CHAPTER III

APPARATUS AND EXPERIMENTAL PROCEDURE

3.1 Apparatus

Block diagrams of the apparatus for measuring T_1 and T_2 are shown in Figs. 3.1 - 3.2. It consists of a waveform generator, 2 units of pulse generators, a pulse amplifier, a gated transmitter, an rf oscillator, a phase sensitive detector, a preamplifier, an rf amplifier, and a storage oscilloscope.

The pulses and time intervals between pulses were obtained from a Tektronix Waveform Generator (type 162) and 2 units of Pulse Generator (type 163). The series of pulses for measuring T_2 were obtained by external battery gating of the waveform generator and by triggering the 161 unit. The oscillator and gating transmitter were devised by Blume and was constructed by W. Senghaphan in 1968.[9]

The preamplifier, rf amplifier and phase sensitive detector were constructed by the author and Phietoon Trivijitkasem who shared the laboratory. The circuits are shown in Figs. I.A, I.B. and I.C. in the appendix. The preamplifier was similar to that devised by Clark.[12] It had high sensitivity, and recovered quickly from large overload signals. The use of a high g_m tube at the input provided low input noise. Fast overload recovery was insured by directly coupling the receiver coil to the input stage and by limiting the voltage with crossed diode at the plate. This prevented overloading the stage that followed. The maximum resonance signal was obtained by tuning the capacitors c_1 and c_2 shown in appendix I.A.

The rf amplifier was tuned to give maximum gain at 10 Mc/sec. The frequency was changed by tuning the capacitors. The cross diode in the first two stages limits the transmitted pulse and prevents overloading the subsequent stages. A low resistance in shunt with the tuning coil on the load of the first stage serves to increase bandwidth and cuts down the gain of the amplifier keeping the signal within the amplifier's linearity. The maximum gain of the rf amplifier is about 1000 times.

The phase sensitive detector was used to remove the rf signal and retain the amplitude. The reference signal was fed onto the controlled grid of the pentode and the reference signal from the rf oscillator was fed onto the suppressor grid. The pentode thus acted as a mixer with excursions of the voltage at the plate being proportional to the product of the voltages applied. If the input signal and the reference signal are in phase the output at the plate was maximum.

The oscilloscope in this investigation was the Tektronix type 549, which can store induced signals.

3.2 T_1 Measurement

If a 180° pulse was applied to invert the magnetization $M_z = M_o$ to $- M_o$, the magnetization returned to equilibrium, because of the spin-lattice relaxation process. However the magnetization can be returned to $- M_o$ if, after a 180° pulse, a further 90° pulse is applied at τ_{null} which is the time for normal 90° magnetization. If the time chosen was exactly equal to τ_{null} the induction tail disappeared. The spin-lattice relaxation time, T_1, could be calculated directly from the formula given by Carr and Purcell.[11]

$$T_1 = \tau_{null} / \log 2$$

3.3 T_2 Measurement

A series of 180° pulses were applied after the time t of a 90° pulse. The series of 180° pulses were applied at the time $t = \tau, 3\tau, 5\tau, \ldots$ etc.and the echoes were formed at the times $t = 2\tau, 4\tau, 6\tau, \ldots$ etc . The amplitude of these echoes decreased exponentially. By plotting the logarithm of the echo amplitude against various values of 2τ, T_2 was obtained from the slope of the resulting straight line. If the time which the amplitude takes to decrease to a half was divided by log 2 the transverse relaxation time, T_2, was obtained.

3.4 Temperature Measurement

The apparatus for measuring temperature consisted of a dc.bridge with a standard variable resistance and a thermistor. The detector for balancing the bridge was a measuring amplifier, a galvanometer and the accuracy was ± 0.1 ohms. The complete apparatus is diagramed in Figs. 3.3 - 3.5. The source of heat was a resistance wire connected to the variable power supply. The circuit of the power supply is shown in Fig. I.E. in the appendix.

The calibration of temperature was made by plotting the resistance against temperature. The calibration graph is shown in Fig. 3.6.

3.5 Sample Preparation

Distilled water was sealed in a Pyrex tube about 5 mm. in diameter. The tube was cleaned by using a cleaning mixture of potassium dichromate and concentrated sulfuric acid. Immediately after distillation the water was heated in the sample tube to remove dissolved oxygen and the tube was then sealed in its own vapor. This avoided contamination by paramagnetic molecules of oxygen.

3.6 Procedure

The sample was warmed until the temperature reached 100°C and then it was cooled by decreasing the heating current from the power supply. For temperatures below room temperature, the system was covered with ice ,allowed to cool and then removed. Measurements were made as the temperature rose to room temperature. Waveforms were generated by a Tektronic Waveform Generator (type 162) and the time interval used was 10sec. The 90° pulses and 180° pulses were obtained from 2 units of a Tektronix Pulse Generator (type 163). The pulse widths for water are μ 5 sec. and μ 10 sec. for 90° and 180° pulses respectively.

For measuring T_1 a 180° pulse was applied and then followed by a 90° pulse. The delay time was varied until null induction signal right after the 90° pulse was obtained. The delay time is then τ_{null}.

For measuring T_2, a Carr-Purcell[11] series of pulses were applied after the 90° pulse. The induction signal was stored on the screen of the oscilloscope and a photograph was taken with polaroid film. The amplitudes of the echoes were calibrated and plotted against time 2τ on a semi-log paper.

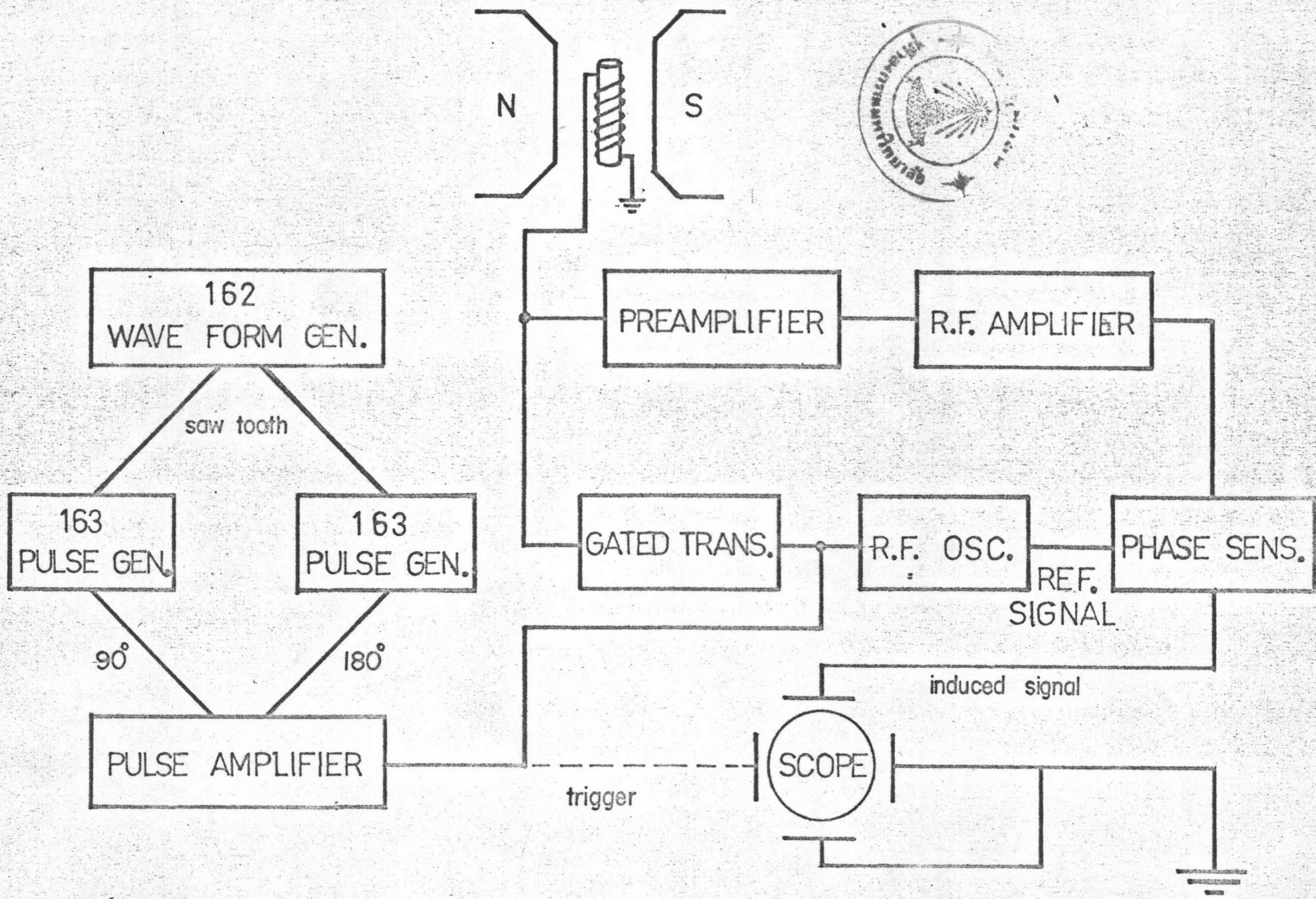

Fig. 3.1 Block diagram for measuring T_1

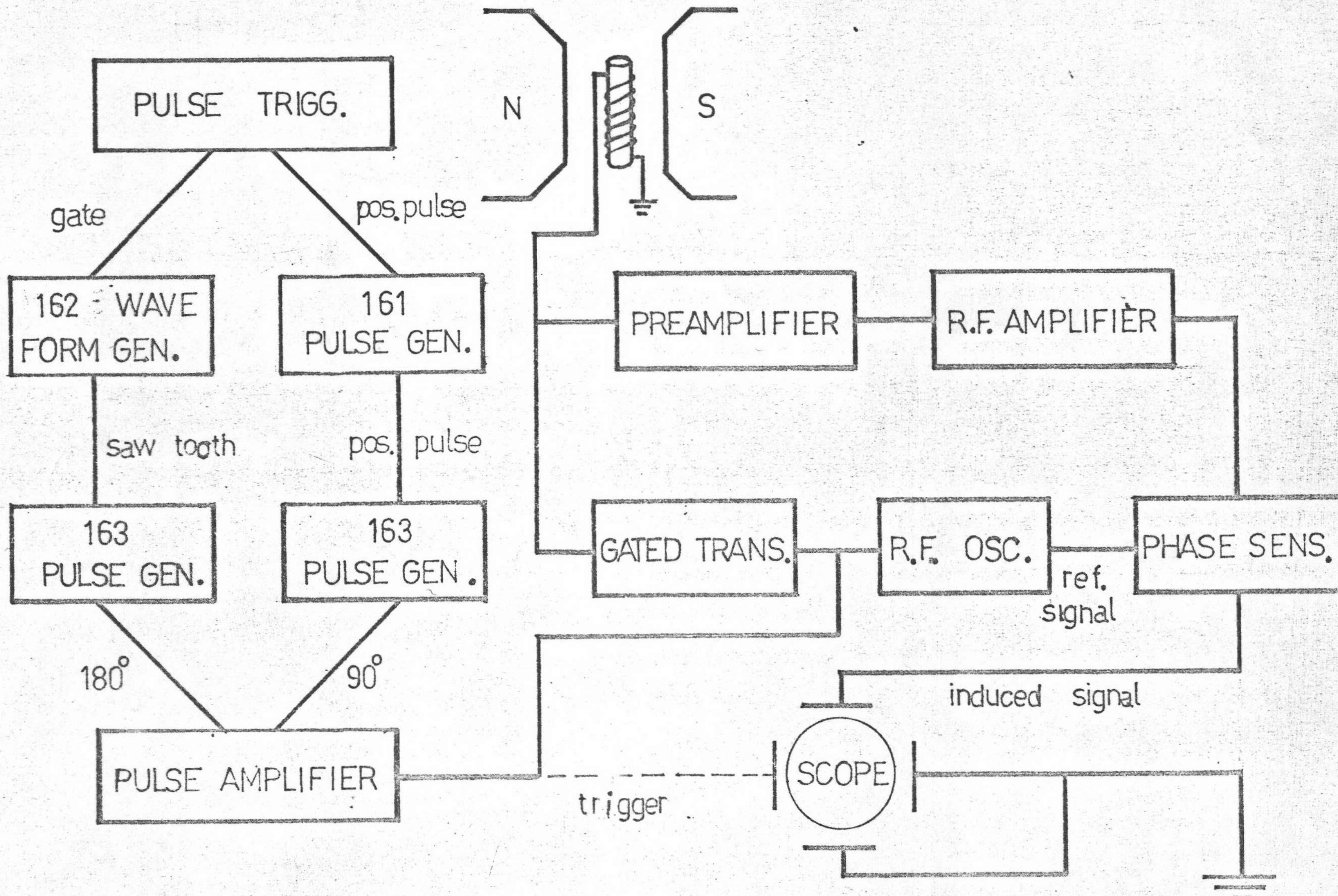

Fig. 3.2 Block diagram measuring T_2

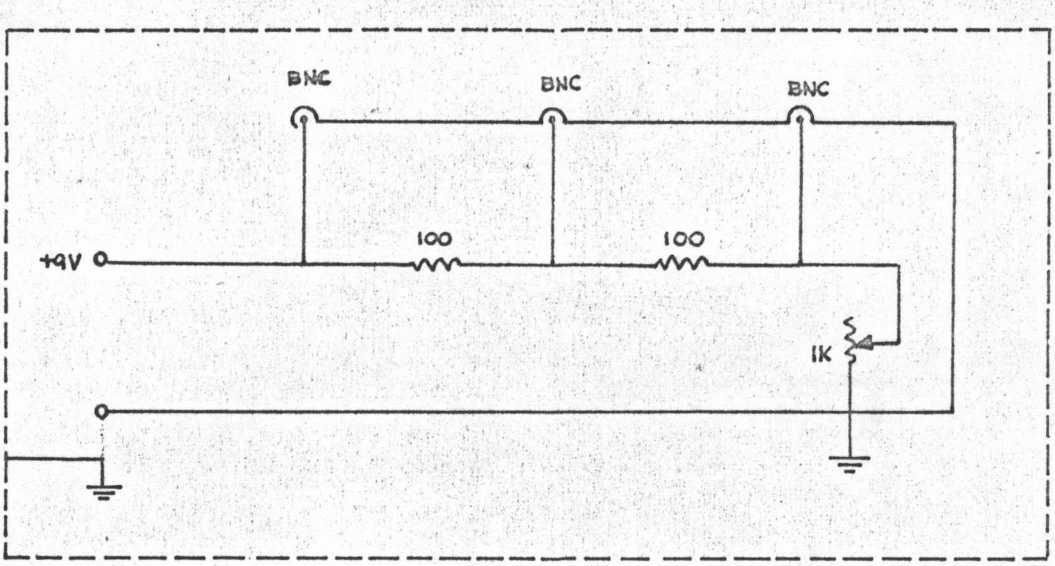

Fig. 3.3 DC· bridge meter

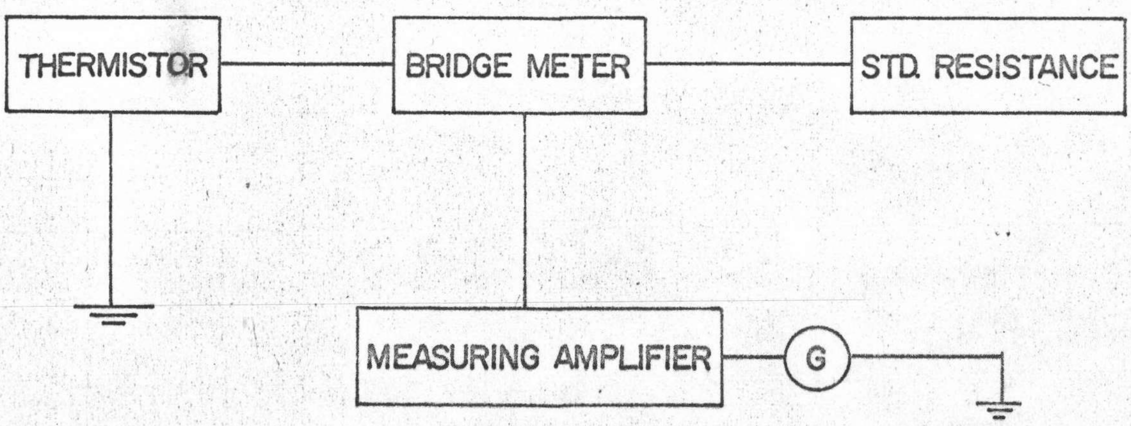

Fig 3.4 DC. bridge circuit

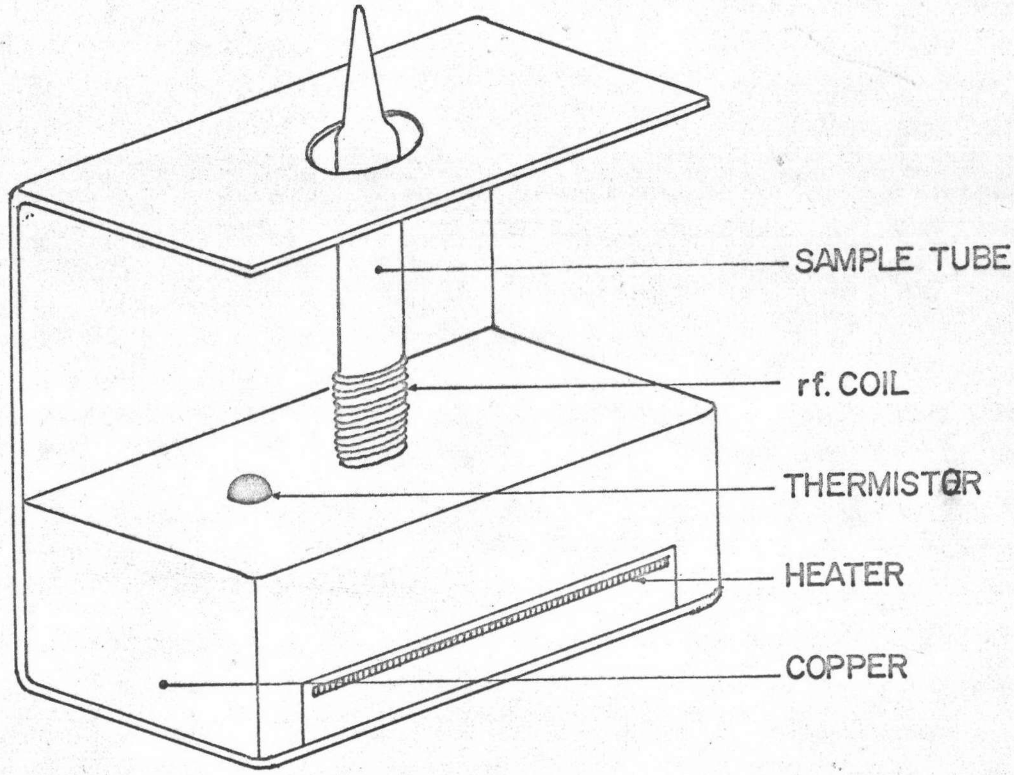

Fig. 3-5 Illustration of sample, thermistor and heater

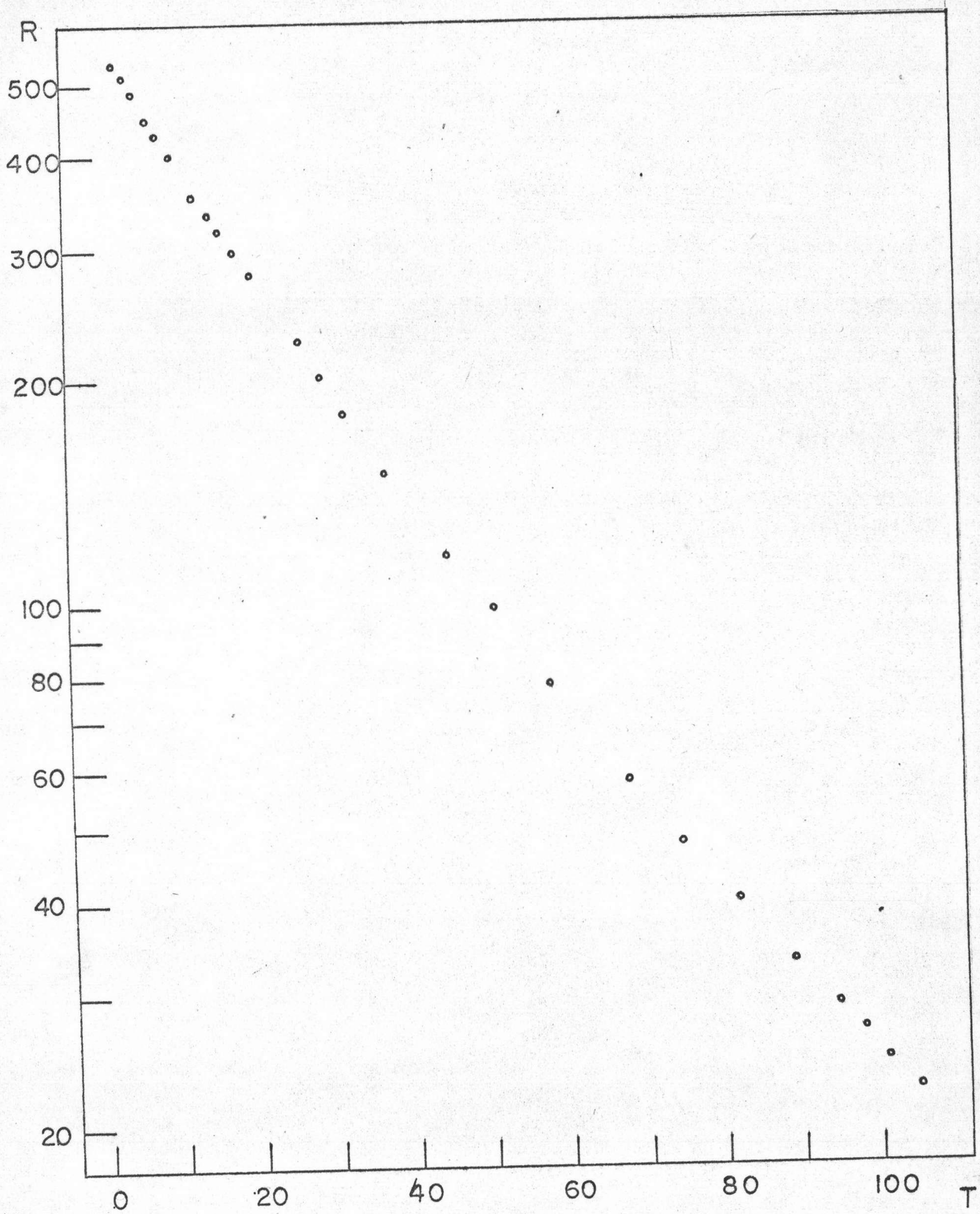

Fig. 3.6 Temperature calibration