

CHAPTER II

EXPERIMENTAL

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Our intension in the present work was mainly to study the electroluminescence properties of pure organic compounds such as pyrene, anthracene, perylene and two phase organic systems like anthracene doped with pyrene and pyrene doped with perylene.

The experimental work done for this study can be divided into three stages, namely,

1. Purification of organic compounds,
2. Preparation of phosphors,
3. Studies on electro-optical properties of pure organic compounds and prepared phosphors.

2:1 PURIFICATION OF ORGANIC COMPOUNDS :

Solid organic compounds isolated from organic reaction were seldom pure. They were usually contaminated with small amounts of other compounds i.e. impurities. Even a trace of impurity in the compound markedly changes its electro-optical properties and therefore, the purification of the organic compounds often must be performed prior to electroluminescence study.

Anthracene procured from C.A.F. Kahalabum, Chemisine Fabrile, Berlin-Adlershof and pyrene obtained from Fluka AG, Buchs SG, Switzerland, were purified separately by the process of adsorption column chromatography⁸⁵ as described below.

ADSORPTION COLUMN CHROMATOGRAPHY :

Solutes having different adsorption coefficient towards a certain solid can be separated by liquid-solid chromatography. It involved preparation of long cylindrical column in which solid adsorbent was packed.

It consisted of glass tube 40 cm long and 16 mm external diameter fitted into a 250 ml suction flask and supporting a 50 ml dropping funnel as shown in Fig-1. The apparatus was first washed with chromic acid and rinsed successively with water and acetone. It was then dried by blowing a current of dry and hot air. The finely divided activated alumina used as adsorbent. A cotton wool plug was inserted at bottom of column and activated alumina wetted in A.R. grade benzene was filled in column to about 3 to 4 cms column length. The column was pressed down gently and firmly by dropping a glass pestle on to it several times from the height of 5 to 10 cms. The process was repeated until a column of 20 cms length was obtained. Lastly a small loose plug of cotton wool was inserted on the top of column. Care should be taken to keep the column always wet by benzene or hexane.

A saturated solution of anthracene was prepared in 50 ml A.R. grade n-hexane and filled in dropping funnel. Solution was allowed to run down from the top of column. As solution penetrates

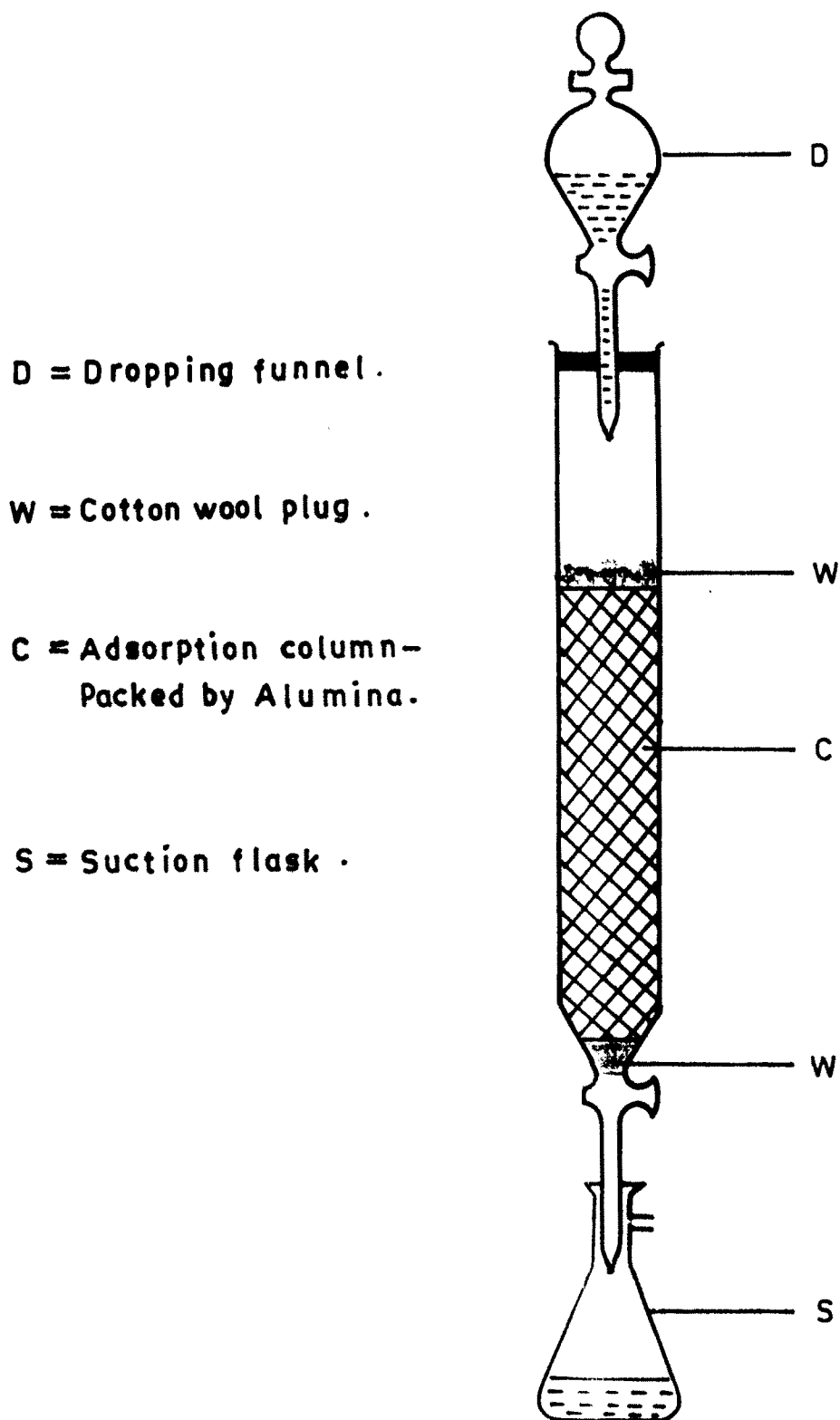


Fig. 1 — ADSORPTION COLUMN CHROMATOGRAPHY APPARATUS.

into the column, the solutes (organic compounds) were adsorbed. When the solution had just completely entered the column, the chromatogram was developed with fresh 200 ml n-hexane and process was followed with u.v. lamp.

- a) top narrow band (ca.3 cms) blue fluorescent band of carbazole,
- b) middle band yellow coloured (Ca. 4 cm) of naphthacene, and
- c) bottom broad (Ca-12 cms) blue-violet fluorescent band of anthracene.

Development of chromatogram was continued till fluorescent material commences to pass into elute. Anthracene was eluted with mixture of benzene and hexane in 1:1 proportion. Solvent was then evaporated from elute under reduced pressure. The process was repeated several times to obtain sufficient quantity of pure anthracene.

Using same procedure pyrene also purified. The compounds obtained in this method were of fluorescent grade and hence further purification was not necessary.

The perylene obtained was fluorescent grade and hence it was used directly from the bottle.

2:2 PREPARATION OF PHOSPHORS :

Basic material (host) in polycrystalline form is doped with doping agent at different dopant concentration (10^{-1} , 5×10^{-2} and 10^{-2} mole / mole). The mixture was dissolved in

hot A.R. benzene and the solution was evaporated to dryness. The mixture of crystals obtained was crushed into a powder form.

Thus, the anthracene (one gm. lot) was mixed with appropriate amounts of pyrene. The mixtures were dissolved in hot A.R. benzene and the solutions were evaporated to dryness as above.

Similarly, the pyrene (one gm. lot) was mixed with appropriate amounts of perylene, and the phosphors were prepared as described above.

2:3 STUDY OF ELECTRO OPTICAL PROPERTIES OF ORGANIC COMPOUNDS AND PHOSPHORS :

The organic compounds and various phosphors, prepared as mentioned above were subjected to the study of spectral energy distribution (S.E.D.) of their El. emission at constant voltage and frequency. The voltage and frequency dependence of the El. intensity (along with the variation in S.E.D.) of the compounds and phosphors prepared were also studied. The instruments used for these studies can be broadly divided into three units, namely,

- a) Electroluminescent cell,
- b) Source of high voltage-high frequency
a.c. field (Excitation unit),
- c) Dispersion unit for analyzing the El. emission
for different wavelengths, and
- d) Detection and measurement unit for measuring
intensity of light emitted.

a) Electroluminescent cell :

Various types of El. cells have been described earlier (Chapter D page 5). We found the following arrangement more

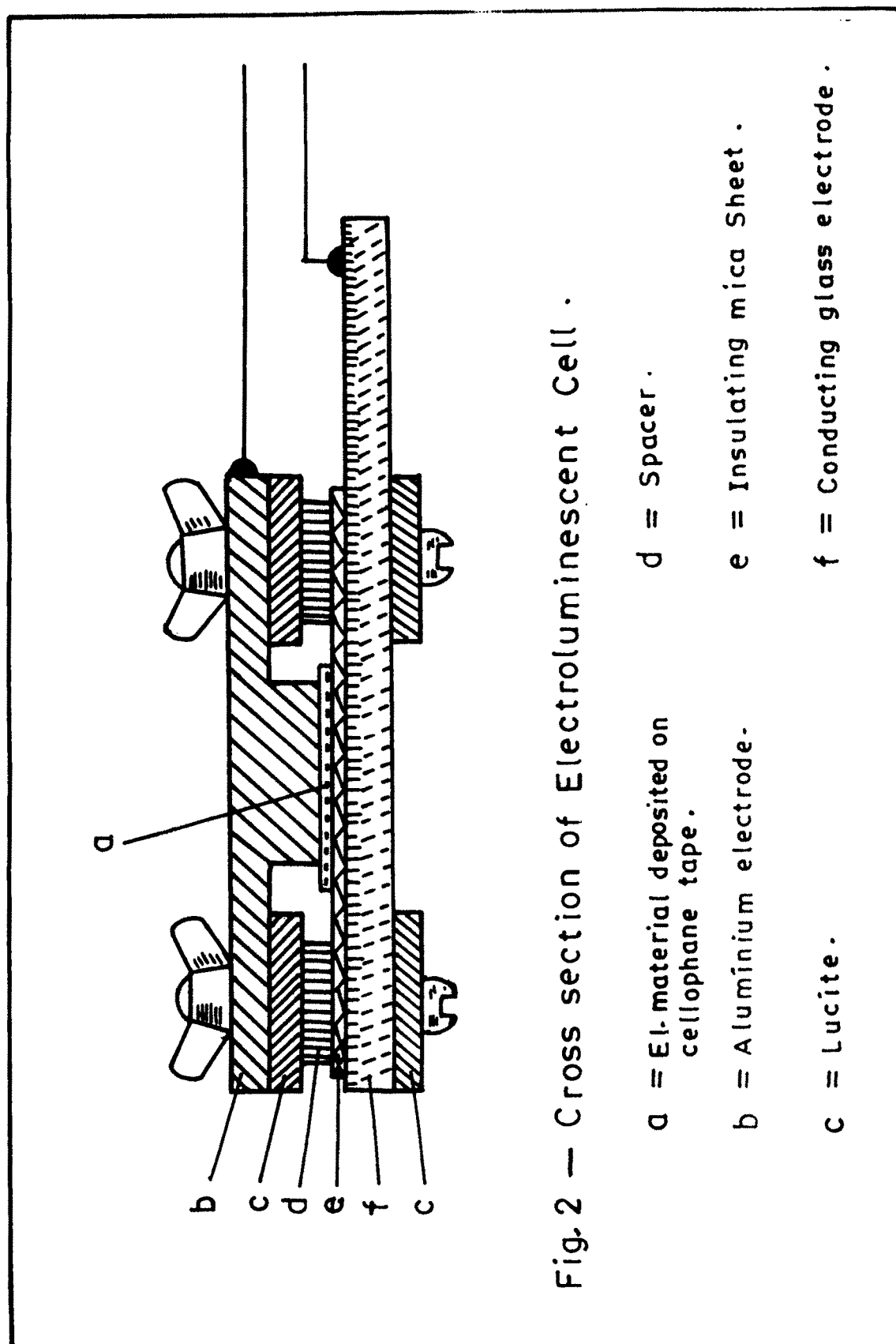


Fig. 2 — Cross section of Electroluminescent Cell .

a = EL-material deposited on
cellophane tape.

b = Aluminium electrode.

c = Lucite .

d = Spacer .

e = Insulating mica Sheet .

f = Conducting glass electrode .

convenient and satisfactory for the measurement of spectral energy distribution and relative intensity of El. emission. (cf. Fig. No. 2)

The emitter was a film of cellophane prepared by dipping it into a hot saturated solution of El. material, drying it, and placing it with melted paraffin wax between a plate of aluminium and a sheet of transparent mica of uniform thickness having high dielectric constant and sufficient breakdown strength. The cell was compressed before cooling to exclude air bubbles. The sheet of mica was covered with conducting glass, having a resistance of 400-500 Ω / cm^2 and optical transmission of the order of 80 % or more, served as second electrode. Such conducting glasses were prepared by spraying a solution of tin salt in alcohol, on to a glass plate heated to just below the melting point. Thus, the cell works as a luminous condenser, the phosphor being sandwiched between two parallel electrodes. This condenser was enclosed in an ebonite frame, provided with a small window in the middle of the top of the surface, and a screw arrangement to adjust the interelectrode distance and the pressure. From the two electrodes, leads were taken out and high electric field was applied to the El. material. The emitted light was observed through the transparent conducting glass. This assembly was found to have the following advantages.

1. A uniform pressure can be applied to the phosphor layer with the help of the screwing arrangement.
2. Inter-electrode distance can be kept constant.
3. The phosphor layers of uniform thickness ($75 \pm 5 \mu$) can easily be prepared.
4. The emission can be observed at low voltages.
5. The extraneous light is completely avoided by fixing the photomultiplier to the constant deviation spectro-meter.

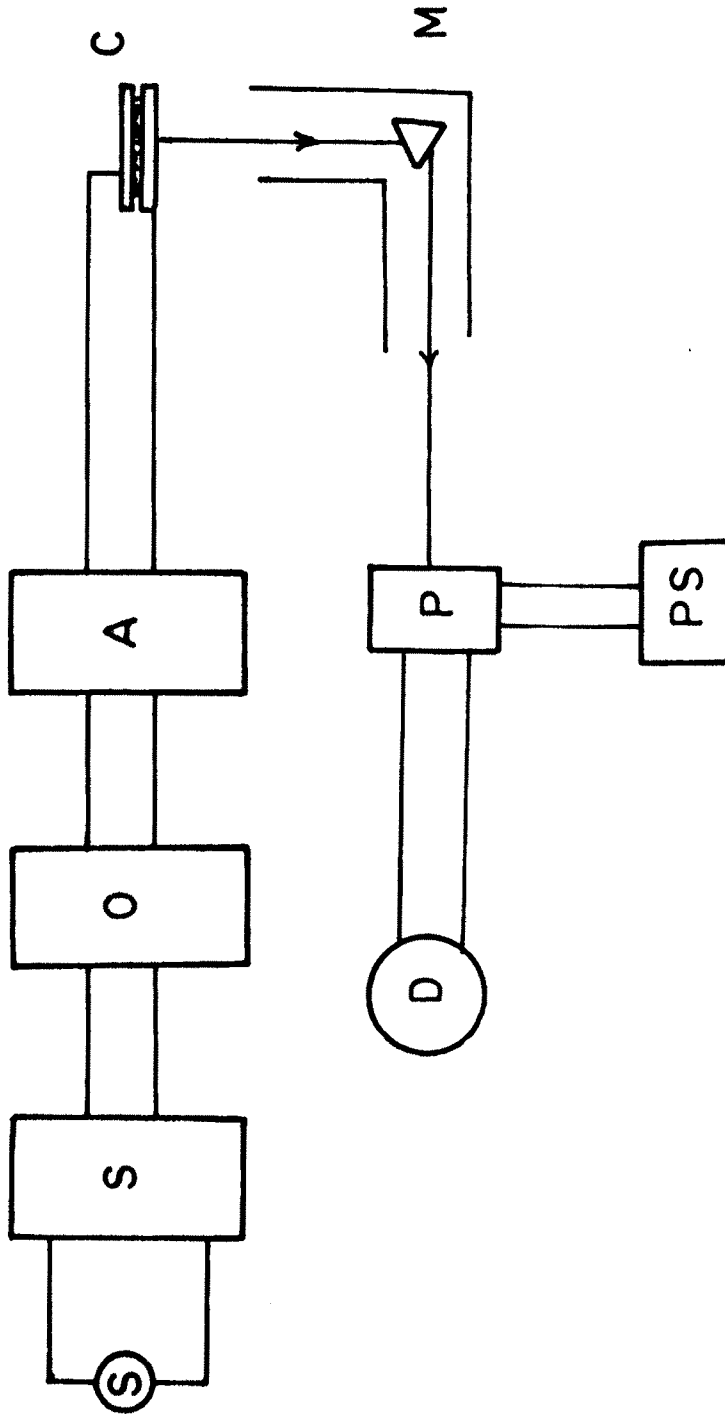
b) Source of high voltage-high frequency a.c. field.
(Excitation unit).

The excitation unit consisted of an audio-frequency oscillator (Philips India make 0-16 KHz, 0-90 volts output) whose output was fed into a high frequency-high voltage transformer (0-5 KV, Telmax, India). The output of this transformer was given to the El. cell, the voltage across the two electrodes being measured with the help of H.T. probe and Simpson Multimeter (Model 260 M).

c) Dispersion unit :

The dispersion unit was a constant deviation spectrometer with a calcite prism (Andhra Scientific Co., India). It was first adjusted for constant deviation position for green-I line of mercury (5460.7 \AA°) and then scale was calibrated for its

Fig. 3 — BLOCK DIAGRAM OF EXPERIMENTAL SET UP.



- S = STABILIZER .
- O = A.F. OSCILLATOR .
- A = STEP UP TRANSFORMER .
- C = ELECTROLUMINESCENT CELL .
- M = C.D. SPECTROMETER .
- P = PHOTOMULTIPLIER TUBE .
- PS = POWER SUPPLY OF P.M.TUBE
- D = SPOT REFLECTING GALVANOMETER

non-linearity with the help of ^amercury spectrum in the visible region (4000-7000 Å^o). The El. cell was placed at a constant distance from the slit of the spectrometer such that El. emitting area was parallel to the slit. The width of the slit and the distance between the cell and the slit were kept under close control throughout the measurement.

d) Intensity Measuring Unit :

The low intensity of El. emission was measured with the help of ^{an}IP-21 photomultiplier tube coupled with a very sensitive spot reflecting galvanometer (Polyflex type, Toshniwal, India, Sensitivity : 1.2×10^{-9} amp per mm deflection). The IP-21 tube holder was provided with a cylindrical opening which could be inserted into the spectro~~meter~~ in the place of its telescope. This arrangement helped to avoid the stray light entering the phototube and hence to minimize the dark current. The P.M. tube holder and the prism of the spectrometer were covered with a thick black cloth to avoid the extraneous light. The P.M. tube was operated at a stabilized d.c. voltage (anode to cathode voltage \approx 1000 volts) and the dark current was reduced to zero by applying an opposing current.

The intensities of El. emission were measured in arbitrary units and were corrected for the photomultiplier response with

the help of standard response curve. A necessary correction to wavelengths measured was also applied to compensate for the non-linearity of the wavelength scale.