Some Guidelines for the Nuclear Magnetic Resonance Experiment

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The Nuclear Magnetic Resonance (NMR) experiment illustrates some important principles of spectroscopy that can be applied to a variety of systems operating at much higher frequencies. NMR has also become an important medical tool for non-invasive imaging of the human body. To avoid the stigma and fear of all things nuclear, this technique has been relabeled "Magnetic Resonance Imaging" or MRI. The apparatus for this experiment, manufactured by TeachSpin, has the controls and signal outputs that allow the complete exploration of the nuclear resonance phenomenon. The following guidelines are meant to help you get a basic quantitative understanding of NMR. By executing them, step by step, you should be able to get a good idea of how all this works.

1. The magnetic resonance phenomenon can be understood by analogy to classical mechanical systems as described in the TeachSpin manual, particularly pages 2-11. Since magnetic resonance is more deeply represented by quantum mechanics, it is suggested that you also read H. Haken & H. C. Wolf, *The Physics of Atoms and Quanta*, sections 20.6, 20.7, then 14.4 and 14.5.

2. The next step is to become familiar with the equipment that will be used. There are basically three components: (1) the actual NMR spectrometer which is enclosed in a metal box housing a modest permanent magnet assembly and crossed transmitter and receiver coils that surround the sample volume, (2) the control and detection electronics modularly housed in a common power supply case, and (3) an Agilent 54642A 500 MHz oscilloscope for monitoring the various control and detection signals. These are illustrated in the TeachSpin manual as Figure 1.2 (page 15), Figure 11.2 (page 24) and Figure 1.3 (page 29). (Consistency of document organization is apparently not a high priority.) Follow the TeachSpin manual, starting at page 26 to gain familiarity with the various controls. Ignore section A.3 (Multiple Pulse Sequence) but continue through page 28. Note that at the present time, the tuning capacitor on the 15 MHz receiver module is broken so leave it alone.

3. The Larmor precession frequency is given by the equation, $\nu_0 = \gamma_p B_0/2\pi$, where γ_p is the proton gyromagnetic ratio and B_0 is the static field in the magnet gap, about 0.36 Tesla (3600 Gauss) for this particular gadget. (A table of useful constants is included at the end of this document.) You now need to find out the intensity of the pulsed RF field, B_1, that induces the spin transitions. There are two sample probes with somewhat similar geometries (see page 25b). For the purpose at hand, select the *Pickup Probe*. Insert the coil in the sample volume and connect to the oscilloscope. With the RF Power modulated by an *A Pulse*, you can expect a fairly hefty signal on the scope. Rotate the probe within the sample volume to maximize the signal amplitude. Note the orientation of the coil and compare with Figure 11.2 to check your understanding of the NMR geometry. TeachSpin claims that the pickup probe coil has a diameter of 6 mm. Use that

information to compute the amplitude of the pulsed RF field (a Physics 240 problem). That is not quite the same as B_1. Remember that the oscillatory field that drives the spin transitions is one of the two rotating components that sum to the RF field that is confined along just one axis of the spectrometer. Thus, the amplitude of B_1 is exactly half of the value just calculated. This allows you to compute the durations of the pulses required to rotate the magnetization through $\pi/2$, $3\pi/2$, or $5\pi/2$ radians using the relation, $\theta = \gamma_p B_1 t$.

4. Follow the instructions in the TeachSpin manual on pages 29 and 30 to observe the Free Induction Decay (FID). Use the estimated value for B_0 given above as an initial starting point for finding the natural precession frequency, ν_0. This can be determined relatively accurately by minimizing the beat frequency observed from the *Mixer Out* port.

5. Tweak the *A-WIDTH* control knob to find the successive maxima of the Free Induction Decay signal (from the *DETECTOR OUT* port) corresponding to the $t_{\pi/2}$, $t_{3\pi/2}$, $t_{5\pi/2}$ pulses. For each value, you will need to patch to the *A+B OUT* port to measure the corresponding time with the oscilloscope. (Note that the scope has a cursor that will enable rather accurate time measurements.) Compare with the calculations you performed in section 4 above.

6. Make sure that the *Mixer Out* signal is really the beat frequency between the natural precession frequency, ν_0, and the applied drive frequency, ν_1. Vary ν_1 starting from near the resonance frequency, ν_0, and compare the *Mixer Out* signal frequency with $|\nu_0 - \nu_1|$.

7. The dynamics of the rotation of the bulk magnetization can be understood in terms of a simple mechanical model, the harmonically forced motion of a simple oscillator. (Think of a playground swing pushed by a klutzy parent.) In the situation at hand, the magnetization is driven by a sinusoidal force of constant amplitude of fixed duration. The number of cycles is simply given by $\nu_1 t$. If $|\nu_1 t - \nu_0 t| = n$, $n \neq 0$, the total torque imparted to the system will be zero. The algebra for this is worked out in detail in the appendix to this note. For *A pulse* lengths corresponding to $t_{\pi/2}$ and $t_{5\pi/2}$, vary ν_1 to find several successive minima in the FID amplitudes. Compare to the predictions provided by the model described above.

8. Find the magnetic field "sweet spot" by cranking the sample holder along the two axes perpendicular to the static field direction. The best position will be found when the apparent FID signal has the longest decay time. (Hint: it is not at the coordinates specified by 0, 0.) Map the magnetic field using the mineral oil sample and twiddling ν_1 to minimize the beat frequency and thus determine ν_0.

9. Measure the spin-lattice relaxation time, T_1, using the π pulse – delay – $\pi/2$ pulse sequence as described on page 33 (Two Pulse – Zero Crossing). Note that after the π pulse, the system will approach thermodynamic equilibrium according to

the formula, $M_z(t) = M_0 (1 - 2 \exp(-t/T_1))$. The factor of two occurs because the magnetization must decay from $-M_0$ back to $+M_0$. Thus, you should find a value for t, τ_0, where the FID signal goes to zero. τ_0 is related to T_1 in a fairly obvious way.

10. Measure the spin-spin relaxation time, T_2, using the $\pi/2$ pulse – delay - π pulse – delay sequence as described on page 34. Verify that this method overcomes problems with magnetic field non-uniformities by also trying this out with the sample intentionally moved to a relatively rapidly varying field region. In fact, you may find that this will be the best way to deal with samples with small values of T_2.

11. In order to see how NMR might be useful in a medical context, find some varieties of biological material and compare T_1, T_2. I would suggest lunch meat and fingernail clippings but you're free to make your own selections. Just make sure no one would be tempted to eat the samples.

Experimental Pitfalls:

1. Make sure the RF power cable with the TNC connector is properly connected to the *RF OUT* jack on the *15 MHz OSC/AMP/MIXER* module before turning on any electrical power.

2. The *REPETITION TIME* controls must be set so that the sample can completely return to thermodynamic equilibrium before the next series of pulses are generated.

3. Make sure that nothing gets accidentally spilled or dropped into the spectrometer housing. Be especially careful to avoid putting any magnetic (ie. steel) objects in the vicinity of the spectrometer box.

Appendix

Forced Harmonic Oscillation

free harmonic oscillator: $m\ddot{x}(t)+kx(t)=0$

forced harmonic oscillator: $m\ddot{x}(t)+kx(t)=F_{ext}(t)$

Assume: $m\ddot{x}(t)+kx(t)=\delta(t-t')$

Then: $x(t)=\frac{1}{m\omega_0}\sin(\omega_0(t-t'));\ \omega_0^2=\frac{k}{m}$

$$G(t,t')=\frac{1}{m\omega_0}\sin(\omega_0(t-t'));\ t>t' \text{ (Green's Function)}$$
$$=0\ ;\ t\le t'$$

For general forcing functions:

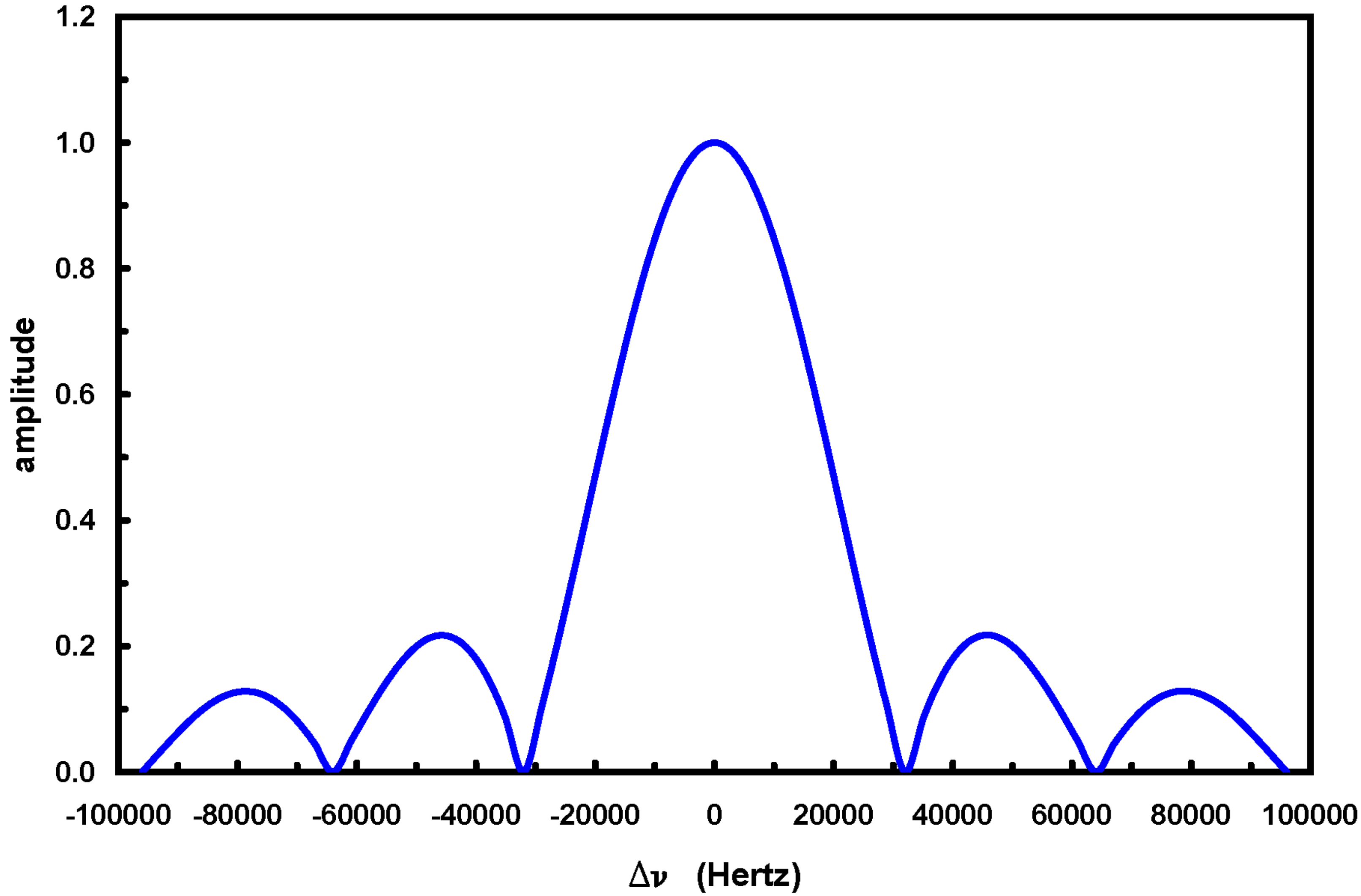
$$x(t)=\int G(t,t')F_{ext}(t')dt'$$

Assume

$$F_{ext}(t')=f_0\ \sin\ (\omega_1 t'+\delta);\ 0\le t'\le T$$
$$=0 \text{ otherwise}$$

$$E_{TOT}=\frac{1}{2}m\ddot{x}^2+\frac{1}{2}kx^2$$
$$=\frac{f_o^{\ 2}}{2m(\omega_0^2-\omega_1^2)^2}\{(\omega_0^2+\omega_1^2)(1-\cos(\omega_0 T)\cos(\omega_1 T))$$
$$+(\omega_0^2-\omega_1^2)(\cos(\omega_0 T)-\cos(\omega_1 T))\cos(\omega_1 T+2\delta)$$
$$-2\omega_0\omega_1\sin(\omega_0 T)\sin(\omega_1 T)\};\ \omega_1\ne\omega_0$$
$$=\frac{f_o^{\ 2}}{16m\omega_0^2}\left\{1+2\omega_0^2T^2-\cos(2\omega_0 T)-4\omega_0 T\sin(\omega_0 T)\cos(\omega_0 T+2\delta)\right\};\ \omega_1=\omega_0$$

From the formula given above, the amplitude of oscillation following a $5\pi/2$ pulse was computed and graphed as shown on the next page as a function of $\Delta\nu=\nu_1-\nu_0$.



amplitude
1.2
1.0
0.8
0.6
0.4
0.2
0.0
-100000
-80000
-60000
-40000
-20000
0
20000
40000
60000
80000
100000
Δν (Hertz)

nuclear magnetic resonance				
Carl W. Akerlof				
7-Mar-08				
e	1.60217649E-19	C		
h	6.62606896E-34	J s		
c	299792458	m/s		
m_p	1.67262164E-27	kg		
μ_N	5.05078324E-27	J/T	$e\hbar/2m_p$	
μ_p	1.41060666E-26	J/T		
μ_p/μ_N	2.792847356			
γ_p	2.675222098E+08	s^{-1} T^{-1}	$\mu_p/½\hbar$	
ν/B	4.25774821E+07	Hz/T	$2\mu_p/h$	$\gamma_p/2\pi$
ν	1.5360000E+07	Hz		
B_0	0.360754	T	$h\nu/2\mu_p$	
$\mathcal{E}_{max}$	0.00	V		
diameter	0.006	m		
area	2.8274E-05	m^2	$¼\pi d^2$	
B_1	0.0000E+00	T	$\mathcal{E}/2\pi\nu A$	
	calculated	measured	meas - calc	
$t_{\pi/2}$				
$t_{3\pi/2}$				
$t_{5\pi/2}$				