

## CHAPTER III

### NUCLEAR MAGNETIC RESONANCE SPECTROMETER



#### 3.1 Components of NMR Spectrometer.

The spectrometer (Fig.3) is of the type first described by Pound and Knight (4) and later analyzed in detail by Watkins (7). This spectrometer consists of an rf unit and calibrator, a lock-in amplifier, an audio oscillator and phase shifter, a recording meter, a constant magnetic field, a cathode rays oscilloscope, a motor and reduction gears. This spectrometer is suitable in searching the resonant frequency for wide range frequency ranging from about 4 to 62 Mc/s, and it is easy in practice to detect the absorption line by means of the changing reactance in sample coil resulting from the absorption of nuclear resonance. The detection is to observe the phase shift in the cathode rays oscilloscope or in the recording meter. We know the resonant frequency from a communication receiver. Each part of the spectrometer will be described in detail.

#### 3.2 RF Unit and Calibrator (7,8,9).

It consists of a calibrator, an rf oscillator, an rf amplifier with avc rf level control, a detector and an audio amplifier. The circuit is shown in Fig. 4. The oscillator is a negative resistance oscillator whose tank coil (sample coil) is placed in the constant magnetic field. The tube, 6 4 6 is used as the oscillating tube because of its low noise, wide range frequencies tuning and higher stability. All parts of the oscillator are kept in a good shielding of

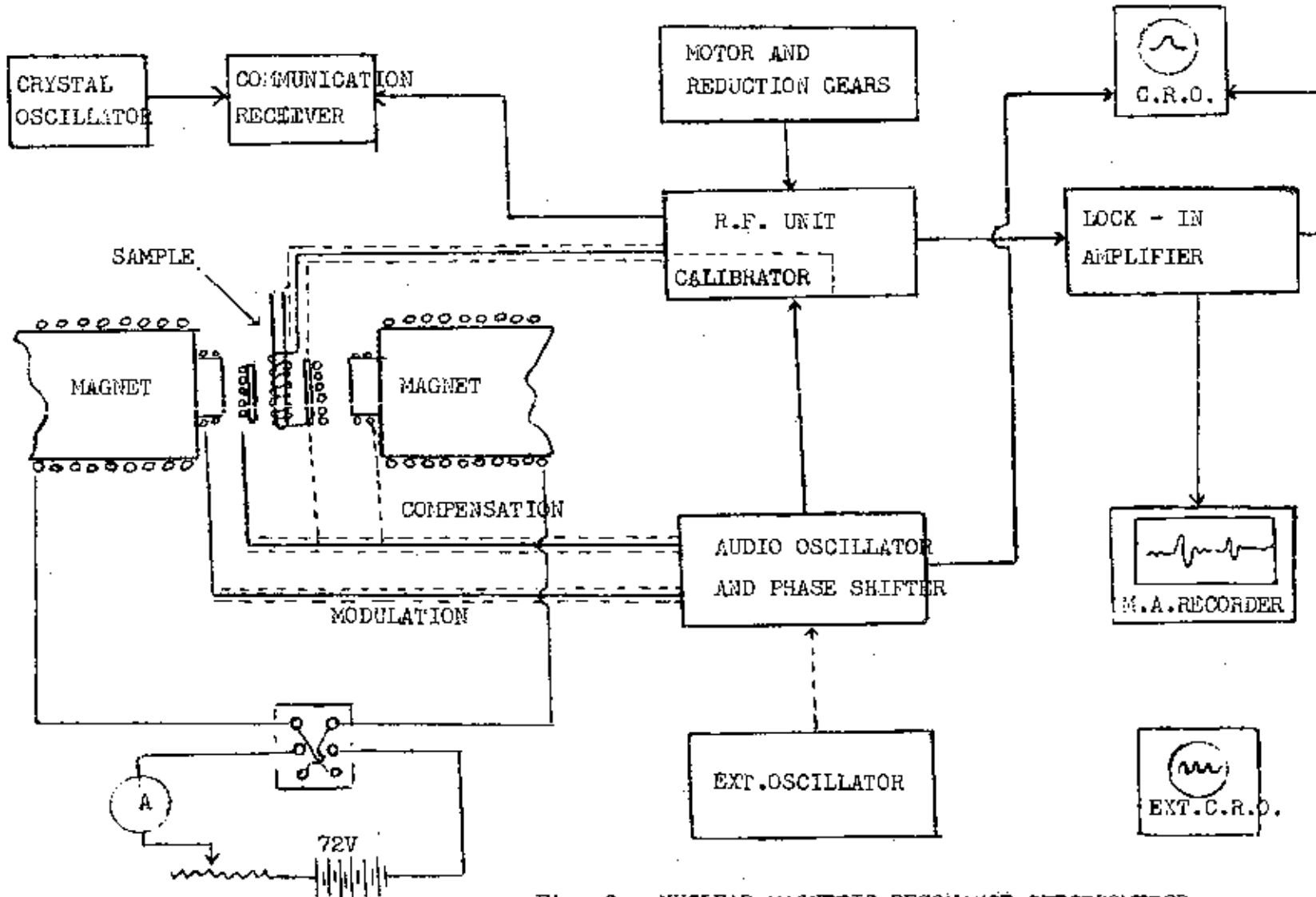
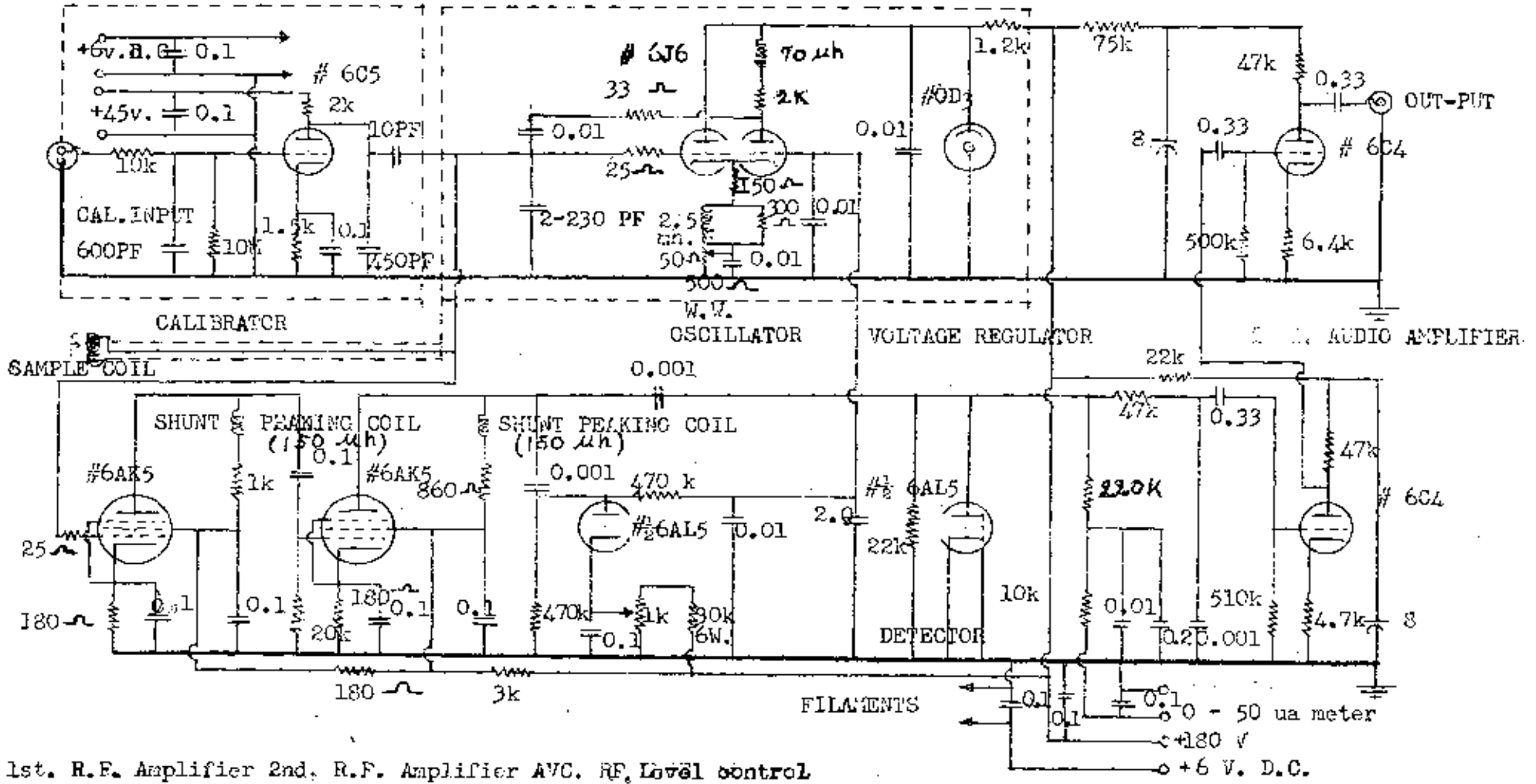


Fig. 3 NUCLEAR MAGNETIC RESONANCE SPECTROMETER .



ALL RESISTORS ARE 1/2 WATTS UNLESS OTHERWISE SPECIFIED  
 ALL CAPPACITORS ARE IN MICROFARADS UNLESS OTHERWISE SPECIFIED.

FIG. 4 R.F. UNIT AND CALIBRATOR

aluminum chassis for reducing extraneous noise of the system.

The rf amplifier has two stages of the tubes, 6AK5. The rf signal from the oscillator is amplified and is fed to a double diode, 6AL5, which is used as an avc rf level control and detector. When the signal is fed from the rf amplifier to the tube 6AL5, where it is rectified at the first half and the audio signal is detected by second half of the tube 6AL5. The dc rectified voltage from the first half of 6AL5, is fed back, through a time constant of two seconds to the oscillator to control the power level in the coil. This feed-back defines an operating point of the oscillator which is far below the natural limiting because of non-linearity of the tube (7). Variations in amplitude of the rf level at audio frequencies, however, are not removed. In the experiment the tuning condenser is tuned by a 10 rpm motor and the rotation can be reduced by reduction gears to  $\frac{1}{25}$  revolution per hour.

The rf level is controlled by adjusting the cathode bias (1 megohm potentiometer) of the first half of the tube 6AL5. The audio frequency signal of the resonance at second half of the 6AL5 is fed to two-stage audio amplifier of 6C4 tubes. The amplification signal from the output is fed to a lock-in amplifier in order to detect the resonant signal of absorption.

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### 3.3 Audio Oscillator and Phase Shifter.

The audio oscillator and phase shifter (Fig. 5) was built by Ketudat (7,8) and is a modification of Watkins'. It is an oscillator with two voltage and two current outputs with separately variable



amplitudes and phases. The oscillator provides a reference voltage for the calibrator which produces a known change in conductance of the tuned circuit of the rf oscillator and thereby give an absolute calibration of the sensitivity of the spectrometer. The current sources supply the Helmholtz coils for the magnetic field modulation and compensation coil which serve to cancel out the direct pick-up of the modulation field by the rf coil. The oscillator circuit of the phase shifter is of RC type. The changing of phase is controlled by a RC network. The audio signal from the external oscillator can be used instead of internal oscillator needed. The signal from the external oscillator can be fed in the point "external oscillator" and then switch to the position " external oscillator" to cut off the internal signal.

#### 3.4 Lock - In Amplifier.

The lock-in amplifier is a part of the spectrometer which compares the phase the resonant absorption signal and the reference signal and feeds this difference into recording meter in order to get the graph of the signal. This lock-in amplifier consists of a narrow-band amplifier, a phase sensitive detector, a time constant RC circuit and a recording meter (milliamp recorder). The circuit of the lock-in amplifier is shown in Fig. 6.

Each part of the lock-in amplifier will be described as follows:

The narrow band amplifier (7,8,9) consists of tubes, 6SN7 and 6SJ7, and twin-T filter. It employs negative feed back by means of twin-T filter in order to reduce other noise signal and let the audio signal at the modulation frequency to the phase sensitive detector.

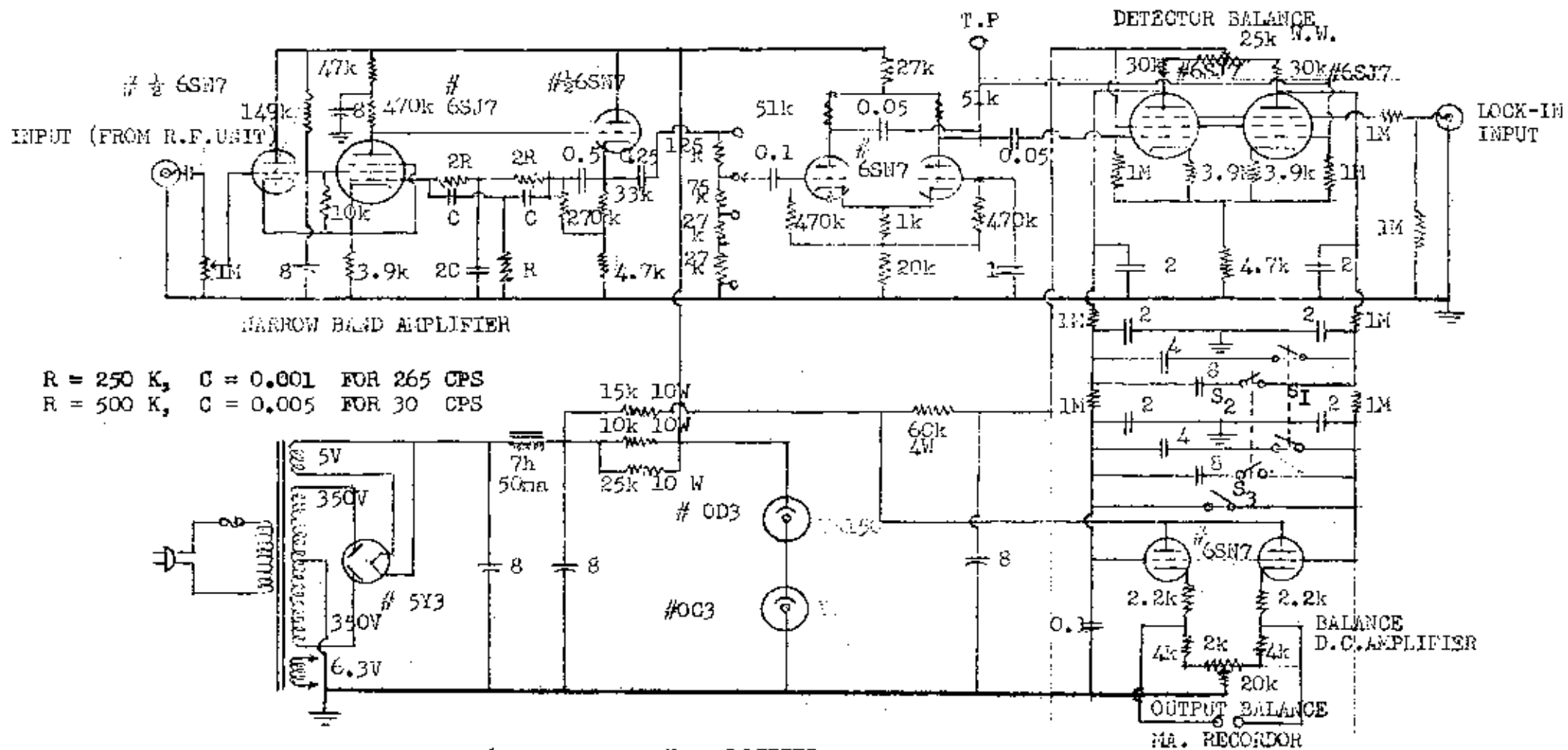


Fig. 6 LOCK - IN AMPLIFIER .

The twin-T component sizes, C should be large enough to minimize the effects of variations in stray capacitance, and R large enough to avoid overloading the cathode follower (10). When the audio signal at the modulation frequency is fed into the lock-in amplifier, it is amplified by the narrow band amplifier and converted to dc by the phase sensitive detector which consists of two tubes 6SJ7. The dc signal is then sent to the recording meter through a variable time constant which serves to control the observation width of the resonant signal.

### 3.5 Sample Coil Construction.

Sample coil is made of # 24 copper wire with 10 turns of diameter 0.2 cm.. It is inserted in the central cylindrical copper

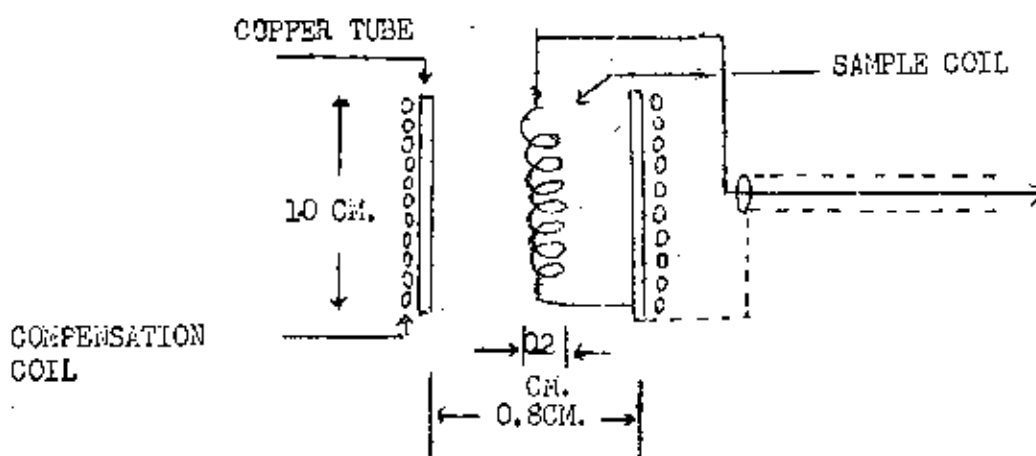


Fig. 7 SAMPLE COIL.

tube of 0.8 cm. diameter and 1.0 cm. long as shown in the Fig. 7. The terminals of the coil are connected with a coaxial cable to coupling with the tuning condenser of the rf oscillator. A compensation coil is wound around the cylindrical copper tube with # 24 copper wire with 15 turns.



### 3.6 Modulation Coil Construction.

The modulation coil is a pair of Helmholtz coils whose radii are 2.5 cm. Each of the Helmholtz coils is constructed with a #28 copper wire, wound around a plastic core of 100 turns. Each coil is fitted with a wooden frame by means of Duco cement. On the top of the

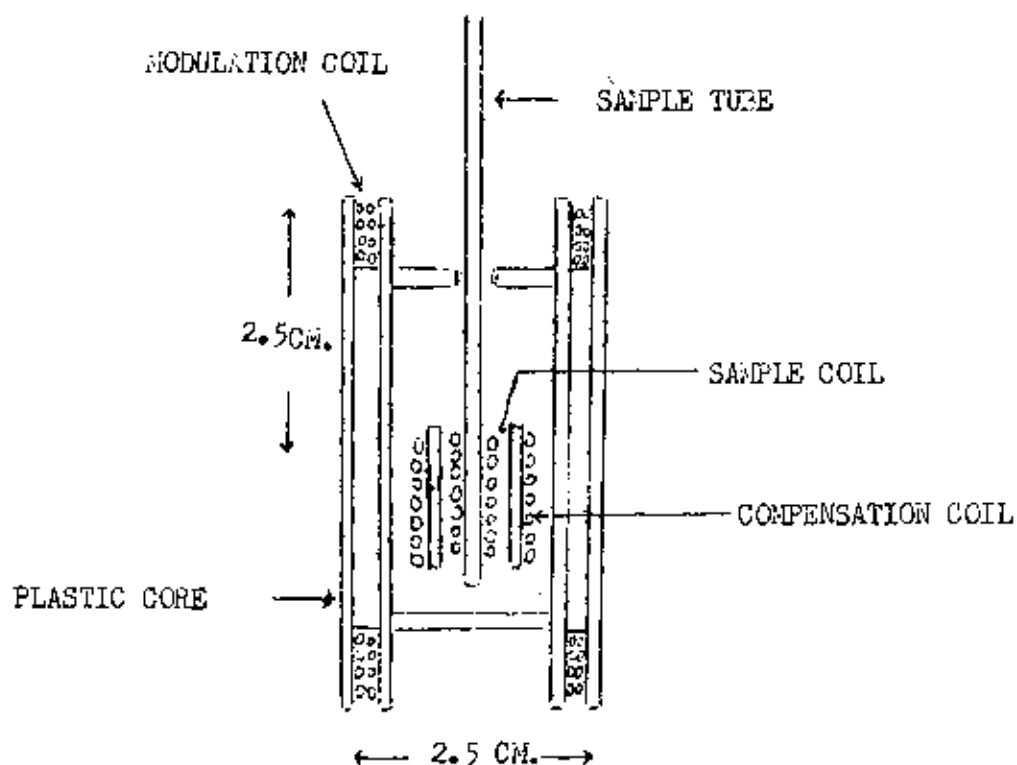


Fig. 8 MODULATION COIL.

wooden frame, is drilled a hole for putting the sample tube inside the sample coil as shown in Fig. 8. The coaxial cable connecting the

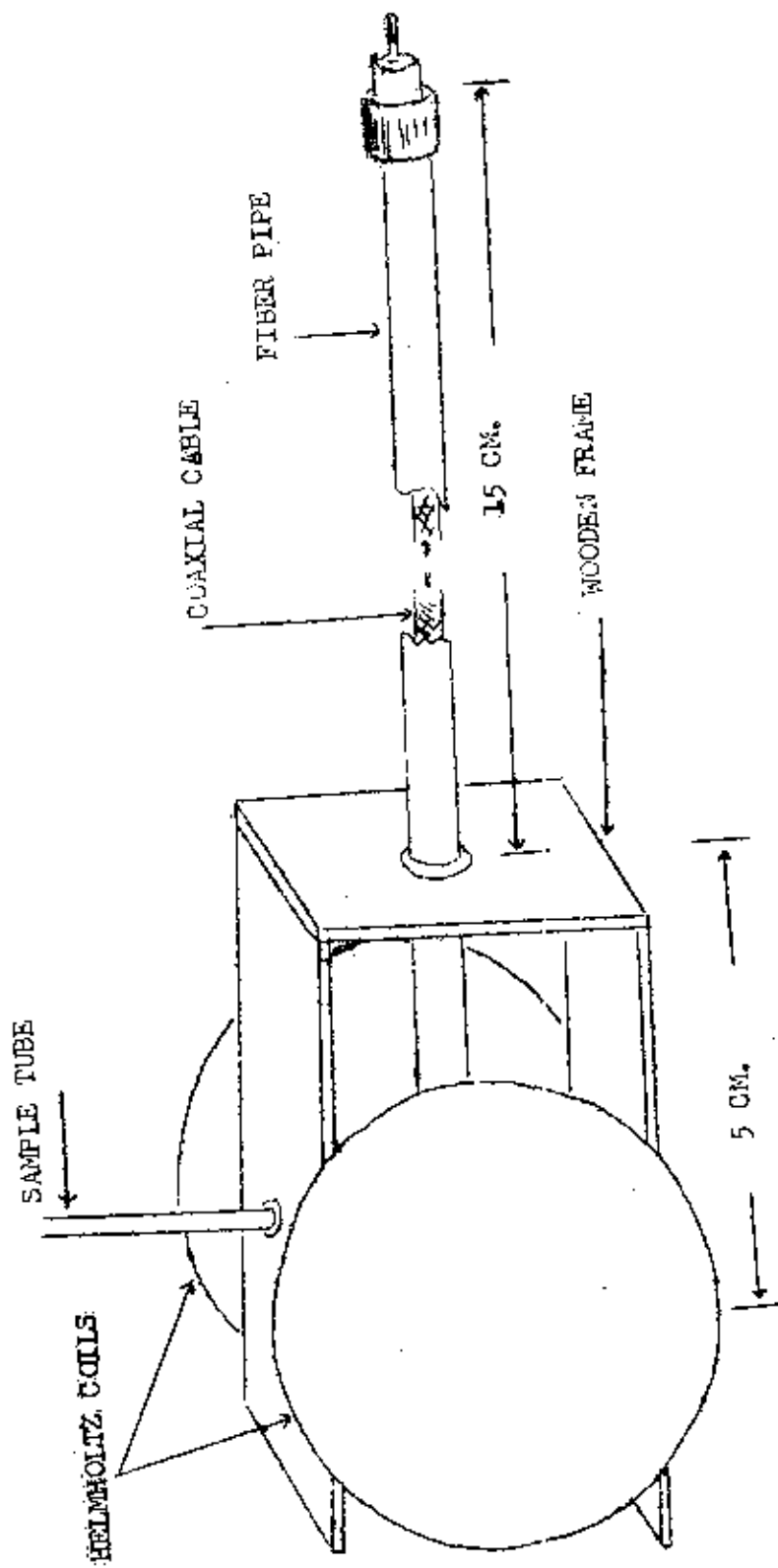
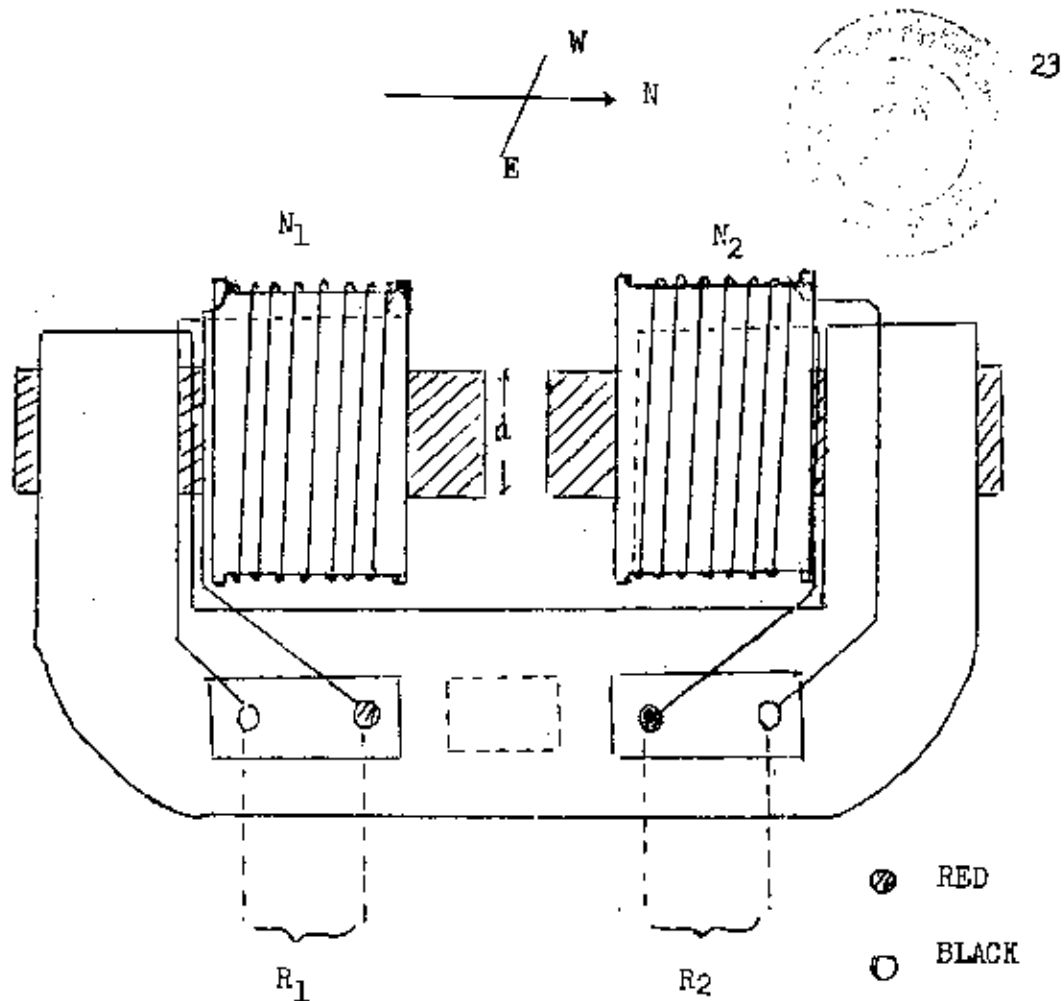


Fig. 9 R.F. PROBE.

sample coil to the rf unit is put in a rigid cylindrical fiber pipe as shown in Fig. 9.

### 3.7 Magnetic Field .

The magnetic field is supplied by an electromagnet of the Tickford Ltd., Electronic Division,, Newport Pagnell Eng., Serial No. NP/182. The pole face diameter is 10 cm., The gap between the pole faces can be adjusted. Total number of turns of the coils, in series, around the poles are 7499 turns with dc resistance of  $38 \Omega$  . The diagram is shown in the Fig. 10. The current for this magnet is supplied by storage batteries .



$N_1 = 3739$  Turns,       $R_1 = 19$  Ohms.

$N_2 = 3760$  Turns,       $R_2 = 19$  Ohms.

Face Diameter,       $d = 10$  cm.

Tickford Ltd. Electronic Division, Newport Pagnell Eng.

Serial No NP/182.

**Fig. 10** THE TICKFORD ELECTROMAGNET.