Simple, Spin-Echo Spectrometer

B. H. MULLER
J. D. NOBLE*
L. J. BURNETT†
J. F. HARMON‡
D. R. McKAY
Department of Physics and Astronomy
University of Wyoming
Laramie, Wyoming 82071
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In an attempt to make pulsed nuclear magnetic resonance (NMR) widely available for teaching purposes, the design and construction of a simple and inexpensive pulsed NMR spectrometer are described in considerable detail. This spectrometer requires only one twelve-volt power supply. A complete procedure for tuning the spectrometer to resonance is also presented.

Solid state electronics has permitted the design and construction of a simple and inexpensive pulsed nuclear magnetic resonance (NMR) spectrometer which we think should prove to be a very effective teaching tool for a wide range of students.[1] A reasonable understanding of pulsed-NMR signals can easily be provided,[2–5] and the display of spin echoes is a vivid exhibition of nuclear magnetism and magnetic resonance.[6] Such signals are also a good demonstration of the model spin system used in Kittel's text on thermal physics.[7] The spin-echo spectrometer lends itself naturally to a variety of experiments including the measurements of relaxation times. Thus, the time constants of the Bloch equations can be clearly illustrated. By the addition of a pair of gradient coils, self-diffusion constants could be measured.[3] If an appropriate temperature control system were constructed, the temperature dependence of these quantities could be observed and melting points could be determined as discussed in Refs. 8 and 9. No doubt other possibilities will suggest themselves for a number of open-ended experiments.

To our knowledge, a genuinely simple and inexpensive pulsed-NMR spectrometer is not now available. Commercial pulsed-NMR spectrometers are complicated and expensive (typically $\$10^4$ to $\$10^5$ or more). While an "inexpensive"[10] and a "simple"[11] NMR pulsed spectrometer have been described in the literature, their cost is of the order of $\$10^3$ and they are much more complicated than ours. Continuous wave (cw) NMR spectrometers of reasonable price are available and widely distributed, although we suspect that they have not been particularly effective teaching instruments.

Pulsed NMR signals are produced when a sample of nuclei of gyromagnetic ratio γ, located in a magnetic field H_0, are exposed to radio frequency (rf) pulses of angular frequency ω (such that $\omega=\gamma H_0$) of the appropriate intensity (H_1), duration, and timing. These signals are observed after they are amplified and exhibited on an oscilloscope whose sweep has been triggered at the onset of each set of pulses.

Figure 1 is a block diagram of the spectrometer. Any triggered oscilloscope will do. While an electromagnet is preferred for ease of tuning, we have also successfully used a permanent magnet of about 2500 G with our spectrometer. The homogeneity requirement is not severe (much less than that needed for cw resonance) and an expensive magnet is not required. Many beginning physics laboratories and most advanced physics laboratories will have a suitable electromagnet on hand. The constructed apparatus consists of four small boxes (whose total weight is under five pounds), sample coil, connecting cables, and a single 12-V power supply (Heathkit IP-18) whose total cost, in parts, is about $200.

Figure 2, which gives a symbolic representation of the envelope of the rf pulses and of the transient nuclear signals, shows the timing instructions the timer must provide the transmitter. At $t=0$, the pulsed oscillator is told to begin and simultaneously the oscilloscope is triggered. At $t=t_1$, the oscillator is shut off and if resonance has been obtained, a nuclear induction signal is seen on the scope once the amplifier has recovered from the saturating effect of the first pulse. At $t=\tau$, a second rf pulse of the same frequency and intensity but different duration is initiated. At $t=\tau+t_2$, this second pulse is terminated and followed at resonance by another free induction decay (unless by careful adjustment, the second pulse is a π pulse). Then a spin echo appears centered at 2τ. At $t=T$ (equal to the reciprocal of the repetition rate), the sequence of two pulses is repeated. The timer must provide the proper signal to the transmitter at the variable times

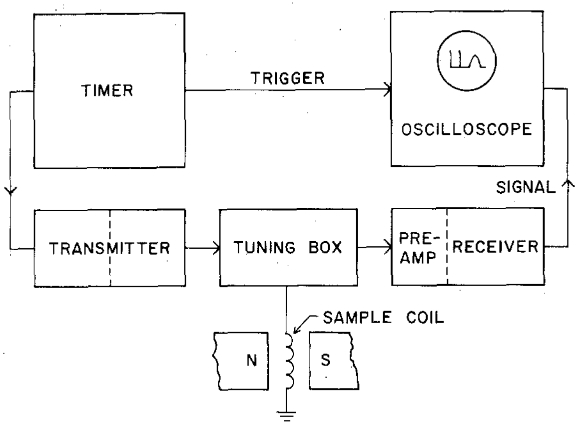

FIG. 1. Block diagram of the apparatus needed for a NMR pulsed spectrometer. The amplifier consists of a preamplifier and a receiver each housed in its own small box with the boxes bolted together.

of t_1, τ, $\tau+t_2$, and T. Figure 3 is a photograph of these signals from the protons of a sample of glycerin taken with this spectrometer.

TIMER

The 2N5431 unijunction transistor and its associated circuit (shown as part of Fig. 4) form a relaxation oscillator used to generate the repetition rate. The period of the oscillator and therefore T are determined by the sum of the 10 kΩ resistance and the variable resistance RT and the 0.47 and 10 μF capacitors. Switch No. 2 ($SW2$) not only allows a choice of capacitance but at its bottom position allows a single shot

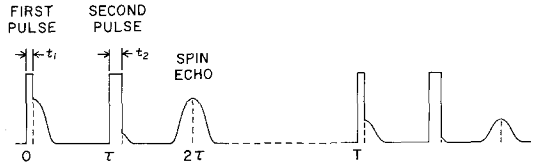

FIG. 2. The timing sequence and the NMR signals at resonance. Typical values for exhibiting a spin echo with a $\pi/2$, ($\gamma H_1 t_1=\pi/2$), and a close to π ($\gamma H_1 t_2=\pi$) pulse with our spectrometer, magnetic field, and a glycerin sample: $t_1=10$ μsec, $t_2=19$ μsec, $\tau=0.5$ msec, $T=0.1$ sec.

pair of pulses whenever the operator pushes the switch No. 1 ($SW1$).

The heart of the timer consists of fourteen NOR gates which are packaged in sets of four in two Motorola MC 717P and two Motorola MC 724P integrated circuits (IC's).[12] The configuration and wiring layout that were found to work well are described in Figs. 4 and 5. Though relatively expensive, the recommended circuit board and its associated connector were found to be very helpful parts of the timer. The pairs of NOR gates ($1B$, $4C$) and ($1C$, $2D$) are used to sharpen the initial timing pulse and to couple it out in the first instance to the scope and in the second instance to the two timing channels (I and II). The pairs of NOR gates ($2A$, $3D$), ($2C$, $1D$), and ($2B$, $3C$) together with the associated resistances and capacitances are delay generators which control the times t_1, τ, and t_2. These times

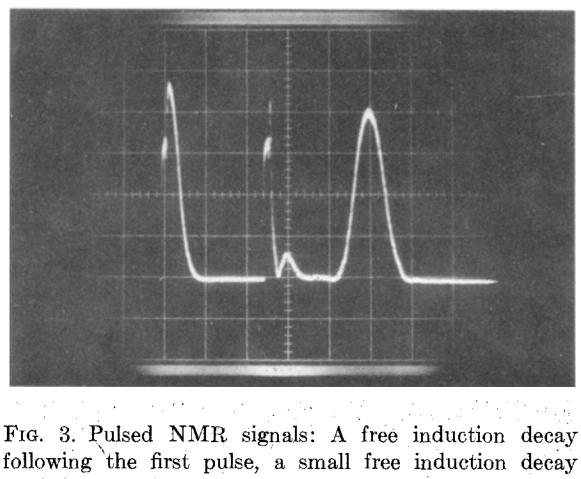

FIG. 3. Pulsed NMR signals: A free induction decay following the first pulse, a small free induction decay following the second pulse, and a spin echo from a sample of glycerin. The vertical and horizontal scales of the oscilloscope screen were 0.5 V/cm and 200 μsec/cm, respectively.

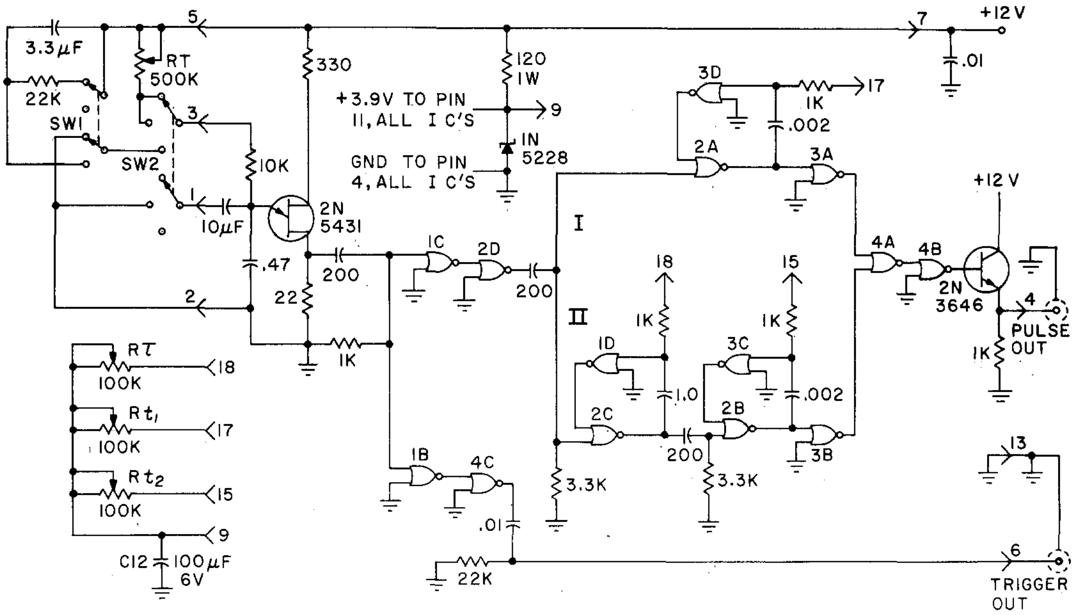

FIG. 4. The timer: The symbol (2A) describes the A NOR gate of IC-2. Its wiring connections are given explicitly in Fig. 5. Potentiometers RT (500 kΩ), Rt_1, R_T, Rt_2 (each 100 kΩ) allow the adjustment of times T, t_1, τ, and t_2, respectively. In all circuits (unless otherwise specified), (a) all resistances are in ohms ±10% and are $\frac{1}{4}$ W; (b) all capacitances numerically less than or equal to 1.0 are in microfarads and those greater than 1.0 are in picofarads. A complete parts list is available from the authors.

can be changed by adjusting the potentiometers Rt_1, R_T, and Rt_2, respectively. NOR gates 3A and 3B invert and couple the timing pulses from channels I and II to the mixing NOR gate 4A. NOR gate 4B inverts and couples the resultant timing train to the emitter follower 2N3646 which provides the appropriate output impedance to drive the transmitter.

TRANSMITTER

The circuit for the transmitter is given in Fig. 6. A positive rectangular pulse from the timer turns on transistor $Q2$ for the duration of the pulse. The grounded-collector Hartley oscillator formed by $Q1$ and its associated circuit then oscillates with a frequency determined mainly by the 50 pF capacitance and the primary inductance of transformer $T1$. The 2N2102 buffer amplifier and the 2N5320 output amplifier both operate Class C. The other components have been picked for proper impedance matching. Figure 7 is a picture of the transmitter.

TUNING BOX

In this system, a single coil is used both for transmitting rf energy to the sample and for re-

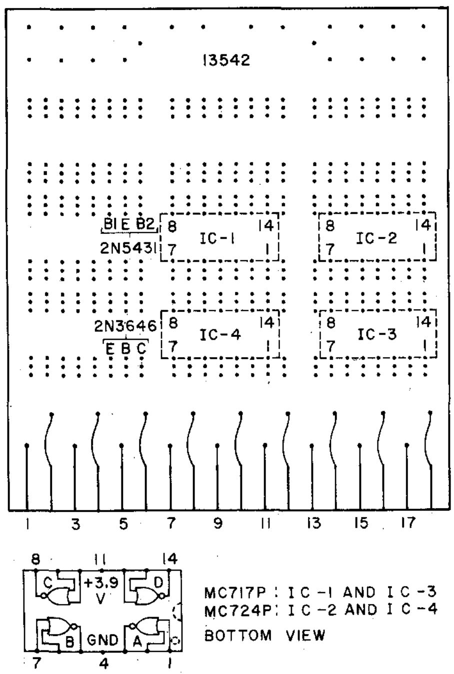

FIG. 5. The timing-circuit layout: The description is for the "Triad Integrated Circuit Card CO-13542" which is to be used with its associated Winchester connector. For example, the NOR gate 2A input connections are at IC-2: 1 and 2 and the output is at IC-2: 3; ground and +3.9 V are at the shared positions for IC-2: 4 and 11.

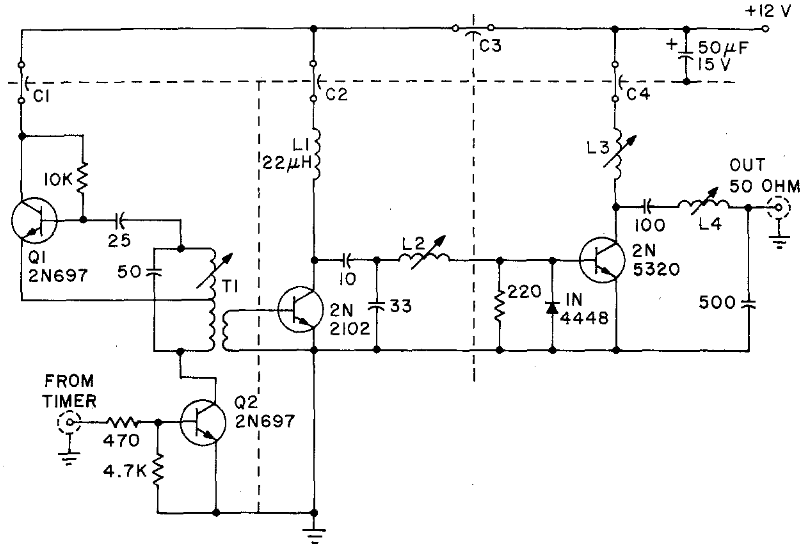

FIG. 6. The transmitter: *C*1, *C*2, *C*3, and *C*4 are 1000 pF feed-thru capacitors. *L*2, *L*3, *L*4, are permeability-tuned inductances made by winding 20, 20, and 15 turns, respectively, of No. 28 wire on J. W. Miller coil form 4400-2. The primary of the transformer *T*1 is made by winding 20 turns of No. 28 wire on a Miller 4400-2 form. The tap is five turns from the *Q*2 end. The secondary has two turns. The transistors *Q*1, 2N2102, and 2N5320 are mounted in a Wakefield type 254-*S*1 heat sink. If different heat sinks are used, their capacity to ground must be taken into consideration.

ceiving the nuclear signal. The coupling circuit is shown in Fig. 8 and is similar to that of Gray, Hardy, and Noble.[13] A resonant impedance transformer is formed by 82 pF and *L*5 which transforms from the low (50 Ω) output impedance of the transmitter to the higher (5 kΩ) parallel impedance of the sample circuit (*C*5, *C*6, and *L*6). Crossed silicon diodes are used to switch the transmitter into the circuit when large rf pulses are applied. In the receiving mode, the small nuclear signal is not large enough to turn the diodes on and the transmitter is disconnected. Since the capacitance (about 30 pF per ft) of the coaxial cable used to connect the sample coil to the system is in parallel with *C*5 and *C*6, the length of this cable is significant. In addition, it is

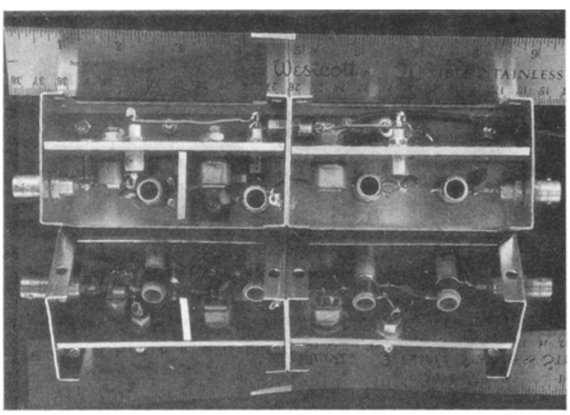

FIG. 7. A bottom view of the transmitter taken with the aid of a mirror. From left to right, the visible coils are *T*1, *L*2, *L*3, and *L*4.

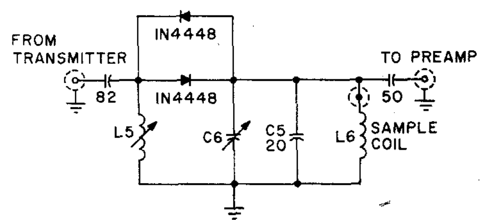

FIG. 8. The tuning box: *L*5 consists of 15 turns of No. 28 wire on a Miller coil form 4400-2. *C*6 is a E. F. Johnson 160-0110-001 tuning capacitor, 2.7–19.6 pF. Our sample coil *L*6 consists of 17 turns of No. 24 enameled wire forming a cylinder approximately 10-mm long with an inside diameter of 11-mm. A 10-mm test tube covered with 5 or 6 layers of Saran wrap lubricated with light oil was used as a coil form. After the coil was soldered to the 2-ft RG-58C/U connecting coaxial cable, it was encapsulated in a thin cylindrical shell of epoxy cement.

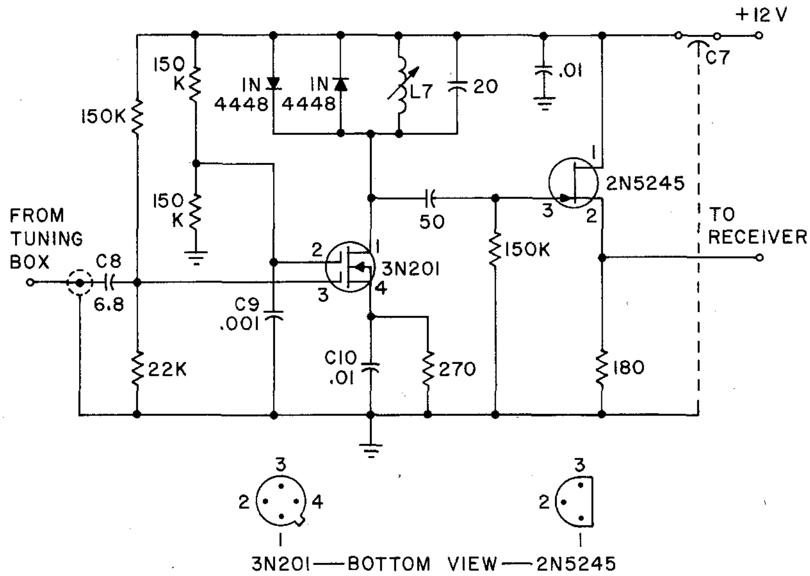

FIG. 9. The preamplifier: *L7* is a Miller-type 4506 adjustable rf coil, 9.9–15 μH.

imperative that this cable and anything else close to the sample coil be nonmagnetic. The fixed capacitance *C*5 should be picked so that *C*6 tunes to resonance at about its midrange position. The sample coil described in the caption to Fig. 8 is the one used with this apparatus, but many different geometries will also work as long as it will tune to the resonant frequency with the cable capacitance, *C*5 and *C*6.

PREAMPLIFIER

The preamplifier circuit shown in Fig. 9 uses a 3N201 transistor, an *N*-channel MOSFET, in the input stage. It is protected from excessive input voltage by internal back-to-back diodes between the gates and the source. The amplified nuclear signal developed at its drain is coupled by 50 pF to the source follower 2N5245 which is used to match the low input impedance of the following IC amplifier. The preamplifier has a gain of about 25 dB and a bandwidth of about 1 MHz. When building this circuit, it is important to keep leads short and to use careful layout. Both *C*9 and *C*10 should have very short leads. *C*8 should not be made much larger than 6.8 pF. Since the 3N201 transistor has high gain at very high frequencies, care must be taken to prevent high frequency parasitic oscillations. It may be necessary to use suppressors such as ferrite beads or highly damped coils on the drain (pin 1) or gate No. 1 (pin 3) circuits if these parasitic oscillations occur. A symptom for such oscillations is low and erratic gain.

RECEIVER

The receiver circuit shown in Fig. 10 consists of two IC amplifiers in cascade, with an envelope detector built into the second IC amplifier. The first IC, a RCA CA3023, is used as a gain-controlled, bandpass amplifier. The gain is controlled by the potentiometer *R*1. The second IC, a RCA CA3002, is adjusted by varying *R*2 until the voltage at pin 8 is about 0.1 V higher than that at pin 7. As with the preamplifier, special care must be taken to keep the leads short and the layout such that unwanted feedback does not take place. For example, sockets were not used for the IC's and their leads were soldered directly to those of the other components in the circuit. The two IC's should be at least an inch apart. A bottom view of the preamplifier and the receiver is given in Fig. 11.

TUNE-UP PROCEDURE

While the best tuning of the spectrometer is done with the aid of a NMR signal, a careful pre-

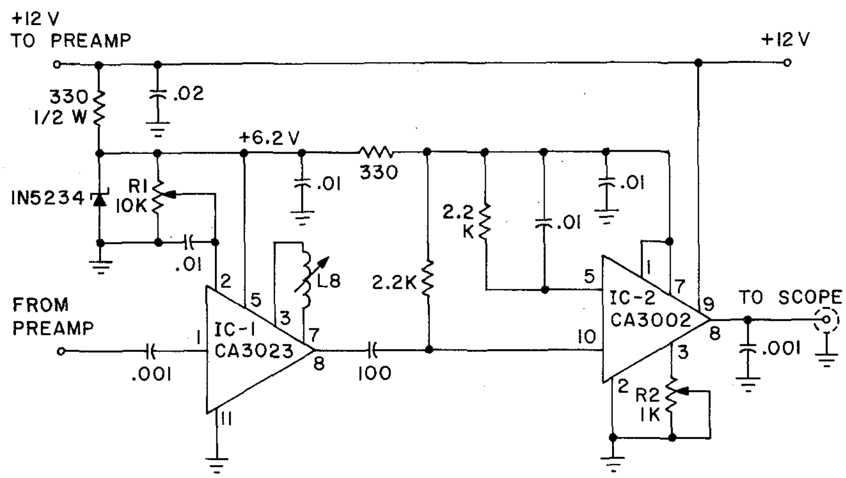

FIG. 10. The receiver: *L8* is a Miller-type 4511 adjustable rf coil, 66–114 μH. *R1* is a 10 kΩ gain control trimpot. *R2* is a 1 kΩ bias adjust trimpot.

liminary tuning which adjusts all the tuned circuits to the desired frequency is needed first to find a signal. This tuning is most conveniently done with the help of a high frequency oscilloscope.

(1) A frequency must be chosen. If a permanent magnet is to be used, its field must be accurately known and the resonant frequency calculated. For use with an electromagnet, the choice of frequency is much less critical; 10.7 MHz is a good value for our circuit. There should be no difficulty within a few hundred kilohertz of this value, but for other frequencies redesign may be necessary.

(2) The core of transformer *T1* is adjusted until the desired frequency is obtained. A communications receiver calibrated against WWV will serve to measure the frequency.

(3) Inductances *L2*, *L3*, and *L4* are tuned to obtain a maximum rf voltage across a 50-Ω resistor temporarily placed across the transmitter output. (With optimal tuning, we observed 45-V peak-to-peak on our oscilloscope.)

(4) The transmitter is then connected to the tuning box and its two IN4448 diodes are temporarily disconnected. *L5* of the tuning box is adjusted for maximum voltage across its terminals. A low-capacity probe should be used for this adjustment.

(5) Check the frequency and, if necessary, repeat steps 2 through 5.

(6) Disconnect the transmitter from the tuning box, reconnect the diodes, and connect the preamp to the tuning box with a coaxial adapter. With the transmitter on (and thus its output *weakly* coupled into the sample coil) tune *C6*, the variable capacitor in parallel with the sample coil, and *L7* of the preamp for maximum voltage at the output of the preamp. Check that *C6* is not at either end of its tuning range.

(7) By adjusting *R1*, set the gain of the re-

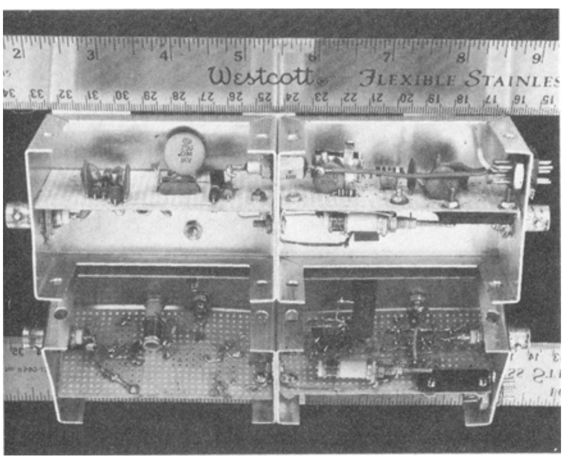

FIG. 11. A bottom view of the amplifier (preamplifier on the left and receiver on the right) taken with the aid of a mirror.

ceiver high enough that some noise is visible on the oscilloscope and low enough that the amplifier does not oscillate.

(8) Set $L8$ at half its maximum value.

(9) With the values given in the caption to Fig. 2, carefully search for a signal by varying the magnetic field until resonance is obtained. Once a signal is found (it will go away if the sample is removed) and the magnetic field is stable, improve the signal by redoing the appropriate adjustments to obtain nuclear signals of high signal-to-noise ratio (25 to 1 can be easily achieved and with a simple low-pass RC filter, 100 to 1 or better is obtainable). Since the amplitude of the spin echo is proportional to $\sin\theta_1 \sin^2(\theta_2/2)$ where $\theta_1 = \gamma H_1 t_1$ and $\theta_2 = \gamma H_1 t_2$, the echo height will be a maximum for $\theta_1 = \pi/2$ and $\theta_2 = \pi$. For $\theta_1 = \pi/2$, the first free induction decay will be a maximum, and for $\theta_2 = \pi$, the second free induction decay will be small (zero for a true π pulse which is only obtainable with a very homogeneous magnetic field). Improvement in the tuning of $L2$, $L3$, $L4$, and $L5$ increases H_1 and therefore decreases the pulse widths t_1 and t_2 needed for $\pi/2$ and π pulses. Improvement in the tuning of $C6$, $L7$, and $L8$ will increase the amplifier gain of the signals and therefore the signal-to-noise ratio. Finally, check that the signal is not being clipped by receiver saturation. If necessary, reduce the gain of the receiver by adjusting $R1$.

* On sabbatical leave from the Department of Physics, Western Illinois University, Macomb, IL 61455, 1971–1972.

† Now at the Department of Physics, California State University, San Diego, CA 92115.

‡ Now at the Department of Physics, Idaho State University, Pocatello, ID 83201.

[1] The first edition of this spectrometer is being used by W. Yost in his physics classes at Cheyenne Central High School as a demonstration device. After seeing the spectrometer in operation at the 1973 AAPT Apparatus Competition, F. W. Martin requested circuits in order to build one for his graduate laboratory at the University of Maryland.

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