Forecast:
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Blown Away

The Relaxation Times

2495 Main Street
Buffalo, NY 14214
716-885-4701
fax 716-836-1077

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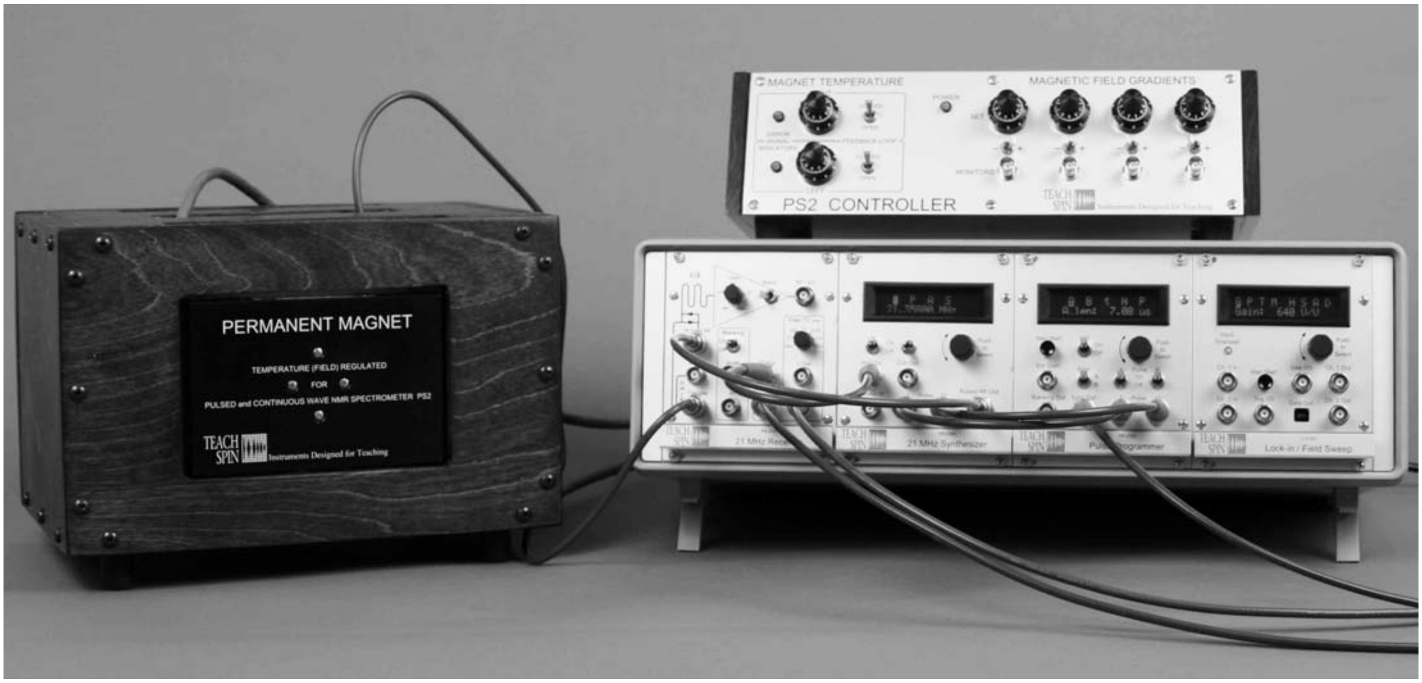

PULSED NMR ON STEROIDS

We heard you! Over the fourteen years since we introduced our first instrument, PS1-A, we have carefully listened to our customers' and even our potential customers' concerns and admonitions for our Pulsed Nuclear Magnetic Resonance Spectrometer. The opportunity to address these ideas came when an important component of the original electronics was no longer manufactured and we needed either to find a substitute or redesign the entire unit. We chose to rethink the experiment and to offer an entirely new and significantly more versatile teaching apparatus. And now, after over two years in development, this unit is finally in production. We have already sold twelve units and have delivered spectrometers to Kassel (Germany), Florida International, and Cal State Stanislaus Universities. Should your school have its name on the PS2-A waiting list?

What's new? Most of the answers to that question can be found in this issue of *The Relaxation Times,* which is completely devoted to the PS2-A, our Pulsed/CW Nuclear Magnetic Resonance Spectrometer.

First of all, PS2-A can observe NMR signals from ***two*** types of nuclei, hydrogen (proton) and fluorine. This opens up the possibility of studying an entirely new collection of molecules in liquids and some solids. Fluorine nuclei are particularly interesting because they typically exhibit large chemical shifts in various compounds. In our original spectrometer, PS1-A, where only proton NMR could be studied, the small chemical shifts were not observable. This was a problem, especially for the chemistry faculty. One might even say that chemical shifts are the "bread and butter" of organic chemistry's NMR analysis. Now, with this unit, chemical shifts in fluorine compounds are easily observable and some can even be seen in proton compounds.

The reason these splittings are observable in PS2-A is not just the fact that it can detect fluorine NMR, but also that the new unit has a larger and more homogeneous magnetic field. The proton resonant frequency is 21 MHz and the T_2* is 10 milliseconds. This unit has a set of four independently controlled electric gradient field coils. Each shim coil has its own current regulated power supply that can be adjusted to increase the homogeneity of the magnetic field over the sample. **Figure 1** shows the free induction decay of a distilled water sample with and without the gradient coil fields.

see Figure 1 inside ➔

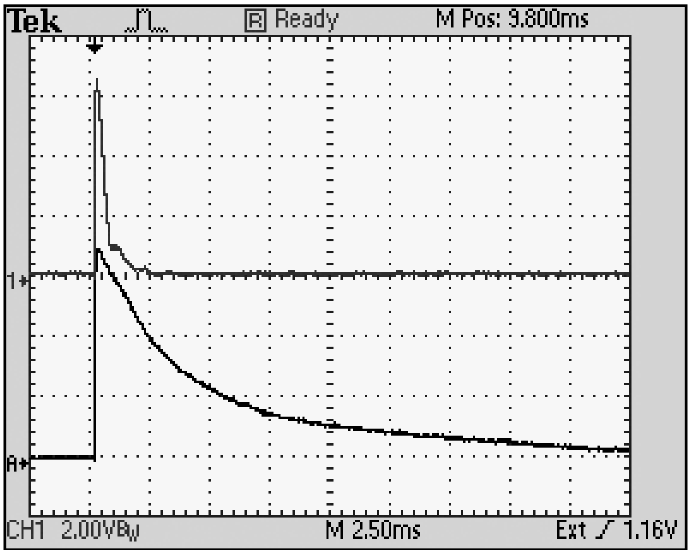

Figure 1 – FID Distilled Water

Not only has the magnetic field been increased in magnitude and homogeneity, it has also been greatly enhanced in stability. Magnetic field stability has been a problem with permanent magnets because the magnetization of the NdFeB alloys is extremely temperature dependent. Our original spectrometer needed to be retuned every five to ten minutes and thus RF phase sensitive detection was impractical. After its forty-five minute warm-up period, PS2-A has a field stability of one part in 10^6 over fifteen minutes. This is accomplished with a proprietary magnet design and PID temperature controller. Both the temperature controller and the current regulated supplies for the gradient coils are housed in the hardwood case that is shown on top of the Mainframe of the instrument.

With such outstanding field stability, students can take advantage of data from the two channel quadrature RF phase-sensitive detectors. The reference RF has a full 360° phase shift with one degree resolution. There is also an envelope detector as well as a buffered output of the direct RF magnetic resonance signal. Again, because of the field stability, students can perform long term signal averaging with the phase-sensitive detector signals, to extract weak signals from the noise. Such signal averaging is now a standard feature on almost all digital oscilloscopes.

This new high-homogeneity, high-field magnet allows students to use the modern form of spectroscopy, Fast Fourier Transforms (FFT), for both fluorine and proton samples. **Figure 2** shows the FID signals of a liquid called Fluorinert, FC-70, with the envelope detector (on resonance) and a RF phase sensitive detector slightly off resonance.

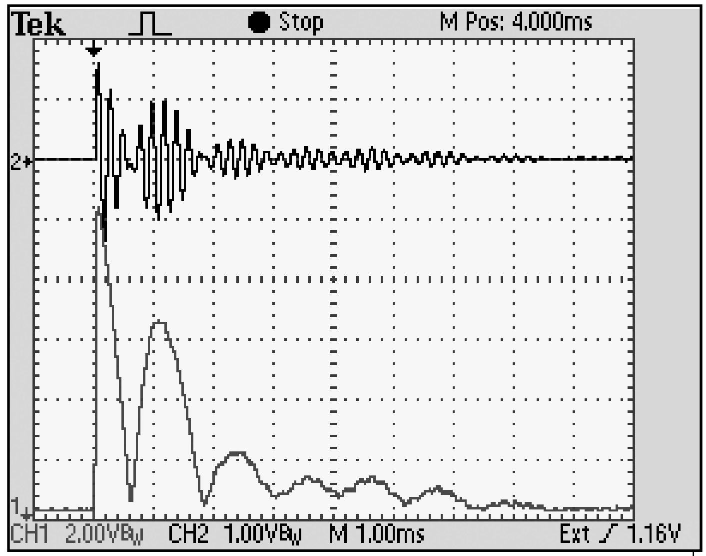

Figure 2 – FID for Fluorinert FC-70

This sample shows a dramatic beat structure, indicating the presence of at least two inequivalent fluorine sites in the sample.

Figure 3 shows the FFT of the phase detector's signal, which has been signal averaged over 16 pulses. The FFT spectrum indicates the presence of three inequivalent fluorine sites and gives their relative splittings. The spectrometer comes with a set of four different fluorine liquids that are all safe for students to use.

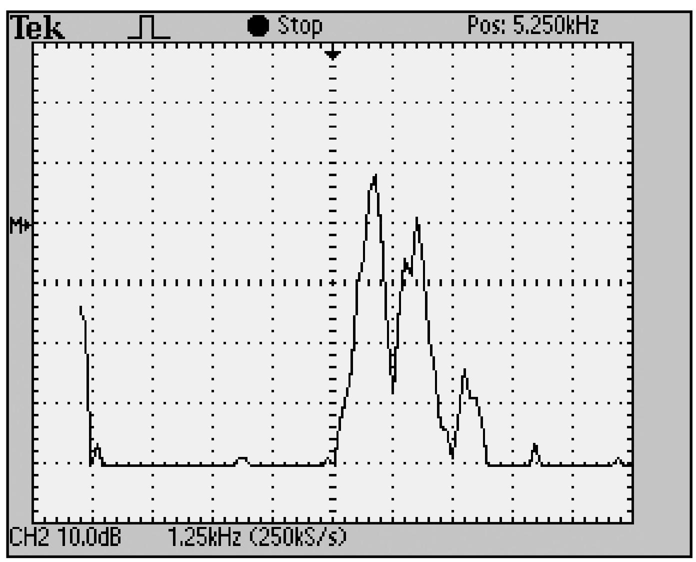

Figure 3 – FFT for Fluorinert FC-70

As with the original PS1-A,B, the unit comes with its own pulse programmer which is capable of producing various pulse trains. Spin-echo signals from a simple two pulse ($\pi/2$, π) sequence (with a variable delay time) are available. This is shown in **Figure 4** for a light mineral oil sample.

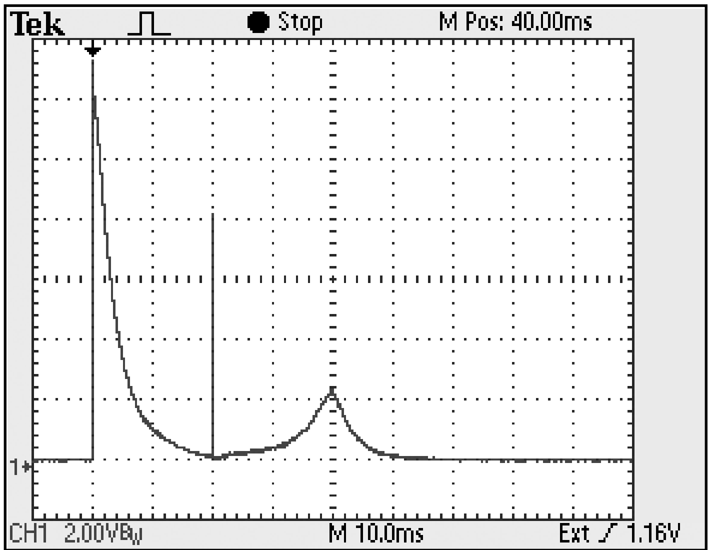

Figure 4 – π/2, π Pulses, Light Mineral Oil

Multi-pulse sequences for the so-called Carr-Purcell and Meiboom-Gill (MG) are also available in the unit. A thirty π-pulse MG sequence, shown in **Figure 5**, was used to measure T_2 in light mineral oil. A two-pulse sequence, with the first π pulse and the second π/2, can be used to measure the spin-lattice relaxation time in any sample. All the pulse widths and time delays are digitally generated for stability and accuracy.

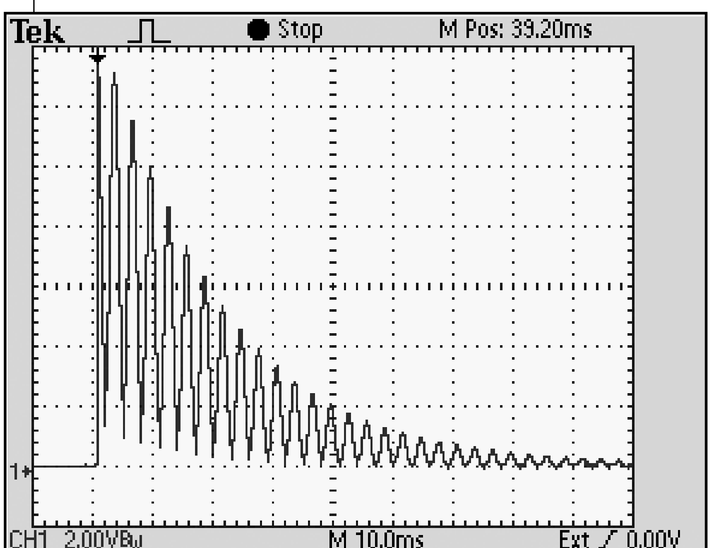

Figure 5 – G-M Pulse Sequence, Light Mineral Oil

Over the years, we have had requests from several universities to create an instrument that could detect magnetic resonance signals two ways; pulsed and the original continuous wave (CW) spectroscopy, so that students could study the relationship between them. That is exactly what we have accomplished. This unit is capable of observing NMR signals for either hydrogen or fluorine nuclei using a continuous RF field and sweeping the magnetic field through resonance. Figure 6 shows the CW resonance signals for the fluorine FC-70 Fluorinert liquid.

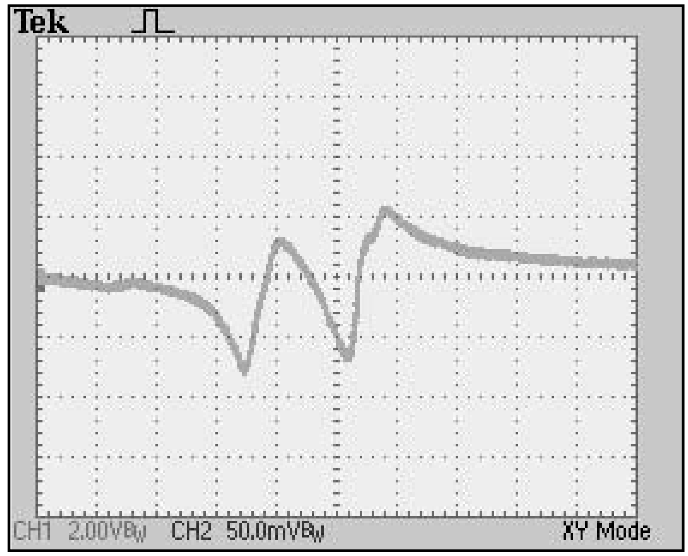

Figure 6 – CW Resonance for FC-70

This signal came from the RF phase sensitive detector with no attempt to separate the real and imaginary part of the nuclear susceptibility using the phase shifter. However, the magnetic field splittings observed in the CW spectrum can now be compared to the frequency splittings detected in the FFT spectrum of the free induction decay. After performing both experiments, it should become clear to the students why the modern spectrometers all use the FFT analysis of the FID.

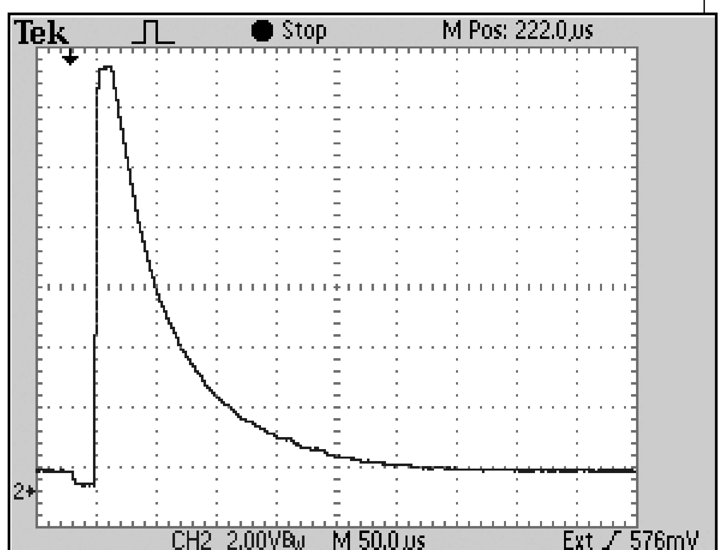

Figure 7 – FID, Teflon

Our new unit has a significantly reduced recovery time after the pulse. This allows the spectrometer to measure signals from many "soft" solids which have T_2 longer than ten microseconds. For example, **Figure 7** shows the FID signal for fluorine nuclei in Teflon. Rubber, greases, plastics and other solid materials with either hydrogen or fluorine nuclei can be studied. With a recovery time of the order of 15 µs, an entire class of solids can now be added to the long list of candidates for exploration.

TEACH SPIN
Tri-Main Center, Suite 409
2495 Main Street
Buffalo, NY 14214-2153

ANNOUNCING THE NEW PULSED/CW NMR

The unit also has a built-in two channel lock-in amplifier. This special purpose, fixed modulation frequency lock-in is used for enhancing the CW signals for broad linewidth signals. It allows students to gain first-hand experience with lock-in detection in NMR. The lock-in module also provides the current for the magnetic field sweep coils as well as an output analog voltage proportional to the field sweep. Various sweep times, sweep ranges, field offsets, voltage amplification, and low-pass time constants are available in the unit.

Unlike our original PNMR spectrometer, PS2-A is a single coil, matched sample probe instrument. The single coil functions efficiently as *both* the transmitter and the receiver. There are many advantages to a single coil unit; including lower power required for RF pulses, smaller size, smaller magnet gap, and an impedance matched sample probe.

For a student, however, operation of the single coil system may appear to be pure “magic”. There are many ways to accomplish this “magic”, but we have chosen a system that can be readily understood by a beginner. A block diagram is shown in **Figure 8**. The instruction manual devotes several pages, with several diagrams, to explain carefully and clearly how this single coil system works for both pulsed and CW resonance. The “magic” turns out to be clever electronics.

The electronics system was designed by Dr. Norman Jarosik of the Princeton University Physics Department. Norman, has been involved with TeachSpin from its inception and designed the electronics for PS1-A,B, whose reputation for reliability, as well as sensitivity is all but legendary. Norman is known to the research community as a staff scientist in the “gravity Group” and the chief engineer of WMAP, the satellite that has been sensing and mapping the anisotropies in the microwave radiation left over from the Big Bang of the early universe. Unsurprisingly, that satellite has been basically trouble free and outlasted its expected lifetime.

Twelve units of PS2-A have already been spoken for, but if you can get your purchase order in soon, we can deliver your spectrometer in time for the fall semester. The current US/Canada price for the spectrometer is $16,750.00. With the Euro so much more valuable than the dollar, the European instrument is practically “free” for those lucky students.

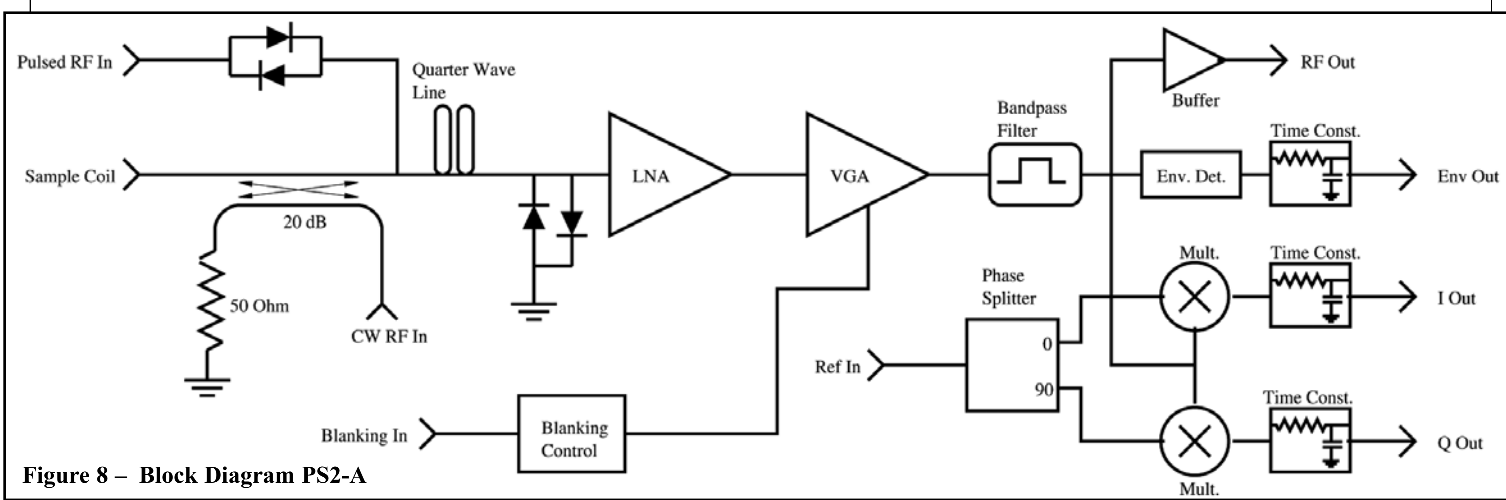

Figure 8 – Block Diagram PS2-A