

A Pulse NMR experiment for an undergraduate physics laboratory

An inexpensive apparatus incorporating some of the recent innovations in electronics has been built for performing nmr experiments in an undergraduate physics laboratory. The student is able to observe spin-echoes and measure T_1 and T_2 .

I. INTRODUCTION

The techniques of pulse nmr have advanced continually in recent years in both the research and teaching laboratories. Most research spectrometers today involve Fourier transform methods requiring the use of digital computers. Without getting into the ongoing pedagogical discussions about the value of computers in the teaching laboratories, one can probably agree that there is still a use for computer-free pulse nmr experiments in which one can actually see and manipulate the signals in the time domain.

Several excellent articles¹ and books² have been published describing such experiments but the rapid advances in electronics have necessitated occasional updates.

One relatively recent technological improvement incorporated in several places in this experiment involves the use of double-balanced mixers (DBM) and broadband power splitters³. Both of these devices use broadband twisted-pair transmission lines⁴, ferrite-core transformers and a matched diode bridge. In order to multiply two signals, some non-linear device such as a diode is needed. The advantage of DBM's is in their improved impedance matching and isolation

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¹ e.g. "Simple, Spin-Echo Spectrometer", B. H. Muller, J. D. Noble, L. F. Burnett, J. F. Harmon, D. R. McKay, Am. J. Phys., **42**, 58 (1974)

² e.g. Fukushima, E. and Roeder, S. B. W., Experimental Pulse NMR, (Addison-Wesley, Reading, MA, 1981)

³ Manufactured by Mini-Circuits Labs, Brooklyn, NY. Either device can be furnished in a small metal box with three bnc connectors for less than \$50 or with leads for circuit-board mounting for less than \$20. The company publishes an excellent catalog which includes many pages on the theory of rf splitters and double balanced mixers (DBM).

⁴ A pair of #40 enamelled coated copper wires twisted together can have a characteristic impedance of 50 ohms. See P. Lefferson, IEEE Trans. PHP-7,4,148 (1971)

among the radiofrequency (rf), intermediatefrequency (if) and local oscillator (lo) signals.

OVERVIEW

Figure 1 is a schematic diagram of the experimental setup which consists of a gated rf pulse generator with variable pulse widths and spacings, a probe circuit that delivers rf power and extracts a signal from the sample, a preamp and a mixer to mix

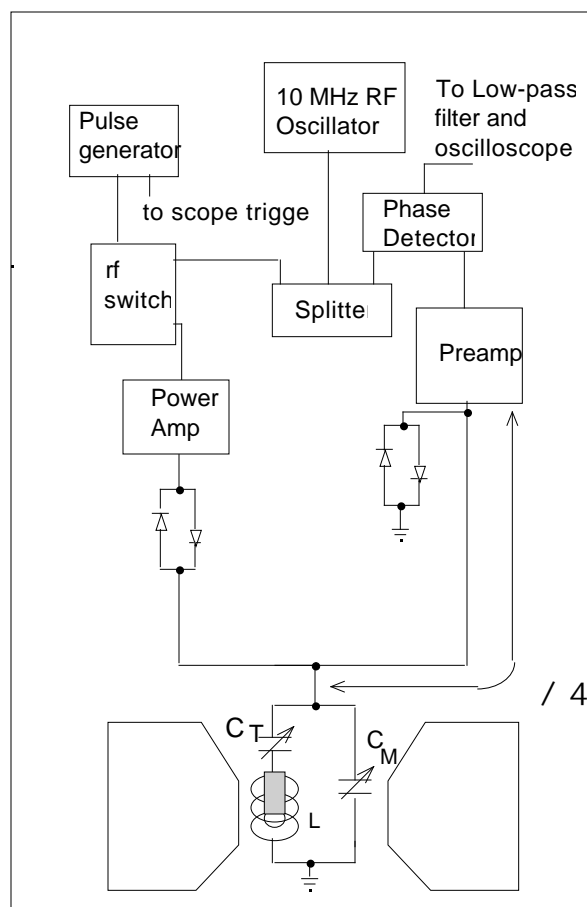


Figure 1. Schematic diagram

the NMR signal down to a frequency where it can be easily manipulated and examined. Any laboratory electromagnet capable of producing 2400 G should be suitable for the experiment. Field homogeneity requirements are less stringent for pulse nmr than for continuous wave experiments, and many laboratories should have magnets on hand that will work nicely.

A 10 Mhz crystal-controlled oscillator⁵ feeds a continuous rf sinewave at 2 volts p-p to a 3 dB power splitter⁶ which is in the circuit to keep all impedances appropriately matched. If cost must be kept to an absolute minimum, a simple rf tee may be substituted for the power splitter. Half of the power exiting the power splitter is fed to the rf port of the first of a pair of double balanced mixers⁷ (DBM) which are used in series as rf switches. They are turned on and off by a direct current passing through their IF ports, under the control of the adjustable gate pulse generator.⁸ The other output of the power-splitter is eventually used as the reference signal in a phase detector, as described below.

After the switching stage, the rf pulses pass through a variable attenuator and a constant-gain 2-watt rf power amplifier⁹ into the probe circuit in the magnet.

The signal from the sample comes out of the probe circuit, and is amplified by a sensitive preamp. The signal then goes into a DBM acting as a phase detector, where it is mixed with the reference signal that comes out of the second port of the power splitter. The dc magnetic field is usually set so that the NMR signal frequency is slightly offset from the transmitter frequency. When the two signals are mixed, a signal is produced at a frequency equal to the difference between f_{Larmor} and f_{rf} . Since we are looking at NMR signals in the vicinity of 10 MHz, mixing this down to a lower frequency makes it easier to see the structure of the signal.

GATE PULSE GENERATOR

The rf pulses are gated on and off under the control of a gate pulse generator (figure 2) built with five 555 timer chips. The circuit operates in two

⁵ Model SW535, Conner-Winfield, Aurora, IL.

⁶ MCL Model ZSC-2-1

⁷ MCL Model ZAD-1

⁸ Each DBM provides 67 dB of isolation between its input and its output in the off state, but it has proven necessary to use two switches in series in order to switch the RF sufficiently off to prevent RF leakthrough that contributes to noise at the preamp, and which may cause saturation of the resonance.

⁹ Motorola MHW-592

modes, *manual* or *repeated*, according to the position of a toggle switch. In the *manual* mode, the gate pulse circuit puts out two TTL pulses of different widths each time the momentary pushbutton switch is closed. Three potentiometers independently control the widths of two pulses and their separations. In the *repeat* mode, the pairs of pulses are repeated after a delay controlled by another potentiometer. In either mode, a scope trigger pulse is generated at the beginning of the first of the pair of pulses.

After the toggle switch, the signal goes on to trigger a second 555 timer, the trigger conditioner, which operates in the monostable mode. This timer starts on the fall of any signal (any one of the right size) on its input. It times for a fraction of a second, and serves only to ensure that the signal that goes on to the next 555 is clean, and free of the switch bounce that the pushbutton switch generates, so that the actual pulse train generator runs cleanly.

The heart of the pulse train generator is a trio of 555 monostable timers with adjustable periods (potentiometer controlled). Timers 1 and 2 are triggered at the same time by the fall of the clean pulse out of the trigger conditioner. Timer 1 pushes its output high for a set time, driving current out through a diode and to the rf switches (DBM's). Timer 2 pushes its output high as well, but does nothing to the rf until its output drops, causing timer 3 to trigger. Timer 3 then pushes its output high, and turns on the rf switches for a selected period. The result is a pair of pulses, each with adjustable time width, and with adjustable spacing between them. This pulse pair is generated each time a trigger pulse comes through the trigger conditioner, either from the pushbutton trigger or from the astable trigger generator.

PROBE CIRCUIT

The probe circuit is a series-tuned LC circuit, impedance matched to 50 ohms with a parallel capacitor. The inductor L in the circuit is the sample coil, a ten turn coil of #18 copper wire wound to accommodate a 12 mm sample tube. It is also important that the probe circuit be well-shielded, well-grounded and mechanically stable.

If signal-to-noise permits, the sample should occupy a small volume at the center of the coil. In any case, no part of the sample should extend outside of the coil. Otherwise, when the pulse width is set for 180° at the center of the coil, there will be some part of the sample, outside of the coil, where the pulse will be some width less than 180° , interfering with the results.

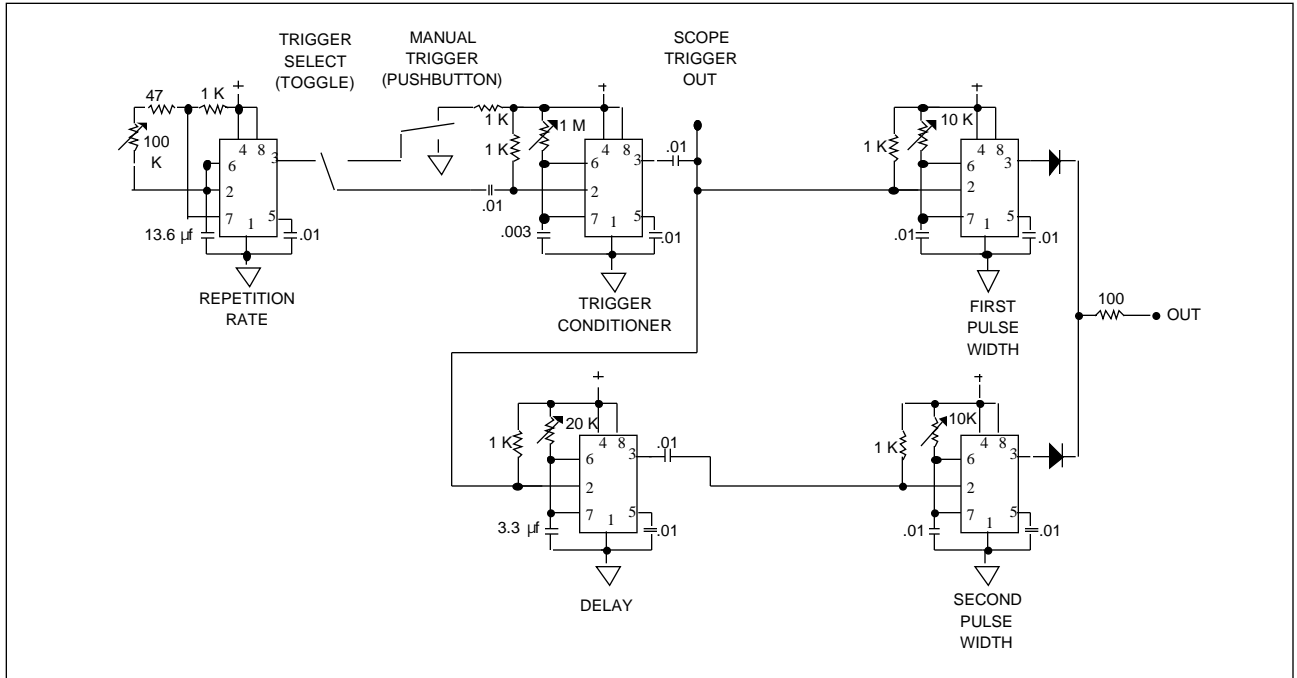


Figure 2. Gate Pulse Generator (all IC's are 555's)

If the inductance of the coil is known, then the approximate values of C_m and C_t can be calculated from the following equations:

$$2 LC_t = 1$$

$$Q LC_t^2 / (C_t + C_m) = 50 \text{ ohms}$$

The final matching of the impedance of the resonant circuit to 50 is best done by monitoring the power reflected back from the circuit and adjusting the two variable capacitors until that is minimized. This is easily done with the help of a 180° hybrid junction or "magic tee"¹⁰, (fig.5). This is a four-port circuit with the characteristic that power flowing into any branch flows out the two perpendicular ports, equally split, and does not affect the fourth port. If rf power is fed into the north port, and the probe circuit and a 50 resistive load are attached to the east and west ports respectively, then any impedance mismatch will reflect some power to the south port where it can be monitored with an oscilloscope. The two capacitors should be alternately varied until the voltage at the south port is minimized.

¹⁰ Model HH-107, Adams-Russell ANZAC, Burlington MA. This company also has an excellent catalog with a good discussion of the theory of mixers and hybrids.

Another device which can be used to help match the probe circuit is an rf directional coupler. This device has 3 ports labelled "in", "out" and "cpl". One percent of the power flowing from the "in" to the "out" port will appear at the "cpl" port. If one connects the "in" port to the probe circuit and the "out" to a suitable 50 test oscillator, then the voltage at "cpl" will give an indication of the amount of power reflected back from the probe. When this voltage vanishes, the probe is matched to 50 .

If a sweep frequency generator is available, it can be used in connection with one of the above devices to make an x-y display of frequency vs. reflected power. Since both capacitors affect both the resonant frequency and the resonant impedance to some extent, this display makes it considerably easier to meet the frequency and impedance criteria simultaneously. If a small amount of power at the crystal frequency is mixed in with the sweep frequency, a beat pattern will appear as a convenient marker.

CROSSED DIODES AND QUARTER-WAVE LINES

In 1968 Lowe and Tarr¹¹ published a scheme for isolating the transmitting and receiving arms of the apparatus from one another while both are

¹¹ I. J. Lowe and C. E. Tarr, Jour. of Physics E, 1, 320 (1968)

simultaneously connected to the probe. This method is so effective that its use has become practically universal, so it will only be briefly summarized here.

Between the power amp and the probe there is a pair of crossed diodes which show a high series impedance when the transmitter is off and a low impedance during the pulses. All silicon diodes exhibit this same non-linear break in their current-voltage characteristics at about .6v. Thus, between pulses, the power amplifier and its associated noise is isolated from the probe circuit.

The signal out of the probe circuit passes through a quarter-wavelength line to reach another pair of crossed diodes which are shunted to ground at the input of the preamp. The diodes short the preamp end of the cable when the transmitter is on, thus protecting the delicate preamp from the high rf power put out by the power amplifier. Between pulses, the voltage across the diodes is too small to turn them on, and they act like an open circuit, allowing the small NMR signal to pass to the preamp.

Any quarter-wave transmission line acts as a transformer which transforms its output impedance according to the equation

$$Z_{\text{input}} = \frac{Z_0^2}{Z_{\text{output}}}$$

where $Z_0 (= 50 \Omega)$ is the characteristic impedance of the transmission line. Thus, during the pulses, when the shunt diodes are acting as a short, the input impedance of the transmission line is infinite acting as if the receiving circuit were completely disconnected from the probe.

THE PREAMP

A Trontech W110F preamp, with a gain of 55 dB and a noise figure of 1.2 dB was used in one version of the spectrometer allowing one to achieve the necessary level of amplification of the NMR signal in one stage. The NMR signal is expected to be of the order of -100 dBm, so it needs to be amplified considerably before it can be observed on an oscilloscope. The amplification can, of course, be performed in multiple stages (most high gain preamps actually have multiple stages). If this is done, it is particularly important to pay attention to amplifier noise figures and the order in which the amplifiers are cascaded. It turns out that a fairly low total noise figure can be achieved with a low noise first stage and a fairly noisy second stage, since the first stage raises the signal to a point where it is relatively safe from the dangers of noise. The total noise figure, ($f =$

signal-to-noise ratio at input/signal-to-noise ratio at output) is only a weak function of second stage noise:

$$f_t = f_1 + \frac{f_2 - 1}{g_1}$$

where f_1 is the noise figure of the first stage, f_2 is that of the second, g_1 is the gain of the first stage.

CARR-PURCELL-MEIBOOM-GILL

A more accurate method for measuring T_2 was developed by Carr and Purcell¹² and later improved by Meiboom and Gill¹³. A version of this circuit using 555 IC timers and Mii-Circuit Lab doubled-balanced mixers has been built.

SAMPLE OUTPUT

Figure 3 shows the typical spin-echo results obtained with a sample of glycerin. The Larmor frequency has been offset from the 10 MHz of the oscillator in order to show a pattern of beats. Figure 4 illustrates a sequence of $180^\circ - 90^\circ$ pulses also in glycerin. This is the most common method for measuring T_1 . The exponential approach to equilibrium after a reversal of the magnetization is clearly demonstrated.

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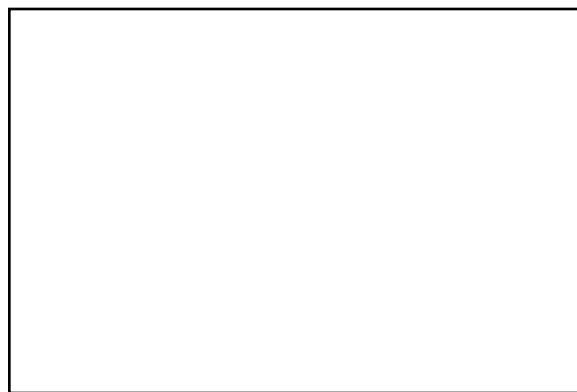


Figure 3. Typical 90-180 spin echo (glycerin)

¹² H. Y. Carr and E. M. Purcell, Phys. Rev. **94**, 630 (1954)

¹³ S. Meiboom and D. Gill, Rev. Sci. Instr. **29**, 688 (1958)



Figure 4. Sequence of 180-90 pairs (glycerin)