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CHAPTER III

INSTRUMENTATION AND EXPERIMENTAL

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CHAPTER- IIII N S T R U M E N T A T I O N A N D E X P E R I M E N T A L .

The details of the Experimental techniques followed in phosphorescence decay and thermoluminescence are as follows :

3-1 Experimental set up for preparation of Phosphors :

Oven :

The charge prepared in the form of paste is dried in the oven. The oven used is Hot air Oven, universal memmer type. It has a finishing like refrigarator bearing model no.MCD 102. The chember inside is made of Aluminium and the size of the Oven is 18" x 18"x 18".

*How come Al is used.  
what is temperature range?*

Hot Plate :

Thermostatically controlled rectangular hot plate was used for drying A.R.grade charcoal. The rectangular plate has dimensions 10" x 12".

Furnace :

Furnace used for preparation of phosphor is Tempo-make crucible furnace (Refractory type, Model No. TI 57 R). The maximum temperature that can be obtained is 1150°C. The electrical rating is 1.4 KW at 240 V A.C. single phase and the temperature control is by energy regulator. The furnace is 10 cm. in diam. having depth 10 cm. The temperature measurements are done with pyrometer. Here a chromel-Alumel thermo couple is used.

### 3-2 Experimental set up for phosphorescence Decay measurements :

Experimental set up for measurement of phosphorescence decay is shown in photograph (fig. 3.1). The housing assembly of photomultiplier tube is fabricated in the laboratory. The phosphorescence decay measurements were carried out at room temperature. The block diagram of the set up is shown in fig.3.2.

The arrangement consists of :

- a) Excitation Source, (b) p.m. housing assembly, (c) pm tube
- d) E.H.T. supply unit, (e) Nanometer, (f) Stabilised supply for all these units.

#### a) Excitation Source :

For photoluminescence study, the excitation source used was a germicidal lamp giving ultraviolet rays typically of the wave length 253.6 nm (15 watts) (model no. G 15 T8). The tube is operated at 230 V. A.C. having wattage 15w. A slit having dimension 4 cm x 1 cm. is made through which the samples are excited.

*1.5 cm x 1 cm  
after 1.5 cm  
3.1 cm x 3.6 cm*

#### b) Photomultiplier tube housing assembly :

A systematic diagram of pm housing assembly is shown in fig. 3.3 A black coloured wooden box having dimensions 22x15x16 cm. is taken. A slit (width 5 cm. x 1 cm.) is grooved to it making an angle of  $45^\circ$  so that the light is incident directly on the sample. A drawer with dimension 20 x 12.5 x 4 cm. has two conducting brass rods attached as shown in fig. 3.4(a). A Kanthal strip is attached to one end of these rods while other having ebonite terminals for the external connections. This arrangement forms the sample holder. pm tube with its slit fully open is mounted above this sample holder. This can be

seen from fig. 3.3.

### Kanthal Strip :

Sample holder consists of a Kanthal strip which is a mixture of (Fe 72%, Cr 23%, Al 3%, CO 2%). The strip is having length 45 mm and thickness 0.025 mm. The width of it is 74 mm. and has a central rectangular depression of about  $\frac{1}{2}$  mm. The Kanthal strip is used for holding the sample for pL studies. TL study is also made with the same strip. A spot welded chromel Alumel thermocouple is attached to read the temperature. The systematic diagram of kanthal strip is shown in fig. 3.4(b). It is worth mentioning that the pm tube housing assembly is fabricated in Research laboratory (Deptt. of Physics ) Dayanand College, Solapur.

### c) Photomultiplier tube :

The intensity of the luminescent light is measured with the help of photomultiplier tube IP 21 made by RCA. The photomultiplier tube was operated at 975 v. dc supply obtained from EHT Unit. The amplification of the tube was  $10^6$ . The dependence of amplification factor and sensitivity of tube on operating voltage is shown in fig. (3.5). The spectral response of the tube Ip 21 is shown in fig. (3.6).

The out put from the photomultiplier tube is a linear function of incident light. The deviation from linearity are about 3% for the variation of the light flux between  $10^{-7}$  and  $10^{-3}$  lumen. The details of performance of the above tube is given below :

3.4a  
fig shows dimensions  
45 x 1.5 mm  
strip with 2mm wide  
give a huge background

What is the response of your tube  
59, 55 or 54 4

Cathode area	:	1.9 cm <sup>2</sup>
Spectral response	:	3000 - 6500 <sup>o</sup> A.
Cathode Sensitivity	:	20-40 uA/lm.
Cathode dark current	:	5 x 10 <sup>-14</sup> A.
Maximum over all voltage	:	1250 v.
Number of stages	:	9
Maximum voltage per stage	:	100 v.
Over all gain	:	2 x 10 <sup>6</sup>
Maximum current	:	0.1 mA.
Anode dark current	:	0.1 uA.
length of the tube	:	9.2 cm.
Diameter of the tube	:	3.3 cm.

d) E.H.T. supply unit for p.m. tube :

High d.c. voltage required to operate the photomultiplier tube was given from an E.H.T. power supply unit (Model EHT 10).

The power supply consists of a stable power oscillator whose out put is controlled by an input signal. A step up transformer is used to boost the out put of this oscillator out put is then rectified and filtered. A portion of this out-put is compared with a high stability temperature compensated reference and the error signal is used in the feed back path to control the out put of oscillator.

Specification of MODEL E.H.T. 10.

OUT PUT Voltage	:	150 v - 1500 v. continuously adjustable.
Current	:	2 mA maximum.
Polarity	:	+ve or -ve as required.
Regulation	:	$\pm$ 0.05% for 0 to 1 mA
Stabilisation	:	0.02% for $\pm$ 10% change in mains voltage
Out put meter	:	BPL panel meter (113 mm scale) with a resolution of 15 V/div.
Connections	:	Output through a Amphenol Coaxial connectors on the front panel
Protection	:	Protected fully against over load and short circuit by current limiting technique.
Power requirement	:	220 V A.C. $\pm$ 10% (single phase 50 Hz.)

e) Nanoammeter :

The out put of the photomultiplier tube is recorded with the help of Nanoammeter. A nanoammeter (model Nm- 121) was used to record the out put. The unit has a facility to record a current ~~deep~~ down to 10 pA.

Specifications of Nanoammeter ( Nm 121)

Applications	:	for low current measurement down to 10 pA.
Range	:	1 nA, 10 nA, 100 nA, 1 uA, and 10 uA.
Polarity	:	Positive or negative through switch.
Meter	:	BPL moving coil meter with 100 mm scale graduated in 100 divisions.
Accuracy	:	2%
Power	:	220 V. mains $\pm$ 10% at 50 Hz.

f) A C Stabiliser :

For all the instruments such as Nanoammeter, EHT Unit, UV tube linear temperature programmer, a stabilised power supply is necessary. This is obtained from AC stabiliser. Details of the stabiliser are as follows :

Model No. : CVL/250 : SER No. 1160/7558

1) Input Voltage : 180 V. to 250 V at 50 Hz.

2) Output Voltage : 230 V. with  $\pm 1\%$

A stop watch having least count 0.1 second was used for decay time measurement.

3-3 Experimental Set Up for Thermoluminescence Study :

Experimental set up for Thermoluminescence study is shown in fig. (3.1 -b) - Photograph. The assembly arrangement is shown in fig. (3.7). Two different excitation sources were used. First being ultraviolet and second gamma source. Details of the first excitation source are discussed earlier.

3-3.1 Gamma Source :

Some phosphors were irradiated using gamma source. The source was made available by B.A.R.C. Bombay Unit.

Gamma irradiation of phosphors was carried out using gamma cell 220 of Atomic energy Canada Ltd. giving 2200 R/min in Feb.83. A 10 minute dose was given in June 1988. However, actual TL study was done in August 1988.

3-3.2 Linear temperature programmer :

A linear temperature programmer Unit was built because of help of HPD. of B.A.R.C. Bombay unit. The programmer provides a constant heating rate which can be adjusted to any suitable

value. Automatic heat control facility is with it. All the samples were heated between range of room temp. to  $300^{\circ}\text{C}$

Linear temperature programmer has a facility of isothermal heating also. A calibrated temp. meter is there on the front panel for temperature measurements.

Other units such as EHT, UV source, PM tube etc. were also used for TL measurement.

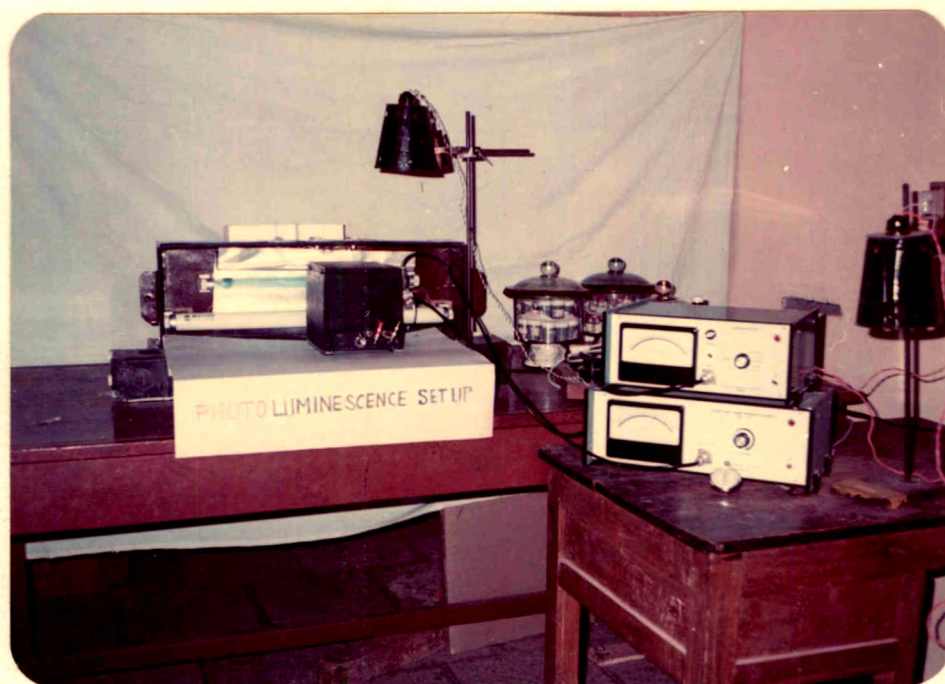
Experimental Procedure: Phosphorescence Decay Measurement :

Phosphorescence decay measurements were carried out at room temperature. A weighed quantity of phosphor (10 mg) was taken in Kanthal Strip was then excited for 5 minutes using ultraviolet source. During excitation, nanoammeter was switched off. After excitation, the uv source was switched off and simultaneously the nanoammeter switch was made on; and decaying intensity was measured. Dark current present was 26 nA.

Experimental Procedure : Thermoluminescence Measurement :

The thermoluminescence measurements were carried out in the temperature range from  $27^{\circ}\text{C}$  to  $300^{\circ}\text{C}$ . The weighed samples were excited with irradiation and ultraviolet source for ten minutes. These samples were then heated by linear temperature programmer with fast heating rate of  $1.32^{\circ}\text{C}/\text{sec}$ . TL intensity was measured by nanoammeter.

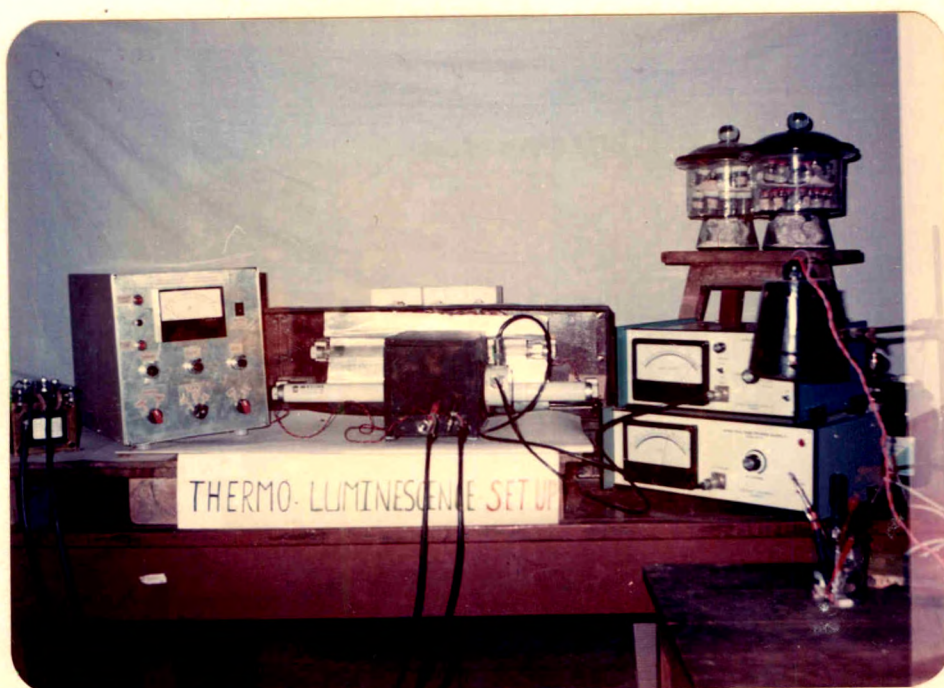




PHOTOLUMINESCENCE SET-UP

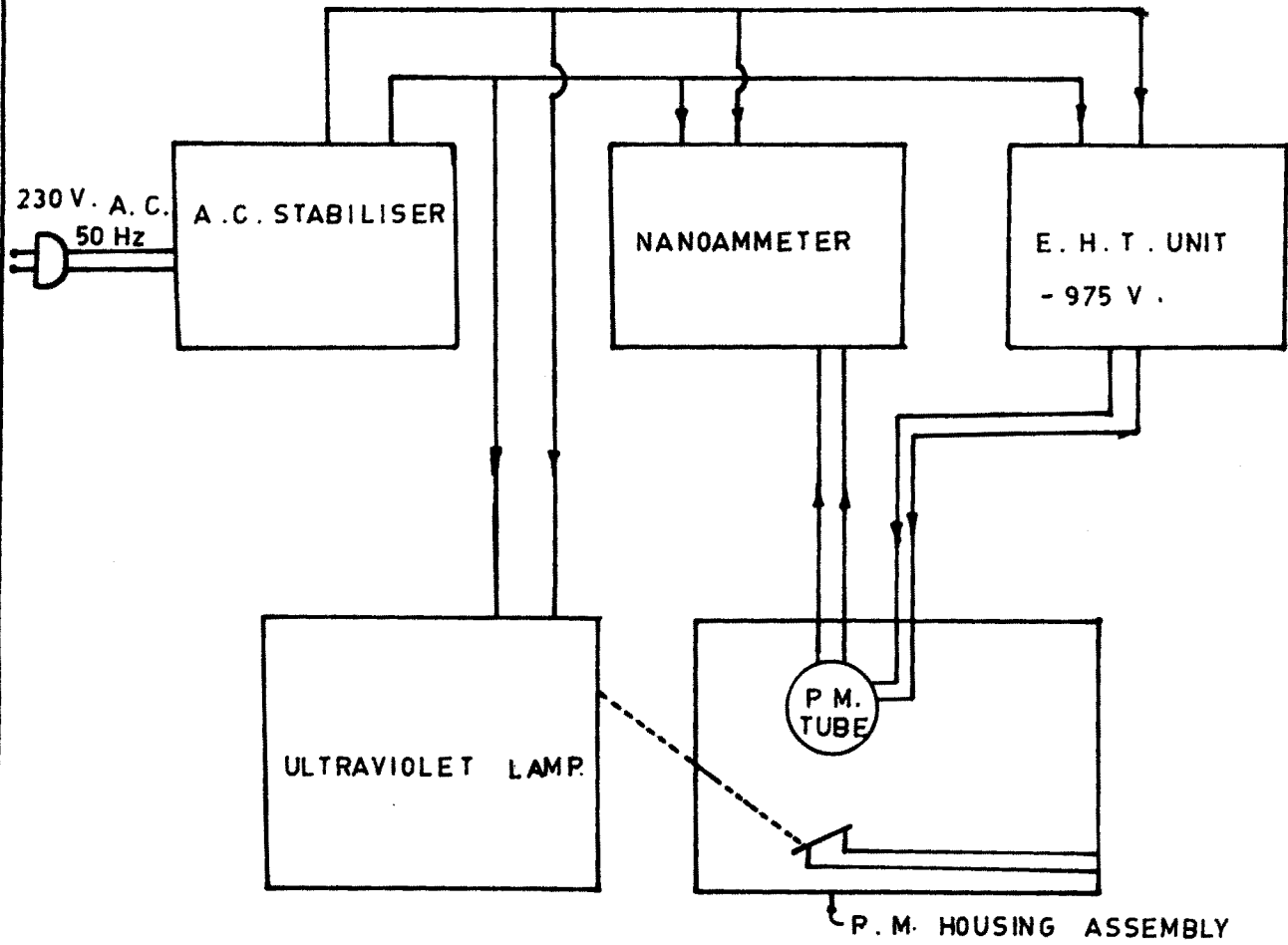
Fig. 3.1(a)





THERMOLUMINESCENCE SET - UP

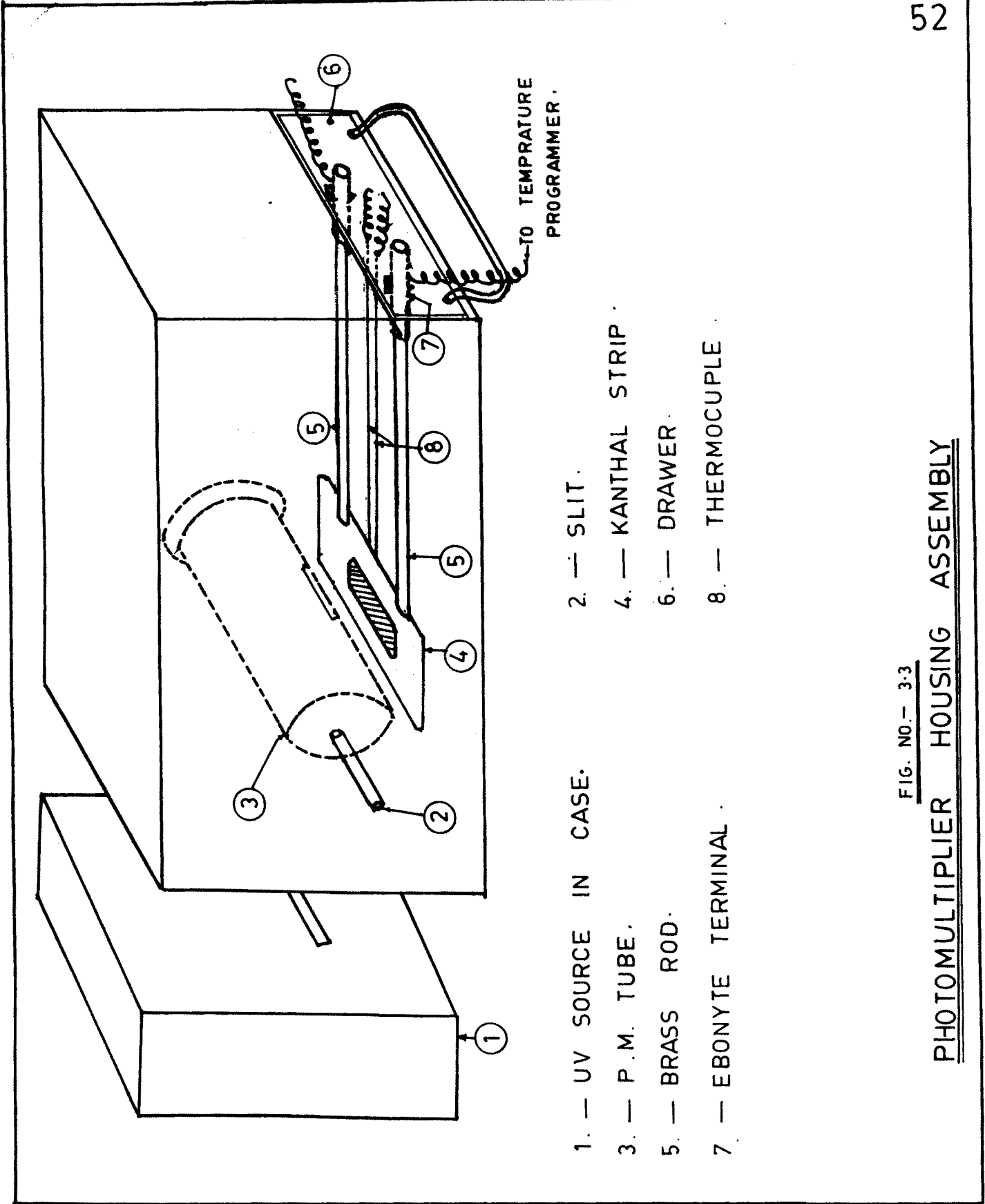
Fig. 3-1 (b)



BLOCK DIAGRAM OF PHOTOLUMINESCENCE

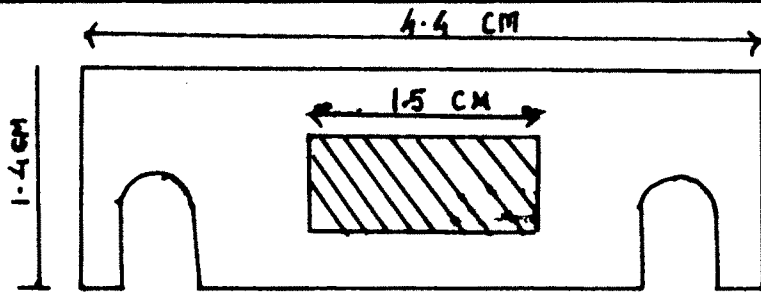
SET UP

FIG. NO.- 3-2



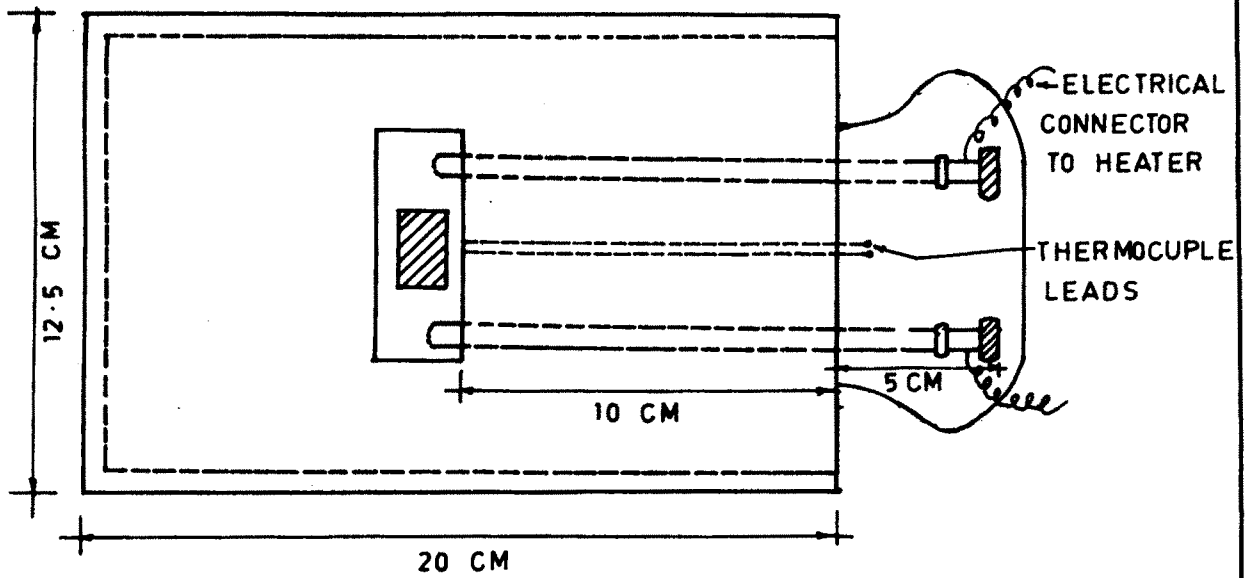
- 1. — UV SOURCE IN CASE.
- 2. — SLIT.
- 3. — P. M. TUBE.
- 4. — KANTHAL STRIP.
- 5. — BRASS ROD.
- 6. — DRAWER.
- 7. — EBONYTE TERMINAL.
- 8. — THERMOCOUPLE.

FIG. NO.— 3-3  
PHOTOMULTIPLIER HOUSING ASSEMBLY



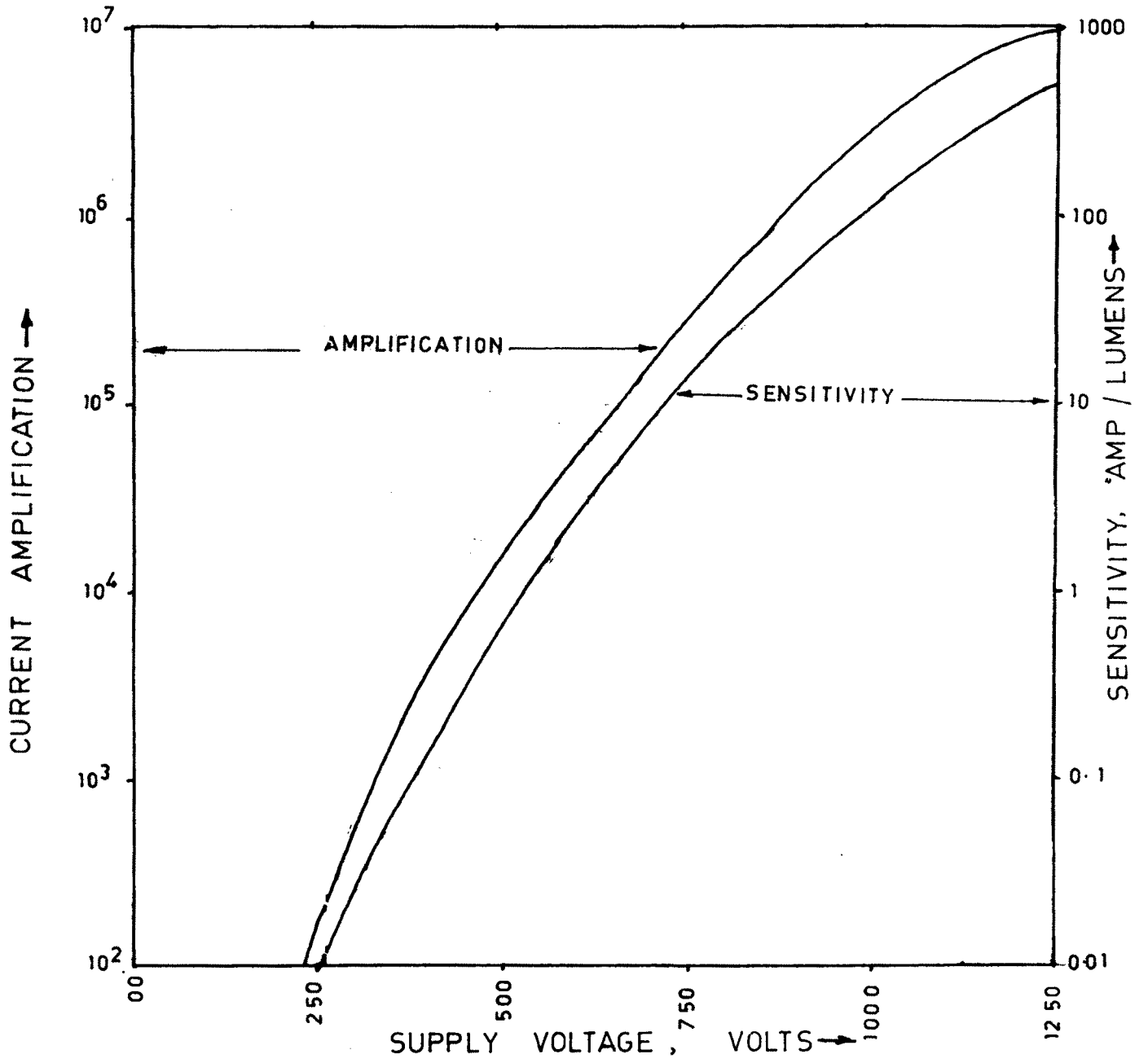
KANTHAL STRIP

FIG. NO. 3-4 (b)

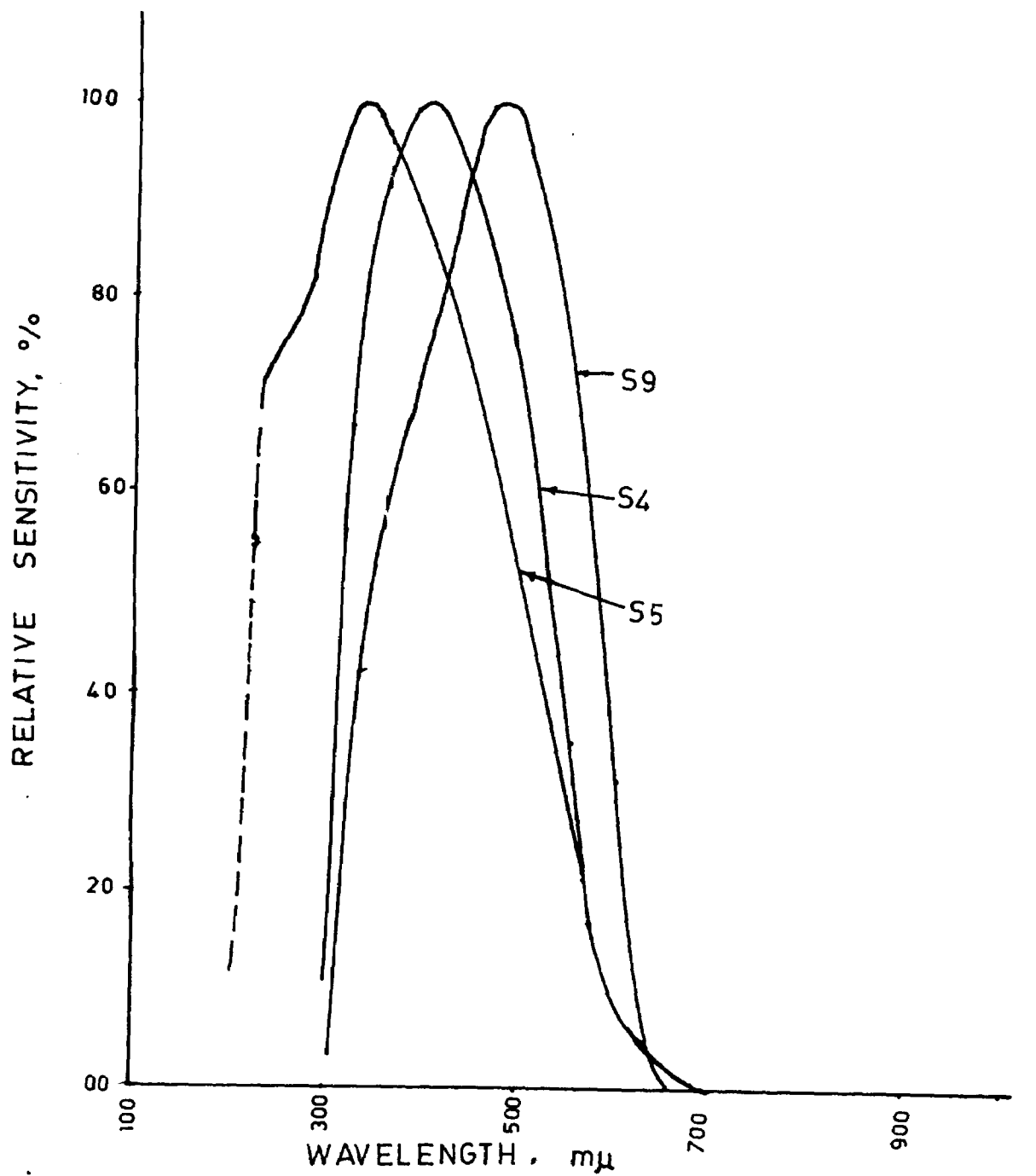


DRAWER ASSEMBLY

FIG. NO. - 3-4 (a)



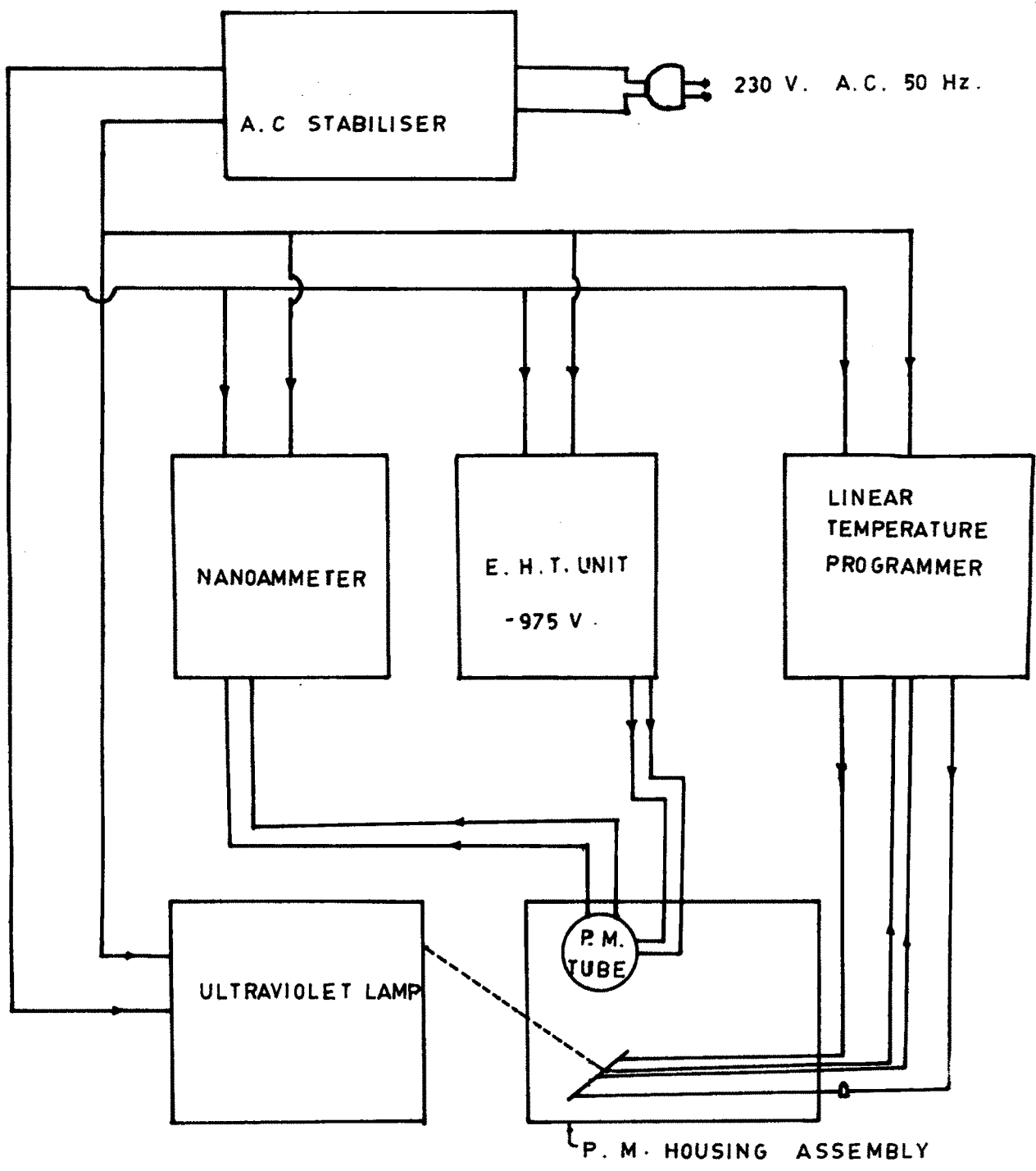
OVERALL AMPLIFICATION & SENSITIVITY OF A PHOTOMULTIPLIER



RELATIVE SENSITIVITY OF DIFFERENT COMMERCIAL

PHOTOCATHODES

IP 21



BLOCK DIAGRAM OF T.L. SET UP

FIG. NO. - 3-7