

## 5. Experimental Content

- 1) Understand the basic physical theory and experimental configuration of a PNMR system. Learn to explain related physical phenomena in PNMR using classical vector model.
- 2) Learn to use signals of spin echo (SE) and free induction decay (FID) to measure  $T_2$  (spin-spin relaxation time). Analyze the influence of magnetic field homogeneity on NMR signal.
- 3) Learn to measure  $T_1$  (spin-lattice relaxation time) using reverse recovery.
- 4) Qualitatively understand the relaxation mechanism, observe the effect of paramagnetic ions on nuclear relaxation time.
- 5) Measure  $T_2$  of copper sulfate solution at different concentrations. Determine the relationship of  $T_2$  with the change of concentration.
- 6) Measure the relative chemical displacement of the sample.

## 6. Experimental Procedure

### 1) Apparatus connection

For the RF Transmitting Unit (on the back panel):

Use the **WHITE (note: must be the white serial cable)** 9-core serial cable to connect the "Signal" port to the serial port of the computer. Use the 2-core cable to connect "Mod Field" port to the "Mod Field" port of the constant temperature unit. Connect the "Amplifier Power" port to "Amplifier Power" port of the constant temperature unit using the 5-core cable. Connect the "RF Signal (O)" port to the "RF Signal (I)" port of the constant temperature unit using the lockable BNC cable. Finally plug in the power cord.

For the Signal Receiving Unit (on the back panel):

Use the **BLACK (note: must be the black serial cable)** 9-core serial cable to connect the "Cont. Temp." port to the "Cont. Temp" port of the constant temperature unit. Use the 4-core cable to connect "Heater Power" port to the "Heater Power" port of the constant temperature unit. Use a BNC cable to connect "Pre-Amp (I)" port to the "Pre-Amp (O)" port of the constant temperature unit. Use the BNC to audio converting cable to connect the "Resonance Signal" port to the audio/microphone socket of the computer. Plug in the power cord.

### 2) Warm-up the apparatus

Turn on the power switches of the two electric units. The current electromagnet temperature is displayed on the Constant Temperature Unit, which is generally equivalent to the local indoor temperature at that time. The temperature increases after a period of time, which shows that the heater is working. After 3 - 4 hours (the time may be different at different seasons or different locations), the temperature will be controlled and stable at 36.50 °C. (Sometimes it changes between 36.44 °C and 36.56 °C, which is normal).

Open the acquisition software, click the "Continuous Acquisition" button, the computer controls the RF signal. The frequency is generally 20.000 MHz. The initial values are generally set at: pulse interval 10 ms, first pulse width 0.16 ms, second pulse width 0.36 ms. Then carefully adjust "Mod Field" amplitude (on the RF Transmitting Unit) to change the magnetic

field with a small range. When it is adjusted to an appropriate value, the FID signal can be observed in the acquisition software interface (spin echo signal can also be observed when adjusted properly), then adjust the “Cont. Field” (for magnetic field uniformity”) on the receiving unit, the change of the tail wave of FID signal can be observed.

### **3) Measurement of apparent lateral relaxation time $T_2^*$**

Adjust the pulse interval to the maximum (60 ms), and adjust the second pulse width to 0 ms. Only the first pulse is kept, carefully adjust the “Mod Field” and “Const Field” using both Coarse and Fine knobs, and adjust the first pulse width within a small range (adjust around 0.16 ms) to maximize the tail wave. Use the software to measure the apparent lateral relaxation time  $T_2^*$  through exponential fitting. Change different samples (such as glycerol samples, oil samples, etc.) for comparison and record their values.

### **4) Measurement of lateral relaxation time $T_2$ with spin echo (SE signal)**

On the basis of the previous step, find the pulse width of  $90^\circ$  pulses (as the first pulse). Adjust the pulse interval to 10 ms, and adjust the second pulse width to twice the first pulse width (for the actual apparatus, it is not exactly twice width relationship) as the  $180^\circ$  pulse, carefully adjust the “Mod Field” and “Const Field” using both Coarse and Fine knobs to maximize the spin echo signal.

Using the software to measure the echo signal size at different pulse intervals, do exponential fitting to obtain the lateral relaxation time  $T_2$ , which is compared with the apparent lateral relaxation time  $T_2^*$  to analyze the effect of magnetic field uniformity on the lateral relaxation time. Change different samples for comparison.

### **5) Measure the lateral relaxation time of hydrogen nuclei in copper sulfate solutions of different concentrations and analyze the relationship between the relaxation time and the concentration change. (Optional)**

The measurement process is the same as the previous step. Measure the transverse relaxation times of five different concentrations of copper sulfate solutions. Use fitting method to find their relationship.

### **6) Learn to measure the longitudinal relaxation time with the inversion recovery method**

The inversion recovery method is to use the  $180^\circ - 90^\circ$  pulse sequence to measure the longitudinal relaxation time  $T_1$ . The method is similar to the spin echo method. Adjust the first pulse as the  $180^\circ$  pulse, adjust the second pulse as  $90^\circ$  pulse, change the pulse interval to measure the amplitude of the coda wave. Do curve fitting to get the longitudinal relaxation time  $T_1$ .

### **7) Measure the relative chemical shift of the sample**

On the basis of achieving the FID signal of glycerin, replace it with the xylene sample. Use the software to analyze the relative chemical shift of xylene (the frequency difference between the two peaks of the xylene spectrum is about 100 Hz)