Forecast:
Proton Chemical Shifts
www.teachspin.com

The Relaxation Times

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

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More Experiments
with our
New Pulsed NMR Spectrometer PS2-A

At TeachSpin, we have been enjoying our new pulsed NMR spectrometer. It's not just the fun of building them (JFR still does construction) but the considerably greater pleasure we get out of "discovering" new experiments for your students to perform with it. This instrument has so many new features – such as the incredible field stability of 1 part in 10^6 over 15 minutes, the quadrature rf phase-sensitive detection with the digital phase shifter, the electric gradient field shim coils, and the cw/lock-in detection – that we are sure that both TeachSpin and the faculty who own the unit will be coming up with even more ideas for student experiments. As these ideas are carefully vetted, we will share them with you, probably in subsequent editions of "The Relaxation Times."

This spectrometer was designed to perform pulsed NMR experiments on **two** types of nuclei: protons (hydrogen) and fluorine. The choice of the proton is obvious, but the reasons for fluorine may be less familiar to some of you. Fluorine has a spin of ½ with a magnetic moment which is only 6% smaller than that of the proton. This means that the resonant frequencies of the two nuclear species are very close. And this makes the design and construction of electronics that can detect both NMR signals relatively easy. But, even more important, is the fact that fluorine nuclei generally experience much larger "chemical shifts" than protons. "Chemical Shifts" are the rather small shifts in the resonant frequencies of the nuclei in solids or liquids due to atomic magnetic fields created by the local electrons. These chemical shifts are especially important for the chemists (hence the name!), since they provide spectral signatures that help identify molecular structures.

TeachSpin wanted to build an affordable, hands-on, table-top instrument that could both observe and measure these chemical shifts using pulsed techniques. Since fluorine shifts are generally larger than proton shifts, we set out to produce a spectrometer that could detect fluorine NMR. This we did. But, after building several units, we realized that ***proton*** chemical shifts in several liquids that are especially important to chemists, should also be observable with our new spectrometer. So we tried it, and thought you might be curious to examine our results.

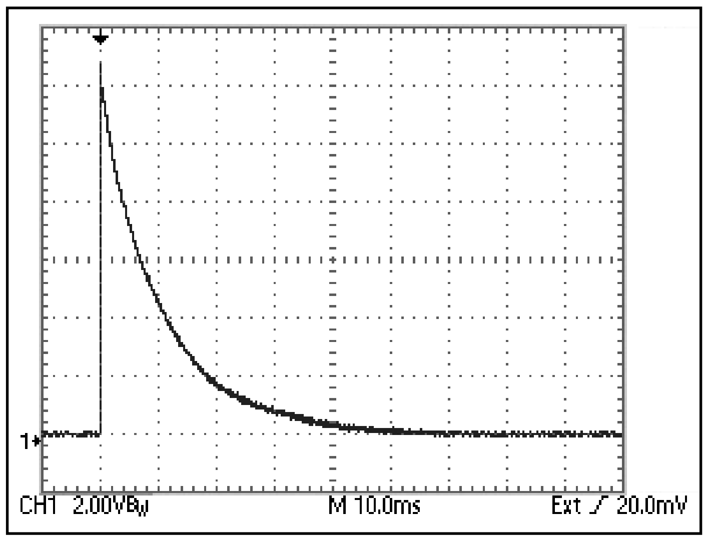


Figure 1 – FID Light Mineral Oil

The first task for the student is to adjust the currents in the gradient coils to achieve the optimum field homogeneity over the sample volume. This is easiest to do using the proton signal from a few drops of light mineral oil. Figure 1 shows our results for the demonstration unit. The time constant of the free induction decay signal (FID) indicates a field homogeneity of about 2 x 10^{-6} Tesla over our sample. The long decay times gave us confidence that proton chemical shifts in some well known chemical solvents should be observable.

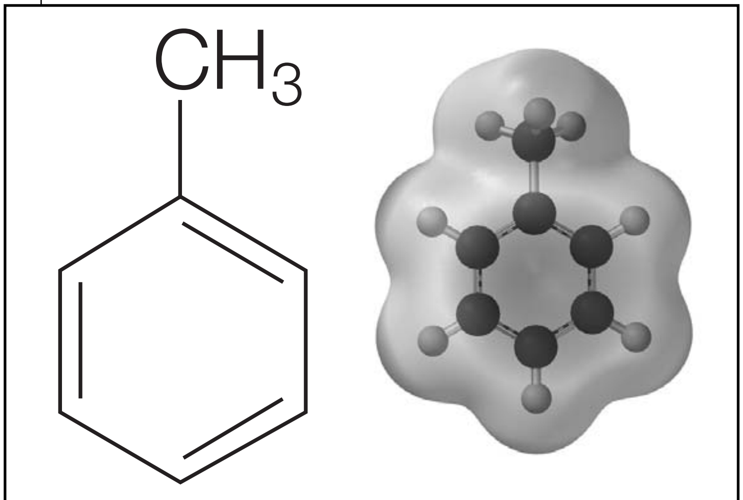


Figure 2: Chemical Structure of Toluene

Our first attempt was toluene, with a molecular formula of $C_6H_5CH_3$. The Wikipedia representation of its chemical structure is shown in Figure 2. There are two groups of hydrogen atoms, three in the methyl cluster and five around the ring.

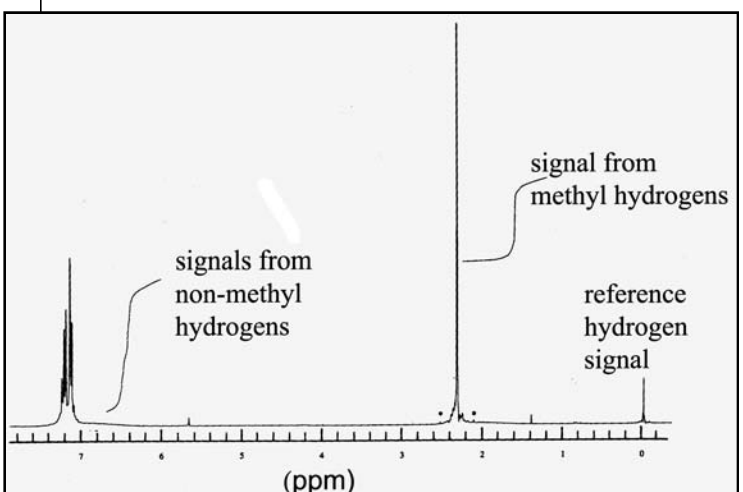


Figure 3: High resolution NMR FFT signals from Toluene

The commercial high-resolution spectroscopy signals are shown in Figure 3, with a hydrogen reference signal from tetramethylsilane.

Figure 4 shows PS2-A's FID signal from our sample of toluene. The beats are clearly observable in both the envelope detector (lower trace) and the phase detector signals. For students familiar with a transient signal summed from two oscillators of nearly the same frequency and amplitude* this FID is an old "friend." This is confirmed by the fast Fourier transform (FFT) of the phase detector's signal shown in Figure 5.

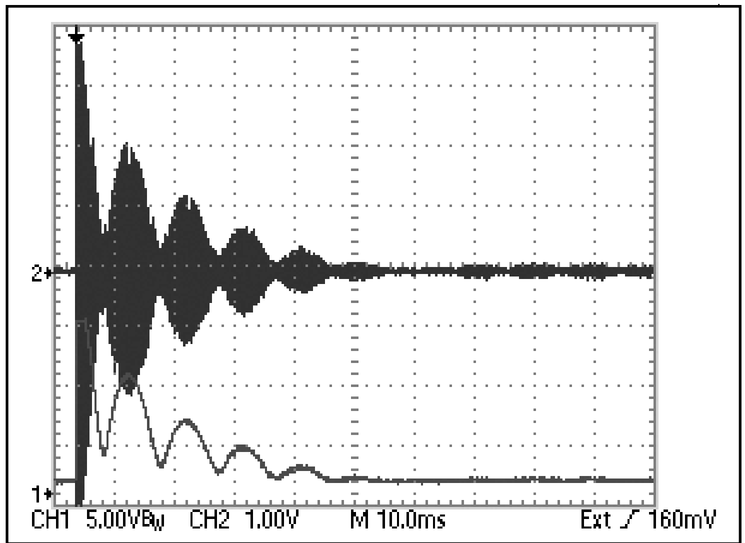


Figure 4: FID of Toluene

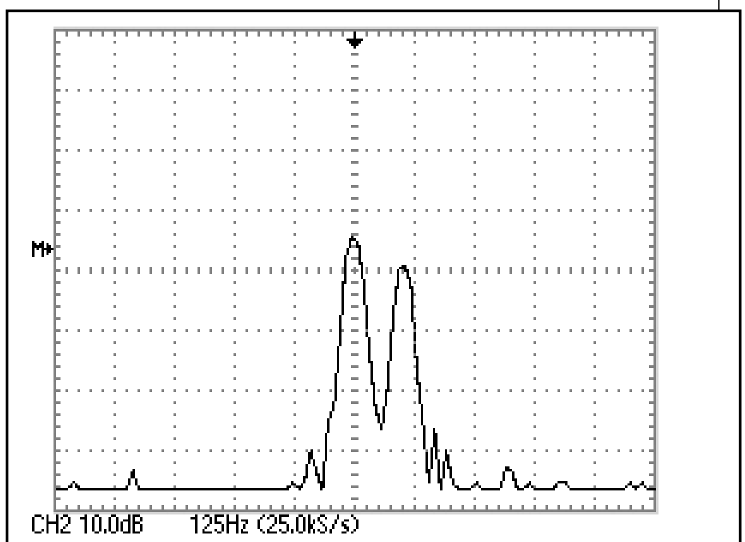


Figure 5: FFT of Toluene

Although the resolution is not nearly what the state-of-the-art commercial NMR spectrometer can achieve, the splitting is clearly observable and measurable. At 125 Hz/div x 0.8 div = 100 Hz splitting out of 21.2 MHz or about 4.7 ppm. The agreement is excellent.

* *TeachSpin's new Torsional Oscillator (TO1-A) is the ideal instrument to show this "beat" signal. See Vol III No. 2 of The Relaxation Times and future issues.*

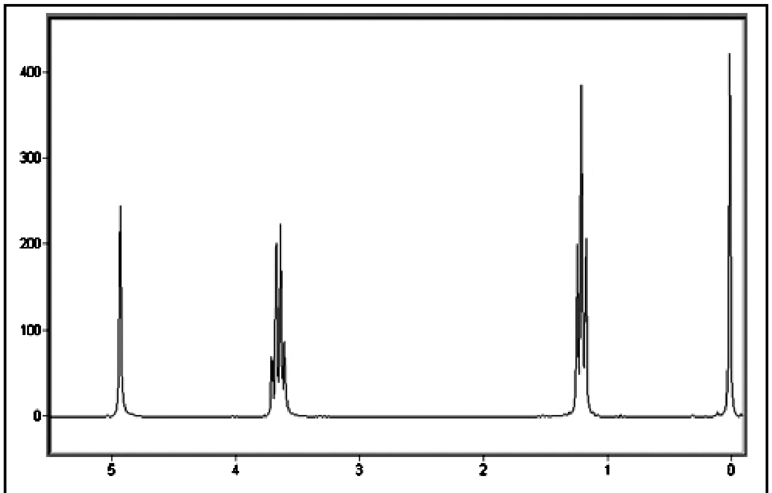


Figure 6: High Resolution NMR FFT Signal from Ethanol

Ethanol (or ethyl alcohol) has three inequivalent proton clusters as shown in the commercial spectrometer data of Figure 6. Here, the overall shift

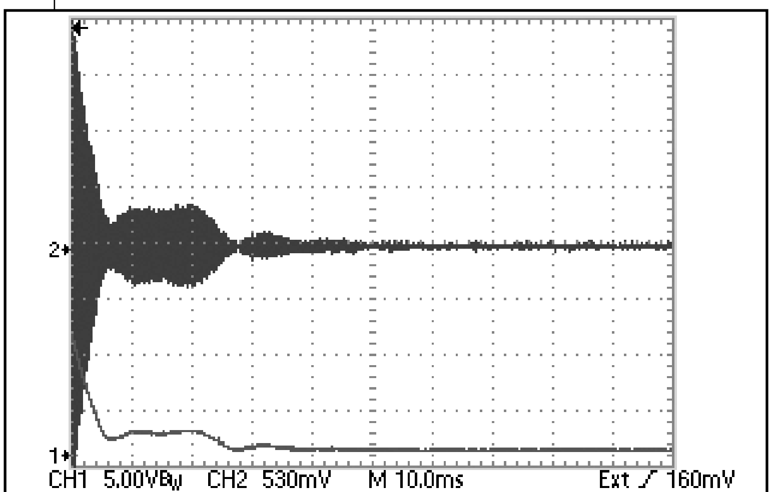


Figure 7: FID of Ethanol

is only about 4.5 ppm. Figures 7 and 8 show the FID and FFT from PS2-A. It might take a bit of imagination to see three lines in the FFT data, but the overall splitting of 0.7 div x 125 Hz/div = 87 Hz out of 21.3 MHz, or about 4.1 ppm and is in good agreement.

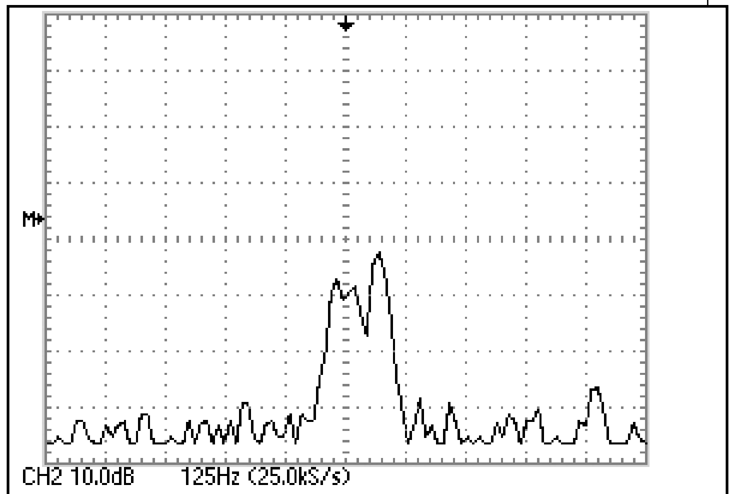


Figure 8: FFT of Ethanol

Obviously, PS2-A will not replace the commercial high resolution spectrometers so essential for modern day organic chemistry and structural biology. The TeachSpin apparatus can, however, be used to teach students the scientific principles that are the basis of the way these important commercial instruments work. It's an instrument students will find very difficult to damage. They can have total control over this apparatus as they carry out their own experiments on a wide variety of solvents. That is the way we at TeachSpin believe that students truly learn science. When will *your* students get their hands on it?

What You Didn't Want to Hear

Sadly, we need to inform our customers that we have been forced to raise our prices. We all have been feeling the pinch, especially when we shell out 50 – 70 dollars to fill up at the gas station. No, we are not raising our prices like the oil companies have, but we have experienced serious cost increases almost everywhere. The cost of shipping goods from our suppliers, the cost of metal parts, particularly copper, brass, and aluminum, the cost of energy, and the cost of health insurance (which every full-time employee of the company has at company expense) have all recently gone up.

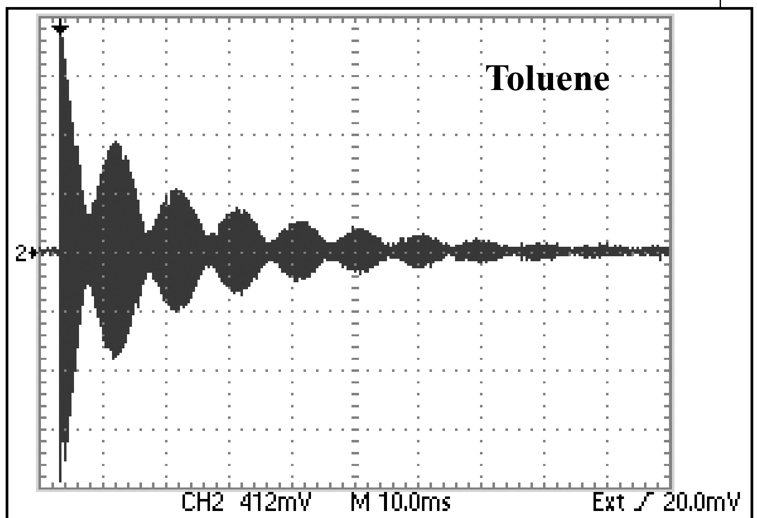
We have had to raise the prices before the end of the year because of these sudden increases in our costs. We hope that we will not have to raise prices again in the near future. We certainly will try, that's a promise. Happily, many of the increases were already taken into account in the pricing of the new instruments so the prices of the Pulsed NMR, Quantum Analogs, and Torsional Oscillator will not have to change at this time.

I know how tight the college and university budgets are for laboratory instruction – especially for

continued >>>>

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

TeachSpin's *NEW* Pulsed/cw NMR Spectrometer *Presents* Signals from Inequivalent Protons



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advanced laboratory instruction in physics. (I taught at universities for over 40 years.) Upper division lab classes are small and the equipment is expensive. But, we hope that each of you can keep making the case to the chairman, the dean, the provost, or even the president of your school for the importance of this part of the educational program. Experimental physics, I claim, is the basic source of technological innovation – technological revolution. It does not begin with engineering; it begins with basic science.

Each of us must play our part in keeping our countries strong in scientific and technological development. Your advanced lab may be the place to inspire that young mind to become the next Charlie Townes, Nicolaas Bloembergen, Robert Dickie or Nicola Tesla. Who knows? We at TeachSpin continue to work extremely hard to keep our prices affordable, while still producing modern, rugged and reliable equipment. But we are swimming in a much larger economic "river," with a recent strong current carrying us along in a direction we do not like.

Even with a price increase of about 6%, we still believe that TeachSpin apparatus is a bargain. We hope you do too.

– Jonathan