

# C-4 Pulsed NMR

## From Physics 191r

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I. Silvera, January 2010

First experiment: yes

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## LEARNING GOALS

- Understand the principles of nuclear magnetic resonance
- Measure relaxation times of protons in mineral oil, glycerin, water and solutions of paramagnetic ions
- Learn how magnetic resonance imaging works
- Observe resonance of protons in gypsum powder
- Perform FFT analysis on nmr signals from fluorine in various chemical environments
- Analyze data using nonlinear curve fits
- Observe NMR in the Earth's magnetic field

## INTRODUCTION

Liquids, solids, and gases can be studied by their interaction with a magnetic field. Nuclei with spin have a magnetic moment (parallel to the spin; electron spins are anti-parallel to their magnetic moments); therefore they interact with applied magnetic fields,  $\mathbf{B}$ , via the Zeeman interaction. Although the interaction is with the magnetic moment, this is connected to the nuclear spin and one generally uses the term spin interaction rather

than magnetic interaction (for spins larger than  $1/2$  there can also be interactions with electric quadrupole moments, etc). For a nucleus with spin  $1/2$ , there are two distinct quantum states in a magnetic field, spin-up and spin-down. Interestingly, although it is basically a quantum system most of the physics can be understood classically with the so-called Bloch equations that consider the relaxation of the nuclear magnetization to equilibrium as a function of time, when disturbed out of equilibrium, as is the case for NMR. We shall use both classical and quantum descriptions. As an example, for  $H = 0$  there is no spin splitting and the magnetization is zero, i.e. there are as many spins pointing say left as right. If a field is suddenly turned on, the magnetic moments will precess around the field at the Larmor frequency,  $\nu_0$ . To develop magnetization the spins must relax to equilibrium. If a spin relaxes from up to down, its energy changes and to conserve energy some excitation must be created in the lattice (or liquid). When the system achieves equilibrium the states will be populated according to the Boltzmann distribution  $N_{up}/N_{down} = \exp(-\mu_N B/kT)$ , where  $\mu_N$  is the nuclear magnetic moment]. In NMR you are studying the interaction with the ensemble of spins, not a single spin. The magnetic moment of the sample is the difference between the down and up moments, so at room temperature there is only a small excess fraction in the down state; for a macroscopic sample this is about  $10^{18}$  nuclei.

Because of the Boltzmann factor and the small magnetic moment (nuclear magnetic moments are typically three orders of magnitude smaller than electronic moments), NMR is an intrinsically insensitive technique, so special techniques are used to study the response and properties. An RF field of the correct frequency, normal to the dc field, can induce transitions. In a dc magnetic field of a few kilogauss, the energy difference between the down and up states is  $h\nu_0$ , where  $\nu_0$  is in the radiofrequency band. A coil can generate an RF magnetic field. Typically the coil is placed in parallel with a capacitor; an RF current is applied by a frequency-tunable circuit that can measure the energy required to maintain oscillation of the LC circuit. The original method of NMR was continuous wave (cw), either sweeping the frequency  $\nu$  of the oscillator at fixed field, or sweeping the field through resonance at fixed frequency. A measured change in RF absorption will occur when  $\nu = \nu_0$ .

Purcell, Torrey, and Pound first observed nuclear magnetism in condensed matter in 1946 by monitoring the Q of a resonant cavity containing paraffin as the proton resonance condition was swept through.<sup>[1]</sup> Soon after Bloch (1946) proposed to observe nuclear magnetic resonance via a technique known as pulsed NMR, which is now well-developed and used in diverse applications. As in the cw experiment, an RF magnetic field is applied to a sample via a coil. However in pulse NMR the magnitude of the RF field is usually greater than in cw NMR and the field is only applied for a short time.

To understand this experiment it is instructive to consider not just the energy difference between states but the net magnetization vector created by the excess population in the down state. The *net* magnetization,  $\mathbf{M}$  is parallel to the external field  $\mathbf{H}_0$  (in the z direction) but spins contributing to it will also have x,y components so that they are oriented at some angle with the external field and will therefore precess about it in the same way that a top spinning about a non-vertical axis precesses in a gravitational field. The frequency of this precession is called the Larmor frequency and at typical laboratory fields it lies in the radiofrequency band.

Now consider the RF magnetic field pulse; the RF field from a coil is linearly polarized and may be thought of as the sum of two oppositely-rotating components. In a frame of reference rotating at the frequency of the RF wave, one of these components will appear stationary, its direction determined by the phase of the oscillating field. Call this component  $\mathbf{H}_1$ . If the transmitter coil is perpendicular to the dc field,  $\mathbf{H}_1$  will be perpendicular to the equilibrium orientation of  $\mathbf{M}$ . If it is also at the Larmor frequency then ( $\mathbf{B}$  is zero in this frame at resonance)  $\mathbf{M}$  must precess about  $\mathbf{B}_1$ . Therefore for a given magnitude of  $\mathbf{B}_1$ , the angle through which  $\mathbf{M}$  will rotate may be

selected by varying the duration of the  $\mathbf{B}_1$  pulse.

Having rotated  $\mathbf{M}$  through some angle and turned off  $\mathbf{B}_1$ ,  $\mathbf{M}$  in turn will precess about the external dc field in the laboratory frame. If a pickup coil is oriented perpendicular to the dc field, then the component of  $\mathbf{M}$  perpendicular to the dc field will induce an RF voltage in the coil. This voltage is proportional to the transverse component of  $\mathbf{M}$  and is the NMR signal; it is usually detected with a phase-sensitive detector. An isolated spin would precess in its new orientation indefinitely, but in condensed matter certain mechanisms cause the system to relax towards equilibrium. The signal therefore decays with time, earning it the name free induction decay (FID). The spin not only interacts with the applied fields, but also its neighboring environment. Thermal variation of the relative positions of neighboring spins lead to an exchange of energy between the nuclear spins and the surrounding medium called the lattice. These are called spin-lattice interactions and they cause  $\mathbf{M}$  to realign with the external dc field; the time constant for this process is called  $T_1$ , the spin lattice relaxation time.

If one views an individual spin as precessing in a local field that is the superposition of the external dc field and magnetic fields due to neighboring spins, one sees that individual spins precess at slightly different frequencies. The result is that even though a group of spins may be parallel to each other at some initial time  $t = 0$ , there is a spin-spin relaxation time  $T_2$  during which the precessing spins get out of phase, and the transverse moment decays. This causes "defocusing" of  $\mathbf{M}$  in the plane perpendicular to the external dc field. Furthermore if the external dc field is inhomogeneous, the Larmor frequency will vary across the sample. Small volumes of sample, which experience an effectively uniform field are called isochromats. If the inhomogeneity is large, isochromats with different precession frequencies get out of phase on a time scale shorter than  $T_2$ , masking the intrinsic  $T_2$  process. The time constant for this instrumental effect is called  $T_2^*$ .

To circumvent the limitations of magnets it is possible to make use of a procedure pioneered by Hahn in 1950. Hahn's idea was to prepare a spin system by rotating  $\mathbf{M}$  through  $90^\circ$ , allowing  $\mathbf{M}$  to defocus via the  $T_2^*$  process, then after a time  $\tau$  to rotate the spins again, this time through  $180^\circ$ . See the figure at right. The second rotation reverses the order of the isochromats, putting the slow ones ahead of the fast ones. When the fast ones catch up with the slow ones,  $\mathbf{M}$  has refocused and a signal is again induced in a coil. The amplitude of a train of such spin echoes decays with  $T_2$  rather than  $T_2^*$ . A number of choices are possible for the phases of the  $180^\circ$  pulses.

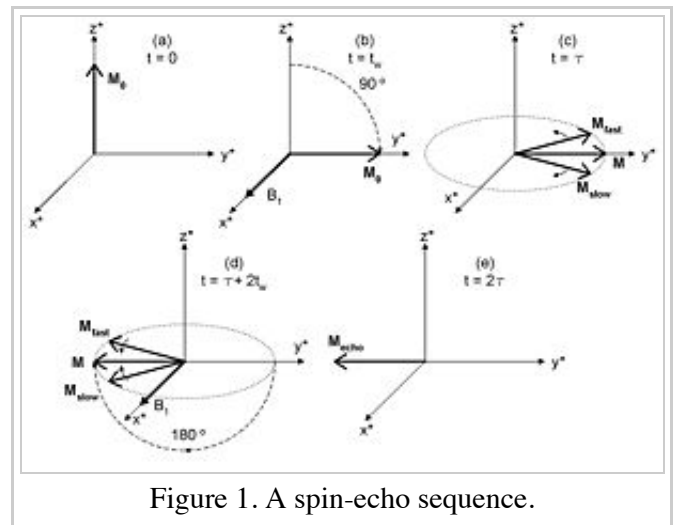


Figure 1. A spin-echo sequence.

## APPARATUS AND INSTRUMENTATION

The NMR experiment, which was originally a cw experiment in the Advanced Lab, has evolved over the years, with the introduction of pulsed NMR with an apparatus supplied by TeachSpin. The original PS1-A apparatus has just (2010) been replaced with the latest PS2-A Pulsed/CW NMR apparatus that you shall use in this experiment. PS1-A used crossed coils, one for the  $\mathbf{B}_1$  pulse, with the pick-up coil at right angles so that the large  $\mathbf{B}_1$  pulse was not picked up, only the NMR response. A deficiency in this apparatus was that the dc magnet was

temperature dependent and the field drifted with temporal variations of the temperature; the signal was not phase detected. In PS2 the magnet with a field strength of 0.5 Tesla is temperature controlled in a tight feedback loop, to overcome this deficiency. In addition there are adjustable shim coils around the sample that enable you to homogenize the field at the sample.

In PS2, a modern system with a single coil is used for both the pulsed field and the NMR signal pick up. Since the pulsed field is quite large, without precautions it would saturate the low-noise amplifier in the pick-up circuit. To circumvent this some rf tricks are used including crossed-diodes, phase shifts in high-frequency coax, and blanking off of the amplifier during the field pulse. Figure 2 is a simplified block diagram of the apparatus; it does not show all the functions of each module, but it does represent the most important functions of each modular component of the spectrometer. You can read the details about this in the *TeachSpin Manual* which is also on the course website, and includes a few chapters of introduction to NMR. You should also read about the magnet, compensating coils, how to set the temperature controllers, and other details of this versatile instrument.

The pulse programmer creates the pulse stream that gates the synthesized oscillator into radio frequency pulse bursts, as well as triggering the oscilloscope on the appropriate pulse (we are using an Tektronix DPO 4032 digitizing oscilloscope; a manual is on the bench or the latest version is on the website). The rate, length, amplitude, and phase of the RF pulses can be controlled by the user for carrying out a number of different measurements. These are the time dependent  $B_1$  fields that produce the precession of the

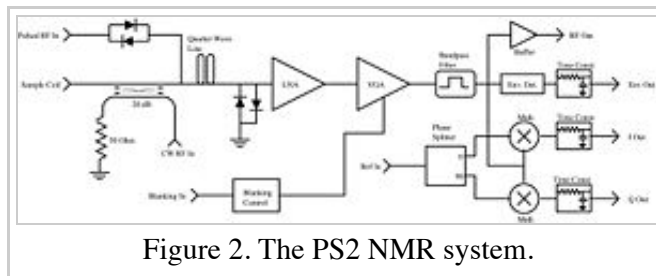


Figure 2. The PS2 NMR system.

magnetization, referred to as the  $90^\circ$  or  $180^\circ$  pulses. The frequency of the RF pulse can be adjusted by the user and initially should be set for protons; later it can be set for fluorine NMR. The coil must be tuned for the frequency used with tuning capacitors (see manual) and this is most easily carried out by replacing the sample with a small provided coil to simulate the sample signal, and tuning to optimize this signal. Nuclear magnetization precession in the direction transverse to the applied constant magnetic field (the so called x-y plane) induces an *emf* in the receiver coil, which is then amplified by the receiver circuitry. The signal is phase detected (see Fig. 2) and you have the choice of either detecting the diode demodulated envelope signal or the in-phase and quadrature signal ( $I_{out}$  and  $Q_{out}$ ); both are useful. By looking at the phase detected signal you can observe the beat signal between the synthesizer frequency and the NMR signal; you are tuned at the resonance when this beat frequency is zero. The PS2 module allows you to set up pulses for measuring relaxation times, Hahn spin-echoes, and correcting for the effect of diffusion in a field gradient using the Carr-Purcell sequences and Meibohm-Gill sequences (see the book by Slichter).

The initial measurements that you will be making are in the time domain, observing the NMR signal as a function of time as it decays to equilibrium. In the original cw experiments, one measured the power spectrum or the absorption as a function of frequency. Ernst and Anderson showed that the power spectrum could be obtained from the Fourier transform of the time dependent signal. Your oscilloscope allows you to Fourier transform the time dependent signal. Although this is useful, it is an amplitude Fourier transform, and the desired transform of the phase detected signal is the cosine transform (see Ernst and Anderson). You can transfer the time dependent signal from the oscilloscope to a computer for further analysis. This versatile apparatus also includes an RF bridge circuit and programs to sweep the field or frequency and carry out cw NMR. The adjustments and tuning for this are quite delicate, and if you attempt this, we recommend that in addition to using the tuning capacitors, when near balance you also make slight retuning of the frequency to achieve balance.

# EXPERIMENTAL PROCEDURE

The first step is to familiarize yourself with the instrumentation. Please read the "Getting Started" section of the *TeachSpin Manual* (after reading the Instrument file), and make the appropriate cable connections. These notes will help you set up the spectrometer and walk you through some measurements. If you're not familiar with digital scopes, you should also read the *Tektronix DPO4000 Manual* and learn how record and transfer either screen images that can be replayed on the scope or data in excel formatted files that can be uploaded into analysis programs such as IGOR or Matlab, both downloadable from the Harvard FASCS site. The easiest way to transfer data to a computer is via a flash drive.

The following suggestions are possibilities for measurements you might choose to undertake. They are by no means exhaustive. They all require reading of the literature for a thorough physical explanation. We would like you to choose experiments that use both proton and fluorine NMR and at least one that requires use of the Fourier transform.

- **1. Learning to do NMR.** It is useful to start out with mineral oil and learn how to set up the instrument, tune and set the frequency, measure relaxation times etc. Play around with this until you feel comfortable with the basics of NMR.
- **2.  $T_1$  and  $T_2$  in water doped with paramagnetic ions.** Paramagnetic ions, with their large electronic magnetic moment, profoundly affect relaxation time of the protons in water. The effects can be measured over a wide range of concentrations. Two possibilities are  $\text{CuSO}_4$  and  $\text{Fe}(\text{NO}_3)_3$ , both of which dissolve in water.
- **3.  $T_1$  and  $T_2$  in Glycerin and water mixtures.** Glycerin and water mix in any ratio. The motion of the protons in glycerin is significantly changed by the change of the liquid viscosity with the addition of water. The relaxation times can be correlated with the viscosity of the liquid, as well as the water concentration.
- **4. Fluorinated compounds.** Several fluorinated compounds are available with this experiment. These have fluorine at different sites with different chemical shifts of the resonance so that there are multiple lines. Reset the frequency and study these compounds and their Fourier transforms.
- **5.  $T_1$  and  $T_2$  in medium where diffusion is restricted.** A sample consisting of a matrix of glass beads of radius  $\sim 100$  microns saturated with water shows effects of restricted diffusion.(reference: see book by P.T. Callaghan)
- **6. The Pake Doublet.** The hydrogen molecule has an unusual spectrum, which requires a quantum mechanical analysis for understanding. Pake studied the NMR spectrum of the water molecules in gypsum and showed that this could be used to determine the orientation of the water, and has led to the use of NMR in chemistry and biology for structural analysis. The two spins on the protons in water can be either in the singlet or the triplet state; the singlet has no magnetic moment, but the spin 1 triplet has transitions between the three  $m_l$  levels. You can be provided with a powdered sample and possibly a single crystal to study this effect. It is important to view the spectrum in the frequency domain.
- **7. Magnetic Resonance Imaging.** Under development.

## NOTES

1. ↑ Purcell, Torrey and Pound (1946)

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C.P. Slichter, Principles of Magnetic Resonance, (Springer-Verlag, Berlin, 1978, 1990 3rd ed). A complete text with problems, clear explanations, appropriate for advanced undergraduate or graduate level students. Excellent bibliography. Any serious student of magnetic resonance should own it. Everyone should read at least some of it. Cabot: QC762.S55 1990.

# BENCH NOTES

- Proton Chemical Shifts ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/chem\\_shift.pdf](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/chem_shift.pdf))
- Tektronix DPO4000 User Guide ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/Tek\\_DPO4000user.pdf](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/Tek_DPO4000user.pdf))
- Glass Beads for Restricted Diffusion ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/glassbeads.pdf](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/glassbeads.pdf))
- Basics of NMR (Rochester Inst. of Tech.) (<http://www.cis.rit.edu/htbooks/nmr/inside.htm>) (J.P. Hornak)
- Mathematica script: FFT Examples (P. Pershan 2011) ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/Fourier.nb](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/Fourier.nb)) (Open in new window - save as text.)
- Example data file for FFT script "cexp.dat" ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/cexp.dat](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/cexp.dat))
- Example data file for FFT script "exp.dat" ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/exp.dat](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/exp.dat))
- Example data file for FFT script "Step.dat" ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/Step.dat](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/Step.dat))
- Mathematica script for making example data files (P. Pershan 2011) ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/MakeData.nb](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/MakeData.nb))
- Mathematica script: NMR Imaging (P. Pershan 2011) ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/NMRImaging.nb](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/NMRImaging.nb))

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- Getting Started ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/PS2A\\_get\\_start\\_aug10.pdf](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/PS2A_get_start_aug10.pdf))
- Experiments ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/PS2A\\_expt.pdf](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/PS2A_expt.pdf))
- Specifications ([http://www.fas.harvard.edu/~phys191r/Bench\\_Notes/C4/PS2A\\_spec.pdf](http://www.fas.harvard.edu/~phys191r/Bench_Notes/C4/PS2A_spec.pdf))
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