5. Experimental Content

a. Observe NMR phenomenon of H-nuclei in water and compare NMR signals between the pure water sample (#5) and samples of water solution with some paramagnetic ions (such as samples #1, #2 and #6) as well as the organic Propanetriol sample #4.

b. Measure the magnetic field strength at the sample location with the Teslameter; measure the resonance frequency with the frequency meter, calculate the gyromagnetic ratio of the sample and compare it with the literature value; and verify the occurrence of NMR under Eq. (4). (For H-nuclei: γ_H=2.6752×10^8 Hz/T; for F-nuclei, γ_F=2.5167×10^8 Hz/T).

c. Based on the known gyromagnetic ratio of H-nuclei, put sample #1, #2, #5 or #6, adjust apparatus while observing the NMR signal on oscilloscope and recording the resonance frequency from frequency meter. Based on equation (4), calculate magnetic field strength B_0. Put sample #3 (hydrofluoric acid) at the same location in the magnetic field, adjust apparatus and observe the NMR signal of F-nuclei (**Note**: the NMR signal of F-nuclei is weak, so optimize the apparatus parameters carefully). From the acquired magnetic field strength B_0 and resonance frequency w_0, the gyromagnetic ratio γF, g-factor gF and nuclear magnetic moment μF of F-nuclei can be hence calculated.

d. If the gyromagnetic ratio of H-nuclei is known, by measuring the resonance frequency under resonance condition, the magnetic field strength can be calculated, so a Teslameter can be calibrated.

e. Use a sample with relatively strong NMR signal, such as #1 or #2, to observe signal coda; move the probe around in the magnetic field to understand the impact of magnetic field uniformity on coda waves.

f. Observe Lissajous graph: by sending the sweep signal and the resonant absorption signal to oscilloscope simultaneously using X-Y mode, symmetric waveform can be observed; by adjusting frequency and phase, two resonant peaks can overlap at the central position; under such condition, Equation (4) is satisfied.

6. Experimental Procedure

1) Preparation

a. Connect the sample probe to the socket on the side panel of the marginal oscillator, and insert one sample into the probe (usually sample #1 should be used first, i.e. water with copper sulfate);

b. Connect "Sweep" (sweep field) of the controller unit to the magnet unit on the corresponding terminals using the red-black wire pairs. Connect "Phase-shifted" on the back panel of the controller unit to CH1 of the oscilloscope using BNC cable. Connect the Teslameter probe to the "Teslameter" port on the back panel of the controller unit using the 5-core cable. Connect the marginal oscillator to the "Magnifier" port on the back panel of the controller unit using the 3-core cable.

c. Connect "CH2" of the marginal oscillator to CH2 of the oscilloscope using BNC cable. Connect "F-meter" of the marginal oscillator to the frequency meter using BNC cable. (**Note:** Read the manual of frequency meter in Section 9 carefully before using it).

Warning: double check for correct wirings.

d. Move the sample probe (with sample inside) into the magnetic field, and adjust its position to locate the sample at the central region of the magnetic field.

e. Turn on the power of the controller unit, preheat for a few minutes.

2) Acquisition of NMR signal

Multiple samples are provided. Since the resonance signal of sample #1 is the strongest, it should be used first by user to be familiar with related experimental operations, before other samples can be tested.

Warning: sample # 3 (HF acid) is a dangerous reagent that can cause serious eye, skin, or bone damage if not handled properly. Follow appropriate safety procedure when handling or refilling HF acid.

a. First turn the sweep "Amplitude" knob in count-clockwise direction to minimum and then turn in clockwise direction about one turn. The sweep field at this time can achieve maximum NMR signal observed on the oscilloscope for sample #1. But for measuring samples of F nuclei, it is recommended to turn sweep amplitude to maximum to achieve a large range for capturing NMR signals.

b. Adjust the "Coarse" knob of the marginal oscillator, and bring the frequency close to the resonance frequency of H-nuclei as shown on the magnet for reference purpose. Then adjust the "Fine" knob to finely change the frequency until the resonance condition described by Equation (4) is met and a NMR signal is observed, as shown in Figure 5. **Note**: the "Fine" knob should be adjusted slowly, as the resonant range is very narrow. Also, since magnetic field strength may vary due to temperature variations, the resonant signal should be sought around the given frequency within ± 1 MHz.

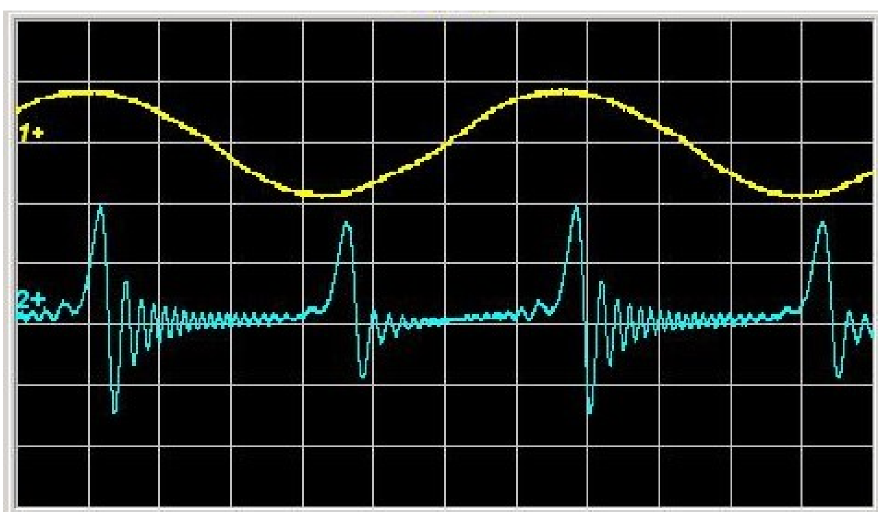

Figure 5 NMR signal observed on oscilloscope

c. After a preliminary resonance signal is obtained, the amplitude of the sweep source should be reduced while adjusting the frequency "Fine" knob to achieve a NMR signal with equal intervals. In the meantime, finely adjust the position of the sample in the magnetic field to achieve a NMR signal with the most coda waves.

d. Measure F-nuclei in HF sample. In theory, the resonance frequency of F-nuclei equals the resonance frequency of H-nuclei divided by 42.577 and multiplied by 40.055 (for example: if the measured resonance frequency of H-nuclei is 20.000 MHz, the resonance frequency of F-nuclei will be 20.000 MHz ÷42.577×40.055=18.815 MHz).

Place the HF sample (#3) in the probe, adjust the frequency close to the resonance frequency, and finely adjust the frequency to get the NMR signal. Since the resonance signal of F-nuclei is relatively small, it is helpful to increase the amplitude of the sweep field.

3) Observation of Lissajous figure

In above experiments, signals are observed on the oscilloscope using time scanning method, so signals are equal-interval sequences.

If the oscilloscope mode is set to X-Y, under resonance conditions, two symmetric waveforms can be observed on the oscilloscope, which correspond to the twice NMR occurrences within one sweep signal period, as seen in Figure 6. Finely adjust the frequency (marginal oscillator), the phase (phase shifter) and X-extension and X-position on the oscilloscope, make the two peaks of the two resonance signals overlap in the middle of the screen. Under such condition, resonance frequency and magnetic field satisfy the condition of $\omega_0 = \gamma \cdot B_0$.

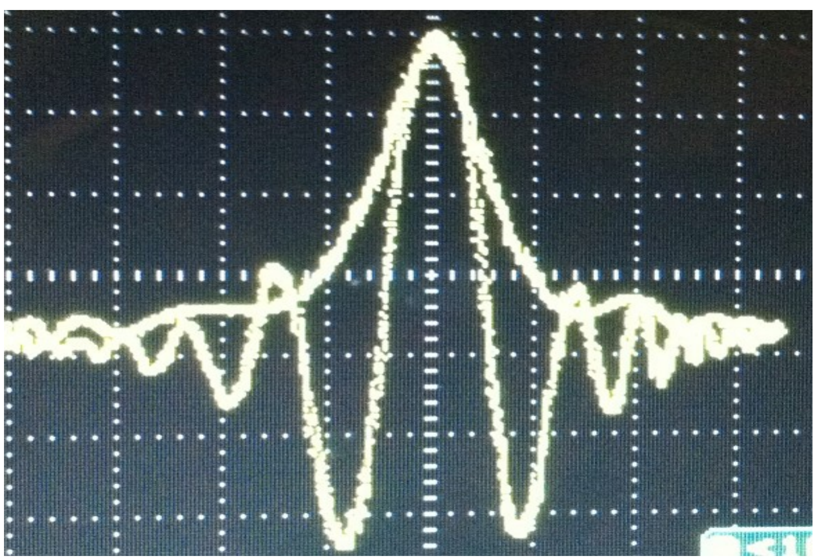

Figure 6 Lissajous figure observed on oscilloscope

4) Calibration of Teslameter

From Equation (4), if the gyromagnetic ratio of H-nuclei and the resonance frequency of a calibration sample are known, the magnetic field strength can be precisely calculated. This can be used for the calibration of a Teslameter..

First, place the Teslameter probe at a place in the absence of a magnetic field; adjust the "Zero" knob of the Teslameter to set it to zero. Then, insert the probe into the magnetic field at the sample position (**Note**: the Tesla probe should be orientated **perpendicularly** to the magnetic field, as otherwise inaccurate magnetic field reading would occur since the Tesla probe is a transverse type). Finally, adjust the "Cali." knob to set the Teslameter equal to the calculated value. Due to the high accuracy, NMR technique is one of the important methods for magnetic field calibration.

Note: before turning off the apparatus power, "Magnetizing", "RF. Amp." and "Amplitude" knobs should be turned all the way to the end in **counterclockwise** direction in order to extend the lifetime of this apparatus.

7. Examples of Data Recording and Processing

Note: Following data are for reference only, not the criteria for apparatus performance:

Experimental samples:

#1 $CuSO_4$ solution (for measuring H-nuclei) and #3 HF acid (for measuring F-nuclei)

Experimental method:

Assuming the gyromagnetic ratio of H-nuclei is known as, γ_N=2.6752×10^8 Hz/T. Use sample #1, adjust apparatus and achieve stable resonance signals; record the resonance frequency from the frequency meter; use Equation (4) to calculate the magnetic field strength at the sample location. Replace sample #1 with sample #3; place sample #3 at the same location in the magnetic field as sample #1; carefully adjust frequency and achieve resonance signal of F-nuclei; use the resonance frequency to calculate the gyromagnetic ratio γ_F, g-factor g_F and nuclear magnetic moment μ_F of F-nuclei.

Experimental results:

If the measured resonance frequency of H-nuclei is f_1=21.1493 MHz, based on Equation (4), the magnetic field strength at sample #1 is calculated as:

$$B_\circ = \frac{\omega_\circ}{\gamma} = \frac{2\pi \cdot f_1}{\gamma_N} = \frac{2\pi \cdot 21.1493 \times 10^6}{2.6752 \times 10^8} = 0.4967\,\text{T, i.e. 4967 Gauss}$$

After changing the sample, the measured resonance frequency of F-nuclei is f_4=19.8980 MHz, using the above magnetic field strength in Equation (4), the gyromagnetic ratio of F-nuclei is calculated as:

$$\gamma_F = \frac{\omega}{B_\circ} = \frac{2\pi \cdot f_4}{B_\circ} = \frac{2\pi \cdot 19.8980 \times 10^6}{0.4967} = 2.5171 \times 10^8\,\text{Hz/T}$$

Compared with the literature value γ_{FO}=2.5167×10^8 Hz/T, the error is very small.

Using $\mu_F = g_F \frac{\mu_N}{\hbar} P_F$ and $\mu_F = \gamma_F P_F$, we can get:

$$g_F = \gamma_F \hbar / \mu_N = 2.5171 \times 10^8 \times 1.0546 \times 10^{-34} / 5.0508 \times 10^{-27} = 5.2557$$

Since $\mu_F = g_F I \mu_N$ (I is the spin quantum number, for F-nuclei, I=1/2), we get:

$$\mu_F = 5.2557 \times 0.5 \times 5.0508 \times 10^{-27} = 13.2727 \times 10^{-27}\,\text{J/T}$$

Changing to unit of nuclear magneton, we have $\mu_{F'} = \frac{\mu_F}{\mu_N} = \frac{13.2727 \times 10^{-27}}{5.0508 \times 10^{-27}} = 2.6278$, while the literature value is $\mu = 2.6273$.

8. Precautions

1) The sweep amplitude is small when observing the resonance signal of hydrogen nucleus, while relatively large for fluorine nucleus.
2) In order to reduce interference, the power socket of this apparatus must have good grounding.
3) Since the detection coil is both a transmitting coil and a signal receiver, it is easily affected by the surrounding environment, so there should be no obvious high-frequency signals and radio interference sources around the laboratory.
4) Handle samples with care, especially HF samples. Remember not to uncover the sample cap of the tube by students.
5) The internal components of the magnet are strong magnetic neodymium iron boron. It is not allowed to place ferromagnetic substances inside the magnet.
6) The six screws on both sides of the magnet must not be twisted at will, otherwise it will affect the experimental results.
7) The surfaces of the electromagnet poles are polished, so avoid scratches or rust to the surfaces. Otherwise, the uniformity of the magnetic field would degrade.