5. Setup and Operation

1) Description of front panel

Constant magnetic field: This apparatus adopts an electromagnet as the excitation magnetic field whose strength is proportional to the excitation current in the electromagnet coil. Thus, magnetic field strength can be changed by adjusting the excitation current in a range from 0 to 3 A using the corresponding "Adjust" knob, read by the digital ampere-meter (A) on the front panel. The current output is via the terminals "+" and "-" under "Magnet Field".

Sweep field: The Sweep Field "Adjust" knob is used to adjust the current flowing through the sweep coil to change the amplitude of the sweeping field. It can vary the width of the resonance signals in the horizontal direction and change the number of coda waves. Usually, this knob is turned all the way to the end in clockwise direction to set the sweeping current at maximum for the best observation of the resonance signal. The sweeping current is displayed on the corresponding digital ampere-meter, and the current output is via the terminals "+" and "-" under "Sweep Field".

Phase shift: The "Phase Adj." knob is used to adjust the phase of the sweeping signal (50/60 Hz) to the X-axis of the oscilloscope in order to change the relative positions of the butterfly-alike resonance signals on the oscilloscope.

Marginal oscillator control: The "Marg. Adj." knob is used to adjust the amplitude of the marginal oscillator. Usually, higher marginal amplitude (i.e. turning the "Marg. Adj." knob in clockwise direction) may bring a stronger NMR signal for easy observation. But, when the marginal amplitude is too high, it may cause self-excited oscillation of the RF coil. In this case, one needs to reduce the amplitude to avoid this phenomenon.

Marginal frequency adjustment: The "Freq. Adj." knob is used to adjust the oscillation frequency of the marginal oscillator.

2) Operation

Refer to Figure 2 to wire the system. Insert the sample probe into the gap of the electromagnet poles carefully. Connect the provided sample cable to the "Sample" terminal (BNC) on the electronic unit. The oscilloscope used in this experiment should have the external scan (X-Y) working mode. Connect the input of a digital frequency counter (40 MHz or above) to the "Freq." terminal (BNC) on the front panel of the main apparatus.

Warning: ensure correct wirings of the magnetic field and the sweep field to prevent damage to the electromagnet and the system ("+" to "+" and "-" to "-", respectively for magnetic field and sweep field), and ground the electromagnet by connecting the post with a grounding sign to the grounding point of the lab properly.

Set the oscilloscope at Y-T mode. Set X-axis sensitivity between 2~5 V/DIV and connect the "X" terminal of the oscilloscope to the "X" terminal (BNC) on the electronic unit. Set Y-axis sensitivity of the oscilloscope between the 0.1~2 V/DIV and connect it to the "Y" terminal (BNC) on the electronic unit.

Turn on the power, and adjust the magnetic field "Adjust" knob to set the constant magnetic field current between 1.2 A and 2.0 A for example. Next, turn the sweep field

"Adjust" knob clockwise to maximum to set the sweeping field current between 0.3 A and 0.7 A as displayed by the current meter while monitoring the scan line on the oscilloscope.

If a non-zero value displayed on the digital frequency meter, the marginal oscillator is functioning. Otherwise, adjust the "Marg. Adj." and "Freq. Adj." knobs until a non-zero value is displayed by the digital frequency meter. Further adjust the "Freq. Adj." knob again until a NMR signal is observed on the oscilloscope (**Note:** during this procedure, may alternatively adjust "Marg. Adj." and "Freq. Adj." to keep the marginal amplitude at a adequately high level but avoid self-excited oscillation). Finally, finely adjust "Marg. Adj." and "Freq. Adj." knobs while adjusting the probe position until an optimal resonance signal is observed. Figure 7 shows an example of both the sweep signal and the resonance signal shown on the oscilloscope screen.

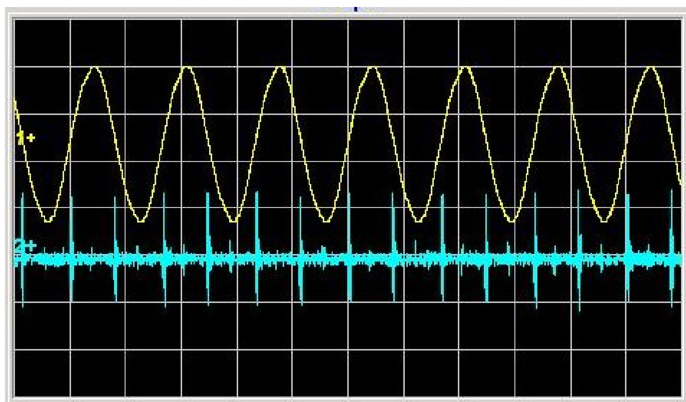

Figure 7 Observed NMR signal of PTFE

When the oscilloscope is changed to X-Y mode, the relative positions of the two resonance signals can be changed by adjusting the "Phase Adj." knob. If the oscilloscope is set at the internal trigger mode (Y-T mode), the "Phase Adj." knob has no effect.

During experiment, in the presence of possible low-frequency interference in the environment, it is recommended to wire and ground the outer shells of the units. After changing the sample or the oscillation frequency, the working status of the marginal oscillator needs to be re-adjusted by using the "Marg. Adj." knob.

The location of the sample probe has been pre-adjusted at factory (e.g. a mark sign), so it normally does not need to be re-adjusted.

This apparatus includes two sample probes (liquid and Fluoride). For using the liquid probe, screw out the cap on the tip, inject liquid to the sample probe with a syringe till full, and screw back the cap to seal.

Prior to delivery, this apparatus was tested and one set of the optimal parameters were achieved (for reference purpose only): (1) for ^{1}H sample, frequency at 11.472 MHz and magnetic current at 1.570 A, (2) for ^{19}F sample, frequency at 11.432 MHz and magnetic current at 1.677 A.

Note: before turning off the power, both "Adjust" knobs of the magnetic field and the sweep field should be turned all the way to the end in **counterclockwise** direction in order to extend the lifetime of this apparatus.

6. **Experimental Content**

1) Understand the classical theory of NMR
2) Learn how to experimentally study NMR phenomena
3) Observe NMR signal of proton (H-nuclei), and measure magnetic field strength
4) Determine gyromagnetic ratio, *g*-factor, and nuclear magnetic moment

7. **Experimental Procedure**

1) <u>Water sample: observe ^{1}H NMR absorption signal and measure magnetic field strength</u>

Pull out the protective plastic tube from the liquid probe and inject test sample. Loosen the small locking nut of the sample probe hole on the magnet unit, insert the liquid probe into the hole with depth to the marked line on the probe, and lock the nut. Note, the larger locking nut is used to adjust the height of the probe. Connect the BNC plug of the liquid sample probe to the "Sample" terminal on the electronic unit.

Turn on power, adjust the marginal oscillator to be oscillating at a certain frequency applied to the sample coil.

According to Eq. (4), NMR can be achieved by changing the strength of the constant magnetic field with a fixed angular frequency of the sweep field, or changing the angular frequency of the sweep field with a fixed magnetic field, or changing both of them.

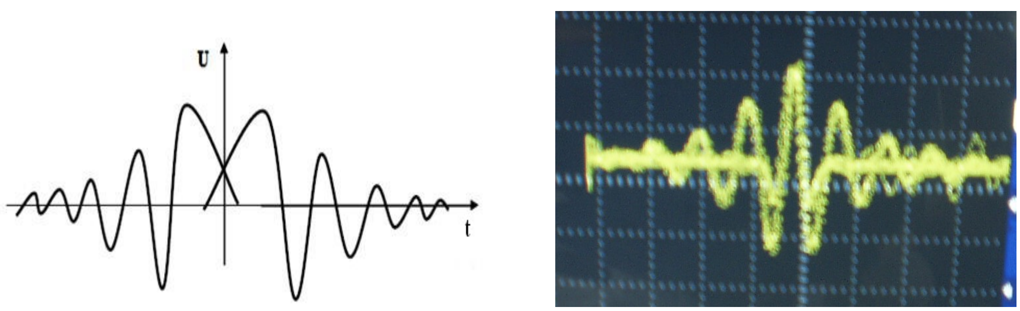

Figure 8 Butterfly-shape signal on oscilloscope

"Sweep field" outputs a signal at 50/60 Hz, which is split into two paths. One is sent to the sweep coil on the electromagnet and the other goes to terminal "X" through an internal phase shifter and is connected to the X-axis of the oscilloscope for synchronized scanning between the two paths. When NMR occurs, two symmetric butterfly-alike waveforms can be observed on the oscilloscope as shown in Figure 8 (left) if the oscilloscope is working at X-Y mode, corresponding to two NMR events in one period of the sweep magnetic field B_m. Finely adjust the phase shifter as well as the strength of the constant magnetic field or the angular frequency of the sweep field to let the two peaks of the waveforms overlap at the center on the oscilloscope as seen in Fig. 8 (right). Now, Eq. (4) is fully satisfied.

To measure the strength of the constant magnetic field, set the oscilloscope at the Y-T scanning mode with the X-axis and Y-axis sensitivities at 5 ms/div and 0.1-0.5 V/div, respectively. Now, resonance absorption signals can be observed on the oscilloscope as seen in Figure 7.

Finely tune the "Freq. Adj." knob until an equal interval of 10 ms (in case of 50 Hz AC frequency) or 8.33 ms (in case of 60 Hz AC frequency) between adjacent signals is observed. The principle is described in Section 4.

Now, record frequency F_H of the frequency meter, which is the frequency of the sweep field under NMR. Since the gyromagnetic ratio of a proton is known (γ_H=2.67522×10^2 MHz/T), the strength of the constant magnetic field (B_0) can be calculated as:

$$B_0 = \frac{w}{\gamma_H} = \frac{2\pi F_H}{\gamma_H} \tag{6}$$

where the unit of frequency is MHz and the unit of magnetic field strength is Gauss.

2) PTFE sample: observe ^{19}F NMR phenomena, determine gyromagnetic ratio, *g*-factor and nuclear magnetic moment

Note: the NMR signal of F^{19} nuclei is relatively weak, careful adjustment is needed.

The experimental procedure is similar to that described above.

The gyromagnetic ratio of ^{19}F (γ_F) can be calculated as:

$$\gamma_F = 2\pi \frac{F_F}{B_F} = \frac{F_F \gamma_H}{F_H} \tag{7}$$

where the gyromagnetic ratio of a proton (γ_H) is known, and F_F and F_H are the NMR frequencies of ^{19}F and ^{1}H, respectively, with identical magnetic field strength set for both cases (B_H=B_F=B_0).

In general, the nuclear magnetic moment can be expressed as

$$\mu = \frac{2\pi g P \mu_N}{h} = \gamma P \tag{8}$$

So we get the *g*-factor of a ^{19}F nucleus as

$$g_F = \frac{\gamma_F h}{2\pi \mu_N} \tag{9}$$

where h is the Planck's constant (h=6.62608×10^{-34} Js), and μ_N is the Bohr nuclear magnetic moment (μ_N=5.0579×10-27 J/T).

Since P_F=$I_F h/2\pi$, from Eq. (8), we get the nuclear magnetic moment of ^{19}F nuclei as

$$\mu_F = g_F I_F \mu_N \tag{10}$$

where I_F is the spin quantum number of ^{19}F nucleus (I_F= ½).

Note: other samples such as Glycerol, $FeCl_3$ or $CuSO_4$ solution can also be used. In fact, $CuSO_4$ solution (concentration around 1%) has a stronger NMR signal than that of pure water or PTFE. For this reason, it is recommended that CuSO4 solution should be used first for an easy observation of experimental phenomenon.