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

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

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

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

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

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

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

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

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.

<u>Procedure</u>

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:

   <u>Variable Voltage but No Current Limiting</u>

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

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

Variable Voltage With Current Limiting

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

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

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

8) Trace out the magnetization curve by plotting the amplitude of the free precession signal versus polarizing time. On the same sheet, graph the output of the NMR AMPLITUDE DETECTOR versus polarizing time. Both curves should have the same shape.

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

EXPERIMENT 2: The Curie Law

Equipment

Earth's-Field NMR instrument

Polarization power supply (floating outputs, 40 volts maximum)

Oscilloscope

125-ml sample bottle

Theory

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

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

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

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

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

Procedure

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

Variable Voltage but No Current Limiting

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

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

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

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

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

9) Repeat Procedures 4-8 for polarizing currents of 2.5, 2.0, 1.5, 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.