CHAPTER III

EXPERIMENTAL EQUIPMENT AND PROCEDURE

3.1 The Apparatus.

The essential features of every nuclear magnetic resonance detector are

(1) the permanent magnet or electromagnet, to provide the steady external applied field H_o ;

(2) the radio frequency oscillator unit, a source of electromagnetic radiation of the appropriate frequency ν ;

(3) provision for examination of the resonance, through variability of either H_o or ν ;

(4) a detector which responds, in some measurable way, to the absorption of energy by the nuclear moments.

A block diagram of a particularly simple and useful N M R detector is shown in Fig. 3-1, and the real one is shown in Fig. 3-2.

The magnetic field H_o was supplied by an electromagnet. The small coil was immersed in the liquid sample. The coil formed the inductive part of a resonant circuit; this gave rise to a rapidly oscillating field, perpendicular to H_o , of magnitude $2H_1 \sin 2\pi\nu t$ in the sample. In the r.f. oscillating detector the frequency ν was swept slowly over a range including the n.m.r. frequency ν_o , by means of a hand driven tuning condenser. The resonating circuit consisted of the sample coil and tuning condenser in parallel. At the instance of resonant condition, the resonant peak was observed on the cathode ray oscilloscope, and the resonant frequency was measured by a frequency meter.

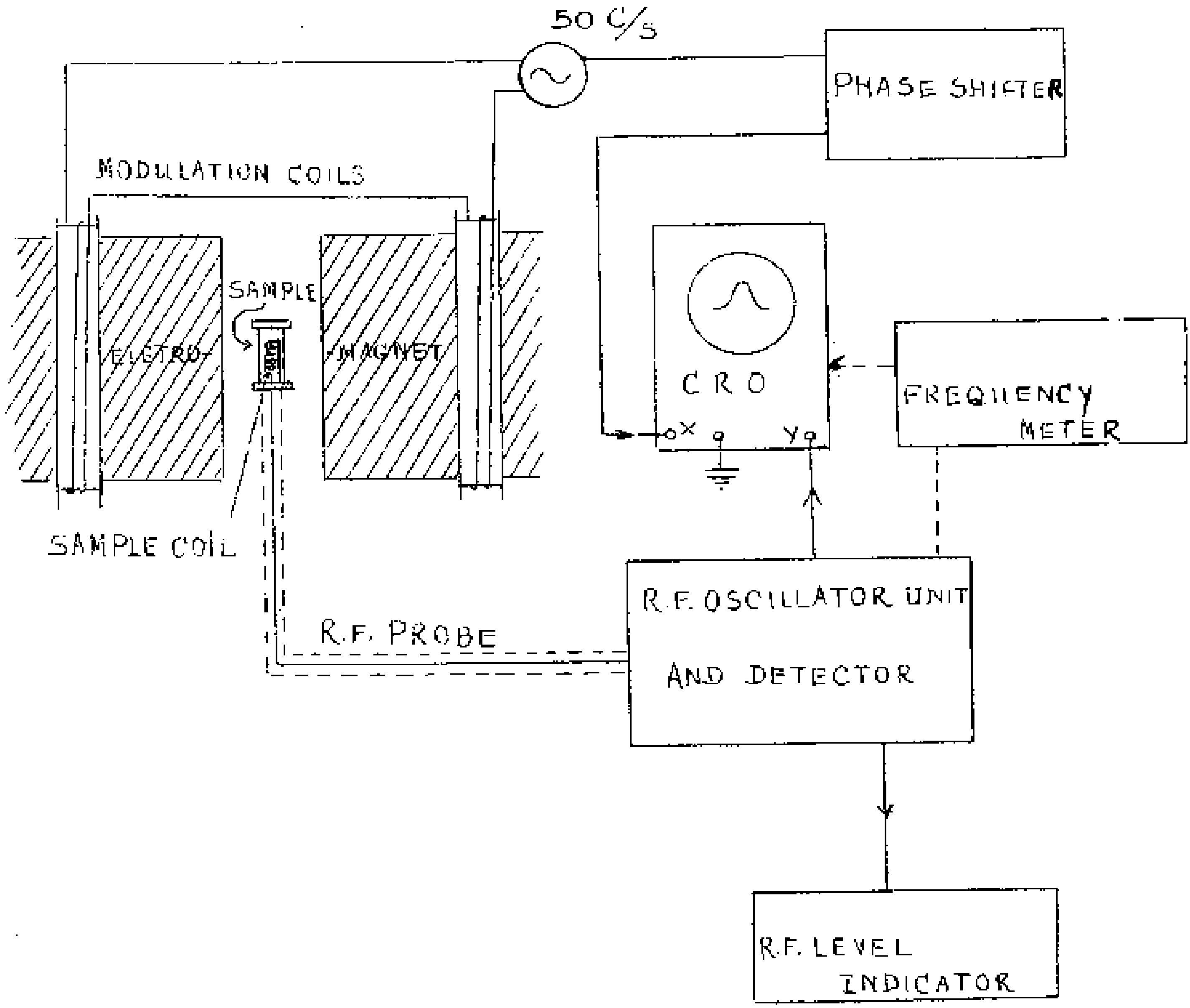


Fig.3.1. Block Diagram of the Apparatus.

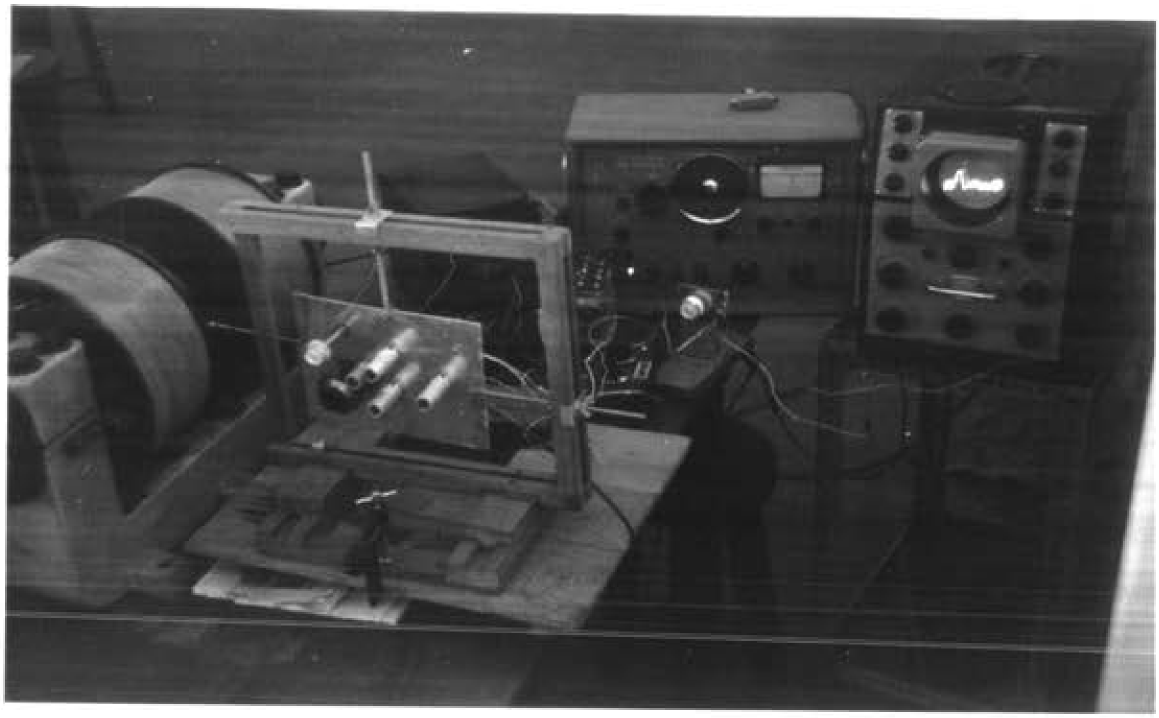


Fig. 3.2. The Apparatus .

The Resonant Peak .

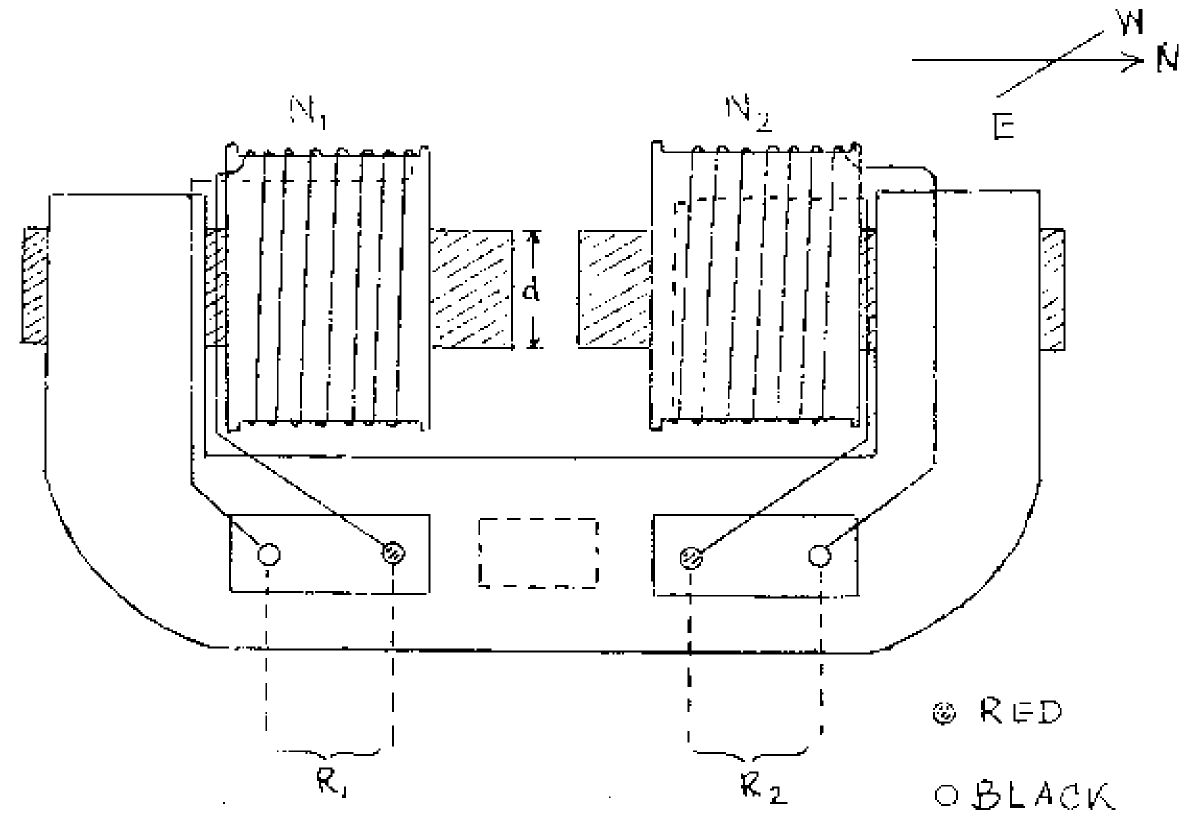
3.2 The Electromagnet.

For this experiment a Tickford electromagnet, serial number EP/182, in the Physics Department, was used. The electromagnet has a 2.5 cm. gap (for this experiment) and 10 cm. pole face diameter. The current for the electromagnet was supplied by 72 volts storage batteries. The total resistance of the coils in series is 38 ohms and total number of turns of the copper coils is 7499. The pole gap of the electromagnet is adjustable by means of screwing the cores with respect to the yoke. The diagram of this electromagnet is shown in Fig. 3.3.

3.3 The Modulation Coils and the Oscillating Field.

A pair of Helmholz coils of 11.2 cm. diameter serves as a magnetic field modulation. Each coils are wound on cylindrical wooden axis with the circular card board frames. They were put on the poles of the electromagnet with a distance of 5.6 cm. apart as shown in Fig.3-1. The total d.c. series resistance, R_{Md}, is 17.5 ohms. The oscillating magnetic field produced at the sample, in the mid-point of central plane of both coils, H_A, is 4.54 gauss peak to peak per 100 mA. rms. sinusoidal current with 50 c/s across the series coils. The diagram of the modulation coils is shown in Fig. 3-4.

If was found that the horizontal width of the C.R.O. trace was just the modulating amplitude in gauss. The method of determining the amplitude was to read the frequency change when the resonance peak was moved from



N_1 =3739 Turns, R_1 = 19 Ohms.

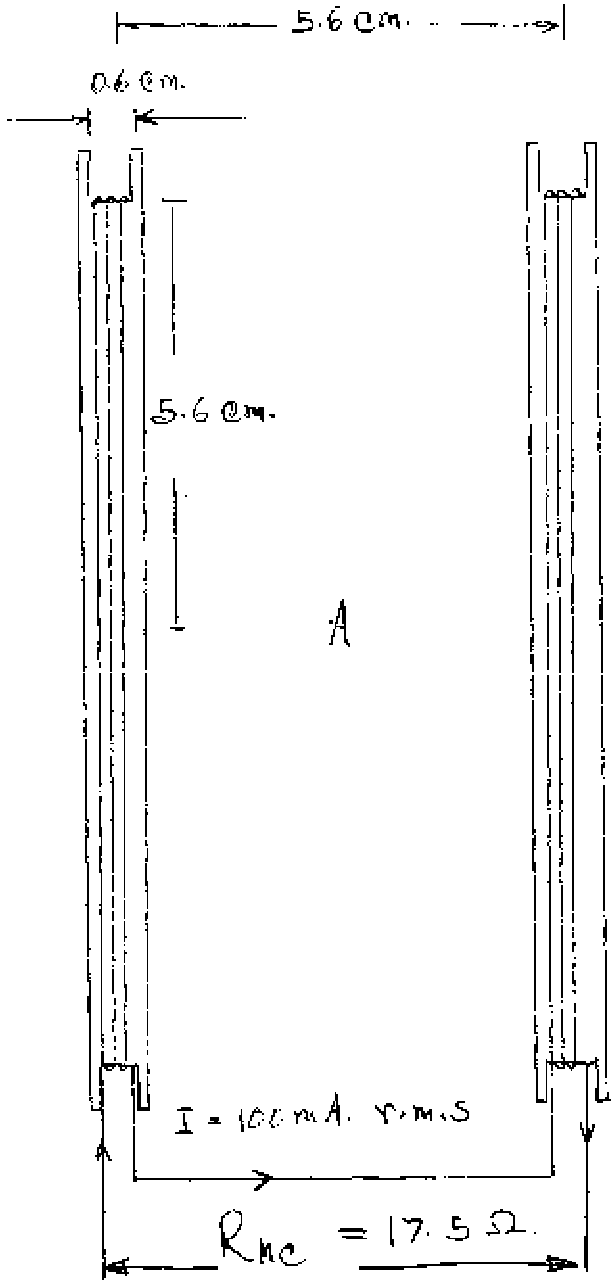
N_2 =3760 Turns, R_2 = 19 Ohms.

Pole Face Diameter, d = 10 cm.

Tickford Ltd. Electronic-Division, Newport Pagnell Eng.

Serial No. NP/182.

Fig.3.3. The Tickford Electromagnet.



EACH COIL HAS 100TURNS

#SWG30 P.O.P. COPPER WIRE.

$$H_A = \frac{3.2\pi NI}{5\sqrt{5}\,a} = \frac{3.2\pi(100\text{turns})(100\text{mA.rms.})}{5\sqrt{5}\,.(5.6\text{cm.})} = 1.604 \text{ Gauss .rms.}$$

$$= 2.268 \text{ Gauss.max.}$$

$$= 4.54 \text{ Gauss. peak to peak.}$$

Fig. 3.4. The Modulation Coils .

one side of the screen to the other. The modulating amplitude was also checked by this method, and it was found that it was about 5.58 gauss, whereas by calculating from its geometrical figure it was about 4.54 gauss. The error depended on the accuracy of the a.c. milli-ammeter.

3.4 Radio Frequency Oscillator Unit and R F Coils.

The radio frequency (r.f.) oscillator which was used for the experiment was the modified Hopkin's autodyne circuit.[12,13] It was constructed by Mr. Bhiyayo Panyarjun.* Its circuit is shown in Fig. 3 - 5. The main part of this circuit consists of one 6AK5 tube, one IN60 semiconductor and three EF91(6AM6) tubes. The 6AK5 tube acts as the oscillator. The IN60 semiconductor serves as a rectifier, the first two EF91 tubes serve as a d.c. amplifier, and the last EF91 tube is the a.f. amplifier. From Fig. 3- 5, the 6AK5 tube behaves as a cathode-follower at r.f. with an inductive cathode load. This causes a negative resistance to appear across the tuned circuit due to the capacitance between grid and cathode. The negative resistance allows the tuned circuit to oscillate with controllable amplitude. Some amplitude stabilization occurs because of the grid rectification. A d.c. voltage proportional to the r.f. amplitude derived from the cathode of the oscillator, is amplified and fed back to the screen via a cathode-follower to maintain the gain of the d.c. amplifier. This circuit gives a voltage variation across the cathode from 0.05 to 0.055 v. over the tuning range. The audio section of the r.f. unit had a high a.f. gain and its output is further amplified with the

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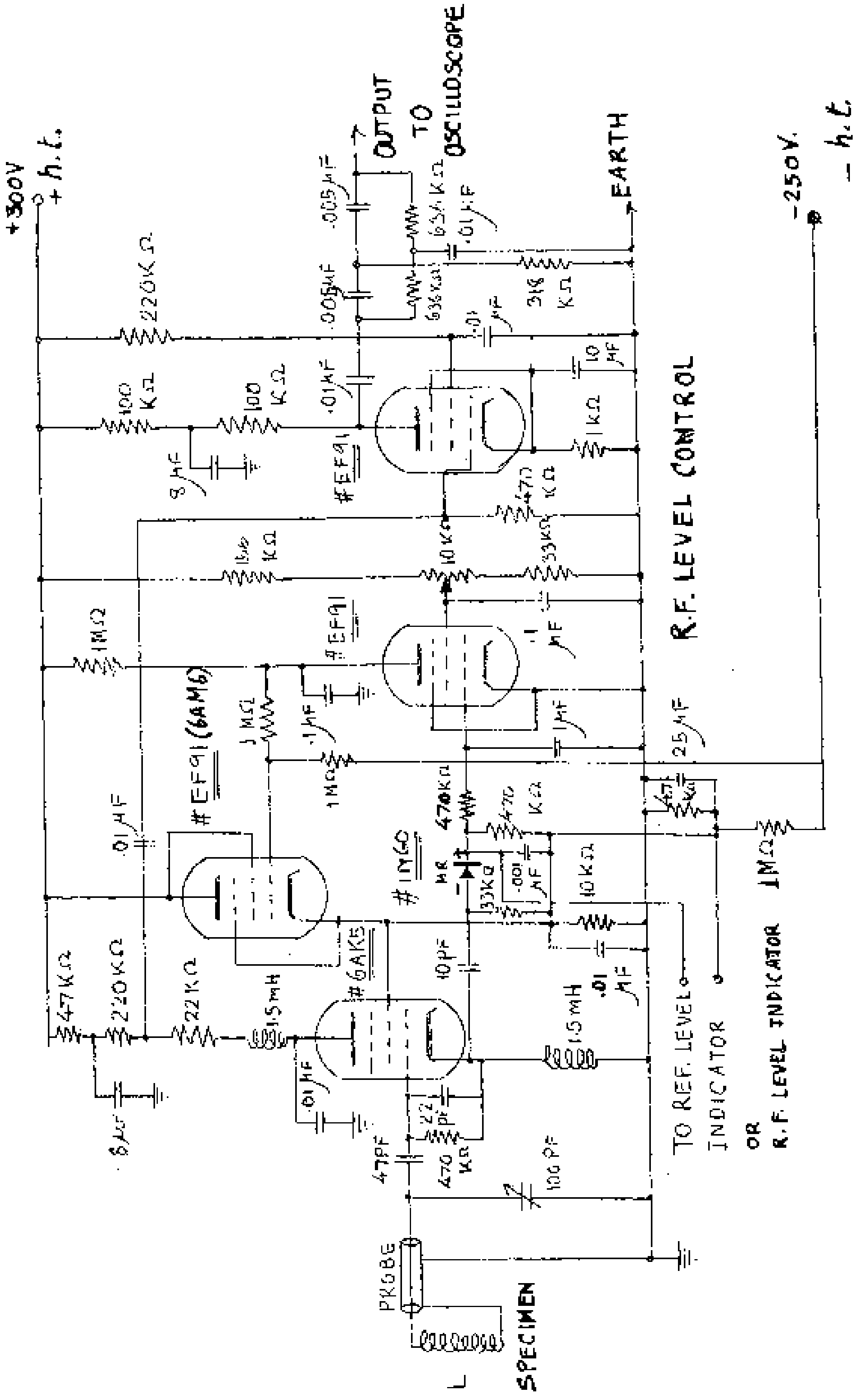


Fig.3.5. **R.F.** Oscillator Unit .

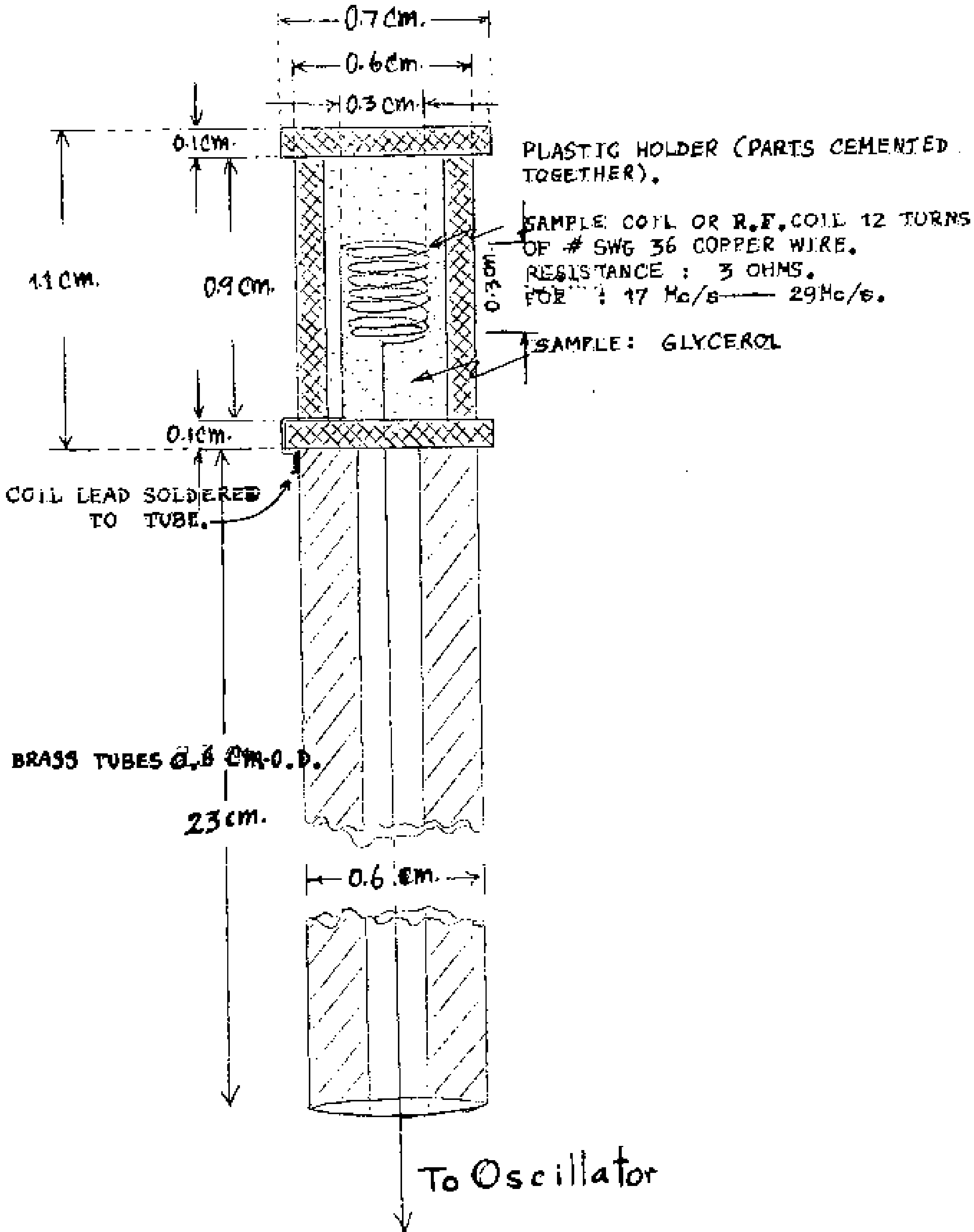


Fig.3.6. Diagram of the Probe.

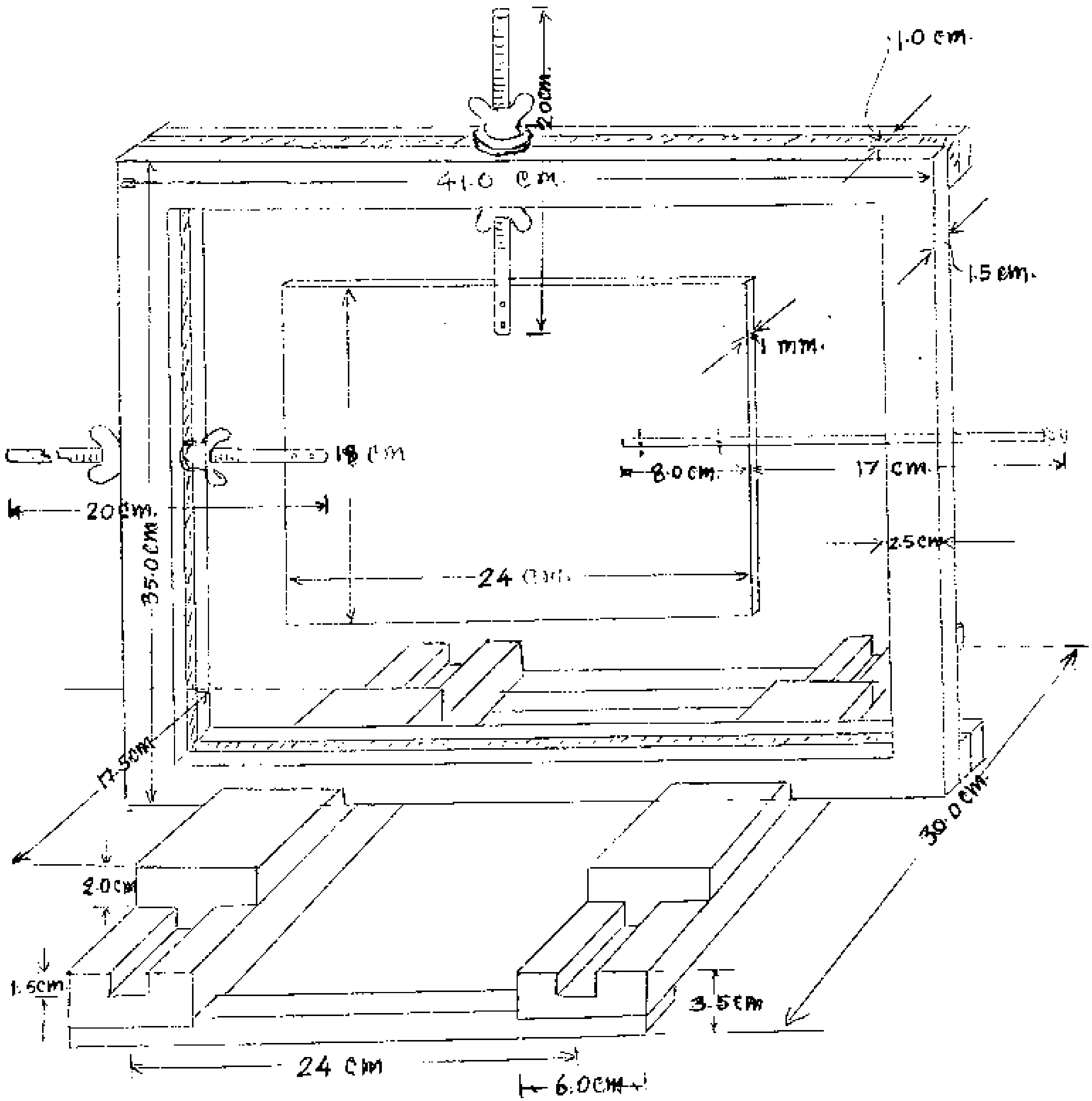


Fig.3.7. The 3-Dimensional-Movable Frame of the R.F.Oscillator Unit.

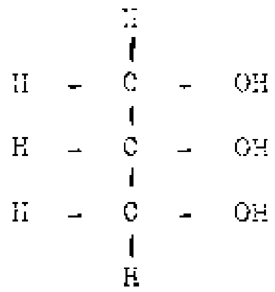
50 c/s components filtered out before being applied to the Y-plates of an oscilloscope.

Fig. 3.6 shows the details of the r.f. probe used. The specimen size was kept as small as possible since the variation of field strength across it due to field inhomogeneity leads to absorption over a corresponding wide range of frequencies and a reduction in sensitivity. The use of glycerol with its low dielectric loss permitts complete immersion of the r.f. coil or sample coil in the specimen. Thus the filling factor is optimized. The r.f. coil is made of 12 turns of # SWG36 copper wire. The coil size is about 0.3 cm x 0.3 cm. or 0.02 c.c. volume. It is immersed in glycerol which is contained in the cylindrical plastic capsule, and is also mounted on the top of the 25 cm. long brass tube as shown in Fig. 3.6.

The r.f. oscillator unit is mounted on the aluminium plate chasis with the size of 16 cm. x 24 cm. The chasis is mounted on the 3-dimensional movable frame as shown in Fig.3.7. The frame consists of the non-magnetic materials. Thus the r.f. oscillator is suitably placed for locating the magnetic field strengths at various positions.

3.5 Chemistry of the Sample.

The sample used was Glycerol (Glycerin); $C_3H_5(OH)_3$, is a trihydroxy propane (or 1,2,3 propanotriol) with the structure



Its physical properties are :-

melting point	=	17.9° C,
boiling point	=	290° C,
density	=	1.26 gm/ml,
molecular weight	=	92.09 ,
dielectric constant	=	43 at 3×10^{5} c/s, 20° C.

The choice of specimen was governed by several factors. These are[12]

(a) range of field strengths to be measured,

(b) relative magnitude of absorption,

(c) dielectric loss.

It was found that water contains a large number of hydrogen atoms per unit volume but has a high dielectric loss. Glycerol has a slightly lower density of hydrogen atoms, but its very low loss results in greatly increased sensitivity.

It was also found by N. Bloembergen[11] that the relaxation time in Glycerol is 10^{2} times smaller than that in water at any temperature. The relaxation times of protons in Glycerol and in water are 0.02 and 2.3 seconds respectively at 20° C, 29 Mc/s. Thus Glycerol was selected as the specimen.

3.6 Frequency Measurement.

The tuning capacitor was tuned until oscillations were just maintained. This point was easily determined by the behavior of the C.R.O. trace when it was reached. A standard variable-frequency source, FM/AM Signal Generator; TF 995 A/1; Marconi Instrument Ltd, acted as a frequency meter by losely coupled with the sample coil. The frequency of the Signal Generator was varied to obtain a zero-beat note on the oscilloscope screen, thus indicating that the two frequencies, the resonant frequency and the standard source frequency, were the same. The value of frequencies could be read from the dial of the standard source. In this way the resonant frequency and hence the magnetic field strength was determined.

3.7 Experimental Procedure.

The apparatus was arranged as shown in Fig. 3.1.Various parts can be listed as follow.

(1) The Tickford electromagnet was supplied by 72 volts storage batteries.

(2) The modulation coils were supplied by 100 mA. rms. 50 c/s.

(3) The r.f. oscillator unit which was constructed by Mr. B. Panyarjun had been set to give 0.03 to 0.05 v. at the reference level (Fig. 3.5) . The r.f. level was indicated by the Galvanometer; type SR 445; No 105780; H. Tinsley & Co.Ltd. London.

(4) The frequency meter used was the FM/AM Signal Generator; TF 995 A/1, No 1940038. Marconi Instruments Ltd. London.

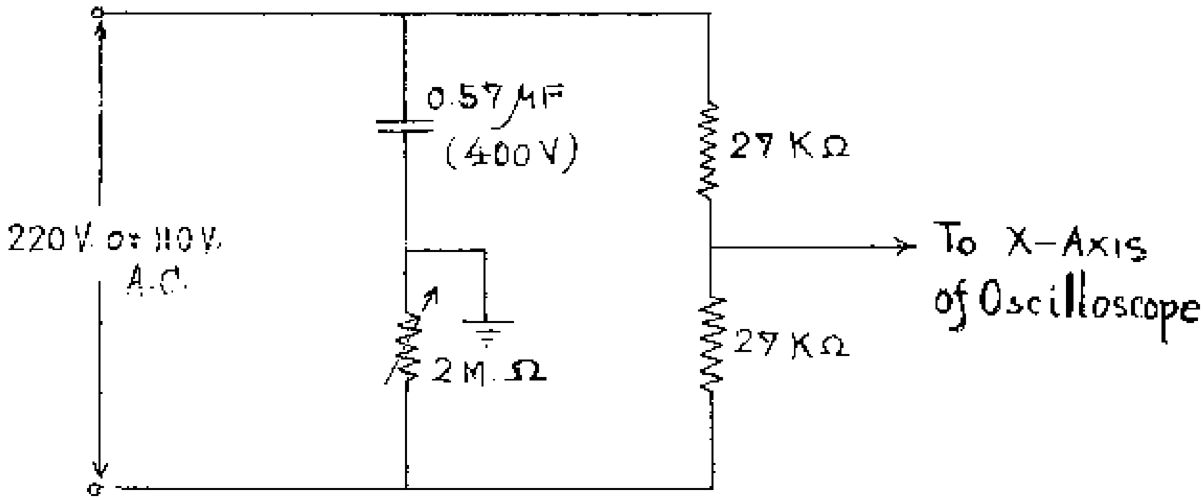


Fig. 3.8. Phase Shifter Circuit .

(5) The Cathode Ray Oscilloscope was the Model 1049 MKIII Oscillograph, Cossor Instruments Ltd. England.

(6) The phase shifter circuit is as shown in Fig. 3.8.

To carry out a measurement the following steps were necessary.

(a) The modulation amplitude was set to about 4.5 gauss peak to peak at 50c/s

(b) The r.f. oscillator was set to give the output of 0.03 to 0.05 v. at reference level.

(c) The Cathode Ray Oscilloscope was set to sweep at 50 c/s on the horizontal axis by 220 or 110 v. a.c.

(d) The tuning capacitor was turned throughout the range to seak for the resonant peaks, then the pulses were located and set to coincidence by the phase shifter.

(e) The resonant frequency ν was measured by the zero-beating method as described in section 3.6. The magnetic field strength can be given by the equation[13]

$$H_o = (234.87 \pm 0.29) \times 10^{-6} \nu \quad \text{gauss, where } \nu$$

is in c/s.