Chapter III

EXPERIMENTAL DETAILS

3.1 Apparatus for Measurement of T_1 and T_2

Electromagnet	(Bruker B.E. 25C8)
Storage Oscilloscope	(Tektronix type 549)
Oscilloscope Camera	(Tektronix type C12)
D.C. Power Supplier	(Tektronix type 160)
Wave Form Generator	(Tektronix type 162)
Pulse Generator	(Tektronix type. 161, 163)
Measuring Amplifier	(Lebold type 532 01)
Function Generator	(Philips PM 5168)
Standard Decade Resistance	(General Radio Company type 1434-X)
Galvanometer	
D.C Ammeter	
Gated RF. Transmitter[18]	
Preamplifier[19]	
RF Amplifier[20]	
Phase Sensitive Detector	
Pulse Amplifier	

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[18] R.J. Blume, "rf Gate with 10^9 Carrier Suppression", The Review of Scientific Instruments, 32(1961)554.

[19] W.G. Clark, "Pulsed Nuclear Resonance Apparatus", Ibid., 35,(1964) 316.

[20] W.. Senghaphan, "An Investigation of Spin-Lattice Relaxation Time of bcc Solid Helium-3", Ph.D.Thesis, Boston University, (1968).

D.C. Power Supplier (for heater used)

Pulse Sequence trigger (for 180° pulse sequences used)

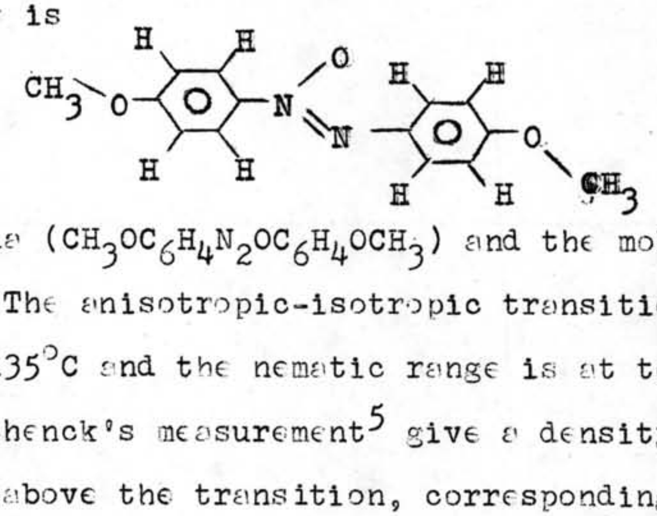
Sample Cell

All of these instruments are illustrated in Fig.3.1 and Fig.3.2 and some of them will be shown in detail description in the next section.

3.2 Sample and Construction of Sample Cell

3.2.1 Sample

The nematic liquid crystal, para-azoxyanisole (PAA) used in this experiment was purchased from the Kodak Chemical Co., Rochester, N.Y., and was patially zone refined. Its chemical structure is



of chemical formula ($CH_3OC_6H_4N_2OC_6H_4OCH_3$) and the molecular weight is 258.3. The anisotropic-isotropic transition occurs at approximately $135^\circ C$ and the nematic range is at temperature $119^\circ C$ to $135^\circ C$. Schenck's measurement[5] give a density of 1.153 g/cm^3 immediately above the transition, corresponding to a molar volume of 225 cm^3. According to Kreutzer and Kast[21], the anisotropic-isotropic heat of transition is 410 cal/mole, and the specific heat at constant volume, which is 0.488 $cal/g^\circ C$

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[21] K. Kreutzer and K. Kast, Naturwiss, 25(1937)233.

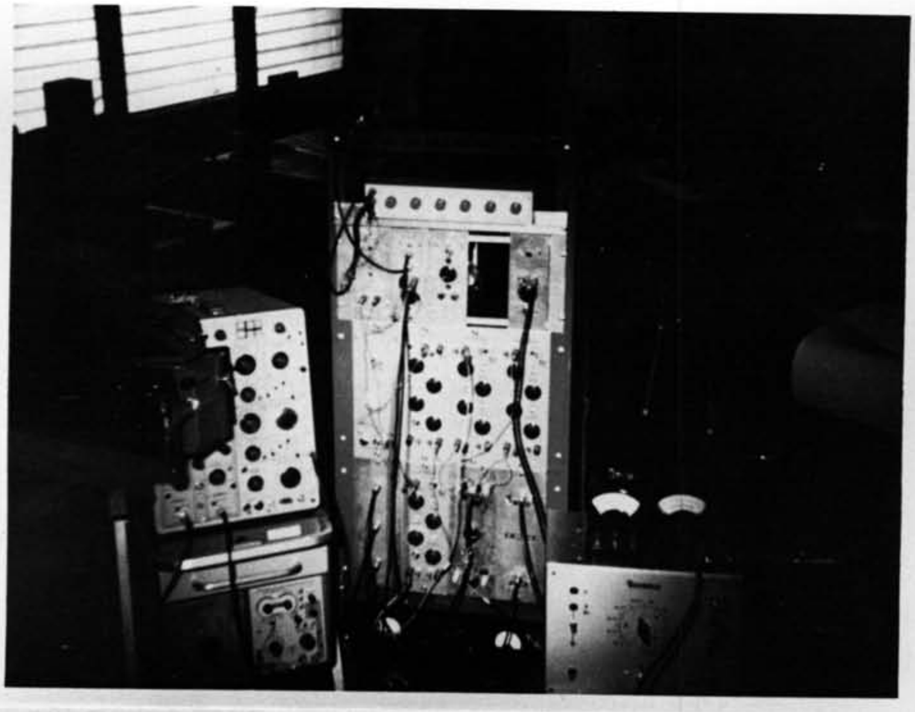


Fig.3.1 Apparatus

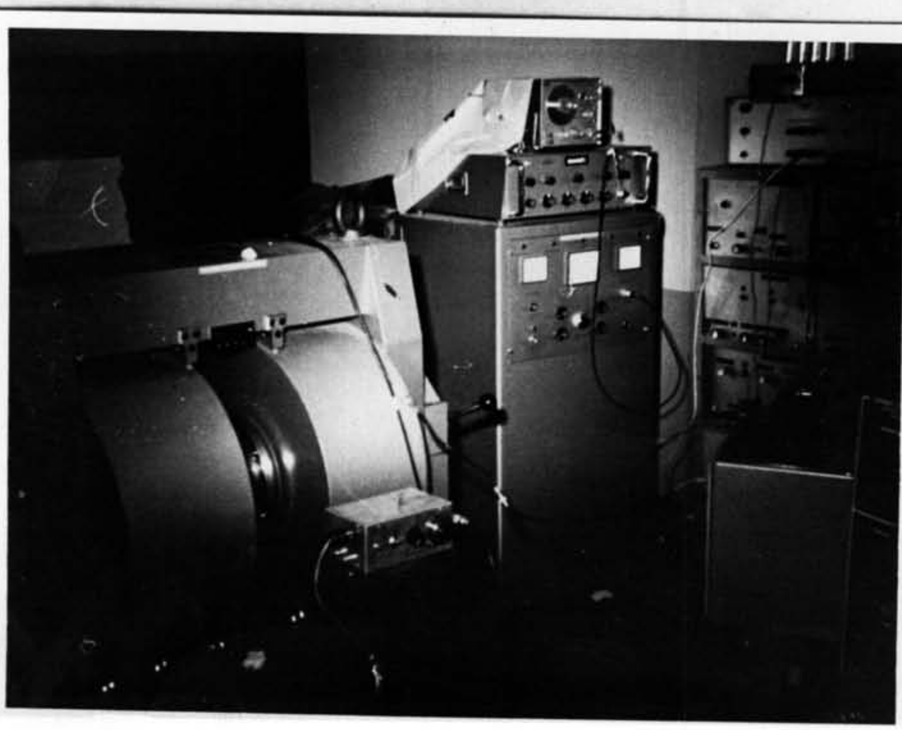


Fig. 3.2 Apparatus

anisotropic phase. The viscosity of isotropic PAA, near the transition, is 3.08 cp, while the viscosity of the anisotropic liquid may be considerably greater in the presence of suitably oriented magnetic field.

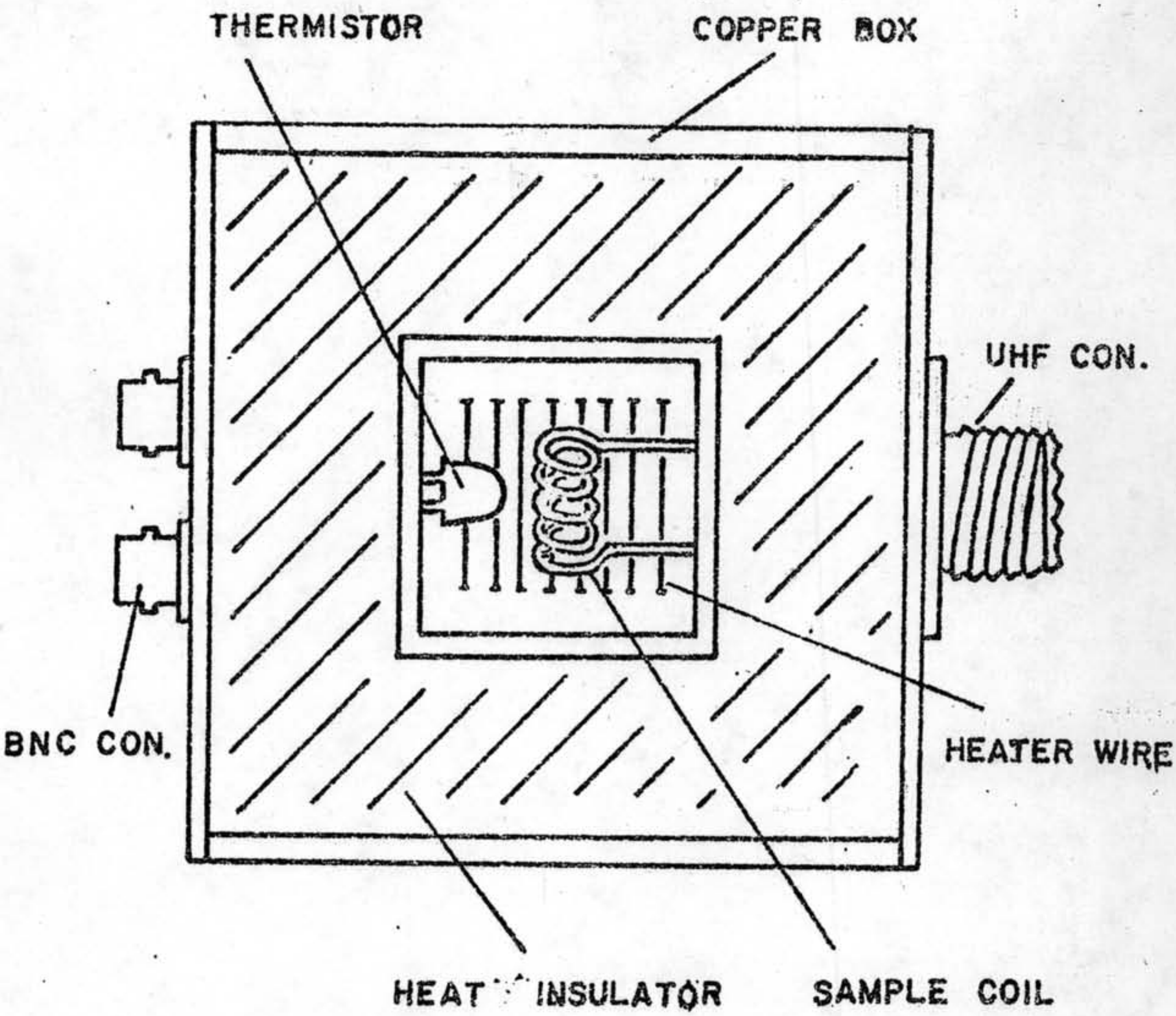
3.2.2 Construction of sample cell

The partially zone refined para azoxyanisole was sealed in a previously cleaned 6-mm-o.d. Pyrex tube. The insulator coated copper wire was wound as a small rf coil about 2 cm. in length, tuned at resonant frequency 10 MHz. and each terminals was connected to a UHF connector fastened on a 1 m.m. thick copper shielding box, dimension 3x8x8 cm. as shown in Fig.3.3. two heater-wire plates were placed between the rf coil. The heat that generated by these two heater plates is controlable by adjusting a suitable d.c. current from the d.c. power supplier. A small thermistor was attached near the sample tube for the temperature calibration. This sample chamber was also surrounded by thermal insulating material preventing the heat radiating from the system.

3.3 Experimental Procedure

3.3.1 T_1 Measurement

The apparatus in the block diagram as shown in Fig.3.4 was set up for the measurement of T_1. A static magnetic field, H_0, was generated by a Bruker B.E. 25C8 magnet. The signal pulse generaters for the rf pulses, the H_1, were Tektronix types 162, 163 and the out put was connected via a home-made pulse



SAMPLE CELL

Fig.3.3

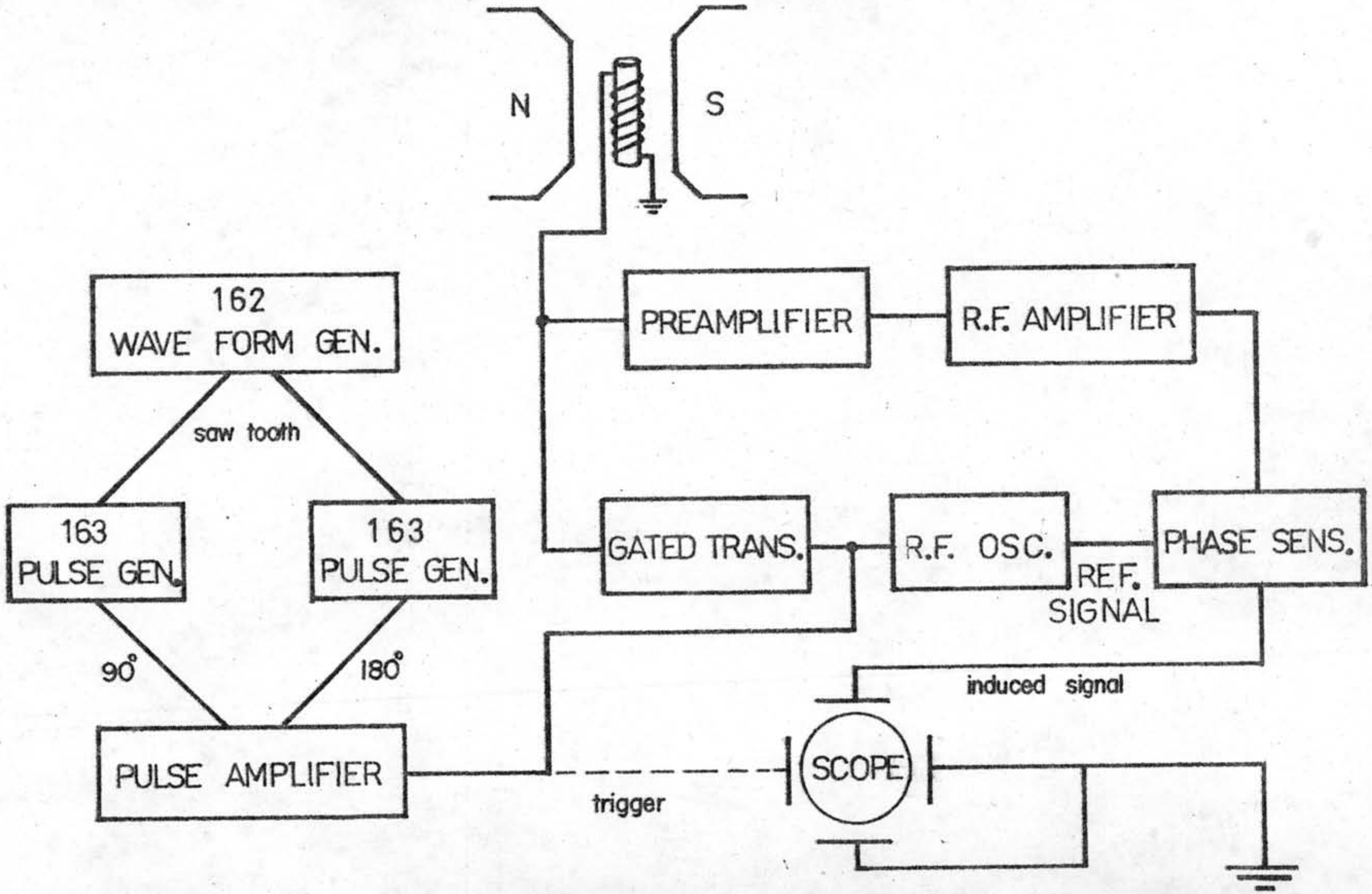


Fig. 3.4 Block diagram for measuring T_1

amplifier feed through a gated transmitter of the type described by Blume[18] (1961). The gate was operated by a pulse of amplitude +60V., with the appropriate delays to provide the desired pulse sequence. The output rf pulse was fed by a coaxial cable to a tuned rf-coil via a UHF connector on the sample cell. The nmr signal was detected on the rf coil and fed to a preamplifier, rf-amplifier and phase sensitive detector. The reference rf signal for the phase sensitive detector was taken directly from the rf crystal oscillator. The amplified induced signal was displayed on Tektronix type 549, a storage oscilloscope.

With an appropriate pulse duration t_{ω}, producing a 90° pulse, a maximum induced signal is performed on the oscilloscope as illustrate in Fig.3.5. Likewise , by adjusting a proper t_{ω} for 180° pulse, a minimum induced signal can be performed as shown in Fig.3.6

Using the measurement method as described in Section 2.5, τ_{null} can be obtained at various different temperatures from 450°K down to 380°K. The experimental data are listed in Appendix II.A.

3.3.2 T_2 Measurement

In order to measure T_2, the apparatus was also set up. as described by the block diagram shown in Fig.3.7. A pulse sequence trigger was connected via the gate trigger of the wave form generator 162, providing a sequence of 180° pulse. At the same time, the pulse sequence trigger was connected with the pulse generator 161 for providing a initial 90°pulse. The spin

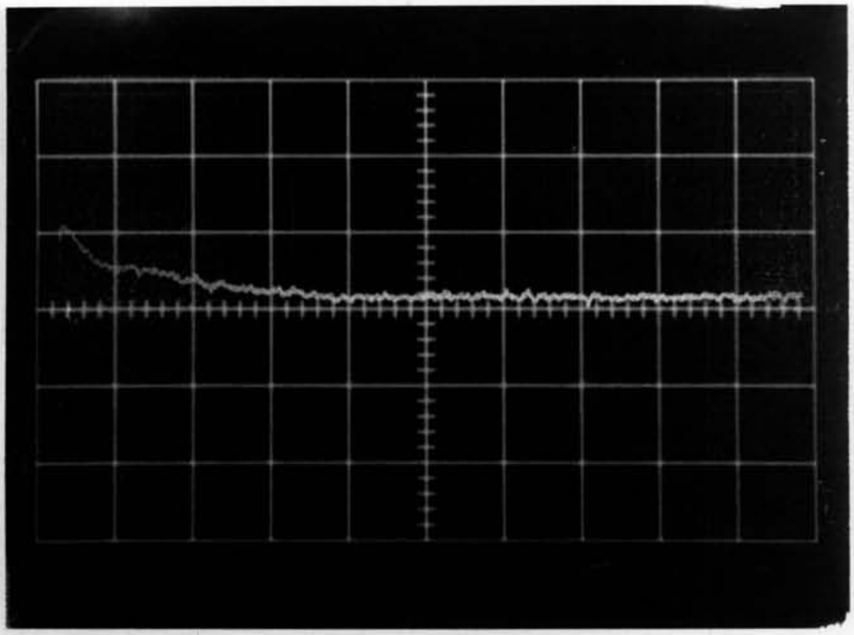


Fig.3.5 Induction tail of 90° pulse

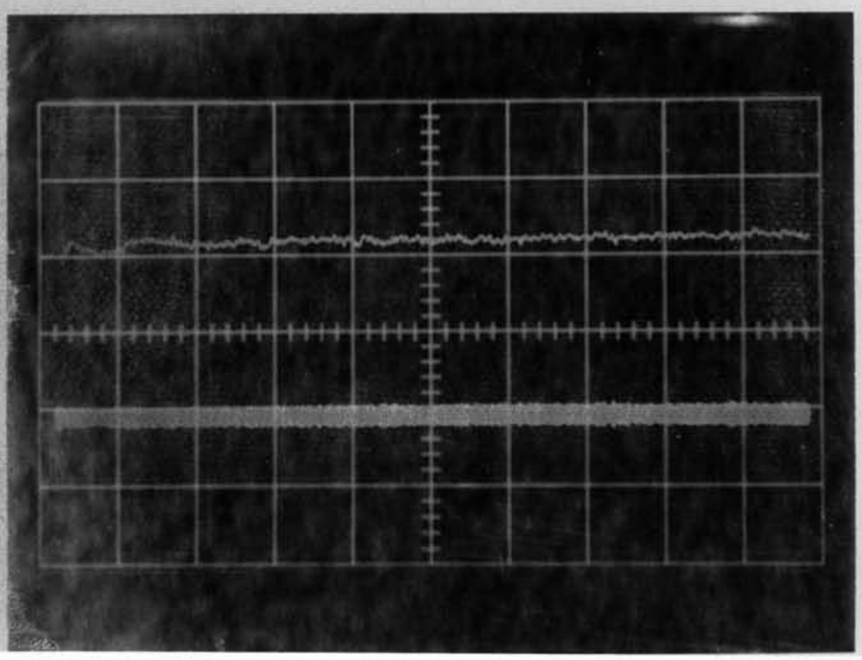


Fig.3.6 180° pulse induced signal

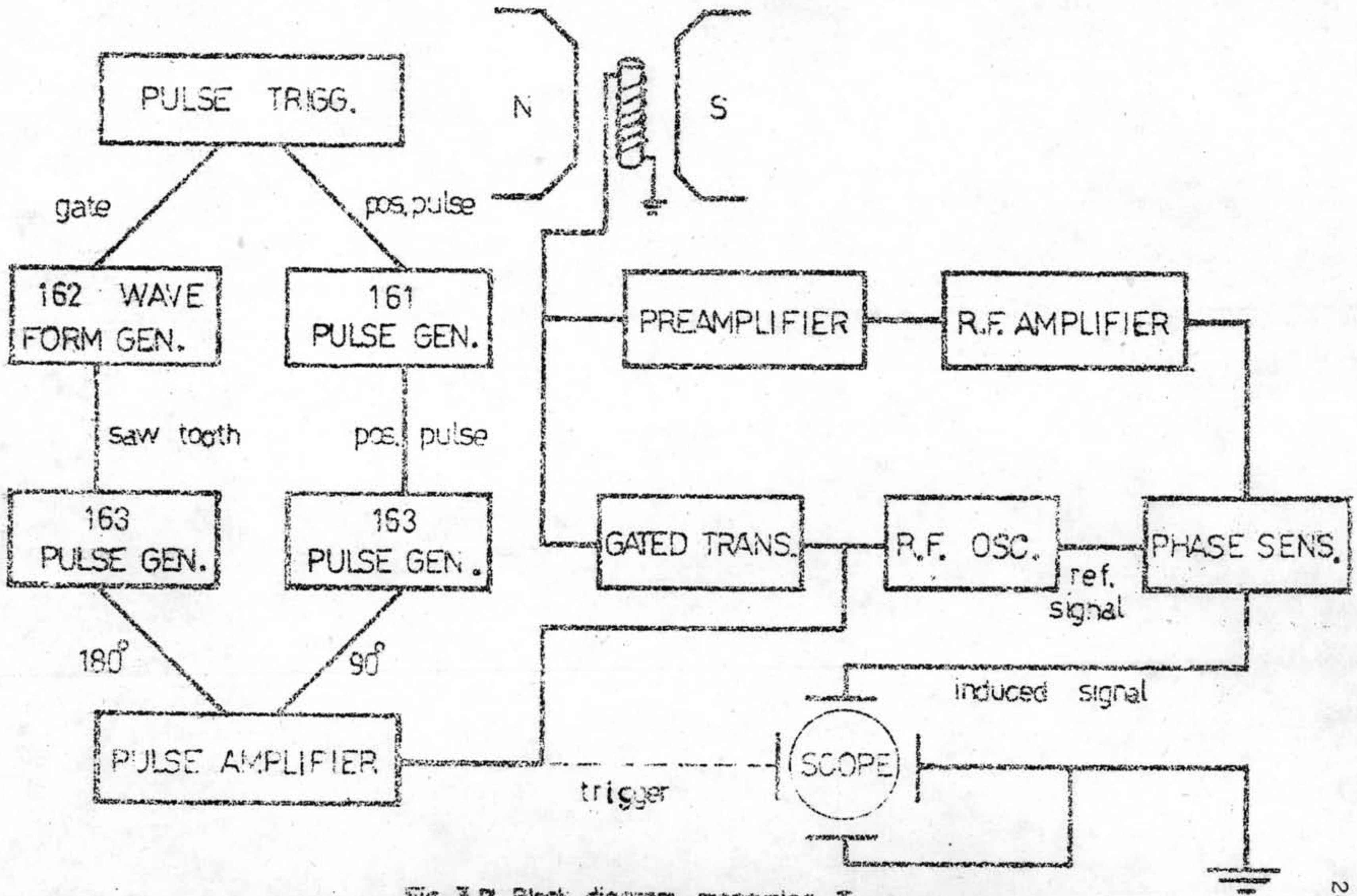


Fig. 3.7 Block diagram measuring T_2

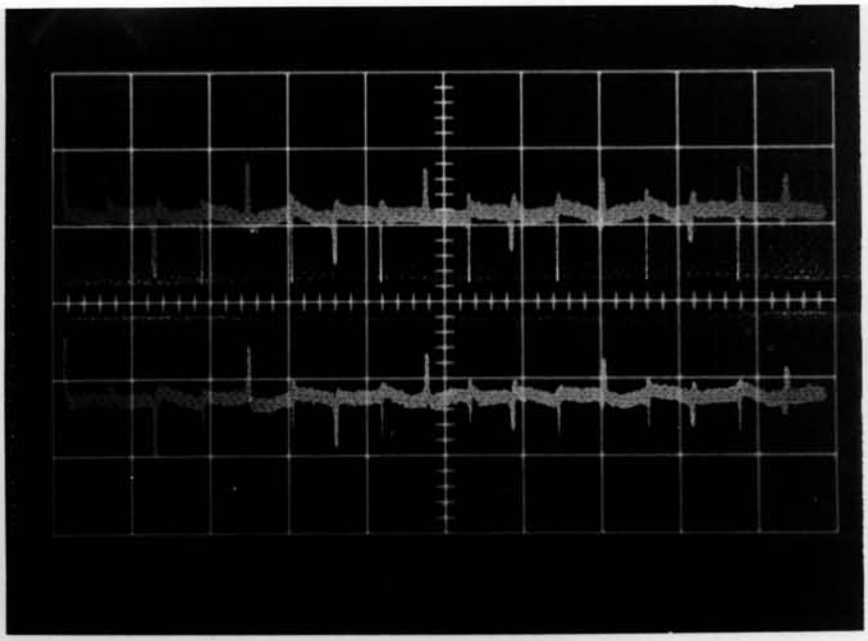


Fig.3.8 Echoes induced by Carr-Purcell pulse series

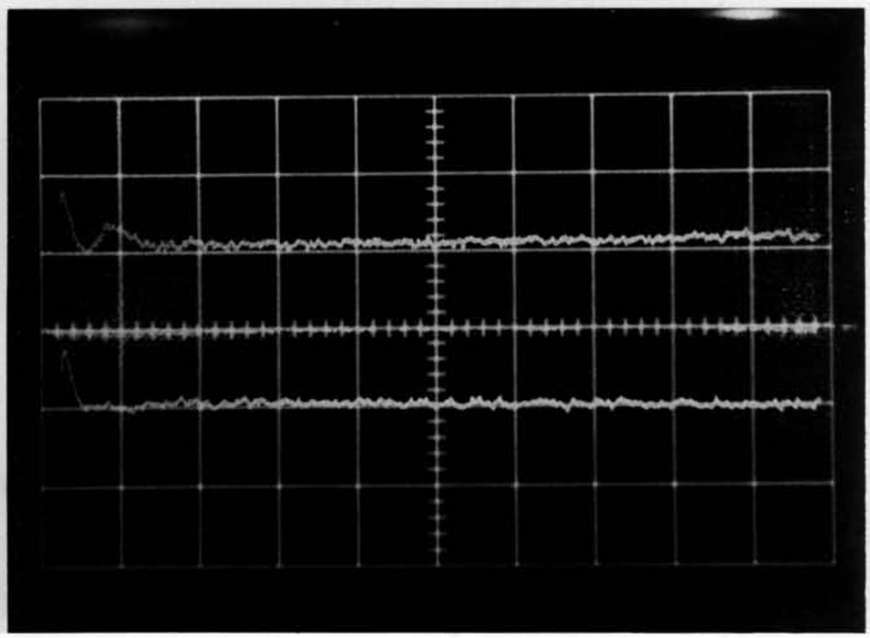


Fig.3.9 90° pulse induction tail in nematic phase

echoes were performed by these 90°-180°pulse sequences. Many photographs of this spin echoes were taken as shown in Fig.3.8 at various temperature. The decay time constant of the echoes amplitude can be calculated to obtain T_2. In the nematic range, the echoes amplitude can not be observed because the signals are very weak (i.e. $T_2 \ll \frac{1}{\gamma \Delta H}$). Therefore the measurement of T_2 is modified by measuring the half width of the induced signal of the 90°pulse as shown in Fig.3.9. That is the decay time constant, $T_{\frac{1}{2}}$ which is used for calculating T_2 by the relation of $T_2 = \frac{T_{\frac{1}{2}}}{\ln 2}$. The experimental data are listed in Appendix II.B.

Some of the electronic units used for measuring $T_1$and T_2 are of the home-built type. They will be described briefly in the following section 3.4.

3.4 Details of Electronics Units

3.4.1 Pre-Amplifier This home-made pre-amplifier is similar to the one published by Clark (1964), but with some modification suitable for the purpose. It was designed with an amplification gain of 20 and the band width of about 3 MHz. The circuit diagram is shown in Appendix I.A. Its function is to work as an amplifier of the small nmr induced signal which is generated by the proton spin of the sample via the rf sample coil, as well as limiting very large rf signal from the transmitter.

3.4.2 RF-Amplifier This rf-amplifier circuit is exactly the same as the one of Senghaphan (1968). The circuit diagram has been illustrated in Appendix I.B. The rf-amplier is work for re-amplifying the induced signal which was amplified by the preamplifier. It is designed with a band width of 3 MHz and the amplification gain of 1000.

3.4.3 Pulse Amplifier The home-made pulse amplifier is designed with a low output impedance as the circuit diagram shown in Appendix I.C. Its function is to amplify the pulse signal which is generated by the Tektronix pulse generator type 163. The amplified pulse of amplitude +60 volts is used for operating the gated rf transmitter.[10]

3.4.4 Phase Sensitive Detector A home-made electronics instrument with high sensitivity is used to extracting the sharp resonant condition. By using the rf reference signal from the rf oscillator superimposed with the rf induced signal at the same resonance frequency, a large AF induced signal envelope will be formed as an output. This output depends on relative phase of the signal and the refference rf signal. They become beats if these two signals have a slightly different frequency. This phase sensitive detector is maintained to have a high amplification gain so that a very weak signal can be well detected. The circuit diagram is illustrated in Appendix I.D.

3.3.5 Pulse Sequence Trigger Unit A positive pulse is provided by a 9V. drycell battery and can be controlled by a button-pushing switch. This pulse is used for triggering the pulse gate on Tektronix type 161, 162 to providing a pulse sequence using for the measuring of T_2. The circuit diagram of the pulse sequence making system is shown in Fig.3.10

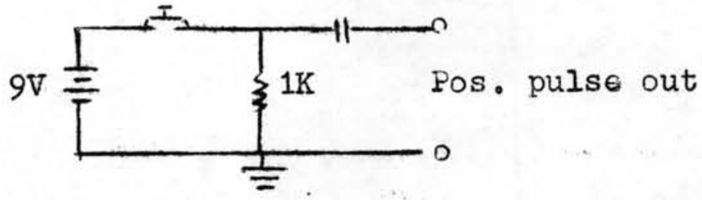


Fig.3.10 Pulse sequence trigger unit

3.5 Temperature Control and Measurement

3.5.1 Temperature Control

In this experiment, T_1 and T_2 are measured in the temperature range 380^o- 450^oK. It is important to control the temperature of the sample to stay constantly at each selected temperature, so that T_1 and T_2 will be measured properly. The temperature of the sample cell were controlled by varying d.c. current which supplies for the two heater plates. The d.c current is generated from a home-made power supplier. The power supplier used in this sample heating system is designed to be a regulated type that can provide a maximum direct current 1.5 ampere, 12 watt. The circuit diagram is illustrated in AppendixI.F.

3.5.2 Temperature Measurement

Since we have to know the temperature of the sample as we measure T_1 and T_2, the method of temperature calibration is importance to be used. The temperature of the sample can be obtained by reading an ordinary thermistor resistance which is strongly temperature dependence. The thermistor resistance is calibrated with an ordinary mercury thermometer. We can obtain a relation between the sample temperature and the thermistor resistance as plotted graphically in Fig.3.11. The thermistor

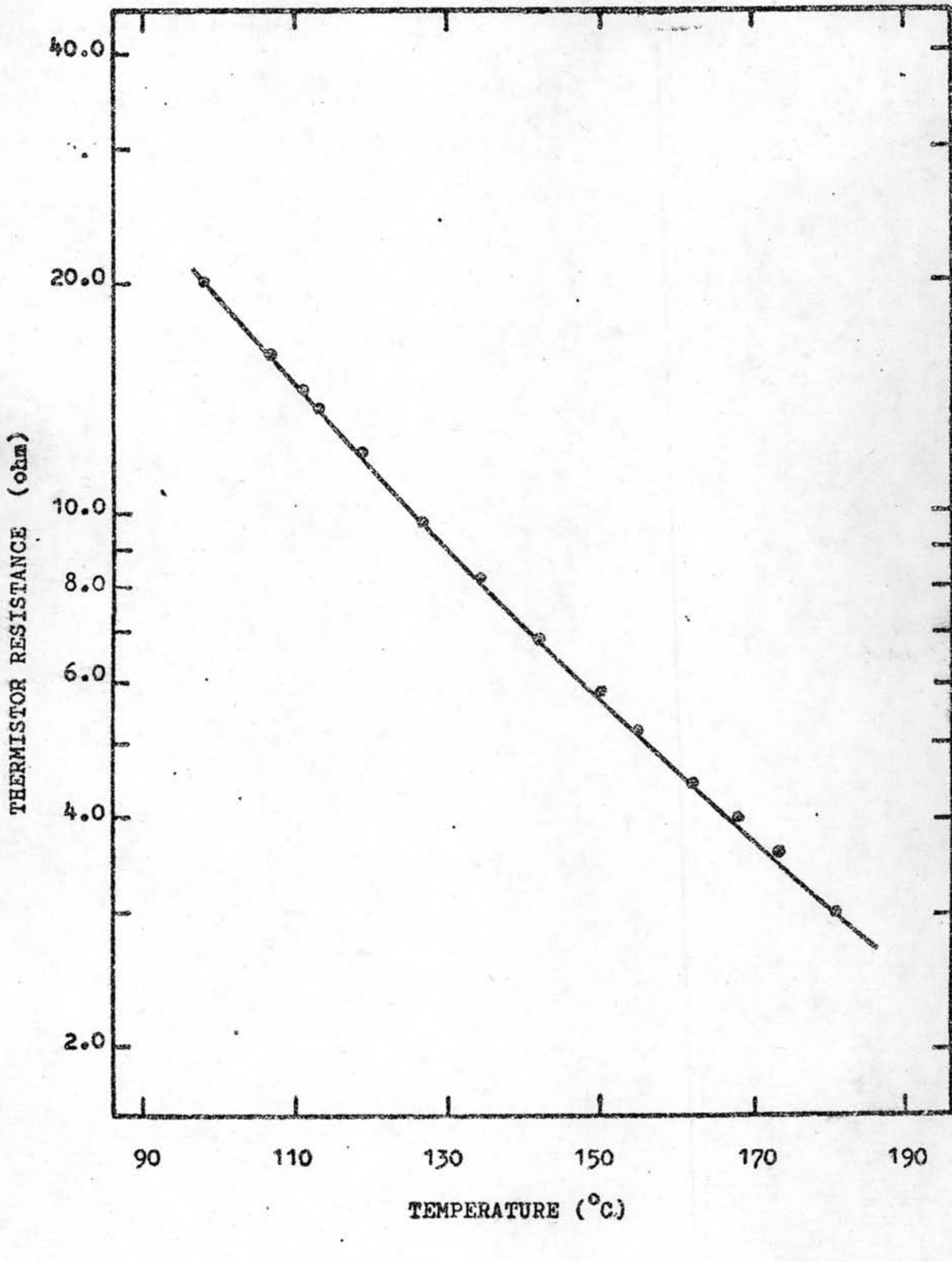


Fig.3.11 Temperature calibration

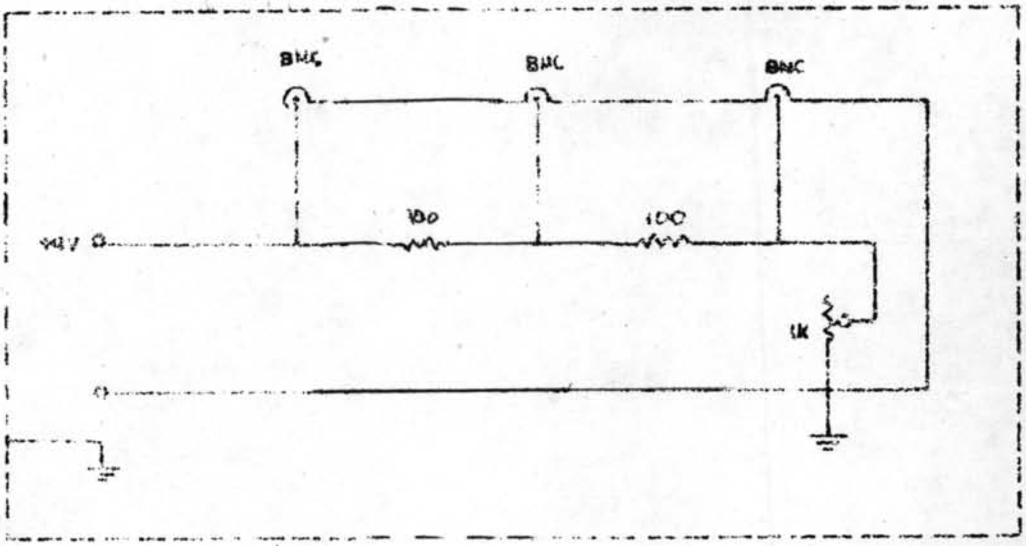


Fig. 3.12a D.C. bridge meter

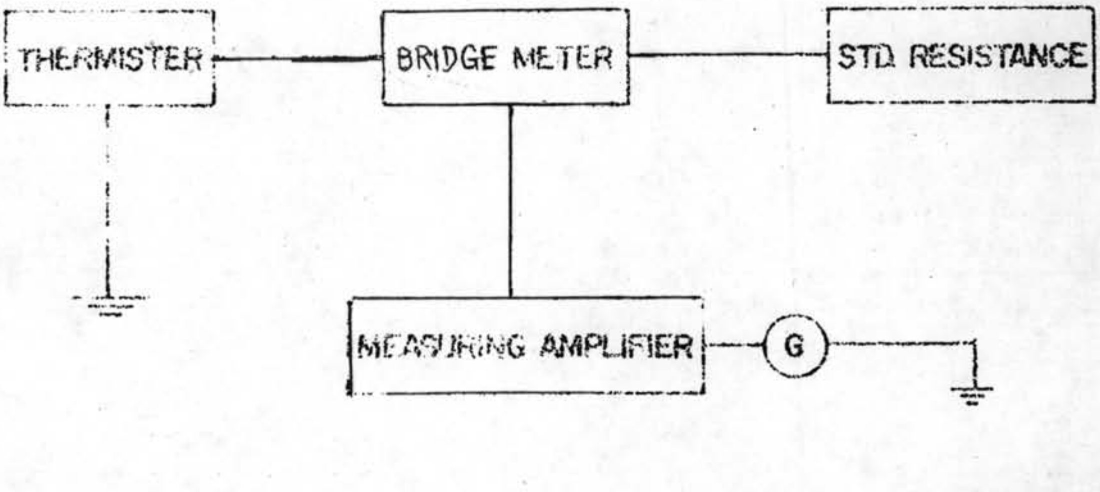


Fig 3.12b D.C. bridge circuit

resistance can be obtained directly by reading from a standard decade resistance box in the resistance bridge. The d.c. bridge circuit for the temperature measurement was set up as in the block diagram shown in Fig.3.12 b.

A bridge meter unit is one part of the Wheastone Bridge designed in a compact form for convenient use. The circuit diagram is shown in Fig.3.12a. There are two identical 100 ohm resistance and a 1K-potentiometer is used for sensitivity controlling. The measuring amplifier is used for amplifying the d.c. current which passes through a galvanometer when the bridge is not in balance condition. The sensitivity of the bridge balancing can be observed from the galvanometer scale.

Then at each temperature which is controlled by varying the d.c. current of the heater, we just only read the resistance from the standard resistance box. Hence the resistance can be calibrated back to obtain the temperature of the sample as we measuring T_1 or T_2.