



## 6.2 : Marginal oscillator-detector

A marginal oscillator makes use of barely enough positive feedback so that an extremely small absorption of energy causes the amplitude of oscillations to vary by a large amount making the direct observation of the NMR signal quite easy. Marginal oscillator circuits have been extensively used as NMR detectors, a well known version being the Pound-Knight-Watkins (PKW) spectrometer [6,7,8].

The principle of this device, called usually as the 'Pound Box' is the following: the level of oscillations of an r.f. oscillator decreases when its load is increased. The lower the initial level of oscillation, sharper is the decrease. A sample containing nuclear spins placed in the coil of the tuned circuit of the oscillator will absorb r.f. energy at resonance and may be considered as an additional load. The drop in the level of oscillations is then used to detect the resonance [1].

A marginal oscillator circuit has the following major advantages as compared with the earlier bridge circuit:

- (a) They are very simple to implement
- (b) The circuit responds only to a variation of the absorption component  $\chi''$ , since the effect of the dispersion component is to cause a frequency modulation which is not detected. The pure absorption is therefore recorded and there are no problems of maintaining good balance to ensure only this type of the signal.
- (c) Wide frequency sweeps are possible when searching for unknown resonances, provided adequate feedback networks are included to keep the oscillation level constant.

**6.2.1 :** Transistorised marginal oscillator NMR detector systems have become much more compact and simple to fabricate. For the present thesis work the initial NMR signal, that of proton in doped water, was observed using a transistorised marginal oscillator circuit. This circuit closely follows the one put forward by Ijaz-ur-Rahman [9], which claims to give improved performance compared with earlier marginal oscillator circuits. The circuit offers the advantage that the amount of feedback and the amplitude of oscillations across the LC circuit can be independently controlled and so its setting as a marginal oscillator becomes extremely easy.

The actual circuit fabricated is shown in Fig.6.1. The circuit has its coil tapped at its centre from where sufficient current can be drawn by transformer (step down) action, to be fed back to the emitter of the oscillator transistor. The feedback capacitor of 400pF and a variable resistance ( $R_2$ ) of 3K $\Omega$  enable one to control the phase and magnitude of this feedback. The circuit was fabricated using BC 178 transistors for both the oscillator transistor and the detector transistor.

The amount of d.c. bias of the detector and the amount of r.f. signal applied to it are important, as the gain of this stage depends on its quiescent point. The optimum combination of the d.c. bias and the r.f. signal amplitude are best found in practice as follows: After having placed the sample tube containing tap water (doped with ferric nitrate solution) in the coil, kept between the pole pieces of the magnet, power to the circuit is switched on. A d.c. voltmeter (20000 $\Omega$ /V) is connected between the collector and emitter of  $T_2$ . If this transistor is not conducting, nearly full supply voltage (about 8V) will be indicated. Otherwise the preset potentiometers  $R_1$ ,  $R_2$  and  $R_3$  are adjusted to obtain this condition.  $R_3$  alone is then adjusted so that the meter reads about a volt less than maximum,

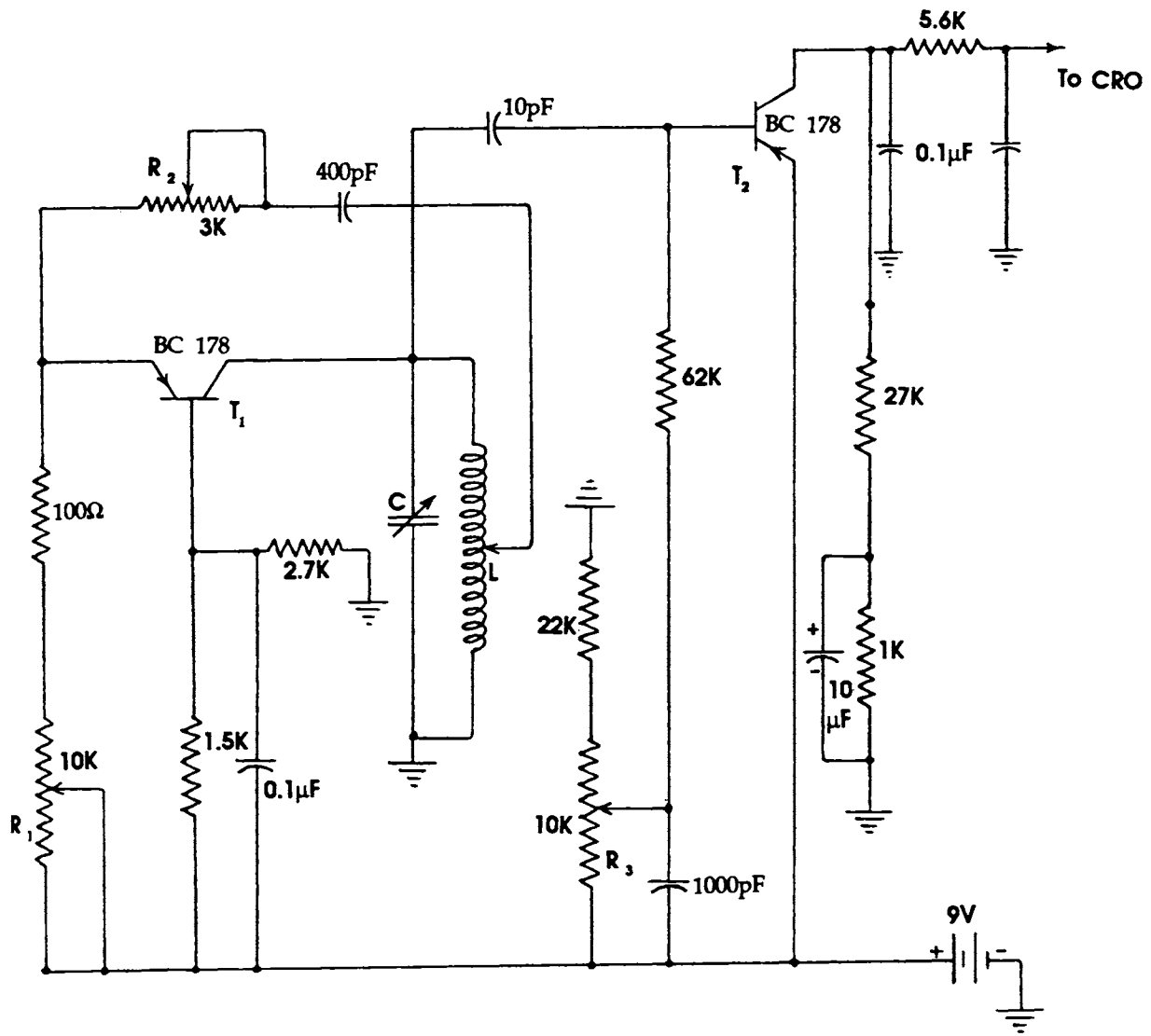


Fig. 6.1 : Circuit diagram of the marginal oscillator

indicating that  $T_2$  is slightly conducting.  $R_1$  and  $R_2$  are then adjusted so that the meter reads about 2V.

After establishing the above bias condition the magnet power supply is switched on. The magnet current is now increased from zero, rather slowly, until the resonance peak appears on the oscilloscope screen. The peak will move sideways on changing the magnet current. In case the amplitude of the resonance peaks is too small, the sensitivity of the marginal oscillator is increased. This is easily done by adjusting  $R_2$  so that the meter reading goes up, say by 1V, and then  $R_1$  is adjusted so that the reading gets restored to its initial value.

Using the above set up proton resonance signal was observed in doped water. However the S/N ratio of the signal was not satisfactory. This was understood to be so because when the circuit is set at a high sensitivity level for obtaining better signal strength it will generate a good deal of noise, mainly due to microphonics, which will mar the appearance of the signal on the oscilloscope screen. Besides, a high sensitivity state will lead to instability in the circuit, whereby the oscillator may flip in and out of oscillation sporadically and the same may have to be reset again and again.

### 6.2.2 : Disadvantages of the marginal oscillator-detector

As mentioned above, the S/N ratio of the marginal oscillator circuit shown in Fig.6.1 was not adequate enough for the satisfactory recording of the resonance signal. Also it is known that the marginal oscillator-detector suffers from a number of drawbacks, as mentioned below, which deter its usage as an efficient NMR detector.

- (i) The main weakness of the marginal oscillator circuit is its non viability for producing very small r.f. fields, which sometimes become necessary for avoiding saturation in specimens like solids possessing long spin-lattice relaxation time. With a marginal oscillator maintaining stable operation it is not possible to reduce the r.f. amplitude across the specimen coil much below 0.1V, whereas for the investigation of several solids it usually becomes necessary to work at a low r.f. level of the order of a few millivolts if saturation is to be avoided [4].
- (ii) Since the operation of the marginal oscillator depends on the non-linear characteristics of the device used [10], their sensitivity tends to be less than that of Q-meter circuits.
- (iii) For a given sample coil the marginal oscillator operates best at only one r.f. level. This precludes convenient choice of r.f. level on the basis of sample saturation properties [11].

These disadvantages of marginal oscillator circuits in general and the poor performance of the circuit shown in Fig.6.1 in particular forced us to go for Robinson oscillator, which was learnt to be capable of providing far better performance in comparison with other NMR detectors.

### 6.3 : Robinson oscillator-detector

Robinson has devised a clever combination of Q-meter and marginal oscillator circuits having all the advantages of both of these, plus the ability to operate at very low r.f. levels on the sample coil [12].

Most of the drawbacks of the marginal oscillator-detector can be overcome by using a Robinson oscillator, where the r.f. feedback needed to sustain oscillations is

derived from a limiter stage. The limited oscillator [13] does not require continuous adjustment nor does it depend critically on the exact characteristics of particular devices. Besides, Robinson oscillators are considerably less sensitive to the unwanted dispersion component [5]. Because of these desirable features Robinson oscillators [13,14] have recently gained much popularity as an efficient NMR detector.

### 6.3.1 : An FET type Robinson oscillator

A typical Robinson oscillator circuit consists of a limited self-oscillator where separate stages are used for regeneration and amplitude control. The essence of the Robinson system is that the only non-linearity in this occurs in the form of a simple limiter, so that both the oscillation conditions and the sensitivity to magnetic resonance absorption are predictable in a straight forward way. This is not the case in other forms of self-oscillating detectors, which are maintained in a more or less unpredictable or 'marginal' mode by the arbitrary operation of a manual or automatic control system.

For the present thesis work a Robinson oscillator employing FETs as active elements was tried out first. The actual circuit fabricated is shown in Fig. 6.2. This circuit is essentially a simplified version of the one put forward by Robinson [15]. Here bipolar transistors in the limiter stage of the Robinson's original circuit [15] have been replaced with the FETs  $Q_1$  and  $Q_2$  (BEW 10s), which possess less  $1/f$  noise than a bipolar pair. This long-tailed pair of FETs acts both as the limiter and as a moderately efficient detector.

If the r.f. input to the gate of  $Q_1$  has an amplitude exceeding about 100mV the current through  $Q_2$  is switched on and off. A fraction (determined by  $C_1$  and  $C_2$ ) of this clipped current is feedback to the tank circuit and this sustains oscillations, the amplitude of which is linearly proportional to the  $Q$  of the circuit. Thus the resonant circuit responds directly to changes in  $Q$  due to NMR absorption. On the negative half-cycle  $Q_1$  is switched off but on the positive half-cycle the current through it continues to rise at a rate governed by  $R$ , even after  $Q_2$  is turned off. Thus the drain current of  $Q_1$  contains a component which is the detected signal.

The transistors  $T_1$  and  $T_2$  (BC 109) and the FET -  $Q_3$  (BFW 10) form the audio amplifier arrangement for enhancing the detected signal. The preset potentiometer  $R_1$  enables one to set the required r.f. level across the specimen coil. The trimmer capacitor  $C_2$  helps to select an appropriate  $C_1/C_2$  ratio for achieving an optimum feedback and hence sensitivity. The +18V dc power for the circuit was provided from two 9V cells.

Using the above mentioned NMR detector circuit a highly satisfactory proton resonance signal from liquid samples could be observed on an oscilloscope. The S/N ratio of the signal was far better than that of the signal obtained using the marginal oscillator circuit of Fig.6.1. The Robinson oscillator circuit was found to have a high degree of frequency stability and very low sensitivity to microphonics as compared with the earlier marginal oscillator. Besides a better control over the r.f. level could be achieved using this circuit. The tracing of a typical derivative signal corresponding to proton resonance in doped water obtained using this circuit is shown in Fig.6.3.

### 6.4 : An improved Robinson Oscillator using MOSFETs

Though the circuit shown in Fig. 6.2 was found to give very good results for the recording of NMR signals in liquids, it could not be used effectively for recording

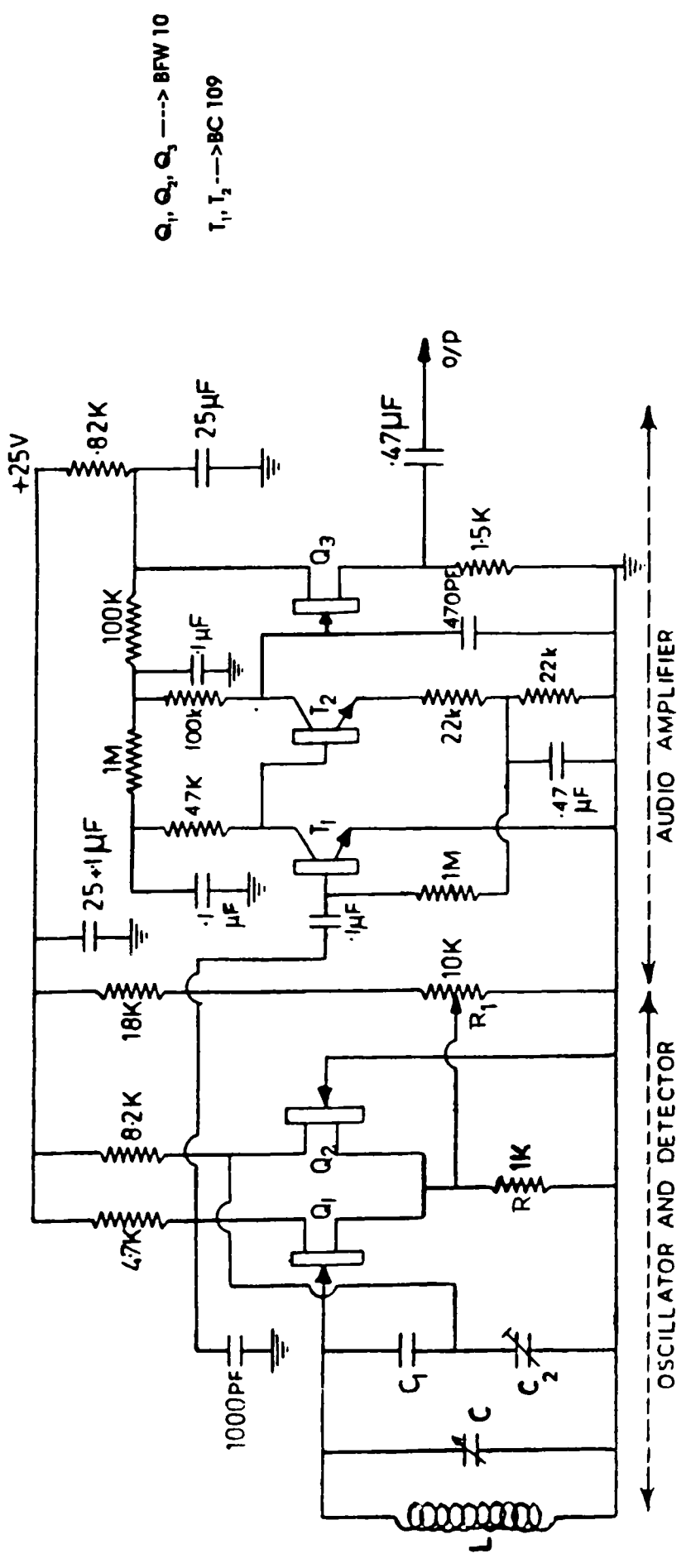


Fig. 6.2 : Circuit diagram of the FET based Robinson's oscillator

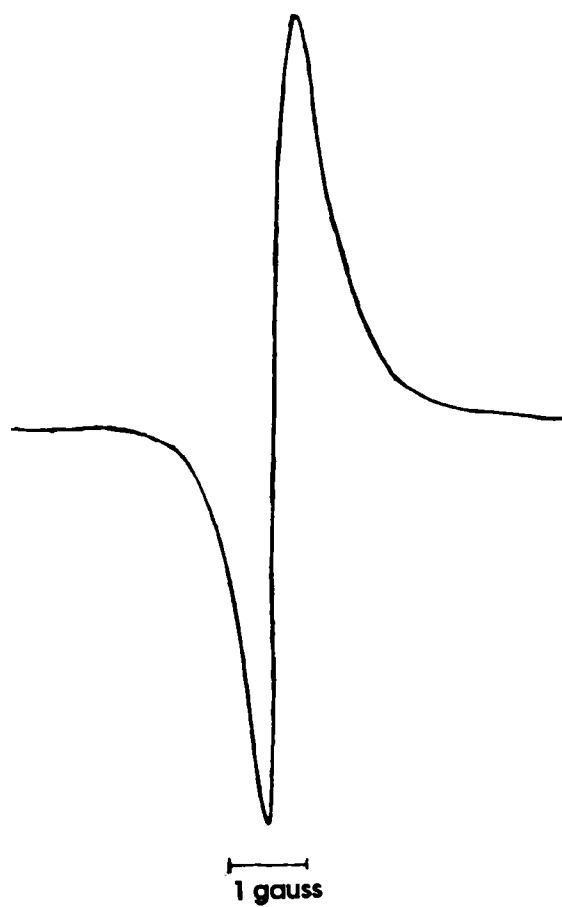


Fig. 6.3 : Tracing of the derivative curve of proton resonance from water doped with  $\text{Fe}(\text{NO}_3)_3$  solution (resonance frequency  $\sim 8\text{MHz}$ ; r.f.level  $\sim 80\text{mV}$ ) obtained using the FET based Robinson oscillator

resonance signals from solids. This was due to the fact that the r.f. level of this circuit could not be brought down to the order of a few millivolts, as required in solids for avoiding saturation. It was found that the r.f. level of the circuit could only be brought down to a value of about 60mV. This circumstances has prompted us to search for a different circuit capable of operating at very low r.f. levels.

Though several modified versions of Robinson oscillators [16, 15, 17] including those employing integrated circuits have been published, most of them suffer from drawbacks like circuit complexity or requirement of highly critical components. The later versions using integrated circuits suffer from reduced sensitivity resulting from relatively low input impedance and the poor noise characteristics of the integrated circuits used.

With the intention of achieving enhanced sensitivity for the detector circuit as well as to obtain the ability to operate the oscillator at very low r.f. levels it was decided to design an improved, but simple, version of Robinson oscillator employing MOSFETs as the active devices for the present work. In this circuit [18] the very high input impedance and better noise characteristics combined with high transconductance of the MOSFETs have been fully exploited for achieving the required sensitivity needed for recording weak NMR signals.

#### **6.4.1 : Description of the circuit**

The circuit, which is shown in Fig. 6.4, comprises of three stages: (i) an r.f. level limited oscillator, (ii) a detector and (iii) a low noise amplifier.

Since the oscillator stage determines the minimum r.f. level at which the circuit can be operated and the maximum sensitivity that can be achieved, much importance had been given to proper design of the same. The input impedance of the active devices used for the oscillator has to be very much higher than the shunt impedance of the nuclear resonance circuit, which is typically a few thousands ohms in practical circuits. Later versions of Robinson oscillators have been using FETs as the active devices. However their input impedance ( $\sim 10^6$  ohms) is not high enough to prevent Q value of the tank circuit getting affected adversely. As a result the Q factors for such circuits are only moderate. Besides operation at very low r.f. levels using such circuits is nearly impossible mainly due to poor transconductance of conventional JFETs.

To overcome these disadvantages MOSFETs (3N 200) were utilised as the active devices for the level limited oscillator in the present circuit. The MOSFETs have an input impedance ( $\sim 10^{13}$  ohms) that is much higher than that of JFETs. This serves to enhance the Q of the new detector circuit considerably. MOSFET also provide excellent high frequency characteristics as well as very low  $1/f$  noise, factors that make them desirable choice for the long-tailed pair in a Robinson oscillator. Besides the high transconductance value, better noise characteristics of MOSFETs and their high tolerance to microphonics are added benefits.

As in many versions of Robinson oscillator [16, 5], in the present design the operating conditions are chosen to give nearly perfect limiting independent of the oscillation level. As a result the absorption signal does not appear as a component of the drain current and hence a separate demodulator is used. Conventional circuits have been employing highly specialised diodes, capable of working at very low levels, as the rectifier [16,5]. To avoid the requirement of critical components and also to facilitate the designing



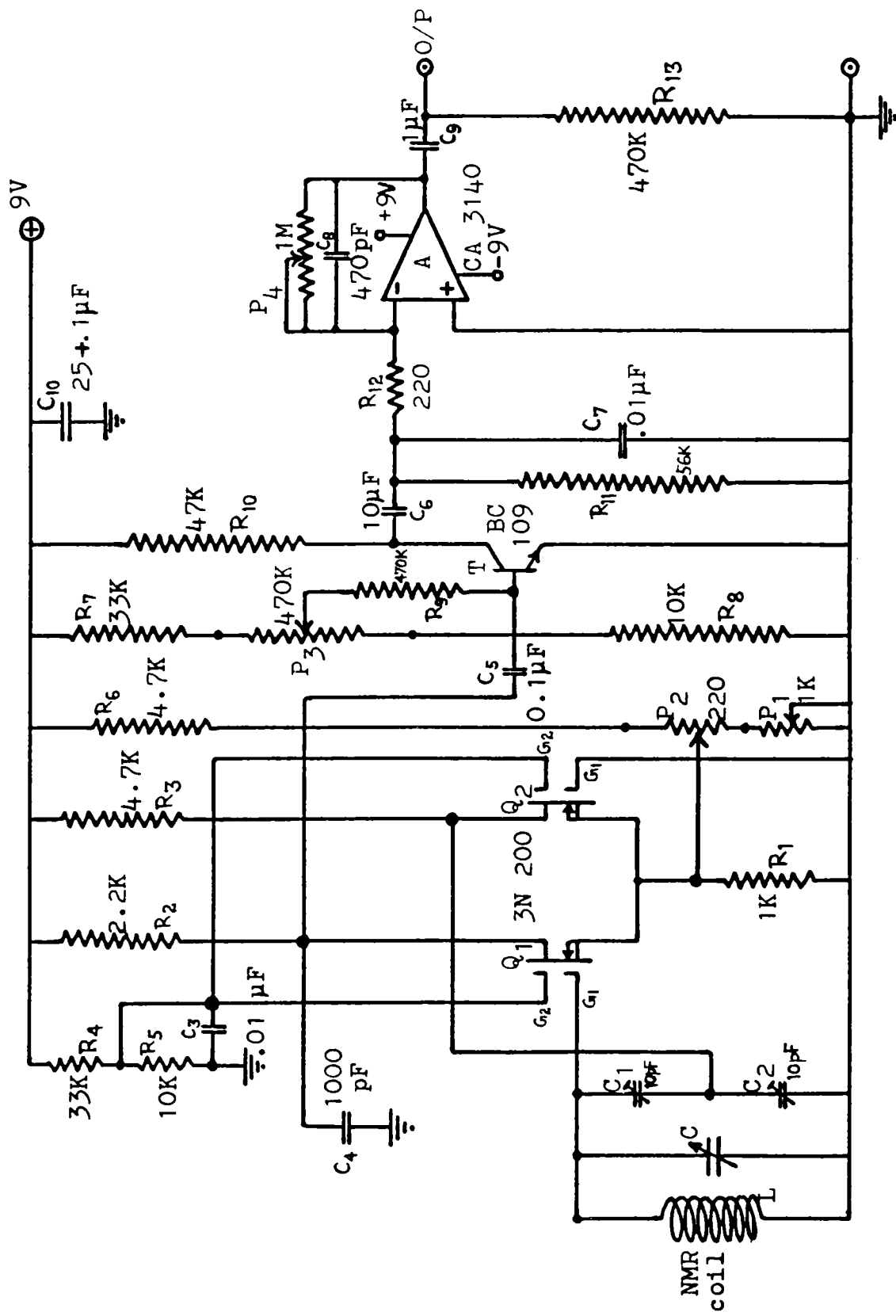


Fig. 6.4 : Schematic circuit of the Improved MOSFET based Robinson oscillator

with commonly available components the base-emitter junction of a silicon transistor (BC 109), properly biased to act as an efficient detector, was utilised for the present circuit. This arrangement can perform well at levels much lower than what the diode rectifier can respond to. Besides, at the collector of the transistor, a stage of a.f. amplification is also obtained as a bonus.

The S/N ratio of the final output signal is considerably affected by the noise contributed from the a.f. stage. In order to minimise the noise generated in the a.f. stage we have employed a single low-noise op-amp (CA 3140) for the final a.f. amplification, instead of using several transistor amplifier stages as in some earlier circuits [15, 17]. The op-amp gain can be conveniently adjusted, either for boosting weak NMR signals or for preventing saturation of the amplifier in the presence of strong signals - by reducing the op-amp gain suitably.

Since the d.c. power requirement for the present design is rather low, it enables standard 9V cells to be used for energizing the complete circuit. As a result the interference due to additional noise generated by power supply stabilizers, as was present in some earlier circuits (e.g.[15]), also gets totally eliminated in the present design.

As in the circuit of Fig 6.2 the trimmer capacitors  $C_1$  and  $C_2$  in the present circuit help to select a suitable sensitivity level. Both the MOSFETs are appropriately forward biased by the positive gate-2 voltage applied to them from the potential divider circuit comprising of resistors  $R_4$  and  $R_5$ . This serves to establish an optimum transconductance for these active devices. The a.c. grounding of gate-2 using the capacitor  $C_3$  helps to reduce the reverse transfer capacitance of the MOSFETs, thereby minimizing stray r.f. feedback [10]. The series combination of the preset potentiometers  $P_1$  and  $P_2$  enables convenient selection of r.f. level. The preset  $P_3$ , connected in the potential divider network constituting resistors  $R_7$  and  $R_8$ , serves to choose an appropriate detector bias level that can yield a good S/N ratio for the detected signal. The resistor  $R_9$  helps to restrict the bias current. The preset  $P_4$  is needed to vary the op-amp gain, and hence the final output signal amplitude, as desired. The +9V and -9V d.c. power for the circuit was provided from two 9V cells.

#### **6.4.2 : Operation of the circuit**

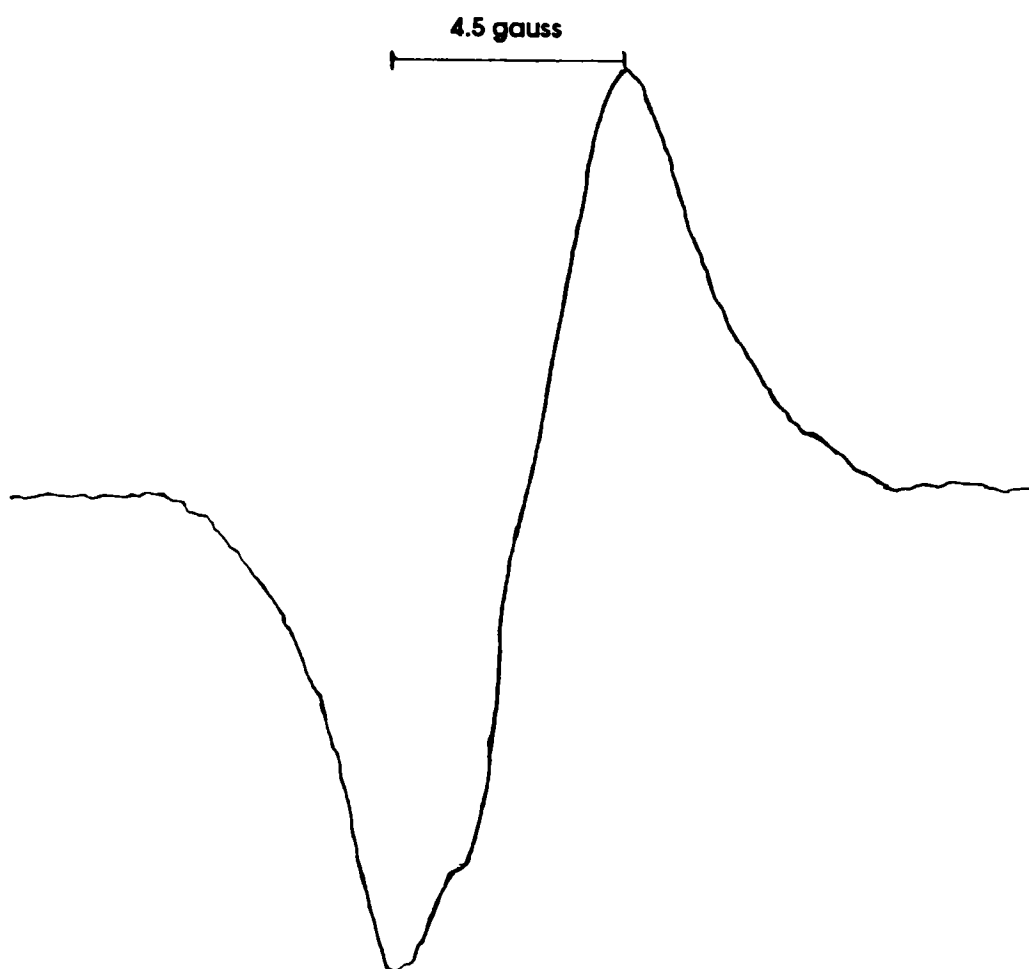
Keeping a reference sample in the NMR coil, initially the presets  $P_1$  and  $P_2$  (Fig.6.4) are adjusted to select the required r.f. level. The biasing of the detecting transistor T is then adjusted, using the preset  $P_3$  such that T is made slightly conducting. Then, viewing the absorption signal pattern on an oscilloscope,  $P_3$  as well  $P_4$  are properly adjusted to get a signal having the best S/N ratio. Under this condition the circuit will yield optimum S/N for any other signal at the particular r.f. level. For recording a signal at a different r.f. level one may reoptimize the S/N by adjusting the presets  $P_3$  and  $P_4$ .

#### **6.5 : Performance of the improved NMR detector**

The circuit shown in Fig. 6.4 has been operated at r.f. levels ranging from 150mV down to 1mV. The silicon transistor rectifier arrangement has been observed to work excellently even when the r.f. level at its base is less than 20mV. This is a real advantage compared to the diode rectifier set up in some earlier circuits (e.g.[5]). The performance of the complete circuit was tested by recording weak NMR signals, especially

from solids, at room temperature. The recording of the signals were entirely satisfactory. Fig.6.5 shows a typical tracing of the derivative curve of proton resonance in polycrystalline  $\text{NH}_4\text{Cl}$  recorded at room temperature ( $30^\circ \text{C}$ ).

It has been found that the new MOSFET based Robinson oscillator circuit gives a much improved performance in comparison with conventional NMR detector circuits, even for room temperature operation. The circuit can be fabricated using commonly available components and requires no critical elements. The simple nature of the circuit enables a compact printed circuit layout ( $\sim 4 \text{ cm} \times 6 \text{ cm}$ ) without necessitating any complex interstage shielding or any critical layout of components. The enhanced sensitivity, capability to operate at very low r.f. fields and the overall simplicity of the circuit makes it an excellent NMR detector, especially useful for probing weak signals.



**Fig. 6.5 : Tracing of the derivative curve of proton resonance from  $\text{NH}_4\text{Cl}$  (resonance frequency  $\sim 12\text{MHz}$ ; r.f.level- $20\text{mV}$ ) obtained using the MOSFET based Robinson oscillator, at room temperature ( $\sim 30^\circ\text{C}$ )**

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Photograph of the Wideline NMR spectrometer fabricated, showing the electromagnet (A), high current stabilizer-cum-field sweep unit (B), NMR detector (C), modulating signal generator (D), lock-in amplifier (E), chart recorder (F), C.R.O. (G) etc.

## Chapter VII

# SOME STUDIES OF PROTON SIGNALS FROM AQUEOUS SOLUTIONS

### Abstract

*This chapter gives a brief account of some preliminary investigations carried out in liquids using the newly fabricated NMR spectrometer. Proton resonance signal recordings from different liquid specimen as well as fluorine resonance signal recording from hydro fluoric acid are described in the first half of the chapter. Study of the effect of different paramagnetic impurities on NMR linewidth, carried out mainly to standardise the spectrometer set up, is discussed in the latter half. At the end of the chapter a discussion of the results of these simple investigations is given.*

### 7.1 Introduction

The NMR spectra of liquid samples differ significantly from those of solids, and so does the information obtained from them. The liquid phase is characterised by greater freedom of molecular motion. The effect of the motion is primarily to reduce the dipolar broadening of the resonance linewidth. i.e. that width due to the direct dipole-dipole interaction between nuclear spins. Secondly, the coupling of the nuclear spins to the lattice is usually reduced, so that spin-lattice relaxation times ( $T_1$ ) are increased. However addition of paramagnetic ions to a liquid will promote relaxation and help to reduce the value of  $T_1$  so that observation of steady NMR signals become possible.

The narrowness of the NMR signals from liquids permits the oscilloscope display of such signals to be achieved very conveniently. This allows standardisation of the NMR spectrometer utilising a standard liquid sample. Further, prior to the recording of the broad NMR signal of a given nucleus from a solid sample, it is convenient and advisable to observe the resonance signal of the particular nucleus from a liquid sample. This will enable one to set the d.c. magnetic field at a value corresponding approximately to that of the centre of the broad resonance signal, whereby the recording of wide-line signals will become easier.

### 7.2 : $H^1$ resonance signals from different liquids

In the present work the first NMR signals obtained were that of  $H^1$  in water doped with  $Fe(NO_3)_3$  salt. For observing the resonance a fixed radiofrequency of about 12MHz was chosen. The r.f.level was chosen to be around 60mV. For obtaining the oscilloscope display the sinusoidal modulating frequency was chosen to be 33Hz and the modulating field strength to be about 1gauss. With the complete spectrometer set up switched on the d.c.magnetic field is varied slowly till the resonance signal appears on the oscilloscope. The corresponding d.c.magnetic field strength was measured to be around 2,800gauss.

For recording the signal the modulating field strength is reduced to about 50milligauss and the modulating frequency is raised to 330Hz. The output of the NMR

detector is connected to the lock-in input. The reference input to the lock-in is derived from the modulating signal generator output. The output of the lock-in is connected to the chart recorder. The field sweep amplitude of the Field Sweep Unit was chosen to be 5 gauss while the sweep speed is selected to be one minute. The chart speed of the recorder was selected to be 10cm/min. Since sweeps generated by the electronic field sweep unit is positive the magnetic field is so adjusted to be slightly less than the value at which the resonance pattern appears on the oscilloscope. With the spectrometer set up switched on the chart drive of recorder is turned on. Then the field sweep is switched on. At the resonance condition the derivative of the absorption curve gets recorded. A tracing of the resonance signal is shown in Fig.7.1.

From the derivative tracing the linewidth in magnetic field unit,  $\Delta H$ , is calculated as the interval between the points of maximum and minimum slope. For the particular tracing of Fig. 7.1,  $\Delta H$  is found to be 0.2 gauss. It has been learnt that for getting a correct measure of the linewidth three conditions have to be satisfied. First of all the radiofrequency field must be small enough to prevent saturation. Secondly, the resonance line must be traversed sufficiently slowly to avoid transient effects which modify the lineshape. Thirdly, the amplifier must have an adequate bandwidth to pass the nuclear magnetic resonance signal without distortion.

Once the linewidth in frequency units,  $\Delta \nu$ , is known the spin-spin interaction time  $T_2$  can be calculated from the relation

$$\Delta \nu = 1/(\sqrt{3}\pi T_2) \text{----- (7.1)}$$

$$\therefore T_2 = 1/(\sqrt{3}\pi \Delta \nu)$$

Since  $\Delta \nu = \gamma \cdot \Delta H / 2\pi$ ,

$$T_2 = 2/(\sqrt{3} \cdot \gamma \cdot \Delta H) \text{Secs ----- (7.2)}$$

The effective proton spin-spin interaction time in doped water, deduced from Fig. 7.1 is found to be  $\sim 2 \times 10^{-4}$  Sec.

Proton resonance signals have been recorded from various other liquid samples like tap water, distilled water, turpentine, acetone, coconut oil etc. The linewidth of these signals are tabulated in Table VII-1.

<b>Table VII-1 :</b> <b>Linewidths of proton resonance signals from different samples</b>	
<b>Sample</b>	<b>Linewidth of H<sup>1</sup> signal (gauss)</b>
Tap Water	0.15
Distilled Water	0.07
Turpentine	0.1
Acetone	0.09
Coconut oil	0.13



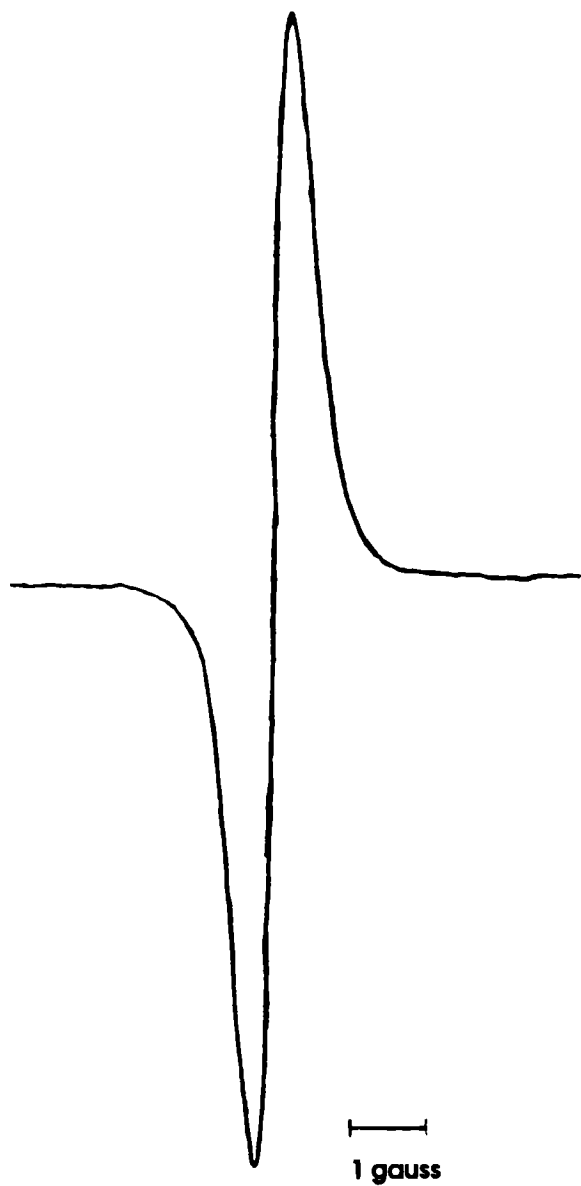


Fig. 7.1 : Tracing of the derivative curve of proton resonance from dilute  $\text{Fe}(\text{NO})_3$  solution (resonance frequency-12MHz; r.f.level-60mV)

### 7.3 : $F^{19}$ resonance

For proving multi-nuclear capability of the spectrometer set up, resonance from dilute hydrofluoric acid has been recorded. 40% hydrofluoric acid was taken in a plastic tube and kept in the sample coil. A radio frequency of about 12MHz and an r.f. level of about 50mV were selected. When the magnetic field is varied upto a value of around 2.8Kgauss the proton resonance signal from the hydrofluoric acid appears on the oscilloscope. When the field is further increased to a value approximately equal to 3Kgauss the  $F^{19}$  resonance signal appears on the oscilloscope. The tracings of the derivative recordings corresponding to these two signals are shown in Fig.7.2 for comparison purpose. The gyromagnetic ratios for  $H^1$  and  $F^{19}$  were calculated from the values of the radiofrequencies and resonant field strengths and were found to be in close agreement with the theoretical values.

### 7.4 : Study of paramagnetic impurity on NMR linewidth

The resonance linewidth in most liquid samples are very small and are in practice determined by the inhomogeneity of the magnetic field over the specimen. For satisfactory observation of resonance line it becomes necessary to broaden the resonance line artificially. Bloch, Hansen and Packard [1] have observed that the addition of paramagnetic ions to water substantially reduces the proton relaxation time, which in turn serves to broaden the resonance linewidth. The effect was further examined by Bloembergen, Purcell and Pound [2], who attributed the relaxation to the diffusional Brownian motion of water molecules in the vicinity of the ion.

For the sake of calibrating our NMR spectrometer set up the study of the effect of  $Fe^{3+}$  ion concentration against spin-spin interaction time ( $T_2$ ) has been repeated. Solutions of  $Fe(NO_3)_3$  in water were prepared for various known concentrations and the linewidth of the proton resonance signal was calculated in each case. From the linewidth the  $T_2$  value was deduced. It has been observed that even a weak ionic concentration markedly lower the  $T_2$  value. This is learnt to be due to the fact that magnetic moment of a paramagnetic ion which is of the order of one Bohr magneton, is about  $10^3$  times larger than a nuclear magnetic moment. Hence the fluctuating local magnetic field is correspondingly larger and the relaxation time shorter. The experimental graph obtained is shown in Fig.7.3 and is in close agreement with that of earlier workers [3]. The graph indicates that the relaxation time of protons in ionic solution is inversely proportional to the ionic concentration.

Study has also been carried out using ions having totally different magnetic moment compared with that of  $Fe^{3+}$ . Ion concentration versus measurements were carried out using  $Cu^{2+}$  and  $Gd^{3+}$  ions in an identical manner as discussed above. The resultant graphs are shown in Fig. 7.4, along with the graph corresponding to  $Fe^{3+}$  ion for comparison purpose. The graphs indicates that for a given concentration the relaxation time of protons in ionic solution is greater for that solution containing paramagnetic impurities having the lowest magnetic moment (e.g.  $Cu^{2+}$ ) and vice versa (e.g.  $Gd^{3+}$ ). The results are found to be in agreement with the prediction of Bloembergen et al [2] that the relaxation time of protons in ionic solutions is inversely proportional to the square of the magnetic moment.



Fig. 7.2 :  $H^1$  and  $F^{19}$  resonances from Hydro Fluoric acid

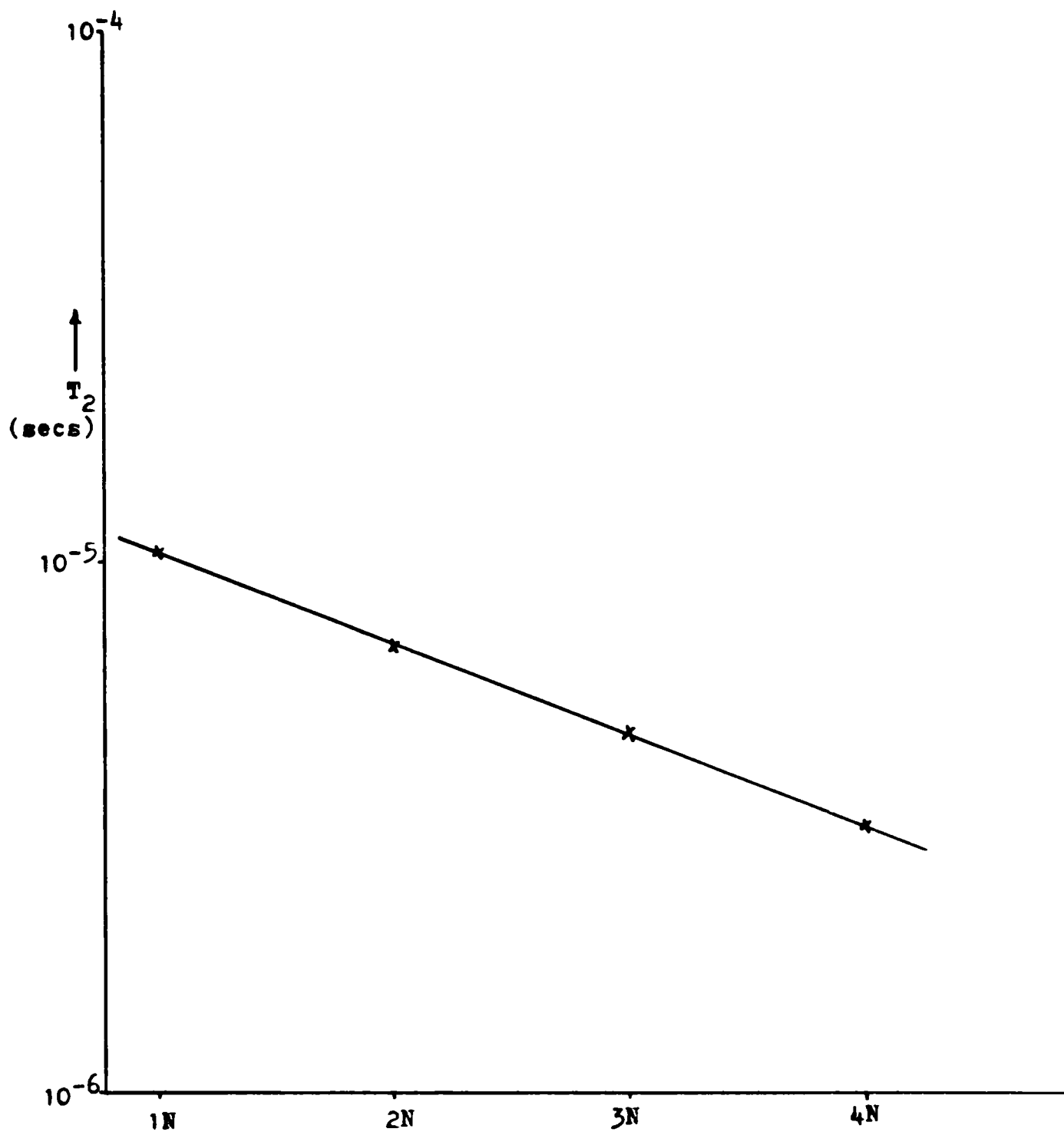


Fig. 7.3 : Graph showing the effect of  $\text{Fe}(\text{NO}_3)_3$  concentration on  $T_2$  value of proton signal

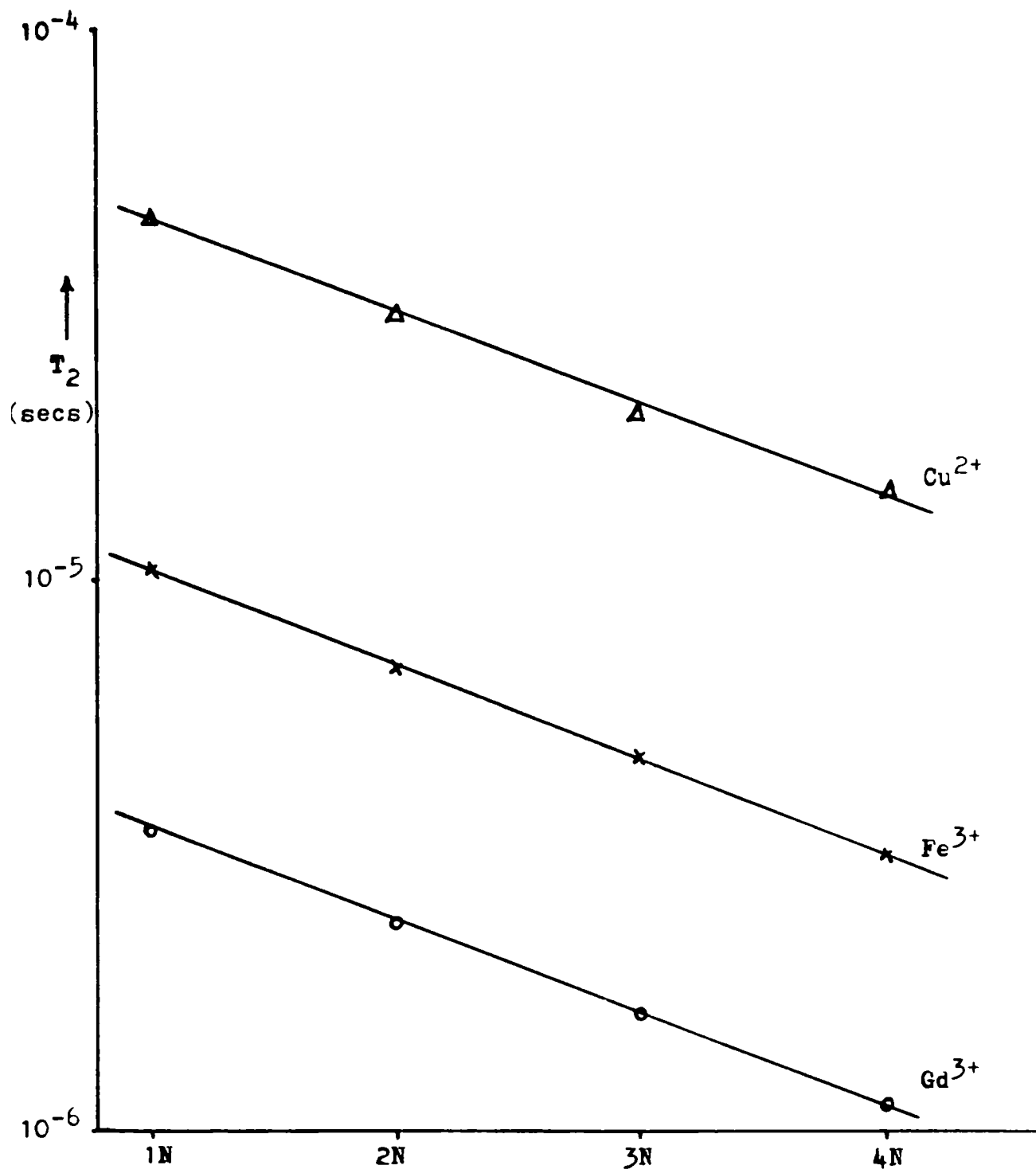


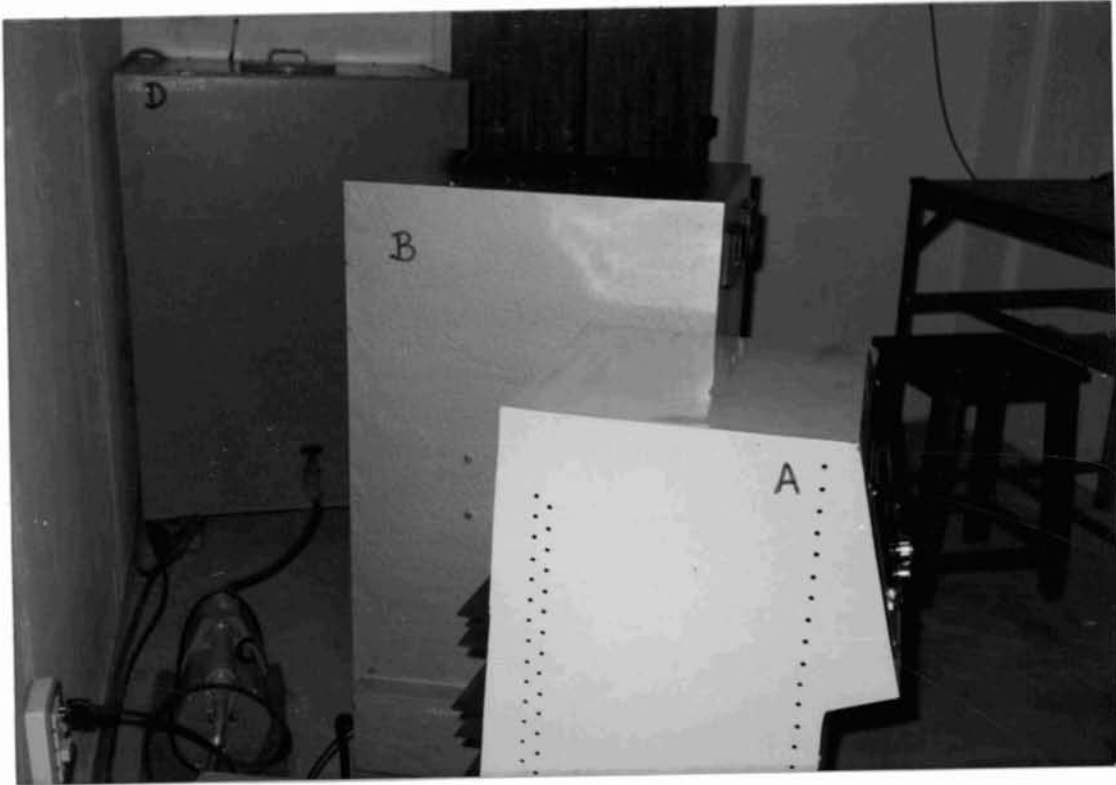
Fig. 7.4 : Graph depicting comparative studies of effect of concentration of  $\text{Fe}^{3+}$ ,  $\text{Gd}^{3+}$  and  $\text{Cu}^{2+}$  ions on  $T_2$  value of proton signal

## 7.5 : Discussion

The indigenously fabricated NMR spectrometer set up has been found to be working satisfactorily. Very stable proton resonance signals could be observed on an oscilloscope using the set up. The derivative recordings of absorption signals were fully satisfactory and dependable. The versatility of the NMR spectrometer has been established by recording  $F^{19}$  resonance as well. The results of the studies carried out for standardising the set up for performing investigation in liquids were found to follow closely those obtained by earlier workers.

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Photograph showing current stabilizer-cum-field sweep unit (A), Rectifier unit (B), monoblock pump (C) for water cooling of the electromagnet and water tank (D)