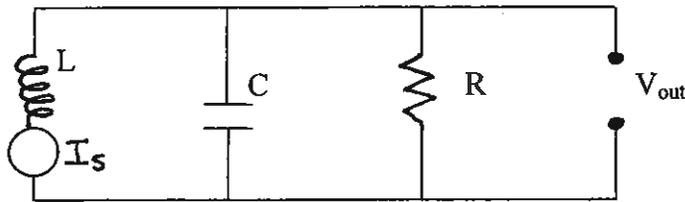
The frequency of the time varying signal indicates the magnetic field in which the nuclei are precessing. The amplitude of the signal is proportional to net number of nuclei that are collectively precessing. This net number of collectively precessing nuclei is called the magnetization, or, in different units, the polarization.

2. Tuning the Coil - What does tuning mean and how does it affect our signal detection?

Nuclear magnetism is generally a rather weak form of magnetism. You cannot attach your water sample to your refrigerator using its nuclear magnetism. It is so weak that special coils and amplifier electronics are required to detect this signal. Even with this, it is essential to “tune” the receiver coil which in our instrument is called the sample coil.

The *emf* generated in the coil depends on the number of nuclei, the rate of precession, the number of turns of the coil, and the coil’s geometry. But the actual voltage at the terminals of the coil can be significantly increased by tuning. (Note that the *emf* and the voltage are not the same for a tuned coil. They are the same for a simple coil which has no appreciable capacitance or inductance.)

Let us consider our sample coil to be part of a parallel circuit which has a capacitor across its terminals. The equivalent circuit is shown in the diagram where *C* is the added capacitance, *L* the coil’s inductance, *R* the coils resistance, and *I_s* is the current generated in the coil by the precessing nuclear moments.



We shall choose our capacitance so that its capacitive reactance is equal to the inductive reactance (but 180° out of phase) at the Larmor precession frequency. As you may recall, capacitive reactance is $\frac{1}{\omega C}$ and inductive reactance is ωL .

In this case, the signal current I_s creates an output voltage V_{out} as it flows through the resistance R .

$$V_{out} = I_s R \quad (a)$$

But the *emf* across an inductor is:

$$emf = I_s \omega L \quad \text{or} \quad I_s = \frac{emf}{\omega L} \quad (b)$$

So, by substitution of (b) into (a)

$$V_{out} = \frac{emf}{\omega L} R \quad (c)$$

The quality factor, or Q , of a coil is defined as the energy stored divided by the energy dissipated per cycle. For a parallel, tuned, resonant, circuit it can be shown that:

$$Q = \frac{R}{\omega L} \quad (d)$$

Substituting into (c)

$$V_{out} = \frac{emf}{\frac{R}{Q}} = (emf) \cdot Q \quad (e)$$

The output voltage of the parallel tuned circuit is a factor of Q times the emf generated by the precessing spins. The special sample coil designed by TeachSpin, has a Q of about 80! This tuned coil gives a giant enhancement of almost two orders of magnitude when the capacitor is adjusted so that its reactance is equal in magnitude to the inductive reactance, at the Larmor precession frequency. If the capacitance is not properly tuned then the voltage output is not so enhanced and the signal is just the emf .

This tuning has another advantage. It partially reduces random noise signals. The voltage at the terminals of the coil depends not only on the magnitude of the time varying flux from the precessing spins, but also on the frequency of that precession. Only noise signals at or near the Larmor frequency are enhanced by the tuning and therefore noise signals outside this range are significantly reduced. In effect the tuned coil acts like a band-pass filter for the system.

B. Creating the "Collective Precession" - Magnetization

Now we turn to the question of how to create a "collective" precession which can induce a measurable emf . With this apparatus, or indeed most NMR instruments, it is not possible to observe a single proton's free precession. The proton's magnetic moment is far too small to detect the voltage at the coil's terminal. Both externally induced pickup noise, and internally created thermal noise from the coil itself, make such detection impossible.

Suppose, however, that we use 125ml of water as a sample. This contains about 10^{25} hydrogen nuclei (protons). If all those protons were aligned with their magnetic moments pointing in the same direction and they were all to precess and remain aligned, then our signal would be 10^{25} times as large as from a single proton!

The degree of alignment in a sample is formally referred to as the polarization or as the magnetization, M . Quantum physics tells us that there are only two possible magnetic states for the proton to reside in, spin along B , spin opposed to B . The magnitude of proton magnetization is proportional to the difference between spin along B and against B or

$$M \propto n_{\uparrow} \mu_{\uparrow} - n_{\downarrow} \mu_{\downarrow}$$

where n_{\uparrow} and n_{\downarrow} refer to the number/unit volume of magnetic moments along and against the magnetic field B .

In this apparatus, the initial signal amplitude is proportional to the magnetization of the sample. You can use this to amplitude to explore two parameters that might control the magnitude of the magnetization, the strength and the duration of the polarizing magnetic field. Both the polarizing field and the polarizing time are under your control!

Does the strength of the polarizing field matter? If so, what is the mathematical form of that dependence? Does the magnetization occur instantaneously when the magnetic field is turned on? If not, your job is to discover the mathematical relationship for the magnetization as a function of the time you leave the polarizing field on.

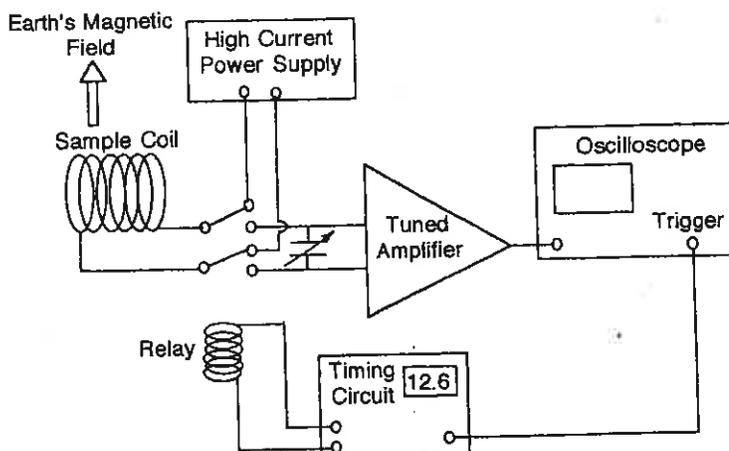
D. Summary of Objectives for the First Set of Experiments

1. Measure the magnitude of your local Earth's magnetic field by determining the frequency of precession of the protons in a water sample.
2. Does the degree of polarization (or the magnetization) of the protons depend on the strength of the magnetic field they are placed in? If so, what is the mathematical relationship between the polarizing magnetic field and the polarization?
3. Does the polarization occur instantaneously? If not, how long does it take to polarize (or magnetize) the water sample? What is the mathematical description of the time dependence of the polarization?
4. Are the degree of polarization and the rate of polarization interrelated? In other words, does the rate of polarization depend upon the polarizing magnetic field?

E. Overview of the Apparatus

Perhaps the best way to explain the apparatus is to examine the simplified block diagram shown at the right.

The high Q sample coil surrounds a 125ml plastic bottle filled with distilled water, or another liquid to be investigated. The sample is placed in a uniform part of the local Earth's magnetic field with the **axis of the coil perpendicular to the Earth's field.**



At the beginning of the experiment, the electronic timers and relays connect the sample coil to the external dc power supply. The current from this external supply produces the so-called "Polarizing Magnetic Field." This polarizing field is perpendicular to the Earth's magnetic field and **much** larger in magnitude. The time the sample coil remains connected to this supply is set by the rotary switches on the electronic controls.

After the predetermined time of "soaking" the nuclei in this large polarizing magnetic field, the polarizing current is disconnected from the coil, the stored magnetic energy in the coil is quickly dissipated, and the coil is connected to the tuning capacitors and the low noise amplifiers. The polarizing magnetic field is removed so quickly that the nuclei remain

polarized along the direction of the polarizing field. With the polarizing magnetic field turned off, the nuclear magnetization precesses in the Earth's magnetic field, producing a time varying flux through the sample coil. The time varying flux creates an *emf*, which creates a time varying voltage at the terminals of the tuned circuit. This voltage is amplified by the preamplifier and tuned amplifier in the electronics.

The precession signal is directed both to the oscilloscope and to the audio amplifier and speaker. You can not only see the signal from the precessing nuclear moments, you can hear it!

F. Investigating the Six Distinct Pieces of the Apparatus

A quick list – You should examine each piece carefully.

1. Coils with special low loss cable.
2. Support stand for coils.
3. Electronic Controls
- 4 Brick-on-a-rope (power supply for electronics)
5. External dc high current power supply.
6. Oscilloscope (or computer with interface)

1. Coils – Note that the instrument has two coils.

The larger outer (halo) coil is essential for dramatically reducing the pickup from external electromagnetic noise fields. Although an important part of the instrument, making it possible to observe the magnetic resonance signal in the presence of extraneous noise, this coil can be ignored by beginning students. They can simply assume that electromagnetic noise is not present in their laboratory. (Turn off all florescent lights and incandescent lights that are on dimmer switches. Dimmers usually produce significant noise fields which are only partly cancelled by this outer coil.)

The inner coil is the sample coil. It has two functions: (please refer to the simplified circuit diagram). First, it provides a large (and variable) magnetic field to polarize (magnetize) the nuclear spins. Then, because of its high Q and multiple turns, it generates an optimum signal voltage from the precessing nuclear spins.

2. Support stand for coils.

This nonmagnetic stand allows the student to conveniently place the coils in a region of the best uniform Earth's magnetic field in the laboratory. It is important to place the sample in the most uniform part of the Earth's magnetic field. If the nuclear moments in the sample find themselves in different magnetic fields they will precess at different rates. In fact, for pure water, the rate at which the signal decays to zero tells the experimenter the field gradient across the sample.

It is usually best to place the coils away from walls, tables with steel parts, off the floor where iron drain pipes may be buried and away from steel support columns. Your equipment might not include this stand, but you must make sure that the sample is placed away from all ferromagnetic materials. Small spatial adjustments, even just a few inches, can often make a significant difference in the observed decay time of the precession signal.

3. Electronic Controls

- a. Timing Circuits. These circuits provide the various timing functions
- The length of polarizing time, which is set by the thumbwheel switches on the panel. This is variable in steps of 0.1s, from 0.1 to 99.9 seconds.
 - Various delay times, such as a 5 second delay after every experimental cycle, an 80 millisecond delay after switching off polarizing current to allow voltage transients to die out, and a delay time equal to polarization time to keep duty cycle to under 50%.
 - Synchronizing pulses for oscilloscope triggering. For NMR signals, the scope should be set to trigger on a negative pulse, negative slope, at minus one volt.
 - Manual Start. A push button switch that start the entire sequence.
 - External Start. A contact closure to ground or a negative TTL pulse performs the same function as pressing the manual start button. Use this only if you need to start the experiment with an external electronic signal.
 - Wait Light. Tell the experimenter that the instrument is still going through its cycle and the operator needs to wait before pushing the start button again.

b. Amplifier

As we discussed earlier, it is essential to tune the sample coil to the frequency of the precessing magnetic moments. Since this precession frequency is uniquely determined by the local Earth's magnetic field in your laboratory, the student must experiment with the controls to determine the proper tuning. There are both COARSE and FINE controls. Note that the tuning frequency increases with counterclockwise rotation. If you find your instrument adjusted to the wrong frequency, you may not be able to detect any signal from the precessing moments.

There are two outputs on the amplifier, one marked preamplifier and the other NMR signal. First adjust the tuning capacitors to maximize the output of the preamplifier. (Note. Use AC coupling on the oscilloscope since the preamplifier has a dc offset imposed on the signal from the electronics). The NMR output comes from the second stage amplifier. This is a tunable bandpass amplifier. It must also be adjusted so that the center of the bandpass is at the Larmor frequency. This adjustment is made by the 10 turn counter dial. All these adjustments can be made on a signal from the distilled water sample.

c. Amplitude Detector

The signal available at the NMR SIGNAL OUTPUT is also connected internally to an Amplitude Detector, which consists of a precision 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 the same shape as the envelope of the free-precession signal, but the amplitude is about $2/3$ as large. This signal is particularly useful for signal averaging of repetitive experiments. Signal averaging is a common feature on digital oscilloscopes. It can enhance the signal to noise ratio for weak signals.

d. Audio Amplifier and Speaker.

The precession frequency for both protons and fluorine nuclei in a typical local Earth's magnetic field is about two thousand cycles per second. This is in the audible frequency range. This instrument has a volume controlled audio amplifier connected to an internal speaker. You can hear the signal generated by the precessing nuclear moments in the sample. If you are lucky to have a particularly uniform field in your lab enough (or you take the instrument outside and away from the building), then you might hear the sound for several seconds.

4. Brick-on-a-Rope (power supply for electronics)

This is the dc regulated power supply that provides ± 15 volts and + 5 volts to operate the electronics. The supply (brick) plugs directly into the ac power line and the dc outputs are connected by cable (rope) to the rear panel of the control box. It is best to leave the dc line plugged into the rear panel and remove the supply from the power line when you have completed the experiments. There is no on-off power switch.

5. External Power Supply

This power supply provides the direct current for the polarizing magnetic field. This supply should be CURRENT REGULATED with a current range between 0.5 and 3 amperes and with a maximum voltage of about 38 volts. Ask your instructor to explain how you adjust the supply for current regulation. Do not run the experiment with less than 0.5 amperes or more than 3.5 amperes.

DO NOT GROUND EITHER SIDE OF THE EXTERNAL POWER SUPPLY. This supply must be floating!

The polarizing field is changed by adjusting the output current. If the supply is not current regulated, the voltage must be adjusted during the experimental run to keep the current and thus the polarizing field, constant. With repeated measurements, the copper wire heats up, the coil resistance increases and, if the supply is only voltage regulated, the current and magnetic field decrease.

DO NOT TURN THE POLARIZING CURRENT ON UNTIL THE INSTRUMENT ITSELF HAS BEEN POWERED BY THE BRICK-ON-A-ROPE.

6. Oscilloscope

A digital scope is highly recommended, but it need not have great bandwidth. A 20 MHz scope is more than sufficient for these measurements. However, "aliasing" can be a major problem for beginning students. Some digital scopes are particularly bad and the signals they present can be extremely confusing. If you are not familiar with this inherent problem with digitizing data, you should carefully read the appendix. One simple test for incorrect data presentation due to aliasing is to change the sweep time to a faster sweep by one unit and observe the same signal. If the signal changes appropriately, it is not aliasing, but if the scope presents significantly different data, then the first data should be ignored.

G. Basic Experiments

The first set of experiments will be done on distilled water sample in the 125ml plastic bottles. This will provide you with a large signal that should be relatively easy to find. Here are a few helpful hints:

1. The preamplifier output maximum signal amplitude should be about 200-300 mv peak-to-peak.
2. The Larmor frequency should be about 2 KHz or about a half a millisecond period. Set the oscilloscope so that you can observe the oscillations. After you have observed them, change the time scale so you can observe the decay of the envelope of these oscillations. What causes this decay?
3. The NMR output from the bandpass amplifier should be about 6 volts peak-to-peak.
4. Use the NMR detector output to measure relative signal amplitude if you want to signal average or avoid aliasing problems on your digital scope.

Play with the apparatus until you feel comfortable with it. Get some ideas on how you will go about designing experiments to answer all the questions in the summary section. Take your data in a systematic way and find an appropriate way to plot it.

It is absolutely essential THAT YOU PLOT THE DATA AS YOU RECORD IT. That means you may want to manipulate this data. Always record the raw data in your data table. For example, 4.5 division, with sensitivity of 5 mv/div. Do not do the multiplication in your head and record 22.5 mv. You are likely to make a mistake. But suppose this magnetization depends on t^2 , what do you plot? If you plot M vs t^2 you should get a straight line. If this data fits a straight line in such a plot, you have very strong evidence that $M \propto t^2$. But a plot of M vs t cannot yield such a conclusion.

Why must you plot the manipulated data as you do the experiments? Suppose you make a mistake. You read the scope incorrectly, you set the time wrong, the polarization field current drifted, the amplifiers became unstable, or some other problem. You will immediately see that you have a problem from your plot. You can now check everything and attempt to find the problem. This gives you the chance to correct it. If you take the data home and plot it on your computer, you will not be able to understand the one or two "crazy" data points. On the other hand, they may not be crazy – you may have made a discovery.

Once again PLOT your manipulated or raw data as you do the experiment. This is the best advice you ever got!

H. Additional Experiments

Once you have determined the basic relationships between magnetization, polarizing current and polarization time for distilled water, you can explore the effect of adding solutes on proton magnetization or the behavior of fluorine nuclei.

1. Does the effect of doping the water with NaCl, table salt, differ from the effect of doping with compounds such as copper sulfate and ferric chloride?
2. How does the concentration of the solute change the way the magnetization behaves.

3. Try using a fluorine compound. You will have to retune the amplifier because the Larmor frequency for your fluorine nucleus will be different from that of the proton.
 - a. Use the Larmor frequencies you have determined for fluorine and water to determine the ratio of the gyro-magnetic ratios of these nuclei. Does this ratio depend on the local magnetic field?
 - b. How does the viscosity of the fluorine sample affect the NMR parameters?
4. If your professor is willing to have you work with volatile, flammable liquids, try studying the proton resonance in a sample such as fluorobenzene which contains both protons and fluorine.

¹ *Phys Rev* 69 (1946) 37-38 "Resonance absorption by Nuclear Magnetic Moments in a solid".

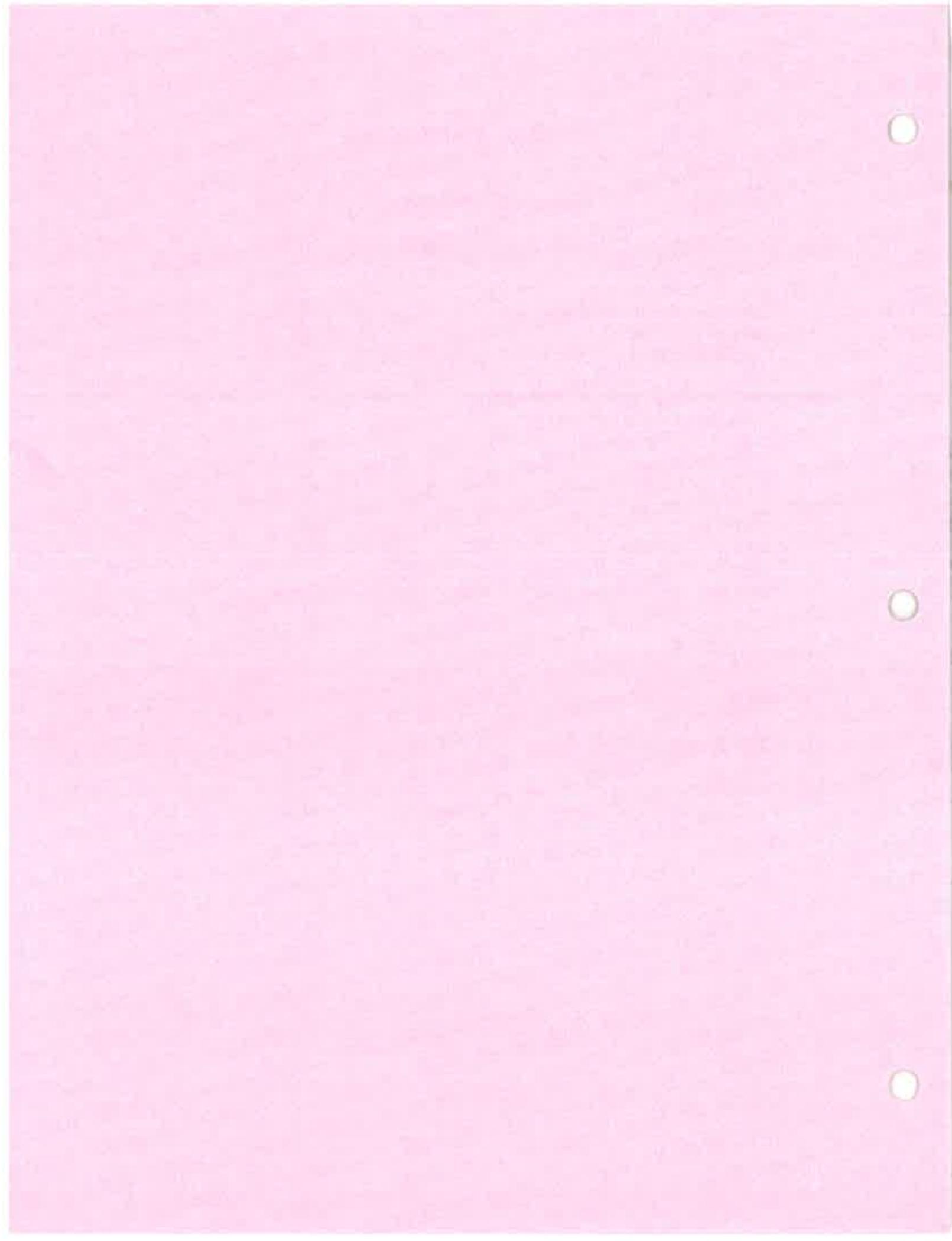
² "The Nuclear Induction Experiments" F. Block, W.W. Hansen and M. Puckard, *Phys Rev* 70 (1946) 474

³ "The Pioneers of NMR and Magnetic Resonance in Medicine" James Mattson and Merrill Simon. Deana Books Co. ISBN 09619243-1-4

Student Manual #2

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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

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

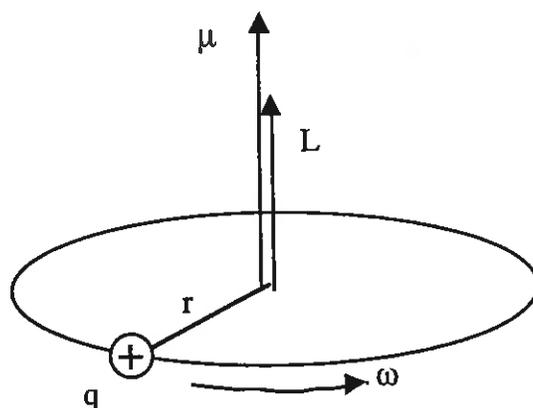


Fig. 1. Magnetic moment and angular momentum vectors for a charged particle rotating at constant angular velocity in a circle of radius r .

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

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

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 $\gamma = q/2m = (1.602 \times 10^{-19} \text{ C})/(1.672 \times 10^{-27} \text{ kg}) = 9.58 \times 10^7 \text{ C/kg}$. The measured value is $\gamma = \bar{\mu} / \bar{L} = 2.675 \times 10^8 \text{ C/kg}$, which is larger than the classically-computed value by a factor of 2.79. 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 \bar{L} and magnetic moment $\bar{\mu}$, both of which point in the same direction along the axis of rotation.

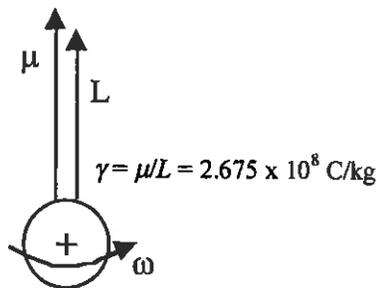


Fig. 2. Classical model of a proton. The magnetic moment and spin angular momentum vectors point in the same direction along the axis of rotation.

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 \bar{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

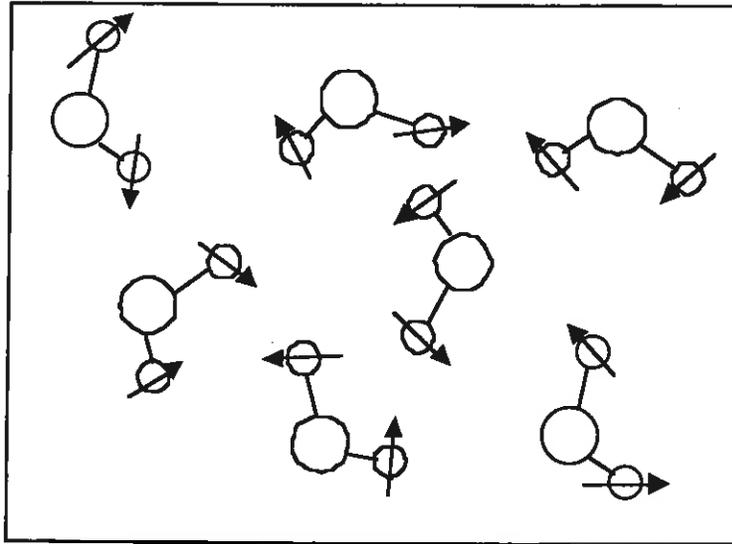


Fig. 3. Schematic representation of molecules of water, H_2O . In the absence of an external magnetic field, the magnetic moment of each proton (hydrogen nucleus) is randomly oriented in space.

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:

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

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.

Actually, a quantum-mechanical correction to the classical expression in Eq. (2) must be made. The corrected result is

$$M_o = \left(\frac{I+1}{I}\right) \frac{N\mu^2 B}{3kT}, \quad (3)$$

where I is the nuclear spin quantum number, equal to $1/2$ for protons. Equation (3) predicts a magnetization proportional to the field B and inversely proportional to the

temperature in kelvins. It is valid so long as $\mu B/kT \ll 1$. 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.

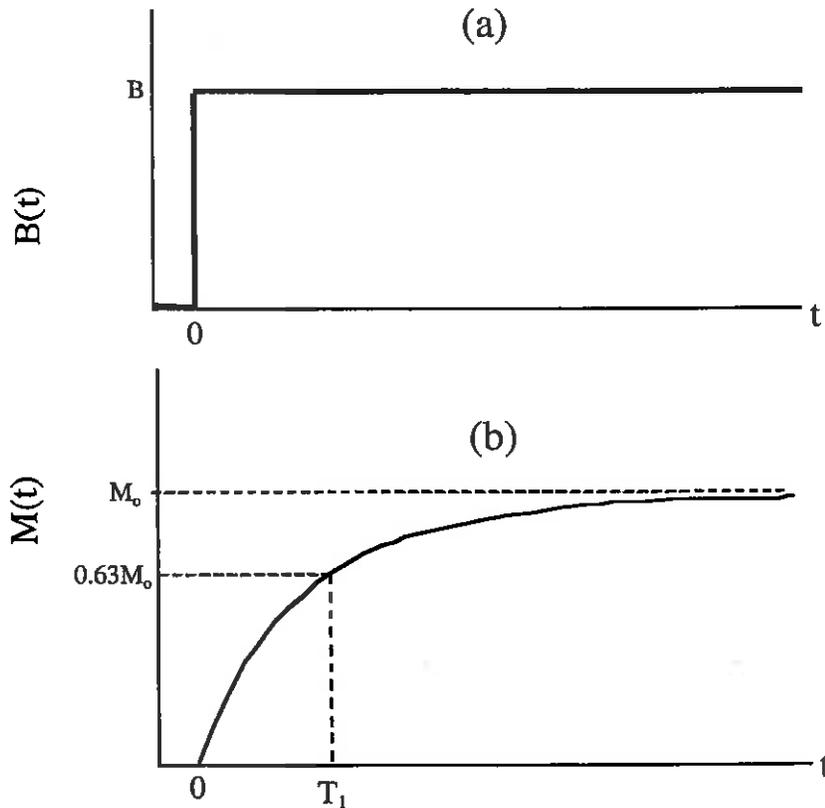


Fig. 4. (a) A constant magnetic field B is switched on at time $t = 0$.
(b) The magnetization grows exponentially with time constant T_1 toward the equilibrium Curie value M_0 .

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

$$M(t) = M_0 \left(1 - e^{-\frac{t}{T_1}} \right) \quad (4)$$

where M_0 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_0 . After two time constants $M(2T_1) = 0.86M_0$. After five time constants $M(5T_1) = 0.99M_0$. 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{B} , it experiences a magnetic torque $\vec{\tau} = \vec{\mu} \times \vec{B}$ 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

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

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_0) is usually very close to 90° .

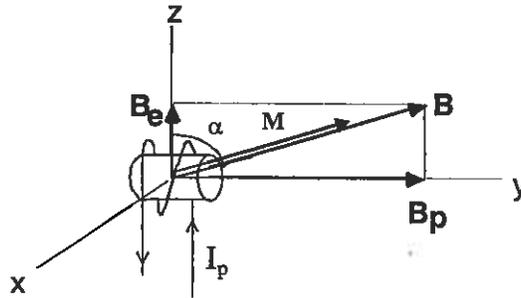


Fig. 5. A schematic representation of the geometry for the earth's-field free precession technique.

When the coil current is reduced to zero, the resultant field \vec{B} shrinks in magnitude and rotates through angle α_0 . 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 $\theta_f \approx \alpha_0 \approx 90^\circ$. 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 $\theta_f \approx \alpha_0 \approx 90^\circ$.

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.

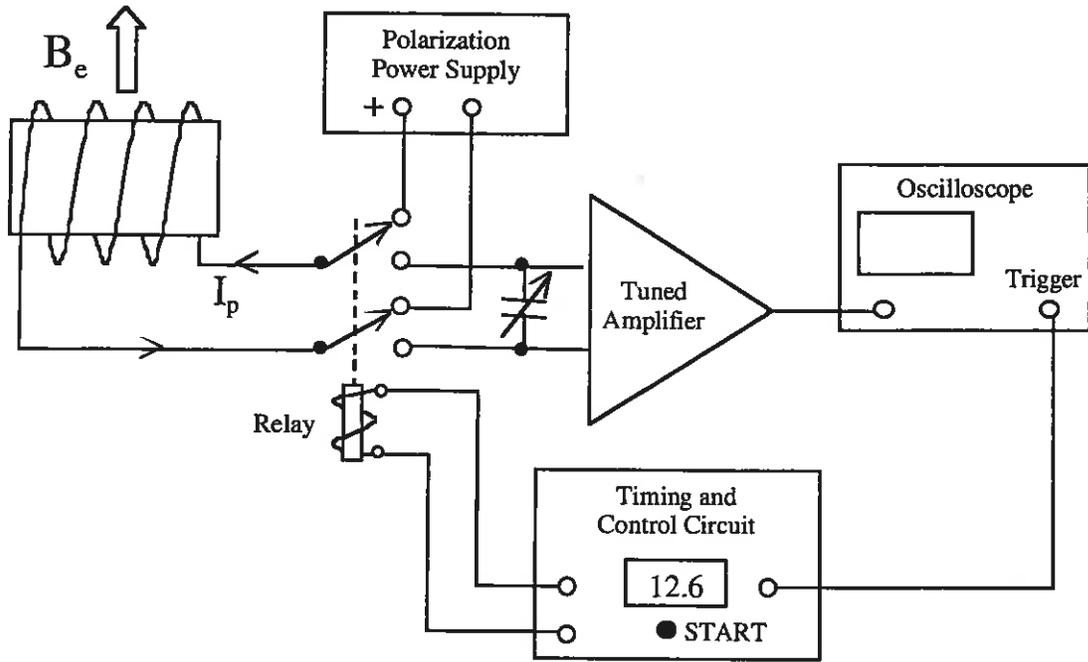


Fig. 6. Simplified block diagram of the apparatus required for the earth's-field free precession technique.

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 \bar{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.

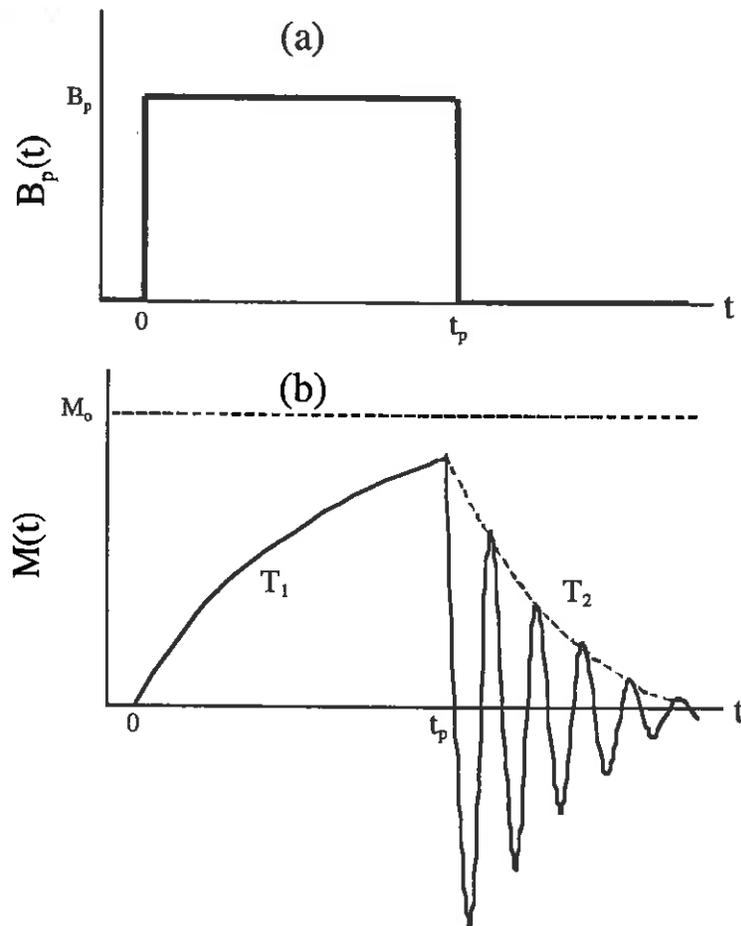


Fig. 7. Variation of polarizing field B_p and magnetization M with time in the earth's-field free precession technique. (a) The polarizing field B_p is switched on at time $t = 0$, and switched off at the end of the polarizing time t_p . (b) When the field is first turned on, magnetization rises exponentially 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.

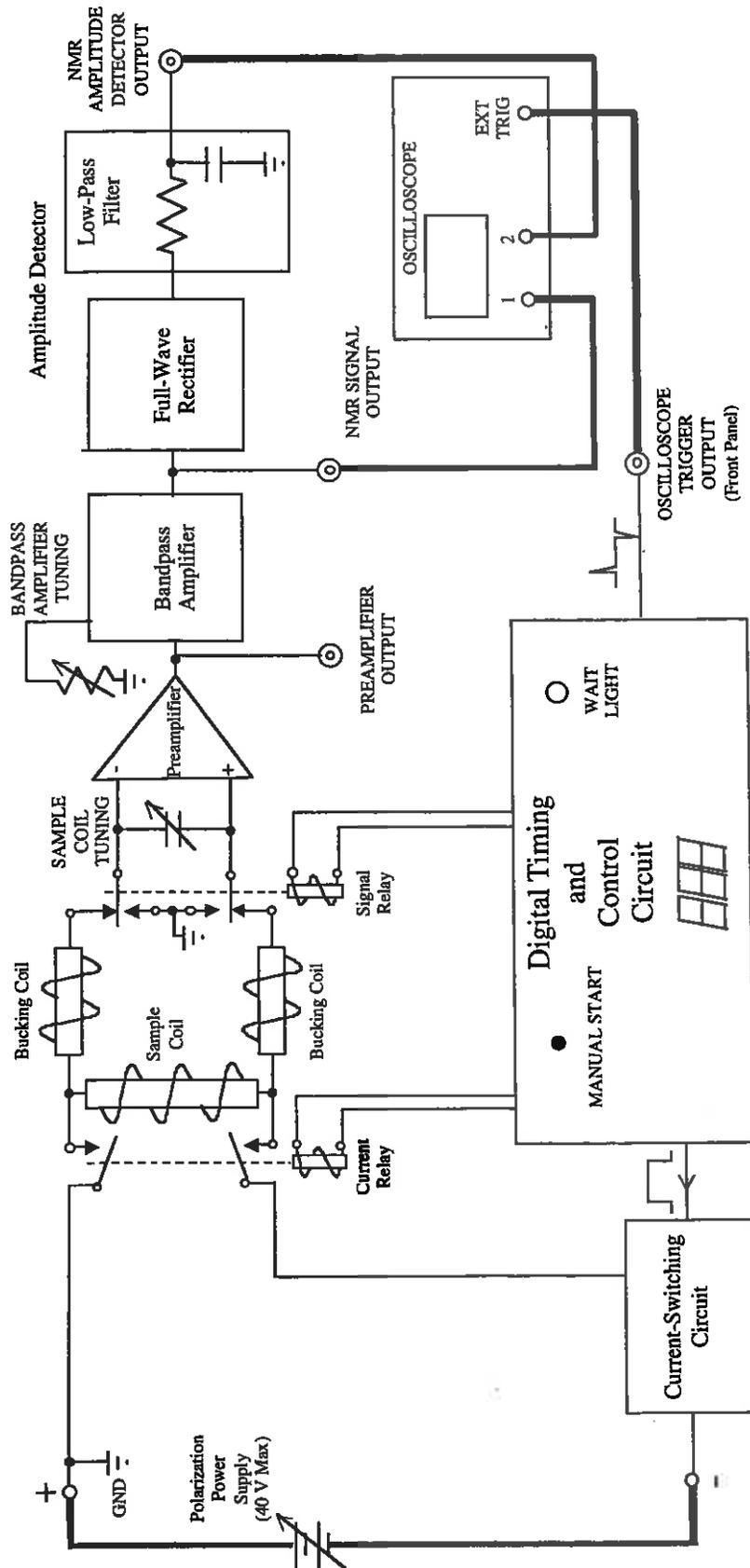


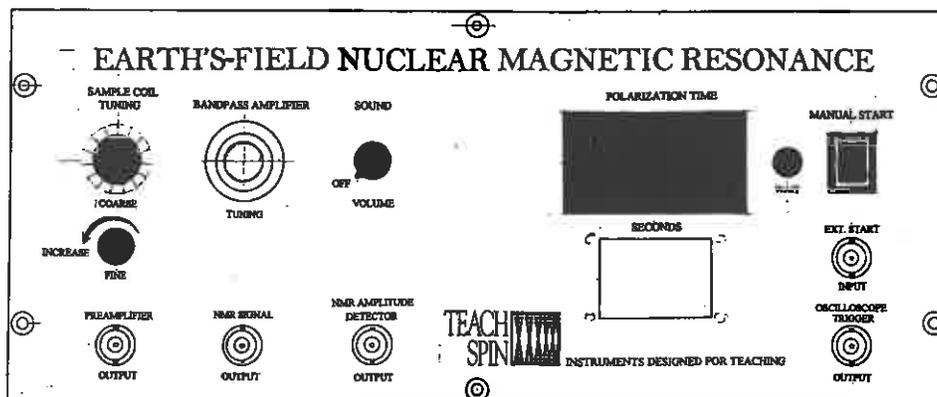
Fig. 8. Block diagram of the Earth's-Field NMR Instrument. External wires and cables are indicated by wide dark lines.

- 4) 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.
- 5) 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.
- 6) 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, $\omega_0 = (LC)^{-1/2}$, 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.

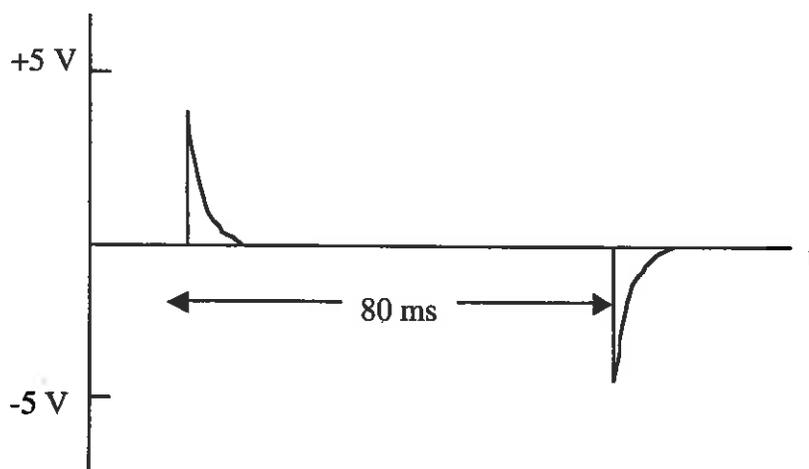
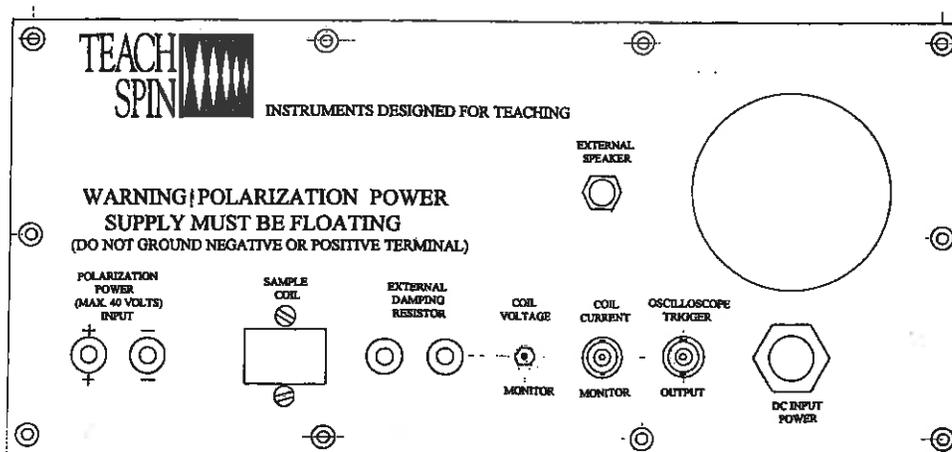


Fig. 9. Pulse sequence at the front-panel OSCILLOSCOPE TRIGGER OUTPUT.

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.

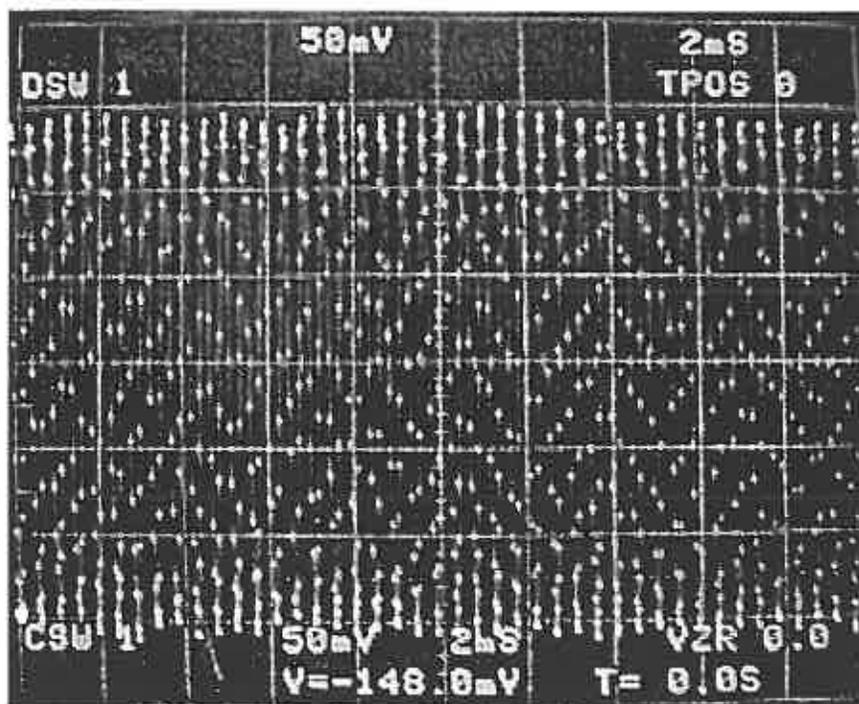


Fig. 12. Free precession signal at the PREAMPLIFIER OUTPUT. The oscilloscope is set at 50 mV/Div and 2 ms/Div. The sample frequency is 100 samples/Div for all oscilloscope waveforms shown in this manual.

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.

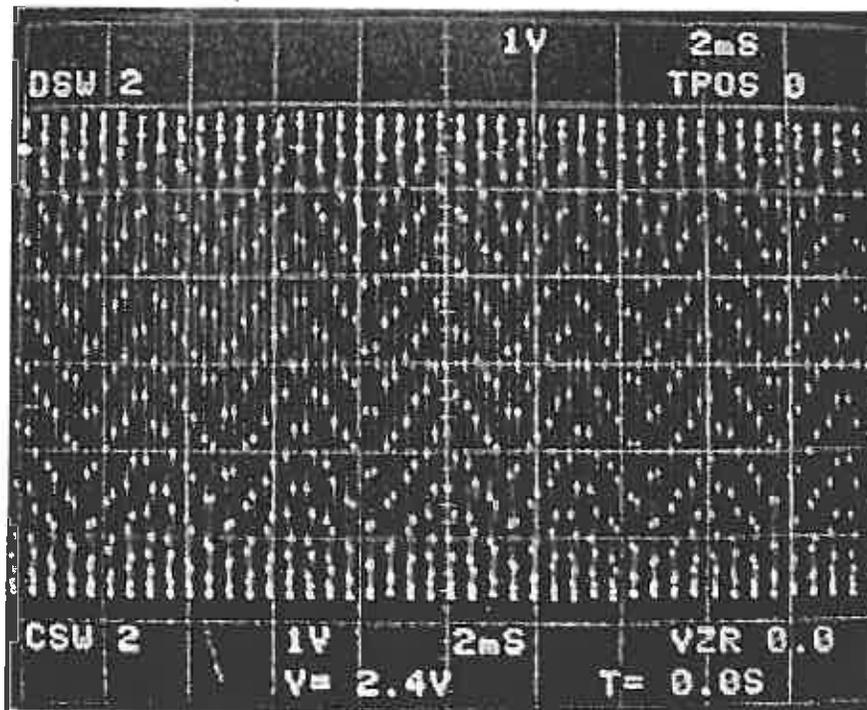


Fig. 13. Free precession signal at the NMR SIGNAL OUTPUT. The oscilloscope is set at 1 Volt/Div and 2 ms/Div.

Figure 15 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. 15 exhibits the Gaussian-shaped decay that is commonly observed where the earth's field indoors is relatively inhomogeneous. Refer to Fig. 17 on page 21 for an example of the longer decays that are observed in more homogeneous earth's magnetic fields.

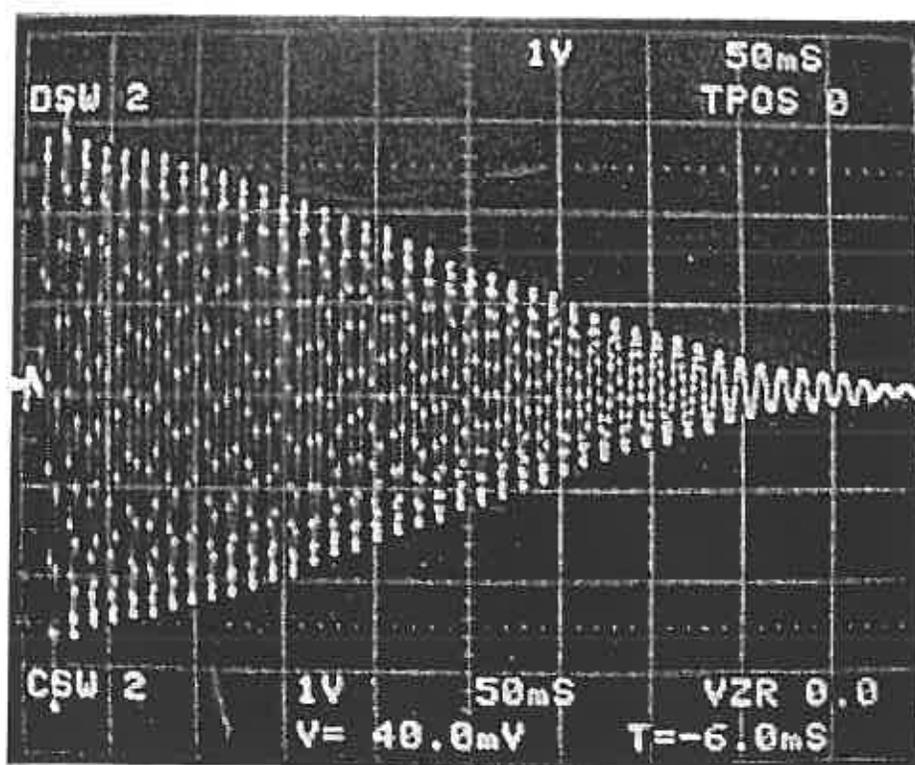


Fig. 15. Water free precession signal at the NMR SIGNAL OUTPUT. Note the Gaussian-shaped decay caused by a relatively inhomogeneous earth's magnetic field. The oscilloscope is set at 1 V/Div and 50 ms/Div.

NMR AMPLITUDE DETECTOR

Figure 17 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. 17 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. 17 are shown in Fig. 18, but there the sweep speed has been increased from 50 ms/Div to 20 ms/Div. Note in Fig. 18 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.

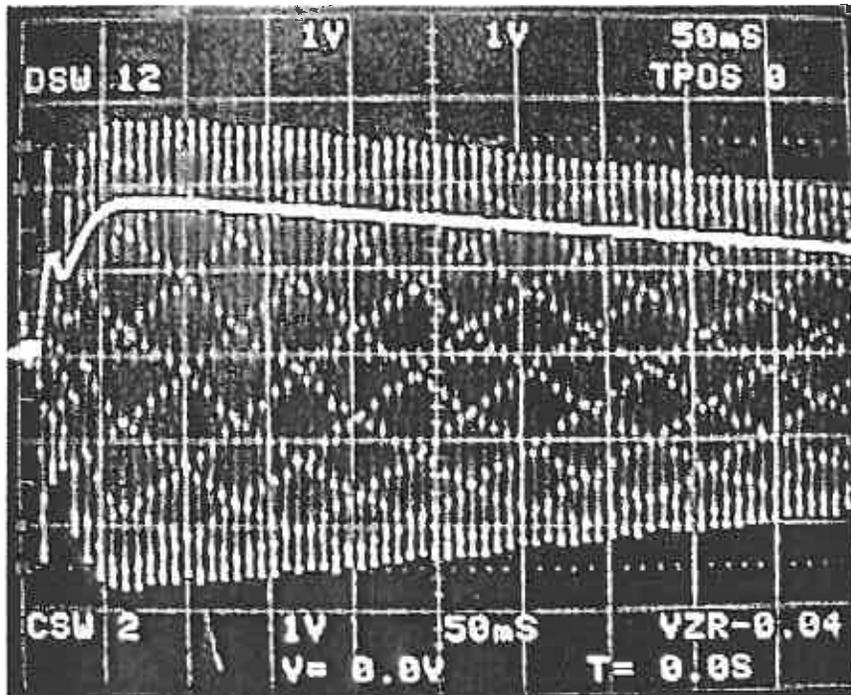


Fig. 17. Water NMR SIGNAL shown along with the output of the NMR AMPLITUDE DETECTOR. Both oscilloscope channels are set at 1V/Div and 50 ms/Div.

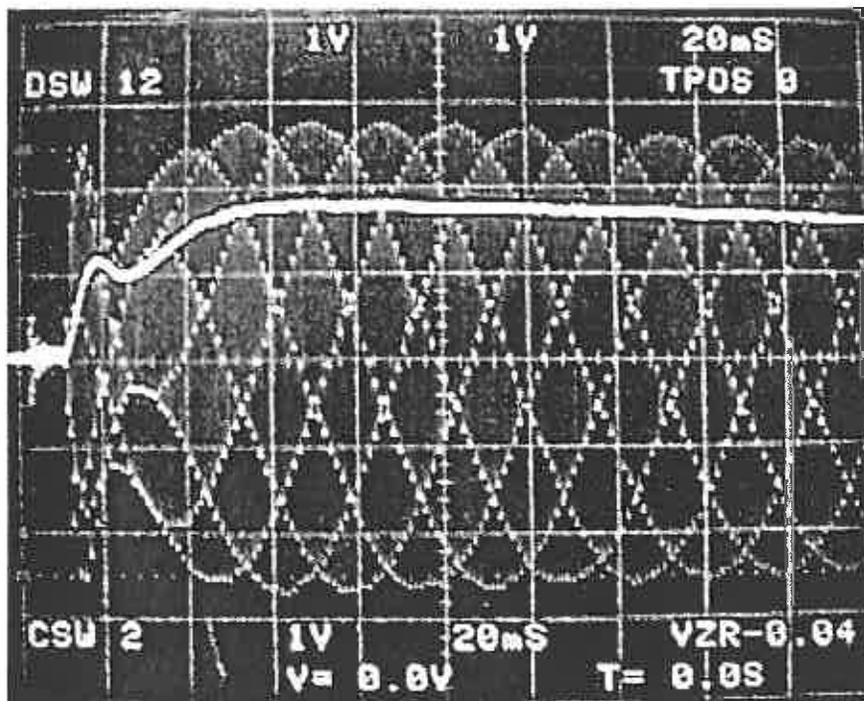


Fig. 18. Same signals as in Fig. 17, but with the oscilloscope sweep speed increased to 20 ms/Div. Note the appearance of "beats" in the NMR SIGNAL OUTPUT caused by problems associated with aliasing.

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.

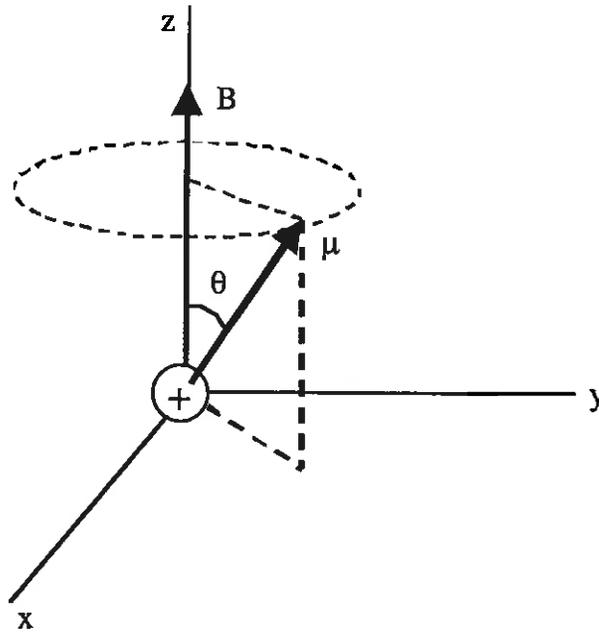


Fig. A1. A positively charged nucleus with its magnetic moment $\vec{\mu}$ oriented at angle θ with respect to a magnetic field \vec{B} pointing along the +z axis. The angular momentum vector \vec{L} , not shown, points in the same direction as $\vec{\mu}$.

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

$$\vec{\tau} = \frac{d\vec{L}}{dt} \quad (\text{A1})$$

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

$$\vec{\tau} = \vec{\mu} \times \vec{B} \quad (\text{A2})$$

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

$$\frac{d\vec{\mu}}{dt} = \gamma \vec{\mu} \times \vec{B} \quad (\text{A3})$$

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:

$$\frac{d\mu_x}{dt} = \gamma B \mu_y, \quad (\text{A4})$$

$$\frac{d\mu_y}{dt} = -\gamma B \mu_x, \quad (\text{A5})$$

and

$$\frac{d\mu_z}{dt} = 0. \quad (\text{A6})$$

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

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

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

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

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

$$\mu_x(t) + i\mu_y(t) = (\mu_x(0) + i\mu_y(0))(\cos \gamma B t - i \sin \gamma B t).$$

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

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

and

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

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 1A shows a 2200 Hz sinusoidal waveform and, for comparison, the sampled approximation. The samples are shown as

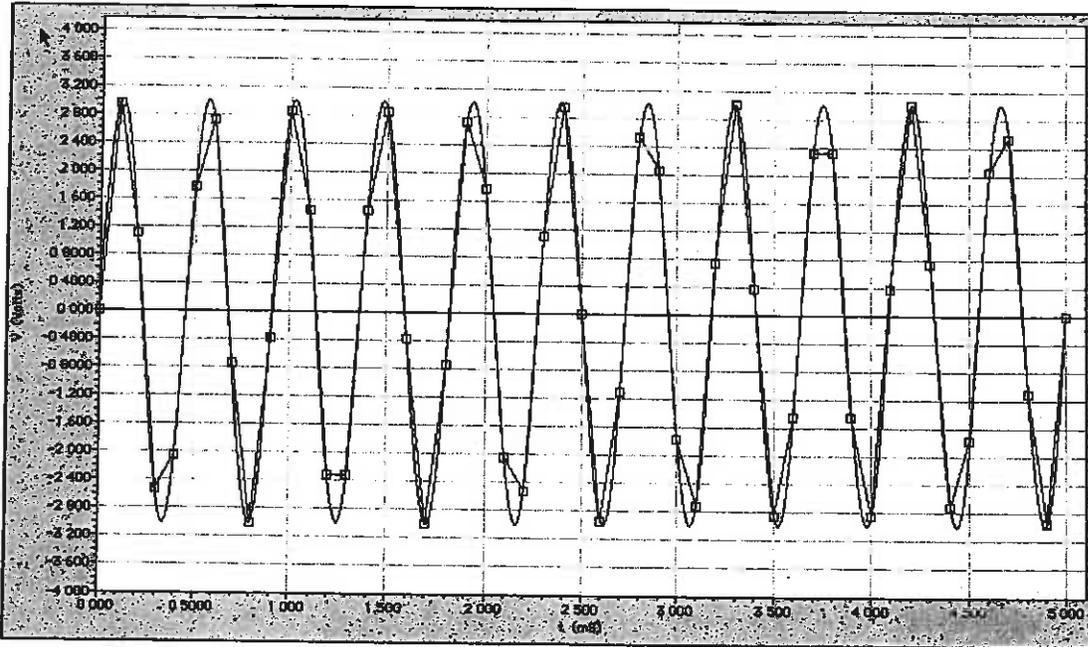


Fig. B1. The rough approximation that results when a 2.2 kHz sinusoidal waveform is sampled at 10 kHz

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.

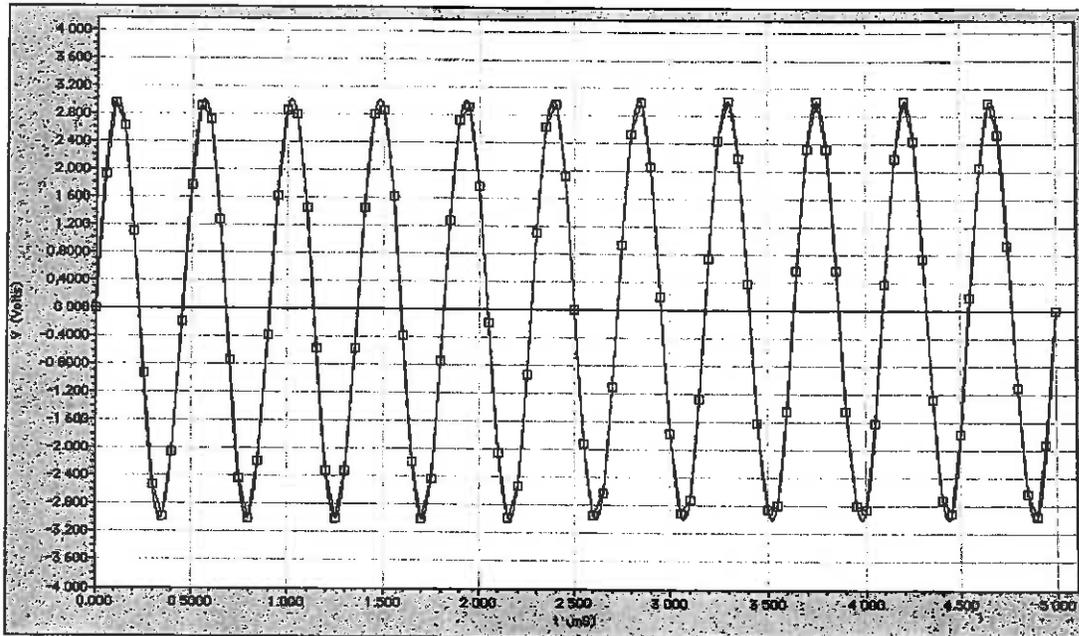


Fig. B2. The relatively smooth waveform that results when a 2.2 kHz sinusoidal waveform sampled at 20 kHz

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

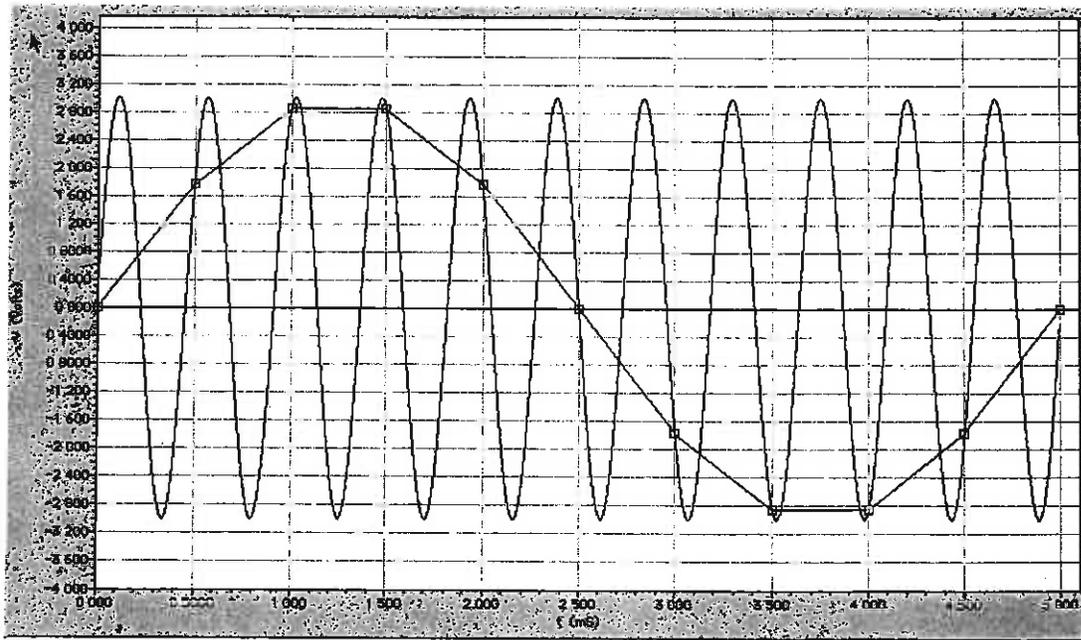


Fig. B3. Missing cycles that result when a 2.2 kHz sinusoidal waveform is sampled at 2 kHz, which does not meet the Nyquist criterion.

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. 18 on p. 22.)

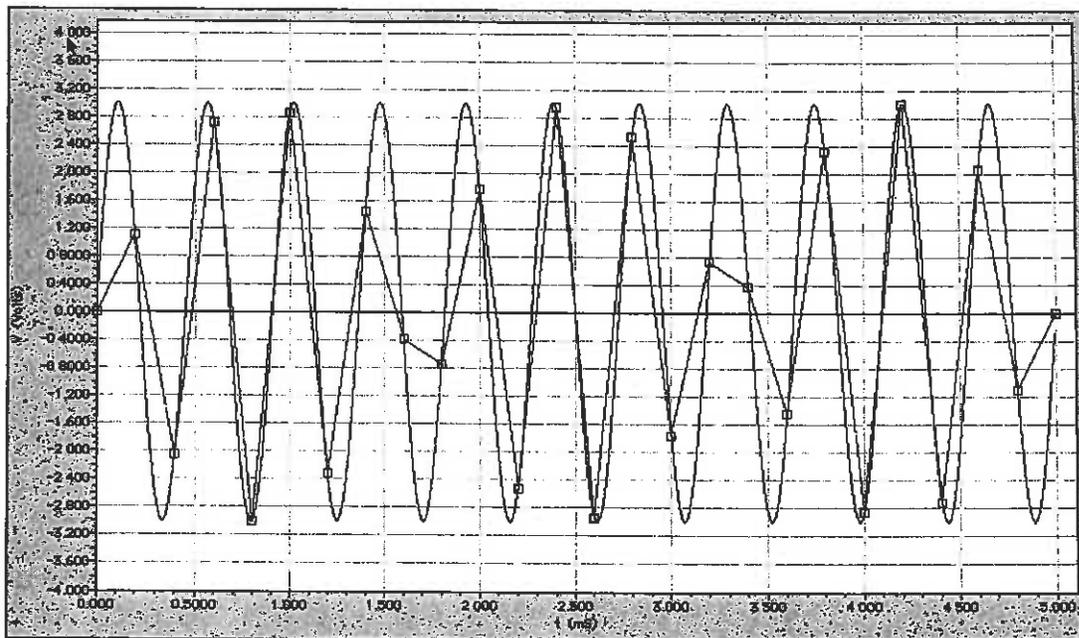


Fig. B4. A 2.2 kHz sinusoidal waveform sampled at 5.0 kHz. There is significant distortion even though the sampling frequency meets the Nyquist criterion.

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

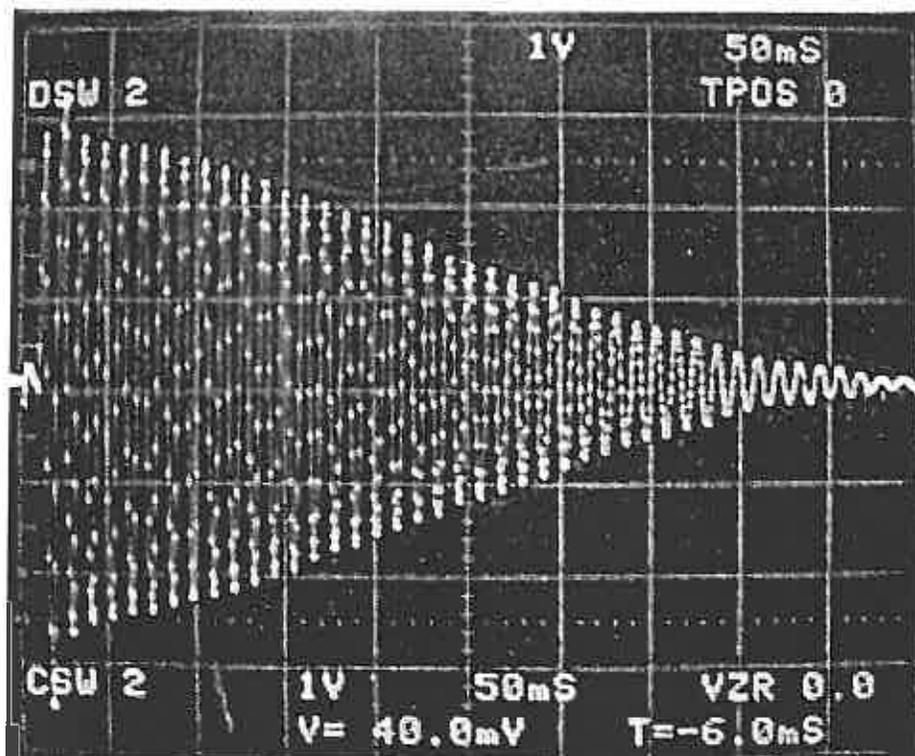


Fig. B5. Apparently smooth free precession signal at a frequency of 2.088 kHz. The sample frequency was 2 kHz.

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

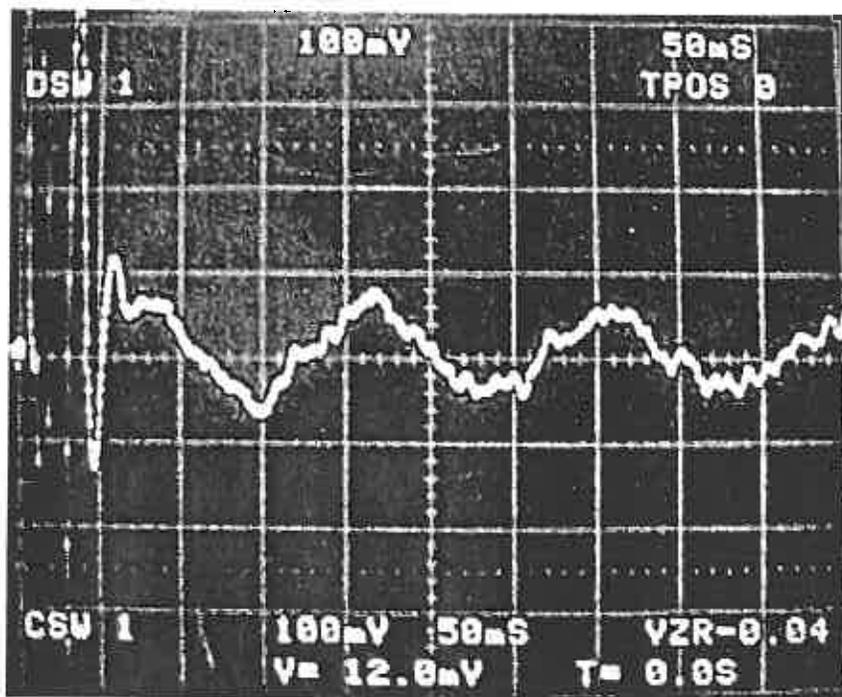


Fig. B6. 2.007 kHz fluorine signal from a 25-gram sample of C_6F_6 . The oscilloscope sample frequency was 2.000 kHz. The digitized waveform appears to have a frequency equal to the difference, 7 Hz.

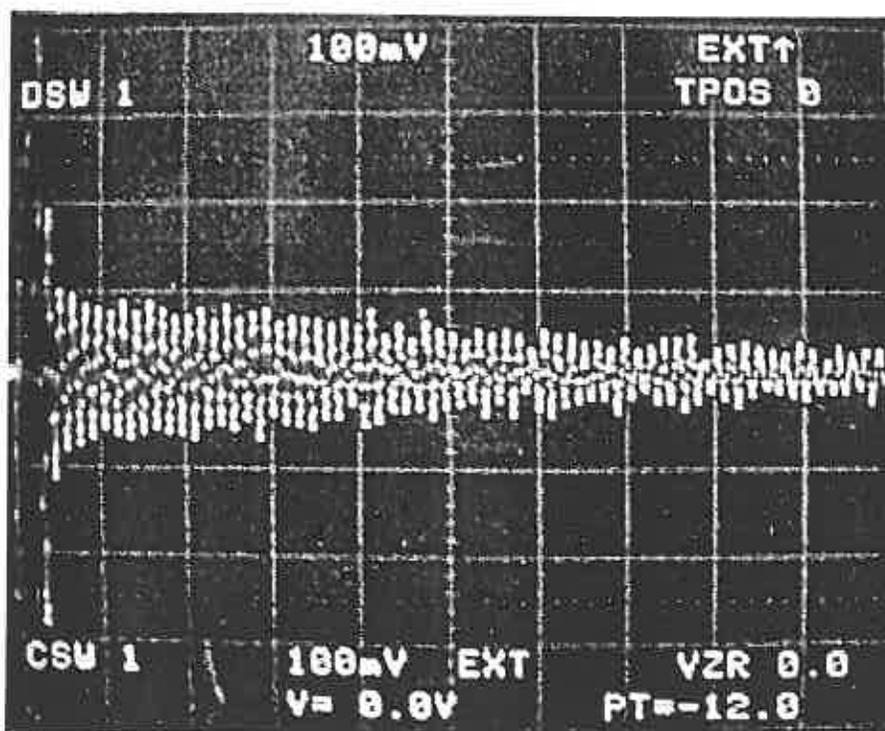
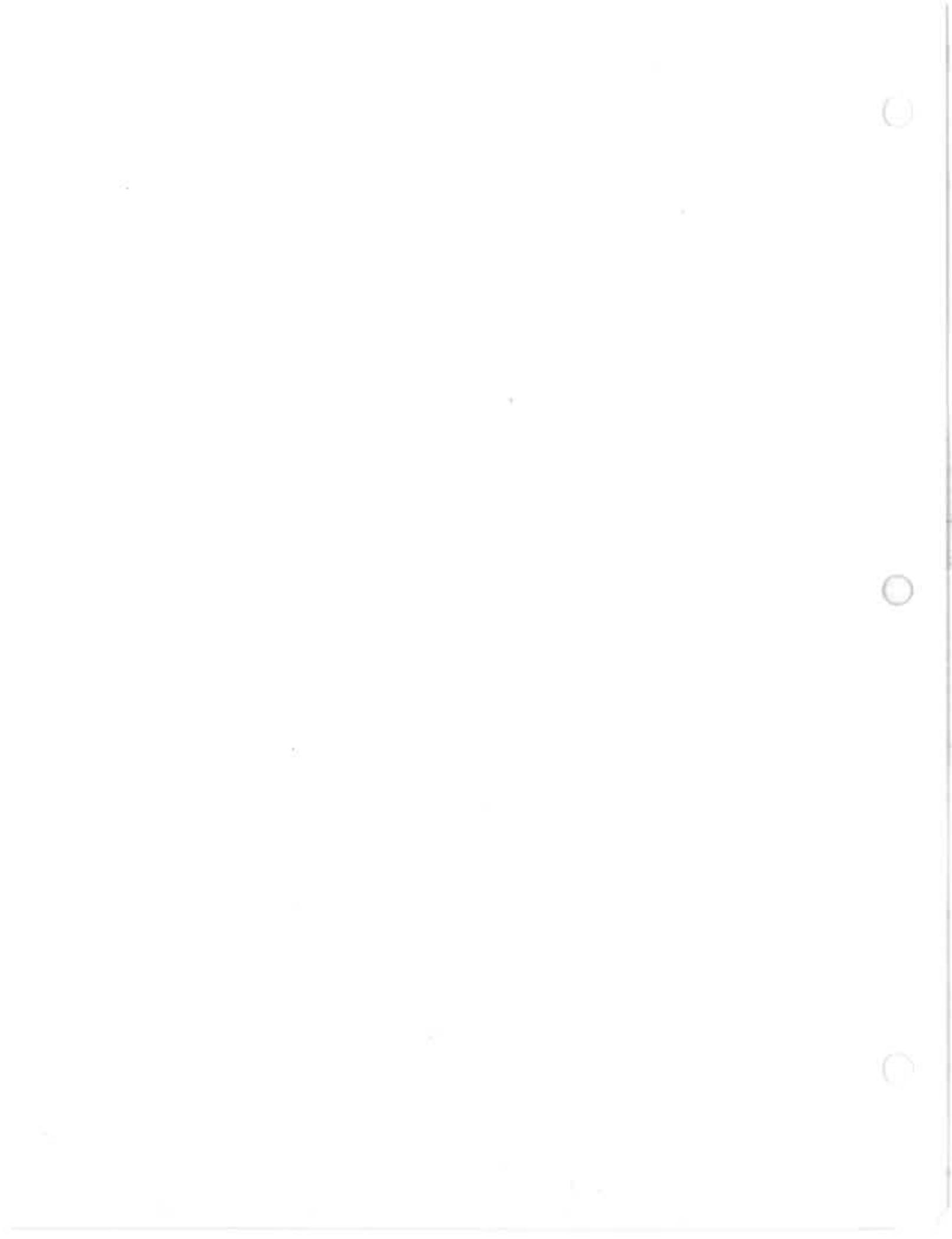


Fig. B7. Same as Fig. B6, except the oscilloscope sample frequency was reduced to 1.040 kHz. Since the oscilloscope displays 100 samples per division, the equivalent sweep speed is 96 ms/Div.

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

$$M_o = N \left(\frac{I+1}{I} \right) \frac{\mu^2}{3kT} B, \quad (1)$$

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

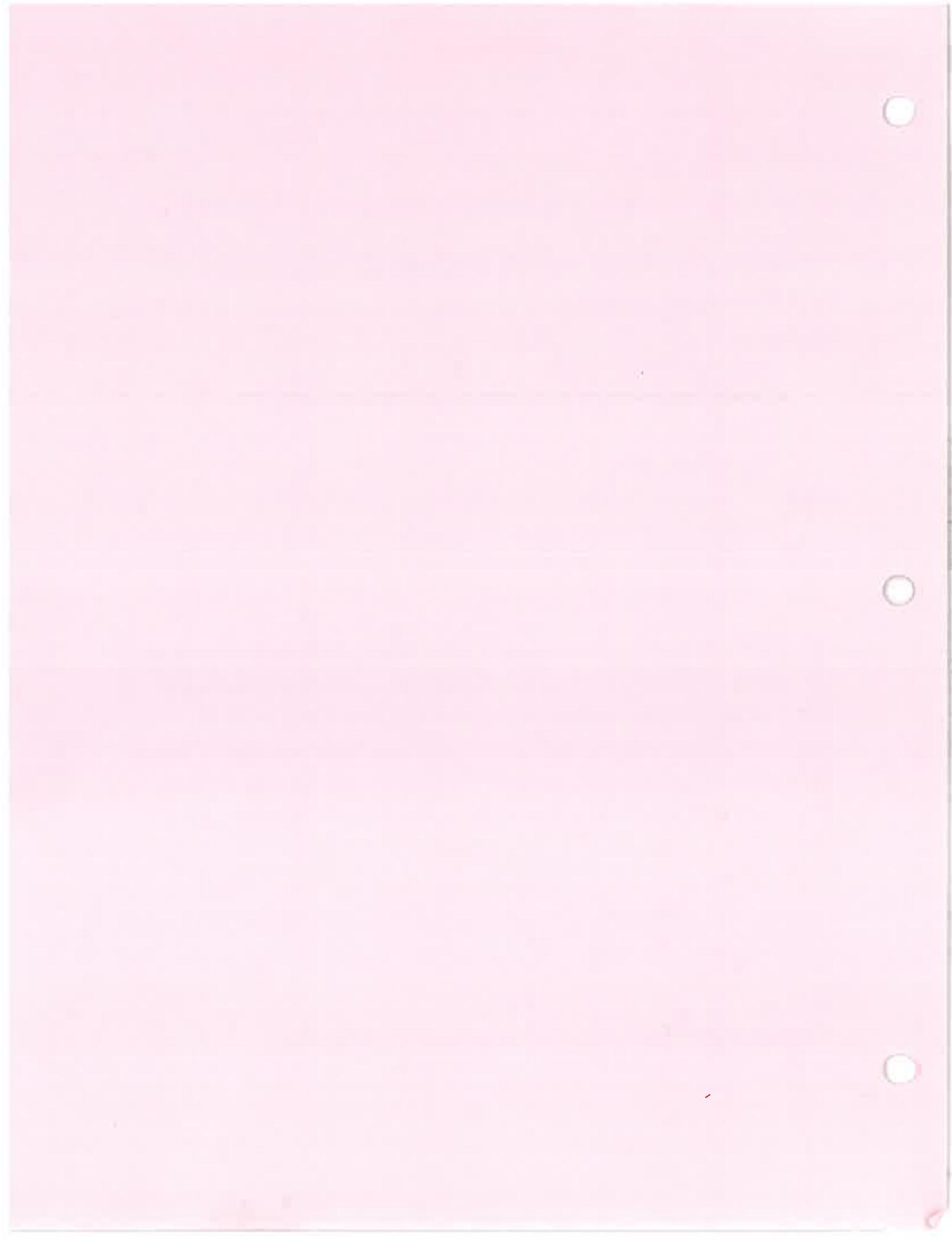
$$M(t) = M_o \left(1 - e^{-\frac{t}{T_1}} \right), \quad (2)$$

where M_o is the equilibrium Curie magnetization, and $M(t)$ is the magnetization at time t .

By rearranging Eq. (2) we obtain

$$M_o - M(t) = M_o e^{-\frac{t}{T_1}} \quad (3)$$

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



$$\ln(M_o - M(t)) = \ln(M_o) - \frac{1}{T_1} t. \quad (4)$$

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_0 and T_1 as adjustable parameters, to either of the data sets plotted in Procedure 8. Alternatively, one can assume that M_0 is approximately equal to the amplitude of the signal at the longest polarizing time (13.0 s in this case), and plot $M_0 - M(t)$ 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 $\ln(M_0 - M(t))$ 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

$$M_o = N \left(\frac{I+1}{I} \right) \frac{\mu^2}{3kT} B, \quad (1)$$

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

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

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 than 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_0 . (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 times 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.

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, 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.

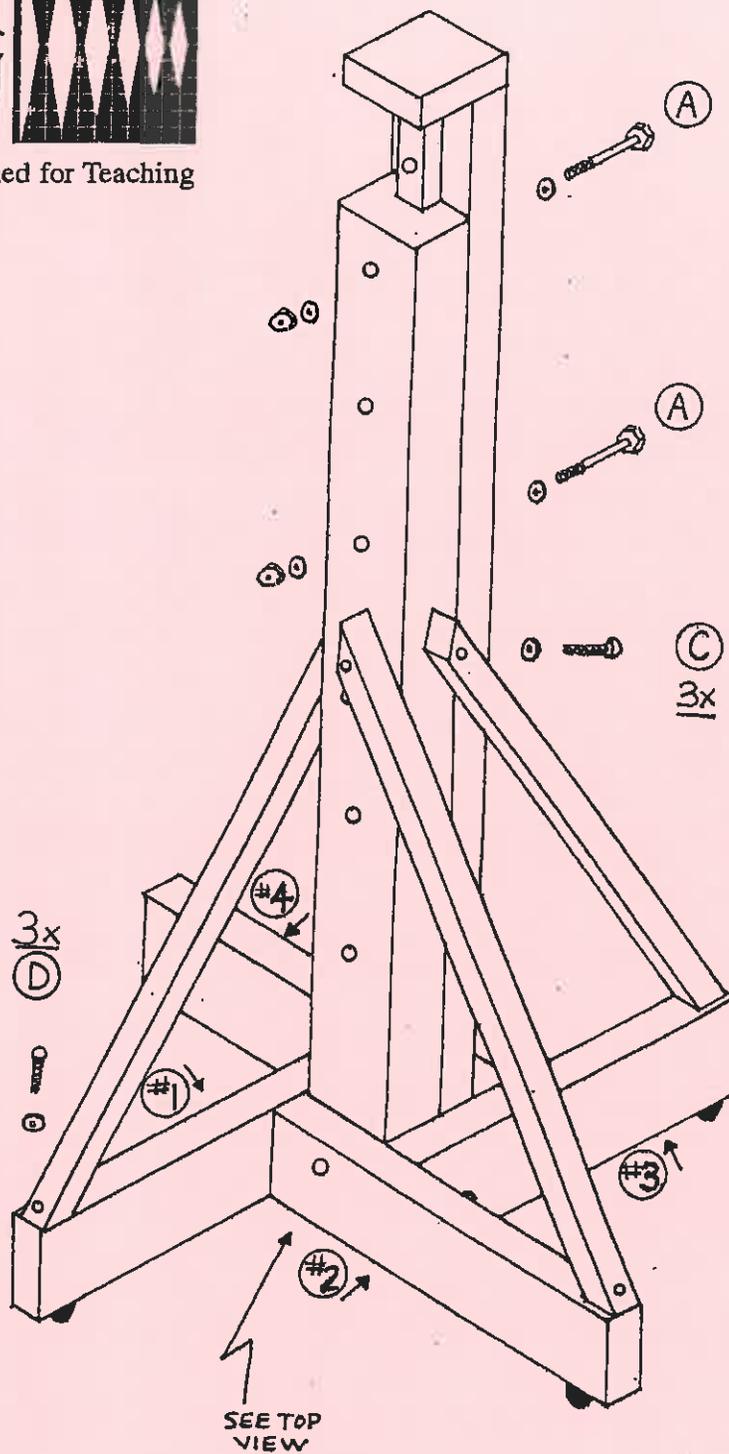
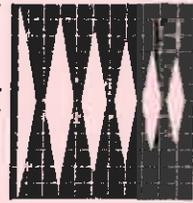
- 7) Once the power supply has been properly adjusted to deliver 3.0 A, you are ready to measure the amplitude of the free precession signal. The amplified free precession signal is available at the NMR SIGNAL OUTPUT. The output of the NMR AMPLITUDE DETECTOR is the NMR SIGNAL after it has been full-wave 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, 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.





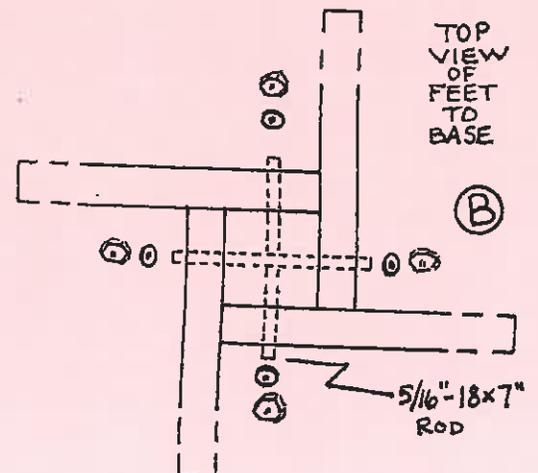
PARTS LIST:

- (A) 2- $\frac{1}{4}$ -20x3" HEX SCREWS
4- 14L WASHERS
2- $\frac{1}{4}$ -20 NUTS
- (B) 2- $\frac{5}{16}$ -18x7" THREADED RODS
4- 20L WASHERS
4- $\frac{5}{16}$ -18 NUTS
- (C) 3- $\frac{1}{4}$ -20x2" ROUND SCREWS
3- 14L WASHERS
- (D) 3- $\frac{1}{4}$ -20x1 $\frac{1}{2}$ " ROUND SCREWS
3- 14S WASHERS

NON-MAGNETIC STAND-

- 1- BASE
- 1- SLIDE
- 3- BRACES
- 4- FEET WITH RUBBER FOOT

NOTE: THE FEET ARE NUMBERED, 1-4, CORALATING TO THE BASE SIDES NUMBERED, 1-4. THE NUMBERS GO ON THE INSIDE, SO THEY ARE UNSEEN.



EARTH'S FIELD NMR NON-MAGNETIC STAND

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