CHAPTER -IV

EXPERIMENTAL

(A) PREPARATION OF SAMPLES

Luminescence is due to the defects in the crystal structure of the host and preparation of phosphors involve creation of defects in crystal matrix. Different conditions of preparation often lead to the formation of new materials. This chapter briefly gives the description of the preparation of NaCl:Ba and KCl:Ba samples, used in the present investigations.

In present work the host materials used are pure "analar grade" NaCl and "GR proanalysi grade" KCl which are respectively supplied by BDH Laboratory Chemical Division, Bombay and Sarabhai Chemicals, Baroda, India. They were certified to have purity of 99.9%. The maximum probable impurities, according to the manufacturers are as follows :

Sr. No.	Material	Inherent impurities
1.	Sodium Chloride "Analar grade"	Insoluble matter $3x10^{-3}$%, Free acid (HCl) $1.8x10^{-3}$%, Free alkali 0.05 ml N/1%, Bromide and Iodide $5x10^{-3}$%, Ferrocyanide $Fe(CN)_6$ 10^{-4}%, Sulphate (SO_4) $2x10^{-3}$%, Nitrate (NO_3) $5x10^{-4}$%, Phosphate (PO_4) $5x10^{-4}$% Arsenic (As) $4x10^{-5}$%, Barium $1x10^{-3}$%, Calcium group and magnesium $4x10^{-3}$%, Iron (Fe) $3x10^{-4}$% heavy metals (Pb) $5x10^{-4}$% and Potassium (K) $1x10^{-2}$%.
2.	Potassium Chloride ("G.R. Proanalysi grade")	Insoluble matter $3x10^{-3}$%, Free acid (HCl) $1.8x10^{-3}$%, Free alkali (KOH) $1.8x10^{-3}$%, Arsenic $4x10^{-5}$%, Barium $1x10^{-3}$%, Iron $3x10^{-4}$%, heavy metals as Pb $5x10^{-4}$%,

Bromide and Iodide $5x10^{-3}$%, Chlorate $3x10^{-3}$%, Phosphate $1x10^{-3}$%, Sulphate $1x10^{-3}$% and total nitrogen $1x10^{-4}$%.

Extreme care was taken with regard to cleanliness and the purity while preparing and handling the samples. All the surfaces which come in contact with phosphor had been cleaned with hydrochloric and nitric acids. After cleaning the surfaces were kept in contact with boiling water for nearly two hours and then dried thoroughly in an oven.

The impurity concerned in the present investigation is barium used in the form of salt viz. barium chloride. Barium chloride is supplied by BDH Laboratory Chemical Division, Bombay, with 99.9% purity. Introduction of the impurity into sodium and potassium chlorides was accomplished by the usual method of recrystallization from aqueous solution. The weighed quantity of sodium or potassium chloride was dissolved in double distilled demineralized water and the weight of impurity as determined by the molar calculation was added to the solution, subsequently the solution was heated slowly on hot plates, untill the excess of water was completely driven out. Microcrystals were collected, dried at 40°C, powdered and mixed homogeneously. The specimens of NaCl:Ba and KCl:Ba with three different Ba-impurity concentrations namely, 10^{-5}, 10^{-2} and 10^{-1} molar fraction prepared in this manner were designated as "as received" samples.

(B) THERMAL ANNEALING TREATMENT

The heat treatments of the specimens, as received from solution involved their annealing at 500°C and 750°C. An air oven having ON/OFF type temperature controller was used which showed the maximum variation of ±2.5°C. The air oven was operated on A.C. mains, 220 volts, 50 Hz, power 2 KW.

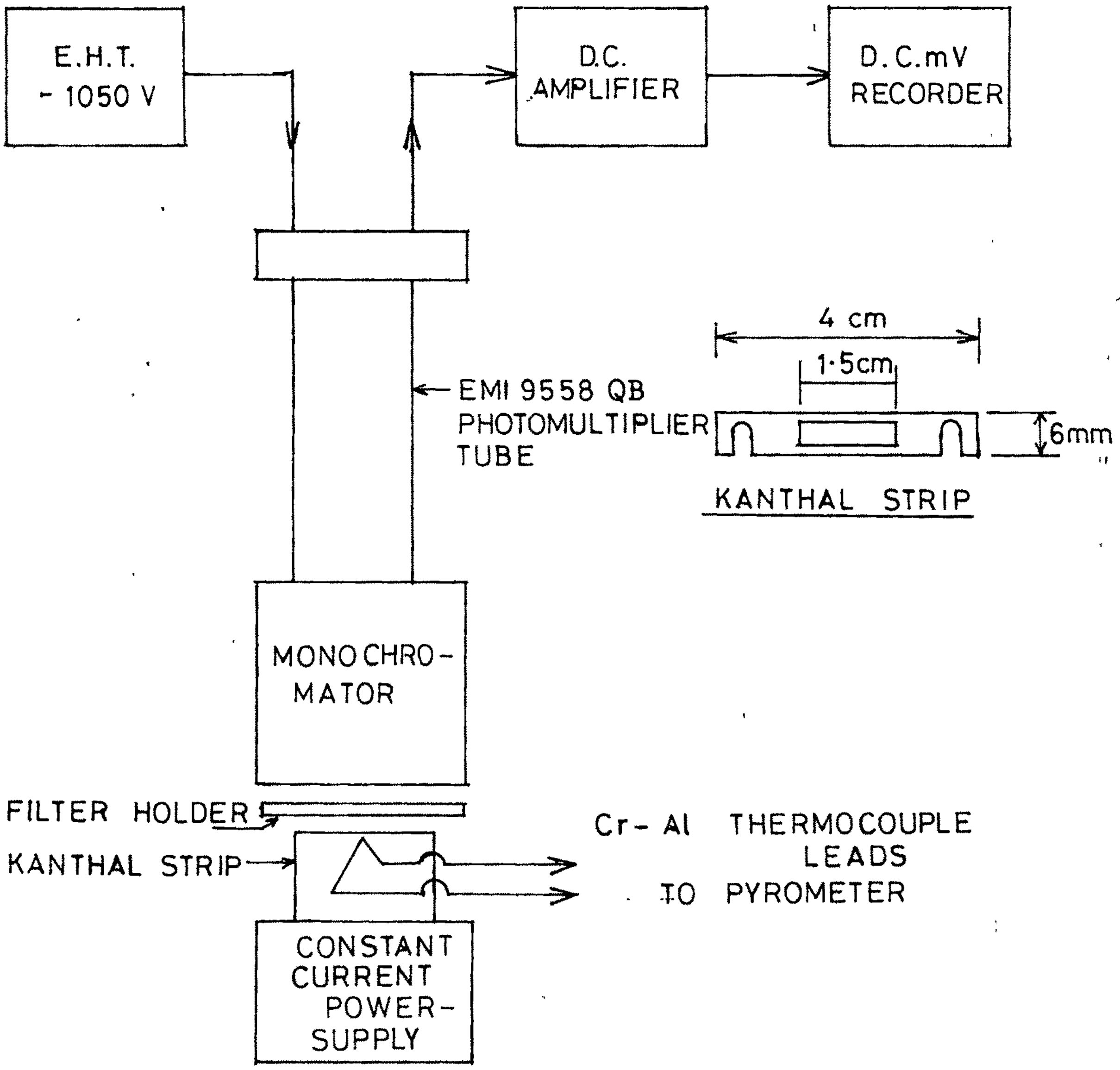


FIG. IV-1: BLOCK DIAGRAM OF TL EMISSION SPECTRA RECORDER.

The samples were annealed for two hours in a silica boat and quenched to room temperature by exposing them to a blast of cold air, such specimens are designated as annealed and quenched or pre-heat treated or thermally treated specimens.

(C) SOURCES OF IRRADIATION

(i) Alpha irradiation

For the alpha irradiation of the specimen ^{241}Am, 200 micro-curie source having the dose rate of 3800 R. min^{-1} was used.

(ii) Beta irradiation

For the beta irradiation of the specimen ^{90}Sr, 20 milli-curie source having the dose rate of 800 R.min^{-1} was used.

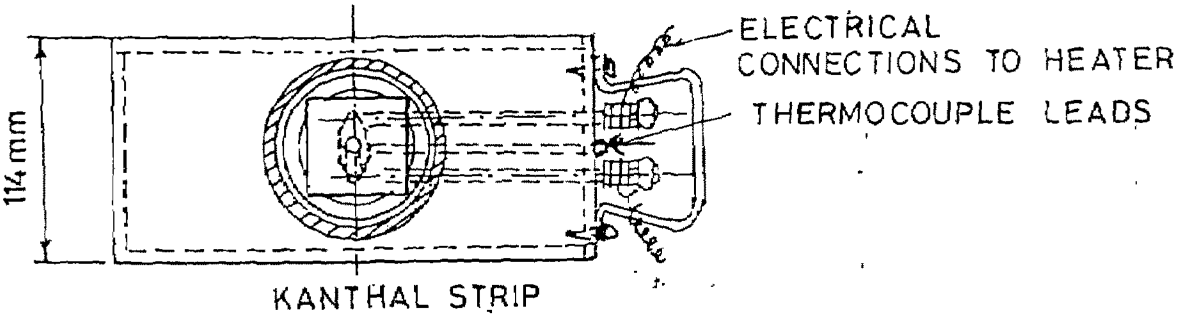
(iii) Gamma irradiation

The specimen in a small packet was exposed to ^{60}Co Gamma cells, having the uniform exposure rate of 800 R. min^{-1}. The dose was standardized and corrected periodically for ^{60}Co decay.

(D) INSTRUMENTATION

(i) TL Glow Curve recorder

A TL glow curve recorder consists basically of a heating arrangement for the sample, a photomultiplier tube (PMT) to sense the TL emission, an amplifier to measure the PMT out-put current and a chart recorder to record this current as a function of temperature. A schematic diagram of the system is given in Fig. IV-1. The sample is spread uniformly on a metallic (Kanthal) strip which is an alloy of Cr - 23 %, Co - 2 %, Fe - 72 % and Al - 3 %, which acts as a heater. Size of the Kanthal



SECTIONAL PLAN OF BAKELITE DRAWER

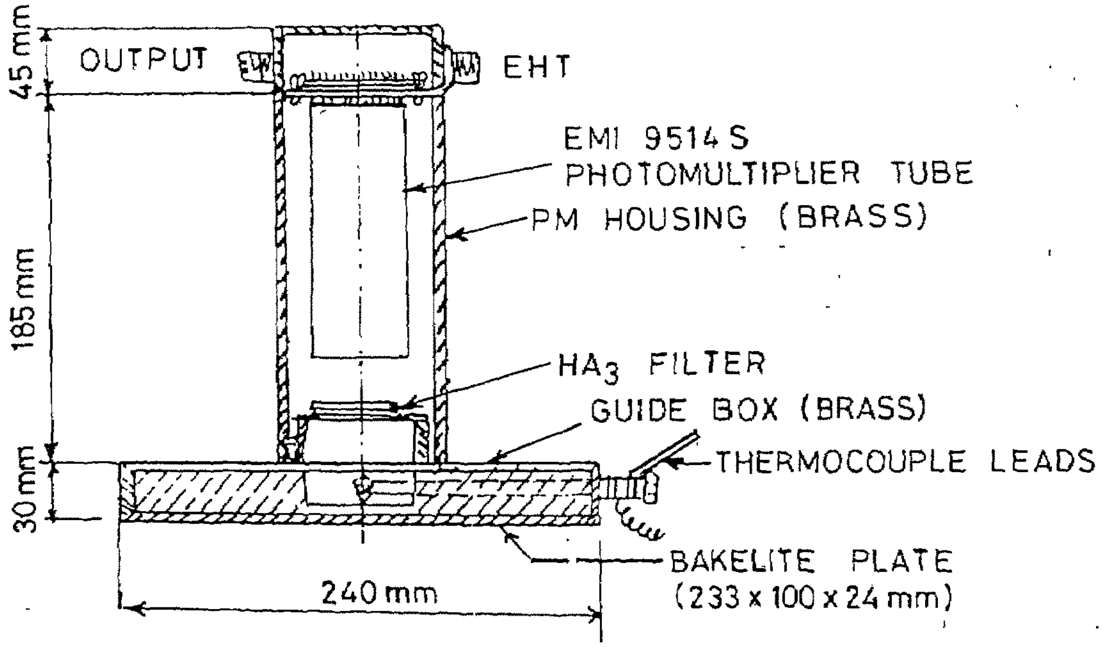


FIG. IV-2: PHOTOMULTIPLIER TUBE HOUSING ASSEMBLY strip is kept as 30 x 5.5 x 0.25 mm^3 with a circular depression of 5 mm diameter and 0.5 mm depth at centre. A Chrome - Alumel thermo couple is spot welded at its centre on the lower side of Kanthal strip. Because of the small size of the heater, high temperature can be attained with low power input and cooling is very fast after the power is switched off. Kanthal strip is secured tightly between two brass electrodes. The electrodes are fitted in 233 x 100 x 24 mm^3 backelite plate which can slide in and out of brass guide box at the top of which an EMI 9514 S photomultiplier tube with S-11 response is housed in a light tight brass cylinder. Negative voltage of 1050 volts is applied to the photomultiplier tube. When the bakelite plate containing the Kanthal strip and the sample is fully inside the guide box, sample is placed in front of the PM tube window. Fig. IV-2 gives the design tube housing assembly. Power to the strip is fed from Hewlett Packard F. and M. Model 240 temperature programmer, which can heat the sample linearly from room temperature to 1000°C, at a rate of 0.5°C/min. to 1000°C/min. and also at constant temperature, anywhere from room temperature to 1000°C can be maintained. In the present work, all the glow curves were recorded with 5 mgm of sample using a linear heating rate was detected by the PM tube and its output was fed to digital 10 mV strip chart recorder through a d.c. amplifier. Response time of the recorder was less than one second (full scale) and the chart speed was 5 cm/minute.

(ii) TL emission spectra recording system

TL emission spectra were recorded by using Jarrel-Ash-Ebert Monochromator. It had grating blazed at 300 nm and 600 nm which could be selected by a knob. The grating were ruled with 1180 lines/mm over an area

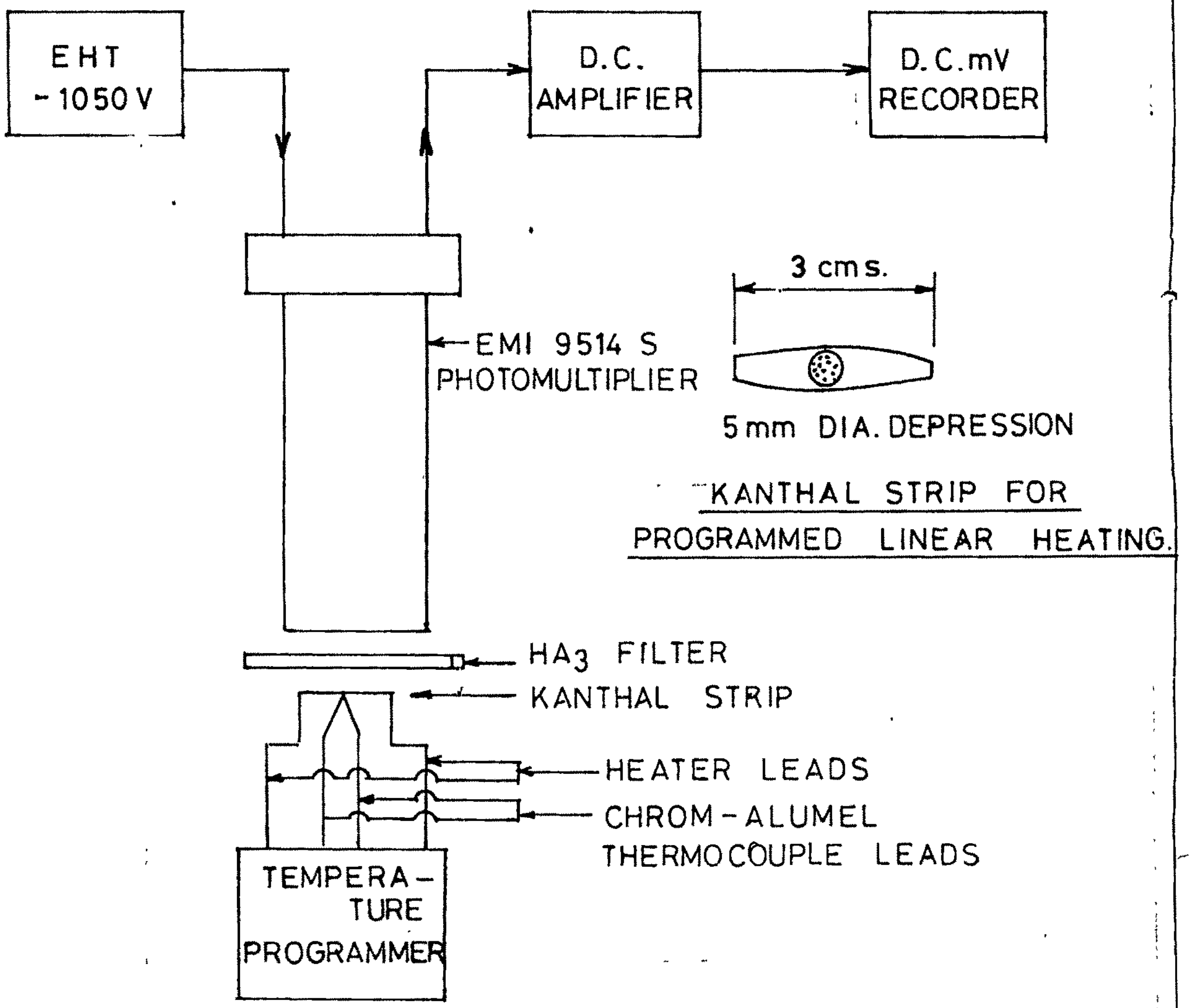


FIG. IV-3: BLOCK DIAGRAM OF PROGRAMMED LINEAR HEATING TYPE GL GLOW CURVE RECORDER of 64 x 64 mm^2 and gave a dispersion of 3.3 nm/mm. 2 mm wide slits were used at both entrance and exit ends of the monochromator.

300 nm blazed grating was used for recording the spectrum in the wavelength range 200-500 nm. About 30 mgm of the sample was spread uniformly on the kanthal strip and placed in front of the entrance slit of the monochromator. The kanthal strip used here had the dimensions 40x6x0.25 mm^3 with 15x5 mm^2 of central area embossed to 0.5 mm depth was secured tightly between the brass electrodes. Specimen was maintained at different constant temperatures, 75°C, 100°C, 130°C and 150°C for the record of TL emission spectra.

For emitting TL light EMI 9558 QB photomultiplier tube having S-20 response was mounted at the exit slit of the monochromator. Output of the photomultiplier tube was fed through a D.C. amplifier to a General Electric 5 mV strip chart recorder. The grating of the monochromator was rotated using a small motor of speed 4 RPM. Wave-length of light at the exit varied at the rate of 100 nm/min. and the chart speed for recording the spectra was 5 cm/min. A block diagram of the experimental arrangement is shown in Fig. IV-3.